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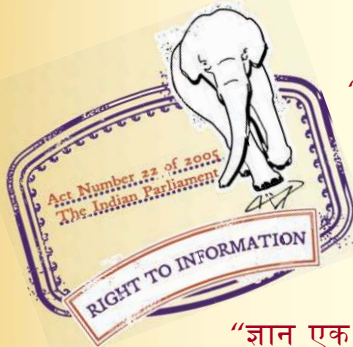
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IS 4032 (1985): Method of chemical analysis of hydraulic cement [CED 2: Cement and Concrete]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 4032 – 1985

(Reaffirmed 2005)

Edition 2.1

(1998-05)

Indian Standard

**METHOD OF CHEMICAL
ANALYSIS OF HYDRAULIC CEMENT**

(First Revision)

(Incorporating Amendment No. 1)

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

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Indian Standard

**METHOD OF CHEMICAL
ANALYSIS OF HYDRAULIC CEMENT**

(First Revision)

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(*Continued on page 2*)

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Indian Standard

METHOD OF CHEMICAL
ANALYSIS OF HYDRAULIC CEMENT
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 October 1985, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 Standard methods of testing cement are essential adjuncts to the cement specifications because faulty test methods may lead to erroneous conclusions as to the quality of cement. The Cement and Concrete Sectional Committee has, therefore, formulated methods of chemical and physical tests of hydraulic cements in details in two separate standards which could be referred to in each of the specification for different types of hydraulic cement. This standard covers methods of chemical analysis of hydraulic cement while methods of physical tests are covered in IS : 4031-1968*.

0.3 This standard was first published in 1968. A need has been felt to introduce rapid methods of chemical analysis of cement to cut down the time taken for determination of major constituents. In this revision, therefore, rapid complexometric (EDTA) methods developed by National Council for Cement and Building Materials have been included as alternate methods for estimation of CaO, MgO, Fe₂O₃ and Al₂O₃ in ordinary and low heat Portland cement, rapid hardening Portland cement, Portland slag cement and Portland pozzolana cement. In case of dispute or doubtful marginal values, the gravimetric methods for estimation of CaO, MgO and Al₂O₃ and potassium permanganate method for estimation of Fe₂O₃ shall be taken as referee method. For rapid complexometric methods of analysis, photometric end point detector shall be used for better detection of sharp end point where sufficient skill is not available.

0.4 This edition 2.1 incorporates Amendment No. 1 (May 1998). Side bar indicates modification of the text as the result of incorporation of the amendment.

*Methods of physical tests for hydraulic cement.

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0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard gives the procedure for the chemical analysis of the different types of hydraulic cement.

1.2 In addition to the tests for chemical requirements prescribed in different cement specifications, this standard also covers test for determination of free lime in Portland cement, pozzolanicity test for Portland pozzolana cement and staining test for masonry cement.

NOTE — Analysis of hydraulic cement may be done using rapid methods such as atomic absorption spectrophotometer method (*see* IS : 12813-1989†) or X-ray fluorescence spectrometer method (*see* IS : 12803-1989‡), or calorimetric analysis (*see* IS : 12423-1988§) not covered in this standard. However, in case of dispute or doubtful marginal values, the method given in this standard shall be taken as referee method.

2. SAMPLING

2.1 The samples of the cement shall be taken as per requirements of IS : 3535-1966|| and the relevant standard specification for the type of cement being tested. The representative sample of the cement selected as above shall be thoroughly mixed before using.

3. REPRODUCIBILITY OF RESULTS

3.1 Blank determinations shall be made on the reagents for each constituent in the cement and the corrections shall be applied where necessary. In all cases check determinations shall be made and repeated if satisfactory checks are not obtained. The difference between check determinations shall not exceed 0.2 percent for silica and alumina, and 0.1 percent for other constituents.

The difference between check determinations by EDTA method shall not exceed 0.2 percent for calcium oxide and magnesia, 0.15 percent for alumina and 0.1 percent for ferric oxide.

*Rules for rounding off numerical values (*revised*).

†Method of analysis of hydraulic cement by atomic absorption spectrophotometer.

‡Method of analysis of hydraulic cement by X-ray fluorescence spectrometer.

§Method for calorimetric analysis of hydraulic cement.

||Methods of sampling hydraulic cements.

4. CHEMICAL ANALYSIS OF HYDRAULIC CEMENTS OTHER THAN PORTLAND SLAG CEMENT AND PORTLAND POZZOLANA CEMENT

4.1 Reagents and Special Solutions — Unless specified otherwise, pure chemicals of analytical reagent grade shall be employed in tests, and distilled water (*see* IS : 1070-1977*) shall be used where the use of water as a reagent is intended. The following reagents and special solutions of analytical reagent grades are required (*see* IS : 2263-1979† and IS : 2316-1968‡).

4.1.1 Reagents

4.1.1.1 Hydrochloric acid — sp gr 1.16 (conforming to IS : 265-1976§).

4.1.1.2 Nitric acid — sp gr 1.42 (conforming to IS : 264-1976||).

4.1.1.3 Sulphuric acid — sp gr 1.84 (conforming to IS : 266-1977¶).

4.1.1.4 Phosphoric acid — sp gr 1.7.

4.1.1.5 Hydrofluoric acid — 40 percent.

4.1.1.6 Ammonium Hydroxide — sp gr 0.90.

4.1.1.7 Acetic acid — glacial.

4.1.1.8 Diethylamine — liquid.

4.1.1.9 Sulphosalicylic acid — solid.

4.1.1.10 Potassium periodate — solid.

4.1.2 Dilute Solutions of Reagents — prepare the following dilute solutions by diluting the reagent with distilled water.

4.1.2.1 Hydrochloric acid — 1 : 1, 1 : 3, 1 : 10 and 1 : 99 (by volume).

4.1.2.2 Nitric acid — 1 : 4, 1 : 33 (by volume).

4.1.2.3 Ammonium hydroxide — 1 : 1 and 1 : 6 (by volume).

4.1.2.4 Sulphuric acid — 1 : 3 (by volume).

4.1.2.5 Phosphoric acid — 1 : 3 (by volume).

4.1.2.6 Sodium hydroxide solution — 4 N (approximately). Dissolve 80 g of sodium hydroxide in 500-ml volumetric flask and make up to the mark with distilled water.

4.1.2.7 Glycerol — 1 : 1 (by volume).

*Specification for water for general laboratory use (*second revision*).

†Methods of preparation of indicator solutions (*first revision*).

‡Methods of preparation of standard solution for colorimetric and volumetric analysis (*first revision*).

§Specification for hydrochloric acid (*second revision*).

|| Specification for nitric acid (*second revision*).

¶Specification for sulphuric acid (*second revision*).

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4.1.2.8 Triethanolamine — 1 : 1 (by volume).

4.1.2.9 Ammonium Acetate — 50 percent solution. Weigh 50 g of ammonium acetate and make up the volume to 100 ml with distilled water.

4.1.3 Stannous Chloride Solution — Dissolve 50 g of stannous chloride (SnCl_2) in 100 ml of hydrochloric acid and dilute to one litre. Keep a few pieces of pure metallic tin in contact with the solution.

4.1.4 Mercuric Chloride Solution — Prepare a saturated solution [5.6 g of mercuric chloride (HgCl_2) in 100 ml of water should give a saturated solution at ordinary room temperature].

4.1.5 Manganese Sulphate Solution — Dissolve 35 g of crystalline manganese sulphate ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$) in 250 ml of water. Add 70 ml of phosphoric acid (sp gr 1.7) and 65 ml of sulphuric acid (sp gr 1.84). Dilute to 500 ml.

4.1.6 Standard Potassium Permanganate Solution — 0.05 N. Dissolve about 1.5 g of potassium permanganate (KMnO_4) in one litre of water. Allow it to stand for a week; filter through asbestos mat and keep in a dark place after standardizing it with pure sodium oxalate.

4.1.7 Ammonium Nitrate Solution — Dissolve 2 g of ammonium nitrate (NH_4NO_3) in 100 ml of water.

4.1.8 Methyl Red — 0.1 percent solution.

4.1.9 Ammonium Oxalate Solution — Dissolve 50 g of ammonium oxalate [$(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$] in one litre of water. Also prepare a dilute solution containing one gram of salt per litre.

4.1.10 Ammonium Hydrogen Phosphate Solution — Dissolve 250 g of ammonium hydrogen phosphate in one litre of water.

4.1.11 Barium Chloride Solution — Dissolve 100 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in one litre of water.

4.1.12 Bismuth Nitrate Solution — Weigh about 4 g of bismuth nitrate pentahydrate [$\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$] in 500-ml beaker. Add 20 ml of acetic acid. Stir and dilute with about 40 ml water. Filter and transfer the solution to one litre volumetric flask and make up the volume to the mark with distilled water.

4.1.13 Buffer Solution — pH 10 — Dissolve 70 g of ammonium chloride in 570 ml of ammonium hydroxide (sp gr 0.90) and make up to one litre with distilled water.

4.1.14 Standard Zinc Solution — 0.01 M. Dissolve accurately weighed 0.6537 g of granulated zinc in a minimum quantity of dilute hydrochloric acid (1 : 1). Make up to the mark with distilled water in one litre volumetric flask.

4.1.15 Standard EDTA Solution — 0.01 M. Dissolve 3.7224 g of disodium ethylenediamine tetra acetate dihydrate in 400 ml hot water and make up the volume in one litre flask. Take 10 ml of standard zinc solution in an Erlenmeyer flask. Add 20 ml buffer solution of pH 10 and warm to 50 to 60°C. Add 50 mg Eriochrome Black-T indicator and titrate with EDTA till the colour changes from wine red to clear blue. Note the volume of EDTA used (V). Calculate the molarity of EDTA as follows:

$$\text{Molarity of EDTA} = \frac{0.01 \times 10}{V}$$

Adjust the molarity to 0.01 M, if required.

4.1.16 Eriochrome Black-T — Grind 100 mg of indicator with 10 g of sodium chloride till homogeneous mixture is obtained and store in an airtight polyethylene bottle.

4.1.17 Xylenol Orange — Grind 100 mg of indicator with 10 g of potassium nitrate till a homogeneous mixture is obtained and store in an airtight container.

4.1.18 Patton-Reeders Indicator — Grind 100 mg of indicator with 10 g of sodium or potassium sulphate till a homogeneous mixture is obtained and store in an airtight bottle.

4.1.19 Thymol Phthalexone Indicator — Grind 100 mg of thymol phthalexone indicator with 10 g of potassium nitrate till a homogeneous mixture is obtained and store in an airtight container.

4.1.20 Thymol Blue — 0.1 percent solution in ethyl alcohol (W/V).

4.2 Loss on Ignition — Heat 1.00 g of the sample for 15 minutes in a weighed and covered platinum crucible (a porcelain crucible may also be used) of 20 to 25 ml capacity by placing it in a muffle furnace at temperature between 900° and 1000°C; cool and weigh. Check the loss in weight by a second heating for 5 minutes and re-weigh. Record the loss in weight as the loss on ignition and calculate the percentage of loss on ignition to the nearest 0.1. Calculate the percent loss on ignition as below:

$$\text{Percent loss on ignition} = \text{loss in weight} \times 100$$

4.3 Silica

4.3.1 Transfer 0.5 g of the sample to an evaporating dish, moisten with 10 ml of distilled water at room temperature to prevent lumping, add 5 to 10 ml of hydrochloric acid, and digest with the aid of gentle heat and agitation until the sample is completely dissolved. Dissolution may be aided by light pressure with the flattened end of a glass rod. Evaporate the solution to dryness on a steam-bath. Without heating the residue any further treat it with 5 to 10 ml of hydrochloric acid and then with an equal amount of water, or pour at once upon the residue 10 to 20 ml

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of hydrochloric acid (1 : 1). Then cover the dish and digest for 10 minutes on the water-bath or hot-plate. Dilute the solution with an equal volume of hot water, immediately filter through an ashless filter paper (Whatman No. 40 or its equivalent), and wash the separated silica (SiO_2) thoroughly with hot water and reserve the residue.

4.3.2 Again evaporate the filtrate to dryness, baking the residue in an oven for one hour at 105 to 110°C. Then treat the residue with 10 to 15 ml of hydrochloric acid (1 : 1) and heat the solution on water-bath or hot-plate. Dilute the solution with an equal volume of hot water catch and wash the small amount of silica it contains on another filter paper. Reserve the filtrate and washings for the determination of combined alumina and ferric oxide.

4.3.3 Transfer the papers containing the residues to a weighed platinum crucible. Dry and ignite the papers, first at a low heat until the carbon of the filter paper is completely consumed without inflaming, and finally at 1 100 to 1 200°C until the weight remains constant.

4.3.4 Treat the ignited residue thus obtained, which will contain small amounts of impurities, with 1 to 2 ml of distilled water, about 10 ml of hydrofluoric acid and 2 drops of sulphuric acid and evaporate cautiously to dryness. Finally heat the small residue at 1 050 to 1 100°C for a minute or two; cool and weigh. The difference between this weight and the weight of ignited sample represents the amounts of silica:

$$\text{Silica percent} = 200 (W_1 - W_2)$$

where

W_1 = weight of silica + (insoluble impurities – residue), and

W_2 = weight of impurities.

NOTE — For routine analysis, treatment with hydrofluoric acid may be omitted.

4.3.4.1 To this amount of silica, add the amount of silica recovered from the residue derived from the combined precipitates of alumina and ferric oxide as indicated under **4.3.5**.

4.3.5 Add 0.5 g of sodium or potassium persulphate to the crucible and heat below red heat until the small residue of impurities is dissolved in the melt. Cool, dissolve the fused mass in water, and add it to the filtrate and washings reserved for the determination of the combined alumina and ferric oxide.

4.4 Combined Ferric Oxide and Alumina

4.4.1 To the filtrate reserved in **4.3.2** which shall have a volume of about 200 ml, add a few drops of the methyl red indicator, and heat to boiling, adding a few drops of bromine water or concentrated nitric

acid during boiling in order to oxidize any ferrous iron to the ferric condition. Then treat with ammonium hydroxide (1 : 1) drop by drop until the colour of the solution becomes distinctly yellow and smells of ammonia. Bring to boiling the solution containing the precipitates of aluminium and ferric hydroxides and boil for one minute. In case of difficulty from bumping experienced while boiling the ammoniacal solution, substitute the one-minute boiling period by a digestion period of 10 minutes on a steam-bath or a hot-plate of approximately the same temperature as of a steam-bath. Allow the precipitate to settle (which should not take more than 5 minutes), filter through an ashless filter paper (Whatman No. 41 or equivalent) and wash with 2 percent hot ammonium nitrate solution. Set aside the filtrate and washings.

NOTE — Two drops of methyl red indicator should be added to the ammonium nitrate solution, followed by ammonium hydroxide (1 : 1) added dropwise until the colour just changes to yellow. If the colour reverts to red at any time due to heating, it should be brought back to yellow by addition of a drop of ammonium hydroxide (1 : 1).

4.4.2 Transfer the precipitate and filter paper to the same beaker in which the first precipitation was effected. Dissolve the precipitate in hot hydrochloric acid (1 : 3), dilute the solution to about 100 ml and re-precipitate the hydroxides (*see* Note). Filter the solution and wash the precipitate with two 10-ml portions of hot ammonium nitrate solution. Combine the filtrate and washings, with the filtrate set aside under **4.4.1** and reserve for the determination of calcium oxide.

NOTE — Alternatively, after the precipitate of the combined hydroxide is dissolved in hydrochloric acid, it may be diluted up to 100 ml in a volumetric flask. A portion may be used for precipitating and estimating the hydroxides and another portion may be used for estimating ferric oxide (instead of dissolving the sample afresh).

4.4.3 Place the precipitate in a weighed platinum crucible, heat slowly until the papers are charred and finally ignite to constant weight at 1 050 to 1 100°C taking care to prevent reduction, and weigh as combined alumina and ferric oxide.

4.4.3.1 Calculation — Calculate the percentage of combined ferric oxide and alumina by multiplying the weight in grams of the residue by 200 (100 divided by weight of sample used):

$$R_2O_3 \text{ percent} = \text{weight of residue} \times 200$$

4.4.4 If silica is suspected to be carried into the filtrate used for this estimation, proceed as given in **4.4.4.1**.

4.4.4.1 Treat the residue in the crucible with a drop of water, about 5 ml of hydrofluoric acid and drop of sulphuric acid and evaporate cautiously to dryness. Finally heat the crucible at 1 050 to 1 100°C for 1 or 2 minutes, cool and weigh. The difference between this weight and the weight previously obtained represents the amount of residual silica. Subtract this amount from the weight of ferric oxide and

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alumina found under 4.4.3 and add the same amount to the amount of silica found under 4.3.4.

4.5 Ferric Oxide

4.5.1 Method 1 (Potassium Permanganate Method) — To one gram of the sample, add 40 ml of cold water and, while the mixture is being stirred vigorously, add 15 ml of hydrochloric acid. If necessary, heat the solution and grind the cement with the flattened end of a glass rod until it is evident that the cement is digested fully. Heat the solution to boiling and treat it with stannous chloride solution added drop by drop while stirring, until the solution is decolourized. Add a few drops of stannous chloride solution in excess and cool the solution to room temperature. Rinse the inside of the vessel with water, and add 15 ml of a saturated solution of mercuric chloride in one lot. Stir, add 25 ml of manganese sulphate solution and titrate with standard solution of potassium permanganate until the permanent pink colour is obtained. Calculate iron as ferric oxide.

4.5.2 Method 2 (EDTA Method)

4.5.2.1 Prepare filtrate as given in 4.3.2 and 4.3.5. Mix the filtrates and make up the volume in a 250-ml volumetric flask.

4.5.2.2 Take 25 ml of solution reserved in 4.5.2.1 and add dilute ammonium hydroxide (1 : 6) till turbidity appears. Clear the turbidity with a minimum amount of dilute hydrochloric acid (1 : 10) and add a few drops in excess to adjust the pH to approximately 1 to 1.5. Shake well. Then add 100 mg of sulphosalicylic acid and titrate with 0.01 M EDTA solution carefully to a colourless or pale yellow solution.

4.5.2.3 Calculation — Calculate the percentage of Fe_2O_3 as below:

1 ml of 0.01 M EDTA \equiv 0.7985 mg of Fe_2O_3

$$\text{Iron oxide (Fe}_2\text{O}_3\text{) percent} = \frac{0.7985 \times V}{W}$$

where

V = Volume of EDTA used in ml, and

W = Weight of the sample in g.

4.6 Alumina

4.6.1 Method 1 (Gravimetric Method) — Subtract the calculated weight of ferric oxide and small amount of silica from the total weight of oxides found under 4.4.3. The remainder is the weight of alumina and small amounts of other oxides which are to be reported as alumina.

4.6.2 Method 2 (EDTA Method) — Take 25 ml of solution reserved under 4.5.2.1 and titrate iron at pH approximately 1 to 1.5 with EDTA

using sulphosalicylic acid as indicator as given in 4.5.2.2. Add 15 ml standard EDTA solution. Add 1 ml of phosphoric acid (1 : 3), 5 ml of sulphuric acid (1 : 3) and one drop of thymol blue into the titration flask. Add ammonium acetate solution by stirring until the colour changes from red to yellow. Add 25 ml of ammonium acetate in excess to obtain pH approximately 6. Heat the solution to boiling for one minute and then cool. Add 50 mg of solid xylenol orange indicator and bismuth nitrate solution slowly with stirring until the colour of the solution changes from yellow to red. Add 2 to 3 ml of bismuth nitrate solution in excess. Titrate with 0.01 M EDTA solution to a sharp yellow end point red colour.

4.6.2.1 Calculation — Calculate the percentage of Al_2O_3 as below:

$$V = V_1 - V_2 - (V_3 \times E)$$

where

V = Volume of EDTA for alumina in ml,

V_1 = Total volume of EDTA used in the titration in ml,

V_2 = Volume of EDTA used for iron (see 4.5.2.2) in ml,

V_3 = Total volume of bismuth nitrate solution used in the titration in ml, and

E = Equivalence of 1 ml of bismuth nitrate solution (see Note below)

1 ml of 0.01 M EDTA \equiv 0.5098 mg of Al_2O_3

$$\text{Aluminium oxide (Al}_2\text{O}_3\text{) percent} = \frac{0.5098 \times V}{W}$$

where

W = Weight of the sample in g.

NOTE — Equivalence of bismuth nitrate solution is obtained as follows:

Transfer 100 ml of bismuth nitrate solution to a 500-ml flask and dilute with about 100 ml distilled water. Add a few drops of thymol blue solution and ammonium acetate solution until the colour changes from red to yellow. Add 50 mg of xylenol orange indicator and titrate with 0.01 M EDTA solution until the colour changes from red to yellow. The equivalence (ml of EDTA) of 1 ml of bismuth nitrate solution is calculated as follows:

$$E = \frac{V_4}{100}$$

where

V_4 = Volume of EDTA solution in ml.

4.7 Calcium Oxide

4.7.1 Method 1 (Gravimetric Method) — Acidify the combined filtrates set aside under 4.4.2 with hydrochloric acid and evaporate them to a

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volume of about 100 ml. Add 40 ml of saturated bromine water to the hot solution and immediately add ammonium hydroxide until the solution is distinctly alkaline. Boil the solution for 5 minutes or more, making certain that the solution is at all times distinctly alkaline. Allow the precipitate to settle; filter and wash with hot water. Wash the beaker and filter once with nitric acid (1.33) that has been previously boiled to expel nitrous acid, and finally with hot water. Discard any precipitate (of manganese dioxide) that may be left on the funnel. Acidify the filtrate with hydrochloric acid and boil until all the bromine is expelled. Add 5 ml of hydrochloric acid, dilute to 200 ml, add a few drops of methyl red indicator and 30 ml of warm ammonium oxalate solution. Heat the solution to 70 to 80°C and add the ammonium hydroxide (1 : 1) dropwise, while stirring, until the colour changes from red to yellow. Allow the calcium oxalate precipitate to stand without further heating for one hour, with occasional stirring during the first 30 minutes; filter through Whatman filter paper No. 42 or equivalent, and wash moderately with cold 0.1 percent ammonium oxalate solution. Set aside the filtrate and washings for estimating magnesia.

4.7.1.1 Dry the precipitate in a weighed, covered platinum crucible, char the paper without inflaming, burn the carbon at as low temperature as possible, and finally heat with the crucible tightly covered in an electric furnace or over a blast lamp at a temperature of 1 100 to 1 200°C. Cool in a desiccator (to guard against absorption of moisture by ignited calcium oxide) and weigh as calcium oxide. Repeat the ignition to a constant weight.

4.7.1.2 *Calculation* — Calculate the percentage of CaO by multiplying the weight in grams of 200 residue (CaO) by 200 [100 divided by the weight of sample used (0.5 g)]:

$$\text{CaO percent} = \text{weight of residue} \times 200$$

4.7.2 *Method 2 (EDTA Method)* — Take 10 ml of solution reserved under **4.5.2.1** in a 250-ml conical flask. Add 5 ml of 1 : 1 glycerol with constant stirring and 5 ml of diethylamine. To this add 10 ml of 4N sodium hydroxide solution and shake well to adjust pH to highly alkaline range of 12 or slightly more. Add approximately 50 ml of distilled water and 50 mg of solid Patton-Reeder's indicator. Titrate against 0.01 M EDTA solution to a sharp change in colour from wine red to clear blue (see Note).

4.7.2.1 *Calculations* — Calculate the percentage of CaO as below:

1 ml of 0.01 M EDTA \equiv 0.5608 mg of CaO

$$\text{Calcium Oxide (CaO) percent} = \frac{0.05608 \times 25 \times V}{W}$$

where

V = Volume of EDTA used in ml, and

W = Weight of the sample in g.

NOTE — Manganese interferes with the estimation of CaO. In presence of manganese, the procedure specified in 6.7.2 may be adopted.

4.8 Magnesia

4.8.1 Method 1 (Gravimetric Method) — Acidify the filtrate set aside under 4.7.1 with hydrochloric acid and concentrate to about 150 ml. Add to this solution about 10 ml of ammonium hydrogen phosphate (250 g/l) and cool the solution by placing in a beaker of ice water. After cooling, add ammonium hydroxide drop by drop, while stirring constantly, until the magnesium ammonium phosphate crystals begin to form, and then add the reagent in moderate excess (5 to 10 percent of the volume of the solution), the stirring being continued for several minutes. Set the solution aside for at least 16 hours in a cool atmosphere and then filter, using Whatman No. 42 filter paper or its equivalent. Wash the precipitate with ammonium nitrate wash solution (100 g ammonium nitrate dissolved in water, 200 ml of ammonium hydroxide added and diluted to one litre). Place in a weighed platinum crucible, slowly char the paper and carefully burn off the resulting carbon. Ignite the precipitate at 1 100 to 1 200°C to constant weight taking care to avoid bringing the pyrophosphate to melting. The product of the weight of magnesia (MgO), pyrophosphate obtained and a factor, 0.3621, shall be the magnesium content of the material tested.

4.8.1.1 Calculation — Calculate the percentage of MgO as below:

$$\text{MgO percent} = W \times 72.4$$

where

W = grams of residue ($\text{Mg}_2\text{P}_2\text{O}_7$), and

72.4 = molecular ratio of 2 MgO to $\text{Mg}_2\text{P}_2\text{O}_7$ (0.362), divided by weight of sample used (0.5 g) and multiplied by 100.

4.8.2 Method 2 (EDTA Method) — Take 10 ml of solution reserved under 4.5.2.1. Add 5 ml of 1 : 1 triethanolamine with constant shaking and 20 ml of buffer solution pH 10. Add 50 mg of the solid thymol phthalexone indicator followed by approximately 50 ml of distilled water. Titrate it against standard 0.01 M EDTA solution until the colour changes from blue to clear pink. This titration gives the sum of calcium and magnesium oxide present in the solution. Titre value of magnesium oxide is obtained by subtracting the titre value of calcium oxide from the total titre value.

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4.8.2.1 Calculations — Calculate the percentage of MgO as given below:

1 ml of 0.01 M EDTA \equiv 0.4032 mg of MgO

$$\text{Magnesium oxide (MgO) percent} = \frac{0.4032 \times 25 \times (V_1 - V)}{W}$$

where

V_1 = Volume of EDTA used in this titration in ml,

V = Volume of EDTA used in CaO determination in ml, and

W = Weight of the sample in g.

4.9 Sulphuric Anhydride — To one gram of the sample, add 25 ml of cold water, and while the mixture is stirred vigorously add 5 ml of hydrochloric acid. If necessary, heat the solution and grind the material with flattened end of a glass rod until it is evident that the decomposition of the cement is complete. Dilute the solution to 50 ml and digest for 15 minutes at a temperature just below boiling. Filter and wash the residue thoroughly with hot water. Set aside the filter paper with the residue. Dilute the filtrate to 250 ml and heat to boiling. Add slowly drop by drop, 10 ml of hot barium chloride (100 g/l) solution and continue the boiling until the precipitate is well formed. Digest the solution on a steam-bath for 4 hours or preferably overnight. Filter the precipitate through a Whatman No. 42 filter paper or equivalent and wash the precipitate thoroughly. Place the filter paper and the contents in a weighed platinum or porcelain crucible and slowly incinerate the paper without inflaming. Then ignite at 800 to 900°C, cool in a desiccator and weigh the barium sulphate obtained, calculate the sulphuric anhydride content of the material taken for the test.

4.9.1 Calculation — Calculate the percentage of SO₃ as follows:

$$\text{SO}_3 \text{ percent} = W \times 34.3$$

where

W = weight of residue (BaSO₄) in g; and

34.3 = molecular ratio of SO₃ to BaSO₄ (0.343), multiplied by 100.

4.10 Insoluble Residue — Digest the filter paper containing the residue set aside under 4.9 in 30 ml of hot water and 30 ml of 2N sodium carbonate solution maintaining constant volume, the solution being held at just below the boiling point for 10 minutes. Filter and wash with dilute hydrochloric acid (1 : 99) and finally with hot water till the residue is free from chlorides. Ignite the residue in a tared crucible at 900 to 1 000°C, cool in a desiccator and weigh.

4.11 Sodium Oxide and Potassium Oxide — This method covers the determination of sodium oxide and potassium oxide by flame photometry using direct intensity procedure.

4.11.1 Reagents and Materials

4.11.1.1 The following reagents and materials are required:

Calcium Carbonate — The calcium carbonate used in the preparation of the calcium chloride stock solution (see 4.11.1.2) shall contain not more than 0.020 percent total alkalies as sulphate.

Hydrochloric Acid (HCl) (*sp gr 1.19*) — concentrated hydrochloric acid.

Potassium Chloride (KCl)

Sodium Chloride (NaCl)

4.11.1.2 Preparation of solution

a) *Calcium Chloride Stock Solution* — Add 300 ml of water to 112.5 g of calcium carbonate in a 1 500-ml beaker. While stirring slowly add 500 ml of hydrochloric acid. Cool the solution to room temperature, filter into a 1-litre volumetric flask, dilute to one litre, and mix thoroughly. This solution contains the equivalent to 63 000 ppm (6.30 percent) calcium oxide.

b) *Sodium Chloride-Potassium Chloride Stock Solution* — Dissolve 1.885 g of sodium chloride and 1.583 g of potassium chloride (both dried at 105 to 110°C for several hours prior to weighing) in water. Dilute to one litre in a volumetric flask and mix thoroughly. This solution contains the equivalent of 1 000 ppm (0.10 percent) each of sodium oxide and potassium oxide.

c) *Standard Solution* — Prepare the standard solutions prescribed in Table 1 (see Note). Measure the required volume of sodium chloride-potassium chloride stock solutions in calibrated pipettes or burettes. The calcium chloride stock solutions may be measured in appropriate graduated cylinders. Place each solution in a volumetric flask, dilute to the indicated volume and mix thoroughly.

NOTE — The solutions listed in Table 1 represent the minimum number required. A greater number of solutions and different concentrations may be used provided there is no more than 25 ppm difference between the alkali concentrations of any solution and the solution containing the next larger amount of alkalies.

4.11.2 Laboratory Containers — All glassware shall be made of borosilicate glass. All polyethylene containers shall be made of a high-density polyethylene having a wall thickness of at least one mm.

4.11.3 Flame Photometer — Any type of flame photometer may be used, provided it may be demonstrated that the required degree of

TABLE 1 STANDARD SOLUTIONS

[Clause 4.11.1.2 (c)]

STANDARD SOLUTION NUMBER	CONCENTRATION OF ALKALI (EXPRESSED) AS Na ₂ O OR K ₂ O IN STANDARD SOLUTION	VOLUME OF CALCIUM CHLORIDE STOCK SOLUTION USED	VOLUME OF NaCl-KCl STOCK SOLUTION USED	FINAL VOLUME OF SOLUTION
	ppm	ml	ml	ml
1	100	200	200	2 000
2	75	100	75	1 000
3	50	100	50	1 000
4	35	100	25	1 000
5	10	100	10	1 000
6	0	100	0	1 000
7	100	0*	100	1 000

*The calcium-free solution is for use only in determining the correct position of the wavelength selector for maximum responses to sodium oxide and potassium oxide.

accuracy and precision as indicated below is obtained (*see* Note). The instrument shall consist of an atomizer and burner; suitable pressure regulating devices and gauges for fuel and air or oxygen; an optical system consisting of suitable light-dispersing or filtering devices capable of preventing excessive interference from wavelengths of light other than that being measured; and a photosensitive indicating device capable of measuring direct intensity.

NOTE — After such accuracy is established for a specific instrument, further tests of instrument accuracy are not required except in cases of dispute when it has to be demonstrated that the instrument gives results within the prescribed degree of accuracy by a single series of tests using the designated standard samples. For normal laboratory testing, it is recommended that the accuracy of the instrument be routinely checked by the use of either a National Bureau of Standards 'Standard Cement' or cement of known alkali content.

4.11.3.1 Precision of the instrument — To establish that an instrument provides the desired degree of precision and accuracy, tests shall be made with the NBS Standard Cement Sample No. 1013, 1015 and 1016, or any other cement samples having alkali oxide (Na₂O) and K₂O contents equal to the appropriate certificate value for NBS Standard Sample No. 1013, 1015 and 1016.

Using the procedures described in **4.11.3.2** make individual determination with each of the three designated standard cements on three different days. The average of the results for each alkali oxide shall not differ from the appropriate certificate value by more than the

following amounts (see Note):

	<i>Sodium Oxide</i> (Na ₂ O)	<i>Potassium Oxide</i> (K ₂ O)
NBS Standard Sample No.1 013	± 0.02	± 0.02
NBS Standard Sample No.1 015	± 0.02	± 0.03
NBS Standard Sample No.1 016	± 0.03	± 0.01

The maximum acceptable difference in the percentage of each alkali between the lowest and highest value obtained shall be 0.04.

NOTE 1 — In the absence of NBS Standard Samples, samples of Indian cements having alkali oxide contents (Na₂O and K₂O) and other requirements equivalent to those of NBS Standard Sample No. 1013, 1015 and 1016 may be used.

NOTE 2 — The presence of silica (SiO₂) in solution effects the accuracy of some flame photometers. In cases where an instrument fails to provide results within the prescribed degree of accuracy, outlined above tests shall be made on solutions from which the SiO₂ has been removed. For this removal, proceed as follows:

Place 1.000 ± 0.001 g of cement in a platinum evaporating dish and disperse with 10 ml of water using a swirling motion. While still swirling, add 5.0 ml of hydrochloric acid all at once. Break up any lumps with a flat-end stirring rod and evaporate to dryness on a steam-bath. Make certain that the gelatinous appearance is no longer evident. Treat the residue with 2.5 ml of hydrochloric acid and about 20 ml of water. Digest on a steam-bath for 5 to 10 minutes and filter immediately through a 9-cm medium texture filter paper into a 100-ml volumetric flask. Wash thoroughly with repeated small amounts of hot water until the total volume of solution is 80 to 95 ml. Cool to room temperature, dilute to the mark, and mix thoroughly.

When it has been demonstrated that the removal of silica (SiO₂) is necessary to obtain the required accuracy described above for a specific flame photometer, silica (SiO₂) shall always be removed when making analysis that are used as the basis for rejection of a cement for failure to comply with specification or where specification compliance may be in question.

4.11.3.2 Calibration of apparatus — Detailed steps for putting the instrument into operation will vary considerably with different instruments and manufacturer's instructions should be followed for special techniques or precautions to be employed in the operation, maintenance, or cleaning of the apparatus. Generally the apparatus shall be calibrated in accordance with the procedure given below:

- a) Turn on the instrument and allow it to warm up in accordance with the manufacturer's instructions (a minimum of 30 minutes is required for most instruments). Adjust fuel and oxygen or air pressures as required by the instrument being used. Light and adjust the burner for optimum operation.
- b) Where required, select the proper photocell for sodium oxide (Na₂O). For instruments with variable slit widths set the slit

opening at the previously determined optimum (*see Note*). Make any other adjustments that may be necessary to establish the proper operating conditions for the instrument.

NOTE — For instruments with a variable slit width, proceed with the slit between fully closed and one fourth open as a preliminary test. Repeat with the slit width at various settings to determine the smallest slit width that does not result in instability of the galvanometer needle when set to give full scale readings with standard solution No. 1. This setting represents the most favourable operating conditions and should be used in all subsequent tests for a specific elements.

- c) For filter instruments, select the proper filter for sodium oxide (Na_2O). For spectral dispersing devices find the correct position on the wavelength dial for the element to be determined by atomizing some of the calcium-free solution (solution No. 7) and moving the wavelength selector slowly back and forth on each side of the indicated wavelength (*see Note*) for the element until the point of maximum transmission or intensity is noted. Set the wavelength selector at the point. The sensitivity controls should be set so as to give about 95 percent of full scale transmission for this determination.

NOTE — Where the desired point for a specific element is not indicated, use a wavelength of 589 m μ for sodium oxide (Na_2O) and 767 m μ for potassium oxide (K_2O) as preliminary setting.

- d) Rinse the atomizer with each solution prior to taking readings on that solution. For instruments having total consumptions-type atomizers rinse with distilled water between tests on each solution.
- e) Atomize standard solution No. 1 (100 ppm) and adjust the proper gain or sensitivity controls to provide full scale readings (meter reading of 100 or 100 percent transmittance).
- f) Atomize standard solution No. 6 (0 ppm). For instruments equipped with a zero adjustment knob, set the meter readings at zero. For dial-transmission instruments record the indicated minimum transmittance without further adjustment. Repeat the procedure described in (e) and (f) until no further sensitivity or zero adjustments are necessary to obtain the full scale reading and zero (or the indicated minimum transmittance) when going from one solution to the other.
- g) Atomize standard solution No. 2 (75 ppm) and note the scale reading. Check the zero of minimum reading with alkali-free standard solution No. 6. Return to the 100 ppm standard solution No. 1. These last two readings serve to evaluate the reading for the 75 ppm standard. If they are within one scale division of zero (or the predetermined minimum) and 100 (or other full scale transmittance reading) respectively, the meter reading obtained

for the 75 ppm solution may be considered correct. If either the reading for the 0 or 100 ppm standard solution is not within one scale division of its predetermined value, then the previously obtained meter reading for the 75 ppm solution No. 2 shall be rejected. In the latter event, repeat the procedure described in (e) and (f) and take another reading for the 75 ppm solution. Only when the 0 ppm and 100 ppm readings are in proper agreement both before and after taking readings on an intermediate standard can be regarded as accurate.

- h) Determine and record meter or transmission reading for the 50, 25 and 10 ppm solution following the same procedure given in (g).
- j) Repeat the entire procedure given in (b) to (h) for potassium oxide (K_2O) making changes in filters, photocells, wavelength, sensitivity, and slit width as required.
- k) Plot calibration curves for each oxide, using suitable linear cross-section paper with meter reading or transmittancy represented on the ordinate and percentage of alkali oxide represented on the abscissa. Draw a smooth curve through the points.

4.11.4 Procedure

4.11.4.1 Solution of cement — Place 1.000 ± 0.001 g of the cement sample in a 150-ml beaker, disperse with 20 ml of water using a swirling motion of the beaker. While still swirling, add 5.0 ml of hydrochloric acid all at once. Dilute immediately to 50 ml with water. Break up any lumps of cement remaining undispersed with a flat-end stirring rod. Digest on the steam-bath or hot-plate for 15 minutes, then filter through a medium texture filter paper into a 100-ml volumetric flask. Wash beaker and paper thoroughly with hot water, cool contents of the flask to room temperature, dilute to 100 ml, and mix the solution thoroughly (see Note 2 under **4.11.3.1**).

4.11.4.2 Procedure for determination of sodium oxide — Warm up and adjust the instrument for the determination of sodium oxide as described in **4.11.3.2**. Immediately following the adjustment and without changing any instrumental setting, atomize the cement solution and note the scale reading (see Note 2). Select the standard solution which immediately bracket the cement solution in sodium oxide content and observe their readings. Their values should agree within one division on the scale with the values previously established during calibration of the apparatus. If not, reset the instrument controls so that the original calibration point is obtained. Finally, alternate the use of the unknown solution and the bracketing standard solution until readings for the unknown agree within one division on the transmission or meter scale and readings for the standards

similarly agree with the calibration points. Record the average of the last two readings obtained as the value for the unknown solution.

NOTE 1 — The order in determining sodium oxide or potassium oxide is optional. In all cases, however, the determination should immediately follow the adjustment of the instrument for that particular constituent.

NOTE 2 — If the reading exceeds the scale maximum, either transfer a 50-ml aliquot to a 100-ml volumetric flask or, if desired, prepare a new solution by using 0.500 g of cement and 2.5 ml of hydrochloric acid (instead of 5.0 ml) in the initial addition of acid. In case the silica has been removed from the 0.5 g sample of cement, treat the dehydrated material with 1.25 ml of hydrochloric acid and about 20 ml of water, then digest, filter and wash. In either case add 5.0 ml of calcium chloride stock solution [see 4.11.1.2 (a)] before diluting to mark with water. Determine the alkali content of this solution as described in 4.11.3.2 and multiply the percentage of alkali oxide obtained by a factor of 2.

4.11.4.3 Procedure for determination of potassium oxide — Repeat the procedure described in 4.11.4.2 except that the instrument shall be adjusted for the determination of potassium oxide.

4.11.4.4 Calculations — From the recorded averages of meter readings for potassium oxide and sodium oxide in the unknown sample, read the percentage of each oxide from its respective calibration curve. Report each oxide to the nearest 0.01 percent.

4.12 Water-Soluble Alkali

4.12.1 The determination of water-soluble alkali should not be considered as a substitute for the determination of total alkali according to 4.11. Moreover, it is not to be assumed that in this method all water-soluble alkali in the cement will be dissolved. Strict adherence to the procedure described is essential where there is a specified limit on the content of water-soluble alkali or where several lots of cement are compared on the basis of water-soluble alkali.

4.12.2 Procedure

4.12.2.1 Weigh 25 to 150 g of sample into a 400-ml beaker and add 250 ml of water (see Note). Stir thoroughly and let stand 30 min at room temperature. Stir again and filter immediately through a Buchner funnel which contains a well-seated retentive filter paper, into a 500-ml filtering flask using slight vacuum. Without washing, transfer the insoluble matter and the paper to the original beaker and rinse the funnel with 150 ml of water into the beaker containing the insoluble matter. Stir thoroughly and let stand 30 min at room temperature. Stir and filter, as above, using a fresh filter paper. Again return the insoluble matter and the paper to the original beaker. Wash the funnel with 100 ml of water into the beaker containing the insoluble matter. Stir thoroughly and let stand 30 min at room temperature. Stir and filter as above. Quantitatively transfer the filtrate to a suitable beaker. Acidify the filtrate with 5 ml of

concentrated HCl (sp gr 1.19). Stir until cloudiness disappears. Evaporate the solution to about 400 ml. Cool to room temperature and transfer quantitatively to a 500 ml volumetric flask. Dilute to 500 ml.

NOTE — The amount of sample taken for analysis should be based on the expected water-soluble alkali content. Twenty-five grams should be used when the expected percentage of either water-soluble alkali (Na_2O or K_2O) is 0.4 or more. When the expected percentage of each water-soluble alkali is 0.06 or less, a 150-g sample should be used. For intermediate amounts of water soluble alkali adjust the weight of sample as follows:

$$\text{Weight of sample} = 8/A$$

where

A = expected percentage of the alkali present in largest amount.

This procedure gives a test solution containing approximately 80 ppm of the alkali present in the largest amount.

4.12.2.2 Transfer a 50-ml aliquot of this solution to a 100-ml volumetric flask. Add 9.0 ml of the stock CaCl_2 solution (63 000 ppm CaO) described in **4.11.1.2** (a) to the 100-ml flask, and dilute the solution to 100 ml. Determine the sodium and potassium oxide content of this solution as described in **4.11.4.2** and **4.11.4.3**. Record the parts per million of each alkali in the solution in the 100-ml flask.

4.12.3 Calculations

4.12.3.1 Calculate the percentage of water-soluble alkali expressed as Na_2O , to the nearest 0.01 as follows:

$$\text{Total water-soluble alkali} = A + E$$

$$A = \frac{B}{W \times 10}$$

$$C = \frac{D}{W \times 10}$$

$$E = C \times 0.658$$

where

A = percentage of water-soluble sodium oxide (Na_2O);

W = weight of cement sample, g;

B = parts per million of Na_2O in the solution in the 100-ml flask;

C = percent of water-soluble potassium oxide (K_2O);

D = parts per million of K_2O in the 100-ml flask;

E = percentage Na_2O equivalent of the K_2O ; and

0.658 = molecular ratio of Na_2O to K_2O .

5. DETERMINATION ON FREE LIME IN PORTLAND CEMENT

5.1 Object — Three methods of test have been described for determining free lime content in Portland cement, namely, Ethanol-Glycerol Method, Ethanol-Glycerol Rapid Method and Ethylene-Glycol Method. The method described under **5.2** is more accurate and in cases of dispute, results obtained by this method shall govern. The other two methods described under **5.3** and **5.4** are given for those who wish to use shorter or more convenient procedures for routine determination of free lime content in cement.

NOTE — The Indian Standard Specification for Portland Cement does not specify requirements regarding free lime content in cement. The determination of free lime in cement is important from the point of soundness of cement mortar or concrete and from the point of view of leaching and efflorescence. Excessive free lime leads to unsoundness in cement; it may also be one of the factors that cause leaching and efflorescence in concrete and masonry work. No definite conclusions, however, have been reached in regard to the optimum content of free lime in cement. The Cement and Concrete Sectional Committee felt that a knowledge of the free lime content in cement will guide the user specially in those cases where careful control over quality of concrete or mortar needs to be exercised or where it will be disastrous to have leaching and efflorescence defacing the exposed surface. This standard has been prepared with a view to providing a method for determining free lime in cement clinker, cement, etc.

5.2 Ethanol Glycerol Method

5.2.1 Reagents — The following reagents are required.

5.2.1.1 Absolute ethanol — Absolute ethanol shall conform to the requirements of IS : 321-1964*.

5.2.1.2 Glycerol — Glycerol shall be of the analytical reagent quality conforming to a purity of about 99 percent. As normally, the water contained in glycerol is not considered an impurity, it is preferable to use anhydrous quality or alternatively, glycerol with not more than 5 percent of water. However, glycerol shall be free from acidic or aldehydic matter. The specific gravity of glycerol when tested with a pycnometer at 25°C shall be not less than 1.249.

5.2.1.3 Phenolphthalein indicator — Dissolve one gram of phenolphthalein in 100 ml of absolute ethanol and neutralize the solution with sodium hydroxide dissolved in absolute ethanol.

5.2.1.4 Glycerol-ethanol solvent — Prepare a solution consisting of one part by volume of glycerol and five parts by volume of absolute ethanol. To each litre of this solution, add 2 ml of the indicator. The solvent should be slightly alkaline (*see* Note 1) to the indicator. If the solvent is colourless, add a dilute solution of sodium hydroxide in absolute ethanol until a slight pink colour appears. If the initial colour is pink, remove it with the standard ammonium acetate solution (*see* **5.2.1.5**)

*Specification for absolute alcohol (*revised*).

and then add the sodium hydroxide solution until a slight pink colour appears. Heat 60 ml of the solvent to boiling. If the pink colour persists, add one small drop of the standard solution of ammonium acetate to the hot solvent. If the pink colour does not disappear, the solvent contains too much alkali, which should be reduced. The solvent, if allowed to stand for a considerable length of time, may become slightly acidic and should be checked and, if necessary, re-adjusted from time to time (*see* Note 2).

NOTE 1 — While a solvent that is exactly neutral at the boiling point is the ideal, it is difficult to prepare due to the end point not being sharp and the danger of adding too much ammonium acetate. The error due to slight alkalinity is not more than 0.01 percent in a determination of free calcium oxide and may be disregarded. In preparing the solvent, the end point may be observed better if the solvent is put in a tall cylinder placed on white paper. The solvent may be observed from the top and the colour matched with that of acidified solvent in a similar cylinder.

NOTE 2 — Glycerol and ethanol are highly hygroscopic. Every effort shall be made to avoid exposing them and other materials unnecessarily to moisture and carbon dioxide in air. Bottles with an outlet near the bottom or fitted with a siphon are convenient. Alternatively, an automatic pipette attached to storage vessel can be used. They can be filled to the top and protected with tubes containing soda lime. Condensers may also be fitted with such tubes at the top.

5.2.1.5 *Standard ammonium acetate solution* (1 ml = 0.005 g calcium oxide) — Prepare the solution by dissolving 16 g of crystalline ammonium acetate (*see* Note) in one litre of ethanol. Standardize this solution by titrating against pure calcium oxide that is freshly prepared by calcining pure calcium carbonate or calcium oxalate in a platinum crucible at 900 to 1 000°C to constant weight. When the calcined calcium oxide has cooled in a desiccator, perform the operations described under **5.2.1.6** in quick succession.

NOTE — Ammonium acetate is generally sold in a damp condition and should be as dry as possible. It may be dried by placing over a dehydrating agent, such as, sulphuric acid or fused anhydrous calcium chloride contained in a desiccator for two weeks or more.

5.2.1.6 Grind it in an agate mortar, weigh out 0.05 to 0.06 g into a dry 200 or 250 ml Erlenmeyer flask, and add to 60 ml of the glycerol ethanol solvent to the flask (*see* Note 1). In order to minimize the danger of hydration and carbonation, separate samples of pure calcium carbonate may be calcined in small sized platinum capsules. The calcium oxide thus produced is cooled in a desiccator, re-weighed for a check and introduced in the flask. The grinding of oxide may be unnecessary if original calcium carbonate is in a finely powdered form. Disperse the calcium oxide in the solution by shaking the flask and attach a reflux condenser (*see* Note 2). Boil the mixture. The ebullition should be positive but not so violent as to cause bumping or excessive evaporation. Then remove the condenser and immediately titrate the solution, while near boiling, with the standard ammonium

acetate solution. Replace the condenser and boil the solution again. Repeat the titrations at intervals (*see* Note 3). Shake the flask frequently between titrations to shorten the time required for the boiling. The titration is complete when no further colour appears in the solution during continuous boiling for one hour (*see* Note 4). Calculate the calcium oxide equivalent of the ammonium acetate solution in grams per millilitre by dividing the weight of calcium oxide used by the volume of solution required.

NOTE 1 — Sometimes pure calcium oxide will take on the bottom of the flask, particularly if the glycerol is anhydrous. This trouble may be avoided putting a few grams of clean, dry quartz sand in the flask before the introduction of calcium oxide and solvent.

NOTE 2 — If a new stopper is used and has white powder on it, it should be thoroughly washed before using. A glass tube about 6 mm in diameter and 50 to 100 cm in length may be used as an air-cooled condenser, but a short condenser is not recommended because a loss of alcohol is liable to occur through evaporation. In such a case it is necessary to add neutralized ethanol from time to time to maintain the same proportion of glycerol to ethanol. If the proportion is greater than one to three, a partial decomposition of cement may take place. Instead of an ordinary flask, plain tubing and a stopper, a flask with a ground-in glass stopper and condenser tube may be used, preferably one with standard taper joint. A water-cooled condenser is the most satisfactory and it may be as short as 30 cm. Some loss of alcohol may also occur through dripping from the condenser during the titrations and should be compensated for by the addition of neutralized ethanol.

NOTE 3 — In general, the intervals may be of 20 minutes but they will chiefly depend on the rapidity of the dissolution of calcium oxide. They may be of 5 to 10 minutes in the beginning and then be increased to 30 minutes or one hour in the end. If the solution contains a large amount of dissolved calcium oxide and the boiling is continued a long time without titration, crystals (probably of calcium glyceride) may form which dissolve slowly and increase the time required for the completion of the titration.

NOTE 4 — The end point will not be the same for a hot solution and a cold solution, especially when a large amount of calcium acetate is present. The titration should always be carried out while the solution is boiling hot. If the end point is determined accurately, the solution will turn pink upon cooling, and this can serve as evidence that the end point has not been greatly overstepped.

5.2.2 Procedure — Weigh one gram of finely ground clinker or Portland cement (*see* 5.2.2.1) into a flask, and add 60 ml of the solvent to the flask and proceed as in the standardization of the ammonium acetate solution (*see* 5.2.1.5). The end point is considered to have been reached when the content of free calcium oxide in the sample does not increase by more than 0.05 percent during the last two hours of boiling.

5.2.2.1 The method is designed especially for fresh clinker. As the method does not differentiate between free calcium oxide and free calcium hydroxide, any free calcium hydroxide that may be present will be included in the determination and calculated as free calcium oxide. The method may be applied to cement or aged clinker, if desired, but the possibility of the presence of free calcium hydroxide should be kept in mind.

The sample shall be fine enough to pass completely through 75-micron IS Sieve. If it is not, take about 1.2 g of the sample and sieve it through a small, clean, 75-micron IS Sieve. Grind the residue in an agate mortar until it is fine enough to pass through the sieve. Mix the ground residue with screened material thoroughly and grind the mixture rapidly in the agate mortar for two or three minutes. Weigh approximately about one gram of the ground mixture into the flask for the determination of free calcium oxide. Avoid unnecessary exposure of the sample to the atmosphere. If the sample is fine enough to pass completely through the sieve, grind slightly over one gram as above before weighing one gram for the determination.

5.2.2.2 No excess of ammonium acetate shall be added at any time because an excess of it may react with calcium aluminate and silicates. To avoid such excess, use a small burette or measuring pipette fitted with a glass stopcock or a rubber pinchcock and small tip which delivers about 50 drops per millilitre. If the titrations are far apart and the reagent evaporates in the tip, a few drops may be discarded before each titration and the drops used in titration may be counted and converted in terms of millilitres. It is a good precaution to allow a slight pink colour to remain in the solution after each of the early titrations.

Vigorous boiling is more essential with cement than with pure calcium oxide and, if possible, should be so conducted that it is not necessary to shake the flask vigorously at intervals. The flask should not be shaken vigorously while it is disconnected from the condenser because the solvent, if superheated, may boil, expel alcohol vapour and then draw in air carrying water and carbon dioxide. A good procedure is to bring the solution and sample to boiling rapidly over a Bunsen flame in the beginning before placing the flask on a hot plate and connecting it with the condenser. If there is a tendency to bump, it may be reduced by adding glass beads or pure quartz sand to the flask or putting a thin mat of asbestos between the flask and the hot plate.

If in the final titrations there is doubt as to the exact end point, add a drop of the phenolphthalein indicator to the quiescent liquid in the flask and carefully observe the point where the indicator strikes the liquid. If no colour appears, the end point has been reached. If the sample settles during boiling and leaves a partially clear layer of solution, another way to overcome difficulty in discerning a change in the colour of the solution is to swirl the flask after the addition of the solution of ammonium acetate so as to avoid the dispersion of the sample. A strong daylight lamp with a reflector may be used as an aid in the discernment of the end point. The contents of the flask may be matched with similar contents in another flask which contains an excess of ammonium acetate.

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If it is necessary to leave the determination incomplete, remove the flask, add just enough ammonium acetate to discharge the pink colour and stopper the flask tightly. When renewing the determination, boil the mixture before repeating the titrations.

5.2.3 Calculation — Calculate the percentage of free calcium oxide to the nearest 0.1 as follows:

$$\text{Free calcium oxide (CaO), percent} = EV \times 100$$

where

E = CaO equivalent of the ammonium acetate solution in grams per millilitre, and

V = millilitres of ammonium acetate solution required by the sample.

5.3 Ethanol-Glycerol Rapid Method

5.3.1 Reagents — The following reagents are required.

5.3.1.1 Phenolphthalein indicator — Prepare the indicator as described under **5.2.1.3**.

5.3.1.2 Glycerol-ethanol solvent — Prepare the solvent as described under **5.2.1.4**. Either anhydrous barium chloride or sodium chloride is used to accelerate the solution of free calcium oxide. If anhydrous barium chloride is used, dissolve it in the solvent on the basis of one gram per 60 ml of solvent before the solvent is neutralized (*see Note*). If sodium chloride is used, add 0.5 g of it to the flask that contains solvent and freshly prepared calcium oxide or sample at the beginning of the standardization or the determination, as the case may be.

NOTE — A convenient way to prepare a large amount of solvent is to heat barium chloride crystals at 120 to 130°C for several hours, dissolve them in hot glycerol (100 to 125°C) without grinding and mix the solution with ethanol. If the salt is dehydrated at a much higher temperature, it may be slow to dissolve in hot glycerol or solvent, even when ground.

5.3.1.3 Standard ammonium acetate solution (0.2 N) — Prepare and standardize a solution of ammonium acetate as described under **5.2.1.5**, using an accelerator as specified under **5.3.1.2**.

5.3.2 Procedure — Determine free calcium oxide in clinker or Portland cement as described under **5.2** using anhydrous barium chloride or sodium chloride as an accelerator (*see 5.3.1.2*). The end point is considered to have been reached when the content of free calcium oxide does not increase by more than 0.05 percent during the last hour of boiling (*see Note*).

NOTE — If the boiling is too prolonged, the sample may be partially decomposed and the end point may be obscured by coloured decomposition products.

5.3.3 Calculation — Calculate the percentage of free calcium oxide as described under **5.2.3**.

5.3.4 Result — This method has a tendency to give higher results than the method described under **5.2**. Result obtained by this method should be checked with the other method wherever possible.

5.4 Ethylene-Glycol Method

5.4.1 The ethylene-glycol method is to be used only for estimating free lime in fresh clinker and freshly ground cement but not with aged cement.

5.4.2 Reagents — The following reagents are required.

5.4.2.1 Ethylene glycol shall not contain more than one percent of water when determined as below:

Take 50 ml of ethylene glycol and 0.5 g of anhydrous copper in a small flask and shake at intervals for one hour at room temperature. A greenish-blue colour indicates the presence of water and the glycol cannot be used. If the colour is only faint green, it indicates that the water content is below one percent. If the water content is below one percent, it can be removed by evaporation by heating the glycol to 120°C for 1 to 2 hours.

5.4.2.2 Bromocresol green indicator — Bromocresol green is blue in alkaline solution and changes through green and yellowish green to yellow in acid solution. In aqueous solution, the various colours correspond to the following pH values:

	pH
Blue	5.2
Green	4.5
Yellow	3.8

5.4.2.3 Standard hydrochloric acid — 0.1 N.

5.4.3 Procedure

5.4.3.1 Take one gram of cement in a long-necked Pyrex flask of 100 ml capacity and add 50 ml of ethylene glycol. Close the flask with a cork stopper and heat the contents on a water bath for one-half hour to bring calcium oxide into solution. Keep the temperature of the bath between 60 to 70°C and shake the flask vigorously every five minutes to prevent the cement from forming lumps. Alternatively, very pure quartz sand initially washed and thoroughly dried is added to the flask to prevent the cement from baking. Moreover, the cement-ethylene-glycol mixture is put under the reflux condenser on a water bath at 60 to 70°C. In the event of stubborn cement necessitating a long time to react, the time can be cut-down by adding purest water-free methanol to the ethylene

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glacol (say 15 cc of former to 30 cc of latter) when on boiling for one-half to one hour under reflux, the solution is ready for titration.

5.4.3.2 Fit into a 5-cc Buchner funnel two filter papers. Carl Schleicher and Schull No. 597 'Selecta' or equivalent, moisten the paper with a little ethylene glycol and such tightly against the funnel. Filter off the solution from the flask quickly using suction, first pouring the clear solution and then the cement on the filter. Wash the cement on filter paper thrice with a 10 ml portion of glycol after filtration. Titrate the filtrate against 0.1 N hydrochloric acid using 3 drops of 1 percent bromocresol green in absolute alcohol as indicator (*see Note*).

NOTE — Instead of the Buchner funnel, it is possible to filter the solution through a Jena sintered glass filter No. 3G3. Sintered glass filter should be used only once and then washed with hydrochloric acid, followed by sodium carbonate solution and distilled water.

5.4.3.3 Conduct a blank determination with known quantities of calcium oxide by dissolving in glycol and titrating against 0.1 N hydrochloric acid as before. Prepare a graph showing the amount of 0.1 N hydrochloric acid required for titrating glycol having different quantities of calcium oxide.

5.4.4 Calculation — Calculate the percentage of calcium oxide present in the sample by comparing the quantity of 0.1 N hydrochloric acid required for titration with the graph and determining the equivalent calcium oxide.

5.5 Precaution

5.5.1 Wherever leaching or bleeding of ethanol glycerol or ethylene glycol is involved, avoid direct contact with flame. Heating over a bath or hot plate is recommended.

6. CHEMICAL ANALYSIS OF PORTLAND SLAG CEMENT

6.1 Reagents and Special Solutions — The following reagents and special solution of analytical reagent grades are required in addition to those specified in 4.1.

6.1.1 Dilute Solutions of Reagents — Prepare the following dilute solutions by diluting the reagent with distilled water.

6.1.1.1 Sodium nitrite — five percent (by weight).

6.1.1.2 Ammonium nitrite — two percent (by weight).

6.1.1.3 Phosphoric acid — 1 : 1 (by volume).

6.1.1.4 Ammonium nitrate wash solution — Hundred g of ammonium nitrate and two hundred g of ammonium hydroxide mixed and diluted to one litre with distilled water.

6.1.2 Standard Potassium Permanganate Solution — 0.03 N. Dissolve about 0.94 g of potassium permanganate (KMnO_4) in one litre of water. Allow it to stand for a week; filter through asbestos mat and keep in a dark place after standardizing it against pure sodium oxalate.

6.1.3 Standard Sodium Thiosulphate Solution — 0.03 N. Prepare a solution of sodium thiosulphate on the basis of 7.4 g of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) per litre.

6.1.4 Standard Potassium Iodate Solution — 0.03 N. Prepare a solution of potassium iodate and potassium iodide on the basis of 1.12 g of potassium iodate (KIO_3) and 12 g of potassium iodide (KI) per litre. Standardize the solution as in **6.1.4.1**.

6.1.4.1 To a cool solution of one gram of potassium iodide in 300 ml of water and 10 ml of hydrochloric acid in a 500-ml flask, add about 25 ml of the standard potassium permanganate solution (0.03 N). Swirl the solution gently, stopper the flask and let it stand for 5 minutes. Titrate the liberated iodine with the standard sodium thiosulphate solution until the colour nearly fades. Add 2 ml of the starch solution, continue the titration until the blue colour is destroyed, and back-titrate with the standard potassium permanganate solution (0.03 N) until the blue colour just reappears. Repeat the titration with the potassium iodate solution substitute for the potassium permanganate solution. Calculate the sulphur equivalent of the standard potassium iodate solution in grams per millilitre as follows:

$$E = \frac{A \times C \times G \times 0.02392}{B \times D \times F}$$

where

- E = sulphur equivalent of the potassium iodate solution g/ml,
- A = weight in g of sodium oxalate used in the standardization of the potassium permanganate solution,
- C = volume in ml of potassium permanganate solution used in the standardization of the potassium iodate solution,
- G = volume in ml of sodium thiosulphate solution required by F ,
- B = volume in ml of potassium permanganate solution required by A ,
- D = volume in ml of sodium thiosulphate solution required by C , and
- F = volume in ml of potassium iodate solution used in the standardization of the potassium iodate solution.

NOTE — One millilitre of a normal solution of potassium permanganate or potassium iodate is equivalent to 0.067 01 g of sodium oxalate or 0.016 03 g of sulphur. The number 0.239 2 is obtained by dividing 0.016 03 by 0.067 01. The potassium iodate

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and potassium permanganate solutions should be standardized frequently, but, as the sodium thiosulphate solution is more stable the potassium iodate solution may sometimes be standardized against the sodium thiosulphate solution alone and without the last values of *A*, *B*, *C* and *D* being changed.

6.1.5 Starch Solution — To 100 ml of boiling water, add a cool suspension of one g of soluble starch in 5 ml of water, and cool. And a cool solution of one g of sodium hydroxide in 10 ml of water, then 3 g of potassium iodide and mix thoroughly.

6.1.6 Ammoniacal Zinc Sulphate Solution — Dissolve 50 g of zinc sulphate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in 150 ml of water and 350 ml of ammonium hydroxide. Filter the solution after allowing it to stand for at least 24 hours.

6.1.7 Ammoniacal Cadmium Chloride Solution — Dissolve 15 g of cadmium chloride ($\text{CdCl}_2 \cdot \frac{1}{2}\text{H}_2\text{O}$) in 150 ml of water and 350 ml of ammonium hydroxide. Filter the solution after allowing it to stand for atleast 24 hours.

6.1.8 Standard Sodium Arsenite Solution — Dissolve 3.0 g of sodium carbonate (Na_2CO_3) and then 0.90 g of arsenic trioxide (As_2O_3) in 100 ml of water. Heat the mixture until the solution is as complete as possible. Filter the solution if it is not clear or it contains a residue. Cool it to room temperature, transfer to a volumetric flask and dilute it to one litre. Standardize the solution as given in **6.1.8.1**.

6.1.8.1 Dissolve 0.58 g of potassium permanganate (KMnO_4) in one litre of water and standardize it against 0.03 g of pure sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$). Put 30 ml of the potassium permanganate solution in a 250-ml Erlenmeyer flask. Add 60 ml of the dilute nitric acid (1 : 4) and 10 ml of a sodium nitrite solution (5 percent) to the flask. Boil the solution until the nitrous acid is completely expelled. Cool the solution, add sodium bismuthate and finish by titrating with standard solution of sodium arsenite in the manner described under **6.10** for the determination of manganic oxide.

NOTE — Each millilitre of the sodium arsenite solution is approximately equivalent to 0.000 3 g of manganic oxide.

6.1.8.2 Calculate the manganic oxide equivalent of the standard sodium arsenite solution in grams per millilitre as follows:

$$C = \frac{0.03 \times 0.236 \times 30}{a \times b}$$

where

C = manganic oxide equivalent of the sodium arsenite solution in g/ml,

- a* = volume in ml of potassium permanganate solution required by 0.03 g of sodium oxalate, and
- b* = volume in ml of sodium arsenite solution required by 30 ml of potassium permanganate solution.

6.2 Loss on Ignition — Loss on ignition shall be determined in accordance with requirements of 4.2. Owing to the presence of sulphide in slag cements which may be oxidised to sulphates, there may be a gain instead of loss in some cases and this should be reported.

6.3 Silica — The method for determining silica in Portland slag cement shall be the same as described in 4.3.

6.4 Combined Ferric Oxide and Alumina — The method for determining combined ferric oxide and alumina in Portland slag cement shall be the same as described in 4.4.

6.5 Ferric Oxide — The method for determining ferric oxide in Portland slag cement shall be the same as described in 4.5.

6.6 Alumina — The method for determining alumina in Portland slag cement shall be the same as described in 4.6.

6.7 Calcium Oxide

6.7.1 Method 1 (Gravimetric Method) — The method for determining calcium oxide shall be the same as described in 4.7.1.

6.7.2 Method 2 (EDTA Method) — Take 10 ml of solution prepared as given in 4.5.2.1 in a conical flask. Warm the solution on a hot plate and add drop by drop 1 : 1 ammonium hydroxide with constant shaking till a turbidity appears. Filter through Whatman filter paper No. 41 and wash the filter paper 2 to 3 times taking 5 to 10 ml to distilled water each time. Add 2 to 3 drops of nitric acid followed by about 50 mg of solid potassium periodate. Keep the flask on water bath till pink colour develops. Shake and allow to cool to room temperature. Add 5 ml of 1 : 1 glycerol with constant stirring and then 5 ml of diethylamine. Add 20 ml of 4 N sodium hydroxide solution and shake well to adjust the pH to 12 or slightly more. Add approximately 50 ml of distilled water and 100 mg of solid Patton — Reeders indicator and titrate against 0.01 M EDTA solution. The end point of titration is reached when one to two drops of EDTA produce a sharp change in colour from violet to blue. Calculate calcium oxide as given in 4.7.2.1.

6.8 Magnesia

6.8.1 Method 1 (Gravimetric Method) — The method for determining magnesia in Portland slag cement shall be the same as described in 4.8.1.

6.8.2 Method 2 (EDTA Method) — Take 10 ml of solution prepared as given in 4.5.2.1 in a conical flask. Warm the solution on a hot plate and add drop by drop 1 : 1 ammonium hydroxide with constant shaking till a turbidity appears. Add 2 ml in excess. Filter the solution through Whatman filter paper No. 41 in another conical flask. Wash the filter paper with hot water 2 to 3 times, taking 10 to 15 ml aliquot each time. Titrate against 0.01 M EDTA solution as in 4.8.2 and calculate magnesium oxide as given in 4.8.2.1.

6.9 Insoluble Residue — The method for determining insoluble residue in Portland slag cement shall be the same as described in 4.10.

6.10 Manganic Oxide

6.10.1 Take a sample of cement 1 to 3 g (see Note 1), weigh and put it into a 250-ml beaker and treat with 5 to 10 ml of water and then with 60 to 75 ml of diluted nitric acid (1 : 4). Boil the mixture until the solution is as complete as possible. Add 10 ml sodium nitrite solution (5 percent) and boil until the nitrous acid is completely expelled (see Note 2) taking care not to allow the volume of the solution to become so small as to cause the precipitation of gelatinous silica. Ignore any separated silica, but if there is still a red or brown residue, use more solution of sodium nitrite to effect a complete decomposition and then boil again to expel the nitrous acid. Filter the solution into a 250-ml Erlenmeyer flask and wash the filter paper with water. The solution should have a volume of 100 to 125 ml.

NOTE 1 — The amount of cement taken for analysis depends on the content of manganese, varying from one gram for about one percent of manganic oxide to 3 g for 0.25 percent or less of manganic oxide.

NOTE 2 — When sodium nitrite is added, the expulsion of nitrous acid by boiling should be complete. If any nitrous acid remains in the solution, it will react with the added sodium bismuthate and decrease its oxidizing value. If there is any manganese in the cement, the first small quantity of sodium bismuthate should bring out a purple colour.

6.10.2 Cool the solution to room temperature. Add a total of 0.5 g of sodium bismuthate in small quantities with intermittent shaking. Complete adding, and shake the solution occasionally for 5 minutes and then add 50 ml of cool dilute nitric acid (1 : 33) which has been previously boiled to expel nitrous acid. Filter the solution through a pad of ignited asbestos in a Gooch crucible or a carbon filter with the aid of suction. Wash the residue four times with cool dilute nitric acid (1 : 33).

6.10.3 Titrate the filtrate immediately with the standard sodium arsenite solution. The end point is reached when a yellow colour is obtained free of brown or purple tints and it does not change upon further addition of arsenic oxide. Calculate the manganese in the cement as manganic oxide.

6.10.4 If a determination of calcium or magnesium is required in addition to determination of manganese, the latter shall be eliminated from the combined filtrate obtained in 4.4.2 in the alumina and ferric oxide determination by the method laid down in 4.7.1.

6.11 Sulphur Trioxide

6.11.1 To one gram of the sample, add 25 ml of cold water, and while the mixture is stirred vigorously, add 5 ml hydrochloric acid. If necessary, heat the solution and grind the material with the flattened end of a glass rod until the decomposition of the cement is complete. Dilute the solution to 50 ml and digest for 15 minutes. Filter and wash the residue thoroughly with hot water. Set aside the filter paper with the residue. Dilute the filtrate to 250 ml and heat to boiling. Add slowly, drop by drop, 10 ml of hot barium chloride (100 g/l) solution and continue the boiling until the precipitate is well formed. Digest the solution on a steam-bath for 4 hours or preferably overnight. Filter the precipitate using Whatman No. 42 filter paper or its equivalent. Wash, place the paper and contents in a weighed platinum or porcelain crucible, and slowly incinerate the paper without inflaming. Then ignite at 800 to 900°C, cool in a desiccator and weigh the barium sulphate.

6.11.2 Calculate the percentage of sulphur trioxide to the nearest 0.1 as follows:

$$\text{Sulphur trioxide percent} = W \times 34.3$$

where

W = weight in g of barium sulphate, and

34.3 = molecular ratio of sulphur trioxide to barium sulphate
(0.343×100)

6.12 Sulphur as Sulphide

6.12.1 Gas Generating Flask — Connect a dry 500-ml boiling flask with a long-stem separating funnel and a small connecting bulb, by means of a rubber stopper. Bend the stem of the funnel so that it does not interfere with the connecting bulb, adjust the stem so that its lower end is close to the bottom of the flask, and connect the opening of the funnel with a source of compressed air. Connect the bulb with an L-shaped glass tube and a straight glass tube about 20 cm in length. Insert the straight glass tube in a tall-form, 400-ml beaker. A three-neck Woulfe bottle with a long glass tubing in the middle opening, placed between the source of compressed air and the funnel, is a convenient aid in the regulation of the air flow. If the air contains hydrogen sulphide or sulphur dioxide, a solution of lead acetate or some other suitable absorbent shall be used in the bottle. Rubber used in the apparatus shall be of pure gum grade low in sulphur and shall be cleaned with warm hydrochloric acid.

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6.12.2 Procedure — Place 15 ml of the ammoniacal zinc sulphate solution (*see* Note 1) and 285 ml of water in the beaker. Put 5 g of the sample (*see* Note 2) and 10 ml of water in the flask and shake the flask gently to wet and disperse the cement completely. This step and the following one should be performed rapidly to prevent the setting of the cement. Connect the flask with the funnel and bulb. Add 25 ml of the stannous chloride solution through the funnel and shake the flask. Add 100 ml of hydrochloric acid (1 : 3) through the funnel and shake the flask. During these shakings, keep the funnel closed and the delivery tube in the ammoniacal zinc sulphate solution. Connect the funnel with the source of compressed air, open the funnel, start a slow stream of air, and heat the flask and contents slowly to boiling. Continue the boiling gently for 5 or 6 minutes, cut off the heat, and continue the passage of air for 3 or 5 minutes. Disconnect the delivery tube and leave it in the solution for use as a stirrer. Cool the solution to 20 to 30°C (*see* Note 3), and 2 ml of the starch solution and 40 ml of hydrochloric acid (1 : 1), and titrate immediately with the standard potassium iodate solution until a persistent blue colour is obtained (*see* Note 4).

NOTE 1 — In general, zinc sulphate solution is preferable to cadmium chloride solution because zinc sulphate is more soluble in ammonium hydroxide than cadmium chloride. The cadmium chloride solution may be used when there is a doubt as to the presence of a trace of sulphur as the yellow cadmium sulphide facilitates the detection of a trace.

NOTE 2 — If sulphur content exceeds 0.20 or 0.25 percent, a smaller sample should be used so that the titration with the potassium iodate solution will not exceed 25 ml.

NOTE 3 — The cooling is important, as the end point is indistinct in a warm solution. A part of the ammonium hydroxide is lost during distillation and the remaining ammonium hydroxide reacts with the acid, raising the temperature of the solution a few degrees without rendering the end point uncertain.

NOTE 4 — If sulphur content is appreciable but not approximately known in advance, the result may be low due to the loss of hydrogen sulphide during a slow titration. In such a case the determination should be repeated with the titration carried out more rapidly.

6.12.3 Calculation — Calculate the percentage of sulphur as follows:

$$\text{Sulphur, percent} = EV \times 20$$

where

E = sulphur equivalent of the potassium iodate solution in g/ml,

V = volume in ml of potassium iodate solution required by the sample, and

20 = 100 divided by the weight of sample used (5 g).

NOTE — Sulphites, thiosulphates, and other compounds intermediate between sulphides and sulphates are assumed to be absent. If such compounds are present, they may cause an error in the determination.

7. CHEMICAL ANALYSIS OF PORTLAND POZZOLANA CEMENT

7.1 Loss on Ignition — The loss on ignition of Portland pozzolana cement shall be determined as specified in **4.2**.

7.2 Magnesia

7.2.1 Method 1 (Gravimetric Method) — The method of determining magnesia content in Portland pozzolana cement shall be the same as that described in **4.8.1** except as given in **7.2.1.1** to **7.2.1.3**.

7.2.1.1 Weigh out a quantity of the ignited sample equivalent to 0.5 g of the moisture-free sample, calculated as follows:

$$\text{Weight of ignited sample, g} = \frac{0.5(100.00 - \text{percentage loss on ignition})}{100}$$

NOTE — The ignited material from the loss on ignition determination may be used for the sample.

7.2.1.2 Thoroughly mix the sample with 4 to 6 g of sodium carbonate by grinding in an agate mortar. Place a thin layer of sodium carbonate on the bottom of a platinum crucible of 20 to 30 ml capacity, add the cement-sodium carbonate mixture and cover the mixture with a thin layer of sodium carbonate. Place the cover crucible over a moderately low flame and increase the flame gradually to a maximum (approximately 1 100°C) and maintain this temperature until the mass is quiescent (about 45 min). Remove the burner, lay aside the cover of the crucible, grasp the crucible with the tongs, and slowly rotate the crucible so that molten contents spread over the side and solidify as a thin shell on the interior. Set the crucible and cover aside to cool. Rinse off the outside of the crucible and place the crucible on its side in a 300-ml casserole about one-third full of water. Warm the casserole and stir until the cake in the crucible disintegrates and can be removed easily. By means of a glass rod, lift the crucible out of the liquid, rinsing it thoroughly with water. Rinse the cover and crucible with hydrochloric acid (1 : 3); then add the rinse to the casserole. Very slowly and cautiously, add 20 ml hydrochloric acid (sp gr 1.19) to the covered casserole. Remove the cover and rinse. If any gritty particles are present, the fusion is incomplete and the test should be repeated, using a new sample.

7.2.1.3 Evaporate the solution to dryness on a steam-bath. Remove silica, the ammonium hydroxide group and calcium in accordance with **4.3.1**, **4.3.2**, **4.3.3**, **4.3.4**, **4.3.5**, **4.4.1** and **4.7.1** respectively. Determine magnesium oxide in accordance with **4.8.1**.

7.2.2 Method 2 (EDTA Method) — Evaporate the solution under **7.2.1.2** to dryness on steam-bath. Remove silica as described in **4.3** and

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make up the volume of 250 ml in a volumetric flask. Determine magnesia as described in 4.8.2.

7.3 Sulphuric Anhydride — The method for determining sulphuric anhydride of Portland pozzolana cement shall be the same as described in 4.9, except that the sample used shall be moisture free and need not be completely decomposed.

7.4 Insoluble Residue — Insoluble residue of Portland pozzolana cement shall be determined as specified in 4.10.

8. POZZOLANICITY TEST FOR PORTLAND POZZOLANA CEMENT

8.1 Purpose — The pozzolanicity test for checking pozzolanic cements enables to observe the absence of hydrated lime in the cement pastes under consideration, owing to the equilibrium which is established between the lime and the alkalies present in the liquid phase in contact with the pastes themselves which are preserved for a given time at constant temperature.

NOTE — Under these conditions, the solutions which are in contact with the Portland cement pastes are generally oversaturated or at least saturated with lime. In case of cement pastes containing a suitable quantity of pozzolana the solutions in contact have a lime content (after a given period), the value of which is always less than that of the saturation, compared with the alkalinity of the solutions themselves. Obviously such a behaviour depends upon the absence in the pozzolanic pastes of hydrated lime, which is gradually fixed by the pozzolana.

8.2 Principle — Pozzolanic activity depends upon the fixation of $\text{Ca}(\text{OH})_2$, released by the hydration of the cement, in the form of more or less definite hydrates whose strength increases with time. In the test described, pozzolanicity is assessed by comparing the quantity of $\text{Ca}(\text{OH})_2$ present in a liquid phase in contact with the hydrated cement, with the quantity of $\text{Ca}(\text{OH})_2$ capable of saturating a medium of the same alkalinity. With a pozzolanic cement, the $\text{Ca}(\text{OH})_2$ in solution is always lower than the saturation concentration.

8.2.1 Experiments show that by using 20 g of cement per 100 ml of water at a temperature of 40°C, equilibrium is practically reached in seven days. The application of the test thus requires knowledge of the solubility at 40°C of $\text{Ca}(\text{OH})_2$ in a solution whose free alkalinity varies from zero to about 100 milli-equivalents of strong base (OH) per litre.

8.2.2 The pozzolanicity test thus offers the advantage of controlling the pozzolanic binder hydrated directly under conditions of use and represents a method of controlling the pozzolanic behaviour of a pozzolanic cement, that is the chemical and physical phenomenon which tend to practically completely remove the lime of hydrolysis from the binder.

8.3 Reagents

8.3.1 *Distilled Water*

8.3.2 *Standard Hydrochloric Acid* — 0.1 N.

8.3.3 *Ammonia Solution* — 0.5 N.

8.3.4 *Ammonium Oxalate* — in saturated solution.

8.3.5 *Methyl Orange*

8.3.6 *Potassium Permanganate Solution* — 0.05 N.

8.3.7 About 20 g paraffin wax (in case a glass flask is used).

8.4 Apparatus

8.4.1 *Conical Flask* — of 300 ml in alkali resisting glass or preferably in plastics material, with a stopper of rubber or waxed cork capable of being securely fitted to the latter so as to permit a vigorous shaking.

8.4.2 *Funnel* — with wide stem.

8.4.3 *Filter* — in sintered glass.

8.4.4 *Conical Flask* — of 250 ml with ground-glass stopper.

8.4.5 *Beaker* — of 250 ml.

8.4.6 *Precision Pipette* — of 50 and 100 ml.

8.4.7 *Thermostatic Space* — at $40 \pm 2^\circ\text{C}$.

8.5 Procedure

8.5.1 In case a conical glass flask is used, coat the inside of the flask with about 20 g of melted paraffin wax. Finally allow the surplus to solidify at the bottom of the flask as it stands on a level surface.

8.5.2 With a pipette, introduce 100 ml of distilled water in the flask and put the stoppered flask into a thermostat at 40°C until the required temperature is reached (about 1 hour).

8.5.3 Then pour into the conical flask, using the funnel with a wide stem, 20.00 ± 0.01 g of the cement to be tested. Close the container and secure the stopper. Shake vigorously for about 20 seconds in order to prevent the formation of cement lumps. Put the conical flask back into the thermostat at 40°C , quite level in order to ensure that the cement settles in a layer of even depth. All the operations outside the thermostat shall be carried out as quickly as possible, to avoid any appreciable lowering of the temperature of the contents of the flask.

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8.5.4 After storage for seven days at 40°C, quickly filter the liquid through sintered glass, collecting the filtrate in a conical flask with ground-glass stopper. Cool the filtrate to room temperature and mix.

8.5.5 Pipette out 50 ml of the filtrate solution into a 250-ml beaker and determine the total alkalinity against standard hydrochloric acid (0.1 N), using methyl orange as indicator. Express the result in milliequivalents (OH) per litre. Now precipitate the calcium as oxalate in ammonia solution, filter and wash with cold water. Titrate the calcium oxalate obtained, using 0.05 N potassium permanganate solution. Express the lime content in millimoles per litre.

8.6 Results — Express the total alkalinity and the calcium oxide content in millimoles per litre. Report on a pozzolanicity diagram* (see Fig. 1), the point representing the calcium oxide content as a function of the total alkalinity.

8.6.1 The cement under test is considered to be pozzolanic if the point representing it lies below the solubility isotherm.

8.7 Retests — If the point obtained is on the isotherm or in the immediate vicinity, re-start the test in the same conditions, but leaving the conical flask in the thermostatic space during fourteen days. In the case of slow but real pozzolanic activity, the test becomes clearly positive.

9. STAINING TEST FOR MASONRY CEMENT (DETERMINATION OF WATER-SOLUBLE ALKALI)

9.1 Procedure

9.1.1 To 150 g of sample of masonry cement in a 400-ml beaker, add 250 ml of distilled water. Stir thoroughly, let stand 30 min at room temperature, and stir again. Filter through a Buchner funnel, transfer the insoluble matter to the original beaker, and rinse the funnel with 150 ml of distilled water into the beaker containing the insoluble matter. Stir thoroughly, let stand 30 min at room temperature, and stir again. Filter as above and again return the insoluble matter to the original beaker. Wash the funnel with 100 ml of distilled water into the beaker containing the insoluble matter. Stir thoroughly, let stand 30 min at room temperature, and stir again. Filter as above. Acidify the filtrate with 5 ml of concentrated hydrochloric acid (HCl, sp gr 1 : 19). Stir until cloudiness disappears. Evaporate the solution to about 400 ml. Cool to room temperature and transfer quantitatively to a 500-ml volumetric flask. Dilute to 500 ml. Transfer a 50-ml aliquot of this solution to a 100-ml volumetric flask.

*The pozzolanicity diagram (Fig. 1) has been established in inclined coordinates, it may also be established in rectangular coordinates.

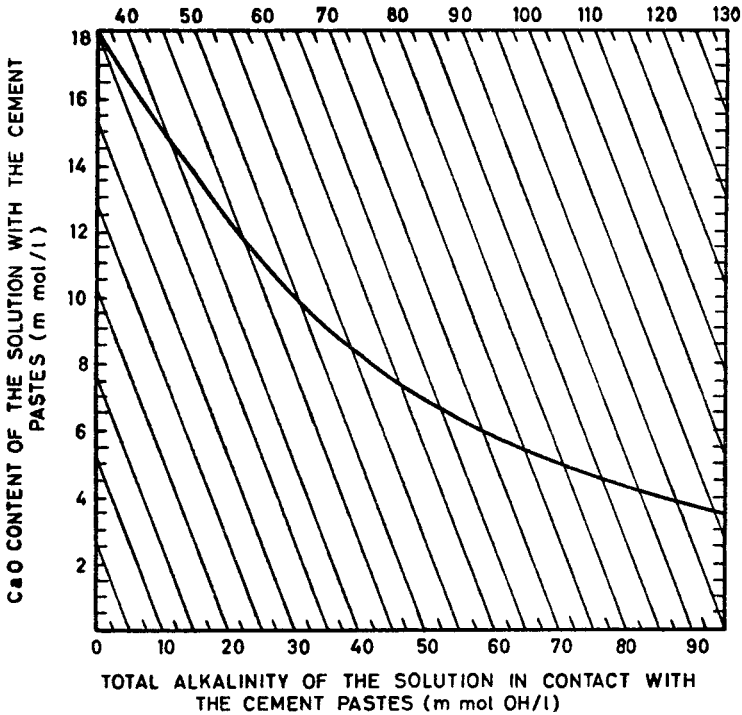


FIG. 1 POZZOLANICITY DIAGRAM — SOLUBILITY OF $\text{Ca}(\text{OH})_2$ IN THE PRESENCE OF ALKALIES AT 40°C

9.1.2 Determine sodium oxide and potassium oxide in the solution described in 9.1.1 following the procedure described in 4.11. Pour 9 ml of the stock calcium chloride (CaCl_2) solution 63 000 ppm (CaO) [see 4.11.1.2 (a)] into the 100-ml flask, and dilute the solution to 100 ml. Determine the solution and potassium oxide content of this solution as described in 4.11.4.2 and 4.11.4.3. Read the parts per million of each solution in the 100-ml flask from the calibration curve.

9.2 Calculation — Calculate the percentage of water-soluble alkali as sodium oxide (Na_2O) to the nearest 0.01 as follows:

$$A = B/1\ 500$$

$$C = D/1\ 500$$

$$E = C \times 0.658$$

$$\text{Total water-soluble alkali} = A + E$$

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where

A = percentage of water-soluble sodium oxide (Na_2O),

B = parts per million of Na_2O in the solution in the 100-ml flask
(see **9.1.1**),

C = percentage of water-soluble potassium oxide (K_2O),

D = parts per million of K_2O in the solution in the 100-ml flask
(see **9.1.1**), and

E = percentage of Na_2O equivalent to the K_2O .

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