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मानक

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IS 12813 (1989): Method of analysis of hydraulic cement by atomic absorption spectrophotometer [CED 2: Cement and Concrete]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHOD OF ANALYSIS OF HYDRAULIC
CEMENT BY ATOMIC ABSORPTION
SPECTROPHOTOMETER**

भारतीय मानक

परमाण्वीय अवशेषण स्पेक्ट्रो फोटोमापी द्वारा जलटूढ़ी सीमेंट की विश्लेषण पद्धतियाँ

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

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 21 September 1989, after the draft finalized by the Cement and Concrete Sectional Committee had been approved by the Civil Engineering Division Council.

With the advent of large scale cement plants and introduction of sophisticated technology for the manufacture of cement, it has become absolutely essential to have a precise control in quarrying, crushing, proportioning of raw materials for raw mix preparation and stable operation of the kiln to get desired quality of clinker. For this purpose, the analytical data of the chemical constituents is essential at more frequent intervals for necessary corrective actions to be taken. The conventional methods of chemical analysis, such as gravimetric and volumetric methods which are generally practised, though accurate and precise, are time consuming resulting in delay for necessary corrective actions. In addition to the conventional methods given in IS 4032 : 1985, the technique of atomic absorption spectrophotometric analysis may be used for routine quality control purposes. The advantages of atomic absorption technique over the conventional analytical methods are its rapidity, relative freedom from interferences (which affords high degree of selectivity) and high degree of sensitivity for over 60 elements. Application of such rapid analytical methods for analysis of major and minor constituents of cement for the routine control purposes will be immensely beneficial. With this in view, the Cement and Concrete Sectional Committee felt necessary to bring out a standard covering atomic absorption spectrophotometric methods for analysis of hydraulic cement. This standard lays down the procedure for conducting atomic absorption spectrophotometric analysis of major and minor constituents of different hydraulic cement. This method may be suitably used for analysis of clinker as well as raw materials and raw mix used in cement manufacture. In case of dispute or doubtful marginal values in estimation of elements covered in IS 4032 : 1985, the methods described in IS 4032 : 1985, shall be taken as referee method.

The composition of the technical committee responsible for the formulation of the standard is given at Annex A.

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

Indian Standard

METHOD OF ANALYSIS OF HYDRAULIC CEMENT BY ATOMIC ABSORPTION SPECTROPHOTOMETER

1 SCOPE

1.1 This standard covers the atomic absorption spectrophotometric procedure for chemical analysis of hydraulic cement and clinker.

1.2 This method covers the determination of SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , Na_2O , K_2O , Mn_2O_3 , TiO_2 and Cr_2O_3 .

NOTE—Compositions are expressed as percent oxide but determined as percent elements in the method.

2 REFERENCES

The Indian Standards given below are necessary adjuncts of this standard.

IS No.	Title
IS 264 : 1976	Specification for nitric acid (second revision)
IS 266 : 1977	Specification for sulphuric acid (second revision)
IS 1070 : 1977	Specification for water for general laboratory use (second revision)
IS 3535 : 1986	Methods of sampling hydraulic cement (first revision)
IS 4032 : 1985	Method of chemical analysis of hydraulic cement (first revision)

3 SAMPLING

3.1 The samples of the cement shall be taken according to the requirement of IS 3535 : 1986 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.

4 OUTLINE OF THE METHOD

4.1 In atomic absorption, a sample solution is aspirated into a flame through which radiation from a line emission source of the element sought passes. The monochromatic radiation is absorbed in proportion to the concentration of the neutral atoms present. The concentration of the analyte is obtained by comparison to calibration solutions.

The basis for using absorption to determine atomic concentration is as follows.

4.1.1 Assume a parallel beam of monochromatic radiant energy of intensity $I_{0\nu}$, at frequency ν is incident on an atomic vapour of path length, l . Then, if the intensity of the transmitted beam is I_ν , the absorption coefficient K_ν of the vapour at frequency ν is defined by:

$$I_\nu = I_{0\nu} \exp(-K_\nu l)$$

Since the absorption line has a finite half width K_ν will vary with ν . However, classical dispersion theory states that the integrated absorption ($K_\nu d\nu$) is given by the relation:

$$\int K_\nu d\nu = (\pi e^2/mc) N_\nu f$$

where

m = electronic mass,

c = velocity of light,

e = electronic charge,

N_ν = number of atoms per cubic centimetre capable of absorbing radiation of frequency ν , and

f = oscillator strength.

4.1.2 This equation is valid only when a transition is initiated from the ground state. N_ν is essentially equal to the total number of atoms per cubic centimetre (N_0). The integrated absorption is then proportional to the concentration of free atoms in the absorbing vapour and is independent of the temperature of the vapour. For most elements, N_ν can be replaced by N_0 which then brings about a simple linear relationship between the integrated absorption coefficient and the concentration. Since it is difficult to measure the integrated absorption coefficients, peak absorption is measured. In practice, the equation for I_ν given in 4.1.1 is valid only when calibration solutions are used.

4.1.3 Although the integrated absorption is proportional to the concentration of free atoms in the absorbing medium and is independent of the temperature of the vapour, the ability of a flame to produce ground state atoms is temperature dependent. The temperature of the flame must be high enough to dissociate the molecules, but not so hot as to produce large numbers of ions.

Elements that form refractory oxides require higher temperatures for their dissociation than such elements as sodium, calcium, copper, or zinc.

4.1.4 The monochromator in atomic absorption and flame emission is used to isolate the resonance line from nearby lines and back ground radiation. The band width is controlled by means of narrow, monochromator slits; however such slits cause reduction of desired radiation at the detector. Flame background radiation (radiation emitted from the flame or from elements therein) is also present and is relatively constant. The effect of this background radiation can be eliminated by modulating the incident, hollow cathode lamp radiation before it reaches the atomic vapours and tuning the amplifier to this modulation frequency. The continuous flame emission is rejected by the tuned amplifier.

4.1.5 The narrow widths of spectral lines cause some of the difficulties in the measurement of integrated absorption coefficients. Some factors affecting the shapes and widths of these lines are; Doppler broadening, pressure broadening, resonance broadening and self-absorption.

4.2 The sample is fused with Lithium metaborate in a crucible. The fused bead is dissolved in dilute nitric acid and the solution is diluted to that concentration which meets optimum requirements for atomic absorption analysis. The standard solutions are prepared from standard reference materials and, after necessary dilutions to the desired analytical range, their absorbance is measured and is plotted against respective concentration to get a calibration curve. The concentration of element in the unknown sample is determined from the absorbance by means of calibration curve.

5 SAFETY PRECAUTIONS

5.1 Operating personnel should adhere to the manufacturer's recommended practice for igniting and extinguishing the burner on the atomic absorption spectrophotometer to avoid an explosion which could cause physical injury.

6 REPRODUCIBILITY OF RESULTS

6.1 In all cases check determination (expressed in percent) shall be made and repeated, if satisfactory checks are not obtained. The difference between check determinations shall not exceed 0.2 for silica, alumina and calcium oxide; 0.1 for Fe_2O_3 and MgO ; 0.02 for Na_2O , K_2O , Cr_2O_3 and 0.05 for TiO_2 .

7 APPARATUS

7.1 Balance

Analytical balance with a precision of weighing accurately up to 0.1 mg.

7.2 Dilution Apparatus

Auto-dilution or precision micro-burette with a least count of 0.05 ml.

7.3 Sample Preparation Equipment

7.3.1 LPG burners capable of fusing sample up to 1 000°C.

7.3.2 Muffle Furnace

Muffle furnace capable of operating up to 1 200°C.

7.3.3 Crucibles

Crucibles made of 95 percent platinum and 5 percent gold or graphite crucibles of about 30 ml capacity.

7.3.4 Mini magnetic stirrer for the dissolution of fused bead.

7.4 Atomic Absorption Spectrophotometric System

7.4.1 Atomic absorption spectrophotometer consisting of atomizer, burner, suitable pressure regulating devices capable of maintaining constant oxidant and fuel pressure for the duration of test, hollow cathode lamp and/or electrode-less discharge lamp for the determination of elements as mentioned in 1.2, spectrometer, photomultiplier tube, detecting and measuring system, amplifying device and read-out system for indicating the absorbed radiation.

7.4.2 Acetylene gas cylinder fitted with two stage pressure reducing regulator.

7.4.3 Nitrous oxide gas cylinder fitted with two-stage pressure reducing regulator.

8 REAGENTS

8.1 Pure chemicals of analytical reagent grade and distilled water conforming to IS 1070 : 1977 shall be used in tests. The following reagents are generally used.

8.1.1 *Lithium Metaborate*, Pure grade anhydrous.

8.1.2 *Lanthanum Oxide*, 99.99 percent.

8.1.3 *Nitric Acid*, Specific gravity 1.42 and conforming to IS 264 : 1976.

8.1.4 *Sodium Chloride*

8.1.5 *Potassium Chloride*

8.1.6 *Manganese Metal*, 99.99 percent,

8.1.7 *Strontium Nitrate*, 99.99 percent.

8.1.8 *Potassium Titanium Oxalate*

8.1.9 *Ammonium Sulphate*

8.1.10 *Sulphuric Acid*, sp gr 1.84 (conforming to IS 266 : 1977).

8.1.11 *Potassium Chromate*

9 STANDARD REFERENCE MATERIAL

9.1 Standard cement samples of National Council for Cement and Building Materials or any other analyzed cement samples meeting the requirements of accuracy of analysis within the specified limits shall be used for calibration. Standard samples for calibration purposes shall be selected in such a way so as to cover the variations in concentrations of the individual constituents for specific materials.

10 PREPARATION OF STANDARD SOLUTIONS

10.1 Standard Solutions for Major Constituents

10.1.1 Prepare the standard solution to match closely the sample solution with respect to both matrix elements and acid content.

10.1.2 Weigh accurately about 0.25 g of standard sample and 1.0 g of lithium metaborate in a crucible. Mix thoroughly and fuse the mix over a burner and/or muffle furnace for about 10 minutes to get a clear melt. Quench the crucible in cold water and tap the bead into a 250 ml beaker containing 100-ml of 1:9 nitric acid. Stir the solution till the dissolution is complete. Transfer the solution to a 250-ml standard volumetric flask and make up the volume to the mark with distilled water. Label this solution as *A*. Prepare the standard solutions in the concentration ranges for each element as given in Table 1.

NOTE — Alternatively the standard solution can be prepared from the salts/metals of individual elements of analytical reagent grade quality.

10.2 Standard Solutions for Minor Constituents

10.2.1 *Alkali Solution*

Weigh 1.9070 g of KCl and 2.5425 g of NaCl and dissolve in distilled water conforming to IS 1070 : 1977. Make up the volume to one litre in a volumetric flask. Dilute 25 ml of the above prepared solution to 250 ml in a volumetric flask to get 100 ppm Na - K. Prepare standards solution of 1, 2, 3, 4 and 5 ppm. Na - K solution by taking 1, 2, 3, 4 and 5 ml from 100 ppm Na - K solution respectively into separate 100 ml volumetric flask. Add 10 ml of blank solution *B* as mentioned in 10.3 to each flask and make up the volume to the mark with distilled water.

10.2.2 *Manganese Solution*

Dissolve 1 g of manganese metal in 50 ml 1:3 nitric acid and make up the volume to one litre with distilled water in a volumetric flask. Dilute 10 ml of this solution to 100 ml in a volumetric flask to get 100 ppm manganese. Prepare standard solutions of 1, 2, 3, 4 and 5 ppm manganese solution by taking 1, 2, 3, 4 and 5 ml of 100 ppm manganese solution respectively into separate 100 ml volumetric flasks. Add 10 ml of blank solution '*B*' as mentioned in 10.3 to each flask and make up the volume to the mark with distilled water.

10.2.3 *Titanium Solution*

Boil a mixture of 7.3924 g potassium titanium oxalate [$K_2 TiO (C_2 O_4)_2 \cdot 2H_2O$], 1 g of ammonium sulphate and 25 ml of concentrated sulphuric acid for 15 minutes. Cool the solution and make up the volume to one litre in a volumetric flask with distilled water to get 1000 ppm titanium solution. Prepare 50, 100, 150, 200 and 250 ppm titanium solution by taking 5, 10, 15, 20 and 25 ml of 1000 ppm titanium solution respectively into separate 100 ml volumetric flasks. Add 10 ml of blank solution '*B*' as mentioned in 10.3 to each flask and make up the volume to the mark with distilled water.

10.2.4 *Chromium Solution*

Weigh 3.7351 g of potassium chromate ($K_2 Cr O_4$), dissolve in distilled water and make up the volume to one litre in a volumetric flask with distilled water to get a 1000 ppm chromium solution. Dilute 25 ml of this solution to 250 ml in a volumetric flask to get 100 ppm chromium solution. Prepare standard solutions of 1, 2, 3, 4 and 5 ppm chromium solution by taking 1, 2, 3, 4 and 5 ml of 100 ppm chromium solution respectively into separate 100 ml volumetric flasks. Add 10 ml of blank solution '*B*' as mentioned in 10.3 to each flask and make up the volume to the mark with distilled water.

10.3 Prepare blank solution by dissolving 1.0 g of lithium metaborate in 10 ml concentrated nitric acid and dilute with distilled water to 250 ml volumetric flask. Label this solution as *B*. Further dilutions shall be carried out as per requirement of individual constituents.

11 PREPARATION OF SAMPLE SOLUTION

11.1 Weigh accurately about 0.25 g of sample and fuse with 1.0 g of lithium metaborate and prepare the solution as indicated in 10.1.2. Label this solution as *C*. Further dilutions of sample solution *C* shall be carried out as given in Table 2.

Table 1 Typical Dilutions Required for Various Constituents Present in Standard Sample for Calibration

(Clause 10.1.2)

Sl No.	Oxide of Element	Approx Percent of Constituent Present	Ml of Solution		Make up to ml	Linearity Range in ppm Calculated on the Basis of Constituent
			'A'	'B' (see 10.3)		
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	SiO ₂	18.0	7.5	17.5	25	54 - 180
			15.0	10.0	25	
			25.0	0.0	25	
		35.0	7.5	17.5	25	105 - 350
			15.0	10.0	25	
			25.0	0.0	25	
ii)	Al ₂ O ₃	5.0	7.5	17.5	25	15 - 50
			15.0	10.0	25	
			25.0	0.0	25	
		12.0	4.0	8.0	25	19.2 - 57.6
			4.0	4.0	25	
			12.0	0.0	25	
iii)	Fe ₂ O ₃	4.0	2.5	5.0	100	1.0 - 3.0
			5.0	2.5	100	
			7.5	0.0	100	
		8.0	1.0	2.0	100	0.8 - 2.4
			2.0	1.0	100	
			3.0	0.0	100	
iv)	CaO (see Notes 2 to 5)	65.0	Ist dilution A ₁			
			5.0	—	100	—
			IIInd dilution			
			5.0 of A ₁	10.00	100	1.63 - 4.88
			10.0 of A ₁	5.00	100	
15.0 of A ₁	0.0	100				
v)	MgO (see Note 5)	4.0	2.5	5.0	100	1.0 - 3.0
			5.0	2.5	100	
			7.5	0.0	100	
		8.0	1.0	2.0	100	0.8 - 2.4
			2.0	1.0	100	
			3.0	0.0	100	

NOTES

1 The typical dilutions indicated in this table is with reference to approximate percentage of constituent present in standard samples in order to obtain the linearity range given in col 7 above. However, if the percentage of constituent in standard sample is different than that indicated, calibration may be done even with the same dilutions.

2 The precision of lime estimation can be further improved by choosing very close range standard samples for calibration.

3 The linearity range can be further extended by reducing the path by rotation of the burner or by using smaller path length burner.

4 Some of the commercially available instruments are having two channel system wherein by use of internal standard element which is not present in the sample, the absorbance ratios of lime with internal standard element are computed resulting in elimination of dilution errors.

5 In case of calcium and magnesium standards 0.1 percent lanthanum/strontium shall be added.

Table 2 Dilutions Required for Various Constituents Present in Unknown Sample for Analysis

(Clause 11.1)

Sl No.	Oxide of Element	Approx Percentage of Constituent	Sample Solution		Blank Solution	
			ml of Solution 'C'	Make up to ml	ml of Solution 'B'	Make up to ml
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	SiO ₂	18.0 - 35.0	15.0	25	15.0	25
ii)	Al ₂ O ₃	5.0 - 12.0	10.0	25	10.0	25
iii)	Fe ₂ O ₃	4.0 - 8.0	5.0	100	5.0	100
iv)	CaO	45.0 - 70.0	Ist Dilution B ₁			
			5.0	100		
			Ind Dilution			
			10.0 of B ₁	100	0.5	100
v)	MgO	4.0 - 8.0	2.5	100	2.5	100
vi)	Na ₂ O & K ₂ O	0.0 - 1.5	5.0	50	5.0	50
vii)	Mn ₂ O ₃	0.0 - 3.0	5.0	50	5.0	50
viii)	TiO ₂	0.1 - 1.0	5.0	50	5.0	50
ix)	Cr ₂ O ₃	0.0 - 0.2	5.0	50	5.0	50

NOTES

1 In case of calcium and magnesium estimation in the sample 0.1% lanthanum/strontium shall be added.

2 If the concentration range of the constituent element varies from that given in col 3 of the table, dilutions shall be adjusted so that it falls within the range of linearity.

3 In case concentrations of Mn₂O₃, TiO₂ and Cr₂O₃ are very low, dilution indicated in Table 2 may not be necessary or higher sample quantity may be taken for analysis. The addition of blank solution to standard shall be respectively matched.

12 ANALYSIS

12.1 The settings and adjustments of the instrument shall be carried out according to details of operation of particular spectrophotometer to achieve optimum performance as per the manufacturer's instructions. The standard conditions with working ranges of the elements and other parameters shall be adjusted as given in Table 3.

12.1.1 Calibration shall be carried out by aspirating the blank solution followed by standard solutions and recording the average absorbance reading for respective known concentration. After aspiration of each standard solution aspirate the blank solution. For a precise calibration curve five standard solutions covering the range shall be used. For routine analysis three standard solutions covering the range shall be used.

12.1.2 The sequence of operation of aspirating the solution for calibration purposes shall be carried out according to manufacturer's instruction. After the calibration with standard solutions aspirate the blank and sample solutions and record its absorbances/concentrations. Aspirate the blank solution to check that there is no drift in the blank/zero reading.

13 CALCULATIONS

$$13.1 \text{ Percent Oxide} = \frac{X \times Y \times DF \times Z \times 10^{-4}}{M}$$

where

X = $\mu\text{g/ml}$ of the element in the aspirated sample solution;

Y = volume of first sample dilution;

DF = dilution factor = $\frac{\text{Final volume of sample solution}}{\text{Aliquot of sample solution C taken for dilution and}}$

M = mass of sample taken for analysis in g;

Z = conversion factor from element to oxide as given below:

To Express	as	Factor, Z
Si	SiO ₂	2.139
Al	Al ₂ O ₃	1.889
Fe	Fe ₂ O ₃	1.430
Ca	CaO	1.399
Mg	MgO	1.658
Na	Na ₂ O	1.348
K	K ₂ O	1.205
Mn	Mn ₂ O ₃	1.437
Ti	TiO ₂	1.668
Cr	Cr ₂ O ₃	1.500

Table 3 Standard Conditions with Working Ranges for the Elements

(Clause 12.1)

Sl No.	Element	Wave-length	Flame Used	*Flame Condition	Interference Encountered	Recommended Steps to Avoid Interference	Working Range of Elements in ppm
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Si	251·6	N ₂ O/C ₂ H ₂	Fuel rich red feather cone	No	—	0 - 150
ii)	Al	309·3	N ₂ O/C ₂ H ₂	Fuel lean, oxidising, red feather cone	Ionisation	Li from the flux is sufficient to overcome the interference	0 - 30
iii)	Fe	248·3	Air/C ₂ H ₂	Fuel lean, oxidising, blue flame	Matrix and excess mineral acid	Samples and standard should be matched in matrix and acidity	0 - 3·0
iv)	Ca	422·7	Air/C ₂ H ₂ or N ₂ O/C ₂ H ₂	Fuel lean, oxidising, blue flame	Depression of signal due to formation of oxysalts with Al, Si, Ti, P	Addition of 0·1% La/Sr as releasing agent	0 - 5·0
v)	Mg	285·2	Air/C ₂ H ₂	Fuel lean, oxidising, blue flame	Depression of signal due to formation of oxysalts with Al, Si, Ti, P	Addition of 0·1% La/Sr as releasing agent	0 - 3·0
vi)	Na	589·0	Air/C ₂ H ₂	Fuel lean, oxidising, blue flame	Small ionization	Li from the flux is sufficient to overcome the interference	0 - 3·0
vii)	K	766·5	Air/C ₂ H ₂	Fuel lean, oxidising, blue flame	Small ionization	Li from the flux is sufficient to overcome the interference	0 - 3·0
viii)	Mn	279·5	Air/C ₂ H ₂	„	No	—	0 - 3·0
ix)	Ti	365·3	N ₂ O/C ₂ H ₂	Fuel rich red, reducing	—	Sensitivity is enhanced by high concentration of Fe and Al	0 - 200
x)	Cr	358	Air/C ₂ H ₂	Fuel rich yellow, reducing	Absorption is suppressed by iron and nickle	Interference can be lessened in a lean flame, 2 percent (m/V) NH ₄ Cl addition control the interference from iron	0 - 5·0

* Flame conditions are to be adjusted to get maximum sensitivity as given in the manufacturer's instructions.

ANNEX A

COMPOSITION OF THE TECHNICAL COMMITTEE

CEMENT AND CONCRETE SECTIONAL COMMITTEE, CED 2

<i>Chairman</i>	<i>Representing</i>
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SHRI P. N. MEHTA	Geological Survey of India, Calcutta
SHRI S. K. MATHUR (<i>Alternate</i>)	
SHRI NIRMAL SINGH	Development Commissioner for Cement Industry (Ministry of Industry), New Delhi
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The Institution of Engineers (India), Calcutta
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Joint Director (Civ Engg), BIS

Cement, Pozzolana and Cement Additives Subcommittee, CED 2 ; 1

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