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( Reaffirmed 1999 )

*Indian Standard*

# METHOD OF ANALYSIS OF HYDRAULIC CEMENT BY X-RAY FLUORESCENCE SPECTROMETER

भारतीय मानक

एक्सरे फ्लूरोसेंस स्पेक्ट्रोमापी द्वारा जलदूढी सीमेंट की विश्लेषण पद्धतियाँ

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BUREAU OF INDIAN STANDARDS
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## FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 29 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 steps 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 X-ray fluorescence (XRF) spectroscopy may be used for routine quality control purposes. The advantage of this technique is its rapidity of analysis and its suitability as 'on-line' as well as 'off-linc' system. Availability of quick data is extremely useful for corrrecting, proportioning and controlling the raw mix to ultimately achieve the desired quality of clinker and cement. With this in view, the Cement and Concrete Sectional Committee felt it necessary to bring out a standard covering the method for X-ray fluorescence spectrometric analysis of hydraulic cement. This standard lays down the procedure for conducting X-ray fluorescence spectroscopy of major and minor constituents of 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 refree 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 X-RAY FLUORESCENCE SPECTROMETER

## 1 SCOPE

**1.1** This standard covers X-ray fluorescence spectrometric procedure for chemical analysis of different hydraulic cements and clinkers.

**1.2** This standard covers the determination of $SiO_2$, $Al_2O_3$, $Fe_2O_3$, CaO, MgO, $SO_3$, $Na_2O$, $K_2O$, $Mn_2O_3$, $P_2O_5$, $TiO_2$, Cl and $Cr_2O_3$.

NOTE — This method determines the element concentration but the convention of expressing composition in terms of oxides is followed.

## 2 REFERENCES

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

| *IS No.* | *Title* |
|---|---|
| IS 3535 : 1986 | Methods of sampling hydraulic cement ( *first revision* ) |
| IS 4032 : 1985 | Method of chemical analysis of hydraulic cement ( *first revision* ) |

## 3 SAMPLING

The samples of cement shall be taken according to the requirements 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 and about 100 g of sample from this shall be taken for analysis.

## 4 OUTLINE OF THE METHOD

### 4.1 Principle

In this method the sample is irradiated by X-ray beam from an X-ray source. These X-rays are absorbed by the elements present in the sample which, in turn, emit X-rays called secondary or fluorescent X-rays. These X-rays are characteristic of the elements present in the sample in terms of their wavelength ( or energy ) by way of their origin, that is, transitions amongst various energy states. Their intensities are directly proportional to the concentration of emitting element in the sample. Using suitable X-ray wavelength dispersion and detection system, the intensities of various X-ray lines are measured and correlated to elemental concentration.

### 4.2 Experimental Procedure

Sample is converted into a suitable tablet form by using either a pressed pellet or fused bead technique. This sample is exposed to primary X-rays from the X-ray tube. The fluorescent X-rays emitted by the elements are analysed by using a set of collimators, dispersing crystals, detectors and intensity measuring system. The intensities of secondary X-rays are proportional to the concentration of the elements. A calibration is carried out using a set of suitable reference standards with varying ranges of oxide concentration. Concentration of the elements are determined from the calibration curves.

## 5 REPRODUCIBILITY OF RESULTS

In all cases, check determination ( expressed in percent ) shall be made and repeated if satisfactory checks are not obtained. The difference between the check determinations shall not exceed the limits given below for individual constituents:

| | |
|---|---|
| $SiO_2$ | ± 0·2 |
| $Al_2O_3$ | ± 0·1 |
| $Fe_2O_3$ | ± 0·1 |
| CaO | ± 0·2 |
| MgO | ± 0·2 |
| $SO_3$ | ± 0·1 |
| $Na_2O$ | ± 0·05 |
| $K_2O$ | ± 0·05 |
| $TiO_2$ | ± 0·03 |
| $P_2O_5$ | ± 0·05 |
| $Mn_2O_3$ | ± 0·05 |
| $Cr_2O_3$ | ± 0·005 |
| Cl | ± 0·005 |

## 6 APPARATUS

### 6.1 Balance

Analytical balance with a precision of weighing accurately up to 0·1 mg.

### 6.2 Sample Preparation Equipment

**6.2.1** *Pressed Pellet Equipment*

**6.2.1.1** *Grinding mill*

Grinding mill with suitable chamber for grinding

the sample with a time control.

**6.2.1.2** *Press*

Press for pellet preparation capable of giving controllable pressure up to 50 tonnes and producing pressed pellet suitable in size for X-ray analysis.

**6.2.1.3** Stainless steel discs/rings or disposable aluminium cups suitable for preparing the pressed pellet in required size for X-ray analysis.

**6.2.2** *Fusion Equipment*

**6.2.2.1** *Melting equipment*

It shall be capable of melting the sample with flux and attaining a minimum temperature of 1 200°C.

NOTE — various types of melting equipment are available commercially, such as resistance heating system, induction heating system and gas heating system.

**6.2.2.2** *Crucibles and casting dishes*

Crucible made of 95 percent platinum and 5 percent gold or graphite curcible or platinum rhodium crucible of suitable dimensions to produce a bead of required size for X-ray analysis.

NOTE — Graphite crucible, though reusable, have limited life compared to platinum-gold crucible or platinum-rhodium crucible.

**6.3 Muffle Furnace**

Furnace capable of continuous operation up to 1 200°C with an indicating pyrometer.

**6.4 X-ray Fluorescence Spectrometer** ( XRF )

**6.4.1** *Spectrometer*

Spectrometer with high voltage generator, X-ray tubes, dispersing crystals, collimators, detectors, measuring system with or without microprocessor/computer, printer, etc, suitable for determination of required elements.

**6.4.2** Chilled water supply system for cooling X-ray tube, suitable vacuum pump and air compressor as specified by the manufacturer.

**6.4.3** Gas cylinder fitted with two stage pressure regulator and containing argon with 10 percent methane gas or any other gas as specified by the manufacturer.

## 7 REAGENTS

Pure chemicals of analytical reagent grade shall be used in analysis. The following reagents are generally used:

a) Lithium metaborate

b) Dilithium tetraborate

c) Sodium tetraborate

d) Lithium fluoride

e) Lithium bromide

f) Sodium bromide

g) Potassium nitrate

h) Lanthanum oxide

j) Dilithium tetraboric acid

k) Disodium tetraboric acid

m) Stearic acid

n) Boric acid

p) Cellulose

q) Polyvinyl alcohol

NOTE — Sodium and potassium reagents shall not be used in estimation of alkalies.

## 8 STANDARD REFERENCE MATERIAL

Standard cement samples of National Council for Cement and Building Materials or any other analysed 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.

## 9 PROCEDURE

**9.1 Preparation of Sample**

**9.1.1** *Pressed Pellet Technique*

A sample of about 100 g is subjected to grinding, using a suitable grinding mill, for a pre-selected time determined by carrying out an experiment to finally yield sample with particle size less than 20 microns. Approximately 15-20 g of this ground sample is then filled in steel disc/ring or aluminium cup. The disc/ring or cup is then placed under the press for a specified time and pressure predetermined by carrying out preliminary experiments to produce stable pallets and to give reproducible XRF intensities for all elements. Particle size, pressure and time of application of pressure shall be kept the same for calibration and test samples. If required, a suitable binder in fixed proportion shall be added to calibration and test sample.

**9.1.2** *Fused Bead Technique*

For this technique, sample shall be taken as such. Determine the loss on ignition as described in IS 4032 : 1985.

**9.1.2.1** A predetermined quantity of sample on ignited basis and flux shall be mixed thoroughly in a crucible and fused to obtain a clear melt

expelling air bubbles, if any. Allow it to cool in the crucible or immediately transfer it to suitable mould made of 95 percent platinum and 5 percent gold, preheated to about 800°C. The temperature and time required for melting would vary depending on the flux material used and sample to flux ratio. These parameters for particular flux shall be predetermined to give a transparent homogeneous glass bead.

NOTES

1 Potassium/sodium nitrate may be incroporated along with the flux whenever an oxidizing atmosphere is required during fusion.

2 Lanthanum oxide acts as a glass forming oxide and may be used for avoiding the cracking of beads.

3 Addition of sodium bromide/lithium fluoride or lithium bromide as releasing agent is required in cases where problem of sticking of glass bead to platinum-gold crucible is faced.

### 9.2 Preparation of Standards for Calibration

Required number of samples shall be taken for calibration purpose. Prepare pellets or beads with these samples as given in **9.1.1** or **9.1.2.**

### 9.3 Measurement of XRF Intensities

#### 9.3.1 *Instrument Stabilization*

For acceptable accuracy of the results, it is necessary to keep the XRF instrument switched on for specified time as per manufacturer's instructions. The detector gas flow, spectrometer chamber temperature, room temperature and chilling water temperature shall be kept within the limits prescribed by the manufacturer.

#### 9.3.2 *Selection of Instrumental Parameters*

The instrumental parameters are to be selected for each element in accordance with the guidelines given by the manufacturer. For a sequential XRF system, operator should carry out the preliminary experiments to select and optimize the instrumental parameters given by the manufacturer.

#### 9.3.3 *Measurements for Calibration*

With the set of standard samples prepared as described in **9.1.1** or **9.1.2**, measure the XRF intensities for all the elements of interest by using the instrumental parameters selected as indicated in **9.3.2** and repeating the measurements 3 times.

#### 9.3.4 *Calibration*

From XRF counts per second ( CPS ) *versus* concentration data for each element, a linear calibration shall be obtained by plotting a graph having the following equation:

$$Y = mX + c$$

where,

$X$ = CPS for an element;

$Y$ = Concentration of element in the sample in percent;

$m$ = Slope of the calibration curve; and

$c$ = Intercept on Y-axis

**9.3.4.1** Alternatively, if XRF system is equipped with a computer, these calibration curves shall be obtained with the help of the computer. The coefficients $m$ and $c$ for each element shall be stored in the computer system for subsequent use while analyzing the samples.

**9.3.4.2** In the case of interference on XRF intensity of one element due to the others in the sample, the equation $Y = mX + c$ takes the form $Y = mX + c +$ terms involving interferences. These terms are calculated with the knowledge of interfering elements and their concentrations. Typical example of interferences for major constituents in cement and related materials are given below:

| *Element Analysed* | *Interfering Element* |
|---|---|
| Si | Mg, Al |
| Al | Mg, S |
| Fe | Ca, Si |
| Ca | K |
| Mg | Ca |

**9.3.4.3.** The interference coefficients are different for each X-ray analyser. In case of minor constituents, interferences are negligible.

#### 9.3.5 *Analysis*

Samples prepared according to **9.1.1** or **9.1.2** shall be subjected to XRF intensity measurements using the same instrumental parameters as for the standard samples. It is absolutely necessary to check the performance of the XRF system with the standard reference samples before taking up analysis. If there is any drift or change in CPS in standard reference sample for any element, calibration curve needs to be corrected. This shall be done by measuring CPS using standard reference samples.

Every day calibration shall be checked as indicated above, and in case of any deviation, recalibration shall be carried out. A standard cement sample shall be then analysed for confirming correctness of calibration.

## 10 CALCULATIONS

### 10.1 Pressed Pellet Technique

Report the concentration values obtained from graph or computer as such. Loss on ignition and insoluble residue, determined according to IS 4032 : 1985, shall be reported alongwith concentration.

**10.2 Fused Bead Technique**

Calculate the concentration values from those obtained on ignited basis and convert it to as received, basis. Loss on ignition and insoluble residue determined according to IS 4032 : 1985, shall be reported alongwith concentration.

## ANNEX A

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002
Telephones : 331 01 38, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

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|---|---|
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