IS : 3535 - 1986
(Reaffirmed 2004)

Indian Standard

METHODS OF SAMPLING HYDRAULIC CEMENTS

(First Revision)

Third Reprint DECEMBER 1998

UDC 666.942.31:620.113

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NEW DELHI 110002

Gr 4

August 1986

Indian Standard

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0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 24 January 1986, after the draft finalized by the Building Materials and Components Sampling Sectional Committee had been approved by the Civil Engineering Division Council.

0.2 With the considerable increase in production and consumption of cement which is of vital importance to the building industry, it is imperative that due consideration is given to sampling procedures which will help in the proper and objective evaluation of the various characteristics of cement.

0.2.1 Proper quality control during process of manufacture would also substantially reduce quality fluctuations of the material. The sampling procedures recommended in the standard, therefore, include the provisions for both process and lot inspection.

0.3 This standard was first issued in 1966. It is being revised so that various modifications necessary in the light of experience gained in its use could be incorporated.

0.4 In this revised version, inspection levels for routine control have been incorporated for those characteristics that have been included in various cement specifications from time to time by Cement and Concrete Sectional Committee, BDC 2. In order to provide a better quality assurance to the consumer, an upper limit to the quantity of material in a lot has been provided. The frequency of testing/inspection in process inspection for various characteristics has been modified so as to bring it in line with current practices in the cement industry. Keeping in view the practical difficulties in some cases in taking the requisite quantity of material in a single operation of the sampling equipment, provision for taking the material in two or three operations has been included. The quantity of material required for the laboratory sample has been increased so that sufficient material is available for carrying out various tests on laboratory samples and the composite sample. In order to obtain

the increased quantity of laboratory sample, the provision for increasing the weight of increment particularly for smaller sub-lots, has been included.

0.5 This standard covers the methods for sampling of hydraulic cements only. The methods for sampling of concrete and cement products will be covered in separate standards.

0.6 In reporting the results of a test or analysis, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods for sampling and the criteria for conformity of hydraulic cements from bags, bulk storage (silos), ship's hold, wagons and conveyors. Broad outlines with regard to the controls to be exercised during the manufacturing process have also been indicated.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Hydraulic Cement — Finely ground material which on addition of requisite quantity of water is capable of hardening both under water and in air by the chemical interaction of its constituents with water, and is also capable of binding together appropriate materials.

2.2 Lot — The quantity of cement from the same manufacturing unit and offered for inspection at one time, not exceeding 2 000 tonnes.

2.3 Sub-Lot — The quantity of cement in each of the parts into which a lot is divided for the purpose of sampling.

2.4 Increment — The quantity of cement taken at a time by the sampling implement.

2.5 Gross Sample — Sample as collected from a sub-lot, that is, the quantity of cement obtained by aggregating together all the increments from the same sub-lot.

2.6 Laboratory Sample — The quantity of cement obtained by reducing the gross sample by following a specified procedure for laboratory testing.

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

2.7 Composite Sample — The quantity of cement obtained by mixing together equal quantities of cement from each of the laboratory samples representing the sub-lots into which the lot has been divided.

3. GENERAL RULES

3.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed:

a) The sampling instrument shall be clean and dry when used.

b) Precautions shall be taken to protect the sample, the material being sampled, the sampling implement and the containers for samples from adventitious contamination.

c) The sample containers shall be of such a size that they are almost completely filled by the sample.

d) The sample containers shall be sealed air-tight after filling and marked with full particulars of the material and the date of sampling.

e) Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

f) The place of sampling shall be clean and dry and free from draught.

3.2 The interval of drawing the increments shall be, as far as possible, uniform in terms of mass throughout the whole quantity of the lot. However, if the rate of handling quantity is uniform, the interval of drawing the increments may be based on time unit in place of quantity unit.

4. PROCESS INSPECTION

4.1 The object of inspecting cement by the purchaser is to ensure its conformity to the specification requirements whereas inspection done by the manufacturer during production is to ensure uniformity and reduce quality fluctuations to minimum. For process control, the manufacturer shall take representative samples of the product at regular intervals to control the quality fluctuations. The inspection levels given in Table 1 are recommended for routine control over the manufacturing process.

4.2 Methods of Drawing Samples

4.2.1 *Clinker* — Every hour, a sample shall be drawn from each kiln immediately after it comes out of the cooling chamber. All the hourly samples drawn during a day shall be mixed together and shall constitute the composite sample for the day. After taking out a sufficient quantity of clinker from this composite sample for chemical analysis, the remaining clinker in the composite sample shall be mixed together and pulverized with a proper proportion of gypsum or both or the additives and tested for all other characteristics of the specification.

TABLE 1 RECOMMENDED INSPECTION LEVELS FOR ROUTINE CONTROL

(*Clause* 4.1)

Sl No.	Characteristics/ Tests	Frequency of Inspection/Tests		
		Clinker/Pozzolana	Cement Grinding	Cement Packing
(1)	(2)	(3)	(4)	(5)
i)	Chemical composition (complete analysis)	One composite sample every day for clinker and every week for pozzolana	One composite sample every week	One composite sample every week
ii)	Fineness	One composite sample every day (clinker taken hourly)	One sample every alternate hour and one composite sample every day	One composite sample every day
iii)	Setting time	do	a) One sample every hour b) One composite sample every day	do —
iv)	Soundness	do	do	One composite sample every day
v)	Compressive strength	do	do	do
vi)	Degree of whiteness (for white Portland cement only)	do	do	do
vii)	Heat of hydration (for low-heat Portland cement only)	do	One composite sample every week	One composite sample every week
viii)	Drying shrinkage (for Portland-pozzolana cement only)	—	do	do
ix)	Transverse strength (for Portland and Portland blast furnace cement only)	—	do	do
x)	Air content (for masonry cement only)	—	do	do
xi)	Water retention (for masonry cement only)	—	do	do
xii)	Hydrophobicity (for hydrophobic Portland cement only)	—	do	do

Note — For physical requirements under col 3, cement samples prepared in the laboratory ball mill by adding clinker with different suitable additives as per the relevant specification, shall be tested.

4.2.2 *Cement Grinding* — Every hour, a sample shall be drawn from the grinding mill and tested for setting time. For fineness, a sample taken every alternate hour shall be tested. The remaining portions of the hourly samples shall be mixed together to give the composite sample for the day. Similarly, a composite sample for a week shall be prepared. The frequency of testing for various characteristics shall be according to col 4 of Table 1.

4.2.3 *Cement Packing* — One composite sample every day from the packing machine shall be tested for fineness, setting time, soundness, compressive strength and degree of whiteness. The remaining portions of the daily samples, shall be mixed together to give the composite sample for the week which shall be tested for other characteristics of the specification.

4.2.4 For effective process control, the use of statistical quality control techniques is recommended and helpful guidance may be obtained in this respect from IS : 397 (Part 1)-1972*, IS : 397 (Part 2)-1975† and IS : 397 (Part 3)-1980‡.

4.2.4.1 The inspection data or the results of tests done at the place of manufacturer may be made available along with the materials supplied to enable the purchaser to judge the acceptability of the lot.

4.2.5 When such information cannot be made available or when the purchaser so desires, the procedure laid down in **5** shall be followed for judging conformity of the lot of hydraulic cements to the requirements of the relevant material specifications.

5. LOT INSPECTION

5.1 The samples shall be selected and examined for each lot separately for ascertaining their conformity to the requirements of the relevant specification.

5.1.1 For obtaining reliable conclusions, it is recommended that as far as possible, cement be sampled when it is in motion; that is, from conveyors or during loading or unloading.

5.2 Sampling from Conveyors

5.2.1 *Sub-Lots* — For the purpose of sampling, a lot while it is being discharged over a conveyor shall be divided into a number of sub-lots in accordance with Table 2.

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*Method for statistical quality control during production: Part 1 Control charts for variables (*first revision*).

†Method for statistical quality control during production: Part 2 Control charts for attributes and count of defects (*second revision*).

‡Method for statistical quality control during production: Part 3 Special control charts.

5.2.1.1 A representative gross sample shall be drawn from each of the sub-lots and shall be kept separately. Thus, there will be as many gross samples as the number of sub-lots into which the lot has been divided.

TABLE 2 NUMBER OF SUB-LOTS INTO WHICH A LOT IS TO BE DIVIDED

(*Clauses* 5.2.1, 5.3.1, 5.4.1, 5.5.2 *and* 5.6.1)

Weight of the Lot (in Tonnes)	No. of Sub-Lots
Up to 100	2
101 " 200	3
201 " 300	4
301 " 500	5
501 " 1 000	6
1 001 " 2 000	7

5.2.2 *Gross Sample* — The gross sample shall be collected by taking a number of increments at regular intervals such that one increment is taken for every 10 tonnes or part thereof from the material discharged. The increment shall preferably be taken from the full cross-section and thickness of the stream and in one operation. The best possible way of taking the increment is to collect the material in a receptacle which cuts across the entire stream at the time when the material is discharged from the conveyor. If it is not possible to take the increment at the point of discharge, it may be taken from the moving belt by means of a scoop which can sweep across the whole cross-section of the material. For this purpose, if practicable, the conveyor may be stopped while the increment is taken. The weight of increment shall not be less than 2 kg. It shall be ensured that the quantity of sample so collected is sufficient for the tests as per **5.7.**

5.2.3 As increments are taken from the sub-lot, they shall be placed directly in moisture-proof, air-tight containers to avoid moisture absorption and aeration of the sample. If the increments are placed in cans, the cans shall be completely filled and sealed immediately. Moisture-proof multiple wall paper bags or plastic bags may be used, if they are strong enough to avoid breakage and if they can be sealed immediately after filling in such a manner as to eliminate excess air in the bag and avoid moisture absorption and aeration of the sample. The containers shall carry suitable identification marks so that they can be related back to the particular sub-lot from which the gross sample has been taken.

5.3 Sampling from Bulk Storage (Silos)

5.3.1 *Sub-Lots* — For the purpose of sampling, the quantity of cement in the bulk storage shall be divided into a number of sub-lots depending upon the weight of the lot, in accordance with Table 2. The division into sub-lots shall be indicated by placing suitable markers on top of the cement.

5.3.2 *Gross Sample* — The gross sample shall be taken from a sub-lot by taking increments at regular intervals when the cement is being charged into the bulk storage or is being discharged from the bulk storage. If there is more than one opening for charging or discharging, the increments shall be taken from each opening. The number of increments shall be such that one increment is obtained for every 10 tonnes of the cement or part thereof. Each increment shall weigh at least 2 kg. It shall be ensured that the quantity of sample so collected is sufficient for tests as per **5.7**.

5.3.3 The increments taken from the sub-lot shall be stored in the manner described in **5.2.3**.

5.4 Sampling from Ship's Hold

5.4.1 *Sub-Lots* — For the purpose of sampling, the quantity of cement in the ship's hold shall be divided into a number of sub-lots depending upon the weight of the lot, in accordance with Table 2.

5.4.2 *Gross Sample* — When the depth of the cement to be sampled does not exceed 2 m, increments may be obtained by a slotted tube sampler as shown in Fig. 1. The slotted tube-sampler shall be between 1·5 to 1·8 m long and about 35 mm in outside diameter and shall consist of two polished brass telescopic tubes with registering slots which are opened or closed by rotation of the inner tube; the outer tube being provided with a sharp point to facilitate penetration. Where applicable, for depths of cement greater than 2 m, a sampling pipe activated by an air jet which is capable of removing cement from different depths may be used. The increments obtained by the slotted tube-sampler or sampling pipe shall be taken from well-distributed points and various depths of the cement in the sub-lot The number of increments shall be such that one increment is obtained for every 10 tonnes or part thereof from the cement. The increment shall weigh at least 2 kg. The material may be drawn in two or three operations. It shall be ensured that the quantity of sample so collected is sufficient for tests as per **5.7**.

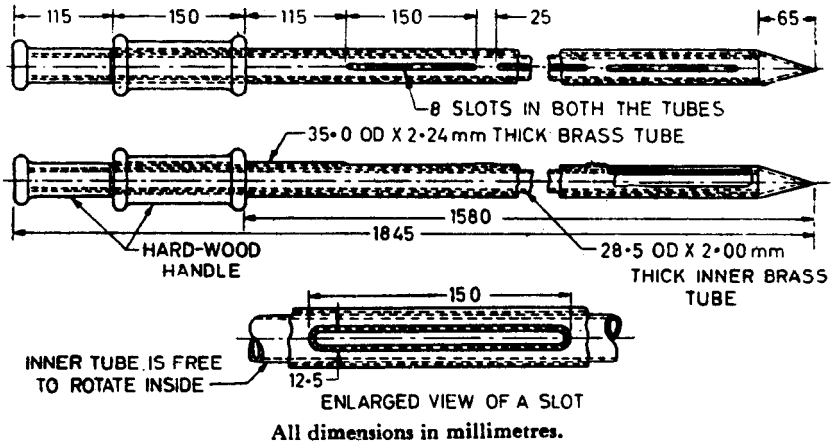
5.4.3 The increments taken from the sub-lot shall be stored in the manner described in **5.2.3**.

5.5 Sampling from Wagons

5.5.1 This method shall apply when loose cement or cement in bulk is received in wagons. If the cement is supplied in bags, **5.6** shall apply.

5.5.2 *Sub-Lots* — For the purpose of sampling, the quantity of cement in wagons shall be divided into a number of sub-lots depending upon the weight of the lot, in accordance with Table 2. The sub-lots shall consist of approximately equal number of wagons.

5.5.3 *Gross Sample* — The gross sample shall be taken from a sub-lot with the help of a slotted tube-sampler of the same design as shown in Fig. 1 but about 60 cm in length. The sampler shall be taken in the manner described in **5.4.2** from at least six evenly distributed points in each selected wagon.



All dimensions in millimetres.

FIG. 1 SLOTTED TUBE-SAMPLER FOR BULK CEMENT

5.5.3.1 From each selected wagon approximately equal number of increments shall be taken and weight of increment shall be not less than 2 kg. The material may be drawn in two or three operations. It shall be ensured that the quantity of sample so collected is sufficient for tests as per **5.7**.

5.5.4 The increments taken for the sub-lot shall be stored in the manner described in **5.2.3**.

5.6 Sampling from Bags

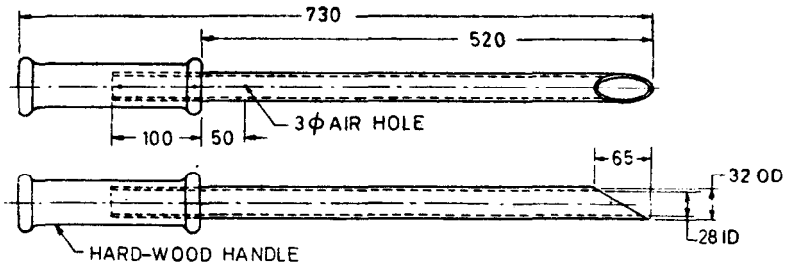
5.6.1 *Sub-Lots* — For the purpose of sampling, the quantity of cement in the lot shall be divided into a number of sub-lots, depending upon the weight of the lot, in accordance with Table 2. The sub-lots shall consist of approximately equal number of bags.

5.6.2 *Gross Sample* — For drawing representative sample from a sub-lot, at least 2 percent of the bags subject to a minimum of five shall be sampled. In case fractional numbers are obtained, the number of bags to be selected shall be taken to be equal to the next higher integer.

5.6.2.1 These bags shall be chosen at random from the sub-lot. To ensure the randomness of selection, a random number table as agreed to between the purchaser and the supplier shall be used (*see* IS : 4905-1968*). In case such a table as not available, the following procedure may be followed:

Starting from any bag at random, count the bags in the sub-lot in one order as 1, 2, 3, up to r and so on. Every rth bag so counted shall be removed, where $r = N/n$, N being the number of bags in the sub-lot and n the number of bags to be selected. In case the value of 'r' comes out to be a fractional number, its value shall be taken as equal to the integral part of it.

5.6.2.2 The sampling tube shown in Fig. 2 shall be inserted diagonally into the valve of the bag and the thumb placed over the air-hole and then shall be withdrawn. The material may be drawn in two or three operations.



All dimensions in millimetres.

FIG. 2 TUBE-SAMPLER FOR PACKAGED CEMENT VOLUME 300 cm³ APPROX

5.6.2.3 From each selected bag, approximately equal number of increments shall be taken and weight of each increment shall be not less than 2 kg. It shall be ensured that the quantity of sample so collected is sufficient for tests as per **5.7**.

5.6.3 The increments taken from the sub-lot shall be stored in the manner described in **5.2.3**.

5.7 Reduction of Gross Sample

5.7.1 Each gross sample shall be reduced separately. The material collected in the gross sample shall be thoroughly mixed, breaking the lumps and removing the foreign materials. It shall then be passed through 850-micron IS sieve. The foreign materials and hardened lumps that do not break on sieving or brushing, shall be discarded.

*Methods for random sampling.

5.7.2 *Coning and Quartering* — The material shall be scooped into a cone-shaped pile. Care shall be taken to drop each scoopful exactly over the same spot as otherwise the central axis of the cone will be slackened. After the cone is formed, it shall be flattened by pressing the top of the cone with the smooth surface of the scoop. Then the cone is cut into quarters by two lines which intersect at right angles at the centre of the cone. The reduction is achieved by rejecting any two diagonally opposite quarters.

5.7.2.1 Sample splitter or riffle samplers of the appropriate size may be used for reducing the gross sample mechanically.

5.7.3 The reduction of the sample in the manner described in **5.7.2** shall be continued till 11 kg of the material required for the laboratory sample is obtained.

5.7.4 Equal quantities of the material shall be taken from each of the laboratory samples representing the sub-lot into which the lot has been divided and mixed together to constitute a composite sample representing the lot as a whole. The weight of the composite sample shall be about 21 kg.

5.7.5 The laboratory sample and the composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample. The referee sample shall be used in case of a dispute between the purchaser and the supplier. Each of these parts shall then be placed in a moisture-proof air-tight container to avoid moisture absorption and aeration of the samples. They shall be labelled with full identification particulars such as supplier's name, the lot and the sub-lot number, the date of sampling, etc.

5.8 Number of Tests

5.8.1 The chemical and physical requirements for which individual laboratory samples shall be tested are:

a) Total sulphur content as sulphuric anhydride, and
b) Fineness.

5.8.2 A composite sample shall be tested for all the requirements.

5.9 Criteria for Conformity

5.9.1 For those characteristics where a composite sample has been tested for a lot, only one test result will be available and that result shall satisfy the requirements of the specification.

5.9.2 When two laboratory samples have been analyzed individually from a lot for any characteristic, the lot shall be considered as conforming to the requirement for that characteristic only if both of them pass.

5.9.3 When three or more laboratory samples have been analyzed individually from a lot for any characteristic, the following procedure shall be followed for judging the conformity to the requirements of that characteristic.

5.9.3.1 For different test results obtained by analyzing different laboratory samples, the average ($\bar{x}$) and the range (R) shall be calculated as follows:

$$\text{Average } (\bar{x}) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum values of the test results.

5.9.3.2 If the specification limit for the characteristic is given as a minimum, the value of the expression ($\bar{x} - 0{\cdot}5\,R$) shall be calculated from the relevant test results. If the value so obtained is greater than or equal to the minimum limit, the lot shall be declared as conforming to the requirement of that characteristic.

5.9.3.3 If the specification limit for the characteristic is given as a maximum, the value of the expression ($\bar{x} + 0{\cdot}5\,R$) shall be calculated from the relevant test results. If the value so obtained is less than or equal to the maximum limit, the lot shall be declared as conforming to the requirement of that characteristic.

5.9.3.4 If the characteristic has two-sided specification limits the value of the expression ($\bar{x} - 0{\cdot}5\,R$) and ($\bar{x} + 0{\cdot}5\,R$) shall be calculated from the relevant test results. If the values so obtained lie between the two specification limits, the lot shall be declared as conforming to the requirement of that characteristic.

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