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भारतीय मानक

सिलिका फ्यूम — विशिष्टि

Indian Standard

SILICA FUME — SPECIFICATION

ICS 91.100.15

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

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Price Group 4

Cement and Concrete Sectional Committee, CED 2

FOREWORD

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

Silica fume is very fine pozzolanic material composed of ultra fine, amorphous glassy sphere (average diameter, 0.10-0.15 μ m) of silicon dioxide (SiO_2) produced during the manufacture of silicon or ferro-silicon by electric arc furnaces at temperature of over 2 000°C. The micro silica is formed when SiO gas produced in the furnace mixes with oxygen, oxidizes to SiO_2 , condensing into the pure spherical particles of micro silica that form the major part of the smoke or fume from the furnace. These fumes are collected and bagged called silica fume. It is also known as condensed silica fume and micro silica. The Committee also felt that chloride content in silica fume shall be declared by the manufacturer so that different samples of silica fume can be compared and Engineer-in-Charge is in knowledge of the amount of chloride entering into concrete through silica fume.

This standard has been formulated to fulfil the need for a specification for this material, in view of its increasing use in the country.

In the preparation of this standard, due weightage has been given to the international coordination among the standards and practices in different countries in addition to relating it to the practices in the field in this country. For this, assistance has been derived from the following:

- a) ASTM C 1240-2000 'Standard specification for use of silica fume as a mineral admixture in hydraulic cement concrete, mortar and grout'; issued by the American Society for Testing and Material
- b) EN 197-1 : 2001 'Cement — Part 1 : Composition, specifications and conformity criteria for common cements'

The composition of the Committee responsible for formulation of the standard is given at Annex B.

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, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SILICA FUME — SPECIFICATION

1 SCOPE

This standard covers the chemical and physical requirements of silica fume for use in concrete and other systems containing hydraulic cement.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	Title
1727 : 1967	Methods of test for pozzolanic materials (<i>first revision</i>)
4082 : 1996	Recommendations on stacking and storage of construction materials and components at site (<i>second revision</i>)
4305 : 1967	Glossary of terms relating to pozzolana
6491 : 1972	Methods of sampling fly ash

3 TERMINOLOGY

3.0 For the purpose of this standard, the definitions given in IS 4305 and the following shall apply.

3.1 Silica Fume — Very fine pozzolanic material, composed mostly of amorphous silica produced by electric arc furnaces as a byproduct of the production of elemental silicon or ferro-silicon alloys.

3.2 Silica Fume in Natural State — Silica fume taken directly from the collection filter. The bulk density typically being in the range of 150-350 kg/m³.

3.3 Densified Silica Fume — Silica fume that has been treated to increase the bulk density by particle agglomeration. The bulk density typically being above 500 kg/m³.

3.4 Silica Fume Slurry — A homogenous, liquid suspension of silica fume particles in water, typically with a dry content of 50 percent by mass, corresponding to about 700 kg/m³ of silica fume.

4 CHEMICAL REQUIREMENTS

Silica fume shall conform to the chemical requirements given in Table 1.

Table 1 Chemical Requirements

(Clause 4)

Sl No. (1)	Characteristic (2)	Requirements (3)	Test Method (4)
i)	SiO ₂ , percent by mass, <i>Min</i>	85.0	IS 1727
ii)	Moisture content, percent by mass, <i>Max</i>	3.0	<i>see</i> Note 1
iii)	Loss on ignition, percent by mass, <i>Max</i>	4.0	IS 1727
iv)	Alkalies as Na ₂ O, percent, <i>Max</i>	1.5	<i>See</i> Notes 2 and 3

NOTES

1 For determination of moisture content, dry a weighed sample as received to constant mass in an oven at 105°C to 110°C. Express in percentage, the loss in mass and record as moisture content.

2 Requirement of limiting alkali shall be applicable in case silica fume is to be used in concrete containing reactive aggregate.

3 For determination of alkalies, method of test used for determination of this in cement may be adopted.

5 PHYSICAL REQUIREMENTS

Silica fume shall conform to the physical requirements given in Table 2.

6 SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Sampling

6.1.1 The methods and procedure of sampling of silica fume shall be same as the method given for fly ash in IS 6491. All samples whether grab or composite shall have a mass of at least 1 kg. Two grab/composite samples shall be taken from the lot for the first 100 t of silica fume. For each subsequent 100 t from the lot of silica fume, one sample shall be taken. However, not less than two samples shall be taken in any sampling programme.

6.1.2 The sample or samples for the purpose of testing may be taken by the purchaser or his representative or by any person appointed to supervise the work for the purpose of which the silica fume is required or by the latter's representative.

6.2 Criteria for Conformity

6.2.1 The samples of silica fume drawn in accordance with 6.1 and then prepared as per 7 and shall be tested as per 4 and 5.

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Table 2 Physical Requirements
(Clause 5)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Specific surface m ² /g, <i>Min</i> (see Note 1)	15	A	—
ii)	Oversize percent retained on 45 micron IS Sieve, <i>Max</i> (see Note 1)	10	—	1727
iii)	Oversize percent retained on 45 micron IS Sieve, variation from average percent, <i>Max</i> (see Notes 1 and 2)	5	—	1727
iv)	Compressive strength at 7 days as percent of control sample, <i>Min</i> (see Note 3)	85.0	—	1727

NOTES

1 Any one of the tests specified in (i) or (ii) and (iii) indicated may be adopted.

2 For (iii) the average shall consist of the ten preceding tests or all of the preceding tests if the number is less than ten.

3 In the test method for determination of compressive strength of silica fume cement mortar in accordance with IS 1727, the value of factor N may be taken as one.

6.2.1.1 Samples representing each 100 t of silica fume shall be tested for moisture content, loss on ignition and oversize.

6.2.1.2 Testing for all other physical and chemical requirements shall be carried out on composite samples representing not more than 400 t material each. The composite samples shall be prepared by combining portions equally from each of 100 t sample.

6.2.2 The lot shall be considered passing if samples meet in all the requirements. The silica fume may be rejected if it fails to meet any of the requirements of this standard. In case of dissatisfaction with the results of tests, the producer or supplier may request re-testing of the failed consignment.

7 SAMPLE PREPARATION

7.1 The grab or composite samples drawn in accordance with 6.1 shall be mixed thoroughly. A clean and dry laboratory concrete drum mixer provides adequate mixing for the purpose. The amount of silica fume shall be 10 to 50 percent of the volume capacity of the mixer. The mixing time shall be 5 ± 1 min. A polyethylene film shall be secured on the drum to keep the material in the drum during mixing of the sample lot.

7.2 A sampling device of appropriate size shall be used to take material from the thoroughly mixed sample for purpose of making the test specimen. At least six random sub-samples shall be taken to prepare the test specimen.

8 STORAGE AND INSPECTION

8.1 The silica fume shall be stored in such a manner so as to permit easy access for proper inspection and identification of each consignment.

8.2 Adequate facilities shall be provided to the purchaser for careful sampling and inspection, either at the source or at the site of work, as may be specified by the purchaser. For guidance on storage of silica fume at site, IS 4082 may be referred to. In general, the material shall be stored similar to cement/fly ash storage depending upon the storage requirement in bags/bulk form.

9 DELIVERY

The supply of silica fume shall be made in suitable quantities mutually agreed upon between the purchaser and the supplier. Where so required by the purchaser, the material shall be supplied in bags (jute laminated, multiply paper or polyethylene lines).

10 MANUFACTURER'S CERTIFICATE

The supplier/manufacturer shall satisfy himself that the silica fume conforms to the requirements of this standard and, if requested by the purchaser, shall furnish a certificate to this effect, indicating the results of the tests carried out on the samples of silica fume.

11 MARKING

11.1 Each bag/consignment of silica fume shall be clearly and permanently marked with the following informations :

- Identifications of the source of silica fume,
- Net mass of silica fume,
- Batch/Control unit number,
- Month and year of packing, and
- Any other identification mark as required by the purchaser.

11.2 BIS Certification Marking

The silica fume may also be marked with the Standard Mark.

11.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

12 HEALTH AND SAFETY

12.1 Owing to its fineness and its high silicon dioxide content, fears have been expressed over the use of the powder forms of micro silica. A large number of X-ray diffraction analysis suggest that it is amorphous material and should therefore be less dangerous than a crystalline material. However, it is advisable to take all necessary precautions while handling and using the

material. In many applications silica fume is handled as a aqueous slurry, reducing the dust problem virtually to zero in such cases.

12.2 Similar information shall be provided in the shipping invoices accompanying the shipment of bulk silica fume.

12.3 The silica fume consignment shall be in good condition at the time of inspection.

ANNEX A

(Table 2)

DETERMINATION OF SPECIFIC SURFACE AREA BY GAS ADSORPTION USING THE BET METHOD

A-1 SCOPE

This method of test specifies the determination of the total specific external and internal surface area of disperse or porous solids by measuring the amount of physically adsorbed gas according to the method of Brunauer, Emmett and Teller (BET method).

The BET method cannot reliably be applied to solids which absorb the measuring gas.

A-2 PRINCIPLE

The method specified involves the determination of the amount of adsorbate or adsorptive gas required to cover the external and the accessible internal pore surfaces of a solid with a complete monolayer of adsorbate. This monolayer capacity can be calculated from the adsorption isotherm using the BET equation. Nitrogen at its boiling point (about 77 K) is usually the most suitable adsorptive.

A-3 PROCEDURE

A-3.1 Sample Preparation

Prior to the determination of an adsorption isotherm, remove physically adsorbed material from the sample surface by degassing, while avoiding irreversible changes to the surface. As certain the maximum temperature at which the sample is not affected by thermogravimetric analysis or by trial experiments using different degassing conditions of time and temperature. When vacuumed conditions are used, degassing to a residual pressure of approximately 1 Pa or better is usually sufficient. Degassing of a sample can also be performed at elevated temperature by flushing with helium or with adsorptive. Degassing is

complete when a steady value of the residual gas pressure p , of its composition or the sample mass m is reached.

A-3.2 Methods of Measurements

Adsorption isotherms may be obtained by volumetric, gravimetric, calorimetric or spectroscopic measurements or by the carrier gas method using continuous or discontinuous operation. The procedure recommended by the manufacturer shall be followed.

The adsorptive gas is admitted to the sample container which is held at a constant temperature. The amounts adsorbed are measured in equilibrium with the adsorptive gas pressure p and plotted against relative pressure, p/p_0 , to give an adsorption isotherm.

A-4 EVALUATION OF ADSORPTION DATA

The amount of gas adsorbed n_a , preferably expressed in moles per gram, is plotted as ordinate against the respective pressure, p/p_0 as abscissa to give the adsorption isotherm. The monolayer capacity n_m is calculated using the BET equation.

$$\frac{p/p_0}{n_a [1 - (p/p_0)]} = \frac{1}{n_m C} + \frac{C-1}{n_m C} \cdot \frac{p}{p_0}$$

where

p = pressure of the adsorptive in equilibrium with the adsorbate, Pa;

p_0 = saturation vapour pressure of the adsorptive, Pa;

n_a = specific amount adsorbed, mol g⁻¹;

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n_m = specific monolayer capacity of adsorbate; amount of adsorbate needed to cover the surface with a complete monolayer of molecules; and

C = BET parameter.

A-5 TEST REPORT

The report of the determination shall include the following information :

- a) Laboratory, type of equipment, date of determination;
- b) Characterization of the sample, for example source, chemical composition, purity, method of sampling, sample division;
- c) Pre-treatment and degassing conditions, for example temperature, residual pressure, partial pressures, duration of degassing, flushing with adsorptive or helium, mass reduction;
- d) Mass of degassed sample, in g;
- e) Experimental procedure for adsorption isotherm determination, for example volumetric, gravimetric, chromatographic,

static or continuous gas admission, single-point determination, calibration of dead volume or buoyancy;

- f) Adsorptive (chemical nature, purity, moisture content);
- g) Adsorption isotherm (n_a , expressed, in mol g⁻¹, plotted against relative pressure, p/p_0), sample temperature in kelvin, saturation vapour pressure, expressed in Pa);
- h) Evaluation parameters: multipoint or single-point determination, BET plot or range of linearity, monolayer capacity n_m expressed in mol g⁻¹, C value, molecular cross-sectional area a_m expressed in square nanometers;
- j) Specific surface area, a_s , expressed in m²/g; and
- k) Reference material(s) used for validation of results.

A-6 USE OF REFERENCE MATERIAL

To ensure proper working conditions and correct data evaluation, the apparatus performance should be monitored periodically using a certified surface area reference material.

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

Cement and Concrete Sectional Committee, CED 2

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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AMENDMENT 1 APRIL 2017
TO
IS 15388 : 2003 SILICA FUME — SPECIFICATION

(*Second cover page, para 5*) — Substitute ‘Annex C’ for ‘Annex A’.

(*Page 1, clause 2*) — Insert the following entry at an appropriate place:

<i>IS No.</i>	<i>Title</i>
4032 : 1985	Method of chemical analysis of hydraulic cement

(*Page 1, Table 1, Note 3*) — Substitute the following for the existing note:

‘3 The alkalis shall be determined as per the procedure given in Annex A.’

(*Page 2, Table 2, row 1, col 4*) — Substitute ‘B’ for ‘A’.

(*Page 3, Annex A*) — Insert the following new annex and renumber the subsequent annexes:

ANNEX A
METHOD FOR DETERMINATION OF AVAILABLE ALKALIS
(*Table 1*)

A-1 PROCEDURE

A-1.1 Weigh 5.0 g of the sample and 2.0 g of hydrated lime on a piece of weighing paper, carefully mix using a metal spatula and transfer to a small plastic vial of approximately 25 ml capacity. Add 10.0 ml of water to this mixture; seal the vial by securing the cap or lid to the vial with tape (*see Note*), blend by shaking until the mixture is uniform, and store at $38 \pm 2^\circ\text{C}$.

NOTE — To ensure that moisture loss from the paste does not occur, place the sealed vial in a sealable container (such as a small sample or mason jar), add sufficient water to cover the bottom of container, and seal.

A-1.2 Open the vial at the age of 28 days and transfer the contents to a 250 ml casserole. Break up and grind the paste with a pestle, adding a small amount of water, if necessary, so that uniform slurry containing no lumps is obtained (*see Note*). Add sufficient water to make the total volume to 200 ml. Let it stand 1 h at room temperature and stir frequently. Filter through a medium-

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textured filter paper onto a 500 ml volumetric flask. Wash thoroughly with hot water (eight to ten times).

NOTE — At times it may be necessary to break the vial and peel off the plastic from the solid cake. In such cases, care should be exercised to avoid the loss of material and to remove all solid material from the fragments of the vial. If the cake is too hard to break up and grind in the casserole, a mortar should be used.

A-1.3 Neutralize the filtrate with dilute HCL (1+3), using 1 to 2 drops of phenolphthalein solution as the indicator. Add exactly 5 ml of dilute HCl (1+3) in excess. Cool the solution to room temperature and fill the flask to the mark with distilled water. Determine the amount of sodium and potassium oxide in the solution using the flame photometric procedure, described in IS 4032, except that the standard solution shall be made up to contain 8 ml of calcium chloride (CaCl₂) stock solution per litre of standard solution, and the solution as prepared shall be used in place of solution of cement.

NOTE — The standard solutions made up with 8 ml of calcium chloride (CaCl₂) stock solution contain the equivalent of 504 ppm of CaO. Tests have shown that this amount closely approximates the amount of calcium dissolved in the test solution.

A-2 CALCULATION AND REPORTING OF RESULTS

Calculate the results as percent by mass of the original sample material. Report as equivalent percentage of sodium oxide (Na₂O), calculated as follows:

$$\text{Equivalent Na}_2\text{O, percent} = \text{Na}_2\text{O, percent} + 0.658 \times \text{K}_2\text{O, percent}$$