भारतीय मानक

IS 18731 : 2024

Indian Standard

फ्यूमड सिलिका — विशिष्टि

Fumed Silica — Specification

ICS 71.060.50

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

May 2024 **Price Group 7**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Fumed silica is a versatile performance additive that is manufactured from chlorosilane compounds by vapour phase flame hydrolysis reaction at about 1 700 °C to 2 000 °C in an oxy-hydrogen flame. Fumed silica finds usage in wide variety of applications in many industries such as adhesives and sealants, composites, elastomers, gel-acid battery, inks and coatings, lubricants and greases, technical powders and agrochemicals, resins, toners, beauty and personal care, etc. Fumed silica provides rheology control and anti-settling, anti-sag, powder free flow, emulsion stability, oil absorption, moisture barrier, reinforcement, corrosion resistance, etc.

Fumed silica is hydrophilic in nature due to presence of surface hydroxyl groups. It has an amorphous structure based on X-ray diffraction pattern, which is caused by the rapid cooling of the molten silica particles to solid state during manufacturing of the product.

Fumed silica particles are non-porous and have moisture content typically less than 1 percent at the time of packaging. This moisture can be removed by heating above 100 °C. The tapped density is about 50 g/l at the time of packaging.

This standard does not cover use of fumed silica in food and pharmaceutical applications. Food grade fumed silica is covered under generic name ‘silicon dioxide, amorphous’ and approved as food additive INS 551 as an anticaking agent, in the *Food Safety and Standards* (*Food Products Standards and Food Additives*) *Regulations*, 2011. The monograph for INS 551 has been published under FAO JECFA monographs 17 (2015). Fumed silica is covered under the name colloidal silicon dioxide, anhydrous silicon dioxide and published as a monograph in the Indian pharmacopeia.

The composition of the Committee responsible for formulation of this standard is given in 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 or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. 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

FUMED SILICA — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and methods of test for hydrophilic, hydrophobic and dispersion products of fumed silica for general applications.

1.2 This standard does not cover fumed silica for use in food and pharmaceutical applications.

2 REFERENCES

The standards given below 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 revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 170 : 2020	Acetone — Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 10332 : 1982	Specification for hydrofluoric acid, aqueous

3 GRADES

The material shall be of the following four grades:

a) *Grade* 1— Hydrophilic fumed silica for general applications;

b) *Grade* 2 — Dimethyl dichlorosilane treated hydrophobic fumed silica;

c) *Grade* 3 — Polydimethyl siloxane treated hydrophobic fumed silica; and

d) *Grade* 4 — Fumed silica aqueous dispersion products.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of fine amorphous powder (hydrophilic and hydrophobic) and as dispersion in aqueous and solvent medium. Fumed silica is insoluble in water, organic solvents and diluted hydrochloric acid, while soluble in hydrofluoric acid and hot alkali solutions.

4.2 The material shall also conform to the requirements specified in Table 1 when tested in accordance with the methods prescribed in Annex A.

5 PACKING AND MARKING

5.1 Packaging

The material may be packed in 3 layer kraft paper bag with an outer LDPE liner or as agreed to between the purchaser and the supplier. Fumed silica aqueous dispersion product may be packed in HDPE drums or as agreed to between the purchaser and the supplier.

5.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

a) Name and grade of the material;

b) Name of the manufacturer and his recognized trade mark, if any;

c) Net mass;

d) Date of manufacture; and

e) Batch/lot number.

5.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

Table 1 Requirements for Fumed Silica

(*Clause* 4.2)

Sl No.	Characteristic	Requirements				Method of Test, Ref to
		Grade 1	Grade 2	Grade 3	Grade 4	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	*p*H (4 percent aqueous slurry)	3.4 to 4.3	–	–	–	A-1.1
ii)	*p*H (4 percent slurry in 80 : 20 IPA: Water), *Min*	–	4.0	–	–	A-1.2
iii)	*p*H (aqueous dispersion)	–	–	–	8.5 to 10.5	A-1.3
iv)	BET specific surface area, m^2/g	90 to 400#	(125 ± 20)	(115 ± 15)	–	A-2
v)	Loss on drying*, percent by mass, *Max*	1.0	1.0	1.0	–	A-3
vi)	Tamped density, g/l, *Max*	60	70	70	–	A-4
vii)	325 Mesh residue (44 micron), percent by mass, *Max*	0.02	0.02	0.02	–	A-5
viii)	Loss on ignition, percent by mass, *Max*	2.0	–	–	–	A-6
ix)	Carbon content, percent by mass	–	(0.85 ± 0.15)	(5.4 ± 0.6)	–	A-7
x)	Assay, percent by mass, *Min*	99.8	–	–	–	A-8
xi)	Viscosity, cPs, *Max*	–	–	–	250	A-9
xii)	Specific gravity	–	–	–	1.114 to 1.130	A-10
xiii)	Solid content, percent by mass	–	–	–	12 to 40	A-11

* at the time of packaging

Different grades of hydrophilic fumed silica available based on their BET specific surface area.

ANNEX A

(*Clause* 4.2)

METHODS OF TEST FOR FUMED SILICA

A-1 DETERMINATION OF *p*H

These test methods are used to determine the *p*H value of the fumed silica.

A-1.1 *p*H of 4 Percent Hydrophilic Fumed Silica Aqueous Slurry

A-1.1.1 *General*

This method measures the hydronium ion concentration of 4 percent aqueous slurry of the test material.

A-1.1.2 *Principle*

The hydronium ion (H_3O^+) concentration is measured by calibrating the chemical potential at a *p*H electrode using buffer solution of *p*H 7.0 and 4.0.

A-1.1.3 *Reagents*

A-1.1.3.1 *Buffer solution* — *p*H 4.0 and *p*H 7.0

A-1.1.3.2 *Distilled water* — (*see* IS 1070)

A-1.1.4 *Instruments*

A-1.1.4.1 *pH meter with glass electrode*

A-1.1.4.2 *Magnetic stirrer*

A-1.1.4.3 *Top pan precision balance* 0.01 g *accuracy*

A-1.1.4.4 *Magnetic stirring bar*

A-1.1.4.5 *PVC beaker* — 250 ml

A-1.1.4.6 *Measuring cylinder* — 100 ml

A-1.1.5 *Procedure*

Before doing the analysis, check whether the *p*H meter is calibrated. *p*H meter should be calibrated daily with buffer solution of *p*H 7.0 followed by *p*H 4.0.

Weigh (4.0 ± 0.01) g of the sample into a 250 ml beaker. Add 100 ml distilled water. Mix thoroughly for 5 min by inserting a magnetic stirring bar into the beaker and placing on to the magnetic stirrer. After 5 min, stop the magnetic stirrer. Immerse the *p*H glass electrode into the solution approximately half-an-inch from the bottom of the beaker. Read the *p*H value of the solution when the reading is stable. Rinse the electrode with distilled water, dry with a tissue paper and immerse electrode in storage solution.

A-1.2 *p*H of 4 Percent Hydrophobic Fumed Silica Slurry in 80 : 20 IPA/Water

A-1.2.1 *General*

This method measures the hydronium ion concentration of 4 percent slurry of the test material in 80 : 20 (*v/v*) IPA: Water mixture.

A-1.2.2 *Principle*

The hydronium ion (H_3O^+) concentration is measured by calibrating the chemical potential at a *p*H electrode using buffer solution of *p*H 7.0 and 4.0.

A-1.2.3 *Reagents*

A-1.2.3.1 *Buffer solution* — *p*H 4.0 and *p*H 7.0

A-1.2.3.2 80 : 20 IPA: *Water mixture*

A-1.2.3.3 *Hydrophobic fumed silica*

A-1.2.3.4 *Distilled water*

A-1.2.4 *Instruments*

A-1.2.4.1 *pH meter with glass electrode*

A-1.2.4.2 *Magnetic stirrer*

A-1.2.4.3 *Top pan precision balance* 0.01 g *accuracy*

A-1.2.4.4 *PVC beaker* — 250 ml

A-1.2.4.5 *Measuring cylinder* — 100 ml

A-1.2.4.6 *Magnetic stirring bar*

A-1.2.5 *Procedure*

Before doing the analysis, check whether the *p*H meter is calibrated. *p*H meter should be calibrated daily with buffer solution of *p*H 7.0 followed by *p*H 4.0.

Weigh (4.0 ± 0.01) g of the sample into a 250 ml beaker. Add 96 g 80 : 20 IPA: Water mixture. Mix thoroughly for 5 min by inserting a magnetic stirring bar into the beaker and placing on to the magnetic stirrer. After 5 min, stop the magnetic stirrer. Immerse the *p*H glass electrode into the solution approximately half-an-inch from the bottom of the beaker. Read the *p*H value of the solution when the reading is stable. Rinse the electrode with distilled water, dry with a tissue paper and immerse electrode in storage solution.

A-1.3 *p*H of Fumed Silica Aqueous Dispersion

A-1.3.1 *General*

This method measures the hydronium ion concentration of fumed silica dispersions.

A-1.3.2 *Principle*

The hydronium ion (H_3O^+) concentration is measured by calibrating the chemical potential at a *p*H electrode using buffer solution of *p*H 4.0, 7.0 and 11.0.

A-1.3.3 *Materials and Reagents*

A-1.3.3.1 *Fumed silica aqueous dispersion product*

A-1.3.3.2 *Buffer solution* — *p*H 7.0 and *p*H 11.0

A-1.3.3.3 *Distilled water*

A-1.3.4 *Instruments*

A-1.3.4.1 *pH meter with glass electrode*

A-1.3.4.2 *Top pan precision balance* — 0.01 g accuracy

A-1.3.4.3 *PVC beaker* — 250 ml

A-1.3.5 *Procedure*

Before doing the analysis, check whether the *p*H meter is calibrated. *p*H meter should be calibrated daily with buffer solution of *p*H 7.0 followed by *p*H 11.0.

Weigh 200 g of the sample into a 250 ml beaker. Immerse the *p*H glass electrode into the solution approximately half-an-inch from the bottom of the beaker. Read the *p*H value of the solution when the reading is stable. Rinse the electrode with distilled water, dry with a tissue paper and immerse electrode in storage solution.

A-2 BET SPECIFIC SURFACE AREA

A-2.1 General

This method describes the multi-point BET surface area measurement of fumed silica.

A-2.2 Principle

The surface area of a solid is measured using Brunauer, Emmett and Teller (BET) theory of multiplayer gas adsorption behavior. Solids adsorb nitrogen gas molecules, and under specific condition, the adsorbed molecules approach a monomolecular layer on the solid surface. The quantity of adsorbed gas in this hypothetical monomolecular layer is calculated using the BET equation. Combining this with the area occupied by the nitrogen molecule yields the total surface area of the solids.

A-2.3 Reagents

A-2.3.1 *Fumed Silica*

A-2.3.2 *Liquid Nitrogen*

A-2.3.3 *Nitrogen and Helium Gas with Purity Greater than* 99.9 *Percent*

A-2.4 Instruments

A-2.4.1 *Glass Sample Cell*

A-2.4.2 *BET Surface Area Instrument*

A-2.4.3 *Degassing Station*

A-2.4.4 *Vacuum Pump*

A-2.4.5 *Dewar Flask for Liquid Nitrogen*

A-2.4.6 *Analytical Balance* 0.000 1 g *Accuracy*

A-2.5 Procedure

Set the regulator pressure of the N_2 and He gas between 15 psi to 18 psi (Regulator pressure should not be more than 30 psi and cylinder pressure should not be less than 200 psi). Set the temperature of the degassing station at 200 °C and allow stabilizing prior to analysis. Switch on the analyzer, vacuum pump and PC. Place one dry and clean sample cell on the analyzer sample port and another to the reference port. Ensure liquid nitrogen has stabilized and is at appropriate level in the dewar. Measure and record the saturation pressure.

A-2.6 Sample Analysis

Record the sample cell weight (with stopper). Weigh 0.06 g to 0.16 g of silica sample into the sample cell. Record the combined weight of sample cell and sample weight (with stopper). Turn on the nitrogen gas flow to the gas injection needle and insert the needle into the sample. Place the sample cell (with stopper) in the heated side of the degassing station. After 10 min, relocate the sample cell to the cold side of the degassing station and allow cooling for 5 min. After cooling, remove the needle and turn off the gas flow. If necessary clean the neck of the sample cell. Record the combined weight of sample cell and sample (with stopper) after degassing. Place the sample cell in to the sample port of the analyzer. Open the sample information dialog box and fill the necessary information like sample ID, operator name, sample weight, degassing condition, analysis condition, adsorptive properties and report option. To start the analysis goes to unit and click start analysis. Choose the file created for the analysis and click ok. After completion of the analysis report will be displayed on the screen. Record the multi-point surface area and c-value.

A-3 LOSS ON DRYING

A-3.1 General

This method is used to determine the content of moisture and volatile materials in silica using controlled temperature drying.

A-3.2 Materials

A-3.2.1 *Fumed Silica*

A-3.3 Instruments and Apparatus

A-3.3.1 *Oven*

A-3.3.2 *Weighing bottles*

A-3.3.3 *Analytical balance*

A-3.3.4 *Desiccator*

A-3.3.5 *Spatula*

A-3.4 Procedure

Switch on the oven. Set the temperature to 105 °C by keep pressing ‘press to set’ button and adjust the temperature using ‘temperature set’ screw. After attaining the temperature, check the temperature of the oven and thermometer reading, which is kept in the oven.

Dry the empty weighing bottles in the drying oven at 105 °C for 15 min. Remove the weighing bottle from the drying oven and allow them to cool in a desiccator. Weigh the weighing bottle and lid together and record the weight. Transfer about 1g of silica sample to the weighing bottle. Close the lid and weigh.

Let the weight be W_1. Place the weighing bottle into the drying oven with lids partially open. Then allow for 2 h at 105 °C in the drying oven. After 2 h carefully close the lid and place the weighing bottle into the desiccator. Allow cooling to room temperature and note the weight of the weighing bottle. Let the weight be W_2.

A-3.5 Calculation

$$\text{Loss on drying, percent by mass} = \frac{(W_1 - W_2) \times 100}{Sample\ weight}$$

where

W_1 = sample weight with bottles; and

W_2 = sample weight with bottles after drying in oven at 105 °C.

A-4 TAPPED DENSITY

A-4.1 General

This method measures the tapped density of fumed silica. The tapped density is the mass of 100 ml of fumed silica after tapping in the tapping volumeter.

A-4.2 Principle

The tapped density is determined by measuring the volume occupied by a weighed amount of fumed silica after tapping the vessel. This causes settling and loose packing of the powder.

A-4.3 Materials

Fumed Silica

A-4.4 Instruments and Apparatus

A-4.4.1 *Tap Density Meter*

A-4.4.2 *Weighing Balance* 0.01 *g*

A-4.4.3 *Measuring Cylinder* — 100 ml

A-4.5 Procedure

Switch on the instrument. Fill the sample up to 100 ml in the measuring cylinder provided with the density meter and weighs the sample without any

disturbance and note down the weight. Now set the time and date in the density meter based on the display message. Select USP –1 method from the display. In this method, the stroke length is 14 mm ± 2 mm and nominal rate of drops is 300 drops/ minute. Select standard mode (As per USP standard) Enter the batch number. Enter the sample weight in grams. Enter the volume of sample in ml. Fix the measuring cylinder in its holder and mount the holder on the port marked USP – 1 (since USP – 1 is selected). Press start key, cylinder will be tapped for 500 times and actual counts will be displayed on the screen. After completion of 500 taps, the tapping will stop and an intermittent beep will indicate that the set tapping is over. Press Enter key to continue. Enter the tapped volume and press start key. Final tapping will be started and it will be tapped for another 750 times and display will indicate the actual number of counts. Enter the tapped volume and press enter key. The display shows the tap density results.

A-5 325 MESH RESIDUES

A-5.1 General

This method measures the amount of residue retained by a 325 mesh screen when the fumed silica slurry is passed through it. For hydrophilic fumed silica the slurry is prepared in water while for hydrophobic fumed silica the slurry is prepared in acetone.

A-5.2 Principle

Fumed silica hard residue greater than 44 microns in diameter is quantified by water wash rinsing the more easily dispersed particles through a 325 mesh screen. The amount of residue in 10 g of material is quantified by weighing the dried residue remaining on the screen after rinsing.

A-5.3 Materials and Reagents

A-5.3.1 *Fumed Silica*

A-5.3.2 *Acetone* — (*see* IS 170)

A-5.3.3 *DM Water*

A-5.4 Instruments and Apparatus

A-5.4.1 *Grit washer*

A-5.4.2 *Analytical Balance* 0.000 1 *g Accuracy*

A-5.4.3 *Top Pan Balance* 0.01 *g Accuracy*

A-5.4.4 *Screen made of Bronze or Stainless Steel* 325 *Mesh* (ASTM 45u)

A-5.4.5 *Clear Mesh Width*

A-5.4.6 *Hot Air Oven*

A-5.4.7 *Water Filter with* 25 micron *Filter Cartridge*

A-5.4.8 *Glass Beaker* — 1 000 ml

A-5.4.9 *Desiccators*

A-5.5 Procedure

Connect the ½’ water line hose from the water tap to the filter. Filter outlet is to be connected to grit washer unit through a pressure gauge. A separate by-pass line from the filter also to be provided for washing the grit washer sides.

Place a clean 1 000 ml beaker on a top loading balance and tare. Weigh 10 g of the sample into the beaker. For hydrophilic fumed silica add 300 ml of filtered water. In case of hydrophobic fumed silica add 300 ml acetone. Wash down sides of beaker as necessary. Stir for approximately 5 min on the magnetic stirrer. Remove beaker from stirrer and remove the stir bar from the beaker. Rinse with filtered water any remaining silica slurry on the stir bar into the beaker. Visually inspect the clean mesh to be used for damage. If damaged, replace mesh. Weigh a clean mesh and place in the mesh holder and carefully screw onto the apparatus. Start water spray and slowly pour the slurry into the grit washer. Wash the sides of the beaker with water and pour into the grit washer. Wash the sides of the grit washer using by-pass water line. After the slurry has passed through the mesh, maintain the water pressure at 1.1 bar for 3 min. Carefully remove the mesh from the holder and place on an aluminum weigh dish. Place in oven at 105 °C for 5 min. Remove the mesh and keep in the desiccators for cooling. Weigh the mesh and calculate residue as follows.

A-5.6 Calculation

325 Mesh residue, percent by mass

$$= \frac{\{(Weight\ of\ screen + Residue - Weight\ of\ screen)\} \times 100}{Sample\ weight}$$

A-6 LOSS ON IGNITION

A-6.1 General

This test measures the quantity of physically and chemically bound water.

A-6.2 Principle

Beside adsorbed water loss, the OH groups on the silica surface get condensed and removed as water.

A-6.3 Materials

A-6.3.1 *Fumed Silica*

A-6.4 Instruments and Apparatus

A-6.4.1 *Muffle furnace*

A-6.4.2 *Platinum Crucible*

A-6.4.3 *Platinum Tip Tong*

A-6.4.4 *Analytical Balance* (0.000 1 g *measurement accuracy*)

A-6.4.5 *Heat Resistant Gloves*

A-6.4.6 *Desiccator*

A-6.5 Procedure

Take about 0.5 g sample, obtained from the test for loss on drying, in a previously weighed platinum crucible. Let the weight of sample be W_1. Let the weight of crucible + sample be W_2. Switch on the muffle furnace and set the temperature to 1 000 °C. Place the sample in the muffle furnace and wait till muffle furnace temperature reach to 1 000 °C. Ignite the sample for 2 h at 1 000 °C, cool in desiccator. Weigh the platinum crucible. Let the weight be W_3.

A-6.6 Calculation

Loss on ignition, percent by mass = $\frac{(W_2 - W_3) \times 100}{W_1}$

where

W_2 = weight of crucible of with sample;

W_3 = weight of crucible with sample with after ignition of sample at 1 000 °C; and

W_1 = sample weight.

A-7 CARBON CONTENT

A-7.1 General

This method is used to determine the carbon content of the hydrophobic fumed silica.

A-7.2 Principle

Carbon content is determined by combusting the treated silica sample at high temperature in a pure oxygen environment. This causes carbon-bearing compounds to break down and release the carbon, which oxidizes to form CO_2. An infrared cell reads the concentration of CO_2 gas present. The instrument converts these values to a percentage value using software stored in the computer module of the instrument.

A-7.3 Materials

A-7.3.1 *Fumed Silica*

A-7.3.2 *Standard Carbon Sample*

A-7.4 Instruments and Apparatus

A-7.4.1 *Analytical Balance with* 0.000 1 g *Accuracy*

A-7.4.2 *KBr Press*

A-7.4.3 *Carbon Analyser*

A-7.4.4 *Boat*

A-7.4.5 *Spatula*

A-7.5 Sample Preparation

Prepare a pellet of silica sample using a KBr press.

A-7.6 Procedure

Check the oxygen gas cylinder pressure. If it more than 10 kg /cm^2, proceed to next step. Otherwise change the cylinder and proceed. Switch on the carbon analyzer and computer simultaneously. Switch on the fan, kept on the carbon analyzer vent. Open the C-144 file in the computer and click the box ‘diagnostics’ wait till the oven temperature to attain 1 100 °C. Click both ‘alarm relay’ and ‘pump/oxygen inlet’ and close the column. Check the oxygen flow in the purge flow and measure flow in the front side rotameter. Click the add sample column in the computer, enter 0.1 g for blank and click Analysis. Insert the empty sample boat into the carbon analyzer. System starts analyzing the carbon content in the sample boat and all carbon will be removed. Click the stop column, when the carbon is completely removed. This blank run should be carried out for sample boat based on number of samples. Now weigh around 0.1 gm of the standard carbon, such as 0.85 percent in the sample boat. Click the add sample, enter the standard sample weight and click analysis. System starts analyzing the sample and gives the carbon content. If the results is not matching with the standard carbon, select the standard analysis, click the calibrate column and press ok button followed by recalculate in the samples menu. Break the

palletized sample and weigh around 0.1 gm in the sample boat and do the analysis as done for standard. Record the result.

A-8 ASSAY

A-8.1 General

This method measures the purity of fumed silica.

A-8.2 Principle

Silicon content is determined on a previously ignited sample from the loss on ignition test. Sample is treated with sulphuric acid and hydrofluoric acid and ignited. Resultant silicon tetrafluoride is volatile and purity can be determined by measuring the loss in weight.

A-8.3 Materials and Reagents

A-8.3.1 *Fumed Silica*

A-8.3.2 *Sulphuric Acid* — (*see* IS 266)

A-8.3.3 *Hydrofluoric Acid* — 48 percent (*see* IS 10332)

A-8.4 Instruments and Apparatus

A-8.4.1 *Balance* (0.000 1 g *Measurement Accuracy*)

A-8.4.2 *Platinum Crucible*

A-8.4.3 *Platinum Tip Tong*

A-8.4.4 *Muffle Furnace*

A-8.4.5 *Electric Burner*

A-8.4.6 *Hot Plate*

A-8.4.7 *Desiccator*

A-8.5 Procedure

After completion of loss on ignition analysis, take the ignited sample for assay analysis. Weigh the platinum crucible with ignited sample. Let the weight be W_1. Add 3 to 4 drops of sulphuric acid and add enough alcohol to just moisten the sample. Add 15 ml of 48 percent hydrofluoric acid and evaporate on a hot plate to dryness in a well ventilated hood. Add 5 ml of hydrofluoric acid and ignite to dryness in an electric burner. Ignite the residue at 1 000 °C for 30 min in a muffle furnace. Cool in desiccator and weigh. Let the weight be W_2.

A-8.6 Calculation

Assay (as SiO_2), percent by mass

$$= \frac{(W_1 - W_2) \times 100}{Ignited\ sample\ weight}$$

where

W_1 = sample weight with crucible; and

W_2 = ignited residue weight with crucible 1 000 °C.

A-9 VISCOSITY

A-9.1 General

This method describes the viscosity measurement of fumed silica dispersions using a viscometer analyzer.

A-9.2 Principle

Viscosity of the dispersions measured using viscometer. The viscometer measures fluid viscosity at given shear rates. Viscosity is a measure of a fluid's resistance to flow. The viscometer rotates a sensing element in a fluid and measures the torque necessary to overcome the viscous resistance to the induced movement. This is accomplished by driving the immersed element, which is called a spindle, through a beryllium copper spring. The degree to which the spring is wound, indicated by the red pointer, is proportional to the viscosity of the fluid. The Viscometer is able to measure over a number of ranges since, for a given spring deflection, the actual viscosity is proportional to the spindle speed and is related to the spindle's size and shape. For a material of given viscosity, the resistance will be greater as the spindle size and/or rotational speed increase. The minimum viscosity range is obtained by using the largest spindle at the highest speed; the maximum range by using the smallest spindle at the slowest speed.

A-9.3 Materials

A-9.3.1 *Fumed Silica Dispersions*

A-9.4 Instruments and Apparatus

A-9.4.1 *Viscometer*

A-9.4.2 *Temperature Sensing Probe*

A-9.4.3 *Spindle Guard*

A-9.4.4 *Beaker* — 250 ml

A-9.5 Procedure

Power on in plug point. Check the bubble level which is available on top of the viscometer and ensure the viscometer level. Power on the instrument. (switch available at the back side of the right hand bottom portion of the stand) Press number '2' for standalone option. Press the 'motor on' key for auto zeroing and wait for two minutes to complete auto zero. After auto zero is complete press any one key to see the viscosity analysis programming page. Then set program by press 'select spindle' option and then press spindle number 61 and press 'enter' key to complete spindle selection, then directly press 60 as number of rotation (RPM) we wanted to run then press 'enter' key to complete (RPM) selection. Selected spindle needs to be attached with a dedicated shaft which is available at the bottom of the main unit by lift the shaft slightly and holding it firmly while screwing the spindle, then fix the spindle by screwing in a shaft with clockwise direction. Ensure spindle guard presence.

Place 250 ml of fumed silica dispersion sample under the main unit and immerse the spindle in the test material until the fluid's level reaches to the immersion groove in the spindle's shaft. With a disc type spindle, it is sometimes necessary to tilt the instrument slightly to avoid trapping air bubbles on its surface while immersing. Ensure the spindle position should be in the horizontally centre point to the sample container. After the spindle is placed at the right position press the 'motor on' key to start viscosity measurement. Use the 'select display' key to change screens for observing viscosity, torque, shear stress and shear rate. Observe all the readings for 2 min and then note the final viscosity reading which is shown on display. Change RPM if required to check viscosity of sample in different RPM and note reading after two minutes observation. Stop motor after completing the analysis. Then lift the spindle by rotating a wheel which is available at the back side of the stand. Remove the spindle by lifting the shaft up and rotate the thread in an anti-clockwise direction. Clean the spindle using tissue paper and store it in a spindle box. Switch off motor and power off the instrument.

A-10 SPECIFIC GRAVITY

A-10.1 General

This method is used to determine the specific gravity of the fumed silica dispersions.

A-10.2 Materials

Fumed silica aqueous dispersion

A-10.3 Instruments

A-10.3.1 *Specific Gravity Bottle* — 25 ml or 50 ml

A-10.3.2 *Analytical Balance*

A-10.3.3 *Hot Air Oven*

A-10.3.4 *Temperature Measuring Device*

A-10.4 Procedure

Weigh the mass of the clean and oven dried volumetric specific gravity bottle and record as W_1. Fill the bottle with deionised water to reference volume line and close the cap and dry the outer area of the flask by tissue, ensure deionised water filled in the flask is free from air bubbles and weigh and record the weight as W_2. Empty the volumetric specific gravity bottle and allow to oven drying at 105 °C for 15 min. Remove the bottle from the drying oven and allow them to cool in a desiccator. Weigh the volumetric bottle and lid together and record the weight as W_3. Fill the volumetric bottle with fumed silica dispersion sample to reference volume line and close the cap and dry the outer area of the flask by tissue, ensure sample filled in the flask is free from air bubbles and weigh and record the weight as W_4.

A-10.5 Calculation

$$\text{Specific gravity} = \frac{W_4 - W_3}{W_2 - W_1}$$

where

W_4 = weight of volumetric specific gravity bottle filled with sample;

W_3 = weight of empty volumetric specific gravity bottle weight after drying again;

W_2 = weight of deionised water filled volumetric bottle; and

W_1 = empty volumetric specific gravity bottle weight after drying.

A-11 SOLID CONTENT

A-11.1 General

This method is used to determine the content of silica in the silica slurry using controlled temperature drying.

A-11.2 Materials

A-11.2.1 *Fumed Silica dispersion*

A-11.3 Instruments and Apparatus

A-11.3.1 *Oven*

A-11.3.2 *Bottles*

A-11.3.3 *Analytical balance*

A-11.3.4 *Desiccator*

A-11.3.5 *Spatula*

A-11.4 Procedure

Switch on the oven. Set the temperature to 105 °C by keep pressing 'Press to set' button and adjust the temperature using 'temperature set' screw. After attaining the temperature, check the temperature of the oven and thermometer reading, which is kept in the oven.

Dry the empty weighing bottles in the drying oven at 105 °C for 15 min. Remove the weighing bottle from the drying oven and allow them to cool in a desiccator. Weigh the weighing bottle without lid and record the weight. Transfer about 5 g of silica dispersion to the weighing bottle. Close the lid and weigh. Let the weight be W_1. Place the weighing bottle into the drying oven. Then allow for 2 h at 105 °C in the drying oven. After 2 h remove weighing bottle from oven and carefully place into the desiccator. Allow cooling to room temperature and note the weight of the weighing bottle. Let the weight be W_2.

A-11.5 Calculation

Solid content, percent by mass

$$= \frac{100 - (W_1 - W_2) \times 100}{Sample\ weight}$$

where

W_1 = sample weight with bottles; and

W_2 = sample weight with bottles after drying in oven at 105 °C.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute, Bhavnagar	DR KANNAN SRINIVASAN **(*Chairperson*)**
Alkali Manufacturers Association of India, Delhi	SHRI K. SRINIVASAN SHRI H. S. DAS (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	DR A. V. R. REDDY DR S. N. ACHARY (*Alternate*)
Central Drugs Standard Control Organization, New Delhi	DR RAMAN MOHAN SINGH
Consumer Voice, Delhi	SHRI M. A. U. KHAN SHRI K. C. CHAUDHARY (*Alternate*)
Delhi Jal Board, New Delhi	SHRI ASHUTOSH KAUSHIK
Directorate General of Quality Assurance (DGQA), New Delhi	DR GURBACHAN SINGH SHRI B. S. TOMAR (*Alternate*)
Geological Survey of India, Kolkata	SHRI PVVR SARMA
Grasim Industries Ltd, Nagda	SHRI ALOK SINGH SHRI PANKAJ GUPTA (*Alternate*)
Global Adsorbents Pvt Ltd, Kolkata	SHRI SANJAY DHANUKA SHRI RAHUL DHANUKA (*Alternate*)
Gujarat Alkalies and Chemicals Ltd, Vadodara	SHRI V. K. MAHIDA SHRI SHAILESH PATEL (*Alternate*)
Hindustan Lever Ltd, Mumbai	MS VRINDA RAJWADE SHRI SOJAN VARGHESE (*Alternate*)
Hindalco, Mumbai	SHRI NAGESWAR KAPURI SHRI AJITH RAMACHANDRA (*Alternate*)
Indian Institute of Chemical Technology, Hyderabad	DR PRAVEEN R. LIKHAR DR RAJENDER REDDY (*Alternate*)
Indian Chemical Council (ICC), New Delhi	DR UMESH SHETKAR DR RAKESH KUMAR (*Alternate*)
Industrial Carbon Pvt Ltd, Ankleshwar	SHRI SATYAN ROHIT KUMAR
Ministry of Chemicals and Fertilizers, New Delhi	DR ROHIT MISRA DR O. P. SHARMA (*Alternate*)
Ministry of Defence (DGQA), Kanpur	SHRI R. N. APARAJIT

Organization	*Representative(s)*
National Chemical Laboratory, Pune	DR DARBHA SRINIVAS DR PARESH DHEPE (*Alternate*)
National Metallurgical Laboratory, Jamshedpur	DR TRILOCHAN MISHRA SHRI DEVBRATA MISHRA (*Alternate*)
National Mineral Development Corporation Ltd, Hyderabad	SHRI RAJAN KUMAR DR PRASHANT SHARMA (*Alternate*)
National Physical Laboratory, New Delhi	DR NAHAR SINGH DR S. P. SINGH (*Alternate*)
National Test House, Ghaziabad	MS RICHA KUNDRA
National Peroxide Ltd, Mumbai	DR JOY ANTHONY
Office of the Development Commissioner (MSME), New Delhi	DR KARTHIKEYAN MS ANNA BACKIAM (*Alternate*)
Shriram Institute for Industrial Research, Delhi	DR LAXMI RAWAT SHRI B. GOVINDAN (*Alternate*)
Tamilnadu Petroproducts Limited, Chennai	SHRI RAVI MUTHUKRISHNAN
Tata Chemicals Ltd, Mithapur	SHRI NAJMUL HASAN KHAN
The Dharamsi Morarji Chemicals Co Ltd, Mumbai	SHRI MANDAR GAIKWAD
Vaibhav Analytical Services, Ahmedabad	SHRI GAURANG OZA
In Personal Capacity (*Hari Nagar Co-Op-Society, Gotri Road, Vadodara - 390007*)	SHRI R. S. BAGHEL
In Personal Capacity (*514 Veer Apt, Sector 13, Rohini, New Delhi - 110085*)	SHRI D. K. JAIN
BIS Directorate General	SHRI A. K. LAL, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (CHEMICAL) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI SAGAR SINGH
SCIENTIST 'D'/JOINT DIRECTOR
(CHEMICAL), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

Review of Indian Standards

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 website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CHD 01 (24464).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Manakalya, 4th Floor, NTH Complex (W Sector), F-10, MIDC, Andheri (East), Mumbai 400093	283 25838

Branches : AHMEDABAD, BENGALURU, BHOPAL, BHUBANESHWAR, CHANDIGARH, CHENNAI, COIMBATORE, DEHRADUN, DELHI, FARIDABAD, GHAZIABAD, GUWAHATI, HARYNA, HUBLI, HYDERABAD, JAIPUR, JAMMU & KASHMIR, JAMSHEDPUR, KOCHI, KOLKATA, LUCKNOW, MADURAI, MUMBAI, NAGPUR, NOIDA, PARWANOO, PATNA, PUNE, RAIPUR, RAJKOT, SURAT, VIJAYAWADA.

Published by BIS, New Delhi