
निस्तापित एल्युमिना — विशिष्टि

Calcined Alumina — Specification

ICS 71.060

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FOREWORD

Calcined alumina is aluminium oxide that has been heated at temperature in excess of 1 050°C to drive off nearly all chemically combined water. In this form, alumina has higher chemical purity, extreme hardness (9 on the Mohs hardness scale, on which diamond is 10), high density and a high melting point (slightly above 2 050°C). However by controlling the calcinations temperature and time, properties of alumina can be controlled. With increasing the temperature during calcinations, alumina transforms to α -alumina which is chemically stable with high melting temperature.

After calcinations, alumina possesses good thermal conductivity, heat and shock resistance and high electrical resistivity at elevated temperatures. This combination of properties makes calcined alumina useful in abrasives, glass, porcelains, spark plugs and electrical insulators but the greatest quantity of calcined alumina is used to produce aluminium.

Commercially available alumina or aluminium oxide is generally known as calcined alumina. On the basis of heat treatment, crystal size, soda content and degree of thermal conversion to alpha phase, calcined alumina is available in number of grades. One of the major factor that decides the final use of alumina is its soda content.

Materials having low soda content are used for electronic applications and refractories and the one having medium level of soda content is used for electrical insulation and porcelains. Further, material with high level of soda content is used for glass, glaze, fibre glass and electrical porcelain.

Calcined alumina is obtained by calcining alumina powder at 1 200-1 300°C in order to get pure Al_2O_3 . As the temperature is increased, alumina transforms into α -alumina which is chemically stable with high melting temperature.

The list of experts who had made significant contribution to the formulation of this 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 or analysis 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.

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Indian Standard

CALCINED ALUMINA — SPECIFICATION

1 SCOPE

This standard covers the requirements and method of test for calcined alumina.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision 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.

<i>IS No.</i>	<i>Title</i>
264 : 2005	Nitric acid — Specification (<i>third revision</i>)
266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
4200 : 2020	Sodium aluminate — Specification (<i>second revision</i>)

3 GRADING

Calcined Al_2O_3 is graded in 3 grades based on soda content. The grades can be named as Grade-1 (low soda content), Grade-2 (medium soda content) and Grade-3 (high soda content).

4 REQUIREMENTS

The material shall consist essentially of calcined alumina which shall be in the form of a white powder. Material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, dry and airtight steel drums or as agreed to between the purchaser and the supplier. The containers shall not be opened until required for use and shall not remain open for a period longer than required for taking out the material.

5.2 Marking

5.2.1 Each container shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Indication of the source of manufacture;
- c) Mass of the material in the container;
- d) Date of packing; and
- e) Batch number.

5.2.2 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.

5.2.2.2 Physical parameters

These parameters depend upon the process involved and controls during production and should be optional *see* Table 2.

Table 1 Requirements for Calcined Alumina
(Clause 4)

SI No.	Characteristic	Requirements			Method of Test, Reference to Clause of Annex A
		Grade-1	Grade-2	Grade-3	
(1)	(2)	Low	Medium	High	(6)
i)	Aluminium oxide (percent by mass, <i>Min</i>)	99.5	99.0	97.5	A-2
ii)	Sodium oxide (Na ₂ O), (percent by mass)	less than 0.1	0.1-0.2	0.2-0.45	A-3 and A-13
iii)	Silica (SiO ₂), (percent by mass, <i>Max</i>)	0.15	0.15	0.1	A-4
iv)	Iron oxide (Fe ₂ O ₃), (percent by mass, <i>Max</i>)	0.03	0.03	0.1	A-5 and A-13
v)	Calcium (CaO), (percent by mass, <i>Max</i>)	0.1	0.1	0.1	A-6 and A-13
vi)	Loss on ignition (percent by mass, <i>Max</i>)	0.5	0.5	2.0	A-7

Table 2 Requirements for Physical Parameters
(Clause 5.2.2.2)

SI No.	Characteristic	Requirements			Method of Test, Reference to Clause of Annex A
		Grade-1	Grade-2	Grade-3	
(1)	(2)	Low	Medium	High	(6)
i)	pH	To be agreed between parties			A-8
ii)	Bulk density (g/cm ³), <i>Min</i>	1.0	1.0	1.2	A-9
iii)	Surface area (m ² /g)	As agreed between the interested parties			A-10 and A-11
iv)	Sieve analysis	As agreed between the interested parties			A-12

ANNEX A

(Clause 4, Table 1)

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF ALUMINIUM OXIDE**A-2.1 Outline of the Method**

From an aliquot of the main solution, after dilution iron and titanium are precipitated by treatment with caustic soda solution. The precipitate is filtered through dry filter and alumina in the filtrate is determined complexometrically.

A-2.2 Reagents

A-2.2.1 Caustic Soda — 20 percent (m/v).

A-2.2.2 Concentrated Hydrochloric Acid - rd = 1.16

A-2.2.3 Sodium Carbonate — Solid.

A-2.2.4 EDTA (0.05 M) — Dissolve about 18.64 g of the disodium salt of EDTA in water and transfer to a 1000 ml volumetric flask, dilute to the mark and mix.

A-2.2.5 Methyl Red Indicator Solution — 0.1 percent (m/v) in rectified spirit.

A-2.2.6 Dilute Hydrochloric Acid — 1 : 1 (v/v).

A-2.2.7 Buffer Solution (pH 5.3) — Dissolve 21.5 g of sodium acetate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$) in 300 ml water, add 2 ml glacial acetic acid and dilute to one litre.

A-2.2.8 Standard Zinc Solution (0.05 M) — Dissolve 3.26 g of electrolytic zinc granules (99.95 percent purity) in dilute hydrochloric acid (1 : 1) and evaporate the solution to 1 to 2 ml, dilute with water add 50 ml buffer solution (pH 5.3), mix and make up the volume to one litre. Standardize against standard aluminium solution following the method for determination of aluminium.

A-2.2.9 Standard Aluminium Solution (0.05 M) — Weigh accurately 1.3490 g of pure aluminium wire (99.9 percent) in a 500 ml beaker containing 50 ml of water and 50 ml concentrated hydrochloric acid. Heat the solution to dissolve the metal. Cool and dilute the solution to 1000 ml in a volumetric flask. (1 ml = 0.002 550 g Al_2O_3).

A-2.2.10 Xylenol Orange Solution — 0.5 percent (m/v) in water acidified with 2 drops of dilute hydrochloric acid (40 percent).

A-2.2.11 Ammonium Fluoride — solid.

A-2.3 Procedure

A-2.3.1 Pipette out 50 ml aliquot from the main solution into 400 ml beaker and neutralize the solution with caustic soda solution. Dissolve the precipitate formed in minimum quantity of concentrated hydrochloric acid and pour the solution slowly with constant stirring to a 200 ml volumetric flask containing 25 ml of caustic soda solution and 1 g sodium carbonate. Keep this on the water-bath for about an hour and then cool to room temperature. Make up the volume to the mark with water and shake thoroughly. Filter dry and collect the filtrate in a dry beaker or dry polythene bottle.

A-2.3.2 Pipette out an aliquot (100 ml) from the filtrate to a 250 ml conical flask. Add about 30 ml of 0.05 M EDTA solution and a drop of methyl red indicator. Neutralize the solution with dilute hydrochloric acid, add 15 ml buffer solution and boil the solution for about 2 min. Cool to room temperature and titrate with standard zinc solution using 2 to 3 drops of xylenol orange indicator. The colour changes from yellow to red. Add about 2 g of ammonium fluoride to the solution and boil the solution for about 2 min. Add 10 ml buffer solution cool to the room temperature and then titrate with standard zinc solution adding a drop of xylenol orange indicator. The end point is from yellow to pinkish red. The percentage of alumina is calculated from the titre value of second titration with zinc.

A-2.4 Calculation

$$\text{Alumina (Al}_2\text{O}_3\text{), percent} = \frac{A \times B}{C} \times 100$$

A = ml of zinc solution required for second titration,

B = equivalent Al_2O_3 (in g) per ml of zinc solution, and

C = mass in g of the sample as represented by the aliquot taken.

NOTE — This method is applicable when CaO and MgO is < 0.3 percent (m/m).

A-3 DETERMINATION OF SODIUM OXIDE (Na_2O)**A-3.1 Apparatus**

A-3.1.1 Flame Photometer, along with digital reading output

A-3.2 Reagents

A-3.2.1 Concentrated Nitric Acid (see IS 264)

A-3.3 Procedure

Weigh 0.5 g of the sample into a 250 ml platinum dish or crucible. Add 10 ml of water and 10 ml of concentrated nitric acid and cover the dish. Evaporate the contents to dryness on a hot plate. Add another 10 ml of concentrated nitric acid and evaporate to dryness again. Extract the residue with 20 ml of nitric acid (1 : 1) by gentle boiling followed by washing the cover with minimum amount of acid solution and take the wash into the dish.

Filter through Whatman No. 40 or equivalent filter paper into a 250 ml volumetric flask. Wash three times with hot water. Cool the flask and dilute to the mark with water. Take 25 ml of the solution and dilute to 100 ml in a volumetric flask.

Aspirate the solution to flame photometer and read the intensity of sodium emission. The instrument shall be calibrated in the range of 0 to 5 mg/l of sodium and the calibration curve shall be plotted with concentration of x-axis and emission intensity on y-axis.

A-3.4 Calculation

Sodium and its compounds (as Na₂O), percent by mass = $\times \times 0.2$

where

\times = mg/l of Na₂O from the calibration curve.

A-4 DETERMINATION OF SILICA (AS SiO₂)**A-4.1 Principle**

The sample is decomposed with an acid mixture and evaporated to fumes. The fumed mass is extracted with water, boiled to dissolve the salts and filtered. The residue is ignited and silica is determined through hydrofluorization.

A-4.2 Reagents

A-4.2.1 Concentrated Sulphuric Acid, rd = 1.84 (conforming to IS 266)

A-4.2.2 Concentrated Nitric Acid, rd = 1.42

A-4.2.3 Concentrated Hydrochloric Acid, rd = 1.16

A-4.2.4 Dilute Sulphuric Acid = 1:20 (v/v) and 1:4 (v/v)

A-4.2.5 Sulphuric Acid Washing Solution = 1:25 (v/v)

A-4.2.6 Sodium Carbonate (Anhydrous)

A-4.2.7 Hydrofluoric Acid = 40 Percent.

A-4.2.8 Mixed Acids-Add slowly 150 ml of concentrated sulphuric acid to 450 ml of water while stirring cool and add 200 ml of concentrated hydrochloric acid and 200 ml of concentrated nitric acid.

A-4.3 Procedure

A-4.3.1 Transfer 1.00 g of accurately weighed sample to a 400 ml pyrex beaker. Add 100 ml of freshly prepared mixed acids, cover at low heat till the brown fumes have disappeared. Remove the cover and wash the sides of beaker with little water. Continue heating until the fumes of sulphur trioxide (SO₃) are given off. After cooling to room temperature add about 100 ml of dilute sulphuric acid (1 : 20) and bring to boiling temperature on a hot plate and continue boiling till the sulphates goes in the solution leaving silica and if any unattacked sample. Allow to settle, filter (use Whatman filter No. 40 with pulp) and wash the residue and the filter paper with hot washing solution until free from dissolved salts, collect the filtrate and washings in 500 ml beaker.

A-4.3.2 Transfer the filter paper in a platinum crucible, ignite in a furnace, cool the ignited residue (containing a little of undissolved sample and silica) and mix with 2 g of sodium carbonate. Cover the platinum crucible with lid, heat slowly on low gas flame in the beginning and increase the temperature with intermittent swirling of the sample. Finally put the lid and keep in a furnace at 1 000°C for about 15 minutes. Remove and cool the crucible to room temperature and dip the crucible along with lid in the beaker containing main solution A. The cake is disintegrated with glass rod and removed from the crucible. Wash the crucible and lid and collect the washings in the sample Solution A. The main solution is evaporated slowly and heating is continued till the fumes of sulphur trioxide (SO₃) are given off. Cool and add 100 ml of dilute sulphuric acid (1 : 20) from the sides of beaker and bring to boiling on a hot plate to dissolve all the solids other than dehydrated silica. Allow to settle, filter (use Whatman filter paper No. 40 with pulp) and wash the residue and the filter paper with hot washing solution until free from dissolved salts. Collect the filtrate and washings in 500 ml volumetric flask (Solution A). Transfer the filter paper to a platinum crucible, ignite in furnace to constant mass at 950 to 1000 °C, cool in a desiccator and weigh. Moisten the precipitate with a few drops of water, add 1 to 2 ml of dilute sulphuric acid (1 : 4) and 10 ml of hydrofluoric acid, evaporate slowly to dryness at a low heat. Repeat the process with the addition of 2 to 5 ml of hydrofluoric acid and evaporate slowly to dryness. Ignite in a furnace at 950 to 1 000°C for 5 to 10 min, cool in a desiccator and weigh. Preserve the residue.

A-4.3.3 Carry out a blank determination following the same procedure and using the same quantity of all reagents without the sample.

A-4.3.4 Preparation of Main Solution

Fuse the residue preserved under **4.3.2** thoroughly with about 1 g of sodium carbonate extract with 20 to 30 ml

of dilute sulphuric acid (1 : 20). Add the extract to the filtrate and washings (Solution A) obtained under 4.3.1 and make up the volume up to the mark with water and mix thoroughly.

A-4.4 Calculation

$$\text{Silica, Percent} = \frac{(A-B)-C}{D} \times 100$$

A = mass in g, of platinum crucible with the residue before volatilization;

B = mass in g, of platinum crucible with the residue obtained after repeated volatilization with HF;

C = mass in g, of silica obtained from the blank determination; and

D = mass in g, of the sample taken.

A-5 DETERMINATION OF IRON OXIDE

A-5.1 Principle

Iron oxide is determined spectrophotometrically using o-phenanthroline.

A-5.2 Reagents

A-5.2.1 Standard Iron Solution (10 µg/ml)

Dissolve 0.4911 g of ferrous ammonium sulphate [(FeSO₄) . (NH₄)₂ SO₄ . 6H₂O)] in water. Add 10 ml of 1 : 1 sulphuric acid and dilute with water to 1 000 ml. Pipette out 50 ml of the solution and dilute with water to 500 ml. One millilitre of this solution is equivalent to 0.01 mg of iron.

A-5.2.2 Sulphuric Acid - 1 : 1.

A-5.2.3 Hydroxylamine Hydrochloride Solution - 10 percent (m/v),

A-5.2.4 O-Phenanthroline Solution

Dissolve 0.1 g of o-phenanthroline in 2 ml of methanol and dilute to 100 ml with water and store in a dark bottle with a ground-glass stopper.

A-5.2.5 Sodium Acetate Solution - 2 M.

A-5.3 Procedure

Take 10, 20, 30, 40 and 50 ml aliquots of standard iron solution in separate 100 ml beakers and add reagent grade water to adjust volume approximately to 50 ml, wherever required. Adjust the pH between 2 to 3 using 1 : 1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water and make up the volume in each beaker to 100 ml. Determine the absorbance after 30 min against reagent blank consisting of same amount of all reagent in reagent grade water at 510 nm using a spectrophotometer. Plot a calibration curve of absorbance against concentration of 1 µg/ml to 5 µg/ml

as obtained by following the above dilution steps using the standard iron solution.

Transfer 1.00 g of accurately weighed sample to a 400 ml pyrex beaker. Add 100 ml of freshly prepared mixed acids, cover it and give low heat till the brown fumes have disappeared. Remove the cover and wash the sides of beaker with little water. Continue heating until the fumes of sulphur trioxide (SO₃) are given off. After cooling to room temperature add about 100 ml of dilute sulphuric acid (1 : 20) and bring to boiling temperature on a hot plate and continue boiling till the sulphates goes in the solution leaving silica and if any unattacked sample. Allow to settle, filter (use Whatman filter No. 40 with pulp) and wash the residue and the filter paper with hot washing solution until free from dissolved salts, collect the filtrate and washings in 500-ml beaker.

Take 10 ml aliquot in a 100 ml beaker. Adjust the pH to 2 to 3 using 1 : 1 sulphuric acid and then between 3 to 4 using sodium acetate solution. Add 2 ml of hydroxylamine hydrochloride solution and 5 ml of o-phenanthroline solution. Dilute with water the contents of the beaker to 100 ml. Determine the absorbance after 30 min against reagent blank at 510 nm using a spectrophotometer. Read the concentration for the absorbance determined using the calibration curve.

A-5.4 Calculation

Iron oxide (as Fe₂O₃), percent by mass = µg/ml of Fe₂O₃ from the calibration curve X 0.05

A-6 DETERMINATION OF CALCIUM (AS CAO)

A-6.1 Outline

Sample is fused with sodium carbonate and boric acid and dissolved in hydrochloric acid and hydrogen-peroxide. Suitable aliquots are aspirated into flame of atomic absorption spectro- photometer using nitrous oxide-acetylene burner for the determination of calcium.

A-6.2 Reagents

Sodium carbonate, anhydrous, boric acid, solid, concentrated hydrochloric acid (rd = 1.16), Hydrogen Peroxide, 30 percent (v/v).

A-6.2.1 Background Solution

Weigh 5.72 g of super purity-aluminium turnings. Dissolve in 200 ml of NaOH solution (5 N). When the aluminium has dissolved, add 20.0 g of boric acid and 400 ml of concentrated hydrochloric acid. Transfer to a 1 000 ml volumetric flask, dilute to volume with water and mix. Store in a polyethylene bottle. Dissolve 2.50 g of iron powder in 100 ml dilute hydrochloric acid (1 : 1). Warm to complete dissolution. Transfer to

a 250 ml volumetric flask, dilute with water and mix. This solution contains 10 mg Fe/ml.

Dissolve 2.50 g of iron powder in 100 ml dilute hydrochloric acid (1 : 1). Warm to complete dissolution. Transfer to a 250 ml volumetric flask, dilute with water and mix. This solution contains 10 mg Fe/ml.

Dissolve 7.4 g of potassium titanium oxalate in 300 ml of water. Transfer to a 500 ml volumetric flask, dilute to volume with water and mix. This solution contains 2 mg Ti/ml.

In a stainless steel beaker, dissolve 1.392 g of silicic acid in 50 ml of NaOH solution (5 N). Warm if necessary to complete dissolution. Transfer to 500 ml polyethylene volumetric flask, dilute to volume with water and mix. Store in a polyethylene bottle. This solution contains 1 mg Si/ml.

Dissolve 26.6 g of lanthanum chloride heptahydrate ($\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$) in water, dilute to 100 ml and mix. This solution contains 100 mg of La/ml.

Into each of the 250 ml volumetric flasks set aside, for the preparation of the calibration solutions, pipette the following:

- 50 ml of aluminium-boric acid solution;
- 10 ml of iron solution;
- 10 ml of titania solution;
- 10 ml of silica solution; and
- 50 ml of lanthanum solution.

A-6.2.2 Standard Calcium Solution, (100 and 10 mg of Ca/litre)

Weigh 0.624 g of pure calcium carbonate in a 250 ml beaker, add 25 ml of water and 10 ml of concentrated hydrochloric acid. When the salt has dissolved, transfer in 250 ml volumetric flask, dilute with water and mix. Store in a polyethylene bottle. This solution contains 1 000 mg Ca per litre.

Transfer 25 ml of this solution in a 250 ml volumetric flask. Transfer into a polyethylene bottle. This solution contains 100 mg Ca per litre (B). Transfer 25 ml of solution (B) to 250 ml volumetric flask. Dilute with water and mix. Transfer it to a polyethylene bottle. This solution contains 10 mg Ca per litre.

A-6.3 Procedure

A-6.3.1 Weigh 1 g of sample in a platinum crucible. Add 3.5 g of sodium carbonate-boric acid fusion mixture (2 : 1) and mix thoroughly by stirring with platinum rod. Place the crucible in a muffle furnace

at 800°C and immediately raise the temperature to 1 000°C. Keep the crucible in the furnace for 1 h. Cool the crucible.

A-6.3.2 Strike the bottom of the crucible with the wooden handle of a spatula to crack the melt. Dislodge the solids by hitting the sides of the crucible with the wooden handle of the spatula and by gently pushing in the bottom and the sides. Fill the crucible with water and gently heat on a hot plate until the melt has softened. Transfer the mass to a 250 ml beaker using a gentle hot water jet. Add to the crucible a few ml of hot water, 5 ml of concentrated hydrochloric acid and 2 ml of hydrogen peroxide.

Gently heat on a hot plate until the residue is well dissolved. Remove the last traces of melt adhering to the sides of the crucible by adding 10 ml of water and 15 ml of concentrated hydrochloric acid. Heat the crucible on the edge of a hot plate until the solids have dissolved. Transfer the contents of the crucible to the 250 ml beaker. Add 2 ml of hydrogen peroxide.

Heat the contents of the beaker gently on the edge of the hot plate until the evolution of small bubbles has stopped.

NOTES

1 Do not heat strongly (silica will be precipitated). Continue heating if necessary until all the melt is dissolved. Transfer the contents of the beaker to a 250 ml volumetric flask, dilute with water and mix. Transfer 25 ml of the solution into a 50 ml volumetric flask. Dilute to volume with water and mix. (Final test solution).

2 For ores having higher calcium contents than 0.84 percent CaO, an intermediate dilution step prior to the 25ml/50 ml of final dilution is required (for example 1/5 or 1/2.5).

A-6.3.3 Blank

Prepare the reagent blank using 3.5 g of fusion mixture and following the same procedure as specified above.

A-6.3.4 Calibration Solution

To 130 ml (50 + 10 + 10 + 10 + 50 ml) of background solution add the volume of respective standard solution given in Table 3. Dilute to 250 ml in volumetric flasks.

Transfer 25 ml of each of the solutions to dry 50 ml volumetric flasks. Dilute to volume with water and mix. These solutions are the final calibration solutions see Table 3.

NOTE — The range of calcium that can be covered may vary from instrument to instrument. More diluted or more concentrated calibration solutions can be used by adjusting accordingly the portions of standard solutions required.

Table 3 Standard Solutions (Calcium) for Calibration Curve
(Clause A-6.3.4)

Percent CaO	Volume in ml of Standard Solution 10 mg Ca/L	Volume in ml of Standard Solution 100 mg Ca/L
0.014	10.0	0
0.028	20.0	0
0.070	0	5.0
0.140	0	10.0
0.210	0	15.0
0.280	0	20.0
0.560	0	40.0
0.840	0	60.0

A-6.3.5 Zero Calibration Solution

Transfer 130 ml (50 + 10 + 10 + 10 + 50 ml) of background solution into 250 ml volumetric flasks, dilute to volume and mix. Transfer 25 ml of this solution into 50 ml volumetric flask, dilute and mix.

A-6.3.6 Adjustment of Atomic Absorption Spectrometer

Fit the burner in accordance with manufacturer's instructions. Light nitrous oxide - acetylene flame. Set the wavelength for magnesium (285.2 nm) and calcium (422.7 nm). After 10 min preheating of the burner, adjust fuel and burner to obtain the maximum absorbance. Aspirate water and calibration solution, to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance.

A-6.3.7 Atomic Absorption Measurements

Aspirate the calibration solutions and the final test solutions in order of increasing absorption starting with diluted blank test solution and the zero calibration solution, with the final test solutions being aspirated at the proper point in the series. Aspirate water between each solution and record the readings when stable responses are obtained. Repeat the measurements twice and average the readings.

Obtain the net absorbance of each calibration solution by subtracting the absorbance of the zero calibration solution. Similarly, obtain the net absorbance of the final test solutions by subtracting the absorbance of the diluted blank test solution.

Prepare a calibration curve by plotting the net absorbance values of the calibration solutions against the concentrations of magnesium or calcium, expressed as percent CaO.

Convert the net absorbance values of the final test solutions to percent CaO by means of the calibration curves.

A-6.3.8 Calculation of Calcium Content

The calcium content, as a percentage by mass, is calculated from the following formula: $\frac{C \times 1.000 \times f}{m}$

where

c = concentration, expressed as percent CaO, in 1.000 0 g test portion;

m = exact mass, in grams, of test portion; and

f = dilution factor.

If any further dilution is required then use the dilutions described.

A-6.3.8.1 From the calcium content calculated above, the percentage of calcium oxide is calculated as follows:

$$\%Ca \times 1.39 = \%CaO$$

A-7 LOSS ON IGNITION

A-7.1 Apparatus

A-7.1.1 Platinum crucible, Muffle furnace

A-7.2 Procedure

Weigh accurately 1.00 g of sample into a previously weighed platinum crucible. Heat slowly first and then at a gradually increasing temperature. Finally ignite at 1 000°C for one hour. Cool in a desiccator containing preferably magnesium perchlorate and weigh. Repeat the heating till constant weight.

A-7.3 Calculation

Loss on heating, percent by mass = $\frac{(A-B)}{C} \times 100$

where

A = initial mass in g, of the crucible with sample,

B = final mass in g, of crucible with residue after ignition, and;

C = mass in g, of the sample taken.

A-8 pH VALUE OF 10 PERCENT SOLUTION

A-8.1 Apparatus

A-8.1.1 pH Meter — a suitable pH meter fitted with glass and calomel electrodes. Calibrate the pH meter using buffer solutions of pH = 4, pH = 7 and pH = 9

A-8.2 Procedure

Prepare an aqueous extract of material by shaking for 1 h, 3 g of the material with 50 ml of water (free from ammonia and carbon dioxide), in a clean polyethylene flask. Decant the clear solution and measure the pH by using any standard pH meter.

A-9 BULK DENSITY

A-9.1 Procedure

Transfer the material as received and pour slowly in a 250 ml stoppered measuring cylinder up to 50-ml mark without knocking. Level the top bed of surface to the mark by gently lifting the cylinder as far as 5 cm from the base pad and releasing it to allow free fall to knock the pad. Repeat this knocking for 4 more times. Add further 50 ml of the sample and knock five times, as above, after every 50 ml addition up to 250 ml. Level the cylinder with the material without any further knocking. Empty out the material from the cylinder and weigh to the nearest 0.5 g. Then heat the sample in an unventilated oven at 250±5°C for 3 h. Place the cylinder in a desiccator, cool and weigh to constant mass.

A-9.2 Calculation

Bulk density, g/ml = 0.004 M

where

M = mass in g, of the material in the cylinder.

A-10 DETERMINATION OF SURFACE AREA

A-10.1 Apparatus

The apparatus consisting of the following is assembled as shown in Fig. 1.

A-10.2 Procedure

Connect the chamber, flow meters and nitrogen cylinder as shown in Fig. 1. Take the sample in the adsorption cell and regenerate it in the tubular furnace at 250°C with dry air/nitrogen purge. Cool the sample in an empty desiccator. Adjust the two nitrogen flow inlets to get a partial pressure of 0.05 to 0.075 mm of Hg. Weigh the cooled sample and place it in the adsorption chamber. Carry out the adsorption for two hours till equilibrium is reached. Weigh the cell. Change the partial pressure of benzene to a higher value, say 0.125

to 0.150 and continue the adsorption by replacing the cell in the chamber for 1 hour. Note four readings in the partial pressure range of 0.05 to 0.35 mm of Hg.

10.3 Calculation

BET equation is

$$\frac{P/P_o}{g \left(1 - \frac{P}{P_o} \right)} = \frac{1}{g m^c} + \frac{c-1}{g m^c} \frac{P}{P_o}$$

Where

g = amount of benzene adsorbed at any P/P_o.

g_m = amount of adsorbate (benzene) required to form monolayer on the surface

C = constant

If $\frac{P/P_o}{g(1 - \frac{P}{P_o})}$ is plotted against P/P_o, a straight line

(BET) plot is obtained in which slope = C-1/g_mC and intercept = 1/g_mC

Slope and Intercept = C-1/g_mC + 1/g_mC - 1/g_m

From the plot, the value of g_m can be obtained.

The constant C = (slope+1)/Intercept is a measure of the difference in heats of adsorption of the 1st layer and the succeeding layers. The specific surface area is given by:

$$S, \text{ m}^2/\text{g} = g_m \cdot N \times B/m \times M$$

where

N = Avogadro's No.;

B = molecular area of the adsorbate (benzene) = 41Å² = 41 X 10⁻²⁰ m²;

M = mass of the sample; and

M = molecular mass of benzene.

On substitution, we get, S = g_m X 3166/m

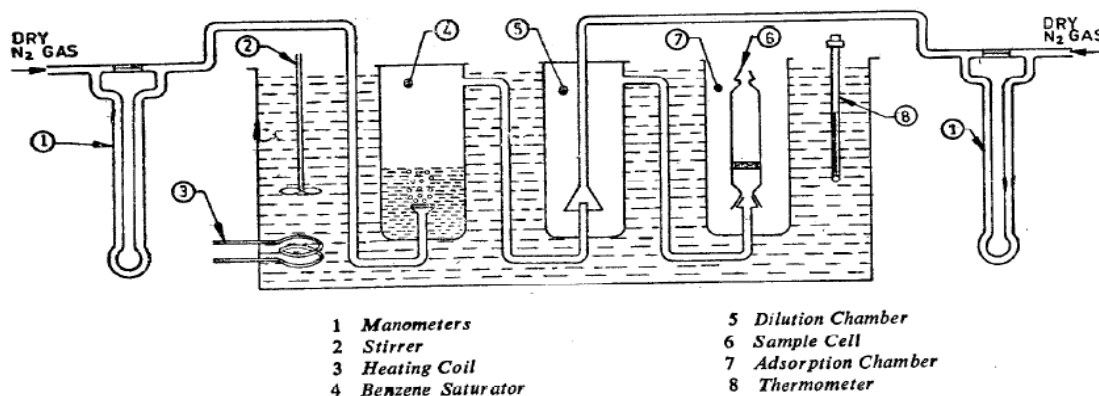


FIG. 1 APPARATUS FOR SURFACE AREA MEASUREMENT

A-11 TOTAL AND EXTERNAL SURFACE AREA BY NITROGEN ADSORPTION METHOD

A-11.1 Outline of the Method

This method is used to determine the total and external surface area of calcined alumina based on nitrogen adsorption. The nitrogen surface area measurement is based on the B.E.T theory.

A-11.2 Apparatus

A-11.2.1 *Multipoint Static Volumetric Gas Adsorption Apparatus with Dewar flasks and Accessories required.*

A-11.2.2 *Sample Cells*

A-11.2.3 *Analytical Balance*

A-11.2.4 *Heating Mantle*

A-11.2.5 *Oven*

A-11.3 Reagents

A-11.3.1 *Liquid Nitrogen, 98 Percent or Higher Purity*

A-11.3.2 *Ultra High Purity Nitrogen Gas*

A-11.3.3 *Ultra High Purity Helium Gas*

A-11.4 Sample Preparation Procedure

A-11.4.1 Dry calcined alumina sample at a temperature of 125°C for about 1 h. This step can be omitted in case the sample is subsequently free from moisture. Empty sample is conditioned for 10 min at conditions which are intended for degassing the sample. Weigh the cell to nearest 0.1 mg and record the mass. Weigh 0.4 g of Calcined Alumina into the sample cell.

A-11.4.2 *Flow Degassing*

Open gas control valve and insert delivery tube into the sample tube. Allow purging with nitrogen or helium for a minute. Place some heat source around sample cell. Degas the sample at 300±10 degree for half an hour or longer in order to ensure that moisture traces are absent. Degassing time that gives a stable surface area may be used for degassing. Once the degassing time has been determined, samples can be degassed on time basis alone. Less than half an hour is required by some sample in case the exposure to moisture is minimum. Minimum time required in order to give a stable surface area is used for the process of degassing. After the degassing process, the tube containing the sample may be directly moved to the analyzer. Else, remove the tube from the source of heat and continue the purging gas flow till it is ready for analysis.

A-11.4.3 *Vacuum Degassing*

Keeping the apparatus at atmospheric pressure, keep the sample cell containing calcined alumina with degassing

apparatus. Start the process of degassing as appropriate for the apparatus. Place a heating mantle or some other heat source around sample cell. Degas the sample at 300 ± 10 degree C for about half an hour or more as required. Hold a pressure at ≤1.4 Pa (10 μm Hg). Once degassing times are determined, future samples can be degassed on time basis alone. Allow sufficient margin of excess time. Some samples requires ≤1/2 h in case the moisture exposure is minimal. Minimum amount of time that gives a stable Surface Area may be used for degassing.

11.5 Procedure

11.5.1 Refer user's manual and instructions for multipoint gas adsorption analyzer so that one can become familiar with the procedures. Number of instruments are available that offer a variety of saturation vapour pressure (P_0) measurement options and Dewar size. Dewar flask is filled with liquid nitrogen and allow it to reach temperature equilibrium. At every hour fill the Dewar flask. This should also be done between each analysis.

11.5.1.1 *Small dewar-*

Cover the Dewar after filling it for a minimum of 2 h. Dewar should be dry and clean at the end of each day.

11.5.2.1 *Large dewar (≥1L)-*

Cover the Dewar for 16 h prior to use, unless continuous P_0 measurements are employed. For continuous P_0 , use Dewar equilibration for 2 h. Once we have reached equilibration, a large Dewar can maintain this equilibration for several days if it is kept covered and filled. The frequency of cleaning is left to the discretion of the operator, but is not to exceed once per week.

11.5.2 *List of P_0 Measurement Options*

Continuous P_0 (measurement at each relative pressure point): The method is one of the best practice, however, the method generally increases analysis time.

11.5.2.1 *Single P_0 per analysis-*

This value can be measured before, during, or after the run, a P_0 value measured at the end of the analysis is preferred since STSA is calculated from last data points. This is significantly influenced by P_0 values. P_0 value in this method is determined prior to initiating measurements to ensure equilibrium of the Dewar.

11.5.2.2 *Daily P_0*

The method is used in case the evidence of stable Dewar is present and no changes in atmospheric pressure that is, greater than 0.13 kPa (1mm Hg) occur.

11.5.2.3 *Calculated P_0*

Value of P_0 is calculated by atmospheric pressure measurement and adding a value between 1.3 and

2.6 kPa (10 mm and 20 mm Hg). In order to determine the constant, operator is responsible, most commonly used pressure is 15 mm Hg.

With exceptions to continuous P_o measurements, it is recommended that P_o value be determined prior to NSA/STSA analysis. P_o value between 10 to 20 mm Hg which is above atmospheric pressure and two consecutive P_o values differing not more than 0.13 kPa (1mm Hg) over 10 min time period are indications of stable Dewar.

Expected difference in P_o and atmospheric pressure in lab is taught to the operator by experience. Free space of the sample cell is determined by measurement with helium or by calculating assumed carbon density of 1.9 g/cm^3 .

Obtain around five data points evenly spaced in relative pressure (P/P_o) range of 0.1 to 0.5. In order to increase the accuracy of measurement, it is recommended to take two additional data points.

Mass of the cell is determined with dry sample to nearest 0.1 mg. This is done before or after measuring nitrogen adsorption.

Avoid use of helium inconsistently, as a buoyancy error of 1 mg/cm^3 of cell volume can occur. Alternatively, mass of Calcined Alumina may be determined directly by pouring it into weighing pan from the sample cell.

11.6 Calculation

At the completion of the analysis, most automated instruments will perform the following computation. Verification of the internal computations by the user must be verified conforming to the following method.

11.6.1 Mass of the Sample

Dried Sample Mass-

(Mass of the cell + sample)-(Mass of the cell)

Record the mass to nearest 0.1 mg

Nitrogen Adsorbed (Volume):

Volume of adsorbed Nitrogen per gram of sample-

$$V_a = \text{Volume of Nitrogen (in cm}^3\text{) / Mass of the sample in (g)}$$

11.6.2 Surface Area

Using B.E.T plot, Nitrogen surface Area can be determined using Brunauer, Emmett and Teller equation:

$$P/V_a(P_o - P) = 1/V_m C + C - 1/V_m C \times P/P_o$$

where

P = manometer pressure in kPa;

P_o = standard vapour pressure of nitrogen (kPa);

V_m = nitrogen volume in gram which covers one monomolecular layer in standard cm^3/g ; and

C = B.E.T constant. Numerical value depends on heat of adsorption of monomolecular layer.

Plot $P/V_a(P_o - P)$ on the Y-axis and P/P_o on X axis for data sets with P/P_o in 0.05 to 0.30 range.

Three or more data points which gives best straight line are used to calculate the slope and Y-intercept. The Y-intercept and the slope are used to calculate the surface area.

Negative Y-intercept in B.E.T plot, may indicate the presence of micropores ($\leq 2 \text{ nm}$ diameter), but other factors can also produce negative intercept.

Calculate Nitrogen Surface Area to the nearest $0.1 \text{ m}^2/\text{g}$ as follows:

$$\text{Surface Area (m}^2\text{/g)} = V_m \times 4.35$$

where

$$V_m = \frac{1}{B + M}$$

B = Y axis intercept, $\pm 10^{-5}$

M = slope of straight line, $\pm 10^{-5}$; and;

4.35 = Area which 1 cm^3 of nitrogen occupy = $(6.02 \times 10^{23}) (16.2 \times 10^{-20}) / 22400$

6.023×10^{23} = Avogadro's Number;

16.2×10^{-20} = Nitrogen molecule area (in m^2); and

22400 = Number of cm^3 occupied by one mole of gas at STP.

11.6.3 Statistical Thickness Surface Area (STSA)

Determine STSA of Calcined Alumina using a plot of Nitrogen gas adsorbed volume per gram of the sample at STP (V_a) versus the statistical layer thickness (t).

Prepare V_a - t plot by plotting t (nm) on the X-axis versus V_a (dm^3/kg at STP) on the Y-axis with data sets having P/P_o equally spaced in the range of 0.2 to 0.5.

where

$$T = \text{statistical layer thickness of calcined alumina} = 0.88 (P/P_o)^2 + 0.645 (P/P_o) + 0.298$$

Slope can be determined by the use of standard linear regression.

Calculate STSA to nearest $0.1 \text{ m}^2/\text{g}$ as follows:

$$\text{STSA} = M \times 15.47$$

Where

M = slope of V_a - t plot and

15.47 = a constant for the nitrogen gas conversion to liquid volume and conversion of units to m^2/g

A-12 SIEVE ANALYSIS (WET METHOD)

If the sample contains substantial quantity of fine particles, wet method for particle analysis is required. Wetting of particles lowers the surface energy and reduces the force of attraction between the particles. Thus, the particles remain separated. Small amount of surfactants is added in case of liquid with high surface tension.

A-12.1 Procedure

Take 5 gm of the sample and soak it in suitable solvent. In case of deflocculation, surfactant may be added. Stir the sample and sample is left for soaking period of 10 min. Pass the material through 350 mesh sieve and wash the material until water filtered becomes clear. Collect the sample on 350 mesh sieve and dry it in oven. Collect the retained material on sieve and weigh.

A-12.2 Calculation

The weight of alumina retained on sieve is calculated as a percentage of the total dry weight of the sample taken.

$$\text{Percentage retained (percent by mass)} = \frac{A}{M} \times 100$$

where

A = mass of residue in g; and

M = mass of sample in g.

A-13 DETERMINATION OF SODIUM, CALCIUM AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-13.1 The solution under analysis is passed with the help of a peristaltic pump through a nebulizer inside a spray chamber. The aerosol formed is aspirated in the form of argon plasma where the atoms are excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and wavelength/intensity of emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

A-13.2 Reagents and Solutions

Magnesium nitrate, 6.67 percent-w/v: Add 66.7 g of magnesium nitrate hexahydrate in 1000ml of deionized water and mix well.

HCl solution: Dilute 83.0 ml conc. HCl to 1 000 ml deionized water.

50Percent nitric aAcid: Dilute 500 ml of Nitric Acid in 500 ml of deionized water.

A-13.3 Standards

(a) Yttrium Internal Standard (to infuse in the instrument T junction), 2 µg/ml- Prepare 1 000 µg/ml of Yttrium stock standard. Transfer 2 ml of this standard stock solution in 1 L volumetric flask and dilute the solution using 1N HCl.

(b) Yttrium Working internal standard: Pipette out 25 ml of 1 000 µg/ml Yttrium stock standard into 50 ml volumetric flask and dilute using 1N HCl.

If Yttrium Internal Standard is used to infuse in the instrument, then there is no need to add Yttrium during the preparation of calibration standards.

Sodium-Calibration standard 0 µg/ml- Add 0 µl of 10 000 µg/ml sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium- Calibration standard 6 µg/ml-Add 30 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium-Calibration standard 12 µg/ml-Add 60 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium-Calibration standard 24 µg/ml-Add 120 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Sodium-Caibration standard 48 µg/ml-Add 240 µl of 10 000 µg/ml of sodium stock solution in 50 ml volumetric flask. To this add 100 µl of Yttrium and dilute to volume with 1 N HCl.

Similar calibration standard solutions can be prepared for iron and calcium analysis.

A-13.4 Sample Preparation

Process the sample until homogeneous.

A-13.5 Procedure (Sample Preparation)

Take sample in a platinum crucible. Add magnesium nitrate (2 ml) to the sample and gently swirl it. Place the sample in a cool muffle furnace with temperature less than 80 °C. Set the furnace program as:

- | | | |
|--------|-----------------|---------------|
| Step 1 | Ramp = 3°C/min | Level = 100°C |
| | Dwell = 360 min | |
| Step 2 | Ramp = 3°C/min | Level = 150°C |
| | Dwell = 60 min | |
| Step 3 | Ramp = 3°C/min | Level = 500°C |
| | Dwell = 480 min | |
| Step 4 | Ramp = end | |

Else, heat the sample in the furnace gradually as per the above steps by controlling the sample through the thermostat.

The sample must not be heated so rapidly that it ignites.

Remove the sample and cool the furnace at room temperature. Add 2 ml 50 percent aqueous HNO₃ and wash with it the sides of the crucible. Dissolve all the ash and transfer in the beaker. Remove excess of acid by slow heating of the sample on a hot plate. Make sure that the sample is free from any black precipitate. If any precipitate is found, again transfer the dry mass of the beaker into the crucible using little water and evaporate the water and heat at 500-550°C for 1 h. Again repeat this procedure till the sample becomes free from black precipitate. Remove the sample from furnace and cool it to room temperature. Add HCl (5 ml, 1 N) and dissolve the ash/mass. Transfer the above solution to 50 ml graduated flask and make up the final volume to

50 ml using 1 N HCl. Fortify the sample with 200 µl of Yttrium working standard and mix it. This step is omitted if T junction is used on the instrument.

Analyse the sample using ICP/OES.

A-13.6 Instrument Settings

Set up the instrument as per the manufacturer's instructions. Relative to the internal standard concentration, the quantification of the ions is done by the system software. Calibrate the instrument using ICP reagent blank and the calibration standard. Monitor the wavelengths for the respective metal ions.

Na = 589.592 nm, Pb = 220nm, Fe = 238.2 nm

A-13.7 Manual Calculation

Use linear regression analysis to determine a standard curve (emission *versus* concentration)

$$\text{ppm(analyzed)} = \frac{\mu\text{g/ml (evaluated from calibration curve)} \times \text{ml (final volume)}}{\text{g (sample weight)}}$$

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This Indian Standard has been developed from Doc No.: CHD 01 (15167).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi