

भारतीय मानक मसौदा
सक्रिय एलुमिना — विशिष्टि

(IS 9700 का दूसरा पुनरीक्षण)

Draft Indian Standard
Activated Alumina — Specification
(*Second Revision of IS 9700*)

(*Not to be reproduced without the permission of BIS or used as an Indian Standard*)

ICS 71.060.20

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 26th March 2025

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*formal clauses will be added later*)

This standard was first published in 1981 and subsequently revised in 1991. In the first revision, new requirements on alumina content, pore volume and surface area were incorporated along with the relevant methods of test. Also, requirements on pH, loss on ignition, density and bed crushing strength were modified.

In this second revision, instrumental test methods for the determination of chloride and sulphate have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Amendment no. 1 has been incorporated. Reference clause and Packing & Marking clause have been updated.

Activated alumina is manufactured by thermal treatment of the following:

- alumina gel manufactured by precipitation technique;
- alumina rehydrated after rapid activation of alumina hydrate; and
- alumina hydrate.

The thermal treatment is given so that eta, gamma and chi alumina phases are formed which are the major constituents of adsorbent alumina.

Activated alumina is used in the industry as a desiccant/adsorbent, primarily intended for application in industrial air drying, gas and liquid drying, packing and in chromatographic analysis. This standard, however, does not cover the material intended for chromatographic use.

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.

Draft Indian Standard
ACTIVATED ALUMINA — SPECIFICATION
(*Second Revision*)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for activated alumina used as a desiccant/adsorbent.

2 REFERENCES

The standards listed 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 editions of the standards indicated below:

<i>IS No</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 460 (Part 1) : 2020	Test sieves — Specification Part 1 Wire cloth test sieves (<i>fourth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)

3 GRADES

There shall be following two grades of the material:

Grade 1 — For high pressure air drying, and

Grade 2 — For general purposes.

4 REQUIREMENTS**4.1 Description**

The material shall consist of activated alumina in spherical or pellet form as agreed to between the purchaser and the supplier.

4.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col (5) of the 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

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.

5.2.1 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 Activated Alumina

(Clause 4.2)

SL No.	Characteristic	Requirement		Method of Test (Ref to Annex of this Standard)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass <i>Max</i>	2.0	2.0	A-2
ii)	pH value	4.6 to 8.0	6.0 to 10.0	A-3
iii)	Aluminium oxide (as Al ₂ O ₂), percent by mass <i>Min</i>	91.0	90.0	A-4
iv)	Water soluble chlorides (as Cl), percent by mass, <i>Max</i>	0.7	—	A-5
v)	Water soluble sulphate (as SO ₄), percent by mass, <i>Max</i>	0.7	—	A-6
vi)	Sodium and its compounds (as Na ₂ O), percent by mass, <i>Max</i>	0.8	—	A-7
vii)	Loss on ignition, percent by mass, <i>Max</i>	8.0	8.5	A-8
viii)	Adsorption capacity, percent, <i>Min</i>	20.0	14.0	A-9
ix)	Pore volume, ml/g, <i>Min</i>	0.40	0.30	A-10
x)	Surface area, m ² /g, <i>Min</i>	350	250	A-11
xi)	Bulk density, g/ml, <i>Min</i>	0.75	0.85	A-12
xii)	Attrition, Loss, percent by mass, <i>Max</i>	—	—	A-13
	a) By tumbling	0.50	1.0	
	b) By rotation	1.50	2.50	
xiii)	Bed crushing strength, percent, <i>Min</i>	90	80	A-14

NOTE — Requirements for moisture, adsorption capacity, pore volume, surface area, loss on attrition and bed crushing strength are critical for adsorbent application.

6 SAMPLING AND CRITERIA FOR CONFORMITY

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A

(Clause 4.2)

METHODS OF TEST FOR ACTIVATED ALUMINA

A-1 QUALITY OF REAGENTS

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

NOTE — ‘pure chemicals’ that do not contain impurities which affect the results of analysis.

A-2 MOISTURE

A-2.1 Apparatus

A-2.1.1 Glass Weighing Dish

Flat bottomed of 20 ml capacity and preferably of following dimensions:

Internal diameter	44 mm
Height	25 mm

A-2.1.2 Desiccator

Vacuum desiccator of 15 cm to 20 cm diameter.

A-2.1.3 Weighing Bottle

Diameter — 6.5 mm to 70 mm

Height — 30 mm to 35 mm

A-2.2 Procedure

Weigh accurately 5 g to 7 g of the sample directly from the packed container in a covered glass weighing dish. Remove the cover and heat the bottle and its contents in an unventilated oven at (250 ± 5) °C for 3 h. Replace the cover, cool in a desiccator and weigh to constant mass. Use this dried material for the determination of loss on ignition given in A-8.2.

A-2.3 Calculation

$$\text{Moisture, percent by mass} = \frac{100(M_1 - M_2)}{M_1}$$

where

M_1 = mass in g, of the material before heating; and

M_2 = mass in g, of the material after heating.

A-3 pH VALUE

A-3.1 Apparatus

A-3.1.1 pH Meter

A-3.2 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-4 ALUMINIUM OXIDE

A-4.1 Apparatus

A-4.1.1 Muffle Furnace**A-4.1.2 Platinum Crucible****A-4.1.3 Steam Bath****A-4.2 Reagents****A-4.2.1 Sodium Carbonate, Anhydrous****A-4.2.2 Boric Acid****A-4.2.3 Nitric Acid — 8 N****A-4.2.4 Ammonia Chloride****A-4.2.5 Bromine water****A-4.2.6 Ammonium Nitrate Solution — 2 percent****A-4.2.7 Ammonium Hydroxide Solution — 1:1****A-4.3 Reagent Preparation****A-4.3.1 Fusion Mixture**

Mix thoroughly 250 g anhydrous sodium carbonate with 100 g boric acid and grind to a fine powder and store in a plastic bottle tightly fitted with a lid.

A-4.4 Procedure**A-4.4.1 Sample Solution Preparation**

Weigh accurately 1 g of the sample with 3.5 g of fusion mixture in clean and dry platinum crucible. Mix it intimately with fusion mixture using a suitable platinum rod. Place it at the door of the muffle furnace for 15 min, then transfer it to the muffle. Maintained at a temperature of 1 000 °C, for further 30 min. It should be noted that a melt and not a sinter is obtained. The melt will dissolve completely in water only if proper heat treatment has been carried out.

Cool the crucible and fill it with as much water as possible without risk of losses. Heat gently till the melt is dissolved. Cool and transfer the content of crucible to a 250 ml beaker containing 17.5 ml of 8 N HNO₃ (10.5 ml for blank). Swirl the content. Cover the beaker and heat the content on a steam bath until clear solution is obtained. Cool, transfer to 250 ml standard flask and dilute to the mark.

A-4.4.2 Estimation of Aluminium

Take 50 ml of aliquot of the solution (*see A-4.4.1*) in a 600 ml beaker, add 4 g to 5 g of ammonium chloride and two drops of bromine water. Heat to boiling. Add 1: 1 ammonium hydroxide solution while stirring until a slight excess (as indicated with litmus paper). Again boil, filter while hot and wash 5 times with hot ammonium nitrate solution. Dry the filter paper containing precipitate. Burn the dry filter paper with precipitate in a previously weighed crucible at the door step of muffle furnace and then at 1 000 °C for 30 min. Weigh the residue to constant mass.

A-4.5 Calculation

$$\text{Alumina (as Al}_2\text{O}_3\text{) percent by mass} = 500 M$$

where

$$M = \text{mass in g, of the residue.}$$

A-5 WATER SOLUBLE CHLORIDES**A-5.1 General**

Two methods are prescribed for determining chlorides, namely, Method A and ion chromatography method as prescribed in **A-15**. In case of dispute, ion chromatography method shall be used as referee method.

A-5.2 Method A**A-5.2.1 Reagents****A-5.2.1.1 Dilute nitric acid** — 1:1**A-5.2.1.2 Standard silver nitrate solution** — 0.05 N**A-5.2.1.3 Standard ammonium thiocyanate solution** — 0.05 N**A-5.2.1.4 Ferric alum indicator solution** — 10 percent m/v**A-5.2.2 Procedure****A-5.2.2.1 Preparation of solution**

Weigh accurately 2g of the material in a platinum crucible of 40 ml capacity previously washed with nitric acid. Heat the crucible and its contents in a furnace at 350 °C for 45 min. Boil for 2 to 3 min and then digest for half an hour. Allow to settle for 10 min and filter through Whatman No. 40 filter paper or equivalent into 200 ml standard flask; wash 3 times with hot distilled water. Cool the solution and make up to the mark.

A-5.2.2.2 Take 10 ml aliquot of the prepared solution (*see A-5.2.2.1*) in a 400 ml conical beaker and acidify with 1 ml of dilute nitric acid. Add enough distilled water to make up the volume to about 100 ml. Cool the beaker and contents. Add 10 ml of standard silver nitrate solution and back titrate against standard ammonium thiocyanate solution to reddish brown end point using 5 to 10 drops of ferric alum indicator. Take a blank reading by adding 10 ml silver nitrate to about 100 ml of water and back titrating against ammonium thiocyanate solution using ferric alum as indicator.

A-5.2.3 Calculation

$$\text{Water soluble chlorides (as Cl), percent by mass} = (V_1 - V_2) \times 10 \times 0.001775 \times 100$$

where

 V_1 = volume in ml, of standard ammonium thiocyanate solution required for the blank; and V_2 = Volume in ml, of standard ammonium thiocyanate solution required for titration.**A-6 WATER SOLUBLE SULPHATES****A-6.1 General**

Two methods are prescribed for determining sulphates, namely, Method A and ion chromatography method as prescribed in **A-15**. In case of dispute, ion chromatography method shall be used as referee method.

A-6.2 Method A**A-6.2.1 Reagents****A-6.2.1.1 Dilute hydrochloric acid** — 1:1**A-6.2.1.2 Barium chloride solution** — 10 percent (m/v)**A-6.2.2 Procedure**

Take 10 ml of the prepared solution (*see A-5.2.2.1*) in a 400 ml beaker. Acidify with 1 ml to 2 ml of dilute hydrochloric acid and heat to boiling. Add 10 ml of hot barium chloride solution drop by drop until all the sulphate is precipitated. Digest on a water bath until the precipitate has settled or preferably keep overnight, filter through a filter paper (Whatman No. 44 or its equivalent) and wash with hot distilled water until washings are free from chlorides. Ignite the precipitate in a tared platinum crucible at 900 °C for 30 min, cool in a desiccator and weigh.

A-6.2.3 Calculation

$$\text{Water soluble, sulphate, percent by mass} = M \times 343.34$$

where

M = mass in g, of ignited residue.

A-7 SODIUM AND ITS COMPOUNDS

A-7.1 Apparatus

A-7.1.1 Flame Photometer

A-7.2 Reagents

A-7.2.1 Concentrated Nitric Acid — see IS 264

A-7.3 Procedure

Weigh 0.5 g of the sample into a 250 ml glass dish previously washed with dilute nitric acid. Add 10 ml of water and 10 ml of concentrated nitric acid and 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. 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. Flame the diluted solution using a flame photometer. The instrument shall be calibrated in the range of 0 to 5 mg/l of sodium.

A-7.4 Calculation

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

where

X = mg/l of Na₂O from the calibration curve.

A-8 LOSS ON IGNITION

A-8.1 Apparatus

A-8.1.1 Platinum Crucible

A-8.1.2 Muffle Furnace

A-8.2 Procedure

Transfer the sample obtained from moisture determination (see A-2.2) and heat at 250°C in a clean and dry platinum crucible, and weigh. Keep the crucible and its contents on a hot plate for 10 min. Heat further at 550 °C for 5 min and finally at 1 000 °C for 30 min. Cool, weigh to constant mass and calculate the percent loss on ignition.

A-8.3 Calculation

Loss on ignition, percent by mass = $\frac{M_2}{M} \times 100$

where

M_2 = mass in g, of the residue; and

M = mass in g, of the regenerated material at 250 °C.

A-9 ADSORPTION CAPACITY

A-9.1 Apparatus

A-9.1.1 Dial Hygrometer

A-9.1.2 Desiccator

A-9.2 Reagents

A-9.2.1 Saturated Solution of Sodium Nitrite

A-9.3 Procedure

Take about 5 g of the material in a previously weighed weighing bottle. Regenerate the sample by placing it in a furnace at 250 °C for about 2 h with a purge of dry air or nitrogen. Remove the sample and quickly stopper it and cool to room temperature in a desiccator, without any desiccant. Weigh the sample bottle with the lid. Place the sample with lid removed along with a dial hygrometer in a desiccator, which is partly filled at its bottom with saturated aqueous solution of sodium nitrite. Evacuate the desiccator for about 15 min. Allow the sample to equilibrate for about 12 h. Release vacuum and weigh the sample bottle with the lid.

NOTE — Saturated solution of sodium nitrite at 30 °C gives a relative humidity of 60 percent. Evacuation of the desiccator will help in the attainment of faster and reproducible equilibrium conditions. It will be more appropriate to place the desiccator at a constant temperature.

A-9.4 Calculation

$$\text{Adsorption capacity, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g, of the water adsorbed; and

M = mass in g, of the regenerated material.

A-10 PORE VOLUME

A-10.1 Apparatus

A-10.1.1 Vacuum Desiccant (without any Desiccant)

A-10.2 Procedure

Take about 10 g of the material in a previously weighed weighing bottle. Regenerate the sample by placing it in a furnace at 250 °C for about 2 h with purge of dry air or nitrogen. Remove the sample and quickly stopper it. Cool to room temperature in a dry desiccator without any desiccant. Weigh the sample bottle with the lid. Transfer the sample into a wire mesh bucket. Place the wire bucket containing the sample in a vacuum desiccator and evacuate for about 15 to 20 min. Then add water till the sample is completely dipped into water. Further evacuate for 30 min. Release vacuum. Take out the sample from wire mesh bucket. Remove all superficial water with moist muslin cloth. Transfer the sample to original weighing bottle and weigh the sample bottle with lid.

NOTE — It will be more appropriate to place the desiccator at a constant temperature.

A-10.3 Calculation

$$\text{Pore Volume, ml/g} = \frac{M_1}{M}$$

where

M_1 = mass in g, of water adsorbed; and

M = mass in g, of the regenerated material.

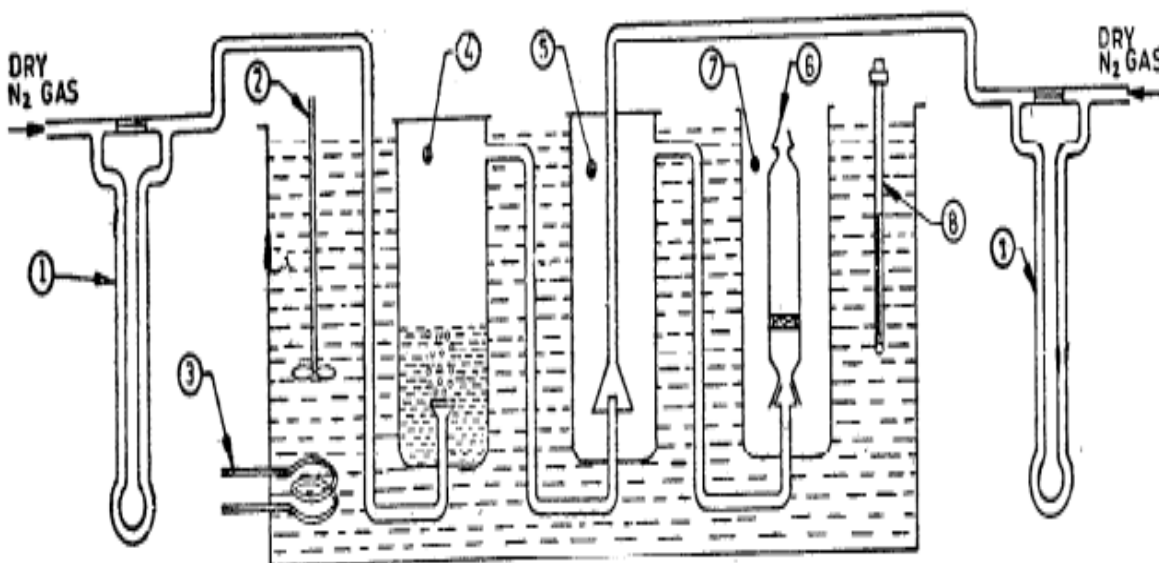
A-11 SURFACE AREA

A-11.1 Apparatus

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

A-11.2 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 mm 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 h. Note four readings in the partial pressure range of 0.05 mm to 0.35 mm of Hg.



- | | |
|----------------------|-----------------------|
| 1. Manometers | 5. Dilution chamber |
| 2. Stirrer | 6. Sample cell |
| 3. Heating Coil | 7. Adsorption Chamber |
| 4. Benzene Saturator | 8. Thermometer |

FIG. 1 APPARATUS FOR THE SURFACE AREA MEASUREMENT

A-11.3 Calculation

BET equation is

$$\frac{P/P_0}{g(1-P/P_0)} = \frac{1}{g_m C} + \frac{C-1}{g_m C} P/P_0$$

where

g = amount of benzene adsorbed at any P/P_0 ,

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

C = constant.

If $\frac{P/P_0}{g(1-P/P_0)}$ is plotted against P/P_0 , a straight line (BET) plot is obtained in which

$$\text{Slope} = \frac{C-1}{g_m C} \text{ and Intercept} = \frac{1}{g_m C}$$

$$\text{Slope} + \text{intercept} = \frac{1}{g_m C} + \frac{C-1}{g_m C} = \frac{1}{g_m}$$

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

The constant $C = \frac{\text{Slope}+1}{\text{Intercept}}$, is a measure of the difference in heats of adsorption of the first layer and the succeeding layers.

The specific surface area is given by:

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

where,

$$N = \text{Avogadro's No.} = 6.023 \times 10^{23}$$

B = molecular area of the absorbate (benzene),

$$= 41 \text{ \AA}^2 = 41 \times 10^{-20} \text{ m}^2,$$

m = mass of the sample, and

M = molecular mass of benzene (78).

On substitution, we get,

$$S = \frac{g_m \times 3 \ 166}{m}$$

A-12 BULK DENSITY

A-12.1 Procedure

Take the material as received and fill it in a 250 ml measuring cylinder up to 50-ml mark without knocking. Shake the sample down by gently lifting the cylinder as far as 5 cm from the base pad and releasing it on the table. 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 $(250 \pm 5)^\circ\text{C}$ for 3 h. Place the cylinder in a desiccator, cool and weigh to constant mass.

A-12.2 Calculation

$$\text{Bulk density, g/ml} = 0.004 M$$

where

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

A-13 ATTRITION LOSS

A-13.1 Attrition Loss by Tumbling

A-13.1.1 Apparatus

A-13.1.1.1 Sieve

850 micron IS sieve [see IS 460 (Part 1)].

A-13.1.2 Procedure

Weigh accurately 100 g of the material, exposed overnight to a constant atmosphere of 95 percent relative humidity and tumble it in plastic jar (12 cm height \times 10 cm diameter) fitted with an edge at 60 rpm for 45 min. Then measure the surface dust by sieving through 850-micron IS sieve and express as percent dust.

A-13.2 Attribution Loss by Rotation

A-13.2.1 Apparatus

A-13.2.1.1 Sieve

850-micron IS sieve [see IS 460 (Part 1)].

A-13.2.2 Procedure

Weigh accurately 100 g of the material, exposed overnight to a constant atmosphere of 95 percent relative humidity and sieved through 850-micron IS sieve. Place 50 g of the material into two steel cylinders of 3.8 cm diameter and

30.5 cm length. Clamp the cylinders at a distance of 7.6 cm from the axis of rotation and rotate at 30 rpm for 1 h. Sieve the material from both the cylinders through 850 micron IS sieve and express the dust collected as percent attribution loss.

A-14 BED CRUSHING STRENGTH

A-14.1 General

Bed crushing strength is a measurement of the amount of fines formed on crushing the material under pressure.

A-14.2 Apparatus

A-14.2.1 Cylinder

150 ml capacity having inner diameter 45 mm, closed at its bottom and fitted with a plunger.

A-14.2.2 Press

1.5 tonne fitted with a pressure gauge.

A-14.2.3 Sieve

850 micron IS sieve [see IS 460 (Part 1)].

A-14.2.4 Graduated Cylinder

200 ml capacity.

A-14.3 Procedure

Measure 120 ml of the material sifted out of the fines through 850-micron IS sieve. Weigh the material. Place the material in the cylinder, insert the plunger and press slowly to 1.1 MT (equivalent to 70 kg per cm²). Hold the pressure for about 30 seconds and release slowly. Pour carefully contents of the cylinder on to the same sieve and sift out the fines. Weigh any material retained on the sieve.

A-14.4 Calculation

$$\text{Bed crushing strength, percent by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g, of the material retained on the sieve; and

M = mass in g, of the material taken for the test.

A-15 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-15.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as chlorides and sulphates, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the pH of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-15.2 Equipment

A-15.2.1 Anion Guard Column — A protector of the separator column.

A-15.2.2 Anion Separator Column — Suitable for selective separation of ions under analysis.

A-15.2.3 Anion Suppressor Device — Anion micro membrane suppressor is used to analyse the data.

A-15.2.4 Detector — Conductivity Detector.

A-15.2.5 Software — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

A-15.2.6 Sample loop of 100 µl, 200 µl, 500 µl or 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

A-15.3 Reagents

A-15.3.1 Glass or Polyethylene Sample Bottles.

A-15.3.2 Distilled Water or Deionized Water free from the Anions of interest.

A-15.3.3 Eluent — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used. For preparation of these solution, 0.285 6 g of sodium bicarbonate and 0.381 6 g of sodium carbonate is dissolved in 2 litres of water.

A-15.3.4 Micro Membrane Suppressor Solution: (0.025 N of sulphuric acid) — Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

A-15.4 Standard Solutions

A-15.4.1 Chloride — Dissolve NaCl, 1.648 5 g in 1 000 ml of reagent water.

A-15.4.2 Sulphate — Dissolve 1.81 g of potassium sulphate in 1 000 ml of reagent water.

A-15.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1 ml to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-15.6 Procedure

Dissolve between 1g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

Add reagent water in an amount equal to 10 times the dry weight of the sample. The slurry is made and stirred for about 10 min using magnetic stirrer. This slurry is filtered from 0.45 µ membrane. This membrane can be directly attached to the end of the syringe. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-15.7 Data analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards.

ANNEX B

(Clause 6)

SAMPLING AND CRITERIA FOR CONFORMITY OF ACTIVATED ALUMINA

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 The sampled material shall be kept in a protected place and shall not be exposed to damp air.

B-1.2 The samples shall be placed in suitable a lot. Containers and each container shall be marked with full details of sampling giving the date sampling; type of material, batch number, if any; and indication of the source of manufacture.

B-2 SCALE OF SAMPLING

B-2.1 All the containers in a single consignment of the material of same grade drawn from a single batch of manufacture shall constitute a lot.

B-2.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.3 The number of containers to be chosen from the lot shall depend on the size of the lot N shall be as given in Table 2.

Table 2 Scale of Sampling

(Clause B-2.3)

Lot Size	Number of Containers to be Selected
N	n
(1)	(2)
up to 15	3
16 to 50	5
51 to 100	8
101 and above	13

B-2.4 The containers to be selected for sampling shall be chosen at random -from the lot. For this purpose a random number table shall be used (*see* 1S 4905). In case such a table is not available, the following procedure shall be adopted:

‘Starting from any container, count them as 123,) r , and so on in a systematic manner, where r is the integral part of N/n . Every r th container thus counted shall be withdrawn to constitute the required sample’.

B-3 PREPARATION OF SAMPLE

B-3.1 From each of the containers selected, draw approximately equal quantity of material with the help of a suitable sampling implement. he material drawn from different containers shall be mixed thoroughly to give a composite sample which shall be sufficient to make triplicate determination for all the characteristics given in the specification.

B-3.2 The composite sample shall then be divided into three parts, one for the purchaser another for the supplier and the third for the referee. These parts shall be transferred to separate containers which shall be suitably closed and marked with all the details of sampling.

B-3.3 The referee sample bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two. It shall be used in case of a dispute between the two.

B-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-4.1 Tests for all the characteristics given in **4** shall be conducted on the composite sample.

B-4.2 The lot shall be considered as conforming to the requirements of this specification if all the test results on the composite sample satisfy the corresponding requirements given in **4**.