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IS 10896 (1984): Aluminium hydroxide, technical [CHD 1: Inorganic Chemicals]



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IS : 10896 - 1984

Indian Standard

SPECIFICATION FOR
ALUMINIUM HYDROXIDE, TECHNICAL

UDC 661.862.23



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR ALUMINIUM HYDROXIDE, TECHNICAL

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Indian Standard

SPECIFICATION FOR ALUMINIUM HYDROXIDE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 19 March 1984, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Aluminium hydroxide of commerce is alumina trihydrate formulated as $Al_2O_3 \cdot 3H_2O$ or $Al(OH)_3$ and is a white crystalline powder prepared from bauxite by caustic digestion by well known Bayer's process. This is normally the technical grade of aluminium hydroxide, which is converted to Al_2O_3 by calcination for use in aluminium metallurgy by the electrolytic process.

0.3 Besides the above major use, it has various other applications, namely, in the manufacture of iron-free aluminium sulphate, sodium aluminate and aluminium salts; in the manufacture of activated alumina and various alumina — silica catalysts used in petroleum cracking processes; in the manufacture of glass and ceramics; used as a base for organic lakes — dyeing processes and in the finely divided form, this is used as a filter or reinforcing agent in rubber, paper coatings and cosmetics.

0.4 The requirements for pharmaceutical grade of the material are not included in this standard as these requirements are covered in the Indian Pharmacopoeia.

0.5 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*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for aluminium hydroxide, technical,

*Rules for rounding off numerical values (revised).

2. REQUIREMENTS

2.1 Description — Aluminium hydroxide, technical shall be in the form of white, light, amorphous powder and gritty to the touch. It shall be odourless and tasteless.

2.2 The material shall comply with the requirements laid down in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR ALUMINIUM HYDROXIDE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay (as Al_2O_3), percent by mass, <i>Min</i>	65.0	A-2
ii)	Silica (as SiO_2), percent by mass, <i>Max</i>	0.12	A-3
iii)	Iron (as Fe_2O_3), percent by mass, <i>Max</i>	0.03	A-4
iv)	Sodium (as Na_2O), percent by mass	0.3-0.5	A-5
v)	Loss on ignition, percent by mass	34.5-35.0	A-6
vi)	Arsenic (as As_2O_3), parts per million, <i>Max</i>	1.0	A-7
vii)	Heavy metals (as Pb), parts per million, <i>Max</i>	5.0	A-8
viii)	pH of 4.0 percent suspension, <i>Max</i>	10.0	A-9
ix)	Free moisture, percent by mass	4.0—8.0	A-10

NOTE — The characteristics tested in (i) to (viii) are on dry basis of the chemical.

3. PACKING AND MARKING

3.1 The material shall be packed in air tight polyethylene lined mild steel containers.

3.2 Each package shall be securely closed and marked with the following information:

- a) Name and grade (technical) of the material;
- b) Net mass of the contents;
- c) Name of the manufacturer and his recognized trade-mark, if any;
- d) Date of manufacture; and
- e) Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as described in Appendix B.

A P P E N D I X A

(Clause 2.2)

METHODS OF TEST FOR ALUMINIUM HYDROXIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

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

A-2. ASSAY

A-2.1 Reagents

A-2.1.1 *Concentrated Hydrochloric Acid* — See IS : 265-1966†.

A-2.1.2 *Methyl Red Indicator Solution* — Dissolve 0.15 g of methyl red in 500 ml of water.

A-2.1.3 *Dilute Ammonium Hydroxide* — 1 : 1 (v/v).

A-2.2 *Procedure* — Weigh accurately about 0.2 g of the material and dissolve in 5 ml of hydrochloric acid, and add 50 ml of water. Heat to

*Specification for water for general laboratory use (second revision).

†Specification for hydrochloric acid (second revision).

boiling, filter through Whatman No. 40 and wash the filter with 50 ml of water; add 2 or 3 drops of solution of methyl red, followed by sufficient dilute ammonia solution to produce a distinct yellow colour in the mixture. Heat to boiling and filter. Wash with ammonium nitrate solution until the precipitate is free from chloride. Dry the precipitate to constant mass at a temperature above 1200°C and weigh the residue of Al_2O_3 .

A-2.3 Calculation

$$\text{Aluminium oxide (as } Al_2O_3 \text{), percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for the test.

A-3. SILICA

A-3.1 Apparatus

A-3.1.1 *125-ml Platinum Crucible*

A-3.1.2 *Hot Water Bath*

A-3.1.3 *Polyethylene Bottles — 125-ml capacity.*

A-3.1.4 *pH Meter-Buffered to pH 4.0*

A-3.1.5 *Bechman Model B Spectrophotometer, Red Phototube, 745 Wavelength*

A-3.1.6 *Volumetric Flasks—100 and 250 ml.*

A-3.1.7 *Burettes and/or Pipettes for Delivering 2, 4, 5 and 50 ml Volumes*

A-3.2 Reagents

A-3.2.1 *Boric Acid*

A-3.2.2 *Sodium Carbonate, Anhydrous — Reagent grade (see IS : 296 - 1974*).*

A-3.2.3 *Dilute Sulphuric Acid — Approximately 10 N. Add 278 ml concentrated sulphuric acid to 700 ml water, cool and dilute to 1 litre.*

A-3.2.4 *Molybdate Reagent — 18.6 g reagent grade of ammonium molybdate ($NH_4 Mo_7O_{24} \cdot 4H_2O$) dissolved in water and diluted to 250 ml. This solution is stable for at least two weeks.*

*Specification for sodium carbonate, anhydrous.

A-3.2.5 Stannous Chloride Stock Solution — 10 g reagent grade $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ dissolved in 10 ml concentrated HCl . This solution is stable for at least a month if stored in polyethylene bottle and kept in the dark.

A-3.2.6 Stannous Chloride Working Solution — 2 ml stock solution is diluted to 100 ml made afresh when needed.

A-3.2.7 Standard Silica Solution — Ignite reagent grade silicic acid that has been tested for impurities by HF evolution. Fuse 0.2500 g of the ignited silicic acid with 2 g Na_2CO_3 at 1000°C for 30 minutes in platinum crucible. Dissolve the melt with water. Carefully add 5.8 ml of 10 NH_4SO_4 and dilute to 1000 ml. This solution is stable for at least six months.

A-3.3 Preparation of Standard Curve

A-3.3.1 Pipette 20 ml of the standard silica solution into a 250 ml volumetric flask.

A-3.3.2 Take 0, 5, 10, 15, 20 and 25 ml aliquots into polyethylene bottles. These aliquots will contain 0, 0.1, 0.2, 0.3, 0.4, 0.5 mg of SiO_2 , respectively.

A-3.3.3 Add sufficient water to each to make the total volume approximately 100 ml.

A-3.3.4 Adjust the pH to 1.25 ± 0.05 and add 4 ml of molybdate reagent without removing the electrodes. Recheck and adjust the pH if necessary (see Note 1 under A-3.3.10).

A-3.3.5 Transfer the sample quantitatively with washing to a polyethylene bottle and heat in a gently boiling water bath for 45 to 180 minutes. Remove the bottles and cool (see Note 2 under A-3.3.10).

A-3.3.6 Transfer the sample to a 250-ml volumetric flask which contains 50 ml of 10N H_2SO_4 , and swirl to mix.

A-3.3.7 Dilute to about 240 ml with wash water (see Note 3 under A-3.3.10).

A-3.3.8 Add 5 ml of the stannous chloride working solution with agitation, dilute to volume, and mix (see Note 4 under A-3.3.10).

A-3.3.9 Determine the absorbance (optical density) of the standard solutions at 745 nm with the meter adjusted to zero absorbance (100 percent transmittance with the blank in the reference cell (see Note 5 under A-3.3.10)).

A-3.3.10 The mg SiO₂ is plotted against the absorbancies on a square graph paper.

NOTE 1 — The permissible range is pH 1.0 to 1.5, but it has been restricted as a safeguard against instrument or operator error.

NOTE 2 — If the bottles are wired or tied together in bundles of three or more, they will not tip over. Do not boil the water violently as it will splash into the bottles. They should not be capped because of the pressure that would build up if they were.

NOTE 3 — Do not allow the sample to stand for more than 30 minutes at this point before proceeding to A-3.3.8.

NOTE 4 — Do not allow the sample to stand for more than two hours before reading.

NOTE 5 — Proper cell corrections must be made as needed.

A-3.4 Procedure

A-3.4.1 Accurately weigh 2.500 g of sample ± 0.000 1 g and place in a clean, dry 125 ml platinum crucible. Weigh 7.50 ± 0.01 g of Na₂CO₃ and 5.0 ± 0.01 g of H₃BO₃ and place in crucible with sample. Mix sample and flux thoroughly. Prepare a fusion blank of flux only (see Note 1 under A-3.4.2). Fuse at 1 000°C for 30 minutes (see Note 2 under A-3.4.2). Remove the crucible and roll it to distribute the melt. When crucible is no longer red, quench it in cold water to crack the melt.

A-3.4.2 Add 75 ml of water and place on a preheated hot plate immediately. Adjust the hot plate so as to simmer the sample as it dissolves. Transfer the hot solution to a polyethylene beaker with washing. Immediately adjust the pH to about 1.5 taking care to avoid loss of sample through violent CO₂ evolution (see Notes 3 and 4 under A-3.4.2). When the sample has redissolved, transfer the acidified solution to a 250-ml volumetric flask, cool and make to volume (see Note 5 under A-3.4.2). Pipette 100 ml aliquots of sample and blank into polyethylene bottles. Proceed as in A-3.3.4 to A-3.3.9 for the preparation of curve. Read optical density with the flux blank in the reference cell.

NOTE 1 — A carefully premixed flux in the ratio, of three parts Na₂CO₃ to 2 parts H₃BO₃, may be used (12.5 g per 2.5 g alumina).

NOTE 2 — Heat slowly to avoid 'boiling' of sample. Do not put directly into 1 000°C furnace.

NOTE 3 — A delay after fusion and before addition of water can result in precipitation of alumina which is difficult to dissolve. A delay after removal from the hot plate and before acidification can also result in precipitation of alumina.

NOTE 4 — In A-3.4.2 the solution is acidified to about pH 1.5, that is, acid enough to prevent alumina precipitation but not below pH 1.25. There is probably no harm

in going lower except that the pH adjustment is more awkward. It has been found that the addition of 33 ml and 14 ml of 10 NH_4SO_4 , respectively to the sample and blank will bring the pH to the proper range if care is taken in weighing flux.

NOTE 5 — No measurable increase in silica results if the acidified sample stands in glass for a few hours; but if overnight delays occur, it is probably best to leave the same in polyethylene.

A-3.5 Calculation

$$\text{Silica (as SiO}_2 \text{), percent by mass} = \frac{X}{4M}$$

where

X = mg/l of SiO_2 from the calibration curve, and

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

A-4. IRON

A-4.1 Apparatus

A-4.1.1 *Platinum Crucible with Cover* — 125 ml.

A-4.1.2 *Muffle Furnace* — 950°C to 1 025°C.

A-4.1.3 *pH Meter*

A-4.1.4 *Spectrophotometer*

A-4.2 Reagents

A-4.2.1 *Hydroxylamine Hydrochloride, 10 percent* — Dissolve 100 g of hydroxylamine hydrochloride in about 500 ml of water. Filter the solution through a tight filter paper into a 1 000 ml volumetric flask and dilute to volume with water.

A-4.2.2 *Ortho Phenanthroline* — 0.2 percent. Dissolve 0.50 g of 1 : 10 ortho-phenanthroline monohydrate in about 100 ml water, warming slightly if necessary up to 80°C. Cool and dilute to 250 ml with water.

A-4.2.3 *Dilute Sulphuric Acid* — 1 : 3 (v/v).

A-4.2.4 *Ammonium Hydroxide* — Concentrated.

A-4.2.5 *Iron Wire* — Reagent grade.

A-4.3 *Procedure* — The sample is fused and prepared as directed in SiO_2 estimation (A-3.4). Pipette a 50 ml aliquot from the 250 ml volume of stock solution into a 150-ml beaker. Carefully adjust the pH to 2.4-2.5 on the pH meter, using 1 : 3 sulphuric acid or NH_4OH as necessary. Carefully transfer the sample into a clean 100 ml volumetric flask. Wash

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the beaker with two or three small portions of wash water and add each washing to the flask. Add 2.0 ml by pipette of 10 percent hydroxylamine hydrochloride to the sample and blank. Mix well. Let the solution stand for 5 minutes. Add, by pipette, 5.0 ml of ortho-phenanthroline solution and mix well. Dilute each flask to volume and mix well. After 30 minutes, determine the optical density (OD) of the sample, using the blank to zero the spectrophotometer, and wave length setting at 508 nm.

A-4.4 Calibration Curve.— Prepare a standard iron solution containing 0.01 mg of Fe_2O_3 per ml by dissolving iron wire, reagent grade, in hydrochloric acid. Add the following volumes of standard Fe_2O_3 solution to 150 ml beakers :

<i>ml 0.01 mg of Fe_2O_3/ml</i>	<i>mg Fe_2O_3</i>
10	0.1
20	0.2
30	0.3
40	0.4

Adjust the volume in each beaker to approximately 50 ml. Prepare a blank with 50 ml water. Carry the blank through each step with the standards. Adjust the pH of each standard and blank to 2.4, 2.5 with 1 : 3 H_2SO_4 or ammonia, as required. Carefully transfer the contents of each beaker into clean 100-ml volumetric flasks. Proceed as in A-4.3. Plot OD against mg Fe_2O_3 per 100 ml on a graph paper.

A-5. SODIUM

A-5.1 Apparatus

A-5.1.1 Flame Photometer

A-5.1.2 Polyethylene Bottles

A-5.2 Reagents

A-5.2.1 Dilute Sulphuric Acid — 50 percent (v/v).

A-5.2.2 Sodium Oxalate

A-5.2.3 Standard Sodium Solution

A-5.2.3.1 Na_2O Primary stock solution — Dissolve 2.1616 g of sodium oxalate in water and make up to 1 litre. 1 ml of this solution contains 1 mg of Na_2O .

A-5.2.3.2 Working standard solution — Transfer 100 ml of stock solution to a 500 ml volumetric flask and make up the volume with water. 1 ml

of this solution contains 0.2 mg of Na_2O . To several 500 ml volumetric flasks add the following and make up the volume with water :

Flask No.	Volume of Working Standard Solution in ml	Volume of Dilute Sulphuric Acid in ml	Na_2O in the Final Volume in mg
1	0	50	0
2	5	50	1
3	10	50	2
4	15	50	3
5	25	50	5
6	35	50	7
7	50	50	10

Transfer the resulting solutions in polyethylene bottles.

A-5.3 Procedure — Weigh 0.5 g of the sample. Transfer it to a 250-ml beaker. Add 50 ml sulphuric acid in the beaker. Cover with a watch glass. Heat gently till all the salts are dissolved. Cool slightly and transfer to a clean 500-ml volumetric flask. Again cool, make up the volume with water. Take the percent transmittance reading of the standard solutions and the sample on a flame photometer. Plot a curve taking percent transmittance on the Y-axis and mg Na_2O on the X-axis. Determine the mg Na_2O of the sample from the curve.

A-5.4 Calculation

$$\text{Sodium (as } \text{Na}_2\text{O), percent by mass} = \frac{\text{mg } \text{Na}_2\text{O}}{10 \times M}$$

where

M = mass in g of the sample taken for the test.

A-6. LOSS ON IGNITION

A-6.1 Apparatus

A-6.1.1 Platinum Crucible

A-6.2 Procedure — Take 1 g of the material in a clean, dry platinum crucible. Keep the crucible and its contents on a hot plate for 10 minutes. Heat further at 550°C for 5 minutes and finally at 1 100°C for 30 minutes. Cool, weigh and calculate the percent loss on ignition.

A-7. ARSENIC

A-7.1 The sample is fused and prepared as directed in A-3.4. Then proceed as per the method given in IS : 2088-1971*.

*Method for determination of arsenic (*first revision*).

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A-8. HEAVY METALS

A-8.1 The sample is fused and prepared as directed in A-3.4. Then proceed as directed in IS : 7017-1973*.

A-9. pH VALUE

A-9.1 Apparatus

A-9.1.1 pH Meter

A-9.2 Procedure — Prepare an aqueous extract of the material by shaking for 1 hour, 2 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-10. FREE MOISTURE

A-10.1 Apparatus

A-10.1.1 Glass Weighing Dish — Flat bottomed 20-ml capacity and preferably of following dimensions :

Internal diameter	44 mm
Height	25 mm

A-10.2 Procedure — Weigh accurately 10 to 12 g of the sample in a covered glass weighing dish. Remove the cover and heat the bottle and its contents in an unventilated oven at $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 3 hours. Replace the cover, cool in a desiccator and weigh to constant mass. Use this dried material for the absorption capacity.

A-10.3 Calculation

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

where

M_1 = mass in g of the sample before heating, and

M_2 = mass in g of the sample after heating.

*Method for colorimetric determination of traces of heavy metals by dithizone.

APPENDIX B

(Clause 4.1)

SAMPLING OF ALUMINIUM HYDROXIDE, TECHNICAL

B-1. SCALE OF SAMPLING

B-1.1 Lot — In any consignment, all the containers of the same size drawn from the same batch of manufacture, shall be grouped together to constitute a lot.

B-1.2 For ascertaining the conformity of the material in the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

NUMBER OF CONTAINERS IN THE LOT	NUMBER OF CONTAINERS TO BE SELECTED
N (1)	n (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 300	7
301 and above	10

B-1.3 These containers shall be selected at random (see IS : 4905-1968*) from the lot. To ensure randomness of selection, a random number table may be used. In case such a table is not available, the following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3,....., etc, up to r and so on, in one order. Every r th container thus counted shall be withdrawn to constitute the sample, where r is the integral part of N/n (N being the number of containers in the lot and n being the number of containers to be selected).

B-2. PREPARATION OF SAMPLES

B-2.1 From different parts of each selected container, a small but approximately equal quantity of material shall be drawn and thoroughly mixed

*Methods for random sampling.

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together to constitute a composite sample weighing not less than 150 g. The total material so obtained shall be divided into three approximately equal parts each of which shall be called a composite sample representing the lot.

B-2.2 Each of the three composite samples obtained in **B-2.1** shall be immediately transferred to appropriate sample containers which shall be sealed air-tight immediately after filling and marked with necessary details for identification.

B-2.3 One of three composite samples shall be marked for the purchaser another for supplier and the third kept as a referee sample.

B-2.4 The referee sample shall be kept at a place and under conditions agreed to between the purchaser and the supplier. The referee sample shall be used in the case of dispute.

B-3. NUMBER OF TESTS AND CRITERION FOR CONFORMITY

B-3.1 Tests for all the requirements of this specification shall be conducted on the composite sample.

B-3.2 The lot shall be declared as conforming to the requirements of the specification if the composite sample satisfies all the tests in **B-3.1**.