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मानक

IS 6015 (1984): Barium hydroxide [CHD 1: Inorganic Chemicals]



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IS : 6015 - 1984

Indian Standard SPECIFICATION FOR BARIUM HYDROXIDE (First Revision)

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

July 1984

Indian Standard

SPECIFICATION FOR BARIUM HYDROXIDE

(First Revision)

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Indian Standard

SPECIFICATION FOR BARIUM HYDROXIDE

(First Revision)

$\mathbf{0.} \mathbf{FOREWORD}$

0.1 This Indian Standard (First Revision) 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 This standard was originally published in 1970. It has been revised in the light of recent developments and experience gained by the industry.

0.3 Taking into consideration the views of producers, consumers and technologists, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the manufacturing and trade practices followed in the country in this field. Indigenous samples of both the technical and analytical reagent grades of the material being manufactured in the country, had been analysed and this revised standard has been based on the data thus collected.

0.4 Barium hydroxide, octahydrate, known in the trade as barium hydrate, barium octahydrate, caustic baryta, is one of the most important barium chemicals excluding those which are used for pigments. It is used in sugar industry, refining animal and vegetable oils and fats, softening of water, manufacture of glass, fresco painting, as boiler scale remedy and in an analytical chemistry.

0.5 Flame photometric methods have been prescribed for the determination of calcium and strontium. Since widely differing types of instruments are available, detailed instructions have not been given.

06 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.

*Rules for rounding off numerical values (revised).

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for barium hydroxide.

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2. GRADES

2.1 There shall be two grades of the material, namely:

- a) Technical (TECH), and
- b) Analytical Reagent (AR)

3. REQUIREMENTS

3.1 Description — The material shall be in the form of colourless crystals or white mass, free from extraneous impurities.

3.2 The material shall comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

	(Clauses 3.2 and	4.2.1)			
Sl No	CHARACTERISTIC .	REQUIRI FOR GI	EMENT RADE	METHOD OF TEST (REF TO CL NO.	
		TECH	AR	IN APPENDIX A)	
(1)	(2)	(3)	(4)	(5)	
i)	Assay [as Ba(OH) ₂ 8H ₂ O], percent by mass, Min	95.0	98·0	A-2	
ii)	Barium carbonate (as BaCO ₃), percent by mass, Max	2.2	2.0	A-3	
iii)	Matter insoluble in dilute hydro- chloric acid, percent by mass, Max	0.04	0.002	A-4	
iv)	Chlorides (as Cl), percent by mass, Max	0 .06	0.005	A-5	
v)	Sulphides	To pass tes	st Topa	ass test A-6	
vi)	Heavy metals (as Pb), percent by mass, Max		0.002	A-7	
vii)	Iron (as Fe), percent by mass, Max	0.003	0.0002	A-8	
viii)	Calcium (as Ca), percent by mass, Max		0.002	A-9	
ix)	Strontium (as Sr), percent by mass, Max		0.2	A-10	
x)	Substances not precipitated by sulphu- ric acid, percent by mass, Max	0.60	0 ·10	A-11	

TABLE 1 REQUIREMENTS FOR BARIUM HYDROXIDE

4. PACKING AND MARKING

4.1 Packing — The material of technical grade shall be packed in mild steel or fibre drums lined with polyethylene. The material of analytical reagent grade shall be packed in airtight glass bottles.

NOTE — Barium hydroxide rapidly absorbs carbon dioxide from air, becoming incompletely soluble in water. The containers should, therefore, be kept tightly closed.

4.2 Marking — The containers shall be legibly and indelibly marked with the following information:

- a) Name and grade of the material;
- b) Net mass of the contents;
- c) Year of manufacture;
- d) Manufacturer's name and/or recognized trade-mark, if any; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

4.2.1 In case of AR grade of the material, complete chemical analysis in respect of the characteristics specified in Table 1 shall also appear on the label.

4.2.2 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.

5. SAMPLING

5.1 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 Appendix B.

$\begin{array}{c} \mathbf{A} \, \mathbf{P} \, \mathbf{P} \, \mathbf{E} \, \mathbf{N} \, \mathbf{D} \, \mathbf{I} \, \mathbf{X} \\ (\, Clause \, 3.2 \,) \end{array}$

METHODS OF TEST FOR BARIUM HYDROXIDE

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 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenol phthalein in 100 ml of 60 percent rectified spirit.

A-2.1.2 Standard Hydrochloric Acid — 1 N.

A-2.2 Procedure — Weigh accurately 4 to 5 g of the material and dissolve it in about 200 ml of carbon dioxide-free water. Add 0.1 ml of phenol-phthalein indicator solution and titrate with standard hydrochloric acid. **Preserve** this solution for the determination of barium carbonate in A-3.

A-2.3 Calculation — Calculate as follows on the basis that 1 ml of 1 N hydrochloric acid is equivalent to 0.1577 g of barium hydroxide:

Barium hydroxide [as Ba (OH)₂ 8H₂O], percent by mass = $\frac{15.77 V N}{M}$

where

V = Volume in ml of standard hydrochloric acid required,

N = normality of standard hydrochloric acid, and

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

A-3. DETERMINATION OF BARIUM CARBONATE

A-3.1 Reagents

A-3.1.1 Standard Hydrochloric Acid – 1 N.

A-3.1.2 Methyl Orange Indicator Solution — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-3.1.3 Sodium Hydroxide Solution -1 N.

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

A-3.2 Procedure — To the solution preserved in A-2.2, add 5 ml of standard hydrochloric acid and heat to boiling. Boil gently to expel all the carbon dioxide and cool. Add 0.1 ml of methyl orange indicator and titrate the excess of hydrochloric acid with sodium hydroxide solution.

A-3.3 Calculation — Calculate as follows on the basis that 1 ml of 1 N hydrochloric acid consumed is equivalent to 0.098 68 g of barium carbonate:

Barium carbonate (as BaCO₃),
$$=\frac{9.868 V N}{M}$$

where

V = volume in ml of standard hydrochloric acid,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test in A-2.2.

A-4. DETERMINATION OF MATTER INSOLUBLE IN DILUTE HYDROCHLORIC ACID

A-4.1 Reagent

A-4.1.1 Dilute Hydrochloric Acid -1:9(v/v).

A-4.2 Procedure — Dissolve about 10 g of the material, accurately weighed, in 100 ml of dilute hydrochloric acid. Heat the solution to boiling and then digest in a covered beaker on steam-bath for one hour. Filter through a tared Gooch crucible or sintered glass crucible No. G 4. Wash thoroughly and dry at $105 \pm 5^{\circ}$ C. Cool and weigh till constant mass is obtained.

A-4.3 Calculation

Matter insoluble in dilute hydrochloric $= \frac{100 \times M_1}{M}$

where

 $M_1 =$ mass in g of the residue obtained, and

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

A-5. DETERMINATION OF CHLORIDES

A-5.1 For Technical Grade

A-5.1.1 Reagents

A-5.1.1.1 Standard silver nitrate solution - 0.1 N.

A-5.1.1.2 Concentrated nitric acid — see IS : 264-1976*.

A-5.1.1.3 Ferric alum indicator solution — saturated.

A-5.1.1.4 Standard ammonium thiocyanate solution – 0.1 N.

A-5.1.1.5 Nitrobenzene

*Specification for nitric acid (second revision).

A-5.1.2 Procedure — Weigh accurately about 5 g of the material and dissolve in about 80 ml of water. Filter the residue, if any, through a filter paper and wash thoroughly with water, collecting the filtrate and washings in a 250-ml conical flask. Add with a pipette 25 ml of silver nitrate solution, 2 ml of concentrated nitric acid and 10 ml of nitrobenzene. Shake vigorously and add 2 ml of ferric alum indicator solution. Titrate the solution with standard ammonium thiocyanate solution to the first persistent colour change. Carry out a blank titration using the same amount of the reagents and following the identical procedure.

A-5.1.3 Calculation

Chlorides (as cl), percent by mass = $\frac{3.545 (V_1 - V_2) N}{M}$

where

- V_1 = volume in ml of standard ammonium thiocyanate solution required in the blank titration,
- V_2 = volume in ml of standard ammonium thiocyanate solution required in the titration with the material,
- N =normality of standard ammonium thiocyanate solution, and
- M = mass in g of the material taken for the test.

A-5.2 For AR Grade

A-5.2.1 Apparatus

A-5.2.1.1 Nessler cylinders — 50 ml capacity.

A-5.2.2 Reagents

A-5.2.2.1 Concentrated nitric acid — See IS : 264-1976*.

A-5.2.2.2 Standard chloride solution — Dissolve 1'649 g of sodium chloride in water and make up the volume to exactly 1 000 ml. Pipette out 10 ml of the solution, dilute with water and again make up the solution to exactly 100 ml. One millilitre of this solution contains 0'1 mg of chloride (as Cl).

A-5.2.2.3 Silver nitrate solution — approximately 2 percent (m/v).

A-5.2.2.4 Phenolphthalein indicator solution — same as in A-2.1.1.

A-5.2.3 Procedure — Weigh 10°0 g of the material, dissolve in 15 ml of water, add a drop of phenolphthalein indicator and neutralize cautiously with concentrated nitric acid. If the solution is not clear, filter through a filter paper Whatman No. 40 or 42 that has been washed free of chloride and dilute the filtrate to exactly 50 ml in a volumetric flask.

^{*}Specification for nitric acid (second revision).

A-5.2.3.1 Take 25 ml aliquot in a Nessler cylinder, add 1 ml of silver nitrate solution and dilute to 50-ml mark. Carry out a control test in the other Nessler cylinder using 1 ml of the standard chloride solution and the same quantities of other reagents and finally diluting to 50-ml mark. Stir both the solutions with glass rod and compare the turbidity produced in the two cylinders after 5 minutes.

A-5.2.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-6. TEST FOR SULPHIDES

A-6.1 Reagents

A-6.1.1 Alkaline Lead Solution — To a lead acetate solution (10 percent m/v), add sodium hydroxide solution (about 1 N) until the precipitate formed gets dissolved.

A-6.1.2 Glacial Acetic Acid - See IS : 695-1975*.

A-6.2 Procedure — Dissolve 1 g of the material in 10 ml of warm water. Add 5 drops of alkaline lead solution and 2 ml of glacial acetic acid.

A-6.2.1 The material shall satisfy the requirements of the test if there is no darkening of the colour as compared with the original.

A-7. DETERMINATION OF HEAVY METALS

A-7.1 Apparatus

A-7.1.1 Nessler Cylinder — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 Concentrated Hydrochloric Acid — See IS: 265-1976[†].

A-7.2.2 Standard Lead Solution — Dissolve 160 g of lead nitrate in water and make up the volume to exactly 1000 ml. Transfer 10 ml of the solution to a volumetric flask and dilute it again with water to 1000 ml mark. One millilitre of this solution contains 001 mg of lead (as pb). The diluted solution shall be freshly prepared.

A-7.2.3 Dilute Acetic Acid — approximately 1 N.

A-7.2.4 Hydrogen Sulphide Solution — saturated.

A-7.2.5 Phenolphthalein Indicator Solution — same as in A-2.1.1.

^{*}Specification for acetic acid (second revision). †Specification for hydrochloric acid (second revision).

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A-7.3 Procedure — Weigh 2'0 g of the material and add 15 ml of water Add 5 ml of concentrated hydrochloric acid and evaporate to dryness over a water-bath. Dissolve it in 20 ml of water and transfer to a Nessler cylinder. Add one drop of phenolphthalein indicator solution and neutralize with sodium hydroxide solution. Add 1 ml of glacial acetic acid and 10 ml of hydrogen sulphide solution. In the second Nessler cylinder, carry out a control test using 1 ml of standard lead solution in place of the sample and the same quantities of other reagents carried through the same procedure. Dilute the contents of each cylinder to 50 ml and shake well. Compare the colour produced in the two cylinders.

A-7.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-8. DETERMINATION OF IRON

A-8.1 Apparatus

A-8.1.1 Nessler Cylinder — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 Concentrated Hydrochloric Acid — See IS : 265-1976*.

A-8.2.2 Ammonium Persulphate — solid.

A-8.2.3 Butanolic Potassium Thiocyanate solution — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-8.2.4 Standard Iron Solution — Dissolve 0.702 g of ferrous ammonium sulphate [Fe SO₄ (NH₄)₂ SO₄·6H₂O] in 10 ml of dilute sulphuric acid (10 percent v/v) and dilute with water to 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-8.3 Procedure — Weigh 1'0 g of the material, dissolve in 10 ml of water and transfer to a Nessler cylinder. Add 5 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Dilute to the mark with water. Shake vigorously for 30 seconds and allow the liquids to separate. Carry out a control test in the other Nessler cylinder with 5 ml of standard iron solution for technical grade of the material in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the two cylinders.

A-8.3.1 For AR grade of the material, use 10'0 g of the material and 5 ml of standard iron solution.

A-8.3.2 The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour of the butanol layer in the test with the material is not deeper than the colour produced in the control test,

*Specification for hydrochloric acid (second revision).

A-9. DETERMINATION OF CALCIUM

A-9.1 Procedure — Dissolve 1 g of the material in about 50 ml of water, add 0.05 ml of phenolphthalein indicator solution, neutralize with dilute hydrochloric acid (1:10 v/v) adding 2 ml in excess and dilute to exactly 100 ml in a volumetric flask. Determine calcium with the help of a flame photometer at 422.7 nm according to the directions of the manufacturer of the apparatus (see 0.5).

A-10. DETERMINATION OF STRONTIUM

A-10.1 Procedure — Dissolve 5 g of the material in 50 ml of dilute hydrochloric acid ($1:10 \nu/\nu$) and make up to 100 ml in a volumetric flask. Take 1 ml aliquot of the solution and dilute to exactly 100 ml in a volumetric flask. Take 1 ml aliquot of the solution and dilute exactly to 100 ml with dilute hydrochloric acid ($1:50 \nu/\nu$). Determine strontium with the help of a flame photometer at 460.7 nm according to the instructions of the manufacturer of the apparatus (see 0.5).

A-11. DETERMINATION OF SUBSTANCES NOT PRECIPITATED BY SULPHURIC ACID

A-11.1 Reagents

A-11.1.1 Concentrated Hydrochloric Acid — See IS : 265-1976*.

A-11.1.2 Dilute Sulphuric Acid — 10 percent (v/v).

A-11.2 Procedure — Dissolve 5 g of the material in 150 ml of water and 5 ml of hydrochloric acid. Heat the solution to boiling and add 25 ml of dilute sulphuric acid. Allow the solution to cool, dilute to 250 ml with water and allow to stand overnight. Decant the solution through a dry filter paper and evaporate 100 ml to dryness in a tared porcelain dish. Heat gently to volatilize the excess of acids and finally ignite at $800 \pm 25^{\circ}$ C for 15 minutes. Cool and weigh till constant mass is obtained.

A-11.3 Calculation

Substances not precipitated by sulphuric $= \frac{100 \times M_1}{M}$

where

 $M_1 = \text{mass in g of the ignited residue, and}$

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

*Specification for hydrochloric acid (second revision).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF BARIUM HYDROXIDE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling the samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken at a place exposed to the adverse effects of weather.

B-1.2 The sampling instruments and sample containers shall be clean and dry.

B-1.3 Before drawing the sample, the contents of the selected containers shall be thoroughly mixed.

B-1.4 After filling, the sample containers shall be sealed and marked with the relevant particulars.

B-1.5 In the case of AR grade, the labels on the bottles shall be examined for the completeness of details before opening and taking out samples.

B-2. SCALE OF SAMPLING

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

B-2.2 Each lot shall be tested separately for all the requirements of this specification. The number of containers to be selected at random from a lot depends on the size of the lot and shall be as given in Table 2.

TABLE 2	SCALE OF SAMPLING
Lot Size	NUMBER OF CONTAINERS TO BE SELECTED
(1)	(2)
Up to 50	3
51 to 100	4
101 to 150	5
151 and above	17

B-2.3 The containers shall be selected at random. In order to ensure the randomness of selection, procedure given in IS : 4905-1968 'Methods for random sampling', may be followed.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 Draw with an appropriate sampling instrument about 100 g of the material (barium hydroxide) from different parts of each of the selected containers. These quantities are called individual samples representing the selected containers. These individual samples shall be kept in different sample containers which shall be marked with full details of sampling.

B-3.2 Equal quantities from each of the individual samples shall be taken and mixed together to form a composite sample weighing approximately 200 g.

B-4 NUMBER OF TESTS

B-4.1 Number of Tests for Technical Grade

B-4.1.1 Tests for the determination of barium hydroxide and barium carbonate contents shall be conducted on each of the individual samples.

B-4.1.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-4.2 Number of Tests for AR Grade — In the case of AR grade, the tests for all the characteristics shall be conducted on each of the individual samples.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For individual Samples — For each of the characteristics for which individual tests have been carried out, the mean (\bar{X}) and the range (R) shall be calculated as follows:

Mean () =
$$\frac{\text{The sum of test results}}{\text{Number of test results}}$$

Pange (P) = The difference between the maximum

Range (R) = The difference between the maximum and the minimum of the test results.

The lot shall be deemed as conforming to the requirements with respect to a characteristic for which minimum limit has been prescribed in Table 1 if \overline{X} —0.6 R is greater than or equal to that limit. In case a maximum limit is specified for a characteristic, the lot shall be deemed as conforming to the requirement if \overline{X} + 0.6 R is less than or equal to that limit.

B-5.2 For Composite Sample — For the remaining characteristics, all the test results shall satisfy the corresponding requirements given in col 3 and 4 of Table 1.

(Continued from page 2)

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units			
Quantity	Unit	Symbol	
Length	matre	m	
Masa	kilogram	ko	Children in a
Time	second		
Electric current	empere	A	
Thermodynamic temperature	kelvin	R	
Luminous intensity	candala	cd	
Amount of substance	mole	mol	
Supplementary Units			
Quantity	UnA	Symbol	
Plane angle	radian	bar	
Solid angle	eteradian		
Derived Unite			
Quantity	Unit	Symbo'	Definition
Force	newton	N I	N = 1 kg.m/s*
Energy	louis	1 1	J = I N.m
Power	watt	W I	W - 1 J/a
Flux	weber	Wb II	ND - IV.
Flux density	testa	T	- I Wb/m'
Frequency	hertz	Hz I	$H_{Z} = 1 c/s (s^{-1})$
Electric conductance	slemena	S 1 !	5 - 1 A/V
Electromotive force	volt	V 11	/ - I W/A
Pressure, stress	pascal	Pa II	Pa = 1 N/m*

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Eastern : 5 Chowringhee Approach	CALCUTTA 700072	27 1	60	90	
Southern : C. I. T. Campus	MADRAS 600113	41 :	24	42	
Northern : B69, Phase VII	S.A.S. NAGAR (MOHALI) 160051	8	78	28	
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