भारतीय मानक मसौदा

रासायनिक उद्योग और तेल-कूप ड्रिलिंग के लिए बेराइट्स — विशिष्टि

(*IS 2881 का तीसरा पुनरीक्षण*)

Draft Indian Standard
Barytes for Chemical Industry and Oil-Well Drilling — Specification
(*Third Revision of IS 2881*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01	Last date of comments: xxxxxx

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1964 and subsequently revised in 1978 and 1984 in the light of the experience gathered in production and consumption patterns of the material. The second revision was undertaken mainly to align the requirements in line with the requirements of Oil and Natural Gas Commission.

In this revision, instrumental test methods for the determination of iron, aluminium, calcium and magnesium have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated. Further, Packing and Marking clause has been updated.

Barytes, the sulphate of barium, also known as 'heavy spar', is extensively mined in Andhra Pradesh and Rajasthan. It is one of the major minerals for export amongst non-metallic minerals. The demand for barytes is mainly for paints and oil-well drilling. Small quantities are also used in the chemical industry for preparation of barium compounds and in rubber and explosive industry. The paint industry takes mostly the best grade white material.

This standard is one of a series of Indian Standards on barytes/ barium sulphate. Other standards published are:

IS 64 Barium sulphate pigments for paints

IS 1683 Barytes for rubber industry — Specification

IS 4156 Methods for sampling of barytes

IS 7588 Barytes for explosives and pyrotechnic compositions — Specification

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
BARYTES FOR CHEMICAL INDUSTRY AND OIL-WELL DRILLING — SPECIFICATION
(*Third Revision*)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for the material commercially known as barytes, intended for the preparation, of barium compounds and for oil-well drilling.

2 REFERENCES

The standards listed in Annex A 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 revision, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of these standards.

3 GRADES AND QUALITIES

3.1 The material shall be of the following two grades:

Grade 1 — suitable for chemical industry, and

Grade 2 — suitable for petroleum industry in oil-well drilling operations.

3.1.1 The material of Grade 1 shall be of two qualities, namely, Quality A and Quality B.

4 REQUIREMENTS

4.1 Description

The material consists essentially of barium sulphate and is white, greyish white or very light pinkish white in colour, free from dirt and foreign matter.

4.2 The material shall comply with the requirements prescribed in Table 1, when tested according to the methods given in Annex B. Reference to relevant clauses of Annex B is given in co1 **6** of the table.

5 PACKING AND MARKING

5.1 Packing

Unless agreed to otherwise between the purchaser and the supplier, the material shall be packed in clean jute bags.

Table 1 Requirements for Barytes for Chemical Industry and Oil-Well Drilling

(*Clause* 4.2, 6.2.2 *and* B-5.3.1)

Sl No.	Characteristics	Requirement			Method of Test, Ref to Cl No. in Annex B
		Grade 1		Grade 2	
		Quality A	Quality B		
(1)	(2)	(3)	(4)	(5)	(6)
i)	Insoluble minus silica, percent by mass, *Max*	97.0	90.0	90.0	**B-2**

ii)	Silica (as SiO_2), percent by mass, *Max*	2.0	6.0*	—	**B-3**
iii)	Aluminium (as Al), percent by mass, *Max*	0.10	—	—	**B-4 or B-11**
iv)	Iron (as Fe), percent by mass, *Max*	0.10	1.5	—	**B-5 or B-11**
v)	Calcium and magnesium (as $CaCO_3$), percent by mass, *Max*	0.10	2.0	—	**B-6 or B-11**
vi)	Fineness (determined by wet method):				
	a) Passing through 75 micron IS Sieve, percent by mass, *Max*	—	—	97	**B-7**
	b) Passing through 53 micron IS Sieve, percent by mass, *Max*	—	—	90 ± 5	**B-7**
vii)	Relative density at 27°/27 °C, *Min*	—	—	4.15[#]	**B-8**
viii)	Matter soluble water, percent by mass, *Max*	—	—	0.02	**B-9**
ix)	Performance			To pass test	**B-10**

*Silica and aluminium oxide together shall be 6.0 percent.

[#] For off-shore drilling the value shall be 4.20.

5.2 Marking

5.2.1 The packages shall be marked legibly and indelibly with the following information:

- a) Name and grade (and quality, if applicable) of the material;
- b) Mass of the material in the package;
- c) Month and year of packing;
- d) Recognized trade-mark, if any; and
- e) Code number to enable the batch of packing to be traced back from records.

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.

6 SAMPLING

6.1 Preparation of Test Samples

Representative test samples of the material shall be prepared as prescribed in IS 1683.

6.2 Number of Tests

6.2.1 Tests for the determination of calcium and magnesium and silica in case of Grade 1, and for the determination of fineness in case of Grade 2 shall be conducted on each of the individual test sample.

6.2.2 Tests for the determination of all the remaining characteristics given in Table 1 shall be carried out on the composite test sample.

6.3 Criteria for Conformity

6.3.1 *For Individual Samples*

For those characteristics which are tested on individual sample, the mean and the range of test results shall be computed as follows:

Mean $(\bar{X}) = \dfrac{Sum\ of\ individual\ test\ results}{Number\ of\ test\ results}$

Range (R) = Difference between the maximum and the minimum values of test results.

For declaring the conformity of the lot:

$\bar{X} - 0.6$ R shall be less than or equal to the maximum specified requirements, and

$\bar{X} - 0.6$ R shall be greater than or equal to the minimum specified requirements.

6.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all characteristics tested on the composite sample, the test results shall satisfy the corresponding specified requirements.

ANNEX A

(*Clause* 2)

(LIST OF REFERRED STANDARDS)

IS No.	*Title*
IS 264 : 2005	Nitric acid — Specification (*third revision*)
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 695 : 2020	Acetic Acid — Specification (*fourth revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1683 : 1994	Barytes for rubber industry specification (*second revision*)
IS 3025(Part 2) : 2019/ISO 11885	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (*first revision*)
IS 3025 (Part 65) : 2022	Methods of sampling and test physical and chemical for water and wastewater part 65 application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*)

ANNEX B

(*Clause* 4.2)

METHODS OF TEST FOR BARYTES FOR CHEMICAL INDUSTRY AND OIL-WELL DRILLING

B-1 QUALITY OF REAGENTS

B-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

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

B-2 INSOLUBLE MINUS SILICA

B-2.1 General

Two methods are prescribed, namely, the classical method and the insoluble-minus-silica method. The latter is recommended for routine determination while the former shall be the referee method.

B-2.2 Classical Method

B-2.2.1 *Outline of the Method*

Barytes powder of known mass (usually not more than 1 g) is fused with 8 to 10 times its mass of sodium carbonate between 800 ℃ to 900 ℃ in a gas-heated or electric furnace. The fused mass is leached out with hot water and filtered. The filtrate is used for the determination of silica and alumina. The residue on the filter paper contains barium, calcium and magnesium as carbonates and iron as oxide. This residue is dissolved in hot dilute hydrochloric acid. The solution obtained contains all the above-mentioned elements in their chloride form and is used for determination of barium, iron, calcium and magnesium. It is made up to 500 ml. For barium determination as aliquot is taken, barium is precipitated as barium sulphate with the addition of ammonium sulphate which is ignited and weighed.

B-2.2.2 *Reagent*

B-2.2.2.1 *Sodium carbonate* — anhydrous.

B-2.2.2.2 *Sodium carbonate solution* — 2 g/1.

B-2.2.2.3 *Dilute hydrochloric acid* — 1 : 4.

B-2.2.2.4 *Ammonium hydroxide* — 20 percent (*m/m*).

B-2.2.2.5 *Concentrated hydrochloric acid* — *see* IS 265.

B-2.2.2.6 *Methyl orange indicator*

Dissolve 0.1 g of methyl orange in 100 ml of water.

B-2.2.2.7 *Ammonium sulphate solution* — 3 percent.

B-2.2.3 *Procedure*

B-2.2.3.1 Weigh about 8 g of sodium carbonate. Place an appropriate quantity out of it in a platinum crucible so as to form a layer about 6 mm deep. Separately, weigh accurately about 1 g of the finely powdered material and place it on top of the sodium carbonate layer in the crucible. Mix both with a glass rod. Add the rest of sodium carbonate also to the crucible. Cover and fuse the mixture at 800 °C to 900 °C in either a gas-heated or electric furnace till the mix melts.

Cool and as the melt cools, rotate the crucible so that the fused mass solidifies in a thin layer. This will shorten the time required for leaching. Leach out the fuse mass with 200 ml of hot water in a 400 ml beaker with thorough stirring. Filter through a 15 cm filter paper (Whatman No. 40 or equivalent). Wash several times by decantation, then remove the crucible from the beaker, transfer the insoluble carbonates to the filter, and wash with hot sodium carbonate solution testing after the twelfth washing to be certain that sulphates have been removed completely. Collect all the filtrate, make up to 500 ml and reserve for the determination of silica in **B-3**.

B-2.2.3.2 Cover the funnel containing the insoluble carbonates with a watch-glass and add hot dilute hydrochloric acid carefully, in small portions at a time to prevent loss, catching the solution in a 600 ml beaker. Add hot dilute hydrochloric acid to the platinum crucible and the beaker in which the leach was made and pour over the filter. Wash the paper with hot water until free from chlorides. Make up the solution to 500 ml. Use it for the determination of barium sulphate content in **B-2.2.3.3**, iron in **B-5** and calcium and magnesium in **B-6.**

B-2.2.3.3 Take 100 ml aliquot of the solution. Neutralize this with ammonium hydroxide using methyl orange as indicator. Add 0.4 ml to 0.6 ml of concentrated hydrochloric acid. Dilute to 400 ml with hot water, bring the solution to boiling, and add 25 ml of hot ammonium sulphate solution dropwise with constant stirring to prevent co precipitation of calcium and magnesium. Transfer the beaker to a warm hot-plate and allow to stand for at least 4 hours. Filter on an ignited/weighed Gooch crucible wash with hot water several times by decantation. The beaker should be scrubbed thoroughly to remove any adhering, precipitate. Continue the washing until free of chlorides. Ignite the crucible in a muffle furnace for 30 min at 850 °C. Cool in a desiccator and weigh. Make a blank determination in a similar manner with an equal amount of sodium carbonate and other reagents.

B-2.2.4 *Calculation*

$$\text{Insoluble minus silica, percent by mass} = \frac{100\,(M_1 - M_2)}{M}$$

where

        M_1 = mass in g of the precipitate with the material,

        M_2 = mass in g of the precipitate in the blank determination, and

        M = mass in g of the material present in the aliquot used.

B-2.3 Insoluble-Minus Silica Method

B-2.3.1 This method presumes that the material other than silica left in the determination is barium sulphate.

B-2.3.2 *Reagents*

B-2.3.2.1 *Concentrated hydrochloric acid — see* IS 265.

B-2.3.2.2 *Concentrated sulphuric acid — see* IS 266.

B-2.3.2.3 *Hydrochloric acid —* 48 percent.

B-2.3.3 *Procedure*

B-2.3.3.1 Weigh about 1 g of the sample (as received) (to the nearest 0.1 mg and place it in a 250 ml beaker. Add 50 ml of concentrated hydrochloric acid and digest over hot water-bath for 10 to 15 min with constant stirring. After digesting, filter it through Whatman No. 48 filter paper (or equivalent) and wash the residue with hot water till free from chloride. Keep the residue (free from chloride) in an oven to become dry, and then incinerate the same in a weighed platinum crucible.

B-2.3.3.2 *Acid treatment*

Cool the platinum crucible and treat the residue with 2 to 3 drops of concentrated sulphuric acid and 10 ml of hydrofluoric acid. Then place the crucible on a hot sand-bath till the acid is completely evaporated. Remove the crucible from sand-bath and gently heat over a low burner flame. Finally heat the crucible till constant mass is obtained. Weigh the residue.

B-2.3.4 *Calculation*

$$\text{Insoluble-minus-silica, percent by mass} = \frac{100\,(M_3 - M_1)}{M_2}$$

where

M_1 = mass in g of the crucible + lid, + residue (after hydrofluoric acid treatment),

M_2 = mass in g of the material taken for the test, and

M_3 = mass in g of the crucible + residue + lid + residue (before hydrofluoric acid treatment).

B-3 SILICA

B-3.1 Outline of the Method

An aliquot of the solution obtained in **B-2.2.3.1** is acidified with dilute hydrochloric acid and evaporated to dryness to dehydrate the silica, and treated with hydrochloric acid and filtered. Silica obtained is ignited and weighed as SiO_2. The silica is hydrofluorized with hydrofluoric acid to obtain the content of pure silica.

B-3.2 Reagents

B-3.2.1 *Dilute Hydrochloric Acid —* 1 : 1.

B-3.2.2 *Hydrochloric Acid —* 40 to 60 percent (*m/v*).

B-3.2.3 *Concentrated Sulphuric Acid — see* IS 266.

B-3.3 Procedure

Take 100 ml aliquot of the solution reserved in **B-2.2.3.1** in a 400 ml beaker, acidify with dilute hydrochloric acid and evaporate to dryness to dehydrate the silica. After dehydrating, take up the residue with dilute hydrochloric acid and filter. The filtrate is again evaporated to dryness, taken up in dilute hydrochloric acid and filtered as before on a new filter paper. Reserve the filtrate for the determination of iron and aluminium oxides in **B-5**. Wash the filter paper thoroughly with hot water and place in a platinum crucible. Ignite at 900°C for 30 min and weigh. Add a few drops of hydrofluoric acid and concentrated sulphuric acid to the crucible, evaporate to dryness, ignite and weigh.

B-3.4 Calculation

$$\text{Silica (as SiO}_2\text{), percent by mass} = \frac{100\,(M_1 - M_2)}{M}$$

where

M_1 = mass in g of the crucible before treatment with hydrofluoric acid,

M_2 = mass in g of the crucible after treatment with hydrofluoric acid, and

M = mass in g of the material present in the aliquot.

B-4 ALUMINIUM

B-4.1 Outline of the Method

The formation of a red aluminium lake with aurin tricarboxylic acid, also known as 'aluminon' is the basis for calorimetric estimation of aluminium present in small quantities. The lake is formed in the presence of acetic acid - acetate buffer. Since silicates and phosphates interfere they have removed first. The optimum intensity of colour is developed around pH 5.3.

B-4.2 Apparatus

B-4.2.1 *Nessler Cylinders* — 50 ml capacity

B-4.3 Reagents

B-4.3.1 *Aluminon Solution*

Dissolve 1.54 g of ammonium acetate in 500 ml of water, add 25 ml of dilute hydrochloric acid 1 : 4 (*v/v*) and mix. Dissolve 0.4 g of ammonium salt of aurin tricarboxylic acid (aluminon) in water, add and mix with the above solution. Finally dissolve 1 g of gum arabic or pure gelatin in water, add to above and dilute the solution exactly to 1 litre. This solution should be stored in amber-coloured bottle.

B-4.3.2 *Standard Aluminium Solution*

Dissolve 1.757 0 g of potash alum (K_2SO_4. $Al_2(SO_4)_3$.$24H_2O$) in water and dilute exactly to one litre. One millilitre of this solution is equivalent to 0.10 mg of aluminium (as Al).

B-4.3.3 *Glacial Acetic Acid* — *see* IS 695.

B-4.3.4 *Ammonium Hydroxide* — 20 percent (*m/m*).

B-4.3.5 *Ammonium Carbonate* — solid.

B-4.4 Procedure

B-4.4.1 Pipette out 25 ml of the solution reserved in **B-2.2.3.2** into a Nessler cylinder. Add 5 ml of dilute hydrochloric acid and 5 ml of glacial acetic acid. Then add 5 ml of ammonium hydroxide and 1 g of ammonium carbonate and mix. The pH of the solution at this stage should be between 5.0 and 6.0. Finally, add 5 ml of aluminon solution and make up to 50 ml. Shake well and allow to stand. A pink colour slowly develops.

B-4.4.2 In four other Nessler cylinders, take 1 ml to 4 ml of the standard aluminium solution and add the same quantities of other reagents as in **B-4.4.1**, finally making up to 50 ml volume and mixing well. Allow the pink colour to develop on standing. Compare the colour given by the test solution against these four standards and determine which is nearest matching colour.

B-4.4.3 If the test solution colour matches with a cylinder

    a) having 1 ml of standard aluminium solution, then the barytes sample contains 0.2 percent aluminium,

    b) having 2 ml standard solution, then the barytes sample contains 0.4 percent aluminium,

    c) having 4 ml of standard solution, then the barytes sample contains 0.6 percent aluminium or; and

    d) having 4 ml of standard solution, then the barytes sample contains 0.8 percent aluminium.

B-4.5 Alternative Method

Aluminium may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-11** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

B-5 IRON

B-5.1 Apparatus

B-5.1.1 *Nessler Cylinders* — 50 ml capacity.

B-5.2 Reagents

B-5.2.1 *Dilute Hydrochloric Acid* — 1: 1 (*v/v*).

B-5.2.2 *Concentrated Nitric Acid* — *see* IS 264.

B-5.2.3 *Ammonium Persulphate* — solid.

B-5.2.4 *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 solution is clear.

B-5.2.5 *Standard Iron Solution*

Dissolve 0.702 g of ferrous ammonium sulphate [$FeSO_6 (NH_4)_2 SO_4.6H_2O$] in 10 ml of 10 percent sulphuric acid and dilute with water to make up to 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make the volume to 100 ml exactly. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

B-5.3 Procedure

Pipette out 25 ml of solution reserved in **B-2.2.3.2** into a Nessler cylinder, add to this 5 ml of concentrated nitric acid and mix thoroughly with a glass rod. Add about 30 mg of ammonium per sulphate and stir to dissolve. Finally, add 15 ml of butanolic potassium thiocyanate solution, make up to 50 ml with water and mix the contents of the cylinder well. Allow the cylinder to stand when the butanolic layer is seen to separate out slowly at the top carrying the pink coloured iron complex in it. Carry out a control test in another Nessler cylinder using 5 ml of the standard iron solution and the same quantities of other reagents used in the test. Compare the colours in the two Nessler cylinders.

B-5.3.1 The limit prescribed for iron in Table 1 shall be taken as not having been exceeded if the colour in the test with material is not deeper than that produced in the control test.

B-5.4 Alternative Method

Iron may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-11** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

B-6 CALCIUM AND MAGNESIUM

B-6.1 Outline of the Method

Calcium and magnesium are determined together by titration of an aliquot of the filtrate obtained in **B-2.2.3.2** with standard EDTA solution.

B-6.2 Reagents

B-6.2.1 *Standard Calcium Chloride Solution*

Dissolve 1 g of calcium carbonate (dried at 120 ℃) in minimum quantity of concentrated hydro chloric acid and make up the solution to exactly one litre.

B-6.2.2 *Ethylenediamine Tetra-Acetic Acid (EDTA) Solution*

Dissolve 7.44 g of disodium ethylenediamine tetra-acetate dihydrate in water and make up the volume to two litres.

B-6.2.3 *Ammonium Hydroxide Ammonium Chloride Buffer Solution*

Dissolve 67.5 g of ammonium chloride in a mixture of 570 ml of ammonium hydroxide (20 percent *m/m*) and 250 ml of water. Dissolve separately a mixture of 0.931 g of EDTA and 0.616 g of magnesium sulphate heptahydrate in about 50 ml of water. Mix the two solutions and make up to one litre.

B-6.2.4 *Eriochrome Black T Indicator Solution*

Dissolve 0.1 g of the material in 20 ml of methanol. This solution shall be prepared fresh at weekly intervals.

B-6.3 Procedure

B-6.3.1 *Standardization of EDTA Solution*

The EDTA solution is standardized against standard calcium chloride solution with eriochrome black T indicator. Pipette out 50 ml of standard calcium chloride solution in a 250 ml conical flask and add 5 ml of ammonium hydroxide ammonium chloride buffer solution (*p*H of the solution approximately 10) and 5 drops of eriochrome black T indicator solution to it. Titrate with EDTA solution until the wine red colour changes to pure blue (A ml).

B-6.3.2 Take 100 ml aliquot of the solution reserved in **B-2.2.3.2** in a 250 ml conical flask and add 5 ml of ammonium hydroxide-ammonium chloride buffer solution and 5 drops of eriochrome black T indicator. Titrate with EDTA solution until the wine red colour changes to pure blue (B ml).

B-6.4 Calculation

$$\text{Calcium and magnesium (as CaCO}_3\text{), percent by mass} = \frac{5\,B}{A\,M}$$

where

        B = volume in ml of EDTA solution required in **B-6.3.2**,

        A = volume in ml of EDTA solution required in **B-6.3.1**, and

        M = mass in g of the material present in the aliquot.

B-6.5 Alternative Method

Calcium and magnesium may alternatively be determined by ICP-OES instrumental test method as prescribed at **B-11** or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

B-7 FINENESS

B-7.1 Procedure

Place 25 g of the material, accurately weighed, on to 75 micron and 53 micron IS sieves placed one above the other, the former being on the top. Shake mechanically or by hand for 15 min. Wash the material on each sieve with water, gently brushing with a soft camel-hair brush, until the washings are clear. Dry the material passing through each sieve to constant mass at (100 ± 2) ºC.

B-7.2 Calculation

$$\text{Fineness, material passing through respective IS Sieve, percent by mass} = 4\,M$$

where

        M = mass in g of the material passing through the sieve.

B-8 RELATIVE DENSITY

B-8.1 Apparatus

B-8.1.1 *Relative Density Bottle* — 50 ml capacity.

B-8.2 Procedure

B-8.2.1 Dry the material to constant mass at 105 °C to 110 °C and place the dried material in a glass-stoppered weighing bottle. Carry out the test in duplicate.

B-8.2.2 Dry the relative density bottle and its stopper at 105 °C to 110 °C, cool in a desiccator and weigh to the nearest milligram. Fill the bottle with kerosine maintained at (27.0 ± 0.5) °C and again weigh (M_1). Empty the bottle and dry again.

B-8.2.3 Partially fill (one-fourth to half of its capacity) the dry relative density bottle with kerosine maintained at (27.0 ± 0.5) °C. Place exactly 40 g of the dried material (M_2) in the bottle and connect to a vacuum pump to remove any adhering air bubbles. Fill it up with kerosine maintained at (27.0 ± 0.5) °C and weigh (M_2).

B-8.3 Calculation

$$\text{Relative density at 27/27 °C} = \frac{M_2}{M_2 - (M_3 - M_1)} \times d$$

where

M_2 = mass in g of the material taken,

M_3 = mass in g of the relative density bottle with the material and kerosine,

M_1 = mass in g of the relative density bottle with kerosine, and

d = relative density of kerosine at 27/27 °C.

B-8.3.1 The two determinations shall agree to within 0.005, otherwise additional determinations shall be made.

B-9 MATTER SOLUBLE IN WATER

B-9.1 Procedure

Weigh accurately about 100 g of the powdered material. Wet it thoroughly with a small quantity of water and then add 200 ml freshly boiled and cooled water. Boil for 5 min. Cool the mixture to room temperature. Make up to 250 ml with freshly boiled and cooled water, shake and filter. Reject the first 50 ml portion of the filtrate. Take a 100 ml portion from the rest of the filtrate in a tared platinum dish and evaporate to dryness on a water-bath. Dry the residue obtained to constant mass in an oven at 105 °C to 110 °C.

B-9.2 Calculation

$$\text{Matter soluble in water, percent by mass} = \frac{250\,A}{M}$$

where

A = mass in g of the residue, and

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

B-10 TEST FOR PERFORMANCE

B-10.1 General

The purpose of the performance test is to observe the effect on the rheological properties of a standard bentonite suspension when it is loaded with the barytes under test. The rheological parameters will increase due to the increase in the content of solid particles in the mud. But if the barytes contain appreciable quantities of soluble salts, especially calcium and magnesium salts, the rheological parameters will increase much more than with the addition of the same amount of salt-free barytes. The degree of fineness of the barytes particles also plays a role in it. A bentonite suspension in water having an apparent viscosity of 15 centipoise is taken more or less as a standard for laboratory testing of mud chemicals.

B-10.2 Apparatus

B-10.2.1 *Direct Indicating Torsion-Viscometer*

The instrument is based on the principle that the torque exerted by a liquid is directly proportional to its viscosity. It consists of two cylinders. Mud is contained in the annular space between the two cylinders. The outer cylinder or rotor sleeve is connected to a motor and is driven at a constant rotational velocity. The rotation of the rotor sleeve in the mud produces a torque on the inner cylinder or bob. A torsion spring restrains the movement. A dial attached to the bob indicates displacement of the bob. The instruments constants have been so adjusted that the apparent viscosity in centipoise (cP) equals the 600 rev/min reading divided by 2.

B-10.2.2 *Electric Stirrer*

A high speed laboratory electric stirrer having a minimum speed of 5 000 rev/min with corrugated impeller.

B-10.3 Material

B-10.3.1 *Bentonite Powder*

An approved sample of bentonite powder which on preparation of 5 to 7 percent suspension in water gives a mud of 15 to 20 centipoise.

B-10.4 Procedure

Prepare a 5 to 7 percent bentonite suspension by making 5 to 7 of approved bentonite per 100 g of suspension in water. Stir for 15 min with the stirrer, and then age for 14 h at room temperature.

After the lapse of ageing period, stir for 5 min. Place the suspension in a suitable container and immerse the rotor sleeve exactly to the inscribed line. With the sleeve rotating at 600 rev/min, wait for the dial reading to reach a steady value. Divide the dial reading at 600 rev/min by 2 to obtain the apparent viscosity. This value should be 15 to 20 centipoise.

B-10.4.1 Load 400 ml of bentonite suspension with 310 g of barytes so that the relative density of the suspension is raised to 1.50 (*see* Note). Stir for 5 min and determine the apparent viscosity as in **B-10.4**.

> NOTE — The relative density of barytes varies. Therefore, the mass of barytes required to be added to, say, one litre of bentonite suspension to raise its relative density to 1.50 cannot be accurately specified. Generally, 310 g of barytes is added to 400 ml of bentonite suspension. The formula given below serves as a guide:

$$V_1 D_1 \pm V_2 D_2 = (V_1 \pm V_2)\, D_3$$

OR

$$M = \frac{V_1 D_2 (D_3 - D_1)}{(D_2 - D_3)}$$

where

V_1 = volume in ml of bentonite suspension,

D_1 = relative density of bentonite suspension,

V_2 = volume in ml of barytes added,

D_2 = relative density of barytes added,

D_3 = desired relative density of the loaded mud, and

M = mass in g of barytes required to be added ($V_2 D_2$).

B-10.4.2 The material shall be considered to possess satisfactory performance if the apparent viscosity of the mud is not more than 50 centipoises.

B-11 DETERMINATION OF ALUMINIUM, CALCIUM, IRON AND MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-11.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get 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 intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

B-11.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clauses* B-11.2 and B-11.4)

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (µg)	Axial viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
ii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr
iii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr

B-11.3 Reagents and Solutions

B-11.3.1. *Nitric Acid (65 percent) Suprapure*

B-11.3.2 *Standard Stock Solution*

Either Prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 µg/ml of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-11.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask & make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

B-11.3.4 *Sample preparation*

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

> NOTE — Sample should be clear before injecting to the instrument.

B-11.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

B-11.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

> NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

B-11.5 Procedure

B-11.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-11.3.5**). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

B-11.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the aluminium (and/or Iron, calcium, magnesium) in the sample solution.

> NOTE — It is recommended that IS 3025(Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

B-11.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.

ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).