

भारतीय मानक मसौदा  
अमोनिया □□□□ – विशिष्टि  
(आईएस 799 का तीसरा का पुनरीक्षण)

*Draft Indian Standard*  
**AMMONIA, LIQUOR — SPECIFICATION**  
(*Third Revision of IS 799*)

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

ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

**Last date of comments: 17<sup>th</sup> December 2022**

## FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was first prepared in 1955 and was first revised in 1978 and 1985 thereby adding a new grade, namely, analytical reagent grade. The requirement for relative density (specific gravity) was deleted in the first revision as it was felt that it need not be a part of the specification. However, a correlation table of relative density and percentage of ammonia was given in Annex A. In the second revision, alternate spectrophotometric methods for the determination of phosphates, silica and iron and an alternate method for the determination of ammonia as prescribed in ISO/DIS 7108 'Ammonia solution for industrial use — Determination of ammonia content — Titrimetric method' issued by the International Organization for Standardization was prescribed.

Liquid ammonia finds application as a water treatment chemical in boiler system.

In this revision, instrumental test methods for the determination of arsenic, calcium, chlorides, copper, iron, lead, sulphates and magnesium have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause and Amendment have been incorporated.

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.

## 1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for ammonia, liquor.

1.1.1 The material is also known as aqua ammonia, and ammonium hydroxide.

## 2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

<i>IS No.</i>	<i>Title</i>
265 : 2021	Hydrochloric acid — Specification ( <i>fifth revision</i> )
336 : 1973	Ether ( <i>third revision</i> )
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
2088 : 1983	Methods for determination of arsenic ( <i>second revision</i> )
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) ( <i>first revision</i> )
4544 : 2000	Ammonia — Code of safety ( <i>first revision</i> )
4905: 2015/ISO 24153	Random sampling and randomization procedures ( <i>first revision</i> )

## 3 GRADES

3.1 There shall be two grades, namely,

- Technical grade intended for uses, such as cleansing agent, refrigerant, accelerator in vulcanization, and in the ammonia process of ferroprinting of drawings.
- Analytical reagent grade.

## 4 REQUIREMENTS

### 4.1 Description

The material shall be a clear and colourless liquid of extremely pungent and characteristic odour, free from suspended matter and sediment. The material absorbs carbon dioxide from air.

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

## 5 PACKAGING AND MARKING

### 5.1 Packaging

The material shall be packed in closed containers as agreed to between the purchaser and the supplier.

## **5.2 Marking**

The packages shall be securely closed and bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and his recognized trade mark, if any;
- c) Gross and net mass;
- d) Date of manufacture and;
- e) Batch number

### **5.2.1 *BIS Certification Marking***

The product may also be marked with the Standard Mark.

**5.2.1.1** 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 there under, and the products may be marked with the standard mark.

**LIQUID CAUSES BURNS – VAPOUR EXTREMELY IRRITATING: HANDLE WITH CARE**

**Table 1 Requirements For Ammonia, Liquor**  
(Clause 4.2)

Sl. No.	Characteristic	Requirement		Method of Test, Ref to Clause No. in Annex B
		Technical Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)
i)	Ammonia, percent by mass, <i>Min</i>	20.0	25.0	B-2
ii)	Residue on evaporation, percent by mass, <i>Max</i>	0.1	0.002	B-3
iii)	Carbonate (as CO <sub>2</sub> ), percent by mass, <i>Max</i>	—	0.01	B-4
iv)	Chloride (as Cl), percent by mass, <i>Max</i>	—	0.00005 (0.05 ppm)	B-5 or B-16
v)	Phosphate (as PO <sub>4</sub> ), percent by mass, <i>Max</i>	—	0.0001 (1 ppm)	B-6
vi)	Silicate (as SiO <sub>2</sub> ), percent by mass, <i>Max</i>	—	0.001 (10 ppm)	B-6
vii)	Sulphate (as SO <sub>4</sub> ), percent by mass, <i>Max</i>	—	0.0002 (2 ppm)	B-7 or B-16
viii)	Sulphide (as S), percent by mass, <i>Max</i>	—	0.00001 (0.01 ppm)	B-8
ix)	Arsenic (as As), percent by mass, <i>Max</i>	—	0.000005 (0.05 ppm)	B-9 or B-17
x)	Calcium and magnesium (as Ca), percent by mass, <i>Max</i>	—	0.0004 (4 ppm)	B-10 or B-17
xi)	Copper (as Cu), percent by mass, <i>Max</i>	—	0.00001 (0.1 ppm)	B-11
xii)	Iron (as Fe), percent by mass, <i>Max</i>	—	0.00002 (0.2 ppm)	B-12 or B-17
xiii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	—	0.00004 (0.4 ppm)	B-13 or B-17
xiv)	Reducing substances (as O), percent by mass, <i>Max</i>	—	To pass test	B-14
xv)	Pyridine and homologues, percent by mass, <i>Max</i>	—	To pass test	B-15

## 6 SAMPLING

**6.1** Representative test samples of the material shall be drawn and adjudged as prescribed in Annex C.

ANNEX A  
(Foreword)

Correlation Table Of Relative Density And Percent By Mass Of Ammonia, Liquor At 27/27°C			
Relative Density at 27/27°C	Percent by Mass	Relative Density at 27/27°C	Percent by Mass
0.9929	1.00	0.933 3	16.48
0.9912	1.50	0.931 8	16.80
0.989 4	2.00	0.930 2	17.25
0.980 7	4.06	0.928 7	17.73
0.979 0	4.37	0.927 2	18.11
0.977 3	4.89	0.925 6	18.57
0.975 6	5.32	0.924 1	19.07
0.973 9	5.78	0.922 6	19.17
0.972 2	5.98	0.921 1	19.89
0.970 5	6.41	0.919 5	20.35
0.968 9	6.84	0.918 0	20.83
0.967 2	7.27	0.9165	21.31
0.965 5	7.61	0.915 0	21.66
0.963 9	8.07	0.913 5	22.11
0.9622	8.49	0.9121	22.53
0.9605	8.93	0.910 6	23.07
0.958 9	9.27	0.909 1	23.42
0.957 3	9.72	0.907 6	23.91
0.955 6	10.17	0.906 1	24.41
0.954 0	10.62	0.904 7	24.93
0.952 4	10.99	0.903 2	25.32
0.950 8	11.44	0.901 8	25.81
0.949 2	11.93	0.900 3	26.30
0.947 5	12.39	0.898 9	26.81
0.945 9	12.76	0.8974	27.11
0.944 4	13.19	0.896 0	27.65
0.942 8	13.64	0.894 6	28.19
0.941 2	14.12	0.893 2	28.68
0.939 6	14.58	0.891 8	29.17
0.938 0	14.90	0.890 2	29.67
0.936 5	15.28	0.8888	30.16
0.934 9	15.93		

## ANNEX B

(Clause 4.2, and Table 1)

## METHODS OF TEST FOR AMMONIA, LIQUOR \

## B-1 QUALITY OF REAGENTS

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

NOTE 1 — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

NOTE 2 — Lunge-Ray pipette is recommended for carrying out analysis of the material.

**B-2 DETERMINATION OF AMMONIA**

**B-2.1** Ammonia may be determined by either of the two methods, namely, Method A and Method B. In case of dispute Method B shall be adopted.

**B-2.2 Method A****B-2.2.1 Reagents**

**B-2.2.1.1** *Standard sulphuric acid* — 0.5 N.

**B-2.2.1.2** *Methyl red indicator*

Dissolve 0.5 g of water soluble methyl red in water and dilute the solution to 1 l.

**B-2.2.1.3** *Caustic soda solution* — 0.5 N, freshly standardized.

**B-2.2.2 Procedure**

Accurately weigh a 15 ml capacity ground glass stoppered weighing bottle containing 10 to 12 ml of water. Open the ammonia bottle and insert a clean and dry glass tube of about 5 mm bore to half the height of the liquid and transfer 1 to 15 ml of the material to the weighing bottle immediately replace the glass stopper and weigh. Place the weighing bottle in about 200 ml of water containing 50 ml of 0.5 N sulphuric acid and a few drops of methyl red indicator.

Due to the mass of the water and ammonia contained in the bottle, it will remain immersed in the acid. Open the stopper under the acid by manipulating with a glass rod and titrate the excess of acid with standardized caustic soda solution to a faint yellow end point.

**B-2.2.2.1** Carry out a blank with all the reagents used for the test.

**B-2.2.2.2 Calculation**

$$\text{Ammonia, percent by mass} = \frac{1.703 (V_1 - V_2)}{M}$$

where,

$V_1$  = volume in ml of standard sodium hydroxide solution used in the blank,

$V_2$  = volume in ml of standard sodium hydroxide solution used in the test with the material,

$N$  = normality of standard sodium hydroxide solution, and

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

**B-2.3 Method B****B-2.3.1 Reagents**

**B-2.3.1.1** *Boric acid* — 2 percent.

**B-2.3.1.2** *Standard sulphuric acid solution* — 0.5 N.

**B-2.3.1.3** *Methyl red* — 0.1 percent.

Dissolve 0.1 g of methyl red in 95 percent (v/v) ethanol and make up to 100 ml with the same ethanol.

**B-2.3.2 Apparatus****B-2.3.2.1 Spherical glass ampoule**

Of thin glass, of suitable capacity and shape, for example, about 20 mm diameter, with one capillary end about 50 mm in length (a typical example is shown in Fig. 1).

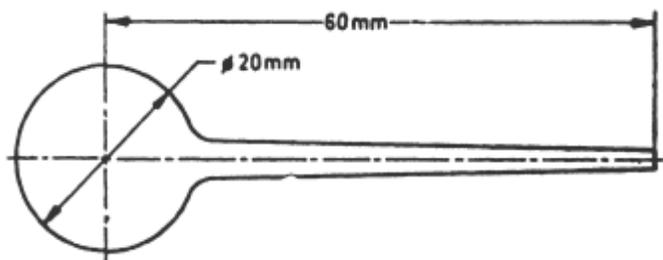


FIG. 1 SPHERICAL GLASS AMPOULE

**B-2.3.3 Procedure**

Weigh the glass ampoule to the nearest 0.01 g. Gently heat the spherical part of the ampoule over a flame and dip the capillary end of the ampoule into the bottle containing the laboratory sample. Ensure that the ampoule is almost completely filled during cooling.

Withdraw the ampoule and dry the capillary tube carefully with filter paper. Seal the end of the capillary tube, without loss of glass, with an oxidizing flame. Allow the capillary tube to cool, wash it with water and wipe it carefully with filter paper. Weigh the sealed ampoule accurately and calculate, by difference, the mass of the test portion.

**B-2.3.3.1** Carefully place the ampoule containing the test portion into a 500 ml conical flask, fitted with a ground glass stopper, to which 50 ml of the boric acid solution, about 250 ml of water and several drops of the methyl red solution have already been added.

Stopper the conical flask and shake carefully so as to break the ampoule. Unstopper the flask, rinse the stopper with water collecting the washings in the same flask.

Using a glass rod, grind the pieces of the ampoule, in particular those parts of the capillary tube which may have remained unbroken. Remove the glass rod, rinse it with water, collecting the washings in the same flask. Titrate with the sulphuric acid solution until the indicator changes from yellow to red.

**B-2.3.3.2 Calculation**

$$\text{Ammonia, percent by mass} = \frac{1.703}{V}$$

where,

$V$  = volume in ml of standard sulphuric acid used, and

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

**B-3 DETERMINATION OF RESIDUE ON EVAPORATION****B-3.1 Procedure**

Measure 500 ml of the material in case of AR grade and 100 ml for technical grade in a volumetric flask. Add the whole material, in small portions, to a tared platinum crucible or other suitable dish, and evaporate to dryness on a steam bath, inside a fume cupboard. Dry the residue at  $105 \pm 2^\circ\text{C}$  to constant mass.

**B-3.2 Calculation**

$$\text{Residue on evaporation, percent by mass} = \frac{100 M}{VS}$$

where

$M$  = mass in g of the residue,

$V$  = volume in ml of the material taken for the test, and

$S$  = relative density of the material.

**B-4 DETERMINATION OF CARBONATE****B-4.1 Apparatus**

**B-4.1.1 Nessler Cylinders** — 25 ml capacity.

**B-4.2 Reagents**

**B-4.2.1 Barium Hydroxide Solution** — saturated.

**B-4.2.2 Standard Sodium Carbonate (Anhydrous) Solution** — 0.01 N.

**B-4.3 Procedure**

Weigh accurately 1.5 g of the material, dilute to 20 ml with carbon dioxide-free water and add 5 ml barium hydroxide solution. Carry out a control test in the other Nessler cylinder by taking 0.5 ml of standard sodium carbonate solution, 20 ml of carbon dioxide-free water and 5 ml of barium hydroxide solution.

**B-4.3.1** The material shall be taken to have passed the test if the turbidity produced in the test with the material is not greater than that produced in the control test.

**B-5 DETERMINATION OF CHLORIDES****B-5.1 Apparatus**

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

**B-5.2 Reagents**

**B-5.2.1 Silver Nitrate Solution** — 0.1 N approximately.

**B-5.2.2 Dilute Nitric Acid** — 4 N approximately.

**B-5.2.3 Standard Chloride Solution A**

Dissolve 1.648 g of sodium chloride (dried at  $105 \pm 2^\circ\text{C}$ ) in water and dilute to 1000 ml in a volumetric flask. One millilitre of this diluted solution contains 1.0 mg of chloride (as Cl).

#### **B-5.2.3.1** *Standard chloride solution B*

Dilute 10 ml of standard chloride solution A (**B-5.2.3**) to 1000 ml with water. One millilitre of this diluted solution contains 0.01 mg of chloride (as Cl).

### **B-5.3 Procedure**

Accurately weigh 20 g of the material and evaporate on a steam bath until reduced to 1 ml. Then dilute with 50 ml of water and add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Carry out a control test in the other Nessler cylinder using 1 ml of standard chloride solution B, 1 ml of dilute nitric acid, 1 ml of silver nitrate solution and 50 ml of water. Wait for 5 min and compare the opalescence in both the cylinders.

**B-5.3.1** The material shall be taken to have passed the test if the opalescence produced in the test with the material is not greater than that produced in the control test.

### **A-5.4 Alternative Method**

Chlorides may alternatively be determined by instrumental test method as prescribed in **B-16**.

## **B-6 DETERMINATION OF PHOSPHATE AND SILICATE**

**B-6.1** Two methods are prescribed for the determination of phosphate and silicate, namely colorimetric method and spectrophotometric method. The spectrophotometric method shall be adopted in case of dispute.

### **B-6.2 Colorimetric Method**

#### **B-6.2.1** *Apparatus*

**B-6.2.1.1** *Separating funnels* — 200 to 250 ml capacity.

**B-6.2.1.2** *pH meter* — with glass electrode.

NOTE — Alternatively, universal standard indicator paper may be used.

**B-6.2.1.3** *Nessler cylinders* — 50 ml capacity.

#### **B-6.2.2** *Reagents*

**B-6.2.2.1** *Dilute Sulphuric Acid* — approximately 5 percent (v/v).

**B-6.2.2.2** *Ammonium Molybdate* — solid.

**B-6.2.2.3** *Concentrated Hydrochloric Acid* — See IS 265

**B-6.2.2.4** *Dilute Hydrochloric Acid* — approximately 10 percent (v/v).

**B-6.2.2.5** *Dilute Hydrochloric Acid* — 1 percent (v/v).

**B-6.2.2.6** *Ethyl Ether* — see IS 336

**B-6.2.2.7** *Stannous Chloride Solution*

Dissolve 2 g of stannous chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in concentrated hydrochloric acid and dilute to 100 ml with the acid.

#### **B-6.2.2.8** *n-Butanol*

#### **B-6.2.2.9** *Standard Phosphate Solution*

Dissolve 1.43 g of potassium dihydrogen orthophosphate ( $\text{KH}_2\text{PO}_4$ ) in water and dilute to 1000 ml in a volumetric flask. One millilitre of this solution contains 1.0 mg of phosphate (as  $\text{PO}_4$ ).

#### **B-6.2.2.10** *Standard Silicate Solution*

Fuse 2.500 g of precipitated silica with 5 g of sodium carbonate in a platinum crucible until all the silica is dissolved in the molten sodium carbonate. Cool and extract the melt with hot water. When all the solid is dissolved, add 2 to 3 g of sodium hydroxide, make up to 250 ml and store in polyethylene bottle. One millilitre of this solution contains ten milligrams of silica (as  $\text{SiO}_2$ ).

#### **B-6.2.2.11** *Standard Phosphate-Silicate Solution*

Take 10 ml of standard phosphate solution (**B-6.2.2.9**) and 10 ml of standard silicate solution (**B-6.2.2.10**) and dilute to 1000 ml with water in a volumetric flask. One millilitre of this diluted solution is equivalent to 0.01 mg of phosphate (as  $\text{PO}_4$ ) and 0.1 mg of silicate (as  $\text{SiO}_2$ ). This standard phosphatesilicate solution should be freshly prepared before use.

#### **B-6.2.3** *Procedure*

Accurately weigh 10 g of the material and evaporate on a steam bath until reduced to 1 ml. Then dilute with water, adjust to a *pH* of about 4 by addition of dilute sulphuric acid and dilute to a volume of about 75 ml. Take 1 ml of standard phosphate-silicate solution. Add 0.5 g of ammonium molybdate each to the test solution and the standard phosphate-silicate solution and when it dissolves, adjust the *pH* to 2 by adding dilute hydrochloric acid. Check the *pH* with the *pH* meter using glass electrode or universal *pH* indicator paper. Heat both the solutions to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid to each and dilute to 100 ml with water. Transfer the solutions to two separating funnels, add 35 ml of ether to each, shake vigorously and allow to separate. Draw off the aqueous phases. Proceed for the determination of phosphates as given in **B-6.2.3.1** in the ether phase. Determine silicate in the aqueous phase as given in **B-6.2.3.2**.

#### **B-6.2.3.1** *Determination of Phosphate*

Wash the ether phase of each funnel from **B-6.2.3**, by shaking with 10 ml of dilute hydrochloric acid, allow to separate, and drain off and discard the aqueous phase. Add 0.2 ml of freshly prepared stannous chloride solution to each ether extract and shake. If the ether extracts are turbid, wash with 10 ml of dilute hydrochloric acid. Transfer the ether extract to the Nessler cylinders. The limit prescribed for phosphate in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

#### **B-6.2.3.2** *Determination of Silicate*

To the two aqueous phases from **B-6.2.3**, add 10 ml of concentrated hydrochloric acid and transfer to separating funnel. Add 40 ml of butanol, shake vigorously, and allow to separate. Draw off and discard the aqueous phase. Wash the butanol solution three times with 20 ml portions of dilute hydrochloric acid, discarding the washing each time. Dilute each butanol solution to 50 ml, take 10 ml aliquot from each and

dilute to 50 ml with butanol. Add 0.5 ml of a freshly prepared 2 percent stannous chloride solution to each and shake. If the butanol extracts are turbid, wash with 10 ml of dilute hydrochloric acid. Transfer the butanol extracts to the Nessler cylinders. The limit prescribed for silicate in Table 1 shall be taken as not having been exceeded if the intensity of blue colour produced in the test with material is not greater than that produced in the control test.

### **B-6.3 Spectrophotometric Method for the Determination of Phosphate**

#### **B-6.3.1 Apparatus**

##### **B-6.3.1.1 Nessler cylinders**

#### **B-6.3.2 Reagents**

##### **B-6.3.2.1 Standard phosphate solution**

Dissolve 0.165 g potassium dihydrogen phosphate dried at 105°C, in water, dilute to 1000 ml. Add 5 ml of  $\text{CHCl}_3$  (1 ml = 0.5 mg  $\text{PO}_4$ ). Prepare standard solution from the above stock having 1 ml = 0.01 mg  $\text{PO}_4$ .

##### **B-6.3.2.2 Strong acid solution**

Cautiously add 300 ml concentrated sulphuric acid to 600 ml water. Cool and add 4.0 ml concentrated nitric acid and dilute to 1000 ml.

##### **B-6.3.2.3 Phenolphthalein indicator**

##### **B-6.3.2.4 Ammonium molybdate solution**

a) Dissolve 31.4 g ammonium molybdate in 200 ml of water.

b) Cautiously add 252 ml of concentrated  $\text{H}_2\text{SO}_4$  to 400 ml of water, mix and cool, add 3.4 ml concentrated  $\text{HNO}_3$ . Add solution (a) to solution (b) and dilute to 1000 ml.

##### **B-6.3.2.5 Amino-naphthol sulphonic acid**

Weigh out separately 0.75 g 1-amino-2-naphthol-4-sulphonic acid, 42 g sodium sulphite and 70 g sodium metabisulphite. Pulverize the amino-naphthol sulphonic acid with a small proportion of sodium metabisulphite in a clean dry mortar. Dissolve the remaining sodium metabisulphite and sodium sulphite in 900 ml water, add to it ground amino-naphthol sulphonic acid — sodium metabisulphite mixture and stir to dissolve. Dilute to 1 l. Store in an amber-coloured bottle.

NOTE — This solution is not stable. Do not keep longer than two weeks.

#### **B-6.3.3 Calibration Curve**

Take 2, 4, 6, 8 and 10 ml standard solutions (1 ml = 0.01 mg) in Nessler cylinders and add 2.0 ml ammonium molybdate, mix well and after 5 min add 2.0 ml amino-naphthol sulphonic acid and dilute to 50 ml mark. Run a reagent blank. Measure the percentage transmittance at 660 nm using 10 mm cell and adjusting the blank at 100 percent transmittance. Draw a calibration curve of O. D. against concentration of  $\text{PO}_4$ .

#### **B-6.3.4 Procedure**

Take a suitable aliquot containing 0.02 mg  $\text{PO}_4$  or more, evaporate on steam bath until reduced to 1 ml. Transfer quantitatively with water into a 100 ml Nessler cylinder. Add phenolphthalein indicator. If pink colour develops, neutralize it with acid. Add 2.0 ml ammonium molybdate reagent and mix well. After 5

min add 20 ml amino naphthol sulphonic acid and dilute to 50 ml mark, mix well. After 5 min measure the percentage transmittance at 660 nm and from the graph find out mg PO<sub>4</sub>.

### B-6.3.5 Calculation

$$\text{Phosphates ( as PO}_4\text{ ), percent by mass} = \frac{A \times 100}{V}$$

where

A = mg/l of PO<sub>4</sub> from the calibration curve, and

V = volume in ml of aliquot of the sample taken for the test.

## B-6.4 Spectrophotometric Method for the Determination of Silicate

### B-6.4.1 Apparatus

#### B-6.4.1.1 Spectrophotometer

#### B-6.4.1.2 Nessler cylinders

### B-6.4.2 Reagents

**B-6.4.2.1 Sodium bicarbonate** — powder.

**B-6.4.2.2 Dilute sulphuric acid** — 1 N.

**B-6.4.2.3 Dilute hydrochloric acid** — 1 : 1 (v/v).

#### B-6.4.2.4 Ammonium molybdate solution

Dissolve 10 g ammonium molybdate in water by warming and dilute to 100 ml. Filter if necessary. Adjust pH 7 to 8 with silica free ammonia or sodium hydroxide. Store the reagent in polyethylene bottle to stabilize.

**B-6.4.2.5 Oxalic acid solution** — 10 percent.

#### B-6.4.2.6 Standard silica solution

Dissolve 4.73 g sodium metasilicate nonahydrate (Na<sub>2</sub>SiO<sub>3</sub>·9H<sub>2</sub>O) in water and dilute to about 900 ml. Check the concentration and adjust the solution to contain 1000 mg/l SiO<sub>2</sub>.

Dilute 10 ml of this solution to 1000 ml. One millilitre of the solution is equivalent to 0.01 mg SiO<sub>2</sub>.

#### B-6.4.2.7 Reducing agent

Dissolve 500 mg 1-amino-2-naphthol-4- sulphonic acid and 1 g of sodium sulphite in 50 ml water and add this to a solution of 30 g sodium hydrogen sulphite in 150 ml water, filter into a plastic bottle.

### B-6.4.3 Calibration Curve

Take 2.5, 5, 10, 15 and 20 ml standard solution (1 ml = 0.01 mg SiO<sub>2</sub>). Add to each 10 ml × (1 : 1) HCl and 20 ml ammonium molybdate reagent. Mix well and allow to stand for 5 to 10 min. Add 5 ml oxalic acid and mix. Wait for 2 min and then add 2.0 ml reducing agent. Make up volume to 50 ml. Run a reagent blank. After 5 min measure percentage transmittance at 660 wavelength adjusting blank at 100 percent transmittance. Draw a calibration curve of O.D. against mg SiO<sub>2</sub>.

**B-6.4.4 Procedure**

Take 10 g of the sample and evaporate on steam bath until the volume reduces to 1 ml. Transfer quantitatively with water into a 100 ml Nessler cylinder and proceed as above (calibration curve). Find mg SiO<sub>2</sub> from the graph.

**B-6.4.5 Calculation**

$$\text{Silicate (SiO}_2\text{), percent by mass} = \frac{A \times 100}{M}$$

where

A = mg of SiO<sub>2</sub> from the calibration curve, and

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

**B-7 DETERMINATION OF SULPHATE****B-7.1 Apparatus**

**B-7.1.1 Nessler Cylinders** — 50 ml capacity.

**B-7.2 Reagents**

**B-7.2.1 Ordinary Denatured Spirit**

**B-7.2.2 Dilute Hydrochloric Acid** — 1 N approximately.

**B-7.2.3 Barium Chloride Solution**

Prepare by dissolving 12 g of barium chloride crystals in water and making up to 100 ml.

**B-7.2.4 Standard Sulphate Solution**

Dissolve 0.148 g of sodium sulphate in water and make up the solution to 1000 ml. One millilitre of the solution contains 0.1 mg of sulphate (as SO<sub>4</sub>).

**B-7.3 Procedure**

Accurately weigh 50g of the material and evaporate on a steam bath until reduced to 1 ml, dilute with 40 ml of water and 10 ml of denatured spirit, and add 1 ml of dilute hydrochloric acid. Mix and add 1 ml of barium chloride solution, mix immediately. Carry out a control test in the other Nessler cylinder by taking 1 ml of standard sulphate solution (**B-7.2.4**), 10 ml of denatured spirit, 40 ml of water and 1 ml of dilute hydrochloric acid; mix, add 1 ml of barium chloride solution and mix immediately. Allow both the solutions to stand for 1 h.

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

**A-5.4 Alternative Method**

Sulphates may alternatively be determined by instrumental test method as prescribed in **B-16**.

**B-8 TEST FOR SULPHIDE**

**B-8.1 Reagent**

**B-8.1.1** *Potassium Plumbite* — sulphide-free.

**B-8.2 Procedure**

Take 10 g (11 ml) of the material, add 2 drops of potassium plumbite solution. No darkening in colour shall be produced. For comparison, take only test solution in another Nessler cylinder.

**B-9 DETERMINATION OF ARSENIC****B-9.1 Procedure**

Take a suitable quantity of the material and evaporate on steam bath so that the test solution contains 1 to 10  $\mu\text{g}$  of arsenic in a final volume of about 5 ml. Determine arsenic by silver diethyl dithiocarbamate method as given in IS 2088.

**A-5.4 Alternative Method**

Arsenic may alternatively be determined by instrumental test method as prescribed in **B-17**.

**B-10 DETERMINATION OF CALCIUM AND MAGNESIUM****B-10.1 Reagents****B-10.1.1** *Ammonia-Ammonium Chloride Buffer Solution*

Dissolve 67.5 g of ammonium chloride in 300 ml of water, add 570 ml of strong ammonia solution and mix. Make up the volume to 1000 ml with water.

**B-10.1.2** *Sodium Sulphide Solution* — M/2 approximately.**B-10.1.3** *EDTA Solution* — M/100.

Dissolve 4.0 g of ethylene diamine tetra acetic acid disodium salt in water, add 20 ml of 1 N sodium hydroxide solution, mix, and dilute to 1000 ml with water.

**B-10.1.4** *Methylthymol Blue Indicator*

Grind 0.1 g of methylthymol blue with 10 g of potassium nitrate. Add 25 to 50 mg of this mixture to 50 ml of the solution to be titrated.

**B-10.2 Procedure**

Weigh 80 g (89 ml) of the material and evaporate on a steam bath until reduced to 1 ml. Dilute to 100 ml with water and add 20 ml of ammonia-ammonium chloride buffer solution, a further 20 g (23 ml) of sample and 5 drops of sodium sulphide solution. Titrate with 0.01 M EDTA, using methylthymol blue as indicator until the blue solution becomes colourless or grey.

**B-10.2.1** The material shall be taken to have passed the test if not more than 1 ml of 0.01 M EDTA is required for titration.

**B-10.3 Alternative Method**

Calcium and magnesium may alternatively be determined by instrumental test method as prescribed in **B-17**.

**B-11 DETERMINATION OF COPPER****B-11.1 Apparatus**

**B-11.1.1** *Nessler Cylinders* — 50 ml capacity.

**B-11.2 Reagents**

**B-11.2.1** *Dilute Sulphuric Acid* — approximately 5 N.

**B-11.2.2** *Diethylammonium Diethyldithiocarbamate Solution* — 0.1 percent (m/v) in carbon tetrachloride.

**B-11.2.3 Standard Copper Solution A**

Accurately weigh 3.93 g of cupric sulphate dissolve in 250 ml of water and add 20 ml of sulphuric acid, make the volume to 1000 ml with water. One millilitre of the solution contains 1.0 mg of copper (as Cu).

**B-11.2.3.1 Standard copper solution B**

Dilute 10 ml of standard copper solution A (**B-11.2.3**) to 1000 ml with water. One millilitre of this solution contains 0.01 mg of copper (as Cu).

**B-11.3 Procedure**

Accurately weigh 100 g (110 ml) of the material, evaporate on a steam bath until volume is reduced to 2 ml, then dilute with 20 ml of water and add 5 ml of dilute sulphuric acid. Extract the solution with 10 ml of a 0.1 percent solution of diethyl ammonium diethyldithiocarbamate in carbon tetrachloride by vigorous shaking. Carry out a control test in the other Nessler cylinder by taking 1 ml of standard copper solution (**B-11.2.3.1**), 20 ml of water, 5 ml of dilute sulphuric acid, and 10 ml of 0.1 percent solution of diethylammonium diethyldithiocarbamate in carbon tetrachloride.

**B-11.3.1** The limit prescribed for copper in Table 1 shall be taken as not having been exceeded if the intensity of any yellow colour produced in the organic layer, in the test with the material, is not greater than that produced in the control test.

**A-11.4 DETERMINATION OF COPPER USING INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY (ICP-OES) METHOD**

**A-11.4.1** Copper can be determined using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) method as prescribed in IS 3025 (Part 2).

**B-12 DETERMINATION OF IRON**

**B-12.1** Two methods are prescribed for the determination of iron, namely, colorimetric and spectrophotometric methods. The spectrophotometric method shall be regarded as the referee method.

**B-12.2 Colorimetric Method****B-12.2.1 Apparatus**

**B-12.2.1.1** *Nessler cylinders* — 50 ml capacity.

**B-12.2.2 Reagents**

**B-12.2.2.1** *Dilute Hydrochloric Acid* — approximately 5 N.

**B-12.2.2.2 Potassium Permanganate Solution** — approximately 0.1 N.

**B-12.2.2.3 Ammonium Thiocyanate Solution** — Dissolve 57.0 g in 100 ml of water.

**B-12.2.2.4 Mixture of Amyl Alcohol and Amyl Acetate** — 1 : 1 (v/v).

**B-12.2.2.5 Standard Iron Solution A**

Dissolve 0.702 g of ammonium ferrous sulphate in 250 ml of water and 15.0 ml of sulphuric acid, make up the volume to 1000 ml with water. One millilitre of this solution contains 0.1 mg of iron (as Fe).

**B-12.2.2.6 Standard iron solution B**

Dilute 50 ml of standard iron solution A (**B-12.2.5**) to 500 ml with water. This solution should be prepared just before use. One millilitre of this solution contains 0.01 mg of iron (as Fe).

**B-12.2.3 Procedure**

Accurately weigh 50 g (57 ml) of the material and evaporate on a steam bath until volume is reduced to about 15 ml, then add 3 ml of dilute hydrochloric acid and boil for 2 min in the same vessel. Add 1 drop of potassium permanganate solution and mix. Transfer to a Nessler cylinder. Add 5 ml of ammonium thiocyanate solution and 10 ml of a mixture of equal volumes of amyl alcohol and amyl acetate, shake vigorously and allow to separate. Carry out a control test in the other Nessler cylinder as follows. Take 1 ml of standard iron solution B (**B-12.2.6**) and 1 ml of dilute hydrochloric acid and dilute with water to the same volume as the acidified solution of the test sample, add 1 drop of potassium permanganate and from this point follow the procedure describe above.

**B-12.2.3.1** The limit prescribed n table 1 shall be taken as not having been exceeded if the red colour in the organic layer produced in the material is not greater than that produced in the control test.

**B-12.3 Spectrophotometric Method**

**B-12.3.1 Apparatus**

**B-12.3.1.1 Spectrophotometer**

**B-12.3.2 Reagents**

**B-12.3.2.1 Dilute Sulphuric Acid Solution** — 1:1 (v/v).

**B-12.3.2.2 Hydroxylamine Hydrochloride Solution** — 10 percent.

**B-12.3.2.3 Ammonium Acetate Solution** — 20 percent.

**B-12.3.2.4  $\alpha$ - $\alpha$ -Dipyridyl Solution** — 1 percent in alcohol.

**B-12.3.2.5 Ammonia Solution (R. D. 0.93)** — 18 percent.

**B-12.3.2.6 Standard Iron Solution A**

Dissolve 0.702 g of ferrous ammonium sulphate [ $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ] in small amount of water. Add 5 ml of concentrated  $\text{H}_2\text{SO}_4$  and dilute with water up to the mark in 1000 ml volumetric flask, mix well 1 ml = 0.1 mg Fe).

**B-12.3.2.7 Standard iron solution B**

Transfer 10 ml of this solution to 1000 ml volumetric flask and dilute to the mark with water, mix well ( 1 ml = 0.001 mg = 1 µg Fe ).

### **B-12.3.3 Calibration Curve**

Transfer 2, 5, 10, 15 and 20 ml of standard iron solution in 5 different 100 ml volumetric flasks. Add to each flask 2 ml of ( 1 : 1 ) sulphuric acid, 2 ml of 10 percent hydroxylamine hydrochloride

Shake and wait for 5 min. Add 100 ml of 20 percent ammonium acetate solution and 1 ml of 1 percent  $\alpha$ - $\alpha$ -dipyridyl solution. Then add 3.5 ml of 18 percent ammonia solution and dilute to the mark, shake well. Run a reagent blank. Allow to stand for 30 min and measure percentage transmittance at 530 nm using 10 mm cell and adjusting blank at 100 percent transmittance. Draw a calibration curve of O.D. against concentration of iron.

### **B-12.3.4 Procedure**

Accurately weigh 50 g (57 ml) of the sample and reduce the volume to 15 ml evaporating on steam bath. Transfer the material quantitatively with small amount of water into 100 ml volumetric flask. Add 2 ml ( 1 : 1 ) sulphuric acid 2 ml of hydroxylamine hydrochloride, shake and allow to stand for 5 min. Add 10 ml of ammonium acetate solution, mix and add 1 ml of  $\alpha$ - $\alpha$ -dipyridyl solution and 3.5 ml of 18 percent ammonia solution. Dilute to the mark with water. Run a reagent blank. Allow to stand for 30 min and measure percentage transmittance at 530 nm adjusting blank at 100 percent transmittance. Find out iron from the graph.

### **B-12.3.5 Calculation**

$$\text{Iron (as Fe), percent by mass} = \frac{X}{M \times 10^4}$$

where,

$X$  = µg iron from the graph, and

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

### **B-12.4 Alternative Method**

Iron may alternatively be determined by instrumental test method as prescribed in **A-17**.

## **B-13 DETERMINATION OF HEAVY METALS**

### **B-13.1 Apparatus**

**B-13.1.1 Nessler Cylinders** — 50 ml capacity.

### **B-13.2 Reagents**

**B-13.2.1 Dilute Hydrochloric Acid Solution** — approximately 5 N.

**B-13.2.2 Dilute Ammonia Solution** — 30 percent (v/v).

**B-13.2.3 Dilute Acetic Acid Solution** — 30 percent (v/v).

**B-13.2.4 Standard Lead Solution**

Dissolve 0.160 g of lead nitrate in 250 ml of water and 40 ml of nitric acid and make up the volume to 1000 ml with water. One milliliter of the solution contains 0.1 mg of lead.

#### **B-13.2.4.1 Standard solution B**

Dilute 50 ml of standard lead solution A (**B-13.2.4**) to 500 ml with water. This solution should be prepared just before use. One milliliter of this solution contains 0.01 mg of lead (as Pb).

#### **B-13.3 Procedure**

Accurately weigh 40 g (45 ml) of the material, boil until the volume is reduced to 15 ml, add 2.5 ml of dilute hydrochloric acid and boil for 2 min in the same vessel. Cool, dilute to 20 ml and add 30 ml of water and 10 g (11 ml) of the sample. Pass hydrogen sulphide through the solution for a few seconds. Carry out a control test in the other Nessler cylinder as follows. Mix 2.0 ml of the standard lead solution B (**B-13.2.4.1**) with 3 ml of dilute acetic acid. Add standard iron solution equivalent to the amount of iron present in the test solution, as per **B-12**, dilute to 40 ml with water, add 10 ml of dilute ammonia solution pass hydrogen sulphide through the solution for a few seconds.

**B-13.3.1** The material shall be taken to have not exceeded the limit given in Table 1 if the turbidity produced in the test with material is not greater than that produced in the control test.

#### **A-5.4 Alternative Method**

Lead may alternatively be determined by instrumental test method as prescribed in **B-17**.

### **B-14 TEST FOR REDUCING SUBSTANCES**

#### **B-14.1 Reagents**

**B-14.1.1 Dilute Sulphuric Acid** — approximately 5 N.

**B-14.1.2 Potassium Permanganate Solution** — 0.1 N.

#### **B-14.2 Procedure**

Weigh 10 g (11 ml) of the material, add 50 ml of dilute sulphuric acid and 1.0 ml of 0.01 N potassium permanganate solution and boil the mixture gently for 5 min.

**B-14.2.1** The material shall be taken to have passed the test if the pink colour of potassium permanganate is not completely discharged.

### **B-15 TEST FOR PYRIDINE AND HOMOLOGUES**

**B-15.1** Measure the optical density of the sample in a 1 cm quartz of silica cell at a wavelength of 252 nm, using a water blank. The optical density should not be greater than 0.045.

### **B-16 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES**

#### **B-16.1 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, 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.

## **B-16.2 Equipment**

**B-16.2.1 Anion guard column** — a protector of the separator column.

**B-16.2.2 Anion Separator column** — suitable for selective separation of ions under analysis.

**B-16.2.3 Anion Suppressor device** — Anion micromembrane suppressor is used to analyse the data  
Detector: Conductivity detector.

**B-16.2.4 Software** — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

Sample loop of 100 µl, 200 µl, 500 µl or 1000 µl be used to determine ionic concentration as per instrument manual and practice.

## **B-16.3 Reagents and Standards**

**B-16.3.1 Glass or polyethylene sample bottles.**

**B-16.3.2 Distilled water or deionized water free from the anions of interest.**

**B-16.3.3 Eluent** — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

**B-16.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid)** — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

## **B-16.4 Standard solutions**

**B-16.4.1 Chloride** — Dissolve NaCl, 1.6485 g in 1 l of reagent water

**B-16.4.2 Sulphate** — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

## **B-16.5 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 0.1-1.0 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.

### **B-16.6 Procedure**

Dissolve between 1 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 (0.1-1.0 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 and deviation of retention force shall not exceed  $\pm 10$  percent of RT of calibration. 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 clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

### **B-16.7 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.

## **B-17 DETERMINATION OF ARSENIC, CALCIUM, IRON, LEAD AND MAGNESIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**

### **B-17.1 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-17.2 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.

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

Sl. No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing ( $\mu\text{g}$ )	Axial viewing ( $\mu\text{g}$ )	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti Cr, Fe
		283.305	(70)		
		217.00		(20)	
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	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
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr
vi)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti

**B-17.3 Reagents and Solutions****B-17.3.1 Nitric acid (65 percent) Suprapure****B-17.3.2 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 1000  $\mu\text{g}/\text{ml}$  of arsenic, calcium, iron, lead and magnesium, in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

**B-17.3.3 Standard solution**

Pipette out 5 ml from 100  $\mu\text{g}/\text{ml}$  standard stock solution into a 100 ml volumetric flask & make up volume with 2 percent nitric acid to prepare 5  $\mu\text{g}/\text{ml}$  solution. From this 5  $\mu\text{g}/\text{ml}$  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  $\mu\text{g}/\text{ml}$  solution of respective elements under reference.

**B-17.3.4 Sample preparation**

Weigh about 2.5 g polyphosphoric acid 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-17.3.5 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, polytetrafluorethylene (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-17.4 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-17.5 Procedure**

##### **B-17.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-17.5.2**). 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-17.5.2** 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  $\pm 5$  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  $\mu\text{g/ml}$  of the arsenic, calcium, Iron, lead and 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-17.6 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).

**ANNEX C**  
*(Clause 6.1)*  
**SAMPLING OF AMMONIA, LIQUOR**

**C-1. GENERAL REQUIREMENTS**

**C-1.1** In drawing samples, the following precautions and directions shall be observed.

**C-1.2** Ammonia, liquor, is volatile and highly toxic and requires care in sampling.

**C-1.2.1** It is advisable that the sampler wears goggles to protect the eyes and observes the other precautions prescribed in IS 4544.

**C-1.3** Samples shall not be taken in an exposed place.

**C-1.4** The sampling instruments shall be clean and dry when used.

**C-1.5** Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

**C-1.6** Use care in opening the container as a portion of the contents may blow out violently due to pressure of ammonia gas. Cool the container before opening, especially during warm season. Hold it away from the eyes and then slowly remove the cap or stopper.

**C-1.7** The samples shall be placed in clean, dry and air-tight screw cap polyethylene bottles.

**C-1.8** The sample containers shall be of such a size that they have about 10 percent ullage when filled with the material.

**C-1.9** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and year of manufacture of the material.

**C-1.10** Samples shall be stored in a cool place.

**C-2 SCALE OF SAMPLING**

**C-2.1 Lot**

All containers in a single consignment of the material of the same grade and drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such batch shall constitute a lot.

**C-2.2** For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested for each lot separately.

**C-2.3** The number of containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with Table 3.

**Table 3 Number of Containers to be Selected for Sampling**

Lot Size (1)	Number of Containers to be Selected (2)
Up to 15	2
16 to 25	3
26 to 50	4
51 to 100	5
101 to 300	6
301 to 500	7
501 to 800	8
801 to 1300	9
1301 and above	10

**C-2.3.1** These containers shall be selected at random. In order to ensure the randomness of selection the procedures given in IS 4905 shall be followed.

### **C-3 TEST SAMPLES AND REFEREE SAMPLE**

**C-3.1** From each of the containers selected according to **C-2.3**, representative portion of the material shall be taken with the help of an appropriate sampling instrument after thoroughly mixing the contents of the container.

**C-3.2** A small but approximately equal quantity shall be taken from each of these portions and well mixed to prepare a composite sample, the quantity of which shall not be less than 3.0 kg. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**C-3.3** The remaining portion of the material from each container shall be divided into three equal parts, each constituting an individual sample. One set of individual samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

**C-3.4** To prevent loss of ammonia when transferring samples from one container to another, all the containers selected for sampling and the receiving containers shall be cooled in ice or cold water-bath at 5 to 10°C. All transfers of the samples shall be done as quickly as possible.

**C-3.5** All the individual and composite samples shall be transferred immediately to thoroughly dried screw cap polyethylene bottles. These bottles shall be sealed and labelled with full identification particulars.

**C-3.6** The referee sample consisting of a composite sample and a set of individual samples shall bear the seals of purchaser and supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of a dispute.

### **C-4 NUMBER OF TESTS**

**C-4.1** Ammonia content shall be tested on each of the individual samples.

**C-4.2** Tests for the determination of the remaining characteristics given in Table 1 shall be performed on the composite sample.

## **C-5 CRITERIA FOR CONFORMITY**

### **C-5.1 Individual Samples**

The lot shall be considered to have satisfied the requirements of ammonia if each of the individual samples tested for ammonia, satisfies the corresponding requirement given in Table 1 for the relevant grade.

### **C-5.2 Composite Samples**

The lot shall be considered to have met the requirement for remaining characteristics given in Table 1 if all the test results on the composite sample satisfy the corresponding requirements given in Table 1.

**C-5.3** The lot shall be declared as conforming to the requirements of this specification if **C-5.1** and **C-5.2** are satisfied.