

भारतीय मानक मसौदा
गंधक का अम्ल — विशिष्टि
(IS 266 का पांचवां पुनरीक्षण)

Draft Indian Standard
Sulphuric Acid — Specification
(*Fifth Revision of IS 266*)

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ICS 71.080.50

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 01.02.2025

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(Formal clause will be added later)

This standard was first published in 1950 and subsequently revised in 1961, 1977, 1993 and 2024. In the third revision, the requirements for iron content for the battery grade acid, oxidizable impurities for the analytical reagent grade, antimony and platinum were suitably amended. The method for determination of oxidizable impurities was modified. Suitable changes were also made in the packing clause.

In the fourth revision, instrumental test methods for the determination of chlorides, nitrates, selenium, copper, iron, lead, arsenic and manganese were added as alternate test methods. Also, a new spectroscopic method for copper analysis was incorporated. Further, reference clause and packing & marking clause were updated and Amendment No.1 was incorporated.

In this revision, some editorial corrections have been done. Also, a new test method for the determination of heavy metal (as lead) has been suitably incorporated.

For general information regarding precautions to be observed in safe handling and use of sulphuric acid, reference may be made to IS 4262 'Code of safety for sulphuric acid'.

In the preparation of this standard, reference has been made to the following ISO standards:

ISO/910-1977 Sulphuric acid and oleum for industrial use — Determination of total acidity and calculation of free sulphur trioxide content of oleum, titrimetric method.

ISO/R 915-1968 Sulphuric acid and oleum for industrial use — Determination of iron content — 2, 2'-bipyridyl spectrophotometric method.

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
SULPHURIC ACID — SPECIFICATION
(Fifth Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sulphuric acid.

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

3.1 There shall be four grades of the material, namely:

- a) Technical (Tech);
- b) Battery;
- c) Chemically pure (CP); and
- d) Analytical reagent (AR).

3.1.1 The battery grade acid shall have two sub-grades, namely, concentrated and dilute.

4 REQUIREMENTS**4.1 Description****4.1.1 Technical Grade**

Sulphuric acid of technical grade shall be a liquid not darker than brown in colour.

4.1.2 Battery Grade (Concentrated and Dilute)

Sulphuric acid of battery grade shall be a colourless liquid. The concentrated acid on dilution with an equal volume of distilled water, and the dilute acid as received, shall be free from suspended matter and other visible impurities.

4.1.3 Chemically Pure and Analytical Reagent Grades

Sulphuric acid of chemically pure and analytical reagent grades shall be a clear and colourless liquid, free from suspended matter and other visible impurities.

4.2 The material shall also 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 (8) of the Table 1.

4.3 Optional Requirements**4.3.1 Technical Grade**

Subject to agreement between the purchaser and the supplier, technical grade acid complying with all the requirements given in col (3) of Table 1 except that for total acidity, may also be supplied as more dilute or more concentrated acid.

4.3.2 Battery Grade

4.3.2.1 Subject to agreement between the purchaser and the supplier, battery grade concentrated acid complying with all the requirements given in col (4) of Table 1 except that for total acidity, may also be supplied as more dilute or more concentrated acid.

4.3.2.2 Subject to agreement between the purchaser and the supplier, requirements for battery grade acid (concentrated and dilute) may include, in addition to the requirements given in Table 1, limits for antimony and platinum as follows:

- a) When tested according to the method prescribed in **B-17**, the concentrated acid shall contain, not more

than 1 ppm, and the dilute acid not more than 0.3 ppm of antimony (as Sb).

- b) When tested according to the method prescribed in **B-18**, the concentrated acid shall contain, not more than 0.2 ppm and the dilute acid not more than 0.06 ppm of platinum (as Pt).

Table 1 Requirements for Sulphuric Acid

(Clauses 4.2, 4.3.1, 4.3.2.1, 4.3.2.2, B-5.2.3.1, B-6.3.3.1, B-10.2.2.1, B-11.2.1, B-12.2.2.1.1, B-13.2.2.1.1, B-14.2.3, B-16.3.1, C-3.1, C-5.2, C-5.3 and C-5.4)

Sl No	Characteristic	Requirement				Method of Test, (Ref to Clause No. in Annex B)	
		Technical Grade	Battery Grade		Chemically Pure Grade		Analytical Reagent Grade
			Concentrated	Dilute			
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	Total acidity (as H ₂ SO ₄), percent by mass, <i>Min</i>	98.0	95.0	30.0	98.0	98.0	B-2
ii)	Residue on ignition, percent by mass, <i>Max</i>	0.05	0.06	0.02	0.02	0.002	B-3
iii)	Iron (as Fe), percent by mass, <i>Max</i>	0.01	0.003	0.001	0.001	0.000 05	B-4
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	—	0.001	0.000 3	0.003 5	0.000 02	B-5
v)	Heavy metals (as lead), percent by mass, <i>Max</i>	0.005	—	—	0.002	0.000 1	B-6
vi)	Arsenic (as As), percent by mass, <i>Max</i>	0.004	0.000 12	0.000 04	0.000 2	0.000 005	B-7
vii)	Oxidizable impurities (as SO ₂), percent by mass, <i>Max</i>	—	0.02	0.02	0.004	0.000 4	B-8
viii)	Organic matter	—	To pass test	—	—	—	B-9
ix)	Nitrates (as NO ₃), ppm, <i>Max</i>	—	—	—	—	0.2	B-10
x)	Ammonia (as NH ₃), ppm, <i>Max</i>	—	—	—	—	2	B-11
xi)	Selenium (as Se), percent by mass, <i>Max</i>	—	0.002	0.000 6	—	—	B-12
xii)	Manganese (as Mn), ppm, <i>Max</i>	—	1	0.3	—	—	B-13

xii)	Copper (as Cu), percent by mass, <i>Max</i>	—	0.003	0.001	—	—	B-14
xiv)	Zinc (as Zn), percent by mass, <i>Max</i>	—	0.003	0.001	—	—	B-15
xv)	Nitrates, nitrites and ammonia (as N), percent by mass, <i>Max</i>	—	0.003	0.001	0.003	—	B-16

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in containers as agreed to between the purchaser and the supplier and subject to the provisions of Red Tariff No. 18 of 1960 issued by the Indian Railway Conference Association, with any alteration or addition made thereafter.

5.1.2 When sulphuric acid is supplied in polyethylene containers or screw-stoppered stoneware bottles or glass carboys or in cans, the containers shall be fitted with leak-tight stoppers.

5.1.3 Sulphuric acid of CP and AR grades shall be supplied in polyethylene containers or in glass bottles or glass carboys, fitted with tight TEFLON washers (of thickness 0.02 mm) and stoppered with HDPE or other suitable stoppers. The use of HDPE or other suitable caps over the stopper is recommended.

5.1.4 When steel drums are used for transport by rail of acid of relative density not less than 1.74, these shall comply with the requirements of the specifications for steel drums prescribed in Red Tariff No. 18 of 1960 issued by the Indian Railway Conference Association, with any alteration or addition made thereafter.

5.1.5 These shall be placed in an upright position on one layer of sand or ashes free from cinders, or chalk, or dry earth and the empty surrounding-space shall also be filled with the same material to prevent movement or they may be packed in thermocol packing (up to 2.5 litres).

5.1.6 In case of dilute sulphuric acid of relative density not more than 1.216, certified in writing by the consignor, the inside packing of the case may be straw or grass or wood shavings or the packing may be thermocol and the mass of each case shall not exceed 130 kg.

5.2 Marking

5.2.1 The containers up to 12 litre capacity and also the packages shall be suitably marked in red letters, the height of the letters shall be such as to be visible with the naked eye. The drums and tank wagons shall be marked with the following information:

- a) Marked in red letters not less than 5 cm high;
- b) Name and grade of the material;
- c) Name of the manufacturer and address;
- d) Net weight of contents;
- e) Lot number;
- f) Expiry date; and
- g) They shall prominently display the words 'CORROSIYE, HANDLE WITH CARE'.

5.2.2 The data of chemical analysis shall also be shown on the containers of the analytical reagent grade of the material.

5.2.3 The packages shall be labelled as shown in Fig. 15 of IS 1260 (Part 1).

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

The method of drawing representative samples of the material shall be as prescribed in Annex C.

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070: 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
IS 1388 (Part 1) : 2019	Laboratory glass ware — Bottles: Part 1 Screw neck bottles (<i>third revision</i>)
IS 2088 : 2023	Methods for determination of arsenic (<i>third revision</i>)
IS 3025 (Part 2) : 2019	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>)
IS 3025 (Part 56) : 2003	Methods of sampling and test (physical and chemical) for water and wastewater: Part 56 Selenium (<i>first revision</i>)
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 (<i>first revision</i>)
IS 4905 : 2015/ ISO 24153 : 2009	Methods for random sampling (<i>first revision</i>)
IS 5296 : 2024	Chloroform, pure and technical — Specification (<i>third revision</i>)
IS 7017 : 1973	Test method for colorimetric determination of traces of heavy metals by dithizone

ANNEX B

(Clauses 4.2)

METHODS OF TEST FOR SULPHURIC ACID

B-1 QUALITY OF REAGENTS

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

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

B-2 DETERMINATION OF TOTAL ACIDITY

B-2.1 General

The material is titrated against standard solution of sodium hydroxide using methyl red as indicator.

B-2.2 Apparatus

B-2.2.1 Lunge-Rey Pipette

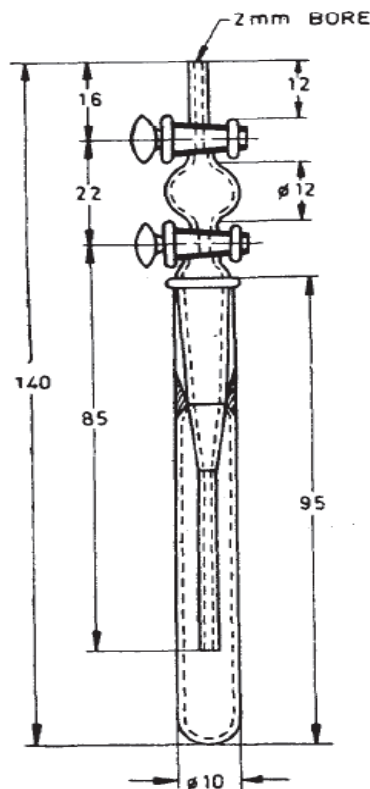
Lunge-Rey pipette shall be of the shape and dimensions shown in Fig. 1. If this pipette is not available, a weighing bottle, or a glass ampoule of the type shown in Fig. 2 may be used.

B-2.3 Reagents

B-2.3.1 *Standard Sodium Hydroxide Solution*, 1 N — freshly standardized

B-2.3.2 *Methyl Red Indicator*

Dissolve 1 g of methyl red in 95 percent (v/v) ethanol and dilute to 100 ml with the same ethanol.



All dimensions in millimetres

FIG. 1 LUNGE RAY PIPETTE

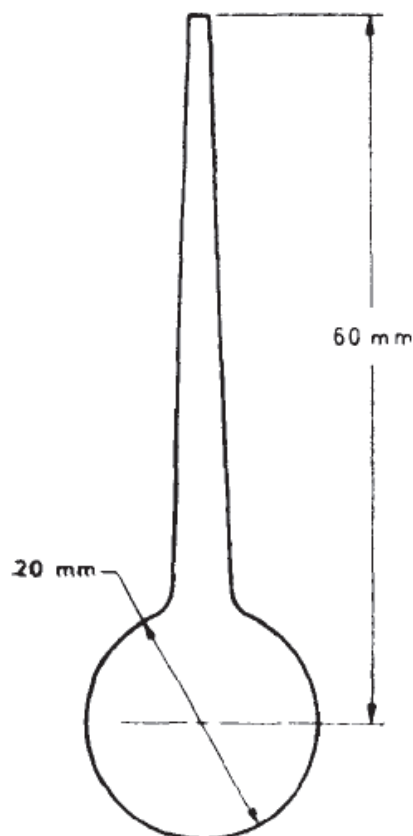


FIG. 2 SPHERICAL GLASS AMPOULS

B-2.4 Procedure

B-2.4.1 Sulphuric Acid Content Higher than 98 Percent by Mass

Accurately weigh about 2 g of material with Lunge-Rey pipette or weighing bottle or glass ampoule. If the glass ampoule is used, carefully mix the test sample by shaking the container. If the acid is partially crystallized, slightly heat the container until the sample is dissolved, then carefully mix again. Take the sample in a beaker or flask and slightly heat the bulb of the glass ampoule in a flame previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule into the flask containing the test sample and ensure that the bulb is filled up to about two-thirds of its volume during cooling (2 ml to 3 ml approximately). Withdraw the ampoule and carefully wipe the capillary end with filter paper. Seal the capillary end in an oxidizing flame, without loss of glass. Remove from the flame and allow the ampoule to cool to room temperature. Wash the capillary and wipe carefully with filter paper. Weigh the ampoule to the nearest 0.1 mg and calculate by difference the mass of the test portion.

Carefully place the ampoule containing the test portion into the conical flask containing 300 ml of cold water. Stopper the flask and shake to break the ampoule containing the test portion. Cool the flask during this operation. Keep cooling and shaking until the vapours are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the conical flask. By means of a glass rod, grind the fragments of the ampoule and in particular the capillary which may have remained intact in spite of shaking. Withdraw the glass rod and wash it with water, collecting the washings in the conical flask. Add two drops of methyl red indicator solution and titrate to the end point with standard sodium hydroxide solution.

B-2.4.2 Sulphuric Acid Content Equal to or Lower than 98 Percent by Mass

Weigh accurately as in B-2.4.1, about 2 g of the sample and transfer to a 500 ml conical flask containing approximately 300 ml of water. Add two drops of methyl red indicator solution and titrate to the end point with standard sodium hydroxide solution.

B-2.5 Calculation

$$\text{Total acidity (as H}_2\text{SO}_4\text{), percent by mass} = \frac{V \times N \times 4.904}{M}$$

where

V = volume in ml, of standard sodium hydroxide solution used for the titration;

N = normality of standard sodium hydroxide solution; and

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

B-3 DETERMINATION OF RESIDUE ON IGNITION

B-3.1 Procedure

Weigh to the nearest 10 mg about 50 g of the sample in a platinum or silica dish of 100 ml capacity, previously ignited at $800\text{ }^\circ\text{C} \pm 50\text{ }^\circ\text{C}$, cooled in a desiccator and weighed. Evaporate the acid carefully on a sand-bath, heating the dish containing the test portion. Heat to dryness. Place the dish containing the residue in an electric furnace heated at $800\text{ }^\circ\text{C} \pm 50\text{ }^\circ\text{C}$ and keep at this temperature for about 15 min. Remove the dish from the furnace, cool in a desiccator and weigh. Repeat heating, cooling and weighing till constant mass is obtained.

B-3.1.1 Preserve the residue obtained from the battery grade concentrated and dilute acids for the test prescribed in **B-14**.

B-3.2 Calculation

$$\text{Residue on ignition, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g, of the residue weighed; and

M_2 = Mass in g, of the sample taken for the test.

B-4 DETERMINATION OF IRON

B-4.1 Four methods are prescribed for determining iron, namely, Method A (Bipyridyl method), Method B (Thiocyanate method), ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-4.2 Method A (Bipyridyl Method)

B-4.2.1 General

After evaporation, iron is reduced by means of hydroxyl ammonium chloride, and the colour developed with 2, 2'-bipyridyl solution is measured by a photometer at a wavelength of about 522 nm.

B-4.2.2 Apparatus

B-4.2.2.1 Photometer

Any spectrophotometer suitable for measurement at a wavelength of about 522 nm or photoelectric colorimeter.

B-4.2.3 Reagents

B-4.2.3.1 Dilute hydrochloric acid — approximately 1N

B-4.2.3.2 Hydroxylammonium chloride solution

Dissolve 10 g of hydroxylammonium chloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in water and dilute to 100 ml

B-4.2.3.3 Ammonium acetate solution — 30 percent (m/v)

B-4.2.3.4 2, 2'-Bipyridyl solution

Dissolve 1 g of 2, 2'-bipyridyl in 10 ml of dilute hydrochloric acid and dilute to 100 ml

B-4.2.3.5 Standard iron solution A

Dissolve 0.7022 g of ferrous ammonium sulphate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water in a 1 000 ml volumetric flask, add 4 ml of concentrated sulphuric acid and make up the volume to the mark with water. One millilitre of this solution contains 0.1 mg of iron (as Fe).

B-4.2.3.6 Standard iron solution B

Take 100 ml of standard iron solution A and dilute to 1 000 ml with water in a 1 000 ml volumetric flask. One millilitre of this solution contains 0.01 mg of iron (as Fe). This solution should be prepared fresh.

B-4.2.4 Procedure

B-4.2.4.1 Preparation of calibration curve

Into each of a series of eleven 100 ml volumetric flasks, place the quantities of standard iron solution B as indicated below:

<i>Sl No.</i>	<i>Standard Iron Solution B</i> <i>ml</i>	<i>Corresponding to Fe</i> <i>µg</i>
(1)	(2)	(3)
i)	0 (compensation solution)	0
ii)	5.0	50
iii)	10.0	100
iv)	15.0	150
v)	20.0	200
vi)	25.0	250
vii)	30.0	300
viii)	35.0	350
ix)	40.0	400
x)	45.0	450
xi)	50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to approximately 50 ml, then 2 ml of hydrochloric acid, 2 ml of hydroxyl ammonium chloride solution and after 5 min, 5 ml of ammonium acetate solution followed by 1 ml of 2, 2'-bipyridyl solution. Dilute to the mark, mix thoroughly and wait for 10 min. Carry out the measurement on the spectrophotometer with a 1 cm cell at a wavelength of about 522 nm (or on a photo electric colorimeter using appropriate filter) adjusting the instrument to zero optical density, using as reference the compensation solution. Prepare a calibration graph with the iron content in micrograms per 100 ml of the standard matching solution as abscissa and the corresponding values of optical density as ordinate.

B-4.2.4.2 Determination

Weigh accurately about 50 g of the sample in a platinum or silica dish (100 ml capacity). Place on a sand bath and carefully evaporate to dryness. Cool, take up with 2 ml of hydrochloric acid and 25 ml of water and heat to facilitate dissolution. Transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark, mix and filter, if necessary. Transfer an aliquot of the sample solution containing between 50 µg and 500 µg of iron, to a 100 ml one-mark volumetric flask. Dilute to approximately 50 ml, if necessary, then add successively 2 ml of hydrochloric acid, 2 ml of hydroxyl ammonium chloride solution and, after 5 min, 5 ml of ammonium acetate solution and 1 ml of 2, 2'-bipyridyl solution. Dilute to the mark, mix and wait for 10 min. Carry out the spectrophotometric (or photoelectric colorimetric) measurement according to the procedure given in B-4.2.4.1, adjusting the instrument to zero optical density using as reference the blank test solution (*see* B-4.2.4.3).

B-4.2.4.3 Blank test

At the same time as the analysis, carry out a blank test using the same procedure and quantities of all reagents employed in the test.

B-4.2.5 Calculation

By reference to the calibration chart (*see B-4.2.4.1*), determine the iron content corresponding to the photometric measurement.

$$\text{Iron (as Fe) percent by mass} = \frac{m \times 100 \times 100}{V \times M}$$

where

m = mass in g, of iron determined in the aliquot of the sample solution;

V = volume in ml, of the sample solution taken for the colour reaction; and

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

B-4.3 Method B (Thiocyanate Method)

B-4.3.1 General

The colour produced by a known quantity of the material with ammonium thiocyanate is compared with a control containing known quantity of iron.

B-4.3.2 Apparatus

B-4.3.2.1 Nessler Cylinders — 50 ml capacity

B-4.3.3 Reagents

B-4.3.3.1 Hydrochloric acid — 1 : 1 (v/v)

B-4.3.3.2 Potassium permanganate solution — 0.1 N approximately

B-4.3.3.3 Ammonium thiocyanate solution — 60 percent (m/v)

B-4.3.3.4 Mixture of amyl alcohol and amyl acetate — 1 : 1 (v/v)

B-4.3.3.5 Standard iron solution B — same as in **B-4.2.3.6**

B-4.3.4 Procedure

B-4.3.4.1 For battery and chemically pure grades

Dilute exactly 1 g of the material to 10 ml with water in a Nessler cylinder. Add one drop of potassium permanganate solution and mix thoroughly. Add 5 ml of ammonium thiocyanate solution and 10 ml of amyl alcohol and amyl acetate mixture. Make up to 50 ml, shake vigorously and allow the layers to separate. Compare the intensity of any red colour produced in the upper layer with a control test carried out in another Nessler cylinder in the same manner using the following amounts of standard iron solutions in place of the sample:

- a) 3 ml of standard iron solution B in the case of battery grade concentrated acid;
- b) 1 ml of standard iron solution B in the case of battery, grade dilute acid; and
- c) 1 ml of standard iron solution B in the case of chemically pure grade acid.

B-4.3.4.2 For technical grade

Dilute exactly 1 g of the material to 100 ml. Take 10 ml of this solution and carry out the test as prescribed under **B-4.3.4.1**, but using 5 ml of standard iron solution B in the control test.

B-4.3.4.3 For analytical reagent grade

Accurately weigh 50 g of the sample in a platinum or silica dish (100 ml capacity). Place on a sand-bath and carefully evaporate to dryness. Cool, add 2 ml of hydrochloric acid and 25 ml of water and heat to facilitate dissolution. Cool and carry out the test with this solution as prescribed in **B-4.3.4.1**, but using 2.5 ml of standard iron solution B in the control test.

B-4.3.4.4 The limit prescribed for the appropriate grade in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the respective control test.

B-5 TEST FOR CHLORIDES

B-5.1 General

Two methods are prescribed for determining chlorides, namely, Method A and ion chromatography method as prescribed in **B-20**. In case of dispute, ion chromatography method shall be used as referee method.

B-5.2 Method A

B-5.2.1 Apparatus

B-5.2.1.1 Nessler cylinders — 50 ml capacity

B-5.2.2 Reagents

B-5.2.2.1 Dilute nitric acid — approximately 4 N

B-5.2.2.2 Silver nitrate solution — approximately 5 percent (*m/v*)

B-5.2.2.3 Standard chloride solution

Dissolve 1.648 g of sodium chloride, previously dried, in 1 000 ml of water. Dilute 10 ml of this solution again to 1 000 ml in a volumetric flask. One millilitre of the diluted solution is equivalent to 0.01 mg of chloride (as Cl).

B-5.2.3 Procedure

Dilute exactly 2 g (25 g in case of dilute battery and AR Grades) of the material to 50 ml with water in a Nessler cylinder. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution and mix. Carry out a control test in the other Nessler cylinder in the same manner using the following quantities of standard chloride solution:

- a) 2 ml in the case of battery grade concentrated acid;
- b) 7.5 ml in the case of battery grade dilute acid;
- c) 7 ml in the case of CP grade acid; and
- d) 0.5 ml in the case of AR grade acid.

B-5.2.3.1 The limit prescribed for the appropriate grade in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test solution is not greater than that produced in the respective control test.

B-6 DETERMINATION OF HEAVY METAL (as Pb)

B-6.1 General

Five methods are prescribed for determining heavy metal (as Pb), namely, Method A (*see* IS 7017), Method B, Method C, ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-6.2 Method B

After evaporation of a test portion, lead is reduced by hydroxylammonium chloride. Complexes are formed of interfering elements with ammonium citrate and potassium cyanide. Lead is extracted at *pH* between 8.5 and 10 by a solution of dithizone in chloroform. Excess of dithizone is eliminated by an ammoniacal solution of potassium cyanide. Lead dithizonate is determined by photometric measurement in chloroform solution at a wavelength of about 520 nm.

A-6.2.1 Range

This method is applicable for the determination of lead (as Pb) contents greater than 1 mg/kg.

B-6.2.2 Apparatus

B-6.2.2.1 Spectrophotometer

Any spectrophotometer suitable for measurement at a wavelength of about 520 nm or photoelectric absorptiometer fitted with filters giving maximum transmission between 500 nm and 540 nm.

B-6.2.3 Reagents

B-6.2.3.1 Chloroform — see IS 5296

B-6.2.3.2 Hydrochloric acid — approximately 12 N (see IS 265)

A.6.2.3.3 Ammonium citrate solution — 10 percent (m/v)

B-6.2.3.4 Purification of the solution

To 100 ml of solution, add ammonia solution, approximately 0.91 g/ml, until the pH reaches a value between 8.5 and 10, checking with the indicator paper. Transfer the solution to a separating funnel, add 10 ml of the dithizone solution and shake vigorously. Allow to separate, withdraw and reject the organic phase. Repeat the extraction, each time with 5 ml of the dithizone solution, until green colour remains. Allow to separate, withdraw and reject the organic phase.

B-6.2.3.5 Potassium cyanide solution — 50 g/l

B-6.2.3.6 Hydroxylammonium chloride solution — 100 g/l

B-6.2.3.7 Ammonia solution — approximately 5 N

NOTE — A freshly prepared solution shall be used in order to avoid too high a value being obtained by the blank test, due to dissolved lead. In fact, dilute ammonia dissolves lead contained in the glass more rapidly than does concentrated ammonia.

B-6.2.3.8 Dithizone solution — 0.025 g/l in chloroform

If dithizone of satisfactory quality is not available, it may be purified by the method as given in **B-6.2.3.8.1**.

B-6.2.3.8.1 Purification of the dithizone

Dissolve 1 g of dithizone (biphenyl thiocarbazon) in 75 ml of chloroform. Filter the solution, collecting the filtrate in a 250 ml separating funnel. Add 100 ml of approximately 0.2 N ammonia solution and shake vigorously. Withdraw the organic phase, collecting it in another separating funnel, and repeat, a further three times, the same operation, using 100 ml of approximately 0.2 N ammonia solution each time. (The dithizone thus passes into the alkaline aqueous phase, which assumes a more or less intense reddish-yellow coloration.) Discard the organic phase, combine the orange coloured aqueous extracts, filter them and transfer them to a 1 000 ml beaker. Precipitate the dithizone by slight acidification with a saturated solution of sulphur dioxide. Allow the precipitate to settle, filter through a sintered glass crucible and wash with water until there is no further acid reaction. Dry the precipitate in a desiccator containing concentrated sulphuric acid under- vacuum and in darkness, for 3 to 4 days. Grind the solid dry product quickly and transfer immediately to a small dark glass bottle. The dithizone, thus purified and stored away from direct sunlight, can be kept for at least 6 months.

B-6.2.3.8.2 Preparation of the solution

Immediately before use, weigh to the nearest 1 mg, 25 mg of the purified dithizone (see **B-6.2.3.8.1**), transfer to a 1 000 ml volumetric flask, dissolve in chloroform, dilute to the mark with chloroform and mix. Store the solution in a dry, dark glass, air-tight bottle.

B-6.2.3.9 Potassium cyanide solution, 1 g/l (ammoniacal).

Transfer 20 ml of the potassium cyanide solution to a 1 000 ml volumetric flask. Dilute with water, add 10 ml of ammonia solution, of relative density approximately 0.88 g/ml, dilute to the mark and mix.

CAUTION — Because potassium cyanide is extremely poisonous, it shall only be handled with all necessary precautions. In particular, do not add acids to solutions containing cyanides, otherwise hydrogen cyanide will be released.

B-6.2.3.10 Standard lead solution A

Weigh 1.6 g of lead nitrate previously dried at 105 °C and cooled in a desiccator and transfer to a beaker. Dissolve in a little water and 1 ml of nitric acid solution, approximately 1.40 g/ml. Transfer the solution quantitatively to a 1 000 ml volumetric flask, dilute to the mark and mix. One millilitre of this standard solution contains 1 mg of lead.

B-6.42.3.11 *Standard lead solution B*

Transfer 10 ml of the standard lead solution A to a 1 000 ml volumetric flask, add 1 ml of nitric acid solution, of relative density approximately 1.40 g/ml, dilute to the mark and mix. One millilitre of this standard solution contains 10 µg of lead. Prepare this solution immediately before use.

B-6.2.3.12 *pH indicator paper* — covering the range from 8.5 to 10**B-6.2.4** *Procedure***B-6.2.4.1** *Test portion*

Fill the pipette with the test sample and, weigh by difference to the nearest 0.02 g, take a test portion of about 50 g. Transfer the test portion to a 250 ml beaker.

B-6.2.4.2 *Blank test*

Carry out, in parallel, a blank test, using the same quantities of reagents as employed for the determination, and following the same procedure for the blank test, the determination and for the preparation of the calibration curve.

B-6.2.4.3 *Preparation of the calibration curve***B-6.2.4.3.1** *Preparation of the standard matching solutions for photometric measurements with a 1 cm cell*

Into eleven separating funnels of 100 ml capacity, fitted with ground glass stoppers, transfer 10 ml of water and add respectively the volumes measured with the burette, of the standard lead solution B indicated below:

<i>Sl. No.</i>	<i>Standard Lead Solution B ml</i>	<i>Corresponding Mass of Lead µg</i>
(1)	(2)	(3)
i)	0	0
ii)	1.0	10
iii)	2.0	20
iv)	3.0	30
v)	4.0	40
vi)	5.0	50
vii)	6.0	60
viii)	7.0	70
ix)	8.0	80
x)	9.0	90
xi)	10.0	100

B-6.2.4.3.2 Add to each of these solutions, 1 ml of the hydroxyl ammonium chloride solution and 10 ml of ammonium citrate solution and adjust the pH 8.5 to 10 by adding ammonia solution drop by drop checking with the indicator paper. Add 2 ml of potassium cyanide solution, and shake, followed by 5 ml of the dithizone solution and extract the lead dithizonate, shaking vigorously for 1 min. Allow to separate and draw off the organic phase, collecting it in a 50 ml volumetric flask. Continue the extraction with successive portions of 5 ml of dithizone solution, until the last portion of the dithizone solution, after swirling, remains green. Collect the various portions of the organic phase, as drawn off, in the same 50 ml volumetric flask, including the portion that remains green. Dilute the organic phase to the mark with chloroform and mix. In order to eliminate the excess of dithizone present in the organic phase, carry out the extraction with the minimum number of manipulations, using 5 ml portions of ammoniacal cyanide solution each time, until the yellow colour of dithizone has disappeared. Then draw off the organic phase which will have a clear pink colour, and pass it through a dry, 'acid-washed' filter paper, collecting the filtrate in a dry-vessel.

NOTE — Dithizonates are particularly sensitive to ultraviolet light and should, therefore, be protected from sunlight and fluorescent light.

B-6.2.4.4 *Photometric measurements*

Carry out the photometric measurements using the spectrophotometer at the maximum of the absorption curve (wavelength of about 520 nm) or with the photoelectric absorptiometer fitted with suitable filters; in each case adjust the instruments to zero absorbance against the compensation solution.

B-6.2.4.5 *Preparation of calibration chart*

Plot a graph having, for example, the lead (as Pb) contents, expressed in micrograms per 50 ml of standard matching solution, as abscissa and the corresponding values of absorbance as ordinate.

B-6.2.4.6 *Determination*

B-6.2.4.6.1 *Preparation of the test solution*

Place the beaker containing the test portion (**B-6.2.4.1**) on a sand-bath and evaporate cautiously to dryness in a well-ventilated fume cupboard. Cool, take up with 2 ml of hydrochloric acid solution and 25 ml of water, and warm moderately to complete the dissolution. Allow to cool, transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

B-6.2.4.6.2 *Extraction of the lead dithizonate*

According to the expected lead content, take an aliquot portion of the test solution (**B-6.2.4.6.1**) containing 10 µg to 100 µg of lead and transfer it to a separating funnel. Then proceed with the determination as given in **B-6.2.4.3.1**.

NOTE — If the lead content to be determined is of the order of 1 mg/kg, use the whole of the test solution, without dilution, for the extraction of the lead dithizonate.

B-6.2.4.6.3 *Photometric measurements*

Carry out the photometric measurements of the chloroform solution of lead dithizonate deriving from the test solution and that deriving from the blank test, according to the methods given in **B-6.2.4.3.2** but after having adjusted the instrument to zero absorbance against the chloroform.

B-6.2.5 *Calculation*

By means of the calibration curve (*see* **B-6.2.4.5**) determine the quantity of lead corresponding to the values of the photometric measurements:

$$\text{Lead (as Pb), ppm} = \frac{(M_1 - M_2) \times D}{M}$$

where

M = mass in g, of the test portion;

M_1 = mass in µg, of lead found in the aliquot portion of the test solution;

M_2 = mass in µg, of lead found in a corresponding aliquot portion of the blank test solution; and

D = ratio of the volume of test solution to the volume of aliquot portion taken for the extraction of the lead dithizonate.

B-6.3 *Method C*

B-6.3.1 *Apparatus*

B-6.3.1.1 *Nessler cylinders* — 50 ml capacity (*see* IS 4161)

B-6.3.2 *Reagents*

B-6.3.2.1 *p-Nitrophenol indicator solution*

Dissolve 0.2 g of *p*-Nitrophenol in hot water and dilute to 100 ml

B-6.3.2.2 *Dilute ammonium hydroxide* — 1 : 9 (v/v)

B-6.3.2.3 Dilute hydrochloric acid — 1 : 99 (v/v)

B-6.3.2.4 Hydrogen sulphide solution — saturated and freshly prepared.

B-6.3.2.5 Standard lead solution

Dissolve 1.60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

B-6.3.3 Procedure

Dissolve 2 g of the material in 25 ml of water in a Nessler cylinder and add 1 drop of *p*-nitrophenol indicator solution. Add dropwise dilute ammonium hydroxide until the solution turns yellow. Add dilute hydrochloric acid dropwise until the solution becomes colourless and then add 0.5 ml of the acid in excess. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test in a similar manner in another Nessler cylinder using 1 ml of standard lead solution.

B-6.3.3.1 The relevant 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.

B-7 DETERMINATION OF ARSENIC

B-7.1 General

Three methods are prescribed for determining arsenic, namely, Method A, ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-7.2 Method A

B-7.2.1 Procedure

To 22 ml acid add 1 ml of nitric acid and evaporate to 10 ml. Add to the cooled residue cautiously 25 ml of water and again evaporate until white fumes appear. Add to the cooled residue 50 ml of water, 0.2 ml of stannous chloride solution (1.5 M) and test as per IS 2088 by silver diethyl dithiocarbamate method.

B-8 TEST FOR OXIDIZABLE IMPURITIES

B-8.1 Reagents

B-8.1.1 Potassium Permanganate Solution — exactly 0.01 N

B-8.2 Procedure

Take a known volume of the material (say 10 to 20 ml) in a 250 ml conical flask and dilute the acid with 5 times its volume of water with continuous cooling in ice. Titrate the solution against standard potassium permanganate to a light, permanent pink colour end point.

B-8.3 Calculation

$$\text{Oxidizable impurities (as SO}_2\text{) percent by mass} = \frac{3.2 \times V \times N}{M}$$

where

V = volume in ml, of standard permanganate solution used for the titration;

N = normality of potassium permanganate solution; and

M = mass in g, (volume × relating density) of the sample taken for the test.

B-9 TEST FOR ORGANIC MATTER

B-9.1 Procedure

Heat the acid in a clean beaker until the acid begins to fume strongly.

B-9.1.1 The material shall be taken to have passed the test if it shows no perceptible charring.

B-10 TEST FOR NITRATES**B-10.1 General**

Two methods are prescribed for determining nitrates, namely, Method A and ion chromatography method as prescribed in **B-20**. In case of dispute, ion chromatography method shall be used as referee method.

B-10.2 Method A**B-10.2.1 Reagents**

B-10.2.1.1 Concentrated hydrochloric acid — see IS 265

B-10.2.1.2 Diphenylamine solution

Prepare by mixing 90 mg of diphenylamine with 60 ml of nitrogen-free sulphuric acid and adding the mixture to 20 ml of water.

B-10.2.2 Procedure

Dilute 6 ml of the material with 2 ml of water, cool to 60 °C and add one drop of concentrated hydrochloric acid and 1 ml of diphenylamine solution.

B-10.2.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if no blue colour is produced.

B-11 TEST FOR AMMONIA**B-11.1 Reagents**

B-11.1.1 Sodium Hydroxide Solution — approximately 30 percent

B-11.1.2 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of water and add to it slowly, with stirring, a saturated aqueous solution of mercuric chloride until a slight permanent precipitate forms. Add 30 g of potassium hydroxide and, when it has dissolved, add 1 ml more of mercuric chloride solution and dilute to 200 ml with water. Allow to settle overnight and then decant the clear solution. Keep the solution in a bottle closed with a well-fitting rubber stopper.

B-11.1.3 Standard Ammonium Chloride Solution

Dissolve 0.314 g of ammonium chloride in 1 litre of water. Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of ammonia (as NH₃).

B-11.2 Procedure

Add exactly 20 g of the material to 40 ml of water, cool in ice and make alkaline with sodium hydroxide solution. Add to the mixture 1 ml of Nessler solution. Carry out a control test using 4 ml of standard ammonium chloride solution in place of the material, finally diluting the contents to the same volume as in the test with the material.

B-11.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test solution is not greater than that produced in the control test.

B-12 TEST FOR SELENIUM**B-12.1 General**

Three methods are prescribed for determining selenium, namely, Method A, ICP-OES method as prescribed in IS 3025 (Part 2) and AAS method as prescribed in IS 3025 (Part 56). In case of dispute, AAS shall be used as referee method.

B-12.2 Method A**B-12.2.1 Reagents**

B-12.2.1.1 Concentrated hydrochloric acid — see IS 265

B-12.2.1.2 Sodium sulphite

B-12.2.1.3 Standard selenium solution

Dissolve 0.1 g of selenium in 5 ml of concentrated nitric acid and 10 ml of concentrated hydrochloric acid. Evaporate to dryness and then take up the residue with water and a little dilute selenium-free sulphuric acid (6 N). Make up the volume to 1 000 ml. One millilitre of this solution contains 0.1 mg of selenium (as Se).

B-12.2.2 Procedure**B-12.2.2.1 For Battery grade concentrated acid**

Cool exactly 5 g of the material contained in a test-tube by surrounding the tube with crushed ice. Carefully add to the acid 10 ml of concentrated hydrochloric acid containing about 10 mg of sodium sulphite. When effervescence ceases, no red colour shall appear at the zone of contact of the two liquids at the end of 3 min from the time the acid is completely covered by the first addition. The reddish brown ring caused by selenium, if present, forms slightly above, and separate from, a yellow ring which may result from the presence of copper and iron. Carry out a control test using 1 ml of standard selenium solution in the same total volume and compare the selenium rings, viewing them transversely against a white background.

B-12.2.2.1.1 The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour of the ring produced by the material is not greater than that produced in the control test.

B-12.2.2.2 For battery grade dilute acid

Carry out the test as described under **B-12.2.2.1** using exactly 5 g of the material for the test and 0.3 ml of standard selenium solution for the control test.

B-13 TEST FOR MANGANESE**B-13.1 General**

Three methods are prescribed for determining manganese, namely, Method A, ICP-OES method as prescribed at **B-19** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-13.2 Method A**B-13.2.1 Reagents**

B-13.2.1.1 Concentrated nitric acid — see IS 264

B-13.2.1.2 Potassium per iodate

B-13.2.1.3 Standard potassium permanganate solution — exactly 0.001 N

B-13.2.2 Procedure**B-13.2.2.1 For battery grade concentrated acid**

Dilute exactly 25 g of the material to 95 ml and add 5 ml of concentrated nitric acid. Add 0.5 g of potassium per iodate and boil the solution vigorously in a covered beaker for 5 min. Cool and compare the colour with that of an equal volume of a solution containing 2.3 ml of standard potassium permanganate solution, 5 ml of nitric acid and 0.5 g of potassium per iodate in water, this solution having been treated in the same manner.

B-13.2.2.1.1 The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced by the material is not greater than that produced in the control test.

B-13.2.2.2 For battery grade dilute acid

Carry out the test as described under **B-13.2.2.1** using exactly 25 g of the material for test and 0.7 ml of standard potassium permanganate solution for the control test.

B-14 TEST FOR COPPER**B-14.1 General**

Four methods are prescribed for determining copper, namely, Method A, Method B, ICP-OES method as prescribed in IS 3025 (Part 2) and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-14.2 Method A

The test shall be carried out only if the residue on ignition as obtained under **B-3.1** exceeds 0.003 percent in case of battery grade concentrated acid and 0.001 percent in case of battery grade dilute acid.

B-14.2.1 Apparatus

B-14.2.1.1 Nessler cylinders — 50 ml capacity

B-14.2.2 Reagents

B-14.2.2.1 Concentrated hydrochloric acid — see IS 265

B-14.2.2.2 Ammonium hydroxide — relative density 0.90

B-14.2.2.3 Standard copper solution

Dissolve 0.393 g of cupric sulphate ($\text{CuSO}_4 \cdot \text{H}_2\text{O}$) in 1 000 ml of water. One millilitre of the solution contains 0.1 mg of copper (as Cu).

B-14.2.3 Procedure

Add to the residue obtained in **B-3.1**, 1 ml of concentrated hydrochloric acid, warm on a steam-bath to dissolve copper and iron oxides, and dilute to 10 ml. Neutralize the solution with ammonium hydroxide and add 4 ml of ammonium hydroxide in excess. Heat sufficiently to coagulate any precipitate, filter into a Nessler cylinder, and dilute to 25 ml. The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour in the solution is not greater than that of a control standard prepared from the following amounts of standard copper solution treated with 4 ml of ammonium hydroxide and diluted to 25 ml in another Nessler cylinder:

- a) 15 ml in the case of battery grade concentrated acid; and
- b) 5 ml in the case of battery grade dilute acid.

B-14.3 Method B**B-14.3.1 Principle**

Copper present in the sample is reduced with the help of ascorbic acid and by addition of 2, 2'-biquinolyl a violet coloured complex is formed.

B-14.3.2 Reagents

Sodium sulphate, Hydrochloric acid solution, Amyl alcohol, (+) Tartaric acid, sodium hydroxide, L-ascorbic acid (100 g/l), 2, 2'-biquinolyl (0.5 g/l), Bromine water-saturated solution, Copper (0.1 g/l), standard solution, Indicator paper (Narrow range) pH 5.5 to pH 7.0, Methyl orange indicator (0.5 g/l) aqueous solution.

B-14.3.3 Apparatus

Spectrophotometer, photoelectric absorptiometer with maximum transmission at a wavelength of 545 nm, Optical cells, 4 cm optical path.

B-14.3.4 Procedure**B-14.3.4.1 Test portion**

Weigh about 10g of the sample to the nearest 0.1 g.

B-14.3.4.2 Blank Test

Carry a test with a blank as the determination follows the procedure.

B-14.3.4.3 Preparation of the calibration graph

Take six 500 ml separatory funnel and introduce the standard copper solutions.

Standard Copper Solutions

Sl No.	Standard copper Solution (ml)	Mass of Copper in μg
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(1)	(2)	(3)
i)	0	0
ii)	2	20
iii)	4	40
iv)	6	60
v)	8	80
vi)	10	100

To all the funnels add 400 ml water and 2 ml of tartaric acid. pH of the solution is adjusted around 6.0 using sodium hydroxide. Add about 2 ml of ascorbic acid and mix the contents thoroughly. Allow the mixture to stand for 5 min. Add 2, 2'-biquinolyl solution (10 ml) and mix well. Now try to extract the copper complex using two 20 ml of amyl alcohol portions. Transfer the extract to 100 ml beaker. Add sodium sulphate (2 g) to extract and remove water traces by stirring thoroughly.

In the next step, filter dry extract into 50 ml one-mark volumetric flask. Wash residual sodium sulphate using 2 ml two portions of amyl alcohol. Dilute the mark using amyl alcohol by transferring to volumetric flask and mix well.

Spectrophotometric Measurements

Measurements are made using spectrophotometer or photoelectric absorptiometer at a wavelength of 545 nm. Measurements are made using filters and adjusting the instruments properly to zero absorbance using amyl alcohol as blank solution.

B-14.3.4.4 Determination

Preparing test solution

To a 400 ml beaker, transfer the test portion. Add 1 drop of methyl orange and 100 ml water. Using Hydrochloric acid, neutralize the solution adding 5 ml in excess. Add bromine water (10 ml) in excess and boil the solution till it is free from bromine. Allow the solution to cool. To a 500 ml separatory funnel fitted with stopper, transfer the contents of the beaker and add 1 ml of HCl solution to it. After the sample preparation and colour development, photometric measurements are made on the test samples using blank test solution (by adjusting the instrument to zero absorbance).

B-14.3.4.5 Calibration graph

The graph for the above standard solutions is plotted between absorbance vs concentration. Take the absorbance of all the prepared solutions and then deduce the concentration of copper in the unknown solution using a calibration graph.

B-14.3.5 Expression of results

Determine the concentration of copper corresponding to the absorbance value. The copper content expressed as mg/kg is given by-

$$\frac{m_1 - m_2}{1000} \times \frac{1000}{m_0} = \frac{m_1 - m_2}{m_0}$$

where

m_0 = the mass of the test sample;

m_1 = the mass of copper in test solution, (in μg);

m_2 = copper in blank solution.

B-15 TEST FOR ZINC

B-15.1 General

The test shall be carried out only if the residue on ignition as obtained in **B-3.1** exceeds 0.003 percent in case of battery grade concentrated acid and 0.001 percent in case of battery grade dilute acid.

B-15.2 Apparatus**B-15.3 Reagents**

B-15.3.1 Sulphuric Acid — 5 percent (m/v) zinc-free

B-15.3.2 Hydrogen Sulphide — gas

B-15.3.3 Ammonium Hydroxide — relative density 0.90

B-15.3.4 Citric Acid Solution — 50 percent (m/v)

B-15.3.5 Ammonium Thiocyanate — 2 percent solution (m/v)

B-15.3.6 Hydrochloric Acid — relative density 1.20

B-15.3.7 Potassium Ferrocyanide Solution — 5 percent (m/v)

B-15.3.8 Standard Zinc Solution

Dissolve 0.1 g of pure zinc in 10 ml of hydrochloric acid and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of zinc.

B-15.3.9 Calcium Carbonate**B-15.4 Procedure**

In a silica dish, evaporate 25 ml (for concentrated grade) or 100 ml (for dilute grade) of battery grade sulphuric acid to dryness. Dissolve in 25 ml of 5 percent sulphuric acid and precipitate heavy metals with hydrogen sulphide.

Filter off the heavy metals and boil the filtrate to remove the hydrogen sulphide, cool, neutralize with ammonium hydroxide and add 10 ml of 50 percent citric acid solution. Heat the solution to boiling and if no calcium citrate separates, add small quantities of calcium carbonate at a time until a precipitate of about 1 g of calcium citrate is formed. Remove from heat and pass a stream of hydrogen sulphide through the solution until it has cooled. Filter the solution through a small filter paper and wash with ammonium thiocyanate solution. Dissolve the precipitate in 3 ml of hydrochloric acid diluted to 10 ml with water and wash the paper with water. Wash the solution into a 100 ml Nessler cylinder and hold until a series of standards covering the range in which the sample falls has been prepared by measuring portions of the standard zinc solution into 100 ml Nessler cylinders. Dilute the standard and sample solutions to about 90 ml and add 3 ml of hydrochloric acid to the standard solution and 2 ml of potassium ferrocyanide solution to all solutions. Dilute the contents of each Nessler cylinder to the mark and mix thoroughly. After standing for at least 5 min compare the turbidity of the standard and the sample. Calculate the percentage of zinc from the quantity of sample taken and the standard solution similar in turbidity to that of the sample.

B-16 TEST FOR NITRATES, NITRITES AND AMMONIA**B-16.1 Apparatus**

B-16.1.1 Nessler Cylinders — 50 ml capacity

B-16.2 Reagents

B-16.2.1 Sodium Hydroxide Solution — approximately 40 percent (m/v)

B-16.2.2 Devarda's Alloy

Containing 45 parts aluminium, 50 parts copper and 5 parts zinc. Heat the aluminium in a Hessian crucible in a furnace until the aluminium begins to melt. Add copper in small portions and heat until liquefied. Now plunge zinc into the molten mass. Cover the crucible and heat the mixture for a few moments and stir with an iron rod. Allow it to cool slowly with the cover on and then pulverize the crystallized mass.

B-16.2.3 Dilute Hydrochloric Acid — approximately 6 N

B-16.2.4 Nessler Solution — same as in **B-11.1.2**

B-16.2.5 Standard Ammonium Chloride Solution

Prepare a solution containing 0.381 9 g of ammonium chloride per litre. One millilitre of this solution contains 0.1 mg of nitrogen (as N).

B-16.3 Procedure

Weigh exactly 20 g of the material and add cautiously to 100 ml of water in a 500 ml flask. After cooling, carefully neutralize with sodium hydroxide solution till the liquid is distinctly alkaline. Add 2 g of Devarda's alloy and fit a distillation head and condenser. After 1 h, distil off 50 ml into a conical flask containing 1ml of dilute hydrochloric acid. Transfer the distillate to a Nessler cylinder, add 1 ml of sodium hydroxide solution and 2 ml of Nessler solution. Carry out a control test following the same procedure using the following quantities of standard ammonium chloride solution:

- a) 6 ml in the case of CP grade acid and battery grade concentrated acid; and
- b) 2 ml in the case of battery grade dilute acid.

B-16.3.1 The relevant 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 respective control test.

B-17 TEST FOR ANTIMONY

B-17.1 Reagents

B-17.1.1 *Hydrogen Sulphide Gas*

B-17.1.2 *Mercuric Chloride Test Paper*

B-17.1.3 *Dilute Ammonium Hydroxide — 1 : 4 (v/v)*

B-17.1.4 *Concentrated Sulphuric Acid*

B-17.1.5 *Standard Antimony Solution*

Dissolve 0.548 g of antimony potassium tartrate in water and make up to 1 000 ml. Dilute 100 ml of this solution to 1 000 ml. One millilitre of this solution contains 0.02 mg of antimony (as Sb).

B-17.1.6 *Sodium Sulphite*

B-17.1.7 *Concentrated Hydrochloric Acid — see IS 265*

B-17.2 Procedure

B-17.2.1 *For Battery Grade Concentrated Acid*

B-17.2.1.1 *Preliminary test*

Take 20 ml of the material, dilute with water to 150 ml and filter from any precipitated lead sulphate. Pass hydrogen sulphide through the solution for 5 min, let it stand for 10 min and filter through a 9 cm quantitative filter paper of close texture without washing. Unfold the paper and examine it closely, especially near the centre, in day-light or artificial light of equivalent colour. If the paper shows neither a black nor a brown colour nor a light yellow or orange colour, antimony is absent within the requirements of the specification, and test for it may be omitted. If precipitation of sulphur occurs during treatment with hydrogen sulphide, this preliminary test may be useless and the actual test as given in **B-17.2.1.2** shall be carried out.

B-17.2.1.2 *Actual test*

- a) If the arsenic content (as As) has been found to be not greater than 0.000 012 percent by mass, antimony be determined without first removing arsenic. In this case, evaporate exactly 20 g of the material to 4 or 5 ml, proceed as for the determination of arsenic by modified Gutzeit method as prescribed in IS 2088 except that the solution is finally warmed to about 60 °C and kept at that temperature for 1 h. Develop the colour by immersing the mercuric chloride test paper strip in dilute ammonium hydroxide. Compare the stain with that obtained under identical conditions from a solution containing 4 ml of concentrated sulphuric acid and 1 ml of standard antimony solution.
- b) If the arsenic content (as As) has been found to exceed 0.000 012 percent by mass, the arsenic present may interfere with the determination of antimony and shall first be removed. In this case, evaporate exactly 100 g of the material to 4 ml or 5 ml in a 250 ml beaker, cool and cautiously add 10 ml of water containing about 0.25 g of sodium sulphite. Evaporate until fumes begin to appear, cool and add 100 ml of concentrated

hydrochloric acid. Boil slowly in an open beaker until the volume is reduced to 40 or 45 ml, keeping the temperature of the liquid below 110 °C. Use boiling tubes or some other device to minimize bumping, and take care not to let the volume get too small or the temperature too high, otherwise some of the antimony may be lost. Transfer to a 50 ml volumetric flask and dilute to 50 ml. To a 10 ml aliquot, add 10 ml of water and 3 ml of concentrated sulphuric acid. Dilute to 50 ml and proceed as directed in **B-17.2.1.2** (a), comparing the stain with that obtained under identical conditions from a solution containing 1 ml of standard antimony solution, 3 ml of concentrated sulphuric acid and 4 ml of concentrated hydrochloric acid.

B-17.2.2 *For Battery Grade Dilute Acid*

B-17.2.2.1 *Preliminary test*

Take 100 ml of the material, dilute with water to 150 ml and proceed exactly in the same manner as described under **B-17.2.1.1**.

B-17.2.2.2 *Actual test*

- a) If the arsenic content (as As) has been found to be not greater than 0.000 004 percent by mass, antimony may be determined without first removing arsenic. In this case, take exactly 100 g of the material and proceed exactly in the manner described under **B-17.2.1.2** (a) but using 1.5 ml of standard antimony solution in the control test.
- b) If the arsenic content (as As) has been found to exceed 0.000 004 percent by mass, the arsenic present may interfere with the determination of antimony and shall be removed. In this case, take exactly 5 g of the material and proceed exactly in the manner described under **B-17.2.1.2** (b) but using 1.5 ml of standard antimony solution in the control test.

B-17.2.3 The relevant limit prescribed in **4.3.2.2** shall be taken as not having been exceeded if the length of the stain produced with the material as well as the intensity of its colour is not greater than that produced in the respective control test.

B-18 TEST FOR PLATINUM

B-18.1 *Reagents*

B-18.1.1 *Aqua Regia*

Prepared by mixing three volumes of concentrated hydrochloric acid with one volume of concentrated nitric acid.

B-18.2 *Procedure*

B-18.2.1 *For Battery Grade Concentrated Acid*

Evaporate 10 ml of the material in a small porcelain evaporating dish on a sand-bath. Evaporate completely but do not heat the residue unnecessarily. Cool and add 2 ml of aqua regia. Cover the dish with a watch-glass and digest on the steam-bath for 10 min. Remove the watch-glass and evaporate the solution to five or six drops. If the solution accidentally evaporates completely, repeat the treatment with aqua regia. Absorb the residual solution in a piece of thin asbestos paper not more than 0.8 mm thick, 5 mm wide and 30 mm long. Hold the paper in a crucible tongs and dip half of it into the solution. Dry gently over a flame and repeat until all of the solution is absorbed. Dry the paper and ignite to redness in a moderate Bunsen flame (8 cm to 10 cm high with good air mixture). Momentarily shut off the gas and then turn it on again so that the stream of unignited gas plays on the hot asbestos paper, held vertically. The limit prescribed in **4.3.2.2** (b) shall be taken as not having been exceeded if there is no glowing of the paper.

B-18.2.2 *For Battery Grade Dilute Acid*

Take 50 ml of the material and carry out the test in exactly the same manner as described under **B-18.2.1**.

B-19 DETERMINATION OF ARSENIC, IRON, LEAD AND MANGANESE BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

B-19.1 *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-19.2 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-19.2 and B-19.4)

Sl No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	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
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-
iii)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)		Cr, Fe
		217.00		(20)	
iv)	Mn	257.610	1	0.4	Cr, Fe, Mo, W
		293.305	(20)	8	Al, Cr, Fe, Ti

B-19.3 Reagents and Solutions

B-19.3.1. Nitric Acid (65 percent) Suprapure

B-19.3.2 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 $\mu\text{g}/\text{ml}$, 100 $\mu\text{g}/\text{ml}$ or 1 000 $\mu\text{g}/\text{ml}$ of lead, iron, manganese and arsenic in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-19.3.3 Standard Solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask and 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 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 $\mu\text{g}/\text{ml}$, 0.3 $\mu\text{g}/\text{ml}$ and 0.5 $\mu\text{g}/\text{ml}$ solution of respective elements under reference.

B-19.3.4 Sample Preparation

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1 ml nitric acid and make up the volume with water.

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

B-19.3.5 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-19.4 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-19.5 Procedure**B-19.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**B-19.3.5**). 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-19.5.2 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 $\mu\text{g/ml}$ of the lead (and/or Iron, manganese, arsenic) 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-19.6 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).

B-20 ION CHROMATOGRAPHY FOR CHLORIDES AND NITRATES**A 20.1 Principle**

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of Nitrates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions as Nitrates, 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-20.2 Equipment

B-20.2.1 Anion Guard Column

A protector of the separator column.

B-20.2.2 Anion Separator Column

Suitable for selective separation of ions under analysis.

B-20.2.3 Anion Suppressor Device

Anion micromembrane suppressor is used to analyse the data.

B-20.2.4 Detector — conductivity detector

B-20.2.5 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 1 000 µl be used to determine ionic concentration as per instrument manual and practice.

B-20.3 Reagents

B-20.3.1 Glass or Polyethylene Sample Bottles.

B-20.3.2 Distilled Water or Deionized Water Free from the Anions of Interest.

B-20.3.3 Eluent

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

B-20.3.4 Micro Membrane Suppressor Solution — (0.025 N of sulphuric acid)

Dilute 2.8 ml of concentrated sulphuric acid in 4 litres of water.

B-20.4 Standard Solutions

B-20.4.1 Chloride — Dissolve NaCl, 1.648 5 g in 1 litre of reagent water.

B-20.4.2 Nitrate Stock Solution

Dry approximately 2 g of sodium nitrate (NaNO₃) at 105 °C for 24 h. Dissolve exactly 1.370 7 g of the dried salt in reagent water, and dilute to 1 litre with reagent water in a volumetric flask.

B-20.5 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 to 1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

B-20.6 Procedure

Dissolve between 1 g to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1 ml to 1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An

automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For solid sample of cobalt acetate, following extraction procedure may be used.

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

B-20.7 Data analysis and Calculations

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

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

ANNEX C

(Clause 6)

SAMPLING OF SULPHURIC ACID

C-1 GENERAL REQUIREMENTS OF SAMPLING

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

C-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.3 To draw a representative sample, the contents of each container shall be mixed thoroughly by rolling, shaking or stirring by suitable means and with necessary caution.

C-1.4 The samples shall be placed in suitable, clean, dry and air-tight glass containers conforming to IS 1388 (Part 1).

C-1.5 Each sample container shall be sealed air-tight after filling and shall be marked with full details of sampling, the date of sampling and the year of manufacture of the material.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

C-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. The number of containers to be selected from the lots of different sizes shall be in accordance with Table 3.

C-2.3 The containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS 4905 may be followed.

C-3 PROCEDURE

C-3.1 Carboys, Drums, Jars and Bottles

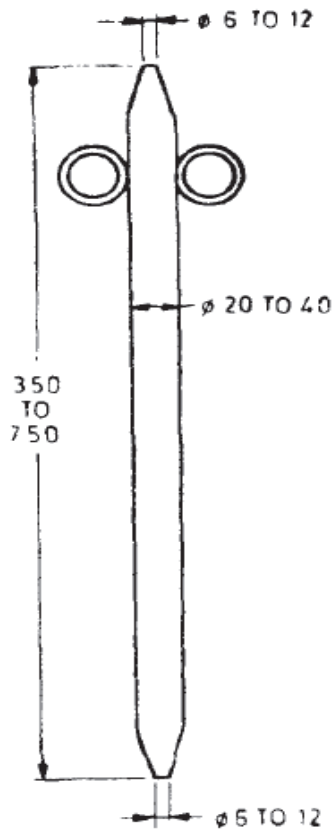
For drawing representative samples from these containers, agitate the material well by rolling or by stirring with a glass rod. Draw the required quantity of the material with the glass sampling instrument shown in Fig. 3 and

transfer it to a clean receptacle. This quantity shall be sufficient to make triplicate determinations for all the characteristics given in Table 1.

The representative samples so drawn shall constitute individual test samples.

Table 3 Number of Containers to be selected for Sampling
(Clause C-2.2)

Sl No.	No. of Containers to be Selected	Lot Size
(1)	(2)	(3)
i)	Up to 15	2
ii)	16 to 25	3
iii)	26 to 50	4
iv)	51 to 100	5
v)	101 to 300	6
vi)	301 to 500	7
vii)	501 to 800	8
viii)	801 to 1000	9
ix)	1001 and above	10



All dimensions in millimeters

FIG. 3 SAMPLING INSTRUMENT

C-3.1.1 A composite sample shall be prepared by mixing thoroughly a small but approximately equal quantity from each of these individual samples. The quantity of material for the composite sample shall be as given in **C-4.1** and **C-4.2**.

C-3.2 Tank Wagons

While loading tank cars, samples shall be drawn at the discharge pipe where the material enters the tank car. The composite sample of not less than 25 litres drawn from each wagon shall consist of small portions of not more than 500 ml each, taken at regular intervals during the period of filling. When samples are drawn at the purchaser's end, equal size samples of not less than 1 litre each shall be taken at 10 cm depth intervals and a composite sample shall be prepared by mixing these in a receptacle.

C-4 TEST SAMPLES AND REFEREE SAMPLE

C-4.1 In case of sulphuric acid of battery grade, three sets of test samples of 600 ml each shall be obtained from the composite sample of each selected container after thorough agitation of the acid. These shall be placed in clean, dry, glass-stoppered bottles, closed tightly, sealed and labelled with all the particulars of sampling.

C-4.2 In case of other grades of sulphuric acid, three sets of test samples of 250 ml each shall be obtained from the composite sample of each selected container after thorough agitation of the acid. These shall be placed in clean, dry, glass-stoppered bottles and closed tightly, sealed and labelled with all the particulars of sampling.

C-4.3 The supplier shall retain one set of sealed samples and deliver one set to the purchaser or his agent, if so required.

C-4.4 The third set of test samples, bearing the seals of the purchaser and the supplier, shall constitute the referee sample to be used in case of dispute between the purchaser and the supplier.

C-5 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

C-5.1 Total acidity shall be tested on each of the individual samples.

C-5.2 The remaining characteristics given in Table 1 shall be tested on the composite sample.

C-5.3 The lot shall be considered to have met the requirements of total acidity if each of the test results on the individual sample meets the corresponding requirements given in Table 1.

C-5.4 The lot shall be considered to have met the remaining requirements given in Table 1 if each of the test results on the composite samples satisfies the corresponding requirements given in Table 1.

C-5.5 The lot shall be declared as conforming to the requirements of this specification if **C-5.3** and **C-5.4** are satisfied.