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मानक

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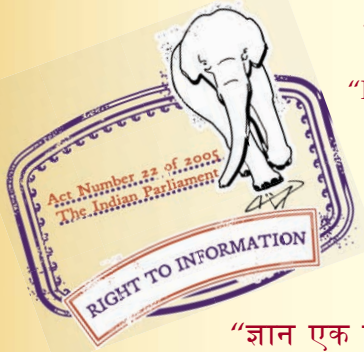
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“Step Out From the Old to the New”

IS 266 (1993): Sulphuric acid [CHD 1: Inorganic Chemicals]



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भारतीय मानक  
गंधक का अम्ल — विशिष्टि  
( तीसरा पुनरीक्षण )

*Indian Standard*  
SULPHURIC ACID — SPECIFICATION  
( *Third Revision* )

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**BUREAU OF INDIAN STANDARDS**  
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## FOREWORD

This Indian Standard ( Third Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1950 and then revised in 1961. The second revision was brought out in 1977 wherein the pure grade was substituted by chemically pure grade. Changes were also made in various requirements, namely, total acidity, iron, chlorides, lead, arsenic, oxidizable impurities and ammonia for both chemically pure as well as analytical reagent grades of sulphuric acid. In the second revision, the specific gravity requirement was deleted as it was felt that this requirement need not be a part of the specification. However, reference may be made to IS 4048 : 1989 'Density composition tables for aqueous solutions of sulphuric acid (*first revision*)' for correlation between density and composition of sulphuric acid.

In this revision, the requirements for iron content for the battery grade acid, oxidizable impurities for the analytical reagent grade, antimony and platinum have been suitably amended. The method for determination of oxidizable impurities has been modified. Suitable changes have been made in the packing clause. This revision also incorporates Amendments No. 1, 2 and 3, issued in 1981, 1984 and 1986 respectively.

For general information regarding precautions to be observed in safe handling and use of sulphuric acid, reference may be made to IS 4262 : 1967 'Code of safety for sulphuric acid'.

In the preparation of this standard, reference has been made to the following standards published by the International Organization for Standardization ( ISO ) :

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/913-1977 Sulphuric acid and oleum for industrial use — Determination of ash — Gravimetric method.

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

ISO/2717-1973 Sulphuric acid and oleum for industrial use — Determination of lead content — Dithizone photometric 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 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## SULPHURIC ACID — SPECIFICATION

### ( Third Revision )

#### 1 SCOPE

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

#### 2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	Title
264 : 1976	Nitric acid ( <i>second revision</i> )
265 : 1993	Hydrochloric acid ( <i>fourth revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods ( <i>first revision</i> )
1388 : 1959	Reagents bottles
2088 : 1983	Methods for determination of arsenic ( <i>second revision</i> )
4905 : 1968	Methods for random sampling
5296 : 1979	Chloroform, pure and technical ( <i>first revision</i> )

#### 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 A. Reference to the relevant clauses of Annex A 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 A-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 A-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 and 4.3 )

SI No.	Characteristic	Requirement					Method of Test, ( Ref to Cl No. in Annex A )
		Technical Grade	Battery Grade		Chemically Pure Grade	Analytical Reagent Grade	
(1)	(2)	(3)	Concentrated	Dilute	(6)	(7)	(8)
i)	Total acidity (as H <sub>2</sub> SO <sub>4</sub> ), percent by mass, <i>Min</i>	98.0	95.0	30.0	98.0	98.0	A-2
ii)	Residue on ignition, percent by mass, <i>Max</i>	0.2	0.06	0.02	0.01	0.002	A-3
iii)	Iron (as Fe), percent by mass, <i>Max</i>	0.05	0.003	0.001	0.001	0.000 05 (0.5 ppm)	A-4
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	—	0.001	0.000 3 (3 ppm)	0.003 5	0.000 02 (0.2 ppm)	A-5
v)	Lead (as Pb), percent by mass, <i>Max</i>	0.005	—	—	0.002	0.000 1 (1 ppm)	A-6
vi)	Arsenic (as As), percent by mass, <i>Max</i>	0.004	0.000 12 (1.2 ppm)	0.000 04 (0.4 ppm)	0.000 2 (2 ppm)	0.000 005 (0.05 ppm)	A-7
vii)	Oxidizable impurities (as SO <sub>2</sub> ), percent by mass, <i>Max</i>	—	0.02	0.02	0.004	0.000 4 (4 ppm)	A-8
viii)	Organic matter	—	To pass test	—	—	—	A-9
ix)	Nitrates (as NO <sub>3</sub> ), percent by mass, <i>Max</i>	—	—	—	—	0.000 02 (0.2 ppm)	A-10
x)	Ammonia (as NH <sub>3</sub> ), percent by mass, <i>Max</i>	—	—	—	—	0.000 2 (2 ppm)	A-11
xi)	Selenium (as Se), percent by mass, <i>Max</i>	—	0.002	0.000 6 (6 ppm)	—	—	A-12
xii)	Manganese (as Mn), percent by mass, <i>Max</i>	—	0.000 1 (1 ppm)	0.000 03 (0.3 ppm)	—	—	A-13
xiii)	Copper (as Cu), percent by mass, <i>Max</i>	—	0.003	0.001	—	—	A-14
xiv)	Zinc (as Zn), percent by mass, <i>Max</i>	—	0.003	0.001	—	—	A-15
xv)	Nitrates, nitrites and ammonia (as N), percent by mass, <i>Max</i>	—	0.003	0.001	0.003	—	A-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 stone-ware 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** The bottles or jars shall be packed in suitable pent top packing cases. 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. Carboys shall be packed in suitable iron hampers or wooden crates, with the inter-space being sufficiently stuffed with whiting, keiselguhr or other noncombustible material.

**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 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 in red letters not less than 5 cm high, showing the name of the acid; indication of the source of manufacture; the grade and mass of the material; and the recognized trade-mark, if any. They shall prominently display the words 'CORROSIVE, 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) : 1973.

## 6 SAMPLING

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

## ANNEX A

( *Clauses 4.2 and 4.3.2.2* )

### METHODS OF TEST FOR SULPHURIC ACID

#### A-1 QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS 1070 : 1992 ) shall be used in tests.

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

#### A-2 DETERMINATION OF TOTAL ACIDITY

##### A-2.0 General

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

##### A-2.1 Apparatus

###### A-2.1.1 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.

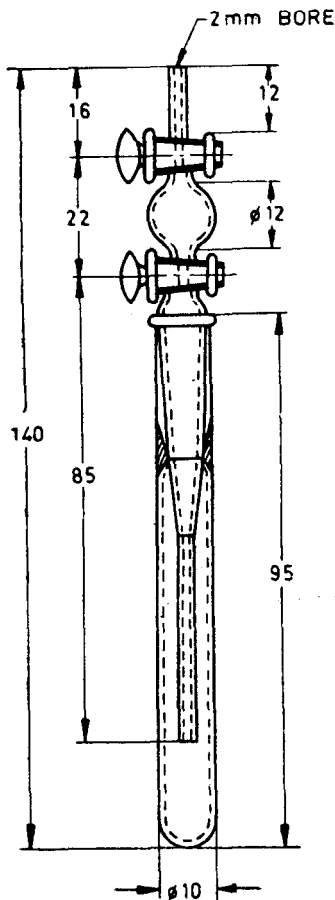
##### A-2.2 Reagents

**A-2.2.1** *Standard Sodium Hydroxide Solution*, 1 N, freshly standardized.

###### A-2.2.2 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-REY PIPETTE

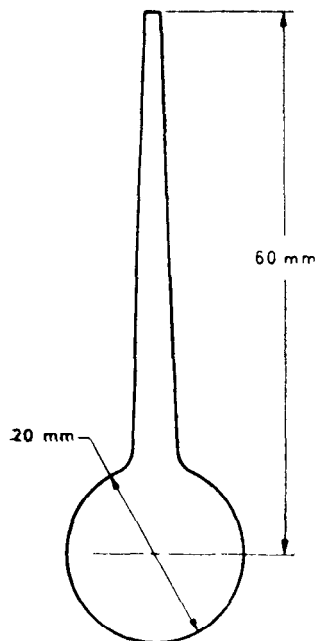


FIG. 2 SPHERICAL GLASS AMPOULES

## A-2.3 Procedure

### A-2.3.1 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 in a flame the bulb of glass ampoule 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 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.

### A-2.3.2 Sulphuric Acid Content Equal to or Lower than 98 Percent by Mass

Weigh accurately as in A-2.3.1, 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.

## A-2.4 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.

### A-3 DETERMINATION OF RESIDUE ON IGNITION

#### A-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  $800 \pm 50^\circ\text{C}$ , 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  $800 \pm 50^\circ\text{C}$  and keep at this temperature for about 15 minutes. Remove the dish from the furnace, cool in a desiccator and weigh. Repeat heating, cooling and weighing till constant mass is obtained.

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

#### A-3.2 Calculation

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

where

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

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

### A-4 DETERMINATION OF IRON

**A-4.0** Two methods are prescribed, namely, Method A (Bipyridyl method) and Method B (Thiocyanate method). In case of dispute, Method A shall be the referee method.

#### A-4.1 Method A (Bipyridyl Method)

##### A-4.1.0 General

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

##### A-4.1.1 Apparatus

###### A-4.1.1.1 Photometer

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

##### A-4.1.2 Reagents

**A-4.1.2.1 Dilute Hydrochloric acid**, approximately 1 N.

##### A-4.1.2.2 Hydroxylammonium chloride solution

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

**A-4.1.2.3 Ammonium acetate solution**, 30 percent (m/v).

##### A-4.1.2.4 2'2'-Bipyridyl solution

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

##### A-4.1.2.5 Standard iron solution A

Dissolve 0.7022 g of ferrous ammonium sulphate [ $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ] 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).

##### A-4.1.2.6 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.

#### A-4.1.3 Procedure

##### A-4.1.3.1 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:

Standard Iron Solution B	Corresponding to Fe
ml	$\mu\text{g}$
0 (compensation solution)	0
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
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 hydroxylammonium chloride solution and after 5 minutes, 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 minutes. Carry out the measurement on the spectrophotometer with a 1-cm cell at a wavelength of about 522 nm (or on a photoelectric

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.

#### A-4.1.3.2 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 and 500  $\mu\text{g}$  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 hydroxylammonium chloride solution and, after 5 minutes 5 ml of ammonium acetate solution and 1 ml of 2,2'-bipyridyl solution. Dilute to the mark, mix and wait for 10 minutes. Carry out the spectrophotometric (or photoelectric colorimetric) measurement according to the procedure given in A-4.1.3.1, adjusting the instrument to zero optical density using as reference the blank test solution (see A-4.1.3.3).

#### A-4.1.3.3 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.

#### A-4.1.4 Calculation

By reference to the calibration chart (see A-4.1.3.1), 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.

### A-4.2 Method B ( Thiocyanate Method )

#### A-4.2.0 General

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

#### A-4.2.1 Apparatus

A-4.2.1.1 Nessler Cylinders, 50-ml capacity.

#### A-4.2.2 Reagents

A-4.2.2.1 Hydrochloric acid, 1 : 1 (v/v).

A-4.2.2.2 Potassium permanganate solution, 0.1 N approximately.

A-4.2.2.3 Ammonium thiocyanate solution, 60 percent (m/v).

A-4.2.2.4 Mixture of amyl alcohol and amyl acetate, 1 : 1 (v/v).

A-4.2.2.5 Standard iron solution B, Same as in A-4.1.2.6.

#### A-4.2.3 Procedure

##### A-4.2.3.1 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:

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

##### 4.2.3.2 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 A-4.2.3.1, but using 5 ml of standard iron solution B in the control test.

##### A-4.2.3.3 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 A-4.2.3.1, but using 2.5 ml of standard iron solution B in the control test.

A-4.2.3.4 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.

## A-5 TEST FOR CHLORIDES

### A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders*, 50-ml capacity.

### A-5.2 Reagents

A-5.2.1 *Dilute Nitric Acid*, approximately 4 N.

A-5.2.2 *Silver Nitrate Solution*, approximately 5 percent (m/v).

### A-5.2.3 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).

### A-5.3 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:

- 2 ml in the case of battery grade concentrated acid,
- 7.5 ml in the case of battery grade dilute acid,
- 7 ml in the case of CP grade acid, and
- 0.5 ml in the case of AR grade acid.

A-5.3.1 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.

## A-6 DETERMINATION OF LEAD

### A-6.0 General

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-6.1 Range

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

### A-6.2 Apparatus

#### A-6.2.1 Spectrophotometer

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

### A-6.3 Reagents

A-6.3.1 *Chloroform*, see IS 5296 : 1979.

A-6.3.2 *Hydrochloric Acid*, approximately 12 N (see IS 265 : 1993).

A-6.3.3 *Ammonium Citrate Solution*, 10 percent (m,v).

#### A-6.3.3.1 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.

A-6.3.4 *Potassium Cyanide Solution*, 50 g/l.

A-6.3.5 *Hydroxylammonium Chloride Solution*, 100 g/l.

A-6.3.6 *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.

A-6.3.7 *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 A-6.3.7.1.

#### A-6.3.7.1 Purification of the dithizone

Dissolve 1 g of dithizone (biphenylthiocarbazone) 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 dith zone, thus purified and stored away from direct sunlight, can be kept for at least 6 months.

#### A-6.3.7.2 Preparation of the solution

Immediately before use, weigh to the nearest 1 mg, 25 mg of the purified dithizone (see A-6.3.7.1), 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.

#### A-6.3.8 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.

#### A-6.3.9 Standard Lead Solution A

Weigh 1.600 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.

#### A-6.3.10 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.

**A-6.3.11 pH Indicator Paper**, covering the range from 8.5 to 10.

### A-6.4 Procedure

#### A-6.4.1 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.

#### A-6.4.2 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.

#### A-6.4.3 Preparation of the Calibration Curve

##### A-6.4.3.1 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:

Standard Lead Solution B ml	Corresponding Mass of Lead µg
0 (compensation solution)	0
1.0	10
2.0	20
3.0	30
4.0	40
5.0	50
6.0	60
7.0	70
8.0	80
9.0	90
10.0	100

**A-6.4.3.2** Add to each of these solutions, 1 ml of the hydroxylammonium chloride solution and 10 ml of ammonium citrate solution and adjust the pH between 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 minute. 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.

#### A-6.4.3.3 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.

#### A-6.4.3.4 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.

#### A-6.4.4 Determination

##### A-6.4.4.1 Preparation of the test solution

Place the beaker containing the test portion (A-6.4.1j) 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.

##### A-6.4.4.2 Extraction of the lead dithizonate

According to the expected lead content, take an aliquot portion of the test solution (A-6.4.4.1) containing 10 to 100 µg of lead and transfer it to a separating funnel. Then proceed with the determination as given in A-6.4.3.1.

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.

##### A-6.4.4.3 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 A-6.4.3.2, but after having adjusted the instrument to zero absorbance against the chloroform.

##### A-6.4.5 Calculation

By means of the calibration curve (see A-6.4.3.4), 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.

## A-7 DETERMINATION OF ARSENIC

### A-7.1 Procedure

Take a suitable quantity of the material so that the test solution contains 1 to 10 µg of arsenic in a final volume of  $5.0 \pm 0.5$  ml. Determine arsenic by silver diethyl dithiocarbamate method as prescribed in IS 2088 : 1983.

## A-8 TEST FOR OXIDIZABLE IMPURITIES

### A-8.1 Reagents

**A-8.1.1 Potassium Permanganate Solution**, exactly 0.01 N.

### A-8.2 Procedure

Take a known volume of the material (say 10 to 20 ml) in a 250-ml conical flask and dilute with an equal volume of water. Titrate the solution against standard potassium permanganate to a light, permanent pink colour end point.

### A-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  $\times$  relating density) of the sample taken for the test.

## A-9 TEST FOR ORGANIC MATTER

### A-9.1 Procedure

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

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

## A-10 TEST FOR NITRATES

### A-10.1 Reagents

**A-10.1.1 Concentrated Hydrochloric Acid** — (see IS 265 : 1993).

**A-10.1.2 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.

**A-10.2 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.

**A-10.2.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if no blue colour is produced.

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

**A-11.1.1 Sodium Hydroxide Solution**, approximately 30 percent.

**A-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.

**A-11.1.3 Standard Ammonium Chloride Solution**

Dissolve 0.314 g of ammonium chloride in one 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  $\text{NH}_3$ ).

**A-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.

**A-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.

**A-12 TEST FOR SELENIUM****A-12.1 Reagents**

**A-12.1.1 Concentrated Hydrochloric Acid** — (see IS 265 : 1993).

**A-12.1.2 Sodium Sulphite****A-12.1.3 Standard Selenium Solution**

Dissolve 0.100 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).

**A-12.2 Procedure****A-12.2.1 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 minutes 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 one millilitre of standard selenium solution in the same total volume and compare the selenium rings, viewing them transversely against a white background.

**A-12.2.1.1** 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.

**A-12.2.2 For Battery Grade Dilute Acid**

Carry out the test as described under **A-12.2.1** using exactly 5 g of the material for the test and 0.3 ml of standard selenium solution for the control test.

**A-13 TEST FOR MANGANESE****A-13.1 Reagents**

**A-13.1.1 Concentrated Nitric Acid** — (see IS 264 : 1976).

**A-13.1.2 Potassium Periodate**

**A-13.1.3 Standard Potassium Permanganate Solution**, exactly 0.001 N.

**A-13.2 Procedure****A-13.2.1 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 periodate and boil the solution vigorously in a covered beaker for 5 minutes. 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 periodate in water, this solution having been treated in the same manner.

**A-13.2.1.1** 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.

**A-13.2.2** *For Battery Grade Dilute Acid*

Carry out the test as described under A-13.2.1 using exactly 25 g of the material for test and 0.7 ml of standard potassium permanganate solution for the control test.

**A-14 TEST FOR COPPER**

**A-14.0 General**

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

**A-14.1 Apparatus**

**A-14.1.1** *Nessler Cylinders* — 50-ml capacity.

**A-14.2 Reagents**

**A-14.2.1** *Concentrated Hydrochloric Acid* — ( see IS 265 : 1993 ).

**A-14.2.2** *Ammonium Hydroxide*, relative density 0.90.

**A-14.2.3** *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 ).

**A-14.3 Procedure**

Add to the residue obtained in A-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.

**A-15 TEST FOR ZINC**

**A-15.0 General**

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

**A-15.1 Apparatus**

**A-15.1.1** *Nessler Cylinders*, 100-ml capacity.

**A-15.2 Reagents**

**A-15.2.1** *Sulphuric Acid*, 5 percent (*m/v*), zinc-free.

**A-15.2.2** *Hydrogen Sulphide Gas*

**A-15.2.3** *Ammonium Hydroxide*, relative density 0.90.

**A-15.2.4** *Citric Acid Solution*, 50 percent (*m/v*).

**A-15.2.5** *Ammonium Thiocyanate*, 2 percent solution (*m/v*).

**A-15.2.6** *Hydrochloric Acid*, relative density 1.20.

**A-15.2.7** *Potassium Ferrocyanide Solution*, 5 percent (*m/v*).

**A-15.2.8** *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.

**A-15.2.9** *Calcium Carbonate*

**A-15.3 Procedure**

**A-15.3.1** 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 1.0 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 minutes 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.

#### A-16 TEST FOR NITRATES, NITRITES AND AMMONIA

##### A-16.1 Apparatus

A-16.1.1 *Nessler Cylinders*, 50-ml capacity.

##### A-16.2 Reagents

A-16.2.1 *Sodium Hydroxide Solution*, approximately 40 percent (*m/v*).

A-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.

A-16.2.3 *Dilute Hydrochloric Acid*, approximately 6 N.

A-16.2.4 *Nessler Solution*, same as in A-11.1.2.

A-16.2.5 *Standard Ammonium Chloride Solution*

Prepare a solution containing 0.3819 g of ammonium chloride per litre. One millilitre of this solution contains 0.1 mg of nitrogen (as N).

##### A-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 one hour, distil off 50 ml into a conical flask containing 1 ml 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.

A-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.

#### A-17 TEST FOR ANTIMONY

##### A-17.1 Reagents

A-17.1.1 *Hydrogen Sulphide Gas*

A-17.1.2 *Mercuric Chloride Test Paper*

A-17.1.3 *Dilute Ammonium Hydroxide*, 1 : 4 (*v/v*).

A-17.1.4 *Concentrated Sulphuric Acid*

A-17.1.5 *Standard Antimony Solution*

Dissolve 0.548 g of antimony potassium tartarate 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).

A-17.1.6 *Sodium Sulphite*

A-17.1.7 *Concentrated Hydrochloric Acid*, See IS 265 : 1993.

##### A-17.2 Procedure

A-17.2.1 *For Battery Grade Concentrated Acid*

A-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 minutes, let it stand for 10 minutes 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 A-17.2.1.2 shall be carried out.

A-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 : 1983 except that the solution is finally warmed to about 60°C and kept at that temperature for one hour. Developed 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 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 A-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.

#### A-17.2.2 For Battery Grade Dilute Acid

##### A-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 A-17.2.1.1.

##### A-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 A-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 A-17.2.1.2 (b) but using 1.5 ml of standard antimony solution in the control test.

A-17.2.3 The relevant limit prescribed in 4.3.2.2 (a) 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.

## A-18 TEST FOR PLATINUM

### A-18.1 Reagents

#### A-18.1.1 Aqua Regia

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

### A-18.2 Procedure

#### A-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 minutes. 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 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.

#### A-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 A-18.2.1.

## ANNEX B

( Clause 6.1 )

## SAMPLING OF SULPHURIC ACID

**B-1 GENERAL REQUIREMENTS OF SAMPLING**

**B-1.0** In drawing samples, the following precautions and directions shall be observed.

**B-1.1** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.2** 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.

**B-1.3** The samples shall be placed in suitable, clean, dry and air-tight glass containers conforming to IS 1388 : 1959.

**B-1.4** 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.

**B-2 SCALE OF SAMPLING****B-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.

**B-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 2.

**B.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 : 1968 may be followed.

**B-3 PROCEDURE****B-3.1 Carboys, Drums, Jars and Bottles**

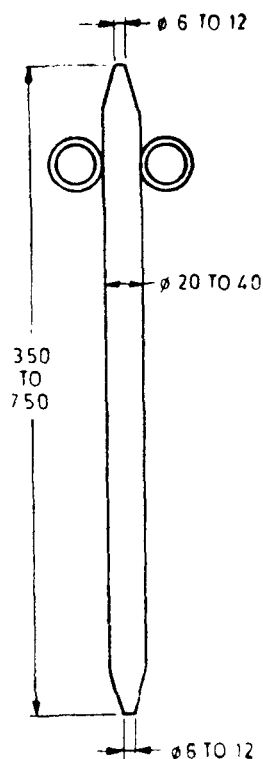
For drawing representative samples from these containers, agitate the material well by rolling or by stirring with a glass rod. Draw the required quantity of the material with the glass sampling instrument shown in Fig. 3 and transfer it to a clean receptacle. This quantity shall be sufficient to make triplicate determinations for all the characteristics given in Table 1.

The representative samples so drawn shall constitute individual test samples.

**Table 2 Number of Containers to be Selected for Sampling**

( Clause B-2.2 )

No. of Containers to be Selected	Lot Size
<i>n</i>	<i>N</i>
(1)	(2)
Up to 15	2
16 ,, 25	3
26 ,, 50	4
51 ,, 100	5
101 ,, 300	6
301 ,, 500	7
501 ,, 800	8
801 ,, 1 000	9
1 001 and above	10



All dimensions in millimetres.

FIG. 3 SAMPLING INSTRUMENT

**B-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 **B-4.1** and **B-4.2**.

#### **B-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 one litre each shall be taken at 10 cm depth intervals and a composite sample shall be prepared by mixing these in a receptacle.

#### **B-4 TEST SAMPLES AND REFEREE SAMPLE**

**B-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.

**B-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.

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

**B-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.

#### **B-5 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY**

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

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

**B-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.

**B-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.

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

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### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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**AMENDMENT NO. 1 FEBRUARY 2003**  
**TO**  
**IS 266 : 1993 SULPHURIC ACID — SPECIFICATION**  
*(Third Revision)*

( Page 1, clause 2.1 ) — Insert the following at the appropriate place:

'IS 7017 : 1973 Test method for colorimetric determination of traces of heavy metals by dithizone'.

[ Page 2, Sl No. Table 1, (ii), (iii) and (v) ] — Substitute the following for the existing:

Sl No.	Characteristic	Requirement					Method of Test (Ref to Clause No. in Annex A)
		Technical Grade	Battery Grade		Chemically Pure Grade	Analytical Reagent Grade	
			Concentrated	Dilute			
ii)	Residue on ignition, percent by mass, <i>Max</i>	0.05	0.06	0.02	0.02	0.002	A-3
iii)	Iron (as Fe), percent by mass, <i>Max</i>	0.01	0.003	0.001	0.001	0.000 05	A-4
v)	Heavy metals (as lead), percent by mass, <i>Max</i>	0.005	—	—	0.002	0.00 1	A-6/ IS 7017

( Page 3, clause 5.1.5, second sentence ) — Substitute the following for the existing:

'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 ).'

( Page 3, clause 5.1.6 ) — Substitute the following for the existing:

'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.'

## Amend No. 1 to IS 266 : 1993

( Page 7, clauses A-6, A-6.0 and A-6.1 ) — Substitute the following for the existing:

### A-6 DETERMINATION OF HEAVY METAL (AS LEAD)

#### A-6.0 GENERAL

Two methods have been specified for determination of lead. Method A (see IS 7017 : 1973) and Method B. However in case of dispute method A shall be the referee method.

#### A-6.1 Method B

Evaporate 11 cc acid to dryness. Add 2 ml dilute hydrochloric acid and 18 ml water. Warm to facilitate dissolution. To this add 30 ml water and 10 ml dilute ammonia solution ( 5N ). Pass hydrogen sulphide gas to the solution for a few seconds. The colour of the solution thus obtained is compared with a solution containing 2 ml standard lead solution (1 ml = 0.01 mg Pb) and 3 ml dilute acetic acid (5N). Add 10 ml of dilute ammonia solution and made up to 50 ml volume with water through which hydrogen sulphide gas is passed for few seconds. The material shall deemed to have passed the test if the colour of the sample solution is lighter than standard solution.

( Page 9, clause A-7.1 ) — Substitute the following for the existing:

#### 'A-7.1 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.'

( Page 9, clause A-8.2 ) — Substitute the following for the existing:

#### 'A-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.'

( CHD 1 )