भारतीय मानक

नाइट्रिक अम्ल — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

NITRIC ACID — SPECIFICATION

(Third Revision)

ICS 71.060.30

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

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Price Group 10

FOREWORD

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

This standard was first published in 1950 and first revised in 1968. It was again revised in 1976 to incorporate a new grade for explosives and pure grade was modified to chemically pure grade. New requirements for iron, manganese, phosphate and silicate for analytical reagent and ammonium salts for technical grade were included. The requirement for specific gravity was deleted but a correlation table for relative density and percent by mass of nitric acid was also incorporated (*see* Annex P).

In this revision, with a view to harmonize Indian Standard with ISO Standard, potentiometric method for chlorides, reduction and titrimetry method for sulphates and spectrophotometric method for ammonium salts have been incorporated.

For general information regarding precautions to be observed in the safe handling and use of nitric acid reference may be made to IS 4560 : 1968 'Code of safety for nitric acid'.

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

ISO 1980 : 1977	Nitric acid for industrial use — Determination of total acidity — Titrimetric method
ISO 1981 : 1971	Nitric acid for industrial use — Determination of nitrous compounds — Titrimetric method
ISO/R 1982 : 1971	Nitric acid for industrial use — Determination of iron content — 2, 2′-Bipyridyl photometric method
ISO 1983 : 1971	Nitric acid for industrial use — Determination of sulphated ash — Gravimetric method
ISO 2991 : 1974	Nitric acid for industrial use — Determination of ammoniacal nitrogen content — Spectrophotometric method
ISO 3328 : 1975	Nitric acid for industrial use — Determination of sulphate content — Method by reduction and titrimetry
ISO 3693 : 1977	Nitric acid for industrial use — Determination of chloride ions content — Potentiometric method — Determination of chloride ions
BS 975 : 1987 (1993)	Schedule for density — Composition tables for aqueous solutions of nitric acid.

The composition of the Committee responsible for formulation of this standard is given in Annex Q.

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

NITRIC ACID — SPECIFICATION

(Third Revision)

1 SCOPE

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

2 REFERENCES

The following standards 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 standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
336 : 1973	Specification for ether (*second revision*)
1070 : 1992	Reagent grade water (*third revision*)
1260 : 1973	Pictorial marking for handling and labelling of goods : (Part 1) Dangerous goods (*first revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
4905 : 1968	Methods of random sampling
7017 : 1973	Method of colorimetric determination of traces of heavy metals by dithizone

3 GRADES

Nitric acid shall be of the following five grades:

a) Technical (Tech),
b) Nitration,
c) Explosive,
d) Chemically pure (CP), and
e) Analytical reagent (AR).

4 REQUIREMENTS

4.1 Description

4.1.1 *Technical, Nitration and Explosive Grades*

The material shall be not darker than pale brown in colour and shall be free from sediment and other visible impurities.

4.1.2 *Chemically Pure and Analytical Reagent Grades*

The material shall be clear and almost colourless, free from sediment 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 to N. Reference to the relevant Annexes is given in Table 1.

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 relevant provisions of Red Tariff No. 18 of 1960 issued by the Indian Railway Conference Association, with any alterations and additions made thereafter.

5.1.2 When nitric acid is supplied in screw-stoppered stone bottles or glass carboys, the containers shall be fitted with leak-tight stoppers and, if required by the purchaser, provided with asbestos washers. The stoppers shall be further sealed by putty made from china clay or a mixture of sodium silicate and asbestos flour or a mixture of sulphur and sand.

5.1.3 Nitric acid of chemically pure and analysis reagent grades shall be supplied in glass bottles or glass carboys fitted with tight fitting TEFLON WASHERS (of thickness 0.2 mm) and stoppered with HDPE or other suitable stopper. The use of HDPE or other suitable caps over the stopper is recommended.

5.1.4 The bottles or jars shall be packed in suitable pent top packing cases. The bottles may also be placed in expanded polystyrene or expanded polyethylene containers and finally in corrugated fibreboard boxes. They shall be placed in an upright position on one layer of sand or ashes free from cinders 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, the interspace being sufficiently stuffed with whiting kieselghur or other non-combustible absorbent material.

5.2 Marking

5.2.1 The containers and also the packages, where possible, shall be suitably marked in red letters, not

Table 1 Requirements for Nitric Acid

(*Clause* 4.2)

Sl No.	Characteristic	Requirements				Method of Test, Ref to Annex
		Technical Grade	Nitration and Explosive	Chemically Pure Grade	Analytical Reagent Grade	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Total acidity (as HNO_3), percent by mass, *Min*	52.0	Nitration Grade : 93.0 Explosive Grade : 98.0	65.0[1)]	69.5	A
ii)	Residue on ignition, percent by mass, *Max*	0.1	0.05	0.01	0.001	B
iii)	Chlorides (as Cl), percent by mass, *Max*	0.03	0.001	0.001	0.000 05 (0.5 ppm)	C
iv)	Sulphates (as H_2SO_4), percent by mass, *Max*	0.2	0.03	0.005	0.000 2 (2 ppm)	D
v)	Heavy metals (as Pb), percent by mass, *Max*	To Pass Test	To Pass Test	To Pass Test	0.000 02 (0.2 ppm)	E
vi)	Nitrous acid (as HNO_2), percent by mass, *Max*	—	0.2	0.05	—	F
vii)	Arsenic (as As), percent by mass, *Max*	—	—	0.000 2 (2 ppm)	0.000 001 (0.01 ppm)	G
viii)	Iodine	—	To Pass Test	—	—	H
ix)	Iron (as Fe), percent by mass, *Max*	—	—	—	0.000 02 (0.2 ppm)	J
x)	Manganese (as Mn), percent by mass, *Max*	—	—	—	0.000 04 (0.4 ppm)	K
xi)	Phosphate (as PO_4), percent by mass, *Max*	—	—	—	0.000 1 (1 ppm)	L
xii)	Silicate (as SiO_2), percent by mass, *Max*	—	—	—	0.000 05 (0.5 ppm)	L
xiii)	Ammonium salts (as NH_3), percent by mass, *Max*	0.0005 (5 ppm)	—	—	—	M

[1)] Higher values shall apply, if agreed to between the purchaser and the supplier.

less than 25 mm high, showing:

a) Name of material;

b) Manufacturer's name and recognized trade-mark, if any;

c) Grade and mass of the material; and

d) Year of manufacture.

They shall prominently display the cautionary notice:

'CORROSIVE, HANDLE WITH CARE'

5.2.2 The data of chemical analysis with respect to the requirements prescribed in col 6 of Table 1 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).

5.2.4 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

5.2.4.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples of the material, the number of tests and criteria for conformity shall be as prescribed in Annex N.

7 QUALITY OF REAGENTS

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

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

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF TOTAL ACIDITY

A-1 OUTLINE OF THE METHOD

The acid is treated with an excess of standard sodium hydroxide solution, and the excess alkali is back titrated with standard sulphuric acid.

A-2 APPARATUS

A-2.1 Lunge-Rey Pipette, 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-3 REAGENTS

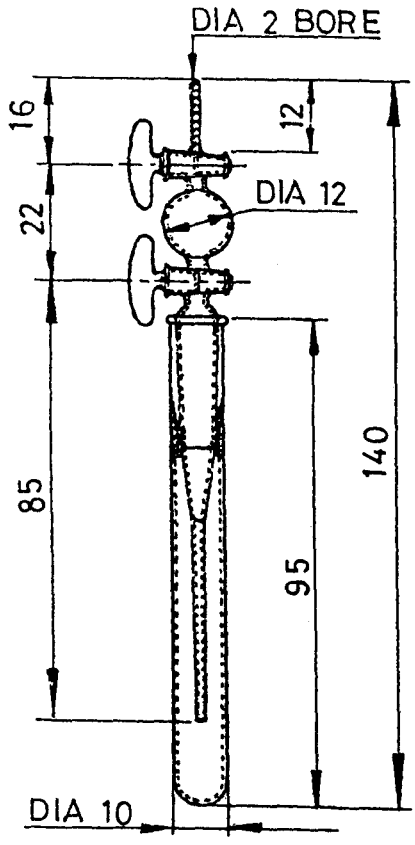
A-3.1 Standard Sodium Hydroxide Solution — 1 N.

A-3.2 Standard Sulphuric Acid — 1 N.

A-3.3 Methyl Orange Indicator — Dissolve 0.05 g of methyl orange in 100 ml of water.

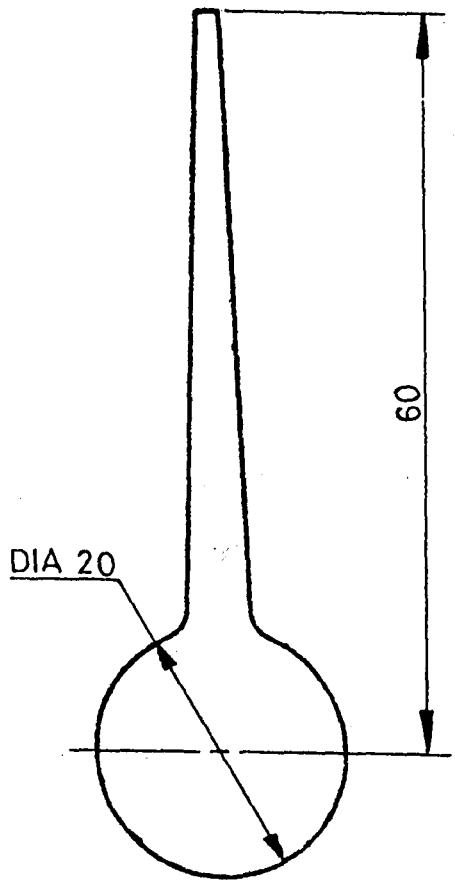
A-4 PROCEDURE

A-4.1 If Lunge-Rey pipette or an ordinary weighing



All dimensions in millimetres.

FIG. 1 LUNGE-REY PIPETTE



All dimensions in millimetres.

FIG. 2 SPHERICAL GLASS AMPOULE

bottle is used for weighing the sample, accurately weigh about 2 g of the material.

If a glass ampoule is used, take sufficient amount of the sample in a beaker or flask. Slightly heat on a flame the bulb of the glass ampoule previously weighed to the nearest 0.1 mg. Immerse the capillary end of the ampoule into the beaker or flask containing the test sample and ensure that during cooling the bulb is almost half-filled (2 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 to cool. 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.

A-4.2 If the sample was weighed in the Lunge-Rey pipette or a weighing bottle, transfer the sample to a stoppered conical flask containing 100 ml of cold water and 50 ml of standard sodium hydroxide solution.

If the sample was weighed in the ampoule, place the ampoule in the conical flask containing 100 ml of water and 50 ml of sodium hydroxide solution. Stopper the flask and while cooling, shake carefully to break the ampoule containing the test portion. 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, break up 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.

Carry out a blank test simultaneously.

A-4.3 Add two drops of methyl orange indicator to the solution in the conical flask, and titrate the excess of sodium hydroxide with standard sulphuric acid solution.

A-5 CALCULATION

$$\text{Total acidity (as } HNO_3\text{), percent by mass} = \frac{(V_1 - V_2) \times 6.3 \times N}{M}$$

where

V_1 = volume, in ml, of standard sulphuric acid required in the blank titration;

V_2 = volume, in ml, of standard sulphuric acid used in titration with the sample;

N = normality of standard sulphuric acid; and

M = mass, in g, of the test portion.

ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF RESIDUE ON INGNITION

B-1 PROCEDURE

Weigh a platinum or silica dish of 100 ml capacity and accurately weigh in it about 100 g of the sample. Evaporate the greater part of the acid (to a final volume of 5 to 10 ml) by carefully heating the dish (for example, on a boiling water bath and then on a sand bath). Remove the dish and allow it to cool to room temperature. Add 1 ml of concentrated sulphuric acid and carry on heating to dryness. Place the dish containing the residue in an electric furnace heated at 800 ± 25°C and keep at this temperature for about 15 min. Remove the dish from the furnace, place in a desiccator and weigh after cooling. Repeat the heating, cooling and weighing to constant mass.

B-2 CALCULATION

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

where

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

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

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF CHLORIDES

C-1 GENERAL

Two methods have been specified for determination of chlorides by visual comparison (Method A) and potentiometric method (Method B). However, in case of dispute, Method A shall be referee method.

C-2 METHOD A

C-2.1 Outline of the Method

Opalescence produced by a known quantity of the material with silver nitrate is compared with that produced in a control containing known quantity of chlorides.

C-2.2 Apparatus

C-2.2.1 *Nessler Cylinders* — 50 ml capacity.

C-2.3 Reagents

C-2.3.1 *Silver Nitrate Solution* — Approximately 0.1 N.

C-2.3.2 *Dilute Nitric Acid* — Approximately 4 N, chloride-free.

C-2.3.3 *Standard Chloride Solution A* — Dissolve 1.648 g of sodium chloride, previously dried at 110°C, in 1 000 ml of water. Further dilute 100 ml of the solution to 1 000 ml with water in a volumetric flask. One ml of the diluted solution contains 0.1 mg of chloride (as Cl).

C-2.3.4 *Standard Chloride Solution B* — Take 100 ml of standard chloride solution A (*see* **C-2.3.3**) and dilute with water to 1 000 ml in a volumetric flask. One ml of the solution contains 0.01 mg of chloride (as Cl).

C-2.4 Procedure

Measure 1 ml of the material in the case of technical grade and 10 ml in the case of other grades into a Nessler cylinder. Add 1 ml of silver nitrate solution and dilute to the mark. Carry out a control test using, the following quantities of dilute nitric acid and standard chloride.solution.

Sl No.	*Grade of the Material*	*Dilute Nitric Acid* ml	*Standard Chloride Solution A* ml	*Standard Solution B* ml
(1)	(2)	(3)	(4)	(5)
i)	Technical	10	4.2	–
ii)	Nitration or explosive	40	1.5	–
iii)	Chemically pure	40	1.4	–
iv)	Analytical reagent	40	–	0.7

C-2.5 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the opalescence produced with the material is not greater than that produced in the control test.

C-3 METHOD B

C-3.1 Outline of the Method

Potentiometric titration of the chloride ions with silver nitrate solution in a nitric acid-acetone-water medium, using a silver measurement electrode and a calomel reference electrode. The method is applicable to products having chloride ions contents, expressed as chloride (as Cl) equal to or greater than 0.000 2 percent *(m/m)*.

C-3.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

C-3.2.1 *Acetone*

C-3.2.2 *Nitric Acid* — Density approximately 1.40 g/ml, about 68 percent *(m/m)* solution.

C-3.2.3 *Silver Nitrate* — Approximately 0.1 N solution.

Dissolve 8.5 g of silver nitrate in water in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution in a brown glass bottle.

C-3.2.4 *Silver Nitrate, Approximately 0.01 N Solution* — Take 50 ml of the silver nitrate solution (*see* **C-3.2.3**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

C-3.2.5 *Silver Nitrate, Approximately 0.004 N Solution* — Take 20 ml of the silver nitrate solution (*see* **C-3.2.3**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

C-3.2.6 *Silver Nitrate, Approximately 0.001 N Solution* — Take 5 ml of the silver nitrate solution (*see* **C-3.2.3**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

C-3.2.7 *Potassium Chloride* — 0.1 N standard reference solution. Weigh, to the nearest 0.000 1 g, 3.727 6 g of potassium chloride, previously dried for 1 h at about 130°C and cooled in a desiccator. Dissolve in a little water, transfer the solution quantitatively to

a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution fresh.

C-3.2.8 *Potassium Chloride* — 0.01 N standard reference solution. Take 50.0 ml of the standard reference potassium chloride solution (*see* **C-3.2.7**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution fresh.

C-3.2.9 *Potassium Chloride* — 0.004 N standard reference solution. Take 20.0 ml of the standard reference potassium chloride solution (*see* **C-3.2.7**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution at the time of use.

C-3.2.10 *Potassium Chloride* — 0.001 N standard reference solution. Take 5.0 ml of the standard reference potassium chloride solution (*see* **C-3.2.7**), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution at the time of use.

C-3.3 Apparatus

C-3.3.1 *Potentiometer* — Sensitivity 2 mV, covering the range – 500 to + 500 mV.

C-3.3.2 *Calomel Electrode*, fitted with a safety reservoir, filled with saturated potassium chloride solution.

C-3.3.3 *Bridge* — Containing a saturated potassium nitrate solution, connected to the calomel electrode and fitted at the ends with porous plugs.

NOTE — This bridge is not necessary, if silver and mercury (I) sulphate electrodes are used.

C-3.3.4 *Silver Electrode*

NOTE — For the determination of chloride ions contents below 0.001 percent (*m/m*), use a silver electrode coated with a layer of silver chloride instead of the ordinary silver electrode. Prepare this electrode as follows: electrolyse a 0.1 N hydrochloric acid solution for about 30 min at a current density of 0.4 mA/cm^2, using a silver electrode (*see* **C-3.3.4**) immersed to a depth of about 5 cm as the anode and a platinum electrode as the cathode. Thoroughly wash the prepared electrode first under running water for at least 24 h then with distilled water.

C-3.3.5 *Magnetic Stirrer*, with a polytetrafluoroethylene (PTFE) coated rod.

C-3.3.6 *Microburette*, with fine-pointed tip, graduated in 0.01 ml divisions.

C-3.4 Procedure

Select the reagent solutions and test portion according to the expected chloride ions content, as indicated below:

Expected Chloride Ions Content Expressed as Cl- Percent m/m	*Silver Nitrate Solution*	*Standard Reference Potassium Chloride Solution*	*Mass of Test Portion*
(1)	(2)	(3)	(4)
For 0.000 2 up to and including 0.001	0.001 N (*see* **C-3.2.6**)	0.001 N (*see* **C-3.2.10**)	50 g, weighed to the nearest 0.01 g
Above 0.001 up to and including 0.01	0.004 N (*see* **C-3.2.5**)	0.004 N (*see* **C-3.2.9**)	20 to 10 g weighed to the nearest 0.01 g
Above 0.01 up to and including 0.1	0.01 N (*see* **C-3.2.4**)	0.01 N (*see* **C-3.2.8**)	10 to 1 g, weighed to the nearest 0.001 g
Above 0.1	0.1 N (*see* **C-3.2.3**)	0.1 N (*see* **C-3.2.7**)	3 to 1 g, weighed to the nearest 0.001 g

C-3.4.1 *Standardization of the Silver Nitrate Solution*

C-3.4.1.1 *Titration*

Take 5.00 ml and 10.00 ml of the appropriate standard reference potassium chloride solution, and place in two low-form beakers of convenient capacity (for example, 250 ml). Carry out the following titration on the contents of each beaker.

Add 5 ml of the nitric acid solution (*see* **C-3.2.2**) 120 ml of the acetone and sufficient water to bring the total volume to about 150 ml. Place the rod of the magnetic stirrer in the beaker, place the beaker on the stirrer. Stirrer and set the stirrer in motion. Immerse the silver electrode and the free end of the bridge in the solution, connect the electrodes to the potentiometer and after having verified the zero of the apparatus, note the value of the starting potential.

Titrate, using the microburette adding initially 4 or 9 ml respectively of the silver nitrate solution corresponding to the standard reference potassium

chloride solution used. Continue the addition in 0.2 ml portions for the 0.001 N solutions, in a 0.1 ml portions for the 0.004 N solutions and in 0.05 ml portions for the 0.01 N and 0.1 N solutions. After each addition, await the stabilization of the potential.

Note the volumes added and the corresponding values of the potential in the first two columns of a table.

In a third column of the table, note the successive increments ($\Delta_1 E$) of the potential E. In a fourth column, note the differences ($\Delta_2 E$), positive or negative, between the potential increments ($\Delta_1 E$) .

The end of the titration corresponds to the addition of the 0.2 or 0.1 or 0.05 ml portion (V_1) of the silver nitrate solution which gives the maximum value of $\Delta_1 E$.

In order to calculate the exact volume (V_{EQ}) of the silver nitrate solution corresponding to the end of the reaction, use the formula:

$$V_{EQ} = V_0 + V_1 \times \frac{b}{B}$$

where

V_0 = total volume, in ml of the silver nitrate solution immediately lower than the volume which gives the maximum increment of $\Delta_1 E$;

V_1 = volume, in ml, of the last portion of silver nitrate solution added (0.2 or 0.1 or 0.05 ml);

b = last positive value of $\Delta_2 E$; and

B = sum of the absolute values of the last positive value of $\Delta_2 E$ and the first negative value of $\Delta_2 E$ (*see* **C-3.6**).

C-3.4.1.2 *Calculation of concentration of the solution*

The concentration T of the silver nitrate solution, expressed as a normality, is given by the formula:

$$T = T_0 \times \frac{5}{V_2 - V_3}$$

where

T_0 = concentration, expressed as a normality, of the standard reference potassium chloride solution used;

V_2 = value, in ml, of V_{EQ} corresponding to the titration of 10 ml of the standard reference potassium chloride solution used; and

V_3 = value, in ml, of V_{EQ} corresponding to the titration of 5 ml of the standard reference potassium chloride solution used;

5 = difference, in ml, between the two volumes of the standard reference potassium chloride solution used.

C-3.4.1.3 *Calculation of the blank test result*

The result of the blank test on the reagents, V_4 is given, in ml, by formula:

$$V_4 = 2V_3 - V_2$$

where V_2 and V_3 have the same meaning as in **C-3.4.1.2**.

C-3.4.2 *Determination*

C-3.4.2.1 *Test portion*

Weigh the test portion indicated in **C-3.4** into a low-form beaker of convenient capacity (for example, 250 ml).

C-3.4.2.2 *Titration*

Add to the test portion (*see* **C-3.4.2.1**) in the beaker, 5 ml of the nitric acid solution, 120 ml of the acetone and sufficient water to bring the total volume to about 150 ml.

NOTE — For test portions of 50 g the final volume of the solution is about 160 ml. It is therefore not necessary to add water. The ratio of acetone/water is still favourable and permits a satisfactory titration.

Introduce the rod of the magnetic stirrer into the beaker, place the beaker on the stirrer and set the stirrer in motion. Immerse the silver electrode and the free end of the bridge in the solution, connect the electrodes to the potentiometer and, after having verified the zero of the apparatus, note the value of the starting potential.

Titrate with the silver nitrate solution corresponding to the test portion taken, by addition, from the microburette of 0.2 ml portions for the 0.001 N solutions of 0.1 ml portion for the 0.004 N solution and 0.05 ml portion for the 0.01 N and 0.1 N solution. After each addition, await the stabilization of the potential.

Continue the titration as specified in **C-3.4.1.1**. Note the volumes added and the corresponding values of the potential.

NOTE — If the chloride ions content is very low and therefore the volume of the appropriate silver nitrate solution used for the titration is less than approximately 1 ml, add to the test solution a known volume, exactly measured (for example, 5.00 ml) of the corresponding standard reference potassium chloride solution. Take this addition into account to the calculation of the result.

C-3.5 Expression of Results

The chloride ions content, expressed as a percentage

by mass of chloride (Cl^-), is given by the formula:

$$(V_5 - V_4) \times T \times 0.035\,45 \times \frac{100}{m}$$

$$= \frac{3.545\,T(V_5 - V_4)}{m}$$

where

T = concentration, expressed at a normality, of the silver nitrate solution used, determined according to **C-3.4.1.2**;

V_4 = result, in millilitres, of the blank test (*see* **C-3.4.1.3**);

V_5 = value, in millilitres, of V_{EQ} corresponding to the determination (*see* **C-3.4.2.2**); and

m = mass in gram of the test portion (*see* **C-3.4.2.1**);

0.035 45 = mass, in gram, of chloride ions corresponding to 1 ml of exactly 1 N silver nitrate solution.

C-3.6 Example

Volume of Silver Nitrate Solution	*Potential*		
V	E	$\Delta_1 E$	$\Delta_2 E$
ml	mV		
(1)	(2)	(3)	(4)
4.80	176		
4.90	211	35	
5.00	283	72	+ 37
5.10	306	23	− 49
5.20	319	13	− 10

$$V_{EQ} = 4.9 + 0.1 \times \frac{37}{37 + 49} = 4.943$$

ANNEX D

[*Table* 1, *Sl No.*(iv)]

DETERMINATION OF SULPHATES

D-1 GENERAL

Two methods have been specified for determination of sulphate, namely, gravimetric (for technical grade), visual comparison (for all other grades) (Method A) and reduction and titrimetry (Method B). However, in case of dispute, Method A shall be referee method.

D-2 METHOD A

D-2.1 Gravimetric Method for Technical Grade

D-2.1.1 *Outline of the Method*

Sulphates are precipitated as barium sulphate and weighed.

D-2.1.2 *Reagents*

D-2.1.2.1 *Sodium chloride* — Analytical reagent grade.

D-2.1.2.2 *Hydrochloric acid* — Analytical reagent grade.

D-2.1.2.3 *Barium chloride solution* — Approximately 10 percent *(m/v)*.

D-2.1.3 *Procedure*

Weigh accurately, using a weighing bottle, an ampoule or a Lunge-Rey pipette, about 50 g of the acid into a 100-ml porcelain dish. Add 0.5 g of sodium chloride and evaporate the contents almost to dryness on a water-bath. Add 5 ml of hydrochloric acid and transfer the contents to a 250-ml beaker using 100 ml of water. Bring the contents to boil over a low flame and, while still hot, add 5 ml of boiling barium chloride solution in slow stream, stirring all the time. Boil the contents for 2 min and allow the precipitate to settle for 4 h. Filter the supernatant liquid through a tared sintered glass crucible (G No. 4) or tared Gooch crucible and transfer the precipitate carefully into the crucible. Wash thoroughly with hot water till the washings are free from chloride. Heat the crucible at 105 to 110°C to constant mass.

D-2.1.4 *Calculation*

$$\text{Sulphate (as } H_2SO_4\text{), percent by mass} = \frac{M_1 \times 42.02}{M_2}$$

where

M_1 = mass, in g, of the precipitate, and

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

D-2.2 Visual Comparison Method for Nitration, Explosive, Chemically Pure and Analytical Reagent Grade

D-2.2.1 *Outline of the Method*

The turbidity produced by a known quantity of the material with barium chloride is compared with that produced in a control test with known quantity of sulphates.

D-2.2.2 *Apparatus*

D-2.2.2.1 *Nessler cylinders* — 50 ml capacity.

D-2.2.3 *Reagents*

D-2.2.3.1 *Sodium carbonate* — Analytical reagent grade.

D-2.2.3.2 *Dilute hydrochloric acid* — Approximately 1 N.

D-2.2.3.3 *Barium chloride solution* — Approximately 10 percent *(m/v)*.

D-2.2.3.4 *Standard sulphate solution* — Dissolve 0.134 7 g of ammonium sulphate in water and make up the solution to 1 000 ml. One millilitre of the solution contains 0.1 mg of sulphate (as H_2SO_4).

D-2.2.4 *Procedure*

Measure out the following volume of the sample in a 100-ml porcelain dish:

a) 10 ml in the case of nitration, explosive and chemically pure grades, and

b) 20 ml in the case of analytical reagent grade.

Add 0.02 g of sodium chloride and evaporate to dryness. Take up the residue with 5 ml of water and 0.5 ml of hydrochloric acid. Filter if necessary and in that case wash with water and collect the filtrate and washings in a Nessler cylinder. Make up to the mark and then add 1 ml of barium chloride solution. Carry out a control test under similar conditions using the quantity of standard sulphate solution as given below:

a) For nitration and explosive grades, 7.5 ml;

b) For chemically pure grade, 7.0 ml; and

c) For analytical reagent grade, 0.55 ml.

D-2.2.5 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the turbidity produced with the material is not greater than that produced in the control test.

D-3 REDUCTION AND TITRIMETRIC METHOD (METHOD B)

D-3.1 Outline of the Method

Evaporation of a test portion of dryness in the presence of sodium carbonate. Reduction of the sulphate to sulphide by a mixture of hydriodic acid and hypophosphorous acid. Absorption of the hydrogen sulphide evolved in a mixture of acetone and sodium hydroxide solution. Titration with a standard volumetric mercury (II) acetate solution in the presence of dithizone as indicator. The method is applicable to products of which the sulphate content, expressed as SO_4 is the equal to or greater than 0.000 1 percent *(m/m)*.

D-3.2 Reagents

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

D-3.2.1 *Hydriodic Acid* — Density approximately 1.71 g/ml, about 57 percent *(m/m)* solution.

D-3.2.2 *Hypophosphorous Acid* [$H(H_2PO_2]$ — Density approximately 1.21 g/ml, about 50 percent *(m/m)* solution.

D-3.2.3 *Hydrochloric Acid* — Density approximately 1.19 g/ml, about 38 percent *(m/m)* solution.

D-3.2.4 *Reduction Solution* — Into a 1 000 ml flask, fitted with a ground glass socket capable of accepting a reflux condenser, introduce, in the following order, under a current of nitrogen (*see* **D-3.2.11**), 100 ml of the hydriodic acid solution (*see* **D-3.2.1**), 25 ml of the hypophosphorous acid solution (*see* **D-3.2.2**) and 100 ml of the hydrochloric acid solution (*see* **D-3.2.3**). Fit the reflux condenser to the flask and, while bubbling a gentle current of nitrogen through the mixture, boil under reflux for 4 h. Then cool to room temperature, maintaining the current of nitrogen. Store the solution in a dark glass flask, previously purged with nitrogen, fitted with a ground glass stopper.

D-3.2.5 *Sodium Sulphate* — 0.001 M *Standard Reference Solution* — Weigh, to the nearest 0.000 1 g, 0.142 0 g of anhydrous sodium sulphate, previously dried at 110°C and cooled in a desiccator. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water dilute to the mark and mix.

One ml of this solution corresponds to 96 µg of SO_4.

D-3.2.6 *Mercury (II) Acetate — 0.001 M Standard Volumetric Solution* — Weigh, to the nearest 0.000 1 g, 0.318 7 g mercury (II) acetate [$Hg(CH_3.COO)_2$]. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

Alternatively, 0.001 M standard volumetric mercury (II) nitrate solution may be used.

D-3.2.6.1 *Mercury (II) nitrate — 0.001 M standard volumetric solution* — Weigh, 10.85 ± 0.01 g of mercury (II) oxide (HgO). Place in a beaker of suitable capacity (for example, 100 ml) and dissolve in 10 ml of nitric acid solution, density approximately 1.40 g/ml, about 68 percent *(m/m)* solution. Dilute the solution, transfer it quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. The solution thus obtained is 0.05 M (corresponding to 0.1 N). Take 20.00 ml of this solution, introduce into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution just before use.

NOTES

1 In most laboratories, an exactly 0.1 N standard volumetric mercury (II) nitrate solution (corresponding to a 0.05 M solution) will be available, this solution being commonly used in the mercurimetric determination of chlorides.

2 The strengths of solutions (*see* **D-3.2.6** and **D-3.2.6.1**) prepared as described above are sufficiently exact, taking into consideration the small quantities of sulphates to be determined. Standardization is therefore, unnecessary.

D-3.2.7 *Acetone*

D-3.2.8 *Dithizone* — 0.5 g/l solution in acetone.

D-3.2.9 *Sodium Hydroxide* — Approximately 1 N solution.

D-3.2.10 *Sodium Carbonate* — Anhydrous.

D-3.2.11 *Nitrogen*

D-3.3 Apparatus

D-3.3.1 *Microburette* — Graduated in 0.001 ml.

D-3.3.2 *Apparatus for Reduction and Distillation* (*see* Fig. 3 for a typical example), off which all the components are fitted together by means of ground glass joints.

D-3.4 Procedure

D-3.4.1 *Test Portion and Preparation of the Test Solution*

Weigh, to the nearest 0.000 1 g, a mass of the test sample containing between 0.5 and 2 mg of SO_4.

Place in a beaker of convenient capacity, add 20 to 30 mg of the sodium carbonate and evaporate to dryness in a fume-cupboard. Dissolve the residue in 10.00 ml of water, slightly acidified with the hydrochloric acid solution. Prepare this solution at the time of use.

D-3.4.2 *Check Test*

The purpose of this test is to check the gas-tightness and functioning of the apparatus (reduction of sulphur compounds and quantitative recovery of the hydrogen sulphide liberated).

Introduce 5 ml of the sodium hydroxide solution 5 ml of the acetone and 0.1 ml of the dithizone solution into the test tube of the apparatus (*see* **D-3.3.2**).

Mix and add, drop by drop, the mercury (II) acetate solution till the colour changes from yellow to pink.

Introduce 2.00 ml of the standard reference sodium sulphate solution (*see* **D-3.2.5**) into a clear and dry reduction flask. Fit the components of the apparatus (*see* **D-3.3.2**) together as indicated in the figure taking care to smear the ground joints lightly with a silicon grease and to tighten them with suitable spring-clips in order to ensure perfect gas-tightness. Pass a gentle flow of nitrogen through the inlet tube (2 to 3 bubbles per second). Then run 15 ml of the reduction solution into the reduction flask through the dropping funnel, under a slight pressure of nitrogen.

Maintaining the current of nitrogen through the inlet tube, immerse the reduction flask partially (about 70 mm) in a boiling water bath for at least 30 min. Then allow the nitrogen to flow through the dropping funnel for 5 min.

The hydrogen sulphide liberated by the reaction is absorbed in the solution contained in the test tube which becomes yellow.

NOTE — Development of a blue-green coloration indicates that a large quantity of hydrochloric acid has been entrained and the test must be repeated.

Disconnect the apparatus and wash the connection tube with several millilitres of a mixture of equal volumes of water and the acetone collecting the washings in the test tube. Titrate the solution obtained with standard volumetric mercury (II) acetate solution (*see* **D-3.2.6**) contained in the microburette, until a new pink coloration is obtained.

The result of the check test is considered satisfactory if the volume of the standard volumetric mercury (II) acetate solution used for the titration is between 1.90 and 2.10 ml , after subtraction of the blank test result. Otherwise, check the apparatus for leaks.

D-3.4.3 *Determination*

Place 1.00 ml of the test solution (*see* **D-3.4.1**) in the reduction flask of the apparatus and connect the different parts of the apparatus. Introduce 5 ml of the sodium hydroxide solution, 5 ml of the acetone and 0.1 ml of the dithizone solution into the test tube. Mix and add, drop by drop, the standard volumetric mercury (II) acetate solution until the colour changes from yellow to pink. Pass a current of nitrogen through the apparatus and run in, through the dropping funnel, 15 ml of the reduction solution under a slight pressure of nitrogen.

Continue as specified on **D-3.4.2** for the check test starting from the fifth paragraph.

D-3.4.4 *Blank Test*

Carry out a blank test under the same conditions as the determination (*see* **D-3.4.3**) but using 1.00 ml of water instead of 1.00 ml of the test solution (*see* **D-3.4.1**).

D-3.5 Calculation

The sulphate content expressed as a percentage by mass of sulphate (SO_4) is given by the formula:

$$\frac{(V_1 - V_0) \times 96 \times 10 \times 100}{1\,000\,000\, m_0} = \frac{(V_1 - V_0) \times 0.096}{m_0}$$

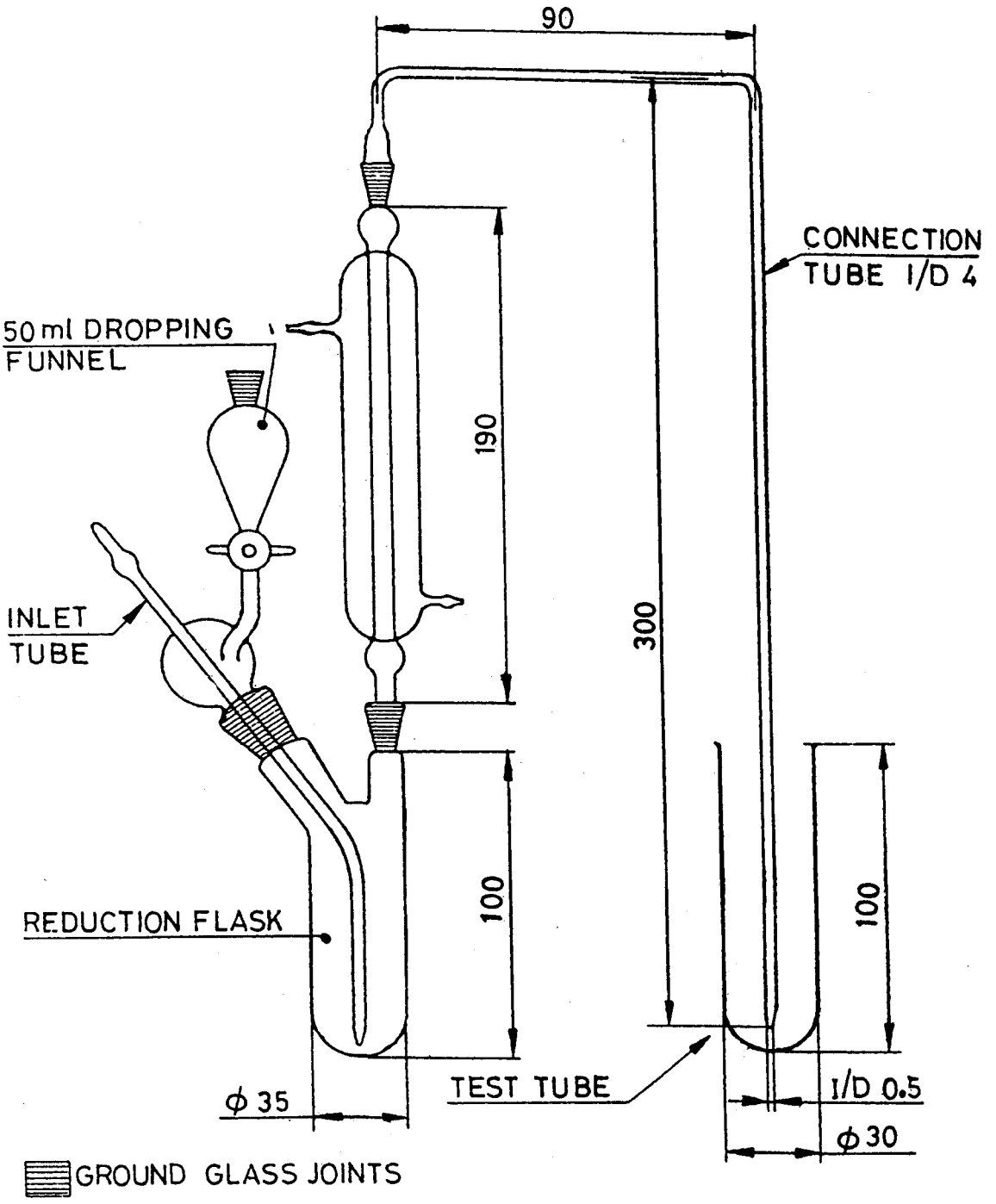
where

V_0 = volume, in ml, of the standard volumetric mercury (II) acetate solution (*see* **D-3.2.6**) used for the blank test;

V_1 = volume, in ml, of the standard volumetric mercury (II) acetate solution (*see* **D-3.2.6**) used for the determination;

m_0 = mass in g of the test portion (*see* **D-3.4.1**); and

96 = mass, in micrograms, of SO_4 corresponding to 1 ml of the standard volumetric mercury (II) acetate solution.



All dimensions in millimetres.

FIG. 3 TYPICAL APPARATUS FOR REDUCTION AND DISTILLATION

ANNEX E

[*Table* 1, *Sl No.* (v)]

TEST FOR HEAVY METALS

E-1 TECHNICAL, NITRATION, EXPLOSIVE AND CHEMICALLY PURE GRADES

E-1.1 Outline of the Method

The acid is neutralized with ammonia, and iron oxide, if precipitated, is removed. Hydrogen sulphide is passed into the solution which is then examined for formation of lead sulphide.

E-1.2 Reagents

E-1.2.1 *Ammonium Hydroxide* — 20 percent *(m/v)*.

E-1.2.2 *Hydrogen Sulphide Gas*, from Kipp's generator.

E-1.3 Procedure

In a conical flask of 250 ml capacity, take 100 ml of water and measure into it 10 ml of the acid. Slowly add ammonium hydroxide, swirling the content of the flask till a distinct smell of ammonia is obtained. Keep aside for 10 min. Filter, if a precipitate of iron oxide is noticed on standing. Bubble hydrogen sulphide gas through the solution rapidly for 5 min.

E-1.3.1 The material shall be taken to have passed the test if the solution does not become darker than light brown in colour or does not deposit any precipitate.

E-2 ANALYTICAL REAGENT GRADE

E-2.1 Procedure

Weigh accurately about 25 g of the material and determine heavy metals (as lead) by the colorimetric method using dithizone as prescribed in IS 7017.

ANNEX F

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF NITROUS ACID

F-1 OUTLINE OF THE METHOD

Nitrous acid is oxidized by standard potassium permanganate solution and excess of potassium permanganate is determined volumetrically.

F-2 REAGENTS

F-2.1 Dilute Sulphuric Acid — Approximately 4 N.

F-2.2 Standard Potassium Permanganate Solution — 0.1 N.

F-2.3 Standard Ferrous Ammonium Sulphate Solution — 0.1 N.

F-3 PROCEDURE

In a ground glass stoppered conical flask, take 100 ml of water previously cooled to 0°C, and 20 ml of dilute sulphuric acid previously cooled to 0°C. Add a known volume V_1 (which will provide an excess of about 10 ml after reaction with nitrous compounds) of standard potassium permanganate solution. Quickly pour about 20 g (accurately weighed) of the sample, close the flask immediately and shake (for approximately 5 min) until all the fumes have disappeared. Add 20.0 ml of ferrous ammonium sulphate solution and titrate the excess with potassium permanganate solution until a pink colour is obtained that lasts for 1 min (V_2). In order to establish the equivalence of the two solutions under the conditions of the determination, add to the same flask a further 20.0 ml of ferrous ammonium sulphate solution and titrate with potassium permanganate solution (V_3).

F-4 CALCULATION

$$\text{Nitrous acid (as } HNO_2\text{), percent by mass} = \frac{[(V_1 + V_0) - V_3] \times 0.235}{m}$$

where

V_1 = volume, in ml, of standard potassium permanganate solution added at the beginning;

V_2 = volume, in ml, of standard potassium permanganate solution used for the first titration;

V_3 = volume, in ml, of standard potassium permanganate solution used for the second titration; and

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

ANNEX G

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF ARSENIC

G-1 REAGENT

G-1.1 Concentrated Sulphuric Acid

G-2 PROCEDURE

G-2.1 For Chemically Pure Grade

Weigh accurately about 2 g of the material and expel nitric acid by evaporation with 5 ml of concentrated sulphuric acid till sulphur trioxide fumes are given out. Determine arsenic in the liquid residue by silver diethyl dithiocarbamate method as prescribed in 1S 2088.

G-2.2 For Analytical Reagent Grade

To 250 g of the sample, accurately weighed, add 5 ml of sulphuric acid and evaporate till fumes of sulphuric acid are evolved. Cool, add 5 ml of water and again evaporate to fuming. Cool, dilute with water to adjust the final volume of the solution to 5 ± 0.5 ml and determine arsenic by silver diethyl dithiocarbamate method as prescribed in IS 2088.

ANNEX H

[*Table* 1, *Sl No.* (viii)]

TEST FOR IODINE

H-1 OUTLINE OF THE METHOD

Iodine is liberated by passing sulphur dioxide in the material. The presence of iodine is indicated by development of violet colour in carbon tetrachloride.

H-2 APPARATUS

H- 2.1 Separating Funnel

Cylindrical, of 150 ml capacity and provided with a 3-holed stopper.

H-3 REAGENTS

H-3.1 Carbon Tetrachloride

H-3.2 Sulphur Dioxide

H-3.3 Carbon Dioxide

H-4 PROCEDURE

Place carbon tetrachloride in the separating funnel in sufficient amount to provide a depth of about 12 mm above the shoulder. Add 20 ml of the material and 30 ml of water and close the funnel with the 3 holed stopper. Fit a soda-lime tube through one of the holes, a tap funnel through another and a gas inlet tube through the third, reaching below the solvent layer. Shake the mixture, allow it. To cool, and pass a steady stream of sulphur dioxide for 5 min. After standing for 15 min, pass carbon dioxide for 2 min in a sufficiently rapid stream to mix the liquid layers. Stop the gas stream and run out the solvent to the height of the shoulder.

H-5 The material shall be considered to have passed the test if there is no violet colour in the solvent.

ANNEX J

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF IRON

J-1 GENERAL

Two methods are prescribed. Method A shall be the referee method and Method B the alternative method.

J-2 METHOD A (BIPYRIDYL METHOD)

J-2.1 Outline of the Method

After evaporation to dryness with hydrochloric acid, iron is reduced to ferrous state, and the colour developed with 2,2′-bipyridyl is measured by a photometer.

J-2.2 Apparatus

J-2.2.1 *Spectrophometer or Photoelectric Absorptiometer* — Any spectrophotometer suitable for measurement at a wavelength of about 522 nm or photoelectric absorptiometer with appropriate filter may be used.

J-2.3 Reagents

J-2.3.1 *Concentrated Hydrochloric Acid*

J-2.3.2 *Dilute Hydrochloric Acid*— 1 N approximately.

J-2.3.3 *Hydroxylammonium Chloride Solution* — Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

J-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 with water.

J-2.3.5 *Ammonium Acetate Solution* — 30 percent (*m/v*).

J-2.3.6 *Standard Iron Solution A* — Dissolve 0.702 2 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4$, $6H_2O$] in water in a 1 000-ml volumetric flask, add 4 ml of concentrated sulphuric acid and make up with water to the mark. One millilitre of this solution contains 0.1 mg of iron (as Fe).

J-2.3.7 *Standard Iron Solution B* — Take 100 ml of standard iron solution *A* and dilute to 1 000 ml in a volumetric flask. One millilitre of this solution contains 0.01 mg of iron (as Fe). This solution should be prepared fresh.

J-2.4 Procedure

J-2.4.1 Weigh accurately about 50 g of the sample in a platinum or quartz dish (100 ml capacity) and place on a boiling water bath and evaporate to dryness. Cool, take up with 2 ml of concentrated hydrochloric acid and 5 ml of water. Evaporate a second time on a boiling water bath. Take up with 2 ml of concentrated hydrochloric acid and 25 ml of water, warming to assist solution. Transfer quantitatively to a 100-ml one-mark volumetric flask, dilute to the mark, mix and filter, if necessary, with a dry filter into a dry vessel. Transfer an aliquot of the sample solution containing between 50 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 dilute, hydrochloric acid 2 ml of hydroxylammonium 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 photometric measurement using either the spectrophotometer at a wavelength of about 522 nm, or the photoelectric absorptiometer with suitable filter, adjusting the instrument to zero absorbance, using the blank test solution as reference.

J-2.4.2 *Blank Test*

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

J-2.4.3 *Preparation of Calibration Curve*

Take the quantities of standard iron solution B indicated in the following table into a series of eleven 100-ml volumetric flask:

Volume of Standard Iron Solution B, ml	*Corresponding Mass of Iron (Fe)*, µg
(1)	(2)
0 (Compensation)	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 dilute hydrochloric acid, 2 ml of hydroxylammonium 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 thoroughly and wait for 10 min.

Carry out the photometric measurements as in **J-2.4.1** using the compensation solution as reference, and prepare a calibration graph having, for example, the iron content in micrograms per 100 ml of the standard matching solution as abscissae and the corresponding values of absorbance as ordinates.

J-2.5 Calculation

By reference to the calibration chart read the iron content corresponding to the photometric measurement.

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

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 test portion.

J-3 METHOD B (THIOCYANATE METHOD)

J-3.1 Outline of the Method

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

J-3.2 Reagents

J-3.2.1 *Hydrochloric Acid* — 1 : 1 (*v/v*).

J-3.2.2 *Potassium Permanganate Solution* — 0.1 N.

J-3.2.3 *Ammonium Thiocyanate Solution* — 60 percent (*m/v*).

J-3.2.4 *Mixture of Amyl Alcohol and Amyl Acetate* — 1:1 (*v/v*).

J-3.2.5 *Standard Iron Solution* — Same as in **J -2.3.7**.

J-3.3 Procedure

J-3.3.1 Accurately weigh 50 g of the sample in a platinum or quartz dish (100 ml capacity), evaporate to dryness on a steam bath, and dissolve the residue

by heating to boiling with a mixture of 3 ml of hydrochloric acid and 10 ml of water. Transfer to a 100 ml (or required volume) separating funnel. Cool and dilute to 50 ml.

J-3.3.2 Add 1 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, shake vigorously and allow to separate.

J-3.3.3 Compare any red colour produced in the upper layer with standard prepared as in **J-3.3.4**

J-3.3.4 Carry out a control test using 1 ml of standard iron solution *B* (*see* **J-3.3.7**), adding 1 ml of hydrochloric acid and diluting with water to the same volume as the acidified solution of the test sample. Proceed as in **J-3.3.2.**

J-3.4 The material shall be taken to have not exceeded the limit prescribed in Table 1 if the intensity of the colour produced in the test with the sample is not greater than that produced in the control test.

ANNEX K

[*Table* 1, *Sl No.* (x)]

TEST FOR MANGANESE

K-1 APPARATUS

K-1.1 Nessler Cylinders — 50 ml capacity.

K-2 REAGENTS

K-2.1 Sodium Carbonate Solution — 1 N approximately.

K-2.2 Sulphuric Acid — 4 N approximately.

K-2.3 Standard Manganese Solution *A* — Dissolve 0.406 g of manganous sulphate tetrahydrate or 0.308 g of manganous sulphate monohydrate in a mixture of 50 ml of dilute sulphuric acid and 100 ml of water, and make up the volume to 1 000 ml with water. One millilitre of the solution contains 0.1 mg of manganese (as Mn).

K-2.4 Standard Manganese Solution *B* — Take 100 ml of standard manganese solution *A* (*see* **K-2.2.1**) and dilute it to 1 000 ml with water immediately before use. One millilitre of this solution contains 0.01 mg of manganese (as Mn).

K-2.5 Sodium Bismuthate — Free from manganese.

K-3 PROCEDURE

Accurately weigh about 25 g of the material in a platinum or silica crucible, add 0.2 ml of sodium carbonate solution and evaporate to dryness on a steam-bath. Dissolve the residue in a mixture of 35 ml of water and 15 ml of the sample. Prepare a standard by mixing 1 ml of standard manganese solution *B* with 35 ml of water and 15 ml of the sample. To each solution add 2 g of sodium bismuthate, boil until just clear and cool rapidly under running water. Any pink colour produced in the test should not be deeper than that of the standard when compared in Nessler cylinders.

ANNEX L

[*Table* 1, *Sl No.* (xi) *and* (xii)]

TEST FOR PHOSPHATE AND SILICATE

L-1 APPARATUS

L-1.1 Separating Funnels — 200 to 250 ml capacity.

L-1.2 *p*H Meter — With glass electrode.

L-1.2.1 Alternatively, universal standard indicator paper may be used.

L-1.3 Nessler Cylinders — 50 ml capacity.

L-2 REAGENTS

L-2.1 Dilute Sulphuric Acid — Approximately 5 percent (*v/v*).

L-2.2 Ammonium Molybdate — Solid.

L-2.3 Concentrated Hydrochloric Acid — Conforming to IS 265.

L-2.4 Dilute Hydrochloric Acid — Approximately 10 percent (*v/v*).

L-2.5 Dilute Hydrochloric Acid — 1 percent (*v/v*).

L-2.6 Ethyl Ether — Conforming to IS 336.

L-2.7 Stannous Chloride Solution — Dissolve 2 g of stannous chloride ($SnCl_2.2H_2O$) in concentrated hydrochloric acid and dilute to 100 ml with the acid.

L-2.8 Butanol

L-2.9 Standard Phosphate Solution — Dissolve 1.43 g of potassium dihydrogen orthophosphate (KH_2PO_4) in water and dilute to 1 000 ml in a volumetric flask. One millilitre of this solution contains 1.0 mg of phosphate (as PO_4).

L-2.10 Standard Silicate Solution

Dissolve 25 g of sodium metasilicate ($Na_2SiO_3.9H_2O$) in water in a polythene beaker, dilute to 500 ml and store in polythene container. Determine the silicate content (as SiO_2) of this solution and dilute a suitable aliquot to give a solution containing 1 mg of silicate (as SiO_2) in 1 ml.

L-2.11 Standard Phosphate-Silicate Solution

Take 10 ml of standard phosphate solution (*see* **L-2.9**) and 5 ml of standard silicate solution (*see* **L-2.10**) and dilute to 1 000 ml with water in volumetric flask. One millilitre of this diluted solution is equivalent to 0.01 mg of phosphate (as PO_4) and 0.005 mg of silicate (as SiO_2). This standard phosphate-silicate solution should be freshly prepared before use.

L-3 PROCEDURE

Accurately weigh and transfer about 10 g of the material into a platinum dish and evaporate on steam-bath until reduced to 0.5 to 1.0 ml. Then dilute it with water, neutralize with cautious addition of sodium carbonate, adjust the *p*H to about 4 by addition of dilute sulphuric acid and dilute to a volume of about 75 ml. Note the quantity of sodium carbonate added. Take 1 ml of standard phosphate-silicate solution. Add to it the same quantity of sodium carbonate as added to the sample solution. Add 0.5 g of ammonium molybdate each to the test solution and the standard phosphate-silicate solution and when it dissolves adjust the *p*H to 2 by adding dilute hydrochloric acid. Check the *p*H with glass electrode or universal *p*H indicator paper. Heat both the solutions to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid to each and dilute to 100 ml with water. Transfer the solutions to two separating funnels, add 35 ml of ether to each, shake vigorously and allow to separate. Draw off the aqueous phases. Proceed for the determination of phosphate in the ether phase as given in **L-3.1**. Determine silicate in the aqueous phase as given in **L-3.2**.

L-3.1 Test for Phosphate

Wash the ether phase of each funnel from **L-3** by shaking with 10 ml of dilute hydrochloric acid, allow to separate, and drain off and discard the aqueous phase. Add 0.2 ml of freshly prepared stannous chloride solution to each ether extract and shake. If the ether extracts are turbid, wash with 10 ml of dilute hydrochloric acid. Transfer the ether extracts to the Nessler cylinders. The limit prescribed for phosphate in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

L-3.2 Test for Silicate

To each of the aqueous phases from **L-3** add 10 ml of concentrated hydrochloric acid and transfer to separating funnels. Add 40 ml of butanol to each, shake vigorously, and allow to separate. Draw off and discard the aqueous phases. Wash the butanol solutions three times with 20-ml portions of dilute hydrochloric acid, discarding the washing each time. Dilute each butanol solution to 50 ml, take a 10-ml aliquot from each and dilute to 50 ml with butanol. Add 0.5 ml of freshly prepared 2 percent stannous chloride solution to each and shake. If the butanol extracts are turbid, wash with 10 ml of dilute hydrochloric acid. Transfer the butanol extracts to the Nessler cylinders. The limit prescribed for silicate in Table 1 shall be taken. As not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

ANNEX M

[*Table* 1, *Sl No.*(xiii)]

TEST FOR AMMONIUM SALTS

M-1 GENERAL

Two methods have been specified for determination of ammonium salts, namely, visual comparison (Method A) and spectrophotometer method (Method B). However in case of dispute, Method A shall be the referee method.

M-2 METHOD A

M-2.1 Apparatus

M-2.1.1 *Kjeldahl Flask* — 800 ml capacity.

M-2.1.2 *Distillation Trap*

M-2.1.3 *Nessler Cylinders* — 50 ml capacity.

M-2.2 Reagents

M-2.2.1 *Sodium Hydroxide Solution* — 42 percent (*m/v*).

M-2.2.2 *Boric Acid* — Saturated solution.

M-2.2.3 *Nessler's Reagent* — Dissolve 35 g of potassium iodide in 100 ml of water and add 4 percent

mercuric chloride solution with stirring until a slight red precipitate remains. Then introduce, with stirring, a solution of 120 g of sodium hydroxide in 250 ml of water and make up to 1 000 ml with distilled water. Add a little more mercuric chloride solution until there is a permanent turbidity. Allow the mixture to stand for 1 day and decant. Keep the solution stoppered in a dark coloured bottle.

M-2.2.4 *Standard Ammonia Solution* — Dissolve 4.706 g of ammonium nitrate (dried at 105°C for 1 h) in water and make up to 1 000 ml. Dilute 1 ml of this solution to 100 ml with water immediately before use. One millilitre of the diluted solution is equivalent to 0.01 mg of ammonia.

M-2.3 Procedure

Take 20 g of the sample in Kjeldahl flask and add about 250 ml of water. Mount the flask on a heater. Take 25 ml of saturated boric acid solution in a 600-ml beaker and keep it below the distillation trap. The tip of the trap should dip in the acid. Run 80 to 90 ml of 40 percent sodium hydroxide solution into the flask and connect it quickly to the distillation trap (2 to 3 drops of methyl red may be added to the flask before the addition of sodium hydroxide to ensure complete neutralization of the acid in the flask). Gradually heat the flask until about 200 ml of distillate is collected. Dilute the distillate to 250 ml in a volumetric flask. Carry out a blank test in a similar way using all the reagents except the sample. Dilute the distillate from the blank test also to 250 ml. Take 5-ml aliquot of the distillate obtained in test with the sample in a Nessler cylinder, add 1 ml of Nessler's reagent and dilute to the mark (if required the aliquot may be further diluted). Similarly take 5-ml aliquot of distillate from blank test in another Nessler cylinder and add standard ammonium solution from the pipette. Add 1 ml of Nessler's reagent. Prepare a series of standards to match the colour with that produced with the sample and calculate the ammonia present in the sample.

M-3 SPECTROPHOTOMETRIC METHOD (METHOD B)

M-3.1 Outline of the Method

Distillation, with entertainment by steam, of the ammonia in the presence of an excess of sodium hydroxide and collection of the distillate in an excess of acid solution. Neutralization of the excess of acid and formation of the coloured complex by treatment with sodium phenate and sodium hypochlorite in the presence of acetone. Spectrophotometric measurement of the indophenol obtained at a wavelength of about 630 nm. The procedure as described, is suitable for contents of ammoniacal nitrogen between 0.000 1 and 0.000 5 percent (*m/m*). If the acid analyzed contain more than 0.000 5 percent (*m/m*) of ammoniacal nitrogen, the mass of the test portion should be decreased accordingly.

M-3.2 Reagents

M-3.2.1 *Sodium Hydroxide* — 350 g/l solution. Boil this solution of 20 min to remove traces of ammoniacal nitrogen and make up to the original volume.

M-3.2.2 *Sulphuric Acid* — Approximately 0.1 N solution.

M-3.2.3 *Sodium Hydroxide* — Approximately 1 N solution.

M-3.2.4 *Sodium Hydroxide* — Approximately 0.1 N solution.

M-3.2.5 *Phenolphthalein*, 10 g/l *Ethanolic Solution* — Dissolve 1 g of phenolphthalein in 95 percent (*v/v*) ethanol and dilute to 100 ml with the same ethanol.

M-3.2.6 *Acetone*

M-3.2.7 *Sodium Phenate* (*Sodium Phenolate*) — Approximately 155 g/l solution. Dissolve 12.5 g of phenol in 27 ml of approximately 5 N sodium hydroxide solution and dilute to 100 ml. Immediately place the solution in the dark. Prepare this solution immediately before it is required for use.

M-3.2.8 *Sodium Hypochlorite* — Solution containing 10 g of available chlorine per litre. Dilute a concentrated solution of sodium hypochlorite (100 to 140 g of available chlorine per litre), previously standardized against a solution of sodium arsenite. Do not use concentrated solutions containing less than 80 g of available chlorine per litre. Store the solution in a cool place and in the absence of light. The solution is stable for about 4 weeks.

M-3.2.9 *Ammonium Chloride* — Standard solution corresponding to 1 g of ammoniacal nitrogen per litre. Weigh, to the nearest 0.000 1 g, 3.819 g of ammonium chloride, previously dried at 100°C and allowed to cool in a desiccator. Place in a beaker of suitable capacity and dissolve in water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. One ml of this solution contains 1 mg of ammoniacal nitrogen. Renew the solution at least once a month.

M-3.2.10 *Ammonium Chloride* — Standard solution corresponding to 0.1 g of ammoniacal nitrogen per litre. Transfer 50.0 ml of the standard solution (*see* **M-3.2.9**) to a 500 ml one-mark volumetric flask, dilute to the mark and mix. One ml of the solution contains 0.1 mg of ammoniacal nitrogen. Renew this solution at least once every 15 days.

M-3.2.11 *Ammonium Chloride* — Standard solution corresponding to 1 mg of ammoniacal nitrogen per litre. Transfer 10.0 ml of the standard solution (**M-3.2.10**) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. One ml of this solution

contains 1 µg of ammoniacal nitrogen. Prepare this solution immediately before it is required for use.

M-3.3 Apparatus

Ordinary laboratories apparatus.

M-3.3.1 *Apparatus with Ground Glass Joints, for Steam Distillation* (*see* Fig. 4, which shows a typical example).

M-3.3.1.1 *Construction*

a) *Steam trap* — for the condensed water, fitted on one side to a steam generator and on the other side to the flask *B*;

b) *Distillation flask* — 500 ml capacity, fitted with an inlet for the introduction of the steam and with a dropping funnel with a PTFE (Polytetrafluoroethylene) stopcock, closed by a tight bung and connected to the condenser, *C*, through a spray trap.

c) *Condenser* — fitted with a tapered extension dipping into the conical flask, *D*, and having on its base a glass collar to stop any external condensation running into the flask *D*;

d) *Conical flask or cylinder* — about 250 ml capacity;

e) Apparatus for heating the flask.

M-3.3.1.2 *Precautions to be taken before, during and after use*

The apparatus should be tightly sealed; ensure that the ground glass joints are not too close to the source of heat. When the apparatus is exposed to the laboratory atmosphere, its wall can absorb traces of ammoniacal nitrogen which are not removed by rinsing with water. It is essential to wash the apparatus by carrying out one or two blank distillations in the presence of sodium hydroxide solution.

After this distillation, and between tests, keep the apparatus sealed from the laboratory atmosphere. Keep the dropping funnel sealed and the end of the delivery tube dipped in water or dilute acid.

M-3.3.2 p*H Meter*

With glass electrode.

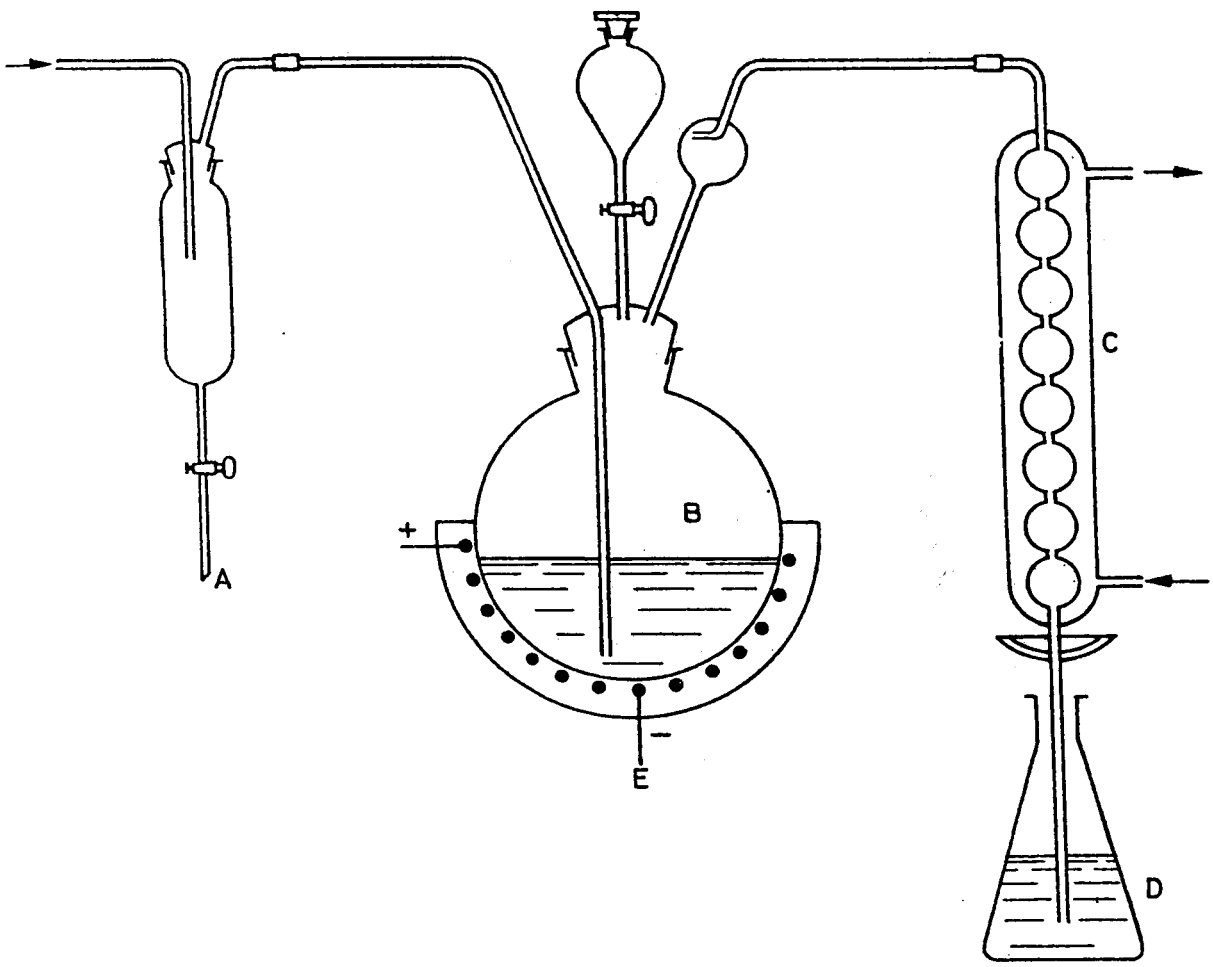


FIG. 4 TYPICAL APPARATUS FOR STEAM DISTILLATION

M-3.3.3 *Spectrophotometer*

M-3.4 Procedure

M-3.4.1 *Test Portion*

Fill a weighing pipette with the test sample and take, weighing by difference to the nearest, 0.010 g, a test portion of about 50 g. Place it in a beaker of convenient capacity (for example, 250 ml). Evaporate the nitric acid on a boiling water bath, in a fume cupboard, until the volume is reduced to about 1 ml.

M-3.4.2 *Blank Test*

Carry out, at the same time as determination, and following the same procedure, a blank test using the same quantities all the reagents used for the test.

M-3.4.3 *Preparation of Calibration Curve*

M-3.4.3.1 *Preparation of standard matching solutions*, relating to measurements carried out with an optical path length of 1 cm.

Into a series of six 50 ml one-mark volumetric flasks, introduce the quantities of the standard ammonium chloride (*see* **M-3.2.11**) solution shown in the following table:

Standard Ammonium Chloride Solution (*see* **M-3.2.11**) ml	*Corresponding Mass of Ammoniacal Nitrogen* µg
(1)	(2)
0 (Compensation)	0
5.0	5
10.0	10
15.0	15
20.0	20
25.0	25

Add to each flask the quantity of water necessary to give a volume of 25 ml, then add 0.3 ml of the acetone and stir. Introduce into each flask stirring in all cases and using a rapid delivery pipette, 10 ml of the sodium phenate solution and, immediately afterwards, 5 ml of the sodium hypochlorite solution, using a rapid delivery pipette for this reagent also. Dilute to the mark and mix. Allow each flask to stand, shielded from light, at ambient temperature for 60 ± 5 min.

M-3.4.3.2 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements, using the spectrophotometer at a wavelength of about 630 nm, after having adjusted the instrument to zero absorbance against the compensation solution.

M-3.4.3.3 *Preparation of calibration chart*

Plot a graph having, for example, the numbers of micrograms of ammoniacal nitrogen in 50 ml of the standard matching solutions as abscissae and the corresponding values of absorbance as ordinates.

M-3.4.4 *Determination*

M-3.4.4.1 *Preparation of the test solution*

Pour 30 ml of the sulphuric acid solution (approximately 0.1 N) into the conical flask (*D*). Transfer the test portion (*see* **M-3.4.1**) to the distillation flask (*B*), washing the beaker thoroughly with water and collecting the washings in the distillation flask. The final volume of the solution should be about 100 ml. Add several drops of the phenolphthalein solution and connect the flask to the distillation apparatus. Neutralize the test solution, stirring continuously, with the sodium hydroxide solution (*see* **M-3.2.1**) added through the dropping funnel. Add 50 ml in excess of the sodium hydroxide solution and the quantity of water necessary to attain a volume of about 300 ml. During these additions, ensure that several drops of solution remain above the stopcock as a seal. Close the funnel with its bung. Warm the flask to the commencement of boiling and distil in a current of steam, controlled to give a drop-by-drop rate of delivery into the conical flask, until a volume of about 150 ml has collected. Thoroughly wash the end of the condenser with water, collecting the washings in the conical flask.

Adjust the *p*H of the solution to between 6 and 7, first by means of the sodium hydroxide solution (*see* **M-3.2.3**) and then by means of the sodium hydroxide solution (*see* **M-3.2.4**) checking the *p*H value with the *p*H meter. Quantitatively transfer the solution to a 250 ml one-mark volumetric flask, dilute to the mark and mix.

M-3.4.4.2 *Colour development*

Take 25.0 ml of the solution (*see* **M-3.4.4.1**) and transfer to a 50 ml one-mark volumetric flask. Stirring throughout each addition, introduce 0.3 ml of the acetone and by means of rapid delivery pipettes, 10 ml of the sodium phenate solution and, immediately afterwards, 5 ml of the sodium hypochlorite solution.

Dilute to the mark, mix and allow to stand, shielded from light, at ambient temperature, for 60 ± 5 min.

M-3.4.4.3 *Spectrophotometric measurements*

Carry out the spectrophotometric measurements on the coloured solution (*see* **M-3.4.4.2**) and on a corresponding aliquot portion of the blank test solution, according to the procedure described in **M-3.4.3.2** after having adjusted the instrument to zero absorbance against water.

M-3.5 Calculation

By means of the calibration chart (*see* **M-3.4.3.3**), determine the quantities of ammoniacal nitrogen

corresponding the values of the spectrophotometric measurements.

$$\text{Ammoniacal nitrogen content, percent by mass} = \frac{(m_1 - m_2) \times D}{1\,000 \times m_0}$$

where

m_0 = mass, in g, of the test portion;

m_1 = mass, in micrograms (µg), of ammoniacal nitrogen found in the aliquot portion of the test solution taken for the colour development;

m_2 = mass, in micrograms (µg), of ammoniacal nitrogen found in the corresponding aliquot portion of the blank test solution; and

D = ratio between the volume of the test solution (*see* **M-3.4.4.1**) and the aliquot portion taken for the colour development.

ANNEX N

(Clause 6)

SAMPLING OF NITRIC ACID

N-1 GENERAL REQUIREMENTS OF SAMPLING

N-1.0 In drawing samples the following precautions and directions shall be observed.

N-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.

N-1.2 To draw a representative sample, the contents of each container shall be mixed thoroughly by rolling, shaking or stirring by suitable means.

N-1.3 The sample shall be placed in suitable clean, dry and airtight glass containers.

N-1.4 Each sample container shall be sealed airtight after filling and shall be marked with full details of sampling, the date of sampling and the year of manufacture of the material.

N-1.5 Sample shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

N-2 SCALE OF SAMPLING

N-2.1 Lot

All the containers in the single consignment of the material 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.

N-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements of the standard. The number of containers to be selected from lots of different sizes shall be in accordance with Table 2.

N-2.3 The containers shall be selected at random from a lot. In order to ensure randomness of selection random number tables shall be used (*see* IS 4905). In case random number tables are not available, the following procedure may be followed:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, 3.....*r*, where *r* is the integral part of *N*/*n* (*N* and *n* being the lot size and the sample size respectively). Every *r*th container thus counted shall be included in the sample till the required number of containers specified in Table 2 is taken out.

Table 2 Number of Containers to be Selected for Sampling

(Clause N-2.2)

Sl No.	Lot Size *N*	No. of Containers to be Selected *n*
(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 1 000	9
ix)	1 001 and above	10

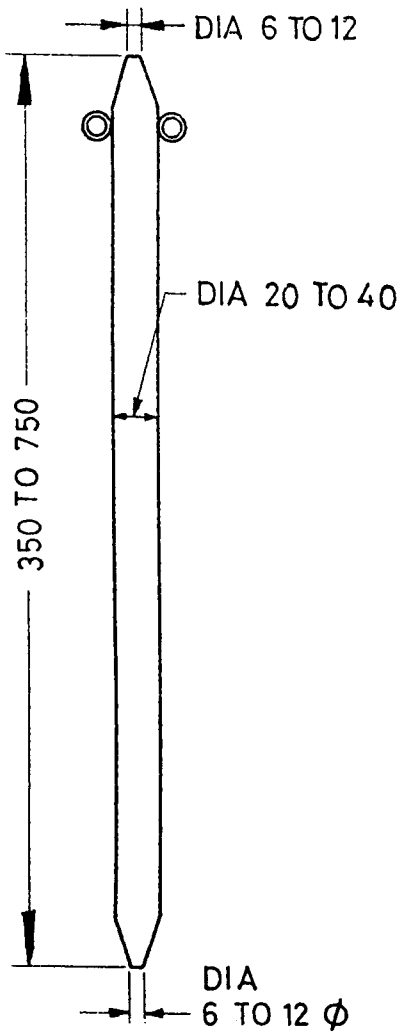
N-3 PREPARATION OF TEST SAMPLES

N-3.1 Sampling Tube

It shall be made of glass and shall be 20 to 40 mm in diameter and 350 to 750 mm in length (*see* Fig. 5). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is

facilitated by two rings at the upper end. For drawing sample, the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

N-3.1.1 For small containers, the size of the sampling tube may be altered suitably.



All dimensions in millimetres.

FIG. 5 SAMPLING TUBE

N-3.2 From each of the containers selected according to **N-2.3**, a small representative portion of the material, about 200 ml, shall be taken out with the help of the sampling tube after thoroughly stirring the acid.

N-3.3 Out of these portions, small but equal quantities of the material shall be taken out and thoroughly mixed to form a composite sample not less than 600 ml. The composite sample shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

N-3.4 The remaining portion of the material from each container shall be divided into 3 equal parts, each forming an individual sample. One set of individual samples representing the *n* containers sampled shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

N-3.5 All the individual and composite samples shall be transferred to separate bottles. These bottles shall be sealed and labelled with full identification particulars.

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

N-4 NUMBER OF TESTS

N-4.1 Total acidity shall be tested on each of individual samples (*see* **N-3.4**).

N-4.2 Tests for all other characteristics listed in Table 1 shall be performed on the composite sample (*see* **N-3.3**).

N-5 CRITERIA FOR CONFORMITY

N-5.1 For Individual Samples

From the individual test results for total acidity, the mean $\bar{x}$ and range R shall be calculated (range being defined as the difference between the maximum and the minimum of the test results). The lot shall be declared as satisfying the requirements of total acidity if the value of the expression $\bar{x} - 0.6\,R$ is greater than or equal to the relevant value specified in Table 1.

N-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirements specified in Table 1.

N-5.3 A lot shall be declared as conforming to this standard, if it satisfies the relevant requirements according to **N-5.1** and **N-5.2** are satisfied.

ANNEX P

(*Foreword*)

CORRELATION TABLE FOR RELATIVE DENSITY AND PERCENT BY MASS OF NITRIC ACID

Relative Density at 28°C/4°C	*Percent by Mass*
(1)	(2)
1.000	0.7
1.005	1.7
1.010	2.6
1.015	3.5
1.020	4.4
1.025	5.4
1.030	6.3
1.035	7.2
1.040	8.1
1.045	8.9
1.050	9.8
1.055	10.7
1.060	11.5
1.065	12.4
1.070	13.3
1.075	14.1
1.080	14.9
1.085	15.8
1.090	16.6
1.095	17.4
1.100	18.3
1.105	19.1
1.110	19.9
1.115	20.7
1.120	21.5
1.125	22.3
1.130	23.1
1.135	23.9
1.140	24.7
1.145	25.5
1.150	26.3
1.155	27.1
1.160	27.8
1.165	28.6
1.170	29.4
1.175	30.1
1.180	30.9
1.185	31.7
1.190	32.4
1.195	33.2

Relative Density at 28°C/4°C	*Percent by Mass*
(1)	(2)
1.200	33.9
1.205	34.7
1.210	35.4
1.215	36.2
1.220	37.0
1.225	37.8
1.230	38.6
1.235	39.4
1.240	40.2
1.245	41.0
1.250	41.8
1.255	42.6
1.260	43.4
1.265	44.2
1.270	45.0
1.275	45.8
1.280	46.6
1.285	47.4
1.290	48.2
1.295	48.9
1.300	49.8
1.305	50.7
1.310	51.6
1.315	52.4
1.320	53.3
1.325	54.2
1.330	55.1
1.335	56.0
1.340	57.0
1.345	57.9
1.350	58.8
1.355	59.8
1.360	60.8
1.365	61.8
1.370	62.9
1.375	63.9
1.380	65.1
1.385	66.2
1.390	67.2
1.395	68.5

Relative Density at 28°C/4°C	*Percent by Mass*
(1)	(2)
1.400	69.7
1.405	70.9
1.410	72.1
1.415	73.4
1.420	74.7
1.425	76.0
1.430	77.4
1.435	78.9
1.440	80.3
1.445	81.8
1.450	83.3
1.455	84.9
1.460	86.6
1.465	88.4
1.470	90.4
1.475	92.7
1.480	95.3
1.485	97.5
1.490	98.7
1.495	99.5
1.498	99.9

ANNEX Q

(Foreword)

COMMITTEE COMPOSITION

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Amendments Issued Since Publication

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