IS : 1089 - 1986
(Reaffirmed 2003)

Indian Standard

(Reaffirmed 2015)

SPECIFICATION FOR OLEUM, TECHNICAL

(Second Revision)

UDC 661·251

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

Gr 7

December 1986

Indian Standard

SPECIFICATION FOR OLEUM, TECHNICAL

(Second Revision)

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Indian Standard

SPECIFICATION FOR OLEUM, TECHNICAL

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

0.2 Oleum, technical (commercially known as fuming sulphuric acid) is an important industrial chemical used in the dyestuff industry, the pharmaceutical industry, for the production of DDT, and in the manufacture of explosives and nonsoapy detergents.

0.3 This standard was originally published in 1957 and was revised in 1973 incorporating two grades, deleting the requirement for relative density and in its place, a statement correlating relative density of oleum and its free sulphur trioxide content had been given (*see* Appendix B).

0.4 The standard is being revised again and in this revision, a new requirement of arsenic and its method of test have been incorporated for both the grades.

0.5 The International Organization for Standardization (ISO) has published the following standards/recommendation for oleum relevant to this standard:

ISO 910-1977 Sulphuric acid and oleum for industrial use — Determination of total acidity and calculation of free SO_3 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.

The provisions of this standard substantially agree with these ISO standards/recommendations.

0.6 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*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for oleum, technical.

2. GRADES

2.1 There shall be two grades of oleum, namely, Grade 1 and Grade 2.

3. REQUIREMENTS

3.1 Description — Oleum may be off-white to brown in colour.

3.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. References to the relevant clauses of the appendix are given in col 5 of Table 1.

3.3 Additional Requirements for Explosives Industry — For oleum of either grade required for manufacture of explosives, the residue on ignition and iron (as Fe) shall be not more than 0·06 percent and 0·01 percent, respectively, when tested according to the methods given in **A-3** and **A-4**. Further, the lead content (as Pb) of oleum of either grade shall be not more than 0·01 percent by mass, when tested as prescribed in **A-8**.

3.4 Relative Density — There shall be no specific requirement for relative density of oleum of either grade. However, since relative density measurement is generally used as a quick indication of the strength of oleum both in plant control as well as by purchasers, a correlation between relative density and oleum strength is given for information and guidance in Appendix B.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR OLEUM, TECHNICAL

(*Clause* 3.2)

Sl No.	Characteristic	Requirement for		Method of Test (Ref to Cl No. in Appendix A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Free sulphur trioxide (as SO_3), percent by mass, *Min*	20	65	A-2
ii)	Residue on ignition, percent by mass, *Max*	0·20	0·20	A-3
iii)	Iron (as Fe), percent by mass, *Max*	0·05	0·05	A-4
iv)	Chlorides (as Cl), percent by mass, *Max*	0·002	0·002	A-5
v)	Total nitrogen (as HNO_3), percent by mass, *Max*	0·005	0·005	A-6
vi)	Sulphur dioxide (as SO_2), percent by mass, *Max*	0·2	0·2	A-7
vii)	Arsenic (as As), percent by mass, *Max*	0·000 5	0·000 5	A-9

4. PACKING AND MARKING

4.1 Packing

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

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

4.1.3 The bottles and 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 dry earth, and the empty surrounding space shall be packed in suitable iron hampers or wooden crates stuffed with chemically inert materials like clay or fullers earth.

4.2 Marking

4.2.1 The containers and also the packages shall be suitably marked in red letters not less than 2·5 cm high, and drums and tank wagons shall be marked in red letters not less than 5 cm high, showing the name of the acid, manufacturer's name, the grade and mass of the material, recognized trade-mark, if any, and the month and year of manufacture.

4.2.2 The packages shall be labelled with the symbol shown in Fig. 1. [*see* IS : 1260 (Part 1)-1973*], the words 'CORROSIVE, HANDLE WITH CARE' being prominently displayed in the lower half of the symbol.

4.2.3 All the containers and packages should be suitably marked with an arrow showing the upright position of containers.

4.2.4 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative test samples of the material shall be drawn as prescribed in Appendix C.

*Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (*first revision*).

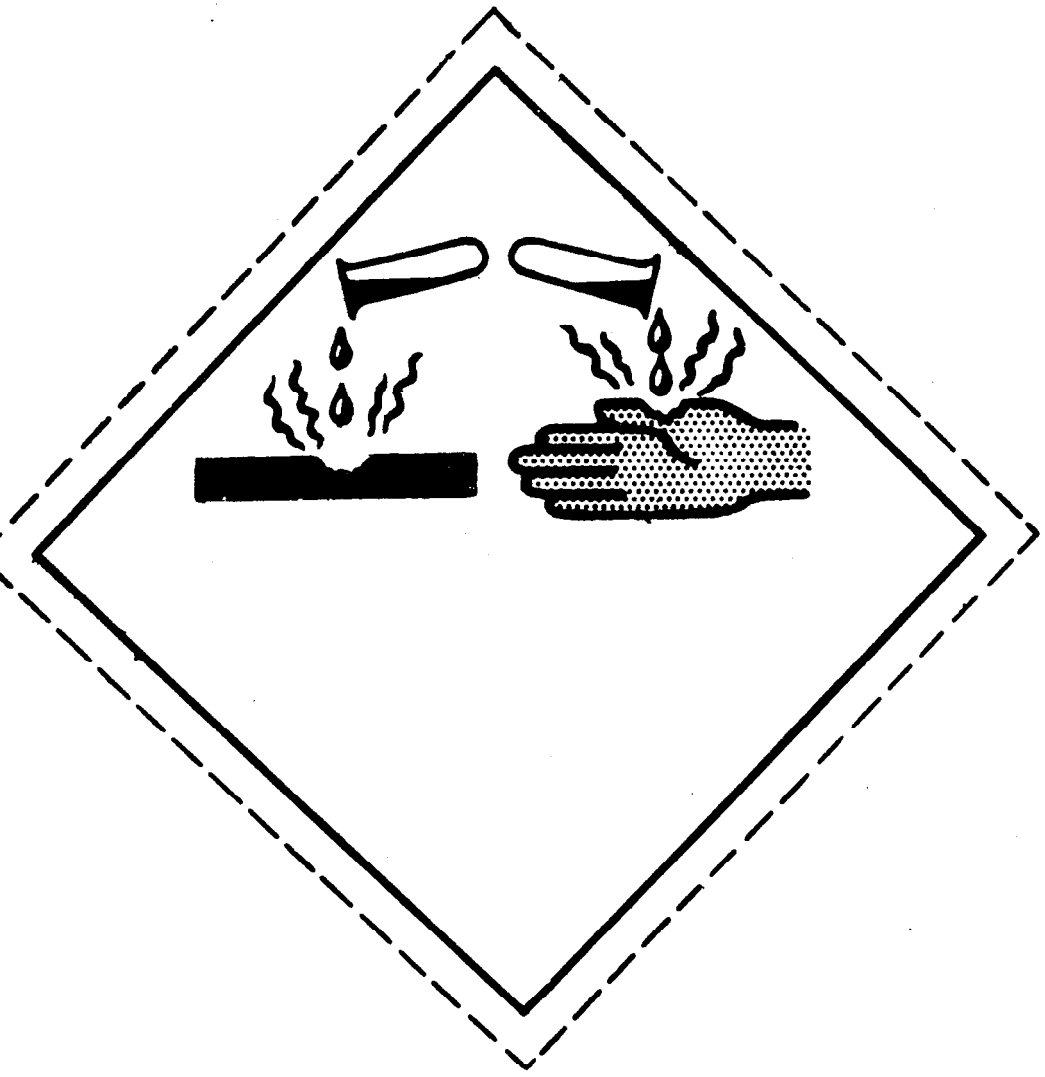


FIG. 1 SYMBOL SHOWING DANGER OF CORROSION

APPENDIX A

(*Clause* 3.2)

METHODS OF TEST FOR OLEUM, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in the tests.

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

A-2. DETERMINATION OF FREE SULPHUR TRIOXIDE

A-2.1 Apparatus

A-2.1.1 *Spherical Glass Ampoule* — of suitable shape and capacity, for example, 20 mm in diameter, having one capillary end of about 50 mm length (*see* Fig. 2).

A-2.1.2 *Dely Weighing Tube* — As shown in Fig. 3.

A-2.2 Reagents

A-2.2.1 *Hydrogen Peroxide* — 60 g/l, neutral to methyl red.

A-2.2.2 *Standard Sodium Hydroxide Solution* — Approximately 1 N.

A-2.2.3 *Methyl Red Indicator* — Dissolve 0·1 g of methyl red in 95 percent ethanol (*see* IS : 323-1959†) and dilute to 100 ml with the same ethanol.

A-2.3 Procedure

A-2.3.1 *Test Portion* — Carefully mix the sample by shaking the container. If the acid is partially crystallized, slightly heat the container until the sample is liquefied, then carefully mix again. Nearly fill a groundglass stoppered flask (capacity approximately 500 ml and neck of about 30 mm diameter) with the test sample. Slightly heat in a flame the bulb of the spherical glass 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

*Specification for water for general laboratory use (*second revision*).
†Specification for rectified spirit (*revised*).

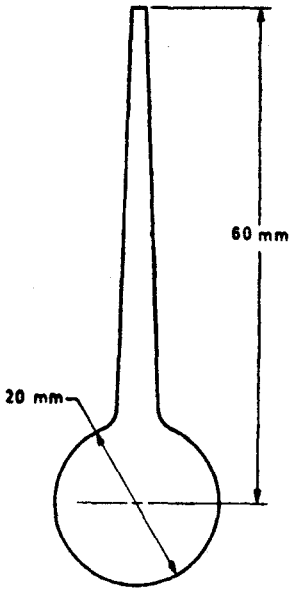
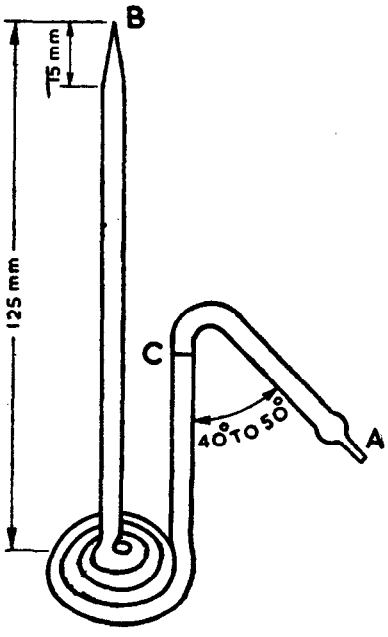


FIG. 2 SPHERICAL GLASS AMPOULE



A — End of the tube.
B — Tube of 5 mm bore and the capillary drawn to 0·25 mm bose at *B*.
C — Mark on the tube.

FIG. 3 DELY WEIGHING TUBE

ampoule and carefully wipe the capillary and with filter paper. Seal the capillary end in the 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. Alternatively, the test sample may be collected with the help of the Dely weighing tube. For this purpose, weigh the empty tube. Draw the sample into it by applying suction through a rubber tube attached to *A*. A mark ascertained by a previous run shall be made to indicate the point to which the acid is to be drawn to give the required quantity (3 to 4 g) of oleum. Wipe the tip *B* carefully with tissue paper and weigh the tube with the sample. The mass of the sample taken shall be obtained by difference.

A-2.3.2 *Preparation of Sample Solution* — Carefully place the ampoule containing the test portion into a thick-walled conical flask (500 ml capacity, with ground-glass stopper) containing 300 ml of cold water.

Stopper the flask and shake to break the ampoule containing the test portion. Cool 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. Transfer quantitatively the solution to a 500-ml one-mark volumetric flask, dilute to the mark and mix thoroughly. Use this solution for test in **A-2.3.3** and **A-5.3**.

A-2.3.3 *Determination* — Transfer 100·0 ml of the sample solution, prepared in **A-2.3.2** to a 500-ml conical flask. Add 5 ml of hydrogen peroxide solution and gently boil for 10 minutes. Allow to cool, add two drops of methyl red solution and titrate to the end point by means of the standard sodium hydroxide solution.

A-2.4 Calculation — For calculating the free sulphur trioxide content of oleum, it is necessary to evaluate: (a) the total sulphur trioxide content (*see* **A-2.4.1**), (b) water combined as sulphuric acid (*see* **A-2.4.2**), and (c) sulphuric acid content (*see* **A-2.4.3**).

A-2.4.1 *Total Sulphur Trioxide*

$$\text{Total sulphur trioxide (as } SO_3\text{), percent by mass} = 20{\cdot}02 \times \frac{VN}{M}$$

where

V = volume in ml of standard sodium hydroxide solution used,

N = normality of the alkali, and

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

A-2.4.2 *Evaluation of Water Combined as Sulphuric Acid*

$$\text{Water combined as sulphuric acid, percent by mass } (C) = 100 - B$$

where

B = total sulphur trioxide content, percent by mass (**A-2.4.1**).

A-2.4.3 *Evaluation of Sulphuric Acid Content*

$$\text{Sulphuric acid content, percent by mass } (D) = C \times 5{\cdot}444$$

where

C = water combined as sulphuric acid (**A-2.4.2**).

NOTE — The conversion factor for water to sulphuric acid is 5·444.

A-2.4.4 *Free Sulphur Trioxide Content of Oleum*

Free sulphur trioxide percent by mass = $100 - D$

where

D = sulphuric acid content, as percent by mass (**A-2.4.3**).

A-3. DETERMINATION OF RESIDUE ON IGNITION

A-3.1 Procedure — Weigh to the nearest 10 mg about 50 g of the test sample in a flat-bottom platinum or quartz dish previously ignited at 800 ± 50°C cooled in a desiccator and weighed. If the sample shows any suspended impurities or any sedimentation at the bottom of the container, the contents of the container shall be thoroughly swirled to offer a uniform and homogeneous material before transferring the same to the dish. Heat the dish gradually on a sandbath until the acid is practically driven off. Then transfer to a muffle furnace maintained, at about 800 ± 50°C and heat for 15 minutes or until there is no further fuming of sulphur trioxide. Cool the dish in a desiccator and weigh. Reserve the residue for determination of iron (*see* **A-4**) and lead (*see* **A-8**).

A-3.2 Calculation

$$\text{Residue on ignition, percent by mass} = \frac{B - A}{M} \times 100$$

where

B = mass in g of the dish with the residue left after ignition,

A = mass in g of the empty dish, and

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

A-4. DETERMINATION OF IRON

A-4.0 General — Two methods are prescribed. Method A is the referee method and method B is the alternative method.

A-4.1 Method A

A-4.1.1 *Apparatus*

A-4.1.1.1 *Nessler cylinder* — 50 ml capacity.

A-4.1.2 *Reagents*

A-4.1.2.1 *Ammonium persulphate*

A-4.1.2.2 *Concentrated hydrochloric acid* — *See* IS : 265-1976*.

*Specification for hydrochloric acid (*second revision*).

A-4.1.2.3 *Butanolic potassium thiocyanate solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-4.1.2.4 *Standard iron solution A* — Weigh, to the nearest 1 mg, 7·022 g of iron (II) ammonium sulphate hexahydrate and place in a beaker of suitable capacity. Add 50 ml of sulphuric acid (about 2 N) and transfer quantitatively to a 1 000-ml one-mark volumetric flask. Dilute to the mark and mix thoroughly. One millilitre of this solution contains 1 mg of Fe.

A-4.1.2.5 *Standard iron solution B* — Transfer 50·0 ml of the standard iron solution A to a 1 000-ml one-mark volumetric flask. Add 5 ml of sulphuric acid (about 2 N), dilute to the mark and mix thoroughly. One millilitre of this solution contains 0·05 mg of Fe. The solution should be prepared just before use.

A-4.1.3 *Procedure*

A-4.1.3.1 Treat the residue left on ignition in **A-3.1** with 2 ml of concentrated hydrochloric acid, add 25 ml of water and heat to facilitate dissolution. Transfer quantitatively to a 500 ml flask, filtering it if necessary, dilute to the mark and mix. Use this solution for determination of iron and lead (*see* A-8).

A-4.1.3.2 Pipette out 10 ml of the solution into a Nessler cylinder. Add about 30 mg of ammonium persulphate, 1 ml of concentrated hydrochloric acid and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml, shake vigorously for 30 seconds and allow the layers to separate. Carry out a control in another Nessler cylinder in the same manner, using 10 ml of standard iron solution B (or 2 ml in the case of explosives grade oleum) and compare the intensity of colour produced in the butanol layers in the two tubes.

A-4.1.3.3 The limit prescribed shall be taken as not having been exceeded, if the intensity of the red colour produced in the butanolic layer with the oleum sample is not deeper than that produced in the control test.

A-4.2 Method B

A-4.2.1 *Apparatus*

A-4.2.1.1 *Spectrophotometer or photoelectric colorimeter*

A-4.2.2 *Reagents*

A-4.2.2.1 *Hydrochloric acid* — approximately 1 N.

A-4.2.2.2 *Hydroxylammonium chloride solution* — Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml.

A-4.2.2.3 *Ammonium acetate solution* — Dissolve 30 g of ammonium acetate in water and dilute to 100 ml.

A-4.2.2.4 *Bipyridyl solution* — Dissolve 1 g of 2, 2'-bipyridyl in 10 ml of approximately 1 N hydrochloric acid and dilute to 100 ml.

A-4.2.2.5 *Standard iron solution C* — Transfer 200 ml of standard iron solution B (A-4.1.2.5) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix thoroughly. One millilitre of this solution contains 10 μg of Fe. The solution should be prepared just before use.

A-4.2.3 *Procedure*

A-4.2.3.1 *Preparation of calibration curve* — Into each of a series of eleven 100-ml one-mark volumetric flasks, place the quantities of standard iron solution C as indicated below:

Standard Iron Solution C	*Corresponding to Fe*
ml	μg
0 (Compensation solution)	0
5·0	50
10·0	100
15·0	150
20·0	200
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 and 1 ml of the 2, 2'-bipyridyl solution. Dilute to the mark, mix thoroughly and wait for 10 minutes. Carry out the measurement on the spectrophotometer in 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 and using as reference the compensation solution. Prepare a calibration chart with the iron content in micrograms per 100 ml of the standard matching solution as abscissae and the corresponding values of optical density as ordinate.

A-4.2.3.2 Transfer an aliquot of the solution, prepared in A-4.1.3.1 containing between 50 and 500 micrograms of iron, to a 100-ml one-mark

volumetric flask. Dilute to approximately 50 ml, add successively 2 ml of hydrochloric acid, 2 ml of the 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-4.2.3.1,** adjusting the instrument to zero optical density using as reference the blank test solution (*see* **A-4.2.3.3**).

A-4.2.3.3 *Blank test* — Simultaneously carry out a blank test using the same procedure and quantities of all reagents employed in the test.

A-4.2.4 *Calculation* — From the calibration chart (*see* **A-4.2.3.1**), determine the iron content corresponding to the photometric measurement.

$$\text{Iron (as Fe), percent by mass} = \frac{A \times 500 \times 100}{V \times M}$$

where

A = mass in g of iron present 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 in **A-3.1**.

A-5. DETERMINATION OF CHLORIDES

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinder* — two, capacity 100 ml.

A-5.2 Reagents

A-5.2.1 *Dilute Nitric Acid* — approximately 10 percent (*m/v*).

A-5.2.2 *Silver Nitrate Solution* — 4·2 percent (*m/v*).

A-5.2.3 *Standard Sodium Chloride Solution* — Dissolve 0·016 5 g of pure ignited sodium chloride in 1 000 ml of water.

A-5.3 Procedure

A-5.3.1 Take with the help of a burette an aliquot of the sample solution prepared in **A-2.3.2** so as to represent 0·5 g of the sample and transfer it to Nessler cylinder. Add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Make it up to the mark and shake well to get a homogeneous solution.

A-5.3.2 In another Nessler cylinder, dilute 1 ml of standard sodium chloride solution with 80 to 90 ml of water and add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution and make the volume to 100 ml. Compare the opalescence developed in the standard solution and the solution containing the sample of oleum.

A-5.3.3 The limit prescribed 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 standard solution.

A-6. TEST FOR TOTAL NITROGEN

A-6.1 Reagents

A-6.1.1 *Sodium Hydroxide Solution* — approximately 40 percent.

A-6.1.2 *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. Then plunge zinc into the molten mass. Cover the crucible and heat the mixture for a few minutes and stir with an iron rod. Allow it to cool slowly with the cover on and then pulverize the crystallized mass to particle size of 0·2 to 0·3 mm.

A-6.1.3 *Concentrated Hydrochloric Acid — See* IS : 265-1976*.

A-6.1.4 *Nessler Solution* — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free 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 the mercuric chloride solution and dilute to 200 ml with ammonia-free water.

A-6.1.5 *Standard Ammonium Chloride Solution* — Prepare a solution containing 0·084 9 g of ammonium chloride per litre. One millilitre of this solution contains 0·1 mg of total nitrogen (as HNO_3).

A-6.2 Procedure

A-6.2.1 Add cautiously 10 ml of the material to 100 ml of water in a 500-ml volumetric flask and, 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 one drop of hydrochloric acid. Transfer the distillate to a Nessler cylinder, add 1 ml of sodium hydroxide solution and 2 ml of Nessler solution.

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*Specification for hydrochloric acid (*second revision*).

A-6.2.2 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the colour of the solution is not deeper than that obtained by nesslerizing 9·5 ml of a standard solution of ammonium chloride in the same total volume of the solution.

A-7. DETERMINATION OF SULPHUR DIOXIDE

A-7.0 Methods — Two methods are prescribed. Method A is for routine analysis and method B is the referee method.

A-7.1 Method A

A-7.1.1 *Reagents*

A-7.1.1.1 *Potassium iodide solution* — approximately 10 percent (*m/v*).

A-7.1.1.2 *Standard iodine solution* — 0·05 N.

A-7.1.1.3 *Standard sodium thiosulphate solution* —0·05 N.

A-7.1.1.4 *Starch solution* — 0·5 percent *m/v*, freshly prepared.

A-7.1.1.5 *Concentrated sulphuric acid — see* IS : 266-1977*.

A-7.1.2 *Procedure* — Weigh accurately about 5 g of the test sample as prescribed in A-2.3.1 and cautiously dilute in 200 ml of water contained in a 500-ml conical flask placed in a deep porcelain dish containing cold water. Add to the diluted solution 10 ml of potassium iodide solution and 20 ml of standard iodine solution. In case the iodine colour disappears altogether, another 20 ml of iodine solution may be added. Titrate the excess iodine with standard sodium thiosulphate using starch as indicator towards the end.

A-7.1.2.1 Carry out a blank test using the same reagents, taking 5 ml of concentrated sulphuric acid under similar conditions.

A-7.1.3 *Calculation*

$$\text{Sulphur dioxide (as } SO_2\text{), percent by mass} = \frac{(B - A)\,N \times 3{\cdot}2}{M}$$

where

B = volume in ml standard sodium thiosulphate solution used in blank test,

A = volume in ml of standard sodium thiosulphate solution used with the test sample,

N = normality of sodium thiosulphate solution, and

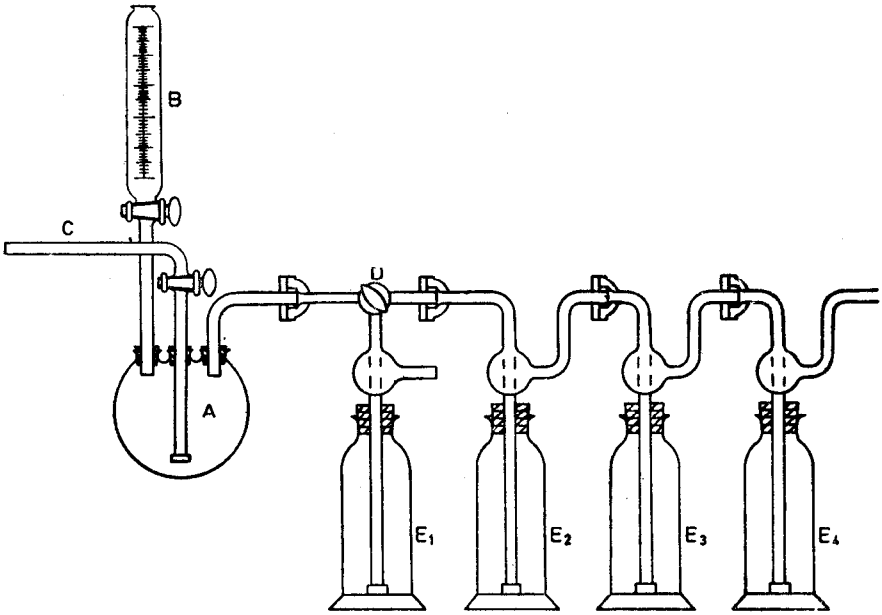
M = mass in g of the test sample taken for the test.

NOTE — In case A is equal to B or higher than B, then there will be no sulphur dioxide in the sample of oleum.

*Specification for sulphuric acid (*second revision*).

A-7.2 Method B

A-7.2.1 *Apparatus*—As shown in Fig. 4.



A — 500 ml, three-necked, round-bottomed flask.

B — 200 ml, graduated separating funnel fitting into one of the three necks of the flask.

C — Tube with cock, inserted into the central neck of the flask, extending about 1 cm from the bottom of the flask. The end of the tube is fitted with a sintered glass diaphragm.

D — Three-way cock.

E — Four wash bottles (E_1, E_2, E_3, E_4), capacity 100 ml, fitted with a sintered glass diaphragm (porosity 3 to 15 μm) at the end of the immersion tube, for gas distribution. One branch of the cock (*D*) is connected to the third neck of the round-bottomed flask (*A*); the second branch is connected to the first wash-bottle (E_1), which can be connected and disconnected according to requirement through the cock (*D*); the third branch is connected to the other wash-bottles linked in series.

FIG. 4 APPARATUS FOR DETERMINATION OF SULPHUR DIOXIDE

A-7.2.2 *Reagents*

A-7.2.2.1 *Nitrogen* — Pure. *See* IS : 1747-1972*.

A-7.2.2.2 *Hydrogen peroxide* — 30 g/l, free from sulphate ions.

*Specification for nitrogen (*first revision*).

A-7.2.2.3 *Hydrochloric acid* — 1·2 N, free from sulphate ions.

A-7.2.2.4 *Barium chloride* — dihydrate, 100 g/l solution.

A-7.2.2.5 *Silver nitrate solution* — Dissolve 0·5 g of silver nitrate in water and dilute to 100 ml.

A-7.2.2.6 *Potassium permanganate solution* — approximately 0·1 N.

A-7.2.2.7 *Sodium hydroxide solution* — approximately 0·1 N.

A-7.2.2.8 *Sulphuric acid* — approximately 36 N.

A-7.2.3 *Procedure*

A-7.2.3.1 *Assembly of apparatus* — Transfer to wash-bottles E_2 and E_3 10 ml of hydrogen peroxide and 50 ml of sodium hydroxide solution. Pour into wash-bottles E_1 and E_4 50 ml of water and one drop of potassium permanganate solution. Remove the air from the round-bottomed flask A by means of a rapid current of nitrogen for 15 minutes. Connect wash-bottles E_2, E_3 and E_4 to the apparatus, excluding the bottle E_1.

A-7.2.3.2 *Test portion* — Fill a ground glass stoppered flask, capacity 250 ml, with the test sample and take a test portion of 100 to 200 g according to the presumed sulphur dioxide content, weighing by difference to the nearest 10 mg. Slowly pour the test portion, cooling to ensure that the temperature is kept below 40°C, on to such a quantity of ice as to produce a final density of approximately 1·70 g/ml.

A-7.2.3.3 *Displacement and absorption of sulphur dioxide* — Introduce the test portion into flask *A* through separating funnel *B*. Pass a current of nitrogen, measured by a flowmeter, of about 20 litres per hour, for 3 hours; the pink colour of the liquid contained in wash-bottle E_4 should remain unchanged. At this point pass the current of nitrogen to wash-bottle E_1, excluding bottles E_2, E_3, and E_4 by means of cock *D*. After half-an-hour, the colour of the liquid in bottle E_1 should still be pink. If this is not the case, repeat the test prolonging the time of operation beyond 3 hours and carefully heating flask *A* at about 50°C. Disconnect wash-bottles E_2 and E_3, rinse the immersion tubes with water and quantitatively transfer the contents of these wash-bottles to a beaker of suitable capacity (600 ml, for example).

A-7.2.3.4 *Determination* — Add drop by drop to the solution obtained in A-7.2.3.3 hydrochloric acid until the contents of the beaker show an acid reaction to the litmus paper. Boil to destroy hydrogen peroxide. Add drop by drop 20 ml of barium chloride solution. (The addition should take about 3 minutes.) Heat to the boil and keep boiling for 2 minutes. Place on a boiling water-bath and leave for 2 hours, then stop heating and leave to stand for about 16 hours. Filter through an ashless slow-speed filter paper (pore diameter between 0·4 and 1 μm approximately), and wash

with boiling water until the filtrate is free from chlorides; the liquid flowing from the funnel should remain clear on addition of a few drops of silver nitrate solution. Place the filter paper and its contents in a platinum crucible, previously tared after igniting in a furnace at 600 to 800°C and cooling in a desiccator. Dry in an oven at about 110°C, and carefully char the filter paper at a low temperature to avoid flaming, then ignite in a furnace at 600 to 800°C. Remove the crucible, add a drop of sulphuric acid and again ignite. Remove the crucible, place in a desiccator, allow to cool and weigh.

A-7.2.4 *Calculation*

$$\text{Sulphur dioxide content } (SO_2), \text{ percent by mass} = \frac{A \times 0{\cdot}274\,4 \times 100}{M}$$

where

A = mass in g of barium sulphate precipitate, and

M = mass in g of the test portion.

NOTE — The conversion factor from barium sulphate to SO_2 is 0·274 4.

A-8. DETERMINATION OF LEAD

A-8.1 Apparatus — All glassware, including the reagent bottles, shall be of borosilicate glass or other quality free from lead, or alternatively plastics materials may be used. It shall be washed with nitric acid solution approximately 7 N, and rinsed with water three times.

A-8.1.1 *Spectrophotometer or Photoelectric Colorimeter* — Fitted with filters giving maximum transmission between 500 and 540 nm.

A-8.2 Reagents

A-8.2.1 *Hydroxylammonium Chloride Solution* — 10 percent (*m*/*v*).

A-8.2.2 *Ammonium Citrate Solution* — 10 percent (*m*/*v*).

A-8.2.3 *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-8.2.4 *Potassium Cyanide Solution* — 5 percent (*m*/*v*).

A-8.2.5 *Chloroform* — redistilled in a borosilicate glass apparatus with ground joints.

A-8.2.6 *Dithizone Solution* — 0·025 g/l, in chloroform.

A-8.2.6.1 *Purification of the dithizone* — Dissolve 1 g of dithizone (biphenyl thiocarbazone) 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, colouring it orange while the oxidation products remain in the organic phase which assumes a more or less intense reddish-yellow colouration). Discard the organic phase, place the orange coloured aqueous extracts together, filter them and transfer them to a 1 000 ml beaker. Precipitate the dithizone by slight acidification with the aid of a saturated solution of sulphur dioxide. Allow the precipitate to settle, filter through a sintered glass crucible and wash until there is no longer an acid reaction. Dry the extracted product thoroughly by means of concentrated sulphuric acid solution under vacuum and in darkness for 3 to 4 days. Grind the solid dry product quickly for a short time and transfer immediately to a small dark glass bottle. The dithizone, thus purified and stored away from direct sunlight, can be kept for at least 6 months.

A-8.2.6.2 *Preparation of the solution* — Immediately before use, weigh to the nearest milligram 25 mg of purified dithizone, transfer to a 1 000-ml one-mark volumetric flask, dissolve in chloroform, dilute to the mark with the same chloroform and mix. Store the solution in a dry, dark-glass bottle, hermetically sealed.

A-8.2.7 *Ammonical Potassium Cyanide Solution* — Transfer 20 ml of the potassium cyanide solution to a 1 000-ml one-mark volumetric flask. Dilute with water, add 10 ml of ammonia solution (density 0·88 g/ml), dilute to the mark and mix.

A-8.2.8 *Standard Lead Solution* — Weigh to the nearest milligram 1·600 g of lead nitrate [$Pb(NO_3)_2$] and transfer to a beaker of suitable capacity. Dissolve in a little water and 1 ml of concentrated nitric acid. Transfer the solution quantitatively to a 1 000-ml one-mark volumetric flask, dilute to the mark and mix. One millilitre of this solution contains 1 mg of lead (Pb). Transfer 10 ml of the solution to a 1 000-ml one-mark volumetric flask, add 1 ml of concentrated nitric acid, dilute to the mark and mix. One millilitre of this solution contains 10 μg of lead (Pb). Prepare this solution immediately before use.

A-8.3 Procedure

A-8.3.1 Transfer an aliquot of the solution prepared in **A-4.1.3.1** containing between 10 and 100 μg of lead to a separating funnel of suitable capacity. Add 1 ml of hydroxylammonium chloride solution and 10 ml of ammonium citrate solution and adjust the *p*H to between 8·5 and 10 by adding ammonia solution drop by drop, checking with an 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 5 minutes, preferably mechanically. Allow to separate and draw off the organic phase, collecting it in a 50-ml one-mark volumetric flask. Continue the extraction with successive portions of 5 ml of the 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 one-mark 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 the ammoniacal cyanide solution. Then draw off the organic phase, which will have a clear pink colour, and pass it through a dry and acid-washed filter paper, collecting the filtrate in a dry vessel.

NOTE — Dithizonates are particularly sensitive ultra-violet light and should, therefore, be protected from sunlight and fluorescent light.

A-8.3.2 Carry out the photometric measurement of the choloform solution of lead dithizonate using the spectrophotometer at the maximum of the absorption curve (at a wavelength of about 520 nm) or with the aid of a photoelectric colorimeter fitted with suitable filters; in each case adjust the instrument to zero absorbance against chloroform.

A-8.3.3 *Preparation of Calibration Curve* — Into eleven separating funnels of 100 ml capacity, fitted with ground-glass stoppers, transfer 10 ml of water and add respectively the volumes of standard lead solution indicated below measured with the burette:

Volume of Lead Standard Solution	*Corresponding Mass of* Pb
ml	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

Then proceed with the determination as described in **A-8.3.1.** Carry out photometric measurement as in **A-8.3.2**, adjusting the instrument to zero

absorbance against the compensation solution. Prepare a chart having, for example, lead (Pb) content in micrograms per 50 ml of standard matching solution as abscissae and the corresponding values of absorbance as ordinates.

A-8.3.4 *Blank Test* — Carry out a blank test using the same quantities of reagents as those employed with the sample solution, and following the same procedure as given in **A-8.3.1** and **A-8.3.2.**

A-8.4 Calculation — By reference to the calibration curve, determine the quantity of lead corresponding to the value of the photometric measurement.

$$\text{Lead (as Pb), percent by mass} = 100 \times \frac{(m_1 - m_2) \times D}{M_0}$$

where

m_1 = mass in g of lead found in the aliquot portion of the sample solution,

m_2 = mass in g of lead found in a corresponding aliquot portion of the blank test solution,

D = ratio of the volume of sample solution (prepared in **A-4.1.3.1**) to that taken for determination of lead in **A-8.3.1**, and

M_0 = mass in g of the sample taken for the test in **A-4.**

A-9. DETERMINATION OF ARSENIC

A-9.1 Reagents

A-9.1.1 *Standard Arsenic Stock Solution* — Weigh, to the nearest 0·1 mg, 0·132 0 g of arsenic trioxide and transfer it to a beaker of suitable capacity (for example, 100 ml). Dissolve the arsenic trioxide with 2 ml of sodium hydroxide solution containing 50 g/l. Transfer the solution to a 1 000 ml volumetric flask. Wash the beaker several times. Collect the washings in the volumetric flask and make up the volume. One millilitre of this standard solution contains 100 μg of arsenic (As).

A-9.1.2 *Diluted Standard Arsenic Solution* — Containing 2·5 mg of arsenic per litre. Take 25 ml of the standard arsenic solution, transfer it to a 1 000-ml volumetric flask and make up the volume. Prepare this solution at the time of use. One millilitre of this standard solution contains 2·5 μg of arsenic (As).

A-9.1.3 *Silver Diethyldithiocarbamate Solution* — Dissolve 1 g of silver diethyldithiocarbamate in water-white pyridine and dilute to 200 ml with pyridine. Store the solution in stoppered-glass bottles away from light.

A-9.2 Procedure — Transfer to a series of 100 ml conical flasks, aliquots of standard arsenic solution corresponding to 0, 5, 10, 15, 20 and 25 μg of arsenic and proceed as given in **A-9.2.1**.

A-9.2.1 Add 10 ml of concentrated hydrochloric acid and dilute to 50 ± 5 ml with water. Add 2 ml of potassium iodide and stannous chloride solution respectively. Mix well and let it stand for 15 to 20 minutes. Pack lightly the top third of the connecting tube with impregnated absorbent cotton wool and assemble on the absorption train. Transfer 5·0 ml of silver diethyldithiocarbamate solution to absorption tube *C*. After 15 to 20 minutes, introduce 5 g of zinc granules into the conical flask *A* and quickly reassemble the apparatus. Allow the reaction to proceed for 45 to 60 minutes at room temperature.

A-9.2.2 *Spectrophotometric Measurements* — Disconnect the absorption tube and tilt the absorber so that the reagent solution flows back and forth between the absorber and bulb to disperse the solid contents, if any, and to mix in the solution well. Transfer the solution to a photometric cell and measure its absorbance at the wavelength of maximum absorption, 540 nm, using water as reference liquid.

NOTE — The colour of the dispersion is not very stable for long time and hence absorptiometric (nephelometric) measurement should be made within 2 hours of the development of colour. Care should also be taken to prevent the evaporation of solution as its volume is small.

In the case of fritted glass absorber raise and lower the connecting tube into the absorber several times, to allow the solution to pass through the frit back and forth effecting the dispersal of the red deposit. Let the connecting tube finally drain into the absorber.

10 μg standard shall have an absorption of the order of 0·4.

A-9.2.3 *Plotting of the Calibration Curve* — Calculate corrected absorbance by subtracting the reading obtained for the solution containing no standard arsenic solution from the observed reading. Plot a graph of corrected absorbance of solution against their arsenic contents.

A-9.2.4 *Blank Test* — Carry out a blank test, as prescribed in A-9 to **A-9.2.2** omitting the sample.

A-9.3 Calculation — Calculate the corrected absorbance by subtracting the value obtained for the blank solution from that obtained for the test solution and read from the calibration curve the corresponding mass of arsenic.

$$\text{Arsenic content, parts per million by mass} = \frac{M_1}{M_2}$$

where

M_1 = mass in g of arsenic found, and

M_2 = mass in g of sample in the solution tested.

APPENDIX B

(*Clause* 3.4)

CORRELATION BETWEEN RELATIVE DENSITY OF OLEUM AND FREE SULPHUR TRIOXIDE CONTENT

Relative Density at 35°C/15°C	*Density in Degree Baume*	*Free Sulphur Trioxide Content, Percent by Mass*
(1)	(2)	(3)
1·818 6	65·3	0
1·822 8	65·5	1
1·827 0	65·6	2
1·831 5	65·8	3
1·836 0	66·0	4
1·839 3	66·2	5
1·842 5	66·3	6
1·846 1	66·5	7
1·849 8	66·6	8
1·853 1	66·8	9
1·856 5	66·9	10
1·859 6	67·0	11
1·862 7	67·2	12
1·866 0	67·3	13
1·869 2	67·4	14
1·872 4	67·6	15
1·875 6	67·7	16
1·879 3	67·8	17
1·883 0	68·0	18
1·887 5	68·2	19
1·891 9	68·4	20
1·896 9	68·6	21
1·902 0	68·8	22
1·905 6	68·9	23
1·909 2	69·1	24
1·912 5	69·2	25
1·915 8	69·3	26
1·918 9	69·4	27
1·922 0	69·6	28
1·925 0	69·7	29
1·928 0	69·8	30

Relative Density at 35°C/15°C	*Density in Degree Baume*	*Free Sulphur Trioxide Content, Percent by Mass*
(1)	(2)	(3)
1·930 9	69·9	31
1·933 8	70·0	32
1·937 2	70·1	33
1·940 5	70·3	34
1·943 9	70·4	35
1·947 4	70·5	36
1·950 4	70·7	37
1·953 4	70·8	38
1·955 9	70·9	39
1·958 4	71·0	40
1·959 8	71·0	41
1·961 2	71·1	42
1·962 7	71·1	43
1·964 3	71·2	44
1·965 8	71·2	45
1·967 2	71·3	46
1·968 7	71·3	47
1·970 2	71·4	48
1·971 7	71·5	49
1·973 3	71·5	50
1·974 1	71·5	51
1·974 9	71·6	52
1·975 5	71·6	53
1·976 0	71·6	54
1·976 6	71·6	55
1·977 2	71·7	56
1·976 3	71·6	57
1·975 4	71·6	58
1·974 6	71·6	59
1·973 8	71·5	60
1·972 3	71·5	61
1·970 9	71·4	62
1·969 1	71·4	63
1·967 2	71·3	64
1·965 4	71·2	65

Relative Density at 35°C/15°C	*Density in Degree Baume*	*Free Sulphur Trioxide Content, Percent by Mass*
(1)	(2)	(3)
1·963 6	71·2	66
1·961 8	71·1	67
1·960 0	71·0	68
1·958 2	71·0	69
1·956 4	70 9	70
1·953 3	70·8	71
1·950 2	70·6	72
1·947 2	70·6	73
1·944 2	70 4	74
1·941 1	70·3	75
1·937 9	70·2	76
1·934 7	70·1	77
1·931 5	69·9	78
1·928 3	69·8	79
1·925 1	69·7	80
1·921 7	69·5	81
1·918 3	69·4	82
1·914 9	69·3	83
1·911 5	69·1	84
1·908 0	69·0	85
1·904 6	68·8	86
1·901 3	68·7	87
1·898 0	68·6	88
1·893 4	68·4	89
1·888 8	68·2	90
1·884 4	68·1	91
1·880 0	68·0	92
1·875 6	67·7	93
1·871 2	67·5	94
1·865 9	67·3	95
1·860 5	67·1	96
1·854 6	66·8	97
1·848 8	66·6	98
1·842 9	66·3	99
1·837 0	66·1	100

APPENDIX C

(*Clause* 5.1)

SAMPLING OF OLEUM, TECHNICAL

C-1. GENERAL REQUIREMENTS

C-1.1 Samples shall not be taken in an exposed place.

C-1.2 The sampling instrument shall be clean and dry.

C-1.3 The material shall be sampled in liquid condition.

C-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

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

C-1.6 The sample shall be placed in suitable, clean, dry and air-tight glass containers, on which the material has no action.

C-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.8 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling and the month and year of manufacture of the material.

C-1.9 Samples shall be stored in a cool and dry place.

C-2. SCALE OF SAMPLING

C-2.1 Lot — All containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture the batches shall be marked separately, and the groups of containers in each batch shall constitute separate lots.

C-2.2 Samples shall be tested from each lot separately for judging the conformity of the material to the requirements of the specification. For this purpose five containers shall be selected at random from each lot.

NOTE — If the number of containers in the lot is five or less, the number of containers to be selected and the criteria for conformity of the lot of the specification shall be as agreed to between the purchaser and the supplier.

C-2.3 The containers shall be selected at random and to ensure randomness of selection, the following procedure is recommended for use:

Starting from any container in the lot, count them as 1, 2 up to r and so on, where r is the integral part of $N/5$ (N being the number of containers in the lot). Every rth container thus counted shall be withdrawn to constitute a sample till the required number of five containers is obtained.

C-3. PREPARATION OF TEST SAMPLES AND REFEREE SAMPLE

C-3.1 **Sampling Tube** — The sampling tube shall be made of glass and shall be 20 to 40 mm in diameter and 350 to 750 mm in length. The upper and lower ends shall be 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.

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

C-3.2 From each of the containers selected according to C-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 with the help of a glass rod.

C-3.3 Out of these portions, a small but equal quantity 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 three equal parts, one for the purchaser, one for the supplier and the third to be used as a referee sample.

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

C-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.

C-3.6 The referee samples consisting of composite sample and a set of 5 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 the case of a dispute between the two.

C-4. NUMBER OF TESTS

C-4.1 Tests for the determination of free sulphur trioxide content shall be performed on each of five individual samples.

C-4.2 Tests for all characteristics given in 3 except free sulphur trioxide shall be performed on the composite sample.

C-5. CRITERIA FOR CONFORMITY

C-5.1 For Individual Samples — From the five test results for free sulphur trioxide content the mean (x) and range (R) of test results shall be calculated. The mean test results shall be greater than or equal to the minimum value specified in col 3 and 4 of Table 1.

C-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 result for each of the characteristics shall satisfy the relevant requirement specified in 3.

(*Continued from page* 2)

Members	*Representing*
SHRI K. P. JOSHI	National Fluorine Corporation, Bombay
SHRI A. J. SHETH (*Alternate*)	
SHRI A. K. MEHRA	Shriram Foods & Fertiliser Industries, New Delhi
SHRI S. K. NANDA (*Alternate*)	
SHRI M. P. MISTRY	Excel Industries Ltd, Bombay
DR ANIL PANDIT	Deepak Nitrite Ltd, Vadodara
SHRI J. T. VORA (*Alternate*)	
SHRI M. D. PATEL	Gujarat State Fertilizers Co Ltd, Vadodara
SHRI S. L. AGGARWAL (*Alternate*)	
SHRI D. N. V. RAO	Tata Chemicals Ltd, Bombay
SHRI C. NEELKANTHAN (*Alternate*)	
SHRI P. ROY	Bengal Chemicals & Pharmaceuticals Ltd, Calcutta
DR A. N. BASU (*Alternate*)	
DR H. C. SHAH	3 K Chemical Co, Thane
SHRIMATI K. H. SHAH (*Alternate*)	
DR M. S. VAIDYA	Dharamsi Morarji Chemical Co Ltd, Bombay
SHRI R. S. VYAS (*Alternate*)	
SHRI H. K. VENKATARAMAIAH	Hindustan Organic Chemicals Ltd, Rasayani

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53/5, Ward No. 29, R. G. Barua Road, 5th Byelane, GUWAHATI 781003	—
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117/418 B Sarvodaya Nagar, KANPUR 208005	21 68 76 21 82 92
Patliputra Industrial Estate, PATNA 800013	6 23 05
Hantex Bldg (2nd Floor), Rly Station Road, TRIVANDRUM 695001	7 66 37
Inspection Offices (With Sale Point):	
Pushpanjali, 205-A West High Court Road, Bharampeth Extension, NAGPUR 440010	2 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	5 24 35

*Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007 — 89 65 28

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Printed at Delhi Printers, Delhi, India