

भारतीय मानक मसौदा
सिलिकॉन टेट्राक्लोराइड, तकनीकी — विशिष्टि
(IS 9398 का दूसरा पुनरीक्षण)

Draft Indian Standard
Silicon Tetrachloride, Technical — Specification
(*Second Revision of IS 9398*)

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ICS 71.060.30

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 9th December 2024

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*formal clauses will be added later*)

This standard was first published in 1979 and subsequently revised in 1987. In the first revision, safety precautions and disposal instructions were incorporated as this material is poisonous and corrosive. Modifications were also made in the test methods for the determination of relative density, distillation yield and free chlorine. Test sample and referee sample clauses were duly amended. In this revision, instrumental test method for the determination of iron has been incorporated. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated and Packing & Marking clause has been updated.

Silicon tetrachloride is widely used in the manufacture of a variety of products such as ethylsilicate, silanes, silicones and semiconductor grade silicon.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

Draft Indian Standard
SILICON TETRACHLORIDE, TECHNICAL — SPECIFICATION
(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for silicon tetrachloride, technical.

2 REFERENCES

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

3 REQUIREMENTS**3.1 Description**

The material shall be a clear liquid, free from sediment and suspended matter, and shall consist essentially of silicon tetrachloride (SiCl₄).

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in col (4) of the table.

Table 1 Requirements for Silicon Tetrachloride, Technical

(Clause 3.2)

Si No.	Characteristic	Requirement	Method of Test (Ref to Clause No. In Annex A)
(1)	(2)	(3)	(4)
i)	Relative density at 27 °C /27 °C	1.470 to 1.480	B-2
ii)	Distillation yield, corrected to 760 mm Hg	Not more than 2 percent by volume below 56 °C and not less than 95 percent by volume between 56 and 59 °C	B-3
iii)	Free chlorine (as Cl), percent by mass, <i>Max</i>	0.01	B-4
iv)	Iron (as Fe), percent by mass, <i>Max</i>	0.01	B-5

4 PACKING AND MARKING**4.1 Packing**

The material shall be packed as agreed to between the purchaser and the supplier. It is preferable that silicon tetrachloride be packed in galvanized iron containers rather than plain steel or cast iron drums.

4.2 Marking

The containers shall be marked with the name and grade (technical) of the material, net mass, manufacturer's name and recognized trade-mark, if any; and the month and year of manufacture.

4.2.1 All containers, in which the material is packed, stored or transported, shall be prominently and clearly marked with the following:

- VOLATILE POISONOUS LIQUID
- USE WITH ADEQUATE VENTILATION
- AVOID PROLONGED BREATHING OF VAPOUR
- AVOID CONTACT WITH FLAME OR HOT SURFACE

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

5 SAFETY PRECAUTIONS AND DISPOSAL INSTRUCTIONS

5.1 Safety Precautions

Since silicon tetrachloride is decomposed by water to yield silicic acid and hydrochloric acid, it is quite corrosive to most metals, when water is present with it. In the absence of water, it has practically no action on iron, steel or the common metals and alloys, and can be stored and handled in metal equipment without danger.

Gas masks to be used while handling.

- First Aid :
- a) If the lungs are affected, remove from exposure and keep warm; in severe cases, get medical attention,
 - b) If skin is affected, drench with plenty of water and remove contaminated clothes.

5.2 Disposal Instructions

While disposing, instruct others to keep at a safe distance. Wear respirator and gloves. Spread soda ash liberally over to spillage and mop up cautiously with plenty of water. Run to waste, diluting greatly with running water.

6 SAMPLING

Representative samples of the material shall be drawn and the conformity of the material to this standard shall be judged according to the procedure prescribed in Annex C.

ANNEX A

(Clause 2)

(LIST OF REFERRED STANDARDS)

<i>IS No</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 2303 (Part 1/Sec 1) : 2021/ ISO 719:2020	Grading glass for alkalinity Part 1 hydrolytic resistance of glass grains Section 1 Determination and classification of hydrolytic resistance at 98 °C (<i>third revision</i>)
IS 2620 : 1963	Specification for distilling flasks
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater
(Part 2) : 2019/ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP – OES) (<i>first revision</i>)
(Part 65) : 2022/ ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (<i>first revision</i>)
IS 4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)

ANNEX B

(Clause 3.2)

METHODS OF TEST FOR SILICON TETRACHLORIDE, TECHNICAL

B-1 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.

B-2 DETERMINATION OF RELATIVE DENSITY

B-2.1 Outline of the Method — The relative density is determined using a suitable hydrometer.

B-2.2 Apparatus

B-2.2.1 Hydrometer — range between 1.400 and 1.600

B-2.2.2 Water bath — maintained at (27 ± 0.5) °C

B-2.2.3 Measuring Jar — 100 ml capacity

B-2.2.4 Thermometer — 0.50 °C

B-2.3 Procedure

Place the material contained in a measuring cylinder in the water bath until its temperature is (27 ± 0.5) °C and read its density with the help of hydrometer. Conduct the experiment in a fuming cup board.

B-3 DETERMINATION OF DISTILLATION YIELD

B-3.1 Outline of the Method

The material is distilled in the specified apparatus under the prescribed conditions of heat input and rate of distillation. 100 ml of the material are distilled and percentages by volume of the distillate recovered at the specified temperatures are recorded.

B-3.2 Apparatus

B-3.2.1 Distillation Flask — of the shape and dimensions shown in Fig. 1 of IS 2620.

B-3.2.2 Thermometer

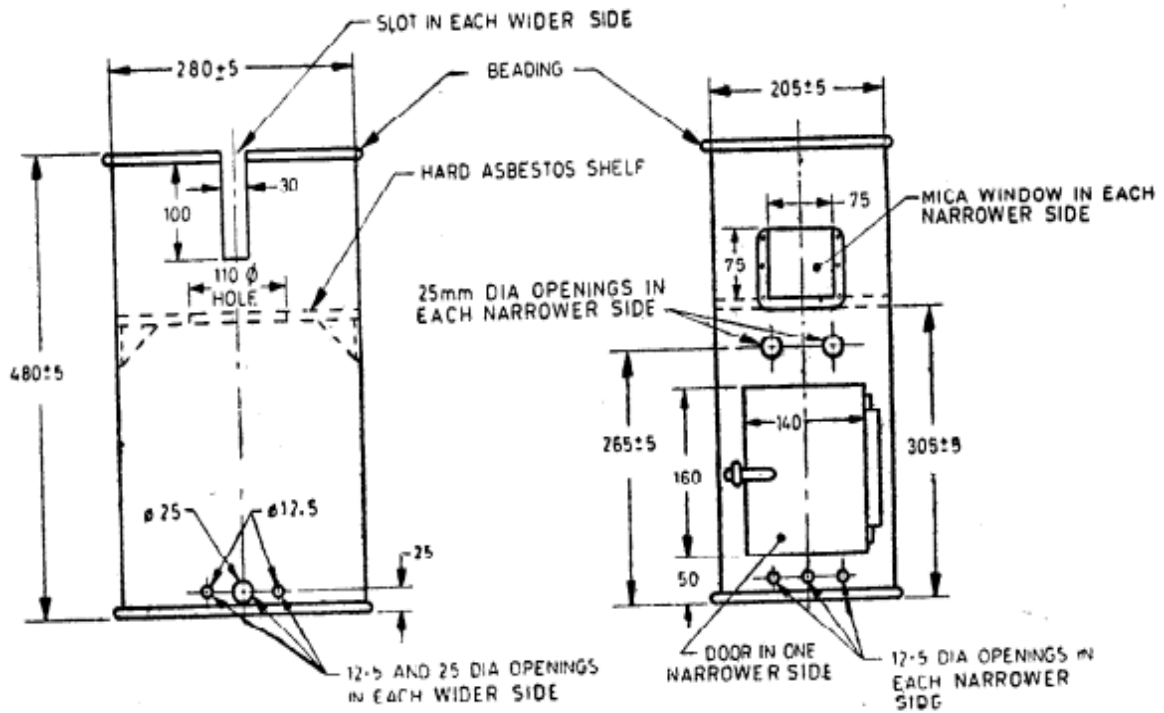
A suitable partial immersion thermometer having a range of 48 °C to 100 °C, graduated at each 0.2 °C and with a maximum scale error of 0.2 °C. The maximum overall length of the thermometer shall be 385 mm and stem diameter shall be 5.5 mm to 8.0 mm.

B-3.2.2.1 The thermometer shall bear a certificate of the National Physical Laboratory, New Delhi or any other organization recognized for such work.

B-3.2.3 Rectangular Draught Screen

Rectangular in cross-section, made of 0.8 mm thick sheet metal with the dimensions shown in Fig. 1, and open at the top and bottom. It shall comply with the following requirements:

- a) In each of the two narrower sides of the draught screen, there shall be two circular holes, each 25 mm in diameter and in each of the four sides of the draught screen, there shall be three holes with their centers 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 1. The diameter of each of the holes, centrally situated in the longer sides, shall be 25 mm and of the remaining ten holes shall be 12.5 mm. At the middle of each of the wider sides, a vertical slot with the dimensions shown in Fig. 1 shall be cut afterwards from the top of the screen. A removable shutter conforming to the dimensions shown in Fig. 2 shall be provided for closing the vertical slot not in use.
- b) A shelf of hard asbestos board, 6 mm in thickness and having a central circular hole 110 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.
- c) In one of the narrower sides of the screen, a door shall be provided having dimensions and position as shown in Fig. 1. In each of the narrower sides of the screen, a mica window shall be placed centrally with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 1
- d) An asbestos board 150 mm × 150 mm × 6 mm in size, having a central hole 50 mm in diameter, shall be so placed on the asbestos shelf that the two holes are approximately concentric and distillation flask, when in position, completely closes the holes of the asbestos board.



All dimensions in millimetres.

FIG. 1 RECTANGULAR DRAUGHT SCREEN

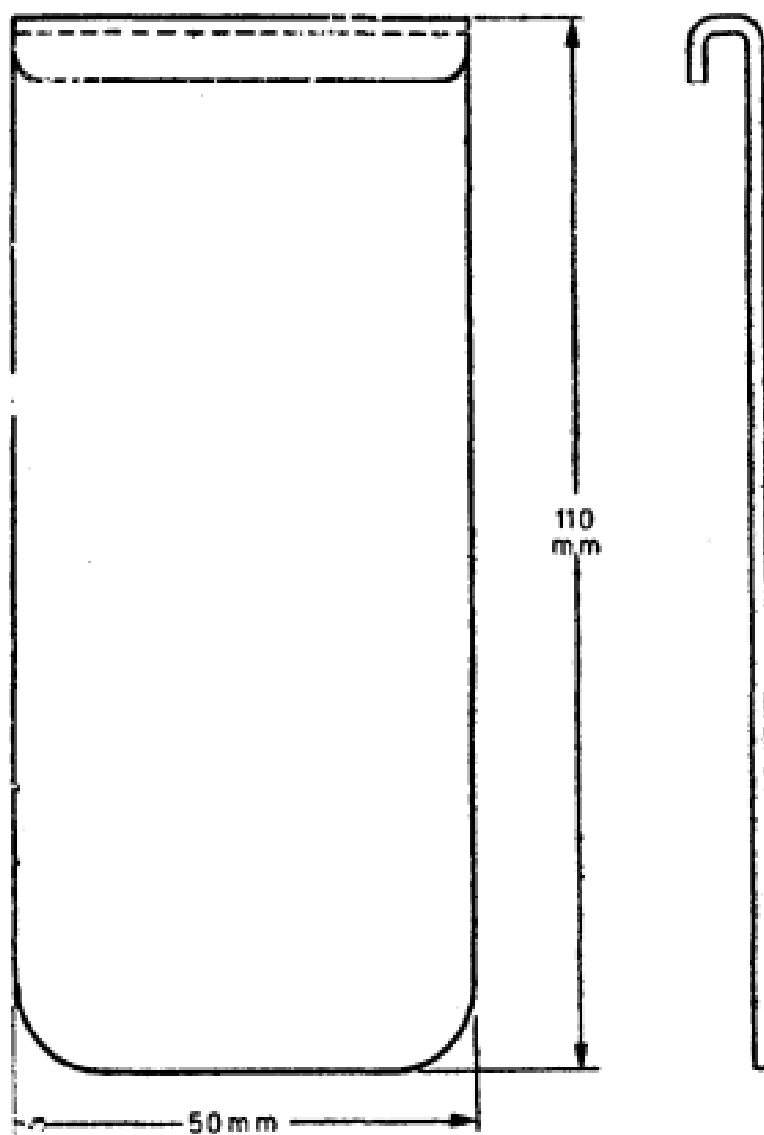
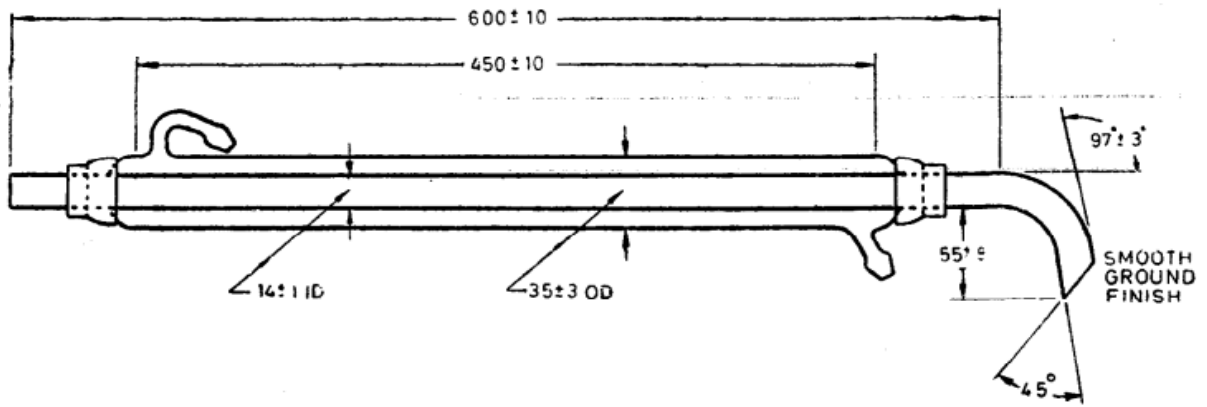


FIG. 2 REMOVABLE SHUTTER

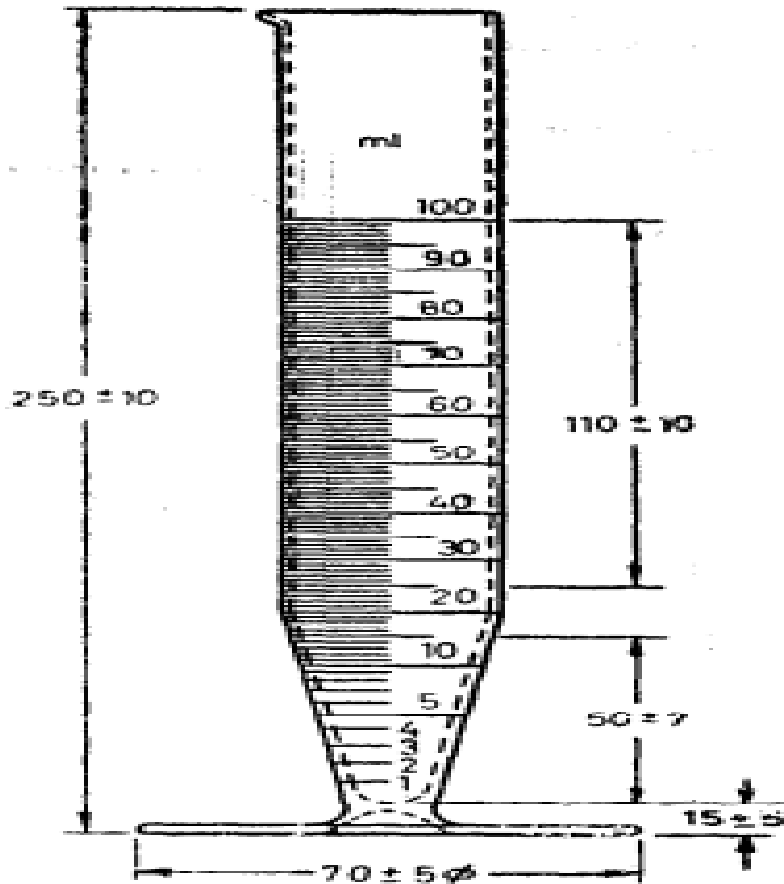
B-3.2.4 *Liebig Condenser* — with the bent end made of Type 1 glass (graded according to IS 2303 Part 1/Sec 1), with a wall thickness of 1.0 mm to 1.5 mm and conforming to the shape and dimensions shown in Fig. 3. The bent portion may, however, be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.

B-3.2.5 *Receiver* — of 100 ml capacity, with dimensions and graduations as shown in Fig. 4.

B-3.2.6 *Electric Heater, Gas Burner or Other Flame Type Heater* — Any suitable heater or burner that enables the distillation to be carried out as described in **B-3.3**



All dimensions in millimetres
FIG. 3 LIEBIG CONDENSER



All dimensions in millimetres
FIG. 4 RECEIVER

B-3.3 Procedure

Assemble the apparatus as shown in Fig. 5. Measure 100 ml of the material at laboratory temperature by means of the receiver and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping, connect the flask to the condenser and insert the thermometer. Fit the thermometer in the flask so that the bottom of the capillary is in level with the lower edge of the side tube joint. Pass an adequate supply of cooling water at 0 °C to 5 °C through the condenser. To receive the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly, especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of the distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be not less than 5 nor greater than 10 min.

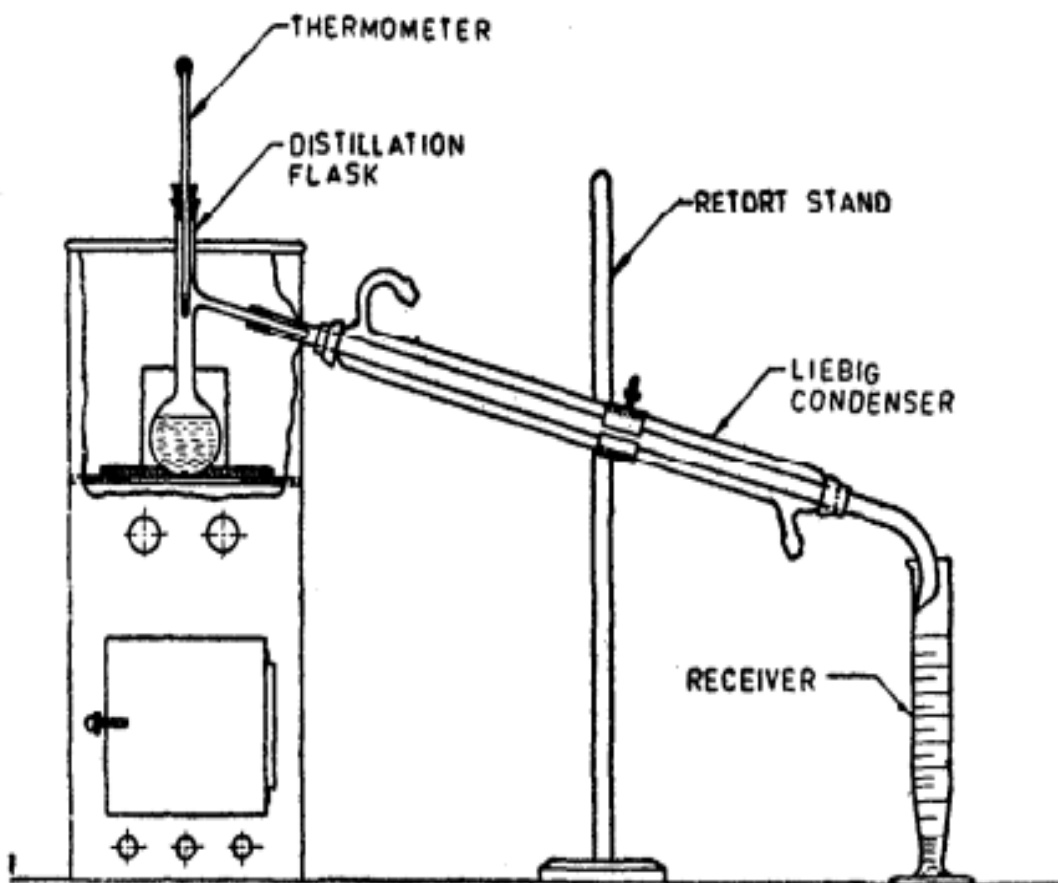


FIG. 5 ASSEMBLY OF APPARATUS FOR DETERMINING DISTILLATION YIELD

Place the receiver in a manner that the condensate flows down its side. Continue the distillation at the rate of 4 ml to 5 ml per minute (about 2 drops per second). Read the volume of the distillate in the receiver, when the thermometer indicates each of the specified distillation temperatures, the temperatures on the thermometer scale being corrected as specified under A-3.4. The difference, between the volumes so recorded is the percentage by volume distilling between the specified temperatures at 760 mm Hg pressure.

B-3.4 Correction of the Thermometer Reading

B-3.4.1 Error of Scale — In all the thermometer reading, make the corrections as indicated on the certificate of the instrument.

B-3.4.2 Correction for Barometric Pressure

If the barometric pressure prevailing during the determination is normal, namely, 760 mm Hg, no correction need be applied to the specified temperatures and the thermometer scale, as corrected under, **B-3.4.1** shall be used as such. If, however, the prevailing pressure deviates from 760 mm Hg, the specified temperatures shall be corrected as follows:

- a) For every 10 mm Hg above 760 mm Hg, add 0.43° to the specified temperature; and
- b) For every 10 mm Hg below 760 mm Hg, subtract 0.43° from the specified temperature.

B-3.4.3 For all joints in the distillation set up, apply fluoro-carbon grease. Conduct the experiment in a fuming cup board.

NOTE — These corrections are valid only for pressures above 700 mm Hg.

B-4 DETERMINATION OF FREE CHLORINE

B-4.1 Outline of the Method

A known quantity of the material is shaken carefully with potassium iodide solution and the liberated iodine is titrated against sodium thiosulphate.

B-4.2 Reagents

B-4.2.1 *Sodium Thiosulphate Solution* — 0.01 N freshly prepared.

B-4.2.2 *Starch Solution*

Triturate 5 g of pure starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste to one, litre of boiling water; boil for 5 min, allow the solution to cool and decant off the clear liquid.

B-4.2.3 *Potassium Iodide Solution* — 5 percent (m/v).

B-4.3 Procedure

B-4.3.1 Take about 500 ml of water containing 10 ml of potassium iodide solution and rundown continuously 10 ml of silicon tetrachloride from a dropping pipette keeping the solution stirring vigorously with the help of a magnetic stirrer. Titrate the liberated iodine with standard sodium thiosulphate solution using starch solution as the indicator. Conduct the experiment in a fuming cup board.

B-4.4 Calculation

$$\text{Free chlorine, percent by mass} = \frac{V \times N \times 3.55}{M}$$

where

V = volume in ml of standard thiosulphate consumed;

N = normality of thiosulphate solution used; and

M = mass in g of the material taken for test [volume multiplied by relative density (*see B-2*)]

B-5 DETERMINATION OF IRON

A-5.1 General

Three methods are prescribed for determining iron, namely, Method A, ICP-OES method as prescribed at **B-6** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-5.2 Method A

A known quantity of the material is evaporated and the residue is extracted with hydrochloric acid and estimated for iron, after removing silica.

B-5.2.1 *Apparatus*

B-5.2.1.1 *Platinum crucible*

B-5.2.1.2 *Nessler cylinder* — 100 ml capacity.

B-5.2.2 Reagents**B-5.2.2.1 Concentrated hydrochloric acid** — see IS 265.**B-5.2.2.2 Ammonium persulphate****B-5.2.2.3 Potassium thiocyanate solution** — 5 percent.**B-5.2.2.4 Hydrofluoric acid****B-5.2.2.5 Standard iron solution**

Dissolve 0.702 g of ferrous ammonium sulphate [$\text{Fe SO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 100 ml water and 10 ml of concentrated sulphuric acid, and dilute with water to 1 000 ml mark. Transfer 100 ml of this solution to 1 litre volumetric flask and dilute to 1 000 ml mark. One milliliter of this solution is equivalent to 0.01 mg of iron (as Fe).

B-5.2.3 Procedure

B-5.2.3.1 Take 20 ml of silicon tetrachloride in a platinum dish and evaporate in a fuming cup-board. Add 5 ml of hydrofluoric acid to the residue and a few drops of sulphuric acid, and again put on a sand bath in a fuming cup-board. When all the fumes have stopped, remove the dish, heat on a light flame, cool and digest the residue with concentrated hydrochloric acid. Extract the residue and make up the volume to 100 ml with water. Pipette out 25 ml of the made up solution, add 30 mg of ammonium persulphate and boil to oxidize the iron. Cool and transfer to a 100 ml Nessler cylinder, add 2 ml of potassium thiocyanate solution and dilute to 100 ml mark with water. In another Nessler cylinder, take the same amount of acid, ammonium persulphate and potassium thiocyanate solution as used with the material and dilute to about 85 ml. Add standard iron solution in small portions at a time so that after dilution to 100 ml, the colour obtained matches with that obtained with the material.

B-5.2.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{V_1}{250 \times D \times V_0}$$

where

 V_1 = volume in ml of standard iron solution; D = relative density according to **B-2.3**; and V_0 = volume in ml of the sample taken for the test.**B-6 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD****B-6.1 Principle**

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively coupled plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

B-6.2 Recommended Wavelength, limit of quantification and important spectral interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

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

(Clauses B-6.2 and B-6.4)

SI No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

B-6.3 Reagents and Solutions**B-6.3.1. Nitric Acid (65 percent) Suprapure****B-6.3.2 Standard Stock Solution**

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 $\mu\text{g}/\text{ml}$ of Iron in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-6.3.3 Standard Solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 $\mu\text{g}/\text{ml}$ solution. From this 5 $\mu\text{g}/\text{ml}$ solution, an aliquot of 1.0, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 $\mu\text{g}/\text{ml}$ solution of respective elements under reference.

B-6.3.4 Sample Preparation

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

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

B-6.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluoroethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

B-6.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

B-6.5 Procedure**B-6.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (B-6.3.5). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

B-6.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in $\mu\text{g/ml}$ of the Iron in the sample solution.

NOTE — It is recommended that IS 3025 (Part 2) /ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

B-6.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

- i) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- ii) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (Dilution factor).

ANNEX C

(Clause 6)

SAMPLING OF SILICON TETRACHLORIDE, TECHNICAL

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 The sampling instrument shall be clean and dry

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

C-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by shaking or stirring or both by suitable means or by rolling.

C-1.4 The samples shall be placed in suitable, clean, dry and air-tight metal or dark or amber glass containers on which the material has no action.

C-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.6 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, and the month and year of manufacture of the material.

C-1.7 Samples shall be stored in the dark.

C-2 SAMPLING INSTRUMENT

C-2.1 The following sampling instrument may be used:

- a) Sampling bottle or can, for taking samples from tanks or drums; and
- b) Sampling tube, for taking samples from bottles or small containers.

C-2.1.1 *Sampling Bottle or Can* — consists of a weighted glass or metal container with removable stopper or top to which is attached a light chain (*see* Fig. 6). The bottle or the can is fastened to a suitable pole. For taking a sample,

the bottle or the can is lowered into the tank to the required depth and the stopper is then removed by means of the chain.

C-2.1.2 Sampling Tube — made of metal or thick glass, 20 mm to 40 mm in diameter and 400 mm to 800 mm in length (see Fig. 7). The upper and lower ends are conical and reach 5 mm to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end.

C-2.1.2.1 For small containers, the size of the sampling tube may be altered suitably.

C-3 SCALE OF SAMPLING

C-3.1 For Tanks and Drums — Each tank or drum shall be sampled separately.

C-3.2 For Bottles and Small Containers — Each lot (see C-3.2.1) shall be sampled separately.

C-3.2.1 Lot

In any consignment, all the containers drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

C-3.2.2 For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested from each lot separately.

C-3.2.3 The number of containers (n) to be selected from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 3.

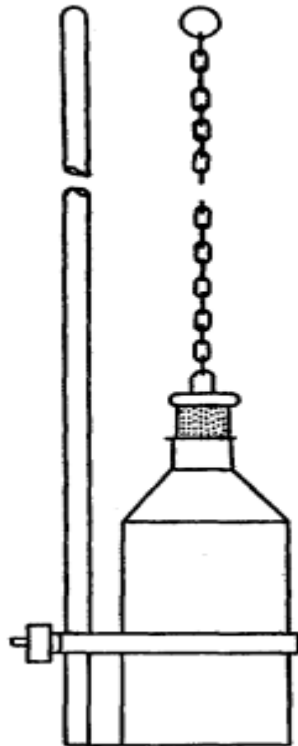
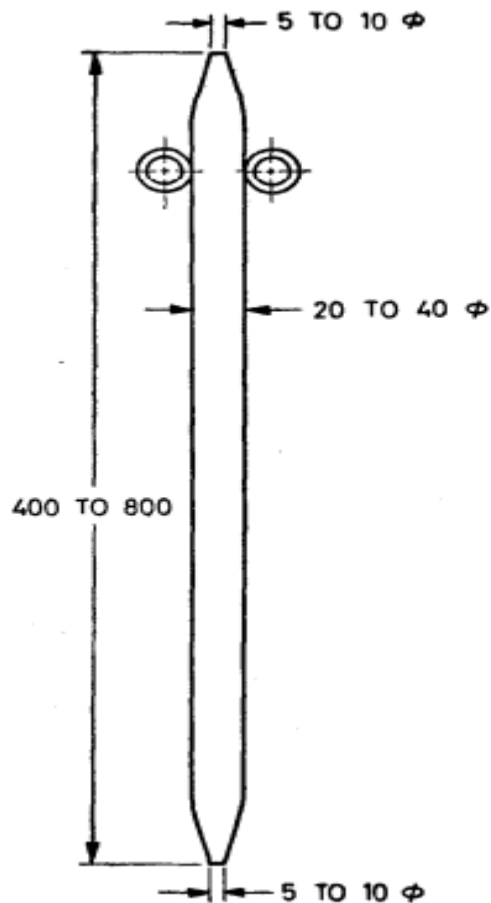


FIG. 6 SAMPLING BOTTLE OR CAN



All dimensions in millimetres.
FIG. 7 SAMPLING TUBE

C-3.2.4 The containers shall be selected at random from the lot. In order to ensure randomness of selection, reference may be made to IS 4905. In case this standard is not available, the following procedure is recommended for use:

Starting from any container in the lot, count them in one order as 1, 2 up to r and so on, where r is the integral part of N/n . Every r^{th} container thus counted shall be withdrawn until the requisite number of containers is obtained.

Table 3 Scale of Sampling

(Clause C-3.2.3)

Lot Size N (1)	No. of Containers to be Selected n (2)
up to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
111 and above	10

C-4 TEST SAMPLE AND REFEREE SAMPLE

C-4.1 From Tanks and Drums

As far as possible, samples from a tank or drum should be drawn during the operation of filling. In that case, equal amounts of the material shall be collected at regular intervals so as to get a total amount of about 1 000 ml. Where it is not possible to take a sample during filling, the material shall be drawn from different depths with the sampling bottle or can, after thoroughly agitating the material so as to ensure a fair amount of homogeneity. The total amount of the material collected shall be thoroughly mixed and divided into three equal portions, one for the purchaser, and another for the supplier and the third for the referee.

C-4.2 From Bottles and Small Containers

From each of the bottles or containers selected according to **C-3.2.4**, a small representative portion of material shall be drawn with the help of the sampling tube. Equal quantities of the material so drawn from the various containers shall be thoroughly mixed to form a test sample of about 1 000 ml. This shall be divided into three equal parts, one for the purchaser, and another for the supplier and the third for the referee.

C-4.3 All the test samples shall be transferred to separate sample containers, and sealed and labelled with full identification particulars. The referee test sample bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two and shall be used in case of a dispute. The sample bottles shall be securely packed in a box containing sufficient amount of soda ash.

C-5 NUMBER OF TESTS

Tests for the determination of all the requirements given in this specification shall be performed on the composite sample obtained in **C-4.1** or **C-4.2**.

C-6 CRITERIA FOR CONFORMITY

The lot shall be declared as conforming to this specification if all the test results satisfy the prescribed requirements.

