

फस्फोरस ऑक्सीक्लोराइड,
तकनीकी — विशिष्टि
(पहला पुनरीक्षण)

Phosphorus Oxychloride,
Technical — Specification
(First Revision)

ICS 71.060

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FOREWORD

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

This standard was originally published in 1986 and is now being revised to incorporate requirements of arsenic, iron and phosphorus trichloride along with their methods of test.

Phosphorus oxychloride finds wide use as a catalyst and chlorinating agent in dyestuff industries, pesticides and pharmaceutical industries. This standard, however, does not cover pharmaceutical grade. Phosphorus oxychloride is an important starting product for the manufacture of phosphoric acid, triesters, obtained from reactions of POCl_3 with alcohols, phenols and epoxides. These esters are used in plasticizers as additives for motor fuels and lubricating oils.

The list of experts who had made significant contribution to the formulation of this standard is given at Annex C.

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 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.

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Indian Standard

PHOSPHORUS OXYCHLORIDE, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for phosphorus oxychloride, technical.

2 REFERENCES

The standards listed below 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:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (<i>third revision</i>)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods (<i>first revision</i>)
4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a colourless to pale yellow fuming liquid with irritating odour.

3.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

**Table 1 Requirements for Phosphorus
Oxychloride**
(Clause 3.2)

SI No.	Characteristic	Requirement	Method of Test (Ref to Cl No. In Annex A)
(1)	(2)	(3)	(4)
i)	Boiling range (95 percent distillation by volume)	104 to 108 °C	A-2
ii)	Relative density at 27°C	1.64 to 1.67	A-3
iii)	Assay (as POC1 ₃ percent by mass, <i>Min</i>)	98	A-4
iv)	Arsenic (percent by mass), <i>Max</i>	0.000 002	A-5
v)	Iron (percent by mass), <i>Max</i>	0.000 5	A-6
vi)	Phosphorus trichloride (percent by mass), <i>Max</i>	0.3	A-7

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in drums with polyethylene liners, in high density polyethylene carboys or in clean glass carboys.

4.2 Marking

The containers shall be marked legibly and indelibly with the following information:

- a) Name of the material;
- b) Name of the manufacturer and recognized trade-mark, if any;
- c) Net mass and batch No.; and
- d) Date of manufacture.

IS 11657 : 2020

NOTE — The word 'Poisonous' together with the symbol as given in Fig. 15 of IS 1260 (Part 1) shall also be given on the label. The label shall have a note as follows:

CAUTION — Phosphorus oxychloride is highly corrosive. When mixed with water, it decomposes with evolution of heat.

4.2.1 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 SAMPLING

Representative samples of the material shall be drawn in accordance with the method prescribed in Annex B.

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ANNEX A

(Clause 3.2)

METHODS OF TEST FOR PHOSPHORUS
OXYCHLORIDE, TECHNICAL

A-1 QUALITY OF REAGENTS

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

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

A-2 BOILING RANGE

A-2.1 Apparatus

A-2.1.1 Round Bottom Flask, of the shape and dimensions as shown in Fig. 1.

A-2.1.2 Thermometer, 98 to 152 °C.

A-2.1.3 Leibig Condenser, Band with long stem as shown in Fig. 2.

A-2.1.4 Receiver Measuring Cylinder, 100 ml with the dimensions and graduations as shown in Fig. 3.

A-2.1.5 Glass Beads

A-2.2 Procedure

Assemble the apparatus as shown in Fig. 4. Take 100 ml sample in the round bottom flask. Heat it, then note down reading (that is, temperature of very first drop) that is starting temperature. Then take a reading after 5 ml and another after 95 ml keeping a rate of 120 to 125 drops per minute.

A-2.3 Result

Boiling range = x °C/5 ml to y °C/95 ml.

where

x = temperature at 5 ml, and

y = temperature at 95 ml.

A-3 DETERMINATION OF RELATIVE DENSITY

A-3.1 Apparatus

A-3.1.1 Hydrometer, 1 600 to 1 700.

A-3.1.2 Cylinder, 250 ml.

A-3.1.3 Thermometer

A-3.2 Procedure

Pour the material to be tested into the clean hydrometer jar, the diameter of which shall be at least 2.5 cm greater

than the diameter of the hydrometer used. Remove all air bubbles that might have formed in the liquid. Keep the jar in a vertical position and in the bath maintained at 30 °C. When the sample in the jar attains the temperature of the bath, that is 30 °C, lower the hydrometer gently into the material. When it has settled depress it about two scale divisions into the liquid. Keep the unimmersed portion of the stem dry as any unnecessary liquid on the stem will change the effective weight of the instrument and affect the reading obtained. Allow the hydrometer to become stationary. Remove all air bubbles that might have formed during lowering of the hydrometer. Read the point on the hydrometer scale to which the sample rises with the eye placed at the principal surface of the material. This reading gives the relative density of the material under test.

A-4 DETERMINATION OF PHOSPHORUS
OXYCHLORIDE

A-4.1 Two methods, namely, acidimetric and excess chloride method have been prescribed for this determination. Excess chloride method shall be the referee method.

A-4.2 Assay (Acidimetric Method)

A-4.2.1 Outline of the Method

Phosphorus oxychloride is hydrolyzed by water to give phosphoric acid and hydrochloric acid. This is determined by acid base titration.

A-4.2.2 Reagents

A-4.2.2.1 Standard sodium hydroxide, 0.1 N.

A-4.2.2.2 Thymolphthalein indicator, 0.1 percent.

A-4.2.3 Apparatus

A-4.2.3.1 pH meter

A-4.2.4 Procedure

Weigh accurately about 1 g of the sample (use a stoppered weighing bottle) in about 100 ml of water and about 30 g of sodium chloride. Add about 0.5 ml of thymolphthalein indicator solution and titrate with 0.1 N sodium hydroxide to a blue colour (not green) pH 9.6. For more accurate work, use a pH meter.

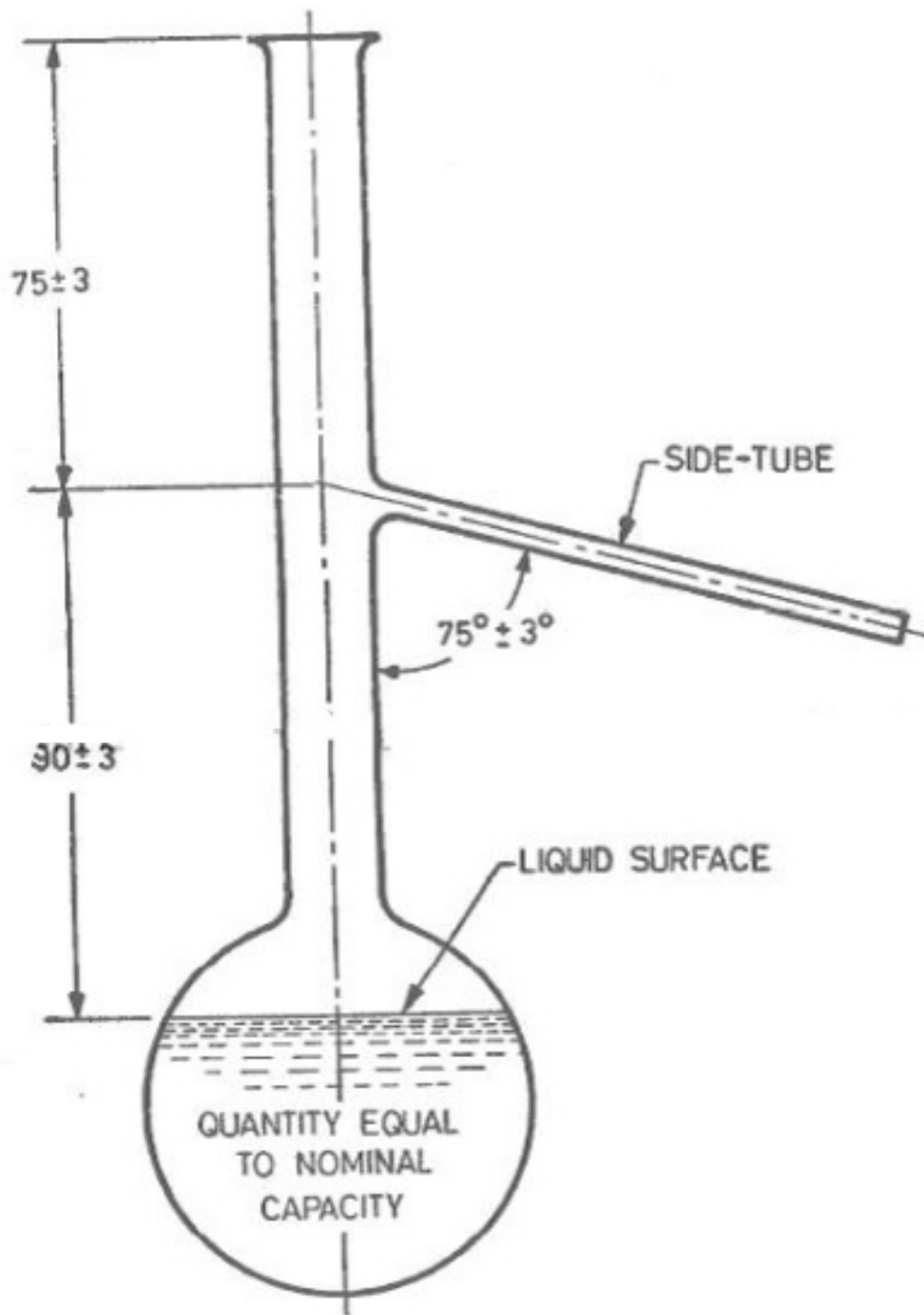
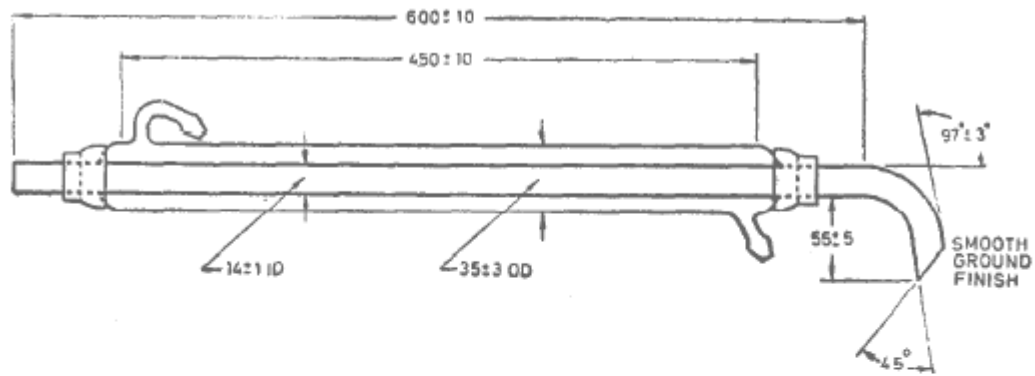
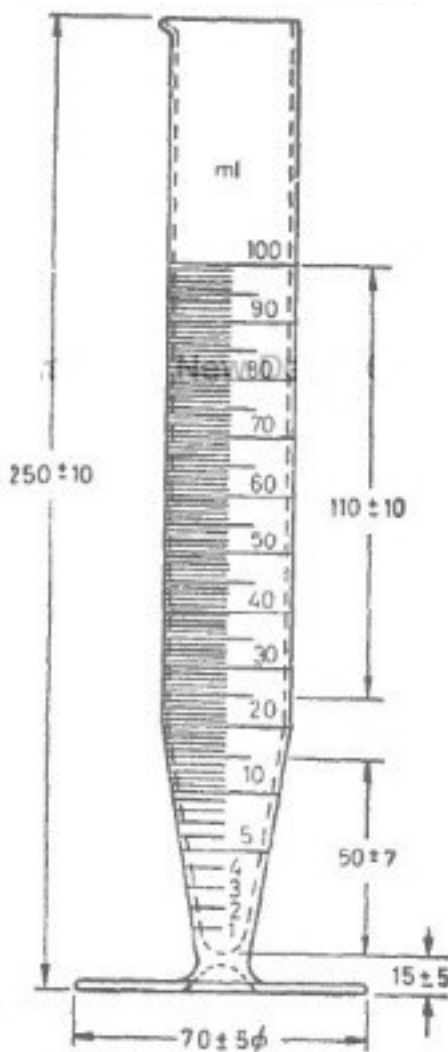


FIG. 1 DISTILLATION FLASK



All dimensions in millimetres

FIG. 2 LEIBIG CONDENSER



All dimensions in millimetres

FIG. 3 RECEIVER

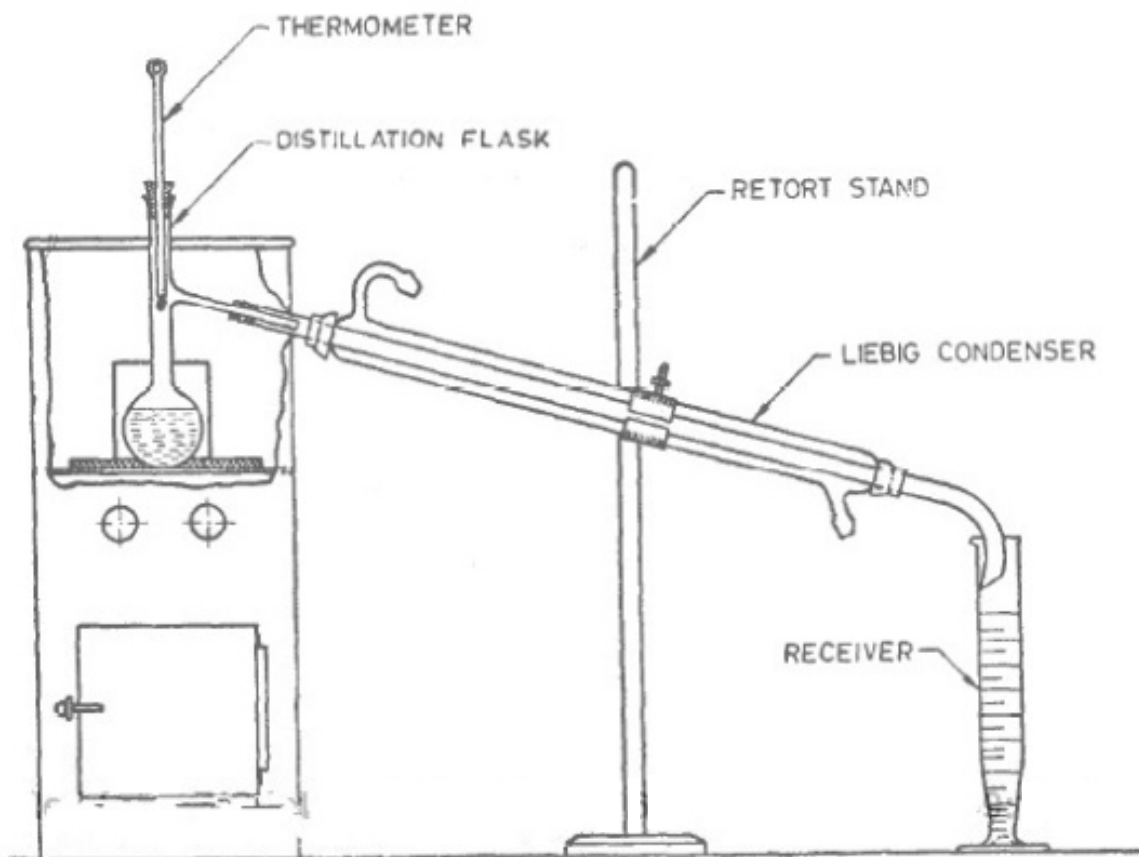


FIG. 4 ASSEMBLY OF APPARATUS

A-4.2.5 Calculation

Phosphorus oxychloride (POCl_3), percent by mass

$$= \frac{V \times N \times 3.07}{M}$$

where

V = volume in ml, of sodium hydroxide,

N = normality of sodium hydroxide, and

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

A-4.3 Assay (Excess Chloride Method)**A-4.3.1 Outline of the Method**

Phosphorus oxychloride is hydrolyzed by water to give phosphoric acid and hydrochloric acid. Hydrochloric acid is determined by excess chloride.

A-4.3.2 Reagents

A-4.3.2.1 Standard silver nitrate solution, 0.1 N.

A-4.3.2.2 Standard ammonium thiocyanate solution, 0.1 N.

A-4.3.2.3 Ferric alum indicator, 40 percent.

A-4.3.2.4 Dilute nitric acid, 6 N.

A-4.3.2.5 Nitrobenzene

A-4.3.3 Procedure

Weigh accurately about 0.2 g of sample in about 50 ml of water contained in a conical flask. Shake well and add 50 ml N/10 silver nitrate and 5 ml of 6 N nitric acid. Add 1 ml ferric alum indicator and 5 ml of nitrobenzene. Back titrate with N/10 ammonium thiocyanate. End point will be brown colour. Carry out a blank filtration.

A-4.3.4 Calculation

Phosphorus oxychloride (POCl_3), percent by mass =

$$\frac{(V_1 N_1 - V_2 N_2) \times 0.51}{M}$$

where

V_1 = volume in ml, of silver nitrate;

N_1 = normality of silver nitrate;

V_2 = volume in ml, of ammonium thiocyanate;

N_2 = normality of ammonium thiocyanate; and

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

A-5 TEST FOR ARSENIC

A-5.1 General

The stain produced by arsenic or mercuricbromide paper is compared with a standard stain.

A-5.2 Reagents

A-5.2.1 Concentrated Hydrochloric Acid

A-5.2.2 Hydrazine Sulphate

A-5.2.3 Sodium Bromide

A-5.3 Preparation of Test Solution

A-5.3.1 For Technical Grade

Weigh 1.0 g of the material and transfer to a small distillation flask. Add 10 ml of water, 15 ml of concentrated hydrochloric acid, 0.25 g of hydrazine sulphate and 0.25 g of sodium bromide. Connect the flask to a condenser and distil the contents till 20 ml of the distillate are collected. Use this distillate for carrying out the test for arsenic, as described in IS : 2088 using for comparison a stain obtained with 0.015 mg of arsenic trioxide.

A-5.3.2 For Analytical Reagent Grade

Weigh 3.0 g of the material and carry out the test for arsenic by the modified Gutzeit method as prescribed in 5.1 or spectrophotometric method as prescribed in 5.2 of IS : 2088 using for comparison in 5.1 a stain obtained with 0.003 mg of arsenic trioxide. In case of dispute, the spectrophotometric method shall be the referee method.

A-6 DETERMINATION OF IRON

A-6.1 Principle

The dissolved iron under slightly acidic medium is reduced using hydroxyl ammonium chloride and the colour is developed with 2,2'-bipyridyl solution [iron (II)-2,2' bipyridyl complex]. The absorbance of the colour is measured by a spectrophotometer at 522 nm.

A-6.2 Apparatus

A-6.2.1 Spectrophotometer, suitable for measurement at 522 nm, with accuracy of 1 nm.

A-6.2.2 Standard Laboratory Glass Apparatus

A-6.3 Reagents

A-6.3.1 Distilled Water

A-6.3.2 Hydrochloric Acid, approximately 6 mol/l (1 : 1 v/v).

A-6.3.3 Hydrochloric Acid, approximately 1 mol/l.

To 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

A-6.3.4 Hydroxylammonium Chloride

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

A-6.3.5 Ammonium Acetate Solution, 30 percent (m/v).

A-6.3.6 2, 2'-Bipyridyl Solution

Dissolve 1 g of 2,2'-Bipyridyl in 10 ml of hydrochloric acid solution (1 mol/l) and dilute to 100 ml.

A-6.3.6.1 Standard iron solution A

Dissolve 0.7022 g of ferrous ammonium sulphate hexahydrate [$\text{FeSO}_4\cdot\text{NH}_4\text{SO}_4\cdot 6\text{H}_2\text{O}$] in water in a 250 ml beaker, add 50 ml of sulphuric acid (about 1 mol/l) and mix. Transfer quantitatively into a one-litre volumetric flask, make up to the mark and mix well. One ml of this solution contains 0.1 mg iron (as Fe).

A-6.3.6.2 Standard iron solution B

Dilute 100 ml of the above solution (solution A) and dilute to 1 litre in a volumetric flask, mix well. One millilitre of this solution contains 10 μg of iron (as Fe).

A-6.4 Procedure

A-6.4.1 Calibration

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

Standard Iron Solution. B (ml)	Corresponding Iron Content as Fe μg
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 50 ml, then 2 ml of hydrochloric acid and 2 ml of hydroxylammonium chloride solution and after 5 min, add 5 ml of ammonium acetate solution and 1 ml of 2,2' bipyridyl solution. Dilute to mark, mix well, and wait for 10 min. Measure absorbance of each solution using spectrophotometer with 1-cm cell at 522 nm against reagent blank (containing equal amount of added reagent) and make up the volume to 100 ml.

A-6.4.2 Blank Test

Prepare a blank test solution using same procedure as used for determination of iron but excluding phosphorus pentachloride.

A-6.4.3 Preparation of Calibration Graph

Prepare a calibration graph with iron as Fe in 1.0 µg/100 ml as abscissa and absorbance as ordinates.

A-6.5 Determination of Iron

Weigh accurately to the nearest 1 mg, an amount of POCl₃ containing 100 to 1 000 µg of iron (as Fe) into a platinum dish. Wet it in minimum amount of distilled water and neutralize with 6 mol/l hydrochloric acid solution and add a few drops in excess. Evaporate to dryness on a water bath. Wet it with a few drops of dilute hydrochloric acid and then dissolve in 20 to 30 ml of distilled water and quantitatively transfer into a 100 ml one-mark volumetric flask, dilute to mark and mix well. If the solution is turbid filter through a Whatman No. 541 filter paper and discard 10 to 15 ml of the initial filtrate.

Transfer a suitable aliquot volume (or the filtrate if filtered) containing 50 to 500 µg of Fe into a 100 ml one-mark volumetric flask. Dilute to around 50 ml (if less), add 2 ml hydrochloric acid and 2 ml of hydroxyl ammonium chloride. Mix well and after 5 min add 5 ml of ammonium acetate solution followed by 1 ml of 2,2'-bipyridyl solution. Dilute to mark, mix well and allow to stand for 10 min. Measure the absorbance using spectrophotometer at 522 nm wave length as described for calibration. Use the same cell size as the one used for calibration and use blank test solution as reference.

A-6.6 Calculation and Expression of Results

By means of the calibration graph determine the concentration of iron in micrograms corresponding to the absorbance value of the test solution. Calculate the iron content as-

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

where

A = mass in g, of iron determined in the sample solution, and

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

A-7 DETERMINATION OF PHOSPHORUS TRICHLORIDE**A-7.1 Principle**

Chromatography is a process of separating the components of gaseous mixture by means of partition

between stationary phase and mobile phase. The stationary phase retains some components strongly which thereby spends less time in the mobile phase and thus passes slowly from the column. On the contrary, the components that are retained weakly will spend more time in the mobile phase and will pass quickly from the column. It is due to the difference in retention time that the components are separated into bands or zones as they pass through the column with the mobile phase.

When the accuracy of analysis is to be achieved at the level of 1 PPM (v/v), the results obtained are relative to the impurities of carrier gas and not absolute values. Hence, the ultrahigh purity carrier gas, that is, Grade 1 helium is to be purified further by a suitable rare gas purifier to achieve zero impurity gas. Argon gas may also be used as a carrier gas.

The purifier system consists of titanium granules at 700 °C for removal of nitrogen and oxygen, a copper oxide furnace for removal of hydrogen and carbon monoxide, and molecular sieve to remove carbon dioxide and moisture. The construction of the gas chromatograph is same as that of any other gas chromatograph with a gas sampling valve and a loop of known volume.

A-7.2 Procedure

Operate the instrument as per the instructions given by the manufacturer. Prepare external standards for PCl₃ and store them in teflon bottles. Use nitrogen phosphorus detector for analysis under following recommended conditions:

- a) *Detector Temperature* — 100 °C;
- b) *Hydrogen pressure* — 15-18 psig; and
- c) *Air Pressure* — 30 psig.

Operate detector in the N-mode. P-mode is suitable for qualitative analysis.

A-7.2.1 Sample Analysis

An aliquot (0.1µl-3µl) of the sample was introduced. During the beginning of the analysis, the switching valve was so positioned that eluate was vented and just the helium carrier gas flowed through the column. As soon as the eluted phosphorus trichloride is sent to the detector through column, the switching valve is turned back out. The concentration of PCl₃ in the sample was determined according to the external standard method of analysis. This is done by comparing the peak height of the standard with that of unknown concentrations.

ANNEX B

(Clause 5)

SAMPLING**B-1 GENERAL REQUIREMENTS OF SAMPLING**

B-1.1 In drawing, storing, preparing, and handling test samples the precautions given in **B-1.2** to **B-1.6** shall be observed.

B-1.2 Samples shall not be taken at a place exposed to weather.

B-1.3 Precautions shall be taken to protect the samples, the sampling instruments and the containers for samples from adventitious contamination.

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

B-1.5 The samples shall be placed in suitable, clean dry and air-tight containers.

B-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 year of manufacture.

B-2 SCALE OF SAMPLING**B-2.1 Lot**

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

B-2.2 The number of containers (n) to be selected from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

(Clause B-2.2)

Sl No.	Lot Size	No. of Containers to be Selected
(1)	(2)	n (3)
i)	Up to 50	3
ii)	51 to 100	4
iii)	101 to 150	5
iv)	151 to 300	7
v)	301 and above	10

B-2.3 These containers shall be selected at random from the lot and in order to ensure the randomness of selection, procedures given in IS 4905 may be followed.

B-2.4 Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of the specification.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected according to **B-2.3** a portion of the liquid, about 200 ml, shall be drawn with the help of a suitable sampling instrument.

B-3.2 Out of these portions drawn from each container, equal quantities of liquid shall be taken and mixed thoroughly to form a composite sample of 600 ml. The composite sample shall be divided into three parts, one for the purchaser, another for the supplier and the third used as a referee sample.

B-3.3 The remaining portion of the liquid drawn from each container, shall be divided into three equal parts each forming an individual sample. One set of individual sample representing 'N' containers sampled shall be marked for the purchaser, another for the supplier and the third used as a referee sample.

B-3.4 All the individual samples and the composite samples shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

B-3.5 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier to be used in case of a dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of phosphorus oxychloride content shall be performed on each of the individual samples.

B-4.2 Tests for the determination of all other characteristics given in Table 1 shall be performed on the composite sample.

ANNEX C

(Foreword)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE
DEVELOPMENT OF THIS STANDARD

COMMITTEE COMPOSITION

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Member Secretary

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SCIENTIST 'C' (CHD), BIS

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: CHD 01 (15165).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi