भारतीय मानक
Indian Standard

IS 11744 : 2020

फस्फोरस पेन्टाक्लोराइड, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

Phosphorous Pentachloride, Technical — Specification

(First Revision)

ICS 71.060

भारतीय मानक ब्यूरो
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December 2020 **Price Group 5**

Inorganic Chemicals Sectional Committee, CHD 01

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 sulphate, iron and lead along with the methods of test.

Phosphorous pentachloride is mainly used as a protective chlorinating agent in the manufacture of pharmaceutical and dyestuffs. Phosphorous pentachloride also serves as a catalyst for condensation and cyclization reactions and for improving grain structure of light metal castings and as a reagent in analytical laboratory. It is also used as dehydrating agent.

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 a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PHOSPHOROUS PENTACHLORIDE TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for phosphorous pentachloride, technical.

2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision 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.

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)
8883 (Part 1) : 2005	Methods of sampling chemical and chemical products: Part 1 General requirements and precautions (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of pale yellow fine granular powder, having an irritating odour, soluble in carbon disulphide and carbon tetrachloride.

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 Annex A is given in col 4 of the table.

Table 1 Requirements for Phosphorous Pentachloride, Technical

(*Clause* 3.2)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to Cl No. of Annex A (4)
i)	Phosphorous pentachloride (as percent by mass, *Min*)	97.0	A-2
ii)	Bulk density, g/ml	0.9	A-3
iii)	Sulphate (ppm), *Max*	50	A-4
iv)	Iron (ppm), *Max*	5	A-5
v)	Lead (ppm), *Max*	5	A-6

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in a polyethylene bag of not less than 50 micron thickness and then put either in a mild steel drum or a high density polyethylene container.

4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Gross and net mass;
d) Date of manufacture; and
e) Batch number.

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 SAMPLING

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

ANNEX A

(*Clause* 3.2 *and* Table 1)

METHODS OF TEST FOR PHOSPHOROUS PENTACHLORIDE, TECHNICAL

A-1 QUALITY OF REAGENTS

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

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

A-2 DETERMINATION OF PHOSPHOROUS PENTACHLORIDE

A-2.1 Two methods, namely, acidimetric and argentometric methods have been prescribed for this determination. Argentometric method shall be the referee method.

A-2.1.1 *Reagents*

A-2.1.1.1 *Standard sodium hydroxide*, 1 N.

A-2.1.1.2 *Standard sulphuric acid,* 1N.

A-2.1.1.3 *Methyl orange indicator*

A-2.1.1.4 *Standard silver nitrate,* 0.1 N.

A-2.1.1.5 *Standard ammonium thiocyanate,* 0.1 N.

A-2.1.1.6 *Ferric ammonium sulphate,* 40 percent.

A-2.1.1.7 *di-n Butyl phthalate*

A-2.2 Procedure

a) *Acidimetric* — Accurately weigh about 1.2 g in a sealed ampoule. Place the ampoule in 50.0 ml of N sodium hydroxide contained in a stoppered 250 ml flask. Break the ampoule below the surface of the liquid and shake, set aside for 10 min, remove the stopper carefully, rinsing with a little water. Heat the flask on a steam bath for 30 min, Cool and titrate with N sulphuric acid using methyl orange indicator to a grey end point. Retain the solution for argentometric method, if required (b).

b) *Arqentometric* — Dilute the contents of the flask retained from acidimetric method (a) to 500.0 ml with water. To 50.0 ml, add 10 ml of nitric acid, 50.0 ml of 0.1 N silver nitrate, 3 ml of din-butyl phthalate and shake vigorously for 1 minute. Titrate the excess 0.1 N silver nitrate with 0.1 N ammonium thiocyanate using ammonium ferric sulphate as indicator shaking vigorously between each successive addition of titrant.

A-2.3 Calculation

a) *Acidimetric*

Phosphorous pentachloride (as $PC1_5$), percent by mass

$$= \frac{(V_1 - V_2) \times 3.47}{M}$$

where

V_1 = volume in ml, of standard sodium hydroxide added,

V_2 = volume in ml, of standard hydrochloric acid, and

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

b) *Argentometric*

Phosphorous pentachloride (as $PC1_5$), percent by mass

$$= \frac{(V_1 - V_2) \times 4.16}{M}$$

where

V_1= volume in, ml of standard silver nitrate solution;

V_2 = volume in ml of standard ammonium thiocyanate; and

M = mass in g, of sample.

A-3 BULK DENSITY

A-3.1 Procedure

Rest the funnel over the top edge of the tared graduated cylinder, Fill the cylinder to the 100 ml mark by pouring the material through the funnel, and drop it 20 times through a height of 15 cm on to a felt pad resting on a bard surface. Note the volume of the material after compacting and weigh the filled cylinder.

A-3.2 Calculation

$$\text{Bulk density, g/ml } \frac{M}{V \times 1\,0000}$$

where

M = mass in g, of the material in the cylinder; and

V = volume in ml, occupied by the material after compacting.

A-4 DETERMINATION OF SULPHATE

A-4.1 General

Sulphate is determined by spectrophotometer, developing the turbidity of barium sulphate. The turbidity of a dilute barium sulphate suspension is difficult to reproduce. It is, therefore, essential to adhere rigidly to the conditions of experimental procedure.

A-4.2 Reagents

A-4.2.1 *Standard Sulphate Solution*

1ml equivalent to 1 mg H_2SO_4. Transfer 50 ml of this stock solution to a 250 ml volumetric flask and dilute to the mark. Five ml of this solution is equivalent to 1 mg H_2SO_4.

A-4.2.2 *Conditioning Reagent*

Mix 50 ml of glycerine with a solution containing 30 ml of concentrated hydrochloric acid, 300 ml of water and 75 g of sodium chloride.

A-4.2.3 *Barium Chloride*

Use crystals of barium chloride that pass through 850-micron IS Sieve and retain on 500 micron IS Sieve.

A-4.3 Procedure

Transfer 5, 10, 15, 20, 25 and 30 ml of the standard sulphate solution (5 ml = 1 mg H_2SO_4) from a calibrated burette into separate 100 m1 volumetric flasks. To each flask add 5 ml of the conditioning reagent and dilute to 100 ml with water. Mix well. Add 3 g of solid barium chloride to each flask, and shake for 1 min by inverting each flask once per second, all the barium chloride should dissolve. Allow to stand for 4 min. Prepare blank for reagents with water. Measure the percent transmission (T) on spectrophotometer at 420 nm wavelength adjusting the blank at 100 percent transmission. Obtain log T and plot a calibration curve of mg H_2SO_4 versus log T passing through origin.

Transfer an aliquot of the solution containing 2 to 4 mg of sulphate to a 100 ml volumetric flask. Add 5 ml of the conditioning reagent and dilute to the mark with water. Mix well. Add 3 g of barium chloride crystals and shake for 1 min by inverting the flask once per second. Allow to stand for four minutes. Prepare a reagent blank. Obtain percent transmission (T) on spectrophotometer at 420 nm wavelength adjusting blank at 100 percent transmission. From the log T, obtain the mg of H_2SO_4 from the calibration curve.

A-4.4 Calculation

$$\text{Sulphate, percent by mass} = \frac{M_1 \times 100 \times F\ (0.979\ 5)}{1\ 000 \times M}$$

where

M_1 = mass in mg, of H_2SO_4 obtained from the calibration curve;

F = dilution factor; and

M = mass of the material taken for the test.

A-5 DETERMINATION OF IRON

A-5.1 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-5.2 Apparatus

A-5.2.1 Spectrophotometer suitable for measurement at 522 nm, with accuracy of 1 nm.

A-5.2.2 *Standard Laboratory Glass Apparatus*

A-5.3 Reagents

A-5.3.1 *Distilled Water*

A-5.3.2 *Hydrochloric Acid,* approximately 6 mol/l (1:1 v/v).

A-5.3.3 *Hydrochloric Acid,* approximately 1 mol/l, to 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

A-5.3.4 *Hydroxylammonium Chloride,* dissolve 10 g of hydroxylammonium chloride ($NH_2OH.HCI$) in water and dilute to 100 ml.

A-5.3.5 *Ammonium Acetate Solution, 30* percent (m/v) *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-5.3.5.1 *Standard iron solution A*

Dissolve 0.7022 g of ferrous ammonium sulphate hexahydrate [$FeSO_4NH_4SO_4.6H_2O$] 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-5.3.5.2 *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-5.4 Procedure

A-5.4.1 *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 l-cm cell at 522 nm against reagent blank (containing equal amount of added reagent) and make up the volume to 100 ml.

A-5.4.2 *Blank Test*

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

A-5.5 Preparation of Calibration Graph

Prepare a calibration graph with iron as Fe in l.0 µg/100 ml as abscissa and absorbance as ordinates.

A-5.6 Determination of Iron

Weigh accurately to the nearest 1 mg, an amount of PCl_5 containing 100 to 1000 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 l00 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 l00 ml one-mark volumetric flask. Dilute to around 50 ml (if less), add 2 ml hydrochloric acid and 2 ml of hydroxylammonium 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-5.7 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-6 DETERMINATION OF LEAD (*REFEREE METHOD*)

A-6.1 Principle

An acidified sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (*p*H 10 to 11.5). The quantity of lead present in the sample is determined spcctrophotometrically by measuring the absorbance at 510 nm of the chloroform extract containing the lead dithizonate complex.

A-6.2 Apparatus

A-6.2.1 *Spectrophotometer,* for use at 510 nm with a path length of 1 cm orlonger.

A-6.2.2 *Standard Volumetric Glasswares*

A-6.2.3 *TEF Beaker,* 100 ml for acid gigestion.

A-6.2.4 *Separatory Funnels,* 250 ml and 500 ml.

All glasswares are to be cleaned with 1 : 1 HNO_3, and rinsed thoroughlywith distilled water.

A-6.2.5 *pH meter*

A-6.3 Reagents

A-6.3.1 *Quality of Reagents*

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

A-6.3.2 *Stock Lead Solution*

Dissolve 0.159 9 g lead nitrate [($Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated HNO_3 and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

A-6.3.3 *Standard Lead Solution*

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

Nitric acid — Concentrated (18 N).

Nitric acid — Dilute — 20 percent, *v/v*.

Ammonium hydroxide — Concentrated (14 N).

Ammonium hydroxide — Dilute 10 percent, *v/v* and 1 percent, *v/v.*

Citrate-Cyanide Reducing Solution Dissolve 200 g anhydrous ammonium citrate [$(NH_4)_2$ $HC_6H_5O_7$] 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH_4OH.

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

Minimum Detection Limit

1.0 µg Pb/10 ml dithizone solution (extract).

A-6.3.4 *Stock Dithizone Solution*

Dissolve 25 mg dithizone in about 50 ml chloroform ($CHCl_3$) taken in a 200 mlbeaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate by giving two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (*v/v*) NH_4OH solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) NH_4OH solution, transfer the $CHCl_3$ layer to another 250 mlseparatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1 : 1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

A-6.3.5 *Working Dithizone Solution*

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

A-6.3.6 *Procedure*

A-6.3.6.1 *Sample digestion*

Digest all samples for lead as per standard digestion procedure using $HNO_3H_2SO_4$ and HNO_3-$HClO_4$.

To 100 ml acidified sample (*p*H = 2) add 20 ml of dilute (20 percent, *v/v*) HNO_3, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30s, allow to stand (to get two separate layers). Discard 1-2 ml $CHCl_3$ layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

A-6.3.6.2 *Calibration curve*

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

A-6.3.6.3 *Calculation*

mg Pb/litre = µg (in 10 ml extract obtained from calibration curve)/Volume of sample (ml)

A-6.4 Determination of Lead (*Alternate Method*)

A-6.4.1 *General*

The colour developed by the material with sodium sulphide solution is compared with that of a control solution.

A-6.4.2 *Apparatus*

Nessler Cylinder — 50-m1 capacity.

A-6.4.3 *Reagents*

A-6.4.3.1 *Ammonium hydroxide* — 5 N.

A-6.4.3.2 *Potassium cyanide solution* — 10 percent.

A-6.4.3.3 *Sodium sulphide solution* — 12 percent.

A-6.4.4 *Standard Lead Solution*

Dissolve 0·160 g of lead nitrate in 90 ml of water to which 1 ml of nitric acid has been added, and dilute to 100 ml. Keep this solution in lead-free stoppered bottle. Further dilute 10 ml of this solution with water to 1 000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-6.5 Procedure

Take 2.0 g of the material in a Nessler cylinder, add 30 ml of ammonium hydroxide and 1 ml of potassium cyanide solution, dilute with water to the mark and add 1 drop of sodium sulphide solution. Carry out a control test using 2 ml of standard lead solution, 5 ml of ammonium hydroxide, 1 ml of potassium cyanide solution, diluting to 50 ml with water and adding 1 drop of sodium sulphide solution.

The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

ANNEX B

(*Clause* 5)

SAMPLING OF PHOSPHOROUS PENTACHLORIDE, TECHNICAL

B-1 GENERAL REQUIREMENTS

For general requirements of sampling, the methods given in IS 8883 may be followed.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In any consignment, all the drums, carbuoys car containers of the same size and of the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or different sizes of containers, the containers belonging to the same batch and same size shall be grouped together and each such group shall constitute a separate lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, tests shall be carried out for each lot separately. For this purpose, the number of containers to be selected from a lot shall be in accordance with Table 2.

Table 2 Scale of Sampling

(*Clause* B-2.2)

Sl No.	Lot Size	Number or Containers/Carbuoys/ Drums to be Selected
(1)	(2)	(3)
i)	Up to 50	3
ii)	51 to 150	4
iii)	151 to 300	5
iv)	301 and above	7

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

B-3 CRITERIA FOR CONFORMITY

B-3.1 The material shall be visually examined for the requirements given in **3.1** of this standard.

B-3.2 From each of the containers selected, adequate material shall be collected for determining the assay percentage.

B-3.3 On the basis of the test results of all the containers, the average (X) and range (R) shall be calculated as follows:

$$\text{Average } (X) = \frac{\text{Sum of the test result}}{\text{Number of tests}}$$

Range (R) = Difference between the maximum and the minimum of the test results.

B-3.4 The lot shall be declared as conforming to the requirements of this standard, if:

$X - 0.6\,R >$ the minimum requirements given in Table 1.

ANNEX C

(*Foreword*)

EXPERTS WHO MADE SIGNIFICANT CONTRIBUTION TO THE DEVELOPMENT OF THIS STANDARD

COMMITTEE COMPOSITION

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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 (15166).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi