

भारतीय मानक मसौदा
फॉस्फोरस पेंटासल्फाइड — विशिष्टि
(IS 10115 का पहला पुनरीक्षण)

Draft Indian Standard
Phosphorous Pentasulphide — Specification
(*First Revision of IS 10115*)

(*Not to be reproduced without the permission of BIS or used as an Indian Standard*)

ICS 71.060.01

Inorganic Chemicals Sectional Committee, CHD 01

Last date of comments: 26th March 2025

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(*Formal clauses will be added later*)

This standard was first published in 1982. In this first revision, instrumental test method for the determination of iron has been incorporated. In addition to this, editorial corrections have been made wherever required. Also, Amendment no. 1 & 2 and reference clause have been incorporated and Packing & Marking clause has been updated.

Phosphorous pentasulphide (P_2S_5) is used as an intermediate in pesticides and oil additives.

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
PHOSPHOROUS PENTASULPHIDE — SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for phosphorous pentasulphide.

2 REFERENCES

The standards given 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 revisions, and parties to agreements based on this Indian Standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

<i>IS No</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 3025 Part 2 : 2019/ISO 11885	Methods of sampling and test (physical and chemical) for water and wastewater Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
Part 65 : 2022	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>)

3 GRADES

The material shall be of the following two grades:

- a) Grade A — for use in oil additives; and
- b) Grade B — for use in pesticides.

4 REQUIREMENTS**4.1 Description**

The material shall be in the form of a fine, free-flowing powder, yellow to pale greenish in colour and free from extraneous in purities.

4.2 Particle Size

The Particle size of the material shall be as agreed to between the purchaser and the supplier.

4.3 The material when tested as prescribed in Annex A, shall also Comply with the requirements given in Table 1. Reference to relevant clauses of Annex A is given in col (5) of the Table 1.

Table 1 Requirements for Phosphorous Pentasulphide

SI No.	Characteristic	Requirement		Method of Test. Ref to Cl No. In Annex A
		Grade A	Grade B	
(1)	(2)	(3)	(4)	(5)
i)	Phosphorus content (as P)	27.2 to 27.7	27.8 to 28.4	A-2
ii)	Sulphuric content (as S)	72.2 to 72.8	71.6 to 72.2	A-3
iii)	Melting point	286 °C to 290 °C	—	A-4
iv)	Iron (as Fe), ppm, <i>Max</i>	100	—	A-5
v)	Reactivity °C/ min, <i>Min</i>	0.8	—	A-6

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in mild steel drums lined with polyethylene film or as agreed to between the purchaser and the supplier.

5.1.1 For Grade A of the material, the container shall also be inerted, using dry nitrogen /carbon dioxide or other inert gas to protect the material from other reaction.

5.2 The container shall be securely closed and shall bear legibly and indelibly the following information and other labelling provisions applicable to the material.

- 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;
- e) Batch number; and
- f) Highly toxic by inhalation, strong irritant, dangerous fire risk, ignite by friction, Contact with water or acids liberates hydrogen sulphide gas.

5.2.1 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.

6 SAMPLING

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

ANNEX A

(Clause 4.3)

METHODS OF TEST

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 PHOSPHOROUS CONTENT

A-2.1 Outline of the method

Phosphorous in phosphorous pentasulphide is oxidized into PO_4^{3-} by oxidation using sulphuric acid invert aqua regia and bromine. The PO_4^{3-} ions are precipitated as quinoline phosphomolybdate, filtered, dissolved in excess of standard alkali and back titrated with standard acid using phenolphthalein as indicator and phosphorus is calculated volumetrically.

A-2.2 Reagents

A-2.2.1 Concentrated Nitric Acid — *see* IS 264

A-2.2.2 Concentrated Hydrochloric Acid — *see* IS 265

A-2.2.3 Bromine

A-2.2.4 Dilute Sulphuric Acid

Relative density 1.21. Slowly add 200 ml of cooled concentrated sulphuric acid to about 700 ml of water contained in a 1 000 ml volumetric flask, Stir and make up to 1 000 ml with water.

A-2.2.5 Citric Molybdate Solution

Weigh 50 g of molybdate anhydride (MoO_3) in a 500 ml beaker. Add 200 ml of water and then 11 g of sodium hydroxide pellets. Heat the beaker until molybdic anhydride dissolves. Dissolve 60 g of citric acid crystals in 250 ml of water in a one litre beaker and add 140 ml of hydrochloric acid. Pour the molybdic solution into the citric acid solution, stirring vigorously all the time. Cool the solution and filter it into a one-liter volumetric flask. Make up to one litre with water. The solution, may be green or blue, depending on exposure to light. If necessary, add a dilute solution of potassium bromate (0.5 to 1 percent) drop wise until green colour disappears. Keep solution in a dark place in a well stoppered bottle, made of polyethylene.

A-2.2.6 Quinoline Solution

Dilute 60 ml of concentrated hydrochloric acid in a beaker with 300 ml to 400 ml of water and heat to 70 °C to 80 °C. Slowly add 25 ml of quinoline solution, stirring all the while. The quinoline dissolves, cool the solution, dilute it with water to one litre and filter. Keep solution in a polyethylene bottle.

A-2.2.7 Standard Sodium Hydroxide — 0.1 N

A-2.2.8 Standard Hydrochloric Acid — 0.1 N

A-2.2.9 Mixed indicator Solution

Two volumes of 0.1 percent of phenolphthalein in 60 percent rectified spirit, three volumes of 0.1 percent thymol blue (0.1 g indicator, 22 ml of 0.1 N NaOH and diluted to 100 ml, 50 ml methylated spirit diluted to 100 ml.

A-2.3 Procedure

A.2.3.1 Grind and weigh accurately about 1 g of the material. Transfer to a 500 ml flask with a ground glass neck, it reflux condenser to it and add 20 ml of concentrated hydrochloric followed by 60 ml concentrated nitric acid via the condenser. Allow reaction to take place under a fume cupboard for about 30 min, and then heat on a steam bath. Add

5 ml of bromine, and reheat in steam bath until bromine disappears. Cool and rinse condenser with water. Cover the flask with small funnel and boil on a low flame till a final volume of few ml is attained, Add water and transfer solution totally into the 1 000 ml volumetric flask. Make up to one litre and filter off any insoluble residue through a dry fluted filter into a dry 1 000 ml flask, discarding the first portion of the filtered liquid.

A-2.3.2 Using volumetric pipette, draw off 25 ml of the solution and pour it into a 500 ml flask. Add 10 ml of dilute sulphuric acid and boil for 5 min. Dilute the solution with water to 150 ml and add 25 ml of citric molybdate solution, heat to boiling and add quinoline solution dropwise from a burette until about one ml has been added. Boil again and to the gently boiling solution add the reagent, a few ml at a time with stirring until 12.5 ml in all have been introduced. A coarsely crystalline precipitate is thus obtained which can easily be filtered.

A-2.3.3 Filter under suction through a thick pad of filter pulp prepared on a perforated disc in a funnel. Wash the precipitate and the flask with water till free from acid (About 10 ml of the washing and 4 drops of the indicator should show colour change from violet to yellow with 1 drop of 0.1 N sodium hydroxide solution). Transfer the pad of filter paper back with the precipitate to the original flask. Add 50 ml to 60 ml of water and then add 0.1 N sodium hydroxide solution with stirring in multiples of 25 ml from a pipette till the precipitate dissolves completely. Add 5 to 6 drops of the indicator solution and titrate the excess of alkali with standard hydrochloric acid till violet colour changes to yellow. The end point is sharp.

A-2.3.4 Run a blank determination on the same quantity of sodium hydroxide solution which was added in the test and titrate with standard hydrochloric acid using 5 to 6 drops of indicator solution.

A-2.4 Calculation

$$\text{Phosphorous (as P) percent by mass} = \frac{(V_1 - V_2) N \times 4.768}{M}$$

where

- V_1 = volume in ml, of standard hydrochloric acid required for the blank;
- V_2 = volume in ml, of standard hydrochloric acid required for the sample;
- N = normality of standard hydrochloric acid; and
- M = mass in g, of the material taken for the test.

A-3 SULPHUR CONTENT

A-3.1 Outline of the Method

Phosphorous pentasulphide is first dissolved in sodium hydroxide and then oxidized first with hydrogen peroxide and then further with bromine to phosphoric acid and sulphuric acid. The sulphate ions are precipitated as barium sulphate and estimated as sulphur.

A-3.2 Reagents

A-3.2.1 *Barium Chloride Solution* ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) — 10 percent (m/v)

A-3.2.2 *Hydrogen Peroxide* — 30 percent (v/v)

A-3.2.3 *Sodium Hydroxide Solution* — 10 percent (m/v)

A-3.2.4 *Silver Nitrate Solution*

A-3.2.5 *Bromine*

A-3.2.6 *Hydrochloric Acid*

A-3.3 Procedure

A-3.3.1 Weigh accurately about 1 g of the material and transfer it to a flask containing 100 ml of sodium hydroxide solution. Warm gently to about 50 °C and stir until the material is completely dissolved. Cool the solution and then add 20 ml of hydrogen peroxide cautiously in small increments. Add one to two boiling chips or glass beads and

carefully boil the solution gently for 10 min. Cool and add another 20 ml of hydrogen peroxide cautiously in small increments. Again boil the solution gently for 10 to 20 min.

A-3.3.2 Dilute the solution to about 100 ml with water and cool. Add 5 ml of bromine, cool and add hydrochloric acid drop wise (*see* Note) until no bromine evolves; then add about 3 ml in excess. Boil until the evolution of bromine ceases. Cool the sample to room temperature and transfer quantitatively to a 250 ml volumetric flask, avoiding the transfer of the boiling chips. Dilute to the mark with water and mix. Pipette out 10 ml of this solution in to a 400 ml tall beaker. Dilute to about 200 ml. Heat the solution to boiling, add drop wise from a burette 10 ml of barium chloride solution, at the rate of about 1 ml/min. Stir the solution constant by during the addition. Allow the precipitate to settle for a minute or two, then test the supernatant liquid for complete precipitation by adding a few drops of barium chloride solution if a precipitate is formed, add more barium chloride solution, repeat this operation again, until an excess of barium chloride is present.

A-3.3.3 Cover the solution and keep it hot, on a water bath, in order to allow time for complete precipitation. Test the clear supernatant liquid with a few drops of barium chloride solution for complete precipitation. Filter through ash less filter paper (Whatman 42 or equivalent) and wash the precipitate with small portions of hot water jet. Continue the washing until the washings are free from chloride ion (5 ml of the wash solution gives no opalescence with a drop or two of silver nitrate solution). Eight or ten washing are usually required.

A-3.3.4 Place the paper and precipitate in a porcelain /silica crucible, previously ignited to redness, cooled in a desiccator and weighed, dry the paper by placing the loosely covered crucible upon a triangle several centimeters above a small flame. Gradually char the paper by increasing heat and finally ignite in a muffle furnace at 775 °C to 800 °C for 10 min. Cool in a desiccator and weigh.

Note — Bromine fumes are released when this solution is boiled, this addition of hydrochloric acid and boiling must be done in fuming cupboard as bromine fumes are very toxic.

A-3.4 Calculation

$$\text{Sulphur, percent by mass} = \frac{M_1 \times 343.4}{M}$$

where

M_1 = mass in g, of the residue; and

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

A-4 DETERMINATION OF MELTING POINT

A-4.1 Apparatus — Fisher-John's melting point apparatus. Cover glasses for microscope slides, 18 mm in diameter.

A-4.2 Procedure

A very small quantity of the finely pulverized maternal is placed between two cover glasses. The cover glasses are placed on the heating stage of the apparatus. The heating current is turned on and the sample is observed in the magnifier the melting point is observed when small droplets appear between the cover glasses. The temperature can be raised rapidly to within 10 of the melting point, but it should be raised slowly after that.

A-4.3 A second cover glass is unnecessary when the melting point of fats, greases and waxes is determined. Because of the uncertainty of there melting points a tiny spike is formed on the cover glass and the melting point is obtained when the tip bends or begins to flow.

A-5 DETERMINATION OF IRON

A-5.1 General

Three methods are prescribed for determining iron, namely, Method A, ICP-OES method as prescribed at **A-7** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

A-5.2 Method A

A-5.2.1 Apparatus

A-5.2.1.1 Electrophotometer — equipped with a blue filter at 440° mμ or a spectrophotometer.

A-5.2.1.2 Graduated cylinders —100 ml

A-5.2.1.3 Filter paper — Munktell's No. 00 or equivalent

A-5.2.1.4 Porcelain crucible

A-5.2.1.5 Muffle furnace

A-5.2.1.6 Volumetric flask — 100 ml capacity

A-5.2.1.7 Pipette — 10 ml capacity

A-5.2.1.8 Burette —50 ml capacity

A-5.2.2 Reagents

A-5.2.2.1 Ammonium thiocyanate

A-5.2.2.2 Hydrochloric acid — see IS 265

A-5.2.2.3 Potassium persulphate — solid

A-5.2.2.4 Nitric acid — see IS 264

A-5.2.2.5 Potassium bisulphate or pyrosulphate — solid

A-5.2.2.6 Standard iron wire

A-5.2.2.7 Ammonium thiocyanate solution — 1 N

A-5.2.2.8 Dilute hydrochloric acid — 1:1 (v/v)

A-5.2.3 Procedure

A sample containing 0.05 mg to 0.1 mg iron is placed in a 250 ml beaker to which has been added about 50 ml water, 5 ml of HCl and 0.1 g of potassium Persulphate. Solution may be accomplished with heat but the resultant solution must be colorless for organic sample, the material is weighed into a porcelain crucible, ignited and fired in a muffle furnace until no carbon remains. The ash is treated with about one gram of potassium bisulphate and heated over a burner until the ash is dissolved. The crucible is cooled and placed in a 250 ml beaker containing 50 ml of water and 5 ml of hydrochloric acid is heated to dissolve the melt.

A-5.2.3.1 The contents of the beaker are brought to a boil and filtered through Munktell's No. 00 paper into a 100 ml graduated cylinder. The paper is washed twice with hot water but care must be taken to avoid a total of more than 80 ml in the graduate. The solution is cooled and treated with 10 ml of 1 N ammonium thiocyanate solution. The volume is made up to 100 ml with water and mixed thoroughly by transferring the solution to a 250 ml beaker.

A-5.2.3.2 The transmission of light through the solution is measured in percent by use of the electro photometer equipped with a blue filter (440 mμ) or a spectrophotometer with the wave length set at 440 mμ. The transmission of light through distilled water is considered 100 percent transmission. The amount of iron present in mg is taken from a curve prepared in the manner described below.

A-5.2.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{\text{mg Fe (obtained from curve)}}{\text{Sample mass taken for test} \times 10}$$

A-5.2.5 Preparation of Standard Calibration Curve

Into eight 250 ml beakers are placed varying amounts of the iron solution (0.010 mg Fe per ml) i.e. 1, 2, 4, 6, 8, 10, 14, 20 ml corresponding respectively to 0.01, 0.02, 0.04, 0.06, 0.08, 0.10, 0.14 and 0.20 mg Fe. To each of these solutions is added about 25 ml to 30 ml water, 10 ml, 1 : 1 hydrochloric acid, 10 ml in NH_4CNS and about 0.1 g potassium persulphate. The solutions are mixed well and diluted to 100 ml by using a 100 ml graduated cylinder. The transmission of light of each solution is measured with 100 percent transmission assumed on pure water. A curve is plotted, the abscissa representing concentration in mg, the ordinate representing the percent transmission of light.

Note 1 — The orange colour of the iron thiocyanate complex is unstable and the light transmission must be measured within ten minutes after the development of the colour.

Note 2 — When the colour is developed in the 100 ml graduated cylinder the total acidity should be equal to 5 ml of HCl or 10 ml of 1:1 HCl if large amounts of phosphate or other buffering agents are present, the acidity must be increased accordingly, but the results will be nevertheless suspect.

Note 3 — In samples containing barium and/or lead, the sulphate and per sulphate iron should be avoided, with a few drops of nitric acid being used as an oxidizing agent.

Note 4 — Since potassium bisulphate contains an appreciable amount of iron, the iron should be determined on a blank containing all the chemicals used in determination. This blank should be subtracted from the total percent Iron.

A-6 REACTIVITY

A-6.1 Apparatus

Dewar flask, wide-mouth, one pint size (Fisher Scientific 10-195 is satisfactory).

A-6.1.1 Stirrer motor, variable speed, capable of operating at 120 rpm, equipped with a two-bladed glass stirrer having the following dimension. The two blades shall have a pitch of 45° . Each blade shall be 13 mm long and the diameter inscribed by the rotating blades shall be not more than 30 mm.

A-6.1.2 *Stopper for Dewar flask* — equipped with glass-lined holes for stirrer and thermometer, and vented for escape of gas.

A-6.1.3 Thermometer, standard laboratory, 650 mm, graduated to 101°C , in 0.1°C divisions.

A-6.1.4 *Mortar and pestle*

A-6.1.5 *IS Sieve* — 300 micron

A-6.1.6 *Graph paper* — $25.4 + 25.4$ per cm.

A-6.2 Reagent — Isopropyl alcohol, technical grade, 99 percent.

A-6.3 Procedure

The stirring rate of the motor is adjusted to 120 rpm. The stirrer and thermometer are inserted through the sleeves of the stopper and positioned 1.27 cm from the bottom of the vessel, making sure that the stirrer does not hit either the slide of the flask or the thermometer. Using a graduate, 100 ml of isopropyl alcohol, adjusted to $(30 \pm 1)^\circ\text{C}$, is added to the reaction flask. Agitation is started and (36 ± 0.1) g of phosphorus pentasulphide, ground and passed through a 60 mesh Sieve, is added to the reaction flask. The reaction is allowed to proceed until temperature rise increments are constant. This is accomplished by plotting a graph, time in minutes versus temperature, recording the reaction temperature every four minutes for one hour.

The end-point is taken as either the maximum temperature reached by the reaction mixture or the point at which the rate of temperature change becomes constant, the point beyond which the curve becomes a straight line.

A-6.4 Calculation

$$\text{Reactivity Rate, } = \text{ }^\circ\text{C/minute} = \frac{\text{Maximum temperature} - \text{Initial temperature}}{\text{Time required for temperature rise}}$$

Reactivity Rate is reported to the nearest 0.1°C/min .

A-7 DETERMINATION OF IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**A-7.1 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.

A-7.2 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 A-7.2 and A-7.4)

Sl No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (μg)	Axial Viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	-	-	-

A-7.3 Reagents and Solutions**A-7.3.1. Nitric Acid (65 percent) Suprapure****A-7.3.2 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 $\mu\text{g}/\text{ml}$, 100 $\mu\text{g}/\text{ml}$ or 1 000 $\mu\text{g}/\text{ml}$ of arsenic, calcium, lead, Iron and magnesium, in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-7.3.3 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 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 $\mu\text{g}/\text{ml}$, 0.3 $\mu\text{g}/\text{ml}$ and 0.5 $\mu\text{g}/\text{ml}$ solution of respective elements under reference.

A-7.3.4 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.

A-7.3.5 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.

A-7.4 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.

A-7.5 Procedure

A-7.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-7.3.5). 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.

A-7.5.2 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 lead (and/or Iron, calcium, arsenic and magnesium) 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.

A-7.6 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 B

(Clause 6.1)

SAMPLE OF PHOSPHORUS PENTASULPHIDE

B-1 SAMPLING

B-1.1 General Precautions — In drawing, preparing, storing and handling samples, the following precautions shall be observed.

B-1.1.1 Samples shall not be taken in a place exposed to the weather.

B-1.1.2 The sampling instruments shall be clean and dry

B-1.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

B-1.1.4 The samples shall be placed in suitable clean, dry airtight, glass or other suitable container on which the material has no action.

B-1.1.5 The sample containers shall be of such a size that they are almost, but not completely, filled with the sample

B-1.1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

B-1.1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-1.2 Scale of Sampling

B-1.2.1 Lot

All the containers in the consignment of the material drawn from a single batch of manufacture shall constitute a lot.

B-1.2.2 For ascertaining the conformity of the lot to the requirements of the specification, samples shall be tested from each lot separately

B-1.2.3 The number of containers to be sampled from each lot shall depend on the size of the lot and shall be in accordance with Table 3.

Table 3 Scale of Sampling

(Clause B-1.2.3)

SI No.	Lot Size	No. of Containers To Be Selected
(1)	N (2)	n (3)
i)	Up to 25	3
ii)	25 to 50	4
iii)	51 to 100	5
iv)	101 to 200	6
v)	201 and above	8

B-1.2.3.1 The sample containers shall be selected at random. In order to ensure the randomness of selection, procedure given in IS 4905 may be followed.

B-1.3 Test Samples and Referee Sample

B-1.3.1 Draw with a suitable sampling instrument approximately 20 g of material from different parts of the container, divide the material so obtained in three equal parts. Each part so obtained shall constitute an Individual sample representing the container and shall be transferred immediately to thoroughly dried bottles which are sealed airtight with glass stoppers These shall be labelled with full particulars of sampling given in **B-1.1.6** One set of test samples shall be sent to the purchaser, another to the supplier and the third kept for the referee.

B-2 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-2.1 Test for all requirements shall be conducted on each of the individual samples.

B-2.2 The lot shall be declared as conforming to a requirement of this specification, if all the samples tested satisfy the relevant requirement.