

भारतीय मानक बोर्ड
भारतीय मानक - 333 - पोटैशियम परमैंगेनेट
) 333 का तीसरा संशोधन (

Draft Indian Standard
POTASSIUM PERMANGANATE — SPECIFICATION
(Third Revision of IS 333)

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

ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01 | **Last date of comments: 17th December 2022**

FOREWORD

This Indian Standard (Third 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 first published in 1951 and was subsequently revised in 1969 and 1980. In this revision, instrumental test methods for the determination of arsenic, chlorides and sulphates have been added as alternate test methods. Also, Packing and Marking clause has been updated. Further, Reference clause has been incorporated.

Potassium permanganate is used as an oxidizing agent mainly in the production of saccharin and benzoic acid; bleaching and decolourising ethereal oils, waxes and fatty materials; leather tanning; staining and preservation of wood; and as a deodorant and disinfectant.

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.

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for potassium permanganate.

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.

<i>IS No.</i>	<i>Title</i>
264:2005	Nitric acid — Specification (<i>third revision</i>)
265:2021	Hydrochloric acid — Specification (<i>fourth revision</i>)
266:1993	Sulphuric acid — Specification (<i>third revision</i>)
1070:1992	Reagent grade water — Specification (<i>third revision</i>)
3025(Part2) :2019/ISO 11885	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP - OES) (<i>first revision</i>)
4161: 1967	Specification for Nessler cylinders

3 GRADES

3.1 The material shall be of the following three grades:

3.1.1 Technical,

3.1.2 Pure, and

3.1.3 Analytical reagent (AR).

4 REQUIREMENTS

4.1 Description

4.1.1 Potassium permanganate of technical grade shall be in the form of dark purple powder or irregular crystals or conglomerates.

4.1.2 Potassium permanganate of pure and analytical reagent grades shall be in the form of dark purple crystals having a metallic lustre.

4.2 The material shall also comply with the requirements laid down in Table 1, when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of the Annex are given in col 6 of the table.

Table 1 Requirements for Potassium Permanganate

Sl. No.	Characteristic	Requirement			Method of Test (Ref to Cl No. in Annex A)
		Technical Grade	Pure Grade	AR Grade	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Potassium permanganate (as KMnO_4) content, percent by mass, <i>Min</i>	98	99	99.5	A-2
ii)	Insoluble matter, percent by mass, <i>Max</i>	1.0	0.3	0.05	A-3
iii)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.1	0.01	0.005	A-4 or A-9
iv)	Sulphates (as SO_4), percent by mass, <i>Max</i>	0.2	0.05	0.005	A-5 or A-9
v)	Nitrogen compounds (as N), percent by mass, <i>Max</i>	—	0.3	0.003	A-6
vi)	Arsenic (as As_2O_3), percent by mass, <i>Max</i>	—	0.01	0.0001	A-7 or A-10
vii)	Loss on heating at 110°C , percent by mass, <i>Max</i>	1.0	0.5	—	A-8

5 PACKAGING AND MARKING

5.1 Packaging

The material shall be packed in closed containers as agreed to between the purchaser and the supplier.

5.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

- Name and grade of the material;
- Name of the manufacturer and his recognized trade mark, if any;
- Gross and net mass;
- Date of manufacture and;
- Batch number

5.2.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1.1 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 there under, and the products may be marked with the standard mark.

6 SAMPLING

6.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

ANNEX A
(Clause 4.2)

METHODS OF TEST FOR POTASSIUM PERMANGANATE

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be employed.

NOTE – ‘ Pure chemicals ’ shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2 DETERMINATION OF POTASSIUM PERMANGANATE CONTENT

A-2.1 Outline of the Method

Potassium permanganate content is determined by titration against standard sodium oxalate solution.

A-2.2 Preparation of Solution

Dry about 2 g of the material at about 110°C to constant mass. Transfer 0.8 g of the dried material, accurately weighed, into a 250 ml volumetric flask. Dissolve in water and make up the volume to the mark.

A-2.3 Reagents

A-2.3.1 *Standard Sodium Oxalate Solution* — 0.1 N. Freshly prepared.

A-2.3.2 *Dilute Sulphuric Acid* — 5 N.

A-2.4 Procedure

Take 25 ml of standard sodium oxalate solution and mix with 15 ml of sulphuric acid and 25 ml of water. Heat the solution to about 60°C and titrate with the prepared solution till a pink colour, persistent for 1 min, is obtained with one drop of it. Record the volume in millilitres of the prepared solution required for the titration.

A-2.5 Calculation

Calculate the mass of potassium permanganate (KMnO_4) on the basis that 1 ml of normal sodium oxalate solution is equivalent to 0.03160 g of potassium permanganate, and express the result as percentage of the mass of the material taken for the test.

A-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure

Weigh accurately about 2 g of the material and dissolve in 100 ml of hot water (at about 50°C). Filter through a Gooch crucible or a sintered glass crucible No. G4. Wash until washings are colourless and dry at about 105°C till constant mass is obtained. For AR grade, use 20 g of the material.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = 100 \frac{M_1}{M_2}$$

where,

M_1 = mass in g of the dried residue, and

M_2 = mass in g of the material taken for the test.

A-4 DETERMINATION OF CHLORIDES

A-4.1 Outline of the Method

Chlorides are determined by comparing the opalescence produced by the material with silver nitrate solution against that produced by a standard chloride solution.

A-4.2 Apparatus

A-4.2.1 Nessler Cylinders — 50 ml capacity (*see* IS 4161).

A-4.3 Reagents

A-4.3.1 Concentrated Nitric Acid — *see* IS 264

A-4.3.2 Silver Nitrate Solution — 5 percent (*m/v*).

A-4.3.3 Standard Chloride Solution

Dissolve 1.649 g of sodium chloride in water and make up the volume to 1000 ml. Pipette out 10 ml of the solution, dilute with water and make up the volume to 100 ml. One millilitre of this solution contains 0.1 mg of chlorides (as Cl).

A-4.3.4 Hydrogen Peroxide — 35 percent (*m/m*).

A-4.4 Procedure

Dissolve 1.0 g of the material in water. Transfer to a Nessler cylinder and add 1 ml of concentrated nitric acid, 1 ml of silver nitrate solution and enough quantity of hydrogen peroxide to completely decolorize the permanganate colour. When the solution is completely decolorized, dilute it to the mark with water. Carry out a control test in the other Nessler cylinder using 10 ml of standard chloride solution in case of technical grade, 1 ml in case of pure grade and 0.5 ml in case of analytical reagent grade of the material, and the same quantities of other reagents in the same total volume of the reaction mixture. Stir both the solutions with glass rods and compare the opalescence produced in the two cylinders after 5 min.

A-4.4.1 The limit shall be taken as not having been exceeded if the opalescence produced in the test with the material is not greater than that produced in the control test.

A-4 Alternative Method

Chlorides may alternatively be determined by instrumental test method as prescribed in **A-9**.

A-5 SULPHATES

A-5.1 For Technical Grade

A-5.1.1 Outline of the Method — Sulphates are determined by precipitating with barium chloride solution.

A-5.1.2 Reagents**A-5.1.2.1 Concentrated hydrochloric acid** — see IS 265**A-5.1.2.2 Hydrogen peroxide** — 35 percent (*m/m*).**A-5.1.2.3 Dilute hydrochloric acid** — approximately 5 N.**A-5.1.2.4 Barium chloride solution** — approximately 10 percent (*m/v*).**A-5.1.3 Procedure**

Weigh accurately about 10 g of the material and dissolve in 100 ml of water. Add this solution slowly to a mixture consisting of 14 ml of concentrated hydrochloric acid, 30 ml of water and 5 ml of hydrogen peroxide. Evaporate the solution to dryness. Dissolve the residue in 10 ml of dilute hydrochloric acid and dilute to 200 ml with water. Heat the solution to boiling and add, drop by drop, 10 ml of barium chloride solution. Boil the solution for 15 min and then allow to stand for 4 h. Filter through a tared Gooch crucible or a sintered glass crucible No. G4. Wash the precipitate thoroughly with hot water till it is free from chlorides and dry to constant mass at 105 to 110°C.

A-5.1.4 Calculation

$$\text{Sulphates (as } SO_4), \text{ percent by mass} = 41.15 \frac{A}{M}$$

where,

A = mass in g of the precipitate, and

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

A-5.2 For Pure and Analytical Reagent Grades**A-5.2.1 Outline of the Method**

Sulphates are determined by comparing the turbidity produced by the material with barium chloride solution against that produced by a standard sulphate solution.

A-5.2.2 Apparatus**A-5.2.2.1 Nessler cylinders** — 50 ml capacity (*see* IS 4161)**A-5.2.3 Reagents****A-5.2.3.1 Dilute hydrochloric acid** — approximately 5 N.**A-5.2.3.2 Standard sulphate solution**

Dissolve 0.1814 g of potassium sulphate in water and make up the volume to 1000 ml. Take 10 ml of this solution and dilute it further to 100 ml. One millilitre of the final solution is equivalent to 0.01 mg of sulphate (as SO_4).

A-5.2.3.3 Barium chloride solution — approximately 10 percent (*m/v*)**A-5.2.3.4 Hydrogen peroxide** — 35 percent (*m/v*)

A-5.2.4 Procedure

Weigh accurately 0.2 g of the material and dissolve in 25 ml of hot water. Add this solution slowly to a mixture consisting of 7 ml of dilute hydrochloric acid, 15 ml of water and 5 ml of hydrogen peroxide. Evaporate the solution to dryness. Dissolve the residue in 1 ml of dilute hydrochloric acid and 10 ml of warm water and filter if necessary. Transfer the solution to a Nessler cylinder and add 2 ml of barium chloride solution, stir the mixture and allow to stand for 20 min. Carry out a control test in the other Nessler cylinder using 10 ml of standard sulphate solution in case of pure grade and 1 ml in case of analytical reagent grade of the material, and the same quantities of other reagents, finally diluting to the mark.

A-5.2.4.1 The limit shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

A-4 Alternative Method

Sulphates may alternatively be determined by instrumental test method as prescribed in **A-9**.

A-6 NITROGEN COMPOUNDS (as N)**A-6.1 Outline of the Method**

Nitrogen compounds are determined with the help of Nessler solution which reacts with ammonia to form a reddish brown colloidal suspension.

A-6.2 Reagents

A-6.2.1 Concentrated Sulphuric Acid — see IS 266

A-6.2.2 Oxalic Acid — solid.

A-6.2.3 Dilute Hydrochloric Acid — 0.1 N.

A-6.2.4 Sodium Hydroxide Solution — 10 percent (m/v)

A-6.2.5 Aluminium Wire

A-6.2.6 Nessler Solution

Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water and add to it slowly with stirring saturated mercuric chloride solution until a slight permanent precipitate is formed. Add 30 g of potassium hydroxide and when it has dissolved, add 1 ml more of mercuric chloride solution, and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

A-6.3 Procedure

Dissolve 1.0 g of the material in 100 ml of water in a flask and add 1 ml of concentrated sulphuric acid and 2.5 mg of oxalic acid. When the solution is clear, connect the flask through a spray trap to a condenser, the end of which is beneath the surface of 10 ml of dilute hydrochloric acid. Add to the flask 40 ml of freshly boiled sodium hydroxide solution and 0.5 g of aluminium wire in small pieces. Allow to stand for 1 h, distil about 60 ml and dilute the distillate to 100 ml. To 25 ml of the distillate add 1 ml of sodium hydroxide solution, dilute to 50 ml and add 2 ml of Nessler solution. Carry out a control test in the similar way using 12 mg of ammonium chloride in case of pure grade of the material, and 0.04 mg in case of analytical reagent grade of the material in place of the material.

A-6.3.1 The limit shall be taken as not having been exceeded if the not deeper than that colour produced in the test with the material is produced in the control test.

A-7 ARSENIC (as As₂O₃)

A-7.1 Reagents

A-7.1.1 *Concentrated Hydrochloric Acid* — see IS 265

A-7.1.2 *Stannous Chloride Solution* — 30 percent (m/v)

A-7.2 Procedure

A-7.2.1 *Preparation of Solution*

Weigh accurately 1.0 g of the material and add a cooled mixture of 10 ml of concentrated hydrochloric acid and 10 ml of water. When the first reaction has subsided, heat to remove chloride. Cool, remove the last traces of chloride with a few drops of stannous chloride solution and add 20 ml of water.

A-7.2.2 Carryout the test for arsenic as prescribed in **5.1** of IS **2088** using for comparison a stain obtained with 0.1 mg of arsenic trioxide (as As₂O₃) in case of pure grade of the material and 0.001 mg in case of analytical reagent grade of the material.

A-7.2.2.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length of the stain as well as the intensity of its colour produced in the test with the material is not greater than that produced in the control test.

A-4 Alternative Method

Arsenic may alternatively be determined by instrumental test method as prescribed in **A-10**.

A-8 LOSS ON HEATING AT 110°C

A-8.1 Procedure

Weigh accurately about 10 g of the material ground to powder into a tared shallow dish and dry to constant mass 110 ± 2°C.

A-8.2 Calculation

$$\text{Loss on heating at 110°C} = 100 \frac{M_1}{M}$$

where,

M_1 = loss in mass in g, and

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

A-9 ION CHROMATOGRAPHY FOR CHLORIDES AND SULPHATES

A-9.1 Principle

Ion Chromatography is an innovative method for the determination of ions. The technique is used for the analysis of chlorides and sulphates. The technique separates ions and polar molecules based on their affinity to ion exchanger. When the method is employed for the determination of the anions, the identification should be made by using a matrix covering the ions of interest. In cation exchange chromatography, the stationary phase is functionalized with anions. These anions will attach cations towards it. These surface bound molecules/ionic species can then be removed by using a suitable eluent containing substituted ions to replace them or they can be removed by changing the *pH* of the column. Similarly, in anion exchange chromatography, the stationary phase is cationic in nature. These cations will then separate the anions.

Conductivity detector is generally used in this method. In case of suppressor ion exchange chromatography, analyte ions are separated on the ion exchange column and these ions together with the eluent move to the matrix suppressor. The eluent conductivity is lowered in the suppressor and the sample ion conductivity is increased leading to the large increase in signal to noise ratio.

A-9.2 Equipment

A-9.2.1 Anion guard column — a protector of the separator column.

A-9.2.2 Anion Separator column — suitable for selective separation of ions under analysis.

A-9.2.3 Anion Suppressor device — Anion micromembrane suppressor is used to analyse the data
Detector: Conductivity detector.

A-9.2.4 Software — Software suitable for control of various operating parameters, receiving inputs and analysis of all data.

Sample loop of 100 μl , 200 μl , 500 μl or 1000 μl be used to determine ionic concentration as per instrument manual and practice.

A-9.3 Reagents and Standards

A-9.3.1 Glass or polyethylene sample bottles.

A-9.3.2 Distilled water or deionized water free from the anions of interest.

A-9.3.3 Eluent — 1.7 mM of sodium bicarbonate and 1.8 mM of sodium carbonate solution is used.

For preparation of these solution, 0.2856 g of sodium bicarbonate and 0.3816 g of sodium carbonate is dissolved in 2 l of water.

A-9.3.4 Micromembrane suppressor solution (0.025 N of sulphuric acid) — Dilute 2.8 ml of concentrated Sulphuric acid in 4 l of water

A-9.4 Standard solutions

A- 9.4.1 Chloride — Dissolve NaCl, 1.6485 g in 1 l of reagent water

A-9.4.2 Sulphate — Dissolve 1.81 g of potassium sulphate in 1 l of reagent water

A-9.5 Calibration and Standardization

For each analyte of interest, prepare calibration standards at three concentration levels and a blank by adding measured stock standards and diluting with reagent water. If the concentration of the sample exceeds the calibration range, the sample may be diluted. Using 0.1-1.0 ml injections of each calibration standard, tabulate area responses or peak height against the concentration. Use these results to prepare calibration curve. Record the retention time during the procedure.

A-9.6 Procedure

Dissolve between 1 to 5 g sample in 25 ml reagent grade water in PTTE/HDPE beaker and use this solution for analysis. Inject a well-mixed sample (0.1-1.0 ml) and flush it through an injection loop using each new sample. Use the loop of same size for the standards and samples. Record the peak in size and area units. An automated constant volume injection system may preferably be used. The width of peak for retention time of ions should be same for sample and standard and deviation of retention force shall not exceed ± 10 percent of RT of calibration. Dilute the sample with the help of reagent water if the response for the peak exceeds the working range of the system for analysis. If required, spike the sample with an appropriate amount of standard and reanalyze in case of absence of distinct resolution. Retention time is inversely proportional to concentration. For clear resolution, the sample can further be diluted. The dilution should be made to an extent till there is no deviation from the method.

A-9.7 Data analysis and Calculations

Prepare a calibration curve for each analyte by plotting instrument response against concentration. Compare the sample response with the standard curve and compute sample concentration. Multiply the value by appropriate dilution factor.

Report results in mg/l or by suitably modifying into percentage. Only report those values that fall within the range of lowest and highest calibration standards

A-10 DETERMINATION OF ARSENIC BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

A-10.1 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-10.2 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.

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

Sl. No.	Element	Wavelength (nm)	Approximately Achievable limits		Interfering Elements
			Radial viewing (μg)	Axial viewing (μg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti

A-10.3 Reagents and Solutions**A-10.3.1 Nitric acid (65 percent) Suprapure****A-10.3.2 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 1000 $\mu\text{g}/\text{ml}$ of arsenic in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

A-10.3.3 Standard solution

Pipette out 5 ml from 100 $\mu\text{g}/\text{ml}$ standard stock solution into a 100 ml volumetric flask & 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.

A-10.3.4 Sample preparation

Weigh about 2.5 g polyphosphoric acid 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-10.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (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-10.4 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-10.5 Procedure

A-10.5.1 Calibration

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-10.5.2). 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-10.5.2 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 arsenic 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-10.6 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)

SAMPLING AND CRITERIA FOR CONFORMITY OF POTASSIUM PERMANGANATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing samples, the following precautions and directions shall be observed.

B-1.2 Samples shall not be taken in an exposed place.

B-1.3 The sampling implement shall be clean and dry when used.

B-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling implement and the containers for samples from adventitious contaminations.

B-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.6 The samples shall be placed in suitable, clean, dry and airtight glass containers which shall be sealed airtight after filling and marked with full details of sampling.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In a single consignment of the material all the containers of the same size and containing the same grade of material and drawn from the same batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different types or sizes of containers, those belonging to the same batch, type and size shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 3.

Table 3 Scale of Sampling

lot size N (1)	Sample Size n (2)
up to 25	3
26 to 50	4
51 to 100	5
101 and above	7

B-2.1.2 The containers to be selected for sampling shall be chosen at random and in order to ensure randomness of selection IS 4905 may be followed.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 From each of the containers selected, draw a representative portion of potassium permanganate approximately 75 g in mass.

B-3.2 Out of these portions, a small but equal quantity of the material shall be taken and mixed thoroughly by suitable means to form a composite sample of mass about 150 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and third to be used as a referee sample.

B-3.3 From the remaining portion of potassium permanganate from each container, about 25 g of the material shall be drawn and divided into three equal parts which shall then be transferred to three separate bottles with full identification particulars of the sample. The material in each bottle shall constitute individual sample. One of these three sets (each set containing n bottles representing n containers supplied) shall be for the purchaser, another for the supplier and the third shall be used as a referee sample.

B-3.4 The referee samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

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

B-4.2 Tests for the determination of all the remaining characteristics given in 3 shall be carried out on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Potassium Permanganate Content

From the set of individual test results for potassium permanganate content, the average (\bar{X}) and the range (R) of the test results shall be computed as in Table 4. (Range R is determined as the difference between the maximum and the minimum test results.)

TABLE 4 CRITERIA FOR CONFORMITY FOR POTASSIUM PERMANGANATE CONTENT

Sl. No.	Grade	Test Results	Average	Range	Criteria For Conformity
(1)	(2)	(3)	(4)	(5)	(6)
i)	Technical	1,2,3....n	\bar{x}_1	R_1	($\bar{x}_1 - 0.6 R_1$) shall be greater than or equal to 98
ii)	Pure	1,2,3....n	\bar{x}_2	R_2	($\bar{x}_2 - 0.6 R_2$) shall be greater than or equal to 99
iii)	Analytical reagent	1,2,3....n	\bar{x}_3	R_3	($\bar{x}_3 - 0.6 R_3$) shall be greater than or equal to 99.5

B-5.2 For declaring the conformity of the lot to the requirements of all the other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in 4.2.