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Draft Indian Standard

PHTHALIC ANHYDRIDE, TECHNICAL — SPECIFICATION

(Third Revision of IS 5158)

(ICS 71.060.99)

Organic Chemicals, Alcohols and
Allied Products Sectional Committee, PCD 9

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FOREWORD

(Formal clauses to be added later)

Phthalic anhydride is largely used in the preparation of alkyd resins and similar high polymeric polyester compounds by reaction with polyhydric alcohols. It is also used in the manufacture of diester of monohydric aliphatic alcohols, which find widespread application as plasticizers and in the manufacture of various types of dyes and intermediates, benzoic acid and some pharmaceuticals. The end-use requirement for purity in most of the applications is quite stringent.

Phthalic anhydride, in the form in which it reaches the market, is a flammable solid and constitutes moderate fire hazard. It has been known to cause allergic symptoms in human beings and repeated or prolonged exposure to the fumes could well be irritating to the mucous membrane, even though there is no record of injury as a result of short exposure even to high concentration. Those handling the material are cautioned to use care ordinarily exercised to avoid bodily contact and exposure to dust or fumes (*see 4.1*).

This standard was originally published in 1969 and subsequently revised in 1977 and 1987. In the first revision, the requirements of crystallizing point, colour of the molten material and total available acidity content were modified.

In second revision, the requirements of crystallizing point, colour of molten material before and after heat treatment, free acidity, total available acidity and maleic anhydride content were modified. The requirements of naphthalene and naphthaquinone content were retained since some of the old plants were reported to still produce phthalic anhydride from naphthalene.

In this (*third*) revision, GC method has been incorporated for determination of purity and its impurities like maleic anhydride and other oxidizable impurities; naphthaquinone; naphthalene and phthalimide. Further, keeping in mind the harmful impacts of naphthaquinone and naphthalene on humans, their limit has been modified to 'Not Detectable'. Phthalimide being harmful impurity

present in the material has also been incorporated as a requirement to be tested along with other parameters.

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 methods of sampling and test for phthalic anhydride, technical, suitable for industrial purpose.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute the 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 standard indicated below:

<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 266: 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 915 : 2012 / ISO 1042 : 1998	Laboratory glassware — One-Mark volumetric flasks (<i>third revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (<i>third revision</i>)
IS 4161 : 2023	Nessler cylinder — Specification (<i>first revision</i>)
IS 4905 : 2015 / ISO 24153: 2009	Random sampling and randomization procedures (<i>first revision</i>)
IS 7420 : 1974	Code of safety for phthalic anhydride

3 REQUIREMENTS

3.1 Description

3.1.1 The material may be either solid or molten. When solid, it shall be in the form of flakes, pellets or crystals free from agglomerates, coloured or stained pieces and other extraneous matter.

3.1.2 When heated to 160 °C, the material shall form a clear, transparent melt without decomposing, which sets to a white cake on cooling.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the method prescribed in col (4) of Table 1.

Table 1 Requirements for Phthalic Anhydride, Technical
(Clauses 3.2 and G-5.1)

SI No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C, <i>Min</i>	130.8	A
ii)	Colour of the molten material, Hazen unit, <i>Max</i>	20	B
iii)	Colour of the molten material after heat treatment, Hazen unit, <i>Max</i>	60	
iv)	Free acidity [as C ₆ H ₄ (COOH) ₂], percent by mass, <i>Max</i>	0.1	C
v)	Total available acidity (as C ₈ H ₄ O ₃), percent by mass, <i>Min</i>	99.8	D
vi)	Maleic anhydride and other oxidizable impurities (as C ₄ H ₂ O ₃), percent by mass, <i>Max</i>	0.1	
vii)	Ash, ppm, <i>Max</i>	50	E
viii)	Iron (as Fe), ppm, <i>Max</i>	3	F
ix)	Naphthaquinone, ppm	Not Detectable	D
x)	Naphthalene, ppm	Not Detectable	
xi)	Phthalimide, ppm	Not Detectable	

3.3 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.

4 PRECAUTIONS IN HANDLING AND STORAGE

4.1 Handling

In view of its moderate fire hazard and irritating properties, precautions shall be taken while handling the material. The material shall not come in contact with skin. Exposure to its vapour, fume and dust shall be minimum and safety goggles shall be worn to protect eyes. Use of a respirator is also recommended if the odour is found to be disagreeable or to have a choking effect (*see* IS 7420).

4.2 Storage

It is recommended to store the material in a cool, ventilated area, away from open flames, other sources of possible ignition and powerful oxidizing agents.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable drums (*see* IS 2552) or multiwalled paper bags or in any other suitable containers as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 Each container shall be securely closed after filling and marked with the following:

- a) Name of the material;
- b) Source of the material;
- c) Name of the manufacturer and his recognized trade-mark, if any;
- d) Month and year of manufacture.
- e) Net mass of the material in the container;
- f) Lot or batch number; and
- g) Any other statutory requirements.

5.2.2 *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.

6 SAMPLING

6.1 The method of drawing representative samples of the material and the criteria for conformity

shall be as prescribed in Annex G.

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF CRYSTALLIZATION POINT

A-1 OUTLINE OF THE METHOD

The material is melted and then slowly cooled to determine its crystallization point by observing the temperature during crystallization under prescribed conditions.

NOTE — This determination shall be carried out without preliminary dehydration but as soon as possible after the sample is opened as the free acid content increases when the sample is in contact with moist air.

A-2 APPARATUS

A-2.1 Electrically-Heated Aluminum Block — Capable of maintaining temperature at $(140 \pm 3)^\circ\text{C}$ with holes 22 mm in diameter and at least 120 mm in depth such that the flat-based tubes project above the surface of the block.

A-2.2 Flat-Based Calorimetric Tube — Approximately 150 mm length and 22 mm diameter and having a graduation mark 100 mm above the base.

A-2.3 Thermometer, with following requirements may be used:

Range	: 98 °C to 152 °C
Graduation	: 0.2 deg
Immersion	: 100 mm
Overall length, <i>Max</i>	: 385 mm
Stem diameter	: 5.5 mm to 8.0 mm
Bulb shape	: Cylindrical
Bulb length	: 15 mm to 20 mm
Length of main scale, <i>Min</i>	: 190 mm
Distance from bottom of bulb to bottom of main scale, <i>Min</i>	: 125 mm
Longer lines at each	: 1 deg
Scale error not to exceed	: $\pm 0.4^\circ\text{C}$

NOTE — Any other thermometer of convenient range and similar requirements and accuracy may also be used.

A-2.3.1 The thermometer shall bear a valid calibration certificate from any institution authorized to issue calibration certificate traceable to international or national measurement standards.

A-3 PROCEDURE

A-3.1 Into the flat based colorimetric tubes, take a quantity of the material, sufficient to reach graduation mark after melting. Place the tube containing the material in the electrically heated block maintained at (140 ± 3) °C. Allow the material to completely melt.

A-3.2 Once the material is melted, carefully remove the tube from the heated block. Begin the cooling process by slowly cooling the melted material. Use a thermometer and insert it into the material two to three times, moving it up and down. Observe the material for the formation of crystals. Report the temperature at which crystals start forming as the crystallization point.

ANNEX B

[Table 1, Sl No. (ii, iii)]

DETERMINATION OF COLOUR OF THE MOLTEN MATERIAL

B-1 OUTLINE OF THE METHOD

The material is melted and the colour of the melted material and that of the melted material after heat treatment are compared with that of the permanent colour standard, and expressed in terms of Hazen colour units. [The Hazen colour unit is defined as the colour of an aqueous solution containing 1 parts per million of platinum in the form of chloroplatinic acid and 2 parts per million cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$)].

B-2 APPARATUS

B-2.1 Identical Flat-Based Colorimetric Tubes, two, heat resistant and approximately 20 mm external diameter, having a graduation mark 100 mm above the base.

B-2.2 One-Mark Graduated Flasks, of 250 and 500 ml capacity (*see* IS 915).

B-2.3 Electrically-Heated Aluminum Block, capable of maintaining temperature at (140 ± 3) °C and (250 ± 3) °C with holes 22 mm in diameter and at least 120 mm in depth such that the flat-based tubes project above the surface of the block.

B-3 REAGENTS

B-3.1 Cobaltous Chloride, hexahydrate

B-3.2 Hydrochloric Acid, relative density 1.16 (*see* IS 265).

B-3.3 Chloroplatinic Acid

Dissolve 250 mg of platinum in a small quantity of aqua regia contained in a glass or porcelain basin by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat the operation twice more.

B-3.4 Potassium Chloroplatinate

B-4 PREPARATION OF COLOUR STANDARD

B-4.1 Dissolve 0.50 g of cobaltous chloride hexahydrate and the whole of the chloroplatinic acid (*see B-3.3*) or 0.6225 g of potassium chloroplatinate in 50 ml of hydrochloric acid. Warm, if necessary, to obtain a clear solution and, after cooling, pour into the 500 ml one-mark graduated flask. Dilute with water up to the mark.

B-4.2 Pipette 15 ml of this solution into one of the 250 ml one-mark graduated flasks and 40 ml into the other one. Dilute with water up to graduation marks. These diluted solutions are equivalent to 30 and 80 Hazen units respectively and should always be freshly prepared.

B-5 PROCEDURE

B-5.1 Introduce into one of the calorimetric tubes (*see B-2.1*), the sample sufficient to reach graduation mark after melting. Pour the specified Hazen colour standard into the other tube to the mark.

A-5.2 Place the tube containing the sample in the electrically heated block maintained at (140 ± 3) °C (*see B-2.3*). As soon as it is melted, compare the colour with that of the colour standard against white background.

B-3.4.3 Immediately after the comparison has been made, raise the temperature of the electrically heated block to reach (250 ± 3) °C (*see B-2.3*) within 15 min and maintain it at that temperature. At the end of 90 min from reaching 250 °C, allow the tube containing the material to cool to about 160 °C and compare the colour with that of the second hazen colour standard.

ANNEX C

[Table 1, Sl No. (iv)]

DETERMINATION OF FREE ACIDITY

C-1 OUTLINE OF THE METHOD

The material is dissolved in ethyl methyl ketone and titrated with standard triethylamine solution using bromophenol blue as indicator. Free acidity is then calculated from the amount of standard triethylamine solution used up.

NOTE — This determination shall be carried out immediately after the sample is opened as the free acidity content increases when the material is in contact with moist air.

C-2 REAGENTS

C-2.1 Phthalic Acid

C-2.2 Ethyl Methyl Ketone, neutralized to bromophenol blue, by addition of an approximately 0.1 N solution of triethylamine in ethyl methyl ketone and containing not more than 0.1 percent (*m/m*) of water.

NOTE — Sufficiently dry ethyl methyl ketone may be obtained by refluxing over calcium chloride for approximately three hours followed by decantation and distillation.

C-2.3 Bromophenol Blue Indicator

Dissolve 0.1 g of bromophenol blue in 100 ml of the ethyl methyl ketone.

C-2.4 Standard Triethylamine Solution (in Ethyl Methyl Ketone), 0.1 N

Standardize as follows:

Weigh accurately about 0.1 g of the phthalic acid. Dissolve in 50 ml of the ethyl methyl ketone contained in a 150 ml conical flask. Add 0.5 ml of the bromophenol blue indicator and titrate with the standard triethylamine solution until the colour changes from green to bluish purple. The factor, *F*, for the standard triethylamine solution will be:

$$F = \frac{M_1}{0.1661 \times V_1}$$

where

*M*₁ = mass of phthalic acid taken, in g; and

*V*₁ = volume of standard triethylamine solution used, in ml.

C-3 PROCEDURE

Weigh, to the nearest 0.1 g, about 10 g of the material and dissolve without heating in 150 ml of the ethyl methyl ketone contained in a 500 ml conical flask. Add 1 ml of the bromophenol blue indicator and titrate with the 0.1 N triethylamine solution until the colour changes from green to blue.

C-4 CALCULATION

$$\text{Free acidity [as } C_6H_4(COOH)_2], \text{ percent by mass} = \frac{16.61 \times V \times F}{M}$$

where

V = volume, in ml, of standard triethylamine solution used;

F = factor for the standard triethylamine solution (*see* C-2.4), and

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

ANNEX D

[Table 1, Sl No. (v)]

DETERMINATION OF TOTAL AVAILABLE ACIDITY (as C₈H₄O₃); MALEIC ANHYDRIDE; NAPHTHAQUINONE; NAPHTHALENE; PHTHALIMIDE AND OTHER OXIDIZABLE IMPURITIES (as C₄H₂O₂)

D-1 Outline of the Method

This test method covers a quantitative determination of phthalic anhydride, maleic anhydride, naphthaquinone, naphthalene, phthalimide and other oxidizable impurities. The sample to be analysed is injected into a gas chromatograph equipped with a flame ionization detector (FID) and a capillary column. The peak area of each component is measured and adjusted using correction factors. The concentration of each component is calculated based on its relative percentages of total adjusted peak area and normalized to 100 percent.

D-2 APPARATUS

D-2.1 Gas Chromatograph

D-2.1.2 Any gas chromatograph available with a flame ionization detector, a split injector and a suitable electronic integrator/software may be used with following accessories and operating condition:

Column	:	Fused silica, stationary phase of crossbond 50 percent phenyl / 50 percent methyl polysiloxane with length 30 m; internal diameter 0.25 mm and film thickness 0.25 µm or equivalent.
Carrier gas	:	Nitrogen
Flow rate, ml/min	:	1.70
Split ratio	:	30 :1
Analysis time, min	:	34
Sample size, µl	:	2.0

D-2.1.2 Temperature Programme of Oven, Detector and Injector:

<i>Injector Temperature, °C</i>	<i>Detector Temperature, °C</i>	<i>Temperature, °C</i>	<i>Oven Hold Time, min</i>	<i>Ramp Rate, °C/min</i>
		60	2	10
230 °C	300 °C	150	3	10
		180	2	10
		220	11	—

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/ capillary, diameter, length, film thickness etc), Calibration technique (internal std, external std, area normalization, percent area etc), carrier Gas (He, H₂, N₂) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

D-2.2 Syringe, 5 µl or 10 µl

NOTE — Auto Sampler may also be used to inject the sample into the column.

D-3 REAGENTS

D-3.1 Acetone

D-3.2 Maleic Anhydride, known purity

D-3.3 *p*-Benzoquinone, known purity

D-3.4 Citraconic Anhydride, known purity

D-3.5 *o*-Tolualdehyde, known purity

D-3.6 Benzoic Acid, known purity

D-3.7 *o*-Toluic Acid, known purity

D-3.8 Phthalide, known purity

D-3.9 4-Methyl Phthalic Anhydride, known purity

D-3.10 Phthalimide, known purity

D-3.11 Naphthalene, known purity

D-3.12 Naphthaquinone, known purity

D-3.13 Phthalic Anhydride, known purity

D-4 PREPARATION OF CALIBRATION STANDARDS

D-4.1 Internal Standard Preparation

Dissolve 0.6 g of n-hexadecane in 1 litre of acetone.

D-4.2 Standard Calibration Mixture

D-4.2.1 Prepare the calibration mixture by adding and mixing the following amount of each component of known purity into 100 ml acetone:

<i>Sl No.</i>	<i>Components</i>	<i>Weight to be taken for 20 g</i>	<i>Actual weight taken for 20 g</i>	<i>Weights present in 1 g value to be entered for calibration</i>	<i>Actual percentage present in final solution</i>
(1)	(2)	(3)	(4)	(5)	(6)
i.	Maleic Anhydride	0.020	0.0202	0.001010	0.1010
ii.	<i>p</i> - Benzoquinone	0.010	0.0101	0.000505	0.0505
iii.	Citraconic Anhydride	0.010	0.0100	0.000500	0.0500
iv.	<i>o</i> -Tolualdehyde	0.010	0.0100	0.000500	0.0500
v.	Benzoic Acid	0.004	0.0040	0.000200	0.0200
vi.	<i>o</i> -Toluic Acid	0.010	0.0100	0.000500	0.0500
vii.	Phthalide	0.010	0.0100	0.000500	0.0500
viii.	4-Methyl Phthalic Anhydride	0.015	0.0150	0.000750	0.0750
ix.	Phthalimide	0.0075	0.0077	0.000385	0.0385
x.	Naphthalene	0.008	0.0088	0.000440	0.0440
xi.	Naphthaquinone	0.0075	0.0072	0.000360	0.0360
xii.	Phthalic Anhydride	-	-	0.9956	99.5455

D-5 CALIBRATION

Pipette 5 ml of the calibration mixture as prepared at **D-4.2.1**. To it add 5 ml internal standard (see **D-4.1**) and dissolve completely. Make sure the GC instrument is adjusted to the conditions stated

as above. Now inject 2 µl of the solution as prepared above into the gas chromatograph and obtain the chromatogram. Calculate the calibration correction factor for each component by dividing its concentration by the respective area in its chromatogram.

D-6 PRECEDURE

Take and dissolve 1 g sample, 5 ml *n*-hexadecane and 5 ml acetone. Now take 2 µl of the sample solution as prepared and inject it into gas chromatograph. Calculate the amount of each component present in the sample.

D-7 CALCULATION

D-7.1 Sum the areas of all unknown components.

D-7.2 Multiply the area of each component of the sample by its relative response correction factor as calculated in **D-5**. Obtain the sum of all corrected areas. Normalize each corrected area to 100 percent and report each as mass percent or use suitable software for auto calculation with respect to calibration standard.

ANNEX E

[Table 1, Sl No. (vii)]

DETERMINATION OF ASH

E-1 APPARATUS

E-1.1 Platinum or Silica Basin

E-1.2 Furnace, capable of maintaining temperature of (600 ± 25) °C.

E-2 PROCEDURE

Slowly burn, in several portions, approximately $(10 \text{ g} \pm 0.1 \text{ mg})$ of the material, weighed to the nearest mg, in a tared platinum or silica basin and ignite finally in a furnace at (600 ± 25) °C until all carbonaceous matter has disappeared. Cool in a desiccator and weigh. Retain the residue for the determination of iron content (*see* Annex F).

E-3 CALCULATION

$$\text{Ash, parts per million} = \frac{M}{M_1} \times 10^6$$

where

M = mass, in g, of the residue after ignition; and
 M_1 = mass, in g, of the material taken for the test.

ANNEX F

[Table 1, Sl No. (viii)]

DETERMINATION OF IRON CONTENT

F-1 GENERAL

Two methods, namely, Method A determination of iron by 2,2'-bipyridyl Method and Method B determination of iron by thioglycolic acid method have been prescribed. In case of dispute, Method A shall be used.

F-2 Method A — 2, 2'- Bipyridyl Method

F-2.1 Outline of the Method

Ferrous iron with 2, 2'-bipyridyl gives a red coloured complex obtained after ignition (*see F-2*) is dissolved in hydrochloric acid and the iron content is determined photometrically using 2,2'-bipyridyl.

F-2.2 Apparatus

F-2.2.1 *One-mark Graduated Flask*, two, 1 000 ml capacity (*see IS 915*).

F-2.2.2 *One-mark graduated Flask*, 100 ml capacity (*see IS 915*).

F-2.2.3 *Spectrophotometer or Electrophotometer*

F-2.2.4 *Stirrer*, of glass or platinum wire.

F-2.3 Reagents

F-2.3.1 *Concentrated Hydrochloric Acid*, 1.16 relative density (*see IS 265*).

F-2.3.2 *Dilute Sulphuric Acid*

Add carefully, with stirring, one volume of sulphuric acid (*see IS 266*) to 6 volumes of water.

F-2.3.3 *Dilute Nitric Acid*

Dilute one volume of nitric acid (*see IS 264*) with 3 volumes of water.

F-2.3.4 *Urea Solution*

Dissolve 100 g of urea in 100 ml of water.

F-2.3.5 *Hydroxyammonium Chloride Solution*

Dissolve 100 g of hydroxyammonium chloride in 1 000 ml of water.

F-2.3.6 *Ammonium Acetate Solution*

Dissolve 50 g of ammonium acetate in 100 ml of water.

F-2.3.7 *2, 2'-Bipyridyl Solution*

Dissolve 0.5 g of 2,2'-bipyridyl in 100 ml of dilute hydrochloric acid (1 N).

F-2.3.8 *Standard Iron Solution*

Dissolve 0.702 g of ammonium ferrous sulphate $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$ in 50 ml of dilute sulphuric acid and then transfer to one of the 1 000 ml one-mark graduated flasks. Dilute with water to 1 000 ml mark. Pipette out 100 ml of this solution into the second 1000 ml one-mark graduated flask. Dilute 100 ml of this solution again to 1 000 ml. 1 ml of this solution contains 10 μg of iron.

F-2.4 *Calibration Charts*

Place in 100 ml one-mark graduated flask 0 ml, 2.0 ml, 4.0 ml, 7.0 ml, 10.0 ml, 15.0 ml and 20.0 ml of the standard iron solution. To each, add 20 ml of dilute nitric acid, 2 ml of urea solution and 2 ml of hydroxyammonium chloride solution. Mix and allow to stand for two minutes. Then add 30 ml of ammonium acetate solution and 5 ml of 2, 2'- bipyridyl solution, and dilute to 100 ml mark. Measure the optical densities of the solutions in the spectrophotometer or electrophotometer at a wavelength between 510 nm and 520 nm. Draw a graph plotting optical densities as a function of quantities of iron (in micrograms) in 100 ml of the solution.

F-2.5 *Procedure*

To the platinum or silica basin containing the residue from the determination of ash (*see E-2*), add 5 ml of hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a stirrer until all the residue has dissolved. Allow to cool and transfer the solution to a 100 ml one-mark graduated flask. Add 2 ml of urea solution, stir, add 2 ml of hydroxyammonium chloride solution, mix and allow to stand for two minutes. Then add 30 ml of ammonium acetate solution, 5 ml of the 2, 2'- bipyridyl solution and dilute to 100 ml. Measure the optical density of the solution on the spectrophotometer or electrophotometer at a wavelength between 510 and 520 nm, and by reference to the calibration chart (*see F-2.4*), read the iron content (μg of iron/100 ml) corresponding to this optical density.

F-2.6 *Calculation*

$$\text{Iron content (as Fe), parts per million} = \frac{M}{M_1}$$

where

M = mass in of iron found, in μg ; and

M_1 = mass of the material taken for determination of ash, in g (*see E-2*).

F-8.2 Method B — Thioglycolic Acid Method

F-8.2.1 Outline of the Method

In an alkaline medium, thioglycolic acid gives a reddish violet colouration with ferrous and ferric iron. The residue from the ash determination is dissolved in hydrochloric acid, thioglycolic acid added and then made alkaline by the addition of ammonia. The colour developed is then compared with that obtained with standard iron solution similarly treated. The iron content is then determined from the amount of standard iron solution required for matching the colour.

F-8.2.2 Apparatus

F-8.2.2.1 *One-mark Graduated Flasks*, 1 000 ml capacity, two (*see IS 915*).

F-8.2.2.2 *One-mark Graduated Flasks*, 25 ml capacity (*see IS 915*).

F-8.2.2.3 *Nessler Cylinders*, two, 100 ml capacity (*see IS 4161*).

F-8.2.3 Reagents

F-8.2.3.1 *Concentrated Hydrochloric Acid*, relative density 1.16 (*see IS 265*).

F-8.2.3.2 *Ammonium hydroxide*, 0.88 relative density.

F-8.2.3.3 *Thioglycolic acid solution*, 10 percent (v/v).

F-8.2.3.4 *Citric acid solution*, 30 percent (m/v).

F-8.2.3.5 *Standard iron solution*, same as in **F-2.3.8**

F-8.2.4 Procedure

To the platinum or silica basin containing the residue from the determination of ash (*see E-2*), add 5 ml of concentrated hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a stirrer until all the residue has dissolved. Allow to cool, transfer to the 25 ml volumetric flask, dilute to the mark with water and mix thoroughly. For each determination, transfer 10 ml of this solution to a 100 ml Nessler cylinder, dilute to about 30 ml and add 0.5 ml of the citric acid solution

followed by 1 ml of thioglycolic acid solution. Add ammonium hydroxide carefully until a reddish-purple colour just appears and then add 0.5 ml in excess. Dilute to 100 ml and mix thoroughly. Take about 90 ml of water in the second 100 ml Nessler cylinder, add 2 ml of the concentrated hydrochloric acid, then 0.5 ml of the citric acid solution followed by 1 ml of the thioglycolic acid solution and 3 ml of the ammonium hydroxide. Add the standard iron solution slowly from a burette shaking with each addition, until the depth of colour in the two cylinders is identical when they are viewed along their axes. Record the volume of standard iron solution added.

F-8.2.5 Calculation

$$\text{Iron content (as Fe), parts per million} = \frac{25 \times V}{M}$$

where

V = volume, in ml, of standard iron solution used; and

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

ANNEX G

(Clause 6.1)

SAMPLING OF PHTHALIC ANHYDRIDE, TECHNICAL

G-1 GENERAL REQUIREMENTS OF SAMPLING

G-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

G-1.2 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

G-1.3 Sampling instrument shall be clean and dry.

G-1.4 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination (*see 4.1*).

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

G-1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

G-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

G-1.8 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and other details given under **5.2**.

G-1.9 Samples shall be stored in a cool and dry place.

G-2 SAMPLING INSTRUMENT

G-2.1 The sampling instrument is a closed type sampling tube, undivided (*see* Fig. 1), consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel) closely fitting into each other throughout their entire length so that it is possible to rotate one tube within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position, the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180°, the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with a locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 150 cm and a diameter of 30 mm can cater for most needs.

G-2.1.1 *Use of Sampling Instrument*

The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes, and finally closing them withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different parts of the same container such that they are at least 75 mm in the case of drums, bags, etc, and 25 mm in the case of small containers from the wall of the container. In all cases, the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

G-3 SCALE OF SAMPLING

G-3.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, the containers belonging to the same batch of manufacture shall be grouped together and each group shall constitute a separate lot.

G-3.2 For ascertaining the conformity of the lot to the requirement of this specification, tests shall be carried out for each lot separately. The number (*n*) of containers to be selected for drawing the samples shall depend upon the size of the lot and shall be in accordance with Table 2.

G-3.3 The containers shall be selected at random from the lot. In order to ensure the randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS 4905 may be referred. In the absence of a random number table, the following procedure may be adopted.

‘Starting from any container, count them as 1, 2, 3,, up to r and so on, where r is an integral part of N/n , N being the lot size and n the sample size respectively. Every r^{th} container thus counted shall be withdrawn so as to give the required sample size.

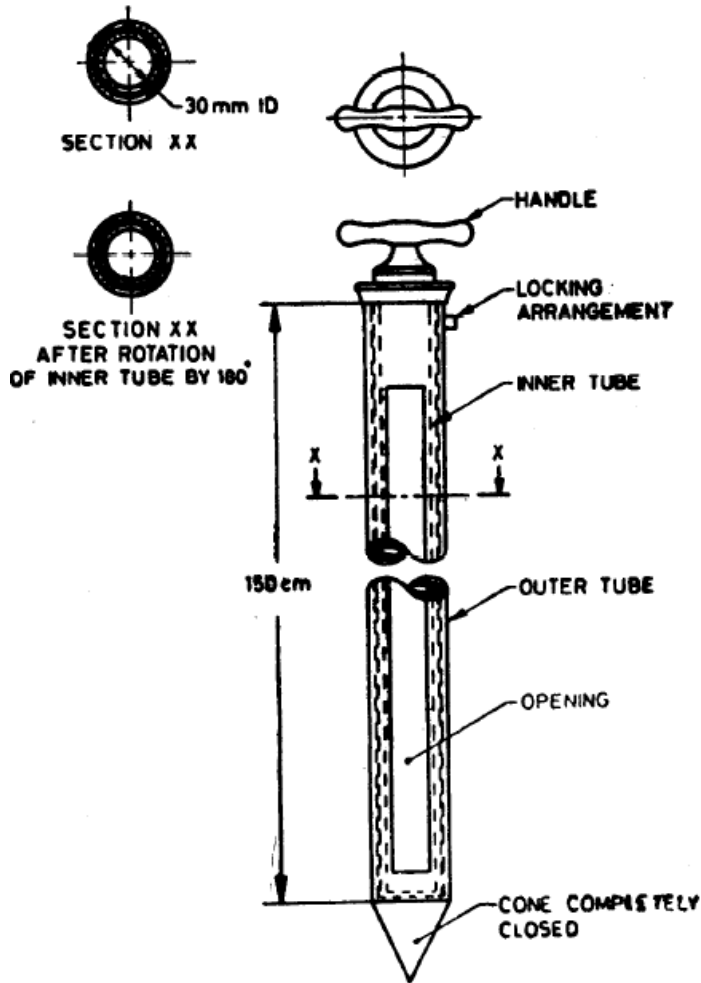


FIG. 1 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

Table 2 Number of Containers to be Selected for Sampling
(Clause G-3.2)

Sl No.	Lot Size	No. of Containers to be Selected
	(N)	(n)
(1)	(2)	(3)

i.	Up to 25	3
ii.	26 to 50	4
iii.	51 to 100	5
iv.	101 to 150	6
v.	151 to 300	7
vi.	301 and above	8

G-4 TEST SAMPLE AND REFEREE SAMPLE

G-4.1 From each of the containers selected according to **G-3.2**, a representative portion of the material about 100 g shall be drawn. These samples shall constitute individual samples.

G-4.2 From each of these individual portions (*see G-4.1*), an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 600 g. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

G-5 NUMBER OF TESTS

G-5.1 Tests for the determination of all the characteristics given in Table 1 shall be carried out on the composite sample.

G-6 CRITERIA FOR CONFORMITY

G-6.1 For declaring the characteristics of the lot to the requirements of all the characteristics, the test results on the composite sample shall meet the corresponding requirement specified.