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मानक

IS 5158 (1987): Phthalic Anhydride, Technical [PCD 9:

Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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IS: 5158 - 1987

Indian Standard SPECIFICATION FOR PHTHALIC ANHYDRIDE, TECHNICAL

(Second Revision)

UDC 661-7:547-584

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Indian Standard SPECIFICATION FOR PHTHALIC ANHYDRIDE, TECHNICAL

(Second Revision)

$\mathbf{0.} \quad \mathbf{FOREWORD}$

0.1 This Indian Standard was (Second Revision) adopted by the Bureau of Indian Standards on 17 December 1987, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1969 and revised in 1977 in view of the quality of the product being made available at that time. In the first revision, the requirements of crystallizing point, colour of the molten material and total available acidity content were modified. However, the Committee decided to revise it again as a result of periodical review in the light of the experience gained in the past and also the prevailing trade practice in the industry. In the present revision, the requirements of crystallizing point, colour of molten material before and after heat treatment, free acidity, total available acidity and maleic content have been modified. The anhydride requirements of naphthalene and naphthaquinone content have been retained since some of the old plants are reported to still produce phthalic anhydride from naphthalene. However, tests need not be conducted for these requirements in case the product is obtained by oxidation of o-xylene (see Table 1).

0.3 The largest single use of phthalic anhydride, the anhydride of benzene *o*-dicarboxylic acid, is in the preparation of alkyd resins and similar high polymeric polyester compounds by reaction with

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for phthalic anhydride, technical, suitable for industrial purposes.

2. REQUIREMENTS

2.1 Description — 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.

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

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 application is quite stringent. It is hoped that this standard will help the consumers in obtaining the supply of an acceptable quality of material.

0.4 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 3).

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with $IS : 2-1960^*$. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

2.2 The material shall also comply with the requirements given in Table 1, when tested according to the method prescribed in Appendix A. Reference to the relevent clauses of Appendix A is given in col 4 of Table 1.

3. PRECAUTIONS IN HANDLING AND STORAGE

3.1 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

TABLE 1 REQUIREMENTS FOR PHTHALIC ANHYDRIDE, TECHNICAL

(Clauses 0.2 and 2.2)

Sl No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO.	
(1)	(2)	(3)	IN APPENDIX A) (4)	
i)	Crystallizing point, °C, Min	130.8	A- 2	
ii)	Colour of the molten material, Hazen unit, Max	20	A-3	
iii)	Colour of the molten material after heat treatment, Hazen unit, Max	60	A-3	
iv)	Free acidity [as C ₆ H ₄ (COOH ₂)], percent by mass, Max	0-1	A-4	
v)	Total available acidity (as C ₈ H ₄ O ₃), percent by mass, <i>Min</i>	99.8	A-5	
vi)	Maleic anhydride and other oxidizable impurities (as C ₄ H ₂ O ₃), percent by mass, Max	0.1	A-6	
vii)	Ash, ppm, Max	50	A- 7	
viii)	Iron (as Fe), ppm, Max	3	A-8	
ix)	*Naphthaquinone, ppm, Max	5	A-9	
x)	*Naphthalene, ppm, Max	20	A-10	
*Applicable for material ex-naphthalene only (see 0.2)				

recommended if the odour is found to be disagreeable or to have a choking effect (see IS: 7420-1974*).

3.2 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.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in suitable drums (see IS : 2552-1979[†]) or multi-walled paper bags or in any other suitable containers as agreed to between the purchaser and the supplier.

4.2 Marking — 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 recognized trade-mark, if any;

- d) Net mass of the material in the container; and
- e) Lot or batch number.

4.2.1 The container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standrads.

5. SAMPLING

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

^{*}Code of safety for phthalic anhydride.

[†]Specification for steel drums (galvanized or ungalvanized) (second revision).

APPENDIX A

(*Clause 2.2*, and *Table 1*)

METHODS OF TEST FOR PHTHALIC ANHYDRIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977*) shall be used in the tests.

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

A-2. DETERMINATION OF CRYSTALLIZING POINT

A-2.0 Outline of the Method — The material is melted and then slowly cooled to determine its crystallizing point by observation of 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-2.1 Apparatus — The crystallizing point apparatus shall be as shown in Fig. 1. A thick glass tube of nominal size 100×25 mm is placed inside a larger glass test tube of nominal size 150×50 mm. The latter tube is flanged so that it may be supported centrally by the metal cover plate.

A-2.1.1 The wider tube is weighted with lead shots, or similar material and the inner tube is closed by means of a cork, which carries a glass stirrer and through its centre a standard thermometer. The stirrer is a loop which surrounds the thermometer. The thermometer is so fixed in the cork that the thermometer's immersion mark is level with the top of the cork and if the thermometer has a contraction chamber, the distance from the bottom of the bulb to the top of the contraction chamber shall not be more than 35 mm.

A-2.1.2 The wider tube is supported in 1 000 ml tall-form beaker filled with cooling liquid to within 20 mm of the top. A thermometer for the cooling bath passes through a hole in the cover plate and is held by a rubber ring.

A-2.1.3 Thermometer — conforming to the following requirements:

Range	98 to 152°C
Graduation	0.2 deg
Immersion	100 mm
Overall length, Max	385 mm

*Specification for water for general laboratory use (second revision).

Stem diameter	5·5 to 8·0 mm
Bulb shape	Cylindrical
Bulb length	15 to 20 mm
Length of main scale, <i>Min</i>	190 mm
Distance from bot- tom 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°C

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

A-2.1.3.1 The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi or any other institution authorized by the Government of India to issue such a certificate.

A-2.2 Procedure — Remove the inner tube from the crystallizing point apparatus and introduce about 30 g of the material for test. Warm the tube in a bath at about 140°C until all but last traces of crystals are melted. Replace the inner tube in its jacket with the cooling liquid in the apparatus maintained at 122 to 124°C. Stir the material gently and continuously, and record thermometer readings at 30 seconds intervals, make sure that a seed crystal is present as the temperature of the material falls to that at which crystal-The crystallizing point lization commences. corresponds to the highest of the first five consecutive readings corrected for thermometer error during which the temperature remains constant within 0.1°C.

A-2.2.1 If supercooling occurs as shown by a rise in temperature, observe the constant temperature after the rise. If five consecutive readings within 0.1 deg are not obtained, record six readings commencing with the point at which the maximum temperature is first attained. Plot the complete cooling curve of temperature against time and draw a staight line to lie evenly between the first and second, and between the fifth and sixth points mentioned above. Extend this line to meet the section of the cooling curve before the temperature rise. Report the temperature corresponding to the point of intersection, corrected for thermometer error, as the crystallizing point.



All dimensions in millimetres. FIG. 1 CRYSTALLIZING POINT (ASSEMBLY OF APPARATUS)

A-3. MEASUREMENT OF COLOUR OF THE MOLTEN MATERIAL

A-3.0 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 part per million of platinum in the form of chloroplantinic acid and 2 parts per million cobalt chloride (CoCl₂.6H₂O)].

A-3.1 Apparatus

A-3.1.1 Identical Flat-Based Colorimetric Tubes — Two, heat resistant and approximately 20 mm external diameter, having a graduation mark 100 mm above the base.

A-3.1.2 One-Mark Graduated Flasks — of 250and 500-ml capacity (see IS : $915-1975^*$). A-3.1.3 Electrically-Heated Aluminium Block — capable of maintaining temperature at $140 \pm 3^{\circ}$ C and $250 \pm 3^{\circ}$ C with holes 22 mm in diameter and at least 120 mm in depth but such that the flat-based tubes project above the surface of the block.

A-3.2 Reagents

A-3.2.1 Cobaltous Chloride, Hexahydrate

A-3.2.2 Hydrochloric Acid — relative density 1.16 (see IS : 265-1976*).

A-3.2.3 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.

A-3.2.4 Potassium Chloroplatinate

A-3.3 Preparation of Colour Standard — Dissolve 0.50 g of cobaltous chloride hexahydrate and the

^{*}Specification for one-mark graduated flask (first revision).

^{*}Specification for hydrochloric acid (second revision).

whole of the chloroplatinic acid (see A-3.2.3) 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.

A-3.3.1 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.

A-3.4 Procedure

A-3.4.1 Introduce into one of the colorimetric tubes (*see* A-3.1.1), a quantity of the material sufficient to reach graduation mark after melting. Pour the specified Hazen colour standard into the other tube to the mark.

A-3.4.2 Place the tube containing the material in the electrically heated block maintained at $140 \pm 3^{\circ}C$ (see A-3.1.3) and as soon as it is melted, compare the colour with that of the colour standard against white background.

A-3.4.3 Immediately after the comparison has been made, raise the temperature of the electrically heated block to reach 250 ± 3 °C (see A-3.1.3) within 15 minutes and maintain it at that temperature. At the end of 90 minutes 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.

A-4. DETERMINATION OF FREE ACIDITY

A-4.0 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.

A-4.1 Reagents

A-4.1.1 Phthalic Acid

A-4.1.2 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.

A-4.1.3 Bromophenol Blue Indicator -- Dissolve 0.1 g of bromophenol blue in 100 ml of the ethyl methyl ketone. A-4.1.4 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 kctone 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:

$$F = \frac{M_1}{0.166 \ 1 \ V_1}$$

where

- $M_1 = \text{mass in g of phthalic acid taken, and}$
- V_1 = volume in ml of standard triethylamine solution used.

A-4.2 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.

A-4.3 Calculation

Free acidity
[as C₆H₄ (COOH)₂] =
$$\frac{16.61 \ V \times F}{M}$$

percent by mass

where

- V = volume in ml of standard triethylamine solution used,
- F = factor for the standard triethylamine solution (see A-4.1.4), and
- M = mass in g of the material taken for the test.

A-5. DETERMINATION OF TOTAL AVAILABLE ACIDITY

A-5.0 Outline of the Method — The material is dissolved in warm water and titrated with standard sodium hydroxide solution using phenolphthalein as indicator. Total available acidity is calculated from the amount of standard sodium hydroxide solution used.

A-5.1 Reagents

A-5.1.1 Potassium Hydrogen Phthalate — previously dried for 2 hours at 120°C.

A-5.1.2 Phenolphthalein Indicator — Dissolve 0.5 g of phenolphthalein in 100 ml of rectified

spirit (see IS : 323-1959*). Add standard sodium hydroxide solution until the indicator is faintly pink.

A-5.1.3 Standard Sodium Hydroxide Solution — 0.5 N, free from carbonate; standardize as follows:

Weigh accurately about 4 g of potassium hydrogen phthalate and transfer to a 500 ml conical flask. Add 120 ml of water and dissolve by warming on a water-bath. Add three drops of the phenolphthalein indicator and titrate hot (about 60° C) with the 0.5 N sodium hydroxide solution to the first pink colour. The factor, *F*, for the standard sodium hydroxide solution will be:

$$F = \frac{M_1}{0.102\,11\,\times\,V_1}$$

where

- $M_1 = \text{mass in g of potassium hydrogen phtha$ $late taken, and}$
- V_1 = volume in ml of standard sodium hydroxide solution used.

A-5.2 Procedure — Weigh accurately about 1.5 g of the material and transfer to a 500-ml conical flask. Add 100 ml of water and dissolve by warming on a water-bath. Add three drops of the phenolphthalein indicator and titrate hot (about 60° C) with the freshly standardized 0.5 N sodium hydroxide solution to the pink colour used in the standardization.

A-5.3 Calculation

Total available acidity
(as
$$C_8H_4O_3$$
) = $\frac{3.703 \times V \times F}{M}$

where

- V = volume in ml of standard sodium hydroxide solution used,
- F = factor for the standard sodium hydroxide solution (see A-5.1.3), and
- M =mass in g of the material taken for the test.

A-6. DETERMINATION OF MALEIC ANHYDRIDE AND OTHER OXIDIZABLE IMPURITIES

A-6.0 Outline of the Method — Phthalic acid is unattacked by cold potassium permanganate solution in the presence of sulphuric acid, whereas for maleic acid, the reaction at room temperature is represented by the equation:

CH. COOH \parallel + 50---->2H₂O + 3CO₂ + CO. CH. COOH Excess of standard permanganate is used and the amount remaining is determined iodometrically. Maleic anhydride and other oxidizable impurities are calculated from the amount of standard potassium permanganate solution used.

Note — This method is not satisfactory for determining amounts of oxidizable impurities greater than 0.5 percent.

A-6.1 Reagents

A-6.1.1 Sulphuric Acid — concentrated (see IS : 266-1977*).

A-6.1.2 Potassium Iodide — solid.

A-6.1.3 Potassium Permanganate — approximately 0.1 N solution.

A-6.1.4 Standard Sodium Thiosulphate Solution — 0.1 N.

A-6.2 Procedure — Take two 50-ml conical flasks. Weigh, to the nearest 0.1 g, into one of the two 500-ml conical flasks about 5 g of the material, add to both flasks 100 ml of water and heat both gently until solution is complete in the flask containing the material. Cool as rapidly as possible to room temperature, thus avoiding the formation of large crystals which may lead to low results being obtained.

A-6.2.1 Add 1 ml of sulphuric acid and again cool. Add 25 ml of the potassium permanganate solution from a pipette. Mix thoroughly and leave to stand for 7 to 10 minutes. At the end of this time, add 1 g of potassium iodide and titrate the iodine released with the standard sodium thiosulphate solution.

A-6.3 Calculation

Maleic anhydride and other oxidizable impurities (as C₄H₂O₃), percent by mass $= \frac{0.098(V - V_1)}{M}$

where

- V = volume in ml of standard sodium thiosulphate (0.1 N) solution used in the determination without the material,
- V_1 = volume in ml of standard sodium thiosulphate (0.1 N) solution used in the determination with the material, and
- M = mass in g of the material taken for the test.

A-7. DETERMINATION OF ASH

A-7.1 Apparatus

A-7.1.1 Platinum or Silica Basin

^{*}Specification for rectified spirit (revised).

^{*}Specification for sulphuric acid (second revision).

A-7.1.2 Furnace — capable of maintaining temperature within $600 \pm 25^{\circ}$ C.

A-7.2 Procedure — Slowly burn, in several portions, approximately $5g \pm 0.1$ mg of the material, weighed to the nearest gram, in a tared platinum or silica basin and ignite finally in a furnace at $600 \pm 25^{\circ}$ C until all carbonaceous matter has disappeared. Cool in a desiccator and weigh. Retain the residue for the determination of iron content (see A-8).

A-7.3 Calculation

Ash, parts per million $= \frac{M}{M_1} \times 100$

where

- M =mass in g of the residue after ignition, and
- $M_1 = \max \inf g \text{ of the material taken for the test.}$

A-8. DETERMINATION OF IRON CONTENT

A-8.0 Methods — Two methods, namely, Method A and Method B have been prescribed. In case of dispute, Method A shall be used.

A-8.1 Method A (2,2-bipyridyl Method)

A-8.1.0 Outline of the Method — Ferrous iron gives with 2,2'-bipyridyl a red coloured complex. The residue obtained after ignition (see A-7.2) is dissolved in hydrochloric acid and the iron content is determined photometrically using 2'2'-bipyridyl.

A-8.1.1 Apparatus

A-8.1.1.1 One-mark graduated flask -1000-ml capacity, two (see IS : 915-1975*).

A-8.1.1.2 One-mark graduated flask — 100-ml capacity (see IS : 915-1975*).

A-8.1.1.3 Spectrophotmeter or electrophotometer

A-8.1.1.4 Stirrer — of glass or platinum wire.

A-8.1.2 Reagents

A-8.1.2.1 Concentrated hydrochloric acid — Relative density 1.16 (see IS : 265-1976[†]).

A-8.1.2.2 Dilute sulphuric acid — Add carefully, with stirring, one volume of sulphuric acid (see IS : 266-1977⁺₊) to 6 volumes of water.

A-8.1.2.3 Dilute nitric acid — Dilute one volume of nitric acid (see IS : 264-1976§) with 3 volumes of water.

A-8.1.2.4 Urea solution — Dissolve 100 g of urea in 100 ml of water.

A-8.1.2.5 Hydroxyammonium chloride solution — Dissolve 100 g of hydroxyammonium chloride in 1 000 ml of water.

A-8.1.2.6 Ammonium acetate solution — Dissolve 50 g of ammonium acetate in 100 ml of water.

A-8.1.2.7 2.2'-bipyridyl solution — Dissolve 0.5 g of 2.2'-bipyridyl in 100 ml of dilute hydrochloric acid (1 N).

A-8.1.2.8 Standard iron solution – Dissolve 0 702 g of ammonium ferrous sulphate [(NH_4)₂ SO₄. FeSO₄, 6 H₂O] 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 1 000-ml one-mark graduated flask. Dilute 100 ml of this solution again to 1 000 ml. One millilitre of this solution contains 10 μ g of iron.

A-8.1.3 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 and 520 nm. Draw a graph plotting optical densities as a function of quantities of iron (in micrograms) in 100 ml of the solution.

A-8.1.4 Procedure — To the platinum or silica basin containing the residue from the determination cf ash (see A-7.2), 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 100ml 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 A-8.1.3), read the iron content (μg of iron/100 ml) corresponding to this optical density.

A-8.1.5 Calculation

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

where

 $M = \text{mass in } \mu g \text{ of iron found, and}$

^{*}Specification for one-mark graduated flasks (first revision).

[†]Specification for hydrochloric acid (second revision). †Specification for sulphuric acid (second revision). §Specification for nitric acid (second revision).

 $M_1 = \text{mass in g of the material taken for deter-}$ mination of ash (see A-7.2).

A-8.2 Method B (Thioglycolic Acid Method)

A-8.2.0 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 The iron content is then determined treated. from the amount of standard iron solution required for matching the colour.

A-8.2.1 Apparatus

A-8.2.1.1 One-mark graduated flasks – 1000 ml capacity, two (see IS : 915-1975*).

A-8.2.1.2 One-mark graduated flasks - 25-ml capacity (see IS : 915-1975*).

A-8.2.1.3 Nessler cylinders — 100-ml capacity, two (see IS : 4161-1967[†]).

A-8.2.2 Reagents

A-8.2.2.1 Concentrated hydrochloric acid relative density 1.16 (see IS: 265-19761).

A-8.2.2.2 Ammonium hydroxide — relative density 0.88.

A-8.2.2.3 Thioglycolic acid solution – 10 percent (v/v).

A-8.2.2.4 Citric acid solution - 30 percent (m/v).

A-8.2.2.5 Standard iron solution - same as in A-8.1.2.8.

A-8.2.3 Procedure — To the platinum or silica basin containing the residue from the determination of ash (see A-7.2), 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. To 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.

A-8.2.4 Calculation

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

where

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

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

A-9. TEST FOR NAPHTHAQUINONE

A-9.0 Outline of the Method — The presence of 1. 4-naphthaquinone causes the development of an intense pink colour when the phthalic anhydride is heated with stannous chloride. The colour produced is compared with that of a freshly prepared reference colour.

A-9.1 Apparatus

A-9.1.1 Two Similar Boiling Tubes -175×38 mm, of Type 1 glass conforming to IS: 2303-1963*

A-9.1.2 Two Similar Thermometers - covering a suitable range.

A-9.2 Reagents

A-9.2.1 Phthalic Anhydride — naphthaquinonefree. (If necessary, recrystallize phthalic anhydride from benzene until the product gives no pink colouration when tested by the method described under A-9.4).

A-9.2.2 Stannous Chloride Dihvdrate

NOTE — The anhydrous material is not satisfactory.

A-9.2.3 Standard 1, 4-naphthaquinone Solution ----Dissolve 0.010 g of 1, 4-naphthaquinone in benzene and dilute to 100 ml. This reagent shall be freshly prepared.

A-9.3 Preparation of Reference Colour --- Weigh 50 g of the naphthaquinone-free phthalic anhydride into one of the clean, dry boiling tubes. Introduce by means of a pipette, 2.5 ml of the standard 1, 4-naphthaquinone solution so that it falls on the phthalic anhydride. Finally, add 0.10 ± 0.01 g of the finely powdered stannous chloride to the contents, and heat in the same way and at the same time as the material under test (see A-9.4).

^{*}Specification for one-mark graduated flasks (first revision).

^{*}Specification for Nessler cylinders. ‡Specification for hydrochloric acid (second revision).

^{*}Method of grading glass for alkalinity.

A-9.4 Procedure — Weigh 50 g of the material into the other clean, dry boiling tube and add 0.10 ± 0.01 g of the finely powdered stannous chloride. Heat to a temperature of $170 \pm 2^{\circ}$ C over a period of 20 minutes side by side with the reference colour described in A-9.3. Stir the contents of the tubes throughout the heating period to ensure that there is no solid material remaining in the tubes, and maintain the temperature for a further 5 minutes. At the end of the time, compare the colour of the molten mixtures against a white background.

A-9.4.1 The material shall be taken to have satisfied the requirement of Table 1, if the colour produced in the test sample is not more intense than that of the prepared colour standard.

A-10. TEST FOR NAPHTHALENE

A-10.0 General — The aromatic hydrocarbon most likely to be present as an impurity in phthalic anhydride is naphthalene, but certain other aromatic hydrocarbons give colours with formaldehyde and sulphuric acid.

A-10.1 Apparatus

A-10.1.1 Round-Bottom Flask — 1 000-ml capacity [see IS: 1381 (Part 1)-1976*], fitted for distillation with a rubber stopper carrying an anti-splash head and a dropping funnel.

A-10.1.2 Separating Funnel — 250-ml capacity (see IS : 1575-1960†).

A-10.1.3 One-Mark Volumetric Flask — 50-ml capacity (see IS : 915-1975 ‡).

A-10.1.4 Glass-Stoppered Test Tubes — 125×16 mm, two.

A-10.2 Reagents

A-10.2.1 Sodium Hydroxide

A-10.2.2 Chloroform — See IS : 5296 - 1979§.

A-10.2.3 Naphthalene

A-10.2.4 Concentrated Sulphuric Acid — relative density 1.84 (see IS : 266-1977*).

A-10.2.5 Formaldehyde Solution — 36 percent (m/v).

A-10.3 Procedure — Weigh 50 g of the sample and 30 g of sodium hydroxide into the roundbottom flask. Close the flask with the rubber stopper, add 40 ml of water through the dropping funnel and immediately close the stop-cock. Heat and distil about 75 ml of distillate into the separating funnel.

A-10.3.1 Extract the distillate obtained with two 20 ml portions of chloroform, first washing out the condenser tube with the chloroform to remove traces of naphthalene. Combine the chloroform extracts, transfer to the 50-ml volumetric flask and dilute with chloroform to the graduation mark.

A-10.3.2 Pipette 5 ml of the solution into one of the dry glass-stoppered test tubes, carefully add, down the side of the tube, 2 ml of sulphuric acid. Shake well and add 5 drops of the formaldehyde solution. Shake vigorously for 2 to 3 minutes. If naphthalene is present, a greenish-blue ring is formed at the junction of the two layers. [This is due to a flocculant precipitate in the acid layer, but it rapidly separates on the surface of the acid layer and is conveniently regarded as a ring at the interface.]

A-10.3.3 Compare the intensity of this ring with that obtained by treating at the same time and in the same manner 0.1 ml of a freshly prepared 0.1 percent (m/v) solution of naphthalene in chloroform. It is preferable to allow the solution to stand for an hour before making the comparison in order to allow the ring to become better defined.

A-10.3.4 The material shall be taken to have satisfied the requirement prescribed in Table 1, if the ring produced in the test sample is not more intense than that produced in the standard.

^{*}Specification for boiling flasks : Part 1 Flasks with plain neck (first revision).

⁺Specification for separating funnels.

Specification for one-mark volumetric flasks.

[§]Specification for chloroform, technical and analytical.

^{*}Specification for sulphuric acid (second revision).

APPENDIX B

(*Clause* 5.1)

SAMPLING OF PHTHALIC ANHYDRIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination (see 3.1).

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

B-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 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 **4.2**.

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type sampling tube, undivided (see Fig. 2), 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.

B-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 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.

B-3. SCALE OF SAMPLING

B-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.

B-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.

B-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 : 4905-1968* 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

^{*}Methods for random sampling.



FIG. 2 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(Clause	B-3	.2)

Lot Size	No. of Containers to be Selected
(N)	(n)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 and above	. 8

and n the sample size respectively. Every rth container thus counted shall be withdrawn so

as to give the required sample size.

B-4. TEST SAMPLE AND REFEREE SAMPLE

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

B-4.2 From each of these individual portions (B-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.

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B-5. NUMBER OF TESTS

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

B-6. CRITERIA FOR CONFORMITY

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

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AMENDMENT NO. 1 NOVEMBER 1992 TO IS 5158:1987 SPECIFICATION FOR PHTHALIC ANHYDRIDE, TECHNICAL

(Second Revision)

(Page 7, clause A-7.2, line 2) — Substitute '10 g \pm 0.1 mg' for '5 g \pm 0.1 mg'.

(PCD 9)

Reprography Unit, BIS, New Delhi, India