IS 6972 (1973): Benzotrichloride, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard

SPECIFICATION FOR BENZOTRICHLORIDE, TECHNICAL

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SPECIFICATION FOR BENZOTRICHLORIDE, TECHNICAL

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0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 July 1973, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Benzotrichloride ($C_6H_5.CCl_3$) also known as α, α, α-trichlorotoluene, toluene trichloride, benzenyl trichloride, benzoic trichloride and phenyl chloroform is mainly used in the dye industry.

0.3 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.

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1. SCOPE

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

2. REQUIREMENTS

2.1 Description — The material shall be clear and colourless to yellowish liquid. It has characteristic penetrating odour. It slowly hydrolyzes to benzoic acid.

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

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR BENZOTRICHLORIDE

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Methods of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Relative density* at 27°C/27°C	1·366 to 1·367†	A-2
ii)	Distillation range at 760 mmHg	5 to 95 ml shall distil within the range of 2·5°C including 215°C	A-3
iii)	Freezing point, *Min*	— 6°C	A-4
iv)	Benzotrichloride content, percent by mass, *Min*	95	A-5
v)	Nuclear chlorine (as Cl), percent by mass, *Max*	0·5	A-6

*This term is used in the same sense as specific gravity with water as reference substance, as adopted by the International Organization for Standardization (ISO).

†The relative density determined at any temperature within the range 25 to 35°C can be adjusted to 27°C/27°C by using the correction factor of + 0·000 8 for every degree celsius fall and — 0·000 8 for every degree celsius rise in temperature.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in glass carboys duly provided in hampers or any other containers as agreed to between the purchaser and the supplier.

3.2 Marking — The material shall be marked with the following information:

a) Name of the material;

b) Manufacturer's name and his recognized trade-mark, if any;

c) Net mass of the material; and

d) Lot or batch number, in code or otherwise.

3.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and Rules and Regulations made thereunder. The ISI 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 ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn and their conformities to the requirements of this standard be judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2 and *Table* 1)

METHODS OF TEST FOR BENZOTRICHLORIDE, TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

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

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 Outline of the Method — In this method, masses of equal volumes of the material and water at the same temperature are compared using relative density bottle.

A-2.1 Apparatus

A-2.1.1 *Relative Density Bottle* — 25 ml capacity.

A-2.1.2 *Water-Bath* — maintained at 27·0 ± 0·2°C.

A-2.1.3 *Thermometer* — Any convenient thermometer of a suitable range with 0·1 or 0·2 deg subdivisions.

A-2.2 Procedure — Clean and dry the relative density bottle, weigh and then fill with recently boiled and cooled water at 27°C. Fill to overflowing by holding the relative density bottle on its side in such a manner as to prevent entrapment of air bubbles. Insert the stopper and immerse in a water-bath maintained at 27 ± 0·2°C. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool and weigh. Calculate the mass of water. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

*Specification for water, distilled quality (*revised*).

A-2.3 Calculation

$$\text{Relative density at } 27°C/27°C = \frac{A - B}{C - B}$$

where

A = mass in g of the relative density bottle filled with the material,

B = mass in g of the clean and dry relative density bottle, and

C = mass in g of the relative density bottle filled with water.

A-3. DETERMINATION OF DISTILLATION RANGE

A-3.1 Procedure — Determine the distillation range following the procedure given in IS : 5298-1969*.

A-4. DETERMINATION OF FREEZING POINT

A-4.1 Procedure — Determine the freezing point following the procedure as given in Method B of IS : 1448 (P : 11)-1967†.

A-5. DETERMINATION OF BENZOTRICHLORIDE CONTENT

A-5.0 Outline of the Method — Benzotrichloride is hydrolyzed to sodium benzoate, which is titrated with standard hydrochloric acid in presence of ether which removes benzoic acid from the solution being titrated, as soon it is produced. This prevents the benzoic acid from interference in the titration.

A-5.1 Reagents

A-5.1.1 *Sodium Hydroxide Solution* — approximately 0·5 N.

A-5.1.2 *Dilute Hydrochloric Acid* — approximately 0·5 N.

A-5.1.3 *Standard Hydrochloric Acid* — 0·1 N.

A-5.1.4 *Phenolphthalein Indicator Solution* — Dissolve 0·1 g of phenolphthalein in 100 ml of 60 percent (*v/v*) rectified spirit.

A-5.1.5 *Bromophenol Blue Indicator Solution* — Dissolve 1 g of bromophenol blue in 20 ml of warm ethanol, and dilute with water to 100 ml.

A-5.1.6 *Ether*

A-5.2 Procedure — Weigh accurately about 0·6 g of the material in a 250-ml round-bottom flask and add to it 50 ml of 0·5 N sodium hydroxide solution. Reflux the above mixture for about 2 hours till it

*Method for determination of distillation range and of distillation yield.

†Method of test for petroleum and its products: P 11 Freezing point (*first revision*)

completely goes into solution. Wash the condenser into the flask after every 30 minutes with about 5 ml of distilled water and shake it thoroughly. Cool the content of the flask to room temperature and neutralize with 0·5 N hydrochloric acid solution, using phenolphthalein as indicator. Then add to this 50 ml of ether and 2 ml of bromophenol blue indicator and titrate with standard hydrochloric acid solution with vigorous shaking till the colour of the aqueous layer starts changing. Remove the ether layer and wash it with about 20 ml of water and mix the washings with the aqueous layer. Add 20 ml of ether to the collective aqueous layer, and titrate further with standard hydrochloric acid until end point is reached.

A-5.3 Calculation

$$\text{Benzotrichloride, percent by mass} = \frac{19{\cdot}55\ VN}{M}$$

where

V = volume in ml of standard hydrochloric acid solution consumed,

N = normaly of standard hydrochloric acid solution, and

M = mass of the material taken for test.

A-6. DETERMINATION OF NUCLEAR CHLORINE

A-6.0 Outline of the Method — The material dissolved in isopropyl alcohol is treated with metallic sodium and refluxed. Under the conditions the organically bound chlorine gets converted to ionizable sodium chloride. The contents are taken up with water, acidified, and treated with known volume of silver nitrate solution. The total chlorine is then determined from the amount of silver nitrate solution used up by titrating the excess silver nitrate solution with standard ammonium thiocyanate solution, using ferric alum solution as an indicator. Similarly, the chlorine in side chain is determined by refluxing the material with ethanolic sodium hydroxide solution and following the above procedure. The chlorine in the nucleus is determined by subtracting the chlorine in side chain from the total chlorine.

A-6.1 Reagents

A-6.1.1 *Isopropyl Alcohol* — conforming to IS : 2631-1964*.

A-6.1.2 *Aqueous Isopropyl Alcohol* — 50 percent (*v/v*).

A-6.1.3 *Metallic Sodium* — pure, in the form of ribbon, or cut in small pieces.

*Specification for isopropyl alcohol.

A-6.1.4 *Dilute Nitric Acid* — 1 : 3 (*v/v*).

A-6.1.5 *Standard Silver Nitrate Solution* — 0·1 N (*see* IS : 2316-1968*).

A-6.1.6 *Ferric Alum Indicator Solution* — Saturate or dissolve 8 g of ferric ammonium sulphate in 400 ml of water containing 3 to 5 ml of concentrated sulphuric acid (conforming to IS : 266-1961†).

A-6.1.7 *Nitrobenzene*

A-6.1.8 *Standard Ammonium Thiocyanate Solution* — 0·1 N (*see* IS : 2316-1968*).

A-6.1.9 *Standard Ethanolic Sodium Hydroxide Solution* — 0·5 N, free from chlorine.

A-6.2 Procedure

A-6.2.1 *Total Chlorine* — Take 50 ml of isopropyl alcohol in a 250-ml round-bottom flask and add 0·2 to 0·3 g of material weighed accurately. Add 3 g of metallic sodium slowly, till there is no reaction. Connect the flask to a reflux condenser and boil vigorously for 1 hour, shaking the flask occasionally. Dissolve the excess metallic sodium by cautiously adding 10 ml of aqueous isopropyl alcohol through the condenser at the rate of one or two drops per second. Boil for additional 10 minutes and then add about 50 ml of water. Cool to room temperature and disconnect the flask from the condenser. Acidify the contents with dilute nitric acid until just acidic (blue litmus paper turning red). Add exactly 50 ml of standard silver nitrate solution, followed by 10 ml of nitrobenzene (*see* Note) and 2 to 3 ml of ferric alum indicator solution.

NOTE — Nitrobenzene is very hazardous when absorbed through skin or when its vapour is inhaled. Such exposure may cause anemia. Do not get nitrobenzene in the eyes, on the skin or on clothing. Avoid breathing its vapour. Use only with adequate ventilation.

Add standard ammonium thiocyanate solution slowly from the burette to the contents in the flask with constant swirling. At the first appearance of red colour, stopper the flask tightly and shake vigorously for 15 seconds to coagulate and remove any precipitate from aqueous phase (*see* Note).

NOTE — Care should be exercised in opening the flask after shaking. Pressure may develop causing small amounts of acid solution to be sprayed from the mouth of the flask as the stopper is removed.

Continue titration slowly until the end point of brick-red colour of aqueous phase is reached. Carry out a blank determination simultaneously following exactly the same procedure as used for the material except the omission of the material.

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

†Specification for sulphuric acid (*revised*).

A-6.2.1.1 *Calculation*

$$\text{Total chlorine (as Cl), percent by mass } (X_1) = \frac{(V_1 - V_2)\,N \times 3{\cdot}546}{M}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution used in the blank determination,

V_2 = volume in ml of standard ammonium thiocyanate solution used in the test with the material,

N = normality of standard ammonium thiocyanate solution, and

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

A-6.2.2 *Chlorine in Side Chain* — Weigh accurately 0·2 to 0·3 g of the material and take in a 250-ml round-bottom flask. Add 50 ml of standard ethanolic sodium hydroxide solution and reflux for 2 hours. Cool the solution, acidify with dilute nitric acid. Add 50 ml of standard silver nitrate solution followed by 10 ml of nitrobenzene (*see* Note under **A-6.2.1**), and 2 to 3 ml of ferric alum indicator solution. Titrate the excess silver nitrate solution against standard ammonium thiocyanate solution to the end point of appearance of brick-red colour of the aqueous phase. Carry out a blank determination.

A-6.2.2.1 *Calculation*

$$\text{Chlorine in side chain, percent by mass } (X_2) = \frac{(V_1 - V_2)\,N \times 3{\cdot}546}{M}$$

where

V_1 = volume in ml of standard ammonium thiocyanate solution used in the blank determination,

V_2 = volume in ml of standard ammonium thiocyanate solution used in the test with the material,

N = normality of standard ammonium thiocyanate solution, and

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

A-6.3 Result — Report the difference of X_1 and X_2 as the percentage by mass of chlorine in the nucleus.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF BENZOTRICHLORIDE, TECHNICAL

B-1. GENERAL REQUIREMENTS FOR SAMPLING

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

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precaution shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

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 suitable, clean, dry and air-tight glass bottles or other containers on which the material has no action.

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

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and details given under **3.2.**

B-2. SAMPLING INSTRUMENT

B-2.1 The following sampling instrument may be used.

B-2.1.1 *Sampling Tube* — It is made of thick glass, 20 to 40 mm in diameter and 400 to 800 mm in length (*see* Fig. 1). The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For small containers the size of the sampling tube may be suitably altered.

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 to consist of different batches of manufacture, the batches shall be marked separately and the containers in each batch shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot

separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

B-3.3 In order to ensure randomness of selection, the following procedure is recommended for use:

Arrange all the containers in the lot in a systematic manner and starting from any one count them as 1, 2, 3,....., up to r and and so on, where r is the integral part of N/n (N and n being the lot size and sample size, respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

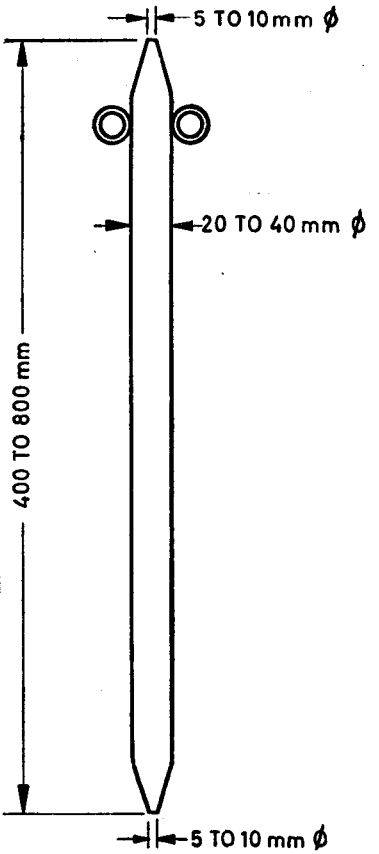

FIG. 1 SAMPLING TUBE

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM LOTS OF DIFFERENT SIZES

(*Clause* B-3.2)

LOT SIZE	SAMPLE SIZE
N	*n*
(1)	(2)
Up to 15	3
16 ,, 40	4
41 ,, 65	5
66 ,, 110	7
111 and above	10

NOTE — When the number of containers is 3 or less all the containers shall be taken for sampling.

B-4. COMPOSITE SAMPLE

B-4.1 From each of the containers selected according to **B-3.2** a representative portion of the material drawn from different parts of the containers shall be drawn with the help of the sampling tube. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample of about 1 500 ml in volume. The 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. These shall be transferred to clean containers and labelled as in full identification particulars of the sample.

B-5. TESTS

B-5.1 Tests for the determination of all characteristics given in **2.1** and Table 1 shall be carried out on the composite sample.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For declaring the conformity of the lot to this specification, the test results on the composite sample shall meet the corresponding requirements specified in the standard.

INDIAN STANDARDS

ON

ORGANIC CHEMICALS (MISCELLANEOUS) MATERIALS

IS:

245-1970	Trichloroethylene, technical (*second revision*)
501-1963	Oxalic acid, technical and analytical reagent (*revised*)
716-1970	Pentachlorophenol (*first revision*)
717-1969	Carbon disulphide, technical (*first revision*)
718-1970	Carbon tetrachloride (*first revision*)
869-1969	Ethylene dichloride (*first revision*)
880-1956	Tartaric acid
3321-1965	Formaldehyde solution
4105-1967	Styrene (vinyl benzene)
4906-1973	Hexamethylenetetramine (hexamine) (*first revision*)
4966-1968	Methylene chloride (dichloromethane), technical
5149-1969	Maleic anhydride, technical
5158-1969	Phthalic anhydride, technical
5254-1969	Acetanilide
5271-1969	Paraformaldehyde
5295-1969	Ethylene glycol
5296-1969	Chloroform, technical and analytical
5297-1969	Perchloroethylene (tetrachloroethylene), technical
5341-1969	Benzyl chloride, technical
5464-1970	Citric acid, monohydrate
5573-1969	Ethylene oxide
5591-1969	Chlorobenzene
5592-1969	Monochloroacetic acid
5992-1970	*p*-Dichlorobenzene, technical
6393-1971	α-Phenylacetamide
6412-1971	Benzoyl chloride, technical
6515-1972	Sodium pentachlorophenate, technical
6712-1972	*o*-Dichlorobenzene
6716-1972	Benzoic acid, technical
6718-1972	Phenoxyacetic acid
6775-1973	Ethyl chloride, technical

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