BUREAU OF INDIAN STANDARDS

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भारतीय मानक मसौदा

2,6-डाई-टर्शियरी ब्यूटाइल फिनॉल — विशिष्टि

Draft *Indian Standard*

2,6-DI-TERTIARY BUTYL PHENOL — SPECIFICATION

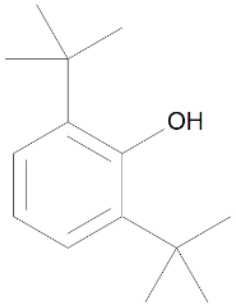
(ICS 71.080.90)

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09	Last date for Comments: **6 January 2025**

FOREWORD

(*Formal clauses to be added later*)

2,6-Di-*tert*-butylphenol [2,6-$(CH_3)_3C)_2C_6H_3OH$] is an organic compound with the following structural formula:



2,6-DTBP is used as an intermediate to manufacture antioxidants that are used in polymers like polypropylene, polyethylene, polystyrene and other products. 2,6-DTBP It is also used directly as an antioxidant in lubes, jet fuels, metal cutting fluids.

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

This standard prescribes the requirements, methods of sampling and testing of 2,6-di-tertiary butyl phenol for industrial uses.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provisions of the standards. 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 given below:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 2362 : 1993	Determination of water by the Karl Fischer method — Test method (*second revision*)
IS 4905 : 2015/ ISO 24153 : 2009	Random sampling and randomization procedures(*first revision*)
IS 5762 : 1970	Methods for determination of melting point and melting range
IS 8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (*second revision*)

3 REQUIREMENTS

3.1 Description

Material shall be white solid below 35 ºC and colourless to pale yellow liquid at 40 ºC and shall be free from any foreign matter.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the method prescribed in col (4) and col (5) of Table 1.

Table 1 Requirements for 2,6-Di-tertiary Butyl Phenol
(*Clause* 3.2)

Sl No.	**Characteristics**	**Requirement**	**Methods of Test, Ref to**	
			Annex	Indian Standards
(1)	(2)	(3)	(4)	(5)
i)	Purity (by GC), percent by mass, *Min*	99.5	A	—
ii)	Moisture content, percent by mass, *Max*	0.03	—	IS 2362
iii)	Colour, Pt-Co, *Max*	20	—	IS 8768

iv)	Melting point, °C	36.0 to 40.0	B	IS 5762
v)	Total impurities, percent by mass, *Max*	0.3	A	—

3.3 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

4 PACKING AND MARKING

4.1 Packing

The material shall be packed and supplied in ISO tankers or stainless-steel tankers or galvanized iron drums (*see* IS 2552).

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

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

4.2.2 For supplies of material in bulk, a test certificate containing the details mentioned at **4.2.1** shall be provided for each consignment. The test certificate shall be certified by an authorized person of the manufacturer's organization.

4.2.3 Each container (including containers for bulk transport) shall be suitably marked with the following information:

WARNING

PROTECT FROM LIGHT AND MOISTURE

4.2.4 *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 there under, and the products may be marked with the Standard Mark.

5 SAMPLING

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex C.

ANNEX A

[*Table* 1, *Sl No.* (i) *and* (v)]

DETERMINATION OF 2,6-DI-TERTIARY BUTYL PHENOL (PURITY) CONTENT AND ITS IMPURITIES BY GAS CHROMATOGRAPHIC METHOD

A-1 OUTLINE OF THE METHOD

This method is used to determine purity of the material and also its impurities. A small volume of sample is injected into a gas chromatograph, which then passes through capillary column, where the separation take place. Flame Ionization Detector (FID) is used for detection.

A-2 APPARATUS

A-2.1 Gas Chromatograph

A-2.1.1 Any gas chromatograph equipped with a flame ionization detector (FID), a split injector and a suitable electronic integrator/software can be used with following accessories and operating condition:

Column	:	100 percent dimethylpolysiloxane (PDMS) phase with 30 m length, 0.32 mm internal diameter and 1.0 µm film thickness or equivalent
Column temperature	:	325 °C
Carrier gas	:	Hydrogen
Gas flow rate, ml/min	:	1.2
Total run time min	:	30
Hydrogen flow rate, ml/min	:	40
Air flow rate, ml/min	:	400
Sample size, µl	:	1.0

A-2.1.2 *Temperature Programme of Oven, Detector and Injector:*

Injector Temperature, °C	*Detector Temperature, °C*	*Oven Temperature, °C*	*Oven Hold Time, min*	*Oven Ramp Rate, °C/min*
250	300	90	2	8
		280	5	—

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 standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) 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.

A-3 REAGENTS

A-3.1 2,4-Di-tertiary-butyl Phenol (2,4-DTBP), certified reference material

A-3.2 Phenol, certified reference material

A-3.3 *o*-Tertiary Butyl Phenol (OTBP), certified reference material

A-3.4 *p*-Tertiary Butyl Phenol (PTBP), certified reference material

A-3.5 2,6-Di-tert-butyl Phenol (2,6-DTBP), certified reference material

A-3.6 2,4,6-Tri-tert-butyl Phenol (2,4,6-TTBP), certified reference material

A-3.7 Methanol

NOTE — In case certified reference material of reagents as mentioned at **A-3.1** to **A-3.6** are unavailable, high purity chemicals (known purity) may also be used as an alternative to certified reference material.

A-4 PREPARATION AND CALIBRATION OF STANDARDS

A-4.1 Standard Preparation

To prepare calibration mixture, weigh each component (*see* item **A-3.1** to **A-3.6**) as per the concentration given in Table 2 in a volumetric flask.

A-4.2 Standardization Procedure

Make sure the GC instrument is adjusted to the conditions stated as above. Weigh 2 g standard solution as prepared at **A-4.1** in 10 ml volumetric flask and dilute it with methanol up to the mark. Inject 1.0 µl of the standard mixture, by using syringe or auto sampler, in column taking care that no air bubble is trapped and obtain the chromatogram. Calculate the relative response factors (RRF) of each impurity by dividing the respective area in its chromatogram by concentration of each impurity.

$$\text{Relative Response Factor, F} = \frac{\text{Area of impurity}}{\text{Concentration of impurity}}$$

Results to be declared on area normalization method. Set the system sensitivity so that all impurity peaks are recorded at adequate levels for data acquisition. Normally, the minimum peak height will be twice that of the baseline noise.

Table 2 Typical Concentration and Relative Retention Time (RRT)
(*Clause* A-4.1)

Sl No.	**Compounds**	**Concentration**	**RRT, min**
(1)	(2)	(3)	(4)

i.	Phenol	0.100 1	0.314
ii.	OTBP	0.100 1	0.714
iii.	PTBP	0.100 1	0.752
iv.	2,6-DTBP	0.100 1	1.000
v.	2,4-DTBP	99.59	1.095
vi.	2,4,6-TTBP	0.001	1.238
	Total	100.00	-

A-5 SAMPLE PREPARATION

Weigh 2.0 g sample in 10 ml volumetric flask. Dilute with methanol up to the mark.

A-6 PROCEDURE

Inject 1 µl of sample by using manual or auto sampler, without any air bubble trapped in the syringe. Allow approximately 30 min for components to elute from the column. Determine the mass concentration of all components by area normalization method.

NOTES

1 When 2,6-DTBP elutes, raise the column oven temperature to 280 °C. The column should remain at 280 °C for approximately 5 min.

2 Before another chromatograph run is attempted, stabilize the oven at 90 °C for at least 10 min.

A-7 CALCULATION

A-7.1 Calculate concentrations of each impurity by correcting with respective response factor:

Concentration of an impurity = Area of an impurity x Response factor

A-7.2 Calculate purity by GC by the following formula:

Purity of 2,6-DTBP, percent by mass = 100 – (Sum of all known and unknown impurities)

A-7.3 Calculate absolute purity by the following formula:

Purity of 2,6-DTBP, percent by mass = 100 – (Water content + Sum of all known and unknown impurities)

Table 3 Typical Retention Time

Sl. No. (1)	**Compound** (2)	**RT, min** (3)
i.	Phenol	3.4

ii.	OTBP	7.5
iii.	PTBP	7.9
iv.	2,6-DTBP	10.7
v.	2,4-DTBP	11.3
vi.	2,4,6-TTBP	13.0

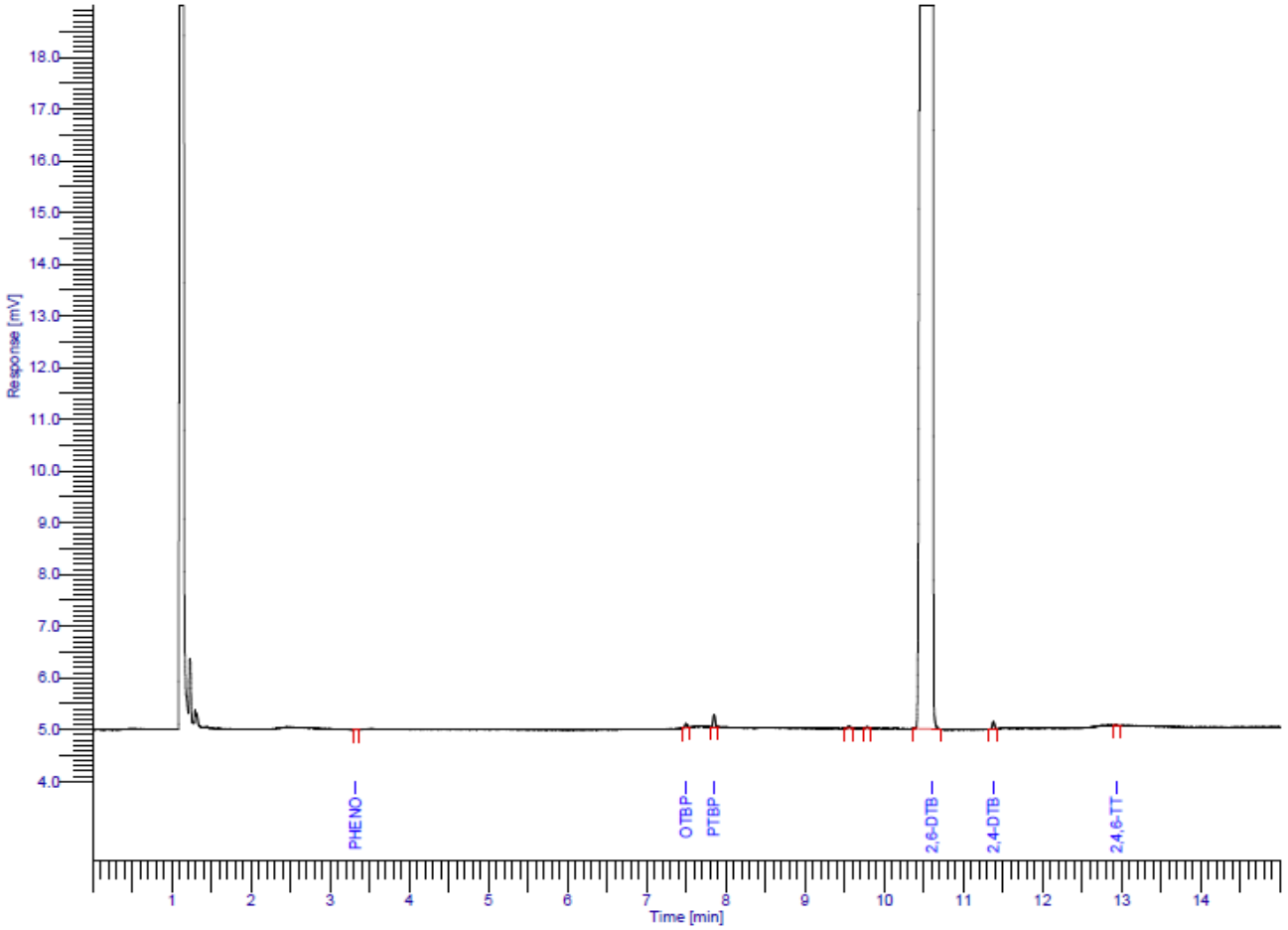


FIG. 3 TYPICAL CHROMATOGRAM

ANNEX B

[*Table* 1, *Sl No*. (iv)]

DETERMINATION OF MELTING POINT

B-1 GENERAL

Melting Point is the temperature at which liquefication of the material occurs, as indicated by the formation of a definite meniscus. This method is used to determination of melting point by heating the sample at a controlled rate in capillary tube.

B-2 APPARATUS

B-2.1 Capillary Melting Point Apparatus, capable with following operating condition:

B-2.1.1 *Melting Point Measurement Range*, 30 °C to 110 °C

B-2.1.2 *Rate of Heating (°C per min)*, 0.2, 0.5, 1, 1.5, 2, 3, 4, 5

B-2.2 Capillary Tubes, made of heat resistant glass with internal diameter 1.0 mm, length 80 mm and outside diameter 1.4 mm.

B-2.3 Thermometer, any convenient thermometer having a range of 0 °C to 110 °C and the least count of 0.1 °C

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

B-3 PROCEDURE

B-3.1 Preparation of Samples

Take 2 g to 5 g of the dry material and grind it in a clean dry porcelain mortar. Take a part of this ground sample and reduce to fine powder in an agate mortar.

B-3.2 Fill the capillary tube with sample up to the height of 3 mm. Now insert the filled capillary into the melting point apparatus and press warming key. The temperature will start rising slowly as per the set value of rate of heating. The graph of light transmittance (y axis) *v/s* melting point temperature (x axis) with initial melting point (S Melt) and Final melting point (F Melt) in degrees Celsius will be displayed. The current temperature reading at the capillary will also be visible. The display screen will record both the initial and final melting values alongside the graph. Once the final melting point is detected, heating will cease, and the temperature will stabilize at the preset initial temperature. Record and report the melting point in deg C.

ANNEX C
(*Clause* 5)
SAMPLING OF 2,6-DI-TERTIARY BUTYL PHENOL

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.1 In drawing, preparing, and handling samples the following precautions shall be observed.

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

C-1.3 The sampling instrument shall be clean and dry when used.

C-1.4 The samples shall be placed in a suitable, clean, dry, air-tight, amber or dark blue glass container or anyother container on which the material has no action.

C-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

C-1.6 Sample shall be protected from light.

C-1.7 Phenolic substance burns the skin and may be absorbed into the system through the skin. The sampler shall wear gloves, preferably of polyvinyl chloride and a face shield. Inhalation of the vapors from hot material shall be avoided.

C-2 SCALE OF SAMPLING

C-2.1 Lot

All the containers in a single consignment of 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 groups of containers in each shall constitute separate lots.

C-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately.

C-3 Sampling from Containers

C-3.1 The number of containers to be selected for sampling shall be in accordance with Table 4.

C-3.2 The container shall be selected at random. In order to ensure randomness of selection, random number tables shall be used (*see* IS 4905).

C-4 PREPARATION OF TEST SAMPLES

C-4.1 When the material consists of crystallized masses, the container selected from the consignment shall be allowed to stand in an open tank, on a grid below which a closed steel coil is fitted. When the material is completely melted stir it thoroughly with a clean, dry, smooth, glass rod, and draw samples from each by means of dry glass sampling tube from different depths, care being taken to reduce atmosphere exposure to the minimum.

Table 4 Number of Containers to be Selected for Sampling from Lots of Different Sizes
(*Clause* C-3.1)

Sl No.	Lot Size	No. of Containers to be Selected
(1)	(2)	(3)
i.	Under 25	5
ii.	25 to 49	5
iii.	50 to 99	10
iv.	100 to 199	15
v.	200 to 299	20
vi.	300 to 499	30
vii.	500 to 799	40
viii.	800 to 1 299	55
ix.	1 300 to 3 199	75
x.	3 200 to 8 000	115

C-4.2 Out of the material drawn from each individual container, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient for carrying out triplicate determinations for all the characteristics specified. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for thereferee.

C-4.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the container selected shall be for the purchaser, another for the supplier and the third forthe referee.

C-4.4 All the individual and composite shall be transferred to separate sample container. This container shall then be sealed air-tight with stoppers and labeled with full identification particulars given in **C-1.5**.

C-4.5 The referee test samples, consisting of a composite sample and a set of individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed between the two,to be used in case of any dispute.

C-5 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

C-5.1 The purchaser may examine and test each of the individual samples separately for compliance with the requirements of this standard, or he may decide at any stage of progress of the examination to test a composite sample (*see* **4.1**) for determining conformity of the lot to this specification.

C-5.2 When the individual samples are separately examined and the results vary from one sample to another, the criteria for judging the quality of the lot for the purpose of acceptance on the results obtained shall be at the discretion of the purchaser, unless otherwise previously agreed to between the purchaser and the supplier.