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Draft Indian Standard

SPECIFICATION FOR BENZYL ALCOHOL

(*First Revision of IS 3924*)

(ICS No. 71.100.60)

Fragrance and Flavour Sectional Committee
PCD 18

Last date for comment is
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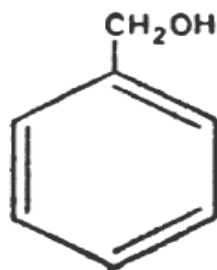
FOREWORD

(*Formal clauses shall be added later*)

This Indian Standard (first revision) was first published in 1966. In this revision, the gas chromatographic analysis for purity determination of vanillin has been upgraded from Packed Column GC to Capillary Column GC for more accurate results. Also the packing and marking clauses have been updated.

Benzyl alcohol (C₇H₈O) and its esters are found in the essential oils of a wide variety of flowers and in balsams obtained from the exudation of trunks of resinous trees. Benzyl alcohol is one of the few chemicals used extensively not only in perfumery but in totally unrelated fields as well, such as pharmaceuticals, lacquers, etc. Large quantities of benzyl alcohol are therefore manufactured of which only a portion is used in the perfumery industry.

Benzyl alcohol has a somewhat weak odour and its main use is as a solvent in perfumes and pharmaceuticals. It is represented by the following structural formula:



BENZYL ALCOHOL
(**Molecular Mass 108.14**)

In the preparation of this standard, considerable assistance has been derived from the Givaudan Index, 1978, published by Givaudan-Delawanna Inc, New York.

Clause 4.3 includes purchaser and seller agreement.

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

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 and the methods of sampling and test for benzyl alcohol used as a solvent in perfumery industry.

2 REFERENCES

The following standards contain provisions which, through reference in text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All the 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 indicated below.

<i>IS No.</i>	<i>Title</i>
IS 326 (Part 2) : 1980	Methods of sampling and test for natural and synthetic perfumery materials: Part ii preliminary examination of perfumery material and samples (<i>Second Revision</i>)
326 (Part 3) : 2006/ ISO 279 : 1998	Method of sampling and test for natural and synthetic perfumery material - Determination of relative density
IS 326 (Part 6) : 2005 ISO 875 :1999	Methods of sampling and test for natural and synthetic perfumery materials: Part 6 evaluation of miscibility in ethanol (<i>Third Revision</i>)
326 (Part 7) : 2006/ ISO 1242 : 1999	Method of sampling and test for natural and synthetic perfumery material - Determination of acid value (<i>Third Revision</i>)
1070 : 1992	Reagent grade water
2284 : 1988	Method for olfactory Assessment of Natural and synthetic perfumery Material
6597 : 2001	Glossary of term related to fragrance and flavour industry (<i>Second Revision</i>)

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 6597 shall apply.

4 REQUIREMENTS

4.1 Description

4.1.1 The material shall generally be obtained by hydrolysis of benzyl chloride.

4.1.2 The material shall be a colourless liquid free from sediment, suspended matter and adulterants.

4.1.3 The material shall be examined for its colour, clarity, suspended matter and sediment as prescribed in IS 326 (Part 2).

4.2 Solubility

4.2.1 *In Ethanol* — The material shall be clearly soluble in **1.5** volumes of ethanol (50 percent v/v), when tested as prescribed in IS 326 (Part 6).

4.2.2 *In water* — one part of the material shall also be soluble in **30** parts of water (by volume) at 27°C.

4.3 The assessment of odour and appearance shall be subject to agreement between the purchaser and seller. The material shall be tested olfactorily, especially for by-odours/ by-notes, and for the presence of adulterants and impurities, if any, as prescribed under **4** and **5** of IS 2284.

4.4 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR BENZYL ALCOHOL
(Clauses 4.4 and 7.1)

Sl no.	CHARACTICRISTIC	REQUIREMENT	METHOD OF TEST, Ref To
(1)	(2)	(3)	(4)
i)	Odour	Faintly aromatic	IS 2284
ii)	Relative density at 27°C/27°C	1.036 to 1.040	IS : 326 (Part 3)
iii)	Refractive index at 27°C	1.535 8 to 1.537 8	IS : 326 (Part 5)
iv)	Acid value, Max	0.3	IS : 326 (Part 7)
v)	Determination of Purity of Benzyl Alcohol percent by mass, <i>Min</i>	99	Annex A

vi)	Freedom from chlorinated compounds	To pass test	Annex B
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5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in glass bottles, or in suitable containers as agreed between the purchaser and the supplier. Aluminium containers shall be avoided. The containers shall be tightly closed and nearly full.

5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

5.2.1 *Name of the material;*

5.2.2 *Name of the manufacturer and his recognized trade-mark, if any;*

5.2.3 *Batch number and date of manufacture; and*

5.2.4 *Net and gross mass.*

5.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian *Standards Act, 2016* and the Rules and Regulations made thereunder. Details of conditions under which a licence for the use of Standard Mark may be granted to manufactures or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material, each sample containing not less than 50 ml shall be drawn as prescribed in IS 326 (Part 1).

7 TEST METHODS

7.1 Tests shall be conducted as prescribed under **4.1, 4.2, 4.3** and the appropriate references specified in col **4** of Table 1.

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (IS 1070 : 1992) shall be employed in tests.

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

ANNEX A
[Table 1, Sl. No. (v)]
GAS CHROMATOGRAPHIC ANALYSIS OF BENZYL ALCOHOL

A-1 GENERAL

The chromatographic conditions given here are for guidance only.

A-2 OUTLINE OF THE METHOD

A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means. The response is related to the amount of a specific component leaving the column.

A-3 APPARATUS

Gas chromatograph equipped with suitable capillary column and flame ionization detector.

A-4 PROCEDURE

Take 1 μL of sample in a GC Vial and inject into gas chromatograph using following operating conditions.

A-5 GC CONDITIONS FOR NON-POLAR COLUMN

Capillary Column	: Fused silica capillary column coated with non-polar stationary phase (100% Polydimethylsiloxane) SH-RXi-1ms
Column temperature	: -40°C to 350°C
Length	: 60 m
Internal diameter	: 0.25 mm
Film Thickness	: 0.25 μm
Injector Temperature	: 240°C
Split Ratio	: 200 : 1
Column flow	: 1 ml/ min
Injection volume	: 0.5 μl
Carrier Gas & Flow	: Helium, at constant pressure of 145 kPa
Make up gas flow	: He – 24ml/min, H ₂ – 32 ml/min, Zero air – 200ml/min
Column oven Temperature	: 60°C to 230°C @ 4°C/min final hold time 20 min.
Detector type	: FID
Detector Temperature	: 290°C
Run time	: 62.5 min

A-6 GC CONDITIONS FOR POLAR COLUMN

Column	: Fused silica capillary column coated with Polar stationary phase (Poly ethylene glycol) SH- Stabilwax
Column temperature	: -20°C to 260°C
Length	: 60 m
Internal diameter	: 0.25 mm
Film Thickness	: 0.25 µm
Injector Temperature	: 240°C
Split Ratio	: 200 : 1
Column flow	: 1.14 ml/ min
Injection volume	: 0.5µl
Carrier Gas & Flow	: Helium, at constant pressure of 158 kPa
Make up gas flow	: He – 24ml/min, H ₂ – 32 ml/min, Zero air – 200ml/min
Column oven Temperature	: 60°C to 230°C @ 4°C/min final hold time 25 min.
Detector type	: FID
Detector Temperature	: 250°C
Run time	: 67.5 min

NOTE — The above gas chromatographic conditions are suggestive /typical. However, any GC with equivalent column may be used provided standardization/calibration are done after setting up chromatographic conditions for the desired/required resolution.

A-7 CALCULATION —

Calculate are percent of each peak by following formulas

Polar and non-polar columns

$$\text{Area \% of individual} = \frac{\text{Peak area of the individual}}{\text{sum of areas of all the peak in the chromatogram}} \times 100$$

NOTE — The modern instruments are equipped with the software, which automatically calculates area percent of each peak.

CHROMATOGRAMS FOR POLAR AND NON-POLAR COLUMN WOULD BE PROVIDED BY CSIR-CIMAP.

ANNEX B
[Table 1, Sl. No. (vi)]
TEST FOR FREEDOM FROM HLORINATED COMPOUNDS

B-1 GENERAL

B-1.1 OUTLINE OF THE METHOD — Absence of even a transient green colour, when the material is ignited on a copper gauze in a non-luminous flame is used for determining freedom from chlorinated compounds.

B-2 APPARATUS

B-2.1 Copper Wire — bent at one end to which a strip of 850-micron copper gauze 1.5 cm wide and 5 cm long is attached.

B-2.2 Dropper

B-2.3 Bunsen Burner - capable of giving good non-luminous flame.

B-3 PROCEDURE

B-3.1 Place the copper strip in the non luminous flame of the Bunsen burner until it glows without imparting a green colour. Cool the gauze and repeatedly ignite it until an oxide coating has formed. Cool the gauze and add 2 drops of the sample by means of a dropper, permitting it to burn in the air. Again cool and add 2 more drops of the test material and burn as before. Continue the procedure until 6 drops have been ignited. Hold the gauze in the outer edge of the non-luminous flame whose height has been adjusted to about 4 cm.

B-3.1 The flame shall be free of even a transient green colour.