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भारतीय मानक मसौदा

प्राकृतिक मेन्थॉल — विशिष्टि

(IS 3134 का दूसरा पुनरीक्षण)

Draft Indian Standard

NATURAL MENTHOL — SPECIFICATION

(Second Revision of IS 3134)

(ICS 71.100.60)

Fragrance and Flavour Sectional Committee,
PCD 18

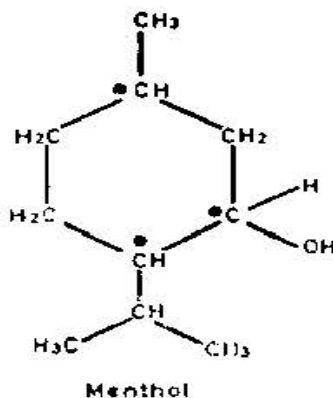
Last date for receipt of comment is
08 March 2025

FOREWORD

(Formal clauses will be added later)

This standard was originally published in 1965 and the first revision was done in 1992 to align with the trade practices prevalent in the perfumery industry and also to align it with the quality level of material being produced and sold in the country.

Menthol (C₁₀H₂₀O) contains three asymmetric carbon atoms* (*see* formula given below). This cyclic, saturated, secondary alcohol may, therefore, exist in four externally compensated and eight optically active forms:



(1-Methyl-4- isopropylcyclohexane-3-ol,
Molecular mass 156.27)

This standard, however, deals with only, laevo-rotatory 1-menthol occurring naturally. In the earlier version, racemic dl-menthol, produced synthetically, was also covered which is no longer commercially available, and hence has been deleted in first revision.

Menthol occurs in the Japanese type oil of mint distilled from *Mentha arvensis*. L which contains 65-70 percent 1-Menthol.

In India, a phenomenal development for the production of 1-Menthol from oil of *Mentha arvensis* and its improved varieties like KOSI, CIM-UNNATI, etc. has taken place during the period up to 2024. Presently, KOSI oil is the main source for the production of 1-menthol. KOSI mentha oil contains 75-78 percent 1-menthol. 1-Menthol is usually obtained by fractional cooling of the mentha oils from + 15 °C to – 40 °C and subsequently separating the cake of (-) -menthol by centrifuging. Large and bold crystals of 1-menthol are then obtained by controlled slow cooling crystallization.

1-Menthol produced from natural sources is available in the form of molten, solid mass, powder, flakes, small crystals, and big crystals. 1-Menthol is also manufactured synthetically for which a separate Indian Standard IS 18250 is available.

Natural menthol is largely used for flavouring in candies, pan masala and chewing gums, in the compounding of pharmaceutical preparations, in the flavouring of tooth pastes, mouth washes, oral preparations, in the preparation of mentholized cigarettes, cosmetic and personal care, etc.

In the first revision, a new requirement of purity of menthol content by GC was included and the requirement for melting range was revised based on data generated through indigenous testing. The requirement for a congealing point was deleted.

The second revision has been brought out to keep pace with the latest technological developments and international practices. In this revision, the following major changes have been made:

- a) The title of the standard has been changed from menthol to natural menthol.

- b) Various forms of natural menthol have been incorporated into the scope of the standard.
- c) Description of the material has been modified.
- d) Improved varieties like KOSI, CIM-UNNATI have been incorporated.
- e) Specific requirement of the specific rotation has been modified.
- f) Specific requirements of colour and appearance have been modified.
- g) New requirement of bio based carbon (p MC Value) and method of test for determination of bio based carbon have been added to identify source of the product.
- h) Equipment's have been added in the Annex B

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 natural menthol crystals, molten, solid mass, flakes and powder extracted from naturally occurring substances.

2 REFERENCES

The standards listed in Annex A 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 listed in Annex A.

3 TERMINOLOGY

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

4 REQUIREMENT

4.1 Description

4.1.1 The material shall consist of colourless/white, acicular or prismatic crystals molten, solid mass, flakes, powder with a penetrating peppermint like odour.

4.1.2 The material extracted from naturally occurring substances. When placed on the tongue the material shall impart a biting sensation and a slightly bitter taste followed by a pleasant feeling of cold.

4.1.3 The material shall be tested olfactory and especially for by-odours/by-notes as prescribed in IS 2284. The assessment of odour and appearance shall be subject to agreement between the purchaser and seller.

4.2 Identification Tests

4.2.1 A five percent (*m/v*) solution of the material in ethyl alcohol (90 percent by volume) shall be neutral to litmus solution.

4.2.2 Dissolve 10 mg of the material in 1 ml of sulphuric acid and add 1 ml of a solution of vanillin in sulphuric acid. An orange-yellow colour shall be produced which, on addition of 1 ml of water, shall further change to violet colour.

4.3 Freedom from Thymol and Other Phenols

4.3.1 Dissolve a few quantity of the material in 1 ml of glacial acetic acid, add three drops of sulphuric acid and one drop of nitric acid. No green colour shall develop.

4.3.2 No colour due to the presence of phenolic substances shall be produced when a solution of the material in ethyl alcohol (90 percent by volume) is added to an aqueous solution of ferric chloride.

4.4 Solubility

The material shall be soluble in all proportions of ethyl alcohol (90 percent by volume) when tested as prescribed in IS 326 (Part 6).

4.5 The material shall also comply with the requirement of Table 1.

Table 1 Requirements for Natural Menthol
(Clause 4.5)

Sl. No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Colour and appearance	Colourless/white acicular or prismatic crystals, molten, solid mass, flakes, and/or powder	IS 326 (Part 2)
ii)	Melting range °C	42 to 44	IS 326 (Part 16)
iii)	Specific rotation at 20 °C (± 1 °C), (Concentration 0.020 g/ml; MeOH)	- 47° to - 50°	IS 326 (Part 4)
iv)	Non-volatile matter, Percent by mass, <i>Max</i>	0.05	Annex B
v)	Menthol content, percent By mass, <i>Min</i>	99.0	Annex C
vi)	Bio based carbon, percent	100	Annex D

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be supplied in well closed containers permitting a minimum of air space, as agreed to between the purchaser and manufacturer/supplier.

5.1.2 The material shall be well protected from light and stored in a cool place.

5.2 Marking

5.2.1 Each container so filled shall be clearly marked with the following information:

- a) Name of the material;
- b) Name of manufacturer and recognized trade mark, if any;
- c) Net and Gross weight of the material;
- d) Lot No./ Batch No. and date of manufacturing; and
- e) Self-life DD/MM/YY or best before by DD/MM/YY.

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

Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

7 TESTS

7.1 Tests shall be carried out as prescribed in **4.1** to **4.4** and in col 4 of Table 1.

7.2 Quality of Reagents

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

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>
IS 326	Methods of sampling and test for natural and synthetic perfumery materials
(Part 1) : 2022	Sampling (<i>fourth revision</i>)
(Part 2) : 2023	Preliminary examination of perfumery materials and samples (<i>third revision</i>)
(Part 4): 2005	Determination of optical rotation (<i>third revision</i>)
(Part 6): 2005	Evaluation of miscibility in ethanol (<i>third revision</i>)
(Part 16): 1989	Determination of melting point (<i>second revision</i>)
IS 1070: 2023	Reagent grade water – Specification (<i>fourth revision</i>)

IS 2284 : 1988	Method of olfactory assessment of natural and synthetic perfumery materials (<i>first revision</i>)
IS 6597: 2001	Glossary of terms relating to fragrance and flavour industry (<i>second revision</i>)

ANNEX B

[(Table 1 and Sl No. iv)]

DETERMINATION OF NON-VOLATILE MATTER

B-1 APPARATUS

B-1.1 Analytical balance — Capable in weighing with least count 0.001g

B-1.2 Hot air oven — Capable maintaining temperature with least count 1 °C

B-1.3 Platinum or silica dish — about 25 mm in diameter.

B-1.4 Desiccator

B-2 PROCEDURE

Place 5 g of the material in an accurately weighted platinum or silica dish, and place it on a water-bath. Continue heating till most of the material has volatilized. Transfer the dish to a hot air-oven maintained at (105 ± 1) °C and heat to constant mass. Cool in a desiccator and weigh it.

B-2.1 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{a \times 100}{b}$$

where

a = mass in g of the residue, and

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

ANNEX C

[(Table 1 and Sl No. (v)]

DETERMINATION OF MENTHOL CONTENT USING GAS CHROMATOGRAPH

C-1 GENERAL

The chromatographic conditions given here are for guidance only.

C-1.1 Outline of the Method

A sample of the material is dissolved in a suitable solvent 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, the 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 whose response is related to the amount of a specific component leaving the column.

NOTE — Hexane, cyclohexane and petroleum ether found more suitable solvent.

C-2 APPARATUS

C-2.1 Any suitable gas chromatograph and column, capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks, may be used. The typical chromatogram for natural menthol with the following chromatographic conditions is shown in Fig. 1.

Sample	Natural Menthol
Column:	
Material	Stainless steel
Length	3m
Orifice	D.32 cm
Stationary phase and solid support	FFAP ¹⁾ 10 percent by mass on Chromosorb WHP, 100 -120 mesh
Carrier gas	Nitrogen
Conditions:	
Column temperature	150 °C
Injection port temperature	210 °C
Detector:	
Type	FID
Temperature	230°C

¹⁾It denotes the free fatty acid phase (FFAP) in carbowax 20 M treated with nitro phthalic acid.

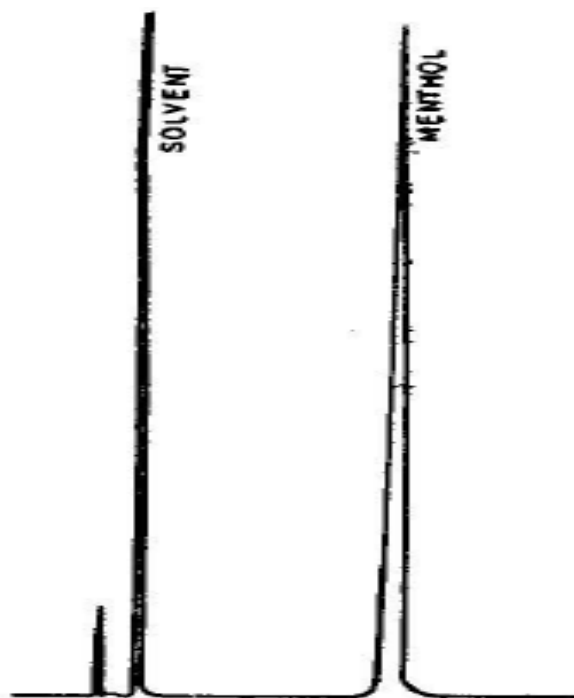


FIG. 1 A TYPICAL CHROMATOGRAM FOR MENTHOL

ANNEX D

[(Table 1 and SI No.vi)]

DETERMINATION OF BIOBASED CARBON USING ACCELERATOR MASS SPECTROMETRY

D-1 GENERAL

D-1.1 Biobased ^{14}C testing is an accurate tool for differentiation of natural menthol from fossil-derived synthetic menthol using accelerator mass spectrometry method.

D-1.2 The living carbon-based materials (present-day) have a strong ^{14}C signature because it has not yet undergone significant radioactive decay and will yield a biobased index of 100 percent. When a plant or animal dies, the amount of ^{14}C in its composition begins to slowly decrease over time at a known rate. For fossil material, the quantities of ^{14}C are much lower, since life activity ceased in the distant past and will therefore be ZERO percent biobased. Measuring ^{14}C content of any natural products is an accurate tool to determine if adulterated with any fossil-derived materials.

D-2 APPARATUS AND REAGENTS

D-2.1 Accelerator Mass Spectrometer (AMS) Apparatus

Biobased evaluation of the samples was done by quantification of ^{14}C content as per the standard protocol ASTM D6866–22 (Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis). It is noteworthy that ISO 16620-2 is equivalent to this standard. Ion accelerator (500kV) based Compact ^{14}C Accelerator Mass Spectrometer (AMS) system shall be used for the quantification of $^{14}\text{C}/^{12}\text{C}$ present in each of the sample.

NOTE — A Pelletron accelerator (500 kV) based Compact ^{14}C Accelerator Mass Spectrometer or NEC XCAMS (National Electrostatic Corp. 1.5SDH tandem accelerator) was used for $^{14}\text{C}/^{12}\text{C}$ ratio measurements in samples and results are normalized to the standard sample OX II.

D-2.2 Sample Preparation

Processed sample were combusted in automated graphitization equipment (AGE) and converted into graphite powder for enabling AMS measurements to quantify $^{14}\text{C}/^{12}\text{C}$ ratio. Method B of ASTM D6866–22 can be used for preparation of graphite prior to dating measurements.

D-2.3 Analysis, Interpretation, and Reporting

D-2.3.1 $^{14}\text{C}/^{12}\text{C}$ and $^{13}\text{C}/^{12}\text{C}$ isotopic ratios are measured using AMS. The isotopic ratios of $^{14}\text{C}/^{12}\text{C}$ or $^{13}\text{C}/^{12}\text{C}$ are determined relative to a standard traceable to the NIST SRM 4990C (oxalic acid) modern reference standard. The calculated “fraction of modern” (fM) represents the amount of ^{14}C in the product or material relative to the modern standard. This is most commonly referred to as percent modern carbon (pMC), the percent equivalent to fM (for example, fM 1 = 100 pMC).

D-2.3.2 All pMC values obtained from the radiocarbon analyses must be corrected for isotopic fractionation using stable isotope data. Correction shall be made using $^{13}\text{C}/^{12}\text{C}$ values determined directly within the AMS where possible.

D-3 pMC CALCULATIONS

D-3.1 The pMC can be greater than 100 percent due to the continuing, but diminishing effects from injection of ^{14}C into the atmosphere with atmospheric nuclear testing programs.

D-3.2 The first version of this standard (ASTM D6866-04) in 2004 referenced a value of 107.5 pMC and the ASTM D6866-10 version (2010) cited 105 pMC. These data points equate to a decline of 0.5 pMC per year. Therefore, on January 2 of each year, the values in Table 2 are used as given in D-3.3

D-3.3 REF through 2024, reflecting 0.3 pMC decrease per year.

D-3.4 It is calculated by dividing pMC by REF and multiplying the result by 100 for example, $[102 (\text{pMC})/102 (\text{REF})] \times 100 = 100$ percent biobased carbon.

D-3.5 Results are reported as percent biobased carbon content or percent biogenic carbon content rounded to the nearest 1 unit with an applied error of 3 percent absolute (Table 4).

D-4 DATA INTERPRETATION

D-4.1 Zero pMC

Represents the entire lack of measurable ^{14}C atoms in a material above background signals thus indicating a fossil (for example, petroleum based) carbon source. Therefore, fossil derived menthol will give a zero pMC value.

D-4.2 If it is a Mixture

A pMC value between 0 and 100 indicates a proportion of carbon derived from fossil vs. modern source.

D-4.3 Reference pMC

A value of 100 pMC indicates an entirely modern carbon source are given in Table 2

Table 2 Reference pMC
(Clause D-4.3)

SI No.	Year	Ref (pMC)
(1)	(2)	(3)
i)	2015	102.0
ii)	2016	101.5
iii)	2017	101.0
iv)	2018	100.5
v)	2019	100.0
vi)	2020	100.0
vii)	2021	100.0
viii)	2022	100.0
ix)	2023	100.0
x)	2024	99.7
xi)	2025	99.4
xii)	2026	99.1

Measured results for all the samples are normalized to the standard sample OX II.

D-4.3.1 AMS delta ^{13}C values are used for the isotopic fractionation correction.

D-4.3.2 Background value during the measurement should be clearly reported. For example, (0.694 ± 0.0242) pMC (Percentage modern carbon) and that corresponds to $^{14}\text{C}/^{12}\text{C}$ ratio $(7.6341 \pm 0.2657) \times 10^{-15}$.

D-4.3.3 Data quality must be monitored with secondary standard sample (IAEA-C7). For example, during measurement, its consensus values (pMC = 49.53 ± 0.12) should match with its experimental result (pMC = 49.46 ± 0.25) and should be within the error range.