भारतीय मानक Indian Standard

प्राकृतिक मेन्थॉल — विशिष्टि

IS 3134: 2025

(दूसरा पुनरीक्षण)

Natural Menthol — Specification

(Second Revision)

ICS 71.100.60

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Price Group 6

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Fragrance and Flavour Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Menthol ($C_{10}H_{20}O$) 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 Weight: 156.27

Menthol occurs in the Japanese type oil of mint distilled from *Mentha arvensis*. *L*, which contains 65 percent to 70 percent l-menthol. l-Menthol produced from natural sources is available in the form of molten, solid mass, powder, flakes, crystals or equivalent. l-Menthol is also manufactured synthetically for which a separate 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 toothpastes, mouth washes, oral preparations, in the preparation of mentholized cigarettes, cosmetic and personal care, etc.

This standard was first published in 1965 and was subsequently revised 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. This standard, however, deals with only, levorotatory, l-menthol occurring naturally. In the original version, racemic dl-menthol, produced synthetically, was also covered which is no longer commercially available, and hence was deleted in the first revision. The first revision was taken up to incorporate a new requirement of purity of menthol content by GC and the requirement for melting range was revised based on data generated through indigenous testing. The requirement for congealing point was deleted.

In India, phenomenal development for the production of 1-menthol from oil of *Mentha arvensis* and its improved varieties like *KOSI*, CIM-UNNATI, etc. had 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 percent to 78 percent 1-menthol. 1-Menthol is usually obtained by fractional cooling of the mentha oils from $+15\,^{\circ}\text{C}$ to $-40\,^{\circ}\text{C}$ and subsequently separating the cake of (–)-menthol by centrifuging. Large and bold crystals of 1-menthol are then obtained by controlled slow cooling crystallization.

This 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 incorporated:

- a) Title of the standard has been changed from menthol to natural menthol;
- b) Scope has been modified by deleting the reference of various applications;
- c) Terminology of synthetic and natural menthol has been incorporated under terminology clause;

(Continued on third cover)

Indian Standard

NATURAL MENTHOL — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and tests for natural menthol.

2 REFERENCES

The standards listed in <u>Annex A</u> 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 edition of these standards.

3 TERMINOLOGY

For the purpose of this standard, following definitions shall apply in addition to those given in IS 6597.

- **3.1 Natural Menthol** Menthol extracted from naturally occurring mentha varieties, without any chemical synthesis.
- **3.2 Synthetic Menthol** Menthol synthesized through chemical routes where fossil fuel/petrochemical are the probable starting material including its blend with natural menthol.

4 REQUIREMENTS

4.1 Description

- **4.1.1** The material shall be colourless liquid or white acicular crystals/prismatic crystals/molten or solid mass/flakes/pellets/powder with a penetrating peppermint like odour.
- **4.1.2** The material shall be tested olfactorily 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 the seller.

4.2 Identification Tests

- **4.2.1** A 5 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 requirements prescribed in <u>Table 1</u>, when tested according to the methods given in col (4) of <u>Table 1</u>.

4.6 Quality of Reagents

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

NOTE — 'Pure chemicals' shall mean chemicals with purity greater than 98 percent and do not contain impurities which affect the result of the analysis.

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 the 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 legibly 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 number or batch number;
- e) Date of manufacturing;
- f) Shelf-life or best before in DD/MM/YY format; and
- g) Any other statutory requirements.

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

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

Table 1 Requirements for Natural Menthol

(Foreword and Clause 4.5)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Melting range, °C	42 to 44	IS 326 (Part 16)
ii)	Specific rotation at 20 °C (± 1 °C), (concentration 0.020 g/ml; MeOH)	(–) 47 $^{\circ}$ to (–) 50 $^{\circ}$	IS 326 (Part 4)
iii)	Non-volatile matter, percent by mass, Max	0.05	Annex B
iv)	Natural menthol content, percent by area, <i>Min</i>	99.0	Annex C
v)	Biobased carbon content, percent ¹⁾	100	Method B of ASTM D6866/Method C of IS 17948 (Part 2) ²⁾

Table 2 Reference pMC (Percent Modern Carbon)

(Foreword and Table 1, Footnote 1)

Sl No.	Year	REF (pMC, percent)
(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

 ${
m NOTE}$ — Calculation of percent biobased carbon content is made by dividing pMC by REF and multiplying the result by 100.

¹⁾ Refer to Table 2 'reference pMC' instead of Table 2 'percent modern carbon (pMC) reference' of ASTM D6866/Table 2 '100 percent biobased carbon values versus year' of IS 17948 (Part 2).

²⁾ In case of disputes, Method C of IS 17948 (Part 2) shall be the referee method for determination of biobased carbon content.

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ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

IS No./ Other Standards	Title	IS No./ Other Standards	Title
IS 326	Methods of sampling and test for natural and synthetic perfumery materials:	IS 2284 : 1988	Method of olfactory assessment of natural and synthetic perfumery materials (first revision)
(Part 1): 2022	Sampling (fourth revision)		
(Part 4) : 2005/ ISO 592 : 1998	Determination of optical rotation (third revision)	IS 6597 : 2001	Glossary of terms relating to fragrance and flavour industry (second revision)
(Part 6): 2005/ ISO 875: 1999	Evaluation of miscibility in ethanol (<i>third revision</i>)	IS 17948 (Part 2) : 2023/ISO 16620-2 : 2019	Plastics — Biobased content: Part 2 Determination of bio-based
(Part 16): 1989	Determination of melting		carbon content
	point and melting range (second revision)	ASTM D6866-24a	Standard test methods for determining the biobased
IS 1070 : 2023	Reagent grade water — Specification (fourth revision)		content of solid, liquid, and gaseous samples using radiocarbon analysis

To access Indian Standards click on the link below:

https://www.services.bis.gov.in/php/BIS 2.0/bisconnect/knowyourstandards/Indian standards/isdetails/

ANNEX B

[Table 1, Sl No. (iii)]

DETERMINATION OF NON-VOLATILE MATTER

B-1 APPARATUS

B-1.1 Analytical Balance — least count 0.001 g

B-1.2 Hot Air Oven — 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-3 CALCULATION

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

where

a =mass, in g, of the residue; and

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

ANNEX C

[Table 1, Sl No. (iv)]

DETERMINATION OF NATURAL MENTHOL CONTENT USING GAS CHROMATOGRAPHY

C-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. The typical chromatogram is shown in Fig. 1.

NOTE — Hexane, cyclohexane or petroleum ether may be used as suitable solvent.

C-2 APPARATUS

C-2.1 Gas Chromatograph — an electronic controlled gas chromatograph (GC) with a flame ionization detector, split/splitless or PSS injector, cyclodextrin capillary column with following accessories and operating conditions shall be used.

C-2.1.1 Gas Chromatographic Conditions

Sample size : 0.5 µl (prepared in

dichloromethane)

Capillary column : Gamma-cyclodextrin

fused silica capillary column with length 30 m;

diameter 0.25 mm and film thickness 0.25 um

Chiral stationary : 2,3-di-acetoxy-6-O-tert-

phase butyl dimethyl silyl

gamma cyclodextrin

Material : 14 percent

cyanopropylphenyl/ 86 percent dimethyl

polysiloxane

Carrier gas : Hydrogen (1.8 ml/min)

Flow split ratio : 1:140

Injector type : Split/splitless/PSS

Injection : 220 °C

temperature

Flow control : Electronic/automatic

Detector:

Type : Flame ionization

detector

Temperature : 230 °C

Flow control : Electronic/automatic

Temperature programme:

Temperature 1 : 70 °C (3 min hold)

Ramp 1 : $3 \, ^{\circ}\text{C/min}$

Temperature 2 : $120\,^{\circ}\text{C}$

Ramp 2 : $5 \, ^{\circ}\text{C/min}$

Temperature 3 : $230 \, ^{\circ}\text{C}$

C-3 CALCULATION

Area percent of each baseline separated peaks is automatically calculated and presented in result section of acquired gas chromatogram.

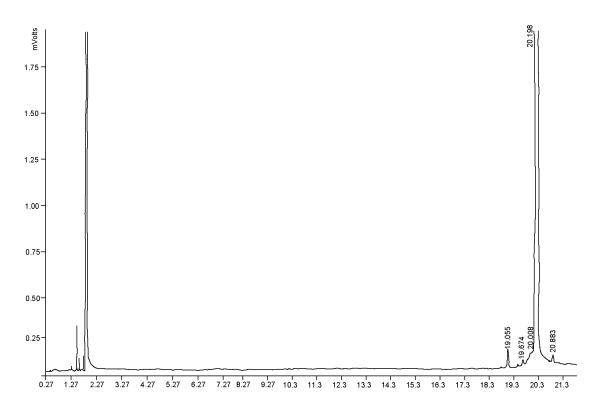


FIG. 1 TYPICAL CHROMATOGRAM OF NATURAL MENTHOL

ANNEX D

(<u>Foreword</u>)

COMMITTEE COMPOSITION

Fragrance and Flavour Sectional Committee, PCD 18

Organization	Representative(s)
CSIR - Central Institute of Medical and Aromatic Plants, Lucknow	DR PRABODH K. TRIVEDI (<i>Chairperson</i>)
All India Agarbathi Manufacturers Association, Bengaluru	SHRI SAPTHAGIRI S. BOGGARAM SHRI THAMBI VENKATA KRISHNA (<i>Alternate</i>)
Central Drugs Standard Control Organization, New Delhi	DR RIKTA SAHA SHRI BIBEKANANDA BEHERA (<i>Alternate</i>)
Central Drugs Testing Laboratory, Chennai	SHRIMATI C. VIJAYALAKSHMI
Central Revenue Control Laboratory, New Delhi	SHRI V. SURESH SHRI SHIVRAJ SINGH (<i>Alternate</i> I) DR MRITUNJOY MAITY (<i>Alternate</i> II)
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CSIR - Central Food Technological Research Institute, Mysuru	DR GIRIDHAR P. SHRI NAGARAJAN S. (<i>Alternate</i>)
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CSIR - Institute of Himalayan Bio-Resource Technology, Palampur	Dr Vijai Kant Agnihotri
CSIR - North East Institute of Science and Technology, Jorhat	Dr Mohan Lal
D.V. Deo Industries, Kochi	Shri Aditya Deo
Essential Oil Association of India, Delhi	SHRI ASHISH JHUNJHUNWALLA SHRI ASHOK MAHAJAN (<i>Alternate</i>)
Forest Research Institute (FRI), Dehradun	DR V. K. VARSHNEY
Fragrance and Flavour Development Centre, Kannauj	DR S. V. SHUKLA SHRI NADEEM AKBAR (<i>Alternate</i>)
Fragrances and Flavours Association of India, Mumbai	SHRI JAIDEEP MOHANLAL GANDHI SHRI KAMLESH SHAH (<i>Alternate</i>)

Organization

Representative(s)

Givaudan India Private Limited, Mumbai

MS SASWATI LAHIRI

DR JAI PRAKASH

MS ARSHDEEP K JOSHI (Alternate I) MS SAMPADA JANDE (Alternate II)

Indian Beauty and Hygiene Association, Mumbai

MS MALATHI NARAYANAN

Indian Pharmacopoeia Commission, Ghaziabad

DR MANAS V. VYAS (Alternate)

DR MANOJ KUMAR PANDEY (Alternate)

Indian Society of Cosmetic Chemists, Mumbai

MS MONISHA MULLICK SHRI BENEDICT MASCARENHAS (Alternate)

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MSME Testing Center, New Delhi

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Rakesh Sandal Industries, Kanpur

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Member Secretary KUMARI ADITI CHOUDHARY SCIENTIST 'C'/DEPUTY DIRECTOR (PETROLEUM, COAL AND RELATED PRODUCTS), BIS This Pade has been Intentionally left blank

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- d) Description of the material has been modified by incorporating various forms of natural menthol;
- e) Requirements of colour and appearance have been deleted from <u>Table 1</u> as already covered under description;
- f) Requirement of the specific rotation has been modified;
- g) New requirement of biobased carbon content and method of test for determination of biobased carbon content have been added to identify source of the material; and
- h) Reference values of percent modern carbon have been updated in Table 2.

Clauses 4.1.2 and 5.1.1 includes purchaser and seller agreement.

The composition of the Committee responsible for the revision of this standard is given in 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.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected	

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