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Draft Indian Standard

OIL OF MENTHA ARVENSIS — SPECIFICATION

(*Fourth Revision* of IS 528)

(ICS No. 71.100.60)

Fragrance and Flavour Sectional Committee
PCD 18

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FOREWORD

(Formal clauses will be added later)

This draft standard was first published in 1954 and subsequently revised in 1970, 1989 and 1999. The third revision was done with a view to bring it in line with trade practices prevalent in perfumery technology and also to align with the quality level of the material being produced, sold in the country and exported. Considerable assistance was derived from the draft of ISO 9776 : 1999 on partially dementholized oil and its values on Indian Oil.

This Standard covers two types of Oil of *Mentha arvensis* L. namely Type 1 Whole oil, and Type 2 Partially Dementholized oil. Type 1 Whole Oil of *Mentha arvensis* L, is primarily used all over the world as a natural raw material for the production of menthol. Type 2, Partially Dementholised Oil, is a by-product obtained during the process of producing Menthol crystals, which after rectification is used in pharmacy and also as a flavouring material in the preparation of dental pastes, powders and mouth washes.

In this revision, the gas chromatographic method has been upgraded from Packed Column GC to Capillary Column GC for more accurate results, which are being progressively used in the country. The requirements of free alcohols, esters, total alcohols and ketones for dementholized oil have been modified in this revision as per the quality of the oil produced and currently available in the market.

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 the Oil of *Mentha arvensis* L, family Labiatae, Whole and Partially Dementholized.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the 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 indicated below:

| <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) : 1980 | Preliminary examination of perfumery materials and samples (<i>second revision</i>) |
| (Part 3) : 2006 | Relative density (<i>third revision</i>) |
| (Part 4) : 2005 | Determination of optical rotation (<i>third revision</i>) |
| (Part 5) : 2006 | Determination of refractive index (<i>third revision</i>) |
| (Part 6) : 2005 | Determination of solubility (<i>third revision</i>) |
| (Part 8) : 2005 | Determination of ester value, content of esters and combined alcohols (<i>third revision</i>) |
| (Part 9) : 2017 | Determination of ester value after acetylation and free alcohols (<i>third revision</i>) |
| (Part 11) : 2017 | Determination of carbonyl value and content of carbonyl compounds (<i>third revision</i>) |
| (Part 19) : 2018 | Gas chromatographic analysis of perfumery materials (<i>first revision</i>) |
| 1070 : 1992 | Reagent grade water (<i>third revision</i>) |
| 2284 : 1988 | Methods for olfactory assessment of natural and synthetic perfumery materials (<i>first revision</i>) |
| 6597 : 2001 | Glossary of terms relating to natural and synthetic perfumery materials (<i>second revision</i>) |

3 TERMINOLOGY

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

4 TYPES

There shall be two types of the oil of *Mentha arvensis* L. as given below:

- 4.1 Type 1 - Whole oil, and
4.2 Type 2 - Partially Dementholized oil.

5 REQUIREMENTS

5.1 Description

5.1.1 The whole oil (Type 1) shall be obtained by steam distillation of the fresh over ground portion of the flowering plant of *Mentha arvensis* L., fam. Labiatae.

5.1.2 The partially dementholized oil (Type 2) shall be the product obtained by subjecting the whole oil (Type 1) to cooling, centrifuging, fractional distillation, rectification and other ancillary processes for the removal of substantial quantities of menthol.

5.1.3 The oil shall be a clear liquid, free from sediment, suspended matter, separated water and adulterants, when examined as prescribed in IS 326 (Part. 2).

5.1.4 The oil shall be tested olfactorily, especially for notes and for the presence of adulterants and impurities, if any, as prescribed in IS 2284.

5.2 The oil shall also comply with the requirements given in Table 1 when tested according to the methods given in col 4 of Table 1.

Table 1 Requirements for Oil of *Mentha arvensis*
(Clauses 5.2)

| SI No. | Characteristic | Requirement | | Method of Test, Ref to |
|--------|--|---|--|------------------------|
| | | Type 1 | Type 2 | |
| (1) | (2) | (3) | (4) | (5) |
| i) | Colour and appearance | Colourless pale yellow or greenish yellow | Colourless pale yellow | Visual Observation |
| ii) | Odour | Characteristic strong minty, herbal followed by cooling sensation | Characteristic minty, intense herbal followed by cooling sensation | IS 2284 |
| iii) | Relative density at 27°/27°C ²⁾ | 0.877 to 0.912 | 0.892 to 0.912 | IS 326 (Part 3) |
| | Relative density at 20°C ²⁾ | 0.8983 to 0.9013 | 0.9010 to 0.9270 | |
| iv) | Optical rotation | -31° to -40° | -20° to -40° | IS 326 (Part 4) |
| v) | Refractive index at | 1.456 to 1.4642 | 1.4512 to 1.4632 | IS 326 (Part 5) |

| | | | | |
|-----|---|------------------|------------------|--------------------------------|
| | $27^{\circ}\text{C}^{2)}$ | | | |
| | Refractive Index at $20^{\circ}\text{C}^{2)}$ | 1.4590 to 1.4606 | 1.4590 to 1.4630 | |
| vi) | Flash point, $^{\circ}\text{C}$, <i>Min</i> ³⁾ | 79 | 81 | 326 (Part 26) |
| vi) | Gas Chromatographic Analysis | | | Annex A |
| | Terpene percent by mass | 4.4 to 7.0 | 2 to 4.5 | IS 326 (Part 8) |
| | Ketones, as menthone (molecular mass 154.25), percent by mass (using free hydroxylamine method) | 4.6 to 11.5 | 12 to 25 | IS 326 (Part 11/ Sec 1) : 2017 |
| | Isomenthone (molecular mass 154.25), percent by mass (using free hydroxylamine method) | 1.5 to 4.5 | 6 to 8 | IS 326 (Part 11/ Sec 1) : 2017 |
| | Esters, as menthyl acetate (molecular mass 198.28), percent by mass | 1.0 to 6.3 | 5 to 7 | IS 326 (Part 24) |
| | Neomenthol (molecular mass 156.26), percent by mass | 1.8 to 2.5 | 3.0 to 4.5 | IS 326 (Part 9/ Sec 2) : 2017 |
| | Free alcohols, as 1-menthol (molecular mass 156.26), percent by mass | 67 to 80 | 40 to 55 | IS 326 (Part 9/ Sec 2) : 2017 |
| | Total alcohols, as menthol (molecular | 71 to 80 | 48 to 58 | IS 326 (Part 9/ Sec 2) : 2017 |

| | | | |
|--|------------|------------|-----------------------------------|
| mass 156.26), percent by mass ¹⁾ | | | |
| Pulegone (molecular mass 152.23), percent by mass | 0.2 to 0.5 | 0.3 to 0.7 | IS 326 (Part 11/ Sec 1) : 2017 |

NOTES —

- 1) 3-Octanol proportions (>1.0) shall be high in Type 2 oil as compared to Type 1
- 2) The correction factors for relative density and refractive index for each degree Celsius change in temperature are 0.00064 and 0.00038 respectively.
- 3) Flash point was measured in a continuous closed cup (CCC) automatic Flash Point tester.

5.3 Chromatographic Profile

Carry out the Gas Chromatography analysis of the essential oil as per Annex A. The proportions of the components in the chromatogram are given in Table 2. The Marker chemicals identified in oil of *Mentha arvensis* L. are listed as per their order of elution in a polar phase capillary column coated with polyethylene glycol as stationary phase.

Table 2 Proportions of marker components in Type 1 Oil
(Clause 5.3)

| (1) | (2) | FID percent | |
|-----|------------------|-------------|------|
| | | (3) | (4) |
| 1 | Limonene | 1.2 | 3.2 |
| 2 | Menthone | 4.6 | 11.5 |
| 3 | Isomenthone | 1.5 | 4.5 |
| 4 | Menthyl acetate* | 1.0 | 6.3 |
| 5 | Neomenthol | 1.8 | 2.5 |
| 6 | Pulegone | 0.2 | 0.5 |

| | | | |
|----------|--------------------|------|------|
| 7 | Menthol | 67.0 | 80.0 |
| | Terpene percentage | 4.4 | 7.0 |

NOTE — Menthyl acetate co-elute with linalool, but the major proportion to this peak is contributed by menthyl acetate

5.4 Menthofuran Test

The oil shall not develop blue colour when tested as follows:

Mix 3 drops of the oil with 5 ml of a solution of 1 volume of nitric acid in 300 volumes of glacial acetic acid in a dry test tube. Place the tube in beaker of boiling water for 5 minutes.

NOTE — If oil *ex Mentha piperita* is present, blue colour develops within 1 to 5 minutes of placing the tube in boiling water, which, on continued heating deepens and shows copper-coloured fluorescence and then fades leaving a golden yellow solution.

5.5 Solubility

Type **1** material shall be soluble in **2.5** to **3** volumes and Type **2** in **3** volumes of ethyl alcohol (70 percent by volume) respectively when tested as prescribed in IS 326 (Part 6).

6 PACKING AND MARKING

6.1 Packing

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

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

6.2 Marking

The material shall be marked with the following information:

- a) Name of the material;
- b) Each container shall be clearly marked with the origin of the material and the menthol content (total alcohols);
- c) Name of the manufacturer and the recognized trade-mark, if any;
- d) Net Mass of material;
- e) Net Volume of material
- e) Batch number;
- f) Manufacturing date.

6.2.1 The containers may also be marked with the Standard Mark.

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

7 SAMPLING

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

7.1.1 The oil, particularly Type 1, shall be warmed to 40°C and agitated to homogenize prior to drawing a representative sample.

NOTE — At low temperatures, menthol is liable to crystallize producing stratification.

7.2 Number of Tests

7.2.1 Test for determination of menthol content (total alcohols) shall be conducted on each of the individual samples.

7.2.2 Tests for all the remaining characteristics shall be conducted on the composite sample.

7.3 Criteria for Conformity

7.3.1 For Individual Samples

For menthol content (total alcohols), determined on the individual samples, the mean (\bar{x}) and range (R) of the test results shall be calculated. If the value of the expression $\bar{x} - 0.4 R$ is greater than or equal to the value specified in Table 1, the lot shall be declared as conforming to the requirement of menthol content.

7.3.2 For Composite Sample

For declaring conformity of the lot to all other requirements of this specification, the results of tests on the composite sample shall meet the corresponding requirements.

8 TEST METHODS

8.1 Tests shall be conducted as prescribed under **5.1.3**, **5.1.4**, **5.3** and **5.4** and the appropriate references to relevant parts and Annexes of the standards as given in col 4 of Table 1.

8.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) 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. vi]
GAS CHROMATOGRAPHIC ANALYSIS OF OIL OF *MENTHA ARVENSIS* L.

A-1 GENERAL

A-1.1 The chromatographic analysis is reported on a polar phase capillary column. The conditions given here are for guidance only.

A-2 PROCEDURE

A-2.1 The analysis shall be done as per IS 326 (Part 19). The typical chromatograms for Type 1 whole oil of *Mentha arvensis* (Fig. 1) and Type 2 Partially Dementholized oil (Fig. 2) obtained as per the experimental conditions given below:

A-2.2 Gas Chromatographic conditions for Polar column

| | |
|-------------------|---|
| Sample size | 0.1 µl (diluted in hexane/ dichloromethane) |
| Capillary column: | Polar phase Fused Silica capillary column |
| Material | Polyethylene Glycol |
| Length | 30 m |
| Internal diameter | 0.32 mm |
| Film thickness | 0.25 µm |
| Carrier gas | Hydrogen/ Nitrogen (1.7 ml/min) |
| Flow split ratio | 1: 40 |
| Injector Type | split/splitless |

| | |
|--------------------------------------|---------------------------|
| Injection temperature | 240°C |
| Flow Control | Electronic/ Automatic |
| Detector: | |
| Type | Flame Ionization Detector |
| Temperature | 250°C |
| Flow Control | Electronic/ Automatic |
| <i>Oven Temperature Programming:</i> | |
| | |
| a) Initial Temperature | 40°C |
| b) Ramp | 3°C/min |
| c) Temperature (Step 1) | 120°C hold for 9 min |
| d) Ramp | 2 °C/min |
| e) Temperature (Step 2) | 140 °C |
| f) Ramp | 5 °C/min |
| g) Final Temperature | 220 °C hold for 9 min |

A-3 CALCULATION

A-3.1 Area percent (%) of each baseline separated peaks is automatically calculated and presented in result section of acquired gas chromatogram of Type 1 and Type 2, respectively.

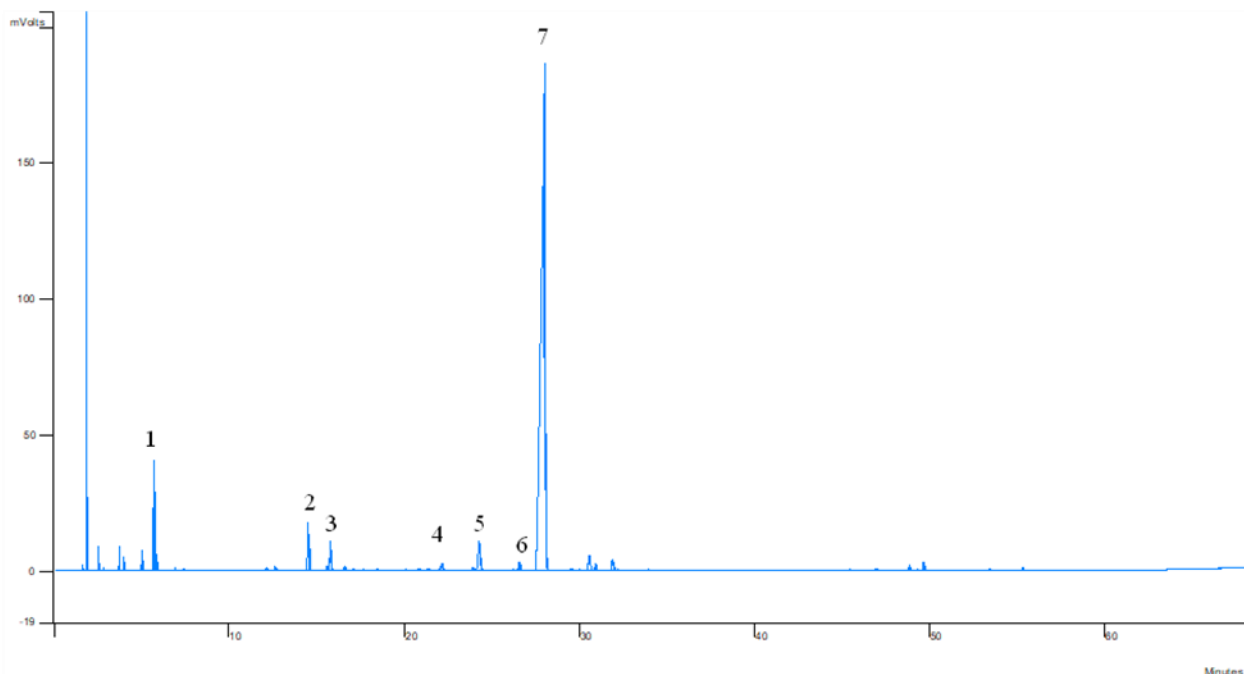


Fig. 1. Gas chromatogram of Type 1 Whole Oil of *Mentha arvensis* L.

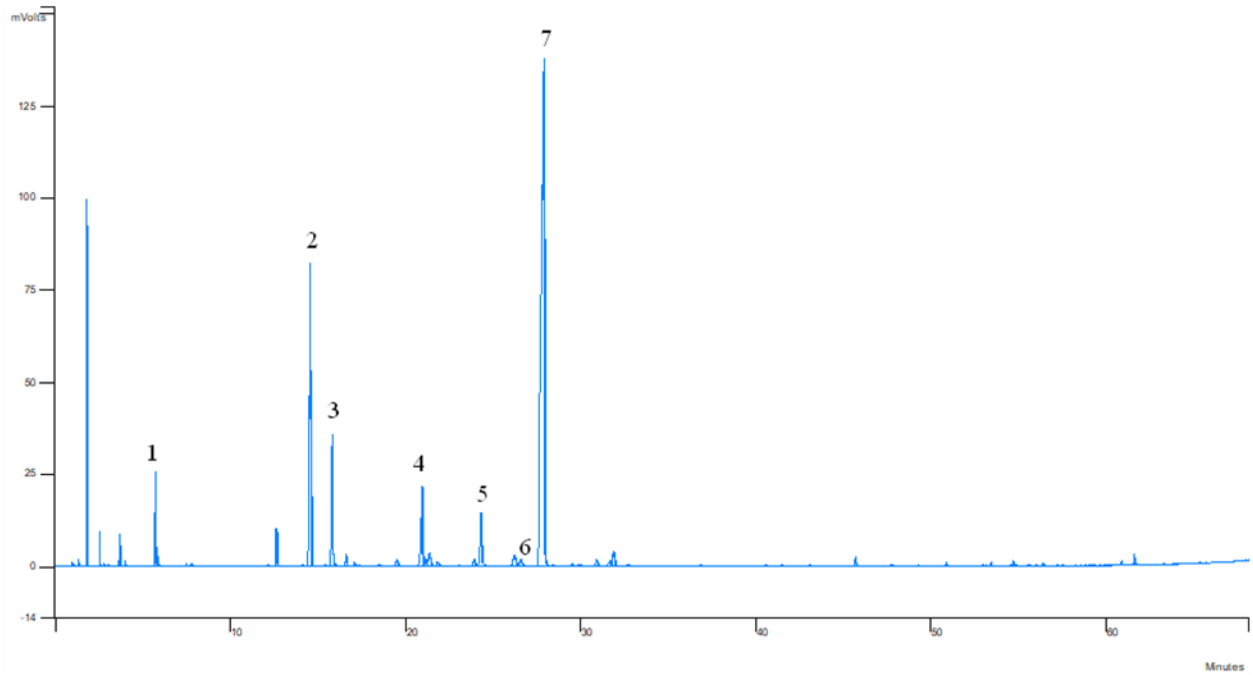


Fig. 2. Gas chromatogram of Type 2 Partially Dementholized Oil