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IS 528 (1999): Oil of Mentha Arvensis [PCD 18: Natural and Synthetic Fragrance Materials]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
मेन्था आरवेन्सिस का तेल — विशिष्टि
(तीसरा पुनरीक्षण)

Indian Standard
OIL OF *MENTHA ARVENSIS* —
SPECIFICATION
(*Third Revision*)

ICS 71.100.60

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1954 and subsequently revised in 1970 and 1989. The Sectional Committee responsible for its preparation felt that it should be revised again 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 currently being produced, sold in the country and exported. Considerable assistance has been derived from the draft of ISO/DIS 9776 on partially dementholized oil and its values on Indian Oil.

In this revision, the gas chromatographic method both on packed and capillary column which are being progressively used in the country has been included giving the percent range of the constituents. The requirements of free alcohols, esters, total alcohols and ketones for dementholized oil have been modified as per the quality of the oil produced and currently available in the market.

The whole oil of *Mentha arvensis L.* is primarily used all over the world as a natural raw material for the production of menthol. Its partially dementholized product, after rectification is used in pharmacy and also as a flavouring material in the preparation of dental pastes, powders and mouth washes.

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 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

OIL OF *MENTHA ARVENSIS* — SPECIFICATION (*Third Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for the oil of *Mentha arvensis* L., fam. *Labiatae*, whole and dementholized.

2 NORMATIVE 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:

IS No.	Title
IS 326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1):1984	Sampling (<i>second revision</i>)
(Part 2):1980	Preliminary examination of perfumery materials and samples (<i>second revision</i>)
(Part 3):1980	Relative density (<i>second revision</i>)
(Part 4):1980	Determination of optical rotation (<i>second revision</i>)
(Part 5):1980	Determination of refractive index (<i>second revision</i>)
(Part 6):1986	Determination of solubility (<i>second revision</i>)
(Part 8):1980	Determination of ester value, content of esters and combined alcohols (<i>second revision</i>)
(Part 9):1980	Determination of ester value after acetylation and free alcohols (<i>second revision</i>)
(Part 11):1980	Determination of carbonyl value and content of carbonyl compounds (<i>second revision</i>)
(Part 19):1998	Gas chromatographic analysis of perfumery materials
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>)
6597:1988	Glossary of terms relating to natural and synthetic perfumery materials (<i>first 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:

- Type 1 Whole oil, and
- 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 overground 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 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 a copper-coloured fluorescence and then fades, leaving a golden-yellow solution.

5.3 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).

IS 528 : 1999

5.4 The material shall also comply with the requirements given in Table 1 when tested according to the methods referred to in col 5 of Table 1.

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) Batch number; and
- 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, 1986* and the Rules and Regulations made thereunder. Details of conditions under which a licence 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.

Table 1 Requirements for Oil of *Mentha Arvensis*
(Clauses 5.4, 7.3.1 and 8.1)

Sl 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	IS 326 (Part 2)
ii)	Odour	Characteri- stic strong minty, herbal followed by cooling sensation	Characteri- stic, minty intense herbal followed by cooling sensation	IS 2284
iii)	Relative density at 27°/27°C ¹⁾	0.877 3 to 0.912 3	0.892 3 to 0.912 3	IS 326 (Part 3)
iv)	Optical rotation	-35° to -45°	-20° to -40°	IS 326 (Part 4)
v)	Refractive index at 27°C ¹⁾	1.456 to 1.464 2	1.451 2 to 1.463 2	IS 326 (Part 5)
vi)	Free alcohols, as l-menthol (molecular mass 156.26), percent by mass, <i>Min</i>	60	40	Annex A
vii)	Esters, as menthyl acetate (molecular mass 198.28), percent by mass	3 to 15	3 to 9	IS 326 (Part 8)
viii)	Total alcohols, as menthol (molecular mass 156.26), percent by mass, <i>Min</i>	65	45	IS 326 (Part 9)
ix)	Ketones, as menthone (molecular mass 154.25), percent by mass (using free hydroxylamine method)	5 to 20	25 to 40	IS 326 (Part 11)

¹⁾The correction factors for relative density and refractive index for each degree Celsius change in temperature are 0.000 64 and 0.000 38 respectively.

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 given on both packed column and 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 oil of *mentha arvensis* for the following conditions are given in Fig. 1, Fig. 2 and Fig. 3, respectively.

A-2.1.1 Packed Column

<i>Sample</i>	<i>Oil of Mentha arvensis L (Partially dementholized)</i>
a) Column	Carbowax
b) Length	3 m
c) I.D.	1/8 inch (0.32 cm)

Column Temperature

a) Initial temperature	100°C
b) Isothermal	1 min
c) Ramp	4°C/m
d) Final temperature	200°C
e) Injection port temperature	250°C

Detector

a) Type	F.I.D.
b) Temperature	250°C.
Carrier gas	Nitrogen
Flow rate	30 ml/min

Capillary Column

<i>Column</i>	<i>Capillary</i>
a) Material	Silica
b) Length	60 m
c) I.D.	0.25 mm
d) Stationary phase film thickness	0.25 µm
Carrier gas	Nitrogen
Flow rate	1 ml/min

Column Temperature

a) Initial	70°C hold for 1 min
b) Ramp	3°C/min
c) Final	210°C
d) Injection port temperature	230°C
e) Injection split ratio	1:100

Volume of the sample injected	0.2 µl
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Detector

a) Type	F.I.D.
b) Temperature	250°C.

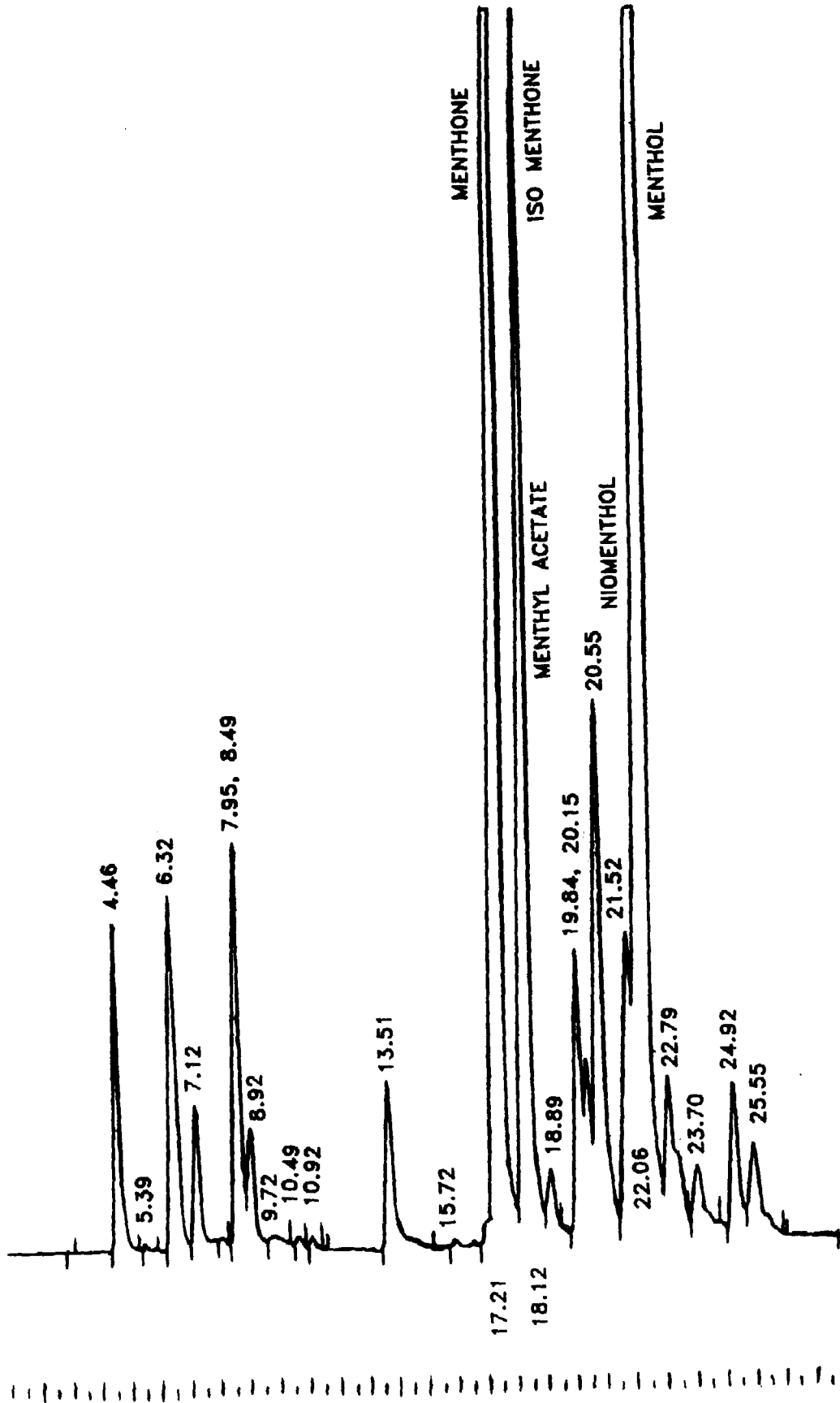


FIG. 1 TYPICAL GAS CHROMATOGRAM OF OIL OF *MENTHA ARVENSIS* (PARTIALLY DEMENTHOLIZED) ON PACKED COLUMN

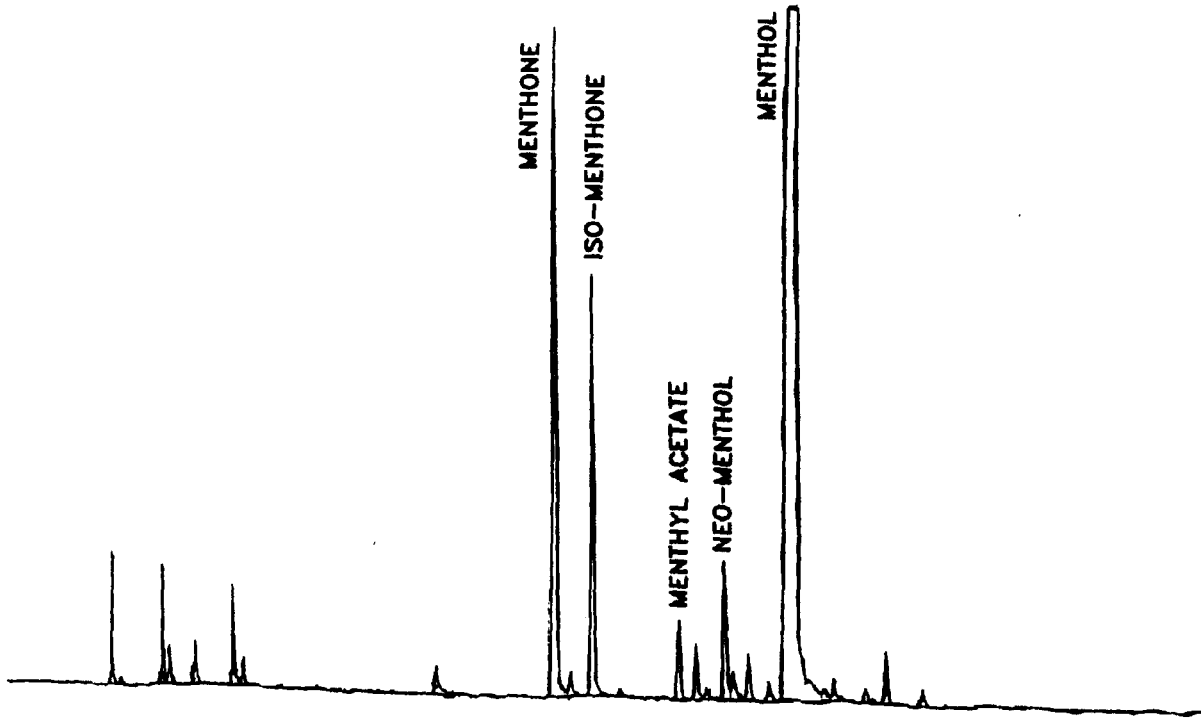


FIG. 2 CAPILLARY GC OF *MENTHA ARVENSIS* OIL

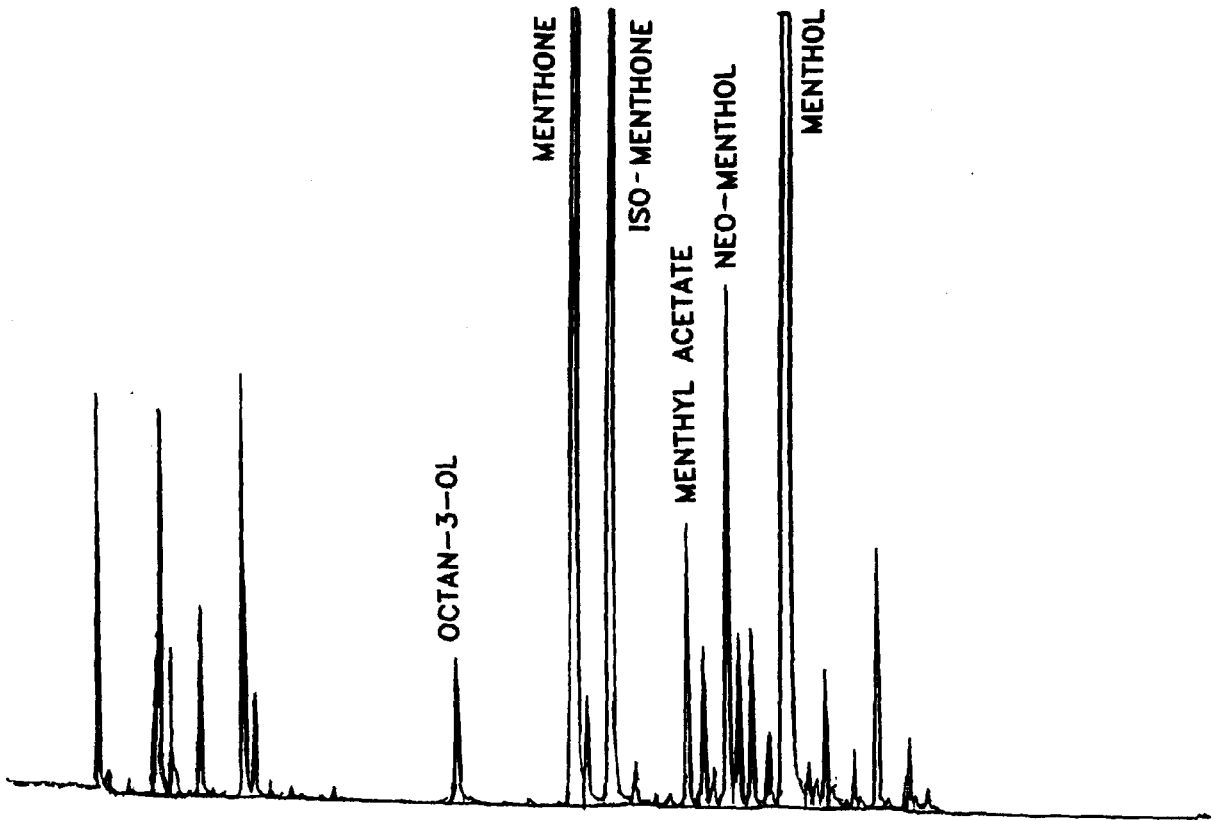


FIG. 3 CAPILLARY GC OF DEMENTHOLIZED OIL

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