INTERNATIONAL STANDARD

ISO 9776

First edition
1999-03-01

Oil of *Mentha arvensis*, partially dementholized (*Mentha arvensis* L. var. *piperascens* Malinv. and var. *glabrata* Holmes)

Huile essentielle de Mentha arvensis, *partiellement démentholée (*Mentha arvensis *L. var.* piperascens *Malinv. et var.* glabrata *Holmes)*

Reference number
ISO 9776:1999(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9776 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

Annexes A and B of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

Oil of *Mentha arvensis*, partially dementholized (*Mentha arvensis* L. var. *piperascens* Malinv. and var. *glabrata* Holmes)

1 Scope

This International Standard specifies certain characteristics of the oil of *Mentha arvensis*, partially dementholized (*Mentha arvensis* L. var. *piperascens* Malinv. and var. *glabrata* Holmes), in order to facilitate assessment of its quality.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO/TR 210, *Essential oils — General rules for packaging, conditioning and storage.*

ISO/TR 211, *Essential oils — General rules for labelling and marking of containers.*

ISO 212, *Essential oils — Sampling.*

ISO 279, *Essential oils — Determination of relative density at 20 °C (Reference method).*

ISO 280, *Essential oils — Determination of refractive index.*

ISO 592, *Essential oils — Determination of optical rotation.*

ISO 709, *Essential oils — Determination of ester value.*

ISO 875, *Essential oils — Evaluation of miscibility in ethanol.*

ISO 1241, *Essential oils — Determination of ester value before and after acetylation and evaluation of the contents of free and total alcohols.*

ISO 1242, *Essential oils — Determination of acid value.*

ISO 1271, *Essential oils — Determination of carbonyl value — Free hydroxylamine method.*

ISO 11024-1, *Essential oils — General guidance on chromatographic profiles — Part 1: Preparation of chromatographic profiles for presentation in standards.*

ISO 11024-2, *Essential oils — General guidance on chromatographic profiles — Part 2: Utilization of chromatographic profiles of samples of essential oils.*

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
oil of *Mentha arvensis*, partially dementholized
essential oil obtained by steam distillation of the flowering tops of *Mentha arvensis* L. var. *piperascens* Malinv. and var. *glabrata* Holmes, of the Lamiaceae family, growing mainly in China and India

NOTE The essential oil is then partially dementholized by freezing and centrifuging.

4 Requirements

4.1 Appearance

Clear, mobile liquid.

4.2 Colour

Almost colourless to amber yellow.

4.3 Odour

Characteristic of mint, menthol-like.

4.4 Relative density at 20 °C, d_{20}^{20}

	China	India	Other origins
Minimum	0,890	0,890	0,890
Maximum	0,908	0,910	0,910

4.5 Refractive index at 20 °C

Minimum: 1,457 0

Maximum: 1,465 0

4.6 Optical rotation at 20 °C

	China	India	Other origins
Minimum	–24°	–22°	–30°
Maximum	–15°	–13°	–10°

4.7 Miscibility in ethanol (volumic fraction 70 %) at 20 °C

1 volume of the oil shall require a maximum of 4 volumes of ethanol (volumic fraction 70 %) at 20 °C to obtain a clear solution. Opalescence may sometimes be observed on further addition of solvent.

4.8 Acid value

Maximum: 1

4.9 Ester value

	China	India	Other origins
Minimum	8	8	8
Maximum	25	25	31
Corresponding to an ester content, expressed as menthyl acetate, of	3 % to 9 %	3 % to 9 %	3 % to 11 %

4.10 Menthol content by determination of ester value after acetylation

	China	India	Other origins
Minimum expressed as free menthol	40 %	40 %	35 %
Maximum expressed as free menthol	60 %	60 %	65 %

4.11 Carbonyl value

	China	India	Other origins
Minimum	91	91	91
Maximum	164	146	164
Corresponding to a menthone content of	25 % to 45 %	25 % to 40 %	25 % to 45 %

4.12 Chromatographic profile

Analysis of the essential oil shall be carried out by gas chromatography. In the chromatogram obtained, the representative and characteristic components shown in Table 1 shall be identified. The proportions of these components, indicated by the integrator, shall be as shown in Table 1. This constitutes the chromatographic profile of the essential oil.

4.13 Flashpoint

Information on the flashpoint is given in annex B.

Table 1 — Chromatographic profile

Component	Proportion					
	China		India		Other origins	
	min. %	max. %	min. %	max. %	min. %	max. %
Octan-3-ol	0,5	3	0,2	1,8	0,2	2
1,8-Cineole	0,3	1,5	0,2	1	0,1	2
Limonene	1,5	4	1	4	1	7
Menthone	18	30	17	26	17	32
Isomenthone	8	12	8	14	6	13
Neomenthol	4	8	4	10	3	11
Menthol	33	45	33	45	30	46
Pulegone	0,5	2,5	0,5	2,5	0,5	2,5
Menthyl acetate	1,5	4	1,5	5	2	7
β-Caryophyllene	0,5	2	0,5	2	0,5	2

NOTE The chromatographic profile is normative, contrary to typical chromatograms given for information in annex A.

5 Sampling

See ISO 212.

Minimum volume of test sample: 50 ml.

NOTE This volume allows each of the tests specified in this International Standard to be carried out at least once.

6 Test methods

6.1 Relative density at 20 °C, d_{20}^{20}

See ISO 279.

6.2 Refractive index at 20 °C

See ISO 280.

6.3 Optical rotation at 20 °C

See ISO 592.

6.4 Miscibility in ethanol (volumic fraction 70 %) at 20 °C

See ISO 875.

6.5 Acid value

See ISO 1242.

Test portion: 2 g.

6.6 Ester value

See ISO 709.

Test portion: 2 g.

Saponification time: 1 h.

Relative molecular mass of menthyl acetate: 198,3.

6.7 Ester value after acetylation

See ISO 1241.

Test portion: 2 g.

Saponification time: 2 h 30.

Relative molecular mass of menthol: 156,3.

6.8 Carbonyl value

See ISO 1271.

Test portion: 2 g.

Standing time: 1 h.

Relative molecular mass of menthone: 154,2.

6.9 Chromatographic profile

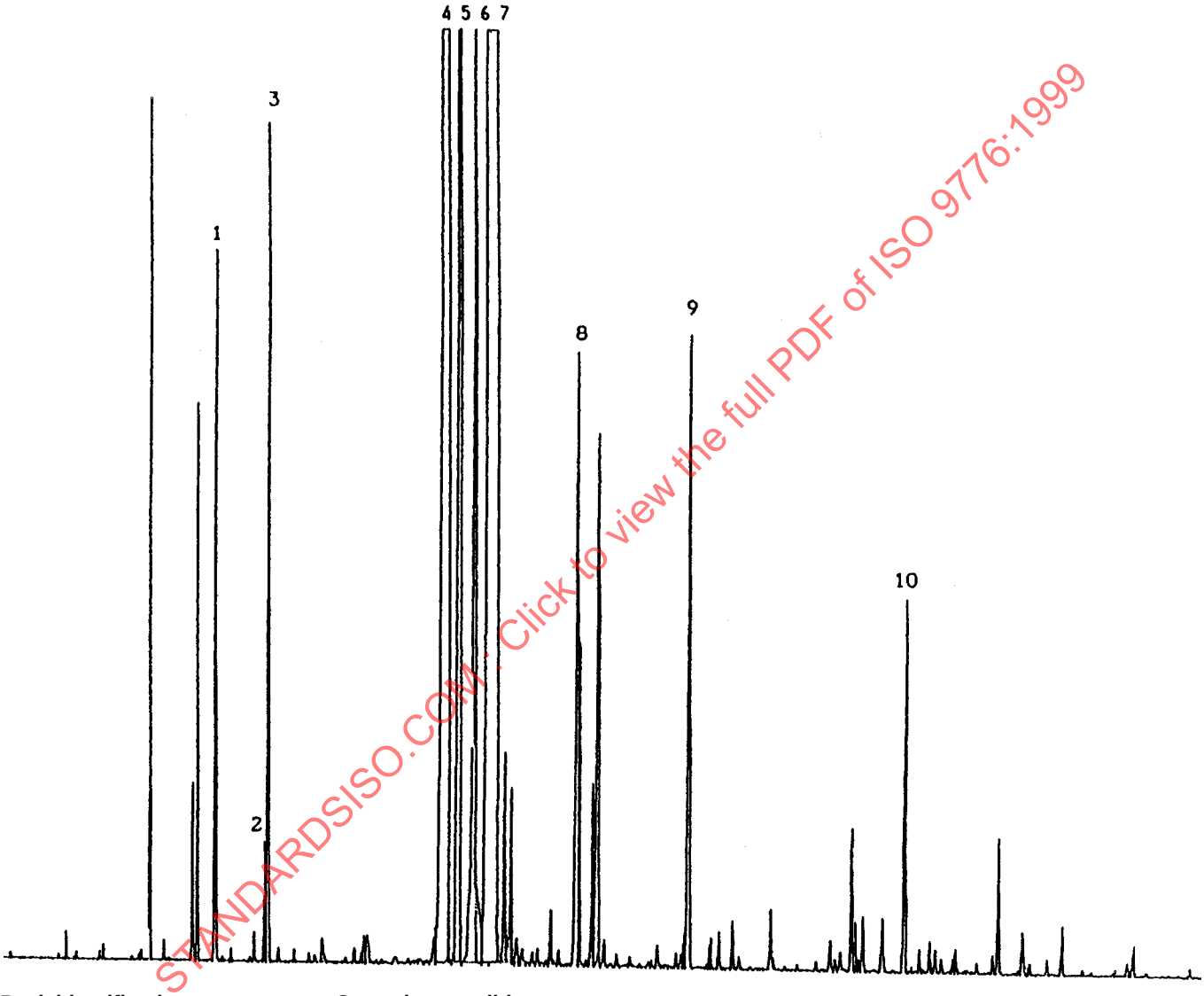
See ISO 11024-1 and ISO 11024-2.

7 Packaging, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

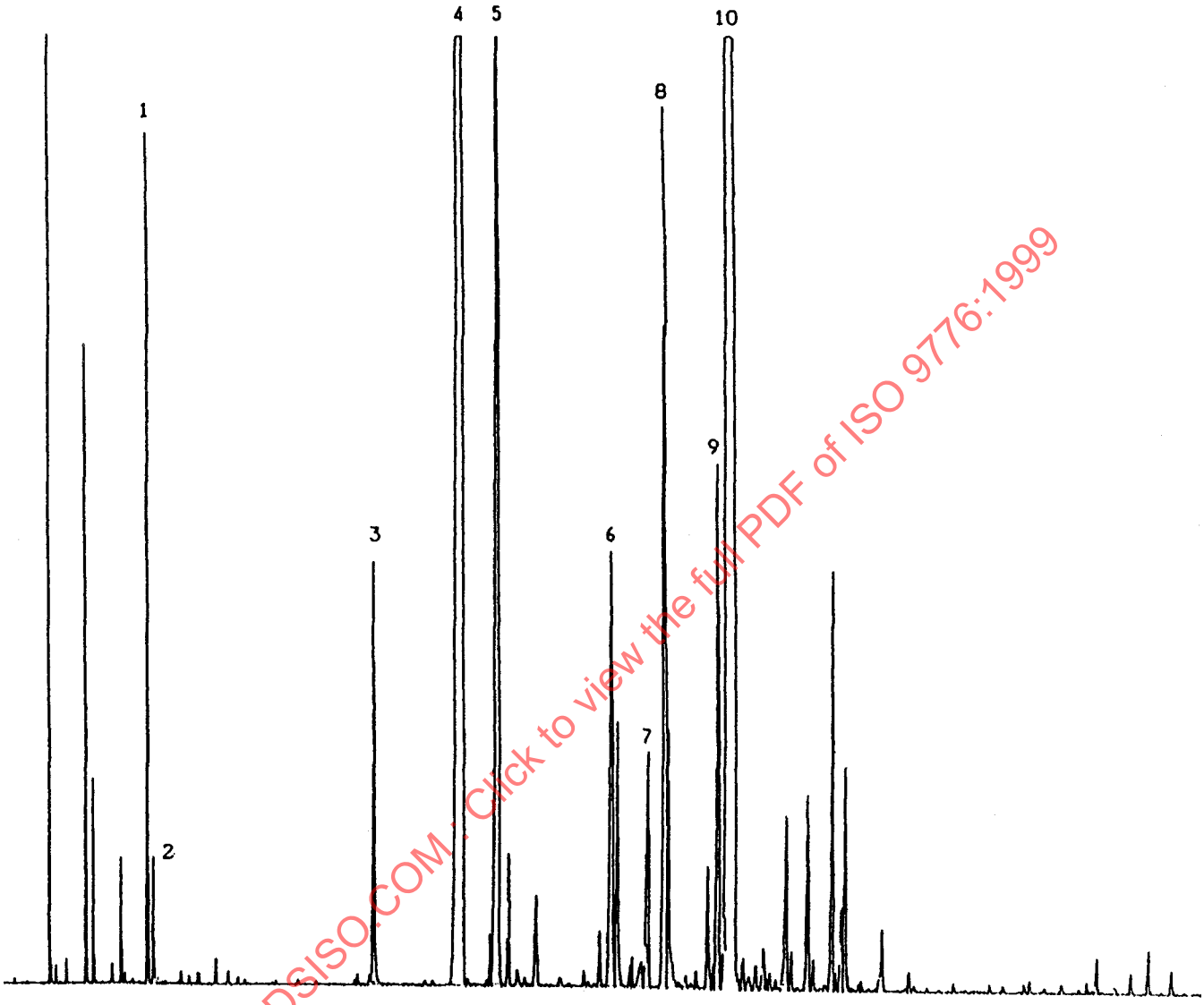
Annex A
(informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of *Mentha arvensis*, partially dementholized (China)



Peak identification	Operating conditions
1 Octan-3-ol + myrcene	Column: capillary, silica, 50 m long and 0,2 mm internal diameter
2 1,8-Cineole	Stationary phase: OV101®
3 Limonene	Film thickness 0,25 µm
4 Menthone	Oven temperature: temperature programming from 65 °C to 200 °C at a rate of 2 °C/min
5 Isomenthone	Injector temperature: 230 °C
6 Neomenthol	Detector temperature: 250 °C
7 Menthol	Detector: flame ionization type
8 Pulegone	Carrier gas: hydrogen
9 Menthyl acetate	Volume injected: 0,2 µl
10 β-Caryophyllene	Carrier gas linear velocity: 0,35 m/s approx.
	Split ratio: 1/100

Figure A.1 — Typical chromatogram taken on an apolar column



Peak identification

1 Limonene
2 1,8-Cineole
3 Octan-3-ol
4 Menthone
5 Isomenthone
6 Menthyl acetate
7 β-Caryophyllene
8 Neomenthol
9 Pulegone
10 Menthol

Operating conditions

Column: capillary, silica, 50 m long and 0,2 mm internal diameter
Stationary phase: polyethylene glycol (CARBOWAX) ®
Film thickness 0,25 µm
Oven temperature: temperature programming from 65 °C to 200 °C at a rate of 2 °C/min
Injector temperature: 230 °C
Detector temperature: 250 °C
Detector: flame ionization type
Carrier gas: hydrogen
Volume injected: 0,2 µl
Carrier gas linear velocity: 0,35 m/s approx.
Split ratio: 1/100

Figure A.2 — Typical chromatogram taken on a polar column

Annex B
(informative)

Flashpoint

B.1 General information

For reasons of safety, transport companies, insurance companies, people in charge of safety services, etc. require information on the flashpoint of essential oils, which in most cases are flammable products.

A comparative study on the relevant methods of analysis (see ISO/TR 11018[1)]) concluded that it was hard to find a single method for standardization purposes, given that:

— essential oils are varied and their chemical compositions differ to a large extent;

— the volume of the sample needed for certain test equipment is incompatible with the high price of essential oils;

— there are different types of equipment that satisfy the desired objective, but users cannot be obliged to use one type of equipment rather than another.

Consequently, it was decided to give a mean value for the flashpoint in an informative annex in each International Standard, for information purposes, in order to meet the requirements of the interested parties.

If possible, the method by which this value was obtained should be specified.

For further information see ISO/TR 11018[1)].

B.2 Flashpoint of the essential oil of *Mentha arvensis*, partially dementholized

The mean value is +75 °C.

NOTE Obtained with "Luchaire" equipment.

1) ISO/TR 11018:1997, *Essential oils — General guidance on the determination of flashpoint.*