INTERNATIONAL STANDARD

ISO 3033-3

First edition
2005-09-01

Oil of spearmint —

Part 3: Indian type (*Mentha spicata* L.), redistilled oil

Huile essentielle de menthe crépue (ou menthe verte) —
*Partie 3: Type Inde (*Mentha spicata *L.), huile bidistillée*

Reference number
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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 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3033-3 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This first edition of ISO 3033-3, together with ISO 3033-1:2005, ISO 3033-2:2005 and ISO 3033-4:2005, cancels and replaces ISO 3033:1988, which has been technically revised.

ISO 3033 consists of the following parts, under the general title *Oil of spearmint*:

— *Part 1: Native type (*Mentha spicata *L.)*

— *Part 2: Chinese type (80 % and 60 %) (*Mentha viridis *L. var.* crispa *Benth.), redistilled oil*

— *Part 3: Indian type (*Mentha spicata *L.), redistilled oil*

— *Part 4: Scotch variety (*Mentha × gracilis *Sole)*

Oil of spearmint —

Part 3: Indian type (*Mentha spicata* L.), redistilled oil

1 Scope

This part of ISO 3033 specifies certain characteristics of the oil of spearmint, Indian type (*Mentha spicata* L.), redistilled oil, in order to facilitate assessment of its quality.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
oil of spearmint, Indian type, redistilled
essential oil obtained by steam distillation from the fresh above-ground parts of the flowering plant of *Mentha spicata* L.

NOTE 1 The steam-distilled oil obtained is redistilled to yield a carvone content of 60 % minimum.

NOTE 2 For information on the CAS number, see ISO/TR 21092.

4 Requirements

4.1 Appearance

Clear mobile liquid.

4.2 Colour

From colourless to pale yellow.

4.3 Odour

Characteristic odour of carvone with an herbaceous note.

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

Minimum: 0,921 0

Maximum: 0,938 0

4.5 Refractive index at 20 °C

Minimum: 1,484 0

Maximum: 1,491 0

4.6 Optical rotation at 20 °C

Between −59° and −48°.

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

It shall not be necessary to use more than 3 volumes of ethanol, 70 % (volume fraction), to obtain a clear solution with 1 volume of essential oil.

4.8 Carbonyl value

Minimum: 224, corresponding to a carbonyl compound content of 60 %, expressed as carvone.

4.9 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.

Table 1 — Chromatographic profile

Component	Minimum %	Maximum %
Limonene [a]	11,5	16,5
3-Octanol	0,6	1,4
Menthone [b]	—	0,15
trans-Sabinene hydrate	0,5	1,0
cis-Dihydrocarvone	1,0	2,5
Carvone [c]	60,0	70,0
trans-Dihydrocarvyl acetate	0,1	0,6
cis-Carvyl acetate	0,1	0,6
cis-Jasmone	0,1	0,4
β-Bourbonene	1,0	2,0
Viridiflorol	not detectable	

[a] The limonene found is regarded to be predominantly L-limonene based on the physical testing. It is believed that there might be a small amount of D-limonene present but the exact quantity is unknown.

[b] The menthone found is regarded to be predominantly L-menthone based on the physical testing. It is believed that there might be a small amount of D-menthone present but the exact quantity is unknown.

[c] The carvone foundis regarded to be predominantly L-carvone based on the physical testing. It is believed that there might be a small amount of D-carvone present but the exact quantity is unknown.

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

4.10 Flashpoint

Information on the flashpoint is given in Annex B.

5 Sampling

See ISO 212.

Minimum volume of test sample: 50 ml.

NOTE This volume allows each of the tests specified in this part of ISO 3033 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, 70 % (volume fraction), at 20 °C

See ISO 875.

6.5 Carbonyl value

See ISO 1271.

Test portion: 1 g.

Reflux time: 3 h.

Relative molar mass of carvone: 150,21.

6.6 Chromatographic profile

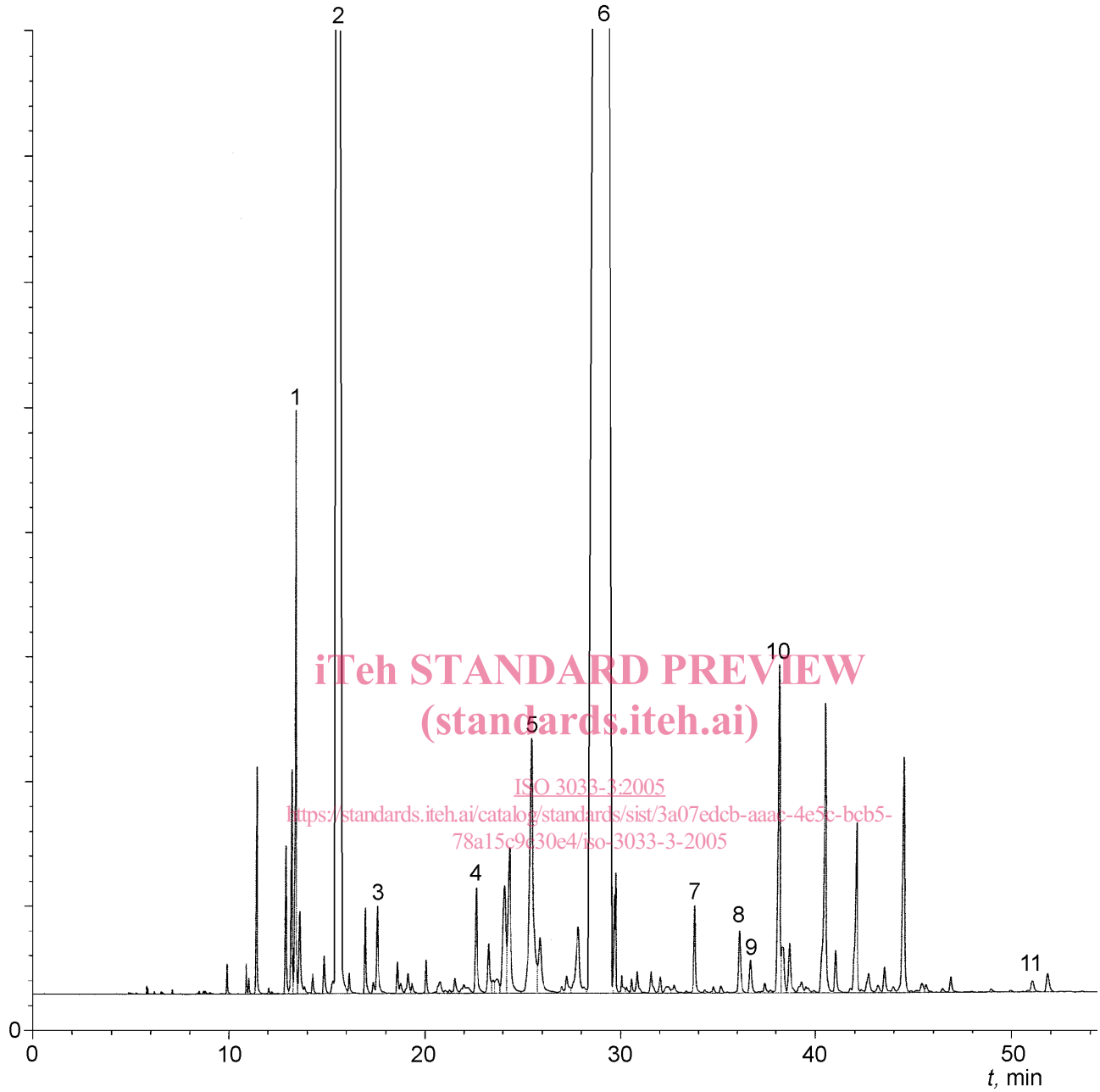
See ISO 11024-1 and ISO 11024-2.

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 spearmint, Indian type (*Mentha spicata* L.), redistilled oil



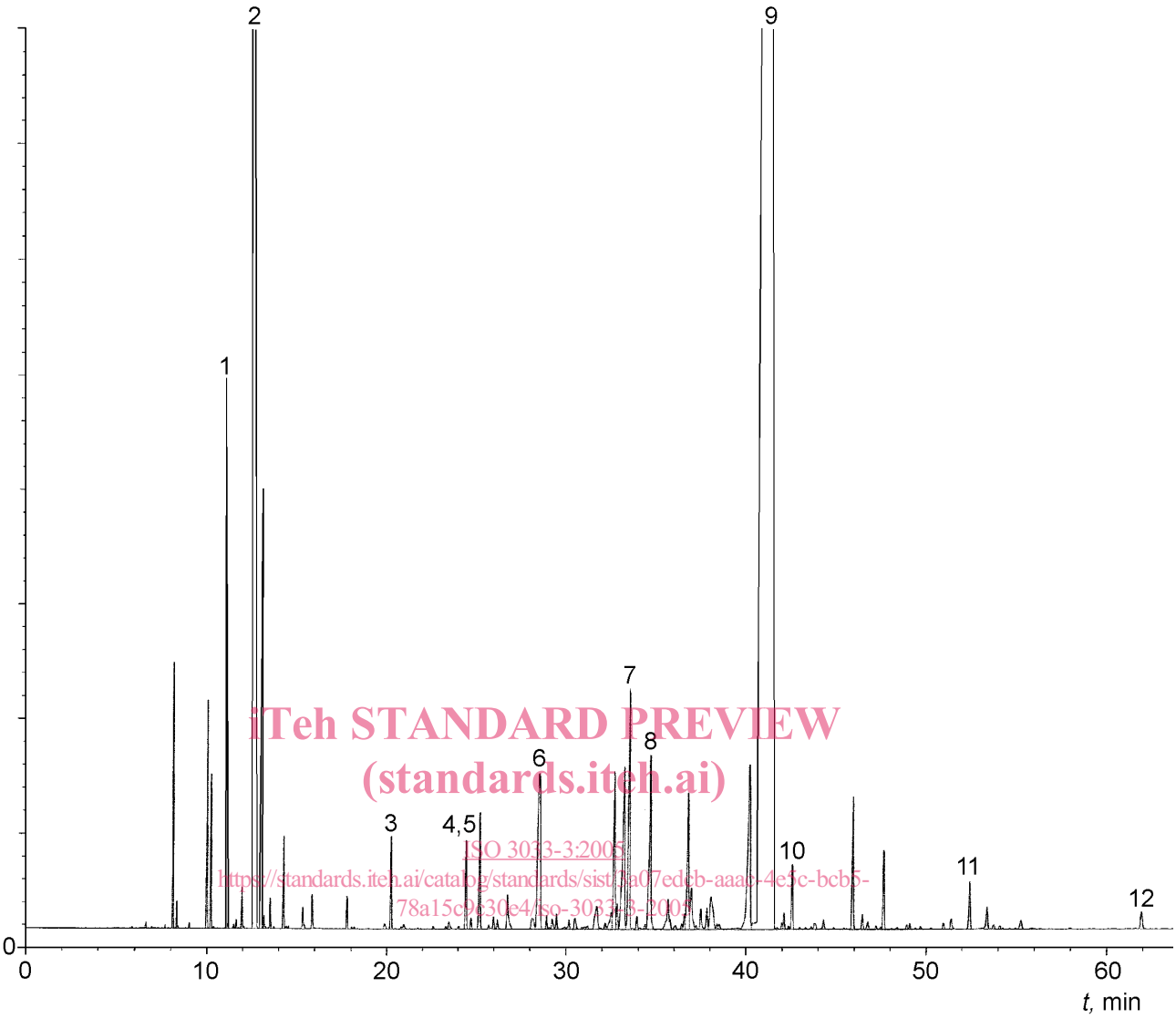
Peak identification

1 Myrcene + 3-octanol
2 Limonene
3 *trans*-Sabinene hydrate
4 Menthone
5 *cis*-Dihydrocarvone
6 Carvone
7 *trans*-Dihydrocarvyl acetate
8 *cis*-Carvyl acetate
9 *cis*-Jasmone
10 β-Bourbonene
11 Viridiflorol

Operating conditions

Column: fused silica capillary; length 30 m; internal diameter 0,25 mm
Stationary phase: poly(dimethyl siloxane) (DB-1®)
Film thickness: 0,25 µm
Oven temperature: isothermal at 75 °C for 5 min, then temperature programming from 75 °C to 100 °C at a rate of 5 °C/min, then from 100 °C to 220 °C at a rate of 6 °C/min and isothermal at 220 °C for 8,34 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Volume injected: 0,1 µl
Carrier gas flow rate: 1 ml/min
Split ratio: 1/100

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



Peak identification

1 Myrcene
2 Limonene
3 3-Octanol
4 Menthone
5 *trans*-Sabinene hydrate
6 β-Bourbonene
7 *cis*-Dihydrocarvone
8 *trans*-Dihydrocarvyl acetate
9 Carvone
10 *cis*-Carvyl acetate
11 *cis*-Jasmone
12 Viridiflorol

Operating conditions

Column: fused silica capillary; length 30 m; internal diameter 0,25 mm
Stationary phase: poly(ethylene glycol) (DB-WAX®)
Film thickness: 0,25 µm
Oven temperature: isothermal at 75 °C for 5 min, then programming temperature from 75 °C to 100 °C at a rate of 5 °C/min, then from 100 °C to 220 °C at a rate of 6 °C/min and isothermal at 220 °C for 8,34 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Volume injected: 0,1 µl
Carrier gas flow rate: 1 ml/min
Split ratio: 1/100

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