INTERNATIONAL STANDARD

ISO 3033-4

> First edition 2005-09-01

Oil of spearmint —

Part 4:

Scotch variety (Mentha × gracilis Sole)

Huile essentielle de menthe crepue (ou menthe verte) —

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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 3033-4 was prepared by Technical Committee ISO/TC 54, Essential oils.

This first edition of ISO 3033-4, together with ISO 3033-1:2005, ISO 3033-2:2005 and ISO 3033-3:2005, 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)

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Oil of spearmint —

Part 4:

Scotch variety (Mentha × gracilis Sole)

1 Scope

This part of ISO 3033 specifies certain characteristics of the oil of spearmint, Scotch variety ($\textit{Mentha} \times \textit{gracilis}$ Sole), 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, Scotch variety

essential oil obtained by steam distillation from the fresh above-ground parts of the flowering plant of *Mentha* × gracilis Sole, of Lamiaceae family

NOTE For information on the CAS number, see ISOUR 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 a 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 | |
| <i>trans</i> -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 | 0.50 | 0,6 | |
| cis-Jasmone | 0,2 | 0,7 | |
| β-Bourbonene | 1,0 | 2,0 | |
| Viridiflorol | Not detectable | | |

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 p-limonene 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

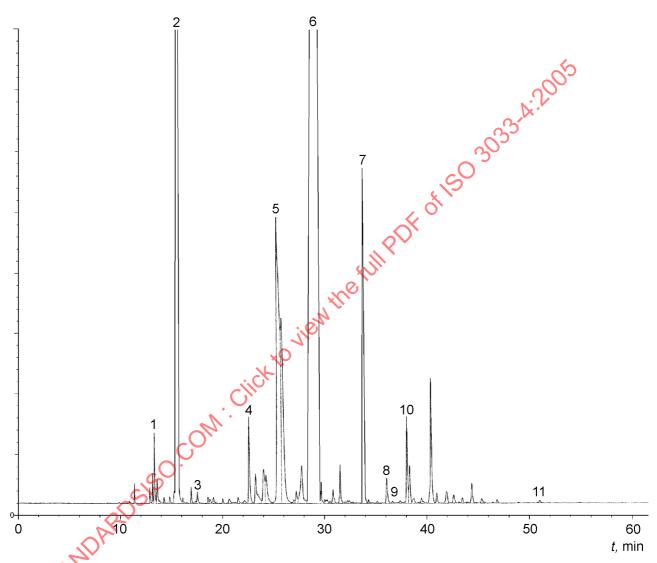
See ISO/TR 210 and ISO/TR 211.

^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 found is regarded to be predominantly L-carvone based on the physical testing. It is believed that there might be a small amount of p-carvone present but the exact quantity is unknown.

Annex A (informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of spearmint, Scotch variety (*Mentha* × *gracilis* Sole)



Peak identification

- 1 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 $^{\otimes}$)

Film thickness: 0,25 µm

Oven temperature: isothermal at 75 $^{\circ}$ C for 5 min, then temperature programming from 75 $^{\circ}$ C to 100 $^{\circ}$ C at a rate of 5 $^{\circ}$ C/min, then from 100 $^{\circ}$ C to 220 $^{\circ}$ C at a rate of 6 $^{\circ}$ C/min

and isothermal at 220 $^{\circ}\text{C}$ for 8,34 min

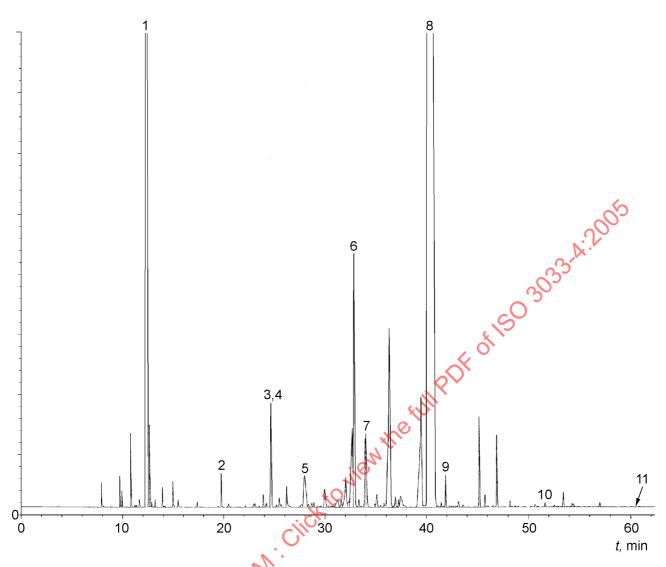
Injector temperature: 230 °C Detector temperature: 260 °C

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 Limonene
- 2 3-Octanol
- 3 Menthone
- 4 trans-Sabinene hydrate
- 5 β-Bourbonene
- 6 cis-Dihydrocarvone
- 7 trans-Dihydrocarvyl acetate
- 8 Carvone
- 9 cis-Carvyl acetate
- 10 cis-Jasmone
- 11 Viridiflorol (not detected)

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 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.2 — Typical chromatogram taken on a polar column

Annex B (informative)

Flashpoint

B.1 General information

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

A comparative study on the relevant methods of analysis (see ISO/TR 11018) concluded that it was difficult to recommend a single apparatus for standardization purposes, given that:

- there is wide variation in the chemical composition of essential oils;
- the volume of the sample needed in certain requirements would be too costly for highpriced essential oils;
- ss there are several different types of equipment which can be used for the determination, users cannot be expected to use one specified type only.

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

The equipment with which this value was obtained should be specified.

For further information, see ISO/TR 11018.

B.2 Flashpoint of the essential oil of spearmint, Scotch variety

The mean value is +58 °C.

NOTE Obtained with "Setaflash" equipment.

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