

Oil of spearmint —

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

ICS 71.100.60

National foreword

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Summary of pages

This document comprises a front cover, an inside front cover, the ISO title page, pages ii and iii, a blank page, pages 1 to 10, an inside back cover and a back cover.

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Amendments issued since publication

Amd. No.	Date	Comments

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 7 November 2005

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INTERNATIONAL
STANDARD

ISO
3033-2

First edition
2005-09-01

Oil of spearmint —

Part 2:

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

Huile essentielle de menthe crépue (ou menthe verte) —

*Partie 2: Type Chine (80 % et 60 %) (*Mentha viridis* L. var. *crispa* Benth.), huile bidistillée*



Reference number
ISO 3033-2:2005(E)

Foreword

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ISO 3033-2 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

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

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

1 Scope

This part of ISO 3033 specifies certain characteristics of the oil of spearmint, Chinese type (80 % and 60 %) (*Mentha viridis* L. var. *crispa* Benth.), 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, Chinese type, redistilled

essential oil obtained by steam distillation from the fresh above-ground parts of the flowering plant of *Mentha viridis* L. var. *crispa* Benth., of Lamiaceae family

NOTE 1 *Mentha viridis* L. var. *crispa* Benth. is also called *Mentha spicata* L. var. *crispa* Benth.

NOTE 2 It may be enriched in carvone by redistillation.

NOTE 3 For the 80 % Chinese type, the oil obtained by steam distillation is redistilled to yield a carvone content of 80 % minimum. For the 60 % Chinese type, the oil obtained by steam distillation is redistilled to yield a carvone content of 60 % minimum.

NOTE 4 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 a herbaceous note.

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

Chinese type, 80 %		Chinese type, 60 %	
min.	max.	min.	max.
0,942	0,955	0,918	0,938

4.5 Refractive index at 20 °C

Chinese type, 80 %		Chinese type, 60 %	
min.	max.	min.	max.
1,488	1,496	1,484	1,491

4.6 Optical rotation at 20 °C

Chinese type, 80 %	Chinese type, 60 %
Between -55° and -50°	Between -60° 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.

Minimum: 299, corresponding to a carbonyl compound content of 80 %, 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	Chinese type, 80 %		Chinese type, 60 %	
	min. %	max. %	min. %	max. %
Limonene ^a	0,5	3,0	10,0	22,0
3-Octanol	traces	0,4	0,2	0,8
Menthone ^b	0,8	2,0	0,8	2,0
<i>trans</i> -Sabinene hydrate	—	0,1	—	0,1
<i>cis</i> -Dihydrocarvone	2,0	4,0	2,0	4,0
Carvone ^c	78,0	84,0	57,0	66,0
<i>trans</i> -Dihydrocarvyl acetate	1,5	4,0	1,5	3,5
<i>cis</i> -Carvyl acetate	0,1	0,3	0,1	0,3
<i>cis</i> -Jasmone	—	0,1	—	0,1
β -Bourbonene	0,5	1,2	0,5	1,2
Viridiflorol	not detectable		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 found is 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.

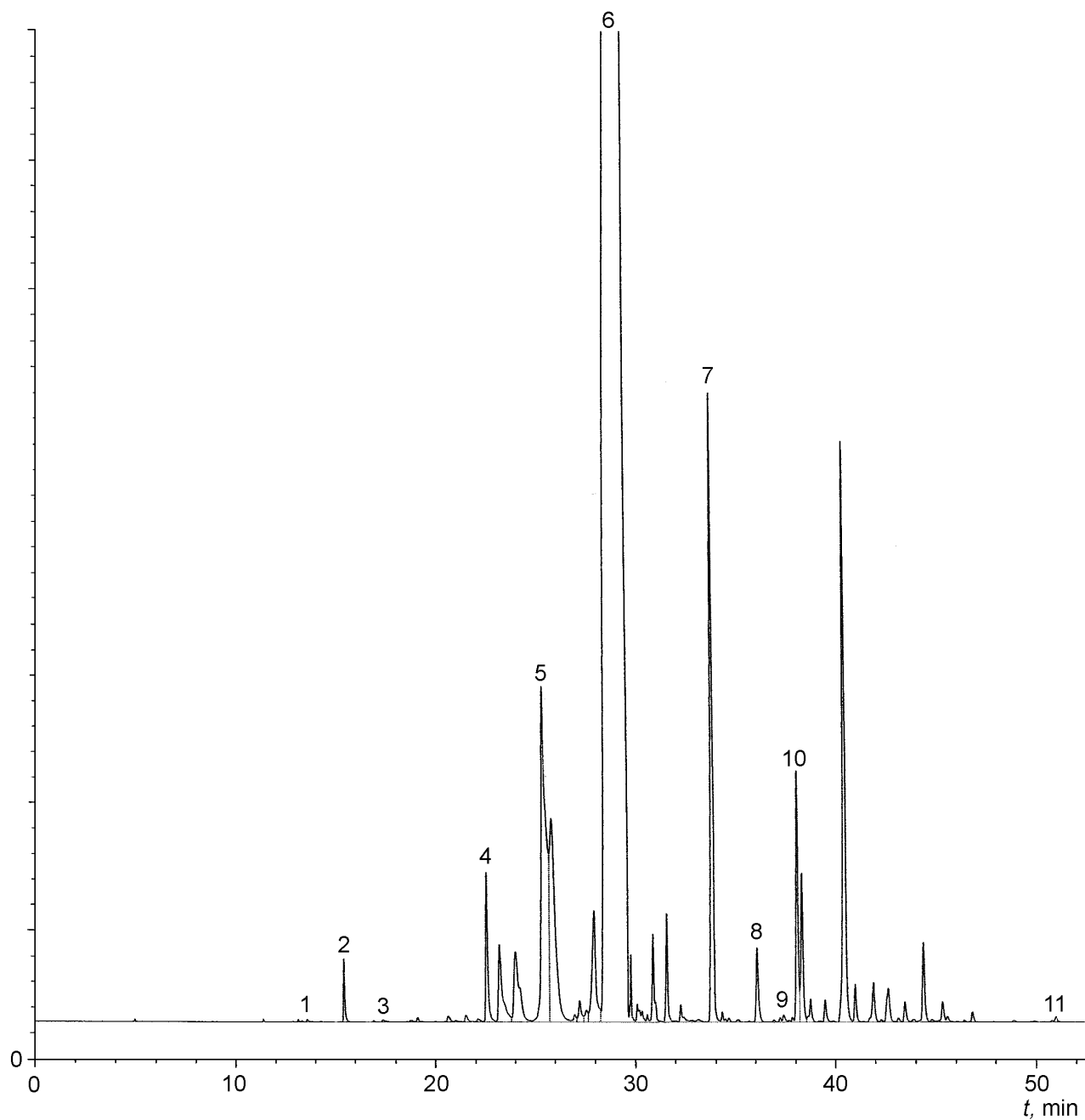
7 Packaging, labelling, marking and storage

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

A.1 Chinese type, 80 %

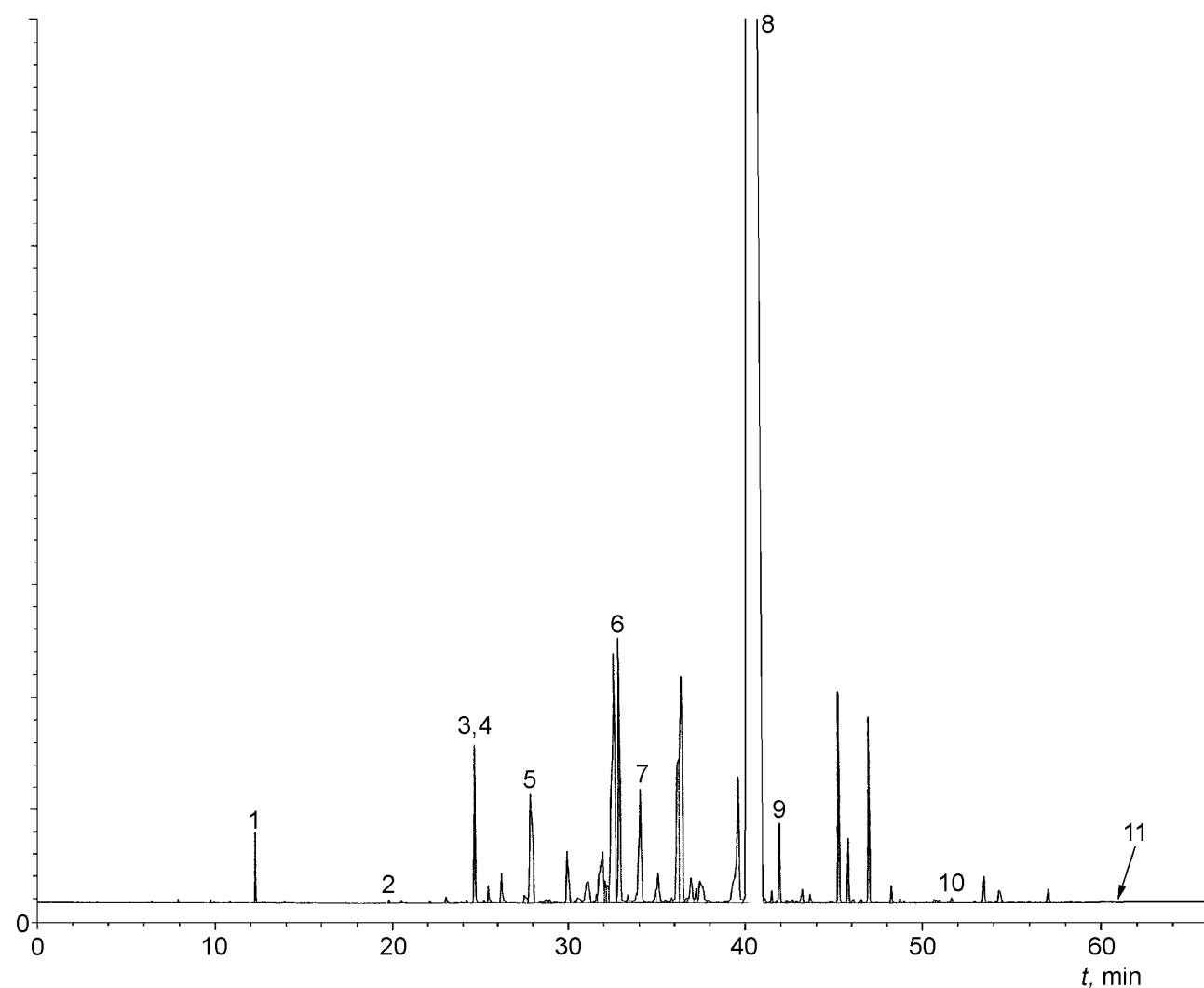
**Peak identification**

- 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[®])
 Film thickness: 0,25 μ m
 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 μ l
 Carrier gas flow rate: 1 ml/min
 Split ratio: 1/100

Figure A.1 — Typical chromatogram of Chinese type, 80 %, taken on an apolar column

**Peak identification**

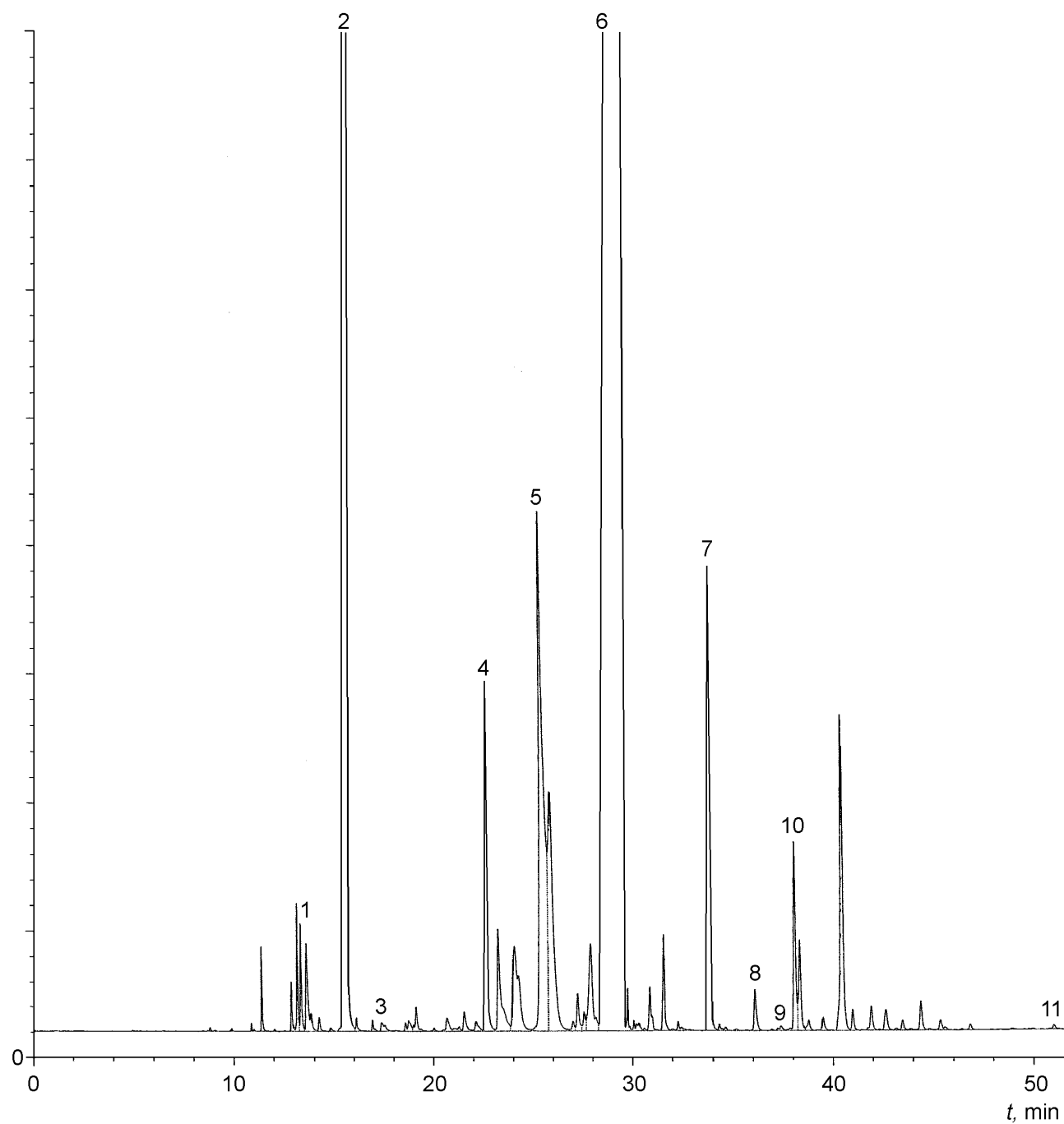
- 1 Limonene
- 2 3-Octanol
- 3 Menthone
- 4 *trans*-Sabinene hydrate
- 5 β -Bourbonene
- 6 *cis*-Dihydrocarvone
- 7 *trans*-Dihydrocarvyl acetate
- 8 Carvone
- 9 *cis*-Carvyl acetate
- 10 *cis*-Jasmone
- 11 Viridiflorol (not detected)

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 μ m
 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 μ l
 Carrier gas flow rate: 1 ml/min
 Split ratio: 1/100

Figure A.2 — Typical chromatogram of Chinese type, 80 %, taken on a polar column

A.2 Chinese type, 60 %

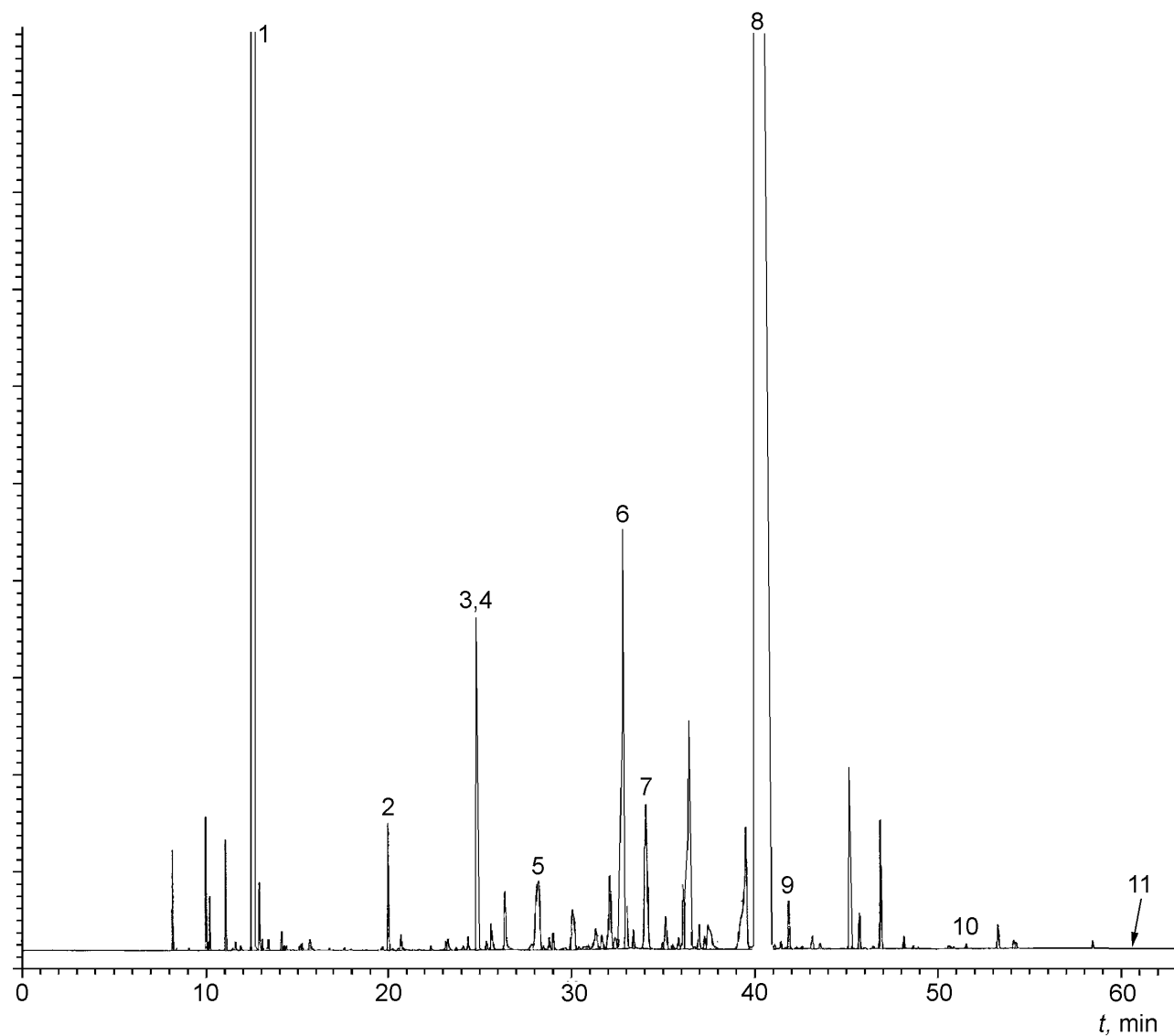
**Peak identification**

- 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[®])
 Film thickness: 0,25 μ m
 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 μ l
 Carrier gas flow rate: 1 ml/min
 Split ratio: 1/100

Figure A.3 — Typical chromatogram of Chinese type, 60 %, 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 μ m
 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 μ l
 Carrier gas flow rate: 1 ml/min
 Split ratio: 1/100

Figure A.4 — Typical chromatogram of Chinese type, 60 %, 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 high-priced essential oils;
- as 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, Chinese type (80 % and 60 %), redistilled oil

The mean value for Chinese type, 60 %, is +58 °C.

The mean value for Chinese type, 80 %, is +74 °C.

NOTE Obtained with “Setaflash” equipment.

Bibliography

- [1] ISO/TR 11018, *Essential oils — General guidance on the determination of flashpoint*
- [2] ISO/TR 21092, *Essential oils — Characterization*

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