INTERNATIONAL STANDARD

ISO 9844

Second edition
2006-03-01

Oil of bitter orange (*Citrus aurantium* L.)

Huile essentielle d'orange amère (Citrus aurantium *L.*)

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Contents

Page

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 9844 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 9844:1991), which has been technically revised.

Oil of bitter orange (*Citrus aurantium* L.)

1 Scope

This International Standard specifies certain characteristics of the oil of bitter orange (*Citrus aurantium* L.), 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 4715, *Essential oils — Quantitative evaluation of residue on evaporation*

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 bitter orange
essential oil obtained by expression, without heating, by mechanical treatment, from the pericarp of the fruit of *Citrus aurantium* L., of the Rutaceae family

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

4 Requirements

4.1 Appearance

Liquid.

4.2 Colour

Pale yellow to brownish green.

4.3 Odour

Characteristic of the outer part of bitter orange peel.

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

American type		Equatorial type		Mediterranean type	
min.	max.	min.	max.	min.	max.
0,840	0,860	0,845	0,860	0,840	0,860

4.5 Refractive index at 20 °C

American type		Equatorial type		Mediterranean type	
min.	max.	min.	max.	min.	max.
1,472	1,476	1,473	1,478	1,472	1,476

4.6 Optical rotation at 20 °C

American type	Equatorial type	Mediterranean type
+ 88° to + 98°	+ 88° to + 95°	+ 88° to + 98°

4.7 Miscibility in ethanol, 90 % (volume fraction), at 20 °C

It shall not be necessary to use more than 8 volumes of ethanol, 90 % (volume fraction), to obtain a clear solution with 1 volume of essential oil.

4.8 Residue on evaporation

American type		Equatorial type		Mediterranean type	
min.	max.	min.	max.	min.	max.
3,5 %	6,0 %	3,0 %	6,0 %	3,5 %	6,0 %

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.

4.10 Flashpoint

Information on the flashpoint is given in Annex B.

Table 1 — Chromatographic profile

Components	American type		Equatorial type		Mediterranean type	
	Minimum %	Maximum %	Minimum %	Maximum %	Minimum %	Maximum %
α-Pinene	0,3	0,7	0,3	0,6	0,2	0,7
β-Pinene	0,3	1,2	0,3	0,8	0,2	1,2
Myrcene	1,6	2,0	1,6	2,0	1,5	3,0
Limonene	93,0	95,0	92,0	95,0	93,0	95,0
Linalool	0,1	0,4	0,1	0,4	0,1	0,4
Linalyl acetate	0,6	1,0	traces	0,5	0,5	1,0
Geranyl acetate	0,1	0,3	nd [a]	0,3	0,1	0,3
β-Caryophyllene	traces	0,2	traces	0,1	traces	0,2
Germacrene D	0,1	0,2	traces	0,1	traces	0,2
trans-β-Nerolidol	traces	0,2	nd [a]	0,3	traces	0,2
Octanal	traces	0,2	nd [a]	0,3	traces	0,1
Decanal	0,1	0,2	nd [a]	0,2	traces	0,3

[a] Undetected.

NOTE The chromatographic profile is normative, contrary to the 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, 90 % (volume fraction), at 20 °C

See ISO 875.

6.5 Residue on evaporation

See ISO 4715.

6.6 Chromatographic profile

See ISO 11024-1 and ISO 11024-2.

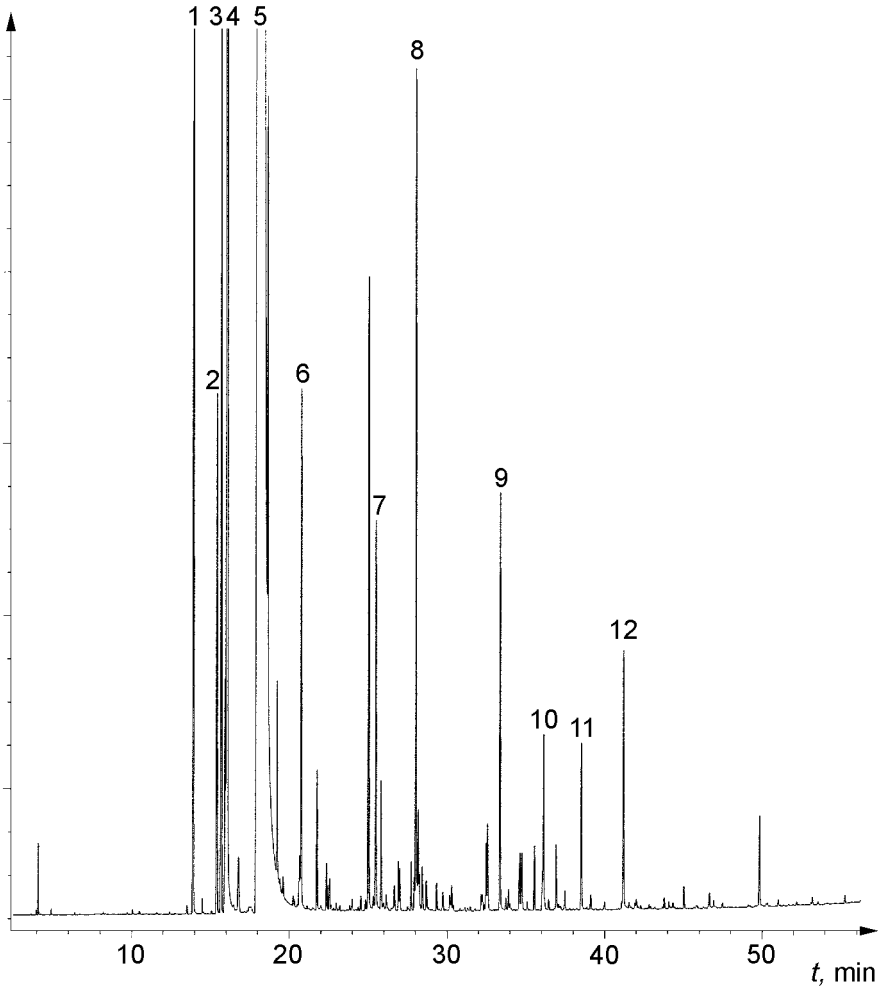
7 Packaging, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

Annex A
(informative)

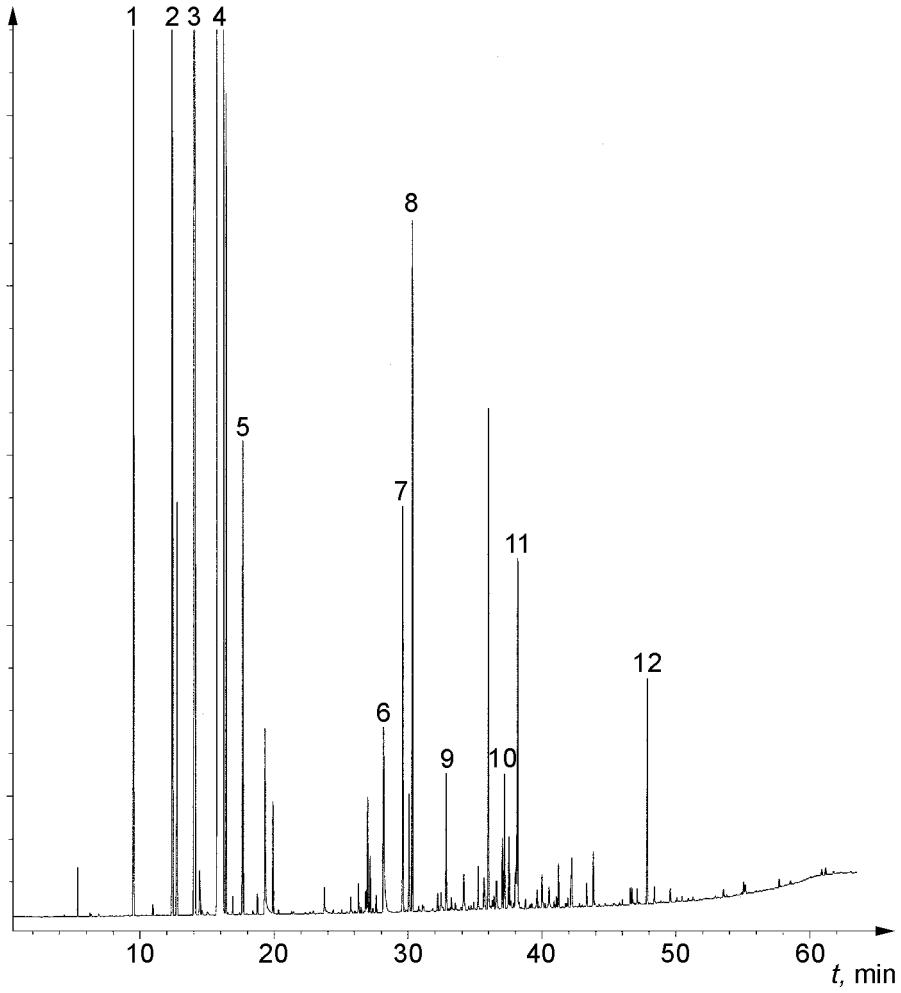
Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.)

A.1 Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.), American type



Peak identification		Operating conditions
1	α-Pinene	Column: methyl silicone; length 50 m; internal diameter 0,2 mm
2	β-Pinene	Stationary phase: poly(dimethyl siloxane) (SP5®)
3	Octanal	Film thickness: 0,33 µm
4	Myrcene	Oven temperature: temperature programming from 60 °C to 240 °C at a rate of 3 °C/min, then
5	Limonene	isothermal at 240 °C for 30 min
6	Linalool	Injector temperature: 270 °C
7	Decanal	Detector temperature: 280 °C
8	Linalyl acetate	Detector: flame ionization type
9	Geranyl acetate	Carrier gas: helium
10	β-Caryophyllene	Volume injected: 0,2 µl
11	Germacrene D	Carrier gas flow rate: 1 ml/min
12	*trans*-β-Nerolidol	Split ratio: 1/50

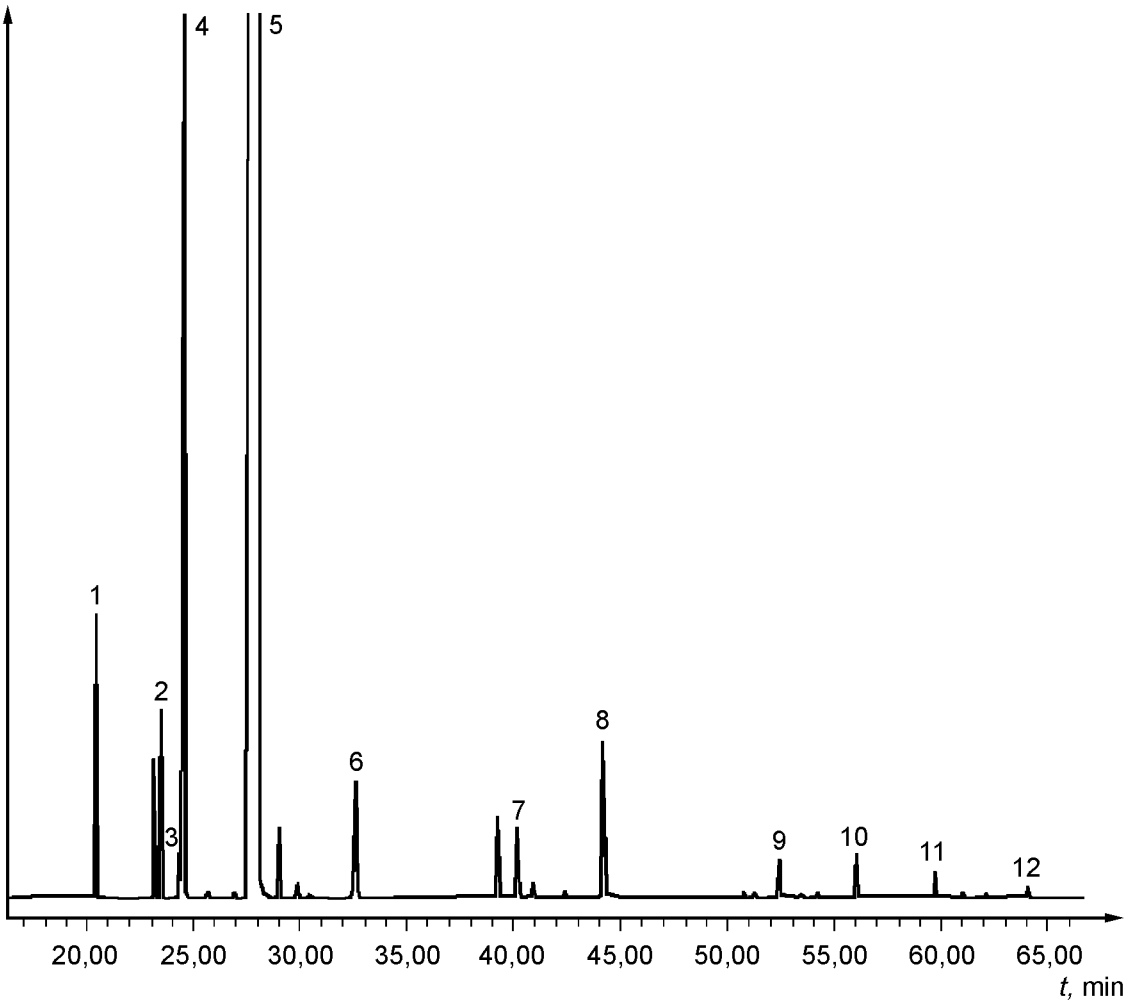
Figure A.1 — Typical chromatogram taken on an apolar column



Peak identification		Operating conditions
1	α-Pinene	Column: Carbowax; length 50 m; internal diameter 0,2 mm
2	β-Pinene	Stationary phase: poly(ethylene glycol) (Suplecowax-10®)
3	Myrcene	Film thickness: 0,4 µm
4	Limonene	Oven temperature: temperature programming from 60 °C to 240 °C at a rate of 3 °C/min, then
5	Octanal	isothermal at 240 °C for 30 min
6	Decanal	Injector temperature: 270 °C
7	Linalool	Detector temperature: 280 °C
8	Linalyl acetate	Detector: flame ionization type
9	β-Caryophyllene	Carrier gas: helium
10	Germacrene D	Volume injected: 0,2 µl
11	Geranyl acetate	Carrier gas flow rate: 1 ml/min
12	*trans*-β-Nerolidol	Split ratio: 1/50

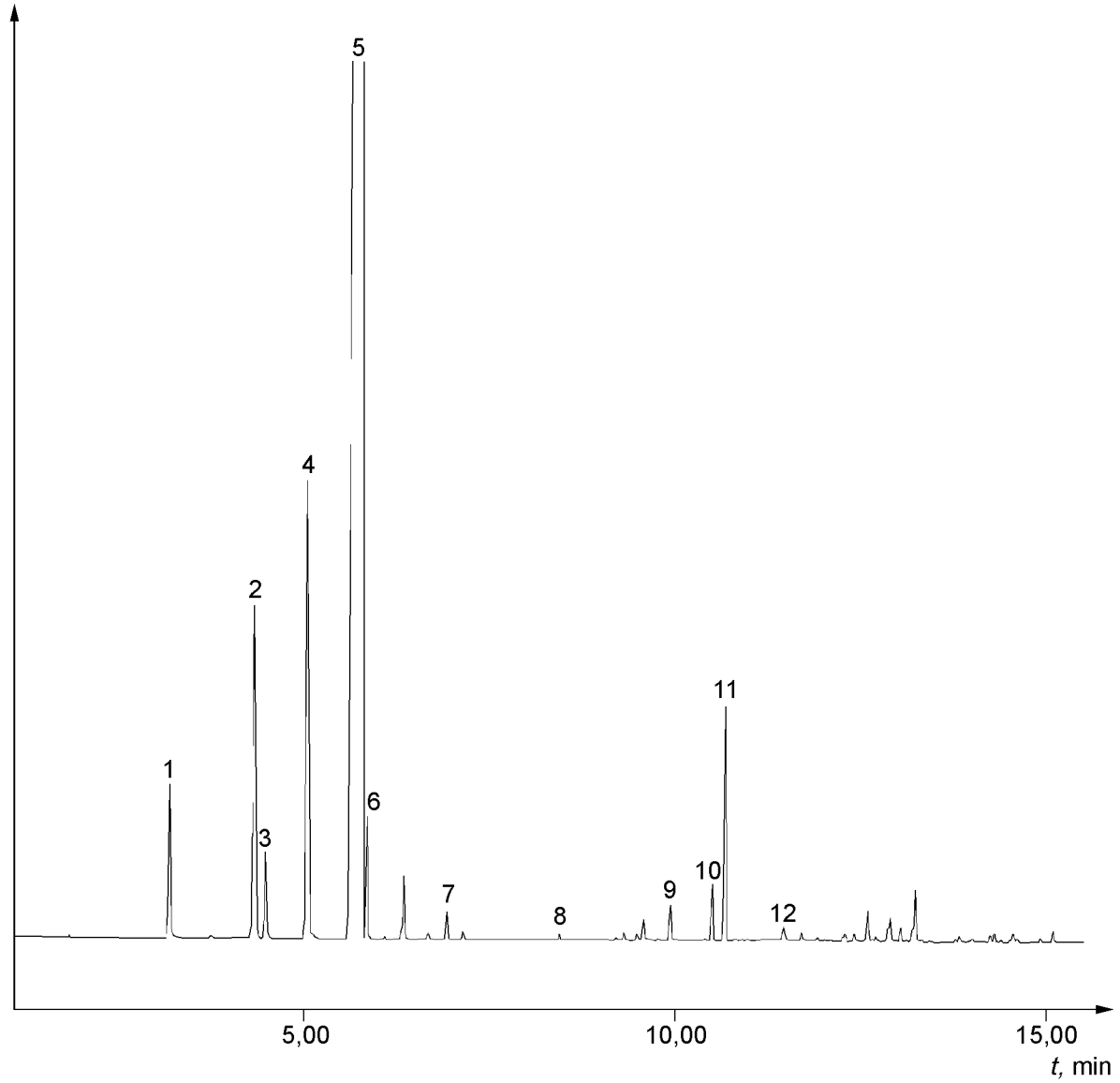
Figure A.2 — Typical chromatogram taken on a polar column

A.2 Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.), Equatorial type



Peak identification		Operating conditions
1	α-Pinene	Column: silica capillary; length 60 m; internal diameter 0,25 mm
2	β-Pinene	Stationary phase: poly(dimethyl siloxane) (SolGel-1ms)
3	Octanal	Film thickness: 0,25 µm
4	Myrcene	Oven temperature: isothermal at 60 °C for 10 min, then temperature programming from 60 °C
5	Limonene	to 300 °C at a rate of 2 °C/min, then isothermal at 300 °C for 20 min
6	Linalool	Injector temperature: 250 °C
7	Decanal	Detector temperature: 250 °C
8	Linalyl acetate	Detector: mass spectrometer
9	Geranyl acetate	Carrier gas: helium
10	β-Caryophyllene	Volume injected: 0,2 µl
11	Germacrene D	Carrier gas flow rate: 1 ml/min
12	*trans*-β-Nerolidol	Split ratio: 1/60

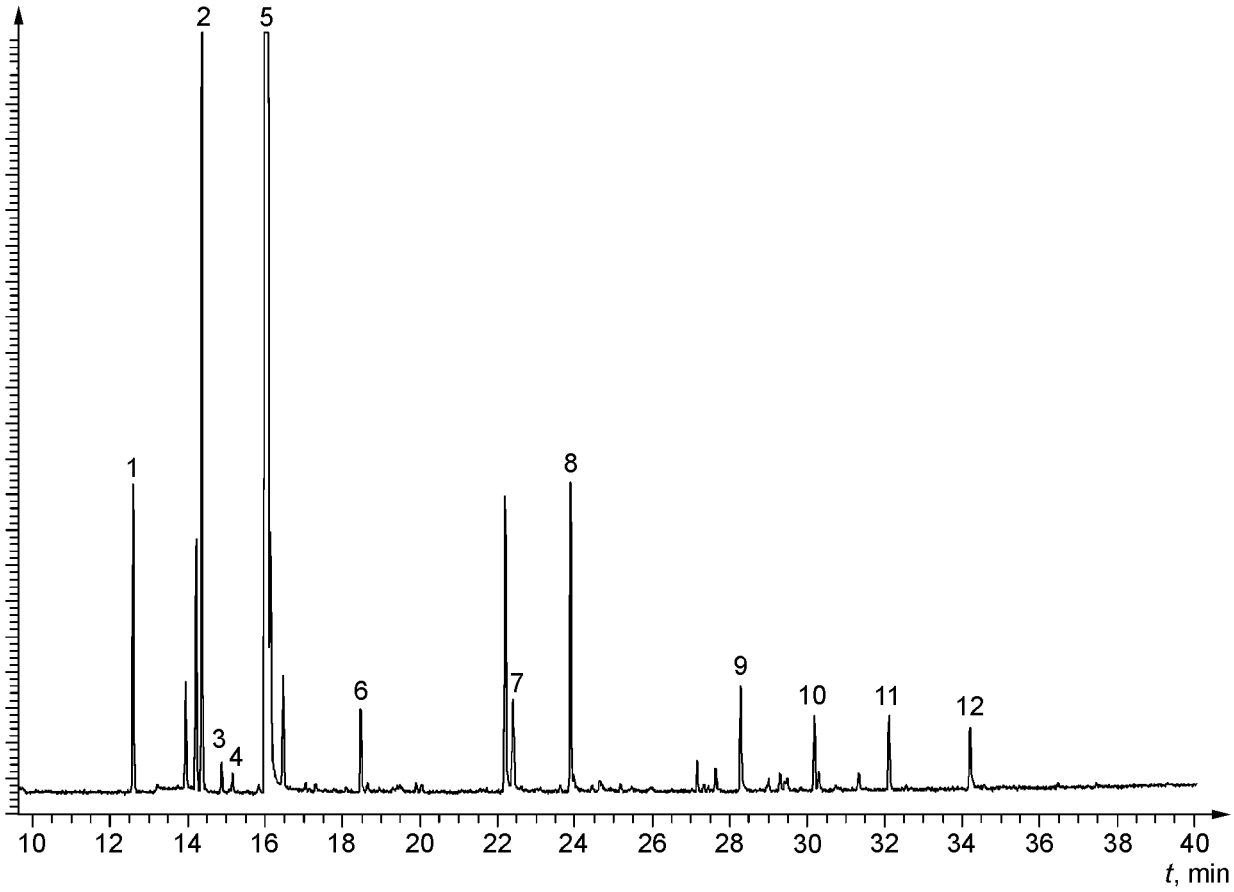
Figure A.3 — Typical chromatogram taken on an apolar column



Peak identification		Operating conditions
1	α-Pinene	Column: silica capillary; length 20 m; internal diameter 0,10 mm
2	β-Pinene	Stationary phase: poly(ethylene glycol) (Suplecowax-10®)
3	Sabinene	Film thickness: 0,20 µm
4	Myrcene	Oven temperature: isothermal at 50 °C for 1 min, then temperature programming from 50 °C
5	Limonene	to 200 °C at a rate of 10 °C/min, then isothermal for 13 min
6	β-Phellandrene	Injector temperature: 250 °C
7	Octanal	Detector temperature: 250 °C
8	Nonanal	Detector: flame ionization type
9	Decanal	Carrier gas: helium
10	Linalool	Volume injected: 0,2 µl
11	Linalyl acetate	Carrier gas flow rate: 30 ml/min
12	β-Caryophyllene	Split ratio: 1/650

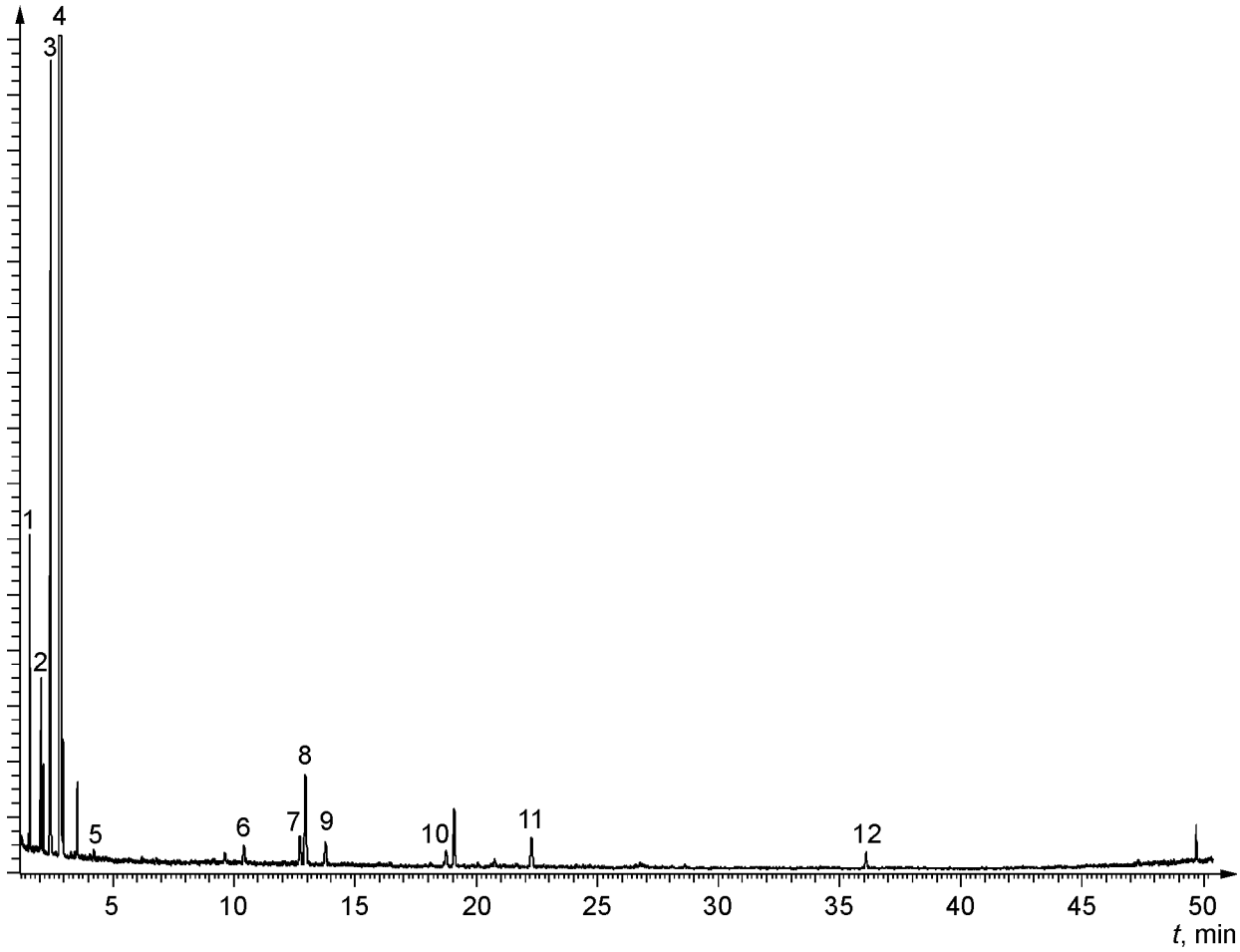
Figure A.4 — Typical chromatogram taken on a polar column

A.3 Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.), Mediterranean type



Peak identification		Operating conditions
1	α-Pinene	Column: silica capillary; length 60 m; internal diameter 0,25 mm
2	β-Pinene	Stationary phase: poly(5 % diphenyl/95 % dimethyl siloxane) (DB5 MS®)
3	Myrcene	Film thickness: 0,25 µm
4	Octanal	Oven temperature: isothermal at 40 °C for 5 min, then temperature programming from 40 °C
5	Limonene	to 220 °C at a rate of 4 °C/min, then isothermal at 220 °C for 5 min
6	Linalool	Injector temperature: 260 °C
7	Decanal	Detector temperature: 280 °C
8	Linalyl acetate	Detector: MS type, Full Scan (50-200)
9	Geranyl acetate	Carrier gas: helium
10	β-Caryophyllene	Volume injected: 0,3 µl
11	Germacrene D	Carrier gas flow rate: 1 ml/min
12	*trans*-β-Nerolidol	Split ratio: 1/150

Figure A.5 — Typical chromatogram taken on an apolar column



Peak identification		Operating conditions
1	α-Pinene	Column: silica capillary; length 60 m, internal diameter 0,32 mm
2	β-Pinene	Stationary phase: poly(ethylene glycol) (Suplecowax-10®)
3	Myrcene	Film thickness: 0,25 µm
4	Limonene	Oven temperature: isothermal at 75 °C for 4 min, then temperature programming from 75 °C
5	Octanal	to 150 °C at a rate of 2 °C/min, then from 150 °C to 240 °C at a rate of 5 °C/min, then
6	Decanal	isothermal at 240 °C for 20 min
7	Linalool	Injector temperature: 225 °C
8	Linalyl acetate	Detector temperature: 225 °C
9	β-Caryophyllene	Detector: MS type, Full Scan (50-200)
10	Germacrene D	Carrier gas: helium
11	Geranyl acetate	Volume injected: 0,03 µl
12	*trans*-β-Nerolidol	Carrier gas flow rate: 1,5 ml/min
		Split ratio: 1/100

Figure A.6 — 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 a wide variation in the chemical composition of essential oils;

— the volume of the sample needed in certain test methods 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 bitter orange

B.2.1 Flashpoint of the essential oil of bitter orange, American type

The mean value is +47 °C.

NOTE Obtained with “Pensky Martens” equipment.

B.2.2 Flashpoint of the essential oil of bitter orange, Equatorial type

The mean value is +50 °C.

NOTE Obtained with “miniflash-petrotest France” equipment.

B.2.3 Flashpoint of the essential oil of bitter orange, Mediterranean type

The mean value is +47 °C.

NOTE Obtained with “Pensky Martens” equipment.

Bibliography

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

[2] ISO/TR 21092, *Essential oils — Characterization*

ICS 71.100.60

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