INTERNATIONAL STANDARD

ISO 3809

Third edition
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Oil of lime (cold pressed), Mexican type [*Citrus aurantifolia* (Christm.) Swingle], obtained by mechanical means

*Huile essentielle de limette (exprimée à froid), type Mexique [*Citrus aurantifolia *(Christm.) Swingle], obtenue par procédés mécaniques*

Reference number
ISO 3809:2004(E)

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Foreword

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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 3809 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This third edition cancels and replaces the second edition (ISO 3809:1987), which has been technically revised.

Oil of lime (cold pressed), Mexican type [*Citrus aurantifolia* (Christm.) Swingle], obtained by mechanical means

1 Scope

This International Standard specifies certain characteristics of the oil of lime (cold pressed), Mexican type [*Citrus aurantifolia* (Christm.) Swingle], 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 1271, *Essential oils — Determination of carbonyl value — Free hydroxylamine method*

ISO 4715, *Essential oils — Quantitative evaluation of residue on evaporation*

ISO 4735, *Oils of citrus — Determination of CD value by ultraviolet spectrophotometric analysis*

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 lime (cold pressed), Mexican type (type A)
essential oil obtained by centrifuging the emulsion of water, juice and oil obtained by crushing the whole fruits of C*itrus aurantifolia* (Christm.) Swingle, of the Rutaceae family

3.2
oil of lime (cold pressed), Mexican type (type B)
essential oil obtained by grating and/or squeezing the peel of fruits of *Citrus aurantifolia* (Christm.) Swingle, of the Rutaceae family, in the presence of water, followed by centrifuging the resulting emulsion of water and oil

NOTE 1 The principal areas of production are Mexico, the countries of Central America and the islands of the Caribbean.

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

4 Requirements

4.1 Appearance

Type A	Type B
Clear liquid, in which a waxy precipitate is usually present	

4.2 Colour

Type A	Type B
From yellowish green to green	Green to dark green

4.3 Odour

Type A	Type B
Characteristic of fresh lime peel. Fresh citrus lime-like odour	Fresh, with a note reminiscent of the pericarp. Fresh citrus lime odour with a pronounced juicy characteristic

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

Type A		Type B	
min.	max.	min.	max.
0,875 0	0,884 0	0,880 0	0,888 0

4.5 Refractive index at 20 °C

Type A		Type B	
min.	max.	min.	max.
1,482 0	1,486 0	1,484 0	1,488 0

4.6 Optical rotation at 20 °C

Type A	Type B
Between +35,0° and +41,0°	This determination is often not possible because oils of this type are intensely coloured

4.7 Carbonyl value

Type A		Type B	
min.	max.	min.	max.
16 (corresponding to 4,5 % of carbonyl compounds expressed as citral)	31 (corresponding to 8,5 % of carbonyl compounds expressed as citral)	18 (corresponding to 5 % of carbonyl compounds expressed as citral)	35 (corresponding to 9,5 % of carbonyl compounds expressed as citral)

4.8 Residue on evaporation

Type A		Type B	
min.	max.	min.	max.
10,0 %	14,5 %	13,0 %	19,0 %

4.9 CD value

Type A	Type B
min.	min.
18,2	23,6

4.10 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 Tables 1 and 2, for type A and type B respectively, shall be identified. The proportions of these components, indicated by the integrator, shall be as shown in Tables 1 and 2. This constitutes the chromatographic profile of the essential oil.

4.11 Flashpoint

Information on the flashpoint is given in Annex B.

5 Sampling

See ISO 212.

Minimum volume of test sample: 25 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 Carbonyl value

See ISO 1271.

Test portion: 5 g.

Standing time: 15 min.

6.5 Residue on evaporation

See ISO 4715.

Test portion: 5 g.

Evaporation time: 6 h.

6.6 CD value

See ISO 4735.

Point B: 370 nm approximately.

Maximum value: 312 nm to 315 nm approximately.

Point A: 280 nm approximately.

Dilution of 0,025 g of oil in 100 ml of 90 % ethanol (volume fraction).

Table 1 — Chromatographic profile (type A)

Component	Minimum %	Maximum %
α-Pinene	2,0	3,0
Sabinene	1,8	4,0
β-Pinene	18,0	24,0
Myrcene	1,0	2,0
p-Cymene	—	0,5
Limonene	42,0	50,0
γ-Terpinene	8,0	11,0
Terpinen-4-ol	0,2	0,6
α-Terpineol	0,2	0,6
n-Decanal	0,05	0,3
Neral	1,2	2,0
Geranial	2,0	3,0
Neryl acetate	0,1	0,35
Geranyl acetate	0,2	0,4
β-Caryophyllene	0,5	1,5
α-Bergamotene	1,0	1,9
α-Farnesene	0,75	1,75
β-Bisabolene	1,0	1,5

NOTE 1 The chromatographic profile is normative, contrary to typical chromatograms given for information in Annex A.

NOTE 2 Expressed oils of lime may contain furo-coumarins. The bergapten content should be less than 2 000 mg/kg.

6.7 Chromatographic profile

See ISO 11024-1 and ISO 11024-2.

7 Packaging, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

Table 2 — Chromatographic profile (type B)

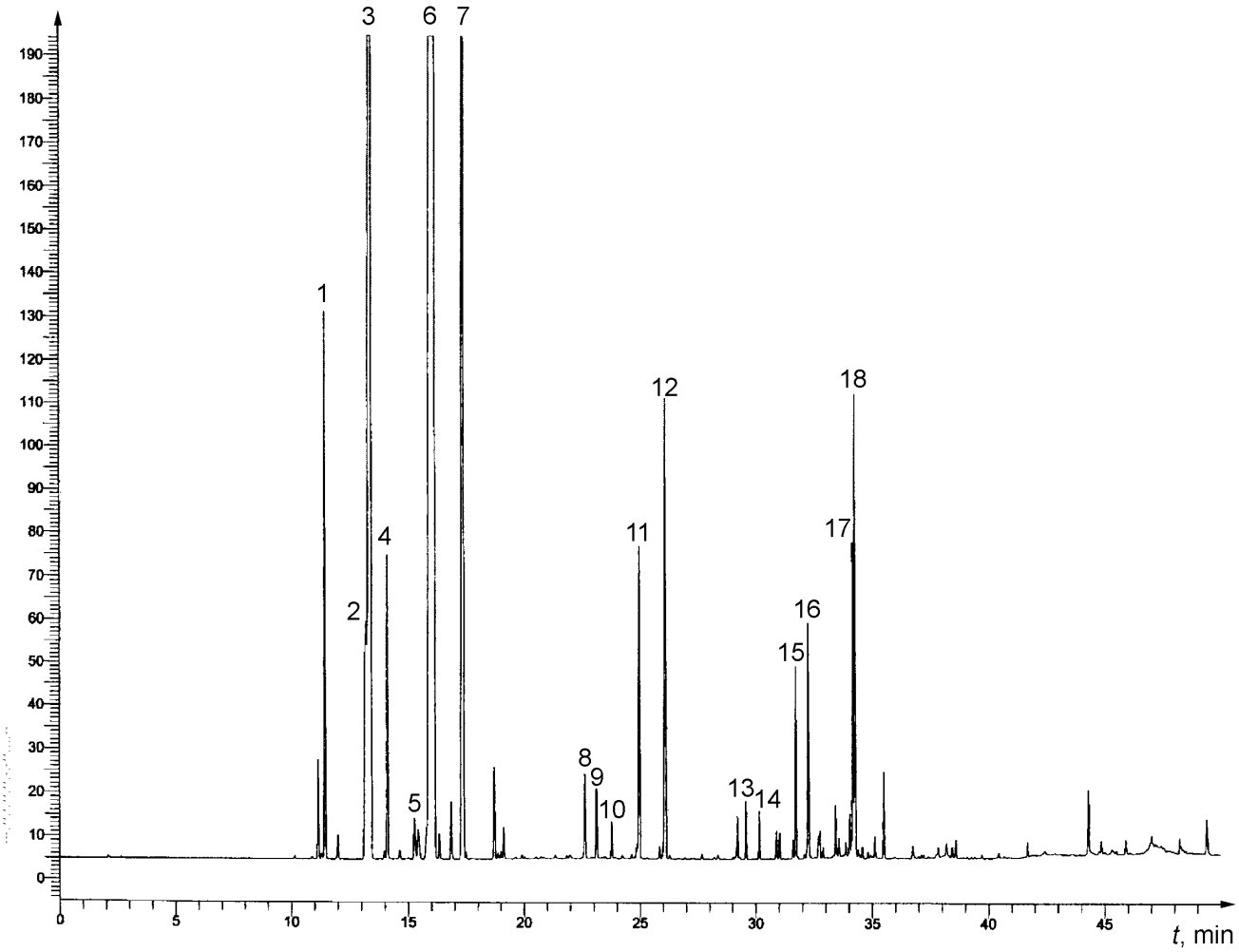
Component	Minimum %	Maximum %
α-Pinene	1,7	2,0
Sabinene	2,0	3,0
β-Pinene	17,0	19,0
Myrcene	1,4	1,8
Neryl acetate	0,0	0,25
γ-Terpinene	9,0	9,7
Terpinen-4-ol	0,2	0,6
p-Cymene	—	0,5
Limonene	38,0	44,0
α-Terpineol	0,3	0,6
n-Decanal	0,15	0,35
Neral	2,0	2,5
Geranial	3,0	3,7
Geranyl acetate	0,3	0,6
β-Caryophyllene	1,5	1,9
α-Bergamotene	0,5	0,7
β-Bisabolene	4,0	4,5

NOTE 1 The chromatographic profile is normative, contrary to typical chromatograms given for information in Annex A.

NOTE 2 Expressed oils of lime may contain furo-coumarins. The bergapten content should be less than 2 000 mg/kg.

Annex A
(informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of lime (cold pressed), Mexican type [*Citrus aurantifolia* (Christm.) Swingle], obtained by mechanical means



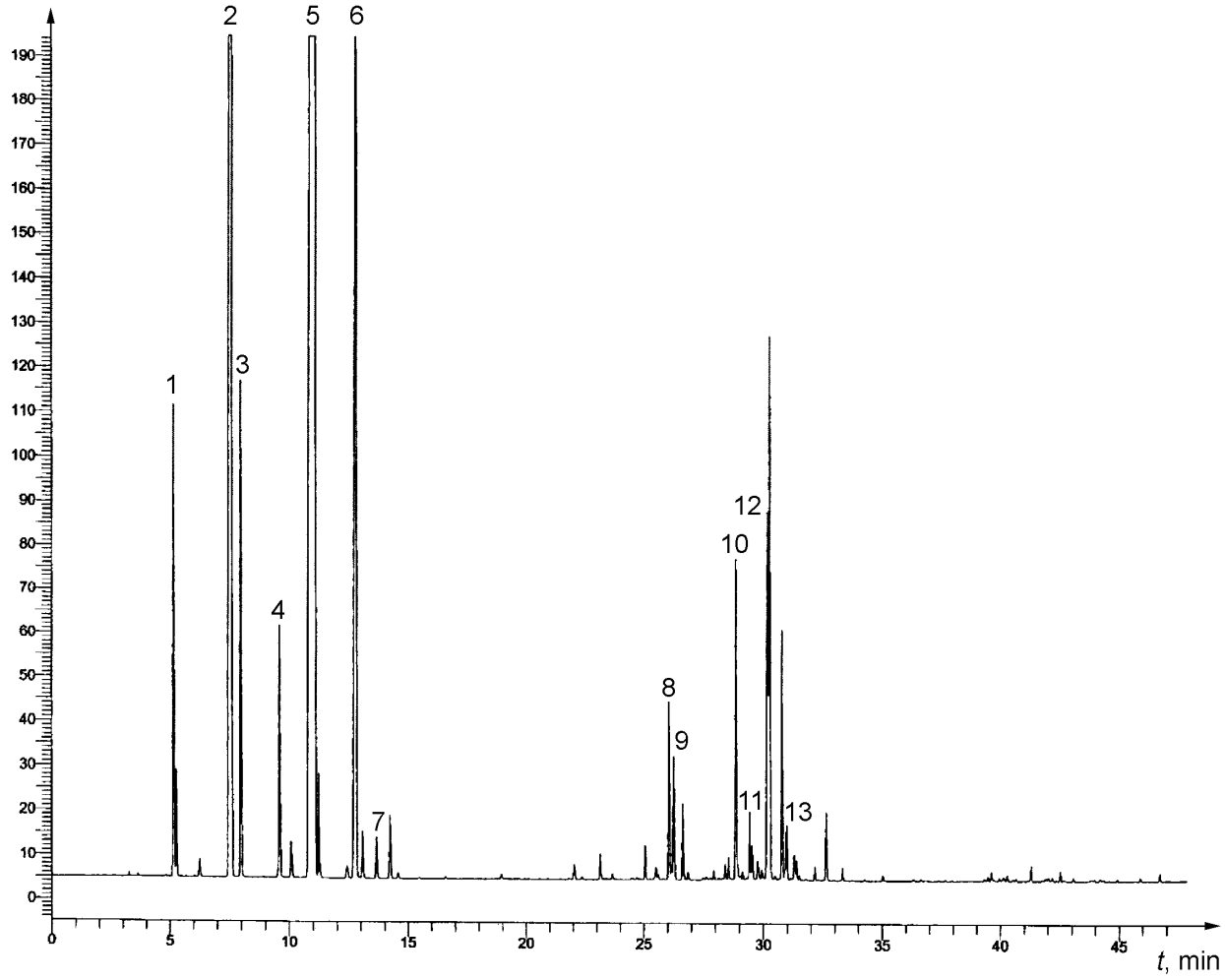
Peak identification

1	α-Pinene	10	*n*-Decanal
2	Sabinene	11	Neral
3	β-Pinene	12	Geranial
4	Myrcene	13	Neryl acetate
5	*p*-Cymene	14	Geranyl acetate
6	Limonene	15	β-Caryophyllene
7	γ-Terpinene	16	α-Bergamotene
8	Terpinen-4-ol	17	α-Farnesene
9	α-Terpineol	18	β-Bisabolene

Operating conditions

Column: capillary; length 30 m; internal diameter 0,20 mm
Stationary phase: poly(5 % diphenyl/95 % dimethyl siloxane) (SP-5®)
Film thickness: 20 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 8,5 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Volume injected: 1 µl
Carrier gas flow rate: 206,84 kPa
Split ratio: 1/100

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



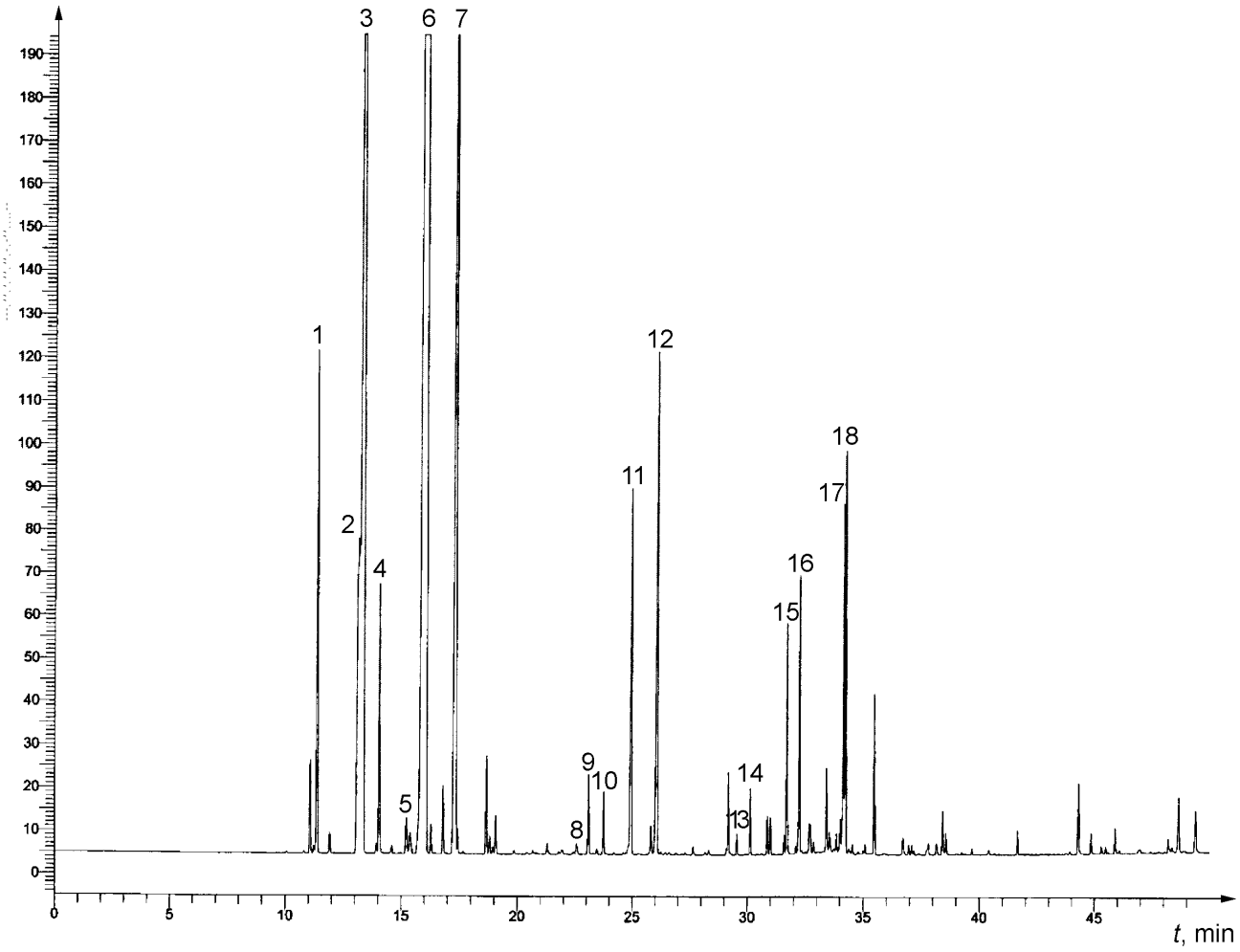
Peak identification

1	α-Pinene	8	α-Bergamotene
2	β-Pinene	9	β-Caryophyllene
3	Sabinene	10	Neral
4	Myrcene	11	α-Terpineol
5	Limonene	12	β-Bisabolene
6	γ-Terpinene	13	Neryl acetate
7	*p*-Cymene		

Operating conditions

Column: capillary; length 30 m; internal diameter 0,20 mm
Stationary phase: poly(ethylene glycol) (Carbowax®)
Film thickness: 20 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 8,5 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Volume injected: 1 µl
Carrier gas flow rate: 206,84 kPa
Split ratio: 1/100

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



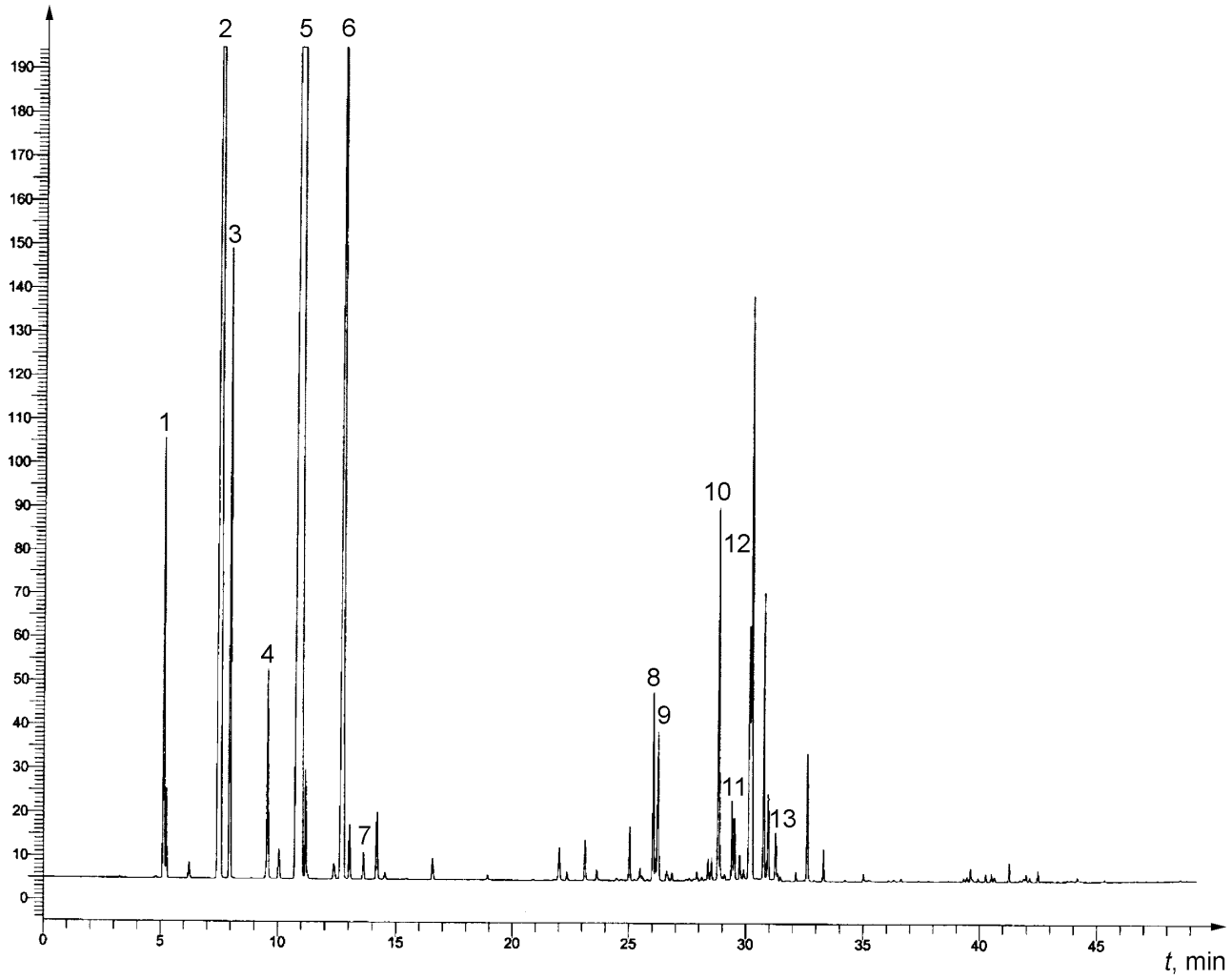
Peak identification

1	α-Pinene	10	*n*-Decanal
2	Sabinene	11	Neral
3	β-Pinene	12	Geranial
4	Myrcene	13	Neryl acetate
5	*p*-Cymene	14	Geranyl acetate
6	Limonene	15	β-Caryophyllene
7	γ-Terpinene	16	α-Bergamotene
8	Terpinen-4-ol	17	α-Farnesene
9	α-Terpineol	18	β-Bisabolene

Operating conditions

Column: capillary; length 30 m; internal diameter 0,20 mm
Stationary phase: poly(5 % diphenyl/95 % dimethyl siloxane) (SP-5®)
Film thickness: 20 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 8,5 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Volume injected: 1 µl
Carrier gas flow rate: 206,84 kPa
Split ratio: 1/100

Figure A.3 — Typical chromatogram taken on an apolar column (type B)



Peak identification

1 α-Pinene
2 β-Pinene
3 Sabinene
4 Myrcene
5 Limonene
6 γ-Terpinene
7 *p*-Cymene
8 α-Bergamotene
9 β-Caryophyllene
10 Neral
11 α-Terpineol
12 β-Bisabolene
13 Neryl acetate

Operating conditions

Column: capillary; length 30 m; internal diameter 0,20 mm
Stationary phase: poly(ethylene glycol) (Carbowax®)
Film thickness: 20 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 8,5 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Volume injected: 1 µl
Carrier gas flow rate: 206,84 kPa
Split ratio: 1/100

Figure A.4 — Typical chromatogram taken on a polar column (type B)

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 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 lime, Mexican type

The mean value is +46 °C.

NOTE Obtained with “Setaflash” equipment.

Bibliography

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

[2] ISO/TR 21092,—[1)], *Essential oils — Characterization*

1) To be published.

ICS 71.100.60

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