INTERNATIONAL STANDARD

ISO 3809

Third edition 2004-05-15

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

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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.

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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 3809:200

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 Citrus 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.

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4 Requirements

4.1 Appearance

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

4.2 Colour

Type A	Туре В
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, d²⁰ STANDARD PREVIEW

Ту	pe A (standa	ırds.iteh.ai)⊤yp	е В
min.	max.	min.	max.
0,875 0	0,884 0) 3809:20 <mark>0,</mark> 880 0	0,888 0
https://standards.html.aveatalog.standards/sis/803Ct230-ac10-402d-a/90-			

dab87531d987/iso-3809-2004

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

4.7 Carbonyl value

Туре А		Туре В		
min.	max.	min.	max.	
16	31	18	35	
(corresponding to	(corresponding to	(corresponding to 5 %	(corresponding to	
4,5 % of carbonyl	8,5 % of carbonyl	of carbonyl	9,5 % of carbonyl	
		compounds expressed		
as citral)	as citral)	as citral)	as citral)	

4.8 Residue on evaporation

Туре А		Туре В	
min.	max.	min.	max.
10,0 %	14,5 %	13,0 %	19,0 %

4.9 CD value

Type A	Туре В
min.	min.
iTel8,2STANDAR	PREVIEV23,6

4.10 Chromatographic profile standards.iteh.ai) 6 Test methods

Analysis of the essential oil shall be carried out by gas 2004 chromatography. In the chromatogram obtained, the skist 6.1 representative and characteristic components shown 3809-20 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.

6.1 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	
<i>p</i> -Cymene	_	0,5	
Limonene	42,0	C'50,0	DA
γ-Terpinene	8,0	11,0	
Terpinen-4-ol	0,2	(Stand	lar
lpha-Terpineol	0,2	0,6	TGC 2
n-Decanal	0,05	0,3	<u>ISO 3</u> g/stan
Neral	1,2	2,0 _{ab875}	31d98
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 furocoumarins. The bergapten content should be less than 2 000 mg/kg.

6.7 Chromatographic profile

See ISO 11024-1 and ISO 11024-2.

Packaging, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

Table 2 — Chromatographic profile (type B)

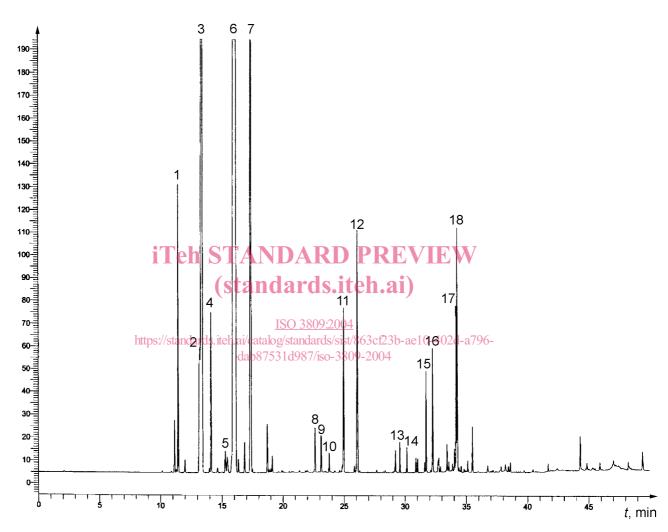
Component	Minimum	Maximum
	%	%
α-Pinene	1,7	2,0
Sabinene	2,0	3,0
β-Pinene	17,0	19,0
Myrcene	1,4	1,8
Neryl acetate	0,0	0,25
γ-Terpinene	9,0	9,7
Terpinen-4-ol	0,2	0,6
p-Cymene D T V T V	7 –	0,5
Limonene	38,0	44,0
α Terpineol • α I)	0,3	0,6
n-Decanal	0,15	0,35
109/2004 Neral ards/sist/863cf23b_ae10_402d_a/	796 <u>-</u> 2,0	2,5
Geranial 2004	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 furocoumarins. 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	<i>p</i> -Cymene	14	Geranyl acetate
6	Limonene	15	β-Caryophyllene
7	γ -Terpinene	16	α -Bergamotene
8	Terpinen-4-ol	17	α -Farnesene
a	a-Ternineol	12	R-Risaholene

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 $^{\circ}$ 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 $^{\circ}$ 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)