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Essential oil of petitgrain, Paraguayan type (*Citrus aurantium* L. var. Paraguay (syn. *Citrus aurantium* var. *bigaradia* Hook f.))

Huile essentielle de petitgrain, type Paraguay (Citrus aurantium L. var. Paraguay (syn. Citrus aurantium var. bigaradia Hook f.))

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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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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 54, *Essential oils*.

This third edition cancels and replaces the second edition (ISO 3064:2000), which has been technically revised.

Essential oil of petitgrain, Paraguayan type (*Citrus aurantium* L. var. Paraguay (syn. *Citrus aurantium* var. *bigaradia* Hook f.))

1 Scope

This International Standard specifies certain characteristics of the essential oil of petitgrain, Paraguayan type (*Citrus aurantium* L. var. Paraguay (syn. *Citrus aurantium* var. *bigaradia* Hook f.)), intended to facilitate the assessment of its quality.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 210, *Essential oils — General rules for packaging, conditioning and storage*

ISO/TS 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 1242, *Essential oils — Determination of acid value*

ISO 11024 (all parts), *Essential oils — General guidance on chromatographic profiles*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

essential oil of petitgrain

essential oil obtained by steam distillation of only leaves and twigs of *Citrus aurantium* L. var. Paraguay (syn. *Citrus aurantium* var. *bigaradia* Hook f.), mainly cultivated in Paraguay, of the *Rutaceae* family

Note 1 to entry: For information on the CAS number, see ISO/TR 21092.^[2]

4 Specifications

4.1 Appearance

Clear liquid.

4.2 Colour

Colourless to pale yellow.

4.3 Odour

Fresh, green, and flowery.

4.4 Relative density, d_{20}^{20}

Minimum: 0,884.

Maximum: 0,892.

4.5 Refractive index at 20 °C

Minimum: 1,454.

Maximum: 1,463.

4.6 Optical rotation at 20 °C

Between -5° and -1°.

4.7 Miscibility in ethanol 70 % (volume fraction) at 20 °C

It shall not be necessary to use more than 3,5 volumes of ethanol 70 % (volume fraction) to obtain a clear solution with one volume of essential oil.

4.8 Acid value

Maximum: 1.

4.9 Chromatographic profile

Carry out the analysis of the essential oil by gas chromatography. Identify in the chromatogram obtained, the representative and characteristic components shown in [Table 1](#). 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
	%	%
β-Pinene	0,5	2,0
Myrcene	1,3	3,0
Sabinene	0,1	0,5
Limonene	0,7	3,5
(Z)-β-Ocimene	0,7	1,5
(E)-β-Ocimene	0,5	3,5
Linalool	15,0	30,0
Linalyl acetate	40,0	60,0

NOTE The chromatographic profile is normative, contrary to typical chromatograms given for information in [Annex A](#).

Table 1 (continued)

Component	Minimum %	Maximum %
β-Caryophyllene	0,3	1,5
α-Terpineol	3,0	7,0
Nerol	0,5	2,0
Geraniol	2,0	4,5
Neryl acetate	1,0	3,0
Geranyl acetate	2,0	5,0

NOTE The chromatographic profile is normative, contrary to typical chromatograms given for information in [Annex A](#).

4.10 Flashpoint

Information concerning the flashpoint is given in [Annex B](#).

5 Sampling

Sampling shall be performed in accordance with ISO 212.

Minimum volume of the test sample: 25 ml.

NOTE This volume makes it possible for each of the tests specified in this International Standard to be carried out at least once.

6 Test methods

6.1 Relative density, d_{20}^{20}

Determine the relative density in accordance with ISO 279.

6.2 Refractive index at 20 °C

Determine the refractive index in accordance with ISO 280.

6.3 Optical rotation at 20 °C

Determine the optical rotation in accordance with ISO 592.

6.4 Miscibility in ethanol 70 % (volume fraction) at 20 °C

Determine the miscibility in accordance with ISO 875.

6.5 Acid value

Determine the acid value in accordance with ISO 1242.

6.6 Chromatographic profile

Determine the chromatographic profile in accordance with ISO 11024.

7 Packaging, labelling, marking, and storage

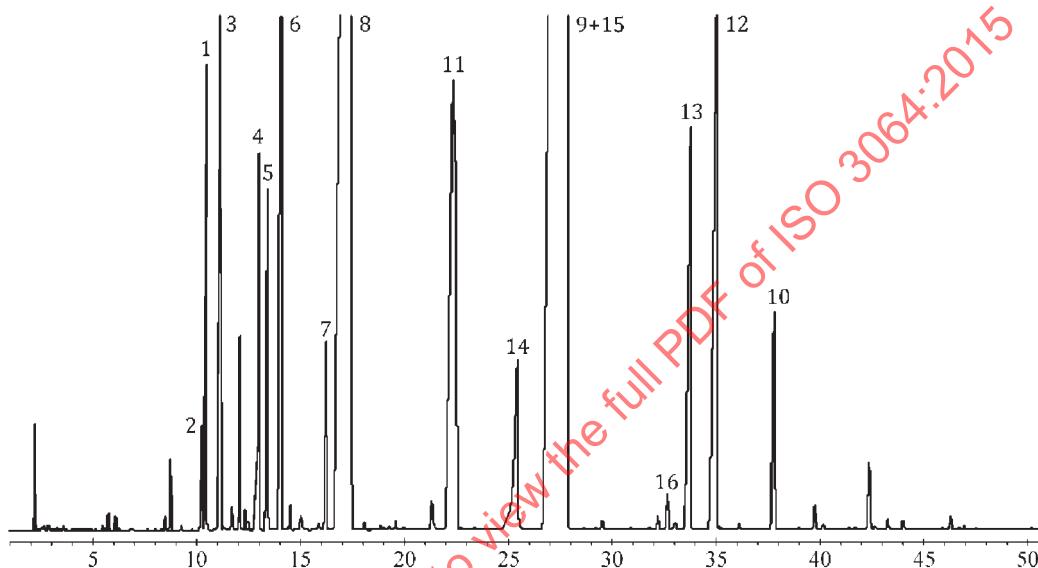
These items shall be in accordance with ISO/TS 210 and ISO/TS 211.

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Annex A

(informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of petitgrain, Paraguay type (*Citrus aurantium* L. var. Paraguay (syn. *Citrus aurantium* L. var. *bigaradia* Hook f.))



Peak identification

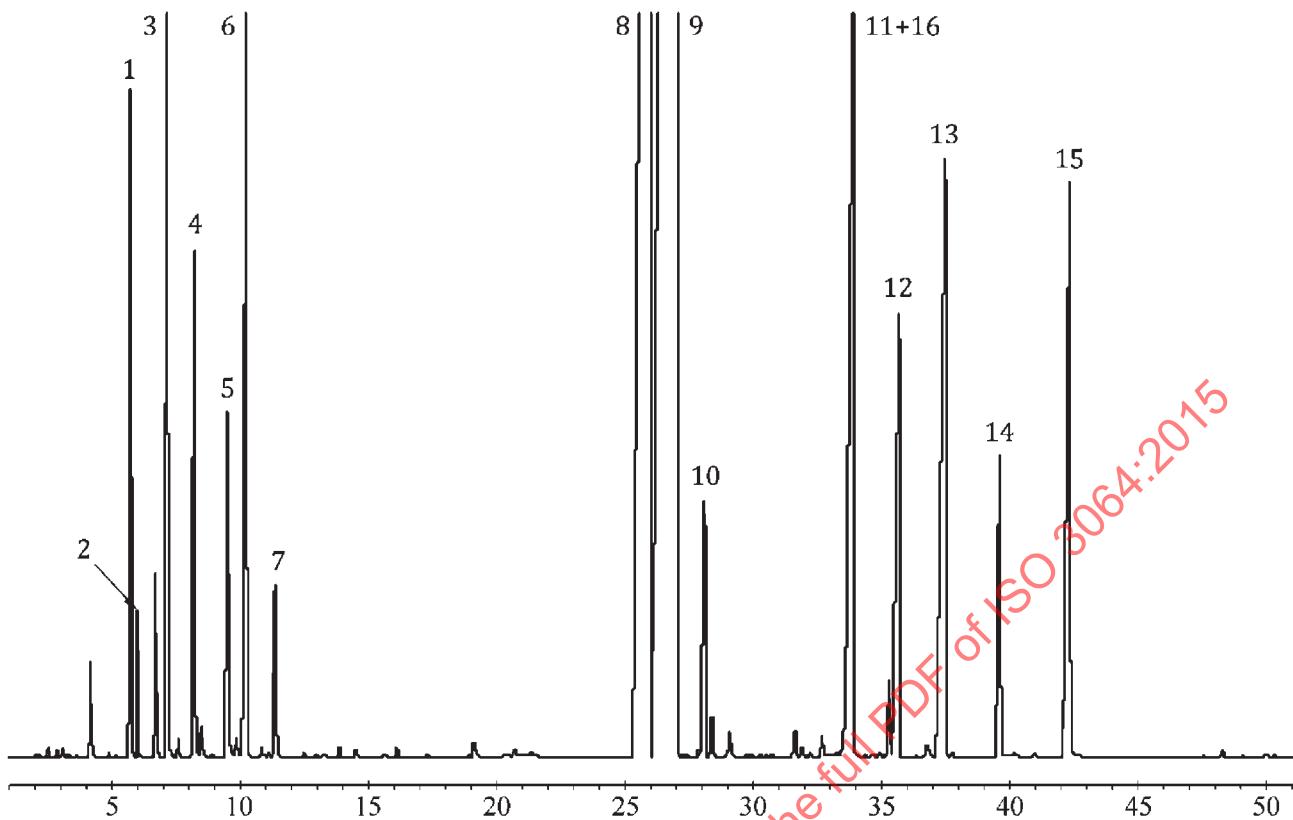
- 1 β -Pinene
- 2 Sabinene
- 3 Myrcene
- 4 Limonene
- 5 (Z)- β -Ocimene
- 6 (E)- β -Ocimene
- 7 Terpinolene
- 8 Linalool
- 9 Linalyl acetate
- 10 β -Caryophyllene
- 11 α -Terpineol
- 12 Geranyl acetate
- 13 Neryl acetate
- 14 Nerol
- 15 Geraniol
- 16 Terpenyl acetate

Operating conditions:

- Column: dimethylpolysiloxane (HP-1 or BP-1^a), length 30 m, internal diameter 0,25 mm
- Film thickness: 0,25 μ m
- Oven temperature: temperature programmed 1 min at 60 °C, then from 60 °C to 240 °C at a rate of 2 °C/min
- Injector temperature: 250 °C
- Detector temperature: 300 °C
- Detector: flame ionization type
- Carrier gas: nitrogen
- Volume injected: 0,5 μ l
- Carrier gas flow rate: 1 ml/min
- Split: 1/100

^a HP-1 and BP-1 are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Figure A.1 — Typical chromatogram of essential oil of petitgrain taken on an apolar column

**Peak identification**

- 1 β -Pinene
- 2 Sabinene
- 3 Myrcene
- 4 Limonene
- 5 (Z)- β -Ocimene
- 6 (E)- β -Ocimene
- 7 Terpinolene
- 8 Linalool
- 9 Linalyl acetate
- 10 β -Caryophyllene
- 11 α -Terpineol
- 12 Geranyl acetate
- 13 Neryl acetate
- 14 Nerol
- 15 Geraniol
- 16 Terpenyl acetate

Operating conditions:

Column: polyethylene glycol (HP Innowax or BP 20^a), length 30 m, internal diameter 0,25 mm
 Film thickness: 0,25 μ m
 Oven temperature: temperature programmed 1 min at 60 °C, then from 60 °C to 240 °C at a rate of 2 °C/min
 Injector temperature: 250 °C
 Detector temperature: 300 °C
 Detector: flame ionization type
 Carrier gas: nitrogen
 Volume injected: 0,5 μ l
 Carrier gas flow rate: 1 ml/min
 Split: 1/100

^a HP Innowax or BP 20 are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Figure A.2 — Typical chromatogram of essential oil of petitgrain 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 flash points of essential oils, which in most cases are flammable products.

A comparative study on the relevant methods of analysis (see ISO/TR 11018^[1]) concluded that it was difficult to recommend a single apparatus for standardization purposes, given the following:

- 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 annexed to each International Standard for information, in order to meet the requirements of the interested parties.

The equipment with which this value was obtained has to be specified.

For further information, see ISO/TR 11018^[1]

B.2 Flashpoint of the essential oil of petitgrain, Paraguayan type

The mean value is +75 °C.

NOTE Obtained with Luchaire¹⁾ equipment.

1) Luchaire is an example of a suitable equipment available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.