INTERNATIONAL STANDARD

ISO 8901

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Oil of bitter orange petitgrain, cultivated (Citrus aurantium L.)

Huile essentielle de petitgrain bigaradier cultivé (Citrus aurantium L.)



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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 8901 was prepared by Technical Committee ISO/TC 54, Essential oils.

This second edition cancels and replaces the first edition (ISO 8901:1987), which has been technically revised.

Oil of bitter orange petitgrain, cultivated (Citrus aurantium L.)

1 Scope

This International Standard specifies certain characteristics of the oil of cultivated bitter orange petitgrain (*Citrus aurantium* L.), in order to facilitate assessment of its quality.

NOTE This International Standard is not applicable to the oil of petitgrain, Paraguayan type, described in ISO 3064.

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 inde

ISO 592, Essential oils — Determination of optical rotation

ISO 709, Essential oils — Determination of ester value

ISO 875, Essential oils — Evaluation of miscibility in ethanol

ISO 1242, Essential oils — Determination of acid value

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 petitgrain

essential oil obtained by steam distillation of the leaves, twigs and small green fruits of *Citrus aurantium* L., of the Rutaceae family, mainly produced in the Mediterranean areas

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

4 Requirements

4.1 Appearance

Clear liquid.

4.2 Colour

Pale yellow to amber yellow, with a slight blue fluorescence.

4.3 Odour

Characteristic (orange like, bitter), ethereal and pleasant.

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

Minimum: 0,885

Maximum: 0,898

Refractive index at 20 °C

1,4550 Minimum:

Maximum: 1.472 0

4.6 Optical rotation at 20 °C

Between -6° and $+2^{\circ}$.

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

It shall not be necessary to use more than 5 volumes of ethanol, 70 % (volume fraction), to obtain a clear solution with 1 volume of essential oil.

Acid value 4.8

Maximum: 2

4.9 Ester value

Minimum: 140

Maximum: 217

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 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.

4.11 Flashpoint

Information on the flashpoint is given in Annex B.

Table 1 — Chromatographic profile

Component	Minimum %	Maximum %
Limonene	1	6
<i>trans</i> -β-Ocimene	1	4
Linalol	10	32
Linalyl acetate	40	72
α-Terpineol	1	7
Geraniol	1	4
Geranyl acetate	1,5	5,5

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

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.

Test methods

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 Acid value

See ISO 1242.

6.6 Ester value

See ISO 709.

Saponification time: 1 h.

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.

Annex A

(informative)

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

Peak identification

- 1 α -Pinene
- 2 Sabinene
- β-Pinene
- 4 Myrcene
- 5 Limonene + *cis*-β-ocimene
- 6 *trans*-β-Ocimene
- 7 Terpinolene
- 8 Linalol
- 9 Terpinen-4-ol
- 10 α -Terpineol
- 11 Nerol
- 12 Linalyl acetate + geraniol
- 13 Neryl acetate
- 14 Geranyl acetate
- 15 β-Caryophyllene
- 16 Humulene
- 17 Bicyclogermacrene
- 18 Nerolidol

Operating conditions

Column: fused silica capillary; length 60 m; internal diameter 0,25 mm

Stationary phase: poly(5 % diphenyl – 95 % dimethylsiloxane)

Film thickness: 0,25 µm

Oven temperature: isothermal at 80 °C for 10 min then temperature

programming from 80 °C to 240 °C at a rate of 3 °C/min

Injector temperature: 250 °C Detector temperature: 270 °C

Detector: flame ionization type

Carrier gas: nitrogen Injection volume: 0,2 µl Carrier gas flow rate: 1 ml/min

Split ratio: 1/100

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

Peak identification

α-Pinene
β-Pinene
Sabinene
Myrcene
Limonene
trans-β-Ocimene

-

7 Linalol

8 Linalyl acetate

9 β-Caryophyllene10 α-Terpineol

11 Neryl acetate

12 Geranyl acetate

13 Nerol

14 Geraniol

Operating conditions

Column: fused silica capillary; length 60 m; internal diameter 0,25 mm Stationary phase: poly(ethylene glycol)-modified TPA (SP-1 000®)

Film thickness: 0,25 µm

Oven temperature: temperature programming from 95 °C to 190 °C at a rate of

4 °C/min.

Injector temperature: 250 °C Detector temperature: 250 °C Detector: flame ionization type

Carrier gas: nitrogen Injection volume: 0,1 µl

Carrier gas flow rate: 1 ml/min

Split ratio: 1/100

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

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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 highpriced 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 in each International Standard, for information, 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 petitgrain, cultivated

The mean value is + 75°.

NOTE Obtained with "Pensky-Martens" equipment.

Bibliography

- [1] ISO 3064, Oil of petitgrain, Paraguayan type (Citrus aurantium L. ssp. aurantium, syn. Citrus aurantium L. ssp. amara var. pumilia)
- [2] ISO/TR 11018:1997, Essential oils General guidance on the determination of flashpoint
- [3] ISO/TR 21092¹⁾, Essential oils Characterization

¹⁾ To be published.

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