INTERNATIONAL STANDARD

ISO 3064

Second edition
2000-08-01

Oil of petitgrain, Paraguayan type (*Citrus aurantium* L. ssp. *aurantium*, syn. *Citrus aurantium* L. ssp. *amara* var. *pumilia*)

*Huile essentielle de petitgrain, type Paraguay (*Citrus aurantium *L. ssp.* aurantium*, syn.* Citrus aurantium *L. ssp.* amara *var.* pumilia*)*

Reference number
ISO 3064:2000(E)

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Printed in Switzerland

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3064 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 3064:1977), which has been technically revised.

Annexes A and B of this International Standard are for information only.

Oil of petitgrain, Paraguayan type (*Citrus aurantium* L. ssp. *aurantium*, syn. *Citrus aurantium* L. ssp. *amara* var. *pumilia*)

1 Scope

This International Standard specifies certain characteristics of the oil of petitgrain, Paraguayan type (*Citrus aurantium* L. ssp. *aurantium*, syn. *Citrus aurantium* L. ssp. *amara* var. *pumilia*), in order to facilitate assessment of its quality.

NOTE The specifications of the oil of bitter orange petitgrain are given in ISO 8901.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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 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 1271, *Essential oils — Determination of carbonyl value — Free hydroxylamine method*.

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 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
oil of petitgrain, Paraguayan type
essential oil obtained by steam distillation of only the leaves and twigs of *Citrus aurantium* L. ssp. *aurantium*, syn. *Citrus aurantium* L. ssp. *amara* var. *pumilia*, mainly cultivated in Paraguay, of the Rutaceae family.

4 Requirements

4.1 Appearance

Clear liquid.

4.2 Colour

Colourless to pale yellow.

4.3 Odour

Characteristic, fresh, rustic, reminiscent of that of the flower of the orange tree.

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

Minimum: 0,882 0

Maximum: 0,893 0

4.5 Refractive index at 20 °C

Minimum: 1,455 0

Maximum: 1,463 0

4.6 Optical rotation at 20 °C

Between – 5 °C and – 1 °C.

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

It shall not be necessary to use more than 4 volumes of 70 % ethanol (volume fraction) to obtain a clear solution with 1 volume of essential oil.

4.8 Acid value

Maximum: 1

4.9 Ester value

Minimum: 122, corresponding to 39 % of ester expressed as linalyl acetate.

Maximum: 190, corresponding to 67 % of ester expressed as linalyl acetate.

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.

Table 1 — Chromatographic profile

Component	Minimum %	Maximum %
β-Pinene	0,5	2
Myrcene	1,3	2,7
Sabinene	0,1	0,5
trans-β-Ocimene	1	3
Linalol	15	30
Linalyl acetate	40	60
α-Terpineol	3,2	6,8
Nerol	0,5	2
Geraniol	2	4,5
Neryl acetate	1	3
Geranyl acetate	2	5
β-Caryophyllene	0,3	1,5

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

4.11 Flashpoint

Information on the flashpoint is given in annex B.

5 Sampling

See ISO 212.

Minimum volume of test sample: 30 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 70 % ethanol (volume fraction) at 20 °C

See ISO 875.

6.5 Acid value

See ISO 1242.

6.6 Ester value

See ISO 709.

Test portion: 2 g.
Saponification time: 30 min.

Relative molecular mass of linalyl acetate: 196,29.

6.7 Chromatographic profile

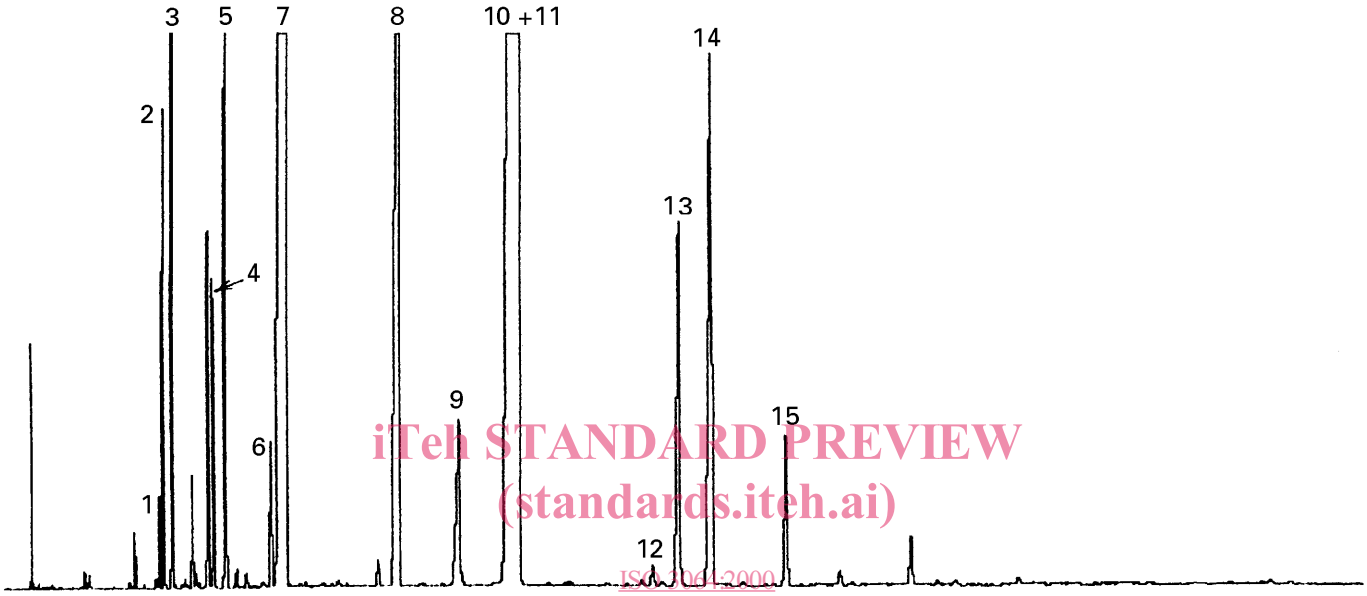
See ISO 11024-1 and ISO 11024-2.

7 Packaging, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

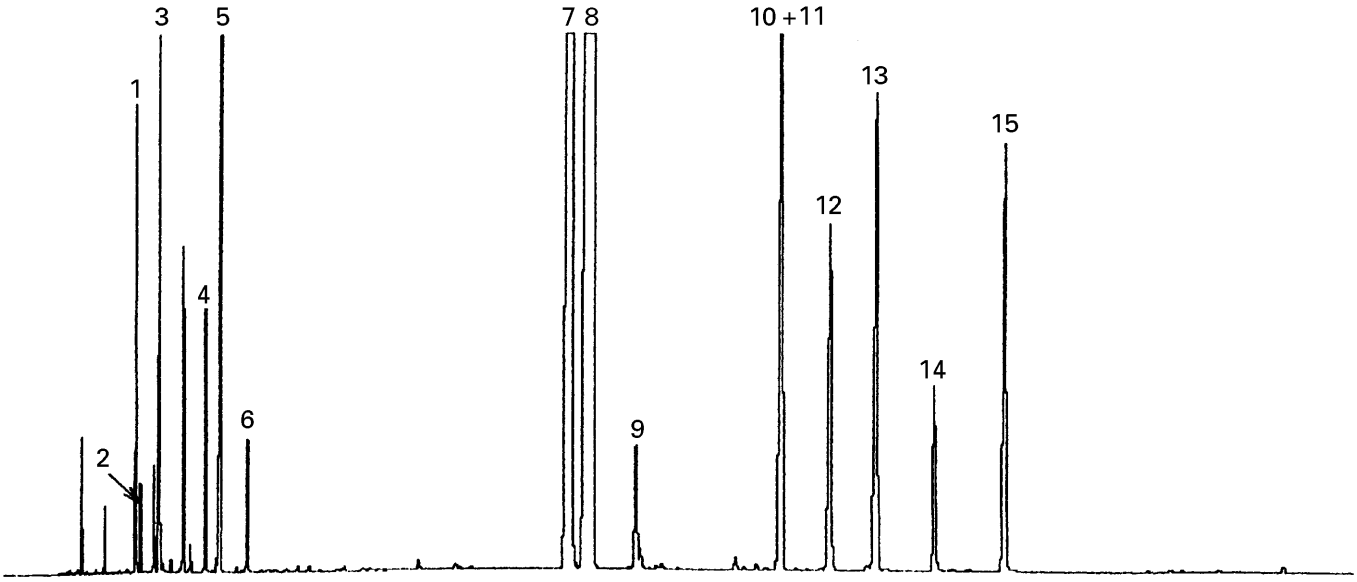
Annex A
(informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of petitgrain, Paraguayan type (*Citrus aurantium* L. spp. *aurantium*, syn. *Citrus aurantium* L. ssp. *amara* var. *pumilia*)



Peak identification		Operating conditions
1	Sabinene	Column: silica capillary; length 30 m; internal diameter 0,25 mm
2	β-Pinene	Stationary phase: polydimethyl siloxane (SE 30)
3	Myrcene	Film thickness: 0,25 µm
4	*cis*-β-Ocimene	Oven temperature: temperature programmed from 70 °C to 250 °C at a rate of 2 °C/min
5	*trans*-β-Ocimene	Injector temperature: 250 °C
6	Terpinolene	Detector temperature: 250 °C
7	Linalol	Detector: flame ionization type
8	α-Terpineol	Carrier gas: nitrogen
9	Nerol	Volume injected: 0,3 µl
10 + 11	Linalyl acetate + geraniol	Carrier gas flow rate: 1 ml/min
12	Terpenyl acetate	Split ratio:1/100
13	Neryl acetate	
14	Geranyl acetate	
15	β-Caryophyllene	

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



Peak identification		Operating conditions
1	β-Pinene	Column: silica capillary; length 30 m; internal diameter 0,25 mm
2	Sabinene	Stationary phase: polyethylene glycol (Supelcowax 10)
3	Myrcene	Film thickness: 0,25 µm
4	*cis*-β-Ocimene	Oven temperature: temperature programmed from 70 °C to 250 °C at a rate of 2 °C/min
5	*trans*-β-Ocimene	Injector temperature: 250 °C
6	Terpinolene	Detector temperature: 250 °C
7	Linalol	Detector: flame ionization type
8	Linalyl acetate	Carrier gas: nitrogen
9	β-Caryophyllene	Volume injected: 0,3 µl
10 + 11	α-Terpineol + terpenyl acetate	Carrier gas flow rate: 1 ml/min
12	Neryl acetate	Split ratio:1/100
13	Geranyl acetate	
14	Nerol	
15	Geraniol	

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