INTERNATIONAL STANDARD

ISO 3528

Second edition
1997-07-15

Oil of mandarin, Italian type (*Citrus reticulata* Blanco)

Huile essentielle de mandarine, type Italie (Citrus reticulata *Blanco*)

Reference number
ISO 3528:1997(E)

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.

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.

International Standard ISO 3528 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 3528:1977), which has been technically revised.

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

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Oil of mandarin, Italian type (*Citrus reticulata* Blanco)

1 Scope

This International Standard specifies certain characteristics of the oil of mandarin, Italian type (*Citrus reticulata* Blanco), in order to facilitate assessment of its quality.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 210:— [1], *Essential oils — General rules for packaging, conditioning and storage.*

ISO 211:— [2], *Essential oils — General rules for labelling and marking of containers.*

ISO 212:1973, *Essential oils — Sampling.*

ISO 279:1981, *Essential oils — Determination of relative density at 20 °C (Reference method).*

ISO 280:1976, *Essential oils — Determination of refractive index.*

ISO 592:1981, *Essential oils — Determination of optical rotation.*

ISO 875:1981, *Essential oils — Evaluation of miscibility in ethanol.*

ISO 1242:1973, *Essential oils — Determination of the acid value.*

ISO 1279:1996, *Essential oils — Determination of carbonyl value — Potentiometric methods using hydroxylammonium chloride.*

ISO 4715:1978, *Essential oils — Quantitative evaluation of residue on evaporation.*

ISO 11024-1:— [3], *Essential oils — General guidance on chromatographic profiles — Part 1: Preparation of chromatographic profiles for presentation in standards.*

ISO 11024-2:— [3], *Essential oils — General guidance on chromatographic profiles — Part 2: Utilization of chromatographic profiles of a sample of essential oils.*

3 Definition

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

1) To be published. (Revision of ISO 210:1961)

2) To be published. (Revision of ISO 211:1961)

3) To be published.

3.1 oil of mandarin, Italian type: Essential oil, obtained without heating by mechanical means, from the fresh epicarp and part of the mesocarp of the fruit of *Citrus reticulata* Blanco, of the Rutaceae family, mainly produced in Italy.

4 Requirements

4.1 Appearance

Mobile liquid.

4.2 Colour

Greenish yellow to reddish orange, with a blue fluorescence light.

4.3 Odour

Characteristic from the fresh pericarp of the fruit.

4.4 Relative density at 20 °C/20 °C

Minimum: 0,848
Maximum: 0,855

4.5 Refractive index at 20 °C

Minimum: 1,474 0
Maximum: 1,478 0

4.6 Optical rotation at 20 °C

Minimum: +64°
Maximum: +75°

4.7 Residue on evaporation

Minimum: 1,6 % (*m/m*)
Maximum: 4,0 % (*m/m*)

4.8 Miscibility with 90 % (*V/V*) ethanol at 20 °C

1 volume of the oil shall not require more than 7 to 10 volumes of 90 % (*V/V*) ethanol, at 20 °C, to give a cloudy solution.

4.9 Carbonyl value

Minimum: 1,4
Maximum: 4,3

Corresponding to a carbonyl compound content of 0,4 % (*m/m*) and 1,2 % (*m/m*), expressed as decanal.

4.10 Acid value

Maximum: 2,0

4.11 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 %
α-Pinene	2	3
β-Pinene	1,2	2
Myrcene	1,5	2
γ-Terpinene	16	22
Linonene	65	75
Methyl *N*-methylanthranilate	0,30	0,60
α-Sinensal	0,20	0,50

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

4.12 Flashpoint

Information on the flashpoint is given in annex B.

5 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.

6 Test methods

6.1 Relative density at 20 °C/20 °C

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 Residue on evaporation

See ISO 4715

Test portion: 5 g.
Evaporation time: 6 h.

6.5 Miscibility with 90 % (V/V) ethanol at 20 °C

See ISO 875.

6.6 Acid value

See ISO 1242.

6.7 Carbonyl value

See ISO 1279, method I.

Test portion: 10 g.
Determination time: 15 min.
M_r of decanal: 156.

6.8 Chromatographic profile

See ISO 11024-1 and ISO 11024-2.

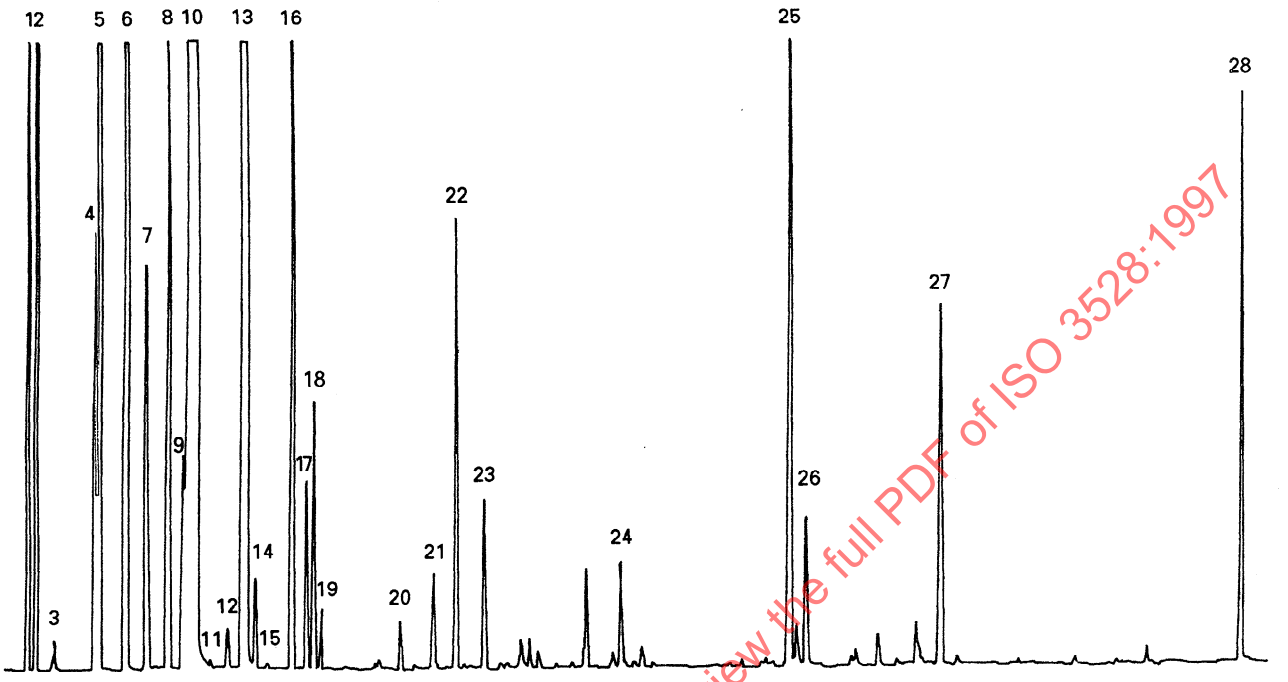
7 Packaging, labelling, marking and storage

See ISO 210 and ISO 211.

NOTE — This essential oil is particularly sensitive to oxidation.

Annex A
(informative)

Typical chromatogram of the essential oil of mandarin, Italian type



Peak identification

1 α-Thujene
2 α-Pinene
3 Camphene
4 Sabinene
5 β-Pinene
6 Myrcene
7 Octanal + α-phellandrene
8 α-Terpinene
9 *p*-Cymene
10 Limonene
11 *cis*-β-Ocimene
12 *trans*-β-Ocimene
13 γ-Terpinene
14 *trans*-Sabinene hydrate
15 Octanol
16 Terpinolene
17 *cis*-Sabinene hydrate
18 Linalol
19 Nonanal
20 Citronellal
21 Terpinen-4-ol
22 α-Terpineol
23 Decanal
24 Thymol
25 Methyl *N*-methylanthranilate
26 Caryophyllene
27 *trans-trans*-α-Farnesene
28 α-Sinensal

Operating conditions

Column: capillary; length 25 m; diameter 0,32 mm
Stationary phase: SE 52 phenyl methylsilicone
Film thickness: 0,4 µm to 0,45 µm
Oven temperature: 60 °C for 8 min, then from 60 °C to 200 °C at a rate of 3 °C/min
Injector temperature: 280 °C
Detector temperature: 280 °C
Detector: flame ionization
Carrier gas: hydrogen (0,5 kg/cm^2)
Volume injected: 0,1 µl
Split ratio: 1/100