INTERNATIONAL STANDARD

ISO 3528

Third edition
2012-07-01

Essential oil of mandarin, Italian type (*Citrus reticulata* Blanco)

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

Reference number
ISO 3528:2012(E)

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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.

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

This third edition cancels and replaces the second edition (ISO 3528:1997), which has been technically revised. It also incorporates the Technical Corrigendum ISO 3528:1997/Cor.1:1998.

Essential 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 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 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
oil of mandarin, Italian type
essential oil obtained by expression, without the aid of heat and with or without previous separation of the pulp and the peel, from the fresh fruit of *Citrus reticulata* Blanco of the *Rutaceae* family

NOTE For information on CAS number, see ISO/TR 21092[2].

4 Requirements

4.1 Appearance

Clear mobile liquid.

4.2 Colour

The colour depends on the season of harvest.

Green	Yellow	Red
Light green to dark green, with a blue fluorescence	Light yellow to dark orange, with a blue fluorescence	Reddish to dark red, with a blue fluorescence

4.3 Odour

The odour from the fresh pericarp of the fruit is characteristic.

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

	Green	Yellow	Red
Minimum	0,847	0,846	0,844
Maximum	0,855	0,854	0,853

4.5 Refractive index at 20 °C

	Green	Yellow	Red
Minimum	1,473 2	1,472 6	1,472 2
Maximum	1,475 8	1,475 3	1,474 6

4.6 Optical rotation at 20 °C

	Green	Yellow	Red
Minimum	+69°	+69°	+70°
Maximum	+75°	+76°	+79°

4.7 Residue on evaporation

	Green	Yellow	Red
Minimum	1,90 % mass fraction	1,80 % mass fraction	1,40 % mass fraction
Maximum	3,90 % mass fraction	3,90 % mass fraction	3,30 % mass fraction

4.8 Miscibility with 90 % (volume fraction) ethanol at 20 °C

It shall not be necessary to use more than 10 volumes of 90 % (volume fraction) ethanol at 20 °C to obtain an opalescent solution with 1 volume of essential oil.

4.9 Acid value

	Green	Yellow	Red
Maximum	2,0	2,0	2,0

4.10 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	Green		Yellow		Red	
	Minimum %	Maximum %	Minimum %	Maximum %	Minimum %	Maximum %
α-Pinene	1,6	2,7	1,6	2,7	1,8	3
β-Pinene	1	2	1	2	1,2	2
Myrcene	1,4	2	1,4	2	1,5	2
n-Octanal	0,08	0,14	0,05	0,14	0,03	0,08
p-Cymene	n.d.[a]	0,4	n.d.[a]	0,5	n.d.[a]	0,8
γ-Terpinene	17	22	16	22	16	22
Limonene	65	74	65	75	65	75
Linalool	0,05	0,2	0,05	0,2	0,03	0,2
n-Decanal	0,04	0,14	0,04	0,14	0,04	0,12
Methyl *N*-methylanthranilate	0,4	0,7	0,3	0,6	0,3	0,6
α-Sinensal	0,2	0,5	0,2	0,5	0,1	0,5

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

[a] Not detectable.

4.11 Flashpoint

Information on the flashpoint is given in Annex B.

5 Sampling

Sampling shall be performed in accordance with 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, 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 Residue on evaporation

Determine the residue on evaporation in accordance with ISO 1242.

Test portion: 5 g

Evaporation time: 6 h

6.5 Miscibility with 90 % (volume fraction) ethanol at 20 °C

Determine the miscibility in accordance with ISO 875.

6.6 Acid value

Determine the acid value in accordance with ISO 1242.

6.7 Chromatographic profile

Determine the chromatographic profile in accordance with ISO 11024.

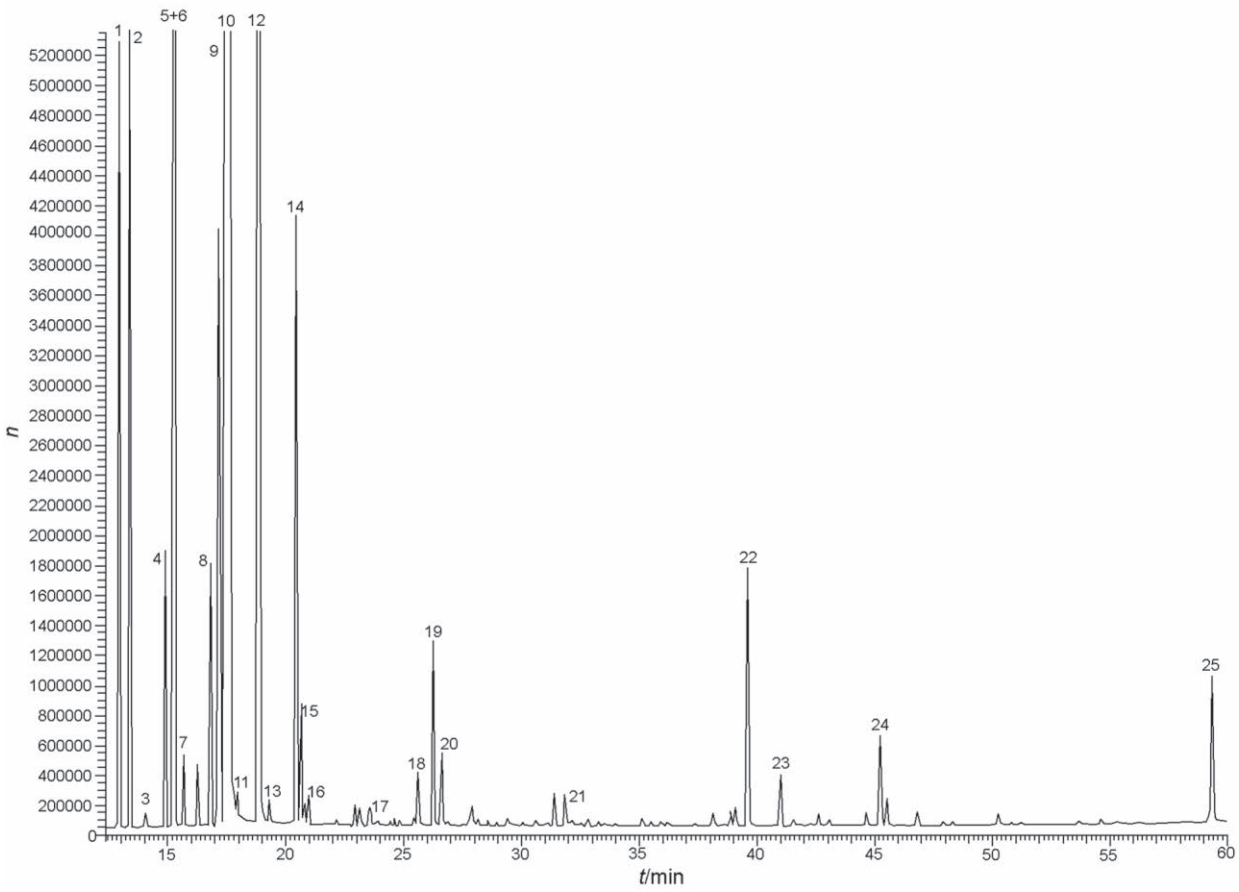
7 Packaging, labelling, marking and storage

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

NOTE This essential oil is particularly susceptible to oxidization.

Annex A
(informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of mandarin, Italian type (*Citrus reticulata* Blanco)



Peak identification

1 α-Thujene
2 α-Pinene
3 Camphene
4 Sabinene
5 β-Pinene
6 Myrcene
7 *n*-Octanal
8 α-Terpinene
9 *p*-Cymene
10 Limonene
11 β-Phellandrene
12 γ-Terpinene
13 (*Z*)-Sabinene hydrate
14 Terpinolene
15 Linalool
16 *n*-Nonanal
17 Citronellal
18 Terpinen-4-ol
19 α-Terpineol
20 *n*-Decanal
21 Thymol
22 Methyl *N*-methylanthranilate
23 β-Caryophyllene
24 (*E*,*E*)-α-Farnesene
25 α-Sinensal

n counts
t time

Operating conditions

Column: capillary, fused silica, 50 m long and 0,25 mm internal diameter

Film thickness: 1,00 µm

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

Oven temperature:

isothermal at 46 °C for 1 min and temperature programming from 46 °C to 100 °C at a rate of 5 °C/min

then from 100 °C to 230 °C at a rate of 2 °C/min, isothermal at 230 °C for 13 min

Injector temperature: 230 °C

Detector temperature: 250 °C

Detector 1: flame ionization type

Detector 2: quadrupole mass spectrometer 40 amu to 300 amu

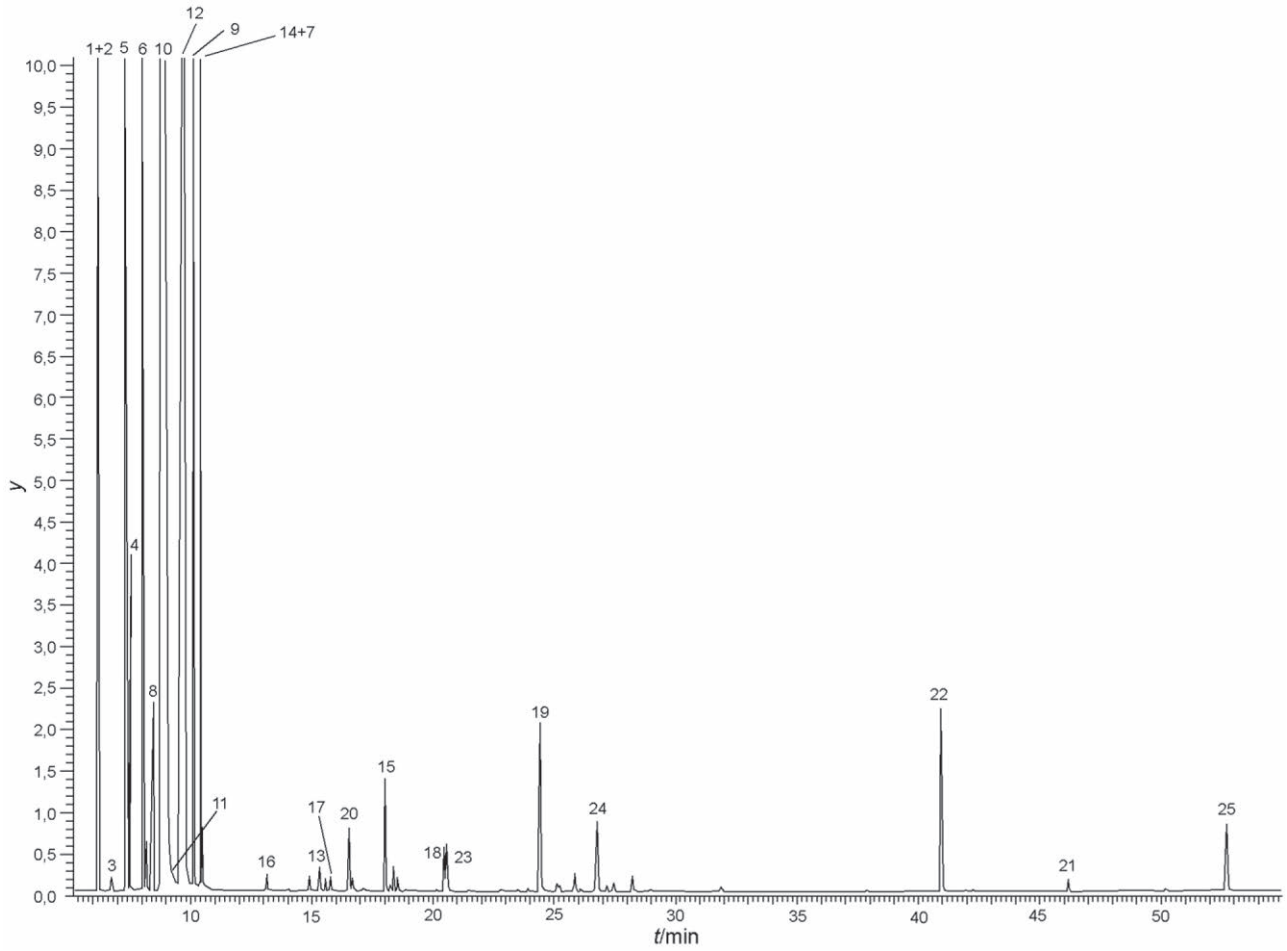
Carrier gas: helium 99,999 % volume fraction

Injection volume: 0,1 µl

Carrier gas flow rate: 1,5 ml/min constant flow

Split ratio: 1:50

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



Peak identification

1 α-Thujene
2 α-Pinene
3 Camphene
4 Sabinene
5 β-Pinene
6 Myrcene
7 *n*-Octanal
8 α-Terpinene
9 *p*-Cymene
10 Limonene
11 β-Phellandrene
12 γ-Terpinene
13 (*Z*)-Sabinene hydrate
14 Terpinolene
15 Linalool
16 *n*-Nonanal
17 Citronellal
18 Terpinen-4-ol
19 α-Terpineol
20 *n*-Decanal
21 Thymol
22 Methyl *N*-methylanthranilate
23 β-Caryophyllene
24 (*E*,*E*)-α-Farnesene
25 α-Sinensal

y relative abundance
t time

Operating conditions

Column: capillary, fused silica, 60 m long and 0,25 mm internal diameter

Film thickness: 0,25 µm

Stationary phase: polyethyleneglycol [CW 20M[1)]]

Oven temperature:

isothermal at 46 °C for 1 min and temperature programming from 46 °C to 100 °C at a rate of 5 °C/min

then from 100 °C to 230 °C at a rate of 2 °C/min, isothermal at 230 °C for 13 min

Injector temperature: 230 °C

Detector temperature: 250 °C

Detector 1: flame ionization type

Detector 2: quadrupole mass spectrometer 40 amu to 300 amu

Carrier gas: helium 99,999 % volume fraction

Injection volume: 0,1 µl

Carrier gas flow rate: 2,2 ml/min constant flow

Split ratio: 1:50

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

1) CW 20M is an example of a suitable product 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.

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[1]) concluded that it was difficult to recommend a single apparatus for standardization purposes, given that:

— 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 mandarin, Italian type

The mean value is +48 °C.

NOTE Obtained with Pensky-Martens[2)] equipment.

2) 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.

Bibliography

[1] ISO/TR 11018, *Essential oils — General guidance on the determination of flashpoint*

[2] ISO/TR 21092, *Essential oils — Characterization*

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