

BS ISO 3528:2012



BSI Standards Publication

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

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of ISO 3528:2012.

The UK participation in its preparation was entrusted to Technical Committee AW/54, Essential oils.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2012

Published by BSI Standards Limited 2012

ISBN 978 0 580 68035 9

ICS 71.100.60

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2012.

Amendments issued since publication

Amd. No.	Date	Text affected
-----------------	-------------	----------------------

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)



COPYRIGHT PROTECTED DOCUMENT

© ISO 2012

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Requirements	1
4.1 Appearance	1
4.2 Colour	2
4.3 Odour	2
4.4 Relative density at 20 °C, d_{20}^{20}	2
4.5 Refractive index at 20 °C	2
4.6 Optical rotation at 20 °C	2
4.7 Residue on evaporation	2
4.8 Miscibility with 90 % (volume fraction) ethanol at 20 °C	2
4.9 Acid value	3
4.10 Chromatographic profile	3
4.11 Flashpoint	3
5 Sampling	3
6 Test methods	3
6.1 Relative density at 20 °C, d_{20}^{20}	3
6.2 Refractive index at 20 °C	4
6.3 Optical rotation at 20 °C	4
6.4 Residue on evaporation	4
6.5 Miscibility with 90 % (volume fraction) ethanol at 20 °C	4
6.6 Acid value	4
6.7 Chromatographic profile	4
7 Packaging, labelling, marking and storage	4
Annex A (informative) Typical chromatograms of the analysis by gas chromatography of the essential oil of mandarin, Italian type (<i>Citrus reticulata</i> Blanco)	5
Annex B (informative) Flashpoint	8
Bibliography	9

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
<i>n</i> -Octanal	0,08	0,14	0,05	0,14	0,03	0,08
<i>p</i> -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
<i>n</i> -Decanal	0,04	0,14	0,04	0,14	0,04	0,12
Methyl <i>N</i> -methylantranilate	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.

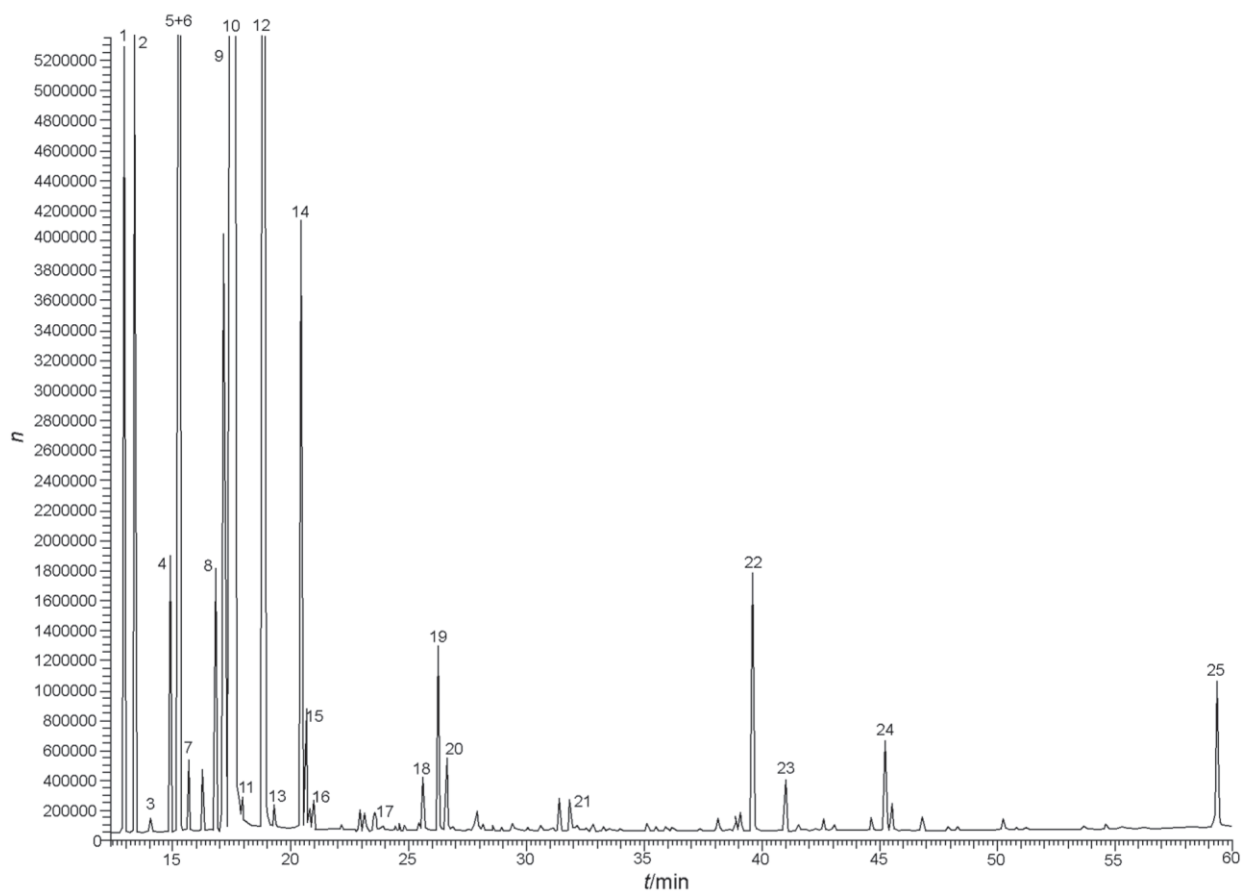
7 Packaging, labelling, marking and storage

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)**



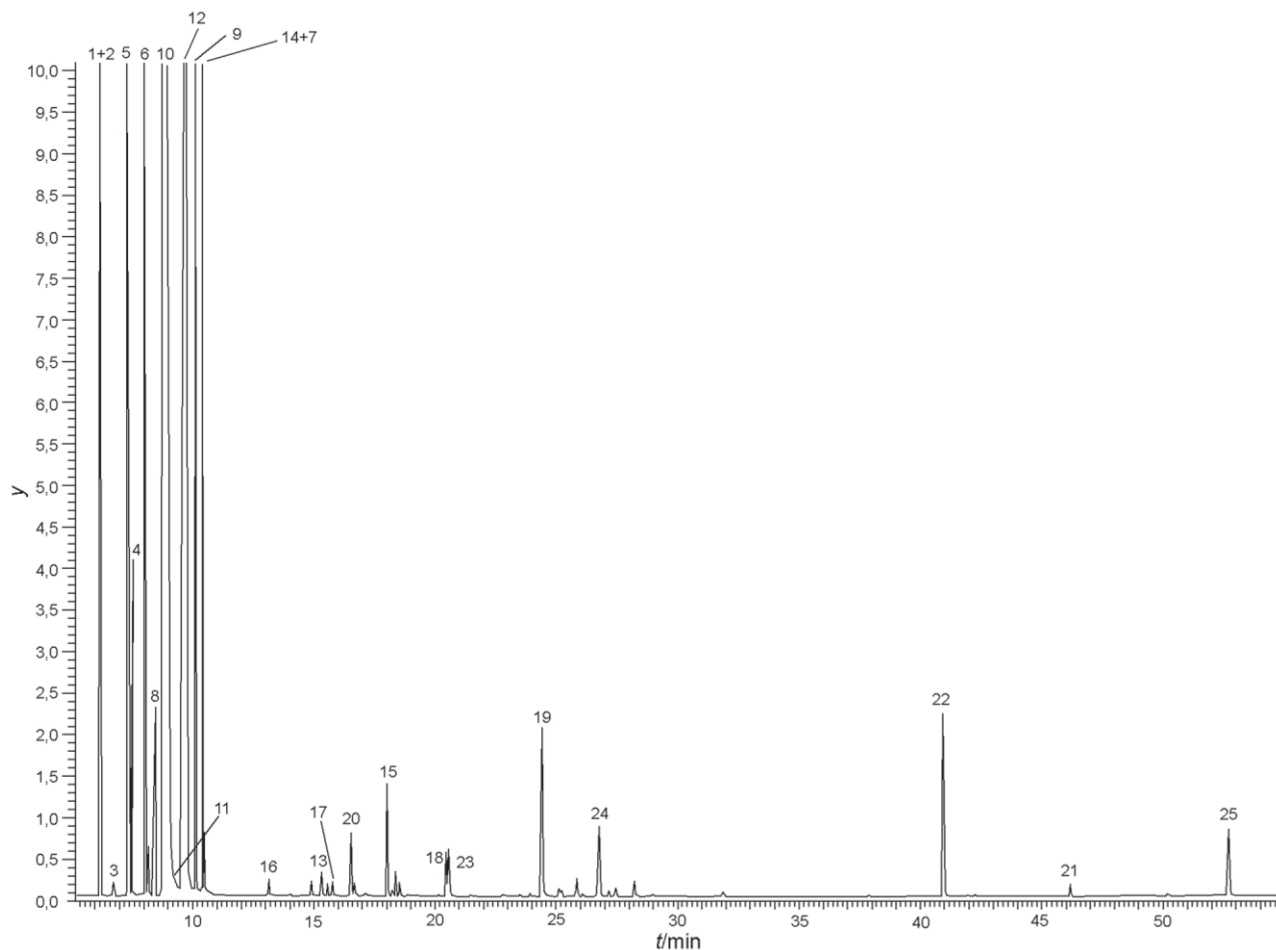
Peak identification

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

Operating conditions

Column: capillary, fused silica, 50 m long and 0,25 mm internal diameter
 Film thickness: 1,00 μ m
 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 μ l
 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	15	Linalool
2	α -Pinene	16	<i>n</i> -Nonanal
3	Camphene	17	Citronellal
4	Sabinene	18	Terpinen-4-ol
5	β -Pinene	19	α -Terpineol
6	Myrcene	20	<i>n</i> -Decanal
7	<i>n</i> -Octanal	21	Thymol
8	α -Terpinene	22	Methyl <i>N</i> -methylantranilate
9	<i>p</i> -Cymene	23	β -Caryophyllene
10	Limonene	24	(<i>E,E</i>)- α -Farnesene
11	β -Phellandrene	25	α -Sinensal
12	γ -Terpinene		
13	(<i>Z</i>)-Sabinene hydrate	<i>y</i>	relative abundance
14	Terpinolene	<i>t</i>	time

Operating conditions

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

Film thickness: 0,25 μ m

Stationary phase: polyethyleneglycol [CW 20M¹]

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 μ l

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-Martens²⁾ 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*

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

Useful Contacts:

Customer Services

Tel: +44 845 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 845 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK



...making excellence a habit.™