INTERNATIONAL STANDARD

ISO 3053

Second edition
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Oil of grapefruit (*Citrus* × *paradisi* Macfad.), obtained by expression

*Huile essentielle de pamplemousse (*Citrus × paradisi *Macfad.), obtenue par expression*

Reference number
ISO 3053:2004(E)

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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 3053 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 3053:1975), which has been technically revised.

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Oil of grapefruit (*Citrus* × *paradisi* Macfad.), obtained by expression

1 Scope

This International Standard specifies certain characteristics of the oil of grapefruit (*Citrus* × *paradisi* Macfad.), obtained by expression, 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 1271, *Essential oils — Determination of carbonyl value — Free hydroxilamine method*

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

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 grapefruit
essential oil obtained, without heating, by mechanical processing from the external part of the pericarp of the fruit of *Citrus* × *paradisi* Macfad., of the Rutaceae family

NOTE 1 The principal areas of production are the United States, Israel, Argentina, Cyprus, South Africa and Brazil.

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

4 Requirements

4.1 Appearance

Clear liquid with possible traces of precipitated waxes.

4.2 Colour

Yellow to pinkish orange.

4.3 Odour

A fresh citrus-like odour, characteristic of grapefruit peel.

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

Minimum: 0,852 0
Maximum: 0,860 0

4.5 Refractive index at 20 °C

Minimum: 1,474 0
Maximum: 1,479 0

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4.6 Optical rotation at 20 °C

Between + 91° and + 96°.

4.7 Total content of aldehydes

Minimum: 0,28 %
Maximum: 2,00 % expressed as decanal (M_r = 156,3)

4.8 Residue on evaporation

Maximum: 10 %

4.9 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.

See ISO 7609.

Table 1 — Chromatographic profile

Component	Minimum %	Maximum %
α-Pinene	0,2	0,6
Sabinene	0,1	0,6
β-Pinene	0,05	0,2
Myrcene	1,5	2,5
Limonene[a]	92	96
n-Octanal	0,2	0,8
n-Nonanal	0,04	0,1
n-Decanal	0,1	0,6
Neral	0,02	0,04
β-Caryophyllene	0,2	0,5
Nootkatone	0,01	0,8

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

[a] In accordance with the present knowledge on this oil and the results of several physical tests carried out in this International Standard, it can be assured that this component is, predominantly, D-limonene.

4.10 Flashpoint

Information on the flashpoint is given in Annex C.

5 Sampling

See ISO 212.

Minimum volume of test sample: 25 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 Total aldehyde content

See ISO 1271.

Test portion: 10 g

Reflux time: 0,5 h

6.5 Residue on evaporation

See ISO 4715.

Test portion: 3 g

Evaporation time: 5 h

6.6 Chromatographic profile

See ISO 11024-1 and ISO 11024-2.

7 Packaging, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

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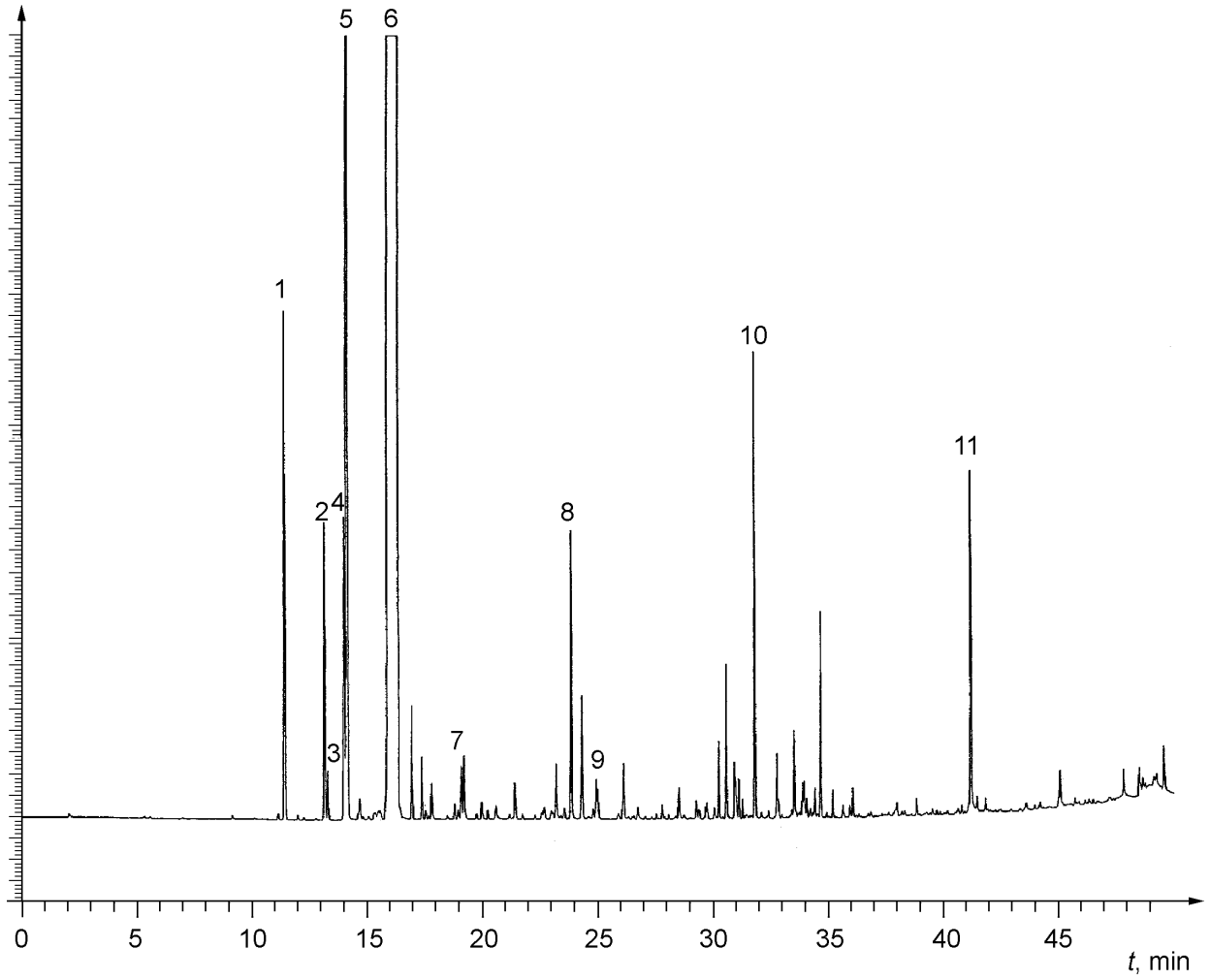
Annex A
(informative)

Typical chromatograms of the analysis by gas chromatography of the essential oil of grapefruit (*Citrus* × *paradisi* Macfad.), obtained by expression



Peak identification

1	α-Pinene
2	β-Pinene
3	Sabinene
4	*n*-Octanal
5	Myrcene
6	Limonene
7	*n*-Nonanal
8	*n*-Decanal
9	Neral
10	β-Caryophyllene
11	Nootkatone

Operating conditions

Column: capillary; length 30 m; internal diameter 0,20 mm
Stationary phase: poly(5 % diphenyl/95 % dimethyl siloxane) (SP-5®)
Film thickness: 20 µm
Oven temperature: isothermal at 75 °C for 5 min, then temperature programming from 75 °C to 100 °C at a rate of 5 °C/min, then from 100 °C to 220 °C at a rate of 6 °C/min and isothermal at 220 °C for 8,5 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Carrier gas flow rate: 22 ml/min
Volume injected: 1 µl
Split ratio: 1/100

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

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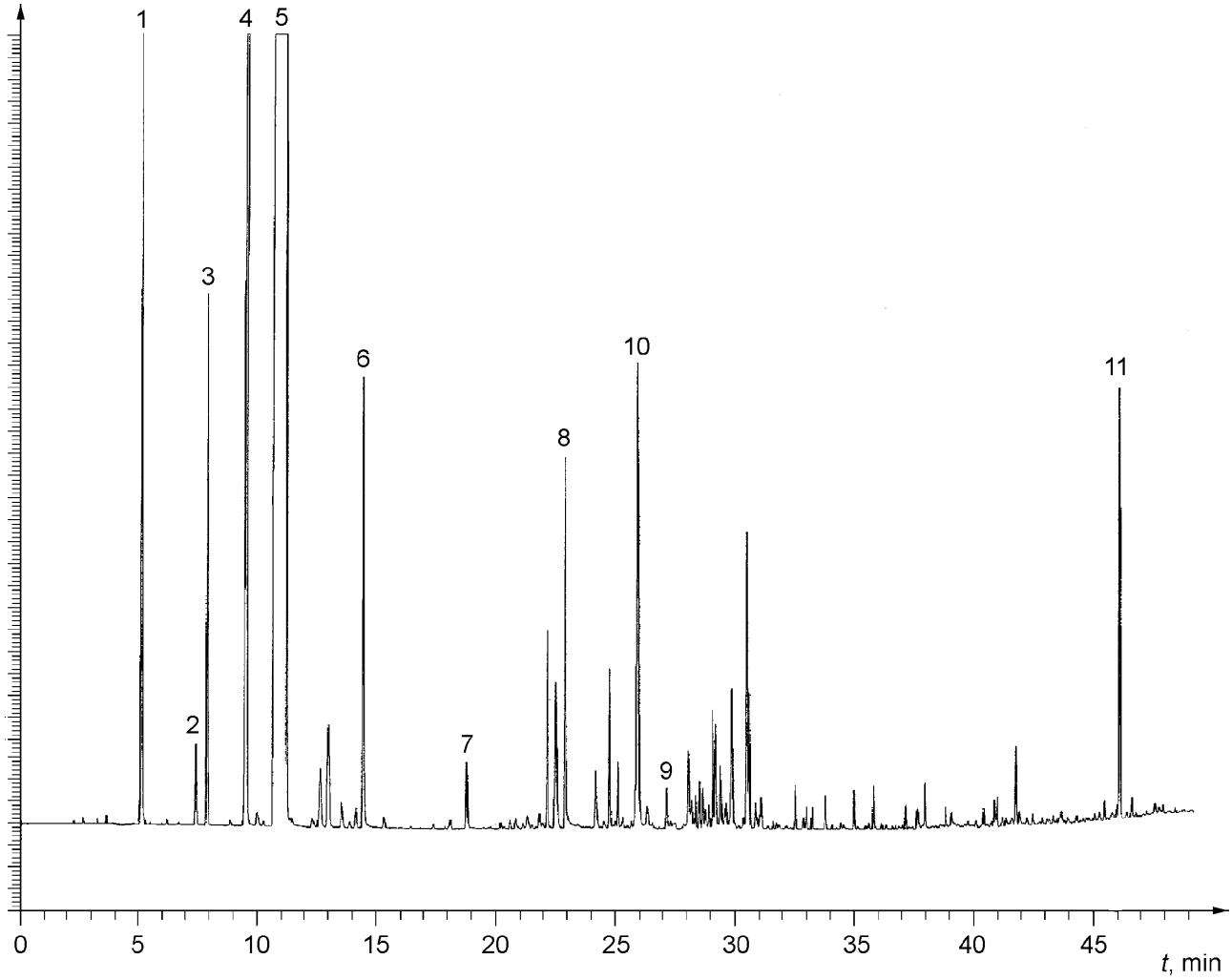

Peak identification	
1	α-Pinene
2	Sabinene
3	β-Pinene
4	Myrcene
5	Limonene
6	*n*-Octanal
7	*n*-Nonanal
8	*n*-Decanal
9	Neral
10	β-Caryophyllene
11	Nootkatone

Operating conditions

Column: capillary; length 30 m; internal diameter 0,20 mm
Stationary phase: poly(ethylene glycol) (Carbowax®)
Film thickness: 20 µm
Oven temperature: isothermal at 75 °C for 5 min, then temperature programming from 75 °C to 100 °C at a rate of 5 °C/min, then from 100 °C to 220 °C at a rate of 6 °C/min and isothermal at 220 °C for 8,5 min
Injector temperature: 230 °C
Detector temperature: 260 °C
Detector: flame ionization type
Carrier gas: helium
Carrier gas flow rate: 22 ml/min
Volume injected: 1 µl
Split ratio: 1/100

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

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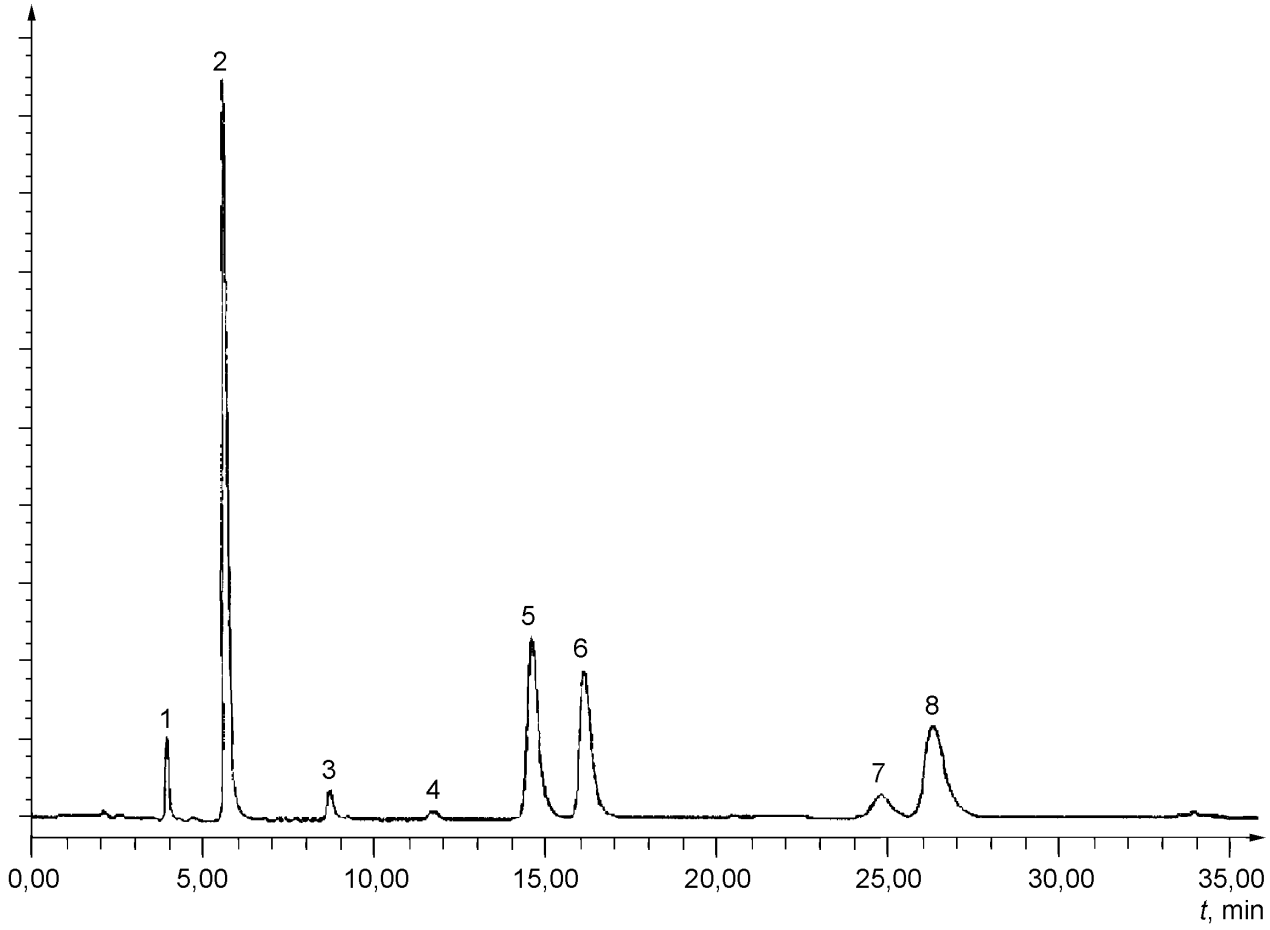
Annex B
(informative)

Typical chromatogram of the analysis by high-pressure liquid chromatography (HPLC) of the essential oil of grapefruit (*Citrus* × *paradisi* Macfad.), obtained by expression



Peak identification

1 Bergamottin
2 Aurapten
3 Osthol
4 Bergapten
5 Epoxybergamottin
6 Epoxyaurapten
7 Isomeranzin
8 Meranzin

Operating conditions

Column: C 18 bonded silica (Sherisorb 5 ods® or equivalent[1)])
Eluents:
A: water/acetic acid (98 %/2 %)
B: acetonitrile
Flow rate 1,5 ml/min
Volume injected: 5,0 µl
UV detection: wavelength of 280 nm from t = 0 min to t = 20 min and then wavelength of 313 nm until the end

Figure B.1 — Typical chromatogram of the analysis by HPLC

1) Sherisorb 5 ods is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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Annex C
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Flashpoint

C.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 a wide variation in the chemical composition of essential oils;

— the volume of the sample needed for 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 in an informative annex to each International Standard, 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.

C.2 Flashpoint of the essential oil of grapefruit

The mean value is + 43 °C.

NOTE Obtained with “Luchaire” equipment.

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Bibliography

[1] ISO/TR 11018:1997, *Essential oils — General guidance on the determination of flashpoint*

[2] ISO/TR 21092:2004, *Essential oils — Characterization*

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