INTERNATIONAL STANDARD

ISO 3061

Second edition
2008-03-15

Oil of black pepper (*Piper nigrum* L.)

*Huile essentielle de poivre noir (*Piper nigrum *L.)*

Reference number
ISO 3061:2008(E)

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Published in Switzerland

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 3061 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 3061:1979), which has been technically revised.

Oil of black pepper (*Piper nigrum* L.)

1 Scope

This International Standard specifies certain characteristics of oil of black pepper (*Piper nigrum* L.), with a view to facilitating the 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 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
essential oil of black pepper
essential oil obtained by steam distillation of the whole or broken unripe fruits of *Piper nigrum* L. of the Piperaceae family.

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

4 Requirements

4.1 Appearance

Clear mobile liquid.

4.2 Colour

Colourless or light coloured (yellow, green, blue).

4.3 Odour

Spicy, woody.

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

	India	Sri Lanka	Indonesia	Madagascar
Minimum	0,864	0,861	0,861	0,864
Maximum	0,880	0,876	0,885	0,884

4.5 Refractive index at 20 °C

	India	Sri Lanka	Indonesia	Madagascar
Minimum	1,478	1,475	1,480	1,475
Maximum	1,487	1,490	1,493	1,490

4.6 Optical rotation at 20 °C

	India	Sri Lanka	Indonesia	Madagascar
Minimum	−18°	−17°	−17°	−18°
Maximum	−7°	−8°	−6°	+20°

4.7 Miscibility with 95 % (volume fraction) ethanol at 20 °C

It shall not be necessary to use more than 3 volumes of 95 % (volume fraction) ethanol to obtain a clear solution with 1 volume of essential oil.

4.8 Chromatographic profile

Analysis of the essential oil shall be carried out by gas chromatography. In the chromatogram obtained, the representative and characteristics components shown in Table 1 shall be identified. The percentages 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

Components	India		Sri Lanka		Indonesia		Madagascar	
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
α–Pinene	3	12	10	16	2,5	10	8	26
β-Pinene	5	12	9	12	nd[a]	3	7	15
Sabinene	6	15	10	17	4	10	nd[a]	4
3-Carene	3	15	5	11	9	20	7	16
Limonene	10	17	13	16	7	12	10	25
δ-Elemene	0,5	3,5	0,5	3	0,5	3	0,5	4,5
α-Copaene	0,5	4,5	1,5	4	0,2	1	nd[a]	0,5
β-Caryophyllene	12	29	12	21	25	40	10	25
Germacrene D	nd[a]	2	nd[a]	1	nd[a]	1	1	6,5
β-Selinene	0,5	3,5	nd[a]	2	3	6	1	4,5
α-Selinene	nd[a]	3	0,5	2	2,5	5	0,5	3
Caryophyllene oxide	nd[a]	1	nd[a]	1	nd[a]	1	nd[a]	1

[a] non-detectable.

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

4.9 Flashpoint

Information on the flashpoint is given in Annex B.

5 Sampling

See ISO 212.

Minimum volume of the final sample: 25 ml.

NOTE This volume is sufficient for 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 with 95 % (volume fraction) ethanol at 20 °C

See ISO 875.

6.5 Chromatographic profile

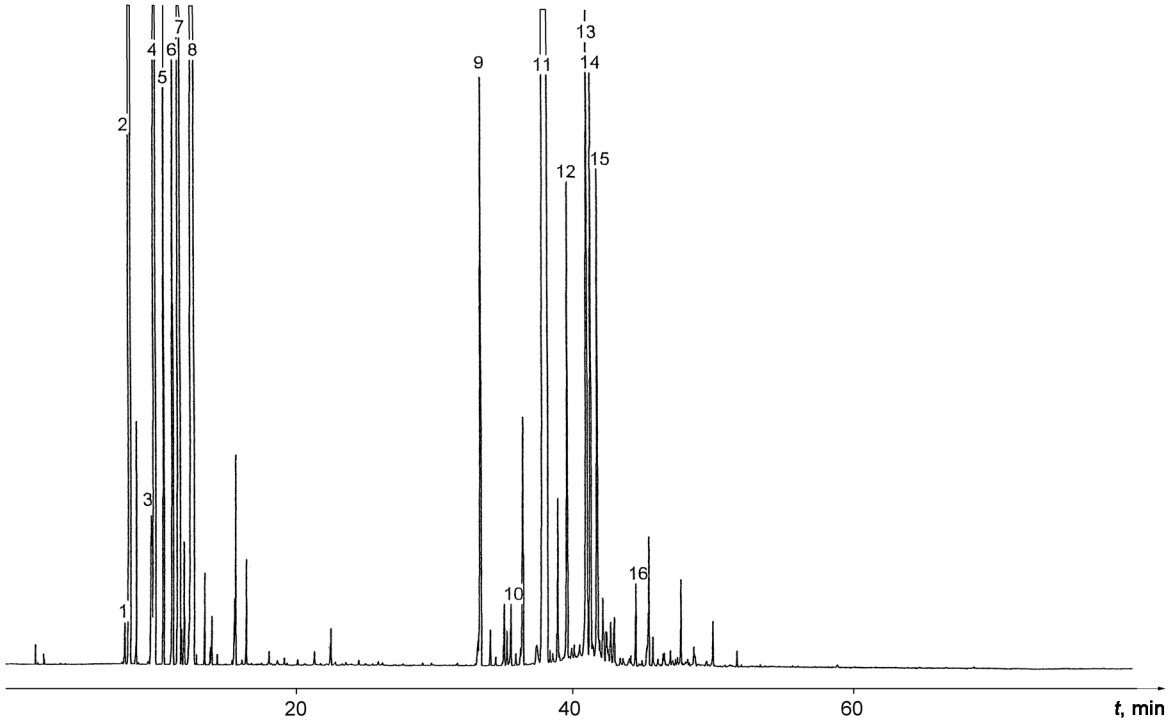
See ISO 11024-1 and ISO 11024-2.

7 Packing, labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

Annex A
(informative)

Typical chromatogram of the analysis by gas chromatography of the essential oil of black pepper (*Piper nigrum* L.)



Peak identification

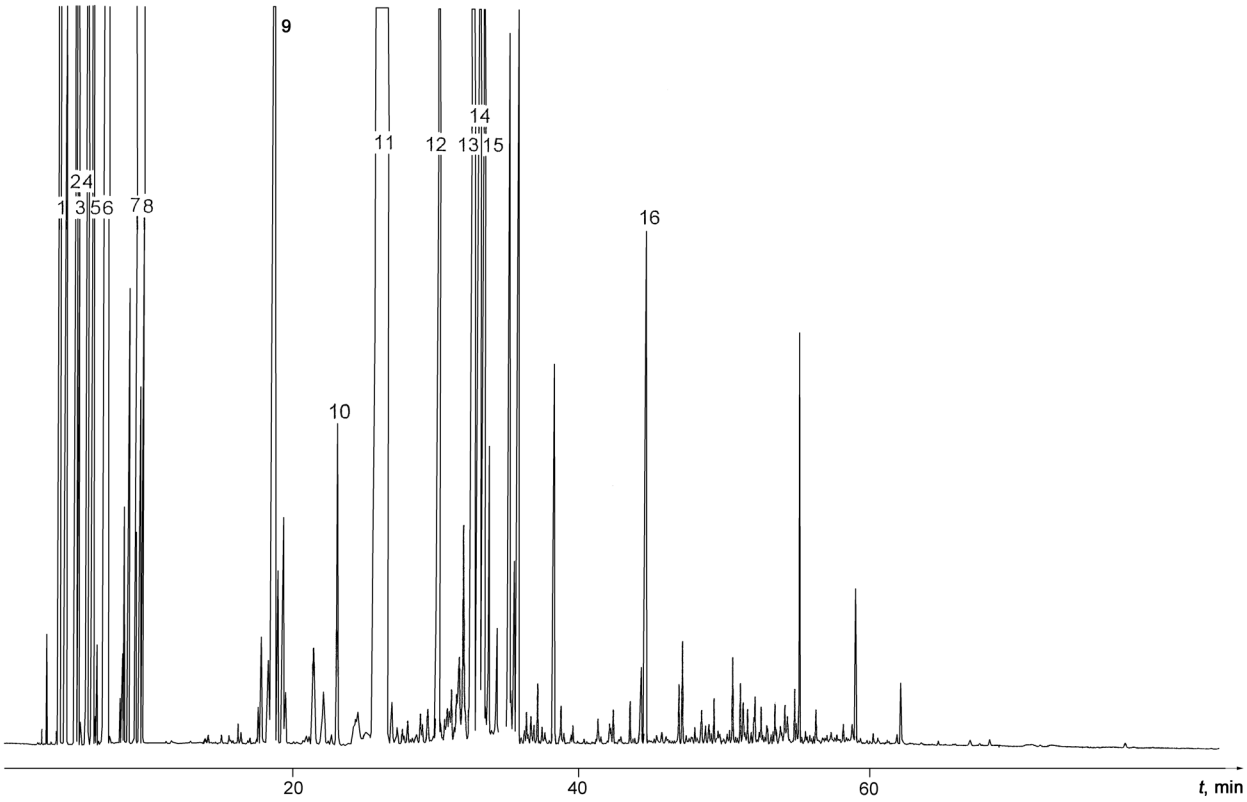
1 α-Thujene
2 α-Pinene
3 Sabinene
4 β-Pinene
5 Myrcene
6 α-Phellandrene
7 3-Carene
8 Limonene + 1,8-Cineole
9 δ-Elemene
10 α-Copaene
11 β-Caryophyllene
12 α-Humulene
13 Germacrene D
14 β-Selinene
15 α-Selinene
16 Caryophyllene oxide

Operating conditions

Column: silica capillary; length 50 m; internal diameter 0,2 mm
Stationary phase: polydimethylsiloxane [HP–1[1)]]
Film thickness: 0,25 µm
Oven temperature: 65 °C to 230 °C, at a rate of 2 °C/min
Injector temperature: 230 °C
Detector temperature: 250 °C
Detector: flame ionization type
Carrier gas: hydrogen
Volume injected: 0,2 µl
Carrier gas flow rate: 1 ml/min
Split ratio: 1/100

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

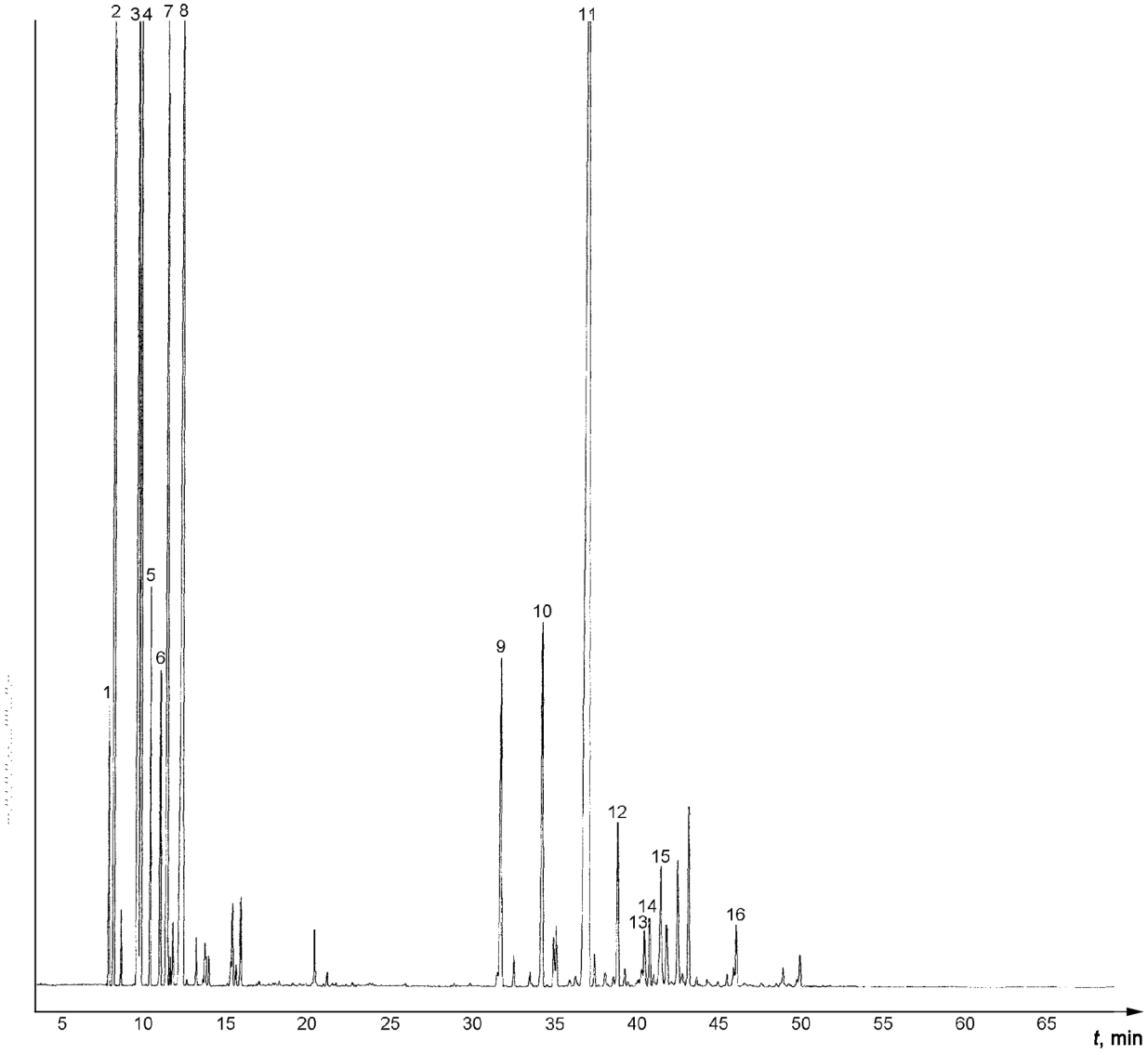
1) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard, and does not constitute an endorsement of this product by ISO. Equivalent products may be used if they can be shown to lead to the same results.



Peak identification	Operating conditions
1 α-Pinene + α-Thujene	Column: silica capillary; length 50 m; internal diameter 0,2 mm
2 β-Pinene	Stationary phase: poly(ethylene glycol) [Carbowax [2)]]
3 Sabinene	Film thickness: 0,25 µm
4 3-Carene	Oven temperature: 65 °C to 230 °C, at a rate of 10 °C/min
5 α-Phellandrene + Myrcene	Injector temperature: 230 °C
6 Limonene	Detector temperature: 250 °C
7 *p*-Cymene	Detector: flame ionization type
8 Terpinolene	Carrier gas: hydrogen
9 δ-Elemene	Volume injected: 0,2 µl
10 α-Copaene	Carrier gas flow rate: 1,1 ml/min
11 β-Caryophyllene + Terpinen-4-ol	Split ratio: 1/100
12 α-Humulene	
13 Germacrene D	
14 β-Selinene	
15 α-Selinene	
16 Caryophyllene oxide	

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

2) 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 of this product by ISO. Equivalent products may be used if they can be shown to lead to the same results.



Peak identification

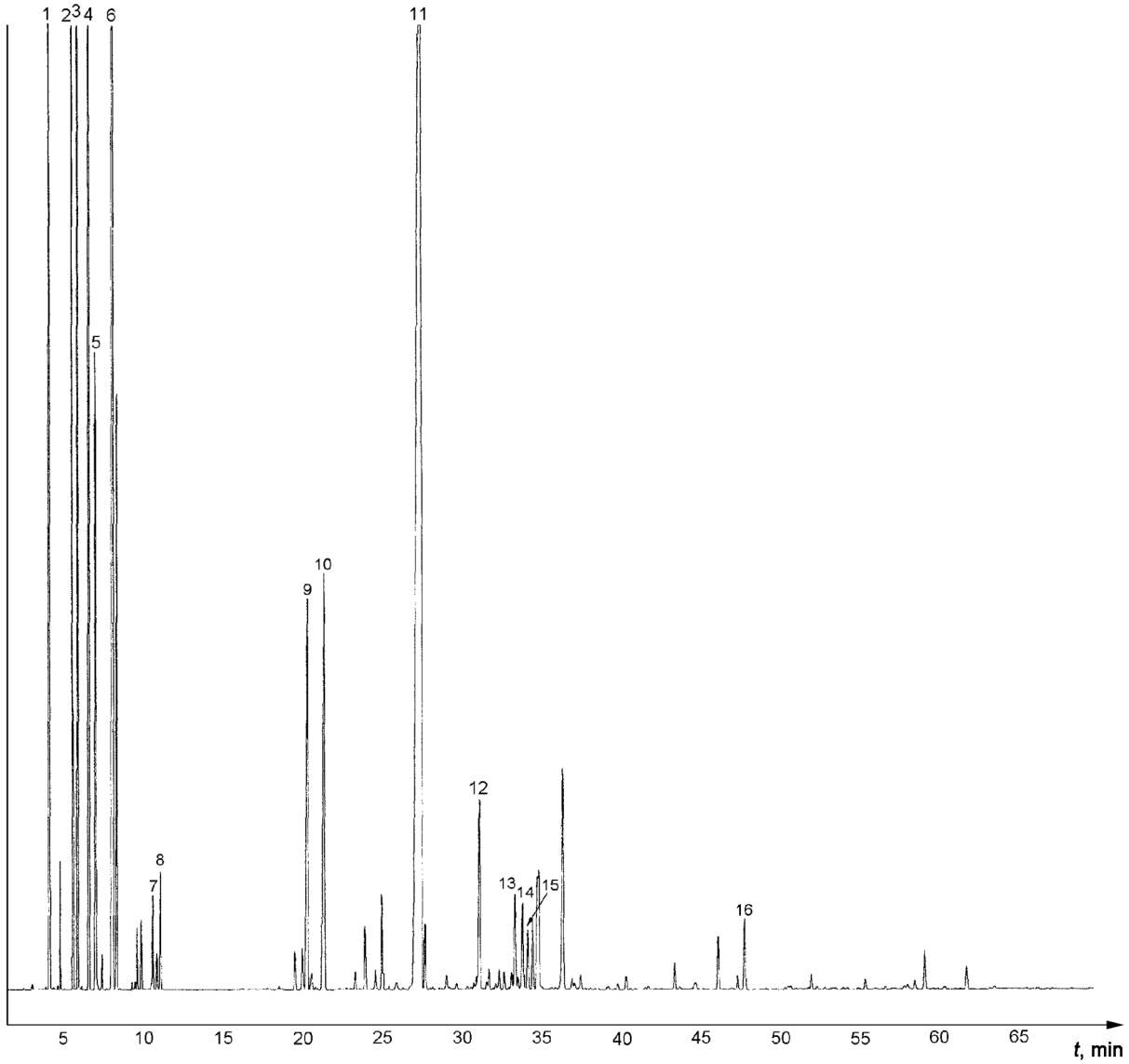
1 α-Thujene
2 α-Pinene
3 Sabinene
4 β-Pinene
5 Myrcene
6 α-Phellandrene
7 3-Carene
8 Limonene + 1,8-Cineole
9 δ-Elemene
10 α-Copaene
11 β-Caryophyllene
12 α-Humulene
13 Germacrene D
14 β-Selinene
15 α-Selinene
16 Caryophyllene oxide

Operating conditions

Column: silica capillary; length 30 m; internal diameter 0,25 mm
Stationary phase: polydimethylsiloxane [HP-1[3)]]
Film thickness: 0,25 µm
Oven temperature: isothermal at 60 °C for 1 min, then programming temperature from 60 °C to 240 °C at a rate of 2 °C/min, then isothermal at 240 °C for 10 min
Injector temperature: 250 °C
Detector temperature: 300 °C
Detector: flame ionization type
Carrier gas: hydrogen
Volume injected: 0,5 µl
Carrier gas flow rate: 100 ml/min
Split ratio: 1/100

Figure A.3 — Typical chromatogram taken on an apolar column, Indian type

3) 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 of this product by ISO. Equivalent products may be used if they can be shown to lead to the same results.



Peak identification

1 α-Pinene + α-Thujene
2 β-Pinene
3 Sabinene
4 3-Carene
5 α-Phellandrene + Myrcene
6 Limonene
7 *p*-Cymene
8 Terpinolene
9 δ-Elemene
10 α-Copaene
11 β-Caryophyllene + Terpinen-4-ol
12 α-Humulene
13 Germacrene D
14 β-Selinene
15 α-Selinene
16 Caryophyllene oxide

Operating conditions

Column: silica capillary; length 30 m; internal diameter 0,25 mm

Stationary phase: poly(ethylene glycol) [Innowax[4)]]

Film thickness: 0,25 µm

Oven temperature: isothermal at 60 °C for 1 min, then programming temperature from 60 °C to 240 °C at a rate of 2 °C/min, then isothermal at 240 °C for 10 min

Injector temperature: 250 °C

Detector temperature: 300 °C

Detector: flame ionization type

Carrier gas: hydrogen

Volume injected: 0,5 µl

Carrier gas flow rate: 100 ml/min

Split ratio: 1/100

Figure A.4 — Typical chromatogram taken on a polar column, Indian type

4) 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 of this product by ISO. Equivalent products may be used if they can be shown to lead to the same results.

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 purchase 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 black pepper (*Piper nigrum* L.)

The mean value is +48 °C (obtained using Madagascar type).

NOTE Obtained with “Setaflash” equipment.

Bibliography

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

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

ICS 71.100.60

Price based on 9 pages