INTERNATIONAL STANDARD

ISO 17412

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Oil of bitter fennel (*Foeniculum vulgare* Mill. ssp. *vulgare* var. *vulgare*)

*Huile essentielle de fenouil amer (*Foeniculum vulgare *Mill. ssp.* vulgare *var.* vulgare*)*

Reference number
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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 17412 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

Oil of bitter fennel (*Foeniculum vulgare* Mill. ssp. *vulgare* var. *vulgare*)

1 Scope

This International Standard specifies certain characteristics of the oil of bitter fennel (*Foeniculum vulgare* Mill. ssp. *vulgare* var. *vulgare*), 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 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 1041, *Essential oils — Determination of freezing point*

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 bitter fennel
essential oil obtained by steam distillation, with or without subsequent rectification, of the fruits and aerial parts of *Foeniculum vulgare* Mill. ssp. *vulgare* var. *vulgare* of the Apiaceae family; some authors use the name *Foeniculum vulgare* Miller ssp. *vulgare* var. *amara*

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

4 Requirements

4.1 Appearance

***trans*-Anethole type**	**Phellandrene type**
Clear liquid or crystalline mass	Mobile liquid

4.2 Colour

***trans*-Anethole type**	**Phellandrene type**
Colourless to pale yellow liquid	Pale yellow to intense yellow liquid

4.3 Odour

***trans*-Anethole type**	**Phellandrene type**
Characteristic, recalling that of anethole	Sweet, herbaceous, characteristic

4.4 Taste

Sweet with a bitter after-taste.

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

	trans-Anethole type	Phellandrene type
Min.	0,944	0,877
Max.	0,973	0,920

4.6 Refractive index at 20 °C

	trans-Anethole type	Phellandrene type
Min.	1,514	1,487
Max.	1,538	1,501

4.7 Optical rotation at 20 °C

	trans-Anethole type	Phellandrene type
Min.	+11°	+42°
Max.	+29°	+68°

4.8 Miscibility in ethanol at 20 °C

trans-Anethole type	Phellandrene type
It shall not be necessary to use more than 10 volumes of ethanol 85 % (volume fraction) to obtain a clear solution with 1 volume of essential oil.	It shall not be necessary to use more than 2 volumes of ethanol 90 % (volume fraction) to obtain a clear solution with 1 volume of essential oil.

4.9 Freezing point

	trans-Anethole type
Min.	+3 °C
Max.	+10 °C
NOTE Freezing points higher than 10 °C often indicate adulteration with aniseed oil or pure anethole.	

The freezing point is not a relevant requirement for the phellandrene type, due to the low anethole content.

4.10 Chromatographic profile

Analysis of the essential oil shall be carried out by gas chromatography. There are two types of bitter fennel oil: *trans*-anethole and phellandrene. In the chromatogram obtained, the representative and characteristics components shown in Table 1 and Table 2 shall be identified. The proportions of these components, indicated by the integrator, shall be as shown in Table 1 and Table 2. This constitutes the chromatographic profile of the essential oil.

Table 1 — *trans*-Anethole type chromatographic profile

Component	Min. %	Max. %
α-Pinene	2,0	11,0
β-Pinene	trace	1,0
Myrcene	0,5	2,0
α-Phellandrene	trace	8,5
Limonene	1,0	6,0
Fenchone	10,0	25,0
Methyl chavicol (estragole)	1,0	6,0
trans-Anethole	50,0	78,0
cis-Anethole	nd [a]	0,5
Anisaldehyde	trace	1,0
1-(4-Methoxyphenyl)propan-2-one (anise ketone)	nd [a]	1,0
NOTE The chromatographic profile is normative, contrary to typical chromatograms given for information in Annex A.		
[a] Non-detectable.		

Table 2 — Phellandrene type chromatographic profile

Component	Min. %	Max. %
α-Pinene	2,0	8,0
β-Pinene	1,0	4,0
Myrcene	1,0	12,0
α-Phellandrene	8,0	25,0
Limonene	8,0	30,0
Fenchone	7,0	16,0
Methyl chavicol (estragole)	2,0	7,0
trans-Anethole	15,0	30,0
cis-Anethole	trace	0,5
Anisaldehyde	trace	0,3
1-(4-Methoxyphenyl)propan-2-one (anise ketone)	trace	0,05

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

4.11 Flashpoint

Information on the flashpoint is given in Annex B.

5 Sampling

See ISO 212.

The minimum volume of test sample is 50 ml.

NOTE The 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 Miscibility in ethanol at 20 °C

See ISO 875.

6.5 Freezing point

See ISO 1041.

6.6 Chromatographic profile

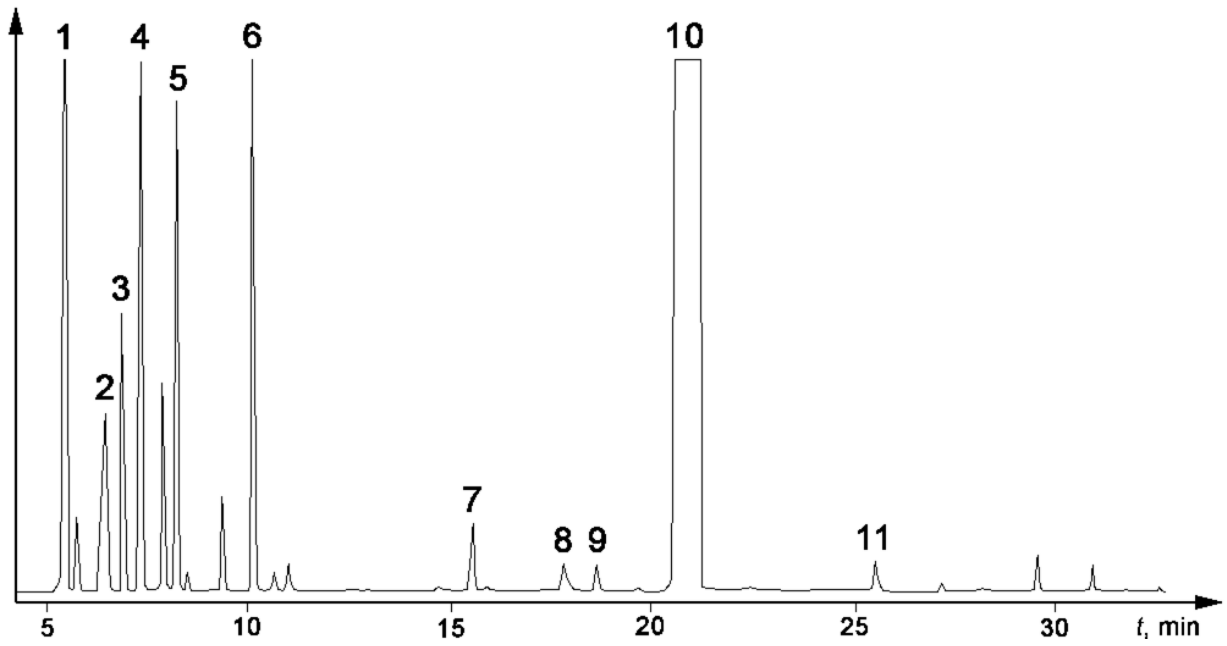
See ISO 11024-1 and ISO 11024-2.

7 Packaging labelling, marking and storage

See ISO/TR 210 and ISO/TR 211.

Annex A
(informative)

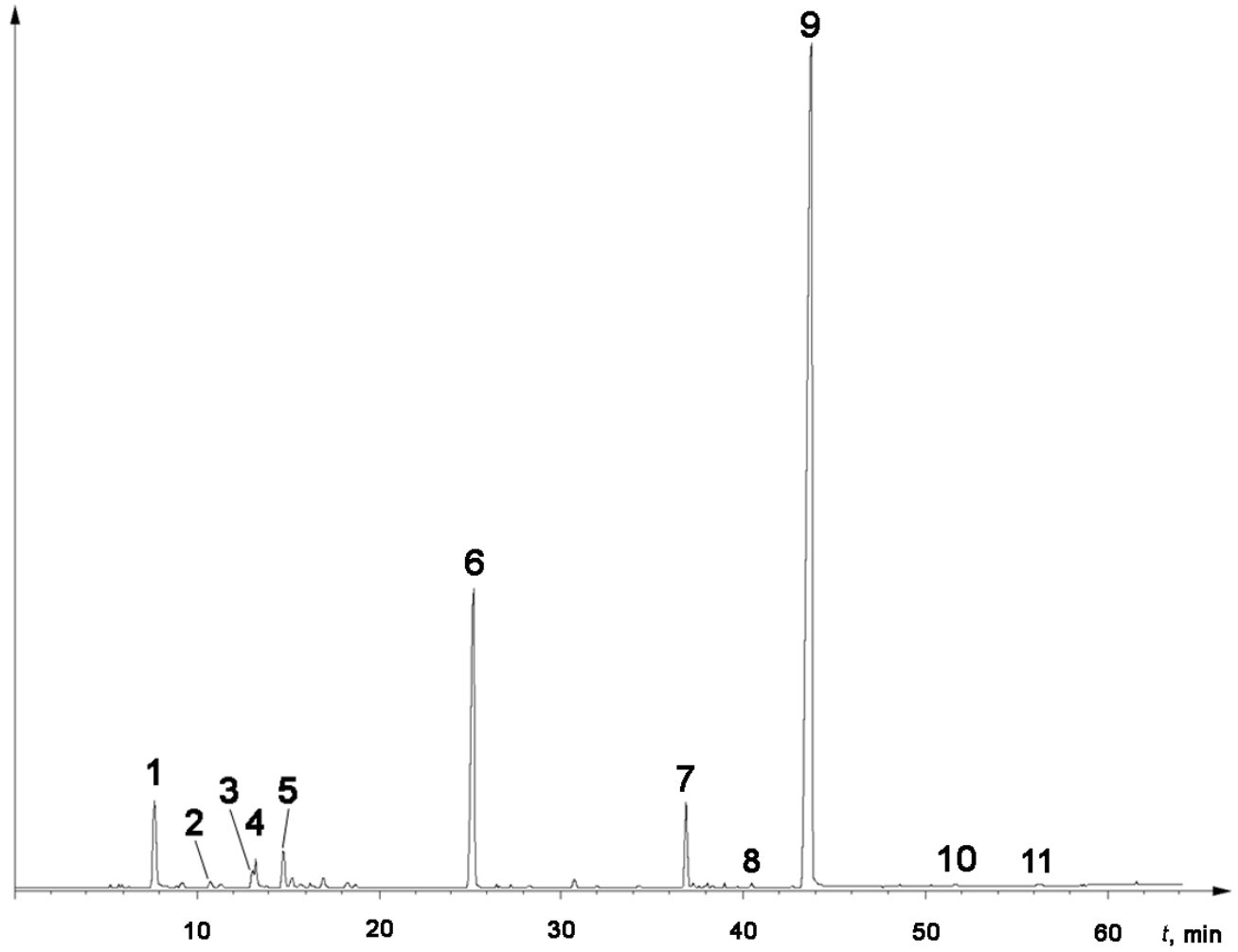
Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter fennel (*Foeniculum vulgare* Mill. ssp. *vulgare* var. *vulgare*)



Peak identification		Operating conditions
1	α-Pinene	Column: FSOT, length 50 m, internal diameter 0,25 mm
2	β-Pinene	Stationary phase: methyl silicone [BP1[1)]]
3	Myrcene	Film thickness: 0,20 µm
4	α-Phellandrene	Oven temperature: isothermal at 65 °C for 1 min, then temperature programming from 65 °C to 180 °C at a rate of 2 °C/min and then isothermal at 180 °C for 10 min
5	Limonene	
6	Fenchone	Injector temperature: 200 °C
7	Methyl chavicol (estragole)	Detector temperature: 300 °C
8	Anisaldehyde	Detector: flame ionization type
9	*cis*-Anethole	Carrier gas: hydrogen
10	*trans*-Anethole	Volume injected: 0,1 µl
11	1-(4-Methoxyphenyl)propan-2-one	Carrier gas flow rate: 3,5 ml/min
		Split ratio: 1:50

Figure A.1 —Typical chromatogram of *trans*-anethole type taken using an apolar column

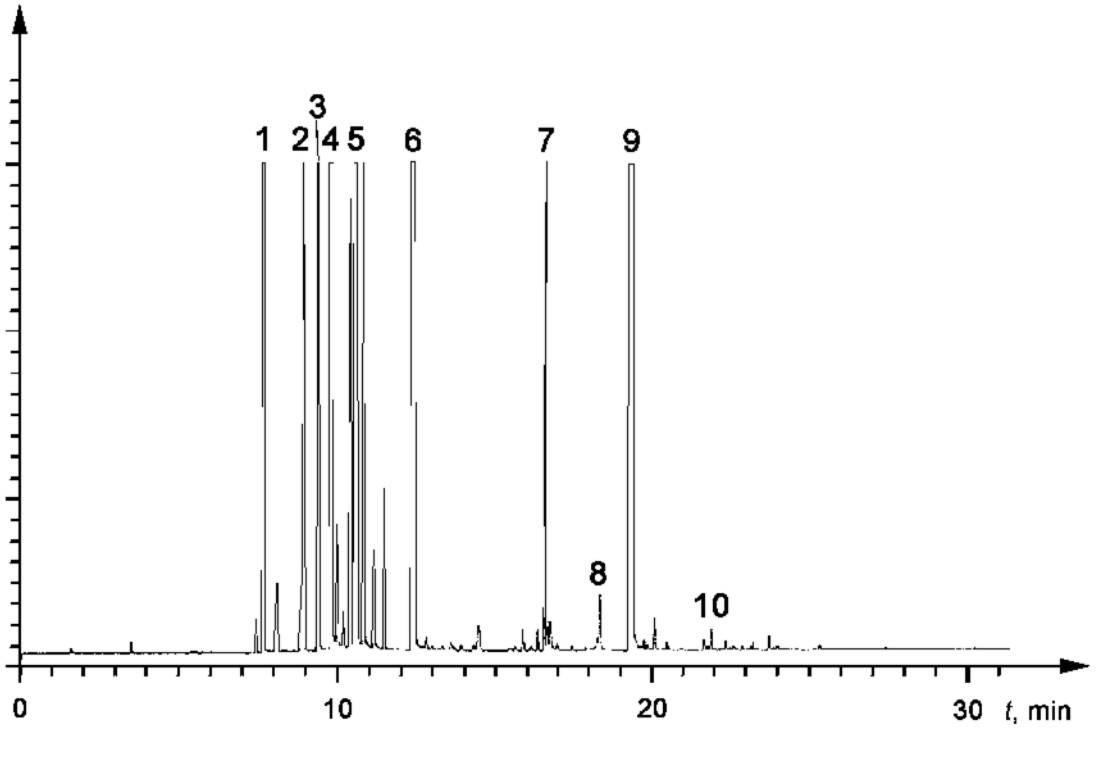
1) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.



Peak identification		Operating conditions
1	α-Pinene	Column: FSOT, length 60 m, internal diameter 0,53 mm
2	β-Pinene	Stationary phase: polyethylene glycol [DB-Wax[2)]]
3	Myrcene	Film thickness: 1,0 µm
4	α-Phellandrene	Oven temperature: isothermal at 50 °C for 5 min, then temperature programming from 50 °C to 220 °C at a rate of 3 °C/min and then isothermal at 220 °C for 15 min
5	Limonene	
6	Fenchone	Injector temperature: 220 °C
7	Methyl chavicol (estragole)	Detector temperature: 220 °C
8	*cis*-Anethole	Detector: flame ionization type
9	*trans*-Anethole	Carrier gas: helium
10	Anisaldehyde	Volume injected: 0,02 µl
11	1-(4-Methoxyphenyl)propan-2-one	Carrier gas flow rate: 4 ml/min
		Split ratio: nil

Figure A.2 — Typical chromatogram of *trans*-anethole type taken using a polar column

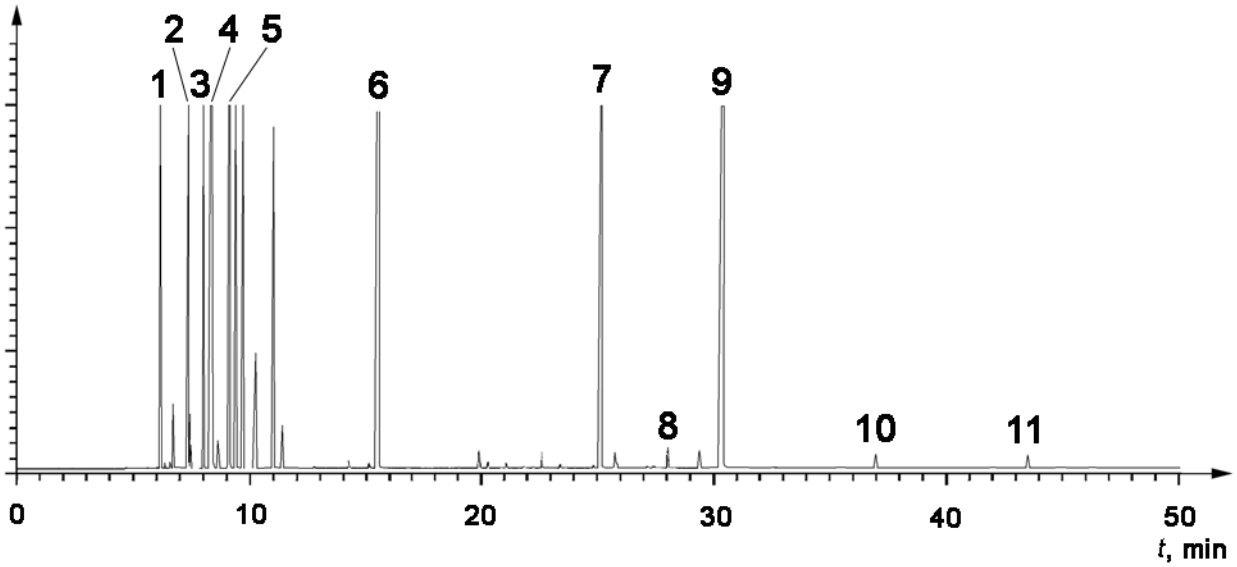
2) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.



Peak identification		Operating conditions
1	α-Pinene	Column: capillary, fused silica, length 30 m, internal diameter 0,25 mm
2	β-Pinene	Stationary phase: 5 % diphenyl-95 % dimethyl siloxane [SPB-5[3)]]
3	Myrcene	Film thickness: 0,25 µm
4	α-Phellandrene	Oven temperature: temperature programming from 55 °C to 100 °C at a rate of 5,5 °C/min, then temperature programming from 100 °C to 200 °C at a rate of 8 °C/min and then isothermal at 200 °C for 10 min
5	Limonene	
6	Fenchone	Injector temperature: 250 °C
7	Methyl chavicol (estragole)	Detector temperature: 250 °C
8	*cis*-Anethole + anisaldehyde	Detector: flame ionization type
9	*trans*-Anethole	Carrier gas: helium
10	1-(4-Methoxyphenyl)propan-2-one	Volume injected: 0,1 µl
		Carrier gas flow rate: 1 ml/min
		Split ratio: 1:100

Figure A.3 —Typical chromatogram of phellandrene type taken using an apolar column

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 by ISO of this product.



Peak identification		Operating conditions
1	α-Pinene	Column: capillary, fused silica, length 60 m, internal diameter 0,25 mm
2	β-Pinene	Stationary phase: polyethylene glycol [SP-1000[4)]]
3	Myrcene	Film thickness: 0,25 µm
4	α-Phellandrene	Oven temperature: temperature programming from 95 °C to 190 °C at a rate of 4 °C/min and then isothermal at 190 °C for 8 min
5	Limonene	Injector temperature: 250 °C
6	Fenchone	Detector temperature: 250 °C
7	Methyl chavicol (estragole)	Detector: flame ionization type
8	*cis*-Anethole	Carrier gas: nitrogen
9	*trans*-Anethole	Volume injected: 0,1 µl
10	Anisaldehyde	Carrier gas flow rate: 1 ml/min
11	1-(4-Methoxyphenyl)propan-2-one	Split ratio: 1:100

Figure A.4 — Typical chromatogram of phellandrene type taken using a polar column

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 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) 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 (see Table B.1) 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.

B.2 Flashpoint of the essential oil of bitter fennel

***trans*-Anethole type**	**Phellandrene type**
+63 °C	+48 °C
NOTE Obtained with Pensky-Martens[5)] equipment.	NOTE Obtained with Setaflash[6)] equipment.

5) 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.

6) 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.

Bibliography

[1] ISO/TR 11018:1997, *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