INTERNATIONAL STANDARD

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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)

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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.

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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.

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ISO/TR 210, Essential oils — General rules for packaging, conditioning and storage

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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 $^{\circ}\text{C}$ — 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

4,1,00 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°
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4.8 Miscibility in ethanol at 20 °C (Standar

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 indicate a	Freezing points higher than 10 °C often 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

J	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
DA	Fenchone CEVEW	10,0	25,0
lar	Methyl chavicol (estragole)	1,0	6,0
	trans-Anethole	50,0	78,0
SO 17	cis-Anethole	nd ^a	0,5
19/stan 02530	Anisaldehyde Spiral of the state of the stat	trace	1,0
	1-(4-Methoxyphenyl)propan-2-one (anise ketone)	nd ^a	1,0
	NOTE The chromatographic profile is n to typical chromatograms given for information		•

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		

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.

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4.11 Flashpoint

(standards.iten.al) 1024-1 and ISO 11024-2.

Information on the flashpoint is given in Annex B.

to typical chromatograms given for information in Annex A.

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5 Sampling

See ISO 212.

The minimum volume of test sample is 50 ml.

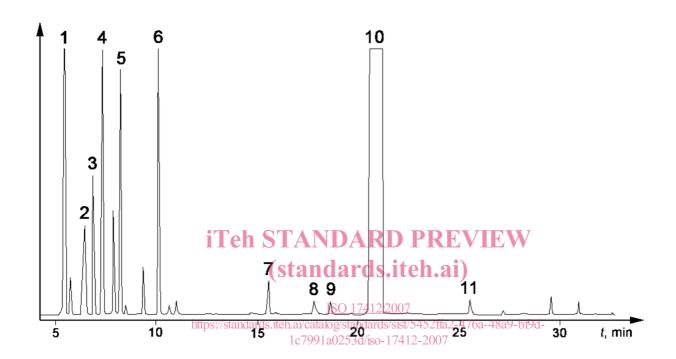
NOTE The volume allows each of the tests specified in this International Standard to be carried out at least once.

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** Column: FSOT, length 50 m, internal diameter 0,25 mm α -Pinene β-Pinene Stationary phase: methyl silicone [BP11) 2 Film thickness: 0,20 µm Myrcene Oven temperature: isothermal at 65 °C for 1 min, then temperature α -Phellandrene

programming from 65 °C to 180 °C at a rate of 2 °C/min and then isothermal at Limonene 5 180 °C for 10 min 6

Fenchone

Injector temperature: 200 °C Methyl chavicol (estragole) 7 Detector temperature: 300 °C Anisaldehyde 8 Detector: flame ionization type cis-Anethole

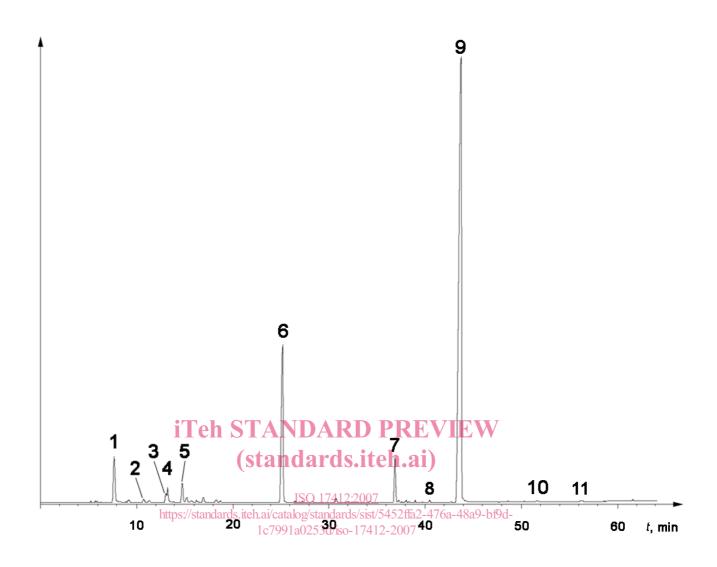
Carrier gas: hydrogen 10 trans-Anethole Volume injected: 0,1 µl 11 1-(4-Methoxyphenyl)propan-2-one

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** α -Pinene Column: FSOT, length 60 m, internal diameter 0,53 mm 1 2 β-Pinene Stationary phase: polyethylene glycol [DB-Wax²)] 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 5 Limonene 220 °C for 15 min 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

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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.