

# Oil of bitter orange (*Citrus aurantium* L.)

ICS 71.100.60

## National foreword

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### Summary of pages

This document comprises a front cover, an inside front cover, the ISO title page, pages ii to iv, pages 1 to 11 and a back cover.

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**Oil of bitter orange (*Citrus aurantium* L.)**

*Huile essentielle d'orange amère (Citrus aurantium L.)*



Reference number  
ISO 9844:2006(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.

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ISO 9844 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 9844:1991), which has been technically revised.

# Oil of bitter orange (*Citrus aurantium* L.)

## 1 Scope

This International Standard specifies certain characteristics of the oil of bitter orange (*Citrus aurantium* L.), 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 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 bitter orange

essential oil obtained by expression, without heating, by mechanical treatment, from the pericarp of the fruit of *Citrus aurantium* L., of the Rutaceae family

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

## 4 Requirements

### 4.1 Appearance

Liquid.

### 4.2 Colour

Pale yellow to brownish green.

### 4.3 Odour

Characteristic of the outer part of bitter orange peel.

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

American type		Equatorial type		Mediterranean type	
min.	max.	min.	max.	min.	max.
0,840	0,860	0,845	0,860	0,840	0,860

**4.5 Refractive index at 20 °C**

American type		Equatorial type		Mediterranean type	
min.	max.	min.	max.	min.	max.
1,472	1,476	1,473	1,478	1,472	1,476

**4.6 Optical rotation at 20 °C**

American type	Equatorial type	Mediterranean type
+ 88° to + 98°	+ 88° to + 95°	+ 88° to + 98°

**4.7 Miscibility in ethanol, 90 % (volume fraction), at 20 °C**

It shall not be necessary to use more than 8 volumes of ethanol, 90 % (volume fraction), to obtain a clear solution with 1 volume of essential oil.

**4.8 Residue on evaporation**

American type		Equatorial type		Mediterranean type	
min.	max.	min.	max.	min.	max.
3,5 %	6,0 %	3,0 %	6,0 %	3,5 %	6,0 %

**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.

**4.10 Flashpoint**

Information on the flashpoint is given in Annex B.



Table 1 — Chromatographic profile

Components	American type		Equatorial type		Mediterranean type	
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum
	%	%	%	%	%	%
$\alpha$ -Pinene	0,3	0,7	0,3	0,6	0,2	0,7
$\beta$ -Pinene	0,3	1,2	0,3	0,8	0,2	1,2
Myrcene	1,6	2,0	1,6	2,0	1,5	3,0
Limonene	93,0	95,0	92,0	95,0	93,0	95,0
Linalool	0,1	0,4	0,1	0,4	0,1	0,4
Linalyl acetate	0,6	1,0	traces	0,5	0,5	1,0
Geranyl acetate	0,1	0,3	nd <sup>a</sup>	0,3	0,1	0,3
$\beta$ -Caryophyllene	traces	0,2	traces	0,1	traces	0,2
Germacrene D	0,1	0,2	traces	0,1	traces	0,2
<i>trans</i> - $\beta$ -Nerolidol	traces	0,2	nd <sup>a</sup>	0,3	traces	0,2
Octanal	traces	0,2	nd <sup>a</sup>	0,3	traces	0,1
Decanal	0,1	0,2	nd <sup>a</sup>	0,2	traces	0,3
<sup>a</sup> Undetected.						
NOTE The chromatographic profile is normative, contrary to the typical chromatograms given for information in Annex A.						

## 5 Sampling

See 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, $d_{20}^{20}$

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, 90 % (volume fraction), at 20 °C

See ISO 875.

### 6.5 Residue on evaporation

See ISO 4715.

### 6.6 Chromatographic profile

See ISO 11024-1 and ISO 11024-2.

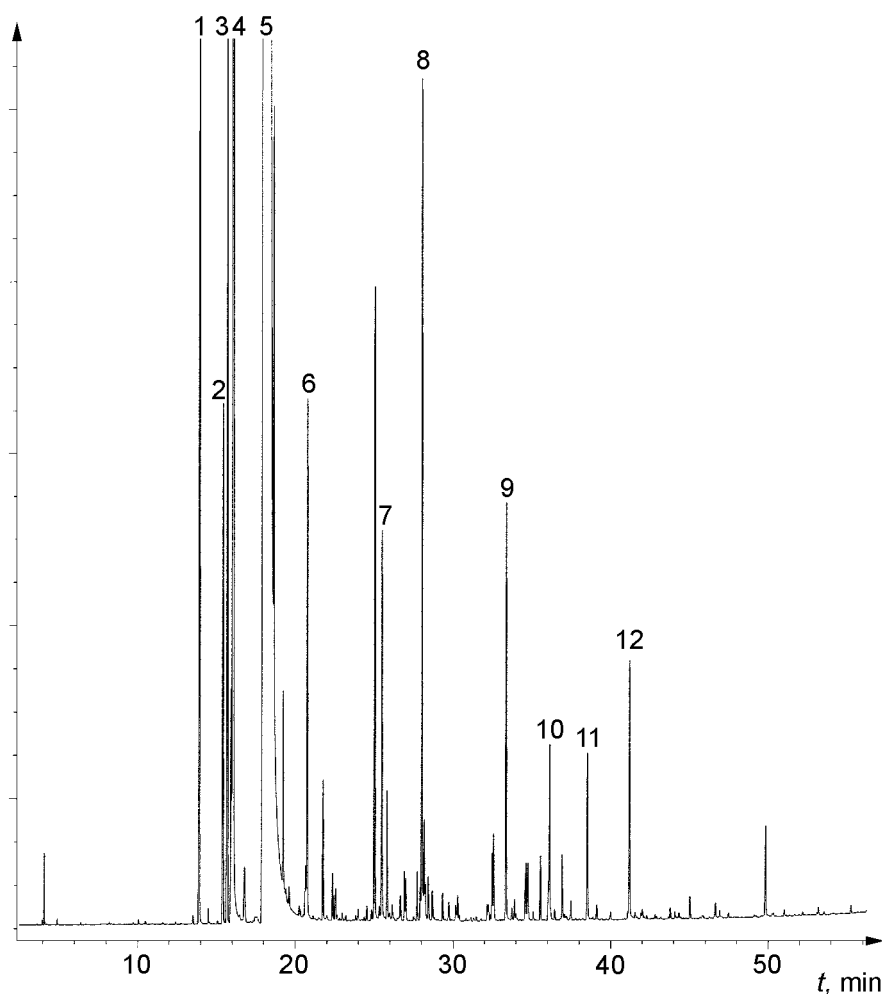
## 7 Packaging, labelling, marking and storage

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 orange (*Citrus aurantium* L.)

#### A.1 Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.), American type



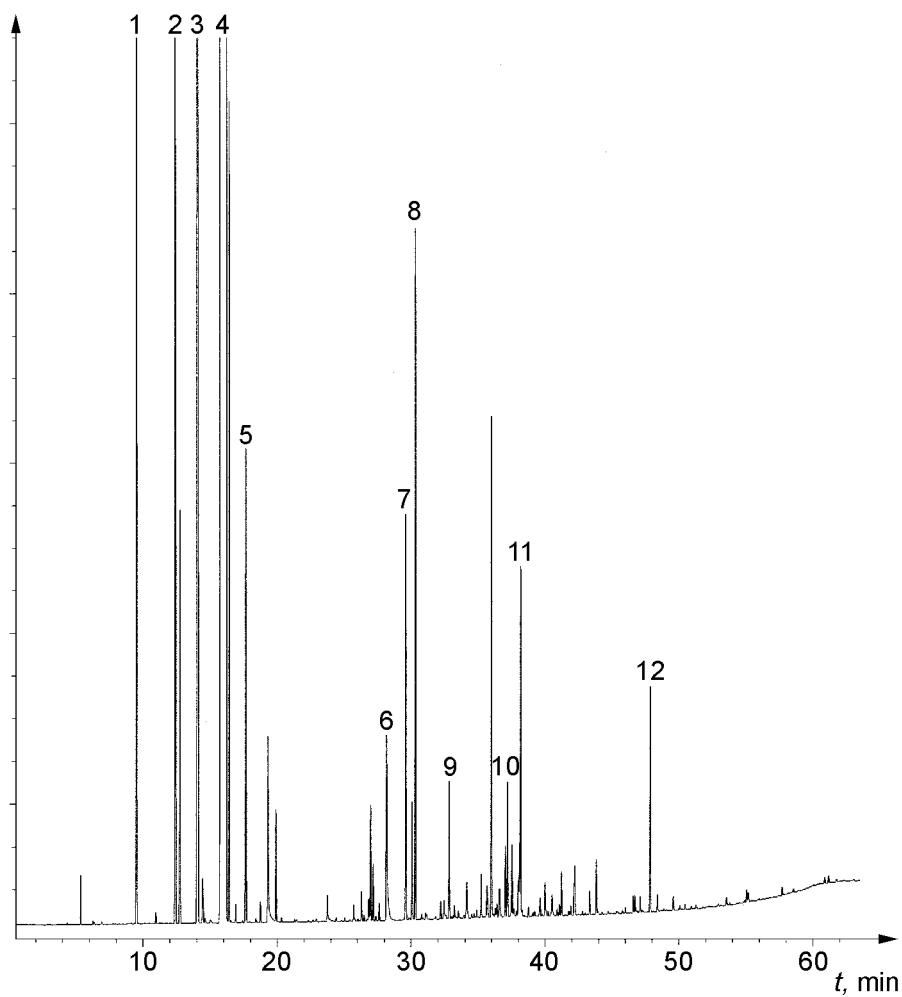
#### Peak identification

- 1  $\alpha$ -Pinene
- 2  $\beta$ -Pinene
- 3 Octanal
- 4 Myrcene
- 5 Limonene
- 6 Linalool
- 7 Decanal
- 8 Linalyl acetate
- 9 Geranyl acetate
- 10  $\beta$ -Caryophyllene
- 11 Germacrene D
- 12 *trans*- $\beta$ -Nerolidol

#### Operating conditions

Column: methyl silicone; length 50 m; internal diameter 0,2 mm  
 Stationary phase: poly(dimethyl siloxane) (SP5®)  
 Film thickness: 0,33  $\mu$ m  
 Oven temperature: temperature programming from 60 °C to 240 °C at a rate of 3 °C/min, then isothermal at 240 °C for 30 min  
 Injector temperature: 270 °C  
 Detector temperature: 280 °C  
 Detector: flame ionization type  
 Carrier gas: helium  
 Volume injected: 0,2  $\mu$ l  
 Carrier gas flow rate: 1 ml/min  
 Split ratio: 1/50

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

**Peak identification**

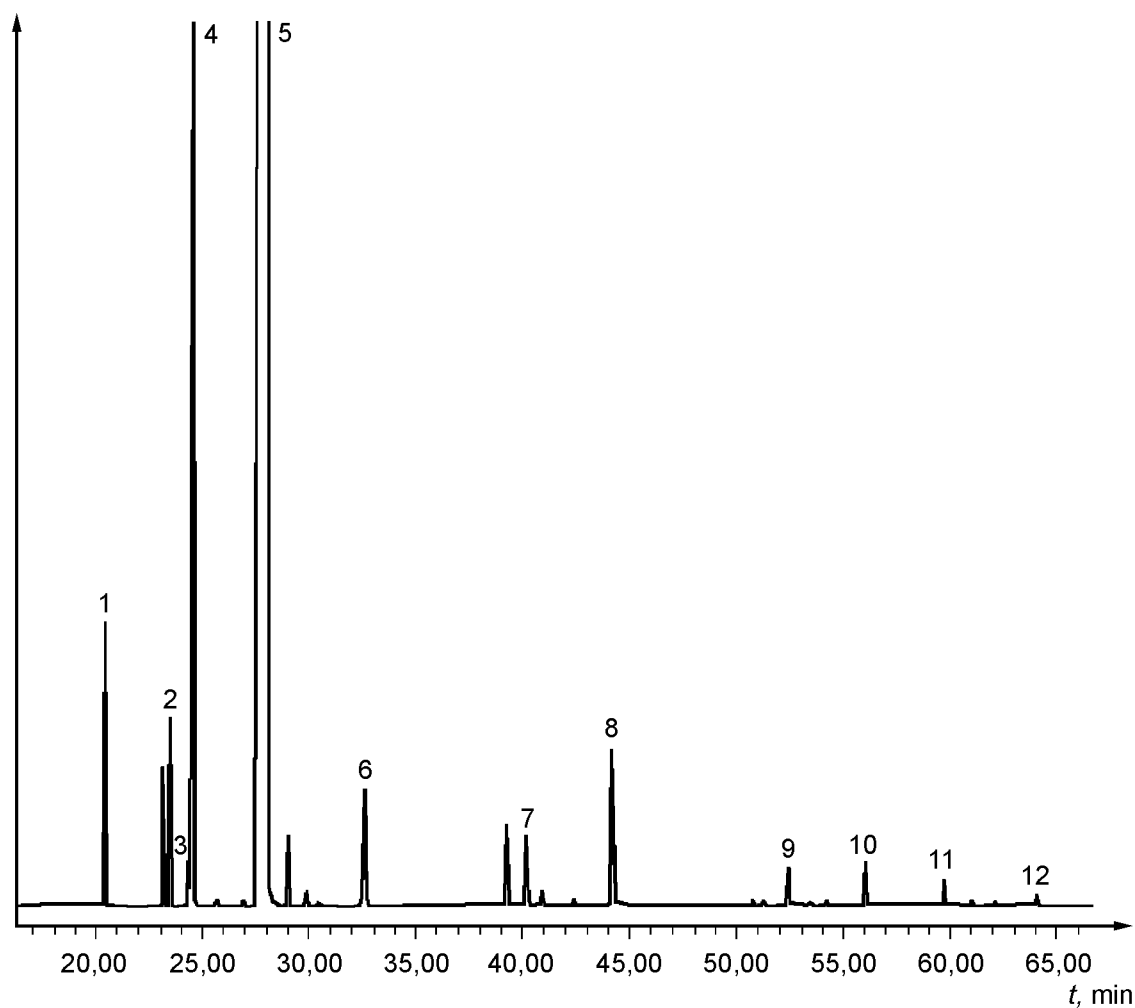
- |    |                                   |
|----|-----------------------------------|
| 1  | $\alpha$ -Pinene                  |
| 2  | $\beta$ -Pinene                   |
| 3  | Myrcene                           |
| 4  | Limonene                          |
| 5  | Octanal                           |
| 6  | Decanal                           |
| 7  | Linalool                          |
| 8  | Linalyl acetate                   |
| 9  | $\beta$ -Caryophyllene            |
| 10 | Germacrene D                      |
| 11 | Geranyl acetate                   |
| 12 | <i>trans</i> - $\beta$ -Nerolidol |

**Operating conditions**

Column: Carbowax; length 50 m; internal diameter 0,2 mm  
 Stationary phase: poly(ethylene glycol) (Suplecowax-10®)  
 Film thickness: 0,4  $\mu$ m  
 Oven temperature: temperature programming from 60 °C to 240 °C at a rate of 3 °C/min, then isothermal at 240 °C for 30 min  
 Injector temperature: 270 °C  
 Detector temperature: 280 °C  
 Detector: flame ionization type  
 Carrier gas: helium  
 Volume injected: 0,2  $\mu$ l  
 Carrier gas flow rate: 1 ml/min  
 Split ratio: 1/50

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

## A.2 Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.), Equatorial type



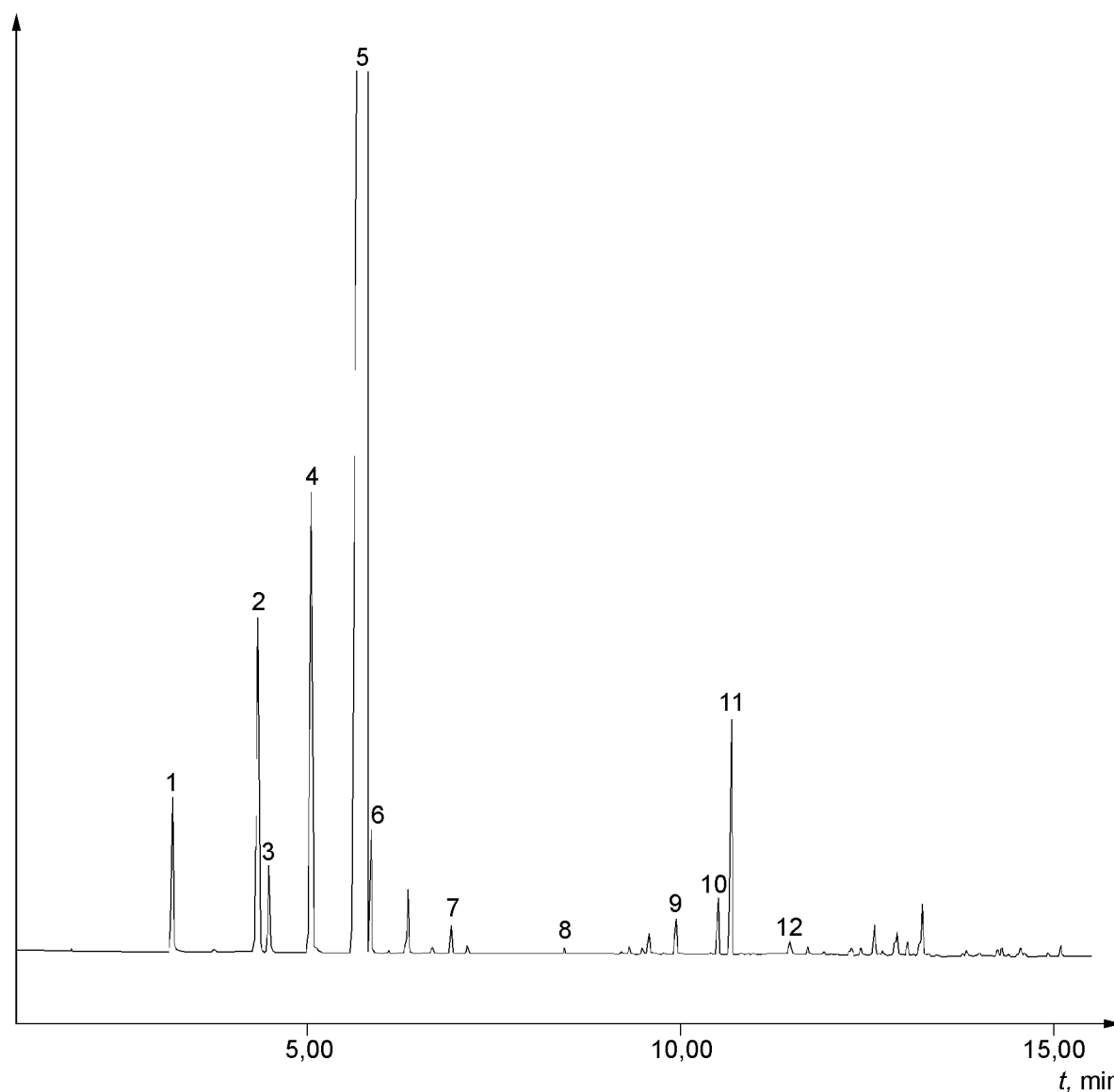
### Peak identification

- |    |                                   |
|----|-----------------------------------|
| 1  | $\alpha$ -Pinene                  |
| 2  | $\beta$ -Pinene                   |
| 3  | Octanal                           |
| 4  | Myrcene                           |
| 5  | Limonene                          |
| 6  | Linalool                          |
| 7  | Decanal                           |
| 8  | Linalyl acetate                   |
| 9  | Geranyl acetate                   |
| 10 | $\beta$ -Caryophyllene            |
| 11 | Germacrene D                      |
| 12 | <i>trans</i> - $\beta$ -Nerolidol |

### Operating conditions

Column: silica capillary; length 60 m; internal diameter 0,25 mm  
 Stationary phase: poly(dimethyl siloxane) (SolGel-1ms)  
 Film thickness: 0,25  $\mu$ m  
 Oven temperature: isothermal at 60 °C for 10 min, then temperature programming from 60 °C to 300 °C at a rate of 2 °C/min, then isothermal at 300 °C for 20 min  
 Injector temperature: 250 °C  
 Detector temperature: 250 °C  
 Detector: mass spectrometer  
 Carrier gas: helium  
 Volume injected: 0,2  $\mu$ l  
 Carrier gas flow rate: 1 ml/min  
 Split ratio: 1/60

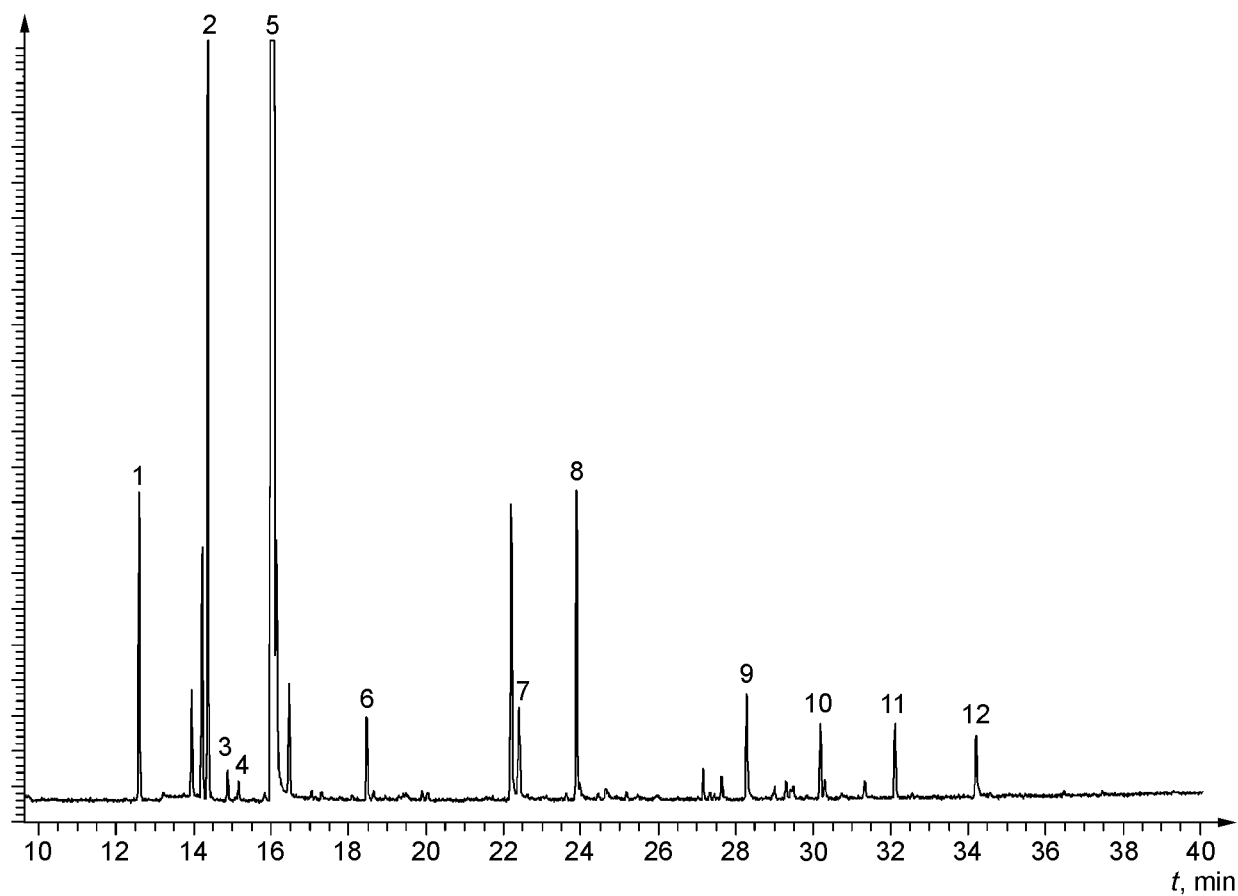
Figure A.3 — Typical chromatogram taken on an apolar column



Peak identification	Operating conditions
1 $\alpha$ -Pinene	Column: silica capillary; length 20 m; internal diameter 0,10 mm
2 $\beta$ -Pinene	Stationary phase: poly(ethylene glycol) (Suplecowax-10®)
3 Sabinene	Film thickness: 0,20 $\mu$ m
4 Myrcene	Oven temperature: isothermal at 50 °C for 1 min, then temperature programming from 50 °C to 200 °C at a rate of 10 °C/min, then isothermal for 13 min
5 Limonene	Injector temperature: 250 °C
6 $\beta$ -Phellandrene	Detector temperature: 250 °C
7 Octanal	Detector: flame ionization type
8 Nonanal	Carrier gas: helium
9 Decanal	Volume injected: 0,2 $\mu$ l
10 Linalool	Carrier gas flow rate: 30 ml/min
11 Linalyl acetate	Split ratio: 1/650
12 $\beta$ -Caryophyllene	

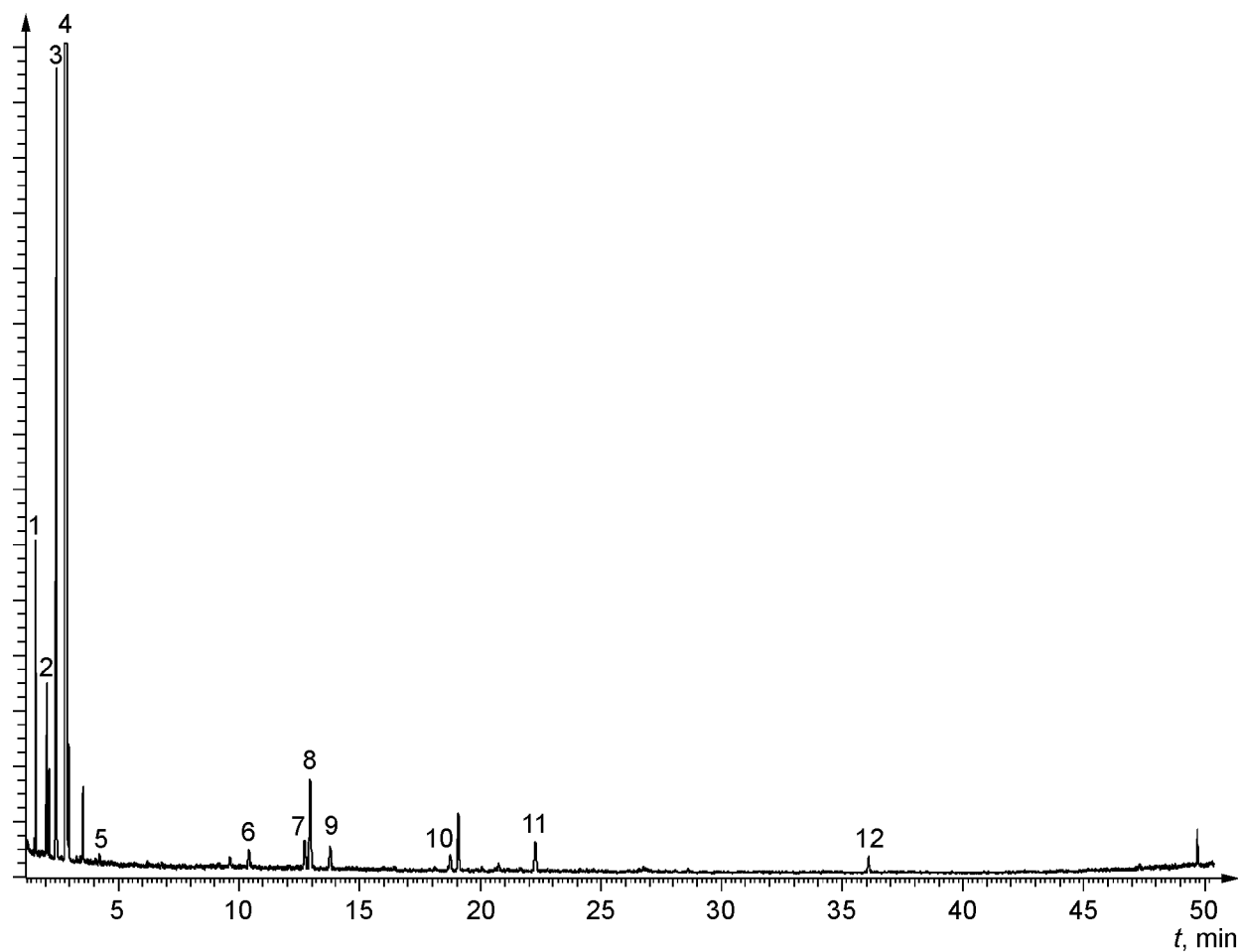
Figure A.4 — Typical chromatogram taken on a polar column

### A.3 Typical chromatograms of the analysis by gas chromatography of the essential oil of bitter orange (*Citrus aurantium* L.), Mediterranean type



Peak identification	Operating conditions
1 $\alpha$ -Pinene	Column: silica capillary; length 60 m; internal diameter 0,25 mm
2 $\beta$ -Pinene	Stationary phase: poly(5 % diphenyl/95 % dimethyl siloxane) (DB5 MS®)
3 Myrcene	Film thickness: 0,25 $\mu$ m
4 Octanal	Oven temperature: isothermal at 40 °C for 5 min, then temperature programming from 40 °C to 220 °C at a rate of 4 °C/min, then isothermal at 220 °C for 5 min
5 Limonene	Injector temperature: 260 °C
6 Linalool	Detector temperature: 280 °C
7 Decanal	Detector: MS type, Full Scan (50-200)
8 Linalyl acetate	Carrier gas: helium
9 Geranyl acetate	Volume injected: 0,3 $\mu$ l
10 $\beta$ -Caryophyllene	Carrier gas flow rate: 1 ml/min
11 Germacrene D	Split ratio: 1/150
12 <i>trans</i> - $\beta$ -Nerolidol	

Figure A.5 — Typical chromatogram taken on an apolar column

**Peak identification**

- |    |                                   |
|----|-----------------------------------|
| 1  | $\alpha$ -Pinene                  |
| 2  | $\beta$ -Pinene                   |
| 3  | Myrcene                           |
| 4  | Limonene                          |
| 5  | Octanal                           |
| 6  | Decanal                           |
| 7  | Linalool                          |
| 8  | Linalyl acetate                   |
| 9  | $\beta$ -Caryophyllene            |
| 10 | Germacrene D                      |
| 11 | Geranyl acetate                   |
| 12 | <i>trans</i> - $\beta$ -Nerolidol |

**Operating conditions**

Column: silica capillary; length 60 m, internal diameter 0,32 mm  
 Stationary phase: poly(ethylene glycol) (Suplecowax-10®)  
 Film thickness: 0,25  $\mu$ m  
 Oven temperature: isothermal at 75 °C for 4 min, then temperature programming from 75 °C to 150 °C at a rate of 2 °C/min, then from 150 °C to 240 °C at a rate of 5 °C/min, then isothermal at 240 °C for 20 min  
 Injector temperature: 225 °C  
 Detector temperature: 225 °C  
 Detector: MS type, Full Scan (50-200)  
 Carrier gas: helium  
 Volume injected: 0,03  $\mu$ l  
 Carrier gas flow rate: 1,5 ml/min  
 Split ratio: 1/100

**Figure A.6 — Typical chromatogram taken on a polar column**

## 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 test methods 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.

#### B.2 Flashpoint of the essential oil of bitter orange

##### B.2.1 Flashpoint of the essential oil of bitter orange, American type

The mean value is +47 °C.

NOTE Obtained with "Pensky Martens" equipment.

##### B.2.2 Flashpoint of the essential oil of bitter orange, Equatorial type

The mean value is +50 °C.

NOTE Obtained with "miniflash-petrotest France" equipment.

##### B.2.3 Flashpoint of the essential oil of bitter orange, Mediterranean type

The mean value is +47 °C.

NOTE Obtained with "Pensky Martens" equipment.



## Bibliography

- [1] ISO/TR 11018, *Essential oils — General guidance on the determination of flashpoint*
- [2] ISO/TR 21092, *Essential oils — Characterization*

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