INTERNATIONAL STANDARD

ISO 11025

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Oil of cassia, Chinese type — Determination of *trans*-cinnamaldehyde content — Gas chromatographic method on capillary columns

Huile essentielle de cannelle type Chine — Détermination de la teneur en trans*-cinnamaldéhyde — Méthode par chromatographie en phase gazeuse sur colonne capillaire*

Reference number
ISO 11025:1998(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 11025 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

Annex A of this International Standard is for information only.

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Introduction

Since the description of methods of analysis by gas chromatography is very long, it is considered useful to establish general methods on the one hand, giving detailed information on all the recurrent parameters, apparatus, products, methods, formulae, etc., and on the other hand standards with short details on the determination of specific constituents on the essential oils, giving only those operating conditions specific to the pertinent determination.

This is the case with the present International Standard, which refers to the general standard ISO 7609 for the general paragraphs.

Oil of cassia, Chinese type — Determination of *trans*-cinnamaldehyde content — Gas chromatographic method on capillary columns

1 Scope

This International Standard specifies a method for the determination of the *trans*-cinnamaldehyde content of the essential oil of cassia, Chinese type (*Cinnamomum aromaticum* Nees, syn. *Cinnamomum cassia* Nees ex Blume).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. As the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 356:1996, *Essential oils — Preparation of test samples.*

ISO 7609:1985, *Essential oils — Analysis by gas chromatography on capillary columns — General method.*

3 Principle

Analysis by gas chromatography on a capillary column, under specified conditions, of oil of cassia, Chinese type. Determination of the *trans*-cinnamaldehyde content using the internal standard method.

4 Reagents and products

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade.

4.1 Reference substance

trans-Cinnamaldehyde, of purity at least 99 %, determined by chromatography under the test conditions.

4.2 Internal standard

Tridecane, of purity at least 99 %, determined by chromatography under the test conditions.

5 Apparatus

5.1 Chromatograph, recorder and electronic integrator

See ISO 7609.

5.2 Column

Length:	25 m to 50 m
Internal diameter:	0,2 mm to 0,3 mm
Stationary phase:	polydimethylsiloxane (OV 101) or polyethylene glycol 20 000 (carbowax 20 M)

5.3 Detector

Flame ionization type.

6 Preparation of test sample

See ISO 356.

7 Operating conditions

7.1 Temperatures

Oven temperature: For example, in the columns, linear temperature programming from 100 °C to 200 °C at a rate of 3 °C/min, or 60 °C to 200 °C at a rate of 1,5 °C/min, then kept constant at 200 °C for 15 min.

Injector temperature: For example, 230 °C to 240 °C.

Detector temperature: For example, 230 °C to 240 °C.

7.2 Carrier gas flow rate and auxiliary gases flow rate

See ISO 7609.

8 Column performance

8.1 Chemical inertness test

Carry out the test as specified in ISO 7609.

8.2 Column efficiency

Determine the column efficiency as specified in ISO 7609.

9 Determination of retention indexes

See ISO 7609.

10 Determination

10.1 Determination of response factor

Determine the response factor as specified in ISO 7609.

10.2 Internal standard method

Carry out the determination of the *trans*-cinnamaldehyde content of the essential oil by the internal standard method specified in ISO 7609, using tridecane (4.2) as internal standard.

11 Expression of results

11.1 Calculation

See ISO 7609.

NOTE — Typical chromatograms are given, for information only, in annex A.

11.2 Repeatability

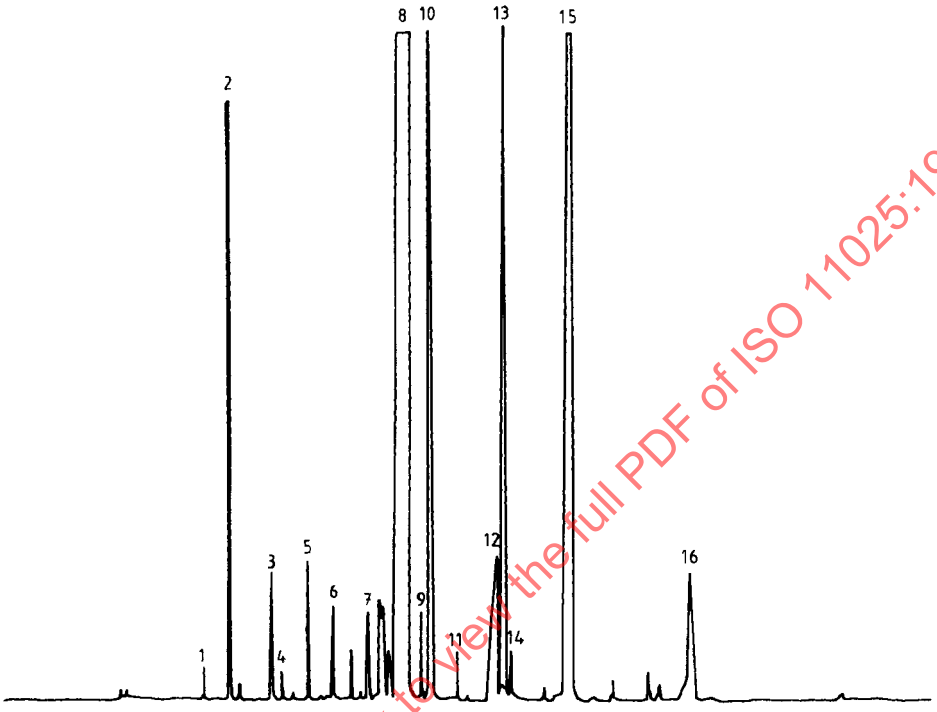
See ISO 7609.

12 Test report

See ISO 7609.

Annex A
(informative)

Typical chromatograms for determination of the *trans*-cinnamaldehyde content in oil of cassia, Chinese type



Peak identification

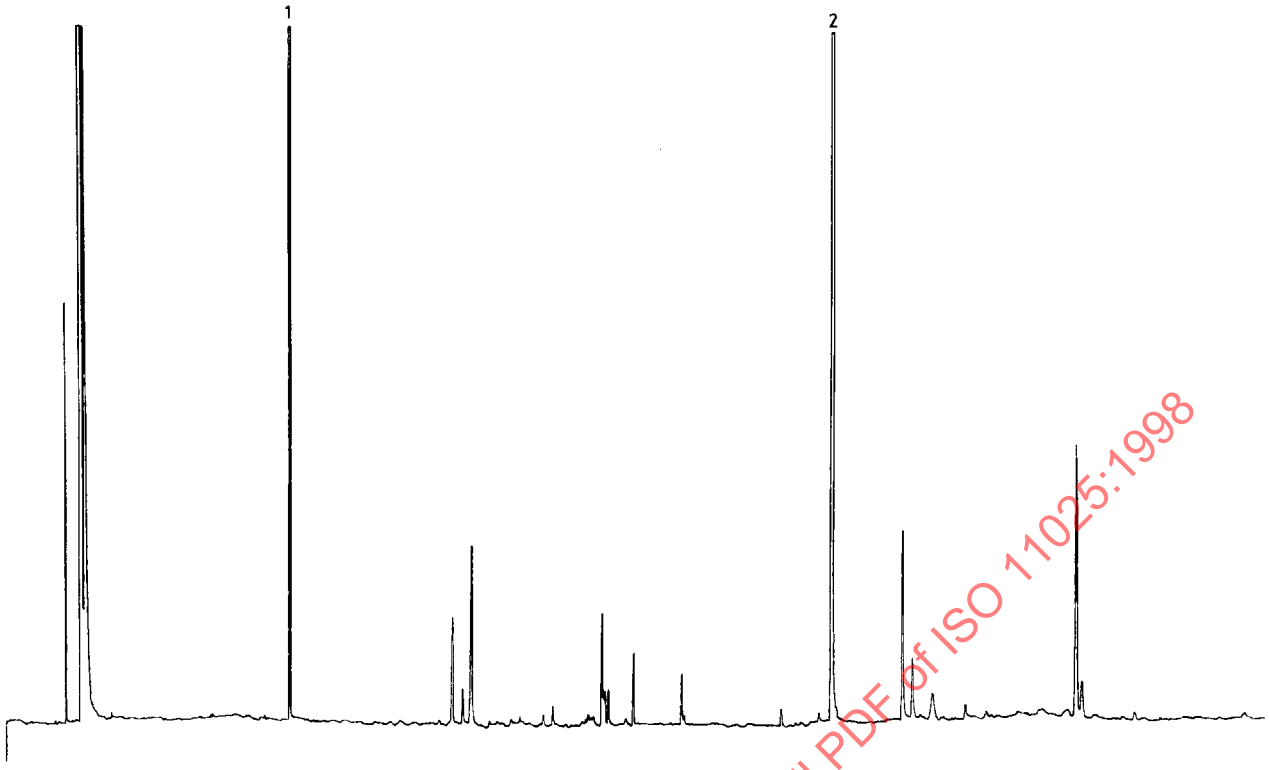
1 Styrene
2 Benzaldehyde
3 Salicylic aldehyde
4 Acetophenone
5 Phenethyl alcohol
6 Dihydrocinnamaldehyde
7 *cis*-Cinnamaldehyde
8 *trans*-Cinnamaldehyde
9 *trans*-Cinnamyl alcohol
10 Tridecane (Internal standard)
11 Eugenol
12 Coumarin
13 Cinnamyl acetate
14 *cis-o*-Methoxycinnamaldehyde
15 *trans-o*-Methoxycinnamaldehyde
16 *o*-Methoxycinnamyl acetate

Operating conditions

Column: fused silica capillary, length 50 m, internal diameter 0,20 mm
Stationary phase: polydimethylsiloxane (OV 101)
Oven temperature: from 100 °C to 200 °C, at rate of 3 °C/min
Injector temperature: 230 °C
Detector temperature: 230 °C
Volume injected: 0,2 µl
Detector: flame ionization
Carrier gas: nitrogen
Carrier gas pressure: 0,4 bar
Split ratio: 1/100

NOTE — The *trans*-cinnamaldehyde peak should not hide any other peak greater than 0,25 %

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



Peak identification

1 Tridecane (internal standard)
2 *trans*-Cinnamaldehyde

Operating conditions

Column: fused silica capillary, length 60 m, internal diameter 0,25 mm
Stationary phase: polyethylene glycol 20 000 (carbowax 20 M)
Oven temperature: from 60 °C to 200 °C at a rate of 1,5 °C/min, then constant at 200 °C for 15 min
Injector temperature: 240 °C
Detector temperature: 240 °C
Volume injected: 0,3 µl
Detector: flame ionization
Carrier gas: hydrogen
Carrier gas pressure: 0,4 bar
Split ratio: 1/100

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