
International Standard



7611

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Oils of lemon and petitgrain citronnier, and oil of lime obtained by a mechanical process — Determination of citral (neral + geranial) content — Gas chromatographic method on capillary columns

iTeh STANDARD PREVIEW

Huiles essentielles de citron et de petitgrain citronnier et huile essentielle de lime obtenue par des procédés mécaniques — Détermination de la teneur en citral (néral + géranial) — Méthode par chromatographie en phase gazeuse sur colonne capillaire

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[ISO 7611:1985](https://standards.iteh.ai/catalog/standards/sist/7399ea1a-0f06-41d9-bbbe-a8a0b02dd2af/iso-7611-1985)

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Descriptors : essential oils, lemon, petitgrain, lime (fruit), chemical analysis, determination of content, gas chromatography.

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7611 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Oils of lemon and petitgrain citronnier, and oil of lime obtained by a mechanical process — Determination of citral (neral + geranial) content — Gas chromatographic method on capillary columns

0 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 in 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.

1 Scope and field of application

This International Standard specifies a gas chromatographic capillary column method for the determination of the citral (neral + geranial) content of oils of lemon and petitgrain citronnier (*Citrus limon* Linnaeus N. L. Burman), and oil of lime obtained by a mechanical process [*Citrus aurantifolia* (Christman) Swingle and *Citrus latifolia* (Tanaka)].

2 References

ISO 356, *Essential oils — Preparation of test sample*.

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

3 Principle

Analysis by gas chromatography on capillary columns, under specified conditions, of small quantities of oils of lemon, petitgrain citronnier or lime. Determination of the citral (neral + geranial) content using the internal standard method.

4 Reagents and products

4.1 Reference substance: mixture of neral and geranial (1 + 2), of purity at least 99 %, determined by chromatography under the test conditions.

4.2 Internal standard: Choose from the following freshly distilled products: acetophenone, hexadecane, methyl nonanoate, methyl dodecanoate, or nonadecane, of purity at least 99 %, determined by chromatography under the test conditions.

The internal standard chosen shall elute as near as possible to the constituents to be determined and shall not superimpose on the peaks of any of the constituents of the essential oils.

5 Apparatus

5.1 Chromatograph, recorder and electronic integrator.

See ISO 7609.

5.2 Column, of length at least 25 to 100 m and internal diameter from 0,2 to 0,5 mm. Stationary phase: polyethylene glycol 20 000.

5.3 Detector, flame ionization type.

6 Preparation of test sample

See ISO 356.

7 Operating conditions

7.1 Temperatures

- Oven:
linear temperature programming from 80 to 180 °C at a rate of 2 °C/min.
- Injection system:
about 190 to 200 °C.
- Detector:
200 to 250 °C, maximum.

7.2 Carrier gas and auxiliary gases flow rates

See ISO 7609.

8 Column performance

8.1 Chemical inertness test

Carry out the test as specified in ISO 7609.

Ensure that the peaks of neral and geranial are not deformed.

8.2 Column efficiency

Determine the column efficiency as specified in ISO 7609.

9 Determination of retention indexes

See ISO 7609.

10 Determination

For the calibration and the determination, assume that neral and geranial have the same response factor K relative to the internal standard.

For calibration, see ISO 7609.

Calculate the total of the peak area of neral and geranial for the calibration and the determination.

10.1 Determination of response factor

Determine the response factor as specified in ISO 7609, using the mixture of neral and geranial (4.1) as the reference substance and one of the internal standards specified in 4.2.

In this case, for the calculation of the response factor K , A_R is the sum of the areas of the peaks for neral and geranial.

10.2 Internal standard method

Carry out the determination of the citral (neral and geranial) content of the essential oil by the method specified in ISO 7609.

11 Expression of results

See ISO 7609.

In this case, A_X is the sum of the areas of the peaks for neral and geranial.

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

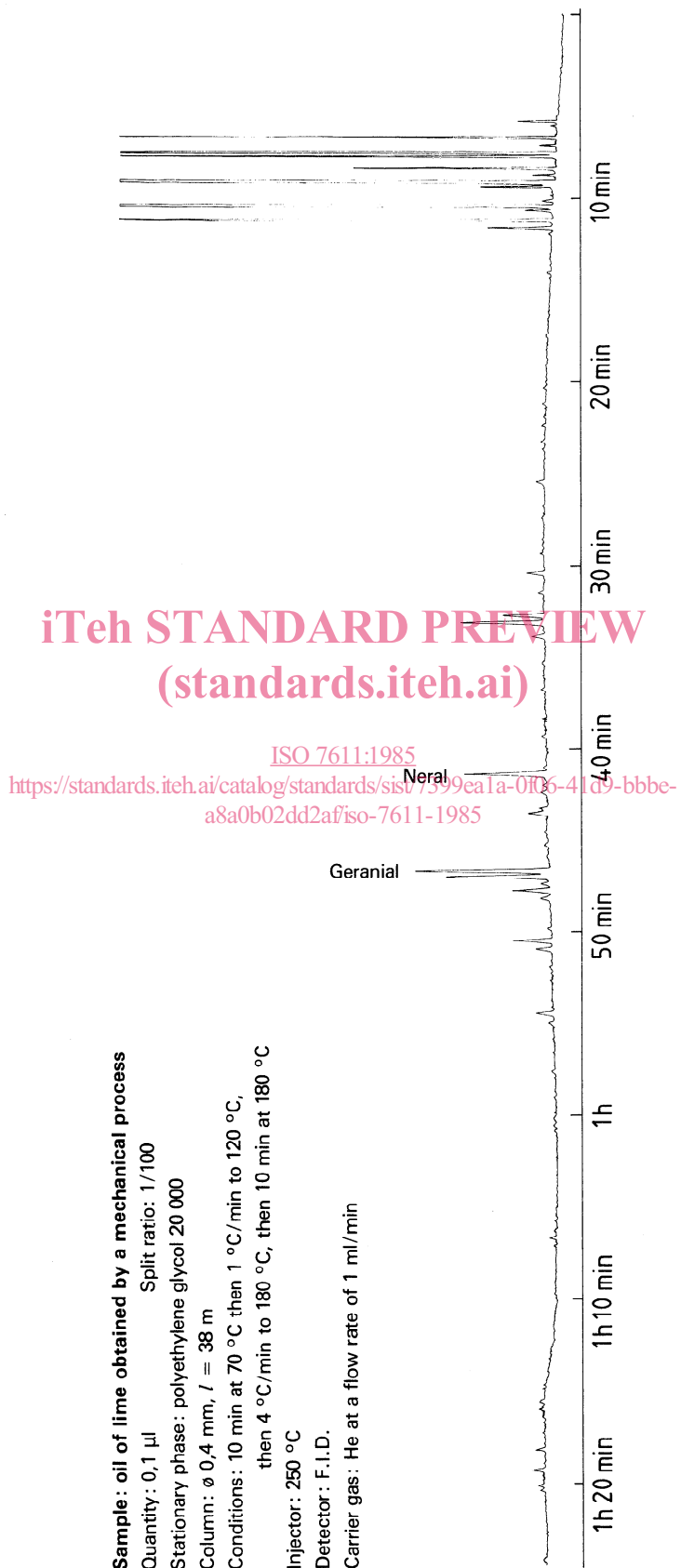
12 Test report

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See ISO 7609.
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Annex

Typical chromatograms

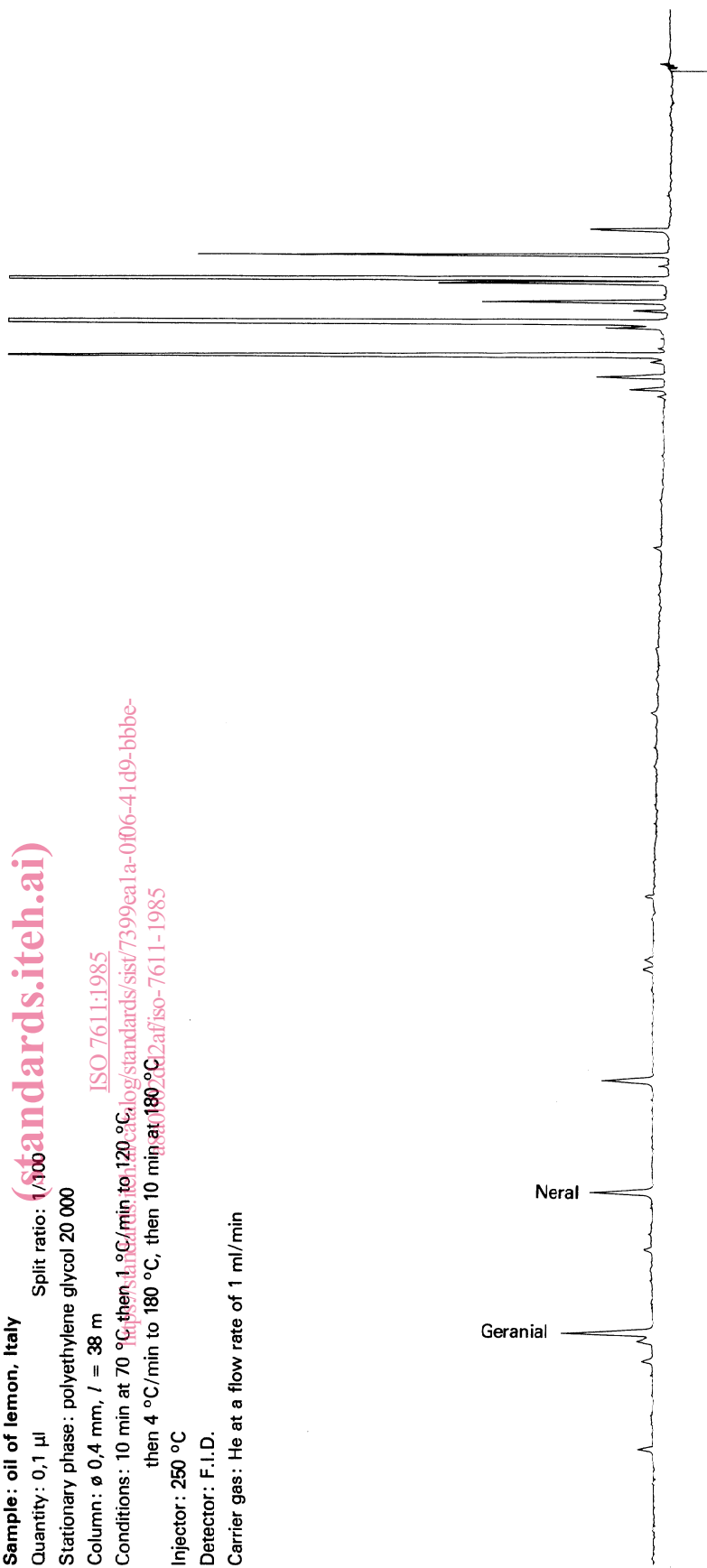
(This annex does not form an integral part of the Standard.)



Sample: oil of lime obtained by a mechanical process
Quantity: 0,1 µl **Split ratio:** 1/100
Stationary phase: polyethylene glycol 20 000
Column: ø 0,4 mm, l = 38 m
Conditions: 10 min at 70 °C then 1 °C/min to 120 °C,
 then 4 °C/min to 180 °C, then 10 min at 180 °C
Injector: 250 °C
Detector: F.I.D.
Carrier gas: He at a flow rate of 1 ml/min

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Sample: oil of lemon, Italy
Quantity: 0,1 µl
Stationary phase: polyethylene glycol 20 000
Column: ø 0,4 mm, l = 38 m
Conditions: 10 min at 70 °C, then 1 °C/min to 120 °C, then 10 min at 120 °C, then 4 °C/min to 180 °C, then 10 min at 180 °C, then 4 °C/min to 220 °C, then 10 min at 220 °C, then 4 °C/min to 250 °C
Injector: 250 °C
Detector: F.I.D.
Carrier gas: He at a flow rate of 1 ml/min



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Sample: oil of petitgrain citromnier, Italy <https://standards.iteh.ai/catalog/standards/sist/7399ca1a-0f06-41d9-bbbe-a8a0b02d0222/af/iso-7611-1985>

Quantity: 0,1 µl Split ratio: 1/100

Stationary phase: polyethylene glycol 20 000

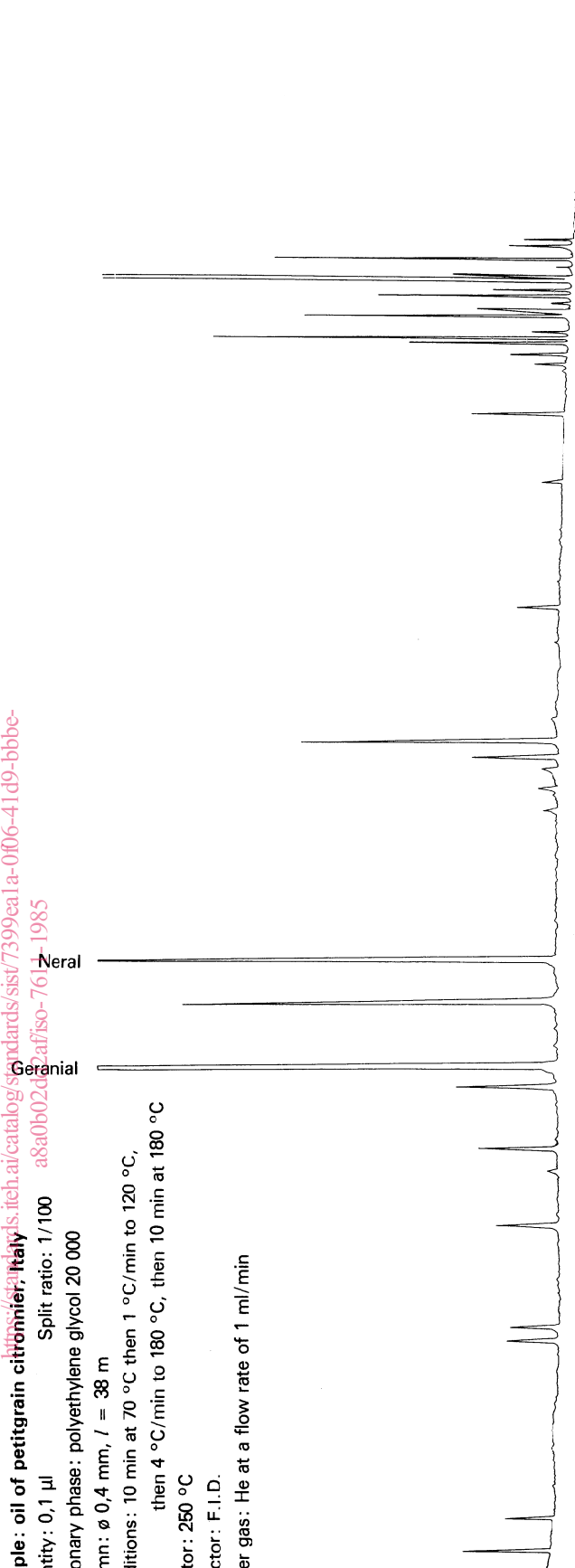
Column: ø 0,4 mm, l = 38 m

Conditions: 10 min at 70 °C then 1 °C/min to 120 °C,
then 4 °C/min to 180 °C, then 10 min at 180 °C

Injector: 250 °C

Detector: F.I.D.

Carrier gas: He at a flow rate of 1 ml/min



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