International Standard 7610

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

Oil of sandalwood — Determination of santalols content (in the form of their trimethylsilyl derivative) — Gas chromatographic method on capillary columns

Huile essentielle de bois de santal — Détermination de la teneur en santalols (sous forme de leur dérivé triméthylsilylé) — Méthode par chromatographie en phase gazeuse sur colonne capillaire

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Foreword

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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 7610 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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Oil of sandalwood — Determination of santalols content (in the form of their trimethylsilyl derivative) — 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.

These short-version standards will either refer to this International Standard for gas chromatographic analyses on capillary columns or to ISO 7359 for analyses on packed columns.

1 Scope and field of application

This International Standard specifies a gas chromatographic capillary column method for the determination of the santalols content (after their transformation into the corresponding trimethylsilyl ether) of oil of sandalwood (*Santalum album* Linnaeus).

2 References

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

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

3 Principle

Analysis by gas chromatography on capillary columns of a small quantity of oil of sandalwood. Determination of the santalols content using the internal standard method after their transformation into the corresponding trimethylsilyl ethers.

4 Reagents and products

All reagents and products shall be anhydrous. Other silylation agents may be used if necessary.

4.1 Reference substances: mixture of α-santalol and β-santalol, freshly distilled, of purity at least 95 % (sum of percentages of α- and β-santalols), determined by chromatography under the test conditions.

4.2 Hexamethyldisilazane, of analytical quality.

4.3 Trimethylchlorosilane, of analytical reagent grade.

4.4 Pyridine, of analytical reagent grade.

4.5 Internal standard: ethyl hexadecanoate, freshly distilled, 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, of length at least 25 m and internal diameter from 0,2 to 0,5 mm. Stationary phase: polyethylene glycol 20 000.

5.3 Detector, flame ionization type.

NOTE — It is important to check frequently the cleanliness of the detector (deposition of silica).

5.4 Conical flasks, of capacity 5 ml, with a ground stopper.

6 Preparation of test sample

See ISO 356.

The prepared test sample shall be silylated before injection, as follows.

6.1 Preparation of silylated test sample for determination of response factor

Weigh, to the nearest 0,001 g, about 100 mg of the mixture of α-santalol and β-santalol (4.1) and 50 mg of the ethyl hexadecanoate (4.5) into a conical flask (5.4). Dissolve in 1 ml of the pyridine (4.4), and add about 0,2 ml of the hexamethyldisilazane (4.2) and about 0,1 ml of the trimethylchlorosilane (4.3). Stopper the flask and shake for 5 min. Allow to stand for at least 15 min.

6.2 Preparation of test sample for determination proper

Weigh, to the nearest 0,001 g, about 100 mg of the sample and 50 mg of the ethyl hexadecanoate (4.5) into a conical flask (5.4). Dissolve in about 1 ml of the pyridine (4.4), and add about 0,2 ml of the hexamethyldisilazane (4.2) and about 0,1 ml of the trimethylchlorosilane (4.3). Stopper the flask and shake for 5 min. Allow to stand for at least 15 min.

7 Operating conditions

7.1 Temperatures

— Oven:

linear temperature programming from 100 to 180 °C at a rate of 2 °C/min.

— Injection system:

200 °C minimum.

— Detector:

about 250 °C.

7.2 Carrier gas and auxiliary gases flow rates

See ISO 7609.

8 Column performance

8.1 Chemical inertness test

Verify the complete separation of the α- and β-isomers of silylated santalol.

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 Methods of determination

For the calibration and the determination, assume that α- and β-santalol have the same response factor K relative to the internal standard.

Use the test samples prepared as described in 6.1 and 6.2 for the determination of the response factor and for the determination proper.

10.1 Determination of response factor

Determine the response factor as specified in ISO 7609, using the mixture of α- and β-santalols (4.1) as the reference substance and the ethyl hexadecanoate (4.5) as the internal standard.

In this case, for calculation of the response factor K, A_r is the sum of the areas of the peaks for α- and β-santalols.

10.2 Internal standard method

Carry out the determination of the santalols content of the essential oil by the method specified in ISO 7609.

Taking into consideration the dilution of the test samples in the pyridine, the quantities injected into the chromatograph should be at least 20 times the normal quantity.

11 Expression of results

See ISO 7609. In this case, A_x is the sum of the areas of the peaks for α- and β-santalols.

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

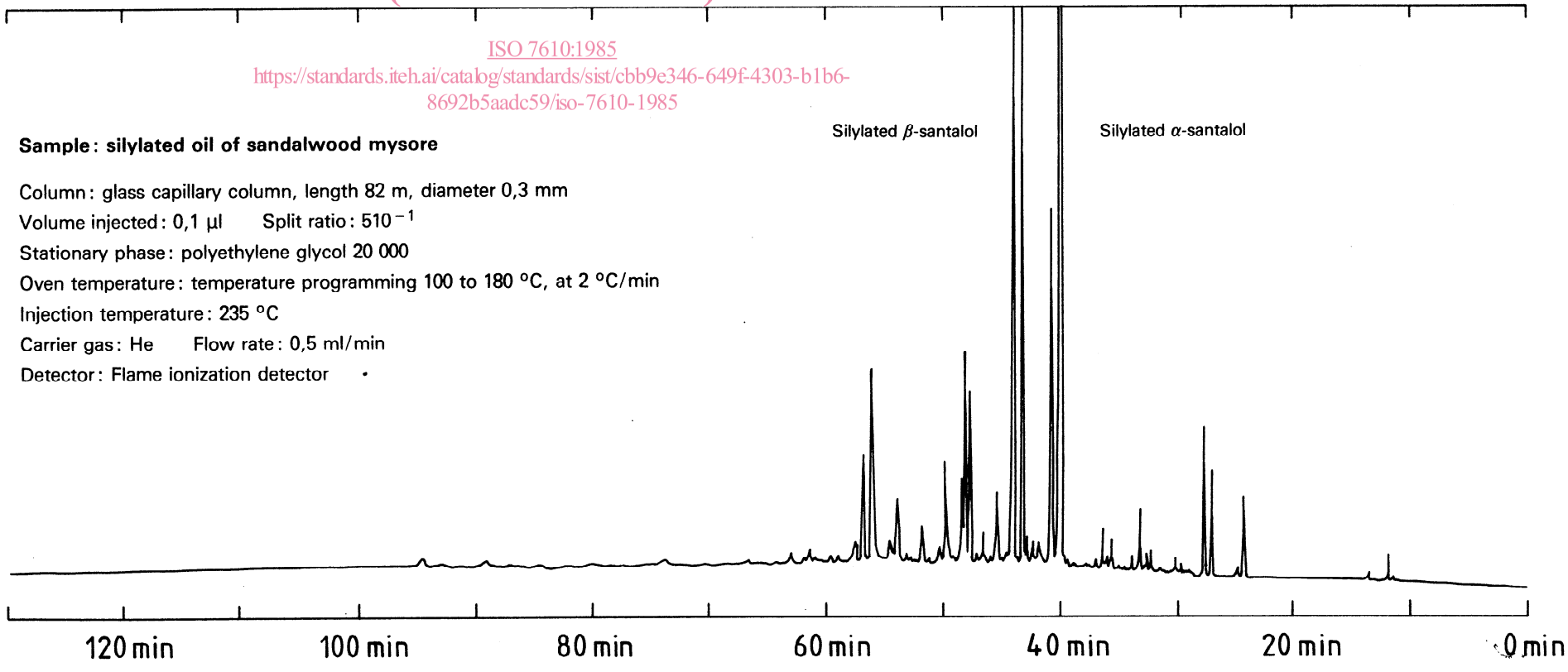
12 Test report

See ISO 7609.

Annex

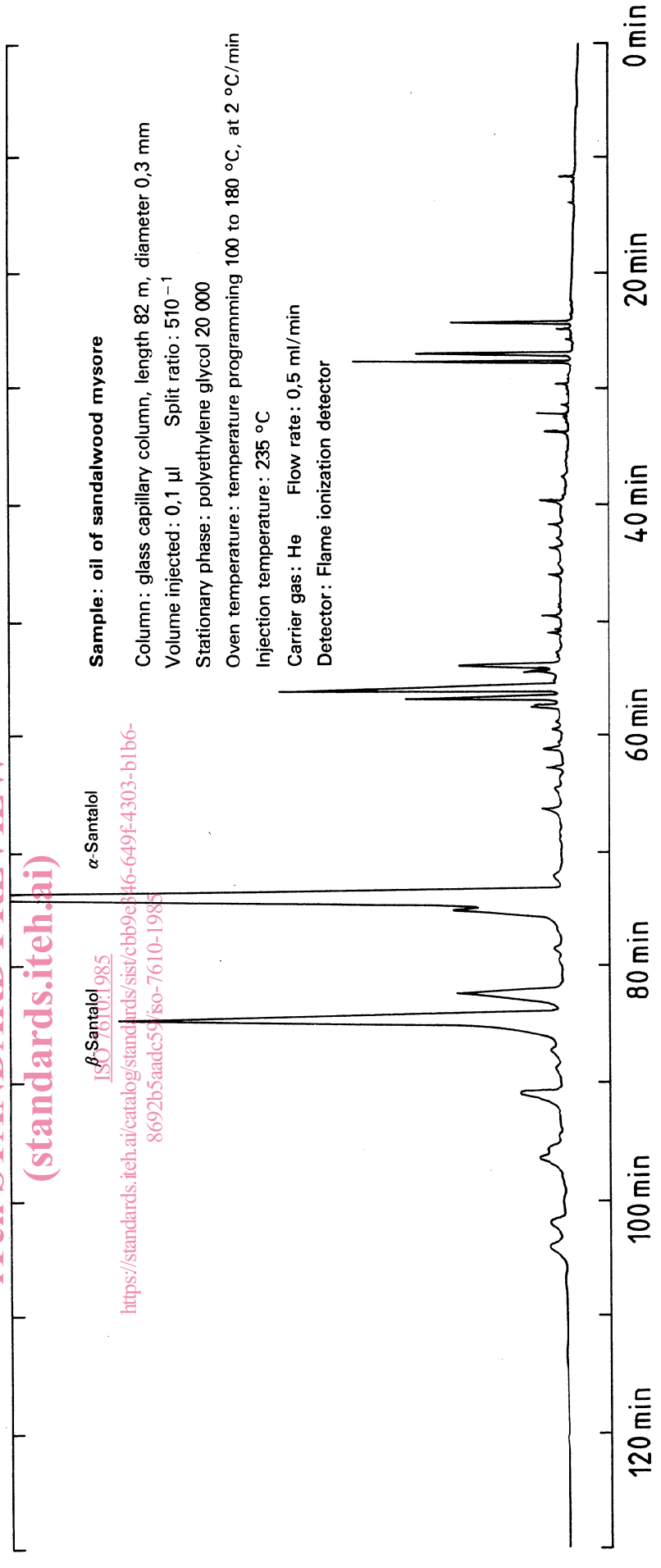
Typical chromatograms

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



Sample: silylated oil of sandalwood mysore

Column: glass capillary column, length 82 m, diameter 0,3 mm
Volume injected: 0,1 µl Split ratio: 510^{-1}
Stationary phase: polyethylene glycol 20 000
Oven temperature: temperature programming 100 to 180 °C, at 2 °C/min
Injection temperature: 235 °C
Carrier gas: He Flow rate: 0,5 ml/min
Detector: Flame ionization detector



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