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Essential oils — Analysis by gas chromatography on chiral capillary columns — General method

Huiles essentielles — Analyse par chromatographie en phase gazeuse sur colonne capillaire chirale — Méthode générale

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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 22972 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

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Introduction

Since the description of gas chromatographic analysis methods is long, it has been considered worthwhile to draw up general standards giving detailed information on all the recurrent parameters (apparatus, products, methods, calculation formulae, etc.), and then short-version standards (abridged analysis sheets) relating to the determination of the enantiomeric excess or distribution of chiral compounds present in essential oils.

This is the case with this International Standard, which makes reference to ISO 7609 for the general clauses.

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Essential oils — Analysis by gas chromatography on chiral capillary columns — General method

1 Scope

This International Standard specifies a general method for the analysis of essential oils by gas chromatography on capillary chiral columns, for the purpose of determining the specific enantiomeric excess or distribution of the chiral compounds contained in the essential oils.

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 356, *Essential oils — Preparation of test samples*

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

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3 Principle

The essentail oil is analysed by gas chromatography, under specified analytical conditions, on a column of appropriate diameter and length, the inside wall of this column having been previously impregnated, either directly with a bonded chiral stationary phase or with a support of this stationary phase.

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The different chiral constituents of interest in the essential oil are indentified with respect to standard substances and, if necessary, by measurement of their retention index and determination of the relative proportions of the enantiomers of a constituent.

4 Reagents and products

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

4.1 Gas.

4.1.1 Carrier gas: hydrogen, helium or nitrogen, or any other gas, depending on the type of detector being used.

If a detector requiring a carrier gas other than those mentioned is used, this should be stated in the test report.

4.1.2 Detector supply gas: all suitable gases, depending on the detector being used:

— for a flame ionization detector, air and hydrogen of high purity;

— for a mass detector, helium of high purity.

4.2 Reference substance, corresponding to the pure and mixed enantiomers to be detected.

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5 Apparatus

5.1 Chromatograph, equipped with an injection system, an appropriate detector and a temperature programmer.

The injection and detection systems shall be fitted with devices for independent control of their respective temperatures.

5.2 Capillary column, made of an inert material, having an inner diameter $\leqslant$ 0,53 mm and a length between 10 m and 60 m.

NOTE Common chiral stationary phases are based on substituted β-cyclodextrins (e.g. 1,3,6-permethylated β-cyclodextrin at 10 % in OV 101®).

5.3 Recorder and electronic integrator.

6 Preparation of the test sample

See ISO 356.

The test sample may have to undergo the following special preparation:

— selective transfer of the relevant constituent(s) (e.g. in-line two-dimensional chromatography or trapping);

— derivatization prior to injection.

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If it is the case, this will be mentioned in the standard on the relevant essential oil.

7 Operating conditions

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7.1 Oven temperature

The oven temperature shall be regulated in such a way as to obtain the required efficiency (see 8.1).

7.2 Carrier gas flow rate

The flow rate shall be regulated in such a way as to obtain the required efficiency (see 8.1).

7.3 Flow rate of detector supply gases

Refer to the appliance manufacturer's instructions in order to obtain the optimum response from the detector.

8 Column performance

8.1 Column efficiency

Determine the column efficiency on the less-retained enantiomer of the selected reference substance (see 4.2), according to the method described in ISO 7609.

The efficiency shall be at least 25 000 theoretical plates.

NOTE When a two-dimensional chromatographic system is used, the temperature programming conditions can be optimized for each component of interest (or chemical family), so as to obtain a satisfactory resolution with a reduced analysis time. In this case, the efficacy may be lower than 25 000 theoretical plates.

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8.2 Resolution and separation

Determine the resolution by injecting a quantity of the selected reference substance (see 4.2) under the test conditions. Carry out the calculations in accordance with ISO 7609:1985, 8.3.1 and 8.3.2.

The injection of the reference substance under analysis conditions shall not lead to any deterioration of the enantiomers or to any variation in their ratio.

9 Determination of the retention indexes

See ISO 7609.

NOTE When a two-dimensional chromatographic system is used, the determination of the retention indexes is optional if the conditions of elution on both chromatographs and the resolution of the enantiomer are similar to those of the reference substances, within the random variations.

10 Determination of the enantiomeric excess or distribution

Use the same temperature and flow rate conditions as those adopted for the column efficiency test (see 8.1). After stabilization of the column temperature, inject an appropriate quantity of the test portion.

On the obtained essential oil chromatogram, verify, using the indications provided by the integrator, compliance with the limits set out in the standards specific to the relevant essential oil.

Particularly in the case of use of a mono-dimensional chromatographic system, it is recommended to verify by all appropriate means (e.g. mass detector) the absence of co-elution for the substances being sought.

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11 Expression of results

11.1 General

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Express the results either in enantiomeric excess or in enantiomeric distribution in accordance with the requirements of the standard specific to the relevant essential oil.

11.2 Enantiomeric excess: E

Determine the enantiomeric excess, E, from the area of each of the enantiomer peaks:

$$E_x = \frac{(A_{\max} - A_{\min})}{(A_{\max} + A_{\min})} \times 100$$

where

x = (R) if $A_{\max}$ is for enantiomer (R);

x = (S) if $A_{\max}$ is for enantiomer (S);

$A_{\max}$ is the peak area presenting the largest surface;

$A_{\min}$ is the peak area presenting the smallest surface.

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11.3 Enantiomeric distribution

$$(\mathrm{R})\,\% = \frac{A(\mathrm{R})}{A(\mathrm{R}) + A(\mathrm{S})} \times 100$$

$$(\mathrm{S})\,\% = \frac{A(\mathrm{S})}{A(\mathrm{R}) + A(\mathrm{S})} \times 100$$

where

$A(\mathrm{R})$ is the enantiomer (R) peak area;

$A(\mathrm{S})$ is the enantiomer (S) peak area.

In the event of the enantiomeric excess or distribution being respectively greater than 95 % or 97,5 %, it is recommended to pay particular attention to the choice of the integration parameters in order to minimize the error on the quantification of the least abundant enantiomer. The latter will be quantified, as far as possible, under acceptable chromatographic resolution conditions.

12 Test report

See ISO 7609.

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