INTERNATIONAL STANDARD

ISO 11024-2

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Essential oils — General guidance on chromatographic profiles —

Part 2:
Utilization of chromatographic profiles of samples of essential oils

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Huiles essentielles — Directives générales concernant les profils chromatographiques —

Partie 2: Utilisation des profils chromatographiques des échantillons d'huiles essentielles

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

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International Standard ISO 11024-2 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

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International Standard ISO 11024 consists of the following parts, under the general title *Essential oils — General guidance on chromatographic profiles*:

— *Part 1: Preparation of chromatographic profiles for presentation in standards*

— *Part 2: Utilization of chromatographic profiles of samples of essential oils*

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Essential oils — General guidance on chromatographic profiles —

Part 2:
Utilization of chromatographic profiles of samples of essential oils

1 Scope

This part of ISO 11024 describes general guidelines on the determination of the compliance of a chromatographic profile of a sample of essential oil under examination with the reference chromatographic profile given in the standard for that oil.

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NOTE Refer also to ISO 11024-1[1)].

2 Normative references

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The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11024. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 11024 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

3 Terms and definitions

For the purposes of this part of ISO 11024, the following terms and definitions apply.

3.1
representative components
components present in all of the samples of the essential oil involved, whether major or minor ones

EXAMPLE Geranyl formate, isomenthone, citronnellal, geraniol, etc. in the essential oil of geranium.

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1) ISO 11024-1, *Essential oils — General guidance on chromatographic profiles — Part 1: Preparation of chromatographic profiles for presentation in standards.*

3.2
characteristic components
one or more representative components, the concentration of which is characteristic for a given essential oil

NOTE The concentration may be nil.

EXAMPLES

Guaia-6,9-diene is present in traces in the Africa geranium and present in higher concentrations in the Bourbon geranium.

10-Epi-gamma-eudesmol is absent in the Bourbon geranium and present in the Africa geranium.

Camphor is present in quantities of less than 0,5 % in lavender.

3.3
typical chromatogram
graphical representation obtained by injection into the chromatograph of a sample of an essential oil considered to be representative of production, together with the operating conditions under which it has been obtained

NOTE The chromatogram is for information only.

3.4
chromatographic profile
list of components selected among the representative and characteristic components of an essential oil, accompanied, for each of them, by concentration limits and, possibly, by the ratios between these concentrations

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4 Principle

A sample of the essential oil under test is analysed by gas chromatography on capillary column.

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Those peaks considered to be representative and characteristic of the essential oil are located on the chromatogram obtained. They are compared with those indicated in the clause "Chromatographic profile" of the standard specific to the essential oil being studied.

Using the results obtained directly from the data-processing system, the compliance of the minimum and maximum concentrations (or of the concentration ratios) of these representative and characteristic components are verified with the limits fixed in the standard specific to the essential oil being studied.

5 Standard matching solution

Following the recommendations of ISO 7609, prepare a standard matching solution by mixing with 1 ml of hexane the reference substances corresponding to the representative and characteristic components indicated in the clause "Chromatographic profile" of the standard concerning the essential oil being studied.

Check that the reference substances are sufficiently pure for chromatography.

6 Apparatus

6.1 Gas chromatograph, provided with split injector, capillary column.

6.2 Flame ionization detector.

6.3 Data-processing system (integrator, calculator, etc.), complying with the indications given in ISO 7609.

Verify the performance of the apparatus using the test described in clause 7.

7 Performance of the apparatus

7.1 General

Set-up the assembly comprising the chromatograph and the data-processing system (6.3) in such a manner that permits the correct resolution and total elution of all volatile components of the essential oil, and the chromatographic profile obtained with the test mixture defined in Table 1 conforms to the chromatographic profile defined by the interlaboratory test (see Table 2).

7.2 Preparation and composition of the standard test mixture

Prepare a standard test mixture as indicated in Table 1.

Table 1 — Composition of the standard test mixture

Chemical name	CAS [a]	EINECS [b]	Minimum purity	Mass fraction %
n-Hexanol	111-27-3	203-852-3	99 %	0,80
α-Pinene	7785-70-8	232-087-8	99 %	5,00
1,8-Cineole (eucalyptol)	470-82-6	207-431-5	99,5 %	50,00
Linalool	78-70-6	201-134-4	99 %	10,00
n-Decanal [c]	112-31-2	203-957-4	98 %	0,20
Linalyl acetate	115-95-7	204-116-4	99 %	25,00
Eugenol	97-53-0	202-589-1	99 %	3,00
β-Caryophyllene	87-44-5	201-746-1	99 %	5,00
Benzyl salicylate	118-58-1	204-262-9	99 %	1,00

[a] Chemical Abstract Service Registration No.

[b] Registration No. of the European Inventory of existing commercially available chemical substances.

[c] Freshly distilled and/or chemically stabilized.

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Check the purity of each of the components by gas chromatography and by the usual physico-chemical methods.

Store the mixture in full sealed bottles, sheltered from the light, and at a temperature between –5° C and +5 °C. Under these conditions, the test mixture may be stored at least a year.

7.3 Procedure

Carry out the chromatographic analysis of the test mixture by injecting the latter under the usual operating conditions for essential oils in practice in the laboratory.

7.4 Results

The results are obtained directly from the data-processing system.

The obtained data, expressed as area percent, shall be within the limits given in table 2.

Table 2 — Chromatographic profile of the standard test mixture

Chemical name	Minimum %	Maximum %
n-Hexanol	0,65	0,75
α-Pinene	5,85	6,25
1,8-Cineole (eucalyptol)	49,0	50,5
Linalool	10,10	10,50
n-Decanal	0,15	0,20
Linalyl acetate	22,80	23,50
Eugenol	2,50	2,75
β-Caryophyllene	5,85	6,30
Benzyl salicylate	0,75	0,95
n-Hexanol/benzyl salicylate ratio	0,75	0,95

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For the peak of the *n*-decanal, the signal-to-noise ratio shall be greater than 100. This ratio may be calculated as follows:

— signal is the height of the peak of decanal;

— noise is half of the difference between the maximum and the minimum signal values in the absence of a peak for 30 s.

8 Preparation of test sample

Comply with the method specified in ISO 356.

9 Location of the representative and characteristic components

Locate on the chromatogram of the essential oil being studied, those peaks which correspond to the representative and characteristic components indicated in the reference chromatographic profile.

This may be carried out in different ways, for example, using one or more of the following methods:

— comparison with a typical chromatogram obtained with a chromatographic column having the same composition;

— use of additions;

— use of retention index;

— coupling with a mass spectrometer.

Alternatively, use a standard matching solution, as follows.

Inject the standard matching solution. Chromatogram A is obtained. Identify the peaks obtained as a function of their order of elution and of their area, which shall correspond approximately to the concentrations used for the preparation of the standard matching solution.

NOTE The orders of elution are given by readings from typical chromatograms obtained using different types of columns.

Inject, under the same procedural conditions, the sample of essential oil whose quality is to be checked. Chromatogram B is obtained.

Using the retention times read on chromatogram A, locate on chromatogram B the peaks corresponding to the representative and characteristic components. All the representative components shall be present in the sample of essential oil being studied.

10 Checking the concentration of the representative and characteristic components

Using the information supplied by the data system corresponding to chromatogram B, method of determination by the peak area normalization method (internal normalization method in accordance with ISO 7609), check that the concentrations (assumed to be equivalent to the peak area percentages taken into consideration) or concentration ratios are included between the minimum and maximum values specified in the clause "Chromatographic profile" of the standard specific to the essential oil being studied.

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11 Expression of results

The chromatographic profile is expressed as a list of the representative and characteristic components found in the sample of essential oil being studied, accompanied by their concentrations which have been assessed by the normalization method. Ratios of these concentrations may also be calculated.

12 Test report

The test report shall specify the method used and the results obtained. It shall also mention all operating conditions not specified in this part of ISO 11024, or regarded as optional, together with details of any incidents which may have influenced the test result.

The test report shall include all information necessary for the complete identification of the sample.