



**International
Standard**

ISO 14714

**Essential oils and aromatic
extracts — Determination of
benzene content**

*Huiles essentielles et extraits aromatiques — Détermination de la
teneur en benzène*

**Second edition
2024-10**

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 14714:1998), which has been technically revised.

The main changes are as follows:

- in the title and scope, the word “residual” has been removed;
- the structure of the document has been changed;
- in the scope, “around 10 ppm” has been replaced by “from 1 to 10 mg/kg”;
- in [6.2](#), the type of the recommended column has been changed;
- in [6.3](#), the title of the subclause has been changed and additional information of the mass spectrometer has been added;
- in [9.1.3](#), precisions have been added for building the calibration curves with Flame ionization detector (FID) or by mass spectrometer detection;
- in [9.2.3](#), precisions have been added for determining benzene content by mass spectrometer detection.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Essential oils and aromatic extracts — Determination of benzene content

1 Scope

This document describes a method for determining the traces of benzene in essential oils and aromatic extracts, using static head space gas chromatography.

It applies to contents from 1 mg/kg to 10 mg/kg in the analysed product.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Essential oils — Analysis by gas chromatography on capillary columns — General method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

m/z

mass-to-charge ratio

dimensionless value formed by dividing the mass of an ion by its charge number in mass spectrometry

4 Principle

The sample is analysed by static headspace gas chromatography on a capillary column, either using a flame ionization detector or detection by means of mass spectrometry.

The benzene content is determined using an external standard calibration method.

5 Reagents and products

5.1 Reference substance: benzene, minimum purity 99 %, determined by gas chromatography.

5.2 Diethyl phthalate, which shall be free from any traces of benzene, as verified under test conditions.

6 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

6.1 Chromatograph, recorder, integrator, in accordance with ISO 7609.

6.2 Column.

It is recommended to use a capillary column with the following characteristics:

- stationary phase: 6 % cyanopropyl/phenyl, 94 % polydimethylsiloxane;
- length: 30 m;
- internal diameter: 0,25 mm;
- film thickness: 1,4 μm .

It is also possible to use other capillary columns with the following characteristics:

- stationary phase: 100 % dimethylpolysiloxane;
- length: 30 m to 60 m;
- internal diameter: 0,25 mm to 0,5 mm.

6.3 Detector, i.e. flame ionization detector or mass spectrometric detector (single quadrupole mass spectrometer in the electron impact mode with conventional 70 eV ionization).

6.4 Headspace sampler, which allows the injection of headspace vapours, in static mode, to the gas chromatograph either with a gas syringe system with leaktightness locks or by an automatic equipment consisting of a loop and valves.

7 Preparation of test sample

The test sample shall be prepared in accordance with ISO 356.

8 Operating conditions

8.1 Chromatographic operating conditions

8.1.1 Injector temperature

The temperature of the injector shall be 150 °C.

8.1.2 Oven temperature

The oven shall have an isothermal value between 40 °C and 60 °C maintained for 15 min, followed by rapid temperature programming to elute any less volatile product.

8.1.3 Temperature of flame ionization detector

The temperature of the flame ionization detector shall be from 200 °C to 250 °C.

8.1.4 Flow rate of carrier gas and auxiliary gases

The flow rate of carrier gas and auxiliary gases shall be in accordance with ISO 7609.

8.1.5 Mass spectrometric detector operating conditions

The mass spectrometer source temperature should be set at 250 °C.

The mass spectrometer detector shall be set on the single ion monitoring (SIM) mode and shall record the three ions with respective mass-to-charge ratios (m/z): 78, 77 and 51.

8.2 Static headspace sampler operating conditions

8.2.1 Thermostatization temperature

The temperature of the thermostatization shall be between 70 °C and 75 °C.

8.2.2 Thermostatization time

The time of thermostatization shall be 30 min minimum.

9 Quantitative analysis method

9.1 External calibration curve

9.1.1 Preparation of standard solutions

Weighing to the nearest 10^{-4} g, prepare a parent solution of benzene in the diethyl phthalate (5.2) to 10 g/kg. By means of successive gravimetric dilutions, prepare a range of standard solutions, with a benzene content between 1 mg/kg and 25 mg/kg, i.e. 25 mg/kg, 10 mg/kg, 5 mg/kg and 1 mg/kg.

9.1.2 Injection of vapour phase of standard solutions

The test specimen is placed in the headspace apparatus flask (6.4) and shall:

- have an identical volume in all cases (during calibration and quantitative analysis);
- be between a tenth and a half of the total volume of the flask.

9.1.3 Graph of the external calibration curve

9.1.3.1 Flame ionization detector (FID)

Record the areas of the benzene peak obtained for each standard solution chromatogram. Plot the curve of the areas as a function of their concentration.

9.1.3.2 Mass spectrometer detector

Record the areas of peak quantifier ion (m/z 78) of the benzene obtained for each standard solution injected. Plot the curve of the areas as a function of their concentration.

9.1.4 Validation of the external calibration curve

Before test specimen analysis, validate the calibration curve by preparing a fresh benzene solution, the content of which is between 1 mg/kg and 25 mg/kg (9.1.1). During the vapour phase, the validation solution shall be injected under the same conditions as during the calibration and shall give a point on the calibration curve.

If not, it is necessary to carry out a complete determination of a new calibration curve, with new weighing and new validation.

9.2 Quantitative analysis

9.2.1 Sample preparation

The volume of the test specimen of the sample in the headspace flask shall be identical to the one used for calibration.

9.2.2 Injection of the sample

Proceed in the same way as described in [9.1.2](#).

9.2.3 Determination of the benzene content

Determine the benzene content using the calibration curve plotted in [9.1](#) to read off the benzene content corresponding to the area of the sample analysis, taking into account any dilution.

If a mass spectrometer is used, the identification of the benzene can be obtained using the quantifier ion m/z 78 and the qualifiers ions m/z 77 and m/z 51, at the retention time used for quantification.

Use of the mass detector is recommended if there are chromatographic coelutions or if the identification of the benzene is required.

If the content is greater than the field of the calibration curve, carry out a dilution in the same solvent and proceed to a new injection and determination.

10 Precision

10.1 Results of the interlaboratory test

An interlaboratory test organized in 1991 with the participation of seven laboratories gave the results shown in [Annex A](#), for information purposes.

10.2 Repeatability

The difference between two individual independent test results obtained using the same method on an identical material subjected to the test in the same laboratory by the same operator using the same apparatus and within a short time interval shall not be greater than 6,5 %.

10.3 Reproducibility

The difference between two individual test results, obtained using the same method on an identical material undergoing the test in different laboratories with different operators using different apparatus shall not be greater than 13,50 %.

11 Test report

The test report shall be prepared in accordance with ISO 7609.