INTERNATIONAL STANDARD

ISO 14714

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Essential oils and aromatic extracts — Determination of residual benzene content

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

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.

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.

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

International Standard ISO 14714 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

Annex A of this International Standard is for information only.

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Essential oils and aromatic extracts — Determination of residual benzene content

1 Scope

This International Standard describes a method for determining the residual traces of benzene in essential oils and aromatic extracts, using static headspace gas chromatography.

It applies to residual contents of around 10×10^{-6} (10 ppm) in the analysed product.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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 Principle

Analysis by gas chromatography of the static headspace on a capillary column, either by flame ionization detector or by detection by means of mass spectrometry.

Determination of residual benzene content using external standard method (by calibration).

4 Reagents

4.1 Reference substance: benzene, of minimum purity 99 %, as determined by gas chromatography.

4.2 Diethyl phthalate, free from any traces of benzene, to be verified under test conditions.

5 Apparatus

5.1 Chromatograph, recorder and integrator

See ISO 7609.

5.2 Capillary column, with the following characteristics:

— length: 30 m to 60 m;

— internal diameter: 0,20 mm to 0,5 mm;

— stationary phase: methyl silicone type is recommended.

5.3 Flame ionization detector or **mass detector**

5.4 Headspace sampling device, for the injection of headspace vapours, comprising either 5.4.1 or 5.4.2.

5.4.1 Gas syringe system (manual), with leaktight locks.

5.4.2 Automatic injector equipment

6 Preparation of test sample

See ISO 356.

7 Operating conditions

7.1 Chromatographic operating conditions

7.1.1 Injector temperature shall be 150 °C.

7.1.2 Oven temperature shall be maintained constant at between 40 °C and 60 °C for 15 min, followed by rapid temperature programming to elute any less volatile product.

7.1.3 Temperature of flame ionization detector shall be between 200 °C and 250 °C.

7.1.4 For flow rate of carrier gas and auxiliary gases, see ISO 7609.

7.2 Operating conditions for headspace sampling device

7.2.1 The sample shall be thermostatted at a temperature of between 70 °C and 75 °C.

7.2.2 This temperature shall be maintained for a minimum of 30 min.

8 Quantitative analysis method

8.1 External calibration curve

8.1.1 Preparation of standard solutions

Weighing to the nearest 0,0001 g, prepare a stock solution of benzene in the diethyl phthalate (4.2) containing a mass fraction of benzene of 1 %. By means of successive gravimetric dilutions, prepare a range of standard

solutions containing a mass fraction of benzene of between 1×10^{-6} and 25×10^{-6} (1 ppm and 25 ppm); i.e. 25×10^{-6}, 10×10^{-6}, 5×10^{-6} and 1×10^{-6} (25 ppm, 10 ppm, 5 ppm and 1 ppm).

8.1.2 Injection of vapour phase of standard solutions

The test specimen is placed in the headspace sampling flask (5.4) and shall be:

— identical in all cases (calibration and quantitative analysis);

— between one-tenth and one-half of the total volume of the flask.

8.1.3 Plotting the external calibration curve

Record the areas of the benzene markers obtained for the successive injections into the headspace of the standard solutions. Plot the curve of the areas as a function of the concentrations.

8.1.4 Validation of the external calibration curve

Before each analysis, validate the calibration curve by preparing, by weighing afresh, a benzene solution containing a mass fraction of between 0 and 25×10^{-6} (25 ppm). Injection of the vapour phase of this validation solution under the same conditions as used for calibration shall give a point which is 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.

8.2 Quantitative analysis

8.2.1 Sample preparation

The test specimen of the sample in the headspace flask shall be identical to the one used for calibration. In the case of contents greater than the field of the calibration curve, carry out a dilution in the same solvent.

8.2.2 Injection of the sample

Proceed in the same way as described in 8.1.2.

8.2.3 Determination of the residual benzene content

Determine the benzene content using the calibration curve plotted in 8.1 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 analytical method is the same. Carry out the calibration and quantitative analysis at least on ion 78, visualizing the spectrum in the chromatographic retention zone of the benzene.

NOTE Use of the mass detector is recommended if there are problems with the separation or identification of the benzene.

9 Accuracy

9.1 Interlaboratory test

An interlaboratory test organized in 1991 with the participation of seven laboratories gave the results shown in annex A, for information purposes.

9.2 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, will in not more than 5 % of cases be greater than 6,5 %.

9.3 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, will in not more than 5 % of cases be greater than 13,50 %.

10 Test report

See ISO 7609.

Annex A
(informative)

Results of interlaboratory tests

Table A.1

Values should be multiplied by 10^{-6} (ppm)

Number of measurements	Laboratories						
	1	2	3	4	5	6[a)]	7
7	9,52	10,20	9,60	10,00	9,20	9,50	10,40
7	9,97	10,30	9,60	11,00	9,90	10,40	10,20
6	10,13	10,30	9,20	10,80	9,30		10,30
6	10,16	10,00	9,20	11,10	9,90		10,10
5	10,58	10,40	8,80	10,40			10,40
5	10,58	10,10	9,20	11,30			9,90
5	9,68	10,60	9,00	11,00			9,60
5	9,54	10,60	8,80	10,00			10,30
4		10,60	8,70	10,50			10,20
2			8,80				9,80
1			9,20				
1			9,20				
54							
Results							
Minimum:	9,52	10,00	8,70	10,00	9,20	9,50	9,60
Maximum:	10,58	10,60	9,60	11,30	9,90	10,40	10,40
Mean:	10,02	10,34	9,11	10,68	9,58	9,95	10,12
Minimum deviation:	0,50	0,34	0,41	0,68	0,38	0,45	0,52
Maximum deviation:	0,56	0,26	0,49	0,62	0,33	0,45	0,28
Maximum relative deviation:	5,59 %	3,33 %	5,40 %	6,35 %	3,92 %	4,52 %	5,14 %
Variance:	0,18	0,05	0,09	0,23	0,14	0,41	0,07
Repeatability standard deviation:	0,42	0,22	0,30	0,48	0,38	0,64	0,27

Minimum:	8,70
Maximum:	11,30
Mean:	9,95
Minimum deviation:	1,25 = 12,55 %
Maximum deviation:	1,35 = 13,58 %
Variance:	0,41
Reproducibility standard deviation:	0,64

a) The values shown are the minima and maxima of the seven measurements.

ICS 71.100.60

Descriptors: oils, essentials oils, chemical analysis, determination of content, chemical residues, benzene, gas chromatography.

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