INTERNATIONAL STANDARD

ISO 709

Second edition
2001-09-01

Essential oils — Determination of ester value

Huiles essentielles — Détermination de l'indice d'ester

Reference number
ISO 709:2001(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
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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 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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 709 was prepared by Technical Committee ISO/TC 54, *Essential oils*.

This second edition cancels and replaces the first edition (ISO 709:1980), which has been technically revised.

Annex A of this International Standard is for information only.

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Essential oils — Determination of ester value

1 Scope

This International Standard specifies a method for the determination of the ester value of an essential oil.

This method is not applicable to essential oils containing lactones or an appreciable proportion of aldehydes.

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 385-1, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 1242, *Essential oils — Determination of acid value*

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
ester value
EV
number of milligrams of potassium hydroxide required to neutralize the acids liberated by the hydrolysis of esters present in 1 g of the essential oil

4 Principle

The esters present in the essential oil are hydrolysed by heating under specified conditions with an excess of a standard volumetric ethanolic potassium hydroxide solution. The excess alkali is determined by back titration with a standard solution of hydrochloric acid.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 Ethanol, 95 % (volume fraction) at 20 °C, freshly neutralized with the potassium hydroxide solution (5.2), in the presence of the coloured indicator (5.4) used for the determination.

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5.2 Potassium hydroxide, standard volumetric ethanolic solution, c(KOH) = 0,5 mol/l at 20 °C, freshly restandardized before each series of tests.

5.3 Hydrochloric acid, standard volumetric solution, c(HCl) = 0,5 mol/l at 20 °C.

It is important that the reagent be taken at the specified temperature of 20 °C, particularly the ethanolic solution of potassium hydroxide, as the volume varies greatly with temperature.

5.4 Coloured indicator.

Use **phenolphthalein**, 2 g/l solution in ethanol (5.1), or **phenol red**, 0,4 g/l solution in ethanol, 20 % (volume fraction), if the essential oil has components that contain phenol groups.

NOTE This particular case will be specified in the specific standard for the essential oil concerned.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Saponification flask, with ground glass neck, of alkali-resistant glass, of capacity 100 ml to 250 ml, to which can be fitted a ground glass air condenser at least 1 m in length with 1 cm to 1,5 cm internal diameter.

If necessary, and particularly for the essential oils with high light fractions and depending on the time placed in the boiling water bath, the glass tube may be replaced by a water-cooled reflux condenser.

6.2 Test tubes, of capacity 5 ml.

6.3 Burettes, of capacity 25 ml, graduated in 0,05 ml, complying with the requirements of ISO 385-1, class B.

6.4 Boiling water bath.

6.5 Analytical balance, accurate to the nearest 0,001 g.

6.6 Potentiometer.

7 Sampling

Sampling is not included in the method specified in this International Standard. A recommended sampling method is given in ISO 212[1)].

It is important that the laboratory receive a representative sample, not damaged or modified during transport or storage before the arrival at the laboratory.

8 Preparation of test sample

The test sample shall be prepared according to ISO 356.

1) ISO 212, *Essential oils — Sampling*.

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9 Procedure

9.1 Test portion

Weigh, to the nearest 0,005 g, 2 g of the test sample.

The test portion may be different from this, if so specified in the specific standard for the essential oil concerned.

9.2 Blank test

Carry out a blank test, in parallel with the determination (9.3), under the same conditions and using the same reagents. (See 9.3.3.)

9.3 Determination

9.3.1 Introduce the test portion (9.1) into the saponification flask (6.1). Add from the burette (6.3) 25 ml of the potassium hydroxide solution (5.2) (see note) and fragments of pumice stone or porcelain.

NOTE If the test portion has been retained from the determination of the acid value, it will not be necessary to neutralize it before adding the potassium hydroxide.

For oils with a high ester value, increase the volume of the potassium hydroxide solution (5.2) used so that $(V_0 - V_1)$ (see clause 10) is at least equal to 10 ml.

For oils with a low ester value, increase the mass of the test portion used.

Attach the air condenser or water-cooled reflux condenser, and place the flask in the boiling water bath (6.4) for a time depending on the essential oil analysed. This time is mentioned in the specification for the oil to be tested.

Allow to cool and remove the tube. Add 20 ml of water and 5 drops of the phenolphthalein solution, or of the phenol red solution (5.4) if the essential oil contains phenols or compounds with phenolic groups.

9.3.2 Titrate the excess potassium hydroxide with the hydrochloric acid (5.3).

9.3.3 This determination may be carried out with the solution resulting from the determination of the acid value, which can be used as the blank test, by adding 5 ml of ethanol (5.1) in this blank test before the addition of the 25 ml of potassium hydroxide solution (this volume corresponds to the volume introduced during the determination of the acid value).

9.4 Potentiometry

Potentiometry may be used for all the essential oils, but it is particularly recommended for highly coloured essential oils for which it is difficult to appreciate the endpoint of the coloured indicator (e.g. vetiver oil). In this case, the same reagents and apparatus shall be used.

NOTE These special cases will be established in the specific standards for the essential oils concerned.

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10 Expression of results

10.1 Calculation

10.1.1 Ester value

The ester value (EV) is given by the formula

$$\text{EV} = \frac{28{,}05}{m}(V_0 - V_1) - \text{AV}$$

where

V_0 is the volume, in millilitres, of hydrochloric acid (5.3) used for the blank test (9.2);

V_1 is the volume, in millilitres, of hydrochloric acid (5.3) used for the determination (9.3.2);

m is the mass, in grams, of the test portion;

AV is the acid value determined according to ISO 1242.

The mass fraction of ester, w, as a percentage, with respect to a stated ester, is given by the formula

$$w = \frac{M_\text{r} \cdot \text{EV}}{561}$$

where

M_r is the relative molecular mass of the ester used to express the results conventionally;

EV is the ester value calculated as above.

Express the ester value to two significant figures when it is less than 100, and to three significant figures when it is 100 or more.

10.1.2 Ester value determined after the acid value

When the determination is carried out on the solution resulting from the determination of the acid value, the ester value (EV) is obtained by the formula

$$\text{EV} = \frac{28{,}05}{m}(V_0 - V_1')$$

where V_1' is the volume, in millilitres, of hydrochloric acid (5.3) used in the new determination.

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11 Test report

The test report shall state:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained.

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Annex A
(informative)

Precision

A.1 General

At the time of publication of this International Standard, precision values for this test method have not been established. Nevertheless, taking into account the data available at present, it was considered useful for the users of this International Standard to include some indications of the values obtained for repeatability and reproducibility, and it is expected to be able to specify definitive values in the next revision of this International Standard.

A.2 Repeatability

The absolute difference between two independent single test results, obtained using this method on the same essential oil tested in the same laboratory by the same operator using the same equipment within a short period of time, was not, in more than 5 % of cases, greater than 0,7 as ester value or 0,25 % for an ester having, for example, a molar mass of 196,29.

NOTE In the case of coloured essential oils, the difference between two measurements is greater when colorimetry is used instead of potentiometry.

A.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on the same essential oil tested in different laboratories with different operators using different equipment, was not, in more than 5 % of cases, greater than 1,4 as ester value or 0,5 % for an ester having, for example, a molar mass of 196,29.

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