International Standard 1241

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Essential oils — Determination of ester value after acetylation and evaluation of free alcohols and total alcohols content

Huiles essentielles — Détermination de l'indice d'ester après acétylation et évaluation de la teneur en alcools libres et en alcools totaux

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1241 was developed by Technical Committee ISO/TC 54, *Essentials oils*.

It was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 1241-1971, which had been approved by the member bodies of the following countries :

Australia	Iran	South Africa, Rep. of
Belgium	Israel	Sweden
Bulgaria	Italy	Thailand
Canada	Japan	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	New Zealand	USSR
Greece	Portugal	
India	Romania	

No member body had expressed disapproval of the document.

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Essential oils — Determination of ester value after acetylation and evaluation of free alcohols and total alcohols content

1 Scope and field of application

This International Standard specifies a method for the determination of ester value after acetylation.

Through the determination of ester value before and after acetylation, it is possible to evaluate free alcohols and total alcohols content.

This method is not applicable to essential oils containing appreciable quantities of

— tertiary alcohols which would not be completely acetylated, or

— phenols, lactones, aldehydes or enolysable ketones, which would be acetylated in addition to free alcohols.

2 References

ISO 212, *Essential oils — Sampling.*

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

ISO/R 385, *Burettes.*

ISO 709, *Essential oils — Determination of ester value.*

3 Definition

ester value after acetylation : The number of milligrams of potassium hydroxide which are required to neutralize the acids liberated by the hydrolysis of the esters contained in 1 g of the acetylated oil.

4 Principle

Acetylation of the essential oil by acetic anhydride in the presence of sodium acetate. Isolation and drying of the acetylated oil, and determination of its ester value in accordance with ISO 709.

Calculation of the free, combined and total alcohol contents from the ester values before and after acetylation.

5 Reagents

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

Reagents specified in ISO 709 and

5.1 Acetic anhydride, concentration not less than 98 %.

5.2 Sodium acetate, anhydrous, freshly melted and powdered.

5.3 Sodium chloride, saturated solution.

5.4 Sodium carbonate /sodium chloride, 20 g/l solution of anhydrous sodium carbonate saturated with sodium chloride.

5.5 Magnesium sulphate anhydrous, or **sodium sulphate** anhydrous and neutral, freshly dried and powdered.

5.6 Litmus paper.

6 Apparatus

Ordinary laboratory apparatus, and

6.1 Acetylation apparatus, including a round-bottomed acetylation flask of capacity 100 to 250 ml with a ground glass neck, provided with a glass tube to act as a reflux condenser, at least 1 m in length and at least 10 mm internal diameter.

Both the round-bottomed flask and the condenser shall be carefully dried before use.

6.2 Measuring cylinders, of capacity 10 ml, graduated in 0,1 ml.

6.3 Measuring cylinders, of capacity 50 ml, graduated in 1 ml.

6.4 Water bath, capable of being controlled at 40 to 50 °C.

6.5 Suitable heating device, for maintaining boiling without local overheating.

6.6 Separating funnel, of capacity at least 250 ml.

6.7 Saponification apparatus, including an alkali-resistant glass flask, of capacity 100 to 200 ml, to which can be fitted a reflux condenser, at least 1 m in length and of at least 10 mm internal diameter.

6.8 Burette, of capacity 50 ml, graduated in 0,1 ml, conforming to the requirements of ISO/R 385.

7 Sampling

See ISO 212.

8 Procedure

8.1 Preparation of test sample

See ISO 356.

8.2 Determination of ester value before acetylation

Determine the ester value before acetylation in accordance with the requirements of ISO 709.

8.3 Determination of ester value after acetylation

Mix approximately 10 ml of the test sample (8.1), 10 ml of the acetic anhydride (5.1) and 2 g of the anhydrous sodium acetate (5.2) in the acetylation flask (6.1). Add fragments of pumice-stone or porcelain and fit the flask with its reflux condenser.

Heat the flask by means of the heating device (6.5) and gently reflux the liquid for 2 h or for the time given in the International Standard for the essential oil being analysed.

At the end of this period, allow the liquid to cool, add 50 ml of distilled water and heat on the water bath, controlled at 40 to 50 °C for 15 min, shaking frequently. Allow to cool to room temperature, remove the reflux tube and transfer the liquid to the separating funnel (6.6). Wash the flask twice with 10 ml of water and collect the washings in the separating funnel. Wait until separation of the phases is complete, then draw off and reject the aqueous phase.

Wash the oil phase by shaking successively with

a) 50 ml of the sodium chloride solution (5.3),

b) 50 ml of the sodium carbonate/sodium chloride solution (5.4),

c) 50 ml of the sodium chloride solution (5.3), and

d) 20 ml of water.

Proceed by shaking the acetylated essential oil with the saturated solutions (a, b, c) and, gently, with the water (d), which, if the washings have been properly conducted, will be neutral to the litmus paper (5.6). Run the oil phase into a dry tube and shake several times over a period of 15 min with at least 3 g of the anhydrous magnesium or sodium sulphate (5.5). Filter. Repeat the contact and shaking with further 3 g portions of magnesium or sodium sulphate until the acetylated oil is free from water.

Determine the ester value, in accordance with the requirements of ISO 709, but using approximately 2 g, weighed to the nearest 0,5 mg, of the acetylated oil and 50 ml of the 0,5 mol/l standard volumetric potassium hydroxide solution.

9 Expression of results

9.1 The ester value after acetylation is given by the formula

$$EV_2 = \frac{28{,}05}{m}(V'_0 - V'_1)$$

9.2 The percentage by mass of free alcohols, with respect to a given ester, is given by the formula

$$\frac{M_r \times (EV_2 - EV_1)}{561 - 0{,}42\ EV_2}$$

9.3 The percentage by mass of combined alcohols, with respect to a given ester, is given by the formula

$$\frac{M_r \times EV_1}{561}$$

9.4 The percentage by mass of total alcohols is obtained by adding the two previous percentages.

In the above formulae,

m is the mass, in grams, of the test portion taken for the determination of the acid value (see ISO 1242) (generally 2 ± 0,05 g);

V'_0 is the volume, in millilitres, of the hydrochloric acid solution (5.3 of ISO 709), used for the blank test (7.2 of ISO 709);

V'_1 is the volume, in millilitres, of the hydrochloric acid solution (5.3 of ISO 709), used for the determination (7.1 of ISO 709) of the ester value after acetylation;

M_r is the relative molecular mass of the ester used to express results conventionally and which is given in the International Standard for the essential oil being analysed;

EV_1 is the ester value of the oil before acetylation (8.2), calculated in accordance with the requirements of ISO 709;

EV_2 is the ester value of the oil after acetylation (8.3), calculated in accordance with the requirements of ISO 709.

10 Test report

The test report shall state the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that might have influenced the results. The test report shall include all details required for the complete identification of the sample.