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Essential oils (containing tertiary alcohols) – Estimation of free alcohols content by determination of ester value after acetylation

Huiles essentielles (contenant des alcools tertiaires) – Évaluation de la teneur en alcools libres par détermination de l'indice d'ester après acétylation

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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 3794 was drawn up by Technical Committee ISO/TC 54, *Essential oils*, and was circulated to the Member Bodies in February 1975.

It has been approved by the Member Bodies of the following countries :

Belgium	Italy	Sri Lanka
Canada	Netherlands	Turkey
France	Portugal	Yugoslavia
Germany	South Africa, Rep. of	
India	Spain	

No Member Body expressed disapproval of the document.

Essential oils (containing tertiary alcohols) — Estimation of free alcohols content by determination of ester value after acetylation

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for estimating the free alcohols content of essential oils, by determination of ester value after acetylation.¹⁾

This method is applicable to essential oils containing an appreciable proportion of tertiary alcohols, namely of linalol and terpineol, ISO/R 1241 not being applicable to those oils.

This method is not applicable to essential oils containing appreciable proportions of phenols, anthranilates, lactones and aldehydes, as stated in ISO/R 1241.

2 REFERENCES

ISO 212, *Essential oils — Sampling*.

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

ISO/R 709, *Determination of ester value and calculation of ester content of essential oils*.

ISO/R 1241, *Essential oils — Estimation of free alcohols content by determination of ester value after acetylation*.

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 acetyl chloride and acetic anhydride, in the presence of dimethylaniline. Isolation and drying of the acetylated oil. Determination of the ester value after acetylation. Calculation of the free alcohols content taking into account the ester value of the same but non-acetylated essential oil.

5 REAGENTS

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

5.1 Acetyl chloride.

5.2 Dimethylaniline, freshly distilled and free from monomethylaniline and aniline.

5.3 Acetic anhydride, concentration not less than 98 %.

5.4 Anhydrous magnesium sulphate or sodium sulphate, recently desiccated and powdered.

5.5 Sodium sulphate (Na_2SO_4), 100 g/l solution.

5.6 Hydrochloric acid solution.

Dilute 5 parts of hydrochloric acid (ρ_{20} 1,19 g/ml) to 100 parts by volume with the sodium sulphate solution (5.5).

5.7 Sodium hydrogen carbonate solution.

Dissolve 50 g of sodium hydrogen carbonate in 1 l of the sodium sulphate solution (5.5).

5.8 Potassium hydroxide, 0,1 N aqueous solution.

5.9 Potassium hydroxide, 0,5 N standard volumetric solution in 95 % (V/V) ethanol.

5.10 Hydrochloric acid, 0,5 N standard volumetric solution.

5.11 Litmus paper.

5.12 Phenolphthalein, 2 g/l solution in 95 % (V/V) ethanol.

6 APPARATUS

Ordinary laboratory equipment, and

6.1 Acetylation apparatus, comprising a 100 ml round-bottomed acetylation flask connected by a ground glass joint to a glass tube at least 1 m long and with an inside diameter of about 10 mm, to act as an air condenser, and a ground glass stopper to fit the flask.

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

1) This method is usually known as the "Fiore method".