# INTERNATIONAL STANDARD



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION MEЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ ORGANISATION INTERNATIONALE DE NORMALISATION

### Essential oils — Estimation of primary and secondary free alcohols content by acetylation in pyridine

vaires et ser ling in the film Huiles essentielles — Évaluation de la teneur en alcools libres primaires et secondaires par acétylation pyridinée

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#### **FOREWORD**

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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 3793 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:

Australia

Belgium

Canada France

Germany

Italy Netherlands

Portugal

Spain Turkey

Yugoslavia

No Member Body expressed disapproval of the document.

## Essential oils — Estimation of primary and secondary free alcohols content by acetylation in pyridine

#### 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for estimating the primary and secondary free alcohols content of essential oils, by acetylation in pyridine.

Primary and secondary alcohols are entirely acetylated by this method. Phenols, anthranilates and aldehydes are acetylated in their entirety or in part. Tertiary alcohols are acetylated only to a negligible extent.

#### 2 REFERENCES

ISO 212, Essential oils - Sampling.

ISO/R 356, Essential oils — Methods of test — Preparation of sample.

ISO 1242, Essential oils — Determination of the acid value.

#### 3 PRINCIPLE

Acetylation of hydroxyl compounds by a mixture of acetic anhydride and pyridine. Hydrolysis of the excess acetic anhydride and determination of the resulting acetic acid.

#### 4 REAGENTS

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

- 4.1 Acetic anhydride, concentration not less than 98 %.
- **4.2** Pyridine, dried, for example, by distillation over solid potassium hydroxide or sodium hydroxide.

#### 4.3 Acetylation mixture.

Mix 1 volume of acetic anhydride (4.1) with 3 volumes of pyridine (4.2).

This mixture shall be prepared fresh daily.

- **4.4 Potassium hydroxide**, approximately 0,5 N standard volumetric solution in 95 % (V/V) ethanol.
- **4.5** Phenolphthalein, 2 g/l solution in 95 % (V/V) ethanol.

#### 5 APPARATUS

Normal laboratory equipment, and

**5.1** 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.

- **5.2** Calibrated burette, capacity 50 ml, graduated in 0.1 ml, complying with class B of ISO/R 385.
- **5.3 Calibrated burette**, capacity 5 ml, graduated in 0.05 ml.
- 5.4 Boiling water bath.
- 5.5 Analytical balance.

#### 6 SAMPLING

See ISO 212.

#### 7 PROCEDURE

#### 7.1 Preparation of test sample

See ISO/R 356.

#### 7.2 Determination of acid value

Determine the acid value of the oil to be tested, in accordance with ISO 1242.

#### 7.3 Test portion

Weigh to the nearest 0,000 1 g, about 1 g of the test sample in the round-bottomed flask of the carefully dried acetylation apparatus (5.1), with its stopper in place.

#### 7.4 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all reagents.