INTERNATIONAL STANDARD ISO 4096

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Essential oils (containing tertiary alcohols) — Evaluation of free alcohols content by determination of ester value after cold formylation

Huiles essentielles (contenant des alcools tertiaires) — Évaluation de la teneur en alcools libres par détermination de l'indice d'ester après formylation à froid

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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 4096 was developed by Technical Committee ISO/TC 54, *Essential oils*, and was circulated to the member bodies in February 1977.

It has been approved by the member bodies of the following countries :

Austria	Italy	Netherlands
Canada	Japan	South Africa, Rep. of
France	Kenya	Thailand
India	Korea, Rep. of	Turkey

No member body expressed disapproval of the document.

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Essential oils (containing tertiary alcohols) — Evaluation of free alcohols content by determination of ester value after cold formylation

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the evaluation of free alcohols content, by determination of ester value after cold formylation, in essential oils containing tertiary alcohols.[1] Total alcohols and combined alcohols content can also be determined with this method.

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.*

3 DEFINITION

ester value after formylation : 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 formylated oil.

4 PRINCIPLE

Cold formylation of the essential oil by a mixture of formic acid and acetic anhydride. Determination of the ester value after formylation. Calculation of free alcohols content taking into account the ester value of the same oil before formylation.

5 REAGENTS

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

The reagents specified in ISO/R 709, and

5.1 Magnesium sulphate or **sodium sulphate,** freshly ignited and powdered.

5.2 Formylation mixture.[2]

Slowly add, while shaking, 1 volume of 100 % (*m/m*) formic acid to 2 volumes of acetic anhydride, concentration not less than 98 % (*m/m*), maintaining the temperature below 15 °C and carefully excluding any traces of moisture through the use of an appropriate device.

After completing the mixture, heat to 50 °C, in 15 min, and cool suddenly in a melting ice bath. Prepare the mixture immediately before its use, on account of its instability.

5.3 Sodium chloride, 100 g/l aqueous solution.

5.4 Sodium chloride-sodium carbonate, aqueous solution.

Dissolve 2 g of sodium carbonate in 100 ml of sodium chloride solution (5.3).

6 APPARATUS

Usual laboratory equipment, and in particular :

6.1 Flask, 50 ml capacity, with a ground stopper.

6.2 Melting ice bath.

6.3 Measuring cylinders, 50 ml capacity, graduated in millilitres.

6.4 Separating funnels, 250 ml, complying with the requirements of ISO 4800.

7 SAMPLING

See ISO 212.

8 PROCEDURE

8.1 Preparation of test sample

See ISO 356.

1) The method is not applicable to the alcohol of patchouli.

2) It is necessary to check the concentration of reagents before use.