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**Olive oils and olive-pomace oils —  
Determination of aliphatic alcohols  
content by capillary gas chromatography**

*Huiles d'olive et huiles de grignons d'olive — Détermination de la teneur  
en alcools aliphatiques par chromatographie en phase gazeuse sur  
colonne capillaire*

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## Contents

Page

Foreword .....	iv
Introduction.....	v
1 Scope .....	1
2 Normative references .....	1
3 Terms and definitions .....	1
4 Principle.....	1
5 Reagents.....	1
6 Apparatus .....	2
7 Sampling.....	3
8 Preparation of the test sample .....	3
9 Procedure .....	4
9.1 Preparation of the unsaponifiable matter .....	4
9.2 Separation of alcoholic fractions .....	4
9.3 Preparation of the trimethylsilyl ethers.....	5
9.4 Gas chromatographic analysis .....	6
10 Precision.....	7
10.1 Interlaboratory test.....	7
10.2 Repeatability .....	7
10.3 Reproducibility .....	7
11 Test report.....	8
Annex A (informative) Determination of the linear velocity of the gas .....	9
Annex B (informative) Results of an interlaboratory test .....	10
Bibliography.....	11

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12871 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

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## Introduction

As part of the *Trade standard applying to olive oils and olive-pomace oils*, the International Olive Oil Council (IOOC) — now known as the International Olive Council (IOC) — published COI/T.20/Doc. 26:2003<sup>[4]</sup>. COI/T.20/Doc. 26 was applicable to olive and olive-pomace oils and was used to distinguish between lampante virgin olive oils and crude olive-pomace oils. Olive pomace is the residual paste which still contains a variable amount of water and oil after pressing or centrifuging.

In 2008, the IOC submitted the document to ISO/TC 34/SC 11 for adoption as an International Standard.

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# Olive oils and olive-pomace oils — Determination of aliphatic alcohols content by capillary gas chromatography

## 1 Scope

This International Standard specifies a procedure for the determination of the content, as a mass fraction expressed as milligrams per kilogram, of aliphatic alcohols in olive oils and olive-pomace oils.

NOTE This International Standard is based on COI/T.20/Doc. 26:2003<sup>[4]</sup>.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

## 3 Terms and definitions

ISO 12871:2010

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### aliphatic alcohols content

sum of the aliphatic alcohols with carbon number  $C_{22}$ ,  $C_{24}$ ,  $C_{26}$ , and  $C_{28}$ , as a mass fraction, determined according to the method specified in this International Standard

## 4 Principle

The oil, to which 1-eicosanol has been added as an internal standard, is saponified with ethanolic potassium hydroxide and the unsaponifiable matter extracted with diethyl ether. The alcoholic fraction is separated from the unsaponifiable matter by chromatography on a basic silica gel plate; the alcohols recovered from the silica gel are transformed into trimethylsilyl ethers (TMSE) and analysed by capillary gas chromatography.

## 5 Reagents

**WARNING — Comply with any local regulations which specify the handling of hazardous substances. Technical, organizational and personal safety measures shall be followed.**

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

### 5.1 Potassium hydroxide, ethanolic solution, $c(\text{KOH}) \approx 2 \text{ mol/l}$ .

Dissolve, while cooling, 130 g potassium hydroxide [ $w(\text{KOH}) = 85 \%$  mass fraction minimum] in 200 ml water and make up to 1 l with ethanol. Store the solution in a well-stoppered opaque glass bottle.

## ISO 12871:2010(E)

**5.2 Potassium hydroxide**, ethanolic solution,  $c$  (KOH)  $\approx$  0,2 mol/l.

Dissolve 13 g potassium hydroxide in 20 ml water and make up to 1 l with ethanol.

**5.3 Diethyl ether**.

**5.4 Anhydrous sodium sulfate**.

**5.5 Glass plates**, coated with silica gel, without fluorescence indicator, 0,25 mm thick.

Suitable ready-for-use products are available commercially.

**5.6 Benzene**, chromatography grade.

**5.7 Acetone**, chromatography grade.

**5.8 Hexane**, chromatography grade.

**5.9 Diethyl ether**, chromatography grade.

**5.10 Chloroform**, chromatography grade.

**5.11 Reference solution for thin-layer chromatography**: 1-eicosanol, 0,5 g/100 ml solution in chloroform, or a fraction of alcohols obtained as indicated in 9.2 from the unsaponifiable matter of an olive-pomace oil.

**5.12 2',7'-Dichlorofluorescein in ethanol**, 0,2 g/100 ml solution. Make slightly basic by adding a few drops of alcoholic potassium hydroxide solution (5.1).

**5.13 Anhydrous pyridine**, chromatography grade.

**5.14 Hexamethyldisilazane (HMDS)**.

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**5.15 Trimethylchlorosilane (TMCS)**.

**5.16 Standard solutions of trimethylsilyl ethers (TMSE)**, of aliphatic alcohols from C<sub>20</sub> to C<sub>28</sub>. Prepare from mixtures of pure alcohols immediately prior to use.

**5.17 Internal standard solution**: solution of 1-eicosanol in chloroform, mass concentration 0,1 g/100 ml.

**5.18 Carrier gas**: hydrogen or helium, gas chromatography grade.

**5.19 Auxiliary gas**: nitrogen, gas chromatography grade.

## 6 Apparatus

Usual laboratory equipment and in particular the following.

**6.1 Round-bottomed flask**, of capacity 250 ml, fitted with a reflux condenser having ground-glass joints.

**6.2 Separating funnel**, of capacity 500 ml.

**6.3 Round-bottomed flasks**, of capacity 250 ml.

**6.4 Chromatographic chamber for thin-layer chromatography**, suitable for glass plates of dimensions 20 cm  $\times$  20 cm.

**6.5 Ultraviolet lamp**, of wavelength 366 nm or 254 nm.



**6.6 Microsyringes**, of capacities 100 µl and 500 µl.

**6.7 Cylindrical filter funnel** with a G3 porous septum (porosity 15 µm to 40 µm), of approximate dimensions: diameter 2 cm and depth 5 cm, with an attachment suitable for filtration under vacuum and a 12/21 male ground-glass joint.

**6.8 Vacuum conical flask**, of capacity 50 ml, with a 12/21 female ground-glass joint which can be fitted to the filter funnel (6.7).

**6.9 Test-tube**, of capacity 10 ml, with a tapering bottom and a sealing stopper.

**6.10 Gas chromatograph**, suitable for use with capillary columns, equipped with the components specified in 6.10.1 to 6.10.4.

**6.10.1 Column oven**, capable of maintaining a temperature to within  $\pm 1$  °C.

**6.10.2 Split injection unit**, temperature-adjustable, with a persilylated glass vaporizing element, or an on-column unit.

**6.10.3 Flame ionization detector**.

**6.10.4 Integration system**.

**6.11 Fused silica capillary column**, of length 20 m to 30 m, internal diameter 0,25 mm to 0,32 mm, with SE-52 or SE-54<sup>1)</sup> liquid phase or equivalent, with a film thickness between 0,10 µm and 0,30 µm.

**6.12 Microsyringe for gas chromatography**, of capacity 10 µl, with hardened needle.

**6.13 Analytical balance**, sensitive to 1 mg (with 0,1 mg display).

**6.14 Desiccator**, with calcium chloride as desiccant.

**6.15 Drying oven**.

## 7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555<sup>[1]</sup>.

It is important that the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

## 8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

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1) SE-52 and SE-54 are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.