SLOVENSKI STANDARD
SIST EN 14103:2003

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Derivati maġļ ob in olj - Metil estri maġļ obnih kislin (FAME) - Doloļ evanje estra in metil estra linolenske kisline

Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of ester and linolenic acid methyl ester contents

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Ester-Gehaltes und des Gehaltes an Linolensäure-Methylester

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la teneur en ester et en ester méthylique de l'acide linolénique

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Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Ester-Gehaltes und des Gehaltes an Linoleinsäure-Methylester

This European Standard was approved by CEN on 2 January 2003.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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Foreword

This document (EN 14103:2003) has been prepared by Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

This document has been prepared under Mandate M/245 on Fatty Acid Methylester (FAME) given to CEN by the European Commission and the European Free Trade Association.

Annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom

1 Scope

The purpose of this European Standard is to determine the ester content of fatty acid methyl esters (FAME) intended for use as pure biofuel or as a blending component for heating and diesel fuels. It also allows to determine the linolenic acid methyl ester content.

It allows one to verify that the ester content of FAME is greater than 90 % (*m/m*) and that the linolenic acid content is between 1 % (*m/m*) and 15 % (*m/m*).

This method is suitable for FAME which contain methyl esters between C_{14} and C_{24}.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 5508, *Animal and vegetable fats and oils - Analysis by gas chromatography of methyl esters of fatty acids (ISO 5508:1990).*

3 Principle

Determination of the percentage of methyl esters of fatty acids present in the sample by gas chromatography according to EN ISO 5508 with internal calibration (methyl heptadecanoate).

Determination of the percentage of linolenic acid methyl ester present in the sample by gas chromatography according to EN ISO 5508.

4 Glassware

4.1 Screw-cap vials with PTFE-faced septa, 10 ml capacity.

4.2 Volumetric flask, 50 ml capacity.

4.3 Pipette, of 5 ml capacity, accurate to 0,02 ml.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Heptane.

5.2 Methyl heptadecanoate of known purity (99 % minimum).

5.3 Methyl heptadecanoate, 10 mg/ml solution: accurately weigh approximately 500 mg of methyl heptadecanoate (5.2) in a 50 ml volumetric flask (4.2) and make up to mark with heptane (5.1).

6 Apparatus

Usual laboratory apparatus and, in particular, the apparatus described in EN ISO 5508.

7 Sampling

Sampling is not part of the method specified in this European Standard. A recommended sampling method is given in EN ISO 5555 [1].

8 Preparation of the sample

Accurately weigh approximately 250 mg of sample in a 10 ml vial (4.1), then add 5 ml of methyl heptadecanoate solution (5.3) using a pipette (4.3).

9 Chromatographic analysis

Refer to EN ISO 5508 and to annex A which described, by way of indication, analysis conditions which may be used.

The chromatographic conditions (injected quantity, oven temperature, carrier gas pressure and split flow rate shall be adjusted so as to correctly visualize the methyl ester peaks of the lignoceric (C_{24}) and nervonic ($C_{24:1}$) acids.

The integration shall be carried out as from the methyl myristate (C_{14}) peak up to that of the methyl ester in $C_{24:1}$ taking all the peaks into consideration, including the minor ones.

NOTE If some unknown peaks are found (others than saturated and mono-unsaturated FAME) between the linolenic acid ($C_{18:3}$) and the nervonic acid ($C_{24:1}$), presence of fish oil in the sample can be suspected.

10 Expression of results

10.1 Determination of ester content

The ester C content, expressed as a mass fraction in percent, is calculated using the following formula:

$$C = \frac{\left(\sum A\right) - A_{EI}}{A_{EI}} \times \frac{C_{EI} \times V_{EI}}{m} \times 100\%$$

where

ΣA is the total peak area from the methyl ester in C_{14} to that in $C_{24:1}$;

A_{EI} is the peak area corresponding to methyl heptadecanoate;

C_{EI} is the concentration, in milligrams per millilitre, of the methyl heptadecanoate solution (5.3) being used;

V_{EI} is the volume, in millilitres, of the methyl heptadecanoate solution (5.3) being used;

m is the mass, in milligrams, of the sample.

NOTE 1 In the case of vegetable oils, the result of the calculation based on relative areas is considered to represent a percentage by mass.

NOTE 2 If the average of two determinations is higher than 100,8 % then discard the results and verify the experimental conditions as well as the purity of internal standard by using this method to determine the ester content of a commercial or prepared mixture.

Express the result to one decimal place.

10.2 Determination of linolenic acid methyl ester

The linolenic acid methyl ester content L, expressed as a mass fraction in percent, is calculated using the following formula :

$$L = \frac{A_L}{\left(\sum A\right) - A_{EI}} \times 100\ \%$$

where

ΣA is the total peak area from the methyl ester in C_{14} to that in $C_{24:1}$;

A_{EI} is the peak area corresponding to methyl heptadecanoate;

A_L is the peak area corresponding to linolenic acid methyl ester.

Express the result to one decimal place.

11 Precision

An interlaboratory test organized in 2000 at European level with the participation of eleven laboratories, each having carried out two determinations on each sample, gave the statistical results indicated in annex B.

11.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short time interval, shall not be greater than:

For ester content	1,6 % (*m/m*)
For linolenic acid methyl ester content	0,1 % (*m/m*)

more than once out of 20 determinations.

11.2 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, shall not be greater than:

For ester content	3,1 % (*m/m*)
For linolenic acid methyl ester content (**L**)	0,311 x L + 0,02 % (*m/m*)

more than once out of 20 determinations.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used if known;

— the test method used, with reference to this European standard;

— all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained, or if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Summary of the analysis conditions of methyl esters by gas chromatography

A.1 Capillary column coated with a polyethylene glycol stationary phase (Carbowax 20M, DBwax, CPwax, etc.).

— Length : 30 m;

— internal diameter : 0,32 mm;

— film thickness : 0,25 µm.

A.2 Variable flow split injector

— Split flow rate : 20 ml/min to 100 ml/min;

— temperature : 250 °C.

A.3 Carrier gas : hydrogen or helium

— Pressure : 30 kPa to 100 kPa;

— flow : 1 ml/min at 2 ml/min (depending on characteristics of column being used).

A.4 Flame ionization detector

— Temperature : 250 °C.

A.5 Oven

— Temperature : 200 °C