SLOVENSKI STANDARD
SIST EN 14331:2004

01-september-2004

Tekoļ i naftni proizvodi - Loļ ba in opredelitev metil estrov maģļ obnih kislin (FAME) v srednjih destilatih - Metoda tekoļ inske (LH)/plinske kromatografije (GC)

Liquid petroleum products - Separation and characterisation of fatty acid methyl esters (FAME) from middle distillates - Liquid chromatography (LC)/gas chromatography (GC) method

Flüssige Mineralölerzeugnisse - Trennung und Bestimmung von Fettsäure-Methylestern (FAME) aus Mitteldestillaten - Flüssigchromatographie (LC)/Gaschromatographie (GC)

Produits pétroliers liquides - Séparation et caractérisation des esters méthyliques d'acides gras (EMAG) dans les distillats moyens - Méthode par chromatographie liquide (CL) et chromatographie en phase gazeuse (CPG)

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Liquid petroleum products - Separation and characterisation of fatty acid methyl esters (FAME) from middle distillates - Liquid chromatography (LC)/gas chromatography (GC) method

Produits pétroliers liquides - Séparation et caractérisation des esters méthyliques d'acides gras (EMAG) dans les distillats moyens - Méthode par chromatographie liquide (CL) et chromatographie en phase gazeuze (CPG)

Flüssige Mineralölerzeugnisse - Trennung und Bestimmung von Fettsäure-Methylestern (FAME) aus Mitteldestillaten - Flüssigchromatographie (LC)/Gaschromatographie (GC)

This European Standard was approved by CEN on 16 January 2004.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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 Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 14331:2004) has been prepared by Technical Committee CEN /TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NEN.

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 September 2004, and conflicting national standards shall be withdrawn at the latest by September 2004.

Annex A is normative. Annexes B and C are informative.

This document includes a Bibliography.

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

1 Scope

This European Standard specifies a method for the separation of fatty acid methyl esters (FAME) from middle distillates by liquid chromatography (LC) and for quantitative determination of the individual esters by gas chromatography (GC).

This method is applicable to FAME of vegetable or animal origin that contain methyl esters between C_{14} to C_{24}. These FAME are mainly composed of C_{16} - C_{18} esters from fatty acids. This method is applicable whatever the origin of the middle distillate.

This test method has been evaluated for the separation and characterisation of FAME present at up to 5 % *(V/V)* in middle distillate.

NOTE For the purposes of this European Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction and the volume fraction.

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 3170, *Petroleum liquids - Manual sampling (ISO 3170:1988, including Amendment 1:1998).*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171:1988).*

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

3 Principle

The method consists of two stages:

— separation of the FAME fraction from the middle distillate by liquid adsorption chromatography at atmospheric pressure on a silica micro-column;

— characterization of the separated FAME fraction by gas chromatography.

4 Reagents and materials

4.1 Hexane, HPLC analytical grade.

4.2 Diethyl ether, HPLC analytical grade.

5 Apparatus

5.1 General

General gas chromatographic and liquid chromatographic equipment shall be used.

5.2 Micro-column, containing approximately 700 mg silica (particle size 55 µm -105 µm), with approximate dimensions of 25 mm of height and 10 mm of diameter.

5.3 Test tube, 20 ml.

6 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

7 Procedure

7.1 Separation on silica column

Add to the top of the silica micro-column (5.1) 0,100 ml of the sample and allow to percolate into the column for about 2 min.

Elute the middle distillate fraction with 10 ml hexane (4.1). The rate of elution shall be slow (drop by drop) at the rate of about 3 ml/min. This fraction is discarded.

Then elute the FAME fraction with 10 ml diethyl ether (4.2) into a test tube (5.2).

NOTE If the FAME content in the middle distillate is greater than 5 % (*V/V*), it is recommended to dilute the sample with a FAME free fuel to obtain a content lower than 5 % (*V/V*).

7.2 Gas chromatographic analysis

Refer to either EN ISO 5508 or annex A, which summarizes the conditions of the analysis.

NOTE In annex B a chromatogram of a sample of fatty acid methyl esters from rapeseed oil is given.

Chromatographic conditions such as injection size and/or split ratio shall be adjusted to detect the minor components e.g. the minor peaks of $C_{24:0}$ and $C_{24.1}$ esters from acids.

A chromatogram obtained from a sample with known FAME composition under identical conditions to those used for the analysis of the unknown sample may be used to establish retention times for purposes of peak identification.

8 Determination of the composition of a mixture of methyl esters

The amount of constituent *X*, expressed as a mass fraction of the FAME fraction, is calculated from the area of the corresponding peak divided by the area of all the peaks, using the equation:

$$X = 100 \frac{A_i}{\Sigma A} \qquad (1)$$

where:

A_i is the area of the peak corresponding to component *i*.

ΣA is the sum of the areas of all the peaks eluted between C_{14} and C_{24} esters.

9 Expression of results

Report the amount of each component of the FAME fraction as % (*m/m*) to the nearest 0,1 % *(m/m)*.

10 Precision

NOTE The precision of the method has been obtained by statistical examination of interlaboratory data for samples containing FAME from rapeseed oil.

10.1 Repeatability

The difference between two results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in twenty.

10.2 Reproducibility

The difference between two single and independent results obtained by different operators in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in twenty.

Table 1 — Precision data

Methyl ester of	Repeatability % (*m/m*)	Reproducibility % (*m/m*)
Palmitic acid (C16:0)	0,5	0,8
Oleic acid (C18:1)	0,6	2,8
Linolenic acid (C18:3)	0,4	1,8

11 Interpretation of results

Table C.1 contains data on the range of fatty acid methyl ester compositions found on typical samples of oils extracted from palm, rapeseed and sunflower.

A significant difference in the composition of a sample from the values given in annex C may indicate the presence of esters derived from sources other than those given, or mixtures of FAME.

12 Test report

The test report shall contain at least the following information:

a) reference to this European Standard;

b) type and complete identification of the product tested;

c) result of the test (see clause 9);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) date of the test.

Annex A
(normative)

Summary of the conditions for analysis of fatty acid methyl esters by gas chromatography

A.1 Column, capillary type, impregnated with a stationary phase of polyethylene glycol type Carbowax 20M, DBwax or CPwax;

length :	30 m
internal diameter :	0,32 mm
film thickness :	0,25 µm

A.2 Sample injector, variable flow split injector, programmable flow rate type;

flow rate:	20 ml/min to 100 ml/min, according to type
temperature:	250 °C

A.3 Carrier gas, hydrogen or helium;

pressure:	30 kPa to 80 kPa

A.4 Oven;

isothermal temperature:	200 °C

A.5 Detector, flame ionization type;

temperature:	250 °C

A.6 Sample injection, size and split ratio to be simultaneously adjusted with the injected volume.