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Automotive fuels - Fatty acid methyl esters (FAME) for diesel engines - Requirements and test methods

Kraftstoffe für Kraftfahrzeuge - Fettsäure-Methylester (FAME) für Dieselmotoren - Anforderungen und Prüfverfahren

Carburants pour automobiles - Esters méthyliques d'acide gras (EMAG) pour moteurs diesel - Exigences et méthodes d'essais

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75.160.20 V^[\ æ\ [!ãæ Liquid fuels

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EUROPEAN STANDARD

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Automotive fuels - Fatty acid methyl esters (FAME) for diesel engines - Requirements and test methods

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This European Standard was approved by CEN on 14 February 2003.

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 Management Centre 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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document EN 14214:2003 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of an identical text or by endorsement, at the latest by January 2004, and conflicting national standards shall be withdrawn at the latest by **January 2004**.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This document has been prepared by CEN /TC 19, "Petroleum products, lubricants and related products".

This European standard exists in parallel with: EN 590 "Automotive fuels - Diesel - Requirements and test methods".

This standard gives all relevant characteristics, requirements and test methods for FAME, which are known at this time to be necessary to define the product to be used as automotive diesel fuel, including iodine value. The stability characteristics of FAME are under investigation in an EU-funded research programme 'BIOSTAB', and suitable limits and test methods may be incorporated into an amended version of this standard upon successful conclusion of this programme, including a possible replacement for iodine value.

Many of the test methods included in this standard were the subject of interlaboratory testing to determine the applicability of the method and its precision in relation to different sources of fatty acid methyl esters. These fatty acid methyl esters were produced from rapeseed and sunflower oil.

Annex A is normative and contains the precision data generated on the test methods which are the result of the interlaboratory testing as mentioned above, carried out by working groups of CEN/TC 19. Annex B and C, also normative, contain details on calculations.

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.

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1 Scope

This European Standard specifies requirements and test methods for marketed and delivered fatty acid methyl esters (FAME) to be used either as automotive fuel for diesel engines at 100% concentration, or as an extender for automotive fuel for diesel engines in accordance with the requirements of EN 590. At 100% concentration it is applicable to fuel for use in diesel engine vehicles designed or subsequently adapted to run on 100% FAME.

NOTE For the purposes of this European Standard, the term “% (m/m)” is used to represent the mass 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.

EN 116:1997, *Diesel and domestic heating fuels – Determination of cold filter plugging point*

EN 590:1999, *Automotive fuels - Diesel - Requirements and test methods*

EN 12662:1998, *Liquid petroleum products - Determination of contamination in middle distillates*

EN 14103:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of ester and linolenic acid methyl ester contents*

EN 14104:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) - Determination of acid value*

EN 14105:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of free and total glycerol and mono-, di- and triglyceride content –(Reference method)*

EN 14106:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME)– Determination of free glycerol content*

EN 14107:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of phosphorus content by inductively coupled plasma (ICP) emission spectrometry*

EN 14108:2003¹⁾, *Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) – Determination of sodium content by atomic absorption spectrometry*

EN 14109:2003¹⁾, *Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) – Determination of potassium content by atomic absorption spectrometry*

EN 14110:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) - Determination of methanol content*

EN 14111:2003¹⁾, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of iodine value*

EN 14112:2003¹⁾, *Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability (accelerated oxidation test).*

1) To be published

prEN 14538:2002 *Fat and oil derivatives – Fatty acid methyl esters (FAME) – Determination of Ca and Mg content by optical emission spectral analysis with inductively coupled plasma (ICP OES).*

EN ISO 2160:1998, *Petroleum products - Corrosiveness to copper - Copper strip test (ISO 2160:1998).*

EN ISO 3104:1996, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104:1994).*

EN ISO 3104:1998/C2:1999, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104:1997, including Cor. 2:1999)*

EN ISO 3170²⁾ *Petroleum liquids – Manual sampling.*

EN ISO 3171:1999, *Petroleum liquids – Automatic pipeline sampling (ISO 3171:1988).*

EN ISO 3675:1998, *Crude petroleum and liquid petroleum products - Laboratory determination of density - Hydrometer method (ISO 3675:1998).*

prEN ISO 3679:2002³⁾, *Determination of flash point - Rapid equilibrium closed cup method (ISO/DIS 3679:2002).*

EN ISO 4259:1995, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259:1992, including Cor. 1:1993).*

EN ISO 5165:1998, *Petroleum products - Diesel fuels - Determination of the ignition quality of diesel fuels - Cetane engine method (ISO 5165:1998).*

EN ISO 10370:1995, *Petroleum products - Determination of carbon residue - Micro method (ISO 10370:1993).*

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EN ISO 12185:1996/C1:2001, *Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185:1996, including Cor. 1:2001).*

EN ISO 12937:2000, *Petroleum products - Determination of water - Coulometric Karl Fisher titration method (ISO 12937:2000).*

EN ISO 13759:1996, *Petroleum products – Determination of alkyl nitrate in diesel fuels – Spectrometric method (ISO 13759:1996).*

prEN ISO 20846:2002, *Petroleum products – Determination of the sulfur content of automotive fuels – Energy-dispersive X-ray fluorescence spectrometry (ISO/DIS 20846:2002).*

prEN ISO 20884:2002, *Petroleum products – Determination of low sulfur content of automotive fuels – Wavelength-dispersive X-ray fluorescence spectrometry (ISO/DIS 20884:2002).*

ISO 3987:1994, *Petroleum products - Lubricating oils and additives - Determination of sulfated ash.*

ASTM D 1160:1999, *Distillation of Petroleum Products at Reduced Pressure.*

3 Sampling

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 automotive diesel fuel. The national

²⁾ To be published (revision of EN ISO 3170:1995)

³⁾ Revision of ISO 3679:1983

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requirements shall be set out in a national annex to this European Standard, either in detail or by reference only.

In view of the sensitivity of some of the test methods referred to in this European Standard, particular attention shall be paid to compliance with any guidance on sampling containers, which is included in the test method standard.

4 Pump marking

Information to be marked on dispensing pumps used for delivering FAME diesel fuel, and the dimensions of the mark shall be in accordance with the requirements of national standards or regulations for the marking of pumps for automotive diesel fuel. Such requirements shall be set out in detail or shall be referred to by reference in a national annex to this European Standard.

5 Requirements and test methods**5.1 Dyes and markers**

The use of dyes or markers is allowed.

5.2 Additives

In order to improve the performance quality, the use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount, to help to avoid deterioration of driveability and emissions control durability. Other technical means with equivalent effect may also be used.

NOTE Deposit forming tendency test methods suitable for routine control purposes have not yet been identified and developed.

5.3 Generally applicable requirements and related test methods

5.3.1 When tested by the methods indicated in Table 1, fatty acid methyl esters (FAME) shall be in accordance with the limits specified in Table 1. The test methods listed in Table 1 have been shown to be applicable to fatty acid methyl esters in an interlaboratory test programme. Precision data from this programme are given in normative Annex A, where these were found to be different from the precision data given in the test methods for petroleum products.

5.3.2 In case of a need for identification of FAME, a recommended method based on separation and characterisation of fatty acid methyl esters by LC/GC is EN 14331 [1].

5.3.3 In case of a need for a check upon FAME quality, iodine value of FAME may be calculated by the method presented in Annex B (normative), but this method does not constitute an alternative to the iodine value requirement of Table 1.

5.3.4 The limiting value for the carbon residue given in Table 1 is based on product prior to addition of ignition improver, if used. If a value exceeding the limit is obtained on finished fuel in the market, EN ISO 13759 shall be used as an indicator of the presence of a nitrate-containing compound. If an ignition improver is thus proved present, the limit value for the carbon residue of the product under test cannot be applied. The use of additives does not exempt the manufacturer from meeting the requirement of maximum 0,30 % (*m/m*) of carbon residue prior to addition of additives.

Table 1 - Generally applicable requirements and test methods

Property	Unit	Limits		Test method ^a
		minimum	maximum	
Ester content ^a	% (m/m)	96,5 ^b		EN 14103
Density at 15 °C ^c	kg/m ³	860	900	EN ISO 3675 EN ISO 12185
Viscosity at 40 °C ^d	mm ² /s	3,50	5,00	EN ISO 3104
Flash point	°C	120	–	prEN ISO 3679 ^e
Sulfur content	mg/kg	–	10,0	prEN ISO 20846 prEN ISO 20884
Carbon residue (on 10 % distillation residue) ^f	% (m/m)	–	0,30	EN ISO 10370
Cetane number ^g		51,0		EN ISO 5165
Sulfated ash content	% (m/m)	–	0,02	ISO 3987
Water content	mg/kg	–	500	EN ISO 12937
Total contamination ^h	mg/kg	–	24	EN 12662
Copper strip corrosion (3 h at 50 °C)	rating	class 1		EN ISO 2160
Oxidation stability, 110 °C	hours	6,0	–	EN 14112
Acid value	mg KOH/g		0,50	EN 14104
Iodine value	gr iodine/100 gr		120	EN 14111
Linolenic acid methyl ester	% (m/m)		12,0	EN 14103
Polyunsaturated (>= 4 double bonds) methyl esters ⁱ	% (m/m)		1	
Methanol content	% (m/m)		0,20	EN 14110
Monoglyceride content	% (m/m)		0,80	EN 14105
Diglyceride content	% (m/m)		0,20	EN 14105
Triglyceride content ^j	% (m/m)		0,20	EN 14105
Free glycerol ^l	% (m/m)		0,02	EN 14105 EN 14106
Total glycerol	% (m/m)		0,25	EN 14105
Group I metals (Na+K) ^k	mg/kg		5,0	EN 14108 EN 14109
Group II metals (Ca+Mg) ^l	mg/kg		5,0	prEN 14538
Phosphorus content	mg/kg		10,0	EN 14107

^a See 5.5.1
^b The addition of non-FAME components other than additives is not allowed, see 5.2.
^c Density may be measured by EN ISO 3675 over a range of temperatures from 20 °C to 60 °C. Temperature correction shall be made according to the formula given in Annex C. See also 5.5.2
^d If CFPP is -20 °C or lower, the viscosity measured at -20 °C shall not exceed 48 mm²/s. In this case, EN ISO 3104 is applicable without the precision data owing to non-Newtonian behaviour in a two-phase system.
^e A 2 ml sample and apparatus equipped with a thermal detection device shall be used
^f ASTM D 1160 shall be used to obtain the 10% distillation residue.
^g See 5.5.3.
^h Pending development of a suitable method by CEN/TC 19, EN 12662 shall be used. The precision of EN 12662 is however poor for FAME products
ⁱ Suitable test method to be developed
^j See also 5.5.1.
^k See 5.5.1. Method under development. See Annex A for precision data for sum of Na + K
^l Method under development. See Annex A for precision data for sum of Ca + Mg.