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Goriva za motorna vozila - Metilni estri maščobnih kislin (FAME) za dizelske motorje - Zahteve in preskusne metode

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

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

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

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EUROPEAN STANDARD
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English Version

Liquid petroleum products - Fatty acid methyl esters (FAME) for use in diesel engines and heating applications - Requirements and test methods

Produits pétroliers liquides - Esters méthyliques d'acides gras (EMAG) pour moteurs diesel et comme combustible de chauffage - Exigences et méthodes d'essai

Flüssige Mineralölerzeugnisse - Fettsäure-Methylester (FAME) zur Verwendung in Dieselmotoren und als Heizöl - Anforderungen und Prüfverfahren

This European Standard was approved by CEN on 20 July 2012.

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Contents

Page

Foreword	3
Introduction.....	5
1 Scope	6
2 Normative references	6
3 Sampling.....	8
4 Pump marking.....	8
5 Requirements and test methods.....	8
5.1 Dyes and markers.....	8
5.2 Additives.....	8
5.2.1 General	8
5.2.2 Oxidation stability enhancing additives.....	8
5.2.3 Non-FAME components.....	9
5.3 Generally applicable requirements and related test methods.....	9
5.4 Climate dependent requirements and related test methods.....	11
5.4.1 General	11
5.4.2 FAME being used at 100 % as fuel for diesel engines and heating applications	11
5.4.3 FAME being used as blend component in diesel fuel	12
5.5 Precision and dispute	13
Annex A (normative) Details of interlaboratory test programme.....	14
Annex B (normative) Correction factor for calculation of density of FAME.....	15
Annex C (informative) Background information for national climate grades selection	16
Bibliography.....	19

Foreword

This document (EN 14214:2012) has been prepared by Technical Committee CEN/TC 19 "Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin", 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 February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 14214:2008+A1:2009, EN 14213:2003 and EN 14213:2003/AC:2003.

This document has originally been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

This document exists in parallel with EN 590.

Significant technical changes between this European Standard and the previous edition are:

- extension towards distillate heating fuels. The former specification for FAME for heating fuel applications (EN 14213) also needed to be aligned, but effectively only one quality was supplied and required by heating fuel suppliers in the European market.
- an update of the specification in the perspective of blending at up to 10 % (V/V) of FAME in automotive diesel fuel;
- discrimination between climate requirements for use for 100 % as fuel for diesel engines and for use as blend component in diesel fuel. This is done by introducing a new Table 3 for seasonal grades to be set nationally. It thus requires countries to present two sets of seasonal choices in a national annex to this standard;
- introduction of additional requirements for FAME for use as a blending component as a first, intermediate, step towards solving precipitation problems observed in the market during cold periods. Further work towards limitation of impurities, more specifically steryl-glycosides, and regarding monoglyceride determination is on-going. A performance test, such as a filterability test, is anticipated to solve this issue in the longer term;
- a decrease of the monoglycerides content limit from 0,8 % (*m/m*) to 0,7 % (*m/m*);
- deletion of the requirement on carbon residue as it is no longer considered necessary;
- an increase of the oxidation stability requirement from 6 h minimum to 8 h minimum;
- introduction of additionally developed test methods for iodine value, phosphorus content and sulfur content, EN 16300, FprEN 16294 and EN ISO 13032, respectively. Plus updating towards revised sulfur and sulfated ash content determination test methods;
- updating of test methods on esters, (mono) glycerides, polyunsaturated fatty acids and stability characteristics of FAME, resulting from work under CEN/TC 19 and in cooperation with CEN/TC 307, either as final publication or as technically assessed and accepted draft texts;

EN 14214:2012 (E)

- combination of all sub-clauses dealing with additives in one and aligning them with similar requirements in EN 590.
- a re-evaluation of Table A.1 has been executed and indicated that FAME conforms to the current precision statements of EN 116 and therefore these row has been deleted from Table A.1.

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

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Introduction

This European Standard gives all relevant characteristics, requirements and test methods for fatty acid methyl esters (FAME), which are known at this time to be necessary to define the product to be used as automotive diesel fuel and in heating applications.

Many of the test methods included in this European Standard have been the subject of several interlaboratory testing to determine their applicability and their precision in relation to different sources of FAME. These FAMEs were produced from vegetable oils available in the market at the time, mainly rapeseed, palm, soy and sunflower oil. This does not imply any restrictions towards the feedstock for the fats and oils from which the FAME is made to comply with this European Standard (see Foreword).

Concerning total contamination, an interlaboratory study with field samples, following a study with artificial samples, is pending and therefore the repeatability and reproducibility of EN 12662 have not yet been fully established. The precision for FAME is investigated.

Concerning cold temperature properties of diesel blends related to the quality of the FAME used as a blending component, strong indications towards the relationship with saturated mono-glycerides and steryl-glycosides have become known. As no test method to detect those components separately has yet been developed, an interim solution in setting CFPP and cloud point limitations has been included in this standard (see Table 3). A study within CEN has indicated that FAME conforms to the current precision statements of EN 116 and EN 23015.

Although there are technical indications that iodine value can be removed as an indication for FAME stability, relaxation at this time would be premature. Until the stability safeguards introduced by this revision have been proven in the market, it is maintained, also preventing unnecessarily high usage of anti-oxidant additives.

In order to meet the needs of the latest technology engines, lowering of the phosphorus limit and the limit of sodium and potassium is under investigation.

EN 14214:2012 (E)

1 Scope

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

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

2 Normative references

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

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

EN 590, *Automotive fuels — Diesel — Requirements and test methods*

EN 12662:2008, *Liquid petroleum products — Determination of contamination in middle distillates*

EN 14103:2011, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of ester and linolenic acid methyl ester contents*

EN 14104:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of acid value*

EN 14105:2011, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of free and total glycerol and mono-, di-, triglyceride contents*

EN 14106:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of free glycerol content*

EN 14107:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of phosphorus content by inductively coupled plasma (ICP) emission spectrometry*

EN 14108:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of sodium content by atomic absorption spectrometry*

EN 14109:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of potassium content by atomic absorption spectrometry*

EN 14110:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of methanol content*

EN 14111:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of iodine value*

EN 14112:2003, *Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of oxidation stability (accelerated oxidation test)*

EN 14538:2006, *Fat and oil derivatives — Fatty acid methyl esters (FAME) — Determination of Ca, K, Mg and Na content by optical emission spectral analysis with inductively coupled plasma (ICP OES)*

EN 15751:2009, *Automotive fuels — Fatty acid methyl ester (FAME) fuel and blends with diesel fuel — Determination of oxidation stability by accelerated oxidation method*

EN 15779:2009, *Petroleum products and fat and oil derivatives — Fatty acid methyl esters (FAME) for diesel engines — Determination of polyunsaturated (≥ 4 double bonds) fatty acid methyl esters (PUFA) by gas chromatography*

FprEN 16294:2011¹⁾, *Petroleum products and fat and oil derivatives — Determination of phosphorus content in fatty acid methyl esters (FAME) — Optical emission spectral analysis with inductively coupled plasma (ICP OES)*

EN 16300:2012²⁾, *Automotive fuels — Determination of iodine value — Calculation method from gas chromatographic data*

EN 23015:1994, *Petroleum products — Determination of cloud point (ISO 3015:1992)*

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

EN ISO 2719:2002, *Determination of flash point — Pensky-Martens closed cup method (ISO 2719:2002)*

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

EN ISO 3170:2004, *Petroleum liquids — Manual sampling (ISO 3170:2004)*

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)*

EN ISO 3679:2004, *Determination of flash point — Rapid equilibrium closed cup method (ISO 3679:2004)*

EN ISO 4259:2006, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259:2006)*

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

EN ISO 12185:1996, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method (ISO 12185:1996)*

EN ISO 12937:2000, *Petroleum products — Determination of water — Coulometric Karl Fischer titration method (ISO 12937:2000)*

EN ISO 13032:2012, *Petroleum products — Determination of low concentration of sulfur in automotive fuels — Energy-dispersive X-ray fluorescence spectrometric method (ISO 13032:2012)*

EN ISO 20846:2011, *Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method (ISO 20846:2011)*

EN ISO 20884:2011, *Petroleum products — Determination of sulfur content of automotive fuels — Wavelength-dispersive X-ray fluorescence spectrometry (ISO 20884:2011)*

ISO 3987:2010, *Petroleum products — Determination of sulfated ash in lubricating oils and additives*

1) At draft stage.

2) To be published.

EN 14214:2012 (E)

DIN 51900-2, *Testing of solid and liquid fuels - Determination of the gross calorific value by the bomb calorimeter and calculation of the net calorific value - Part 2: Method using isoperibol or static jacket calorimeter*

DIN 51900-3, *Testing of solid and liquid fuels - Determination of gross calorific value by the bomb calorimeter and calculation of net calorific value - Part 3: Method using adiabatic jacket*

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 or heating fuel. The national 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 respective 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**5.2.1 General**

In order to improve performance, the use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount to help 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.

5.2.2 Oxidation stability enhancing additives

In order to improve the oxidation stability of FAME, it is strongly recommended to add oxidation stability enhancing additives to FAME at the production stage and before storage, providing an oxidation stability similar to that obtained with 1 000 mg/kg of 2,6-di-tert-butyl-4-hydroxytoluene (BHT).

CAUTION — There is a potential risk of precipitate formation with oxidation stability enhancing additives at low temperatures in low aromatic arctic fuel. Caution should therefore be taken in the choice of oxidation stability enhancing additives to arctic grade FAME.

5.2.3 Non-FAME components

The addition of non-FAME components (other than additives) such as diesel or other hydrocarbons prior to testing FAME for compliance with the EN 14214 specification is not permitted.

5.3 Generally applicable requirements and related test methods

5.3.1 When tested by the methods indicated in Table 1, 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 FAME in an interlaboratory test programme. Precision data from this programme are given in 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 in a hydrocarbon blend, the recommended method based on separation and characterisation of FAME by LC/GC is EN 14331 [1].

5.3.3 FAME shall be free from any adulterant or contaminant that may render the fuel unacceptable for use in diesel engine vehicles and heating applications.

NOTE For further information on preventing contamination by water or sediment that may occur in the supply chain it is advisable to check CEN/TR 15367-1 [2].

5.3.4 When used for heating fuel purposes, either at 100 % or as blend component, FAME shall have a minimum net calorific value of 35 MJ/kg determined by DIN 51900-2 or DIN 51900-3.

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