

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

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 15779:2009 sisaldab Euroopa standardi EN 15779:2009 ingliskeelset teksti.

Standard on kinnitatud Eesti Standardikeskuse 31.12.2009 käskkirjaga ja jõustub sellekohase teate avaldamisel EVS Teatajas.

Euroopa standardimisorganisatsioonide poolt rahvuslikele liikmetele Euroopa standardi teksti kättesaadavaks tegemise kuupäev on 07.10.2009.

Standard on kättesaadav Eesti standardiorganisatsioonist.

This Estonian standard EVS-EN 15779:2009 consists of the English text of the European standard EN 15779:2009.

This standard is ratified with the order of Estonian Centre for Standardisation dated 31.12.2009 and is endorsed with the notification published in the official bulletin of the Estonian national standardisation organisation.

Date of Availability of the European standard text 07.10.2009.

The standard is available from Estonian standardisation organisation.

ICS 75.160.20

Standardite reprodutseerimis- ja levitamiseõigus kuulub Eesti Standardikeskusele

Andmete paljundamine, taastekitamine, kopeerimine, salvestamine elektroonilisse süsteemi või edastamine ükskõik millises vormis või millisel teel on keelatud ilma Eesti Standardikeskuse poolt antud kirjaliku loata.

Kui Teil on küsimusi standardite autorikaitse kohta, palun võtke ühendust Eesti Standardikeskusega:
Aru 10 Tallinn 10317 Eesti; www.evs.ee; Telefon: 605 5050; E-post: info@evs.ee

Right to reproduce and distribute Estonian Standards belongs to the Estonian Centre for Standardisation

No part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying, without permission in writing from Estonian Centre for Standardisation.

If you have any questions about standards copyright, please contact Estonian Centre for Standardisation:
Aru str 10 Tallinn 10317 Estonia; www.evs.ee; Phone: +372 605 5050; E-mail: info@evs.ee

ICS 75.160.20

English Version

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

Produits pétroliers et produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) pour moteurs diesel (gazole) - Détermination de la teneur en esters méthyliques d'acides gras polyinsaturés (≥ 4 doubles liaisons) (PUFA) par chromatographie en phase gazeuse

Mineralölzeugnisse und Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäuremethylester (FAME) für Dieselmotoren - Bestimmung von mehrfach ungesättigten (≥ 4 Doppelbindungen) Fettsäuremethylestern (PUFA) mittels Gaschromatografie

This European Standard was approved by CEN on 22 September 2009.

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

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



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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

Foreword.....	3
Introduction	4
1 Scope	5
2 Normative references	5
3 Principle.....	5
4 Apparatus	5
5 Reagents and materials	6
6 Sampling	6
7 Procedure	6
8 Calculation.....	7
9 Expression of results	8
10 Precision	8
10.1 General.....	8
10.2 Repeatability, r	8
10.3 Reproducibility, R	9
11 Test report	9
Bibliography	10

Foreword

This document (EN 15779:2009) 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 April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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.

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

Introduction

Polyunsaturated fatty acid methyl esters are considered as a critical component in FAME since they show a low stability against oxidation and polymerization reactions. The specification on polyunsaturated ester content is needed in FAME and biodiesel products to limit the content of polyunsaturated FAME with more than three double bonds. At the time of the first FAME fuel specifications no test method was available for such a complicated determination in terms of identification and quantification, so technical work has been done in a joint working group with CEN/TC 307 before any standardisation steps could be taken.

The method has been prepared by the partners of the project "BIOScopes" (Lot 1, Task a) funded by the European Commission, DG TREN, with the purpose to execute a Pan-European round robin test to determine the precision data and the usability of this new and other revised determination methods for FAME.

1 Scope

This European Standard specifies a method for the determination of the polyunsaturated (≥ 4 double bonds) fatty acid (PUFA) methyl esters content of fatty acid methyl ester (FAME) as a whole between 0,6 % (*m/m*) and 1,5 % (*m/m*).

The method covers the predominant four polyunsaturated fatty acid methyl esters of eicosatetraenoic acid (C 20:4 (n-6)), eicosapentaenoic acid (C 20:5 (n-3)), docosapentaenoic acid (C 22:5 (n-3)), and docosahexaenoic acid (C 22:6 (n-3)).

Studies have indicated that based on the linearity of results from this European Standard, PUFA methyl esters can be determined in FAME in the range between 0,3 % (*m/m*) to 3,0 % (*m/m*). However, the precision was not established in that range, as no samples within the upper ranges were included in the final interlaboratory test (see 10.1).

Although the method is applicable to all uses, it is predominantly for FAME for use in diesel engines.

NOTE 1 For the purposes of this document, the term “% (*m/m*)” is used to represent the mass fraction of a material.

NOTE 2 This European Standard is based on A.O.C.S Official Method Ce 1b-89 [1].

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.

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

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

3 Principle

Determination of the percentage of polyunsaturated (≥ 4 double bonds) fatty acid (PUFA) methyl ester present in FAME is done by gas chromatography/FID detection using internal calibration with C 23:0 methyl ester. The theoretical detector correction factors relative to C 23:0 internal standard for different poly-unsaturated ester types are applied to the analytical data for optimum accuracy.

4 Apparatus

4.1 Capable gas chromatograph, consisting of a capillary injection system (preferable split mode at a split ratio of 1:50), a flame ionization FID detector and the following:

4.1.1 Injector, temperature 220 °C.

4.1.2 Detector, temperature 275 °C.

4.1.3 Oven temperature profile, initial temperature 150 °C, initial hold time 1 min; program rate 15 °C/min up to 200 °C; 2 °C/min up to 250 °C final temperature.