

**Petroleum products and fat and oil derivatives - Fatty acid methyl esters (FAME) for diesel engines - Determination of polyunsaturated ( $\geq 4$  double bonds) fatty acid methyl esters (PUFA) by gas chromatography**

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

See Eesti standard EVS-EN 15779:2009+A1:2013 sisaldab Euroopa standardi EN 15779:2009+A1:2013 ingliskeelset teksti.	This Estonian standard EVS-EN 15779:2009+A1:2013 consists of the English text of the European standard EN 15779:2009+A1:2013.
Standard on jõustunud sellekohase teate avaldamisega EVS Teatajas.	This standard has been endorsed with a notification published in the official bulletin of the Estonian Centre for Standardisation.
Euroopa standardimisorganisatsioonid on teinud Euroopa standardi rahvuslikele liikmetele kättesaadavaks 25.09.2013.	Date of Availability of the European standard is 25.09.2013.
Standard on kättesaadav Eesti Standardikeskusest.	The standard is available from the Estonian Centre for Standardisation.

Tagasisidet standardi sisu kohta on võimalik edastada, kasutades EVS-i veebilehel asuvat tagasiside vormi või saates e-kirja meiliaadressile [standardiosakond@evs.ee](mailto:standardiosakond@evs.ee).

ICS 75.160.20

### Standardite reprodutseerimise ja levitamise õigus kuulub Eesti Standardikeskusele

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

Kui Teil on küsimusi standardite autorikaitse kohta, võtke palun ühendust Eesti Standardikeskusega:  
Aru 10, 10317 Tallinn, Eesti; [www.evs.ee](http://www.evs.ee); telefon 605 5050; e-post [info@evs.ee](mailto:info@evs.ee)

### The right to reproduce and distribute 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 a written permission from the Estonian Centre for Standardisation.

If you have any questions about copyright, please contact Estonian Centre for Standardisation:  
Aru 10, 10317 Tallinn, Estonia; [www.evs.ee](http://www.evs.ee); phone 605 5050; e-mail [info@evs.ee](mailto:info@evs.ee)

English Version

Petroleum products and fat and oil derivatives - Fatty acid methyl esters (FAME) for diesel engines - Determination of polyunsaturated ( $\geq 4$  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 ( $\geq 4$  doubles liaisons) (PUFA) par chromatographie en phase gazeuse

Mineralölerzeugnisse und Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) für Dieselmotoren - Bestimmung von mehrfach ungesättigten ( $\geq 4$  Doppelbindungen) Fettsäuremethylestern (PUFA) mittels Gaschromatographie

This European Standard was approved by CEN on 22 September 2009 and includes Amendment 1 approved by CEN on 5 August 2013.

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

CEN members are the national standards bodies of 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 United Kingdom.



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

CEN-CENELEC 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 .....	7
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+A1:2013) 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 March 2014, and conflicting national standards shall be withdrawn at the latest by March 2014.

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 includes Amendment 1 approved by CEN on 5 August 2013.

This document supersedes EN 15779:2009.

The start and finish of text introduced or altered by amendment is indicated in the text by tags **A1** **A1**.

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.

## 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 ( $\geq 4$  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

**[A1]** 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. **[A1]**

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 ( $\geq 4$  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.

**4.1.4 Capillary column**, fused silica; 30 m in length, 0,25  $\mu\text{m}$  film thickness and 0,20 mm to 0,32 mm internal diameter. The liquid phase shall be bonded Carbowax or an equivalent polyethylene glycol type.