



BSI Standards Publication

Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of ester and linolenic acid methyl ester contents

National foreword

This British Standard is the UK implementation of EN 14103:2020. It supersedes BS EN 14103:2011, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/307, Oilseeds, animal and vegetable fats and oils and their by-products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Date	Text affected
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English Version

**Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) -
Determination of ester and linolenic acid methyl ester
contents**

Produits dérivés des corps gras - Esters méthyliques
d'acides gras (EMAG) - Détermination de la teneur en
ester et en ester méthylique de l'acide linoléique

Erzeugnisse aus pflanzlichen und tierischen Fetten und
Ölen - Fettsäure-Methylester (FAME) - Bestimmung
des Ester-Gehaltes und des Gehaltes an Linolensäure-
Methylester

This European Standard was approved by CEN on 18 November 2019.

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Contents

Page

European foreword	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions	4
4 Principle	4
5 Reagents	5
6 Apparatus	5
7 Sampling.....	5
8 Procedure	5
8.1 Operating conditions	5
8.2 Internal standard GC purity determination	6
8.3 FAME sample preparation and analysis	6
8.4 Identification	7
9 Calculation.....	7
9.1 Determination of ester content	7
9.2 Determination of linolenic acid methyl ester.....	10
9.3 Expression of results	10
10 Precision	10
10.1 General.....	10
10.2 Repeatability	10
10.3 Reproducibility.....	11
11 Test report.....	11
Annex A (informative) Chromatograms of FAME samples - Determination of ester content and linolenic acid methyl ester content – Summary of the analytical conditions.....	12
Annex B (informative) Results of an interlaboratory trial	16
Annex C (informative) Calculation of the pattern of fatty acid methyl esters.....	18
Bibliography	19

European foreword

This document (EN 14103:2020) has been prepared by Technical Committee CEN/TC 307 “Oilseeds, vegetable and animal fats and oils and their by-products — Methods of sampling and analysis”, the secretariat of which is held by AFNOR.

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 July 2020, and conflicting national standards shall be withdrawn at the latest by July 2020.

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

This document supersedes EN 14103:2011.

In comparison with the previous edition, the following technical modifications have been made:

- a) note on natural nonadecanoic acid methyl ester added in the scope;
- b) new procedure to check nonadecanoic acid methyl ester purity, with new GC conditions, and reduction of the minimum GC purity (99,5 to 99,0 % (*m/m*));
- c) calculation of results revised by incorporation of theoretical flame ionization detector correction factor (TCF), this gives a better accuracy of the calculated contents in case of presence of methyl esters with short chains;
- d) new inter-laboratories study (ILS) conducted and precision adopted;
- e) new sample chromatograms recorded and added;
- f) calculation of the pattern of fatty acid methyl esters incorporated as informative Annex C;
- g) modification of the way of integration in 8.4 by taking all the peaks into consideration whereas in the previous edition all the peaks identified as fatty acid methyl esters were taken into consideration;
- h) increase of the FAME sample test portion in 8.3 to 250 mg whereas in the previous edition the sample test portion was 100 mg;
- i) document revised editorially.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

The purpose of this document is to describe a procedure for the determination of the ester content in fatty acid methyl esters (FAME) intended for incorporation into diesel oil. It also allows determining the linolenic acid methyl ester content. It allows verifying that the ester content of FAME is greater than 90 % (*m/m*) and that the linolenic acid methyl ester content is between 1 % (*m/m*) and 15 % (*m/m*).

The precision was established using FAMEs with an ester content of 95 % (*m/m*) and 100 % (*m/m*) only, thus covering the range of the limit value. The method is also suitable outside of this range; however, precision for lower concentrations is subject to further work.

This method is suitable for FAME which contains methyl esters between C6 and C24.

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

This method was elaborated for FAME samples from usual raw material. For FAME sample from unidentified raw material, a solution of the test sample is prepared without any internal standard addition, in order to verify the absence of natural nonadecanoic acid methyl ester or other unknown substances co-eluting with the IS.

NOTE 2 The calculation method of the pattern of fatty acid methyl esters is given in Annex C.

WARNING — The use of this method may involve hazardous equipment, materials and operations. This method does not purport to address to all of the safety problems associated with its use, but it is the responsibility of the user to search and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

Determination of the mass percentage of total methyl esters of fatty acids and the mass percentage of linolenic acid methyl ester present in the sample, by gas chromatography according to a procedure using internal calibration (nonadecanoic acid methyl ester).

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

5.1 Toluene, analytical grade, no impurities of toluene eluting after methyl ester C6:0.

5.2 Nonadecanoic acid methyl ester (FAME C19:0), GC purity, min. 99,0 % (*m/m*).

GC Purity of nonadecanoic acid methyl ester shall be checked for every new lot according to the procedure described in 8.2.

Standard shall be kept at room temperature in a dry storage (e.g. desiccator) in order to limit its water absorption. Its water content shall be below 0,2 % (*m/m*) and verified by Karl Fischer determination when a new lot of standard is opened.

If the purity is lower than 99,0 % (*m/m*) do not use it for this determination.

5.3 Carrier gas, hydrogen or helium, 99,999 5 % pure or better, gas chromatography quality, dried, oxygen removed by suitable filters (<0,1 mg/kg), free from organic impurities.

5.4 Auxiliary gases, hydrogen and air, gas chromatography quality, free from organic impurities.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Gas chromatograph, equipped with a variable split flow injector or equivalent device, a temperature programmable oven and a flame ionization detector.

6.2 Capillary column, coated with polyethylene glycol (Carbowax 20M) stationary phase, the following characteristics have been found suitable: length: 30 m, internal diameter: 0,25 mm, film thickness: 0,25 µm.

Stationary phase other than polyethylene glycol should be tested first before being selected as co-elution between the internal standard (FAME C19:0) and other fatty acid methyl esters may exist.

NOTE Indeed, there is a co-elution between FAME C19:0 and linoleic acid methyl ester (FAME C18:2) when using a column with a stationary phase such as 70 % cyanopropyl-polysilphenylene-siloxane (BPX70).

6.3 Glass mono-use tubes, equipped with plastic mono-use stopper, 10 ml capacity.

6.4 Pipette, 10 ml capacity.

6.5 Analytical balance, accuracy $\pm 0,1$ mg.

7 Sampling

Sampling is not part of the method specified in this European Standard. A recommended sampling method is given in EN ISO 5555 [1].

8 Procedure

8.1 Operating conditions

The chromatographic analysis conditions will be chosen taking into account the characteristics of the column being used and the type of carrier gas (hydrogen or helium).