SLOVENSKI STANDARD
SIST EN 14110:2019

01-julij-2019

Nadomešča:
SIST EN 14110:2003

Derivati maščob in olj - Metilni estri maščobnih kislin (FAME) - Določevanje metanola

Fat and oil derivatives - Fatty Acid Methyl Esters - Determination of methanol content

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Methanolgehaltes

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Produits dérivés des corps gras - Esters méthyliques d'acides gras - Détermination de la teneur en méthanol

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Ta slovenski standard je istoveten z: **EN 14110:2019**

ICS:

67.200.10	Rastlinske in živalske maščobe in olja	Animal and vegetable fats and oils

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14110

April 2019

ICS 67.200.10

Supersedes EN 14110:2003

English Version

Fat and oil derivatives - Fatty Acid Methyl Esters - Determination of methanol content

Produits dérivés des corps gras - Esters méthyliques d'acides gras - Détermination de la teneur en méthanol

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung des Methanolgehaltes

This European Standard was approved by CEN on 13 December 2019.

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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.

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 14110:2019) 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of an identical text or by endorsement, at the latest by October 2019, and conflicting national standards shall be withdrawn at the latest by October 2019.

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 14110:2003.

This document has been prepared under mandate M/245 on Fatty Acid Methyl Ester (FAME) given to CEN by the European Commission and the European Free Trade Association.

Significant changes between this document and EN 14110:2003 are:

— Addition of Formula (1) — resolution between methanol and 2-propanol

— Correction of the Formula to calculate the methanol content based on external calibration

— Addition of Clause 2 — Normative References

— Addition of Clause 7 — Sampling

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1 Scope

This document specifies a method for the determination of the methanol content of fatty acid methyl esters (FAME) for use as diesel fuel and domestic heating fuel. The method is applicable to methanol contents between 0,01 % (*m*/*m*) and 0,5 % (*m*/*m*). The method is not applicable to mixtures of FAME containing other low boiling components.

NOTE For the purposes of this document, the terms "% (*m*/*m*)" and "% (*V*/*V*)" are used to represent respectively the mass fraction and the volume fraction.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of the standard, and fulfil statutory and regulatory requirements for this purpose.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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)*

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

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3 Terms and definitions

No terms and definitions are listed in this document.

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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

The sample is heated at 80 °C in a hermetically sealed vial to allow desorption of contained methanol into the gas phase. When the equilibrium is reached a defined part of the gas phase is injected into a gas chromatograph, where methanol is detected with a flame ionization detector.

The amount of methanol can be determined either by internal calibration (procedure A) or by external calibration (procedure B).

If only manual equipment is available then only internal standard calibration should be used.

5 Reagents

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

5.1 Methanol, of known purity greater than 99,5 %.

5.2 2-propanol, of known purity, greater than 99,5 % (for procedure A, internal calibration).

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5.3 Reference FAME, with a methanol content less than 0,001 % (*m/m*).

Reference FAME can either be obtained from commercial sources or by washing FAME three to five times with distilled water in a separator funnel. FAME should be then dried by heating at 90 °C under stirring and reduced pressure.

5.4 Carrier gas, nitrogen, helium or hydrogen, of know purity greater than 99 %.

6 Apparatus

6.1 Head space vials, 20 ml capacity.

6.2 Inert septa (e.g. TFE or Viton [1])) **and metallic caps.**

6.3 Syringe, 10 µl, accurate to 0,1 µl.

6.4 Gas syringe 500 µl, fitted with a valve (for manual procedure).

6.5 Crimping pliers.

6.6 Pipettes, of 1 ml, 2 ml, 5 ml capacity.

6.7 Volumetric flasks, capacity 10 ml and 25 ml.

6.8 Gas chromatograph, equipped with a capillary column, suitable injector (automatic headspace system or a split/splitless injector) and flame ionization detector, integrator.

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6.9 Capillary column, the column shall elute methanol as a symmetrical peak. Stationary phases like methylpolysiloxane (e.g. DB1 [2]), SE30 [2])) or polyethylenglycol (e.g. DBWAX [2]), CARBOWAX [2])) can be used successfully and a film thickness of minimum 0,5 µm is recommended.

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The use of a packed column equipped by one of the above mentioned stationary phases or of Chromosorb 101[2]) is also allowed.

6.10 Automatic headspace equipment

The automatic headspace equipment used shall have an accuracy of 1 % or better concerning experimental conditions like equilibrium temperature, heating times, and headspace sampling volume. This can be checked, if necessary, by repeated analysis of the same sample.

Automatic headspace equipment is recommended because apart from its better repeatability it allows for automated and fast analysis using external calibration. Manual equipment can also be used, however, extreme care should be taken when gas volumes are sampled manually from the vials and are injected manually.

6.11 Analytical balance, with an accuracy of ± 0,1 mg.

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6.12 Thermostatically controlled bath or oven, capable of heating up to 80 °C. The exact temperature of the bath or oven has no influence on the test result as long as it is the same when running the calibration and sample sequence, because the temperature dependent equilibrium of gaseous and liquid phase will be the same in measurement and calibration. Since it is technically not possible to verify the temperature by direct measurement, the verification may be conducted by proof of concept, e. g. via measurement of a sample for quality assurance.

6.13 Thermostatically controlled oven, capable of heating up to 60 °C. The exact temperature of the oven has no influence on the test result as long as it is the same when running the calibration and sample sequence, because the temperature dependent equilibrium of gaseous and liquid phase will be the same in measurement and calibration. Since it is technically not possible to verify the temperature by direct measurement, the verification may be conducted by proof of concept, e. g. via measurement of a sample for quality assurance.

7 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 petroleum products.

8 Operation conditions

8.1 Analytical conditions

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The GC operating conditions shall be chosen taking into account the characteristics of the column and the type of carrier gas in order to reach the desired resolution of minimum 1,5 between the methanol and 2-propanol peaks.

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The resolution, *Rs*, between methanol and 2-propanol is calculated using Formula (1):

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$$Rs = 1,18\frac{t_B - t_A}{w_A + w_B} \quad (1)$$

where

t_A is the retention time of methanol;

t_B is the retention time of 2-propanol;

w_A is the peak width at half-height of methanol;

w_B is the peak width at half-height of 2-propanol;

Ensure that values for retention time and peak width are in the same units.

For an example of a chromatogram see Annex A.

NOTE 1 The following parameters are given as an example:

— column DB1 (length = 30 m, internal diameter = 0,32 mm, film thickness = 3 µm);

— split injector flow rate: 50 ml/min;

— injector and detector temperature: 150 °C;

— oven temperature: 50 °C;

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— carrier gas (hydrogen) pressure: 40 kPa;

— volume injected: 500 µl.

NOTE 2 The following head space sampling conditions are given as an example:

— equilibrium temperature: 80 °C;

— equilibrium time: 45 min;

— sampling volume: 500 µl.

8.2 Operation

The gas chromatograph shall be set up and operated according to the manufacturer's instructions.

9 Calibration solutions

9.1 Three calibration solutions with approximately the following methanol concentrations in FAME (5.3) shall be prepared as described below.

The exact mass shall be determined by weight measurement. It is necessary to ensure thorough mixing by vigorous shaking.

NOTE Three calibration solutions have proven sufficient in daily practice, covering the scope of this method. For other concentration ranges more and/or different calibration solutions can be chosen.

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9.2 Calibration solution A (0,5 % (*m*/*m*) methanol in FAME).

Fill a 25 ml volumetric flask (6.7) with 25 ml of FAME (5.3) and add (112 ± 0,1) mg (142 µl) methanol (5.1) into the liquid phase using a syringe (6.3). The exact masses shall be determined by weight measurement. It is necessary to ensure thorough mixing by vigorous shaking.

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9.3 Calibration solution B (0,1 % (*m*/*m*) methanol in FAME).

Transfer 5 ml of calibration solution A into a 25 ml volumetric flask (6.7) and carefully fill to the mark with FAME (5.3).

9.4 Calibration solution C (0,01 % (*m*/*m*) methanol in FAME).

Transfer 1 ml of calibration solution B into a 10 ml volumetric flask (6.7) and fill to the mark with FAME (5.3).

10 Procedure

10.1 General

Two alternative procedures, the first using internal calibration and the second using external calibration are described in 10.2 and 10.3, respectively.

10.2 Procedure A — Internal calibration

10.2.1 General

This procedure is generally preferred when only a small number of samples is analysed and when automatic head space equipment is not available. For manual procedure, see information in 10.2.2.