SLOVENSKI STANDARD
SIST EN 17057:2018

01-april-2018

Goriva za motorna vozila ter maščobni in oljni derivati - Določevanje nasičenih monogliceridov v metilnih estrih maščobnih kislin (FAME) - Metoda z GC-FID

Automotive fuels and fat and oil derivates - Determination of saturated monoglycerides content in Fatty Acid methyl Esters (FAME) - Method by GC-FID

Autoraftstoffe und erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Bestimmung des Gehalts von gesättigter Monoglyceride in Fettsäure-Methylester (FAME) - Methode mittels GC-FID

Combustibles et produits dérivés des corps gras - Détermination de la teneur des monoglycerides saturés en esters méthyliques d'acides gras (EMAG) - Méthode par GC-FID

Ta slovenski standard je istoveten z: **EN 17057:2018**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

SIST EN 17057:2018 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 17057

January 2018

ICS 75.160.20

English Version

Automotive fuels and fat and oil derivates - Determination of saturated monoglycerides content in Fatty Acid Methyl Esters (FAME) - Method by GC-FID

Carburants pour automobiles et produits dérivés des corps gras - Détermination de la teneur en monoglycérides saturés (SMG) des esters méthyliques d'acides gras (EMAG) - Méthode par GC-FID

Kraftstoffe und Erzeugnisse aus Fetten und Ölen - Bestimmung des Gehalts an gesättigten Monoglyceriden in Fettsäure-Methylester (FAME) - Verfahren mit GC-FID

This European Standard was approved by CEN on 19 November 2017.

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

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Ref. No. EN 17057:2018 E

Contents

Page

European foreword

This document (EN 17057:2018) 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of an identical text or by endorsement, at the latest by July 2018 and conflicting national standards shall be withdrawn at the latest by July 2018.

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1 Scope

This document specifies a method to determine the saturated monoglyceride content in Fatty Acid Methyl Esters (FAME). This method only identifies and quantifies the following saturated monoglycerides: 1-C16:0, 2-C16:0 and 1-C18:0. The total saturated monoglyceride content is calculated by the summation of the contents of these three saturated monoglycerides. The precision has been established for FAMEs having saturated monoglycerides in the (200 to 1 500) mg/kg range.

This method is not suitable for FAME produced from or containing coconut and palm kernel oil derivatives because of overlapping of various peaks.

NOTE This Standard determines only three saturated monoglycerides, i.e. 1-C16:0, 2-C16:0 and 1-C18:0. FAMEs can contain also other saturated monoglycerides such as 1-C17:0, but these are generally much lower than the three targeted saturated monoglycerides and are therefore not included in the Standard's scope.

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard 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, 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 ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

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

3 Principle

The saturated monoglycerides are converted into more stable and volatile silyl derivatives in the presence of pyridine and N-methyl-N-trimethylsilyltrifluoroacetamide (MSTFA).

After silylation, the sample is analysed by gas chromatography on a capillary column with thin film thickness, an on-column injector or equivalent device (e.g. programmed-temperature vaporizing injector), and a flame ionization detector.

The quantification of the saturated monoglycerides is performed against the internal standard (IS) Glyceryl monononadecanoate (mono C19), using a relative response factor of 1,00.

4 Reagents and materials

Use only reagents of recognized analytical grade. The reagents should not contain components that chromatographically co-elute with the target components, i.e. 1-C16:0, 2-C16:0 and 1-C18:0.

NOTE Annex A shows a list of suppliers for saturated monoglyceride standards.

4.1 N-methyl-N-trimethysilyltrifluoroacetamide (MSTFA) [CAS: 24589-78-4], p.a.

4.2 Pyridine, [CAS: 110-86-1], max. 0,1 % water, stored on molecular sieve.

4.3 n-Heptane, [CAS: 142-82-5], p.a.

4.4 2-Hexadecanoyl-glycerol ($2 - C_{16:0}$)**, [CAS: 23470-00-0],** of > 99,0 % purity.

4.5 1-Hexadecanoyl-rac-glycerol ($1 - C_{16:0}$)**, [CAS: 542-44-9],** of > 99,0 % purity.

4.6 1-Octadecanoyl-rac-glycerol ($1 - C_{18:0}$)**, [CAS: 123-94-4],** of > 99,0 % purity.

4.7 1-Nonadecanoyl-rac-glycerol ($1 - C_{19:0}$)**, [CAS: 112340-30-4],** of > 99,0 % purity.

4.8 Squalene [CAS: 111-02-4] ((6E,10E,14E,18E)-2,6,10,15,19,23-Hexamethyltetracosa-2,6,10,14,18,22-hexaene), of > 99,0 % purity.

4.9 n-Hexacosane [CAS: 630-01-3] (n-C26), of > 99,0 % purity.

4.10 n-Octacosane [CAS: 630-02-4] (n-C28), of > 99,0 % purity.

4.11 Carrier gas, hydrogen or helium, minimum 99,999 % purity.

4.12 Auxiliary gases, such as air, hydrogen and nitrogen, minimum 99,995 % purity.

5 Apparatus

Usual laboratory apparatus and glassware, together with the following:

5.1 Gas chromatograph, equipped with an on-column injector or equivalent device (e.g. PTV: Programmed Temperature Vaporisor), a temperature programmable oven and a flame ionization detector.

5.2 Capillary column, capable of being programmed up to 400 °C ("high temperature" type), e.g. 5.2.1 or 5.2.2. Other columns with similar performance may be used. Such columns should be tested first before being selected as co-elution may occur.

5.2.1 DB 5HT (5 % phenyl, 95 % methylpolysiloxane), 30 m x 0,25 mm, 0,1 µm film thickness.

5.2.2 CP9078, 15 m x 0,32 mm, 0,1 µm film thickness.

5.3 Volumetric flasks, 10, 20 and 50 ml capacity.

5.4 Screw-cap vials with PTFE-faced septa, 10 ml capacity.

5.5 Precision pipette, 1 ml capacity.

5.6 Microsyringes, 100 µl and 500 µl capacity.

5.7 Graduated cylinder, 10 ml capacity.

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

6 Preparation of solutions

6.1 Glyceryl monononadecanoate stock solution, 2,5 mg/ml

Accurately weigh approximately 50 mg (accuracy ± 0,1 mg) of glyceryl monononadecanoate (1-C19:0, 4.7) in a 20 ml volumetric flask (5.3) and make up to the mark with pyridine (4.2).

The solution shall be perfectly clear at ambient temperature. After storage in refrigerator at 4 °C the solution might show a precipitate that shall spontaneously re-dissolve when put at ambient temperature, without any external heating.

NOTE If stored at 4 °C the solution is stable for at least 3 months.

6.2 Reference solution

Prepare a stock solution of 1-C16:0 (4.5), 2-C16:0 (4.4), 1-C18:0 (4.6), squalene (4.8), n-C26 (4.9) and n-C28 (4.10) by weighing approximately 50 mg (accuracy ± 0,1 mg) of each in a 50 ml volumetric flask (5.3), dissolve and make up to the mark with pyridine (4.2).

7 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national regulations for the sampling of the product under test.

8 Procedure

8.1 Operating conditions

The chromatographic analysis conditions shall be chosen taking into account the characteristics of the column being used and the type of carrier gas (hydrogen or helium). It is however recommended to observe an analysis time of about 30 min to 35 min. Ensure that the triglycerides are fully eluted to prevent heavy ends built up in the column.

By way of indication, an example of analysis conditions is described in Table 1.

Table 1 — Exemplary analysis conditions

Column temperature program for column: DB 5HT (5 % phenyl, 95 % methylpolysiloxane), 30 m x 0,25 mm, 0,1 µm film thickness	60 °C hold for 1 min; 60 °C to 200 °C at 20 °C/min; 200 °C to 250 °C at 2 °C/min; 250 °C to 370 °C at 20 °C/min; 370 °C hold for 10 min
Column temperature program for column: CP9078, 15 m x 0,32 mm, 0,1 µm film thickness	60 °C hold for 1 min; 60 °C to 230 °C at 20 °C/min; 230 °C to 370 °C at 10 °C/min; 370 °C hold for 10 min
Detector temperature:	375 °C
Carrier gas Hydrogen or Helium	1,5 ml/min (to be optimized for the applied gas type)
Injection mode: volume injected:	 On-column or PTV 1 µl

8.2 Column evaluation

In order to establish that a column will perform as required, the following specifications shall be determined for new column acceptability and are useful for periodic column evaluation.

During robustness studies, n-C26, n-C28 and squalene were identified as components that can chromatographically co-elute with the target components 1-C16:0 and 1-C18:0. Therefore, resolution requirements are specified for these components in Table 2.

Table 2 — Resolution requirements

Pair	Required resolution for this pair
n-C26 and 1-C16:0	≥ 1,0
Squalene and 1-C18:0	≥ 1,0
n-C28 and 1-C18:0	≥ 1,0

NOTE Meeting these requirements, it is still possible that co-eluting of other components with the target components, i.e. 1-C16:0, 2-C16:0 and 1-C18:0 occurs. In that case, the calculated saturated monoglyceride content will be somewhat overestimated.

The column temperature programming profile is dependent upon the individual column characteristics. The temperature profile is to be optimized in order to establish satisfactory separations for the sets of components listed in 6.2.

Analyse the reference solution as described in 8.3.

The resolution, R, is calculated using the following formula:

$$R = \frac{1,18\left(t_2 - t_1\right)}{\left(W_{h_1} + W_{h_2}\right)} \quad (1)$$

where

t_2 is the retention time of peak 2;

t_1 is the retention time of peak 1;

W_{h_1} is the peak width at half height of the first peak;

W_{h_2} is the peak width at half height of the second peak.

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

NOTE Annex B shows an example for the calculation of the resolution.

8.3 Analysis of the reference solution

Using microsyringes (5.6), transfer 200 µl of the reference solution (6.2) and 150 µl of MSTFA (4.1) into a 10 ml vial (5.4). Avoid contact with humidity.

Hermetically close the vial and shake vigorously.

Store 15 min at room temperature, and then add approximately 8 ml of n-heptane (4.3) and homogenize the mixture.