



**SLOVENSKI STANDARD**  
**SIST EN 16270:2012**  
**01-november-2012**

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**Goriva za motorna vozila - Določevanje komponent z visokim vreliščem v nafti - Metoda plinske kromatografije**

Automotive fuels - Determination of high-boiling components in petrol - Gas chromatographic method

Kraftstoffe für Kraftfahrzeuge - Bestimmung von hoch-siedenden Komponenten in Ottokraftstoff - Gaschromatographisches Verfahren

Carburants pour automobiles - Détermination des composants haute-bouillantes dans l'essence - Méthode par chromatographie en phase gazeuse

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**Ta slovenski standard je istoveten z: EN 16270:2012**

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**ICS:**

75.160.20      Tekoča goriva      Liquid fuels

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EUROPEAN STANDARD

EN 16270

NORME EUROPÉENNE

EUROPÄISCHE NORM

September 2012

ICS 75.160.20

English Version

## Automotive fuels - Determination of high-boiling components including fatty acid methyl esters in petrol - Gas chromatographic method

Carburants pour automobiles - Détermination des composés à haut point d'ébullition dont les esters méthyliques d'acides gras dans l'essence - Méthode par chromatographie en phase gazeuse

Kraftstoffe für Kraftfahrzeuge - Bestimmung hochsiedender Komponenten einschließlich Fettsäure-Methylester in Ottokraftstoff - Gaschromatographisches Verfahren

This European Standard was approved by CEN on 27 July 2012.

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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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## Foreword

This document (EN 16270:2012) 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of an identical text or by endorsement, at the latest by March 2013, and conflicting national standards shall be withdrawn at the latest by March 2013.

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.

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## EN 16270:2012 (E)

## 1 Scope

This European Standard specifies a determination method of high boiling components in petrol according to EN 228 [1] by capillary gas chromatography using flame ionisation detection. This method is applicable to high boiling material, such as fatty acid methyl ester (FAME) or diesel fuel, having a boiling point greater than or equal to 1-methyl-naphthalene.

This European Standard is applicable to materials having a vapour pressure low enough to permit sampling at ambient temperature and a boiling range of at least 100 °C. This method pays special attention to fatty acid methyl esters.

The measurement range for the high boiling fraction is from 0,7 % (m/m) to 2,5 % (m/m). For the FAME fraction, the range is from 0,2 % (m/m) to 2 % (m/m).

NOTE 1 When calculating the FAME fraction, this method only takes the C18- methyl esters compounds into account.

NOTE 2 For the purposes of this standard, the terms “% (m/m)” and “% (V/V)” are used to represent the mass fraction ( $\mu$ ) and the volume fraction ( $\varphi$ ) of a material respectively.

**WARNING — The use of this European Standard may involve hazardous materials, operations and equipment. This European 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 establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.**

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## 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 14214, *Liquid petroleum products — Fatty acid methyl esters (FAME) for use in diesel engines and heating applications — Requirements and test methods*

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

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

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1

#### start of high boiling fraction

first peak to be included in the high boiling fraction

Note 1 to entry: In this determination method, this is the peak of 1-methyl-naphthalene.

### 3.2

#### end of high boiling fraction

last peak to be included in the high boiling fraction

Note 1 to entry: In this determination method, this is the peak of dotriacontane (n-C32).

## 3.3

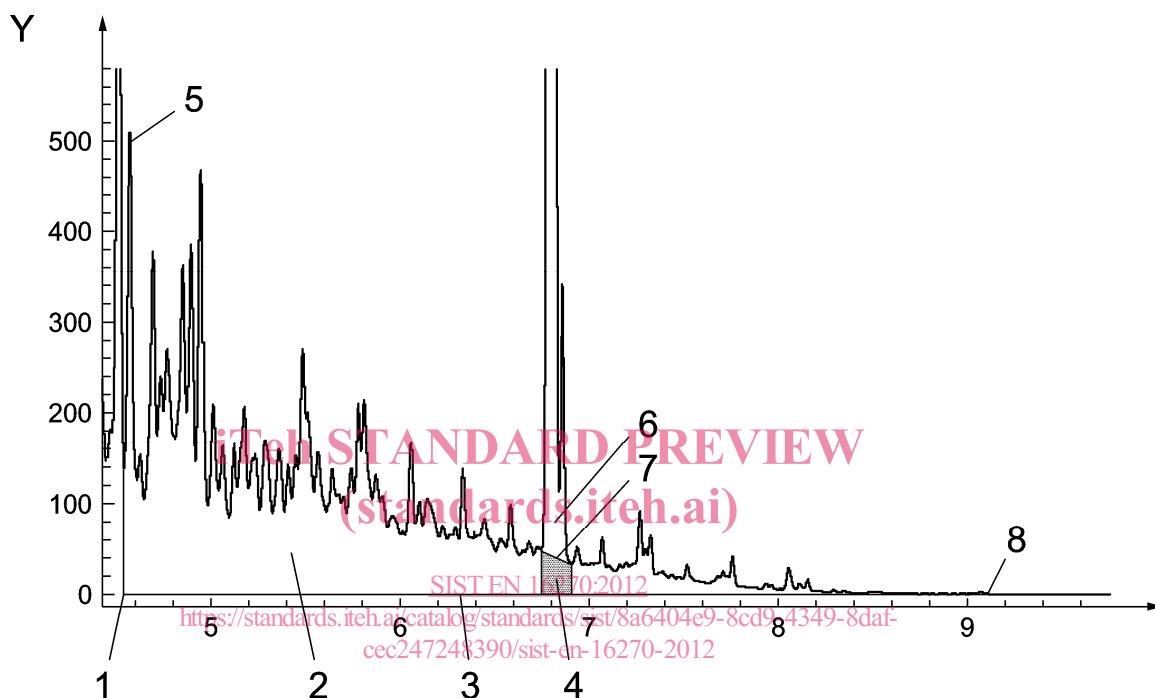
## fatty acid methyl esters fraction

## FAME fraction

combined area of the C18:0, C18:1, C18:2 and C18:3-FAME peaks

Note 1 to entry: The area is defined as shown in Figure 1. This only takes the C18-compounds into account as other methyl esters compounds such as C16:0 can only be present in very limited amounts. In addition, C16:0 co-elutes with an *n*-paraffin which would affect the quantification.

Note 2 to entry: Figure 1 presents visual explanation of the definitions on the basis of an exemplary chromatogram.



## Key

1	start of high boiling fraction from 1-methyl-naphthalene elution	6	FAME peak used for FAME calculation
2	area corresponding to high boilers (including FAME peaks)	7	baseline for FAME calculation
3	integration baseline	8	end of C32's elution
4	area under FAME baseline is high boiling material and is not considered FAME	<i>t</i>	time (min)
5	1-methyl-naphthalene	Y	FID Signal

Figure 1 — Example chromatogram with explanation of definitions

## 3.4

## total high boiling area for a calibration mixture

 $A_{tc}$ 

sum of the areas of 1-methylnaphthalene and dotriacontane

Note 1 to entry: See Figure 2 for further explanation.

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**3.5**  
**total FAME area for a calibration mixture**  
 $A_{fc}$   
 cumulative area from the start of the FAME C18-fraction until the end of the FAME C18-fraction for the calibration mixture

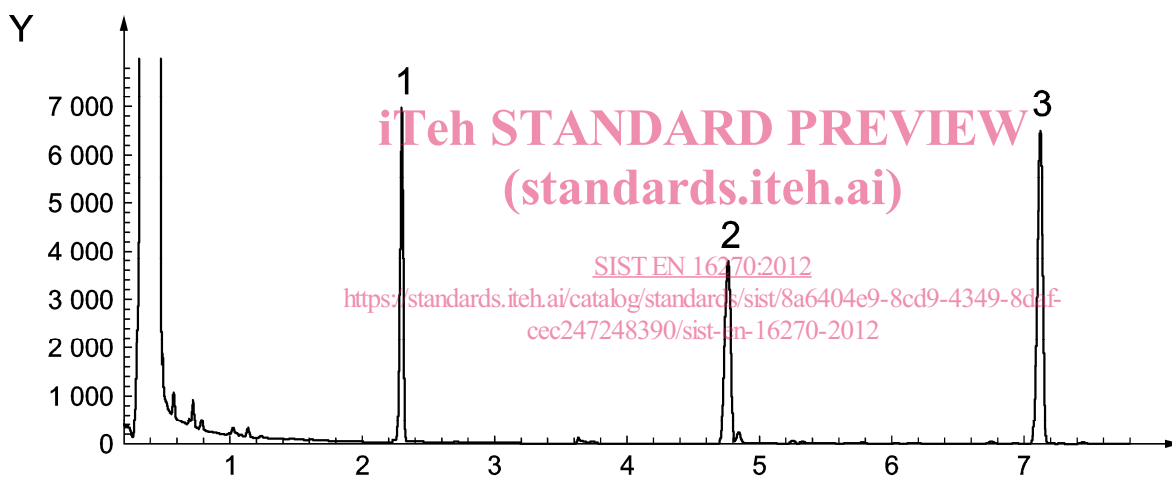
Note 1 to entry: See Figure 2 for further explanation.

**3.6**  
**total high boiling area for a sample**  
 $A_s$   
 cumulative area from the start of the high boiling fraction until the end of high boiling fraction for a sample

Note 1 to entry: See Figure 1 for further explanation.

**3.7**  
**total FAME fraction area for a sample**  
 $A_f$   
 cumulative area from the start of the FAME C18-fraction until the end of the FAME C18-fraction for a sample

Note 1 to entry: See Figure 1 for further explanation.



**Key**  
 1 1-Methyl-naphthalene – start of high boiling fraction  
 2 C18-FAME  
 3 dotriacontane – end of high boiling fraction  
 $t$  time (min)  
 $Y$  FID signal

**Figure 2 — Example of a calibration mixture chromatogram**

## 4 Principle

A test portion is introduced into a gas chromatographic column, which separates hydrocarbons in the order of increasing boiling point. The column temperature is raised at a linear reproducible rate and the area under the chromatogram is recorded throughout the analysis. The beginning and the end of the fractions are determined with a calibration mixture.

## 5 Reagents and materials

Unless otherwise stated, only chemicals of recognised analytical quality shall be used.



**5.1 Carrier gases**, helium, nitrogen or hydrogen, of at least 99,999 % (V/V) purity; any oxygen present should be removed, e.g. by a chemical resin filter.

**IMPORTANT — Follow the safety instructions from the filter supplier.**

**5.2 Hydrogen**, of a grade suitable for flame ionisation detectors.

**5.3 Compressed air**, regulated for flame ionisation detectors.

**5.4 Calibration components**, 1-methyl-naphthalene ( $\geq 98\%$ ), a fatty acid methyl ester (as specified in EN 14214) and dotriacontane ( $C_{32}H_{66}$ ,  $\geq 97\%$ ).

Fatty acid methyl esters that are mainly consisting of methyl ester C18 compounds such as rapeseed or soy are suitable components. Other methyl esters, such as palm oil, with higher amounts of C16 compounds, are not suitable

**5.5 Calibration mixture**

Prepare about 100 g of a calibration mixture by gravimetrically blending the components mentioned in 5.4. Typically a blend of 0,1 % (m/m) 1-methyl-naphthalene, 0,2 % (m/m) dotriacontane and 0,2 % (m/m) FAME in *n*-heptane is used.

Record the masses to the nearest 0,1 mg when weighing the components. Calculate the mass percentage of each calibration component in the mixture and round to the nearest 0,001 % (m/m).

A typical solvent to be used is *n*-heptane ( $\geq 99\%$  purity). Other solvents or mixtures of hydrocarbons may be used provided their boiling point is in the range of about 80 °C to about 160 °C to limit possible evaporation when preparing the mixture and overcome interference with the boiling point of 1-methyl-naphthalene. The boiling point range of the solvent shall not interfere with the calibration components.

The calibration sample may contain mixtures of different origin of FAME's such as rapeseed or soy, as is most representative for a specific application.

## 6 Apparatus

**6.1 Gas chromatograph**, with the following performance characteristics:

**6.1.1 Flame ionisation detector**, which shall be capable of operating at a temperature at least equivalent to the maximum column temperature employed in the method.

It is recommended that the capillary column sits just below the flame tip and that the orifice of the jet has an 0,6 mm minimum to prevent frequent blocking with silicones.

**6.1.2 Column temperature programmer**, capable of linear programmed temperature operation over a range from ambient temperature to 350 °C.

**6.1.3 Sample inlet system**, consisting of a programmable temperature vaporiser (PTV) or a temperature programmable cool on-column (COC) injection port.

The maximum temperature of the injection device shall be higher than the final oven temperature. The minimum temperature shall be low enough to prevent sample or solvent flashback, but high enough to allow sample focusing at the front of the column. Table 1 presents typical operating conditions.