

---

**Goriva za motorna vozila - Določevanje metanola v motornem gorivu etanol (E85) s plinsko kromatografijo - 1. del: Metoda z uporabo enojne kolonske tehnike**

Automotive fuels - Determination of methanol in automotive ethanol (E85) fuel by gas chromatography - Part 1: Method using single column technique

Kraftstoffe für Kraftfahrzeuge - Bestimmung von Methanol im automotive Ethanol (E85) als Kraftstoff durch Gaschromatographie - Teil 1: Gaschromatographisches Verfahren

Carburants pour automobiles - Détermination de la teneur en méthanol dans l'automobile d'éthanol (E85) de carburant par chromatographie en phase gazeuse - Partie 1 : Méthode par chromatographie en phase gazeuse

**Ta slovenski standard je istoveten z: EN 16761-1:2015**

**ICS:**

71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis
75.160.20	Tekoča goriva	Liquid fuels

**SIST EN 16761-1:2016****en,fr,de**

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

SIST EN 16761-1:2016

<https://standards.iteh.ai/catalog/standards/sist/c7eb68a1-9ffa-4870-a643-c17be9e9fb72/sist-en-16761-1-2016>

EUROPEAN STANDARD

EN 16761-1

NORME EUROPÉENNE

EUROPÄISCHE NORM

December 2015

ICS 75.160.20

English Version

## Automotive fuels - Determination of methanol in automotive ethanol (E85) fuel by gas chromatography - Part 1: Method using single column technique

Carburants pour automobiles - Détermination de la teneur en méthanol dans le carburant éthanol (E85) pour automobiles par chromatographie en phase gazeuse - Partie 1: Méthode par colonne simple

Kraftstoffe für Kraftfahrzeuge - Bestimmung des Methanolgehalts in Ethanolkraftstoff (E85) mittels Gaschromatographie - Teil 1: Einsäulenverfahren

This European Standard was approved by CEN on 10 October 2015.

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**

<b>Contents</b>	<b>Page</b>
European foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions .....	4
4 Principle .....	6
5 Reagents and materials.....	6
6 Apparatus.....	7
7 Sampling.....	8
8 Preparation of the apparatus .....	8
9 Determination of density .....	9
10 Calibration .....	9
11 Linearity check.....	9
12 Procedure.....	10
13 Calculation.....	10
14 Expression of results.....	11
15 Precision.....	11
15.1 General.....	11
15.2 Repeatability, <i>r</i> .....	11
15.3 Reproducibility, <i>R</i> .....	11
16 Test report.....	11
Bibliography.....	12

iTeh STANDARD PREVIEW

(standards.iteh.ai)

SIST EN 16761-1:2016

[https://standards.iteh.ai/catalog/standards/sist/c7eb68a1-9fa-4870-a645-](https://standards.iteh.ai/catalog/standards/sist/c7eb68a1-9fa-4870-a645-c17be9e9fb72/sist-en-16761-1-2016)[c17be9e9fb72/sist-en-16761-1-2016](https://standards.iteh.ai/catalog/standards/sist/c7eb68a1-9fa-4870-a645-c17be9e9fb72/sist-en-16761-1-2016)

## European foreword

This document (EN 16761-1:2015) 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 June 2016, and conflicting national standards shall be withdrawn at the latest by June 2016.

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.

The determination of a significant amount of methanol in ethanol (E85) automotive fuels was deemed to be necessary to check the product for compliance against EU emission regulations. The CEN/TC 19 Ethanol Fuels Task Force requested the development of such a determination technique.

In EN 16761, *Automotive fuels — Determination of methanol in automotive ethanol (E85) fuel by gas chromatography*, two test methods were developed that comply with this scope:

— *Part 1: Method using single column technique* [the present document];

— *Part 2: Method using heart cut technique.*

According to the CEN/CENELEC Internal Regulations, the national standards organizations 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.

## EN 16761-1:2015 (E)

## 1 Scope

This European Standard specifies a method for the determination of methanol in automotive ethanol (E85) fuel (also designated as ethanol (E85) automotive fuel or shortly "E85") by capillary gas chromatography using flame ionization detection. Fuel quality specifications for this product exist, see Bibliography Entry [1].

The measurement range for the methanol is from about 0,5 % (V/V) to about 1,5 % (V/V). Other methanol contents can also be determined, however no precision data for results outside the specified range is available.

NOTE For the purposes of this European Standard, the terms "% (m/m)" and "% (V/V)" are used to represent respectively the mass fraction,  $\mu$ , and the volume fraction,  $\varphi$ .

**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 users 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 228, *Automotive fuels — Unleaded petrol — Requirements and test methods*

EN ISO 3170, *Petroleum liquids — Manual sampling (ISO 3170)*  
<https://standards.iteh.ai/catalog/standards/sist/c7eb68a1-9ffa-4870-a643-c17be9e9f772/sist-en-16761-1-2016>

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

EN ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density — Hydrometer method (ISO 3675)*

EN ISO 3838, *Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pycnometer and graduated bicapillary pycnometer methods (ISO 3838)*

EN ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method (ISO 12185)*

## 3 Terms and definitions

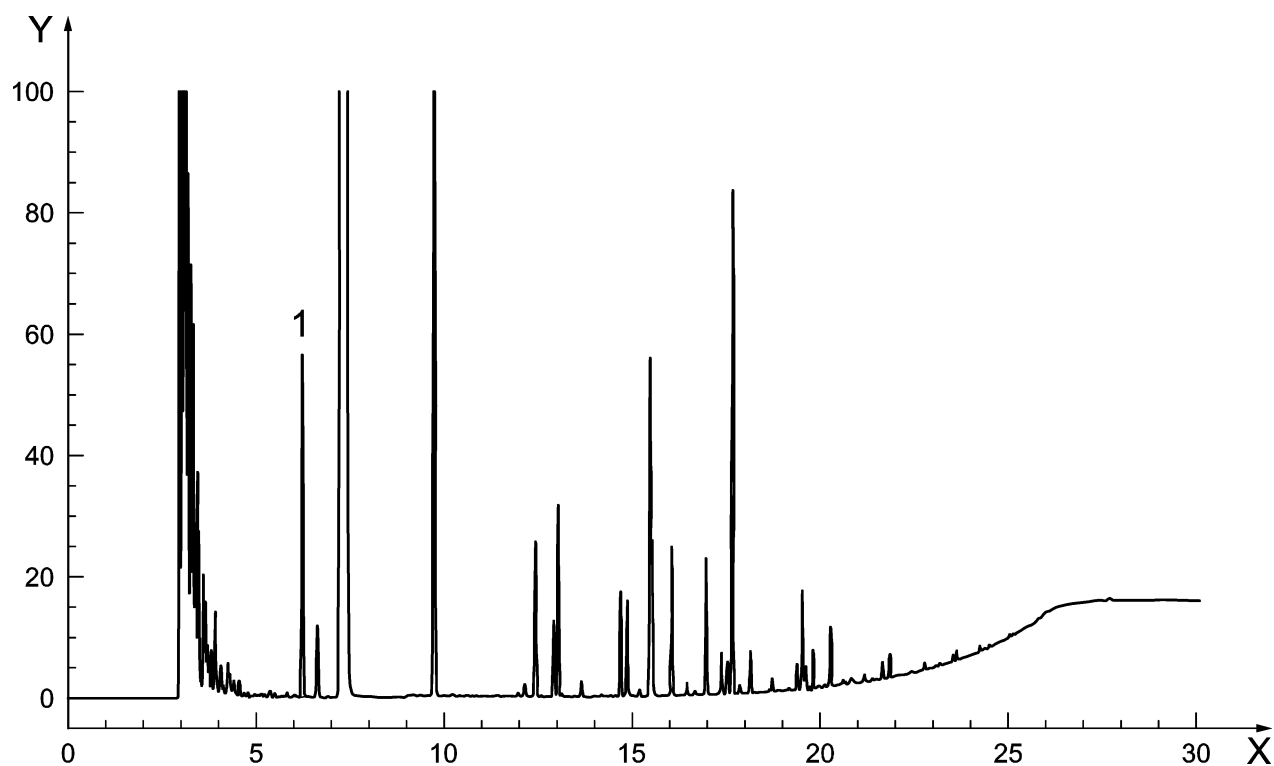
For the purposes of this document, the following terms and definitions apply.

### 3.1

#### **methanol calibration area**

area of the methanol peak in the calibration chromatogram

Note 1 to entry: Figure 1 shows an example of a calibration chromatogram.



Key

- 1 methanol
- X time (min)
- Y FID signal

iTeh STANDARD PREVIEW  
(standards.iteh.ai)

SIST EN 16761-1:2016

<http://standards.iteh.ai/en/standards/SIST-EN-16761-1-2016/c17be9e9fb72/sist-en-16761-1-2016>  
Figure 1 — Example chromatogram of a calibration solution

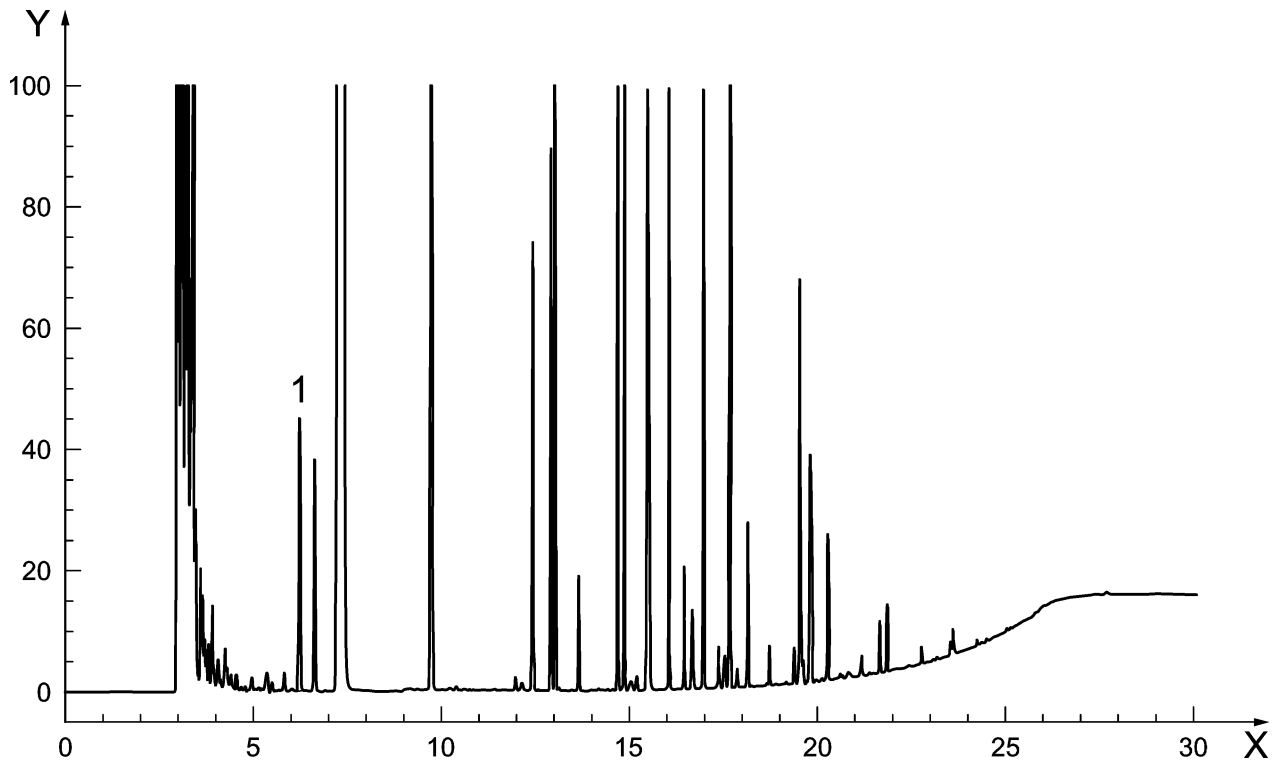
### 3.2

#### methanol sample area

$A_s$

area of methanol peak in the sample chromatogram

Note 1 to entry: Figure 2 shows an example of a sample chromatogram



## Key

- 1 methanol  
 X time (min)  
 Y FID signal

iTeh STANDARD PREVIEW  
 (standards.iteh.ai)

SIST EN 16761-1:2016

Figure 2 <https://standards.iteh.ai/catalog/standards/sist-en-16761-1-2016/c17be9e9fb72/sist-en-16761-1-2016>  
 Example chromatogram of an automotive ethanol (E85) fuel sample

## 4 Principle

This standard describes the analysis of methanol content in automotive ethanol (E85) fuels using a single high polarity capillary column to separate methanol from the hydrocarbon matrix and other oxygenate components.

The method uses external standard calibration.

A test portion is introduced into a gas chromatographic column, which separates oxygenated and non-oxygenated hydrocarbons in the order of increasing polarity and boiling point. The column temperature is raised at a linear reproducible rate and the area under the chromatogram is recorded throughout the analysis. The retention time as well as the calculation of the methanol amount is carried out with reference to calibration solution.

## 5 Reagents and materials

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

**5.1 Carrier gases**, helium, nitrogen or hydrogen, of at least 99,995 % (V/V) purity, any oxygen present should be removed, e.g. by a chemical resin filter.

Follow the safety instructions from the filter supplier.

**5.2 Hydrogen**, grade suitable for flame ionization detectors.

**5.3 Compressed air**, suitable for flame ionization detectors.



**5.4 Calibration components**, methanol (anhydrous,  $\geq 99,8\%$  (m/m)) and ethanol (pro-analysis  $\geq 99,8\%$  (m/m) by GC). It should be ensured that the ethanol contains no methanol detectable by this test method in a mixture (85/15 % (V/V)) of ethanol and petrol (5.5).

**5.5 Oxygenate-free petrol** (EN 228 compliant).

**5.6 Calibration solution:**

Prepare a calibration solution by gravimetrically blending 1,0 % (m/m) methanol (5.4) in an 85:15 (V/V) mixture of ethanol (5.4) and oxygenate-free petrol (5.5).

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

The hydrocarbon matrix shall not interfere with the methanol.

## 6 Apparatus

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

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

**6.1.2 Column temperature programmer**, capable of linear programmed temperature operation over a range from ambient temperature to 150 °C.

**6.1.3 Sample inlet system**, consisting of an injector with variable split flow or similar device.

Table 1 contains the typical operating conditions.

**6.2 Column**, a 50 m capillary column is typically being used, with 0,25 mm internal diameter and coated with 1,2,3-tris [2-cyanoethoxy]propane (TCEP).

See Table 1 for further advised conditions.

A different column material is suitable as long as similar separation efficiency is achieved. Precision values are however only available for TCEP material.

**6.3 Carrier gas control**, the chromatograph shall be able to deliver a constant carrier gas flow over the whole temperature range of the analysis.