

**Ethanol as a blending component for petrol -
Determination of higher alcohols, methanol and other
impurities - Gas chromatographic method**

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ICS 75.160.20

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English Version

Ethanol as a blending component for petrol - Determination of higher alcohols, methanol and other impurities - Gas chromatographic method

Éthanol comme base de mélange à l'essence -
Détermination de la teneur en alcools supérieurs, méthanol
et autres impuretés - Méthode par chromatographie en
phase gazeuse

Ethanol zur Verwendung als Blendkomponente in
Ottokraftstoff - Bestimmung von höheren Alkoholen,
Methanol und anderen Verunreinigungen -
Gaschromatographisches Verfahren

This European Standard was approved by CEN on 12 July 2013.

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Foreword

This document (EN 15721:2013) 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of an identical text or by endorsement, at the latest by February 2014, and conflicting national standards shall be withdrawn at the latest by February 2014.

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.

This document supersedes EN 15721:2009.

EN 15721:2013 includes the following significant technical changes with respect to EN 15721:2009: the method has been simplified and more tailored towards the determination of the higher alcohols as mentioned in EN 15376 (propan-1-ol, butan-1-ol, butan-2-ol, 2-methylpropan-1-ol (isobutanol), 2-methylbutan-1-ol, 3-methylbutan-1-ol, methanol). All other alcohol compounds are summed as impurities. The response factor check and the listed example response factors have been taken out.

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, 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.

Introduction

This document specifies a gas chromatographic (GC) test method for the determination of a number of compounds present in ethanol for use as a blending component in petrol according to the CEN ethanol blending component specification EN 15376 ^[1]. The test method comprises of GC identification and analysis of a number of molecules, which are then attributed to several classes ("impurities", "methanol", "higher alcohols"), which are needed for calculation of the specified values as required in EN 15376.

The method described in this document was prepared by CEN/TC 19's Working Group 9 and is based on two methods (^[2] and ^[3]) published from a European Regulation on wine and on other internationally published analytical methods on spirits ^[4]. The method is modified for determinations in ethanol for automotive applications.

1 Scope

This European Standard specifies a gas chromatographic method for ethanol, in which higher alcohols (propan-1-ol, butan-1-ol, butan-2-ol, 2-methylpropan-1-ol (isobutanol), 2-methylbutan-1-ol, and 3-methylbutan-1-ol) from 0,1 % up to 2,5 % (*m/m*), methanol from 0,1 % up to 3 % (*m/m*) and other impurities, in the range from 0,1 % up to 2 % (*m/m*) are determined.

Impurities are all the compounds not attributed to the groups of higher alcohols or methanol.

NOTE 1 The European ethanol blending component specification^[1] sets a limit for the combined result of ethanol + higher alcohols, not the ethanol content itself.

Due to possible interferences, the method is not applicable to denatured ethanol samples.

Water, if present in the sample, is not included in this analysis, because a signal for water is not visible in the chromatogram. Therefore, if "alcohol content" is called up in a specification, water needs to be considered separately in the calculations.

NOTE 2 For the purposes of this European Standard, the term "% (*m/m*)" is used to represent the mass fraction (ω).

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 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

3 Principle

The compounds specified in the scope are determined by direct injection of a test portion into a gas chromatograph (GC) system. An internal standard is added to the sample prior to the injection. The compounds are separated with suitable GC equipment using temperature programming with the option to also use flow programming on a suitable column. They are detected using a flame ionisation detector (FID). The concentration of each compound is determined from response factors with respect to the internal standard.

The response factors are obtained during calibration using the same chromatographic conditions as those for the analysis of the ethanol samples.

Two procedures ("Procedure A" and "Procedure B") are specified which differ mainly in the optional use of a water dilution step prior to the analysis. Both variants have been validated to produce identical results and precision in extensive RR tests.

4 Reagents and materials

All reagents shall be of recognised analytical grade (minimum 99 %) or of higher purity, if commercially available. They shall be stored in closed dark glass bottles and can be used for some long time. Other internal standards may also be used when there is sufficient proof that their GC signal does not interfere with the other signals in the chromatogram.

4.1 Water which, for analytical laboratory use, shall conform to grade 2 of EN ISO 3696.