
Goriva za motorna vozila - Določanje tipov ogljikovodikovih skupin in izbira ogljikovodikov in kisikovih spojin - Metoda plinske kromatografije z vakuumsko ultravijolično absorpcijsko spektroskopijo (GC-VUV)

Automotive fuels - Determination of hydrocarbon group types and select hydrocarbon and oxygenate compounds - Gas chromatography with vacuum ultraviolet absorption spectroscopy (GC-VUV) method

Kraftstoffe für Kraftfahrzeuge - Bestimmung von Kohlenwasserstoffgruppentypen und Auswahl von Kohlenwasserstoff- und Sauerstoffverbindungen - Gaschromatographie mit Vakuum-Ultraviolett-Absorptionsspektroskopie (GC-VUV)

Carburants pour automobiles - Détermination des types de groupes d'hydrocarbures et sélection des composés d'hydrocarbures et d'oxygénation - Chromatographie gazeuse avec spectroscopie d'absorption ultraviolette sous vide (GC-VUV) méthode

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Automotive fuels - Determination of hydrocarbon group types and select hydrocarbon and oxygenate compounds - Gas chromatography with vacuum ultraviolet absorption spectroscopy (GC-VUV) method

Carburants pour automobiles - Détermination de groupes d'hydrocarbures et de sélections d'hydrocarbures et de composés oxygénés - Chromatographie en phase gazeuse avec une détection par spectroscopie d'absorption ultraviolette sous vide (GC-VUV)

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Contents		Page
European foreword		3
Introduction		4
1	Scope.....	5
2	Normative references.....	6
3	Terms, definitions and abbreviations	6
3.1	Terms and definitions	6
3.2	Abbreviations.....	7
4	Principle	7
5	Reagents and materials	8
6	Apparatus	10
7	Sampling.....	11
8	Preparation of apparatus.....	11
9	Calibration.....	12
10	Pre-measurement validation.....	13
11	Procedure	14
12	Calculation.....	16
13	Report	21
14	Precision	21
Annex A (normative) Generating absorbance spectra from intensity scan data.....		23
A.1	Generating scan intensity data to VUV absorbance spectra.....	23
A.2	Modelling absorbance data	23
Annex B (informative) Relative response factors as function of molecular absorption cross sections.....		25
Bibliography		27

European foreword

This document (EN 18015:2024) 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 January 2025, and conflicting national standards shall be withdrawn at the latest by January 2025.

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EN 18015:2024 (E)**Introduction**

This document is used for the determination of saturated (paraffinic, isoparaffinic and naphthenic), olefinic, aromatic and oxygenated hydrocarbons in automotive motor gasoline according to European fuel specifications such as EN 228 [1].

The test method described in this document is based on ASTM D8071 [2] with modifications to the gas chromatographic oven temperature profile.

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1 Scope

This test method is a standard procedure for the determination of saturates, olefins, aromatics and oxygenates in unleaded petrol using gas chromatography and vacuum ultraviolet detection (GC-VUV).

Concentrations of compound classes and certain individual compounds are determined by mass fraction % (*m/m*) or volume fraction % (*V/V*). The concentration ranges for which the method is applicable are given in Table 1.

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, respectively.

This test method has been tested for unleaded petrol according EN 228 [1]; Although specifically developed for the analysis of automotive motor gasoline including those that contain oxygenates this test method applies to other hydrocarbon streams having similar boiling ranges, such as naphthas and reformates.

The method is found to be applicable to petrol containing other oxygenates than indicated in Table 1, such as isopropanol, iso-butanol, tert-butanol, n-propanol, acetone, tert-pentanol and di-isopropyl ether (DIPE), however precision has not been determined.

Table 1 — Application ranges

Compound or group	Units	Concentration range
Saturates	% (<i>V/V</i>)	21,48 to 80,87
Olefins	% (<i>V/V</i>)	0,22 to 41,90
Aromatics	% (<i>V/V</i>)	2,35 to 64,55
Benzene	% (<i>V/V</i>)	0,20 to 2,54
Toluene	% (<i>V/V</i>)	0,87 to 30,97
Ethylbenzene	% (<i>V/V</i>)	0,20 to 3,45
Xylenes	% (<i>V/V</i>)	0,49 to 18,59
Methanol	% (<i>V/V</i>)	0,07 to 15,30
Ethanol	% (<i>V/V</i>)	0,08 to 24,96
MTBE	% (<i>V/V</i>)	0,22 to 22,21
ETBE	% (<i>V/V</i>)	0,13 to 23,44
TAME	% (<i>V/V</i>)	0,24 to 21,96
TAAE	% (<i>V/V</i>)	0,24 to 8,60
Total oxygen content	% (<i>m/m</i>)	0,52 to 12,19

Individual hydrocarbon components are typically not baseline-separated by the procedure described in this test method. The coelutions are resolved at the detector using VUV absorbance spectra (Annex A) and deconvolution algorithms.

While this test method reports by mass fraction % (*m/m*) or volume fraction % (*V/V*) for several specific components that can be present in unleaded petrol, it does not attempt to speciate all possible components that can occur in unleaded petrol. In particular, this test method is not intended as a type of detailed hydrocarbon analysis (DHA).

EN 18015:2024 (E)

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 the user of this document to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

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)*

3 Terms, definitions and abbreviations**3.1 Terms and definitions**

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1.1**response filter**

mathematical operation performed on an absorbance spectrum for the purpose of converting the spectrum to a single-valued response suitable for representation in a two-dimensional chromatogram plot

3.1.2**library reference spectrum**

absorbance spectrum representation of a molecular species stored in a library database and used for identification of a compound/compound class or deconvolution of multiple coeluting compounds

3.1.3**response area**

response summed over a given time interval having units of absorbance units

Note 1 to entry: Time factor necessary to convert a response area to a true mathematical area cancels out of all critical calculations and is omitted.

3.1.4**relative response factor****RRF**

function of the compound's absorption cross section (expressed in cm² per molecule) and methane's cross section

Note 1 to entry: The absorption cross section is averaged over the 125 nm to 240 nm wavelength region.

Note 2 to entry: A compound's relative response factor is a function of the type and number of chemical bonds.

3.2 Abbreviations

For the purposes of this document, the following abbreviations apply.

AU	absorbance units
DHA	detailed hydrocarbon analysis
GC	gas chromatograph
GC-VUV	gas chromatography with vacuum ultraviolet spectroscopy detection
RI	retention index
RRF	relative response factor
MTBE	methyl tertiary butyl ether
ETBE	ethyl tertiary butyl ether
TAME	tertiary amyl methyl ether
TAEF	tertiary amyl ethyl ether
DIPE	diisopropyl ether
TID	time interval deconvolution
iC4	isobutane
C4	butane
iC5	isopentane
C5	pentane
C15	pentadecane
VUV	vacuum ultraviolet

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4 Principle

A sample is introduced to a gas chromatographic (GC) system. After volatilization, the effluent is introduced onto a GC column for separation, and then detected by a vacuum ultraviolet absorption spectroscopy detector¹. The separation is accomplished using a 30 m, nonpolar phase capillary column and a moderately fast temperature ramp (typical operating parameters of this test method are given in Clause 11). Coelutions are resolved by the detector using vacuum ultraviolet (VUV) absorbance spectra and deconvolution, more precisely, an automated time interval deconvolution (TID) algorithm.

The TID uses approximate retention indices, the VUV spectral library, and the concept of additive absorption according to Beer–Lambert Law principles to bin pure or deconvolved mixture spectra into class or species-specific responses in an automated fashion. The chromatogram is interrogated in defined time intervals that can be adjusted depending on the complexity of the sample.

The response area for each sequential time interval over the entire chromatogram is determined. The calculation of the results is based on the deconvoluted response areas of the saturates, olefins, aromatics and oxygenates, in addition to several individual species components. The percent mass concentrations are calculated from the response areas using class-based or compound-specific relative response factors (RRFs). The volume percent concentrations are calculated from the mass concentrations by applying specific component or class and carbon number-based density values. The mass and volume percent

¹ VGA100, VGA101, VUV Analyze, VUV Vision are the trade name of products supplied by VUV Analytics, Cedar Park, Texas, USA 78613. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of the product named.

EN 18015:2024 (E)

calculations are software automated, whereby the RRFs and densities are a function of elution time in a static database library.

5 Reagents and materials**5.1 Purity of reagents**

Reagent grade chemicals shall be used in all tests. Use only reagents of recognized analytical grade. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Helium carrier gas for gas chromatograph, 99,999 % pure.

NOTE Method performance has not been studied for other carrier gases such as hydrogen or nitrogen.

5.3 Purge or makeup gas, nitrogen, helium or argon, for vacuum ultraviolet detector, 99,999 % pure.**5.4 Methylene chloride or carbon disulfide**, reagent grade, used as a solvent test sample and GC rinse solvent. (**Warning** — Toxic material can be combustible at high temperatures.)

5.4.1 Alternate reagent grade solvents may be used provided that autosampler syringe carryover does not interfere with sample analysis and that a suitable baseline (see 8.7) is achieved.

5.5 Retention time standard consisting of isobutane (iC₄), butane (C₄), isopentane (iC₅), and pentane (C₅) through pentadecane (C₁₅) linear alkanes, approximately 1 % by mass each, in suitable solvent such as methylene chloride, used as retention time markers.

5.6 System validation mixture, having the components and approximate concentrations given in Table 2. The actual concentration levels of the prepared system validation mixture in Table 2 are not critical but shall be accurately known.

5.6.1 The components of the system validation mixture may be modified to include linear alkanes or other compounds necessary for determining a retention index file (see 9.1 and 9.2).

5.6.2 The concentrations in Table 2 may be suitably modified to accommodate additional or substituted components. The concentrations shall otherwise be accurately known.

Table 2 — System validation mixture

Component	Concentration % (m/m)	Component	Concentration % (m/m)
Cyclopentane	1,1	Benzene	2,2
n-Pentane	1,1	Toluene	2,2
Cyclohexane	2,1	trans-Decahydronaphthalene	4
2,3-Dimethylbutane	2,1	n-Tetradecane	4,5
n-Hexane	2,1	Ethylbenzene	4,5
1-Hexene	1,5	o-Xylene	4
Methylcyclohexane	4	n-Propylbenzene	5