

SLOVENSKI STANDARD oSIST prEN ISO 22854:2023

01-november-2023

Tekoči naftni proizvodi - Določanje vrste ogljikovodikov in oksigenatov v motornem bencinu in bencinu na osnovi etanola (E85) - Metoda multidimenzionalne plinske kromatografije (ISO/DIS 22854:2023)

Liquid petroleum products - Determination of hydrocarbon types and oxygenates in automotive-motor gasoline and in ethanol (E85) automotive fuel - Multidimensional gas chromatography method (ISO/DIS 22854:2023)

Flüssige Mineralölerzeugnisse - Bestimmung von Kohlenwasserstoffgruppen und sauerstoffhaltigen Verbindungen in Ottokraftstoffen und in Ethanolkraftstoff (E85) - Multidimensionales gaschromatographisches Verfahren (ISO/DIS 22854:2023)

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Produits pétroliers liquides - Détermination des groupes d'hydrocarbures et de la teneur en composés oxygénés de l'essence pour moteurs automobiles et du carburant éthanol pour automobiles E85 - Méthode par chromatographie multidimensionnelle en phase gazeuse (ISO/DIS 22854:2023)

Ta slovenski standard je istoveten z: prEN ISO 22854

ICS:

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

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en,fr,de

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DRAFT INTERNATIONAL STANDARD ISO/DIS 22854

ISO/TC 28

Voting begins on: **2023-08-30**

Secretariat: NEN

Voting terminates on: 2023-11-22

Liquid petroleum products — Determination of hydrocarbon types and oxygenates in automotivemotor gasoline and in ethanol (E85) automotive fuel — Multidimensional gas chromatography method

Produits pétroliers liquides — Détermination des groupes d'hydrocarbures et de la teneur en composés oxygénés de l'essence pour moteurs automobiles et du carburant éthanol pour automobiles E85 — Méthode par chromatographie multidimensionnelle en phase gazeuse

ICS: 75.080

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Reference number ISO/DIS 22854:2023(E)

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Published in Switzerland

ISO/DIS 22854:2023(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 22854:2021), which has been technically revised. The main changes compared to the previous edition are as follows:

- the scope and precision have been clarified in terms of total oxygenates and corrected for previous mistakes in oxygen and ethanol contents, as well as corrected for rounding as required by the reporting requirements;
- a new procedure C has been implemented (and precision thereof determined by an ILS) to allow determination of very low aromaticity, benzene, toluene and hexane contents required for small engine petrol fuel for which CEN/TC 19 has developed a specification;
- the text has been further harmonized with ASTM D6839^[7].

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <u>www.iso.org/members.html</u>.

Introduction

Previous editions of this document were used for determination of saturated, olefinic, aromatic and oxygenated hydrocarbons in automotive motor gasoline according to European fuel specifications.

An interlaboratory study has shown that the method can be used for gasolines with a higher concentration of oxygenated compounds, including methanol. The interlaboratory study also provided data to calculate precision for toluene in gasoline. A further study focussed on higher ether contents. <u>Annex B</u> includes example chromatograms of gasolines with a variety of oxygenates for use for the correct identification of these oxygenates.

Another interlaboratory study has shown that the method is applicable for gasolines with a very low aromaticity. The study delivered optimalization of a validation step (Procedure C).

The test method described in this document is harmonized with ASTM D6839,^[7] except for Procedure C which has a merely European product focus.

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Liquid petroleum products — Determination of hydrocarbon types and oxygenates in automotivemotor gasoline and in ethanol (E85) automotive fuel — Multidimensional gas chromatography method

1 Scope

This document specifies the gas chromatographic (GC) method for the determination of saturated, olefinic and aromatic hydrocarbons in automotive motor gasoline, small engine petrol and ethanol (E85) automotive fuel. Additionally, the benzene and toluene content, oxygenated compounds and the total oxygen content can be determined.

NOTE 1 For the purposes of this document, the terms % (m/m) and % (V/V) are used to represent respectively the mass fraction, w, and the volume fraction, φ .

This document defines three procedures, A, B and C. The application ranges are given in <u>Table 1</u>.

NOTE 2 Procedure A has been assessed for total oxygenates content, and later for individual oxygenates. The ranges given are considered to apply to individual oxygenated compounds or the total group of (unidentified, not further precised) oxygenates.

Although specifically developed for the analysis of automotive motor gasoline that contains oxygenates, this test method can also be applied to other hydrocarbon streams having similar boiling ranges, such as naphthas and reformates.

NOTE 3 For Procedure A, applicability of this document has specifically been verified for the determination of *n*-propanol, acetone, and di-isopropyl ether (DIPE). However, no precision data have been determined for these compounds.

Procedure B describes the analysis of oxygenated groups (ethanol, methanol, ethers, C3 – C5 alcohols) in ethanol (E85) automotive fuel containing ethanol between 50 % (V/V) and 85 % (V/V). The gasoline is diluted with an oxygenate-free component to lower the ethanol content to a value below 20 % (V/V) and 85 % (V/V) and 85 % (V/V).

The sample can be fully analysed including hydrocarbons. Precision data for the diluted sample are only available for the oxygenated groups.

NOTE 4 An overlap between C9 and C10 aromatics can occur. However, the total is accurate. Isopropyl benzene is resolved from the C8 aromatics and is included with the other C9 aromatics.

Procedure C describes the analysis of small engine petrol fuel containing low contents of aromatics and olefins.

Component or group	Procedure A	Procedure B	Procedure C		
Saturates, %(V/V)	26,9 - 79,3				
Total aromatics, %(<i>V/V</i>)	19,3 - 46,3		0,4 - 2,7		
Total olefins, %(V/V)	0,4 - 26,9		0,1 - 2,4		
Benzene, %(V/V)	0,38 - 1,98		0,04 - 0,11		
Toluene, %(<i>V/V</i>)	5,85 - 31,65				
<i>n</i> -Hexane, % (<i>V</i> / <i>V</i>)			0,1 - 2,1		
^a Oxygenated compounds (as individual component or group)					

Table 1 — Application ranges for each procedure

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Component or group	Procedure A	Procedure B	Procedure C
Napthenes, % (V/V)			0,2 - 3,8
Oxygenates ^a , %(<i>V</i> /V)	0,61 - 9,85		0,08 - 0,86
Total oxygen content, %(<i>m/m</i>)	0,50 - 12,32		
Methanol, %(V/V)	1,05 - 16,96	1,05 - 16,96	
Ethanol, %(V/V)	0,50 - 17,86	> 50,0 and < 85,0	0,06 - 0,39
C3 – C5 alcohols, %(<i>V/V</i>)		> 1,4 and < 2,5	
Ethers, %(<i>V</i> / <i>V</i>)		> 0,5 and < 1,6	
MTBE, %(<i>V/V</i>)	up to 22,79	1,0 - 15,7	0,01 - 0,70
ETBE, %(<i>V/V</i>)	up to 21,36	1,0 - 15,5	0,09 - 0,73
TAME, %(<i>V/V</i>)		1,0 - 5,9	
TAEE, %(<i>V/V</i>)	0,98 - 15,59	1,0 - 15,6	
^a Oxygenated compounds (as individu	al component or group)	_ ·	

 Table 1 (continued)

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.

ISO 3170, Petroleum liquids — Manual sampling

ISO 3171, Petroleum liquids — Automatic pipeline sampling

3 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 6c851e6273e/osist-pren-iso-22854-2023

— IEC Electropedia: available at <u>https://www.electropedia.org/</u>

3.1 hydrocarbon hydrocarbon group HG family of hydrocarbons such as saturated hydrocarbons, olefinic hydrocarbons 3.1.1 saturate saturated hydrocarbon type of hydrocarbon (3.1) that contains no double bonds with a carbon number of 3 to 12 EXAMPLE *n*-Paraffins, *iso*-paraffins, naphthenes and poly-naphthenes.

3.1.2 olefin

olefinic hydrocarbon

type of *hydrocarbon* (3.1) that contains double or triple bonds with a carbon number of 3 to 10

EXAMPLE *n*-Olefins, *iso*-olefins and cyclic olefins.

3.1.3 aromatic aromatic hydrocarbon

type of cyclic *hydrocarbon* (3.1) with alternating double and single bonds between carbon atoms forming the rings

EXAMPLE Benzene, toluene and higher homologous series with a carbon number of 6 to 10 and naphthalenes, with a carbon number of up to 12.

3.2

oxygenate

oxygenated compound

type of *hydrocarbon* (3.1) that contains an oxygen group, the addition of which is allowed according to current petrol specifications

EXAMPLE Alcohols and ethers.

Note 1 to entry: See Note 2 to <u>Clause 1</u>.

4 Principle

4.1 All procedures described use the same separation technique and analysis procedure. The difference between the main Procedure A and that for Procedure B is that the sample is diluted. The diluting solvent is not considered in the integration. This makes it possible to report the results of the undiluted sample after normalization to 100 %.

Procedure C differs from Procedure A that it requires an additional tuning step to assure that the individual oxygenates, aromatics and olefins are correctly identified by optimizing the pre-column temperatures and valve settings.

4.2 The gasoline sample being analysed is separated into hydrocarbon groups by means of GC analysis using special column-coupling and column-switching procedures.

The sample is injected into the GC system and, after vaporization, is separated into the different groups. Detection is always done by a flame ionization detector (FID).

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4.3 The mass concentration of each detected compound or hydrocarbon group is determined by the application of relative response factors (see 9.2) to the area of the detected peaks, followed by normalization to 100 %. For automotive motor gasoline samples containing oxygenates that cannot be determined by this test method, the hydrocarbon results are normalized to 100 % minus the value of oxygenates as determined by another method. The liquid volume concentration of each detected compound or hydrocarbon group is determined by the application of density values (see 9.3) to the calculated mass concentration of the detected peaks followed by normalization to 100 %.

IMPORTANT — It is essential to the correct execution of the method that great care be taken to ensure that all compounds are correctly identified. This is especially true for the identification of oxygenated compounds because of their wide range of response factors. It is, therefore, highly recommended for correct identification to verify possibly unknown oxygenates using a reference mixture that contains these pure compounds.

4.4 After this analysis, the automotive motor gasoline is separated into hydrocarbon groups and then by carbon number. Using the corresponding relative response factors, the mass distributions of the groups in the automotive motor gasoline sample can be calculated.