SLOVENSKI STANDARD
oSIST prEN ISO 22854:2020

01-maj-2020

Tekoči naftni proizvodi - Določevanje vrste ogljikovodikov in oksigenatov v motornem bencinu in bencinu na osnovi etanola (E85) - Metoda multidimenzionalne plinske kromatografije (ISO/DIS 22854:2020)

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 22854:2020)

Flüssige Mineralölerzeugnisse - Bestimmung der Kohlenwasserstoffgruppen und der sauerstoffhaltigen Verbindungen in Ottokraftstoffen und in Ethanolkraftstoff (E85) - Multidimensionales gaschromatographisches Verfahren (ISO/DIS 22854:2020)

Produits pétroliers liquides - Détermination des groupes d'hydrocarbures et des composés oxygénés de l'essence pour moteurs automobiles et du carburant pour automobiles éthanol (E85) - Méthode par chromatographie multidimensionelle en phase gazeuse (ISO/DIS 22854:2020)

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

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

ICS: 75.080

IMPORTANT — Please use this updated version dated 2020-01-27, and discard any previous version of this DIS. [Corrections have been made to Annex B.]

This document is circulated as received from the committee secretariat.

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

This fourth edition cancels and replaces the third edition (ISO 22854:2016), which has been technically revised.

The main changes compared to the previous edition are as follows:

— new examples of typical chromatograms in Annex B.

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 www.iso.org/members.html.

Introduction

This document is a small update of the second edition (ISO 22854:2014), which in turn was a revision to extend the scope of the first edition. Originally ISO 22854:2008 (and its predecessor EN 14517:2004) was used for determination of saturated, olefinic, aromatic and oxygenated hydrocarbons in automotive motor gasoline according to European fuel specifications. Recent round-robin work has shown that the scope of the method can be updated without alteration to include petrol with higher oxygen percentages than mentioned in the first edition and will now be applicable for automotive motor gasoline up to and including E10.

An interlaboratory study organized by CEN has shown that the method can also be used for high-ethanol gasoline [also called ethanol (E85) automotive fuel], provided that the sample is diluted with a component that will not interfere with any of the components or group of components that need to be analysed. Details of how to perform such analysis are given in 8.2.

The derived precision data for methanol do not comply with the precision calculation as presented in this document. No precision calculation for methanol has been established as the need for such data has not been expressed. If methanol is present in the automotive motor gasoline sample, it is recommended that its contents is verified by the use of an appropriate test method, for instance as given in EN 228[1].

The test method described in this document is harmonized with ASTM D6839[2].

Liquid petroleum products — Determination of hydrocarbon types and oxygenates in automotive-motor 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 and ethanol (E85) automotive fuel. Additionally, the benzene content, oxygenate 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, μ, and the volume fraction, φ.

This document defines two procedures, A and B.

Procedure A is applicable to automotive motor gasoline with total aromatics of up to 50 % (*V/V*); total olefins from about 1,5 % (*V/V*) up to 30 % (*V/V*); oxygenates from 0,8 % (*V/V*) up to 15 % (*V/V*); total oxygen from about 1,5 % (*m/m*) to about 3,7 % (*m/m*); and benzene of up to 2 % (*V/V*). The system can be used for ethers with 5 or more C atoms up to 22 % (*V/V*) but the precision has not been established up to this level.

Although this test method can be used to determine higher-olefin contents of up to 50 % (*V/V*), the precision for olefins was tested only in the range from about 1,5 % (*V/V*) to about 30 % (*V/V*).

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 2 For Procedure A, precision data have been established for the oxygenate compounds in automotive motor gasoline samples containing ethyl-tert-butyl ether (ETBE), methyl-tert-butyl ether (MTBE), tert-amyl-methyl ether (TAME), *iso*-propanol, iso-butanol, tert-butanol, methanol and ethanol. The derived precision data for methanol do not comply with the precision calculation as presented in this document. Applicability of this document has also 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 procedure for 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*) before the analysis by GC. If the ethanol content is unknown, it is advisable to use a dilution of 4:1 when analysing the sample.

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

NOTE 3 For Procedure B, the precision can be used for an ethanol fraction from about 50 % (*V/V*) up to 85 % (*V/V*). For the ether fraction, the precision as specified in Table 6 can be used for samples containing at least 11 % (*V/V*) of ethers. For the higher alcohol fraction, too few data were obtained to derive a full precision statement and the data presented in Table 6 are therefore only indicative.

NOTE 4 While developing this test method, the final boiling point was limited to 215 °C.

NOTE 5 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.

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*

ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
hydrocarbon group
family of hydrocarbons such as saturated hydrocarbons, olefinic hydrocarbons, etc

3.1.1
saturated hydrocarbon
saturate
type of hydrocarbon 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
olefinic hydrocarbon
olefin
type of hydrocarbon 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 hydrocarbon
aromatic
type of cyclic hydrocarbon 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 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 Clause 1, Note 2.

3.3
partial group
PG
one carbon number in an individual group, being either a single compound like toluene or an isomeric mixture

EXAMPLE *n*-Butane and *iso*-butane.

4 Principle

4.1 Procedure A and Procedure B use the same separation technique and analysis procedure. The difference between the parts is that for Procedure B 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 %.

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

The automotive motor gasoline 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).

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 oxygen - containing 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. By the use of the corresponding relative response factors, the mass distributions of the groups in the automotive motor gasoline sample can be calculated.

5 Reagents and materials

5.1 Gases

Installation of suitable moisture filters is recommended for hydrogen, helium and nitrogen lines.

5.1.1 Hydrogen, 99,995 % pure.

DANGER — Hydrogen is explosive when mixed with air at concentration between 4 % (*V/V*) and 75 % (*V/V*). See the equipment manufacturers' manuals concerning leaks in the system.

5.1.2 Helium or nitrogen, 99,995 % pure.

The system's operating parameters such as column and trap temperatures, carrier gas flows and valve switching times are depending on the type of carrier gas used. The use of nitrogen as carrier gas is not possible on all configurations. Contact the equipment manufacturer for specific information or instructions on the use of nitrogen.