SLOVENSKI STANDARD
SIST EN ISO 3924:2019

01-november-2019

Nadomešča:
SIST EN ISO 3924:2016

Naftni proizvodi - Določevanje destilacijskega območja - Metoda plinske kromatografije (ISO 3924:2019)

Petroleum products - Determination of boiling range distribution - Gas chromatography method (ISO 3924:2019)

Mineralölerzeugnisse - Bestimmung des Siedeverlaufs - Gaschromatographisches Verfahren (ISO 3924:2019)

Produits pétroliers - Détermination de la répartition dans l'intervalle de distillation - Méthode par chromatographie en phase gazeuse (ISO 3924:2019)

Ta slovenski standard je istoveten z: **EN ISO 3924:2019**

ICS:

71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis
75.080	Naftni proizvodi na splošno	Petroleum products in general

SIST EN ISO 3924:2019 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 3924

September 2019

ICS 75.080

Supersedes EN ISO 3924:2016

English Version

Petroleum products - Determination of boiling range distribution - Gas chromatography method (ISO 3924:2019)

Produits pétroliers - Détermination de la répartition dans l'intervalle de distillation - Méthode par chromatographie en phase gazeuse (ISO 3924:2019)

Mineralölerzeugnisse - Bestimmung des Siedeverlaufs - Gaschromatographisches Verfahren (ISO 3924:2019)

This European Standard was approved by CEN on 30 June 2019.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, 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: Rue de la Science 23, B-1040 Brussels

Ref. No. EN ISO 3924:2019 E

Contents

Page

European foreword

This document (EN ISO 3924:2019) has been prepared by Technical Committee ISO/TC 28 "Petroleum and related products, fuels and lubricants from natural or synthetic sources" in collaboration with 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 March 2020, and conflicting national standards shall be withdrawn at the latest by March 2020.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 3924:2016.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Endorsement notice

The text of ISO 3924:2019 has been approved by CEN as EN ISO 3924:2019 without any modification.

INTERNATIONAL STANDARD

ISO 3924

Fifth edition
2019-07

Petroleum products — Determination of boiling range distribution — Gas chromatography method

Produits pétroliers — Détermination de la répartition dans l'intervalle de distillation — Méthode par chromatographie en phase gazeuse

Reference number
ISO 3924:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 method was originally based on the joined IP 406[3] and ASTM D2887[4] methods.

This fifth edition cancels and replaces the fourth edition (ISO 3924:2016), which has been technically revised. The main changes compared with the previous edition are as follows.

— The accelerated procedure has been moved from Annex B to the main body text. It is described as Procedure B and has a precision and bias calculation in relation to Procedure A (the original procedure).

— A new annex has been added with the newly defined boiling points for n-alkanes to keep the method technically equivalent with IP 406 and ASTM D2887.

— Annexes E and F have been added with information on the use of alternative carrier gases.

— Several safety warnings and editorial updates have been made.

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.

Petroleum products — Determination of boiling range distribution — Gas chromatography method

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of the document.

1 Scope

This document specifies a method for the determination of the boiling range distribution of petroleum products. The method is applicable to petroleum products and fractions with a final boiling point of 538 °C or lower at atmospheric pressure as determined by this document. This document does not apply to gasoline samples or gasoline components. The method is limited to products having a boiling range greater than 55 °C and having a vapour pressure sufficiently low to permit sampling at ambient temperature.

The document describes two procedures.

a) Procedure A allows a larger selection of columns and analysis conditions, such as packed and capillary columns as well as a thermal conductivity detector in addition to the flame ionization detector. Analysis times range from 14 min to 60 min.

b) Procedure B is restricted to only three capillary columns and requires no sample dilution. The analysis time is reduced to about 8 min.

Both procedures have been successfully applied to samples containing fatty acid methyl esters (FAME) up to 20 % (volume fraction).

NOTE For the purposes of this document, the terms "% (mass fraction)" and "% (volume fraction)" are used to represent the mass fraction (μ), the volume fraction (φ) of a material.

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 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
initial boiling point
IBP
temperature corresponding to the retention time at which a net area count equal to 0,5 % of the total sample area under the chromatogram is obtained

3.2
T10, T30, T50, T70, T90
temperature (T) corresponding to the retention time at which a net area count equal to the 10 %, 30 %, 50 %, 70 % or 90 % of the total sample area under the chromatogram is obtained

3.3
final boiling point
FBP
temperature corresponding to the retention time at which a net area count equal to 99,5 % of the total sample area under the chromatogram is obtained

3.4
slice rate
number of data slices acquired per unit of time used to integrate the continuous (analogue) chromatographic detector response during an analysis

Note 1 to entry: The slice rate is expressed in Hz (for example, slices per second).

4 Principle

A sample is introduced into a gas chromatographic column, which separates hydrocarbons in the order of increasing boiling point. The column temperature is raised at a reproducible rate and the area under the chromatogram is recorded throughout the analysis. Boiling temperatures are assigned to the time axis from a calibration curve, obtained under the same conditions by running a known mixture of hydrocarbons covering the boiling range expected in the sample. From these data, the boiling range distribution is obtained.

Annex A presents a correlation model for the calculation of physical distillation[1][5][6] equivalent data from boiling range distribution analysis by gas chromatography determined following this document.

5 Reagents and materials

5.1 Stationary phase for columns, non-polar, that elutes hydrocarbons in boiling point order.

NOTE The following materials have been used successfully as liquid phases, other stationary phases can be used, see 6.2.

For packed columns:

— silicone gum rubber UC-W98;

— silicone gum rubber GE-SE-30;

— silicone gum rubber OV-1;

— silicone gum rubber OV-101.

For capillary columns:

— polydimethylsiloxane.

5.2 Solid support for packed columns, usually consisting of crushed fire brick or chromatographic diatomaceous earth.

The particle size and support loading shall be such as to give optimum resolution and analysis time.

NOTE In general, support loadings of 3 % to 10 % have been found most satisfactory.

5.3 Carrier gas, with a minimum purity of 99,995 %, constituted of:

a) helium for use with flame ionization detectors (FIDs) or thermal conductivity detectors;

b) for the use of nitrogen or hydrogen as a carrier gas, see Annexes E and F.

CAUTION — Helium and nitrogen are compressed gases under high pressure. Hydrogen is an extremely flammable gas under high pressure.

5.4 Hydrogen, grade suitable for FIDs.

CAUTION — Hydrogen is an extremely flammable gas under high pressure.

5.5 Compressed air, free of oil and water, regulated for FIDs.

CAUTION — Compressed air is a gas under high pressure and supports combustion.

5.6 Calibration mixture, consisting of an accurately weighed mixture of n-alkanes covering the range from C_5 to C_{44} and dissolved in carbon disulfide (5.8).

For packed columns, the final concentration in mass should be approximately 10 parts of the n-alkane mixture to 100 parts of carbon disulfide. For capillary columns, the final concentration in mass should be approximately 1 part of the n-alkane mixture to 100 parts of carbon disulfide.

The following mixture of n-alkanes has been found to be satisfactory for most samples: C_5, C_6, C_7, C_8, C_9, C_{10}, C_{12}, C_{14}, C_{16}, C_{18}, C_{20}, C_{24}, C_{28}, C_{32}, C_{36}, C_{40}, C_{44}. At least one component of the mixture shall have a boiling point lower than the initial boiling point (IBP) of the sample and at least one component shall have a boiling point higher than the final boiling point (FBP) of the sample. The boiling points of n-alkanes are listed in Table 1.

If the test sample contains significant quantities of n-alkanes that can be identified on the chromatogram, these peaks can be used as internal boiling point calibration points. However, it is advisable to use the calibration mixture to be sure of peak identifications.

Propane and butane can be added non-quantitatively to the calibration mixture, if necessary, to conform to 5.6. This can be done by bubbling a small amount of the gaseous hydrocarbon into a septum-sealed vial of the calibration mixture using a gas syringe.

If stationary phases other than those listed in the note in 5.1 are used, the retention times of a few alkylbenzenes across the boiling range, such as *o*-xylene, *n*-butylbenzene, 1,3,5-tri-isopropylbenzene, *n*-decylbenzene and *n*-tetradecylbenzene, shall also be checked to make certain that the column is separating according to the boiling point order (see Annex C).

5.7 Reference material, the primary reference material used shall be ASTM reference gas oil no. 1 or no. 2 (as specified in Annex B).

5.8 Carbon disulfide, reagent grade or better (CAS RN 75-15-0).

CAUTION — Carbon disulfide is extremely volatile flammable and toxic.

