



SLOVENSKI STANDARD

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Naftni proizvodi - Določanje porazdelitve območja vrelišč z metodo plinske kromatografije - 1. del: Srednji destilati in mazalna olja

Petroleum products - Determination of boiling range distribution by gas chromatography method - Part 1: Middle distillates and lubricating base oils

Mineralölerzeugnisse - Gaschromatographische Bestimmung des Siedeverlaufes - Teil 1: Mitteldestillate und Grundöle

Produits pétroliers - Détermination de la répartition dans l'intervalle de distillation par méthode de chromatographie en phase gazeuse - Partie 1: Distillats moyens et huiles lubrifiantes

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ICS:

75.080	Naftni proizvodi na splošno	Petroleum products in general
75.100	Maziva	Lubricants, industrial oils and related products

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English Version

**Petroleum products - Determination of boiling range
distribution by gas chromatography method - Part 1:
Middle distillates and lubricating base oils**

Produits pétroliers - Détermination de la répartition
dans l'intervalle de distillation par méthode de
chromatographie en phase gazeuse - Partie 1: Distillats
moyens et huiles lubrifiantes

Mineralölerzeugnisse - Gaschromatographische
Bestimmung des Siedeverlaufes - Teil 1:
Mitteldestillate und Grundöle

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 19.

If this draft becomes a European Standard, 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.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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<https://standards.iteh.ai/catalog/standards/sist/a6b5a1a0-520f-440f-97f5-e98951b205c7/sist-en-15199-1-2021>

prEN 15199-1:2019 (E)**European foreword**

This document (prEN 15199-1:2019) 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 document is currently submitted to the CEN Enquiry.

EN 15199 consists of the following parts, under the general title *Petroleum products — Determination of boiling range distribution by gas chromatography method*:

- *Part 1: Middle distillates and lubricating base oils*
- *Part 2: Heavy distillates and residual fuels*
- *Part 3: Crude oil*
- *Part 4: Light fractions of crude oil*

This part of the standard describes the determination of boiling range distribution of materials with initial boiling points (IBP) above 100 °C and final boiling points (FBP) below 750 °C. For testing materials with initial boiling points (IBP) above 100 °C and final boiling point (FBP) above 750 °C, Part 2 of the standard may be used. For testing materials with initial boiling points (IBP) below 100 °C and final boiling points (FBP) above 750 °C, such as crude oils, Part 3 may be used.

This second edition cancels and replaces the first edition (EN 15199-1:2006) which is updated editorially as well as that the precision is extended for the recovery points between 10 % and 50 %.

This part of the standard is based on IP Test Method IP 480 [1] and ASTM Test Method ASTM D6352 [2].

1 Scope

This document specifies a method for the determination of the boiling range distribution of petroleum products by capillary gas chromatography using flame ionization detection. The standard is applicable to materials having a vapour pressure low enough to permit sampling at ambient temperature and a boiling range of at least 100 °C. The standard is applicable to distillates with initial boiling points (IBP) above 100 °C and final boiling points (FBP) below 750 °C, for example, middle distillates and lubricating base stocks.

The test method is not applicable for the analysis of petroleum or petroleum products containing low molecular weight components (for example naphtha's, reformates, gasolines, diesel). Components containing hetero atoms (for example alcohols, ethers, acids, or esters) or residue are not to be analysed by this test method.

NOTE 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 .

WARNING — The use of this European Standard may involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard 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 3170:2004)*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling (ISO 3171:1988)*

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 <http://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

Note 1 to entry: Explanation of some of the terms is given in Figure 1.

3.1

initial boiling point

IBP

temperature corresponding to the retention time at which a net area counts equal to 0,5% of the total sample area (3.6) under the chromatogram is obtained

3.2

final boiling point

FBP

temperature corresponding to the retention time at which a net area counts equal to 99,5% of the total sample area (3.6) under the chromatogram is obtained

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3.3

area slice

area resulting from the integration of the chromatographic detector signal within a specified retention time interval

Note 1 to entry: In area slice mode peak detection parameters are bypassed and the detector signal integral is recorded as area slices of consecutive, fixed duration time interval.

3.4

corrected area slice

area slice (3.3) corrected for baseline offset by subtraction of the exactly corresponding area slice in a previously recorded blank (non-sample) analysis

3.5

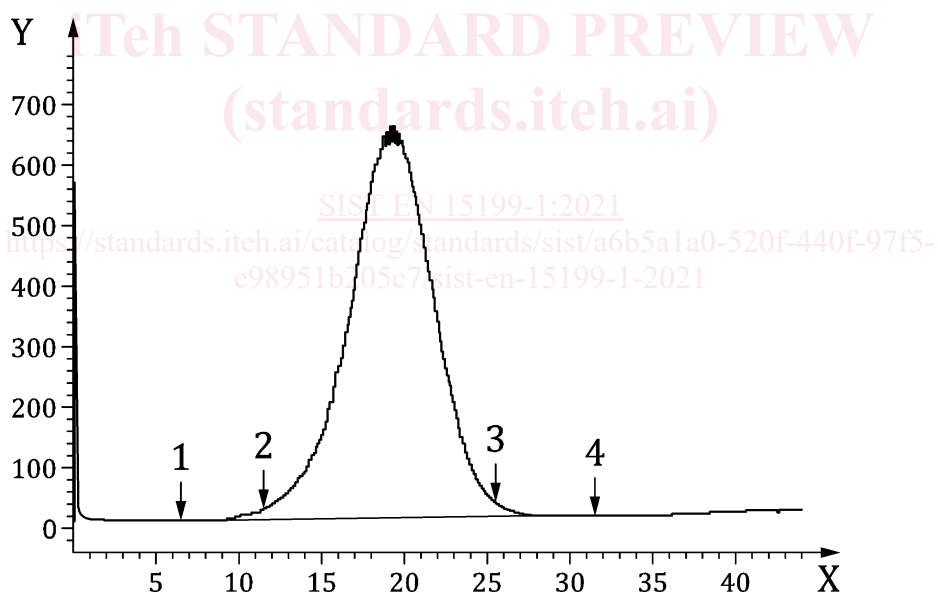
cumulative corrected area

accumulated sum of corrected area slices (3.4) from the beginning of the analysis through a given retention time, ignoring any non-sample area for example of solvent

3.6

total sample area

cumulative corrected area (3.5), from the initial area point to the final area point, where the chromatographic signal has returned to baseline after complete sample elution

**Key**

- 1 start of elution
- 2 initial boiling point (IBP) (3.1)
- 3 final boiling point (FBP) (3.2)
- 4 end of elution
- X min
- Y pA

Figure 1 — Typical chromatogram

4 Principle

A test portion is introduced into a gas chromatographic column, which separates hydrocarbons in the order of increasing boiling point. The column temperature is raised at a linear reproducible rate and the area under the chromatogram is recorded throughout the analysis. Boiling points are assigned to the time-axis from a calibration curve obtained by running a mixture of known normal alkanes (5.5), covering the sample boiling range, under the same conditions. From these data, the boiling range distribution is obtained.

5 Reagents and materials

Unless otherwise stated, only chemicals of recognized analytical quality shall be used.

5.1 Liquid stationary phase, a methyl silicone stationary phase for the column.

5.2 Carrier gas, helium, nitrogen or hydrogen, of at least 99,999 % (V/V) purity. Any oxygen present is removed by a chemical resin filter.

WARNING — Follow the safety instructions from the filter supplier.

5.3 Hydrogen, grade suitable for flame ionization detectors

5.4 Compressed air, regulated for flame ionization detectors.

5.5 Alkanes, normal alkanes of at least 98 % (m/m) purity from C₅ to C₁₀, C₁₂, C₁₄, C₁₆, C₁₈, C₂₀, C₂₄ and C₂₈ to be used with Polywax (5.6).

NOTE The calibration mixture from ISO 3924 [3] is also suitable.

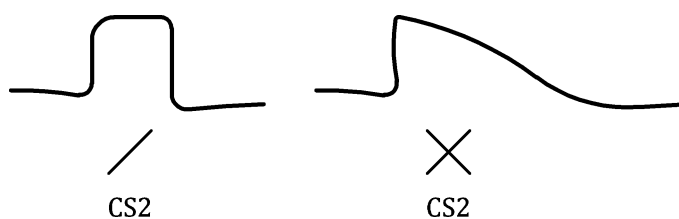
5.6 Polywax 655 or 1000

5.7 Carbon disulphide, purity 99,7 % (V/V) minimum.

WARNING — Extremely flammable and toxic by inhalation.

CAUTION — It is recommended that all work with carbon disulphide is carried out in an explosion protected fume cupboard.

To confirm the suitability of the carbon disulphide (5.7) as a solvent, it is recommended to check elution profiles (see Figure 2).



Key

- / good
- × bad

Figure 2 — Solvent peak shapes

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5.8 Calibration mixture

The mixture shall contain at least one normal alkane with a boiling point lower than the IBP of the sample, and at least one normal alkane with a boiling point higher than the FBP of the sample.

Dissolve 0,1 g of Polywax (5.6) in 7 ml carbon disulphide (5.7), warming gently if necessary. Prepare an equal volume mixture of alkanes (5.5) and add 10 μ l to the Polywax solution.

NOTE 1 Commercially available alkane standards are suitable for column performance checks.

NOTE 2 The calibration mix is used to determine the column resolution, skewness of the C₂₀ peak, and retention time versus boiling point calibration curve.

5.9 Reference materials (RM)

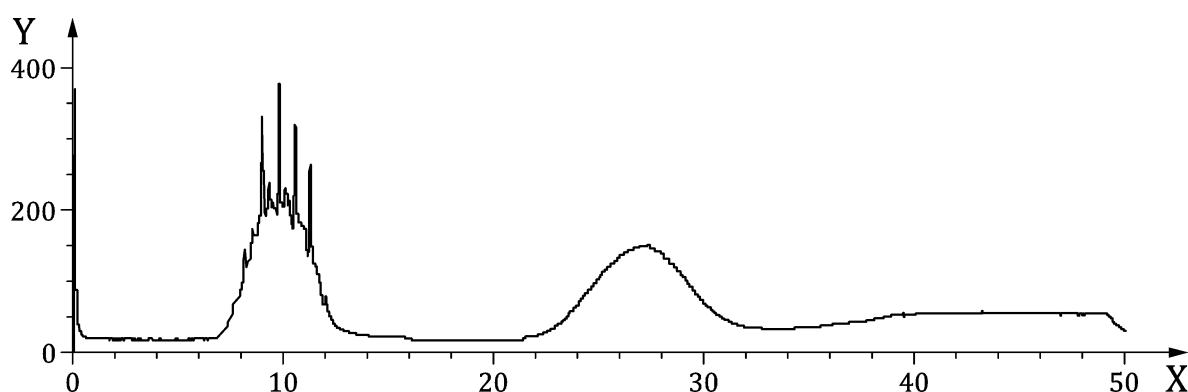
5.9.1 A reference material has two functions:

- External Standard: to determine the recovery of samples by comparing the total sample area (3.6) of the reference material with the total sample area (3.6) of the unknown sample;
- Boiling Point Distribution Standard: to check the proper functioning of the system by comparing the results with a known boiling point distribution on a routine basis. A typical example is given in (5.9.2).

5.9.2 **Reference Material 5010**, a reference sample that has been analysed by laboratories participating in the test method cooperative study. Consensus values for the boiling range distribution of this sample are given in Table 1. (standards.iteh.ai)

5.9.3 **Cyclohexane**. (C₆H₁₂)—(99+ % pure) may be used in place of CS₂ for the preparation of the calibration mixture. [SIST EN 15199-1:2021](https://standards.iteh.ai/catalog/standards/sist/a6b5a1a0-520f-440f-97f5-)
<https://standards.iteh.ai/catalog/standards/sist/a6b5a1a0-520f-440f-97f5->

5.9.4 **Binary gravimetric blend**, a binary distillate mixture with boiling points ranges that gives a baseline at the start, a baseline between the two peaks and an end time that is as close to the end of the chromatogram as possible (see Figure 3 and B.3). This mixture is used to check the relative response of the two distillates and to check the baselines at the start, middle and end of the chromatogram.



Key

Y response

X retention time

Figure 3 — Typical chromatogram of binary gravimetric blend distillate

Table 1 — Reference Material 5010 composition

% OFF	Average °C	Allowable difference 95,5 % CI °C
IBP	428	9
5	477	3
10	493	3
15	502	3
20	510	3
25	518	4
30	524	4
35	531	4
40	537	4
45	543	4
50	548	5
55	554	4
60	560	4
65	566	4
70	572	4
75	578	5
80	585	4
85	593	4
90	602	4
95	616	4
FBP	655	18

6 Apparatus

6.1 Gas chromatograph, with the following performance characteristics.

6.1.1 Flame ionization detector, connected to the column to avoid any cold spots. The detector shall be capable of operating at a temperature at least equivalent to the maximum column temperature employed in the method.

6.1.2 Column temperature programmer, capable of linear programmed temperature operation over a range of 10 °C above ambient to 450 °C.

6.1.3 Sample inlet system, consisting of a programmable temperature vaporizer (PTV) or cold on-column (COC) injection port. The maximum temperature of the injection device shall be equal to, or higher than, the final oven temperature. The minimum temperature shall be low enough to prevent sample or solvent flashback, but high enough to allow sample focusing at the front of the column. Table 2 contains the typical operating conditions.