



**BSI Standards Publication**

# **Petroleum products — Determination of boiling range distribution by gas chromatography method**

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Part 1: Middle distillates and lubricating base oils

## National foreword

This British Standard is the UK implementation of EN 15199-1:2020. It is dual numbered as BS 2000-480:2020 in the UK. It supersedes BS EN 15199-1:2006 (dual numbered as BS 2000-480:2006), which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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### BS 2000 Series

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| Date            | Text affected                            |
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| 31 January 2021 | Correction to dual numbering information |

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

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## European foreword

This document (EN 15199-1:2020) 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of an identical text or by endorsement, at the latest by June 2021, and conflicting national standards shall be withdrawn at the latest by June 2021.

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 15199-1:2006.

The main changes in this edition are:

- the precision is extended for the recovery points between 10 % and 50 %;
- the text has been updated editorially in order to give better guidance to operators executing the test.

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 document specifies 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 can 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 can be used. Part 4 describes the determination of boiling range distribution of hydrocarbons up to *n*-nonane in crude oil.

This document is based on IP Test Method IP 480 [4] and ASTM Test Method ASTM D6352 [3].

According to the CEN-CENELEC Internal Regulations, the national standards organisations 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.

## 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) or middle distillates like Diesel and Jet fuel.

Petroleum or petroleum products containing blending components which contain heteroatoms (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 and the volume fraction.

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 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)*

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

## 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 counts equal to 0,5 % of the total sample area (3.6) under the chromatogram is obtained (see Figure 1)

### 3.2 final boiling point

#### FBP

temperature corresponding to the retention time at which a net area (3.7) counts equal to 99,5 % of the total sample area (3.6) under the chromatogram is obtained (see Figure 1)

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

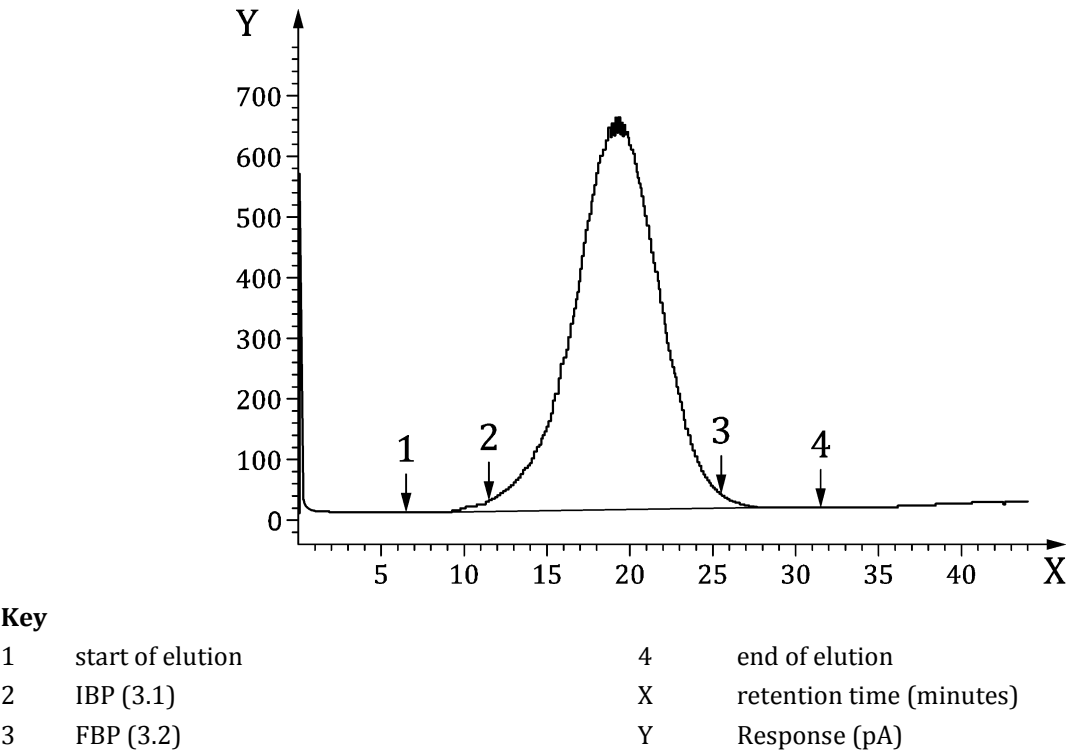


Figure 1 — Typical chromatogram

3.7  
net area

cumulative area counts for the sample minus the cumulative area count for the blank

### 3.8

#### recovery

ratio of the cumulative area count of the sample to that of the reference material (external standard) corrected for dilution and material weights combined with the percentage of light ends, if applicable

## 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 n-alkanes, covering the sample boiling range, under the same conditions. From these data, the boiling range distribution is obtained.

Several SIMDIS methods are standardized test methods and each one is dedicated to a certain boiling point range or product.

EN ISO 3924 [1] is limited to products having an initial boiling point greater than 55 °C, a final boiling point lower than 538 °C and having a vapour pressure sufficiently low to permit sampling at ambient temperature.

EN 15199-2 is applicable to materials with initial boiling points (IBP) above 100 °C and final boiling points (FBP) above 750 °C, for example, heavy distillate fuels and residuals. The method is not applicable to bituminous samples.

EN 15199-3 is applicable to crude oils. The boiling range distribution and recovery (3.8) up to C<sub>100</sub> or C<sub>120</sub> can be determined.

## 5 Reagents and materials

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

**5.1 Carrier gas**, helium, 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.

**5.2 Hydrogen**, grade suitable for flame ionization detectors.

**5.3 Compressed air**, suitable for flame ionization detectors.

**5.4 Alkanes**, n-alkanes of at least 98 % (m/m) purity from C<sub>5</sub> to C<sub>10</sub>, C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, C<sub>18</sub>, C<sub>20</sub>, C<sub>24</sub> and C<sub>28</sub>.

NOTE The calibration mixture from EN ISO 3924 [1] is also suitable.

**5.5 Polywax 655® or 1000®**

**5.6 Carbon disulfide**, (CS<sub>2</sub>) purity 99,7 % (V/V) minimum.

WARNING — Extremely flammable and toxic by inhalation.

CAUTION — It is recommended that all work with CS<sub>2</sub> is carried out in an explosion protected fume cupboard.

Cyclohexane ( $C_6H_{12}$ )—(>99 % pure) can be used in place of  $CS_2$  for the preparation of the calibration mixture. However, the precision of this method is based on calibration mixtures, reference material and samples prepared with  $CS_2$  only.

### 5.7 Calibration mixture

Dissolve 0,1 g of Polywax (5.5) in 7 ml  $CS_2$  (5.6), warming gently if necessary. Prepare an equal volume mixture of alkanes (5.4) and add 10  $\mu$ 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  $C_{20}$  peak, and retention time versus boiling point calibration curve.

### 5.8 Reference materials

**5.8.1** 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 (3.5) 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 (5.8.2).

**5.8.2 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**.

**5.8.3 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 2** 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.