BRITISH STANDARD

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

Part 1: Middle distillates and lubricating base oils

The European Standard EN 15199-1:2006 has the status of a British Standard

ICS 75.080

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National foreword

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A list of organizations represented on PTI/13 can be obtained on request to its secretary.

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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 par chromatographie en phase gazeuse - Partie 1: Distillats moyens et huiles lubrifiantes

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

This European Standard was approved by CEN on 28 August 2006.

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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 Central Secretariat has the same status as the official versions.

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Foreword

This document (EN 15199-1:2006) 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of an identical text or by endorsement, at the latest by April 2007, and conflicting national standards shall be withdrawn at the latest by April 2007..

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*

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 part of the standard is a joint development between the EI [1], ASTM [2] and CEN.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This European Standard specifies a method for the determination of the boiling range distribution of petroleum products by capillary gas chromatography using flame ionisation 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 naphthas, reformates, gasolines, diesel). Components containing hetero atoms (for example alcohols, ethers, acids, or esters) or residue are not to be analyzed by this test method.

NOTE For the purposes of this European Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction and the volume fraction.

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 referenced documents are indispensable for the application 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.

NOTE 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 count equal to 0,5 % of the total sample area under the chromatogram is obtained

3.2
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

3.3
area slice
area resulting from the integration of the chromatographic detector signal within a specified retention time interval

NOTE 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.

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3.4
corrected area slice
area slice corrected for baseline offset by subtraction of the exactly corresponding area slice in a previously recorded blank (non-sample) analysis

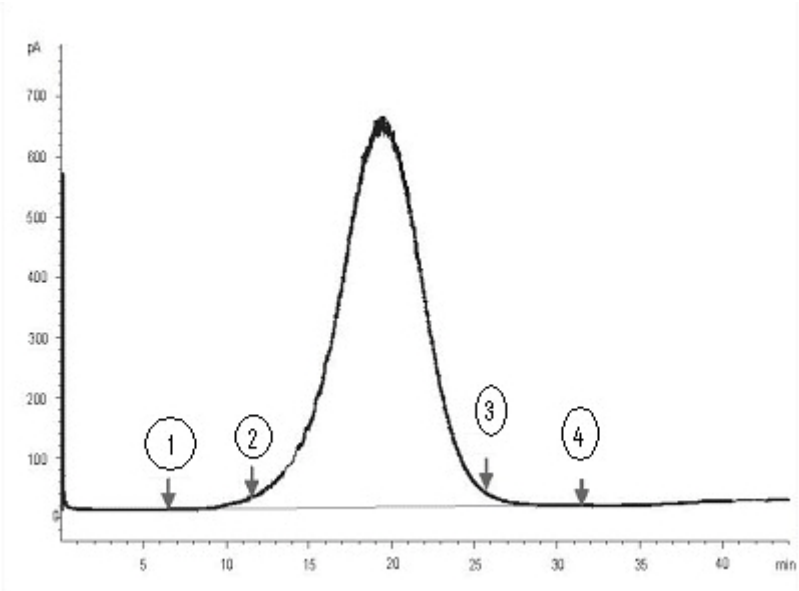
3.5
cumulative corrected area
accumulated sum of corrected area slices from the beginning of the analysis through a given retention time, ignoring any non-sample area for example of solvent

3.6
slice rate
time interval used to integrate the continuous (analogue) chromatographic detector response during an analysis

NOTE The slice rate is expressed in Hz (for example integrations per second or slices per second).

3.7
slice time
analysis time associated with each area slice throughout the chromatographic analysis

NOTE The slice time is the time at the end of each contiguous area slice.



Key:

1 start of elution
2 initial boiling point (IBP)
3 final boiling point (FBP)
4 end of elution

Figure 1 — Typical chromatogram

3.8
total sample area
cumulative corrected area, from the initial area point to the final area point, where the chromatographic signal has returned to baseline after complete sample elution

3.9
net area
cumulative area counts for the sample minus the cumulative area count for the blank

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3.10
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 normal alkanes, 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 gases, 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 ionisation detectors

5.4 Compressed air, regulated for flame ionisation detectors.

5.5 Alkanes, normal alkanes of at least 98 % (*m/m*) purity from C_5 to C_{10}, C_{12}, C_{14}, C_{16}, C_{18}, C_{20}, C_{24} and C_{28} 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 disulfide, purity 99,7 % (*V/V*) minimum.

WARNING — Extremely flammable and toxic by inhalation.

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

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