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Petroleum products — Determination of cloud point — Automated step-wise cooling method

Produits pétroliers — Détermination du point de trouble — Méthode automatisée par refroidissement par paliers

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Foreword

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This document was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, in collaboration with ISO Technical Committee TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, in accordance with the agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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 describes an automated method for the determination of the cloud point, based on the manual determination technique described in ISO 3015[2]. In parallel with the revision of the manual method, the scope has been extended to new fuels.

Petroleum products — Determination of cloud point — Automated step-wise cooling method

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 users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of this document and to fulfil other applicable requirements for this purpose.

1 Scope

This document specifies a method to determine cloud point using a step-wise cooling technique that is executed by means of automated equipment types with optical detection mode.

The method is applicable to distillate fuels, fatty-acid methyl esters (FAME) and paraffinic diesel fuels, including blends thereof, as well as those containing flow-improvers or other additives, intended for use in diesel engines and domestic heating installations.

The method can be applied to other products such as vegetable oils or lubricants, but these kinds of products have not been evaluated during the interlaboratory study (ILS), no precision data are available.

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
cloud point
temperature at which a cloud of wax crystals first appears in a liquid when it is cooled under specified conditions

4 Principle

A sample is cooled at a specified rate and examined periodically or continuously. The temperature at which a cloud is first observed in the test jar is recorded as the cloud point.

5 Reagents and materials

5.1 Lintless filter paper, with particle retention of (5 ± 1) µm.

6 Apparatus

6.1 Test jar, cylindrical, of clear glass, flat-bottomed, 33,2 mm to 34,8 mm in outside diameter and 80 mm to 125 mm in height.

The inside diameter of the jar may range from 30,0 mm to 32,4 mm, within the constraint that the wall thickness be no greater than 1,6 mm. The jar shall be marked with a line to indicate a sample height (54 ± 3) mm above the inside bottom.

6.2 Sample temperature probe, cylindrical, vertical in the centre of the test jar and plunged in the sample having the sensitive area located at maximum 10 mm from the bottom of the jar.

A thermometer with digital display shall be used for measuring the sample temperature with a resolution of 0,1 °C and an accuracy of 0,5 °C (see Figure 1).

NOTE The exact location of the probe depends on the equipment design manufacturer.

6.3 Anti-moisture device, to close the test jar and the jacket, avoiding any moisture to be introduced.

6.4 Jacket, watertight, cylindrical, metal, flat-bottomed, minimum 90 mm in depth with an inside diameter of 44,2 mm to 45,8 mm. It shall be supported in a vertical position and it shall be capable of being cleaned.

6.5 Disc (optional, depending on the design of the equipment), made of any insulating material (for example cork or felt), 6 mm in thickness, to fit loosely inside the jacket to maintain the test jar and avoid any direct contact with the jacket.

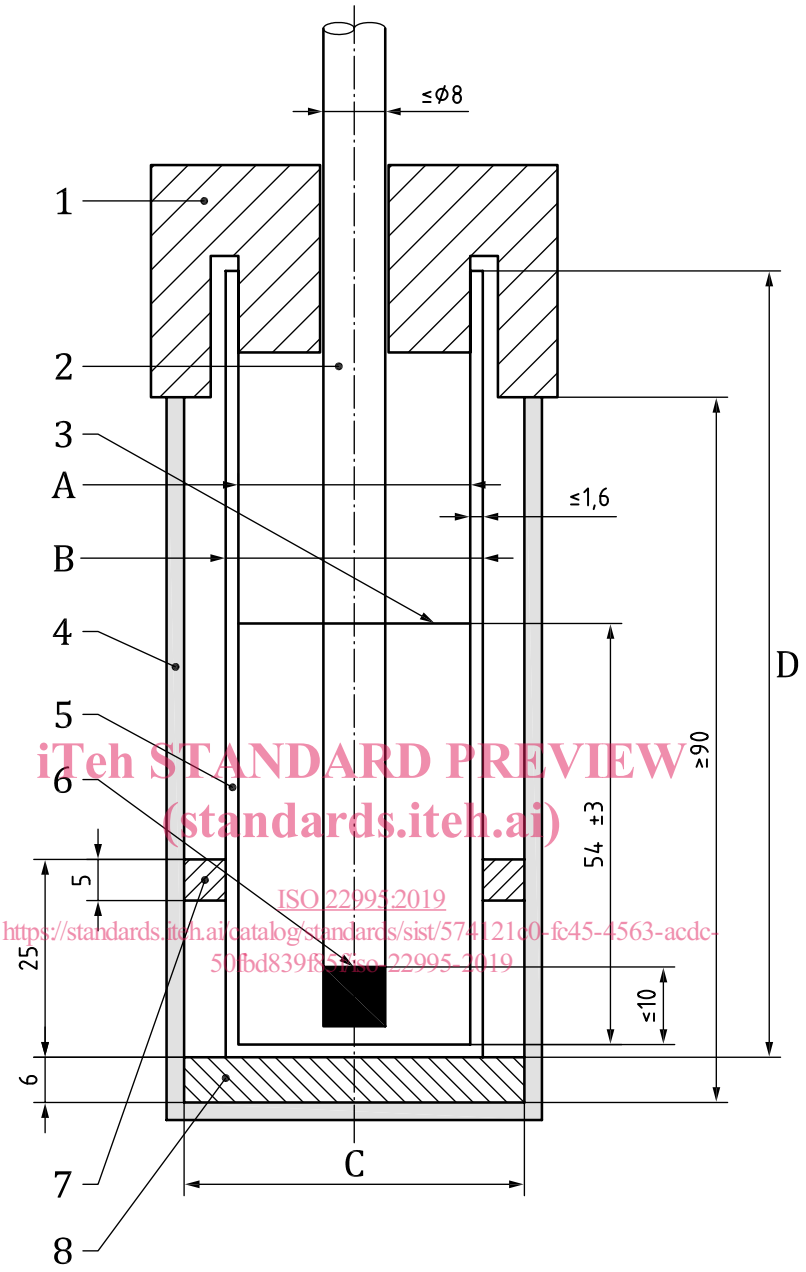
6.6 Gasket (optional, depending on the design of the equipment), ring form, about 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket.

This gasket can be made of rubber, leather or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

6.7 Automated detection device, consisting of an optical detection system able to detect cloud or wax crystals appearance in the sample every 1 °C at minimum.

6.8 Cooling device, cooling bath or device capable of maintaining the temperature of the jacket as required in Table 1.

Dimensions in millimetres



Key

1	anti-moisture device	5	test jar	A	Ø 30,0 mm to 32,4 mm
2	sample temperature probe	6	sensitive area	B	Ø 33,2 mm to 34,8 mm
3	sample filling mark	7	gasket (optional)	C	Ø 44,2 mm to 45,8 mm
4	jacket	8	disk (optional)	D	80 mm to 125 mm

Figure 1 — General overview of the measurement cell

7 Sampling

Unless otherwise specified in the commodity specification, samples shall be taken as described in ISO 3170 or ISO 3171.