INTERNATIONAL STANDARD

ISO 22995

First edition
2019-04

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

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

Reference number
ISO 22995:2019(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 ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Principle **1**
5 Reagents and materials **2**
6 Apparatus **2**
7 Sampling **3**
8 Sample preparation **4**
9 Preparation of the apparatus **4**
10 Procedure **4**
11 Expression of result **4**
12 Precision **5**
12.1 General 5
12.2 Repeatability, *r* 5
12.3 Reproducibility, *R* 5
13 Test report **5**
Bibliography **6**

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 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.

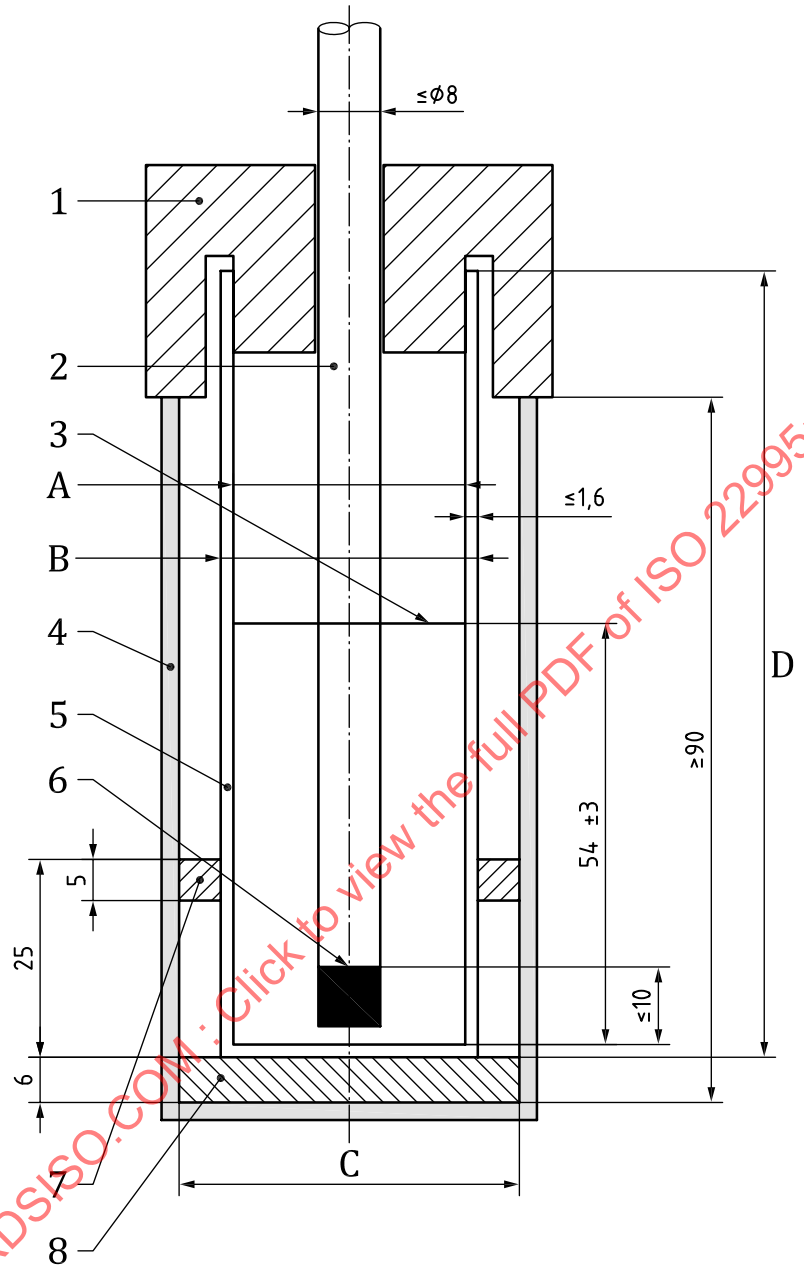
6.6 Gasket (optional, depending on the design of the equipment), ring form, about 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket.

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.

8 Sample preparation

The product to be tested shall be homogeneous and free of any visible particle and water. If not, bring the sample to be tested to a temperature at least 14 °C above the approximate cloud point, but not above 49 °C. Remove any moisture present by filtration through dry lintless filter paper (5.1), until the sample is perfectly clear, still working at the temperature of at least 14 °C above the approximate cloud point.

9 Preparation of the apparatus

Prepare the instrument for the test, according to the manufacturer's instructions.

10 Procedure

10.1 Pour the clear sample into the test jar (6.1) to the level mark.

10.2 Install the test jar into the jacket following the prescription of the manufacturer.

10.3 Ensure that the test jar does not touch the jacket. Gasket (6.6) and disk (6.5) may optionally be used.

10.4 Stepped cooling profiles

Maintain the temperature of the cooling bath at 0 °C ± 1,5 °C. As soon as the temperature of the sample reaches +9 °C, cool down the jacket to the next step (see Table 1) until the sample temperature reaches the low limit of the range and continue to the next step until detection of cloud point.

Table 1 — Bath temperature setting versus sample temperature range

Bath number	Bath temperature setting °C	Sample temperature range °C
1	0 ± 1,5	Start to +9
2	−18 ± 1,5	+9 to −6
3	−33 ± 1,5	−6 to −24
4	−51 ± 1,5	−24 to −42
5	−69 ± 1,5	−42 to −60

A maximum of 200 s is allowed to change the jacket temperature between each step.

If the detection is done outside the jacket, the test jar shall be extracted quickly but without disturbing the sample at each 1 °C sample temperature. The complete operation of cloud inspection shall not take more than 3 s.

If the detection is done inside the jacket, the cloud inspection may be done continuously or every 0,1 °C for instance.

11 Expression of result

Report as the cloud point the temperature at which any cloud is detected in the test jar.

If the analyser is able to give a better resolution than 1 °C, the temperature at which the cloud is detected shall be rounded down to the next lower integer (for example, if the cloud point is detected at +4,9 °C, the result is rounded down to +4 °C and if the cloud point is detected at −3,1 °C, the result is rounded down to −4 °C).

NOTE This way of reporting the result does not meet the resolution requirement of ISO 4259[3].

For internal use, the result given with the resolution of the equipment may be reported.

For comparing results between different automated equipment with different resolution and the manual procedure (see ISO 3015[2]), results should be rounded down to the next integer.

12 Precision

12.1 General

The precision, as determined by statistical examination in accordance with ISO 4259:2006[4], is given in 12.2 and 12.3.

The precision analysis is based on the results of an interlaboratory study (ILS) carried out in 2016. This study included FAME, distillate marine fuels (ISO-F-DMA), diesel fuels as well as paraffinic diesel fuels, covering the blending range from (0 to 30) % volume of FAME.

The range of cloud point values for the ILS was from −33 °C to +13 °C.

The ILS consisted of petroleum products of ISO 2049[1] colour of 5,0 and lower. The precisions stated in this document may not apply to samples with colour determined according to ISO 2049 higher than 5,0.

NOTE The ILS is documented in Reference [5].

12.2 Repeatability, *r*

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, is 1,1 °C.

12.3 Reproducibility, *R*

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, under different test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, is 2,5 °C.

13 Test report

The test report shall include at least the following information:

a) the type and complete identification of the product tested;

b) a reference to this document, i.e. ISO 22995:2019;

c) the result of the test (see Clause 11);

d) any deviation, by agreement or otherwise, from the procedure described (see Clause 10);

e) the date of test.