
**Petroleum and related products
from natural or synthetic sources —
Determination of pour point —**

**Part 1:
Automated step-wise cooling method**

*Produits pétroliers et connexes d'origine naturelle ou synthétique -
Détermination du point d'écoulement —*

Partie 1: Méthode automatisée par refroidissement par paliers

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

A list of all parts in the ISO 23877 series can be found on the ISO website.

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 the determination of pour point by automated instruments that mirrors ISO 3016 (which comprises a manual procedure only). This document is based on the techniques used in the instruments available on the market in 2021.

This test method does not contain any precision at this stage. As this is a newly described technique, no immediate precision or interim repeatability can be given either. At the time of publication, an interlaboratory study was ongoing, the results of which will be taken into account in future standardization activities.

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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 users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of this document, and to determine the applicability of any other restrictions.

1 Scope

This document specifies a method for determining the pour point of petroleum products by means of automated equipment, in which movement of the test specimen is detected using a step-wise cooling technique.

A separate procedure suitable for the determination of the lower pour point of fuel oils, heavy lubricant base stock, and products containing residual fuel components is also described.

The procedure described in this document is not suitable for crude oils.

Test results from this method are determined in 3 °C testing intervals.

NOTE Some equipment can also generate results at 1 °C testing intervals, which is an acceptable alternative procedure, but for which precision and bias have not been determined.

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*

ISO 17034, *General requirements for the competence of reference material producers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1

pour point

lowest temperature at which a sample of petroleum product will continue to flow when it is cooled under specified standard conditions

3.2

no-flow point

temperature of the test specimen at which a wax crystal structure or viscosity increase, or both, impeding movement of the surface of the test specimen under specified standard conditions

4 Principle

After preliminary heating, the sample is cooled at a specified rate and examined at intervals of 3 °C for flow characteristics. The lowest temperature at which movement ("pour" or "flow") of the sample is detected, by the automated equipment, is recorded as the pour point.

5 Reagents and materials

5.1 Certified reference materials (CRM), from suppliers in accordance with ISO 17034.

6 Apparatus

6.1 Test jar, cylindrical, of clear glass, flat-bottomed, 33,2 mm to 34,8 mm outside diameter and 110 mm to 125 mm in height. The test jar shall have an inside diameter of 30,0 mm to 32,4 mm, with the constraint that the wall thickness be no greater than 1,6 mm.

The jar shall be marked with a line to indicate a contents level, corresponding to a volume of 45 ml $\pm$ 1 ml.

6.2 Sample temperature probe, cylindrical, vertical in the test jar and plunged in the sample, depth and location specified by the manufacturer of the testing device (see also References [3] and [4]).

A thermometer with digital display (e.g. PT100) shall be used for measuring the sample temperature with a resolution of at least 0,1 °C and an accuracy of at least 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 bath, preventing the introduction of any moisture.

6.4 Jacket (test jar container), watertight, cylindrical metal, flat-bottomed, dimensions as per the manufacturer of the testing device. It shall be supported in a vertical position in the cooling device ([6.7](#)), and 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) approximately 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, approximately 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket.

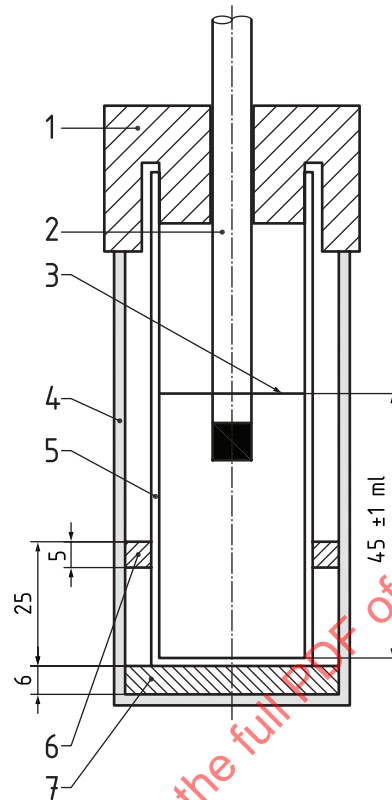
This gasket can be made of rubber, leather or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the gasket is to prevent the test jar from touching the jacket.

6.7 Cooling device, cooling bath or device with a firm support to hold the jacket vertical, capable of maintaining the temperature of the jacket as required in [Table 1](#).

6.8 Automated detection device, detection system able to detect movement of the sample by means of optics (optical reflection), charged-coupled device (CCD) camera, temperature change (thermal sense) or ultrasonics (sensor), as specified in [Annex A](#).

6.9 Timing device, capable of measuring up to 30 s with an accuracy of 0,2 s.

Dimensions in millimetres



Key

- | | | | |
|---|--------------------------------|---|-------------------------|
| 1 | anti-moisture device (6.3) | 5 | test jar (6.1) |
| 2 | sample temperature probe (6.2) | 6 | gasket (optional) (6.6) |
| 3 | sample filling mark (6.1) | 7 | disc (optional) (6.5) |
| 4 | jacket (6.4) | | |

Figure 1 — Apparatus for pour point test

7 Sampling

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

8 Preparation of apparatus

8.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

8.2 Clean and dry the automated detection device and test jar using suitable solvents as instructed by the manufacturer.

8.3 Turn on the main power switch of the automated instrument.

9 Calibration

9.1 Ensure that all of the manufacturer's instructions for calibrating, checking and operating the automated apparatus are followed.

9.2 Check the position of the temperature sensor and glass jar according to the manufacturer's instructions and, when necessary, make appropriate adjustments.

10 Verification of the performance of the apparatus

The correct functioning and performance of the automated apparatus should be verified preferably at least twice a year and, where possible, using certified reference materials (5.1)

The apparatus should also be checked more frequently (e.g. weekly) using an in-house secondary reference material with a known pour point value, or using an SQC sample (intralaboratory or intra-company) according to ISO 4259-4.

Deviations outside established statistical quality control limits should be investigated and be resolved. The manufacturer's instruction manual should provide guidance on ensuring that the equipment is correctly set up and calibrated.

11 Procedure

11.1 Pour the sample into the test jar (6.1) to the level mark. If necessary, heat the sample until it is just sufficiently fluid to pour into the test jar.

If it is necessary to heat the sample to a temperature greater than 45 °C to transfer to the test jar, keep the sample in the test jar at room temperature for 24 h before testing it. When it is known that a sample has been heated to a temperature higher than 45 °C during the preceding 24 h, or when the thermal history of the sample is not known, the sample shall be kept at room temperature for 24 h before testing it.

11.2 Subject the sample in the test jar (6.1) to a preliminary treatment, appropriate to its pour point, in accordance with 11.3 or 11.4, or use the instrument's automatic preheat option and insert the test jar into the apparatus as per manufacturer's instructions (also see 11.6).

11.3 Samples having pour points above –33 °C shall be treated as follows.

Heat the sample without stirring to 9 °C above the expected pour point in the bath maintained at 12 °C above the expected pour point, or to 45 °C in a bath maintained at 48 °C, whichever is greater.

11.4 Samples having pour points of –33 °C and below shall be treated as follows.

Heat the sample without stirring to 45 °C in the bath maintained at 48 °C ± 1,5 °C.

11.5 If the automatic preheat option has not been used, install the test jar into the jacket of the apparatus following the instructions of the manufacturer.

11.6 Check that the test jar does not touch the jacket (6.4), as per the manufacturer's instructions. Gasket (6.6) and disk (6.5) may optionally be used.

11.7 Select the test interval of 3 °C.

11.8 Enter the expected pour point (EP). If the EP is unknown, enter the room temperature. The EP shall be a multiple of 3 °C.

11.9 For stepped cooling profiles, the temperature of the cooling/heating device shall be regulated in accordance with the programmed temperature settings obtained from [Table 1](#). The instrument shall automatically change the block temperature in accordance with the sample temperature (see [Table 1](#)). Continue to lower (change) the temperature of the cooling/heating device to the next temperature step until detection of pour point. A maximum of 200 s is allowed to change the jacket temperature between each step.

NOTE This temperature is recorded by the sample temperature probe ([6.2](#)) and stored by the instrument.

Table 1 — Bath and sample temperature ranges

Bath	Bath temperature setting °C	Sample temperature range °C
1	12 above expected pour point, or $48 \pm 1,5$, whichever is greater	Preheat to at least 45 or 9 above expected pour point, whichever is greater
2	$24 \pm 1,5$	Start to 27
3	$0 \pm 1,5$	27 to 9
4	$-18 \pm 1,5$	9 to -6
5	$-33 \pm 1,5$	-6 to -24
6	$-51 \pm 1,5$	-24 to -42
7	$-69 \pm 1,5$	-42 to -60

11.10 Start the test in accordance with the manufacturer's instructions.

11.11 At this point the instrument shall monitor the test specimen for flow by means of the automatic detection device ([6.8](#)). The instrument shall start tilting the test specimen when the temperature of the test specimen is at 9 °C above EP. If movement of the test specimen is detected the specimen is returned to a vertical position.

11.12 The test will continue in this manner until a temperature is reached where the detector is not detecting any movement (no-flow point) in the test specimen when the test jar is in a complete horizontal position for at least 5 s.

11.13 If the instrument detects the no-flow point on the first tilting cycle (EP+9 °C), disregard the result and start with using a higher expected pour point.

11.14 For fuel oils, heavy lubricant base stock and products containing residual fuel components, the result obtained by the procedure given in [11.1](#) to [11.13](#) is the upper (maximum) pour point. If required, determine the lower (minimum) pour point by heating the sample while stirring to 105 °C, pouring it into the jar, and determining the pour point as given in [11.2](#) to [11.13](#).

12 Expression of results

When 3 °C testing interval is selected, add 3 °C to the temperature recorded in [11.12](#) and [11.14](#) and report this as the pour point. For fuel oils, heavy lubricant base stock and products containing residual fuel components (see [11.14](#)), add 3 °C to the temperature recorded in [11.12](#) and [11.14](#) and report this as the upper pour point or lower pour point, as required.

When an alternative testing interval is selected, for example 1 °C, proceed as above but add 1 °C to the temperature recorded instead of 3 °C. For ISO 3016, similar format determinations, an interval of 3 °C is required in any case.

13 Precision

The precision for lubricating oils, as determined by statistical examination in accordance with ISO 4259-1, is under development.

The precision for middle distillate and residual fuels, as determined by statistical examination in accordance with ISO 4259-1, is under development.

14 Test report

The test report shall contain at least the following information:

- a) sufficient details for complete identification of the product tested;
- b) a reference to this document, i.e. ISO/TS 23877-1:2023;
- c) used method of sampling (see [Clause 7](#));
- d) the result of the test (see [Clause 12](#));
- e) any deviation, by agreement or otherwise, from the procedures specified;
- f) the date of the test;
- g) report if an alternative testing interval than 3 °C was used.

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Annex A (normative)

Detection principles

A.1 Optics (optical reflection)

The test jar starts in a vertical position as in [Figure A.1, a\)](#). The test jar is mechanically manipulated according to the programmed test procedure, while optically observing the surface of the specimen for movement, by means of a light emitter (L) and light receiver (R) and recording the temperature of the specimen by means of a PT-100 probe [see [Figure A.1, b\)](#)]. If the test specimen is liquid after tilting, it is moved back to the vertical position for further cooling. If no movement is detected in the horizontal stage [[Figure A.1, c\)](#)], the specimen is considered to be solid, and the temperature at this point is recorded.

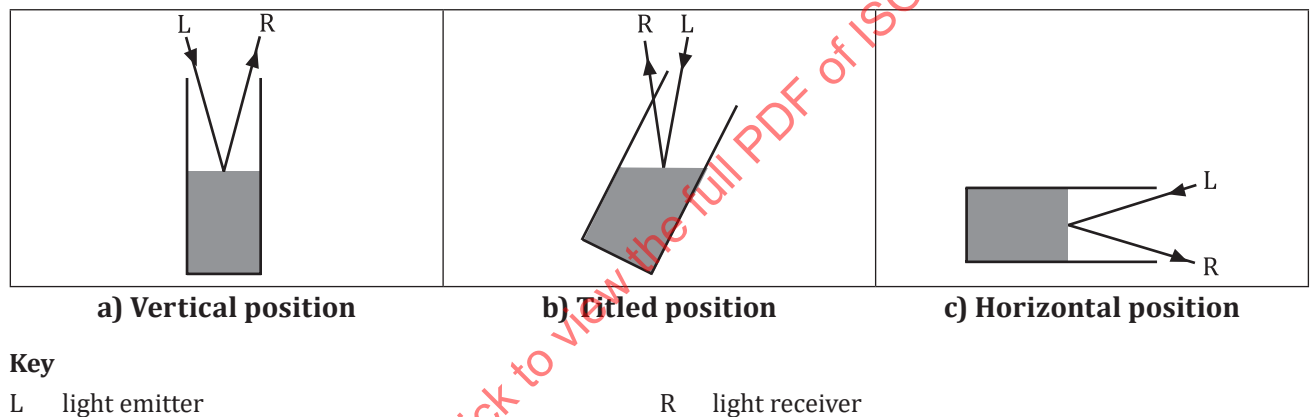
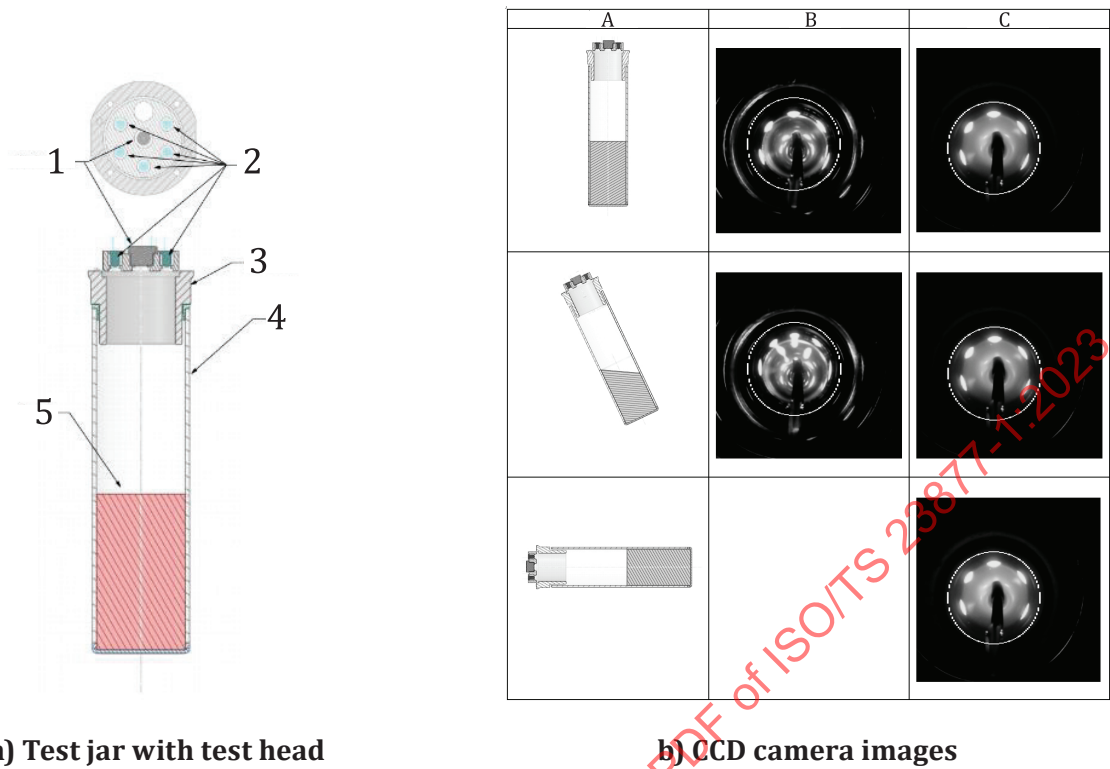


Figure A.1 — Detection using optics (optical reflection)

A.2 Charged-coupled device (CCD) camera

According to the programmed test procedure, the test jar (4) is lifted out of the bath jacket and tilted mechanically [see [Figure A.2 a\)](#)]. During this procedure, a CCD camera (1) system mounted on top of the test jar (4) optically observes the surface of the sample (5) for movement [see [Figure A.2 b\)](#)]. The test jar is tilted in different angles [see [Figure A.2 b\)](#), column A] after it is lifted out of the bath jacket. During this procedure, the five LED (2) mounted in the test head are activated, and the small light reflections coming from the surface of the sample are monitored by the camera for movement: flow [[Figure A.2 b\)](#), column B] and no flow [[Figure A.2 b\)](#), column C].



- Key**
- | | | | |
|---|------------|---|------------|
| A | angle | 2 | LED |
| B | flow | 3 | jar holder |
| C | no flow | 4 | test jar |
| 1 | CCD camera | 5 | sample |

Figure A.2 — Detection using a CCD Camera

Then the temperature of the specimen is recorded by means of a PT-100 probe. If no movement is detected when the specimen is in a fully horizontal position [Figure A.2 b), column A], the temperature at this point is recorded.

A.3 Temperature change (thermal sense)

The test jar is mechanically manipulated according to the programmed test procedure, while the surface of the specimen is observed for movement, using two PT-100 probes [pour point sensors, see Figure A.3, a) and b)] on top of the specimen (just touching the sample) in the test jar and recording the temperature of the specimen by means of another PT-100 probe [see Figure A.3, a) and b)].

When the specimen is in a fully horizontal position and there is no temperature change sensed by the two PT-100 probes (pour point sensors), it means no movement is detected. The temperature at this point is recorded.