Technical Specification

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

Part 2: Automated linear cooling method

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

Partie 2: Méthode automatisée par refroidissement linéaire

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Foreword

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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 automatic instruments. This document is based on the techniques used with 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. At the time of publication, an interlaboratory study was ongoing, the results of which will be taken into account in future standardization activities.

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

Part 2:
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1 Scope

This document specifies a method for determining the pour point of petroleum products by means of automatic equipment for detecting movement of the test specimen using a linear 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 can be determined in either 1 °C or 3 °C testing intervals

NOTE The equipment referenced in this method 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 the viscosity increase, or both, impede(s) movement of the surface of the test specimen under specified standard conditions

4 Principle

After preliminary heating, the sample is cooled at a specified linear rate and examined at intervals of 3 °C or 1 °C for flow characteristics. The pour point is recorded as the lowest temperature at which movement ("pour" or "flow") of the sample is observed.

5 Reagents and materials

Certified reference materials (CRM) shall be used from suppliers in accordance with ISO 17034.

6 Apparatus

The following apparatus shall be used.

6.1 Specimen cup (vial), automatic instruments, as specified by the manufacturer of the instrument.

6.2 Sample temperature probe, which is either:

a) cylindrical, vertical in the test jar and plunged in the sample; the depth and location are specified by manufacturer of the testing device; or

b) embedded in specimen holder.

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

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

6.3 Anti-moisture device, to close the specimen holder, avoiding any moisture to be introduced.

6.4 Specimen holder, location where the specimen cup/vial is placed for testing, e.g. metallic block or chamber.

6.5 Cooling device, device (integrated in the analyser) capable of controlling the specimen chamber temperature as specified by the manufacturer.

6.6 Automated detection device, detection system able to detect movement of the sample e.g. by means of optics or pressure, as specified in Annex A.

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 specimen cup (vial) (6.1) using suitable solvents and material or use a new cup, as instructed by the manufacturer.

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

9 Calibration

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

9.2 Check the position of the sample temperature probe (6.2) and specimen cup (vial) (6.1), in accordance with the manufacturer's instructions. 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 (Clause 5).

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 a statistical quality control (SQC) sample (intralaboratory or intra-company) according to ISO 4259-4.

Deviations outside established SQC 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 Introduce the sample into the specimen cup (vial) (6.1) to the level mark or required volume. If necessary, heat the sample until it is just sufficiently fluid to pour into the specimen cup (vial).

If it is necessary to heat the sample to a temperature greater than 45 °C to transfer to the specimen cup (vial) (6.1), keep the sample 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 specimen cup (vial) (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 specimen cup (vial) (6.1) into the apparatus as per manufacturer's instructions.

11.3 Samples having pour points above –33 °C shall be heated, 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 heated, 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 specimen cup (vial) (6.1) into the specimen holder (6.4) following the instructions of the manufacturer.

11.6 Select the test interval of 3 °C.

11.7 Enter the expected pour point (EP). If the EP is unknown, enter room temperature. If an interval of 3 °C has been selected, the EP shall be a multiple of 3 °C.

11.8 Follow the manufacturer's instructions (i.e. the cooling profiles) for regulating the temperature of the instruments. The cooling profiles are:

— Automatic pressure pulsing: 1,5 °C/min ± 0,1 °C/min.

— Automatic air pressure: 3 °C/min to 4 °C/min until EP 40 °C, after this 0,8 °C/min to 1,1 °C/min.

— Automatic pressure sensing: 10 °C/min ± 1 °C/min until EP 30 °C, after this 1,5 °C/min ± 0,15 °C/min .

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

11.10 At this point the instrument shall monitor the test specimen for flow by means of the automatic detection device (6.6). The instrument shall start when the temperature of the test specimen is at 9 °C above EP. If movement of the test specimen is detected, the specimen is cooled to the next testing interval.

11.11 The test will continue in this manner until a temperature is reached where the test specimen shows no movement when the specimen cup is checked (no-flow point). This temperature is recorded by the sample temperature probe (6.2) and stored by the instrument.

11.12 If the instrument detects the no-flow point during the first observation, disregard the result and start with using a higher expected pour point (EP).

11.13 For fuel oils, heavy lubricant base stock and products containing residual fuel components, the result obtained by the procedure given in 11.1 to 11.12 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 11.1 to 11.12.

12 Expression of results

When a testing interval of 3 °C is selected, add 3 °C to the temperature recorded in 11.11 and 11.13 and report this as the pour point. For fuel oils, heavy lubricant base stock and products containing residual fuel components (see 11.13), add 3 °C to the temperature recorded in 11.11 and 11.13 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 determinations that have a similar format to ISO 3016, an interval of 3 °C is required in any case.

13 Precision

The precision for lubricating oils, and 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 23877-2:2024;

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 a testing interval other than 3 °C was used;

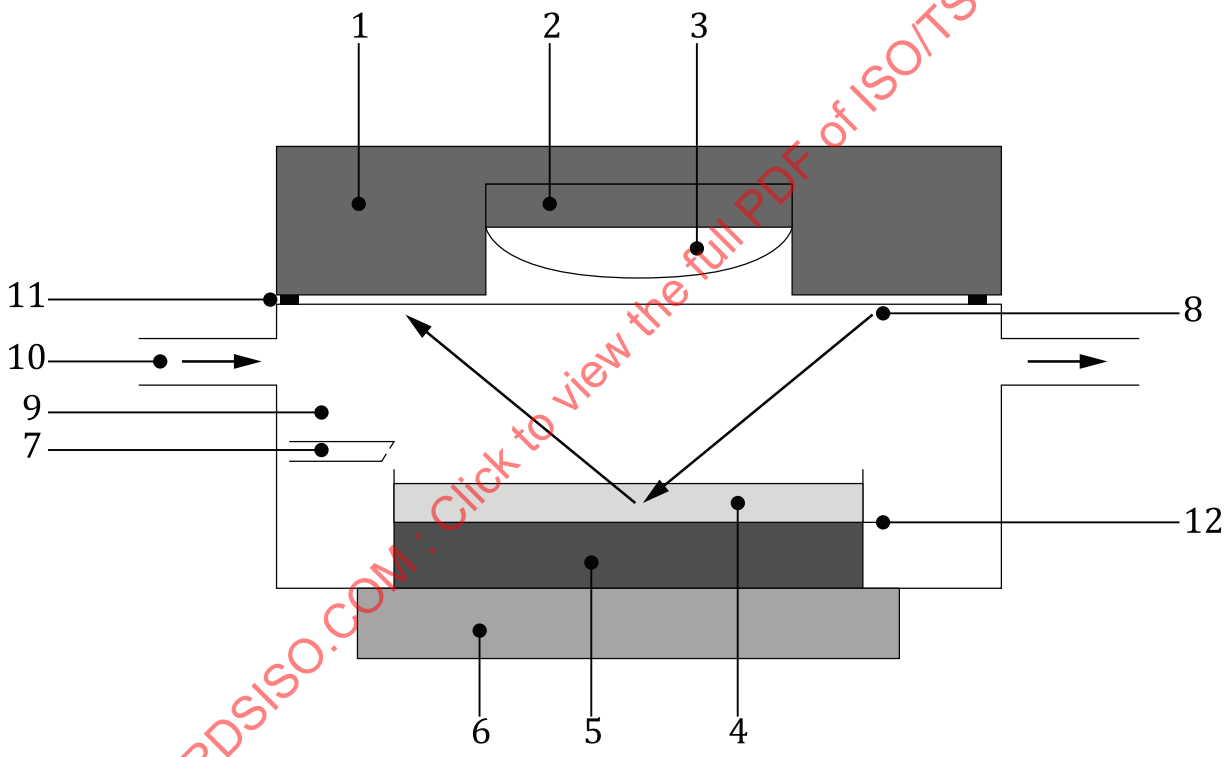
h) any unusual features observed.

Annex A
(normative)

Detection principles

A.1 Automatic pressure pulsing

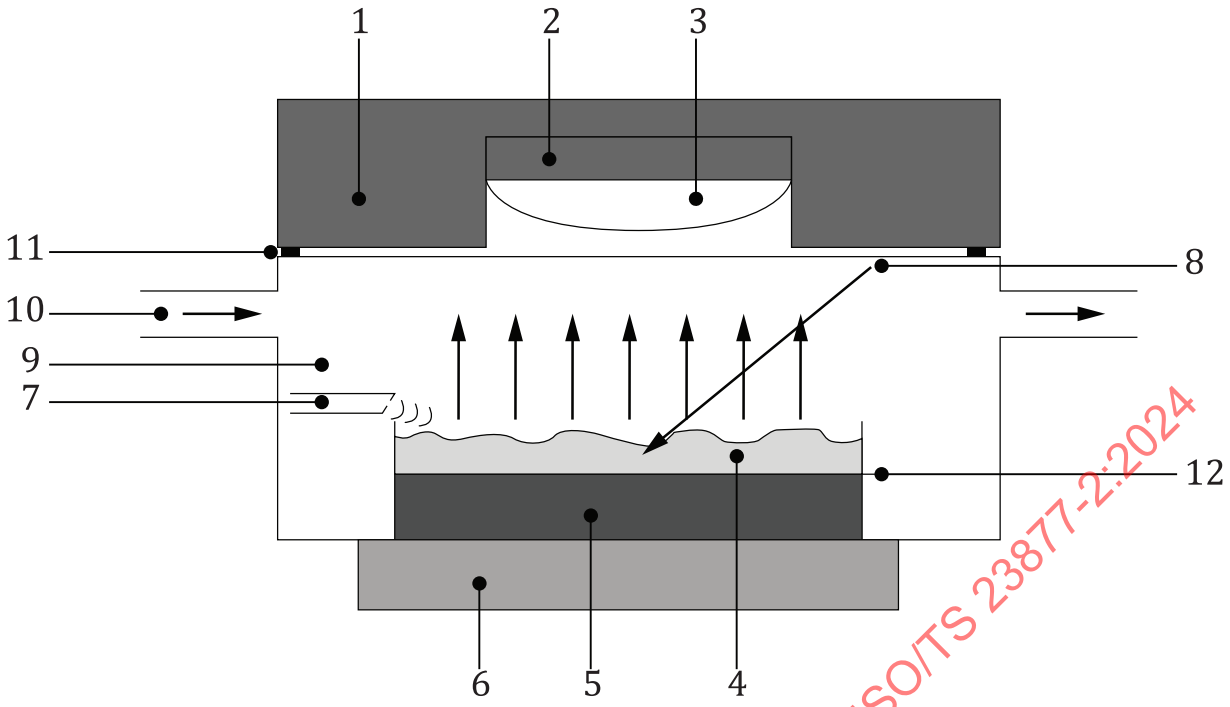
The specimen is illuminated by the light source (see Figure A.1) and the movement of the specimen surface upon application of a pulse is monitored by an array of optical detectors (see Figure A.2). The test will continue until application of a pulse of compressed gas causes no observable movement on the specimen surface. This is the no-flow point of the specimen. The pour point is recorded as the lowest temperature at which movement of the specimen surface is observed upon application of the pulse of compressed gas. The pour point value shall be displayed as an integer temperature in multiples of 1 °C or 3 °C, depending on the selected testing interval.



Key

1	lid	7	pressure pulse nozzle
2	optical detectors	8	light source
3	lens	9	chamber
4	specimen (cup) with mirrored surface	10	flow of purge gas
5	peltier cooling device	11	O-ring seal
6	heat sink with liquid or air cooled medium	12	temperature sensor

Figure A.1 — Schematic of test chamber



Key

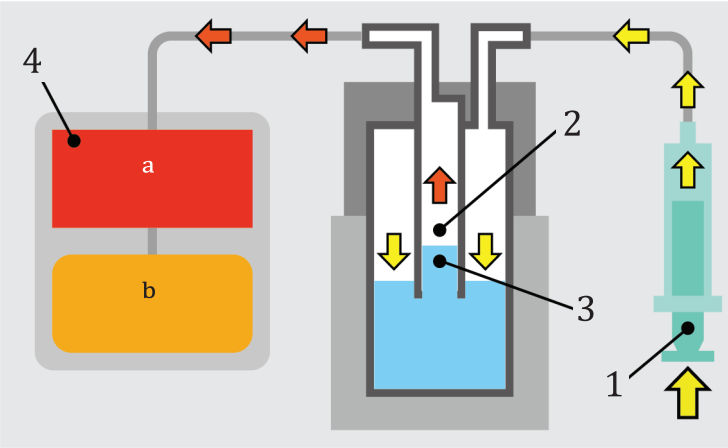
1	lid	7	pressure pulse nozzle
2	optical detectors	8	light source
3	lens	9	chamber
4	specimen (cup) with mirrored surface	10	purge gas
5	peltier cooling device	11	O-ring seal
6	heat sink with liquid or air cooled medium	12	temperature sensor

Figure A.2 — Detection of specimen movement using optical reflection

A.2 Automatic air pressure

The test specimen is preheated up to a specific temperature, then cooled at a controlled rate by the sequence programme. At certain preset points reached during cooling, the specimen surface is subjected to air pressure from the connected pressurizing syringe.

As shown in Figure A.3, pressure from the pressure syringe (key reference 1) is applied onto the circumference of the specimen surface. The central surface area of the specimen has the pressure conducting tube (key reference 2 of Figure A.3) inserted into it. The increased air pressure onto the circumference surface causes its surface level to fall slightly. As a consequence (using the principle of the U-tube), the surface level of the specimen inside the pressure conducting tube rises (key reference 3 of Figure A.3) and the pressure change caused by this rise is detected by the pressure sensor (key reference 4 of Figure A.3) confirming that the specimen has not yet solidified. Cooling of the specimen continues and the pour point detection is performed at the preset detection intervals. As the specimen movement reduces (which means solidification has started), the surface level outside the conducting tube becomes steady (see key reference 1 of Figure A.4), despite the application of increased air pressure. Therefore, no more pressure change occurs inside the conducting tube and no flow point has been detected. The last point at which pressure change was detected is determined as the pour point.

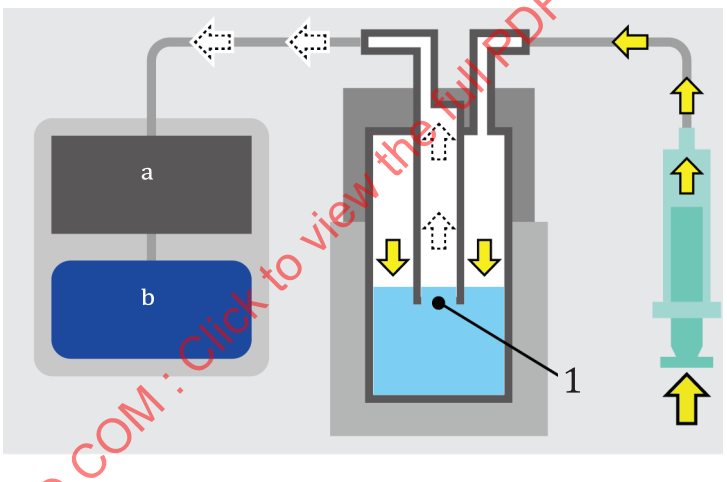


Key

1 pressure syringe
2 pressure conducting tube
3 level of test specimen
4 pressure sensor

a Pressure rise detected.
b Specimen is still flowing.

Figure A.3 — Schematic of fluid stage



Key

1 level of test specimen

a No pressure rise detected.
b Specimen is not flowing.

Figure A.4 — Detection of no flow point

A.3 Automatic pressure sensing

At the time the cooling begins, the pressure measurement system is engaged to continuously monitor specimen behaviour. A decrease in pressure, as determined by the apparatus (see Figures A.5 and A.6), is measured in the specimen vial, signifying that the test specimen has ceased to flow due to a crystal structure formation in the specimen or its viscosity has increased, or both. At this point, the temperature of the specimen chamber is recorded as the no flow point and held on a digital display. The test chamber is then reheated and the test sequence is terminated.