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

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

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 3016:2019

<https://standards.iteh.ai/catalog/standards/sist/363a5427-ffa3-4813-8fda-88c88d7e2877/iso-3016-2019>



iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO 3016:2019

<https://standards.iteh.ai/catalog/standards/sist/363a5427-ffa3-4813-8fda-88c88d7e2877/iso-3016-2019>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2019

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Apparatus	2
6 Sampling	3
7 Procedure	4
8 Expression of results	6
9 Precision	6
9.1 General	6
9.2 Lubricating oils	6
9.2.1 General	6
9.2.2 Repeatability	6
9.2.3 Reproducibility	6
9.3 Middle distillate and residual fuels	6
9.3.1 General	6
9.3.2 Repeatability	7
9.3.3 Reproducibility	7
10 Test report	7
Annex A (normative) Specifications of temperature measuring devices	8
Annex B (informative) Commonly used chemicals and freezing mixtures	10
Annex C (informative) Actual derived precision values	11
Bibliography	12

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 Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*. <https://standards.iteh.ai/catalog/standards/sist/363a5427-f8a3-4813-8fda-8b8817e28710/iso-3016-2019>

This third edition cancels and replaces the second edition (ISO 3016:1994), which has been technically revised. The main changes compared to the previous edition are as follows:

- inclusion of digital contact thermometer in [5.2.1](#);
- update of normative references in [Clause 2](#);
- chemicals and mixtures moved from former Clause 4 to [Annex B](#) (alignment with ISO 3015[1]);
- bath and sample temperature ranges have been aligned with ASTM D97[2], changes in bath temperature and the temperatures at which the test jars are moved to the batch with the next lower temperature have over the years (1994 up to the time of publication of this document) not led to observation of a bias versus test results obtained with the former edition;
- option for using automatic apparatus has been removed as being non-applicable;
- automated apparatus no longer being addressed as its use is at the discretion of the laboratory and the precision does not apply to that equipment;
- addition of sampling instructions in [Clause 6](#);
- alignment of [Clause 9](#) on precision, with ASTM D97[2] and introduction of [Annex C](#);
- addition of a Bibliography.

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.

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

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 the determination of the pour point of petroleum products. 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.

NOTE There is equipment available that uses an automated procedure similar to the one described in this document. However, the precision thereof has not been established¹⁾.

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*

ASTM D7962, *Practice for Determination of Minimum Immersion Depth and Assessment of Temperature Sensor Measurement Drift*

ASTM E2877, *Guide for Digital Contact Thermometers*

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

pour point

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

1) ISO develops an automated test method standard.

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 observed is recorded as the pour point.

5 Apparatus

5.1 Test jar, cylindrical, of clear glass, flat-bottomed, 33,2 mm to 34,8 mm outside diameter and 115 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 54 mm \pm 3 mm above the inside bottom representing 45 ml \pm 1 ml. See [Figure 1](#).

5.2 Temperature measuring device, one of the following:

5.2.1 Digital contact thermometer (DCT), meeting the requirements specified in [A.1](#).

5.2.2 Liquid-in-glass thermometers, partial immersion type conforming to the specifications given in [A.2](#).

Since separation of liquid column thermometers occasionally occurs and may escape detection, thermometers should be checked immediately prior to the test and used only if they prove accurate within ± 1 °C (for example ice point).

5.3 Cork, to fit the test jar, bored centrally for the test temperature measuring device.

5.4 Jacket, watertight, cylindrical metal, flat-bottomed, 115 mm \pm 3 mm in depth with inside diameter 44,2 mm to 45,8 mm, and a wall thickness of approximately 1 mm. It shall be supported in a vertical position in the cooling bath ([5.7](#)) so that no more than 25 mm projects out of the cooling medium, and shall be capable of being cleaned.

5.5 Disc, of cork or felt approximately 6 mm in thickness, to fit loosely inside the jacket.

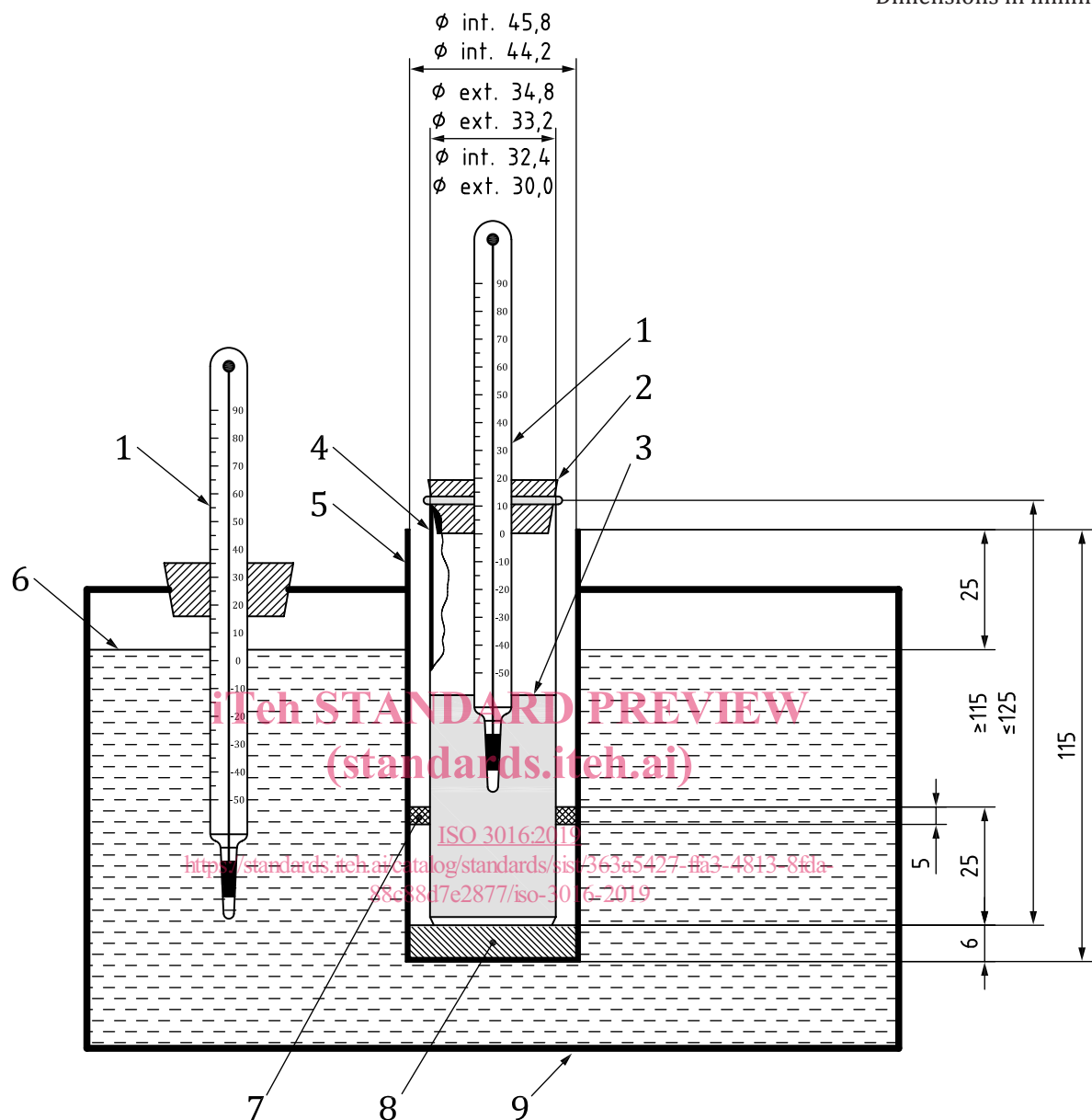
5.6 Gasket, ring form, approximately 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket. This gasket shall be made of rubber, leather or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape.

NOTE The purpose of the ring gasket is to prevent the test jar from touching the jacket.

5.7 Cooling baths, maintained at prescribed temperatures with a firm support to hold the jacket vertical. The required bath temperatures may be obtained by refrigeration if available, otherwise by suitable cooling mixtures. Cooling mixtures commonly used for bath temperatures are given in [Annex B](#).

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

Dimensions in millimetres



Key

- | | | | |
|---|-------------------------|---|--------------------|
| 1 | thermometer (5.2) | 6 | coolant level |
| 2 | cork (5.3) | 7 | gasket (5.6) |
| 3 | surface of the specimen | 8 | disc (5.5) |
| 4 | test jar (5.1) | 9 | cooling bath (5.7) |
| 5 | jacket (5.4) | | |

Figure 1 — Apparatus for pour point test

6 Sampling

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

7 Procedure

7.1 Pour the sample into the test jar to the level mark. If necessary, heat the sample in a water bath 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 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.

7.2 In the case of pour points above 36 °C, use a higher range temperature measuring device (5.2) such as IP 63C or ASTM 61C, or a digital contact thermometer. Close the test jar with the cork carrying the test temperature measuring device (5.2). Adjust the position of the cork and temperature measuring device so the cork fits tightly, the temperature measuring device and the jar are coaxial, and the temperature measuring device is immersed to the correct depth.

7.2.1 For liquid-in-glass, the thermometer bulb should be immersed so the beginning of the capillary is 3 mm below the surface of the specimen.

7.2.2 For digital contact thermometers, the probe should be immersed so the end of the probe is 10 mm to 15 mm below the surface of the specimen.

7.3 Subject the sample in the test jar to a preliminary treatment, appropriate to its pour point, in accordance with 7.4 or 7.5.

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

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

7.4.2 Transfer the test jar to a bath maintained at 24 °C ± 1,5 °C and commence observations for pour point. When using a liquid bath, ensure that the liquid level is between the fill mark on the test jar and the top of the test jar.

7.4.3 When the sample temperature reaches 9 °C above the expected pour point (estimated as a multiple of 3 °C), commence observations for flow in accordance with 7.7.

7.4.4 If the sample has not ceased to flow when the temperature has reached 27 °C, carefully remove the test jar from the bath, wipe the outside surface with a clean cloth moistened with wiping fluid and place it in the 0 °C bath (5.7) in accordance with 7.6. Make observations for flow in accordance with 7.7 and cool as specified in the schedule given in 7.8.

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

7.5.1 Heat the sample without stirring to 45 °C in a bath maintained at 48 °C ± 1,5 °C. Transfer the test jar to a bath maintained at 24 °C ± 1,5 °C. When using a liquid bath, ensure that the liquid level is between the fill mark on the test jar and the top of the test jar. When the specimen temperature reaches 27 °C, and if using liquid-in-glass thermometers, remove the high cloud and pour thermometer, and place the low cloud and pour thermometer in position. Transfer the test jar to the cooling bath (see 7.8).

7.6 Ensure that the disc (5.5), gasket (5.6) and the inside of the jacket (5.4) are clean and dry, and place the disc in the bottom of the jacket. The disc and jacket shall have been placed in the cooling medium (5.7) a minimum of 10 min before the test jar is inserted. Place the gasket around the test jar approximately

25 mm from the bottom, and insert the test jar into the jacket. Never place a test jar directly into the cooling medium.

7.7 Carry out observations for flow.

7.7.1 At each thermometer reading that is a multiple of 3 °C below the temperature of the first observation, remove the test jar from the bath or jacket, as applicable, and tilt it just sufficiently to ascertain whether there is movement in the sample in the test jar. Typically, the complete operation of removal, wiping and replacement should not require more than 3 s.

7.7.2 Continue observations at each thermometer reading that is a multiple of 3 °C below the temperature of the first observation.

After the specimen has cooled to allow the formation of paraffin wax crystals, take great care not to disturb the mass of specimen nor permit the thermometer to shift in the specimen; any disturbance of the spongy network of wax crystals will lead to low and erroneous results.

At low temperatures, condensed moisture can limit visibility. This may be removed by wiping the outside surface of the test jar with a clean cloth moistened with wiping fluid (e.g. ethanol or methanol) close to the bath temperature.

7.7.3 As soon as the sample does not flow when tilted, hold the test jar in a horizontal position for 5 s, as measured by the timing device (5.8), and observe carefully. If the sample shows any movement, replace the test jar immediately in the bath or jacket, as applicable, and repeat the observation at the next temperature, 3 °C lower.

7.7.4 Continue in this manner until a temperature is reached where the sample shows no movement when the test jar is held in a horizontal position for 5 s. Record the observed reading of the thermometer.

7.8 If the sample has not ceased to flow when its temperature has reached 27 °C, transfer the test jar to a jacket in a cooling bath maintained at 0 °C ± 1,5 °C. As the specimen continues to get colder, transfer the test jar to a jacket in the next lower temperature cooling bath in accordance with Table 1.

Table 1 — Bath and sample temperature ranges

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

7.9 To determine conformity with existing obsolescent specifications having pour-point limits at temperatures not divisible by 3 °C, it is acceptable practice to conduct the pour-point measurement according to the following schedule.

Begin to examine the appearance of the sample when the temperature of the sample is 9 °C above the specification pour point. Continue observations at 3 °C intervals in accordance with 7.7 and 7.8 until the specification temperature is reached. Report the sample as passing or failing the specification limit.