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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

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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

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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 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).

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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*.

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 established[1].

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

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[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 ± 3 mm above the inside bottom representing 45 ml ± 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 ± 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.

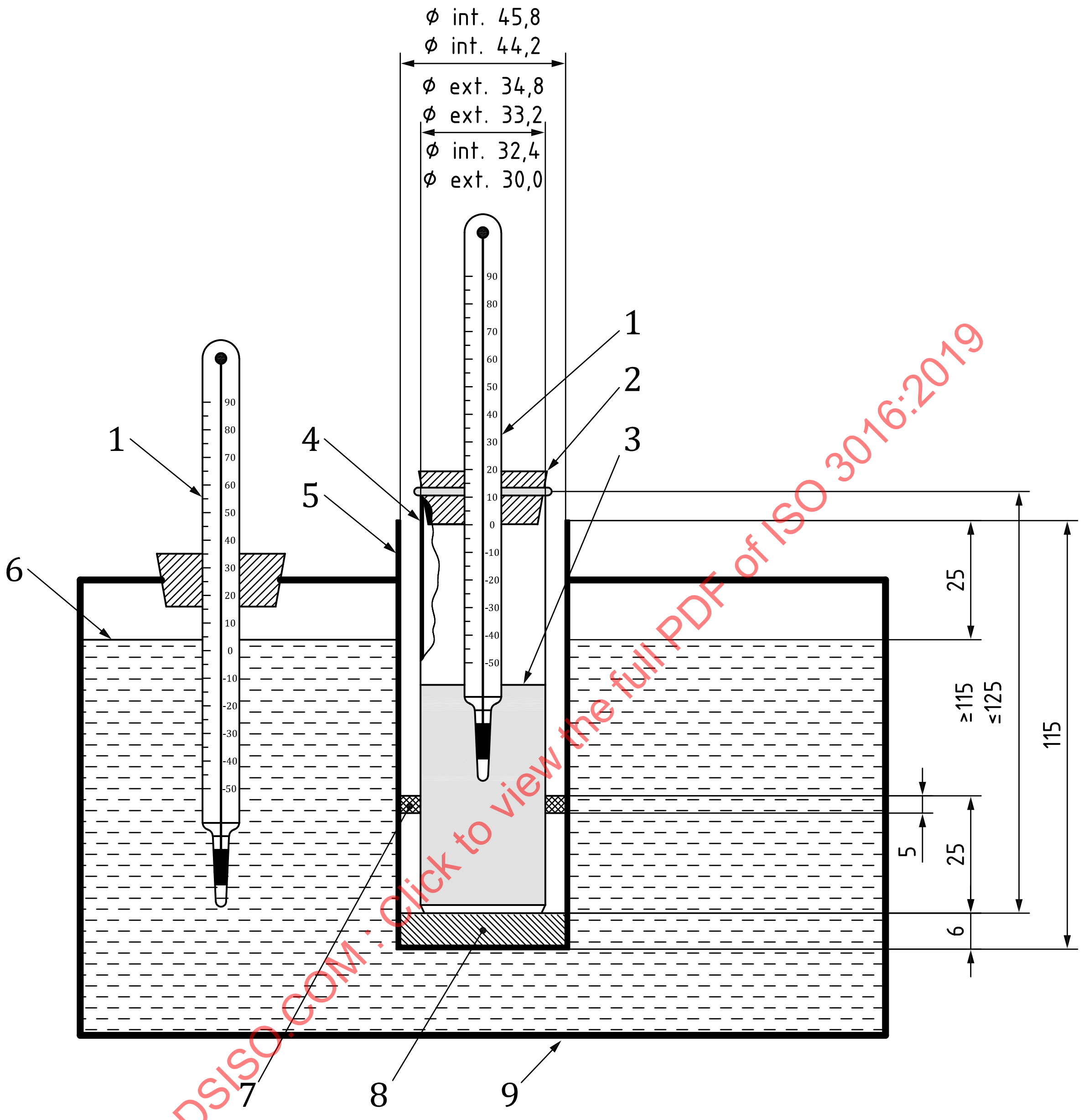
5.6 Gasket, ring form, approximately 5 mm in thickness, to fit snugly on the outside of the test jar and loosely inside the jacket. This gasket shall be made of rubber, leather or other suitable material, elastic enough to cling to the test jar and hard enough to hold its shape.

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.

7.10 For fuel oils, heavy lubricant base stock and products containing residual fuel components, the result obtained by the procedure given in 7.1 to 7.8 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 7.2 to 7.8.

8 Expression of results

Add 3 °C to the temperature recorded in 7.7.4 and 7.10 and report this as the pour point, or lower pour point, as applicable.

9 Precision

9.1 General

The precision statements were developed using liquid-in-glass thermometers corresponding to those in ASTM E1[6] or IP specifications for IP standard thermometers.

The precision statements are the derived values rounded up to the next testing interval value. The actual derived precision values are given in Annex C.

9.2 Lubricating oils

9.2.1 General

The precision statements were derived from a 1998 interlaboratory test program using ASTM D6300[3]. Participants analysed five sets of duplicate base oils, three sets of duplicate multigrade lubricating oils, and one set each of duplicate hydraulic oils and automatic transmission fluid in the temperature range of −51 °C to −11 °C. Seven laboratories participated with the manual test method[2]. Information on the type of samples and their average pour points are given in Reference [4].

9.2.2 Repeatability

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 6 °C only in one case in 20. Differences greater than this should be considered suspect.

9.2.3 Reproducibility

The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 9 °C only in one case in 20. Differences greater than this should be considered suspect.

9.3 Middle distillate and residual fuels

9.3.1 General

The precision statements were prepared with data on 16 middle distillate and residual fuels tested by 12 co-operators[5]. The fuels had pour points ranging from −33 °C to +51 °C.

NOTE Based on the results of the 1983 interlaboratory cooperative test program.

9.3.2 Repeatability

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed 3 °C only in one case in 20. Differences greater than this should be considered suspect.

9.3.3 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed 9 °C only in one case in 20. Differences greater than this should be considered suspect.

10 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 3016:2019;

c) the result of the test (see Clause 8);

d) any deviation, by agreement or otherwise, from the procedures specified (see Clause 7);

e) the date of the test.

Annex A
(normative)

Specifications of temperature measuring devices

A.1 Electronic

A digital contact thermometer (DCT) conforming to the specifications in Table A.1 shall be used.

Table A.1 — Digital contact thermometer (DCT) requirements

Parameter	Requirement
DCT	ASTM E2877 Class F or better
Nominal temperature range[a]	High pour: −38 °C to +50 °C Low pour: −80 °C to +20 °C Melting point: 32 °C to 127 °C
Display resolution	0,1 °C minimum
Accuracy[b]	±500 mK (±0,5 °C)
Sensor type	Platinum resistance thermometer (PRT), thermistor
Sensor sheath[c]	4,2 mm O.D. maximum
Sensor length[d]	Less than 10 mm
Immersion depth[e]	Less than 40 mm per ASTM D7962
Sample immersion depth	Tip of sheath between 10 mm and 15 mm below sample meniscus. See Figure 1
Response time[f]	Less than or equal to 4 s[f]
Measurement drift	Less than 500 mK (0,5 °C) per year
Calibration error	Less than 500 mK (0,5 °C) over the range of intended use
Calibration range	Consistent with temperature range of use
Calibration data	Four data points evenly distributed over the calibration range that is consistent with the range of use. The calibration data are to be included in the calibration report.
Calibration report	From a calibration laboratory with demonstrated competency in temperature calibration which is traceable to a national calibration laboratory or metrology standards body

[a] The nominal temperature range temperature may be different from the values shown, provided that the calibration and accuracy criteria are met.

[b] Accuracy is the combined accuracy of the DCT unit which is the display and sensor.

[c] Sensor sheath is the tube that holds the sensing element. The value is the outside diameter of the sheath segment containing the sensor element.

[d] The physical length of the temperature sensing element.

[e] As determined by ASTM D7962 or an equivalent procedure.

[f] Response time is a percentage of the time it takes a DCT to respond to a step change in temperature. The response time is 63,2 % of the step change time as determined per Section 9 of the test method in ASTM E644-11[8]. The step change evaluation begins at 20 °C ± 5 °C air to 77 °C ± 5 °C with water circulating at 0,9 m/s ± 0,09 m/s past the sensor.

When the DCT display is mounted on the end to the probe's sheath, the test jar with the probe inserted will be unstable. To resolve this, it is recommended that the probe be less than 30 cm in length but no less than 15 cm. A 5 cm long stopper that has a low thermal conductivity, with approximately half of it inserted in the sample tube, will improve stability.

When making measurements below −40 °C with a PRT, it may be necessary to use a 1 000 Ω sensor in order to obtain accurate measurements.

The DCT calibration drift shall be checked at least annually. This check is done by either measuring the ice point, or against a reference thermometer in a constant temperature bath at the prescribed immersion depth to ensure conformity with the measurement drift as in Table A.1. See the practice in ASTM D7962.

NOTE A DCT calibration drift in one direction over several calibration checks, is an indication of deterioration of the DCT.

A.2 Liquid-in-glass thermometers

Partial-immersion type thermometers conforming to the specifications in Table A.2 shall be used.

Table A.2 — Specification of glass-in-liquid thermometers

Specification	Low cloud and pour	High cloud and pour	Melting point
Range, °C	−80 to +20	−38 to +50	32 to 127
Immersion length, mm	76	108	79
Graduation at each °C	1	1	0,2
Longer lines at each °C	5	5	1
Figured at each °C	10	10	2
Scale error, max °C	1 (≥−33 °C) 2 (<−33 °C)	0,5	0,2
Expansion chamber to permit heating to °C	60	100	150
Overall length, mm	230 ± 5	230 ± 5	380 ± 5
Stem diameter, mm	6 to 8	6 to 8	6 to 8
Bulb length, mm	7,0 to 10	7,0 to 10	18 to 28
Bulb diameter, mm	5,0 to stem	5,5 to stem	5,0 to 6,0
Distance from bottom of bulb to line at °C in mm	−70 100 to 120	−38 120 to 130	32 105 to 115
Length of scale/range, mm	70 to 100	65 to 85	200 to 240

NOTE 1 The emergent-stem temperature is 21 °C throughout the scale range.

NOTE 2 Liquid-in-glass thermometers, having ranges shown in Table A.3 and conforming to the requirements as prescribed in ASTM E1[6] or ASTM E2251[7], or specifications for IP standard thermometers[9], meet the specifications in Table A.2.

Table A.3 — Conforming liquid-in-glass thermometers

		Thermometer number	
Thermometer	Temperature range	ASTM	IP
High cloud and pour	−38 °C to +50 °C	5C, S5C	1C
Low cloud and pour	−80 °C to +20 °C	6C	2C
Melting point	+32 °C to +127 °C	61C	63C

Annex B
(informative)

Commonly used chemicals and freezing mixtures

The following reagents and materials should be considered.

B.1 **Acetone**, technical grade, is suitable for the cooling bath, provided it does not leave a residue on drying.

WARNING — Extremely flammable.

B.2 **Carbon dioxide (solid).**

B.3 **Ice**, commercial grade of dry ice, is suitable for use in the cooling bath.

B.4 **Petroleum naphtha,** commercial or technical grade, is suitable for use in the cooling bath.

WARNING — Combustible. Vapor harmful.

B.5 **Sodium chloride crystals**, commercial or technical grade sodium chloride, is suitable for use in the cooling bath.

B.6 **Sodium sulfate**, reagent grade of anhydrous sodium sulfate should be used when required.

B.7 **Ethanol,** Ethyl Alcohol, commercial or technical grade of dry ethanol, is suitable for the cooling bath.

WARNING — Flammable. Denatured, cannot be made nontoxic.

B.8 **Methanol**, Methyl Alcohol, commercial or technical grade of dry methanol, is suitable for the cooling bath.

WARNING — Flammable. Vapor harmful. Toxic.

B.9 **Cooling mixtures**, as described in Table B.1, commonly used for bath temperatures.

Table B.1 — Cooling mixtures and bath temperatures

Cooling mixture	Bath temperature
Ice and water	0 °C ± 1,5 °C
Crushed ice and sodium chloride crystals, or acetone or petroleum naphtha or methanol or ethanol with solid carbon dioxide added to give the desired temperature	−18 °C ± 1,5 °C
Acetone or petroleum naphtha or methanol or ethanol with solid carbon dioxide added to give the desired temperature	−33 °C ± 1,5 °C
Acetone or petroleum naphtha or methanol or ethanol with solid carbon dioxide added to give the desired temperature	−51 °C ± 1,5 °C
Acetone or petroleum naphtha or methanol or ethanol with solid carbon dioxide added to give the desired temperature	−69 °C ± 1,5 °C