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Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity

Produits pétroliers — Liquides opaques et transparents — Détermination de la viscosité cinématique et calcul de la viscosité dynamique

Reference number
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Contents

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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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*, in collaboration with 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 accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 3104:2020), which has been technically revised.

The main changes are as follows:

- manual Procedure A has been designated as the referee test method in case of dispute;
- the DCT requirements have been updated in Table 1;
- allowable DCT drift in 7.3 has been aligned with Table 1;
- extra instructions for quality control have been added referring to ISO 4259-4;
- complying thermometers have been updated in Table B2;
- the calculation has been corrected in Annex D.

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

Many petroleum products and some non-petroleum materials are used as lubricants. The correct operation of equipment depends upon the appropriate viscosity of the liquid being used. In addition, the viscosity of many petroleum fuels is important for the estimation of optimum storage, handling and operational conditions. Thus, the accurate measurement of viscosity is essential to many product specifications.

This document describes two test methods: Procedure A (manual) and Procedure B (automated). Procedure A is the referee test method (or reference test method) to resolve doubts or dispute.

Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity

WARNING — This document does not purport to address all of the safety problems, if any, 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 the application of this document, and to determine the applicability of any other restrictions.

1 Scope

This document specifies Procedure A, using manual glass viscometers, and Procedure B, using glass capillary viscometers in an automated assembly, for the determination of the kinematic viscosity, ν, of both transparent and opaque products. The scope includes liquid petroleum products, fatty acid methyl ester (FAME), paraffinic diesel, hydrotreated vegetable oil (HVO), gas to liquid (GTL) and biofuel diesel mixtures up to 50 % FAME. The kinematic viscosity is determined by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity, η, is obtained by multiplying the measured kinematic viscosity by the density, ρ, of the liquid. The range of kinematic viscosities covered in this test method is from 0,2 mm^2/s to 300 000 mm^2/s over the temperature range −20 °C to +150 °C.

NOTE The result obtained from this document is dependent upon the behaviour of the sample and is intended for application to liquids for which primarily the shear stress and shear rates are proportional (Newtonian flow behaviour). If, however, the viscosity varies significantly with the rate of shear, different results can be obtained from viscometers of different capillary diameters. The procedure and precision values for residual fuel oils, which under some conditions exhibit non-Newtonian behaviour, have been included.

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 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ASTM E2877-12, *Standard 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 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
kinematic viscosity

ν

resistance to flow of a fluid under gravity

Note 1 to entry: For gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density, ρ. For any particular viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, ν:

$$\nu = \eta / \rho$$

where η is the *dynamic viscosity* (3.2) coefficient.

3.2
dynamic viscosity

η

ratio between the applied shear stress and rate of shear of a liquid

Note 1 to entry: It is a measure of the resistance to flow or deformation of a liquid.

Note 2 to entry: The term dynamic viscosity is also used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence.

Note 3 to entry: Dynamic viscosity may also be called coefficient of dynamic viscosity or absolute viscosity.

3.3
density

ρ

mass per unit volume of a substance at a given temperature

4 Principle

The time is measured for a fixed volume of liquid to flow under gravity through the glass capillary of a calibrated viscometer under a reproducible driving head and at a known and closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

5 Reagents and materials

5.1 Cleaning solution, strongly-oxidizing cleaning solution or alkaline cleaning solutions can be used.

Alkaline cleaning solutions with a pH of greater than 10 are not recommended as they have been shown to change the viscometer calibration. If these are used, then the viscometer calibration should be verified to ensure there is no change.

5.2 Sample solvent, completely miscible with the sample. A prewash of an aromatic solvent such as toluene or heptane can be necessary to remove asphaltenic material. When cleaning capillaries inside the bath, the boiling point of the cleaning solution shall be higher than the bath temperature.

5.3 Drying solvent, suitable and volatile at the used temperature. Filter before use. If moisture remains, use a drying solvent miscible with water (5.4).

NOTE When cleaning capillaries inside the bath and if the bath temperature is higher than 50 °C, acetone is not suitable.

5.4 Water, deionized or distilled, conforming to Grade 3 of ISO 3696. Filter before use.

5.5 Certified viscosity reference standards (CRM), produced by a reference material producer and meeting the requirements of ISO 17034. They shall be characterized in accordance with a standard practice for the basic calibration of master viscometers and characterization of viscosity oils, such as in ASTM D2162-21. The certified values shall be traceable to the international agreed value of distilled water (1,003 4 mm^2/s at 20 °C as specified in ISO/TR 3666).

6 Apparatus

6.1 Drying tubes, consisting of a desiccant drying system of either externally mounted drying tubes or an integrated desiccant drying system which is designed to remove ambient moisture from the capillary tube. Ensure that they are packed loosely and that the desiccant is not saturated with water.

6.2 Sample filter, micron screen or fretted (sintered) glass filter, no more than 75 µm.

6.3 Reagent filter, micron screen or fretted (sintered) glass filter, no more than 11 µm.

6.4 Ultrasonic bath, unheated, with an operating frequency between 25 kHz to 60 kHz and a typical power output of ≤100 W, of suitable dimensions to hold container(s) placed inside of bath, for use in effectively dissipating and removing air or gas bubbles that can be entrained in viscous sample types prior to analysis. It is permitted to use ultra-sonic baths with operating frequencies and power outputs outside this range. However, it is the responsibility of the laboratory to conduct a data comparison study to confirm that the results determined with and without the use of such ultrasonic baths do not materially impact results.

6.5 Manual apparatus

6.5.1 Glass capillary viscometer, calibrated in accordance with ISO 3105.

The viscometer shall have a certificate of calibration provided by a laboratory that meets ISO/IEC 17025. The calibration constant should be checked before first use of the capillary and only changed if necessary.

The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration. The variation in the value of g across the earth's surface is about 0,5 % due to latitude plus approximately 0,003 % per 100 m altitude. Apply a gravity correction to the viscometer calibration constant as in Formula (1), if the acceleration of gravity of the testing laboratory differs by more than 0,1 % of the calibration laboratory.

$$C_2 = \left(\frac{g_2}{g_1}\right) C \qquad (1)$$

where the g_1 and g_2 are, respectively, the calibration laboratory and the testing laboratory.

NOTE Calculation of acceleration of gravity values can be found in Reference [25].

IMPORTANT — Viscometers used for silicone fluids, fluorocarbons and other liquids, which are difficult to remove using a cleaning agent, shall be reserved for the exclusive use of those fluids, except during their calibration. Subject such viscometers to calibration checks at frequent intervals. The solvent washings from these viscometers shall not be used for the cleaning of other viscometers. If the viscometer is cleaned using the material in 5.1 then the user shall verify the calibration before further use.

6.5.2 Viscometer holder or mounting device within the temperature-controlled bath, enabling the glass viscometer to be suspended so that the upper meniscus is directly above the lower meniscus vertically within 1° in all directions.

Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within 0,3° in all directions in accordance with ISO 3105.

The proper alignment of vertical parts may be confirmed by using a plumb line, but for rectangular baths with opaque ends, this may not be possible.

6.5.3 Temperature-controlled bath, containing a transparent liquid of sufficient depth such that at no time during the measurement is any portion of the sample in the viscometer less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

Temperature control of the bath liquid shall be such that, for each series of flow-time measurements, within the range of 15 °C to 100 °C, the temperature of the bath medium does not vary by more than ±0,02 °C from the selected temperature over the length of the viscometer, and/or between the position of each viscometer, and/or at the location of the temperature measuring device. In other words, the temperature shall be constant at the capillary and at the position of the temperature measuring device within a maximum difference of 0,04 °C. For temperatures outside this range, the deviation from the desired temperature shall not exceed ±0,05 °C.

Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.5.3, in accordance with the conditions given in Annex B and any corrections supplied on the certificates of calibration for the temperature measuring device. Maintain the bath temperature at the test temperature using the readings of the temperature measuring device with the corrections supplied by the certificate of calibration.

The temperature measuring device shall be held in an upright position under the same conditions of immersion as when calibrated.

6.5.4 Temperature-measuring device, for the range 0 °C to 100 °C, being either:

a) a calibrated liquid-in-glass thermometer, as listed in Annex B with a calibration and measurement capability (CMC) of ±0,04 °C after correction or better, or

b) a digital contact thermometer (DCT) as described in Table 1 for this temperature with equal or better CMC.

NOTE 1 A DCT is preferred due to the lower uncertainty of measurement.

The calibration data should be traceable to a calibration or metrology standards body and meet the uncertainty of measurement required. The calibration certificate shall include data covering the series of temperature test points which are appropriate for its intended use. When two temperature measuring devices are used in the same bath in this range, they shall agree within 0,04 °C.

If calibrated liquid-in-glass thermometers are used, the use of two thermometers is recommended.

Outside the range 0 °C to 100 °C, a calibrated liquid in-glass thermometer with a CMC of ±0,1 °C or better shall be used, and when two temperature measuring devices are used in the same bath, they shall agree within ±0,1 °C.

When using liquid-in-glass thermometers, use a magnifying device to read the thermometer to the nearest 1/5 division (e.g. 0,01 °C or 0,02 °C) to ensure that the required test temperature and temperature control capabilities are met. It is recommended that thermometer readings (and any corrections supplied on the certificates of calibrations for the thermometers) be recorded on a periodic basis to demonstrate compliance with the test method requirements.

A DCT which meets the requirements in Table 1 shall be used. The DCT shall be dependent upon temperature range in use.

NOTE 2 The resulting uncertainty of calibration can be dependent upon the immersion depth.

The DCT probe shall be immersed no less than the immersion depth stated on the calibration certificate.

NOTE 3 With respect to DCT probe immersion depth, a procedure is available in ASTM E563-11:2019, Section 7,[1] for determining the minimum depth. With respect to an ice bath, ASTM E563-22 provides guidance on the preparation of an ice bath, however variance from the specific steps is permitted, provided preparation is consistent as it is being used to track change in calibration.

The DCT probe calibration drift should be verified periodically while in use, and not less than once a year. If the measurement of DCT calibration drift exceeds the specified limit, it shall be fully recalibrated consistent with its range-of-use. If the drift exceeds the noted limit for calibration drift, then it shall be reverified at a shorter time period, and not less than once per month, until this is noted as stable.

NOTE 4 The procedures contained in ASTM E563-22 and ASTM E644-11 provide guidance on the steps required to check calibration drift.

Table 1 — DCT requirements

Criteria	Minimum requirements
DCT	ASTM E2877-12
Display resolution	0,01 °C, recommended 0,001 °C
Display maximum permissible error for combined probe and sensor	Range: −80 °C −0 °C, 0,05 °C Range: 0 °C −100 °C, 0,02 °C Range: >100 °C, 0,05 °C
Sensor type	Resistance temperature detector (RTD), such as a platinum resistance thermometer (PRT) or thermistor
Drift	less than 20 mK (0,02 °C) per year
Linearity	Less than 0,01 °C over range of intended use
Calibration report	The DCT shall have a report of temperature calibration which should be traceable to a national calibration or metrology standards body issued by a calibration laboratory with demonstrated competency in temperature calibration
Calibration data	The calibration report shall include at least 3 calibration temperatures including 0 °C and two other points including the test temperature of use and state the immersion depth under which this was calibrated and the resulting uncertainty. The calibration data should be distributed over the calibration range of the DCT

6.6 Automated apparatus

6.6.1 General

Automated viscometers, which use the technical principles of this document, are acceptable provided they meet the accuracy and precision of all the equipment listed in 6.5. In addition, if they are used to measure viscosity in samples subject to conditioning using the steps in Annex C, a heated sample tray shall be used if the sample is not analysed immediately after conditioning. This sample tray (6.6.2) shall be heated to a temperature which will ensure the sample will not drop below its WAT (wax appearance temperature) or 20 °C above its pourpoint. For samples required to be analysed at 100 °C or above, heating the sample above these temperatures can cause evaporation of light components and is not appropriate.

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[1] Withdrawn.

Flow times of less than 200 s are acceptable, however, the kinetic energy correction shall be calculated and should not exceed 3 % of the measured viscosity. Where a value of greater than 3 % is achieved, the analysis should be repeated using a smaller diameter viscometer tube.

NOTE ISO 3105 describes in more detail the principles and calculation of kinematic viscosity as related to the dimensions of the viscometer.

6.6.2 Sample trays

Some automated equipment contain sample loading trays for analysis of multiple samples. When a sample has been subjected to conditioning using the steps in Annex C, the sample shall not be allowed to cool below the testing temperature on the loading tray as this will result in an increase in measured viscosity as compared to the manual procedure. The sample delivery path should be heated as the temperature of the conditioned sample can drop very quickly. For analysis of these samples, the sample loading trays shall be heated above the test temperature where practical, (see 6.6.1 for samples analysed at 100 °C or above) to ensure the temperature of the sample has reached the test temperature at the time of analysis and the nature of the sample is not changed. For analysis of these samples at 50 °C, a sample tray heated at 54 °C has been shown to be sufficient to maintain the sample temperature above 50 °C for at least 40 min. For analysis of these samples at other temperatures, the lab should establish the correct sample tray temperature and time before analysis.

6.6.3 Temperature measuring device

If embedded, a temperature measuring device shall fully meet the requirements of 6.5.4 and be removable for an external calibration. The embedded device provides an independent reference temperature read-out, allowing the temperature control of the automated apparatus to be adjusted at the required set-point of test.

6.7 Timing device, capable of taking readings with a discrimination of 0,1 s or better, and having an uncertainty within ±0,07 % of the reading when tested over intervals of 200 s and 1 000 s.

Regularly verify these readings and maintain records of such checks.

The time signals which are broadcast by the National Institute of Standards and Technology (NIST), National Physical Laboratory (NPL) or other time signal stations, are a convenient and primary standard reference for calibrating timing devices.

NOTE Many broadcast networks put out a standard frequency signal, as do many telephone networks. Such signals are suitable for checking the timing devices used to an accuracy of 0,1 s.

Timing devices employed in automated viscometers can be an integral part of the apparatus and typically are digital (using a precision crystal oscillator) with precision discriminations of 0,01 s or better. As such, it is possible that the timing devices are not able to be individually verified once installed. Documentation of the accuracy of the timing device over the intended measuring range of the viscometer tube should therefore be provided by the manufacturer. Independent verification of timing devices should be provided in cases where the above-mentioned limits are not satisfied.

Electrical timing devices may be used if the current frequency is controlled to an uncertainty of 0,05 % or better. Alternating currents, as provided by some public power systems, are controlled intermittently rather than continuously. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

7 Verification

7.1 Viscometer

Verify the calibration of the viscometer using a certified viscosity reference standard (5.5) following Procedure A (manual, Clause 11) or Procedure B (automated, Clause 12). Acceptable tolerance bands for this verification check shall be as detailed in Annex D. If the measured kinematic viscosity does not fall within this acceptable range, recheck each step in the procedure, including thermometer and viscometer calibrations and cleaning to locate the source of error. ISO 3105:1994, Table 1 gives details of standards available.

Alternatively, verify working viscometers against a reference viscometer having a certificate of calibration in accordance with ISO 3105.

Verification is required at least prior to first use of the viscometer and whenever a physical change is made to the apparatus e.g. re-calibrating the temperature set-point, after cleaning or investigating the failure of quality assurance (QA)/ quality control (QC) protocol in place.

7.2 Liquid-in-glass thermometer

Verify the calibration of the liquid in a glass thermometer on a periodic basis in line with B.2 at least at the frequencies described, and maintain records of such checks. A complete new recalibration of the thermometer, while permitted, is not necessary in order to meet the accuracy ascribed to the design of the thermometer until the ice point change from the last full calibration amounts to one scale division, such as 0,05 °C. Some thermometers have an ice point scale for this activity.

7.3 Digital contact thermometer

Verify the calibration of the DCT at least annually. The probe shall be recalibrated, when the check value differs by more than 0,02 °C from the last probe calibration.

Verification can be accomplished with the use of a water triple point cell or an ice bath.

ASTM standard practices given in ASTM E563-22, ASTM E1750, and ASTM E2593 may be used as references for checking calibrations.

7.4 Timer

Verify the precision of the timer in use at regular periods and maintain records of such checks.

NOTE Many broadcast networks put out a standard frequency signal, as do many telephone networks. Such signals are suitable for checking the timing devices to a maximum permissible error of 0,1 s.

8 Re-calibration

Glass capillary viscometer recalibration, if required, shall be undertaken using the procedures in ISO 3105.

CAUTION — Users are cautioned that recalibrating equipment in situ when a verification fails potentially calibrates in an error. The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It should be appreciated that a correct result obtained on standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

9 Quality control

Use statistical control charts to validate “in-statistical-control” status for the execution of a standard test method in a single laboratory. For examples, see ISO 4259-4.

Use a quality control (QC) sample that is representative of the product(s) routinely tested by the laboratory to confirm that the instrument is in statistical control.

This may not be possible if the product under test cannot be used as a QC material due to its nature (unstable or subject to thermal treatment).

Where the testing facility lab uses a CRM material as a measure of quality control, Annex D shall be used as the procedure to construct suitable acceptable tolerance zones for the QC/QA system.

10 Sample preparation

10.1 Pre-analysis sample conditioning

10.1.1 Using Table 2, verify whether the sample type under analysis requires conditioning before analysis and, if so, follow the relevant procedure and steps noted, before charging the viscometer.

10.1.2 If the sample is transparent, but not liquid at room temperature, for example samples with a high pour point value, then the sample shall be sufficiently heated to ensure it can flow freely before charging the viscometer.

10.2 Visual inspection and filtering

When the sample is liquid, visually inspect it. If the sample contains fibres or solid particles, use a sample filter (6.2) prior to or during charging of the viscometer tube. At all stages, protect the sample from contamination.

Table 2 — Sample types and conditioning procedures

Sample type	Is sample conditioning required before analysis?	Is sample filter required?	Conditioning procedure reference
Base oils	No	No[a]	Not required
Gas oils	No	No[a]	Not required
Distillate	No	No[a]	Not required
Kerosene	No	No[a]	Not required
Jet fuel	No	No[a]	Not required
Formulated oils	No	No[a]	10.1.2
Lubricant additives	No	No[a]	10.1.2
Biofuel blend	No	No[a]	10.1.2
Biodiesel B100 (transparent)	No	No[a]	10.1.2
Petroleum wax (transparent)	No	No[a]	10.1.2
Biodiesel B100 (opaque)	Yes	Yes	Annex C
Petroleum wax (opaque)	Yes	Yes	Annex C
Residual fuel oils	Yes	Yes	Annex C
Steam-refined cylinder oils	Yes	Yes	Annex C
Black lubricating oils	Yes	Yes	Annex C
Used oils	No	Yes	Not required

[a] Unless it contains fibres, solid particles or is not transparent.

11 Procedure A — Manual equipment (referee test method)

11.1 Procedure A is the referee test method (or reference test method) to resolve doubts or dispute.

Check the bath temperature using the temperature measuring device, ensuring that the set point is within the acceptable tolerance and accuracy of the test temperature, and record this data.

With certain products which exhibit “gel-like” behaviour, users should take care that measurements are made at temperatures sufficiently high for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameter.

11.2 Select suitable clean, dry calibrated viscometer(s) having a range covering the estimated kinematic viscosity at the specified test temperature. The flow time shall not be less than 200 s, or less than the minimum flow time for viscometer types and/or sizes specified in Annex A, and the maximum flow time stated in ISO 3105.

The specific details of operation vary depending on the type of viscometer. The operating instructions for the different types of viscometers listed in Table A.1 are given in ISO 3105. In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table A.1 (type C).

For the measurement of kinematic viscosity of jet fuels at −20 °C, only suspended-level type ISO 3105 viscometers as noted in Table A.1 shall be used. The suspended-level type viscometer types used for jet fuel do not require a correction to the calibration constant for the test temperature being used.

11.3 Charge the viscometer with the sample in accordance with the operating instructions in ISO 3105 for the particular type of viscometer, as described in Annex A. Then place in the bath in the manner dictated by the design of the instrument. This operation shall be in conformity with that employed when the instrument was calibrated.

Allow the charged viscometer to remain in the bath long enough to reach the test temperature, and to ensure that all air bubbles in the sample have dispersed. As the equilibration time in the bath varies for different products, different viscometer tubes, for different temperatures and for different kinematic viscosities, establish and document a safe equilibrium time by trial. A minimum of 30 min is required for jet fuel at −20 °C.

Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

NOTE Some viscometers require charging outside of the bath due to their design.

When the test temperature is below the ambient dew point, the use of loosely packed drying tubes affixed to the open ends of the viscometer is permitted but not mandatory. These are designed to prevent water condensation. However, it is essential that they do not set up a pressure differential and affect the rate of flow. Before first use of drying tubes, it is recommended that a certified viscosity reference standard is used to verify the correct use of the viscometer with and without drying tubes in order to ensure that there is no restriction in the flow.

Special conditions apply when the test temperature is below the dew point, such as for analysis at −20 °C. To ensure moisture does not condense or freeze on the walls of the capillary, it is recommended to charge the viscometer outside the bath and if used affix loosely packed drying tubes to the open ends of the viscometer. The drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by changing the pressure in the instrument. Carefully flush the moist room air from the viscometer by applying vacuum to one of the drying tubes.

Finally, it is recommended before placing the viscometer in the bath to draw up the sample into the working capillary and timing bulb and allow to drain back, as an additional safeguard against moisture condensing or freezing on the walls.

Do not use viscometers which cannot be removed from the constant temperature bath for charging the sample portion.

Where one bath is used to accommodate several viscometers, never add or withdraw a viscometer while any other viscometer is being used to measure flow time.

For samples which have been subjected to conditioning as described in Annex C, ensure that the determinations are started within 1 h of completing this pre-treatment.

11.4 Perform the determinability test.

11.4.1 Use suction (if the sample contains no volatile constituents) or pressure to position the sample level in accordance with the operating instructions as set out in ISO 3105 for the type of viscometer in use. With the sample flowing freely, measure, in seconds, to within 0,1 s the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum, select a viscometer with a capillary of smaller diameter and repeat the operation.

11.4.2 On all fluids except those subject to conditioning in Annex C, repeat the procedure. For direct flow viscometers described in 11.4.1, without recharging the viscometer make a second measurement of flow time. In the case of a reverse flow viscometer, either charge two viscometer tubes, or carry out a complete cleaning and drying of a single tube before re-charging and performing a second measurement of flow time.

11.4.3 Repeat 11.3 and 11.4.1. Record both flow time measurements. The intent is that the same sample of fluid is measured in the same timing bulb successively. In the case of a reverse flow viscometer with two bulbs, only the lower bulb shall be used for flow time measurement.

On fluids subject to conditioning in Annex C, charge two viscometer tubes and follow the procedure described in 11.4.1. Record both measurements.

From the two measurements of flow time, calculate two determined values of kinematic viscosity as shown in 14.1.1.

If the two measurements agree with the stated determinability figure for the product, calculate the average of the two determinations and use this value to report the kinematic viscosity. Record the result.

11.4.4 If two successive measurements do not meet the determinability figure for the product, repeat the above operation from 7.1.

Poor determinability can be attributed to: an unsuccessfully filtered sample, ice forming in the capillary, a contaminated viscometer or sample, unstable temperature conditions, insufficient equilibrium time, heterogeneity of sample or that the viscometer may require cleaning. Consider these points before starting a new analysis.

If the material or temperature (or both) is not listed in Clause 16 then use 0,01 y for temperatures between 15 °C and 100 °C and for temperatures outside this range use 0,015 y as an estimate of determinability, where y is the average of the determinations being compared. In addition, the user can establish a reasonable determinability by trial. Document and use this “trial” determinability value against which to judge if determinability has been met in subsequent analysis.

12 Procedure B — Automated equipment

12.1 Set up the automated viscometer in accordance with the manufacturer’s instructions for use and verify that the equipment is fit for use by applying suitable quality control measures.

12.2 Check the bath temperature using the temperature measuring device, ensuring that the set point is within the acceptable accuracy and tolerance of the test temperature. Do not start the test if the measurement is outside the tolerance.

12.3 Perform the determinability test.

12.3.1 Charge the viscometer, on completion of the equilibrium time, ensure that the sample is drawn up the capillary to a position in the capillary tube above the timing bulb.

The sample may also be drawn up once and released without timing to “wet” the tube if required.

For sub-zero temperature measurements, the sample shall be held in the viscometer tube during the temperature equilibrium period. The time required is dependent upon the sample type, viscometer tube and temperature of analysis, and should be determined individually for each automated equipment. For analysis of jet fuel at −20 °C, the automated viscometer shall maintain the sample in position for a minimum of 20 min for the temperature to equilibrate.

12.3.2 Allow the sample to flow freely and measure in seconds, to a resolution of at least 0,01 s, the time required for the bottom of the meniscus to pass from one timing position to the next timing position.

Flow times of less than 200 s are permitted as long as the kinetic energy correction is calculated and does not exceed 3 % of the measured viscosity. If the kinetic energy correction exceeds 3 %, select a viscometer with a smaller diameter capillary and repeat the test.

12.3.3 On all fluids except those subject to conditioning in Annex C, repeat the procedure described in 12.3.1 to 12.3.2, without recharging the viscometer, to make a second measurement of flow time. The equilibrium time in this case is not required. Record the flow time measurements, kinetic energy correction and the bath temperature readings at the beginning of each flow time measurement.

12.3.4 On fluids subject to conditioning in Annex C, two viscometer tubes can be charged and follow the procedure described in 12.3.1 to 12.3.3, or all measurements can be undertaken in one viscometer tube if the sample is sufficiently fluid, and the sample is not allowed to cool below the test temperature. Record the flow time measurements, kinetic energy correction and the bath temperature readings at the beginning of each flow time measurement. These may be automatically captured by the equipment or manually by the analyst.

12.3.5 Repeat the procedure described in 12.3.1 to 12.3.2 until two successive determinations agree within the required determinability given in this document. If the two determinations agree with the stated determinability figure for the product, calculate the average of the two measurements and use this value to report the kinematic viscosity. Record the result.

12.3.6 If two successive determinations do not meet the determinability figure for the product, repeat the above operation from 12.3.1.

If the material or temperature or both is not listed in Clause 16 then use 0,01 y for temperatures between 15 °C and 100 °C and for temperatures outside this range use 0,015 y as an estimate of determinability (where y is is the average of the determinations being compared). In addition, the user can establish a reasonable determinability by trial. Document and use this “trial” determinability value against which to judge if determinability has been met in subsequent analysis.

13 Cleaning of the viscometer tube

13.1 After each analysis, clean the viscometer thoroughly by several rinses with the sample solvent (5.2), followed by the drying solvent (5.3). Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

13.2 When the verification of the viscometer calibration using certified reference material is outside of the acceptable tolerance band, the viscometer should be cleaned using the manufacturer's recommended cleaning solution and methodology to remove organic deposits.

Sometimes insoluble particles can be removed by use of an ultrasonic bath. Inorganic deposits may be removed using acid treatment before the use of cleaning solution, particularly if the presence of barium salts is suspected. Following the removal of inorganic deposits, clean with the cleaning solution (5.1) to remove residual traces of organic deposits, rinse thoroughly with water (5.4) and drying solvent (5.3), and dry with filtered dry air or a vacuum line as required by the manufacturer of the equipment.

14 Calculation

14.1 Procedure A — Manual viscometers

14.1.1 Calculate the determinability value, d, from two measured flow times, t^1 and t^2, and the viscometer constant, C, by means of Formulae (2) and (3):

$$\nu^1 = C \times t^1 \tag{2}$$

$$\nu^2 = C \times t^2 \tag{3}$$

where

ν^1 is the kinematic viscosity, in millimetres squared per second for the first flow time;

ν^2 is the kinematic viscosity, in millimetres squared per second for the second flow time;

C is the calibration constant of the viscometer, in millimetres squared per second squared;

t^1 is the measured flow time, in seconds of the first analysis;

t^2 is the measured flow time, in seconds of the second analysis.

Calculate the difference between ν^1 and ν^2 and compare to the determinability value stated in Table 3. If the difference between ν^1 and ν^2 is less than the determinability value stated in Table 3, calculate the final reported kinematic viscosity result ν as an average ν^1 and ν^2 as given by Formula (4):

$$\nu = (\nu^1 + \nu^2)/2 \tag{4}$$

where ν is the kinematic viscosity, in millimetres squared per second of the final reported result.

14.1.2 If required, calculate the dynamic viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of Formula (5):

$$\eta = \nu \times \rho \times 10^{-3} \tag{5}$$

where

η is the dynamic viscosity, in millipascal seconds;

ρ is the density, in kilograms per cubic metre, at the same temperature used for the determination of the kinematic viscosity.

The density of the sample may be determined by an appropriate method such as ISO 12185 ·or ISO 3675 and corrected to the temperature of determination by means of ISO 91:2017.

14.2 Procedure B — Automated viscometers

14.2.1 The equipment should calculate the determinability values as described in 14.1.1. The determinability values should be recorded and available for reference. If the difference between v^1 and v^2 is less than the determinability value stated in Table 4, the equipment should calculate the kinematic viscosity value for the sample. If it is greater than the value stated in Table 4, then the equipment should indicate that the result should not be reported.

14.2.2 Calculate the kinematic viscosity, *v,* from the average of the measured flow time, *t*, and the viscometer constant, *C*, taking into account the kinetic energy factor *E*, by means of Formula (6):

$$v = C \times t - (E / t^2) \tag{6}$$

where

v is the kinematic viscosity, in millimetres squared per second;

C is the calibration constant of the viscometer, in square millimetres per second squared;

t is the mean flow time, in seconds;

E is the kinetic energy factor, in square millimetre seconds.

Although the kinetic energy factor, *E*, is not a constant, it can be approximated by means of Formula (7):

$$E = \frac{52{,}5V^{\frac{3}{2}}}{L(C \times d)^{\frac{1}{2}}} \tag{7}$$

where

V is the volume of the timing bulb, in millilitres;

L capillary working length, in millimetres;

d capillary diameter, in millimetres;

C is the calibration constant of the viscometer, in square millimetres per second squared.

15 Expression of results

Report the test results for kinematic viscosity or dynamic viscosity, or both, to four significant figures, together with test temperature.

16 Precision

16.1 Determinability, *d*

The difference between successive determinations obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, in the normal and correct operation of this test method, would exceed in the long run the values indicated in Table 3 with an approximate probability of 5 % due to random variation.

16.2 Repeatability, *r*

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method, would exceeed the values calculated in Table 3 with an approximate probability of 5 % due to random variation.

16.3 Reproducibility, *R*

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the method, would exceed the values calculated in Table 3 with an approximate probability of 5 % due to random variation.

16.4 Procedure A — Manual viscometers

Precision estimates are summarized in Table 3.

16.5 Procedure B — Automated viscometers

Precision estimates are summarized in Table 4.

Table 3 — Precision estimations Procedure A

Type of product	Determinability d mm²/s	Repeatability r mm²/s	Reproducibility R mm²/s
Base oils at 40 °C [15]	0,003 7 y	0,010 1 X	0,013 6 X
Base oils at 100 °C [15]	0,003 6 y	0,008 5 X	0,019 0 X
Formulated oils at 40 °C [15]	0,003 7 y	0,007 4 X	0,012 2 X
Formulated oils at 100 °C [15]	0,003 6 y	0,008 4 X	0,013 8 X
Formulated oils at 150 °C [16]	0,015 y	0,005 6 X	0,018 X
Petroleum wax at 100 °C [17]	0,008 0 y	0,014 1 $X^{1,2}$	0,036 6 $X^{1,2}$
Residual fuel oils at 100 °C [18]	0,03 y	0,080 88 X	0,120 6 X
Residual fuel oils at 50 °C [18]	0,024 4 y	0,078 85 X	0,084 61 X
Lubricant additives at 100 °C [19]	0,001 06 $y^{1,1}$	0,001 92 $X^{1,1}$	0,008 62 $X^{1,1}$
Gas oils at 40 °C [20]	0,001 3 $(y + 1)$	0,004 3 $(X + 1)$	0,008 2 $(X+1)$
Jet fuel at −20 °C [21]	0,007 608	0,001 368 $X^{1,4}$	0,002 899 $X^{1,4}$
Marine fuels, diesel, biofuel and biofuel blends at 40 °C [22]	0,008 093	0,004 094 (x + 1,5)	0,006 133 (x + 1,5)

Key

y average of determinations being compared

X average of results being compared

NOTE 1 The precision values refer to kinematic viscosity only, not dynamic viscosity.

NOTE 2 See references [15] to [22] for further details on how the precision values were obtained.

NOTE 3 The precision values for used (in-service) formulated oils at 40 °C and 100 °C using Procedure A has not been determined as most laboratories use automated equipment.

Table 4 — Precision estimations Procedure B

Type of product	Determinability d mm^2/s	Repeatability r mm^2/s	Reproducibility R mm^2/s
Used (in-service) formulated oils at 40 °C [23]	[a]	$0{,}000\,233\ X^{1{,}722}$	$0{,}000\,594\ X^{1{,}722}$
Used (in-service) formulated oils at 100 °C [23]	[a]	$0{,}001\,005\ X^{1{,}463\,3}$	$0{,}003\,361\ X^{1{,}463\,3}$
Marine fuels, diesel, biofuel and biofuel blends at 40 °C [22]	0,004 667	0,010 07	0,020 92

Key

X average of results being compared

NOTE 1 The precision values refer to kinematic viscosity only, not dynamic viscosity.

NOTE 2 The precision values for automated viscometers has been determined for base oils at 40 °C and 100 °C and formulated oils at 40 °C and 100 °C. These were shown to be no worse than the precision for manual instruments, and the results between manual and automated instruments were considered practically equivalent for these sample types[23].

NOTE 3 See references [22] and [23] for further details on how the precision values were obtained.

[a] The determinability for used (in-service) formulated oils has not been determined, however a limit of 0,01 y where y is the average of the determinations being compared for temperatures between 15 °C and 100 °C may be used. Alternatively, users may reference DIN 51659-1 as a guide.

17 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 3104:2023;

c) the procedure used i.e. Procedure A (as specified in Clause 11) or Procedure B (as specified in Clause 12);

d) the result of the test (see Clause 15);

e) any deviation, by agreement or otherwise, from the procedure specified;

f) any unusual features observed;

g) the date of the test;

h) the name and address of the test laboratory (if relevant to the customer).

Annex A
(normative)

Viscometer types, calibration and verification

Table A.1 lists capillary viscometers commonly in use for viscosity determinations on petroleum products. For specifications and operating instructions, see ISO 3105.

Table A.1 — Viscometer types

Type	Viscometer identification	Kinematic viscosity range[a] mm²/s
A	**Ostwald types for transparent liquids** Cannon-Fenske routine[b]	0,5 to 20 000
	Zeitfuchs	0,6 to 3 000
	BS/U-tube[b]	0,9 to 10 000
	BS/U/M miniature	0,2 to 100
	SIL[b]	0,6 to 10 000
	Cannon-Manning semi-micro	0,4 to 20 000
	Pinkevitch[b]	0,6 to 17 000
B	**Suspended-level types for transparent liquids** BS/IP/SL[b]	3,5 to 100 000
	BS/IP/SL (S)[b]	1,05 to 10 000
	BS/IP/MSL	0,6 to 3 000
	Ubbelohde[b]	0,3 to 100 000
	FitzSimons	0,6 to 1 200
	Atlantic[b]	0,75 to 5 000
	Cannon-Ubbelohde (A), Cannon-Ubbelohde dilution (B)[b]	0,5 to 100 000
	Cannon-Ubbelohde semi-micro	0,4 to 20 000
	DIN Ubbelohde	0,35 to 50 000
C	**Reverse-flow types for transparent and opaque liquids** Cannon-Fenske opaque	0,4 to 20 000
	Zeitfuchs cross-arm	0,6 to 100 000
	BS/IP/RF U-tube reverse-flow	0,6 to 300 000
	Lantz-Zeitfuchs type reverse-flow	60 to 100 000

[a] Each range quoted requires a series of viscometers. To avoid the necessity for a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s, except where noted in ISO 3105.

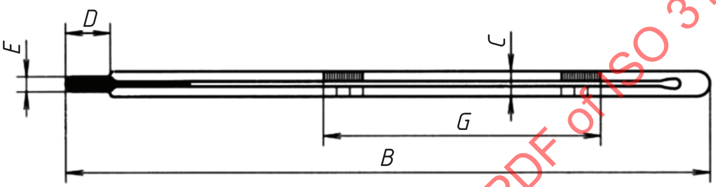
[b] In this series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

Annex B
(normative)

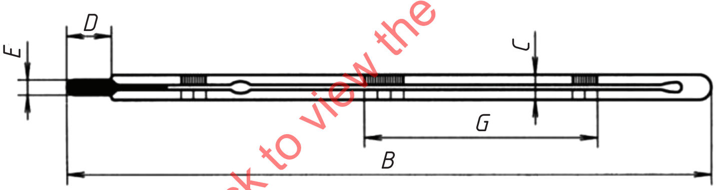
Thermometers for kinematic viscosity test

B.1 Thermometer designs and specifications

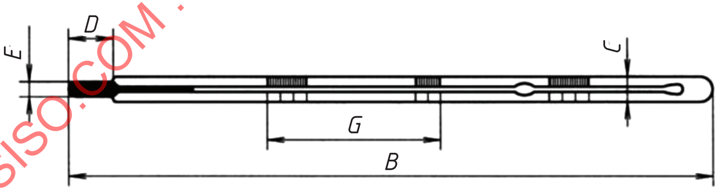
Use a short-range specialized thermometer conforming to the generic specification given in Table B.1 and to one of the designs shown in Figure B.1 a) to c). Table B.2 gives a range of ASTM, IP and ASTM/IP thermometers that comply with the specification in Table B.1, together with their designated test temperatures. As an alternative, use a DCT as defined in 6.5.4.



a) Design 1



b) Design 2



c) Design 3

Key

B	total length	*C*	stem outside diameter
D	bulb length	*E*	bulb outside diameter
G	length of scale range		

NOTE The difference in the designs rests mainly in the position of the ice point scale. In design 1, the ice point is within the scale range, in design 2, the ice point is below the scale range and in design 3, the ice point is above the scale range.

Figure B.1 — Thermometer designs

Table B.1 — General specification for thermometers

Immersion depth	Unit	Total
Scale marks:		
— Subdivisions	°C	0,05
— Long lines at each	°C	0,1 and 0,5
— Numbers at each	°C	1
— Maximum line width	mm	0,1
Scale error at test temperature, max.	°C	0,1
Expansion chamber to permit heating to	°C	105 for thermometer scale up to 90, 120 between 90 and 95, 130 between 95 and 105, 170 above 105
Total length, *B*	mm	300 to 310
Stem outside diameter, *C*	mm	6,0 to 8,0
Bulb length, *D*	mm	45 to 55
Bulb outside diameter, *E*	mm	no greater than stem
Length of scale range, *G*	mm	40 to 90

Table B.2 — Examples of complying thermometers

Thermometer number	Test temperature °C
ASTM 132C/IP 102C	150
ASTM 110C/IP 93C	135
ASTM 121C/IP 32C	98,9 and 100
ASTM 129C/IP 36C	93,3
ASTM 48C/IP 90C	82,2
IP 100C	80
ASTM 47C/IP 35C	60
ASTM 29C/IP 34C	54,4
ASTM 46C/IP 66C	50
ASTM 120C/IP 92C	40
ASTM 28C/IP 31C	37,8
ASTM 118C	30
ASTM 45C/IP 30C	25
ASTM 44C/IP 29C	20
ASTM 128C/IP 33C	0
ASTM 72C/IP 67C	−17,8
ASTM 127C/IP 99C	−20
ASTM 126C/IP 71C	−26,1
ASTM 73C/IP 68C	−40
ASTM 74C/IP 69C	−53,9

B.2 Thermometer calibration and verification

B.2.1 Report of temperature calibration

When using a liquid-in-glass thermometer, it shall have a report of temperature calibration traceable to a national calibration or metrology standards body, issued by a competent calibration laboratory with demonstrated competency in temperature calibration. The calibration report shall include data for a series of temperatures which are appropriate for its intended use.

B.2.2 Ice point verifications

The scale correction of liquid-in-glass thermometers can change during storage and use. Therefore, regular in-house ice point verifications are required, and can be achieved within the working laboratory, using an ice melting bath. However, these checks may not be an adequate means of recalibration as the use of a single-point recalibration at the ice point adds an additional uncertainty to the updated thermometer calibration result at every temperature except for the ice point. The user shall determine if the thermometer requires complete re-calibration to continue to meet the expanded measurement uncertainty requirements of this clause.

NOTE NIST Special Publication 1 088, 2009, Section 7.9[14] on determining the uncertainty of correction can be helpful to the user in making this decision. If in-house ice point verification brings the expanded measurement uncertainty out of the requirements of B.2.1, a complete recalibration at a laboratory meeting the requirements of B.2.1 is required.

B.2.3 Liquid-in-glass thermometer checks

For liquid-in-glass thermometers, the interval for ice-point verification shall be no longer than six months. For new thermometers, monthly checking for the first six months is recommended. A change of one or more scale divisions in the ice point means that the thermometer may have been overheated or damaged, and it may be out of calibration. Such thermometers shall be removed from service until inspected, or recalibrated, or both.

B.2.4 Records of calibration

Keep records of all recalibrations.

B.2.5 Procedure for ice-point verification of liquid-in-glass thermometers

B.2.5.1 Unless otherwise listed on the certificate of calibration, the recalibration of calibrated kinematic viscosity thermometers requires that the ice-point reading shall be taken within 60 min after being at test temperature for not less than 3 min.

NOTE NIST Special Publication 1 088 from the NIST website[14] describes an effective procedure for ice point verification, including all formulae necessary for calculations of change of correction and measurement uncertainty.

B.2.5.2 Select clear pieces of ice, preferably made from distilled or pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the crushed ice and add sufficient water to form slush, but not enough to float the ice. As the ice melts, drain off some of the water and add more crushed ice. Insert the thermometer, and pack the ice gently about the stem, to a depth approximately one scale division below the 0 °C graduation.

B.2.5.3 After at least 3 min have elapsed, tap the thermometer gently and repeatedly at right angles to its axis while making observations. Successive readings taken at least 1 min apart shall agree within 0,005 °C. Record the ice-point readings and determine the thermometer correction at this temperature from the mean reading. If the correction is found to be higher or lower than that corresponding to a previous calibration, change the correction at all other temperatures by the same value. During the procedure, apply the following conditions.

a) The thermometer shall be supported vertically.

b) View the thermometer with an optical aid that gives a magnification of approximately five and also eliminates parallax.