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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

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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

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

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 [24].

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, 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
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.

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