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ISO/FDIS 23581

Petroleum products and related products — Determination of kinematic viscosity — Method by Stabinger type viscometer

Produits pétroliers et produits connexes - Détermination de la viscosité cinématique - Méthode par viscosimètre type Stabinger

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO 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 second edition cancels and replaces the first edition (ISO 23581:2020), which has been technically revised. [://standards.iteh.ai/catalog/standards/iso/2257a218-12c0-4197-b85f-61ddb35db936/iso-fdis-23581](http://standards.iteh.ai/catalog/standards/iso/2257a218-12c0-4197-b85f-61ddb35db936/iso-fdis-23581)

The main changes are as follows:

- base oils, formulated oils, jet fuels and residual fuel oils have been included in the scope;
- the apparatus description, sample handling procedures and determinability criteria have been updated to accommodate the new scope.

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 products and related products — Determination of kinematic viscosity — Method by Stabinger type viscometer

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 users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of this document and fulfil other applicable requirements for this purpose.

1 Scope

This document specifies a procedure for the determination of kinematic viscosity, ν , by calculation from dynamic viscosity, η , and density, ρ , of both transparent and opaque liquid petroleum products and crude oils using the Stabinger type viscometer.

The result obtained using the procedure described in this document depends on the rheological behaviour of the sample. This document is predominantly applicable to liquids whose shear stress and shear rate are proportional (Newtonian flow behaviour). If the viscosity changes significantly with the shear rate, comparison with other measuring methods is not possible except at similar shear rates.

The precision has been determined only for the materials, density ranges and temperatures described in [Clause 13](#). The test method can be applied to a wider range of viscosity, density, temperature and materials. It is possible that the precision and bias are applicable for materials which are not listed in [Clause 13](#).

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 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 12185, *Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method*

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

dynamic viscosity

η

ratio of the applied shear stress to the resulting shear rate of a liquid

3.2

kinematic viscosity

ν
ratio of the *dynamic viscosity* (3.1) to the *density* (3.3) of a liquid at the same temperature and pressure

Note 1 to entry: The kinematic viscosity is a measure of a liquid's resistance to flow under gravity.

3.3

density

ρ
mass of a substance divided by its volume at a given temperature

3.4

determinability

d
quantitative measure of the variability associated with the same operator in a given laboratory obtaining successive determined values using the same apparatus for a series of operations leading to a single result

Note 1 to entry: Determinability is the difference between two such single determined values that would be exceeded about 5 % of the time (one case in 20 in the long run) in the normal and correct operation of the test method.

3.5

test specimen

portion or volume of the sample obtained from the laboratory sample, which is delivered to the measuring cells

4 Principle

A test specimen is introduced into the measuring cells, at a controlled and known temperature. The measuring cells consist of a pair of rotating concentric cylinders and an oscillating U-tube. The dynamic viscosity is determined from the equilibrium rotational speed of the inner cylinder under the influence of the shear stress of the test specimen and an eddy current brake in conjunction with adjustment data. The density is determined by the oscillation frequency of the U-tube in conjunction with adjustment data. The kinematic viscosity is calculated by dividing the dynamic viscosity by the density.

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5 Reagents and materials

5.1 Cleaning solvent, able to remove the sample from the measuring cell after the measurement and completely miscible with all constituents of the sample. Commercially available volatile petroleum spirit or cleaner's naphtha of technical grade or better have been proven suitable as cleaning solvents.

5.2 Drying solvent, highly volatile and miscible with the cleaning solvent, shall be filtered before use and shall be of an appropriate purity so that no residues remain in the instrument. *n*-Hexane, *n*-heptane (recommended due to lower toxicity) or, depending on the sample, concentrated ethanol ($\geq 96\%$) are suitable.

NOTE 1 A separate drying solvent is not needed if the cleaning solvent also meets the requirements of the drying solvent.

NOTE 2 When measuring residual fuel, asphaltic material can be removed by pre-washing with an aromatic solvent (e.g. toluene or xylene).

5.3 Compressed air, oil-free and filtered with a dew point lower than the lowest measuring cell temperature at which the instrument should be dried.

The pressure should be limited to 100 kPa.

It is also possible to use inert gases, for example technical nitrogen. The requirements given for compressed air are also valid here.

5.4 Certified reference liquids, for kinematic viscosity and density, which shall be identical to the reference standards for kinematic viscosity and density cited in ISO 3104 and ISO 12185, respectively.

5.5 Reference thermometer and probe, for verification of the temperature calibration.

The measuring uncertainty of the reference thermometer, including the probe, shall not exceed 0,01 °C. The resolution shall be at least 0,001 °C.

The probe used for the calibration (with an adapter if necessary) shall have a shape which fits the geometry of the viscosity cell. The probe replaces the measuring system (tube and measuring rotor).

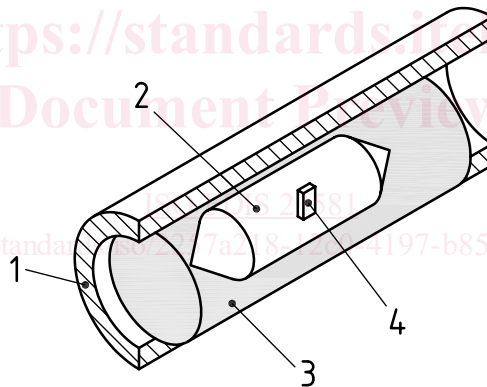
6 Apparatus

Usual laboratory apparatus and glassware shall be used, in particular the following.

6.1 Stabinger type viscometer.

6.1.1 Viscosity measurement

The Stabinger type viscometer is a concentric rotating viscometer, containing an outer rotor and an inner rotor (see [Figure 1](#)). The small concentric gap between these rotors is filled with the sample. The outer rotor is driven at constant speed, which makes the inner rotor rotate due to the sample's viscosity. The lightweight inner rotor is centred in the heavier sample due to the centrifugal forces. The equilibrated speed ratio depends on the driving viscous shear force and the opposing magnetic induction force (eddy current). The dynamic viscosity is a function of the equilibrated speed ratio and adjustment constants. The kinematic viscosity is obtained by dividing the measured dynamic viscosity by the measured density.



Key

- | | | | |
|---|------------------------------|---|--------------|
| 1 | outer rotor (constant speed) | 3 | sample fluid |
| 2 | inner rotor (measured speed) | 4 | magnet |

Figure 1 — Viscosity cell

6.1.2 Density measurement

The Stabinger type viscometer has an integrated density measurement based on the oscillating U-tube principle. The sample-filled U-tube is oscillated and the instrument calculates the density from the measured natural frequency of the filled tube using adjustment factors. The viscosity-dependent error of this procedure is corrected using the measured viscosity value.

6.1.3 Temperature control

The Stabinger type viscometer has an integrated temperature control which keeps the viscosity and density measurement at the same temperature.

Using Peltier elements, a highly conductive measuring cell block which surrounds the measuring cells is set to the target temperature with a stability of $\pm 0,005$ °C over the whole temperature range at the position of the viscosity cell.

The measurement uncertainty of the temperature calibration ($k = 2$; 95 % confidence level) shall be within $\pm 0,03$ °C over the range from 15 °C to 100 °C and within $\pm 0,05$ °C outside this range.

6.2 Syringes.

Commercially available syringes with a Luer tip of at least 5 ml in volume shall be used. The user shall ensure full chemical compatibility of the syringe construction materials with all sample and cleaning liquids by consulting the manufacturer's documentation.

6.3 Flow-through or pressure adapter.

A flow-through or a pressure adapter may be used as an alternative to a syringe for introduction of the test specimen into the measuring cells. Such adapters apply either pressure or suction to the test specimen, therefore, care shall be taken to avoid the formation of bubbles. The user shall ensure full chemical compatibility of the flow-through or pressure adapter construction materials with all sample and cleaning liquids by consulting the manufacturer's documentation.

6.4 Hot filling adapter.

A hot filling adapter maintains the sample temperature during manual introduction of the test specimen into the measuring cells. It may be added to the Stabinger type viscometer to improve the flow of the test specimen and prevent precipitation of wax crystals while filling and cleaning the measuring cells.

6.5 Sample changer or sample handler.

The sample changer or sample handler shall be designed to ensure and maintain the integrity of the test specimen before and during the analysis. It shall be capable of transferring a representative aliquot of the test specimen into the measuring cells. It shall be able to mimic the procedure for sample handling described in 7.2. The sample changer or sample handler may have heating capability, if required.

6.6 Screen.

If a screen is used, it shall have an aperture of 75 μm to remove particles from the sample.

6.7 Magnet.

If an external magnet is used, the magnet shall be strong enough to remove ferromagnetic materials from the sample. Magnetic stirring rods may be used.

6.8 Ultrasonic bath, unheated.

To dissipate and remove air or gas bubbles from the sample, an ultrasonic bath may be used prior to analysis. The bath should be sized to hold the container(s) placed inside, and operate at an operating frequency between 25 kHz and 60 kHz and a typical power output of ≤ 100 W. Ultrasonic baths with operating frequencies and power outputs outside this range may be used, but it is in the responsibility of the user to confirm via a data comparison study that the results obtained after sample preparation with and without such baths do not show significant deviations.

7 Sampling and sample handling

7.1 Sampling

Samples shall be taken as described in ISO 3170 or ISO 3171.

NOTE National regulations can apply.

7.2 Sample handling

7.2.1 General sample handling

For samples such as viscous lube oils that are likely to contain air or gas bubbles, an ultrasonic bath (6.8) without the heater turned on (if so equipped) may be used for typically 1 min to dissipate the bubbles.

The test specimen shall be obtained as follows:

- a) The laboratory sample shall be mixed if it is not homogeneous. The mixing should be carried out in a closed, pressurized container or at sub-ambient temperatures to avoid the loss of volatile material.
- b) The test specimen may be drawn with an appropriate syringe from a properly mixed laboratory sample. Alternatively, the sample may be delivered from the mixing container directly to the measuring cells by means of a flow-through or pressure adapter or sample changer or sample handler, provided that the proper attachments and connecting tubes are used. For waxy or other samples with high pour point, before drawing the test specimen, heat the sample to the desired temperature, which shall be high enough to dissolve the wax crystals.
- c) Samples that are prone to contain particles, such as used oils or crude oils, shall be passed through a 75 µm screen prior to measurement. If these particles are ferromagnetic in nature, a magnet (6.7) should be used instead.

7.2.2 Conditioning of residual fuel oils

7.2.2.1 Place the required number of disposable syringes or sample vials for batch analysis in a sample preheat apparatus (e.g. oven, heating block or bath) held between 60 °C and 65 °C. When manually filling the measuring cells, the Stabinger type viscometer shall be equipped with a hot filling adapter and the injection adapters shall be pre-warmed together with the syringes.

7.2.2.2 Heat the sample in its tightly closed original container in the sample preheat apparatus, held between 60 °C and 65 °C, for 1 h. After heating, the sample shall be sufficiently fluid for easy shaking and stirring.

7.2.2.3 For samples of a very waxy nature or oils of high kinematic viscosity, it may be necessary to increase the heating temperature above 60 °C to achieve proper mixing. The sample should be sufficiently fluid after heating for ease of stirring and shaking.

7.2.2.4 Thoroughly stir the sample with a suitable rod of sufficient length to reach the bottom of the container. Continue stirring until the sample is entirely homogeneous and there is no sludge or wax adhering to the rod.

7.2.2.5 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

7.2.2.6 Immediately thereafter, pour a sufficient amount of the sample into a glass flask to fill the apparatus' sampler system and loosely stopper the flask.

7.2.2.7 Immerse the flask in a heated liquid bath or another equipment which will maintain the sample between 100 °C and 105 °C for 30 min.

Care shall be taken at this step, as vigorous boil-over can occur when opaque liquids which contain high levels of water are heated to high temperatures. Appropriate personal protective equipment should be worn when handling hot materials.

7.2.2.8 Remove the flask from the bath (or other equipment), stopper tightly and shake for 1 min.

7.2.2.9 If the sample is manually introduced into the viscometer, fill the sample into a preheated syringe by using a preheated injection adapter and measure immediately.

7.2.2.10 If a heated sample changer is used, set the vial magazine temperature to be held between 60 °C and 80 °C. Load each sample into a preheated sample vial and insert the vials into the vial magazine. Wait for 10 min to 15 min before starting the measurement.

7.2.2.11 If a heated sample handler is used, load the preheated sample vial or syringe (depending on the model) with the first sample to be tested in the batch and place the sample vial or syringe in the sample handler that is held between 60 °C and 80 °C. Set the temperature of the sample preheat apparatus to between 60 °C and 80 °C and place the other containers in it.

7.2.2.12 Analysis of samples shall be completed within 1 h from completion of the step specified in [7.2.2.8](#).

8 Calibration and verification

8.1 General

The calibration shall be verified periodically using certified reference liquids ([5.4](#)).

Due to the measuring range of the viscosity and temperature, more than one certified reference liquid can be required. If a reference liquid gives no reference value or if the given reference value is not sufficiently precise for one of the two parameters (viscosity or density), (e.g. a density standard without viscosity values), the affected parameter shall be verified with another suitable reference liquid.

Verify the calibration of the temperature measurement periodically by using a reference thermometer ([5.5](#)).

The recommended interval to verify viscosity and density calibration is once a month; for temperature control, once a year.

8.2 Instrument

Ensure that the instrument is leak tight and the measuring cells have been cleaned and dried before verification of the calibration is undertaken.

The verification of the calibration (calibration check measurement) should be carried out according to the instrument manufacturer's instructions.

Calculate an acceptable verification limit in accordance with [Annex A](#). If, despite the correct condition of the instrument, the measured viscosity or density does not lie within the acceptable verification limit 19 out of 20 times, then the viscosity or density cell or both shall be adjusted according to the instrument manufacturer's instructions. Perform a calibration check measurement for verification after the adjustment.

NOTE Measurement values of viscosity and density that do not correspond to the certified values within the limits given are typically attributable to deposits in the measuring cells which are not removed by a routine flushing procedure. Refer to [10.2.7](#) and the manufacturer's instructions for more thorough cleaning methods.