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

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Foreword

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

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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).

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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 (v) at 40 °C in the range from 2 mm^2/s to 6 mm^2/s by calculation from dynamic viscosity (η) and density (ρ) of middle distillate fuels, fatty acid methyl ester fuels (FAME) and mixtures of these 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). However, if the viscosity changes significantly with the shear rate, comparison with other measuring methods is only permissible at similar shear rates.

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 terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
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, defined as the difference between two such single determined values

4   Principle

A test portion of a sample is introduced into the measuring cells, which are at closely 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.

5   Reagents and materials

5.1   Cleaning solvent, able to remove the sample from the measuring cell after the measurement and be completely miscible with all constituents of the sample.

5.2   Drying solvent, highly volatile and miscible with the cleaning solvent, shall be filtered before use and of an appropriate purity so that it does not leave any residues in the instrument.

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

NOTE 2   Commercially available volatile petroleum spirit or cleaner's naphtha of technical grade or better has proven suitable.

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

The pressure should be limited to 100 kPa.

Instead of compressed air, 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 viscosity and density, identical to the reference standards referenced 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

6.1   General

Usual laboratory apparatus and glassware should be used.

6.2   Stabinger type viscometer

6.2.1   Viscosity measurement

The Stabinger type viscometer, a concentric rotating viscometer, contains 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.

6.2.2   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.
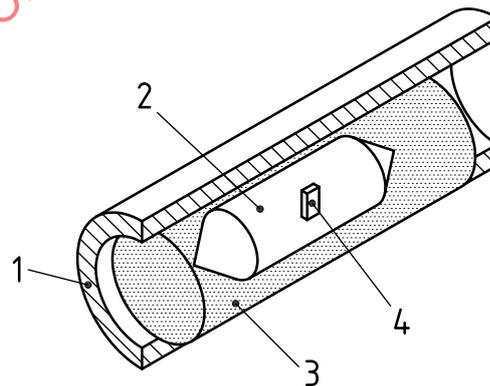
Key

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

Figure 1 — Viscosity cell

6.2.3 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 ± 0,005 °C.

The measurement uncertainty of the temperature sensor shall be within ± 0,03 °C at 40 °C.

6.2.4 Stability

The instrument automatically ensures the temperature equilibration of the sample by checking the stability of the continuously recorded viscosity and density values by limiting the maximum fluctuation range to 0,07 % for the dynamic viscosity and ± 0,03 kg/m^3 for the density within 60 s.

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

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.

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 calibration fluid may 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), for example 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 should be carried out according to the instrument manufacturer's instructions.

If, despite the correct condition of the instrument, the measured viscosity does not correspond to the certified value with a deviation of less than 0,35 %, then the viscosity measurement shall be adjusted according to the instrument manufacturer's instructions.

If, despite the correct condition of the instrument, the measured density does not correspond to the certified value with a deviation of less than 0,001 g/cm³, then the density measurement shall be adjusted according to the instrument manufacturer's instructions.

If, despite the correct condition of the instrument, the measured temperature does not correspond to the certified value with a deviation of less than 0,03 °C, then the temperature measurement shall be adjusted according to the instrument manufacturer's instructions.

9 Apparatus preparation

Ensure that the measuring cells are clean and dry before filling with the sample. The displayed density value of air can be used as an indicator.

Set the test temperature to 40,00 °C. The Stabinger type viscometer automatically ensures temperature control and temperature equilibration of the sample.

If the test temperature is below the dew point of the ambient air, a suitable air-drying apparatus shall be connected to the air pump inlet of the instrument. When using external compressed air or inert gases, ensure that the dew point is lower than the lowest test temperature which can be expected.

Set the determinability limits for viscosity and density to ± 0,1 % and ± 0,2 kg/m³, respectively.

10 Procedure

10.1 Filling and cleaning

There are three different filling and cleaning procedures which may be used; details are given in 10.2, 10.3 and 10.4.

10.2 Manual filling and cleaning using syringes

10.2.1 Load a sufficient amount of sample to a syringe and remove any air bubbles. Ensure that for the first filling enough sample is used to fill all measuring cells. If sufficient sample is available, it is recommended to fill until the sample is visible in the drain hose. Leave the syringe connected to the instrument during the whole measurement procedure. Typically, a 5 ml syringe entirely filled with sample is enough for a measurement with the viscometer.

10.2.2 Switch on the motor for a short time (5 s to 10 s) to ensure that the measuring cells are pre-wetted. In this way any residues are also absorbed into the sample. By subsequently filling at least a further 0,25 ml of sample, the sample in the measuring cell is replaced by fresh sample. If there is enough sample available, it is recommended to refill 1 ml.

10.2.3 Start the motor again for the first determination of viscosity and density. The instrument automatically and continuously checks the stability of the measured values until the set criteria are met (6.2.4). Then the instrument requests to refill sample for the next determination. The procedure is the same as for pre-wetting the cells (10.2.2).

10.2.4 If the difference between the determinations is within the set determinability limits (Clause 9), then the values of the last determination shall be reported as valid measured results.

10.2.5 If it is not possible to obtain a valid result within an applicable number of repetitions, report the robust mean value and the standard deviation (*k* = 2; 95 % confidence level) together with a remark indicating that the determinability exceeded the limits stated in Clause 9 for this product.

10.2.6 Remove the sample from the measuring cells and clean the cells with a suitable cleaning solvent (5.1). Repeat this procedure until the sample is completely removed. Starting the motor for a short period of time and reading the viscosity of the solvent gives an indication of the quality of the cleaning. If the cleaning solvent is not volatile enough, rinse the cells with a suitable drying solvent (5.2) and dry the cells with compressed air (5.3) or by suction. The displayed density value can be used as an indication of the drying.

10.3 Manual filling using sample displacement

10.3.1 The sample volume shall be sufficient to ensure complete displacement of the previous sample. Load the sample to the syringe (typically 25 ml) and remove any air bubbles.

10.3.2 The first filling should be approx. 15 ml. Leave the syringe connected to the instrument during the whole measurement procedure.

10.3.3 Switching on the motor for a short time (5 s to 10 s) ensures that the measuring cells are pre-wetted. In this way, any residues are also absorbed into the sample. By subsequently filling a further 2 ml of sample, the sample in the measuring cell is replaced by fresh sample.

10.3.4 After a new start of the motor, the first determination of viscosity and density is carried out. The instrument automatically and continuously checks the stability of the measured values until the set criteria are met (6.2.4). Then the instrument requests to refill the sample for the next determination.

10.3.5 If the difference between the determinations corresponds with the set determinability limits (Clause 9), then the values of the last determination are given as valid measured result.

10.3.6 If it is not possible to obtain a valid result within an applicable number of repetitions, report the robust mean value and the standard deviation ($k = 2$; 95 % confidence level) together with a remark indicating that the determinability exceeded the limits stated in Clause 9 for this product.

10.3.7 Clean the viscosity measuring cell if necessary (e.g. significant product change, instrument not in use, problems with the determinability limits) according to 10.2.6.

10.4 Automatic filling and cleaning by a sample changer

10.4.1 Before starting the measuring procedure, set appropriate filling, cleaning and drying parameters unless the instrument can adapt these automatically to the sample.

10.4.2 Fill sufficient sample volume into a suitable container for the sample changer and place the container into the magazine. If the sample changer or the sample requires it, close the containers with appropriate covers.

10.4.3 Before starting the measuring cycle, make sure there is sufficient cleaning solvent (5.1) and drying solvent (5.2) and enough space in the waste vessel for gathering the sample and solvent.

10.4.4 After starting the measuring cycle, the measuring cells are automatically filled and pre-wetted and the first determination of viscosity and density is carried out. The instrument automatically and continuously checks the stability of the measured values until the set criteria are met (6.2.4). Then the instrument refills the sample for the next determination.

10.4.5 If the difference between the determinations is within the set determinability limits (Clause 9), then the values of the last determination are given as valid measured result.

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10.4.6 If it is not possible to obtain a valid result within an applicable number of repetitions, report the robust mean value and the standard deviation (k = 2; 95 % confidence level) together with a remark indicating that the determinability exceeded the limits stated in Clause 9 for this product.

10.4.7 After the measurement cycle, the cells are cleaned and dried fully automatically and the cycle is repeated for all vessels in the magazines.

11 Calculation

Record the dynamic viscosity, in millipascal seconds, to four significant digits and the density to the nearest 0,1 kg/m³. Calculate the kinematic viscosity in square millimetres per second using dynamic viscosity/density.

The calculation of kinematic viscosity is usually performed automatically by the instrument using Formula (1):

$$v = 1\,000 * \eta / \rho \tag{1}$$

where

v is the kinematic viscosity, expressed in square millimetres per second (mm²/s);

η is the dynamic viscosity, expressed in millipascal seconds (mPa · s);

ρ is the density, expressed in kilograms per cubic metre (kg/m³).

12 Expression of results

Express the dynamic (η) and kinematic (v) viscosity to four significant digits, the density (ρ) to the nearest 0,1 kg/m³ and the test temperature to the nearest 0,01 °C.

13 Precision

13.1 General

The precision statement was developed by statistical evaluation of the results of a CEN interlaboratory study with 21 laboratories on a matrix of 12 samples, in accordance with ISO 4259:2006[1].

The set of samples comprised single FAME types (e.g. palm-kernel- and rape-seed-based), blends of methyl esters, different middle distillates and a mix (50/50) of paraffinic diesel fuel (GTL) and arctic diesel fuel MK1 covering a range of viscosity at 40 °C from 2,61 mm²/s to 5,50 mm²/s.

Only the equations for kinematic viscosity are given; for other precision data see Reference [2].

13.2 Repeatability, r

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the value indicated in Formula (2) in only one case in 20.

$$r = 0,01\,05 - 0,000\,3X \tag{2}$$

where X is the average of the two results being compared, in mm²/s.