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Petroleum products and related products — Determination of kinematic viscosity by calculation from the measured dynamic viscosity and density – Method by constant pressure viscometer

Produits pétroliers et produits connexes — Détermination de la viscosité cinématique par calcul à partir des mesures de viscosité dynamique et de masse volumique — Méthode par viscosimètre à pression constante

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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 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).

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

The purpose of this document is to specify a procedure for measuring dynamic viscosity and density, and then calculating kinematic viscosity from these measurements, when applied to petroleum and related liquids. Kinematic viscosity is often a characteristic that is specified in product specifications and is a frequent measurement in testing laboratories. The constant pressure viscometer provides a versatile and efficient technique using less time and labour for the laboratory.

Petroleum products and related products — Determination of kinematic viscosity by calculation from the measured dynamic viscosity and density – Method by constant pressure viscometer

1 Scope

This document specifies a procedure for determining dynamic viscosity, η, and density, ρ, for the calculation of kinematic viscosity, ν, of middle distillate fuels, fatty acid methyl ester fuels (FAME) and mixtures thereof, up to 60 % with middle distillate fuels, and lubricating oils (e.g. base oils, formulated oils), and synthetics, using a constant pressure viscometer. The range of kinematic viscosities covered in this test method is from 0,5 mm^2/s to 2 000 mm^2/s, with precision at 40 °C from 1,0 mm^2/s to 1 286 mm^2/s, and precision at 100 °C from 3,0 mm^2/s to 157 mm^2/s.

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 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

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
density
ρ
mass per unit volume of a substance at a given temperature

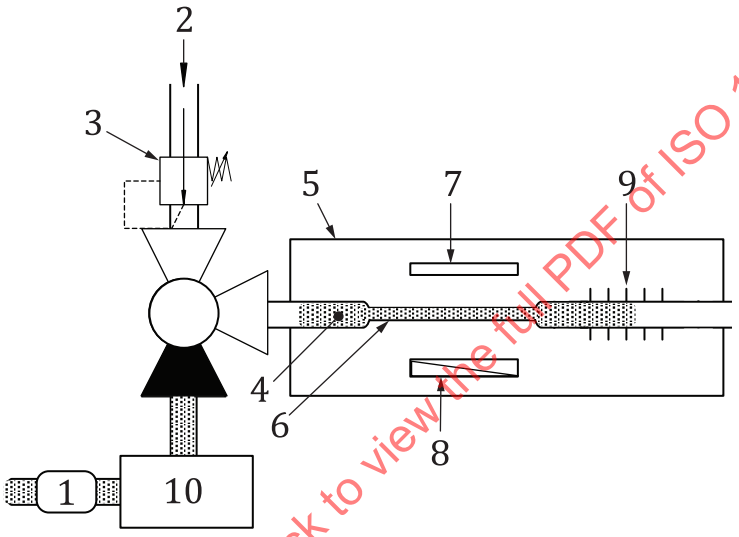
3.2
dynamic viscosity
η
ratio between the applied shear stress and rate of shear of a liquid at a given temperature

3.3
kinematic viscosity
ν
resistance to flow of a fluid under gravity.

Note 1 to entry: The kinematic viscosity can be calculated by dividing the *dynamic viscosity* (3.2) with *density* (3.1).

4 Principle

A test portion is delivered to the temperature-controlled measuring section consisting of a horizontal capillary tube with optical sensors and an oscillating U-tube densitometer (see Figure 1). The test portion is forced through the capillary viscometer under pressure and the time taken for a fixed volume to flow through a length of the tube and optical array is measured and automatically recorded. The density is determined by the oscillation frequency of the U-tube in conjunction with calculations. The kinematic viscosity is calculated by dividing the dynamic viscosity by the density.



Key
1 75 µm screen
2 compressed air
3 pressure regulator
4 specimen
5 thermal block
6 capillary
7 temperature sensor
8 thermoelectric cooling/heating
9 detector array
10 densitometer

Figure 1 — Overview of the measurement principle

5 Reagents and materials

5.1 Sample solvent, completely miscible with the sample.

For samples that are mutually soluble such as jet fuels and light middle distillate test specimens, it is suitable to use the same or similar middle distillates, or the next sample, as a solvent. If the solvent dries up without

residues in an applicable time frame, the use of a separate drying solvent is not required. For more viscous test specimen, a solvent such as heptane or toluene is suitable.

5.2 Drying solvent, a volatile solvent miscible with the sample solvent, *n*-pentane is suitable.

5.3 Dry air, for blowing and drying of the measuring cells.

5.4 Certified reference material, one or more certified reference materials depending on the range of viscosity, density, and temperature typically tested. The uncertainty of the reference standards shall be stated for each certified value (k = 2; 95 % confidence level).

6 Apparatus

6.1 Constant pressure viscometer, of the Hagen-Poiseuille principle of capillary flow, to determine the dynamic viscosity.

A capillary tube of known dimension enclosed horizontally in a controlled thermoelectric thermal block maintained at a constant temperature. The test portion is driven to flow along the tube by a constant and regulated pressure of compressed air. The transit time of the test portion as it flows past an array of optical detectors is measured (see Figure 1). The dynamic viscosity is proportional to the measured transit time as the product of the measured flow time and the calibration constant of the viscometer.

6.2 Densitometer, of a U-shaped oscillating sample tube with a system for electronic excitation and frequency counting that is suitable for calculating kinematic viscosity from dynamic viscosity and capable of achieving the stated precision for kinematic viscosity in this document.

6.3 Regulated air pressure device, able to maintain constant air pressure between 6,89 kPa to 68,9 kPa and cause a test specimen to flow along the capillary tube.

6.4 Controlled thermoelectric heating and cooling system, containing the viscosity measuring capillary tube maintained at a constant temperature, with temperature stability within ±0,01 °C from the programmed temperature (see Figure 1).

6.5 Sample flow line, 75 µm screen to remove particles or fibres from samples.

6.6 Sampling device, such as an autosampler, equipped to transfer a representative volume of test specimen into the measuring cells, which can have heating capability to lower the viscosity of the sample when filling the measuring cells. Approximately 20 ml of sample may be consumed.

7 Test portion preparation

7.1 Sampling

Samples shall be taken as described in ISO 3170 when sampling manually or ISO 3171 for automatic sampling.

7.2 Sample preparation

7.2.1 Mix the sample to homogenize at room temperature in a sample container. If loss of volatile material can occur in an open container, mixing in closed containers, or at sub-ambient temperature is recommended. Samples of a waxy nature or high viscosity may be heated to higher temperature to achieve proper mixing.

7.2.2 Deliver the test specimen from a properly mixed laboratory sample to the measuring cells using a sampling device such as an autosampler, approximately 20 ml is consumed. For waxy or samples with a high

pour point, before delivering the test specimen, heat the laboratory sample to a temperature high enough to dissolve wax crystals. Samples having high viscosity may be heated enough to allow for delivery to the measuring cells.

8 Calibration and verification

8.1 Use calibrated apparatus (6.1 and 6.2). Verify the calibration of the apparatus at least once a year by testing certified reference materials (CRM). It is recommended that more frequent verification checks are made using CRMs. Use one or more certified reference materials depending on the range of viscosity, density, and temperature typically tested.

8.2 Verify the calibration and operation of the apparatus according to the manufacturer's instructions.

9 Apparatus preparation

Prepare the apparatus for operation in accordance with the manufacturer's instructions.

10 Procedure

10.1 Place the prepared sample into position for sampling and programme the apparatus for the desired temperature or temperatures.

10.2 Begin the analysis. The test portion is driven to flow along the capillary tube by a constant and regulated pressure of compressed air, as determined by the apparatus, and not to exceed a flow time of 3 600 s. The transit time of the test portion as it flows past an array of optical detectors is measured (see Figure 1.). The dynamic viscosity is proportional to the measured transit time as the product of the measured flow time and the calibration constant of the viscometer.

Use two optimum transit flow times for determining the (average) dynamic viscosity.

10.3 Measure the density of the test portion and calculate the kinematic viscosity as in Clause 11.

11 Calculation

Calculate the kinematic viscosity in square millimetres per second using dynamic viscosity/density.

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

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

where

ν is the kinematic viscosity, expressed in square millimetres per second (mm^2/s);

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

ρ is the density, expressed in kilograms per cubic metre (kg/m^3).

12 Expression of results

Report the calculated kinematic viscosity, ν, in mm^2/s with four significant digits and the programmed test temperature.

Dynamic viscosity, η, in mPa s and density, ρ, in kg/m^3 can be reported, but the accuracy of these values has not been evaluated by this test method.

13 Precision

13.1 General

The precision statement for the kinematic viscosity was developed by statistical evaluation of the calculation of dynamic viscosity and density results from an international interlaboratory study with eight laboratories on a matrix of 23 samples at 40 °C including the viscosity range of 1,0 mm^2 to 1 286 mm^2, and 10 samples at 100 °C including the viscosity range of 3,0 mm^2 to 157 mm^2, in accordance with ISO 4259-1:2017/Amd.2:2020. Required degrees of freedom were exceeded for all formulae as presented in Table 1.

The multi-national sample set comprised different middle distillates, blends with methyl esters up to 60 %, FAME, heating oil, kerosine, base oil, formulated engine oil, synthetic engine oil, gear lube, and polyalphaolefins , tested at 40 °C. Samples tested at 100 °C were comprised of base oil, formulated engine oil, synthetic engine oil, gear lube, and polyalphaolefins.

13.2 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 that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using the functions as in Table 1.

13.3 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 that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using the functions as in Table 1.

Table 1 — Precision for kinematic viscosity

Test temperature	Repeatability, *r*	Reproducibility, *R*
40 °C	0,007 281(X+1)	0,008 485(X+1)
100 °C	0,003 713$X^{1,147\,4}$	0,006 039$X^{1,147\,4}$
X is the average of the two results being compared, in mm^2/s.		

14 Test report

The test report shall contain at least the following information:

a) reference to this document, i.e. ISO 18335:2024;

b) type and complete identification of the product tested;

c) result of the test (see Clause 12);

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

e) any unusual features observed;

f) date of the test.