INTERNATIONAL STANDARD 3104

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3104 was drawn up by Technical Committee ISO/TC 28, *Petroleum products,* and circulated to the Member Bodies in April 1973.

It has been approved by the Member Bodies of the following countries :

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Bulgaria	Israel	Sweden
Canada	Mexico	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Norway	U.S.A.
Germany	Poland	U.S.S.R.
Ghana	Portugal	

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies a procedure for the determination of the kinematic viscosity of liquid petroleum products, both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid.

1.2 This International Standard is intended for application to liquids for which, for practical purposes, the shear stress and shear rate may be considered proportional, i.e. the coefficient of viscosity is independent of the rate of shear. This ideal behaviour is commonly called Newtonian flow behaviour. If a liquid departs significantly from this behaviour, different results may be obtained from viscometers with different capillary diameters through which the rate of shear will be different.

1.3 This International Standard is not intended for the measurement of the viscosity of bitumens.

2 DEFINITIONS

For the purpose of this International Standard, the following definitions apply :

2.1 kinematic viscosity : The ratio between the viscosity and the density of the liquid. It is a measure of the resistance to flow of a liquid under gravity. In the SI the unit of kinematic viscosity is the square metre per second (m^2/s); for practical use a submultiple is more convenient. The centistokes (cSt) is $10^{-6}\ m^2/s$ (i.e. 1 cSt = 1 mm^2/s) and is customarily used.

2.2 dynamic viscosity (coefficient of) : The ratio between the applied shear stress and the rate of shear. This coefficient is thus a measure of the resistance to flow of the liquid. In the SI the unit of dynamic viscosity is the pascal second (Pa·s); for practical use a submultiple is more convenient. The centipoise (cP) is 10^{-3} Pa·s (i.e. 1 cP = 1 mPa·s) and is customarily used.

2.3 Newtonian liquid : A liquid having a viscosity that is independent of the shear stress or shear rate. If the ratio of shear stress to shear rate is not constant, the liquid is non-Newtonian.

3 PRINCIPLE

The time is measured, in seconds, for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the meaured flow time and the calibration constant of the viscometer derived by direct or step-up calibration with distilled water, which is the primary kinematic viscosity standard.

4 APPARATUS

4.1 Viscometers

Viscometers of the glass capillary type, calibrated and capable of measuring kinematic viscosity within the limits of precision given in 7.2, are acceptable (see note 1). The viscometers listed in table 1 meet these requirements (see note 2). It is not intended to restrict this International Standard either to the use of only those viscometers listed in table 1 or to the use of U tube viscosimeters.

NOTES

1 Procedures for the calibration of viscometers are given in ISO 3105, *Glass capillary kinematic viscometers — Specification and operating instructions.*

2 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this must, therefore, be supplied by the calibration laboratory together with the instrument constant. Where the acceleration of gravity, g, in the two locations differs by more than 0,1 %, correct the calibration constant as follows :

$$C_2 = \frac{g_2}{g_1} \times C_1$$

where the subscripts 1 and 2 indicate respectively the calibration laboratory and the testing laboratory.

4.2 Viscometer holders

The holder shall allow the viscometer to be suspended in a position similar to that adopted for calibration. The proper alignment of a vertical datum part may be confirmed by using a plumb line.

4.3 Viscometer thermostat and bath

Any transparent liquid or vapour bath may be used provided that it is of sufficient depth that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

The temperature control must be such that for the range of 15 to 100 °C, the temperature of the bath medium does not vary by more than 0,01 °C over the length of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the variation must not exceed 0,03 °C.

4.4 Temperature measuring device

Calibrated liquid-in-glass thermometers (see table 2) of an accuracy after correction of 0,02 °C may be used, or any other thermometric device of equal accuracy. All readings and corrections shall be made to the nearest 0,01 °C.

4.5 Timing device

Any timing device may be used provided that the readings can be taken with a discrimination of 0,2 s or better, and that it has an accuracy within ± 0,07 % when tested over intervals of 15 min.

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

5 CALIBRATION

5.1 Viscometers

Use only calibrated viscometers with constants measured and expressed to the nearest 0,1 % of their respective values.

5.2 Thermometers

Calibrated liquid-in-glass thermometers shall be checked to the nearest 0,01 °C by direct comparison with a suitable calibrated reference thermometer.

5.2.1 Calibrated thermometers shall be checked for accuracy at total immersion, which means immersion to the top of the mercury column, with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature; the expansion bulb should not be immersed.

5.2.2 The ice point of calibrated thermometers shall be determined periodically and the certified correction shall be adjusted to conform to any change in the ice point. The possible change in the ice point reading of new thermometers may require checking every week.

5.3 Timers

Standard time signals available in some countries may be used for checking the accuracy of timing devices.

5.4 Viscosity oil standards[1] (table 3)

These may be used as confirmatory checks on the procedure in the laboratory. If the measured kinematic viscosity does not agree within ± 0,35 % of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration, to locate the source of anomalous result. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of anomalous result.

6 PROCEDURE

6.1 Kinematic viscosity

6.1.1 The specific details of operation vary for the different types of viscometers listed in table 1. In all cases, however, proceed in accordance with 6.1.2 to 6.1.6 inclusive.

6.1.2 Maintain the bath at the test temperature according to the corrected readings of the thermometer within the limits given in 4.3. Locate the thermometer in the bath with the top of the mercury column below the surface of the bath liquid, and the emergent stem of the thermometer above the cover of the bath.

6.1.2.1 Ascertain that the ice point of the thermometer has been determined recently and the corrections, if any, applied to the calibration values. For thermometers with an auxiliary ice point scale, a change in the ice point determination is an indication of a need for recalibration.

6.1.2.2 Select a clean, dry, calibrated viscometer having a range covering the estimated viscosity (i.e. a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time should not be less than 200 s.

Viscometers used for silicone fluids, fluorocarbons, and other liquids which are difficult to remove by the use of a cleaning agent, shall be used only for the determination of the kinematic viscosity of such fluids except when calibrating. Such viscometers shall be subjected to calibration checks at frequent intervals.

1) Viscosity oil standards having the approximate kinematic viscosities shown in table 3 are available in certain countries from national laboratories or other authorized sources.

6.1.2.3 When the temperature of the test is below the dew point, fit loosely packed drying tubes onto the open ends of the viscometer to prevent water condensation. Drying tubes must fit the design of the viscosimeter and not restrict the flow of the sample under test by pressures created in the instrument.

For determinations at temperatures below 0 °C, it may be advisable to charge the sample into the viscometer at ambient temperature; allow the viscometer to cool to bath temperature, keeping the sample in the working capillary to prevent slight accumulation of frost on the walls of the capillary.

6.1.3 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample contains solid particles, filter through a 75 μm screen during charging.

6.1.3.1 With certain products which exhibit gel-like behaviour, take care that measurements are made at sufficiently high temperatures for such materials to flow freely so that similar results will be obtained in viscometers of different capillary diameters (see 1.2).

6.1.3.2 The viscosity of steam-refined cylinder oils, black lubricating oils, residual fuel oils, and similar waxy products can be affected by the previous thermal history. The following preheating procedure should be followed to obtain uniform results for viscosities below 95 °C :

To obtain a representative sample, heat the sample in the original container to about 50 °C with stirring and shaking. Probe the bottom of the container with a rod to be certain that all waxy materials are in solution. Pour 100 ml into a 125 ml conical flask. Stopper loosely with a cork or rubber stopper. Immerse the flask in a bath of boiling water for 30 min. Mix well, remove the sample from the bath, and strain it through a 75 μm screen directly into the viscometer already in the thermostatically controlled bath. Complete the viscosity test within 1 h after preheating.

6.1.4 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because this time will vary for the different instruments and for different temperatures, establish a safe temperature equilibrium time by trial (30 min should be sufficient). Where the design of the viscometer requires it, adjust the volume of the test sample after the sample has reached temperature equilibrium. One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring flow time, to avoid disturbing the temperature equilibrium.

6.1.5 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 5 mm ahead of the first timing mark. With the sample flowing freely, measure in seconds, to within 0,2 s (see 4.5), the time required for the meniscus to pass from the first timing mark to the second. If this flow time is less than the specified minimum (see 6.1.2.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

6.1.6 For modified Ostwald and suspended-level types, repeat the procedure described in 6.1.5 to make a second measurement of the flow time. If two measurements agree within 0,2 %, use the average for calculating the reported kinematic viscosity.

For reverse-flow types, duplicate determinations must be made; flow times should agree within 0,35 %. If these agreements are not obtained, reject the test results.

6.2 Dynamic viscosity

6.2.1 Determine the kinematic viscosity as described in 6.1.

6.2.2 Determine the density of the sample to 0,001 g/cm³ at the same temperature as the kinematic viscosity, in accordance with a suitable standard method.

6.3 Cleaning of viscometer

6.3.1 Between successive determinations, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. 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.

6.3.2 Periodically clean the instrument with chromic acid to remove organic deposits, rinse thoroughly with distilled water and acetone, and dry with clean dry air. Inorganic deposits may be removed by hydrochloric acid treatment before the use of cleaning acid, particularly if barium salts are suspected.

7 EXPRESSION OF RESULTS

7.1 Calculation

7.1.1 Calculate the kinematic viscosity ν from the measured flow time t and the instrument constant C by means of the equation.

$$\nu = C \cdot t$$

where

ν is the kinematic viscosity, in square millimetres per second;

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

t is the flow time, in seconds.

7.1.2 Calculate the dynamic viscosity η from the calculated kinematic viscosity ν and the density ρ by means of the equation

$$\eta = \rho \cdot \nu$$

where

η is the dynamic viscosity, in millipascal seconds;

ρ is the density, in grams per cubic centimetre, at the same temperature as used for measuring the flow time t;

ν is the kinematic viscosity, in square millimetres per second.

7.2 Precision[1)]

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is as follows :

7.2.1 *Repeatability*

The difference between successive 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 following value only in one case in 20 :

0,35 % of the mean

7.2.2 *Reproducibility*

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in 20 :

0,7 % of the mean

8 TEST REPORT

Report the test results for both the kinematic and dynamic viscosity, rounded to the nearest 0,01 % of the value measured or calculated, respectively, and make reference to this International Standard.

TABLE 1 – Viscometer types

Viscometer identification	Range[1)] mm²/s
A. Modified Ostwald types for transparent liquids :	
1 Cannon-Fenske routine[2)]	0,5 to 20 000
2 Zeitfuchs	0,6 to 3 000
3 BS/IP U-tube[2)]	0,9 to 10 000
4 BS/IP-tube miniature	0,2 to 20 000
5 SIL[2)]	0,6 to 10 000
6 Cannon-Manning semi-micro	0,4 to 20 000
7 Pinkevitch[2)]	0,6 to 17 000
B. Suspended-level types for transparent liquids :	
1 BS/IP suspended-level[2)]	3,5 to 100 000
2 BS/IP suspended-level, shortened form[2)]	1,05 to 10 000
3 BS/IP miniature suspended-level	0,6 to 3 000
4 Ubbelohde[2)]	0,3 to 100 000
5 FitzSimons	0,6 to 1 200
6 Atlantic[2)]	0,75 to 5 000
7 Cannon-Ubbelohde, Cannon-Ubbelohde dilution[2)]	0,5 to 100 000
8 Cannon-Ubbelohde semi-micro	0,4 to 20 000
C. Reverse-flow types for transparent and opaque liquids :	
1 Cannon-Fenske opaque	0,4 to 20 000
2 Zeitfuchs cross-arm	0,6 to 100 000
3 BS/IP U-tube reverse flow	0,6 to 300 000
4 Lantz-Zeitfuchs	60 to 100 000

1) Each range quoted requires a series of viscometers. To avoid the necessity of making a kinematic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted.

2) In each of these series, the minimum flow time for the viscometers with lowest constants exceeds 200 s.

NOTE – Specifications and operating instructions for all these viscometers have been assembled in ISO 3105.

1) The precision applies only to clean, transparent liquids tested between 15 and 100 °C.

TABLE 2 – **Kinematic viscosity test thermometers**

Thermometer number[1]		For tests at	Subdivisions
ASTM	IP	°C	°C
	69 C	– 53,9	0,05
	65 C	– 51 to – 35	0,1
73 C	68 C	– 40	0,05
	67 C	– 17,8	0,05
	33 C	0	0,05
44 C	29 C	20 and 21,1	0,05
45 C	30 C	25	0,05
118 C		30	0,05
	31 C	37,8	0,05
120 C		40	0,05
46 C	66 C	50	0,05
	34 C	54,4	0,05
47 C	35 C	60	0,05
	90 C	82,2	0,05
	36 C	93,3	0,05
	32 C	98,9 and 100	0,05
121 C		100	0,05
110 C		135	0,05

1) The essential requirements for these thermometers are given in ASTM specification E1, for ASTM thermometers, and in the specifications for IP standard thermometers.

TABLE 3 – **Typical viscosity oil standards**[1]

Viscosity standard designation	Approximate kinematic viscosity, mm^2/s, at								
	– 53,89 °C	– 40 °C	20 °C	25 °C	37,78 °C	40 °C	50 °C	98,89 °C	100 °C
3	300	80	4,6	4,0	3,0	2,9	–	1,2	1,2
6	–	–	11	8,9	6,0	5,7	–	1,8	1,8
20	–	–	44	34	20	18	–	4,0	3,9
60	–	–	170	120	60	54	–	7,4	7,2
200	–	–	640	450	200	180	–	17	17
600	–	–	2 400	1 600	600	520	280	33	32
2 000	–	–	8 700	5 600	2 000	1 700	–	78	75
8 000	–	–	37 000	23 000	8 000	6 700	–	–	
30 000	–	–	–	81 000	27 000	23 000	11 000	–	

1) The actual values for these standards are established and reaffirmed annually by co-operative tests. In 1971, tests were made using 15 different types of viscometer in 26 laboratories located in 9 countries.

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ERRATUM

Page 4

Replace table 1 by the new table shown below, in which the viscometer identifications are aligned on those given in ISO 3105-1976, and a corrected kinematic viscosity range is given for the viscometer type A.4.

TABLE 1 — **Viscometer types**

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