

INTERNATIONAL
STANDARD

ISO
3104

Second edition
1994-10-15

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

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Produits pétroliers — Liquides opaques et transparents — Détermination de la viscosité cinématique et calcul de la viscosité dynamique

ISO 3104:1994

<https://standards.iteh.ai/catalog/standards/iso/49d746f2-d9f1-4714-814c-c16ae25f59a/iso-3104-1994>



Reference number
ISO 3104:1994(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3104 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 3104:1976), of which it constitutes a technical revision.

Annexes A, B and C form an integral part of this International Standard.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Introduction

Many petroleum products, and some non-petroleum materials, are used as lubricants, and 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.

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

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a procedure for the determination of the kinematic viscosity, ν , 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, η , can be obtained by multiplying the measured kinematic viscosity by the density, ρ , of the liquid.

NOTE 1 The result obtained from this International Standard 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 may 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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most re-

cent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3105:—¹⁾, *Glass capillary kinematic viscometers — Specifications and operating instructions*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 kinematic viscosity, ν : Resistance to flow of a fluid under gravity.

NOTE 2 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 , where $\nu = \eta/\rho$, and where η is the dynamic viscosity coefficient.

3.2 density, ρ : Mass per unit volume of a substance at a given temperature.

3.3 dynamic viscosity, η : Ratio between the applied shear stress and rate of shear of a liquid. It is sometimes called the coefficient of dynamic viscosity, or simply viscosity. Thus dynamic viscosity is a

1) To be published. (Revision of ISO 3105:1976)

measure of the resistance to flow or deformation of a liquid.

NOTE 3 The term dynamic viscosity can also be used in a different context to denote a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence.

4 Principle

The time is measured 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 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 Chromic acid cleaning solution, or a nonchromium-containing, strongly-oxidizing acid cleaning solution.

WARNING — Chromic acid is a health hazard. It is toxic, a recognized carcinogen, highly corrosive and potentially hazardous in contact with organic materials. If used, wear a full face-shield and full-length protective clothing including suitable gloves. Avoid breathing vapour. Dispose of used chromic acid carefully as it remains hazardous. Nonchromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.

5.2 Sample solvent, completely miscible with the sample. Filter before use.

NOTE 4 For most samples a volatile petroleum spirit or naphtha is suitable. For residual fuels, a prewash with an aromatic solvent such as toluene or xylene may be necessary to remove asphaltenic material.

5.3 Drying solvent, volatile and miscible with both the sample solvent (5.2) and water (5.4). Filter before use.

NOTE 5 Acetone is suitable.

5.4 Water, deionized or distilled, conforming to Grade 3 of ISO 3696. Filter before use.

5.5 Certified viscosity reference standards, for use as confirmatory checks on the procedure in the laboratory.

6 Apparatus

6.1 Viscometer, calibrated, of the glass capillary type, capable of measuring kinematic viscosity within the limits of precision given in clause 14 (see annex A).

NOTE 6 Viscometers listed in table A.1, whose specifications meet those given in ISO 3105, meet these requirements. It is not intended to restrict this test method to the use of only those viscometers listed in table A.1; annex A gives further guidance.

Automated viscometers, which have been shown to measure kinematic viscosity within the limits of precision given in clause 14, are acceptable alternatives. Apply a kinetic energy correction (see ISO 3105) to kinematic viscosities less than $10 \text{ mm}^2/\text{s}$ and flow times less than 200 s.

6.2 Viscometer holder, enabling all viscometers which have the upper meniscus directly above the lower meniscus to be suspended vertically within 1° in all directions. Those viscometers whose upper meniscus is offset from directly above the lower meniscus shall be suspended vertically within $0,3^\circ$ in all directions (see ISO 3105).

NOTE 7 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 wholly satisfactory.

6.3 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 $\pm 0,02^\circ\text{C}$ from the selected temperature over the length of the viscometer, or between the position of each viscometer, or at the location of the thermometer. For temperatures outside this range, the deviation from the desired temperature shall not exceed $\pm 0,05^\circ\text{C}$.

6.4 Temperature-measuring device, for the range 0 °C to 100 °C, either calibrated liquid-in-glass thermometer (annex B) of an accuracy after correction of $\pm 0,02$ °C or better, or any other thermometric device of equal or better accuracy. When two thermometers are used in the same bath, they shall agree within 0,04 °C.

NOTE 8 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 of an accuracy after correction of $\pm 0,05$ °C or better shall be used, and when two thermometers are used in the same bath they shall agree within $\pm 0,1$ °C.

6.5 Timing device, capable of taking readings with a discrimination of 0,1 s or better, and having an accuracy within $\pm 0,07$ % (see annex C) of the reading when tested over intervals of 200 s and 900 s.

NOTE 9 Electrical timing devices may be used if the current frequency is 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 actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

7 Calibration and verification

7.1 Verify the viscometer calibration following the laboratory procedure using a certified viscosity reference standard (5.5). If the measured kinematic viscosity does not agree within $\pm 0,35$ % of the certified value, recheck each step in the procedure, including thermometer and viscometer calibrations, to locate the source of error. Table 1 in ISO 3105 gives details of standards available.

NOTE 10 The most common sources of error are caused by particles of dust lodged in the capillary bore and temperature measurement errors. It should 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 error.

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

$$C_2 = (g_2/g_1)C_1$$

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

8 General procedure for kinematic viscosity determination

8.1 Adjust and maintain the viscometer bath at the required test temperature within the limits given in 6.3, taking account of the conditions given in annex B and of the corrections supplied on the certificates of calibration for the thermometers.

Thermometers shall be held in an upright position under the same conditions of immersion as when calibrated.

NOTE 11 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used (see 6.4). They should be viewed with a lens assembly giving approximately $\times 5$ magnification and be arranged to eliminate parallax errors.

8.2 Select a clean, dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time shall not be less than 200 s or the longer time noted in ISO 3105.

NOTE 12 The specific details of operation vary depending on the type of viscometer. The operating instructions for the different types of viscometers listed in table A.1 are given in ISO 3105.

8.2.1 When the test temperature is below the dew point, affix loosely-packed drying tubes to the open ends of the viscometer. The drying tubes shall fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument. Carefully flush the moist room air from the viscometer by applying vacuum to one of the drying tubes. Finally, before placing the viscometer in the bath, draw up the sample into the working capillary and timing bulb and allow to drain back, as an additional safeguard against moisture condensing or freezing on the walls.

8.2.2 Viscometers used for silicone fluids, fluorocarbons and other liquids which are difficult to remove by the use of 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.

9 Determination of viscosity of transparent liquids

9.1 Charge the viscometer and place in the bath 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 during charging through a (75 µm) filter (see ISO 3105).

NOTE 13 In general, the viscometers used for transparent liquids are of the type listed in table A.1, types A and B.

9.1.1 With certain products which exhibit "gel-like" behaviour, take care that measurements are made at temperatures sufficiently high for such materials to flow freely, so that similar kinematic viscosity results are obtained in viscometers of different capillary diameter.

9.1.2 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Where one bath is used to accommodate several viscometers, never add or withdraw a viscometer while any other viscometer is being used to measure flow time.

Because time in the bath will vary for different instruments, for different temperatures and for different kinematic viscosities, establish a safe equilibrium time by trial.

NOTE 14 30 min should be sufficient, except for the highest kinematic viscosities.

9.1.3 Where the design of the viscometer requires it, adjust the volume of the sample to the mark after the sample has reached temperature equilibrium.

9.2 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 approximately 7 mm above the first timing mark, unless any other value is stated in the operating instructions for the viscometer. With the sample flowing freely, measure, in seconds to within 0,1 s (see 6.5), the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 8.2), select a viscometer with a capillary of smaller diameter and repeat the operation.

9.2.1 Repeat the procedure described in 9.2 to make a second measurement of flow time. Record the result.

9.2.2 If the two measurements agree with the stated determinability figure (see 14.1) for the product, calculate the average of the two measurements and use this value to calculate the kinematic viscosity. If the measurements do not agree, repeat the determination after thorough cleaning and drying of the viscometer and filtering of the sample. Record the result.

10 Determination of viscosity of opaque liquids

10.1 For steam-refined cylinder oils and black lubricating oils, proceed to 10.2 ensuring a thoroughly representative sample is used. For residual fuel oils and similar waxy products, whose kinematic viscosity can be affected by their thermal history, the procedure described in 10.1.1 to 10.1.6 shall be followed to minimize this effect.

NOTE 15 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in table A.1, type C.

10.1.1 Heat the sample in its original container, in an oven, at 60 °C ± 2 °C for 1 h.

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

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

NOTE 16 With samples of a very waxy nature or samples 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.

10.1.4 Immediately thereafter pour sufficient sample to fill two viscometers into a 100 ml glass flask and loosely stopper the flask.

10.1.5 Immerse the flask in a bath of boiling water for 30 min.

NOTE 17 Care should be taken, as vigorous boil-over may occur when opaque liquids which contain high levels of water are heated to high temperatures.

10.1.6 Remove the flask from the bath, stopper tightly and shake for 1 min.

10.2 Charge two viscometers and place in the bath in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 75 µm filter into two viscometers previously placed in the bath. For samples subjected to heat pretreatment (10.1), use a preheated filter to prevent the sample coagulating during the filtration.

NOTE 18 Viscometers which are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample. This is to ensure that the sample will not be cooled below test temperature.

10.2.1 After 10 min in the bath, adjust the volume of the sample (where the design of the viscometer requires) to coincide with the filling marks as in the viscometer specifications (see ISO 3105).

10.2.2 Allow the charged viscometers enough time in the bath to reach the test temperature. (See note 14.) Where one bath is used to accommodate several viscometers, never add or withdraw a viscometer while any other viscometer is being used to measure flow time.

10.3 With the sample flowing freely, measure, to the nearest 0,1 s (see 6.5), the time required for the advancing ring of contact to pass from the first timing mark to the second. Record the result.

In the case of samples requiring heat pretreatment (10.1), complete the determinations within 1 h of completing the pretreatment.

10.4 Calculate the mean kinematic viscosity, ν , in mm^2/s , from the two determinations.

For residual fuel oils, if the two measurements agree within the stated determinability figure (see 14.1), calculate the average of the two measurements and use this value to calculate the kinematic viscosity to be reported. If the measurements do not agree, repeat the determination after thorough cleaning and drying of the viscometer and filtering of the sample. Record the result.

NOTE 19 For other opaque liquids, no precision data is available.

11 Cleaning of the viscometer

11.1 Between successive determinations, clean the viscometer thoroughly by several rinsings with the sample solvent (5.2), followed by the drying solvent

(5.3). 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.

11.2 Periodically clean the viscometer with the cleaning solution (CAUTION — see 5.1) for several hours to remove residual traces of organic deposits, rinse thoroughly with water (5.4) and drying solvent (5.3), and dry with filtered dry air or a vacuum line. Remove any inorganic deposits with hydrochloric acid treatment before the use of cleaning solution, particularly if the presence of barium salts is suspected.

11.3 Alkaline cleaning solutions shall not be used, as changes in the viscometer calibration may occur.

12 Calculation

12.1 Calculate the kinematic viscosity, ν , from the measured flow time, t , and the viscometer constant, C , by means of the following equation:

$$\nu = C \times t$$

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 [mm^2/s^2];

t is the mean flow time, in seconds.

12.2 Calculate the dynamic viscosity, η , from the calculated kinematic viscosity, ν , and the density, ρ , by means of the following equation:

$$\eta = \nu \times \rho \times 10^{-3}$$

where

η is the dynamic viscosity, in millipascal seconds;

ρ is the density, in kilograms per cubic metre, at the same temperature used for the determination of the kinematic viscosity;

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

NOTE 20 The density of the sample may be determined by an appropriate method such as ISO 3675, and corrected to the temperature of determination by means of ISO 91-1.

13 Expression of results

Report the test results for kinematic viscosity and/or dynamic viscosity to four significant figures, together with the test temperature.

14 Precision

14.1 Determinability (d)

The difference between successive determinations obtained by the same operator in the same laboratory using the same apparatus for a series of operations leading to a single result, would in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty:

Base oils at 40 °C and 100 °C²⁾:
0,0020y (0,20 %)

Formulated oils at 40 °C and 100 °C³⁾:
0,0013y (0,13 %)

Formulated oils at 150 °C⁴⁾:
0,015y (1,5 %)

Petroleum wax at 100 °C⁵⁾:
0,0080y (0,80 %)

Residual fuel oils at 80 °C and 100 °C⁶⁾:
0,011(y + 8)

Residual fuel oils at 50 °C⁶⁾:
0,017y (1,7 %)

where y is the average of determinations being compared.

14.2 Repeatability (r)

The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test ma-

terial would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty:

Base oils at 40 °C and 100 °C²⁾:
0,0011x (0,11 %)

Formulated oils at 40 °C and 100 °C³⁾:
0,0026x (0,26 %)

Formulated oils at 150 °C⁴⁾:
0,0056x (0,56 %)

Petroleum wax at 100 °C⁵⁾:
0,0141x^{1,2}

Residual fuel oils at 80 °C and 100 °C⁶⁾:
0,013(x + 8)

Residual fuel oils at 50 °C⁶⁾:
0,015x (1,5 %)

where x is the average of results being compared.

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14.3 Reproducibility (R)

The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of this test method, exceed the values indicated below only in one case in twenty:

Base oils at 40 °C and 100 °C²⁾:
0,0065x (0,65 %)

Formulated oils at 40 °C and 100 °C³⁾:
0,0076x (0,76 %)

Formulated oils at 150 °C⁴⁾:
0,018x (1,8 %)

Petroleum wax at 100 °C⁵⁾:
0,0366x^{1,2}

2) These precision values were obtained by statistical examination of interlaboratory results from six mineral oils in the range 8 mm²/s to 1 005 mm²/s at 40 °C and 2 mm²/s to 43 mm²/s at 100 °C, and were first published in 1989.

3) These precision values were obtained by statistical examination of interlaboratory results from seven fully formulated engine oils in the range of 36 mm²/s to 340 mm²/s at 40 °C and 6 mm²/s to 25 mm²/s at 100 °C, and were first published in 1991.

4) These precision values were obtained by statistical examination of interlaboratory results for eight fully formulated engine oils in the range of 7 mm²/s to 19 mm²/s at 150 °C, and were first published in 1991.

5) These precision values were obtained by statistical examination of interlaboratory results from five petroleum waxes in the range 3 mm²/s to 16 mm²/s at 100 °C, and were first published in 1988.

6) These precision values were obtained by statistical examination of interlaboratory results from fourteen residual fuel oils in the range 30 mm²/s to 1 300 mm²/s at 50 °C and 5 mm²/s to 170 mm²/s at 80 °C and 100 °C, and were first published in 1984.