## INTERNATIONAL STANDARD



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# Glass capillary kinematic viscometers — Specifications and operating instructions

Viscosimètres à capillaire, en verre, pour viscosité cinématique — Spécifications et mode d'emploi





Reference number ISO 3105:1994(E)

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### Glass capillary kinematic viscometers and operating instructions

## **Specifications**

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

#### 1 Scope

This International Standard gives specifications and operating instructions for glass capillary viscometers widely used for the determination of kinematic viscosity of petroleum products by the procedure described in ISO 3104. The calibration of these viscometers is also described.

The types of viscometers described are modified Ostwald viscometers (annex A), suspended-level viscometers (annex B) and reverse-flow viscometers (annex C). Other viscometers of the glass capillary type which are capable of measuring kinematic viscosity within the limits of precision given in ISO 3104 may be used.

#### 2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3104:1994. Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity.

#### 3 Symbols for viscometer parts

Letters are used to designate specific parts of each viscometer described in the annexes. These letters are also used in the text of this International Standard when reference to the viscometers is given. The more frequently used letters on the figures in the annexes are as follows:

- A Lower reservoir
- B Suspended level bulb
- C and J Timing bulbs
- D Upper reservoir
- E, F and Timing marks
- G and H Filling marks
- K Overflow tube
- L Mounting tube
- M Lower vent tube
- N Upper vent tube
- P Connecting tube
- R Working capillary

# 4 Viscometer materials and manufacture

**4.1** Fully annealed, low-expansion borosilicate glass shall be used for the construction of all viscometers. The size number, serial number and manufacturer's designation shall be permanently marked on each viscometer. All timing marks shall be etched and filled with an opaque colour, or otherwise made a permanent part of the viscometer.

**4.2** With the exception of the FitzSimons and Atlantic viscometers, all viscometers shall be designed to fit through a 51 mm hole in the lid of a constant-temperature bath having a liquid depth of at least 280 mm; it is assumed that the surface of the Tth liquid will be not more than 45 mm from the top

the bath lid.

NOTE 1 For certain constant-temperature baths, especially at low or high temperatures, it may be necessary to construct the viscometers with the uppermost tubes longer than shown in the annexes to ensure adequate immersion in the constant-temperature bath. Viscometers so modified can be used to measure kinematic viscosity within the precision of the test method. The lengths of tubes and bulbs shown in the figures should be maintained within  $\pm$  10 % or  $\pm$  10 mm, whichever is less, such that the calibration constant of the viscometer does not vary by more than  $\pm$  15 % from the nominal value.

#### 5 Viscometer holder and alignment

All viscometers which have the upper meniscus directly above the lower meniscus (Cannon-Fenske mutine in annex A and all viscometers in annex B)

Il be mounted in a constant-temperature bath with tube L held within 1° of the vertical, as observed with a plumb bcb or other equally accurate inspection means.

NOTE 2 A number of commercially available holders are so designed that the tube L is held perpendicular to the lid of a constant-temperature bath; nevertheless, the viscometer should be tested with a plumb line in order to ensure that the tube L is in a vertical position.

Those viscometers whose upper meniscus is offset from directly above the lower meniscus (all other viscometers in annex A and all viscometers in annex C) shall be mounted in a constant-temperature bath with tube L held within 0,3° of the vertical.

NOTE 3 Round metal tops, designed to fit above a 51 mm hole in the lid of the bath, are frequently cemented on to Zeitfuchs, Zeitfuchs cross-arm and Lantz-Zeitfuchs viscometers, which then are permanently mounted on the lid of the bath. Also a rectangular metal top,

25 mm  $\times$  59 mm, is often cemented on to Zeitfuchs cross-arm and Zeitfuchs viscometers. Viscometers fitted with metal tops should be set vertically in the constant-temperature bath with the aid of a plumb line.

In each figure, the numbers which follow the tube designation indicate the outside tube diameter, in millimetres. It is important to maintain these diameters and the designated spacing to ensure that holders will be interchangeable.

#### 6 Calibration of viscometers

#### 6.1 Procedures

Calibrate the kinematic glass capillary viscometers covered by this International Standard using the procedures specified in annexes A to C.

#### 6.2 Reference viscometers

**6.2.1** Select a clear petroleum oil, free from solid particles and possessing Newtonian flow characteristics, with a kinematic viscosity within the range of both the reference viscometer and the viscometer to be calibrated. The minimum flow time shall be greater than that specified in the appropriate table of the annex in both the reference viscometer and the viscometer which is to be calibrated in order that the kinetic energy correction (see 7.1) may be less than 0,2 %.

**6.2.2** Select a calibrated viscometer of known viscometer constant  $C_1$ .

NOTE 4 This viscometer may be a reference viscometer (driving head at least 400 mm) that has been calibrated by the step-up procedure using viscometers of successively larger diameters, starting with distilled water as the basic kinematic viscosity standard, or a routine viscometer of the same type that has been calibrated by comparison with a reference viscometer.

The calibration of the reference viscometer should only be carried out by a reputable laboratory meeting the requirements of, for example, ISO Guide 25.

Mount the calibrated viscometer together with the viscometer to be calibrated in the same bath and determine the flow times of the petroleum oil in accordance with ISO 3104.

**6.2.3** Calculate the viscometer constant  $C_1$  as follows:

$$C_1 = (t_2 \times C_2)/t_1 \tag{1}$$

where

- C<sub>1</sub> is the constant of the viscometer being calibrated;
- *t*<sub>1</sub> is the flow time, to the nearest 0,1 s, in the viscometer being calibrated;
- C<sub>2</sub> is the constant of the calibrated viscometer;
- *t*<sub>2</sub> is the flow time, to the nearest 0.1 s, in the calibrated viscometer.

**6.2.4** Repeat 6.2.1 to 6.2.3 with a second oil whose flow times are at least 50 % longer than the first oil. If the two values of  $C_1$  differ by less than 0.2 % for those viscometers listed in annexes A and B and less than 0.3 % for those viscometers listed in annex C, use the average as the viscometer constant for the viscometer being calibrated. If the constants differ by more than this value, repeat the procedure, taking care to examine all possible sources of errors.

6.2.5 The calibration constant, C, is dependent upon the gravitational acceleration at the place of calibration and this must, 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:

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

#### 6.3 Viscosity reference standards

Kinematic viscosity reference standards<sup>11</sup> are available having the approximate kinematic viscosity shown in table 1. Certified kinematic viscosity values are established by cooperative tests and are supplied with each delivery.

**6.3.1** Select from table 1 a viscosity reference standard with a kinematic viscosity at the calibration temperature within the kinematic viscosity range of the viscometer to be calibrated and a minimum flow time greater than that specified in the appropriate table of the annex. Determine the flow time, to the nearest 0,1 s, in accordance with ISO 3104, and calculate the viscometer constant, *C*, as follows:

$$C = v/t \tag{3}$$

where

- v is the kinematic viscosity, in millimetres squared per second, for the reference standard liquid;
- *i* is the flow time, in seconds.

Designation	Approximate kinematic viscosity mm <sup>2</sup> /s						
reference standard	°C I I I I I I I I I I I I I I I I I I I						
	- 40	20	25	40	50	100	
3	80	4,6	4.0	2.9	-	1,2	
6		11	8,9	5,7	-	1,8	
20		44	34	18	-	3,9	
60		170	120	54	—	7,2	
200		640	450	180	_	17	
600		2 400	1 600	520	280	32	
2 000		8 700	5 600	1 700	—	75	
8 000		37 000	23 000	6 700	_	-	
30 000		_	31 000	23 000	11 000		

Table 1 — Typical viscosity reference standards

 Viscosity reference standards are available in certain countries from national laboratories or other authorized sources. These reference liquids cover the range of all viscometers described in this international Standard. **6.3.2** Repeat with a second reference standard whose flow times are at least 50 % longer than the first reference standard. If the two values of C differ by less than 0,2 % for those viscometers listed in annexes A and B and less than 0,3 % for those viscometers listed in annex C, use the average as the viscometer constant for the viscometer being calibrated. If the constants differ by more than this value, repeat the procedure, taking care to examine all possible sources of errors.

#### 6.4 Expression of viscometer constant

Report the viscometer constant, *C*, to the nearest 0,1 % of the determined value. This generally means four significant figures from  $1 \times 10^{N}$  to  $6,999 \times 10^{N}$ .

#### 7 Kinematic viscosity calculation

#### 7.1 Basic formula

In principle, the calculation of kinematic viscosity is related to the dimensions of the viscometer according to the Hagen-Poiseuille law as follows:

$$v = (10^{6} \pi g D^{4} H t / 128 V L) - E / t^{2} \qquad \dots (4)$$

where

is the kinematic viscosity, in millimetres squared per second;

- g is the acceleration due to gravity, in metres per second squared;
  - D is the diameter of the capillary, in metres;
  - L is the length of the capillary, in metres;
  - *H* is the average distance between the upper and lower menisci (average driving head), in metres;
  - v is the timed volume of liquids passing through the capillary, in metres cubed (approximately the volume of the timing bulb);
  - *E* is the kinetic energy factor, in square millimetre seconds;

is the flow time, in seconds.

If the viscometer is selected so that the minimum flow time shown in the tables of annexes A to C is exceeded, the kinetic energy term,  $E/r^2$ , becomes insignificant and equation (4) may be simplified by

grouping the nonvariable terms into a constant, C, as follows:

$$v = Ct \tag{5}$$

#### 7.2 Kinetic energy correction

The viscometers described in the annexes A to C are designed such that the kinetic energy correction term,  $E/t^2$ , is negligible if the flow time is more than 200 s. In the case of several sizes of viscometers for the measurement of low kinematic viscosity liquids, a minimum flow time greater than 200 s is required in order that the kinetic energy correction term,  $E/t^2$ , shall be negligible. The minimum flow times required are given in footnotes to the appropriate tables of viscometer dimensions in the annexes A to C.

For viscometers whose constants C are  $0.01 \text{ mm}^2/\text{s}^2$  or less, the kinetic energy correction may be significant if the minimum 200 s flow time is not observed.

#### 7.3 Maximum flow time

A limit of 1 000 s has been set arbitrarily for convenience as the recommended maximum flow time for the viscometers covered by this International Standard. Longer flow times may be used.

#### 7.4 Surface tension correction

If the two menisci have different average diameters during the flow time and if the surface tension of the sample differs substantially from the calibrating liquid, a surface tension correction is necessary. The corrected constant,  $C_{\rm corr}$ , is given approximately as follows:

$$C_{\rm corr} = C[1 + (2/gH)(1/r_{\rm u} - 1/r_{\rm i})(\gamma_{\rm c}/\rho_{\rm c} - \gamma_{\rm t}/\rho_{\rm t})]$$
(6)

where

- g is the acceleration due to gravity, in metres per second squared;
- *H* is the average length of the driving head, in metres;
- is the average radius of the upper meniscus, in metres;

is the average radius of the lower meniscus, in metres;

is the surface tension, in newtons per metre;

ρ is the density, in kilograms per cubic metre.

Subscripts c and t relate to values obtained with the calibrating liquid and the test portion, respectively.

While this correction applies to all viscometers, a number of viscometers are designed to minimize the surface tension correction. The greatest correction normally encountered is with a viscometer calibrated with water and used for oils. Generally, viscometers are calibrated and used with hydrocarbons whose surface tensions are close enough for these corrections to be insignificant.

#### 7.5 Effect of temperature

**7.5.1** The viscometer constant, *C*, is independent of temperature for all those viscometers which have the volume of sample adjusted at bath temperature and for all suspended-level viscometers.

**7.5.2** The following viscometers, which have a fixed volume charged at ambient temperature, have a viscometer constant, *C*, which varies with temperature: Cannon-Fenske routine, Pinkevitch, Cannon-Manning semimicro, Cannon-Fenske opaque.

**7.5.2.1** The following equation can be used to calculate the viscometer constant at temperatures other than the calibration temperature for the Cannon-Fenske routine, Pinkevitch and Cannon-Manning semimicro viscometers:

$$C_2 = C_1 \left\{ 1 + [4 \ 000V(\rho_2 - \rho_1)] / (\pi D^2 H \rho_2) \right\}$$
(7)

where

- C<sub>1</sub> is the constant of the viscometer when filled and calibrated at the same temperature;
- V is the volume of charge, in millilitres;
- D is the average diameter of the meniscus in the lower reservoir for the Cannon-Fenske routine, Pinkevitch and Cannon-Manning semimicro viscometers, and in the upper reservoir of the Cannon-Fenske opaque viscometer, in millimetres;
- *H* is the average length of the driving head, in millimetres;
- $\rho$  is the density of the test liquid at the filling temperature, in kilograms per litre [kg/(m<sup>3</sup> × 10<sup>-3</sup>)];
- $\rho_2$  is the density of the test liquid at the test temperature, in kilograms per litre [kg/(m<sup>3</sup> × 10<sup>-3</sup>)];

**7.5.2.2** The temperature dependence of C for the Cannon-Fenske opaque (reverse-flow) viscometer is given as follows:

$$C_2 = C_1 \left\{ 1 - \left[ 4 \ 000V(\rho_2 - \rho_1) \right] / \left( \pi D^2 H \rho_2 \right) \right\} \dots (8)$$