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Glass capillary kinematic viscometers — Specification and operating instructions

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

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FOREWORD

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Glass capillary kinematic viscometers — Specification and operating instructions

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard gives specifications and operating instructions for glass capillary kinematic viscometers of all the types described in detail in annexes A, B, and C as follows :

- modified Ostwald viscometers, annex A
- suspended-level viscometers, annex B
- reverse-flow viscometers, annex C

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1.2 The calibration of the viscometers is described in clause 5.

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1.3 This International Standard covers some widely used viscometers suitable for use in accordance with ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*. 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 NOMENCLATURE FOR FIGURES

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 I	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

3 MATERIALS AND MANUFACTURE

3.1 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.

3.2 With the exception of the FitzSimons and Atlantic viscometers, all viscometers are 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; and it is assumed that the surface of the liquid will be not more than 45 mm from the top of the bath lid. 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 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 on the figures should be held within ± 10 % or ± 10 mm, whichever is less, such that the calibration constant of the viscometer does not vary more than 15 % from the nominal value.

4 VISCOMETER HOLDER AND ALIGNMENT

4.1 All viscometers shall be mounted in a constant-temperature bath with tube L held within 1° of the vertical as observed with a plumb bob or other equally accurate inspection means. 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.

4.2 Round metal tops, designed to fit above a 51 mm hole in the lid of the bath, are frequently cemented on to the 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 × 59 mm, is often cemented on to the Zeitfuchs cross-arm and Zeitfuchs viscometers. Viscometers fitted with metal tops should also be set vertically in the constant-temperature bath with the aid of a plumb line.

4.3 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.

5 CALIBRATION OF VISCOMETERS

5.1 Procedures

Calibrate the kinematic glass capillary viscometers covered by this International Standard using the procedures described in annexes A, B and C.

5.2 Viscosity oil standards

Viscosity oil standards[1] are available having the approximate kinematic viscosity shown in table 1. Certified kinematic viscosity values are established by co-operative tests and are supplied with each delivery.

5.3 Using oil standards of accurately known viscosity

Select from table 1 a viscosity oil 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 0,2 s in accordance with ISO 3104, clause 6, and calculate the viscometer constant, C, as follows :

$$C = \frac{\nu}{t}$$

where

ν is the kinematic viscosity, in square millimetres per second,[2] for the standard liquid;

t is the flow time, in seconds.

5.4 Using reference viscometers of accurately known viscometer constant

5.4.1 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 6.1) may be less than 0,2 %.

5.4.2 Select a calibrated viscometer of known viscometer constant C_1. This viscometer may be a reference viscometer (driving head at least 40 cm) 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. Mount the calibrated viscometer together with the viscometer to be calibrated in the same bath and determine the flow times of the oil in accordance with ISO 3104, clause 6.

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TABLE 1 — **Typical viscosity oil standards***

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	–	–

* 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.

1) Viscosity oil standards are available in certain countries from national laboratories or other authorized sources.

2) In this International Standard, kinematic viscosity is expressed in square millimetres per second (mm^2/s), which is a recommended sub-multiple of the SI unit (m^2/s) for this quantity. In practice, however, the centistokes (cSt) is generally used in this case in the petroleum and petrochemical industries. The values of the kinematic viscosity are unaffected by this practice in view of the fact that 1 cSt = 1 mm^2/s.

5.4.3 Calculate the viscometer constant C_1 as follows :

$$C_1 = \frac{t_2 \times C_2}{t_1}$$

where

C_1 is the constant of the viscometer being calibrated;

t_1 is the flow time to the nearest 0,2 s in the viscometer being calibrated;

C_2 is the constant of the calibrated viscometer;

t_2 is the flow time to the nearest 0,2 s in the calibrated viscometer.

6 KINEMATIC VISCOSITY CALCULATION

6.1 Basic formula

Kinematic viscosity, expressed in square millimetres per second, can be calculated from the viscometer dimensions as follows :

$$\nu = \frac{100\,\pi\,g\,D^4\,h\,t}{128\,V\,l} - \frac{E}{t^2} \qquad \ldots (1)$$

where

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

g is the acceleration due to gravity, in centimetres per second squared;

D is the diameter of the capillary, in centimetres;

l is the length of the capillary, in centimetres;

h is the average vertical distance between the upper and lower menisci, in centimetres;

V is the timed volume of liquids passing through the capillary, in cubic centimetres (approximately the volume of the timing bulb);

E is the kinetic energy factor, in square millimetre seconds;

t is the flow time, in seconds.

If the viscometer is selected so that the minimum flow time shown in the tables of annexes A, B, and C are exceeded, the kinetic energy term, E/t^2, becomes insignificant and equation(1) may be simplified be grouping the non-variable terms into a constant, C, as follows :

$$\nu = C \cdot t \qquad \ldots (2)$$

6.2 Kinetic energy correction

The viscometers described in annexes A, B, and 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 viscometer for the measurement of low-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 set out as footnotes to the appropriate tables of viscometer dimensions given in annexes A, B and C.

For viscometers whose constants are 0,05 mm^2/s^2 or less, the kinetic energy correction may be significant if the minimum 200 s flow time is not observed.

6.3 Maximum flow time

The limit of 1 000 s has been set arbitrarily as the maximum flow time for the viscometers covered by this International Standard.

6.4 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 changed C constant, C_2, is given approximately as follows :

$$C_2 = C_1 \left[1 + \frac{2}{g\,h}\left(\frac{1}{r_u} - \frac{1}{r_l}\right)\left(\frac{\alpha_1}{\rho_1} - \frac{\alpha_2}{\rho_2}\right)\right] \qquad \ldots (3)$$

where

g is the acceleration due to gravity, in centimetres per second squared;

h is the average driving head, in centimetres;

r_u is the average radius of the upper meniscus, in centimetres;

r_l is the average radius of the lower meniscus, in centimetres;

α is the surface tension, in millinewtons per metre (dynes per centimetre);

ρ is the density, in grams per cubic centimetre.

Subscripts 1 and 2 relate to values with the calibrating liquid and with 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.

6.5 Effect of temperature

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

6.5.1 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 semi-micro, and Cannon-Fenske opaque.

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6.5.2 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 semi-micro viscometers :

$$C_2 = C_1 \left[1 + \frac{4\,V\,(\rho_2 - \rho_1)}{\pi\,D^2\,h\,\rho_2} \right] \qquad \ldots (4)$$

where

C_1 is the constant of the viscometer when filled and calibrated at the same temperature;

V is the volume of charge, in cubic centimetres;

D is the average diameter of the meniscus in the lower reservoir for the Cannon-Fenske routine, Pinkevitch and Cannon-Manning semi-micro viscometers, and in the upper reservoir of the Cannon-Fenske opaque viscometer, in centimetres;

h is the average driving head, in centimetres;

ρ_1 is the density of the test liquid at the filling temperature, in grams per cubic centimetre;

ρ_2 is the density of the test liquid at the test temperature, in grams per cubic centimetre.

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

$$C_2 = C_1 \left[1 - \frac{4\,V\,(\rho_2 - \rho_1)}{\pi\,D^2\,h\,\rho_2} \right] \qquad \ldots (5)$$

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ANNEX A

MODIFIED OSTWALD VISCOMETERS

A.1 GENERAL

The following viscometers of the modified Ostwald type for transparent liquids follow the basic design of the Ostwald viscometer, but are modified to ensure a constant volume test portion in the viscometer :

a) **at the filling temperature :**

Cannon-Fenske routine viscometer

Cannon-Manning semi-micro viscometer

Pinkevitch viscometer

b) **at the test temperature :**

Zeitfuchs viscometer

SIL viscometer

BS/U-tube viscometer

BS/U miniature viscometer

The above viscometers are used for the measurement of the kinematic viscosity of transparent Newtonian liquids up to 20 000 mm^2/s.

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A.2 APPARATUS

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For the modified Ostwald viscometers, detailed drawings, size designations, nominal viscometer constants, kinematic viscosity range, capillary diameter and bulb volumes for each viscometer are shown in figures 1 to 7.

A.3 OPERATING INSTRUCTIONS

A.3.1 A standard operating procedure applicable to all glass capillary kinematic viscometers is contained in ISO 3104. Operating instructions for the modified Ostwald viscometers are outlined in A.3.2 to A.3.8 with emphasis on procedures that are specific to this group of viscometers.

A.3.2 Select a clean, dry calibrated viscometer which will give a flow-time greater than 200 s or the minimum shown in the table of dimensions, whichever is the greater.

A.3.3 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 is thought to contain fibres or solid particles, filter through a 75 μm screen during charging.

A.3.3.1 To charge the Cannon-Fenske routine, Cannon-Manning semi-micro, and Pinkevitch viscometers, invert the viscometer and apply suction to tube L (the Pinkevitch viscometer has a side arm O to which vacuum is applied, with the finger on tube L being used to control the liquid flow) with the tube N immersed in the liquid sample. Draw the sample to timing mark F for the Cannon-Fenske routine and Pinkevitch viscometers and to filling mark G for the Cannon-Manning semi-micro viscometer. Mount the viscometer upright in the constant-temperature bath, keeping tube L vertical.

A.3.3.2 Mount the Zeitfuchs viscometer in the constant-temperature bath, keeping tube L vertical. Pour the sample through tube L to filling mark G. Allow 15 min for the sample to reach the bath temperature and become free of air bubbles. Attach the vacuum line with stopcock and trap to tube K. Slowly draw the sample into timing bulb C by partially opening the stopcock in the vacuum line and partially closing tube N with the finger. Allow the excess liquid to flow into bulb D and through tube K into the trap in the vacuum line. When the liquid in tube L reaches a point 2 to 5 mm above filling mark H, hold it at this point by alternately closing and opening tube N to the atmosphere with the finger for the time shown below to permit the sample to drain from the walls of tube L.

TABLE 2 — **Drainage time for various kinematic viscosity ranges**

Kinematic viscosity of sample mm^2/s	Drainage time s
Under 10	10 to 20
10 to 100	40 to 60
100 to 1 000	100 to 120
Over 1 000	180 to 200

Adjust the working volume by drawing the meniscus at the bottom of the column of liquid exactly to filling mark H, making sure that the sample completely fills the viscometer between filling mark H and the tip of the over-flow in bulb D; after this final adjustment of the working volume, remove the finger and close or remove the connection of the vacuum source. The final adjustment may be more conveniently made by disconnecting the vacuum and applying pressure to the mounting tube L by use of a rubber bulb.

A.3.3.3 Charge the SIL viscometer by tilting it about 30° from the vertical, with bulb A below capillary R. Introduce enough of the sample into tube L for bulb A to fill completely and overflow into the gallery. Return the viscometer to the vertical position and mount it in the constant-temperature bath so that tube L is vertical. The quantity of sample charged should be such that the level in the lower reservoir is 3 to 14 mm above opening S. The sample will rise in capillary R somewhat higher than opening S. After the temperature equilibrium has been reached, remove any excess sample from the gallery by suction applied to tube K.

A.3.3.4 Mount the BS/U-tube or BS/U/M miniature viscometer in the constant-temperature bath keeping tube L vertical. Using a long pipette to minimize any wetting of tube L above filling mark G, fill bulb A with a slight excess of the sample. After allowing the sample to attain the bath temperature, adjust the volume of the sample to bring the liquid level within 0,2 mm of filling mark G by withdrawing the sample with a pipette.

A.3.4 Allow the viscometer to remain in the constant-temperature bath a sufficient time to ensure that the sample reaches temperature equilibrium (for liquids of low kinematic viscosity 10 min at 40 °C, 15 min at 100 °C, or 20 min at 135 °C).

A.3.5 Use vacuum (or pressure if the sample contains volatile constituents) to draw the sample through bulb C to about 5 mm above the upper timing mark E. Release the vacuum, and allow the sample to flow by gravity.

A.3.6 Measure, to the nearest 0,2 s, the time required for the leading edge of the meniscus to pass from timing mark E to timing mark F. If this flow time is less than the minimum flow time specified for the viscometer, select a viscometer with a smaller diameter capillary and repeat steps A.3.3 to A.3.6.

A.3.7 Repeat steps A.3.5 and A.3.6 making a duplicate measurement of flow time. If the two measurements agree within 0,2 %, use the average for calculating kinematic viscosity.

A.3.8 Clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by rinsing with a completely volatile solvent. Dry the viscometer by passing a slow stream of filtered, dry air through the viscometer for 2 min, or until the last trace of solvent is removed.

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Dimensions in millimetres

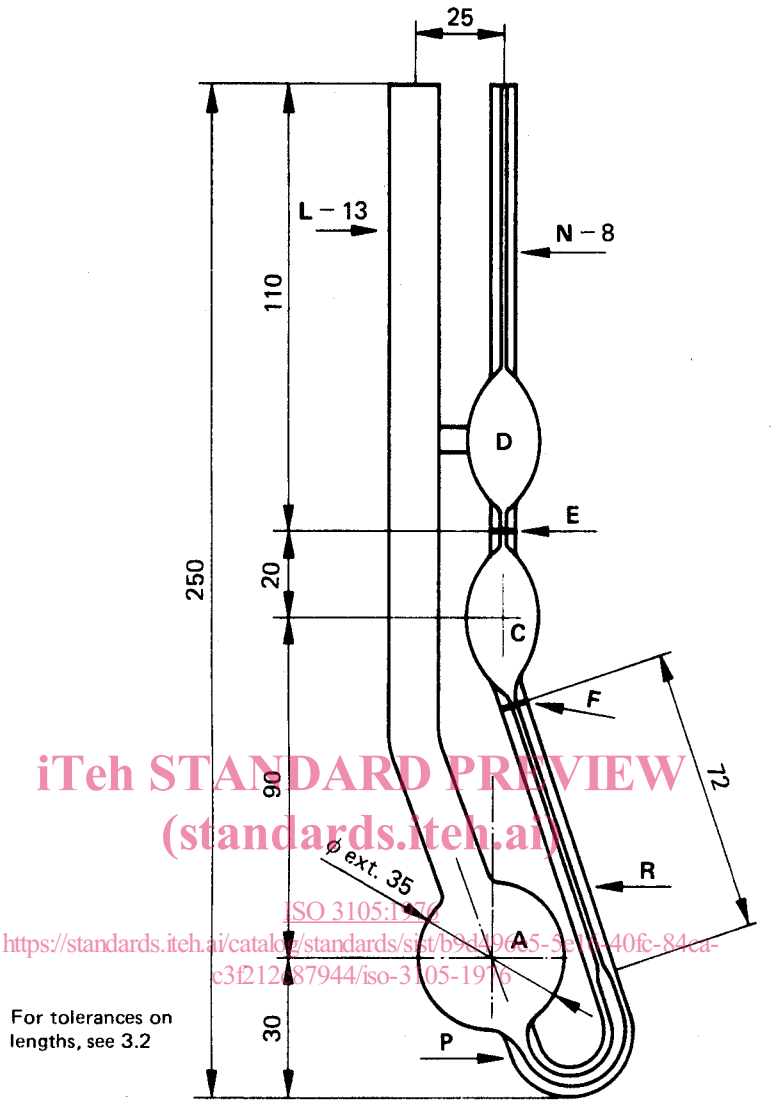

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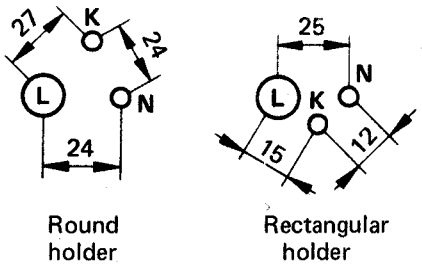
FIGURE 1 – **Cannon-Fenske routine viscometer**

TABLE 3 – **Dimensions and kinematic viscosity ranges**

Size No.	Nominal viscometer constant $(mm^2/s)/s$	Kinematic viscosity range mm^2/s	Inside diameter of tube R mm (± 2 %)	Inside diameter of tubes N, E and P mm	Bulb volume ml (± 5 %)	
					D	C
25	0,002	0,5* to 2	0,30	2,6 to 3,0	3,1	1,6
50	0,004	0,8 to 4	0,44	2,6 to 3,0	3,1	3,1
75	0,008	1,6 to 8	0,54	2,6 to 3,2	3,1	3,1
100	0,015	3 to 15	0,63	2,8 to 3,6	3,1	3,1
150	0,035	7 to 35	0,78	2,8 to 3,6	3,1	3,1
200	0,1	20 to 100	1,01	2,8 to 3,6	3,1	3,1
300	0,25	50 to 250	1,27	2,8 to 3,6	3,1	3,1
350	0,5	100 to 500	1,52	3,0 to 3,8	3,1	3,1
400	1,2	240 to 1 200	1,92	3,0 to 3,8	3,1	3,1
450	2,5	500 to 2 500	2,35	3,5 to 4,2	3,1	3,1
500	8	1 600 to 8 000	3,20	3,7 to 4,2	3,1	3,1
600	20	4 000 to 20 000	4,20	4,4 to 5,0	4,3	3,1

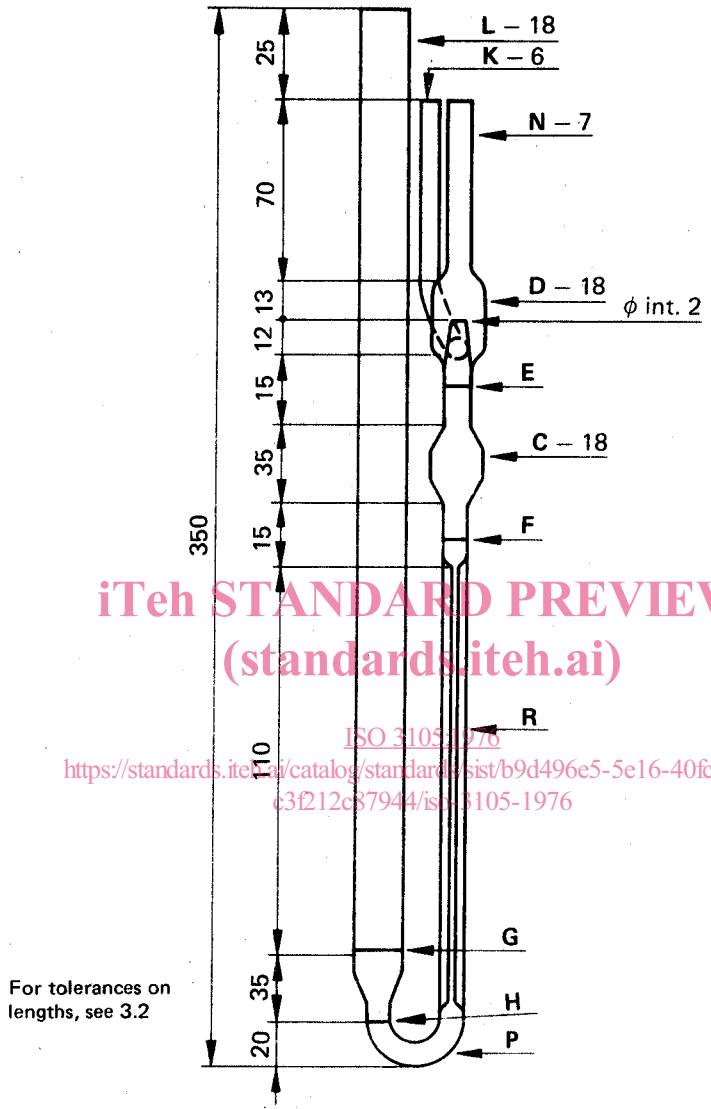
* 250 s minimum flow time; 200 s minimum flow time for all other sizes

Dimensions in millimetres



Round holder

Rectangular holder



For tolerances on lengths, see 3.2

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FIGURE 2 – **Zeitfuchs viscometer**

TABLE 4 – **Dimensions and kinematic viscosity ranges**

Size No.	Nominal viscometer constant (mm²/s)/s	Kinematic viscosity range* mm²/s	Inside diameter of tube R mm (± 2 %)	Inside diameter of tubes P, E and F mm	Volume bulb C ml (± 5 %)
1	0,003	0,6 to 3	0,42	3,8 to 4,2	3,0
2	0,01	2 to 10	0,59	3,8 to 4,2	4,0
3	0,03	6 to 30	0,78	3,8 to 4,2	4,0
4	0,1	20 to 100	1,16	3,8 to 4,2	5,0
5	0,3	60 to 300	1,54	3,8 to 4,2	5,0
6	1,0	200 to 1 000	2,08	3,8 to 4,2	5,0
7	3,0	600 to 3 000	2,76	3,8 to 4,2	5,0

* 200 s minimum flow time for all sizes

Dimensions in millimetres

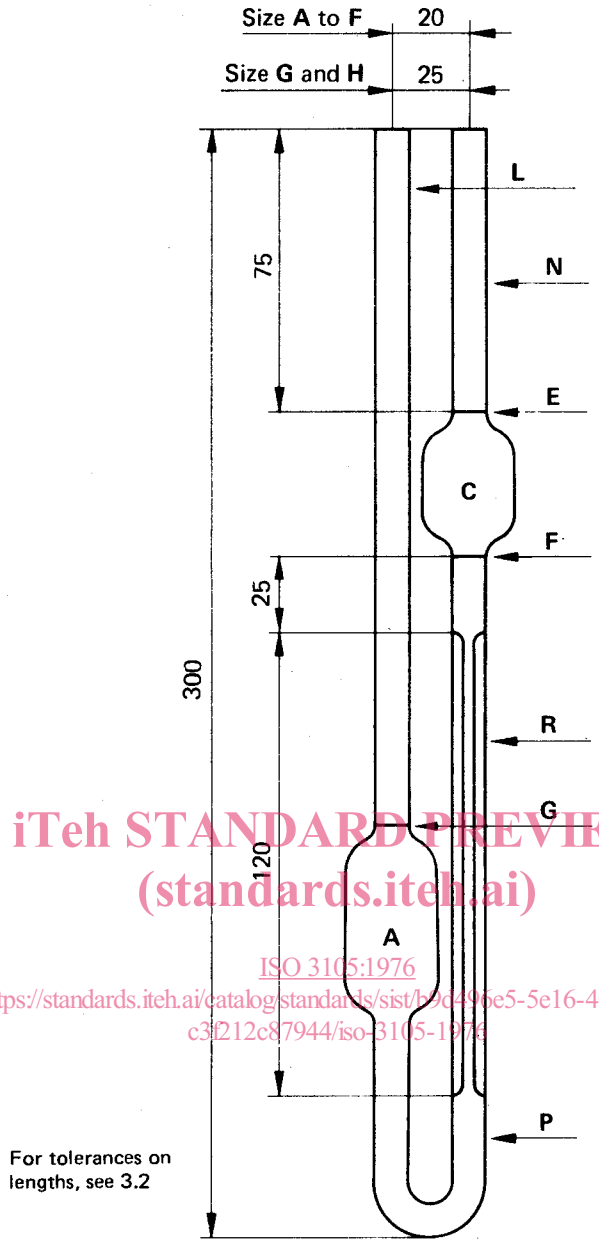

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FIGURE 3 – **BS/U-tube viscometer**

TABLE 5 – **Dimensions and kinematic viscosity ranges**

Size No.	Nominal viscometer constant	Kinematic viscosity range	Inside diameter of tube R	Outside diameter of tubes**		Volume bulb C	Vertical distance F to G	Outside diameter of bulbs A and C
				L and P	N			
	$(mm^2/s)/s$	mm^2/s	mm (± 2 %)	mm	mm	ml (± 5 %)	mm	mm
A	0,003	0,9* to 3	0,50	8 to 9	6 to 7	5,0	91 ± 4	21 to 23
B	0,01	2,0 to 10	0,71	8 to 9	6 to 7	5,0	87 ± 4	21 to 23
C	0,03	6 to 30	0,88	8 to 9	6 to 7	5,0	83 ± 4	21 to 23
D	0,1	20 to 100	1,40	9 to 10	7 to 8	10,0	78 ± 4	25 to 27
E	0,3	60 to 300	2,00	9 to 10	7 to 8	10,0	73 ± 4	25 to 27
F	1,0	200 to 1 000	2,50	9 to 10	7 to 8	10,0	70 ± 4	25 to 27
G	3,0	600 to 3 000	4,00	10 to 11	9 to 10	20,0	60 ± 3	32 to 35
H	10,0	2 000 to 10 000	6,10	10 to 11	9 to 10	20,0	50 ± 3	32 to 35

* 300 s minimum flow time; 200 s minimum flow time for all other sizes.

** Use 1 to 1,25 mm wall tubing for N, P, and L.

Dimensions in millimetres

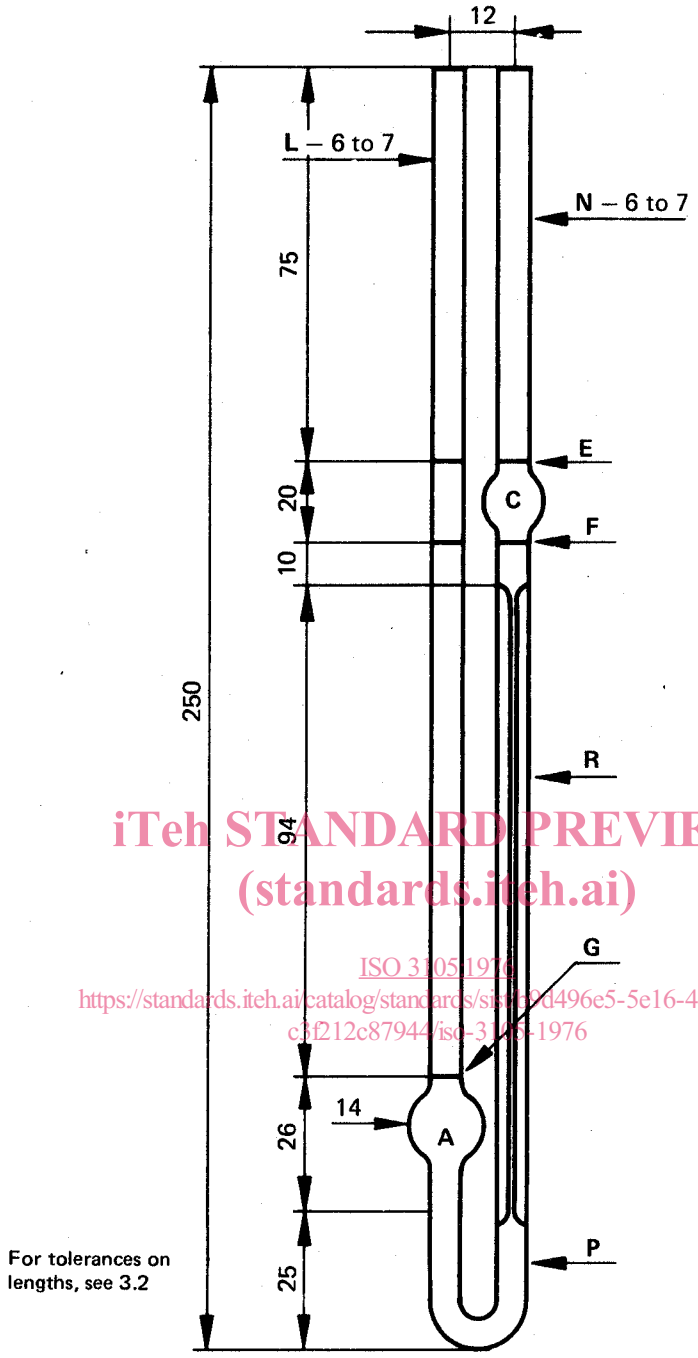

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FIGURE 4 – **BS/U/M miniature viscometer**

TABLE 6 – **Dimensions and kinematic viscosity ranges**

Size No.	Nominal viscometer constant (mm²/s)/s	Kinematic viscosity range* mm²/s	Inside diameter of tube R mm (± 2 %)	Outside diameter of tubes L, N and P** mm	Volume bulb C ml (± 5 %)
M1	0,001	0,2 to 1	0,20	6 to 7	0,50
M2	0,005	1 to 5	0,30	6 to 7	0,50
M3	0,015	3 to 15	0,40	6 to 7	0,50
M4	0,04	8 to 40	0,50	6 to 7	0,50
M5	0,1	20 to 100	0,65	6 to 7	0,50

* 200 s minimum flow time for all sizes.

** Use 1 to 1,25 mm wall tubing for N, P and L.