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Standard Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers[1]

This standard is issued under the fixed designation D446; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 These specifications cover operating instructions for glass capillary kinematic viscometers of all the types described in detail in Annex A1, Annex A2, and Annex A3 as follows:

Modified Ostwald viscometers, Annex A1
Suspended-level viscometers, Annex A2
Reverse-flow viscometers, Annex A3

1.2 The calibration of the viscometers is described in Section 6.

1.3 This standard covers some widely used viscometers suitable for use in accordance with Test Method D445. Other viscometers of the glass capillary type which are capable of measuring kinematic viscosity within the limits of precision given in Test Method D445 may be used.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

2.2 *ISO Documents:*[3]

ISO 3104 Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity

ISO 3105 Glass Capillary Kinematic Viscometers—Specifications and Operating Instructions

ISO 5725 Basic Methods for the Determination of Repeatability and Reproducibility of a Standard Measurement Method

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

ISO Guide 25 General Requirements for the Calibration and Testing Laboratories

2.3 *NIST Standards:*[4]

NIST 1297 Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results

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 color, or otherwise made a permanent part of the viscometer. See detailed description of each type of viscometer in Annex A1, Annex A2, and Annex A3.

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 by more than ±15 % from the nominal value.

[1] These specifications and operating instructions are under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and are the direct responsibility of Subcommittee D02.07 on Flow Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

***A Summary of Changes section appears at the end of this standard**

4. Nomenclature for Figures

4.1 The figures in the annexes contain letters to designate specific parts of each viscometer. These letters are also used in the text of the 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 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

5. Viscometer Holder and Alignment

5.1 All viscometers which have the upper meniscus directly above the lower meniscus (Cannon-Fenske routine in Annex A1 and all in Annex A2) 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.

5.1.1 Those viscometers whose upper meniscus is offset from directly above the lower meniscus (all others in Annex A1 and all in Annex A3) shall be mounted in a constant-temperature bath with tube L held within 0.3° of the vertical.

5.2 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.

5.3 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.

6. Calibration of Viscometers

6.1 *Procedures:*

6.1.1 Calibrate the kinematic glass capillary viscometers covered by this standard using the procedures described in Annex A1, Annex A2, and Annex A3.

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 and 7.2) may be less than 0.2 %.

6.2.2 Select a calibrated viscometer of known viscometer constant C_1. 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 capillary 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. See Test Method D2162.

6.2.3 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 Test Method D445.

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

6.2.4 Calculate the viscometer constant C_1 as follows:

$$C_1 = (t_2 \times C_2)/t_1 \quad (1)$$

where:

C_1 = the constant of the viscometer being calibrated,
t_1 = the flow time to the nearest 0.1 s in the viscometer being calibrated,
C_2 = the constant of the calibrated viscometer, and
t_2 = the flow time to the nearest 0.1 s in the calibrated viscometer.

6.2.5 Repeat 6.2.1 – 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 Annex A1 and Annex A2 and less than 0.3 % for those viscometers listed in Annex A3, use the average. If the constants differ by more than this value, repeat the procedure taking care to examine all possible sources of errors.

6.2.5.1 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:

$$C_2 = (g_2/g_1) \times C_1 \quad (2)$$

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

6.3 *Certified Viscosity Reference Standards:*

6.3.1 Certified viscosity reference standards shall be certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. Certified viscosity reference standards shall be traceable to master viscometer procedures described in Practice D2162.

6.3.1.1 The uncertainty of the certified viscosity reference standard shall be stated for each certified value (k=2, 95% confidence). See ISO 5725 or NIST 1297.

6.3.2 Select from Table 1 a certified 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

TABLE 1 Certified Viscosity Reference Standards

Designation	Approximate Kinematic Viscosity, mm ^{2}/s					
	20°C	25°C	40°C	50°C	80°C	100°C
S3	4.6	4.0	2.9	...	...	1.2
S6	11	8.9	5.7	...	...	1.8
S20	44	34	18	...	...	3.9
S60	170	120	54	...	...	7.2
S200	640	450	180	...	...	17
S600	2400	1600	520	280	67	32
S2000	8700	5600	1700	...	...	75
S8000	37 000	23 000	6700	...	...	...
S30000	...	81 000	23 000	11 000	...	...

flow time to the nearest 0.1 s in accordance with Test Method D445 and calculate the viscometer constant, C, as follows:

$$C = \nu/t \quad (3)$$

where:

ν = the kinematic viscosity, mm^2/s, for the certified viscosity reference standard, and
t = the flow time, s.

6.3.3 Repeat with a second certified viscosity reference standard whose flow times are at least 50 % longer than the first certified viscosity reference standard. If the two values of C differ by less than 0.2 % for those viscometers listed in Annex A1 and Annex A2 and less than 0.3 % for those viscometers listed in Annex A3, 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 Constant:*

6.4.1 Report the constant to the nearest 0.1 % of the determined value. This generally means four significant figures from 1×10^N to 6.999×10^N and three significant figures from 7×10^N to 9.99×10^N.

7. Kinematic Viscosity Calculation

7.1 *Basic Formula:*

7.1.1 Kinematic viscosity, expressed in mm^2/s, can be calculated from the viscometer dimensions as follows:

$$\nu = (10^6 \pi g D^4 H t/128\, VL) - E/t^2 \quad (4)$$

where:

ν = the kinematic viscosity, mm^2/s,
g = the acceleration due to gravity, m/s^2,
D = the diameter of the capillary, m,
L = the length of the capillary, m,
H = the average distance between the upper and lower menisci, m,
V = the timed volume of liquids passing through the capillary, m^3 (approximately the volume of the timing bulb),
E = the kinetic energy factor, mm^2·s, and
t = the flow time, s.

7.1.2 If the viscometer is selected so that the minimum flow time shown in the tables of Annex A1, Annex A2, and Annex A3 are exceeded, the kinetic energy term, E/t^2, becomes insignificant and Eq 4 may be simplified by grouping the non-variable terms into a constant, C, as follows:

$$\nu = C \cdot t \quad (5)$$

7.2 *Kinetic Energy Correction:*

7.2.1 The viscometers described in the Annex A1, Annex A2, and Annex A3 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 set out as footnotes to the appropriate tables of viscometer dimensions given in the Annex A1, Annex A2, and Annex A3.

7.2.2 For viscometers whose constants are 0.05 mm^2/s^2 or less, a kinetic energy correction can be significant if the minimum 200 s flow is not observed. Where this is not possible, Eq 5 takes on the following form:

$$\text{kinematic viscosity, mm}^2/\text{s} = Ct - E/t^2 \quad (6)$$

where:

E = kinetic energy factor, mm^2 × s,
C = viscometer constant, mm^2/s^2,
t = flow time, s.

7.2.3 Although the kinetic energy factor, E, is not a constant, it may be approximated by means of the following equation:

$$E = 52.5\ V^{3/2}\ /\ L\ (Cd)^{1/2} \quad (7)$$

where:

(using the units given in Figs. A1.1-A3.4)

V = volume of the timing bulb, mL,
L = capillary working length, mm,
d = capillary working diameter, mm,
C = viscometer constant, mm^2/s^2.

NOTE 1—The kinetic energy factor for certain viscometer designs and flow time use can result in significant kinematic viscosity errors. Determine the effect of the kinetic energy factor for viscometers not described in this specification.

7.3 *Maximum Flow Time:*

7.3.1 The limit of 1000 s has been set arbitrarily for convenience as the recommended maximum flow time for the viscometers covered by this standard. Longer flow times may be used.

7.4 *Surface Tension Correction :*

7.4.1 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