Designation: D 2162 - 86

Standard Test Method for **Basic Calibration of Master Viscometers and Viscosity Oil** Standards¹

This standard is issued under the fixed designation D 2162; 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 (e) indicates an editorial change since the last revision or reapproval.

1. Scope

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1.1 This test method covers the calibration of master viscometers and viscosity oil standards both of which may be used to calibrate routine viscometers as described in Test Method D 445 over the temperature range from 15 to 100°C.

1.2 This 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. For specific precautionary statements see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

- D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 1250 Petroleum Measurement Tables²
- D1480 Test Method for Density and Relative Density (Specific Gravity) of Viscous Materials by Bingham Pycnometer⁴
- D 1590 Test Methods for Surface Tension of Water and Waste Water³
- E 1 Specification for ASTM Thermometers⁴

3. Descriptions of Terms Specific to This Standard

3.1 basic calibration-calibration based on the primary standard, water. Pure water has a kinematic viscosity of 1.0038 cSt (mm²/s) at 20°C.⁵

3.2 master viscometers-glass capillary viscometers with a liquid driving head of at least 40 cm which are specially designed to minimize errors due to surface tension, kinetic energy, and capillary end effects.

3.3 viscosity oil standards-stable Newtonian liquids, the kinematic viscosities of which have been related to the kinematic viscosity of water through the step-up procedure described in this method.

4. Summary of Test Method

4.1 Two or more master viscometers, having calibration constants in the 0.001 to 0.003-cSt/s (mm²/s²) range, are calibrated with water at 20°C. The kinematic viscosities of two, or more, oil standards are measured at 40°C in these two master viscometers. Corrections are made for buoyancy and, where necessary, for temperature and surface tension.

4.2 A third master viscometer, with a calibration constant of 0.003 to 0.009 cSt/s (mm²/s²), is then calibrated at 40°C with the two standard oils and its calibration factor calculated at standard conditions for water at 20°C. In like manner additional viscosity oil standards and additional master viscometers are calibrated at 40°C using the average results from at least two master viscometers or two oil standards as the case may be. Steps between successive calibration constants or viscosities increase by a factor of three or less until the desired viscosity range is covered.

4.3 Oils are calibrated at other temperatures using the average result from at least two master viscometers.

5. Significance and Use

5.1 In order to determine the kinematic viscosity of liquids whose kinematic viscosity and surface tensions, or both, are significantly different from the primary kinematic viscosity standard, water at 20°C, special long-capillary master viscometers are needed. This test method describes the special step-up procedure for the calibration of master viscometers and kinematic viscosity oil standards.

6. Apparatus

6.1 Master Viscometers: Cannon⁶or Ubbelohde⁷ Type---Acceptable viscometers are shown in Figs. 1 and 2. Two masters are required with calibration constants in the 0.001 to 0.003-cSt/s (mm²/s²) range. Additional masters have factors increasing in three-fold steps.

6.2 Thermometers-Kinematic viscosity thermometers having a range from 18.5 to 21.5°C, or 38.5 to 41.5°C, and conforming to the requirements for Thermometers 44C and 120C, as prescribed in Specification E 1, and calibrated to 0.005°C by the National Bureau of Standards or other qualified agency. A standard platinum resistance thermometer together with a Mueller resistance bridge having equivalent or better accuracy is preferable, where available. Other Thermometers 46C, 121C, etc. as required for standardizing

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Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 11.01.

⁴ Annual Book of ASTM Standards, Vol 14.03.

⁵ Swindells, J. F., Coe, Jr., J. R., and Godfrey, T. B., "Absolute Viscosity of Water at 20°C," Journal of Research, National Bureau Standards, Vol 48, No. 1, January, 1952, p. 1 (RP 2279).

^{*} Cannon, M. R., "Viscosity Measurement, Master Viscometers," Industrial and Engineering Chemistry: Analytical Edition, Vol 16, 1944, p. 708. ⁷ Ubbelohde, L., "The Suspended Lever Viscometer," Journal Institute Petro-

leum Technologists (London), Vol 22, 1936. p. 37.

oil viscosities at other temperatures may be used.

6.3 Bath—A thermostated bath containing water or other transparent liquid deep enough to immerse the master viscometers so that the upper fiducial mark is at least 50 mm below the surface. The efficiency of stirring and the balance between heat loss and input must be such that the temperature of the water does not vary by more than $\pm 0.01^{\circ}$ C over the length of the viscometer or from one viscometer position to another. The working section of the bath should be shielded from direct radiation from heaters and lights. A standard platinum resistance thermometer, approximately 450 mm in length, may be used to ensure that the variation in temperature does not exceed $\pm 0.01^{\circ}$ C. Firm supports should be provided to hold the master viscometer in a rigid and reproducible position within 0° 15 min of vertical.

6.4 Timer—Either a spring-wound or electric timer capable of measuring time intervals of 300 to 10,000 s with an accuracy of ± 0.03 %. The stop watch, fully but not tightly wound, must be used and tested in the same position. For example, if used at 45° inclination, it should have been tested previously in that position. Electric timers must be operated on circuits, the frequencies of which are controlled. Commercial power sources, the frequencies of which are intermittently and not continuously controlled, are not satisfactory. Both mechanical and electric timers may be sensitive to abnormally low ambient temperature and should not be used when cold.

NOTE 1—Time signals as broadcast by the National Bureau of Standards, Station WWV, at 2.5, 5, 10, 15, 20, 25, 30 and 35 MHz are a convenient and primary standard reference for calibrating timing devices. The signals are broadcast 24 h daily. Station CHU from Ottawa, Canada, at 3.330, 7.335, and 14.670 MHz or Station MSF at Rugby, United Kingdom, at 2.5, 5 and 10 MHz may be received better in some locations.

7. Reagents

7.1 Acetone, reagent grade.

NOTE 2-Warning-Extremely flammable.

7.2 Chromic Acid Cleaning Solution—Carefully pour 1 L of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) into 35 mL of a saturated solution of technical grade sodium dichromate ($Na_2Cr_2O_7$) in water.

NOTE 3-Warning-Causes severe burns.

7.3 Petroleum Ether, technical grade.

NOTE 4-Warning-Combustible. Vapor harmful.

7.4 Primary Standard Water, deionized or distilled, then distilled fresh the same day of use. Store in a glass-stoppered chromic acid-cleaned bottle of borosilicate glass.

7.5 Viscosity Oil Standards—Stable petroleum oils selected to cover the desired kinematic viscosity range. They should be transparent and have vapor pressures below 10 mmHg at 40°C. Store away from heat and light in suitable containers, preferably glass.

8. Calibration of Master Viscometers with Water at 20°C

8.1 Maintain the water bath at $20 \pm 0.01^{\circ}$ C applying the necessary thermometer corrections. Two check thermometers are recommended to detect any change in calibration.

8.2 Clean a master viscometer having a calibration constant of 0.001 to 0.003 cSt/s (mm²/s²) with chromic acid cleaning solution to remove organic deposits, rinse tho oughly with distilled water and acetone and dry with stream of filtered air.

8.3 Clean a 50-mL Erlenmeyer flask with cleaning solution and rinse thoroughly with distilled water. Add freshl distilled primary standard water and bring to a boil t remove dissolved gases. Cover the flask to prevent entrance of dust and allow to cool. If a Cannon master viscometer being calibrated, cool the water to $20 \pm 3^{\circ}$ C.

8.4 Charge the Cannon master as follows:

8.4.1 Connect a rubber tube to large arm J. Invert th viscometer and dip the end of small arm A into the beaker c water. Apply suction to arm J and draw liquid through bult B and D to etched line E. Turn the viscometer back to it normal vertical position, wipe tube A clean, insert the viscometer into a holder and place it in the constant temperature bath.

8.4.2 Support the master viscometer firmly and align it in a vertical position with the aid of a plumb bob with a length of about 560 mm. One can be made by use of a cork to fit the large tube J, a piece of thread, and an 18-mm length o 3-mm diameter solder lead. Remove the cork (or slot it during the test so that no back pressure is developed.

8.5 Charge the Ubbelohde master as follows:

8.5.1 Tilt the instrument about 30° from the vertical with bulb A below the capillary and then introduce enough water into large arm 1 to bring the level up to the lower filling line. The level should not be above the upper filling line when the viscometer is returned to the vertical position and the liquid has been drained from tube 1. Charge the viscometer in such a manner that the U-tube at the bottom fills completely without trapping air.

8.5.2 Place the viscometer into a holder and place it in the constant temperature bath. Align the large tube 1 in a vertical position with a plumb bob as described for the Cannon master.

8.6 Allow the charged viscometer to stand in the bath long enough for the sample to reach bath temperature. Fifteen minutes are usually sufficient.

8.7 With gentle vacuum or pressure, force the liquid about 5 mm above the upper timing mark. Avoid splashing liquid in the upper bulb or forming any bubbles in the liquid. When using a Ubbelohde viscometer, hold a finger over the upper end of tube 3 during this operation; then remove it and immediately place it over tube 2 until the liquid drops away from the lower end of the capillary.

8.8 Measure the efflux of water from upper to lower timing mark (which should be 300 s or more) to the nearest 0.1 s. Repeat this measurement two additional times and average if the lowest and highest times agree within 0.1 %. If the measurements do not agree within this tolerance, repeat the procedure paying particular attention to cleaning the viscometer, filtering the sample, avoiding contamination during filling and afterwards, checking the temperature control and the timing device.

8.9 Clean the viscometer and dry with filtered air. Reload the viscometer and measure the efflux time in triplicate. Average if the lowest and highest times agree within 0.1 %.

8.10 If the two average times fall within 0.1 %, average the two to obtain an average of the two fills.

8.11 Calculate the viscometer constant as follows:

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c = the calibration constant of the viscometer with water at $20^{\circ}C$, cSt/s (mm²/s²), and

= efflux time, s.

8.12 Repeat the operations described in 8.1 to 8.8 with a second master viscometer having a constant not greater than $0.003 \text{ cSt/s}^2 (\text{mm}^2/\text{s}^2).$

NOTE 5-Normally master viscometers are calibrated and used in the same location. If subsequently the viscometer is used at a laboratory other than the calibrating one, the c constant should be corrected for the difference in the acceleration of gravity, g at the two locations as follows:

$$c_2 = (g_2/g_1) \times c_1$$

where:

 c_2 and g_2 = the calibration constant and gravity at the new locations, and

 c_1 and g_1 = the calibration constant and gravity at the original location.

Certificates for viscometers should state the value of g at the location of the calibrating laboratory. Failure to correct a viscometer constant for change of gravity can result in errors as high as 0.2 %, which is twice the error permitted between checks in this test method.

9. Calibration of Viscosity Oil Standards at 40°C

9.1 Choose an oil with a kinematic viscosity of about 3 cSt/s (mm²/s) at 40°C. Filter a portion into a clean beaker through a 200-mesh (75-µm) sieve, or other suitable filter, to remove dirt or sediment. If necessary, adjust the temperature of the oil to within 3°C of 20°C.

9.2 Adjust the temperature of the bath to $40 \pm 0.01^{\circ}C$ applying the necessary thermometer corrections.

NOTE 6-The procedure is given for 40°C because kinematic viscosities of petroleum oils are traditionally measured at this temperature and so viscosity oil standards at 40°C are in common use. Master viscometers may, however, be calibrated at any convenient bath temperature and used, with any necessary corrections, at other temperatures.

9.3 Charge one of the master viscometers, already calibrated with water, with the oil. Allow the charged viscometer to stand in the bath long enough to reach bath temperature.

9.4 Measure the efflux time to the nearest 0.1 s three times and average if the lowest and highest times agree within 0.1 %.

9.5 Clean the viscometer with petroleum ether followed by acetone and dry with filtered air. Reload the viscometer with oil and measure the efflux time in triplicate. Average if the lowest and highest times agree within 0.1 %.

9.6 If the average times fall within 0.1 %, average the two.

9.7 Using the second calibrated master viscometer, repeat the procedure described in 9.1 to 9.6 on the same oil.

10. Corrections and Calculation of Kinematic Viscosity at 40°C

10.1 Buoyancy Correction (Applicable to both Cannon and Ubbelohde Viscometers):

10.1.1 In kinematic viscometers the driving head of liquid is slightly reduced by the counterpoised head of air in the empty arm of the viscometer. This buoyancy effect is measured by the density of air divided by the density of the liquid. In going from water at 20°C to oil at another temperature, the net effect is the difference between the two as follows:

$$c_b = (d_{a20}/d_{w20}) - (d_{aT}/d_{oT})$$

TABLE 1 Density of Moist Air^A

Temperature °C	Density, g/mL
-54	0.00162
-40	0.00150
0	0.00128
20	0.00120
25	0.00118
40	0.00113
60	0.00105
100	0.00094

AThese densities assume an atmospheric pressure of 760 mm and, at 20°C and above, a dew point of 150°C (the mean of an anticipated 5 to 25°C).

where:

= buoyancy correction expressed as a fraction of the Сь driving head.

 $d_{a20} =$ density of moist air at 20°C (0.00120 g/mL), $d_w^{20} =$ density of water at 20°C (0.998 g/mL),

 d_{aT} = density of moist air at the test temperature in g/mL (see Table 1), and

 d_{oT} = density of oil in g/mL at the test temperature.

10.1.2 Determine the density of the oil standard at 40°C in accordance with Test Method D 1480.8 Alternatively, determine the specific gravity of the oil at some convenient temperature and obtain the specific gravity at 40°C in accordance with Standard D 1250. Specific gravity is close enough to density for the purposes of this correction.

10.1.3 Calculate the buoyancy correction by the equation given in 10.1.1, inserting the determined oil density at 40°C and the density of air at 40°C from Table 1. For most petroleum oils the approximate buoyancy correction is -0.0002 when correcting from water at 20°C to oil at 40°C.

10.2 Temperature Correction (Necessary only with Cannon-type viscometer and only when test temperature differs from the calibration temperature, 20 °C):

10.2.1 The effects of thermal expansion of glass capillarytube viscometers of the kinematic type are virtually selfcompensating and usually may be neglected. However, in viscometers in which the working volume is fixed at 20 ± 3°C but which are used at another temperature, the driving head changes with expansion or contraction of the liquid. The correction is given as follows:

$$c_T = V(d_T - d_{20})/\pi R^2 h d_T$$

where:

 c_T = temperature correction expressed as a fraction of the driving head,

V = the total volume of fill at 20°C, in mL,

 d_{20} = the density of the liquid when filled at 20°C in g/mL,

 d_T = the density of the liquid at the test temperature in g/mL,

R = inside radius of the reservoir in centimetres, and

h = driving head in centimetres at 20°C.

NOTE 7-The volume of fill, the inside radius of the reservoir and the driving head may be measured approximately since errors in these measurements have only slight effects on the final corrected viscosity. For Cannon masters conforming to the dimensions shown in Fig. 1 the equation reduces to:

$$c_T = 0.023 \times [(d_T - d_{20})/d_T].$$

⁸ Swindells, J. F., Hardy, R. C., and Cottington, R. L., "Precise Measurements with Bingham Viscometers and Cannon Master Viscometers" Journal of Research, National Bureau Standards, Vol 52, No. 3, March, 1954, p. 105 (RP 2479).

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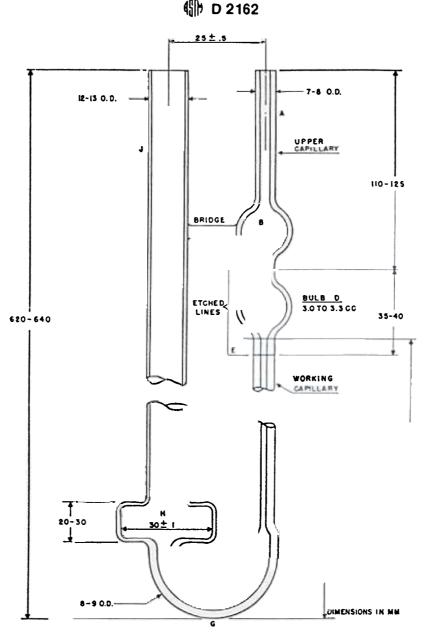


FIG. 1 Cannon Master Viscometer

10.2.2 Determine the density of the oil at 20°C in accordance with Test Method D 1480. Alternatively, determine the specific gravity of the oil at some convenient temperature and obtain the specific gravities at 20°C and 40°F in accordance with Standard D 1250. Specific gravities may be substituted for density in this equation.

10.2.3 Calculate the temperature correction by the equation given in 10.2.1, inserting the density of the oil at 20°C and the density of the oil at 40°C as determined in 10.1 or as calculated in accordance with Standard D 1250. For most petroleum oils the approximate temperature correction is -0.0003 when correcting from 20°C to 40°C.

10.3 Surface Tension Correction (Necessary only with the Cannon-type viscometer, when calibrated with one liquid and used with another liquid having a different surface tension and density):

10.3.1 If the upper liquid meniscus in a viscometer flows in a narrower tube than the bottom meniscus, the driving head is slightly reduced by the capillary rise in the smaller diameter tube or bulb. The surface tension and capillary rise of water are greater than that of oil. Consequently, the driving head is reduced more with water in the viscometer and the driving head is less with water in the viscometer than it is with oil. A correction is required for the difference in capillary rise of water and oil.

10.3.2 Equations have been derived relating capillary rise to surface tension in tubes of small diameter. These equations are not exact when applied to larger tubes. The corrections given in Table 2 are based upon actual measurements of capillary rise with both water and oil in a bulb simulating the Cannon master efflux bulb.⁸ They are empirical corrections to be applied only to Cannon master viscometers having the dimensions shown in Fig. 1. The

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TABLE 2 Surface Tension Correction	TABLE	2	Surface	Tension	Correction
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Surface Tension Divided by Density	Correction		
30	+0.0016		
35	+0.0014		
40	+0.0012		
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efflux bulb must be spherical and have a volume of 3.0 cm³, the lower reservoir must have a diameter of 30 mm and the head must be 470 ± 40 mm.

10.3.3 Determine the surface tension of the oil standard at 40°C in accordance with Test Method D 1590.

10.3.4 Divide surface tension by density at 40°C as determined in 10.1.2. Select the surface tension correction from Table 2. The correction is expressed as a fraction of the driving head.

NOTE 8-In Cannon master viscometers, the approximate surface tension correction for most petroleum oils is +0.0014 when correcting from water at 20°C to oil at 40°C. In Ubbelohde master viscometers the surface tension correction will be nearly zero if the diameter of the meniscus in the vent bulb below the capillary is 75 % that of the meniscus in the efflux bulb at the maximum diameter. If the diameter of the lower meniscus varies from 70 to 80 % of the maximum upper meniscus diameter, the surface tension correction, c_s , will vary from -0.0002 to +0.0002 (-0.02 to +0.02 %); when the viscometer is made as shown in Fig. 2, the surface tension correction should be within these limits. If the diameter of the lower meniscus varies from 60 to 90 % of the maximum upper meniscus diameter, the surface tension correction, c_{s} , will vary from -0.0005 to +0.0005 (-0.05 to +0.05 %).

10.4 Kinematic Viscosity Calculation:

10.4.1 For a Cannon master viscometer, calculate the kinematic viscosity of the oil standard as follows:

$$v = (1 + c_h + c_T + c_s) \times Ct$$

where:

where:

v = kinematic viscosity of the oil at 40°C in cSt/s (mm²/s²),

 c_b = buoyancy correction (see 10.1),

 c_T = temperature correction (see 10.2),

- c_s = surface tension correction (see 10.3), C = calibration constant of master viscometer with water at 20°C, in cSt/s (mm^2/s^2) and

= average efflux time in seconds,

10.4.2 For a Ubbelohde master viscometer, calculate the kinematic viscosity of the oil standard as follows:

$$v = (1 + c_b) \times Ct$$

v, c_h , C, and t are as given in 10.4.1.

NOTE 9—The value of $(1 + c_h + c_T + c_s)$ will average about 1.0009 for the Cannon master and 0.9998 for the Ubbelohde master viscometer. The total correction is therefore of the same order or below the repeatability limit of this method.

10.5 Repeat the calculation for the data obtained in 9.7 with the second master viscometer. Average the kinematic viscosities if they agree within 0.1 %.

11. Calibration of Additional Master Viscometers and Viscosity Oil Standards at 40°C

11.1 Choose an oil with a kinematic viscosity of 6 to 10 cSt (mm²/s) at 40°C. Determine the average kinematic viscosity in the two calibrated master viscometers as directed in Sections 9 and 10.

11.2 Select a new, third master viscometer with a calibration constant in the 0.003 to 0.009 cSt/s (mm²/s²) range. Determine its calibration constant using the 3 and 10 cSt (mm²/s²) kinematic viscosity oil standards following the procedure given in 9.1 to 9.6.

11.3 Calculate the calibration constant as follows:

11.3.1 For Cannon master viscometers:

$$C = v/[(1 + c_b + c_T + c_s) \times t]$$

where:

 C_T = the calibration factor in mm²/s² for water at 20°C

v = the kinematic viscosity of the oil standard in mm²/s,

 c_b = the buoyancy correction (10.1),

 c_T = the temperature correction (10.2),

 c_s = the surface tension correction (10.3), and

= the average efflux time in seconds. 1

11.3.2 For Ubbelohde master viscometers:

$$C = v/[(1+c_b) \times t]$$

where:

 C, v, c_b , and t are as given in 11.3.1.

NOTE 10-For consistency, all viscometer calibration factors are corrected back to water at 20°C. Corrections for other oils and temperatures are made from this standard condition. Calibration factors with oil at 40°C are used in routine calibration work.

11.4 Average the calibration factors found with the two oil standards if they agree within 0.1 %.

11.5 Continue to calibrate viscometers and viscosity oil standards in like manner using two or more viscosity oil standards to determine the factor of each viscometer and two viscometers to determine the kinematic viscosity of each oil standard.

11.6 Periodically clean all master viscometers with chromic acid cleaning solution to remove organic deposits.

12. Calibration of Viscosity Oil Standards at Other Temperatures

12.1 Select a master viscometer having an efflux time between 300 and 3000 s with the oil at the temperature of test.

12.2 Filter a portion of the oil into a clean beaker. When calibrating with Cannon viscometers adjust the temperature to $20 \pm 3^{\circ}C$.

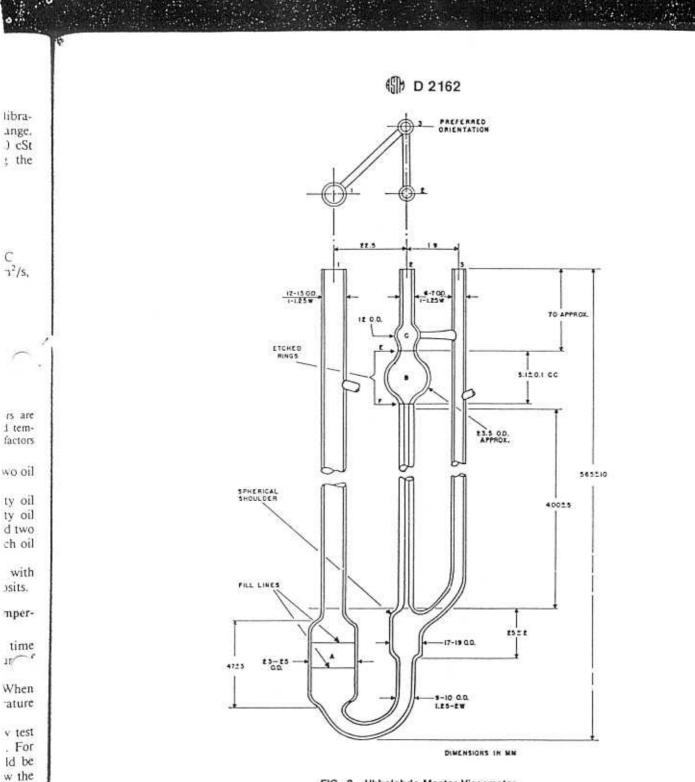
12.3 Adjust the temperature of the bath to the new test temperatures within ± 0.01 °C of the test temperature. For temperatures below 15°C, the bath temperature should be constant within ±0.02°C. When working near or below the dew point, use drying tubes to prevent condensation of moisture in the viscometer.

12.4 Determine efflux time of the oil at the new test temperature as directed in 9.3 to 9.6. Select another master viscometer having an efflux time between 300 and 3000 s and repeat the determination of efflux time.

12.5 Calculate the kinematic viscosity as directed in Section 10, substituting air density, oil density, and surface tension at the new test temperature for the densities and surface tension at 40°C.

13. Precision and Bias

13.1 The precision of this test method as determined by statistical examination of interlaboratory test results is as follows:





13.2 Repeatability—The difference between two 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 this test method, exceed 0.1 % of their mean only in 1 case in 20. 13.3 Reproducibility—The difference between two singland independent test results, obtained by different operator working in different laboratories on identical test materia would, in the long run, in the normal and correct operation of this test method, exceed 0.35 % of their mean only in case in 20.

13.4 Bias—Since there is no accepted reference materia suitable for determining the bias for the procedure in Tes Method D 2162, no statement on bias is being made.

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