

APPENDIX

METHODS OF OBTAINING THE LOW-TEMPERATURE VISCOSITY OF ENGINE OILS AT HIGHER SHEAR RATES AND STRESSES

The following methods supplement the papers in this symposium by describing how the various viscous results were obtained. It should be noted that none of the methods has been evaluated for accuracy or reproducibility.

METHODS OF TEST FOR VISCOSITY OF ENGINE OILS BY PENN STATE SINGLE-PASS CAPILLARY VISCOMETER

Scope

1. Two methods are described for determining the viscosity of engine oils at varying

rates of shear. The apparatus is useful in determining viscosities at reduced temperatures (0 F and below).

METHOD A

Apparatus

2. The PRL single-pass capillary viscometer is designed to fit into most conventional constant-temperature baths. The minimum bath size required is 6 in. in diameter by 10 in. in depth. A dry ice - acetone bath may be used for temperatures below 32 F, a water bath for temperatures of 32 to 130 F, an oil bath for temperatures of 130 to 300 F, and a saturated vapor bath for temperatures above 300 F.

Cleaning Procedure

3. (a) Drain the viscometer, disassemble the parts and then scrub them with a suitable solvent. The choice of the solvent depends on the nature of the test fluid. Following the solvent wash, clean the parts with petroleum ether to remove any solvent residue. Then air dry the parts and place them in a convection oven at 200 F for an additional 1-hr drying period.

(b) The capillary requires a special cleaning technique. Pull solvent through the capillary into a suction flask using a water aspirator as the source of vacuum. Use about 200 ml of solvent to back-flush the capillary followed by 50 ml of petroleum ether. Then

dry the capillary and place it in the convection oven for the final drying.

(c) Additional precautions must be taken with the test fluid samples to protect the capillaries from foreign material which may partially plug the capillary. Filter all test fluid samples through a medium-porosity sintered-glass filter at room temperature to remove insoluble foreign material. As an additional check on capillary condition, check each capillary unit periodically with a mineral oil calibrating fluid. A typical schedule calls for a calibration run after every 10 experimental tests with unknown fluids. Periodic calibrations conducted over a span of several years show no measurable changes in the capillary flow characteristics with use.

Viscometer Assembly

4. (a) Select the desired capillary, depending upon the viscosity of the fluid to be tested, using the relationship shown in Fig 14.¹ Attach the capillary to the reservoir and

¹ Figures referred to in this method are those in "Development and Application of the PRL Single-Pass Capillary Viscometer" by E. Erwin Klaus, Francis M. Angeloni, and Merrell R. Fenske, see p. 28.

fill the reservoir with a measured quantity of test fluid and insert the stirrer. Put the reservoir cap with the rubber O-ring in place on the reservoir and turn it to the end of the thread, then back off one-eighth turn. Use a few drops of the test fluid on the shoulder of the pot as a lubricant for the O-ring. Then place an efflux bulb of appropriate volume on the capillary housing and fasten it by means of two brass springs to act as a positive seal for the ground glass joint. Then place the entire viscometer assembly in the constant-temperature bath as shown in Fig. 1.¹ Connect the stirrer shaft to a stirrer motor (30 rpm) with a removable sleeve. Start the stirrer and allow the temperature to come to the bath temperature. Approximately 1 hr is required for temperature equilibrium to be established between the test fluid and the bath in the case of viscous samples. After equilibrium is achieved, disconnect the stirrer motor and connect the pressure inlet line from the ballast system to the fluid reservoir by means of the stainless steel tubing fitting shown in Fig. 2.¹

(b) Adjust the pressure in the ballast system to the desired level at least 15 min before the start of a test. The pressure may be adjusted and the temperature of the ballast system allowed to come to equilibrium while the fluid sample is being stirred.

Procedure

5. (a) When the ballast system has reached a constant pressure value and the fluid sample the desired temperature, pressurize

the fluid reservoir by opening valve 2, Fig. 1.¹ This pressurizes the fluid reservoir and causes the test fluid to flow through the capillary. Measure the time necessary to fill the efflux bulb with test fluid from the lower to the upper etched line with a calibrated stop watch reading in 0.1-sec increments. After the fluid reaches the top etched line, vent the pressure to the viscometer by closing valve 2 and opening valve 1, Fig. 1.¹ Record the pressure and time for the test. The viscometer must be vented promptly following each determination to prevent excessive flow of fluid from the reservoir which will result in overflow from the efflux bulb into the constant-temperature bath.

(b) The efflux bulb of the viscometer unit is then changed to allow a second viscosity determination to be made under a new set of shear conditions. Raise the entire viscometer assembly in the constant-temperature bath until the ground-glass joint of the efflux bulb is above the liquid level. Dry the joint and remove the springs. Then remove the efflux bulb from the system. Sealing the top of the efflux bulb with a rubber suction bulb or a finger will prevent fluid spilling into the constant-temperature bath. Attach a clean efflux bulb with the brass springs and lower the entire unit into the bath. Readjust the ballast pressure system for the second test and allow it to stand for 15 min to come to equilibrium. Determine the viscosity by the procedure described in Paragraph (a). Using this same technique a third, fourth, and fifth determination may be made from one charge to the reservoir.

METHOD B

Summary of Method

6. The sample of oil is contained in a reservoir the cover of which includes a capillary holder and a pressure inlet. The reservoir should be capable of withstanding operating pressures to 300 psi, min. The oil sample is put in the reservoir and brought to equilibrium temperature in a constant-temperature bath. Following this, nitrogen under pressure is introduced into the reservoir, forcing the oil through a capillary into an efflux bulb of calibrated volume. Viscosity and rate of shear are determined through calculations

involving the efflux time, the pressure applied, and the dimensions of the capillary.

Apparatus

7. (a) *Reservoir*.—The reservoir is a cylindrical brass vessel with walls approximately $\frac{1}{4}$ in. thick having a capacity of approximately 85 ml. The cover of the reservoir is secured by means of 6 machine screws. The cover is sealed to the reservoir with an O-ring seal. The reservoir cover contains a capillary holder and a fitting for introducing gas under pressure.

(b) *Capillary*.—Special Pyrex glass capillaries are used. A typical capillary is shown in Fig. 1. The inside diameter of the capillary and the volume of the efflux bulb will vary,

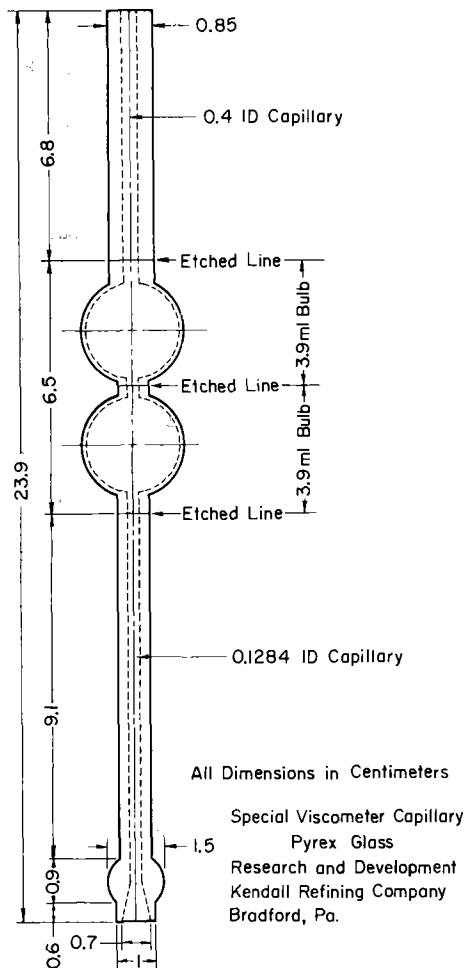


FIG. 1.—Typical Viscometer Capillary for Use with Penn State Single-Pass Capillary Viscometer.

depending upon the viscosity range and shear rate being investigated.

(c) *Constant - Temperature Bath*.—Constant temperature in the reservoir is obtained by immersing the assembled reservoir and capillary in a dry ice - acetone bath. A Dewar flask approximately 145 mm in inside

diameter by 300 mm deep with unsilvered strips on opposite sides will hold an adequate quantity of acetone and will permit observation of the flow of fluid into the efflux bulb. A stirrer is used to circulate the fluid in the cooling bath. The temperature of the bath is determined with a precision thermometer. Temperature is regulated by increasing or decreasing the quantity of dry ice added periodically to the bath.

(d) *Pressure System*.—Pressure to the reservoir is supplied from a cylinder of nitrogen with suitable reducing valves and regulators. A ballast cylinder of the same size as that in which gas is commercially supplied is also attached to the supply line. The pressure indicator used should be readable within ± 0.5 psi. A three-way valve is used to introduce pressure to the reservoir at the start of a viscosity determination and to shut off the pressure and vent the reservoir at the end of the determination.

Procedure

8. (a) Fill the reservoir with approximately 70 ml of the sample. Assemble the reservoir and viscometer capillary.

(b) Place the assembled reservoir in the constant-temperature bath and connect to the pressure system. Maintain the constant-temperature bath at the desired test temperature for a minimum of 45 min.

(c) When equilibrium temperature has been reached, introduce nitrogen under pressure into the reservoir and record the time required to fill the efflux bulbs of the capillary assembly. Turn off the pressure immediately after the fluid reaches the second timing mark to prevent the possibility of blowing nitrogen through the capillary.

Calculation

9. The viscometer capillary assemblies are calibrated using oils of known viscosity characteristics. From these calibration runs and the dimensions of the capillary, constants can be determined which relate the efflux time to the viscosity of the fluid and the shear rate at which the determination was made.

METHOD OF TEST FOR VISCOSITY OF ENGINE OILS BY THE HIGH-RATE-OF-SHEAR ROTATIONAL VISCOMETER

Scope

1. This method describes a procedure for determining the viscosity of engine oils at low temperatures by the high-rate-of-shear rotational viscometer.

Apparatus

2. The viscometer is basically the same as that developed by Barber, Muenger, and Villforth.² Figure 2³ shows the essential features of this apparatus. Concentric cylinders (stator and rotor) with small clearance are separated by a film of test oil (approximately 0.001 in.). The apparatus was designed so that the heat paths, radially inward and outward, from the film are equal. This and the very small clearance minimize any temperature gradient across the oil film. The desired film temperature is obtained by circulating a coolant through the rotor and around the stator and is measured by four thermocouples embedded in the stator with their junctions very close to the test oil film. The rotor is driven by a multispeed drive and the stator is supported by a frictionless hydrostatic support bearing. Shear stress is obtained by measuring the torque transmitted to the stator by the test oil film.

Procedure

3. (a) In determining viscosities, it is necessary to know the precise oil film thickness at the test temperature. For this purpose, the film thickness is calculated by running a Newtonian reference oil of known viscosity at the test temperature. Then by obtaining the slope of the torque versus rpm curve for the Newtonian reference oil, the film thick-

ness can be calculated from the following equation:

$$\mu = \frac{\tau}{R} = \frac{\tau}{\frac{V}{h}}$$

where:

μ = absolute viscosity,

τ = shear stress,

R = rate of shear,

V = velocity of moving surface, and

h = film thickness.

(b) Charge the apparatus with a sample of the test oil and drive the rotor at a speed corresponding to the lowest shear rate desired. Circulate coolant at a temperature low enough to give the desired film temperature. When the proper film temperature is reached and conditions stabilized, record the torque. Then drive the rotor at a higher speed and change the coolant temperature if necessary to maintain the same film temperature, at which time again record the torque. Repeat this process until the highest desired shear rate is obtained. In order to check for any shearing-down of the test oil or loss of oil film, repeat the entire process in reverse, that is, from the high speed down. If the descending viscosity points fall on the curve of the ascending points, no appreciable shearing-down or loss of oil film has occurred.

Calculation

4. Using the film thickness determined in Section 3(a), calculate the viscosity of the non-Newtonian test oil at the same temperature by the formula in Section 3(a).

METHOD OF TEST FOR VISCOSITY OF ENGINE OILS BY THE PRESSURE VISCOMETER (ASTM METHOD D 1092 MODIFIED)

Scope

1. This method describes a procedure for determining the viscosity of engine oils at low temperatures by the pressure viscometer.

² E. M. Barber, J. R. Muenger, and F. J. Villforth, Jr., "A High Rate of Shear Rotational Viscometer," *Analytical Chemistry*, Vol. 27, p. 425, March, 1955.

Summary of Method

2. Viscosity measurement utilizing the pressure viscometer consists of recording the pressure drop through a very accurately

³ This figure is not shown here but appears as Fig. 5 in G. A. Kohl and R. D. Skoglund, "An Investigation of Low-Temperature Viscosity Characteristics of Motor Oils," see p. 123.

calibrated capillary. The test lubricant is forced through the capillary by means of a floating piston actuated by a hydraulic system as shown in Fig. 3.⁴ The volumetric flow through the capillary is established by calibrating the output of the Zenith pump with respect to pressure and pump speed. Poiseuille's equation is used to calculate the viscosity and shear rate.

Apparatus

3. The apparatus used is essentially that specified by ASTM Method D 1092, Test for Apparent Viscosity of Lubricating Greases,⁵ with a minor modification to facilitate oil viscosity determinations. Basically, the modification of the apparatus consists of inverting the cylinder in contrast to the standard setup. This has the beneficial effect of enabling a change in capillaries without loss of test lubricant and the resulting air entrainment. Also, gravity flow does not occur with the cylinder inverted. The apparatus is contained within a temperature environmental chamber.

METHODS OF TEST FOR VISCOSITY OF LUBRICATING OILS BY THE FERRANTI-SHIRLEY VISCOMETER

METHOD A. ESSO RESEARCH AND ENGINEERING CO. PROCEDURE

Scope

1. (a) This method for determining viscosity at 0 F by the Ferranti-Shirley viscometer is applicable to motor oils whose viscosities at 0 F are in the 5W, 10W, or 20W range. It is also applicable to waxy or non-waxy mineral or synthetic oils with or without polymeric or other types of additives.

(b) This procedure is designed to obtain high-shear, steady-state viscosities of lubricating oils at low temperatures for the purpose of relating these viscosities to the cold-crinking behavior of such oils. It is not intended to measure the time-dependent or

Procedure

4. Using the modified apparatus, follow the procedure described in ASTM Method D 1092.⁵

Calculation

5. At very low pressures, corrections are made for the weight of the piston, fluid friction losses in the hydraulic system, and head effects. These corrections are made using the energy equation and the equation for head loss in laminar flow. These corrections become negligible above about 10 psi.

NOTE.—At high shear rates, the pressure viscometer has been observed to give decreasing viscosity with increasing shear rate for Newtonian fluids. This has been attributed to heat dissipation within the capillary from the work input required to force the test fluid through the capillary. Recent work⁶ has indicated that it may be possible to predict the temperature rise, assuming adiabatic flow and a value for the specific heat of the oil.

thixotropic flow behavior of many oils, nor is it intended to relate to lubricating oil pumping rates, ability to be poured, or other low-shear phenomena.

Equipment

2. (a) *Viscometer*.—Ferranti-Shirley low-temperature range, plate-cone viscometer consisting of a measuring unit, a power supply, and a speed control unit.

(b) *Refrigeration Unit*.—A refrigeration unit capable of maintaining a bath of isopropanol or other suitable circulating fluid at 0 ± 0.1 F. The more accurate the temperature control, the better. Associated with this is a pump which circulates the isopropanol through thermally insulated pipes to the plate of the viscometer through which the coolant flows at a rate of 2 gal per min. Also immersed in the isopropanol reservoir is

⁴ This figure is not shown here but appears as Fig. 1 in G. A. Kohl and R. D. Skoglund, "An Investigation of Low-Temperature Viscosity Characteristics of Motor Oils," see p. 123.

⁵ Method of Test for Apparent Viscosity of Lubricating Greases (D 1092-58 T), 1961 Book of ASTM Standards, Part 7, p. 553; see also ASTM Standards on Petroleum Products and Lubricants, Oct. 1961.

⁶ G. A. Kohl and R. D. Skoglund, "An Investigation of Low-Temperature Viscosity Characteristics of Motor Oils," see p. 123.

a cooling coil through which predried nitrogen (or air) is passed through thermally insulated tubing to the cone area.

(c) *Guard*.—A circular guard of rigid transparent plastic for isolating the cone area. It is mounted on two pins in the housing surrounding the cone bearings. The guard is wide enough almost to touch the plate when it is in position. A nozzle in the guard admits the predried, precooled nitrogen, which escapes through the gap between the guard and the plate.

(d) *Recorder*.—A Bristol single pen multi-range temperature recorder for recording the thermocouple outputs. The range usually extends from -20 to $+30$ F and covers the full scale. Three thermocouple leads from the viscometer plate and one each from the refrigeration bath and the air around the cone enter a multiposition switch plate connected to the input of the recorder.

An optional feature is a single pen recorder for recording the torque as a function of time. A special attachment can be made on the back of the speed control unit to bring the torque signal out to the recorder without affecting the torque indicator ordinarily used.

(e) *Enclosure*.—The viscometer measuring unit is enclosed in a box made of clear rigid plastic having a hinged front panel with a friction snap to hold it shut. The nitrogen escapes through holes in the box for thermocouple and power leads.

Procedure

3. (a) *Preparation*.—Install the 1-cm radius cone using the three knurled screws on the cone-holder, always aligning the reference marks. Turn on the refrigeration and circulating unit and allow it to equilibrate at a temperature near 0 F overnight or for several hours prior to the measurements. Keep the dried nitrogen running throughout, to prevent ice formation on the plate. During this time keep the plate about $\frac{1}{16}$ in. below the cone tip with a drop of oil between the two. Turn on the "main" and "motor" switches, the second 5 min after the first.

(b) *Cleaning*.—With the drive gear in neutral, clean the instrument by lowering the plate quickly and wiping it and the cone

with a folded industrial tissue soaked in acetone. This may need to be repeated several times. After each wiping, leave the wet pad on the plate and bring the latter to within $\frac{1}{2}$ to $\frac{1}{4}$ in. of the cone. Then slide the tissue out and bring the plate to within $\frac{1}{4}$ to $\frac{1}{8}$ in. of the cone. After the last wiping, allow the plate surface to dry in the nitrogen stream. This takes a few seconds, and can be observed by the disappearance of the shine on the plate at a glancing angle.

(c) *Setting Cone*.—After the cleaning, bring the plate into contact with the cone. This is accomplished by raising the plate by turning the knurled cylinder till the stop is hit. Set the switch on the indicator or speed control unit at "Cone and Plate" so that a small current will pass and be indicated when the plate touches the cone tip. Make a final adjustment to 0.0001 in. with the micrometer while the cone is rotating at 5 to 10 rpm. When intermittent contact is apparent, turn the plate back about 0.0001 in. and lock it in position. There should be no contact now, but hand pressure on the top of the unit will cause intermittent contact. Once this position has been reached the instrument is ready for use. The plate can be lowered and raised back to the stop over and over again using the knurled cylinder, without change in position, although occasional checks should be made during a day's run to correct for thermal expansion.

(d) *Charging*.—Place one drop of the sample on the plate under the center of the cone with a hypodermic needle or medicine dropper, after lowering the plate slightly. Then raise the plate back to the contact position. Excess oil will ooze out around the edges. To avoid bubbles or unwet portions of the surfaces, it is best to have the cone rotating at about 10 rpm during this operation. The whole step should be done rapidly to avoid ice formation on the plate.

(e) *Measurement*.—Allow the temperature, as measured by the centermost thermocouple, to return to its equilibrium value, using the temperature recorder. This should take 5 to 10 min or less. Set the drive gears in the "high" position and increase the speed to the desired value. Allow the instrument to run until constant values of the

torque and temperature are reached (about 10 min). Record the speed, torque, and temperature. This constitutes a measurement. Speeds corresponding to 4000 to 9000 reciprocal seconds have been used. The viscosities at the various speeds differ little in this range and correlate fairly well with engine performance.

(f) *Calibrations*.—Calibrate the instrument with a reference oil which is Newtonian at 0 F and has an accurately measurable viscosity in the range of interest, for example, 25 ± 10 poises or stokes at 0 F for 10W oils. Make a series of runs at different speeds. Then plot the torque in the arbitrary units versus the product of the speed (in rpm) times the viscosity. This is the calibration curve for the instrument. In making these measurements it will be found that unless great pains are taken, the equilibrium temperature will not be 0 F. If the starting temperature is selected properly, the temperature at the lowest speeds will be -2 F and at the highest speeds $+2$ F. The calibration line must be drawn using the viscosities at the test temperatures. These can be calculated if the variation of the viscosity of the calibration oil with temperature is known. For example, for ASTM Low Temperature Reference Oil No. 3 the variation of viscosity with temperature at 0 F is 7.75 per cent per deg Fahr for small changes in temperature. For changes of more than a few per cent use the following formula:

$$\log \frac{\eta}{\eta_0} = -\frac{T}{29.7}$$

where:

η = viscosity,

η_0 = viscosity at 0 F (33.0 stokes), and

T = temperature, deg Fahr.

Corrections larger than 15 per cent should not be made. The runs should be repeated at temperatures closer to 0 F.

(g) *Viscosity Determinations*.—To determine the viscosity of an unknown oil read the value of the viscosity times the speed corresponding to the observed torque from the calibration curve. Dividing this value

by the known speed gives the viscosity at the observed temperature. Often the observed temperature is not exactly 0 F due to temperature rises that are not estimable in advance. Two correction methods may be employed to obtain viscosities at 0 F: (1) Make constant-speed runs at different coolant temperatures until the desired temperature is reached or bracketed, or (2) by interpolation to obtain the viscosity at exactly 0 F. A less exact method is to change the speed of rotation at constant coolant temperature setting until the desired test temperature is bracketed. This latter procedure is simpler but has the disadvantage of giving results at varying shear rates. However, as it has been found that shear rate in this range has a small effect on the viscosity, this drawback is not serious. The manufacturer's calibration may be used to relate the speed to the shear rate.

Precautions and Maintenance Requirements

4. There are a number of minor adjustments that must be made from time to time, which are discussed in the instruction manual furnished by the manufacturer. Principal among these are the zero speed and torque adjustments. There are also various lubrication and maintenance requirements suggested by the manufacturer for the precision bearings, gears, motor commutator, and so on.

Precision and Correlation

5. In the one extensive study carried out to date (July, 1961) the steady-state viscosities as measured at 8600 sec^{-1} on this instrument were shown to correlate with engine cranking viscosities with a standard deviation of ± 9.4 per cent. A large part of this variation is due to the error in the cranking tests themselves (estimated standard deviation = 8.7 per cent). The intralaboratory repeatability appears to vary with the type of oil tested and with the standard deviation as high as 5 per cent is possible. Interlaboratory reproducibility data are sparse but the standard deviation may be slightly higher than 5 per cent.

METHOD B. AMERICAN OIL CO. PROCEDURE

Scope

6. This method of test describes a procedure for using the Ferranti-Shirley cone and plate viscometer to measure, in poises, the apparent viscosity of polymer-thickened oils at low temperatures. The procedure applies in particular to the use of the smallest cone with 10W/30 motor oils, where the results may be correlated with cranking effort in engines.⁷

Summary of Method

7. One drop of sample is placed upon the plate of the viscometer. Cold isopropyl alcohol is circulated through the plate until the sample is chilled to a suitable subzero temperature as measured by a thermocouple. The plate is raised to contact the cone, and the cone is then rotated at a fixed speed for $\frac{1}{2}$ hr. Torque readings are taken at prescribed intervals. Viscosities are computed by multiplying the average of the final torque to speed ratios by a calibration constant established with Newtonian reference oils.

Apparatus

8. The apparatus comprises the Ferranti-Shirley viscometer⁸ plus a mechanically refrigerated cold box (regulated by a potentiometer-type recorder-controller) for cooling the alcohol circulating through the plate, a bath of dry ice and alcohol for cooling a nitrogen purge stream, and a precision potentiometer for measuring the plate temperatures. Modifications to the viscometer are insulation of the plate with asbestos paper, use of a cork ring about the cone, and incorporation of thermometers, graduated in 0.2 F, in glass sleeves attached to the ports in the plate by rubber tubing.

Standardization of Viscometer

9. (a) Check the thermocouple embedded in the plate in melting ice against a cali-

brated thermocouple in a drop of oil maintained at 0 F. Record the millivolts corresponding to 0 F.

(b) Establish the calibration constant for the viscometer operating at a given shear rate by plotting observed torque to speed ratios against the known viscosities at 0 F, in poises, of at least two Newtonian reference oils bracketing the range of interest. Use of the instrument is not recommended at shear rates above which the indicated plots are no longer linear.

Procedure

10. (a) 1. Place a drop of the sample in the center of the lowered plate. Add the cork ring, and raise the assembly into the approximate operating position by means of the coarse screw adjustment. Introduce the nitrogen purge stream into the cone (ordinarily the smallest available) by means of a flexible tubing passing through the wall of the cork ring. Adjust the temperature of the nitrogen gas to approximately 0 F by regulating the flow rate through the copper tube in the cooling bath.

(b) Start the gear pump and circulate alcohol at 2 gal per min from a 4-gal reservoir inside the cold box to the plate of the viscometer. Continue circulation until equilibrium temperatures, as measured by thermometers in the ports of the plate and the controller thermocouple in a well in the reservoir, are reached.

(c) Make the final positioning of the plate to the cone by means of the fine screw adjustment until electrical contact is just broken. Take an initial set of temperature readings (oil and alcohol), select the cone speed, and start the $\frac{1}{2}$ -hr run. After the first 10 min, read and record the torque and temperature every 5 min. Average the last three readings to provide the equilibrium values. To minimize interpolation errors, accept the run only if the oil temperature is within 1 deg of 0 F.

(d) Readjust the temperature of the circulating alcohol and repeat the run with the objective of bracketing the 0 F target temperature. Plot torque readings (log

⁷ E. O. Forster and H. H. Horowitz, "Application of the Cone-Plate Viscometer to Non-Newtonian Systems," see p. 85.

⁸ R. McKennel, "Cone-Plate Viscometer—Comparison with Coaxial Cylinder Viscometer," *Analytical Chemistry*, Vol. 28, p. 1710 (1956).

scale) versus millivolts (linear scale), and determine nominal reading at 0 F by interpolation.

(e) Clean and dry the plate with tissues moistened in hexane and in acetone.

Calculation

11. Calculate the viscosity of the sample as follows:

$$\eta = K \frac{T}{S}$$

where:

η = apparent viscosity, in poises at 0 F,

T = torque reading of the instrument,

S = speed of the cone in rpm, and

K = slope of the calibration line.

METHOD OF TEST FOR VISCOSITY OF ENGINE OILS BY THE FORCED-BALL VISCOMETER

Scope

1. This method describes a procedure for measuring the viscosity of engine oils at low temperatures by the forced-ball viscometer. Measurements are made at a shear rate of $2000 \pm 500 \text{ sec}^{-1}$ which approximates the shear rates existing in internal combustion engines at cranking speeds.

Apparatus

2. (a) *Viscometer*.—The viscometer used is the forced-ball viscometer,⁹ essentially a falling ball viscometer. In operation the ball is forced down through the closed-end cylinder filled with the oil under test. The time required for the ball to fall a selected distance through the cylinder (fall time) is recorded on an electric timer.

(b) *Bath*.—The oil sample, cylinder, and ball are held at a constant temperature by means of a modified Zeitfuchs bath. Commercial alcohol is used as the bath liquid.

Calibration of Viscometer

3. (a) Calibration of the viscometer is carried out by putting standard fluids of known viscosities in the cylinder and determining the length of time for the ball to fall 2 in. under various loads. The load is then plotted against reciprocal fall time (proportional to shear stress and shear rate respectively) and the best line through the points is determined by the method of least squares. The intercept of this best line with

the load coordinate is the value of the friction associated with the apparatus. This load correction is usually below 2 g for viscometers equipped with air bearings and remains essentially constant for extended periods if the air bearings are kept reasonably clean.

(b) The calibration factor k is determined from the plot of load versus reciprocal fall time by the equation:

$$k = \frac{\text{slope}}{\text{viscosity}} = \frac{\text{net load} \times \text{fall time}}{\text{viscosity}}$$

where the slope is the net ball load (platform weight minus the load correction) in grams divided by the reciprocal fall time (for a 2-in. fall) in seconds, and the viscosity of the calibration fluid at the test temperature is in centipoises. The calibration factor varies with ball and cylinder diameters as well as with temperature.

Procedure

4. (a) Condition 50 ml of the oil to be analyzed at a temperature of $120 \pm 5 \text{ F}$ for $\frac{1}{2}$ hr.

(b) Fill the cylinder and cup with about a 25-ml sample of the conditioned oil.

(c) Place the cup in the alcohol bath, and connect the ball. Bring the ball, cup, and test oil to a temperature of $0 \pm 0.05 \text{ F}$. Move the ball up and down in the cylinder occasionally during the cooling. The ball may be raised manually or by placing weights on the counterbalance. When raising the ball, extreme care must be taken to prevent pulling air into the sample.

(d) When the temperature of the sample reaches 0 F, adjust the weights on the platform to provide a shear rate of 2000 ± 500

⁹ For a complete description of this instrument see: Theodore W. Selby and Norman A. Hunstad, "The Forced-Ball Viscometer and Its Application to the Rheological Characterization of Mineral Oil Systems," p. 98. See Fig. 1 of that paper for a diagram of the forced-ball viscometer.

reciprocal seconds. The ball must be pushed to the side of the cylinder prior to each measurement of fall time.

(e) When the fall rate becomes constant under a given load as indicated by 8 out of 10 falls varying less than ± 0.01 sec, record the ball load and fall time.

Calculations

5. (a) Calculate the viscosity of the sample as follows:

$$\text{viscosity of sample} = \frac{\text{net load} \times \text{fall time}}{k}$$

where k = calibration factor of the viscometer.

(b) Calculate the shear rate, which is related to the fall time, as follows:

$$\bar{G} = \frac{V}{a^2} \left[3r + a + \frac{2a^2}{2r + a} \right]$$

where:

\bar{G} = average shear rate, in reciprocal seconds,

V = ball velocity, in cm per sec,

a = average annulus width, in cm, and

r = ball radius, in cm.