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An American National Standard

Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature[1]

This standard is issued under the fixed designation D 4684; 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 Department of Defense.

~~ε1 NOTE—Added research report to existing footnote for Section 13 editorially in December 2008.~~

1. Scope*

1.1 This test method covers the measurement of the yield stress and viscosity of engine oils after cooling at controlled rates over a period exceeding 45 h to a final test temperature between –10 and –40°C. The viscosity measurements are made at a shear stress of 525 Pa over a shear rate of 0.4 to 15 s^{-1}. The viscosity as measured at this shear stress was found to produce the best correlation between the temperature at which the viscosity reached a critical value and borderline pumping failure temperature in engines.

~~1.2Procedure A of this test method has precision stated for a yield range from less than 35 Pa to 210 Pa and apparent viscosity range from 4300 to 270 000 mPa·s. The test procedure can determine higher yield stress and viscosity levels.~~

~~1.3This test method is applicable for unused oils, sometimes referred to as fresh oils, designed for both light duty and heavy duty engine applications. It also has been shown to be suitable for used diesel and gasoline engine oils. The applicability to petroleum products other than engine oils has not been determined.~~

~~1.4This test method uses the milliPascal second (mPa·s) as the unit of viscosity. For information, the equivalent unit, centiPoise (cP), is shown in parentheses.~~

~~1.5This test method contain two procedures: Procedure A incorporates several equipment and procedural modifications from Test Method D4684–02 that have shown to improve the precision of the test, while Procedure B is unchanged from Test Method D4684–02. Additionally, Procedure A applies to those instruments that utilize thermoelectric cooling technology and those that use indirect refrigeration technology of recent manufacture for instrument temperature control. Procedure B can use the same instruments used in Procedure A or those cooled by circulating methanol.~~

1.2 This test method contain two procedures: Procedure A incorporates several equipment and procedural modifications from Test Method D 4684–02 that have shown to improve the precision of the test, while Procedure B is unchanged from Test Method D 4684–02. Additionally, Procedure A applies to those instruments that utilize thermoelectric cooling technology or direct refrigeration technology of recent manufacture for instrument temperature control. Procedure B can use the same instruments used in Procedure A or those cooled by circulating methanol.

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1.4 This test method is applicable for unused oils, sometimes referred to as fresh oils, designed for both light duty and heavy duty engine applications. It also has been shown to be suitable for used diesel and gasoline engine oils. The applicability to petroleum products other than engine oils has not been determined.

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

1.5.1 *Exception*—This test method uses the milliPascal second (mPa·s) as the unit of viscosity. For information, the equivalent unit, centiPoise (cP), is shown in parentheses.

1.6 *This standard does not purport to address all of the safety concerns, if any, 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.*

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

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

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 3829 Test Method for Predicting the Borderline Pumping Temperature of Engine Oil

2.2 *ISO Standard:*[3]

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

ISO Guide 34 General Requirements for the Competence of Reference Material Producers

ISO Guide 35 Certification of Reference Materials

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity*—the determined viscosity obtained by use of this test method.

3.1.2 *Newtonian oil or fluid*—an oil or fluid that at a given temperature exhibits a constant viscosity at all shear rates or shear stresses.

3.1.3 *non-Newtonian oil or fluid*—an oil or fluid that at a given temperature exhibits a viscosity that varies with changing shear stress or shear rate.

3.1.4 *shear rate*—the velocity gradient in fluid flow. For a Newtonian fluid in a concentric cylinder rotary viscometer in which the shear stress is measured at the inner cylinder surface (such as this apparatus, described in 6.1), and ignoring any end effects, the shear rate is given as follows:

$$G_r = \frac{2(\Omega)R_s^2}{R_s^2 - R_r^2} \quad (1)$$

$$= \frac{4(\pi)R_s^2}{t(R_s^2 - R_r^2)} \quad (2)$$

where:

G_r = shear rate at the surface of the rotor in reciprocal seconds, s^{-1},
Ω = angular velocity, rad/s,
R_s = stator radius, mm,
R_r = rotor radius, mm, and
t = time in seconds for one revolution of the rotor.

For the specific apparatus being described in 6.1.1,

$$G_r = 63/t \quad (3)$$

3.1.5 *shear stress*—the motivating force per unit area for fluid flow. For the rotary viscometer being described, the rotor surface is the area under shear or the shear area.

$$T_r = 9.81\, M\,(R_o + R_t) \times 10^{-6} \quad (4)$$

$$S_r = \frac{T_r}{2(\pi)R_r^2 h} \times 10^9 \quad (5)$$

where:

T_r = torque applied to rotor, N·m,
M = applied mass, g,
R_o = radius of the shaft, mm,
R_t = radius of the string, mm,
S_r = shear stress at the rotor surface, Pa, and
h = height of the rotor, mm.

For the dimensions given in 6.1.1,

$$T_r = 31.7\, M \times 10^{-6} \quad (6)$$

$$S_r = 3.5\, M \quad (7)$$

3.1.6 *viscosity*—the ratio between the applied shear stress and rate of shear, sometimes called the coefficient of dynamic viscosity. This value is thus a measure of the resistance to flow of the liquid. The SI unit of viscosity is the Pascal second [Pa·s]. A centipoise (cP) is one milliPascal second [mPa·s].

3.2 *Definitions of Terms Specific to This Standard:*

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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 International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

3.2.1 *calibration oils*—those oils that establish the instrument's reference framework of apparent viscosity versus speed, from which the apparent viscosities of test oils are determined. Calibration oils, which are essentially Newtonian fluids, ~~are available commercially~~shall be obtained from suppliers complying with ISO Guide 34, ISO Guide 35, and ISO 17025 ~~and traceable~~with traceability to a national metrology institute ~~(NMI), and~~(NMI). These calibration oils will have an approximate viscosity of 30 Pa·s (30 000 cP) at −20°C or 60 Pa·s (60 000 cP) at −25°C.

3.2.2 *cell constant*—the ratio of the calibration fluid viscosity to the time required to complete the first three measured revolutions of the rotor.

3.2.3 *test oil*—any oil for which the apparent viscosity and yield stress are to be determined by this test method.

3.2.4 *unused oil*—an oil which has not been used in an operating engine.

3.2.5 *used oil*—an oil which has been used in an operating engine.

3.2.6 *yield stress*—the shear stress required to initiate flow. For all Newtonian fluids and many non-Newtonian fluids, the yield stress is zero. An engine oil can have a yield stress that is a function of its low-temperature cooling rate, soak time, and temperature.

4. Summary of Test Method

4.1 An engine oil sample is held at 80°C and then cooled at a programmed cooling rate to a final test temperature and held for a specified time period. At the end of this period, a series of increasing low torques are applied to the rotor shaft until rotation occurs to determine the yield stress, if any is exhibited. A higher torque is then applied to determine the apparent viscosity of the sample.

5. Significance and Use

5.1 When an engine oil is cooled, the rate and duration of cooling can affect its yield stress and viscosity. In this laboratory test, a fresh engine oil is slowly cooled through a temperature range where wax crystallization is known to occur, followed by relatively rapid cooling to the final test temperature. These laboratory test results have predicted as failures the known engine oils that have failed in the field because of lack of oil pumpability.[4] These documented field failing oils have all consisted of oils normally tested at –25°C. These field failures are believed to be the result of the oil forming a gel structure that results in either excessive yield stress or viscosity of the engine oil, or both.

5.2 *Cooling Profiles*:

5.2.1 For oils to be tested at −20°C or colder, Table X1.1 applies. The cooling profile described in Table X1.1 is based on the viscosity properties of the ASTM Pumpability Reference Oils (PRO). This series of oils includes oils with normal low-temperature flow properties and oils that have been associated with low-temperature pumpability problems **(1-5)**.[5] Significance for the −35 and −40°C temperature profiles is based on the data collected from the "Cold Starting and Pumpability Studies in Modern Engines" conducted by ASTM **(6,7)**.

5.2.2 For oils to be tested at −15 or −10°C, Table X1.2 applies. No significance has been determined for this temperature profile because of the absence of appropriate reference oils. Similarly, precision of the test method using this profile for the −10°C test temperature is unknown. The temperature profile of Table X1.2 is derived from the one in Table X1.1 and has been moved up in temperature, relative to Table X1.1, in consideration of the expected higher cloud points of the viscous oils tested at −15 and −10°C.

6. Apparatus

6.1 *Mini-Rotary Viscometer*—An apparatus that consists of one or more viscometric cells in a temperature-controlled block made of a metallic material with high thermal conductivity. Each cell contains a calibrated rotor-stator set. The rotor shall have a crossbar near the top of the shaft extending in both directions far enough to allow the locking pin (6.6) to stop rotation at successive half turns. Rotation of the rotor is achieved by an applied force acting through a string wound around the rotor shaft.

6.1.1 The mini-rotary viscometric cell has the following ~~typical~~ dimensions:

~~Diameter of rotor~~	~~17.0 ± 0.05 mm~~
Diameter of rotor	17.06 ± 0.08 mm
Length of rotor	20.0 ± 0.14 mm
~~Inside diameter of cell~~	~~19.0 ± 0.05 mm~~
Inside diameter of cell	19.07 ± 0.08 mm
Radius of shaft	3.18 ± 0.13 mm
Radius of string	0.1 mm

6.1.2 *Cell Cap*~~—A cover inserted into the top of the viscometer cell to minimize room air circulation into the cells. The cell cap is a stepped cylinder 38 ± 1 mm (1.5 ± 0.05 in.) in length made of a low thermal conductivity material such as an acetyl copolymer **like Delrin**®. The top half is 28 ± 1 mm (1.10 ± 0.05 in.) in diameter and the bottom half is 19 mm (0.745 in.) in diameter with a tolerance consistent with the cell diameter. The tolerance on the bottom half is such that it will easily fit into cell but not allow cap to contact rotor shaft. The piece has a center bore of 11 ± 1 mm (0.438 ± 0.05 in.). The cap is made in two~~

[4] Pumpability Reference Oils (PRO) 21 through 29.

[5] The boldface numbers in parentheses refer to the references at the end of this standard.

~~halves to facilitate placement in the top of the cell.~~ —A cover inserted into the top of the viscometer cell to minimize room air circulation into the cells is required for thermometrically cooled instruments. The cell cap is a stepped cylinder 38 ± 1 mm (1.5 ± 0.05 in.) in length made of a low thermal conductivity material, for example, thermoplastic such as acetyl copolymers that have known solvent resistivity and are suitable for use between the temperature ranges of this test method. The top half is 28 ± 1 mm (1.10 ± 0.05 in.) in diameter and the bottom half is 19 mm (0.745 in.) in diameter with a tolerance consistent with the cell diameter. The tolerance on the bottom half is such that it will easily fit into cell but not allow cap to contact rotor shaft. The piece has a center bore of 11 ± 1 mm (0.438 ± 0.05 in.). The cap is made in two halves to facilitate placement in the top of the cell.

6.1.2.1 Cell caps shall not be used in the direct refrigeration instruments, since such use would block the flow of cold, dry air into the stators to keep them frost-free.

6.2 *Weights*:

6.2.1 *Yield Stress Measurement*—A set of ten weights, each with a mass of 10 ± 0.1 g. One of the weights is a holder for the other weights.

6.2.2 *Viscosity Measurement*—Weight with mass of 150 ± 1.0 g.

6.3 *Temperature Control System* —Regulates the mini-rotary viscometer block temperature in accordance with the temperature requirements described in Table X1.1 or Table X1.2.

6.3.1 *Temperature Controller*—As a very critical part of this procedure, a description of the requirements that the controller shall meet are included in Appendix X2.

6.3.2 *Temperature Profile*—The temperature profile is fully described in Table X1.1 and Table X1.2.

6.4 *Thermometers*— For measuring the temperature of the block. Two are required, one graduated from at least +70 to 90°C in 1°C subdivisions, the other with a scale from above +5°C down to at least –41°C or lower, in 0.2°C subdivisions. Other thermometric devices of equal accuracy and resolution may be used to measure the temperature, such as digital meters using a resistance temperature detector (RTD) or a thermistor sensor.

6.4.1 When using metal encased thermometric devices, care should be taken that the metal case does not create a biased temperature reading. It has been observed that some metal sheathed devices indicate a higher than actual temperature of the sample. This is typically caused by heat conduction through the metal sheath but there can be other causes.

6.5 *Supply of Dry Gas*—A supply of dry filtered dry gas to minimize moisture condensation on the upper portions of the instrument.

6.5.1 For thermoelectric cooled instruments, which use cell caps, the dry gas supply is connected to the housing cover. The supply of dry gas is discontinued when the cover is removed for the measurement phase of the test.

6.6 *Locking Pin*—A device to keep the rotor from turning prematurely and able to stop the rotor at the nearest half revolution by interaction with the rotor crossbar.

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7. Reagents and Materials

7.1 *Newtonian Oil*— Low cloud-point of approximately 30 Pa·s (30 000 cP) viscosity at –20°C for Procedure B or 60 Pa·s (60 000 cP) at –25°C for Procedure A for calibration of the viscometric cells.

7.2 *Methanol*—Commercial or technical grade of dry methanol is suitable for the refrigerated cooling bath required for some units. (**Warning**—Flammable.)

7.3 *Oil Solvent*—Commercial heptanes or similar solvent that evaporates without leaving a residue is suitable. (**Warning**—Flammable.)

7.4 *Acetone*—A technical grade of acetone is suitable provided it does not leave a residue upon evaporation. (**Warning**—Flammable.)

Procedure A

8. Sampling

8.1 A representative sample of test oil free from suspended solid material and water is necessary to obtain valid viscosity measurements. If the sample in its container is received below the dew-point temperature of the room, allow the sample to warm to room temperature before opening the container.

9. Calibration and Standardization

9.1 *Calibration Procedure*—For those instruments in which the temperature sensor is not permanently attached to the temperature controller, calibrate the temperature sensor in the MRV block while the sensor is attached to the temperature controller.

9.1.1 The sensed temperature calibration shall be verified using a reference thermometer noted in 6.4 at a minimum of three temperatures.

~~9.1.2During the calibration all the cells are to contain 10 mL of a typical fluid with the rotor and cell caps in place.~~

9.1.2 During the calibration all the cells are to contain 10 mL of a typical fluid with the rotor and, if required, cell caps in place. Cell caps shall not be used for direct refrigeration instruments (see 6.1.2).

9.1.3 Make these temperature measurements at least 5°C apart and include both –5°C and the lowest test temperature used to establish a calibration curve for this combination of temperature sensor and controller. Make at least two temperature measurements at every calibration temperature with at least 10 min between observations. For instruments using an independent temperature controller, see X2.1 for calibration guidance.

NOTE 1—All temperatures in this test method refer to the actual temperature and not necessarily the indicated temperature.

9.2 The calibration constant of each rotor/stator combination is determined by conducting two tests at –25°C using a viscometric standard as a test sample.

9.2.1 Each cell shall be calibrated twice and the resulting calibration constant is to be calculated from the average of the two determinations of the time for three revolutions of the rotor. When the two cell calibrations are consecutive, the second test shall be on a new sample of standard with cleaning between the steps.

NOTE 2—Once a set of rotors have been calibrated in an instrument, subsequent calibration checks can be single determinations if the criteria of 9.11 are met.

9.2.2 The same 150 g weight is to be used for both calibration and viscosity measurements. However, different weights may be used for calibration and viscosity measurements if the masses of the two weights are certified to be 150 ± 0.1 g.

9.3 Using steps in 10.1, prepare the cells for the calibration test cycle.

NOTE 3—Before inserting the rotors in the cells, inspect each rotor to be sure that the shaft is straight, that the rotor surface is smooth and free from dents, scratches and other imperfections. For ~~thermoelectric cooled instruments, where the~~ rotors ~~have~~with a bearing point at the bottom of the shaft, ensure that the point is sharp and centered on the rotor shaft. If these conditions are not met, repair or replace the rotor.

9.4 Using either the calibration temperature profile given for the instrument (or, alternatively, the cooling profile given in Test Method D 3829) for the test temperature of the reference fluid, follow the owner's manual instructions for the instrument to initiate the cooling profile program.

NOTE 4—The use of the calibration temperature profile makes it possible to complete two cell constant determinations in one day.

9.5 Place the thermometer in the thermowell. The same thermowell location is to be used for all measurements.

9.5.1 The thermometer must be placed into the thermowell at least one hour prior to completion of the test.

9.6 At the completion of the temperature profile, check that the final test temperature is at the desired calibration temperature within ±0.1°C. Final test temperature is to be verified independently of the temperature controller using the thermometer in the thermowell.

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9.7 Beginning with the cell farthest to the left facing the instrument, follow this procedure for each cell in turn.

9.7.1 Align the pulley wheel with the rotor shaft for the cell to be tested.

9.7.2 Hang the string over the timing wheel.

9.7.3 Suspend the weight holder plus a 10-g weight (total mass 20 g) from string.

9.7.4 Disengage the locking pin.

9.7.5 As soon as the crossbar is clear of the locking pin, reengage the locking pin. This will stop the rotation at approximately one half revolution.

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9.7.6 Remove weight holder and 10-g weight from the string.

9.7.7 Suspend the 150-g weight from the string.

9.7.8 Disengage the locking pin and simultaneously start timing as soon as the rotor is released.

9.7.9 Determine the time for exactly three revolutions of the rotor.

NOTE 5—For some instruments, the timing may be done automatically.

9.7.10 After three revolutions, reengage the locking pin and remove the weight from the string.

9.7.11 Record the time for three revolutions and the cell number.

9.8 Repeat 9.7.1-9.7.11 for each of the remaining cells in numerical order.

9.9 Repeat 9.3-9.8 for a second set of calibration data.

9.10 For each cell (rotor/stator combination) calculate of the calibration constant using Eq 8 and 9.

$$t = (t_1 + t_2)/2 \tag{8}$$

$$C = \eta/t \tag{9}$$

where:

η = viscosity of the standard oil, mPa·s (cP) at test temperature,
C = cell constant,
t_1 = time of three rotor revolutions for first calibration,
t_2 = time of three rotor revolutions for second calibration, and
t = average time of three rotor revolutions.

9.11 After the calibration constants have been determined, check to see if any cell has a calibration constant differing by more than 4% from the average of all cells or if the difference between t_1 and t_2 for any cell is greater than 4% of the average of t_1 and t_2. If so, then one or both of the results should be considered suspect. If these criteria are not met, examine the indicated rotor for damage, repair or replacement as necessary, and repeat the cell calibrations.

9.12 If corrected values for the controller temperature and thermometer deviate by more than the tolerance (±0.1°C), use the procedure in X2.2 to assist in determining the cause and correction.

10. Yield Stress and Viscosity Measurement Procedure

10.1 *Test Sample and Viscometric Cell Preparation*:

10.1.1 If the cells are not clean, see 10.7 for the cleaning procedure.

10.1.2 Place 10 ±0.2 mL of test oil samples into the clean cells.

10.1.2.1 All cells should contain a fluid and rotor; if there are less than a full set of samples to run, fill each of the unused cells with a typical test sample.

10.1.3 *Loading Cells with Test Oils*—Place each rotor and test oil in its cell, and place upper pivot pin in position, including any unused cells.

~~10.1.4Install a cell cap on all cells, including any unused cells.~~

10.1.4 When use is required, install a cell cap on all cells, including any unused cells.

10.1.5 For each cell, except any unused ones, place a loop of the nominal 700-mm long string over the crossbar. Hang the string over the timing wheel with a small weight attached such as a large paper clip. Wind the string around the shaft until the end is about 100 mm below the wheel. Do not overlap windings.

NOTE 6—The strings can be pre-wound around the shafts before they are installed in 10.1.3.

10.1.5.1 Engage the locking pin to prevent the rotor from turning.

10.1.5.2 Lay the remaining string over the top of the bearing plate letting it hang over the back of the plate.

10.1.5.3 Repeat 10.1.5-10.1.5.2 until all cells with samples to be measured are prepared.

10.1.6 Place the housing cover over the viscometric cells.

10.1.7 Connect the dry gas supply to the housing cover, as noted in 6.5. Set the dry gas flow to approximately 1 L/h. Increase or decrease the flow as necessary to minimize frost or moisture condensation around the cells.

10.2 Select the cooling profile for the desired test temperature and follow the instrument instructions to initiate the program. Table X1.3 lists the nominal times to reach a particular test temperature.

10.3 Place the thermometer in the thermowell at least one hour prior to completion of the test. The same thermowell location is to be used for all measurements and must be the same one as was used in the calibration.

10.4 At the completion of the cooling profile, check the time-temperature plot for the run to ensure that the time-temperature profile is within tolerance and that the test temperature as measured in the thermowell is within ±0.2°C of the final test temperature. Both of these checks may be done automatically by the control software incorporated in some instruments. Final test temperature is to be verified independently from the temperature controller. If the final test temperature is more than 0.1°C from the set point on two consecutive runs, the temperature sensor must be recalibrated according to 9.1.

10.5 If the temperature profile is within tolerance, proceed with measurements. If not, then abort the test and recalibrate temperature controller as in 9.1.

10.6 *Measurement of the Yield Stress and Viscosity*:

10.6.1 Immediately prior to starting measurements, take the cell housing cover off the instrument.

10.6.2 *Yield Stress Determination*—Starting with the cell farthest to the left while facing the instrument, use the following procedure for each cell in turn, bypassing the unused cells.

10.6.2.1 Align the pulley wheel with the rotor shaft of the cell to be tested.

10.6.2.2 Hang the string over the timing wheel.

10.6.2.3 Suspend the 10-g weight holder from the string.

10.6.2.4 For instruments with automatic timing, start timing and then release the locking pin. For manual timing, start timing immediately after the locking pin is disengaged.

10.6.2.5 Observe whether the end of the crossbar moves more than 3 mm in 15 s. (This 3 mm is approximately twice the diameter of the crossbar.) An alternative procedure is the use of a marked rotation of the timing wheel equivalent to a rotor shaft rotation of 3 mm.

10.6.2.6 Electronic or timing wheel motion-sensing devices, which are available on some instruments, are suitable alternatives to direct observation.

10.6.2.7 If rotor movement of more than 3 mm, or alternative, in 15 s is observed in 10.6.2.5, remove all of the 10-g weights from the end of the string, and proceed to 10.6.3.

10.6.2.8 If a rotor movement of less than 3 mm in 15 s is observed in 10.6.2.5, stop timing and lift the weight holder so it is not supported by the string. Then add an additional 10-g weight to weight holder.

NOTE 7—As additional weights are added to the weight holder it is necessary to suspend the holder with the additional weights from the string and restart timing without the use of the locking pin for the remainder of the yield stress assessment. When using software available for some instruments, ensure that the mass applied is the mass requested by the program.

10.6.2.9 Carefully and gently, suspend the weight holder with the additional weights from the string and start timing.

10.6.2.10 Repeat steps in 10.6.2.8 and 10.6.2.9 until the accumulated weights causes rotation of the rotor. At this point, remove all the weights from the string.

10.6.2.11 If no rotation is observed with a total of 100 g, record that the yield stress is >350 Pa, and proceed with 10.6.3.

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