



Designation: ~~D6335~~—~~09~~ D6335 – 16

Standard Test Method for Determination of High Temperature Deposits by Thermo- Oxidation Engine Oil Simulation Test¹

This standard is issued under the fixed designation D6335; 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.

1. Scope*

1.1 This test method covers the procedure to determine the amount of deposits formed by automotive engine oils utilizing the thermo-oxidation engine oil simulation test (TEOST²).³ An interlaboratory study (see Section 17) has determined it to be applicable over the range from ~~10~~ 10 mg to ~~65~~ 65 mg total deposits.

NOTE 1—Operational experience with the test method has shown the test method to be applicable to engine oils having deposits over the range from ~~22~~ 22 mg to ~~180~~ 180 mg total deposits.

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

1.2.1 Milligrams (mg), grams (g), ~~millilitres~~ milliliters (mL), and ~~litres~~ liters are the units provided, because they are an industry accepted standard.

1.2.2 ~~Exception—Provided~~ psig—Pounds per square inch gauge (psig) is provided for information only in 6.2.

1.3 ~~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.~~

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 ~~ceramic isolator~~ isolator, *n*—~~the fitting that compresses the O-ring into the depositor rod casing and isolates the depositor rod casing from the voltage applied to the depositor rod.~~

2.1.2 ~~depositor rod~~ rod⁴, *n*—~~the steel rod a patented, specially made, numbered, and registered steel rod (used once for each test) on which the deposits are collected. It is resistively heated through a temperature cycle during the test series of twelve temperature cycles during the test to temperatures established and controlled by a thermocouple inserted to a pre-determined depth in the hollow rod.~~

2.1.3 ~~depositor rod casing~~ casing, *n*—~~the sleeve that surrounds the depositor rod and allows the flow of specimen the test oil up and around the outside of the rod.~~ rod at a flow rate such that every volume element of the test oil is exposed to the same heating cycle.

2.1.4 ~~drain tube~~ tube, *n*—~~the tube connecting the upper outlet of the depositor rod casing to the reaction chamber.~~

2.1.5 ~~end cap~~ cap, *n*—~~the fitting to tighten the ceramic isolators down onto the O-rings at the both ends of the depositor rod casing.~~

2.1.6 ~~filter deposits~~ deposits, *n*—~~the mass in mg milligrams of the deposits collected on the filter cartridge after test on a special multi-layer filter cartridge used once for each test.~~

2.1.7 ~~pump~~ pump, *n*—~~the gear pump that controls is used to control the flow rate of sample the test oil through the depositor rod casing.~~

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.09.0G on Oxidation Testing of Engine Oils.

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² TEOST is a trademark of the Tannas Co. (Reg. 2001396), Tannas Company, 4800 James Savage Rd., Midland, MI 48642.

³ The Development of Thermo-Oxidation Engine Oil Simulation Test (TEOST), Society of Automotive Engineers (SAE No. 932837), 400 Commonwealth Dr., Warrendale, PA 15096-0001.

⁴ The sole source of supply of the apparatus known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

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

2.1.8 *pump inlet tube*—*tube, n*—the tube connecting the reactor chamber to the pump.

2.1.9 *pump outlet tube*—*tube, n*—the tube connecting the pump to the depositor rod casing.

2.1.10 *reactor chamber*—*chamber, n*—the heated reservoir that contains the bulk of the sample throughout the test. It has a drain valve for removing sample at the end of the test and an inlet valve for adding gases to the sample. The chamber contains a magnetic stir bar well in the bottom in which a stir bar is placed to mix the reactor (approximately 100 mL) of the 116 mL of test oil sample circulated past the deposit rod during the test. The reactor is equipped with a magnetic stir-bar to continuously mix the chamber contents.

2.1.10.1 Discussion—

In the reaction chamber, moist air and nitrous oxide are each bubbled at a controlled rate of 3.5 mL/min through a channel opening at the bottom of the reaction chamber. This channel is also used to drain the test oil from the reaction chamber for filtration when the test is completed.

2.1.11 *rod deposits*—*deposits, n*—the mass, in milligrams, of the deposits collected on the depositor rod.

2.1.12 *rod O-rings*—*O-rings, n*—the O-rings that seal the outside of the rod and the depositor rod casing to prevent sample leaks.

2.1.13 *side nut*—*nut, n*—the fitting creates a seal to prevent sample leaking from the front holes of the depositor rod casing.

2.1.14 *thermocouple lock collar*—*collar, n*—a fitting that tightens on the thermocouple to ensure the thermocouple is at the correct depth when placed inside the rod.

2.1.15 *total deposits*—*deposits, n*—the rod deposits plus the filter deposits.

3. Summary of Test Method

3.1 A 116 mL sample of the engine oil to be tested containing 100 mg/kg ferric naphenate is put into the reaction chamber and heated and stirred at a temperature of 100°C that contains ferric naphenate and is in contact with nitrous- 100 °C. Nitrous oxide and moist air is pumped at a set flow rate are injected from a bottom channel opening, each at a flow rate of 3.5 mL/min. This catalyzed oil is pumped past a tared depositor rod. The rod that is resistively heated through twelve, 9.5 min-9.5 min temperature cycles that go from 200-200 °C to 480°C-480 °C. When the twelve cycle-twelve-cycle program is complete, the depositor rod is rinsed of oil residue and dried and the gross rod mass obtained. The sample-remaining test oil sample, including washing from the deposit rod, is flushed from the system and filtered through a tared filter. The mass of deposits on the rod plus the mass of deposits on the filter is the total deposit mass.

4. Significance and Use

4.1 The test method is designed to predict the high temperature deposit forming tendencies of an engine oil subject to the added oxidizing stress of a turbocharger. This test method can be used to screen oil samples or as a quality assurance tool.

5. Apparatus

5.1 *Thermo-oxidation engine oil simulation test (TEOST) test instrument.*⁴ See Fig. 1.

5.2 *Balance*, capable of weighing to the nearest ~~0.1 mg~~. 0.1 mg.

5.3 *Vacuum Source*, hand held, floor model, or house vacuum.

5.4 *Magnetic stirrer and stir bars.*

5.5 *Digital timer.*

5.6 *Petroleum and temperature resistant O-rings.*

5.7 *Ceramic isolators.*

5.8 *Polypropylene filters.*

5.9 *Plastic filter holder.*

5.10 *Plastic Petri Dishes*, for filter storage.

5.8 *Filtering Flask*—~~1000 mL~~. 1000 mL.

5.9 *Graduated Filter Funnel*—~~500 mL~~. 500 mL with Luer lock fitting.

5.10 *Graduated Cylinder*—~~150 mL~~. 150 mL.

5.11 *Beakers*—One small (for example, ~~25 mL~~). 25 mL). One beaker large enough to clean the depositor rod casing (for example, ~~600 mL~~). 600 mL).

5.12 *Graduated Cylinder*—~~10 mL~~. 10 mL.

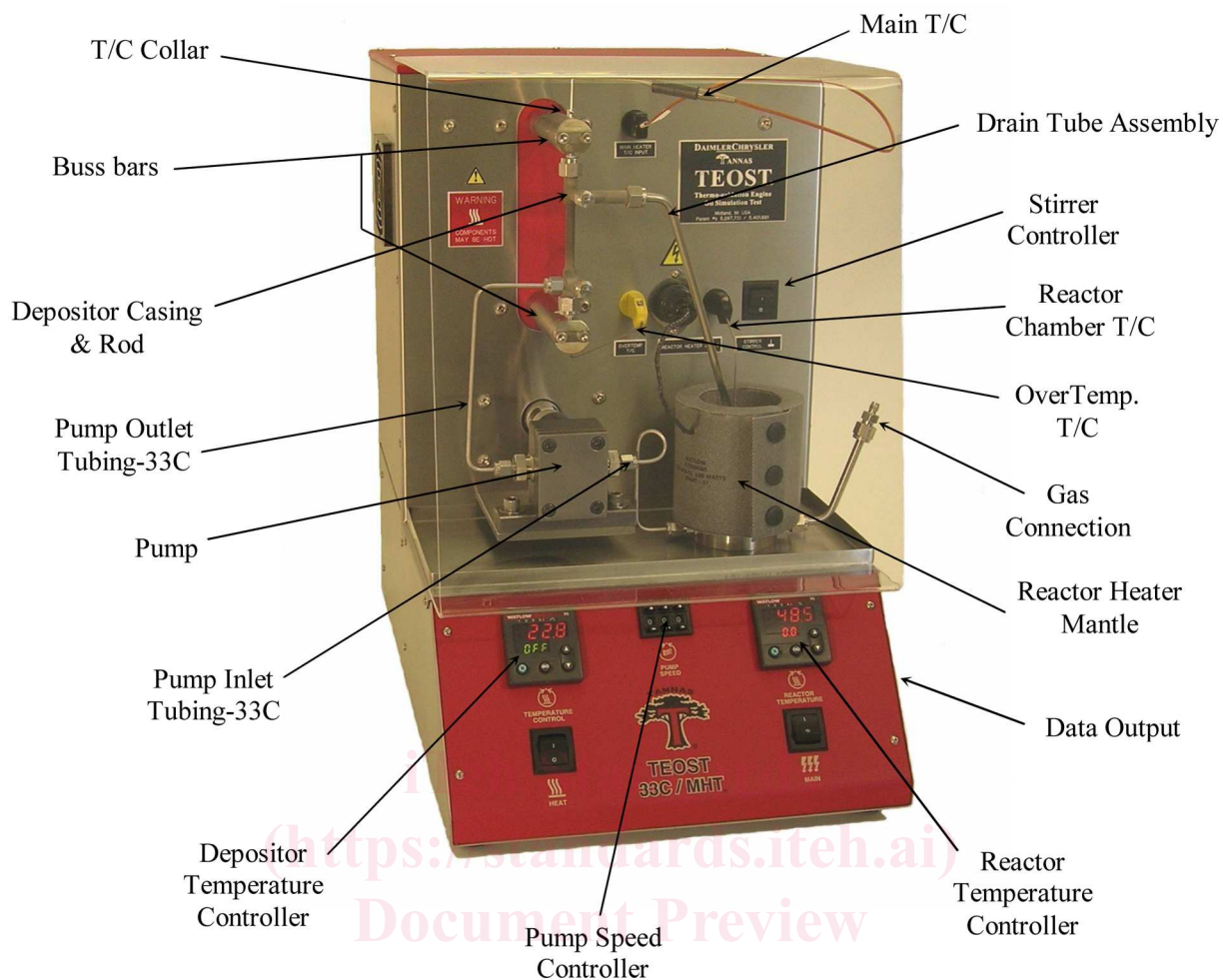


FIG. 1 Thermo-oxidation Engine Oil Simulation Test (TEOST)

5.13 Erlenmeyer Flask—50 mL. 50 mL.

5.14 Adjustable hex wrench.

5.15 Pipe Cleaners—33 mm × 304.8 mm. 304.8 mm.

5.16 Steel Wool—4/0 (ultra fine).

5.17 Brass Brush—0.22 caliber. caliber (5.588 mm).

5.18 Glass Syringe—100 µL. 100 µL. (Optional—A calibrated pipet can also be used with chemically inert disposable tips.)

5.19 Tannas one piece cartridge filters. (Optional One-piece disposable—Items 5.8, multi-layer 5.9, and cartridge filters. 5.10 are not needed if the optional Tannas one-piece cartridge filters is used.)⁴

5.20 Flow Meters, Meters—capable of measuring 60 mL/min to 1010 mL -mL- of air per min./min of gas.

5.21 Weighing Boat, Boat—light, circular or oblong open container, preferably made of aluminum with a diameter or length of approximately 77 cm to 10 cm-10 cm and notched in two diametrically opposed places to prevent the rod from rolling. (See Fig. 2.)

5.22 Wire Rod—a clean, thin (about 1 mm diameter), somewhat flexible, stainless steel wire rod (approximately 150 mm in length) for dislodging any deposits adhering to the filter funnel walls into the filter cartridge.

6. Reagents and Materials

6.1 Nitrous Oxide (N₂O)—USP compressed Compressed gas cylinder, medical grade. 99.6 % purity or higher.

6.2 Moist Air—Hydrocarbon-free air regulated to 103.4 kPa (15 psig) before 103.4 kPa (15 psig) before entering the flow meter and then bubbled through approximately 30 mL-30 mL of water in a small Erlenmeyer flask-flask before combining with the nitrous oxide and entering the reaction chamber.

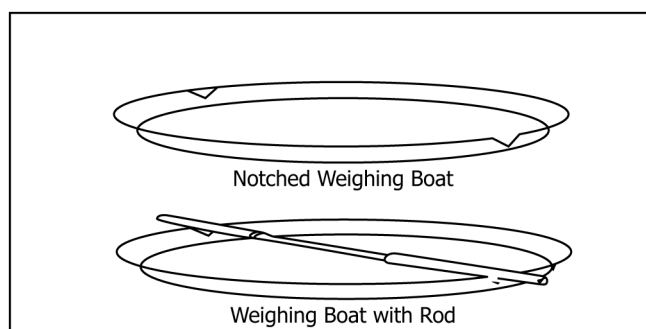


FIG. 2 Weighing Boat and Rod

6.3 *Ferric Naphthenate*—Six percent iron content in mineral spirits.

6.4 *Cyclohexane or Heptane—Cyclohexane, Heptane, or Other Alkane Hydrocarbon Solvent of Equivalent Volatility—Industrial Reagent* grade. (**Warning—Flammable.**) Cyclohexane is the only allowed naphthenic hydrocarbon. Do not use other naphthenic or any aromatic hydrocarbons. Throughout the further description of the test, the solvent selected is referred to as “hydrocarbon solvent.”

6.4.1 The volatility of the hydrocarbon solvent selected should ensure timely evaporation from the deposits on the rod and filter. In general, the higher the purity of the solvent, the more quickly the solvent should evaporate.

6.5 *Low Deposit Reference Oil*—CG-1 reference oil⁴ is a petroleum oil capable of generating total deposits in the ~~20~~20 mg to 30 mg range. The acceptable deposit range of a specific lot is provided by the supplier of that lot.

6.6 *Intermediate Deposit Reference Oil*—CF-1 reference oil⁴ is a petroleum oil capable of generating total deposits in the ~~50~~50 mg to ~~60~~60 mg range. The acceptable deposit range of a specific lot is provided by the supplier of that lot.

6.7 *Pump Calibration Fluid*—TPC.⁴

7. Calibration

7.1 The TEOST instrument is calibrated by performing the procedure described in Section 8. At that point, either a low or high deposit reference oil shall be run. The results shall be within the repeatability limits established by the supplier of the reference oils.

7.2 The calibration should be performed a minimum of every six months, as recommended by the instrument manufacturer.

7.3 If the repeatability is not within the established limits, the instrument setup steps in Section 8 should be performed. Then the reference oil should be rerun.

8. Setup of the Test Instrument

8.1 *Pump Speed Calibration*—The pump speed should be calibrated using the instructions found in the operations manual. It is recommended that this calibration be done every six months.

8.2 *Thermocouple Depth*—The thermocouple depth setting (distance from tip to locking collar) should be determined using the procedure in the operations manual. The depth setting should be checked daily and should be redetermined whenever a new thermocouple is installed.

8.3 *Thermocouple Calibration*—The thermocouple shall be calibrated every six months or when replaced. This can be done by placing the thermocouple into a liquid or sand bath while simultaneously measuring the temperature by a certified liquid or digital thermometer. The temperature controller may then be offset to display the correct temperature.

8.4 *Flow Calibration*—Ensure the proper operation of the flow meters by connecting a digital flow meter to the output. The flow for the air shall be $3.53.5 \text{ mL/min} \pm 1 \text{ mL/min/min}$ and the N₂O flow shall be $3.53.5 \text{ mL/min} \pm 1 \text{ mL/min/min}$.

8.5 *PID Settings*—The PID settings on the temperature controller MUST be set to Pb: 80, Re: 2.0, and Ra: 0.2. Consult the operations manual for further guidance.

8.6 *Power Adjustments*—This procedure, used only for instruments made prior to 1999, is for making power adjustments and is given in the operations manual. It is recommended that the power adjustments be made by a qualified instrument technician.

8.7 Verify that the temperature program shown in Table 1 is entered. When verifying the temperature program, always be sure NOT to select *guaranteed* or *assured soak*.

9. Assembly of Apparatus

9.1 Assemble the TEOST system by placing the reaction chamber in the bolt seats on the TEOST platform with the drain and gas inlet tubing facing the right side of the instrument.

TABLE 1 Temperature Program

| Program Mode | Value |
|--------------|----------|
| Set point 0 | 200°C |
| Set point 0 | 200 °C |
| Time 1 | 1.15 min |
| Set point 1 | 200°C |
| Set point 1 | 200 °C |
| Time 2 | 1.00 min |
| Set point 2 | 480°C |
| Set point 2 | 480 °C |
| Time 3 | 2.00 min |
| Set point 3 | 480°C |
| Set point 3 | 480 °C |
| Time 4 | 4.00 min |
| Set point 4 | 200°C |
| Set point 4 | 200 °C |
| Time 5 | 1.15 min |
| Set point 5 | 200°C |
| Set point 5 | 200 °C |
| Time 6 | 0 min |
| Cycles | 12.00 |

9.2 Connect the pump inlet tube to the outlet connection of the reaction chamber and the inlet connection of the pump. Finger tighten the connections.

9.3 Connect the pump outlet tube to the outlet connection of the pump, and place a ~~10 mL~~ 10 mL graduated cylinder directly under the open end of the pump outlet tube.

9.4 Place the lid containing the thermocouple on the reaction chamber, making sure that the thermocouple is touching the bottom of the reaction chamber.

9.5 Wrap the heating jacket around the reaction chamber and secure it with the provided straps. Connect the heating jacket and the thermocouple to the labeled connections on the back wall of the instrument. The connectors shall be inserted and twisted to obtain a proper connection. Use the reactor temperature controller to set the temperature of the reaction chamber to ~~100°C~~ 100 °C.

9.6 Connect the gas tubing to the reaction chamber, and set the flow meters for the moist air and N₂O to ~~3.5 mL/min~~ 3.5 mL/min ± ~~1 mL/min~~ 1 mL/min. These are allowed to flow to purge out the lines before the test begins.

NOTE 2—Be sure the valve on the reaction chamber is pointing up to allow the gases to enter into the chamber.

10. Sample Preparation

10.1 After thoroughly mixing the test sample, use a graduated cylinder to measure ~~116 mL~~ 116 mL ± ~~1 mL~~ 1 mL of the fluid and pour it into a large beaker until only drips are coming from the graduated cylinder. Use a syringe to add ~~193 ± 1 µL~~ 193 ± 1 µL glass syringe or an optional calibrated pipet to add 193 µL ± 1 µL of the approximately 6 % ferric naphthenate solution to the ~~116 mL~~ 116 mL of test fluid. The resulting concentration of iron in the test fluid will be about ~~100-100 mg wt ppm/kg~~ 100-100 mg wt ppm/kg.

10.2 Use a magnetic bar and stirrer to mix the oil and ferric naphthenate for at least ~~5 min~~ 5 min but not more than ~~15 min~~ 15 min. Make certain that a vortex is not created. Periodically move the beaker around the stirrer (hence moving ~~the~~ the magnet around the bottom) to ensure the best possible stirring.

10.3 After the stirring is completed, inspect the oil solution by holding it up to a light to make sure it is homogenous. If not, stir for ~~5 min~~ 5 min more.

11. Procedure

11.1 When the reaction chamber is ~~100°C~~ 100 °C ± ~~5°C~~ 5 °C, pour the test sample, along with the stirrer bar, into the reaction chamber. Using the speed dial on the right side of the instrument, turn on the stirrer. ~~The older equipment has~~ Older instruments have a stirrer control versus a switch; this control must be set so that the stirrer is on but a vortex is not formed. The sample temperature should reach ~~100°C~~ 100 °C in approximately ~~15 min~~ 15 min.

11.2 Set the pump speed to 999, using the dial on the front panel of the instrument. Allow the pump to flush out ~~10 mL~~ 10 mL of fluid into the ~~10 mL~~ 10 mL graduated cylinder placed at the open end of the pump outlet tube. When flushing is complete, set the pump dial to 000 to stop the pump. Discard the ~~10 mL~~ 10 mL of oil.

11.3 Use ~~cyclohexane or heptane~~ hydrocarbon solvent to rinse off an unused rod, both on the outside and down the center. Clean each of the three sections of the rod lightly with 4/0 steel wool by stroking (up and down action) each section 20 times while turning the rod. Rinse the rod with acetone inside and out. Using a pipe cleaner soaked in acetone, clean the interior of the rod. Repeat the interior cleaning with a clean pipe cleaner through the rod in the opposite direction. Dry the rod with a vacuum or blowing dry air while holding the rod between the thumb and index finger. Make sure to dry the center of the rod as well. Handle

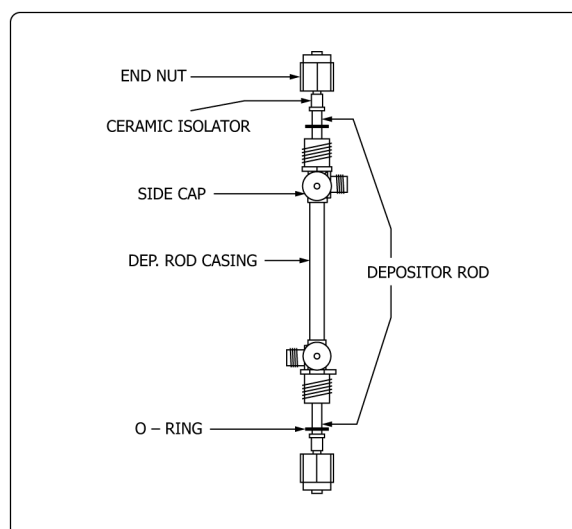


FIG. 3 Diagram of Depositor Rod Assembly

the rod as little as possible to avoid adding mass from oils on the skin and be sure not to set the rod down until after a mass is taken. Take extra care not to touch the center area of the rod where the deposits are formed. Weight the rod to $0.1 \text{ mg} \pm 0.1 \text{ mg}$ and record as the initial mass once the rod ~~weight~~ mass has come to equilibrium.

NOTE 3—Heptane may be substituted for cyclohexane throughout the test. The use of heptane will require longer drying times.

11.4 Inspect the bus bars to make sure they are clean. If not, clean the bus bars according to the operations manual. Slide the pre-weighed rod into the clean depositor rod casing with an even amount of the rod protruding from either end. Slide a new petroleum and temperature resistant O-ring over each end of the rod, and slide them up to the depositor rod casing. Place the ceramic isolators over each end of the rod with the thin portion pointing toward the end of the rod. Place the nuts on the end, and start to secure them, but do not tighten. Align the rod in the depositor rod casing until an even amount of the rod is protruding from both ends (equally spaced) or the shoulder between the deposit area and the bus bar connection areas of the rod is centered in the inlet or outlet connections of the depositor rod casing. When this is achieved, the nuts may be tightened. See Fig. 3.

11.5 Place the depositor rod casing/test rod assembly vertically in the bus bars, and slide the overtemp thermocouple in the bottom of the rod at the same time. Slide this in as far as it will go without bending it (be sure the rod has not shifted within the depositor rod casing). Make certain that the larger connection on the depositor rod casing is at the top. Connect the top end by placing the rod into the indentation of the bus bar and swinging the cap over the rod. Insert the hex screw to begin the securing process. The top of the rod should be approximately ~~2 mm~~ 2 mm above the top of the circular bus bar cap. Tighten all four hex screws. The gap in the bus bar should be the same on both sides of the depositor rod for the top and bottom bus bars. A solid contact between the rod and the bus bars is all that is required.

NOTE 3—Do not overtighten.

11.6 The open end of the pump outlet tube can now be connected to the input connection of the depositor rod casing. The drain tube may now also be connected to the outlet connection of the depositor rod casing and finger tightened. Use a wrench to tighten the end caps on the two open fronts of the depositor rod casing. The unit assembly should now be complete.

11.7 Set the pump dial to 999 once again to complete the fluid flow through the entire system. When the system is completely filled with test fluid and the oil is flowing out of the drain tube back into the reactor, set the pump dial to the setting determined from the pump speed calibration to give a flow rate of ~~0.490~~ 0.49 mL/min/min (0.40 g/min/min flow rate with TPC pump calibration fluid).

11.8 Make sure the depositor rod thermocouple is clean, and place the depositor rod thermocouple down the center of the test rod. If it is not clean, rub the thermocouple with an abrasive pad of 500 grit or finer or emery paper to remove the oxidation. A caliper may be used to ensure proper depth of the thermocouple as determined by the thermocouple depth calibration. Be careful to prevent bends in the thermocouple, which may slightly change its depth within the rod.

11.9 Turn on the main heat switch on the front panel of the instrument to start the test, and press the START/STOP button on the controller. Note that during the test the temperature excursions may differ between samples because of deposit formation. Some excursions may be in excess of 50°C .

NOTE 4—During the test, the temperature excursions may differ between samples because of deposit formation. Some excursions may be in excess of 50°C .