



Designation: D6335 – 16

# Standard Test Method for Determination of High Temperature Deposits by Thermo- Oxidation Engine Oil Simulation Test<sup>1</sup>

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 ( $\epsilon$ ) 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 (TEOST<sup>2</sup>).<sup>3</sup> An interlaboratory study (see Section 17) has determined it to be applicable over the range from 10 mg to 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 2 mg to 180 mg total deposits.

1.2 The values stated in SI units are to be regarded as standard.

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

1.2.2 *Exception*—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, 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<sup>4</sup>, n*—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 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, n*—the sleeve that surrounds the depositor rod and allows the flow of the test oil up and around the outside of the 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, n*—the tube connecting the upper outlet of the depositor rod casing to the reaction chamber.

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

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

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

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

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

2.1.10 *reactor chamber, n*—the heated reservoir that contains the bulk (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

<sup>1</sup> 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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<sup>2</sup> TEOST is a trademark of the Tannas Co. (Reg. 2001396), Tannas Company, 4800 James Savage Rd., Midland, MI 48642.

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

<sup>4</sup> 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,<sup>1</sup> which you may attend.

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

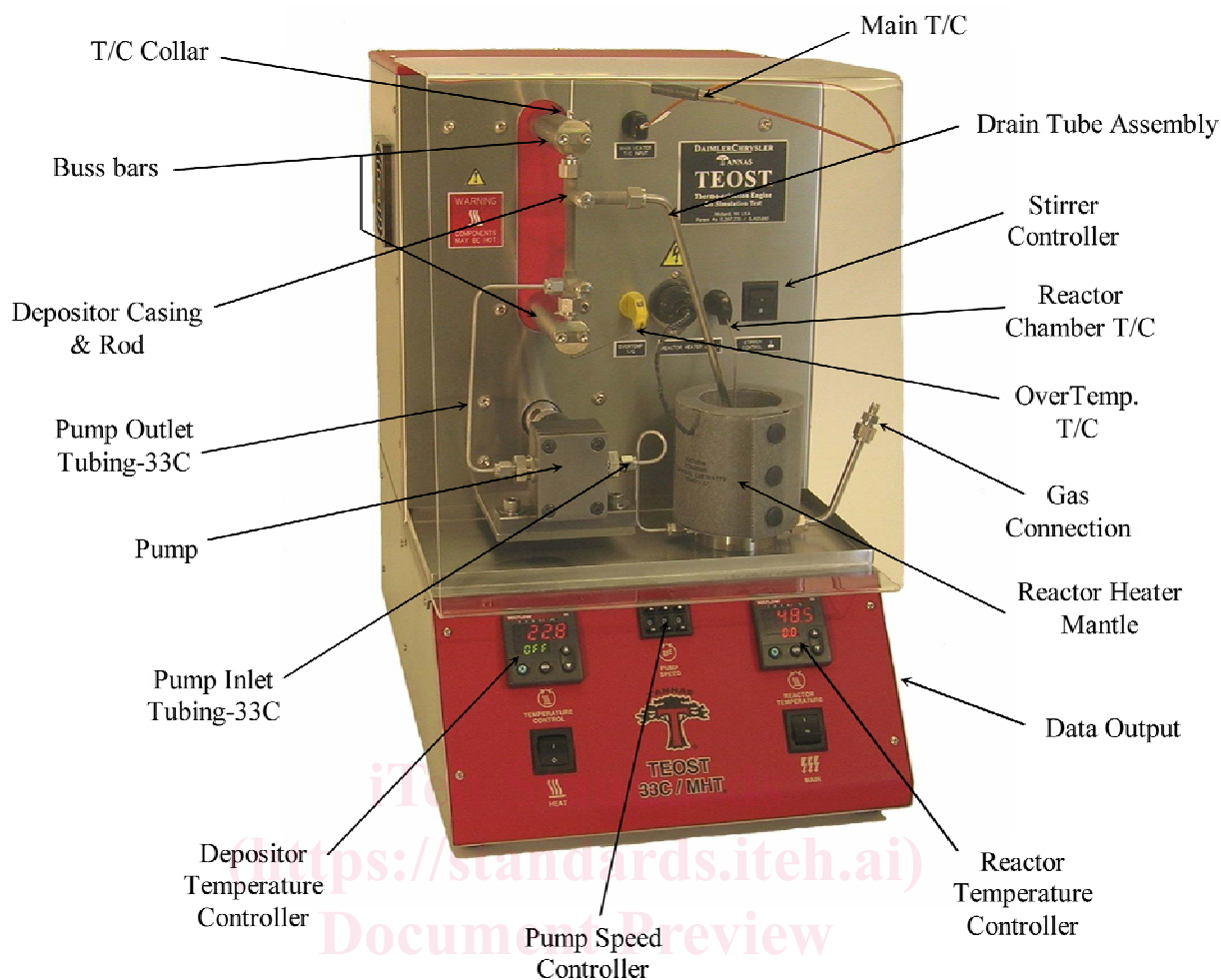


FIG. 1 Thermo-oxidation Engine Oil Simulation Test (TEOST)

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

2.1.12 *rod 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, 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, 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, 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 naphthenate is put into the reaction chamber and heated and stirred at a temperature of 100 °C. Nitrous oxide and moist air 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 that is resistively heated through twelve, 9.5 min temperature cycles that go from

200 °C to 480 °C. When the twelve-cycle program is complete, the depositor rod is rinsed of oil residue and dried and the gross rod mass obtained. The 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.*<sup>4</sup> See Fig. 1.

5.2 *Balance*, capable of weighing to the nearest 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.*

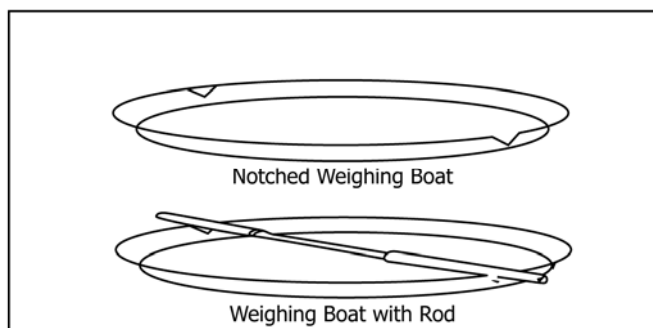


FIG. 2 Weighing Boat and Rod

5.7 *Ceramic isolators.*

5.8 *Filtering Flask*—1000 mL.

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

5.10 *Graduated Cylinder*—150 mL.

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

5.12 *Graduated Cylinder*—10 mL.

5.13 *Erlenmeyer Flask*—50 mL.

5.14 *Adjustable hex wrench.*

5.15 *Pipe Cleaners*—3 mm × 304.8 mm.

5.16 *Steel Wool*—4/0 (ultra fine).

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

5.18 *Glass Syringe*—100  $\mu$ L. (Optional—A calibrated pipet can also be used with chemically inert disposable tips.)

5.19 *One-piece disposable multi-layer cartridge filters.*<sup>4</sup>

5.20 *Flow Meters*—capable of measuring 0 mL/min to 10 mL/min of gas.

5.21 *Weighing Boat*—light, circular or oblong open container, preferably made of aluminum with a diameter or length of approximately 7 cm to 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* ( $N_2O$ )—Compressed gas cylinder, 99.6 % purity or higher.

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

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

6.4 *Cyclohexane, Heptane, or Other Alkane Hydrocarbon Solvent of Equivalent Volatility*—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 oil<sup>4</sup> is a petroleum oil capable of generating total deposits in the 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 oil<sup>4</sup> is a petroleum oil capable of generating total deposits in the 50 mg to 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.<sup>4</sup>

## 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 3.5 mL/min  $\pm$  1 mL/min and the  $N_2O$  flow shall be 3.5 mL/min  $\pm$  1 mL/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.

**TABLE 1 Temperature Program**

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

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.

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 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.

9.6 Connect the gas tubing to the reaction chamber, and set the flow meters for the moist air and N<sub>2</sub>O to 3.5 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 ± 1 mL of the fluid and pour it into a large beaker until only drips are coming from the graduated cylinder. Use a glass syringe or an optional calibrated pipet to add 193 µL ± 1 µL of the approximately 6 %

ferric naphthenate solution to the 116 mL of test fluid. The resulting concentration of iron in the test fluid will be about 100 mg/kg.

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

## 11. Procedure

11.1 When the reaction chamber is 100 °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. 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 in approximately 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 of fluid into the 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 of oil.

11.3 Use 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 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 mg and record as the initial mass once the rod mass has come to equilibrium.

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](#).