Designation: D6335 – 09

Standard Test Method for Determination of High Temperature Deposits by Thermo-Oxidation Engine Oil Simulation Test[1]

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 (TEOST[2]).[3] An interlaboratory study (see Section 17) has determined it to be applicable over the range from 10 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 to 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 (mL), and litres are the units provided, because they are an industry accepted standard.

1.2.2 *Exception*—Provided psig 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*—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*—the steel rod on which the deposits are collected. It is resistively heated through a temperature cycle during the test.

2.1.3 *depositor rod casing*—the sleeve that surrounds the depositor rod and allows the flow of specimen around the outside of the rod.

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

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

2.1.6 *filter deposits*—the mass in mg of the deposits collected on the filter cartridge.

2.1.7 *pump*—the gear pump that controls the flow rate of sample through the depositor rod casing.

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

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

2.1.10 *reactor chamber*—the 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 contents.

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

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

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

2.1.14 *thermocouple lock collar*—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*—the rod deposits plus the filter deposits.

3. Summary of Test Method

3.1 A sample of the engine oil at a temperature of 100°C that contains ferric napthenate and is in contact with nitrous oxide and moist air is pumped at a set flow rate past a tared depositor rod. The rod is resistively heated through twelve, 9.5 min temperature cycles that go from 200 to 480°C. When the

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

Current edition approved June 1, 2009. Published July 2009. Originally approved in 1998. Last previous edition approved in 2003 as D6335–03b. DOI: 10.1520/D6335-09.

[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.

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

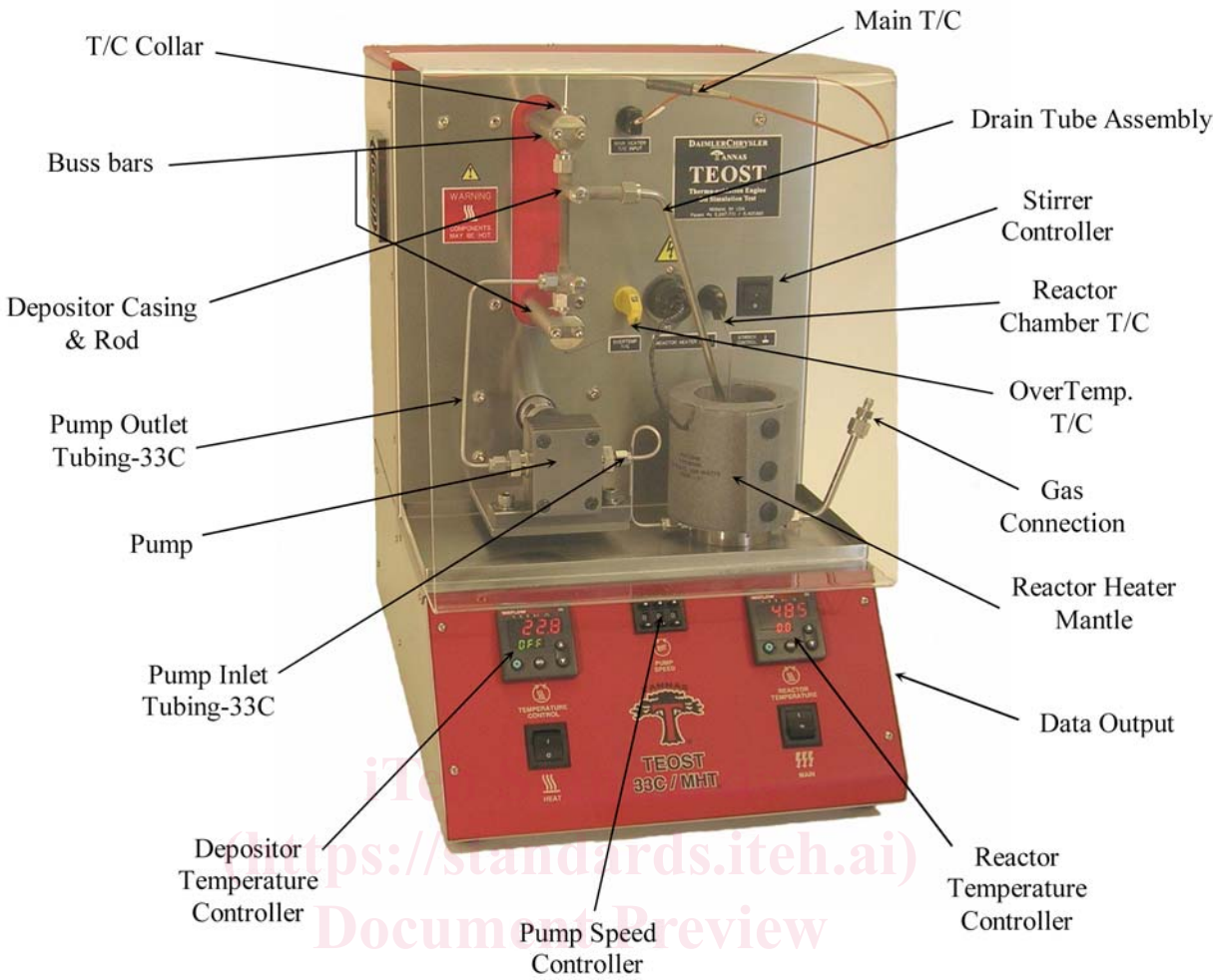

FIG. 1 Thermo-oxidation Engine Oil Simulation Test (TEOST)

twelve cycle program is complete, the depositor rod rinsed of oil residue and dried and the gross rod mass obtained. The sample 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. 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.

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.

5.11 *Filtering Flask*—1000 mL.

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

5.13 *Graduated Cylinder*—150 mL.

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

5.15 *Graduated Cylinder*—10 mL.

5.16 *Erlenmeyer Flask*—50 mL.

5.17 Adjustable hex wrench.

5.18 *Pipe Cleaners*—3 × 304.8 mm.

5.19 *Steel Wool*—4/0 (ultra fine).

[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.

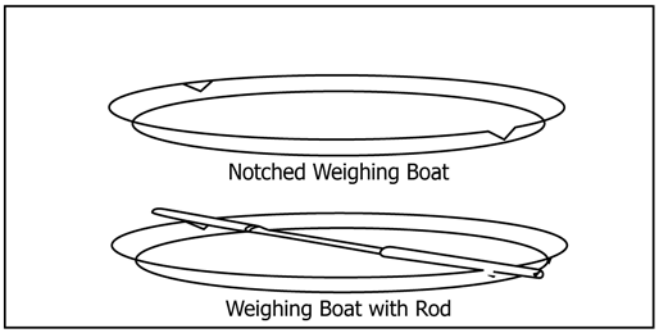

FIG. 2 Weighing Boat and Rod

5.20 *Brass Brush*—0.22 caliber.

5.21 *Glass Syringe*—100 µL.

5.22 Tannas one piece cartridge filters. (*Optional*—Items 5.8, 5.9, and 5.10 are not needed if the optional Tannas one-piece cartridge filters is used.)[4]

5.23 *Flow Meters,* capable of measuring 0 to 10 mL of air per min.

5.24 *Weighing Boat,* light, circular or oblong open container, preferably made of aluminum with a diameter or length of approximately 7 to 10 cm and notched in two diametrically opposed places to prevent the rod from rolling. (See Fig. 2.)

6. Reagents and Materials

6.1 *Nitrous Oxide* (N_2O)—USP compressed gas cylinder, medical grade.

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

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

6.4 *Cyclohexane or Heptane*—Industrial grade.

6.5 *Low Deposit Reference Oil*—CG-1 reference oil[4] is a petroleum oil capable of generating total deposits in the 20 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[4] is a petroleum oil capable of generating total deposits in the 50 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.[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.

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

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 ± 1 mL/min and the N_2O flow shall be 3.5 ± 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.

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.