

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 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.
- 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 Oct. 1, 2016. Published November 2016. Originally approved in 1998. Last previous edition approved in 2009 as D6335-09. DOI: 10.1520/D6335-16.
- 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.

- 2.1.2 depositor rod⁴, 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

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.

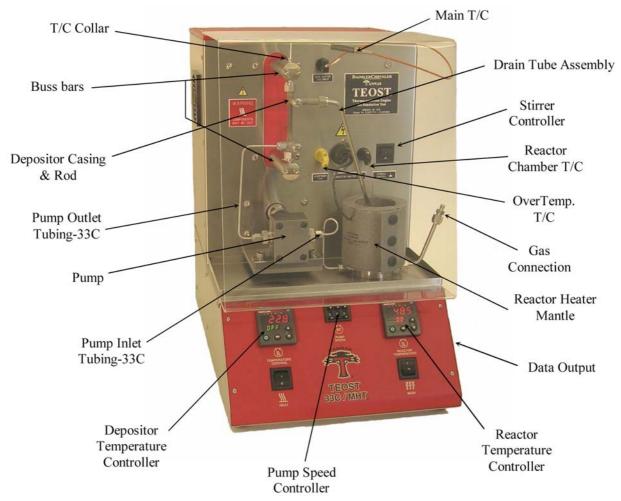


FIG. 1 Thermo-oxidation Engine Oil Simulation Test (TEOST)

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 napthenate 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.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.

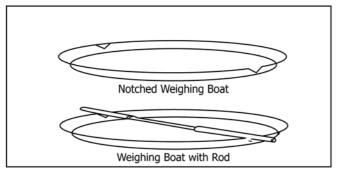


FIG. 2 Weighing Boat and Rod

- 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 \times 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.⁴
- $5.20 \; Flow \; Meters$ —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 no 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 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 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.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 3.5 mL/min \pm 1 mL/min and the N₂O 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_2O to 3.5 mL/min \pm 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 \pm 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 \pm 1 μL of the approximately 6 %

ferric napthenate 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 $100 \,^{\circ}\text{C} \pm 5 \,^{\circ}\text{C}$, 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 $100 \,^{\circ}\text{C}$ 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.

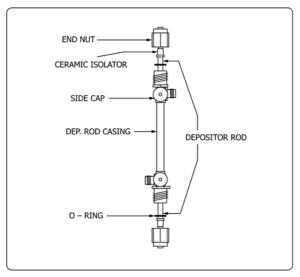


FIG. 3 Diagram of Depositor Rod Assembly

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 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 0.49 mL/min (0.40 g/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 4—During the test, the temperature excursions may differ between samples because of deposit formation. Some excursions may be in excess of $50\,^{\circ}\text{C}$.

12. Test Completion

- 12.1 Test time is 1 h, 54 min. When the test is complete, the heat function will discontinue, and the rod will go back to room temperature. Turn off the main heat switch, set the pump dial to 000, turn off the reaction chamber heater control, and remove the reaction chamber heating jacket. (Warning—The reactor and the reactor heating jacket are hot.)
- 12.2 Place a small beaker beneath the connection between the depositor rod casing inlet and pump outlet, and remove the pump outlet connection, making sure to catch any oil that drips out
- 12.3 Carefully remove the depositor rod casing drain tube and rinse it with hydrocarbon solvent into a beaker sufficient in size to accommodate the rod casing.
- 12.4 Loosen the hex screws on the bus bars. One hex screw will need to be removed on the top bus bar and the others loosened to get the depositor rod casing out of the bus bars.
- 12.5 Place the depositor rod casing in the large beaker. Remove residual oil and loose deposits by squirting hydrocarbon solvent into the casing containing the rod.
- 12.6 Remove the securing nuts, ceramic isolators and O-rings from the assembly. This should be done gently so that the deposits on the rod are not disturbed.
- 12.7 Carefully remove the depositor rod from the casing by pulling it straight out so that the deposits remain intact on the rod. Return the depositor rod casing to the large beaker.
- 12.8 Hold the end of the rod with the thumb and index finger over the beaker. Rinse the inside and outside of the rod with hydrocarbon solvent, and allow the rinsed material to drain into the beaker.
- 12.9 Fill three test tubes or similar containers with hydrocarbon solvent and let the rod soak sequentially in each test tube for approximately 10 min. It is also possible to use one test tube that is rinsed into the collection beaker between each soaking.
- 12.10 Remove the rod from the last test tube and rinse one last time with hydrocarbon solvent.
- 12.11 Pour the contents of all three test tubes into the collection beaker. Rinse the test tubes out thoroughly with hydrocarbon solvent, and drain the contents into the collection beaker.
- 12.12 After rinsing the rod, use a vacuum source to completely dry the inside of the rod.
- Note 5—Only use a vacuum source. Do not use a pressurized dry air source to dry the rod as this may blow off deposit particles.
- 12.13 Pre-weigh and tare a weighing boat and set the rod across the boat so that it does not touch the deposits. Do not set

Filtering Flask Assembly

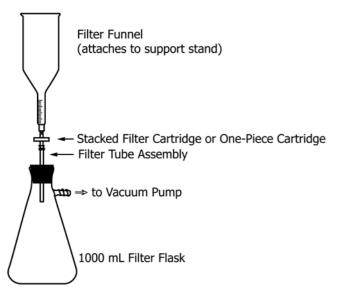


FIG. 4 Diagram of Vacuum Filtration System

the rod down before weighing. Leave the rod on the balance for a minute to make sure that all of the solvent has been removed. A loss in mass indicated that further drying is needed before a final mass can be recorded. When a constant mass is obtained, record the value.

- 12.14 Drain the rest of the oil from the reaction chamber to the large beaker. Thoroughly rinse the depositor rod casing with hydrocarbon solvent, and remove it from the beaker.
- 12.15 Disconnect the pump inlet tube from the pump but leave it connected to the reaction chamber. Allow the reaction chamber to cool, then rinse it thoroughly with hydrocarbon solvent and drain into the beaker. (**Warning**—Do not use hydrocarbon solvent when the chamber is hot. Fumes are flammable.)
- 12.16 With the 25 mL beaker from 12.2 beneath the pump outlet tube, set the pump speed to 999 and empty the oil from the tube and pump into the beaker. When the oil is completely exhausted, set the pump to 000. Remove the pump inlet tube from the reaction chamber and flush it with hydrocarbon solvent into the large beaker.
- 12.17 Add the oil in the small beaker to the contents of the large beaker, rinsing the beaker with hydrocarbon solvent.
- 12.18 Rinse the reaction chamber with hydrocarbon solvent from where the pump inlet tube was connected and drain it into the large beaker.

13. Fluid Filtering

- 13.1 Obtain a new filter cartridge and determine and record the tare mass of the fresh filter on a balance to the nearest 0.1 mg. This number will be used as the initial filter mass.
- 13.2 Connect the filter cartridge to the filter system shown in Fig. 4, turn on the vacuum source, and slowly pour the contents of the container into the glass filter funnel; draw these

contents into and through the filter cartridge where the solid contents will be trapped by the filter and the liquid contents will pass into the vacuum flask connected to the vacuum source. To ensure that no stray deposits are lost, rinse the inside of the container with hydrocarbon solvent into the funnel and through the filter cartridge.

Note 6—Do not permit the hydrocarbon solvent to remain in contact with the filter cartridge any longer than needed for flushing the hydrocarbon solvent through the filter since the plastic of the filter cartridge may absorb some mass from the hydrocarbon solvent if the two are left in contact for an extended time of 20 min or more.

13.3 If deposits from the depositor rod are lodged in the neck of the funnel above the filter, extend a wire rod (see 5.22) down from the top of the funnel to break up and/or dislodge any deposits that may have collected on the wall and wash these deposits (which may also adhere to the wire) into the filter. To avoid puncturing the layered filters, do not push the wire rod into the filter cartridge.

Note 7—Particulate deposits often adhere to the funnel outlet because of static charge; the wire rod also helps to dissipate this charge and permit the particles to travel to the filter when washed.

13.4 Rinse the sides of the funnel with about 10 mL of hydrocarbon solvent, and then add about 90 mL of the hydrocarbon solvent through the filter in three aliquots, each of 30 mL, to allow the hydrocarbon solvent to percolate slowly through the filter and solubilize any oil remaining in the deposits.

Note 8—On rare occasions, the filter may become clogged because of both numerous and very fine deposits. Under these conditions, a second or even third pre-weighed fresh filter cartridge may be used.

13.5 When filtering is completed, allow the filter cartridge(s) to dry under vacuum for 30 min and weigh. Continue drying until the cartridge(s) come to constant mass as indicated by agreement to within \pm 0.1 mg of two weighings taken 10 min apart

Note 9—If the drying time becomes lengthy, flush the filter cartridge(s) with the hydrocarbon solvent twice more with about 5 mL to $10\,\mathrm{mL}$ of hydrocarbon solvent and draw pre-warmed air by vacuum through the filter to obtain complete drying. This avoids the use of air circulating ovens, which prolong the time of bringing the filter cartridge to constant mass because of the added time to bring the filter back to ambient temperature.

14. Cleaning

- 14.1 The TEOST apparatus components need to be cleaned of the oil residue remaining from the test before another test is run. This can be done by rinsing the components with hydrocarbon solvent. If the components are to be used right away, then an air source or vacuum can be used to facilitate the drying. The reaction chamber, the pump inlet tube, the pump outlet tube, and the depositor rod casing drain tube shall all be cleaned prior to use.
- 14.2 In addition to rinsing with hydrocarbon solvent, the inner walls of the depositor rod casing shall be scrubbed to remove any deposit residues. Use a 0.22 caliber (5.588 mm) brass brush to scrub the inside of the casing, and then wash it again with hydrocarbon solvent. Be sure not to use this residue for filtering from a test. The brass bristles do break free and would give an erroneous mass in the filter.

Note 10—The brush will need to be replaced occasionally due to the wearing of the brass bristles.

- 14.3 The pump inlet and outlet tubes both have finger nuts on each end. The fittings are sealed with petroleum and temperature resistant O-rings. New O-rings should be used for every other run to ensure the seal. The depositor rod O-rings should be replaced every run.
- 14.4 Periodically remove the pump and clean in a sonic cleaner in accordance with the manufacturer's instructions.
- 14.5 When the reaction chamber becomes discolored with varnish, clean the chamber with a varnish remover to remove the stains.

15. Calculation

15.1 *Rod Deposits*—The rod deposits are obtained by subtracting the initial rod mass from the final rod mass and recording to 0.1 mg.

Rod deposits = Final rod mass
$$-$$
 Initial rod mass (1)

15.2 Filter Deposits—The filter deposits are obtained by subtracting the initial filter mass from the final filter mass and recording to 0.1 mg.

Filter deposits = Final filter mass
$$-$$
 Initial filter mass (2)

15.3 *Total Deposits*— The sum of rod and filter deposits equal the total deposits.

Total deposits = Rod deposits + Filter deposits
$$(3)$$

16. Report

16.1 Report the filter deposit mass, the rod deposit mass, and the total deposit mass, all to the nearest 0.1 mg.

17. Precision ⁵

17.1 Interlaboratory study test results are shown in Table 2.

TABLE 2 Interlaboratory Test Results

Oil Sample	Average Total Deposit (mg)	Repeatability (mg)	Reproducibility (mg)
AROP 124	51.79	9.90	13.40
AROP 125	26.72	8.46	9.68
AROP 126	10.75	7.14	11.01
AROP 127	64.63	10.66	23.30
AROP 128	48.13	12.03	20.36
AROP 130	40.23	12.04	13.13

- 17.2 The precision of this test method as determined by the statistical examination of the interlaboratory study test results is as follows:
- 17.2.1 Repeatability— The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

Repeatability = $3.058 (X^{0.33})$, where X is the mean of the two values

17.2.2 *Reproducibility*— The difference between two single and independent results, obtained by different operators working in different laboratories on identical test materials, would, in the long run, in the normal and correct operation of the test method, exceed the following values in only one case in twenty:

Reproducibility = $4.774 (X^{0.33})$, where X is the mean of two values

18. Keywords

18.1 deposits; engine oil; oxidation; pyrolysis; TEOST; thermal decomposition; thermal oxidation; turbocharger

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D6335 – 09) that may impact the use of this standard. (Oct. 1, 2016.)

- (1) Subcommittee D02.09 has re-edited the method on the basis of user experience with the goal of generally simplifying and clarifying the content of the standard. A few more specific locations of changes to this standard are as follows.
- (2) Modified language in subsections 2.1.10 and 12.14 when referring to drain valve and gas inlet valve of the reaction chamber due to different fabrication period of equipment, and added subsection 2.1.10.1 to clarify the channel used to introduce gases to the reaction chamber and the use of this channel to drain the reaction chamber after test to collect loose deposit particles in the test oil.
- (3) Added a calibrated pipet as an optional device to use in subsection 5.18.
- (4) Added a wire rod in subsection 5.22 to aid in filtering.
- (5) Eliminated former subsections 5.8, 5.9, and 5.10 to reduce the potential of errors associated with self-constructed multi-layer filters.
- (6) Statement in subsection 6.1 has been changed from requiring medical grade nitrous oxide to 99.6% purity or better.
- (7) Included a warning note for flammable solvent in subsection 6.4. Clarified subsection 6.4 in regards to the purity of hydrocarbon solvents allowed for use. Also, added a note

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:D02-1391.



referring cyclohexane and other alkane hydrocarbon solvent as hydrocarbon solvent instead.

- (8) Added subsection 6.4.1 on the drying time and the purity of different solvents and removed former Note 3 since this item is referred to in 6.5.
- (9) Inserted Note 5 to warn against using pressurized air to dry the solvent-moist depositor rod.
- (10) Modified Section 13 for clarity in filtering.
- (11) Updated Eq 2 in subsection 15.2 to refer "postclean filter mass" as "initial filter mass" instead.
- (12) Made various minor changes for clarity in descriptions and techniques.

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