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

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.09.0G 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.

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

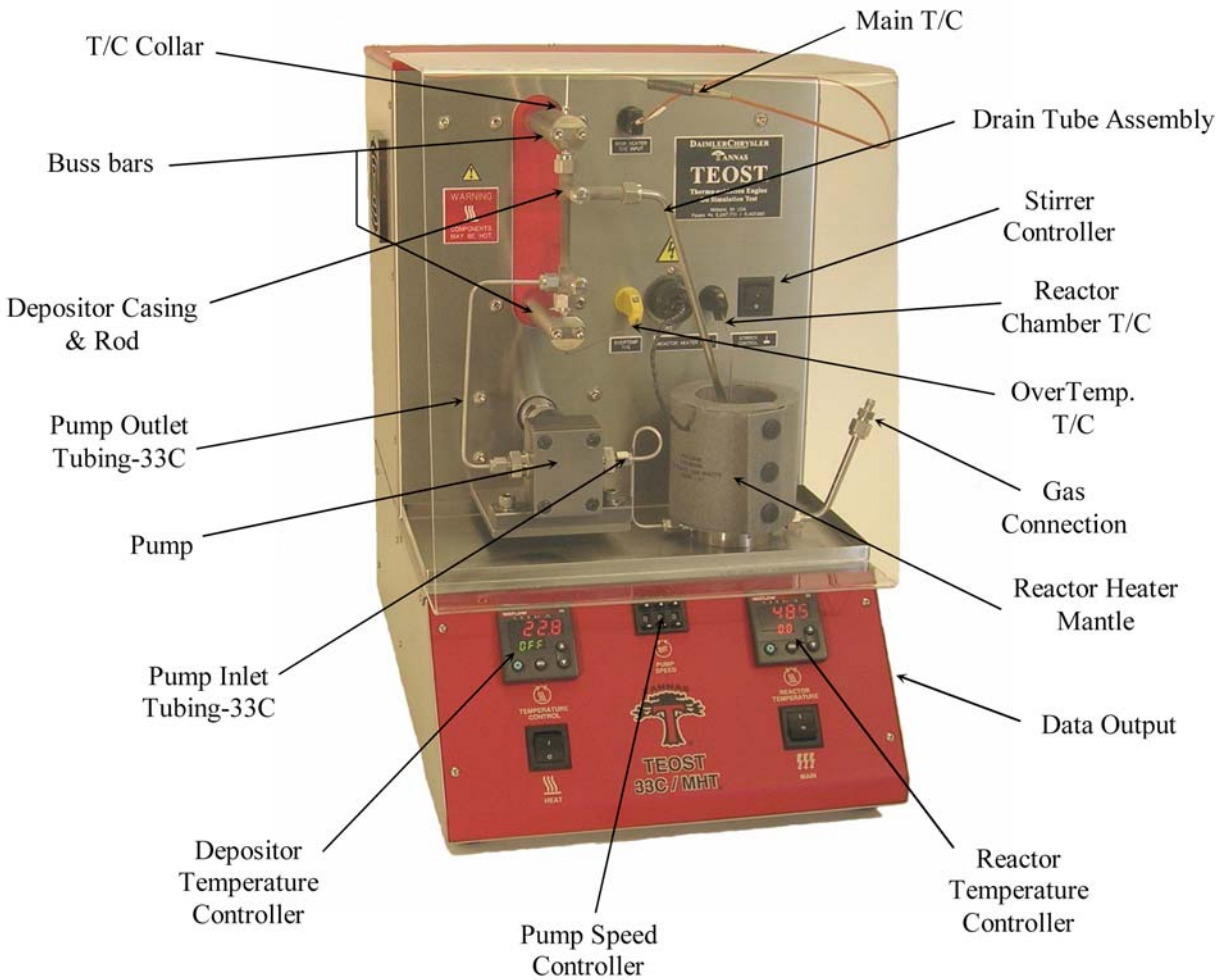

FIG. 1 Thermo-oxidation Engine Oil Simulation Test (TEOST)

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).

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.

[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.

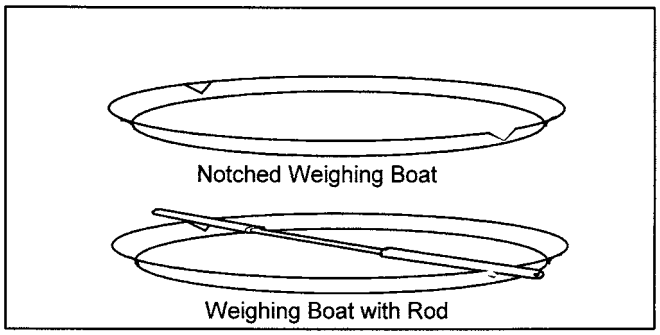

FIG. 2 Weighing Boat and Rod

5.24 *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.

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*.

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

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 ± 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 ± 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 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 wt ppm.

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 hte 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 ± 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 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 cyclohexane or heptane to rinse off an unused rod, both on the outside and down the center. Clean each of the three sections of the rod 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 weight 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

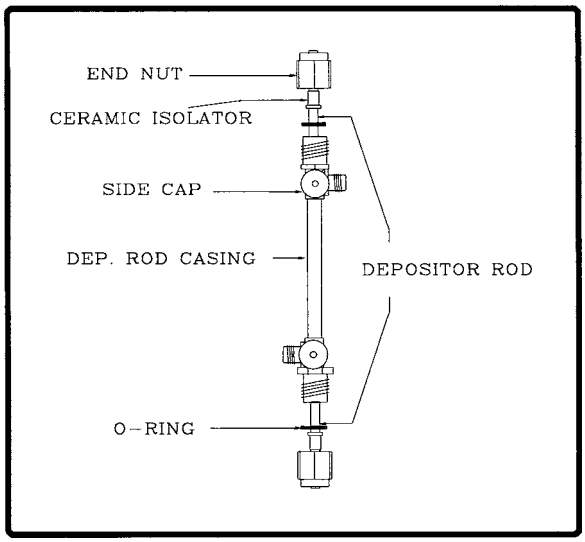

FIG. 3 Diagram of Depositor Rod Assembly

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 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 4—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 that during the test the temperature excursions may differ between samples because of deposit formation. Some excursions may be in excess of 50°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 cyclohexane or heptane 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 cyclohexane or heptane 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 cyclohexane or heptane, and allow the rinsed material to drain into the beaker.

12.9 Fill three test tubes or similar containers with cyclohexane or heptane 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 cyclohexane or heptane.

12.11 Pour the contents of all three test tubes into the collection beaker. Rinse the test tubes out thoroughly with cyclohexane or heptane, 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—Do not use a dry air source to dry the rod.

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 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 Place the large beaker under the drain pipe of the reaction chamber, and turn the valve to allow the oil in the reaction chamber to drain. Thoroughly rinse the depositor rod casing with cyclohexane or heptane, and remove it from the beaker.

12.15 Disconnect the pump inlet tube from the pump but leave is connected to the reaction chamber. Allow the reaction chamber to cool, then rinse it thoroughly with cyclohexane or heptane and drain into the beaker. (**Warning—**Do not use cyclohexane or heptane 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 cyclohexane or heptane into the large beaker.

12.17 Add the oil in the small beaker to the contents of the large beaker, rinsing the beaker with cyclohexane or heptane.

12.18 Rinse the reaction chamber with cyclohexane or heptane from where the pump inlet tube was connected and drain it into the large beaker.

13. Fluid Filtering

13.1 To assemble the filter cartridge, stack five polypropylene disks in the plastic filter holder, screw the top and bottom parts together, and tighten to seal. The use of five disks compensates for the gaps and passages that may occur in the filters.

NOTE 6—Optional one-piece filter cartridges[4] may be used. If so, start at 13.2.

13.2 Record the tare mass of the filter cartridge as the preclean filter mass. Attach the top of the filter cartridge to the Luer lock connection of the filter funnel and the bottom to the filter flask. Connect the vacuum source to the side arm of the filter flask. The vacuum should be off. See Fig. 4.

13.3 Pour 5 mL of cyclohexane or heptane through the system ten times (a total of 50 mL) to remove any solubles from the filters. It may be necessary to apply vacuum briefly to break the vapor lock. Turn the vacuum on and allow the filters to dry for 15 min.

13.4 Weigh the filter cartridge. Repeat drying and weighing the filter cartridge until constant mass is reached. Record this mass as the postclean filter mass.

13.5 When the postclean filter weight is established, connect the filter cartridge to the filter system again, and slowly pour the contents of the large beaker through the filter with the

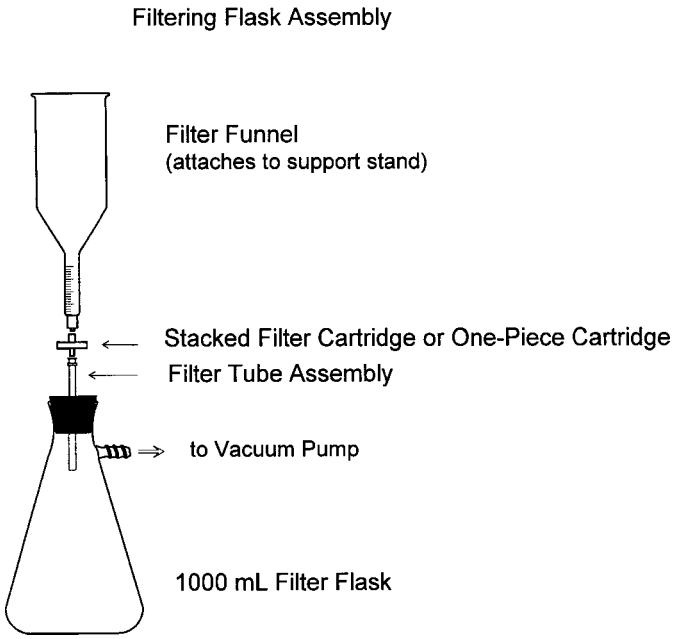

FIG. 4 Diagram of Vacuum Filtration System

vacuum on. Rinse down the sides of the funnel with about 10 mL of cyclohexane or heptane, and then add 100 mL of cyclohexane or heptane through the filters in 10-mL aliquots. This should be done with the vacuum off to allow the cyclohexane or heptane to percolate through the filters and ensure a thorough rinse. Once again, turn the vacuum on briefly to remove vapor lock and to keep the cyclohexane or heptane flowing through the filters.

13.6 When filtering is complete, allow the filter cartridge to air dry for 15 min.

13.7 Remove the filter cartridge from the filter assembly and weigh. Return the cartridge to the filter assembly and dry for 5 min more and reweigh. Repeat until the mass of the cartridge is constant. Record this mass as the final filter mass.

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 cyclohexane or heptane. 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 cyclohexane or heptane, the inner walls of the depositor rod casing shall be scrubbed to remove any deposit residues. Use a .22 caliber brass brush to scrub the inside of the casing, and then wash it again with cyclohexane or heptane. Be sure not to use this residue for filtering from a test. The brass bristles do break free and would give an erroneous weight in the filter.

NOTE 7—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.

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

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.

$$\text{Rod deposits} = \text{Final rod mass} - \text{Initial rod mass} \quad (1)$$

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

$$\text{Filter deposits} = \text{Final filter mass} - \text{Postclean filter mass} \quad (2)$$

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

$$\text{Total deposits} = \text{Rod deposits} + \text{Filter deposits} \quad (3)$$

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.

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:

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

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,

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

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

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D6335–03b) that may impact the use of this standard.

(*1*) Changed the solvent from hexane to cyclohexane and heptane.
(*2*) Added oil and catalyst tolerances into Section 10.
(*3*) Modified Section 11 for clarity and added the procedure for cleaning of the thermocouple. Also added stirring instructions so that a vortex is not formed during the mixing causing air to be drawn into the system.
(*4*) Added a statement about not filtering after using the brass brush and that the pump must be periodically cleaned to Section 14.