



Standard Test Method for Determination of Moderately High Temperature Piston Deposits by Thermo-Oxidation Engine Oil Simulation Test— TEOST MHT¹

This standard is issued under the fixed designation D7097; 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 mass of deposit formed on a specially constructed test rod exposed to repetitive passage of 8.5 g of engine oil over the rod in a thin film under oxidative and catalytic conditions at 285 °C. The range of applicability of the Moderately High Temperature Thermo-Oxidation Engine Test (TEOST MHT²) test method as derived from an interlaboratory study is approximately 10 mg to 100 mg. However, experience indicates that deposit values from 1 mg to 150 mg or greater may be obtained.

1.2 This test method uses a patented instrument, method and patented, numbered, and registered depositor rods traceable to the manufacturer³ and made specifically for the practice and precision of the test method.⁴

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

1.3.1 Although not an SI unit, the special name liter (L) is allowed by SI for the cubic decimeter (dm³) and the milliliter (mL) for the SI cubic centimeter (cm³). Likewise, the special name millimeter (mm) is allowed by SI as a measurement of length.

1.4 *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. Referenced Documents

2.1 *ASTM Standards:*⁵

[D4485 Specification for Performance of Active API Service Category Engine Oils](#)

[D6335 Test Method for Determination of High Temperature Deposits by Thermo-Oxidation Engine Oil Simulation Test](#)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *bubble airflow gauge, n*—a precision bore glass tube marked in tenths of a milliliter used to measure accurately the flow rate of air around and past the depositor rod and to calibrate mass air flow controllers recommended for use in the procedure.

3.1.2 *depositor rod deposits, n*—particulate matter formed on the depositor rod surface by oxidation of the thin film of passing oil exposed to the rod temperature and air, and weighed after appropriate washing and drying to obtain the net mass gain.

3.1.3 *filter deposits, n*—particulates washed from the depositor rod after the test and collected on a special multi-layer filter cartridge.

3.1.4 *TEOST², n*—an acronym for Thermo-Oxidation Engine Oil Simulation Test.

3.1.5 *total rod deposits, n*—the mass of deposits collected on the depositor rod plus any mass of deposits washed from the depositor rod and later extracted on a filter.

3.1.6 *volatilized oil, n*—oil vapor coalesced on the mantle wall, and subsequently collected in a vial.

3.2 *Abbreviations:*

⁵ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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 and MHT are registered trademarks of the Tannas Co. (Reg. 2001396), Tannas Company, 4800 James Savage Rd., Midland, MI 48642.

³ The sole source of supply of the apparatus known to the committee at this time is Tannas Company, 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.

⁴ The TEOST instrument, method and rod are patented. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented technology to ASTM 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

3.2.1 *MHT*², *n*—moderately high temperature.

3.2.1.1 *Discussion*—The TEOST MHT procedure evaluates deposit formation at temperatures that are closely related to those of the piston ring zone in reciprocating engines (as distinguished from the much higher temperatures associated with the TEOST 33C, Test Method **D6335**, procedure for determining potential deposits in turbochargers).

4. Summary of Test Method

4.1 Deposit-forming tendencies of an engine oil under oxidative conditions are determined by circulating an oil-catalyst mixture comprising a small sample (8.4 g) of the oil and a very small (0.1 g) amount of an organo-metallic catalyst. This sample mixture is then circulated for exactly 24 h in the TEOST MHT instrument over a special wire-wound depositor rod heated by electrical current to a controlled temperature of 285 °C at the hottest location on the rod. The depositor rod is weighed before and after the test and any deposit formation on the rod as well as any deposits collected from rod washings are determined. During the test, precisely controlled and directed air is caused to bathe the oil flowing down the depositor rod and, thereby, to provide opportunity for oxidation. Precision of the test is strongly influenced by the care in manufacture of the wire-wound steel depositor rods and the treatment of the coating of the wound wire, the rate of air flow, and the amount and degree of mixing of the catalyst.

5. Significance and Use

5.1 The test method is designed to predict the deposit-forming tendencies of engine oil in the piston ring belt and upper piston crown area. Correlation has been shown between the TEOST MHT procedure and the TU3MH Peugeot engine test in deposit formation. Such deposits formed in the ring-belt area of a reciprocating engine piston can cause problems with engine operation and longevity. It is one of the required test methods in Specification **D4485** to define API Category-Identified engine oils.⁶

6. Apparatus

6.1 *TEOST MHT Instrument*,³ with specific fittings for the MHT procedure including parts and assemblies are as follows:

6.1.1 *Depositor Rod Casing Assembly*:

6.1.1.1 *Ceramic Isolators*, special non-conductive fittings that compress the depositor rod O-rings into the end-caps and centers the depositor rod in the end-caps to prevent leakage of oil from the lower end-cap. (See **Figs. 4 and 5**.)

6.1.1.2 *Depositor Rod, Wire-Wound*, a specially patented, numbered, and registered steel tube wound with pretreated steel wire. The steel tube is formed to a selected interior diameter to precisely contact the surface of a metal-sheathed thermocouple. The registered depositor rods are required to run the TEOST MHT procedure. (See **Fig. 4, Fig. 5, and Fig. 7**.)

NOTE 1—Precision of the TEOST MHT procedure is highly dependent

⁶ Selby, T. W., and Florkowski, D. F., “The Development of the TEOST Protocol MHT Bench Test of Engine Oil Piston Deposit Tendency,” *Supplement to the Proceedings of the 12th Esslingen Colloquium*, Esslingen, Germany, January 11-13, 2000, pp. 55-62.

on the uniformity of manufacture and use of patented and registered depositor rods. Each depositor rod is numbered and traceable to the manufacturer and raw steel tubing mill.

6.1.1.3 *End-cap, Upper*, holds the upper end of the glass mantle and depositor rod in place and allows air and oil to enter the deposit-forming zone separately. (See **Fig. 4 and Fig. 7**.)

6.1.1.4 *End-cap, Lower*, holds the lower end of the glass mantle and depositor rod in place and provides an outlet for the oil to pass into the sample flask and subsequently to the recirculating pump inlet tubing. (See **Fig. 6**.)

6.1.1.5 *End-cap Nuts, Four*, used for compressing small O-rings around depositor rod and for positioning and sealing the oil feed tube and sealing the air inlet tubing. (See **Fig. 4 and Fig. 5**.)

6.1.1.6 *Glass Mantle*, the glass casing that surrounds the depositor rod and diverts volatilized oil into a collecting vial. (See **Figs. 4-6**.)

6.1.1.7 *Mantis Clip*, a wire-spring device holding the sample flask in place on the lower end-cap. (See **Fig. 2 and Fig. 6**.)

6.1.1.8 *Lower End-cap Seal*, a flexible oil temperature resistant rubber seal (see **Fig. 9**).

6.1.1.9 *Oil Feed Tube*, the avenue for oil to be delivered from the pump to the top of the depositor rod.

6.1.1.10 *End cap O-rings, Large, Petroleum-resistant*, create a seal between the end-caps and glass mantle. (See **Fig. 5**.)

6.1.1.11 *End cap O-rings, Small, Petroleum- and Heat-resistant*, creates an air and fluid seal between depositor rod and end-caps. (See **Fig. 5**.)

6.1.1.12 *Pump Outlet Tubing*, a flexible transparent vinyl tube of 3.2 mm outer diameter with a flared end used to transport the oil sample from the oil pump to the oil feed tube. (See **Fig. 6**.)

6.1.1.13 *Sample Flask*, a small (~25 mL), modified form of an Erlenmeyer flask with sidearm into which the catalyst and sample are first weighed, then later used to feed the sample to the circulating system. (See **Fig. 2 and Fig. 6**.)

6.1.1.14 *Stainless Steel Hex Screws and Busbar End Piece*, these secure the depositor rod to the busbars.

6.1.1.15 *Thermocouples, Two*, stainless steel sheathed, 1.57 mm diameter by 150 mm length. One, a J-type, is used for controlling the test temperature (depositor rod) while the other, a K-type, is used to protect against an over-temperature condition.

6.1.1.16 *Thermocouple Locking Collar*, a fitting that can be tightened on the thermocouple to ensure the thermocouple tip is at the correct position when placed inside the depositor rod. (See **Fig. 4**.)

6.1.1.17 *Volatiles Vial Clip*, the device that holds the volatiles collection vial in place on the mantle. (See **Fig. 4**.)

6.1.2 *Airflow Control Assembly*, sets air flow at chosen flow rate.

6.1.2.1 *Bubble Airflow Gauge*, a device for precisely establishing the airflow rate and calibrating the flow meter from 1 mL/min to 30 mL/min. (See **Fig. 1**.)

6.1.2.2 *Calibrated Flow Meter*, capable of measuring approximately 1 mL/min to 20 mL/min of air and providing a continuous reading on airflow rate when calibrated.

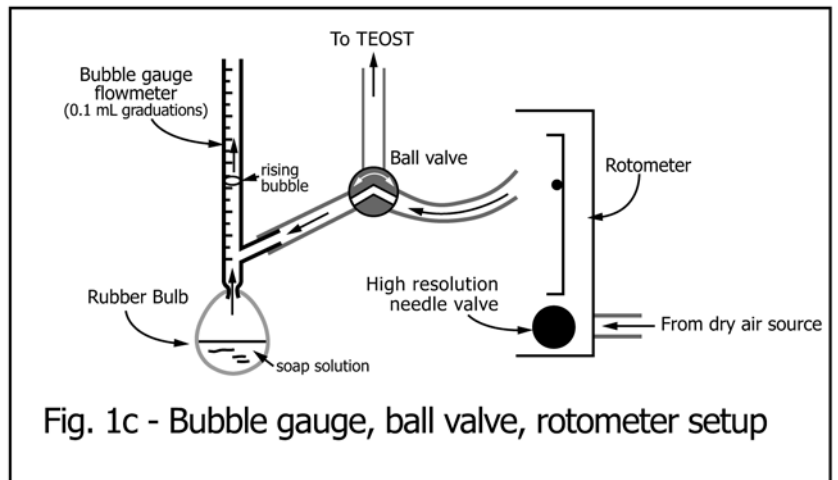
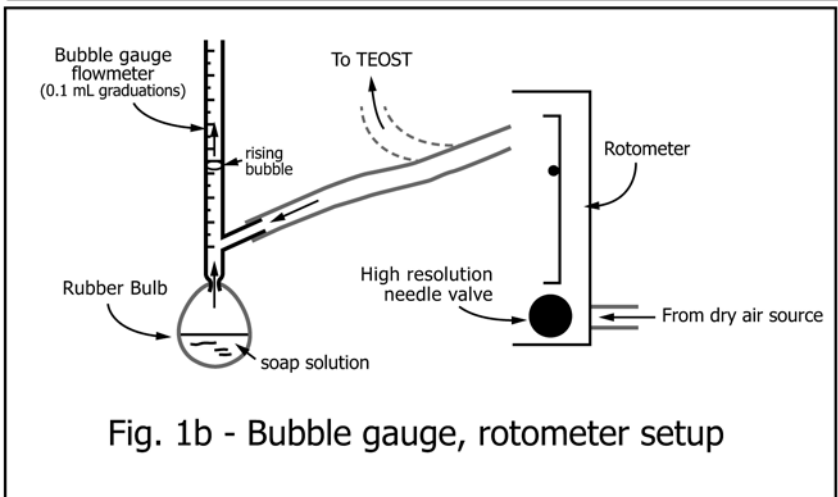
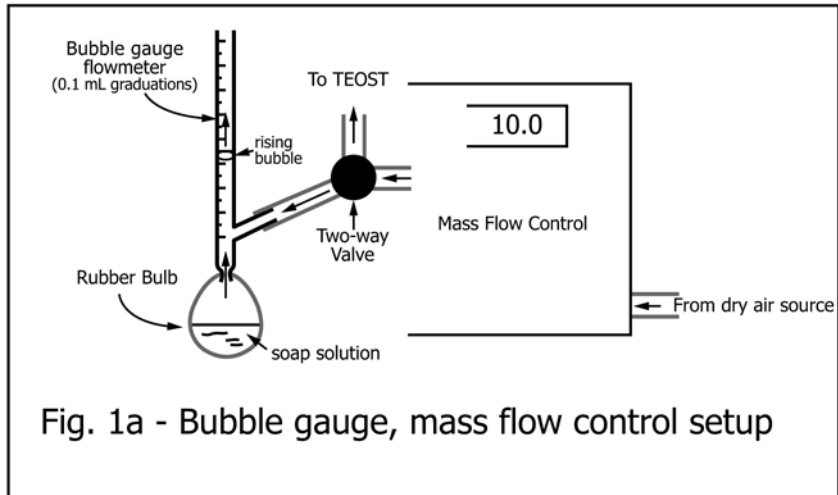


FIG. 1 Bubble Gauge

6.1.2.3 *Handheld Digital Flow Meter*, an optional device to monitor air flow to or out of the mantle, capable of reading a flow rate of 10.0 mL/min \pm 0.1 mL/min of air.

6.1.2.4 *Precision Digital Mass Flow Controller*, an optional device that allows the precise control of the input air flow. (See Fig. 1a.)

6.1.2.5 *Stopwatch*, reading to 1/100 s.

6.1.3 *Filtering Flask Assembly*, provides the means for filtering particles washed from the depositor rod. (See Fig. 8.)

6.1.3.1 *Filter Cartridge*, a special multilayer filter made for the TEOST MHT procedure fitting the end of the filter funnel also made for the TEOST procedure. (See Fig. 8.)

6.1.3.2 *Filter Funnel*, a special combination funnel of ~400 mL capacity, necking down to a 10 mL graduated or

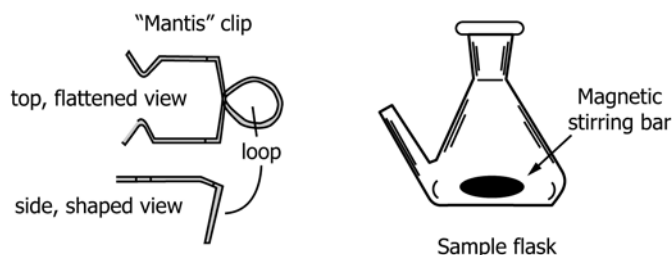


FIG. 2 Sample Flask with Stirring Bar and Mantis Clip

non-graduated section that, in turn, ends in a glass or Luer-lock tip fitting the special filter cartridge used in the procedure. (See Fig. 8.)

6.1.3.3 *Filter Tube Assembly*, a metal or polyethylene tube inserted through a No. 8 rubber stopper in the vacuum flask to fit the lower outlet of the filter cartridge. (See Fig. 8.)

6.1.3.4 *Vacuum Flask*, 1000 mL capacity for collecting the hydrocarbon solvent and oil during the filter rinse.

6.1.3.5 *Vacuum Source*, a vacuum source sufficient to draw the hydrocarbon solvent and oil through the filter and provide the necessary filter drying.

6.1.3.6 *Wire Rod*, a thin, clean, stainless steel wire rod, for dislodging any deposits trapped in the narrow portion of the filter funnel just above the filter.

6.2 *Ancillary Equipment*, needed or helpful:

6.2.1 *Balance*, capable of weighing deposits to the nearest 0.1 mg with a minimum capacity of 100 g.

6.2.2 *Catalyst Syringe*, a small glass syringe that uses either a glass or PTFE plunger (do not use rubber plunger) of 100 μ L capacity, for carefully metering the catalyst being weighed into the sample flask. (An optional approach is to use a small disposable glass pipet.)

6.2.3 *Oil Sample Transfer Pipettes*, disposable glass or plastic pipettes or droppers.

6.2.4 *Oil Extraction Test Tubes*, three glass test tubes of sufficient height to cover all but the upper 20 mm of an inserted deposit-carrying rod. Plastic tubes are not acceptable.

6.2.5 *Temperature Recorder*, an optional device for tracking the temperature of the upper depositor rod thermocouple over the 24 h period of the test.

6.2.6 *Thermocouple Depth Insertion Gauge*, an optional measurement device fabricated for simple setting and checking of the thermocouple insertion depth, using a millimeter graduation scale.

6.2.7 *Vials and Caps*, a vial and matching cap of 10 mL or more in volume with an 11.5 mm diameter mouth and an outer diameter of 20 mm to collect the volatile material emitted by the oil and collected on the mantle wall during the test as well as the recovered, end-of-test oil sample. (See Fig. 4.)

6.2.8 *Weighing Boat*, a light, circular or oblong weighing container, preferably made of aluminum with a diameter or length of 7 cm to 10 cm and notched in two diametrically opposed places to prevent the rod from rolling. (See Fig. 3.)

6.2.9 *Air-Flow Restrictor*—a small PTFE washer designed to limit the amount of air allowed to pass between the sample flask and the drain on the lower end-cap.

7. Reagents and Materials

7.1 *Abrasive Paper*, 800-grit emery (aluminum oxide).

7.2 *Acetone*, particle-free, reagent grade, for final cleaning of new depositor rods. (**Warning**—Flammable. Health hazard.)

7.3 *Air*, oil-free, clean, and dry, obtained from cylinder gas or house line, regulated to 15 kPa to 100 kPa (2 psi to 15 psi) at more than 690 kPa (100 psi).

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

7.4.1 The volatility of the cyclohexane as the solvent ensures timely evaporation of the deposits on the rod and filter. If another alkane hydrocarbon is used as the solvent, longer drying times may be required. The higher the purity of the solvent, the quicker the solvent should evaporate.

7.5 *Catalyst*³—Catalyst contains iron, lead, and tin in ratios chosen for emulating engine deposit conditions.

7.5.1 For long term storage, it is acceptable to refrigerate the catalyst until a few hours before use (let catalyst warm to room temperature before opening to eliminate condensation). Temporary unopened storage, up to four weeks, may be at room temperature.

7.6 *Certified Reference Oils*,³ certified low deposit fluid (LDF, about 10 mg to 15 mg), medium deposit fluid (MDF, about 40 mg to 50 mg), and high deposit fluid (HDF, about 70 mg to 90 mg).

7.7 *Combination Pump Calibration and Temperature Control Thermocouple Depth Setting Oil, TPC-1*,³ a highly deposit-resistant oil used in setting pump calibration and temperature control calibration without forming significant deposits on the depositor rod during these calibrations.

7.8 *Varnish Cleaning Liquid*, used in cleaning varnish from mantle, end-caps, and other components of the equipment after test. Other glass cleaners with varnish removing capabilities also may be used.

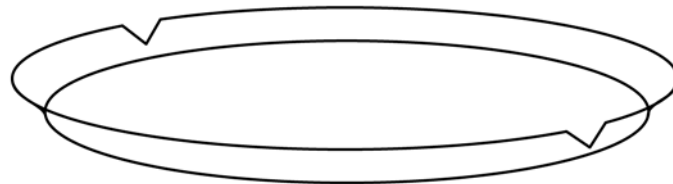
8. Programming the Apparatus

8.1 *PID (proportional, integral, and derivative) Settings for Temperature Control*—In order for the thermocouple sensitivity and response values (PID settings) to have the minimum excursion from the temperature value desired during operation set them to the following settings: Pb 160 Re: 1.0 Ra 0.1. See Instrument Manual³ for more details on the adjustment technique.

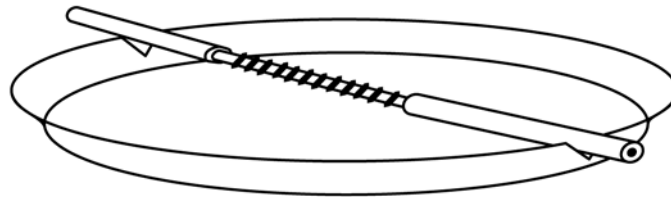
8.2 *Temperature Controller Setting*—Set the temperature control program to maintain 285 °C for 24 h according to the instructions in the Instrument Manual.

8.3 If using a strip chart recorder, turn on the strip chart, set the chart speed to 10 mm/h, but do not lower the pen(s) or turn on the chart drive at this time.

8.4 If using other means of continuously recording temperatures, prepare these for receiving information.



Notched Weighing Boat



Weighing Boat with Rod

FIG. 3 Weighing Boat and Rod

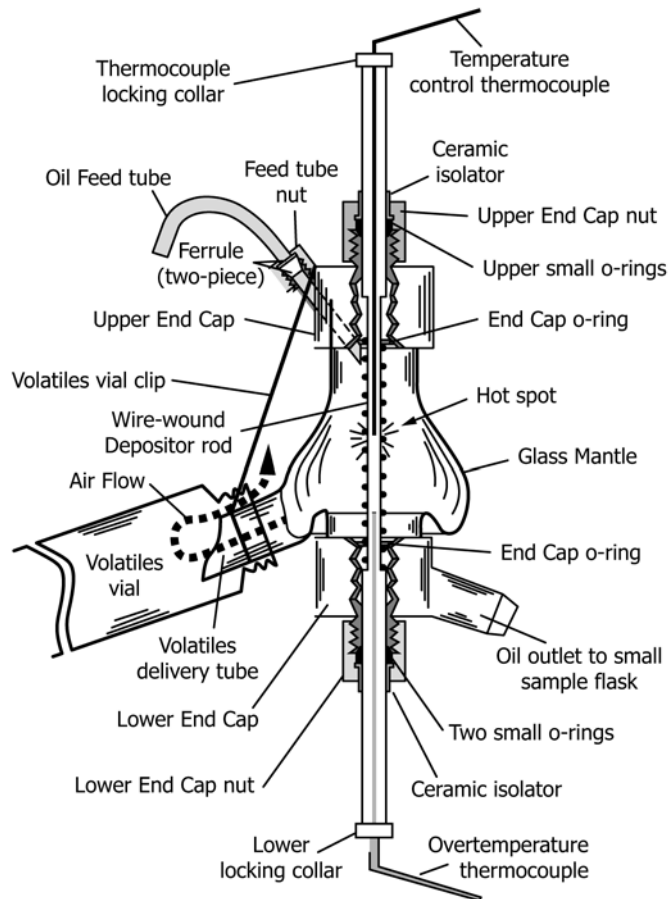


FIG. 4 Depositor Assembly (Cut-away View)

9. Calibration and Standardization

9.1 Calibration of Air Flow Rate (alternative procedures, follow 9.1.1 and 9.1.2):

9.1.1 Calibration of Air Flow Rate Using a Mass Flow Controller:

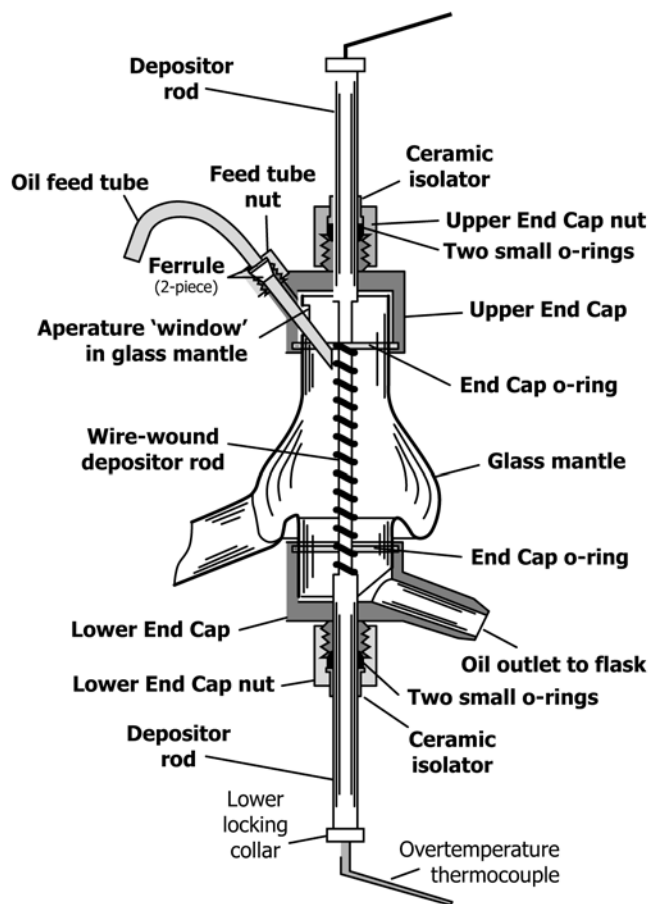


FIG. 5 Insertion of Oil Inlet Tube (Cut-away View)

9.1.1.1 Use the bubble gauge or other primary calibration device before each test to check or calibrate a mass flow controller.

(1) The TEOST MHT protocol is sensitive to flow rate, therefore primary calibration of mass flow meters or other forms of air flow control such as analog or digital flow meters is desired to ensure proper flow rate.

NOTE 2—Models of some mass flow meters may permit adjustment of the readout to the calibration value when the appropriate air flow is reached.

9.1.2 Calibration of Air Flow Rate Using an Air Flow Meter:

9.1.2.1 Use the bubble gauge or other primary calibration device before each test to calibrate analog or digital flow meters (see Note 3).

9.1.2.2 Set up the bubble gauge and flow meter equipped with a fine needle valve as shown schematically in Fig. 1a with a three-way stopcock or Fig. 1c with a ball valve.

NOTE 3—A handheld flow meter may also be used.

9.1.2.3 Connect the dry air source to the flow meter and set the source's regulator to a pressure value no greater than allowed by the tolerances of the flow meter used.

9.1.2.4 Insert the end of the air inlet tube into the soft rubber tubing attached to the bubble gauge and check that the joint is leak tight with soap solution.

9.1.2.5 Adjust the flow meter rate and retest bubble rise rate to bring the flow rate to $10.0 \text{ mL/min} \pm 0.2 \text{ mL/min}$.

9.2 Oil Pump Rate Calibration—Follow the technique in the manufacturer's Instrument Manual to set a test oil flow rate of $0.25 \text{ g/min} \pm 0.01 \text{ g/min}$.

9.3 Temperature Controller Setting—Follow the technique in the manufacturer's Instrument Manual to set a test temperature of $285 \text{ }^\circ\text{C}$ on the temperature controller.

9.4 Calibration of Control Thermocouple:

9.4.1 Calibrate the depositor rod temperature control thermocouple in a liquid or sand bath maintained at $285 \text{ }^\circ\text{C} \pm 50 \text{ }^\circ\text{C}$ and, if necessary, adjust the temperature offset of the temperature controller to match the bath temperature for this thermocouple. In the absence of either a liquid, block or sand bath, boiling distilled water may be used to calibrate at $100 \text{ }^\circ\text{C}$. The temperature should be able to be calibrated within $\pm 0.1 \text{ }^\circ\text{C}$.

9.4.2 Before each reuse of the thermocouple, clean any corrosion or other deposits from the thermocouple surface using a fine abrasive pad (500 grit or finer) or emery paper (800 grit). The resulting cleaned surface shall show bright metal, particularly in the temperature-sensitive area at the end of the thermocouple. Do not overclean the thermocouple surface or use coarse abrasives, as the thermocouple wall could be thinned and damaged.

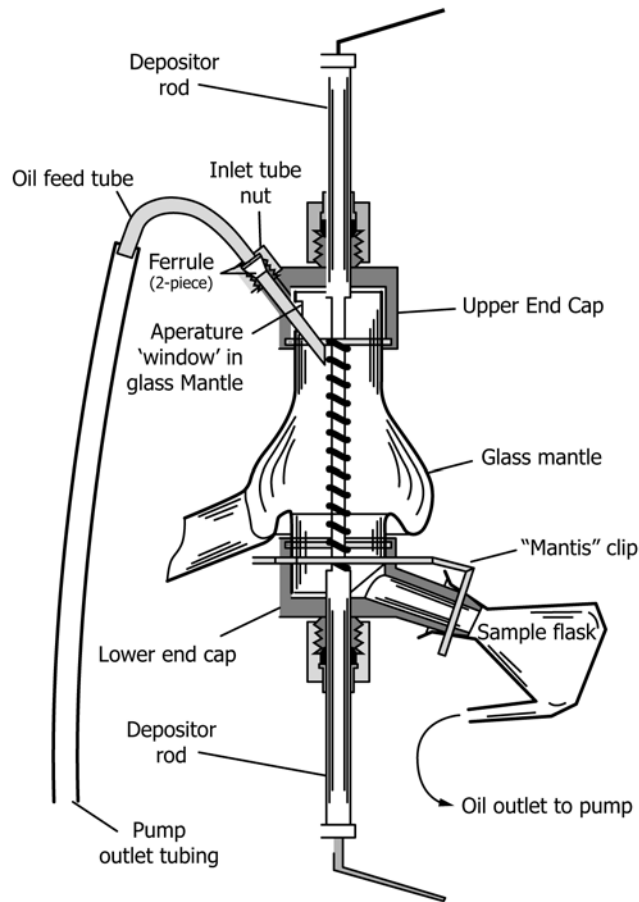


FIG. 6 Flexible Tube Connection to Oil Feed Tube and Sample Flask Placement with Mantis Clip (Cut-away View)

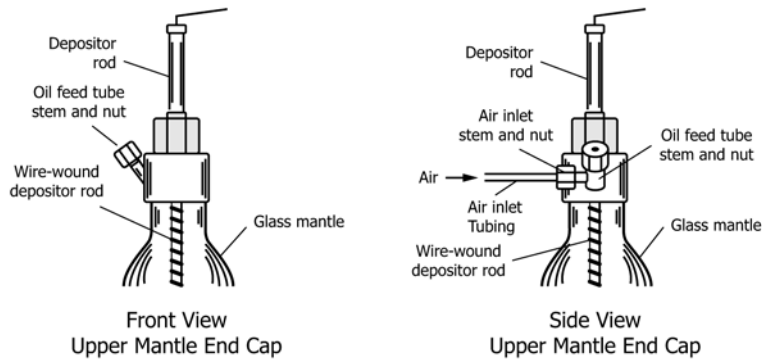


FIG. 7 Front and Side Views of Upper Mantle End Cap Showing Air Inlet

9.5 *Determination, Setting, and Use of Appropriate Position for the Temperature Control Thermocouple*—Follow the technique in the manufacturer’s Instrument Manual to find the hottest point within the bore of the depositor rod.

9.5.1 Insert the thermocouple gently (see **Note 5**) into the bore of the depositor rod to bring the collar into contact with the emergent top of the rod when it is in position within the depositor rod casing assembly positioned in the busbars.

NOTE 4—The locking collar may slip if the thermocouple is forced into the depositor rod thus resulting in a wrong position for the temperature sensing area of the thermocouple.

9.5.2 If desired, adjust the strip chart recorder or other temperature-recording device to record temperature sensed by the temperature control thermocouple.

NOTE 5—Continuously record the temperature of the controlling thermocouple at maximum sensitivity setting to determine any aberration in temperature during a run that may be caused by temporary electrical failure or brownout of the local power supply.

9.6 Install the over-temperature thermocouple according to the manufacturer’s Instrument Manual.

9.7 Standardize the TEOST MHT procedure using certified reference oils in accordance with Sections 10 and 15.

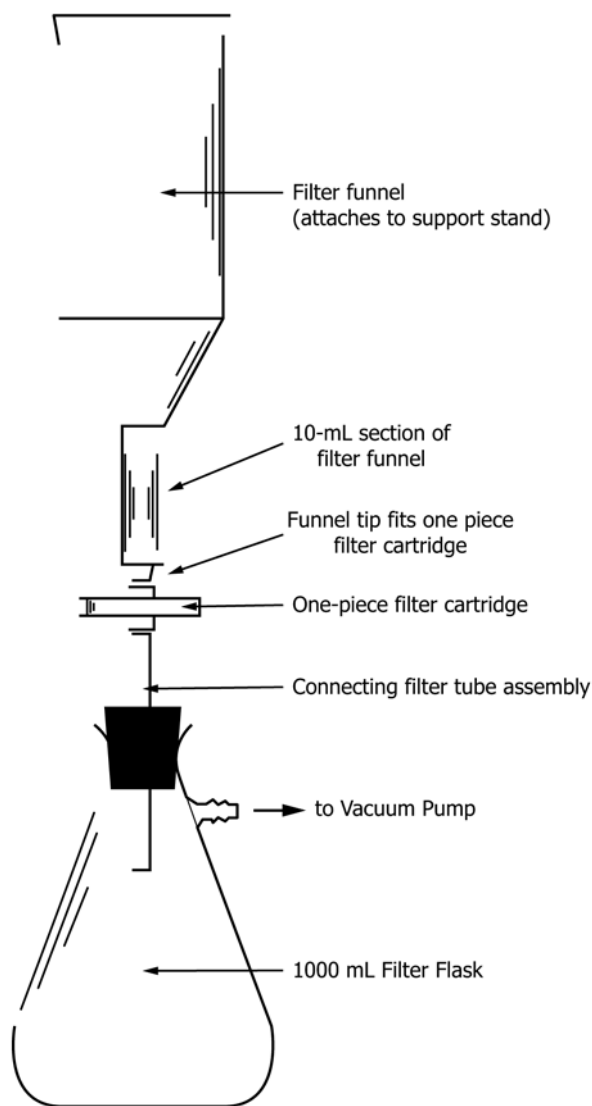


FIG. 8 Filter Funnel Setup

10. Instrument and Sample Preparation

10.1 Before testing unknown samples, confirm the functionality of the TEOST MHT instrument by testing one of the certified reference oils (see 7.6). Choosing one of the certified reference oils mentioned in 7.6, follow the directions in Section 10.

10.2 Make sure that the TEOST heat switch is in the off position to prevent any accidental startup of the test. Then turn on the main power switch and allow 30 min or more for the instrument electronics to warm up. Ensure that the pump switch is off.

10.3 Make sure that the thermocouples are clean (see 9.4.2).

10.4 Invert both the catalyst vial and the oil container at least three times to ensure homogeneity of both components prior to use.

10.5 Place a clean sample flask (see Fig. 2) on a precision balance and tare the balance (to bring the indicated mass of the container to zero).

10.6 Using a microliter glass syringe that uses either a glass or PTFE plunger (do not use rubber plunger), or optional disposable glass pipet, add the pre-calculated mass of catalyst required to make 8.5 g of sample-catalyst mixture based on the certified value of the catalyst and record the mass to the nearest 0.0001 g. The range for the mass of catalyst to be added shall be ± 0.0003 g of the mass required.

NOTE 6—The mass of oil required for the appropriate mixture of catalyst-to-oil ratio is stated on the label of the vial of certified catalyst.

10.7 Again, tare the balance and add the required mass of oil to the sample flask to make the sample-catalyst mixture total $8.5 \text{ g} \pm 0.05 \text{ g}$. The range for the mass of the oil to be added shall be $\pm 0.01 \text{ g}$ of the mass required to obtain the catalyst/oil ratio shown on the catalyst bottle. If more oil than required is added, make a new sample.

NOTE 7—An electronic file containing a generic calculation table to determine the appropriate catalyst/sample weights can be obtained from the ASTM Test Monitoring Center.⁷

10.8 Add a TFE-fluorocarbon-coated magnetic stirring bar to the sample flask, and place the flask on the magnetic stirrer incorporated on the platform of the TEOST (or other appropriate magnetic stirrer) and stir for 30 min to 60 min. Do not heat the mixture. Do not stir the mixture too vigorously, and particularly avoid creating a large vortex where the mixture may splash out. Be sure to load the sample and start the test within 20 min of the completion of the mixing process.

NOTE 8—For planning purposes, the hardware setup and the finish of sample mixing can be arranged to coincide, so the test can be started in a shorter preparation time.

11. Hardware Setup

11.1 Install new end-cap O-rings in both end-caps.

NOTE 9—At this point, any interest of the operator in determining the mass of volatiles, recovered end-of-test oil, and mantle deposits will require weighing and recording the initial mass of the volatiles collection vial, the recovered oil vial, and the cleaned and dried mantle.

11.2 Using a gentle twisting motion, insert the upper end of the glass mantle squarely into the upper end-cap. Avoid chipping the mantle glass, and make sure that the hole or window (see Fig. 5) in the upper mantle faces the corresponding oil feed tube opening in the upper end-cap. Set this assembly aside in a safe place until required to complete the depositor rod casing assembly.

11.3 *Cleaning and Weighing a Depositor Rod; Use of Weighing Boat:*

11.3.1 Select a weighing boat (see Fig. 3) for the depositor rod and obtain the boat's mass to $\pm 0.0001 \text{ g}$. Keep covered when not in use to avoid contamination from particles falling from the air.

NOTE 10—If fluctuations are seen on the balance, momentarily touch the boat to a grounding pad to eliminate static.

11.3.2 Once the depositor rod preparation has begun, handle the rod with care and do not set the rod down except on the weighing boat. Keep covered when not in use.

⁷ ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206. www.astmtmc.cmu.edu.

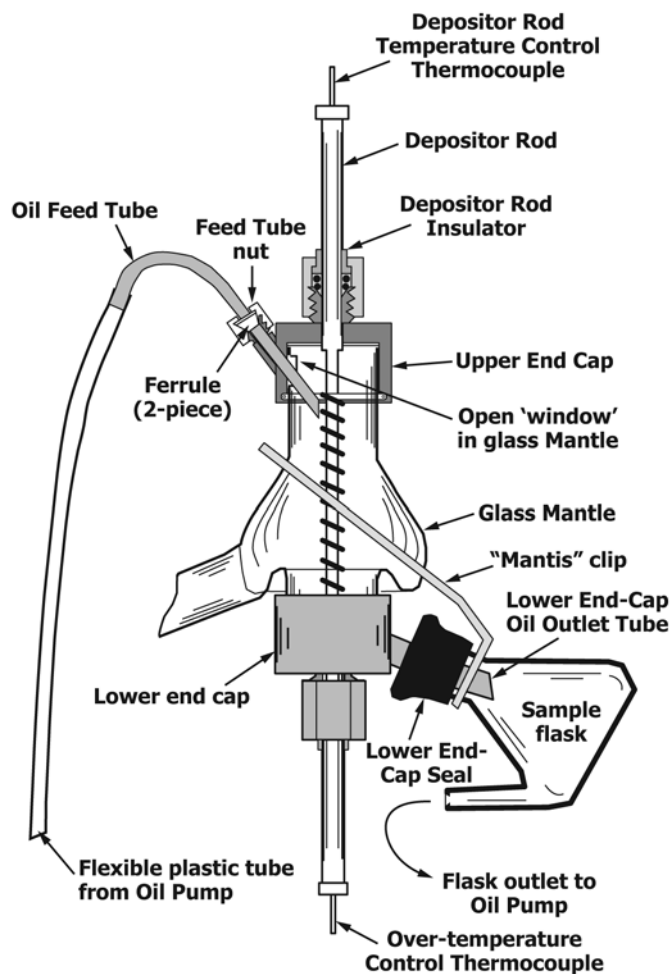


FIG. 9 Lower End-cap Seal

11.3.3 When handling and cleaning the wire-wound depositor rod, be careful not to distort the length and pitch of the wire coils.

NOTE 11—The wire coils on the depositor rod are preset to a specified pitch by the manufacturer and are adjusted to a tension to just be able to slide up and down on the machined narrower section of the rod around which the wire coil is wrapped without being too loose.

11.3.4 Use hydrocarbon solvent, to rinse both the outside and inside of the rod. Use thin, solvent-resistant laboratory gloves or finger cots when handling the rod to keep natural skin oils from affecting the mass of the rod.

NOTE 12—If the cots or gloves are chemically attacked by the hydrocarbon solvent, a film or residue will be left on the surface of the gloves.

11.3.5 Using a pipe cleaner soaked in acetone, swab the inside bore of the depositor rod by pushing the pipe-cleaner all the way through the bore in one direction, then repeat the cleaning of the inside bore in the opposite direction with a fresh pipe cleaner soaked in acetone. Exercise particular care in handling and cleaning the depositor rod during this stage, avoiding any distortion of the wire coil.

11.3.6 Rinse the outside and the inside bore of the depositor rod with acetone using appropriate solvent-resistant gloves.

11.3.7 Vacuum-dry or blow clean, dry air on the inside of the depositor rod while holding it between the thumb and index fingers. Dry the outside of the rod only by exposure to ambient air.

11.3.8 Set the depositor rod down in the pre-weighed weighing boat from 11.3.1 (see Fig. 3). Weigh the depositor rod and boat to a constant mass within ± 0.0001 g. Record this value as the initial depositor rod and boat mass. Subtract the boat mass from the total mass for later determination of the depositor rod mass carrying deposits from the test.

11.3.9 After the boat and depositor rod masses have been obtained, put the covered boat aside for later use when the depositor rod has completed its exposure to deposits for 24-h.

11.4 *Completing the Depositor Rod Casing Assembly* (see Figs. 4-6):

11.4.1 Pick up the pre-assembled mantle and upper end-cap (see 11.2) and carefully slide the depositor rod up through the bottom of the glass mantle/upper-end-cap assembly until the end of the depositor rod emerges through the threaded stem of the upper end-cap.

11.4.2 Place two new small O-rings on the emergent depositor rod.

11.4.3 Place one of the ceramic isolators over the top of the depositor rod with the wider diameter of the isolator facing the O-rings. Pushing with the ceramic isolator, bring the O-rings in contact with the top of the threaded stem of the end-cap as shown in Fig. 4.

11.4.4 Place an end-cap nut on the emergent depositor rod and insert the narrower diameter of the isolator through the clearance hole in the nut. Finger-tighten the nut onto the threaded end-cap stem to compress the two-stacked O-rings around the depositor rod thus sealing and insulating the depositor rod from the end-cap.

11.4.5 Make sure that the bottom of the depositor rod helical wire winding is resting on the bottom shoulder of the narrower midsection of the rod.

11.4.6 Insert the bottom end of the depositor rod through the top of the lower end-cap and continue by inserting the lower end of the mantle into upper end of the lower end-cap (see Figs. 4 and 5). Again, use a careful twisting motion so as not to stress or fracture the glass mantle.

11.4.7 Repeat the same process for the bottom of the depositor rod and lower end-cap to complete the depositor rod casing assembly as shown in Fig. 5 with the exception of the introduction of the oil feed tube.

11.4.8 Make sure that the top and bottom emergent ends of the depositor rod in the depositor rod casing assembly are about the same length. If necessary, loosen the end-cap nuts and move the depositor rod as necessary to ensure this equivalence.

11.5 *Connecting the Depositor Casing Assembly to the Bus Bars:*

11.5.1 Make sure that both the upper and lower faces of the busbar assemblies are free of oil or other material that might interfere with electrical continuity when the depositor rod is in place and the busbar caps are screwed in place. If necessary, use a paper towel and moisten and wipe the busbar contacting area with hydrocarbon solvent (see 7.4) or a varnish removing solvent, rinse with water, and allow to dry.

11.5.2 Insert the depositor rod assembly into the bus bar assembly and tighten as described in the operations manual.

11.5.3 Slide the over-temperature thermocouple up into the depositor rod until its collar touches either the bottom of the depositor rod or the lower busbar.

11.5.4 Before every test, check to ensure that the temperature control thermocouple's lock collar is tight and correctly positioned for the proper insertion depth. This depth was previously established in 9.5.

11.5.5 Place the temperature control thermocouple down the top center bore of the depositor rod to the lock collar. During insertion of the thermocouple in either the depositor rod or thermocouple depth gauge, be careful not to bend the thermocouple.

11.6 *Connecting the Rigid Oil Feed Tube and the Flexible Oil Flow Tubing:*

11.6.1 *Connecting a New Oil Feed Tube (see Fig. 5) for First-time Use:*

11.6.1.1 Ensure that the depositor rod casing assembly has been completed before proceeding. Then do a first setting of the ferrule according to 11.6.1.2 to 11.6.1.7.

(1) It is a preferred technique to install and hold the depositor rod casing assembly fixed in the busbars when setting the ferrule.

11.6.1.2 Place the two-piece ferrule in position to clamp the outer diameter of the oil feed tube by inserting its beveled tip first through the oil feed tube inlet nut and then the two-piece ferrule with the ferrule's conical end facing the beveled end of the oil feed tube.

11.6.1.3 Lightly push the oil feed tube carrying the two-piece ferrule into the interior matching tapered socket of the oil feed tube inlet stem of the upper end-cap (see Fig. 7). Bring the beveled tip of the oil feed tube into light contact with the helically wound wire on the depositor rod (see Fig. 5) as viewed up through the mantle.

11.6.1.4 Using the oil feed tube inlet nut, push the two-part ferrule into the feed tube inlet stem and screw the nut onto the stem until resistance of the ferrule is felt.

11.6.1.5 Ensure that the beveled end of the oil feed tube is still in light contact with the depositor rod wire and that the opposite end of the oil feed tube is pointed downward when the depositor rod casing assembly is held upright (see Fig. 5), then tighten the nut about ½-turn to set the conical ferrule onto the rigid tubing.

11.6.1.6 Test the effectiveness of the ferrule's contact with the oil feed tube by lightly trying to move the oil feed tube back from contact with the wire winding on the depositor rod while the oil feed tube inlet nut is still tightened.

11.6.1.7 If the oil feed tube can be moved back from contact with the depositor rod wire winding when the oil feed tube inlet nut is tight, once more bring the beveled tip in light contact with the rod wire and further tighten the inlet nut just enough to stop this back-and-forth motion of the oil feed tube.

NOTE 13—This series of steps positions the ferrule on the plastic tube so that the beveled tip will always be in proper position in relation to the depositor rod when the non-beveled end is pointed downward. However, the ferrule has not been set firmly enough to prevent side-to-side motion of the non-beveled end. To set the ferrule so firmly as to prevent this side-to-side movement is not necessary in most installations but if it is desired to do so, contact the manufacturer for instruction.

11.6.2 *Connecting a Previously Used Oil Feed Tube (see Fig. 5) to its Assigned Upper End Cap:*

11.6.2.1 With the depositor rod casing assembled and connected to the bus bars, insert the beveled end of the oil feed tube into and through the oil feed tube inlet stem of the upper end-cap until the tip reaches and lightly touches the wire winding on the depositor rod. Tighten the oil feed tube inlet nut.

11.6.2.2 *Position of Oil Feed Tube Tip:*

(1) The oil feed tube tip shall either touch the wire or be within close proximity on the depositor rod to ensure that the test oil runs down the specially treated helical wire guide on the rod and does not bypass the rod by dripping straight to the bottom end-cap or run down the inside mantle wall.

(2) Through regular use, the beveled end of the oil feed tube will eventually degrade, becoming charred and less pliable. To ensure proper delivery of oil to the depositor rod, replace the oil feed tube after 25 tests or at any time the beveled tip becomes charred or the test oil bypasses the rod.

NOTE 14—Occasionally, when the tip of the oil feed tube is sufficiently degraded, the test oil will move back up the outside of the tube and run down the inside of the mantle into the mantle trough and then into the volatiles vial, thus spoiling the test.

11.7 Connect the oil feed tube to the pump outlet with 3.2 mm (0.125 in.) outer diameter clear, oil-resistant, flexible plastic tubing.

11.8 *Verification of Airflow:*

11.8.1 Adjust the air supply to the proper value of 10.0 mL/min and then check the airflow to the depositor rod casing assembly as directed in 9.1.

11.8.2 Connect the air inlet tubing to the air inlet stem on the upper end-cap and finger-tighten the nut (see Fig. 7). Place a rubber plug into the lower end-cap oil outlet to prevent any leakage of air from that opening.

11.8.3 To ensure that the air flow rate passing through the mantle is 10 mL/min \pm 0.2 mL/min, connect the bubble gauge or calibrated handheld digital flow-meter to the volatiles delivery tube so that all air exiting from that opening passes through the bubble gauge or handheld digital flow-meter.

11.8.4 Measure the air flow rate from the outlet of the volatiles delivery tube. Be sure that the pump switch is in the *off* position before such measure of air flow.

11.8.4.1 If the flow rate is less than 10 mL/min \pm 0.2 mL/min, tighten the oil feed tube nut on the upper end-cap more firmly as well as the upper and lower end-cap nuts that press against the ceramic isolator and the two small O-rings holding the depositor rod (see Fig. 5 and Fig. 7).

11.8.4.2 If the flow rate remains less than 10 mL/min \pm 0.2 mL/min, check for air leakage around the upper and lower end-cap O-rings (see Fig. 5) with an eyedropper filled with detergent/water solution and check for bubbles. If bubbles are seen, first remove the mantle from the end-cap and check the mantle for cracks or chipping. If none are found, replace the end-cap O-ring and test again. If bubbles are still found, contact the manufacturer.

11.8.4.3 If the flow rate is still less than 10 mL/min \pm 0.2 mL/min, disconnect the air line from the upper end-cap and then check the flow rate at this point (see 9.1).

11.8.4.4 If the flow rate is correct at this point, replace the steel ferrule on the air inlet tubing with a plastic ferrule and firmly tighten the air supply connection to the air inlet stem top mantle end-cap (see Fig. 7).

11.8.4.5 Recheck the airflow rate from the volatiles delivery tube.

11.8.4.6 If the flow rate is more than 10 mL/min \pm 0.2 mL/min, recheck the flow rate from the air source using the bubble flow meter or handheld digital flow-meter.

11.8.4.7 If the flow rate cannot be set to 10 mL/min \pm 0.2 mL/min, contact the manufacturer.

11.8.4.8 If the airflow is acceptable, remove the connection from the volatiles delivery tube and the plug from the oil outlet of the lower end-cap.

11.8.4.9 The acceptable range of air flow fluctuation during the test is 10.0 mL/min \pm 1.0 mL/min.

12. Sample Installation

12.1 After the sample and catalyst have been well mixed (see 10.8) in the sample flask, inspect the mixture to determine

if it appears homogeneous. If not, continue stirring and inspect the mixture occasionally until it has become homogeneous.

NOTE 15—For higher viscosity or dark samples, such as SAE 20W-40 engine oils, achieving homogeneity is somewhat more difficult and may require viewing the mixture with a light to ensure thorough mixing.

12.2 Connect the pump inlet tubing to the sample flask's glass sidearm.

12.3 Remove the pump outlet tubing to the sample flask's glass sidearm.

13. Procedure—Startup

13.1 Using the manufacturer's Instruction Manual, check the program's PID settings (see 8.1) and the temperature profile to ensure that no settings have been changed.

13.2 While the depositor rod is still at room temperature, start the pump at maximum flow rate on the pump control and run until the test oil flows out of the pump outlet tubing and into the sample flask with no bubbles.

13.3 At this point, temporarily turn the pump setting to zero flow rate and return the pump outlet tubing into the upper end cap.

13.4 Snap the special sample flask mantis clip shown in Fig. 2 around the neck of the sample flask as shown in Fig. 6.

13.4.1 Hold the sample flask securely upright on a flat table-top or bench-top with one hand and press the lower end-cap seal in place over the mouth and neck of the sample flask. Use just sufficient force to place the lower end-cap seal in place then insert the lower end-cap oil outlet tube through the lower end-cap seal opening into the sample flask as shown in Fig. 9.

13.4.2 If not already aligned, rotate the sample flask so that the flask sidearm is directly below the flask (see Fig. 6.)

13.5 Place the volatiles collector vial in position on the volatiles delivery tube from the mantle and carefully snap on the volatiles vial clip (see Fig. 4).

13.6 Make sure that the volatiles collector vial does not block air flow from the mantle's volatiles delivery tube and that the volatiles delivery tube outlet is well within the vial (see Fig. 4).

13.7 If desired, turn on the recording device for continuously recording depositor rod temperature indicated by the controlling thermocouple.

13.8 Install the operator poly(methylmethacrylate) shield for safety and test consistency.

NOTE 16—The shield not only protects the operator but also allows a constant temperature within the rod area by keeping passing room or hood airflow to a minimum.

13.9 Turn on the heater switch.

13.10 Start the program on the temperature controller but leave the pump speed at zero.

13.11 When the depositor rod temperature controller shows rod temperature between 200 °C and 250 °C, increase the pump speed from zero to the value determined during calibration for sample delivery of 0.25 g/min \pm 0.02 g/min (see 9.2).

Make sure that oil circulation is functioning with no leaks and the oil is flowing down the depositor rod.

13.12 After any initial temperature overshoot and stabilization of the temperature, temperature control shall remain at $285\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. If, after an hour, the temperature is still not stable, abort the test and contact the manufacturer.

13.13 Run the test for the required $24\text{ h} \pm 10\text{ min}$ test time.

NOTE 17—If desired, a timer can be placed between the instruments power cord and the wall socket to shut off the instrument when the test is completed.

13.14 The instrument will indicate that the test is finished by either showing the word *off* on the temperature controller or, in older models, by the temperature dropping to room temperature (see respective Instrument Manuals).

14. Preparation and Disassembly for Weighing

14.1 Preparation:

14.1.1 Prepare the three test tubes for extracting any remaining oil from the depositor rod by filling the test tubes with hydrocarbon solvent, enough to cover at least $\frac{3}{4}$ of the rod. If desired, one test tube may be used and simply rinsed into the material to be filtered.

14.1.2 Bring out the depositor rod weighing boat from 11.3.9 but keep it under a cover to prevent contamination from airborne particles.

14.2 Disassembly:

14.2.1 Remove the volatiles collector vial from the depositor rod assembly and cap the vial.

14.2.2 Disconnect the air supply line from the upper end-cap.

14.2.3 Carefully remove the temperature control thermocouple from the depositor rod to avoid bending it.

14.2.4 Remove the oil feed tube inlet nut and withdraw the oil feed tube from the upper end-cap.

14.2.5 Place the beveled tip of the oil feed tube into a 10 mL recovered end-of-test oil vial held securely in a clamp or vial stand.

14.2.6 Use the pump to drain the remaining end-of-test oil in the flask, pump, and lines into the clean residual oil vial by turning the pump on at maximum speed until no more end-of-test oil is recoverable (approximately 5 min). If there is a desire to measure the mass of this material, allow at least 10 min to 15 min for the remnant oil to flow off the rod into the flask.

NOTE 18—Save these oils for further information and analysis if desired.

14.2.7 When as much of the end-of-test oil is removed as possible, remove the tip of the pump outlet tubing from the end-of-test vial, cap the vial, and turn off the pump.

NOTE 19—If the operator has elected to determine the mantle mass, mass of volatiles, or recovered end-of-test oil mass collected during the test, record the final mass of the mantle to the nearest 0.0001 g and the mass of the vials to the nearest 0.01 g or less. Subtract the initial masses of the mantle and vials from the final masses and report the resulting values.

14.2.8 After pumping out all recoverable residual oil, place the tip of the pump outlet tubing onto a vacuum source that is

connected to a vacuum flask. Remove the sample flask, mantis clip, and lower end-cap seal from the lower end-cap, set aside mantis clip and the lower end-cap seal for future use. Protect the sample flask from being upset and fill the sample flask half full with hydrocarbon solvent.

14.2.8.1 Turn on the vacuum to the vacuum flask and set the pump speed to a maximum of 500 to flush the hydrocarbon solvent through both the pump inlet and pump outlet tubing.

14.2.9 Repeat the flush of the pump tubing by again filling the sample flask with a hydrocarbon solvent and continue to vacuum. If the hydrocarbon solvent is colored at the end of this second flush, repeat the flushing until the hydrocarbon solvent is clear.

14.2.10 When clear hydrocarbon solvent emerges from the pump, continue to vacuum for 10 min.

14.2.10.1 This flush is done to avoid a sludge buildup in the pump. If the oil residue is allowed to sit in the pump longer than several hours, perform an initial flush with about 10 mL of fresh SAE 50 engine oil in the same manner prior to the hydrocarbon solvent flush and then follow by the hydrocarbon solvent flush as needed.

14.2.10.2 For more effective cleaning, periodically remove the metal pump inlet/outlet tubing connectors from the pump and place in a sonic bath containing varnish remover for 20 min.

14.2.11 Remove the sample flask, mantis clip, and tubing to the pump. Thoroughly rinse the sample flask and magnetic stir bar with the hydrocarbon solvent. Remove the bar, and dry both the flask and bar for reuse.

14.2.12 If needed, place the sample flask in a plastic beaker for protection against breakage. Cover the flask with varnish remover and let soak until the flask washes clean with warm water.

14.3 Disassembly of Depositor Rod Casing Assembly:

14.3.1 Loosen the hex screws on the busbars and carefully remove the depositor rod casing assembly.

NOTE 20—In lifting the depositor rod casing assembly off the bottom over-temperature thermocouple, exercise care not to bend the thermocouple.

14.3.2 Place the entire depositor rod assembly in a clean and dry rinse container such as a 600 mL beaker or other suitable container.

14.3.3 Hereinafter, use a plastic squeeze bottle containing the hydrocarbon solvent for carefully cleaning the assembly and its components so as to not lose any deposits except into the rinse container.

14.3.4 Refer to the manual for further cleaning details.

14.3.5 Carefully place the mantle and lower end-cap in the container for later washing and drying. Do not use a sonic bath to clean the glassware.

14.3.6 Hold the end of the depositor rod with the thumb and index finger and gently rinse the inside and outside of the rod with the hydrocarbon solvent, while allowing the residue to fall into the container.

14.4 Washing, Drying, and Weighing the Depositor Rod:

14.4.1 Place the depositor rod immediately into the first of the three test tubes previously filled with hydrocarbon solvent

(see 14.1.1) for 10 min progressively in each of the three test tubes to soak the rod deposits and extract any residual oil. If the deposit is heavy, longer soak times may be required. Again, one test tube may be used as long as it is rinsed into the deposit rinse container.

14.4.2 Thoroughly rinse the depositor rod with hydrocarbon solvent over the chosen container. Use a vacuum hose to dry the inside bore of the rod thoroughly but do not bring the vacuum hose near the deposit area of the depositor rod. *Do not use any air source to dry the rod because this will blow some of the deposits off the rod and spoil the test.*

14.4.3 Immediately set the depositor rod onto the pre-weighed weighing boat (see Fig. 3 and 14.1.2) and allow about 10 min for the hydrocarbon solvent residue to evaporate.

14.4.4 After placing the depositor rod in the weighing boat, do not remove the rod from the weighing boat until the weighing process is completed.

NOTE 21—Deposits may fall off the rod during the weighing period but will be caught by the boat.

14.4.5 Weigh the rod and boat to the nearest 0.0001 g. Reweigh after 10 min to determine if all of the hydrocarbon solvent has evaporated. If not, continue to redetermine the mass of the rod and boat every 10 min until it reaches constant mass of ± 0.0001 g. Subtract the empty mass of the boat (determined in 11.3.1) from this value and record the difference as the final mass of the rod.

NOTE 22—If fluctuations are seen on the balance, touch some part of the boat containing the depositor rod momentarily to a grounding mat, grounding wire, or equivalent technique to discharge the static.

14.5 *Collecting the Mass of Depositor Rod Deposits into the Container of Hydrocarbon Solvent:*

14.5.1 Pick up the lower end-cap from the container and rinse it with hydrocarbon solvent into the container and set the lower end-cap aside.

14.5.2 Pick up and rinse the glass mantle with hydrocarbon solvent into the container and set it aside.

14.5.3 Add and rinse the contents of the three test tubes from 14.4.1 into the container. Rinse thoroughly to ensure that no depositor rod deposits are lost.

14.6 *Filtering the Contents of the Wash Hydrocarbon Container:*

14.6.1 Select a filter cartridge and inspect the filter for debris or oil. If acceptable, determine and record the tare mass of the filter on a balance to the nearest 0.0001 g. This number will be the initial filter mass.

NOTE 23—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 adsorb some mass from the hydrocarbon solvent if the two are left in contact for an extended time of 20 min or more.

14.6.2 Connect the filter cartridge to the filter system shown in Fig. 8, turn on the vacuum source and slowly pour the contents of the container into the glass filter funnel and draw these contents into and through the filter cartridge where the solid contents are trapped and the liquid contents 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.

14.6.2.1 If larger deposits from the depositor rod are lodged in the neck of the funnel above the filter, use a wire rod (see 6.1.3.6) extended down from the top of the funnel to break up and dislodge any deposits that may collect. Wash off any deposits adhering to the wire into the filter. To avoid puncturing the layered filters, do not push the wire rod into the filter cartridge.

NOTE 24—Deposits often adhere to the funnel outlet because of the influence of static electrification and the wire rod also helps to dissipate this charge and permit the particles to travel on to the filter when washed.

14.6.3 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 30 mL aliquots, to allow the hydrocarbon solvent to percolate slowly through the filter and solubilize any oil remaining in the deposit.

NOTE 25—On rare occasions the filter may become clogged because of both numerous and very fine deposits. Under these conditions, a second or possibly even third pre-weighed filter cartridge shall be used.

14.6.4 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 of two weighings taken 5 min apart to within ± 0.0001 g.

NOTE 26—If the drying time becomes lengthy, draw the 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.

14.7 *Cleaning the Reusable Components:*

NOTE 27—Since this is a 24-h test, depending on the operator's interest in quickly starting another test, it is helpful to have two complete sets of casing components (upper and lower end-caps and mantle) so that following tests can be initiated quickly.

14.7.1 Place end-caps in a glass or plastic container and place the mantle in a separate plastic container for protection against breakage. Cover both contents with varnish cleaning material that does not etch glass or adversely affect metal, and let soak until the various components wash clean with warm water.

14.7.2 Soak all depositor rod-casing components in acetone to take up water and other hydrocarbon insoluble materials. Remove the O-rings and discard.

NOTE 28—After soaking in acetone for about 1 h, the end-cap O-rings will swell and usually dislodge themselves from the end-caps.

14.7.3 Rinse the depositor rod casing components with hydrocarbon solvent and store for next use.

14.7.3.1 Inspect the mantle for any chips or cracks. If the mass of the cleaned mantle is less than its mass obtained at the initiation of the test, this is an indication that some chipping has occurred. Replace the mantle if chipping or breakage does not permit air flow control (see 11.8.4).

15. Test of Instrument Functionality

15.1 After setting up the TEOST MHT instrument for analysis according to Sections 10 through 12, first test the

instrument and supporting equipments to determine if it will produce acceptable results.

15.1.1 Chose one or more of the three certified reference fluids available (see 7.4) and run such oil or oils through the test sequence of Sections 13 and 14.

15.1.2 Determine the mass of combined depositor rod and filter deposits (see Section 16) and compare this mass to the acceptable range for the chosen certified reference fluid(s).

15.1.3 If the value(s) obtained is within the acceptable calibration range, proceed to test oils.

15.1.4 If the value obtained is not within the acceptable calibration range, recheck and verify control thermocouple depth, air flow rate, and oil circulation rate. If these are acceptable, contact the manufacturer. If not, reset these conditions and rerun the reference fluid(s).

16. Calculations

16.1 Calculate the following to the nearest 0.0001 g by subtracting the pretest mass of each item from the post-test mass:

16.1.1 Depositor rod deposits equals final rod mass (see 14.4.5) minus initial rod mass (see 11.3.8).

16.1.2 Filter deposits equals final filter mass (see 14.6.4) minus clean filter mass (see 14.6.1).

NOTE 29—If the mass of mantle deposits, volatiles and recovered oil are desired, calculate by subtracting the initial mass from the final mass of the mantle and vials, respectively. Report mantle deposits in milligrams. However, volatile material and recovered end-of-test oil are normally reported in grams.

16.2 Convert results in 16.1.1 and 16.1.2 to milligrams ($g \times 1000 = mg$).

17. Report

17.1 For reference oil tests, use the standardized report form set and data dictionary for reporting test results and for summarizing the operation data.

17.2 Report the following to the nearest 0.1 mg:

17.2.1 Filter deposits,

17.2.2 Rod deposits, and

17.2.3 Total deposits (rod deposits plus filter deposits).

NOTE 30—If desired, report the mass of mantle deposits, volatiles, and recovered oil.

TABLE 1 Example for Calculating Repeatability (r) and Reproducibility (R)

Total Deposits, in mg	Formula Utilized (r and R)	Example Result
30	$r = 1.98(x^{0.556})$	$r = 13.1$
30	$R = 2.19(x^{0.556})$	$R = 14.5$

18. Precision and Bias

18.1 *Precision*:

18.1.1 *Repeatability (r)*—The difference between successive 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 only in one case in 20 (see Table 1):

$$r = 1.98(x^{0.556}) \quad (1)$$

in which (x) is the mean of two test results.

18.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would in the long run exceed the following values only in one case in 20 (see Table 1):

$$R = 2.19(x^{0.556}) \quad (2)$$

in which (x) is the mean of two test results.

18.2 *Bias*—No information can be presented on the bias of the procedure in this test method for measuring deposit mass because no material having an accepted reference value is available.

18.3 *General Considerations*—The interlaboratory study on the above precision statement was performed according to this test method with eight oils run in duplicate at ten laboratories. The range of applicability of this test method, as derived from the interlaboratory study, is approximately 10 mg to 100 mg. See RR:D02-1841 for additional details and data on severity.⁸

19. Keywords

19.1 deposits; engine oil; oxidation; thermal decomposition; thermal oxidation

⁸ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: RR:D02-1841.

SUMMARY OF CHANGES

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D7097 – 16) that may impact the use of this standard. (Approved September 1, 2016.)

(1) Revised Section 18.

Subcommittee D02.09 has identified the location of selected changes to this standard since the last issue (D7097 – 09) that may impact the use of this standard. (Approved July 1, 2016.)

- | | |
|---|--|
| (1) Added disposable glass pipet as another option for use in subsection 6.2.2. | (5) Clarified statement in 14.6.1. |
| (2) Clarified language in 6.2.2 and 10.6 on the type of syringe and plunger to use. | (6) Updated SI units. |
| (3) Removed dimensions for wire rod in 6.1.3.6 and for oil extraction test tubes in 6.2.4. | (7) Added seal to the top of the sample flask assembly during operation. |
| (4) Changed language in 7.4 in regards to the type of hydro-carbon solvent allowed for use. | (8) Updated precision with new round robin data using sealed flask. |

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