



Designation: D7097 – 16

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

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 ( $\epsilon$ ) 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 MHT<sup>2</sup>) 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 manufacturer<sup>3</sup> and made specifically for the practice and precision of the test method.<sup>4</sup>

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 (dm<sup>3</sup>) and the milliliter (mL) for the SI cubic centimeter (cm<sup>3</sup>). 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*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.09.0G on Oxidation Testing of Engine Oils.

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<sup>2</sup> TEOST and MHT are registered trademarks of the Tannas Co. (Reg. 2001396), Tannas Company, 4800 James Savage Rd., Midland, MI 48642.

<sup>3</sup> 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,<sup>1</sup> which you may attend.

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

*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:*<sup>5</sup>

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 *TEOST<sup>2</sup>, 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:*

<sup>5</sup> 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.

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

3.2.1 *MHT*<sup>2</sup>, *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.<sup>6</sup>

#### 6. Apparatus

6.1 *TEOST MHT Instrument*,<sup>3</sup> 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

<sup>6</sup> 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.

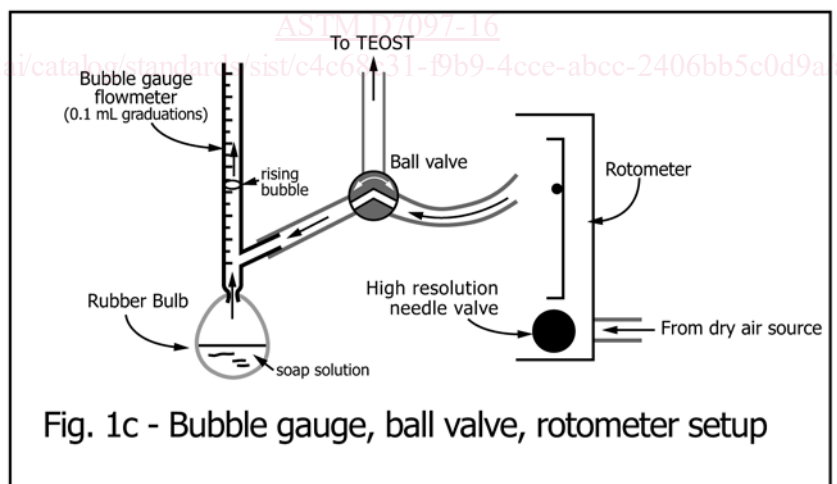
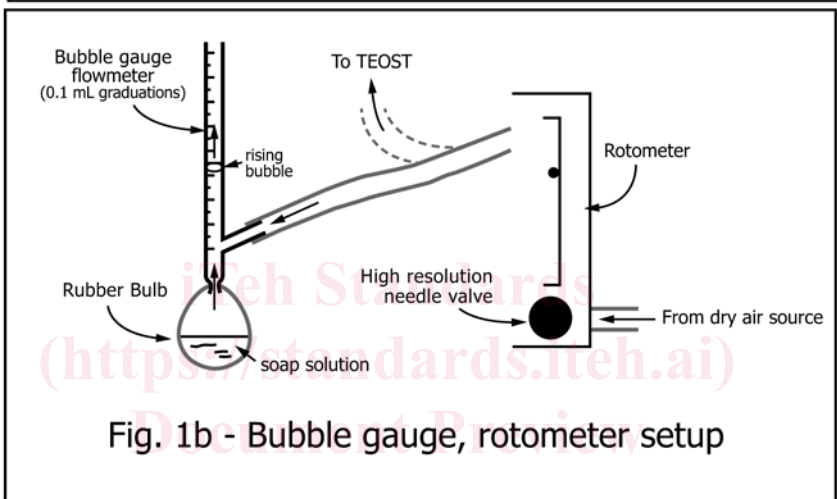
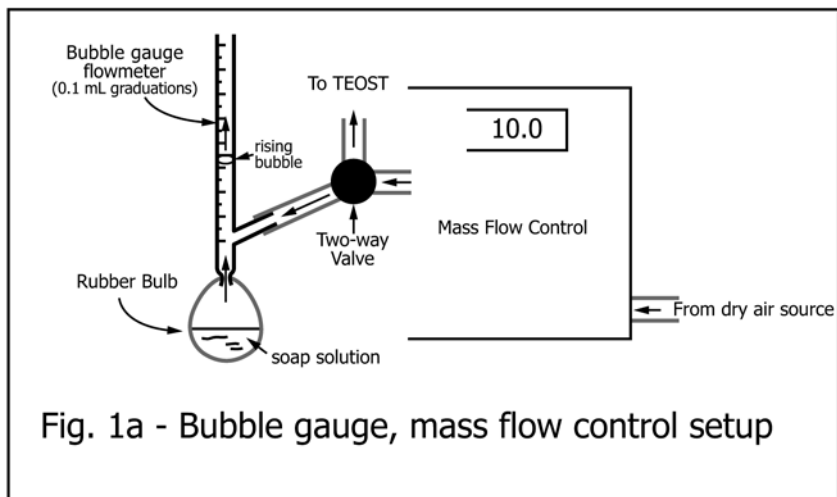


FIG. 1 Bubble Gauge

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

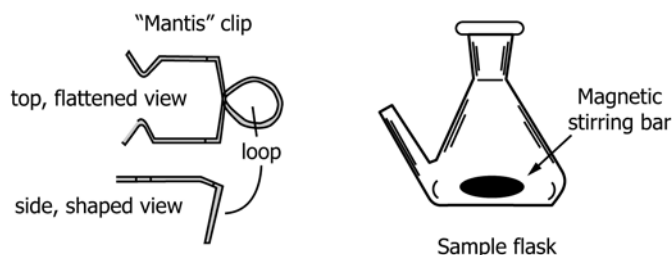


FIG. 2 Sample Flask with Stirring Bar and Mantis Clip

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  $\mu$ 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*<sup>3</sup>—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*,<sup>3</sup> 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*,<sup>3</sup> 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 Manual<sup>3</sup> 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.

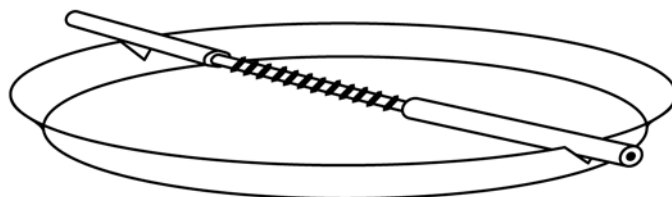
8.3 If using a strip chart recorder, turn on the strip chart, set the chart speed to 10 mm/h, but do not lower the pen(s) or turn on the chart drive at this time.

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

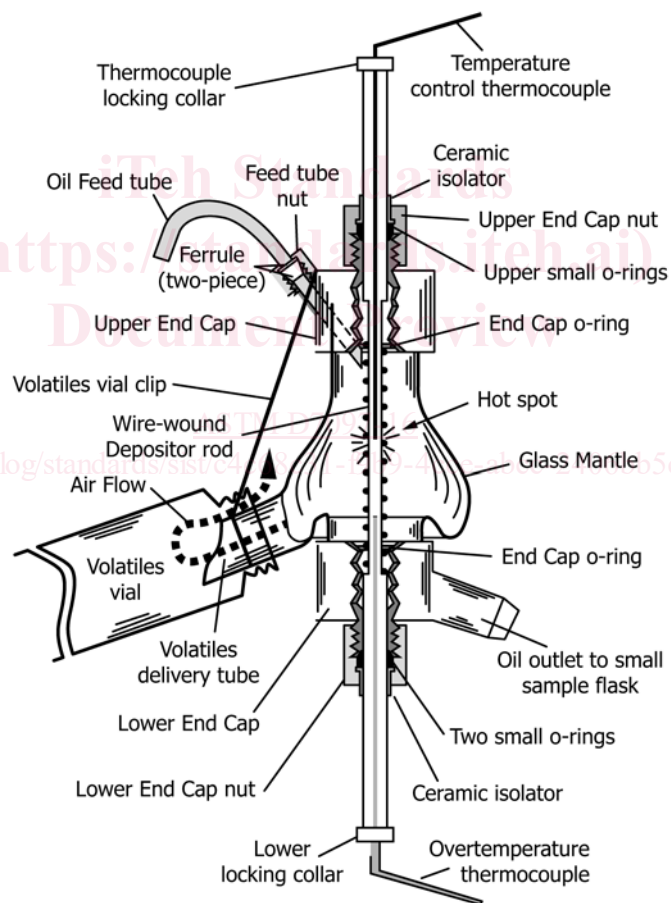


FIG. 4 Depositor Assembly (Cut-away View)

**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:

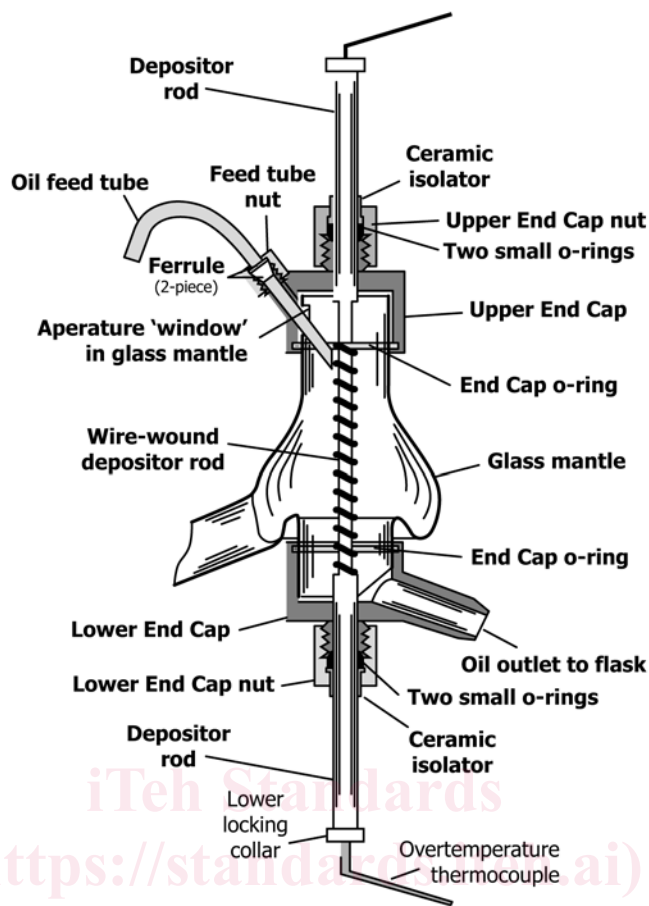


FIG. 5 Insertion of Oil Inlet Tube (Cut-away View)

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.