Designation: D 7097 – 05

An American National Standard

Standard Test Method for Determination of Moderately High Temperature Piston Deposits by Thermo-Oxidation Engine Oil Simulation Test—TEOST MHT[1]

This standard is issued under the fixed designation D 7097; 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 MHT[2]) test method as derived from an interlaboratory study is approximately 10 to 100 mg. However, experience indicates that deposit values from 1 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[3] and made specifically for the practice and precision of the test method.[4]

NOTE 1—ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

1.3 The values stated in SI units are to be regarded as standard. Although not an SI unit, the special name, litre (L) is allowed by SI for the cubic decimetre (dm^3) and the millilitre (mL) for the SI cubic centimetre (cm^3).

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]

D 4485 Specification for Performance of Engine Oils

D 6335 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 gage*, *n*—a precision bore glass tube marked in tenths of a millilitre 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 *MHT*[2], *n*—an acronym for moderately high temperature.

3.1.4.1 *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 D 6335, procedure for determining potential deposits in turbochargers).

3.1.5 *TEOST*[2], *n*—an acronym for Thermo-Oxidation Engine Oil Simulation Test.

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

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

[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 and MHT are registered trademarks of the Tannas Co. (Reg. 2001396).

[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,[3] 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,[4] which you may attend.

[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.

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 D 4485 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 pre-treated 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 2—Precision of the TEOST MHT procedure is highly dependent 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 re-circulating 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.

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

6.1.1.9 *O-rings, Larger, Petroleum-resistant*, create a seal between the end-caps and glass mantle. (See Fig. 5.)

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

6.1.1.11 *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.)

6.1.1.12 *Sample Flask,* a small (~25 mL), modified form of an Erlenmeyer flask with side-arm 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.)

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

6.1.1.14 *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.

6.1.1.15 *Thermocouple Lock 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.)

6.1.1.16 *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-tube Airflow Gage*, a device for precisely establishing the airflow rate and calibrating the flow meter from 1 to 30 mL/min. (See Fig. 1.)

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

6.1.2.3 *Hand-held 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 ± 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] 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.

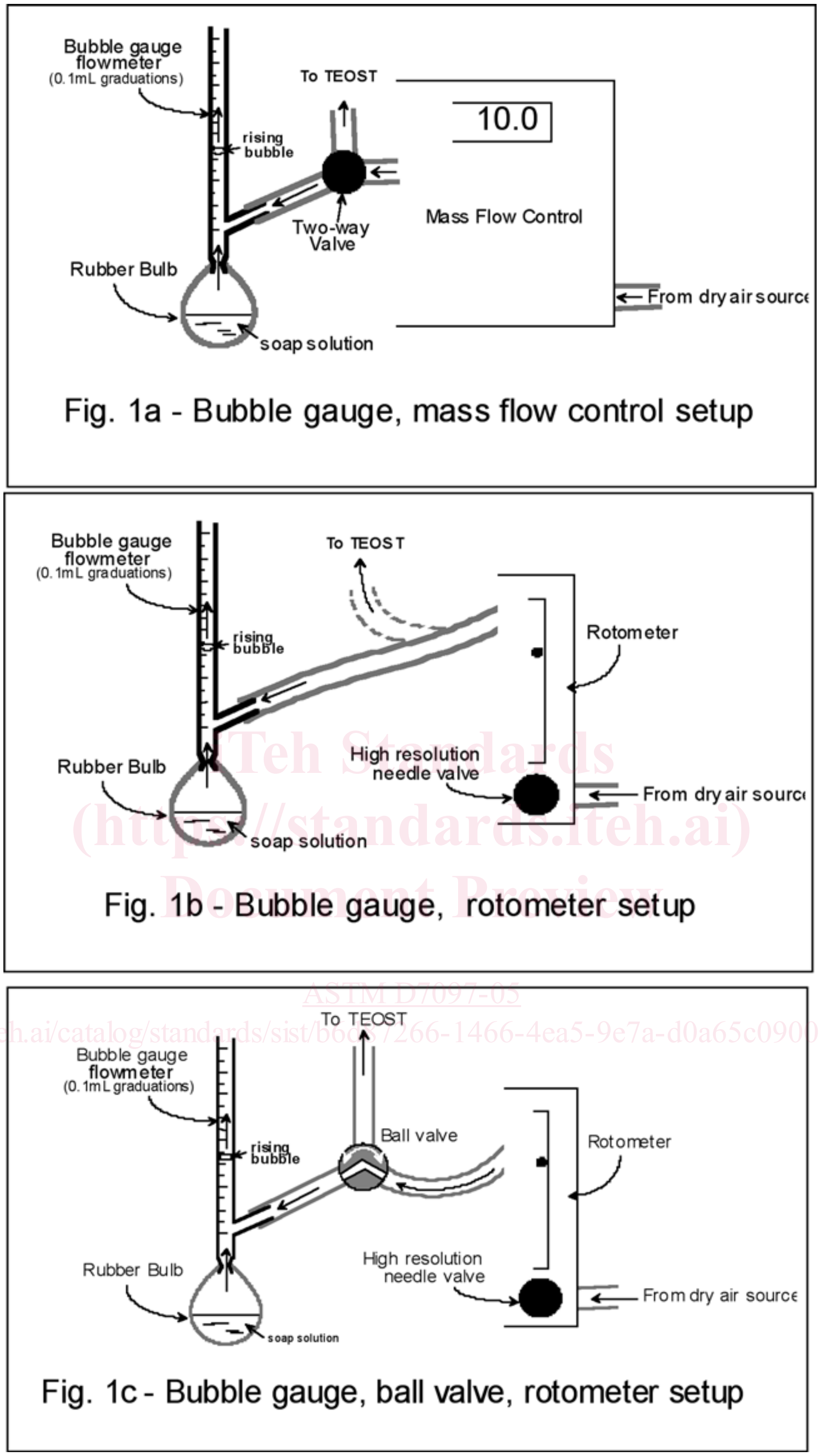

Fig. 1a - Bubble gauge, mass flow control setup

Fig. 1b - Bubble gauge, rotometer setup

Fig. 1c - Bubble gauge, ball valve, rotometer setup

FIG. 1 Bubble Gage

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.

6.1.3.2 *Filter Funnel,* a special combination funnel of ~400-mL capacity, necking down to a 10-mL graduated or 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 filter rinse hydrocarbon solvent.

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

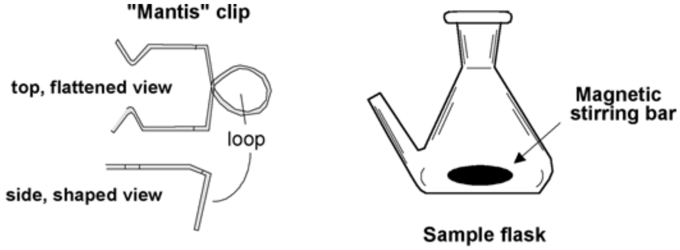

FIG. 2 Sample Flask with Stirring Bar and Mantis Clip

6.1.3.6 *Wire Rod*, stainless steel, 1 to 1.5 mm diameter, 300 to 350 mm length, 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 syringe of 100 µL capacity, for carefully metering the catalyst being weighed into the sample flask.

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

6.2.4 *Oil Extraction Test Tubes*, three, 120-mm tall, made of glass or hydrocarbon solvent-resistant plastic.

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 Gage,* an optional measurement device fabricated for simple setting and checking thermocouple insertion depth, using a millimetre 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 to 10 cm and notched in two diametrically opposed places to prevent the rod from rolling. (See Fig. 3.)

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 to 100 kPa (1.5 to 15 psi) at more than 690 kPa (100 psi).

7.4 *Cyclohexane or Other Alkane Hydrocarbon Solvent of Equivalent Volatility*, reagent grade. (**Warning**—Flammable.) Do not use naphthenic or aromatic hydrocarbons.

7.4.1 *Discussion*—The volatility of the alkane hydrocarbon solvent ensures timely evaporation of the deposits on the rod and filter.

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

7.5.1 *Discussion*—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 sealed storage, up to four weeks, may be at room temperature.

7.6 *Certified Reference Oils,*[3] certified low deposit fluid (LDF, about 10 to 15 mg), medium deposit fluid (MDF, about 40 to 50 mg), and high deposit fluid (HDF, about 70 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*—Adjust the thermocouple sensitivity and response values (PID settings) to have the minimum excursion from the temperature value desired during operation. See Instrument Manual[3] for recommended settings and 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.

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*:

9.1.1.1 Use the bubble gage 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 and 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.

9.1.1.2 Set up the bubble gage and mass flow controller sketched in Fig. 1a.

9.1.1.3 Connect the clean, dry air source to the mass flow controller and set the source's regulator to a value no greater than allowed by the tolerances of the flow controller (typically between 100 to 140 kPa (15 to 20 psi).

9.1.1.4 Connect the mass flow controller to the bubble airflow gage (bubble gage).

9.1.1.5 Start the flow of air through the flow controller and bubble gage.

9.1.1.6 Squeeze the rubber bulb of the bubble gage, (half filled with water containing three drops of liquid soap) to force the soapy water half-way up into the glass cylinder to wet the tube walls. Avoid making the mixture froth.

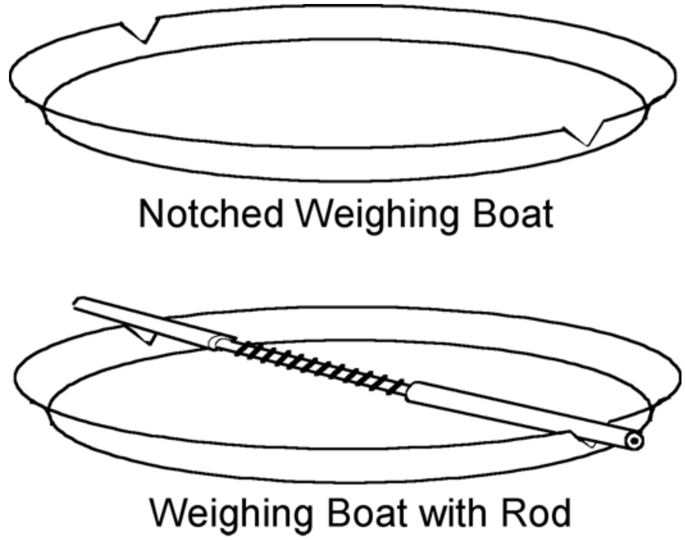

FIG. 3 Weighing Boat and Rod

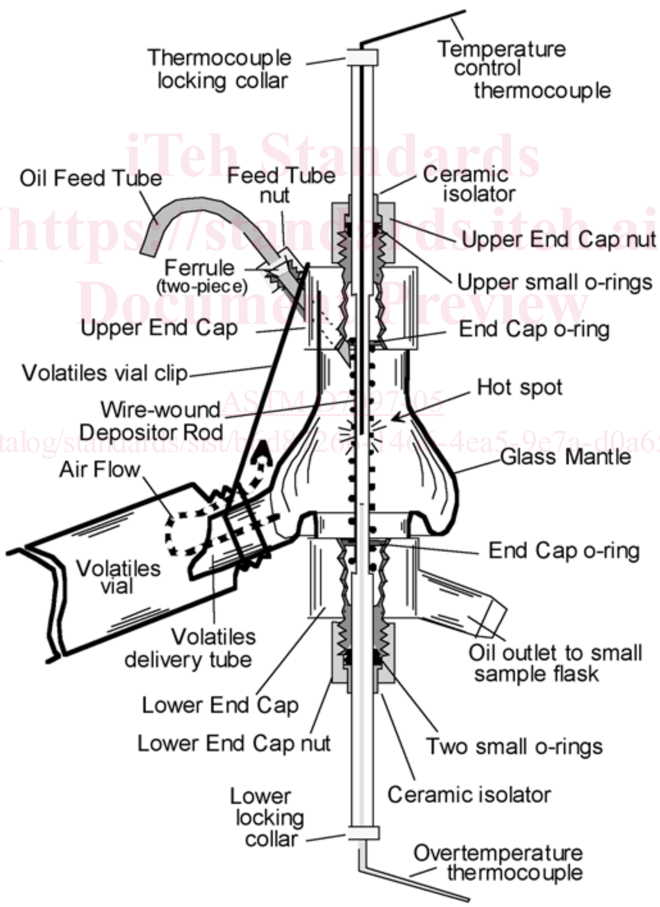

FIG. 4 Depositor Assembly (Cut-away View)

9.1.1.7 Squeeze the rubber bulb gently to bring a bubble just above the air inlet near the bottom of the bubble gage. Let the airflow catch the bubble and raise it toward the top of the bubble tube.

9.1.1.8 With a stopwatch, measure the time in seconds that it takes for the bubble to advance 5.0 mL.

9.1.1.9 Calculate the flow rate in mL/min by the following equation:

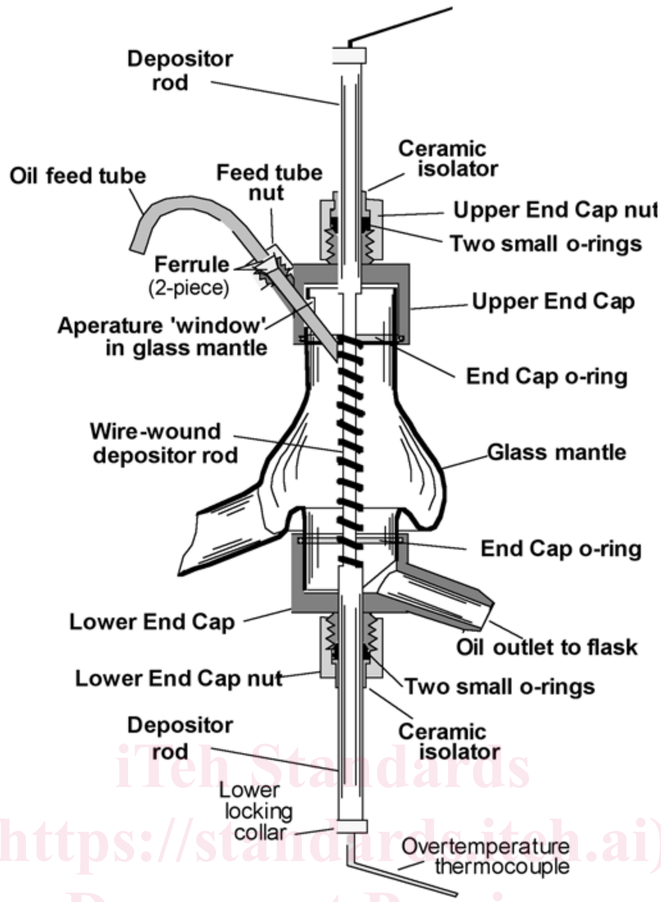

FIG. 5 Insertion of Oil Inlet Tube (Cut-away View)

$$\text{Rate} = 5.0 \text{ mL} \times 60 \text{ s/min} \div \text{bubble rise time in seconds (s)} \quad (1)$$

or more simply,

$$\text{Rate} = 300 \div \text{bubble rise time in seconds using a 5.0 mL volume} \quad (2)$$

9.1.1.10 Calibrate the mass flow controller by adjusting the needle valve controlling air flow to the mass flow meter to bring the bubble tube flow rate to 10.0 ± 0.2 mL/min. The resulting value on the mass flow controller is equivalent to 10.0 ± 0.2 mL/min.

NOTE 3—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 gage or other primary calibration device before each test to calibrate analog or digital flow meters (see Note 4).

9.1.2.2 Set up the bubble gage 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 4—A hand-held 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 gage and check that the joint is leak tight with soap solution.

9.1.2.5 Follow 9.1.1.4 through 9.1.1.8.

9.1.2.6 Adjust the needle valve on the flow meter and retest bubble rise rate to bring the flow rate to 10.0 ± 0.2 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 ± 0.01 g/min.

9.3 *Temperature Controller Setting*—Follow the technique in the manufacturer's Instrument Manual to set a test temperature of 285°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 at 285°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 or sand bath, boiling distilled water may be used to calibrate at 100°C.

9.4.2 Before each reuse of the thermocouple, clean any corrosion or other deposits from the thermocouple surface using 800 grit emery (aluminum oxide) paper or coated wire cleaning pad. The resulting cleaned surface shall show bright metal, particularly in the temperature-sensitive area at the end of the thermocouple. Do not over-clean the thermocouple