Designation: D7528 – 09

Standard Test Method for Bench Oxidation of Engine Oils by ROBO Apparatus[1]

This standard is issued under the fixed designation D7528; 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.

INTRODUCTION

Any properly equipped laboratory, without outside assistance, can use the procedure described in this test method. However, the ASTM Test Monitoring Center[2] (TMC) provides reference oils and an assessment of the test results obtained on those oils by the laboratory. By these means, the laboratory will know whether its use of the test method gives results statistically similar to those obtained by other laboratories. Furthermore, various agencies require that a laboratory utilize the TMC services in seeking qualification of oils against specifications. For example, the U.S. Army imposes such a requirement in connection with several Army engine lubricating oil specifications.

Accordingly, this test method is written for use by laboratories that utilize the portions of the test method that refer to the TMC services. Laboratories that choose not to use the TMC services may simply ignore these portions.

This test method may be modified by means of information letters issued by the TMC. In addition, the TMC may issue supplementary memoranda related to the method.

1. Scope

1.1 This test method describes a bench procedure to simulate the oil aging encountered in Test Method D7320, the Sequence IIIG engine test method. These aged oils are then tested for kinematic viscosity and for low-temperature pumpability properties as described in the Sequence IIIGA engine test, Appendix X1 of Test Method D7320.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 *Exceptions*—There are no SI equivalents for some apparatus in Section 6, and there are some figures where inch units are to be regarded as standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in Sections 7 and 8.

1.4 This test method is arranged as follows:

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[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.B0.07 on Development and Surveillance of Bench Tests Methods.

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[2] ASTM Test Monitoring Center, 6555 Penn Avenue, Pittsburgh, PA 15206-4489. www.astmtmc.cmu.edu.

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2. Referenced Documents

2.1 *ASTM Standards:*[3]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants

D4485 Specification for Performance of Engine Oils

D4684 Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature

D5293 Test Method for Apparent Viscosity of Engine Oils and Base Stocks Between −5 and −35°C Using Cold-Cranking Simulator

D7320 Test Method for Evaluation of Automotive Engine Oils in the Sequence IIIG, Spark-Ignition Engine

2.2 *SAE Standard:*[4]

SAE J300 Engine Oil Viscosity Classification

3. Terminology

3.1 *Definitions:*

3.1.1 *candidate oil*, *n*—an oil that is intended to have the performance characteristics necessary to satisfy a specification and is to be tested against that specification. **D4175**

3.1.2 *reference oil*, *n*—an oil of known performance characteristics, used as a basis for comparison.

3.1.2.1 *Discussion*—Reference oils are used to calibrate testing facilities, to compare the performance of other oils, or to evaluate other materials (such as seals) that interact with oils. **D4175**

3.1.3 *non-reference oil*, *n*—any oil other than a reference oil, such as a research formulation, commercial oil or candidate oil. **D4175**

3.1.4 *test oil*, *n*—any oil subjected to evaluation in an established procedure. **D4175**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aged oil*, *n*—a test oil after it has been subjected to the 40-h aging process in a ROBO apparatus.

3.3 *Acronyms:*

3.3.1 *ROBO*, *n*—Romaszewski Oil Bench Oxidation[5]

4. Summary of Test Method

4.1 The test oil is combined with a small amount of iron ferrocene catalyst and placed in a 1-L reaction vessel. That mixture is stirred and heated for 40 h at 170 ºC with air flowing across the liquid surface under negative pressure. In addition, nitrogen dioxide and air are introduced below the reaction surface. After cooling, the oxidized, concentrated test oil is subjected to pertinent viscometric tests. Evaporated oil is condensed in order to weigh it and calculate evaporative loss.

5. Significance and Use

5.1 This bench test method is intended to produce comparable oil aging characteristics to those obtained with ASTM TMC Sequence IIIGA matrix reference oils 434, 435 and 438 after aging in the Sequence IIIG engine test.

5.2 To the extent that the method generates aged oils comparable to those from the Sequence IIIG engine test, the measured increases in kinematic and MRV viscosity indicate the tendency of an oil to thicken because of volatilization and oxidation, as in the Sequence IIIG and IIIGA (see Appendix X1 in Test Method D7320) engine tests, respectively.

5.3 This bench test procedure has potential use in specifications and classifications of engine lubricating oils, such as Specification D4485.

6. Apparatus

6.1 *Balances*:

6.1.1 *Analytical Balance*—Capable of weighing 200 g with a minimum indication resolution of 0.1 g.

6.1.2 *Analytical Balance*—Capable of weighing 0.1 g with a minimum indication resolution of 0.001 g.

6.2 *Fume Hood*, that vents to the outside atmosphere (see Section 8).

6.3 *Reaction Vessel* (ACE Glass, Inc. part number D120676),[6,7] a 1-L, thick-walled glass vessel having a nominal 100-mm inner diameter and with a bottom, sample/drain valve. The lower half has an Instatherm[7,8] coating, rated at approximately 400 W, for heating the test mixture. A diagram is shown in Fig. A1.1.

6.4 *Vessel Head*—The vessel head is a stainless steel plate of sufficient diameter to completely cover the lower glass vessel and provide ample material for a sturdy mounting system. Reimel Machine, Inc. part number RMI-1002-DH[7,9] has been shown to be suitable for this application. The vessel head may also be constructed as described in Annex A2. Users may also source some parts from Reimel Machine, Inc. and some in-house. Ensure the plate has a center hole for an agitator shaft and threaded ports to allow filling and for the attachment of air/nitrogen dioxide lines, vacuum control and relief valves, and a temperature probe. Fig. A2.1 defines the locations of these ports. Mill the bottom surface of this stainless steel plate to accept a polytetrafluoroethylene (PTFE) ring seal for centered attachment of the glass vessel as described in Annex A3. Reimel Machine, Inc. part number RMI-1007-DH[7,9] has been found suitable for this purpose.

[3] 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] Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, http://www.sae.org.

[5] Kinker, B. G., Romaszewski, R. A., and Palmer, P. A., "ROBO–A Bench Procedure to Replace Sequence IIIGA Engine Test," *Journal of ASTM International (JAI)*, Vol 4, No. 10, 2007, Paper ID JAI 100916. Available online from www.astm.org.

[6] The sole source of supply of the apparatus known to the committee at this time is Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ 08362-0688.

[7] If you are aware of alternative suppliers, please provide this information to ASTM. Your comments will receive careful consideration at a meeting of the responsible technical committee[1] which you may attend.

[8] Instatherm is a registered trademark of Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ 08362-0688.

[9] The sole source of supply of the apparatus known to the committee at this time is Reimel Machine, Inc., 2575 Wyandotte Rd., Willow Grove, PA 19090.

6.5 *Stirrer Motor*—An electric motor with drill chuck collet capable of sustained operation at 200 ± 5 r/min.

6.6 *Stirrer*—An 8-mm diameter stainless steel rod, 30-mm long with a means of attaching a blade assembly at the bottom. The turbine blade assembly diameter is 2.58 in. with 1.4-mm thick blades attached at a 45° pitch with an overall blade height of 0.985 in. Construct the stirrer as described in Annex A4. Reimel Machine, Inc. part number RMI-1001-DH[7,9] has been found suitable for this purpose. Attach the stirrer to the reactor head by means of a packing gland constructed as described in Annex A5. Reimel Machine, Inc. part number RMI-1004-DH[7,9] has been found suitable for this application. Attach the stirrer to the stirrer motor by inserting the 8-mm steel rod through the opening in the reactor head and the packing gland, and insert PTFE rope packing to create a seal. Position the blade 6 mm from the bottom of the vessel.

6.7 *Air Supply System*—Capable of delivering an uninterrupted flow of dry air into the test oil via a subsurface feed throughout the reaction time period. An in-line, desiccant-charged, drying system has been found suitable. Ensure the subsurface feed tube opening remains below the surface of the test fluid for the duration of the test.

NOTE 1—As the amount of test oil remaining at the end of the test is not always known at the beginning of the test, it is advisable to configure the dry-air tube location such that the opening of the tube is as close to the agitator and as close to the bottom of the reactor as practical (without contacting the agitator or blocking the tube opening).

6.8 *Graduated Tube* (Ace Glass, Inc., part number D120677),[6,7] 12-mL capacity, with 0.1 mL graduations and having appropriate provisions for connection to the reaction vessel's subsurface gas delivery system—see Annex A6 for more details. By receiving liquid phase nitrogen dioxide from a gas bottle, this tube allows measurement of nitrogen dioxide depletion from the tube over the course of the reaction.

6.9 *Temperature Control System*—A controller and probe capable of being programmed to control reaction temperature via low output wattage at or below 40 VAC and with an operational hysteresis of 0.1 °C using an on/off algorithm. Alternatively, a proportional-integral-derivative (PID) algorithm may also be used. Position the temperature probe tip so that it is level with the bottom of the turbine blade with a distance of 8 mm between the probe center and the blade edge.

6.9.1 As the temperature may not be uniform throughout the reactor, it is important from the point of view of precision that the temperature is always monitored and controlled at the specified position inside the reactor. When reassembling the reactor for a new run, reposition the probe, if necessary, as it is easily bent.

6.10 *Flow Meters*:

6.10.1 *Acrylic Block Airflow Meter* (King Instrument Co., 7520 Series, Order number 2C-17),[7,10] having a scale of 0.4 to 4 Standard Cubic Feet per Minute (SCFM), with ¼-in. NPT threaded female pipe end. It is used for measuring air flow in 10.3.2. The machined fitting for the top of the flow meter shall accommodate the vacuum line from the condenser to the reactor with a ⅜-in. inside diameter or larger. The machined fitting for the bottom of the flow meter shall accommodate the ¼-in. vacuum control valve.

NOTE 2—SCFM is the volumetric flow rate of a gas corrected to *standardized* conditions of temperature, pressure, and relative humidity, thus representing a precise mass flow rate. However, the definitions of *standard* conditions vary. In this method, the flow meter is calibrated with air at *standard* conditions defined as a temperature of 70°F, a pressure of 14.6 psia and 0 % relative humidity.

6.10.2 *Airflow Meter*, with a scale calibrated in mL/min for measuring subsurface airflow of 185 mL/min in 10.3.1 and 10.3.2.

6.11 *Vacuum System*—A pump with a free air capability of at least 160 L/min is required to ensure a constant air flow across the reaction surface in the vessel of 2.0 ± 0.1 SCFM with 61 kPa vacuum for 40 h. Instructions for constructing the vacuum plumbing for the vessel are given in Annex A7. As explained in Annex A7, it is critical to follow these instructions precisely.

6.12 *Vacuum Control Valve*—A stainless steel needle valve with ¼-in. outside diameter tube connections and a flow coefficient (Cv) of 0.37. A McMaster-Carr Supply Company needle valve part number 45585K86[7,11] has been found suitable for this application.

6.13 *Vacuum Trap System*—Supplies coolant at an inlet temperature < 20 °C to the vacuum trap condensers in order to remove vapors from the effluent prior to entering (and possibly damaging) the vacuum system and has a means of recovering the distillate for weighing. Redundant (serial) condensers are beneficial as long as the required airflow across the reaction surface is maintained. Annex A8 provides information on two systems that have been found to be satisfactory.

6.14 *Time Controller*—A timing device accurate to 1 min is used to deactivate the heat source.

6.15 *Precision Needle Valve*—Having a low Cv for precise control of the flow of nitrogen dioxide. Examples of valves that have been found satisfactory are given in Appendix X3.

6.16 *Beaker*—300-mL capacity.

6.17 *Glass Jar*—250-mL capacity which can be sealed.

6.18 *Shaker*—Use either a reciprocal or an elliptical shaker.

6.19 *Assembled ROBO Apparatus*—Fig. X4.1 shows an example of an assembled ROBO apparatus. However, because it is assembled from different components, some of which are site specific (e.g., geometry of fume hood, local safety considerations, use of different parts such as temperature controllers, and so forth), there is no standard ROBO apparatus assembly. As an aid to building and setting up a new ROBO apparatus, a package of information is available on the TMC website.[2] This (non-mandatory) information supplements that given in Section 6. An index to the contents of this information package is given in Appendix X5.

7. Reagents and Materials

7.1 *Liquid Nitrogen Dioxide*—Produces a reddish-brown gas with a pungent odor. (**Warning—**VERY TOXIC if inhaled

[10] The sole source of supply of the apparatus known to the committee at this time is King Instrument Co., 12700 Pala Drive Garden Grove, CA 92841.

[11] The sole source of supply of the apparatus known to the committee at this time is McMaster-Carr Supply Company, P.O. Box 740100, Atlanta, GA 30374-0100.

or ingested. Explosive if mixed with combustible material. Irritating to eyes and respiratory system. Danger of very serious irreversible health effects.)

7.2 *Iron Ferrocene*—98 % or higher purity. (**Warning—**Do not breathe dust. Harmful if swallowed.)

7.3 *Oil*—100 Neutral, API Group II, for mixing with iron ferrocene catalyst.

7.4 *Cleaning Solvent*—Commercial heptanes, or similar solvents that evaporate without leaving a residue, are suitable. (**Warning—**flammable.)

7.5 *Acetone*—A technical grade of acetone is suitable provided it does not leave a residue upon evaporation. This is used for a final cleaning rinse. Acetone will degrade fluoroelastomer seals and can dissolve or deteriorate acrylics. (**Warning—**flammable.)

7.6 *Dry Air*—Desiccated air is suitable.

7.7 *Reference Oils*—The reference oils TMC 434, 435, and 438 are required for setting up the ROBO apparatus test stand (see Section 9). The TMC[2] maintain and distribute these oils. These oils are formulated or selected to represent specific chemical types or performance levels, or both.

7.7.1 The TMC is responsible for managing a system that ensures the performance and formulation consistency of the reference oils. Store the reference oils in locations where the ambient temperature does not exceed 32 °C. Under these conditions, the expected shelf life of a reference oil is five years. In some circumstances, however, the TMC may specify a shelf life longer than five years. In such cases, they will use documented analysis procedures to justify the longer shelf life.

7.7.2 Unless specifically authorized by the TMC, do not analyze TMC reference oils, either physically or chemically. The testing laboratory tacitly agrees to use the TMC reference oils exclusively in accordance with the TMC's published Policies for Use and Analysis of ASTM Reference Oils, and to run and report the reference oil test according to TMC guidelines.

NOTE 3—Policies for the Use and Analysis of ASTM Reference Oils are available from the TMC.[2]

8. Hazards

8.1 *Specific Hazards*—Due to nitrogen dioxide toxicity, with the exception of weighing, perform steps 10.3 to 10.8 of the procedure in the fume hood. See also 7.1.

9. Reference Oil Testing and Test Stand Calibration

9.1 *Set-up Guidelines for a New ROBO Apparatus Test Stand*:

9.1.1 Obtain the three reference oils TMC 434, 435, and 438 (or reblends approved by the ROBO surveillance panel) from the TMC.[2] (See 7.7.2 for conditions of use for the TMC reference oils.)

9.1.2 Test the three oils according to the procedure described in Section 10 in the following suggested sequence: TMC 434, 435, 438.

9.1.3 Determine the viscometric properties of the aged reference oils as described in Sections 12 and 13 (with the exception of volatiles which are excluded from reference oil testing).

9.1.4 Compare the viscometric proprieties of the aged reference oils with the guidelines given in Table 1.

9.1.5 If the viscometric measurements of all three aged TMC reference oils fall within the guidelines given in Table 1, consider the ROBO apparatus test stand to be operating in an acceptable range.

9.1.6 If any of the viscometric measurements of any of the three aged reference oils fall significantly outside the guidelines in Table 1, evaluate the operational validity of the tests, rectify any obvious problems, and repeat 9.1.2 to 9.1.4 for the reference oil(s) that failed to meet the guidelines.

9.1.7 If the problem is not obvious, re-check the viscometric measurements and all test-related equipment (e.g., air-flow gauges, vacuum gauge, temperature controller, thermocouple, vacuum pump, plumbing integrity, any evidence of leaks)—see also A9.4. Following the re-check, and provided the control valve setting has not been altered from that set initially, repeat 9.1.2 to 9.1.4 for the reference oil(s) that failed to meet the guidelines.

9.1.8 If the control valve setting was altered, repeat 9.1.2 to 9.1.4 for all three reference oils.

9.2 *For Laboratories Using the TMC Services*—This section describes the involvement of the TMC in respect to maintaining ROBO apparatus test stand calibration status for ROBO engine oil testing. Those laboratories choosing not to use these services may ignore this section.

9.2.1 *Reference Oil Test Frequency*—Periodic calibration with the TMC is required. This involves testing one of the reference oils on the stand according to the ASTM Test Monitoring Center's requirements.[2]

9.2.1.1 Prior to conducting a reference oil test, procure a supply of reference oil directly from the TMC. The reference oils are usually supplied directly to a testing laboratory under code numbers to ensure that the laboratory is not influenced by prior knowledge of acceptable results in assessing the test results. The TMC will determine which specific reference oil the laboratory shall test.

9.2.1.2 Assign a test number to each ROBO test.

9.2.1.3 During the time of conducting a reference oil test on one test stand, non-reference oil tests may be conducted on other previously calibrated stands.

9.2.2 *Reporting of Reference Oil Test Results*—Report the results of all reference oil tests to the TMC according to the following directive:

9.2.2.1 Transmit results to the TMC within 5 days of test completion via electronic data transfer protocol as outlined in

TABLE 1 Set-up Guidelines for ROBO Apparatus/Test Stand

TMC Reference Oil Code	Viscosity at 40 °C,[A] mm^2/s	Yield Stress at –30 °C,[B] Pa	Apparent Viscosity at –30 °C,[B,C] mPa·s
438	97 to 122	<35	22 000 to 35 000
434	107 to 138	<35	39 000 to 50 000
435	135 to 178	>35[D]	>70 000

[A] Determine by Test Method D445.
[B] Determine by Test Method D4684.
[C] This viscosity is commonly referred to as the Mini Rotary Viscometer (MRV) viscosity (see also 12.2.1.1).
[D] Yield stress may be missed due to Test Method D4684 test variability.

the Data Communication Committee, Electronic Test Report Transmission Model (ETRTM). The ETRTM can be obtained from the TMC.[2]

9.2.3 *Evaluation of Reference Oil Test Results*—The TMC evaluates the reference-oil test results for both operational validity and statistical acceptability. The TMC may consult with the test laboratory in case of difficulty, as follows:

9.2.3.1 Upon receipt of the reference-oil test results from the test laboratory, the TMC will evaluate the laboratory's reported operational parameters for compliance with the current test method. For operationally valid tests, the TMC will then evaluate the pass/fail parameters for statistical validity according to the ROBO calibration guidelines. The TMC will send a test confirmation report to the test laboratory indicating the overall validity of the calibration test results, and disclosing the non-blind industry reference oil code.

9.2.3.2 In the event the reference oil test is unacceptable, the test laboratory shall provide an explanation of the problem relating to the failure. If the problem is not obvious, carry out the re-checks described in 9.1.7. Following the re-checks, the TMC will assign another reference oil for testing by the laboratory. If this reference oil test is unacceptable, a reassessment of the stand set-up, as described in 9.1 may be necessary.

9.2.3.3 The TMC will decide, with consultation as needed with industry experts (testing laboratories, members of the ASTM Technical Guidance Committee and of the surveillance panel, and so forth), whether the reason for any failure of a reference oil test is a false alarm, testing apparatus, testing laboratory, or industry-related problem. The ROBO surveillance panel shall adjudicate all industry problems.

10. Procedure

10.1 *Vacuum Control Valve Setting*—For a new ROBO apparatus test stand, set the vacuum control valve as described in Annex A9. The control valve setting is critical as it affects the severity of the test. For all subsequent runs involving test oils, use exactly the same control valve setting to that used in 9.1.5, or, for those laboratories using the TMC services, for the last successful calibration run.

10.2 *Catalyst Preparation*:

10.2.1 Weigh 0.1 ± 0.001 g of iron ferrocene (see warning in 7.2) into an appropriate container such as a 250-mL glass jar.

10.2.2 Add 99.9 ± 0.1 g of API Group II 100 Neutral oil to obtain 0.100 ± 0.001 mass % iron ferrocene.

10.2.3 Mix thoroughly, until the catalyst is completely in solution, by shaking on a reciprocal shaker for at least 10 min, or in an elliptical shaker for at least 5 min.

10.3 *Vessel Seal Check*:

10.3.1 Start subsurface dry-air flow at a rate of 185 mL/min.

10.3.2 On an assembled vessel, install the flow meter between the top connection of the vacuum control valve and the vacuum source. Apply vacuum to the vessel and block the vacuum relief orifice long enough to assure the system will attain 85 kPa with a subsurface airflow of 185 mL/min. The air flow meter shall read less than 0.6 SCFM.

10.4 *Preset Vacuum Flow*—Apply vacuum to the vessel and set the air flow through the reactor to 2.0 ± 0.1 SCFM by bleeding air, if needed, into the vacuum line between the vacuum source and the condenser. Maintain the vacuum pressure at 61 ± 1.7 kPa by adjusting the vacuum relief valve. Once these parameters are set, remove the flow meter from the system. Shut off the vacuum.

10.5 *Sample Preparation and Charging Nitrogen Dioxide*:

NOTE 4—Steps 10.5.1 to 10.5.3 may be carried out in any order or simultaneously.

10.5.1 *Sample Preparation*—Introduce 3.0 ± 0.1 g of prepared iron ferrocene catalyst solution and 197.0 ± 1.0 g test oil to the reaction vessel. See Appendix X1 for suggested mixing procedures.

NOTE 5—The total mass of oil in the reactor is 200 ± 1.0 g (197.0 ± 1.0 g from the test oil and 3.0 g from the catalyst solution).

10.5.1.1 Start the stirrer motor and agitate at 200 ± 5 r/min.

10.5.2 Make the electrical connections to the heater. (**Warning—**To avoid electric shock and possible ignition spark, check that the power is de-energized before making electrical connections.)

10.5.3 *Charging Nitrogen Dioxide*—Transfer 2.0 ± 0.1 mL of liquid nitrogen dioxide (see Section 8 and warning in 7.1) into the graduated tube. See Appendix X2 for examples of how the transfer may be made.

10.6 *Oil Aging*:

10.6.1 *General*—Begin the oil aging by setting the time and temperature and turning on the vacuum.

10.6.1.1 Complete steps 10.6.2 to 10.6.5 within 1 min; the order in which they are carried out is not important.

10.6.2 Set the time controller to 40 h to initiate the oil aging.

10.6.3 Set the temperature controller to 170 °C and commence heating.

10.6.4 Adjust the temperature controller voltage output to 25 to 40 V.

10.6.5 Turn the vacuum system on.

10.6.6 Adjust the nitrogen dioxide precision needle valve to allow introduction of nitrogen dioxide in a controlled and gradual manner into the inlet flow stream. Ensure that the nitrogen dioxide is completely depleted from the tube and introduced into the reactor within 12 ± 1 h.

10.6.6.1 Because changes to the nitrogen dioxide flow rate can affect precision, it is imperative that nitrogen dioxide be introduced to the reactor in a controlled and gradual manner. Using a flow rate of 0.167 mL/h, monitor nitrogen dioxide depletion closely in the first 2 to 4 h, the aim being to introduce 0.5 mL during that time period. Introduce the remaining 1.5 mL at a similar flow rate, ensuring that the total of 2.0 mL is delivered between 11 and 13 h.

10.7 *Shutdown*:

10.7.1 At the end of the 40-h cycle, allow the system to cool to room temperature while maintaining the airflow and agitation.

10.7.2 Turn off the vacuum. (The vacuum flow can be turned off at any time after completion of the 40-h cycle.) Bleed the pressure by opening a port, e.g., the sample addition port. Drain the aged oil into a suitable container.

10.8 *Mass Percent Volatiles Collected*:

10.8.1 Drain the condensed liquid from the vacuum trap system into a tared vessel. Determine and record the mass of the condensed liquid to the nearest 0.1 g.

10.8.2 Calculate as follows:

$$\text{Mass \% volatiles, \% m/m} = 100\,\frac{\text{M(volatiles)}}{\text{M(fresh)}} \quad (1)$$

where:

M(fresh) = 200 g = the mass of fresh oil added to the reactor in 10.5.1, and

M(volatiles) = mass, g, of condensate collected in 10.8.1.

NOTE 6—The significance of the % volatiles parameter is under investigation.

11. Cleaning

11.1 Clean the reaction vessel with cleaning solvent (see warning in 7.4).

11.1.1 Scrub any residual material off the glass surface while taking care not to scratch the inside of the vessel. Perform a final rinse with acetone (see warning in 7.5).

11.2 Clean the vacuum control valve.

11.2.1 Flush the valve with cleaning solvent or carburetor cleaner, followed with an acetone rinse to remove and avoid any carbon deposits that could reduce or plug the valve orifice.

11.2.2 Additional optional cleaning may be needed in cases where there is insufficient vacuum flow (see 10.4). If vacuum flow is sufficient, skip to step 11.3.

11.2.2.1 Disassemble the valve and remove any carbon deposits from the plug and inside seat of the valve body.

11.2.2.2 Flush as in 11.2.1.

11.2.2.3 Reassemble the vacuum control valve, ensuring that the valve setting is at exactly the same position to that used in 9.1.5 or, if using the TMC services, to that used at the last successful calibration run.

11.3 Clean the underside of the reactor cap and all shafts or probes protruding downward into the vessel with cleaning solvent and a lightweight, lint-free towel. Rinse with acetone.

11.4 Ensure that subsurface air supply lines are clear, then clean them with cleaning solvent and reassemble when dry.

11.5 Clean the acrylic block flow meter with cleaning solvent. Do not use acetone which can dissolve or deteriorate acrylics.

12. Calculations and Determination of Test Results

12.1 *Increase in Kinematic Viscosity at 40 °C*:

12.1.1 Calculate as follows:

$$\text{Percent viscosity increase (PVIS)} = 100\,\frac{[\text{KV(aged)} - \text{KV(fresh)}]}{\text{KV(fresh)}} \quad (2)$$

where:

KV(aged) = kinematic viscosity, mm^2/s, at 40 °C of the aged oil as determined by Test Method D445, and

KV(fresh) = kinematic viscosity, mm^2/s, at 40 °C of the fresh oil as determined by Test Method D445.

12.2 *Low-Temperature Viscometric Properties*:

12.2.1 Using Test Method D5293, measure the Cold Cranking Simulator (CCS) viscosity of the ROBO-aged oil at the temperature specified for the SAE W grade of the fresh oil. This temperature can be found in the SAE J300 Viscosity Classification System (hereafter referred to as SAE J300).

12.2.1.1 If the measured CCS viscosity is less than or equal to the maximum CCS viscosity specified in SAE J300 for the SAE W grade of the fresh oil, measure the MRV viscosity by Test Method D4684 at the MRV temperature specified in SAE J300 for the SAE W grade of the fresh oil.

12.2.1.2 If the measured CCS viscosity is higher than the maximum CCS viscosity specified in SAE J300 for the SAE W viscosity grade of the fresh oil, measure the MRV viscosity by Test Method D4684 at 5 °C higher than the MRV temperature specified in SAE J300 for the original SAE W viscosity grade of the fresh oil (i.e., at the MRV temperature specified in SAE J300 for the next higher SAE W viscosity grade).

13. Report

13.1 *Report Forms*—For TMC reference oil tests, use the standardized report form set and data dictionary.

NOTE 7—The non-reference oil test results should also be reported on these same forms if the results are intended to be submitted as candidate oil results against a specification.

13.1.1 Report reference oil test results according to the protocols described in 9.2.2.

13.2 *Reporting Units*—Report results in SI units.

13.3 Report the following:

13.3.1 Kinematic viscosity at 40 °C, by Test Method D445, of the test oil before and after aging.

13.3.1.1 Report to two decimal places for viscosities between 10 and 100 mm^2/s and to one decimal place for viscosities >100 mm^2/s.

13.3.2 Percent increase in kinematic viscosity at 40 °C after aging (PVIS)—see 12.1.

13.3.2.1 Report to nearest 0.1 %.

13.3.3 SAE W grade of the fresh oil.

13.3.4 The CCS viscosity and temperature of measurement of the ROBO-aged oil by Test Method D5293.

13.3.5 The MRV viscosity, yield stress and temperature of measurement of the aged oil by Test Method D4684—see 12.2.1.1 and 12.2.1.2.

14. Precision and Bias [12]

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory tests results is given in Table 2.

14.1.1 *Intermediate Precision Conditions*—Conditions where test results are obtained with the same test method using the same oil, with changing conditions such as operators, measuring equipment, test apparatus, and time.

NOTE 8—Intermediate precision is the appropriate term for this test method, rather than repeatability, which defines more rigorous within-laboratory conditions.

14.1.1.1 *Intermediate Precision Limit (i.p.)*—The difference between two results obtained under intermediate precision conditions that would in the long run, in the normal and correct conduct of the test method, exceed the values shown in Table 2 in only one case in twenty. When only a single test result is

[12] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report D02-1660.

TABLE 2 Test Precision[A]

Variable	Intermediate Precision		Reproducibility	
	$S_{i.p.}$[B]	i.p.[C]	S_R[B]	R[C]
PVIS[D]	0.191	0.535	0.267	0.748
MRV viscosity[D]	0.25	0.70	0.40	1.12

[A] These statistics are based on results obtained from an interlaboratory program in which seven samples were tested in seven laboratories on ten test rigs (see 14). The samples consisted of SAE 5W-XX and 10W-30 multigrade engine oils including ASTM Test Monitoring Center Reference Oils 434, 435 and 438.

[B] S = Standard deviation.

[C] This value is obtained by multiplying the standard deviation by 2.8.

[D] The original units for PVIS are percent viscosity increase. The original units for MRV viscosity are mPa·s. These parameters are transformed using ln(result). When comparing two test results on these parameters, first apply this transformation to each test result. Compare the absolute difference between the transformed results with the appropriate (intermediate precision or reproducibility) precision limit.

available, the Intermediate Precision Limit can be used to calculate a range (test result ± Intermediate Precision Limit) outside of which a second test result would be expected to fall about one time in twenty.

14.1.2 *Reproducibility Conditions*—Conditions where test results are obtained with the same test method using the same test oil in different laboratories with different operators using different equipment.

14.1.2.1 *Reproducibility Limit (R)*—The difference between two results obtained under reproducibility conditions that would, in the long run, in the normal and correct conduct of the test method, exceed the values in Table 2 in only one case in twenty.

14.2 *Bias*—No estimate of the bias for this procedure is possible because the performance results for an oil are determined only under the specific conditions of the test and no absolute standards exist.

15. Keywords

15.1 evaporative loss; low-temperature pumpability; MRV viscosity; oil aging; oil oxidation; oil viscosity; ROBO test; sequence IIIG test; sequence IIIGA test; volatiles

ANNEXES

(Mandatory Information)

A1. REACTION VESSEL

A1.1 A diagram of the reaction vessel (ACE Glass, Inc. part number D120676[6,7]) is shown in Fig. A1.1.

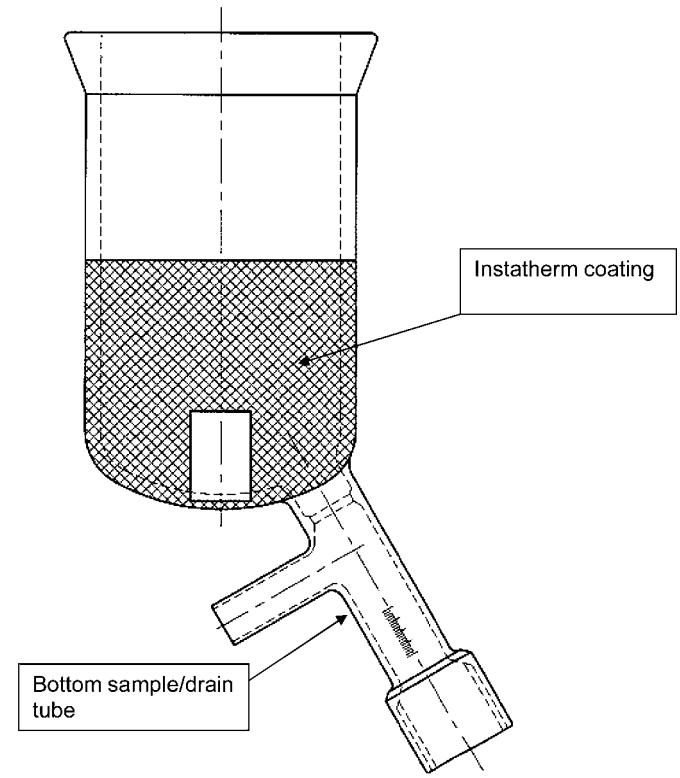

FIG. A1.1 Reaction Vessel

A2. REACTION VESSEL HEAD

A2.1 Construct the vessel head as described in Fig. A2.1. Reimel Machine, Inc. part number RMI-1002-DH[7,9] has also been shown to be suitable for this application.

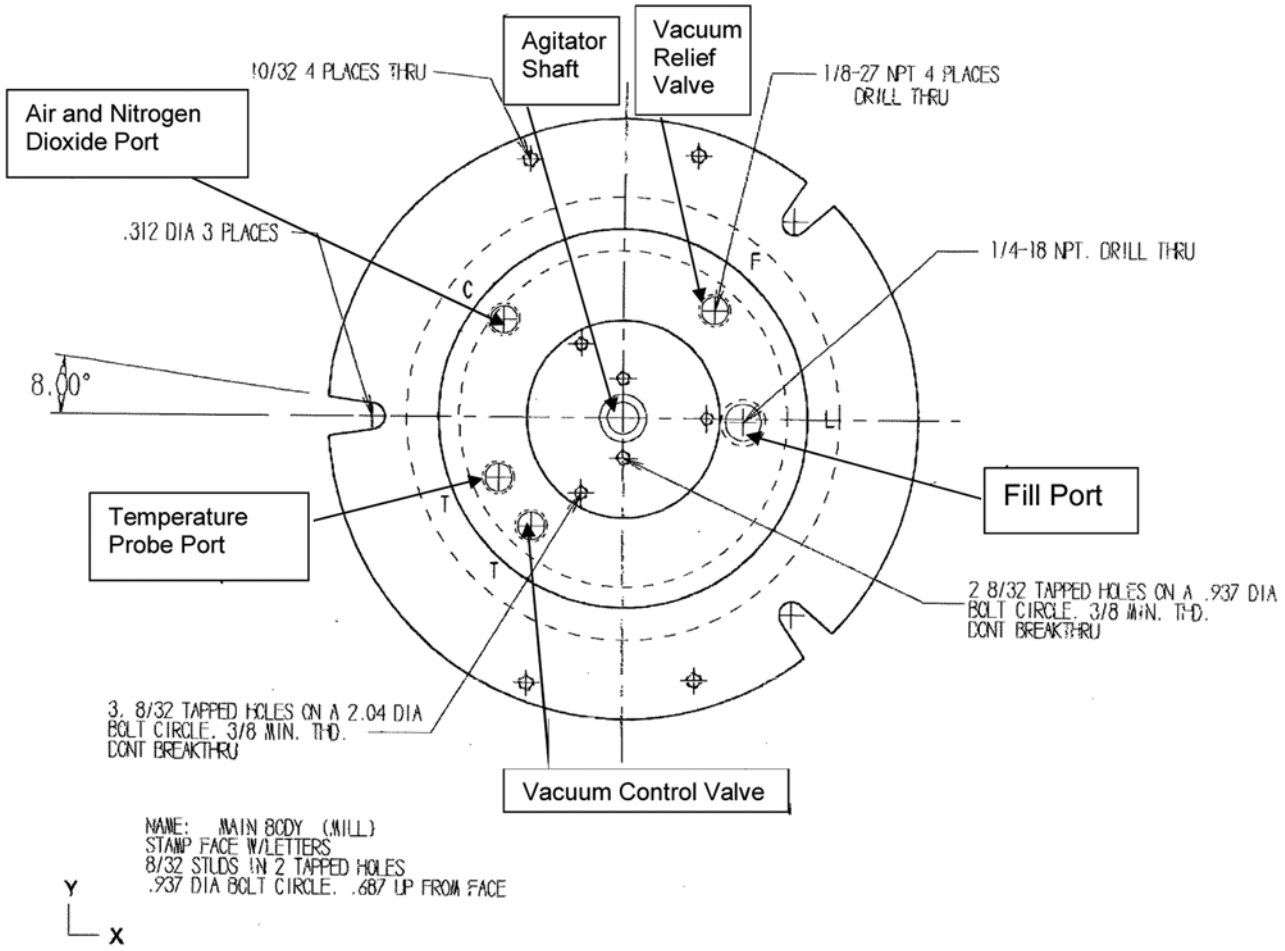

FIG. A2.1 Reaction Vessel Head (all dimensions are in inches)

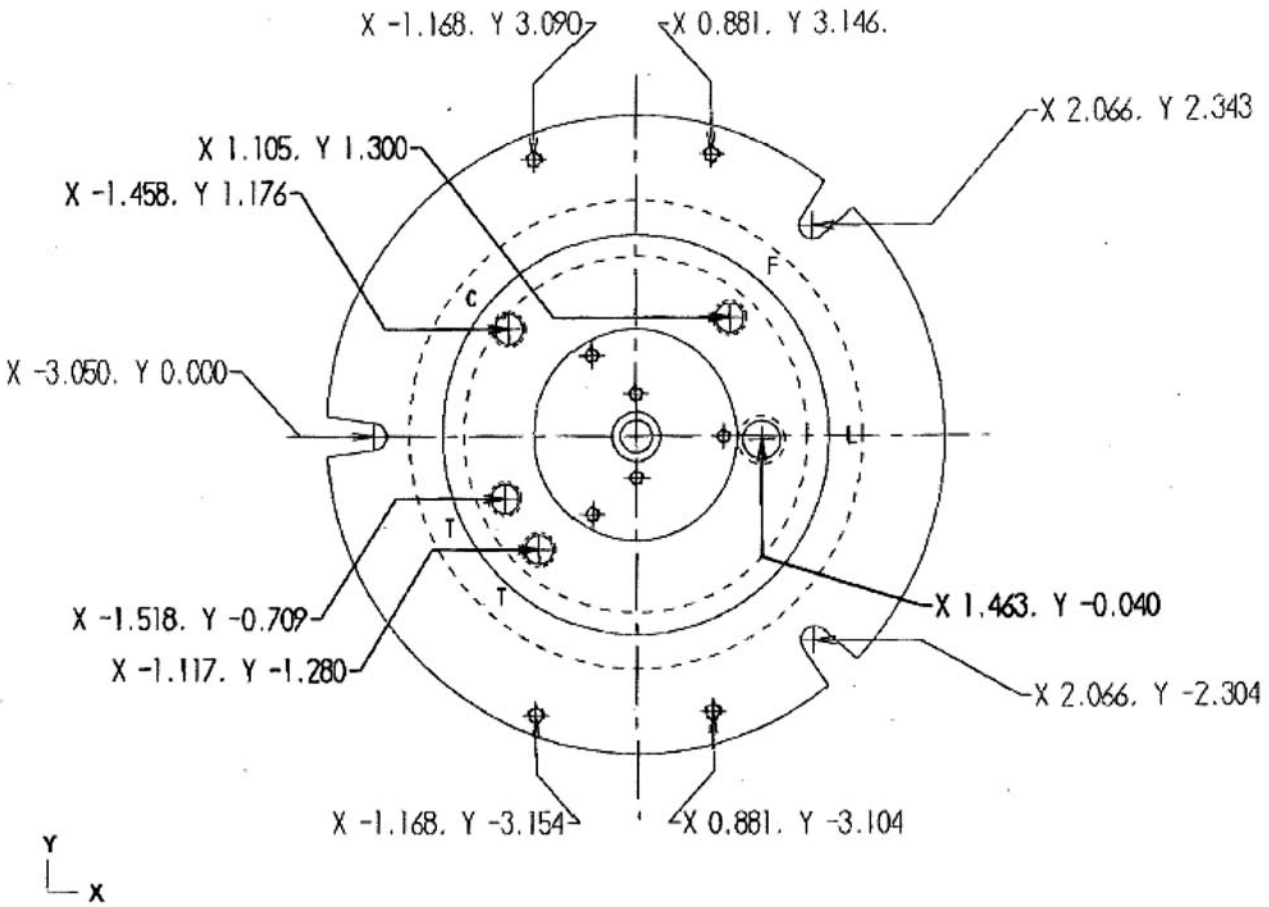

FIG. A2.1 Reaction Vessel Head (all dimensions are in inches) *(continued)*

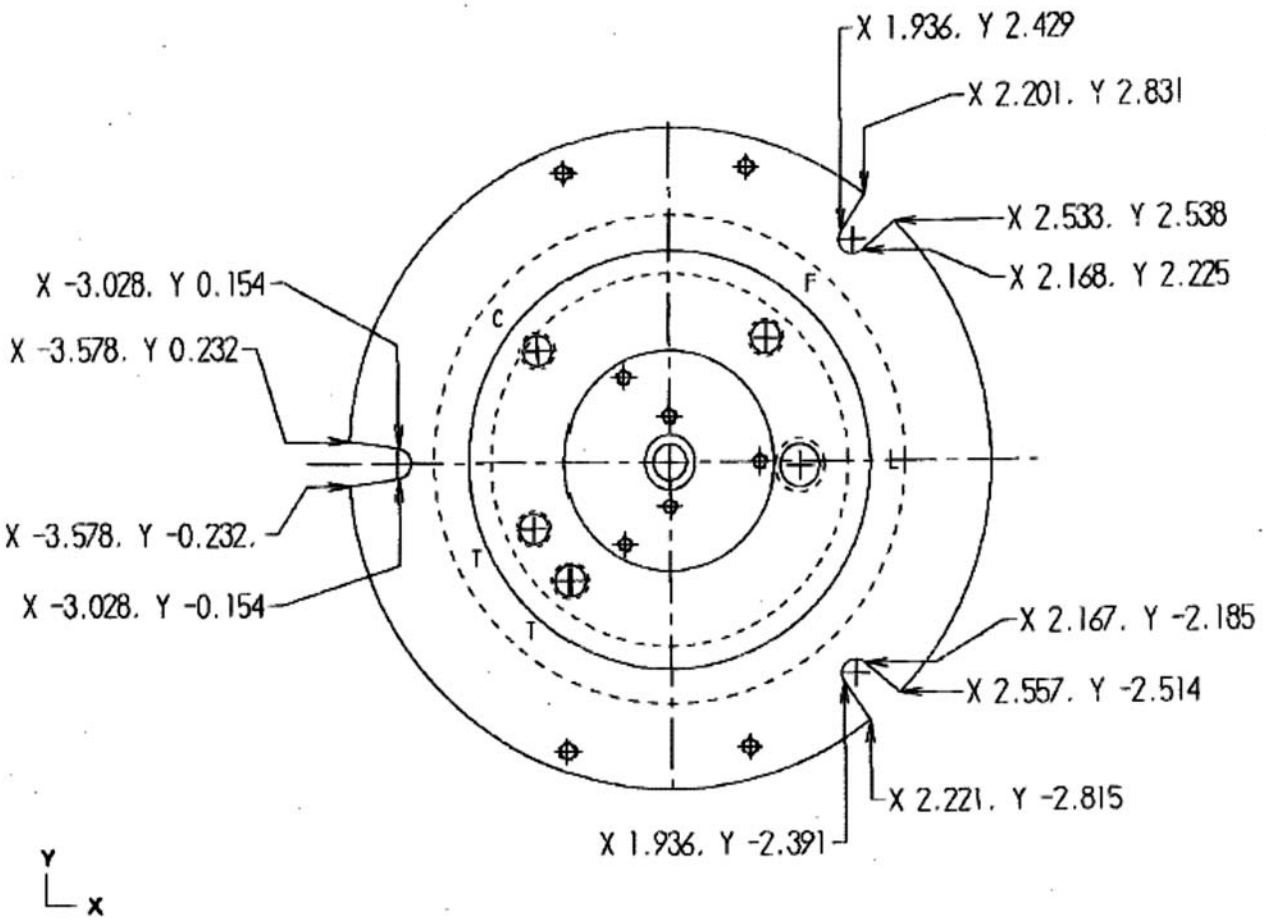

FIG. A2.1 Reaction Vessel Head (all dimensions are in inches) *(continued)*

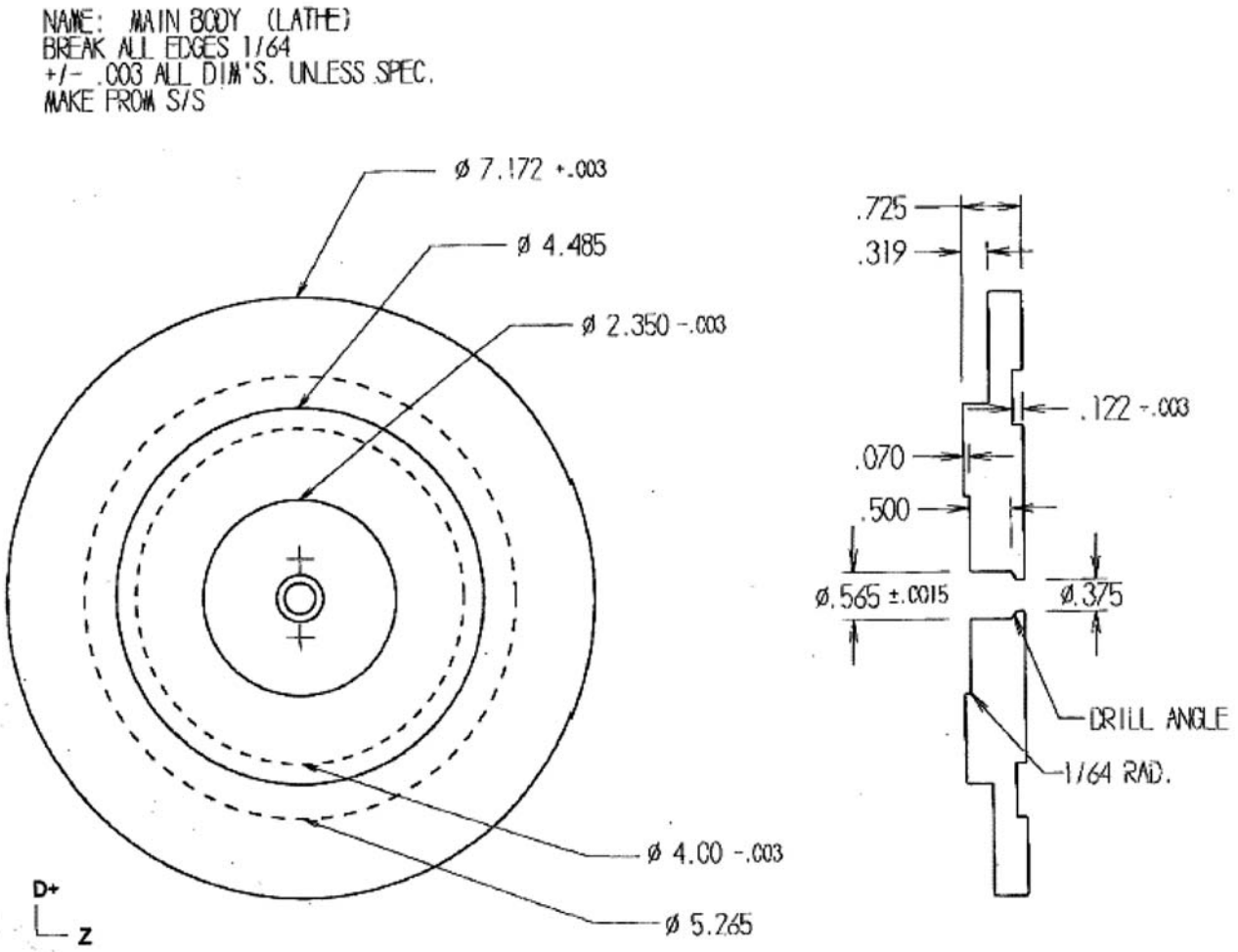

FIG. A2.1 Reaction Vessel Head (all dimensions are in inches) *(continued)*

A3. REACTION VESSEL-TO-HEAD SEAL

A3.1 Fig. A3.1 shows details and dimensions for the reaction vessel-to-head seal. Reimel Machine, Inc. part number RMI-1007-DH[7,9] has been found suitable for this purpose.

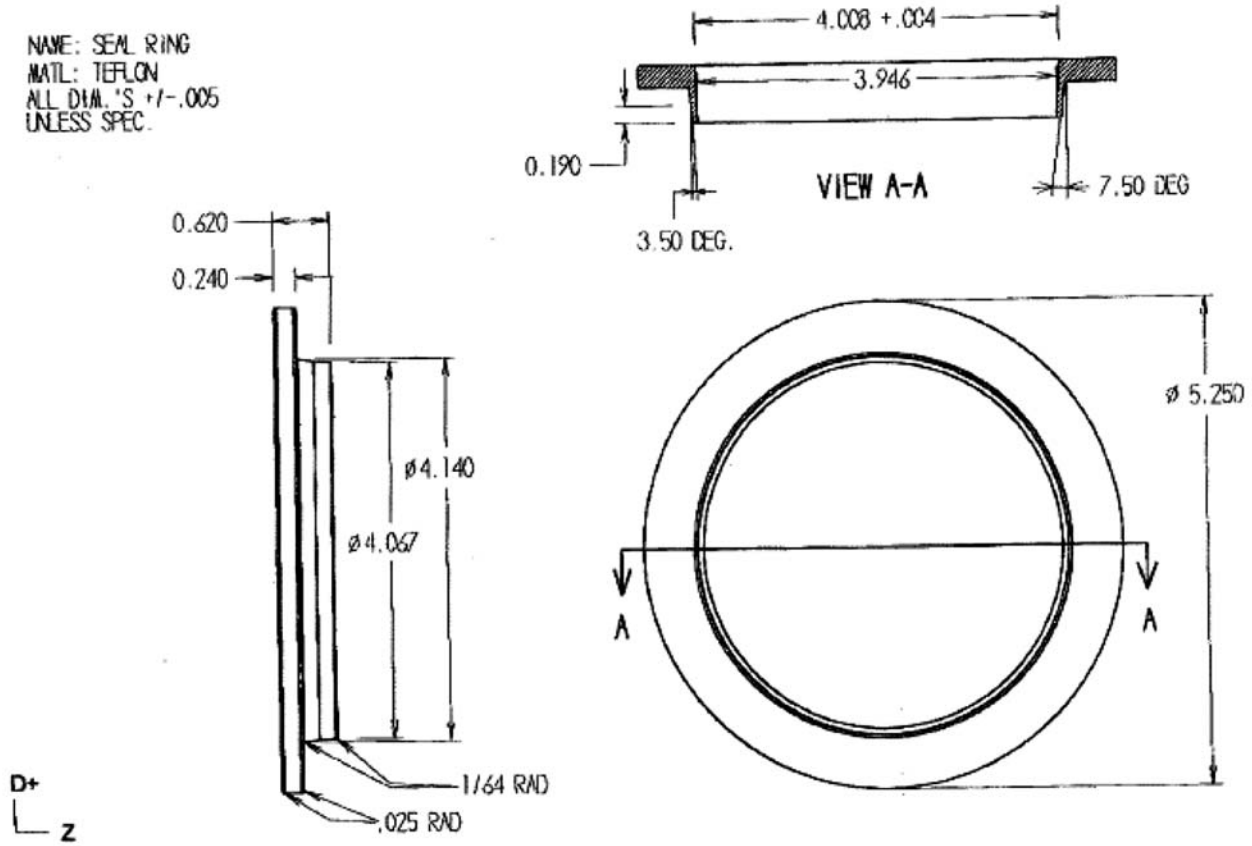

FIG. A3.1 Reactor Vessel to Head Seal (all dimensions are in inches)

A4. AGITATOR TURBINE BLADE

A4.1 Fig. A4.1 shows details and dimensions of the stainless steel agitator turbine blade. Reimel Machine, Inc. part number RMI-1001-DH[7,9] has been found suitable for this purpose.

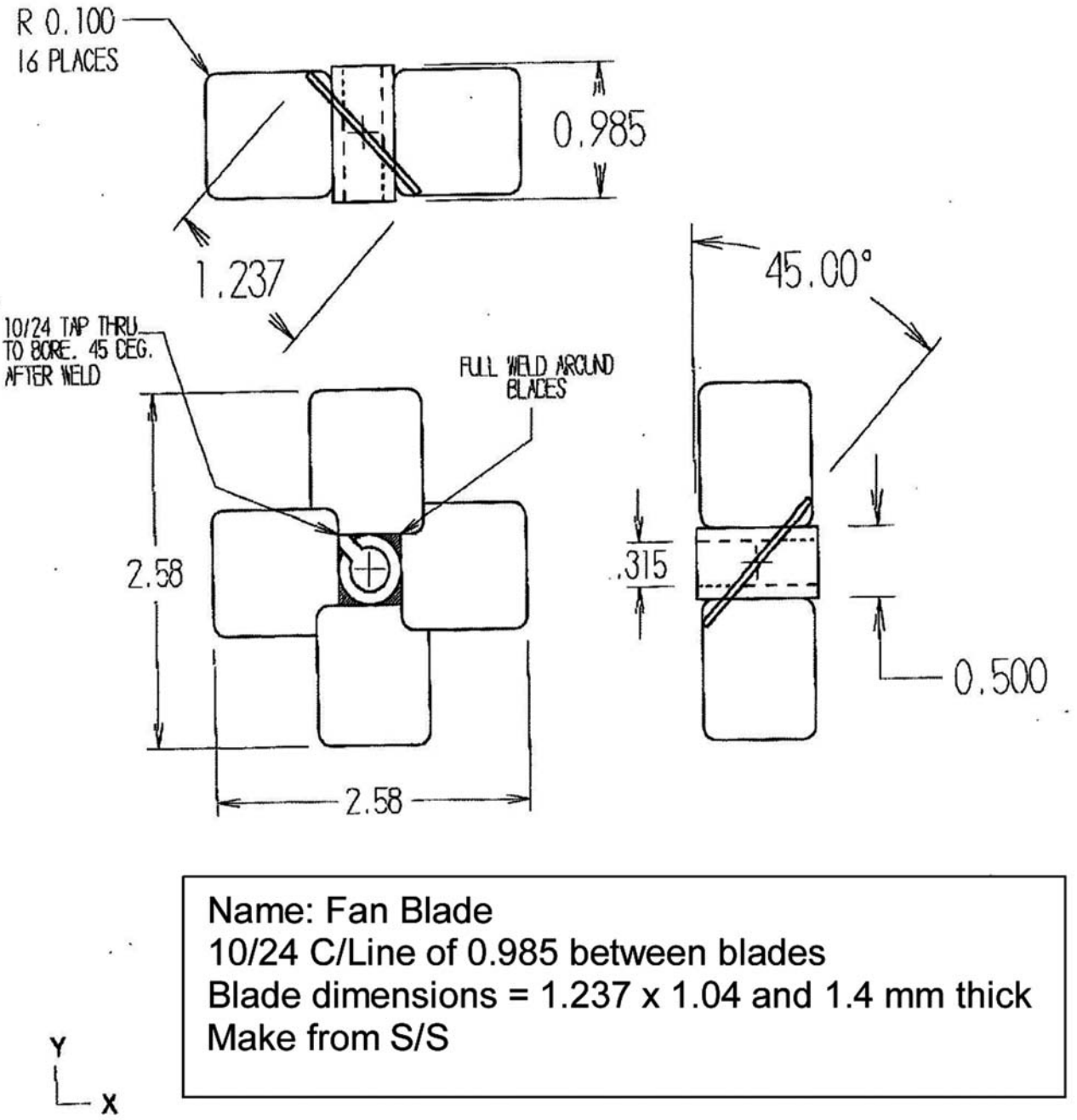

FIG. A4.1 Specification for Agitator Turbine Blades (all dimensions are in inches unless otherwise stated)

A5. AGITATOR PACKING GLAND

A5.1 Fig. A5.1 shows details and dimensions of the agitator packing gland. Reimel Machine, Inc. part number RMI-1004-DH[7,9] has been found suitable for this purpose.

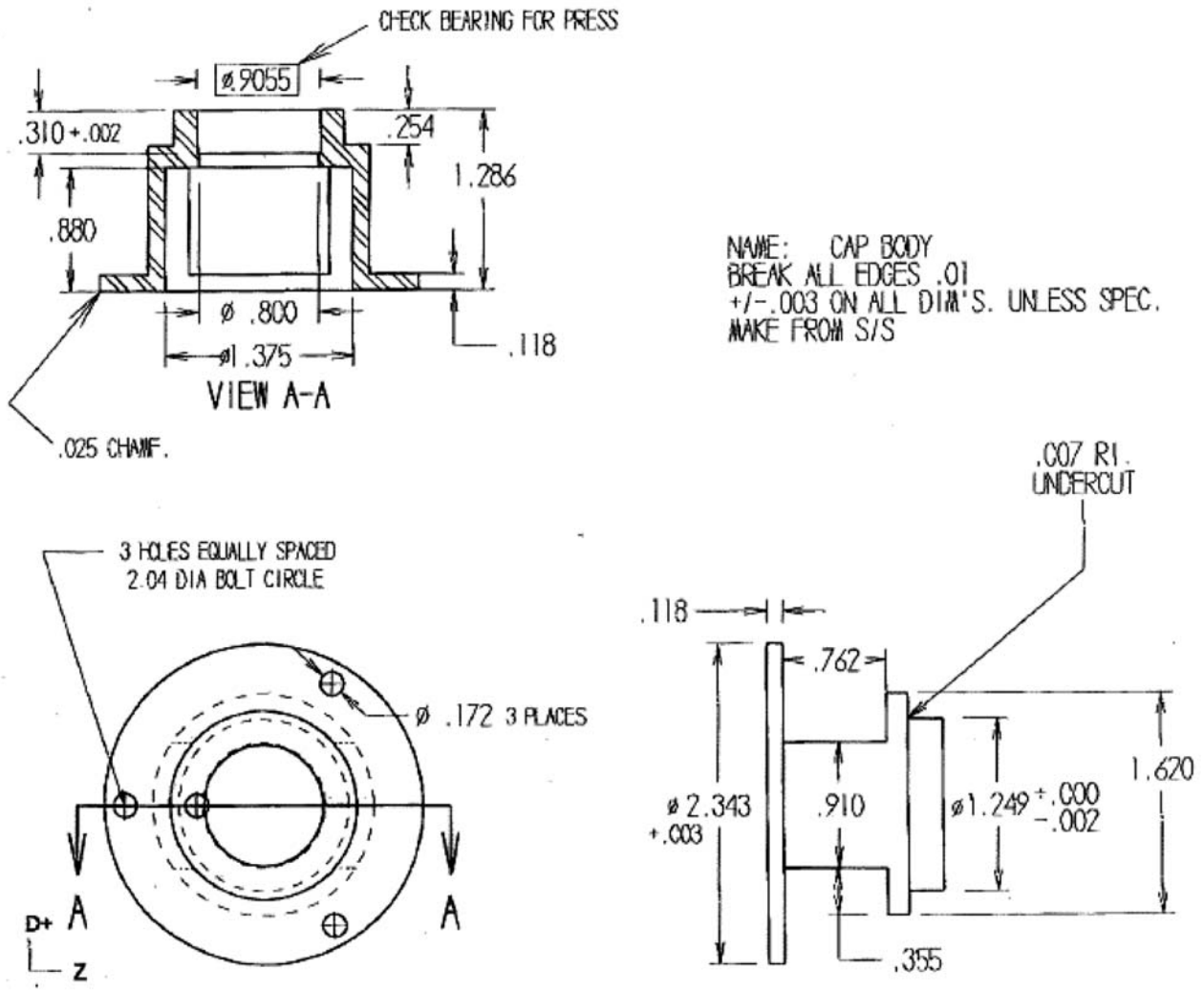

FIG. A5.1 Agitator Packing Gland (all dimensions are in inches)

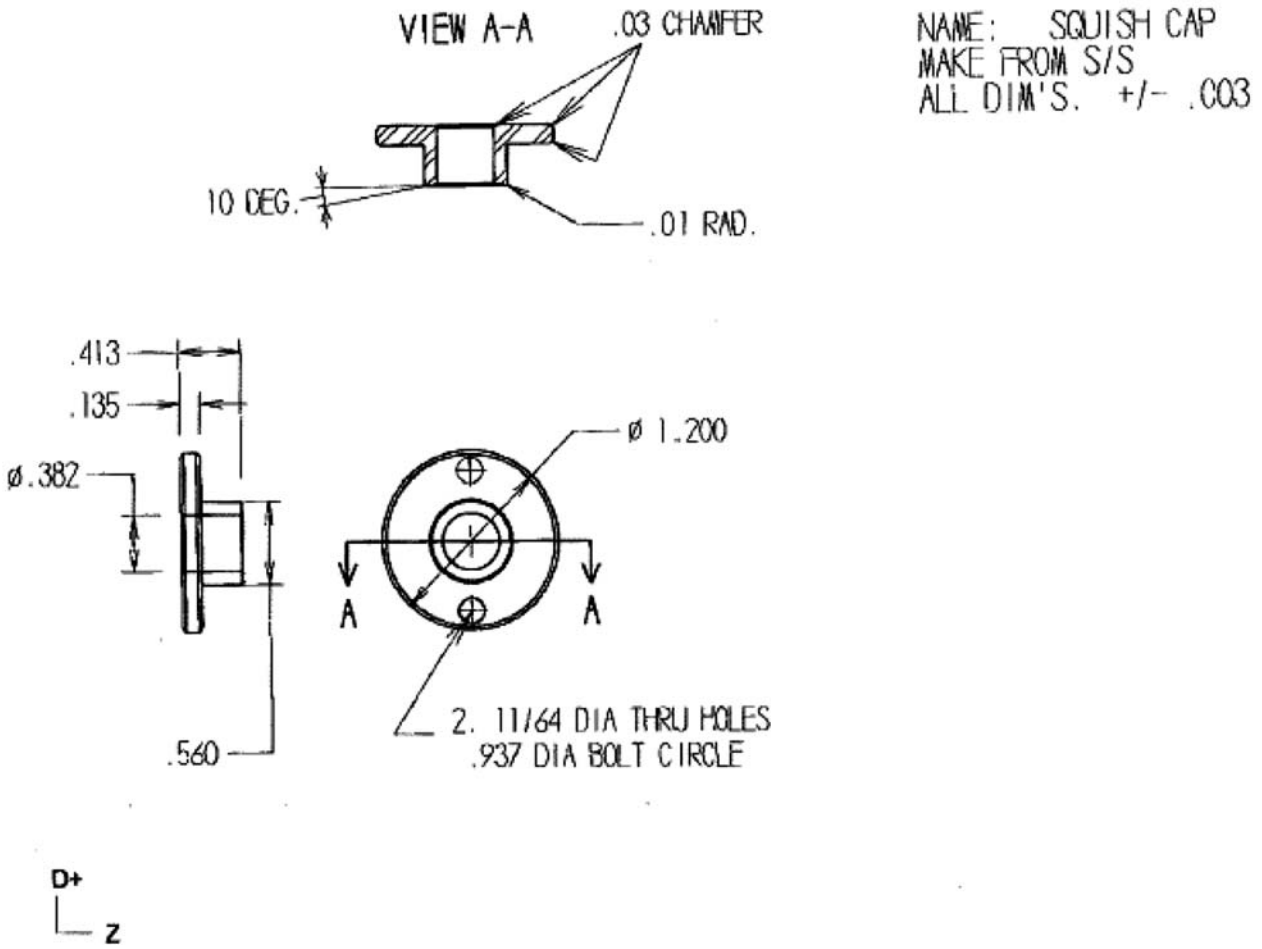

FIG. A5.1 Agitator Packing Gland (all dimensions are in inches) *(continued)*

A6. NITROGEN DIOXIDE GRADUATED TUBE

A6.1 A diagram of the Ace Glass, Inc. 12-mL, graduated centrifuge tube, part number D120677[6,7] is shown in Fig. A6.1.

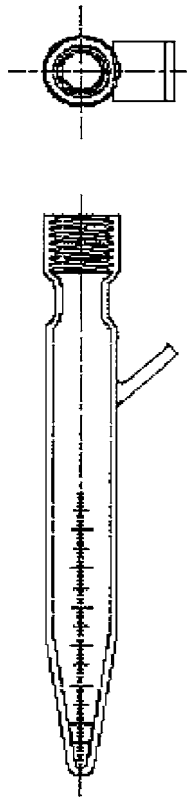

FIG. A6.1 Nitrogen Dioxide Graduated Tube

A7. VACUUM SYSTEM PLUMBING

A7.1 *General*—It is critical to follow these instructions precisely when constructing the vacuum plumbing for the reaction vessel. This is because restrictions in the vacuum system may act as a partially closed valve. As a consequence, the vacuum control valve, which is used to *tune* the system, cannot be opened sufficiently to compensate and the tuning range may be inadequate to allow calibration with all three reference oils in section 9.1.

A7.2 Construct vacuum lines (flexible or rigid stainless steel) from the vacuum source to condenser from tubing with a minimum inside diameter of 0.375 in. If construction is rigid tubing, use machined fittings for bends (do not use bent tubing).

A7.3 Construct vacuum lines from the condenser to the top connection of the vacuum control valve at the reactor head from tubing with a minimum inside diameter of 0.375 in. and reduced to ¼ in. tubing with inside diameter of 0.18 in. to attach onto the vacuum control valve. If construction is rigid tubing, use machined fittings for bends (do not use bent tubing). Ensure that rigid vacuum lines from the reactor to the vacuum trap condenser slope downward to the condenser and that flexible vacuum lines from the reactor to the condenser do not have any dips or constrictions.

A7.4 Construct the vacuum lines from the bottom connection of the vacuum control valve to the reactor connection from ¼ in. tubing with inside diameter of 0.18 in. and no more than 25 mm long.

A8. VACUUM TRAP CONDENSERS

A8.1 *General*—A vacuum trap/condenser is used to protect the vacuum system from harmful effects of reaction gases and to collect volatilized oil.

A8.2 A dual trap, assembled from Ace Glass, Inc. part numbers 8748-12, 7506-15, and 8751-20,[6,7] has been found to provide sufficient system protection.

A8.3 An alternative vacuum trap/condenser for ½ in. tubing comprising an Ace Glass, Inc. condenser (part number D127507) and trap (part number D127590) has also been found to be satisfactory.[6,7] This alternative offers larger diameter connections to the vacuum system than that specified in A8.2.

A9. SETTING THE VACUUM CONTROL VALVE

A9.1 On a completely assembled reactor including the flow meter, apply vacuum to the system, open the vacuum control valve fully, and block the vacuum relief orifice long enough to assure system will attain 85 kPa with a subsurface airflow of 185 mL/min. The air flow meter shall read less than 0.6 SCFM.

A9.2 Adjust the vacuum control valve and the vacuum relief orifice to attain an airflow of 2.0 ± 0.1 SCFM through the reactor while the vacuum pressure is maintained at 61 ± 1.7 kPa.

A9.3 The system is now ready to begin reference oil calibration runs as described in section 9.1. Do not change the vacuum control valve setting during the calibration runs as it affects the severity of the test. If the system achieves "in-calibration status" in 9.1, use the same vacuum control valve setting for all subsequent test oil runs.

A9.4 Should the system not achieve calibration in 9.1, first check that the vacuum plumbing instructions in Annex A7 were followed precisely. If they were, make the following adjustments:

A9.4.1 If a reference oil exhibits a mild response, open the vacuum control valve, further. If the reference oil response is too severe, close the vacuum control valve further. Resetting the vacuum control valve is a matter of some trial and error; it is suggested that no more than one revolution of the handle be made at any one time. After resetting the vacuum control valve, adjust the vacuum relief orifice to attain an airflow of 2.0 ± 0.1 CFM through the reactor while the vacuum pressure is maintained at 61 ± 1.7 kPa.

A9.4.1.1 For systems using a laboratory vacuum pump, it may be necessary to install an air bleed to achieve the latter conditions.

APPENDIXES

(Nonmandatory Information)

X1. SAMPLE PREPARATION AND ADDITION

X1.1 *General*—There is no prescribed method of introducing the fresh oil and the sample with the catalyst into the reactor. Examples of techniques that have been successfully used are given below.

X1.1.1 *Premix Procedure*:

X1.1.1.1 Tare a clean, 300-mL beaker using the balance described in 6.1.1 and weigh 3.0 ± 0.1 g of prepared iron ferrocene catalyst into beaker.

X1.1.1.2 Add the test oil to the beaker until a total mass of 200.0 ± 1.0 g is attained.

X1.1.1.3 Stir catalyst/test oil mixture with a glass rod for 1 min.

X1.1.1.4 Transfer the mixture into the reaction vessel through the fill port.

X1.1.1.5 Seal the fill port with a threaded plug.

X1.1.2 *Direct Weighing Procedure*:

X1.1.2.1 Remove the reactor vessel from the apparatus, clean, if necessary, as described in 11.1 and tare using the balance described in 6.1.1.

X1.1.2.2 Weigh 3.0 ± 0.1 g of prepared iron ferrocene catalyst into the vessel.

X1.1.2.3 Add test oil until a total mass of 200.0 ± 1.0 g is achieved.

X1.1.2.4 Reassemble the vessel to the apparatus.

X1.1.2.5 Carry out the vessel seal check as described in 10.3.

X1.1.2.6 Preset the vacuum flow and pressure as described in 10.4.

X2. CHARGING THE LIQUID NITROGEN DIOXIDE

X2.1 *General*—There is no prescribed method of charging the liquid nitrogen dioxide. An example of a technique that has been successfully used is given below.

X2.1.1 *Using a 3-Way or a 4-Way Valve*:

X2.1.1.1 To reduce pressure inside the graduated tube, either place an ice/water mixture at the bottom of the tube or a piece of dry ice at the top of the tube.

NOTE X2.1—Nitrogen dioxide boils at 21.1 °C.

X2.1.1.2 Close all valves into the apparatus.

X2.1.1.3 Turn the selector valve so that it is pointed down towards the graduated tube in the case of a 3-way valve and towards the reaction vessel in the case of a 4-way valve.

X2.1.1.4 Open the nitrogen dioxide gas bottle valve for several seconds to allow some of the liquid phase to collect in the connecting tube above the valve.

X2.1.1.5 Securely close the nitrogen dioxide gas bottle valve and slowly open the nitrogen dioxide charging valve to allow some liquid to drip into the nitrogen dioxide graduated tube. (**Warning—**Because of the toxicity of nitrogen dioxide, exercise care when opening the charging valve.)

X2.1.1.6 Repeat the sequence of chilling, and valve opening and closing, until 2.0 mL of nitrogen dioxide is present in the tube.

X2.1.1.7 Alternatively, the application of vacuum can also be used to charge the graduated tube.

NOTE X2.2—If using a vacuum to remove excess nitrogen dioxide due to an overcharge, exercise great care to avoid sucking out the entire charge.

X3. NITROGEN DIOXIDE PRECISION NEEDLE VALVE

X3.1 A needle valve positioned between the vessel and nitrogen dioxide graduated tube has been successfully used to control the flow of nitrogen dioxide gas into the inlet flow stream. Because of the small amount of gas required per hour, it is recommended that the valve have a low Cv for controlling the flow in a precise manner. Examples of valves that have been found satisfactory are a 10-turn, vernier-handle, needle valve with a Cv of 0.004 and a 20-turn, needle valve with a Cv of 0.019.

X4. EXAMPLE OF AN ASSEMBLED ROBO APPARATUS

X4.1 Fig. X4.1 shows an example of an assembled ROBO apparatus test stand. As pointed out in 6.19, however, there is no standard ROBO apparatus assembly. As an aid to building and setting up a new ROBO apparatus, a package of information is available on the TMC website.[2]

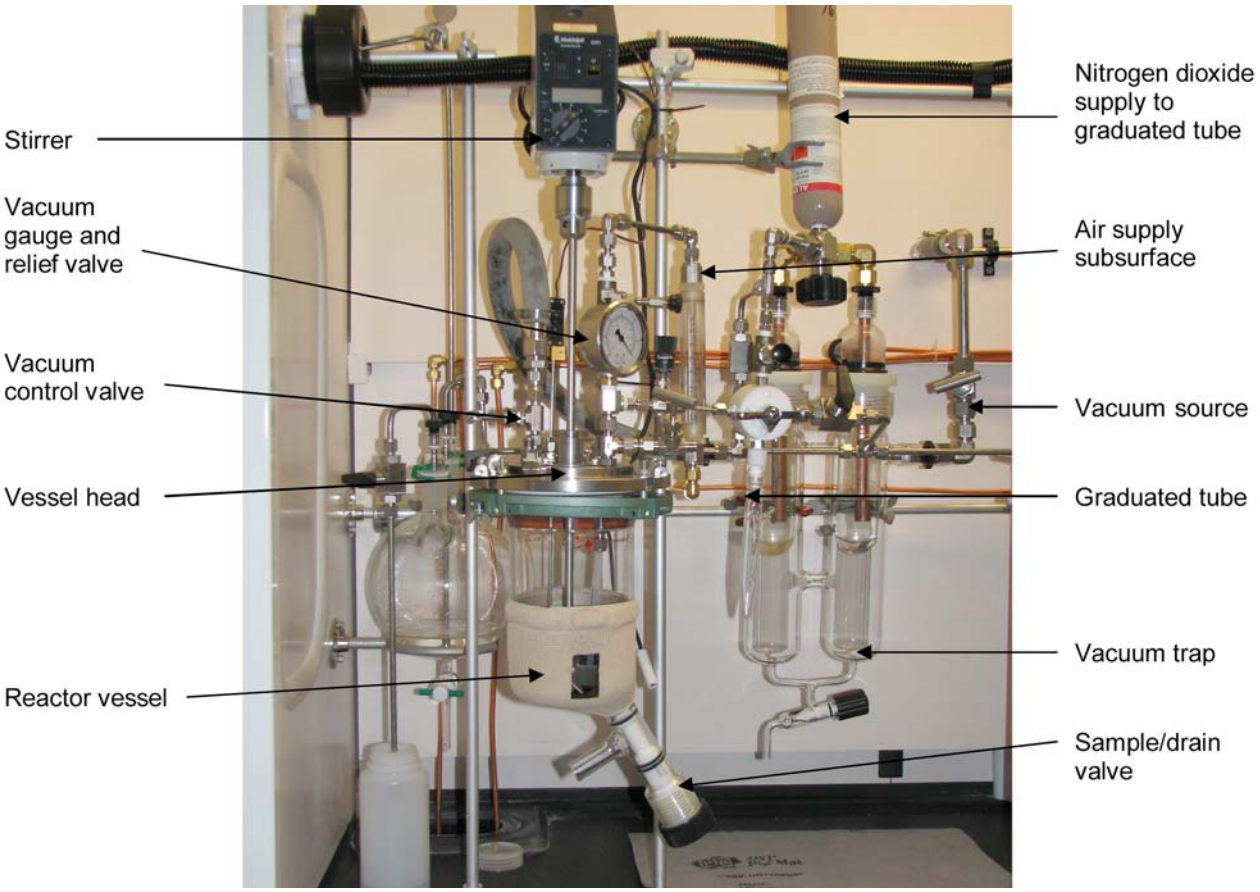

FIG. X4.1 Example of a Fully-Assembled ROBO Apparatus Test Stand

X5. INFORMATION PACKAGE TO AID SETTING UP A NEW ROBO APPARATUS

X5.1 Although the information provided in ROBO Test Method D7528 is sufficient to allow a new user to build a ROBO apparatus, this package provides (non-mandatory) information, supplementary to that in Section 6 of the Method, on components and techniques that have been found suitable when setting up an apparatus for the first time. Some of the general laboratory issues associated with handling nitrogen dioxide are also discussed as are emergency shut down procedures.

X5.1.1 The information package was provided to the laboratories that built the ROBO apparatus used in the precision round robin. Section 6 of this Method, however, incorporates improvements and clarifications of the method and takes precedent over the Information Package in the event there are discrepancies.

X5.2 The following is an index to the contents of the Information Package on TMC's website:[2]

Subject	Applicable Section of this Test Method
Background	
Agitation System	6.6
General	
Installation of the Packing Gland	
Agitation System Components	
Air Supply System	6.7
General	
Air Supply and Subsurface Feed	
Schematic of Air Supply System	
Air Supply System Components	
Miscellaneous	
General Precautions for Operating Equipment	
Emergency Shutdown Procedure	
Emergency Shutdown Procedure in the	
Event of a Nitrogen Dioxide Release in	
the Laboratory	
Nitrogen Dioxide	6.15, 10.5.3, 10.6.6
General	
Nitrogen Dioxide Physical Characteristics	
Charging the Nitrogen Dioxide to the	
Reaction Vessel	
Schematic	
Adjusting the Level of Nitrogen Dioxide from	
the Graduated Tube	

Subject	Applicable Section of this Test Method
Schematic	
Nitrogen Dioxide System Components	
Temperature Control System	6.9
General	
Initial Setup Parameters	
Temperature Control System	
J-Thermocouple placement inside reactor	
Temperature Control System Components	
Vacuum System	6.10, 6.11
General	
Vacuum Trap Components	
Vacuum Trap	
Water Safety	
Vacuum Pressure Measurement	
Water Flow Diagram	
Vacuum Flow Diagram	
Vacuum System Check	
Vacuum System Components	
Vessel Head and Vessel Components	6.4
General	
Diagram and Photographs of Vessel Head	
More photographs	
Vessel and Vessel Head Components	