



# Standard Test Method for Evaluation of Rust Preventive Characteristics of Automotive Engine Oils<sup>1</sup>

This standard is issued under the fixed designation D 6557; 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 a Ball Rust Test (BRT) procedure for evaluating the antirust ability of fluid lubricants. The procedure is particularly suitable for the evaluation of automotive engine oils under low-temperature, acidic service conditions.

1.2 Information Letters are published occasionally by the ASTM Test Monitoring Center (TMC)<sup>2</sup> to update this test method. Copies of these letters can be obtained by writing the center.

1.3 The values stated in either SI units or in other units shall be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, use each system independently of the other, without combining values in any way.

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.* See 7.1.1-7.1.3, 8.2.1.1, and Table 1.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

D 5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID)

<sup>1</sup> 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 on Automotive Lubricants.

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<sup>2</sup> Until the next revision of this test method, the ASTM Test Monitoring Center will update changes in the test method by means of information letters. Information letters may be obtained from the ASTM Test Monitoring Center, 6555 Penn Avenue, Pittsburgh, PA 15206-4489, Attention: Administrator. This edition incorporates revisions in all information letters through No. 03-1. The TMC supplies reference oils.

<sup>3</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.

E 344 Terminology Relating to Thermometry and Hydrometry

## 3. Terminology

3.1 *Definitions:*

3.1.1 *calibrate, v*—to determine the indication or output of a measuring device with respect to that of a standard. **E 344**

3.1.2 *corrosion, n*—the chemical or electrochemical reaction between a material, usually a metal surface, and its environment that can produce a deterioration of the material and its properties. **D 5844**

3.1.3 *non-reference oil, n*—any oil other than a reference oil, such as a research formulation, commercial oil, or candidate oil. **D 5844**

3.1.4 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison. **D 5844**

3.1.4.1 *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.

3.1.5 *rust, n*—of ferrous alloys, a corrosion product consisting primarily of hydrated iron oxides. **D 5844**

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *average gray value (AGV), n*—measurement of brightness units on test specimens, indicating the degree of rust protection.

3.2.2 *specimen, n*—a carbon steel ball,  $\frac{7}{32}$  in. (AISI 1040).

## 4. Summary of Test Method

4.1 Multiple test tubes, each containing test oil and a specimen, are placed in a test tube rack, which is attached to a mechanical shaker. The shaker speed and temperature are controlled.

4.2 Air and an acidic solution are continuously fed into each test tube over an 18 h period to create a corrosive environment.

4.3 The specimens are then removed, rinsed, and analyzed by an optical imaging system designed to quantify the antirust capability of each test oil.

TABLE 1 Organic Solvent

	Composition
Ethyl acetate, <sup>A</sup> 99.5+ %	(37.5 % vol)
Denatured ethyl alcohol <sup>B</sup>	(27.5 % vol)
Butanol, <sup>C</sup> 99 %	(5.0 % vol)
Tetrahydrofuran (THF), <sup>C</sup> 99+ %	(30.0 % vol)

<sup>A</sup> **Warning**—See the appropriate Materials Safety Data Sheet.

<sup>B</sup> **Warning**—Flammable. Cannot be made nontoxic. Health hazard.

<sup>C</sup> **Warning**—Flammable. Health hazard.

## 5. Significance and Use

5.1 This bench test method was designed as a replacement for Test Method D 5844. Test Method D 5844 was designed to measure the ability of an engine oil to protect valve train components against rusting or corrosion under low temperature, short-trip service, and was correlated with vehicles in that type of service prior to 1978.<sup>4</sup>

5.1.1 Correlation between these two test methods has been demonstrated for most, but not all, of the test oils evaluated.

## 6. Apparatus

6.1 *Specimen Preparation System*—Obtain the specimens from the Central Parts Distributor (CPD).<sup>5,6</sup>

6.1.1 Specimen preparation equipment includes various common laboratory apparatus and an ultrasonic cleaning bath.

6.2 *Air Supply System*—A compressed air supply is required, with two air filters, two pressure regulators, a gas purifier, a gassing manifold (25 port outlet), TFE-fluorocarbon tubing (25 600-ft lengths) or equivalent multiport flow control system, and a gas mass flowmeter (see Annex A1 and Figs. 1 and 2).

6.3 *Acid Solution Delivery System*—An acid solution delivery system that includes a multiple syringe pump with a ten position rack is required. The flow rate range minimum is 0.0001  $\mu\text{L/h}$  (using a 0.5- $\mu\text{L}$  syringe) to a maximum 220.82 mL/min (using a 140-mL syringe) (see Figs. 3 and 4).

6.4 *Test Tube Assembly*—The test tube assembly consists of 24 disposable plastic syringes and other common laboratory apparatus.

6.5 *Temperature and Shaking Speed Control System*—A mechanical shaker, Bench-Top Environ Shaker Model 4628,<sup>6,7</sup> provides an orbital shaking motion in a controlled speed and temperature environment.

6.5.1 A special test tube assembly rack<sup>6,8</sup> (see Figs. 5 and 6) has 24 tube positions and is attached to the shaker platform (18 in. by 18 in.).

6.6 *Gassing Manifold*, required.

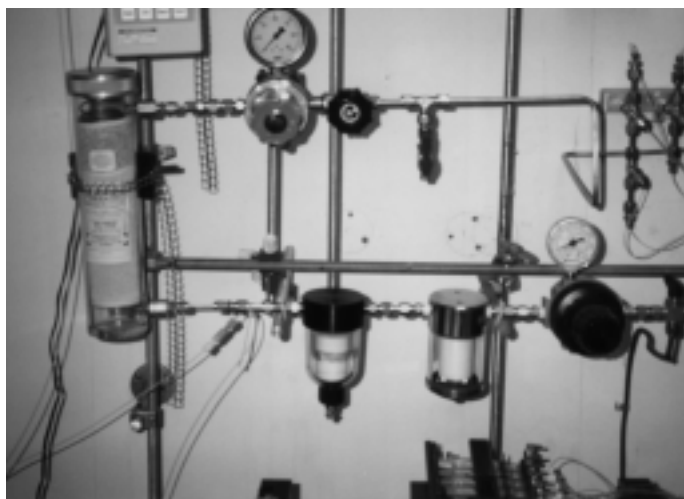


FIG. 1 Photograph of Air Delivery System

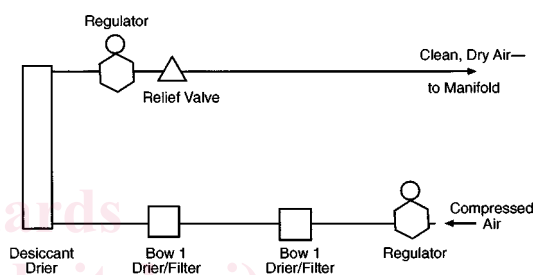


FIG. 2 Schematic of Air Delivery System



FIG. 3 Photograph of Acid Delivery System

6.7 *Venting System*—Common laboratory apparatus is employed for the required venting system (see Fig. 7).

6.8 *Image Analysis System*—A specific imaging analysis system<sup>6,9</sup> is required. This system is composed of:

6.8.1 *Optics and Illumination:*

6.8.1.1 Nikon Epiphot 200 inverted metallurgical microscope,

<sup>4</sup> Special Technical Publication, "Multicylinder Test Sequences for Evaluating Automotive Engine Oils, Part, Sequence IIID *ASTM STP 315H*, Available from ASTM Headquarters.

<sup>5</sup> The sole source of supply of the apparatus known to the committee at this time is Central Parts Distributor, Test Engineering Inc., 12718 Cimmaron Path, San Antonio, TX 78249.

<sup>6</sup> If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

<sup>7</sup> The sole source of supply of the apparatus known to the committee at this time is Labine, Inc., 15<sup>th</sup> and Bloomingdale, Melrose Park, IL 60160.

<sup>8</sup> The sole source of supply of the apparatus known to the committee at this time is West End Machine and Weld, Inc., P.O. Box 9444, Richmond, VA 23228.

<sup>9</sup> The sole source of supply of the apparatus known to the committee at this time is Meyer Instruments, Inc., 1304 Langham Creek, Suite 235, Houston, TX 77084.

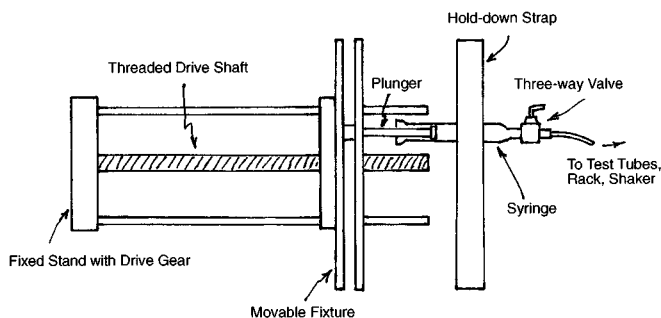


FIG. 4 Schematic of Acid Delivery System

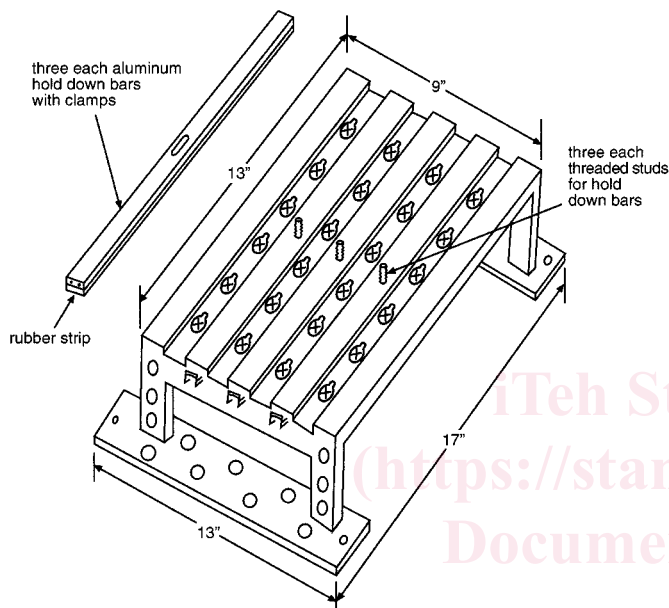


FIG. 5 Test Tube Assembly Rack

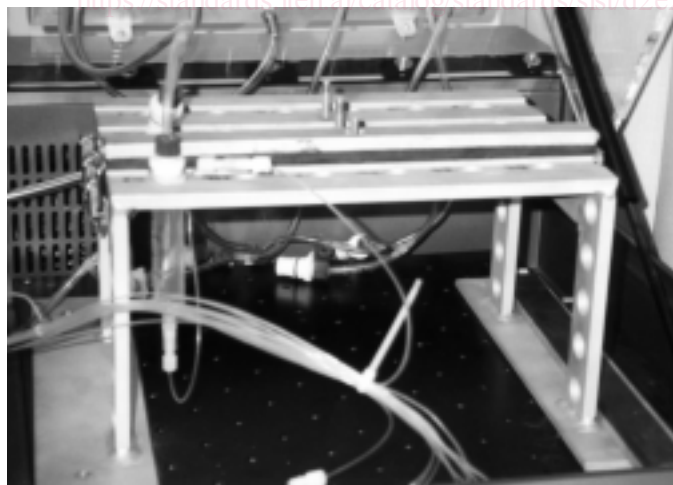


FIG. 6 Photograph of Test Tube Assembly Rack

- 6.8.1.2 BZ binocular head,
- 6.8.1.3 RV 3 plate mechanical stage,
- 6.8.1.4 CFWN 10× wide field eyepiece, high point eyepiece,
- 6.8.1.5 Manual BD 5 place nosepiece,
- 6.8.1.6 Epiphot 300 EB block,



FIG. 7 Gassing Manifold for Venting

- 6.8.1.7 DF module,
- 6.8.1.8 CF BD plan 5×/0.13 plan achromat objective,
- 6.8.1.9 CF BD plan 10×/0.13 plan achromat objective,
- 6.8.1.10 EPI polarizer,
- 6.8.1.11 Analyzer,
- 6.8.1.12 Lamphouse for 12 V/100 W quartz halogen light source,
- 6.8.1.13 Lamphouse adapter,
- 6.8.1.14 12 V/100 W halogen bulbs,
- 6.8.1.15 300/200 100 W power supply.
- 6.8.1.16 Remote control cable,
- 6.8.1.17 C-mount coupler for video camera,
- 6.8.1.18 NCB 11 filter,
- 6.8.1.19 Power cords, and
- 6.8.1.20 Ultracentrifuge tube spacer with a 5-mm hole drilled in the center (used as a sample holder and sample randomizer for sample orientation).

6.8.2 Image Capture Hardware and Software:

- 6.8.2.1 Research grade, high resolution, NTSC RGB/RS-170 camera system,
- 6.8.2.2 Research grade, high resolution, NTSC RGB/RS-170 frame grabber,
- 6.8.2.3 The host computer system (shall meet or exceed the following specifications):

(1) *Hardware*—Pentium 133 MHz CPU, 16 MB RAM, 540 MB hard drive, 1.44 MB 3.5-in. floppy, 1.44 MB 5.25-in. floppy (optional), CD-ROM (highly recommended option), 101 or Windows 95 keyboard, SVGA local bus video card with 2 MB RAM (4 MB recommended), 2 button serial mouse with pad, 2 parallel ports, and 2 serial ports.

(2) *Software*—Windows 3.x/DOS 6.22 or Windows 95 Operating System, Microsoft Excel 7.0 (Microsoft Office 97 recommended).

(3) *Monitor*—Medical grade high-resolution 19-in. NTSC RGB color video monitor, all necessary cables, connectors, and adapters (including a surge and spike suppressing power strip).

6.8.3 BRT Image Analysis Software:

- 6.8.3.1 BRT macro program, and
- 6.8.3.2 Optimate image analysis engine.

## 7. Reagents and Materials

### 7.1 Reagents:

7.1.1 *Acid Solution* (**Warning**—Corrosive. Combustible. Health hazard)—Obtain the acid solution from the CPD.

NOTE 1—For information only. Appendix X1 contains details of the acid solution.

7.1.2 *Acetone, 99.5 %*. (**Warning**—Flammable. Health hazard.)

7.1.3 *n-Heptane, 38 to 42 %* (purity), commercial grade, with C<sub>7</sub> isomers. (**Warning**—Flammable. Health hazard.)

7.1.4 *Organic Solvent*—Blend as shown in Table 1.

### 7.2 Materials:

7.2.1 *TFE-fluorocarbon FEP Tubing*, 1/32-in. I.D. by 1/16-in. outside diameter (O.D.); 17 rolls of 1000 ft/roll. (Not required if the optional NRS flow controller<sup>6,10</sup> is used.)

7.2.2 *Vinyl Tubing*, 1/8 in.-inside diameter (I.D.) by 1/4 in.-O.D.; about 15 ft.

7.2.3 *Miscellaneous Common Laboratory Equipment*, including glassware, tubing fittings, trays, vials, and plastic syringes.

## 8. Preparation of Apparatus

### 8.1 Specimens:

8.1.1 Remove the appropriate number of specimens from vacuum-sealed packages, into a 4-oz bottle (clear, medium-round with cap). Add sufficient heptane (see 7.1.3), approximately 2 oz, to cover specimens.

8.1.2 Cap the bottle loosely and place it in an ultrasonic cleaning bath. Sonicate for 30 min, and then decant the heptane.

8.1.3 Rinse two more times with heptane and follow with an acetone (see 7.1.2) rinse to ensure the specimens are free of contamination. Dry the specimens with nitrogen for 3 min.

NOTE 2—The specimens can be prepared up to one week in advance and stored in heptane until needed for testing.

### 8.2 Test Tube Assembly, Tube Rack, and Shaker for Each Test Tube:

8.2.1 Cut 24 separate pieces of TFE-fluorocarbon FEP tubing, each piece to be 9.5-in. (24-cm) long.

8.2.1.1 Use compressed air (for technical use only), 50 psig minimum, to remove most of the water/oil emulsion that may be trapped inside the short lengths of capillary tubing. Clean the tubing with heptane (see 7.1.3), followed by acetone (see 7.1.2), and dry with compressed air.

8.2.2 Check the flangeless fitting for 1/16-in. O.D. TFE-fluorocarbon tubing for deterioration, and replace as necessary.

8.2.3 Remove and discard the plunger from a new 20-mL disposable plastic syringe (Luer-Lok), and securely fasten the syringe barrel to the short capillary tubing, using couplings, 1/4-28 thread, and female Luer CTFE fittings, 1/4-28 thread, and with a 1.5-mm bore.

8.2.3.1 Label the syringes (test tubes) from 1 to 24.

8.2.4 Place the assembled test tube in the tube rack with the capillary tubing facing upward in the adjacent small hole.

8.2.4.1 The test tube assembly rack is a specially designed aluminum fabrication. It holds 24 test tubes with easy snap-on lock, wing nuts, and hold-down bars (see Fig. 5).

8.2.5 Place one precleaned specimen into each test tube, using extra-long forceps (7 in. with serrated tips) to avoid contamination.

8.2.6 Insert 10 mL of test oil into each test tube, using a 10-mL disposable syringe.

8.2.7 Secure the test tubes to the tube holder with three hold-down bars and three wing nuts.

8.2.8 Fasten the test tube assembly rack to the shaker platform with four custom-made wing bolts.

### 8.3 Acid Delivery System:

8.3.1 Withdraw 6 mL of acid solution by hand from a wide-mouth beaker into an individual 5-mL disposable plastic syringe (Luer-Lok).

8.3.1.1 Attach the syringe to an acid inlet port of one of the 24 three-way switching valves, with 1/4-28 thread. (The other two ports are used for air inlet and mixed air/acid outlet.)

8.3.2 Turn the three-way valve to *two-way open* and eject, by hand, about 0.5 mL of acid solution into a waste beaker, while ensuring that no air bubbles remain in the syringe.

8.3.2.1 Place the syringe that now contains about 5.5 mL of acid solution on the holder of the multiple syringes pump.

8.3.3 Repeat the above procedure for the other 23 acid delivery syringes.

8.3.4 There are three multiple syringe pumps, and eight of the 5-mL syringes are attached to each of the pumps.

8.3.4.1 The pumps each have a ten-position rack and are required to satisfy a flow rate range of 0.0001  $\mu$ L/h, minimum, to 220.82 mL/min, maximum. Required accuracy is  $\pm 1\%$ , and reproducibility is  $\pm 0.1\%$ .

8.3.5 Ensure that the syringe barrel flange and the plunger flange are firmly held by the retaining clamps (six 2-in. C-clamps that secure the ends of the hold-down bars of the multiple syringes pump).

8.3.5.1 Good alignment of all 24 acid delivery syringes against the retaining brackets is crucial to ensure repeatability. (See Fig. 3 for a photograph of the acid delivery system, and Fig. 4 for a schematic.)

8.3.6 Cut 24 pieces of TFE-fluorocarbon tubing; each piece to be  $51 \pm 1$  in. in length.

8.3.6.1 Attach each of these tubes to the mixed air/acid outlet ports of the three-way switching valves.

### 8.4 Air Delivery System:

8.4.1 Clean, dry air, compressed to at least 50 psig, is required.

8.4.2 A single stage, high-purity stainless steel pressure regulator is the first in the line; this is equipped with a 0 to 160-psi maximum pressure gage.

8.4.3 Two compressed air filters capable of removing particles and mists are required, and are next in the line.

8.4.3.1 The first filter in the line is an A912-DX type, followed by an A912-BX type. These have polycarbonate

<sup>10</sup> Brooks Model 8744 NRS Flow Controller has been determined to be acceptable for this application. The sole source of supply of the apparatus known to the committee at this time is McPac Process Automation and Control, 8040 Bavaria Rd., Twinsburg, OH 44087.

bowls and should be equipped with aluminum shields. They have ¼-in. NPT (F) ports and will withstand 150-psig pressure maximum.

NOTE 3—Alternatively, zero grade air cylinders can be used and will not require the extensive filtering outlined above.

8.4.4 A Drierite gas purifier, with a maximum working pressure of 100 psig, is next in the line.

8.4.4.1 The first portion of the purifier (about 75 %) contains a molecular sieve, activated, Type 4A, 8 to 12 mesh.

8.4.4.2 The remaining portion of the purifier (about 25 %) contains Drierite absorbent, color indicating type, 8 mesh.

8.4.5 Next in the line is the downstream regulator, single stage high-purity stainless steel, which is equipped with a 0 to 60-psi pressure gage.

8.4.6 The next installation is a relief valve, in-line adjustable CA series, 50 to 150-psi cracking pressure range, set at 80 psi (optional to control over pressure).

8.4.7 Lastly, a gassing manifold with 25 port outlets and ¼-in. tube fittings is installed. (See Figs. 5 and 6 for a schematic and photograph of the air system.)

8.4.8 Cut 25 pieces of the TFE-fluorocarbon tubing, each piece to be approximately 600 ft in length.

8.4.8.1 These long tubes provide the necessary backpressure to allow good control of the very low airflow rate. The individual lengths may need to be adjusted slightly to ensure the same flow rates at a given delivery pressure. An equivalent multiport flow control system can be used.<sup>6,10</sup>

8.4.8.2 Connect these tubes to the gassing manifold.

(1) One of these tubes is then connected to a gas mass flowmeter, capable of measuring up to 200 mL/min, and with 0.1-mL/min resolution (see Annex A1).

(2) The other 24 tubes are then connected to the air inlet ports on the 24 three-way valves.

## 9. Procedure

9.1 Turn the three-way valve to *two-way open*, activate the syringe pumps, and eject 1 mL of acid solution into a waste beaker at 0.1-mL/min speed.

9.1.1 Make sure that the retaining brackets properly align all 24 syringe plungers.

9.2 Adjust the acid solution flow rate to 0.193 mL/h (test flow rate), and run for 1 h to ensure that all syringe plungers are properly aligned at the retaining brackets of the pusher block.

9.3 Set the actual air flow rate to 40 mL/min.

9.3.1 Monitor the airflow rate with the digital mass flow meter connected to the reference air capillary tubing. Refer to the calibration chart developed in Annex A1 to determine the actual air flow rate setting.

9.4 Turn the three-way valves to the *three-way open* position, and dry the nominal 51-in. lengths of capillary tubing with air for 30 min.

9.5 Measure and record the airflow rates of all lines, and then shut off the main airflow valve.

9.6 Connect the long capillary tubing (that is, the nominal 51-in. lengths) to the short capillary tubing from the test tubes.

9.7 Connect all 24 stopper vent lines to the top of the test tubes.

9.7.1 TFE-fluorocarbon thermometer adapters are used as stoppers, <sup>19</sup>/<sub>22</sub> joint.

9.7.2 Attach the vent lines to barbed tee connectors (clear polypropylene, for ⅛-in. I.D. tubing).

9.7.3 Then, connect the vent outlets to a gassing manifold (8 port inlet with ¼-in. hose barb; use vinyl tubing, ⅛-in. I.D. by ¼-in. O.D. (see Fig. 7).

9.7.4 Finally, connect the gassing manifold's outlet to a condensate trap flask, with side arm (1000 mL), placed such that gravity drains the condensate from the test tubes. *Make sure that all of the capillary tubes are free to move with the shaker platform.*

9.8 Set the shaker temperature to maintain  $48 \pm 0.1^\circ\text{C}$ , as measured in an actual oil sample, and warm up the entire system to the control temperature in the oil sample. Refer to the calibration chart developed in Annex A2 to determine the shaker temperature setting.

9.9 Following the warm-up period, turn on the shaker, set the shaker speed to 300 r/min, and ensure that each ball freely rotates against each syringe wall.

9.10 Start introducing the acid solution at an actual flow rate of 0.193 mL/h. Refer to the calibration chart developed in Annex A3 to determine the actual flow rate setting.

9.11 Check and adjust, if necessary, the upstream pressure (that is, upstream from the three-way valves) to ensure that the actual air flow rate is 40 mL/min.

9.12 Maintain the above test conditions for 18 h.

9.13 At the end of the test, stop the syringe pumps, shut off the airflow, and turn off the shaker.

9.14 Disconnect the acid/air delivery tubes from the test tube assembly, and remove the stoppers from the test tubes.

NOTE 4—The preceding procedure described the use of three multiple syringe pumps and 24 test tubes. However, as a minimum, one multiple syringe pump with a maximum of 10 test tubes can also be utilized.

## 10. Reference Oil Testing

10.1 Procure a supply of reference oils directly from the TMC.

10.1.1 These oils have been formulated or selected to represent specific chemistry types or performance levels, or both. Each reference oil is identified with a unique identification code on the container label.

10.2 Request a reference oil assignment from the TMC. Assignments will be made by the unique identifying codes on the reference oil samples.

10.2.1 Provide the TMC with the bath identification for the test.

10.3 Test the assigned reference oil along with each batch of non-reference oil tests, simultaneously with and in the same bath as the non-reference oils.

10.3.1 Run the reference oil test in accordance with the same procedure used for the non-reference oil tests.

10.3.2 Inclusion of this coded (that is, blind) reference oil helps protect against the possibility of bias in the testing.

NOTE 5—Annex A4 discusses the involvement of the TMC with respect to the reference oil-monitoring program.

10.4 The testing laboratory tacitly agrees to use the reference oils in accordance with Policies for Use and Analysis of

ASTM Reference Oils,<sup>2</sup> and to run and report the reference oil test results in accordance with TMC guidelines.

10.5 Report the reference oil test results to the TMC in accordance with the following guidelines:

10.5.1 Use the data reporting formats detailed in Annex A5 (see Figs. A5.1 through A5.4) for reporting all reference oil test results to the TMC.

10.5.2 Do not include any non-reference oil test results.

10.5.3 Complete all of the required blank fields on the forms.

10.5.4 Transmit reference oil test data by electronic means or by telephone facsimile to the TMC immediately upon completion of the test analysis.

10.5.4.1 Include all of the reporting forms in the transmission.

NOTE 6—Specific protocols for the electronic transmission of test data are available from the TMC.

10.5.5 In addition to the previously transmitted data, send by mail or other courier one copy of the final reference oil test report to the TMC.

10.5.5.1 The signatory line on the mailed Final Report Cover Sheet (see Fig. A5.1) requires an original signature by an authorized representative of the testing laboratory. The signature affirms the statements made in the affidavit on the Final Report Cover Sheet.

10.5.5.2 Mail the final test report so that it is received by the TMC within 30 days from the test completion date.

10.6 Upon receipt of the initial reference oil test results, the TMC will review the data for operational adherence to the published procedure.

10.6.1 If the test is determined to be operationally valid, the test results will then be evaluated, using statistical acceptance criteria established by the governing surveillance panel.

10.6.1.1 The acceptance criteria are subject to change at the discretion of the surveillance panel.

10.6.2 If the initial transmitted data is determined to be both operationally valid and statistically acceptable, the TMC will so notify the testing laboratory.

10.6.2.1 The TMC will also disclose the uncoded reference oil identification to the testing laboratory.

10.6.2.2 The TMC's first determinations are considered preliminary until the formally signed final report is received and reviewed by the TMC. Discrepancies between the initial transmitted data and the mailed final report may result in reversal of the preliminary determinations.

10.7 In the event that a reference test is determined unacceptable by the TMC, the TMC will provide an explanation to the testing laboratory.

10.7.1 If there is an obvious operational problem for the unacceptable test results, the problem has to be corrected before requesting another reference oil assignment from the TMC.

10.7.2 If the reason for the unacceptable results is not obvious, all test-related equipment shall be rechecked for compliance with the procedure and good laboratory practice.

10.7.3 Following this rechecking process, the TMC will assign another coded reference oil for testing.

10.8 The batch of non-reference oil tests, which accompany the coded reference oil test, is considered valid only if the results of the reference oil test meet the predetermined acceptance criteria for the particular reference oil tested.

## 11. Test Results

### 11.1 Prepare Specimens for Image Analysis:

11.1.1 Remove the specimens from the test tubes, using extra-long forceps (7 in. with serrated tips).

11.1.2 Swirl each specimen in a 400-mL beaker containing heptane (see 7.1.3) to remove most of the adsorbed oil layer.

11.1.3 Place each specimen into a separate 20-mL scintillation wash vial (make one perforation in the bottom to facilitate cleaning) in a vial holder.

11.1.4 Put the vial holder into a utility tray (stainless steel, 12¼ by 7¾ by 2¼ in.), pour enough heptane into the tray to cover the specimens, and shake the tray gently for 2 min before decanting the heptane.

11.1.5 Put enough organic solvent (see 7.1.4 and Table 1, Footnotes A through C) into the tray to cover the specimens, and soak the specimens for 10 min, to remove any remaining organic deposits, before decanting the solvent.

11.1.6 Put enough heptane into the tray to cover the specimens, and shake the tray gently for 2 min before decanting the heptane.

11.1.7 Transfer the specimens into clean, dry 20-mL scintillation vials that have been previously labeled the same as the test tubes in 8.2.3.1.

11.1.8 Dry the specimens with nitrogen gas, and then securely fasten the vial caps.

### 11.2 Prepare Image Analysis System for Rust Evaluation:

11.2.1 Set the microscope adjustments as follows:

11.2.1.1 Illumination change-over knob B/D—D (dark field).

11.2.1.2 ND16 filter slider—IN (push to first click).

11.2.1.3 A filter slider—IN (push to second click).

11.2.1.4 F (field diaphragm control lever)—OPEN.

11.2.1.5 A (aperture diaphragm control lever)—OPEN.

11.2.1.6 Neutral density filter—ND16.

11.2.1.7 Objective lens—5×.

### 11.3 Optronics Controller:

11.3.1 Shutter speed—1/60 + (manual).

11.3.2 White balance—ON.

11.4 Turn on the Image Analysis Computer and initiate the *Optimate* software program.

11.5 Select the macro titled *BRT51.MAC* (BRT Macro Version 5.1, or later).

### 11.6 Rust Rating Procedure:

11.6.1 Wipe each specimen, using a lint-free laboratory wipe; remove all solvent film and loose surface deposits from the rating surfaces prior to the digital image rating.

11.6.2 Place the Calibration Reference Specimen onto the microscope stage, and follow the dialog box instructions that appear on the screen (operator interface) (see Fig. 8).

NOTE 7—Refer to the software manual for the detailed procedure of imaging analysis.

11.6.2.1 Answer the operator interface questions to allow automatic recording of the data into the spreadsheet program.