Designation: D 6557 – 00

An American National Standard

Standard Test Method for Evaluation of Rust Preventive Characteristics of Automotive Engine Oils[1]

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 (ε) 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)[2] 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, 7.1.2, 7.1.3, 8.2.1.1, and Table 1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 5844 Test Method for Evaluation of Automotive Engine Oils for Inhibition of Rusting (Sequence IID)[3]

E 344 Terminology Relating to Thermometry and Hydrometry[4]

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, 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.

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.[5]

---

[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.B0.01 on Passenger Car Engine Oils.

Current edition approved June 10, 2000. Published August 2000.

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

[3] *Annual Book of ASTM Standards*, Vol 05.03.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Special Technical Publication, "Multicylinder Test Sequences for Evaluating Automotive Engine Oils, Part, Sequence IIID *ASTM STP 315H*, Available from ASTM Headquarters.

FIG. 1 Photograph of Air Delivery System

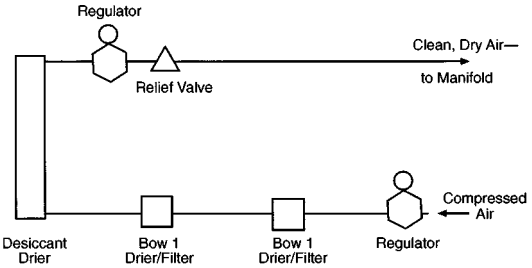

FIG. 2 Schematic of Air Delivery System

FIG. 3 Photograph of Acid Delivery System

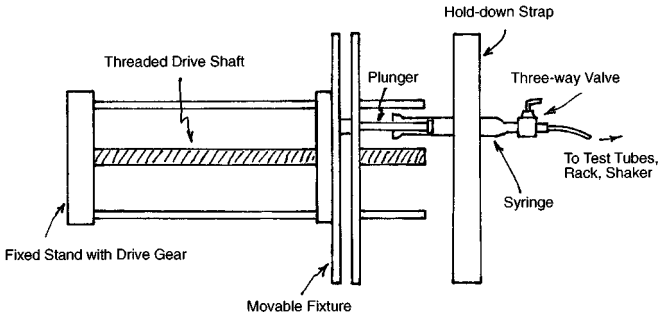

FIG. 4 Schematic of Acid Delivery System

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).[6]

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 µL/h (using a 0.5-µL 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,[7]

[6] 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. 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.

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

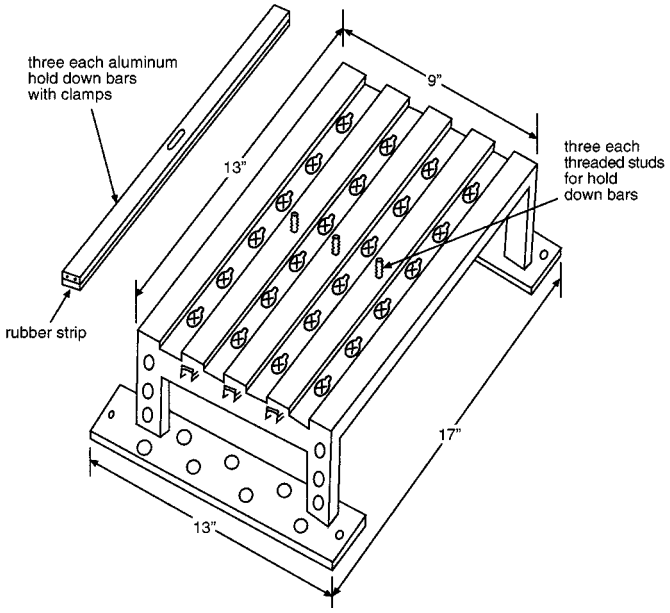

FIG. 5 Test Tube Assembly Rack

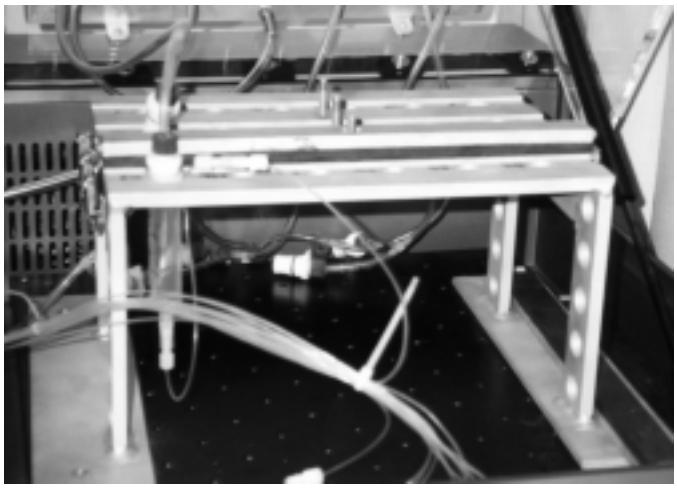

FIG. 6 Photograph of Test Tube Assembly Rack

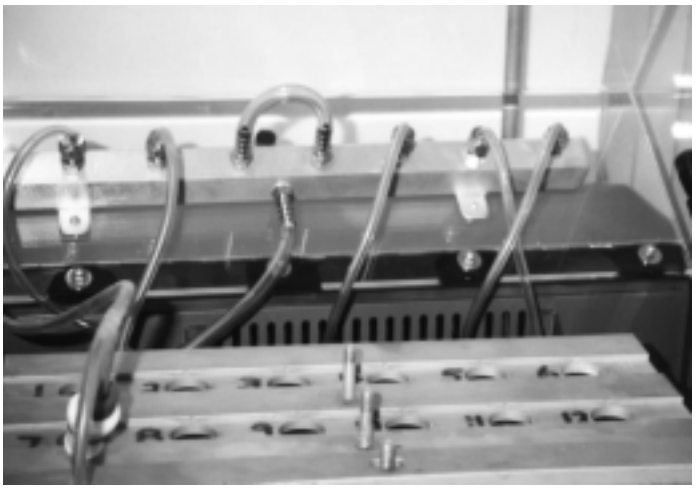

FIG. 7 Gassing Manifold for Venting

provides an orbital shaking motion in a controlled speed and temperature environment.

6.5.1 A special test tube assembly rack[8] (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.

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[9] is required. This system is composed of:

6.8.1 *Optics and Illumination*:

6.8.1.1 Nikon Epiphot 200 inverted metallurgical microscope,

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,

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):

(a) 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.

[8] 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. 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.

[9] 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. 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.

TABLE 1 Organic Solvent

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

[A] **Warning**—See the appropriate Materials Safety Data Sheet.
[B] **Warning**—Flammable. Cannot be made nontoxic. Health hazard.
[C] **Warning**—Flammable. Health hazard.

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

(c) 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_7 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[10] 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 (**Warning**—see 7.1.3), approximately 2 oz, to cover the 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 (**Warning**—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 (**Warning**—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 (**Warning**—see 7.1.3), followed by acetone (**Warning**—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 µL/h, minimum, to 220.82 mL/min, maximum. Required accuracy is ± 1 %, and reproducibility is ± 0.1 %.

[10] 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. 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.

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 ± 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 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.[10]

8.4.8.2 Connect these tubes to the gassing manifold.

(a) 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).

(b) 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, $^{19}/_{22}$ 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 ± 0.1°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,[11] 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 (**Warning**—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) (**Warning**—See 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] Available from the TMC.

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.

11.6.3 Rotate (without skin contact) the Calibration Reference Specimen, and take 20 different readings.

11.6.4 Repeat the previous step for each specimen. (The Calibration Reference Specimen is the first and last specimen analyzed to ensure no drift has occurred during analysis.)

11.6.5 Print the spreadsheet program results.

12. Precision and Bias

12.1 *Precision:*[12]

[12] See RR:D02-1483 for details. Available from ASTM Headquarters.

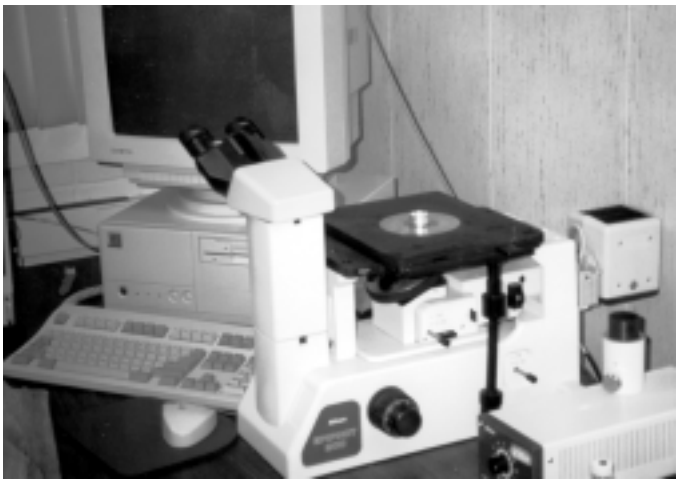

FIG. 8 Microscope, Stage, and Computer

12.1.1 *Intermediate Precision (formerly called repeatability)*—The difference between two results obtained with the same oil, using the same test method, in the same laboratory, using the same apparatus (different test tubes), would, in the normal and the correct operation of the test method, exceed the following value in only one case in twenty:

15.15 *AGV*

12.1.2 *Reproducibility*—The difference between two single and independent test results obtained with the same oil in different laboratories would, in the normal and the correct operation of the test method, exceed the following value in only one case in twenty:

18.89 *AGV*

12.2 *Bias*—No bias is believed to exist. However, this aspect of the test will be reevaluated after the test method has been in use by several laboratories over an appropriate period of time.

13. Keywords

13.1 Ball Rust Test; corrosion; engine oil; rust

ANNEXES

(Mandatory Information)

A1. DIGITAL AIR FLOWMETER CALIBRATION

A1.1 Connect the reference air line to the digital gas flowmeter inlet port, and attach capillary tubing from the flowmeter outlet to a calibrated bubble meter.

A1.2 Calculate the actual air flow rate for the digital flowmeter by using the following formula:

$$\text{air flow rate (mL/min)} = \frac{100 \text{ mL}}{\text{time (min)}} \quad \text{(A1.1)}$$

A1.3 Repeat A1.2 for digital indicator readings of 35.0, 40.0, 45.0, and 50.0 mL/min.

A1.4 Plot the calibration curve of digital airflow set point versus actual airflow rate to provide an airflow correction chart.

A1.5 *Alternatively*, connect the reference air line to the digital gas flowmeter inlet port, and attach capillary tubing from the flowmeter outlet to a large (1 L or larger) open beaker, partially filled with water.

A1.6 Fill a 100-mL volumetric cylinder with deionized water, and secure the cylinder in an inverted position to a stand. Make sure the cylinder is perpendicular to the water level.

A1.6.1 Attach capillary tubing from the top of the cylinder, and place the tubing outlet into the partially filled beaker.

A1.7 Adjust the pressure gage valve to set the digital indicator at 30.0 mL/min. Allow 5 min to ensure that the airflow has stabilized.

A1.8 Remove the capillary air line from the beaker, and attach it to the bottom valve of the inverted volumetric cylinder.

A1.9 Measure the minutes required to displace 100 mL of water from the cylinder.

A1.10 Record the set pressure, digital air flowmeter reading, and displacement time.

A1.11 Repeat directions provided in A1.2-A1.4 to calculate the actual airflow rate for the digital flowmeter.

A2. SHAKER TEMPERATURE CALIBRATION

A2.1 Insert 10 mL of a representative oil sample into a 20-mL plastic syringe, as part of a test tube assembly, and place the assembly in the test tube rack at rack location No. 10 (second row from the vent line manifold and fourth slot from the left).

A2.2 Immerse a calibrated digital thermometer into the oil sample without blocking the air inlet port that is left open.

A2.3 Introduce 40 cm^3/min of air to circulate the oil.

A2.4 Adjust the shaker temperature control to 40°C. Record the actual temperature from the calibrated digital thermometer every 30 min for 4 h.

A2.5 Repeat A2.2-A2.4 for shaker temperature set points of 50°C and 60°C.

A2.6 Plot the calibration curve of shaker set point temperature versus actual temperature to provide a temperature control correction chart.

A3. MULTIPLE SYRINGE PUMP LIQUID FLOW CALIBRATION

A3.1 Determine the density of the acid solution, in g/mL. It is convenient to weigh the 1 L of solution after it has been prepared (see 7.1.8.2). Use a calibrated balance to obtain the weight of the 1 L of solution, and subtract the weight of the empty flask to obtain the actual weight of the solution. (Or, use a calibrated density meter.)

A3.2 Weigh 24 of the 20-mL scintillation vials (labeled 1 to 24) individually on a calibrated balance.

A3.3 Withdraw 6 mL of acid solution from a wide-mouth beaker into each of the twenty-four 5-mL plastic syringes by hand, and attach the syringes to the acid inlet ports of the three-way valves.

A3.3.1 Turn the three-way valves to the *two-way open* position, and withdraw by hand about 0.5 mL of solution from each syringe into a waste beaker while ensuring that no air bubbles remain in the syringes.

A3.4 Place the individual syringes, which now contain 5.5 mL of solution, on the holder of the multiple syringe pumps (consists of three separate pumps with eight syringes operated by each pump).

A3.4.1 Press the retaining brackets firmly against the syringe barrel flange and the plunger flange and tighten the retaining screws. *A good alignment of all 24 syringes against the retaining brackets is crucial for good calibration.*

A3.5 Activate the multiple syringe pumps and withdraw 1 mL of solution from each of the 24 plastic syringes into a waste beaker for 2 min at 0.5 mL/min. During this 2 min of pump operation, make sure that all 24 syringe plungers are well seated in the retaining brackets.

A3.6 Reduce the pump speed to 0.150 mL/h, and operate for 1 h. Ensure that all syringe plungers are properly aligned at the retaining brackets of the pusher block.

A3.6.1 Stop the pump, and ensure that no air bubbles remain in the capillary lines.

A3.7 Place filled capillary lines into corresponding 20-mL vials.

A3.7.1 Seal the vial opening with parafilm to avoid any evaporation of the acid solution during the calibration.

A3.8 Start the pump, and also start a calibrated clock for 18 h.

A3.8.1 At the end of 18 h, stop the pump and remove the parafilm and capillary lines.

A3.9 Reweigh the vials, and calculate the weight gain for each vial. Total the weight gains for each bank of the appropriate eight vials to obtain the weight gain for an individual pump.

A3.10 Calculate the actual pump flow rate for the individual three pumps by using the following formula:

$$\text{flow rate (mL/h)} = \frac{\text{weight gain (g)}}{\text{density (g/mL)} \times \text{time (h)}} \quad \text{(A3.1)}$$

A3.11 Repeat A3.3-A3.10 for pump flow rate set points of 0.20 and 0.25 mL/h.

A3.12 For each pump, plot the calibration curve of set flow rate versus actual flow rate to provide a pump flow correction chart.

A4. THE ROLE OF THE ASTM TEST MONITORING CENTER (TMC)

A4.1 The TMC is a nonprofit organization located in Pittsburgh, PA.

A4.2 *Operation*—The TMC operates in accordance with the Rules and Regulations Governing the ASTM Test Monitoring System, which was developed and approved by Subcommittee D02.B0, and subsequently, approved by Committee D02.

A4.3 *Management*—The management of the ASTM Test Monitoring System is vested in the Test Monitoring Board (TMB) elected by Subcommittee D02.B0. The TMB, in turn, selects the TMC Administrator who is responsible for directing the activities at the TMC.

A4.4 *Duties*—The TMC is staffed to administer technical studies, conduct laboratory visits, perform statistical analyses of reference oil test data, provide reference oils to test laboratories, and maintain the calibration programs for various test methods as directed by the TMB.

A4.5 *Coordination*—The TMC coordinates its activities among the test sponsors, test developers, surveillance panels, and testing laboratories.

A4.6 *Income*—The TMC's operating income is obtained from fees levied on the reference oils supplied to the test laboratories and on the calibration tests conducted.

A4.7 *Laboratory Participation*—For those laboratories choosing to utilize the services of the TMC in maintaining the calibration of a monitored ASTM test method, calibration testing is required at regular intervals as determined by the responsible surveillance panel.

A4.7.1 These calibration tests are conducted using coded reference oils as outlined in Section 10 of this test method.

A4.7.2 It is the laboratories' responsibility to maintain the calibration of the test method, and to keep on-site reference oil inventory at or above the minimum level specified by the TMC.

A4.8 *New Laboratories*—New laboratories desiring to participate in the ASTM Test Monitoring System should contact the TMC Administrator. Information concerning fees, laboratory inspections, testing practices, appropriate surveillance panel membership, and rater training will be provided.

A4.8.1 Initially, a new laboratory will be requested to conduct reference oil tests to ensure that the laboratory is using the proper testing techniques.

A4.9 *New Reference Oils*—When new reference oils are introduced, participating laboratories will be requested to run and donate the tests necessary to develop performance targets and precision, and performance acceptance limits.

A4.9.1 The appropriate surveillance panel with TMC input will design the test program for prospective new reference oils.

A4.10 *Information Letters*—Occasionally, it may become necessary to change a test method, and notify participating laboratories of the change, prior to approval of the change by Subcommittee D02.B0. In such a case, the TMC will issue an Information Letter.

A4.10.1 Normally, prior to each Subcommittee D02.B0 meeting held in conjunction with the semiannual Committee D02 meeting, the accumulated information letters are balloted by Subcommittee D02.B0.

A4.10.2 Subsequently, test methods that are affected by changes in Information Letters are revised accordingly, balloted in Committee D02, and finally approved by ASTM.

A4.10.3 By these actions, ASTM due process procedures are applied to the TMC Information Letters.

A4.10.4 The ASTM Committee on Technical Committee Operations (COTCO) in 1984 gave authority for the issuance of information letters, as follows: "COTCO recognizes that D02 has a unique and complex situation. The use of Information Letters is approved providing each letter contains a disclaimer to the effect that such has not obtained ASTM consensus. These Information Letters should be moved to such consensus as rapidly as possible."

A4.10.5 Information letters issued subsequent to the issue date of this test method update the test method and should be obtained from the TMC.

A4.11 *Memoranda*—The TMC may also issue memoranda to convey information, such as clarification of the test procedure and approval for alternative test parts or materials or for any other matters having no affect on test performance, results, precision, and bias.

A5. REPORT FORMAT AND DATA DICTIONARY

A5.1 Use the standardized report form package detailed in A5.2 to report all test results for both reference and non-reference oils.

A5.2 *Test Report Forms with Data Dictionary Variable Mnemonics*—The following report forms contain field names for all reported variables. Use these variables in electronic data transmission as defined by the Data Communications Committee (see Figs. A5.1-A5.4).

A5.2.1 The test report variable data dictionary (see Fig. A5.4) contains all of the field names used in the A5.2 report form package.

VERSION 20000120

CONDUCTED FOR

TSTSPON1

TSTSPON2

LABVALID	V = VALID
	I = INVALID

Test Number			
Shaker Table ID:	*SHKTBLID*	Test Run No.:	*TSTRUNNO*
End of Test Date:	*DTCOMP*	End of Test Time:	*EOTTIME*
Oil Code: [A]	*CMIR/OILCODE*		
Alternate Codes	*ALTCODE1*	*ALTCODE2*	*ALTCODE3*

In my opinion this test *OPVALID* been conducted in a valid manner in accordance with the Test Method DXXXX and the appropriate amendments through the information letter system. The remarks included in the report describe the anomalies associated with this test.

[A] CMIR or Non-Reference Oil

SUBLAB

Testing Laboratory

SUBSIGIM

Signature

SUBNAME

Typed Name

SUBTITLE

Title

FIG. A5.1 Final Report Cover Sheet

Summary of Results

Lab	*LAB*		
Shaker Table ID	*SHKTBLID*	Test Run No.	*TSTRUNNO*
Test Start Date	*DTSTRT*	Test Start Time, hh:mm	*STRTTIME*
Test Finish Date	*DTCOMP*	Test Finish Time, hh:mm	*EOTTIME*
Image Analysis Software Version	*IMANLVER*		
Fresh Ball Gray Value	SOT: *SOTGRVAL*	EOT:	*EOTGRVAL*

Reference Oil				**Non-reference Oil**			
CMIR Code	*CMIR*			Oil Code	*OILCODE*		
TMC Oil ID	*IND*						
Lab Oil Code	*RLABOCOD*			Lab Oil Code	*LABOCODE*		
SAE Viscosity	*RSAEVISC*			SAE Viscosity	*SAEVISC*		
Acid Batch Serial No.	*RACIDBNO*			Acid Batch Serial No.	*ACIDBNO*		
	Tube 1	**Tube 2**	**Average**		**Tube 1**	**Tube 2**	**Average**
Instrument Position	*RINSTP1*	*RINSTP2*		Instrument Position	*INSTP1*	*INSTP2*	
Air Flow Rate, cc/min	*RAFR1*	*RAFR2*		Air Flow Rate, cc/min	*AFR1*	*AFR2*	
Number of Image Readings Taken	*RREADNO1*	*RREADNO2*		Number of Image Readings Taken	*READNO1*	*READNO2*	
Average Gray Value	*RAGRYVL1*	*RAGRYVL2*	*RAGRYVL*	Average Gray Value	*AGRYVL1*	*AGRYVL2*	*AGRYVL*
Tube to Tube Percent Deviation	*RTB12PDV*			Tube to Tube Percent Deviation	*TB12PDV*		

Ball Set Serial Number			
Reference Oil		**Non-reference Oil**	
Tube 1	*RBSETNO1*	Tube 1	*BSETNO1*
Tube 2	*RBSETNO2*	Tube 2	*BSETNO2*

FIG. A5.2 Summary of Results

Comments

Lab: *LAB*	Shaker Table ID: *SHKTBLID*	Test Run No.: *TSTRUNNO*
EOT Date: *DTCOMP*		EOT Time: *EOTTIME*
Start Date: *DTSTRT*		Start Time: *STRTTIME*
Oil Code: *CMIR/OILCODE*		

Number of Comment Lines	*TOTCOM*
OCOMR001	

FIG. A5.3 Comments

20-jan-2000

Data Dictionary

Sequence	Form	Test Area	Field Name	Field Length	Decimal Size	Data Type	Units/Format	Description
10	1	BRT	VERSION	8	0	C	YYYYMMDD	BRT VERSION 20000120
20	1	BRT	TSTSPON1	40	0	C		CONDUCTED FOR, FIRST LINE
30	1	BRT	TSTSPON2	40	0	C		CONDUCTED FOR, SECOND LINE
40	1	BRT	LABVALID	1	0	C	V or I	TEST LAB VALIDATION (V or I)
50	1	BRT	SHKTBLID	15	0	C		SHAKER TABLE ID
60	1	BRT	TSTRUNNO	15	0	C		TEST RUN NUMBER
70	1	BRT	DTCOMP	8	0	C	YYYYMMDD	DATE COMPLETED (YYYYMMDD)
80	1	BRT	EOTTIME	5	0	C	HH:MM	END OF TEST TIME (HH:MM)
90	1	BRT	OILCODE	38	0	C		NON-REFERENCE OIL CODE
100	1	BRT	CMIR	6	0	C		CMIR
120	1	BRT	ALTCODE1	10	0	C		ALTERNATE OIL CODE 1
130	1	BRT	ALTCODE2	10	0	C		ALTERNATE OIL CODE 2
140	1	BRT	ALTCODE3	10	0	C		ALTERNATE OIL CODE 3
150	1	BRT	OPVALID	8	0	C	HAS/HAS NOT	OPERATIONAL VALIDITY (HAS/HAS NOT)
160	1	BRT	SUBLAB	40	0	C		SUBMITTED BY: TESTING LABORATORY
170	1	BRT	SUBSIGIM	70	0	C		SUBMITTED BY: SIGNATURE IMAGE
180	1	BRT	SUBNAME	40	0	C		SUBMITTED BY: SIGNATURE TYPED NAME
190	1	BRT	SUBTITLE	40	0	C		SUBMITTED BY: TITLE
200	2	BRT	LAB	2	0	C		LAB CODE
210	2	BRT	DTSTRT	8	0	C	YYYYMMDD	START DATE (YYYYMMDD)
220	2	BRT	STRTTIME	5	0	C	HH:MM	START TIME (HH:MM)
230	2	BRT	IMANLVER	10	0	C		IMAGE ANALYSIS SOFTWARE VERSION
240	2	BRT	SOTGRVAL	4	0	N		START OF TEST FRESH BALL GRAY VALUE
250	2	BRT	EOTGRVAL	4	0	N		END OF TEST FRESH BALL GRAY VALUE
260	2	BRT	IND	6	0	C		TMC OIL CODE
270	2	BRT	RLABOCOD	12	0	C		REFERENCE LABORATORY INTERNAL OIL CODE
280	2	BRT	LABOCODE	12	0	C		NON-REFERENCE LABORATORY INTERNAL OIL CODE
290	2	BRT	RSAEVISC	7	0	C		REFERENCE SAE VISCOSITY GRADE
300	2	BRT	SAEVISC	7	0	C		NON-REFERENCE SAE VISCOSITY GRADE
310	2	BRT	RACIDBNO	10	0	C		REFERENCE ACID BATCH SERIAL NUMBER
320	2	BRT	ACIDBNO	10	0	C		NON-REFERENCE ACID BATCH SERIAL NUMBER
330	2	BRT	RINSTP1	2	0	C		REFERENCE INSTRUMENT POSITION TUBE 1
340	2	BRT	RINSTP2	2	0	C		REFERENCE INSTRUMENT POSITION TUBE 2
350	2	BRT	INSTP1	2	0	C		NON-REFERENCE INSTRUMENT POSITION TUBE 1
360	2	BRT	INSTP2	2	0	C		NON-REFERENCE INSTRUMENT POSITION TUBE 2
370	2	BRT	RAFR1	5	1	N	cc/min	REFERENCE AIR FLOW RATE TUBE 1 (cc/min)
380	2	BRT	RAFR2	5	1	N	cc/min	REFERENCE AIR FLOW RATE TUBE 2 (cc/min)
390	2	BRT	AFR1	5	1	N	cc/min	NON-REFERENCE AIR FLOW RATE TUBE 1 (cc/min)
400	2	BRT	AFR2	5	1	N	cc/min	NON-REFERENCE AIR FLOW RATE TUBE 2 (cc/min)
410	2	BRT	RREADNO1	3	0	N		REFERENCE NUMBER OF IMAGE READINGS TAKEN TUBE 1
420	2	BRT	RREADNO2	3	0	N		REFERENCE NUMBER OF IMAGE READINGS TAKEN TUBE 2
430	2	BRT	READNO1	3	0	N		NON-REFERENCE NUMBER OF IMAGE READINGS TAKEN TUBE 1
440	2	BRT	READNO2	3	0	N		NON-REFERENCE NUMBER OF IMAGE READINGS TAKEN TUBE 2
450	2	BRT	RAGRYVL1	4	0	N		REFERENCE AVERAGE GRAY VALUE TUBE 1
460	2	BRT	RAGRYVL2	4	0	N		REFERENCE AVERAGE GRAY VALUE TUBE 2
470	2	BRT	RAGRYVL	4	0	N		REFERENCE AVERAGE GRAY VALUE AVERAGE
480	2	BRT	AGRYVL1	4	0	N		NON-REFERENCE AVERAGE GRAY VALUE TUBE 1
490	2	BRT	AGRYVL2	4	0	N		NON-REFERENCE AVERAGE GRAY VALUE TUBE 2
500	2	BRT	AGRYVL	4	0	N		NON-REFERENCE AVERAGE GRAY VALUE AVERAGE
510	2	BRT	RTB12PDV	5	1	N	%	REFERENCE TUBE 1 TO TUBE 2 PERCENT DEVIATION (%)
520	2	BRT	TB12PDV	5	1	N	%	NON-REFERENCE TUBE 1 TO TUBE 2 PERCENT DEVIATION (%)
530	2	BRT	RBSETNO1	20	0	C		REFERENCE BALL SET SERIAL NUMBER TUBE 1
540	2	BRT	BSETNO1	20	0	C		NON-REFERENCE BALL SET SERIAL NUMBER TUBE 1

FIG. A5.4 Data Dictionary

20-jan-2000 Report: ASTM Data Dictionary

Sequence	Form	Test Area	Field Name	Field Length	Decimal Size	Data Type	Units/Format	Description
550	2	BRT	RBSETNO2	20	0	C		REFERENCE BALL SET SERIAL NUMBER TUBE 2
560	2	BRT	BSETNO2	20	0	C		NON-REFERENCE BALL SET SERIAL NUMBER TUBE 2
570	3	BRT	TOTCOM	3	0	Z		TOTAL LINES OF COMMENTS & OUTLIERS
580	3	BRT	OCOMRxxx	70	0	C		OTHER DOWNTIME COMMENTS XXX

FIG. A5.4 Data Dictionary *(continued)*

```
#########################################################################
#                                                                       #
#           D a t a  D i c t i o n a r y  R e p e a t i n g             #
#              F i e l d  S p e c i f i c a t i o n s                    #
#                                                                       #
#########################################################################
# The following contains specifications and field groupings for fields in the
# Data Dictionary that are REPEATING Fields.  These fields can be identified
# in the Data Dictionary by the Hxxx or Rxxx in the last four positions of the
# field name.
#
# Repeating fields are used to specify repeating measurements.
#
# The format for a repeating field name is 4 descriptive characters followed
# by the letter H or R followed by 3 characters for the actual interval
# the measurement was taken. The field will always be a total of 8 characters.
#
# Example ABCDHxxx.
#
# The following is the format of this specification:
#
# Column 1 - 8:   Repeating Field Name
# Column 10 - 17: The Parent Field Name of the Group
# Column 19 - 26: The Measurement Interval Group Name
# Column 30 - 80: Comments about the Repeating Field Group.
#
# The lines following the Repeating Field Name Record will contain the required
# measurements for the particular field.  Multiple 80 characters lines
# can be specified.  A blank line marks the end of each specification.
#
# The Field Name in Column 10-17 designates the the Group in which the field
# belongs.  The First field name in a group is the Parent of the grouping
# and can be used to determine how fields should be grouped.
# The changing of the Parent Field marks the end of a repeating group
# specification.
#
# Example:
#
# VIS_Hxxx, DVISHxxx and PVISHxxx expanded for transmission (8 and 16 hours):
#
#           VIS_H008
#           DVISH008
#           PVISH008
#           VIS_H016
#           DVISH016
#           PVISH016
#
# Note:  During electronic transmission, repeating field groups must be kept
#        together within the specified group but the order within the group
#        does not have to be maintained.
#
#########################################################################
#                 Start of Field Grouping Specifications                #
#########################################################################
#
BRT VERSION 20000120
OCOMRxxx OCOMRxxx OCOMRxxx   OTHER DOWNTIME COMMENTS XXX
```

FIG. A5.4 Data Dictionary *(continued)*

APPENDIX

(Nonmandatory Information)

X1. PREPARATION OF ACID SOLUTION

TABLE X1.1 Acid Solution

Component	Amount
	(ensure accuracy within 0.2 %)
Acetic acid	6.646 g
HBr	4.892 g
HCl	103.232 g
Deionized water	Fill to 1-L mark

X1.1 *Acid Solution Preparation (Example: 1-L of solution)*:

X1.1.1 Accurately weigh each of the chemicals listed in Table X1.1 into separate clean, dry 50-mL beakers (100-mL beaker for the HCl 1.0 *N*), using a three-place balance. Flush and rinse a 1-L flask with a small amount of deionized water, and empty the individual beakers into the flask. Fill the 1-L flask with deionized water up to the 1-L mark.

X1.1.2 Put a magnetic stir bar into the 1-L flask, put a stopper in the flask, and place the flask on a stirring plate. Adjust to a moderate stirring speed, and allow stirring for 30 min at room temperature. Remove the stir bar from the flask.