



Designation: D5800 – 21

Standard Test Method for Evaporation Loss of Lubricating Oils by the Noack Method¹

This standard is issued under the fixed designation D5800; 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 four procedures for determining the evaporation loss of lubricating oils (particularly engine oils). The evaporation measured is reported as percent total loss. The test method relates to one set of operating conditions but may be readily adapted to other conditions as required.

1.2 Procedure B and Procedure D that are in the main section of the test method provide equivalent results. Procedures A and C, which are in [Annex A1](#) and [Annex A2](#), have equivalent results. It has been determined that Procedures A and C show a slight bias when compared to Procedures B and D. Procedures B and D give slightly higher results versus Procedures A and C on formulated engine oils, while Procedures B and D give lower results versus Procedures A and C on basestocks. Thus, a correction factor is utilized to convert between the two sets of Procedures based on the fluid type.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.06 on Analysis of Liquid Fuels and Lubricants.

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² 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.

[D4057 Practice for Manual Sampling of Petroleum and Petroleum Products](#)

[D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products](#)

[D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance](#)

[D6300 Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products, Liquid Fuels, and Lubricants](#)

2.2 *DIN Standards:*³

[DIN 1725 Specification for Aluminum Alloys](#)

[DIN 12785 Specifications for Glass Thermometers](#)

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *evaporation loss, n*—of a lubricating oil by the Noack method, that mass of volatile oil vapors lost when the oil is heated in a test crucible through which a constant flow of air is drawn.

3.1.2 *volatility, n*—the tendency of a liquid to form a vapor.

4. Summary of Test Method

4.1 A measured quantity of sample is placed in an evaporation crucible or reaction flask that is then heated to 250 °C with a constant flow of air drawn through it for 60 min. The loss in mass of the oil is determined.

4.2 Interlaboratory tests have shown that Procedure A, Procedure B, Procedure C, and Procedure D yield statistically equivalent precision, with a correlation coefficient of $R^2 = 0.996$ (see research report).

5. Significance and Use

5.1 The evaporation loss is of particular importance in engine lubrication. Where high temperatures occur, portions of an oil can evaporate.

5.2 Evaporation may contribute to oil consumption in an engine and can lead to a change in the properties of an oil.

³ Available from Deutsches Institut für Normung e.V.(DIN), Am DIN-Platz, Burggrafenstrasse 6, 10787 Berlin, Germany, <http://www.din.de>.

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

5.3 Many engine manufacturers specify a maximum allowable evaporation loss.

5.4 Some engine manufacturers, when specifying a maximum allowable evaporation loss, quote this test method along with the specifications.

5.5 The apparatuses used in Procedure C (see Annex A2) and Procedure D, also permit collection of the volatile oil vapors for determination of their physical and chemical properties. Elemental analysis of the collected volatiles may be helpful in identifying components such as phosphorous, which has been linked to premature degradation of the emission system catalyst.

Procedure B—Non-Woods Metal Apparatus

6. Apparatus

6.1 The following procedure, Procedure B, describes an automated test method that uses the same principle, and the same crucible as Procedure A. Only the heat transfer medium to the sample and lid are different. It does not use Woods alloy, and the sample temperature is directly monitored.

6.2 *Noack Evaporative Testers*⁴, (see Fig. 1 and Fig. 2 for both models), comprising the following:

6.2.1 *Heating Block Unit*, electrically heated by base and jacket heaters, having a total power consumption sufficient to ensure a specimen temperature profile similar to the one

recorded in the specimen when heated with the Woods metal heater block. In the center of the heating block, there is a circular recess to insert the evaporative crucible. The jacket heater is configured to ensure a direct contact with the crucible. A mechanism is provided to open the jaws for crucible insertion. Two catches on the block prevent the crucible from rising, and the base heater is spring loaded to ensure a direct contact with the crucible.

6.2.2 *Evaporative Crucible*, with screw cover (see Fig. 3). The crucible is made of stainless steel (see Fig. 4). Above the support ring is the thread for the cover. The nickel-plated brass cover (see Fig. 5) is hermetically sealed to the crucible by an internal conical sealing surface. Three nozzles of hardened steel (see Fig. 6) permit the air stream to pass through the cover. The extraction tube (see Figs. 7 and 8), which slopes downward, leads from a threaded and sealed connection in the center of the cover.

6.2.3 *Temperature Probe*—The specimen temperature measuring device shall have an accuracy of 0.5 °C, or better, and a resolution of 0.1 °C, or better. The probe is provided with a calibration certificate of 250.0 °C with a precision of ±0.1 °C. Its diameter is 4 mm, and its position is as indicated in Fig. 4. It should be calibrated with appropriate procedure at appropriate frequency (minimum once a year).

6.3 *Balance*, capable of weighing at least 500 g to the nearest 0.01 g.

6.4 *Crucible Clamp and Spanner*.

6.5 *Reamer*, 2 mm diameter.

6.6 *Ball Bearing*, 3 mm to 5 mm diameter.

6.7 *Glassware Assembly*, strictly identical to the description in A1.1.6 – A1.1.12 and A1.1.15 of Procedure A.

⁴ The sole source of supply of the apparatus known to the committee at this time for the Model A/B (NCK2/NCK2 5G) is ISL (PAC LP), B.P. 70285 14653 Carpiquet Cedex, Verson, France. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.



FIG. 1 Model A

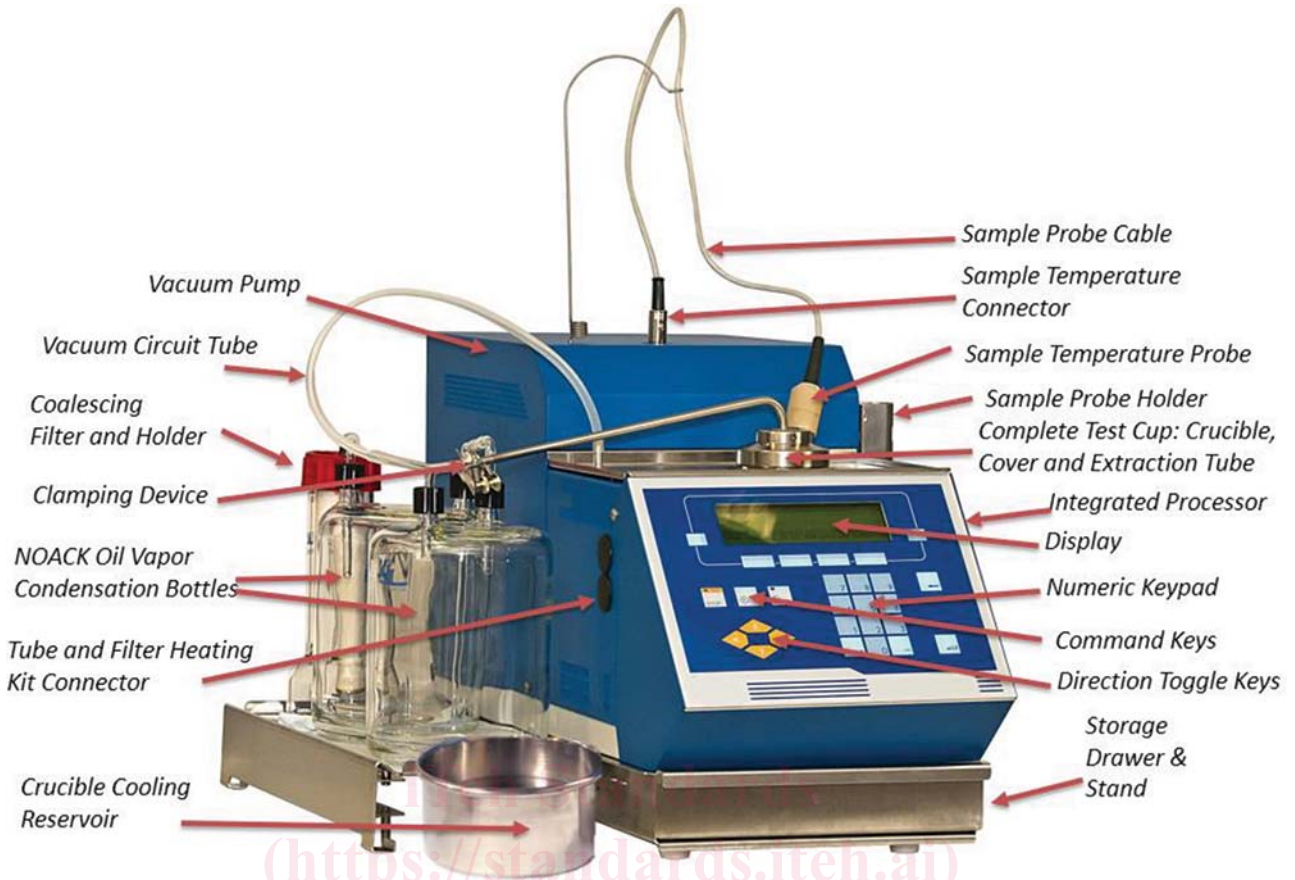
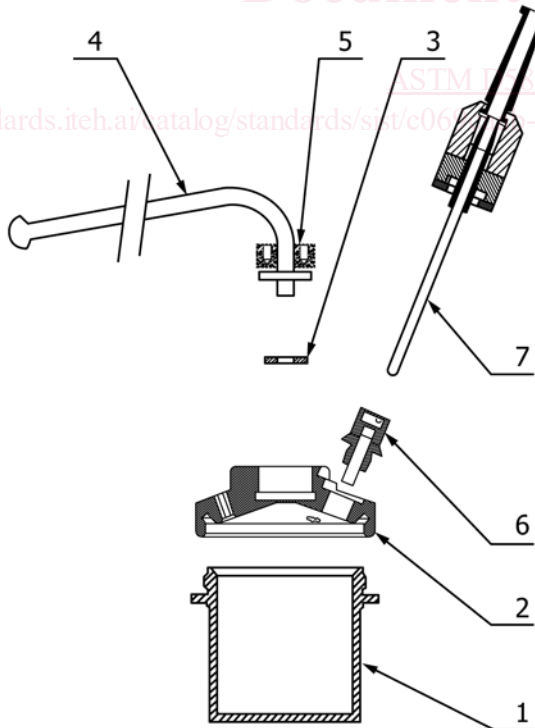


FIG. 2 Model B



- 1 - Cup
- 2 - Cover
- 3 - Extraction tube seal
- 4 - Extraction tube
- 5 - Extraction tube nut
- 6 - Temperature probe locking device
- 7 - Temperature probe

FIG. 3 Crucible with Temperature Probe

6.8 Vacuum Pump.

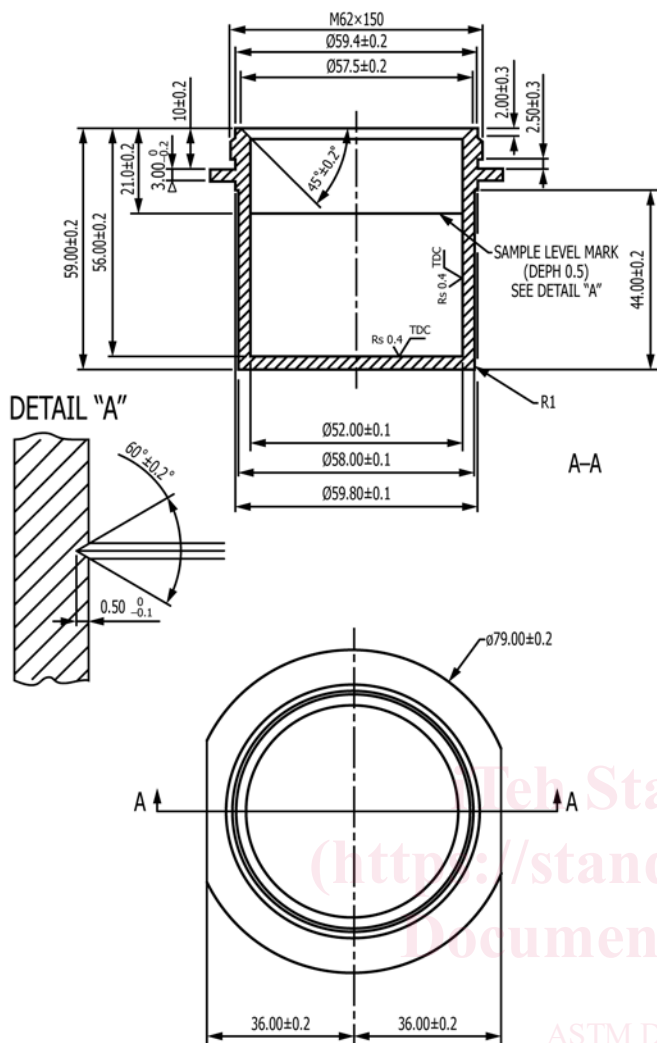


FIG. 4 Noack Cup (Detail 1 of Fig. 3)

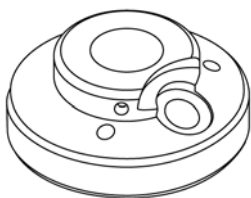


FIG. 5 Crucible Cover (Detail 2 of Fig. 3)

6.9 *Central Processing Unit (CPU)*, capable of controlling the specimen temperature, the vacuum, the time, the heating, and the printing. The specimen is heated to $245.2\text{ }^{\circ}\text{C} \pm 0.5\text{ }^{\circ}\text{C}$ with the temperature profile recorded in the specimen when tested with a Woods metal apparatus (1 h at $250\text{ }^{\circ}\text{C}$) with automatic test duration compensation. The automatic test duration compensation is used because a test may be started with a heating block at room temperature or at hot temperature when several tests are carried without cooling phase. The CPU automatically adjusts the pressure differential of $20\text{ mm} \pm 0.2\text{ mm}$. These conditions can be checked with the printed report.

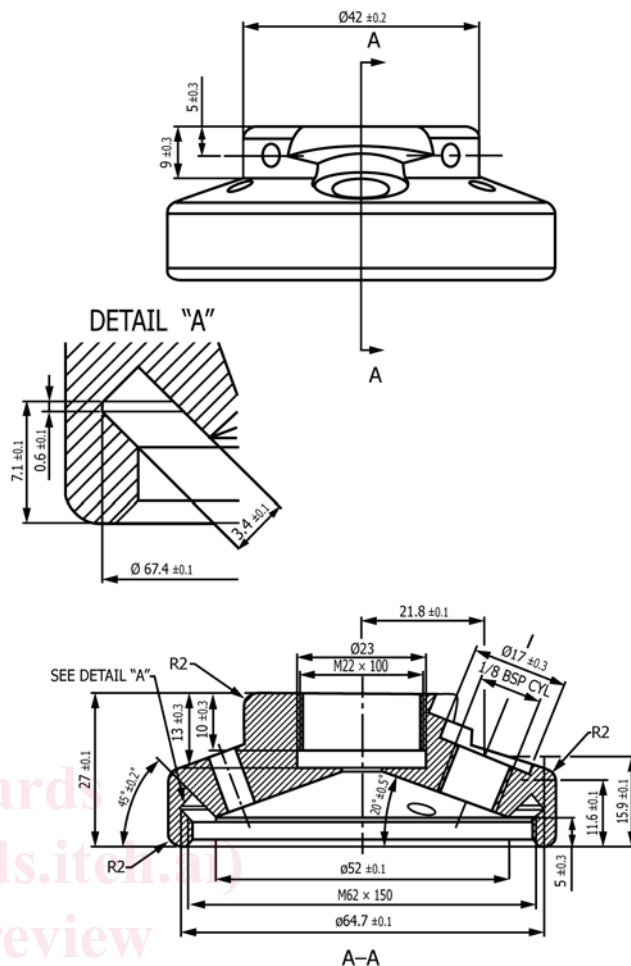


FIG. 5 Crucible Cover (continued)

6.10 *Printer*, to print the graphs of the specimen temperature and the vacuum recorded during the test.

6.11 TMC calibration of the instrument is required for results used in API licensing of formulated automotive engine oils. The calibration procedure is defined in the Lubricant Test Monitoring System (LTMS) document that is maintained by the ASTM Test Monitoring Center (TMC)⁵ and is governed by the D02.B0.07 Volatility Surveillance Panel. LTMS severity adjustments shall be applied to results used in API licensing of automotive engine oils (12.2.1).

NOTE 1—It is the perception of the D02.B0.07 Volatility Surveillance Panel that maintaining crucible cup and lid pairings is a measure of good practice which minimizes variability. Under authority of the D02.B0.07 Volatility Surveillance Panel, it has determined that for D5800 Procedure B tests to be considered operationally valid on TMC calibrated instruments, crucible cups and lids are to be initially paired by the testing lab and then remain paired for the entire life-cycle of the crucible set. Moreover, labs may implement and retire paired crucible sets as needed but once initially paired for calibration and candidate product testing, crucible cups and lids must remain paired for all subsequent test runs. Cups and lids shall be engraved or indelibly marked to maintain unique identification. Note that the use of multiple cup-lid pairings does not affect the TMC calibration status of an instrument.

⁵ ASTM Test Monitoring Center, 6555 Penn Avenue, Pittsburgh, PA 15206-4489, <http://www.astmtmc.org>.

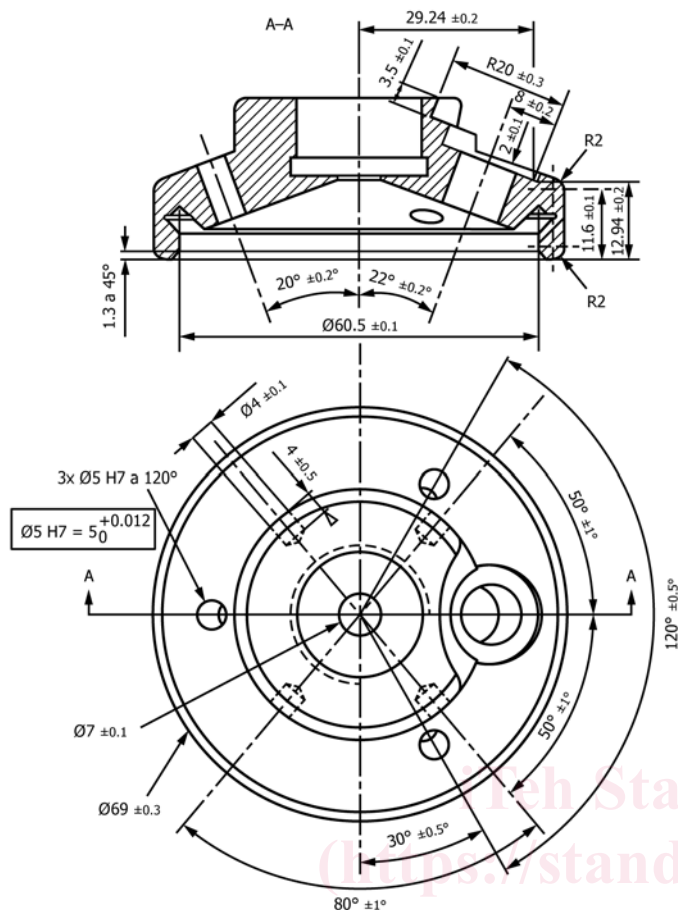


FIG. 5 Crucible Cover (continued)

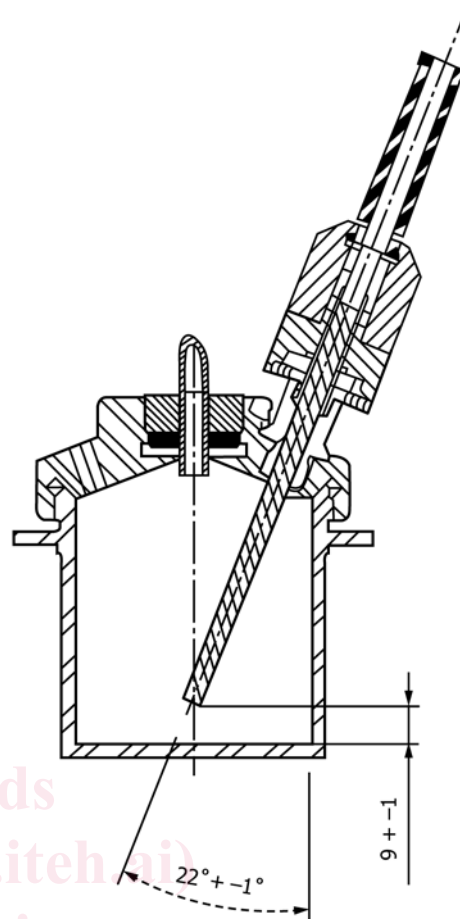


FIG. 6 Specimen Temperature Probe Positioning

7. Reagents and Materials

7.1 *Cleaning Solvent*—A mixture of naphtha and toluene is recommended for cleaning the crucible. (**Warning**—Flammable, vapor harmful.) Overnight soaking may be necessary.

7.2 *Noack Reference Fluid*—Oil such as NCO-12⁶ having a known evaporative loss for use with all D5800 method procedures. The value of which is provided by the manufacturer.

7.3 *Insulated Gloves.*

7.4 *Drying Paper.*

8. Hazards

8.1 *Safety Hazards*—It is assumed that anyone using this test method will either be fully trained and familiar with all normal laboratory practices, or will be under the direct supervision of such a person. It is the responsibility of the operator to ensure that all local legislative and statutory requirements are met.

⁶ The sole source of supply of the reference fluid known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

NOTE 2—It has been reported during testing high Noack oils using some models that the thermocouple probe can become exposed when evaporation loss is high. While the instrument will alarm to indicate that the temperature fluctuation is greater than allowed in the test method, the heater will not shut off. If not noticed, the oil can continue to heat to close to its flash point, and one laboratory has reported the sample to flash when the probe was removed at the end of the test. Hence, it is suggested to contact the instrument manufacturer to remedy possible malfunction.

8.2 (**Warning**—Though the test method calls for a draft-free area, the exhaust fumes from the evaporating oil must be ventilated to an outside source. Precaution shall be taken to avoid any possibility of fire or explosion.) (See **Note A1.3**.)

8.3 An alternate means for preventing draft described in **Appendix X3** was not used in the development of the test method precision statement.

9. Preparation of Apparatus

9.1 To avoid disturbing the thermal equilibrium, the apparatus shall be assembled in a draft-free area. Plexi-glass draft shields are available from the manufacturer, for the model shown in **Fig. 2**, if drafts in the room cause thermal disturbances (see **Appendix X3**).

9.2 Prepare the automated apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment.

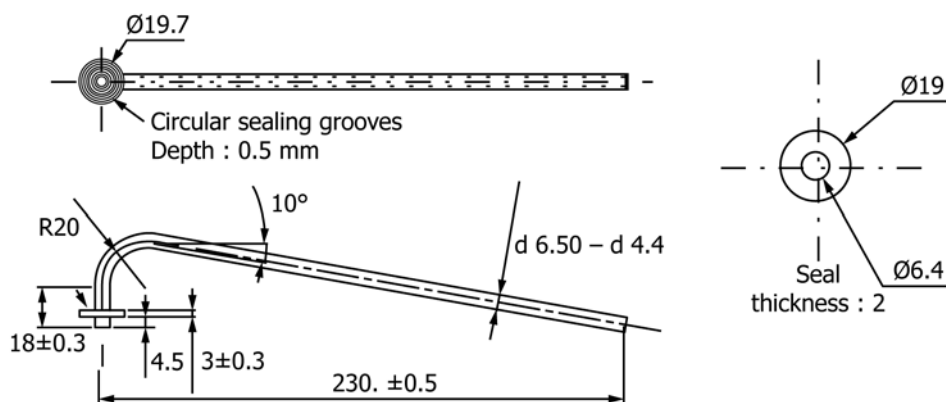


FIG. 7 Extraction Tube (Stainless Steel) with Its Seal (Details 3 and 4 of Fig. 3)

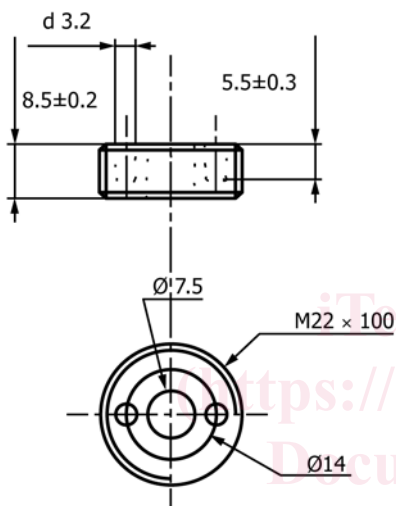


FIG. 8 Extraction Tube Nut (Stainless Steel) (Detail 5 of Fig. 3)

9.3 Clean the glass bottles, the glass tubing, and the Y-piece to prevent a build-up of condensate.

NOTE 3—Condensate should not be allowed to build up in the 2 L glass bottles. These should be washed out with solvent and dried before a maximum 2 cm of condensate collects.

10. Verification

10.1 It is recommended that a Noack reference oil check, such as NCO-12, be run each day of operation. Switch the instrument on a minimum of 30 minutes before running the test to allow temperature stabilization of measurement circuitry

10.2 Make sure that the glassware assembly and the vacuum pump are cleaned and all the connections are sealed.

10.3 Thoroughly clean and dry all parts of the test cup and its accessories before starting the test. Check that the crucible and cover are free from lacquer. Stubborn lacquer should be removed by soaking in the appropriate solvent (see manufacturer's manual). Pass the reamer through each of the three nozzles in the cover to ensure that they are clear. (**Warning**—Using a reamer with a diameter larger than 2 mm can enlarge the nozzles. This can lead to a wrong losses result due to increased air flow.)

10.4 Run the ball bearing through the extraction tube to ensure that it is clear of contaminants.

10.5 Calibrate the vacuum measuring device in accordance with the manufacturer's instructions.

10.6 Weigh the empty cup without its cover to the nearest 0.01 g.

10.7 Weigh into the tared crucible 65.0 g \pm 0.1 g of reference fluid to a precision of 0.01 g. This mass is called M_1 .

10.8 Screw on the cover using the clamp and the spanner. During this phase, make sure that the specimen will never splash on the inside part of the cover. If this occurs, even only one time, the test shall be repeated from 10.3.

10.9 Connect the specimen temperature probe to the instrument.

10.10 Press down on the locking lever located on the front of the heating block. Place the crucible in the heating block. Rotate the crucible, securing the flange under the screw heads. Adjust the final position of the extraction tube so that it is located in front of the arm of the glass Y-piece, and release the locking lever.

10.11 Connect the extraction tube to the arm of the glass Y-piece, and secure the connection with the clamp. Be sure that the stainless extraction tube, the male connection, and the right arm of the Y-piece are properly aligned.

10.12 Start the test by following manufacturer's instructions. When the audible alarm signals the last 3 minutes of the test, turn off the alarm. Stay in front of the equipment, and be prepared to disconnect the extraction tube.

10.13 After 60 min, the test is automatically stopped, and the end of the test alarm sounds. Remove the specimen temperature probe. Disconnect the extraction tube within 15 s maximum. Press down the locking lever. Remove the crucible. Stop the audible alarm.

10.14 Stand the crucible in a cold DI or distilled water bath to a minimum depth of 30 mm.

10.15 Check the printed report to ensure that the specimen temperature and vacuum plotted curves stayed within the indicated limits. Occasionally the electronics will generate erroneous noise spikes in the graphs. These spikes are evident

by a rapid temperature or pressure excursion followed by a rapid return to baseline conditions. These spikes are not cause for invalidating a test. If however, there are gradual temperature and/or pressure drifts or excessive noise spikes beyond specified limits, perform the necessary system maintenance and/or calibrations. In addition, ensure the apparatus complies with the manufacturer's instruction and that the procedure has been adhered to. After these checks, rerun the test from 10.6.

10.16 After 30 min, remove the crucible from the water bath, dry the outside, and carefully remove the lid. This phase is very critical. Make sure that the sample is never in contact with the inside part of the lid.

NOTE 4—It is very important during the manipulation of the crucible, at the start and the end of the test, to not splash the internal face of the cover with the specimen in the cup. When this occurs, it leads to higher losses and the test must be rerun.

10.17 Reweigh the crucible without the lid to the nearest 0.01 g.

10.18 Calculate the M_2 mass by subtracting the empty cup mass from the mass measured in 10.7.

10.19 Calculate to the nearest 0.1 % M/M the evaporation loss of the reference fluid, using the following equation:

$$[(M_1 - M_2)/M_1] \times 100 \quad (1)$$

where:

M_1 = specimen mass before the test, and

M_2 = specimen mass after the test at 245.2 °C.

10.20 Compare the result obtained against the given value for the reference fluid. If the result is within limits, proceed to Section 11.

10.21 If the result is not within the limits, check that the apparatus complies with the manufacturer's instruction and that the procedure has been adhered to.

10.22 Recheck the evaporation loss of the reference oil. To do so, proceed as described in 10.2.

11. Procedure

11.1 Weigh into a tared crucible 65 g ± 0.1 g representative of test specimen to a precision of 0.01 g.

NOTE 5—Sample in accordance with Practice D4057 or Practice D4177.

11.2 Proceed as described in 10.3 – 10.17.

11.3 Calculate to the nearest 0.1 % M/M the evaporation loss of the specimen, using Eq 1.

12. Calculation

12.1 Evaporation loss is obtained from the difference in weight before and after test. The specimen is heated in accordance with the temperature profile recorded in the specimen when tested with a Woods metal apparatus (1 h at 250 °C) with automatic test duration compensation. The automatic test duration compensation is used because a test may be started with a heating block at room temperature or at hot temperature when several tests are carried without cooling phase. The

checking of these conditions can be done with the printed report. Calculate evaporation loss, using the following equation:

$$[(M_1 - M_2)/M_1] \times 100 \quad (2)$$

where:

M_1 = $B - A$,

M_2 = $C - A$,

A = empty crucible weight,

B = crucible plus specimen weight, and

C = crucible plus specimen after the test.

12.2 Procedure B and D provide equivalent results (see RR:D02-1887).⁷ Some consistent differences in results determined using Procedures A/C and B/D have been observed depending on the type of sample tested. A test result obtained using one of the procedures can be transformed to an estimated result on the basis of the other procedure as follows:

12.2.1 *Formulated Engine Oils*—The following relationships are based on the round robin test results on formulated engine oils with volatilities in the range of 10.5 % to 21.5 % Noack:

Value by Noack Procedure B/D = 1.030

× Value by Noack Procedure A/C(3)

Value by Noack Procedure A/C = 0.970

× Value by Noack Procedure B/D(4)

The 95 % confidence limits for the regression coefficient in Eq 3 are 1.021 to 1.033; those for the coefficient in Eq 4 are 0.968 to 0.980.

When results are utilized for API licensing of automotive engine oils, a severity adjustment (SA) shall be applied to the final result of Eq 2. The severity adjustment is determined from the TMC calibration procedure, as stated in 6.11.

12.2.2 The following relationships are based on round robin test results on basestocks with volatilities in the range of 4 % to 25 % Noack:

Value by Noack Procedure B/D = 0.962

× Value by Noack Procedure A/C(5)

Value by Noack Procedure A/C = 1.039

× Value by Noack Procedure B/D(6)

The 95 % confidence limits for the regression coefficient in Eq 5 are 0.950 to 0.959; those for the coefficient in Eq 6 are 1.043 to 1.053.

NOTE 6—The results of Noack residue should not be rounded up before using the multiplication factors given in Eq 3-6.

13. Report

13.1 Report the following information:

13.1.1 The nearest 0.1 % M/M as evaporation loss (Test Method D5800, Procedure B).

13.1.2 If a severity adjustment is applicable to the result, the non-adjusted value, the severity adjustment, and the severity-adjusted value are to be reported.

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1887. Contact ASTM Customer Service at service@astm.org.

13.2 Conversion of values from either D5800 A/C or D5800 B/D to the other:

13.2.1 Only if the nature of the test specimen is known with certainty, in other words, it is known to be either a basestock or a formulated engine oil, the evaporation loss calculated in 12.2.1 on the basis of either Procedure A/C or Procedure B/D may be converted to an equivalent result on the basis of the other procedure. The appropriate equation from those above should be selected and applied according to the type of the sample tested (formulated engine oil or basestock).

13.3 Report the converted result from Procedure A/C to B/D, or Procedure B/D to A/C to the nearest 0.1 m % as evaporation loss of the test sample as converted from the original procedure to the calculated basis procedure.

13.4 If the nature of the test specimen is not known as being either a basestock or a formulated engine oil, the results of the test using Procedure B or D must be identified as being run under Procedure B or D and the value of percent evaporation so obtained will require additional information on the nature of the test specimen for calculations to be made to generate the standard value produced by Procedure A or C.

13.4.1 Converted results should be reported as D5800 A/C (converted from the results obtained by D5800 B/D) or as D5800 B/D (converted from the results obtained by D5800 A/C).

14. Precision and Bias⁸

NOTE 7—Noack S2, Procedure D equipment has been shown to be equivalent in two separate studies (see RR:D02-1887).⁷

⁸ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1785. Contact ASTM Customer Service at service@astm.org. As determined by the round robin, no significant difference in results exists between Procedure B and Procedure C.

14.1 To estimate the precision of Procedure B, the test results from the interlaboratory study were analyzed following Practice D6300.

14.2 The interlaboratory study included ten oils, three base oils, and seven finished oils, tested in thirty laboratories.

14.3 The precision of this test method, as determined by the statistical examination of the interlaboratory study test results, is as follows:

14.3.1 *Repeatability*—The quantitative expression for the random error associated with the difference between two independent results obtained under repeatability conditions that would be exceeded with an approximate probability of 5 % (one case in 20 in the long run) in the normal and correct operation of the test method:

$$\text{Repeatability} = 0.1331 \cdot X^{0.7443} \quad (7)$$

where:

X = average of the two determinations under consideration.

14.3.2 *Reproducibility*—A quantitative expression for the random error associated with the difference between two independent results obtained under reproducibility conditions that would be exceeded with an approximate probability of 5 % (one case in 20 in the long run) in the normal and correct operation of the test method:

$$\text{Reproducibility} = 0.2411 \cdot X^{0.7443} \quad (8)$$

where:

X = average of the two determinations under consideration.

14.4 *Bias*—Since there is no accepted reference suitable for determining the bias for this procedure, no statement on bias is being made.

Procedure D—Automated Non-Woods Metal Noack S2 Volatility Test Apparatus

15. Apparatus⁹

15.1 *Noack S2 (Trademark) Instrument*—Fig. 9, including parts as follows:

15.1.1 *Reaction Vessel and Heater*, capable of heating a sample quickly to test temperatures of 250 °C.

15.1.2 *Teflon Lid Assembly*.

15.1.3 *Coalescing Filter*.

15.1.4 *Pump Filter*.

15.1.5 *Orifice Tube*.

15.1.6 *Magnetic Cross Stir Bar*.

15.1.7 *Thermocouple, PT100*.

15.1.8 *Viton O-rings*.

15.1.9 *Coalescing Filter Cartridges*.

15.1.10 *Pump Filter Cartridges*.

15.2 *Balance*, capable of weighing at least 500 g to the nearest 0.01 g.

15.3 *Beaker*, 600 mL.

15.4 *Cork spacer*, capable of supporting reaction vessel during weighing.

16. Reagents and Materials

16.1 *Cleaning Solvent*, such as VarClean, capable of removing varnish.

⁹ The sole source of supply of the apparatus known to the committee at this time is Tannas Co., 4800 James Savage Rd., Midland, MI 48642. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

16.2 *Hydrocarbon Solvent*, such as hexane.

16.3 *Medium Volatility Reference Oil*.

16.4 *High Volatility Reference Oil*.

17. Preparation and Setup of the Apparatus

17.1 Locate the instrument on a laboratory bench if possible (rather than a hood) to minimize atmospheric pressure fluctuation. Vent the vacuum pump outlet provided on the back of the instrument with a metal or plastic tube leading to a suitable exhaust outlet.

17.2 Insert the thermocouple connector into the thermocouple receptacle on the back of the cabinet.

17.3 Be sure that the inside of the reactor vessel is clean and that the other hardware and tubing are free of any oil residue.

17.4 If collection of the volatilized oil is desired, clean the coalescing filter housing with a hydrocarbon solvent, dry, and install new filter cartridge.

17.5 Turn on the main power switch located on the front panel.

18. Calibration

18.1 The Noack S2 should be warmed up at the beginning of the day. The unit should be turned on with the heat and vacuum pump on for 15 min to 30 min before the first test. If the electronics have already been on for at least 15 min, it is only necessary to wait for the instrument to control the heater temperature to 175 °C ± 5 °C before the first run.

18.2 Calibrate the thermocouple at 250 °C against a certified thermometer or other standard temperature measuring device and, if necessary, adjust the calibration offset on the

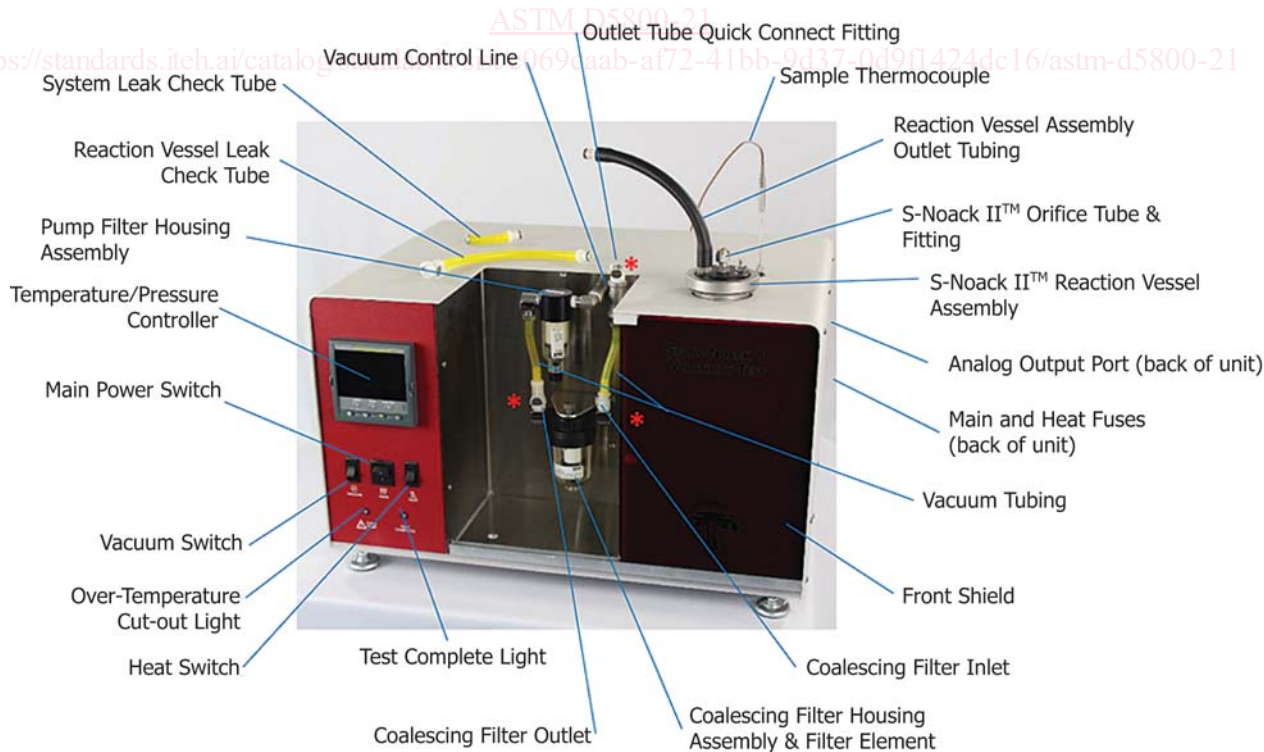


FIG. 9 Noack S2 Test Equipment

temperature controller according to the manufacturer's instructions. This shall be performed semi-annually.

18.3 Calibrate the vacuum pressure to 2.0 cm of water using the manufacturer's instructions. This shall be performed weekly.

18.4 Set stir motor speed to/by manufacturer's recommendations/instructions and shall be performed semi-annually.

18.5 In addition to the procedures described above, TMC calibration of the instrument is required for results used in API licensing of automotive engine oils. The calibration procedure is defined in the Lubricant Test Monitoring System (LTMS) document that is maintained by the ASTM Test Monitoring Center (TMC) and is governed by the D02.B0.07 Volatility Surveillance Panel. LTMS severity adjustments shall be applied to results used in API licensing of automotive engine oils (22.1.1).

19. Reference Check

19.1 It is recommended that the Noack reference oil check, NCO-12, be run each day of operation and the orifice adjusted if necessary (see manufacturer's manual). If applicable, see 18.5 for the TMC certification process as defined by the LTMS document.

19.2 Allow the unit to warm up with the pump on while preparing the sample. The heater can stay at 175 °C continuously.

19.3 Clean the reaction vessel and stir bar with solvent (hydrocarbon solvents such as cyclohexane, heptane, and so forth) and dry it prior to the next test.

19.4 Clean the lid assembly with solvent and wipe dry prior to the next test. Periodically, run a pipe cleaner through the orifice tube to ensure no blockage or restrictions exist.

19.5 Place a clean reaction vessel with the stir bar on a scale. Record and compare the weight with the previous run. The weight of the empty, clean reaction vessel and stir bar should consistently be within ± 0.05 g. If the comparative weight is within tolerance of the previous weight, tare the scale. If not, check the reaction vessel and/or stir bar for cleanliness. Reweigh, compare, and tare the scale.

19.6 Place $65 \text{ g} \pm 0.02 \text{ g}$ of reference oil in the reaction vessel. The stir bar magnet may affect some balances.

NOTE 8—It is recommended to use the stopper (supplied with the instrument) to minimize any interference between the magnet and the balance.

19.7 Place the lid assembly in the reaction vessel and close by tightening the lid retaining ring.

19.7.1 If measuring the percent volatiles collected, follow the manufacturer's recommendations.

19.8 Check the system temperature. To start the test, the system temperature must be between 170 °C and 180 °C.

19.9 Place the reaction vessel assembly on top of the unit and insert the sample thermocouple.

19.10 Perform a leak check of the reaction vessel assembly as per the manufacturer's instructions.

19.11 Remove the heat-resistant stopper, turn the vacuum switch "ON," place the reaction vessel assembly in the heater assembly, and connect the reaction vessel lid tubing to the system connector.

19.12 Start the reference run using the controller.

19.13 When the test is complete, the system will shut down the vacuum pump and the heater (even with the switches still "ON"). In addition, an audible buzzer will sound, test complete light will illuminate, and the test complete screen will be displayed.

19.14 Remove the sample thermocouple. Be careful not to bend the sample thermocouple during removal.

19.15 Disconnect the Teflon lid tubing quick connect.

19.16 Use heat-resistant gloves to remove the reaction vessel assembly.

19.17 Reset the controller.

19.18 Carefully place the reaction vessel assembly in the cooling cup of cold water. Avoid tilting or jostling the reaction vessel assembly during this process so residual oil does not get transferred to the Teflon lid assembly. The water level should be lower than the flange on the reaction vessel assembly to prevent any contamination to the sample.

19.19 Remove the coalescing filter housing assembly. Drain the collected volatiles. Replace the filter element as required. If needed, reassemble coalescing filter housing assembly and replace in the unit.

19.20 Check the pump filter housing assembly to be sure the pump filter it is not wet or yellowed. Never leave liquid in the pump filter housing assembly. Replace the filter as needed.

19.21 Carefully remove the sample thermocouple from the thermocouple holder and clean with a scrub pad.

19.22 The reaction vessel assembly should be cool after approximately 15 min.

19.23 Carefully open the reaction vessel assembly. Slowly move the Teflon lid assembly up to avoid splashing liquid onto the underside of the lid.

NOTE 9—Some condensed volatiles may cling to the Teflon lid assembly. This is not concerning and has been taken into account in the method. However, care should be taken not to splash additional material.

19.24 Place the stopper on the scale, weigh, and record the weight of the reaction vessel and contents.

19.25 Determine the amount of oil volatilized and divide by the initial sample weight to determine the weight loss percentage (see Section A2.8).

19.26 Compare the result to the given value of the reference fluid. If results are within limits, proceed to the sample procedure. If the result is not within the limits of the reference fluid, check that the procedure has been followed and that the apparatus is set up properly with no leaks. Check the calibration of the temperature controller and pressure sensing device.

NOTE 10—Procedures for calibration of the temperature controller and pressure controller can be found in the Operations Manual for the Selby-Noack II Volatility test.

19.27 If no errors in setup or procedure are identified, proceed to changing the size of the orifice tube. The size should be changed in increments of 0.05 mm, with each change corresponding to a directly related change of about 0.3 % evaporation loss. A larger orifice tube increases the volatile value and a smaller orifice tube decreases the volatile value. Rerun the reference fluid after making any changes.

19.28 If problems persist, contact the instrument manufacturer.

19.29 Drain and rinse the reaction vessel, stir bar, and lid assembly and O-ring, and wipe with a clean towel to ensure that both are clean and dry.

19.30 Check the vacuum flow with the inclined manometer assembly once per month or if the daily reference oil value begins to drift.

19.31 Once the system has reached $175\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$, it is now ready for the next run.

20. Sample Procedure

20.1 Perform the steps from Section A2.5 substituting a test sample for the reference fluid.

20.2 Calculate the percent evaporation loss of the test sample to the nearest 0.1 %.

20.3 Collect the volatilized oil (if desired for further analysis) from the coalescing filter.

21. Cleaning

21.1 Clean all of the hardware, with the exception of the reaction flask bottom, with a suitable hydrocarbon solvent (for example, hexane, heptane, cyclo-hexane).

21.2 If the coalescing filter is to be used for collecting volatilized oil, it can be cleaned while disassembled. Unscrew the collection cup, remove the filter cartridge, and clean with a suitable hydrocarbon solvent (for example, hexane, heptane, cyclohexane). Upon reassembly, the filter cartridge can be replaced with a new, clean cartridge. The filter can also be removed from the bracket, if necessary, for cleaning.

21.3 Place 10 mL of a varnish removing solvent into the reaction flask bottom. Insert clean paper towel into the solvent and wipe inside of flask thoroughly, removing any varnish that may be present on the wall. Rinse carefully with hot water and dry.

21.4 If other parts develop a varnish film, clean these with the same procedure as indicated in A2.6.5 or put the parts into a half/half water or full-strength solution of varnish remover overnight.

21.5 With a towel dampened with varnish removing solvent, clean the end of the thermocouple. Wipe with towel dampened with hot water and dry to remove any remaining cleaning solvent or wipe with emery paper.

22. Calculation

22.1 Percent volatility is determined by mass loss found by subtracting the combined weight of the flask bottom and oil after testing from their combined weight before the test.

NOTE 11—The percent volatility is obtained by taking the mass loss of the reaction flask and dividing that by the exact mass of the test oil sample recorded earlier: that is, if weight of the oil sample is 65.1 g and weight of the oil lost is 10.2 g, then $(10.2\text{ g}/65.1\text{ g}) \times 100 = 15.67\%$.

22.1.1 When results are utilized for API licensing of automotive engine oils, a severity adjustment (SA) shall be applied to the final result of Eq 2. The severity adjustment is determined from the TMC calibration procedure, as stated in 18.5.

NOTE 12—When results are utilized for API licensing of automotive engine oils, a severity adjustment (SA) shall be applied to the final result of Eq 3. The severity adjustment is determined from the TMC calibration procedure, as stated in 18.5.

22.2 The mass of volatiles collected is obtained by subtracting the coalescing filter assembly weight before the test from its weight at the end of the test.

NOTE 13—The percent of volatiles collected is obtained by dividing the mass of volatiles collected by the mass loss: that is, if the weight of the empty coalescing filter assembly is 163.2 g and the weight of the filter assembly after volatilization gained 9.8 g, and if the weight loss shown by 22.1 is 10.2 g, then $(9.8\text{ g}/10.2\text{ g}) \times 100 = 96.08\%$ of the volatiles were collected.

$$\text{Evaporation Loss \%} = \frac{[(B - A) - (C - A)]}{(B - A)} \times 100 \quad (9)$$

where:

A = reaction vessel + stir bar weight,
 B = A + sample weight, and
 C = B after 1 h of heating.

23. Report

23.1 Report the evaporation loss to the nearest 0.1 %.

24. Precision⁷

NOTE 14—Procedures B and D were shown to be equivalent in RR:D02-1887 and therefore the same precision is reported as Procedure B.

24.1 *Repeatability*—The quantitative expression for the random error associated with the difference between two independent results obtained under repeatability conditions that would be exceeded with an approximate probability of 5 %, one case in 20, in the normal and correct operation of the test method:

$$\text{Repeatability} = 0.1331X^{0.7443} \quad (10)$$

24.2 *Reproducibility*—A quantitative expression for the random error associated with the difference between two independent results obtained under reproducibility conditions that would be exceeded with an approximate probability of 5 % (one case in 20 in the long run) in the normal and correct operation of the test method:

$$\text{Reproducibility} = 0.2411X^{0.7443} \quad (11)$$

25. Quality Control for Procedures A, B, C, and D

25.1 Procedures A, B, C, and D require confirmation of the performance of the apparatus by analyzing a quality control