Designation: D6375 – 09 (Reapproved 2014)

Standard Test Method for Evaporation Loss of Lubricating Oils by Thermogravimetric Analyzer (TGA) Noack Method[1]

This standard is issued under the fixed designation D6375; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the procedure for determining the Noack evaporation loss of lubricating oils using a thermogravimetric analyzer test (TGA). The test method is applicable to base stocks and fully formulated lubricant oils having a Noack evaporative loss ranging from 0 to 30 mass %. This procedure requires much smaller specimens, and is faster when multiple samples are sequentially analyzed, and safer than the standard Noack method using Wood's metal.

1.2 The evaporative loss determined by this test method is the same as that determined using the standard Noack test methods.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D5800 Test Method for Evaporation Loss of Lubricating Oils by the Noack Method

D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance

D6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

E1582 Practice for Calibration of Temperature Scale for Thermogravimetry

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Noack reference oil*—the oil provided by Noack equipment manufacturers to check proper operation of the Noack evaporation tester.

3.1.2 *Noack reference time*—the time (in minutes) required for the Noack reference oil to reach its known Noack evaporative loss under the conditions used in this test method.

3.1.3 *TGA Noack volatility*—the evaporative loss (in mass percent) of a lubricant as determined in this test method.

4. Summary of Test Method

4.1 A lubricant specimen is placed in an appropriate TGA specimen pan. The pan is placed on the TGA pan holder and quickly heated to between 247 and 249°C under a stream of air, and then held isothermal for an appropriate time. Throughout this process, the TGA monitors and records the mass loss experienced by the specimen due to evaporation. The Noack evaporation loss is subsequently determined from the specimen weight percent loss versus time curve (TG curve) as the mass percent lost by the specimen at the Noack reference time determined under the same TGA conditions.

5. Significance and Use

5.1 This test method is a safe and fast alternative for determination of the Noack evaporation loss of a lubricant.

5.2 The evaporation loss of a lubricant is important in the hot zones of equipment where evaporation of part of the lubricant may increase lubricant consumption.

5.3 Some lubricant specifications cite a maximum allowable evaporative loss.

6. Apparatus

6.1 *Thermogravimetric Analyzer* , with the capability to meet all the conditions required for this test method, along with the software necessary to complete the required analyses.

6.2 *Aluminum Specimen Pan*—This shall be cylindrical, and have a minimum inside diameter/height ratio of 0.45 and a volume of 50 ± 3 µL. If the pans provided by the particular TGA manufacturer do not meet these criteria, alternative pans

[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.

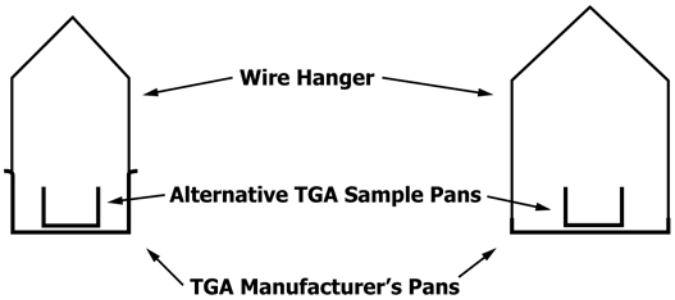

FIG. 1 Examples Showing Adaptation of Alternative Sample Pans

may be used and adapted to fit the pan holder of the TGA. Examples of some of the adaptations used during the evaluation of this test method are shown in Fig. 1.

6.3 *Pressure Regulator,* capable of maintaining air delivery pressure at the level required by the TGA instrument.

6.4 *Flowmeter,* with a flow control valve capable of setting and measuring the air throughput required by the TGA instrument.

7. Reagents and Materials

7.1 *TGA Temperature Calibration Standards*—These materials will depend on the particular TGA apparatus and its capabilities. The TGA manufacturer typically provides them and describes their use in the operating manual for the instrument.

7.2 Compressed air at a pressure suitable for operation of the TGA instrument. Reagent grade air is not necessary but may be used if there are concerns over possible contamination of the internal parts of the TGA.

7.3 *Noack Reference Oil*—Oil having a known Noack evaporative loss, the value of which is provided by the manufacturer.

8. TGA Preparation and Calibration (see Note 1)

NOTE 1—This section only needs to be done if TGA has been idle for an extended period of time, has had significant repairs made to it, or has been mishandled or its location changed.

8.1 Check the temperature correlation between the specimen and control temperatures in accordance with TGA manufacturer's recommendations or Practice E1582. Use calibration standards that will bracket 250°C. When necessary, recalibrate, and regenerate correlation.

8.2 When necessary, burn out the TGA to remove any condensed liquids or deposits, which may have formed on its inside surfaces. Generally, burn out is accomplished by raising the temperature of the TGA to a minimum of 800°C with an air purge from 200 to 500 mL/min, and by maintaining it at this high temperature until no smoke is detected from the TGA gas exhaust tube. Normally 15 to 20 min at these conditions are enough to remove most deposits. (**Warning**—Do not place a specimen pan in the TGA during this operation. It will melt and may damage the balance or furnace mechanisms.)

8.3 Check operation of TGA balance and adjust when necessary. Follow manufacturer's procedure and recommendations.

9. Procedure

9.1 *Determination of Specimen Mass :*

9.1.1 Determine the nominal internal diameter (in centimetres) of the specimen pans by measuring the internal diameter of 10 different pans and averaging the results. A caliper shall be used to make this measurement.

9.1.2 Calculate the specimen mass using following equation:

$$M_s = 350\,(\mathrm{ID})^3 \quad (1)$$

M_s = Specimen mass, mg (round to closest whole mg.)
ID = Nominal inside diameter of specimen pan, cm (see 9.1.1).

9.2 *Air Flowrate*— Set air flowrate to that recommended by the TGA manufacturer or higher if during the initial tests with the Noack reference oil there appears to be condensation on any part of the TGA balance mechanism or furnace lining. Repeat 8.1 with the new flow rate.

9.3 *Temperature Program (see Note 2):*

NOTE 2—This section only needs to be done during the initial set up of the method in the TGA.

9.3.1 Using the correlation from 8.1, determine the final program temperature required to obtain a final specimen temperature of 249°C.

9.3.2 Program the TGA to heat the specimen from 50°C to the final program temperature determined in 9.3.1 at heating rate(s) that will simulate the specimen heating rate of the standard Noack methods (~100°C/min to 220°C and 10°C/min from 220°C to 249°C). Some guidance on how to achieve acceptable heating rates can be obtained from the examples shown in Fig. 2. Maintain the final program temperature for 30 min (see Note 3).

NOTE 3—The 30 min isothermal hold may be adjusted after the Noack reference oil has been tested and the Noack reference time for the instrument has been established (see 9.4). The isothermal hold can then be set to be 2 min longer than the measured Noack reference time.

9.3.3 Tare an empty specimen pan in accordance with the TGA operating manual.

9.3.4 Add the required mass (± 3 mg) (as determined in 9.1) of the Noack reference oil to the tared pan.

9.3.5 Place the pan on the TGA pan holder, and run specimen through the temperature program, as described in 9.3.2.

9.3.6 From the data obtained in 9.3.5, generate a plot of time versus specimen temperature. Determine whether at any time

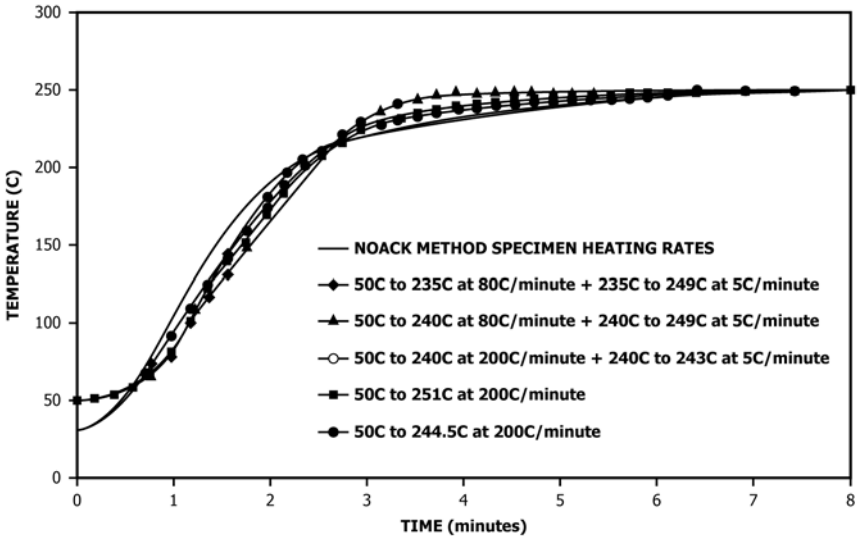

FIG. 2 TGA Noack Programs and Resulting Specimen Heating Rates

the specimen temperature was above 249°C. When this occurs, proceed to 9.3.8. When it does not occur, proceed to 9.3.7.

9.3.7 Temperature **not** over 249°C: Determine the Noack reference time in accordance with 9.4.6. When the Noack reference time is less than 7 min, return to 9.3.2 and the reduce heating rate to extend the Noack reference time beyond 7 min. Check the specimen temperature at the Noack reference time to be sure it is between 248 and 249°C. When it is lower, determine how much lower than 248.5°C and increase the final program temperature by this amount. Go to 9.4.

9.3.8 Temperature **over** 249°C: Modify the TGA temperature program to eliminate the temperature overshoot. This is generally accomplished by splitting the program into two stages, by reducing the heating rate by the final program temperature, or by a combination thereof. An example of how temperature overshoot was eliminated in a particular instrument is shown in Fig. 3. Repeat 9.3.3 – 9.3.6 until an appropriate temperature program has been obtained.

9.4 *Determination of Noack Reference Time:*

NOTE 4—**Important:** This determination shall be completed each day prior to evaluating any test specimens.

9.4.1 Set air flowrate in accordance with 9.2.

9.4.2 Enter final temperature program established in 9.3.

9.4.3 Tare an empty specimen pan in accordance with the TGA operating manual.

9.4.4 Add the required mass (as determined in 9.1) of the Noack reference oil to the tared pan. Whether specimen is added volumetrically or gravimetrically, the actual mass shall be within ±3 mg of the calculated specimen mass. Adjust specimen mass to meet this requirement.

9.4.5 Place the pan on the TGA pan holder, and run specimen.

9.4.6 From the thermogravimetric curve generated in 9.4.5, determine the time (if possible to the closest 0.01 min) required for the Noack reference oil to reach its Noack evaporative loss. This time is the Noack reference time. Record this time, as it will be used in 9.5 to determine the TGA Noack volatility of the test lubricants. An example of a TG curve for the Noack reference oil and how to use it to determine the Noack reference time is shown in Fig. 4 (Curve 1). The isothermal hold of the TGA temperature program can now be modified to the Noack reference time plus 2 min. This will expedite future determinations.

9.4.7 Check that the specimen temperature at the Noack reference time is between 247 and 249°C. When the temperature is outside this range, burn out TGA in accordance with 8.2 and repeat 9.4.

9.4.8 Compare the measured Noack reference time to those measured in prior days. When the difference is more than 10 %, check operation of the TGA in accordance with Section 8. When significant repairs or modifications, such as replacement of the balance mechanisms, temperature sensor, and so forth, have been made to the TGA since the previous time the Noack reference time was measured, the test method shall be repeated starting with Section 8.

9.5 *Determination of TGA Noack Volatility of Test Lubricant:*

9.5.1 Using a new specimen pan, repeat 9.4.1 – 9.4.5 using the test lubricant in place of the Noack reference oil.

9.5.2 Using the TG curve for the test lubricant and the Noack reference time from 9.4.6, determine the mass loss (in mass %) of the test lubricant at the Noack reference time. This is the TGA Noack volatility for the test lubricant. Examples of how to determine the TGA Noack volatility of test lubricants are shown in Fig. 4 (Curves 2 and 3). Check that the specimen temperature at the Noack reference time is between 247 and 250°C. When it is not, reinitiate the test starting with Section 8.

9.5.3 The TGA shall be burned out (see 8.2) on a regular basis. An estimate of how many tests can be done on a particular TGA before it needs to be burned out can be obtained by performing consecutive tests with the Noack reference oil until the difference in the Noack reference time between any of

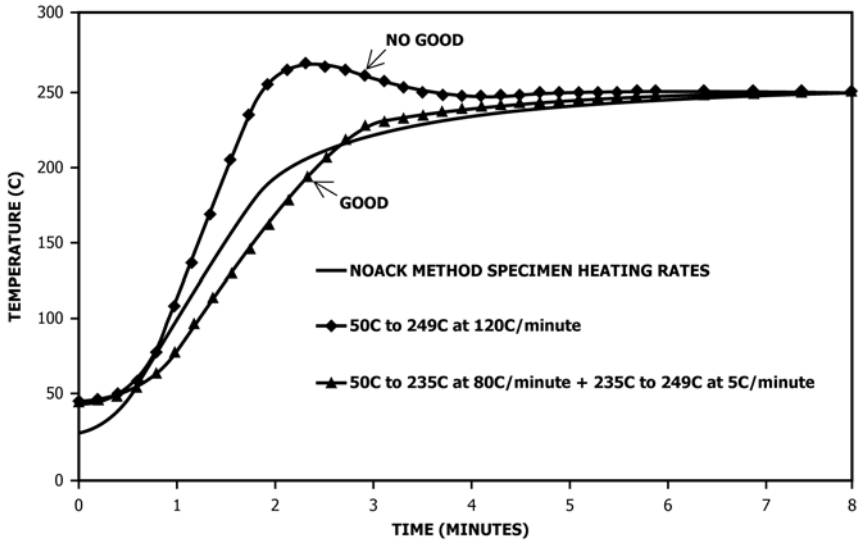

FIG. 3 TGA Noack Programs and Resulting Specimen Heating Rates Showing Modification of TGA Program to Eliminate Overshoot in Specimen Temperature

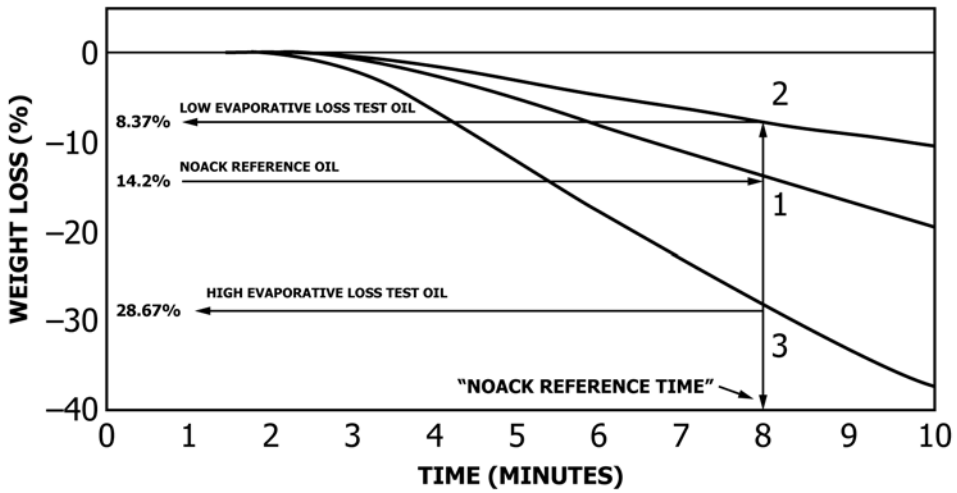

FIG. 4 Determination of Noack Reference Time and Test Oils Evaporative Loss

the determinations is greater than 10 %. The number of tests between burn outs may be increased by operating at a higher air flowrate (see 9.2).

10. Report

10.1 Report the TGA Noack volatility of the test lubricant, as determined in 9.5.2, to the closest 0.01 mass percent.

11. Quality Control (QC)

11.1 Confirm the performance of the instrument or the test procedure by analyzing a QC sample.

11.1.1 If a suitable QC oil or basestock sample is not available, prepare QC sample from a stock of such material.

11.1.2 When QC/Quality Assurance (QA) protocols are already established in the testing facility, they may be used when they confirm the reliability of the test result.

11.1.3 When there is no QC/QA protocol established in the testing facility, Appendix X1 can be used as the QC/QA system.

12. Precision and Bias[3]

12.1 On the basis of an interlaboratory round robin consisting of nine laboratories testing eight oils with TGA instruments from five different manufacturers, the following precision and bias were determined for this procedure.

12.2 *Repeatability*— Two determinations made on the same sample within a short interval of time, by the same operator using the same TGA equipment, in the normal and correct

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1447.

operation of this test method, should differ by more than the following value only in one case in twenty.

$$\text{Repeatability} = 0.31\,(\text{TGA Noack Volatility})^{0.60} \quad (2)$$

12.3 *Reproducibility*— Two determinations made on the same sample by different operators or using different TGA equipment, in the normal and correct operation of this test method, should differ by more than the following value only in one case in twenty.

$$\text{Reproducibility} = 0.39\,(\text{TGA Noack Volatility})^{0.60} \quad (3)$$

12.4 *Bias*—Within the repeatability of this test method, no significant bias was found between the Noack volatility determined by this test method and those determined using Test Method D5800.

13. Keywords

13.1 evaporation loss of lubricants; Noack evaporative loss by TGA; Noack volatility by TGA; TGA Noack volatility; TGA volatility; thermogravimetry; volatility of lubricants

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL (QC)

X1.1 Confirm the performance of the instrument or the test procedure by analyzing a quality control sample.

X1.2 Prior to monitoring the measurement process, the user of this test method needs to determine the average value and control limits of the QC sample. See Practice D6299 and MNL 7.[4]

X1.3 Record the QC results, and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process. See Practice D6299, Guide D6792, and MNL 7.[4]

X1.4 In the absence of explicit requirements given in the test method, the frequency of QC testing is dependent on the criticality of the quality being measured, the demonstrated stability of the testing process, and customer requirements. Generally, a QC sample is analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC frequency may be reduced. The QC sample precision should be checked against the ASTM test method precision to ensure data quality.

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material routinely analyzed. An ample supply of QC material should be available for the intended period of use, and it must be homogenous and stables under the anticipated storage conditions. See Practice D6299, Guide D6792, or MNL 7,[4] or a combination thereof, for further guidance on QC and control charting techniques.

[4] MNL7, *Manual on Presentation of Data Control Chart Analysis*, 6th ed., ASTM International, W. Conshohocken, PA.