Designation: D 6375 – 99a

An American National Standard

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

This standard is issued under the fixed designation D 6375; 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 analysed, 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 value stated in S.I. units are to be regarded as the 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]

D 5800 Test Method for Evaporation Loss of Lubricating Oils by the Noack Method

E 473 Terminology Relating to Thermal Analysis

E 1582 Practice for Calibration of Temperature Scale for Thermogravimetry

2.2 *Other Documents:*

DIN 51-581 Determination of Evaporation Loss of Lubricating Oils[3]

CEC L-40-T-87 Evaporation Loss of Lubricating Oils[4]

JPI-5S-41-93 Determination of Evaporation Loss of Engine Oils (Noack Method)[5]

SAE 962035 The Thermogravimetric Noack Test: A Precise, Safe and Fast Method for Measuring Lubricant Volatility[6]

IP 421 Evaporation Loss of Lubricating Oils[7]

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.

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

[3] Available from Deutsches Institut fur Normunge, Beuth Verlag GmbH, Burggrafenstrasse 6, 1000 Berlin 30, Germany.

[4] Available from CEC, Madou Plaza, 25th floor, Place Madou, B-1210, Brussels, Belgium.

[5] Available from Petroleum Assoc. of Japan, Keidanren No. 90-4, 1 Chrome, Ohtemachi, Chiyoda-Ku, Tokyo 100-0004, Japan.

[6] Available from Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

[7] Available from American National Standards Institute, 11 W. 42nd St., 13th floor, New York, NY 10036.

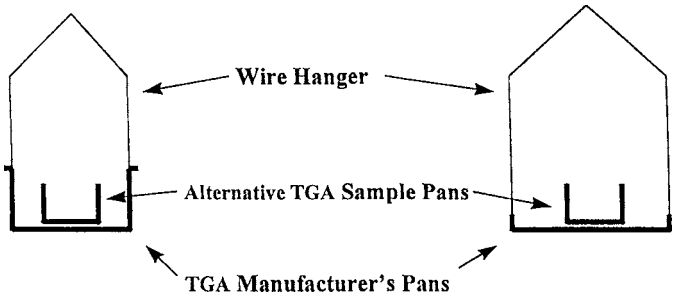

FIG. 1 Examples Showing Adaptation of Alternative Sample Pans

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 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 E 1582. 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. (**Caution:** 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