



Standard Test Method for Volatilization Rates of Lubricants in Vacuum¹

This standard is issued under the fixed designation D2715; 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 determination of the rates of volatilization of lubricants in a thermal-vacuum environment at pressures and temperatures necessary to obtain a measurable rate of evaporation, or evidence of decomposition.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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

[E296 Practice for Ionization Gage Application to Space Simulators](#)

[E297 Methods for Calibrating Ionization Vacuum Gage Tubes](#)³

3. Summary of Test Method

3.1 A known quantity of specimen is placed in a thermal vacuum balance system and the evaporated material is condensed on a cold plate. The weight of the specimen is continually recorded as a function of time for nominal constant surface area.

4. Significance and Use

4.1 This test method provides data for comparison of the evaporation rate of lubricants used in unshielded bearings in the space environment.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.11 on Engineering Sciences of High Performance Fluids and Solids.

Current edition approved May 1, 2007. Published June 2007. Originally approved in 1968. Last previous edition approved in 2002 as D2715 – 92 (2002). DOI: 10.1520/D2715-92R07.

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³ Withdrawn.

5. Apparatus

5.1 *Recording Vacuum Microbalance*, with capacity of 1 g or more, sensitivity of 0.01 mg or less, zero stability of 0.025 mg or less for 8 h with ranges of weight change of 10 mg or more, and 0.1 mg or less, capable of being pumped to 10^{-5} Pa (10^{-7} torr) or less.

5.1.1 When Procedure B for the more volatile samples is used, the vacuum requirement shall be 10^{-2} Pa (10^{-4} torr) or less.

5.2 *Vacuum System*—A pumping system capable of maintaining a starting pressure of 10^{-6} to 10^{-5} Pa (10^{-8} to 10^{-7} torr) (5.1.1). An optically dense baffle system should be used to ensure freedom from back-streaming. A conventional bell jar system with an oil diffusion pump, a mechanical back-up pump, and an optically dense, liquid, nitrogen-cooled baffle has been found satisfactory on the configuration as shown in Fig. 1.

5.3 *Furnace*, with thermocouple indicator, capable of maintaining a constant sample temperature $\pm 3^\circ\text{C}$. All parts of this furnace must be proved to be usable at the highest temperature and vacuum contemplated.

5.4 *Recorder*, capable of recording weight changes continuously with the balance used, to the performance specified in 5.1.

5.5 *Specimen Container*, made of 300 series stainless steel in the form of a straight cylinder with an aspect ratio of height to diameter of approximately 1:14. Where chemical reactions are experienced with the container, alternative materials may be used.

5.6 *Contacting Thermocouple*, touching solid or immersed in liquid specimens, with the leads brought out in such a way as not to influence balance indication.

5.7 *Cold Plate*—A condensing shield cooled with liquid nitrogen to immobilize molecules evaporated from the lubricant which subtends, at least, a 160° arc from the center of the sample.

5.8 *Nude Ionization Gage*, installed as described in Practice E296 and calibrated as described in Methods E297.

5.9 *Optional Supplemental Equipment:*

5.9.1 *Mass Spectrometer*, to identify degassing products and evaporating species.

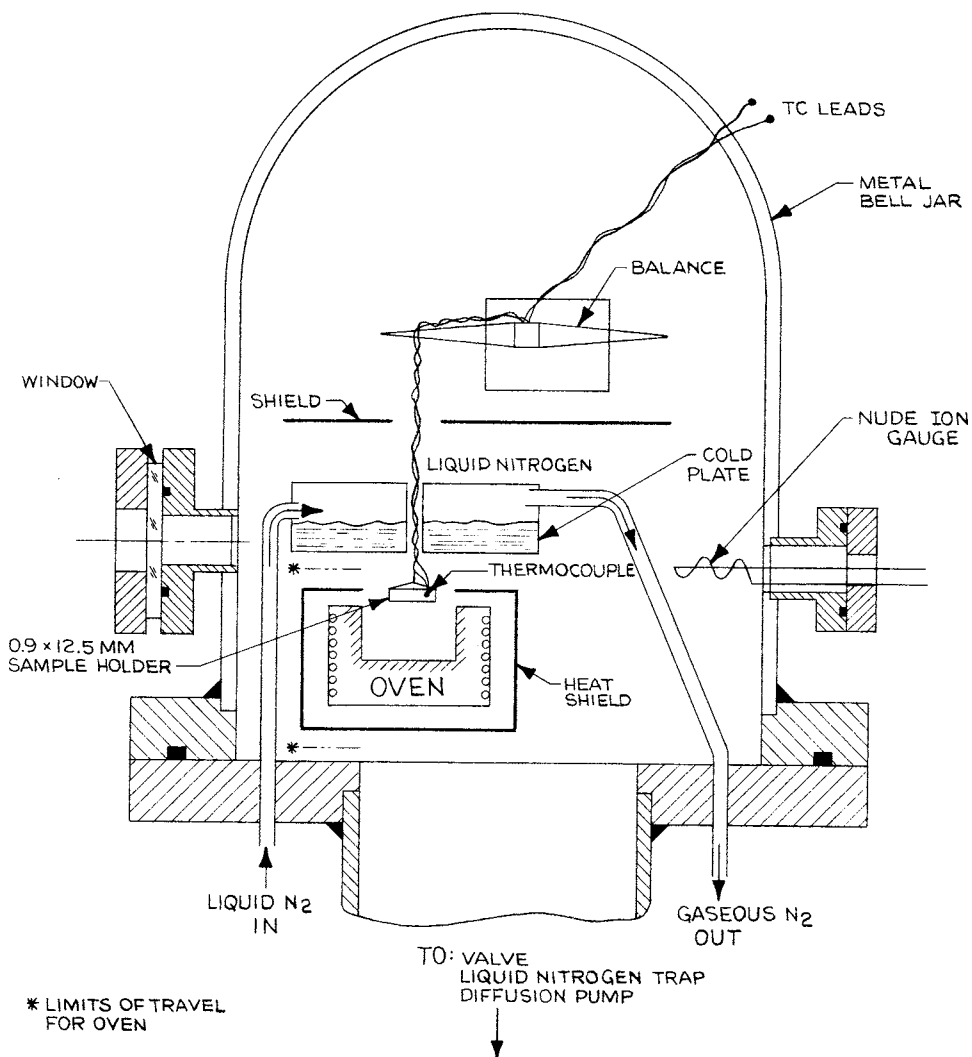


FIG. 1 Apparatus for Measuring Evaporation Rates in Vacuum

5.9.2 *Infrared Optical Pyrometer System*, for determining the specimen temperature. This must be calibrated against the thermocouple for each material used, due to emissivity effects.

5.9.3 *Copper Tab*, on a cold plate facing the specimen, for X-ray analysis of the condensate.

5.9.4 *Noncontact Specimen Thermocouple*, calibrated against 5.5.

5.9.5 *Pressure Recording Pen*, added to the recorder.

5.9.6 *Time Derivative Computer*, to report the rate directly.

6. Reagents and Materials

6.1 *Antiwetting Agent*—A low-surface tension material for coating the specimen container and the thermocouple. Its volatility must be low enough to contribute less than 5 % to the evaporation rate of any sample to be tested.

6.2 *Calibration Material*—Pure compound of suitable physical properties to simulate the lubricant under investigation. (*N*-heptadecane and *bis m*-(*m*-phenoxyphenoxy) phenyl ether have been found satisfactory. Tin provides a low evapo-

ration rate material, the performance of which can be checked by the Langmuir equation.)⁴

6.3 *Liquid Nitrogen*, commercial grade.

6.4 *Helium*, ACS purified grade.

7. Specimen Preparation

7.1 Remove dissolved gases from the bulk lot prior to test using a separate vacuum chamber. Break the vacuum in the chamber with helium. A large enough sample of material should be degassed in this pretreatment so that it will suffice for all anticipated test runs. A mass spectrometer can be used to verify complete degassing.

7.2 If required as evidenced by creepage of lubricant in first run, coat the container and the thermocouple with the antiwetting agent (6.1). Silicones are especially likely to require this precaution.

7.3 Add to the container the required amount of sample, $75 \pm 5 \text{ mg/cm}^2$ of area exposed for evaporation. Press solids and

⁴ Freundlich, M. M., "Microbalance for Measuring Evaporation Rates in Vacuum," *Vacuum*, Vol 14, 1963, pp. 293–297.

semisolids into the container with sufficient pressure to assure the apparent surface area approximates the real surface area. If a coherent surface cannot be achieved, note this fact in the report.

8. System Calibration

8.1 Calibrate the system in the vacuum, using one of the calibration materials, over the temperature range to be used, following the procedure shown in 9.1-9.8.

8.2 The rates obtained are compared with those predicted by the Langmuir equation:⁵

$$G = 7.77p\sqrt{MT} \quad (1)$$

where:

G = evaporation rate, g/cm²·s,

p = vapor pressure, Pa,

M = molecular weight, and

T = temperature, K.

8.2.1 If the measured rates differ by more than ±20 % from those calculated, take all possible corrective steps to locate the source of the discrepancy. Use of a calibration factor is not encouraged, but may be tolerated in some cases if so reported. A factor greater than 2 or less than 0.5 casts such doubt on the results as to practically invalidate them and require corrective action.

9. Procedure A

9.1 Immerse the thermocouple in the sample, and bring the furnace to approximate operating temperature.

9.2 Suspend the sample and the container in position over the furnace, and tare to near the upper limit of the range.

9.3 Assemble the vacuum apparatus and pump the system to give a chamber pressure of 10⁻⁶ to 10⁻⁵ Pa (10⁻⁸ to 10⁻⁷ torr).

9.4 Start the liquid nitrogen flowing and cool the cold plate to 143 K (-200°F) or lower. Stabilize the furnace temperature.

9.5 Measure the pressure near the furnace position with the nude ionization gage.

9.6 Move the furnace into operating position surrounding the specimen. Start the recorder, and mark the recorder chart *start of heat*.

9.7 Hold the temperature constant at the required level for sufficient length of time to measure the rate of weight change and determine constancy of this rate.

NOTE 1—A time derivative computer may be used to report rate of weight change directly.

9.8 Monitor pressure changes manually or by the second pen on the recorder when available. When the test temperature is reached, and a steady weight loss condition attained, establish the sample weight and measure the evaporation rate for this percentage point of the original weight. If the material has uniform molecular weight throughout, the rate will not change with progressing evaporation. If the rate changes, continue measurement until the time for a single rate determination exceeds 3 h.

9.9 Determine rates for several temperatures, using a fresh sample for each determination. Temperature intervals of 25 K, which approximate a ten-fold increase in rate, are usually suitable.

NOTE 2—If the sample is known to be an essentially pure compound, repetitive measurements are permissible. If such purity is merely suspected, judgment may be made on the basis that a sample is not to be reused after a determination in the course of which the rate has changed more than 25 % at a single temperature. However, if the supply is limited, it is possible to obtain some meaningful data on a spot basis, as indicated below.

9.10 After primary data have been obtained at increasing temperature levels on a sample which meets the above criterion of less than 25 % change during any single measurement, make spot measurements at decreasing temperature levels to detect any changes in the specimen.

10. Procedure B

10.1 Immerse the thermocouple, suspend the sample, and position the furnace as described in 9.1-9.3.

10.2 Assemble the vacuum apparatus and pump the system to give a chamber pressure of 10⁻³ to 10⁻² Pa (10⁻⁵ to 10⁻⁴ torr).

10.3 Conduct the rest of the test as described in 9.4-9.10.

11. Calculations

11.1 When the evaporation rate proves to be constant within the limit of a 25 % decrease during a determination, or 25 %/h if the determination takes less than 1 h, the evaporation rate for each temperature is as follows:

$$R = (w_0 - w_1)/(t_1 - t_0) \quad (2)$$

where:

R = evaporation rate, g/s,

w_1 = weight of sample at the end of the test, g,

w_0 = initial weight of the sample, g,

t_1 = time at the end of the test, s, and

t_0 = initial time of the test, s.

11.2 If the sample has a changing rate, this rate is calculated for each of the standard degrees of evaporation required in 12.2 as follows:

11.2.1 The weight required at each evaporation level is:

$$w_r = (100w_0 - Ew_0)/100 \quad (3)$$

where:

w_r = weight at specified evaporation loss, g,

w_0 = initial weight of sample, g, and

E = evaporation loss, %.

11.2.2 Draw a line tangent to the curve on the recorder chart at each weight corresponding to the evaporation loss from 11.2.1 and calculate the evaporation rate as follows:

$$R = (w_a - w_b)/(t_b - t_a) \quad (4)$$

where:

R = evaporation rate, g/s,

w_a = weight at one point on the tangent line, g,

w_b = weight at a second point on the tangent line, g,

t_a = time at a point on the tangent line corresponding to w_a , s, and

⁵ Buckley, D. H., and Johnson, R. L., "Evaporation Rates for Various Organic and Solid Lubricants in Vacuum to 10⁻⁸ Millimetres of Mercury at 55 to 1100°F," *National Aeronautics and Space Administration Technical Note D-2081*, 1963.

t_b = time at a point on the tangent line corresponding to w_b , s,

11.3 The evaporation rate per unit area is:

$$E = (R/A)C \quad (5)$$

where:

E = evaporation rate per unit area, g/cm²·s,

R = evaporation rate from 11.1 or 11.2, g/s,

A = surface area of sample exposed for evaporation, cm², and

C = calibration factor from 8.2, if applicable.

11.4 If the molecular weight of the sample is known, the rates may be converted to vapor pressures by the equation given in 8.2. As the molecular weight enters as square root, the allowable error is twice that for the vapor pressure.

12. Report

12.1 For specimens of constant rate according to 11.1, the report shall consist of the evaporation rate per unit area for each temperature, plus a statement of any deviations in coherence of surface as in 7.3, or variation in chamber pressure beyond the limits in 9.3, or decomposition found in 9.10.

12.2 For specimens of variable rate according to 11.2, the report shall consist of the evaporation rate per unit area for each temperature at intervals of 5 % (based on the sample weight in 7.3) from the first obtainable one as far as the data go but not to exceed a running time of 3 h unless this is specifically required. Any deviations (see 12.1) are to be reported.

12.3 For specimens of variable rate, and such limited supply as to require reuse at another temperature, the report will contain the data which could be obtained. For example, such a report might indicate:

5 %, 10 % measured at 473 K,
 15 %, 20 % measured at 498 K,
 25 %, 30 %, 35 % measured at 523 K,
 40 %, 45 %, 50 % remeasured at 498 K.

13. Precision and Bias

13.1 The data shown in Fig. 3 of the paper⁵ were used to prepare the following statement on Procedure A. Cooperative testing to prepare a statement on Procedure B is being planned.

13.1.1 *Repeatability*—Duplicate results by the same operator should be considered suspect if they differ by more than 45 % of their mean value (95 % confidence level).

13.1.2 *Reproducibility*—There is no immediate plan to determine the data necessary to develop the reproducibility statement.

13.2 *Bias*—No general statement is made on bias for this standard since the data used to determine the correlation cannot be compared with accepted reference material.

14. Keywords

14.1 lubricants; volatilization; volatilization rates

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