



## Standard Test Method for Volatilization Rates of Lubricants in Vacuum<sup>1</sup>

This standard is issued under the fixed designation D 2715; 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 ( $\epsilon$ ) 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:

E 296 Practice for Ionization Gage Application to Space Simulators<sup>2</sup>

E 297 Method for Calibrating Ionization Vacuum Gage Tubes<sup>3</sup>

### 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.

### 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.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.11 on Engineering Sciences of High Performance Fluids and Solids.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.03.

<sup>3</sup> Discontinued; see 1985 *Annual Book of ASTM Standards*, Vol 15.03.

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  $160^\circ$  arc from the center of the sample.

5.8 *Nude Ionization Gage*, installed as described in ASTM Practice E 296 and calibrated as described in Method E 297.

#### 5.9 *Optional Supplemental Equipment:*

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

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

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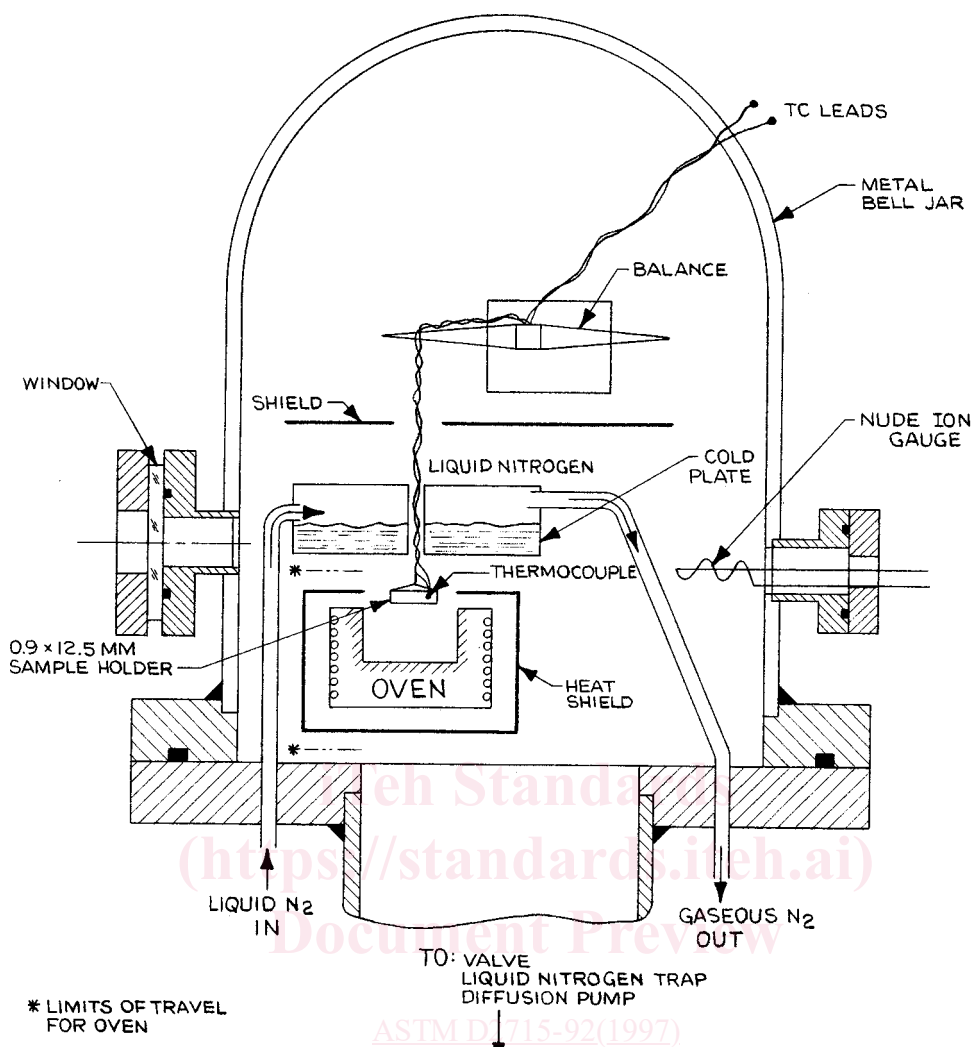


FIG. 1 Apparatus for Measuring Evaporation Rates in Vacuum

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 evaporation rate material, the performance of which can be checked by the Langmuir equation.)<sup>4</sup>

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 anti-wetting 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 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:<sup>5</sup>

$$G = 7.77p\sqrt{M/T} \quad (1)$$

<sup>4</sup> Freundlich, M. M., "Microbalance for Measuring Evaporation Rates in Vacuum," *Vacuum*, Vol 14, 1963, pp. 293-297.

<sup>5</sup> 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.