



## Standard Test Method for Hermeticity of Sealed Devices by a Radioisotope Test<sup>1</sup>

This standard is issued under the fixed designation F 785; 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.

<sup>ε1</sup> NOTE—Added Test Method F 866 and renumbered Sections 3, 4, and 5, editorially in July 1991.

### 1. Scope

1.1 This test method covers procedures for detecting leaks in sealed devices by means of a radioactive tracer gas.

1.2 This test may be conducted on any sealed device having an internal cavity that either contains gas or is evacuated.

1.3 The minimum detectable leak size depends upon the amount of activation, while the maximum detectable leak size depends upon the quantity of radioactive gas initially accumulated within the cavity and its subsequent rate of escape. In normal applications this test can detect leak sizes greater than  $1 \times 10^{-8}$  atm·cm<sup>3</sup>/s ( $\sim 1 \times 10^{-9}$  Pa·m<sup>3</sup>/s), but leak sizes smaller than  $1 \times 10^{-8}$  atm·cm<sup>3</sup>/s can be detected by prolonged activation. The detection of leaks greater than  $1 \times 10^{-5}$  atm·cm<sup>3</sup>/s ( $\sim 1 \times 10^{-6}$  Pa·m<sup>3</sup>/s) is affected by the cavity volume available for gas collection and by internal absorbent or gettering materials.

1.4 The range and precision of measurement can be increased by performing the test in multiple steps with a sequential increase in activation at each step and minimum delay between activation and detection. Such a sequential procedure which starts with small activation for the larger leak sizes also prevents excessive radioactivity in large volume devices.

1.5 This test method can be applied to single device or batch testing but is particularly effective for 100 % screening of large batches.

1.6 Other methods for determining hermeticity may be found in Practices F 97, F 98, and Test Methods F 134.

1.7 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazards see Section 9.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- E 425 Definitions of Terms Relating to Leak Testing<sup>2</sup>
- F 97 Practices for Determining Hermeticity of Electron Devices by Dye Penetration<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.09 on Hybrid Microelectronics.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 03.03.

<sup>3</sup> Annual Book of ASTM Standards, Vol 10.04.

F 98 Practices for Determining Hermeticity of Electron Devices by a Bubble Test<sup>3</sup>

F 134 Test Methods for Determining Hermeticity of Electron Devices with a Helium Mass Spectrometer Leak Detector<sup>3</sup>

F 784 Method for Calibrating Radioisotope Hermetic Test Apparatus<sup>3</sup>

F 866 Test Method for Measuring the Package Attenuation Coefficient of a Sealed Device for Radioisotope Hermetic Test<sup>3</sup>

### 3. Terminology

#### 3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *standard leak rate or leak size*—the rate of flow of dry air of dew point less than  $-25^{\circ}\text{C}$  through a leak under standard conditions specified as follows: (1) the inlet pressure shall be 1 standard atmosphere  $\pm 5\%$  ( $101 \pm 5$  kPa), (2) the outlet pressure shall be less than 1 kPa (0.01 atm), and (3) the temperature shall be  $23 \pm 3^{\circ}\text{C}$ .

3.1.2 *measured leak rate,  $Q$* —the leak rate of a given package as measured under specified conditions and employing a specified test medium. Usually, leak rates measured under different conditions or by different methods cannot be compared unless flow mechanisms are assumed and corresponding leak sizes calculated.

3.1.3 *minimum detectable leak rate,  $Q_{\min}$* —that value calculated from Eq 1 (see 13.1) for given values of pressurization and pressurization time and for a specified minimum detectable count rate.

3.1.4 *minimum detectable count rate,  $R_{\min}$* —the smallest unambiguous count rate above background that can be derived from the particular counting station at the test site at the time of use.

3.1.5 *background signal*—the smallest unambiguous count rate that can be assigned to the particular radiation counter at the test site at the time of use with an empty, shielded detector. It is the sum of the averaged base signal and drift.

3.1.6 *maximum acceptable leak size,  $L_A$* —the largest value of leak size that can be tolerated in parts deemed acceptable with respect to hermeticity. This value must be mutually agreed upon between parties to the test.

3.1.7 *specific activity,  $s$* —the amount of radioactivity per unit quantity of test gas.

3.1.8 *counting efficiency or  $k$ -factor*—the measured count rate per unit quantity of radioactivity from within the device for a specific counting station and position within the crystal detector.

3.1.9 *package attenuation coefficient, C*—numerical value for the fractional decrease in gamma radiation from the entrained tracer gas due to the device enclosure. This is a property of the package and device type only.

3.1.10 *counter sensitivity, G*—the measured count rate per unit quantity of radioactivity. This is a characteristic of the individual counting station and position within the crystal detector.

3.1.11 *pump-down time*—the interval of time required to evacuate the activation chamber from the selected activation pressure down to a specified low pressure at which the tank is backfilled with air and vented to atmosphere.

3.1.12 *dwel time*—the interval of time between the removal of activation pressure and the measurement of package radioactivity.

3.1.13 For other definitions relating to leak testing see Definitions E 425.

3.2 *Units:*

3.2.1 *Pressure:*

3.2.1.1 1 Pa = 1 kg·m/s<sup>2</sup>·m<sup>2</sup> = 0.0075 torr = 1.451 × 10<sup>-4</sup> psi

3.2.1.2 1 standard atm = 1.01325 × 10<sup>5</sup> Pa

3.2.1.3 1 torr = 1/760 standard atm = 133.322 Pa

3.2.2 *Leak Rate:* 1 Pa·m<sup>3</sup>/s = 9.869 atm·cm<sup>3</sup>/s

3.2.3 *Radioactivity:* 1 Ci = 3.7 × 10<sup>10</sup> Bq (s<sup>-1</sup>)

3.2.4 *Reference Temperature Range:* 23 ± 3°C (see 5.1.1)

3.2.5 *Specific Activity:* μCi/atm·cm<sup>3</sup>

3.2.6 *Counting Efficiency:* counts per minute per μCi.

4. Summary of Test Method

4.1 In this test a gas mixture of nitrogen containing a small concentration of <sup>85</sup>Kr is used to pressurize sealed devices, thus allowing a quantity of <sup>85</sup>Kr to enter any nonhermetic device.

4.2 The gamma radiation from the disintegrating <sup>85</sup>Kr molecules that have entered the defective device are measured with a scintillation crystal-equipped detection system. Normally the beta radiation from the disintegrating <sup>85</sup>Kr gas is attenuated by the wall of the device, thus allowing the gamma radiation from within the device to be equated to the number of molecules of <sup>85</sup>Kr that entered the device. To prevent false rejection due to <sup>85</sup>Kr gas adsorbed externally on the device, a surface adsorption measurement should be made, using either a Geiger-Mueller tube or scintillation crystal.

4.3 The correlation between leak rate and detector response is calculated as a function of the pressurization and the time of pressurization in the test gas mixture of given radioactivity. See Appendix X1 for further details.

5. Significance and Use

5.1 This test method provides a quantitative measurement procedure for determining leak rates in small sealed packages such as are used with electron devices. Interior package volumes may range from less than 1 × 10<sup>-3</sup> cm<sup>3</sup> to 10 cm<sup>3</sup> or more. The method may be applied on a go-no-go basis as a screening process capable of examining thousands of components per hour.

5.2 Contaminants that reduce the effective life of the device may enter the cavity of the device through a leak. These leaks are found at seal points between dissimilar

materials or are due to porosity of a defective portion of the package.

5.3 The maximum leak size that may be allowed is a function of the package cavity volume, the nature of the device, and the expected lifetime in the intended environment. The value is usually listed in acceptance specifications as evolved from operational experience and test capability.

6. Interferences

6.1 Leak testing with radioactive gas following moisture-type environmental tests or any liquid immersion is unsatisfactory since small leaks may become clogged or the leak rate may be reduced by the liquid. For this reason wet gross-leak tests must follow the radioisotope hermetic test.

6.2 Organic materials such as plastics, inks, labels, etc., on the surface of parts will cause erroneous readings when parts are tested according to this method because of <sup>85</sup>Kr absorption in such materials unless the appropriate precautions are used.

6.3 Entrapment of <sup>85</sup>Kr in surface coatings, in minute pin-hole voids in solder surfaces, or in surface porosities may produce false readings when the part is tested according to this method.

7. Apparatus

7.1 *Gas Pressurization System* (or activation unit), comprised of an activation tank, transfer system, storage tank, and control system as indicated in the schematic of Fig. 1. The system should be capable of safely handling radioactive <sup>85</sup>Kr with provision for evacuation of the activation chamber to 0.5 torr (67 Pa), of transferring the <sup>85</sup>Kr-nitrogen gas from the storage tank into the activation tank at pressures greater than 2 atm (203 kPa), and of returning the gas mixture to the storage tank within 2 min. The unit shall have been calibrated in accordance with Method F 784.

7.2 *Counting Station*, comprised of a scintillation crystal of the thallium-activated sodium iodide or equivalent type, a photomultiplier tube, power supply, preamplifier, and ratemeter as indicated in the schematic of Fig. 2. In order to maximize sensitivity, a well-type crystal should be used with as deep a well as possible. The ratemeter system should have a precision of 2%. A surface or beta radiation counter may also be available. This shall have been calibrated in accordance with Method F 784.

8. Materials

8.1 A mixture of <sup>85</sup>Kr and nitrogen gas of known specific

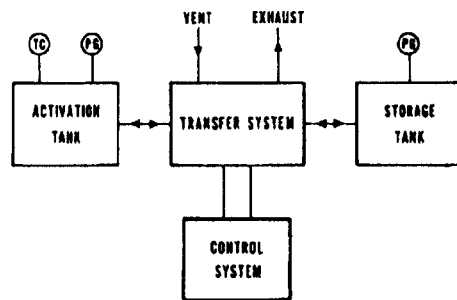


FIG. 1 Activation Unit

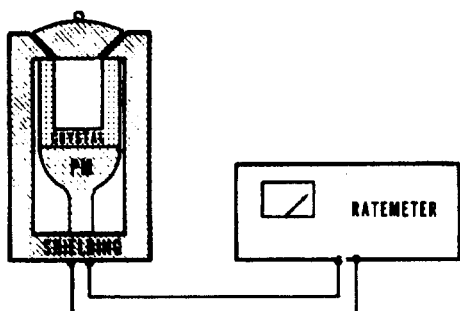


FIG. 2 Counting Station

activity. A minimum concentration of  $100 \mu\text{Ci}/\text{atm}\cdot\text{cm}^3$  ( $365 \times 10^5 \text{ Bq}/\text{Pa}\cdot\text{m}^3$ ) is required.

### 9. Hazards

9.1 *Radioactivity*—Both Federal and some state and local governmental regulations require a license for the possession and use of  $^{85}\text{Kr}$  leak test equipment. In the use of radioactive gas these regulations and the maximum permissible exposure and tolerance levels prescribed by law shall be observed.

9.2 Large-volume, large-leak devices may have excessive radiation levels after testing and must be deactivated prior to removal from the controlled access test area or appropriately marked as required by law, or both.

9.3 The structural limits of many types of packages are readily exceeded by the pressurization capability of the activation unit and by some radioisotope hermetic test specifications. Attention should be given to this factor in selecting activation parameters for any given package type.

### 10. Sampling

10.1 This test method may be applied on either a sample or 100 % basis. The quantity of small-cavity devices pressurized at one time should be limited to the number that can be checked at the counting station within the dwell time specified and not to exceed 1 h.

### 11. Test Specimen

11.1 The package attenuation coefficient shall be determined in accordance with Test Method F 866, or alternatively, the counting efficiency shall be measured for the specific counting station and position within the detector in accordance with Test Method F 866 just prior to the hermetic test procedure.

### 12. Conditioning

12.1 Inspect all devices for count rate. If any exhibit values much greater than 1000 counts per minute above counting-station background, they may be deactivated in vacuum or in a vacuum oven until the count-rate has diminished to a suitable value.

12.2 In cases where retest is required at some time following liquid immersion, determine the surface activity and deactivate the devices by allowing to stand in air or vacuum-oven until beta radiation is undetectable or two successive gamma count rates differ by less than 5 % over a 10-min interval.

### 13. Procedure

13.1 *Activation Parameters*—Determine activation pressure and soak time as follows:

$$Q_{\min} = (R_{\min} P_0^2) / (skT (P_e^2 - P_i^2)t) \quad (1)$$

where:

$Q_{\min}$  = minimum detectable leak rate specified,  $\text{atm}\cdot\text{cm}^3/\text{s}$ .

$R_{\min}$  = minimum detectable count rate above background selected, counts per minute. This is the reject count above the background of both the counting equipment and the component.  $R_{\min}$  shall be  $\geq 1000$  counts per minute.

$s$  = specific activity,  $\mu\text{Ci}/\text{atm}\cdot\text{cm}^3$ .

$k$  = counting efficiency, counts per minute per  $\mu\text{Ci}$ .

$T$  = soak time that the devices are to be activated, h.

$P_0$  = 1 standard atmosphere.

$P_e$  = activation pressure, atmospheres absolute.

$P_i$  = original internal pressure of the devices, atmospheres absolute.

$t$  = conversion factor, 3600 s/h.

The activation pressure ( $P_e$ ) may be established by specification, or if a convenient soak time ( $T$ ) has been established, the activation pressure ( $P_e$ ) can be adjusted to satisfy Eq 1.

13.2 *Counting Efficiency,  $k$* —Calculate the counting efficiency as follows:

$$k = C \times G \quad (2)$$

where:

$C$  = package attenuation coefficient, dimensionless quantity, and

$G$  = counter sensitivity, counts per minute per  $\mu\text{Ci}$ .

13.3 *Background Signal*—The count rate is normally 600 to 800 counts per minute with the shield plug in place and greater without the plug.

13.3.1 Observe the ratemeter and record its indication for a 5-min period as follows. Do not readjust the controls (gain, voltage) for the duration of this procedure.

13.3.2 Record the pointer deflection, in scale divisions, at  $T = 0$  min.

13.3.3 Record the minimum and maximum pointer deflections occurring in the interval from  $T = 0$  to  $T = 1$  min.

13.3.4 In like manner record the minimum and maximum deflections for each minute interval to  $T = 5$  min.

13.3.5 Record the deflection occurring at  $T = 5$  min.

13.3.6 Average the minimum and maximum values of pointer deflections together.

13.3.7 Subtract the values for pointer deflections at  $T = 5$  min and at  $T = 0$  min. Divide the difference by 5. This is the drift.

13.3.8 Add the average value of pointer deflections and the drift. This shall be the background signal.

13.4 *Evaluation of Surface Sorption*—Evaluate all device configurations consisting of glass, metal, and ceramic, or combinations thereof, including coatings and external sealants, for surface sorption of  $^{85}\text{Kr}$  before establishing the leak test parameters.

13.4.1 Select a number of representative specimens of the devices to be tested.

13.4.2 Measure the background count rate for each specimen.

13.4.3 Activate the specimens with the pressure and soak time values selected in 13.1.

13.4.4 Record the time at which the activation tank is vented.

13.4.5 Remove the specimens from the activation tank and measure the count rate (beta or gamma) for each specimen individually.

13.4.6 Record the time of measurement for each specimen.

13.4.7 Repeat the measurement of count rate and record the time of measurement for each specimen periodically until for each specimen the beta radiation is constant to within 5 % or the net gamma count rate above background is  $\leq 0.5 R_{\min}$  as selected in 13.1.

13.4.8 Determine the lapsed time required from the time of venting for each specimen for the condition of 13.4.7 to be established.

13.4.9 The shortest value of lapsed time so obtained shall be the minimum usable dwell time.

13.4.10 Alternatively, open representative specimens of the devices and subject all parts of each device as a unit to steps 13.4.2 through 13.4.8. The averaged values of the lapsed times so obtained shall be the minimum usable dwell time.

13.4.11 If the minimum usable dwell time exceeds 1 h, a larger value of  $R_{\min}$ , and consequent pressurization parameters, may be selected. It is noted that sorption may vary with pressure and time of exposure so that some trial may be required before satisfactory exposure values are obtained.

#### 13.5 Hermetic Test:

13.5.1 Determine background count rate for each device to be tested. Where no observable effect on background is obtained, use the value from 13.3.

13.5.2 Place the devices in the activation tank with any free volume in the tank reduced with solid filler blocks to minimize loss of  $^{85}\text{Kr}$  on venting and to minimize chamber pump down time.

13.5.3 With the value of counting efficiency calculated in 13.2, calculate the activation pressure and time in accordance with 13.2 and 13.4.

13.5.4 Program the activation unit for the calculated exposure pressure  $P_e$  and time  $T$ .

13.5.6 Start the activation unit and evacuate the activation tank to 0.5 torr (67 Pa).

13.5.7 Expose the devices to the prescribed activation conditions.

13.5.8 Return the test gas to storage, achieving a vacuum of 0.5 torr (67 Pa) in the chamber.

NOTE 1—When the minimum detectable leak rate selected is  $\geq 1 \times 10^{-6}$  torr (133  $\mu\text{Pa}$ ), the chamber may be evacuated to 2.0 torr (267 Pa) to reduce resultant dwell time and, hence, to minimize gas escape from leaking devices.

13.5.9 When the activation tank has been vented, remove the devices, and transfer to the counting station after the appropriate minimum usable dwell time has elapsed according to 13.4, measure, and record.

13.5.10 Separate out those devices which give a count rate above background  $\geq R_{\min}$ .

13.5.11 Rejected devices may be reexamined for surface adsorption in the manner of 13.4 to verify that they are truly leakers.

13.5.12 With the exception of cavity-free volumes  $< 1 \times 10^{-3} \text{ cm}^3$  and leaks  $\geq 1 \times 10^{-5} \text{ atm} \cdot \text{cm}^3/\text{s}$ , a dwell time of up to 1 h may be used.

13.5.13 Calculate the leak rate value as follows:

$$L = Q_{\min}(R)/(R_{\min}) \quad (3)$$

where:

$L$  = leak size of the test device,  $\text{atm} \cdot \text{cm}^3/\text{s}$ , and

$R$  = net count rate of the test device, counts per minute.

13.5.14 Steps 13.5.2 through 13.5.13 may then be repeated with greater activation pressure or longer time, or both, to obtain a smaller minimum detectable leak rate for those devices which evidenced net count rates of approximately or less than  $R_{\min}$ .

## 14. Deactivation of Rejects

14.1 Reject devices contain  $^{85}\text{Kr}$ , and exert control over their release. Generally, 1  $\mu\text{Ci}$  or more of  $^{85}\text{Kr}$  is of concern. For reference a standard type of electronic component will read 15 000 counts per minute for 1  $\mu\text{Ci}$ . Since 1000 counts per minute is usually used as reject level, very few devices in the fine leak rate range will exceed this. Those which do are usually larger leaks and will deactivate overnight in vacuum, or, if gross leakers, in a few hours standing.

14.2 Devices that are to be retested may be deactivated in vacuum or in a vacuum oven until the count-rate has diminished to a suitable value. Before retest, however, record any residual reading as a background value for that part (see 13.5.2).

## 15. Report

15.1 For later reference or for purposes of comparison, the following data should be reported:

15.1.1 Part type.

15.1.2 Date of test.

15.1.3 Date of equipment calibration.

15.1.4 Specific activity.

15.1.5 Counting efficiency.

15.1.5.1 Package attenuation coefficient.

15.1.5.2 Counter sensitivity.

15.1.6 Leak test:

15.1.6.1 background count rate, station.

15.1.6.2 minimum detectable leak size,  $Q_{\min}$ .

15.1.6.3 minimum detectable count rate,  $R_{\min}$ .

15.1.6.4 activation pressure,  $P_e$  (true).

15.1.6.5 activation time,  $T$ .

15.1.6.6 minimum usable dwell time.

15.1.6.7 initial count rate, each part, before activation.

15.1.6.8 indicated count rate, each part.

15.1.6.9 corrected net count rate,  $R$ .

15.1.6.10 dwell time.

15.1.6.11 calculated leak size (Eq 3).

## 16. Precision

16.1 A round-robin evaluation of the precision of this method was carried out on 100 specimens of a 14-lead

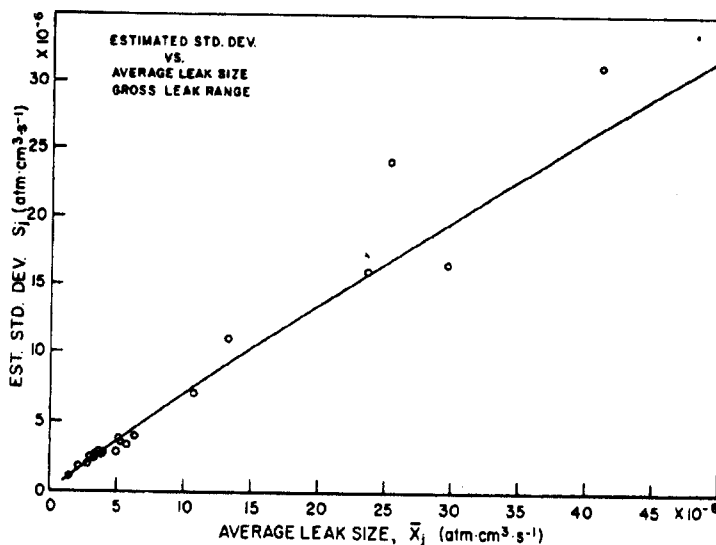


FIG. 3 Estimated Standard Deviations  $S_j$  of Measured Values in the Gross Leak Range with a Least Squares Fitted Curve

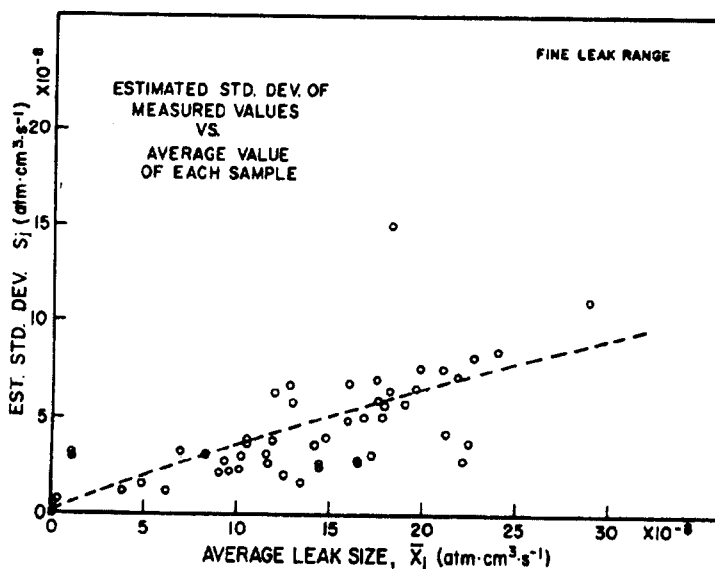


FIG. 4 Estimated Standard Deviations  $S_j$  of Measured Values in the Fine Leak Range with a Least Squares Fitted Curve

ceramic-glass dual-in-line package device of 0.02-cm<sup>3</sup> internal free volume.

16.2 The test sequence was done in two steps. The first step was for a minimum detectable leak rate,  $Q_{min}$ , of  $1 \times 10^{-6}$  atm·cm<sup>3</sup>/s; the second was with increased activation for a  $Q_{min}$  of  $1 \times 10^{-8}$  atm·cm<sup>3</sup>/s.

16.3 Typical results are described by the following equation:

$$\log (S_j/X_0) = a_0 + a_1 \log (\bar{X}_j/X_0) \quad (4)$$

where:

$S_j$  = calculated standard deviation,  
 $X_0 = Q_{min}$ , and  
 $\bar{X}_j$  = leak rate.

With  $X_0$  being  $1 \times 10^{-6}$ ,  $a_0 = -0.106$ ,  $a_1 = 0.947$ , and  $r = 0.989$ , where  $r$  is the correlation coefficient. With  $X_0$  being  $1 \times 10^{-8}$ ,  $a_0 = -0.280$ ,  $a_2 = 0.839$ , and  $r = 0.980$ . See Figs. 3 and 4.

16.4 *Limitations*—The precisions listed are relevant to the type of test specimen used. Values would probably be affected by change in package materials and internal free volumes. See Appendix X1 for test details.

APPENDICES

(Nonmandatory Information)

X1. Round-Robin Results

X1.1 *Results*—A round robin was carried out to determine the precision (between-laboratory agreement) of this radioisotope hermetic test method as applied to a representative semiconductor device package with commercially available test equipment. The test sequence employed a minor variation on that prescribed above in order to allow calculation and data manipulation after the experimental data were obtained at ten independent test sites. To reduce the number of variables involved, one specific model of commercially available equipment was used so that all installations were able to follow the identical operational sequence in using their equipment to carry out the prescribed test procedure. The test procedure is, however, compatible with any available test equipment that meets the requirements listed above.

X1.2 *Test Samples*—One hundred specimens of a 14-lead ceramic-glass dual-in-line packaged device were selected by radioisotope test from a production run of several thousands of parts of a commercial integrated circuit with a nominal internal free volume of 0.02 cm<sup>3</sup>. Manufacture of these packages was by normal methods, except that these devices were not subjected to fluid immersion at any time. Selected indicated leak sizes ranged from <1 × 10<sup>-8</sup> atm·cm<sup>3</sup>/s to ~5 × 10<sup>-5</sup> atm·cm<sup>3</sup>/s. The test specimens were serialized at random.

X1.3 *Calibration*—The counting station at each test site was calibrated against the same pulse generator and <sup>85</sup>Kr reference standards. Each pressure gage, of the Bourdon type, was calibrated at atmospheric pressure against a mechanical reference gage included in each console, which reference gage was characterized by an accuracy of better than 1 %. Each vacuum gage, a thermocouple type with readable scale of 0 to 20 torr (0 to 2666 Pa), was calibrated against a similar transfer thermocouple gage, which in turn had been compared against a precision liquid manometer with an uncertainty limited only by the readability of the gage scale. Repeated calibrations of the transfer thermocouple gage before and after the round robin showed agreement of within 1 %.

X1.4 *Specific Activity*—At each site the sample vial was filled to an indicated pressure of approximately 2.0 torr (267 Pa). The specific activity was determined from the value of the indicated pressure and was used to calculate the activation parameters for a threshold count rate  $R_{\min}$  of 1000 counts per minute and the selected minimum detectable leak rate. For data reduction following the tests, the precise indicated fill pressure was converted to true pressure with the gage factor, the true specific activity was determined, and a corrected threshold count rate was calculated for each site.

X1.5 *Procedure*—Two successive cycles were employed; the first cycle was a test for the indicated range of leak sizes  $\geq 1 \times 10^{-6}$  atm·cm<sup>3</sup>/s. A single measurement was made on each of the 100 sample packages. The second cycle was a fine

leak test for the indicated range  $\geq 1 \times 10^{-8}$  atm·cm<sup>3</sup>/s. Packages that unambiguously evidenced a leak  $> 3 \times 10^{-6}$  atm·cm<sup>3</sup>/s in the first cycle were set aside. Those packages which evidenced borderline values of  $< \sim 3 \times 10^{-6}$  atm·cm<sup>3</sup>/s were retested in the second cycle along with the remaining packages which had passed the first cycle. Activation parameters for the first cycle were arbitrarily set at a nominal 3 atm (304 kPa) absolute pressure and 0.01 h with a final pump-down to 2 torr (267 Pa) to minimize dwell time. Actual times ranged from 15 to 40 min. For the fine leak test, activation was nominally 5 atm (667 kPa) absolute with a pressurization time determined from Eq X1.1 for an uncorrected  $R_{\min} = 1000$  counts per minute. Final venting was at 0.5 torr (67 Pa), and activation times were typically 12 to 18 min. Actual dwell times ranged from 17 to 80 min.

X1.6 *Deactivation* was by storage in a vacuum chamber for about 48 h, plus variable wait times between tests. Initial count rates for the packages were nominally 2000 counts per minute or less.

X1.7 *Results:*

X1.7.1 Of those packages which evidenced borderline values of leak size of  $\sim 1 \times 10^{-6}$  atm·cm<sup>3</sup>/s, most exhibited a well-defined test value in the second cycle, which was about one decade smaller than the value measured in the first cycle, in which case the fine leak values were assigned to them. The remaining borderline leakers formed a second class which exhibited a large count rate in the second cycle of the order of 10<sup>5</sup> counts per minute, in which case the value measured in the first cycle was assigned. Each package then had a single measured value from each site. With this distinction, the between-laboratory agreement of whether leaks were  $> 1 \times 10^{-6}$  atm·cm<sup>3</sup>/s or not was 100 %. This agreement would not have resulted had the borderline specimens not been retested in the fine leak cycle. The agreement as to whether packages were leakers or not ( $< 1 \times 10^{-8}$  atm·cm<sup>3</sup>/s) was 95 %; this value was derived from 11 measurements on each of 25 packages initially selected as nonleakers with 14 individual measurements evidencing leak size greater than  $1 \times 10^{-8}$  atm·cm<sup>3</sup>/s. The results of the final repeated test made at the first laboratory showed a statistical independence from the first set of data, and so data were treated as derived from 11 independent test sites.

X1.7.2 An average value

$$\bar{X}_j = (1)/(n) \sum_i X_{ij} \quad (X1.1)$$

was derived for each sample  $j$  from the single measurement at each of  $i$  laboratories. The estimated standard deviation

$$S_j^2 = (1)/(n - 1) \times \sum X_{ij}^2 - n \bar{X}_j^2 \quad (X1.2)$$

represents the dispersion in measurements for each sample. The between-laboratory agreement is summarized in Fig. 3 as a plot of  $S_j$  versus  $\bar{X}_j$  for the gross leak range and similarly in Fig. 4 for the fine leak range. Regression calculations for these data result in the equation

$$\log (S_j/X_0) = a_0 + a_1 \log \bar{X}_j/X_0 \quad (X1.3)$$

with results as listed in Table X1.1.

X1.7.3 The expected agreement that would be obtained

TABLE X1.1 Regression Calculation Values for Eq X1.3 for the Gross and Fine Leak Ranges

Range	$a_0$	$a_1$	$X_0$	$r$	$S_{yx}$	$S_0$	$S_1$
Gross	-0.106	0.947	$1 \times 10^{-6}$	0.989	0.060	0.025	0.028
Fine	-0.280	0.839	$1 \times 10^{-8}$	0.980	0.308	0.036	0.022

$r$  = correlation coefficient.  
 $S_{yx}$  = estimated standard deviation for the dispersion in  $\log (S_j/X_0)$ .  
 $S_0$  = estimated 1 standard deviation error in  $a_0$ .  
 $S_1$  = estimated 1 standard deviation error in  $a_1$ .

between any two laboratories making a single measurement on the same specimen is derived from the fitted curves of Eq X1.3. The difference between two such measurements would not be greater than  $2.77 \sigma$  for 95 % confidence level, where  $\sigma$  is the standard deviation at the mean value of the two measurements ( $X_j$ ) for which the fitted values for  $S_j$  are the estimated standard deviation for 11 measurements.

X1.7.4 More detailed examination of the data indicates individual measurement biases for each test site.<sup>4</sup>

<sup>4</sup> Ruthbera, S., Neff, G. R., and Martin, B. D., "Radioisotope Hermetic Test Precision," Proceedings 1977 International Microelectronics Symposium, pp. 131-137.

## X2. CONCEPTS

X2.1 Krypton-85 is a radioisotope with a half life of about 10.76 years which decays to stable rubidium-85. About 99 % of the disintegrations involve the emission of beta particles with a maximum energy of about 0.7 MeV, and about 0.46 % of the disintegrations lead to gamma emission with an energy of approximately 0.52 MeV. In practice, <sup>85</sup>Kr is diluted with nitrogen gas, commonly in the ratio of about one part of krypton to  $10^4$  parts nitrogen. The devices to be tested are placed in an activation tank, which is then sealed and evacuated. The <sup>85</sup>Kr-nitrogen gas mixture is pumped into the tank to a prescribed activation pressure. During the prescribed "soaking" time, the gas passes through any package leaks and accumulates in the interior of the component. After the <sup>85</sup>Kr gas mixture is pumped from the activation tank and stored for reuse, the tank is vented and the components are examined with radiation counters. The beta radiation from the <sup>85</sup>Kr atoms that have entered the defective device is normally attenuated by the device wall, but the gamma radiation easily penetrates the device walls and is measured with an external scintillation crystal-equipped detection system; thus, the intensity of gamma radiation from within the device can be equated to the number of <sup>85</sup>Kr atoms that have entered. Adsorption of <sup>85</sup>Kr on the device wall can occur with some materials, or the gas can be captured in porosities near the exterior surface. In such cases a measure of the surface effect can be obtained by determining the beta radiation from the surface with a thin-windowed Geiger-Mueller tube or by monitoring the gamma radiation from selected samples.

X2.2 The flow of gas through the microchannels, cracks, and porosities that compromise the leaks in real packages is not readily described from first principles except for two special regimes, molecular flow and laminar flow. For any other case, solution is obtained on a semitheoretical basis which utilizes the superposition of molecular flow and laminar flow expressions with adjustable parameters that are functions of the mean free path of the gas molecules and known dimensions of uniform channels. Turbulence is possible under some of the leak test conditions, and this further obscures solution. Therefore, to obtain workable

equations for selection of pressurization parameters and quantification of results, simplified gas flow models are used. Traditionally the radioisotope hermetic test method has been described by a laminar viscous flow model.

X2.3 The flow rate of gas through a uniform cylindrical channel of radius  $r$  under a pressure difference between  $P_c$  upstream and  $P_i$  downstream is

$$q = (d)/(dt) (PV) = (\pi r^4)/(16 \eta l) (P_c^2 - P_i^2) \quad (X2.1)$$

where  $\eta$  is the viscosity of the gas. Other cross-sectional shapes require modification of the dimensional term. In general this flow rate is

$$q = K (P_c^2 - P_i^2) \quad (X2.2)$$

but from the definition of standard leak rate,

$$L = K (P_0^2) \quad (X2.3)$$

Since the tracer gas is nitrogen with a small concentration of <sup>85</sup>Kr, and since the viscosities of nitrogen and air are essentially equal,

$$q = (Q)/(P_0^2) (P_c^2 - P_i^2) \quad (X2.4)$$

where it is assumed that laminar flow is persistent at the activation pressures and the measured leak rate  $Q$  with the tracer gas is  $L$ . Under the assumption that the accumulation of gas within the package cavity is small, or that it grows linearly in time under pressurization, and that no <sup>85</sup>Kr escapes after activation, the radioactivity of the interior gas is the product of  $q \times T \times s$ , where  $T$  is the pressurization time,  $s$  is the specific activity, and  $q$  is the flow rate. If the counting efficiency is  $k$ , the resultant count rate is then

$$R = (Q)/(P_0^2) (P_c^2 - P_i^2) \times T \times s \times k \quad (X2.5)$$

X2.4 One microcurie of <sup>85</sup>Kr produces on the average ~10 210 counts per minute of gamma radiation (branching ratio of 0.46 %). Actually, counter sensitivities vary from <12 000 to ~15 000 counts per minute per  $\mu$ Ci, so that a large fraction of the radiation counter response is due to secondary ionization effects. Thus, the actual count rate is determined by counter geometry, crystal properties, shielding, position of test object, photomultiplier sensitivity, and amplifier characteristics.

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