RADIOISOTOPE HERMETIC TEST PRECISION

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ABSTRACT

Leak testing is an essential element of the screening process for hermetic devices, where test methods are, designed to reject all devices with leak rates greater than some prescribed value; nevertheless, poor measurement correlations between test methods and between test installations continue to be a major problem. An effort within ASTM Committee F-1 on Electronics is being made to resolve such measurements problems. An inter-laboratory comparison has been completed on the radioisotope test method using commercial test equipment and commercial glass-sealed ceramic packages (Cerdip). One hundred test packages with indicated leak ranging from less than 1 x 10^{-8} atm•cm^3/s to about 5 x 10^{-5} atm•cm^3/s were tested sequentially by 11 laboratories using a 2-step sequence in a specified procedure. These test results demonstrate conclusively that the inter-laboratory comparison of hermetic packages can be accomplished under appropriate guidelines with a precision (one standard deviation) ranging from 0.5 x 10^{-8} at 1 x 10^{-8} atm•cm^3/s to 3.2 x 10^{-5} at 5 x 10^{-5} atm cm^3/s. It is demonstrated that this precision could be increased significantly through elimination of the individual laboratory biases that were found to occur.

INTRODUCTION

Hermetic testing of semiconductor device packages has been part of the screening process for many years. Even with this long experience, severe measurements problems continue to exist. Although specifications on leak rate reject levels, such as in the military standards, are set in terms of definite numerical values, little agreement in the leak rates measured has actually been demonstrated between different installations using the same test method or for different test methods at the same installation [1]. As a matter of fact, one or more orders of magnitude differences in measurements agreement are the rule. Such measurement uncertainty has caused difficulties at the producer user interface on acceptability of parts; it has affected production yields; it leads to faulty packages escaping detection; and it has obscured the technical evaluation of the correlation of leak rate to reliability both in the attempts to set D.O.D and NASA screening levels to realistic values and to determine the true sensitivity of semiconductor devices to environmental contaminants such as moisture.

An effort is being made within ASTM Committee F-1 on Electronics to develop standards for hermetic test methods which will produce quantitative measurements and then to evaluate such test methods through inter-laboratory comparisons. A part of this effort has been completed with a round robin

The material reported here was derived from an effort within Committee F-1 on Electronics of the American Society for Testing and Materials, 1916 Race St. Philadelphia, PA 19103, for the purpose of developing a voluntary consensus standard as an aid to industry, government agencies, and the general public on one radioisotope method.

The purposes of this round robin were both to: determine the precision (between laboratory agreement) inherent in the radioisotope method when applied to production devices with commercially available test equipment and to generate a data base for further refinement of the draft method. To reduce the number of variables involved, one specific model of equipment was used so that all installations could follow the identical operational sequence in using their equipment to carry out the prescribed test procedure. The test procedure itself, however, can be performed with other suitable commercially available test equipment, although actual controls may vary. All equipment was serviced and calibrated prior to test.

Even though it was not clear at the onset that real package leaks were sufficiently stable for such an exercise. the test samples were circulated through 10 laboratories with a final repeated test made by the first laboratory to close the loop. All samples survived.

THEORY OF THE RADIOISOTOPE METHOD

Krypton-85 is a radioisotope with a half life of approximately 10 years. Over 99% of the disintegrations involve the emission of beta particles with a maximum energy of about 0.7 MeV, and about 0.5% of the disintegrations lead to gamma emission with an energy of approximately 0.5 MeV. In use, Kr^{-85} is diluted with nitrogen gas in the ratio of

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About one to 10^4 to obtain an increased quantity of test gas with a viscosity equivalent to that of air. The components are tested with apparatus as depicted in Fig. 1. The devices are placed in the activation tank, which is then sealed and evacuated. The Kr^{-85}-N_2 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 components. After the Kr^{-85} gas mixture is pumped from the activation tank and stored for reuse, the tank is vented and the com-ponents are examined with a gamma radiation counter, such as represented in Fig. 2, to measure the amount of Kr^{-85} that remains in the device interior.

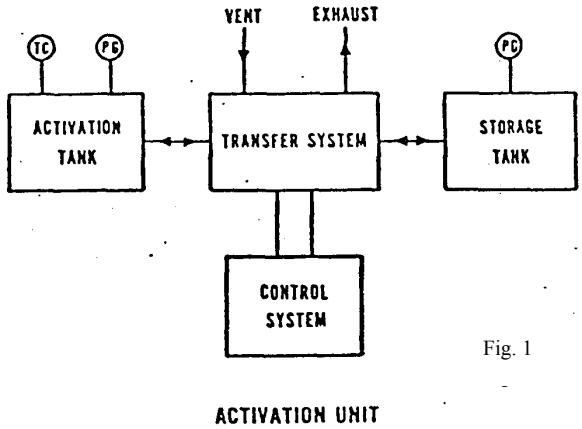

Fig. 1

Figure 1. Block diagram of Radioisotope Activation Console. TC - thermocouple vacuum gauge.

PG - pressure gauge. The equation traditionally used to re-late the gamma count rate to the leak rate is [2-4]

$$Q = \frac{R\,Po^2}{SKT\,(Pe^2 - Pi^2)} \qquad (1)$$

where:

Q = the leak rate (atm•cm^3/s or equivalent)**
R = count rate above ambient background
S = specific activity (μCi/atm cc of the Kr^{-85}-N_2 Mix)
K = overall counting efficiency of the detec-tor for the package type (counts/min/μCi)
T = soak times
Po = 1 standard atmosphere pressure
Pe = activation pressure (atm absolute)
Pi = initial internal package pressure (atm absolute)

This formula assumes that the gas flow through the leak is laminar that the gas accumulates within pa linear y in time, and that little pressure change occurs in the interior No provision is included for gas escape after pressurization

Since the emphasis of this first round robin was on measure-ments agreement rather than on absolute value, these as-sumptions were not of consequence except for the omission of a factor on gas escape from the package interior, and count rate determi-nations were to be completed within a specified time to minimize this effect of gas escape.

EXPERIMENT

Equipment

Activation Unit

The transfer system, Fig. 1, comprises vacuum pumps, com-pressor, gas transfer lines, valves, and filters all under com-mand of the control system which is linked to the various transducers for use, in automatic or manual operation. The activation chamber was normally evacuated to 0.5 torr prior to pressurization, and to 0.5 torr or 2.0 torr after pressuriza-tion as dependent upon the time allowed to transfer the parts to the counting sta-tion (dwell time). Pump-down time of 2 minutes or less was required to minimize evacuation of gas from packages with large leaks.††

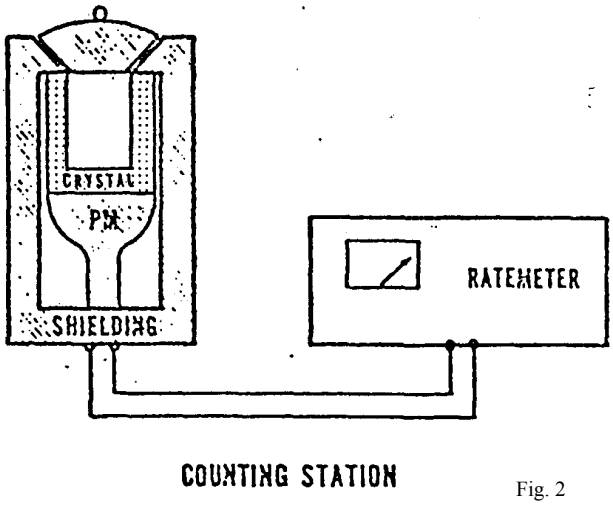

Fig. 2

Figure 2. Counting station components.

**Although the SI system of metric units is now preferred, present engineering practice uses the mixed units of atm-cm /s for leak rate, torr for vacuum, and psi for pressures near or greater than 1 atmosphere. Conversion factors are 1 Pa m^3/s = 9.869 atm-cm^3/s, and 1 Pa = 0.0075 torr or 1.451 x 10^{-4} psi; one curie = 3.7 x.10^{10} events/s.

††Actual test equipment was the IsoVac Engineering, Inc. Radiflo ® Mark IV Leak Detector. This equipment is identified in order to adequately specify the experimental proce-dure. Such identification does not imply recommendation or evalua-tion either by the ASTM or the NBS.

Counting Station

As shown in Fig. 2, the station comprised a shielded scintillation crystal, into which a well has been formed of about 2 in. diameter and 3 in. depth, a photomultiplier tube, preamplifier, and Ratemeter. The maximum unit rate of the stations used was 105 counts/min.††

Test Samples

One hundred 14-lead ceramic-glass dual-in-line packaged devices as pictured in Fig. 3 were select-ed by radioisotope test from a production run of several thousands of parts of a commercial inte-grated circuit with a nominal internal free volume of 0.02 cm^3. Manufacture was by normal methods, but these devices were not subjected to fluid immersion at any time. Selected indicated leak sizes ranged from < 1 x 10^{-8} atm-cm^3/s to ~ 5 x 10-s atm-cm^3/s. The test specimens were serialized at Random.

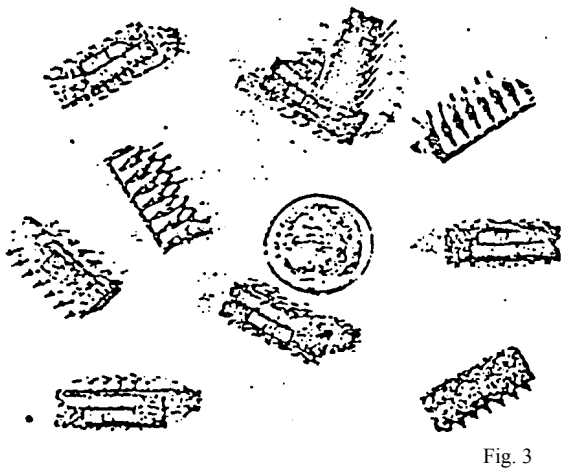

Figure 3. Ceramic-glass 14 lead dual-in-line test packages.

Calibration

The counting station at each of the test sites was calibrated against the same pulse generator and three Kr^{-85} reference standards [5]. The Ratemeter indications on all scales were adjusted for agreement to the pulse generator. The Ratemeter was then connected to the crystal/photomultiplier system and the counter was set into operation. One of the Kr^{-85} reference standards was inserted into the detector well, and amplifier gain was adjusted. until a plateau was reached for signal to noise ratio. Then the three Kr^{-85} reference standards, each of different activity, were used to calibrate the gamma count rate for each of the three larger Ratemeter scales, i.e., 0-10^4 counts/min, 0-3 x10^4 counts/min, 0-10^5 counts/min, and an average value was derived for the counter sensitivity in courts/min-μCi.

Pressure gauges were calibrated at atmospheric pressure against a mechanical reference gauge included in each console. These reference gauges were characterized by the manufacturer as having an accuracy of better than 1% and were not individually calibrated for this round robin. The thermocouple vacuum gauge in each console was calibrated against a transfer thermocouple gauge which calibrated against a precision liquid monometer [6].

The specific activity of the test gas was deter-mined by transferring a sample of gas into a glass vial of known volume at an indicated pressure of ~ 2.0 torr on the internal thermocouple gauge. The glass vial was of similar construction to the Kr^{-85} reference samples. The radioactivity was measured with the calibrated counting station, and a correction factor was applied for "true" pres-sure according to the calibration data on the thermocouple gauge. Three independent samples were so taken, measured, and an average value of specific activity in μCi/atm-cm^3 was computed.

Test Procedure

In practice, the hermetic screening process is not a measurement procedure but is a detection opera-tion for indicated leak rates greater than some set value. Normally the radioisotope method is used is a fine leak test procedure with the rejection limit set at such a value as to allow the detection of some of the larger leak sizes [3,4,7]. The purpose of this round robin was to determine the precision of radioisotope testing; therefore, a test procedure was used which would give definitive measurements. Two successive cycles were employed in order to obtain accurate count rates and to prevent overfill with the radio-active gas. The first cycle was a test for the range of leak sizes > 1 x 10^{-6} atm cm^3/s , and the second cycle was a fine leak test for the indicated range > 1 x 10^{-8} atm cm2/s. Packages which unambiguously evidenced a leak > 1 x 10^{-6} atm-cm^3/s in the first cycle were set aside and assigned values as measured. Those packages which evidenced borderline values of ~ 1 x 10^{-6} atm-cm^3/s were re-tested in the second cycle along with the remaining packages which had passed the first cycle.

The count rate for each device was measured initially at each installation to establish the back-ground level for each device. Activation parameters for the first cycle were nominally 3 atm-abs. and 36 s with a final pump-down to 2 torr to mini-mize dwell time and possible gas escape back through the larger leaks. Individual packages were measured in the counting station; all measurements were scheduled for completion within 15 minutes after removal from the activation tank. Actually the time taken for completion ranged from 15 min-utes to 40 minutes. Leak rate values were calcu-lated from equation (1).

The fine leak test was performed in the same manner as the first cycle leak test. Pressurization was nominally 5 atm-abs; soak time was determined from equation (1) as based upon each particular value of indicated specific activity in order to derive a count rate of ~ 1000 counts/min above back-

ground. for a 1 x 10^{-8} atm-cm^3/s leak size. Final venting was done at 0.5 torr, and soak times were typically 12 to 18 minutes. After measurement, correction was applied for "true" specific activity to derive "real" leak rate. Although not considered neces-sary for fine leakers, a 30 min time limit for completion of counting is good practice. Actual times ranged from 17 min to 80 min.

After testing at each site, the specimens were deactivated of Kr^{-85} by storing in a vacuum chamber for about 48 h. At each of the succeeding installations, initial device background count rates before test were nominally 2000 counts/min or less.

RESULTS

Data

Of those packages which evidenced borderline values of leak rate of ~ 1 x 10^{-6} atm-cm^3/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 lame count rate in the second cycle of the order of 10^5 counts/min, in which case the value measured in the first cycle was assigned. With this distinction, the between-laboratory agreement of whether leaks were greater than 1 x 10^{-6} atm-cm^3/s or not was 100%.This agreement was have resulted had the borderline specimens not been retested in the fine leak cycle. The agreement as to whether packages were leakers or no (< T x 10'a atm-cm^3/s) was 95%; this value derived from 14 individual measurements evidencing leak size greater than and 261 evidencing leak size less than 1 x 10^{-8} atm-cm^3/s as deter-mined from 11 measurements on each of 25 packages that were initially selected as non-leakers, and for every sample the agreement on no-leak was at least 9 out of 11.

Between-Laboratory Agreement

An average value

$$\bar{x}_j = \frac{1}{n}\sum_i x_{ij} \qquad (2)$$

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

$$s_j^2 = \frac{1}{n-1}\sum_i x_{ij}^2 - n\bar{x}_j^2 \qquad (3)$$

represents the dispersion in measurements for each. sample. The between laboratory agreement is sum-marized in Fig. 4 as a plot of s_j vs. x_j for all data, except for one specimen which gave a data point far from the mainstream and was later found to have a small external void. Between laboratory uncertainty increases continuously with leak size for both leak ranges.

It is noted that a gap in leak size occurred in the fine leak range. It is not known if this is a result of the sample selection process or the or the nature of the leaks in this type of package. The large dispersion for the range > 1 x 10^{-6}.atm-cm^3/s is certainly affected by the variation in dwell times that occurred at some of the installations.

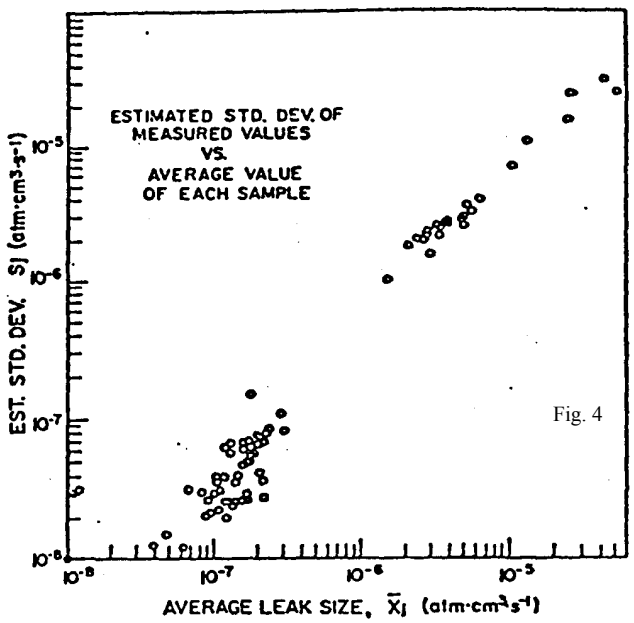

Fig. 4

Figure 4. Estimated standard deviations sj of measured values for all samples as a function of the sample average leak size xj.

Regression calculations show that the variation in between-laboratory reproducibility is well described by equations of the form

$$\log s_j = a_0 + a_1 \log \bar{x}_j \qquad (4)$$

for both the fine and larger leak range. The fitted curves are shown in Fig. 6 for the fine leak range (2nd test cycle) and in Fig. 6 for the larger leak range (1st test cycle). The expected agreement that would be obtained between any two laboratories making a single measurement on the same specimen is then derived from these curves. The difference between two such measurements has a variance of twice the square of the standard deviation, and on a 95% confidence level the difference would not be neater than 1.96 standard deviation or 1.96 (2sj2)1/2 2.77 sj, where sj is the value taken from the fitted curves.†††

††† Some adjustment on these values is required in principle to correct for the relationship of average value of sj to the standard deviation as. based upon the number of degrees of freedom in the measurements, but the correction is relatively small in the present instance.

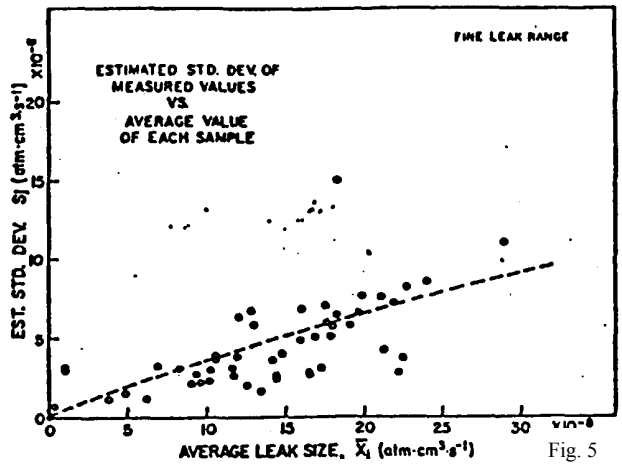

Figure 5. Estimated standard deviations sj of measured values in the fine leak range with a least squares fitted curve.

Laboratory Bias

More detailed examination of the data indicates that part of the overall systematic dependence of dispersion on leak size is the result of individual laboratory measurement biases. Sorting out the data on a laboratory by laboratory basis gives results such as shown in the two examples of Fig. 7 for the fine leak range and in Fig. 8 for the larger leak range where the individual laboratory measurement for each of the test specimens x_{ij} is compared against the average value x_j. A linear regression calculation of the form. $x_{ij} = a + bj$ was applied to each laboratory's results, and each laboratory showed a characteristic bias as indicated in Figs. 7 and 8; the bias differed between the two ranges at some laboratories. In fitting the lines to the data, an occasional value was found to fall far from the mainstream in the fine leak range, presumably random events. An occasional value was found to fall considerably below the mainstream in the larger leak range as marked by a slash in Fig. 8. In the latter case, the points are presumed to result from overly long dwell times.

Possible Precision

It is of interest to speculate on the precision that could possibly be obtained were laboratory biases eliminated. This possible precision was anticipated by taking the apparent precision at each laboratory and pooling the results for each test specimen. Specifically, the residuals were calculated for each test specimen as derived from each laboratory's regression line as x_{ij} - $_{ij}$, where xij is again the actual value measured by the i^{th} laboratory for the j^{th} sample and $_{ij}$ is the expected value as calculated from the regression line for each laboratory at the corresponding value of $_j$. A standard deviation s_r was then calculated from all the residuals for each specimen and, again, a regression curve was fitted. Results a represented by curve a in Fig. 9 for the fine leak range and by curve a in Fig. 10 for the larger leak range. Actual precisions that were obtained in the test, when laboratory biases are included are repeated again in curves b. Thus, from the comparison of curves a to b, precision might be improved by a factor of ~ 2 if laboratory bias could be eliminated. Identification of factor causing such bias remains for further effort. In the larger leak range one might expect further improvement in precision were stricter adherence maintained for short dwell time.

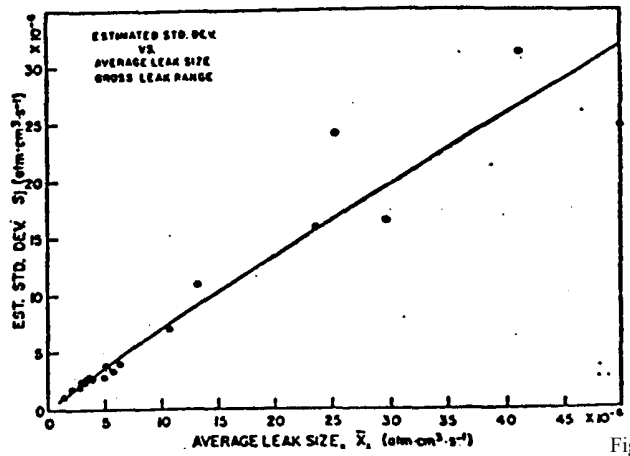

Figure 6. Estimated standard deviations sj of measured values in the gross leak range with a least squares fitted curve.

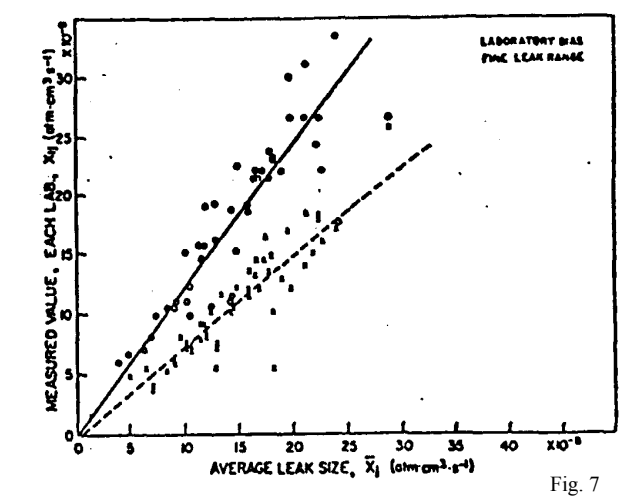

Figure 7. Example of individual laboratory bias. Measured values xij as a function of average leak size j for the fine leak range.

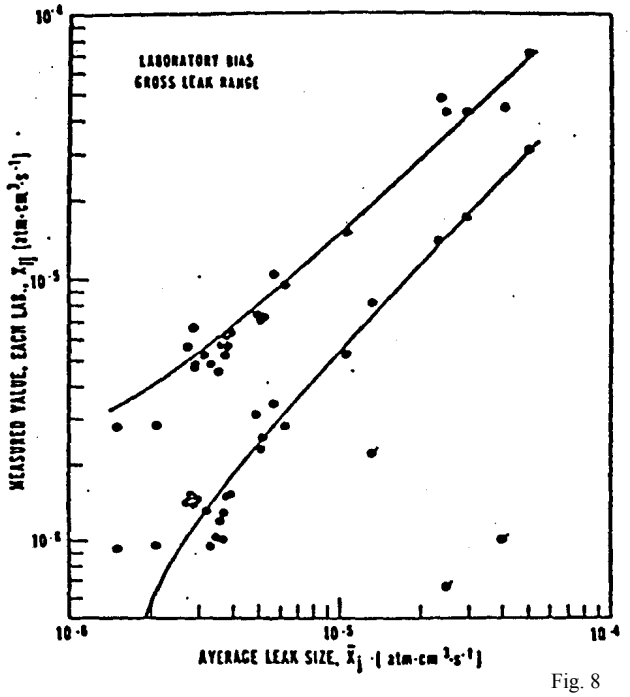

Fig. 8

Figure 8. Example of individual laboratory bias. Measured values xij as a function of average leak size xj for the "gross" leak range.

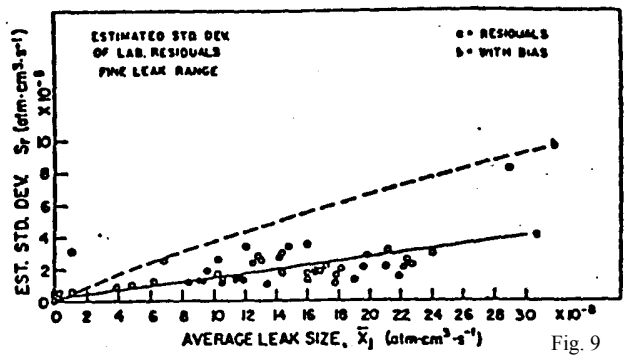

Fig. 9

Figure 9. Estimated standard deviations sr for the fine leak range as derived from residual values for each sample from each fitted line for all laboratories, est. std. dev. for all (xij – xij) where xij.= a0 + a1xj, with results in curve a. Curve b is the one std. dev. reproducibility derived from original measurements which include laboratory bias effects.

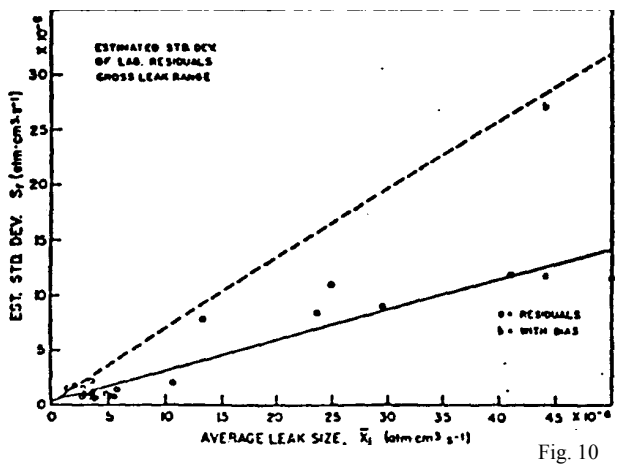

Fig. 10

Figure 10. Estimated standard deviation sr for the "gross" leak range as derived from residual values for each sample from each fitted line for all laboratories, est. std. dev. for all (xij - xij) with results in curve a. Curve b is the reproducibility (one std, dev.) with laboratory bias included.

Impact

These results demonstrate that an inter-laboratory comparison of hermetic packages can be accomplished with this radioisotope method and that one can achieve measurements precision greatly improved over the one or more orders of magnitude differences that have been the rule. Although the precision achieved here is probably better than that in the normal production environment, the guidelines imposed can be transferred readily and should lead to improvements there also, which in turn should help to resolve the producer user difficulties, yields, and technical evaluation problems which exist.

The actual precisions determined in this round robin depend on the guidelines imposed and on the type of test specimen used. Values would probably be affected by change in package materials and internal free volumes. Future efforts are planned to assess these effects.

It has also been shown that package leak rates, even for the small leaks, can remain stable provided reasonable care is taken in package transportation. Although these particular specimens were hand carried between installations, the significance is that even the finest of these leak sizes remained open in the field and therefore could pose a threat to reliability were they to escape initial screening.

A knowledge of measurement precisions such as reported here is one factor important to achieving a rational basis for hermetic testing. Leak test philosophy, as embodied in existing test specifications. has been essentially to set leak size reject limits as based upon general instrumentation capabilities and to observe the net result through failure analysis of operational devices some time later. However, efforts are now being made to determine the sensitivity of devices to

internal moisture, the major degradent [8], and to measure the infusion rate of moisture as a function of leak size [9]. Once the effects of such environmental contaminants on circuit operation are known definitively, once the infusion rates of such contaminants are known quantitatively as a function of leak size, and once the measurements uncertainties are established, realistic operational specifica-tions could be set to eliminate all leakers to a chosen confidence limit.

ACKNOWLEDGMENT

To carry out an inter-laboratory comparison of this nature requires considerable preparation and the cooperation of many personnel. Those organizations which gracefully participated in the measurements were the Burroughs Corp., Fairchild Semiconductor, Hughes Aircraft, IBM Corp.(2 installations), IsoVac Engineering, Inc., Lockheed Missiles and Space Co., Motorola, Inc. (2 installations), and TRW Systems.

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