PACKAGE INTEGRITY MEASUREMENT TECHNOLOGY AND QUALITY ASSURANCE

Raytheon Company
Aaron DerMarderosian and Vincent Gionet

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APPROVED: 

BENJAMIN A. MOORE
Project Engineer

FOR THE COMMANDER:

JOHN J. BART
Chief Scientist, Reliability Sciences
Electromagnetics and Reliability Directorate

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This report is the result of a study to evaluate two procedures in MIL-STD-883D, Test Method 1014.9 (Seal) and Test Method 1018.2 (Internal Water-Vapor Content). The first portion of this report reviews and investigates the current test procedures included in Test Method 1014 and explores new test methods for recommendation of incorporation into a new revision of the test method. Included in Appendix A are the results of a survey of industry for comments and recommendations for improving and clarifying the present test method. Appendix A of this report presents the proposed new version of Test Method 1014.

The second portion of this report details the results of a study that provided and distributed Test Method 1018 moisture measurement correlation samples to various Residual Gas Analysis (RGA) laboratories. This was done in order to determine the accuracy and precision of mass spectrometric measurement of moisture in microelectronics packages at facilities deemed suitable or candidates to be deemed suitable by the Defense Electronic Supply Center (DESC) to perform MIL-STD-883D, Test Method 1018.2, Procedure 1 (Mass Spectrometry), moisture testing.
This study was initiated to improve the present methods found in MIL-STD-883D test, Method 1014.9 (Seal) and Method 1018.2 (Internal Water Vapor Content). The scope of the concern is to reduce the incidence of gaseous ambient induced failures by improving the present Mil-Standards.

The study focuses on reviewing present practices, exploring new ones and suggesting recommendations for revisions. The aim of the study was to try and gather as much useful information, i.e., data, comments, recommendations, ideas and new leak tests from the micro-electronic industry at large, and use this information to make improvements to the Mil-Standard.

The response to an industry wide survey of testing practices, comments and recommendations in the form of a questionnaire was minimal. Of the one-hundred-one (101) persons surveyed, only thirty-two (32) replied. For the most part, little information of value to this study was obtained with the exception of a few respondees who elaborated more with their replies and indicated a genuine concern for change. The inputs from all respondees were channeled into making the recommendations that would benefit everyone.

The study was initiated with a search and review of new technology and procedures which would demonstrate potential for inclusion in Method 1014. These included studying laser optical techniques and the use of a 37% He tracer gas. A study was also performed to evaluate the use of a pre mass spectrometer bake at 125°C to remove helium gas from package surfaces caused from the bombing process.

We have studied the behavior of so called one-way leakers. This was accomplished by varying the test pressure and temperature. Special fixturing was designed and fabricated for these tasks. The results of these experiments show that most parts leak bidirectionally and behave according to molecular flow. There were some examples, however, of directional flow behavior as well as those whose leak rates were severely affected by temperature. Because of the unpredictable nature of these parts (the directionallity is not always predictable as to effect and direction), we cannot recommend a particular test method which can detect them consistently. We do feel, however, that the tighter limits (< 1 x 10^-8 ATM cc/sec) coupled with package integrity design guidelines will go a long way towards their elimination.

The survey test data generated, along with a review of the existing procedures in MIL-STD-883D, Method 1014 for fine and gross leak testing, led us to the following major conclusions and recommendations: (The complete revision of Method 1014 is shown in Appendix A).
The present failure criteria for helium and Krypton 85 fine leak testing (Test Condition A and B) is too lenient. We recommend a maximum allowable air leak rate of $1 \times 10^{-8}$ ATM cc/sec for all tests and packages regardless of package internal volume.

The helium fine leak fixed method ($A_t$) is a compromise and should be eliminated.

A post bomb bake prior to fine leak test at $100-125^\circ C$ for 10 to 15 minutes should be allowed in order to rid the package of absorbed tracer gas. This will reduce background noise levels and allow for reliable multiple part tests as well as increase the sensitivity of the test.

The Krypton 85 Test (Condition B) should be rewritten for molecular flow (in place of viscous flow at present) and account for the loss of gas after depressurization, i.e., same principle as the flexible helium leak test method (Howl and Mann Equation).

Replace the fixed method with an alternative helium backfill method at seal. This would simplify testing and assure detection of leaks in larger packages down to $1 \times 10^{-8}$ ATM cc/sec.

The gross leak bubble test should limit the number of parts tested at one time, to a maximum of four (4).

Simplify the Howl and Mann expression as described in 1014; $A_t$.

The results of the 1018 correlation study revealed that many of the R.G.A. test facilities had "drifted" somewhat out of calibration and indicated problems with both ends of the volume range tested (.01 cc and 5.5 cc). The testing was performed in two trials. The first trial indicated a calibration problem with 2 of the 3 RGA houses while the second trial indicated a potential problem with the small volume correlation samples, since three (3) of the four (4) facilities were in reasonably close agreement with each other. The effects of the larger volume package, however, were still evident as shown in the first trial.

As a result of these findings we recommend that:

A. Qualified RGA facilities should have several hundred correlation samples to test over a 3-6 month period in order to establish a meaningful statistical basis for their calibration, measurement approach, and procedure.
B. Rome Laboratory should evaluate their data and procedures and establish a firm set of procedures which can be audited on an ongoing basis.

C. Evaluate the use of a rolled gold interior for the correlation samples to eliminate any variabilities in oxide thickness levels within the package cavity.
ACKNOWLEDGEMENTS

The authors would like to acknowledge the efforts of those who participated in the hermeticity survey. The responses from AMD and NASA were particularly thorough and helpful in our preparation during the rewriting of Method 1014. We would also like to thank Messrs. L. Bergquist and T. Greene of Martin Marietta for their efforts in examining their "single leak test" method for part of this study and Mr. J. Tyson of Laser Technology for his testing method for gross leak detection based on laser interferometry. The latter has shown promise as a new method for inclusion into Method 1014 and joins the Kr85 and weight test methods for their ability to detect certain types of one-way leakers.
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EVALUATION

The objective of this effort was to assure the reliability of state of the art microelectronics used in Air Force systems by improving existing package integrity test methods found in MIL-STD-883D. The package in which a microelectronic device is contained not only prevents mechanical damage to the enclosed device but also should assure a benign gaseous atmosphere to prevent catastrophic failure mechanisms and/or electrical parameter drift to out of tolerance conditions. If the package is not hermetic or if the package contains potentially dangerous contaminants (i.e., water as vapor or adsorbed on internal surfaces), failure mechanisms, both short and long term, could be activated. MIL-STD-883D contains test methods to confirm the hermeticity (Test Method 1014) and limit the internal moisture content (Test Method 1018) of military microelectronics. However, packaging technology has become much more complex since the implementation of Test Method 1014. Also, new procedures for fine and gross leak testing have been developed. In addition, these larger, more complex packaging schemes, along with the inclusion of new materials within the package (glasses, die attaches, organics) have caused problems in correlating moisture measurements among certified laboratories.

Raytheon Company has accomplished the main objectives of the contract with respect to hermeticity testing. They surveyed industry for comments and suggestions for improvements to Test Method 1014. Raytheon has developed and tested a new procedure for fine leak testing that involves backfilling devices with known quantities of helium during the package sealing operation. This allows fine leak testing without pressure bombing and is especially appropriate for large surface area and "delicate" packages. The acceptable leak rate for this procedure is proposed to be $8 \times 10^{-9}$ std cc/sec air. Raytheon also recommends removal of the fixed method for fine leak testing due to inconsistencies related to package volume ranges. In order to facilitate use of the alternate flexible fine leak testing method, the contractor has simplified the Howl-Mann equation used to determine test conditions. Raytheon also confirmed that the flow assumption (viscous rather than molecular) used to develop the radioactive krypton test procedure was in error. They have corrected the equations in this procedure to reflect molecular flow. Raytheon studied the "One Way Leaker" phenomena and discovered that, in most cases, that the fine leak criteria now in Test Method 1014 are much too liberal. Raytheon proposes for all package sizes for the existing procedures in Test Method 1014 an acceptable fine leak rate of $1 \times 10^{-8}$ std cc/sec air.

Raytheon also manufactured moisture correlation samples and distributed them to commercial gas analysis facilities in order to determine the accuracy of analysis at each facility. Not all laboratories correlated. The presence of helium in correlation samples surfaced problems at labs that did not accurately calibrate for this gas. As a result, a second set of samples were produced without helium and distributed to the same laboratories. Again, not all labs agreed. Raytheon has sent the remaining samples to Rome Laboratory for continuation of the correlation study. This study emphasizes the need to conduct correlation studies with a matrix of samples more frequently than has been done previously.

Benjamin A. Moore/Program Manager
INTRODUCTION

The rapid changes of the state-of-the-art technologies in the microelectronics industry has placed a major priority on manufacturing high reliability devices in the military industry. As a consequence of this, Rome Labs, in an effort to maintain this level of reliability consciousness, has undertaken a review of the current test methods found in MIL-STANDARD-883D, Methods 1014 (Seal Test) and Method 1018 (Internal Water-Vapor Content). The scope of their concern is to reduce the incidence of gaseous ambient induced failures by improving the present MIL-STANDARD Methods 1014 and 1018.

Raytheon Co., under contractual agreement with Rome Labs, has undertaken the task of providing a detailed study to investigate the current version of MIL-STD-883D, Method 1014 and explore and investigate new test methods for incorporation of a new revision to the present test methods. As part of this agreement, Raytheon was asked to provide correlation moisture standards for the purpose of surveying commercial RGA (Residual Gas Analysis) companies deemed certified by DESC to perform analysis for the military per MIL-STANDARD-883D, Method 1018.2. The present procedures and practices are to be closely scrutinized and recommendations made for improving the method for the purpose of achieving commonality with calibration and parity with test results.

The key elements of this study are contained in the following outline.

TEST METHOD 1014 (SEAL)

- Study and Review Package Measurement Technology as it pertains to MIL-STD-883D, Method 1014
- Survey the industry for recommendations to changes in Method 1014
- Identify potential new test methods and techniques
- Report findings
- Review and study one-way leakers
- Report findings
- Make recommendations
TEST METHOD 1018 (INTERNAL VAPOR CONTENT)

Conduct a laboratory correlation study involving RGA tests of hermeticity sealed packages.

2. Distribute to suitable laboratories.
3. Collect and analyze all data.
4. Report findings.
5. Make recommendations.
To begin our study, we had to decide whether the existing procedures in MIL-STD-883D, Method 1014 were effective in screening out hermeticity failures in the fine and gross leak tests. In order to get an objective opinion of these leak tests, it was important to survey the rest of the industry and determine the likes and dislikes as well as any problems associated with the use of these test procedures. A questionnaire was prepared for this purpose as shown in Appendix B. The questionnaire was prepared in four (4) sections.

1. **General questions** about leak testing procedures, type of packages tested, thru-put, failures, likes, dislikes, recommendations, etc.

2. **One-Way Leaker Phenomena** - Knowledge of, experience with and data to share.

3. **Equipment Manufacturers** - Types of tests used, training of customers, changes in test specifications which would produce better equipment and recommendations.

4. **Failure Analysis** - Types and percentages of leakers, their leak sites and methods for finding their location.

A list of prospective questionees was drawn up from several sources to include names of persons supplied by Mr. B. Moore of Rome Labs, vendor lists, authors of pertinent papers and recommendations of other associates. Approximately three hundred (300) people were contacted via telephone, of this number, one-hundred-one (101) people expressed a willingness to answer a questionnaire if mailed to them. Out of the one-hundred-one (101) questionnaires mailed, we received thirty-two (32) replies, the replies were summarized and are enclosed in Appendix C. The replies from this survey seemed to express only a mild concern from most people, with the exception of less than ten (10) people whose replies were more in-depth with a greater concern to share and express their knowledge, experience, data and recommendations on the subject.

In the interim, we conducted a literature search through our Library Technical Search Service for the purpose of gathering for review all new as well as old hermetic seal testing information which might be made available. We were also interested in trying to obtain any relevant data pertinent to the one-way leaker phenomenon. The material searched included the following:

- ASTM and MIL-STD tests.
IEEE papers on hermetic seal tests.

Manufacturer's test equipment data and specs.

All other papers concerning seal testing.

The list of papers which surfaced from this literature search are listed in the bibliography of this report.

One of the latest developments in leak testing technology to surface is a combined fine and gross leak helium leak test utilizing a modified cryopump which reportedly achieves a greater range of test sensitivity. According to the developers, Bergquist and Shertz, quoting their findings and conclusions, "either the helium that has escaped from the component is measured or the rate in which it escapes is measured". Also "if the leak is gross, the helium will quickly escape to the level in the atmosphere which is 5 ppm in air. The differences between a gross and fine leak are easily detected because in the gross leak all the helium escapes into the manifold". Unfortunately we were unable to perform any correlation studies with this equipment during the contract period.

Another recent leak test is an optical method developed by LTI, Laser Technology, Inc. of Norristown, PA. which utilizes a laser illumination and video interferometry system and can accommodate singular components in a tray or complete circuit boards. The equipment measures the deformation of the device cover with an applied pressure or vacuum. Reducing the ambient pressure will cause the lid to bulge and if a leak is present the lid deformation will change as it "leaks down" thus relating to a leak rate. Knowing the geometry and the stiffness of the lid it can be factored into a leak rate equation to determine the actual leak rate. This system of detection and measurement works well for large electronic packages e.g., hybrids and devices with large covers but may prove ineffective with small and stiffer lidded devices.

This test method appears to have potential for study and for possible inclusion with Method 1014. We received a group of 20, 40 and 48 lead metal covered integrated circuits from Laser Technology, Inc. which were tested by them utilizing the laser optical method. Kr85 and helium leak tests were also performed at two other companies. We in turn performed our own leak study on these parts to determine if there was correlation between the optical and the helium leak test. Our test results shown in Table 1 indicate close correlation with that of Laser Optical Leak Rates. Based on these results, we feel that this technique shows promise.

(1) Lyle E. Bergquist, Stephen R. Shertz, Helium Leak Test for Small Components, Martin Marietta, Denver Aerospace, Denver, Colorado, USA.
# Table I. Laser Optical Correlation Leak Study Results

<table>
<thead>
<tr>
<th>Device Serial Number</th>
<th>Laser Optical Leak Rate</th>
<th>Texas Instruments (Krypton) 1.5 Yrs. Ago</th>
<th>Hughes (Krypton) 8.0 Mos. Ago</th>
<th>Raytheon Helium Leak Test Results</th>
<th>96 Hr. Bake Weight Loss Milligrams</th>
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<tr>
<td>A10</td>
<td>2.10E-05</td>
<td>6.70E-05</td>
<td>1.20E-04</td>
<td>2.60E-06</td>
<td>0.40</td>
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<tr>
<td>A2</td>
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<td>&lt;1E-10</td>
<td>0.40</td>
</tr>
<tr>
<td>B3</td>
<td>&gt;1E-4</td>
<td>4.40E-06</td>
<td>1.00E-05</td>
<td>&gt;1E-4</td>
<td>42.40</td>
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<tr>
<td>B5</td>
<td>&gt;1E-4</td>
<td>2.80E-06</td>
<td>7.00E-06</td>
<td>&gt;1E-4</td>
<td>32.70</td>
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<tr>
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<td>0.00</td>
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</table>

*Somewhat pressure sensitive*
The library search for information regarding one-way leakers turned up nothing significant on the subject. A technical article was found through a questionnaire response pertaining to one-way leakers (2) but did not provide any new information or methods for identifying one-way leakers. It rather focuses on RGA analyses of a large group of various devices from 1.2 to 17.8 cc volumes and attempts to correlate moisture ingress with package sizes as they relate to bombing pressures. The questionnaire didn’t provide anymore revealing information from respondees about this phenomenon. Of the thirty-two (32) questionnaires returned, sixteen (16) responded that they were aware of this phenomenon and nine (9) responded with methods for detecting one-way leakers, they were: RGA, dye penetrant, Krypton 85 and the weight gain test (See Appendix C).

ONE WAY LEAKER STUDY

In preparation for our one-way leaker experiments we planned on enlisting the aid of other sources from the respondees of our questionnaire to help supply us with potential one-way leaker candidates. There were no positive responses. We, therefore, had to rely on our own inventory of parts, leakers and non-leakers to perform our experiments. It is important to note that all of the leakers that we used for this study were detected by the flexible method (A) utilizing a failure criteria of $1 \times 10^{-8}$ ATM cc/sec; air. It is also important to note that we typically pressure bomb devices at 60 to 100 psig for periods of time in excess of sixteen (16) hours and as much as 100 hours prior to testing. This method increases the signal in the mass spectrometer and increases the internal pressure of the device. The increased pressure also helps to assure detection since some devices are pressure sensitive leakers. This pressure sensitivity will be shown in some of the devices we had tested.

A dual chambered test fixture was designed to perform these experiments. This fixture allowed for helium leak testing of a component in two directions; inwardly and outwardly so that a differential pressure could be applied either internally or externally to the device under test. The test fixture shown in Figure (1) has a dividing stainless steel test plate/tube assembly separating the two halves of the fixture. A test device is soldered onto the brass tube and plate assembly. The device/plate was then either placed upright or inverted in the fixture and clamped together depending on the direction of test. The total assembly was then attached to the inlet port of a helium leak detector.

(2) Dan Epstein, How to Test for One Way Leakers, ICL Data Device Corp., Bohemia, N.Y., USA,
ONE - WAY LEAKER FIXTURE

Figure 1
There were some problems associated with attempting to fasten a
test device to the brass tubes on the plate. First of all, the
interconnect had to be of a material which was impervious to
helium. This ruled out the use of rubber, plastics and nylon etc.
After some trial experiments it was decided that the best method
was to attach a copper or brass tube directly to the test device
which had a drilled or sand blasted hole to the package interior.
Again this attachment also had to be impervious to helium. This
was accomplished by soft soldering the tube directly to the device.
Ceramic lidded devices had to be prepared by ion sputtering around
the hole site with 100 to 200Å of chrome, 25,000Å nickel and 5,000Å
of gold metallization (See Figures 2 and 3). Considerable care had
to be exercised with the soldering because of the possibility of
flux vapors plugging leak sites and solder plugging the inlet hole.

Once the device was attached to the tube/plate assembly, the
internal pressure of the device was increased while it was
submerged in fluorocarbon fluid. In this way, we could determine
the leak pressure and leak site of the part as well as the quality
of the solder connections. Figure (4) illustrates the attachment
of a hybrid device to our tube/plate assembly.

TEST PROCEDURE WITH ONE-WAY LEAKER FIXTURE

The test device plate was clamped into the test fixture and placed
on the helium leak detector port as shown in Figure (1). The upper
chamber was blanked off by three valves leading to a vacuum pump
from a tee on one side and a helium tank and regulator on the other
and a center closure needle valve. The upper chamber was then
evacuated by opening the valves to the vacuum pump providing a zero
"0" psi differential pressure by removing all the ambient air in
the system. After a period of approximately 10 to 15 minutes, a
zero or background leak detector reading was recorded. The vacuum
line was then blanked off and helium pressure was slowly released
into the upper chamber monitored by a vacuum/pressure gage
graduated in 1 psi increments. Depending on the response of the
helium leak detector, the device was incrementally pressurized and
helium readings recorded. At 15 psi of 100% helium, the leak rate
of the device can be simply converted to the standard air leak rate
by dividing the value by 2.7. By incrementally increasing the
pressure and observing the behavior of the leak readings it can be
observed if the device is a pressure sensitive leaker. For
example, if a small increase in pressure causes a large change in
leak rate (up or down), then the device would be considered to be
pressure sensitive. This effect can be seen clearly in figures 9-
12. Subsequent testing of the device in the opposite direction
will determine if the device is a one-way leaker and/or pressure
sensitive.

We performed over fifty (50) experiments, often times repeating the
same experiment on the same device several times to determine
repeatability.
Figure 2

GOLD SPUTTERED I.C. 1/4 x 3/8

Figure 3

I.C. WITH SOLDERED CU TUBULATION
TUBULATED HYBRID DEVICE

Figure 4
ROOM TEMPERATURE TESTS:

There were a total of forty-five (45) devices tested. Fifteen (15) exhibited leak rates much greater than $1 \times 10^{-6}$ ATM cc/sec and could not be used. Fifteen (15) were non-leakers and were used essentially as controls to assure that the results were not affected by "false" signals. Two (2) devices were damaged and hence not used. Nine (9) devices were equal leakers in both directions (molecular flow) and four (4) were found to leak greater in one direction than the other and were pressure sensitive as well.

"NORMAL" DEVICES:

The "molecular flow" devices were characterized by both (a) equal leak rates at all pressures in each direction and (b) followed the classic molecular flow equation prediction which describes the leak rate as one which is directly proportional to the pressure difference (i.e., doubling of the pressure, doubles the leak rate). See Figures 6-8 for the details of this type of leaker (Serial #55 and 351). As stated before, there were a total of nine (9) devices which behaved similar to these two (2). None of the devices tested in any of these experiments indicated a leak rate behavior which would be predictable by either viscous or transitional flow equations. We have concluded from these tests as well as others we have observed over several years, that the molecular flow assumptions of the flexible method of fine leak testing (A of Method 1014) are correct and that the viscous flow assumptions of the Kr85 radioactive fine leak test are not valid and hence must be corrected in order to obtain reasonable correlation between these two (2) test methods.

PRESSURE SENSITIVE DEVICES:

Four (4) of the devices (16 lead flat packs) examined were clearly pressure and direction sensitive leakers. The leak behavior of these parts were somewhat predictable and at times erratic. These characteristics suggest that they were probably contaminated (flux, fluorocarbon etc.) In spite of this, it was felt that they represented some of the general population of non-hermetic devices and may help to shed some light on "confusing" residual gas analysis results. In examining Figures 9-12, some interesting behavior can be seen. As an example, Serial #216 (Figure 9) shows that little to no tracer gas could get into the device (external pressure) until about 100 psia and if it had leaked in, the internal pressure would have to exceed 75 psia to be "rejected" by using current Test Method 1018 criteria. It is clear that this part could easily escape detection at this time and would probably fail the requirements of Method 1018 residual gas analysis. Since this device had previously been detected as a leaker using the "flexible method" (A2), we feel that it had somehow become
contaminated and is the root cause of this "new" behavior. Any number of environments could have provided the contamination for the part (i.e., soldering fluxes, thermal shock fluids, cleaning solvents, etc.). In any case, it is clear that the part, at present, could be classified as being a pressure sensitive leaker.

In examining the behavior of Serial #41 (Figure 10) another category of pressure sensitivity emerges. This part shows a clear direction sensitivity i.e., helium flows easily into the device following the molecular flow predictions yet does not flow out of the part until the pressure reaches about 60 psia and then rather dramatically increases its leak rate by nearly three (3) orders of magnitude at 75 psia! We suspect that this device is truly a pressure sensitive leaker and not afflicted with contamination. Since this part was originally rejected using the flexible method, which uses 90 psia as a bombing pressure for periods of time up to 60 plus hours, we would/could expect to detect this part as a leaker. In this case the longer bomb times can be advantageous in culling leakers.

In examining the behavior of Serial #214 (Figures 11 and 12) it is evident that the device is a pressure sensitive leaker in both directions. At approximately 75 to 90 psia the device changes its leak rate from < 1 \times 10^{-6} \text{ ATM cc/sec He} to > 1 \times 10^{-6} \text{ ATM cc/sec He! As with the previous part (Serial #41), we feel that our practice of long pressurization periods helped to detect this device in the original leak tests. A standard "fixed" bomb time of just a few hours probably would have not detected this unusual behavior.

**TEMPERATURE SENSITIVITY TESTING:**

A test fixture was fabricated for the purpose of performing experiments at hot and cold temperatures. This fixture shown in Figure (5) incorporates a thermoelectric element for the purposes of heating and cooling the device under test (DUT). This fixture worked sufficiently well for heating a device but had its limitations when trying to cool a device below 0°C. Several experiments were performed with this test fixture and it worked sufficiently well. The results of our temperature tests indicated a net effect of slightly decreasing the leak rate when there was an elevated temperature of 100°C by a factor of 0.6 to 0.7 and had a reverse effect of slightly increasing the leak rate with an approximate 15°C drop in temperature from room ambient.

There was an exception in experiments #47 and #48 when the tests were repeated on device Serial #038 of varying temperature; see Figures (13) and (14). The results during these tests indicated that, by heating the device and holding the pressure constant, the leak rate was lowered and the leak was effectively closed. Cooling the device produced only a slight increase in the leak rate.
The results of the temperature sensitivity test for this one device indicates a dramatic effect from increasing temperature which is not clearly understood at this time. Although this device was found originally as a leaker that can be easily confirmed utilizing typical test procedures, it does create some concern in attempting to predict its behavior in future tests. Previous studies by others also noted a temperature sensitivity to some leakers but concluded that "temperature bombing" of parts would add little value to hermeticity testing. We also conclude the same based on our results.

CONCLUSIONS:

The results of these tests, although limited in nature, indicate that:

A. One-way leakers clearly exist and that their presence can cause confusing RGA results.

B. Molecular flow is the predominant regime for fine leakers.

C. Pressure bombing at the higher pressures for longer periods of time (i.e., > 60 psia for > 12 hours on devices with cavity volumes less than ~ 0.1 to 0.2 cc) appear to increase one-way leaker capture rates. More work would be needed to obtain a clear statistical basis for this finding.

D. The temperature test results support previous findings which have concluded that its use would be of little to no real value.
ONE-WAY LEAKER FIXTURE

CUTAWAY VIEW OF ONE-WAY LEAKER FIXTURE
WITH HEATER / COOLER ELEMENT

FIGURE 5.
ONE-WAY LEAKER STUDY
EXPERIMENT #15

PART TYPE: 16 LEAD FLAT PAK
PART SERIAL# 55
LOT #: QUAL. LOT TR787-2002
TEST DATE: 10-2-90

INTERNAL PRESSURE

LEAK RATE ATM. CC/SEC. (HELIUM)

TIME (MINUTES)

FIGURE 6.
FIGURE 7.
ONE-WAY LEAKER STUDY
EXPERIMENT #42

PART TYPE: 16 LEAD FLAT PAK
PART SERIAL# 351
LOT # : V89-017
TEST DATE: 11-14-90

INTERNAL & EXTERNAL PRESSURE

LEAK RATE ATM. CC/SEC. (HELIUM)

10^-5

10^-6

10^-7

10^-8

10^-9

10^-10

0 PSIA (VAC.) "BACKGROUND SIGNAL"

0 PSIA - 120 PSIA

15 PSIA - 75 PSIA

30 PSIA

45 PSIA

60 PSIA

75 PSIA

90 PSIA

105 PSIA

120 PSIA

INT LEAK RATE

EXT LEAK RATE

TIME (MINUTES)

FIGURE 8.

17
NOTE:
1. WITH INTERNAL PRESSURE LEAKER INCREASED DRASTICALLY AT 105 PSIA
2. WITH EXTERNAL PRESSURE LEAKER INCREASED DRASTICALLY AT 120 PSIA

FIGURE 9.
ONE-WAY LEAKER STUDY
EXPERIMENT #16

PART TYPE: 16 LEAD FLAT PAK
PART SERIAL: #41
LOT #: 10-2
TEST DATE: 10-2-90

INTERNAL & EXTERNAL PRESSURE

EXTERNAL LEAK RATE

INT LEAK RATE

0 (VAC.) TO 45 PSIA

FIGURE 10.
ONE-WAY LEAKER STUDY
EXPERIMENT #11

PART TYPE: 16 LEAD FLAT PAK
PART SERIAL #214
LOT #: V89-017
TEST DATE: 9-27-90

INTERNAL PRESSURE

LEAK RATE ATM. CC/SEC. (HELUM)

TIME (MINUTES)

FIGURE 11.
ONE-WAY LEAKER STUDY

PART TYPE: 16 LEAD FLAT PAK
PART SERIAL #214
LOT #: V89-017
TEST DATE: 9-28-90

EXPERIMENT #11

EXTERNAL PRESSURE

LEAK RATE ATM. CC/SEC. (HELIUM)

TIME (MINUTES)

10^-5
10^-6
10^-7
10^-8
10^-9
10^-10

1.7E-6 (OFF SCALE)

LEAK OPENS AT 105 PSIA

'O' RING BLEW OUT AT 75 PSIA ON THE FIXTURE

FIGURE 12.
ONE-WAY LEAKER STUDY
EXPERIMENT #47
RUN #1 INTERNAL PRESSURE
RUN #2 EXTERNAL PRESSURE
RUN #3 EXTERNAL PRESSURE & TEMPERATURE

LEAK RATE ATM. CC/SEC. (HELIUM)

TIME (MINUTES)

FIGURE 13.
As outlined in the Statement of Work we were requested to conduct a laboratory correlation study. The purpose of this study was to evaluate the accuracy and precision of mass spectrometric gas analysis facilities that are presently suitable, or are candidates to be deemed suitable by the Defense Electronics Supply Center (DESC), to perform Method 1018 (Internal Water Vapor Content), Procedure 1 of MIL-STD-883C, dated 4 November 1986. Note: This study was not for the purpose of determining technical certification or suitability.

The government supplied a list of five (5) commercial RGA facilities for the purpose of performing analyses for this study. They are listed as follows:

COMMERCIAL LABORATORIES

Atlantic Analytical Laboratory
Whitehouse, New Jersey

AT&T Microelectronic Analytical Services
Allentown, Pennsylvania

IT International Technology Corp.
Cerritos, California

Oneida Research Services, Inc.
Whitesboro, New York

Pernicka Corporation
Fort Collins, Colorado

We were requested to provide three hundred fifty (350) correlation samples to be equally divided and distributed between commercial and non-commercial analytical laboratories, the latter half being directly distributed to non-commercial laboratories by the government (Rome Labs). The samples were fabricated from various all nickel T.O. series transistor packages, caps and bases in assorted combinations to approximate five (5) different volumes. We were instructed to seal with known quantities of moisture as shown in Table (2). Included among these were packages sealed with a military qualified organic "epoxy" properly cured (per manufacturer's instructions), die or substrate attach equal to that normally employed in microelectronics processing for die or substrate attach in 1.0 cc volume packages. The following is a list of the moisture standard samples provided:
TABLE 2. MOISTURE STANDARD CORRELATION SAMPLES

<table>
<thead>
<tr>
<th>INTERNAL VOLUME</th>
<th>(CC) ACTUAL</th>
<th>MOISTURE CONTENTS AND QUANTITIES</th>
</tr>
</thead>
<tbody>
<tr>
<td>IDEAL (Requested)</td>
<td>2000 ppmv</td>
<td>5000 ppmv</td>
</tr>
<tr>
<td>.01</td>
<td>.016</td>
<td>0</td>
</tr>
<tr>
<td>.02</td>
<td>.028</td>
<td>50</td>
</tr>
<tr>
<td>0.10</td>
<td>.094</td>
<td>0</td>
</tr>
<tr>
<td>1.00</td>
<td>.89</td>
<td>0</td>
</tr>
<tr>
<td>10.0</td>
<td>5.60</td>
<td>0</td>
</tr>
</tbody>
</table>

TOTAL 350 Pieces

During the course of this study we fabricated a total of 700+ samples for this effort. The fabrication of these samples took place at two different time intervals and in two groups of 350 pieces. They are referred to as Lot #1 (pilot devices) and Lot #2 RGA correlation specimens. The first group of devices Lot #1 (pilot devices) were used to confirm our design values at DESC suitable commercial laboratories. Lot #2 became the group we considered as the standard for our correlation studies. The study proceeded as outlined in the contractor's Statement of Work (SOW).

.02 CC Vol. With 2,000 and 5,000 PPM Moisture:

To fabricate this particular 0.02 volume package we welded a tall profile 0.175" high TO-18 header to a TO-18 base sealed in our dry box at 2000 ppmv and 5000 ppmv respectively. The moisture level in the dry box was measured with a General Eastern Hygro-M1, Dewpoint monitor.

.01, 1.0 and 5.6 CC Vol. with 5,000 PPMV Moisture:

The 0.01 cc specimens were fabricated from low profile 0.135" high TO-18 headers and bases. The 0.1 cc volumes were fabricated from two 0.135" high TO-18 caps welded together. The 1.0 cc volume specimens were fabricated by welding two (2) TO-8 caps together. We were unable to obtain suitable packages that could be handled by our welding apparatus for obtaining a 10.0 cc volume package and settled for a smaller, 5.6 cc volume. These devices were fabricated by welding two 0.750" high TO-8 caps together. All
these samples were sealed in our dry box at 5000 ppmv. The completed devices are shown in Figure (15) and (16).

$1.0 \text{ CC Vol. with 5,000 PPM Moisture and Organic Die Attach}$

In fifty (50) of the 1.0 cc volume packages, a 0.250 x 0.250 inch silicon die was mounted with Ablestik 570K, insulating preform epoxy and attached per the manufacturer's instructions. This manufacturer was deemed qualified by DESC and chosen from the document list of MIL-STD-883C, Method 5011, qualified epoxies and their manufacturers.

The names of four suppliers were given to us by DESC, they were: Ablestik, Epotech, Amicon and A-I Technology. According to DESC these were the only ones at the time of selection to conform to MIL-STD-5011. We chose to go with Ablestik because of some prior experience with the product at our hybrid facility.

We submitted the proposed use of Ablestik 570K insulating preform epoxy along with manufacturing data and specifications to Rome Laboratory as specified in CDRL, A006. Included was an independent test report prepared by Mr. James McGrath, Raytheon Co., Quincy, MA. (3)

The test design samples from Lot #1 (pilot devices) were sent out to three (3) commercial RGA laboratories. Twenty-one devices, three (3) of each type were sent to each laboratory. The results of these analyses are tabulated in Table (3) and the graph as shown in Figure (17), entitled RGA Correlation Test Results Lot #1 (Pilot Groups). The results show very good design correlation with Lab I results whereas the other two laboratories data are somewhat scattered. The data in the Table 3 does not include the results of other analyses performed on additional devices at Rome Laboratory and Lab F, those devices were submitted to Rome Laboratories for their own analysis and distribution. Based on these results, we prepared Lot #2 devices to be used as the formal 350 piece sample for the lab correlation study. Due to the depletion of the inventory of devices in Lot #1 for use as pilot devices to confirm our design values, it was necessary to seal another lot of devices for use as our formal correlation standards. Therefore, another group of three hundred fifty (350) devices were sealed and are referred to as Lot #2.

Lot #2 devices were distributed to four (4) commercial laboratories. The fifth laboratory was unable to perform any analysis due to equipment failure.

CORRELATION MOISTURE STANDARDS

Figure 15

Figure 16
<table>
<thead>
<tr>
<th>Sample Parameters</th>
<th>LAB A</th>
<th>LAB B</th>
<th>LAB C</th>
<th>LAB D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sealed In Air</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>With Organic</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Note:** This table does not include the results of the RGA analytical tests performed at Harris. These results were submitted by Home Laboratory.
RGA CORRELATION TEST RESULTS LOT #1 (PILOT GROUPS)

VOLUME CC

MEAN PPM (WATER)

LAB A
LAB D
LAB I

○ 2000 PPM SEAL LEVEL

*NOTE: ORGANIC TEST RESULTS NOT INCLUDED ON THIS GRAPH (SEE TABLE)

FIGURE 17.
We instructed all the analytical laboratories to analyze sixty percent (60%) of the devices of the total (35) sent for MIL-STD-883C, Method 1018.2, Procedure I and report on these devices before proceeding with the remaining devices. We also requested that the devices to be tested per Paragraph 3 of Method 1018.2 of MIL-STD-883C with a prebake of 24 hours and that bake time and temperatures shall be reported in the analysis report for all devices.

After careful scrutiny of all of the reported analytical data from sixty percent (60%) of the devices tested at four (4) laboratories, Rome Labs decided to end further testing and recalled the remaining devices. The recalled devices were later shipped to Rome Labs at their request.

The analytical data from Lots #1 and #2 devices was tabulated in Tables (3) and (4) and graphical representations are shown in Figures (17) and (18).

Upon receipt of the analytical results from each testing laboratory, the data was statistically analyzed to determine the mean and standard deviation. These results are tabulated along side our mean averages.

A plus (+) or minus (−) twenty (20) percent criteria allows for levels between 4000 and 6000 ppmv respectively for 5000 ppmv sealed levels and 1600 and 2400 ppmv for 2000 ppmv sealed devices.

It is interesting to note that the only analytical service to report within these boundaries was Lab I, in the Lot #1 group of analyses. The Lot #2 analytical results indicated levels far beyond the ±20% criteria with the exception of two labs who tested within specifications in the .1 to 1.0 cc volume ranges. Refer to Lot #2 data (Lab I and Lab D) test results in Figure 18.

In accordance with CLIN 001, Statement of Work Paragraph 4.1.3.3 the remaining one-hundred seventy-five (175) devices were shipped to Rome Laboratory for their inspection and acceptance.

**MANUFACTURE AND SEALING OF MOISTURE CORRELATION SAMPLES**

The samples were fabricated from various all nickel (Ni) plated T.O. series transistor packages as shown in Figures (15) and (16). The following table (Table 5) provides the dimensional data on those parts used for fabricating the correlation samples.
<table>
<thead>
<tr>
<th>SEAL PARAMETERS</th>
<th>LAB A</th>
<th>LAB B</th>
<th>LAB C</th>
<th>LAB D</th>
<th>LAB E</th>
</tr>
</thead>
<tbody>
<tr>
<td>VOLUME</td>
<td>SEAL</td>
<td>NITROGEN</td>
<td>SERIAL</td>
<td>RGA</td>
<td>ROA</td>
</tr>
<tr>
<td>0.01</td>
<td>5400</td>
<td>100</td>
<td>A01</td>
<td>85</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.01</td>
<td>5400</td>
<td>100</td>
<td>A02</td>
<td>86</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.01</td>
<td>5400</td>
<td>100</td>
<td>A03</td>
<td>87</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.02</td>
<td>5400</td>
<td>100</td>
<td>A04</td>
<td>88</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.02</td>
<td>5400</td>
<td>100</td>
<td>A05</td>
<td>89</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.02</td>
<td>5400</td>
<td>100</td>
<td>A06</td>
<td>90</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.02</td>
<td>5400</td>
<td>100</td>
<td>A07</td>
<td>91</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.02</td>
<td>5400</td>
<td>100</td>
<td>A08</td>
<td>92</td>
<td>/ I.D.</td>
</tr>
<tr>
<td>0.02</td>
<td>5400</td>
<td>100</td>
<td>A09</td>
<td>93</td>
<td>/ I.D.</td>
</tr>
</tbody>
</table>

**TABLE 4. Correlation Study Lot #2 RGA Results**
<table>
<thead>
<tr>
<th>INTERNAL VOLUME CC</th>
<th>PART DESCRIPTION AND SIZE</th>
<th>MOISTURE CONTENTS AND QUANTITIES</th>
</tr>
</thead>
<tbody>
<tr>
<td>IDEAL ACTUAL</td>
<td>COMPONENT NO. 1 HEIGHT/DIA. COMPONENT NO. 2 HEIGHT/DIA.</td>
<td>2000 5000 5000 PPMV</td>
</tr>
<tr>
<td>0.01 0.16</td>
<td>T.O.-18 CAP W.R. .135 X .175 ID T.O.-18 BASE .100 X .175 OD</td>
<td>0 50 0</td>
</tr>
<tr>
<td>0.02 0.28</td>
<td>T.O.-18 CAP W.R. .175 X .175 ID T.O.-18 BASE .100 X .175 OD</td>
<td>50 50 0</td>
</tr>
<tr>
<td>0.1 0.94</td>
<td>T.O.-18 CAP W.R. .135 X .175 ID T.O.-18 CAP .135 X .175 ID</td>
<td>0 50 0</td>
</tr>
<tr>
<td>1 0.89</td>
<td>T.O.-8 CAP W.R. .125 X .06 ID T.O.-8 CAP .125 X 0.6 ID</td>
<td>0 50 50</td>
</tr>
<tr>
<td>10 5.6</td>
<td>T.O.-8 CAP W.R. .75 X .06 ID T.O.-8 CAP .75 X 0.6 ID</td>
<td>0 50 0</td>
</tr>
</tbody>
</table>

NOTE: W.R. = WITH WELD RING

TOTAL = 350 PIECES
LOT #1 PRE-CONDITIONING

Prior to sealing the Lot #1 components were cleaned with several cleaning solutions then baked for 16 hours at 125°C (overnight). The sealing chamber (dry box) containing the welding apparatus was pre-conditioned overnight (purged with 90% dry N₂ and 10% He). The R.H. in the dry box was controlled by bubbling dry nitrogen through a cylinder containing water. The flow was adjusted to provide the required dew point in the dry box. A fan was included in the dry box to circulate the N₂ and He and H₂O atmosphere. The dew point was sampled periodically utilizing a General Eastern Co. (HYGRO-M1-PACER). The measuring instrument samples the gas and measures its dew point automatically on a mirrored surface. The dew points were monitored periodically during pre-conditioning and during sealing. In addition to these samples, we sealed some devices at ambient room condition at dew points approaching room temperature. These samples were included in the analysis to provide us with a method of "verifying" the testing of each RGA vendor. The serial number of the device, time of day and dew point were recorded for each device during sealing.

SEALING OF LOT #1 DEVICES

The parts were removed from the pre-conditioning bake in sealed containers and transferred to a remote sealing site. The parts were placed in the dry box temperature/vacuum ante chamber where upon the devices were given an additional thermal/vacuum bake for approximately one (1) hour then transferred to the sealing dry box which was pre-conditioned overnight to a dew point of -2.5°C (5000 ppmv). All the 5000 ppmv parts were sealed first then the dry box was re-conditioned by dropping the dew point to -13°C or (2000 ppmv H₂O + N₂ + He atmosphere for sealing the 0.02 cc volume, 2000 ppmv devices.

After sealing, all the devices were subjected to a helium tracer gas fine leak test and a fluorocarbon FC-77 weight gain gross leak test. Only those devices with a leak rate < 1 x 10⁻⁶ ATM cc/sec air were considered acceptable.

LOT #2 PRECONDITIONING

The Lot #2 group of parts were preconditioned similar to Lot #1, with the exception that the overnight bake was at 100°C rather than 125°C. The devices saw an additional bake at the sealing facility similar to the procedures of Lot #1 device conditioning except that we were instructed by Rome Labs to omit helium gas in our sealing
procedure. The reason for this omission was based on problems that surfaced during analysis of Lot #1 devices at two laboratories. These problems were traced to inaccurate calibration for helium. It was not known why the presence of helium caused the problems. If any gas in the ambient matrix is not assayed properly, the results for all other gases in the package ambient matrix will be skewed. In order to direct emphasis to moisture measurement correlation, Rome Laboratory requested that helium be omitted from Lot 2 samples. Rome Laboratory, upon completion of the laboratory survey, will recommend procedures to assure analytical accuracy for moisture in all normally encountered microelectronic device ambients. It was not known why the presence of helium caused the problems. This time we chose to seal all the 2000 ppmv devices first since conditioning the dry box from a low dew point to a higher dew point would hopefully solve the problem with the higher ppm levels that we experienced with the 0.02 cc "2000 ppmv" devices in the Lot #1 analyses. Again, the serial number, time of seal and dew point were recorded for each device.

**Moisture Analysis (Figures and Tables)**

The lot #1 RGA data clearly shows that Lab I provided mean values on all volume devices which were within the target values chosen (5,000 ppm). The standard deviation is also shown to be small and indicates that the parts and the test are reasonably consistent.

The lot #1 RGA data from Lab D shows a trend of higher readings for the smaller volumes (.01 and .02 cc) and a fairly even response for the .01, 1, and 5.5 cc. The standard deviation indicates more spread in the data than Lab I thus raising an issue of consistency.

The lot #1 RGA data from Lab A shows mean values similar to Lab D for the volume range of .01 to 1 cc but shows a significant departure at 5.5 cc (variation on the high side by a factor of 2 to 3 as compared to Lab I and Lab D). There is also a significant difference in the standard deviation (much more spread in data) than the others. It would appear that they have "volume effects" as well as test consistency problems.

There were three (3) devices in lot #1 (Serial #3’s 362, 379 and 386) which were intentionally sealed in a dramatically different ambient air to assure that the test houses were able to detect outliers in a population of devices. These parts were sealed in a room air ambient with a dewpoint of 15.4°C. This dewpoint converts to ~17,200 ppm. As shown in the data, each of the R.G.A. facilities (lot #1) showed high values of moisture ranging from 21,000 to 30,000 ppmv. Although there were significant differences between the test houses in the moisture values for these parts, they clearly were able to identify the devices as outliers.
The 2,000 ppmv values obtained from all three vendors were considerably higher than the target values. It was felt that this could have been attributed to the order of seal (i.e., 5,000 ppmv groups were sealed first followed by 2,000 ppmv. The 2,000 ppmv parts probably had not equilibrated). Another thought was the possibility of a minimum quantity of moisture adsorbed onto the internal surfaces of the devices in an ambient of 2,000 ppmv. As an example, if we assume that one (1) monolayer were adsorbed on the interior surface, this could amount to approximately 2,000 ppmv for the .016 cc volume (surface roughness factor of unity). Combining this value with the water entrapped in the cavity volume would result in a total moisture content of ~4,000 ppmv. For the 0.028 cc volume in an ambient of 2,000 ppmv this single monolayer would amount to a total moisture content of approximately 3,000 ppmv. In order to confirm this hypothesis a series of follow-on tests should be performed. This work is critical for small volume, low moisture level standards.

The preparation of the correlation samples has evolved over several years to a procedure which we feel is rigorous in execution and as consistent as practical. The solid nickel headers and caps are initially inspected at 10-30X magnification. Any visual anomalies is cause for rejection i.e., specks, dents, etc. This is followed by a thorough cleaning step designed to remove any residual greases, finger prints and loose particles. The parts are then rinsed, blown dry and baked for 24 hours. They are then stored in a desiccator and finally sealed in a dry box which has been stabilized at the appropriate moisture level and ambient gas content. The moisture level is monitored with a dew point instrument (General Eastern) throughout the entire seal process. All appropriate parameters are recorded (time, seal schedule, gas mix, moisture level, serial number, etc.). We have conformed to this procedure for the past 8-10 years and have found it to be effective and a sound method. This has been evidenced by the several round robin trials as well.

In spite of the divergent results reported by the laboratories in this recent correlation study, we feel that the correlation samples are consistent and are properly filled with each of the stated target values. This conclusion is based on the fact that each of the RGA test facilities were reasonably consistent within their own readings. In previous trials we had noted a great degree of scatter in the intra laboratory data whenever our correlation samples were not properly prepared.

In summary we feel that, in spite of the results of these trials, the correlation samples are sufficiently consistent in moisture content to have highlighted the problems noted with some of the RGA facilities.
CONCLUSIONS

The data obtained from the first trial seen at the three (3) laboratories strongly suggests the following:

1. RGA testing can be consistent and accurate when performed carefully and when calibrations are performed frequently.

2. The correlation samples were themselves accurate and consistent within each lot.

3. The high helium content placed in the samples for leak testing purposes in general, did not adversely affect the moisture measurements.

4. Parts sealed with an approved organic die attach material consistently indicated higher moisture levels than sister packages that did not contain them. The moisture levels of those containing the die attach material varied from 20 to 400% greater than those without it. We suspect that this difference can be attributed to the prebake period and/or the method of moisture sampling (integration vs instantaneous burst). In any event, this area needs further exploration in order to shed more light on this important issue.

The data obtained from the second trial run was somewhat mixed. The following are our conclusions to date:

1. Three (3) laboratories (Lab I, Lab G, and Lab D) had similar results for moisture content in the volume range from .01 to 1.0 cc. Lab I and Lab G followed each other out to the 5.6 cc volume, while Lab D diverged considerably (> a factor of 2 higher) similar to the results of Lab B. Lab B's data for the volumes ranging from .01 to 0.1 cc were much lower than the others but exceeded all others above that. These results all suggest that Lab I, Lab D and Lab G are consistent in volumes less than 1-2 cc and have significant variations above that. The Lab B data suggests calibration and/or test methodology problems exist in their technique.

2. At this time we do not know why the smaller volume devices (.01 to .02 cc) appear to have moisture values significantly higher than the target values. There is an ongoing investigation which is attempting to address this issue. Until a clear answer is found, conclusions which fault either the correlation samples or the RGA houses can only be based on conjecture.
A) The procedures should be consistent among the RGA facilities. In order to accomplish this, we feel it would be necessary to provide each of the facilities with hundreds of correlation samples each to be evaluated over a several month period. At the conclusion of these tests, the participants, under the auspices of Rome Laboratory, should generate a detailed step-by-step method and procedure for RGA tests.

B) In order to accomplish the above recommendation, it is necessary to produce several thousand correlation samples for distribution. At present and in the past, the only accepted mechanism was through Rome Laboratory. It would probably be more efficient if they could be fabricated directly for the RGA facilities under the guidance of Rome Laboratory or their designee.

C) Finally, there are still some unresolved issues regarding the correlation samples themselves. Although they have been reasonably consistent for the last several trials, the following area remains and should be addressed:

The absolute accuracy needs to be worked out with an independent method. We have basically relied on the dewpoint measurements in the dry box for our guide in combination with agreement from RGA facilities. This method is particularly delicate for the small volume devices (0.01 to 0.02 cc) which are vulnerable to the effects of surface to volume ratios (i.e., a single monolayer of water could have a major affect on the reading) as well as thick or thin oxides on the nickel surface. It may be prudent to fabricate the samples from a rolled gold composite to eliminate any effects due to oxidation layers. In addition, heating of the devices during the sealing process may be useful in minimizing or eliminating adsorbed moisture.
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Development of the NID System, A New Test Condition (F) for Performing Gross Leak Detection in Compliance with MIL-STD-883C, Edward Etess, Web Technology, Inc., Dallas, TX, USA.

Permissible Leak Rates and Moisture Ingress, A. DerMarderosian, Raytheon Co., Sudbury, MA, U.S.A.


ASTM STANDARDS


1.0 PURPOSE

The purpose of this test is to determine the effectiveness (hermeticity) of the seal of microelectronic and semiconductor devices with designed internal cavities.

1.1 Definitions

A. **Standard Leak Rate** - Standard leak rate is defined as that quantity of dry air at 25°C in atmospheric cubic centimeters flowing through a leak or multiple leak paths per second when the high-pressure side is at 1 atmosphere (760 mm Hg absolute) and the low-pressure side is at a pressure of not greater than 1 mm Hg absolute. Standard leak rate shall be expressed in units of atmospheric cubic centimeters per second (atm cc/s).

B. **Measured Leak Rate** - Measured leak rate (Rₘ) is defined as the leak rate of a given package as measured under specified conditions and employing a specified test medium. Measured leak rate shall be expressed in units of atmospheric cubic centimeters per second (atm cc/s, He). For the purpose of comparison with rates determined by other methods of testing, the measured leak rates must be converted to equivalent standard leak rates.

C. **Equivalent Standard Leak Rate** - The equivalent standard leak rate (L) of a given package, with a measured leak rate (Rₘ) is defined as the leak rate of the same package with the same leak geometry, that would exist under the standard conditions of 1.1A. The equation in 3.1.1.2 and 3.2.1 represents the R'R ratio and gives the equivalent standard leak rate (L) of the package with a measured leak rate (Rₘ) where the package volume and leak test conditioning parameters influence the measured value of (Rₘ). The equivalent standard leak rate shall be expressed in units of atmospheric cubic centimeters per second STD or air (atm cc/s) air.

2.0 APPARATUS

The apparatus required for the seal test shall be as follows for the applicable test conditions:
2.1. **Test Conditions A., A., and A. Tracer Gas Helium (He) Fine Leak** — Apparatus required shall consist of suitable temperature, pressure and vacuum chambers and a mass spectrometer-type leak detector preset and properly calibrated for a helium leak rate sensitivity sufficient to read measured helium leak rates of $10^{-9}$ atm cc/s, He and greater. The volume of the chamber used for leak rate measurement should be held to the minimum practical, since this chamber volume has an adverse effect on sensitivity limits. The leak detector indicator shall be calibrated using a diffusion-type calibrated standard leak at least once during every working shift. For Test Condition A₁, the following apparatus is required:

a. Fixtures, gages, meters and appropriate fittings for an enclosed environment (i.e., dry box, etc.) capable of controlling and maintaining a gaseous ambient of helium gas and dry air or nitrogen.

b. A hermetic sealing apparatus capable of sealing the device within the controlled environment.

c. A small fan for circulating the enclosed gaseous ambient.

For Test Condition A, the following apparatus is required:

a. Fixture and fittings to mate the package to be tested to the leak detector.

b. Surgical rubber gasket.

c. Apeizon grease (type M or N), perfluorocarbon fluid, or equivalent, if required to obtain seal.

2.2 **Test Condition B. Radioisotope Fine Leak** — Apparatus for this test shall consist of:

a. Radioactive tracer gas activation console.

b. Counting equipment consisting of a scintillation crystal, photomultiplier tube, preamplifier, ratemeter, and krypton-85 reference standards. The counting station shall be of sufficient sensitivity to determine through the device wall the radiation level of any krypton-85 tracer gas present within the device. The counting station shall have a minimum sensitivity corresponding to a leak rate of $10^{-2}$ atm cc/s of krypton-85 and shall be calibrated at least once every working shift using krypton-85 reference standards and following the equipment manufacturer's instruction.

---

11 Perfluorocarbons contain no chlorine or hydrogen
A tracer gas consisting of a mixture of krypton-85 and dry nitrogen. The concentration of krypton-85 in dry nitrogen shall be no less than 100 microcuries per atmospheric cubic centimeter. This value shall be determined at least once every 30 days and recorded in accordance with the calibration requirements of this standard (See 4.5.1 of MIL-STD-883).

2.3 Test Condition C - Perfluorocarbon Gross Leak

Apparatus for this test shall consist of:

a. A vacuum/pressure chamber for the evacuation and subsequent pressure bombing of devices up to 90 psia up to 23.5 hours.

b. A suitable observation container with provisions to maintain the indicator fluid at a temperature of 125° and a filtration system capable of removing particles greater than 1 micrometer in size from the fluid (Condition C1 only).

c. A magnifier with a magnification in the range between 1.5X to 30X for observation of bubbles emanating from devices when immersed in the indicator fluid (Condition C1 only).

d. Sources of Type I detector fluids, and Type II indicator fluids as specified in Table I.

e. A lighting source capable of producing at least 15 thousand foot candles in air at a distance equal to that which the most distant device in the bath will be from the source. The lighting source shall not require calibration but the light level at the point of observation (i.e., where the device under test is located during observation for bubbles), shall be verified (Condition C1 only).

f. Suitable calibrated instruments to indicate that test temperatures, pressures, and times are as specified.

g. Suitable fixtures to hold the device(s) in the indicator fluid (Condition C1 only).

h. A perfluorocarbon vapor detection system capable of detecting vapor quantities equivalent to 0.28 milligram of Type I fluid (Condition C3 only).

i. The vapor detector used for Condition C3 shall be calibrated at least once each working shift using a Type I fluid calibration source, and following the manufacturer's instructions.
3.4 Test Condition E. Penetrant Dye Gross Leak

The following apparatus shall be used for this test:

a. Ultraviolet light source with peak radiation at approximately the frequency causing maximum reflection of the dye (3650Å for Zyglo; 4935 Å for fluorescein; 5560 Å for Rhodamine B, etc.

b. Pressure chamber capable of maintaining 105 psia.

c. Solution of fluorescent dye (such as Rhodamine B, Fluorescein, Dye-check, Zyglo, Fl-50, or equivalent) mixed in accordance with the manufacturer’s specification.

d. A magnifier with a magnification in the range between 1.5X to 30X for dye observation.

2.5 Test Condition E. Weight Gain Gross Leak

Apparatus for this test shall consist of:

a. A vacuum/pressure chamber for the evacuation and subsequent pressure bombing of devices up to 90 psia up to 10 hours.

b. An analytical balance capable of weighing the devices accurately to 0.1 milligram.

c. A source of Type III detector fluid as specified in Table I.

d. A filtration system capable of removing particles greater than 1 micrometer in size from the perfluorocarbon fluid.

e. Suitable calibrated instruments to measure test pressure and times.
<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>TYPE I</th>
<th>TYPE II</th>
<th>TYPE III</th>
<th>ASTM TEST METHOD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boiling Point (°C)</td>
<td>50-95</td>
<td>140-200</td>
<td>50-110</td>
<td>D-1120</td>
</tr>
<tr>
<td>Surface Tension (Dynes/cm) at 25°C</td>
<td>&lt; 20</td>
<td></td>
<td></td>
<td>D-971 D-1331</td>
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<tr>
<td>Density at 25°C (gm/ml)</td>
<td>&gt; 1.6</td>
<td>&gt; 1.6</td>
<td>&gt; 1.6</td>
<td>D-941</td>
</tr>
<tr>
<td>Density at 125°C (gm/ml)</td>
<td></td>
<td>&gt; 1.5</td>
<td></td>
<td>D-941</td>
</tr>
<tr>
<td>Dielectric Strength (volts/mil)</td>
<td>&gt; 300</td>
<td>&gt; 300</td>
<td>&gt; 300</td>
<td>D-877</td>
</tr>
<tr>
<td>Residue (µgm/gm)</td>
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<td>&lt; 50</td>
<td>&lt; 50</td>
<td>D-2109</td>
</tr>
<tr>
<td>Appearance</td>
<td>Clear</td>
<td>Colorless</td>
<td>N/A</td>
<td></td>
</tr>
</tbody>
</table>

Perfluorocarbons contain no chlorine or hydrogen
3.0 PROCEDURE

Fine and gross leak tests shall be conducted in accordance with the requirements and procedures of the specified test condition. Testing order shall be fine leak (Condition A or B) followed by gross leak (Condition C, D, or E). When specified (See 4.0), measurements after test shall be conducted following the leak test procedures. Where the pressure specified exceeds the microcircuit package capability, alternate pressure, exposure time, and dwell time conditions may be used provided they satisfy the leak rate, pressure, time relationships which apply, and provided a minimum of 30 psia (2 atmospheres absolute) bomb pressure is applied in any case. When Test Condition A, is used, gross leak testing is not required. However, A, shall not be used in lieu of the required seal testing of lidded packages. When batch testing (more than one device in the leak detector at one time) is used in performing Test Condition A or B and a reject condition occurs, it shall be noted as a batch failure. Each device may then be tested individually for acceptance if all devices in the batch are retested within one hour after removal from the tracer gas pressurization chamber. For Condition C, only, devices that are batch tested, and indicate a reject condition, may be retested individually one time using the procedure of 3.3.3.1 herein, except that repressurization is not required if the devices are immersed in detector fluid within 20 seconds after completion of the first test, and they remain in the bath until rejected.

3.1 Test Condition A, A, or A, Tracer Gas (He) Fine Leak

Test condition A, is a "backfill" method which seals a specified quantity of helium tracer gas in packages with an internal cavity volume $\geq 0.2$ cc. This method replaces the "fixed" method and eliminates the long pressurization times required to detect leaks, in larger volume packages, near the limit of acceptability. Test Condition A, is a "flexible" method that allows the variance of test conditions in accordance with the equation of 3.1.1.2 to detect the specified equivalent standard leak rate (L) at a predetermined leak rate (R). Test Condition A, is a method that will detect the required measured leak rate (R,) of an unsealed package.
3.1.1 Test Condition A, Backfilled Method

This test is an alternate method for $A_2$ which may only be used for packages with an internal cavity volume $\geq 0.2$ cc. The devices shall be sealed in a dry gas ambient mixture of $37\%$ (by volume) helium with the balance (unless otherwise specified) of air or nitrogen. After seal and removal from the specified ambient, the packages shall be tested with a mass spectrometer type leak detector. The parts shall be tested within the time as specified in Table II. These maximum dwell times are provided to assure detection of leakage up to $1 \times 10^{-5}$ ATM cc/sec air and provide overlap with gross leak tests used in this method.

**TABLE II**

MAXIMUM ALLOWABLE TIMES BETWEEN MEASUREMENTS FOR CONDITION A.

<table>
<thead>
<tr>
<th>VOLUME OF PACKAGE (V) IN CM$^3$</th>
<th>MAXIMUM DWELL TIME (HOURS)</th>
<th>REJECT LIMIT ($R_i$) (ATM CC/S, HE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\geq 0.2 - &lt; 0.5$</td>
<td>10</td>
<td>$8 \times 10^{-9}$</td>
</tr>
<tr>
<td>$\geq 0.5 - &lt; 1.0$</td>
<td>30</td>
<td>$8 \times 10^{-9}$</td>
</tr>
<tr>
<td>$\geq 1.0 - &lt; 2.0$</td>
<td>70</td>
<td>$8 \times 10^{-9}$</td>
</tr>
<tr>
<td>$\geq 2.0 - &lt; 4.0$</td>
<td>120</td>
<td>$8 \times 10^{-9}$</td>
</tr>
<tr>
<td>$\geq 4.0 - &lt; 10.0$</td>
<td>300</td>
<td>$8 \times 10^{-9}$</td>
</tr>
<tr>
<td>$\geq 10.0 - &lt; 20.0$</td>
<td>700</td>
<td>$8 \times 10^{-9}$</td>
</tr>
<tr>
<td>$\geq 20.0 - &lt; 40.0$</td>
<td>1200</td>
<td>$8 \times 10^{-9}$</td>
</tr>
</tbody>
</table>

The value of $37\%$ was chosen to simplify leak rate readings; in this case, $37\%$ helium leak readings ($R$) are equivalent to the standard leak rate ($L$). This is shown in the following equation.

$$L = \frac{R_i \left( \frac{100}{\text{He}\%} \right)}{2.7} \quad \text{OR} \quad R_i = \frac{2.7L}{\left( \frac{100}{\text{He}\%} \right)}$$

Where $L$ = Equivalent Standard Leak Rate (ATM CC/S, Air)

$R_i$ = Measured Leak Rate (ATM CC/S, He)

He% = Volume % of Helium gas sealed into the package under standard conditions (20°C, ±5°C at 14.7 ±3.0 psia)
Other percentages of helium may be used, provided the reject criteria of \(1 \times 10^{-2}\) ATM cc/sec, air (L) is maintained and detectable.

Parts that are acceptable may be retested at future times without the need for pressurization in helium (e.g., as in \(A_2\)) provided that the time between such tests does not exceed the maximum dwell times specified in Table II.

If the maximum dwell time as specified in Table II is exceeded, then the part(s) shall be subjected to Test Condition \(A_2\) for a period of time equal to 10% of the excess dwell period (one (1) hour minimum) at 2 atmospheres absolute. This will assure detection of leakage (L) from \(1 \times 10^{-5}\) ATM cc/sec to \(1 \times 10^{-3}\) ATM cc/sec. The reject limit of Table II shall apply on such retested material. The maximum dwell time after release from the pressure bomb for these retested devices, shall be 4 hours.

The reject limit \(R_i\) of \(8 \times 10^{-9}\) ATM cc/sec, He is intentionally slightly less than \(1 \times 10^{-7}\) ATM cc/sec, He to provide a small guardband in the dwell times allowed in order to assure detection of leaks equal to \(1 \times 10^{-5}\) ATM cc/sec (i.e., since an \(R_i\) value of \(1 \times 10^{-7}\) ATM cc/sec, He is equal to \(1 \times 10^{-5}\) ATM cc/sec, air the dwell times allowed on the smaller volume packages would show a very slight decrease in the measured value \(R_i\)). Table II reject limit \(R_i\) is based on 37% He backfilled in the cavity. The reject limit for other percentages of helium sealed into the cavity can be calculated by using the following expression:

\[
R_i = 8 \times 10^{-9} \left[ \frac{\text{He}\%}{37} \right]
\]

Where:

- \(R_i\) = Mass spectrometer reject limit in ATM cc/sec, He
- \(\text{He}\%\) = Volume % of helium sealed into the cavity.

NOTE: The maximum allowable dwell times between measurements remain the same as those shown in Table II.
3.1.1.2 Test Condition A, Flexible Method

The completed device(s), shall be placed in a sealed chamber which is then pressurized with a tracer gas of 100 ±0, -5 percent helium for the required time and pressure. The pressure shall then be relieved and each specimen transferred to another chamber or chambers which are connected to the evacuating system and a mass-spectrometer-type leak detector. When the chamber(s) is evacuated, any tracer gas which was previously forced into the specimen will thus be drawn out and indicated by the leak detector as a measured leak rate \( R_i \). The number of devices removed from pressurization for leak testing shall be limited such that the test of the last device can be completed within the chosen value of dwell time \( t_2 \).

Values for bomb pressure exposure time, and dwell time shall be chosen such that actual measured tracer gas leak rate \( R_i \) readings obtained for the devices under test (if defective) will be greater than the minimum detection sensitivity capability of the mass spectrometer. The devices shall be subjected to a minimum of 2 atmospheres absolute of helium atmosphere. If the chosen dwell time \( t_2 \) is greater than 60 minutes, graphs shall be plotted to determine an \( R_i \) value which will assure overlap with the selected gross leak test condition. The chosen values, in conjunction with the value of the internal volume of the device package to be tested and the maximum equivalent standard leak rate \( L \) limit (as shown below or as specified in the applicable acquisition document), shall be used to calculate the measured leak rate \( R_i \) limit using the following equation:

\[
R_i = 2.7LP_0 \left\{ 1 - \frac{2.7Lt_1}{V} \right\} \left\{ 1 - \frac{2.7Lt_2}{V} \right\}
\]

Where:

- \( R_i \) = The measured leak rate of tracer gas (He) through the leak in atm cc/s He.
- \( L \) = The equivalent standard leak rate in atm cc/s; air.
- \( P_0 \) = The pressure of exposure in atmospheres absolute.
- \( t_1 \) = The time of exposure to \( P_0 \) in seconds.
- \( t_2 \) = The dwell time between release of pressure and leak detection, in seconds.
- \( V \) = The internal volume of the device package cavity in cubic centimeters.
3.1.1.2.1 Failure Criteria

Unless otherwise specified, devices shall be rejected if the equivalent standard leak rate (L) exceeds $1 \times 10^{-6}$ ATM cc/s air.

To minimize the effects of surface sorption of tracer gas, it is permissible to bake devices after pressurization for a period of 10 to 15 minutes at $100 \pm 20^\circ$C prior to testing in the mass spectrometer. The bake period shall be added to the dwell period ($t_2$) in calculations for the total dwell time.

The constant 2.7 in the equation is the calculated value of $\left(\frac{M_A}{M}\right)^{1/2}$ in the complete Howl and Mann equation shown below:

$$R_1 = \frac{LP_0}{P_0} \left(\frac{M_A}{M}\right)^{1/2} \left\{ 1 - \left[ \frac{Lt_1}{VP_0} \left(\frac{M_A}{M}\right)^{1/2} \right] \right\} e^{-\left[ \frac{Lt_2}{VP_0} \left(\frac{M_A}{M}\right)^{1/2} \right]}

Where:

- $R_1$, $L$, $P_0$, $t_1$, $t_2$, and $V$ are defined above in the abbreviated version and
- $M_A = $ The molecular weight of air in grams (28.7)
- $M = $ The molecular weight of the tracer gas (helium) in grams (4)
- $P_0 = $ The atmospheric pressure in atmospheres absolute (1)

3.1.2 Test Condition A, Procedure Applicable to the Unsealed Package Method

The fixture and fittings of 2.1 Test Condition A, shall be mounted to the evacuated port of the leak detector. Proof of fixturing integrity shall be verified by sealing a flat surfaced metal plate utilizing the gasket of 2.1 (and grease or fluid of 2.1 if required to obtain seal) and measuring the response of the leak test system. Testing shall be performed by sealing the package(s) to the evacuation port and the package cavity evacuated to 0.1 torr or less. Care shall be
taken to prevent contact of grease with package (seal ring not included), to avoid masking leaks. The external portion of the package shall be flooded with Helium gas either by the use of an envelope to obtain essentially a 100% helium atmosphere or a spray gun, set at a pressure of 45 psi and a flow rate of at least 1 STD. cu. ft./min. The package shall be tested at these conditions for 10 seconds minimum for both methods.

3.1.2.1 **Failure Criteria**

Unless otherwise specified, devices shall be rejected if the measured leak rate \( R_i \) exceeds \( 1 \times 10^{-8} \text{ atm cc/s He} \).

3.2 **Test Condition B. Radioisotope Fine Leak Test**

3.2.1 **Activation Parameters**

The activation pressure and soak time shall be determined in accordance with the following equation:

\[
R_i = SKVP_E \left\{ 1 - e^{-\left[ \frac{.58Lt_1}{V} \right]} \right\} \left\{ e^{-\left[ \frac{.58Lt_2}{V} \right]} \right\} \tag{1}
\]

The parameters of equation (1) are defined as follows:

\( R_i \) = Counts per minute above the ambient background after activation if the device leak rate were exactly equal to \( L \). This is the reject count above the background of both the counting equipment and the component, if it has been through prior radioactive leak tests.

\( S \) = The specific activity, in microcuries per atmospheric cubic centimeter, of the Krypton-85 tracer gas in the activation system.

\( K \) = The overall counting efficiency of the scintillation crystal in counts per minute per microcurie of Krypton-85 in the internal void of the specific component being evaluated. This factor depends upon component configuration and dimensions of the scintillation crystal. The counting efficiency shall be determined in accordance with 3.2.2.
t₁ = Soak time, in seconds, that the devices are to be activated.

t₂ = The dwell time between release of pressure and leak detection, in seconds.

P₀ = The activation pressure in atmospheres absolute.

L = Equivalent standard leak rate in ATM cc/sec; air.

V = The internal volume of the device package cavity in cubic centimeters.

3.2.2 Determination of Counting Efficiency (K)

The counting efficiency (K) of equation (1) shall be determined as follows:

a. Five representative units of the device type being tested shall be tubulated and the internal void of the device shall be backfilled through the tubulation with a known volume and known specific activity of Krypton-85 tracer gas and the tubulation shall be sealed off.

b. The counts per minute shall be directly read in the shielded scintillation crystal of the counting station in which the devices are read. From this value, the counting efficiency, in counts per minute per microcurie, shall be calculated.

3.2.3 Evaluation of Surface Sorption

All device encapsulations consisting of glass, metal and ceramic or combinations thereof, including coatings and external sealants, shall be evaluated for surface sorption of Krypton-85 before establishing the leak test parameters. Representative samples of the questionable material shall be subjected to the predetermined pressure and time conditions established for the device configuration as specified by 3.2.1. The samples shall then be counted every 10 minutes, with count rates noted, until the count rate becomes asymptotic with time. (This is the point in time at which surface sorption is no longer a problem). This time lapse shall be noted and shall determine the "wait time" specified in 3.2.4.
3.2.4 Procedure

The devices shall be placed in a radioactive tracer gas activation tank. The activation chamber may be partially filled with inert material to reduce pumpdown time. The tank shall be evacuated to 0.5 torr minimum. The devices shall be subjected to a minimum of 2 atmospheres absolute pressure of Krypton-85/dry nitrogen mixture. Actual pressure and soak time shall be determined in accordance with 3.2.1. The $R_1$ value in counts per minute shall not be less than 600 above background. The Krypton-85/dry nitrogen gas mixture shall be evacuated to storage until 0.5 to 2.0 torr pressure exists in the activation tank. The storage cycle shall be completed in 3 minutes maximum as measured from the end of the activation cycle or from the time the activation tank pressure reaches 60 psia if a higher bombing pressure is used. The activation tank shall then immediately be backfilled with air (air wash). The devices shall then be removed from the activation tank and leak tested within 1 hour after gas exposure with a scintillation-crystal-equipped counting station. Device encapsulations that come under the requirements of 3.2.3 shall be exposed to ambient air for a time not less than the "wait time" determined by 3.2.3. This exposure shall be performed after gas exposure but before determining leak rate with the counting station. Device encapsulations that do not come under the requirements of 3.2.3 may be tested without a "wait time". (The number of devices removed from pressurization for leak testing shall be limited such that the test of the last device can be completed within 1 hour). If the dwell time is greater than 1 hour, graphs shall be plotted to determine an $R_1$ value which will assure overlap with the selected gross leak test condition.

NOTE: CAUTION. Discharge of Krypton-85 into the atmosphere must not exceed limits imposed by local and Federal regulations.

3.2.5 Failure Criteria

Unless otherwise specified, devices shall be rejected if the equivalent standard leak rate ($L$) exceeds $1 \times 10^{-6}$ ATM cc/sec; air.

3.2.6 Personnel Precautions

Federal, some state and local governmental regulations require a license for the possession and use of Krypton-85 leak test equipment. In the use of radioactive gas, these regulations and their maximum permissible exposure and tolerance levels prescribed by law should be observed.
NOTE: FOR TEST CONDITION A, (3.1.1.2) and B (3.2):

It is permissible to release the chamber pressure periodically for less than 15 minutes at a time, in order to insert or remove devices. This chamber "downtime" must, however, be accounted for in the total pressurization period \( t_1 \). This allowance is made in order to provide for more efficient use of bombing chambers.

3.3 Test Condition C, or C2, Perfluorocarbon Gross Leak

Test Condition C, is a fixed method with specified conditions that will ensure overlap with the fine leak test. Test Condition C, has been replaced by C2. Test Condition C2, which also assures overlap with the fine leak test, is a fixed method that uses a vapor detection system instead of an indicator bath.

3.3.1 Procedure Applicable to Fixed (C,) Method

The devices shall be placed in a vacuum/pressure chamber and the pressure reduced to 50 torr or less and maintained for 30 minutes minimum, except for devices with an internal volume \( > 0.1 \text{ cm}^3 \) this vacuum cycle may be omitted. A sufficient amount of Type I detector fluid shall be admitted to cover the devices. When the vacuum cycle is performed, the fluid will be admitted after the minimum 30 minute period, but before breaking the vacuum. The devices shall then be pressurized in accordance with Table IV. When the pressurization period is complete, the pressure shall be released and the devices removed from the chamber without being removed from a bath of detector fluid for greater than 20 seconds. A holding bath may be another vessel or storage tank. When the devices are removed from the bath they shall be dried for 2 ±1 minutes in air prior to immersion in type II indicator fluid, which shall be maintained at 125°C ±5°C. The devices shall be immersed with the uppermost portion, at a minimum depth of 2 inches below the surface of the indicator fluid, one at a time or in such a configuration that a single bubble from a single device out of a group under observation may be clearly observed as to its occurrence and source. Under no circumstances shall more than 4 devices be tested at one time. The devices shall be observed against a dull, non-reflective black background through the magnifier, while illuminated by the lighting source, from the instant of immersion until, expiration of a 30 second minimum observation period, unless rejected earlier.

3.3.1.1 Test Condition C2, Fixed Method

Allowable fixed method conditions shall be as shown in Table III, herein.
TABLE III. CONDITION C, AND C, PRESSURIZATION CONDITIONS.

<table>
<thead>
<tr>
<th>PRESSURE PSIA</th>
<th>MINIMUM PRESSURIZATION TIME (HOUR)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>C₁</td>
</tr>
<tr>
<td></td>
<td>23.5</td>
</tr>
<tr>
<td>45</td>
<td>8</td>
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<td>105</td>
<td>0.5</td>
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</tbody>
</table>

3.3.2 Failure Criteria

A definite stream of bubbles or two or more large bubbles originating from the same point shall be cause for rejection.

3.3.3 Test Condition C, Perfluorocarbon Vapor Detection

3.3.3.1 Procedure

The devices shall be placed in a vacuum/pressure chamber and the pressure reduced to 50 torr or less and maintained for 30 minutes minimum. A sufficient amount of Type I detector fluid shall be admitted to the pressure chamber to cover the devices. The fluid shall be admitted after the 30 minute minimum vacuum period but before breaking the vacuum. The devices shall then be pressurized in accordance with Table III. Upon completion of the pressurization period, the pressure shall be released, the devices removed from the pressure chamber without being removed from a bath of detector fluid for more than 20 seconds and then retained in a bath of perfluorocarbon fluid. When the devices are removed from the fluid they shall be air dried for a minimum of 20 seconds and a maximum of 5 minutes prior to the test cycle. If the type I detector fluid has a boiling point of less than 80°C, the maximum drying time shall be 3 minutes.
The devices shall then be tested with a perfluorocarbon vapor detector that is calibrated in accordance with 2.3h and 2.3i. "Purge" time shall be in accordance with Table IV. Test time shall be a minimum of 3.5 seconds (unless the device is rejected earlier) with the perfluorocarbon vapor detector purge and test chambers at a temperature of 125 ±5°C, or 2.5 seconds minimum with the purge and test chambers at a temperature of 150 ±5°C.

NOTE: Air dry, purge and test limits for each device shall be complied with in all cases, including stick to stick handling.

NOTE: Test temperature shall be measured at the chamber surface that is in contact with the device being tested.

3.3.3.2 Failure Criteria

A device shall be rejected if the detector instrumentation indicates more than the equivalent of 0.28 milligrams of type I detector fluid in accordance with Table I.

TABLE IV. PURGE TIME FOR CONDITION C.

<table>
<thead>
<tr>
<th>PACKAGE WITH INTERNAL FREE VOLUME (CM³)</th>
<th>PURGE TIME (SECONDS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;0.01</td>
<td>≤ 5</td>
</tr>
<tr>
<td>≥0.01 &lt;0.10</td>
<td>≤ 9</td>
</tr>
<tr>
<td>≥0.10</td>
<td>&lt;13</td>
</tr>
</tbody>
</table>

NOTE: Maximum purge time can be determined by cycling a device with a 0.02 to 0.05 inch hole and measuring the maximum purge time that can be used without permitting the device to escape detection during the test cycle.

3.3.4 Precautions

The following precautions shall be observed in conducting the perfluorocarbon gross leak test:

a. Perfluorocarbon fluids shall be filtered through a filter system capable of removing particles greater than 1 micrometer prior to use. Bulk filtering and storage is permissible. Liquid which has accumulated observable quantities of
particulate matter during use shall be discarded or reclaimed by filtration for re-use. Precaution should be taken to prevent contamination.

b. Observation container shall be filled to assure coverage of the device to a minimum of 2 inches.

c. Devices to be tested should be free from foreign materials on the surface, including conformal coatings and any markings which may contribute to erroneous test results.

d. A lighting source capable of producing at least 15 thousand foot candles in air at a distance equal to that which the most distant device in the bath will be from the source. The lighting source shall not require calibration but the light level at the point of observation (i.e., where the device under test is located during observation for bubbles) shall be verified.

e. Precaution should be taken to prevent operator injury due to package rupture or violent evolution of bomb fluid when testing large packages.

3.4 Test Condition D. Penetrant Dye Gross Leak

This test shall be permitted only for destructive verification of devices (See 3.6). The pressure chamber shall be filled with the dye solution to a depth sufficient to completely cover all devices. The devices shall be placed in the solution and the chamber pressurized at 105 psia minimum for 3 hours minimum. For device packages which will not withstand 105 psia, 60 psia minimum for 10 hours may be used. The devices shall then be removed and carefully washed, using a suitable solvent for the dye used, followed by an air-jet dry. The devices shall then be immediately examined under the magnifier using an ultraviolet light source of appropriate frequency.

3.4.1 Failure Criteria

Any evidence of dye penetration into the device cavity shall constitute a failure.

3.5 Test Condition E. Weight Gain Gross Leak

3.5.1 Each device shall be weighed and the initial weight recorded or the devices may be categorized into cells as follows. Devices having a volume of <0.01 cc shall be categorized in cells of 0.5 milligram increments and devices with a volume ≥0.01 cc shall be categorized in cells of 1.0 milligram increments. The devices shall be placed in a vacuum/pressure chamber and the pressure reduced to 50 torr or less and maintained for 1 hour except that for devices with an
internal cavity volume >0.1 cc, this vacuum cycle may be omitted. A sufficient amount of Type III detector fluid shall be admitted to the pressure chamber to cover the devices. When the vacuum cycle is performed, the fluid shall be admitted after the 1 hour period but before breaking the vacuum. The devices shall then be pressurized to 75 psia minimum except that 90 minimum psia shall be used when the vacuum cycle has been omitted. The pressure shall be maintained for 2 hours minimum. If the devices will not withstand the 75 psia test pressure, the pressure may be lowered to 45 psia minimum with the vacuum cycle and the pressure maintained for 10 hours minimum.

Upon completion of the pressurization period, the pressure shall be released and the devices removed from the pressure chamber and retained in a bath of the perfluorocarbon fluid. When the devices are removed from the fluid they shall be air dried for 2 ±1 minutes prior to weighing. Transfer the devices singly to the balance and determine the weight or weight category of each device. All devices shall be tested within 4 minutes following removal from the fluid. The delta weight shall be calculated from the record of the initial weight and the post weight of the device. Devices which were categorized shall be separated into two groups, one group which shall be devices which shifted one cell or less and the other group which shall be devices which shifted more than one cell.

3.5.2 Failure Criteria

A device shall be rejected if it gains 1.0 milligram or more and has an internal volume of <0.01 cm³ and 2.0 milligrams or more if the volume is > 0.01 cm³. If the devices are categorized, any device which gains enough weight to cause it to shift by more than one cell shall be considered a reject. A device which loses weight of an amount which if gained would cause the device to be rejected may be retested after it is baked at 125°C for a period of 8 hours.

3.6 Retest

Devices which fail gross leak (Test Condition C or E) may be retested destructively. If the retest shows a device to pass, that was originally thought to be a failure, then the device need not be counted as a failure in the accept number of LTPD calculations. Single devices which fail fine leak (Test Condition A₁, A₂, A, or B) shall not be retested for acceptance unless specifically permitted by the applicable acquisition document. Where fine leak retest is permitted, the entire leak test procedure for the specified test condition shall be repeated. That is, retest of a single failed device consisting of a second observation on leak detection without a re-exposure to the tracer fluid or gas under the specified test condition shall not be permissible under any circumstances. Preliminary measurement to detect residual tracer gas is advisable before any test.

A₁, A₂, or B only.
4.0 SUMMARY

The following details shall be specified in the applicable acquisition document:

a. Test condition letter when a specific test is to be applied (See 3).

b. Accept or reject leak rate for Test Condition A or B when other than the accept or reject leak rate specified herein applies (See 3.1.1, 3.1.1.2, 3.1.2, and 3.2.4).

c. Where applicable, measurements after test (See 3).

d. Retest acceptability for Test Conditions A and B (See 3.6).

e. Order of performance of fine and gross if other than fine followed by gross (See 3).

f. Where applicable, the device package pressure rating shall be specified if that rating is less than 75 psia.
SUBJECT: RADC MIL-STD-883C, Method 1014 Questionnaire

Dear

INTRODUCTION

The continual changes in the state-of-the-art in microelectronics has placed a high priority on the efforts to maintain reliability in the military electronics industry. In an effort to maintain this level of reliability consciousness, the Air Force Systems Command and Rome Air Development Center, is undertaking a review of a current test method included in MIL-STD-883C, Method 1014, "Seal Test". The scope of the concern is to reduce the incidence of ambient induced failures by improving the present MIL-STD Test Method 1014.

Raytheon Co., under contractual agreement with RADC, has undertaken the task of providing a detailed study to investigate the current version of MIL-STD-883C, Method 1014, and explore and investigate new test methods for incorporation in a revision of the test method at some later date.

In order to provide as much information as possible to the government, we are soliciting the microelectronics industry, including I.C. vendors and users as well as manufacturers of test equipment, to help furnish us information, pertinent to Method 1014 (Seal).

We have compiled a brief questionnaire which we are including that will aid us in this effort. We are requesting any relevant information, methods, new or old along with recommendations which may be beneficial to this study and the community at large.

It is the intent of this study to review replies from as many inputs and sources as possible, and to present our findings to the government. Recommendations from this study will be submitted to the JEDEC Committee (JC-13) for possible adoption into MIL-STD-883C.
Your cooperation and time to prepare this response is greatly appreciated. We will respect the confidentiality of your replies and ask that you fill out only whatever information you feel is proper and approved by your company. Please do not submit any proprietary data or information.

Sections 1, 2 and 4 questions are directed to package manufacturers and users. Section 3 questions are directed to equipment manufacturers.

If you have any questions, please contact us during normal business hours at telephone number (508) 440-2791 or our 24 hour FAX service at (508) 440-3920. Thank you for your cooperation.

Sincerely yours,

A. DerMarderosian
Instrumentation Section Manager
Environmental Engineering Dept.
Sudbury, MA 01776
PLEASE RETURN FORM IN THE ENCLOSED SELF-ADDRESSED, STAMPED ENVELOPE AND MAIL TO:

RAYTHEON CO.
C/O A. DERMARDEROSIAN, BOX 1F6
EQUIPMENT DIVISION
528 BOSTON POST ROAD
SUDBURY, MA 01776

RESPONDENT'S NAME:

JOB POSITION:

DATE:

COMPANY:

ADDRESS:

PHONE #:
**SECTION 1.**

**QUESTIONNAIRE**

1. Do you perform post production hermetic seal testing, or have performed for you?
   - Yes ___  No ___
   If yes, please continue.

2. What kinds of devices are tested? Please describe product types, sealing type and quantities.  (See Matrix)

<table>
<thead>
<tr>
<th>QUESTION 2 MATRIX</th>
<th>SEAL METHOD</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SOLDIER</td>
</tr>
<tr>
<td>1. Platform</td>
<td></td>
</tr>
<tr>
<td>a. TO-Series</td>
<td></td>
</tr>
<tr>
<td>Round</td>
<td></td>
</tr>
<tr>
<td>b. Square or</td>
<td></td>
</tr>
<tr>
<td>Rectangular</td>
<td></td>
</tr>
<tr>
<td>2. All Metal Package</td>
<td></td>
</tr>
<tr>
<td>a. All Metal Flat Pack</td>
<td></td>
</tr>
<tr>
<td>b. Modular Sidewall</td>
<td></td>
</tr>
<tr>
<td>c. Butterfly</td>
<td></td>
</tr>
<tr>
<td>d. Vertical Sidewall</td>
<td></td>
</tr>
<tr>
<td>e. Solid Sidewall</td>
<td></td>
</tr>
<tr>
<td>f. Dihedral</td>
<td></td>
</tr>
<tr>
<td>g. Uniwall</td>
<td></td>
</tr>
<tr>
<td>h. Unibody</td>
<td></td>
</tr>
<tr>
<td>3. Glass Flatpack/DIP</td>
<td></td>
</tr>
<tr>
<td>4. Cerdip/Cerpack</td>
<td></td>
</tr>
<tr>
<td>5. All Ceramic Package</td>
<td></td>
</tr>
<tr>
<td>a. Leads</td>
<td></td>
</tr>
<tr>
<td>b. Leadless</td>
<td></td>
</tr>
<tr>
<td>6. Other - Please Describe</td>
<td></td>
</tr>
</tbody>
</table>
3. Which hermetic seal test specification do you use?

(a) MIL-STD-202F, Method-112E
(b) MIL-STD-750C, Method 1071.4
(c) MIL-STD-883C, Method 1014.8
(d) ASTM
(e) Other

If ASTM, please specify method designation number. If other specifications are used, please specify:

ASTM Number:

Other Specifications/Methods:

4. If MIL Standards or ASTM hermeticity specifications/methods are used, which specific tests within Table 1 (next page) do you perform and why? (Please check appropriate boxes on next page)

Please explain:
<table>
<thead>
<tr>
<th>Test Method</th>
<th>Test Description</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Method 112E</td>
<td>Gross Leak</td>
<td>Cond. A. Bubble Test: Mineral Oil-Peanut Oil</td>
</tr>
<tr>
<td>Method 1071.4</td>
<td>Gross Leak</td>
<td>Cond. B. Radiosotope Dry Gross Leak Test (Mineral Oil-Krypton 85)</td>
</tr>
<tr>
<td>Method 1014</td>
<td>Gross Leak</td>
<td>Cond. D. Dye Penetrant Test</td>
</tr>
<tr>
<td>Method 1014</td>
<td>Gross Leak</td>
<td>Cond. E. Weight Gain Test</td>
</tr>
<tr>
<td>Method 1014</td>
<td>Gross Leak</td>
<td>Cond. F. Fluorocarbon Vapor Detection Test</td>
</tr>
</tbody>
</table>

**Fine Leak**

<table>
<thead>
<tr>
<th>Test Method</th>
<th>Test Description</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>F780-77</td>
<td>Method A - Hot Bubble (Immersion)</td>
<td>Method A - Hot Bubble (Immersion)</td>
</tr>
<tr>
<td>F780-81</td>
<td>Method B - Backfill and Immersion</td>
<td>Method B - Backfill and Immersion</td>
</tr>
<tr>
<td>F780-81</td>
<td>Method C - Vacuum Bubble</td>
<td>Method C - Vacuum Bubble</td>
</tr>
</tbody>
</table>

**HIL-STS and ASTM Tests**

- Tracer Gas Tests
- Procedure I and II for parts with evacuation tubes
- Procedure III for sealed parts back pressurization, II/A and IIIC-Helium, II/A-Krypton 85
5. What percentage failures do you experience with the following seal type:
   Ceramic to glass  
   Ceramic to metal  
   Glass to metal  
   Metal to metal  
   Other, (Define)  

6. How many parts are you capable of leak testing per day?
   Fine ___  Gross ___  Qty/Day  

7. What is your opinion of the tests you perform? Do you see any weaknesses or inconsistencies in the tests you perform? Include any problems you may be experiencing. Please answer in detail and attach reports, relevant data, etc. if available.
8. Do you see inconsistencies or inaccuracies in the present MIL-STD-883C, 1014 Test Method, if so, where? What method do you consider best for large volume packages, i.e., hybrids, VLSI, etc?

9. Are there any other tests that you feel should be considered for inclusion in Test Method 1014 of MIL-STD-883C?  
   Yes ___  No ___  
   If yes, describe in detail.
SECTION 2.
One-Way Leaker Phenomena

The one-way leaker phenomena is somewhat of an enigma in the leak testing community. It is not known how many people are aware of the problem or whether they have addressed the problem in their leak testing procedures. Basically, the one-way leaker phenomena functions similar to a check valve, allowing a leak to pass only in one direction. It is usually very sensitive to pressure and/or temperature. It can allow gas or a liquid to become entrapped in a device. These leakers have in the past been identified mainly by destructive residual gas analysis tests. Another aim of this study is to focus on this phenomenon.

1. Are you familiar with the one-way leaker phenomenon?

Yes _____ No _____

If no, continue on to Section 3.

If yes, do you use or know of a test or tests that can identify these leakers? Please explain:

2. Do you have any sample parts which were determined to be one-way leakers?

Yes _____ No _____

Do you have any samples you wish to contribute to this study?

Yes _____ No _____

3. Do you have any detailed reports describing test results and analyses on one-way leakers that you would be willing to contribute to this study.

Yes _____ No _____
SECTION 3.

EQUIPMENT MANUFACTURERS

1. Do you perform leak testing?  
   __________ No  
   __________ Yes - Please detail the types of test used.

2. Do you provide leak test training to your customers?  
   __________ No  
   __________ Yes - What type of tests - please specify.

3. Can you identify changes in hermeticity test specifications which would enable you to produce better test equipment?  
   __________ No  
   __________ Yes - Explain
4. Do you know of other methods of leak testing which you would recommend for incorporation into Test Method 1014 of MIL-STD-883?

[ ] No
[ ] Yes - Explain

5. Do you have technical reports to support your recommendation?

[ ] No
[ ] Yes - Please enclose a copy.
FAILURE ANALYSIS

1. Do you perform failure analysis on parts which fail leak tests?
   
   No
   Yes

2. If your answer to (1) was Yes, please describe the locations of leak sites and percent of each type e.g.: To-Series, 85% glass to metal seal, 10% weld area and 5% cracked metal.

3. Describe the techniques used to determine the locations of the leak sites.
APPENDIX C
QUESTIONNAIRE SUMMARY

The following questionnaire was delivered by mail to 101 individuals previously mentioned. There were 32 responses collected and condensed on to a copy of the same questionnaire. Some responses have been paraphrased for brevity, tables were drawn to reflect the scope of responses and in a particular instance, ranges of quantities and volumes are listed in lieu of exact numbers.

The responses, with the above exceptions, are the true transcriptions of the respondent's reply to the question asked. Every effort has been made to reflect the response of each respondent on its own merit. The condensing of this information gives one a sense that the community at large is at best, not particularly enthusiastic nor interested in learning about the technical issues associated with hermeticity testing and as such relegate its execution to production throughput concerns. In short, it's just another menial test to perform.
### SECTION 1.
**QUESTIONNAIRE**

1. **Do you perform post production hermetic seal testing, or have performed for you?**
   - Yes 29
   - No 3

   If yes, please continue.

2. **What kinds of devices are tested? Please describe product types, sealing type and quantities. (See Matrix)**

<table>
<thead>
<tr>
<th>QUESTION 2 MATRIX</th>
<th>SEAL METHOD</th>
<th>SOLDER</th>
<th>WELD</th>
<th>GLASS</th>
<th># TESTED</th>
<th>% FINE LEAK</th>
<th>% GROSS LEAK</th>
<th>INT. CAVITY VOL. CC.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Platform</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. TO-Series Round</td>
<td>1</td>
<td>9</td>
<td>1</td>
<td>150</td>
<td>20</td>
<td>.25</td>
<td>.01</td>
<td>1</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2. All Metal Package</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>a. All Metal Flat Black</td>
<td>1</td>
<td>2</td>
<td>1</td>
<td>150</td>
<td>2</td>
<td>.05</td>
<td>&lt;2.0</td>
<td>&lt;1</td>
</tr>
<tr>
<td>b. Modular Sidewall</td>
<td>-</td>
<td>2</td>
<td>-</td>
<td>-</td>
<td>3</td>
<td>&lt;.001</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3. Glass Flatpack/DIP</td>
<td></td>
<td>1</td>
<td>1</td>
<td>2</td>
<td>1.5-1000</td>
<td>0.4&lt;-2</td>
<td>1</td>
<td>.15</td>
</tr>
<tr>
<td>4. Cerdip/Cerpack</td>
<td>4</td>
<td>2</td>
<td>4</td>
<td>1-10,000</td>
<td>.001&lt;-2</td>
<td>.001&lt;-1</td>
<td>.02&lt;-2</td>
<td></td>
</tr>
<tr>
<td>5. All Ceramic Package</td>
<td></td>
<td>8</td>
<td>4</td>
<td>662</td>
<td>3</td>
<td>.001</td>
<td>.01</td>
<td>.01</td>
</tr>
<tr>
<td>a. Leads</td>
<td>8</td>
<td>4</td>
<td>-</td>
<td>-</td>
<td>662</td>
<td>2</td>
<td>1.0</td>
<td>.5</td>
</tr>
<tr>
<td>b. Leadless</td>
<td>10</td>
<td>3</td>
<td>1</td>
<td>0.5-1000</td>
<td>.001-2.0</td>
<td>.001-1.0</td>
<td>.02&lt;-2.0</td>
<td></td>
</tr>
<tr>
<td>6. Other: Please Describe</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Matrix Question #6

Other 5 responses.

1. Multi-Layer ceramic with metal lids attached with AuSn solder, dips, PGA, lead less (no statistical data provided)

2. All ceramic body with metal lid (solder sealed, 600K per yr, < 1% F.L., Vol 0.3cc)

3. Axial lead solid glass diode (no statistical data provided)

4. DO-7 Glass Pkg. - Axial leads (no statistical data)

5. Ceramic Pkg. with glass window - Init and epoxy sealed (no statistical data)
3. Which hermetic seal test specification do you use?

(a) MIL-STD-202F, Method-112E  (3)
(b) MIL-STD-750C, Method 1071.4  (11)
(c) MIL-STD-883C, Method 1014.8  (22)
(d) ASTM  (0)
(e) Other  0

If ASTM, please specify method designation number. If other specifications are used, please specify:

ASTM Number:

Other Specifications/Methods:

No responses

4. If MIL Standards or ASTM hermeticity specifications/methods are used, which specific tests within Table 1 (next page) do you perform and why? (Please check appropriate boxes on next page)

Please explain:

1) Customer requirements

2) Individual preference
### MIL-STD
202F
METHOD 112E
NOTICE B OCT 11, 1968
GROSS LEAK
- Cond. A. Bubble Test
  - Mineral Oil-Peanut Oil
- Cond. B. Bubble Test
  - Silicone Oil at Vacuum
- Cond. D. Bubble Test
  - Hot Fluorocarbon
- Cond. E. Bubble Test
  - Two (2) Fluorocarbon Liquids
- Cond. F. Fluorocarbon Vapor Detection Test

### MIL-STD
750C
METHOD 1071.4
NOTICE 2 SEPT 17, 1987
GROSS LEAK
- Cond. A. Radioisotope Wet Gross Leak Test
  - (Mineral Oil - Krypton 85)
- Cond. B. Radioisotope Dry Gross Leak Test
- Cond. C. Bubble Test
  - Fluorocarbon (Bomb and Bubble)
- Cond. E. Dye Penetrant Test
- Cond. J. Weight Gain Test
- Cond. K. Fluorocarbon Vapor Detection Test

### MIL-STD
882C
METHOD 1014
NOTICE 6 MAY 29, 1987
GROSS LEAK
- Cond. C. Fluorocarbon Gross Leak Bubble Test
  - C1 = Fixed Method
  - C2 = Flexible Method
  - C3 = Fluorocarbon Vapor Detection Test
- Cond. D. Dye Penetrant Test
- Cond. E. Weight Gain Test

### ASTM
VOLUME 10.04
GROSS LEAK
- E38-72
  - Method A - Hot Bubble (Immersion)
  - Immersion Fluids Are:
    - Fluorocarbons, Denatured Alcohol and Non-Corrosive Ethylene Glycol
- Method B - Backfill and Immersion Gas Backfills Are:
  - Helium, Air, Nitrogen, Argon
  - Helium Mixture
  - Liquid Backfills Are:
    - Low B.P. Fluorocarbons
- Method C - Vacuum Bubble Immersion Fluids Are:
  - Fluorocarbons, Denatured Alcohol and Non-Corrosive Ethylene Glycol
- Method D - Tubulation and Pressure in a Bath. Test Gases Are:
  - Helium, Air, Nitrogen, Argon

### MIL-STD
202F
METHOD 112E
NOTICE B OCT 11, 1968
FINE LEAK
- Tracer Gas Tests
- Procedure I and II
  - For Parts with Evacuation Tubes
  - Procedure III
    - For Sealed Parts: Back Pressurization 12a and 15c-Helium 12-15c-Krypton 85

### MIL-STD
750C
METHOD 1071.4
NOTICE 2 SEPT 17, 1987
FINE LEAK
- Cond. G. - Tracer Gas Radioisotope Krypton 85
- Cond. H. - Helium Tracer Gas
  - H1 = Fixed Method
  - H2 = Flexible Method
- Cond. B. - Radioisotope Krypton 85

### MIL-STD
882C
METHOD 1014
NOTICE 6 MAY 29, 1987
FINE LEAK
- Cond. A. Helium Tracer Gas
  - A1 = Fixed Method
  - A2 = Flexible Method
  - A4 = External Helium Source

### ASTM
VOLUME 10.04
FINE LEAK
- E34-85
  - Helium Tracer Gas
    - Method A - Internal (Flexible Method)
    - Pressurization and Detection
    - Method B - External Helium Probing

### MIL-STD
202F
METHOD 112E
NOTICE B OCT 11, 1968
COMBINED FINE AND GROSS
- F316-83
  - Vent Hole Evacuation - Mass Spec and External Helium Tracer Gas Internal Package Size of 0.6cc or Greater

### MIL-STD
202F
METHOD 112E
NOTICE B OCT 11, 1968
F134-85E1
- Method B. Package Attachment to Mass Spec and External Helium Tracer Gas

---

**MIL-STDs and ASTM Tests**

**Table 1**
5. What percentage failures do you experience with the following seal type:

<table>
<thead>
<tr>
<th>Percentage Failures (%)</th>
<th>0</th>
<th>.01</th>
<th>.1</th>
<th>.3</th>
<th>.4</th>
<th>.5</th>
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<th>3.0</th>
<th>8.0</th>
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<td>5</td>
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<td>Other</td>
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</tbody>
</table>

Other = None 2, No Response 4, Proprietary 1, Blow Hole 1

6. How many parts are you capable of leak testing per day?

<table>
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<th>Devices Per Day</th>
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<th>100</th>
<th>300</th>
<th>400</th>
<th>500</th>
<th>600</th>
<th>1K</th>
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<th>3K</th>
<th>4K</th>
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<th>70K</th>
<th>100K</th>
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<tbody>
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<td>1</td>
<td>4</td>
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<td>1</td>
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<td>1</td>
</tr>
<tr>
<td>Gross</td>
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<td>1</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>6</td>
<td>1</td>
<td>2</td>
<td>2</td>
<td>1</td>
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<td>2</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

7. What is your opinion of the tests you perform? Do you see any weaknesses or inconsistencies in the tests you perform? Include any problems you may be experiencing. Please answer in detail and attach reports, relevant data, etc. if available.

<table>
<thead>
<tr>
<th>Reliable</th>
<th>Unreliable</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>Retesting/Repeatability 3</td>
</tr>
<tr>
<td></td>
<td>Subjectivity (Bubble Test) 1</td>
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<tr>
<td></td>
<td>Procedure Adherence 1</td>
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<tr>
<td></td>
<td>False Leakers (Bake-out) 3</td>
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<tr>
<td></td>
<td>Test Inadequate 1</td>
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<tr>
<td>No</td>
<td>Large Volumes (Collapsing) 1</td>
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<tr>
<td>Response</td>
<td>Correlation (Leak Vs RGA) 1</td>
</tr>
<tr>
<td></td>
<td>Detailed (Respondee #100) 1</td>
</tr>
<tr>
<td>10</td>
<td>Accuracy (Respondee #22) 13</td>
</tr>
</tbody>
</table>
8. Do you see inconsistencies or inaccuracies in the present MIL-STD-883C, 1014 Test Method, if so, where? What method do you consider best for large volume packages, i.e., hybrids, VISI, etc?

<table>
<thead>
<tr>
<th>Yes</th>
<th>No</th>
<th>No Response</th>
<th>Other*</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>15</td>
<td>14</td>
<td>3</td>
</tr>
</tbody>
</table>

*Add Bake (fine leak) (1)

Correlation of He to Kr85 (fixed method) (1)

Contaminated Test Parts and False Leakers (1)

9. Are there any other tests that you feel should be considered for inclusion in Test Method 1014 of MIL-STD-883C?

Yes ___ No __

If yes, describe in detail.

Yes* (4)

No (18)

No Response (10)

*Alcohol Bomb
Faster Gross Leak
Dry Gross Leak
Helium Bubble Test
SECTION 2
One-Way Leaker Phenomena

The one-way leaker phenomena is somewhat of an enigma in the leak testing community. It is not known how many people are aware of the problem or whether they have addressed the problem in their leak testing procedures. Basically, the one-way leaker phenomena functions similar to a check valve, allowing a leak to pass only in one direction. It is usually very sensitive to pressure and/or temperature. It can allow gas or a liquid to become entrapped in a device. These leakers have in the past been identified mainly by destructive residual gas analysis tests. Another aim of this study is to focus on this phenomenon.

1. Are you familiar with the one-way leaker phenomenon?
   Yes 15   No 31   No Response 0
   If no, continue on to Section 3.
   If yes, do you use or know of a test or tests that can identify these leakers? Please explain:
   RGA (3)
   DYE (1)
   KR85 and Weight Gain (5)
   Total 9 Responses

2. Do you have any sample parts which were determined to be one-way leakers?
   Yes 1   No 31
   Do you have any samples you wish to contribute to this study?
   Yes 0   No 30   No Response 1   Maybe 1

3. Do you have any detailed reports describing test results and analyses on one-way leakers that you would be willing to contribute to this study.
   Yes 0   No 32
SECTION 3

Equipment Manufacturers

1. Do you perform leak testing?
   - No
   - Yes - Please detail the types of test used.

2. Do you provide leak test training to your customers?
   - No
   - Yes - What type of tests - please specify.

3. Can you identify changes in hermeticity test specifications which would enable you to produce better test equipment?
   - No
   - Yes - Explain
     1) Increase NID drying time from 5 min (current) to 7 min.
     2) Combined fine and gross leak test using mass spectrometer.

4. Do you know of other methods of leak testing which you would recommend for incorporation into Test Method 1014 of MIL-STD-883?
   - No
   - Yes - Explain

5. Do you have technical reports to support your recommendation?
   - No
   - Yes - Please enclose a copy.
SECTION 4

Failure Analysis

1. Do you perform failure analysis on parts which fail leak tests?

   [ ] 22 No
   [ ] 8 Yes

   2 No Response

2. If your answer to (1) was Yes, please describe the locations of leak sites and percent of each type e.g.: Tö-Series, 85% glass to metal seal, 10% weld area and 5% cracked metal.

   Glass to metal (soldered and welded) Poor wetting
   Metal lid to ceramic Cracked ceramic
   Glass Feed thru
   Seal voids
   Ceramic to glass
   Horizontal/vertical crack

3. Describe the techniques used to determine the locations of the leak sites.

   [ ] 3 Visual examination
   [ ] 9 Bubble test
   [ ] 17 Dye penetrant
   [ ] 8 SEM
   [ ] 1 Tubulation
   [ ] 2 Cross section
   [ ] 1 Fine leak
   [ ] 1 Gross leak

   8 No answer
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