Moisture Analysis:

History, Sampling and Case Studies

By

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Note: This material was presented as an invited guest at the JEDEC meeting on January 13, 2004 in Phoenix Arizona.

This presentation was intended to provide an overview of the technology and history of Internal Water Vapor Analysis of hermetic electronic devices by mass spectrometry per MIL-STD-883, TM1018, Procedure 1 and MIL-STD-750, TM1018, Procedure 1.

A few case studies of successes and failures in using these MIL-STD mass spectrometry methods are presented.

Introduction:

Mass spectrometry analysis of electronic components is more than an Internal Water Vapor test as the titles of the Military Standards 883 and 750 might imply. The test provides a quantitative measurement of all gases inside a device and should be used as such. To ignore the other gases is like throwing away a winning ticket to a lottery. The other gases matter. The other gases are relevant indicators of sealing quality and potential product reliability issues. Because the test measures all of the gases, the test method provides the only way to evaluate the quality of sealing processes. It provides the only way to conclusively identify out-of-control sealing batches. Because it measures all of the gases, the method is best used as a tool to *develop* sealing processes and then *monitor* sealing processes. Only then can it be effective as a qualification tool.

The current procedure to "qualify" high reliability product for the military once every 3 to 6 months using the criteria of 3+2, accept with 1 reject, is statistically ineffective and inconsistent with high reliability concepts.

Moisture problems can be extreme and devastating to military and commercial systems. Out of control sealing processes or sub-processes lead to a built in wear-out failure mechanism where every device has the potential to fail prematurely causing severe system consequences. Test data has also shown that leak-test-escapes are real and prevalent and lead to failures, yet the Military Standard is silent on the subject of "other" gases. Only moisture limits are specified.

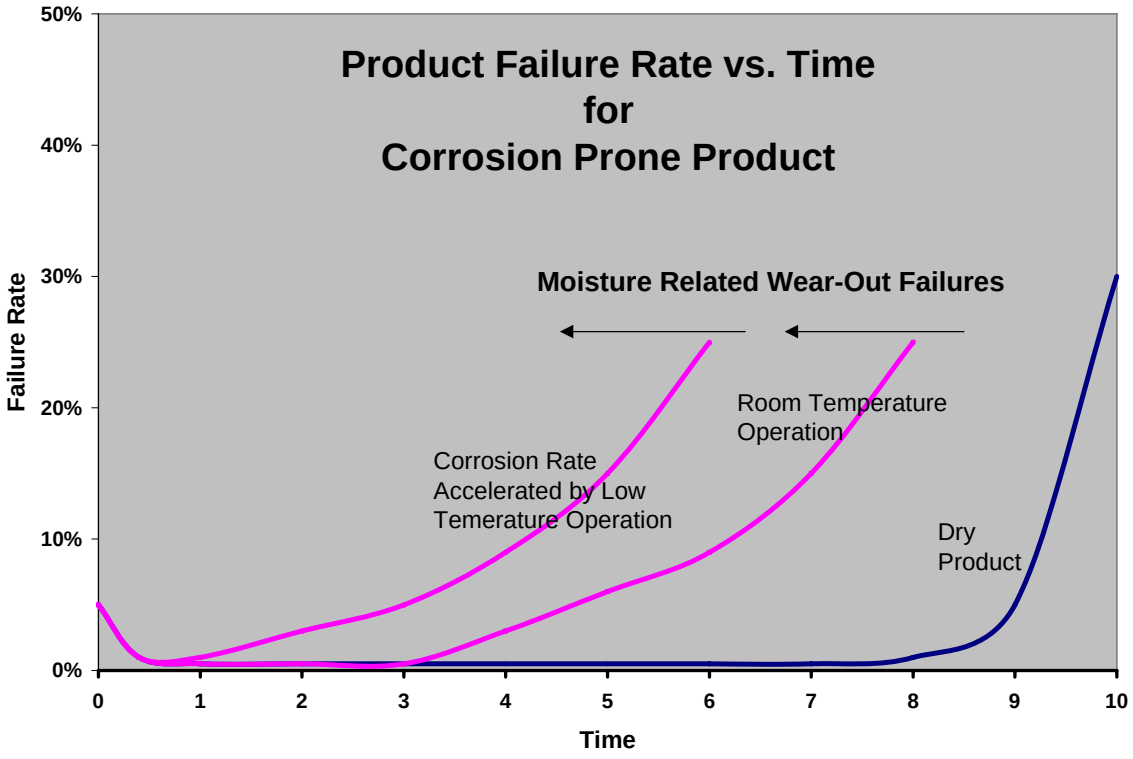
Users need to inspect the laboratories and have the laboratory demonstrate the validity of their protocols. There is far more technical detail in achieving accuracy and sensitivity then required by the Military Standard test method.

The Military Standards are intended to qualify product for the government. The MIL-STD test parameters are limiting and may not be modified. Process development, failure analysis and commercial products often need other parameters such as different mass ranges, sample temperature or pre-bake conditions to provide pertinent engineering data.

History:

Many have never experienced a serious moisture problem with their products. Some question why internal water vapor analysis is required by the military to qualify hermetic components. However, those who have experienced a serious moisture problem will never forget it. The reason that they will never forget it is that moisture related failures could be extreme. So extreme in fact, that the failure rate may take on the properties of one of the few true wear-out failure mechanisms where virtually every unit of the product might fail in the field. The consequences can be severe.

The failure mechanism, as it relates to moisture, is usually corrosion of the metallurgy. Corrosion is induced by the condensation of moisture and accelerated by many factors, including, low temperature operation, voltage, usage cycle, ionic contamination, and the design of the device. Moisture induced failures are one of the few failure mechanisms that are accelerated by operation at low temperature. It can corrode the metallurgy or cause a soft error such as excessive leakage current via the condensation of moisture accelerated by ionic contamination.



Prior to the implementation of 1018 test methods in the late 1970's, there were numerous commercial and military system problems due to microcircuit internal corrosion. Measuring moisture in microcircuits proved difficult at first because of the physical property of water that makes it tend to stick to surfaces. Special sample mounting techniques and transfer passages had to be designed to efficiently transfer the moisture from the sample cavity to the detector of the mass spectrometer. At the heart of the technology are designs and methods to achieve efficient moisture

transfer efficiency along with good calibration techniques and good mass spectrometry techniques to collect and quantitate the data.

MIL-STD-883, TM 1018 was implemented in 1979 with substantial controversy. Since then, the mass spec technology (via much R&D and investment by the laboratories) continued to evolve with new concepts, better transfer passage design to minimize moisture signal loss, better calibration methods, many new generations of computers and software and new generations of computer controlled mass spectrometers.

By contrast, the MIL-STD's, Test Methods 1018 saw no substantive change over that time. Worse, the acceptance criteria remained unchanged. It was never the intent that this would happen. The initial "loose" criteria were intended to get the industry started and then, over time, tighten up the criteria.

Ineffective Qualification Sampling Plan:

The current procedure to qualify high reliability product for the military once every 3 to 6 months using the 3+2 sampling allowing 1 reject is statistically ineffective.

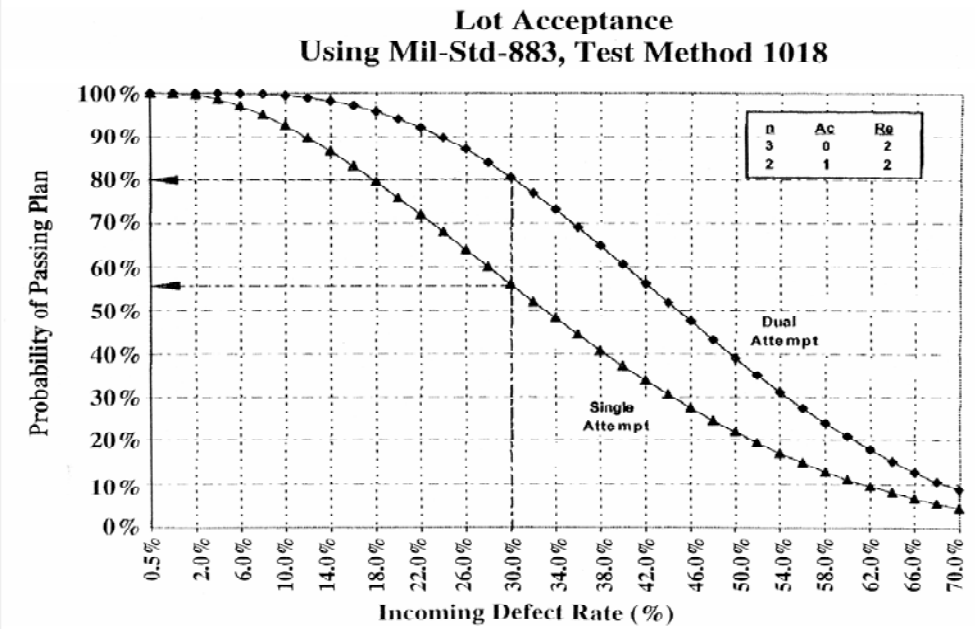


Figure 2

Figure 2 shows the statistical probability of passing large lots of product versus the sample reject rate of the lot. Lots having 30% rejects (e.g. if 30% of the product had water vapor content > 5,000 ppm) have a 55% chance of accepting the entire lot on the 1st attempt and an 80% chance of accepting of the entire lot if a dual attempt is applied.

Defining the Objective:

On face value, this sampling plan and acceptance criteria is not compatible with high reliability concepts. It should cause one to pause and question what the objective is for performing water vapor testing.

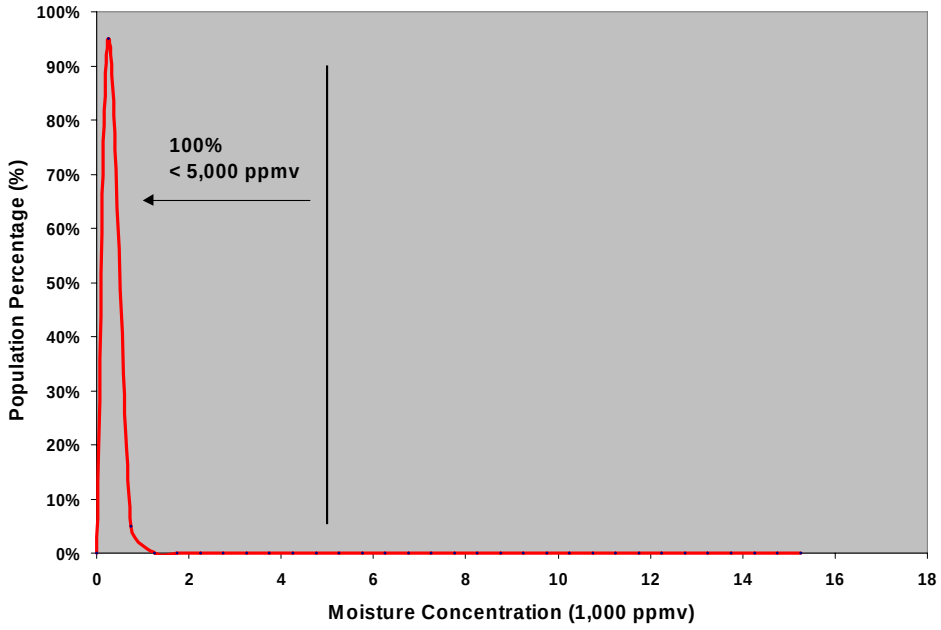
I would like to propose the following statement that might serve as an objective. It was extracted from a DESC letter to the industry (Ref DSN 296-6048 dated February 7, 1996).

> *"... to establish that the device manufacturer has a technical basis from which he can ascertain that the product delivered has sufficient design and process margin at the time of delivery to maintain a package ambient gas atmosphere compatible to high reliability performance in reasonable Military or Space application environments as defined in ....."*

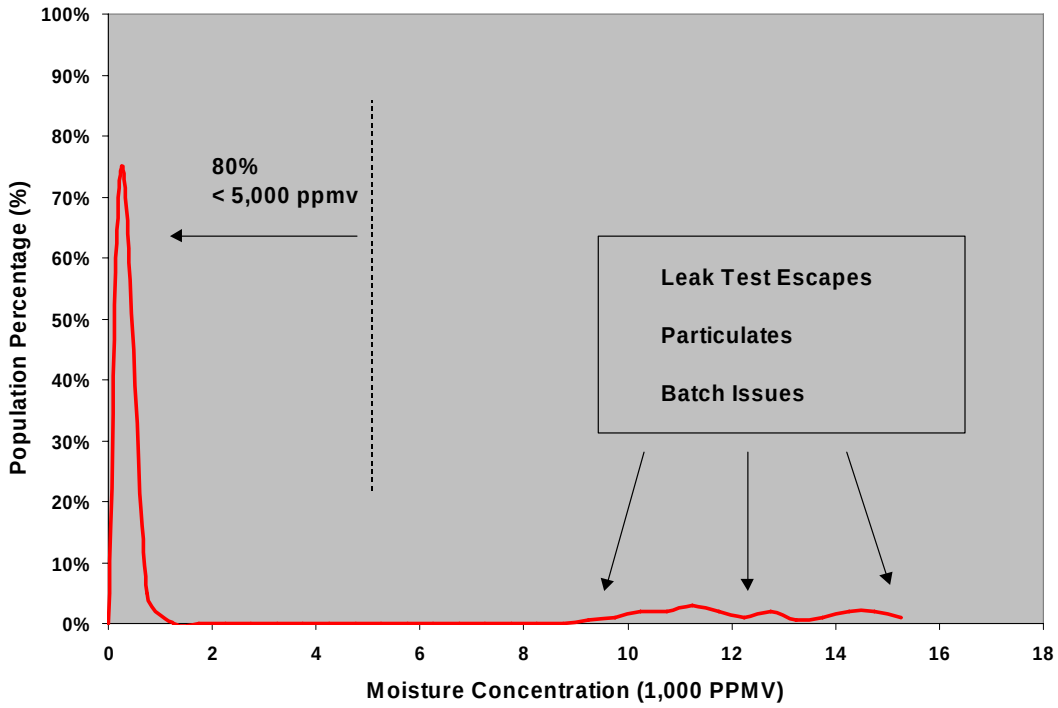
This objective can not be achieved by testing 5 samples every 3-6 months. Aside from the obvious statistical shortcomings, there are other reasons why it can not be achieved.

Statistical Data Patterns for Moisture:

A well controlled TO type device with no organic materials might have a distribution pattern for moisture concentration as shown below. The entire population might have moisture content below 1,000 ppm, with nitrogen as the sealing gas.



All processes are not that ideal. A distribution shown below is often seen. The Figure below indicates that a manufacturer has the basis technology to hermetically seal dry parts but there are other factors that impact the quality of a sub population of the product. There may be a variety of causes. Some may be random rejects and some may be an out-of-control sub batches.



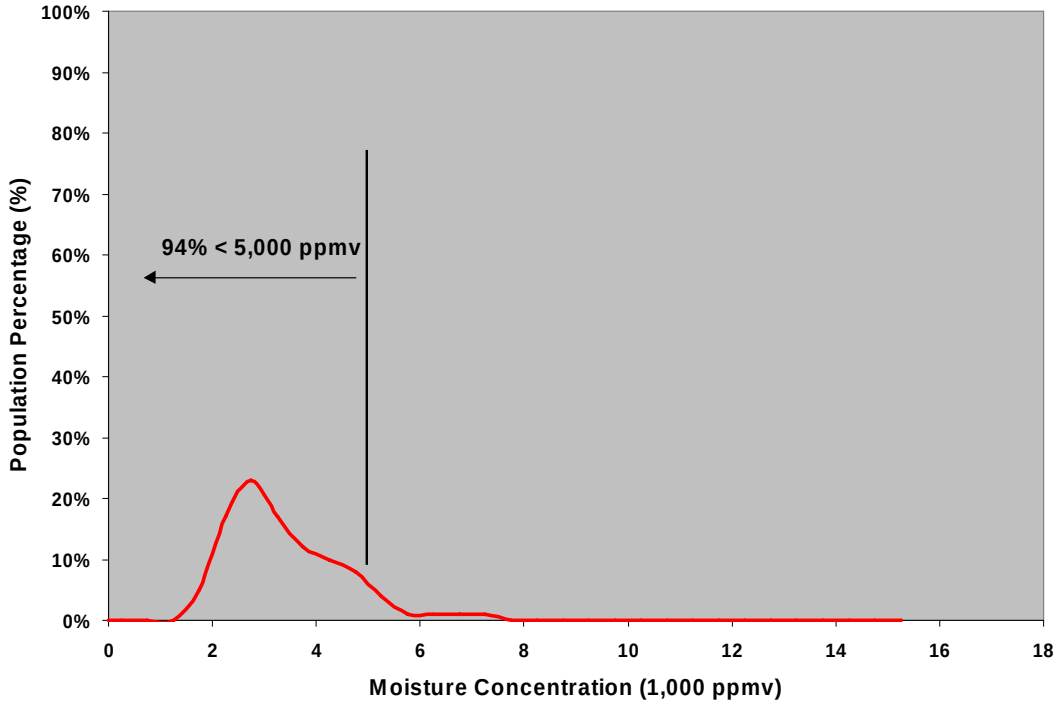
There are two serious types of problem that can create this type of distribution.

One cause could be a single out of control sealing machine (possible for only a day). The seal chamber may not have been purged sufficiently or the chamber was not sealed properly for that day. There is the potential that every device in that batch has been sealed with high moisture content. What appears to be a random event might actually be and entire sub batch that is out of control. Sampling a few devices from many lots may make the problem appear random when in fact, it may be a very lot dependent problem.

The other serious problem that contributes to this type of distribution is Leak-Test-Escapes. The mass spectrometry data often shows the presence of helium and/or fluorocarbons. Sometimes there is the presence of air in devices that are intended to have pure nitrogen. This problem has existed since MIL-STD 883, TM 1018 was implimented in the late 1970's. Through out the 1980's the problem was very significant. It has improved during the 1990's but it is still prevalent in the industry. Some of the Leak-Test-Escape product even passes the moisture level of 5,000 ppm. The problem has also been labled "One-Way-Leakers". Some examples will be shown later.

Other causes could be purely random defect issues such as particulate matter inside the device, defective feed throughs, human spittle, etc.

Product lines that use organic materials inside the devices face another problem. It usually takes an R&D effort on the part of the manufacturer to develop a process that achieves less than 5,000 ppm. Manufacturers usually have to work hard to develop a process and then closely monitor the materials and process to keep it under control. The figure below shows a typical distribution of moisture for a mature organic materials process. It is very difficult to achieve results below 1,000 ppm and the process often has a larger distribution spread. It is not uncommon to have samples that "tail" above 5,000.



During the presentation I was asked what I thought about the 5,000 ppm requirement. Was 5,000 ppm a good choice? One reason that 5,000 ppm makes sense is because the dew point temperature is less than 0 °C. In practice, the actual electrical device failures due to corrosion that I can recall were above 10,000 ppm (> 1%) except for one special case. That is not to say that failures can not occur in the 5,000 to 10,000 ppm range. I think that a good way to look at it is that 5,000 ppm is a good strategic choice as a pass/fail limit and that the range between 5,000 and 10,000 ppm provides a practical safety margin. As for the case shown above where there is a portion of the distribution that tails above 5,000 ppm, you might make the case that the distribution above would be acceptable.

Case Study 1:

A military system was in the pre-production, system test, and qualification stage. The system developer began to observe failures in a specific device type after a relatively short operating time. The mass spectrometry analysis of the failed samples showed high moisture. Failure analysis showed obvious corrosion.

The developer then performed Internal Vapor Analysis on two samples from every lot that was procured for the system build. Hundreds of lots were tested. There was tight lot identification control.

The data fell into two groups depicted by the chart below. They were either, very clean, very dry, hermetic and obviously sealed under tight control (similar to Lot 1), or, they were clean, very wet, but still hermetic (similar to Lot 2).

The samples in each lot were either all good or all bad. There were multiple sealing facilities involved. The problem was traced to a specific sub process within 1 of the

	Lot 1	Lot 1	Lot 2	Lot 2
N_2 (%)	99.9	99.9	98.6	98.6
O_2 (ppm)	ND	ND	ND	ND
Ar (ppm)	ND	ND	ND	ND
CO_2 (ppm)	<100	<100	<100	<100
H_2O (ppm)	157	210	13,500	18,450

facilities. Once the Lot 2 types were pulled from the system and the stockpile, the system had a reliable life.

This is a classic case that describes moisture induced wear-out prone product. This is what the test method was originally intended for. Had this product entered into the operational system, the consequences would have been devastating. Had the device manufacturer not had good lot identification control, the source of the problem might have be treated as a random failure mode and production might have proceeded only to result in a field recall later on.

Given the percentage of defective product, it is highly likely that the product would have passed the qualification requirements if the lots were mixed and/or if only 5 random samples were selected to reflect 6 months of production. The client was fortunate that early failures highlighted the problem.

Case Study 2:

The figure below shows test results from a recent military component. It demonstrates some significant issues. Sample 2 demonstrates that the supplier has the basic technology to seal high quality, dry product. However, samples 1 and 3 show a disturbing trend. They show the presence of helium and air. Sample 2 does not show helium so it can be concluded that the supplier does not intentionally seal in helium as a leak test agent. Samples 1 and 3 show that helium and air (O_2 and Ar) have entered the devices as Leak-Test-Escapes. Note that all three samples passed the < 5,000 ppm moisture requirement.

	1	2	3
N_2 (%)	98.1	99.9	96.5
O_2 (%)	1.09	ND	2.05
Ar (ppm)	583	ND	1,099
CO_2 (ppm)	801	230	1,033
H_2O (ppm)	1,833	178	3,600
He (ppm)	5,262	ND	8,560
Fluoro (ppm)	ND	ND	ND

The point is that the other gases (other than moisture) matter. The data indicates that the product has hermeticity issues. The cause of this should be investigated before the product gets into systems, even though it "passed" the MIL-STD test. Samples 1 and 2 are probably "leakers" and will probably continue to leak and continue to increase the moisture content and most likely be a reliability risk.

Case Study 3:

The following data was obtained from a military system. The first sample was a device that failed in system use and failed due to corrosion inside the device. Samples 2 thru 6 were samples of the same manufacturing vintage found in the replacement stores that would be used to repair the system.

The interesting thing is that all of the samples shown below were over ten years old at the time of the test. Even more interesting is that archive data showed the same general patterns when originally tested 10 years earlier.

This data shows several interesting facts.

	System Failure	2	3	4	5	6
N_2 (%)	98.1	99.9	99.9	88.0	99.9	96.5
O_2 (%)	5.39	ND	ND	9.92	ND	2.05
Ar (ppm)	3,165	ND	ND	6,102	ND	1,099
CO_2 (ppm)	801	380	422	306	651	1,033
H_2O (ppm)	16,811	103	547	14,870	216	4,600
He (ppm)	5,262	ND	ND	ND	ND	8,560
Fluoro (ppm)	8,305	ND	ND	ND	ND	182

First, samples 2, 3 and 5 demonstrate that the manufacturer had the ability to seal high quality product.

Second, the "System Failure" sample (1) and samlpes 4 and 6 demonstrate hermeticity problems as described in the prior case study. These problems are not lot dependent as described in case 1. They are not even product dependent. They reflect a sealing technology of the period. Many manufacturers had this problem to varying degrees during that time period. This was one of the worst examples.

Third, after more than 10 years, the product vapor content did not change, suggesting that the devices are indeed hermetic "now" but at one point in time, probably during the leak test process, the devices leaked temporailly, but then resealed and remained hermetic for many years.

Fourth, samples 2, 3 and 5 baseline the sealing capabilities and the process (Dry Nitrogen). No air or leak test gases appear. The other samples show the presence of air (O_2 and Ar) which is not present in the baseline. The helium and fluorocarbons indicate hermeticity problems and shows that, in this case, the air is most likely getting into the devices at the same time as the leak test gases. (The air could have come from a sealer that was not purged well but this is unlikely because of the frequency of finding helium and fluorocarbons.)

Fifth, only 2 of the 6 samples are rejects per the MIL-STD. The probability of accepting lots having 33% rejects is 50% on the first attempt and 80% if a dual attempt was used (which was allowed at that time).

Sixth, there was substantial testing of this product when the product was originally built. The original data replicates the above data very closely. It is purplexing why this product was used in systems. It is likely that the tests probably passed the "3+2 with 1 failure" requirement. The other gases should have raised a red flag that the product had hermeticity problems.

When TM 1018 was first proposed to the industry, most proponents were trying to prevent the senerio presented in Case 1, what I call the wear-out potential, lots where the "process" was out of control. The concept of qualifying 3+2 samples every 3 to 6 months was a gross over simplification of how to use a valuable analytical test method. Many manufacturers who have had moisture problems have learned the hard way why the method is more than just a "moisture" test. Many manufacturers, military and commercial, use it to develop and control their process on a daily or weekly basis in order to achieve high quality sealing atmospheres. Others use it just for the moisture values and disregard everything else as seems to have happened in case study #3.

What seems to be overlooked is that internal vapor analysis can validate the hermeticity test procedures but only if the test is performed "after" hermeticity testing.

Conclusions and Recommendations:

Internal vapor analysis is the only way to evaluate the quality of the sealing processes. It is best used as a tool to *develop* sealing processes and then *monitor* sealing and hermeticity testing processes.

The methods are more than an "Internal Water Vapor" test. They measure <u>all</u> gases inside a device and should be used as such. The other gases matter and are often indicators of the overall hermetic sealing process. The data is a valuable engineering tool to validate the integrity of the package, the stability of the materials inside, the controls on the sealing chambers, and, the effectiveness (and potential destructiveness) of the fine and gross leak test procedures. All this information is available from the test.

Moisture problems can be extreme and devastating to military and commercial systems. Out of control processes can (and have) lead to a built in wear-out failure mechanisms where every device may fail prematurely causing severe system consequences.

The current procedure to qualify high reliability product for the military once every 3-6 months using the 3+2 accept with 1 reject criteria is statistically ineffective. Such a

small sampling plan may only be effective if it is supported by a process monitor database.

Many moisture problems are lot dependent. Small quantity sampling from multiple lots allows sub-process quality issues to go undetected with this sampling plan.

The 5,000 ppm industry standard for acceptance is probably a good strategic choice as a pass/fail limit.

For more than 25 years, test data has shown that leak-test-escapes are real and prevalent.

The Military Standards are intended to qualify product for the government. The MIL-STD test parameters are limiting but may not be modified. Process development, failure analysis and commercial products often need other parameters such as different mass ranges or sample temperature or pre-bake conditions.

Users need to inspect the laboratories and have the laboratory demonstrate the validity of their protocols. Find out how the test is done, exactly. There is far more technical detail in achieving accuracy and sensitivity than is specified by the Military Standards. Quality inspections of a laboratory that focus only on compliance to TM 1018 are only a small factor in the ability of a laboratory to produce precision and accuracy in this technology. TM 1018 does not describe any way near enough critical properties of the laboratories method. I am not proposing that the TM include such information because there are many valid ways to do it and it may be an impossible task. What is needed is that each laboratory should be able to "Validate" their method to users and demonstrate how robust the methods are. The following are some common questions:

> How do they calibrate for moisture? How often, what moisture levels, what volumes? What type of calibrators are used? What is the moisture reference standard? How precise is it? How is it calibrated? Can the lab validate moisture accuracy?
>
> How do they measure the primary peaks? (Note: the method calls for measuring moisture in Parts-Per-Million. You therefore have to accurately measure the "million", the primary peaks, all of them, accurately!) Can the lab validate primary gases accuracy and identification?
>
> How do they tune the spectrometer? What parameters are adjusted, how are they monitored, and how are they set?
>
> What is the moisture transfer efficiency? How much moisture is lost in the transfer passage?
>
> How is the data acquired? How fast, how many data points per mass peak? How is the data stored?

How is the data quantitated? Are there any special correction factors?

Does the lab require any pre-test warnings (Vacuum sealed or high pressure sealed samples, primary gases other than nitrogen)?

How specific can the lab identify the type of fluorocarbons or other solvents?

Over all, the mass spectrometry technique is the only test method that can provide a complete process control picture of hermetic sealing technology.

It is a powerfull engineering tool.

About the Author

Mr. Rossiter received a B.S. in Physics and a M.S. in Solid State Science and Technology from Syracuse University. He worked at the Rome Air Development Center in Rome NY for 9 years in the field of Physics of Failure of Microelectronic Devices. He was the manager of the R&D Analytical Lab at Harris Semiconductor for 4 years and also held the position of QC Manager at Harris Semiconductor for 2 years.

He founded Oneida Research Services, Inc. in 1977 and currently is President.