# Measuring mass flows in hermetically sealed MEMs & MOEMs to ensure device reliability

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## Abstract

Many MEMs and MOEMs devices require controlled ambient environments for successful operation. Controlled ambients are usually obtained via hermetic packaging. These controlled environments must first be obtained and then maintained to prevent their degradation over the device lifetime. Controlled ambients decay in quality over time due to various mechanisms including leaks, permeation, poor processing and outgassing of species like hydrogen, water and organics external and internal to the package. The key to controlling the process of degradation is to understand in a quantitative manner which species are present and their mass flow rates into the controlled ambient.

The current work describes a new technique for determining these species and mass flow rates. This new technology provides tremendous sensitivity to package volumes < 0.01cc compared to standard quadrupole techniques, which are applicable to samples larger than 0.01 cc. The technology is based on a high speed, high mass resolution, and highly sensitive Time-Of-Flight (TOF) spectrometer to test the tiniest of devices with significant advancement in signal-to-noise ratios. Key operational parameters demonstrated include:

- Spectra Acquisition speed: 1 full spectra every 20 µs.
- Mass Range: mass 2 to 150 standard (2-500 capable)
- Mass resolution: 0.1 AMU
- Calibration Fixtures: 0.0001, 0.0005, 0.001, 0.005 and 0.01 cc
- Sample temperature: 100°C standard (room temperature to 150°C capable)

**Keywords:** MEMs, MOEMs, Reliability, RGA, Outgassing, Leak, Analytical, Time of Flight, TOF

## 1. INTRODUCTION/BACKGROUND

Quantitative analysis of the internal gas content inside hermetic devices was originally developed in the 1970's. It was implemented in MIL-STD-883, TM-1018 and MIL-STD-750, TM-1018 in the late 1970's in response to significant failures from internal corrosion and soft electrical upsets due to moisture. The military focused almost exclusively on measuring and qualifying the moisture content in military devices by setting a pass-fail criterion of less than 5,000 ppmv moisture in devices. The original rational behind the 5,000 ppmv limit is arguable, but the dominant argument is derived from the fact that a dew point temperature of 5,000 ppmv is -2.3°C, below the freezing point. Mass spectrometry is the method of choice for performing such analysis, because it has the ability to directly measure all of the gases in a hermetic device. With proper calibration techniques, mass spectrometry can accurately determine moisture as well as all of the other gases in units of parts-per-million by volume (ppmv).

It is important to note that Military Standards are for the specific purpose of qualifying devices for the military. The test conditions (pre-bake temperatures, test temperatures, limits of sample volume for which labs are Suitable, etc.) may not be changed and may not be appropriate for things like medical devices, process development studies and materials research applications. Testing labs are prohibited from altering the test parameters and still labeling the report as a Military Standard test.

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The Military Standards focused on moisture content. The original title was "Internal Water Vapor Content," but for almost three decades, the test data has shown numerous systemic problems related to leak test quality and outgassing of materials inside devices as evidenced by other gases found in devices. These undesirable and uncontrolled gases have been all but ignored in the Military qualification criteria. This is changing. The title of the Military Standard has been changed to "Internal Gas Analysis". The purpose has also changed from measuring moisture to measuring all gases with an emphasis on moisture. (Note: Mil Std 750 has implemented this change and Mil Std 883 will most likely follow in the next revision).

Until recently, the technology for testing the internal gases in hermetic devices was limited to internal cavities of 0.01 cc (at 1 atm.) and larger. Poor signal to noise issues prohibited accurate measurement below 0.01cc. New device technologies such as MEMs are beginning to succeed in the marketplace, while many newer devices are much smaller. In addition, the use of vacuum packaged devices is increasing. Vacuum packaging to include the use of materials like getters that actively pump gas from the cavity result in an effective quantity of measurable gas far less than that of a 0.01cc @ 1 atm device. Some of these technologies use sealing gases other than nitrogen or air, with some having sealing gases that have a mass greater than 100 AMU.

## 2.  MASS FLOWS IN MEMS/MOEMS

In order to understand the nature of the problem to be analyzed in small volume cavity devices it is important to understand what the critical factors are. A simple pictorial model of a cavity shows the following mass flows of gases and other volatile species within a hermetically sealed device:



P = pressure
C = concentration
V = volume
T = temperature
L = length
Ø = diameter
Prm = permeation rate
Ps = physisorption rate
Og = outgassing rate
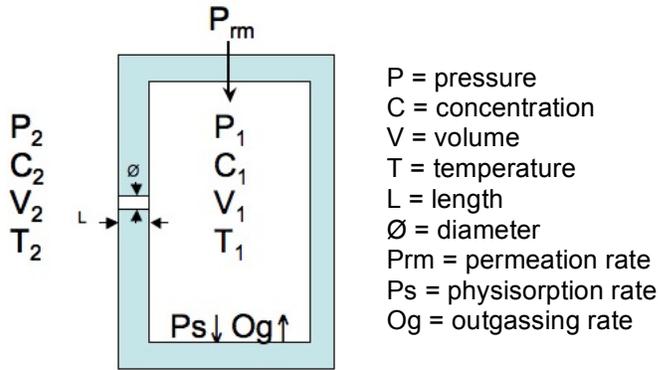
Fig. 1 Pictorial model of a hermetically sealed cavity and the mass flows in the system so defined.

Practically speaking, experience has shown that with good choices of materials and processes, fine leak testing per MIL-STD-883E results in hermetic seals with practical device lifetimes.

"3.1.1.2.1  Failure criteria.  Unless otherwise specified, devices with an internal cavity volume of 0.01 cc or less shall be rejected if the equivalent standard leak rate (L) exceeds 5 x 10-8 atm cc/s air.  Devices with an internal cavity volume greater than 0.01 cc and equal to or less than 0.4 cc shall be rejected if the equivalent standard leak rate (L) exceeds 1 x 10-7 atm cc/s air. Devices with an internal cavity volume greater than 0.4 cc shall be rejected if the equivalent standard leak rate (L) exceeds 1 x 10-6 atm cc/s air."

Yet devices still fail, particularly as the volumes drop below 0.01 cc. Proposals are under discussion in industry and governmental forums to tighten leak testing requirements, but ongoing analysis of MEMS devices shows that leaks are not necessarily the culprit. Qualitative analysis in multiple labs has shown species indicative of other failure mechanisms that should be considered.

Detailed consideration of the pictorial model shown above points to two other mass flows within the system that should be studied in depth for proper understanding of potential failure mechanisms. These mass flows are permeation and outgassing. Both have experimentally derived mass flow rates in the ranges proposed for improved leak testing. However, without knowing the constituent species of these mass flows, it is impossible to determine if they are due to leaks, permeation or outgassing. An ideal analytical technique would not only be able to determine any internal pressure rise within a discrete cavity without inducing potential leak paths by stressing the device containing the cavity, but it would be able to identify the chemical species present and their quantities so that the cause of the pressure rise could be isolated from the choices of leaks, permeation or outgassing.

## 3.   HIGH RESOLUTION INTERNAL VAPOR ANALYSIS

ORS has developed its High Resolution Internal Vapor Analyzer (HR-IVA[TM]) technology to test very small internal volume devices like MEMS and MOEMS. These devices may have internal volumes as small as .25 nanoliters. The technology uses time-of-flight (TOF) mass spectrometry to achieve the necessary spectra acquisition speed and sensitivity to acquire the miniscule amount of gas emitted from such a small hermetic device. ORS has developed all metal (no organics) calibrators having internal cavities of 0.0001cc, 0.0005cc, 0.001cc, 0.005cc, and 0.01cc. The calibration fixtures allow the injection of small calibration gas bursts to identically simulate a sample test.
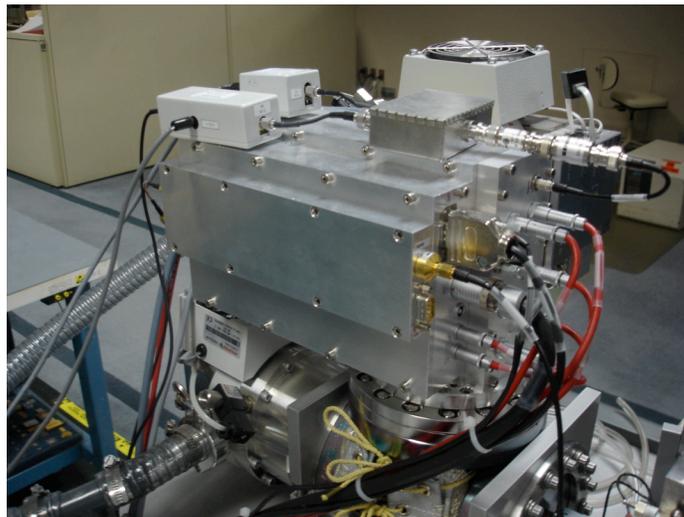


Fig.2. HR-IVA time-of-flight mass spectrometer.

## 4.   BASIC APPROACH TO INTERNAL GAS ANALYSIS USING HR-IVA

ORS has historically used a dynamic approach to measuring moisture and other gases. The dynamic method measures the sample gas in transit, as it is emitted from the sample and passes by the ion source to be pumped away. The process of transferring the sample gas to the mass spectrometer ion source becomes very critical as the device's internal volume gets ever smaller. In order to achieve optimum signal-to-noise in the mass spectrometer ion source, ORS has significantly improved the transfer passage efficiency over the past few years. A comparison of the signal-to-noise values for quadrupole and TOF technology are shown in Figure 3:
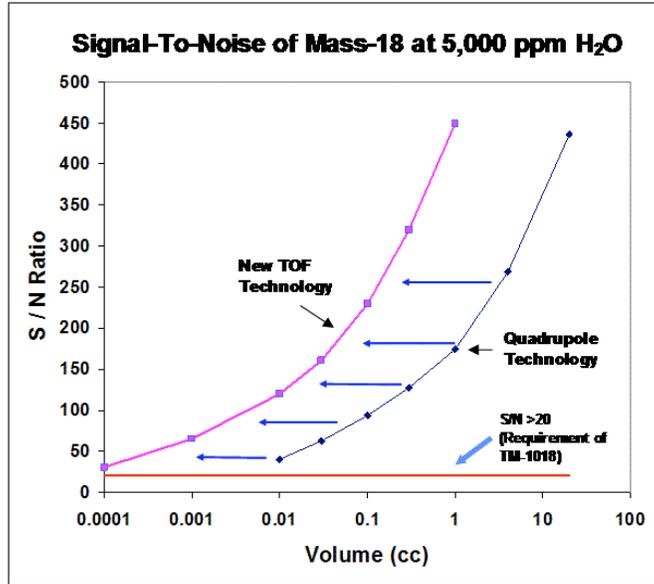
Fig. 3. Signal to noise comparison of quadrupole versus TOF.

The internal volume of the transfer passage of the High Resolution IVA TOF system has been drastically reduced to minimize interaction effects between the sample and the transfer passage. The smaller transfer passage volume also serves to increase the gas pressure in the ion source of the spectrometer, increasing the signal-to-noise ratio in the mass spectra. For small samples in the range of 0.0001cc increasing ion count is critical to achieving adequate signal-to-noise requirements.

Figure 4 shows a block diagram of a traditional dynamic internal vapor analysis system. This figure depicts the concept of both existing IVA equipment, which uses a quadrupole mass spectrometer, and HR-IVA systems that use a TOF mass spectrometer. The key differences between IVA and HR-IVA systems are the type of mass spectrometer and data acquisition systems used. In fact, the majority of the components are identical in both systems.
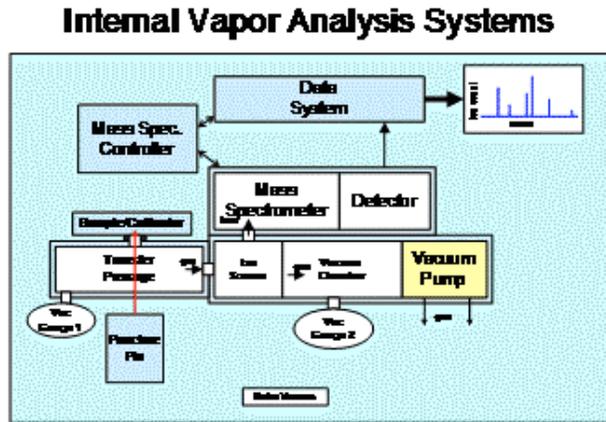


Fig. 4. Block diagram of a typical internal vapor analysis system.

When a measurement is taken with either system type, a dynamic test method is used where the gas from the sample dynamically passes through the ionization source and is pumped away. When the device to be analyzed is punctured, the gas passes into the transfer passage, then through a leak orifice that is adjusted for the estimated amount of gas in the

sample device. The smaller the sample, the larger the leak orifice and the faster the sample gas will pass through the chamber. As the sample exits the leak orifice, it passes through the ion source of the spectrometer where the gasses are ionized and detected by the detector.

Quadrupole technology is unable to achieve the necessary signal-to-noise ratios for very small samples because the gas pulse from the sample is too fast for the limited spectra sweep speeds possible with these instruments. To measure these very small gas samples we developed a custom time-of-flight mass spectrometer to acquire high speed data.

## 5.  TIME-OF-FLIGHT MASS SPECTROMETRY

### 5.1  TOF System
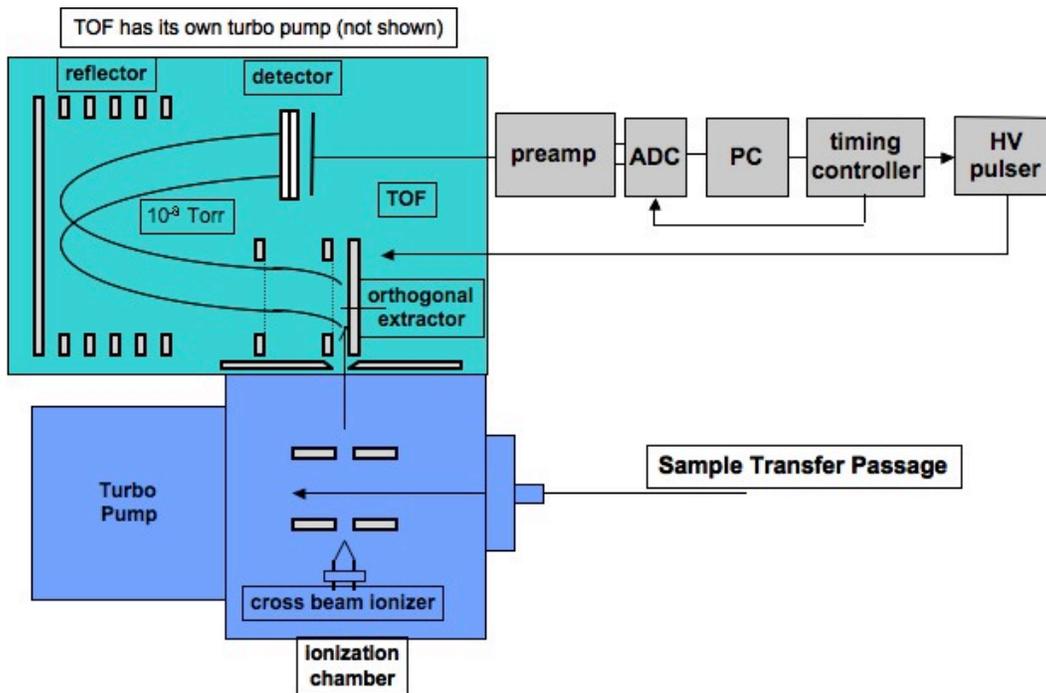


Fig. 5. The basic design of a time-of-flight spectrometer. The gas from the test sample passes through the transfer passage, leak valve and then the ion source and is pumped away.

Ions are continuously created in the ion source as the sample gas passes through. The TOF flight tube is above the ion source. The flight tube chamber is under high vacuum, evacuated with its own turbo pump. There is a small entry point where ions are orthogonally extracted into the flight tube by a very short, high voltage pulse. Each extracted packet of ions contains ions of the entire gas mixture. The packet of extracted ions is then pulled along the flight tube to the ion detector, which is at the end of the flight path. The ion detector simply counts the number of ions that hit the detector over a 1 μs period.

The time that it takes each ion to traverse the flight tube is a function of the mass-to-charge ratio. The detector counts the ions that hit the detector and saves the number of counts in a memory array where each element in the array represents the ion count per nanosecond. Each data array of ion counts verses time represents a mass spectrum where the index of the array represents time in nanoseconds. The time is later converted to atomic mass units (AMU) by a direct calibration function. The flight tube is cleared of ions after 20 μs and another extraction event will be triggered to collect another mass spectra from the newly extracted ions.

A mass spectra array that ranges from 1 AMU to 168 AMU has, for example, 7,680 array elements. Each element is measured 1ns apart. The system is capable of measuring out to 560 AMU but is typically set to measure to 168 AMU as a practical range. The HR-IVA system collects ion counts over the mass range of 1 AMU to 580 AMU on every extraction. However, spectra data from 1 AMU to 168 AMU is saved because saving data beyond 168 AMU consumes a great deal of memory and is unlikely to offer any benefit. The software can be quickly reset to collect data beyond 168 AMU if needed.

## 5.2 Typical Spectra Output

Figure 6 displays a typical spectra output. The scale for the x axis is reported both in units of time (1720ns to 2100ns) and as m/e (14 to 18). The fine resolution of the peaks (the time separation between the peaks) demonstrated enables the HR-IVA to distinguish fractional masses. By contrast, quadrupole systems are not capable of distinguishing fractional masses. Xenon, for example, has several mass peaks that appear at half mass units.
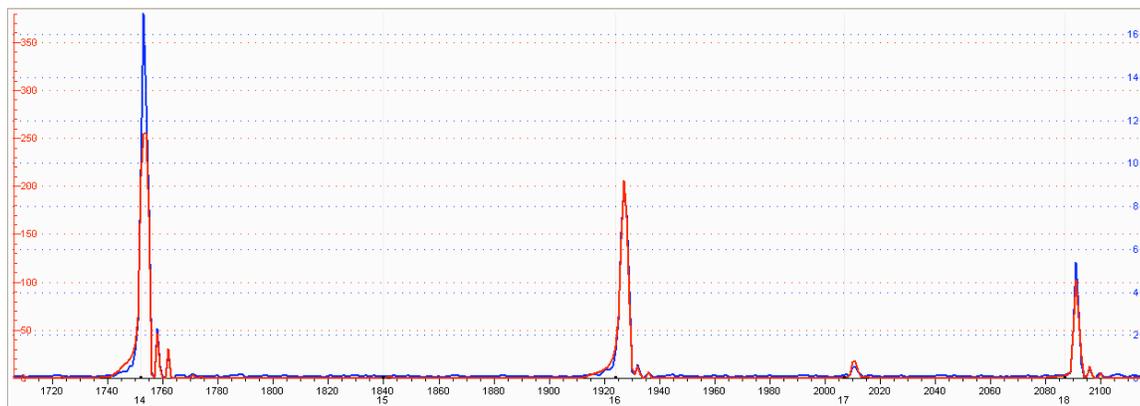


Fig.6. Mass spectra of peaks 14 through 18 showing typical peak resolution.

In figure 6 the red curve is the highly amplified channel output and the blue curve is the lesser amplified output channel. The highly amplified channel is used to quantify trace levels and the lesser amplified channel is used to quantify major levels. Note that for AMU 14, the sensitive channel (red) is saturated. The amplitude of the blue peak would then be used for quantifying AMU 14 while the amplitude of the red peaks would be used to quantify AMUs 16, 17 & 18.

## 5.3 Moisture Calibration Curve

Improved linear response for moisture as measured at 5000 ppmv is a critical factor when quantifying data. The dynamic method reduces surface effects and conductance issues to provide a linear response for moisture. Figure 7 shows a typical moisture calibration curve for a 0.0005cc calibration volume:

**0.0005cc Calibration Curve**
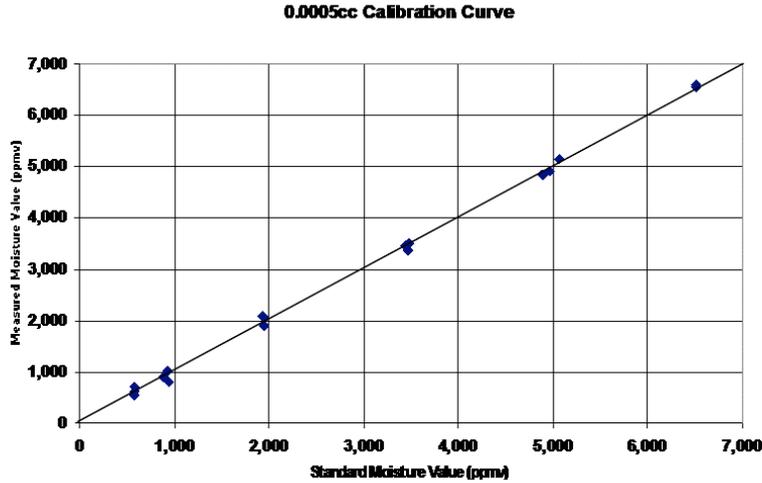
Fig.7. Moisture linearity curve for a 0.0005cc volume.

# 6. MEASURING MASS FLOWS WITH THE HR-IVA SYSTEM

## 6.1 Example 1

A 0.001cc cavity sample was analyzed for internal gas content utilizing the HR-IVA. The purpose of the analysis was to determine the concentration of moisture in the package. The package cavity contained a moisture getter and was prebaked at 100°C for 16-24 hours.

Outgassing of surfaces and materials can affect the total volume of gases measured during the time of analysis. Measurements taken where the sample is punctured and allowed to "sit" in the vacuum chamber while sampled over long periods (10 seconds to several minutes) are not capable of measuring these affects. In fact, the interactions of small sample volumes within a large test chamber allow for unwanted surface reductions, oxide formations, absorption of moisture to "dry" surfaces, and dilution of the gas mixture. All of these effects can change the capabilities and results of the test, as well as reducing test sensitivity. In comparison, the instantaneous HR-IVA method, where the gases are measured at the exact time of puncture, limits or eliminates these issues.

Figures 8 and 9 represent a calibration of a 0.001cc volume with 5000 ppmv moisture in a nitrogen balance. The moisture content was determined using a NIST traceable dew point hygrometer in a stable gas flow of humidified nitrogen. Each data point represents the summation of 500 spectra, which were acquired 20 μs apart.

The measured pump out curve for nitrogen, mass 28 was normal and fit a typical exponential function with little to no conduction effects. The white data points represent the displayed mass. The green curve represents the total ion count. The measurement time to generate the main portion of the pump out curve is approximately 1.0 seconds.
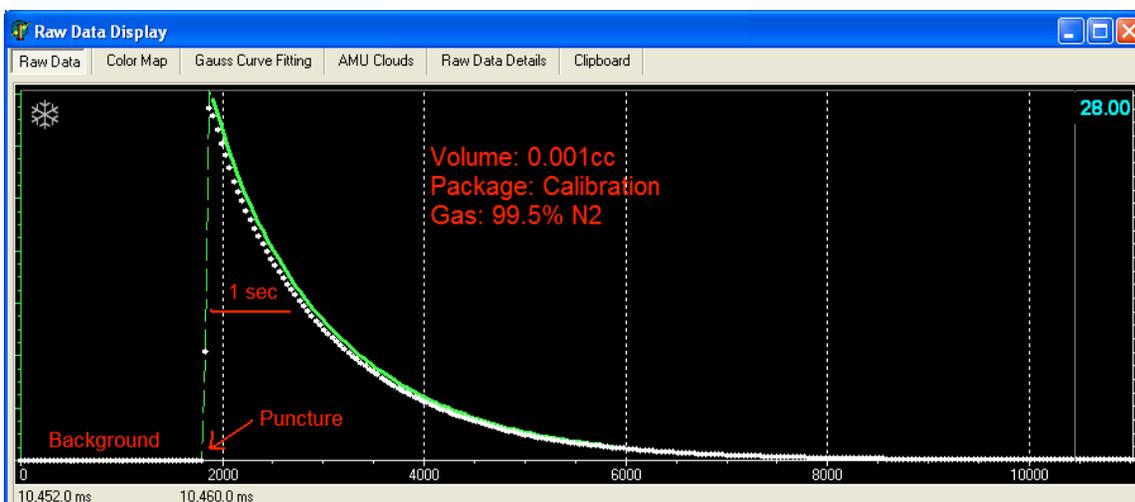
Fig. 8. Mass 28 pump out curve for 0.001cc calibration at 99.5% $N_2$.

Figure 8 shows the 5000 ppmv moisture pump out curve for the calibration run. The large number of data points on the graph actually shows the signal rise at the time of puncture. The rise portion of the curve is important.
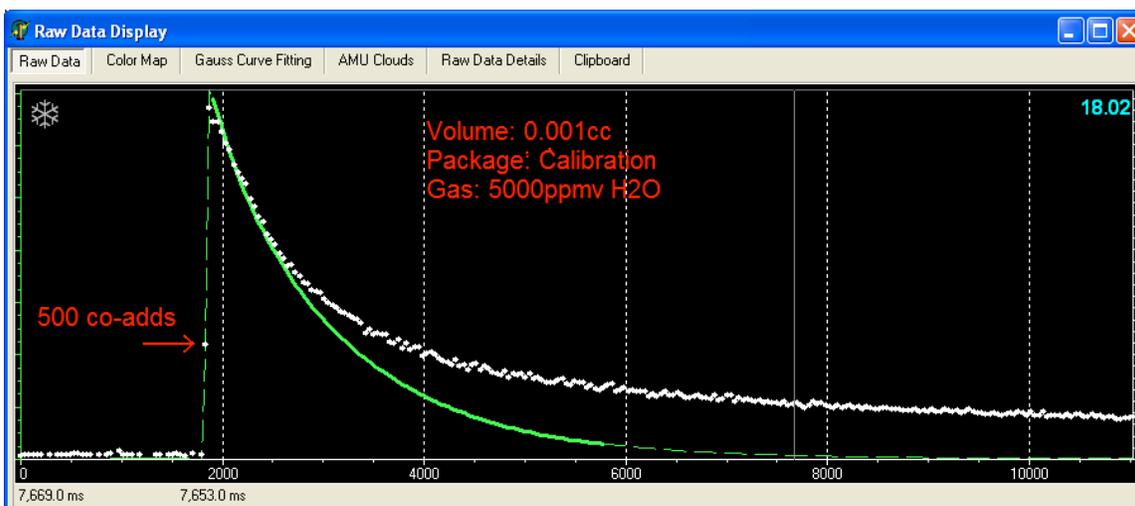


Fig. 9. Mass 18 pump out curve for 0.001cc calibration at 5000ppmv.

The sample was then tested. The nitrogen pump out curve was very similar to the calibration sample (figure 8.) The moisture pump out curve from the test sample package is show in figure 9. Note that the total ion count (green curve) is similar to the calibration pump out curve but the moisture behavior is quite different. The initial point of puncture is preceded by a level background spectra and followed by a steady increase in moisture content. It is evident from the curve that the device continually outgassed moisture over time after puncture. The device, when punctured, is exposed to an initial high vacuum ($\sim 1 \times 10^{-9}$ torr) and comes to a maximum equilibrium pressure of $\sim 1 \times 10^{-5}$ torr during analysis. This drop in internal pressure changes the moisture vapor pressure over the getter and causes it to release moisture. For reference, the moisture in the atmosphere before puncture and at the instant of puncture is essentially only a few ppm. The question becomes, "What are you trying to measure?" Are you measuring real or induced effects in the device?
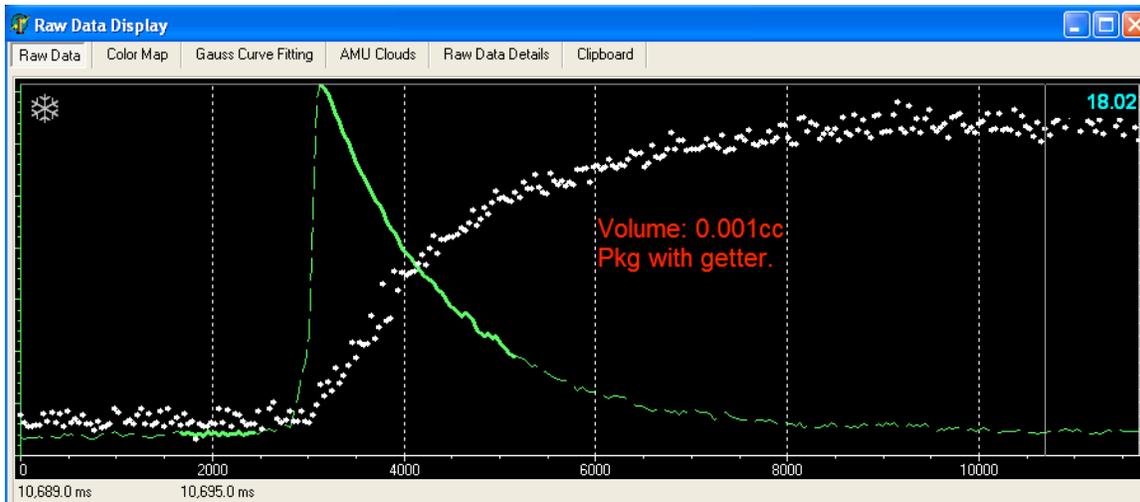
Fig. 10. Mass 18 pump out curve for 0.001cc package.

The speed of the TOF system allows one to look at the outgassing affects from the time of puncture and eliminates surface interactions that may result from long analysis times. At the time of puncture, there is virtually no moisture in the atmosphere of the device. The moisture getter is doing its job. After exposure to vacuum, the getter gives up its moisture. The amount that it gives up is known qualitatively from the graph but not quantitatively, because there is no way to calibrate the meaning of the curve.

## 6.2 Example 2

A MEMS device with a $2X10^{-4}$ cc internal volume with an internal pressure of 0.1 torr was analyzed to determine the baseline gas composition and any outgassed species. This device is with out a getter. The effective volume at 1 atm was calculated to be 0.25 nL. The device was heated to 100°C for 16-24 hours and tested at 100°C. The raw data for mass 18 (Moisture), 28 (Nitrogen), 40 (Argon) and 44 (Carbon Dioxide) are shown in figures 11 through 12.

The pump out curve for mass 40 is shown in figure 11. The background level was extremely low at $<1X10^{-9}$ torr prior to puncture. The rise in mass 40 intensity can clearly be shown with 5 data points covering the front part of the curve. The number of data points can be changed by increasing or decreasing the co-adds. For faster sampling, when a pressure pulse is small, a lower co-add is necessary. A secondary view of the selected segment line or single co-add data point is shown below the pump out curve. The peak intensities for all masses from 0 to 106 AMU are shown. The gas concentrations reveal a mixture of ~55% argon, ~30% nitrogen, ~10% $CO_2$, ~5% $H_2$ and no detectable levels of $H_2O$.

Below the main curve in figure 11 is the complete mass spectrum present at the data point called out by the label "Segment Data" in the main curve. This figure demonstrates that all masses, not just the balance gas of argon, are recorded and available for review for each segment captured.
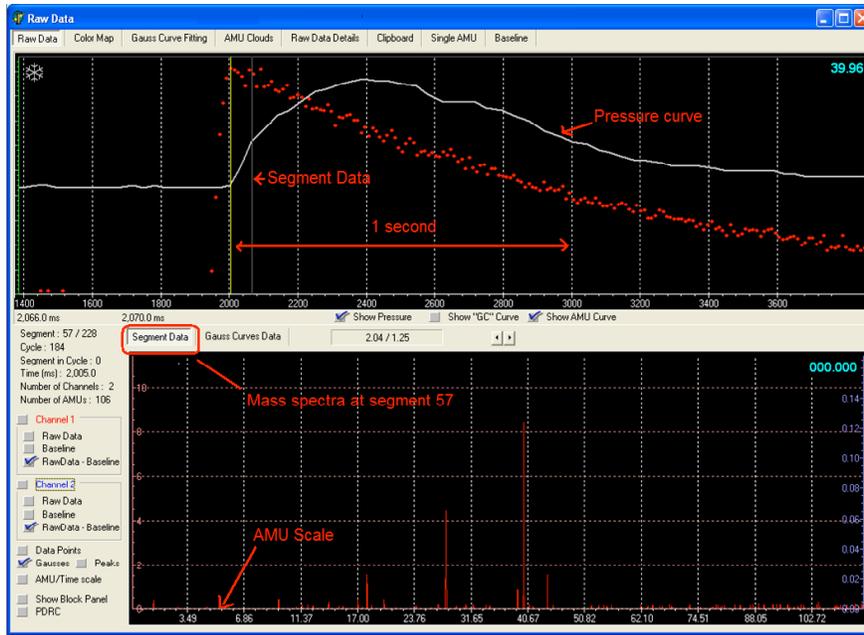
Fig. 11. Mass 40 argon pump out curve for .25nL volume.

Figure 12 shows the moisture (AMU 18) data. As shown before, the white curve is the total pressure over time and the red curve is for the species of interest, in this case moisture. There is no increase in the moisture data above the signal noise level during the gas pulse after puncture indicative of a getter dumping moisture. The data also points to a good hermetic seal with no influx of water through a leak. The package is bone dry.
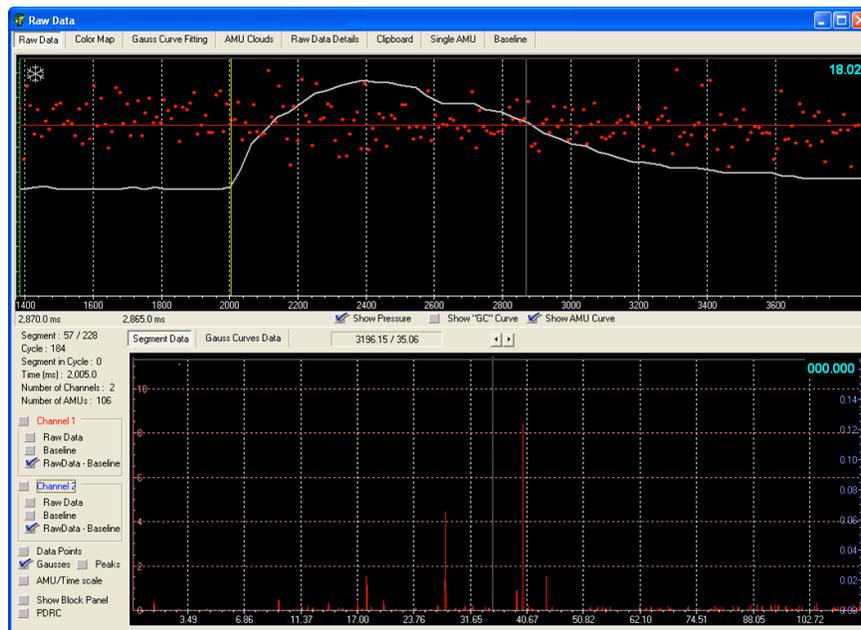


Fig. 12. Mass 18 moisture pump out curve for .25 nL volume.

# 7.  CONCLUSIONS

The ability to measure single sample volumes below 1 nL is new to the industry with ORS' development of the HR-IVA. The HR-IVA technique allows process engineers and material scientists a way to quantify, diagnose and differentiate between leaks, outgassing, and permeation in very small samples. This is particularly important in these very small volume devices where the surface area to volume ratios are extremely large and failure mechanisms other than classical leaks loom large. In addition, the sealing environments and test process effects of pressure and temperature can be studied to generate accurate and reproducible data.