

# Inadvertent Under-Reporting of Moisture

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## ABSTRACT

The author has been involved in the analysis of moisture in hermetic structures since 1966, and has designed and built numerous commercial systems over the past 50 years based on gas chromatography and mass spectrometry principles in order to achieve accurate measurements. This paper is, in fact, an attempt to bring attention to a number of significant problems with the above test method and to generate interest in resolving these problems in a clear, unambiguous way.

## INTRODUCTION

The purpose of TM-1018 is to determine how much moisture is contained in a hermetic structure and is available to cause a moisture related failure. Between 1973 and 1978 numerous moisture measurement workshops were held at NBS (now NIST) in order to establish criteria for the calibration and measurement of semiconductor packages using mass spectrometry. Since water is a polar molecule, it doesn't follow any accepted vacuum theory such as the ideal gas law. The proper use of moisture calibration transfer standards and the generation of known amounts of moisture in atmospheric air were detailed, as well as, the proper analytical technique for sample introduction and analysis by mass spectrometry. The workshops were attended by representatives of all the analytical laboratories involved in these measurements at the time, so when the Mil-Std TM-1018 was introduced in 1978 and did not contain any of the above criteria, there was very little concern. Correlation studies were initially conducted by Rome Laboratory using actual production packages and it was quickly determined that the use of a multivolume calibrator was necessary, since the standard deviations of moisture contained in actual production packages at the time were too broad to prove useful. EAG Laboratories agreed to design, build, and deliver multivolume calibrators to any requesting laboratory for our actual manufacturing cost. Over 150 calibrators were manufactured and supplied to mass spectrometer laboratories in over 22 countries. It became apparent that the multivolume calibrator was very useful for many mass spectrometer applications other than the analysis of semiconductor packages.

## THE PROBLEM

Since TM-1018 does not contain any information as to the proper use of moisture transfer standards nor the appropriate criteria for sample introduction and analysis by mass spectrometry, it is no

wonder that laboratories which began operation in the 80's, 90's, and later are at a disadvantage. We have all heard the term, "shop for the lowest moisture measurements", since the amount of moisture measured in a package has a pass/fail limit, and would result in lot jeopardy if two packages out of 5 failed the 5000ppm limit established by DESC (now DLA). The following paragraphs are an attempt to identify the independent variables in the calibration procedures and design of a mass spectrometer system which can result in under-reporting the measured moisture, as well as Oxygen.

## MEASURING MOISTURE

A mass spectrometer can be a very precise instrument able to achieve a precision of better than +/- 1% of measured value, however, accuracy will be determined and possibly limited by the accuracy of the standards used to calibrate the instrument. In 1952 NBS published information on the measurement of moisture in atmospheric air, and again in 1961 on the NBS standard hygrometer. Transfer standards are used to calibrate commercial hygrometers which we use to determine the moisture levels being introduced to the mass spectrometer during calibration. The NBS standard and transfer standards are all based on making measurements of moisture in atmospheric air at a flow rate of 1.0 cubic foot hour. Using a commercial hygrometer with gasses other than atmospheric air at flow rates higher than 1.0 CFH brings into question the accuracy of the measurement. In general, the dew point measured will most likely be lower for a given amount of moisture than would be measured using air and the proper flow rate. This will result in biasing the mass spectrometer to provide lower measured values of moisture, since a larger amount of moisture is being defined as a smaller value during calibration.

## MULTIVOLUME CALIBRATOR

The multivolume calibrator developed by EAG Laboratories is a convenient method of introducing calibration gasses into a mass spectrometer. The proper use of the calibrator when introducing moisture standards is required to ensure accuracy of the measured values. It is imperative that the calibrator be placed in the flow stream just ahead of the hygrometer and operated at the same temperature (room temperature). The practice of elevating the temperature of the calibrator will result in the introduction of a greater amount of moisture than is being measured by the hygrometer, which again, biases the mass spectrometer to provide lower measured values.

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## SAMPLE INTEGRATION

During the transfer and introduction of gas samples containing moisture, the measured data must be integrated over time to insure that all the moisture in the sample is accounted for. The practice of using short integration times or no integration time, again, results in under-reporting the amount of moisture contained in the sample.

## SAMPLE INTERACTIONS

Many mass spectrometers utilize an RGA as the mass analyzer. Mass separation techniques can include magnetic, double focused magnetic, monopole, quadrupole, three dimensional quadrupole, or time-of-flight mechanisms. Most RGA instruments utilize a space charge limited ion source which is highly sensitive but also very nonlinear as a function of partial pressure. Making measurements at pressures greater than  $1.0E^{-6}$  Torr can result in ion/molecule interactions making quantitative measurements difficult. This is usually of little concern since an RGA is a qualitative instrument not a quantitative instrument. The effective ion source temperature is also important, since we know that thermal disassociation of water molecules occur at around 2200 deg K. Exposing the moisture sample to ion gauge filaments also results in disassociation. TM-1018 originally required the volume of the sample being analyzed to be measured using a capacitance manometer (Baratron) which is a fundamental pressure measuring device whose measurement is independent of the composition of the gas sample and more importantly doesn't react or modify the gas sample. Since these high temperature filaments disassociate water and consume Oxygen, the net result is under reporting of both moisture and Oxygen and usually indicating an increased level of Hydrogen in the sample.

## DIRTY VACUUM SYSTEMS

The background of a mass spectrometer is usually measured prior to sample introduction and is subtracted from the measured data

when producing the final analytical result. One of the problems with this procedure is: "at what level or pressure is subtraction valid?" When total background pressures are greater than  $1.0e^{-8}$  Torr prior to sample introduction, making background subtractions are not analytically viable. The reason is, that when the sample is introduced, the total pressure in the system increases which tends to suppress or decrease the partial pressures of the background. In most cases, the principle background is moisture, thus subtracting a larger amount than is actually present results in under-reporting the amount contained in the sample.

## CONCLUSION AND RECOMMENDATIONS:

The above variables become dominant as the package size is reduced. Small packages such as a UB or even smaller MEMS packages can be used to identify measurement problems when comparing laboratory data. Since the under reporting of moisture can have unexpected consequences, one recommendation would be to change the method of package submission for qualification. It is suggested that four packages be randomly selected from a production lot and two each be submitted to two different DLA suitable laboratories having different mass spectrometers. If the data does not correlate, then DLA must investigate the situation and mitigate the problem. One only need be reminded of the one month delay in the launch of the very first Space Shuttle because a power transistor failed in a power supply during the count down. The failure was cause by 1.5% moisture level contained in the package, and all the spare parts had similar moisture levels. How did this part ever get qualified? The cost of this failure was estimated to be more than \$20 million. Changing measurement and calibration recipes to force small packages to pass the 5000ppm moisture limit should not be allowed. It is further recommended that moisture pass/fail levels be increased for small packages to reflect the internal surface area to volume ratio adjustment previously proposed to DLA, JEDEC, and G-12 by Micropac and EAG.