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U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Semiconductor Measurement Technology:

ARPA/NBS Workshop V
Moisture Measurement Technology for Hermetic Semiconductor Devices

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Proceedings of the ARPA/NBS Workshop V
held at the National Bureau of Standards,
Gaithersburg, MD

March 22-23, 1978

Edited by

Harry A. Schafft, Stanley Ruthberg, and Elaine C. Cohen

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PREFACE

The Workshop on Moisture Measurement Technology for Hermetic Semiconductor Devices was conducted as part of the Semiconductor Technology Program in the Electron Devices Division of the National Bureau of Standards (NBS). This Workshop is the fifth in a series dedicated to the furtherance of the measurement technology needed by the semiconductor device industry in its attempt to provide to its customers products that are based on the most advanced technology, yet have high reliability and the affordable costs which result from high yields. It was held at the Gaithersburg, Maryland, facility of the National Bureau of Standards on March 22-23, 1978, under the cosponsorship of the National Bureau of Standards and the Defense Advanced Research Projects Agency, with the concurrence of the Rome Air Development Center. Representatives from industrial, governmental, and academic organizations concerned with device manufacture, analysis, and instrument design participated in this workshop. Speakers were selected from among the finest and most active researchers in the field. The outcome was a large and enthusiastic workshop attendance.

The workshop was concerned with present problems with the measurement of moisture in hermetic semiconductor devices. While moisture-induced failure modes and mechanisms had received considerable attention in the published literature and in meetings, the accurate and reliable measurement of the moisture content had not; yet, this lack of measurement of moisture reliability is a major obstacle to meaningful efforts to limit and control this pervasive contaminant. The workshop featured 20 formal talks and several group and panel discussions. The 150 attendees represented a broad spectrum of the semiconductor and related communities concerned with moisture measurement.

The workshop was chaired by H. A. Schafft of NBS. S. Ruthberg of NBS served as Technical Advisor and R. W. Thomas of RADC was the third member of the Planning Committee. E. C. Cohen of NBS coordinated workshop arrangements. J. S. Halapatz of NBS typed the workshop report.

It is hoped that this report will convey the spirit of the workshop to those who could not attend.

Disclaimers

Semiconductor Measurement Technology: Moisture Measurement Technology for Hermetic Semiconductor Devices

— A Workshop Report —

Chairman: Harry A. Schafft
Technical Advisor: Stanley Ruthberg
Coordinator: Elaine C. Cohen

ABSTRACT

The workshop, fifth in a series concerned with measurement problems in integrated circuit processing and assembly, served as a forum to examine present problems with the measurement of moisture in hermetic semiconductor devices. While moisture-induced failure modes and mechanisms had received considerable attention in the published literature and in meetings, the accurate and reliable measurement of the moisture content had not; yet, this lack of measurement of moisture reliability is a major obstacle to meaningful efforts to limit and control this pervasive contaminant. Manuscripts and summaries are provided of 19 talks, panel meetings, and group encounters on three major topics: mass spectromoter measurements of internal package moisture, moisture sensors, and package analysis and qaulity assurance.

Key Words: Analysis of moisture content; mass spectrometer measurement; moisture; moisture generators; moisture sensors; quality control; reliability of semiconductor devices; semiconductor devices.

1. INTRODUCTION

A Workshop on Moisture Measurement Technology for Hermetic Semiconductor Devices was conducted by the National Bureau of Standards at its Gaithersburg, Maryland site on March 22-23, 1978. It was the fifth in a series of ARPA/NBS workshops concerned with the measurement problems in integrated circuit processing and assembly.

The workshop served to bring together workers in the field to examine present problems with the measurement of moisture in hermetic semiconductor devices. While moisture-induced failure modes and mechanisms had received considerable attention in the published literature and in meetings, serious problems with the accurate and reliable measurement of moisture content had not. The workshop was designed to address this gap.

The accurate determination of moisture content in hermetic semiconductor devices is fraught with pitfalls. These pitfalls present a major obstacle to meaningful efforts to limit and control this dangerous and pervasive contaminant. At the time of the workshop it appeared that while single-laboratory precision of mass spectrometer measurements were adequate, interlaboratory measurement precision was poor. That made the accuracy of reported values of moisture content suspect, and thwarted efforts in relating moisture content to device failure. It appeared that the use of moisture sensors was becoming more practical. While the availability of such sensors appeared to open up new opportunities for measuring and monitoring moisture content, it also required that a number of questions be addressed. Of significance to the reliability community was that both mass spectrometer and moisture sensor measurements were to be used in Test Method 1018 of MIL-STD-883B (Interval Water-Vapor Content) which was soon to become effective. It was in this context that the workshop was held.

It was only natural, therefore, that the measurement approaches addressed in detail were those using mass spectrometers and moisture sensors (aluminum oxide and surface conductivity types). The main conclusions reached at the workshop were: (1) for all of the methods used to make measurements in device packages, one can only talk about measurement precision — not accuracy; (2) none of the measurement techniques available is ready for use by inexperienced personnel; and (3) the precision or repeatability of moisture measurement will not be satisfactory until much more is done to characterize mass spectrometers and moisture sensors in terms of all the variables, conditions, and applications that can affect the measurement.

The format of the workshop included a mix of formal presentations, group discussions, and group encounters with some of the speakers. The first day was devoted to presentations on the use of mass spectrometers and moisture sensors to measure moisture content, and to discussions for developing responses to questions regarding the variables which affect measurement results and the specification of measurement data. Two panels met in the evening to define the state-of-the-art and what needs to be done for each of the two approaches. The work of the panels was presented for refinement and discussion in the afternoon of the second day. In the morning of the second day, presentations were given on aspects of military standard tests for moisture content and on a number of different approaches related to moisture measurement. Following these presentations, the audience was divided into three groups. Three pairs of speakers moved separately to each group, in turn, for questions and discussions. Summaries of these encounters were given in the afternoon followed by the panel reports and open discussions.

2. SESSION I MASS SPECTROMETER MEASUREMENTS

2.1 Mass Spectrometer Measurements at RADC

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Since the late 1960s RADC has been actively involved in mass spectrometric microelectronic package ambient gas analyses. As reported by Thomas *et al.* [1], a system was developed and used to determine the effects of various contamination of the package ambient on the reliability of the contained device. It soon became apparent that moisture, even in small quantities, could have serious detrimental effects on the operational integrity and reliability of integrated circuits. The severity of these effects depends upon the matrix of contamination with a package, i.e., moisture concentration, organic and/or inorganic contamination, the technology of the contained device (whether classical digital, linear, or MOS type), and the various stresses to which a package could be subjected. The result of these experiences in moisture measurements led to the creation of Method 1018 of MIL-STD-883B which sets an upper bound on the acceptable moisture concentration contained within a militarily acceptable device. This upper limit of 5000 parts per million by volume (ppm_v) was chosen so that if and when the package was cooled, the moisture within the package ambient would freeze out (frost point) rather than condense on the surface of the contained device. In order to assure accurate measurements of moisture vapor, requirements for certification of analytical facilities and methods were established. This paper will describe efforts to assure mass spectrometric moisture measurements at RADC and will present some of the interesting phenomena discovered during this program.

During approximately ten years' experience of mass spectrometric measurements at RADC, a set of criteria for a desirable analysis system evolved. These criteria included refinements of the original batch processing system, removal of inherent limitations on the size and type of package that could be handled, and improvements in the flexibility and throughput of the overall system.

These features included a rapid cycle capability. The original batch system required a 12- to 24-h 125°C vacuum bake process to reduce the system moisture background to an acceptable level. All packages were therefore subjected to a time-temperature stress that made it impossible to separate moisture contained in the package ambient from moisture adsorbed and/or outgassed from materials within the package. In order to circumvent the long bake cycle, a system was needed that would allow minimum exposure of system walls to atmospheric contamination during package loading. Also, by reducing or eliminating this bake cycle, time-critical analyses would not be delayed. Secondly, the batch system did not have the ability to reliably and reproducibly control the gaseous constituents under more than one condition. Therefore, as requirements for analysis of larger and larger microelectronic packages increased, the capability of the system to perform accurate analysis decreased, since the pressure in the mass analyzer rose into a nonlinear operating range. Attempts to limit the pressure within the analyzer resulted in distortion of the gaseous ambient due to selective separation of components by limited conductance vacuum valves. Therefore, a system was required that would feature variable conductances, thus controlling the pressure in the mass analyzer independent of the size of the package analyzed. An additional requirement was the ability to compare different techniques used for mass spectrometric gaseous analyses. These included both static sample-and-hold techniques and dynamic flow sampling. In order to study the difference between ambient water vapor and absorbed moisture, the system should also feature variable sample temperature.

The result of our criteria is the Rapid Cycle mass spectrometer system pictured in figure 1 and schematically presented in figure 2. Sample holddown and release is accomplished through manipulation of V6, a three-way valve. The sample chamber is roughed by a mechanical pump through V4. V5 provides access to a calibration and backfill port that includes a micrometer valve. A high-voltage probe is mounted in the sample area and when high voltage is applied at less than 5×10^{-2} and more than 1×10^{-3} Torr, a plasma discharge occurs that helps to clean the system surface. A capacitive micrometer, D1, monitors the sample chamber pressure. Valve V2/C1 has an orifice machined into its seat and therefore can act as either a high or a low conductance. V3 can be used to regulate the amount of gas flowing into the mass analyzer through C2, a fixed orifice, by controlling upstream pressure. V1, when open, gives a large direct conductance to the mass analyzer and is used mainly to reduce the sample system background prior to analysis. An ionization gage is supplied which is helpful in measuring ionizer pressures during ambient gas analyses. The mass analyzer is pumped by a direct drive turbomolecular pump backed by a mechanical pump. By choosing combinations of valves V2 and V3, one can allow for dynamic (V2 open, V3 closed), static (C1 in, V3 closed), or differential (C1 in, V3 open) type flow systems. The choice of sampling depends, in part, on the sample volume of gas, the maintenance of acceptable pressure levels within the mass analyzer, and the selected sensitivity of the mass analyzer to partial pressures of gas (gain of instruments, emission current). The key to effective, clean system operation is that the sample gas-handling portion of the system is contained within one small block of stainless steel. This block is shown in figure 3.

The scale of this drawing is approximately 1:1, indicating that care has been taken to minimize the total gas sampling surface area. The proximity of the valves and the sample to the analyzer, along with the relatively small exposed surface area promote a clean background that can be quickly attained even after sampling a wet volume of gas from a microelectronic package. The inlet block is maintained at 120°C to minimize surface adsorption of moisture and other condensible species. A minimum amount of deviation in the line of sight of the spectrometer to the sample has also been designed into the inlet block. The puncture pin assembly and V3 are perpendicular to the plane of the drawing, and thus does not show in this cross section. The sample holder is also perpendicular to the plane of the drawing and mates to the sample lid O-ring. The sample holder features removable chucks to allow rapid compensation for change in sample size and configuration. The sample holder is surrounded by fluid lines that allow either cooling (cold water) or heating (hot gas). Not shown is a thermocouple which mounts through the sample holddown vacuum line and abuts directly on the sample chuck. The chuck assembly is held in place by a vise and is securely sealed to the sample chamber by a viton O-ring. Isolating the sample chuck from the sample chamber is accomplished by a viton O-ring which is concentric to the puncture pin channel and impinges on the top of the sample. Therefore, only a very small area of the package undergoing analysis is exposed to the interior of the high vacuum gas inlet block. This precludes the necessity of vacuum baking the surface area of the package and the sample holder prior to analysis. Application of a positive pressure of an inert, dry gas through V5 will prevent atmospheric diffusion into the sample chamber during loading. A typical analysis from sample loading to puncture takes about 10 min.

A sample is loaded on the sample chuck and vacuum is applied via V6 with V3 on. The vise is then closed and V4 opened. As V4 is evacuating the roughing chamber, TC2 is monitored to assure that gas is not being leaked by the O-ring seal to the top of the package. V2 is then opened and the system is evacuated to a pressure of 10 μm as read on TC2. V3 is then closed and V1 opened. The mass spectrometer is now used to monitor gases in the sample chamber. After no major change is noted in the background peaks for 1 min, V1 is closed and V2 opened. Once equilibrium is obtained, a background is taken. The package is then punctured. After analysis, V2 and V4 are closed, the locking vise is released and V6 is moved to the sample removal (eject) position. V5 is opened to allow positive flow of an inert gas out of the sample chamber. Another sample can then be loaded and the process repeated.

The principal means of water vapor calibration of the batch system consisted of measuring the room relative humidity and converting to percent moisture using a relationship developed from Amagat's Law of Partial Volumes

$$\%H_2O = \frac{(RH)(P_{H_2O})}{P_{ATM}}$$

. However, this gave only a one-point calibration whose level was uncontrollable. In order to determine the moisture sensitivity of the Rapid Cycle system over an expected range of interest (1000-10 000 ppm_V), a divided flow moisture generation system was built. Figure 4 is a block diagram of this system. Dry nitrogen flow from a liquid nitrogen source is divided into two sections. One section is bubbled through 18 MΩ water maintained at 50°C. The other acts as a dilution flow. The resultant wet nitrogen flows through a short section of tubing also maintained at 50°C and is bubbled through 18 MΩ water at 19°C. Since the incoming flow is saturated with respect to the 19°C ambient, 100-percent saturation is assured leaving the second saturator. This saturated carrier gas is then immediately combined with the dilution flow. This effectively drops the dew point such that moisture will not condense in the combined flow at room temperature (20°C or greater). The combined gas flow goes through a temperature stabilization coil into a stainless steel moisture sensor calibration module maintained at 25°C. The calibration gas then leaves this manifold and flows by the Rapid Cycle system inlet where it is sampled. The flowmeters have been calibrated at the outlet of the system using a mercury piston flowmeter calibrator. This negated the need to compensate for the back pressure of the saturators.

A comparison was made between the Rapid Cycle mass spectrometer system, when calibrated with room relative humidity, the determination of flow stream concentration of water vapor and the predicted moisture, converting for nonideal behavior generated by the divided flow system. The results of this experiment are given in figure 5. Over the range of calibration the Rapid Cycle mass spectrometer system has a linear response that exhibits practically a one-to-one relationship with the divided flow system. Therefore, the relative humidity calibration technique is accurate from 1000 to 7000 ppm_v of moisture. The short-term (8-h) stability of the Rapid Cycle mass spectrometer system calibration factors for water vapor was confirmed by measuring relative humidity and determining this factor periodically during a typical analysis run. The results of this experiment are shown in figure 6.

Prior to the moisture calibration experiments, attempts were made to correlate analyses performed on the batch system with the Rapid Cycle system measurements. It was discovered that an appreciable amount of moisture detected by our batch system was not being seen by the Rapid Cycle system. A subsequent investigation revealed that the plasma discharge preanalysis technique used to accelerate system cleanup between analyses was the apparent loss factor. Later it was discovered that, in addition to the plasma discharge effect, backfilling and purging the system with argon or other inert gases resulted in similar behavior. Figure 7 shows the behavior of moisture released during a typical gas analysis run in the Rapid Cycle system after the system had been backfilled to 1 atm with room air with no positive pressure of gas flowing from the sample chamber. The long-term pump-out exponential curve was anticipated based upon our previous experience. Next, an analysis on a similar part was performed with air backfill and no positive pressure. However, during system evacuation prior to analysis, a plasma discharge was activated for 1 min. The water vapor peak intensity was then monitored as a function of time after package puncture. As pictured in figure 8, the peak decreased rapidly within a few seconds after puncture and decreased to background levels within 4 min. Figure 9 shows the effect of replacing the air backfill with argon at a positive pressure preventing atmospheric diffusion into the sample chamber. Again, the intensity of the water vapor peak decreased dramatically (approximately 40 percent) within the first few seconds after package puncture and background level was achieved within 4 min. Apparently, both plasma and again backfill were conditioning the system surfaces such that moisture was immediately adsorbed onto them and did not desorb within the time span of normal analysis. Figure 10 shows the result of triplicate analysis run on similar samples for the purpose of determining the magnitude of this adsorption-desorption phenomena.

Since the air backfill technique resulted in long moisture residence times within the system, extended analyses for longer than normal times could integrate increasing quantities of moisture into package ambient gas analysis. Triplicate determinations on similar samples were made at increasing analysis (integration) times. The results of this experiment are shown in figure 11. The data points represent the average of three determinations with the deviation bars representing the minimum and maximum values obtained. Within the limits of our data handling and storage capabilities (200 s of data accumulation), there is no appreciable change in reported moisture concentration with increased percent of available gas sampled.

One possible way of distinguishing between ambient moisture within a microelectronic package and total moisture (moisture adsorbed on surfaces and absorbed in materials used within the package) is to perform comparison analyses of similar samples at room temperature (25°C) to determine the ambient water vapor concentration and at elevated temperature (100°C) to liberate "bound" moisture within the package. The results of increasing sample temperature on the determined moisture concentration are shown in figure 12. The samples used in this experiment contained an epoxy die attach material so that differentiation between the contribution of adsorbed and absorbed moisture to the total is not possible. However, it has been shown by Czanderna [2] that epoxy materials release moisture with increasing temperature. Figure 13 illustrates the results of an experiment to determine if (1) the package had obtained thermal equilibrium with the normal analysis time (approximately 10 min), and (2) if the desorption of moisture with the package would continue once thermal equilibrium was reached. Generally, the package did reach thermal equilibrium and no additional moisture outgassed within the experimental time frame.

In order to compare analytical processes requiring an extended high temperature bake prior to analysis with the Rapid Cycle analytical technique (no bake), a sample time at 125°C experiment was performed. The results of this determination are presented in figure 14. The zero time figure is from data taken at 100°C under normal analytical conditions. The 1-h point suggests increased moisture outgassing from the epoxy die attach at 125°C. Some of the ambient moisture then apparently reacts with materials within the package as evidenced by the depressed moisture concentrations at the 4- and 8-h points. This reduction in moisture has been noted in other analyses where the packages had been subjected to elevated temperatures (125, 200, 250°C) for varying lengths of time up to 4000 h). After 24 h at 125°C, the average moisture concentration returned to the 1-h level, but the large deviation between the minimum and maximum suggests emphasis of differences in the quality of the epoxy die attach material and its subsequent handling (curing, etc.).

After characterization of the Rapid Cycle system was completed, several analyses of packages containing known amounts of moisture were performed. Good agreement was obtained on those samples containing 4000 $\mathrm{ppm_v}$ moisture or more. However, when analyses were performed on samples containing 500 $\mathrm{ppm_v}$ or less moisture, the Rapid Cycle system reported moisture concentrations from 2000 to 3000 $\mathrm{ppm_v}$. This suggested that the air backfill technique, which was necessary to prevent adsorption of moisture from relatively "wet" packages onto sample chamber walls, was conditioning the chamber walls such that moisture was being desorbed during analysis of dry samples. The results of an experiment to characterize this phenomena are detailed in figure 15. A valve with minimum dead space was attached to the Rapid Cycle system inlet and used to simulate a microelectronic package analysis by bursting gas into the system to a pressure similar to that encountered during analysis of the moisture standards. Room air backfill was necessary to accurately analyze relatively high moisture samples but introduced error in analysis of low moisture concentration samples. The results of this experiment indicate that more work must be done to characterize and compensate for the system moisture adsorption-desorption behavior.

Other experiments and work to be done include further comparison of moisture determinations from solid-state sensors, the Rapid Cycle mass spectrometer system, and the divided flow generation system. The necessity of acquiring a dew point apparatus will be investigated, and, if needed, one will be acquired as transfer standard. Comparisons should be made between *in situ* sensor measurements and Rapid Cycle mass spectrometer system calibrated in different modes (burst, flow, low, and high moisture) in order to determine possible correction factors (transfer functions) to compensate for various package volumes and concentrations. Traceability to the National Bureau of Standards (NBS) can be obtained by having a dew point apparatus calibrated at NBS and/or having solid-state moisture sensors calibrated at NBS and used in the laboratory as a laboratory standard.

In conclusion, it appears that known concentrations of moisture can be generated and independently measured accurately by several different analytical instruments. Furthermore, assurance of generation of accurate quantities of moisture should allow characterization of the mass spectrometer system response with respect to adsorption-desorption phenomena and for calibration of solid-state sensors which, in turn, may be used to confirm the Rapid Cycle system moisture response. Also, the Rapid Cycle technique for quantitative mass spectrometric analysis of microelectronic package gaseous ambients promises to be a viable analysis technique.

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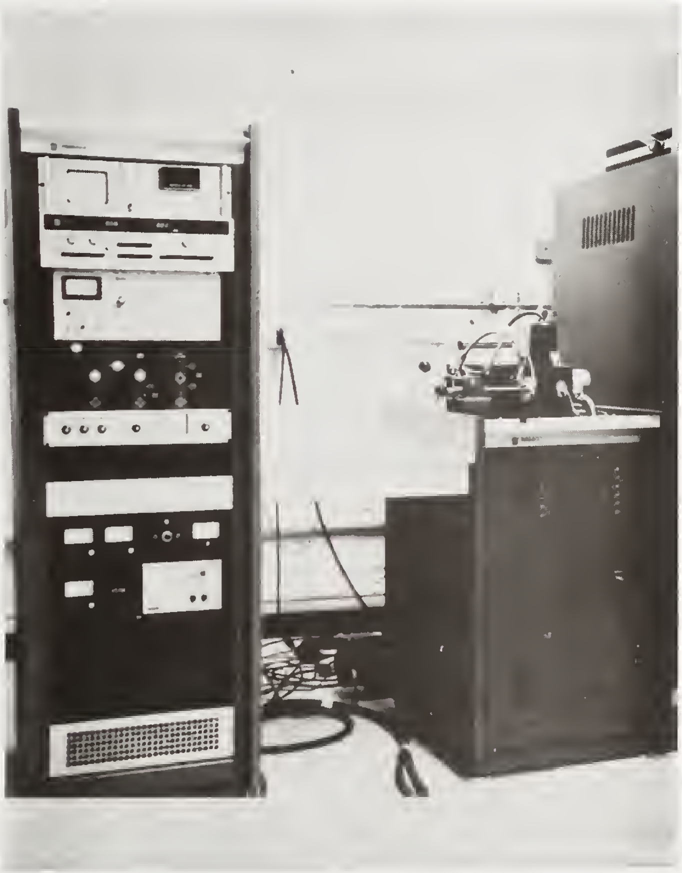

Figure 1. Photograph of a Rapid Cycle system.

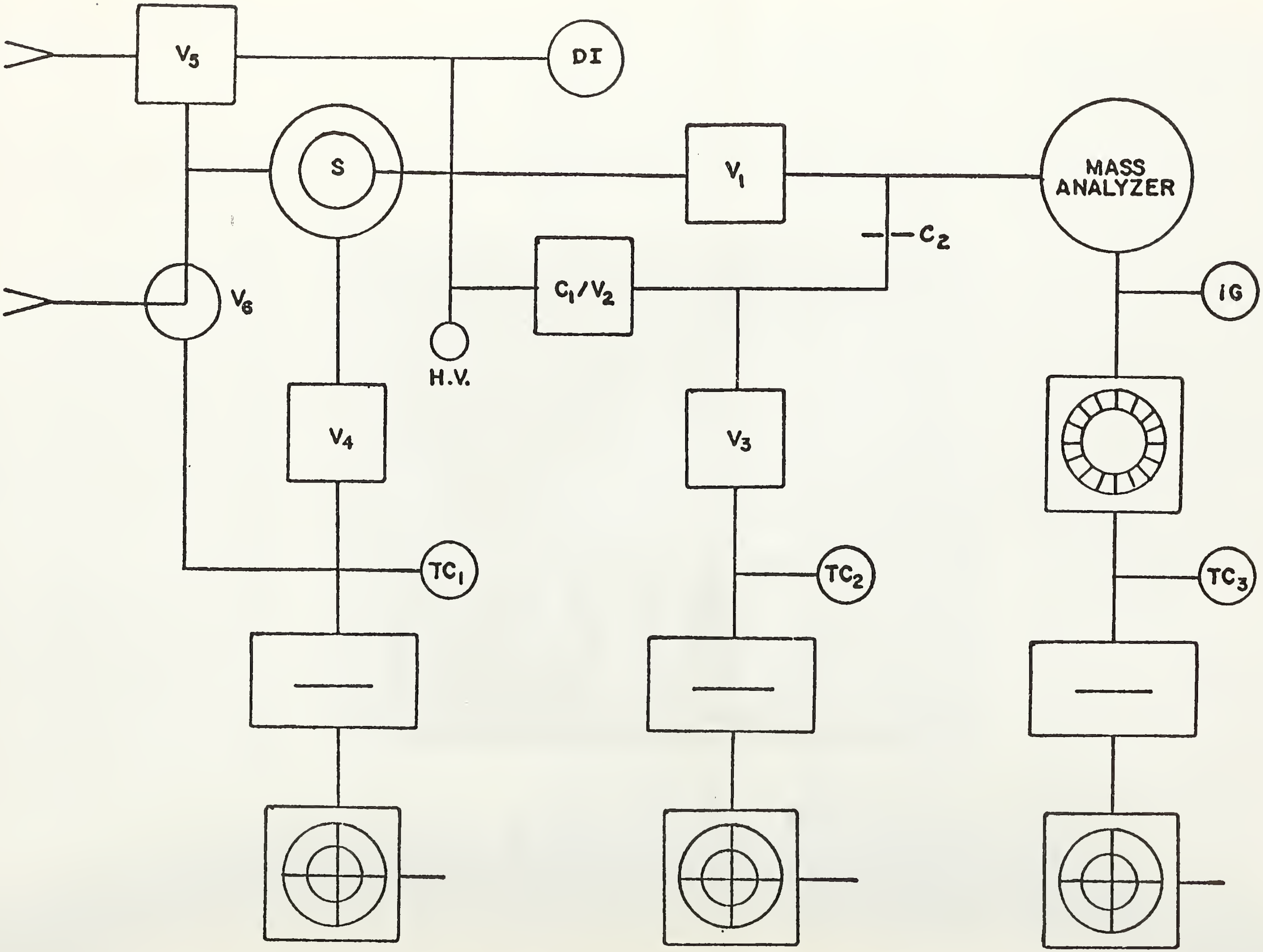

Figure 2. Rapid Cycle mass spectrometer system.

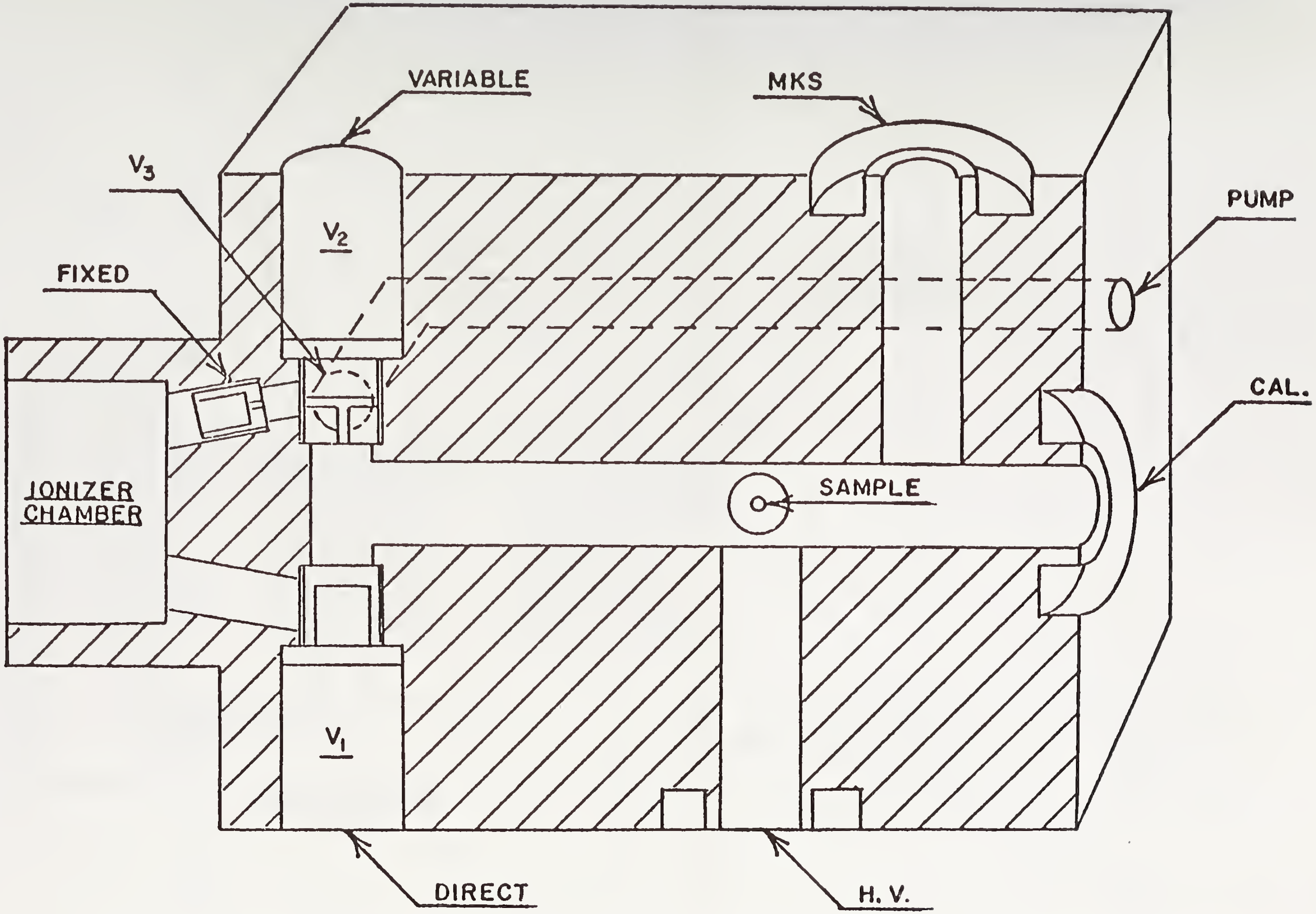

Figure 3. Rapid Cycle inlet block.

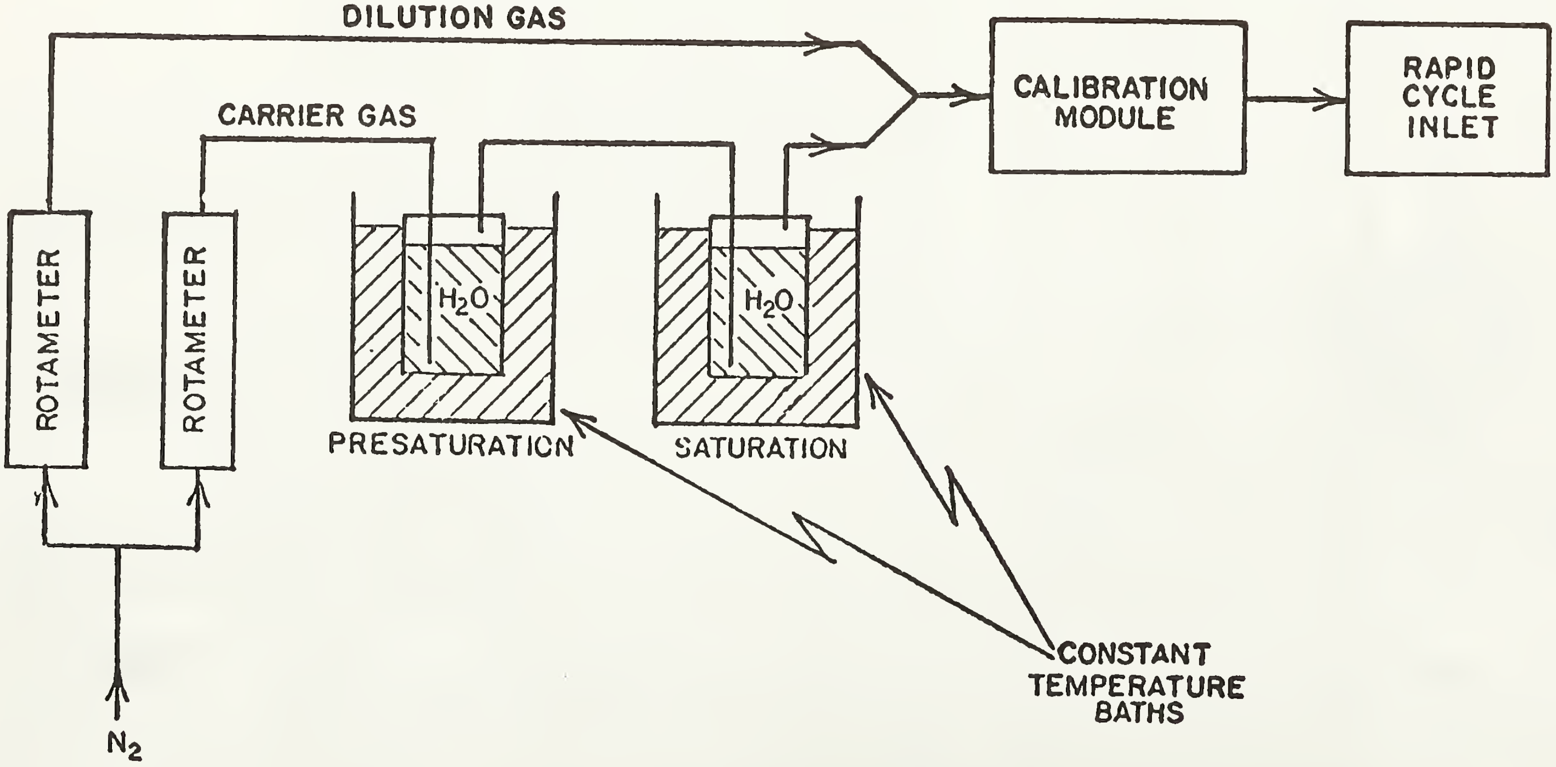

Figure 4. Divided flow system.

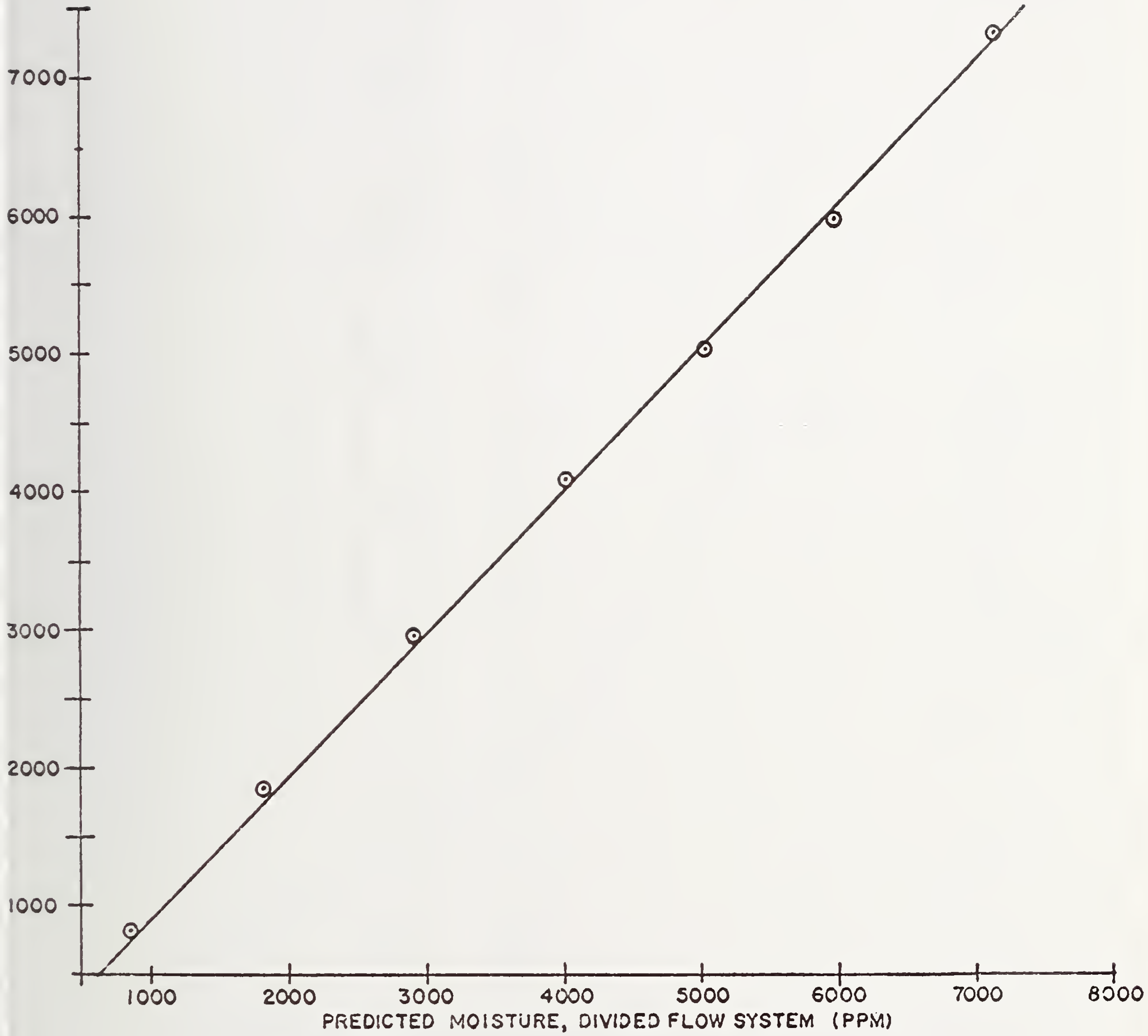

Figure 5. Moisture measurement comparison.

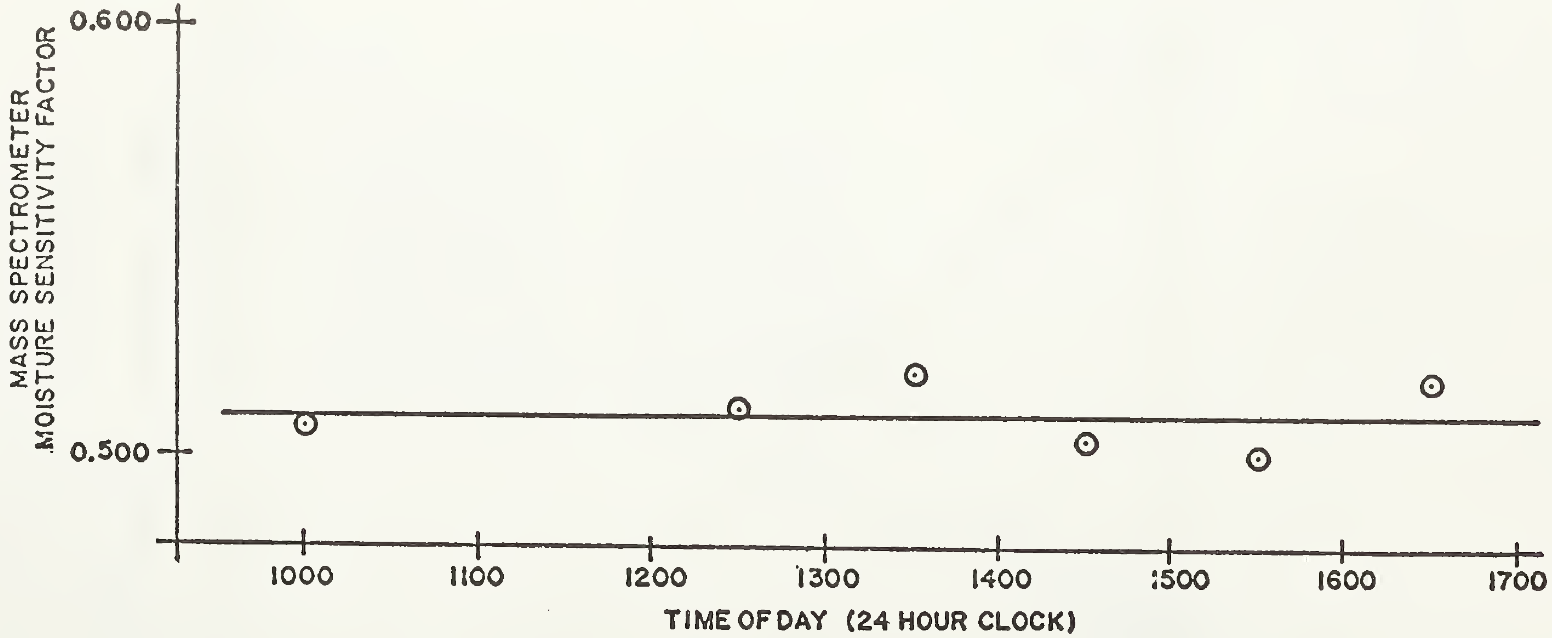

Figure 6. Short-term stability.

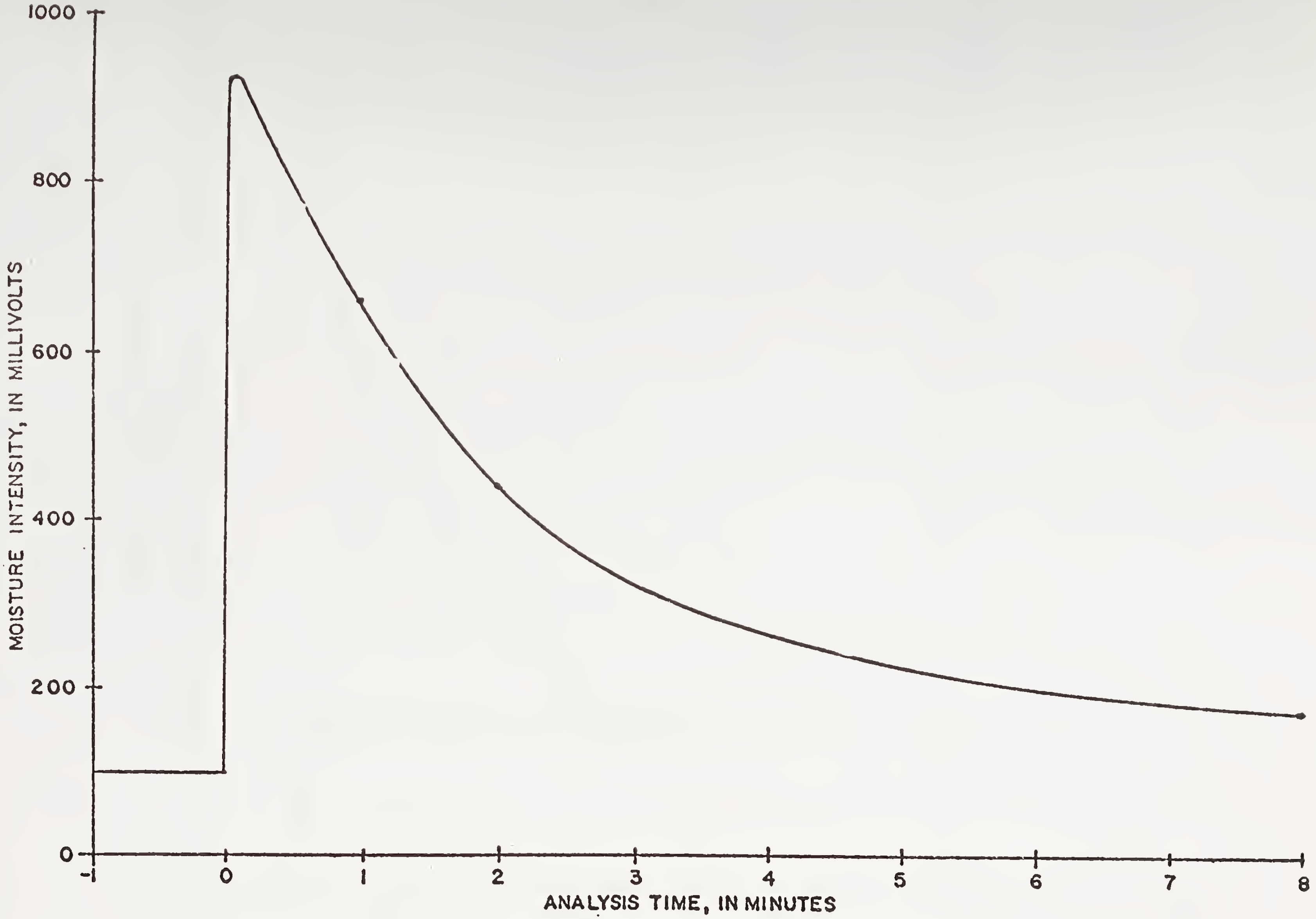

Figure 7. Air backfill.

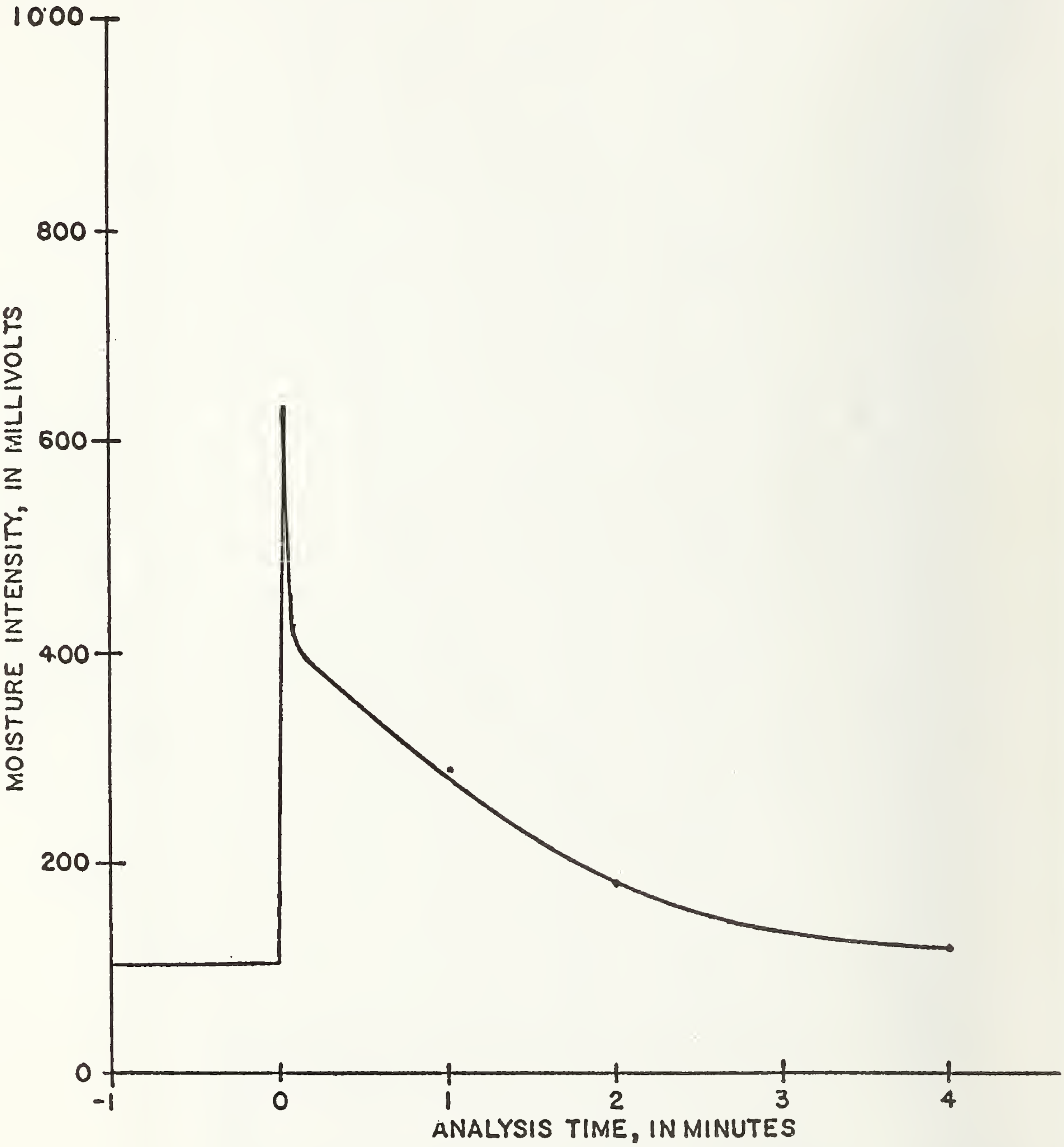

Figure 8. Plasma discharge.

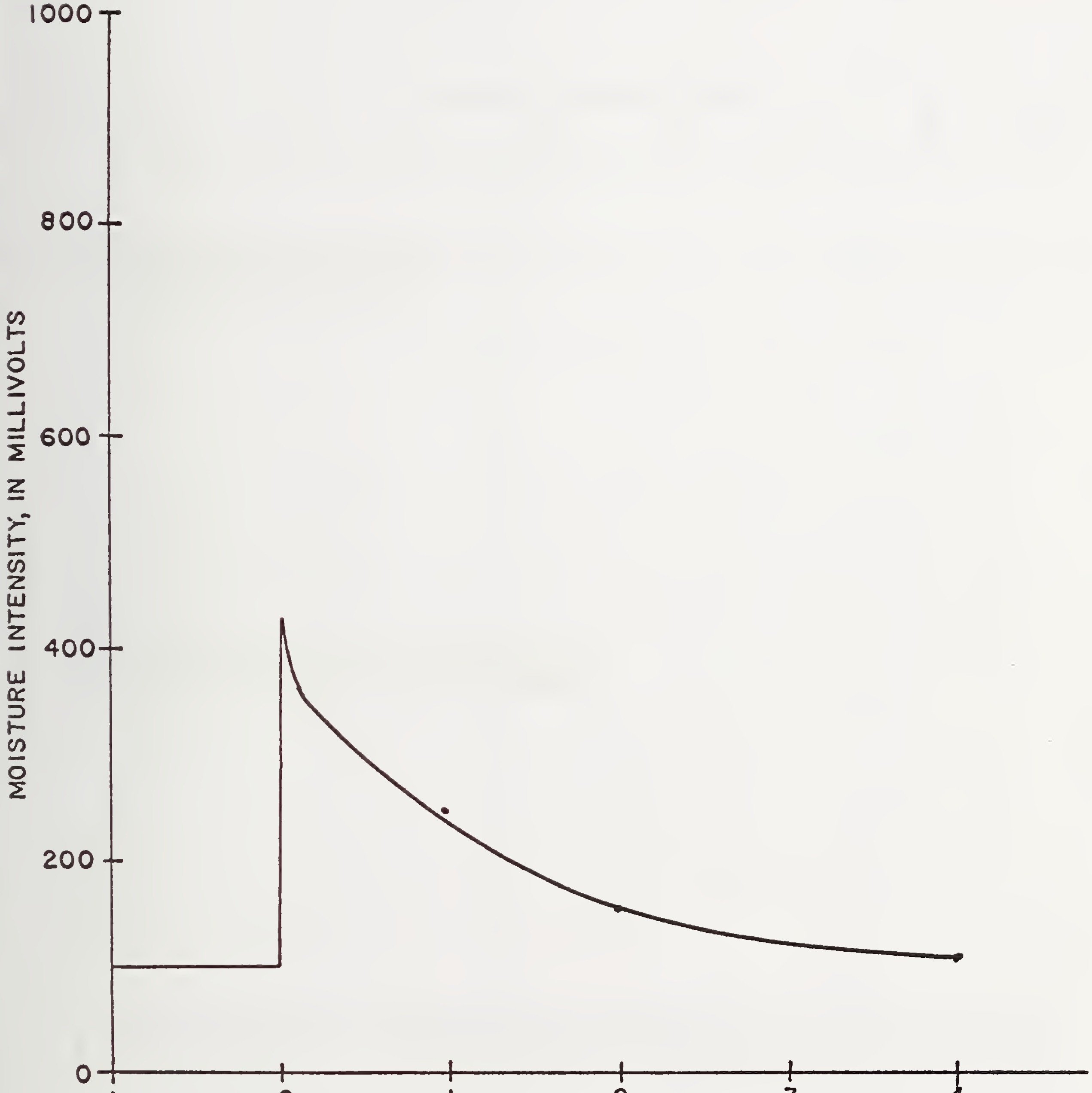

Figure 9. Argon backfill.

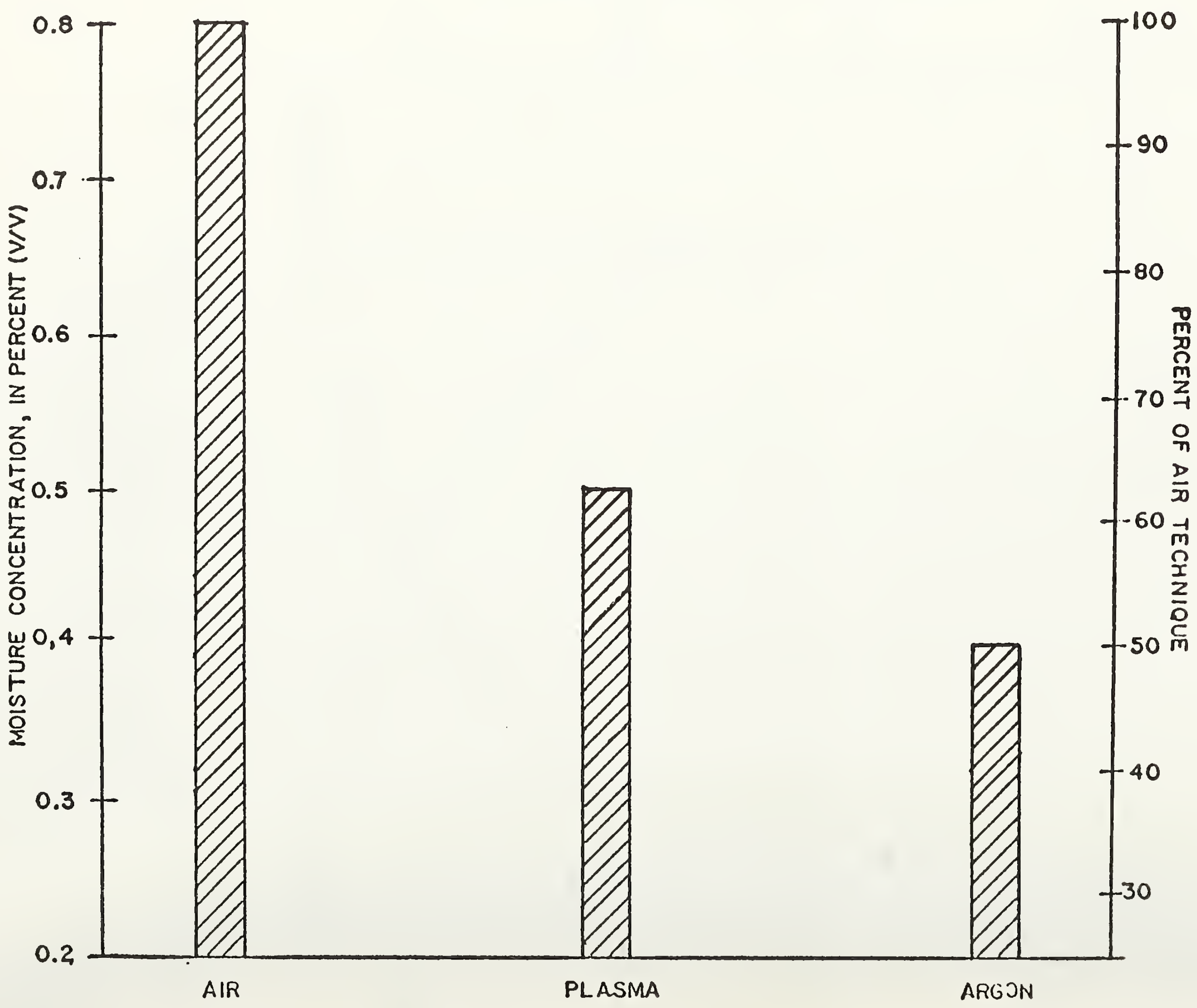

Figure 10. Preanalysis technique.

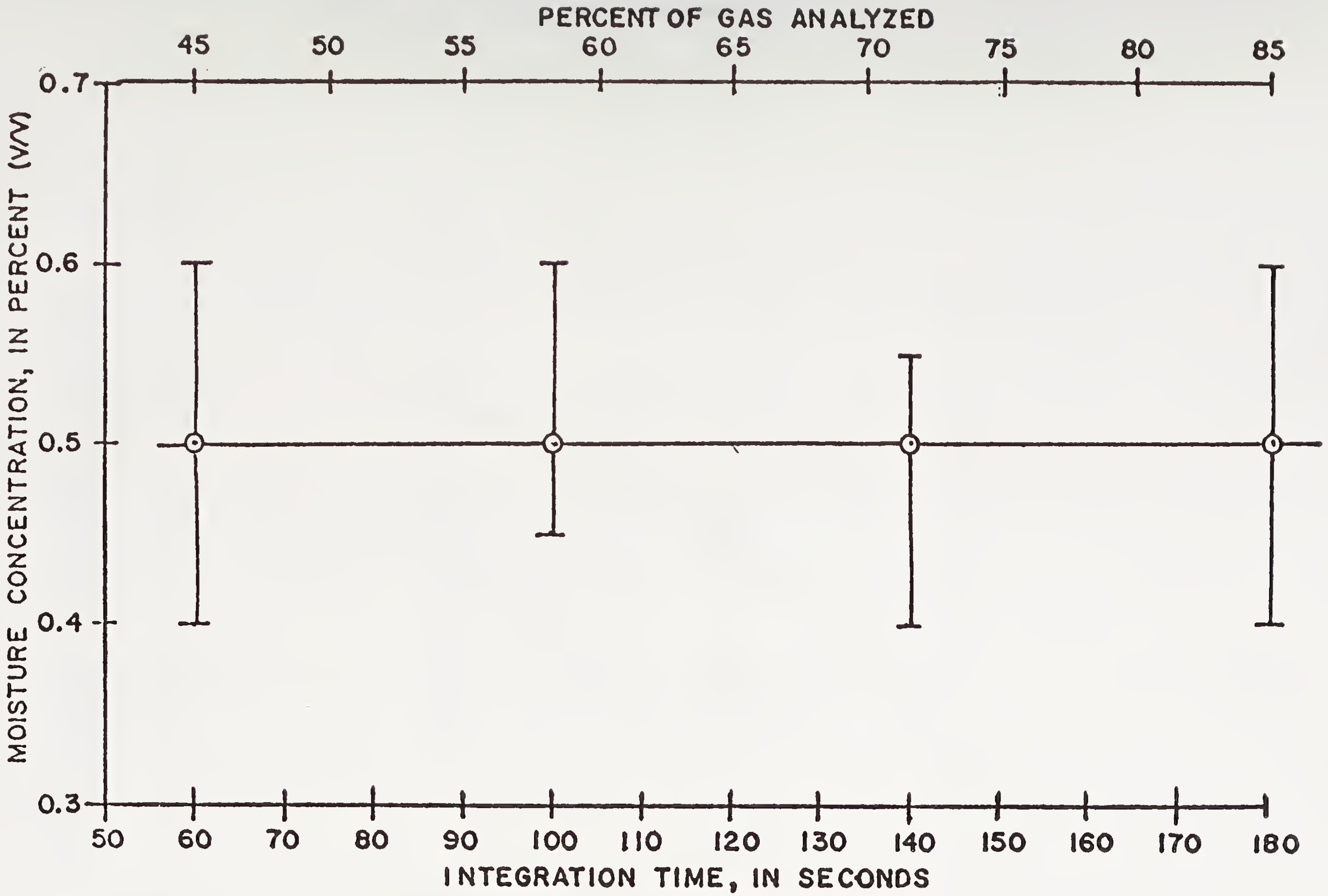

Figure 11. Influence of integration time.

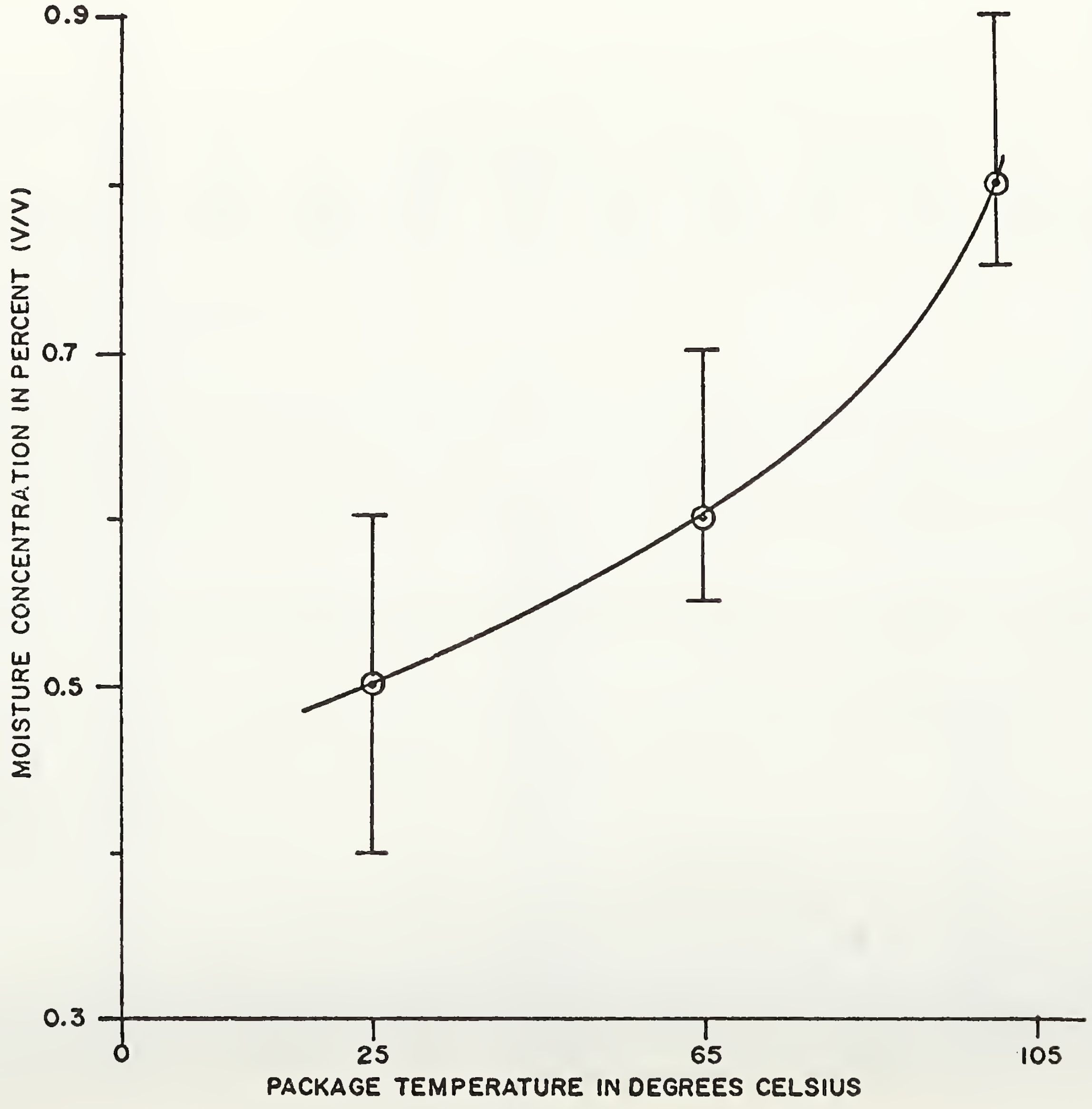

Figure 12. Package temperature experiment.

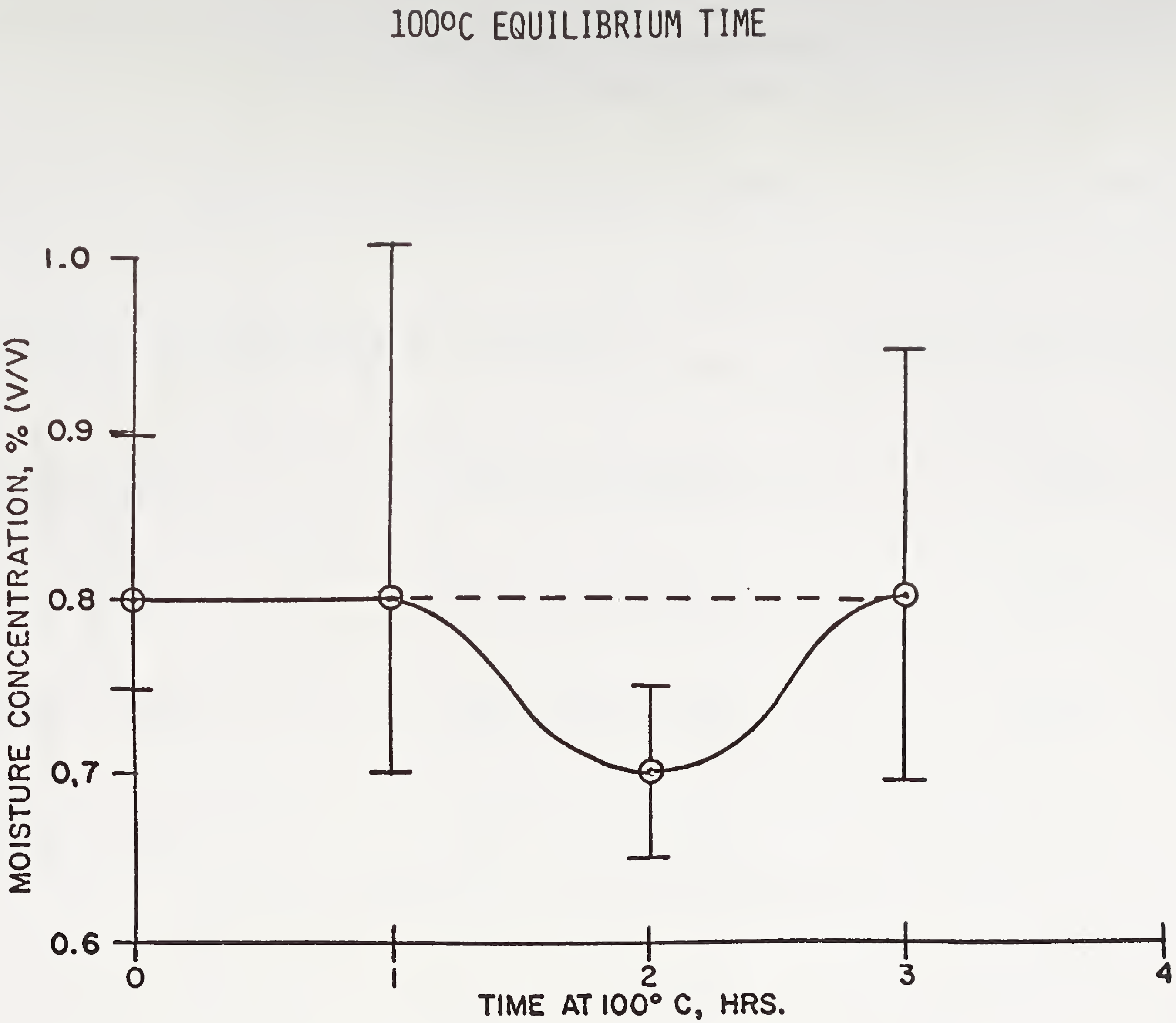

Figure 13. 100°C equilibrium time.

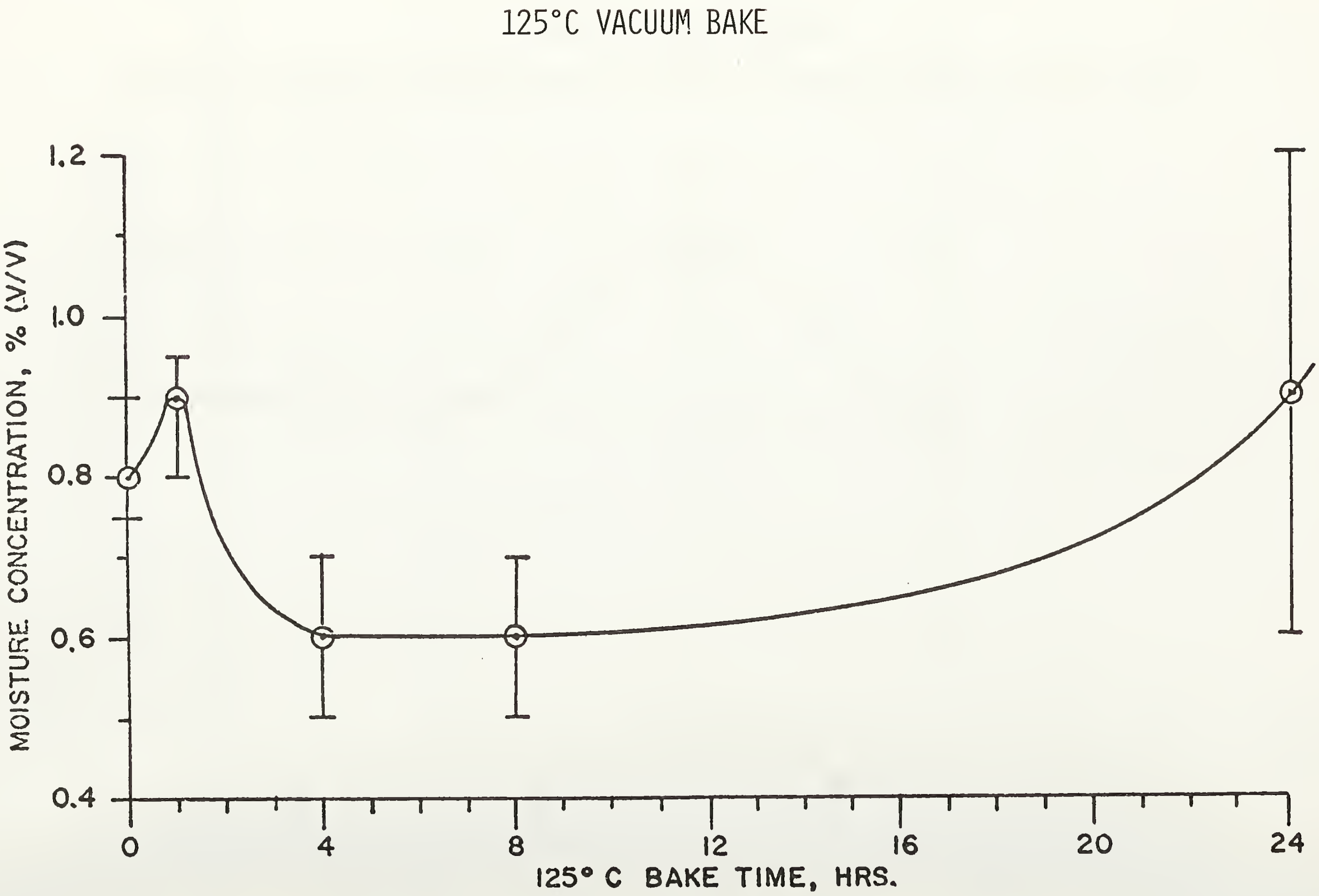

Figure 14. 125°C vacuum bake.

RAPID CYCLE SYSTEM MOISTURE RESPONSE

GAS SOURCE & MOISTURE (PPM_V)	PROCESS	REPORTED MOISTURE (PPM_V)
NITROGEN - 500	INERT GAS BACKFILL	500
ROOM AIR - 9000	INERT GAS BACKFILL	2000
NITROGEN - 500	ROOM AIR BACKFILL	3000
ROOM AIR - 9000	ROOM AIR BACKFILL	9000

Figure 15. Rapid Cycle system moisture response.

2.2 A Method for Measuring the PPM Moisture-Sensing Limitations of Mass Spectrometers in Testing Small Packages

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SUMMARY

Using a known ppm moisture generator, the water-vapor-measurement limitations of two mass spectrometers have been measured. It has been shown that the lower detectability limit for moisture in a gas is a complex function of the variables of each system employed. These variables include package and inlet volumes, inlet and ion source temperatures, type of high-vacuum pumping, and length of pumpout between analyses and/or calibrations. The influence of these variables has been measured to establish instrument characteristics at ambient temperature and at 110°C.

INTRODUCTION

A series of extensive tests was made to characterize the performance of two mass spectrometers for measurement of water content in small, hermetically sealed packages. Water measurement studies were made using an IC package simulator in which package free volume, pressure, and moisture content were known. Problems associated with actual package opening and testing are yet to be studied.

Primary interest in this study was to provide a final method of mass spectrometer water calibration which is compatible with the testing of packages sized from 2 to 0.07 cm^3. The calibration is intended to provide a method of determining either the moisture vapor content of a package or the moisture vapor plus the package wall desorbed moisture.

Description

Figures 1 and 2 are schematic diagrams of the two mass spectrometers used in this evaluation. Shown in figure 1 is a typical oil diffusion pumped, liquid nitrogen trapped, permanent magnet residual gas analyzer. Temperature of the analyzer is regulated at 225°C while the batch inlet system is at ambient. A fixed conductance of 0.072 cm^3 per second is provided between the inlet system and analyzer.

Most of the time spent in water measurement evaluation has been in characterizing the system in figure 2. Turbo-molecular pumping is used without cold trapping to attain an analyzer pressure of 10^{-9} Torr. Gas is transferred from the batch inlet system into the analyzer through variable conductance adjusted to provide an initial pressure rise in the analyzer of about 5×10^{-6} Torr. The inlet sample chamber and quadrupole mass filter are regulated at 110°C during package testing.

A six-port package simulation valve is attached to both systems shown in figures 1 and 2. This valve connected to the known ppm moisture source is shown in figure 3. "Carrier" gas is made to flow through the generator, as shown in figure 3, either through humidifier H1 or H2 where a known water vapor is added to the flowing gas. Humidified gas is vented after passing through the sample loop attached to the six-port valve. Inherent in the sample loop is the simulated package volume, pressure, and gas mixture. Further description of the package simulator is given in paper 5.1 of this workshop.

Experimental Results

Experimentally derived characteristic curves for water calibration are given in Graph A for the diffusion-pumped, fixed-conductance mass spectrometer. For simulated package sizes of 2, 1, and 0.5 cm^3, the moisture-sensing limitations are 1000, 1700, and 2500 ppm, respectively, assuming 1-atm initial pressure.

Characteristic curves for water calibration are given in Graph B for the turbo-molecular pumped quadrupole mass spectrometer having a variable conductance. For simulated package sizes of 0.5, 0.11, and 0.07 cm^3, the moisture-sensing limitation is extended to 250 ppm. As the package size is reduced, larger uncertainties are seen in the ppm moisture measurements.

If mass spectrometers are to be used as moisture detectors, it is essential that instrument precondition be used and pump-out time be carefully controlled. Given in table 1 are test results of further measurements made to determine the influence on inlet sample chamber pump-out time upon NET ion peak integral. Tests were made at two levels of moisture in nitrogen: 500 and 1000 ppm. No significant difference was measured in NET integrals after 5 and 10 min of pump-out time. However, after 20 min of continuous turbomolecular pumping, a 30-percent reduction in NET integral was measured.

As expected, the water vapor sensitivity of the quadrupole mass spectrometer increased when the sample chamber and analyzer were elevated from ambient to 110°C. The NET ion signal at 110°C was two to four times larger than the NET measurement at ambient temperature. Results are given in table 2.

CONCLUSION

Moisture measurement characteristics of two mass spectrometers have been established for use in testing hermetically sealed packages.

The smallest measurable ppm moisture content in sealed packages was determined using a package simulator.

ACKNOWLEDGMENT

The author is indebted to Dr. F. B. Burns for his assistance in data acquisition and data interpretation, and for his preparation of tubing used for package siumlation. The project could not have been completed on schedule without his contributions.

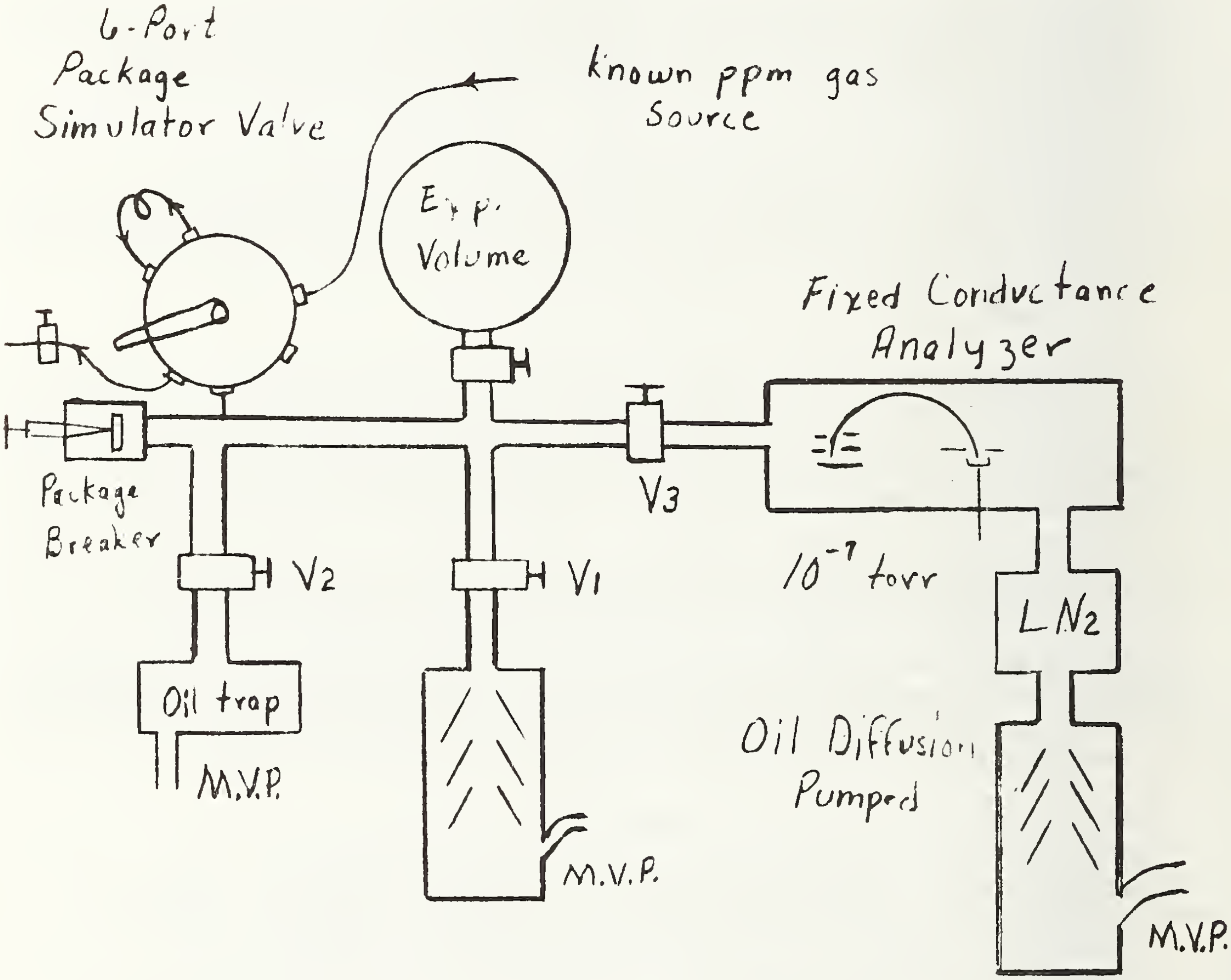


Figure 1. Constant conductance permanent magnet mass spectrometer.

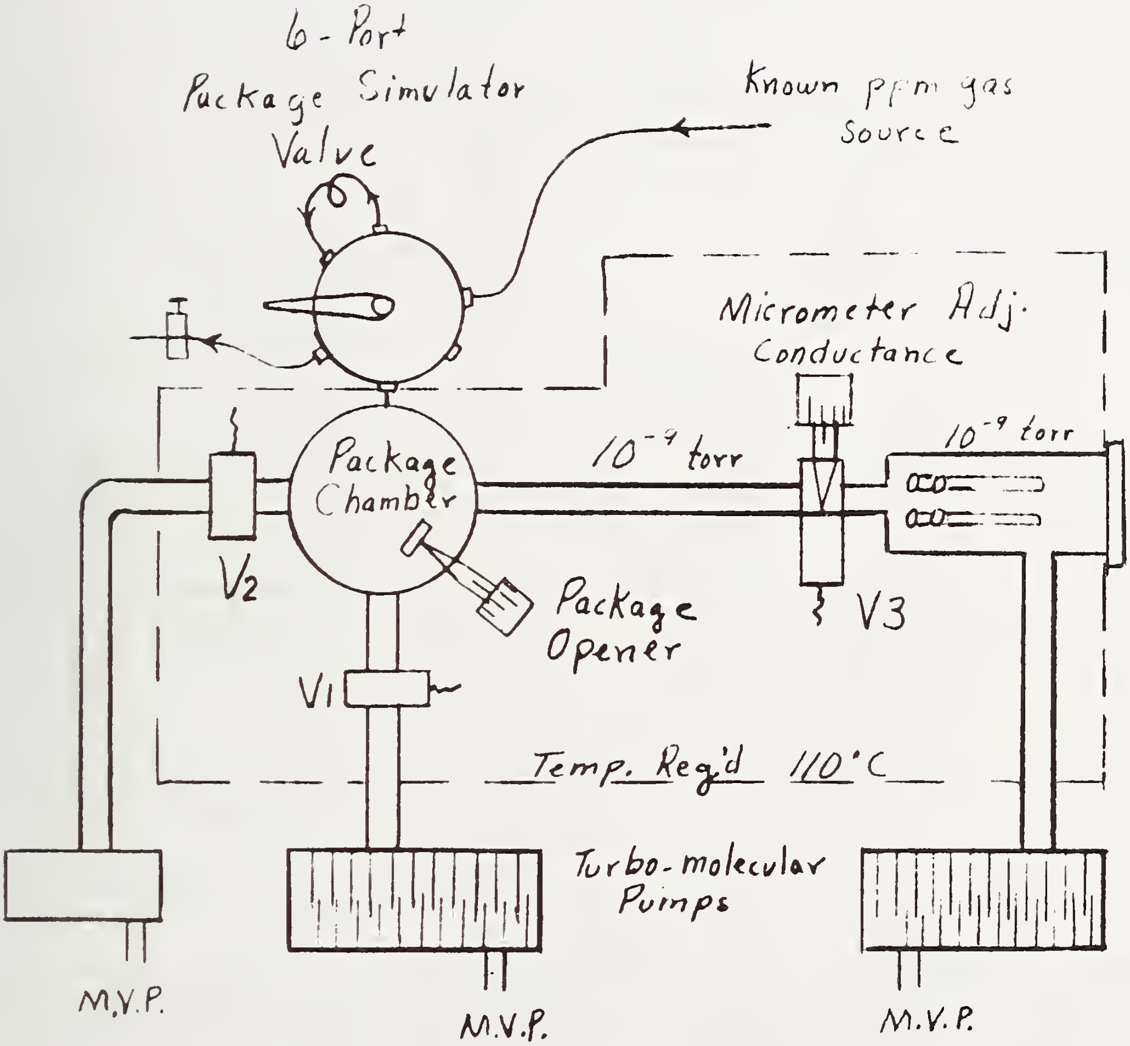


Figure 2. Variable conductance quadrupole mass spectrometer.

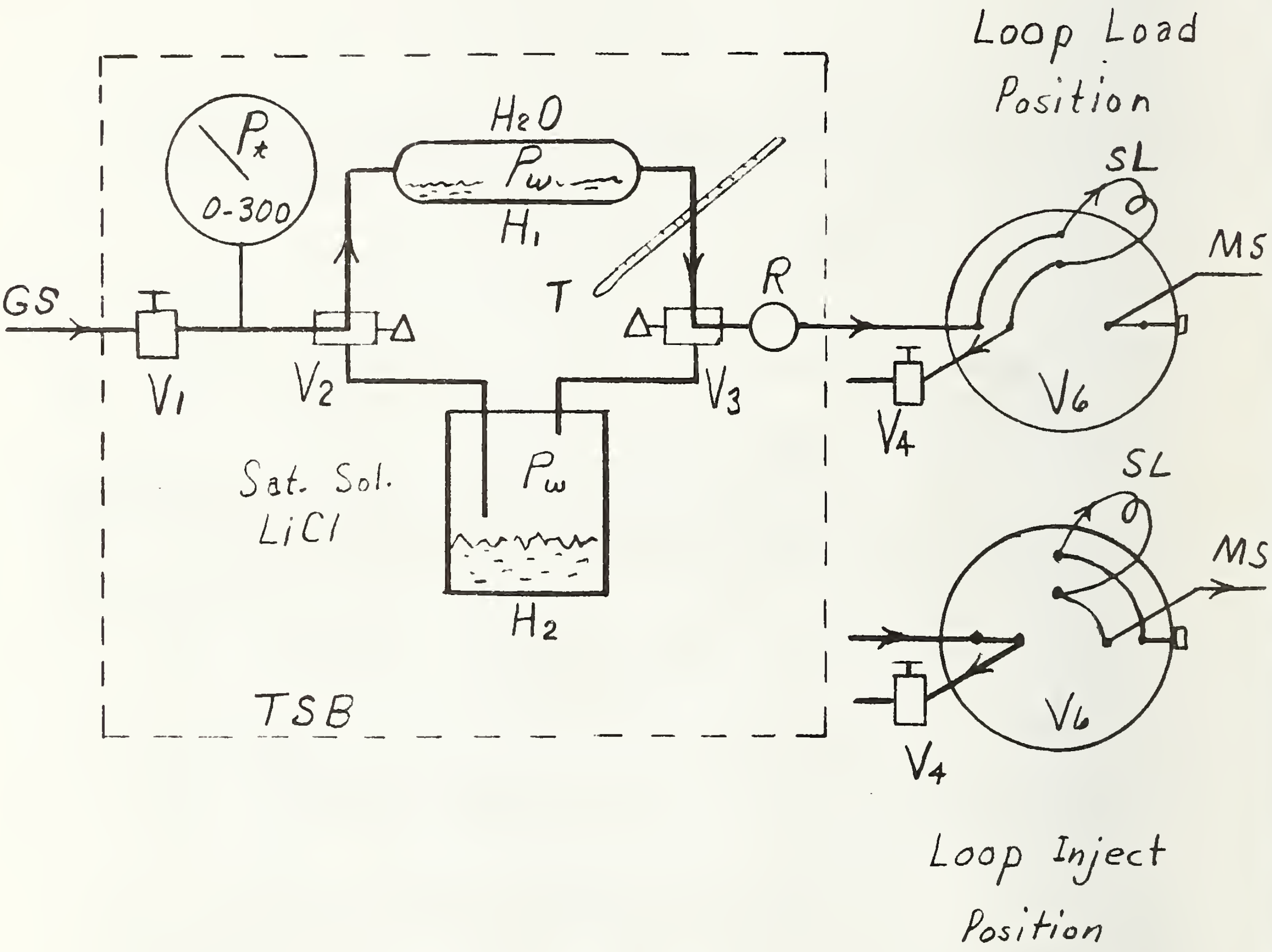
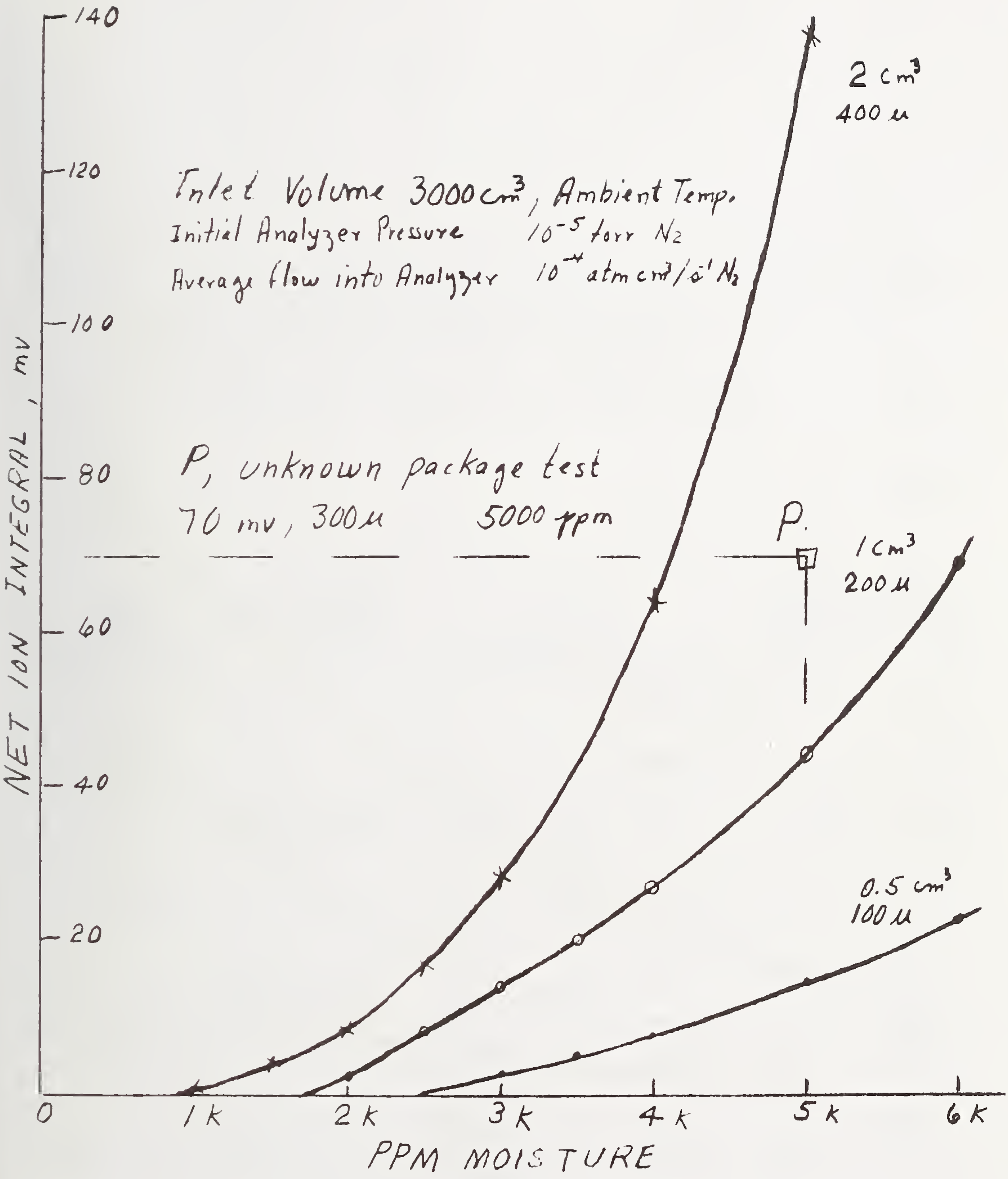
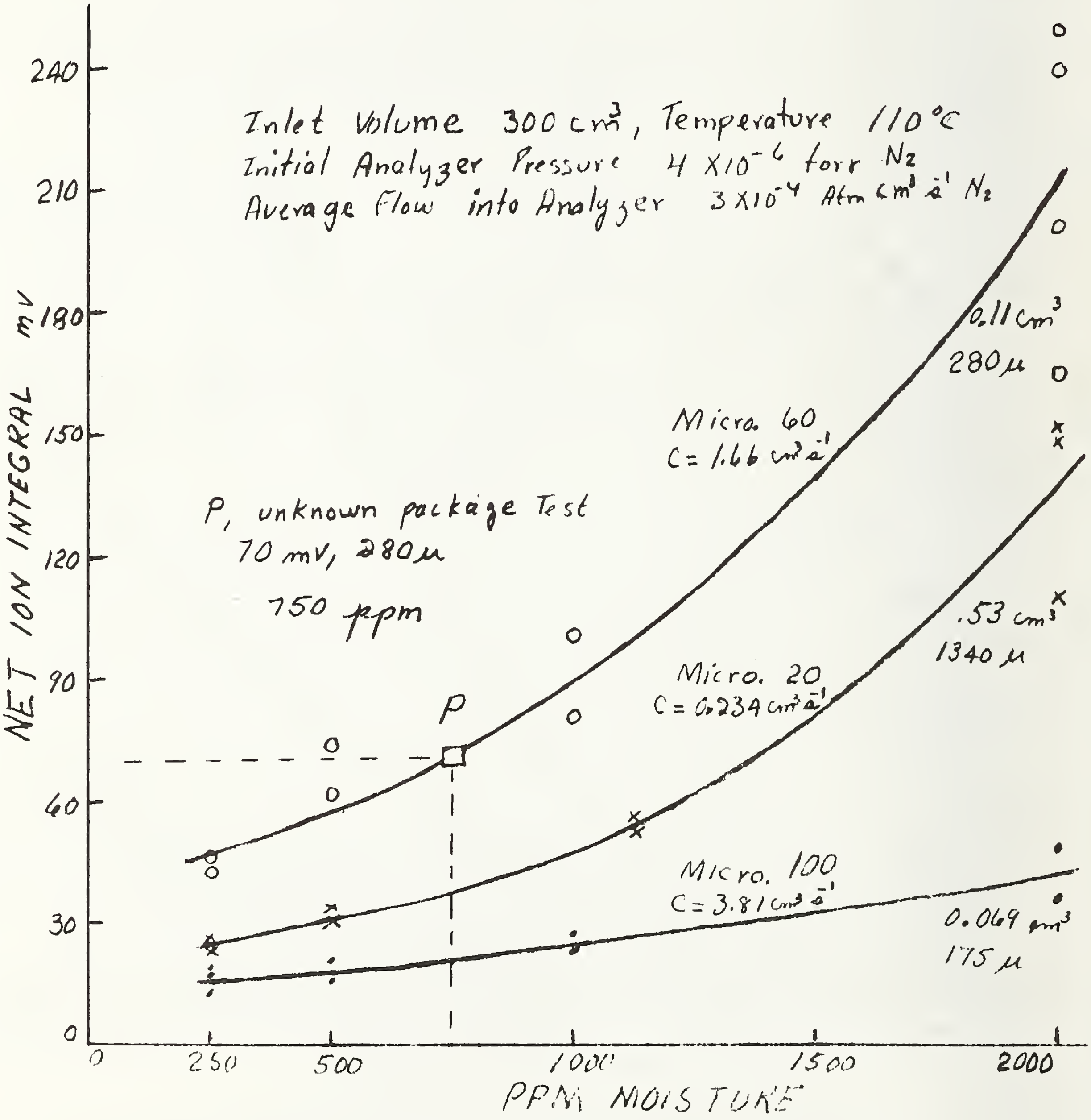


Figure 3. Hermetically sealed package simulator. GS - dry gas source; V1, V2, and V3 - gas control valves; V4 - sample loop pressure adjust and vent valve; R - gas flow regulator; V6 - six-port loop load and inject valve; SL - sample loop used to simulate package volumes; MS - mass spectrometer connection; P - total gas pressure (gage); P_w - water vapor pressure; T - humidifier temperature; TSB - temperature stabilizing box; H1 - liquid water humidifier (1 L); H2 - lithium chloride saturated water solution humidifier (3 L).



Graph A. Constant conductance mass spectrometer water calibration.



Graph B. Variable conductance mass spectrometer water calibration.

Table 1. Change in Mass Spectrometer ppm Sensitivity Due to 5-, 10-, and 20-Min Vacuum Pumping Between Observations.

Simulated package volume 0.5 cm^3

Temperature 110°C Humidified N_2 gas

ppm Water (By Volume)	Pumpout Time (Min.)	NET Integral (Millivolts)	Sensitivity (Millivolts per Millitorr)
500	5	2.46	2.55
	10	2.49	2.58
	20	1.77	1.83
1000	5	5.24	1.36
	10	5.78	1.50
	20	3.66	0.95

Table 2. Measure NET Ion Peak Integrals at Ambient and 110°C Temperature.

ppm Water (By Volume)	NET Ion Peak Integrals	
	Ambient Temperature	110°C Temperature
250	225	423
500	259	440
1000	329	980
2000	568	2230

2.3 A Dynamic Method of Calibrating a Mass Spectrometer Used For Measuring the Water Content of Semiconductor Encapsulations

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INTRODUCTION

The principal problem concerning the accurate measurement of the moisture content of small packages using a mass spectrometer arises from adsorption by the internal walls of the instrument. The problem can be overcome in part by the use of a small volume system maintained at an elevated temperature. Calibration of the system is, however, essential so a dynamic technique has been developed to characterize the instrument and hence enable accurate and reproducible measurements of the water content.

The important parameters of the instrument are the volume, V, of the expansion chamber into which the gaseous contents of the package is released, the conductance, U, of the leak connecting this chamber to the mass spectrometer, and the conductance, U_p, controlling the pressure in the latter. The volume of the chamber and the size of the leak are such as to ensure that there is molecular flow throughout the system. If the specimen of gas contains an amount, Q, of an inert species, the partial pressure, P, of that species in the spectrometer will be described by the equation:

$$P = \frac{Q}{\tau_d U_p} \cdot \exp(-t/\tau_d) , \tag{1}$$

where τ_d, the depletion time, is equal to V/U. As there is molecular flow, U depends on the molecular weight of the species.

Each time a water molecule impinges on an uncovered surface, it is physically adsorbed for a short time and then desorbed. Consequently, when water is released into the expansion chamber, equilibrium between the amount of water adsorbed and that in the gas phase is reached extremely rapidly if physical adsorption is the only process removing water from the gas phase. For most systems the time taken to reach equilibrium will be much less than the depletion time so the two phases will remain in equilibrium as the pressure falls in response to the loss of water to the spectrometer. When the specimen contains a small quantity of water, the amount adsorbed will be proportional to the pressure, and the equation describing the decay in pressure is the same as that for an inert gas. The only difference is that the depletion time becomes (1 + n) V/U, where n is the quotient of the amount of water adsorbed to that in the gas phase. It follows from eq (1) that the rise in the partial pressure also depends on the value of n.

Figure 1 shows how the partial pressures of nitrogen and water decayed after the puncturing of a package. The ratio of the depletion time for nitrogen to that for the other inert gases in the package confirmed that there was molecular flow so the value of V/U for water could be deduced from the depletion time of nitrogen. Thus, it was possible to predict the rate of decay of the pressure of water in the absence of adsorption ($n = 0$). In this ideal case, the plot of pressure against time should be parallel to the broken line on figure 1. As the measured decay rate was half that indicated by this line, the value of n must have been 1. So half of the water released into the chamber was adsorbed.

These calculations assume that the adsorption in the spectrometer has a negligible effect compared with that of adsorption in the expansion chamber, and that the pressure in the former is always proportional to that in the latter. This will be true if the two parts of the instrument are at about the same temperature and if $U_p > U$, conditions will usually be met. Adsorption in the spectrometer just delays the response of the pressure in this part of the system to changes of pressure in the expansion chamber (as demonstrated by fig. 1).

Calculation of Water Content

The simplest method of calculating the water content of a specimen is to compare the rise in the partial pressure of this gas with that observed when a known quantity of an inert gas is released into the system. As there is molecular flow throughout the system the equation to be used is:

$$\frac{\text{(amount of water)}}{\text{(amount of inert gas)}} = \frac{(1 + n)\ \text{(rise in pressure of water)}}{\text{(rise in pressure of inert gas)}} . \qquad (2)$$

The numerator of the RHS of this equation is the pressure which would have been observed in the absence of adsorption. A simple technique has been developed to determine the value of the unknown $(1 + n)$. It is based on an analysis of the time dependence of the rise in pressure of water when this vapour is leaked into the expansion chamber at a constant rate.

Assessment of the Amount of Water Adsorbed by the Expansion Chamber

Nitrogen was bubbled through water and passed over the entrance to a needle valve connected directly to the expansion chamber which was evacuated by a diffusion pump. When the chamber was subsequently isolated from the pump, the partial pressure of water in the chamber, and hence in the spectrometer, slowly rose towards a limiting value. If the rate of entry of water into the expansion chamber is q, then the amount which will have entered within a time, t, since starting the flow will be $q \cdot t$. The amount extracted from the chamber by the spectrometer during this period will be $U \cdot \int_0^t P_e \cdot dt$, where P_e is the partial pressure in the chamber. As the amount of water in the gas phase is $V \cdot P_e$ it follows that n, the quotient of the amount adsorbed and that in the gas phase will be:

$$n = (q \cdot t - U \cdot \int_0^t P_e \cdot dt - V \cdot P_e)/V \cdot P_e . \qquad (3)$$

The partial pressure in the spectrometer is related to P_e by the equation:

$$P \cdot U_p = P_e \cdot U . \qquad (4)$$

Equilibrium is eventually reached when the flow of water through each part of the system is constant. The pressure in the spectrometer rises to the limiting value, P_s, which is given by the equation:

$$P_s = q/U_p \; . \qquad (5)$$

Equations (3), (4), and (5) can be combined to give:

$$n = (P_s \cdot t - \int_0^t P \cdot dt - P \cdot V/U) \cdot U/P \cdot V \; . \qquad (6)$$

As the value of V/U can be deduced from the depletion time of nitrogen, all the quantities on the RHS of this equation can be measured. Figure 2 shows plots of the reciprocal of n plotted against the pressure in the spectrometer. They were obtained when the system was at 165 and 150°C. The linear relationship is predicted by a model based on physical adsorption. If the amount of water adsorbed is described by Langmuir's eq (1), the value of n will be:

$$n = \frac{A \cdot \gamma \cdot m \cdot \beta}{V \cdot (1 + \beta \cdot P)} \qquad (7)$$

where A is the surface area of the expansion chamber, the roughness of this surface, m is a constant, and β a parameter which depends on the heat of adsorption, ϕ, the temperature, T, and the oscillatory period, τ_o, of the adsorbed molecule. If the pressure is measured in mbars, volume in 1, and area in m^2, then eq (7) becomes:

$$\frac{1}{n} = P \cdot \left(\frac{1.37 \times 10^3 \cdot V \cdot U_p}{A \cdot \gamma \cdot T \cdot U} \right) + \left(\frac{1.17 \times 10^{-4} \cdot V)}{A \cdot \gamma \cdot \tau_o \cdot T^{1/2}} \right) \cdot \exp - \frac{\phi}{R \cdot T} \; . \qquad (8)$$

The values of V, A and U_p/U are known; the latter is determined by calibration of the instrument using a known quantity of an inert gas. Thus the slope of the plot of 1/n against pressure can be used to calculate γ; a value of 4 is needed to account for the results presented here. The intercept of the straight line can be used to calculate the heat of adsorption. The value of 14.3 kcal/gram mole needed for a reasonable fit to the results are at the high end of the range of values quoted for physical adsorption in the literature [2] but it is possible that the calculation is affected by the substantial temperature gradients in the oven enveloping the system.

Equation (8) is only valid if equilibrium between the amount of water adsorbed and that in the gas phase has been reached. In equating the values of n given by this equation to that given by eq (6), it has been assumed that the rise in pressure is slow enough for the two phases to remain in equilibrium. This is an assumption which can be checked by comparing the rate of decay of pressure, when the flow is stopped, with the value calculated using the value of n deduced during the period when the pressure was rising. It can be shown that if n is described by an equation of the same form as eq (8), then the rate of decay of pressure is a solution of:

$$- \frac{1}{P} \frac{dP}{dt} = \frac{U}{V \cdot (1 + n^2/n_o)} , \tag{9}$$

where n_o is the value of n when P is zero. All the factors on the right of this equation are known so the measured value of $\frac{1}{P} \frac{dP}{dt}$ can be compared with the calculated one. The measured values plotted in figure 3 are all normalised by multiplying by V/U. These values, which were obtained from the results of four experiments, are in reasonable agreement with the solid line which was calculated using the value of n given by figure 2. Therefore, the assumption that the two phases were always in equilibrium is shown to be valid. We have not shown that equilibrium is reached immediately after water is released into the expansion chamber (as it would be if there were only physical adsorption), but let us proceed as if we had.

Reappraisal of the Calculation of the Water Content of Packages

When a package is punctured, the water released would almost immediately be shared between the two phases and the rise in pressure is $(1 + n)^{-1}$ times lower than it would have been in the absence of adsorption. As the value of n is known, it can be used to convert the measured pressure rise into that which would have been observed in the absence of adsorption and hence the water content can be calculated. Figure 4 shows the relationship between the measured and the calculated pressures. It was obtained using the value of 1/n plotted in figure 2. The accuracy of this calibration curve can be checked by releasing a known quantity of water into the expansion chamber. The point on figure 4 was obtained by puncturing a TO-5 package which had been sealed in an atmosphere having a dew point of 12°C. The agreement indicates that the model is substantially correct. The self-consistency of the analysis can also be checked by comparing the rate of decay of pressure after puncturing a package with that predicted by the equations.

When small quantities of water were released into the instrument (at 165°C), the time constant controlling the decay in pressure was found to vary with the range 2 to 3.8 times V/U, whereas the value of n obtained from figure 2 predicts a time constant of 3.5 V/U. The discrepancy can be shown to be due to the fact that the amount of water adsorbed depends on the previous history of the expansion chamber.

None of the measurements of the amount of water adsorbed, which have been described so far, had been made after a prolonged period of outgassing. The first measurement of the pressure dependence of n after such a period always gave lower values of n than those found subsequently, so it would appear that the expansion chamber was capable of adsorbing more water than it did during subsequent exposure to moisture. The increased adsorption may be due to the transfer of water to a tightly held state – a mechanism which may be akin to the chemisorption studied by Hayashi [3]. As only the first measurement after outgassing is substantially affected, it would appear that the amount of water which can be held by this state is limited and that the rate of desorption is low. If the flow of water into a previously outgassed expansion chamber is stopped after only a short time, the subsequent rate of decay of pressure is far more rapid than that found when all the sites responsible for this second type of adsorption were full. This rapid decrease in pressure can be explained by postulating that the transfer of water to the tightly held state continues when the pressure begins to fall. As this behaviour indicates that the adsorbed and the gas phases were not in equilibrium, the rate of transfer to the tightly held state must have been small. Thus, immediately after water is released from a punctured package, we need only consider the amount of water physically adsorbed. The transfer to the tightly held state will, however, cause the pressure to fall more rapidly than expected. As the rate of transfer depends on the amount of water already chemisorbed, the variability of the time constant describing the rate of decay of pressure after puncturing a package can be explained.

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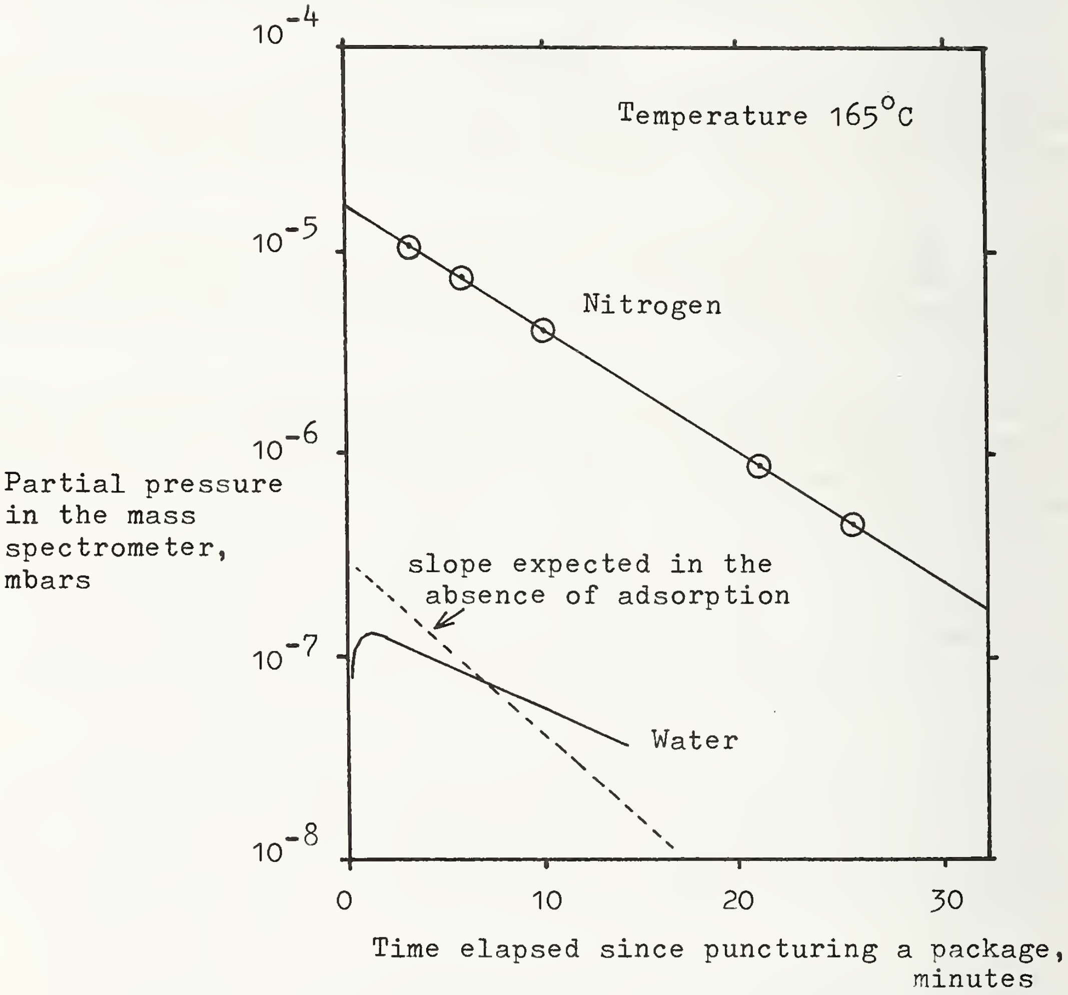

Figure 1. Decay of pressure after puncturing a package.

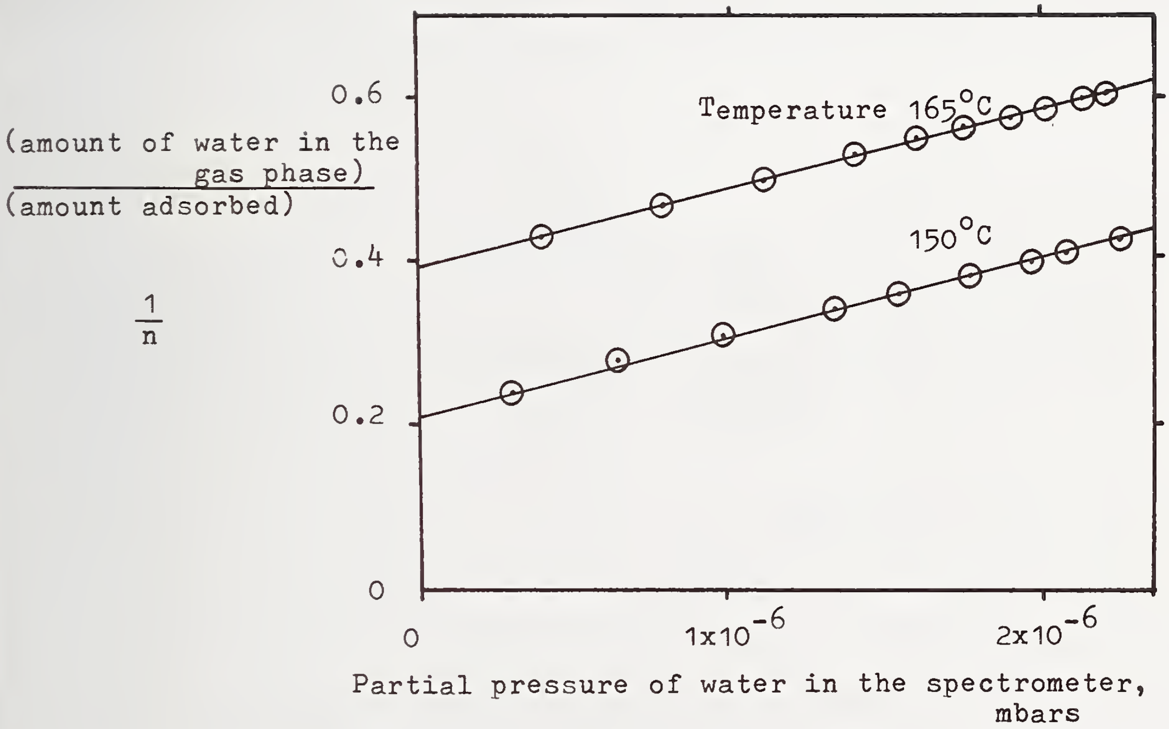

Figure 2. Pressure dependence of the reciprocal of the amount of water adsorbed.

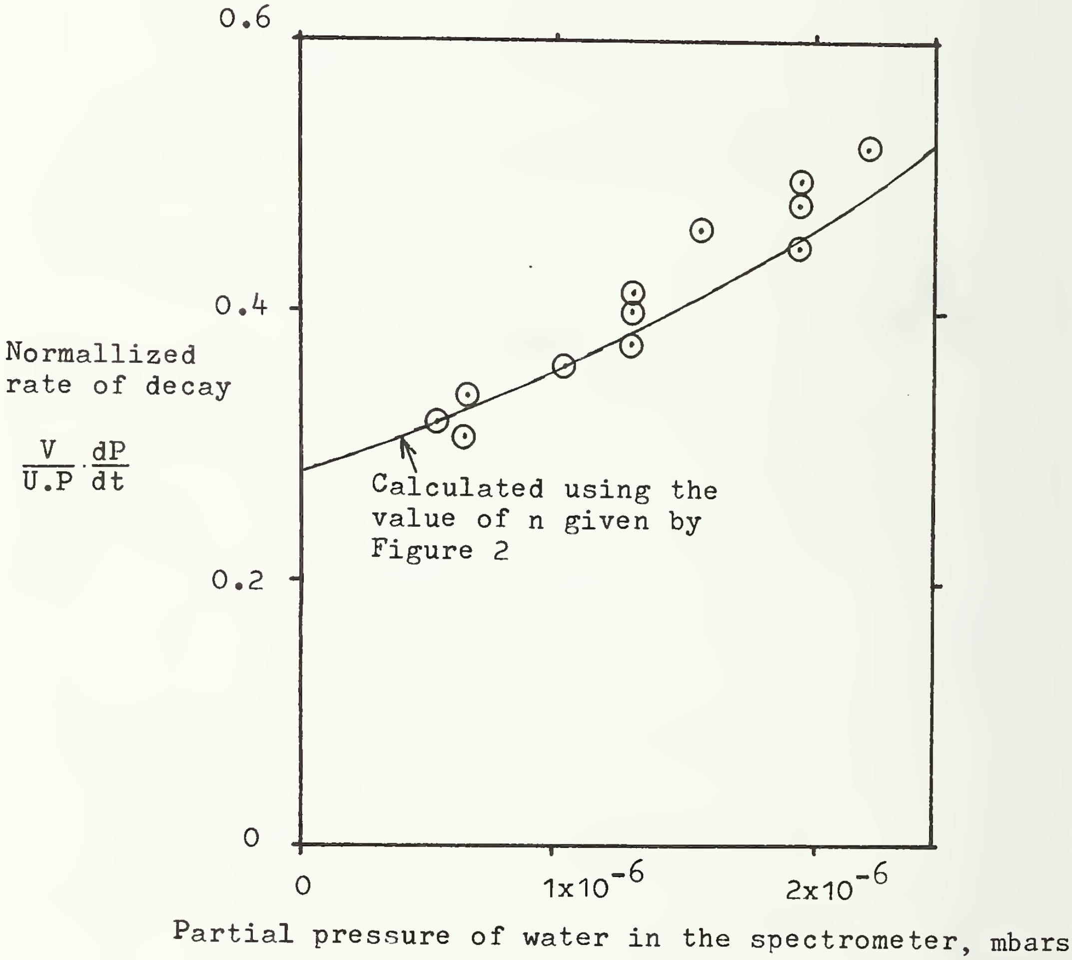

Figure 3. Pressure dependence of the rate of decay of the partial pressure of water in the spectrometer.

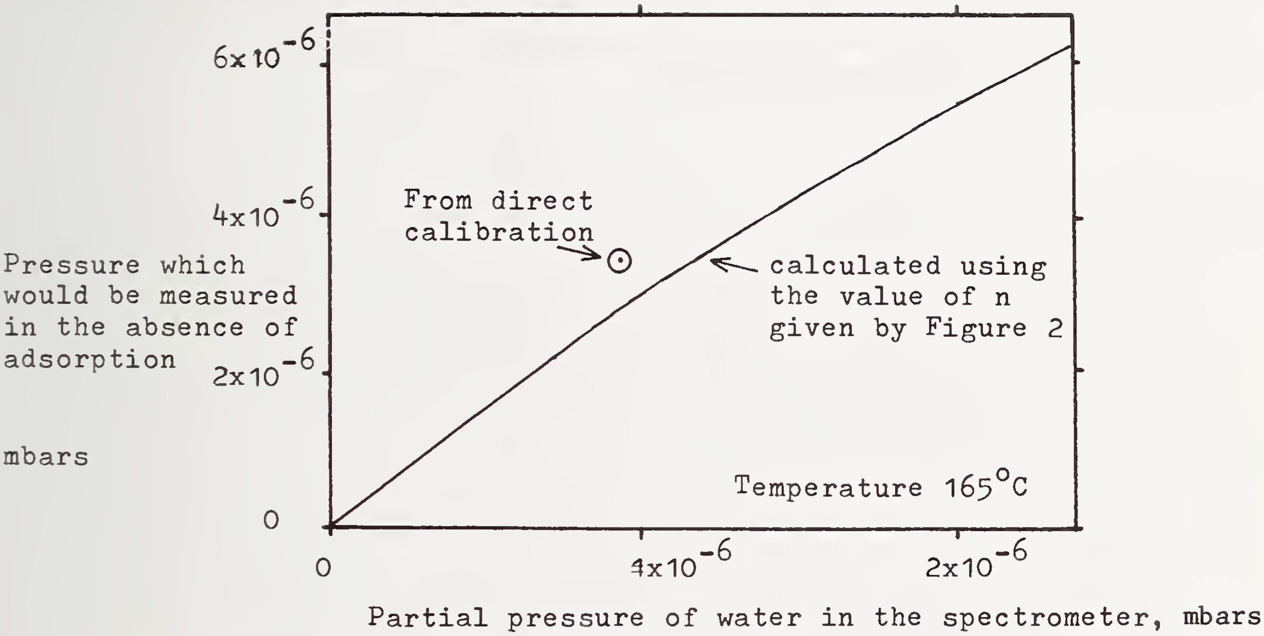

Figure 4. Calibration curve.

2.4 Moisture Measurement by Mass Spectrometer

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SUMMARY

Accuracy and precision in any measurement can only be achieved by a thorough understanding of the dependent and independent variables associated with the measurement problem. This paper addressed the identification of variables and their impact on: 1) obtaining a valid sample, 2) transferring the sample to the analyzer, 3) analyzing the sample, and 4) calibrating the analyzer. A test method was discussed which provides a high degree of precision, sensitivity, and accuracy. Examples of instrumentation capable of reliably implementing the above test method were described. A summary of test data over a one-year period was presented.

2.5 Microcircuit Package Gas Analysis*†§

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Quality control [1] and failure analysis of hermetically sealed integrated circuit packages has become increasingly important in establishing the criteria which should be satisfied for reliability and long-term performance of the IC package. The failure to preclude solvent, moisture, and other contaminants can lead to migrated-gold resistive shorts [2].

Failure analysis as outlined below furnishes a wealth of information for determining the nature of the problem and what factors need to be considered for improvement in reliability.

A. Nondestructive Testing
   1. external visual
   2. electrical characterization
   3. radiographic (x-rays)
   4. hermeticity test

B. Destructive Testing
   1. package gas analysis
   2. internal visual
   3. internal electrical characterization
   4. SEM analysis

C. Report
   1. part history
   2. analysis results
   3. conclusions
   4. recommended action

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* This paper represents the results of one phase of research carried out at the Jet Propulsion Laboratory, California Institute of Technology, under Contract No. NAS7-100, sponsored by the National Aeronautics and Space Administration.

† The views and conclusions expressed are those of the authors and do not necessarily represent the official policies of the Department of Defense, Department of Commerce, or the United States Government.

§ Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

Mass spectrometry is an effective method for package gas analysis but problems associated with moisture determinations [3,4] require that the method be carefully executed and that reliable calibrations are available.

The microcircuit analysis accessory device (MAAD)[5] used at JPL is a multisample inlet system (fig. 1) with a high vacuum environment for the preanalysis bakeout, a package puncturing fixture, and a gas transfer line to the mass spectrometer. The MAAD consists of a heated chamber (fig. 2) containing a carousel upon which a maximum of twelve packages may be mounted. By varying the carousel base plate design, ceramic packages, cans, or flatpaks can be accommodated. The carousel may be detented to a specific package location and then the package is punctured with a manual micrometer-driven puncture device. All fabricated materials, pneumatically operated valves, rotary advance, and the puncture mechanism, are of stainless steel. Gaskets are OFHC copper. The complete system is maintained at 100°C during the analysis sequence.

The MAAD, with its own vacuum system, capacitance nanometer, and inlet to the mass spectrometer, enables one to subject the samples to a bake-out sequence and then to analyze in succession the contents of each package. A data system provides the necessary versatility and speed for such an analysis.

PROCEDURE

Chamber Loading

The IC packages are carefully rinsed with acetone and methanol, and then mounted (avoid lead stress) on the MAAD carousel. Standard cleanliness procedures are observed during the handling of any parts of the sample environment. Upon reassembly of the chamber, the system is purged with dry nitrogen for 15 min before high vacuum initiation and then undergoes a bake-out period of ∿16 hours at 125°C with the ion pump operating between 10^{-6} and 10^{-7} Torr. For analysis, the chamber, inlet line, and mass spectrometer manifold are operated at 100°C.

System Verification

The Finnigan Model 3200 is calibrated for the range 18 to 800 amu in the conventional manner using FC-43 ($C_{12}F_{27}N$). For quantitative analysis in the mass range 2 to 50 amu, the Mass Fragmentography Mode is capable of monitoring 4 different m/e values. In order to achieve maximum sensitivity and accuracy, the Diagnostic Mode (fig. 3) is used to determine the proper m/e value to the nearest 0.1 amu. The background spectrum or that of a calibrating gas mixture (fig. 1) may be used to establish the mass defect.

At the conclusion of the bake-out period and during the 1-h equilibration period (125°C → 100°C), the system is conditioned and the response level and stability checked by inletting a 10 ppm DI water in nitrogen standard [6] (baseline reference) as shown in table 1. The amount of gas, the variable leak-rate setting, and the scanning parameters are determined from previous experience.

Package Analysis Sequence

Prior to analysis of the first package (table 1), the background spectrum of the instrument is compared to that obtained with the chamber open (valves V_2, V_4, and V_6 open, fig. 1) to the instrument. Immediately upon penetration of the package lid with the puncture device, the Tru-Torr gage indicates the pressure of the expanded gas volume in the chamber. Simultaneous observation of the gage readout and the puncture device torque prevents undue penetration of the package cavity and circuit damage. The available volume of the chamber varies from 110 to 140 cm^3 depending upon the size of the carousel jig used. At this point, the proper leak rate is selected and rapid qualitative scans of the gas sample are made with either the light beam oscillograph (mass range 2 to 90) or the data system (mass range 2 to 800) by opening the inlet valve to the mass spectrometer for $\sim$3 s (with proper leak rate, less than five percent of the total sample is used for this survey). From the rapid scan result, a selection is made as to which four m/e peaks are to be monitored by the data system for the quantitative analysis of the gas sample. Since nitrogen and water are usually monitored, there are only two available choices. The list of general interest includes hydrogen, helium, methane, argon, and carbon dioxide.

For a sample package which produces a chamber pressure of 100 millitorr, the mass flow rate is 0.55 Torr-ℓ/s and a total scan time of 4.3 min is used. For small package gas volumes, all of the gas is inletted to the mass spectrometer in order to account for transport characteristics for different species as shown in figure 4. In particular, relatively long sampling times are required for moisture. In order to analyze large gas volumes which would produce chamber pressures >1 Torr, a sampling volume (SV) is available between V_4 and V_2 (fig. 1). By allowing the sample gas to expand and equilibrate into this segment and then isolating it from the chamber via V_2, this sampling volume ($\sim$4.5 cm^3) can be analyzed in a reasonable length of time and in a similar fashion to smaller samples. Again, conditions for the sample and for the baseline reference are duplicated.

Following the analysis of the sample, the inlet valve is closed and the chamber opened to the ion pump for $\sim$5 min. The gate valve to the ion pump is then closed and a sample of the moisture standard (10 ppm water in nitrogen) is added to the chamber. The pressure and analysis conditions are identical to that of the previously analyzed package sample. Thus, one establishes the baseline for the moisture calculation of the sample, the sensitivity factor for nitrogen, and the background state of the MAAD chamber before puncturing the next sample. In the event of a very wet sample (>1-percent water), the sequence with the moisture standard may have to be repeated in order to determine the recovery of the system (see table 1). Area integration of the curves (fig. 4) generated by $\sim$500 scans is made with a background correction using the first and last 10 scans of the respective species being monitored. The 10-scan intervals represent the instrument background before and after closure of the sample inlet valve, V_2. For the moisture measurement (fig. 5), the total data intensity of scans 10 to 468 represent the moisture "area." Reproducibility of the results for repetitive backfilling of the chamber to the same pressure is shown by the integrated areas of the nitrogen curves (table 1). The behavior of the moisture background during the sequence is likewise indicated. These data printouts are used to ascertain the stability of the system.

Following each set of samples analyzed (daily), the moisture standard (1000 ppm water in nitrogen) is analyzed under conditions identical to that of the samples and the baseline standard (10 ppm water in nitrogen (fig. 5)). These standards are inletted to the MAAD chamber from the cylinders through heated stainless steel regulators, lines, and valves. Room air is used for calibration for higher levels of moisture.

Sensitivity Factors

Because the mass spectrometer has varying sensitivities for the different species being analyzed, it is required that these relationships be determined in order to complete quantitative measurements. The sensitivity factor determination for water is most important and also exhibits the most problems. Ideally, it would be desired to puncture a package similar to the sample in size, gas composition, pressure, and having a predetermined amount of moisture. Calibration standards are under development [7]. For species other than water, a 0.2-percent by volume mixture of water, helium, methane, oxygen, argon, and carbon dioxide in nitrogen is used to determine the respective sensitivity factors. Results indicate that, during normal mass spectrometer operation, these need to be checked only once per week. Limitations for this system are: the sensitivity for oxygen is low; for the <10 amu range, both a mass defect and lower sensitivity require that a sensitivity factor be determined with a standard similar in composition to the sample being analyzed and that the amu positions of the hydrogen and helium peaks are established.

Identification of Organic Contaminants

Caution: This method is used only for the identification of species and is not applicable for the obtaining of quantitative data. One reason for this is that the mass transfer effect will be pronounced for species of widely different mass values. Several scans of a small portion of the sample are made. A representative scan is selected and normalized with respect to the major component, usually nitrogen (table 2A). Clean packages will not exhibit significant values above m/e 28 and 29 with the exception of 40 and 44. A significantly large peak may represent the molecular ion of a compound and/or its base peak (most abundant peak). Therefore, the m/e 45 peak was selected as a possibility and all data normalized with respect to it (table 2B). Mass spectral matching indicated that i-propyl alcohol was a possibility. Plausibility in this case was confirmed by reviewing the package assembly process with the manufacturer; also a corrective step of improved bake-out of the components following the cleaning with the alcohol was adopted.

The validity of a qualitative analysis of a single sample can be increased by knowledge of sample history, etc. For example, the contaminant, methyl ethyl ketone (table 3A), was conclusively identified by mass spectral matching. Normalization of the data to m/e 43 is shown in table 3B. Both the Library File (generated with the instrument using a known compound) and the ASTM listing (table 3C) were used for the matching. Inquiries concerning the package epoxy preform were made because an evaluation report on the type of adhesive and its specification listed acetone as the solvent. Part history (preform batch no.) was unavailable but the vendor stated that methyl ethyl ketone or acetone were used as solvents in different batch processes.

Quantitative Results

The gas analysis (table 4) comprises a portion of a typical failure analysis report at JPL. Leak tests results for the three accepted packages are nominal but the gas analysis shows dramatic dissimilarities. Because of the low sensitivity for oxygen (common to many quadrupole instruments), argon (0.9 percent in air) is used to indicate air leakage. S/N 1193 shows the presence of helium, argon, and a low percentage of nitrogen, and this is what would be expected if the package had a temporary loss of hermeticity prior to the helium leak test. S/N 1388 is similar to S/N 1193 except that it may have been hermetic during a helium leak test. S/N 1293 contained a large trace of organic contaminants which were identified as Freon TF and an alcohol. Both of these are customarily used as cleaning solvents during package assembly. In accordance with the nature of the contaminants' spectra (contribution to m/e 44), the entry for carbon dioxide is qualified. A comprehensive gas analysis can aid in establishing the probable time at which the desired fill gas and/or hermeticity of the device was not maintained. The excessive amount of moisture (3.4 to 5.8 percent) found in all of the packages as well as the large trace amounts of organic solvents in one of the packages clearly point to problems in preseal bakeout and sealing.

SUMMARY

During the past 2-1/2 years, hundreds of packages (volume range 5×10^{-3} cm^3 to several cubic centimeters) have been analyzed as a part of the failure analysis program. Gas analysis has been a very useful tool for the evaluation of the packaging technology. A great deal of information has been obtained which has significantly helped the manufacturer to control the sealing process. Recently, participation with other laboratories in testing "standard" packages of a certain moisture level was completed. The need exists for the extension and continued effort in this area so as to provide an available technique for the certification via gas analysis which is acceptable by the concerned parties.

Total moisture as determined by the mass spectrometric method can be a reliable method provided the following points are observed:

1. thermal equilibration of the system,
2. sample/standard matching,
3. stability of system response, and
4. availability of standard packages of a well-established moisture level for calibration.

ACKNOWLEDGMENT

The authors are grateful to Dr. Robert Thomas of RADC and to John Pernicka of Pernicka Corporation for their many suggestions and aid in the successful development and application of the package gas analysis system. Also, they

gratefully acknowledge the help given by the members of the JPL Parts Failure Analysis Group.

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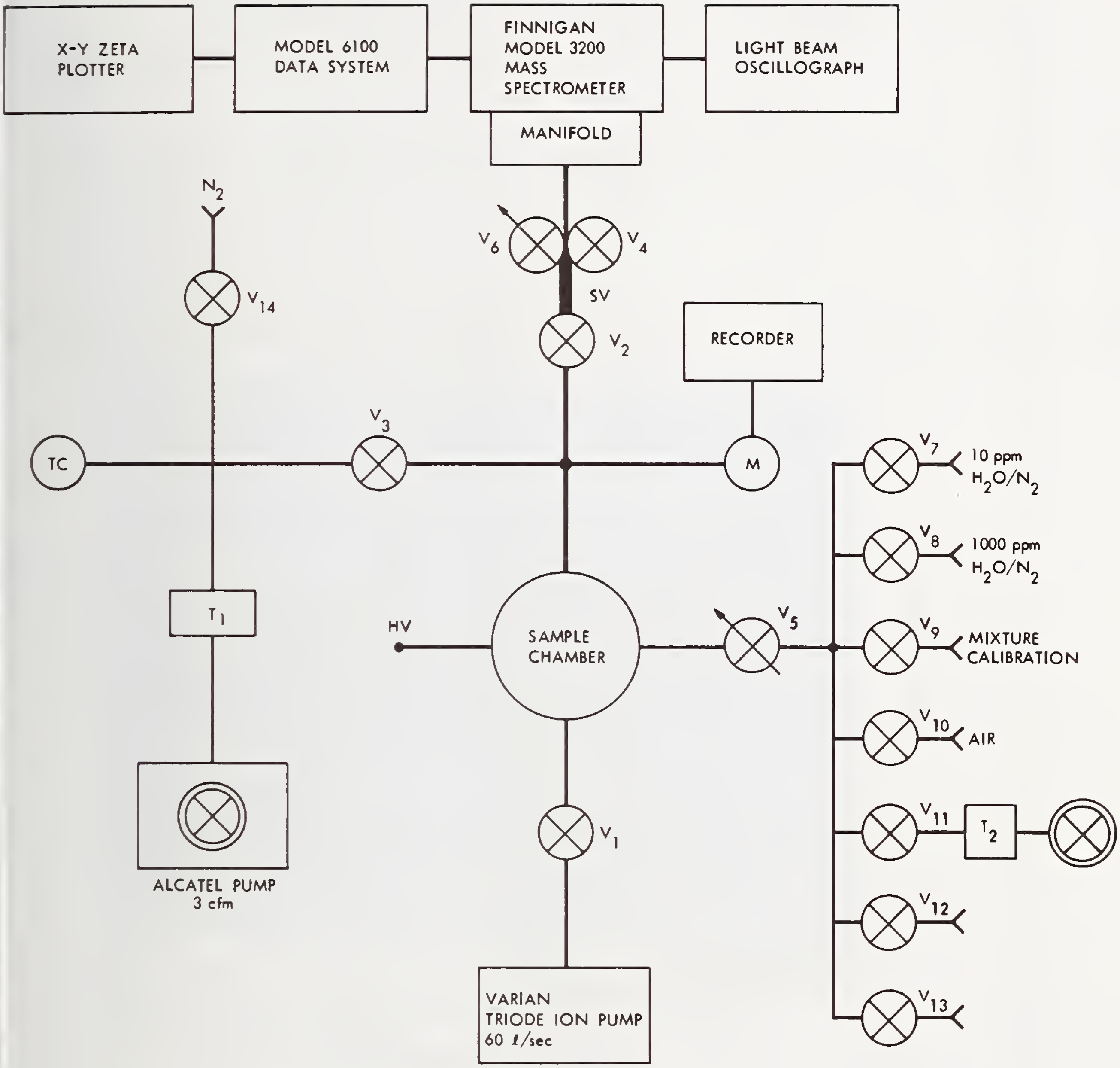
4. Thomas, R. W., private communication, Rome Air Development Center, Solid State Applications Section, Griffiss AFB, Rome, New York.

5. Submitted by Pernicka Corporation, Sunnyvale, CA, under contract to Jet Propulsion Laboratory, Pasadena, California, Task Order #RD-147.

6. Moisture standards (10 ppm water in nitrogen and 1000 ppm water in nitrogen) as obtained from Airco Industrial Gases, Riverton, New Jersey. These standards are of the type described by S. Wechter and F. Kramer, Jr., in *Evaluation of Gas Phase Moisture Standards Prepared in Treated Aluminum Cylinders* (presented at the 21st National Symposium, Analysis Instrumentation Division, Instrument Society of America, May 8, 1975).

7. Cordasco, V. T., Control of Microcircuit Contamination, A Microsensor for Moisture Measurement, RADC-TR-75-317, Final Technical Report (March 1976).

8. American Society for Testing and Materials, Committee E14 on Mass Spectrometry, Index of Mass Spectral Data, listed by Molecular Weight and the Six Strongest Peaks (1969).



V_1	4" GATE VALVE, PNEUMATIC, THERMIONICS
V_2 - V_4	SS BELLOWS VALVE, PNEUMATIC, NUPRO
V_5	THROTTLE VALVE, MONEL DIAPHRAGM, HOKE
V_6	VARIABLE LEAK, MICROMETER ADJUST, NUPRO
V_7 - V_{14}	SS-4H, NUPRO
M	CAPACITANCE MANOMETER, MKS TRU-TORR, 0.000 - 8.000 TORR, DIGITAL DISPLAY
T_1, T_2	MOLECULAR SIEVE TRAP
HV	HIGH VOLTAGE FEEDTHROUGH, MAY BE USED WITH ARGON PURGE UNDER GLOW DISCHARGE (OPTIONAL)
SV	SAMPLING VOLUME

Figure 1. IC package gas analysis.

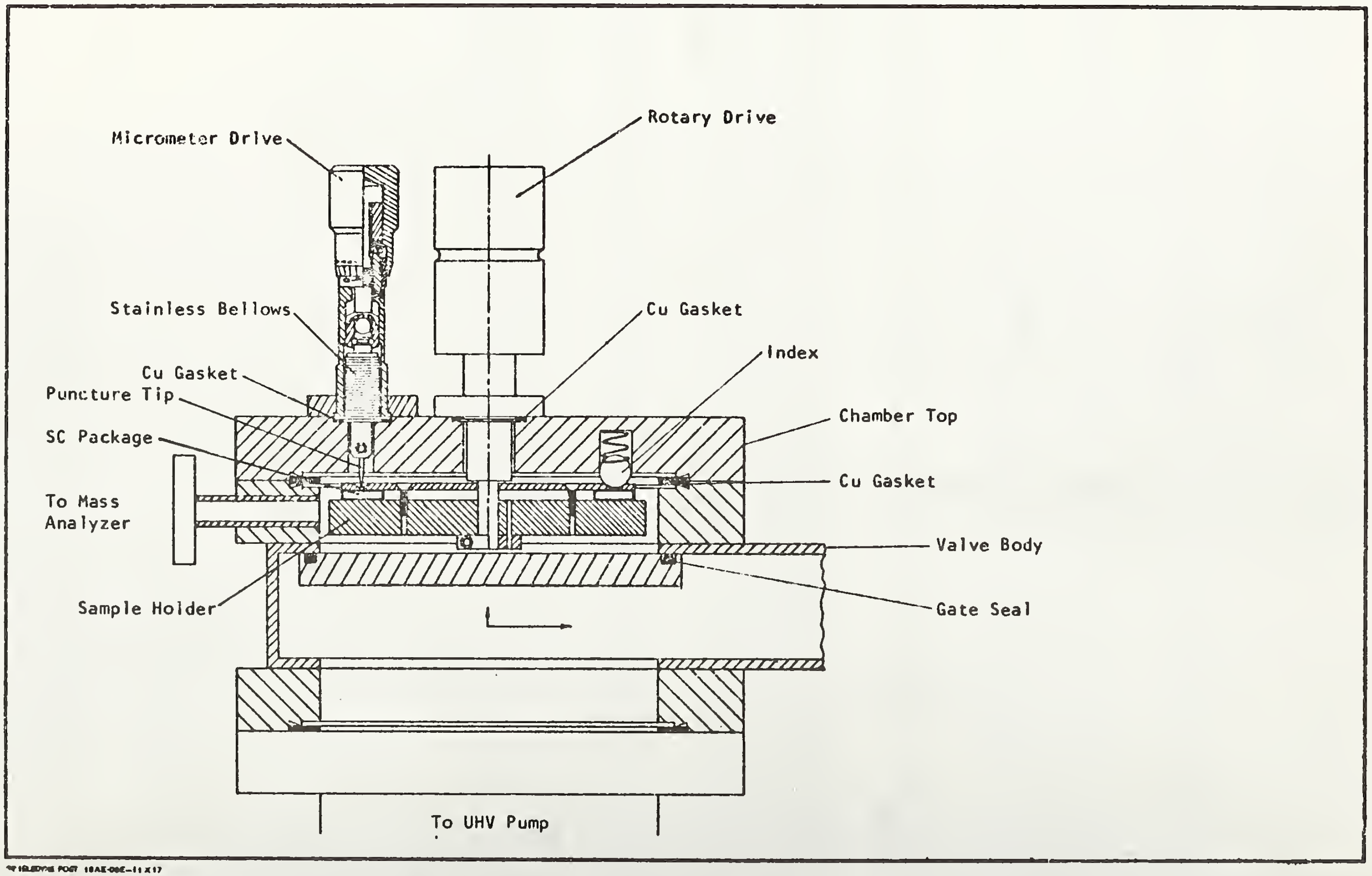


Figure 2. Chamber detail - microcircuit sealed-package gas analysis accessory for running sequential samples in mass spectrometer.

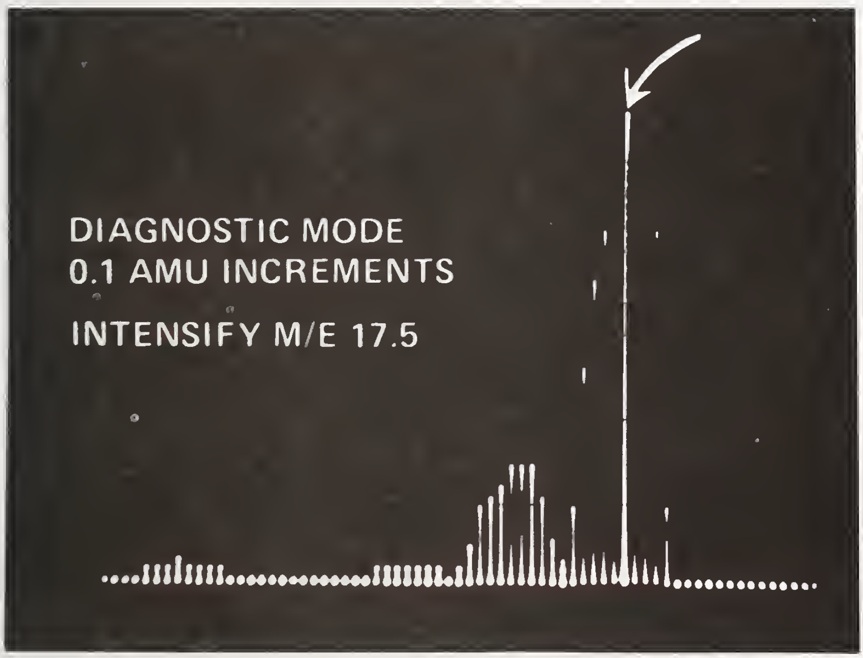


Figure 3. Diagnostic mode - Finnigan Model 6100 Data System 0.1 amu increments, m/3 17.5 intensified.

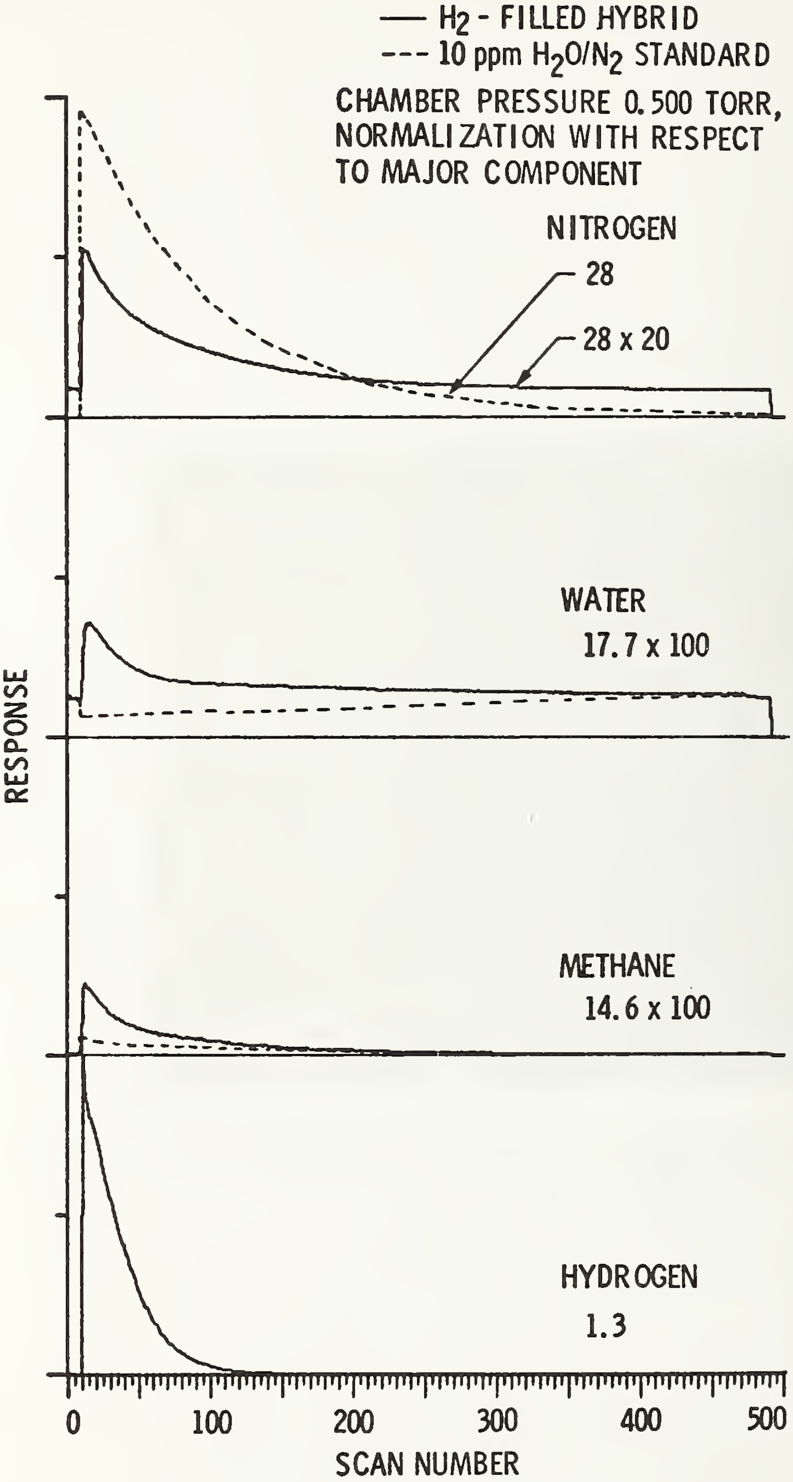


Figure 4. Mass fragmentogram for hydrogen-filled hybrid.

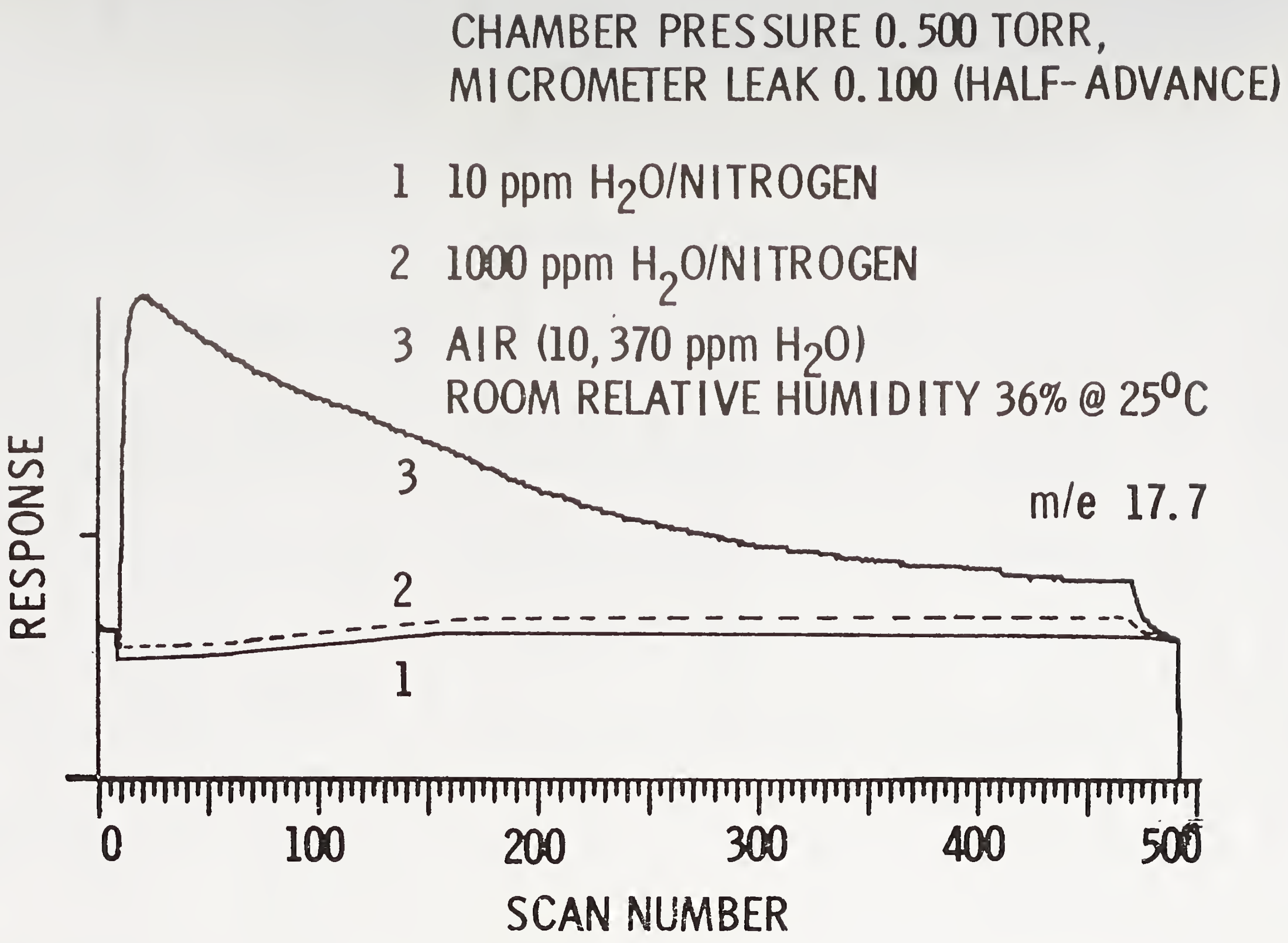


Figure 5. Mass fragmentogram for moisture calibration.

Table 1. Package Analysis Sequence.

1. Conditioning (ascertain background level)
2. Package #1 analysis
3. Major constituent calibration and/or background equilibration
4. Package #2 analysis
5. Repeat step #3
6. Calibration
    a. Moisture
    b. Other constituents (H_2, He, CH_4, Ar, CO_2)

FINNIGAN MODEL 3200 GC/MS DATA SHEET

- Control Settings -

File #	Sample	Pressure (Torr)	Micrometer Setting	Nitrogen		Water Scan 10 - 468 Total Intensity x 10^3	ΔH_2O Area
				Area x 10^6	Bkg x 10^3		
92201	N_2 Std.	5.995	0.010	2.634	74	50.78	
		6.001	↓	2.592	74	51.03	
92202	7-2	7.273		3.010	72	70.88 ←	20.42*
92203	N_2 Std.	7.272		2.945	72	52.36	
		7.273		2.875	71	50.46 ←	
- Remaining Samples -							
92207	N_2 Std.	7.173		2.711	65	48.94 ←	
92208	Air (1.44% H_2O)	7.169		1.900	62	87.49 ←	38.55

* Determined moisture 0.70%

Table 2. Mass Spectral Data (Contaminant - isopropyl alcohol).

```
IIA
MASS RANGE 1-200
FILE 60702 SCAN 22-14
8E
SPEC. #22

  0      .00    .28    .00  16.48   .00   .00    .00
  7      .00    .00    .00    .00   .00   .00   2.13
 14      .00    .00    .00    .00   .16   .52    .00
 21      .00    .00    .00    .00   .00   .16    .96
 28   100.00   1.44    .00    .44   .00   .00    .00
 35      .00    .00    .08    .12   .40   .08    .52
 42      .32   1.36    .64   6.03   .12   .00    .00
 49      .08    .00    .00    .00   .00   .00    .00
 56      .00    .00    .08    .20   .00   .00    .00
 63      .00    .00    .00    .00   .00   .00    .00
 70      .00    .00    .00    .00   .00   .00    .00
 77      .00    .00    .00    .00   .00   .00    .00
 84      .00    .00    .00    .00   .00   .00    .00
 91      .00    .00    .00    .00   .00   .00    .00
 98      .00    .00    .00    .08   .00   .06    .00
105      .00    .00    .00    .00   .00   .00    .00
112      .00    .00    .00    .00   .00   .00    .00
119      .00    .00    .00    .00   .00   .00    .00
126      .00    .00    .00    .00   .00   .00    .00
133      .00    .00    .00    .00   .00   .00    .00
140      .00    .00    .00    .00   .00   .00    .00
147      .00    .00    .00    .00   .04   .00    .00
```

```
IIB
FILE 60702 SCAN 22-14 NORM M/E 45
8E
SPEC. #22

  0      .00   4.66    .00 273.33   .00   .00    .00
  7      .00    .00    .00    .00   .00   .00  35.33
 14      .00    .00    .00    .00  2.66  8.66    .00
 21      .00    .00    .00    .00   .00  2.66  16.00
 28      >    24.00    .00   7.33   .00   .00    .00
 35      .00    .00   1.33   2.00  6.66  1.33   8.66
 42     5.33  22.66  10.66 100.00  2.00   .00    .00
 49     1.33    .00    .00    .00   .00   .00    .00
 56      .00    .00   1.33   3.33   .00   .00    .00
```

ISOPROPYL ALCOHOL
(Gas, ASTM)

m/e	%
45	100
43	17
27	16
41	7
39	6
31	6
29	10

Table 3. Mass Spectral Data (Contaminant - methyl ethyl ketone).

```
IIIA
MASS RANGE 1-200
FILE 50505 SCAN 39-14
S/N 97
SPEC. #39

  0     .00     .00     .19  1.98    .00    .00     .00
  7     .00     .00     .00   .00    .58    .61   41.99
 14    4.66    1.62     .54  1.42    .00    .00     .00
 21     .00     .00     .00   .00    .17   1.39    6.06
 28  100.00   11.90     .24   .27  14.04    .00     .07
 35     .00     .04     .22   .29    .95   1.30     .93
 42    1.66   28.93    4.98   .46    .00    .00     .00
 49     .09     .22     .17   .04    .17    .04     .19
 56     .07    1.76     .36   .04    .00    .00     .00
 63     .00     .00     .00   .00    .00    .00     .00
 70     .00     .12    3.06   .14    .00    .00     .00
 77     .00     .00     .00   .00    .00    .00     .00
 84     .00     .07     .00   .00    .00    .00     .00
 91     .14     .00     .00   .00    .00    .00     .00
 98     .00     .00     .04   .00    .00    .00     .00
105     .00     .04     .00
```

```
IIIB

FILE 50505 SCAN 39-14 NORM M/E 43
S/N 97
SPEC. #39

  0     .00     .00     .67  6.87    .00    .00     .00
  7     .00     .00     .00   .00   2.03   2.12  145.12
 14   16.11    5.59    1.86  4.91    .00    .00     .00
 21     .00     .00     .00   .00    .59   4.83   20.94
 28       >   41.13     .84   .93  48.51    .00     .25
 35     .00     .16     .76  1.01   3.30   4.49    3.22
 42    5.76  100.00   17.21  1.61    .00    .00     .00
 49     .33     .76     .59   .16    .59    .16     .67
 56     .25    6.10    1.27   .16    .00    .00     .00
 63     .00     .00     .00   .00    .00    .00     .00
 70     .00     .42   10.60   .50    .00    .00     .00
 77     .00     .00     .00   .00    .00    .00     .00
 84     .00     .25     .00   .00    .00    .00     .00
 91     .50     .00     .00   .00    .00    .00     .00
 98     .00     .00     .16   .00    .00    .00     .00
105     .00     .16     .00
```

```
IIIC
LIBRARY FILE - METHYL ETHYL KETONE

 21     .00     .00     .00   .07    .74   6.45   21.39
 28    5.31   36.20     .85   .51    .00    .00     .00
 35     .00     .12     .85  1.08   3.12    .36    2.16
 42    7.32  100.00    3.40   .95    .00    .00     .07
 49     .33     .82     .49   .15    .74    .20     .72
 56     .30    8.00     .28   .00    .00    .00     .00
 63     .00     .00     .00   .00    .00    .00     .00
 70     .00     .85   20.36  1.16    .05
```

METHYL ETHYL KETONE
(Gas, ASTM)[8]

m/e	%
43	100
29	24
72	17
27	16
57	6
42	5
26	5

Table 4. Typical Results - Package Gas Analysis.

S/N	Fine Leak Test[a] (x 10^{-8} cc/sec He)	Relative Burst Pressure	COMPONENTS (% VOLUME/VOLUME)								
			He	CH_4	H_2O	N_2	O_2	Ar	CO_2	H_2	Other
1193	0.78	1.000	Large Trace	N.D.[b]	3.4	∿ 84.3	∿ 10.5	0.62	1.23	N.D.	N.D.
1212	30.2 ← Reject for G/A										
1293	0.60	0.932	N.D.	0.10	4.2	95.39	Trace	Trace	(m/e 44 0.32)	N.D.	Trace Freon TF,[c] alcohol
1388	0.72	1.300	N.D.	N.D.	5.8	∿ 75.0	∿ 17.6	0.97	0.64	N.D.	N.D.

[a] Gross Leak Test: No Leaks Observed

[b] None detected

[c] Library File: 1,1,2-Trichloro-1,2,2-trifluoroethane and an alcohol (i-propyl?)

2.6 Comparison of Mass Spectrometric Moisture Measurements by Different Laboratories

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INTRODUCTION

As part of an MM&T (Manufacturing Methods and Technology) Study on the screen testing of electronic components performed for Army/MIRADCOM, an investigation was made to determine the quality of the existing capability for measuring the moisture contents of microcircuit packages. This investigation consisted of preparing moisture standards or standard packages (i.e., packages filled under precisely controlled conditions with nitrogen containing known concentrations of moisture), having identical sets of these packages analyzed by different laboratories that are recognized experts in moisture measurement, and comparing the results.

The primary purpose of this paper is to present and discuss these results. However, since the validity of these results and the credibility of this entire effort depends on the propriety of the standard packages, they will be discussed first.

The Standard Packages

The basic method selected for preparing the standard packages is depicted in figure 1. Nitrogen of the desired moisture content is flushed through the lidded package via two attached tubes while the moisture content at the input and output is monitored by an optical dew point hygrometer. When equilibrium is established at the desired moisture content as indicated by the equivalence of the optical dew point hygrometer readings, the package is sealed by simultaneously sealing off the two tubes.

The package used for this application is an empty 2.54-cm (1-in.) square gold-plated Kovar package blank or box (i.e., a package without electrical feedthroughs) sealed with a gold-plated Kovar lid containing two 0.316-cm (1/8-in.) outside diameter gold-plated copper tubes located diagonally in two of its corners. The lid is seam-sealed to the package case using a gold-tin preform prior to processing, and final seal is accomplished by simultaneously cold welding (pinching off) the two tubes. A photograph of this package is shown in figure 2.

The method by which nitrogen of the desired moisture content is produced is illustrated in figure 3. Nitrogen partially saturated with moisture is generated by flushing dry nitrogen from the laboratory supply through a flask containing deionized water and mixing it with dry nitrogen directly from the laboratory supply. This partially saturated nitrogen is then circulated through a double wound coil of copper tubing immersed in a controlled temperature bath set at the value required to produce nitrogen of the desired moisture content. The precise value of the moisture content of the output nitrogen is measured using an optical dew point hygrometer.

A diagram of the setup used for preparing the standard packages is given in figure 4. This setup consists of a manifold to which five packages can be connected, a laboratory source of dry nitrogen for purging the packages and the optical dew point hygrometer, a source of nitrogen of controlled moisture content for filling the packages, an optical dew point hygrometer for monitoring the moisture content of the nitrogen, pressure gages to assure that the packages are sealed at atmospheric pressure, provision for connecting a leak detector to troubleshoot the system, and the necessary plumbing (tubing, couplings, and valves), pressure regulators, flow gages, and exhausts to interconnect these major components as required and assure proper operation of the setup.

A photograph of the manifold portion of this setup and connection of the ancillary equipment completing the setup is shown in figure 5. As just stated, the manifold can accommodate five packages; however, valving is provided to isolate the packages from each other so that any number of packages from one to five can be processed as desired. Valves also are provided so that the moisture content (dew point) can be measured at both the package input and output. Also, magnehelic pressure gages (0 to ±10 in. of water) are attached at both the input and output to assure that the packages are sealed at atmospheric pressure. Flow gages are provided at the output and at the optical dew point hygrometer to assure that the flow is within the 0.5 to 5 SCFH (standard cubic feet per hour) range required for proper operation of the optical dew point hygrometer.

Provision (not shown) also has been made to heat the system to 125°C. The packages are heated by twining a heater strip among them and covering them with an insulated metal box. The temperature is monitored by a thermocouple attached to one of the packages. The plumbing inside the large manifold box is heated by serpentining a long heater strip. The inside bottom and top of the box is insulated to prevent heat loss. The plumbing extends below the insulation and the temperature is monitored by a thermocouple attached to one of the tubes or valves. The manifold box is aluminum and all plumbing (tubing, valves, and couples) is stainless steel.

The procedure used in preparing the standard packages is as follows:

1. The package cases and lids are cleaned in an ultrasonic Freon TF bath. The lids are checked for cleanliness by immersing each lid in a circulating deionized water bath (10 MΩ minimum) with a conductance monitor at the overflow and noting that the resistance of the water does not decrease. The tubes are then flushed with isopropanol and Freon TF and blown dry with nitrogen.

2. The package cases and lids are assembled by seam-sealing them in a normal dry nitrogen sealing environment (dry box) using gold-tin preforms.

3. The packages are mounted on the manifold and checked for leaks using the leak detector.

4. The packages are purged simultaneously by flushing them with dry nitrogen from the laboratory supply. Progress of the purging cycle is monitored by periodically measuring the moisture content at the manifold output using the optical dew point hygrometer and is considered completed when this measurement indicates a moisture content well below that at which the packages are to be filled.

5. After purging, the laboratory nitrogen supply is shut off and the source of nitrogen of controlled moisture content is turned on, and valves are switched to permit each package to be filled individually. Moisture content at the package output is monitored using the optical dew point hygrometer and when equilibrium is reached, the reading is recorded, valves are closed to isolate the package, and the copper tubes are pinched off. During this fill cycle, pressure is monitored to assure that the package is at atmospheric pressure at pinch off.

6. The packages are leak tested (both fine and gross) and the tips of the tubes are dipped in solder.

Standard Packages for the Present Investigation

Ninety standard packages were prepared for the present investigation, 30 each at moisture contents of approximately 1000, 6000, and 10 000 parts per million by volume (ppm_v). Fifteen of the 30 packages prepared at each moisture content were processed and sealed off at room temperature and 15 at 125°C. The packages were prepared in batches of five and systematically labeled. After all of the packages were prepared, they were grouped into five sets consisting of one package from each batch to assure that the sets of packages were essentially identical. These five sets of packages were then sent to the five selected laboratories for moisture analysis. Each laboratory was sent a total of 18 packages, six each prepared at moisture contents of 1000, 6000, and 10 000 ppm_v, of which three of each moisture content were processed and sealed off at room temperature and three at 125°C.

Results

The results obtained from the four laboratories that responded are presented in tables 1 and 2 for the packages filled at room temperature and at 125°C, respectively. The results obtained by the laboratories differ widely and none agree with the fill values. The values obtained by Lab 2 were in closest agreement with the fill values. Those obtained by Lab 3 were higher than the fill values by factors of 3 to 4, and those obtained by Labs 4 and 5 were lower than the fill values by factors of 3 to 5 for the high moisture content packages (5950 and 10 000 ppm_v), and by factors of 4 or 5 to 15 for the 1050 ppm_v moisture content packages.

These results are significant in that they are representative of the state-of-the-art capability of the moisture measurement technology and attest to the need for improvement.

There also is another interesting observation to be made from these results. It was expected that the measured moisture contents would be higher for the packages filled at room temperature than for those filled at 125°C at the three different moisture contents due to the fact that more moisture would be adsorbed on the walls of the packages at the lower temperature, and this moisture would be released at the higher temperature at which the moisture measurements are made. However, a comparison of tables 1 and 2 shows that this was not the case — the measured moisture contents of the packages filled at 125°C, in general, were at least as high as or higher than those filled at room temperature.

A special comment must be made regarding the results obtained by Lab 3. After discussing the large difference between the moisture contents, they measured for the packages and the fill moisture contents of the packages; they carefully examined their system and discovered that a thermal runaway that damaged their moisture calibration system had occurred causing the results to be high by a factor of three. The corrected values obtained by Lab 3 are given in table 3. With this correction, the Lab 3 results are in much closer agreement with the fill values than any of the other laboratories.

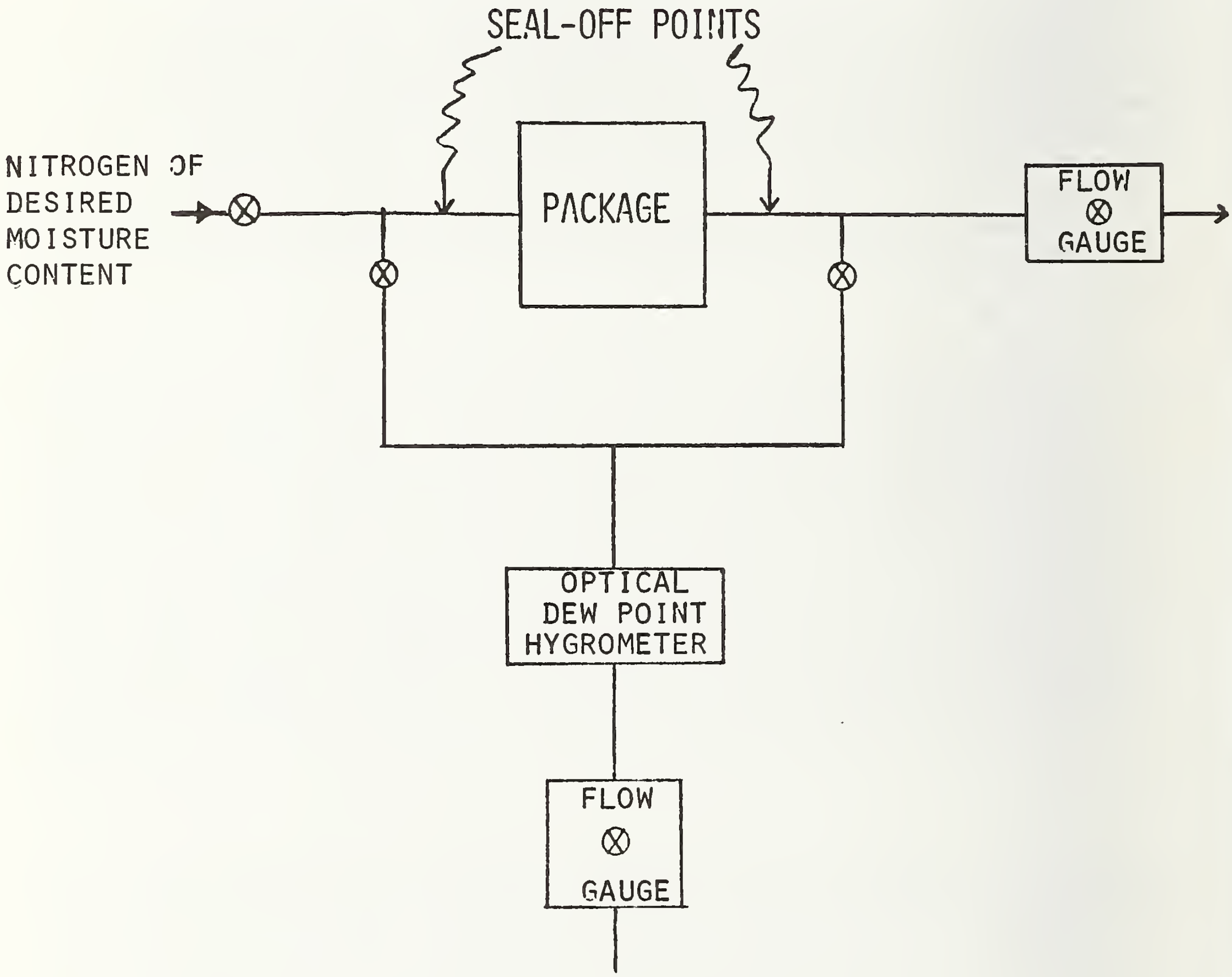

Figure 1. Basic method for preparing standard packages.

Figure 2. Moisture standard package.

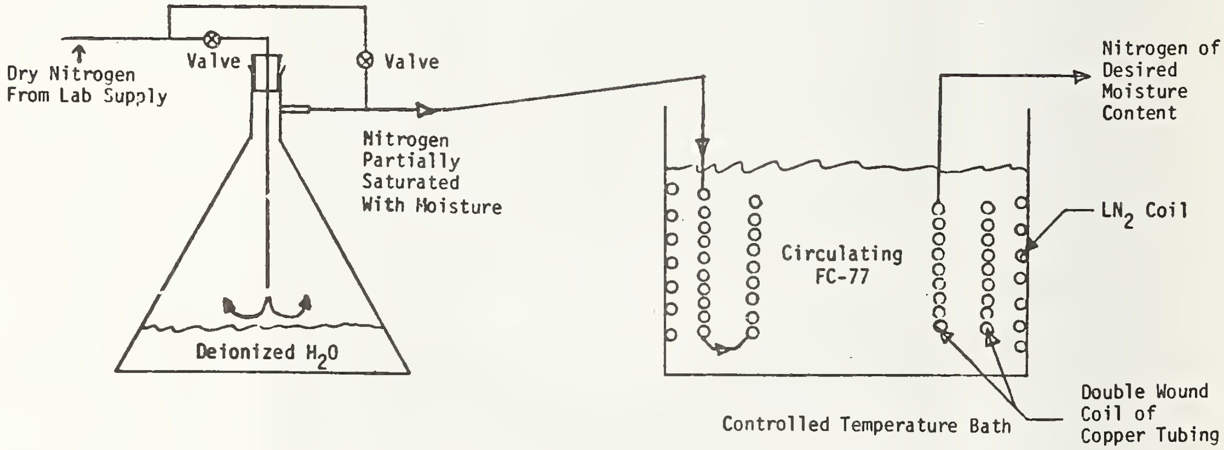

Figure 3. Method for producing nitrogen of desired moisture content.

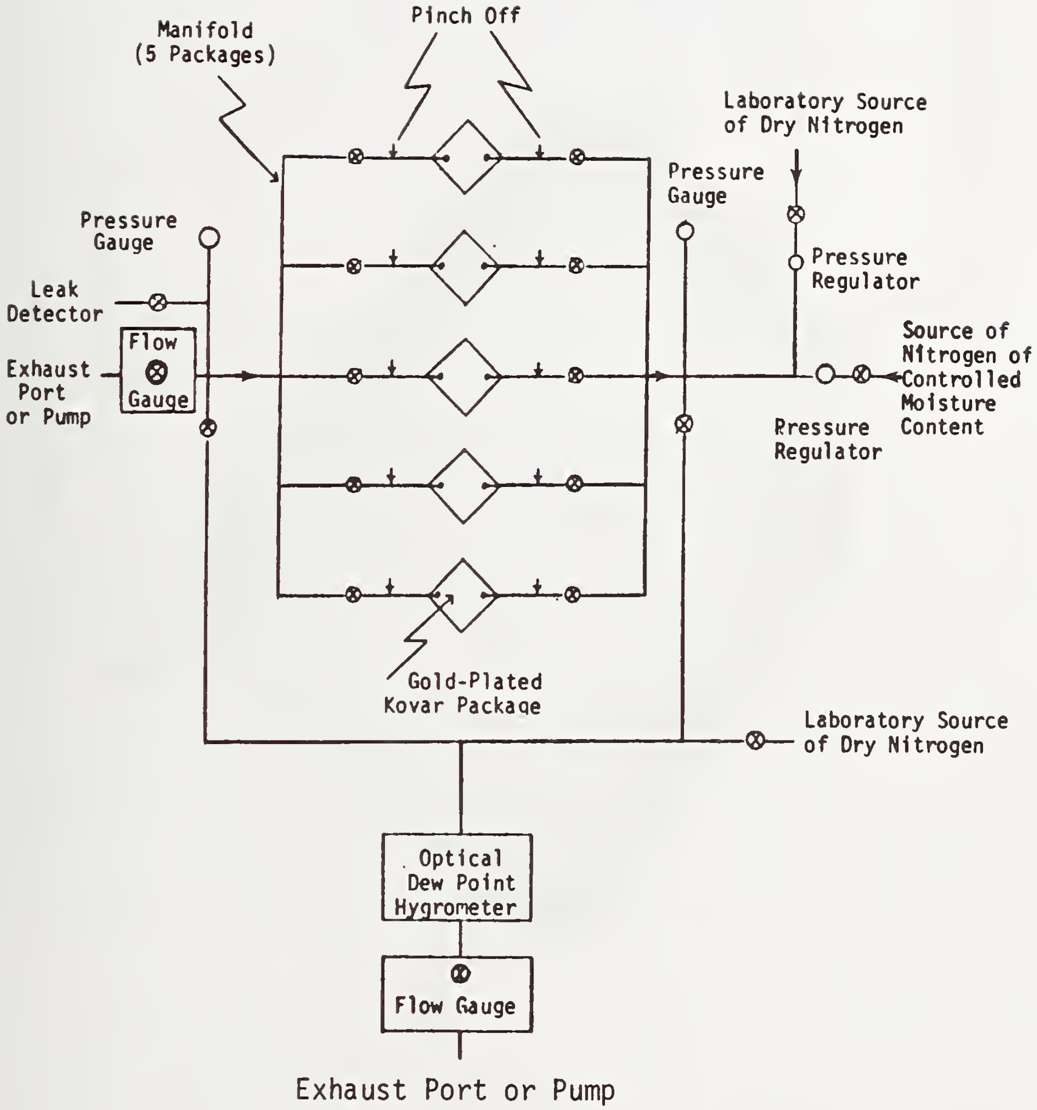

Figure 4. Diagram of setup for preparing standard packages.

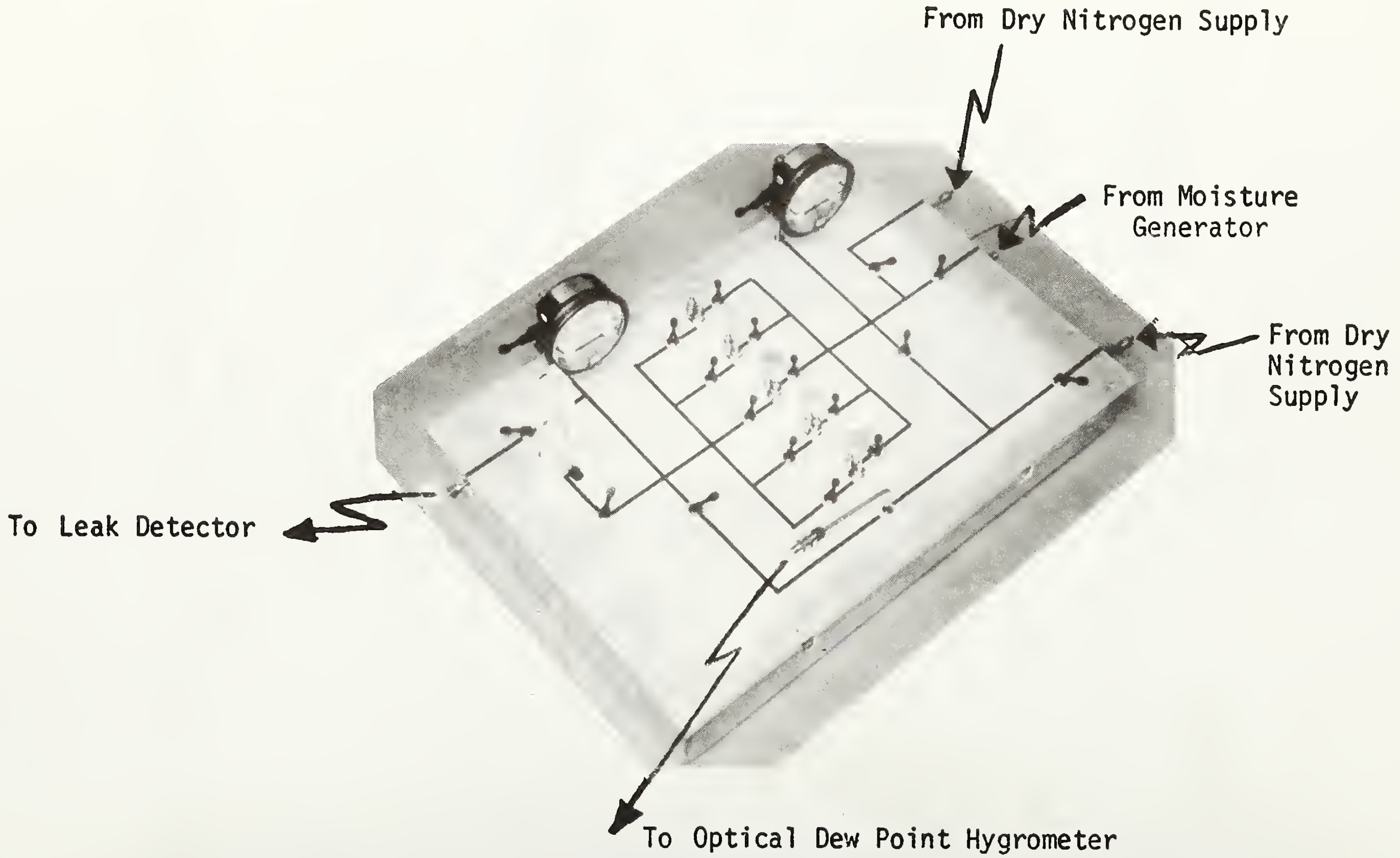

Figure 5. Manifold portion of setup for preparing standard packages with ancillary equipment indicated.

Table 1. Moisture Analysis Results for Standard Packages Filled at Room Temperature.

Filled Moisture Content (ppm_v)	Measured Moisture Content (ppm_v)			
	Lab 2	Lab 3	Lab 4	Lab 5
1,050	500	3,000	158	60
	900	2,700	60	90
	600	3,100	83	170
5,950	7,000	19,400	1,448	1,200
	9,800	20,400	1,525	1,160
	11,600	23,000	1,124	2,580
10,000	14,500	37,000	2,864	2,870
	17,600	--	3,095	2,820
	17,100	33,000	3,140	2,560

Table 2. Moisture Analysis Results for Standard Packages Filled at 125°C.

Filled Moisture Content (ppm_v)	Measured Moisture Content (ppm_v)			
	Lab 2	Lab 3	Lab 4	Lab 5
1,050	500	11,000	186	200
	500	3,100	135	250
	500	3,100	88	390
5,950	13,100	24,000	1,458	1,360
	13,900	21,000	1,419	1,510
	15,600	20,500	1,540	1,580
10,000	14,000	42,000	2,967	2,750
	17,700	40,000	2,750	2,230
	17,800	34,000	2,730	2,370

Table 3. Corrected Lab 3 Results for Standard Packages Filled at Room Temperature and at 125°C.

Fill Moisture Content (ppm$_v$)	Measured Moisture Content (ppm$_v$)	
	RT	125°C
1,050	1,000	3,700
	900	1,030
	1,030	1,030
5,950	6,500	8,000
	6,800	7,000
	7,600	6,800
10,000	12,000	14,000
	—	13,000
	11,000	11,000

3. SESSION II MOISTURE SENSORS

3.1 The Effects of Assembly Techniques Upon the Performance Characteristics of Al_2O_3 *In-Situ* Moisture Sensors*†

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INTRODUCTION

Aluminum oxide moisture-sensing chips have been described in previous literature [1,2]. Information of a preliminary nature with respect to how hybrid processing affects the performance characteristics has also been reported. In an effort to expand information that had been reported by Kovac [2], several experiments were conducted in order to augment present understanding of process variables which affect the moisture *versus* admittance characteristics of aluminum oxide sensors.

In the discussion of aluminum oxide moisture-sensing chips, the term "aluminum oxide" should be interpreted in the context of aluminum oxide produced with the design, process, and applications compromises peculiar to the Panametrics MMA moisture-sensing chip. All information is based on the use of standard TO-5 used as a package study media.

Design Considerations

The Panametrics MMA has been designed with the hybrid and semiconductor manufacturer in mind. The moisture sensor is fabricated on a 60 by 60 mil square silicon chip, where the silicon is thermally oxidized and the resultant purity of silicon dioxide is comparable to that of an MOS. Symmetrical 6-mil square bonding pads are forgiving of any random orientation which the chip might assume when reaching its terminal position on the substrate or header. If the gold silicon eutectic system is to be used for die bonding, it is possible to die bond Mini-Mod-A's without the use of a preform.

Admittance *Versus* Moisture Characteristics

The theory of calibration of aluminum oxide sensors is simple and straightforward and is based on Henry's Law. At Panametrics dry nitrogen from a liquid source is bubbled through a saturator where the saturator is

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maintained at approximately room temperature, but the temperature is precisely measured so that one knows the partial pressure of the water vapor in the nitrogen/water stream. The stream can then be diluted with known volumetric quantities of nitrogen, so that dew points or parts per million by volume (ppm_v) can be generated with fair precision, and sensors are thereby calibrated. To give some idea of correlatability with a reference standard, one can refer to figure 1.

In this set of curves a calibration of Mini-Mod-A sensors is shown using an industrial foil sensor such as a Panametrics Type M22 as a primary standard. This foil sensor was calibrated by the method previously mentioned. Although seemingly indirect, it is a convenient technique to use eliminating constant temperature baths and precision-calibrated flowmeters. Still the results correlate within 2°C of a direct calibration using the National Bureau of Standards' two-pressure system. The results are averages of four Mini-Mod-A sensors calibrated by NBS and then calibrated by Panametrics' simplified laboratory technique previously described.

Effect of Die Attach

The volatile by-products which in many cases includes ammonia have been found to affect the admittance *versus* moisture characteristics of aluminum oxide moisture-sensing chips. There is one exception. It has been shown that chips from the same wafer wire bonded by holding the chips with tweezers on headers during the ultrasonic wire bonding are precisely comparable to their counterparts mounted with Eastman 910 epoxy. We shall define such a calibration curve as a so-called "neutral" curve from time to time as a tool of reference. Figure 2 shows the differences when groups of chips from the same wafer are mounted with various epoxies and ultrasonically wire bonded at room temperatures. Eppotech 31LV is similar to the "neutral" Eastman 910 curve, but shows divergence at -15°C dew/frost point. A gold-filled Eppotech is considerably different.

Another case of die bond effects can be shown in figure 3. Again, the "neutral" curve is shown for purposes of reference. The "Indium" curve is actually a 50-50 weight percent indium-lead alloy, where the liquidus forms at 208°C. The gold-silicon curve was generated by using eutectic goldsilicon preforms which melt at 370°C. Heating rearranges the pore structure of the aluminum oxide. The trend of heat treatment can either result in steepening of the calibration curve at the moist end or the lowering of admittance or both.

Since we are dealing with sensors fabricated on silicon substrates, they can be manufactured with gold backing so that they can be die bonded directly to a gold substrate without the use of a preform.

Effects of Wire Bonding

The effects of wire bonding are similar to the effects of braze or eutectic die bonding and are determined by the heating column temperature during wire bonding. Such effects are observable only if the wire bonding is performed at a temperature appreciably higher than a prior die bond temperature.

Effects of Baking and Sealing

Four groups of individually calibrated sensors mounted on TO-5s, vacuum baked for 4 h at 150°C and resistance welded in nitrogen of 70 ppm_v moisture level, showed no changes after puncturing the cap and recalibrating the sensors.

A CERDIP process was simulated by taking the TO-5 units used to generate the gold-silicon curve of figure 3 and dividing them into two subgroups. The first subgroup was heated in nitrogen for 10 min at 450°C. The second subgroup was heated under the same conditions, except that the maximum temperature was 500°C. The results are shown in figure 4. The left curve is actually two curves superimposed, indicating no change from original values in the case of the TO-5s heated at 450°C. This is not surprising, since the maximum heating column temperature for gold-silicon die bonding is probably quite close to 400°C. At 500°C there is a noticeable change as the pore structure of the aluminum oxide sensors again undergoes modification due to heat stressing at a temperature moderately higher than a prior process temperature.

Temperature Coefficient

Aluminum oxide chip sensors work on a surface adsorption principle which becomes severely limited at temperatures approaching 100°C. This is further complicated by the present state of mass spectrometry art where correlation from one mass spectrometry system to another system often results in orders of magnitude differences. There is reasonable expectation that with refinement of readout electronics and measurements made at some elevated temperature, say 50°C, better correlation can be established. Such an objective is being seriously considered at Panametrics.

Presently, measurement temperatures have to be limited to a range of 23°C to 39°C.

CONCLUSIONS

Aluminum oxide moisture-sensing chips equivalent to the Panametrics Type MMA can be used in process temperatures up to 500°C. If environments which affect the calibration characteristics of sensing chips are taken into account, estimates of ambient water vapor level within engineering accuracy are readily obtainable. The maximum temperature of process exposure appears to be the governing variable affecting the final characteristics of the aluminum oxide chip sensors, if no epoxies are present in the system. It would, therefore, appear possible to temperature condition chips or wafers so that the final characteristics would not be heat-cycle-dependent.

The cost effectiveness of the aluminum-oxide-sensing chip should be reviewed in the light of many cost advantages. Very low capital investment is required. No specialized training or skill is required to conduct measurements and to interpret results. The tests are nondestructive. The cost of fine and gross leak tests can be eliminated. There is monitoring capability for the life of the product. Finally, confidence levels associated only with 100-percent testing of hermetically sealed packages are possible.

ACKNOWLEDGMENTS

The author would like to acknowledge data supplied by the Rome Air Development Center of the calibrations data obtained from the National Bureau of Standards.

REFERENCES

1. Kovac, M. G., Chlech, D., and Goodman, P., A New Moisture Sensor for *"In-Situ"* Monitoring of Sealed Packages, *15th Annual Proceedings, Reliability Physics 1977,* Las Vegas, Nevada, April 1977, pp. 85-91.

2. Kovac, M. G., Performance Characteristics of Al_2O_3 Moisture Sensor Inside Sealed Hybrid Packages, *Proc. 1977 Int. Microelectronics Sym.,* Baltimore, Maryland, October 24-16, 1977, pp. 249-252.

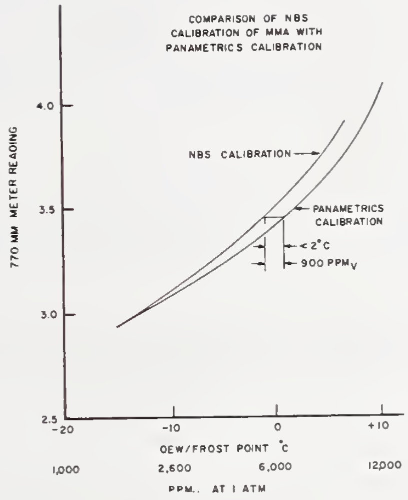

Figure 1. Calibration results for Mini-Mod-A sensors as compared against a Type M22 fail sensor as reference.

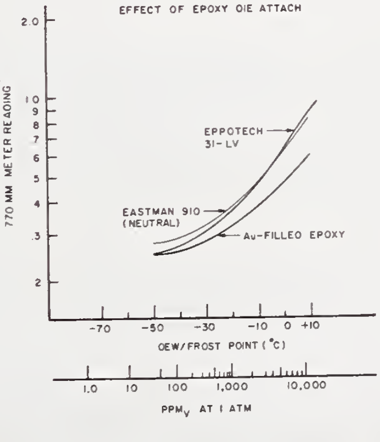

Figure 2. Effect of epoxy die attach on the response of MMA oxide sensors.

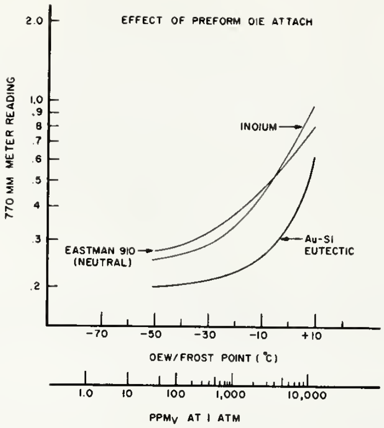

Figure 3. Effect of preform die attach on the response of MMA oxide sensors.

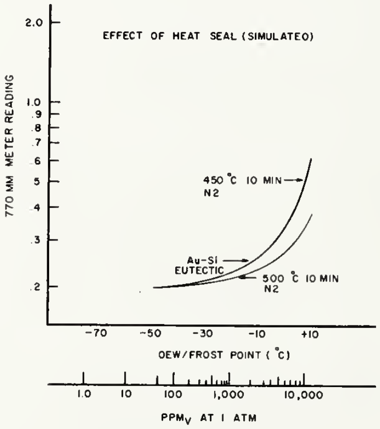

Figure 4. Effect of various package heat-sealing procedures on the response of MMA oxide sensors.

3.2 Application Considerations of Solid State Moisture Chips for Hybrids and Associated Calibration Measurement Techniques*†

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BR-101B-SS Moisture Chip

The Thunder Scientific BR-101B-SS Moisture Chip, a solid-state thin-film bulk effect device, is the result of two years of intensive research and development.

It appears to have fast response, minimum hysteresis, and immunity to saturation which with its small size permit unique applications, we believe, beyond the capability and range of other humidity sensors. Specifically, because of the extremely small size and broad range, the sensor as a substrate chip may be die bonded into hybrid packs and ICs for analysis of moisture leak rates prevalent in most instances.

Theory of Sensor Operation

The moisture chip is literally an array of many interstitial spaces within the sensor accessed by electrodes. Water molecules can drift freely and randomly into and throughout the structure in such a manner as to electrically change its bulk impedance.

Because of the ability of moisture molecules to drift into and out of the structure, the sensor exhibits response times of less than 300 ms for 63 percent of reading. As physical size of the substrate is reduced, response time is equivalently reduced.

Specifications

We find the accuracy to be typically better than 2-percent indicated RH over a range of from 0-percent to 100-percent RH. Inaccuracy is less than 10 parts per million by volume (ppm_v) over the range of 1 ppm_v to 3000 ppm_v. The worst case hysteresis taken under the most extreme conditions is less than 2-percent indicated RH. The best case is less than 1-percent indicated

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RH or nonexistent. The stated guaranteed accuracy is conservative, being ±2-percent indicated RH.

The sensor has been found to also be sensitive to changes in moisture down to a minimum of 0.1 ppm. Therefore, it may be used in applications where it is desired to detect minimal moisture conditions.

The sensor has been tested over temperature ranges from -40°C to as high as 450°C with no apparent degradation.

The chip has also been exposed to altitudes ranging from sea level to 500 000 feet and also to ultra-high vacuum. In all instances, whether exposed for hours or days, the chip continues to function measuring the various amounts of moisture present. Other sensors take considerable time to respond once desiccated by a long-term vacuum.

Applications

The BR-101B-SS moisture chip may be used in a myriad of requirements such as environmental control, pollution studies, process controls, metrology, etc. In particular the intent of this paper is to discuss how well the sensor lends itself for monitor of leak rates in hybrid packs because of its minute size when used in die form.

Mass Spectrometer Method

Analysis of microelectronic packages has to date been done with very sensitive mass spectrometers using special sampling apparatus made specifically for puncturing microelectronic packages.

This method has worked reasonably well, allowing detection of moisture in the package in many instances of device failure analysis.

Analysis by mass spectrometry is not only expensive but time consuming, always resulting in a hole in the package or destruction of same. In addition the mass spectrometer does not lend itself to on-line testing or analysis of hundreds or perhaps thousands of microelectronic and hybrid packages.

Hybrid Package Application

Presently, the maximum allowable moisture level that can be tolerated in a package has not been determined. Therefore, it is suggested, the necessity is to monitor the internal atmosphere of the package not only during sealing operations but also over an extended life period.

Various packages and device types are more sensitive to moisture than others. As an example, a 741 IC chip is extremely sensitive to moisture even at levels of moisture from 20 ppm and up. Higher values of moisture, i.e., 6000 to 8000 ppm, can initiate dendritic growths in small geometry packages between opposite polarities under the proper combination of metallization.

Epoxy-sealed circuits are even more subject to failure because of moisture. Tests have been conducted that indicate the beginning of moisture leakage just after a sealing bake at 125°C. Moisture levels between inside and out completely equilibrate in approximately 12 h at standard temperature and atmosphere if the epoxy is leaking.

The moisture chip in die form measures 0.070 in. by 0.075 in. by 10 mils thick. This size lends itself well to standard die bonding techniques when using tin-gold or other preforms at a temperature of approximately 270°C. The die is constructed in such a manner so as to allow ball bonding, ultrasonic, or thermal welding of gold leads from the pads provided to the hybrid itself.

Due to the small size of the sensor, the intent is to leave the sensor sealed within the package to allow monitoring of package moisture over extended periods of time.

Improved Sensor Characteristics

The standard aluminum oxide chip exhibits a number of deleterious characteristics such as:

1. It is affected by process temperatures and can be permanently damaged by this parameter.
2. It shows hysteresis.
3. Response time is 15 s or more.
4. It exhibits continual aging throughout the life of the sensor.
5. Dynamic range is limited.

We claim that The Thunder Scientific moisture chip exhibits many improved characteristics shown as follows:

1. The chip is linear.
2. Hysteresis is temporary and reduced to 20 ppm or better.
3. Response is 300 ms around reading.
4. Sensitivity is three orders of magnitude better.
5. Repeatability can be as good as 10 ppm or better.
6. Small size — it can be made smaller.
7. The chip does not age as do normal aluminum oxide sensors.
8. It exhibits minimum change with process temperature.

Broader Process Methods

The improved moisture chip due to its inherent construction may be subjected to broader process methods without normally resultant damage or deterioration caused by high temperature, etc. In other words, it more nearly approaches a semiconductor due to added flexibility in process.

Die Bonding

The chip may be die bonded using a number of different materials and methods heretofore not available.

In practice the chip can be constructed upon either amorphous quartz or silicon. In the case of the quartz type, each chip is gold backed. Tin or gold germanium or other eutectics may be used. When the sensor is constructed upon silicon, standard silicon gold eutectic bonding may be used negating the necessity of preforms, etc. In this case, bonding temperatures as high as 270°C may be used without damage.

Lead Bonding

The Thunder moisture chip is ruggedly constructed. Pads provided are greater than 1000 nm thick providing a bond area of approximately 10 mils in diameter. Because of this, the die can be reused if desired.

Any type of bonding method may be used with little difficulty, i.e., ultrasonic, thermal compression, pulse, etc. Stage heat may be run at 100°C to 150°C with no damage.

Bakeout

Bakeout when epoxies are used may be at 120°C or higher.

Vacuum bake-out temperature prior to sealing of the hybrid will not harm the sensor.

High-Temperature Capability

A high-temperature version of the Thunder moisture chip is available where process temperatures may be as high as 450°C as in the case of DIPS or ceramic glass-sealed packages. This is the Model BR-101B-SS-HT.

Calibration Methods

For calibration of the moisture chips after mounting in the hybrid and prior to sealing, Thunder Scientific provides calibration systems covering the full range of moisture from essentially zero to 100-percent RH.

The series 4000 provides a calibration accuracy guaranteed to be better than ± 0.5°C. We claim that typical accuracy is ± 0.2°C over a range of -75° to -8° dew point (1 to 3060 ppm_v).

The series 7000 covers an overlapping range from as low as 5-percent RH to 96-percent RH with an accuracy of better than ±0.5-percent RH. It contains a small chamber in a stable bath which will hold a considerable number of hybrids for calibration.

The series 6500 provides a large upright chamber where a larger volume may be required for quantity calibrations. The series 6500 covers the range from 5-percent RH to 96-percent RH with a guaranteed accuracy of better than ±1-percent RH.

The Model 4A-1 or 4AB-1 Humidity Standard

Where it is necessary to know precisely the RH in an air stream or chamber, the model 4A-1 or 4AB-1 standard is recommended.

This system will provide an accurate measurement to better than ±0.5-percent RH over a range of 1-percent RH or less to 99.9-percent RH.

SUMMARY

In review, the BR-101B-SS moisture chip provides a useful source for determination of the moisture content of hybrid assemblies during manufacture and final operation of these devices.

In addition, equipment for calibration of the moisture chip in hybrids has been delineated and typical accuracies stated.

The Thunder Scientific model BR-101B-SS moisture sensor is an advanced method for determining moisture level in hybrids in production requirements.

3.3 Hysteresis and Long-Term Repeatability of Aluminum Oxide Humidity Sensors

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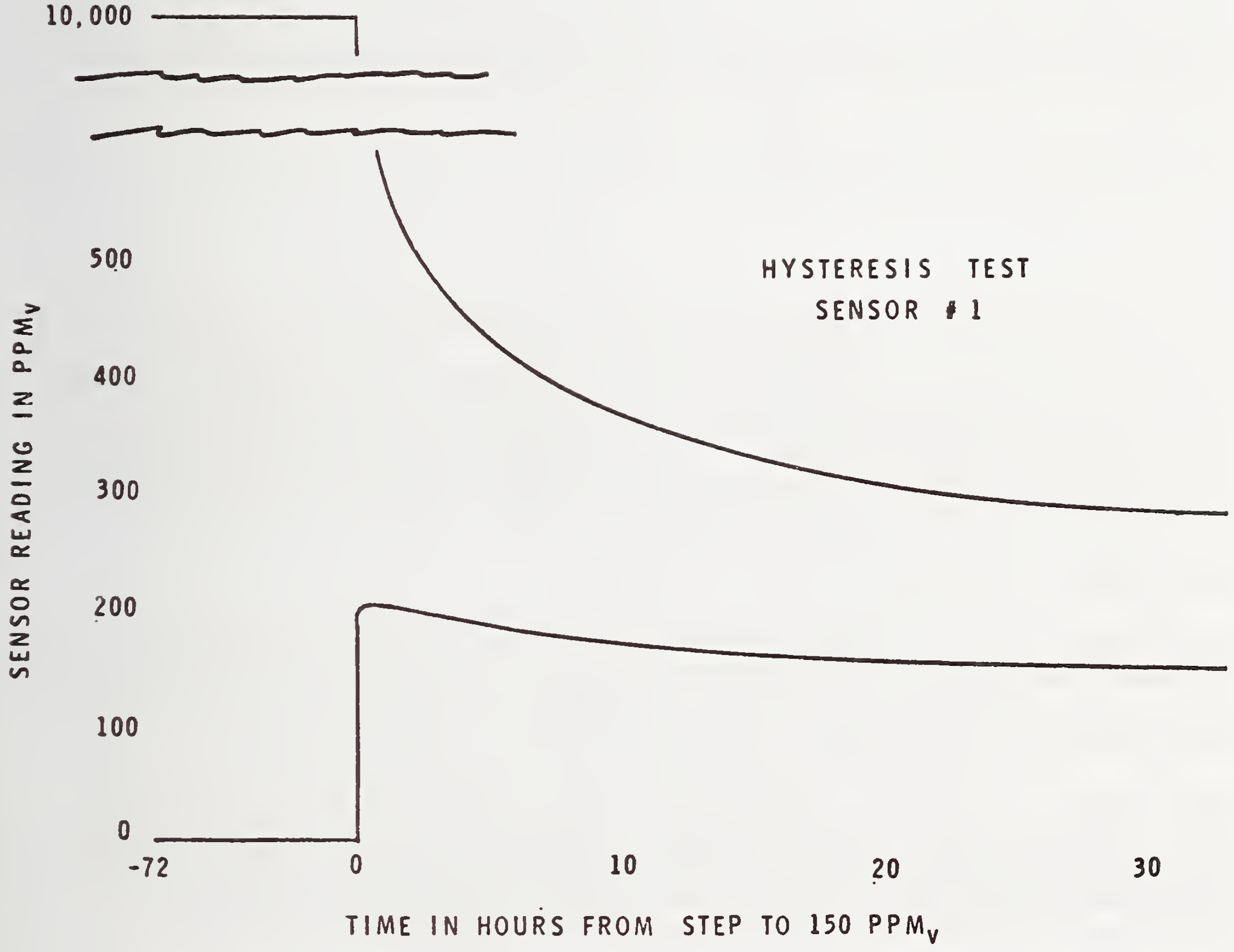
Aluminum oxide humidity sensors calibrated at Sandia Laboratories have all shown a hysteresis following water vapor pressure variations and/or temperature variations. To make the aluminum oxide hygrometer calibrations more useful for many measurement applications at Sandia, hysteresis and long-term repeatability measurements have been made.

Three sensors were tested for hysteresis by first stepping them from dry to 150 parts per million by volume ($\mathrm{ppm_v}$), then from 10 000 $\mathrm{ppm_v}$ to 150 $\mathrm{ppm_v}$. Typical response is shown in the figure. The typical worst case hysteresis was 360 $\mathrm{ppm_v}$ at 1 h and 130 $\mathrm{ppm_v}$ at 30 h.

The drift of each of the three sensors stored for six months at 10 000 $\mathrm{ppm_v}$ and at a constant temperature of 21.1 ± 1°C was approximately 20 $\mathrm{ppm_v}$ downward (to a lower humidity reading). However, when the sensors were exposed for 1 h to a humidity of 150 $\mathrm{ppm_v}$ and a temperature of 100°C, the 150-$\mathrm{ppm_v}$ room temperature calibration of each unit shifted downward approximately 100 $\mathrm{ppm_v}$.



Hysteresis of aluminum oxide humidity sensors.

3.4 Moisture Standards and Sensors*†

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The inclusion of the Internal Water-Vapor Content Test, Test Method 1018, into MIL-STD-883B required the development of moisture standards for the correlation and calibration of various laboratory mass spectrometry equipment in use throughout the United States and for subsequent DESC certification. The intention of standards is to provide a controlled and predictable moisture environment of three ranges of water vapor content inside a package. The packages were to contain an internal moisture sensor to measure the moisture content before gas analysis and provide for a means of traceability to an NBS standard measurement with a recalibration of the sensor after the analysis was completed. Several problems were encountered in development of the moisture standards. These problems along with the results of the three Round Robins of standards sent to various gas analysis labs will be discussed in this paper.

Processing

The development of a moisture standard called for changes in the normal processing steps used in the sealing of a package. The only component included in the package was an aluminum oxide moisture sensor which was die attached using a gold-tin preform at 280°C. The sensor was wire bonded using gold wire and ultrasonic techniques. The packages with the sensors were then vacuum baked at 125°C for 16 to 24 h and then hermetically sealed in nitrogen at a controlled level of water vapor.

Water Vapor Generation

Dry nitrogen gas is bubbled through a heated flask of DI water (≈80°C). The saturated nitrogen is mixed with dry nitrogen to the desired level in a reservoir whose pressure is 20 psi. The wet nitrogen gas is then introduced into the Research Instrument Corporation (RICO) solder sealer.

The water vapor content introduced into the package is monitored using an aluminum oxide moisture sensor mounted internally to the sealing chamber of the RICO. Figure 1 shows a graph of the sensor operation during the seal cycle *versus* the heater temperature monitored at the same time. The operation of the sealer cycle is outlined in figure 2.

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Predictability

For the moisture standards sealed in Round Robin I, a moisture sensor was mounted in the package using the processing steps described earlier. The packages were sealed in two moisture ranges of 5000 ppm and 10 000 ppm. The predicted moisture in the package was based on the value read using the aluminum oxide moisture sensor mounted in the RICO seal chamber. The moisture sensor in the seal chamber gives a reading of the sealing ambient water vapor content. This was assumed to be the amount of water vapor introduced into the package. The moisture sensors mounted internal to the standard package in Round Robin I were monitored to provide a reading of the actual internal standard package ambient. The readings recorded by these sensors showed that the actual water vapor content is considerably lower than the predicted value as shown in figure 3. This difference in water vapor content is attributed to package wall adsorption effects and to shifts in the calibration of the sensor inside the standard package.

Adsorption of moisture onto the walls of the package appears to be the most significant effect in developing a predictable moisture standard. Packages vacuum baked and subsequently sealed in a controlled wet environment of 10 000 ppm are found to contain 5000 to 6000 ppm when gas analyzed. The effect occurs in packages in volume ranges of 0.05 cm^3 and 0.2 cm^3. If the packages are not vacuum baked, the effect is considerably reduced.

Measurement of the internal moisture content of the standards was accomplished in Round Robin I using an aluminum oxide moisture sensor. Work with these sensors has indicated that the sensors exhibit a shift in calibration due to various processing techniques. The original calibration curve was supplied by the manufacturer. The processing steps affecting the sensors and the maximum shift due to these steps are shown in figure 4. It should be noted that the effects of room air storage and die attach on the sensor can be corrected by recalibrating the sensor before vacuum baking. This procedure would limit the worst case maximum shift to -4°C dew point. The overall effect of the adsorption of moisture on the package walls and the shift of the sensor is shown in figure 5, where the first shift from the predicted values of 10 000 ppm and 5000 ppm are due to the sensor shift and adsorption phenomenon and the exponential decrease is due to adsorption.

The aluminum oxide moisture sensors are temperature dependent, displaying a negative temperature coefficient. As the sensors are cooled, the sensor reading increases. Figure 6 shows a graph of a sensor calibrated at three different temperatures. Therefore, the sensor must be calibrated at use temperature.

Moisture Standards

In the three round robins completed for the correlation and calibration of various gas analysis labs, only Round I had an internal moisture sensor enclosed in the standard package. In Rounds II and III, the moisture in the package was predicted using the value recorded by the moisture sensor in the seal chamber. The sensors used in Round I were calibrated after opening to provide a more accurate reading of the standard package ambient seen before gas analysis.

Figure 7 shows the results of Round I. Of the four analytical labs participating, only one observed a reading close to the internal moisture sensor reading. The chart also shows the difference in the sensor readings after one to two weeks. This downward trend in moisture content is due to adsorption on the package walls. There was also a difference in readings due to the sensor shifting which was corrected for through sensor recalibration after opening the package. Applying the recalibration data to the last sensor reading gives a good indication of what the package ambient was at the time of gas analysis. It is the last sensor reading value that correlates with Lab C gas analysis.

The correction factors for the labs were based on the corrected moisture sensor readings and the moisture content the labs actually found. Since a moisture sensor was not mounted internal to the package in Rounds II and III, there is no cross check of the actual package ambient before analysis. The predicted values in Rounds II and III agree closely with two analysis labs, Labs C and D. The packages used in Round II were not vacuum baked out, thus lowering the effect of package wall adsorption. Packages used in Round III were vacuum baked and the effect of adsorption is seen in the difference in readings. Applying the correction factors from Round I to the labs' readings, the correct range of moisture is established in the package. Rounds II and III results are shown in figures 8 and 9, respectively.

From the experiments performed in the development of the moisture standards, it was found that moisture can be introduced into packages at controlled levels. Monitoring of the package ambient during and after sealing shows that two major factors are in play which affect the final moisture level in the package, these being aluminum oxide sensor shifts and package wall adsorption. Correction of the sensor reading after recalibration and comparison to gas analysis of the packages shows that a predictable moisture standard can be developed for correlation and calibration of mass spectrometry gas analyzers.

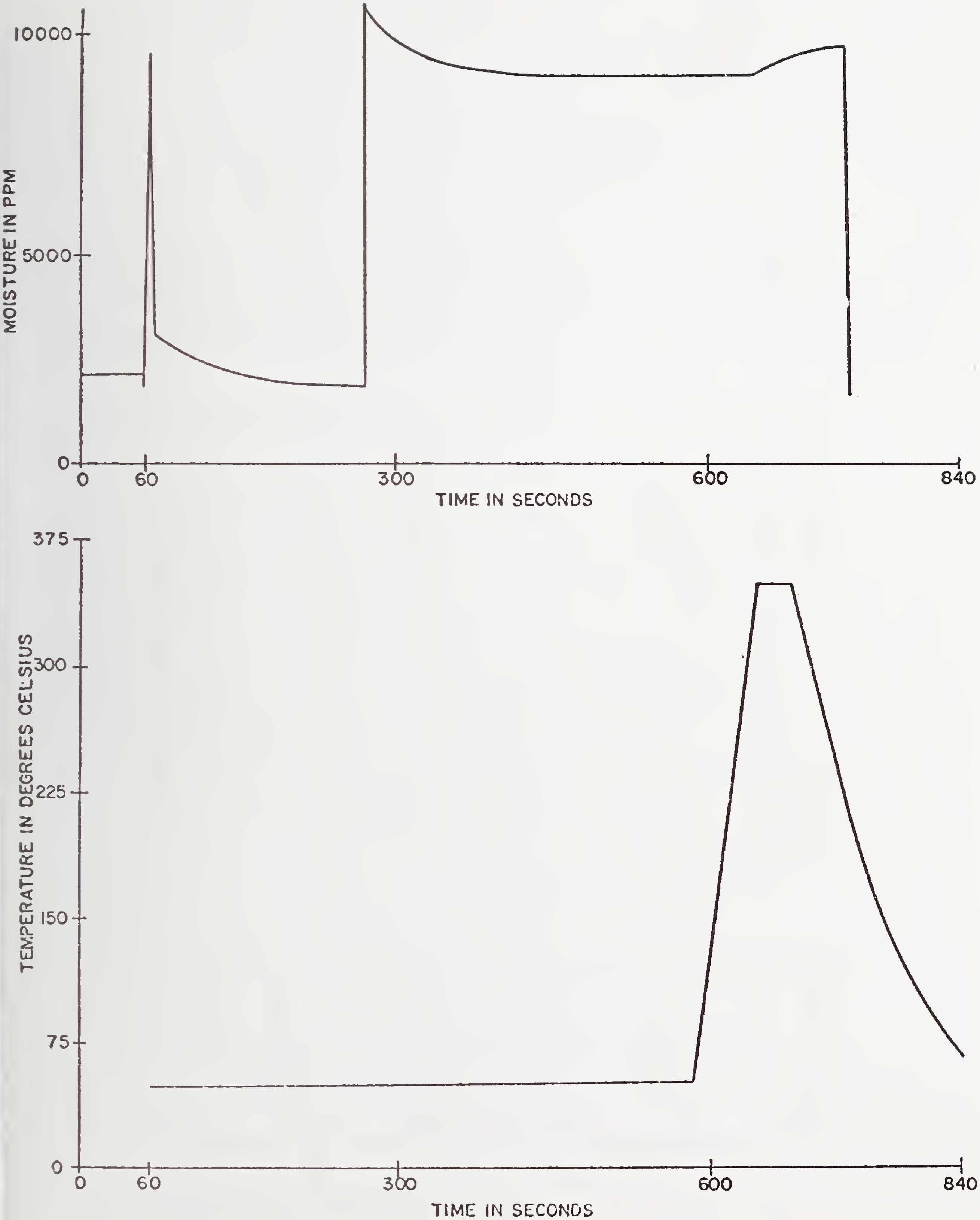

Figure 1. Sensor and sealer temperature profile.

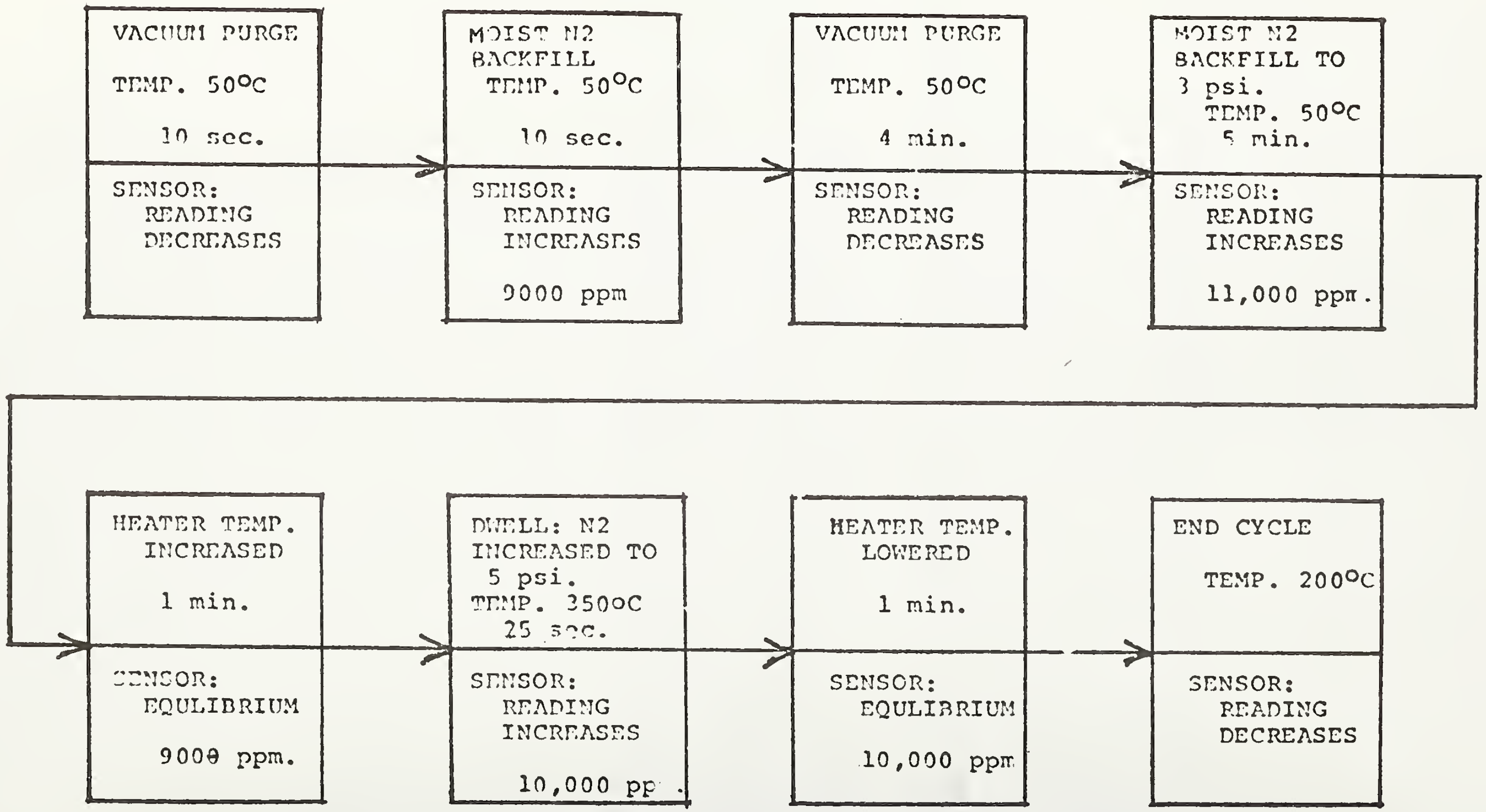

Figure 2. Sealer operation.

PACKAGE	PREDICTED	INTERNAL SENSOR 1 hr after seal
1	6700 ppm	4600 ppm
2	5300	2400
3	4200	1700
4	10700	5400
5	11800	4600
6	11800	5100
7	11800	3800
8	11800	5100
9	11800	3800
10	10700	4000
11	10700	4000

Figure 3. Moisture sensor reading *versus* predicted moisture content.

DRY N_2 STORAGE	NO CHANGE
ROOM AIR STORAGE	-3°C SHIFT
DIE ATTACH AT 280°C	-2°C SHIFT
VACUUM BAKE @125°C 16-24 hr	-2°C SHIFT
SEALING AND GAS ANALYSIS	-2°C SHIFT
TCE WASH	TEMPORARY RECOVERY

Figure 4. Processing effects on sensor.

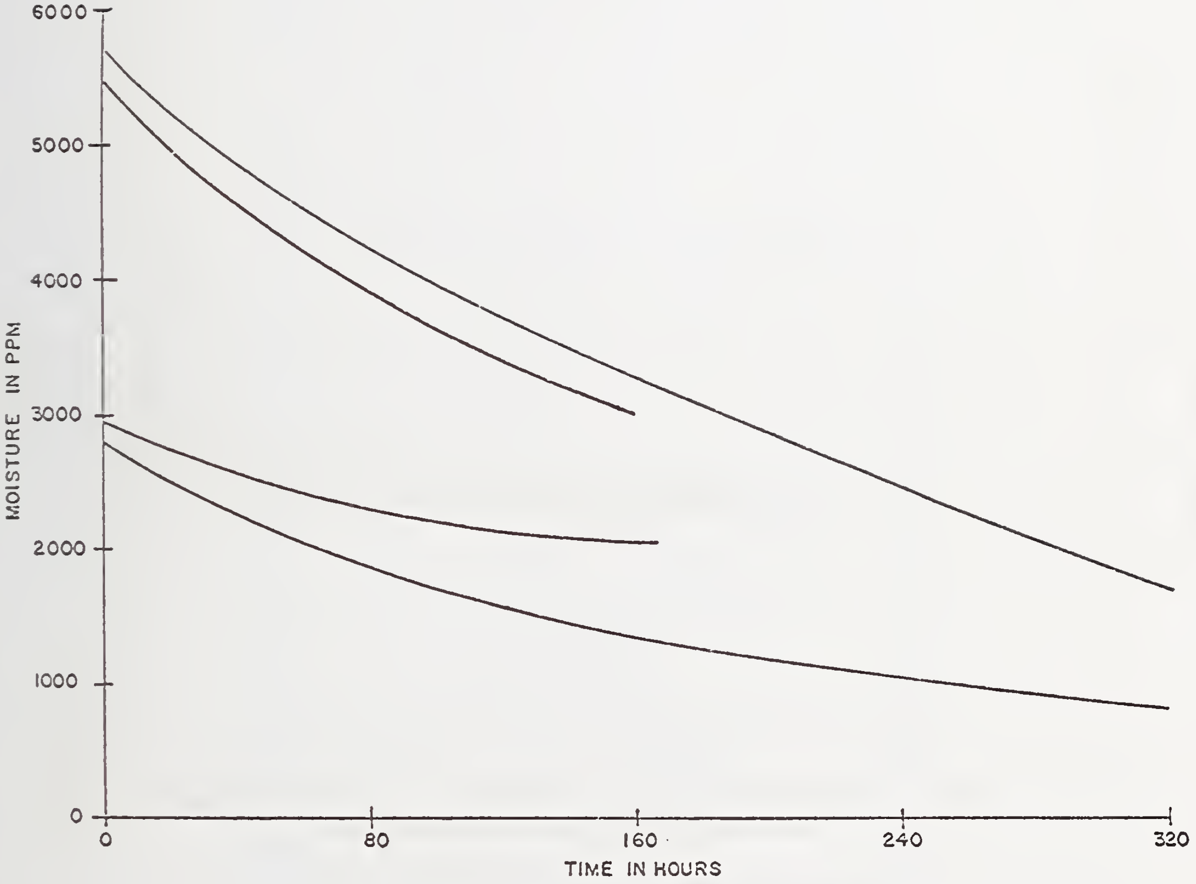

Figure 5. Adsorption effects on package ambient.

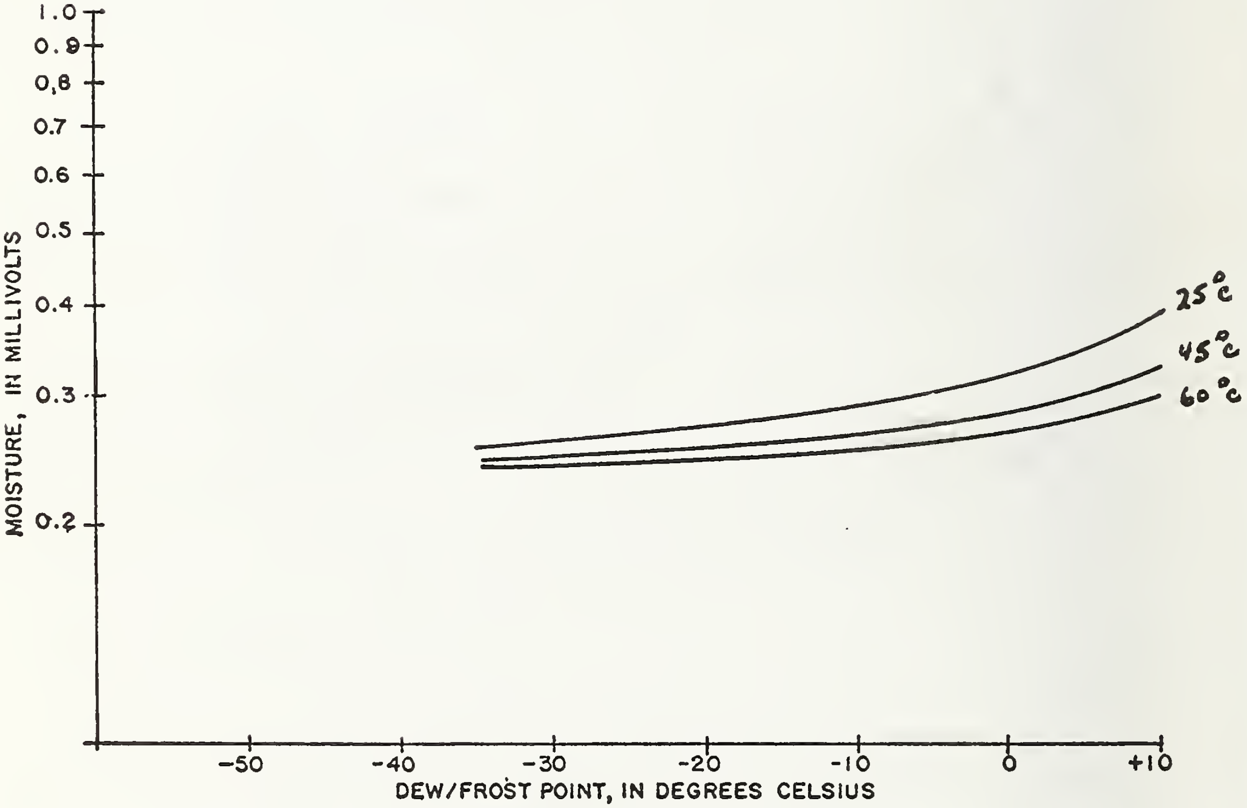

Figure 6. Temperature effects on sensors.

Vacuum Bake 89 Hrs 125°C

Package Sensor								Sensor Recalibration		Mass Spect Uncorrected				Mass Correction Factor			
Ser # Package	Scaler Sensor	2/12/78 @ Seal	2/12/78 2 hrs. after	2/13/78	2/17/78	2/21/78	2/24/78	Correction Factor	Corrected Reading	Lab A	Lab B	Lab C	Lab D	Lab A	Lab B	Lab C	Lab D
1	7200	3300	3800	leaker				1.26	leaker		-						
2	6000	2100	2100	1800		1300		1.27	1650				650				2.5
3	5000	2000	1800				900	1.36	1220	139				8.8			
4	11000	5000	4400					1.70			-						
5	13000	4700	3800	2200		2300		0.98	2250				1000				2.25
6	13000	5000	4200		2400			1.88	4510			5000				0.90	
7	13000	4100	3500		2600			1.70	4420			1800				2.45	
8	13000	4400	4100	2100		2000		-									-
9	13000	4200	3500				1600	1.87	2990	376				7.9			
10	11000	4400	3800				1700		3180		401				7.9		
11	11000	4200	3800				1700	1.80	3180	459				6.9			
													Ave C. F.	7.8	*	0.90	2.4

* One reading not sufficient to compute average used factor of 5 in Round II and III.

Figure 7. Round I results.

No Bake or Insitu · Sensor

Serial #	Sealing Sensor	Mass Spectrometer Reading				Round I Correction Factor				Corrected Reading
		Lab A	Lab B	Lab C	Lab D	Lab A	Lab B	Lab C	Lab D	
8	5100				New Tech 6000				**	6000
9	5600			4500				0.9		4000
10	5800		1204				5.0			6000
11	5800	872				7.8				6800
12	5600				4000				**	4000
13	5600			4900				0.9		4400
14	5600		1365				5.0			6800
15	5500	667				7.8				5200
16	5400				7000				**	7000
17	5400			5000				0.9		4500
18	5200		1025				5.0			5100
19	5200	534				7.8				4100
										4000 - 7000 5500±1500 Spread ± 27%

** Between Round I and Round II, Lab D Technique changed to optimize accuracy at 5000 ppm.

Figure 8. Round II results.

Vacuum Bake   No Sensor

Serial #	Corrected to 3 psig Seal Sensor	Mass Spectrometer Reading				Round I Correction Factor				Corrected Reading
		Lab A	Lab B	Lab C	Lab D	Lab A	Lab B	Lab C	Lab D	
16	14200		560				5.0			2800
17	11000			5000				0.9		4500
18	13000									
19	11000				5000				1.0	5000
20	11000		681				5.0			3400
21	11000			4300				0.9		3900
22	11000	600				7.8				4700
23	Leaker									
24	9600		1029				5.0			5100
25	9400			6800				0.9		6100
26	9600	241				7.8				1900
27	8700				4000				1.0	4000
								Spread		1900 - 6100 4000 ± 2100 ± 52%

Figure 9. Round III results.

3.5 Assessment of the Use of Measurement of Surface Conductivity as a Means of Determining Moisture Content of Hermetic Semiconductor Encapsulations

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INTRODUCTION

The one failure mode which cannot be combatted by any means other than reducing the water content of the internal atmosphere is electrochemical corrosion of aluminium metallization. As this depends on moisture-induced surface conductivity, a small sensor which would permit sensitive measurement of surface conductivity could provide a useful means of monitoring, or approving, a sealing process. Two types of sensors which can be made using conventional silicon technology are considered here. Their performance is gauged in terms of the corrosion rate expected when packages have a water content on the limit of detection using the sensor.

Surface Conductivity of Silicon Dioxide

Koelmans [1] used an interdigital structure to measure the surface conductivity of silicon dioxide as a function of the relative humidity and temperature. We have confirmed his results. The measured conductivity can be described by the equation:

$$\sigma = \sigma_o \exp\left(- \frac{\varepsilon}{R \cdot T}\right) , \tag{1}$$

where T is the temperature while, as figures 1 and 2 show, ε and σ_o are parameters which depend on the relative humidity. Koelmans [1] noted that the conductivity appeared to be consistent with the model used by Kawasaki and Hackerman [2] to describe the conductivity of porous silicon. They showed that σ was a function of the amount, m, of water adsorbed:

$$\sigma \propto m \cdot \exp\left(- \varepsilon_o \cdot \frac{(1 - \beta \cdot m)}{R \cdot T}\right) . \tag{2}$$

At low relative humidities ε_o and β are constants. Equation (2) reduces to eq (1) if m is expressed as a function of relative humidity. These two quantities are related by the adsorption isotherm which is likely to be described by the BET equation:

$$m = \frac{m_o \cdot c \cdot r}{(1 - r)(1 + [c - 1]r)} \tag{3}$$

where $c = \exp(\phi/R \cdot T)$, r is the relative humidity and m_o is a constant. ϕ is the difference between the latent heat of evaporation of water and the heat of adsorption of this vapour. If with hindsight c is assumed to be approximately three, then the simple expression for m, which is given below, is accurate to within 11 percent over the range $0.1 < r < 0.6$:

$$m \approx m_o \cdot c \cdot r \; . \qquad (4)$$

Over a range of temperature about a mean, T_o, the value of m is given by the equation:

$$m \approx m_o \cdot r \left(1 - \frac{\phi \cdot T}{R \cdot T_o^2} + \frac{\phi}{R \cdot T_o}\right) . \qquad (5)$$

If this value of m is substituted into eq (2), it is found that:

$$\sigma_o \propto r \cdot m_o \cdot \exp\left(- \frac{\varepsilon_o}{R \cdot T} + \frac{\phi}{R \cdot T} \cdot \beta \cdot r \cdot m_o \cdot \exp \frac{\phi}{R \cdot T_o}\right), \text{ and} \qquad (6)$$

$$\varepsilon = \varepsilon_o \cdot \left(1 - \beta \cdot r \cdot m_o \cdot \left(1 + \frac{\phi}{R \cdot T_o}\right) \cdot \exp \frac{\phi}{R \cdot T_o}\right) - \phi \; . \qquad (7)$$

Over the range $0.1 < r < 0.7$ the results presented in figures 1 and 2 can be described by the equations:

$$\sigma_o = r \cdot 2 \times 10^{-3} \cdot \exp(-11.5 \cdot r) \quad (\Omega/\square)^{-1}, \text{ and} \qquad (8)$$

$$\varepsilon = 19 - 13.6\ r \text{ kcals/gram.mole.} \qquad (9)$$

Comparison of eqs (6) and (7) with (8) and (9) yields a value of 925 cals/mole for ϕ, a value near the 1.5 kcals/mole needed to account for the adsorption isotherm measured by Kawasaki and Hackerman [2]. The value of ϕ can be used to estimate the range of validity of the approximation on which eq (5) is based. It can be shown that it is accurate to within 10 percent if the temperature is within 70°C of the mean of the range of temperatures over which the conductivity was measured (30 to 100°C).

The values of σ_o derived from Koelmans' and Sim's results had the same dependence on relative humidity, but they differed by an order of magnitude. The higher value (Koelmans) is used in the calculations presented here.

The dependence of the corrosion rate on current is complicated by two processes not yet mentioned. The current flowing between aluminium electrodes was a decreasing function of time. Koelmans attributed this behaviour to interfacial polarisation of one or both of the electrodes. A field effect structure, which formed the basis of the sensor to be discussed shortly, was used to monitor the voltage of the oxide adjacent to the anode. It showed that the majority of the polarisation was due to the applied voltage being dropped at the edge of the anode. As this voltage drop disappeared, immediately the current ceased to flow. The polarisation is likely to be due to the formation of an insulating film on the anode. The film will reduce the corrosion rate because it lowers the current, but the possibility of it breaking down under certain conditions, such as C1 contamination [1], cannot be ignored. Thus in calculating corrosion rates, we must use the current which flows before there is a significant amount of polarisation. The other complicating factor is localisation of corrosion. As some of the failures observed during corrosion tests have occurred because of thinning at the grain boundaries, there is no doubt that the corrosion can be localised. Unfortunately, it is not yet possible to build this into the calculations of corrosion rates; the error introduced will be at least partly offset by ignoring the decrease in current due to polarisation.

In the worst case, the passage of one electron will result in the corrosion of one aluminium atom. The time taken to consume half a 10-μm wide aluminium track of thickness 1 μm, which is separated by 10 μm from another whose potential is 10 V higher, is then:

$$t_f = \frac{1.35 \times 10^{-11}}{\sigma} \text{ hours} . \tag{10}$$

The value of t_f can be deduced using eq (8) and (9). Figure 3 shows the value of t_f plotted against the relative humidity at 25°C. The high relative humidity end of this plot is in rough agreement with service experience. MOS circuits, which were subsequently shown to be in packages having an internal atmosphere with a relative humidity of about 0.7, failed within several months of entering service. These calculations indicate that relative humidities of less than 0.1 at 25°C do not present a significant reliability hazard. Such levels of moisture can be detected by measuring the surface conductivity.

Measurement of Water Content Using an In-Package Sensor

The sensitivity of any in-package sensor will be affected by the amount of water adsorbed by the walls of the package. The calculations presented here are based on the assumption that the adsorption can be described by a BET isotherm having ϕ equal to 1.5 kcals/gram mole. The choice of a value of ϕ is not very critical since it exerts little influence on the amount of water adsorbed at the high relative humidities produced by lowering the temperature of the package. Figure 4 shows the calculated relationship between the temperature and the relative humidities in two types of packages. (The surface roughness of the metal TO-5 package was taken to be 3 and that of the ceramic package 10.) The relative humidity in the packages never reached saturation, but if there had been no adsorption, saturation would have occurred at the temperatures defined by the crosses marked on the plots. Figure 4, in conjunction with the equations for surface conductivity, can be used to determine the temperature dependence of the conductivity of an encapsulated silicon element.

Figure 5 shows the temperature dependence of the calculated leakage current of a large interdigital structure encapsulated in a ceramic dual-in-line package. (This package is one of the worst cases to consider because its large surface-area-to-volume ratio prevents high relative humidities from being reached when the temperature is lowered.) The leakage current goes through a shallow maximum as the temperature is reduced so the effect of the increasing relative humidity on the surface conductivity more than compensates for the reduction in temperature, a result which will be familiar to all users of the MIL SPEC dew point test.

Figure 5 shows that the leakage current is not very sensitive to temperature when the water content of a package is small so the operating temperature would not appear to be critical. However, the validity of the conductivity equations have not been proved for relative humidities below 0.1, so it is desirable to raise the relative humidity by lowering the temperature. Measurements on interdigital structures encapsulated in TO-5 packages showed a sharp drop in conductivity when the temperature was less than about -20°C. As this drop is not predicted by the conductivity equation, the sensor should not be used below -20°C.

As the bulk leakage current was always less than 10^{-13} A, the interdigital structure should be capable of detecting the presence of 3×10^{-4} mbar.1 of water in a CERDIP. Reference to figures 4 and 6 will show that this amount of water would produce a relative humidity of 0.2 in the package at 25°C and that the corrosion time, t_f, would be approximately 10 years. Similar calculations can be performed to determine the sensitivity of a test for moisture content which relies on naturally occurring surface leakage paths on the interior of the package or on the semiconductor chip. Our experience is that it is very rare to find that the sensitivity is comparable with that of the interdigital structure. One of the exceptions is a microwave transistor whose base emitter fingers form an ideal structure.

The size of the interdigital structure limits its usefulness, its 40 500-μm long fingers can only be accommodated on a chip about 1 mm square. Thus, it is not practical to consider adding the sensor to a process validation module. Fortunately a smaller and more sensitive sensor can be made using a field effect element to detect the charging of the surface of the oxide surrounding a biased electrode. The structure is similar to that used by Schlegel *et al.* [3] in their studies of surface conductivity. The sensor and the time dependence of the potential of the oxide are illustrated in figure 6. The time, t_d, which elapses between the application of the bias and the end of the field effect structure being turned on is a solution of eq (3):

$$V_t = V_o \operatorname{erfc} \left(\frac{x^2 \cdot C}{4 \cdot \sigma \cdot t_b} \right)^{1/2} , \tag{11}$$

where V_t is the turn-on voltage (23 V), V_o is the bias voltage (90 V), x is the distance between the electrode and the edge of the field effect structure (30 μm), and C is the capacitance per unit area of the thermally grown oxide (70 uF/m^2). The figures in brackets refer to a structure used in our experiments for which:

$$t_b = \frac{2.4 \times 10^{-4}}{\sigma} \text{ seconds.} \tag{12}$$

Figure 7 shows the calculated turn-on time of a sensor encapsulated in a CERDIP package containing various quantities of water. This type of graph can be used to calculate the sensitivity of the sensor when encapsulated in various types of packages. If the sensor does not turn on within 10^5 s of applying bias when the temperature is -10°C, the water content will be less than the figures given in the table. The water content is expressed in terms of the relative humidity the water would produce at 25°C and in terms of the dew point (which could only be measured if the package had nonadsorbent walls). Reference to figure 3 will show that the sensor should be able to provide adequate protection against corrosion. It is also worth noting that the sensitivity of the sensor will be increased if the surface conductivity, and hence the corrosion rate, is enhanced by ionic contamination.

Amount of Water Which Can be Detected by the Field Effect Sensor.

Type of package	Amount of Water		
	mbar.1	dew point °C	relative humidity at 25°C
TO-5	1.3×10^{-4}	-19	2%
CERDIP	1×10^{-4}	- 4	5%

Field effect sensors having various geometries have been exposed to relative humidities in the range 0.3 to 0.85 at 85°C, and eq (11) has been shown to provide an adequate description of their behaviour. In addition a small number of sensors have been encapsulated in TO-5 packages and the temperature dependence of the turn-on time shown to be in reasonable agreement with the model presented here.

Use of the Sensor

It is envisaged that the sensor could be used to detect a deterioration in a sealing process or to approve a sealing process. An example of what happens when a sealing process goes "out of tune" is illustrated by figure 8 which shows the water content in low profile TO-5 packages produced by one manufacturer plotted against the date of sealing. Packages sealed during weeks 31 and 32 of 1972 had an abnormally high water content — it was high enough to cause rapid failure due to electrochemical corrosion of the aluminium metallisation. Had the field effect sensor been used to monitor the process at least once a week, the deterioration would have been detected before the components entered service.

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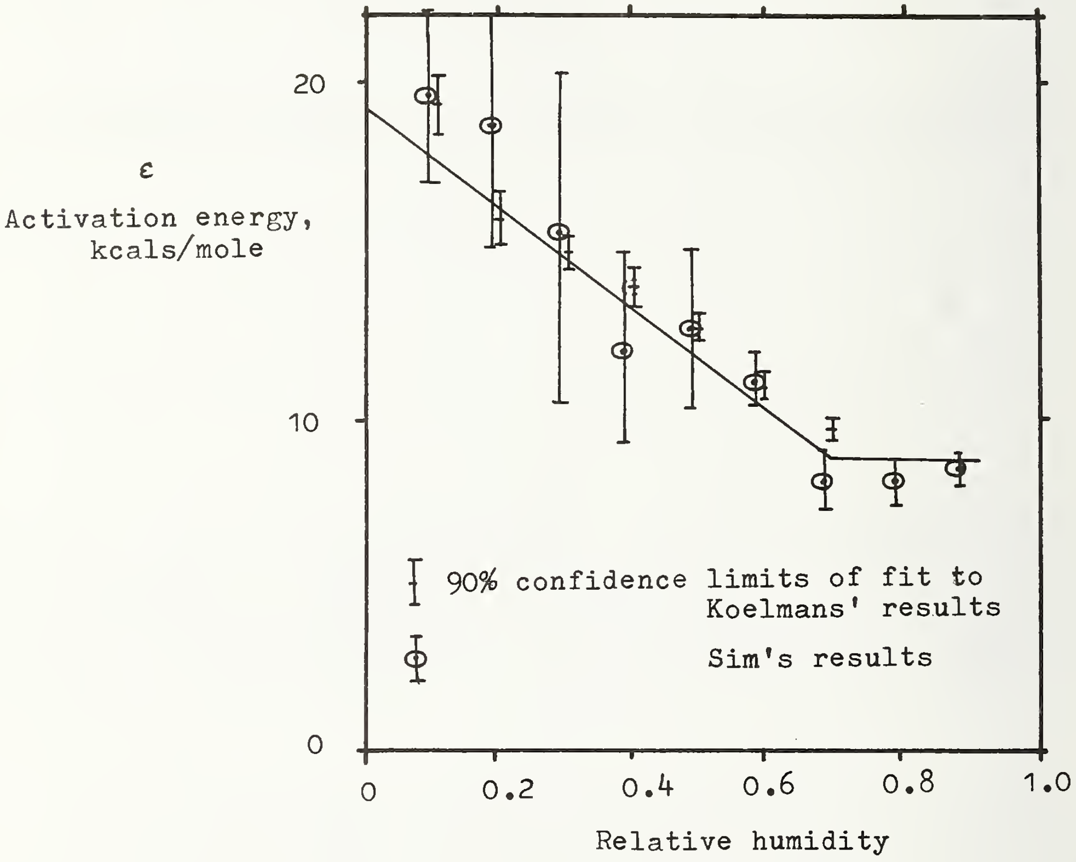

Figure 1. Dependence of the activation energy on the relative humidity.

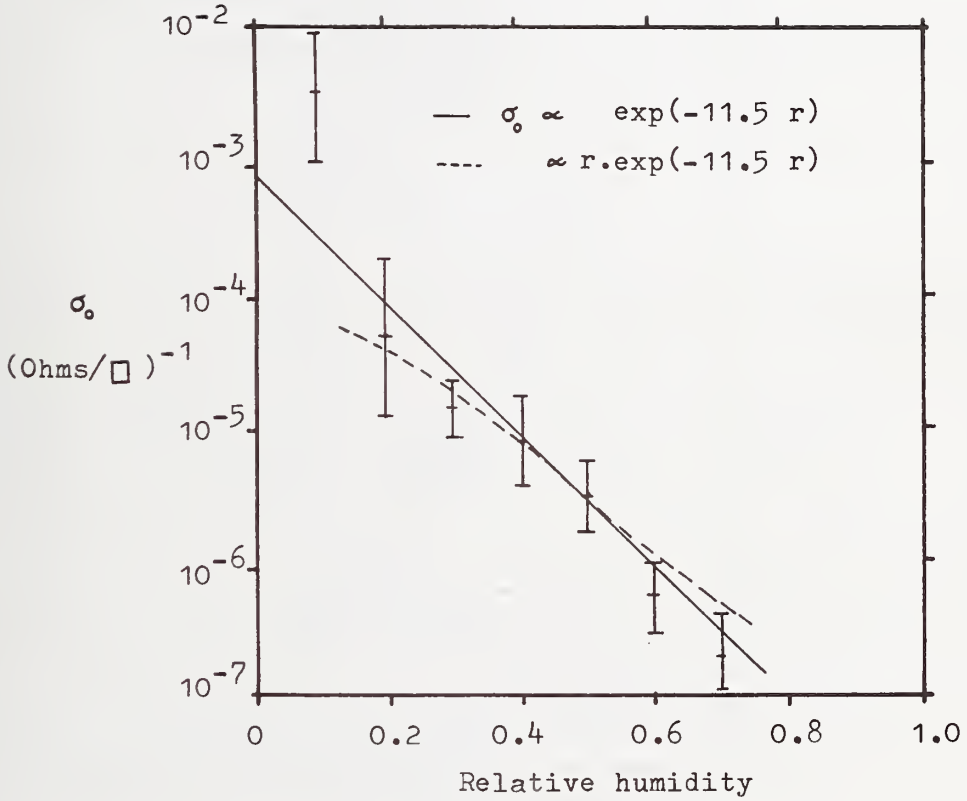

Figure 2. Dependence of σ_o on the relative humidity.

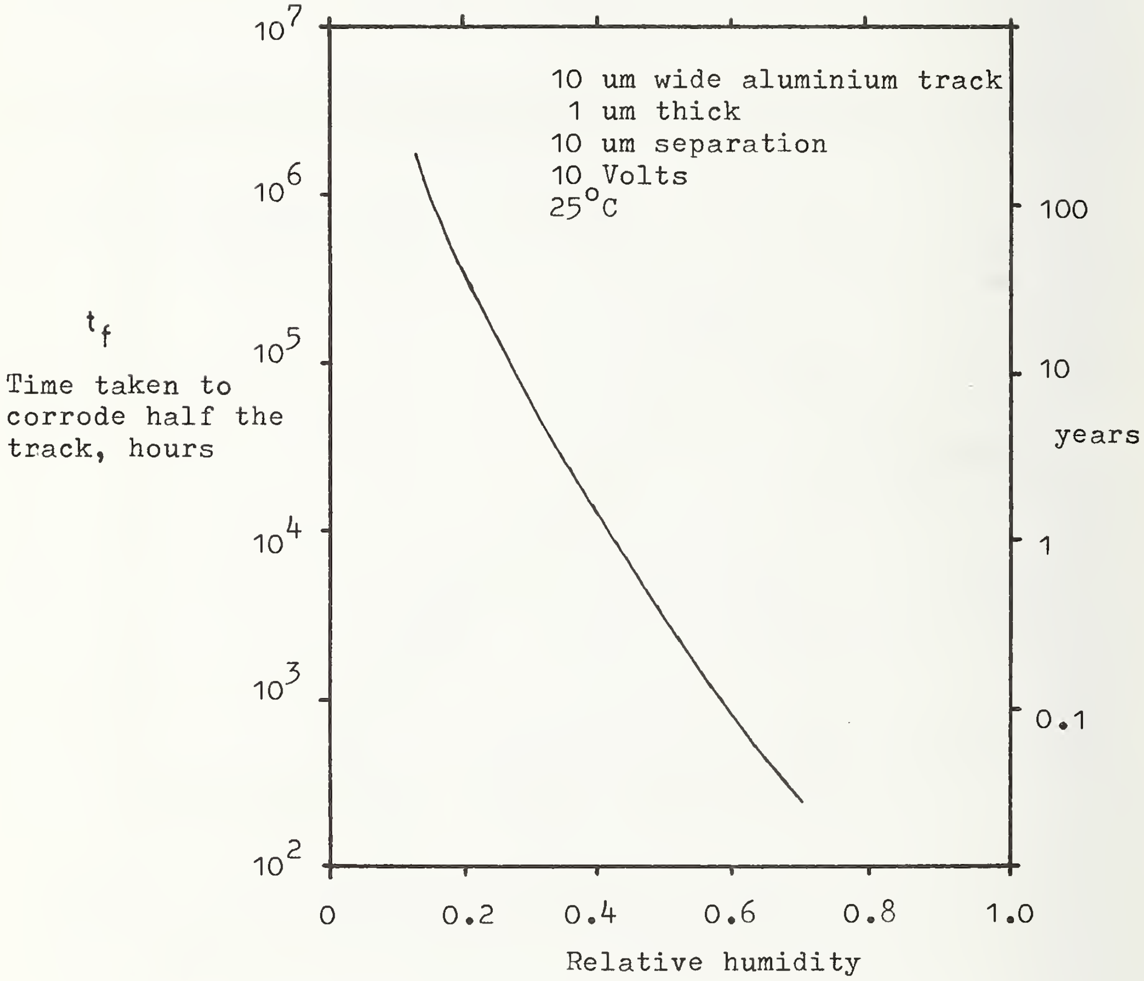

Figure 3. Time taken to corrode half an aluminium track.

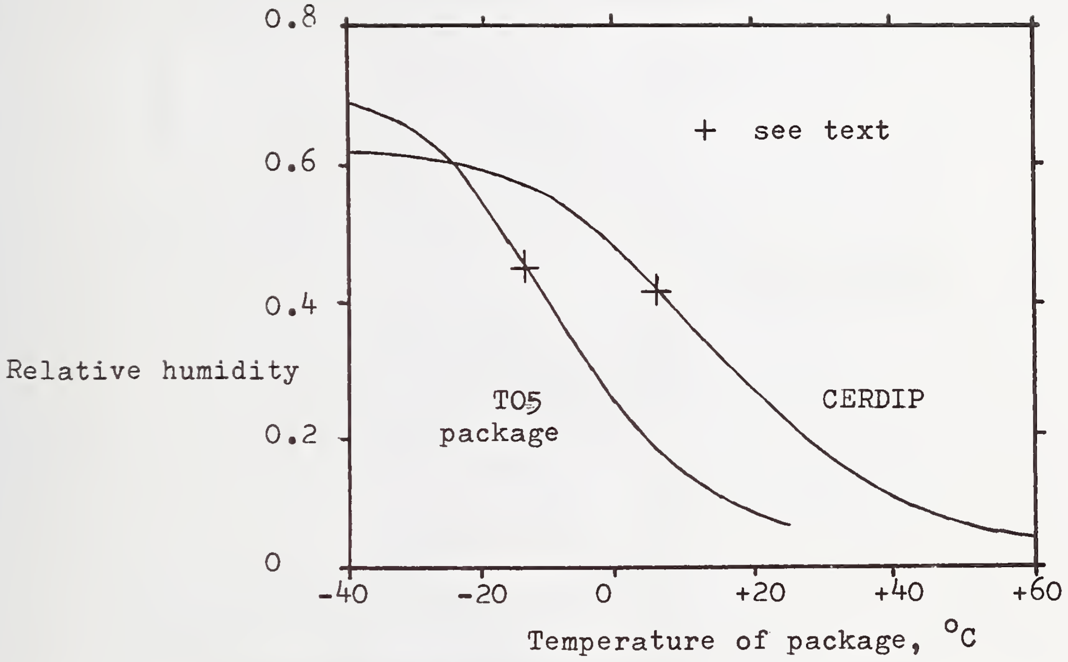

Figure 4. Temperature dependence of the relative humidity inside packages containing 3 x 10^{-4} mbar. l of water.

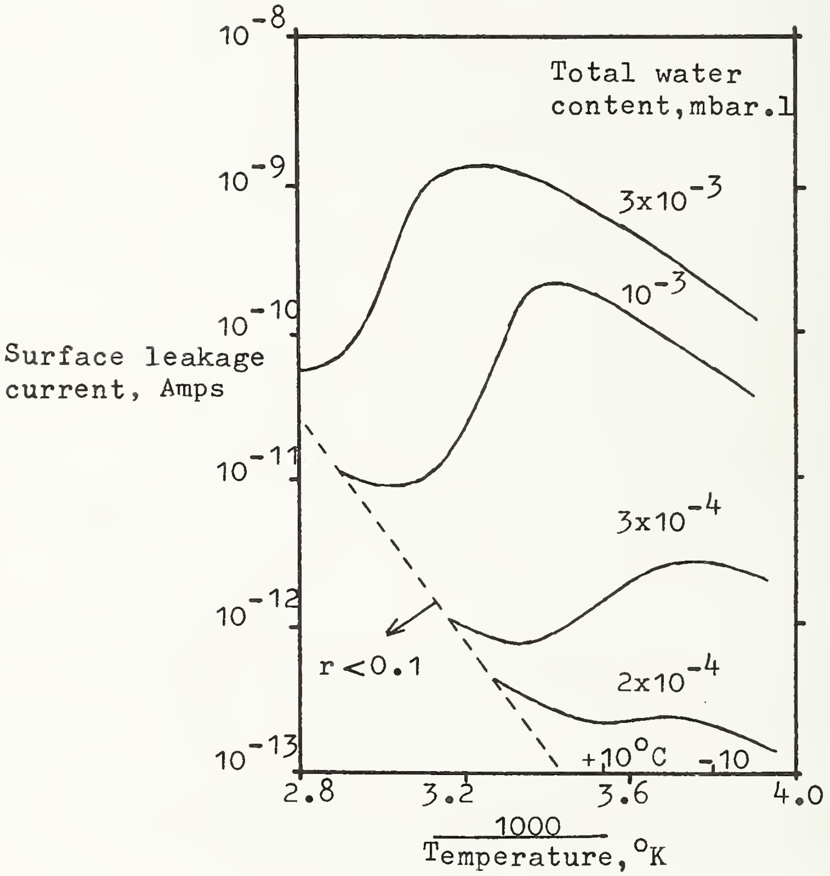

Figure 5. Calculated surface leakage current of an interdigital structure in a CERDIP.

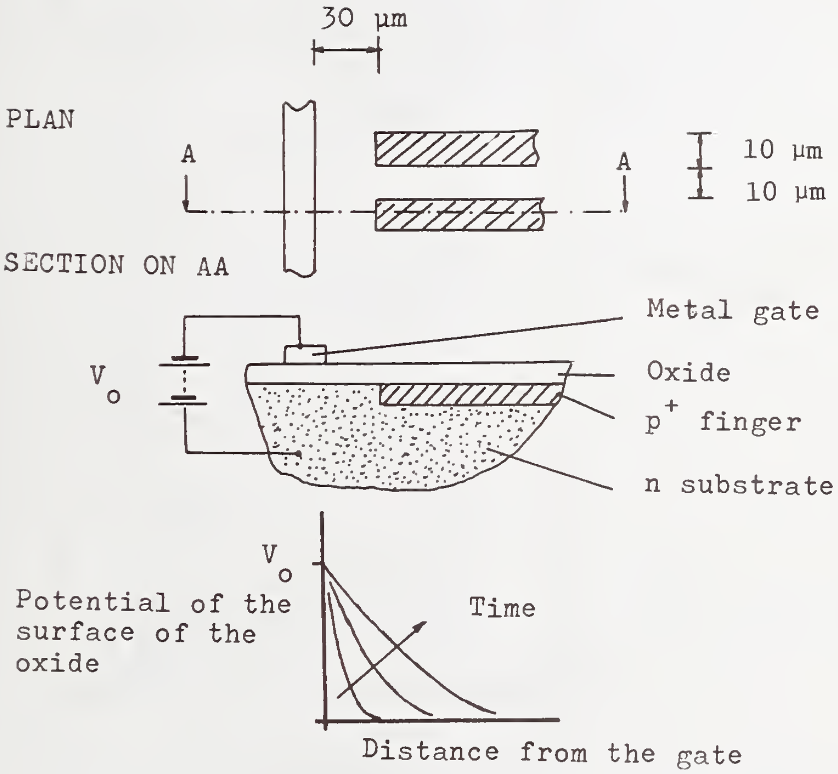

Figure 6. The field effect sensor.

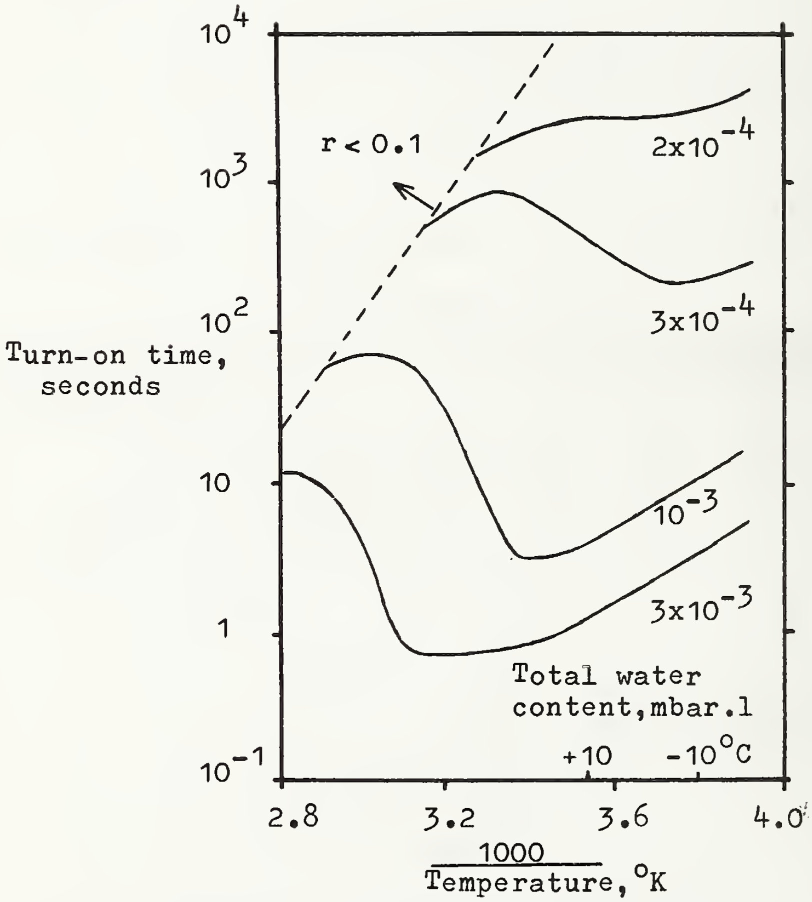

Figure 7. Calculated turn-on time of a field effect sensor in a CERDIP.

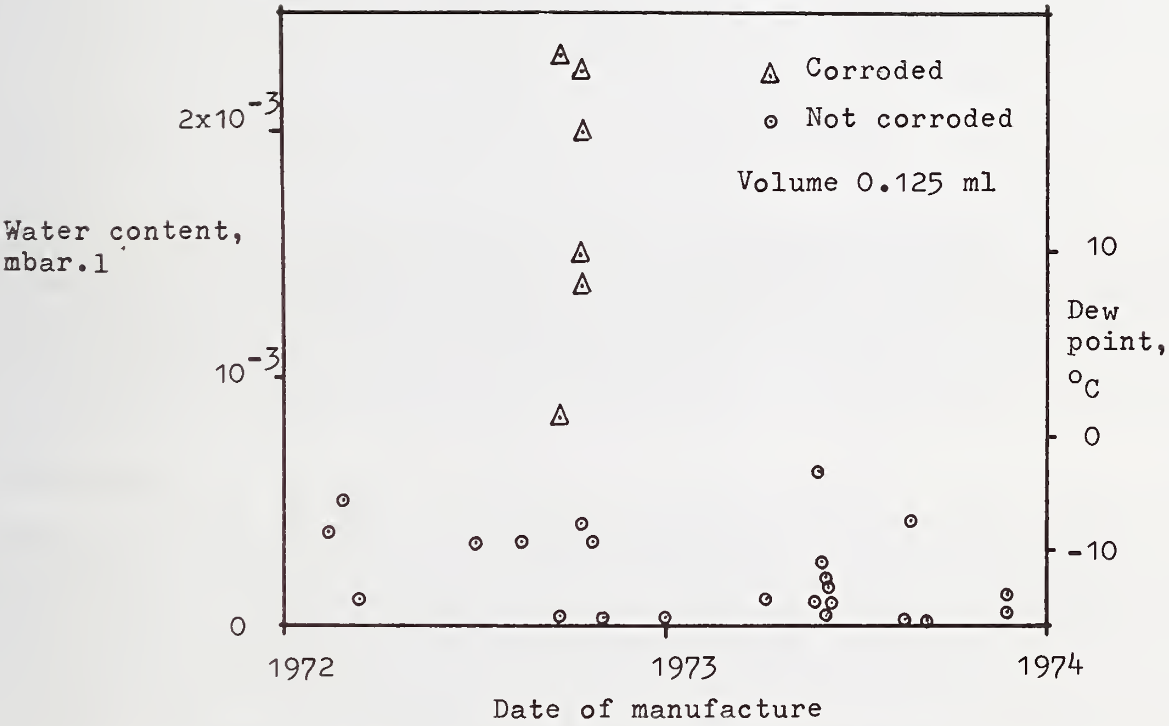

Figure 8. Water content of packages produced by the same manufacturer.

3.6 Moisture Sensing With the Charge-Flow Transistor*

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INTRODUCTION

The charge-flow transistor (CFT) was developed to provide an MOS-compatible method for monitoring the sheet conductance of gas- or moisture-sensitive thin films [1,2]. The principle of operation of the device is most easily described with reference to a simpler structure, the "charge-flow capacitor" (fig. 1). The substrate is conducting, and the two portions of the upper electrode are presumed to be connected together. The thin film of resistive material (called the polymer in fig. 1) is located within the gap in the upper electrode. When a voltage is suddenly applied between the electrode and substrate, the capacitor charges in two steps. First, the electrodes charge very quickly to the applied voltage, yielding the situation depicted in figure 1(b). Second, charge gradually flows through the resistive polymer film (fig. 1(c)) until, finally, a state is reached (fig. 1(d)) where the polymer-film portion of the capacitor is uniformly charged to the applied voltage. The dynamics of the charging process resemble the dynamics of an RC transmission line, with the resistive portion being the sheet resistance of the polymer film, and the capacitive portion being the polymer-insulator-substrate capacitance.

The charge-flow transistor (shown in cross section in fig. 2) incorporates a charge-flow capacitor into the gate structure of a MOSFET. For an enhancement-mode device, the dynamics of turn-on lead to a time delay between the application of a gate-to-source voltage and the appearance of a complete conducting channel between drain and source. Because this time delay depends on the sheet resistance of the polymer film, the turn-on delay serves to sense any variable that causes a variation in that sheet resistance. This paper presents some turn-on delay and static-characteristic measurements for CFTs that incorporate the moisture-sensitive polymer poly (p-aminophenylacetylene), abbreviated PAPA.

We have fabricated p-channel aluminum-gate enhancement-mode CFTs with a variety of gate-oxide thicknesses and substrate dopings [2,3,4]. Typical device dimensions are a channel width of 10 mils, a total channel length L of 2 mils, and gate gap widths W that vary from 0.25 mils to 1.5 mils. Once fabricated, the devices are spin coated with a thin-film of PAPA (thickness $\sim$100 nm). Independent measurements of the variation of sheet conductance of PAPA films with moisture show a very strong dependence, nearly exponential. It was expected, therefore, that CFTs coated with PAPA would exhibit turn-on delays that also depended strongly on exposure to moisture. This paper reports some preliminary results of our experiments.

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* Work supported by the Office of Naval Research under Contract N00014-77-C-0361.

Static Characteristics

A very interesting result was obtained when we examined the static characteristics of the CFTs, that is, the current-*versus*-voltage characteristics of the device after the completion of the transient. These results are summarized in figure 3. When operated in the saturated or pinched-off region, a normal MOSFET exhibits a quadratic dependence of drain current on gate voltage. This dependence is usually demonstrated in graphs of the square root of drain current *versus* gate voltage. The typical plot is linear, and extrapolates to a threshold voltage. Figure 3 shows a comparison of the behavior of a normal MOSFET with six CFTs, with gap widths that increase from 0.25 mil (device 1) to 1.5 mils (device 6) in quarter-mil steps. The data for the metal gate MOSFET extrapolate to a threshold voltage of about -9 V. For the CFTs well above threshold, an approximately linear behavior of $\sqrt{I}$ on V is also observed, but the CFT threshold voltage increases with increasing gap width to a maximum value of about -16 V (extrapolated threshold voltage for devices 5 and 6). This increase in threshold voltage with gap width is believed to arise from the perturbation of the energy levels within the polymer by the metal-polymer contact. At gap widths above about 1 mil, the threshold voltage is determined by the polymer-semiconductor work-function difference, but at smaller gap widths, the presence of the aluminum gate causes a shift in the effective work function of the polymer. These results represent an unusual way to examine the effect of contacts between metals and near-insulators. This technique may also turn out to be useful for examining the effect of metal-surface contacts, as in the oxide-only MOSFET moisture-sensing devices described by Merrett and Sim [5]. The startling result of our data on PAPA is that contact effects seem to propagate 0.5 mil, or 12 μm, into the polymer, a surprisingly large distance.

Moisture Dependence of Turn-On

We examine now some turn-on data typical of CFTs coated with PAPA. Figure 4 shows turn-on delay times at room temperature for four different moisture levels. Although the data are sparse, the apparent exponential dependence of turn-on delay time *versus* relative humidity is typical of our CFTs. This device, however, is the first one on which we have observed delay-time variations over five orders of magnitude.

As the data suggest, the CFT is a very sensitive device. It remains to be seen whether further experiments will demonstrate sufficient long-term stability and reproducibility to make this a genuinely useful moisture-measuring technique. Nevertheless, these preliminary results are very promising, and point a clear direction for future efforts.

ACKNOWLEDGMENT

The devices reported on here were fabricated in the Microelectronics Facility of the MIT Center for Materials Science and Engineering. This Facility is supervised by Professor Clifton Fonstad, who supplied many suggestions and criticisms. Masks for the devices were made with the kind assistance of Dr. Dan Smythe of the MIT Lincoln Laboratory. The polymer material was supplied by NASA/Lewis Research Center, under grant NSG-3061.

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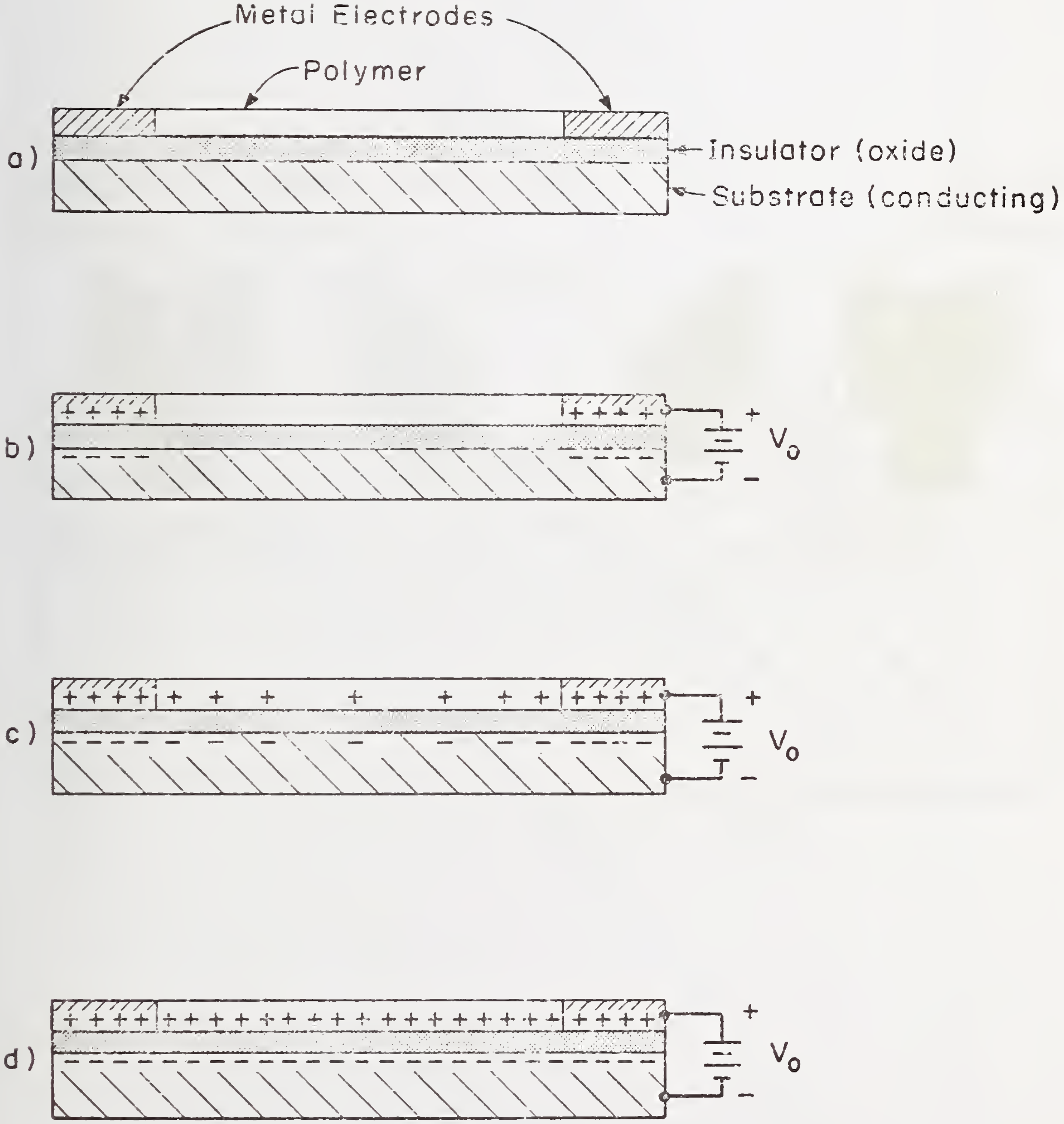

Figure 1. The charge-flow capacitor.

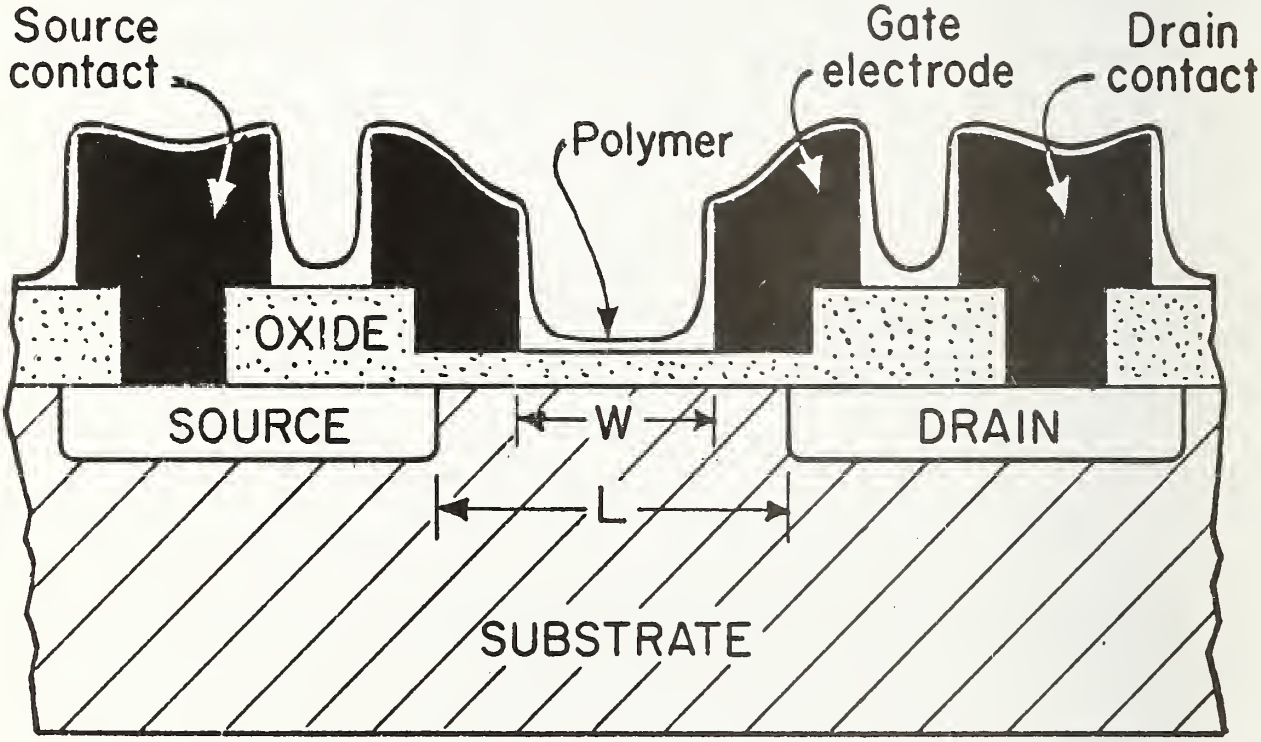

Figure 2. The charge-flow transistor.

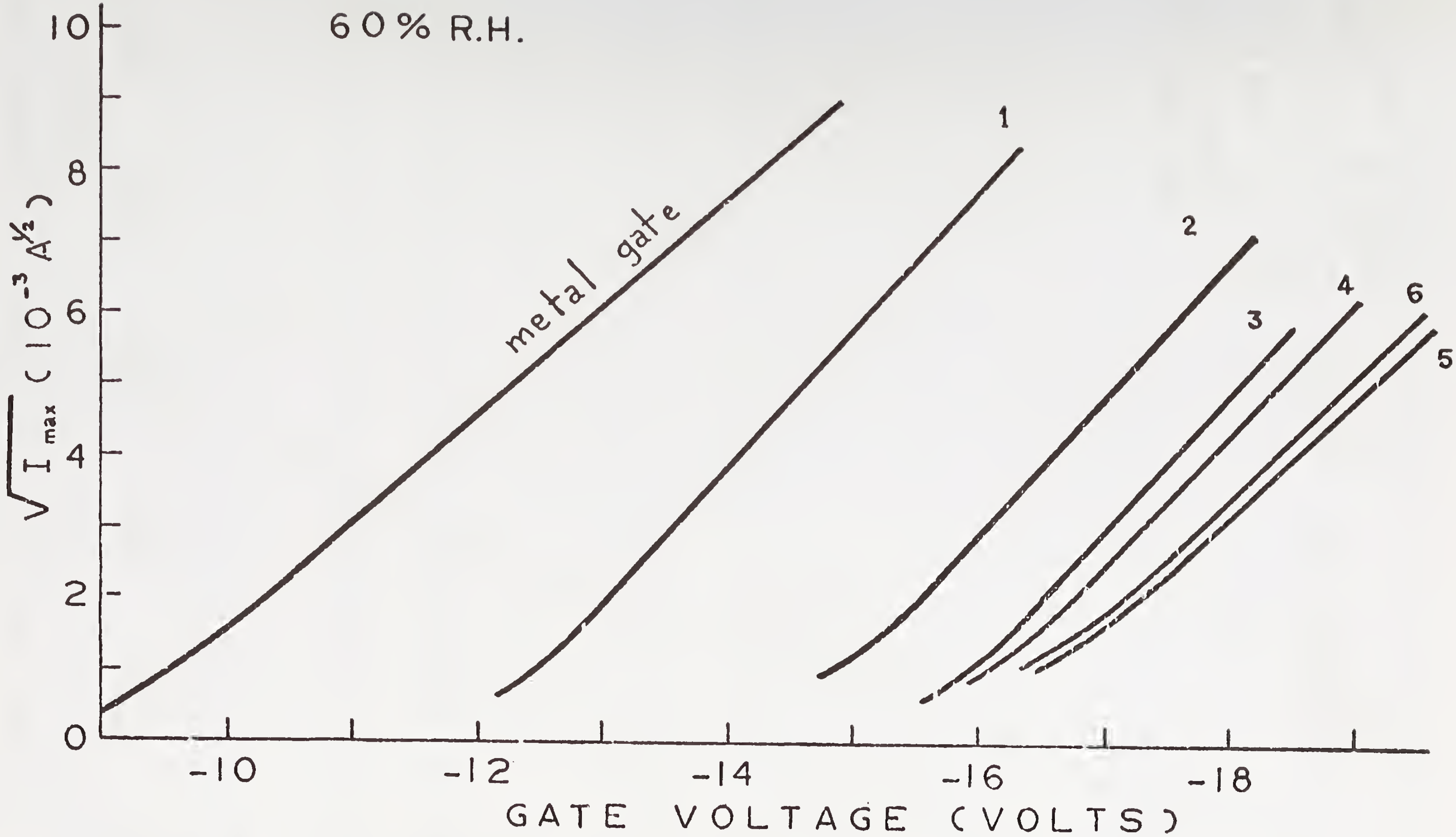

Figure 3. Maximum drain current *versus* gate voltage for a whole array. The number gives the gap width in quarter mils.

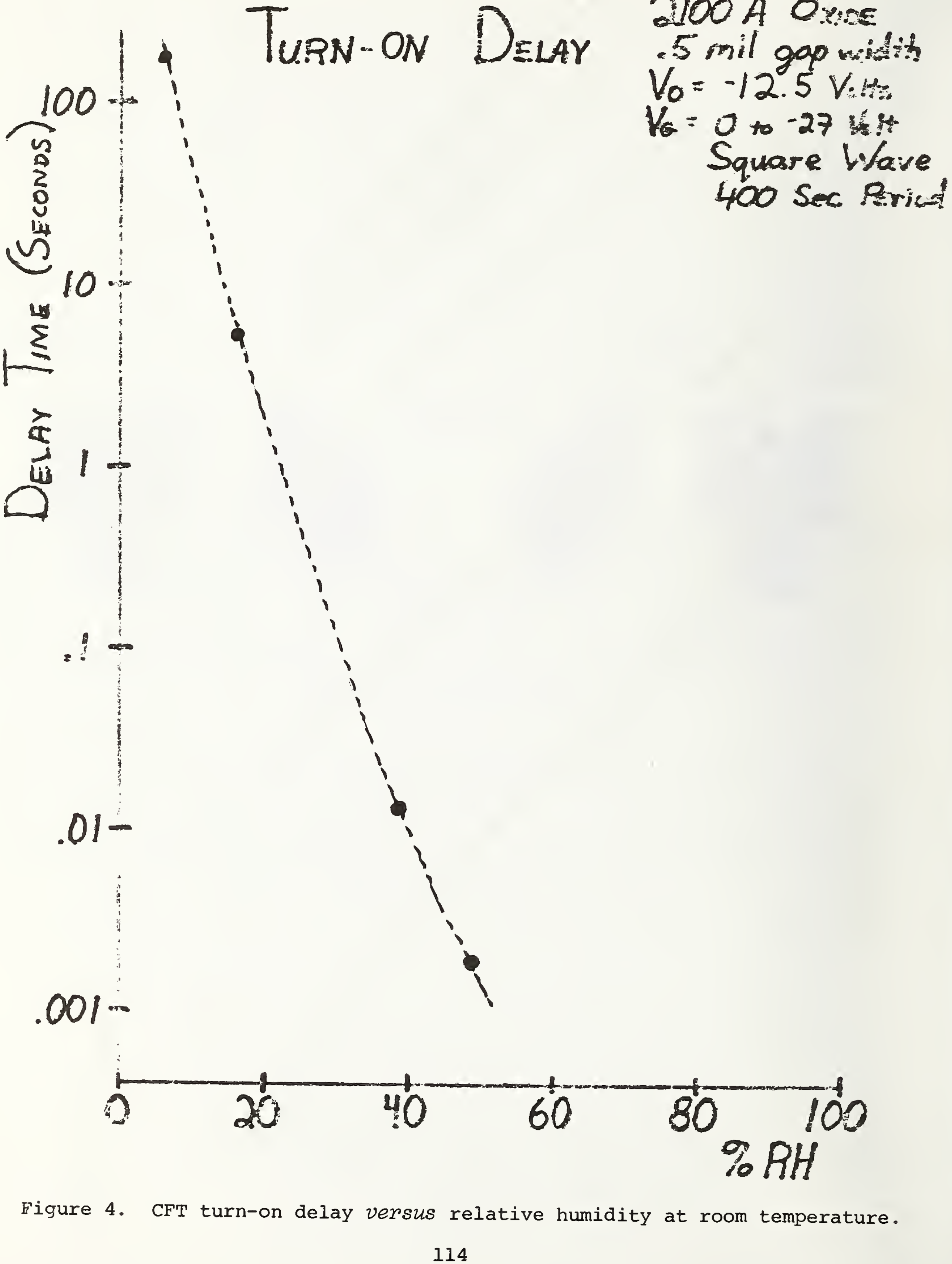

Figure 4. CFT turn-on delay *versus* relative humidity at room temperature.

3.7 A Surface Conductance Technique for the Evaluation of Internal Package Environments*

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We report some results of a study of moisture monitors as a check on their application in packages for device reliability. We wanted a sensitive method that could be used by technical personnel for moisture determination, but as we got into the study, other advantages of the surface conductance sensor appeared that might be more relevant to reliability.

Before going into detail, we note the correlation of dew point to the specification Method 1018 of MIL-STD-883B, which places a limit of moisture content at 5000 parts per million by volume (ppm_v), namely, that the dew point will vary with different packages as based upon the seal temperature because of the final total pressure. Table 1 lists such data. Sources of moisture are the furnace ambient, reaction products, and desorption. For reliability purposes, the dew point is a more relevant measure than the relative concentration of moisture since the dew point relates to surface current which correlates to corrosion, to charge spreading instability (in MOS structures), and to modification of transistor gain. Whether the dew point or frost point is obtained does not appear to be a factor, as we have not seen a change in conductivity to indicate a change in phase.

The surface conductance sensor is an interdigitated aluminum pattern on a thermally oxidized silicon chip 0.095 in. on a side. The die were attached by eutectic means in a variety of LSI packages.

Surface conductance is monitored over a temperature range of [illegible]°C to -70°C and displays the following:

1. Increasing surface current with decreasing temperature as the relative humidity inside the package rises and modifies the surface conductance of the silica.

2. A region in which the surface current increases and then peaks as additional water trapped in the package condenses on the sensor surface.

3. A region of declining surface current with temperature as no significant further condensation takes place and decreasing specific conductance of the surface layer with temperature becomes the predominant effect.

The absolute current observed depends on water content of the package and temperature and may be substantially increased by the presence of mobile ions

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* This paper was originally given in the Panel Meeting on Moisture Sensors but is incorporated here for continuity.

on the surface of the die. This renders the system particularly effective in evaluating new package materials and the potential reliability of devices.

We have done some modeling to anticipate the surface current as a function of temperature for a given package ambient. Figure 1 represents the surface sheet resistivity of silicon dioxide as a function of relative humidity. Figure 2 indicates the calculated film thickness of water as condensed within a 22-pin DIP for several initial concentrations of moisture. Figure 3, then, is the expected surface current as a function of temperature for the three cases under a 50-V bias, where the characteristic is affected by the surface conductance of the silica and the conductivity of the water film. Derived results depend upon the assumed value for the conductivity of water and the temperature dependence. We could only use approximate values since little data exist for temperatures lower than 0°C.

Figure 4 displays results with real packages. Two types of sealing glass were involved. Glass #6 is, presumably, a replacement for glass #2 by some vendors, but note that the sensor current peaked at a higher temperature for #6 which indicates a higher water content, and the current amplitude is greater which indicates greater contamination. The two curves in each case represent extremes in data. Although the position (and amplitude) of the peak is not that of the dew point, it is a sensitive correlation to surface behavior.

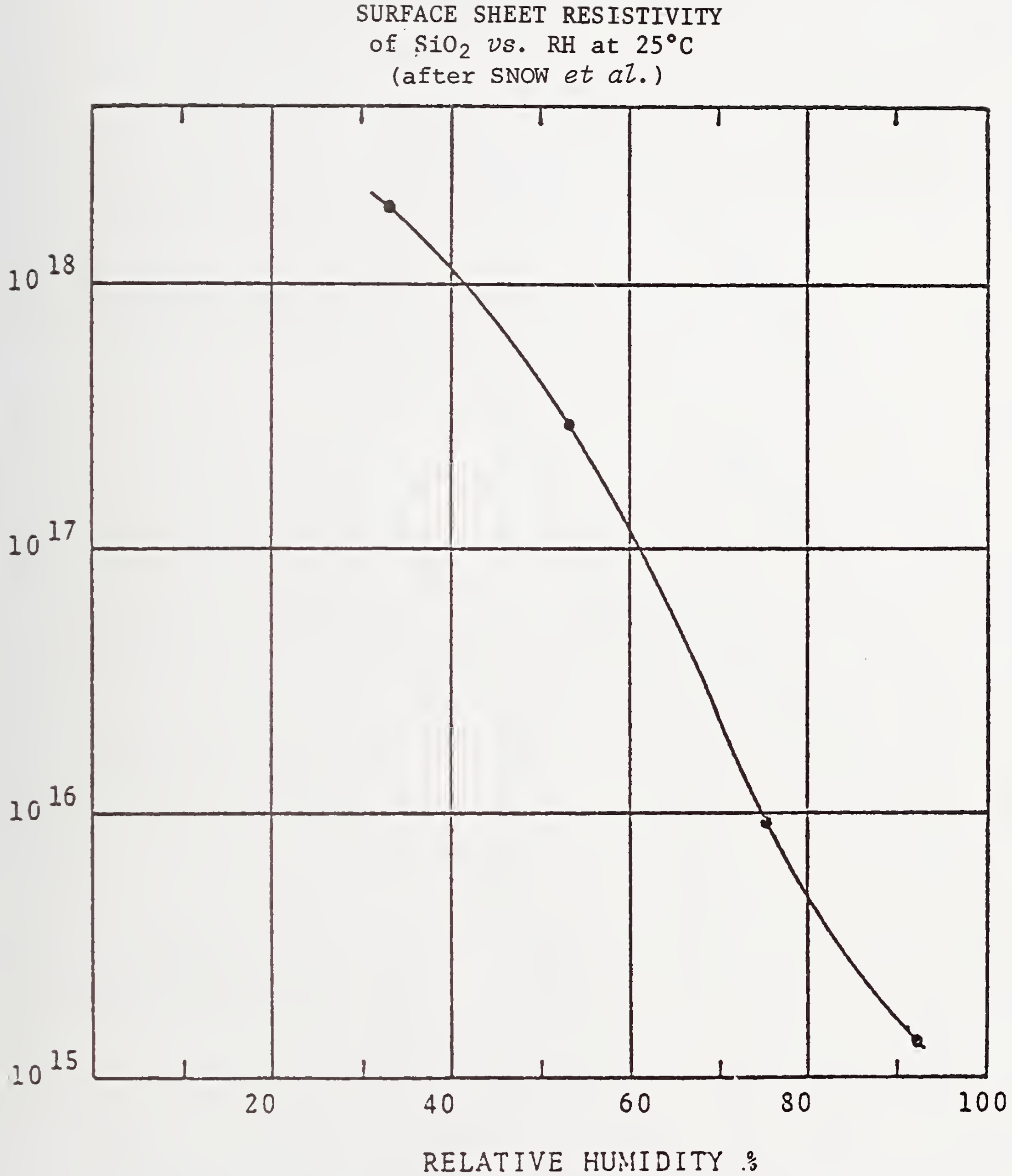
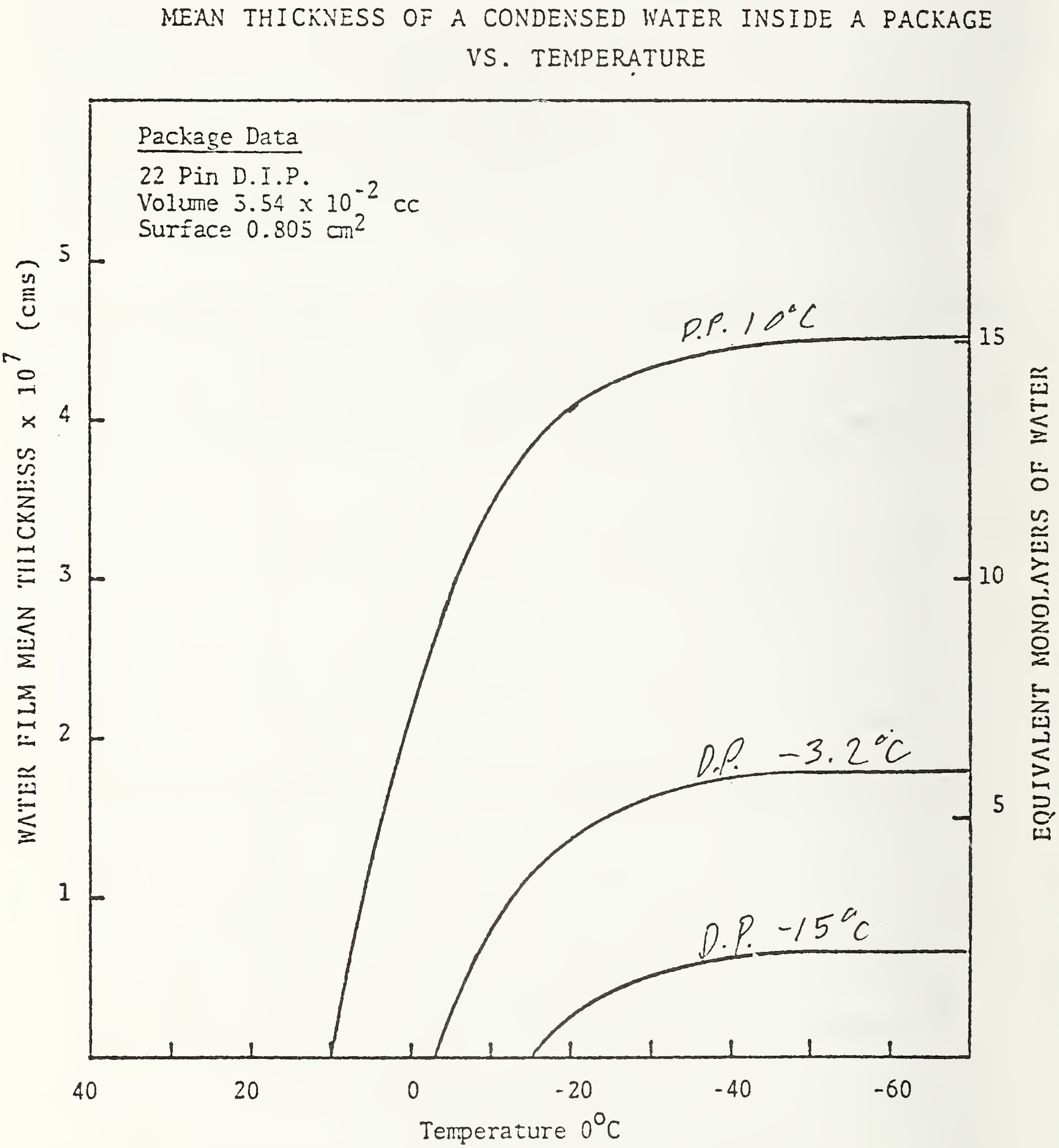


Figure 1. Surface sheet resistivity of SiO_2 as a function of relative humidity at 25°C (after SNOW *et al.*).



MEAN THICKNESS OF A CONDENSED WATER INSIDE A PACKAGE VS. TEMPERATURE

Figure 2. The calculated film thickness of water condensed within a 22-pin DIP for several initial concentrations of moisture.

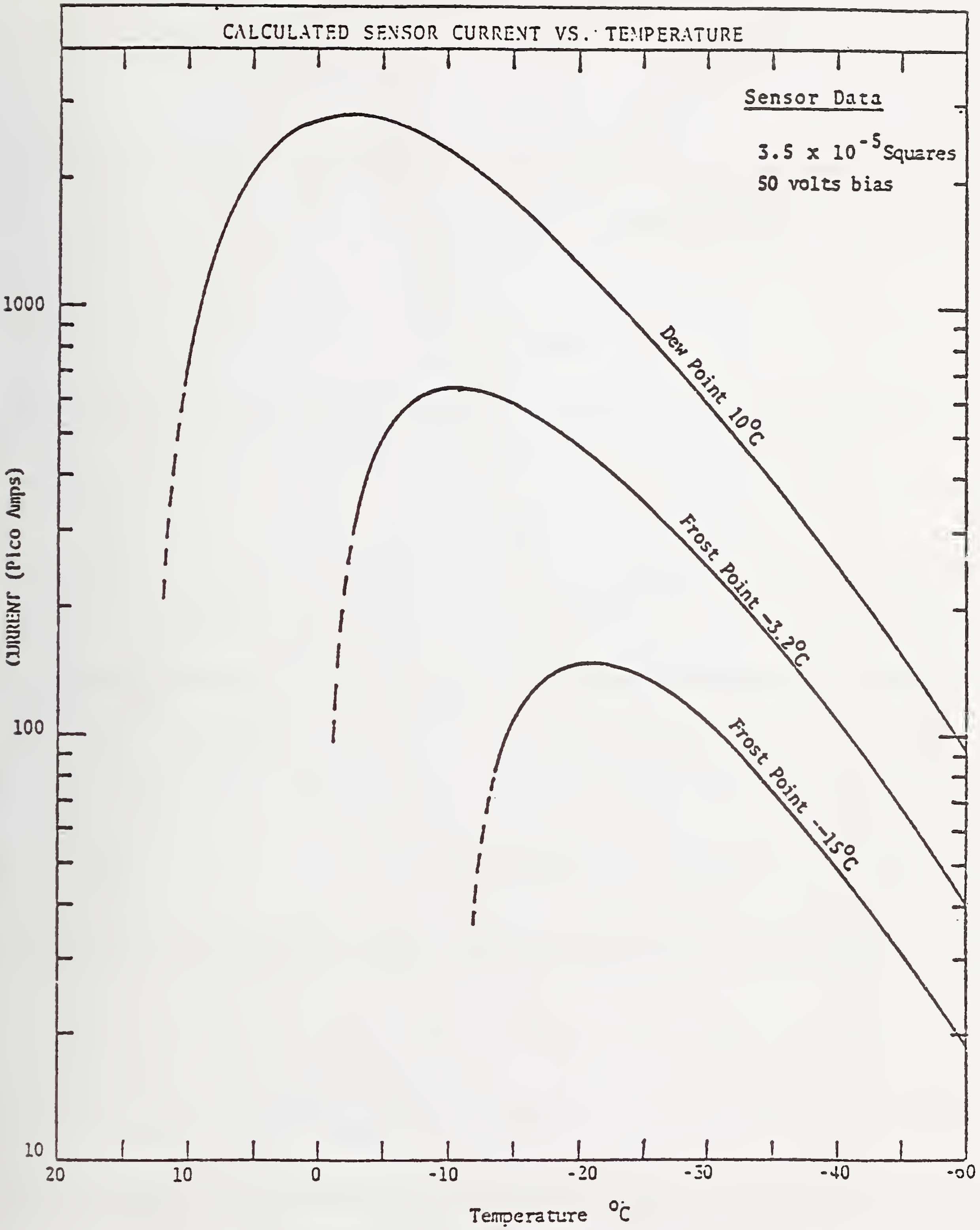


Figure 3. Expected surface current as a function of temperature for the three cases indicated in figure 2 with a 50-V bias or affected by surface conductance of SiO_2 and the conductivity of the water film.

SENSOR CURRENT VS. TEMPERATURE 2 TYPES OF GLASS SEAL

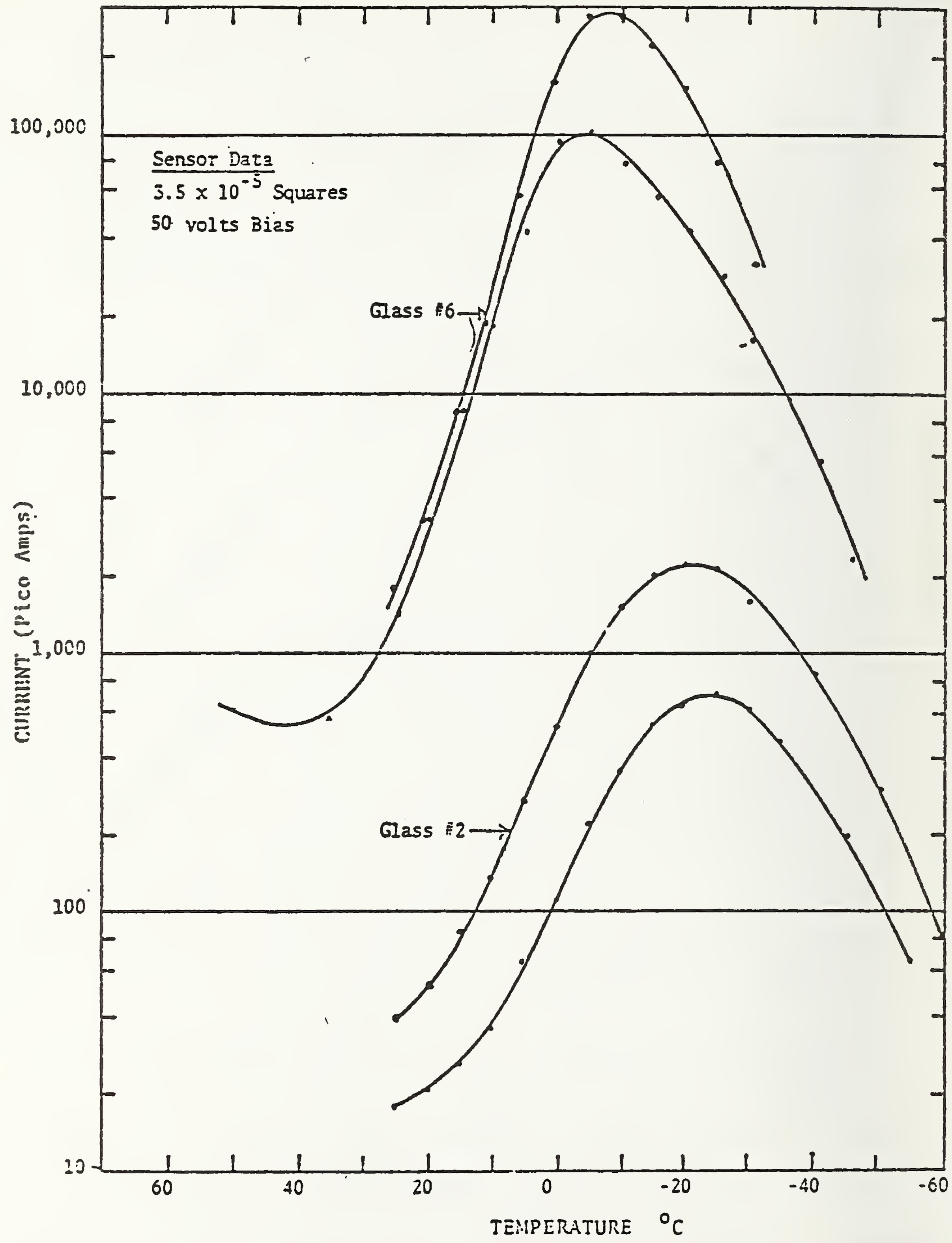


Figure 4. Experimental values of sensor current as a function of temperature for two types of sealing glass.

Table 1. The Correlation of Indicated Dew Point to a Package Moisture Content of 5000 ppm_v for Packages Sealed off at Various Temperatures.

DEW/FROST POINT VS. SEAL TEMPERATURE (5000 ppm H_2O)				
Seal Temperature	25°C	300°C	400°C	500°C
Cavity Pressure	760 mm	395mm	336mm	293mm
Dew/Frost Point	-3.2°C	-11.3°C	-13.3°C	-14.9°C
Typical Seal Process	Weld	Solder	Vitreous Glass	Devitrifying Glass

4. PANEL MEETINGS

4.1 Discussion Group Summary of Mass Spectrometer Measurements

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The discussion on mass spectrometry started with the techniques used to calibrate a flow system, for example, the advantages and disadvantages of circulating a gas bottle full of moisture. It was felt that the gas bottle is most reproducible at 100 ppm. There are problems if you try to contain 1000 or 5000 ppm moisture for a long period of time. Another commoner calibration technique is, of course, the dilution flow technique, which a number of people are using. One other calibration technique was brought up that I was not familiar with. That is, the gravometric technique where you flow the moist gas over phosphorus pentoxide, and you weigh it before and afterwards. This discussion was prompted by the need for a method of accurate calibration at the high ppm level, 5000 to 10 000 ppm. It was suggested that the gravometric technique could be used very effectively, but I do not think anybody has evaluated the absolute accuracy and the tolerances one might expect using the phosphorous pentoxide system on 0.01 cm^3 gas volumes.

A good portion of the discussion centered around the problems that one gets into when you calibrate with a constant flow of gas, and then puncture a package, and have a small volume burst of gas. I think the most important thing that we learned at the conference was that there is a lot more work that has to be done like Bob Merrett reported from England. This is really the type of work we need to determine the moisture loss to surfaces in the measuring system and how it does vary as a function of temperature. A discussion followed on surface preparation of the stainless-steel systems and whether there are other materials which might be used. One technique suggested was to anneal stainless at a high temperature in oxygen. Chrome in the stainless diffuses to the surface, where it is oxidized giving a rather inert surface. But it then becomes hygroscopic so you solve one problem and generate a new one. Bob Merrett mentioned another technique that they used in England, which they came upon rather serendipitously. They found that nickel packages would transmit hydrogen through the package wall at a very constant and reproducible rate. In fact, you can take a nickel can, insert it into an oven with a high temperature hydrogen environment, and quite accurately predict that water will be generated on the inside of the package. Hydrogen reacts with the nickel oxide on the inside of the package and converts it catalytically to moisture. Mr. Merrett said he could get upwards to 3-percent water inside the package if he waited long enough. In an actual failure analysis, they discovered packages that became wet after exposure to a hydrogen environment for an extended period of time. This might solve the problem of sealing standards in wet environments. Most sealing apparati don't like to be operated in 10 000 ppm moisture, preforms oxidize, and the gold actually forms nonwetting surface layers. When sealing in moist ambients, you are contaminating the clean surfaces that you want to mate with either the solder or the welder, so Mr. Merrett was suggesting that this might be a technique for filling a package that was already sealed. The technique would generate moisture inside the package after it has been sealed, and, therefore, you eliminate the problems of contaminating the surface. It is an intriguing technique, and I would be glad to offer moisture analysis services to anybody that wants to seal up some nickel caps using this technique. There still is the problem determining how much water is in the package after hydrogen treatment. Since only one in every 10 atoms of hydrogen that get inside of the package produces water, there are questions on how reproducible is this ratio and what is the effect of wall thickness and temperature? These all have to be worked out, but it is a rather intriguing physical-chemical process that might be useful in developing standards.

We discussed the round robin for the better part of an hour. The purpose was to explain some of the differences of the technique involved in measuring. The data presented were uncorrected, but if you look down each column for each analytical lab, you could start seeing the thing that we are calling the transfer function appearing: one analytical lab having a lower sensitivity to a small volume of moisture than another. It certainly pointed out that our statistics are poor, and that you cannot make conclusions on results that differ as much as we had on the round robins. In preparing the standards, I pointed out that we were under the assumption that to start with a clean package was the best case and as Chuck Messenger mentioned yesterday, half of the moisture disappears within the first couple of hours, so you are not in equilibrium, if you dry out your package. You are better off to seal it wet and then try to backfill it. Less moisture is needed from your backfill to bring the package ambient into equilibrium and it looks like the uniformity is better when you use a package that has not been baked out, as in Round II. The elimination of the vacuum bake-out process is one of the few things that makes it easier to assemble the standards. Some of the problems in making the standards were the calibration shifts that are observed in mounting the sensors used inside the packages. We now know a little bit more why we are getting the shifts and the importance of calibrating the sensors after the mass spectrometry correlations. In succeeding round robins we would hope to bring all of the package sensors that are used in the standards back to the Bureau of Standards and have them recalibrated, since this is a prime source for calibration. At RADC, we have a group of sensors which we use as a voting scheme. The unknown and five sensors are in the same chamber, and we assume that none of those have drifted the same amount, but that by no means is a prime calibration source. We will also probably in the next few months purchase an optical dew point measurement system so that we will have another independent method of determining moisture content.

4.2 Comments on Round-Robin Results

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Test packages for Round I were gold-plated, all-metal, solder-sealed packages with a volume of approximately 0.07 cm^3. The packages were baked out for 89 h at 125°C before backfilling. Two sensors were used to measure the moist backfilling gas. One is denoted the sealer sensor and is located within the inner seal chamber of the RICO sealer. The other, the package sensor, is mounted inside the sample package. The sealer sensor is monitored during seal. Since the backfilling gas is at 5 psig, this reading has been corrected to 3 psig for comparison with the package sensor which has a 3 psig ambient pressure after cooling to room temperature. Since the package sensor indicated a decrease in moisture over a period of several weeks, a precise correlation of the package moisture at the time of opening and the mass spectrometer analysis cannot be performed. Since all packages were returned to RADC, it was possible to measure the package sensor after opening and check its calibration. The correction factors for the sensors appear in Column 9. Using these factors, combined with the sensor reading nearest the analysis data, yields the best measure of content inside the packages at the time of opening. An additional correction may be necessary since the package moisture measurement is made at 25°C and the mass spectrometer measurement is performed according to Method 1018 at 100°C. This correction is not expected to be more than 20 percent. It will be determined experimentally on the RADC Rapid Cycle system which has the capability of making mass spectrometer readings at a package temperature of 25°C.

Data from the four analytical labs were then compared with the corrected sensor reading. The ratio was used to compute the correction factor for each laboratory. Lab B had only one package in Round I with gas remaining after evacuation; therefore, as noted, the correction factor could not be computed. A value of five was assumed for Lab B in Rounds II and III since it brought Lab B's readings into agreement. Lab D made some changes in technique which improved the accuracy of the measurements at 5000 ppm; therefore, no correction factors were applied for Rounds II and III. This should not be interpreted as indicating that Lab D readings are the correct ones, since calibrated sensor standards were not used to compute an accurate correction factor, as was done in Round I.

Round II packages were not baked so, therefore, much less moisture was adsorbed onto the walls after seal. In fact, the seal sensor readings are very close to the corrected mass spectrometer readings indicating that a vacuum bake is not desirable in the preparation of moisture standards. This is important if one is trying to fill the standard to a specific value.

One other change was necessary between Rounds I and II. Because of poor sealing yield, the sample package was changed from an all-metal to a metal-ceramic side-brazed package which worked better in the RICO sealer under wet seal conditions.

Round III was vacuum baked in hopes of improving uniformity. The large difference in seal sensor and mass spectrometer readings again demonstrates the loss in moisture from the ambient to the dry package walls after seal.

The results of the round robins indicated the need for moisture standards and validated the concept of using a moisture sensor within a semiconductor package. Rounds II and III demonstrated the predicted precision by the analytical labs since a common correction factor for each one could be assigned which would correct for system moisture losses. The results of Round II indicate that with properly prepared samples, the labs were within ±27 percent of a median value.

With this experience behind us, RADC will prepare additional standards with and without sensors with the goal of demonstrating less than 20-percent error before full certification of the analytical labs is granted on June 5, 1978.

In addition, standards will be prepared with volumes of 0.1 and 1 cm^2 with moisture contents of 5000, 1000 and 500 ppm to insure accuracy for larger packages with high and low moisture content.

These samples will be sent to two additional labs which have requested certification. Results of the above experiments will be presented to JEDEC Core 14 as they become available.

4.3 Moisture Sensors

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The miniature humidity sensors which are to be enclosed in hermetic semiconductor devices are still in the beginning stages of development. However, a great deal of effort is being expended to rapidly advance the state of the art for measurement of humidity and/or the total moisture content of hermetic semiconductor packages.

Except for the requirement of small physical dimensions, the sensor specifications for the operating characteristics is generally similar to those in other fields of humidity measurements. To make meaningful measurement of the water vapor content of the sealed atmosphere using the electric hygrometer sensor, the following are some of the operating parameters which must be determined.

Stability: The stability of the sensor must be measured after exposure to various ambient conditions to which the sensors will be exposed during the packaging process as well as during actual usage.

Hysteresis: The absolute quantity of water in the package should be either fixed or increasing with time. The relative humidity in the package can increase or decrease as the ambient temperature decreases or increases.

Range: The ambient humidity and temperature range of the sensor.

Temperature Coefficient: The effect of ambient temperature on the calibration of the sensor.

Pressure Coefficient: The effect of ambient pressure on the calibration of the sensor.

Repeatability

Sensitivity: The characteristics outlined above can be determined with the calibration of the sensors in humidity-producing equipment with known accuracy. However, for the calibration to be meaningful, the reproducibility of the sensor characteristics must not only be from within one manufactured lot but also be reproducible from different manufactured lots produced at different periods of time. The sensors should be tested to determine the effects of exposure to high temperature, of bonding of electrical leads to the sensor's electrodes, contamination effects, etc. The same electrical parameters such as voltage and frequency used for *in-situ* measurements should also be used during calibration.

At surface temperatures warmer than the dew point temperature, the amount of water film adhering to the surface can be made thicker or thinner by cooling or heating the sensor surface. This change in film thickness, which produces a corresponding change in surface conductivity, can be related to ambient humidity by calibration and testing procedures outlined for the electric hygrometer sensors. The degree of surface conductivity will depend not only on the thickness of water film but also on the amount of contaminants on the surface. Therefore, this method would provide an assessment of the reliability of the semiconductor device operating at low temperatures due to the presence of both water and contaminants, i.e., the stability of a MOS or linear devices due to the effects of spreading surface charges.

Correlation Between Humidity Sensors and Mass Spectrometry Measurements

The mass spectrometer measures either the total or some fraction of the total water in the package while the humidity sensor measures the equilibrium water vapor content in the package. The total water content will depend on such factors as internal volume, cleanliness, and the amount as well as the hygroscopicity of the material inside the package. The total water which can be driven out of the hygroscopic material will be a function of pressure, temperature, and time. (The details of the precautions necessary to ensure no addition or subtraction of water molecules between the sampling system from the package to and through the mass spectrometer are described in the Mass Spectrometer Section of this report.) The equilibrium water vapor pressure in the package is a function of ambient temperature.

The tests to determine the feasibility of correlating the humidity sensor and mass spectrometer measurements should be made with calibrated humidity sensors and tested for specific size and material of package, as well as the components inside the package. Tests should also be made to determine the effects of different test conditions, i.e., temperature, pressure and duration of test, on the correlation of the two types of measurements.

The consensus was that there should be a similar workshop next year. Another suggestion was that a panel of interested parties from industry, government, users, and vendors be organized to interchange information and to edit and disseminate such information to interested parties.

5. SESSION III PACKAGE ANALYSIS AND QUALITY ASSURANCE

5.1 Parts-Per-Million Water-Vapor Generating System Used to Simulate Moisture in Small Integrated Circuit Packages*

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INTRODUCTION

The object of this project was to develop a device which could be used to simulate the gas contents of hermetically sealed packages. Essential in such simulation are three important variables: package gas pressures, package gas mixtures of known moisture content, and package volumes. Previous generating systems have provided the required gases of known moisture content [1-3]. However, no known system has heretofore simulated a variety of package volumes and package pressures. The device described below provides independent control of all three essential variables. A final variable, package internal adsorbing materials is not simulated in this system.

DESCRIPTION

Figure 1 shows the unit that was constructed and tested. During operation, pressurized gas flows through the package simulator - starting at valve V1 through V2, through humidifier H1 or H2, through V3, through gas-flow regulator R, through a variety of sample loop tubes attached to valve V6, and finally venting through an adjustable orifice of valve V4. Flow regulator R can be set to gas-flow rates ranging from 5 to 200 cm^3 per minute. Humidifier H1 is chosen to provide a calibrating gas with a moisture content ranging from 3000 to 20 000 ppm. Humidifier H2 is used to provide a calibrating gas from 250 to 3000 ppm moisture.

The free volume of a package is simulated by changing the length of tubing in the sample loop, SL, attached to valve V6. In the "loop load" position, gas of known moisture content is pumped through this loop continuously; this purging is essential to minimize wall adsorption. Then, to simulate package puncture in the inlet vacuum system of a mass spectrometer, the six-port valve is moved to the "loop inject" position. The simulated package sample of gas is introduced into the mass spectrometer immediately.

The moisture content of the flowing gas is calculated by taking the ratio of the vapor pressure of the water, P_w, for that humidifier to the total pressure of the flowing gas, P_t:

$$\text{ppm} = \frac{P_w}{P_t} \times 10^6 .$$

Distilled water was used in humidifier H1 during these tests. From the

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* This work was supported by the U. S. Department of Energy.

measured temperatures of H1, values of P_w were obtained using standard water-vapor pressure tables [4]. A saturated solution of water and lithium chloride was used in humidifier H2. From the measured temperatures of H2, values of P_w were obtained from tests reported by Wexler and Hasegawa [5]. For a constant temperature, the ppm moisture content of the flowing gas was set by adjusting the total gas pressure, P_t.

Direct proof of performance was obtained by comparing the ppm moisture generator with Sandia Laboratories' Transfer Humidity Standard. In table 1 it can be seen that agreement between the humidity standard and the generator is typically within 15 percent.

CONCLUSION

This water-vapor-generating system can simulate three important variables in hermetically sealed packages:

- package gas mixtures including water content
- package free volume
- package gas pressure.

The generator has been useful as a fundamental tool in calibrating mass spectrometers for use in gas analysis of hermetically sealed packages.

ACKNOWLEDGMENTS

Without the design modification contributed by Dr. F. B. Burns, this package simulator would not have been equipped with the variable volume feature used in the final model. Also, his continued interest in the project prompted numerous discussions during which he gave valuable advice on data acquisition and interpretation.

Special thanks are also due Mr. F. Koletar for his work on design and construction of the generating system.

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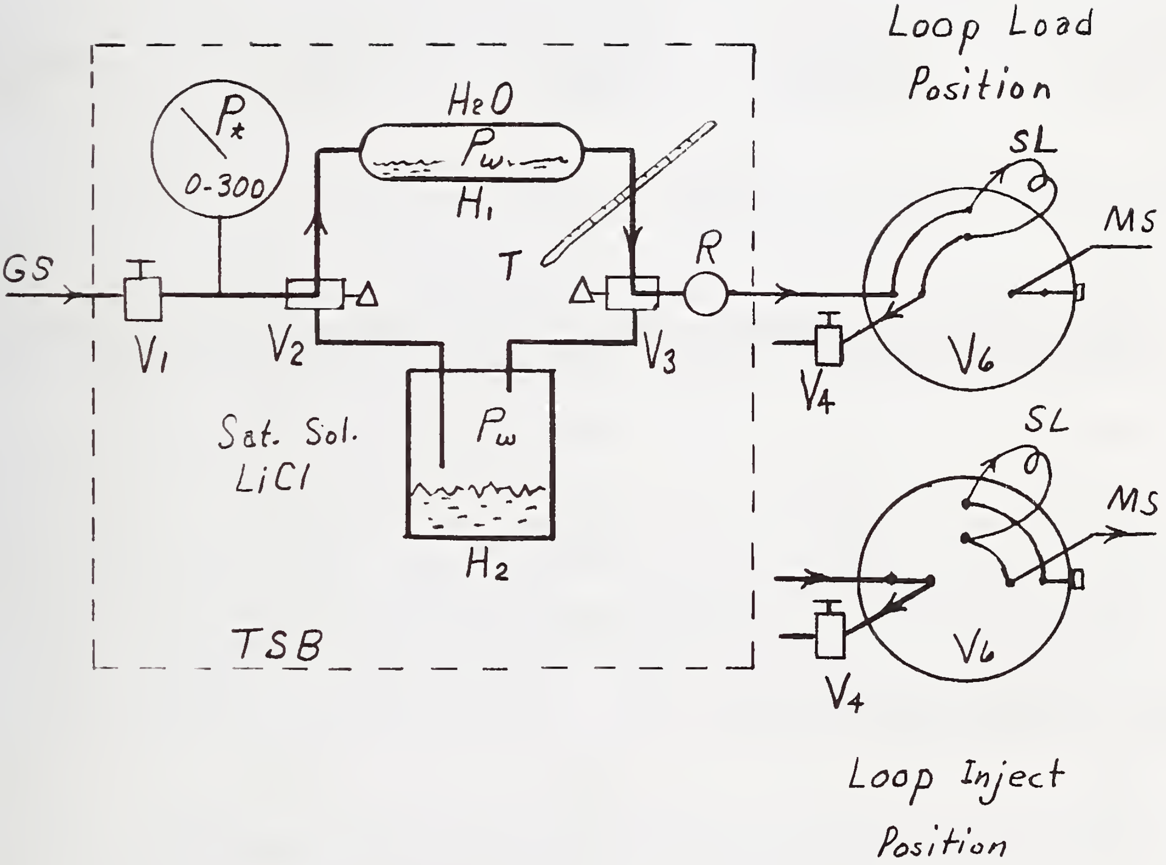

Figure 1. Hermetically sealed package simulator. GS - dry gas source; V1, V2, and V3 - gas control valves; V4 - sample loop pressure adjust and vent valve; R - gas flow regulator; V6 - six-port loop load and inject valve; SL - sample loop used to simulate package volumes; MS - mass spectrometer connection; P - total gas pressure (gage); P_w - water vapor pressure; T - humidifier temperature; TSB - temperature stabilizing box; H1 - liquid water humidifier (1 L); H2 - lithium chloride saturated water solution humidifier (3 L).

Table 1. Generator Compared with Transfer Humidity Standard.

Stabilizing Time (min)	Generator Flow Rate (cm^3/min)	Generator Setting (ppm)	Transfer Std. Reading (ppm)	Difference (%)
Lithium chloride saturated water solution humidifier, H2				
25	50	372	427	12
80	173	372	420	11
17	190	741	741	0
25	70	741	743	0
16	110	1468	1435	- 2
7	74	2798	2345	- 16
45	32	2798	2633	- 6
Distilled water humidifier, H1				
140	11	2968	3267	10
23	28	5840	5227	- 11
960	10	5828	5655	- 3
30	50	11 480	9770	- 15

5.2 A Total Moisture Analyzer for Hermetic Devices

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Most of the commercially available instrumentation for total moisture analysis of hermetic packages is based on the expensive and relatively complex mass spectrometer. This paper describes an inexpensive, easy-to-operate moisture analysis system using an aluminum-oxide-type moisture sensor as the detector element. Moisture is transferred from the package to the detector using either carrier gas or vacuum techniques. The instrument was originally designed to analyze heart pacemakers, but with slight modifications it can also be used to make moisture determinations on hybrid packages.

Instrument Description

A block diagram of the instrument is shown in figure 1. Note the similarities to a conventional gas chromatograph. The aluminum-oxide moisture-sensor output is monitored with a hygrometer, and a permanent record of the analysis is made with an x-y plotter and digital integrator. A port for injecting water via microliter syringe is located between the chamber O-ring seal and the detector. This allows direct calibration of the instrument without the need for packaged moisture standards.

Instrument Operation - Carrier Gas Flow

Figure 2 is a block diagram of the instrument with the carrier gas plumbing shown as solid, dark lines. The instrument is first calibrated via moisture injections bracketing the expected moisture levels to be found in the sample packages. Calibration is repeated following analysis of samples to verify that detector sensitivity has not changed due to contamination or some other variable. If necessary, the second calibration series can cover a slightly different moisture concentration range to allow for unexpectedly dry or moist samples.

To analyze a package, it is held at 100°C for 24 h, then rapidly transferred to the heat and sample chamber. A hole is then made in the package with the outlet-side puncture device, then an inlet hole is made with the second device. If necessary, flow rate is adjusted to the value used during calibration. Data are recorded and integrated until the original baseline response is reached. The integral under the peak is in units of micrograms per cubic centimeter per second. Since flow rate is a constant, multiplying by seconds per centimeter gives an integral value expressed in micrograms directly. If required, free volume of the package can be measured and moisture content calculated as parts per million by volume (ppm_v).

Figure 3 shows typical calibration injections in the 10- to 80-μg range. Figure 4 is a complete calibration curve for the range 10 to 10 000 μg. Figure 5 shows actual analysis results for four packages built at the same time under similar conditions. Total moisture content of these packages was expected, based on previous mass spectrometer studies, to be somewhere near 50 μg.

Figure 6 shows the variation in detector response to repeated 100-μg injections at various flow rates. These data are plotted in figure 7. As expected, instrument sensitivity increases as flow rate is decreased. However, analysis times lengthen considerably at flow rates much below 2 cm^3 per minute. Since a longer analysis time results in increased background from moisture residual in the carrier gas, analytical error increases with analysis time at low moisture concentrations. The resulting practical detection limit is about 5 μg, obtained at a 1 or 2 cm^3 per minute flow rate.

Instrument Operation - Vacuum-Sampling System

The vacuum-sampling system was intended for use with packages where it would be difficult to make two puncture holes, such as hybrids, or where more absolute sensitivity is required. To analyze a package with this system, the package is first heated to 100°C and held for 24 h. The package is then transferred to the sample chamber. A vacuum pump evacuates the plumbing shown in heavy dark lines in figure 8. When the vacuum reaches 10 μm, the valve between the pump and system is closed and a single hole is made in the package. Initial and final hygrometer readings are recorded, the difference being proportional to the moisture content of the package. Calibration of this system requires packages containing known quantities of moisture. Since such standards are currently unavailable to this laboratory, little work has been done so far with this sampling system.

SUMMARY

The instrument satisfies the need for a total moisture-analysis system which is inexpensive and easy to operate. As many as 30 packages containing 100-μg moisture or less can be analyzed in an 8-h period. Calibration is simple and repeatable. No standard moisture packages are required for its operation in the carrier-gas mode. The system as described meets the requirements for a carrier-gas-flow type moisture analyzer as described in the Internal Water-Vapor Content Test, Test Method 1018 of MIL-STD-883B.

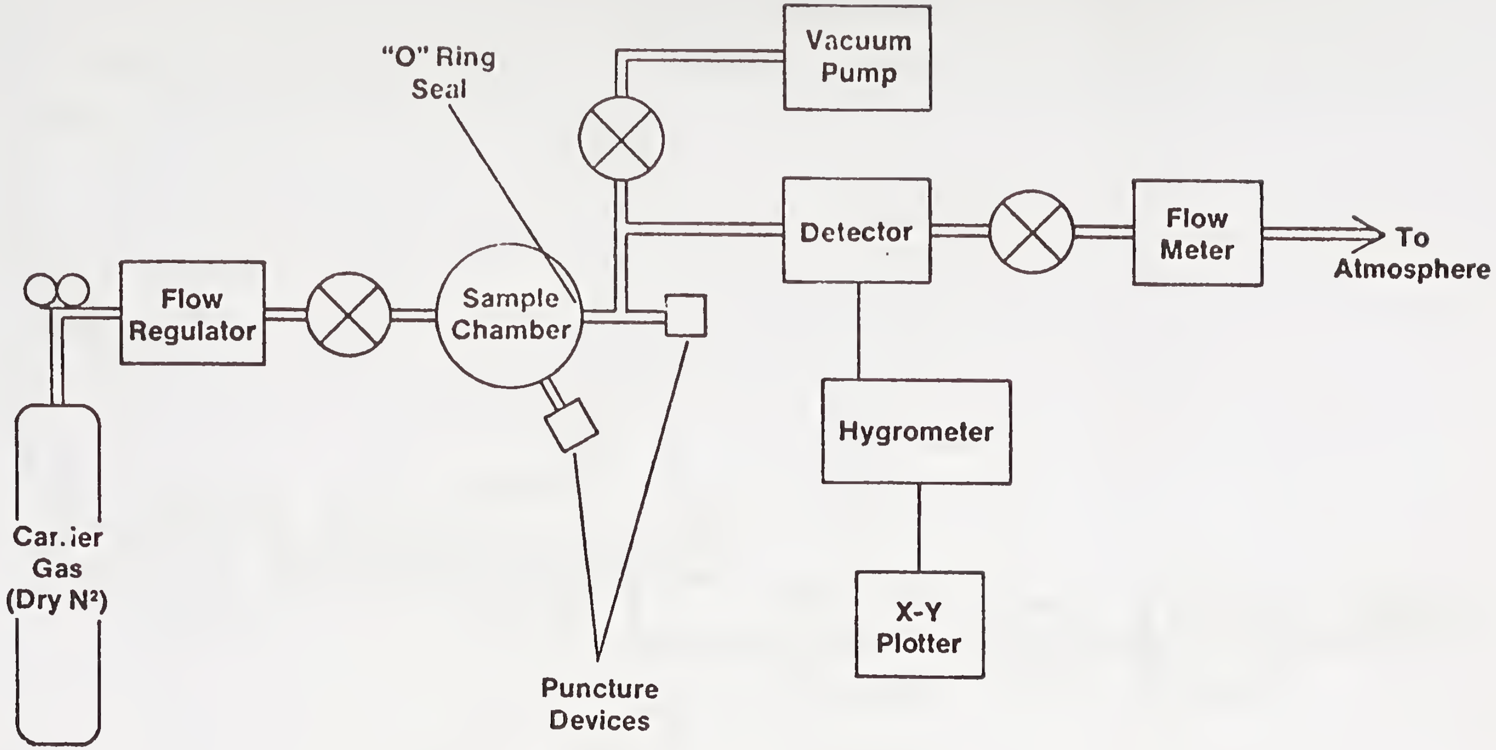

Figure 1. The block diagram of the moisture analyzer.

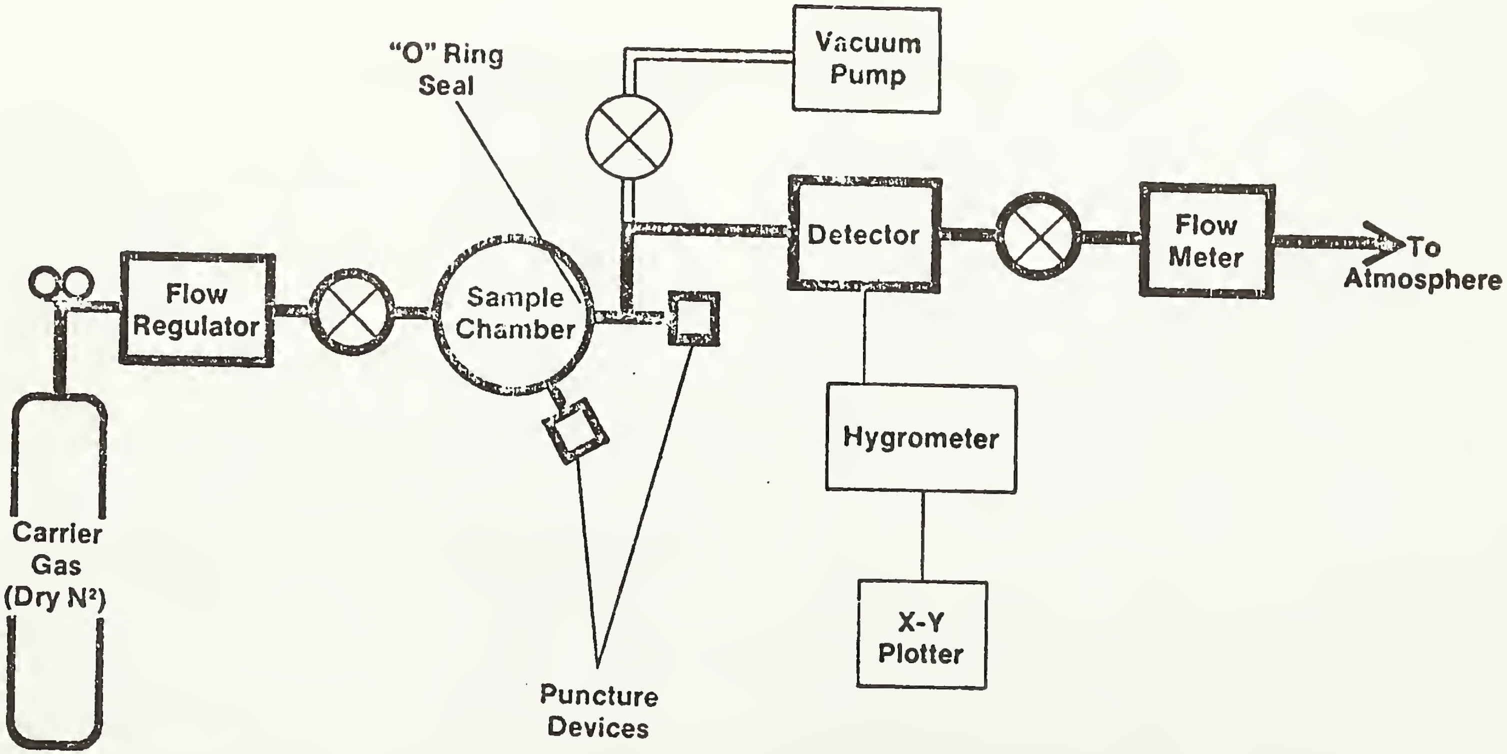

Figure 2. A schematic diagram of the carrier gas flow route for the moisture analyzer.

Figure 3. Detector response of the moisture analyzer to calibration injections of 10 to 80 μg of water into the sample chamber.

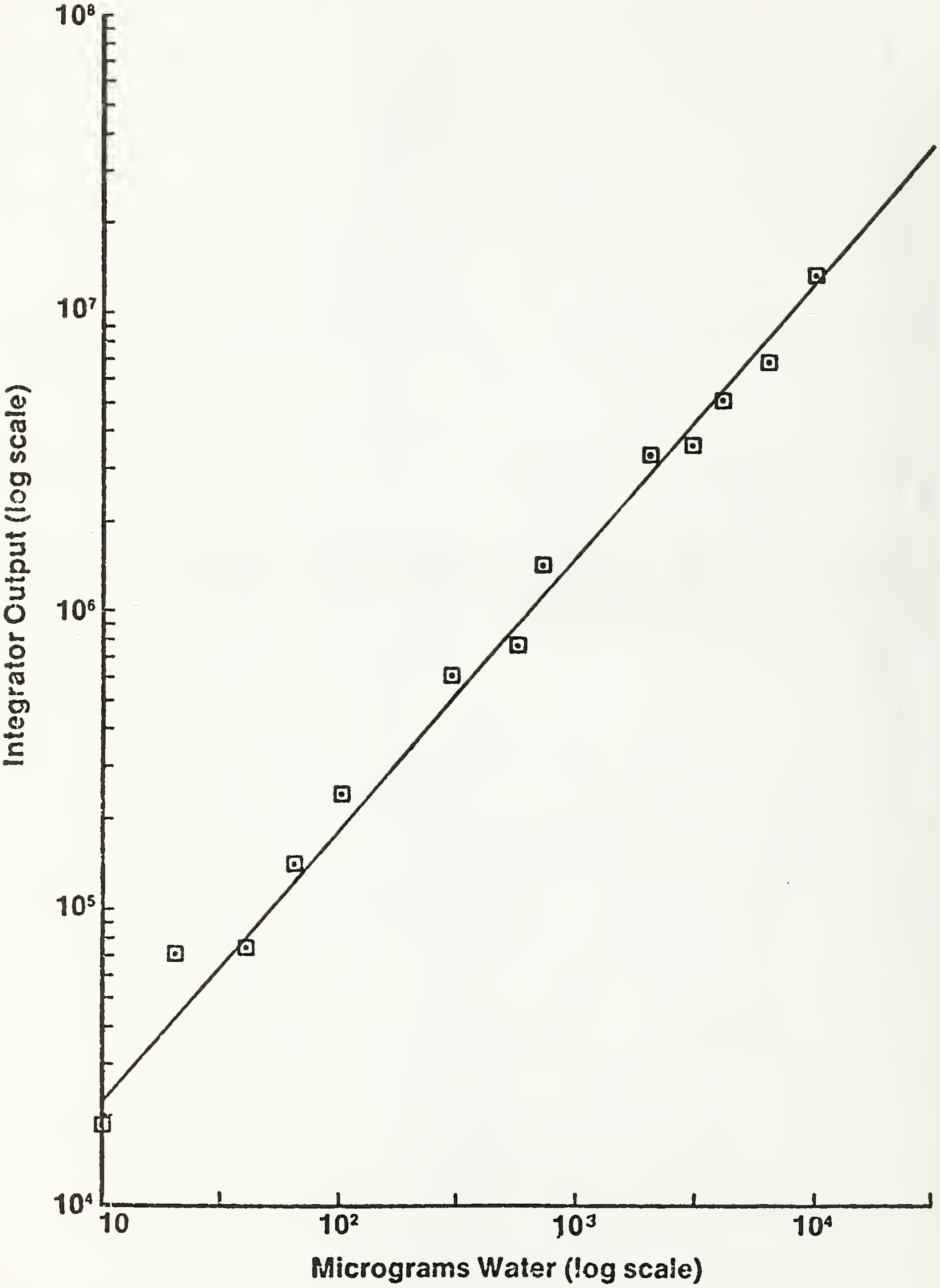

Figure 4. A typical calibration result for the moisture analyzer for an injection range of 10 to 10 000 µg of water.

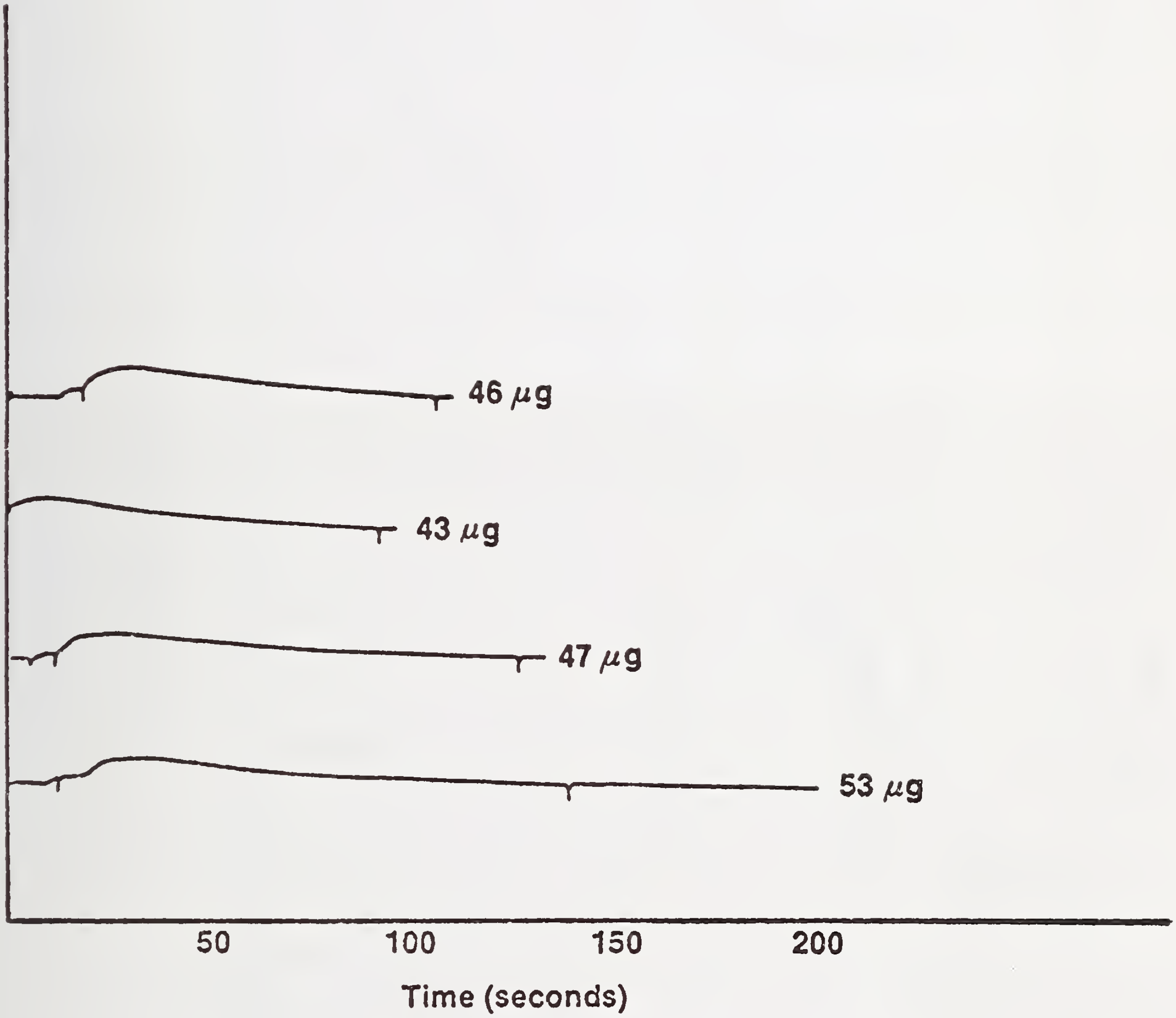

Figure 5. Response of the analyzer for four packages. The base for each trace is shifted for clarity.

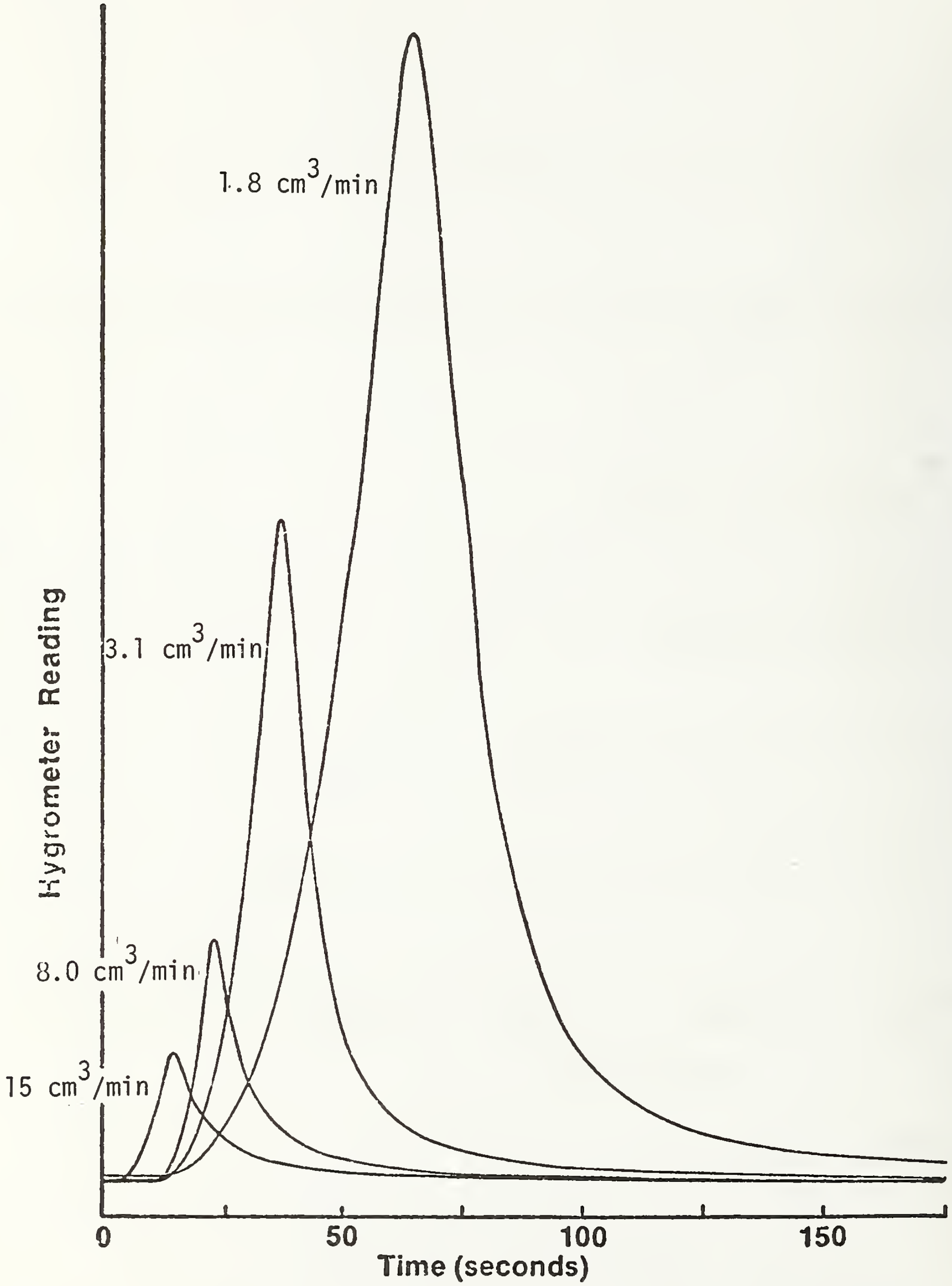

Figure 6. Variation in detector response to carrier gas flow rate for 100-μg water injection.

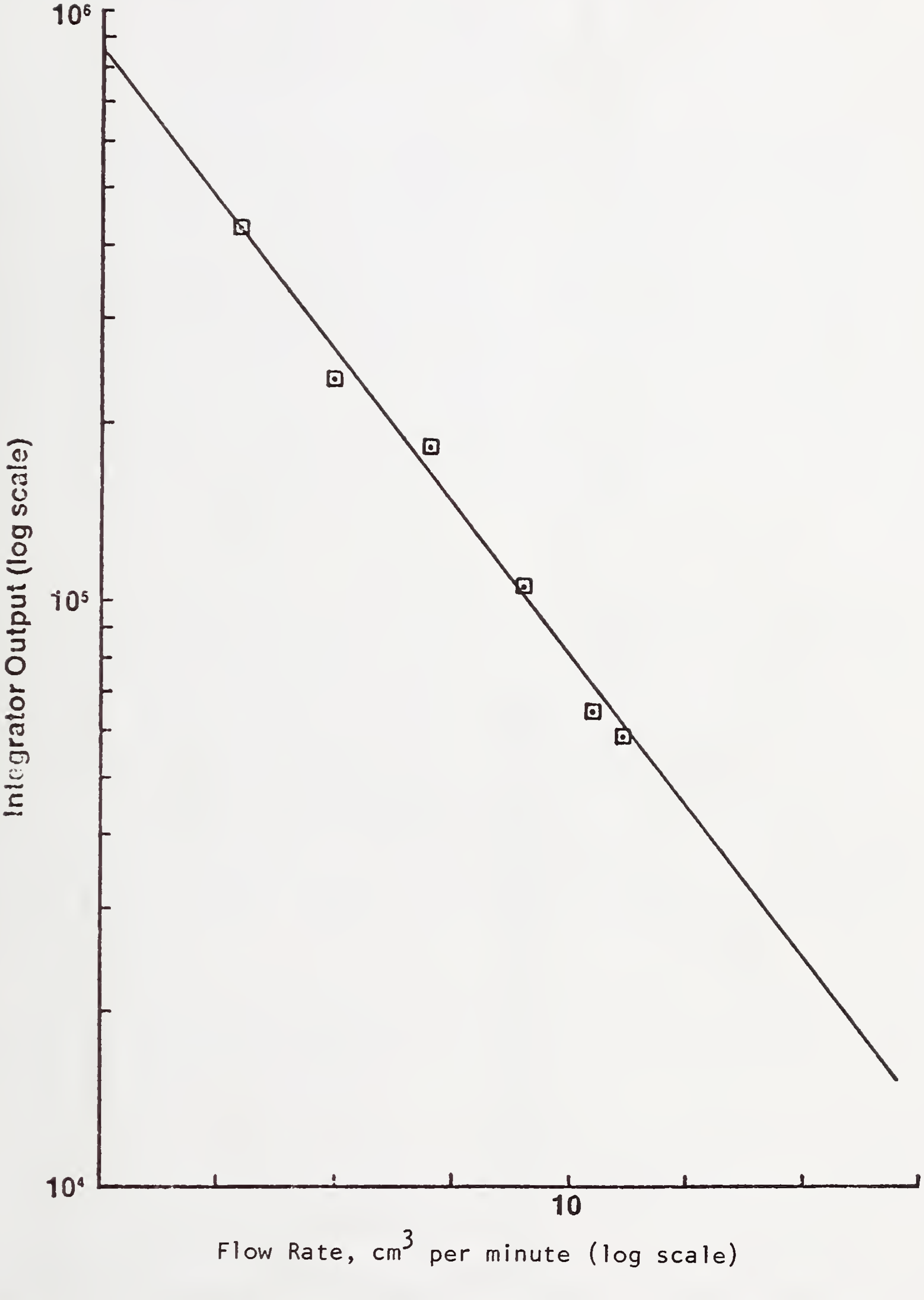

Figure 7. Integrated output as a function of carrier gas flow rate for constant for 100-μg water injection.

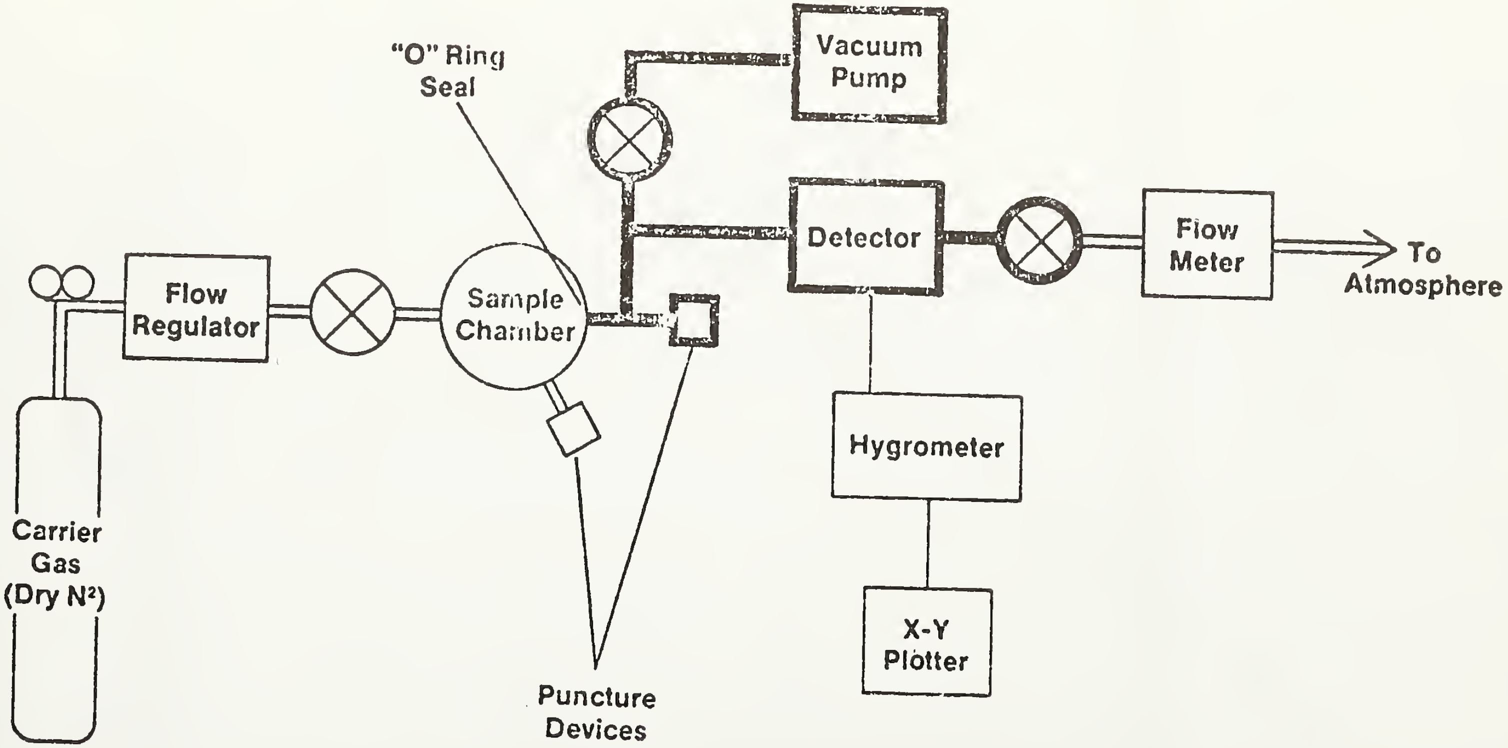

Figure 8. A schematic diagram for a vacuum-sampling procedure. The part of the flow system in heavy outline is first evacuated. When the pressure falls to 10 µm, the vacuum pump is isolated, and the test procedure is applied.

5.3 Analysis of Water Vapor in Purified Gas Systems

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In many scientific and technical activities, there is need for purified gases which have low concentrations of water vapor and other oxygen containing gaseous species. Atmospheres such as hydrogen and argon are used to reduce oxygen species or to establish an inert environment. Although there are many accepted practices for gas purification, there is considerable controversy concerning the accurate and reliable analysis of the minor impurity species in the gas at levels in the ppm range or below. Concentrations at these levels, however, are significant in materials processing activities, particularly of semiconductors. We discuss two methods of gas analysis that are appropriate analytical techniques under specific conditions – molecular beam mass spectroscopy (MBMS) and solid state electrochemistry.† In describing each of these methods, we will indicate the conditions under which they are appropriate, briefly describe the method, indicate problems of calibration and correction factors, and present typical results.

Molecular Beam Mass Spectroscopy

The Molecular Beam Mass Spectroscopy Method (MBMS) is suitable for analyzing steady-state gas flow systems with flow rates $\gtrsim$1 liter per minute, pressures $\sim$10 to 1000 Torr and gas temperatures from room temperature to $\sim$1000°C. The main features of the sampling system, shown in figures 1 and 2, are: a source region (i.e., the gas to be sampled); a sampling orifice (0.08-mm diameter-conical nozzle); first stage chamber at a pressure of $\sim 10^{-3}$ Torr; skimmer orifice (0.12 mm) and second state chamber at a pressure of $\sim 10^{-5}$ Torr; detector slit (13 mm); and detector chamber (10^{-7} Torr) with mass analyzer. The source gas expands through the sampling orifice into the first stage where the skimmer transfers the center of the free jet into the second stage to form a molecular beam. After mechanical chopping of the beam in the second stage, it passes into the third stage where it is phaselock-detected by a quadrupole analyzer.

We consider three important aspects of the sampling process: influence of a skimmer location on the beam density, the signal-to-background ratio, and the change in composition upon sampling.

Beam Density

A movable skimmer orifice is valuable for characterizing the dynamics of the sampling process. For a conical nozzle the variation of the gas density

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† General references, describing the techniques, are given in the Bibliography at the end of this paper.

along the beam axis, $\rho_{r=0}$ was found to be a simple function of the skimmer-sampling orifice distance (fig. 3) and may be represented as:

$$\rho = B/(\varepsilon - \varepsilon_c)^2 ,$$

where $\varepsilon = z/d^*$ is the reduced skimmer position (where z is the skimmer location, d^* is the sampling orifice diameter), ε_c is the intercept in figure 3 and B is a constant which depends on the source gas properties. To obtain the same beam density for different source gases, the skimmer must be located at different positions along the beam axis, and this location may be predicted from a single experimentally determined parameter for each source gas condition.

Background and Accuracy

There may be significant differences between the composition of the beam and the background in the detector chamber under steady-state sampling conditions. This may result from differences in the pumping speeds, leak rates, and adsorption and desorption kinetics for the species in the detector chamber. These differences are illustrated by comparing the unchopped (dc) and the chopped (ac) signals as is shown in figure 4 and table 1 for the mass spectrum of air. The dc signal corresponds to a lower oxygen and a higher argon concentration than for the air composition due principally to the different pumping speeds for the two species; however, the ac signal gives a faithful representation of the expected air composition. In addition, it is noted that some species, such as water vapor clusters (masses 55, 73), are detected only in the ac mode since these species decompose upon collision with the walls of the chamber.

Composition Change Upon Sampling

Composition changes upon sampling may arise for the following reasons: pressure diffusion separation, skimmer interference, background penetration, and molecular diffusion from the beam. The latter three are most important in the present sampling procedure. Of particular interest is the sampling of minor gaseous species in a gas of significantly different mass, for example, heavier gas molecules in hydrogen or helium. Figure 5 shows the enhanced composition for a source gas of hydrogen containing 1-percent nitrogen or 1-percent argon and compares the observed enhancement with predictions based on molecular diffusion. For smaller skimmer-nozzle separation distances, there is evidence of skimmer interference, and for larger distances there is evidence of background penetration. At intermediate distances the mass enrichment is close to that predicted for molecular diffusion. The lighter hydrogen molecules with higher thermal velocity diffuse more rapidly from the center of the beam causing relative enhancement of the heavier species.

Typical Results of Palladium-Diffused Hydrogen

We have measured the purity of palladium-diffused hydrogen and have evaluated the interaction of this gas with a quartz chamber at temperatures from room temperature to 900°C. Typical spectra are shown in figure 6 with a summary of results given in table 2. The minimum level of water vapor detected is a few ppm, with substantial increases when hydrogen interacts with quartz at an elevated temperature. The latter is due to the reduction of silicon dioxide (and the concurrent appearance of silicon oxide gas) as well as outgassing of the quartz.

An increased sensitivity for the detection of polar gaseous species has been achieved by the use of a trapping-desorption technique. The purified gas was flowed for 26 h over a molecular sieve trap cooled in liquid nitrogen, and then the trap was desorbed over a 4-h period by heating to 450°C. The resulting spectrum shown in figure 7 allows semiquantitative detection in the ppb level.

Solid-State Electrochemical Cells

Solid-state electrochemical determination of water vapor in hydrogen provides a valuable check for the MBMS techniques. Solid-state electrochemical cells are extremely sensitive for the detection of the oxygen chemical potential, but they do not detect individual molecular species. The gas sample must be introduced into a cell at a temperature of 600 to 1200°C. Relatively small samples may be used ($\sim 1\ cm^3$) or flow systems may be analyzed with a response time of a few seconds for hydrogen-water gas mixtures.

Basic Cell and Electrolyte Considerations

There are a number of solids that exhibit high ionic conduction, exclusive of electronic conduction, over a wide range of temperature and composition. Such solids form the basis of a powerful technique for the selective detection of a specific chemical potential by the selective transport of one ionic species. For example, certain solid oxides such as zirconia, ZrO_2, or thoria, ThO_2, with additions of heterovalent oxides, such as calcia, CaO, and yttria, Y_2O_3, show high selectivity for the conduction of oxide ions and form the basis for oxygen solid-state electrochemical cells. Such a cell is illustrated in figure 8 with the relevant electrode reactions which involve oxygen gas, oxide ions, and electrons at the electrolyte gas interface. Porous platinum electrodes at this interface catalyze the chemical and the electrode reaction. When the conduction in the electrolyte is exclusively ionic, the Nernst equation and Faraday's law may be applied to the cell:

$$\varepsilon = RT/4\ F\ \ln\ (P_{O_2}''/P_{O_2}')\text{: The Nernst Equation (oxygen sensor)}$$

$$\frac{I}{4F} = \frac{dn_{O_2}}{dt}\ \text{: Faraday's Law , (oxygen pump)}$$

where ε is the open circuit EMF, F the Faraday, n_{O_2} the moles of oxygen, and P_{O_2}' and P_{O_2}'' the oxygen pressure at the respective electrode interfaces.

If the oxygen pressure at one of the electrodes is known, the measurement of ε of the cell will determine the unknown pressure using the Nernst equation. If an EMF is imposed on the cell and the electron current in the external circuit is measured, then the number of moles of oxygen "pumped" from one compartment to the other may be obtained from Faraday's law. Thus, such a cell can act as a sensor or as an oxygen pump. (It is worth noting that the current of 0.1 mA for 4 s corresponds to the transfer of $\sim 10^{-9}$ moles of oxygen!) These two relations, however, are valid only for pure ionic conductivity; any electronic conduction leads to an internal electron leakage current which tends to short out some of the open circuit EMF of the Nernst equation as well as invalidating Faraday's law. As a consequence, there is a vital interest in the range of temperature and oxygen pressure over which the conduction is essentially completely ionic. This range is referred to as an electrolytic domain. At very low and at very high oxygen pressures, slight deviations from a perfect stoichiometric compound introduce electrons or electron holes in sufficient quantity to invalidate the use of the above equations. Thus, one should determine that a particular electrolyte is being used within its established electrolytic domain.

The detection of ppm of water vapor in purified hydrogen corresponds to very low oxygen potentials. Referring to the literature for the equilibria constants for the reaction,

$$H_2 + \frac{1}{2} O_2 = H_2O; \; K = P_{H_2O}/P_{H_2} P_{O_2}^{1/2}$$

for temperatures from 600 to 900°C, one establishes that ppm levels of water correspond to oxygen pressures of 10^{-25} to 10^{-35} atm. At these temperatures in the presence of the catalytic porous platinum, one expects equilibrium to be attained. These pressures are outside to the electrolytic domain for zirconia base electrolytes but within the domain for thoria base electrolytes which extend to ppb range for water in hydrogen. The results for measurements made for palladium-diffused hydrogen are shown in table 3 and indicate a few ppm water vapor. Of particular significance is the fact that the ratio of water to hydrogen is, within experimental error, independent of temperature and this result indicates that there is no significant outgassing of the electrolyte tube. These levels of water vapor are in good agreement with the MBMS results.

SUMMARY

- For a conical nozzle-beam-sampling system, the gas density depends upon the skimmer-nozzle distance and this dependence may be expressed with a single characteristic parameter for each source gas condition.
- For different source gas conditions, the skimmer must be located at different positions in order to achieve identical beam pressures.
- Molecular beam chopping is necessary to obtain accurate analysis since the background is not representative of the source gas composition.
- Mass enhancement may be predicted from molecular diffusion for specific locations of the skimmer position.
- MBMS measurements indicate that palladium-diffused hydrogen contains a few ppm of water and, upon interaction with quartz at elevated temperatures, outgassing and reduction leads to higher water levels.

- These levels of water vapor in palladium-diffused hydrogen were also detected using a solid-state electrochemical technique.

ACKNOWLEDGMENT

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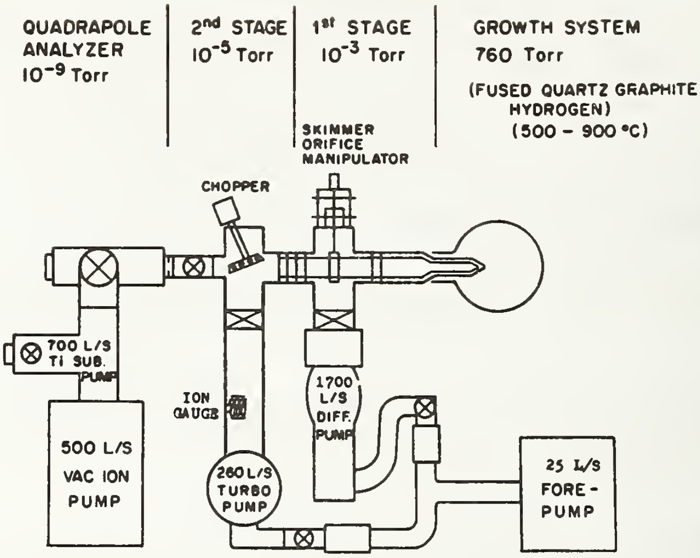

Figure 1. A schematic representation of the molecular beam-mass spectrometry system.

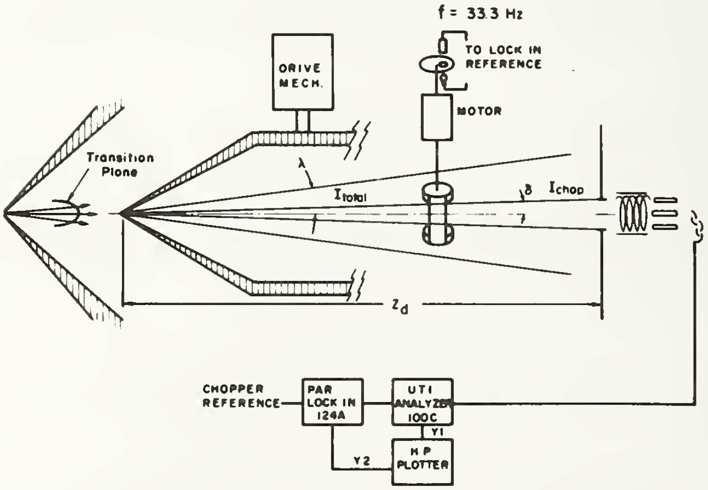

Figure 2. The interior structure of the molecular beam-mass spectrometry system.

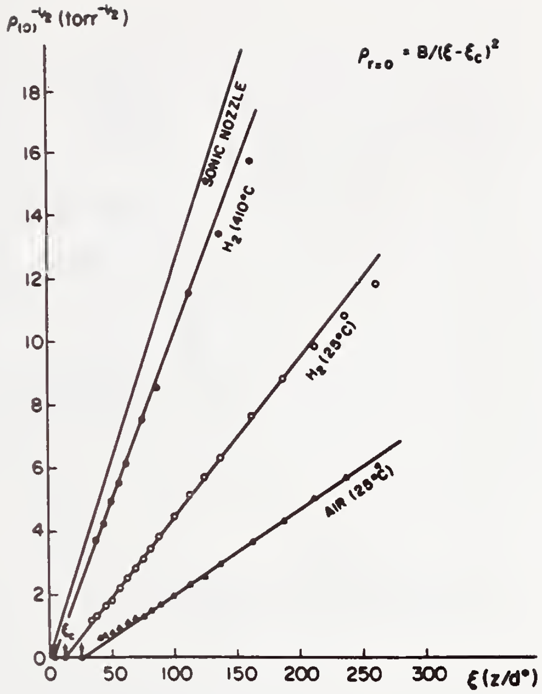

Figure 3. Dependence of gas density on the skimmer-orifice distance.

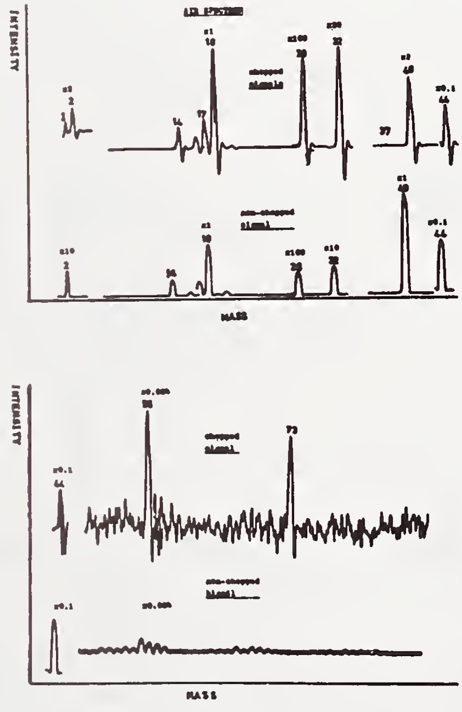

Figure 4. Air composition spectra measured by chopped and unchopped modes of detection.

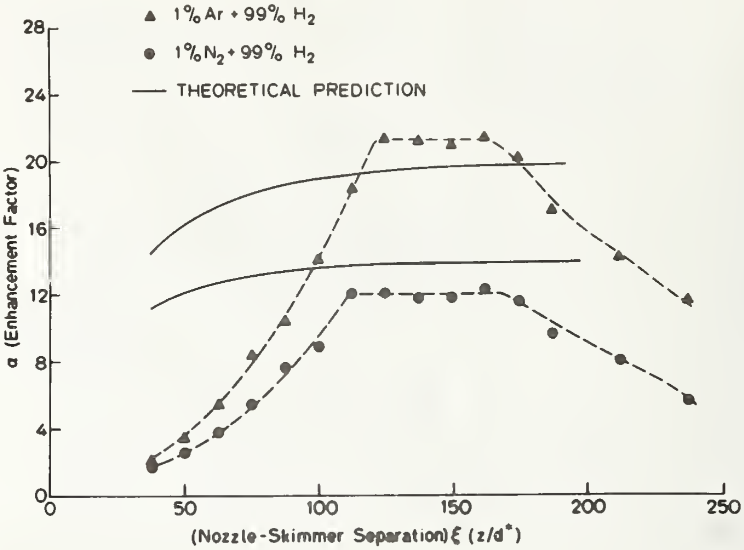

Figure 5. The mass enhancement factor, α, represented as a function of nozzle-skimmer separation.

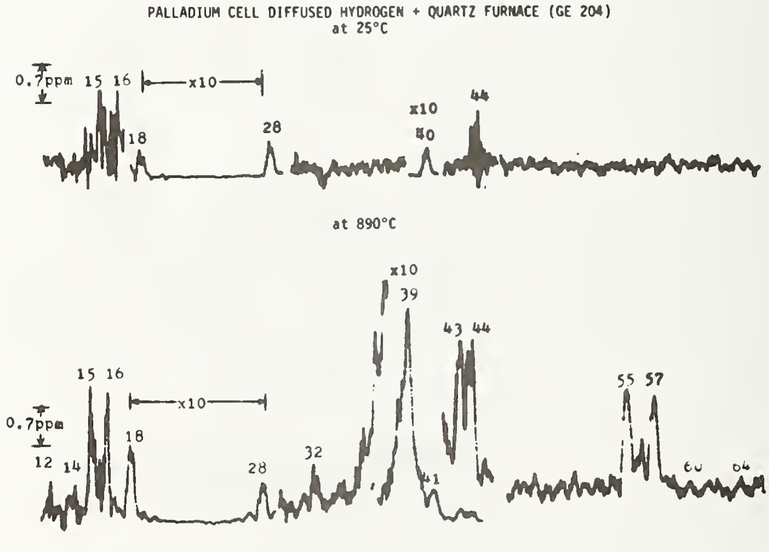

Figure 6. Comparison of the mass spectrum of palladium cell-diffused hydrogen plus quartz system at 25°C and 890°C.

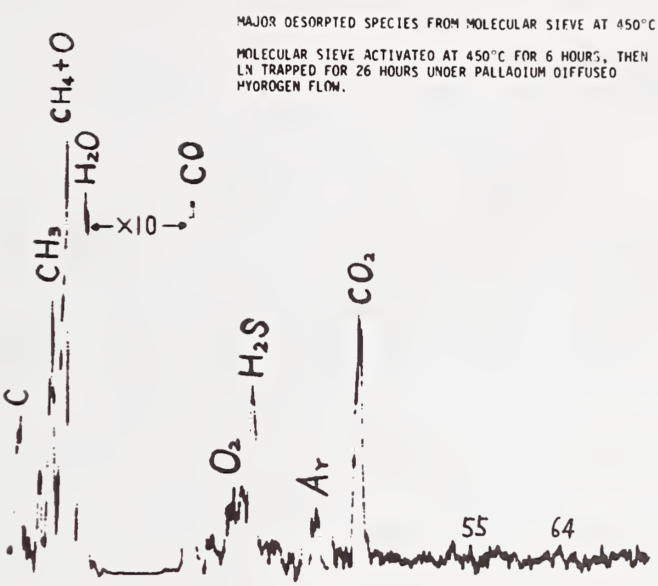

Figure 7. The desorption spectrum of molecular sieve trapped hydrogen.

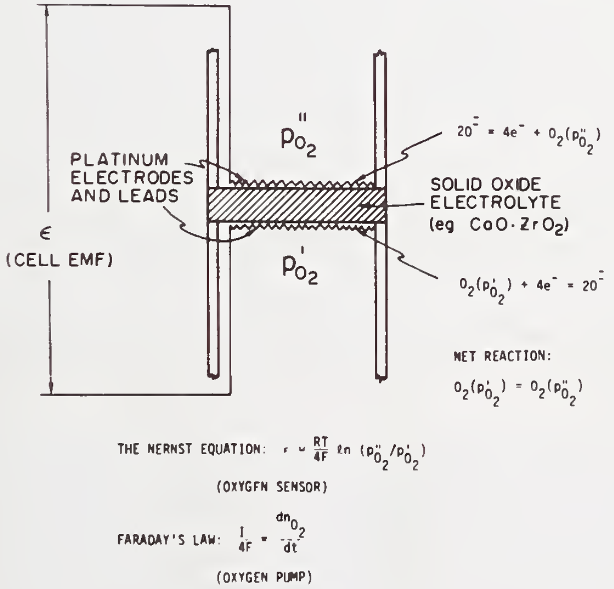

Figure 8. A schematic representation of a solid oxide electrolytic cell.

Table 1. Comparison of the Chopped and the Non-Chopped Mode of Detection for an Air Sample.

	STANDARD	CHOPPED	NON-CHOPPED
N_2	1	1	1
O_2	26.8%	23.1%	12.7%
Ar	1.2%	1.5%	4.9%
CO_2	0.042%	0.049%	0.26%
H_2	0.64ppm	0.44%	13.5%
H_2O	-	1.22%	2.5%
$(H_2O)_3H^+$	-	40ppm	-
$(H_2O)_4H^+$	-	35ppm	-

Table 2. Major Vapor Species in Palladium-Diffused Hydrogen in Contact with Quartz.

T(°C)	25	400	600	890
H_2O(ppm)	3	4	7	13
N_2(ppm)	~2	~2	~2	~2
CO(ppm)	~1	~1	~1	~1
CH_4(ppm)	2	2	2	2
Ar(ppm)	1.5	1.5	1.5	1.5
H_2S(ppb)	~50	-	-	-
K(ppm)	-	-	-	15
C_3+C_4(ppm)	-	-	~0.1	~2
SiO(ppm)	-	-	-	~0.8

Table 3. Measured Values of the Cell EMF and Corresponding Values of $\log_{10}P_{O_2}$ and P_{H_2O} and P_{H_2O}/P_{H_2}.

T(°C)	emf(V)	$\log_{10}P_{O_2}$	P_{H_2O}/P_{H_2} (X 10^6)
600	0.5295	-34.72	3.7
700	0.6175	-31.80	3.2
800	0.6860	-29.08	4.4
900	0.7605	-26.90	5.0
		AV.	4.1

5.4 Mass Spectrometric Moisture Analysis of Hermetic Semiconductor Devices by Acetylene Conversion*†

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Quantitative mass spectrometric gas analysis was developed during and following the second world war with the introduction of commercial gas mass spectrometers by Consolidated Electrodynamics Corporation and by Westinghouse Electric Corporation. The early instruments were limited to the analysis of low molecular weight gases. The technique was rapidly advanced to perform quantitative analysis of high molecular weight gases. Early users of the instruments recognized the limitations of the mass spectrometer in analyzing some of the low molecular weight gases. In general, efforts to mass spectrometrically analyze for moisture and the halogen acids were unsuccessful. Many of the speakers at this workshop have addressed the problems of mass spectrometric moisture analysis and reported means for alleviating or minimizing factors which can influence moisture determinations.

In the determination of the moisture content of hermetically sealed electronic packages, the problem is twofold: (1) acquiring the moisture sample for analysis, and (2) analysis of the moisture sample. The approach used in this paper minimizes the risks in acquiring the sample and allows the mass spectrometric analysis to be performed by conventional mass spectrometric gas analysis methods.

The method of moisture analysis employed is that reported by Carlson and Morgan [1]. A variety of chemical reagents are known to react quantitatively with moisture and generate simple gaseous products. These reagents include: sodium or calcium hydride giving hydrogen, magnesium nitride or sodium amide giving ammonia, and calcium carbide giving acetylene. In this work, the calcium carbide-acetylene conversion was selected. The most intense mass spectrum peak of acetylene falls at mass 26. This region of the mass spectrum is usually free from background and other gas interferences. In addition, the physical and chemical properties of acetylene make it a much-preferred gas over moisture for quantitative mass spectrometric analysis.

The essential features of the system used for moisture and total gas analysis of hermetic electronic devices are shown in figure 1. The device is held in

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* The views and conclusions expressed are those of the authors and do not necessarily represent the official policies of the Department of Defense, Department of Commerce, or the United States Government.

† Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

a mechanical stainless steel housing equipped with temperature monitoring and heating. The device is punctured by means of a needle attached to a valve stem. Calcium carbide is enclosed in a stainless steel tube which has provisions for both heating and cooling. Valves provide the various required pumping options. The system is miniaturized to facilitate the transfer of moisture from the device to the trap. The system is attached directly to the inlet system of the 21-104 Dupont mass spectrometer and its associated inlet pumping system is used to evacuate the drive and trap.

Preconditioning of the system for device analysis includes pumping and baking at 125°C for 1/2 to 1 h, based on background monitoring. Prior to puncture of the package, the calcium carbide trap is cooled with liquid nitrogen. The device is punctured and the gas partitioned as to condensables at liquid nitrogen temperature. The condensables are retained in the trap and the noncondensables are measured as to quantity, are mass spectrometrically analyzed, and are pumped away. The trap is then warmed to 100°C for release of the gases and conversion of the moisture to acetylene. This fraction is measured, analyzed, and combined with the results of the noncondensable fraction to provide a total analysis. The calculations for water determinations allow for its conversion to acetylene, wherein one mole of water reacts to form 0.5 mole of acetylene.

The accuracy of the method was evaluated by analyzing 300 cm^3 atm gas samples of known moisture content as determined with an Alnor Dew Point Meter (see table 1). The decomposition of the hydrated salts of known water content (see table 2) was also used to test the accuracy.

Typical results obtained for four devices are given in tables 3 and 4.

In conclusion a method for the moisture analysis of hermetically sealed electronic packages has been described. The moisture is collected, reacted with calcium carbide to form acetylene, and then analyzed mass spectrometrically as acetylene. The approach minimizes or eliminates many of the problems encountered in direct mass spectrometric analysis of moisture. A complete analysis of all gases is made and the total gas quantity is determined. The method allows for calculation of device fill pressure and measurement of device leak rate.

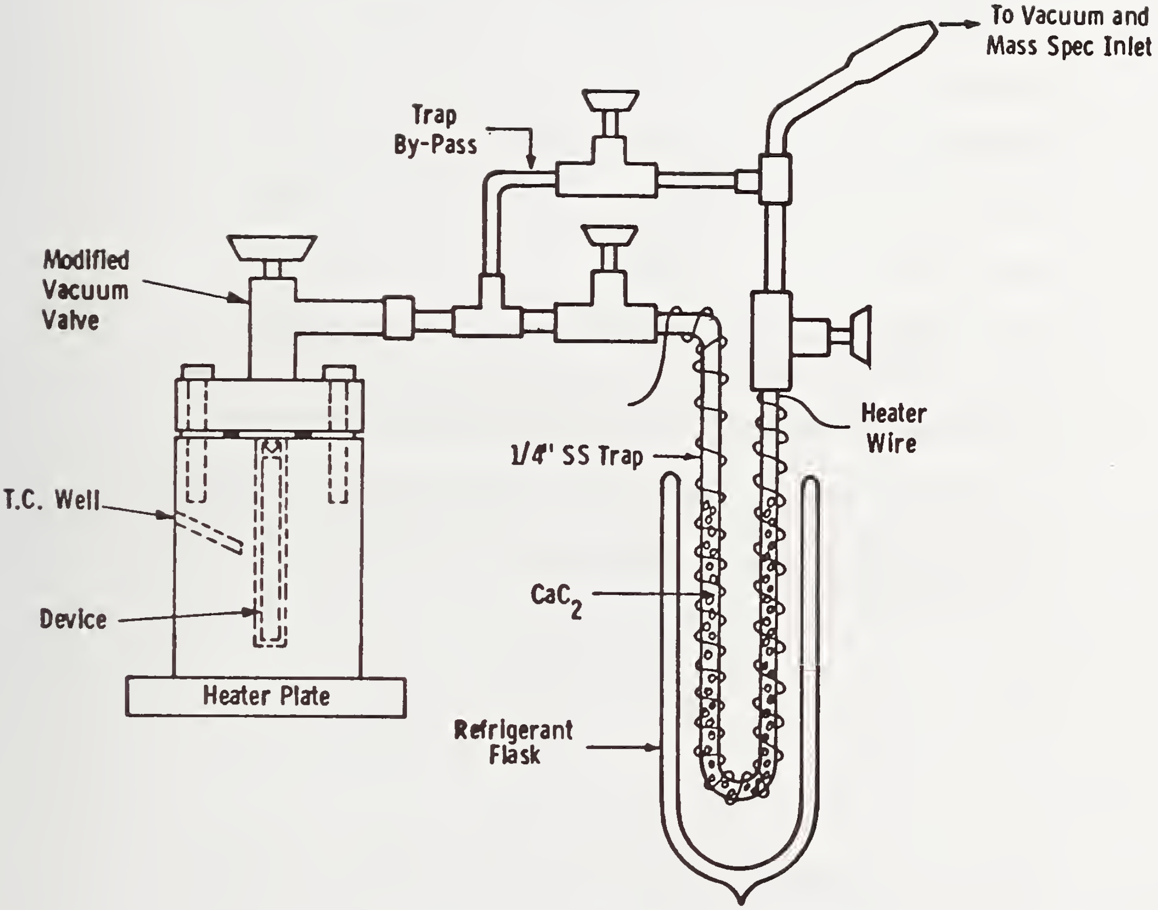

Figure 1. Trapping system for concentration and conversion of water to acetylene.

Table 1. Determination of Moisture in Gases.

Gas	Vol (ppm) Alnor Method	Mass Spec. Method
Nitrogen	<5[(a)]	2[(c)]
Nitrogen	28	25
Nitrogen	90	92
Dry Air	655	655
Ambient Air	8600	7950
Ambient Air	15,000[(b)]	13,700

(a) Liquid N_2 boil off - moisture content below Alnor.

(b) Moisture content above Alnor - determined by hygrometry.

(c) Sensitivity of mass spectrometric method approximates 1 μg of water.

Table 2. Determination of Moisture in Hydrated Salts.

Salt	mg	wt % H_2O Found	Theoretical
$BaCl_2 \cdot 2H_2O$	53.18	14.35	14.74
$K_2Fe(CN)_6 \cdot 3H_2O$	155.15	12.46	12.78

Table 3. Typical Results of Mass Spectrometric Analysis With Acetylene Conversion of Water. (Total gas quantity of approximately 0.5 $cm^3 \cdot atm$)

Mole %

He	CH_4	N_2	Ar
12.92	0.05	87.02	0.01

ppm

H_2O	Ethane	Acetone	Benzene
58	88	350	25

Total gas = 0.50 $cm^3 \cdot atm$

Device #2 Analysis

Mole %

He	CH_4	N_2	Ar
10.23	0.07	87.69	0.01

ppm

H_2O	Acetone	Benzene
2220	125	45

Total gas = 0.52 $cm^3 \cdot atm$

Table 4. Typical Results of Mass Spectrometric Analysis With Acetylene Conversion of Water. (Total gas quantity of approximately 0.02 $cm^3 \cdot atm$)

TABLE IV

Device #3 Analysis

Mole %

H_2	CH_4	N_2	C_2H_4
3.74	0.33	95.13	0.09

ppm

H_2O	CO_2	Propane	Acetone	Benzene
300	400	2400	3600	400

Total gas = 0.022 $cm^3 \cdot atm$

Device #4 Analysis

Device leaked.

Leak rate = 7.7×10^{-6} $cm^3 \cdot atm$

5.5 Moisture in Packages — A User's Viewpoint

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INTRODUCTION

Moisture-related problems in semiconductor packages persist in spite of the many documented accounts describing such experiences. This is a rather disturbing situation but to a certain degree is understandable. To some, the literature describing these problems was enough to convince them that measures should be taken to eliminate moisture from their product, yet others have been reluctant to do so. The reasons for this reluctance, in the writer's opinion, are varied and range from simple disbelief ("we're making too much of this") to those who raise honest questions concerning threshold levels of moisture, the validity of moisture measurement techniques, the role of polymeric materials in packages, the effects of contamination, hermeticity, etc. With these considerations in mind, the following paper discusses the histories of two moisture-related problems in semiconductor devices with the purpose of detailing some of the many factors involved in corrosion phenomena and, in particular, the threshold for such reactions and how subtle differences in "identical" parts can imply to some that moisture is not a serious problem.

Moisture in Integrated Circuits

In the early 1970s corrosion problems were noted with thin-film nichrome resistors used in some integrated circuits [1-3]. Extensive analysis by this author and several groups of other investigators from various agencies concluded that the problem was related to moisture trapped within the packages. This failure mode escaped detection at the manufacturing plant, incoming inspection, extensive burn-in tests, and several electrical tests. The reason for the escape, in retrospect, was simple: none of the tests energized the devices at room temperature or below for periods of time of more than a few seconds . . . one of the "advantages" of high-speed test equipment. Unfortunately, the failures were not detected until system operational temperature-cycling tests were performed or in a few cases in the field. The impact on the program was enormous and was a key factor in the decision to pursue the failure mechanism in as much detail as possible. This and other related test results are summarized in the following paragraphs.

Construction Details

The problem devices were triple three-input NAND gates utilizing thin-film stabilized nickel chromium resistors 20 nm thick. A protective coating of evaporated silicon monoxide was used as an overglaze for scratch and particle protection. The parts dissipated only a few milliwatts and were encased in a 1/4- by 1/4-in. ceramic package which utilized a high-lead content, low temperature glass for sealing purposes (typical trade name "Cerpak"). The free air internal volume varied considerably (0.005 cm^3 to 0.015 cm^3) because of the glass application method (sprayed). Sealing was accomplished by using a conventional belt furnace. The ambient gas in the oven was principally nitrogen or nitrogen and oxygen (50/50). The temperature/time profile was adjusted to devitrify the low temperature glass (peak temperature was ∿500°C), a process which reportedly protects the seal area from possible damage due to subsequent thermal shock tests.

Failure Mode

The failures were noted in operational system tests at ambient temperatures ranging from -10°C to ∿30°C. The devices which failed did so in a time frame of from a few minutes to several hours. Very few lasted beyond ∿100 h. Conversely, high-temperature burn-in at 125°C for several hundred hours did not produce any failures related to this phenomenon. Visual examination of failed resistors by optical means indicated a circular void formation ranging from ∿0.0001 in. to several thousandths of an inch (fig. 1).

Failure Mechanism

In the early portions of the investigation, several mechanisms were pursued (diffusion, chemical, electromigration, etc.) to no avail. None of these could explain the dependence of these phenomena on the inverse relationship with temperature and the fact that only sealed packages had resistor failures. It was felt that the mechanism was probably associated with internal materials and that the most likely one would be the gaseous environment. This preliminary conclusion prompted several gas analysis tests involving the package ambient. The initial results from various testing laboratories were perplexing to say the least. The differences between the normally expected gases were slight yet the difference in moisture content varied by orders of magnitude. Only one of the laboratories performed water analysis as a separate test and could also make these measurements at elevated temperatures. The elevated (100°C) temperature measurements produced the highest levels and much controversy since previous room temperature measurements had indicated no moisture at all. This controversy continued for some time and subsided somewhat through the use of a miniature surface conductivity sensor. The sensors indicated a dew point for these parts at or near room temperature which correlated closely with the failure history of these devices. Several ensuing experiments confirmed that the "disappearance" of the nichrome was indeed related to moisture, applied power and minute cracks, and/or pin holes in the silicon monoxide glassivation at or near the nichrome in aluminum step (fig. 2).

As a result of these findings, changes in the manufacturing process and electrical test procedures were instituted. They are summarized as follows: (1) providing a crack and pin-hole-free two-layer glassivation system of silicon dioxide over silicon oxide; (2) sealing in a dry package, referred to as a "hot cap" seal process; and (3) powered temperature-cycling screen tests at the component level (-20°C to +70°C).

Discussion and Conclusions

The extent and details of the entire investigation encompassed a two- to three-year time frame and involved as many as six large semiconductor companies and most of the government agencies at one time or another. The key points uncovered in the study were as follows: (1) moisture could be generated and trapped within a sealed package which had been processed by "acceptable" practices; (2) it was difficult to subsequently detect and quantify the levels of moisture by conventional methods (mass spectroscopy and gas chromatography); and (3) miniature surface conductivity sensors were useful in dew point analysis for these and other small volume packages, helpful in defining the failure mechanism (see figs. 3 through 5 for a pictorial summary) and, finally, used to determine the source of moisture [4].

Although the mechanism of "disappearing" nichrome was defined during this investigation, a controversy remained over the use of the mass spectrometer for the measurement of moisture within small volume devices. That controversy is apparently still with us and, in the author's opinion, will persist because of the complexities associated with measurement techniques and the interpretation of the data obtained from such methods. These issues and others will be addressed in more detail later in this paper.

Moisture in Hybrid Devices

More recently, problems involving moisture entrapment in hybrid devices have been detailed which address various thresholds for gold migration (fig. 6) as a function of relative humidity, type and quantity of contaminant, and voltage [5]. This study was initiated because of (1) poor correlation between package moisture content and gold dendrite growth, (2) poor correlation between moisture measuring agencies, and (3) a definite correlation between metallization failures (shorts for dendrite growth) and an electrical "dew point" noted during temperature tests.

The highlights of this study are discussed in the following paragraphs with emphasis on the effects of the variables on the migration rate and how they alter the threshold for such reactions.

Experimental Studies

Initial experiments were performed on "as-processed" alumina substrates with an unknown degree of contamination. A small drop of distilled water was placed between two parallel metallization runs while they were biased from 2 to 8 VDC. Gold migration was noted in this case, while none was noted on substrates which had been rigorously cleaned. Scanning electron microscope and E.D.A.X. analysis revealed active salts in the first substrate, namely, sodium and potassium chloride. In subsequent experiments, the substrates were intentionally contaminated with known quantities of sodium and potassium chloride and potassium iodide (an etchant used for gold metallization). These contaminated specimens were then exposed to various saturated salt constant humidity environments ranging from 10- to 98-percent RH at 23°C. Leakage currents and dendrite growth rates were monitored as a function of relative humidity and quantity of contaminant. The results are shown in figures 7 through 10 and establish a threshold for migration which ranges from 44- to 98-percent RH at 23°C depending on the type and quantity of contaminant. No migration has been detected to date (∿4000 h) below the relative humidity as noted by the dotted lines on each of the curves. Additional experiments involving the threshold voltage under worst case (crystal bridging the 0.005-in. gap between the runs) conditions indicate a value of ∿0.6 VDC

(98-percent RH at 23°C) while mildly contaminated parts in lower relative humidities (70 percent at 23°C) indicate values as high as 50 to 100 VDC. In all of these cases, the key factor is simply the presence or absence of ionic mobility, suggesting that all of these variables must be above a given value at the same time in order to initiate migration or other types of corrosion phenomena.

Discussion

It became apparent early in this study that there are many other factors which could have a significant effect on migration or corrosion phenomena besides moisture, contaminants, and voltage. These include such considerations as spacing between runs; steady-state operating temperature (is it high enough to preclude migration by virtue of lowering the relative humidity?); thermal cycling or occasional low-temperature operation (thereby increasing the relative humidity); package volume and its effect on the total quantity of moisture available for condensation; and substrate materials which can affect the surface leakage characteristics as a function of relative humidity and, hence, corrosion or migration thresholds [6-8]. Considering the effects of these and other parameters, it is no wonder that various users of "identical" parts are distraught over differences in device performance; e.g., running parts a few degrees warmer can be the difference between success and failure or simply being above or below the threshold for moisture, voltage, contamination, etc.

General Discussion

We have in this paper attempted to illustrate part of the complex nature of corrosion phenomena associated with two common semiconductor device types: a small flatpak and a larger hybrid package. In addition to this, the literature [9-16] over the past several years has been conspicuously filled with similar accounts encompassing many military, medical, and aerospace programs. It should be noted, at this point, that corrosion is not the only problem posed by the presence of moisture in packages (e.g., inversion, surface leakage, etc). These have been intentionally omitted from this discussion, yet are mentioned at this time to provide additional impetus for (1) "drying out" semiconductor packages, and (2) aggressively pursuing cost-effective manufacturing methods which will, by design, accomplish this task. Without a conscientious effort on the part of the supplier, we, the users of such goods, will be doomed to live through these problems time and again. It is, therefore, incumbent upon those of us in the reliability community to provide well-documented, detailed data on moisture-related phenomena, how it is introduced into a package, what the acceptable levels are, and precisely what is necessary to remove it. Although much of this information has been available for some time, certain aspects remain to be detailed while others are open to interpretation. Some of these areas are discussed in the following.

Moisture Measurement Techniques

The principle techniques used today for the measurement of moisture are (1) mass spectroscopy at 100°C, (2) electrolytic hygrometry at 100°C, (3) aluminum oxide sensors, and (4) surface conductivity devices. Each of these methods have its place in moisture measurement. None is completely fool-proof, however; yet when judiciously used in combination, they can be effective in detecting and measuring moisture in packages in a cost-effective manner.

The mass spectrometer, when set up according to Thomas of RADC, can be useful in determining total water content in packages, other gas species, and desorption and absorption properties of various materials. The disadvantages are mainly its setup and test cost and its inability to distinguish between water which is absorbed and that which is in the vapor phase. In addition, the test is also destructive, making it more cumbersome to track parts through a process.

The electrolytic hygrometer measures total water only and is not useful for other gases. It also suffers from the same disadvantage as the mass spectrometer.

The aluminum oxide transducer is a nondestructive method for determining the *in-situ* moisture content of a sealed package [17]. The device typically is mounted in the package, sealed along with the rest of the circuitry, and may be monitored for moisture content as a function of each of the processing steps and periodically during use in the field. The device has excellent sensitivity and is typically monitored at room temperature. Its disadvantage to date has been its high cost (for engineering evaluations, however, its cost is not considered to be a factor) and its relatively low high-temperature limit.

The surface conductivity sensor is by far the oldest method used to detect moisture on surfaces. Its main feature is its direct readout of surface leakage, and hence, corrosion current, which we are trying to control and understand! The device, if properly instrumented, can be very useful since it also is a simple *in-situ* device, is inexpensive, can withstand high temperature-sealing operations, and hence can be placed in production quantity parts in a cost-effective manner. The main disadvantage is its lack of repeatability and poor sensitivity to low moisture levels. It is also somewhat more tedious to operate than the aluminum-oxide sensor due to the need for multiple-point temperature measurement requirements (needed usually for "dew point" measurements).

If we consider each of these methods for what they can do best, then it becomes apparent that when used in combination they enable the designer to (1) choose materials with desirable properties (low outgassing, stable temperature properties) through the use of mass spectroscopy and electrolytic hygrometry; (2) evaluate each of these materials with and without process contaminants for surface conductivity as a function of relative humidity (surface conductivity sensor approach); and (3) determine the internal moisture content of the sealed parts as they are processed and as a function of system usage (aluminum-oxide sensor or surface-conductivity sensor). Only in performing these types of detailed tests and other related tasks will we be able to authoritatively specify, to the user and manufacturer, which methods, materials, cleanliness levels, and ambient moisture levels are acceptable for long-term reliable device operation.

Hermeticity

The discussion to this point has been limited to moisture and contaminants which have been inadvertently sealed into packages. This, however, is only part of the problem since failure analyses of many devices over the past several years have indicated that faulty seals have also allowed corrosive materials to come in contact with the contents of these parts. Consequently, this raises serious questions as to the structural integrity of these enclosures and/or the validity of hermeticity test procedures and practices. Controversies surrounding these topics have periodically surfaced over the past twenty years or more and, until recently, very little has been accomplished to help alleviate them. Most of this current activity has centered around leak test correlation studies [18], moisture ingress rates as a function of air leak rates [19-21], and some improvements in the test procedures [22]. More must be done, however, in these and other related areas if we are to intelligently address the subject of hermetically sealed devices.

Until the hermeticity problems are resolved, I am afraid we will continue to have moisture-related (from in-leakage) problems in so-called hermetic devices. It certainly seems fruitless to vacuum bake parts for hours, seal them in super dry atmospheres, involving inexpensive time-consuming equipment, when on the other hand, we specify maximum leak rates for such parts that are too lenient,* to say nothing of the general apathy of the leak testing community and their lack of understanding in this area.

CONCLUSIONS

1. Corrosion phenomena in semiconductor packages is very complex. The threshold for these reactions depend on such things as the quantity of moisture; the location, type, and quantity of ionic contaminants; the applied potential; conductor spacing; electrode materials; surface resistivity of insulators; device operating temperature; chemical reactions; and the internal free volume of the package, to name a few.

2. Moisture measurement techniques, at present, are probably sufficient to detect minute quantities of moisture present in small volume devices. The problem remaining is a matter of interpretation of such levels and how they affect device reliability when all of the variables are considered (e.g., end use environment, conductor spacing, voltages applied, absorption into materials, etc.).

3. Contamination specifications need to be defined as to what is and what is not acceptable. The use of surface conductivity sensors in combination with test coupons may go a long way towards defining these levels. In addition, more needs to be understood about the role of polymers in

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* E.g., a package with an internal volume of $\sim$0.2 cm^3 has a maximum allowable leak rate of 1×10^{-7} atm cm^3 s [22]. According to ref. [20] (see fig. 11), it would take only $\sim$100 days for the dew point in the package to exceed 0°C when exposed to an ambient condition of 80-percent RH at 23°C.

packages and how they react in regards to introducing water and other contaminants into such devices.

4. Hermeticity specifications leave much to be desired in terms of adequacy, maximum allowable leak rates, and how they relate to device reliability. Without answers to these questions, I am afraid we will do little to eliminate the confusion and apathy which exists among those involved in leak testing activities.

RECOMMENDATIONS

In view of the complex nature of the problems discussed in this paper, it would seem prudent, for those involved in high reliability programs, to specify for use only those semiconductor products which have undergone the rigors of manufacture which would assure them to be as clean, dry, and hermetic as possible.

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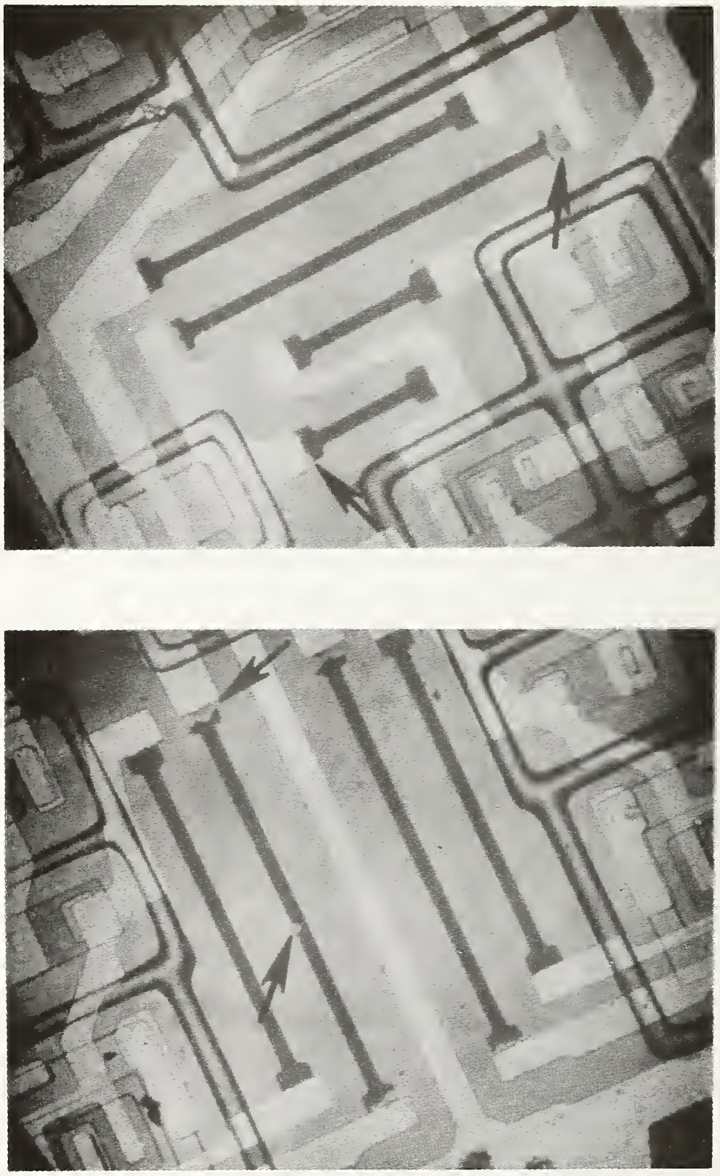
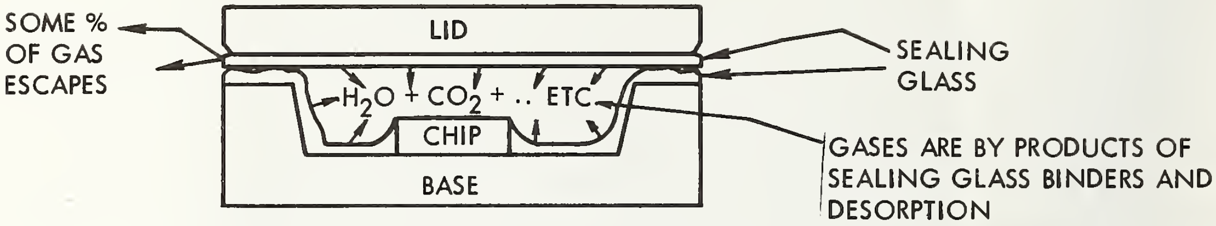
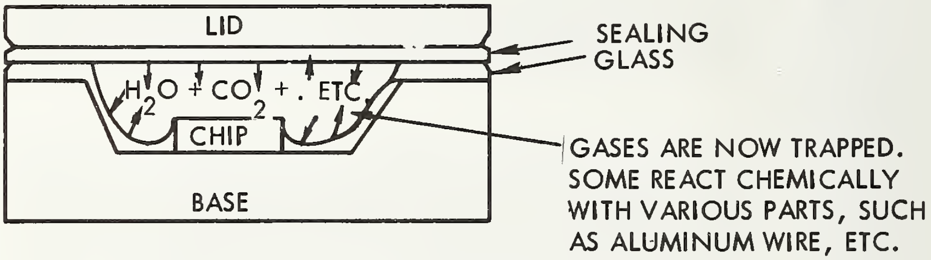


Figure 1. Photographs of 1509 NAND Gates depicting typical voids in resistors after failure at ambient temperatures ranging from -10°C to ∿30°C within times from a few minutes to several hours. Voids are indicated by locating arrows.

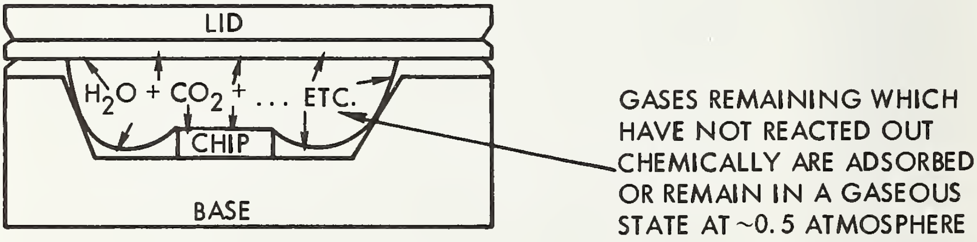
Figure 2. Scanning electron microscope photographs of cracks and holes in the silicon monoxide passivation coat near the nicrome in aluminum step of the 1509 Gate devices.



DEVICE AT SEALING TEMPERATURE
BUT NOT SEALED (~500°C)



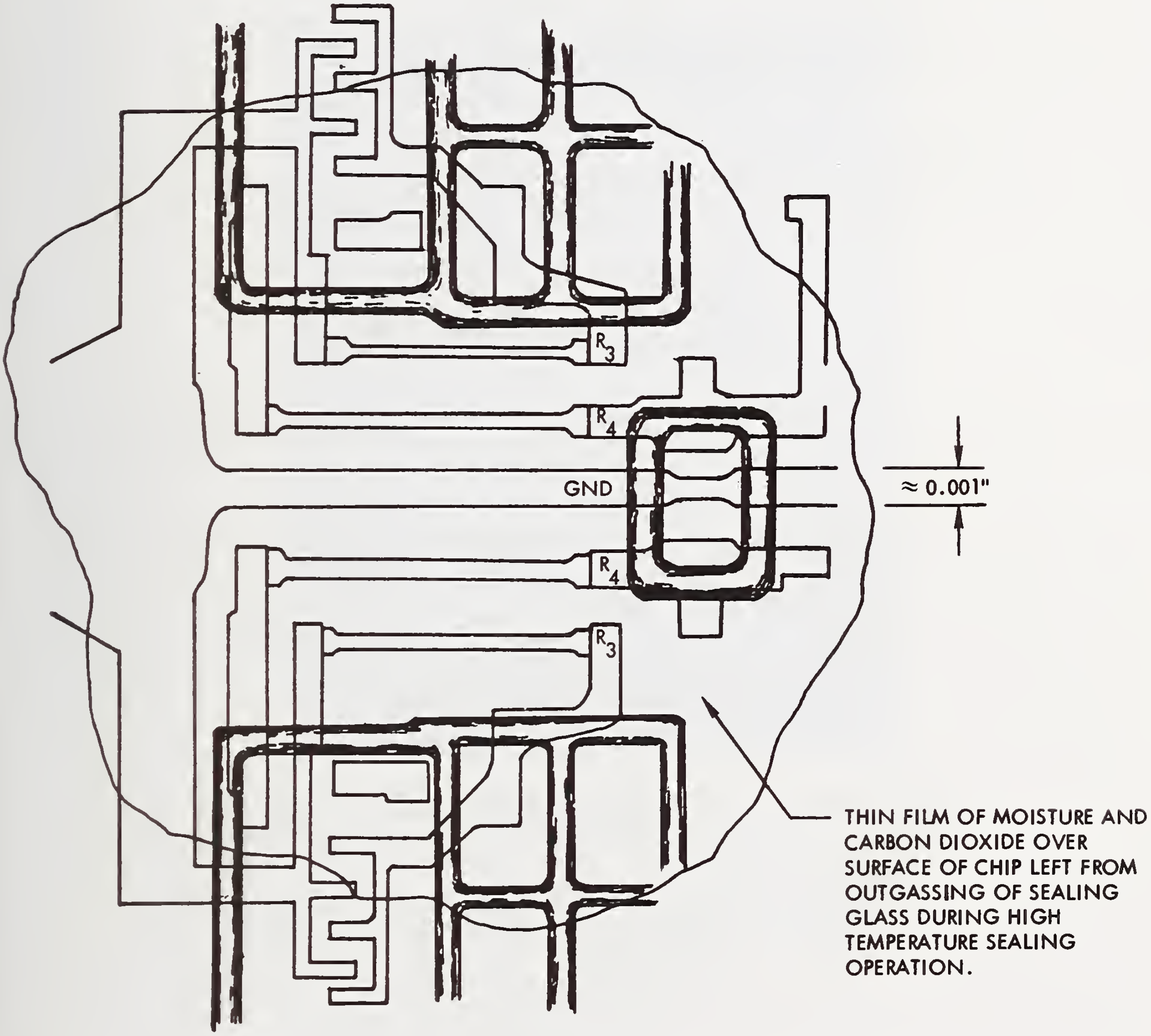
DEVICE AT ~500°C AND SEALED



DEVICE AT ROOM TEMPERATURE

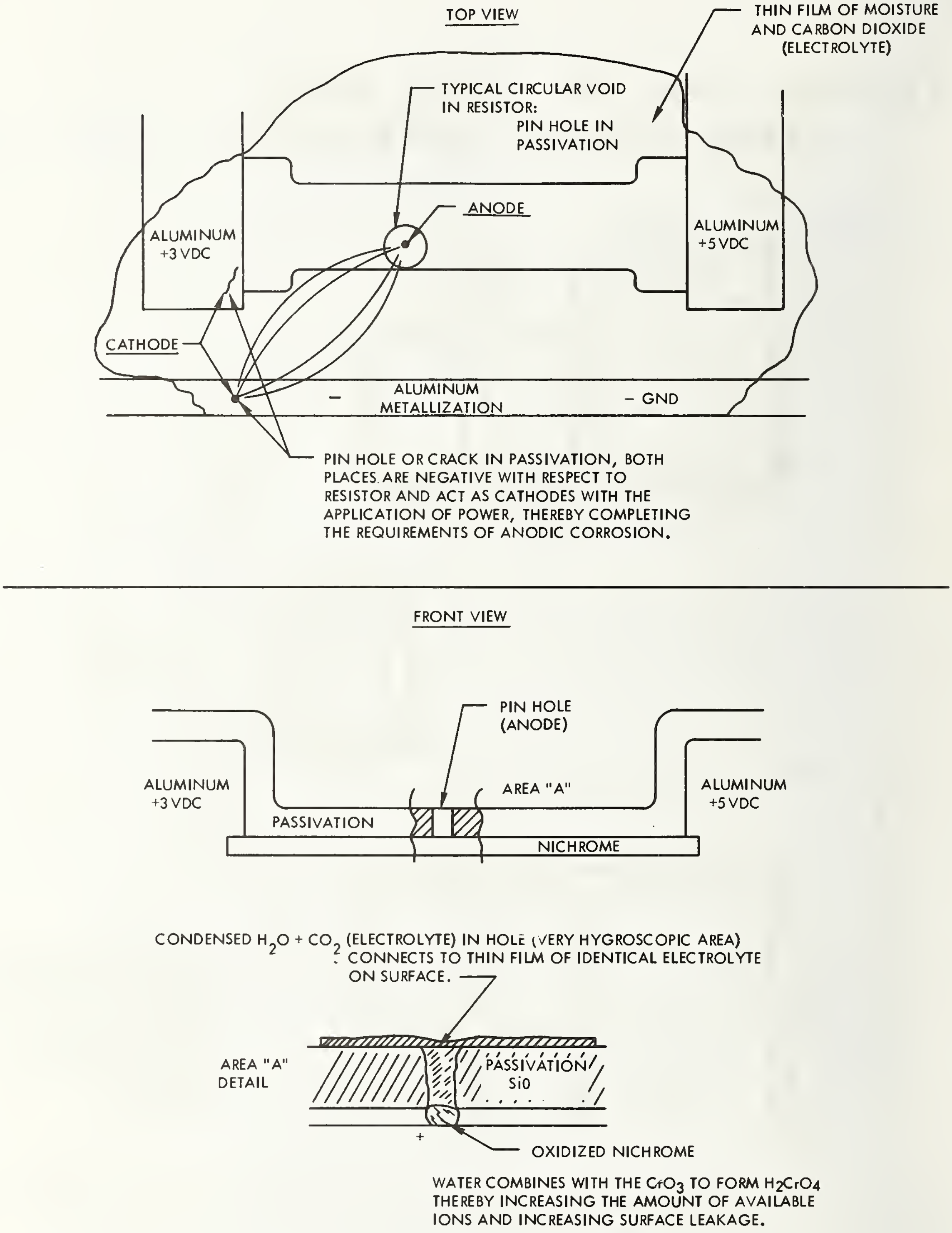
Figure 3. Model of the failure mechanism of the nicrome resistors in 1509 Gates: internal gas composition before and after seal.

PARTIAL VIEW OF 1509 GATE AFTER SEALING OPERATION



THIN FILM OF MOISTURE AND CARBON DIOXIDE OVER SURFACE OF CHIP LEFT FROM OUTGASSING OF SEALING GLASS DURING HIGH TEMPERATURE SEALING OPERATION.

Figure 4. Model of the failure mechanism in 1509 Gates: moisture and carbon dioxide distribution after sealing operation.



THIN FILM OF MOISTURE AND CARBON DIOXIDE (ELECTROLYTE)

PIN HOLE OR CRACK IN PASSIVATION, BOTH PLACES ARE NEGATIVE WITH RESPECT TO RESISTOR AND ACT AS CATHODES WITH THE APPLICATION OF POWER, THEREBY COMPLETING THE REQUIREMENTS OF ANODIC CORROSION.

CONDENSED $H_2O + CO_2$ (ELECTROLYTE) IN HOLE (VERY HYGROSCOPIC AREA) CONNECTS TO THIN FILM OF IDENTICAL ELECTROLYTE ON SURFACE.

WATER COMBINES WITH THE CrO_3 TO FORM H_2CrO_4 THEREBY INCREASING THE AMOUNT OF AVAILABLE IONS AND INCREASING SURFACE LEAKAGE.

Figure 5. Model of the failure mechanism in 1509 Gates: details of anodic corrosion associated with visible voids in nichrome resistors.

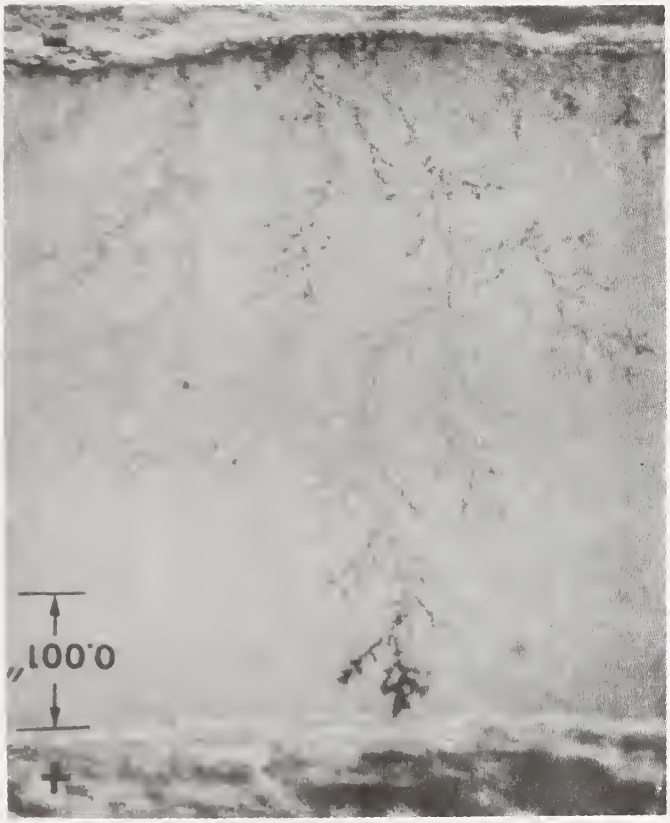


Figure 6. Gold dendrite formation noted in hybrid package between closely-spaced conductors. Alumina substrate was found to be contaminated with traces of sodium and potassium chloride while package ambient was found to contain a relatively high percentage of moisture.

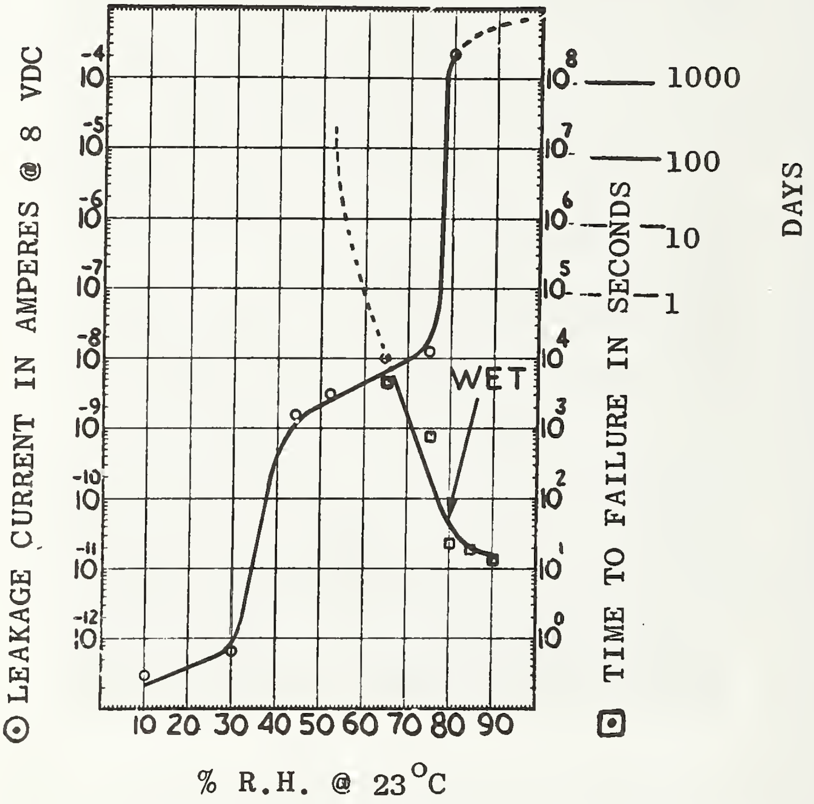

POTASSIUM CHLORIDE CONTAMINATION
WORST CASE

Figure 7. Leakage current and time to circuit failure as a function of relative humidity parallel gold metallization runs at 8 VDC bias with potassium chloride contamination.

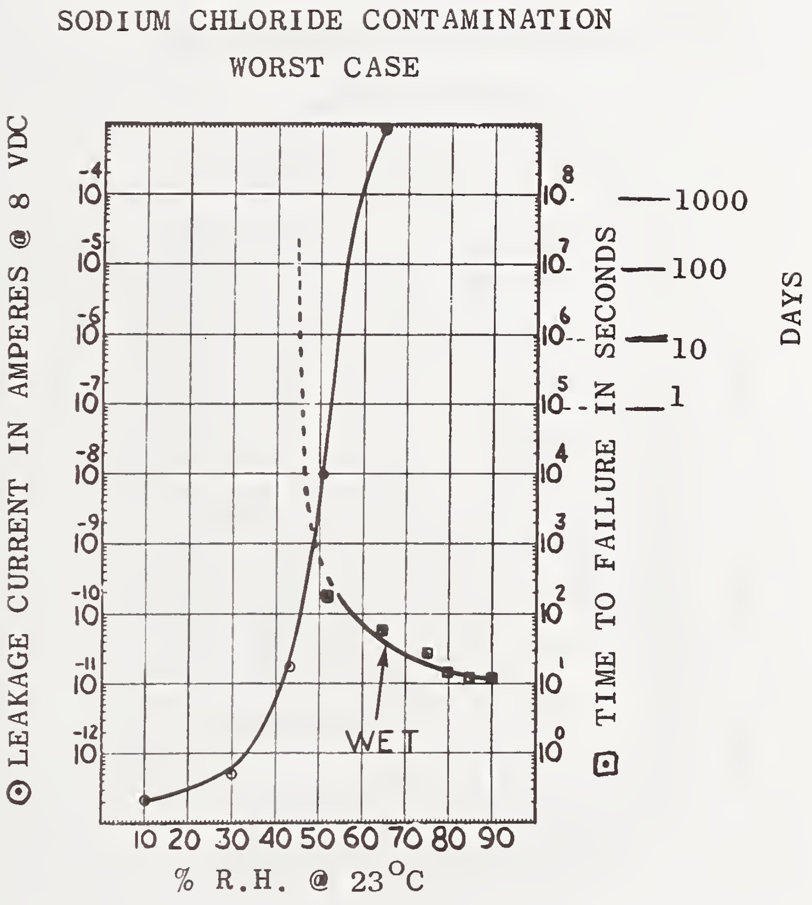


Figure 8. Leakage current and time to circuit failure as a function of relative humidity parallel gold metallization runs at 8 VDC bias with sodium chloride contamination.

POTASSIUM IODIDE CONTAMINATION

WORST CASE

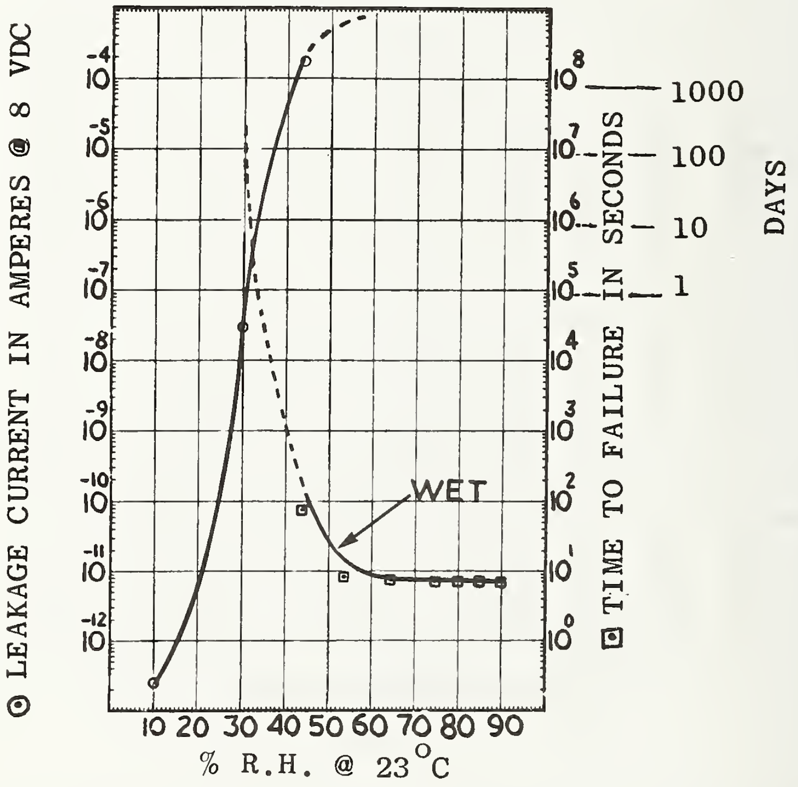


Figure 9. Leakage current and time to circuit failure as a function of relative humidity parallel gold metallization runs at 8 VDC bias with potassium iodide contamination.

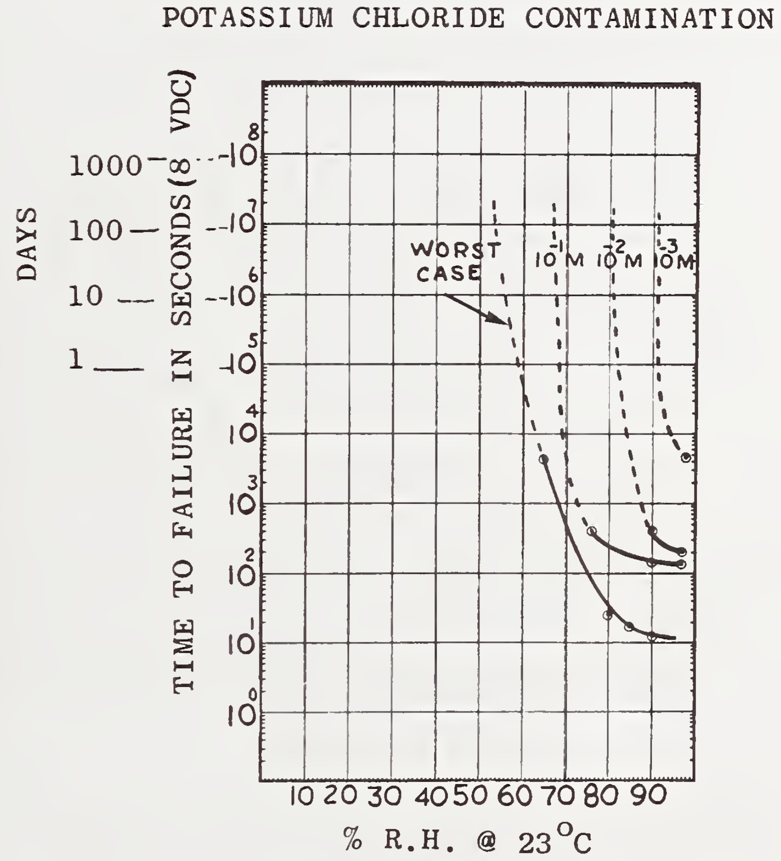


Figure 10. Leakage current and time to circuit failure as a function of relative humidity parallel gold metallization runs at 8 VDC bias with potassium chloride contamination.

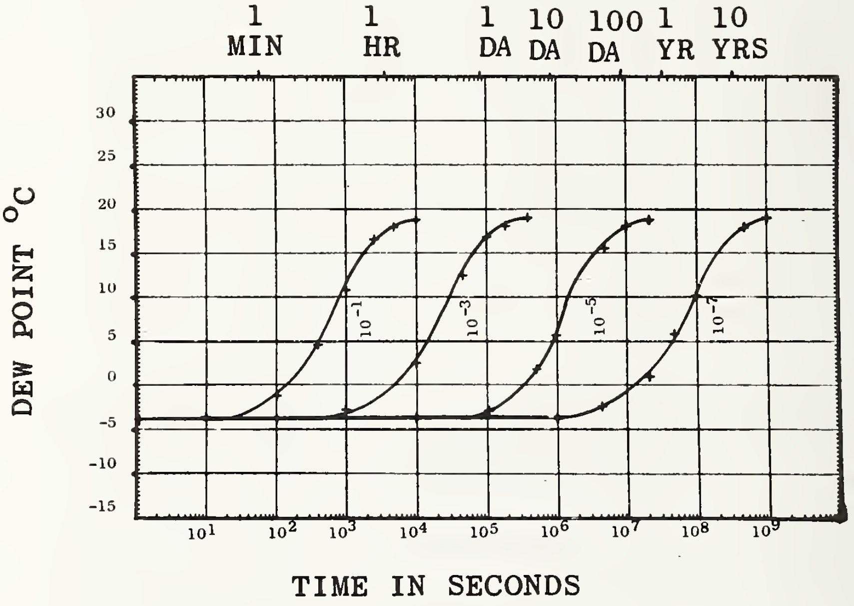


Figure 11. Change in dew point to TO-5 package *versus* time as a function of standard air leak rate in an ambient environment of 80-percent RH at 22°C.

5.6 Problems in Specifying and Measuring Moisture Content Within Electronic Devices

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To put the military specification requirement on moisture content into focus, I would like to discuss the philosophy, including intent of the specification and the inclusive nature of the MIL-STD-883 Test Method 1018 on internal water-vapor content for microelectronic devices.

The specification was prepared based on a large volume of published data relating corrosion failures to the presence of moisture within a hermetically sealed package. These data clearly showed the necessary but not always sufficient condition that moisture plays in corrosion failures. Moisture measurements indicate that the first order failure threshold for corrosion failures occurs when liquid water forms on the surface of the device, or the interconnection pattern in the case of hybrids. For packages at 1 atm the lowest (liquid) dew point occurs at 0°C. Under these conditions, the moisture content is 6025 parts per million by volume (ppm_v). If a package contains at least this amount of moisture or more, liquid water will be present within the cavity at some temperature above freezing. If the pressure is less than 1 atm, then greater amounts of moisture in ppm_v are required to produce condensation at any given temperature. Since many CERDIPs have 0.5 atm, there is a safety factor built into any 1 atm moisture specification.

To visually illustrate the relationship between moisture content, dew point, relative humidity, failure threshold, specification limit, and measurement capability, a moisture spectrum has been assembled in figure 1. Several precautions are in order before using the chart. It has been assumed that the moisture is present in a gas at 1 atm. To convert to relative humidity, one must assume a fixed temperature, in this case 20°C. A hermetic package containing 23 039 ppm_v at 1 atm and at 20°C would be completely saturated and therefore have a relative humidity of 100 percent. It would also have a dew point of 20°C. A package containing 9883 ppm_v at 1 atm pressure would have a relative humidity of 43 percent at 20°C and would reach a dew point if cooled to 7°C.

It is obvious that the inside of the package reaches a dew point if it contains more than 6025 ppm_v and a frost point if less than 6025 ppm_v. MOS instability has been observed for devices containing 2000 ppm_v moisture, corrosion may occur if the moisture content is greater than 6025 ppm_v and gold dendrites have been shown to form quickly if the moisture content is greater than 15 000 ppm_v. For less than 500 ppm_v moisture in a small IC package, the absolute quantity of moisture is less 5 ng, and it can be argued that the quantity of moisture is insufficient to complete a corrosion process even if located at a single point on the IC surface. The surface ionic mobility at less than 500 ppm_v is also likely to be too small to induce oxide threshold instability. Measurement apparatus does exist to make moisture measurements of small IC volumes. The aluminum oxide sensor has a useful range from 100 $\mathrm{ppm_v}$ to slightly less than saturation. The mass spectrometer is capable of detecting 10 $\mathrm{ppm_v}$ moisture in 0.01 $\mathrm{cm^3}$ of nitrogen; however, it is currently considered accurate only over a range of 500 to 21 000 $\mathrm{ppm_v}$.

Part of our inhouse research program has been a program to evaluate on a random basis JAN parts procured for use in military systems. As part of this program, each of the ten devices purchased per slash sheet were gas analyzed. The results are shown in figures 2 and 3. Before discussing the data, it should be made very clear that the majority of these parts would have operated reliably in military equipment. Moisture alone, as mentioned previously, is not a sufficient condition for failure in most cases. However, the presence of moisture in most of these parts indicates that for high-reliability military parts, some control must be exercised over the moisture sealed within the part if the lowest possible failure rate is to be attained. It should be noted that all of the devices are CERDIPs and that, at the time of manufacture, most manufacturers' packages contained enough moisture to saturate the package at some temperature above 0°C. The monitoring program will continue with the hope that, with improved processing and assembly techniques, the moisture content in all military IC packages can be reduced to less than 500 $\mathrm{ppm_v}$.

Turning now to the problem of writing a specification, let me begin by noting that the test method was written with the objective of determining whether the package passes or fails the specified moisture limit of 5000 $\mathrm{ppm_v}$ for IC, 6000 $\mathrm{ppm_v}$ for hybrids, and 1000 $\mathrm{ppm_v}$ for desiccated parts. The initial draft was prepared jointly with Mr. C. Zierdt of Bell Telephone Laboratories. The intent was to make the options all-inclusive so that all techniques which were available at that time could be used to ascertain the moisture content of the package. Comments were solicited at the time the test method was coordinated and are welcome at any time. It is hoped that with full cooperation from government and industry, it will evolve into a clear, concise, and accurate test method for measuring moisture content within microelectronic packages. With that goal in mind, I would like to discuss some proposed changes which have been incorporated into the draft of a revised Method 1018, dated March 20, 1978. Since this summary is being written after the NBS meeting, I have taken the liberty to include other changes suggested by attendees during and since the meeting on March 20. A summary and rationale for the major changes follows by paragraph:

Para 2.1.a - This paragraph was extensively changed to improve the calibration procedures for small volume packages. The sensitivity factor is defined under flow conditions. The transfer function is more specifically defined for three volume ranges and three moisture limits.

Para 2.1.b - The temperature of the passage is specified as 125°C to insure uniformity from lab to lab. Higher temperatures can cause secondary reactions which would make the transfer function highly variable. Likewise, lower temperature would result in unpredictable losses of moisture during analysis. A statement is also added to ensure that the entire package reaches 100°C and is maintained at that temperature for 10 min, sufficient time for the physically adsorbed moisture on the package walls to enter the vapor state. Longer bake-out times will add a small amount of more deeply absorbed moisture; however, for eutectically mounted ICs, this effect is minimal. The increased vapor pressure of moisture in the cavity brings the net desorption rate to zero in a short period of time. Studies were reported by Mr. Moore earlier at this conference indicating that, even for packages containing epoxy die attach material, the equilibrium time is very short.

Para 2.2.a - The sensitivity factor definition based on 0.01 cm^3 package volume was too restrictive for this procedure which is partially intended for larger packages. The definition is replaced by an absolute definition involving micrograms of water and the weight of the gas in the package.

Para 2.3.a - The minimum moisture sensor sensitivity was raised to 300 ppm to allow the use of conductivity type sensors in addition to the aluminum oxide sensors.

Para 3.1 - A requirement was added that the devices be verified as hermetic prior to testing per Method 1018. This will prevent samples from being sent to the analytical labs which cannot be analyzed. The cleaning requirement was clarified to indicate that only those substances such as varnish epoxies or silicones need be removed from the external surface and only if they interfere with the water vapor measurement. The maximum bake-out temperature is limited to 125°C. This reduces the reaction of moisture metal surfaces and gives a more accurate determination of moisture sealed into the cavity.

Para 3.1.a - A more precise statement of abnormal pressure rise replaces an ambiguous guideline.

Para 3.1.b - A statement concerning the desirability of reporting other gases contained in the cavity has been added. While reporting is not mandatory, the presence and relative amount of helium, argon, oxygen, and fluorocarbons may indicate the source of moisture or other seal problems.

Para 3.1.c - A statement was added defining data reduction procedures in the case of desiccants or organics in order to limit the inclusion of moisture desorbing from the material after the package has been opened. This would unduly penalize manufacturers using these materials and would not simulate a use condition.

Para 3.2 - A modification was made to allow for a newly developed technique utilizing a holding chamber, a moisture sensor, and a pressure sensor.

Para 3.3 - A change was made in measurement temperature which would permit the use of a conductivity type sensor, in this procedure, and a requirement for mass spectrometer correlation was added.

This concludes the changes proposed for Method 1018. An addition to Method 5008 for hybrids will correct an omission from the published version which will allow the 100-percent use of moisture sensors in lieu of fine and gross leak testing. As the sensors become more widespread in usage and the price comes down, the potential for true moisture leak measurements by a more cost-effective technique will be realized.

A point should be made about calibration accuracy for any of the analytical techniques described previously. The accuracy of the instrument does not appear in the test method. Instead, it will appear in a checklist being made up by RADC and used by DESC in surveying laboratories wishing to become certified. It is proposed that, before complete certification is granted, the laboratory must be able to measure standards to within ±20 percent of the absolute value.

Revisions as outlined in the March 20 draft will be sent out for coordination before the June JC-13 meeting. A notice to MIL-STD-883B should follow within 60 days.

Future limits for moisture in microelectronic packages will await the results of current government- and industry-sponsored research to determine moisture failure thresholds for a number of devices and technologies and the development of more sensitive measurement techniques.

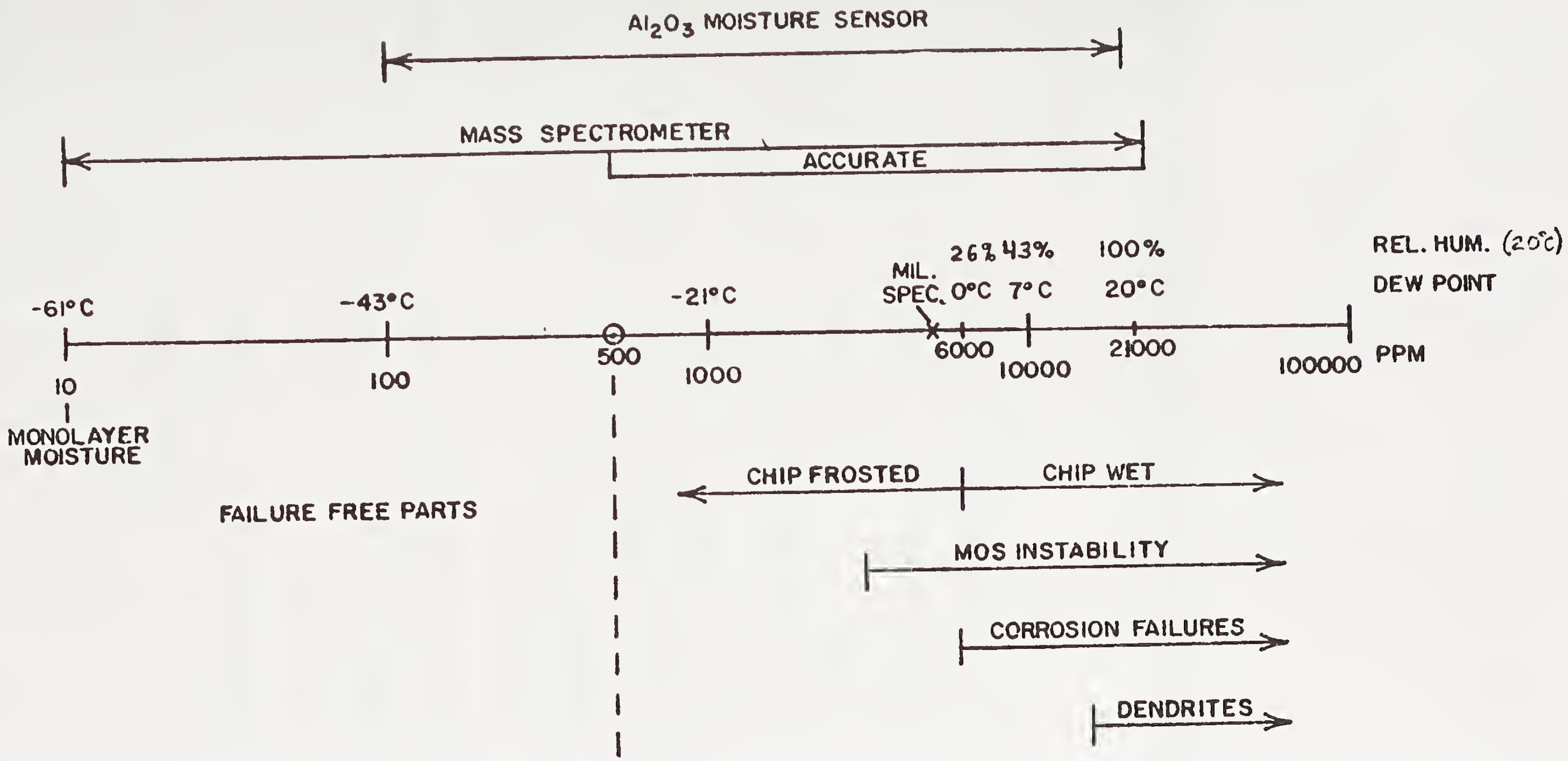

Figure 1. Relationship between moisture content, dew point, relative humidity, failure threshold, specification limit, and measurement capability.

PE#	MANUFACTURER	DEVICE #	DATE CODE	H_2O CONTENT (0.01%=100PPM, 0.1%=1000PPM)		
				AVG	LOW	HIGH
21	SIGNETICS	JM38510/00101BCB	7415 & 7340	1.75	0	3.4
22	SIGNETICS	JM38510/02304BCB	7346	1.8	0.6	2.9
23	TEXAS INST	38510B	T7537	0.70	0.3	1.4
34	MOTOROLA	JM38510/00202BAC	7516	0.5		
35	SIGNETICS	JM38510/00202BDB	7549 & 7543	0.2		
36	SIGNETICS	JM38510/02304BDB	7542	0.7		
50	MOTOROLA	JM38510/00303BCB	7541	2.4	1.5	3.8
51	I.T.T.	JM38510/00502BAB	7540	0.1	0.02	0.2
52	SIGNETICS	JM38510/00502BCB	7437	0.4	0.04	0.7
53	NATIONAL	JM38510/00502BDB	7543	1.2	0.7	2.0
54	FAIRCHILD	JM38510/101BCB	7540	1.9	0.7	4.0
55	FAIRCHILD	JM38510/10101BGB	7640	0.2	0.1	0.3

Figure 2. Moisture content of some sampled JAN parts. Ten specimens per slash sheet.

6. Group Encounters

Speaker Pairs:

1. Robert W. Thomas, Aaron Der Marderosian
2. Johnny L. Hartley, Steven R. Loucks
3. David A. Stevenson, William M. Hickam

Moderators:

Saburo Hasegawa (NBS)
Ernest E. Hughes (NBS)
Stanley Ruthberg (NBS)

SUMMARY

Moisture Sensors - What are the practical considerations for calibration, when should calibration be done, and how can one take into account the shifts in calibration that occur? What is the uniformity in response for sensors obtained from the same wafer? When are they stable and uniform enough for use?

An example of sensor precision and stability under benign conditions is found in the analyzer of Loucks and Burnham (Session III). Calibration is obtained by direct injection of water into the courier gas stream by microsyringe transfer with 10-μg to 10-mg samples. Calibration is performed on a day-to-day basis, and a precision of ∿10 percent at 50 μg can be obtained daily, though sensitivity shifts from day to day. The smallest package volume that can be accommodated is ∿0.5 cm^3. The minimum detectable quantity of water is ∿3 μg in a 6-cm^3 package. Package bakeout to 100°C can be made. They have not had to replace the original sensor.

Evidence appears to indicate that sensor response will not change drastically even under thermal treatment to 150°C, provided thermal shock is absent. Alternatively, thermal shocks such as obtained with gold-tin solder or eutectic die attach will cause a large calibration shift. Calibration should then be obtained following such a process; however, successive thermal shock will again alter the calibration characteristic. Uniformity of sensors from the same wafer appears to be fair.

Mass Spectrometers - What is the relative merit of one type of instrument over another; why is the limit of 500 parts per million by volume (ppm_v) of water being established; are organics allowed within the package; what is the potential of the acetylene conversion scheme; are any people having difficulty in analyzing oxygen; what about ion abundances of M/e 17 and 18?

With appropriate procedure, there should not be any difference in measurements made with one type of instrument *versus* another, say the quadrupole *versus* the sector. The 500 ppm_v as stated in MIL-STD-1018 is, for the present, a condition on the minimum detectable capability of the analyzers rather than as a limit on packages. However, corrosion effects do occur at concentrations of ∿4000 ppm_v and oxide effects are seen to occur at ∿1000 ppm_v. We need more definitive studies on these thresholds, which studies should better be implemented as the measurements precision increases. As instrument capability increases, we will expect the lower limit to be applied. Organics within the package will not prohibit measurements, though the kinetics of moisture emission may be affected. Organics on the outside of the package are not desirable. The acetylene conversion method (Hickam and Morgan, Session III) has a potential conversion efficiency of 90 percent. The smallest volume analyzed to date is 0.02 cm^3, and analysis usually takes 25 min. There should not be any difficulty in analyzing oxygen, but the abundance of M/e 17 *versus* 18 shall remain a question.

Failure Mechanisms - There is a need for more fundamental studies on thresholds for corrosion, dendritic growth, oxide effects, etc. The consensus is that packages should be as dry as possible; water is a catalyst; removing contaminants *per se* is not a complete answer.

Other Procedures - These would be acceptable if qualified against the mass spectrometer, at least for the present, e.g., acetylene conversion and surface conductivity cells.

The 125°C limit set forth in MIL-STD-1018 is really for the mass spectrometer and is a compromise value on getting enough ambient water into the machine *versus* chemical reactions which could occur at greater temperatures. The 100°C to 125°C is to protect the devices from the operating environment.

Vacuum-sealed devices can be analyzed, but the moisture content would have to be given in appropriate units, i.e., not ppm_v.

Many lengthy discussions were directed to moisture generators, materials, arrangements, valves, capability, needs. The cost of a generator such as described by Hartley (Session III) would be from $2,000 to $3,000, but could be reduced. Sampling times are important to avoid desorption. Both two-temperature and two-pressure systems were considered.

6.1 List of Attendees

MOISTURE MEASUREMENT WORKSHOP March 22-23, 1978, NBS, Gaithersburg, MD

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11\. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

The workshop, fifth in a series concerned with measurement problems in integrated circuit processing and assembly, served as a forum to examine present problems with the measurement of moisture in hermetic semiconductor devices. While moisture-induced failure modes and mechanisms had received considerable attention in the published literature and in meetings, the accurate and reliable measurement of the moisture content had not; yet, this lack of measurement of moisture reliability is a major obstacle to meaningful efforts to limit and control this pervasize contaminant. Manuscripts and summaries are provided of 19 talks, panel meetings, and group encounters on three major topics: mass spectrometer measurements of internal package moisture, moisture sensors, and package analysis and quality assurance.

12\. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

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