

C76-597/201

DEVELOPMENT OF LOW COST,
HIGH RELIABILITY SEALING TECHNIQUES
FOR HYBRID MICROCIRCUIT PACKAGES

PHASE II
FINAL REPORT

August 1977

Contract NAS8-31992

(NASA-CR-150420) DEVELOPMENT OF LOW COST,
HIGH RELIABILITY SEALING TECHNIQUES FOR
HYBRID MICROCIRCUIT PACKAGES, PHASE 2 Final
Report (Rockwell International Corp.,
Anaheim, Calif.) 108 p HC A06/MF A01

N77-33349

Unclass

G3/27 50763



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1.0 INTRODUCTION

1.1 STUDY BACKGROUND

An investigation to determine the suitability of using adhesive package sealing as an alternate to metallurgical package sealing for hybrid microcircuits for NASA/MSFC applications was started in June 1975. The initial effort directed to a preliminary evaluation of selected adhesives to assess their feasibility for this application was performed during the period from June 1975 to April 1976 under Contract NAS8-31517. The major effort performed under that contract consisted of (1) surveying representative hybrid manufacturers to assess the current use of adhesives for package sealing, (2) making a cost comparison of metallurgical versus adhesive package sealing, and (3) determining the seal integrity of gold-plated Kovar butterfly-type packages sealed with selected adhesives after they had been subjected to MIL-STD-883A, Class A, test environments.

This study is documented in detail in a NASA Contractor Report, NASA CR 144339 issued in April 1976. Results showed that adhesive-sealed packages retained their seal integrity (as determined by the seal test specified in MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁) after they had been sequentially subjected to MIL-STD-883A, Class A Thermal Shock, Temperature Cycling, Mechanical Shock, and Constant Acceleration test environments. Specifically, 1.27 cm (1/2 inch) square gold-plated Kovar butterfly-type packages sealed with the film adhesives Ablefilm 507, 529, and 550 and the paste adhesive Epo-Tek H77 retained their seal integrity after all tests, and similar 2.54 cm (1 inch) square packages retained their seal integrity after all tests except the 10,000g's constant acceleration test. While these results were by no means considered to be sufficient to establish adhesives as suitable for sealing high reliability hybrid microcircuit packages, they were considered encouraging and indicative that adhesive package sealing warranted further evaluation. As a result, a follow-on study was authorized.

1.2 SCOPE OF THE PRESENT STUDY

The general objective of the overall study is to investigate low cost, high reliability sealing techniques for hybrid microcircuit packages. The

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specific objective of this portion of the study (Phase II) was to continue the evaluation of adhesives (begun in Phase I) to determine whether or not they qualify for this application. This effort consisted of the following three tasks:

Task 1: Seal gold-plated Kovar packages with selected adhesives and determine their seal integrity after they have been exposed to temperature-humidity environments.

Task 2: Seal both gold-plated Kovar and ceramic packages with the four best adhesives identified in Task 1 and determine their seal integrity after they have been subjected to MIL-STD-883A test environments.

Task 3: Subject the best adhesive-package combination identified in Task 2 to a 60°C/98% RH environment and determine its susceptibility to moisture permeation.

2.0 TECHNICAL DISCUSSION

2.1 EFFECT OF TEMPERATURE-HUMIDITY EXPOSURES ON SEAL INTEGRITY

The objective of this effort was to determine if adhesive-sealed gold-plated Kovar packages can pass the seal test requirements of MIL-STD-883A, Method 1014.1 after they have been exposed to selected temperature-humidity environments. Ten adhesives, as listed in Table 1, and the following four temperature-humidity environments were selected for this evaluation.

Level 1 - 50°C/60% RH for ten days

Level 2 - 60°C/98% RH for ten days

Level 3 - Moisture resistance test environment per Method 1004.1 of MIL-STD-883A

Level 4 - 85°C/85% RH for ten days

Seal integrity was determined before and after exposing the packages to these environments by performing fine and gross-leak tests in accordance with MIL-STD-883A, Method 1014.1, Test Conditions A₂ and C₁ and C₂, respectively. For the fine-leak test, the packages were bombed at 30 psig helium for three hours. For the C₂ gross-leak test, the packages were pressurized at 30 psig for two hours. Packages sealed with all ten adhesives were exposed to the Level 1 and Level 2 environments. Level 3 and Level 4 testing was conducted only on packages sealed with the adhesives which passed exposure to the Levels 1 and 2 environments. In all cases, three packages sealed with each of the adhesives and three seam-sealed packages (to serve as controls) were tested. A different set of packages was exposed to each environment.

2.1.1 Package Assembly Method

Since a large number of packages had to be assembled, a special fixture was designed and fabricated for this purpose. A photograph of the fixture containing an assembled package is shown in Figure 1, and a close-up showing the package assembly region and assembled package in greater detail is given in Figure 2. The fixture has eight precisely positioned pins to ensure proper alignment. Teflon-coated, stainless-steel plates, 0.152 cm (60 mils) thick, are placed on each side of the package to ensure that it is not distorted when the clamping pressure required during cure is applied.

Table 1. Adhesives Selected for Evaluation

Adhesive	Type	Manufacturer
Ablefilm 507	Nonconductive Film	Ablestik Laboratories
Ablefilm 550	Nonconductive Film	Ablestik Laboratories
Alebond 36-2	One Component Silver-filled Paste	Ablestik Laboratories
Alebond 58-1	One Component Gold-filled Paste	Ablestik Laboratories
Epo-Tek H20E	Two Component Silver-filled Paste	Epoxy Technology, Inc.
Epo-Tek H81	Two Component Gold-filled Paste	Epoxy Technology, Inc.
Epo-Tek H77	Two Component Non-conductive Paste	Epoxy Technology, Inc.
Alebond 789-1	One Component Non-conductive Paste	Ablestik Laboratories
Alebond 873-1	Epoxy Novolak Paste	Ablestik Laboratories
AF-30	Nitrile Phenolic Film	3M Company

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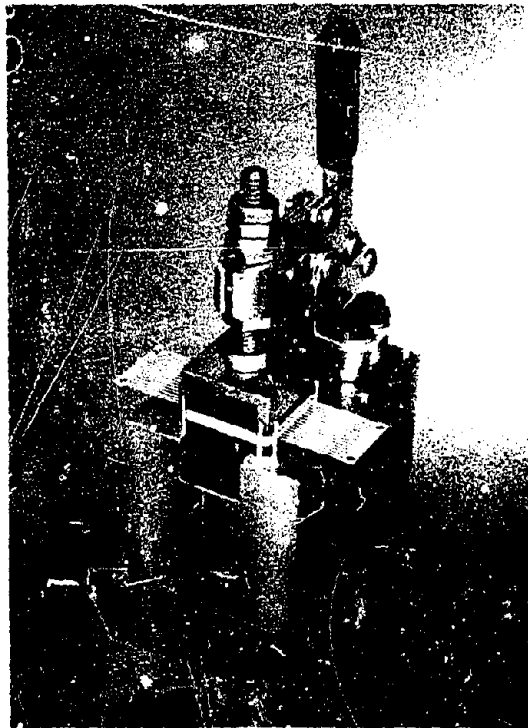


Figure 1. Package Assembly Fixture

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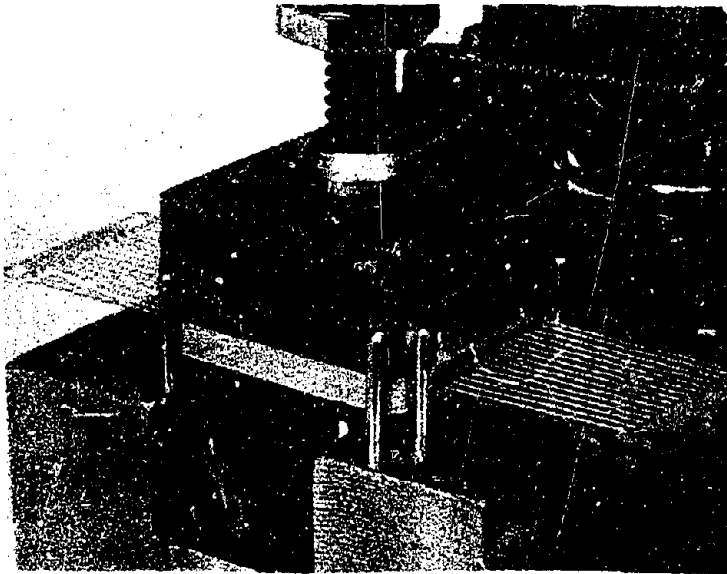


Figure 2. Close-up View of Package Assembly Region and Assembled Package

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The procedure for preparing a package was as follows: The package (package case, adhesive, package lid) was assembled by placing the parts between the alignment pins with a teflon-coated plate on each side (bottom and top). A metal yoke was then placed on top of this assembly and the toggle clamp closed, forcing the attached bolt down on the metal yoke and the metal yoke down on the package. This is shown in Figures 1 and 2. The force exerted on the package was controlled by a spring on the bolt and was just sufficient to keep the package parts from shifting with respect to each other while a spring clamp was applied to the package assembly (package and teflon-coated plates). Also, the end of the bolt which contacts the metal yoke was free to swivel to ensure that the force exerted on the metal yoke was perpendicular to its top surface and could not cause any misalignment of the parts of the package assembly. A spring clamp of proper force was then placed on the package assembly, the toggle clamp was opened, and the clamped package assembly was removed from the fixture. A photograph of a package assembly ready for cure is shown in Figure 3. The package was leveled by adjusting the bolt on the opposite end of the clamp.

2.1.2 Packages and Package Processing

The packages selected for testing were 2.54 cm (1 inch) square, gold-plated Kovar, butterfly-type packages of solid ring frame construction with gold-plated Kovar lids. Their rim width was 0.102 cm (40 mils) and their sealing area 1.03 cm² (0.16 inch²). These packages were selected because of their wide use in NASA/MSFC equipment. Package blanks (or sealing boxes) were used rather than completed packages. These were identical to the completed packages except that the holes for the feedthroughs had not been drilled and the feedthroughs installed. Package blanks are less expensive than completed packages and their use for the evaluation of sealing is preferable. Since there were no feedthroughs as possible sources of leaks, the leak rates measured were entirely due to the package seals.

The packages (and lids) were cleaned by brushing them successively in deionized water, acetone, and isopropyl alcohol and then spray rinsing with Freon TF. The cleaned packages were stored in a chamber containing a nitrogen ambient and used within a few hours after they were cleaned. The adhesive preforms were removed from the freezer, placed in the nitrogen chamber with the packages, and allowed to stand at room temperature for approximately

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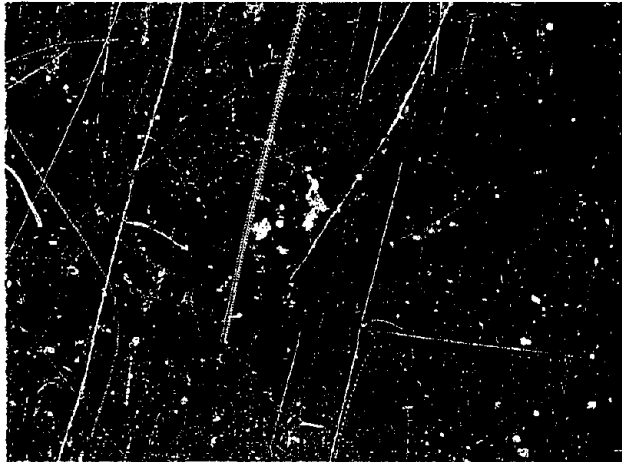


Figure 3. Package Assembly Ready for Cure

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one hour before they were used. The packages were assembled in room ambient using the assembly fixture and method described in the previous section (2.1.1). The film adhesives were placed on the package case in the fixture. The paste adhesives were manually applied to the rim of the package case before the package case was placed in the fixture. The adhesive was applied using a sharpened Q-tip stick while the procedure was monitored under a microscope to assure complete coverage and the absence of bubbles.

The packages had a small hole 0.033 cm (13 mils) in diameter in their lids which aligned with a 0.318 cm (1/8 inch) diameter hole in the top teflon-coated plate to allow the packages to vent during cure. The packages were cured in a nitrogen environment and the holes were subsequently solder sealed in room environment. Clamping pressures applied during cure were $7.0 \times 10^4 \text{ N/m}^2$ (10.1 psi) for the paste adhesives, $1.1 \times 10^5 \text{ N/m}^2$ (16.3 psi) for the film adhesives Ablefilm 507 and 550, and $2.0 \times 10^5 \text{ N/m}^2$ (29.0 psi) for the film adhesive AF-30. In the case of the paste adhesives, since their viscosities are initially very low, only sufficient pressure was applied to assure that the parts remained in alignment during cure. The cure schedules used for the adhesives are given in Table 2. These cure schedules meet or exceed those recommended by the manufacturers.

2.1.3 Results for Level 1 Environment Exposure

Results obtained for packages subjected to the Level 1 environment (50°C/60% RH for ten days) are given in Table 3. The nitrile phenolic film adhesive, AF-30, was eliminated from consideration because 11 of 12 packages sealed with it failed the initial C_2 gross leak test. Since this problem was not encountered with any of the other adhesives, it was felt that this adhesive was not worth further effort. The only adhesive affected by exposure to this environment was Epo-Tek H20E. All three packages sealed with this adhesive were found to be gross leakers when tested according to Test Condition C_2 . However, it is interesting to note that these packages were not found to be gross leakers when previously tested according to Test Condition C_1 , and their fine-leak rates measured prior to C_1 and C_2 gross-leak testing were as follows:

- Package 1: 3.6×10^{-7} atm cc/sec (air equivalent)
- Package 2: 4.2×10^{-7} atm cc/sec (air equivalent)
- Package 3: 4.0×10^{-7} atm cc/sec (air equivalent)

Table 2. Adhesive Cure Schedules

Adhesive	Cure Schedule
Ablefilm 507	70 minutes at 175°C
Ablefilm 550	2-1/2 hours at 150°C
Ablebond 36-2	40 minutes at 150°C
Ablebond 58-1	40 minutes at 150°C
Epo-Tek H20E	15 minutes at 150°C
Epo-Tek H81	15 minutes at 150°C
Epo-Tek H77	30 minutes at 150°C
Ablebond 789-1	2 hours at 170°C
Ablebond 873-1	2 hours at 170°C
AF-30	70 minutes at 175°C

Note: These cure schedules meet or exceed those recommended by the adhesive manufacturers.

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Table 3. Effect of Level 1 Environment Exposure
(Ten Days at 50°C/60% RH)

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507 1 2 3	8.0×10^{-8} 6.2×10^{-8} 8.0×10^{-8}	9.0×10^{-8} 8.8×10^{-8} 8.8×10^{-8}
Ablefilm 550 1 2 3	3.6×10^{-8} 1.5×10^{-8} 1.4×10^{-8}	4.0×10^{-8} 1.4×10^{-8} 3.0×10^{-8}
Ablebond 36-2 1 2 3	1.2×10^{-7} 1.6×10^{-7} 1.0×10^{-7}	1.7×10^{-7} 1.2×10^{-7} 2.0×10^{-7}
Ablebond 58-1 1 2 3	1.0×10^{-8} 9.5×10^{-9} 4.2×10^{-8}	2.8×10^{-3} 7.0×10^{-8} 5.2×10^{-8}
Epo-Tek H20E 1 2 3	1.6×10^{-7} 1.4×10^{-7} 1.4×10^{-7}	Gross (C ₂) Gross (C ₂) Gross (C ₂)
Epo-Tek H81 1 2 3	2.4×10^{-8} 2.1×10^{-8} 4.6×10^{-8}	1.4×10^{-8} 1.8×10^{-8} 3.8×10^{-8}
Epo-Tek H77 1 2 3	1.1×10^{-8} 1.8×10^{-8} 8.4×10^{-9}	1.2×10^{-8} 2.0×10^{-8} 8.7×10^{-9}
Ablebond 789-1 1 2 3	4.4×10^{-8} 4.4×10^{-8} 6.3×10^{-8}	4.8×10^{-9} 5.0×10^{-9} 1.6×10^{-8}
Ablebond 873-1 1 2 3	5.4×10^{-8} 3.0×10^{-8} 1.3×10^{-8}	2.1×10^{-8} 1.6×10^{-8} 1.3×10^{-8}
Seam Sealed 1 2 3	1.2×10^{-9} 4.4×10^{-9} 0.9×10^{-9}	1.2×10^{-9} 2.0×10^{-9} 1.8×10^{-9}

2.1.4 Results for Level 2 Environment Exposure

Results for packages subjected to the Level 2 environment (60°C/98% RH for ten days) are given in Table 4. Packages were first tested for fine leaks, then gross leaks per Test Condition C₁, and finally gross leaks per Test Condition C₂. Packages found to be gross leakers when tested per Test Condition C₁ were not tested per Test Condition C₂. All packages for which fine leaks could not be measured were found to be gross leakers when tested per Test Condition C₁ and vice versa. Seven additional packages (three sealed with Ablefilm 550, three with Ablebond 58-1 and one with Ablebond 789-1) were found to be gross leakers when tested per Test Condition C₂. The fine-leak rates measured for these packages prior to C₁ and C₂ gross-leak testing were as follows:

<u>Adhesive & Package No.</u>	<u>Leak Rate atm cc/sec (air equivalent)</u>
<u>Ablefilm 550</u>	
Package 4	6.8×10^{-9}
Package 5	1.0×10^{-8}
Package 6	3.8×10^{-9}
<u>Ablefilm 58-1</u>	
Package 4	2.2×10^{-6}
Package 5	$>3.0 \times 10^{-6}$
Package 6	5.2×10^{-8}
<u>Ablebond 789-1</u>	
Package 4	3.0×10^{-9}

The gross leak in the package sealed with Ablebond 789-1 (Package 4) was not at the adhesive seal but was due to an imperfection in the factory weld (bottom to rim). For all of the other leaky packages, failures occurred due to separation of the adhesive from the gold metal, indicating that the failure mode was adhesive rather than cohesive. Since gold is a difficult metal to bond to, this is the expected failure mode. Also, failure of the adhesives in this case does not necessarily mean that they would not be adequate for other applications such as sealing ceramic to ceramic.

Review of the results given in Table 4 shows that several adhesives were severely degraded by exposure to this environment and should be eliminated from further consideration. These were Ablebond 58-1, Epo-Tek H20E, Epo-Tek H81,

Table 4. Effect of Level 2 Environment Exposure
(Ten Days at 60°C/98% RH)

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507 4 5 6	1.0×10^{-7} 6.4×10^{-8} 8.6×10^{-8}	3.2×10^{-7} 4.6×10^{-8} 5.4×10^{-8}
Ablefilm 550 4 5 6	1.4×10^{-8} 4.6×10^{-8} 5.3×10^{-8}	Gross (C ₂) Gross (C ₂) Gross (C ₂)
Ablebond 36-2 4 5 6	2.4×10^{-7} 2.7×10^{-7} 1.8×10^{-7}	2.7×10^{-8} 9.6×10^{-8} Gross (C ₁)
Ablebond 58-1 4 5 6	1.8×10^{-8} 1.4×10^{-8} 3.0×10^{-8}	Gross (C ₂) Gross (C ₂) Gross (C ₂)
Epo-Tek H20E 4 5 6	2.1×10^{-7} 2.4×10^{-7} 1.6×10^{-7}	Gross (C ₁) Gross (C ₁) Gross (C ₁)
Epo-Tek H81 4 5 6	8.4×10^{-8} 2.5×10^{-8} 4.0×10^{-8}	Gross (C ₁) Gross (C ₁)* 1.8×10^{-8}
Epo-Tek H77 4 5 6	2.0×10^{-8} 1.6×10^{-8} 1.0×10^{-8}	6.0×10^{-9} 1.5×10^{-8} Gross (C ₁)
Ablebond 789-1 4 5 6	5.8×10^{-8} 1.5×10^{-8} 1.8×10^{-8}	Gross (C ₂ at factory weld 1.2×10^{-8} 1.9×10^{-8}
Ablebond 873-1 4 5 6	5.9×10^{-8} 2.0×10^{-8} 2.1×10^{-8}	Gross (C ₁) 4.4×10^{-9} Gross (C ₁)*
Seam Sealed 4 5 6	0.8×10^{-9} 0.7×10^{-9} 1.6×10^{-9}	1.2×10^{-9} 1.0×10^{-9} 1.4×10^{-9}

*Lid came off during fine-leak testing

and Ablebond 873-1. Even though all three packages sealed with Ablefilm 550 were found to be C_2 gross leakers, it was felt that this adhesive should be evaluated further because low fine-leak rates were measured for all three packages sealed with it, and all of these packages passed the C_1 gross-leak test. Review of the results given in Table 4 also indicates that two of the adhesives, Ablefilm 507 and Ablebond 789-1, were unaffected by exposure to this environment (all packages retained their seal integrity). Also, for two other adhesives, Ablebond 36-2 and Epo-Tek H77, only one package lost seal integrity. These adhesives are worthy of further testing at the more severe temperature-humidity conditions.

In summary, as a result of exposure to the Level 2 temperature-humidity environment (60°C/98% RH for ten days), four adhesives (Ablebond 58-1, Epo-Tek H20E, Epo-Tek H81, and Ablebond 873-1) were eliminated from further consideration for package sealing; and five adhesives (Ablefilm 507, Ablebond 789-1, Ablebond 36-2, Epo-Tek H77 and Ablefilm 550) were selected for further testing.

2.1.5 Results for Level 3 Environment Exposure

Results for packages exposed to the Level 3 environment (moisture resistance test environment per Method 1004.1 of MIL-STD-883A) are given in Table 5. A comparison of these results with those given in Table 4 for similar packages exposed to the Level 2 environment (60°C/98% RH for ten days) indicates that the Level 2 environment is possibly more severe than the Level 3 environment. This conclusion suggests that continuous exposure at 50°C for ten days is more degrading than the effects due to cycling the temperature between 25°C and 65°C at six-hour intervals for ten days.

2.1.6 Results for Level 4 Environment Exposure

Results for packages exposed to the Level 4 environment (85°C/85% RH for ten days) are given in Table 6. The only packages that retained their seal integrity after exposure to this environment were the three that were seam sealed, two of the three that were sealed with Ablefilm 507, and the three that were sealed with Ablebond 789-1. All others (one of the three sealed with Ablefilm 507, and all three sealed with Ablefilm 550, Ablebond 36-2 and Epo-Tek H77)

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Table 5. Effect of Level 3 Environment Exposure
(Moisture Resistance Test Environment
per Method 1004.1 of MIL-STD-883A)

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507		
7	1.1×10^{-7}	5.4×10^{-8}
8	1.4×10^{-7}	5.8×10^{-8}
9	1.1×10^{-7}	4.0×10^{-8}
Ablefilm 550		
7	7.3×10^{-8}	5.0×10^{-8}
8	9.4×10^{-8}	Gross (C ₂)
9	3.3×10^{-8}	2.6×10^{-8}
Ablebond 36-2		
7	1.3×10^{-7}	9.4×10^{-8}
8	1.3×10^{-7}	1.0×10^{-7}
9	3.2×10^{-7}	1.1×10^{-7}
Epo-Tek H77		
7	1.1×10^{-8}	1.2×10^{-8}
8	7.8×10^{-9}	8.2×10^{-9}
9	1.6×10^{-8}	Gross (C ₁)
Ablebond 789-1		
7	1.8×10^{-8}	7.0×10^{-9}
8	1.0×10^{-8}	1.1×10^{-8}
9	1.9×10^{-8}	2.0×10^{-8}
Seam Sealed		
7	$<1.0 \times 10^{-9}$	2.0×10^{-9}
8	2.4×10^{-9}	2.0×10^{-9}
9	$<1.0 \times 10^{-9}$	1.4×10^{-9}

Table 6. Effect of Level 4 Environment Exposure
(Ten Days at 85°C/85% RH)

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507 10 11 12	1.1×10^{-7} 1.1×10^{-7} 1.3×10^{-7}	6.4×10^{-8} 6.3×10^{-8} Gross (C_1)
Ablefilm 550 10 11 12	2.9×10^{-7} 3.3×10^{-7} 6.5×10^{-8}	Gross (C_1) Gross (C_1) Gross (C_1)
Ablebond 36-2 10 11 12	1.7×10^{-7} 1.4×10^{-7} 1.6×10^{-7}	Gross (C_1) Gross (C_1) Gross (C_1)
Epo-Tek H77 10 11 12	1.3×10^{-8} 1.1×10^{-8} 6.4×10^{-8}	Gross (C_1) Gross (C_1) Gross (C_1)
Ablebond 789-1 10 11 12	4.0×10^{-8} 2.1×10^{-7} 6.4×10^{-8}	1.3×10^{-8} 9.6×10^{-9} 1.5×10^{-8}
Seam Sealed 10 11 12	$<1.0 \times 10^{-9}$ $<1.0 \times 10^{-9}$ $<1.0 \times 10^{-9}$	1.3×10^{-9} 1.0×10^{-9} 1.4×10^{-9}

were found to be C_1 gross leakers. A comparison of these results with those given in Tables 4 and 5 for similarly sealed packages exposed to the Level 2 and Level 3 environments (60°C/98% RH for ten days and the moisture resistance test environment specified in Method 1004.1 of MIL-STD-883A) substantiates that this environment (85°C/85% RH for ten days) is without doubt the most severe.

2.1.7 Results for Extended Level 1 Environment Exposure

The packages which previously were exposed to the Level 1 environment (50°C/60% RH for ten days), and retained their seal integrity, were exposed to this same environment an additional three times. These packages therefore were exposed to 50°C/60% RH for a total of 40 days or approximately 1000 hours. Seal test results after each ten day exposure are given in Table 7. As can be seen from a comparison of the last two columns of this table, only one additional package failed (the only remaining package sealed with Epo-Tek H77) as a result of the fourth ten-day exposure.

The important result is that at least three and perhaps five of the nine adhesives tested provide package seals that retain their integrity (i.e., pass the MIL-STD-883A seal test) after long-term exposure (approximately 1000 hours) to a temperature-humidity environment of 50°C/60% RH. Since 50°C/60% RH is a relatively mild temperature-humidity environment, this result alone is not particularly significant. However, it complements the results obtained for the adhesives tested for a shorter time under more severe conditions.

2.1.8 Adhesives for Further Evaluation

A summary of the results obtained from this evaluation is given in Table 8. The data are simplified by using an "X" to indicate the packages that retained their seal integrity after exposure to each temperature-humidity environment, and a dash (-) to indicate those that did not. The asterisks (*) in the Ten-day 60°C/98% RH Column indicate that these adhesives were eliminated from further consideration because of their failure during exposure to this environment. On the basis of these data, the four best adhesives were Ablebond 789-1, Ablefilm 507, Ablebond 36-2 and Epo-Tek H77.

2.1.9 Results of Infrared Analysis of Selected Adhesives

Infrared spectrographic analyses were made for two adhesives (Ablefilm 507 and Ablebond 789-1) that passed exposure to all of the temperature-humidity

Table 7. Effect of Extended Exposure at 50°C/60% RH

Adhesive	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After First 10 Day Exposure Air Equivalent (atm cc/sec)	Leak Rate After Second 10 Day Exposure Air Equivalent (atm cc/sec)	Leak Rate After Third 10 Day Exposure Air Equivalent (atm cc/sec)	Leak Rate After Fourth 10 Day Exposure Air Equivalent (atm cc/sec)
Ablefilm 507	1	8.0 x 10 ⁻⁸	9.0 x 10 ⁻⁸	7.2 x 10 ⁻⁸	3.2 x 10 ⁻⁸
	2	6.2 x 10 ⁻⁸	8.8 x 10 ⁻⁸	6.0 x 10 ⁻⁸	3.1 x 10 ⁻⁸
	3	8.0 x 10 ⁻⁸	8.8 x 10 ⁻⁸	8.4 x 10 ⁻⁸	3.7 x 10 ⁻⁸
Ablefilm 550	1	3.6 x 10 ⁻⁸	4.0 x 10 ⁻⁸	Gross (C1) 2.6 x 10 ⁻⁸	---
	2	1.5 x 10 ⁻⁸	1.4 x 10 ⁻⁸	2.8 x 10 ⁻⁸	---
	3	1.4 x 10 ⁻⁸	3.0 x 10 ⁻⁸	2.8 x 10 ⁻⁸	Gross (C1) Gross (C2)
Ablebond 3F-2	1	1.2 x 10 ⁻⁷	1.7 x 10 ⁻⁷	3.1 x 10 ⁻⁷	---
	2	1.6 x 10 ⁻⁷	1.2 x 10 ⁻⁷	1.6 x 10 ⁻⁷	Gross (C1) 6.4 x 10 ⁻⁸
	3	1.0 x 10 ⁻⁷	2.0 x 10 ⁻⁷	1.6 x 10 ⁻⁷	6.3 x 10 ⁻⁸
Ablebond 5B-1	1	1.0 x 10 ⁻⁸	2.8 x 10 ⁻⁸	3.4 x 10 ⁻⁸	---
	2	9.5 x 10 ⁻⁹	7.0 x 10 ⁻⁸	3.6 x 10 ⁻⁸	Gross (C2) Gross (C2)
	3	4.2 x 10 ⁻⁸	5.2 x 10 ⁻⁸	6.5 x 10 ⁻⁸	Gross (C1) Gross (C1)
Epo-Tek H20E	1	1.6 x 10 ⁻⁷	Gross (C2)	---	---
	2	1.4 x 10 ⁻⁷	Gross (C2)	---	---
	3	1.4 x 10 ⁻⁷	Gross (C2)	---	---
Epo-Tek H81	1	2.4 x 10 ⁻⁸	1.4 x 10 ⁻⁸	Gross (C2) 2.6 x 10 ⁻⁸	---
	2	2.1 x 10 ⁻⁸	1.8 x 10 ⁻⁸	2.8 x 10 ⁻⁸	1.4 x 10 ⁻⁸ 1.9 x 10 ⁻⁸
	3	4.6 x 10 ⁻⁸	3.8 x 10 ⁻⁸	2.8 x 10 ⁻⁸	---
Epo-Tek H77	1	1.1 x 10 ⁻⁸	1.2 x 10 ⁻⁸	2.1 x 10 ⁻⁸	---
	2	1.8 x 10 ⁻⁹	2.0 x 10 ⁻⁸	3.0 x 10 ⁻⁸	Gross (C1) 1.2 x 10 ⁻⁸
	3	8.4 x 10 ⁻⁹	8.7 x 10 ⁻⁹	1.7 x 10 ⁻⁸	Gross (C1)
Ablebond 789-1	1	4.4 x 10 ⁻⁸	4.8 x 10 ⁻⁹	5.0 x 10 ⁻⁸	4.4 x 10 ⁻⁹
	2	4.4 x 10 ⁻⁸	5.0 x 10 ⁻⁹	8.8 x 10 ⁻⁸	3.9 x 10 ⁻⁹
	3	6.3 x 10 ⁻⁸	1.6 x 10 ⁻⁸	6.2 x 10 ⁻⁸	1.1 x 10 ⁻⁸
Ablebond 873-1	1	5.4 x 10 ⁻⁸	2.1 x 10 ⁻⁸	5.4 x 10 ⁻⁸	2.4 x 10 ⁻⁸
	2	3.0 x 10 ⁻⁸	1.6 x 10 ⁻⁸	4.3 x 10 ⁻⁸	1.9 x 10 ⁻⁸
	3	1.3 x 10 ⁻⁸	1.3 x 10 ⁻⁸	3.7 x 10 ⁻⁸	1.6 x 10 ⁻⁸
Seam Sealed	1	1.2 x 10 ⁻⁹	1.2 x 10 ⁻⁹	1.2 x 10 ⁻⁹	1.0 x 10 ⁻⁹
	2	4.4 x 10 ⁻⁹	2.0 x 10 ⁻⁹	1.0 x 10 ⁻⁹	<1.0 x 10 ⁻⁹
	3	0.9 x 10 ⁻⁹	1.8 x 10 ⁻⁵	1.2 x 10 ⁻⁹	<1.0 x 10 ⁻⁹

Table 8. Simplified Summary of Results of Temperature-Humidity Evaluation

Adhesive	Ten Days 50°C/60% RH	Forty Days 50°C/50% RH	Ten Days 60°C/98% RH	Ten Day Moisture Resistance Test	Ten Days 85°C/85% RH
Ablefilm 507	XXX	XXX	XXX	XXX	XX-
Ablefilm 550	XXX	---	---	XX-	---
Ablebond 36-2	XXX	XX-	XX-	XXX	---
Ablebond 58-1	XXX	---	---	---	---
Epo-Tek H20E	---	---	---	---	---
Epo-Tek H81	XXX	XX-	X--*	---	---
Epo-Tek H77	XXX	---	XX-	XX-	---
Ablebond 789-1	XXX	XXX	XXX	XXX	XXX
Ablebond 673-1	XXX	XXX	X--*	---	---
Seam Sealed	XXX	XXX	XXX	XXX	XXX

X = Package retained seal integrity after exposure

- = Package was gross leaker after exposure

* = Adhesive was eliminated from further consideration

environments and for two adhesives (Ablefilm 550 and Epo-Tek H77) that failed. In each case, spectra were run on uncured samples and on cured samples before and after they were exposed to 85°C/85% RH for ten days. For each adhesive, the spectrum obtained for the uncured sample was compared with that obtained for the cured sample before temperature-humidity exposure to ascertain the degree of cure. Then the spectrum for the cured sample before temperature-humidity exposure was compared with the spectrum for the cured sample after exposure to note any chemical changes resulting from the ten-day 85°C/85% RH exposure.

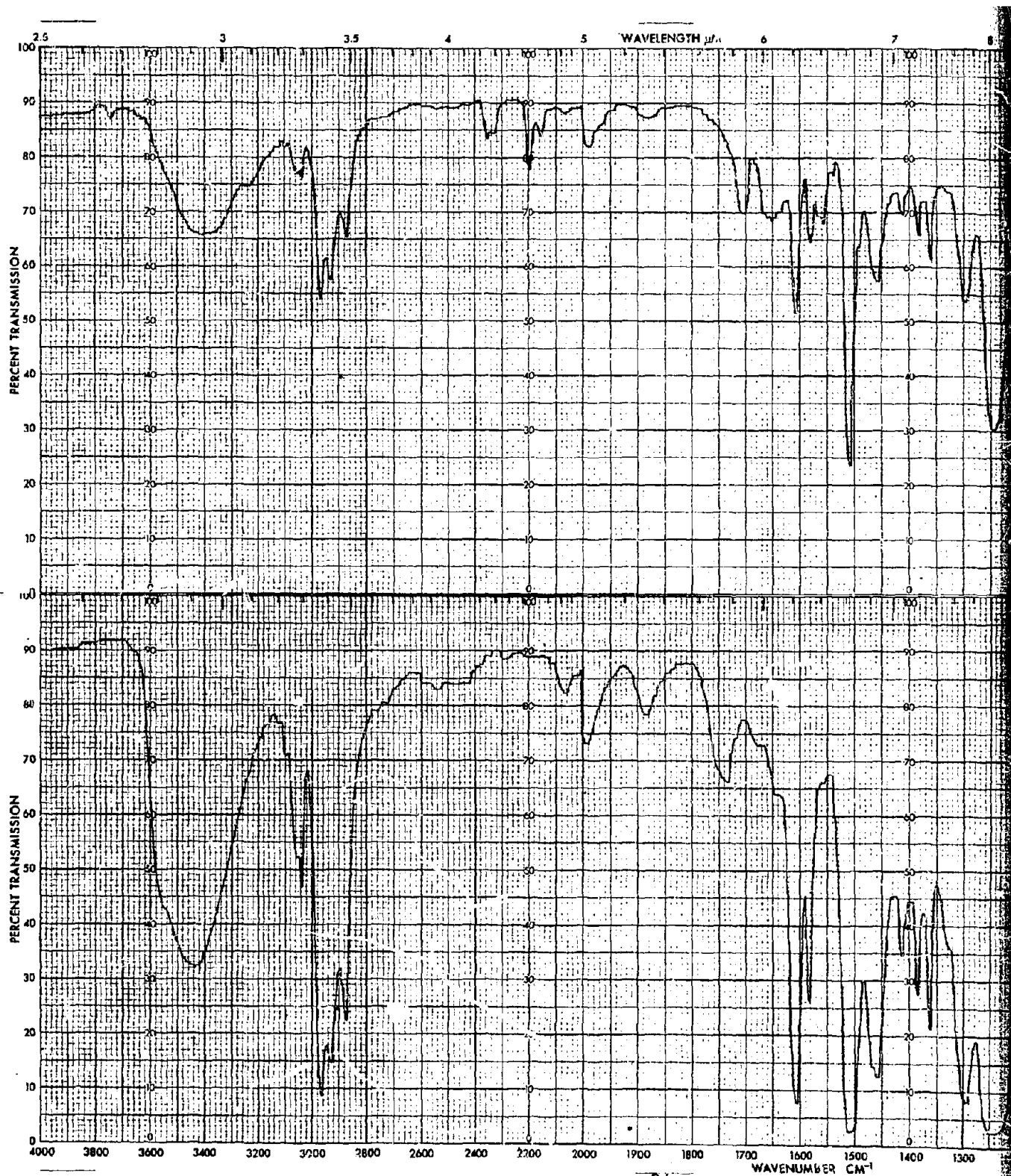
Spectra of uncured and cured samples are shown in Figures 4 and 5 for Ablefilm 507 and Ablefilm 550, respectively. The spectrum for the uncured sample is at the top. Since both of these adhesives are supplied in film form on a fiberglass mat, the adhesives were extracted by dissolving in acetone and evaporating. Ablefilm 507 consists of a DGEBA (diglycidal ether of bis phenol A) resin and a dicyandiamide (Dicy) curing agent. The presence of Dicy is indicated by the nitrile group doublet at 4.56 and 4.65 μm . Comparison of the spectra in Figure 4 shows that this doublet has disappeared in the spectrum of the cured sample indicating that cure was completed. Also, this fact is substantiated by the retreat (decrease in absorption) of the epoxide group absorption band at 10.8 μm . Ablefilm 550 is a nitrile modified DGEBA resin with a dicyandiamide curing agent and an accelerator. Comparison of the spectra for the cured and uncured samples for this adhesive (Figure 5) shows that the doublet characteristic of Dicy has degraded, but that a strong absorption peak still remains at 4.58 μm . This may be due to the nitrile portion of the modified epoxy or to excess unreacted Dicy. The fact that complete curing has occurred is substantiated by the disappearance of the epoxide peak at 10.8 μm .

Spectra of cured samples before and after exposure to 85°C/85% RH for ten days for the four adhesives (Ablefilm 507, Ablefilm 550, Epo-Tek H77, and Ablebond 789-1) are shown in Figures 6 through 9, respectively. In all cases, the spectrum for the cured adhesive before exposure is at the top. Comparison of the spectra in Figures 6, 8 and 9 for Ablefilm 507, Epo-Tek H77, and Ablebond 789-1 indicates that they are essentially the same. Therefore, the conclusion is that the chemical structures of these adhesives have not been affected by the temperature-humidity exposure. Comparison of the spectra for

EXHIBIT FRAME 1

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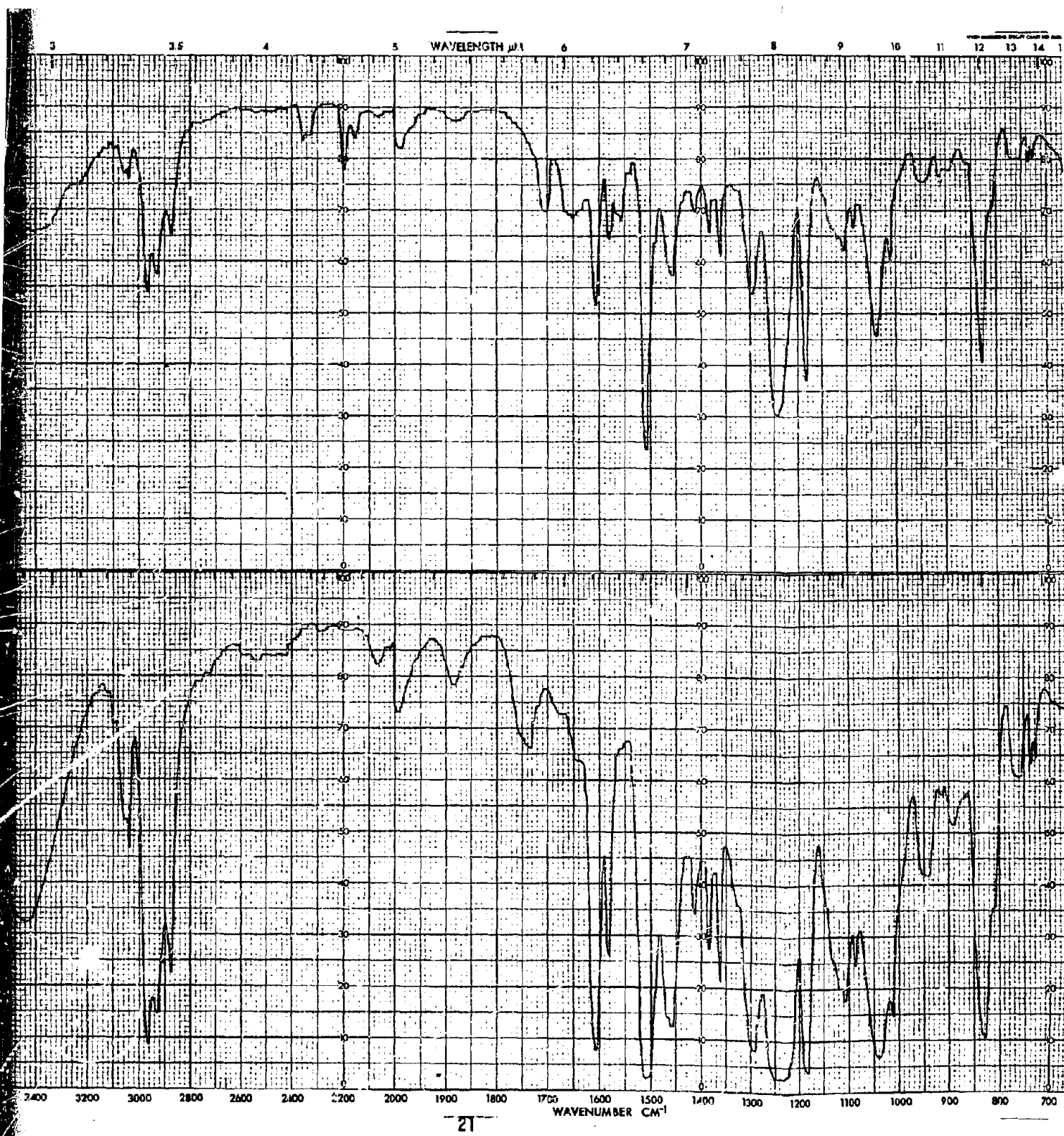
Figure 4. IR Spectra for Ablefilm 507
Top - Uncured
Bottom - Cured (70 Min. at 175°C)



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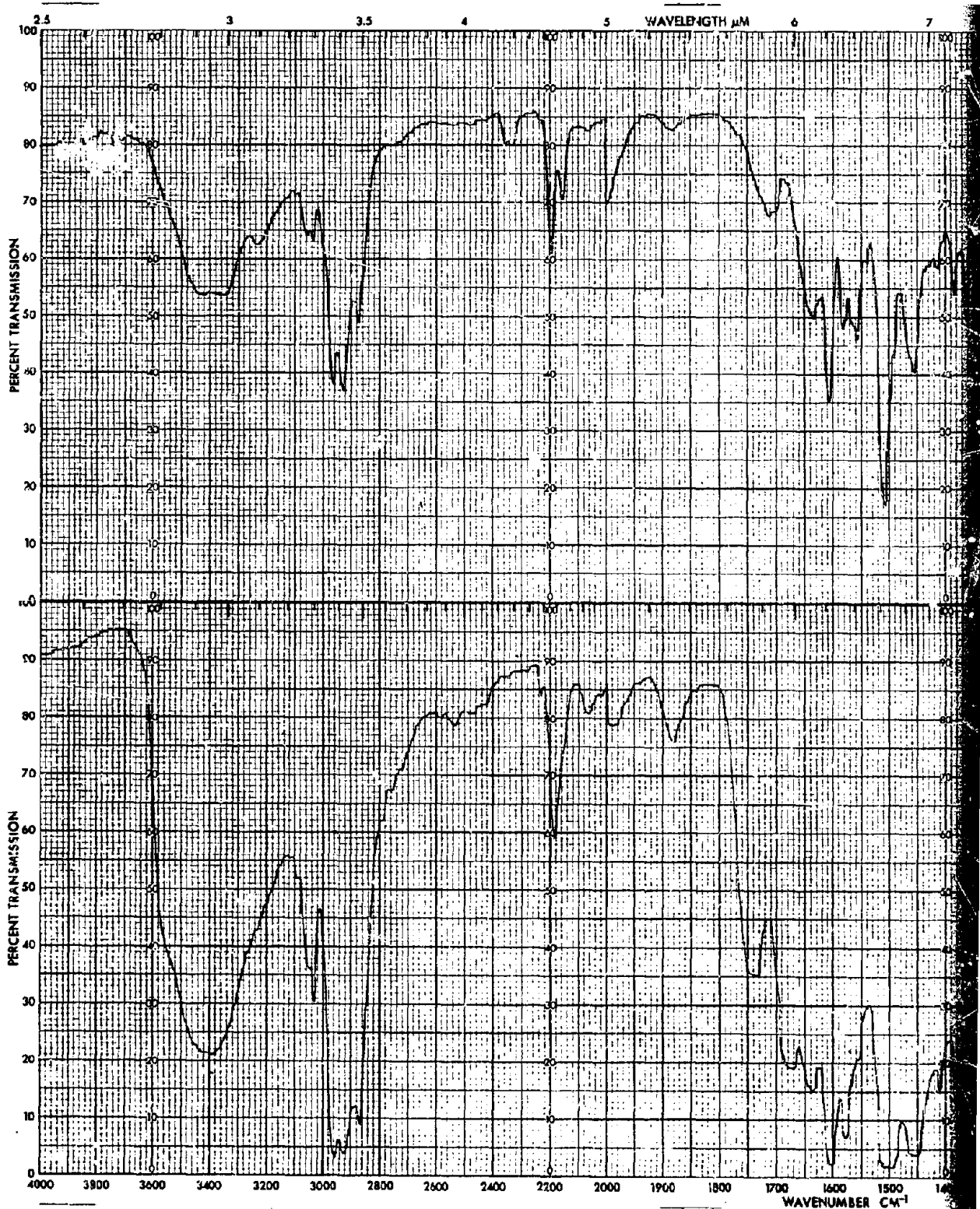
Figure 4. IR Spectra for Ablefilm 507
Top - Uncured
Bottom - Cured (70 Min. at 175°C)



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Figure 5. IR Spectra for Ablefilm 550
Top - Uncured
Bottom - Cured (2-1/2 Hrs. at 150°C)



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Figure 5. IR Spectra for Ablefilm 550
Top - Uncured
Bottom - Cured (2-1/2 Hrs. at 150°C)

~~SECRET~~ **FRAME 2**

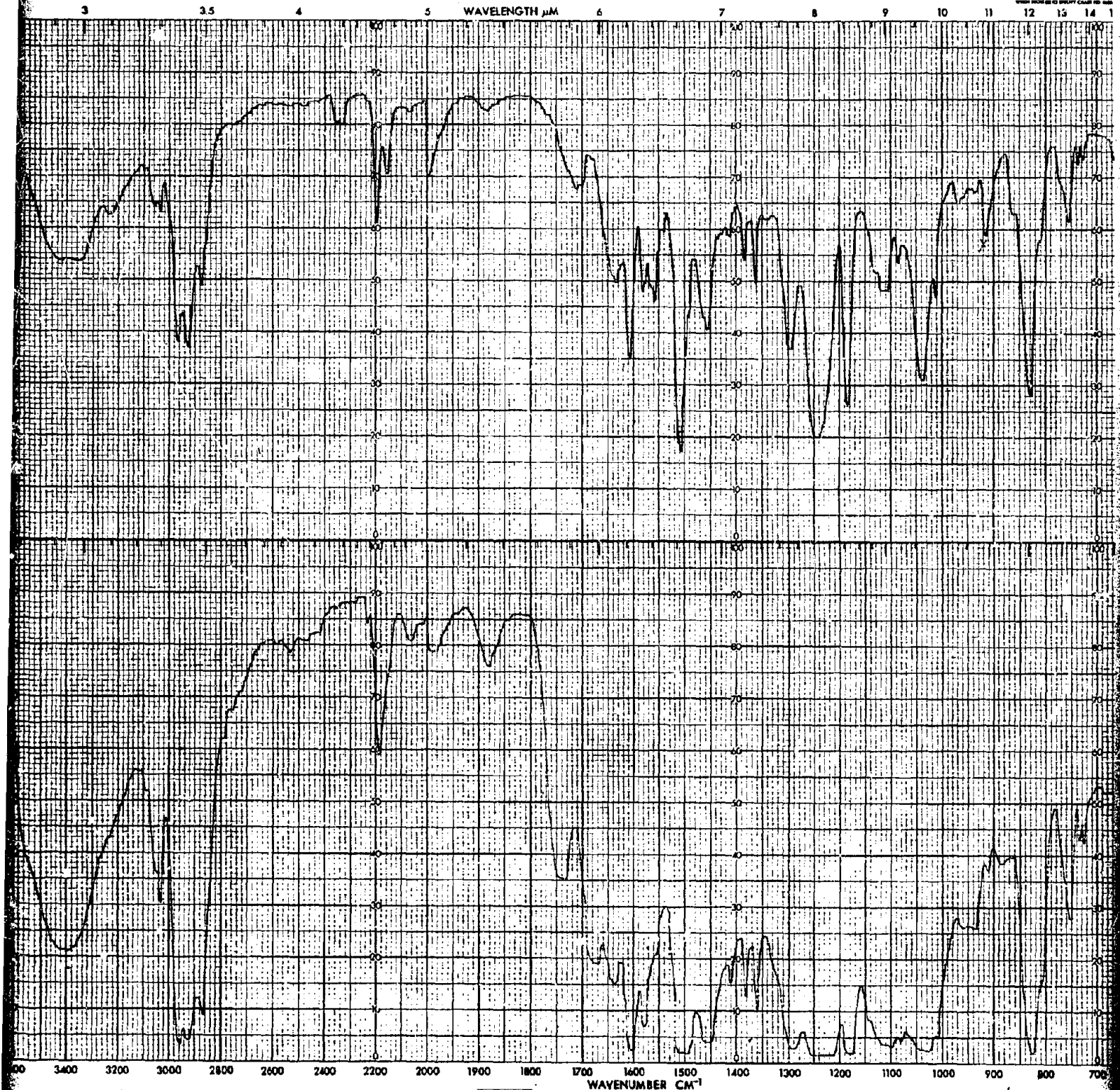
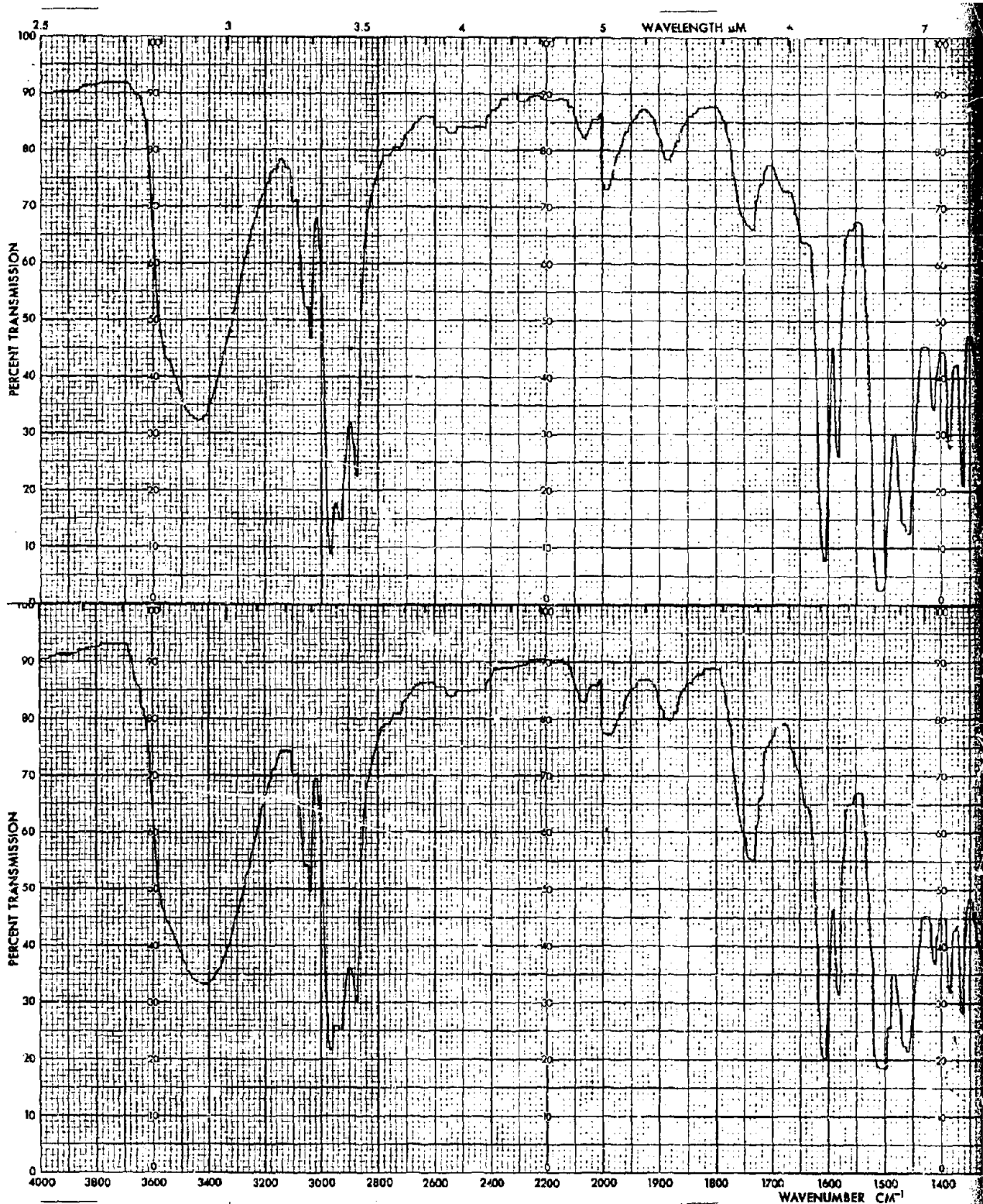


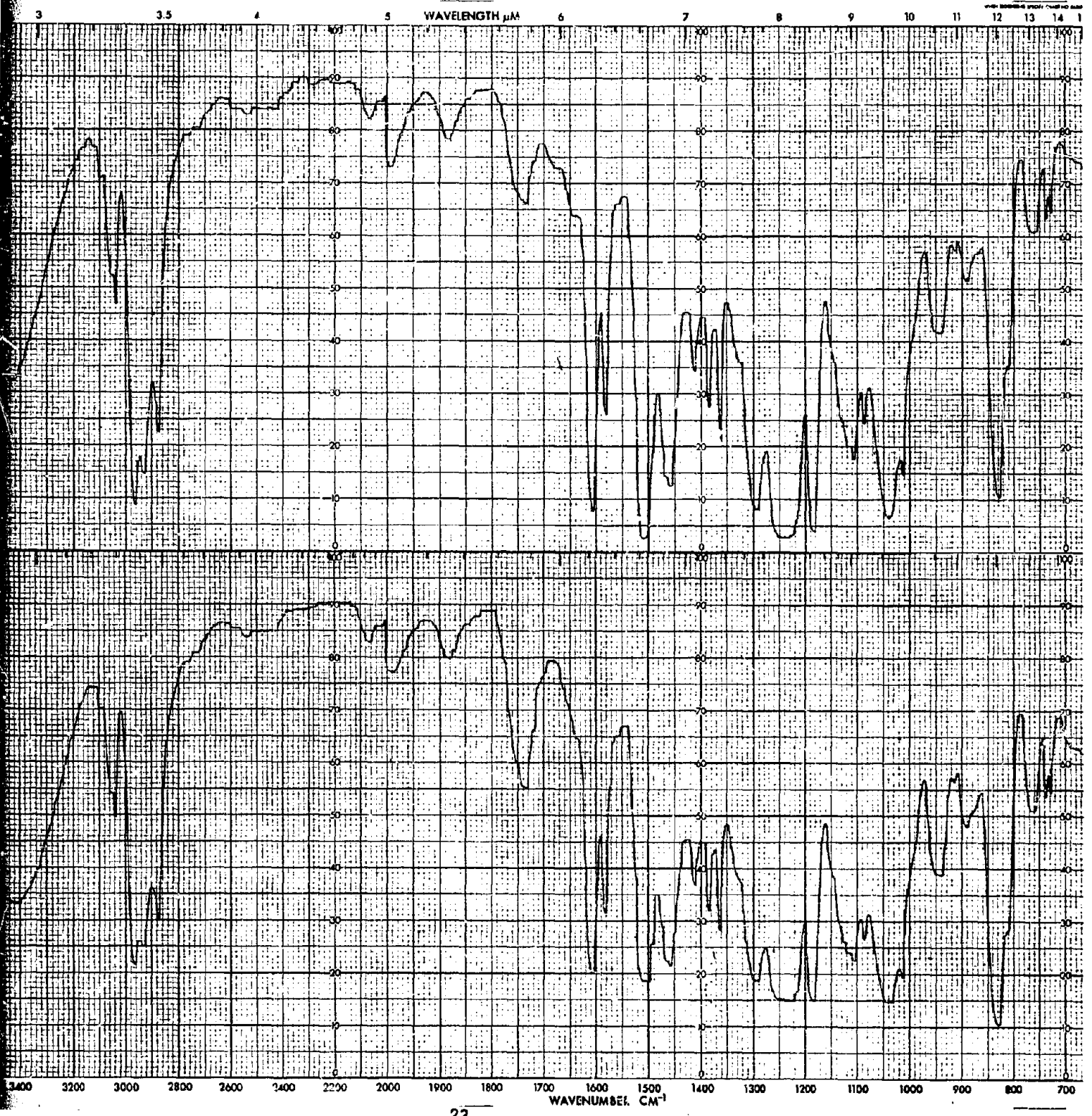
Figure 6. IR Spectra for Ablefilm 507
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



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~~FOR USE~~ FRAME 2

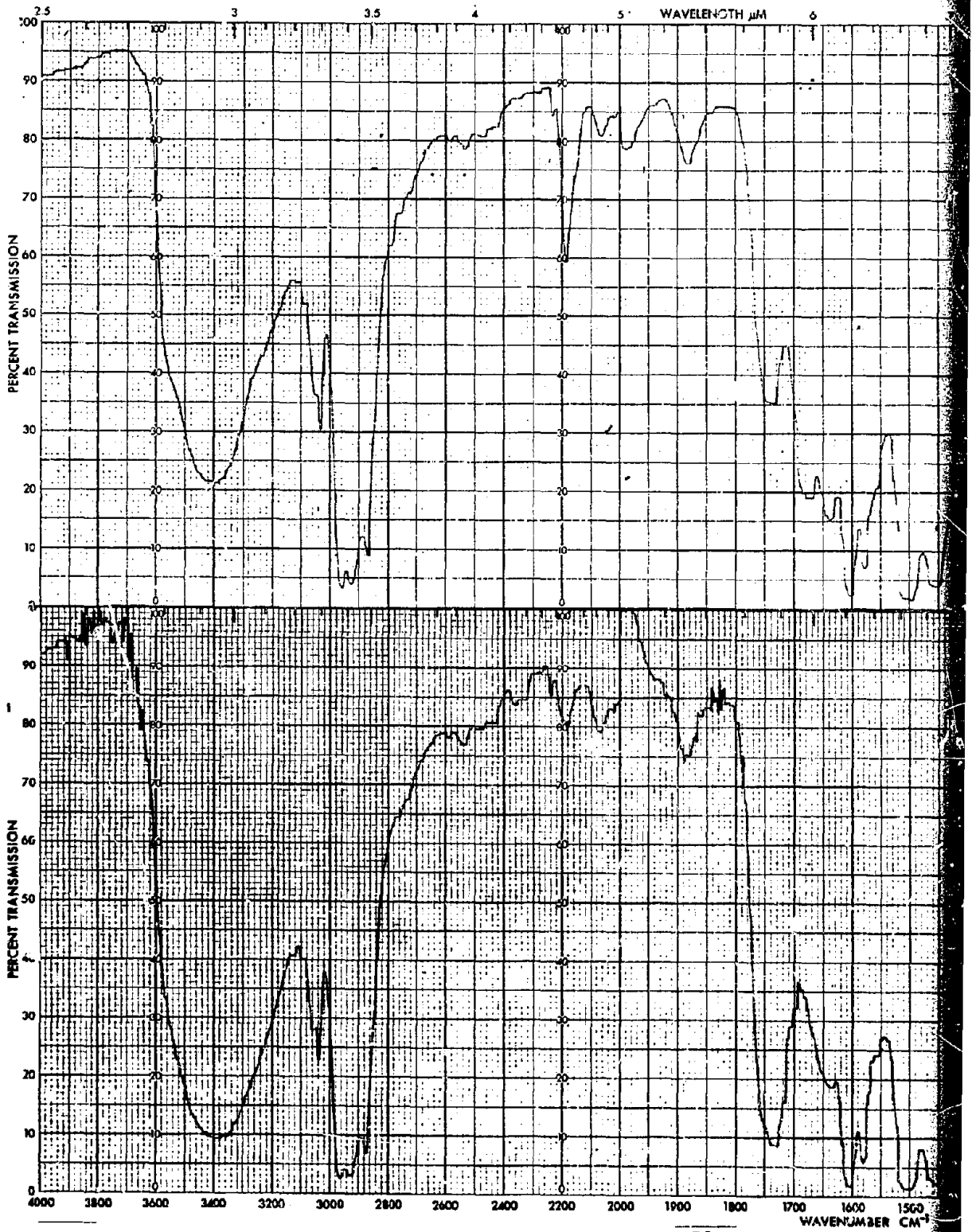
Figure 6. IR Spectra for Ablefilm 507
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



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Figure 7. IR Spectra for Ablefilm 550
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



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Figure 7. IR Spectra for Ablefilm 550
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure

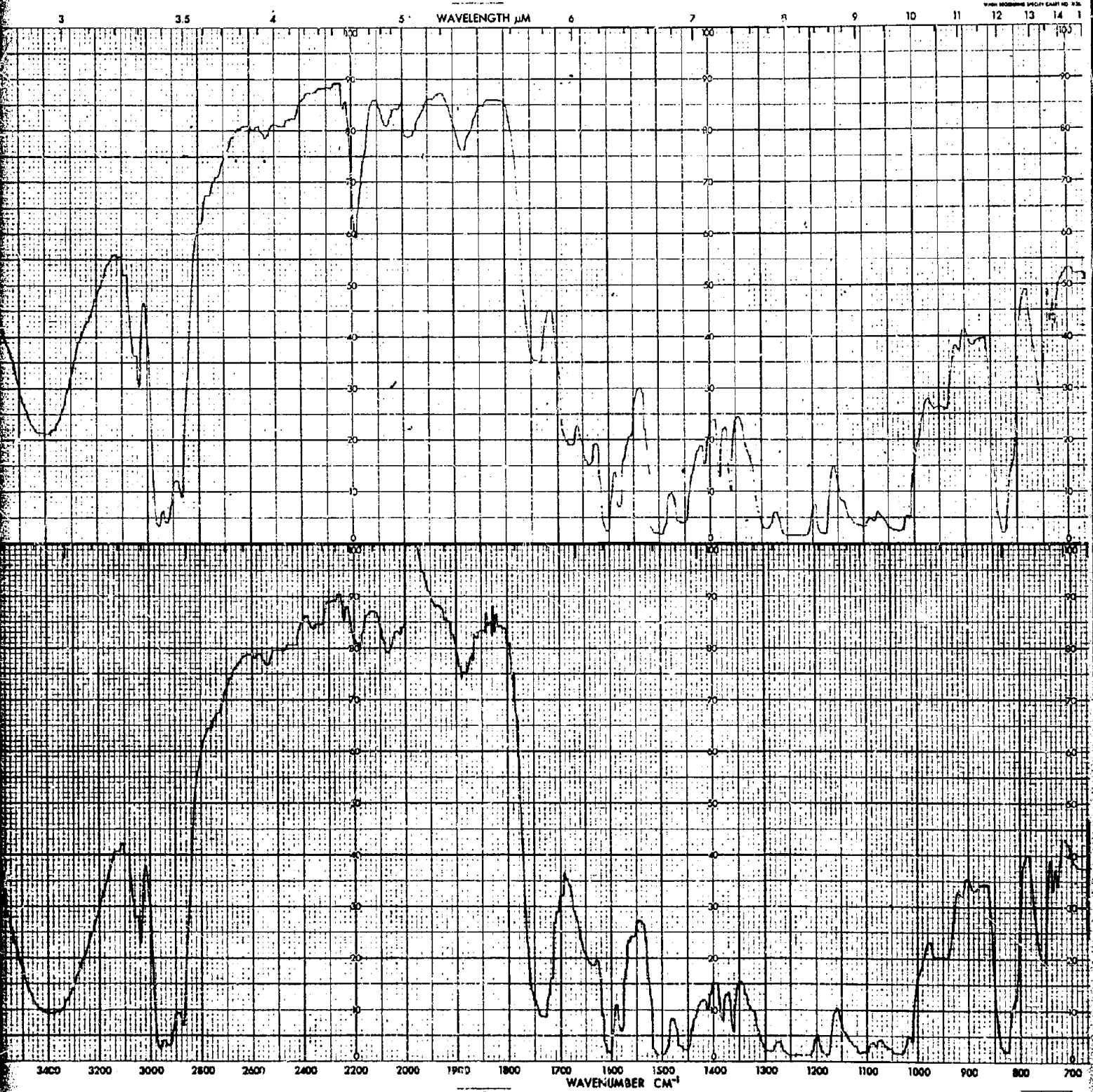
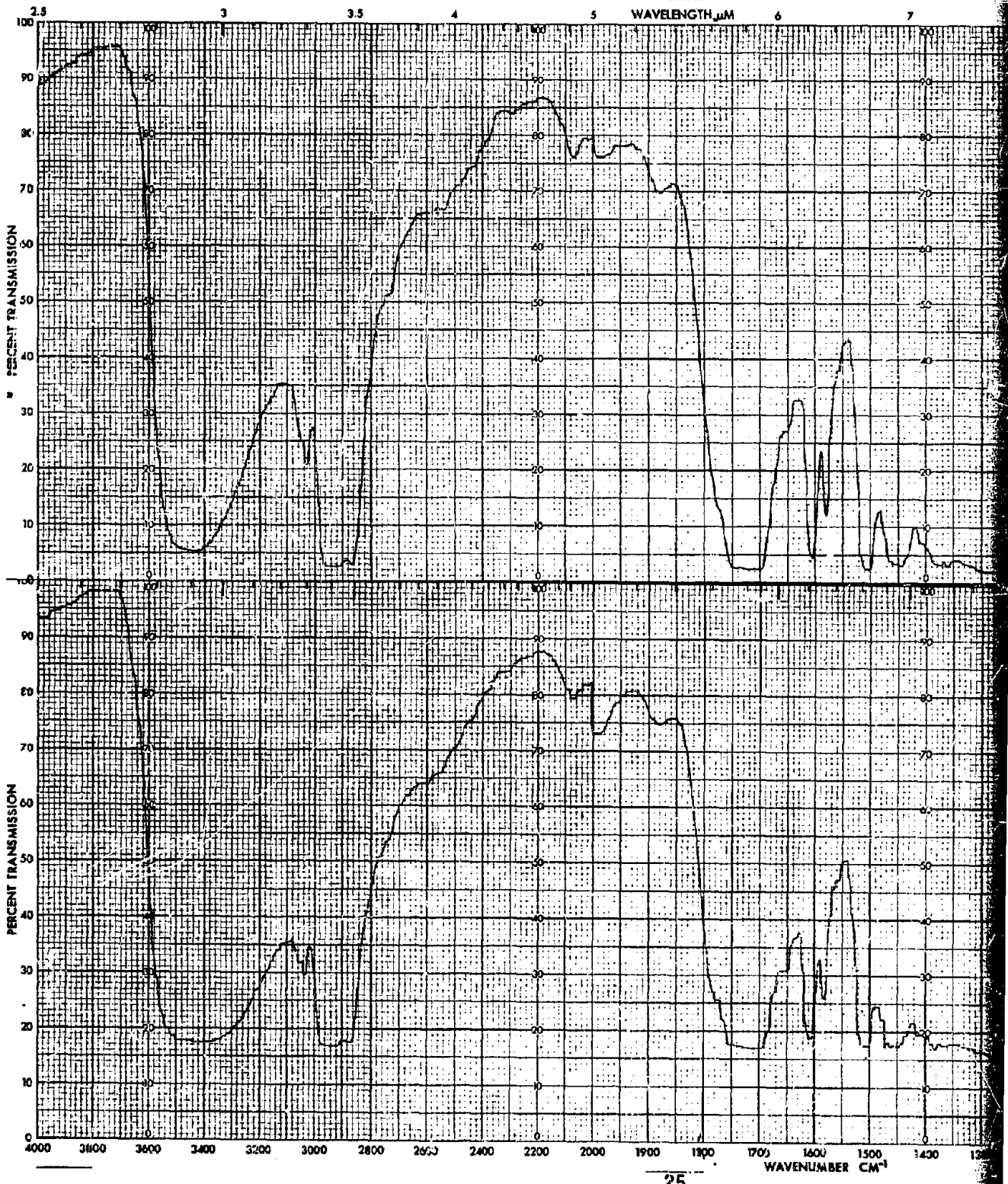


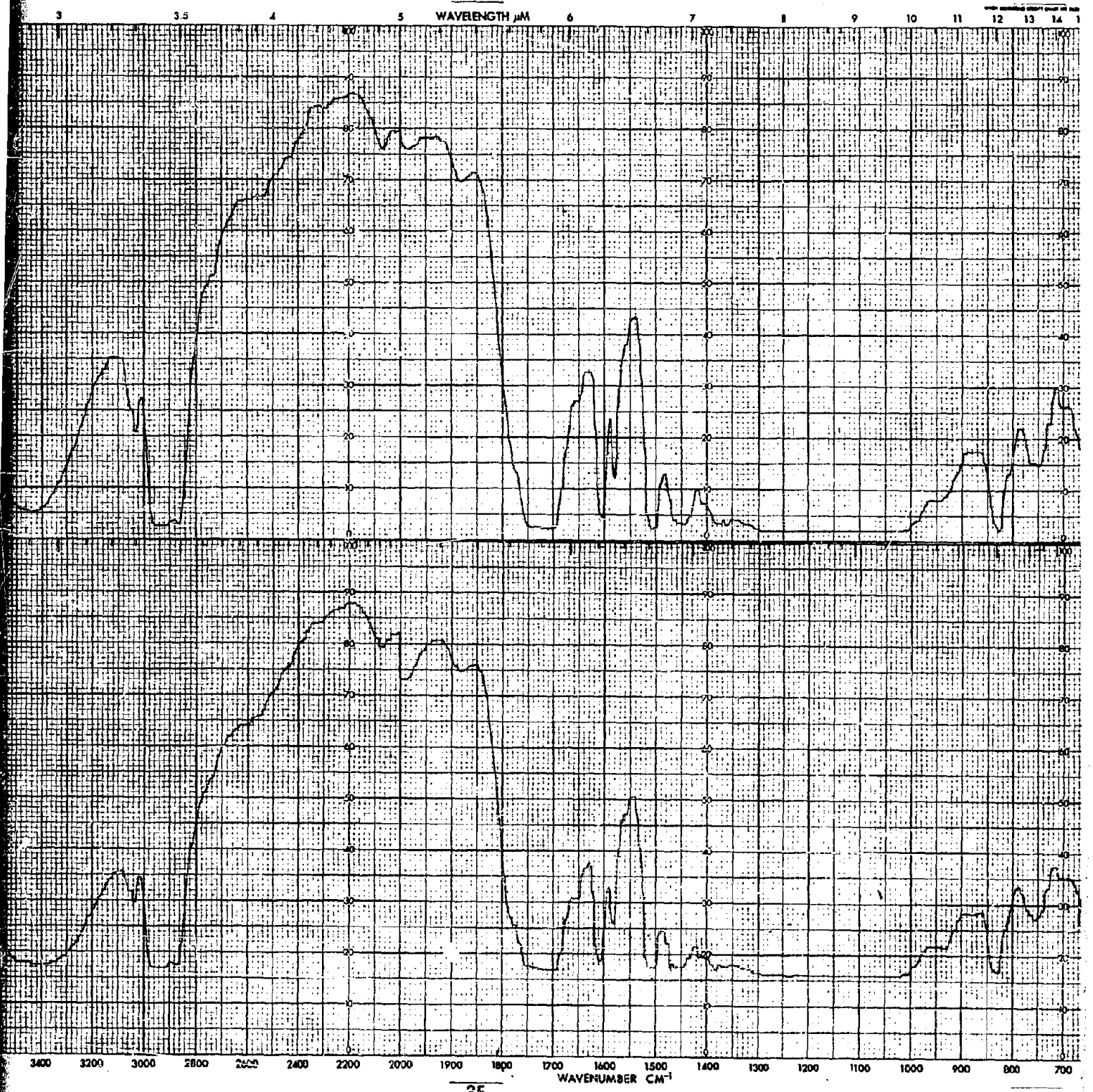
Figure 8. IR Spectra for Epo-Tek H77
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



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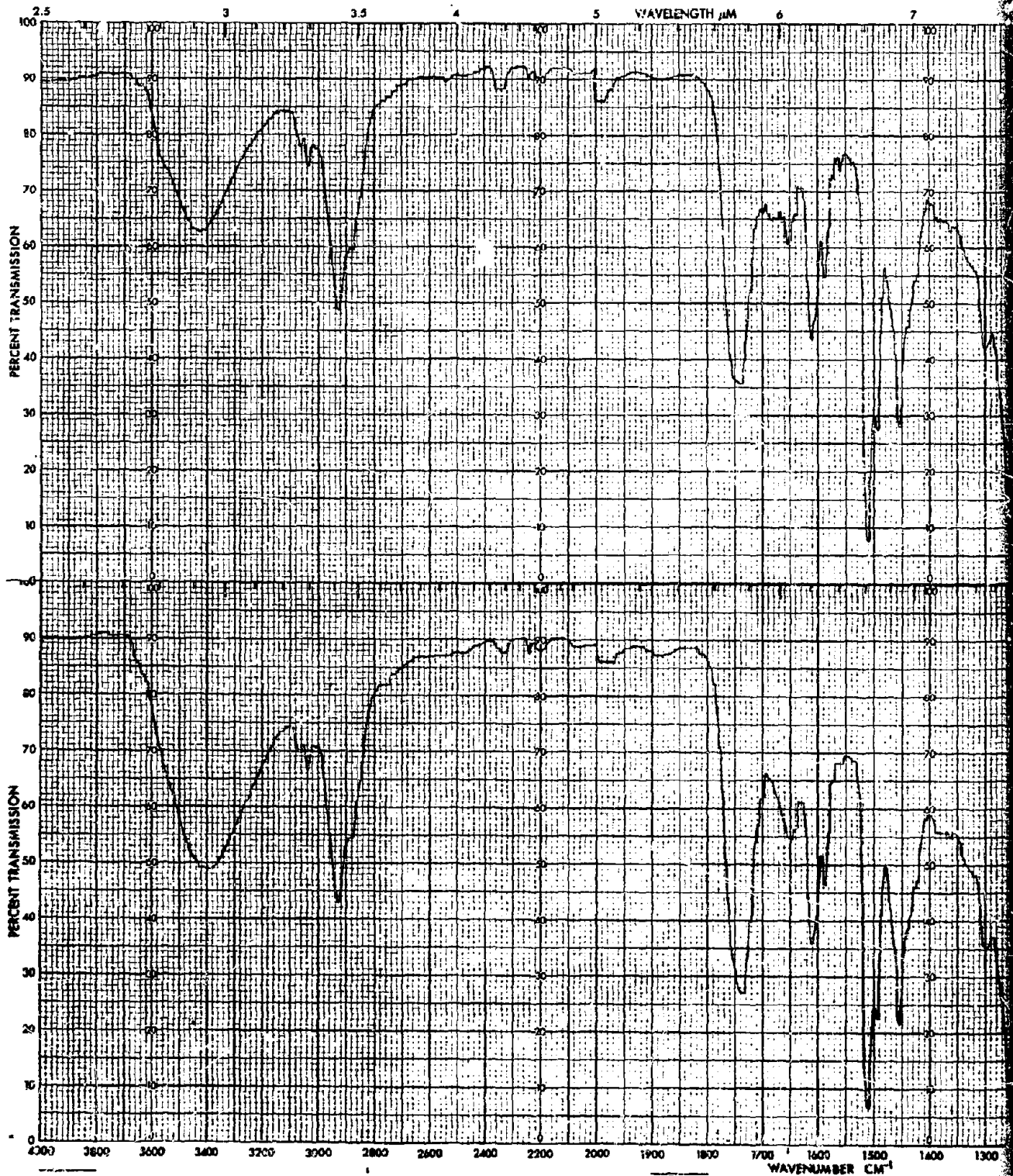
Figure 8. IR Spectra for Epo-Tek H77
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



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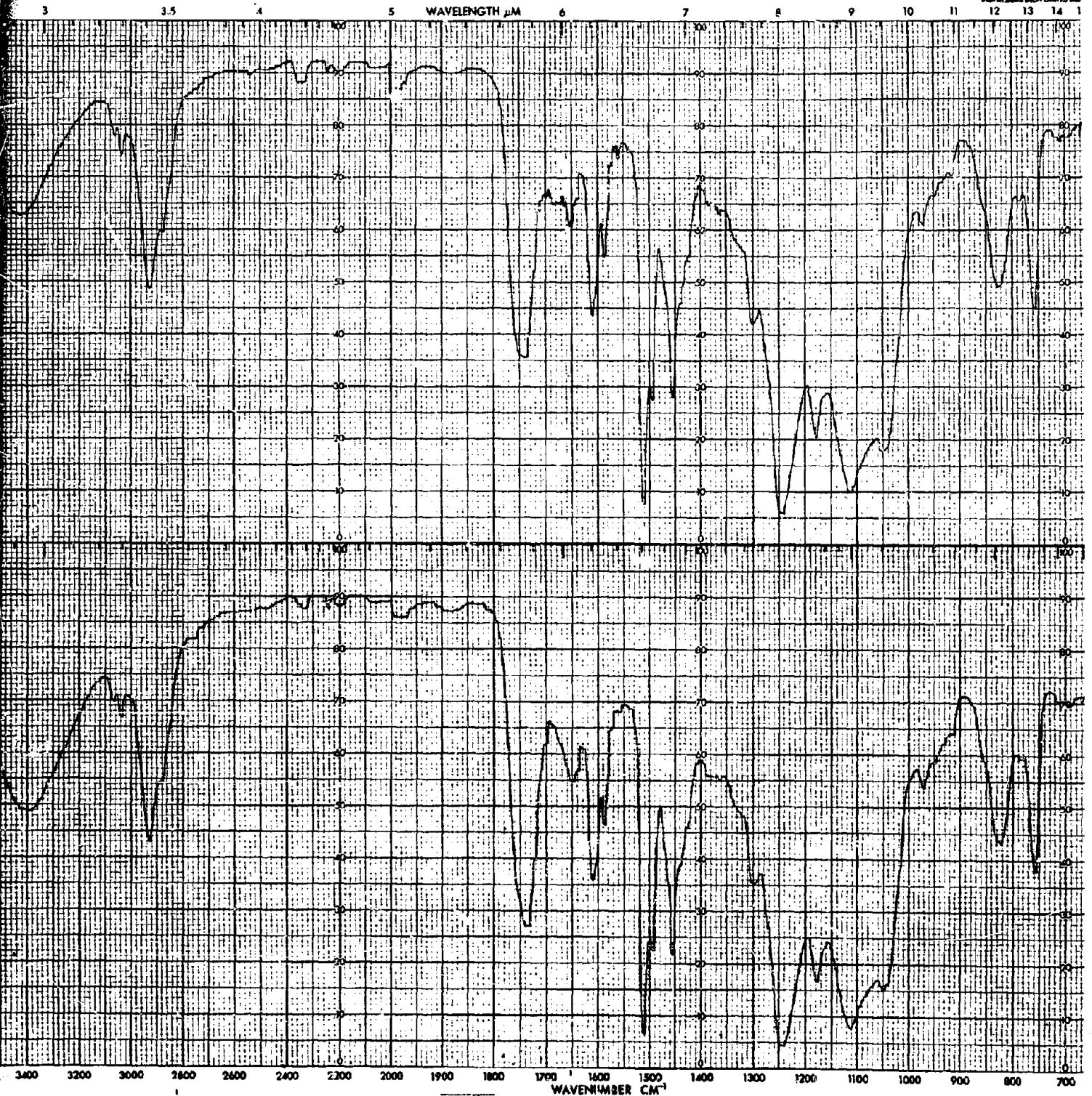
Figure 9. IR Spectra for Ablebond 789-1
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



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Figure 9. IR Spectra for Ablebond 789-1
Top - Before Ten Days Exposure at 85°C/85% RH
Bottom - After Exposure



Ablefilm 550 (Figure 7), however, shows a substantial reduction in the absorption band at 4.58 μm , indicating that the nitrile group has been hydrolyzed to a carboxyl group during the temperature-humidity exposure. This conclusion is supported by the growth (increased absorption) of the carbonyl peak (5.7 to 5.8 μm). Since the purpose of adding the nitrile was to increase the ability of the adhesive to adhere to gold, its hydrolysis during temperature-humidity exposure could explain why the performance of this adhesive (Ablefilm 550) was degraded.

In conclusion then, comparison of the IR spectra before and after temperature-humidity exposure suggests a possible reason for the failure of Ablefilm 550 but not for the failure of Epo-Tek H77.

2.2 EFFECT OF MIL-STD-883A TEST ENVIRONMENTS ON SEAL INTEGRITY

The objective of this effort was to determine if gold-plated Kovar packages and/or ceramic packages sealed with the four best adhesives identified in the previous effort, retain their seal integrity after they have been subjected to the following Class A test environments specified in Method 5004.2 of MIL-STD-883A:

- (1) Thermal Shock - Method 1011.1, Test Condition C (i.e., 15 cycles, -65°C to +150°C)
- (2) Temperature Cycling - Method 1010.1, Test Condition C (i.e., 15 cycles, -65°C to +150°C)
- (3) Mechanical Shock - Method 2002.1, Test Condition B (i.e., 5 shock pulses at 1500 g's in the Y_1 plane)
- (4) Constant Acceleration - Method 2001.1, Test Condition A (i.e., 5000 g's in the Y_1 plane)
- (5) Temperature Aging (240 hours at 125°C)

To accomplish this objective, six gold-plated Kovar packages and six ceramic packages sealed with each of the four adhesives (Ablefilm 507, Ablebond 36-2, Epo-Tek H77, and Ablebond 789-1) were subjected to the test environments both individually and sequentially in the order listed. The integrity of the package seals was determined before and after exposure to each test environment by testing the packages in accordance with MIL-STD-883A, Method 1014.1, Test Conditions A_2 , and C_1 and C_2 . In each case, six seam-sealed gold-plated Kovar packages were used as controls.

2.2.1 Packages and Package Processing and Assembly

The gold-plated Kovar packages selected for testing were the same as those used previously (described in Section 2.1.2). The ceramic packages (or boxes since they did not have electrical feedthroughs) selected were 2.29 cms (0.9 inches) square. These packages have a 0.127 cm (50 mils) wide rim and a sealing area of 1.16 cm² (0.18 inch²). Ceramic lids 0.064 cm (25 mils) thick were laser cut to fit these boxes and a hole approximately 0.04 cm (16 mils) in diameter was sandblasted in them so that the packages could vent during cure.

The same package assembly method described in Section 2.1.1 was used. An assembly fixture similar to the one used to assemble the

gold-plated Kovar packages was fabricated to accommodate the smaller ceramic packages, and smaller teflon coated steel backing plates were made. The same cleaning procedure as that described in Section 2.1.2 was used for the gold-plated Kovar packages. The ceramic packages were cleaned by brushing them in toluene and isopropyl alcohol and then spray rinsing them with Freon TF. This cleaning procedure was selected because of the necessity of pretesting the ceramic boxes to be sure that they did not contain cracks or holes. Testing was performed using a helium-leak detector and required the use of Apiezon N grease on the rims of the packages to form a temporary seal. Since any residual Apiezon N would degrade the adhesive seals, it had to be completely removed. Toluene proved to be a very effective solvent for this purpose.

The remainder of the package processing and sealing procedures were the same as those described in Section 2.1.2, except that the vent holes in the ceramic packages were sealed with Epo-Tek H77. The cure schedules and clamping pressures used in preparing these packages also were the same as those used previously in preparing packages for the temperature-humidity evaluation effort. Clamping pressures applied during cure were approximately $7.0 \times 10^4 \text{ N/m}^2$ (10 psi) for the paste adhesives, and $1.1 \times 10^5 \text{ N/m}^2$ (16 psi) for the film adhesive. Curing schedules were:

Ablefilm 507	70 Minutes at 175°C
Ablebond 36-2	40 Minutes at 150°C
Epo-Tek H77	30 Minutes at 150°C
Ablebond 789-1	2 Hours at 170°C

2.2.2 Comments on Ceramic Packages and Cleaning Methods

Initial seal testing of the first group of adhesive-sealed ceramic packages revealed that 16 of 48 were C_1 gross leakers with the leaks occurring not at the seal area, but through the center portion of the bottom of the ceramic packages. This was unexpected since the packages were bought from 3M (American Lava Division) in accordance with an existing company specification. As a result of this experience, it was decided to pretest the ceramic packages for hermeticity. Testing of 200 packages showed that approximately 50% failed. Apparently small holes or cracks existed due either to inadequate compaction of the alumina or a poorly controlled firing schedule. The significance of this unexpected situation is that it flags a problem area associated with the

use of ceramic packages. The situation experienced may not be typical but it does indicate that a large portion of a batch of ceramic packages can be leakers, and that pretesting should be considered by manufacturers using ceramic packages in order to minimize their circuit rework or rejection rates (and associated costs).

Since the present method for cleaning the gold-plated Kovar packages and lids (lightly scrubbing them successively in deionized water, acetone, and isopropanol and spray rinsing with Freon TF) was arbitrarily selected and is time consuming, it was decided to clean them using the simple, standard, automated procedure of successively immersing them in four ultrasonically-agitated Freon TF baths followed by a Freon TF vapor rinse. Packages were cleaned by this method and 12 each were sealed with Ablebond 36-2 and Epo-Tek H77. Initial seal testing of the packages showed that all 24 were C₁ gross leakers. At this point, packages were cleaned by the original method and 12 each sealed with Ablebond 36-2 and Epo-Tek H77. Of this group, 23 passed the seal test (one package sealed with Ablebond 36-2 was a C₂ gross leaker). On this basis, it was decided to retain the original cleaning method. It is felt that the demonstrated superiority of the method used is essentially due to the effectiveness of acetone and isopropanol in removing fingerprint contamination accumulated during handling (particularly when the small holes are drilled in the lids).

2.2.3 Results for Thermal Shock Testing

Results for the packages subjected to 15 cycles of thermal shock between -65°C and +150°C (MIL-STD-883A, Method 1011.1, Test Condition C) are given in Tables 9 and 10. As shown, all ceramic packages retained their seal integrity after exposure to this environment, but all the gold-plated Kovar packages failed except those that were seam sealed and five of the six that were sealed with Ablefilm 507. The other Ablefilm 507 sealed package had a fine leak rate of 1.6×10^{-7} atm cc/sec (air equivalent) similar to the fine leak rates of those that retained their seal integrity, and also passed the C₁ gross leak test, but was found to be a gross leaker when tested in accordance with Test Condition C₂. All the Ablebond 36-2 and Epo-Tek H77 sealed gold-plated Kovar packages were C₁ gross leakers, while one of those sealed with Ablebond 789-1 was a C₁ gross leaker and the other five were C₂ gross leakers.

Table 9. Effect of Thermal Shock on Seal Integrity of Gold-Plated Kovar Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Thermal Shock 15 Cycles (-65°C, +150°C) Air Equivalent (atm cc/sec)
Ablefilm 507		
1	9.8×10^{-8}	1.7×10^{-7}
2	1.2×10^{-7}	1.9×10^{-7}
3	9.2×10^{-8}	1.9×10^{-7}
4	9.6×10^{-8}	1.4×10^{-7}
5	8.4×10^{-8}	1.8×10^{-7}
6	7.4×10^{-8}	1.6×10^{-7} , Gross (C ₂)
Ablebond 36-2		
1	2.1×10^{-7}	2.6×10^{-7} , Gross (C ₁)
2	1.5×10^{-7}	$>3.0 \times 10^{-6}$, Gross (C ₁)
3	1.9×10^{-7}	$>3.0 \times 10^{-6}$, Gross (C ₁)
4	1.5×10^{-7}	$>3.0 \times 10^{-6}$, Gross (C ₁)
5	1.7×10^{-7}	4.2×10^{-7} , Gross (C ₁)
6	1.2×10^{-7}	2.3×10^{-7} , Gross (C ₁)
Epo-Tek H77		
1	5.0×10^{-8}	$>3.0 \times 10^{-6}$, Gross (C ₁)
2	6.8×10^{-8}	Lid came off during fine leak test
3	2.2×10^{-8}	6.8×10^{-8} , Gross (C ₁)
4	3.6×10^{-8}	$>3.0 \times 10^{-6}$, Gross (C ₁)
5	5.6×10^{-8}	$>3.0 \times 10^{-6}$, Gross (C ₁)
6	1.4×10^{-8}	1.5×10^{-8} , Gross (C ₁)
Ablebond 789-1		
1	1.9×10^{-7}	$>3.0 \times 10^{-6}$, Gross (C ₂)
2	1.8×10^{-7}	$>3.0 \times 10^{-6}$, Gross (C ₂)
3	1.0×10^{-7}	$>3.0 \times 10^{-6}$, Gross (C ₁)
4	8.0×10^{-8}	$>3.0 \times 10^{-6}$, Gross (C ₂)
5	4.9×10^{-8}	$>3.0 \times 10^{-6}$, Gross (C ₂)
6	4.2×10^{-8}	$>3.0 \times 10^{-6}$, Gross (C ₂)
Seam Sealed		
1	$<1.0 \times 10^{-9}$	3.4×10^{-9}
2	$<1.0 \times 10^{-9}$	2.6×10^{-9}
3	$<1.0 \times 10^{-9}$	3.4×10^{-9}
4	$<1.0 \times 10^{-9}$	3.6×10^{-9}
5	$<1.0 \times 10^{-9}$	4.0×10^{-9}
6	$<1.0 \times 10^{-9}$	2.0×10^{-9}

Table 10. Effect of Thermal Shock on Seal Integrity of Ceramic Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Thermal Shock 15 Cycles (-65°C, +150°C) Air Equivalent (atm cc/sec)
Ablefilm 507		
1	1.3×10^{-7}	1.5×10^{-7}
2	2.8×10^{-8}	9.2×10^{-8}
3	2.4×10^{-8}	1.4×10^{-7}
4	4.0×10^{-8}	1.6×10^{-7}
5	1.6×10^{-7}	1.0×10^{-7}
6	2.0×10^{-7}	1.6×10^{-7}
Ablebond 36-2		
1	1.0×10^{-7}	2.2×10^{-7}
2	1.2×10^{-7}	1.6×10^{-7}
3	1.0×10^{-7}	1.4×10^{-7}
4	7.6×10^{-8}	1.8×10^{-7}
5	5.8×10^{-8}	6.5×10^{-8}
6	8.0×10^{-8}	1.0×10^{-7}
Epo-Tek H77		
1	5.0×10^{-8}	2.2×10^{-8}
2	5.0×10^{-8}	3.4×10^{-8}
3	1.3×10^{-7}	6.8×10^{-8}
4	3.4×10^{-8}	2.6×10^{-8}
5	6.8×10^{-8}	6.0×10^{-8}
6	9.2×10^{-8}	9.0×10^{-8}
Ablebond 789-1		
1	1.8×10^{-7}	1.7×10^{-7}
2	1.7×10^{-7}	1.6×10^{-7}
3	6.5×10^{-8}	1.5×10^{-7}
4	1.5×10^{-7}	1.1×10^{-7}
5	1.2×10^{-7}	1.2×10^{-7}
6	7.4×10^{-8}	1.7×10^{-7}

It is felt that the explanation of the contrasting results obtained for the ceramic and the gold-plated Kovar packages lies simply in the well-known fact that adhesives form a stronger bond with alumina than they do with gold. The result not so easily explained is that the gold-plated Kovar packages sealed with the film adhesive retained their seal integrity while those sealed with the three paste adhesives did not. A possible explanation is that the bond lines were adhesive starved in the case of the paste adhesives due to excessive clamping pressure, whereas this could not be the case for the film adhesive since it has a 0.005 cm (2 mil) glass carrier.

The measured fine-leak rates are given for all gold-plated Kovar packages including those which were subsequently found to be gross leakers. This has been done to emphasize two points. First, some packages which pass the fine-leak test are subsequently found to be gross leakers when subjected to the conditions of the gross-leak tests. As shown in Table 9, this is the case for Package 6 sealed with Ablefilm 507, Packages 1, 5 and 6 sealed with Ablebond 36-2, and Packages 3 and 6 sealed with Epo-Tek H77. Second, the C_2 gross-leak test is a more sensitive test than the C_1 gross-leak test and consequently, gross leakers that may escape detection by the C_1 test are often caught by the C_2 test. (this is the case for Package 6 sealed with Ablefilm 507). These results substantiate the well known fact that both fine and gross leak tests are necessary to assure that seals of adequate strength and integrity are obtained.

2.2.4 Results for Temperature Cycling Testing

Results for the packages temperature cycled in accordance with MIL-STD-883A, Method 1010.1, Test Condition C (15 cycles between -65°C and $+150^{\circ}\text{C}$) are given in Tables 11 and 12. All the adhesive sealed ceramic packages retained their seal integrity. However, all the adhesive sealed gold-plated Kovar packages failed except those sealed using Ablefilm 507.

In review, both the results of the effect of thermal shock on seal integrity and the effect of temperature cycling on seal integrity show that ceramic packages sealed with all four selected adhesives (Ablefilm 507, Ablebond 36-2, Epo-Tek H77 and Ablebond 789-1) retain their seal integrity after exposure to these environments, but the only gold-plated Kovar packages that retain their seal integrity after these exposures are those that were seam sealed and those sealed with Ablefilm 507. It was felt that perhaps the failure of the paste adhesives on the

Table 11. Effect of temperature Cycling on Seal Integrity of Gold-Plated Kovar Packages

Adhesive Package No.	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Temperature Cycling 15 Cycles (-65°C, +150°C) Air Equivalent (atm cc/sec)
Ablefilm 507		
7	1.2×10^{-7}	3.0×10^{-7}
8	7.2×10^{-8}	1.5×10^{-7}
9	8.2×10^{-8}	2.0×10^{-7}
10	6.4×10^{-8}	1.4×10^{-7}
11	5.6×10^{-8}	1.3×10^{-7}
12	6.4×10^{-8}	1.5×10^{-7}
Ablebond 36-2		
7	1.8×10^{-7}	Gross (C ₁)
8	8.0×10^{-8}	Gross (C ₁)
9	1.3×10^{-7}	Gross (C ₁)
10	2.2×10^{-7}	Gross (C ₁)
11	2.8×10^{-7}	Gross (C ₁)
12	1.4×10^{-7}	Gross (C ₁)
Epo-Tek H77		
7	1.1×10^{-7}	Gross (C ₁)
8	3.6×10^{-8}	Gross (C ₁)
9	2.7×10^{-8}	Gross (C ₁)
10	3.7×10^{-8}	Gross (C ₁)
11	4.4×10^{-8}	Gross (C ₁)
12	1.0×10^{-7}	Gross (C ₂)
Ablebond 789-1		
7	6.3×10^{-8}	Gross (C ₂)
8	3.0×10^{-8}	Gross (C ₂)
9	1.5×10^{-8}	Gross (C ₁)
10	2.2×10^{-8}	Gross (C ₂)
11	2.1×10^{-8}	Gross (C ₂)
12	1.6×10^{-8}	Gross (C ₂)
Seam Sealed		
7	$<1.0 \times 10^{-9}$	2.0×10^{-9}
8	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
9	$<1.0 \times 10^{-9}$	1.0×10^{-9}
10	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
11	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
12	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$

Table 12. Effect of Temperature Cycling on Seal Integrity of Ceramic Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent ($\mu\text{m cc/sec}$)	Leak Rate After Temperature Cycling 15 Cycles (-65°C , $+150^\circ\text{C}$) Air Equivalent ($\mu\text{m cc/sec}$)
Ablefilm 507		
7	1.6×10^{-8}	2.0×10^{-7}
8	9.0×10^{-8}	2.8×10^{-7}
9	2.1×10^{-8}	1.3×10^{-7}
10	1.4×10^{-8}	1.2×10^{-7}
11	1.7×10^{-7}	2.5×10^{-7}
12	2.8×10^{-8}	2.0×10^{-7}
Ablebond 36-2		
7	8.2×10^{-8}	2.3×10^{-7}
8	8.4×10^{-8}	2.3×10^{-7}
9	3.4×10^{-7}	3.0×10^{-7}
10	9.4×10^{-8}	2.4×10^{-7}
11	3.8×10^{-8}	1.1×10^{-7}
12	7.2×10^{-8}	2.0×10^{-7}
Epo-Tek H77		
7	1.0×10^{-7}	1.8×10^{-7}
8	1.5×10^{-8}	8.4×10^{-8}
9	2.5×10^{-8}	1.0×10^{-7}
10	1.6×10^{-8}	8.7×10^{-8}
11	1.0×10^{-8}	4.9×10^{-8}
12	1.2×10^{-8}	5.6×10^{-8}
Ablebond 789-1		
7	1.1×10^{-7}	1.5×10^{-7}
8	2.0×10^{-7}	7.7×10^{-8}
9	1.0×10^{-7}	1.0×10^{-7}
10	7.6×10^{-8}	8.3×10^{-8}
11	1.6×10^{-7}	9.6×10^{-8}
12	1.0×10^{-7}	7.6×10^{-8}

gold-plated Kovar packages was due to the fact that the clamping pressure of $7.0 \times 10^4 \text{ N/m}^2$ (10 psi) applied during cure was too great resulting in adhesive starvation of the bond lines. To investigate this possibility, twelve gold-plated Kovar packages were sealed using Epo-Tek H77 with a clamping pressure of only $3.5 \times 10^4 \text{ N/m}^2$ (5 psi) applied during cure. Initial seal test results for these packages are given in Table 13. As shown, all the packages except one which had a gross leak at the breather hole in the lid had low fine-leak rates. However, immersion in the 125°C fluorocarbon indicator fluid, as required in the C1 gross-leak test, caused sufficient pressure buildup in the packages to cause the seals to rupture after approximately 30 seconds, resulting in gross leakers. Apparently, seals formed with such a low clamping pressure used during cure have an even lower bond strength than those formed with the higher clamping pressure.

2.2.5 Results for Mechanical Shock Testing

Results for the packages that were mechanically shocked per MIL-STD-883A, Method 2002.1, Test Condition B (i.e., five 1500g shock pulses in the Y_1 plane), are given in Tables 14 and 15 for the gold-plated Kovar and ceramic packages, respectively. As noted in Table 14, attempts to seal gold-plated Kovar packages with Epo-Tek H77 were unsuccessful. Two attempts were made: one with the adhesive used to seal previous packages and one with fresh adhesive. Fine-leak testing indicated that all packages had leak rates in the low 10^{-8} atm cc/sec (air equivalent) range. However, during the C₁ gross leak test, all but four developed gross leaks after 15 to 60 seconds immersion in the 125°C test fluid. Apparently, the seals are good initially but the bond strength is so weak at 125°C that the internal pressure built up at this temperature is sufficient to rupture them.

Since the purpose of this test was to determine the effect of mechanical shock on the lid to package seal, the packages had to be held in such a way that the lids were unrestrained. This was accomplished by bonding the packages (bottom down) to 5/32 inch thick aluminum tabs using Ablefilm 529. These aluminum tabs were then clamped in a fixture so that the packages were free. This mounting method is essential for proper execution of the test; however, it does have an undesirable effect in that the adhesive used to mount the packages adsorbs or absorbs helium during bombing and subsequently releases

Table 13. Seal Test Results for Gold-Plated Kovar Packages
Sealed With Reduced ($3.5 \times 10^4 \text{ N/m}^2$ or 5 psi)
Clamping Pressure Applied During Cure

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)
Epo-Tek H77	
1	5.6×10^{-9} , Gross (C_1)
2	5.2×10^{-9} , Gross (C_1)
3	1.4×10^{-8} , Gross (C_1)
4	7.9×10^{-9} , Gross (C_1)
5	4.6×10^{-9} , Gross (C_1)
6	8.7×10^{-9} , Gross (C_1)
7	7.4×10^{-9} , Gross (C_1)
8	6.1×10^{-9} , Gross (C_1)
9	6.3×10^{-9} , Gross (C_1)
10	9.4×10^{-9} , Gross (C_1)
11	5.0×10^{-9} , Gross (C_1)
12	Gross Leak at Breather Hole

Table 14. Effect of Mechanical Shock on Seal Integrity of Gold-Plated Kovar Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Mounting Packages On Aluminum Tabs Air Equivalent (atm cc/sec)	Leak Rate After Mechanical Shock 5 Shocks at 1500 g's Air Equivalent (atm cc/sec)
Ablefilm 507			
13	1.4×10^{-7}	1.3×10^{-7}	1.8×10^{-7}
14	1.6×10^{-7}	1.4×10^{-7}	1.6×10^{-7}
15	1.8×10^{-7}	1.8×10^{-7}	2.0×10^{-7}
16	2.8×10^{-7}	1.8×10^{-7}	2.0×10^{-7}
17	2.0×10^{-7}	1.6×10^{-7}	2.0×10^{-7}
18	1.4×10^{-7}	1.5×10^{-7}	1.8×10^{-7}
Ablebond 36-2			
13	2.8×10^{-7}	2.1×10^{-7}	Gross (C ₂)
14	2.7×10^{-7}	2.5×10^{-7}	Gross (C ₂)
15	2.2×10^{-7}	2.4×10^{-7}	1.9×10^{-7}
16	2.0×10^{-7}	1.7×10^{-7}	1.4×10^{-7}
17	1.7×10^{-7}	1.9×10^{-7}	1.6×10^{-7}
18	2.2×10^{-7}	2.4×10^{-7}	2.0×10^{-7}
Epo-Tek H77	Attempts to seal packages with Epo-Tek H77 were unsuccessful (i.e., packages sealed with this adhesive failed gross C ₁ testing).		
Ablebond 789-1			
13	4.0×10^{-8}	2.5×10^{-7}	1.8×10^{-7}
14	1.6×10^{-8}	1.1×10^{-7}	1.4×10^{-7}
15	1.8×10^{-8}	1.3×10^{-7}	8.8×10^{-8}
16	1.3×10^{-8}	1.3×10^{-7}	9.4×10^{-8}
17	8.2×10^{-9}	8.9×10^{-8}	7.3×10^{-8}
18	5.2×10^{-9}	3.4×10^{-8}	3.3×10^{-8}
Seam Sealed			
13	1.1×10^{-9}	4.0×10^{-8}	7.6×10^{-8}
14	$<1.0 \times 10^{-9}$	2.6×10^{-8}	4.0×10^{-8}
15	1.3×10^{-9}	2.3×10^{-8}	3.6×10^{-8}
16	$<1.0 \times 10^{-9}$	3.0×10^{-8}	4.7×10^{-8}
17	$<1.0 \times 10^{-9}$	1.4×10^{-8}	2.4×10^{-8}
18	1.5×10^{-9}	3.4×10^{-8}	4.5×10^{-8}

Table 15. Effect of Mechanical Shock on Seal Integrity of Ceramic Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Mounting Packages On Aluminum Tabs Air Equivalent (atm cc/sec)	Leak Rate After Mechanical Shock 5 Shocks at 1500 g's Air Equivalent (atm cc/sec)
Ablefilm 507			
13	1.3×10^{-7}	1.6×10^{-7}	1.8×10^{-7}
14	1.8×10^{-7}	1.3×10^{-7}	1.6×10^{-7}
15	6.4×10^{-8}	1.2×10^{-7}	1.2×10^{-7}
16	1.2×10^{-7}	1.5×10^{-7}	1.3×10^{-7}
17	8.4×10^{-8}	1.4×10^{-7}	1.4×10^{-7}
18	1.7×10^{-7}	1.9×10^{-7}	1.9×10^{-7}
Ablebond 36-2			
13	1.5×10^{-7}	2.1×10^{-7}	2.9×10^{-7}
14	3.2×10^{-7}	2.9×10^{-7}	2.8×10^{-7}
15	1.1×10^{-7}	2.1×10^{-7}	2.0×10^{-7}
16	1.1×10^{-7}	2.2×10^{-7}	2.5×10^{-7}
17	2.3×10^{-7}	3.5×10^{-7}	2.9×10^{-7}
18	1.0×10^{-7}	3.0×10^{-7}	3.0×10^{-7}
Epo-Tek H77			
13	1.2×10^{-7}	1.9×10^{-7}	2.1×10^{-7}
14	1.3×10^{-7}	1.9×10^{-7}	2.0×10^{-7}
15	1.1×10^{-7}	1.4×10^{-7}	2.0×10^{-7}
16	6.0×10^{-8}	1.9×10^{-7}	2.0×10^{-7}
17	3.8×10^{-8}	1.4×10^{-7}	1.6×10^{-7}
18	2.3×10^{-8}	1.6×10^{-7}	1.6×10^{-7}
Ablebond 789-1			
13	5.4×10^{-8}	1.6×10^{-7}	1.9×10^{-7}
14	3.2×10^{-7}	2.0×10^{-7}	1.9×10^{-7}
15	1.3×10^{-7}	1.3×10^{-7}	1.5×10^{-7}
16	9.5×10^{-8}	1.5×10^{-7}	1.6×10^{-7}
17	1.0×10^{-7}	1.4×10^{-7}	1.5×10^{-7}
18	9.5×10^{-8}	1.6×10^{-7}	1.4×10^{-7}

it, increasing the apparent fine leak rate. To allow for this, the packages were fine-leak tested after they were mounted on the aluminum tabs. These results are given in Column 3 of Tables 14 and 15, and are the leak rates that should be compared to those obtained after the packages were mechanically shocked in order to determine the effects of this test environment on retention of seal integrity. The increase in apparent leak rate, due to the release of adsorbed or absorbed helium from the adhesive used to mount the packages (in this case four-mil thick Ablefilm 529) can be determined by comparing the results given in Columns 2 and 3 of Table 14 for the seam-sealed gold-plated Kovar packages. This comparison indicates that the helium released from the mounting adhesive during fine-leak testing is equivalent to a leak rate in the low $(1.5 \text{ to } 4)10^{-8}$ atm cc/sec (air equivalent) range. In general, this is negligible compared to the measured leak rates (low 10^{-7} atm cc/sec air equivalent range) of the adhesive-sealed packages and to the variation in the repeatability of fine-leak rate measurements.

Comparing the last two columns of Tables 14 and 15 shows that only two packages failed after mechanical shock, gold-plated Kovar Packages 13 and 14 sealed with Ablebond 36-2. However, it is not conclusive that these packages failed as a result of mechanical shock. These packages showed fine-leak rates after shock of 1.7×10^{-7} and 1.9×10^{-7} atm cc/sec (air equivalent), respectively (similar to the leak rates of the other four packages sealed with this adhesive), and passed the C_1 gross leak test but showed as C_2 gross leakers. A later C_1 gross leak test showed that Package 13 was now a C_1 gross leaker but that Package 14 was not. Previous experience with gold-plated Kovar packages suggests that the failure of these packages may have been due to the stress of the C_1 and C_2 gross-leak tests rather than as a result of exposure to the mechanical shock environment. In any case, the fact that the adhesive failed, indicates that it is inadequate for sealing gold-plated Kovar packages.

2.2.6 Results for Constant Acceleration Testing

Results for the packages that were subjected to constant acceleration per MIL-STD-883A, Method 2001.1, Test Condition A (i.e., 5,000g's in the Y_1 plane) are given in Tables 16 and 17. As in the case of the mechanical shock test,

Table 16. Effect of Constant Acceleration on Seal Integrity of Gold-Plated Kovar Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Mounting Packages On Aluminum Tabs Air Equivalent (atm cc/sec)	Leak Rate After Constant Accel. at 5000 g's Air Equivalent (atm cc/sec)
Ablefilm 507			
19	1.9×10^{-7}	9.4×10^{-8}	2.4×10^{-7}
20	2.0×10^{-7}	9.5×10^{-8}	2.4×10^{-7}
21	2.3×10^{-7}	1.0×10^{-7}	3.2×10^{-7}
22	2.0×10^{-7}	9.6×10^{-8}	2.3×10^{-7}
23	1.6×10^{-7}	7.8×10^{-8}	2.2×10^{-7}
24	1.9×10^{-7}	9.6×10^{-8}	4.0×10^{-7}
Ablebond 36-2			
19	1.2×10^{-7}	1.0×10^{-7}	Gross (C ₁)
20	9.0×10^{-8}	7.8×10^{-8}	*
21	1.2×10^{-7}	1.2×10^{-7}	*
22	8.1×10^{-8}	8.0×10^{-8}	*
23	1.2×10^{-7}	7.6×10^{-8}	Gross (C ₁)
24	9.8×10^{-8}	1.3×10^{-7}	3.2×10^{-7}
Epo-Tek H77	Attempts to seal packages with Epo-Tek H77 were unsuccessful (i.e., packages sealed with this adhesive failed gross C ₁ testing)		
Ablebond 789-1			
19	6.7×10^{-9}	1.6×10^{-7}	9.4×10^{-8}
20	4.8×10^{-9}	2.0×10^{-7}	1.8×10^{-7}
21	6.0×10^{-9}	5.8×10^{-8}	4.4×10^{-8}
22	5.6×10^{-9}	9.1×10^{-8}	7.2×10^{-8}
23	5.1×10^{-9}	6.8×10^{-8}	6.4×10^{-8}
24	3.6×10^{-9}	7.4×10^{-8}	9.5×10^{-8}
Seam Sealed			
19	$<1.0 \times 10^{-9}$	2.0×10^{-8}	7.0×10^{-8}
20	1.2×10^{-9}	2.8×10^{-8}	5.6×10^{-8}
21	$<1.0 \times 10^{-9}$	2.9×10^{-8}	6.2×10^{-8}
22	$<1.0 \times 10^{-9}$	5.8×10^{-8}	1.0×10^{-7}
23	1.1×10^{-9}	1.6×10^{-8}	4.2×10^{-8}
24	1.2×10^{-9}	5.1×10^{-8}	1.8×10^{-7}

*Lid Came Off During Constant Acceleration Test

Table 17. Effect of Constant Acceleration on Seal Integrity of Ceramic Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Mounting Packages on Aluminum Tabs Air Equivalent (atm cc/sec)	Leak Rate After Constant Accel. at 5000 g's Air Equivalent (atm cc/sec)
Ablefilm 507			
19	2.2×10^{-7}	4.0×10^{-7}	3.0×10^{-7}
20	1.2×10^{-7}	4.4×10^{-7}	3.6×10^{-7}
21	9.5×10^{-8}	3.3×10^{-7}	3.0×10^{-7}
22	7.5×10^{-8}	3.2×10^{-7}	2.8×10^{-7}
23	1.1×10^{-7}	3.2×10^{-7}	3.0×10^{-7}
24	8.8×10^{-8}	4.4×10^{-7}	4.7×10^{-7}
Ablebond 36-2			
19	1.2×10^{-7}	5.8×10^{-7}	3.7×10^{-7}
20	1.6×10^{-7}	6.0×10^{-7}	5.6×10^{-7}
21	1.9×10^{-7}	6.6×10^{-7}	5.0×10^{-7}
22	1.1×10^{-7}	4.4×10^{-7}	3.8×10^{-7}
23	9.8×10^{-8}	3.8×10^{-7}	4.4×10^{-7}
24	1.0×10^{-7}	5.8×10^{-7}	5.6×10^{-7}
Epo-Tek H77			
19	5.3×10^{-8}	1.3×10^{-6}	3.6×10^{-7}
20	1.9×10^{-7}	1.4×10^{-6}	4.0×10^{-7}
21	3.7×10^{-8}	5.6×10^{-7}	2.3×10^{-7}
22	6.2×10^{-8}	5.3×10^{-7}	2.2×10^{-7}
23	4.2×10^{-8}	6.0×10^{-7}	2.2×10^{-7}
24	1.7×10^{-8}	5.4×10^{-7}	2.5×10^{-7}
Ablebond 789-1			
19	1.2×10^{-7}	3.2×10^{-7}	2.0×10^{-7}
20	3.3×10^{-8}	3.2×10^{-7}	2.7×10^{-7}
21	2.8×10^{-8}	3.2×10^{-7}	2.6×10^{-7}
22	1.4×10^{-8}	3.0×10^{-7}	2.1×10^{-7}
23	1.6×10^{-8}	4.2×10^{-7}	3.4×10^{-7}
24	2.1×10^{-8}	3.0×10^{-7}	2.6×10^{-7}

the packages had to be held so that the lids were free, so again the packages were bonded bottom down on 5/32 inch thick aluminum tabs using four-mil thick Ablefilm 529.

Comparison of the initial leak rates and the leak rates after mounting for the seam-sealed gold-plated Kovar packages, indicates that the helium released from the mounting adhesive during fine-leak testing is equivalent to a leak rate in the low $(1.5 \text{ to } 5)10^{-8}$ atm cc/sec (air equivalent) range. This agrees with the corresponding range of $1.5 \text{ to } 4 \times 10^{-8}$ atm cc/sec (air equivalent) found for the packages that were mechanically shocked. As previously stated, this leak rate is negligible in comparison with the measured leak rates [low 10^{-7} atm cc/sec (air equivalent) range] of the adhesive-sealed packages and the variation in the repeatability of fine-leak rate measurements.

The only packages that failed the 5,000g's constant acceleration test were the gold-plated Kovar packages sealed with Ablebond 36-2. Five of the six packages failed. The lids came off three of the packages during test, and of the others subsequent leak testing showed one to be a fine leaker and one to be a C_1 gross leaker. Gold-plated Kovar packages sealed with Epo-Tek H77 were not tested because, as previously noted, attempts to seal these packages were unsuccessful.

2.2.7 Results for Temperature Aging Testing

Results for the packages exposed to a 125°C dry nitrogen environment for 240 hours are given in Tables 18 and 19. This test was selected to correspond to the temperature/time requirement associated with the burn-in test per MIL-STD-883A, Method 1015.1. All of the ceramic packages sealed with the four different adhesives, the gold-plated Kovar packages sealed with Ablebond 789-1, and the seam-sealed gold-plated Kovar packages retained their seal integrity after this exposure. However, the gold-plated Kovar packages sealed with Ablefilm 507 and Ablebond 36-2 did not. Epo-Tek H77 again was not tested on the gold-plated Kovar packages because attempts to seal these packages with this adhesive were unsuccessful. As noted in Table 18, this also was the case for three of the six gold-plated Kovar packages sealed with Ablebond 36-2.

Table 18. Effect of Temperature Aging on Seal Integrity of Gold-Plated Kovar Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Temperature Aging for 240 Hrs. at 125°C (atm cc/sec)
Ablefilm 507		
25	1.3×10^{-7}	Gross (C ₁)
26	1.4×10^{-7}	Gross (C ₁)
27	1.6×10^{-7}	2.0×10^{-7}
28	1.5×10^{-7}	Gross (C ₁)
29	1.8×10^{-7}	Gross (C ₁)
30	1.6×10^{-7}	Gross (C ₁)
Ablebond 36-2		
25	4.0×10^{-7}	Gross (C ₁)
26	1.2×10^{-7}	Gross (C ₁)
27	*	--
28	3.0×10^{-7}	Gross (C ₁)
29	*	--
30	*	--
Epo-Tek H77	Attempts to seal packages with Epo-Tek H77 were unsuccessful (i.e., packages sealed with this adhesive failed gross C ₂ testing)	
Ablebond 789-1		
25	1.7×10^{-8}	1.4×10^{-7}
26	1.7×10^{-8}	8.4×10^{-8}
27	1.0×10^{-8}	2.8×10^{-8}
28	7.8×10^{-9}	3.0×10^{-8}
29	1.2×10^{-8}	4.0×10^{-8}
30	9.6×10^{-9}	6.2×10^{-8}
Seam Sealed		
25	$<1.0 \times 10^{-9}$	1.0×10^{-9}
26	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
27	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
28	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
29	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$
30	$<1.0 \times 10^{-9}$	$<1.0 \times 10^{-9}$

*These packages failed gross C₂ testing

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Table 19. Effect of Temperature Aging on Seal Integrity of Ceramic Packages

Adhesive Package Number	Initial Leak Rate Air Equivalent (atm cc/sec)	Leak Rate After Temperature Aging for 240 Hrs. at 125°C (atm cc/sec)
Ablefilm 507		
25	2.5×10^{-7}	1.5×10^{-7}
26	1.8×10^{-7}	1.2×10^{-7}
27	2.5×10^{-7}	1.6×10^{-7}
28	7.6×10^{-7}	1.0×10^{-6}
29	1.9×10^{-7}	1.3×10^{-7}
30	1.9×10^{-7}	1.1×10^{-7}
Alebond 36-2		
25	9.8×10^{-8}	1.9×10^{-7}
26	8.8×10^{-8}	1.6×10^{-7}
27	9.1×10^{-8}	1.9×10^{-7}
28	7.6×10^{-8}	1.5×10^{-7}
29	3.1×10^{-7}	3.0×10^{-7}
30	3.5×10^{-7}	3.5×10^{-7}
Epo-Tek H77		
25	3.4×10^{-8}	1.6×10^{-8}
26	3.4×10^{-8}	1.9×10^{-8}
27	3.0×10^{-8}	1.5×10^{-8}
28	3.3×10^{-8}	1.5×10^{-8}
29	1.5×10^{-8}	9.4×10^{-9}
30	4.3×10^{-8}	1.1×10^{-8}
Alebond 789-1		
25	1.9×10^{-8}	1.8×10^{-8}
26	1.0×10^{-7}	2.3×10^{-8}
27	1.0×10^{-7}	3.0×10^{-8}
28	7.4×10^{-8}	2.2×10^{-8}
29	3.0×10^{-7}	2.2×10^{-7}
30	7.3×10^{-8}	2.4×10^{-8}

2.2.8 Results of Sequential Testing

The results obtained for the packages subjected sequentially to the specified test environments are given in Tables 20 and 21 for the gold-plated Kovar and ceramic packages, respectively. As shown in Table 20, none of the adhesive sealed gold-plated Kovar packages survived the sequential exposure. Only those sealed with Ablefilm 507 passed thermal shock (5 of 6 packages), but even these (4 of the remaining 5 packages), subsequently failed temperature cycling. Contrarily, a review of Table 21 indicates that all ceramic packages sealed with all four adhesives passed the sequential exposure. In reviewing the data, it is important to recall that the packages had to be mounted bottom down on aluminum tabs to perform the mechanical shock and constant acceleration tests. In this case, five-mil thick Ablefilm 529 was used rather than four-mil thick Ablefilm 529 as was used previously (Sections 2.2.5 and 2.2.6). This was somewhat unfortunate because comparison of the measured leak rates of the seam-sealed gold-plated Kovar packages before and after the packages were mounted on the aluminum tabs, indicates that with the thicker mounting adhesive, the adsorbed and absorbed helium released during fine-leak testing is equivalent to a leak rate of about 2 or 3×10^{-7} atm cc/sec (air equivalent). In contrast to the conclusion of the discussion of this subject in Sections 2.2.5 and 2.2.6, this value is not negligible in comparison with the measured leak rates of the adhesive sealed packages. As a result, the apparent leak rates for all of the packages are increased. However, comparison of the indicated leak rates after the packages were mounted on the aluminum tabs with those obtained after the packages were subsequently subjected to mechanical shock, constant acceleration, and temperature aging shows that the seal integrity of the packages was not degraded.

2.2.9 Summary of MIL-STD-883A Testing

A summary of the results obtained from this evaluation is given in Tables 22 and 23 for the individual and sequential exposures, respectively. The data are simplified by using an "X" to indicate the packages that retained their seal integrity and a dash (-) to indicate those that did not. The asterisks (*) indicate that packages were not tested since attempts to seal them were unsuccessful. The two important results evident from these tables are the following:

Table 20. Effect of Sequential Testing per MIL-STD-883A, Method 5004.2, Class A, Test Environments on Seal Integrity of Gold-Plated Kovar Packages

Adhesive Package No.	Initial Leak Rate Air Equiv. (atm cc/sec)	Leak Rate After Thermal Shock 15 Cycles (-65°C, +150°C) Air Equiv. (atm cc/sec)	Leak Rate After Temp. Cycling 15 Cycles (-65°C, +150°C) Air Equiv. (atm cc/sec)	Leak Rate After Mounting Packages On Aluminum Tabs Air Equivalent (atm cc/sec)	Leak Rate After Mechanical Shock 5 Shocks at 1500 g's Air Equivalent (atm cc/sec)	Leak Rate After Constant Accel. at 5,000 g's Air Equivalent (atm cc/sec)	Leak Rate After Temperature Aging 240 Hrs at 125°C Air Equivalent (atm cc/sec)
Ablefilm 507	1	9.8 x 10 ⁻⁸	1.7 x 10 ⁻⁷	---	---	---	---
	2	1.2 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	3	9.2 x 10 ⁻⁸	1.9 x 10 ⁻⁷	---	---	---	---
	4	9.6 x 10 ⁻⁸	8.5 x 10 ⁻⁸	---	---	---	---
	5	8.4 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	6	7.4 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
Ablebond 36-2	1	2.1 x 10 ⁻⁷	Gross (C ₁)	---	---	---	---
	2	1.5 x 10 ⁻⁷	Gross (C ₁)	---	---	---	---
	3	1.9 x 10 ⁻⁷	Gross (C ₁)	---	---	---	---
	4	1.5 x 10 ⁻⁷	Gross (C ₁)	---	---	---	---
	5	1.7 x 10 ⁻⁷	Gross (C ₁)	---	---	---	---
	6	1.2 x 10 ⁻⁷	Gross (C ₁)	---	---	---	---
Epo-Tek H77	1	5.0 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	2	6.8 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	3	2.2 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	4	3.6 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	5	5.6 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
	6	1.4 x 10 ⁻⁸	Gross (C ₁)	---	---	---	---
Ablebond 789-1	1	1.9 x 10 ⁻⁷	Gross (C ₂)	---	---	---	---
	2	1.8 x 10 ⁻⁷	Gross (C ₂)	---	---	---	---
	3	1.0 x 10 ⁻⁸	Gross (C ₂)	---	---	---	---
	4	8.0 x 10 ⁻⁸	Gross (C ₂)	---	---	---	---
	5	4.9 x 10 ⁻⁸	Gross (C ₂)	---	---	---	---
	6	4.2 x 10 ⁻⁸	Gross (C ₂)	---	---	---	---
Seam Sealed	1	<1.0 x 10 ⁻⁹	3.4 x 10 ⁻⁹	2.8 x 10 ⁻⁷	4.3 x 10 ⁻⁷	4.4 x 10 ⁻⁷	3.2 x 10 ⁻⁷
	2	<1.0 x 10 ⁻⁹	2.6 x 10 ⁻⁹	2.2 x 10 ⁻⁷	2.8 x 10 ⁻⁷	2.4 x 10 ⁻⁷	2.6 x 10 ⁻⁷
	3	<1.0 x 10 ⁻⁹	3.4 x 10 ⁻⁹	2.6 x 10 ⁻⁷	4.5 x 10 ⁻⁷	4.0 x 10 ⁻⁷	3.9 x 10 ⁻⁷
	4	<1.0 x 10 ⁻⁹	3.6 x 10 ⁻⁹	2.6 x 10 ⁻⁷	2.5 x 10 ⁻⁷	5.0 x 10 ⁻⁷	7.8 x 10 ⁻⁷
	5	<1.0 x 10 ⁻⁹	4.0 x 10 ⁻⁹	2.6 x 10 ⁻⁷	3.6 x 10 ⁻⁷	4.7 x 10 ⁻⁷	3.4 x 10 ⁻⁷
	6	<1.0 x 10 ⁻⁹	2.0 x 10 ⁻⁹	2.1 x 10 ⁻⁷	3.1 x 10 ⁻⁷	3.1 x 10 ⁻⁷	3.2 x 10 ⁻⁷

*Lid came off during fine leak test.

Table 21. Effect of Sequential Testing per MIL-STD-883A, Method 5004.2, Class A, Test Environments on Seal Integrity of Ceramic Packages

Adhesive Package No.	Initial Leak Rate Air Equiv. (atm cc/sec)	Leak Rate After Thermal Shock 15 Cycles (-65°C, +150°C) Air Equivalent (atm cc/sec)	Leak Rate After Temp. Cycling 15 Cycles (-65°C, +150°C) Air Equivalent (atm cc/sec)	Leak Rate After Mounting Packages On Aluminum Tabs Air Equivalent (atm cc/sec)	Leak Rate After Mechanical Shock 5 Shocks at 1500 g's Air Equivalent (atm cc/sec)	Leak Rate After Constant Accel. at 5000 g's Air Equivalent (atm cc/sec)	Leak Rate After Temp. Aging 240 Hrs at 125°C Air Equivalent (atm cc/sec)
Abletilla 507	1	1.3 x 10 ⁻⁷	1.5 x 10 ⁻⁷	1.2 x 10 ⁻⁶	2.2 x 10 ⁻⁶	1.6 x 10 ⁻⁶	1.2 x 10 ⁻⁶
	2	2.8 x 10 ⁻⁸	9.2 x 10 ⁻⁷	1.0 x 10 ⁻⁶	1.8 x 10 ⁻⁶	1.4 x 10 ⁻⁶	9.5 x 10 ⁻⁶
	3	2.4 x 10 ⁻⁸	1.4 x 10 ⁻⁷	1.2 x 10 ⁻⁶	1.8 x 10 ⁻⁶	1.4 x 10 ⁻⁶	1.1 x 10 ⁻⁶
	4	4.0 x 10 ⁻⁸	1.6 x 10 ⁻⁷	1.2 x 10 ⁻⁶	1.6 x 10 ⁻⁶	1.4 x 10 ⁻⁶	1.3 x 10 ⁻⁶
	5	1.6 x 10 ⁻⁷	1.0 x 10 ⁻⁷	1.6 x 10 ⁻⁶	2.0 x 10 ⁻⁶	1.6 x 10 ⁻⁶	1.0 x 10 ⁻⁶
	6	2.0 x 10 ⁻⁷	1.6 x 10 ⁻⁷	1.3 x 10 ⁻⁶	1.5 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.0 x 10 ⁻⁶
Ablebond 36-2	1	1.0 x 10 ⁻⁷	2.2 x 10 ⁻⁷	1.0 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.1 x 10 ⁻⁶	1.4 x 10 ⁻⁶
	2	1.2 x 10 ⁻⁷	1.6 x 10 ⁻⁷	1.0 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.2 x 10 ⁻⁶
	3	1.0 x 10 ⁻⁸	1.4 x 10 ⁻⁷	1.0 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.3 x 10 ⁻⁶
	4	7.6 x 10 ⁻⁸	1.8 x 10 ⁻⁷	1.0 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.6 x 10 ⁻⁶
	5	5.8 x 10 ⁻⁸	6.5 x 10 ⁻⁸	1.0 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.1 x 10 ⁻⁶	1.4 x 10 ⁻⁶
	6	8.0 x 10 ⁻⁸	1.0 x 10 ⁻⁷	1.0 x 10 ⁻⁶	1.1 x 10 ⁻⁶	1.2 x 10 ⁻⁶	1.3 x 10 ⁻⁶
Epo-Tek H77	1	5.0 x 10 ⁻⁸	2.2 x 10 ⁻⁸	6.9 x 10 ⁻⁷	9.2 x 10 ⁻⁷	7.1 x 10 ⁻⁷	7.8 x 10 ⁻⁷
	2	5.0 x 10 ⁻⁷	3.4 x 10 ⁻⁸	5.1 x 10 ⁻⁷	5.9 x 10 ⁻⁶	4.8 x 10 ⁻⁷	5.2 x 10 ⁻⁷
	3	1.3 x 10 ⁻⁷	6.8 x 10 ⁻⁸	9.1 x 10 ⁻⁷	1.0 x 10 ⁻⁶	8.2 x 10 ⁻⁷	8.2 x 10 ⁻⁷
	4	3.4 x 10 ⁻⁸	2.6 x 10 ⁻⁸	5.3 x 10 ⁻⁷	5.7 x 10 ⁻⁷	4.6 x 10 ⁻⁷	5.6 x 10 ⁻⁷
	5	6.8 x 10 ⁻⁸	6.0 x 10 ⁻⁸	3.9 x 10 ⁻⁷	5.3 x 10 ⁻⁷	4.5 x 10 ⁻⁷	5.0 x 10 ⁻⁷
	6	9.2 x 10 ⁻⁸	9.0 x 10 ⁻⁸	1.0 x 10 ⁻⁷	4.8 x 10 ⁻⁷	3.9 x 10 ⁻⁷	4.0 x 10 ⁻⁷
Ablebond 789-1	1	1.8 x 10 ⁻⁷	1.7 x 10 ⁻⁷	3.4 x 10 ⁻⁷	4.8 x 10 ⁻⁷	5.6 x 10 ⁻⁷	7.3 x 10 ⁻⁷
	2	1.7 x 10 ⁻⁷	1.6 x 10 ⁻⁷	4.0 x 10 ⁻⁷	4.9 x 10 ⁻⁷	5.0 x 10 ⁻⁷	6.3 x 10 ⁻⁷
	3	6.5 x 10 ⁻⁸	1.5 x 10 ⁻⁷	3.9 x 10 ⁻⁷	5.4 x 10 ⁻⁷	5.8 x 10 ⁻⁷	6.6 x 10 ⁻⁷
	4	1.5 x 10 ⁻⁷	1.1 x 10 ⁻⁷	3.7 x 10 ⁻⁷	4.6 x 10 ⁻⁷	4.8 x 10 ⁻⁷	7.0 x 10 ⁻⁷
	5	1.2 x 10 ⁻⁷	7.2 x 10 ⁻⁸	4.4 x 10 ⁻⁷	5.5 x 10 ⁻⁷	9.1 x 10 ⁻⁷	6.0 x 10 ⁻⁷
	6	7.4 x 10 ⁻⁸	1.7 x 10 ⁻⁷	4.5 x 10 ⁻⁷	5.0 x 10 ⁻⁷	6.2 x 10 ⁻⁷	6.2 x 10 ⁻⁷

Table 22. Simplified Summary of Results of Individual Testing Per MIL-STD-883A, Method 5004.2, Class A Test Environments

Package Type Adhesive	Thermal Shock	Temperature Cycling	Mechanical Shock	Constant Acceleration	Temperature Aging
<u>Control</u>					
Seam-Sealed Gold-Plated Kovar	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
<u>Gold-Plated Kovar</u>					
Ablefilm 507	XXXX-	XXXXXX	XXXXXX	XXXXXX	X-----
Ablebond 36-2	-----	-----	XXXX--	X-----	---(*)
Epo-Tek H77	-----	-----	(*)	(*)	(*)
Ablebond 789-1	-----	-----	XXXXXX	XXXXXX	XXXXXX
<u>Ceramic</u>					
Ablefilm 507	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
Ablebond 36-2	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
Epo-Tek H77	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
Ablebond 789-1	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX

X = Package retained seal integrity
 - = Package was gross leaker
 (*) = Packages were not tested because attempts to seal them were unsuccessful

Table 2C. Simplified Summary of Results of Sequential Testing Per MIL-STD-883A, Method 5004.2, Class A Test Environments

Package Type Adhesive	Thermal Shock	Temperature Cycling	Mechanical Shock	Constant Acceleration	Temperature Cycling
<u>Control</u>					
Seam-Sealed Gold-Plated Kovar	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
<u>Gold-Plated Kovar</u>					
Ablefilm 507	XXXXX-	X-----			
Ablebond 36-2	-----				
Epo-Tek H77	-----				
Ablebond 789-1	-----				
<u>Ceramic</u>					
Ablefilm 507	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
Ablebond 36-2	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
Epo-Tek H77	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX
Ablebond 789-1	XXXXXX	XXXXXX	XXXXXX	XXXXXX	XXXXXX

X = Package retained seal integrity

- = Package was gross leaker

- (1) All ceramic packages sealed with all four adhesives retained their seal integrity after exposure to all test environments, both individually and sequentially.
- (2) None of the adhesive sealed gold-plated Kovar packages survived sequential exposure to the test environments. Only those sealed with Ablefilm 507 survived thermal shock but subsequently failed temperature cycling.

On the basis of these results, it is recommended that no further consideration be given to adhesive sealing gold-plated Kovar packages.

2.2.10 Selection of Best Adhesive - Package Combination for the Moisture Permeation Study

From the summary of the results of this effort given in the last section (2.2.9), it is obvious that the ceramic package is the proper package choice for the moisture permeation study. However, since all four of the adhesives performed equally well with the ceramic packages, there is no reason to select one over the others. Also, since the previous evaluation of the effect of temperature-humidity exposures on the seal integrity of adhesive sealed packages discussed in Section 2.1 was performed using gold-plated Kovar packages, it is felt that the results might not be completely valid for ceramic packages, and should not be used as the basis for further selection among the four candidate adhesives. As a result, ceramic packages sealed with these adhesives were subjected to the two most severe temperature-humidity exposures previously used (ten days at 60°C/98% RH and ten days at 85°C/85% RH) and seal tested.

Results for the packages subjected to ten days at 60°C/98% RH are given in Table 24. All packages passed (i.e., retained their seal integrity). The packages used for this test had previously been temperature cycled for 15 cycles between -65°C and +150°C.

Results for the packages subjected to ten days at 85°C/85% RH are given in Tables 25 and 26. The packages in Table 25 had previously been temperature cycled for 15 cycles between -65°C and +150°C. All of these packages passed the fine and C_1 gross leak tests. However, all three of the packages sealed with Ablefilm 507 and one each sealed with Ablebond 36-2 and Epo-Tek H77

Table 24. Effect of Ten Days Exposure at 60°C/98% RH on Adhesive Sealed Ceramic Packages

Adhesive Package No.	Leak Rate Before Exposure Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507		
10	1.2×10^{-7}	8.3×10^{-8}
11	2.5×10^{-7}	1.9×10^{-7}
12	2.0×10^{-7}	1.1×10^{-7}
Ablebond 36-2		
10	2.4×10^{-7}	3.2×10^{-7}
11	1.1×10^{-7}	1.9×10^{-7}
12	2.0×10^{-7}	2.4×10^{-7}
Epo-Tek H77		
10	8.7×10^{-8}	2.3×10^{-7}
11	4.9×10^{-8}	1.2×10^{-7}
12	5.6×10^{-8}	1.1×10^{-7}
Ablebond 789-1		
10	9.3×10^{-8}	3.3×10^{-7}
11	9.6×10^{-8}	3.2×10^{-7}
12	7.6×10^{-8}	2.6×10^{-7}

Table 25. Effect of Ten Days Exposure at 85°C/85% RH on Adhesive Sealed Ceramic Packages

Adhesive Package Number	Leak Rate Before Exposure Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507		
7	2.0×10^{-7}	1.2×10^{-7} Gross (C ₂)*
8	2.8×10^{-7}	1.9×10^{-7} Gross (C ₂)
9	1.3×10^{-7}	1.0×10^{-7} Gross (C ₂)
Ablebond 36-2		
7	2.3×10^{-7}	1.0×10^{-7}
8	2.3×10^{-7}	1.0×10^{-7}
9	3.0×10^{-7}	1.4×10^{-7} Gross (C ₂)*
Epo-Tek H77		
7	1.8×10^{-7}	5.5×10^{-8}
8	8.4×10^{-8}	4.2×10^{-8}
9	1.0×10^{-7}	1.3×10^{-7} Gross (C ₂)
Ablebond 789-1		
7	1.5×10^{-7}	8.6×10^{-8}
8	7.7×10^{-8}	3.9×10^{-8}
9	1.0×10^{-7}	5.1×10^{-8}

* Leak Occurred at Vent Hole

Table 26. Effect of Ten Days Exposure at 85°C/85% RH on Adhesive-Sealed Ceramic Packages

Adhesive Package Number	Leak Rate Before Exposure Air Equivalent (atm cc/sec)	Leak Rate After Exposure Air Equivalent (atm cc/sec)
Ablefilm 507		
25	1.5×10^{-7}	Gross (C ₁)
26	1.2×10^{-7}	Gross (C ₁)
27	1.6×10^{-7}	Gross (C ₁)
28	1.0×10^{-6}	Gross (C ₁)
29	1.3×10^{-7}	Gross (C ₁)
30	1.1×10^{-7}	1.4×10^{-7} , Gross (C ₂)
Ablebond 36-2		
25	1.9×10^{-7}	2.2×10^{-7} , Gross (C ₂)
26	1.6×10^{-7}	Gross (C ₁)
27	1.9×10^{-7}	2.2×10^{-7}
28	1.5×10^{-7}	Gross (C ₁)
29	3.0×10^{-7}	3.2×10^{-7} , Gross (C ₂)
30	3.5×10^{-7}	3.6×10^{-7}
Epo-Tek H77		
25	1.6×10^{-8}	Gross (C ₁)
26	1.9×10^{-8}	6.8×10^{-8} , Gross (C ₂)
27	1.5×10^{-8}	7.1×10^{-8}
28	1.5×10^{-8}	6.3×10^{-8}
29	9.4×10^{-9}	3.8×10^{-8} , Gross (C ₂)
30	1.1×10^{-8}	4.0×10^{-8} , Gross (C ₂)
Ablebond 789-1		
25	1.8×10^{-8}	Gross (C ₁)
26	2.3×10^{-8}	1.6×10^{-7}
27	3.0×10^{-8}	Gross (C ₁)
28	2.2×10^{-8}	1.3×10^{-7}
29	2.2×10^{-7}	Gross (C ₁)
30	2.4×10^{-8}	1.2×10^{-7} , Gross (C ₂)

failed the C_2 gross leak test. For two of the packages (one sealed with Ablebond 36-2 and one sealed with Ablefilm 507), the gross leak occurred at the vent hole which also was sealed with adhesive. For the other three packages, the gross leak occurred in the seal area. The packages in Table 26 had previously been temperature aged at 125°C for 240 hours. Thirteen out of 24 packages passed the fine and C_1 gross leak tests (one sealed with Ablefilm 507, four sealed with Ablebond 36-2, five sealed with Epo-Tek H77, and three sealed with Ablebond 789-1). However, seven of these subsequently failed the C_2 gross-leak test. Only six packages retained their seal integrity (two each sealed with Ablebond 36-2, Epo-Tek H77, and Ablebond 789-1). All six packages sealed with Ablefilm 507 failed.

The results shown in Tables 25 and 26 indicate that of the four adhesives evaluated, Ablefilm 507 was degraded the most when exposed to the 85°C/85% RH environment. Also, it appears that Ablebond 789-1 was degraded the least and the other two, Ablebond 36-2 and Epo-Tek H77, about equally and somewhere in between. Based on these results, Ablebond 789-1 was selected as the adhesive to be used in the moisture permeation study.

2.3 SUSCEPTIBILITY OF ADHESIVE-SEALED PACKAGES TO MOISTURE PERMEATION

The objective of this effort was to subject the best adhesive-package combination identified in the previous effort to a 60°C/98% RH environment and determine its susceptibility to moisture permeation. To accomplish this, three ceramic packages were sealed with Ablebond 789-1 with Panametrics Aquamax-type moisture sensors inside them. These packages were then exposed to a 60°C/98% RH environment and their moisture content monitored. A seam-sealed gold-plated Kovar package also containing an Aquamax-type moisture sensor was used as a control.

2.3.1 The Panametrics Aquamax-Type Moisture Sensor

A photograph of a Panametrics Aquamax-type moisture sensor is shown in Figure 10. The overall dimensions of the sensor chip are 0.368 cm (145 mils) square and the active region of the sensor is 0.19 by 0.23 cm (75 by 90 mils). The physical structure and a representative cross-section of the moisture sensor are shown in Figure 11, along with the equivalent circuit of a single pore. Essentially, the sensor is an aluminum-oxide capacitor. The sensor is made by evaporating aluminum on silicon, anodizing it, and then evaporating a thin layer of gold over the porous oxide layer formed. Gold bonding pads are attached to the aluminum and gold layers which form the two electrodes of the capacitor.

According to the vendor's brochure, the gold electrode is so thin that water vapor rapidly permeates it and is adsorbed by the porous oxide. The amount of water adsorbed is functionally related to the vapor pressure of the water present in the atmosphere surrounding the sensor. The conductivity of the oxide structure is determined by the number of water molecules adsorbed and provides a distinct value of electrical impedance which is a direct measure of the water vapor pressure. The sensor can be calibrated to measure dew points ranging from +20°C to -110°C (i.e., moisture content ranging from approximately 23,000 ppm_v to 1 ppb_v).

2.3.2 Sensor Mounting for Present Application

The moisture sensors normally are supplied mounted on TO headers, a form unusable in the present application where they are to be directly mounted in gold-plated Kovar and ceramic packages. At special request, Panametrics supplied the sensors eutectically mounted on 0.635 cm (1/4 inch) square gold-plated

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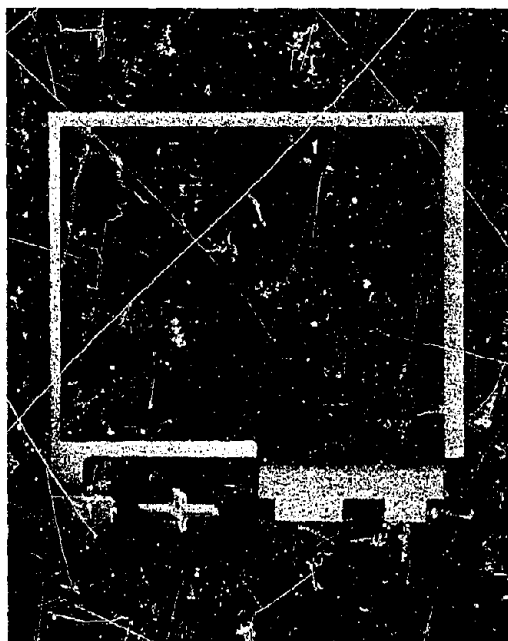
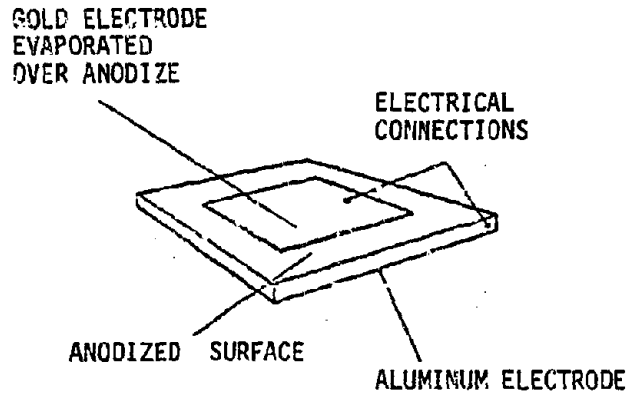
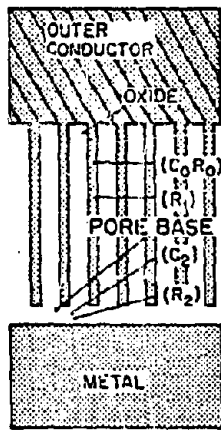


Figure 10. Photograph of a Panametrics
Aquamax-Type Moisture Sensor

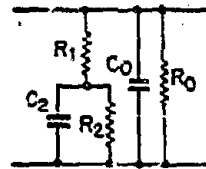
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(a) Representative Physical Structure



(b) Representative Cross-section



C_0 = CAPACITANCE OF ENTIRE OXIDE LAYER;
 C_2 = PORE-BASE CAPACITANCE;
 R_0 = RESISTANCE OF SOLID ALUMINUM OXIDE;
 R_1 = PORE-SIDE RESISTANCE;
 R_2 = PORE-BASE RESISTANCE;

(c) Equivalent Circuit of a Single Pore

Figure 11. Physical Structure, Representative Cross-section, and Single Pore Equivalent Circuit of a Panametrics Aquamax-Type Moisture Sensor

Kovar tabs as shown in Figure 12. Also, since the bonding pads on the sensors are small and bonds must be made and broken several times during the course of this investigation, special carrier substrates on which the sensors are permanently mounted were designed and fabricated. A photograph of one of these substrates is shown in Figure 13. These substrates are ceramic with three large thin-film gold metallized areas approximately $2 \mu\text{m}$ (20,000Å) thick, one U-shaped for attachment of the gold-plated Kovar tabs containing the moisture sensors, and two L-shaped to serve as device terminals. The gold-plated Kovar tabs are attached to the U-shaped thin film gold metallized regions by micro-gap bonding 0.0025 cm (1 mil) thick gold ribbon between them. Electrical connection between the thin film gold pads on the moisture sensors and those on the substrates are made by ultrasonically bonded 0.0025 cm (1 mil) diameter gold wire. A photograph of a carrier substrate with a moisture sensor attached is shown in Figure 14. These substrates with the moisture sensors attached are then the units that are removed and rebonded. Due to their large terminal areas, they can be used repeatedly. It turned out to be extremely fortunate that carrier substrates were used because difficulty was encountered in bonding to the smaller pads on the moisture sensors (the ones on the left in Figure 10) due to poor pad adhesion. For some sensors, several attempts had to be made before a bond was obtained; and in all cases, the final bond had marginal strength (around one gram).

2.3.3 Ceramic Package Bases

Bases for the ceramic packages were designed and fabricated. A photograph of one is shown in Figure 15. These bases are approximately 2.31 cms (0.910 inches) wide and 2.95 cms (1.160 inches) long with three thin-film gold metallized areas approximately $2 \mu\text{m}$ (20,000Å) thick, one U-shaped to accommodate the carrier substrates with the moisture sensors, and two L-shaped to serve as package terminals. The carrier substrates with the moisture sensors attached are placed in the U-shaped regions and attached to them by micro-gap bonding 0.0025 cm (1 mil) thick gold ribbon. Electrical connection between the terminals of the carrier substrates and those of the package bases are made by ultrasonically bonded 0.0038 cm (1.5 mils) diameter gold wire. A photograph of a package base with a carrier substrate (and moisture sensor) attached is shown in Figure 16.

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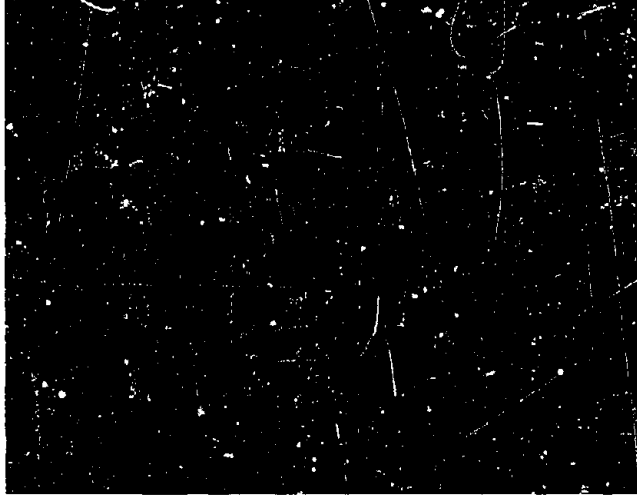


Figure 12. Moisture Sensor Mounted on Gold-Plated Kovar Tab as Supplied by Panametrics

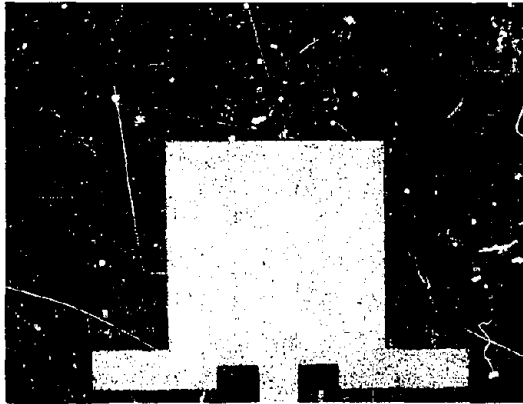


Figure 13. Carrier Substrate for Panametrics Moisture Sensor

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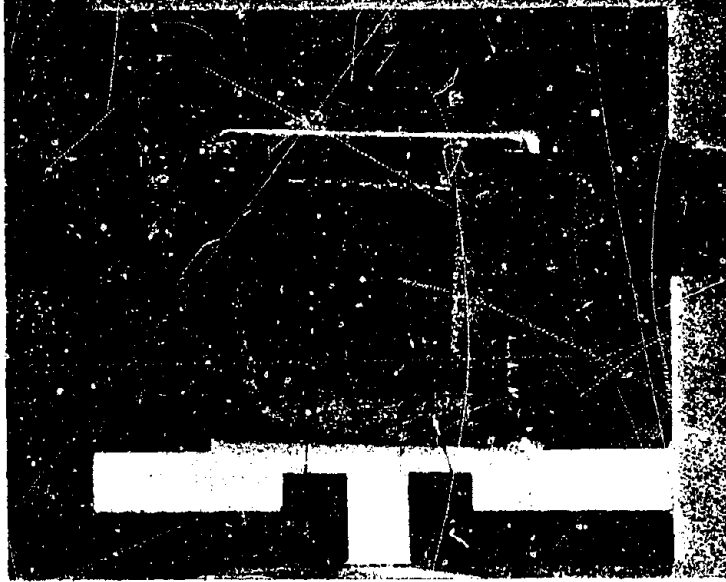


Figure 14. Carrier Substrate with Moisture Sensor Attached

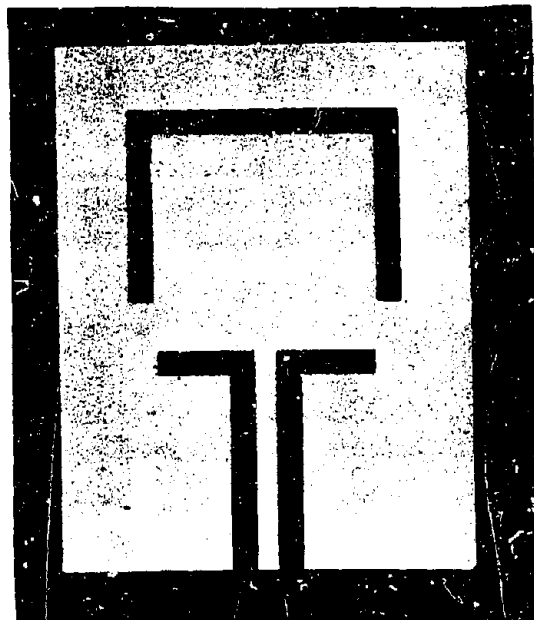


Figure 15. Base for Ceramic Package

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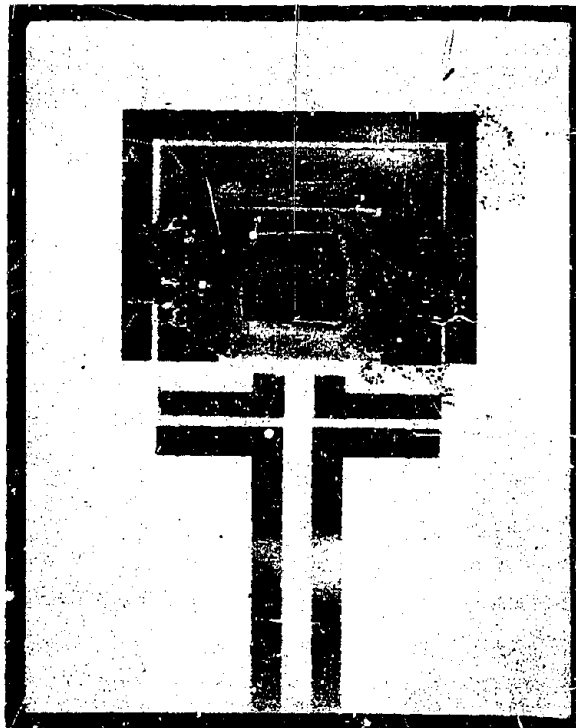


Figure 16. Ceramic Package Base With
Carrier Substrate (and
Moisture Sensor) Attached

2.3.4 Necessity for In-House Calibration of the Moisture Sensors

Conversations with Panametrics personnel revealed that the present proposed use of the Aquamax-type moisture sensors differed from normal application in two respects. First, the sensors normally are used at room temperature ($25 \pm 2^\circ\text{C}$), and second, the maximum recommended storage temperature is $+70^\circ\text{C}$. In the present application where the sensors are to be sealed in adhesive-sealed packages, the sensors must be exposed to the cure temperature of the adhesive which is 170°C for Ablebond 789-1. Also, in the present application, it is desirable to monitor the moisture content of the packages at the exposure temperature of 60°C rather than at room temperature.

Further conversations with Panametrics personnel indicated that operating the sensors at 60°C would not present a serious problem, but would only require that the sensors be calibrated at that temperature. Also, it was felt that while exposing the sensors to 170°C (as required to cure the adhesive) would permanently change their calibrations, they would stabilize at new values after a few hours (4 to 6) exposure. However, since no data exist confirming this speculation, it was recommended that the sensors still be recalibrated after testing is completed. As a result of these conversations, it was decided to proceed as follows:

- (a) Calibrate the sensors as received at room temperature and at 60°C .
- (b) Expose the sensors at 170°C for six hours in dry nitrogen and recalibrate them at room temperature and at 60°C .
- (c) Seal the sensors in the packages, subject the packages to the $60^\circ\text{C}/98\% \text{ RH}$ environment, and monitor the sensor outputs versus time.
- (d) Remove the sensors from the packages, recalibrate them at 60°C to see if they have changed, and interpret the output readings obtained in (c).

2.3.5 Chamber for Calibrating Moisture Sensors

Since the above procedure requires repeated calibration, the only practical approach was to develop an in-house capability. Consequently, a chamber for use in calibrating the moisture sensors was designed and fabricated. An exterior view of the chamber is shown in Figure 17 and a view of the interior of the two parts is shown in Figure 18. The chamber was fabricated from stainless steel, and the plate was electroplated with gold so that the ceramic carrier substrates on which the moisture sensors are mounted could be strapped to it with gold

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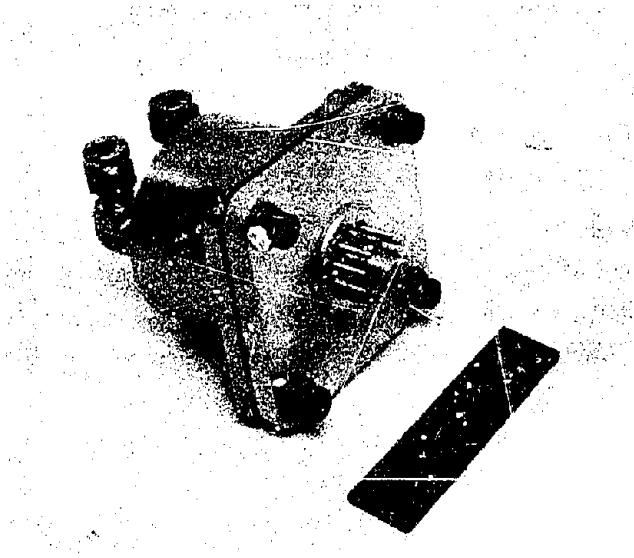


Figure 17. Chamber for Use in Calibrating Moisture Sensors



Figure 18. Interior of Calibration Chamber

ribbon. This attachment can be seen in Figure 18 and in greater detail in Figure 14. As can be seen in Figures 17 and 18, the chamber is equipped with two ports with swage-type fittings for gas flow-through and an eleven-pin electrical feedthrough for attaching five moisture sensors and a thermocouple (iron-constantan). The capability of calibrating more than one moisture sensor at a time is desirable not only to reduce the time required, but also to determine the uniformity of response between the moisture sensors. Electrical connection between the moisture sensors and the feedthrough pins was made by ultrasonically bonded 0.0038 cm (1-1/2 mil) diameter gold wire.

A stainless steel disc was mounted in the cylindrical part of the chamber (visible in Figure 18) to provide a baffle so that the gas would not impinge directly on the moisture sensors. This disc is of proper size to provide a 0.32 cm (1/8 inch) gap between it and the chamber wall and was mounted 1.27 cms (1/2 inch) from the gas ports on a 0.64 cm (1/4 inch) wide stainless steel bar. The bar was cut to slip-fit into the chamber and was positioned between the two ports to assure that the gas would not be short-circuited between the inlet and outlet ports. The chamber was sealed using an O-ring as shown in Figure 18.

2.3.6 Calibration of As-Received Sensors

Calibration runs were made for five Panametrics moisture sensors at room temperature and at 60°C. Readings were taken with the Panametrics Model 2000 Control Unit operated in both the normal and expanded scale modes. The five sensors were calibrated simultaneously using the calibration chamber just described. Calibration was made at five points covering a dew point range from approximately -36°C to +8°C, corresponding to moisture concentrations ranging from 200 to 10,000 ppm_v. The setup depicted in Figure 19 was used to generate the nitrogen of various moisture contents required for the calibration. The precise value of the dew point at each calibration point was measured with an optical dew point hygrometer.

Calibration curves obtained for one of the sensors (Sensor No. 1) are given in Figure 20. These curves are typical of those obtained for all five sensors. The calibration curves are extremely temperature dependent. The calibration curve obtained at 60°C differed considerably from that obtained at room temperature (27°C). Also, the calibration curves cross at a dew point of about

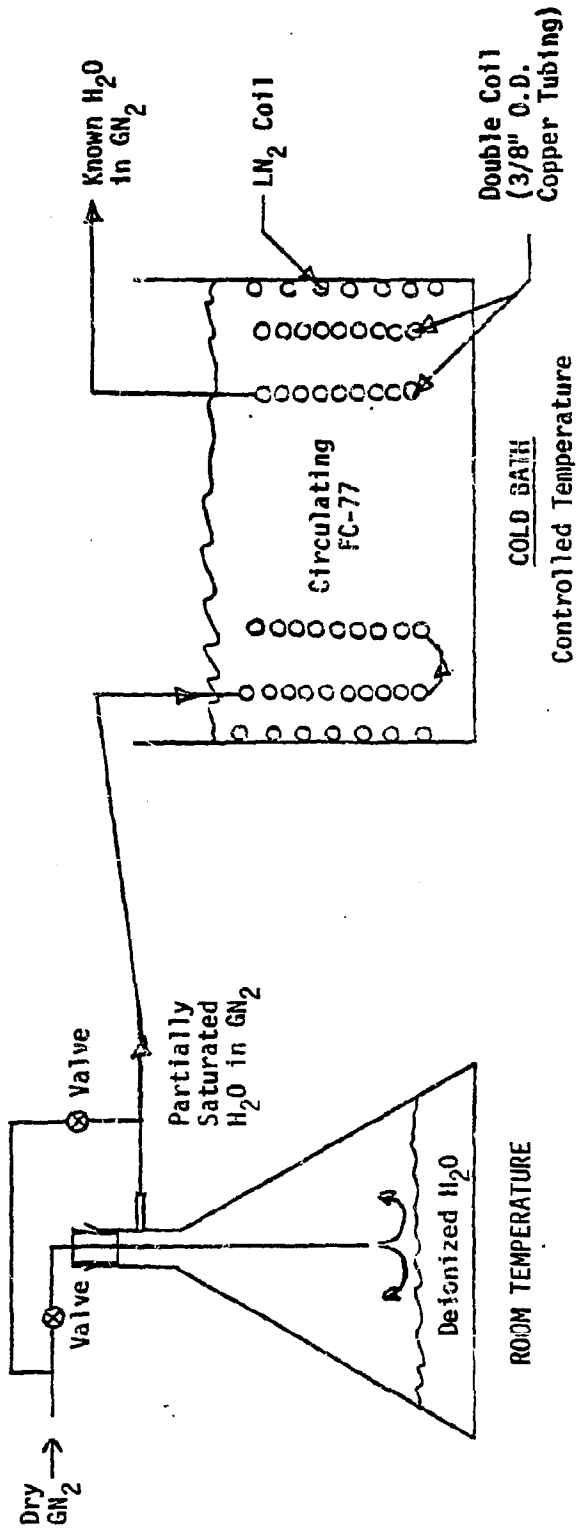


Figure 19. Setup for Producing Nitrogen of Known Moisture Content

EUCHE DIETZEN CO.
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NO. 340R-L220 DIETZEN GRAPH PAPER
SEMI-LOGARITHMIC
2 CYCLES X 20 DIVISIONS PER INCH

PARAMETER MODE 2000 METER READING

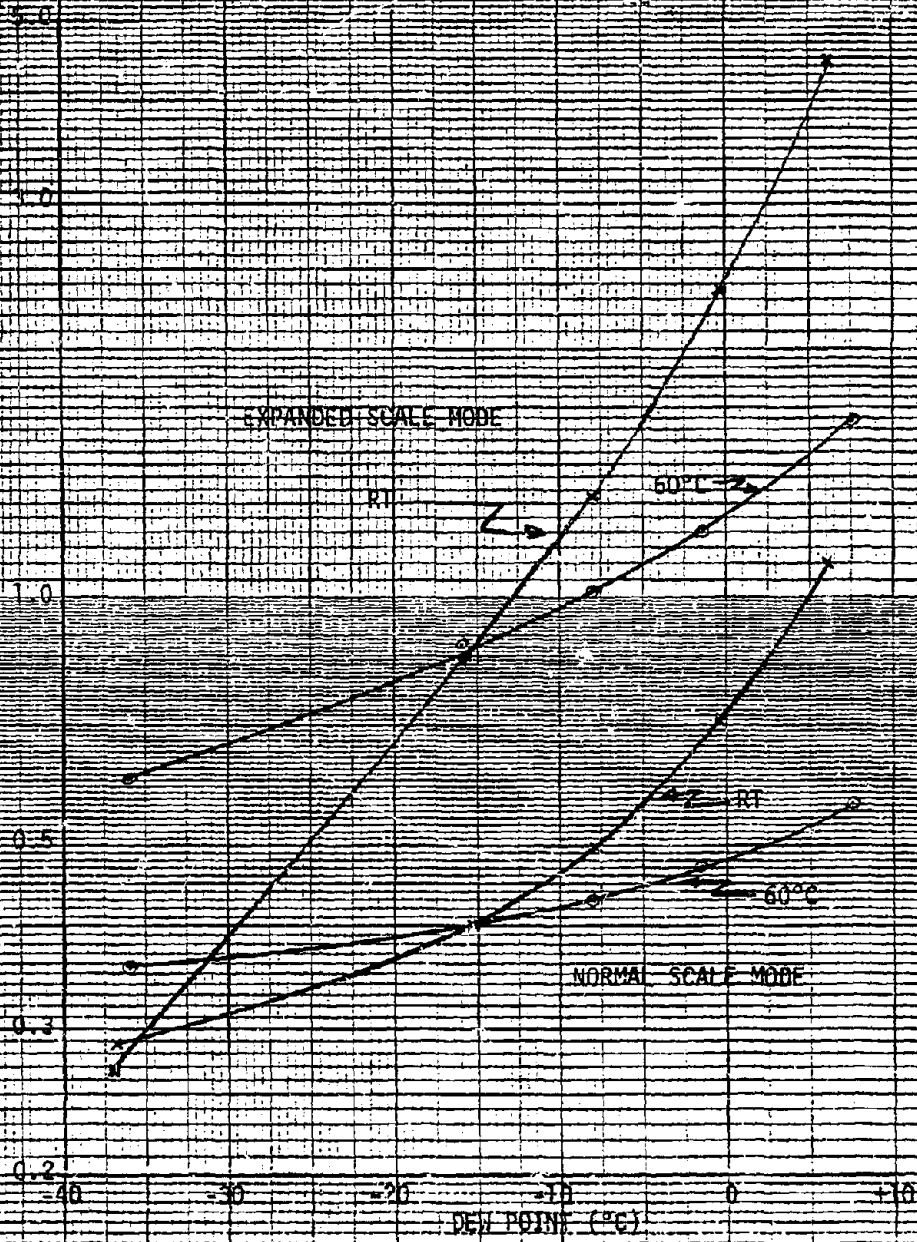


Figure 20. Calibration Curves for Sensor No. 1
at Room Temperature and at 60°C

-15°C. These results point out a serious limitation in the use of these sensors. For accurate results, they must be used at very nearly the same temperature at which they are calibrated. Also, their sensitivity is degraded at 60°C. The calibration curves for all five sensors (with the Control Unit operated in the expanded scale mode) are plotted in Figure 21. These curves are shown to emphasize the fact that if accurate dew point measurements are desired, the sensors must be calibrated over the entire dew point range, especially if they are to be used at the higher temperatures. Single point calibration would not be sufficient since the curves for the different sensors are not parallel. The relation of the curves of Sensor No. 5 to the others merits comment. The curves for this sensor fell quite a bit lower than the others, indicating that it had generally lower sensitivity. The surface of this sensor had been scratched in handling, which could explain the observed reduced sensitivity since such damage probably reduces the active area or volume of the sensor.

Additional calibration runs were made on the same sensors to check their repeatability. Typical results are shown in Figure 22 (Sensor No. 2). In general, the results are reasonably good for the sensors operated in the expanded scale mode (difference in dew points range from 1 to 4°C). In the worst case (4°C), at a dew point of -10°C, this would represent a possible value of moisture content ranging from 2150 to 3050 ppm_v; or at a dew point of 0°C, a possible value ranging from 5100 to 6950 ppm_v. This represents an error in ppm_v of approximately 15 to 17%.

2.3.7 Recalibration of Sensors After Six Hours at 170°C

Results of the recalibration of the sensors after they were exposed at 170°C for six hours in dry nitrogen are summarized in Figures 23 through 26. The calibration curves for both the normal and the expanded scale modes obtained for Sensor No. 1 at room temperature and at 60°C are given in Figures 23 and 24, respectively. Corresponding calibration curves obtained previously (before the six-hour bake at 170°C) are also shown for comparison. These curves are typical of those obtained for all five sensors. It is evident from this comparison that the calibration curves have changed considerably because of the high temperature exposure. The sensitivity of the sensors is substantially reduced; however, they certainly are still usable.

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NO. 340R-1220 DIETZGEN GRAPH PAPER
SEMI-LOGARITHMIC
2 CYCLES X 20 DIVISIONS PER INCH

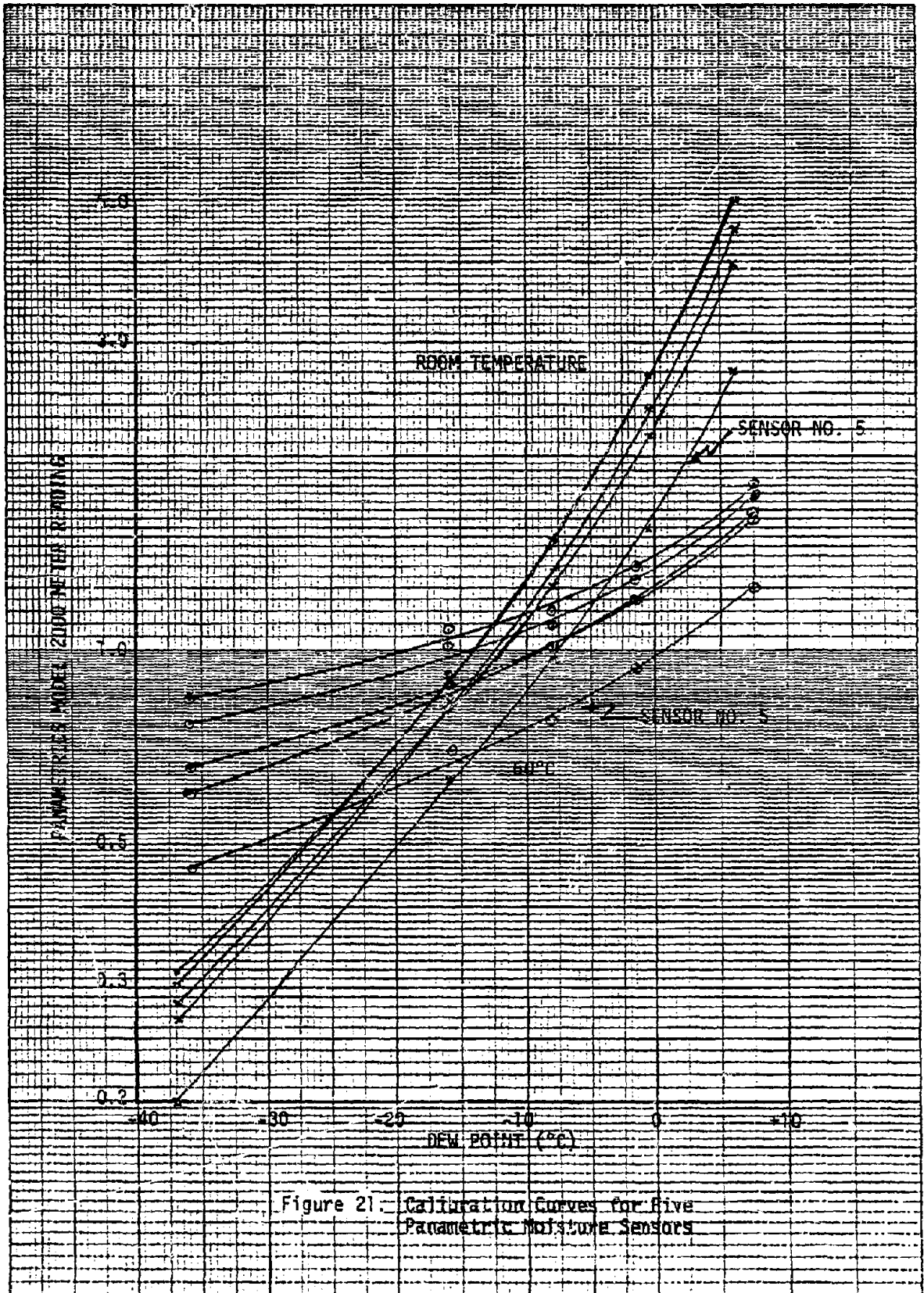


Figure 21. Calibration Curves for Five Parametric Moisture Sensors

EUGENE DIETZEN CO.
MADE IN U. S. A.

MD. 34DR-L220 DIETZEN GRAPH PAPER
SEMI-LOGARITHMIC
2 CYCLES X 20 DIVISIONS PER INCH

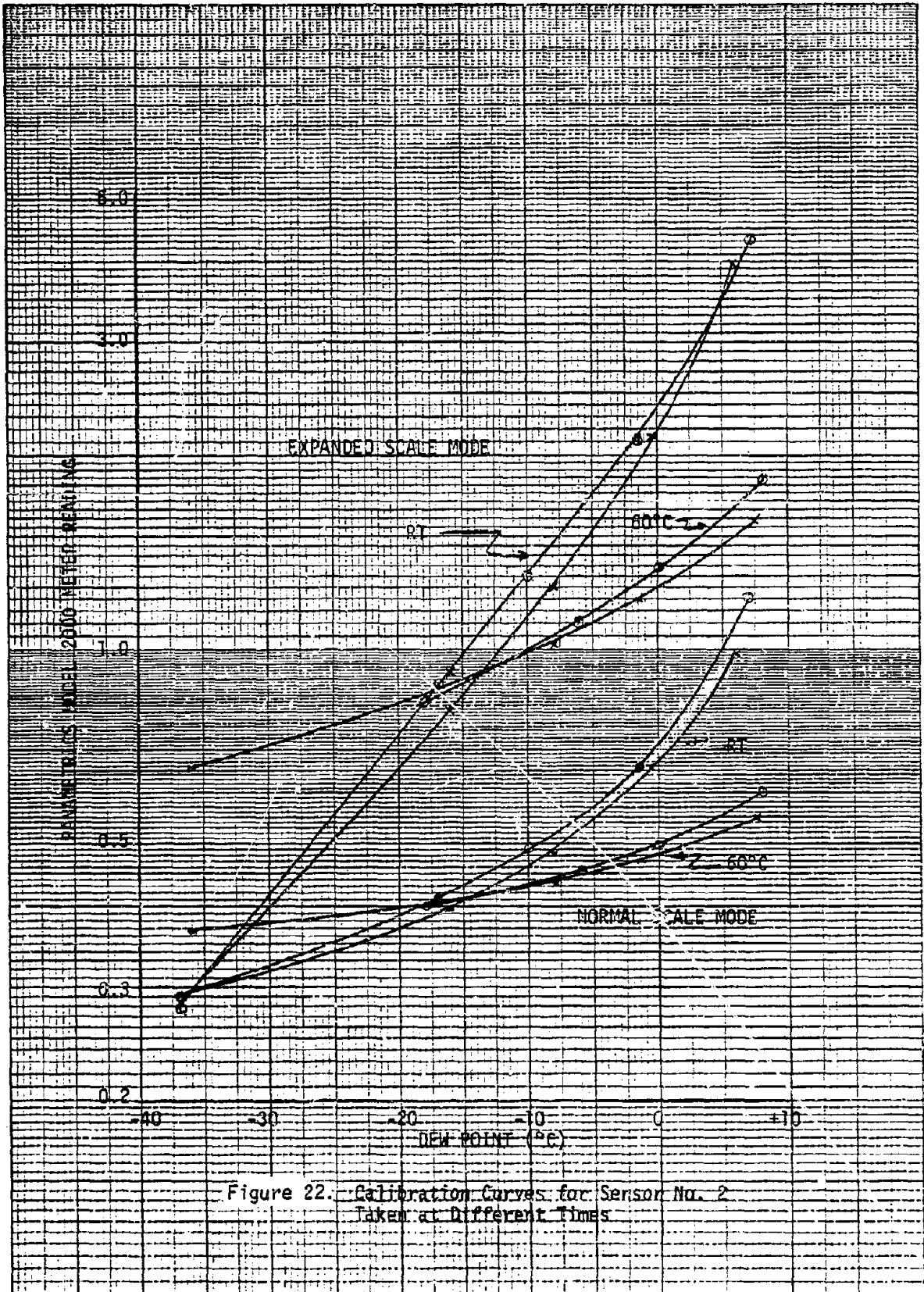


Figure 22. Calibration Curves for Sensor No. 2
Taken at Different Times

350T-716
 SEMI-LOGARITHMIC
 REFLECTOR TYPE CO.
 1 CYCLES X 10 DIVISIONS
 MODEL 2000
 MADE IN U.S.A.
 AT DALLAS, TEX.

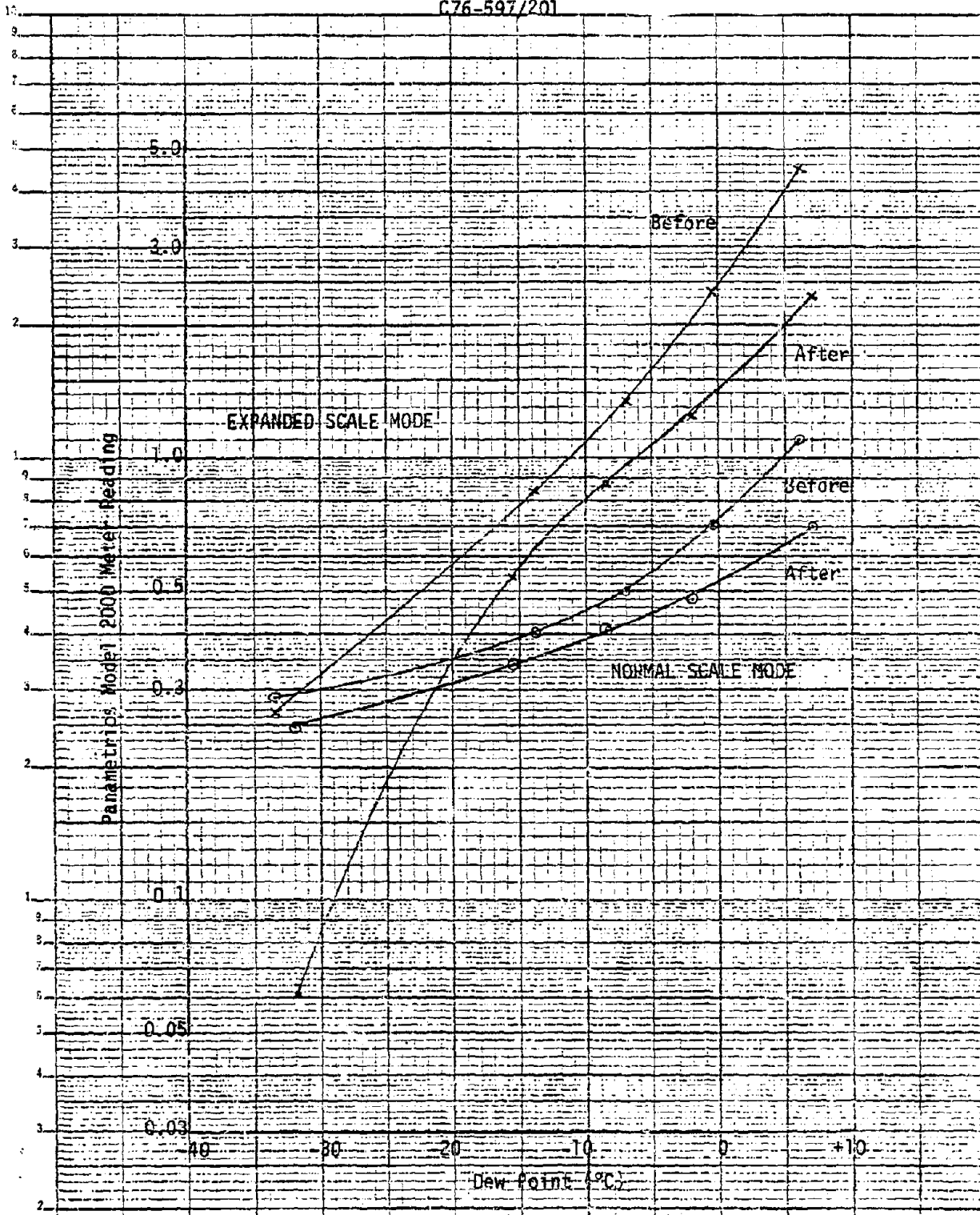


Figure 23. Calibration Curves for Sensor No. 1 at Room Temperature Before and After Six-Hour Bake at 170°C in N₂

17-20 BERTECH/AMETEK 3501 716
 MODEL 1000
 1 CYCLE 10 DIVISIONS

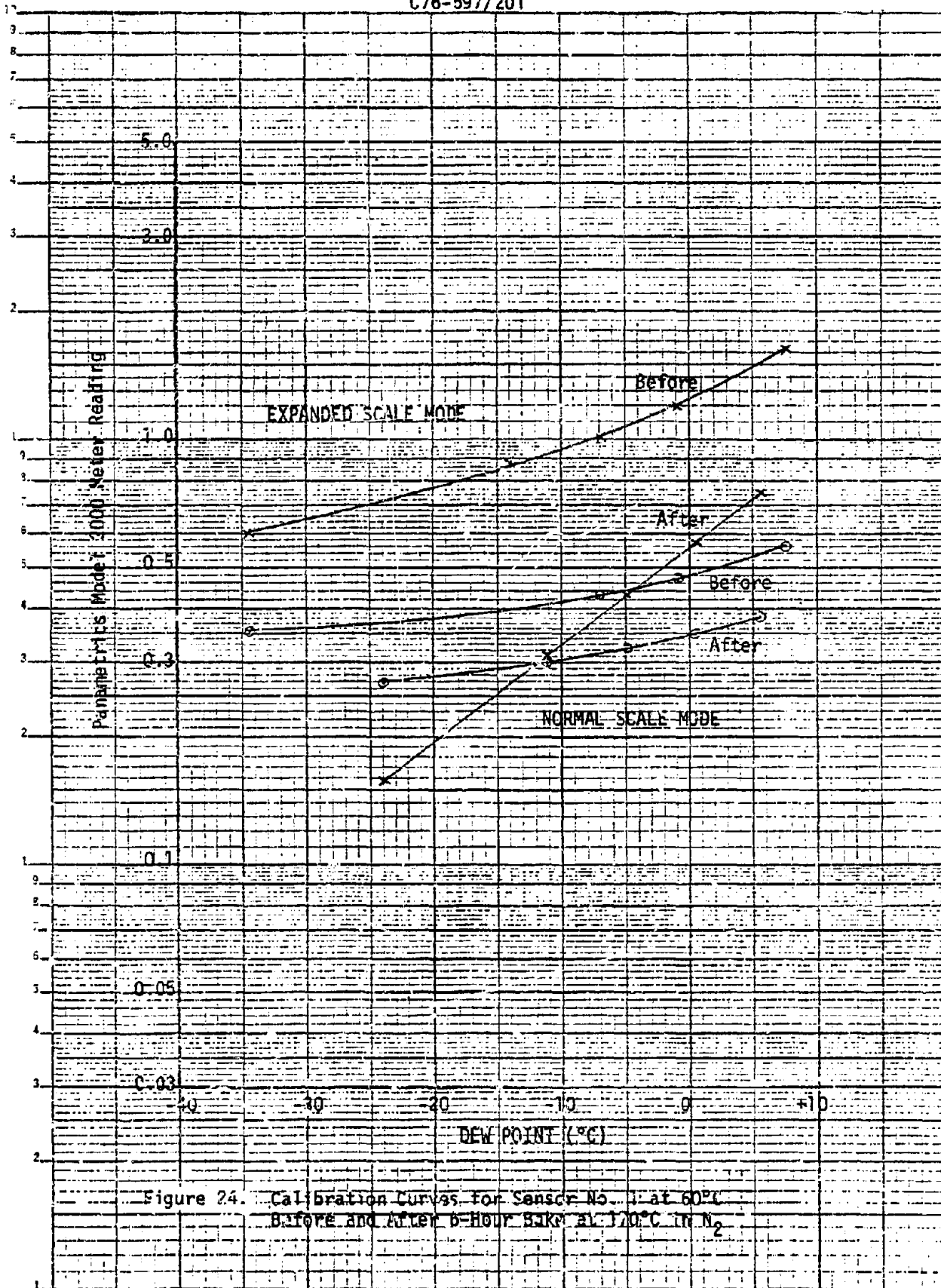
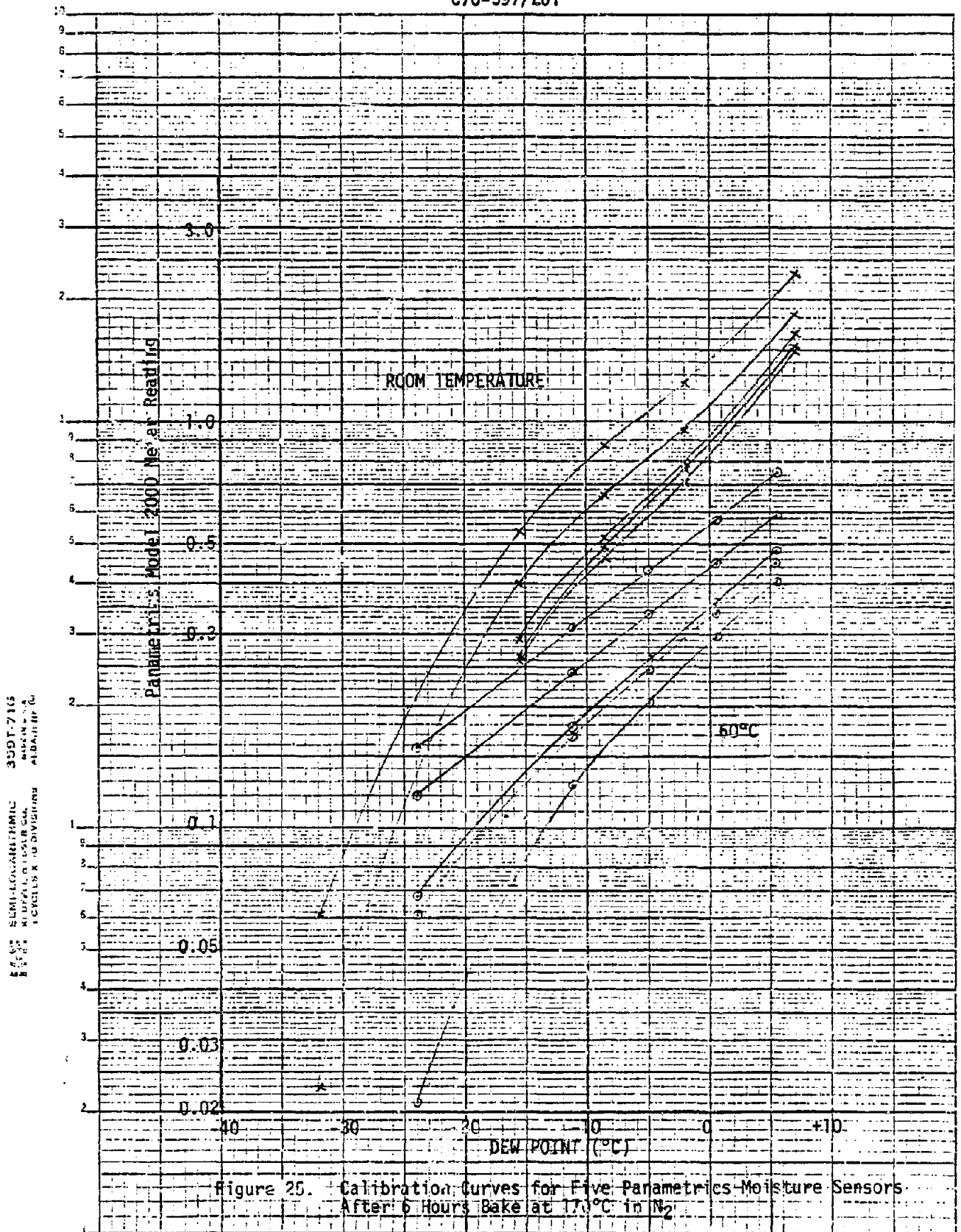


Figure 24. Calibration Curves for Sensor No. 1 at 60°C Before and After 6-Hour Soak at 170°C in N₂



339T-716
 SEMI-LOGARITHMIC
 ALUMINUM
 1 CIRCLES X 10 DIVISIONS

3505-716
ALUMINUM
ALUMINUM
ALUMINUM

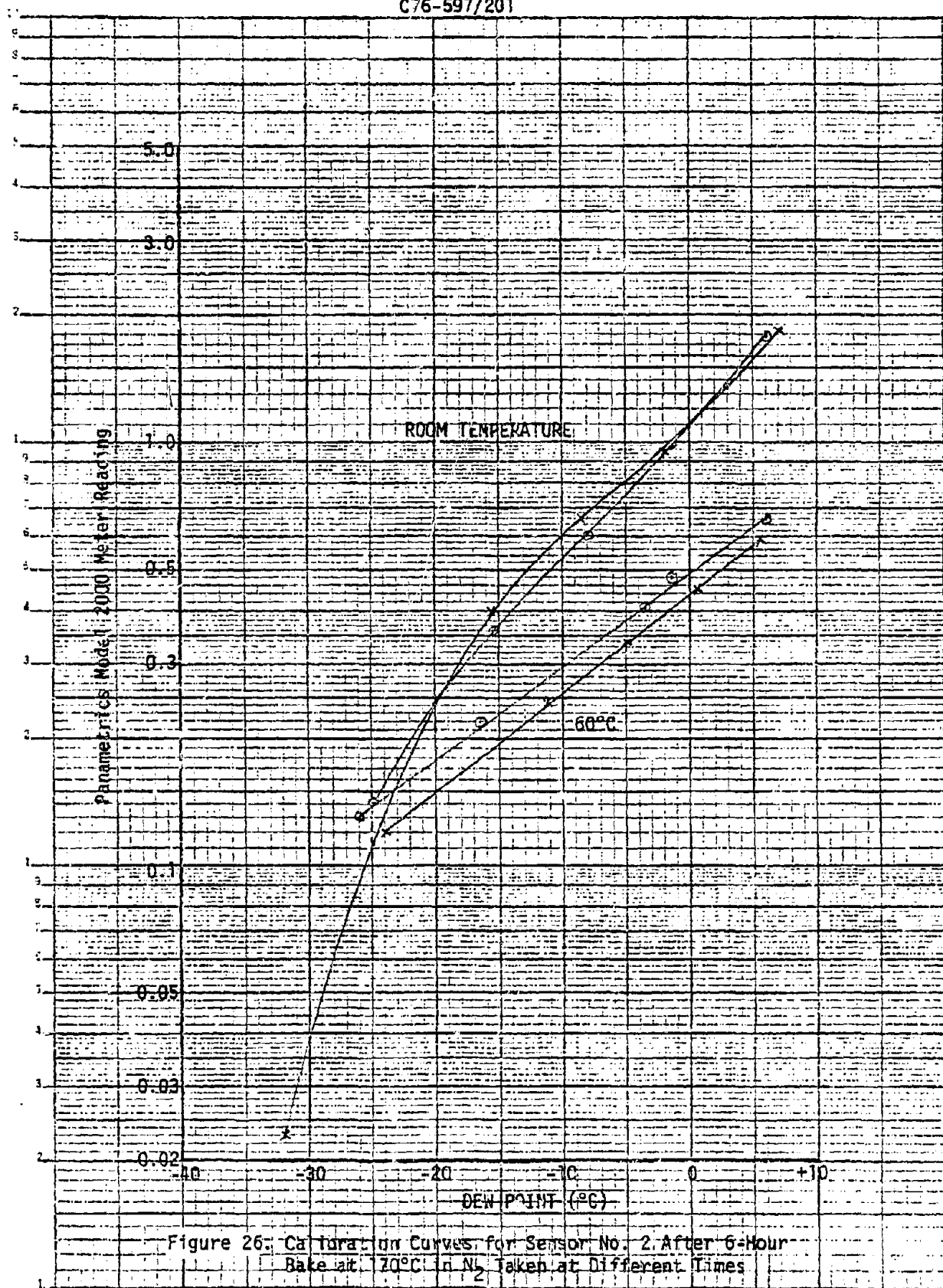


Figure 26: Capture Curves for Sensor No. 2 After 6-Hour Bake at 70°C in N₂ Taken at Different Times

The new calibration curves for all five sensors (with the control unit operated in the expanded scale mode) at room temperature and at 60°C are shown in Figure 25. Typical results showing repeatability are given in Figure 26 (Sensor No. 2). In general, the repeatability is reasonably good (not worse than 2 or 3°C). At a dew point of -10°C this would represent a possible value of moisture content ranging from 2300 to 2900 ppm_v; or at a dew point of 0°C, a possible value ranging from 5350 to 6700 ppm_v. This represents an error in ppm_v of approximately 11 or 12%.

2.3.8 Packages for 60°C/98% RH Exposure

2.3.8.1 Package Preparation

Three ceramic packages and one gold-plated Kovar package were prepared for exposure to the 60°C/98% RH environment. Preparation procedures were as follows:

Ceramic Packages - The carrier substrates containing the moisture sensors Numbers 2, 3 and 4, were removed from the calibration chamber by breaking the leads connecting their terminals to the chamber pins and the gold ribbon strapping them to the chamber plate. They were then strapped to the ceramic package bases and electrical connections made to the package terminals as described in Section 2.3.3 and shown in Figure 16. During this operation, precautions were taken to assure that the sensors were protected from exposure to static charges; and to assure such protection in subsequent operations, the package terminals were shorted together by bonding a wire between them.

The packages were assembled using the assembly fixture designed for the ceramic packages discussed in Section 2.2.1, and the assembly method described in Section 2.1.1. In this case, to accommodate the larger ceramic package bases, the two alignment pins on the right side of the assembly fixture were removed. Also in this case, the small hole (approximately 0.04 cm or 16 mils in diameter) required to allow the packages to vent during curing of the adhesive, was sandblasted in the ceramic boxes which served as the package lids. These lids were cleaned using the same procedure as described in Section 2.2.1 (i.e., brushed in toluene and isopropyl alcohol and spray rinsed with Freon TF). The paste adhesive (Ablebond 789-1) was manually applied on the rims of the lids as described in Section 2.1.2. The packages were assembled in room ambient

and cured in a dry nitrogen environment for two hours at 170°C with a clamping pressure of approximately $7.0 \times 10^4 \text{ N/m}^2$ (10 psi) applied to them. The packages were then removed from the oven directly into the attached dry nitrogen glove box (maintained at a dew point of approximately -60°C), the clamps and teflon-coated plates removed, the vent holes filled with Epo-Tek H77, and the packages replaced in the dry nitrogen oven and cured for 30 minutes at 150°C. After this, the packages were stored in the dry nitrogen glove box. A photograph of a completed package is shown in Figure 27.

Gold-Plated Kovar Package - The carrier substrate containing moisture sensor Number 1 was removed from the calibration chamber as described above and strapped directly to the bottom of the gold-plated Kovar package using gold ribbon. The substrate terminals were connected to two of the package leads by ultrasonically bonded 0.0038 cm (1.5 mil) diameter gold wire. The package leads were then shorted together to protect the sensor from static charges, and the package was vacuum baked for four hours at 135°C and seam-sealed in a dry nitrogen environment.

2.3.8.2 Package Conditions Prior to Exposure

Immediately after the ceramic packages were sealed, they were removed from the dry nitrogen glove box and readings were taken for them and the seam-sealed gold-plated Kovar package using the Panametrics Model 2000 Control Unit. The packages (including the seam-sealed gold-plated Kovar package) were then stored in the entry lock to the dry box (which also is purged with dry nitrogen) until they could be seal tested approximately four days (88 hours) later. Readings were repeated at that time and six hours later after seal testing was completed. The packages were returned to the entry lock for an additional six days (138 hours) before they were removed and placed in the temperature-humidity chamber for testing. These readings and the measured leak rates are given in Table 27.

In all cases, for all four packages, the readings were made with the control unit operating in the normal scale mode because they were too low to be detected with the unit operating in the expanded scale mode. The adhesive-sealed ceramic package containing moisture Sensor No. 2 was found to be a gross leaker because of a hole in the ceramic lid. This problem was discussed previously in Section 2.2.2. The lids were pretested, but either this one was

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Figure 27. Adhesive-Sealed Ceramic Package
for Moisture Permeation Testing

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Table 27. Package Conditions Prior to 60°C/98% RH Exposure

Package Type	Panametrics Model 2000 Meter Readings (Normal Scale)				Leak Rate Air Equivalent (atm cc/sec)
	Immediately After Sealing (Zero Hours)	Before Seal Test (88 Hours)	After Seal Test (94 Hours)	At Start of 60°C/98% RH Test (232 hrs)	
Moisture Sensor					
Seam-Sealed Gold-Plated Kovar					
Sensor No. 1	0.205	0.205	0.208	0.215	2.0×10^{-8}
Adhesive-Sealed Ceramic					
Sensor No. 2	0.230	0.220	0.282	0.270	Gross C_1 (Hole in Cover)
Sensor No. 3	0.210	0.202	0.202	0.215	3.6×10^{-7}
Sensor No. 4	0.203	0.199	0.200	0.218	2.4×10^{-7}

missed or the defect developed when the small vent holes were sandblasted.

2.3.9 Exposure of Packages to 60°C/98% RH Environment and Discussion of Results

As previously discussed in Section 2.3.4, since it was not known to what extent the calibration of the sensors was affected by the additional high temperature exposure during package sealing, this effort also involved recalibrating the sensors. The procedure for this effort was then as follows:

- (1) The packages were exposed to the 60°C/98% RH environment and the sensor outputs monitored.
- (2) The sensors were removed from the packages and recalibrated at 60°C.
- (3) The sensor output readings were interpreted as dew points using the calibration curves, converted to ppm_v, and plotted versus exposure time.

A detailed presentation of the data and discussion of the results follows.

2.3.9.1 Exposure Data

Prior to installing the packages, the temperature-humidity chamber (Blue M Model FR-256PB) was stabilized at 60°C/98% RH. An empty card-type connector used with the ceramic packages was then installed in the chamber and tested for electrical leakage using both the Panametrics Model 2000 Control Unit operated in the Normal Scale Mode and a Delta FET VOM. For no leakage, the Control Unit gives a zero reading and the VOM shows infinity. Both instruments indicated that leakage was occurring (the Control Unit gave a reading of 0.042 and the VOM indicated 6 megohms). The wet bulb control setting was lowered slightly and the leakage, as indicated by the Control Unit reading, slowly decreased. No further adjustments were made. By the next morning the leakage (as indicated by the Control Unit reading) was essentially zero. The solder lug side of the connector was then sprayed with Permatex DLF, and the Control Unit reading dropped to zero. Also, the Delta FET VOM indicated infinity. An empty ceramic package base was then inserted in the connector. The leakage, as indicated by the Control Unit reading, initially increased, (apparently due to the fact that moisture condensed on the ceramic package base since it was initially at RT) and then decreased to zero (as the ceramic package base warmed up to 60°C). As a result of the small adjustment in the wet bulb temperature, the relative humidity is slightly less than 98%.

All packages, including the one that was a gross leaker, were then installed in the temperature-humidity chamber and sensor outputs monitored with the Panametrics Model 2000 Control Unit. The exposure was continued for 15 days. While data was taken much more frequently, an adequate summary of the data for the seam-sealed gold-plated Kovar package and the two good adhesive-sealed ceramic packages is given in Table 28. As this data shows, the output of the moisture sensor in the seam-sealed gold-plated Kovar package remained at a low constant value throughout the entire 15-day exposure, while the outputs of the sensors in the adhesive-sealed ceramic packages slowly but steadily increased. The behavior of the output of the moisture sensor in the adhesive-sealed ceramic package that was a gross leaker was completely different. It increased very rapidly to a value of 2.59 on the Normal Mode Scale after 7-1/2 hours (the evening of the first day) and was found to be high off scale (i.e., greater than 30) the next morning. It remained high off scale until about one o'clock of the second day (a total of 28 hours exposure); then decreased rather steadily to a value of 0.028 by the end of 96 hours, and remained essentially constant (0.025 to 0.032) at this value for the duration of the 15-day exposure.

2.3.9.2 Condition of Sensors After Exposure

At the end of the 15-day exposure, the packages were opened and visually examined. All sensors except the one from the ceramic package that was a gross leaker appeared the same as they did originally. A photograph of this sensor is shown in Figure 28. As the photograph shows, this sensor was extensively corroded everywhere (even the alignment cross was corroded) except under the gold terminal attached to the gold electrode. It was speculated and later verified by Panametrics personnel that there was no aluminum under this terminal. A close-up view of the terminal to the aluminum electrode (Figure 29) shows that the aluminum neck between the gold-plated terminal and the aluminum electrode was completely corroded.

This result is considered serious. It raises a question concerning the long-term and perhaps even the relatively short-term reliability of these sensors. While it is admitted that this sensor was exposed to a rather severe environment, approximately 19% moisture (190,000 ppm_v) at 60°C, it failed in approximately 24 hours. The question is then to what extent and how quickly

Table 28. Sensor Outputs as Read by the Panametrics Model 2000 Control Unit During 15-Day 60°C/98% RH Exposure*

Elapsed Days	Time of Day	Seam-Sealed Gold-Plated Kovar Package Sensor No. 1		Adhesive-Sealed Ceramic Packages			
		Normal Scale	Expanded Scale	Sensor No. 3		Sensor No. 4	
				Normal Scale	Expanded Scale	Normal Scale	Expanded Scale
0	0900	0.215	Off-Scale Low	0.215	Off-Scale Low	0.218	Off-Scale Low
1	0900	0.203	↓	0.210	↓	0.214	↓
2	0900	0.215	↓	0.228	↓	0.230	↓
3	0900	0.215	↓	0.231	0.002	0.238	0.027
4	0900	0.214	↓	0.238	0.031	0.242	0.059
5	0900	0.215	↓	0.242	0.060	0.250	0.095
6	0930	0.213	↓	0.250	0.091	0.258	0.129
7	0900	0.213	↓	0.255	0.119	0.263	0.162
8	0900	0.214	↓	0.261	0.148	0.271	0.195
9	0900	0.214	↓	0.268	0.178	0.279	0.229
10	0900	0.215	↓	0.272	0.207	0.284	0.261
11	0900	0.214	↓	0.279	0.235	0.291	0.292
12	1210	0.215	↓	0.286	0.270	0.299	0.331
13							
14	0920	0.217	↓	0.298	0.328	0.312	0.398
15	0900	0.217	↓	0.305	0.361	0.320	0.432

* Relative Humidity was slightly less than 98%.

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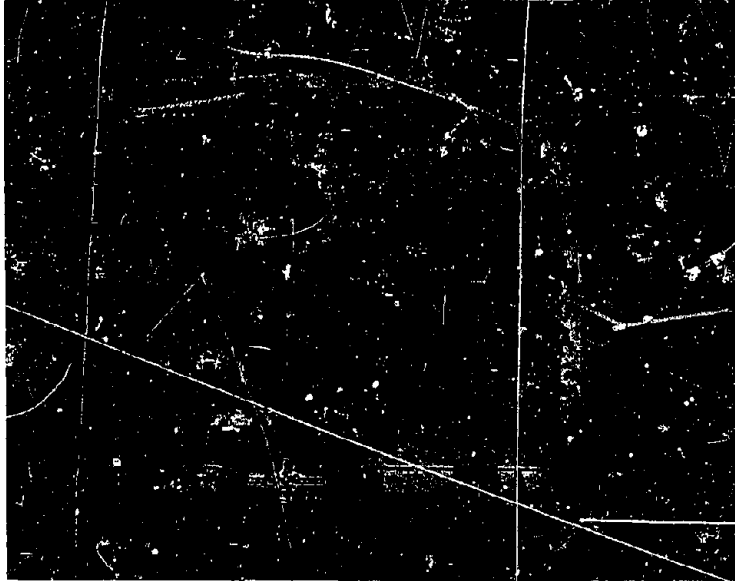


Figure 28. Moisture Sensor Removed From Package That Was a Gross Leaker

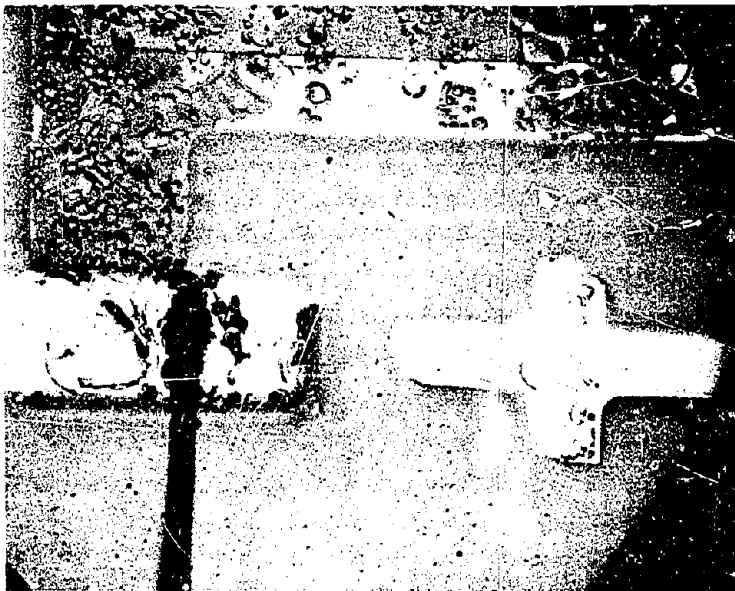


Figure 29. Close-up View of Terminal to Aluminum Electrode

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are these sensors degraded at lower temperatures and/or lower moisture concentrations. It was learned in a telecon with Panametrics personnel that a drift in sensor characteristics has been observed during prolonged exposure under much less severe conditions. This could be due to the very slow occurrence of corrosion. Also, Panametrics does not recommend the sensors (either the Aquamax-type or the new Mini-Mod A-type) for long-term applications. Based on the observed severe corrosion of the Aquamax-type moisture sensor, it is our opinion that a fundamental redesign eliminating the aluminum electrode is required. It is proposed that an improved sensor can be fabricated by sputtering, screening, anodizing, or otherwise applying a porous aluminum oxide or other dielectric on a gold base-electrode, thus eliminating the aluminum-gold bimetallic couple. A patent disclosure for this improved sensor has been made to the Rockwell Patent Department.

2.3.9.3 Recalibration of Sensors After Exposure

The three sensors were removed from the seam-sealed gold-plated Kovar package and the two good adhesive-sealed packages, mounted in the calibration chamber, and recalibrated at 60°C. Results are given in Figures 30 through 32 for Sensor Numbers 1, 3 and 4, respectively. As shown, the sensors were calibrated twice (on different days) for both the normal and expanded scale modes. The results of the two calibrations are nearly identical. Corresponding calibration curves obtained after the six-hour bake at 170°C in nitrogen (i.e., before the sensors were sealed in the packages) are also shown for comparison. It is evident that this exposure (6 hours at 170°C) did not stabilize the calibration curves of the sensors at new values. Contrary to the speculation made in Section 2.3.4, the calibration curves were changed substantially by the additional high-temperature exposures of the sensors during package sealing. Also, it is seen that the calibration curves for Sensor Numbers 3 and 4, which were subjected to the same high temperature exposures (2 hours at 170°C and one hour at 150°C), changed by nearly identical amounts, and that the change in the calibration curves for these sensors was greater than the change in the calibration curves for Sensor No. 1 which was exposed to a lower temperature for a longer period of time (4 hours at 135°C).

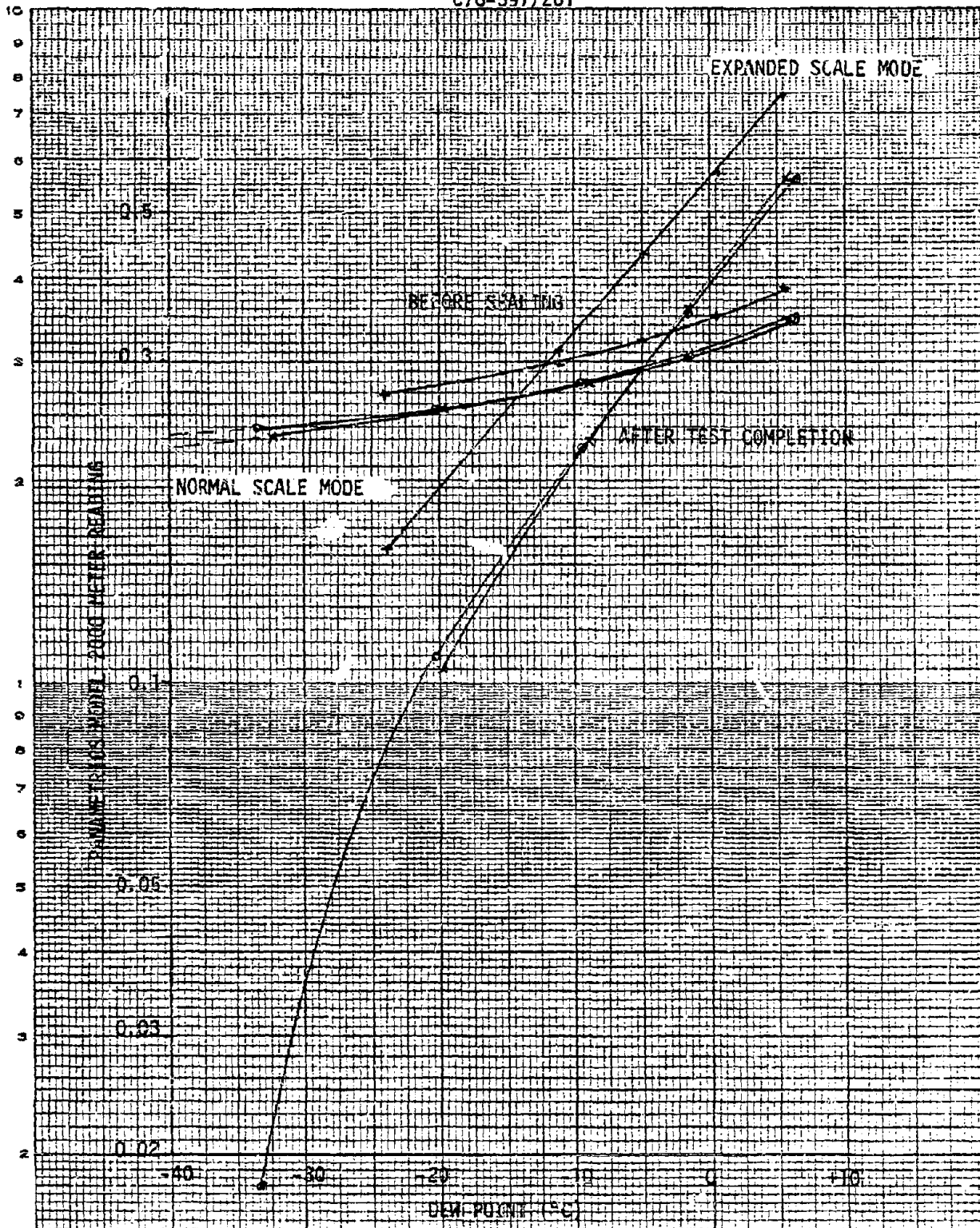


Figure 30. Calibration Curves for Sensor No. 1 at 60°C Before Sealing in Seam-Sealed Gold-Plated Kovar Package and After Test Completion

MIL-STD-2000-2000
 SEMI-LOGARITHMIC
 2 CYCLES X 20 DIVISIONS PER INCH

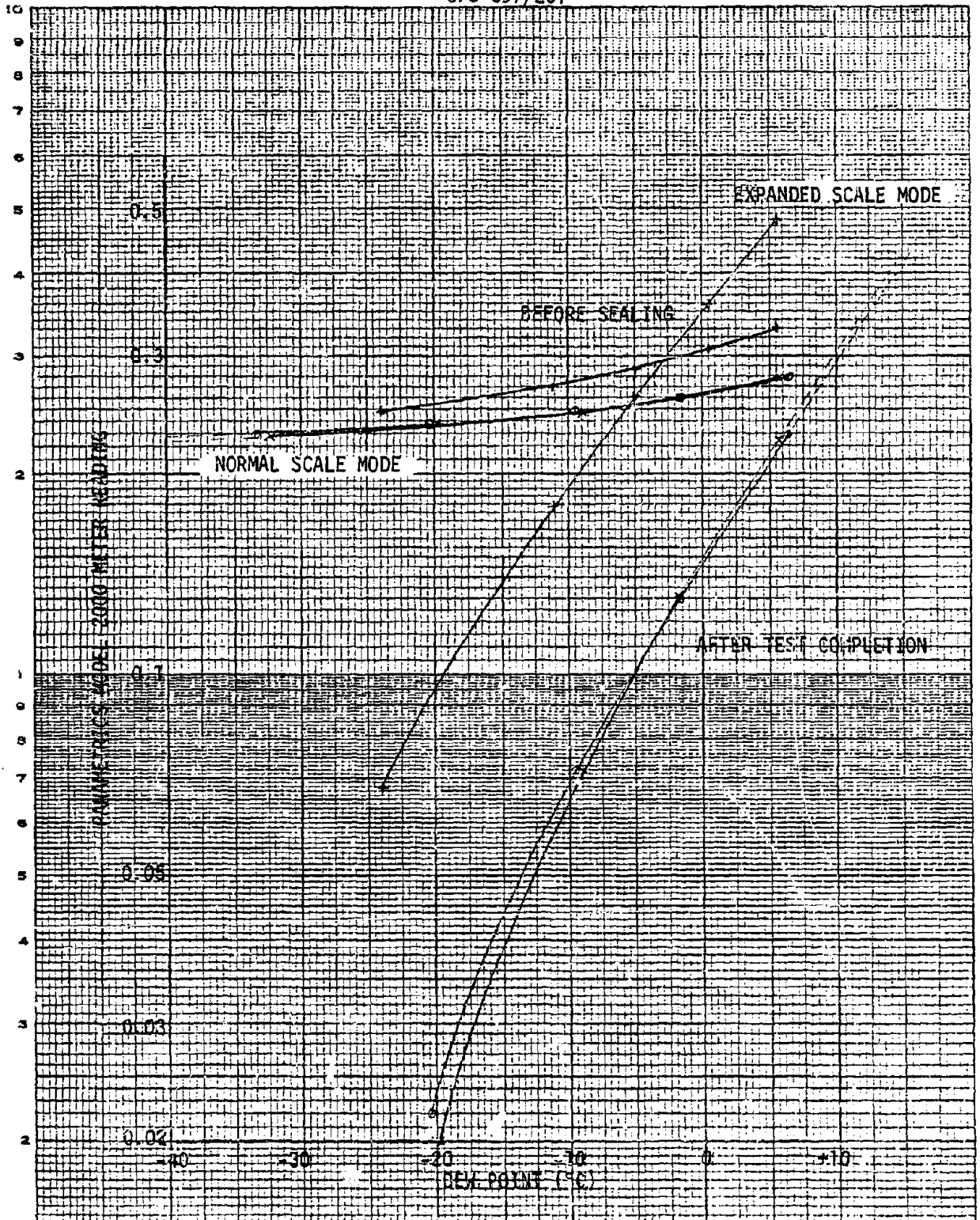


Figure 31. Calibration Curves for Sensor No. 3 at 60°C Before Sealing in Adhesive-Sealed Ceramic Package and After Test Completion

MODEL 2000
 SEMI-CARTESIAN
 2 CYCLES X 20 DIVISIONS PER INCH

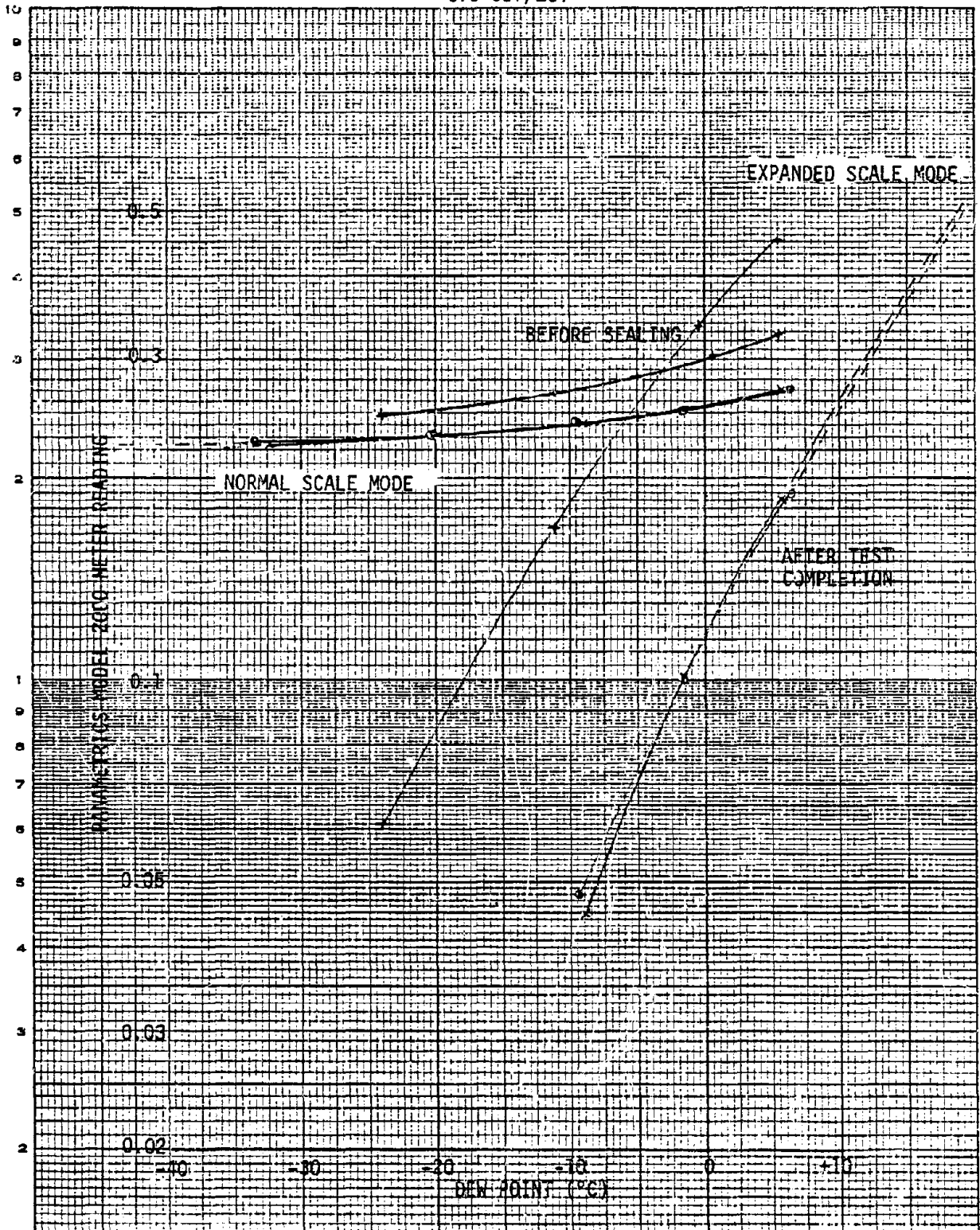


Figure 32. Calibration Curves for Sensor No. 4 at 60°C Before Sealing in Adhesive-Sealed Ceramic Package and After Test Completion

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2.3.9.4 Results of 60°C/98% RH Exposure

Interpretation of the sensor output readings recorded during the 15-day exposure of the packages to the 60°C/98% RH environment (Table 28) using the new calibration curves is given in Table 29 for the two good adhesive-sealed ceramic packages. The readings for the seam-sealed gold-plated Kovar package were too low to be accurately interpreted, but the dew point remained essentially constant throughout the 15-day exposure and certainly was less than -40°C and probably around -55°C. Therefore, the moisture content of this package was less than 130 ppm_v and probably only about 20 ppm_v. As shown in Table 29, the dew points for the adhesive-sealed ceramic packages were initially the same as that for the seam-sealed gold-plated Kovar package but steadily increased with elapsed exposure time. At the end of the 15-day exposure, the dew point of the package that contained Sensor No. 3 was +12.8°C and that of the package that contained Sensor No. 4 was +16.9°C, indicating that the moisture contents of the packages were 14,620 and 19,030 ppm_v, respectively.

These data (moisture content versus exposure time) are plotted in Figure 33. After the first few days (four days for the package that contained Sensor No. 3 and two days for the package that contained Sensor No. 4), the curves are linear for the remainder of the test (15 days). During this time, moisture permeated into the package that contained Sensor No. 3 at the rate of 1,230 ppm_v per day and into the package that contained Sensor No. 4 at the rate of 1400 ppm_v per day. Further discussion of the results obtained for the adhesive-sealed ceramic packages from several viewpoints follows.

2.3.9.4.1 Measured Fine-Leak Rates

The fine-leak rates measured for these packages (per MIL-STD-883A, Method 1014.1, Test Condition A₂) prior to the 60°C/98% RH exposure were 3.6×10^{-7} and 2.4×10^{-7} atm cc/sec (air equivalent) for the packages that contained Sensor Numbers 3 and 4, respectively (see Table 27). On this basis, it would be expected that moisture would permeate more readily into the package that contained Sensor No. 3. However, the above results showed the opposite. There are four possible explanations for these conflicting results: (1) the leak rate measurements were in error, (2) the leak rates changed prior to exposure, (3) there was more adhesive on the package that contained Sensor Number 3, and

Table 29. Interpretation of Sensor Output Readings for Adhesive-Sealed Ceramic Packages

Elapsed Days	Time of Day	Package Containing Sensor No. 3			Package Containing Sensor No. 4		
		Panametrics Model 2000 Meter Reading	Dew Point (°C)	Moisture Content (ppm _v)	Panametrics Model 2000 Meter Reading	Dew Point (°C)	Moisture Content (ppm _v)
0	0900	0.215*	< -40	< 130	0.218*	< -40	< 130
1	0900	0.210*	< -40	< 130	0.214*	< -40	< 130
2	0900	0.228*	-35.0	220	0.230*	-22.0	840
3	0900	0.231*	-29.0	415	0.238*	-11.5	2250
4	0900	0.031	-17.7	1260	0.059	- 7.0	3320
5	0900	0.060	-11.3	2290	0.095	- 2.3	5000
6	0930	0.091	- 6.4	3510	0.129	+ 1.1	6570
7	0900	0.119	- 3.0	4700	0.162	+ 3.9	8000
8	0900	0.148	Zero	6020	0.195	+ 6.4	9420
9	0900	0.178	+ 2.6	7240	0.229	+ 8.3	10820
10	0900	0.207	+ 4.7	8400	0.261	+10.2	12290
11	0900	0.235	+ 6.5	9570	0.292	+11.7	13580
12	1210	0.270	+ 8.4	10900	0.331	+13.3	15100
13	--	--	--	--	--	--	--
14	0920	0.328	+11.2	13150	0.398	+15.8	17720
15	0900	0.361	+12.8	14620	0.432	+16.9	19030

*Sensor outputs read with the Panametrics Model 2000 Control Unit operated in the Normal Scale Mode.
 All others are sensor outputs read with the Control Unit operated in the more sensitive but restricted range Expanded Scale Mode

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K&E 10 X 10 TO THE CENTIMETER 10 X 25 CM.
KEUFFEL & ESSER CO., MANHATTAN, N.Y.

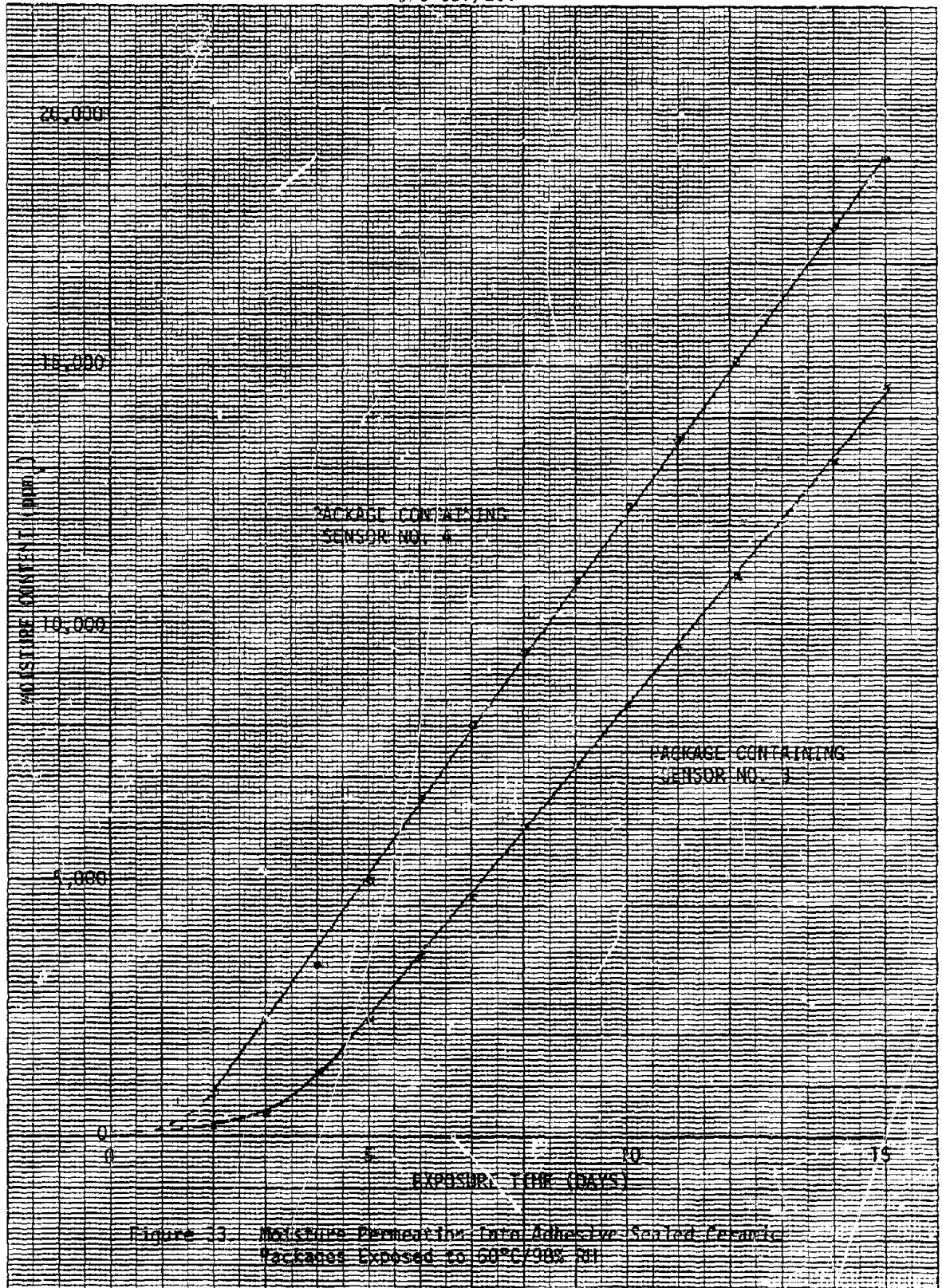


Figure 3. Moisture Permeation Into Adhesively Sealed Ceramic Packages Exposed to 60°C/90% RH

(4) the helium leak test is inappropriate for measuring leak rates when the dominant mechanism is diffusion rather than convection. Explanations 1 and 2 are unlikely. Since the packages were exposed to identical conditions after the leak rates were measured, there is no reason why one should have changed in comparison to the other. The absolute values of the leak rates may have been in error, but their relative values should have been accurate since they were leak tested at the same time. Explanation 3 cannot be completely discounted, however, it is implausible because the leak rate for the package that contained Sensor No. 3 is 50% greater than the leak rate for the package that contained Sensor No. 4. Even if the observed leak rates were due entirely to adsorbed and/or absorbed helium, this difference would require that the package that contained Sensor No. 3 had 50% more adhesive on it than the package that contained Sensor No. 4. Explanation 4 is the most likely. As evidenced by the curves shown in Figure 33, diffusion requires hours or even days to stabilize, and the fine-leak test is performed in minutes.

2.3.9.4.2 Difference in Moisture Permeation Rates

The observed difference in the rates at which moisture permeated into the two packages is simply explained as due to a small difference in the thickness of their bond lines (or seals). The only variable parameter for the two packages that affects the permeation rate is the seal area. The seal perimeter is the same for both packages, but the seal thickness is not controlled.

2.3.9.4.3 Time to Reach 6000 ppm_v

The maximum moisture content allowed in a hybrid package has been set at 6000 ppm_v (MIL-STD-883A, Method 5008). As seen from the curves in Figure 33, this level was reached at the end of eight days exposure at 60°C/98% RH for the package that contained Sensor No. 3 and after 5-1/2 days for the package that contained Sensor No. 4.

2.3.9.4.4 Permeability

Seal permeabilities for the two adhesive-sealed ceramic packages can be calculated from the 15-day exposure data using a simple rearrangement of the following equation reported by R. K. Traeger of Sandia Laboratories.

$$t = \frac{VL}{PART} \ln \left(\frac{p_0 - p_1}{p_0 - p_2} \right)$$

where,

- t is the time to reach p_2 (sec)
- V is the internal free volume of the package (cm^3)
- L is the diffusion path length or seal width (cm)
- P is the permeability of the sealant (gm/cm sec Torr)
- A is the total cross-sectional area of the seal (cm^2)
- R is the gas constant ($3465 \text{ Torr cm}^3/\text{°K gm}$)
- T is the temperature ($^{\circ}\text{K}$)
- p_0 is the external water vapor pressure (Torr)
- p_1 is the initial, internal water vapor pressure (Torr)
- p_2 is the final, internal water vapor pressure (Torr)

Rearranging this equation to solve for the permeability gives:

$$P = \frac{VL}{ARTt} \ln \left(\frac{p_0 - p_1}{p_0 - p_2} \right)$$

In the present case, all terms are known, or can be calculated, except the bond line thickness which is required to calculate A. However, it is known that the bond line thickness is not greater than 3 mils, so calculations were made for bond line thicknesses of 1, 2 and 3 mils. The values of the various terms used in the present calculations are given in Table 30, and the calculated permeabilities are given in Table 31. As shown, the values ranged from 1.29 to 4.42×10^{-13} gm/cm sec Torr. These values for 60°C are in excellent agreement with the value of 4.2×10^{-13} gm/cm sec Torr reported by Traeger as measured by Kass at 51°C . However, Traeger states that the epoxy for which Kass measured this value was a very highly filled material that was not usable as a lid sealant. This was not so in the present case. It is important to note that (except for the value measured by Kass) our calculated permeability is approximately two orders of magnitude smaller than those previously reported in the literature.

2.3.9.4.5 Time to Half Ambient

The calculated permeabilities were used to calculate the time required for the moisture content of the packages to reach a value equal to half that of

Table 30. Values Used in Calculating Permeabilities

Both Packages	Package Containing Sensor No. 3	Package Containing Sensor No. 4
<p>V = 1.05 cm³</p> <p>L = 0.127 cm</p> <p>A = 0.0232 cm² (1 mil bond line)</p> <p>= 0.0465 cm² (2 mil bond line)</p> <p>= 0.0697 cm² (3 mil bond line)</p> <p>R = 3465 Torr cm³/°K gm</p> <p>T = 333°K</p> <p>P₀ = 146.39 Torr (98% RH)</p>	<p>P₁ = 1.74 Torr (2290 ppm_v)</p> <p>P₂ = 11.11 Torr (14620 ppm_v)</p> <p>t = 10 days = 0.864 x 10⁶ sec</p>	<p>P₁ = 0.64 Torr (840 ppm_v)</p> <p>P₂ = 14.46 Torr (19030 ppm_v)</p> <p>t = 13 days = 1.123 x 10⁶ sec</p>

Table 31. Calculated Permeabilities
(10⁻¹³ gm/cm sec Torr)

Assumed Bond Line Thickness	Package Containing Sensor No. 3	Package Containing Sensor No. 4
0.00254 cm (1 mil)	3.86	4.42
0.00508 cm (2 mils)	1.93	2.21
0.00762 cm (3 mils)	1.29	1.47

the external environment. This calculation was made using the previous equation:

$$t = \frac{VL}{PART} \ln \left(\frac{p_0 - p_1}{p_0 - p_2} \right)$$

For this calculation, p_1 was assumed to be zero and p_2 was assumed to be $1/2 p_0$, giving:

$$t_{1/2} = \frac{VL}{PART} \ln (2)$$

or

$$t_{1/2} = 0.693 \frac{VL}{PART}$$

For the two extreme values calculated for the permeability (i.e., 1.29 and 4.42 $\times 10^{-13}$ gm/cm sec Torr), the times to half ambient were calculated to be approximately 103 days and 90 days, respectively.

Simple mathematical extrapolation of the curves of Figure 33 gives times to half ambient of 83 days and 71 days for the packages that contained Sensors Numbers 3 and 4, respectively. These values are expected to be low because no provision has been made for the fact that the rate at which moisture permeates into the packages will decrease as the moisture vapor pressure in the packages increases.

In any case, these calculations show that at 60°C, the moisture content of the packages will reach a value equal to half that of the external environment in approximately three months.

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3.0 SUMMARY

A systematic study was performed to evaluate the suitability of adhesives for sealing hybrid packages for NASA/MSFC applications. This study consisted of three parts.

- (1) Screening ten selected adhesives on the basis of their ability to seal gold-plated Kovar butterfly-type packages that retain their seal integrity after individual exposures to the following four increasingly severe temperature-humidity environments:
 - (a) Ten days at 50°C/60% RH
 - (b) Ten days at 60°C/98% RH
 - (c) The ten-day moisture test per Method 1004.1 of MIL-STD-883A
 - (d) Ten days at 85°C/85% RH
- (2) Screening the four best adhesives, as determined from the temperature-humidity screen, on the basis of their ability to seal gold-plated Kovar butterfly-type packages and ceramic packages that retain their seal integrity after both individual and sequential exposure to the following Class A test environments specified in Method 5004.2 of MIL-STD-883A:
 - (a) Thermal Shock - Method 1011.1, Test Condition C (i.e., 15 cycles, -65°C to +150°C)
 - (b) Temperature Cycling - Method 1010.1, Test Condition C (i.e., 15 cycles, -65°C to +150°C)
 - (c) Mechanical Shock - Method 2002.1, Test Condition B (i.e., 5 shock pulses at 1500 g's in the Y_1 plane)
 - (d) Constant Acceleration - Method 2001.1, Test Condition A (i.e., 5,000 g's in the Y_1 plane)
 - (e) Temperature Aging corresponding to the temperature/time requirement associated with the burn-in test of Method 1015.1 (i.e., 240 hours at 125°C)
- (3) Subjecting the best adhesive-package combination, as identified by the MIL-STD-883A screen, to a 60°C/98% RH environment and continuously monitoring the moisture content using Panametrics Aquamax-type moisture sensors to determine susceptibility to moisture permeation.

In all cases, seam-sealed gold-plated Kovar packages were used as controls. The ten adhesives selected for temperature-humidity screening were Ablefilm 507, Ablefilm 550, Ablebond 36-2, Ablebond 58-1, Epo-Tek H20E, Epo-Tek H81, Epo-Tek H77, Ablebond 789-1, Ablebond 873-1, and AF-30. The four best adhesives selected for MIL-STD-883A screening were Ablefilm 507, Ablebond 36-2, Epo-Tek H77, and Ablebond 789-1. Packages sealed with two of these, Ablefilm 507 and Ablebond 789-1, retained their seal integrity after exposure to all temperature-humidity environments. Packages sealed with the other two, Ablebond 36-2 and Epo-Tek H77, failed exposure to the 85°C/85% RH environment. The best adhesive-package combination selected for moisture permeation testing was the ceramic package sealed with Ablebond 789-1. All of the gold-plated Kovar packages sealed with the four adhesives failed sequential exposure to the MIL-STD-883A test environments, and all of the ceramic packages passed.

Results for two ceramic packages sealed with Ablebond 789-1 exposed to the 60°C/98% RH environment (moisture concentration of 193,000 ppm_v) for 15 days showed that the moisture content (which initially was only about 20 ppm_v) slowly increased to 1000 ppm_v after the first few days (3-3/4 days for one package and two days for the other), and thereafter steadily increased at constant rates to 14,600 ppm_v in one package and 19,000 ppm_v in the other by the end of the 15-day test. The moisture content was 6,000 ppm_v after eight days for one package, and after only a little over 5-1/2 days for the other. Permeabilities and times to half ambient were calculated to be of the order of 1.3 to 4.2 x 10⁻¹³ gm/cm sec Torr and 90 to 100 days, respectively.

The seam-sealed gold-plated Kovar packages used as controls passed all tests. In the case of the 15-day exposure to 60°C/98% RH, the moisture content of the seam-sealed gold-plated Kovar package remained unchanged at about 20 ppm_v.

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4.0 ACKNOWLEDGMENTS

The authors wish to acknowledge Mr. S. V. Caruso of NASA for his guidance and assistance throughout this program; Mr. M. W. McMurrin, Director of Special Microcircuits at Rockwell, Anaheim, for his support and engineering direction; and W. J. Olson, Advanced Technology Laboratories at Rockwell, Anaheim, CA, for his dedication in performing much of the laboratory work.