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INVESTIGATION OF THE EFFECTS OF HUMIDITY EXPOSURE ON 3501-6/AS PREPREG MATERIAL

Final Report

For period 30 Sep 1980 - 29 Sep 1981

Prepared under Contract N00019-80-C-0400 for

Department of the Navy
Naval Air Systems Command
Washington, DC 20361

19951228 069

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April 30, 1982



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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER R82-915381-5	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) INVESTIGATION OF THE EFFECTS OF HYMIDITY EXPOSURE ON 3501-6/AS PREPREG MATERIAL		5. TYPE OF REPORT & PERIOD COVERED Final Report 30 Sep 1980 - 29 Sep 1981
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) D. A. Scola		8. CONTRACT OR GRANT NUMBER(s) N00019-80-C-0400
9. PERFORMING ORGANIZATION NAME AND ADDRESS United Technologies Research Center East Hartford, CT 06108		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Department of the Navy Naval Air Systems Command Washington, DC 20361		12. REPORT DATE April 30, 1982
		13. NUMBER OF PAGES 114
14. MONITORING AGENCY NAME & ADDRESS (If different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) DTIC QUALITY INSPECTED 2		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
3501-6 Epoxy Resin	Humidity Effects	IR
3501-6 Epoxy Prepreg	Mechanical Properties	DSC
Humidity	Physiochemical Analyses	
Aging	HPLC	
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
Humid aging studies of AS/3501-6 prepreg and 3501-6 neat resin at 25°C at humidity levels of 0, 31, 51, and 93% RH for time periods of 12, 24, 48, 72, 168 and 336 hrs were carried out. The chemical changes of the resin were followed by infrared spectroscopy (IR), high pressure liquid chromatography (HPLC), and differential scanning calorimetry (DSC) studies. Mechanical properties of composites fabricated from humid aged prepreg were also determined.		

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The IR, HPLC and DSC studies reveal that slow advancement of the resin occurs with time, and the concentration of advancement species increase with humidity level. Indications are that dynamic mechanical analyses may be used to follow changes in the cure cycle caused by the advancement species. The mechanical properties of composites fabricated from humid aged AS/3501-6 prepreg are equivalent to the properties of composites fabricated from unaged "as received" prepreg. High humidity exposure of AS/3501-6 is not detrimental to composite mechanical properties.

PREFACE

This work is an account of the work performed by United Technologies Research Center, East Hartford, CT on the effects of moisture on 3501-6/AS prepreg for the NAVAL AIR SYSTEMS COMMAND, Contract N00019-80-C-0400 from September 30, 1980 to September 29, 1981. The work was performed in the Materials Sciences section of the Materials Technology Laboratory. The manager and principal investigator was Dr. Daniel A. Scola. The project monitor was Mr. Richard Dempsey, Naval Air Systems Command, Washington, DC. This report was reviewed and approved by Maxwell Stander, NAVAIR.

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Investigation of the Effects of Humidity
Exposure on 3501-6/AS Prepreg Material

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I. INTRODUCTION

This is the final report of a twelve (12) month program covering the period September 30, 1980 to September 29, 1981.

1.1 Program Objectives

The objectives of this program were to evaluate the effects of humidity exposures of AS graphite/3501-6 prepreg material on resin composition (chemistry), shelf life, processing characteristics and mechanical properties of finished laminates prepared from the prepreg. Other objectives were to define the limits of acceptability of moisture content, to demonstrate that these limits fall within acceptable processing and mechanical property characteristics, and if required, to suggest modification of the cure cycle to accommodate humidity exposure effects.

1.2 Program Tasks

The program consisted of three tasks. Task I consisted of physiochemical analysis and physical property determination of humidity exposed prepregs. In Task II, the processing characteristics of the humidity exposed prepregs were determined. Task III comprised fabrication of laminates and determination of mechanical properties. The effects of humidity exposed AS/3501-6 prepreg on mechanical properties of the composites was established.

II. RESULTS AND DISCUSSION

2.1 Moisture and Volatile Content of "As Received" 3501-6 Resin and AS/3501-6 Prepreg

2.1.1 Gravimetric Method

Table 1 summarizes the moisture and volatile content of as-received 3501-6 epoxy resin and AS/3501-6 prepreg as determined by treatment at RT in vacuum and at 48°C. The total moisture and volatile content of the 3501-6 resin at RT or 48°C for a 24 hr period is ~0.100%. For the prepreg at 48°C, after 24 or 48 hrs, the moisture and volatile content is ~0.20 wt%, twice the amount in the epoxy resin. Continued vacuum treatment of the resin and prepreg increases the loss to ~0.40 to 0.50 wt%, respectively, suggesting that some component, such as curing agent 4,4'-diaminodiphenylsulfone, is being slowly removed.

2.1.2 Mass Spectroscopic Analysis of Total Volatile in AS/3501-6 Prepreg

A sample of prepreg was subjected to high vacuum treatment at 25°C and the off gases were analyzed by mass spectroscopy. This analysis gave the following: 780 ppm H₂O, 61 ppm methylethylketone, and 4.7 ppm toluene. The total volatile content as determined by this technique was 0.085%. The moisture content was 0.078%, while the volatile solvent content was 0.0065%. The volatile and moisture content measured by this technique is in good agreement with the method of weighing prepreg before and after exposure to vacuum at 48°C. Desorption experiments (Table 2) show moisture contents varying from 0.03 to 0.10%.

2.2 Moisture Desorption-Absorption Characteristics of 3501-6 Resin and AS/3501-6 Prepreg at 0% RH

Table 2 lists the desorption-absorption behavior of 3501-6 epoxy resin exposed to air and several desiccants: drierite, P₂O₅ and conc. H₂SO₄. The data from this table for the epoxy resin is illustrated graphically in Fig. 1. The moisture content of 3501-6 resin is approximately 0.1 wt% as determined by P₂O₅ and conc. H₂SO₄ desorption data. The effect of prevailing humidity conditions and sample inhomogeneity on desorption-absorption is illustrated by comparing the first series of tests with the second series of tests for samples exposed to air and drierite (Table 2). In the first series, both in air and drierite, moisture is desorbed while in the second set, moisture is absorbed both from air, and from drierite. Table 3 lists the desorption-absorption behavior of AS/3501-6 prepreg in air and several desiccants: drierite, P₂O₅ and H₂SO₄.

The moisture content of AS/3501-6 prepreg is approximately 0.14 wt% (Table 3). Here, as with the 3501-6 epoxy resin, the prevailing humidity condition and sample inhomogeneity of the AS/3501-6 prepreg from sample to sample is illustrated by the fact that in the first series, in drierite, P₂O₅ and conc. H₂SO₄

moisture is actually absorbed, while in the second series of tests, moisture is desorbed. The ability of the prepreg to behave as a desiccant which is stronger than P_2O_5 or conc. H_2SO_4 suggests that the prepreg contains $BF_3 \cdot MEA$ accelerator distributed inhomogeneously throughout the prepreg, instead of being homogeneously distributed throughout the prepreg. The data for the desorption-absorption behavior of AS/3501-6 prepreg is shown graphically in Fig. 2.

2.3 Moisture Absorption-Desorption Characteristics at Various Humidity Levels

2.3.1 3501-6 Epoxy Resin

As mentioned in section 2.2, the moisture absorption and desorption characteristics of the neat resin and AS/3501-6 prepreg are dependent on several factors: prevailing ambient conditions, surface area (sample size) and sample history. Considerable scatter in the data is obtained due to daily changes in temperature and humidity conditions in the laboratory. A series of moisture absorption experiments were carried out both on neat 3501-6 epoxy resin and AS/3501-6 prepreg. Initially, samples were weighed in an air conditioned laboratory where the relative humidity was supposed to be controlled to ~50%. However, the large scatter in the data suggested that the laboratory air conditioning system did not adequately control the humidity level. Therefore, a special room controlled to 25°C and 50% RH was provided to carry out the experiments and prepreg handling. Even when weighings were carried out in a room controlled for humidity and temperature, considerable variations in moisture absorption and desorption behavior from sample to sample from the same resin or prepreg batch was noted. The data for 3501-6 resin are summarized in Tables 4-7 and shown graphically in Figs. 3, 4, and 5. As noted in Table 4, the dry condition (0% RH) causes a weight loss of ~0.10%, and this doesn't vary very much from the 12 to 336 hr period tested. Within each group of samples, for instance for the 0% and 31% RH samples, there is a considerable difference in the percent desorption or absorption of samples 1, 2 and 3 (Table 4). This suggests that the resin system may be heterogeneous, resulting in a difference in capacity for desorption or absorption of moisture. This variation shows up more consistently for samples listed in Table 5. Each data point for desorption or absorption represents a specimen which was subsequently analyzed by HPLC.

Another factor which is also responsible for these variations is the ambient condition which varies daily and also on a seasonal basis. Examples of this are the data at 51% RH in Tables 4 and 5. Resin samples tested in March 1981 absorbed moisture (0.20 to 0.50 wt%) at 51% RH (Table 4), while samples tested in April 1981 either desorbed moisture (-0.34 wt%) or absorbed moisture (+0.36 wt%) at 51% RH (Table 5), depending on the prevailing humidity level.

Samples tested in May 1981 also desorbed moisture at 51% RH, as shown in Table 6. Apparently if the prevailing humidity levels in the laboratory are greater than 51% on initial weighing, and less than 51% after final weighings, a net loss in weight is experienced. And conversely, as shown in Table 7, epoxy

resin sample measured in September 1981 absorbed moisture under the dry (0%) and 31% RH conditions. In these instances, the prevailing humidity level in the laboratory during initial weighing was extremely low, and the humidity level in the final weighing was very high.

Therefore, it is clear that the moisture desorption and absorption capacity of 3501-6 epoxy resin is dependent on the homogeneity of the sample, and prevailing ambient humidity conditions. Under dry conditions, approximately 0.2 wt% moisture can be lost; under humid conditions approximately 0.5 to 1.0% moisture can be absorbed.

2.3.2 AS/3501-6 Prepreg (Single Ply)

The moisture absorption behavior of the AS/3501-6 prepreg is listed in Tables 8-10 and shown graphically in Figs. 6-8. As can be seen from the data at 0% and 31% RH exposures, a drying out occurs. However, at 51% RH, two samples gained moisture during 12 and 24 hr exposures, while the remainder of the samples lost moisture under this exposure, suggesting that for most of the weighings the RH conditions in the laboratory exceeded 51%. A 93% RH, moisture absorption ranged from 1.7 to 4.0% over the two week period. Here, as with the resin material, the desorption and absorption characteristics depend on prevailing relative humidity conditions in the laboratory. It is significant to point out that a comparison of the quantity of moisture desorbed by AS/3501-6 prepreg (~2.0%) (Table 8) is approximately 10 times greater than the quantity of material desorbed by neat 3501-6 epoxy resin (~0.20%) (Table 6), while the quantity of moisture absorbed at 93% RH by AS/3501-6 prepreg (~3.0%) (Table 8) is approximately six times greater than the quantity of moisture absorbed by neat 3501-6 epoxy resin (0.50%) (Table 5). This suggests that the prepreg is much more sensitive to ambient conditions (perhaps due to its physical form) than the epoxy resin. The presence of volatile materials, other than water in the prepreg does not account for this difference, according to the data in Table 1, and as described in Section 2.1.2. The moisture absorption characteristics of single ply prepreg which were measured in September 1981 shows that ~1% moisture absorption occurred even at 0 and 31% RH. At 51 and 93% RH, approximately 1.8% moisture was absorbed. The quantity of moisture absorbed by AS/3501-6 prepreg at all humidity levels is about twice the quantity absorbed by 3501-6 neat resin (Table 7) under the same conditions.

2.3.2.1 AS/3501-6 (10 plies)

The moisture desorption-absorption characteristics of two miniature AS/3501-6 prepreg lay-ups, a 10 ply prepreg disk (2.54 cm diameter) and a rectangular lay-up (2.34 cm long (0.92")) x 0.63 cm wide (0.25") x 10 plies, are demonstrated in Tables 11 and 12 respectively. Desorption occurs at 0% RH and 31% RH; insignificant desorption and absorption occurs at 51% RH, while approximately 0.25 wt% absorption occurs within 24 hrs at 93% RH. This is less than 10% of moisture absorbed by single ply prepreg samples, showing how surface area is important in desorption and absorption behavior.

2.3.2.2 Summary of Moisture Desorption-Absorption Behavior

The desorption-absorption data for both the neat resin and prepreg are summarized in Tables 13 and 14 for two exposure times, 24 and 48 hrs over the seasons for which the measurements were made. For a 24 hr period, the desorption-absorption behavior of the resin and prepreg are shown in Figs. 9 and 10 respectively. Both for the epoxy resin and AS/3501-6 prepreg the absorption data is more consistent for the 93% RH exposure than the other three humidity levels. For the 0% and 31% RH, there appears to be a drying out effect, and as with the other levels, this varies with sample history and prevailing relative humidity levels at the time the sample is measured. At the 51% RH level the data reflects to a greater extent than the other RH conditions the impact of the ambient relative humidity condition, since the 51% RH is a mid point condition. The second important aspect of the data in Tables 13 and 14 is that greater changes are experienced for the single ply prepreg than for the neat 3501-6 epoxy resin, or for 10 ply prepreg sample showing the effect of surface area on desorption-absorption behavior. It is significant that after 2 weeks at 93% RH (Tables 11 and 12) the 10 ply AS/3501-6 prepreg systems absorbed only 0.60% moisture while the single plies absorbed about 2.0% moisture in only 1 day.

2.3.3 Reversibility Effects of 3501-6 Epoxy Resin and AS/3501-6 Prepreg

Epoxy resin specimens, 3501-6 and AS/3501-6 prepreg were exposed to two highest humidity levels, 51 and 93%, for various time periods, and then subjected to 0% RH to constant weight. This returns the resin or prepreg to near zero moisture content. This required approximately 4 days. The samples were then analyzed by HPLC and DSC analyses to compare with samples exposed only to the same RH condition. Since the experiments were carried out in the summer season, in some cases a weight loss was experienced for several of the samples at the 51 and 93% RH levels. The results of the exposures are listed in Table 15. The results of the HPLC and DSC analyses will be discussed in section 2.4.1.

In the moisture absorption-desorption process which takes place with 3501-6 resin and AS/3501-6 prepreg, a certain fraction of the moisture should react irreversibly with $\text{BF}_3 \cdot \text{MEA}$ to produce boric acid and $\text{CH}_3\text{CH}_2\text{NH}_2 \cdot \text{HF}$ salt and/or fluoroborate ($\text{F}_{4-n}\text{B}(\text{OH})_n^-(\text{CH}_3\text{CH}_2\text{NH}_3^+)$) salts. However, because of the variability of daily humidity conditions and inhomogeneity of resin and prepreg samples, small changes in weights caused by reaction with a component in the resin system present at only ~1 wt% would not be detected in these experiments. As mentioned above these changes are irreversible and could possibly effect resin reactivity and cure. Physical moisture absorption-desorption behavior should be reversible, and this is shown by the data in Table 15. The fact that this moisture absorption is reversible shows that $\text{BF}_3 \cdot \text{MEA}$ in prepreg has already been converted by moisture to other species indicated above. This may occur during manufacture of the prepreg.

2.3.4 $\text{BF}_3 \cdot \text{MEA}$ Content in 3501-6 and Its Moisture Absorption Characteristics

There is one component in 3501-6 which has a great capacity for moisture absorption. This is boron trifluoride monoethylamine, $\text{BF}_3 \cdot \text{MEA}$. It is reported (Ref. 1) to be present in the resin at a concentration of about 1%. Atomic absorption analysis for boron of the neat 3501-6 resin and 3501-6 derived from prepreg showed that the neat resin contained 1.67% $\text{BF}_3 \cdot \text{MEA}$ and that the prepreg derived resin contained 0.94% $\text{BF}_3 \cdot \text{MEA}$. The higher concentration of $\text{BF}_3 \cdot \text{MEA}$ in the neat resin should result in a greater moisture absorption capacity than found, and these values should be greater than found for prepreg. This was not the case. This emphasizes the greater influence prepreg surface area has on moisture absorption-desorption properties.

The moisture absorption behavior of this component is shown in Tables 16 and 17. The first experiments were carried out in March 1981. The second series of experiments were carried out in May 1982. The differences in the laboratory relative humidity conditions show up in the desorption and absorption characteristics of the $\text{BF}_3 \cdot \text{MEA}$. It should be noted that at 0% RH (dry condition) a weight loss of about 0.5% occurs, which is about three times greater than experienced by the 3501-6 epoxy resin. However, at the higher humidity levels, $\text{BF}_3 \cdot \text{MEA}$ absorbed considerable quantities of moisture within the first 12 hr period (Table 17); 4.7% at 31% RH, 9.9% at 51% RH and 19.2% at 93% RH. These two latter RH conditions are fairly representative of conditions in the spring or summertime in many parts of the country, and point out that prepreg exposed to atmospheric conditions in the summer will absorb moisture immediately, due to the presence of $\text{BF}_3 \cdot \text{MEA}$. The effect of moisture on $\text{BF}_3 \cdot \text{MEA}$ would be hydrolysis of $\text{BF}_3 \cdot \text{MEA}$ to fluorinated boric acids or boric acid and $\text{C}_2\text{H}_5\text{NH}_3^+\text{F}^-$, and loss of activity. Loss of accelerator activity should cause a decrease in reactivity of the resin, and should be reflected in the processing studies. However, the results of the present study suggests that the possibility of decreased reactivity due to deactivation of $\text{BF}_3 \cdot \text{MEA}$ in humid atmosphere, is not reflected in processing characteristics or composite properties discussed in section 2.7.

2.4 Development of Chromatographic Procedures for Analysis of AS/3501-6 Prepreg

One of the most important aspects in determining the effects of moisture and aging on the processing of prepreg and mechanical properties of composites fabricated therefrom is the ability to observe changes in chemistry as a function of time and moisture content, and to be able to separate time and moisture effects. High pressure liquid chromatography (HPLC) has the ability to make separations of mixtures in a relatively short time (<1 hr) but its usefulness in the present program is not the ability to separate every component in the mixture, but key components that undergo changes in concentration as a function of time/moisture, and which can be followed. This is the approach taken in the present program in order to determine moisture and time effects in 3501-6/AS prepreg.

Initial liquid-liquid chromatographic (LLS) studies were carried out on resin samples DEN 438, MY720, DER 332 and DDS curing agent using Waters μ Bondapak C₁₈ column in the reverse-phase (RPLLC) mode with an acetonitrile/water solvent system (mobil phase) over a solvent composition range.

The reverse-phase liquid-liquid chromatography (RPLLC) mode was selected based on discussions with investigators in the field, and on literature references (Refs. 1,2). The μ Bondapak C₁₈ column was selected because of its good component separation, moderate capacity and fast analyses. The column material consists of C₁₈H₃₇-chemically bonded to 10 μ size μ porasil (silica) to yield a low polarity surface.

The Waters Model ALC/GPC-244 liquid chromatograph equipped with a Waters Intelligent Information Processing (WIIP) unit for control (Model 720 system controller) and data reduction (Model 730 data module) was used. The module yields a chromatogram in which the run conditions, chromatographic data, pump parameters, time, date, solvent/sample/method and operator identification are on a single comprehensive report. The module integrates and labels each detector/signal (peak) for retention time, and automatically re-zeros base line. The module is programmed to determine the area of each component eluting from the column, and to calculate the amount eluted in terms of the area percent. It enables the operator to set conditions for automatic operation.

Initially chromatographic studies were made on 3501-6 resin in the "as-is" state and advanced state (100°C/100 min), varying solvent composition and run time in the isocratic mode and solvent gradient elution mode. The isocratic conditions, varying solvent composition from 90/10 CH₃CN/H₂O to 50/50 CH₃CN/H₂O gave poor resolution of 3501-6 epoxy resin. The gradient elution technique was then used, in which the solvent composition and polarity was changed in a linear fashion. This is controlled by selection of the curve previously programmed to change solvent composition during the course of the analysis. The gradient elution technique resulted in good separation of the 3501-6 resin components starting with a 50/50 acetonitrile/H₂O mobil phase and running a linear gradient to 100/0 acetonitrile/H₂O phase.

The chromatograms for the as-is 3501-6 resin and advanced 3501-6 resin are shown in Figs. 11 and 12. The peaks for each component are labeled where these are known. With each chromatogram, a printout of each signal or peak in terms of its retention time and quantity in micrograms (if calibrated) or area percents (uncalibrated) is derived. In the "as-is" 3501-6 prepreg, the peak emerging at 10.58 min was defined as the advancement peak.

Table 18 gives the ratios of DDS/MY720, DDS/advancement peak, and MY720/advancement peaks for "as received" and advanced 3501-6 epoxy resin. Comparison of Fig. 11 with Fig. 12 reveals that the peak at 10.58 in Fig. 11 has split into two peaks at 10.60 and 11.17 min (Fig. 12). This complicates the issue of calculating peak ratios. The peak ratios were calculated assuming both peaks were advancement peaks. In addition, microgram quantities represented for each peak were combined and the peak ratio was calculated. These data are shown in Table 18. If these assumptions are correct, the combined weights represented by peaks at 10.6 and 11.17 min indicate that the concentration of the advancement peak has increased due to the partial cure. Table 18 also shows that the ratios for the peak at 11.17 min most likely represents the bulk of the advanced resin component.

Due to the separation in the "advancement" peak, an alternative solvent system was considered in this chromatographic analysis. This led to the selection of the tetrahydrofuran/water system for the mobil phase. A series of chromatographic runs were made with this solvent system using μ Bondapak C₁₈ column in the reverse phase mode, under both isocratic (fixed solvent composition) and solvent gradient conditions.

In the initial studies, the elution times for each component were established using epoxy resin and DDS samples and mixtures of components. This was followed by analysis of 3501-6 resin, with and without the internal standard, benzanilide. A summary of pertinent runs are shown in Figs. 13, 14, 15 and 16 for as-is 3501-6 resin, as-is + benzanilide internal standard, advanced 3501-6, and advanced 3501-6 + internal standard. These represent conditions which could be used to analyze the resin with respect to changes in the pertinent components, DDS, MY720 and advancement peak. A complete run required a total of 70 min including the time to return the solvent pair to its initial solvent composition. The actual run required 55 min. Several runs were made to reduce the total run time without compromise of the accuracy of the method by changing the solvent gradient curve and time, and solvent composition, and also by changing the initial and final times of the run. The rate of change in solvent composition vs time can be controlled by setting the solvent gradient curve.

Examples of these runs are illustrated in Figs. 17, 18, 19 and 20. Figure 17 is an example of changes in initial and final solvent composition (weaker solvent) and gradient time, which shows no resolution of the MY720 epoxy resin and advancement peak, and considerable tailing (retention) of the less polar components. Figure 18 is an example in which the same initial and final solvent composition of 20/80 (THF/H₂O) to 60/40 (THF/H₂O) was used but a different gradient curve (curve 2) was used to arrive at 60/40.

This shows poor resolution, considerable tailing (retention), and no separation of the advancement peak. Figure 19 shows that reduction in the gradient time 20/80 → 60/40, using the same gradient curve as in Fig. 13, also results in poor resolution of the advancement peak and excessive tailing of the less polar components. Figure 20 shows that a reduction of the initial time at 20/80 and time to return to this initial solvent composition of 20/80 reduces overall run time by 20 min without compromise in the separation. Figure 20 is an example of the final chromatographic conditions settled on. The 3501-6 was partially advanced by heating at 100°C for 125 min. The chromatogram in Fig. 21 when compared with the chromatogram for as-is 3501-6 for the same concentrations shows decreases in DDS, MY720 and an increase in the advancement peak. The ratios of peaks will be followed in determining the effects of moisture and aging on AS/3501-6 prepreg.

The final conditions used for separation of the 3501-6 components in this study are listed as follows:

Column: μ Bondapak C₁₈ 4 mm ID x 30 cm length
Flow Rate: 1.2 ml/min (constant)
Solvent System (mobile phase): Tetrahydrofuran (THF)/water
Gradient: linear gradient 20/80 to 60/40
 THF/H₂O over 45 min
Detector: U.V. 254nm at 0.5 or 1.0 AUFS
Sample Size: 15 or 20 μ l of resin solution (THF)
Sample Conc: 0.84 to 1.28 μ g/ μ l
Instrument: Waters ALC/GPC 244 equipped with WIIP modules
 (Model 720 systems controller and Model 730 data module)

Several standard solutions were prepared for HPLC analysis by dissolving the quantities of materials listed in Table 19 in 100 ml tetrahydrofuran using a volumetric flask. A 5 ml aliquot of the solution was further diluted to 25 ml in a volumetric flask. For HPLC analysis, 15 μ l to 20 μ l portion was used.

Table 20 is a summary of the ratios for peaks DDS/MY720, DDS/advancement peak and MY720/advancement peak for 3501-6 resin, 3501-6 + internal standard, advanced 3501-6 and advanced 3501-6 + internal standard. The ratio of MY720/advancement peak shows a regular decrease with time, as expected, and appears to be more consistent than the DDS/advancement ratio. Therefore, the ratio of MY720/advancement peak was followed in subsequent analyses of resins after humidity exposures.

2.4.1 HPLC Analysis of Humidity Aged 3501-6 Epoxy Resin Samples

Utilizing the chromatographic conditions represented by Figs. 20 and 21 for "as-is" and advanced epoxy resin, a series of 3501-6 resin samples were exposed to the relative humidities of 0, 31, 51 and 93% at 25°C after 12, 24, 48, 72, 168, 211, 250 and 363 hrs, and under vacuum at 25°C for 12, 24, and 168 hrs. Samples were then subjected to HPLC, IR, and DSC analyses.

Table 21 summarizes the HPLC analysis in terms of the MY720/advancement peak ratio for 3501-6 samples exposed to various humidity and time conditions. The data is shown graphically in Fig. 22a. For the vacuum and dry condition, it can be seen that the ratio decreases with time, with a significant decrease after 72 hrs (dry condition). A similar trend occurs in the 31% RH condition, which is considered a relatively dry condition, since under these conditions moisture may be desorbed (see Tables 3 or 4).

The 51% and 93% relative humidity conditions, and particularly the 93% conditions show a sharper decrease in the MY720/advancement peak ratio after 72 hrs and longer than the dry samples, suggesting the influence of moisture in catalyzing the cure reaction. Moisture definitely appears to influence the cure of the resin, as shown by these HPLC results.

2.4.2 HPLC Analysis of AS/3501-6 Prepreg

Samples of AS/3501-6 epoxy prepreg were exposed to humidity conditions and times as described for the 3501-6 epoxy resin and then subjected to HPLC analysis. The results, expressed in terms of the MY720/advancement peak ratio, are listed in Table 22 and shown graphically in Fig. 22b. Comparison of the AS/3501-6 prepreg data with the 3501-6 resin data shows a similar trend of a decrease in the MY720/advancement peak ratio with increasing humidity level, for the same time period. This suggests that moisture exerts a catalytic effect on the cure process under these conditions. The results also show that even under dry conditions, the resin advances after aging to 72 hrs. Of particular significance is the fact that the initial MY720/advancement ratios of prepreg are lower than the resin. This indicates that the resin undergoes some advancement during the prepreg manufacture.

These results clearly show that aging at various humidity levels causes a slow cure of the epoxy resin.

2.4.2.1 Reversibility Effects of Humidity Exposed 3501-6 Resin and AS/3501-6 Prepreg

A series of 3501-6 and AS/3501-6 prepreg samples was subjected to 51 and 93% RH for various time periods, dried at 0% RH to an equilibrium weight (4 days) and then subjected to HPLC analysis. The results of these tests are listed in

Tables 23 and 24. Comparison of the data in Tables 23 and 24 for the same time periods and RH conditions with data listed in Tables 21 and 22 shows that for a time period up to 72 hrs, after exposure to 93% RH, the moisture absorption/desorption process does not affect the MY720/advancement peak ratio. This shows that the moisture absorption process is reversible and that the resin aging (cure) process is irreversible, as expected. This is significant in that prepreg humidity aged 72 hrs or longer and which has undergone partial advancement can be dried at 25°C if necessary and then subjected to a cure process tailored to the curing properties of the aged prepreg.

2.5 Infrared Study of Humidity Exposed 3501-6 Resin and AS/3501-6 Prepreg

Epoxy resin was removed from AS/3501-6 prepreg by dissolving in tetrahydrofuran. Neat resin was obtained from Hercules, Inc. and was also studied. Films of the neat resin and resin from prepreg on sodium chloride crystals were prepared from a THF solution of the resin by evaporation of the solvent at room temperature, followed by vacuum treatment at RT for 2 hrs. A similar film on a zinc sulfide crystal was prepared for use in the 93% RH exposure. The change in the epoxy peak at 915 cm^{-1} was followed by calculating the ratio of the epoxy peak to the aromatic peak at 1605 cm^{-1} as a function of time at 0, 31, 51 and 93% at 25°C. This latter peak was selected because it should remain constant with time since it is not involved in a chemical change. Since the film thickness should remain constant and the aromatic peak at 1605 cm^{-1} is not involved in the reaction, any change in this ratio is due to the disappearance of the epoxy peak. This ratio of absorbance $915\text{ cm}^{-1}/1605\text{ cm}^{-1}$ should decrease as the cure reaction proceeds.

The results of this study are shown in Tables 25 and 26 and Figs. 23-28 for neat resin and resin removed from prepreg. It should be mentioned that the scatter in the data makes it difficult to interpret the results. However, for the neat resin system, which has not been subjected to the same manufacturing heat process as the resin removed from the prepreg, there is no apparent change in the epoxy concentration with time in the dry condition (0% RH), 31% and 51% RH for the first seven days. After this time there is a definite decrease in the epoxy concentration with time at these three conditions. For the 93% RH exposure (Fig. 24), no sharp change occurred after the first seven days, but there appears to be a very small decrease on the absorption ratio with time over a 33 day period. The change in the first seven days is insignificant. After two weeks the absorption peak ratio for all four conditions is similar, ~ 0.220 .

However, for the resin removed from prepreg, the results suggest that for the 0%, 31% and 51% RH exposures, there is little or no change in the first week of exposure, and only a very small change after 33 days. This is in contrast to the HPLC analyses which shows an increase in the advancement peak ratio with

time at these humidity levels. It should be pointed out that for the 0% relative humidity exposure, the prepreg becomes dry, boardy and brittle over this time period. For the 93% exposure, the prepreg resin is tacky and pliable, even though it appears to be undergoing a slow advancement in the first seven days and this continues over the 33 day period. After one week, the absorption peak ratio for the 0%, 31% and 51% RH exposed prepreg samples vary from 0.27 to 0.31, while for the 93% RH it is 0.256 indicating that epoxy is reacting with some component in the resin. This suggests that moisture appears to be catalyzing the cure reaction. This is confirmed by the HPLC studies which show a greater decrease in the MY720/advancement peak ratio with time at 93% RH than at 0, 31 and 51% RH levels.

The prepreg after 93% RH has considerable flexibility. The flexible and tacky nature of the prepreg after exposure to 93% RH is caused by moisture which is plasticizing the resin, leading to a flexible, pliable system, even after two weeks exposure.

The 3501-6 epoxy resin consists of epoxy components MY720 resin, CY179 cycloaliphatic epoxy ester, and o-cresol novalac; $\text{BF}_3 \cdot \text{MEA}$ accelerator; and 4,4'-diaminodiphenylsulfone, amine curing agent. Inspection of the infrared spectra of the humidity exposed neat resin films show that four component peaks decrease in intensity with time and at all humidity exposures. These are the amino peaks at 3450 and 3500 cm^{-1} , the ester peak at 1720 cm^{-1} and the epoxy peak at 910 cm^{-1} . However, the infrared spectra of the humidity exposed resin films derived from prepreg show that only the amino and epoxy absorption peaks decrease in intensity (concentration) during these exposures.

This suggests that in the process of manufacturing AS/3501-6 prepreg, some chemical changes have taken place. Since the neat epoxy resin has not been subjected to a manufacturing process involving the same thermal treatment as resin for prepreg, the aging behavior of the resin is expected to be different than the prepreg. The extent to which the epoxy ester may be consumed during prepreg production, and the effect of this treatment on $\text{BF}_3 \cdot \text{MEA}$ reactivity most likely will effect its overall behavior in humidity aging studies relative to neat resin. This may account for the differences observed between neat and prepreg resin in the infrared studies.

2.6 Differential Scanning Calorimetric (DSC) Analysis of 3501-6 Epoxy Resin and AS/3501-6 Prepreg

Samples of 3501-6 epoxy resins and AS/3501-6 prepreg were exposed to humidity conditions and times mentioned for HPLC and IR studies, and then subjected to DSC analysis. For each sample DSC runs were made at heating rates of 5°C/min, 10°C/min and 20°C/min. The peak temperature of the major exotherm peak

at each heating rate was determined from the DSC scan for each sample. The data are listed in Tables 27 and 28. Typical DSC thermograms are listed in Figs. 29-31. Plots of the $\log \phi$ (heating rate 5°C/min, 10°C/min and 20°C/min) as a function of $1/T$ (K) for the major exotherm peak were prepared for several samples as shown in Figs. 32 and 33. The plots for the prepreg aged for 12 hrs at the various humidity condition (Fig. 30) do not deviate significantly from the plot for "as received" prepreg (Fig. 29). However, considerable deviation is noted for prepreg aged 336 hrs (Fig. 31). The calculated total energy evolved in the major exotherm peak, discussed below, suggests that this deviation is not significant. The DSC scan derived from heating a sample at the various rates yields a major exothermic peak which is related to A, the activation energy required for the cure reaction. The area of the exothermic peak is related to the heat of the reaction. From the plots of $\log \phi$ (heating rate) vs $1/T$, the activation energy can be calculated as follows:

$$\log \phi = -\frac{A}{2.303 RT} + B$$

where ϕ = heat up rate,
 T = temperature, K
 A = constant related to the activation energy $\approx E_a$
 B = constant, related to the Arrhenius frequency factor
 R = gas constant = 1.987.

The slope of $\log \phi$ vs $1/T$ can be expressed as follows:

$$\text{slope} = -\frac{E_a}{2.303 R}$$

The energy term E_a can be calculated:

$$E_a \text{ (Activation Energy of the cure reaction)} = -\text{slope} \times 2.303 (1.987)$$

The values of E_a were calculated for "as received" and several humidity exposed prepreps. The results for three time periods are shown in Table 29. The data suggest that the time period of two weeks in dry condition or at the various humidity conditions do not affect the activation energy of the major cure reaction.

However, inspection of the many DSC curves for the series of runs made on 3501-6 resin and AS/3501-6 prepreg reveals that the onset temperature of the minor peak in the thermogram is affected by 93% relative humidity exposure. The data for the onset temperatures of the minor peak in humidity aged prepreg is listed in Table 30. After 250 and 336 hrs of exposure in the 93% RH condition, the on-set temperature at the higher heatup rates (10 and 20°C/min), increased by 15 to 33°C. Furthermore, the minor exotherm peak has virtually disappeared as is illustrated by comparing Figs. 29 (as received prepreg) and 30 (93% RH, 12 hr prepreg) with Fig. 31 (336 hr humidity aged prepreg). These data show that the DSC can be used to detect small changes in resin composition which occur due to humidity aging, if the DSC sensitivity can be improved to follow the minor exotherm peak.

2.6.1 Reversibility Effects of Humidity Exposed 3501-6 Epoxy Resin and AS/3501-6 Prepreg

A series of 3501-6 epoxy resin and AS/3501-6 prepreg samples were subjected to 51 and 93% RH for various time periods, dried at 0% RH to an equilibrium weight (~4 days) and then subjected to DSC analysis. The results of the DSC scans are tabulated in terms of the major exotherm peak temperature experienced at the three heat rates; 5°C/min, 10°C/min and 20°C/min (Tables 31 and 32). Comparison of these data with the data in Tables 27 and 28 respectively reveals that the moisture absorption-desorption process has no detectable effect on the thermal behavior of the heat resin, or resin from prepreg. This suggests that moisture has little influence on the cure behavior of the epoxy resin.

2.7 Processing Characteristics of Humidity Aged Prepreg

2.7.1 Dielectrometric Changes in AS/3501-6 Prepreg During Processing

A series of composites were fabricated from "as received" AS/3501-6 prepreg for the long cure cycle listed in section 3.5. The results of this process study would be compared with changes which occur in the dielectrometric behavior of prepreg as a function of exposure and time. The Audrey II Model 203 Monoprobe dielectrometer was used to follow the dielectric changes in the cure with time and temperature.

In the initial stages of heat-up to 240°F, the dissipation factor remains constant for about 45 min, then undergoes a gradual increase yielding a broad peak over a 55 min period, with the broad maximum reached in 35 min, before a temperature of 240°F is reached. The temperature is maintained at 240°F for 80 to 120 min (60 min in the UTRC runs) at which point another gradual increase in dissipation factor occurs. At the end of this hold period at 240°F, pressure is increased (from 85 psi to 100 psi), a dramatic change in dissipation occurs,

followed by the development of another broad peak as the temperature is increased to 275°F, and held for 40 min. This is followed by an increase in temperature to 350°F with a gradual decrease in the dissipation factor to a low value of 0.020 as the temperature is maintained at 350°F for 120 min, 100 psi pressure.

The very broad peaks generated during the cure process are caused by long hold times at the lower temperature and the gradual increase to the higher cure temperature. This causes gradual changes in the dielectric properties of the resin. Therefore subtle changes in the chemistry of the system caused by moisture absorption and aging time cannot be detected by this long cure process. In order to detect changes due to moisture and aging, it would be necessary to shorten the cure cycle in efforts to sharpen the peaks due to abrupt changes in the dielectric properties of the resin matrix. Therefore, this technique was abandoned in favor of using a more sensitive method such as dynamic mechanical analysis or a technique measuring the rheological properties of the resin over a narrow temperature range, and/or at a fixed temperature as a function of time, but not necessarily tied to the complete cure cycle.

2.7.2 DMA Study of AS/3501-6 Prepreg

DuPont Model 981 Dynamic Mechanical Analyzer (DMA) is being used in determining the effects of moisture on the rheological properties of the AS/3501-6 prepreg, and to relate these properties to the processing behavior of the prepreg. Samples are being tested by raising the sample temperature from room temperature to 135°C at 1.5°C/min, and then holding at 135°C until a change in rheological properties are observed. Thus, this thermal treatment corresponds to the first stage in the cure cycle used to fabricate AS/3501-6 composites, prepared from humidity exposed prepreg.

Initial studies to establish the conditions required to obtain meaningful results were carried out on "as received" prepreg. After several investigative runs, it was found that meaningful data on prepreg could be obtained on prepreg samples sandwiched between aluminum foil. Changes in Young's modulus, loss modulus $\tan \delta$ and damping (Figs. 34 and 35) are being recorded as a function of the thermal treatment and time. The results show (Fig. 34) that a time period of 30 min at 135°C was required before a small change in the rheological properties (indicated by very gradual change in damping and $\tan \delta$) was observed, and that a time period of 100 min at 135°C was required before these changes became significant. Complete gelation occurred after 170 min at 135°C, which in Fig. 34 is indicated as the point of maximum damping.

A DMA run of AS/3501-6 prepreg aged at 93% RH for 5 weeks is shown in Fig. 35. Two significant differences relative to unaged sample are noted; (1) there is a damping peak within 40 min during the heat-up to 135°C, which may be related to a melt of the advancement species; and (2) the point of maximum damping occurs after 140 min at 135°C, compared to 170 min for the "as received" prepreg. The data suggest that the processing cycle must accommodate this change in gel time to prevent the production of laminates with poor mechanical properties. Additional studies are to be conducted in this area.

2.7.3 Tack and Drapage Properties of Humid Aged AS/3501-6 Prepreg

Table 33 lists the tack and drapage properties of humid aged prepreg. The data show that after three days at the lowest and highest humidity levels, there is a decrease in the tack and drapage properties of the prepreg. However, as will be shown in section 2.7, excellent composites were fabricated from prepreg aged two weeks at 0% or 93% RH.

2.8 Physical and Mechanical Properties of Composites Fabricated from Humidity Aged AS/3501-6 Prepreg

A series of unidirectional AS/3501-6 composites were fabricated from humidity exposed AS/3501-6 prepreg for the following humidity conditions: 0%, 31%, 51% and 93% RH at 25°C for time periods of 12, 24, 72, 168 and 336 hrs. Some experiments were also carried out at 100% RH at 29°C for 12, 24, 48 and 160 hrs. For the shear and transverse tensile tests, samples were cut from 10.2 cm x 10.2 cm x 0.25 cm (4" x 4" x .100") composites. For the longitudinal tensile and compression tests, samples were cut from 15.2 cm x 15.2 cm x 0.14 cm (6" x 6" x 0.055") composites. Tests were carried out at room temperature and 122°C for the "as fabricated" composites and for composites exposed to a humidity condition of 87% RH, 160°F (71°C) to saturation.

2.8.1 Physical Properties

The 4" x 4" x 0.100" composite densities, thicknesses and mils/ply thicknesses are listed in Table 34. The 6" x 6" x 0.055" composites are listed in Table 35.

2.8.2 Void Content of Composites

Photomicrographs of longitudinal cross sections of the 4" x 4" x 0.100" composites were taken. A few representative micrographs are shown in Figs. 36 and 37. Table 36 lists the observations from photomicrographs of all composites tested.

From the data in Table 36, 3 points can be made; (1) most composites are void free, (2) there is no correlation between relative humidity exposure of prepreg and void content, (3) long aging times of prepreg at dry (0%) or wet (93% RH) conditions have no detrimental effect on composite quality; that is, void free composite can be produced under all prepreg exposures.

2.8.3 Interlaminar Shear Strength

The room temperature and 122°C shear strengths of the "as fabricated" and humidity exposed (wet) are listed in Tables 37 and 38 respectively. The data are presented graphically in Figs. 38-41. Inspection of the data in the figures suggests several trends: (1) For the first 72 hr exposure at all humidity conditions, there is no apparent effect on RT "as fabricated" shear strength. Beyond this time period, particularly for the 2 weeks exposure prepreg exposed to 0% RH yielded composites with poorer shear strength than prepreg exposed to 93% RH. (2) The RT wet strength data appears to show prepreg exposure effects to a greater extent than the RT dry strength data. For time periods up to 1 week, prepreg exposed to all conditions (0% RH, 31, 93 and 100% RH) yielded composites with at least equivalent or superior shear strength than "as received" prepreg. These exposures, therefore, show no adverse effects in finished composites. However, for 2 week exposure of prepreg the more humid conditions yielded composites with superior shear strength than the dry (0% RH) condition. (3) Inspection of the dry and wet strength data for composites tested at 122°C shows similar trends as shown by the RT shear strength data. (4) The data suggests that prepreg can be stored at 25°C for at least a week with no detrimental effect to composite shear strength and further suggests that for exposure times up to 2 weeks, high humidity exposure improves the shear strength, while a dry condition is detrimental to shear strength.

The shear strength results appear to be in conflict with HPLC data. These data show that the highest humidity exposure yields a greater concentration of the advancement species in the prepreg, and it is expected that these species would decrease flow and cause premature gelation of the resin system. However, since the data suggests that the presence of advancement species actually improves composite shear strength and these species are also present in prepreg aged for 2 weeks at 0% RH, moisture must be influencing the flow during processing to counteract reduced flow that is expected of the advanced resin. Apparently, moisture is absorbed by the resin, and behaves as a plasticizer. This will decrease the glass transition temperature of the prepreg containing these advanced resin species and help the flow as the temperature is increased in the process. Prepreg aged at 0% RH for 2 weeks is boardy in nature, and apparently does not possess the flow properties of the moisture containing prepreg.

2.8.4 Transverse Tensile Properties

Transverse tensile data for AS/3501-6 composites fabricated from humidity aged prepreg is listed in Tables 39 and 40. Considerable difficulty was encountered in fabricating these specimens. The first difficulty was that in bonding the doublers to both ends of the composite in a press at low pressure (35 psi), several composites cracked. This was circumvented by bonding the doublers in the free state using spring loaded clamps on both ends to hold the doubler in place for bonding. The other difficulty was that many specimens broke in the cutting process. Some specimens broke in handling.

The fragile nature of these specimens therefore prevented the testing of a full compliment of specimens as was planned.

The room temperature and 122°C transverse tensile data for dry and wet specimens is listed in Tables 39 and 40. Inspection of the tables for the RT dry and wet tensile data shows that there is no apparent effect of humid aging of AS/3501-6 prepreg on the transverse tensile properties of AS/3501-6 composites. The same conclusions can be arrived from the 122°C dry and wet data. The transverse tensile specimen are very fragile, and some of the scatter in the data can be ascribed to precracked specimens, as is indicated by comparing RT and 122°C dry strength of composite 66 with the RT and 122°C wet strength of this composite. In both cases the dry RT and 122°C strength properties are much lower than the wet RT and 122°C strengths. This is unexpected as the data in Tables 39 and 40 show. Therefore this section of the composite used to test the dry RT and 122°C strength properties most likely contained cracks, thereby yielding lower test data.

The fact that the transverse tensile data did not reveal a similar trend as the shear data even though both tests are matrix dominated properties suggests that the transverse test is too flaw sensitive to be used as a useful test to follow humidity aging effects.

2.8.5 Longitudinal Tensile Properties

The tensile properties of composites fabricated from humidity aged AS/3501-6 prepreg are listed in Tables 41-44. Since the tensile properties are fiber controlled, it is not expected that the tensile properties would be affected to any great extent by these humid aging experiments, unless the humid aging caused very poor bonding and created high void composites. It is apparent that neither of the two latter conditions were present in these composites, as is shown by inspection of the data in Tables 41-44. The RT dry and wet tensile properties, and the 122°C cry and wet tensile properties show no trends in either improvement or degradation of the tensile properties due to humidity aging of the AS/3501-6 prepreg.

2.8.6 Compressive Properties

Tables 45 and 46 list the dry RT and 122°C compressive properties of composites fabricated from humidity aged AS/3501-6 prepreg. The compressive strength and modulus data show that humid aging of prepreg under the conditions listed has no apparent effect on this mechanical property.

2.8.7 Conclusions on Effects of Humidity Aging on Mechanical Properties

The results of the mechanical tests show that humidity aging of AS/3501-6 prepreg up to 2 weeks has no detrimental effect on the mechanical properties of composites fabricated from the humid aged prepreg. In fact, the shear strength data suggest that aging of the prepreg at the high humidity levels 51-93% RH over 2 week time periods causes an improvement in the interlaminar shear strength relative to "as received" and dry 0% and 31% aged prepreg.

III. EXPERIMENTAL METHODS AND PROCEDURES

3.1 Materials

The resin and prepreg materials investigated in this program were Hercules 3501-6 epoxy resin and Hercules AS1/3501-6 graphite epoxy prepreg. The epoxy resin 3501-6 batch 135AS was manufactured by Hercules Inc. on Jan. 27, 1981 and received at United Technologies Research Center on Feb. 13, 1981. The AS1/3501-6 graphite epoxy prepreg was manufactured Feb. 11, 1981 and received at United Technologies Research Center Apr. 6, 1981. The Hercules Quality Assurance Certification and final inspection reports are shown in Tables 47 and 48.

3.2 Humidity Aging Conditions for Prepreg

AS/3501-6 prepreg and 3501-6 epoxy resin were aged in a controlled temperature (25°C) at four humidity levels: 0, 31, 51 and 93%. The materials were placed in stainless steel desiccators 31 cm (H) x 32 cm (W) x 31 cm (D) with fixed wire shelf supports every 13 mm and with 4.8 mm glass on both sides and door. The humidity levels were controlled as follows: 0% RH (dry condition) - indicating drierite (anhydrous CaSO_4); 31% RH - saturated solution of hydrated calcium chloride $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$; 51% RH - saturated solution of hydrated calcium nitrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$; 93% RH - saturated solution of ammonium hydrogen phosphate $\text{NH}_4\text{H}_2\text{PO}_4$. Some experiments were carried out at room temperature under vacuum for several days and at 29°C, 100% RH for up to 2 weeks in a humidity chamber.

3.3 Humidity Exposure of AS/3501-6 Laminates

The fabricated laminates were tested in two states of condition, dry and wet. In the "as fabricated" ambient condition (dry), the moisture content of composites varied from 0.1 to 0.3%, depending on ambient conditions. The wet condition consisted of exposure of composites to a relative humidity of 87% at 160°F (71°C) until saturation was achieved. This usually required approximately seven days.

3.4 Physiochemical Methods

3.4.1 Differential Scanning Calorimetry (DSC)

Thermal analysis of 3501-6 epoxy resin and AS/3501-6 prepreg samples were carried out on a DuPont 990 thermal analysis unit equipped with a differential scanning calorimeter (DSC) unit. Sample sizes ranged from 5 to 6.0 mg for resin samples and 13 to 17.0 mg for prepreg samples. DSC scans were run at three rates for each sample: 5°C/min, 10°C/min and 20°C/min. Other conditions used in runs are shown on a thermogram (Fig. 29), which is an example of the many DSC's run on this program.

3.4.2 Infrared Spectroscopy

Initial studies on resin and prepreg resin were carried out on a Perkin-Elmer 298 infrared spectrophotometer. This is a double beam continuous recorder instrument which records infrared transmittance as a function of frequency (expressed in wave number) over a frequency range $2.5\mu - 40\mu$ ($4000-600\text{ cm}^{-1}$ wave number). Films of the neat 3501-6 epoxy resin or 3501-6 epoxy resin from prepreg on sodium chloride crystals or zinc sulfide were prepared from a tetrahydrofuran solution of the resin by evaporation of the solvent at room temperature, followed by vacuum treatment at room temperature for 2 hrs. Epoxy resin was removed from AS/3501-6 prepreg by dissolving the resin in tetrahydrofuran and filtering the solution through a millipore filter.

3.4.3 Dielectrometry

An Audrey II Model 203 Monoprobe Dielectrometer was used to monitor and measure the dielectric changes which occurred on curing the 3501-6 epoxy. Dielectric analysis is a method of following the changes which occur in the cure of the resin, and the information generated can be used to determine when pressure should be applied to a laminate to produce a void-free specimen. The apparatus has a frequency range between 100 and 1000 Hz. The Audrey II operates on the principle of electrical impedance; heat is an ac voltage (V) is impressed upon a sample, the current (I) that flows through the sample is measured, and the impedance is the ratio V/I . It can be shown that $e = I_C/2fV$, where I_C is the capacitive component of the current through the sample and F is the variable frequency. The dissipation factor is defined as $D = 1/2 \text{ FRC} = I_R/I_C$, where I_R is the resistive component of current through the sample. Then what one measures in using Audrey II, is the dissipation factor D_X as a function of temperature, or D_X as a function of time at a given temperature.

3.4.4 Dynamic Mechanical Analysis

A DuPont 981 Dynamic Mechanical Analyzer (DMA) attached to the DuPont 1090 Thermal Analyzer was used to measure dynamic rheological properties of prepreg. Prepreg samples were sandwiched between aluminum foil and the damping response as a function of time, at a specific temperature was recorded.

3.4.5 Thermal Mechanical Analyses

The response of the prepreg to a mechanical load while heating it at a specific rate was measured using a DuPont 973 Thermomechanical Analyzer (TMA), attached to the DuPont 1090 thermal analyzer. To make these measurements, samples were sandwiched between aluminum foil. This prevented adhesion of the resin to the mechanical probe used in the test.

The composites fabricated by this procedure are listed in Tables 33 and 34. The majority of composites were fabricated by the cure cycle listed above. A series of laminates (Table 49) were fabricated from "as received" prepreg using the same lay-up procedure shown in Fig. 42, but the cure cycle differed in that pressure was applied at room temperature. The cure cycle employed for these laminates was as follows:

1. Vacuum bag 19 or 20 ply unidirectional lay-up.
2. Maintain 20" of mercury (minimum) vacuum and apply 85 ± 5 psi autoclave pressure.
3. Raise laminate temperature to $115^{\circ}\text{C} \pm 5^{\circ}\text{C}$ in 80 to 120 min (1.5 to $3^{\circ}\text{C}/\text{min}$).
4. Hold at 115°C for 90 to 120 min while maintaining 20" of mercury (minimum) and 85 ± 5 psi autoclave pressure.
5. After completion of 90 min hold, at $115 \pm 5^{\circ}\text{C}$, raise temperature to $135^{\circ}\text{C} \pm 15^{\circ}\text{C}$ in 5 to 15 min (1.2 to $3.6^{\circ}\text{C}/\text{min}$). At the same time increase autoclave pressure to 100 ± 5 psi, and vent the vacuum line to the atmosphere.
6. Hold at $135^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 30 to 45 min, 100 ± 5 psi autoclave pressure.
7. Then raise temperature to $177^{\circ}\text{C} \pm 5^{\circ}\text{C}$ in 15 to 40 min (1.3 to $3.5^{\circ}\text{C}/\text{min}$), and hold at $177^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 120 to 140 min maintaining 100 ± 5 psi autoclave pressure.
8. Cool to 93°C in not less than 30 min under at least 7.5 psi pressure.
9. Postcure in an oven at 177°C for 8 hrs.

The objective of these runs was to determine if the Audrey II Dielectrometer could monitor small changes in processing behavior. After making several runs, it was concluded the long processing time involved in this cure cycle prevented the detection of minor dielectric changes which may occur due to aging. Therefore this approach and cycle was abandoned in favor of a shorter cure cycle and use of other techniques to follow changes in process due to humid aging.

3.4.6 High Pressure Liquid Chromatographic (HPLC) Analysis

HPLC analysis of 3501-6 neat resin and 3501-6 resin removed from pre-preg was made on a Waters ALC/GPC 244 chromatogram equipped with WIIP (Waters Intelligent Information Processes) modulus (Model 720 Systems Controller and Model 730 (data module), an automated M-710B WISP (Waters Intelligent Sample Processor) sample processor and a Model 440 UV/VIS tunable spectrophotometer. The final conditions used in following changes in neat 3501-6 and 3501-6 resin dissolved from prepreg are listed as follows:

Column: μ Bondapak C₁₈ 4 mm ID x 30 cm length
Flow Rate: 1.2 ml/min (constant)
Solvent System (mobile phase): Tetrahydrofuran (THF)/water
Gradient: linear gradient 20/80 to 60/40
THF/H₂O over 45 min
Detector: Model 440, U.V., 254 nm at 0.5 or 1.0 AUFS
Sample Size: 15 or 20 μ l of resin solution (THF)
Sample Conc: 0.84 to 1.28 μ g/ μ l
Instrument: Waters ALC/GPC 244 equipped with WIIP modules
(Model 720 systems controller and Model 730 data module)

Several standard solutions were prepared for HPLC analysis by dissolving the quantities of materials listed in Table 19 in 100 ml tetrahydrofuran using a volumetric flask. A 5 ml aliquot of the solution was further diluted to 25 ml in a volumetric flask. For HPLC analysis, a 15 μ l portion was used.

In the initial stages of this study, an internal standard was used to determine quantitatively the amount of DDS, MY720 and advancement peak as a function of the humidity aging conditions. The model 720 system controller and Model 730 data module system were integrated into the HPLC equipment; it was possible to determine the concentration of these components in terms of the areas of each peak. The WIIP system (Waters Intelligent Information Processes) is programmed to calculate the percent of each component eluting from the column based on areas. Therefore, for neat resin 3501-6 and AS/3501-6 HPLC studies, it was not necessary to use internal standard in each solution.

The procedure used for analysis of the 3501-6 resin samples was as follows: approximately 0.64g of 3501-6 resin ("as received" or humidity aged sample) was dissolved in tetrahydrofuran in a volumetric flask to a volume of 100 ml. A 5 ml aliquot of this sample was further diluted in a volumetric flask to a volume of 25 ml. A 15 μ l sample was injected into the column for analysis. The approximate concentration of resin solution used in the HPLC analysis was 1.28×10^{-3} g/ml. A 15 μ l sample contains about 19.2 mg of resin.

The procedure used for analysis of AS/3501-6 prepreg was as follows: approximately 1.5g (3" x 3") of AS/3501-6 prepreg ("as received" or humidity aged) was treated with about 30 ml of tetrahydrofuran (THF) to dissolve the resin. The solution was filtered through a 0.45 μ pore millipore membrane. The fiber was washed with 3 x 15 ml portions of THF. These washings were also filtered, as above in the same setup. The combined filtered solutions were transferred to a 100 ml volumetric flask using proper wash procedure to transfer all resin to the flask, and were diluted with THF to a volume of 100 ml. A 5 ml aliquot portion was transferred to a 25 ml volumetric flask and diluted with THF to a volume of 25 ml. A 20 μ l sample was used in the HPLC analysis. The approximate concentration of resin removed from the prepreg and analyzed as follows:

$$\begin{aligned}
 1.5\text{g prepreg} \times .28 \text{ (fraction of resin on prepreg)} &= 0.42\text{g} \\
 0.42\text{g}/100 \text{ ml} &= 0.0042\text{g/ml} \\
 0.0042\text{g/ml} \times 5 \text{ ml} &= 0.021\text{g} \\
 0.021\text{g}/25 \text{ ml} &= 0.00084\text{g/ml} = 0.84 \text{ g}/\mu\text{l}
 \end{aligned}$$

A 20 μ l sample contains about 16.8 μ g resin.

3.5 Fabrication of Composite Test Laminates

Two sizes of laminates were fabricated: 10.2 cm x 10.2 cm x 19 ply (~0.0254 cm, 0.100 in.) and 15.24 cm x 15.24 cm x 10 ply (~0.14 cm, ~0.055 in.). The ply lay-up and bleeder plies and release films used in the vacuum-bag-pressure fabrication process is shown in Fig. 42. The cure cycle used in the fabrication process was as follows:

1. vacuum bag 10 or 19 ply unidirectional lay-up
2. maintain 20 in. of mercury (minimum)
3. raise laminate temperature to 135°C (275°F), 1-2°C/min (2-4°F/min) (50-70 min)
4. hold 135°C 0.5 hr, apply 85 psi, then maintain these conditions for an additional 0.5 hr, then vent line
5. raise temperature to 178°C (350°F), 1.5-2.6°C/min, (3-5°F/min), 85 psi (15-25 min)
6. hold 178°C, 120 min, 85 psi
7. cool to 100°C, release pressure
8. postcure in an oven at 178°C for 4 hrs, free state.

3.6 Fabrication of Test Specimens

Short beam interlaminar short beam shear specimens, nominally 0.65 cm wide x 1.2 cm long x 0.25 cm thick were cut from the 10.2 cm x 10.2 cm x .25 cm (4.0" x 4.0" x .100") laminate specimens.

The transverse tensile specimens, nominally 0.64 cm wide x 10 cm long x 0.25 cm thick (1/4" x 4.0" x 0.100"), were also cut from the same laminate. Glass/epoxy doublers 0.32 cm thick (0.125") were bonded using Hysol EA 9649R film adhesive onto the laminate before the transverse tensile specimens were cut. The doublers were bonded to the laminate one end at a time by clamping the doubler and adhesive film in position, and allowing the other end to remain in the free state. The cure cycle used for bonding the doublers to the composite was to place the specimen in the preheated oven at 350°F, apply 10-20 psi pressure (by clamping), allow to cure at 350°F for 2 hrs. Attempts to bond doublers to both ends of the laminate simultaneously using a press resulted in cracked laminates. A 1" gage section was provided for each specimen.

Longitudinal tensile and compression specimens were fabricated from the 15.2 cm x 15.2 cm x 0.14 cm (6" x 6" x 0.055") laminates. Since the Celanese compression test specification calls for stainless steel doublers, these were bonded onto the laminate in one bonding operation to produce both tensile specimens and compression specimens with stainless steel doublers. The tensile specimens were nominally 15.2 cm long x 1.27 cm wide x 0.14 cm thick (6" x 0.5" x 0.055") with a 5.0 cm (2") gage section. The compression specimens were nominally 6.4 cm wide x 14.0 cm long x 14 cm thick with a gage section of 0.127 cm (0.5"), according to the ASTM D-3410-70 procedure. Tensile and compression specimens were cut using a diamond cut-off wheel.

3.7 Testing Procedures

The ASTM D-2344 procedure was used for the interlaminar short beam shear tests. The span-to-depth ratio used was 4/1 and the test was run at a crosshead speed of 0.127 cm/min.

The compression test was run according to ASTM procedure D3410-75, at a crosshead speed of 0.127 cm/min (0.05 in/min). The longitudinal tensile specimens were 15.2 cm in length by 1.27 cm in width by 0.14 cm in thickness. The gage section dimension was 5.0 cm and the tab sections were also 5.0 cm in length. The tests were run at a crosshead speed of 0.127 cm/min. The transverse 90° tensile tests were run on specimens 0.25 cm thick x 0.64 cm wide x 10 cm long, with a 2.54 cm gage length. The specimens were protected with tabs at each end, each 3.81 cm in length.

IV. CONCLUSIONS

Humid aging of AS/3501-6 prepreg at 25°C at humidity levels of 0, 31, 51 and 93% RH causes an increase in the concentration of advanced resin species and the concentration increased with humidity level. This was observed by HPLC analyses, and to a lesser extent in the infrared spectroscopic analysis. The Differential Scanning Calorimetric (DSC) technique was not sufficiently sensitive to detect changes in the heat of the major exotherm reaction due to the advanced resin species. There are indications that DSC may be used to detect changes in the on-set exotherm temperature of the minor exotherm peak due to an increase in the concentration of advanced resin species. The Audrey II dielectrometric techniques were also not sufficiently sensitive to detect changes in the processing due to the presence of these advanced resin species. There are indications that dynamic mechanical analyses (DMA) may be sensitive to detect changes in viscoelastic properties of the prepreg due to changes in resin composition caused by humid aging.

The mechanical properties (interlaminar shear, transverse tensile, longitudinal tensile and compressive properties) of composites fabricated from humid aged AS/3501-6 prepreg are equivalent to the properties of composites fabricated from unaged "as received" prepreg. In fact, there are strong indications that the interlaminar shear strengths are improved due to aging for 1 to 2 weeks at the higher humidity levels (51 and 93% RH), contrary to what might be expected. The DSC studies suggest that the curing process time at the cure cycle temperatures used may be reduced by these advanced resin species. This requires further investigation for verification.

REFERENCES

1. H. Borstell, "Chemical Composition and Processing Specifications for Air Force Advanced Composite Matrix Materials", Technical Report AFML-TR-79-4166, Nov. 1979
2. J. F. Carpenter, "Test Program Evaluation of 2501-6 Resin", Report No. N00019-77-C-0155, May 1978.

Table 1

Moisture and Volatile Content of 3501-6 Epoxy
Resin and AS/3501-6 Prepreg

Testing Dates		Time in Full Vac. hrs	Temp. °C	Wt% Volatile + Moisture	
In	Out			3501-6 Resin	AS/3501-6 Prepreg ³
4/24/81	4/24/81	5 min	RT	+0.15 ¹	-
4/22/81	4/23/81	24	RT	+0.080 ¹	-
4/22/81	4/29/81	163	RT	+0.59 ¹	-
3/17/82	3/17/82	5 min	48	0.045 ²	0.02
3/17/82	3/18/82	24	48	0.082 ²	0.20
3/17/82	3/19/82	48	48	.092 ²	0.24
3/17/82	3/23/82	144	48	0.25	0.35
3/17/82	3/24/82	168	48	0.25	0.40

¹ sample weight ~0.64g

² sample weight ~3.0g

³ sample weight ~0.7g

Table 2

Moisture Desorption-Absorption Behavior
of 3501-6 Epoxy Resin¹

Testing Dates		Time hrs	Wt% Change ¹				Conc. H ₂ SO ₄ ⁸
In	Out		Air ⁴	Drierite ⁵	Drierite ⁶	P ₂ O ₅ ⁷	
3/15/82 ²	3/16/82	24		-.064	-.035	-.070	-.045
3/17	3/18	24	-.027	-	-	-	-
3/15	3/18	24	-	-	-.035	-.076	-.046
3/15	3/22	168	-.014	-.089	-.042	-.10	-.080
3/15	3/23	192	-.007	-.105	-.093	-.067	-.048
3/15	3/24	216	-.014	-.089	-.042	-	-.077
3/15	3/25	240	+0.003	-.089	-.042	-	-.070
3/15	3/26	264	-.089	-.089	-.005	-	-.084
3/23 ³	3/24	24	+0.020	+0.073	-	-.097	-.030
3/23	3/25	48	+0.047	+0.086	-	-.083	-.097
3/23	3/26	72	+0.077	+0.069	-	-.10	-.023

¹ resin weight ~2.7g² first series of tests³ second series of tests⁴ ~28% RH⁵ new batch⁶ older batch⁷ 0% RH⁸ 0% RH

Table 3

Moisture Desorption-Absorption Behavior
of AS/3501-6 Prepreg¹
(single ply)

Testing Dates		Time hrs	Air ⁴	Drierite ⁵	Drierite ⁶	P ₂ O ₅ ⁷	Conc. H ₂ SO ₄ ⁸
In	Out						
3/15/82 ²	3/16/82	24		-.123	+1.69	+0.72	+0.12
3/17	3/18	24	-.044	-	-	-	-
3/15	3/18	72		-	+1.68	+0.73	+0.13
3/15	3/22	168	-.041	-.123	+1.69	+0.72	+0.12
3/15	3/23	192	-.063	-.105	+1.70	+0.73	+0.13
3/15	3/24	206	-.089	-.123	+1.70	-	-
3/15	3/25	230	-.033	-.100	+1.73	-	-
3/15	3/26	254	-.02	-.120	1.70	-	-
3/23 ³	3/24	24	-.045	-.107	-	-.144	-.140
3/23	3/25	48	.00	-.067	-	-.013	-.099
3/23	3/26	72	-.003	-.103	-	-.140	-.139

¹ sample weight ~2.7g

² first series of tests

³ second series of tests

⁴ ~28% RH

⁵ new batch

⁶ older batch

⁷ 0% RH

⁸ 0% RH

Table 4

Wt% Change of 3501-6 Epoxy Resin Due to
Humidity Exposure at 25°C¹

Exposure Time hrs	Sample		Wt% Change																															
			0% RH Dry			31% RH			51% RH			93% RH																						
			In	Out	Sample	1	2	3	1	2	3	1	2	3																				
24	3/23/81	3/24	-0.20, -0.13, 0	-0.003, -0.70, +0.06	+0.20, +0.19, +0.06	+0.36, +0.32, +0.29	48	3/23	3/25	-0.19, -0.13, 0	+0.036, -0.68, +0.10	+0.27, +0.26, +0.15	+0.49, +0.46, +0.44	72	3/23	3/26	-0.13, 0	-0.66, +0.10	+0.30, +0.17	+0.56, +0.53	163	3/23	3/30	-0.10, +0.03	-0.58, +0.18	+0.45, +0.32	+0.86, +0.82	336	3/23	4/6/81	-0.13, 0	-0.52, +0.26	+0.61, +0.52	+1.45, +1.37

¹ samples tested 3/23 to 4/6/81 (3.0g sample)

Table 5

Wt % Change of 3501-6 Epoxy Resin Due to
Humidity Exposure at 25°C¹

(each sample then subjected to HPLC analysis)

Exposure Time hrs	Sample		% Change			
	In	Out	0% RH	31% RH	51% RH	93% RH
12	4/6/81	4/6	-.17	-.89	-.34	-.59
24	4/13	4/14	-.31	-.09	-.06	+ .46
48	4/13	4/15	-.99	-1.03	-.13	+ .22
72	4/3	4/6	-.13	+ .15	+ .29	+ .59
168	4/6	4/13	-.47	-.88	-.23	+ .03
250	4/9	4/20	-.20	0.00	+ .36	+ .31
336	4/7	4/21/81	-.34	-.02	+ .03	+ .40

¹ samples tested 4/6 to 4/20/81 (0.64g sample)

Table 6

Wt % Change of 3501-6 Epoxy Resin Due to
Humidity Exposure at 25°C¹

Exposure Time hrs	Sample		51% RH Wt % Change			Ave.
	In	Out	1	2	3	
12	5/12/81	5/13	-0.11	-0.14	-.21	-.15
24	5/12	5/14	+0.21	-.12	-.18	-.17
48	5/12	5/16	+0.17	-.10	-.14	-.14
72	5/12	5/17	-.16	-.10	-.13	-.13
168	5/12	5/19	-.01	-.05	-.03	-.03
336	5/12	5/26/81	+1.13	+1.18	+1.13	+1.15

¹0.7g samples used

Table 7

Moisture Absorption Behavior of 3501-6 Epoxy
Resin at 25°C (~3.0g sample)

Aging Time	Sample		Wt% Moisture Absorbed			
	In	Out	0% RH	31% RH	51% RH	93% RH
12	9/14/81	9/15	+0.52	+0.73	+0.52	+0.75
24	9/14	9/15	+0.54	+0.74	+0.57	+0.80
48	9/14	9/16	+0.54	+0.78	+0.62	+0.52
72	9/14	9/17	+0.49	+0.72	+0.62	+0.43
168	9/14	9/21	+0.44	+0.72	+0.66	+0.63
216	9/14	9/24	+0.47	+0.62	-	+0.80
336	9/14	9/27/81	+0.52	+0.89	+0.71	+0.90

Table 8

Wt % Change of AS/3501-1 Prepreg Due to
Humidity Exposure at 25°C¹
(samples used in HPLC studies)
(single ply)

Exposure Time hrs	Sample		Wt % Change			
	In	Out	0% RH	31% RH	51% RH	93% RH
12	5/5/81	5/6	-0.67	+0.05	+0.67	+4.1
24	5/6	5/7	-1.81	-.79	+0.04	+2.27
48	5/6	5/8	-2.23	-.81	-.20	+3.2
72	5/11	5/14	-2.84	-1.19	-.85	+1.81
168	5/8	5/15	-2.32	-.68	+0.21	+3.30
250	5/11	5/20	-3.05	-1.36	-.91	+2.50
336	5/7	5/20/81	-2.81	-	-.30	+2.07

¹ 1.69g samples used

Table 9

Wt % Change of AS/3501-6 prepreg Due to
Humidity Exposure at 25°C¹
(samples used in DSC studies)
(single ply)

Exposure Time hrs	Sample		Wt % Change			
	In	Out	0% RH	31% RH	51% RH	93% RH
12	5/5/81	5/6	-1.7	-2.19	+2.83	+3.23
24	5/11	5/12	-1.8	-1.36	-.56	+1.71
48	5/6	5/8	-1.6	-.60	-.05	+1.98
72	6/2	6/5	-	-	-.61	+2.10
168	5/7	5/14	-	-.52	-.24	+2.06
168	5/21	6/3	-2.6	-	-	-
336	5/12	5/26/81	-2.97	-	-	-

¹ 0.4 to 0.8g samples used

Table 10

Moisture Absorption of AS/3501-6 Prepreg
 (~1.4g sample - single ply)

Exposure Time hrs	Sample		<u>0% RH</u>	<u>31% RH</u>	<u>51% RH</u>	<u>93% RH</u>
	<u>In</u>	<u>Out</u>				
12	9/14/81	9/15	+ .99	1.17	1.96	1.78
24	9/14	9/15	1.19	1.26	1.95	1.86
48	9/14	9/16	1.23	1.23	1.98	1.99
72	9/14	9/17	1.14	1.09	1.70	1.70
168	9/14	9/21	.99	1.03	1.50	1.78
216	9/14	9/24	-	-	1.8	1.83
336	9/14	9/27	1.15	1.19		1.88
720	9/14	10/14/81	-	-	-	2.06

Table 11

Moisture Absorption Characteristics
 AS/3501-6 Prepreg
 (1" diameter disks x 10 plies)
 (sample weights ~1.3g)

Exposure Time hrs	Sample		0% RH	31% RH	51% RH	93% RH
	In	Out				
12	8/13/81	8/14	-.23	-.06	+.02	+.25
24	8/13	8/14	-.17	-.05	+.02	+.27
48	8/14	8/16	-.28	-.10	-.02	+.27
72	8/13	8/17	-.28	-.20	-.05	+.25
168	8/13	8/21	-.26	-.12	+.03	+.30
336	8/14	8/28/81	-.20	-.02	+.20	+.60

Table 12

Moisture Absorption Characteristics of AS/3501-6 Prepreg
 (2.36 cm x 0.64 cm x 10 plies)
 (sample weights ~1.00g)

Exposure Time hrs	Sample		0% RH	31% RH	51% RH	93% RH
	In	Out				
12	8/20/81	8/20	-.05	+.02	+.05	+.15
24	8/20	8/21	-.09	+.01	+.05	+.24
48	8/14	8/16	-.10	-.01	+.07	+.26
72	8/13	8/17	-.13	-.04	+.02	+.22
168	8/14	8/21	-.15	+.02	+.09	+.43
336	8/14	8/28/81	-.10	+.023	.23	0.65

Table 13

Summary of Desorption-Absorption Characteristics
of 3501-6 Epoxy Resin Measured on Different Months

Exposure Time hrs	Sample		Wt% Change			
	In	Out	0% RH	31% RH	51% RH	93% RH
24	3/23/81	3/24	-0.10 ¹	-.25 ¹	+.15 ¹	+.32 ¹
48	3/23	3/25	-0.10 ¹	-.27 ¹	+.23 ¹	+.46 ¹
24	4/13	4/14	-0.31 ²	-.09 ²	-.06 ²	+.46 ²
48	4/13	4/16	-.99 ²	-1.03 ²	+.13 ²	+.22 ²
24	5/12	5/13	-	-	-.18 ¹	-
48	5/12	5/14	-	-	-.14 ¹	-
24	9/14	9/15	+.54 ¹	+.74 ¹	+.57 ¹	0.70 ¹
48	9/14	9/16/81	+.54 ¹	+.78 ¹	+.62 ¹	+.52

¹ same sample for each exposure time

² different samples for each time exposure

Table 14

Summary of Desorption-Absorption Characteristics of
AS/3501-6 Prepreg Measured on Different Months

Exposure Time hrs	Sample		Wt% Change			
	In	Out	0% RH	31% RH	51% RH	93% RH
24	5/6/81	5/7	-1.81 ¹	-.79 ¹	+.04 ¹	+2.27 ¹
48	5/6	5/8	-2.23 ¹	-.81 ¹	-.20 ¹	+3.20 ¹
24	5/11	5/12	-1.80 ¹	-1.36 ¹	-.56 ¹	+1.71 ¹
48	5/6	5/8	-1.60 ¹	-0.60 ¹	-.05 ¹	+1.98 ¹
24	8/13	8/14	-0.17 ²	-.05 ²	+.02 ²	+.27 ²
48	8/14	8/16	-.28 ²	-.10 ²	-.02 ²	+.27 ²
24	8/20	8/21	-.09 ²	+.01 ²	+.05 ²	+.24 ²
48	8/14	8/16	-.10 ²	-.01 ²	+.07 ²	+.26 ²
24	9/14	9/15	+1.19 ³	+1.26 ³	+1.95 ³	+1.86 ³
48	9/14	9/16/81	+1.23 ³	+1.23 ³	+1.98 ³	+1.99 ³

¹ single ply, different samples for each exposure time

² 10 ply, different samples for each exposure time

³ single ply, same specimen for each exposure time

Table 15

Absorption-Desorption Studies of 3501-6 Resin¹ and
AS/3501-6 Prepreg¹ Reversibility Studies at 25°C
(sample used in HPLC)

Solution # ^{2,3}	Exposure Time hrs	Sample		% RH	Wt % Change	Dried at 0% RH to Equil. Wt% from Orig.
		In	Out			
78	12	8/12/81	8/13	51	-0.14	-0.73
80	24	8/12	8/14	51	-0.76	-0.91
82	72	8/12	8/15	51	-0.15	-0.47
79	12	8/12	8/13	93	-0.20	-0.58
81	24	8/12	8/14	93	-	-
83	72	8/12	8/15	93	+3.32	-0.88
70	12	6/5	6/6	51	-	-3.7
72	24	6/5	6/7	51	-1.03	-4.2
74	72	6/5	6/8	51	-0.28	-3.5
76	168	6/5	6/19	51	-1.1	-
71	12	6/5	6/6	93	-	-4.2
73	24	6/5	6/7	93	+2.80	-4.0
75	72	6/5	6/8	93	+4.16	-3.8
77	168	6/5	6/19/81	93	+3.70	-

¹ 1.6g samples of resins were used and ~1.5g of prepreg samples were used.

² resin samples were used to prepare solutions 78 thru 83 for HPL analyses

³ resin from prepreg samples were used for preparing solutions 70 thru 77
for HPLC analyses

Table 16

Wt% Change of $\text{BF}_3 \cdot \text{MEA}$ Due to Humidity Exposures at 25°C
(sample weight ~5.0g)

Exposure Time hrs	Sample		Wt% Change			
	In	Out	12% RH (Dry)	31% RH	51% RH	93% RH
65	3/19	3/27	-0.58	+9.48	liquefied	liquefied
90	3/19	3/24	-0.64	+9.40	↓	↓
142	3/19	3/25	-0.57	+9.68	↓	↓
262	3/19	3/30	-0.63	+9.46	↓	↓

Table 17

Wt% Change of $\text{BF}_3 \cdot \text{MEA}$ Accelerator¹ Due to
Humidity Exposures at 25°C

Exposure Time hrs	Sample		Wt% Change			
	In	Out	0% RH	31% RH	51% RH	93% RH
12	5/12	5/13	0.05	4.7	9.9	19.2
24	5/12	5/14	-.36	7.3	14.3	34.0
48	5/12	5/16	-.51	7.9	20.4	54.3
72	5/12	5/17	-.40	8.2	27.0	69.8
168	5/12	5/19	-.58	7.7	33.2	106.6
336	5/12	5/26	-.15	8.0	31.0	138.7

¹ 3.0g samples used

Table 18

HPLC Analysis of Standard Solutions of 3501-6 Epoxy Resin

Component Ratio	Ratio of Peaks	
	"as received" Resin	Advanced Resin
DDS/MY720 (major)	0.56	0.49
DDS/Advancement	5.12	8.15 ¹ 2.14 ² 2.19 ³
MY720/Advancement	9.16	16.5 ¹ 4.3 ² 3.44 ³

- ¹ ratio for advancement peak emerging in 10.60 min
² ratio for advancement peak emerging in 11.17 min
³ ratio for combined peaks at 10.60 and 11.17 min

Table 19

Composition of Solutions Used in HPLC Analyses

<u>Material</u>	<u>Quantity, g</u>	<u>THF Dilution Volume (Sol. A) ml</u>	<u>Dilution Vol. of 5 ml Sol. A, ml</u>	<u>Solution No.</u>
3501-6 "as received"	0.64	100	25	7
3501-6 humid aged	0.64	100	25	11 thru 41 78 thru 83
3501-6 + benzanilide (internal standard)	0.64 to 0.30	100	25	8
advanced ¹ 3501-6	0.64	100	25	9
advanced ¹ 3501-6 + benzanilide (internal standard)	0.64 + 0.30	100	25	10
AS/3501-6 prepreg "as received" and humid aged	1.5	100	25	42 thru 77

¹ partially advanced by heating at 100°C/min

Table 20

HPLC Analyses of Standard Solutions of 3501-6 Epoxy Resin

Epoxy Resin Solution No.	Run No.	Solution Age at RT hrs	Ratio of peaks		
			DDS/ MY720	DDS/ Advancement Peak	MY720/ Advancement Peak
7 (as-is)	33	<1	0.333	3.80	11.4
7 "	57	96	0.400	3.90	9.75
7 ¹ "	59	96	0.400	4.00	10.1
7 ² "	63	168	0.323	2.93	9.08
7 "	91	264	0.500	5.03	10.02
8 (as-is) + int. std.	34	<1	0.470	4.30	9.26
8 "	58	96	0.420	1.80	4.28
8 ¹ "	60	96	0.450	3.20	7.29
8 ² "	64	168	0.430	4.54	10.42
9 adv. 3501-6	35	<1	0.371	1.27	3.43
9 "	41	24	0.282	0.876	3.11
9 "	92	264	0.360	1.67	4.63
10 adv. 3501-6 + int. std.	36	1	0.346	1.16	3.37
10 "	42	24	0.161	0.353	2.18
10 "	45	30	0.217	0.424	1.95

¹ change in gradient time to 30 min

² change in gradient time to 22 min

Table 21

HPLC Analysis of Humidity Exposed 3501-6 Epoxy Resin at 25°C

Exposure Time hrs	MY720/advancement Peak Ratio				
	Vacuum	0% RH (Dry)	31% RH	51% RH	93% RH
baseline	9.82 (5 min)	9.70	10.4	10.4	10.4
12		10.2	10.2	10.1	10.0
24	10.35	9.38	9.91	9.87	9.56
48		9.38	9.10	9.40	9.36
72		8.82	8.92	9.01	8.02
168	7.68	8.97	8.86	8.17	7.71
250		7.54	7.49	7.08	6.36
336		7.34	7.02	6.63	5.92

Table 22

HPLC Analysis of Humidity Exposed
AS/3501-6 Prepreg at 25°C

Exposure Time hrs	MY720/Advancement Peak Ratio			
	<u>0% RH</u>	<u>31% RH</u>	<u>51% RH</u>	<u>93% RH</u>
12	9.06	9.04	9.58	8.87
24	8.50	9.62	8.43	9.00
48	8.92	9.16	9.34	9.01
72	9.93	9.62	8.83	8.24
168	8.34	8.12	7.67	6.53
250	8.16	7.03	6.37	5.42
336	7.64	6.96	7.34	5.22

Table 23

HPLC Analysis of Humidity Exposed and Then Dried 3501-6
Epoxy Resin at 25°C
(Reversibility Studies)

<u>Solution No.</u>	<u>Exposure Time hrs</u>	<u>% RH</u>	<u>Wt% Change</u>	<u>Dried at 0% RH to Equil.¹Wt% from Orig.</u>	<u>Ratio of MY720/ Advancement Peak</u>
78	12	51	-0.14	-0.73	9.43
80	24	51	-0.76	-0.91	9.65
82	72	51	-0.15	-0.47	8.77
79	12	93	-0.20	-0.58	9.58
81	24	93	-2.38	-2.80	9.24
83	72	93	+0.32	-0.88	8.32

¹ approximately 4 days to equilibrium dry weight

Table 24

HPLC Analysis of Humidity Exposed and Then Dried
AS/3501-6 Prepreg at 25°C
(Reversibility Studies)

<u>Solution No.</u>	<u>Exposure Time hrs</u>	<u>% RH</u>	<u>Wt% Change</u>	<u>Dried at 0% RH to Equil.¹Wt% from Orig.</u>	<u>Ratio of MY720/ Advancement Peak</u>
70	12	51	-	-3.7	8.73
72	24	51	-1.03	-4.2	8.98
74	72	51	-0.28	-3.5	8.24
76	168	51	-0.77	-	7.01
71	12	93	-	-4.2	8.87
73	24	93	+2.80	-4.0	8.40
75	72	93	+4.16	-3.8	8.19
77	168	93	+3.70	-	6.28

¹ approximately 4 days to equilibrium dry weight

Table 25

Infrared Analysis of Humidity Exposed
3501-6 Epoxy Resin at 25°C

Exposure Time		Epoxy Peak/Aromatic Peak Ratio (915 cm ⁻¹ /1605 cm ⁻¹)			
<u>hrs</u>	<u>Days</u>	<u>0% RH</u>	<u>31% RH</u>	<u>51% RH</u>	<u>93% RH</u>
base line	(zero time)	0.300	0.283	0.249	0.209
8	0.33	-	-	-	0.232
12	0.67	0.299	0.293	-	-
16	-	-	-	-	0.207
24	1	0.278	0.271	0.257	0.202
48	2	0.309	0.307	0.268	0.193
72	3	0.306	0.307	0.261	-
120	5	-	0.300	-	0.200
136	5.6	-	-	-	0.186
168	7	0.264	0.276	0.262	0.203
211	8.8	0.223	0.224	0.241	0.199
250	10.4	-	0.227	0.210	-
336	14	0.224	-	-	0.224
343	14.3	-	0.218	0.209	-
384	16				0.195
450	19				0.183
550	23				0.194
575	24				0.175
672	28				0.186
792	33				0.177

Table 26

Infrared Analysis of Humidity Exposed
AS/3501-6 Prepreg at 25°C

Exposure Time		Epoxy Peaks/Aromatic Peaks Ratio (915 cm ⁻¹ /1605 cm ⁻¹)			
		<u>0% RH</u>	<u>31% RH</u>	<u>51% RH</u>	<u>93% RH</u>
<u>hrs</u>	<u>Days</u>				
base line (zero time)		0.264	0.319	0.289	0.263
8	0.33	0.267	0.317	0.298	0.282
16	0.67	0.269	0.289	0.312	0.270
24	1	0.278	0.289	0.303	0.266
48	2	0.278	0.300	0.290	0.259
72	3	0.272	0.293	0.290	0.241
136	5.7	0.272	0.318	0.285	0.256
206	8.6	0.276	0.286	0.295	0.236
336	14	0.264	0.289	0.295	0.254
384	16	0.266	0.280	0.291	0.233
450	19	0.264	0.277	0.298	0.237
550	23	0.261	0.289	0.285	0.229
575	24	0.262	0.281	0.299	0.210
672	28	0.267	0.276	0.280	0.229
792	33	0.265	0.278	0.271	0.208

Table 27

DSC Data for Humidity Exposed
3501-6 Resin at 25°C

Time, hrs	Heating Rate °C/min	Major exotherm Peak Temp, °C			
		-----% Relative Humidity			
		<u>0</u>	<u>31</u>	<u>51</u>	<u>93</u>
"as received"	5			216 ¹	
	10			235	
	20			255	
12	5	218	216	218	215
	10	238	237	237	235
	20	255	255	253	254
24	5	217	218	220	218
	10	236	235	238	238
	20	255	255	255	257
48	5	218	216	218	218
	10	235	235	235	235
	20	255	255	253	258
72	5	223	222	220	218
	10	240	240	238	238
	20	259	258	258	255
168	5	218	218	218	220
	10	239	235	235	238
	20	258	255	255	255
336	5	218	218	218	218
	10	238	238	238	238
	20	258	258	255	255

¹ the "as received" resin was run under prevailing lab conditions and was not exposed to any humidity conditions as implied here

Table 28

DSC Data for Humidity Exposed AS/3501-6 Prepreg at 25°C

Time, hrs	Heating Rate °C/min	Major Exotherm Peak Temp °C			
		— % Relative Humidity —			
		0	31	51	93
"as received"	5			217 ¹	
	10			238	
	20			255	
12	5	218	220	220	220
	10	237	236	237	240
	20	257	257	258	257
24	5	218	218	220	220
	10	237	237	238	238
	20	257	255	258	257
48	5	216	220	220	220
	10	235	238	238	238
	20	255	260	258	259
72	5		220	220	220
	10		238	238	238
	20		257	257	257
168	5	220	218	220	220
	10	238	237	237	238
	20	257	257	258	258
336	5	222	220	222	223
	10	238	238	242	240
	20	258	258	260	262

¹ the "as received" prepreg was run under prevailing lab conditions and was not exposed to any humidity conditions as implied here

Table 29

Energies of Cure for Humidity Aged AS/3501-6 Prepreg

Exposure Time hrs	E_a , Activation Energy of Major Cure Reaction, Kcal/mole				
	<u>"as received"</u>	<u>0% RH</u>	<u>31% RH</u>	<u>51% RH</u>	<u>93% RH</u>
	18.87				
24	-	18.36	19.26	18.99	19.54
168	-	19.54	18.36	18.99	18.99
336	-	20.09	18.72	19.13	18.74

Table 30

DSC On Set Temperature of Minor Peak in
Humidity Aged AS/3501-6 Prepreg

Exposure Time hrs	Heating Rate °C/min	On-Set Temp, °C			
		0% RH	31% RH	51% RH	93% RH
"as received"	5			110 ¹	
	10			120	
	20			130	
12	5	110	110	110	110
	10	115	118	120	120
	20	120	130	133	133
24	5	110	110	110	110
	10	120	115	120	120
	20	133	130	132	133
48	5	105	110	110	110
	10	115	118	118	120
	20	128	130	130	133
72	5	-	112	110	105
	10	-	118	120	120
	20	-	133	133	133
168	5	110	110	110	110
	10	120	120	125	120
	20	130	130	135	138
250	5	110	115	110	115
	10	120	122	120	125
	20	133	130	133	138
336	5	118	113	115	112
	10	122	118	125	135
	20	133	135	135	153

¹ the "as received" prepreg was run under prevailing lab conditions and was not exposed to any humidity conditions as implied here

Table 31

Effect of Moisture Absorption-Desorption Process on
DSC Behavior of 3501-6 Epoxy Resin
(Reversibility Studies)

Exposure Time hrs	% RH	Wt% Change	Dried at 0% RH to Equil. Wt% from Orig. ¹	Major Exotherm Peak Temp. °C at Heating Rates		
				5°C/min	10°C/min	20°C/min
12	51	-0.24	-0.57	218	235	252
24	51	-0.20	-2.1	215	233	253
72	51	-0.14	-0.24	215	233	255
168	51	+0.23	-0.07	218	235	255
12	93	-0.15	-0.38	218	230	250
24	93	-1.6	-1.8	215	233	253
72	93	-0.35	-0.13	218	230	253
168	93	+0.53	-0.10	218	237	255

¹ approximately 4 days to equilibrium dry weight

Table 32

DSC Analysis of Humidity Exposed and Then
Dried AS/3501-6 Prepreg at 25°C
(Reversibility Studies)

Exposure Time hrs	% RH	Wt% Change	Dried at 0% RH to Equil. ¹ Wt% from Orig.	Major Exotherm Peak Temp. °C at Heating Rates		
				5°C/min	10°C/min	20°C/min
12	51	-	-2.11	212	230	248
24	51	-0.09	-2.27			
72	51	-0.17	-2.34	220	237	253
168	51	-0.31	-2.37	218	237	257
12	93	-	-2.29	218	237	255
24	93	-0.84	-5.49	219	237	257
72	93	+3.40	-2.35	218	237	258
168	93	+2.13	-2.83	219	237	257

¹ approximately 4 days to equilibrium dry weight

Table 33

Effect of Humid Aging on Tack and Drape
Behavior of AS/3501-6 Prepreg

<u>Prepreg Sample</u>	<u>Prepreg Treatment</u>		<u>Tack</u>	<u>Drape</u>
	<u>Time, hrs</u>	<u>% RH</u>		
unaged "as received"			excellent	good
1	12	0	"	"
2	12	31	"	"
3	12	51	"	"
4	12	93	"	"
5	24	0	"	"
6	24	31	"	"
7	24	51	"	"
8	24	93	"	"
9	72	0	"	"
10	72	31	"	"
11	72	51	"	"
12	72	93	"	"
13	168	0	good	{ good but poorer than 3 days, but improves with humidity level.
14	168	31	"	
15	168	51	"	
16	168	93	"	{ good but poorer than 1 week, im- proves with humidity level
17	336	0	poor	
18	336	31	"	
19	336	51	"	
20	336	93	"	

Table 34

Composites Fabricated from 25°C Humidity Exposed AS/3501-6 Prepreg
(4" x 4" x 19 plies)

Composite No.	Prepreg Exposure Time, hrs	% RH	Prepreg Moisture Description or Absorption Wt%	Composite		
				Density g/cc	Thickness in.	Ply Thickness mils/ply
Nav-19	"as received"	-	+ .20	1.54	0.095	5.0
-61	12	0	- .43	1.55	0.095	5.0
-32	12	31	+ .06	1.54	0.102	5.37
-56	12	31	+ .06	1.55	0.100	5.26
-36	12	51	+ .16	1.57	0.099	5.21
-43	12	93	+ .43	1.50	0.098	5.16
-52	12	93	+ .43	1.58	0.099	5.05
-40	24	0	- .09	1.54	0.100	5.26
-21	24	31	+ .02	1.55	0.097	5.10
-42	24	51	+ .19	1.57	0.100	5.26
-54	24	93	+ .50	1.55	0.096	5.05
-24	24	93	-	1.53	0.100	5.26
-58	72	0	- .11	1.54	0.102	5.37
-25	72	31	- .05	1.52	0.100	5.26
-60	72	31	+ .08	1.52	0.101	5.32
-22	72	51	+ .20	1.55	0.097	5.10
-20	72	93	+ .65	1.55	0.094	4.94
-27	168	0	- .11	1.53	0.105	5.52
-35	168	31	+ .06	1.54	0.099	5.21
-39	168	51	+ .21	1.55	0.102	5.36
-30	168	93	+ .60	1.57	0.098	5.15
-50	336	0	- .11	1.53	0.099	5.21
-51	336	31	+ .07	1.55	0.103	5.42
-48	336	51	+ .21	1.56	0.097	5.10
-46	336	93	+ .70	1.58	0.097	5.10
-63	12	100	0.50	1.56	0.096	5.05
-65	24	100	+ .90	1.56	0.100	5.26
-68	48	100	1.37	1.56	0.107	5.63
-66	160	100	1.54	1.57	0.100	5.26

Table 35

Composites Fabricated from 25°C Humidity
Exposed AS/3501-6 Prepreg
(6" x 6" x 10 plies)

Composite No.	Prepreg Exposure Time, hrs	% RH	Prepreg Moisture Desorption or Absorption, Wt%	Thickness in.	Ply Thickness mils/ply
Nav-16	"as received"	-	+0.20	0.051	5.1
-37	12	0	-.43	0.051	5.1
-53	12	31	+0.06	0.054	5.4
-33	12	51	+0.16	0.052	5.2
-59	12	93	+0.43	0.053	5.3
-17	24	0	-.09	0.053	5.3
-55	24	31	+0.02	0.052	5.2
-57	24	51	+0.19	0.052	5.2
-23	24	93	+0.50	0.050	5.0
-34	72	0	-.11	0.051	5.1
-28	72	31	-.05	0.050	5.0
-18	72	51	+0.20	0.051	5.1
-29	72	93	+0.65	0.052	5.2
-26	168	0	-.11	0.051	5.1
-41	168	31	+0.06	0.048	4.8
-31	168	51	+0.21	0.051	5.1
-38	168	93	+0.60	0.049	4.9
-49	336	0	-.11	0.053	5.3
-44	336	31	+0.07	0.054	5.4
-47	336	51	+0.21	0.055	5.5
-45	336	93	+0.70	0.054	5.4
-62	12	100	+0.50	0.056	5.6
-64	24	100	1.37	0.052	5.2
-69	48	100	+0.90	0.052	5.2
-67	160	100	1.54	0.054	5.4

Table 36

Observation of Photomicrographs of Composite
Cross-Section for Void Content

	<u>Prepreg Treatment</u>		<u>Observation of Photomicrograph Long. Cross Section</u>
	<u>Prepreg Time hrs</u>	<u>% RH @ 25°C</u>	
NAV-19	unaged, "as received"		void free
-61	12	0	void free
-32	12	31	many microvoids between plies
-56	12	31	void free
-36	12	51	void free
-43	12	93	void free
-52	12	93	void free
-40	24	0	void free
-21	24	31	very few microvoids between plies
-42	24	51	void free
-54	24	93	several large voids between plies
-24	24	93	very few microvoids between plies
-58	72	0	void free
-25	72	31	very few microvoids between plies
-60	72	31	void free
-22	72	51	very few voids between plies
-20	72	93	void free
-27	168	0	void free
-35	168	31	a few microvoids between plies
-39	168	51	void free
-30	168	93	void free
-50	336	31	void free
-51	336	51	void free
-48	336	93	void free
-46	336	93	void free
-63	12	100	void free
-65	24	100	void free
-68	48	100	several large voids between plies
-66	160	100	void free

Table 37

Interlaminar Shear Strength of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		RT Shear Strength ¹			
	Time hrs	% RH @ 25°C	Dry ²		Wet ³	
			ksi	MPa	ksi	MPa
NAV-19	Unaged "as received"		15.1	106.1	12.1	83.4
-61	12	0	12.9	83.9	13.8	95.1
-32	12	31	7.63	52.6	6.75	46.6
-56	12	31	15.0	103	13.7	94.5
-36	12	51	15.5	113	12.7	87.6
-43	12	93	14.2	97.9	13.5	93.1
-52	12	93	15.2	108	13.1	90.3
-40	24	0	15.8	110	14.1	97.2
-21	24	31	13.5	93.1	11.1	76.5
-42	24	51	14.5	100	13.1	90.3
-54	24	93	14.6	100.8	13.1	90.3
-24	24	93	9.79	67.5	9.60	66.2
-58	72	0	15.5	107.5	14.3	98.6
-60	72	31	14.4	100.2	13.0	89.6
-22	72	51	14.6	100.5	13.5	93.1
-20	72	93	14.2	98.3	13.6	93.8
-27	168	0	14.3	98.6	-	-
-35	168	31	14.2	101.8	12.9	88.9
-39	168	51	15.6	107	13.5	93.1
-30	168	93	15.5	107.2	14.5	100
-50	336	0	12.7	87.8	10.1	69.6
-51	336	31	14.8	102.3	12.9	88.9
-48	336	51	14.6	100	13.5	93.1
-46	336	93	16.8	116	14.3	98.6
-63	12	100 ⁴	13.1	90.7	12.5	86.2
-65	24	100 ⁴	14.6	100.6	14.0	96.6
-68	48	100 ⁴	15.0	102.6	15.0	103.4
-66	160	100 ⁴	16.2	111	13.3	91.7

¹ average of two specimens

² dry - as fabricated condition

³ wet - after exposure to 87% RH, 71°C to saturation

⁴ at 29°C

Table 38

Interlaminar Shear Strength of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		122°C Shear Strength ¹			
	Time	% RH	Dry ²		Wet ³	
	hrs	@ 25°C	ksi	MPa	ksi	MPa
NAV-19	unaged "as received"		12.2	83.9	8.13	56.0
-61	12	0	10.4	71.7	7.80	53.8
-32	12	31	6.29	43.4	6.30	43.4
-56	12	31	8.30	57.2	8.30	54.8
-36	12	51	12.2	84.5	9.25	63.7
-43	12	93	10.7	74.1	8.25	56.8
-52	12	93	11.6	83.0	8.88	61.2
-40	24	0	11.7	80.7	8.11	55.9
-21	24	31	10.2	70.1	6.49	44.7
-42	24	51	11.8	81.4	9.43	65.0
-54	24	93	11.2	77.2	8.68	59.8
-24	24	93	7.50	57.7	-	-
-58	72	0	12.2	82.8	7.72	53.1
-60	72	31	11.3	77.9	8.10	55.8
-22	72	51	11.6	80.2	8.30	57.2
-20	72	93	11.7	80.9	9.99	68.8
-27	168	0	11.1	76.5	7.70	53.1
-35	168	31	10.6	73.0	7.60	52.4
-39	168	51	12.3	84.8	8.78	60.6
-30	168	93	11.2	77.6	9.02	62.2
-50	336	0	9.9	68.2	6.39	44.1
-51	336	31	11.3	87.8	9.30	64.1
-48	336	51	12.0	82.4	8.40	57.9
-46	336	93	11.8	81.0	9.01	62.1
-63	12	100 ⁴	10.7	73.8	7.00	48.3
-65	24	100 ⁴	11.8	81.3	8.60	59.3
-68	48	100 ⁴	11.9	82.0	7.00	48.3
-66	160	100 ⁴	11.4	78.6	7.70	53.1

¹ average of two specimens

² dry - as fabricated condition

³ wet - after exposure to 87% RH, 71°C to saturation

⁴ at 29°C

Table 39

Transverse Tensile Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		RT Transverse Tensile Properties ¹									
	Time hrs	% RH @ 25°C	Dry ²			Wet ³						
			Strength ksi	Modulus 10 ⁶ psi	εf %	Strength MPa	Modulus 10 ⁶ psi	εf %				
NAV-19		unaged										
-61	12	0	6.58	1.25	8.62	0.50	4.10	1.43	28.3	9.90	0.28	
-32	12	31	5.40	1.50	10.34	0.35	2.64	1.52	18.3	10.5	0.18	
-56	12	31	6.40	1.29	8.90	0.52	5.40	1.50	37.2	10.3	0.38	
-36	12	51	broken before test				5.40	1.62	37.2	11.1	0.32	
-43	12	93	broken before test				broken before test					
-52	12	93	7.50	1.35	9.31	0.52	4.00	1.54	27.6	10.6	0.27	
-40	24	0	5.19	1.48	10.21	0.37	4.80	1.40	33.1	9.70	0.35	
-21	24	31	broken before test				broken before test					
-42	24	51	7.94	1.38	9.52	0.57	4.00	1.59	27.6	11.0	0.26	
-54	24	93	broken before test				broken before test					
-24	24	93	7.20	1.27	8.76	0.51	4.89	1.52	33.7	10.5	0.26	
-58	72	0	6.20	1.18	8.14	0.52	broken before test					
-25	72	31	5.80	1.48	10.21	0.41	3.45	1.22	23.8	8.48	0.23	
-60	72	31	5.10	1.33	9.17	0.38	3.43	1.47	23.6	10.1	0.25	
-22	72	51	5.61	1.49	10.28	0.38	2.08	1.30	14.3	9.0	0.17	
-20	72	93	7.03	1.59	10.97	0.52	5.50	1.72	37.9	11.9	0.31	
-27	168	0	6.87	1.46	10.07	0.61	5.72	1.80	39.5	12.4	0.36	
-35	168	31	7.83	1.27	8.76	0.61	3.90	1.47	26.9	11.5	0.26	
-39	168	51	broken before test				3.40	1.58	23.5	10.9	0.28	
-30	168	93	broken before test				broken before test					
-50	336	0	7.55	1.42	9.79	0.54	5.90	1.81	40.7	12.5	0.32	
-51	336	31	4.70	1.91	13.17	0.24	4.15	1.49	28.6	10.3	0.29	
-48	336	51	6.60	1.42	9.79	0.52	5.66	1.46	39.0	10.0	0.32	
-46	336	93	7.68	1.35	9.21	0.74	5.95	1.41	41.0	9.72	0.38	
-63	12	100 ⁴	6.15	1.70	11.72	0.37	4.92	1.75	33.9	12.1	0.31	
-65	24	100 ⁴	6.20	1.56	10.76	0.33	4.00	1.28	27.6	8.83	0.31	
-68	48	100 ⁴	broken before test				broken before test					
-66	160	100 ⁴	3.54	1.54	10.62	0.23	broken before test					
			3.85	1.67	11.52	0.23	4.23	1.70	29.2	11.7	0.26	

¹ average of two specimens

² dry - as fabricated condition

³ wet - after exposure to 87% RH, 71°C to saturation
⁴ at 29°C

Table 40

Transverse Tensile Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		122°C Transverse Tensile Properties									
	Time hrs	% RH @ 25°C	Dry ²					Wet				
			Strength		Modulus	Strength		Modulus	Strength		Modulus	
			ksi	MPa		10 ⁶ psi	GPa		ksi	MPa		10 ⁶ psi
εf %		εf %		εf %		εf %						
NAV-19		unaged	7.40	51.0	1.41	9.72	0.66	1.42	9.79	0.73	5.00	0.34
-61	12	"as received"	2.02 ³	13.9	1.38	9.52	0.15	1.92	13.2	0.80	10.0	0.27
-32	12		5.0	34.4	1.07	7.38	0.49	1.35	9.31	1.04	7.17	0.14
-56	12		broken before test					broken before test				
-36	12		broken before test					broken before test				
-43	12		7.40	51.0	1.50	10.34	0.51	1.14	7.86	0.75	5.17	0.19
-52	12		5.40	37.2	1.35	9.31	0.47	1.51	10.4	0.86	1.45	0.24
-40	24		broken before test					2.36	16.3	0.95	6.55	0.28
-21	24		7.30	50.3	1.33	9.17	0.56	1.26	8.69	0.76	5.24	0.19
-42	24		broken before test					broken before test				
-54	24		4.54	31.3	1.36	9.38	0.38	2.47	17.0	0.92	2.21	0.27
-24	24		5.32	36.6	1.07	7.38	0.45	broken before test				
-58	72		7.44	51.3	1.43	9.86	0.47	2.50	17.2	0.93	6.41	0.18
-25	72		4.60	31.7	0.98	6.76	0.46	broken before test				
-60	72		5.70	39.3	1.37	9.45	0.38	2.04	13.7	0.77	5.31	0.34
-22	72		5.68	39.1	1.39	9.59	0.43	1.67	11.5	0.78	5.37	0.28
-20	72		8.07	55.6	1.38	9.52	0.59	3.49	24.0	0.85	5.86	0.40
-27	168		8.41	58.0	0.87	6.00	0.99	1.22	8.41	0.67	4.62	0.21
-35	168		broken before test					1.61	11.1	0.82	5.66	0.22
-39	168		broken before test					broken before test				
-30	168		broken before test					3.39	11.1	1.72	11.8	0.52
-50	336		5.30	36.5	1.75	12.07	0.29	broken before test				
-51	336		5.90	40.6	1.27	8.76	0.49	3.50	24.1	1.30	8.96	0.30
-48	336		5.40	37.2	1.33	9.17	0.45	3.67	25.3	1.30	8.96	0.36
-46	336		7.01	48.3	1.46	10.07	0.51	3.93	27.1	0.62	4.27	0.69
-63	12		5.15	35.5	1.40	9.66	0.37	0.90	6.21	0.74	5.10	
-65	24		broken before test					broken before test				
-68	48		4.21	29.0	1.30	8.97	0.32	1.51	10.4	0.80	5.51	0.41
-66	160		4.17	28.8	1.38	9.52	0.30	2.33	16.0	0.79	5.45	0.34

¹ average of two specimens
² dry - as fabricated condition
³ suggests that specimen is precracked
⁴ wet - after exposure to 87% RH, 71°C to saturation
⁵ at 29°C

Table 41

Longitudinal Tensile Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		RT Tensile Properties ¹				
	Time hrs	% RH @ 25°C	Strength		Modulus		ϵ_f %
			ksi	MPa	10 ⁶ psi	GPa	
NAV-16	unaged "as received"		227	1560	19.5	135	0.85
-37	12	0	226	1559	19.3	133	0.94
-53	12	31	252	1737	20.0	138	1.03
-33	12	51	doubler slipped				
-59	12	93	doubler slipped				
-17	24	0	251	1735	18.2	126	1.09
-55	24	31	234	1613	18.0	124	1.65
-57	24	51	215	1429	21.0	145	0.92
-23	24	93	doubler slipped				
-34	72	0	246	1697	19.4	134	0.89
-28	72	31	260	1792	18.2	126	1.09
-18	72	51	doubler slipped				
-29	72	93	182	1255	18.8	130	0.90
-26	168	0	259	1789	18.9	130	1.09
-41	168	31	doubler slipped				
-31	168	51	223	1537	18.3	126	1.05
-38	168	93	238	1639	18.6	128	0.85
-49	336	0	269	1852	20.3	140	1.06
-44	336	31	doubler slipped				
-47	336	51	249	1716	18.0	124	0.90
-45	336	93	266	1832	18.3	126	1.12
-62	12	100 ³	196	1124	17.0	117	1.40
-64	24	100 ³	198	1365	18.2	126	1.42
-69	48	100 ³	195	1345	17.1	118	1.46
-67	160	100 ³	192	1324	17.0	117	1.32

¹ average of two specimens

² dry - "as fabricated" condition

³ at 29°C

Table 42

Longitudinal Tensile Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		RT Tensile Properties ¹				
	Time hrs	% RH @ 25°C	Strength		Modulus		ϵ_f %
			ksi	MPa	10 ⁶ psi	GPa	
NAV-16	Unaged "as received"		doubler slipped				
-37	12	0	207	1426	15.4	106	1.02
-53	12	31	268	1848	23.1	159	1.04
-33	12	51	237	1635	20.2	139	0.95
-59	12	93	280	1931	17.3	119	1.20
-17	24	0	doubler slipped				
-55	24	31	217	1496	19.8	136	0.98
-57	24	51	234	1613	17.9	123	1.26
-23	24	93	219	1510	19.8	136	0.94
-34	72	0	250	1724	22.4	155	0.99
-28	72	31	238	1641	19.5	134	1.00
-18	72	51	224	1545	16.7	115	0.99
-29	72	93	doubler slipped				
-26	168	0	234	1614	16.8	116	1.01
-41	168	31	doubler slipped				
-31	168	51	doubler slipped				
-38	168	93	235	1621	18.0	124	.4
-49	336	0	220	1517	19.1	132	0.73
-44	336	31	231	1593	18.2	126	1.29
-47	336	51	245	1689	18.8	130	1.06
-45	336	93	262	1806	17.1	118	1.00
-62	12	100 ³	214	1476	18.3	126	0.92
-64	24	100 ³	238	1641	19.9	137	0.99
-69	48	100 ³	225	1551	19.4	134	0.95
-67	160	100 ³	222	1531	20.9	144	0.89

¹ average of two specimens

² wet - after exposure to 87% RH, 71°C to saturation

³ at 29°C

⁴ disengaged prior to failure

Table 43

Longitudinal Tensile Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		122°C Tensile Properties ¹				
	Time hrs	% RH @ 25°C	Strength		Modulus		ϵ_f %
			ksi	MPa	10 ⁶ psi	GPa	
Nav-16	Unaged "as received"		247	1703	20.9	144	1.02
-37	12	0	205	1410	22.7	156	0.92
-53	12	31	275	1899	20.0	138	1.14
-33	12	51	doubler slipped				
-59	12	93	doubler slipped				
-17	24	0	256	1772	19.6	139	1.06
-55	24	31	191	1317	18.2	126	1.45
-57	24	51	217	1500	21.5	149	0.93
-23	24	93	doubler slipped				
-34	72	0	204	1343	18.5	127	0.90
-28	72	31	doubler slipped				
-18	72	51	doubler slipped				
-29	72	93	215	1481	20.2	139	0.95
-26	168	0	doubler slipped				
-41	168	31	doubler slipped				
-31	168	51	doubler slipped				
-38	168	93	208	1452	20.6	142	0.90
-49	336	0	247	1702	21.5	147	1.04
-44	336	31	188	1296	16.6	115	1.36
-47	336	51	239	1645	21.1	146	1.00
-45	336	93	232	1599	18.5	128	1.05
-62	12	100 ³	169	1166	18.7	129	1.20
-64	24	100 ³	170	1172	20.1	139	1.30
-67	160	100 ³	204	1407	20.7	143	1.35
-69	48	100 ³	188	1296	18.1	125	1.38

¹ average of two specimens

² dry - as fabricated condition

³ at 29°C

Table 44

Longitudinal Tensile Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		122°C Tensile Properties ¹				
	Time hrs	% RH @ 25°C	Strength		Modulus		ϵ_f %
			ksi	MPa	10 ⁶ psi	GPa	
NAV-16	Unaged "as received"		227	1566	17.9	123	1.32
-37	12	0	206	1421	22.2	153	1.10
-53	12	31	249	1717	22.5	155	1.12
-33	12	51	235	1621	19.8	137	1.11
-59	12	93	257	1772	15.8	109	1.54
-17	24	0	242	1669	18.0	124	1.49
-55	24	31	226	1559	19.2	132	1.06
-57	24	51	209	1441	21.0	145	1.08
-23	24	93	199	1372	18.8	130	1.10
-34	72	0	216	1489	21.7	150	1.10
-28	72	31	doubler slipped				
-18	72	51	doubler slipped				
-29	72	93	187	1421	23.6	163	- 4
-26	168	0	doubler slipped				
-41	168	31	248	1710	21.8	150	1.15
-31	168	51	doubler slipped				
-38	168	93	208	1334	21.4	148	1.10
-49	336	0	249	1717	22.7	157	0.86
-44	336	31	208	1434	23.3	161	0.88
-47	336	51	220	1517	20.6	142	1.00
-45	336	93	252	1739	18.0	124	1.52
-62	12	100 ³	209	1441	21.6	149	0.85
-64	24	100 ³	218	1503	17.1	118	1.40
-69	48	100 ³	184	1269	23.1	159	0.66
-67	160	100 ³	223	1538	19.0	131	1.00

¹ average of two specimens

² wet - after exposure to 87% RH, 71°C to saturation

³ at 29°C

⁴ disengaged prior to failure

Table 45

Compression Properties of AS/3501-6 Composites
Fabricated From Humidity Aged Prepreg

Composite No.	Prepreg Treatment		RT Compression Properties				
	Time hrs	% RH @ 25°C	Strength		Modulus		ϵ_f %
			ksi	MPa	10^6 psi	GPa	
NAV-16	Unaged "as received"		148	1020	17.2	119	1.43
-37	12	0	164	1127	16.5	114	1.37
-53	12	31	155	1026	15.0	100	1.00
-33	12	51	137	945	13.0	90	1.76
-59	12	93	150	1034	18.3	126	0.58
-17	24	0	141	970	15.8	110	1.37
-55	24	31	140	965	13.0	90	1.77
-57	24	51	125	861	14.0	96	1.20
-23	24	93	150	1034	16.5	114	1.10
-34	72	0	144	1002	17.2	129	1.40
-28	72	31	124	854	16.4	113	1.40
-18	72	51	135	931	15.0	100	1.33
-29	72	93	144	989	14.9	100	
-26	168	0	155	1066	17.3	120	1.48
-41	168	31	131	903	14.2	98	1.80
-31	168	51	174	1199	17.2	120	-
-38	168	93	145	996	13.6	94	1.65
-49	336	0	154	1062	16.0	98	1.30
-44	336	31	150	1034	16.0	110	0.74
-47	336	51	158	1097	18.1	124	1.18
-45	336	93	159	1099	16.7	105	1.75
-62	12	100 ³	171	1179	15.4	106	1.40
-69	48	100 ³	132	910	15.2	105	0.80
-64	24	100 ³	151	1041	20.8	143	0.67
-67	160	100 ³	146	1007	19.3	133	0.71

¹ average of two specimens

² dry - as fabricated condition

³ at 29°C

Table 46

Compression Properties of AS/3501-6 Composites
Fabricated from Humidity Aged Prepreg

Composite No.	Prepreg Treatment		122°C Compression Properties ¹				
	Time hrs	% RH @ 25°C	Strength		Modulus		ε _f %
			ksi	MPa	10 ⁶ psi	GPa	
NAV-16	Unaged "as received"		140	968	17.7	122	-
-37	12	0	127	878	17.3	119	0.64
-53	12	31	104	717	15.2	105	0.90
-33	12	51	110	759	17.6	121	0.80
-59	12	93	130	89.6	19.4	134	0.47
-17	24	0	133	922	16.3	111	-
-55	24	31	126	86.9	17.3	119	0.56
-57	24	51	143	985	16.2	113	1.0
-23	24	93	118	81.3	13.6	94	1.1
-34	72	0	122	854	17.3	119	0.97
-28	72	31	doubler slipped				
-18	72	51	90	61.7	19.2	132	0.47
-29	72	93	130	894	17.1	118	0.70
-26	168	0	doubler slipped				
-41	168	31	108	745	16.5	114	1.32
-31	168	51	doubler slipped				
-38	168	93	125	860	16.9	116	0.79
-49	336	0	123	862	14.1	98	1.46
-44	336	31	146	1007	17.3	119	0.64
-47	336	51	136	937	19.6	132	1.15
-45	336	93	157	1080	16.3	112	1.62
-62	12	100 ³	166	1144	19.6	135	0.95
-69	48	100 ³	145	1000	17.6	121	1.32
-64	24	100 ³	158	1089	15.9	109	1.20
-67	160	100 ³	160	1103	19.6	135	1.04

¹ average of two specimens

² dry - as fabricated condition

³ at 29°C

Table 47

Hercules Incorporated Quality Assurance Certification

CUSTOMER: United Technologies
 PURCHASE ORDER NO: 27539
 MATERIALS: Graphite Fiber/Epoxy Material, 3501-6/AS1, 12" prepreg tape
 SPECIFICATION: MMS 549, Rev. A, Type I
 QUANTITY: 16.00 lbs.
 LOT NO: 1823 Manufactured February 11, 1981
 SPOOL NO: 2A
 RESIN LOT NO: 136 Manufactured by Hercules Inc.
 FIBER LOT NO: 185-20 Manufactured by Hercules Inc.

I. Fiber Properties

	<u>Spec Req</u>	<u>Lot Average</u>
Tensile Str., ksi	410 minimum	524
Tensile Mod., msi	32-36	34
Density, lb/in ³	0.0640-0.0660	0.0656

II. Prepreg Physical Properties

	<u>Spec Req</u>	<u>Average/Individual</u>
Spool No.		1
Resin Flow, %	10-25	19
Volatiles, %	1.5 max	1.0/1.1, 1.0, 0.8
Tack	Table I, spec	Conforms

III. Laminate Mechanical Properties

	<u>Spec Req</u>	<u>Panel #</u>	<u>Average/Individual</u>
	(min. ind)	Spool 1	
0° Tensile Str., RT, ksi*	200	12267	281/284, 260, 299
0° Tensile Mod., RT, msi*	18.0	12267	19.6/20.3, 18.0, 20.5
0° Elongation, RT, $\mu\text{in}/\text{in} \times 10^3$	10.0	12267	14.3/14.2, 14.4, 14.3
Short Beam Shear, RT, ksi	15.0	12268	17.4/17.7, 17.3, 17.3
Short Beam Shear, 250°F, ksi	9.0	12268	11.5/11.1, 11.6, 11.9
Short Beam Shear, 250°F, ksi (24 hr H ₂ O boil)	7.5	12268	11.9/11.9, 11.9, 11.8

*Normalized to 0.0416 panel thickness.

Table 47 (Cont'd)

IV. Panel Physical Properties

	<u>Spec Req</u>	
Spool No./Panel No.		1/12267
Ply Thickness, inches	0.0052 \pm 0.0003	0.0052

V. Individual Spool Physical Properties

<u>Spec Req</u>	<u>Average/Individual</u>	<u>Average/Individual</u>
	42 \pm 3	145-155
<u>Spool No.</u>	<u>Resin Content, %</u>	<u>Fiber Areal Wt, gm/m²</u>
2A	42/42,42,42	155/155,155,155

Table 48

Final Inspection Report - 12" Wide Prepreg
Hercules Incorporated

<u>Lot. No.</u>	<u>Spool No.</u>	<u>Product</u>		
1823	2A	Magnamite ^R	3501-6/AS	
<u>Feet from Start</u>	<u>Defect Length</u>	<u>Zone</u>	<u>TMB</u>	<u>Defect Type/Remarks</u>
178	2'			Gap
292	1'			Hole
300	End of Spool			

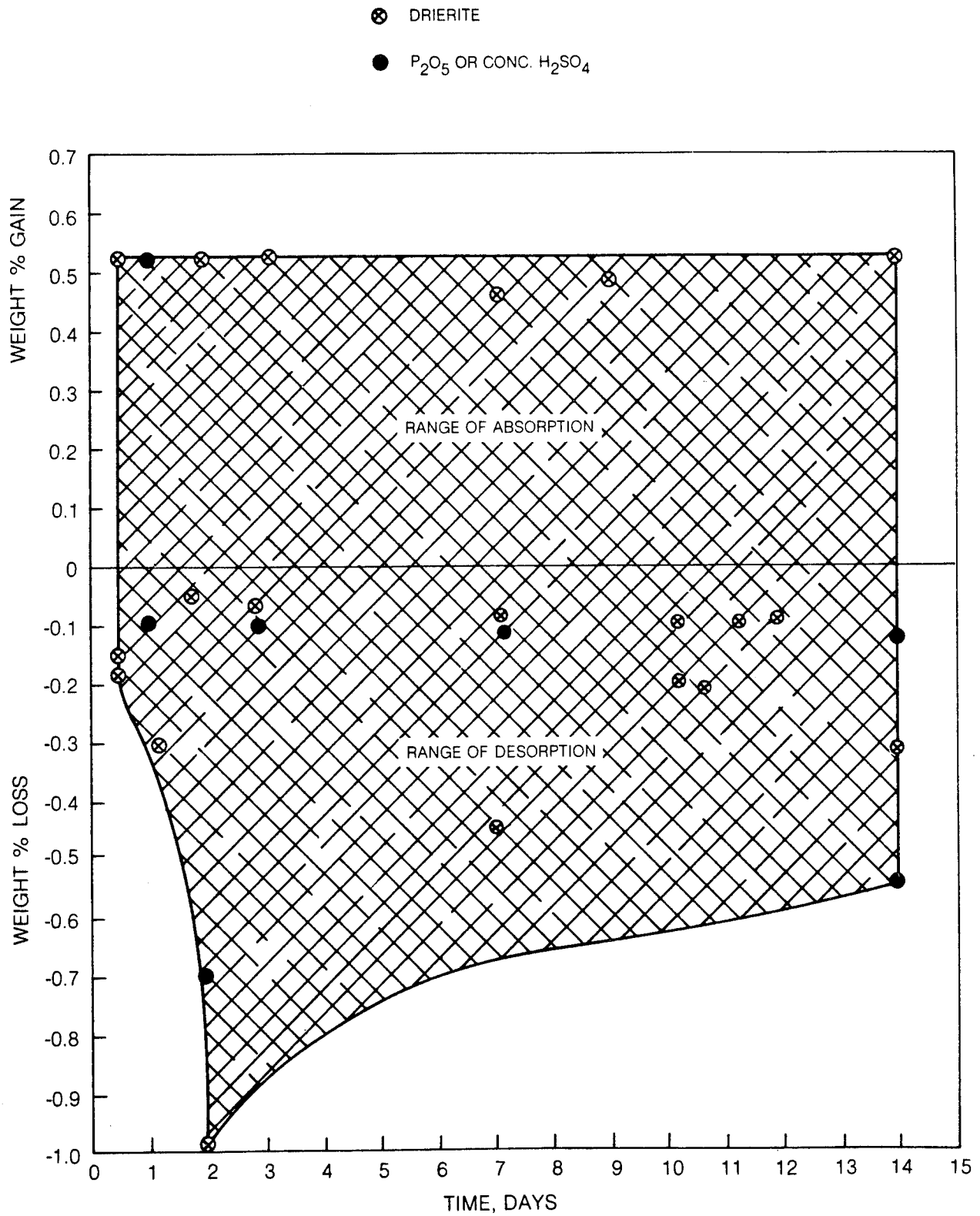
Table 49

Autoclave Fabrication of AS/3501-6 Graphite Epoxy Composites¹
 (Audrey II Model 203 Monoprobe Monitored)

Composite No.	Plies	Density g/cc	Vol %			Thickness in.	Mils/Ply
			Resin	Fiber	Void		
NAV-1	10	1.53	34.6	62.4	2.8	0.054	5.4
-2	10	1.52	32.5	64.2	3.2	0.054	5.4
-8	19	1.58	24.8	72.3	2.9	0.095	5.0
-9	20	1.58	25.1	72.3	2.6	0.100	5.0
-10	20	1.50	27.9	65.2	6.3	0.101	5.05
-11	20	1.56	26.5	71.8	3.7	0.098	4.9
-12	20	-	-	-	-	0.096	4.8

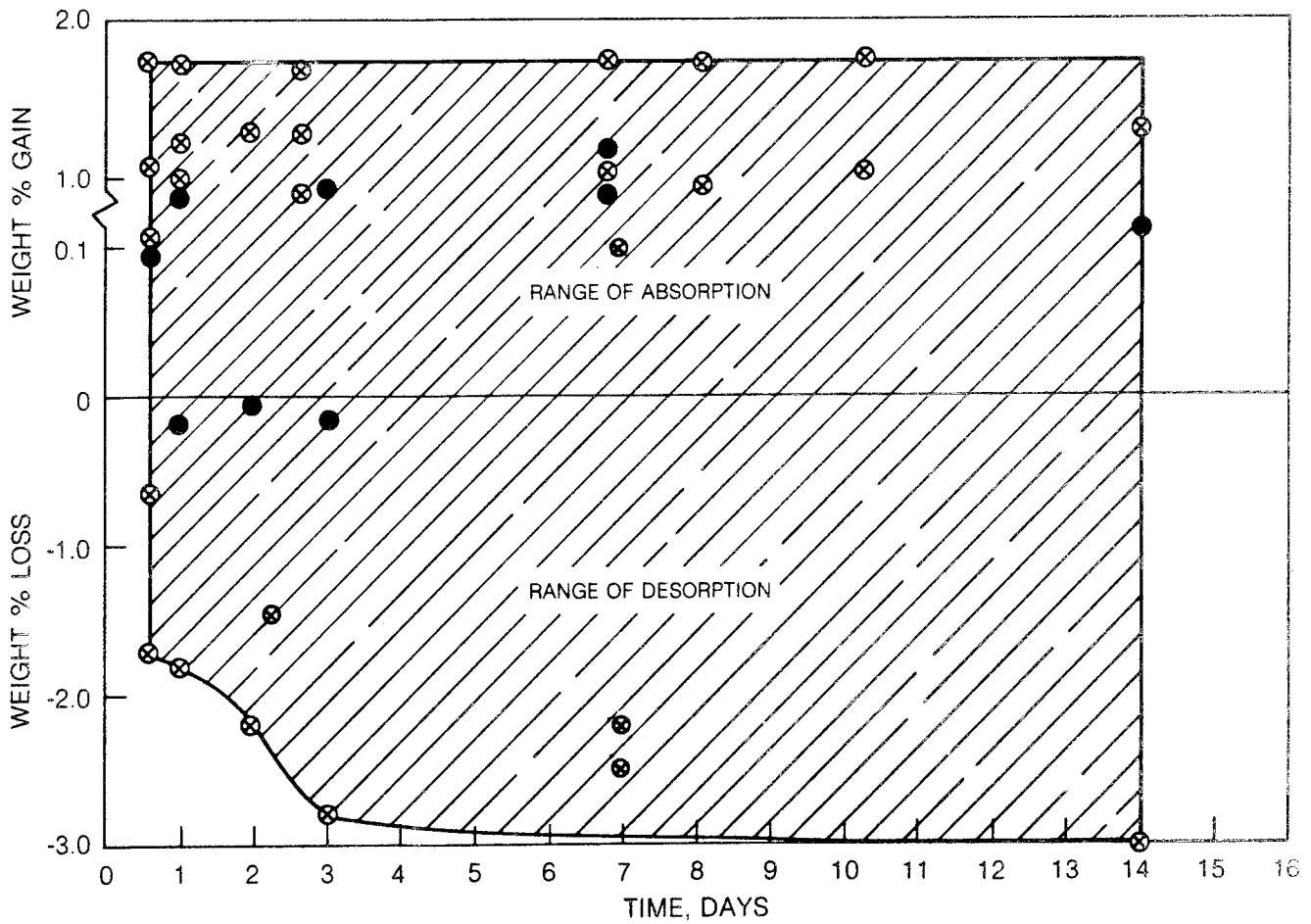
¹ as received prepreg used in these runs

MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF 3501-6 EPOXY RESIN AT LOW HUMIDITY LEVELS AT 25°C

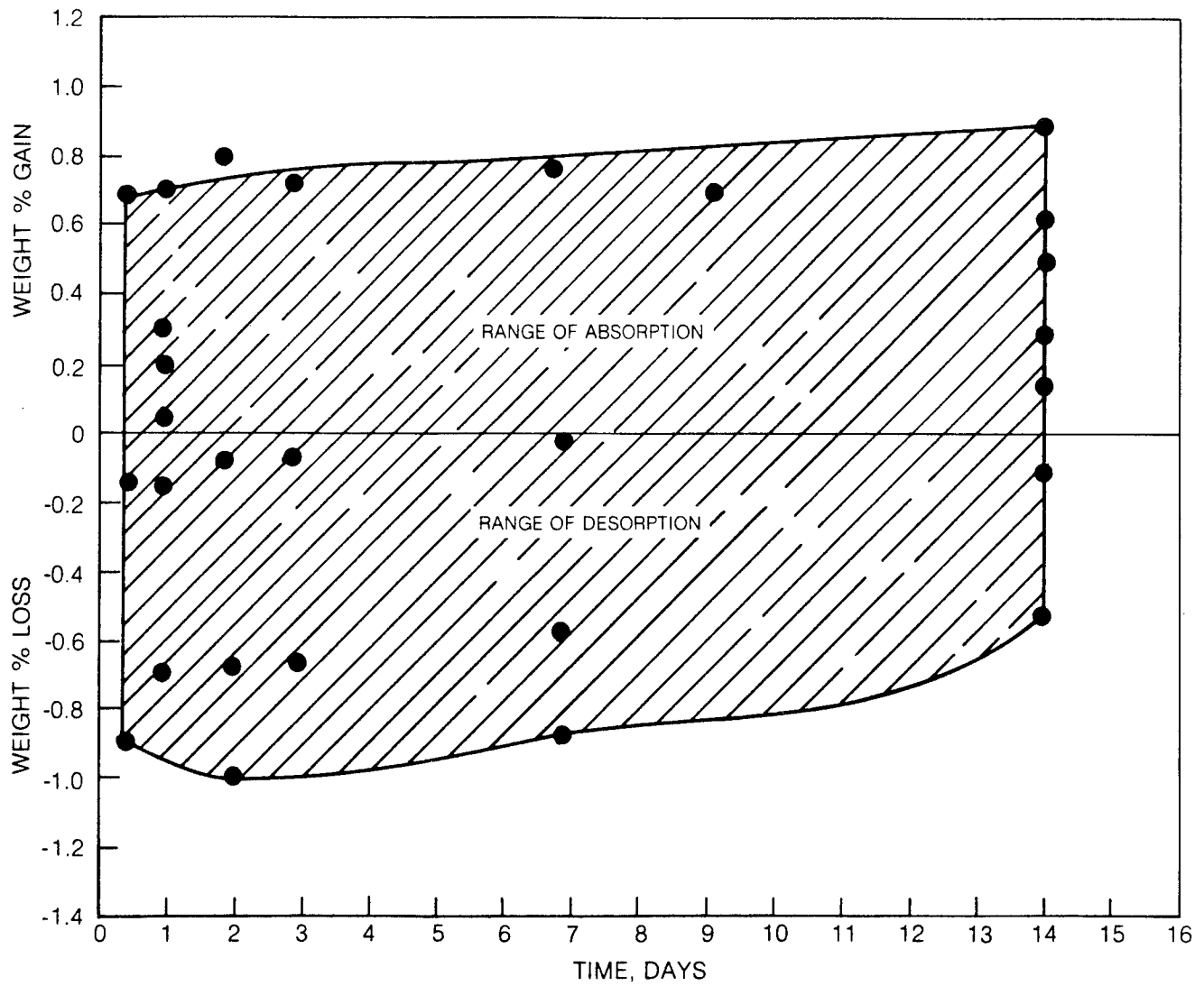


MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF AS/3501-6 PREPREG AT LOW HUMIDITY LEVELS AT 25°C

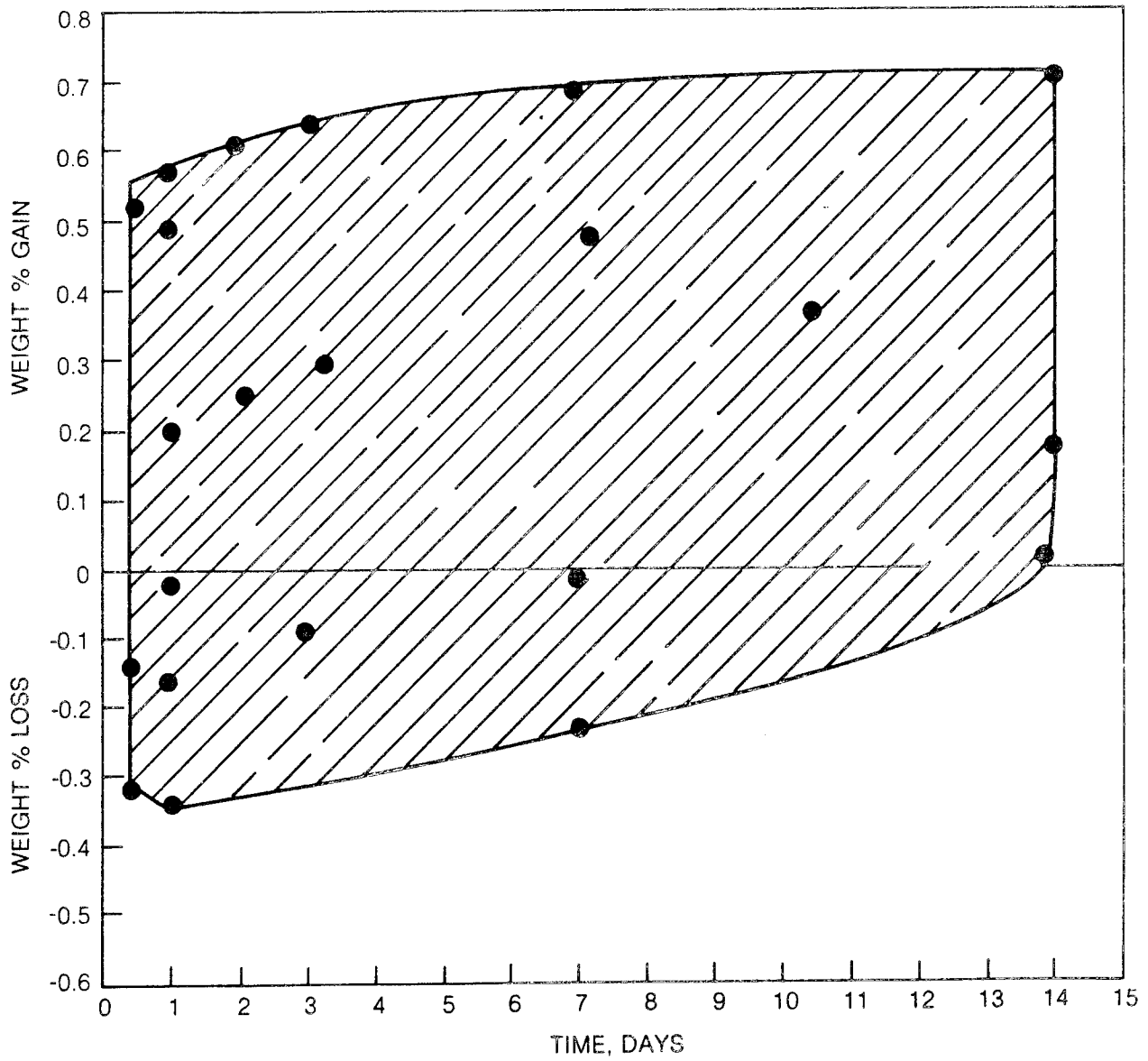
- ⊗ DRIERITE
- P₂O₅ OR CONC H₂SO₄



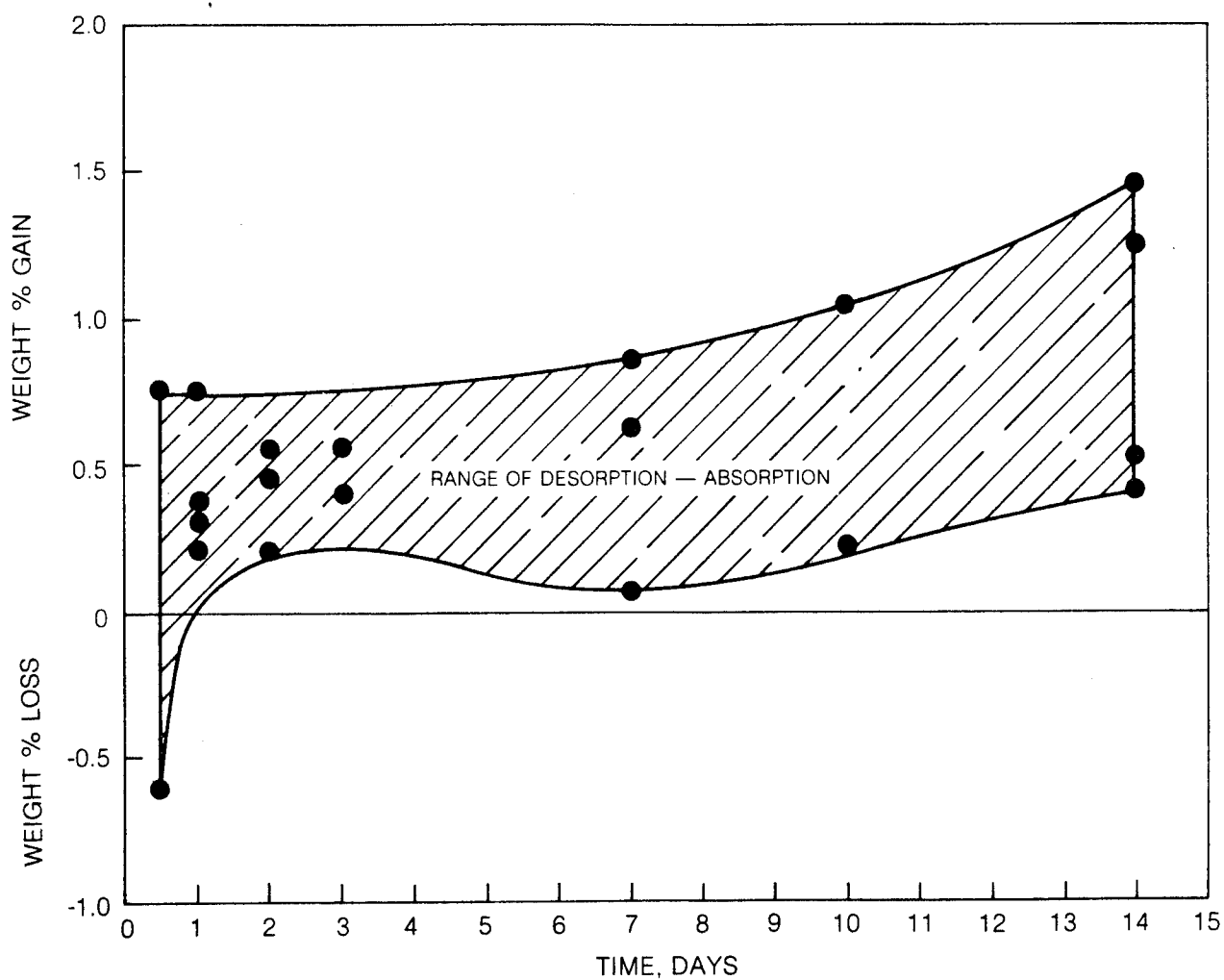
MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF 3501-6 EPOXY RESIN AT 31% R.H., 25°C



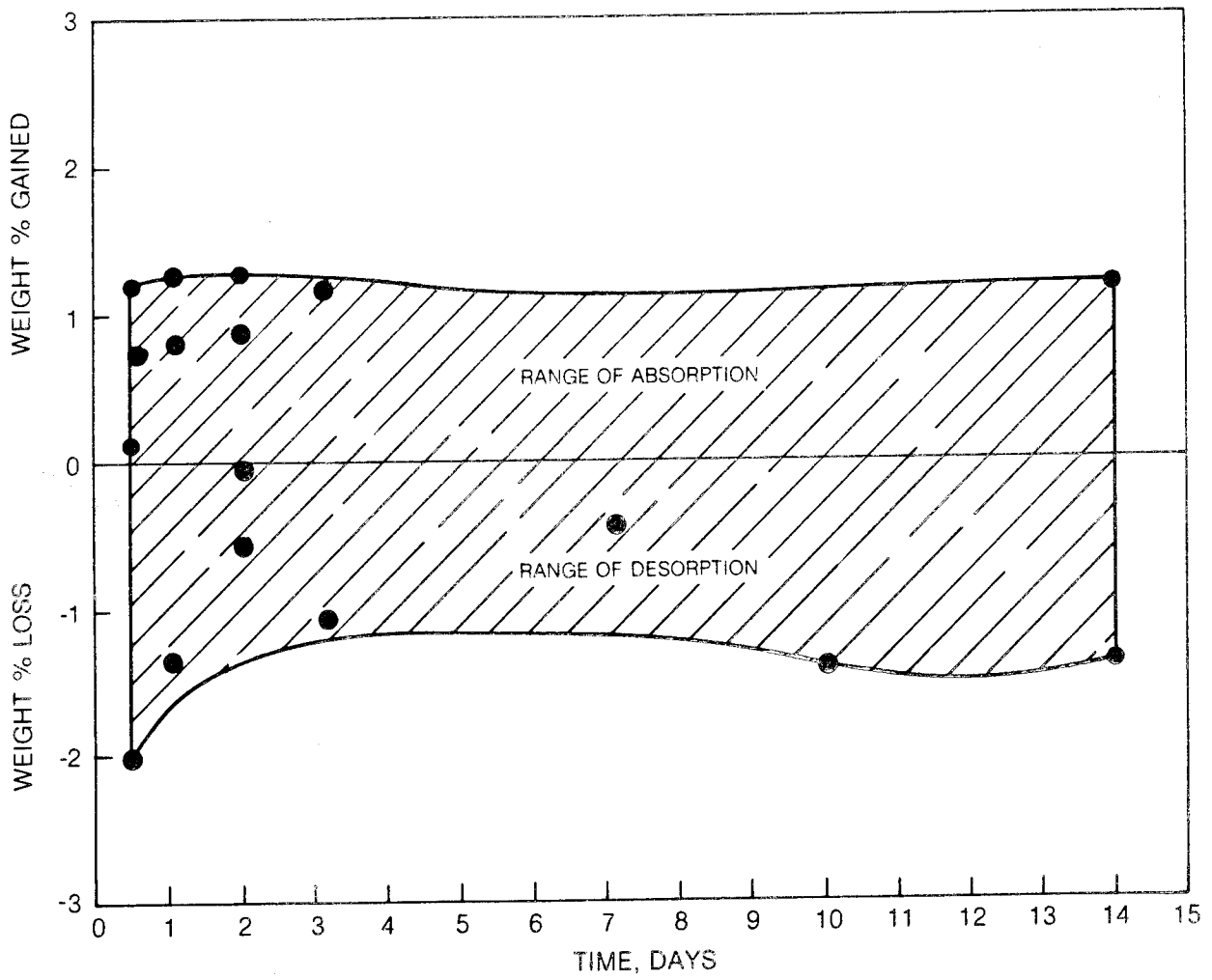
**MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF 3501-6 EPOXY RESIN
AT 51% R.H., 25°C**



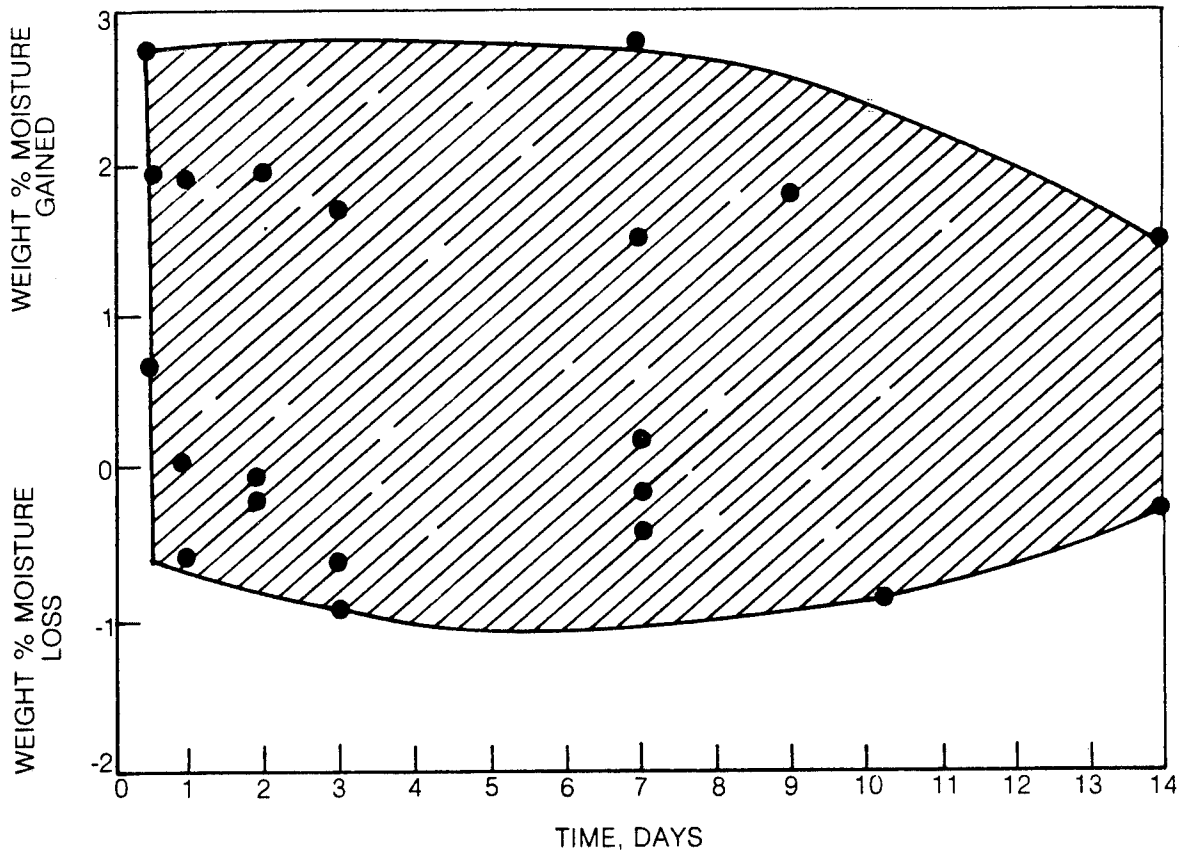
MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF 3501-6 EPOXY RESIN
AT 93% R.H., 25°C



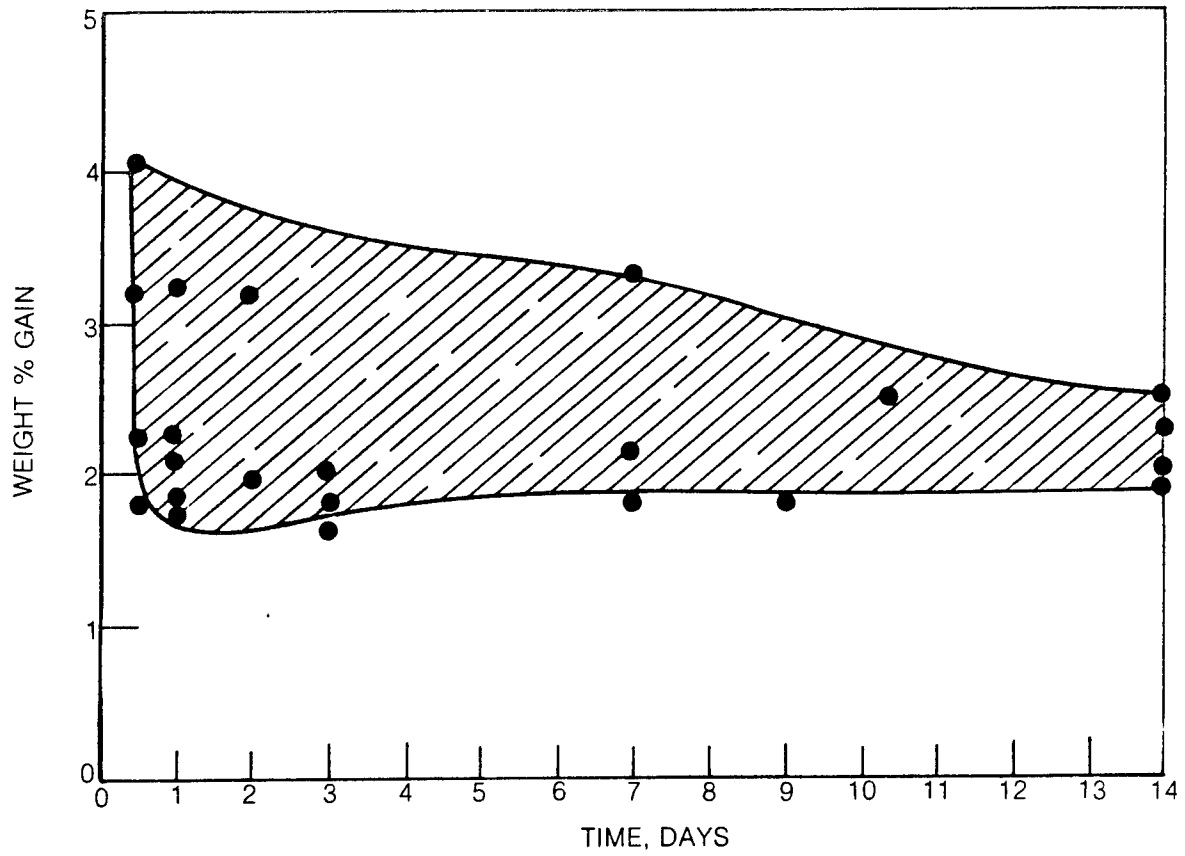
MOISTURE DESORPTION - ABSORPTION BEHAVIOR OF AS/3501-6 PREPREG
AT 31% R.H., 25°C



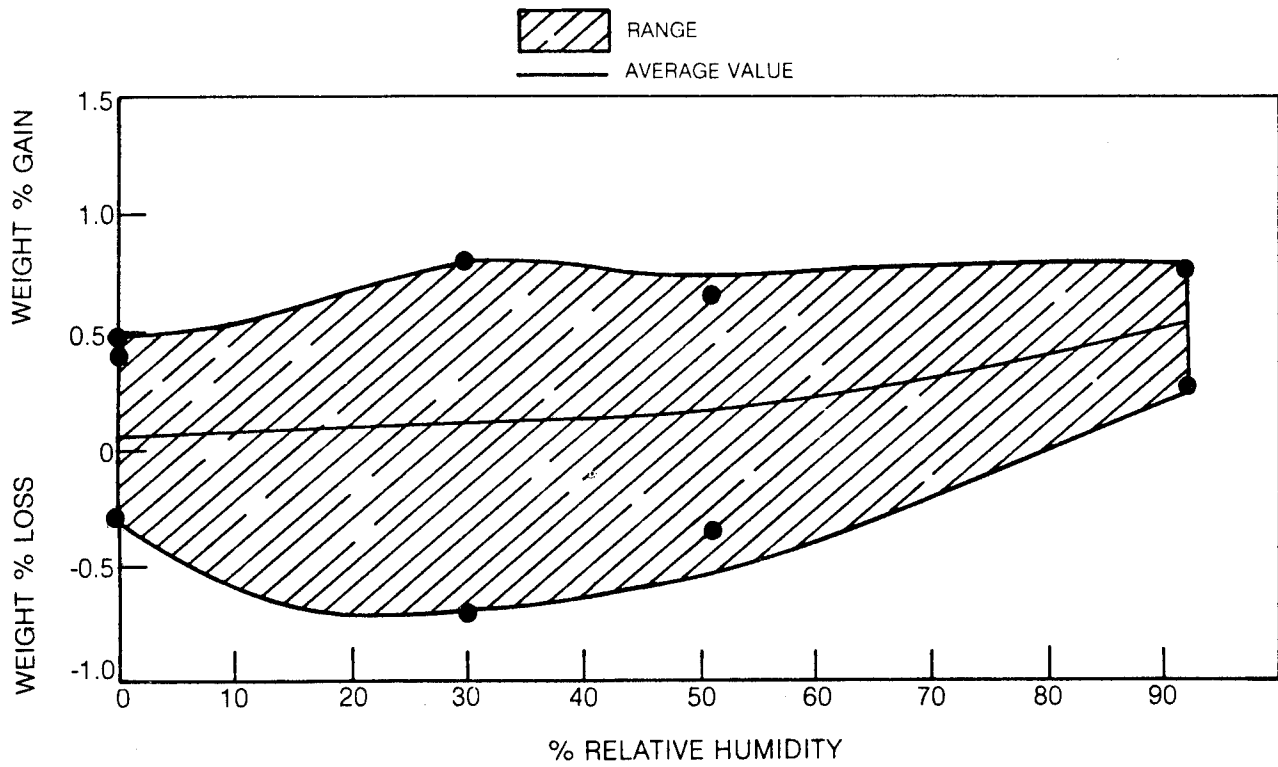
MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF AS/3501-6 PREPREG
AT 51% R.H., 25°C



MOISTURE ABSORPTION BEHAVIOR OF AS/3501-6 PREPREG AT 93% R.H., 25°C

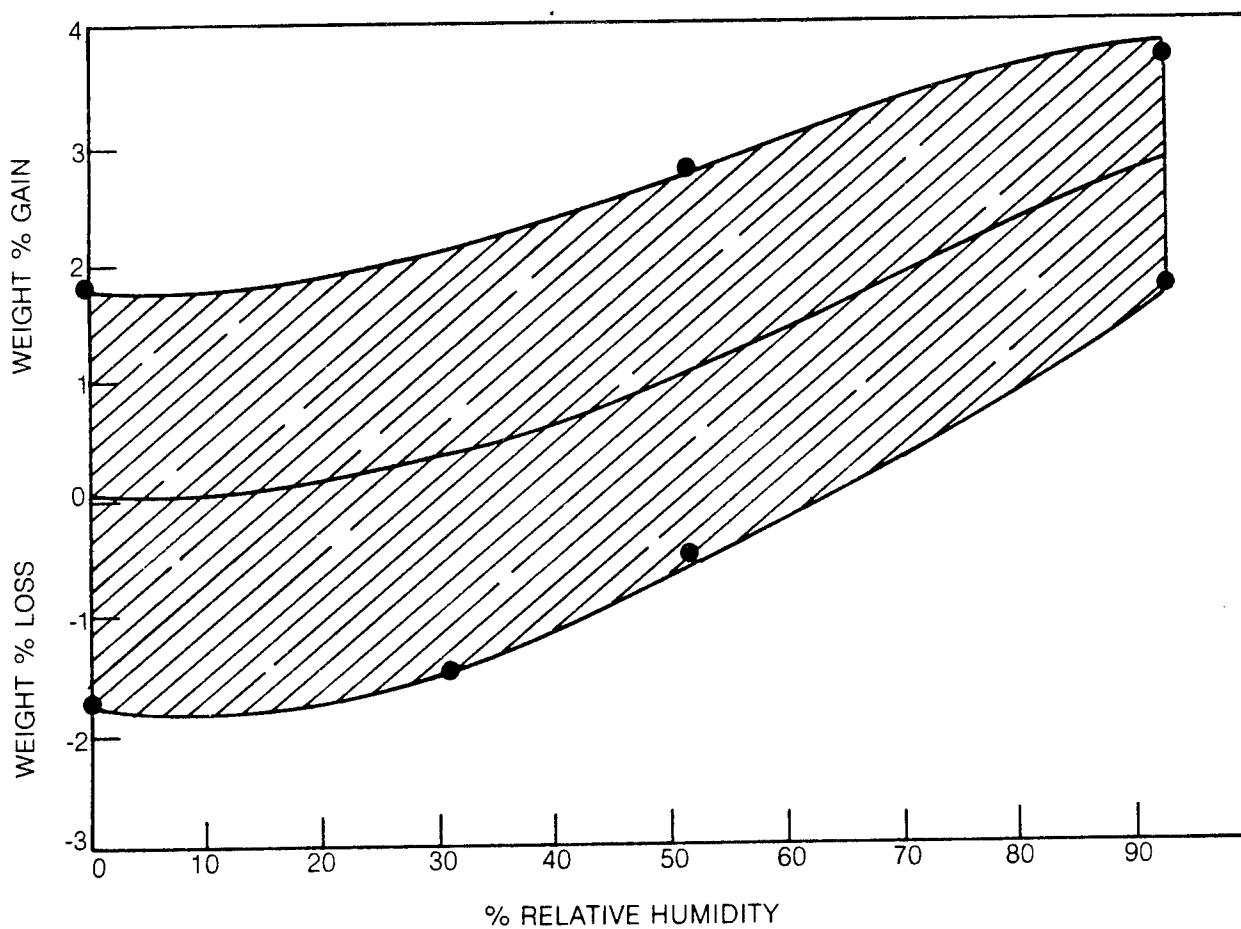


MOISTURE DESORPTION-ABSORPTION BEHAVIOR OF 3501-6 EPOXY RESIN AT 25°C, 24 HOURS



MOISTURE DESORPTION — ABSORPTION CAPABILITIES OF AS/3501-6 PREPREG AT 25°C, 24 HOURS

 RANGE
 AVERAGE VALUE



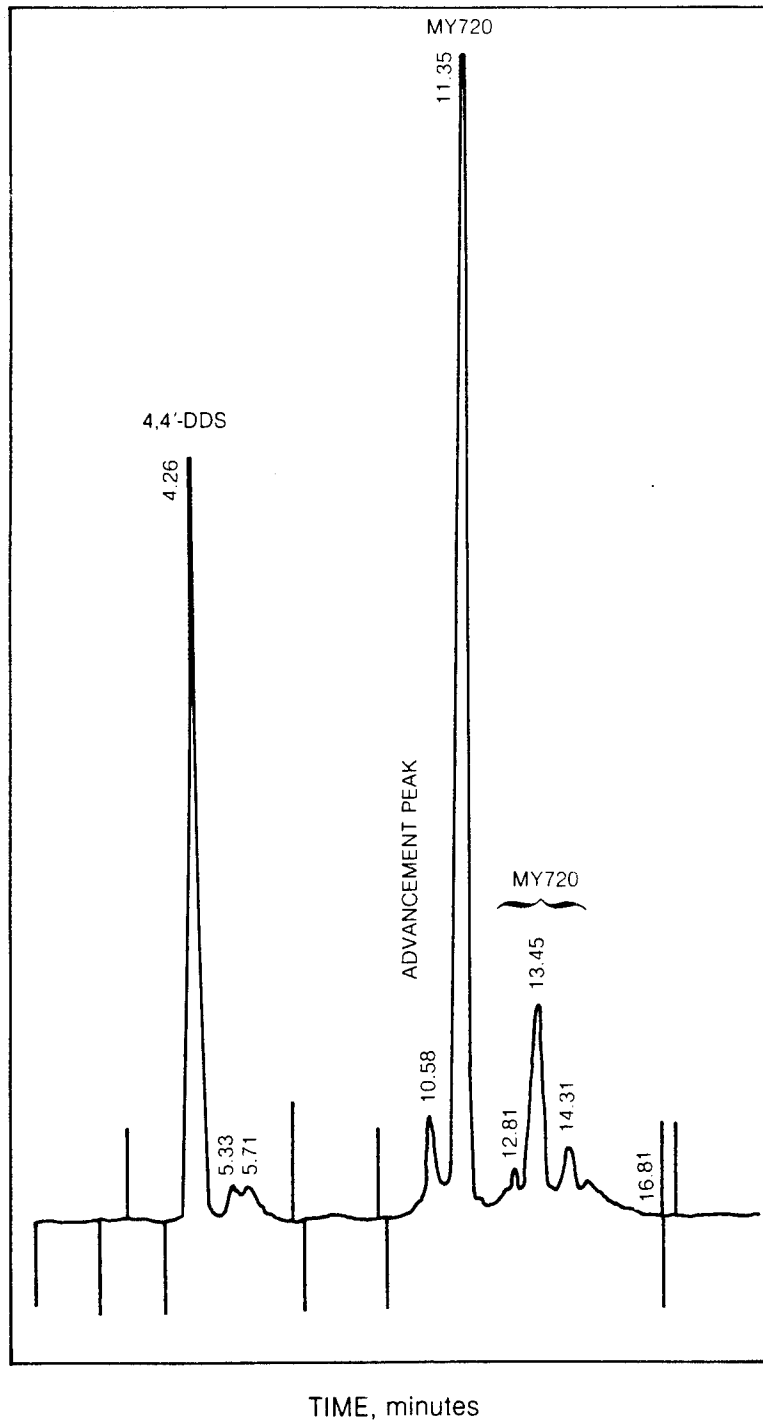
HPLC CHROMATOGRAM OF "AS-IS" 3501-6 EPOXY RESIN

CONDITIONS

COLUMN: μ BONDAPAK C₁₈
 SAMPLE SIZE: 10 μ l
 ATTENUATION: UV 1.0
 CHART SPEED: 0.5 cm/min
 INSTRUMENT: WATERS MODEL ALC/GPC-244

PROGRAM:

TOTAL TIME, min	FLOW cc/min	% CH ₃ CN	% H ₂ O	CURVE
0	1.20	50	50	—
4	1.20	50	50	6
7	1.20	100	0	6



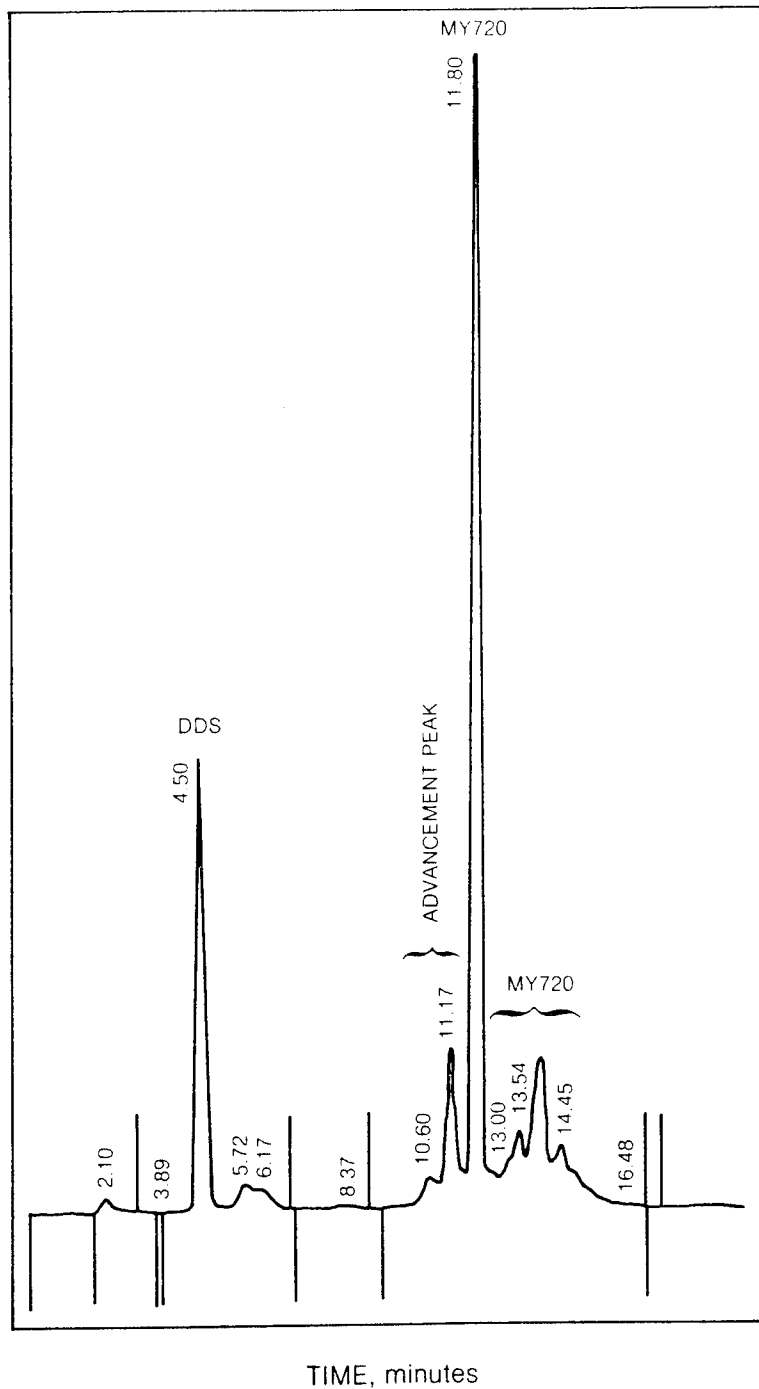
HPLC CHROMATOGRAM OF ADVANCED 3501-6 EPOXY RESIN

CONDITIONS

COLUMN: μ BONDAPAK C₁₈
 SAMPLE SIZE: 10 μ l
 ATTENUATION: UV 1.0
 CHART SPEED: 0.5 cm/min
 INSTRUMENT: WATERS MODEL ALC/GPC-244

PROGRAM

TOTAL TIME, min	FLOW cc/min	% CH ₃ CN	% H ₂ O	CURVE
0	1.20	50	50	—
4	1.20	50	50	6
7	1.20	100	0	6



HPLC CHROMATOGRAM OF "AS-IS" 3501-6 EPOXY RESIN

PROGRAM:

TOTAL TIME, min	FLOW cc/min	% THF	% H ₂ O	CURVE
INITIAL	1.20	20	80	—
5.00	1.20	20	80	6
45.00	1.20	60	40	6
55.00	1.20	60	40	6
70.00	1.20	20	80	6

CONDITIONS:

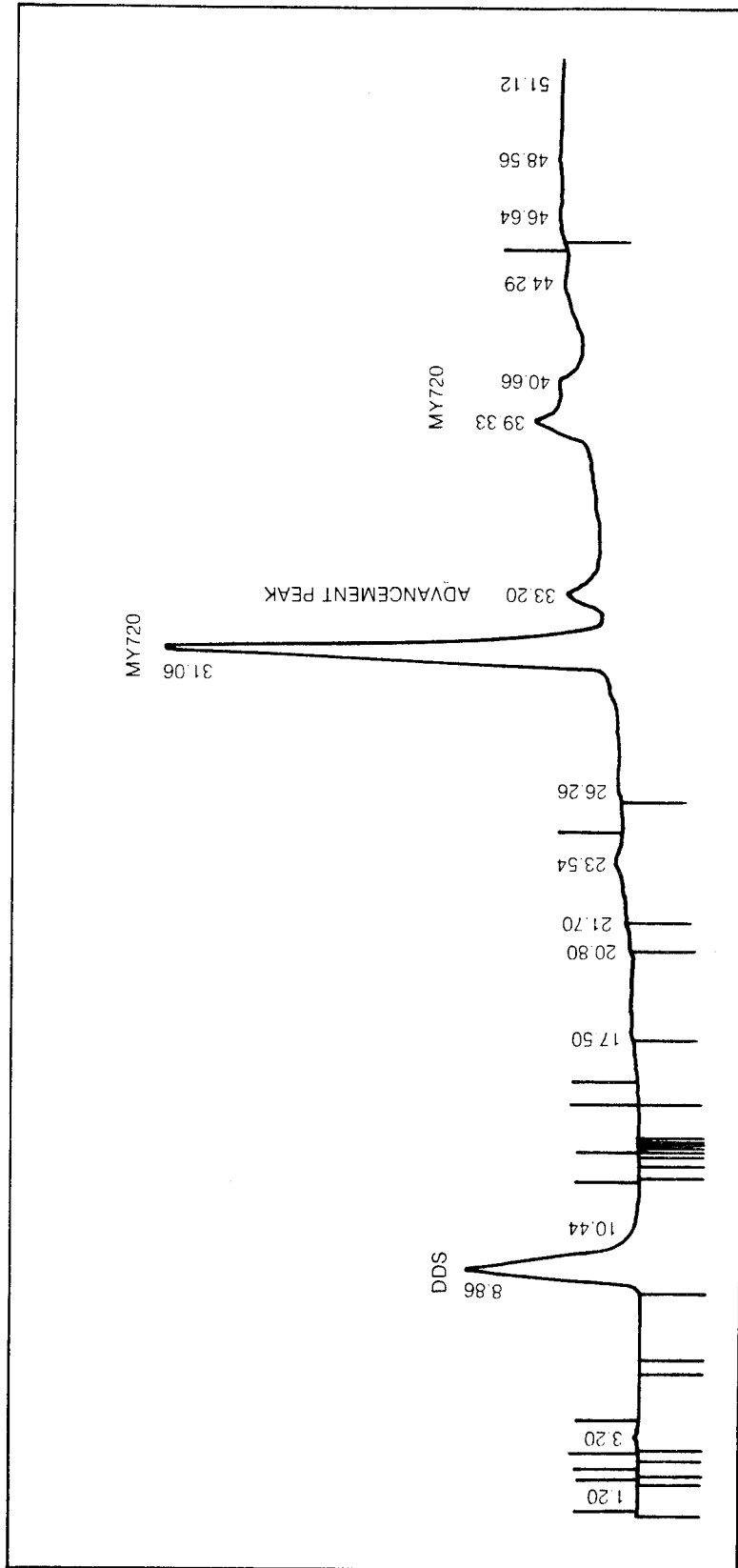
COLUMN: μ BONDAPAK C₁₈

SAMPLE SIZE: 15 μ l

ATTENUATION: UV 1.0

CHART SPEED: 0.5 cm/min

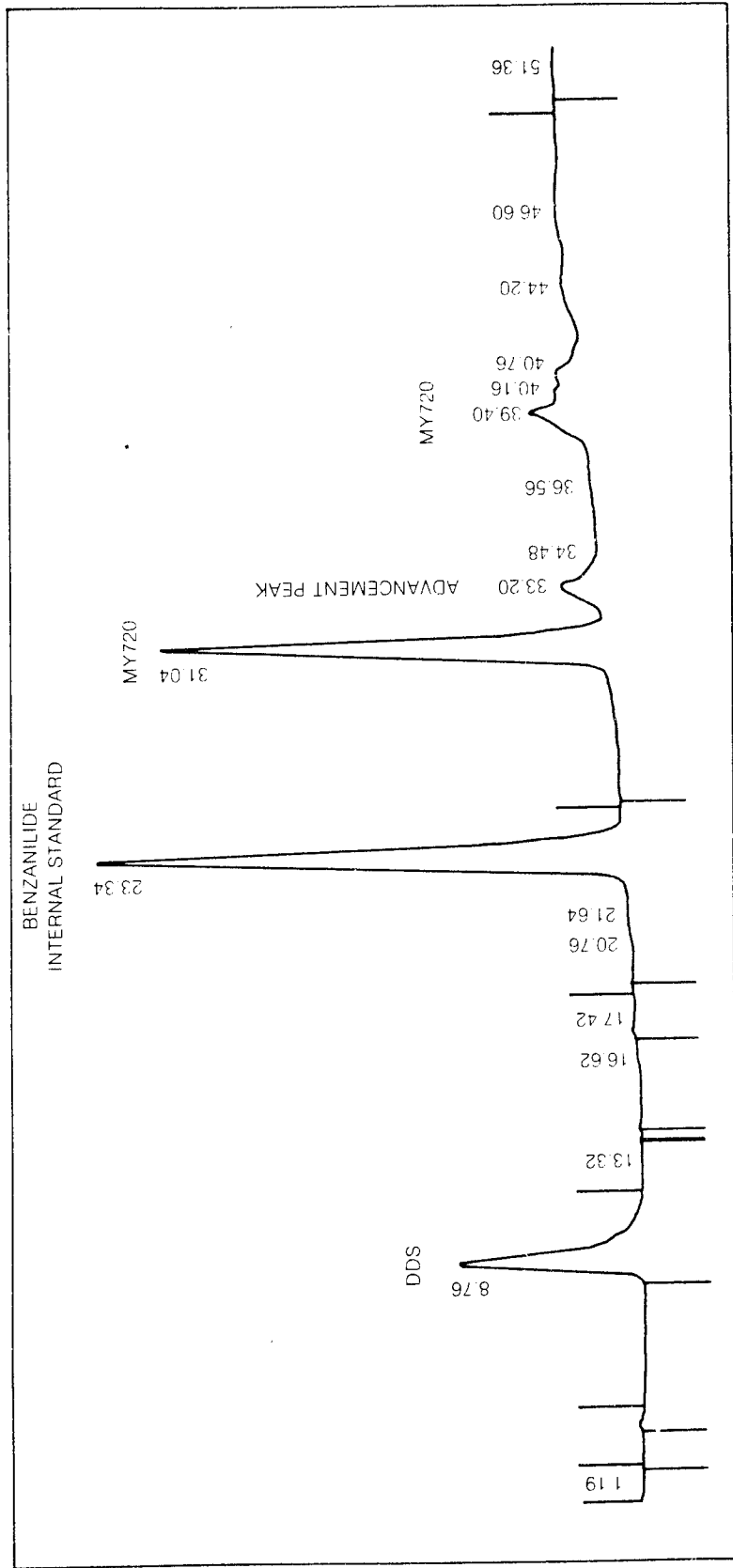
INSTRUMENT: WATERS MODEL ALC/GPC-244



HPLC CHROMATOGRAM OF "AS-IS" 3501-6 + INTERNAL STANDARD

PROGRAM			
TOTAL TIME, min	FLOW cc/min	% THF	% H ₂ O
INITIAL	1.20	20	80
5.00	1.20	20	80
45.00	1.20	60	40
55.00	1.20	60	40
70.00	1.20	20	80

CONDITIONS	
COLUMN	μ BONDAPAK C ₁₈
SAMPLE SIZE	15 μl
ATTENUATION	UV 1.0
CHART SPEED	0.5 cm/min
INSTRUMENT	WATERS MODEL ALC/GPC-244



HPLC CHROMATOGRAM OF ADVANCED 3501-6 EPOXY RESIN

CONDITIONS:

COLUMN: μ BONDAPAK C₁₈

SAMPLE SIZE: 15 μ l

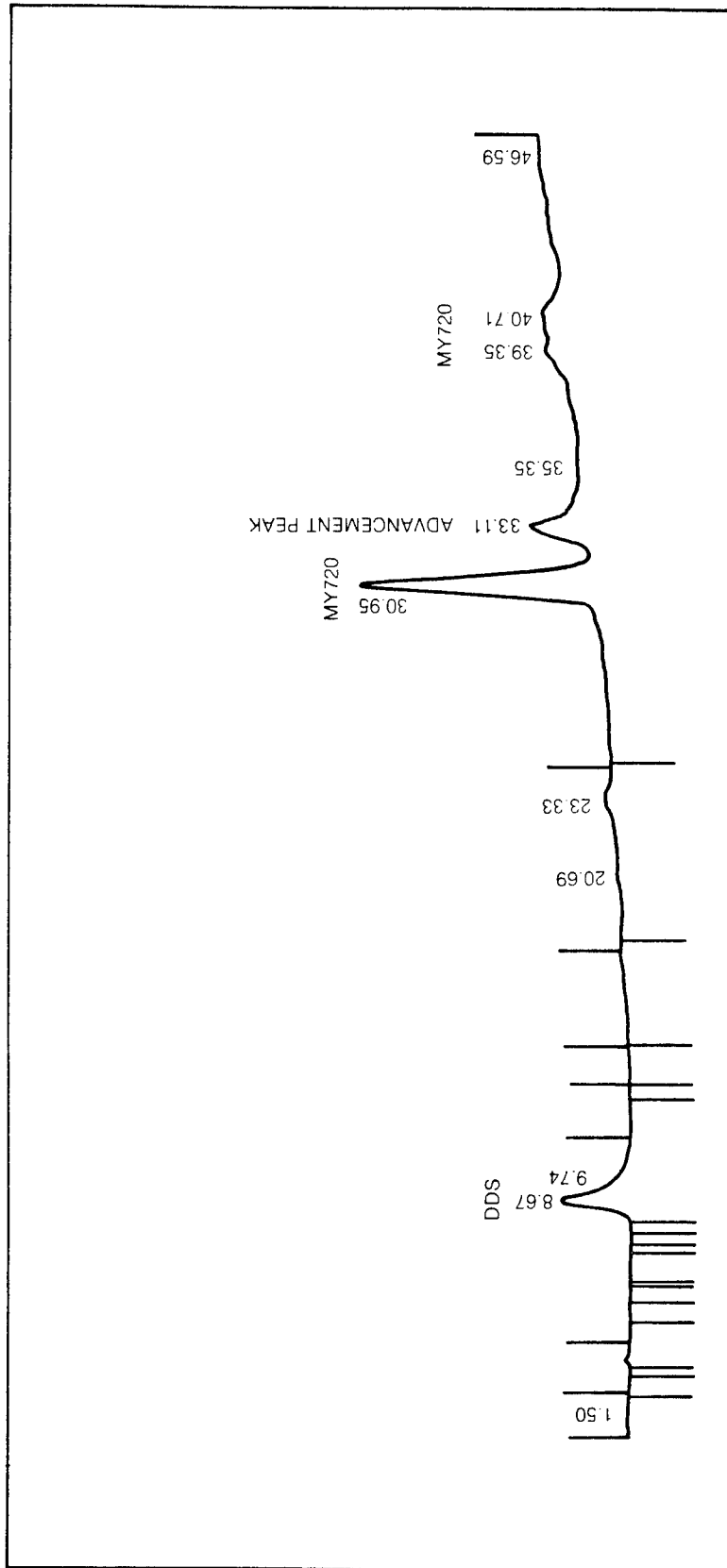
ATTENUATION: UV 1.0

CHART SPEED: 0.5 cm/min

INSTRUMENT: WATERS MODEL ALC/GPC-244

PROGRAM:

TOTAL TIME, min	INITIAL	FLOW cc/min	% THF	% H ₂ O	CURVE
5.00	1.20	1.20	20	80	---
45.00	1.20	1.20	20	80	6
55.00	1.20	1.20	60	40	6
70.00	1.20	1.20	60	40	6



TIME, minutes

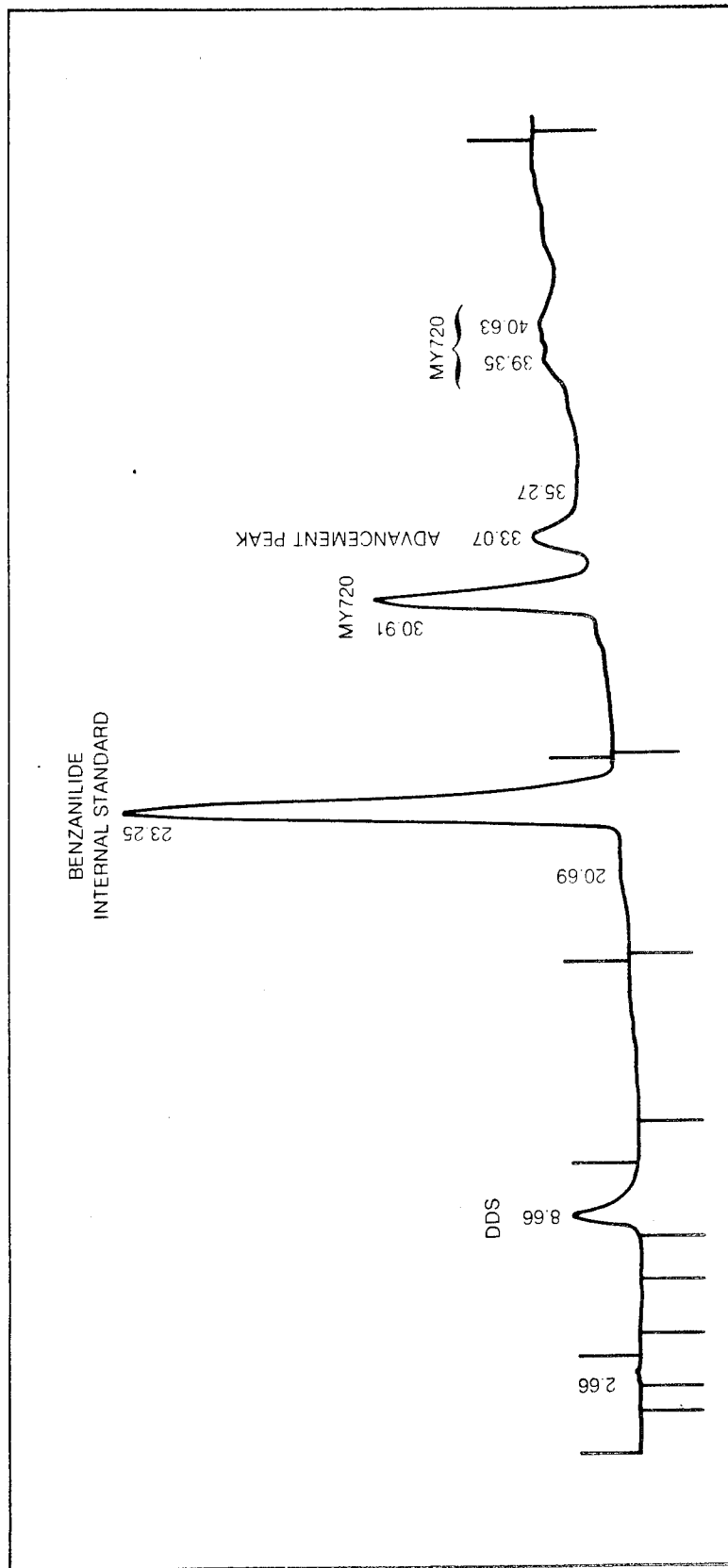
HPLC CHROMATOGRAM OF ADVANCED 3501-6 + INTERNAL STANDARD

CONDITIONS:

COLUMN: μ BONDAPAK C₁₈
 SAMPLE SIZE: 15 μ l
 ATTENUATION: UV 1.0
 CHART SPEED: 0.5 cm/min
 INSTRUMENT: WATERS MODEL ALC/GPC-244

PROGRAM:

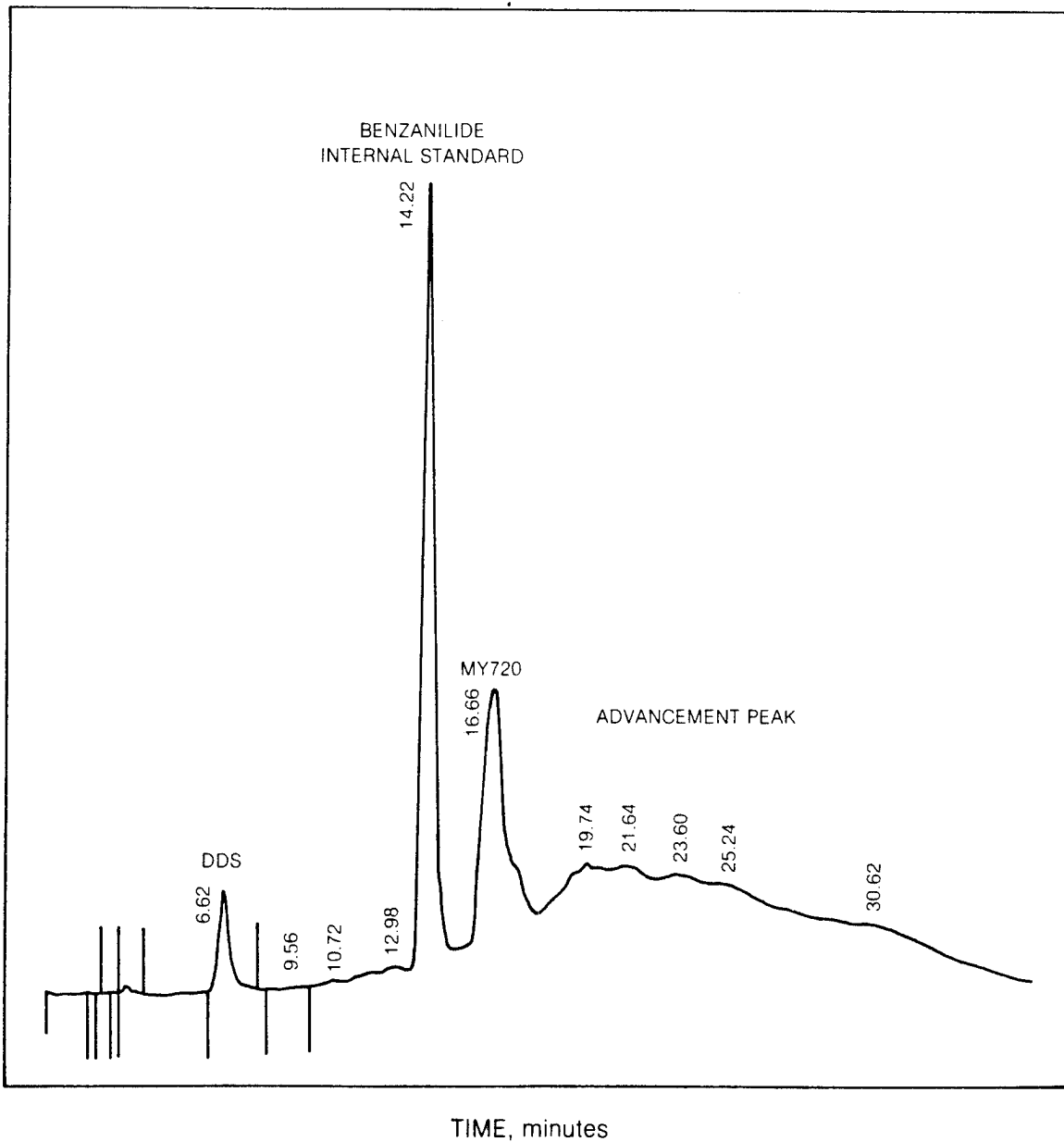
TOTAL TIME, min	FLOW, cc/min	% THF	% H ₂ O	CURVE
INITIAL	1.20	20	80	—
5.00	1.20	20	80	6
45.00	1.20	60	40	6
55.00	1.20	60	40	6
70.00	1.20	60	80	6



TIME, minutes

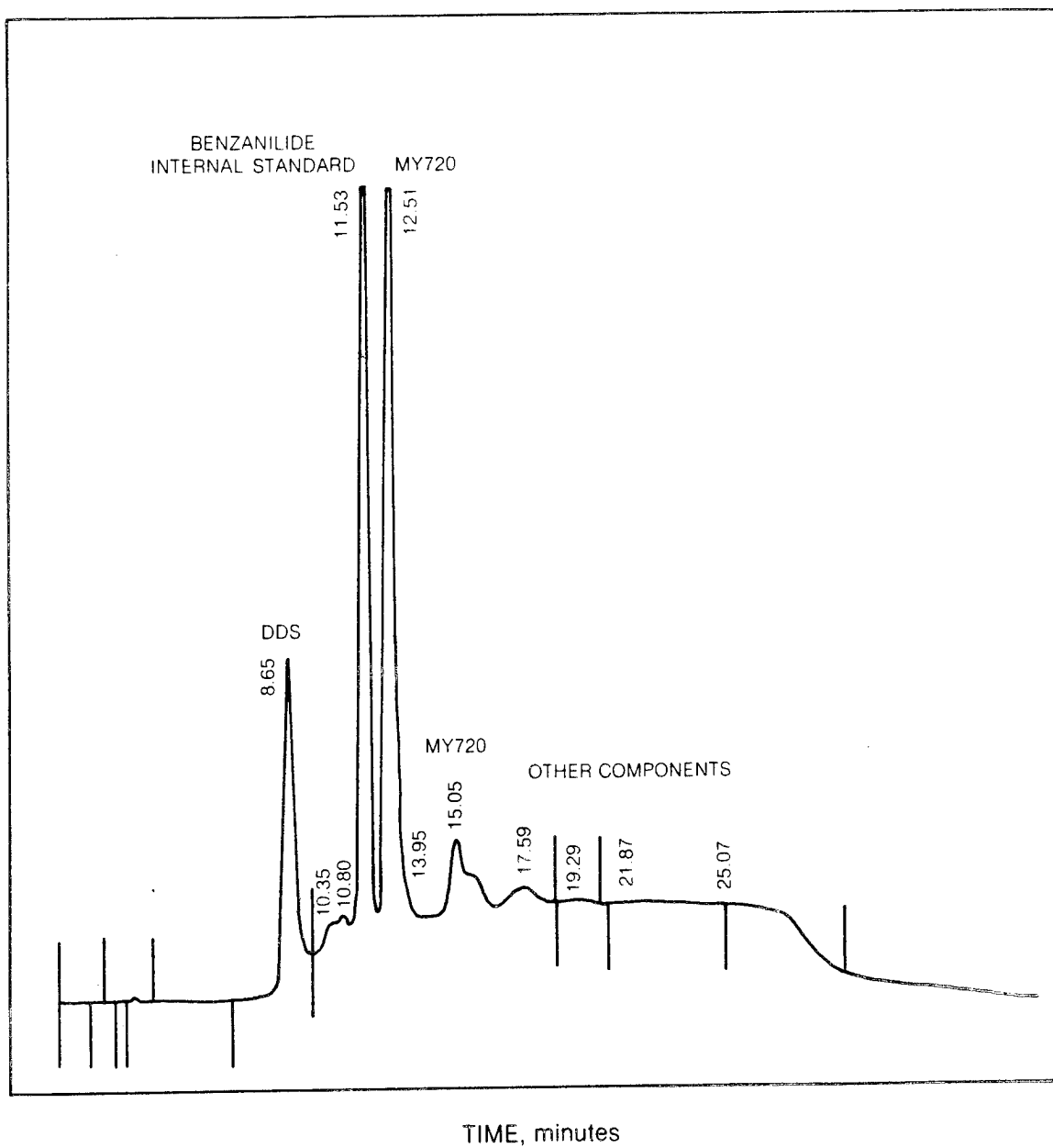
**HPLC CROMATOGRAM OF ADVANCED 3501-6 + INTERNAL STANDARD
SHOWING EFFECT OF SOLVENT COMPOSITION**

CONDITIONS	PROGRAM:				
	TOTAL TIME, min	FLOW cc/min	% THF	% H ₂ O	CURVE
COLUMN: μ BONDAPAK C ₁₈	INITIAL	1.20	25	75	—
SAMPLE SIZE: 15 μ l	2.00	1.20	25	75	6
ATTENUATION: UV 1.0	22.00	1.20	75	25	6
CHART SPEED: 0.5 cm/min	32.00	1.20	25	75	6
INSTRUMENT: WATERS MODEL ALC/GPC-244					



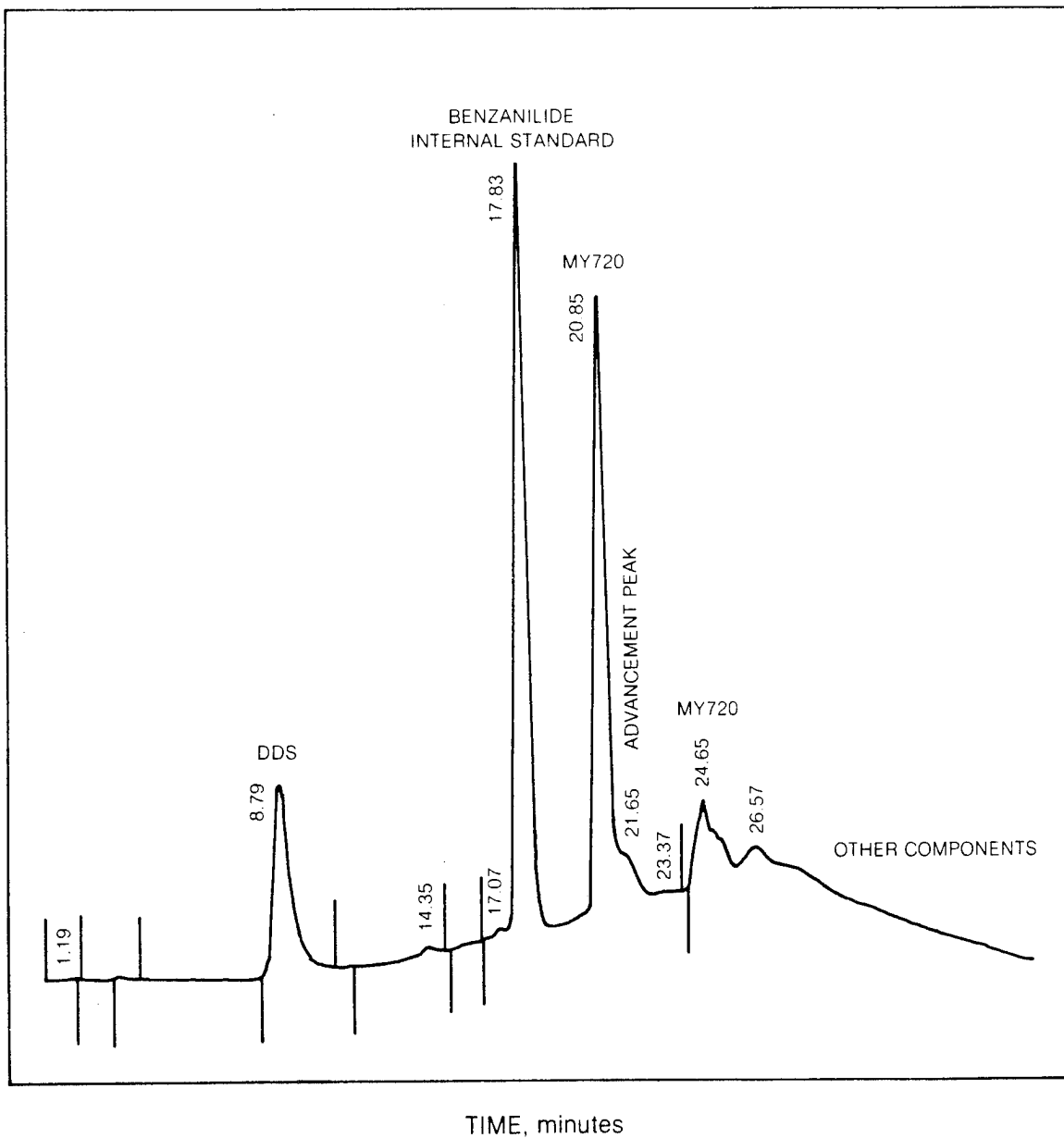
HPLC CHROMATOGRAM OF "AS-IS" 3501-6 + INTERNAL STANDARD SHOWING EFFECT OF RATE OF SOLVENT GRADIENT CHANGE

CONDITIONS	PROGRAM:				
	TOTAL TIME, min	FLOW cc/min	% THF	% H ₂ O	CURVE
	INITIAL	1.20	20	80	—
COLUMN: μ BONDAPAK C ₁₈	2.00	1.20	20	80	2
SAMPLE SIZE: 15 μ l	22.00	1.20	60	40	2
ATTENUATION: UV 1.0	32.00	1.20	20	80	2
CHART SPEED: 0.5 cm/min	37.00	1.20	20	80	2
INSTRUMENT: WATERS MODEL ALC/GPC-244	40.00	1.20	30	70	6



**HPLC CHROMATOGRAM OF "AS-IS" 3501-6 + INTERNAL STANDARD
SHOWING EFFECTS OF GRADIENT TIME CHANGE**

CONDITIONS	PROGRAM				
	TOTAL TIME, min	FLOW cc/min	% THF	% H ₂ O	CURVE
COLUMN: μ BONDAPAK C ₁₈	INITIAL	1.20	20	80	—
SAMPLE SIZE: 15 μ l	2.00	1.20	20	80	6
ATTENUATION: UV 1.0	22.00	1.20	60	40	6
CHART SPEED: 0.5 cm/min	32.00	1.20	20	80	6
INSTRUMENT: WATERS MODEL ALC/GPC-244					



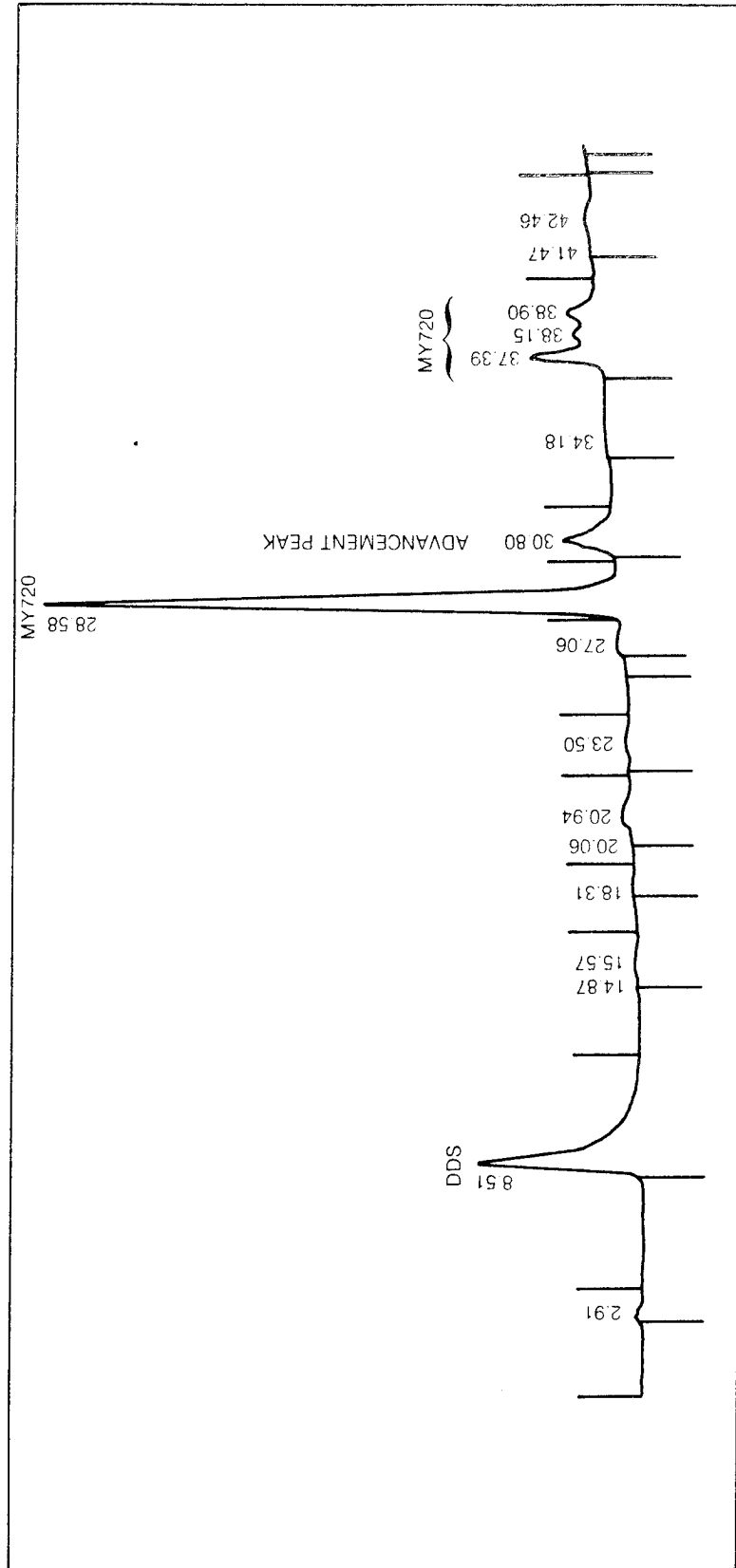
**HPLC CHROMATOGRAM OF "AS-IS" 3501-6 SHOWING EFFECTS OF
REDUCING INITIAL AND FINAL RUNTIME**

CONDITIONS:

COLUMN: μ BONDAPAK C₁₈
 SAMPLE SIZE: 15 μ l
 ATTENUATION: UV 1.0
 CHART SPEED: 0.5 cm/min
 INSTRUMENT: WATERS MODEL ALC/GPC-244

PROGRAM:

TOTAL TIME, min	INITIAL	5.00	45.00	50.00	FLOW cc/min	% THF	% H ₂ O	CURVE
	1.20	1.20	1.20	1.20	1.20	20	80	—
					1.20	20	80	6
					1.20	60	40	6
					1.20	20	80	6



TIME, minutes

HPLC CHROMATOGRAM OF ADVANCED 3501-6 EPOXY RESIN

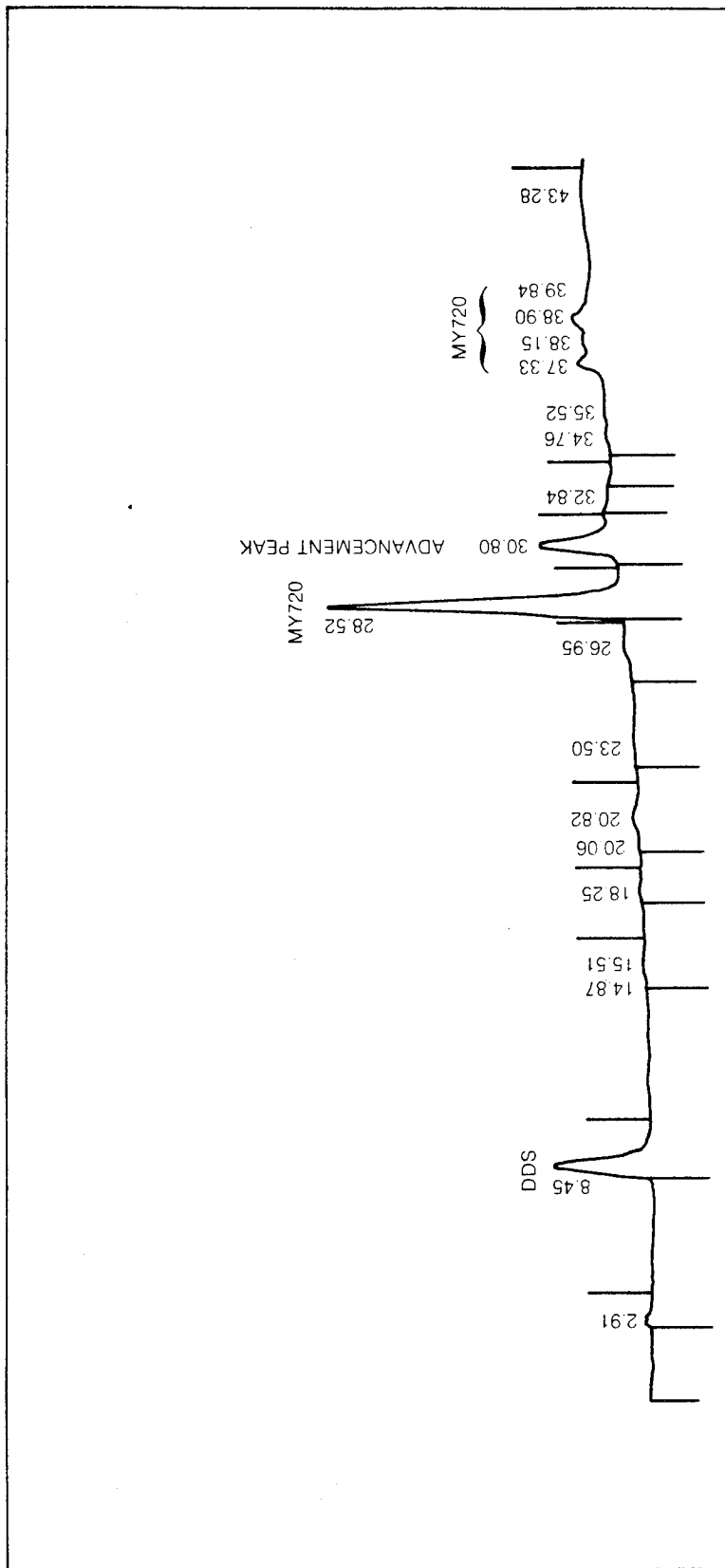
CONDITIONS:

COLUMN: μ BONDAPAK C₁₈
 SAMPLE SIZE: 15 μ l
 ATTENUATION: UV 1.0
 CHART SPEED: 0.5 cm/min
 INSTRUMENT: WATERS MODEL ALC/GPC-244

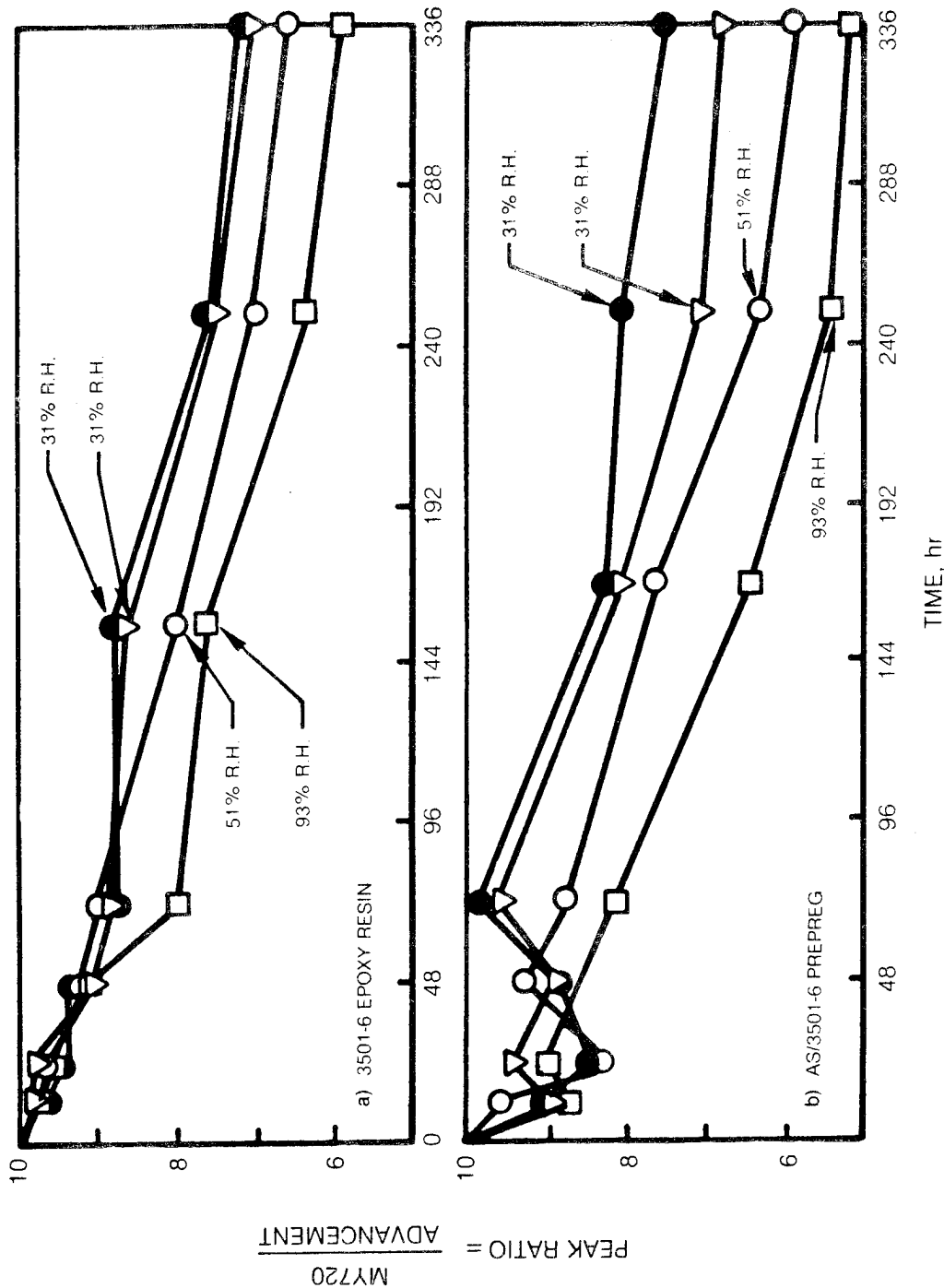
PROGRAM:			
TOTAL TIME, min	FLOW cc/min	% THF	% H ₂ O
INITIAL	1.20	20	80
5.00	1.20	20	80
45.00	1.20	60	40
50.00	1.20	20	80

CURVE

— 6 6 6



HPLC ANALYSIS OF 3501-6 EPOXY RESIN AND AS/3501-6 PREPREG



INFRARED ABSORPTION RATIO VS TIME OF HUMIDITY EXPOSURE OF 3501-6 EPOXY RESIN AT 25°C

FIG. 23

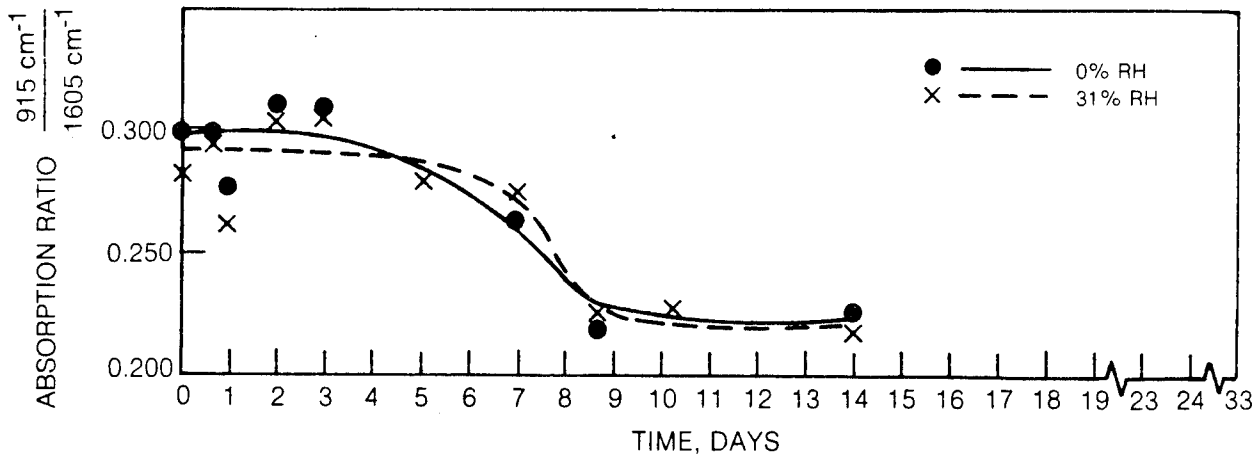
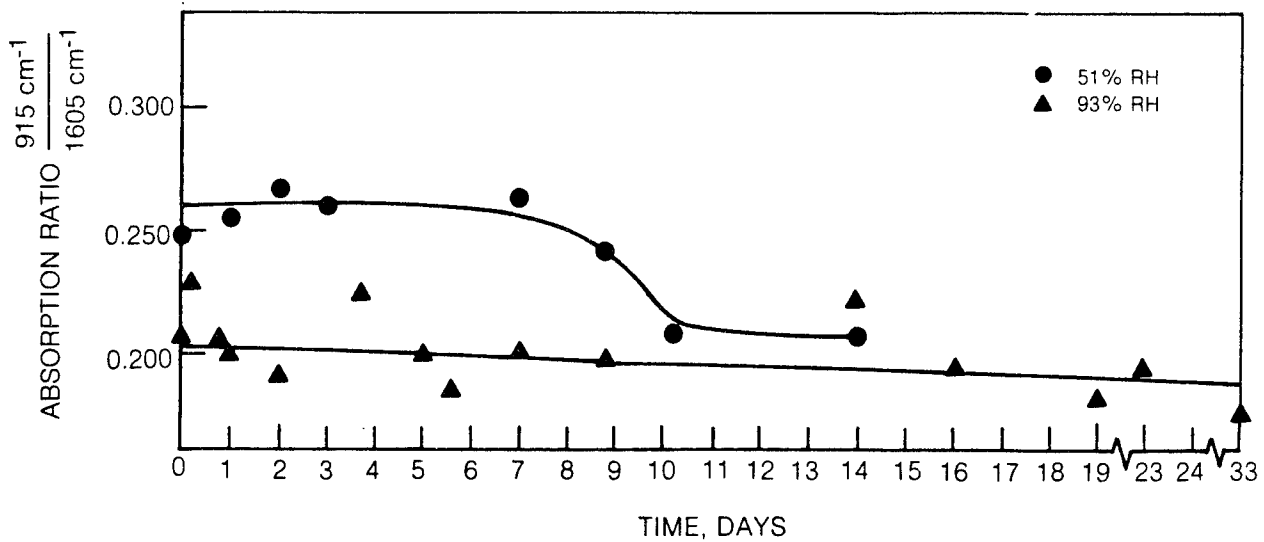


FIG. 24



INFRARED ABSORPTION RATIO VS TIME OF HUMIDITY EXPOSURE OF AS/3501-6 PREPREG AT 25°C

FIG. 25

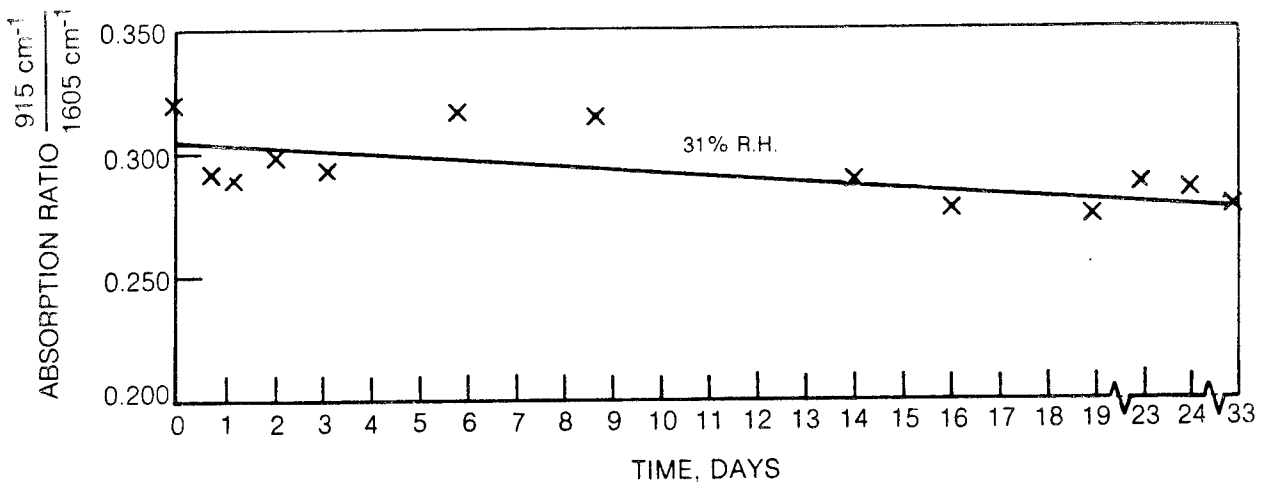
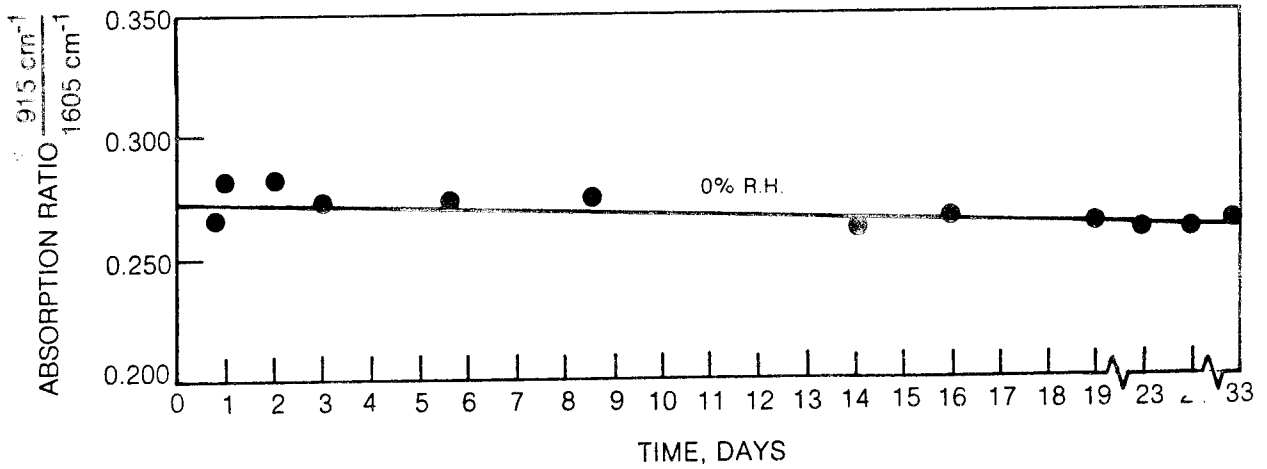


FIG. 26



INFRARED ABSORPTION RATIO VS TIME OF HUMIDITY EXPOSURE OF AS/3501-6 PREPREG AT 25°C

FIG. 27

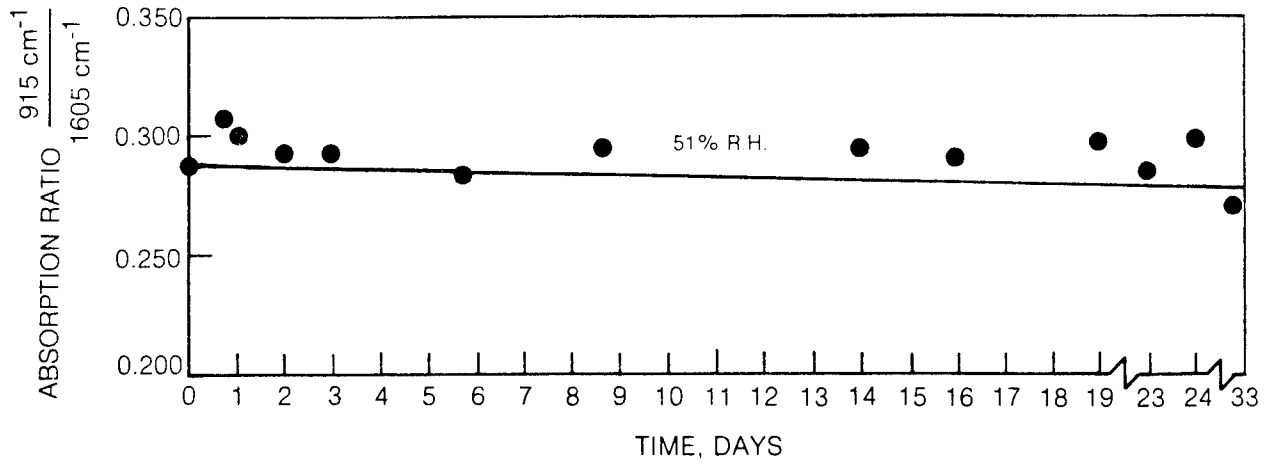
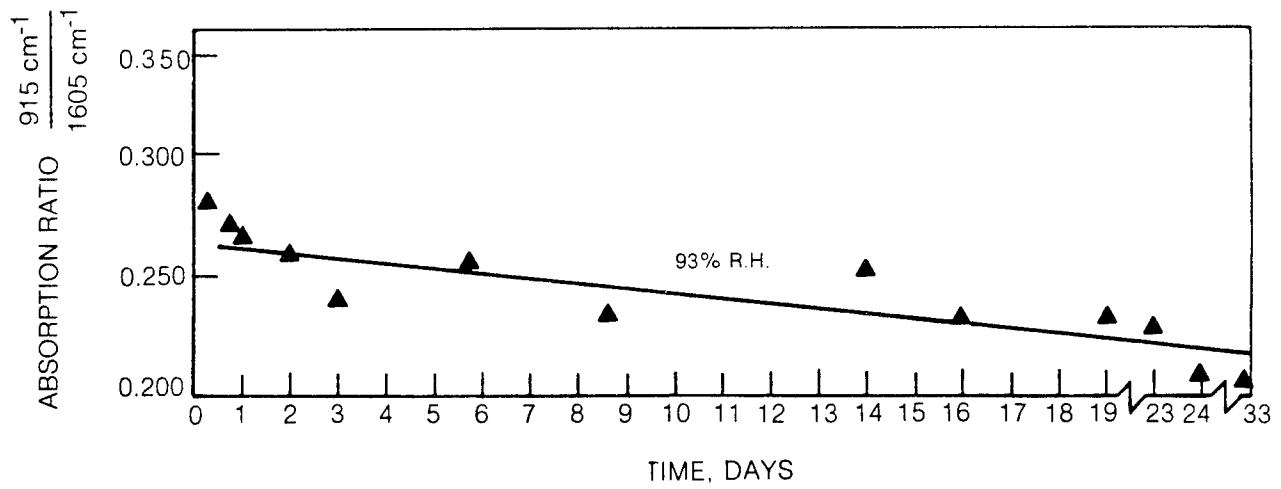
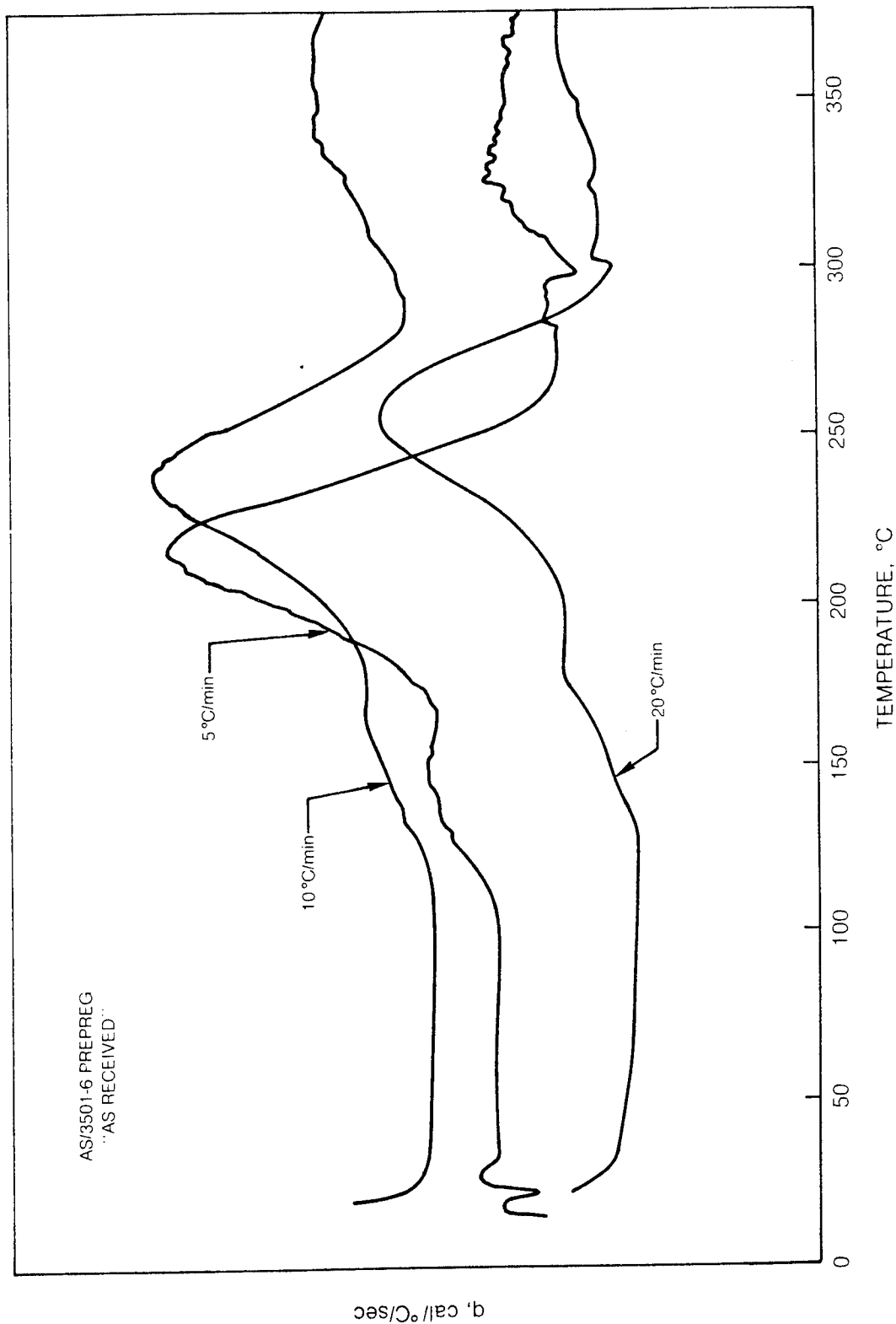


FIG. 28



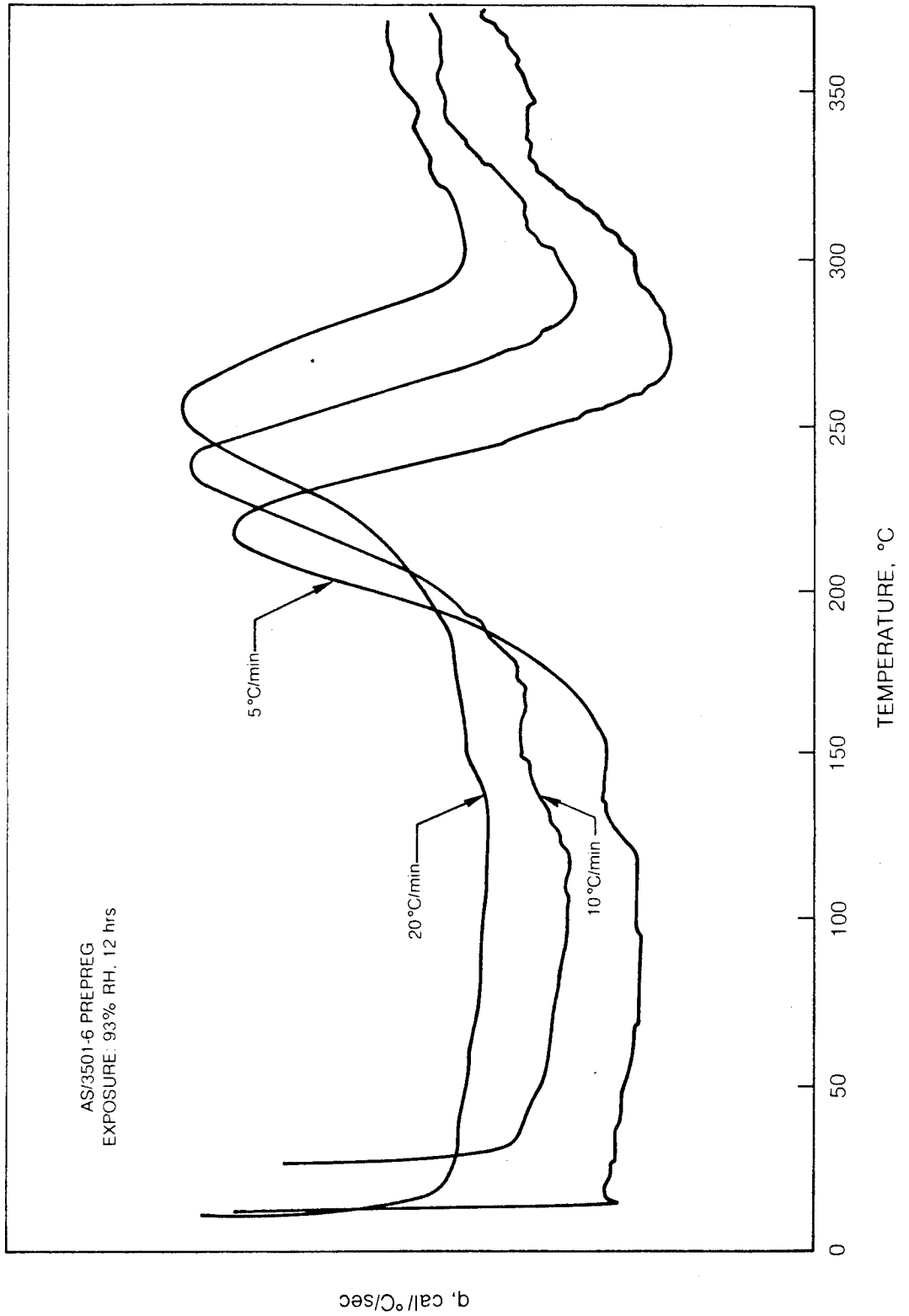
DSC THEROGRAM OF AS/3501-6 PREPREG

	WEIGHT, mg
SENSITIVITY AT 5 °C/min, 0.5 mcal/sec/in.	14.3
SENSITIVITY AT 10 °C/min, 1.0 mcal/sec/in.	13.7
SENSITIVITY AT 20 °C/min, 2.0 mcal/sec/in.	13.1



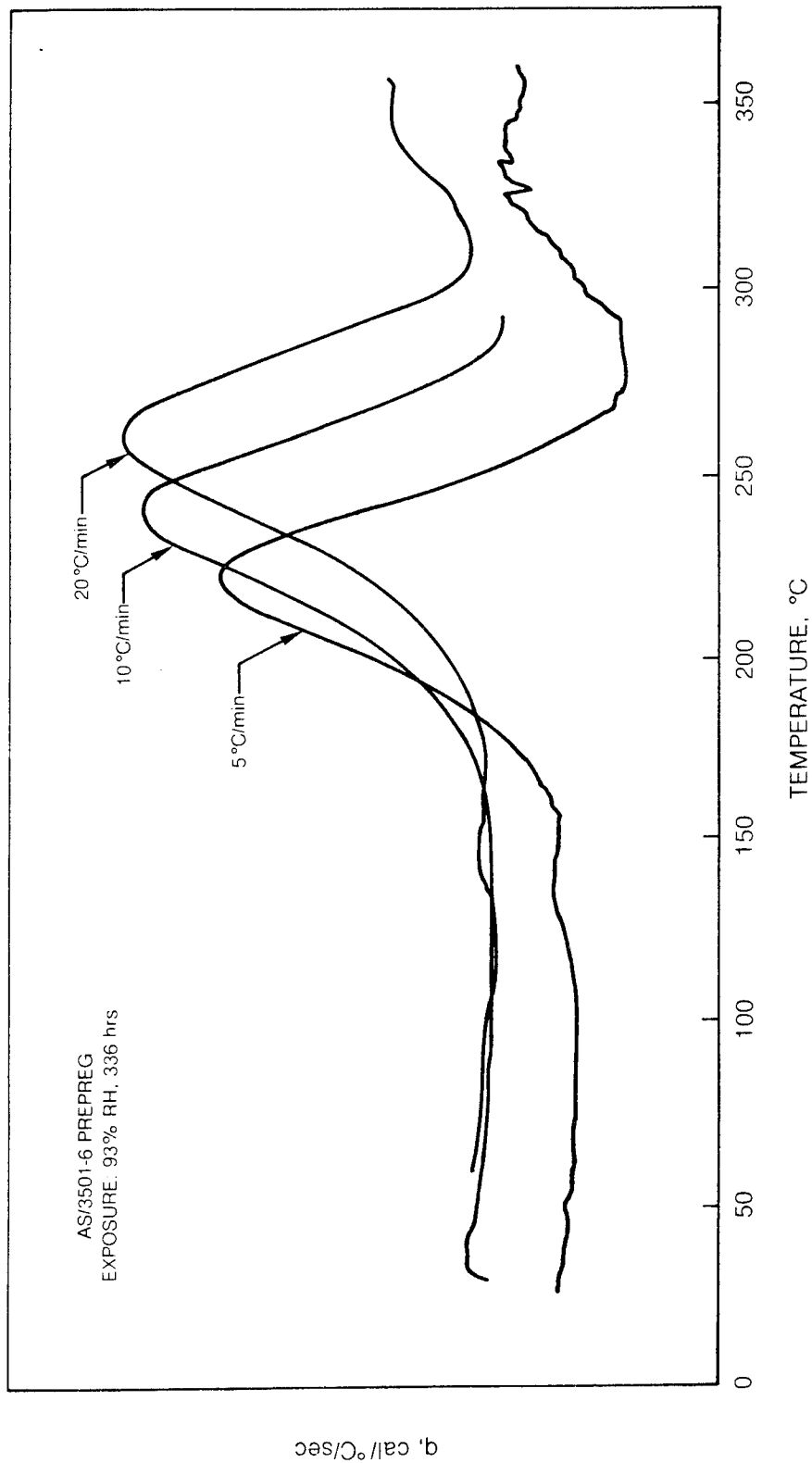
DSC THERMOGRAM OF AS/3501-6 PREPREG

	WEIGHT, mg
SENSITIVITY AT 5°C/min, 0.5 mcal/sec/in	17.0
SENSITIVITY AT 10°C/min, 1.0 mcal/sec/in.	17.8
SENSITIVITY AT 20°C/min, 2.0 mcal/sec/in.	13.6

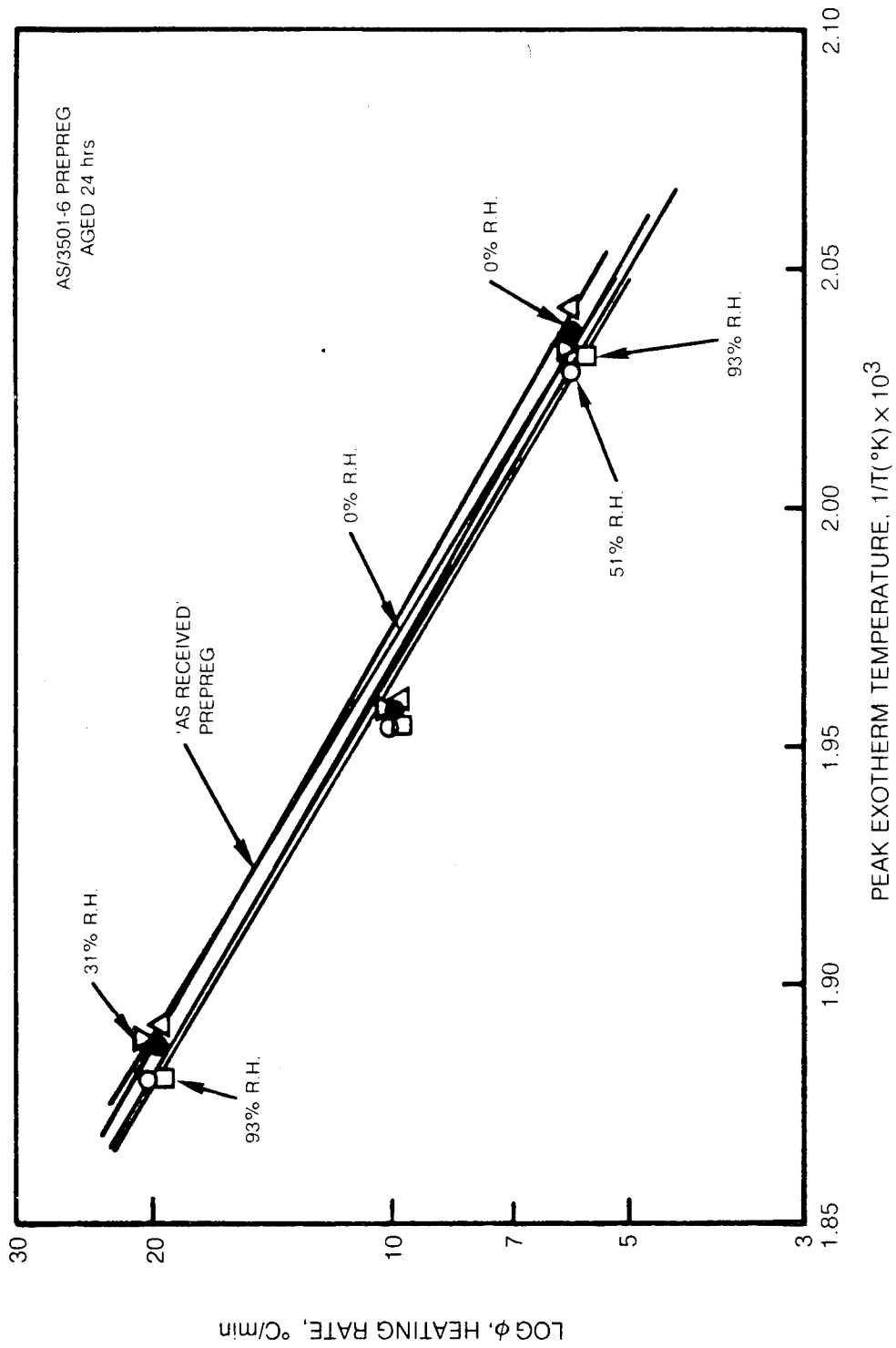


DSC THERMOGRAM OF AS/3501-6 PREPREG

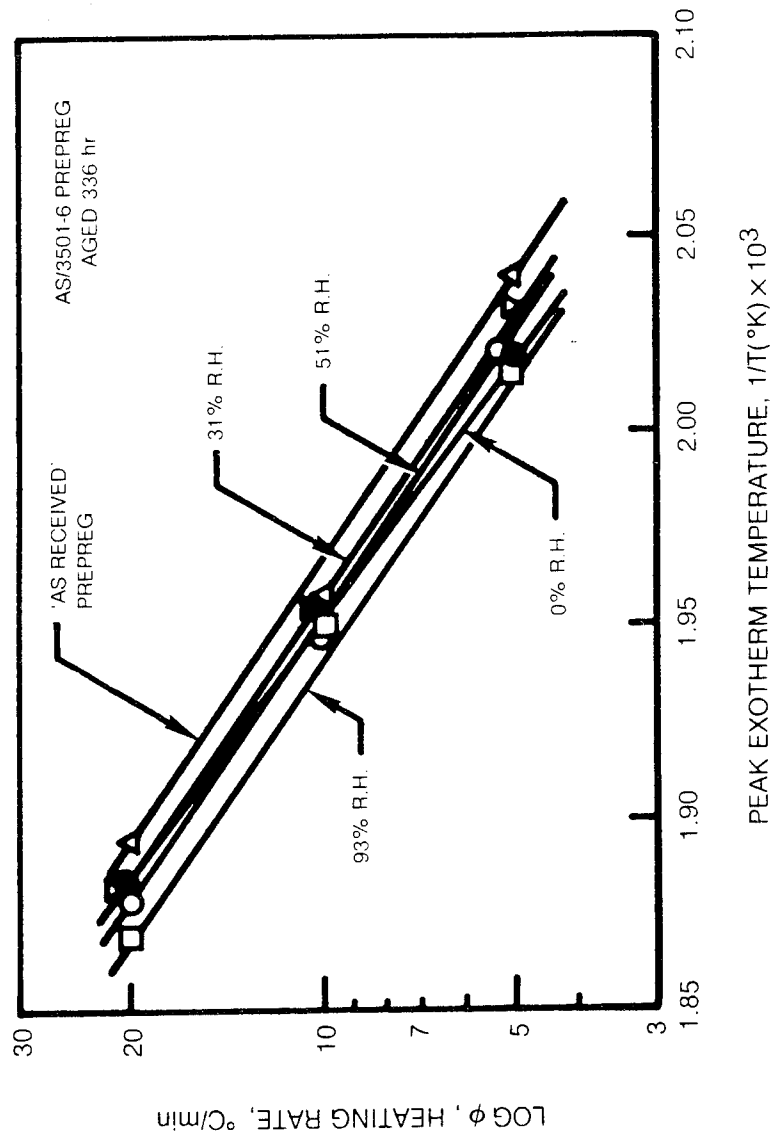
	WEIGHT, mg
SENSITIVITY AT 5°C/min, 0.5 mcal/sec/in.	14.7
SENSITIVITY AT 10°C/min, 1.0 mcal/sec/in.	14.9
SENSITIVITY AT 20°C/min, 2.0 mcal/sec/in.	15.0



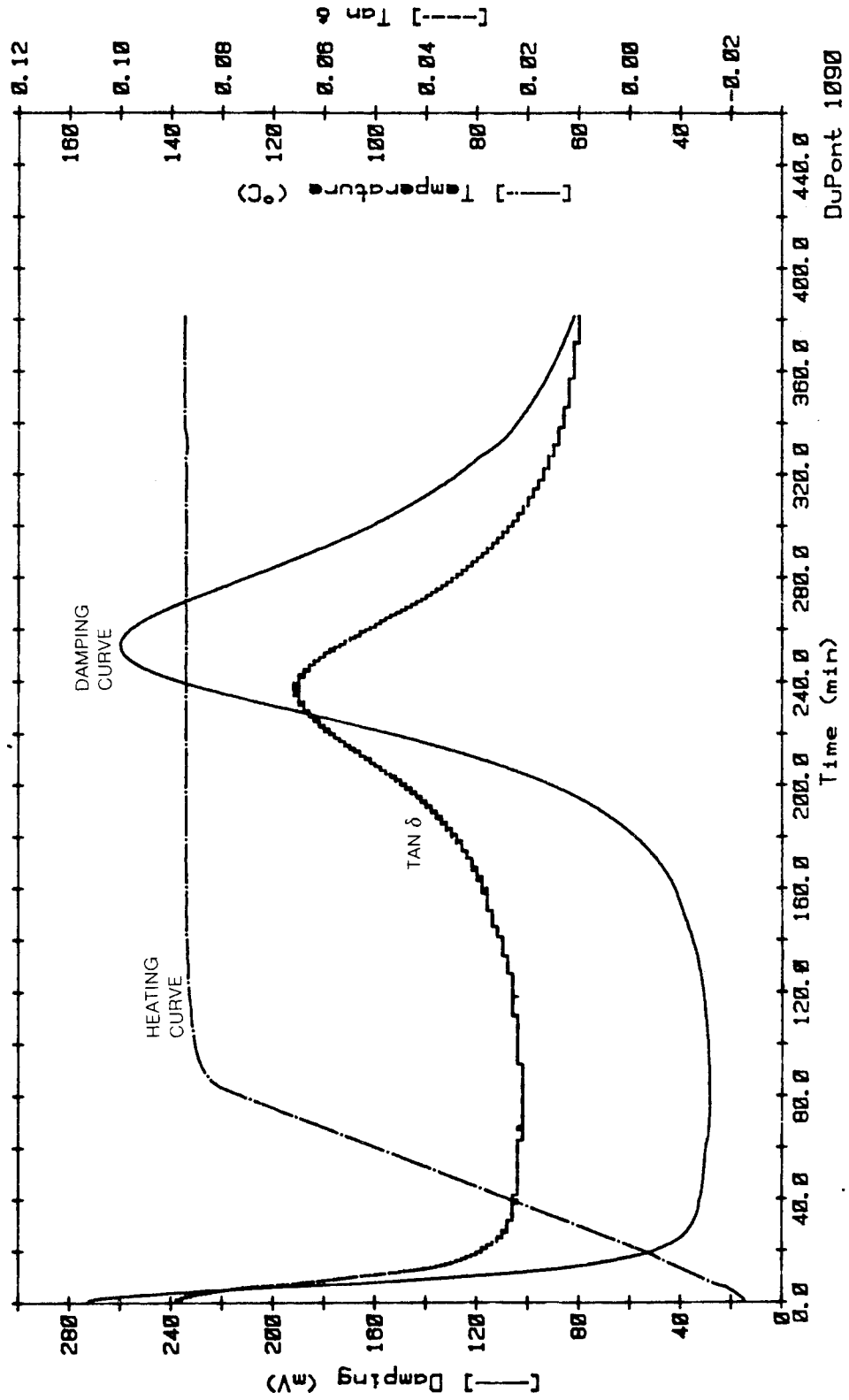
DSC PLOTS OF HEATING RATE VS PEAK EXOTHERM TEMPERATURE



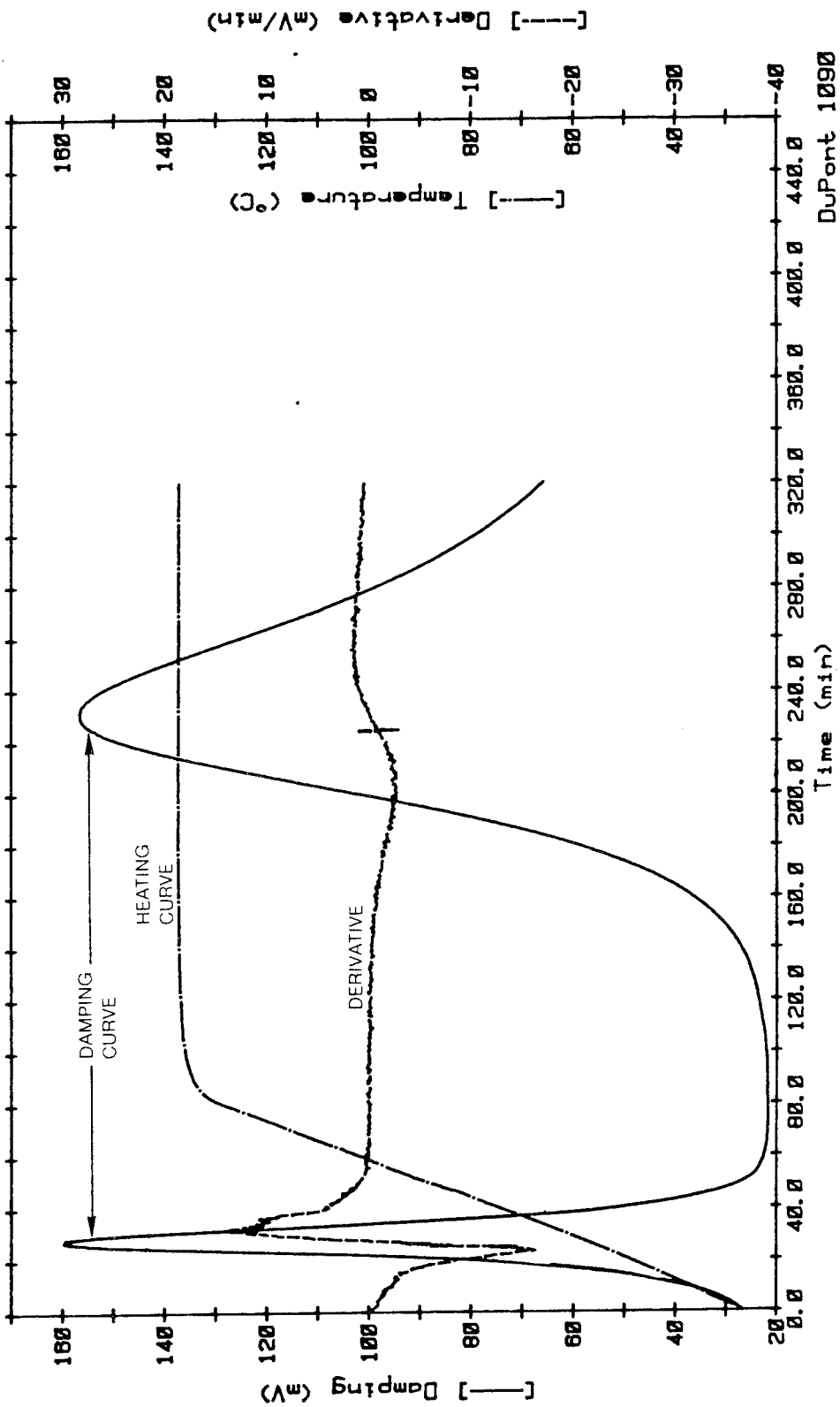
DSC PLOTS OF HEATING RATE VS PEAK EXOTHERM TEMPERATURE



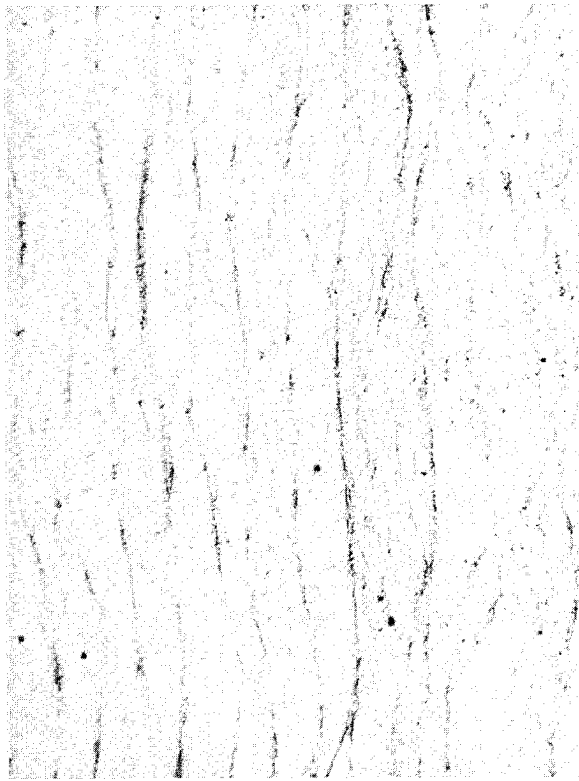
DMA OF "AS RECEIVED" AS/3501-6 PREPREG



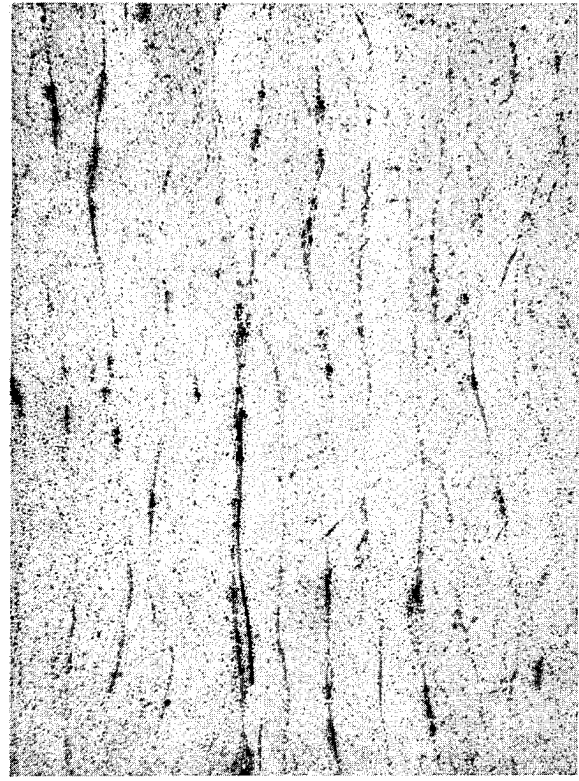
DMA OF AS/3501-6 PREPREG AFTER EXPOSURE TO 93% RH, 840 HRS (5 WKS)



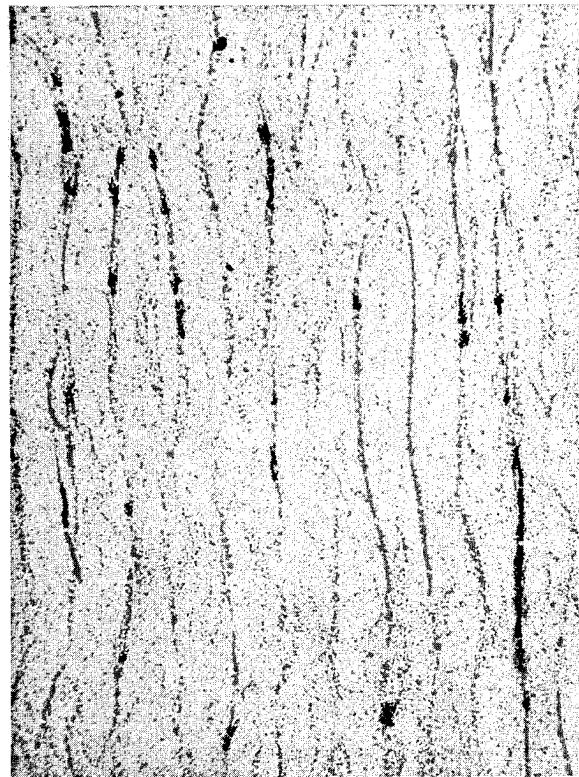
OPTICAL MICROGRAPHS OF TRANSVERSE CROSS SECTIONS OF AS/3501-6 COMPOSITES



a) NAV-19 "AS-RECEIVED" 200μm



b) NAV-21 PREPREG HUMID AGED
24 hrs, 31% RH 200μm

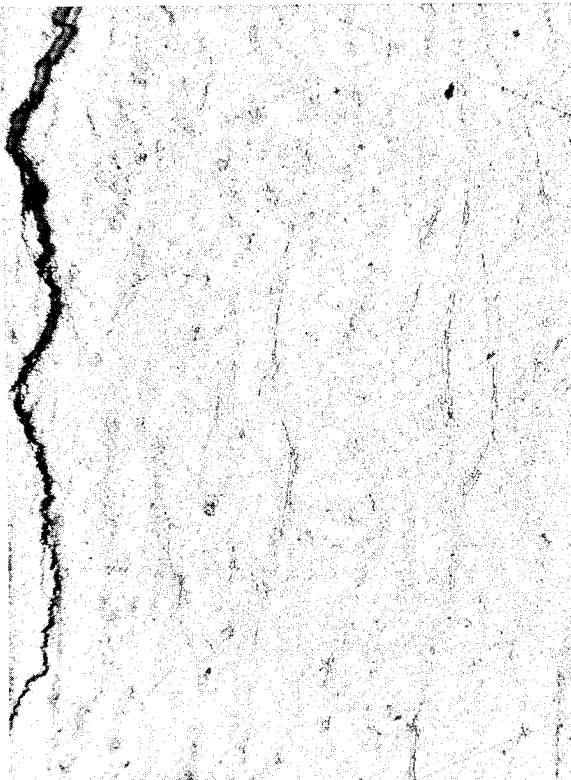


c) NAV-25 PREPREG HUMID AGED
24 hrs, 93% RH 200μm

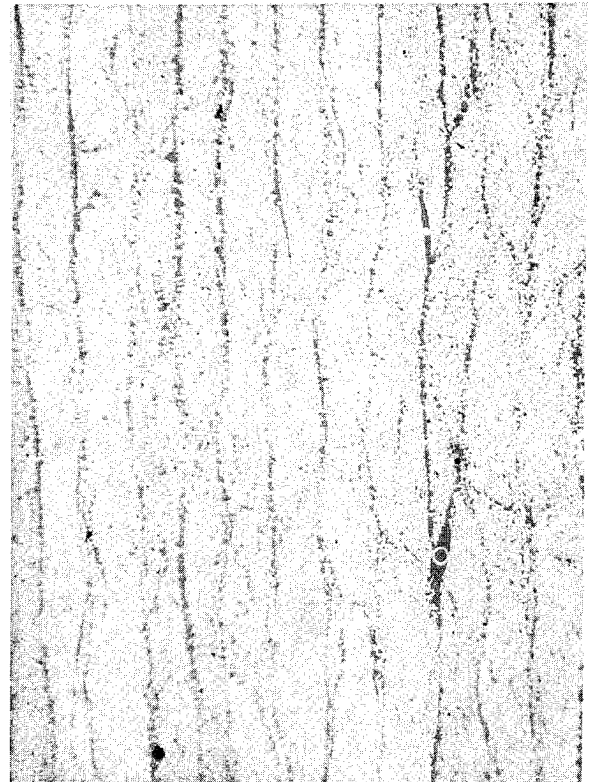


d) NAV-22 PREPREG HUMID AGED
72 hrs, 51% RH 200μm

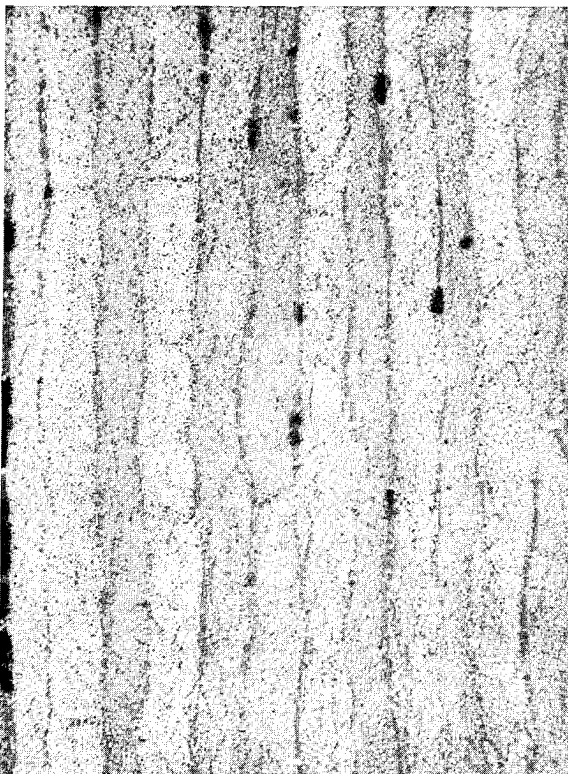
OPTICAL MICROGRAPHS OF TRANSVERSE CROSS-SECTIONS OF AS/3501-6 COMPOSITES



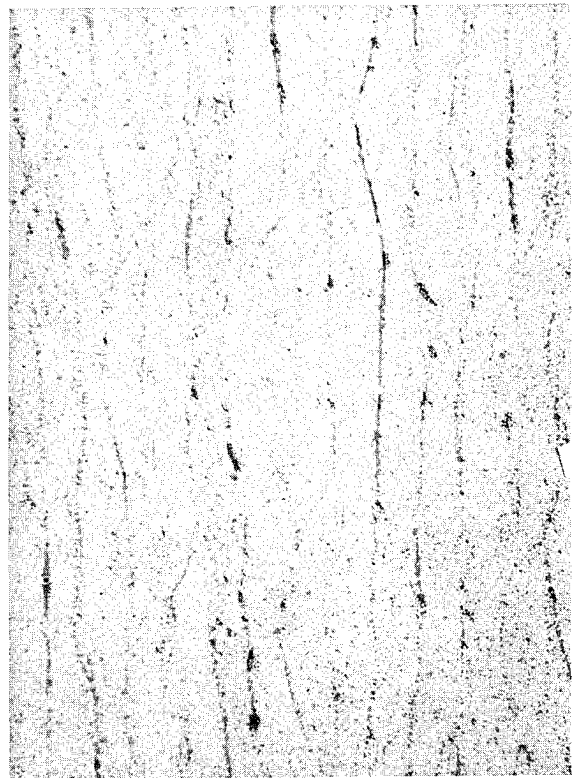
a) NAV-20 PREPREG HUMID AGED
72 hrs, 93% RH 200µm



b) NAV-27 PREPREG HUMID AGED
168 hrs, 0% RH 200µm

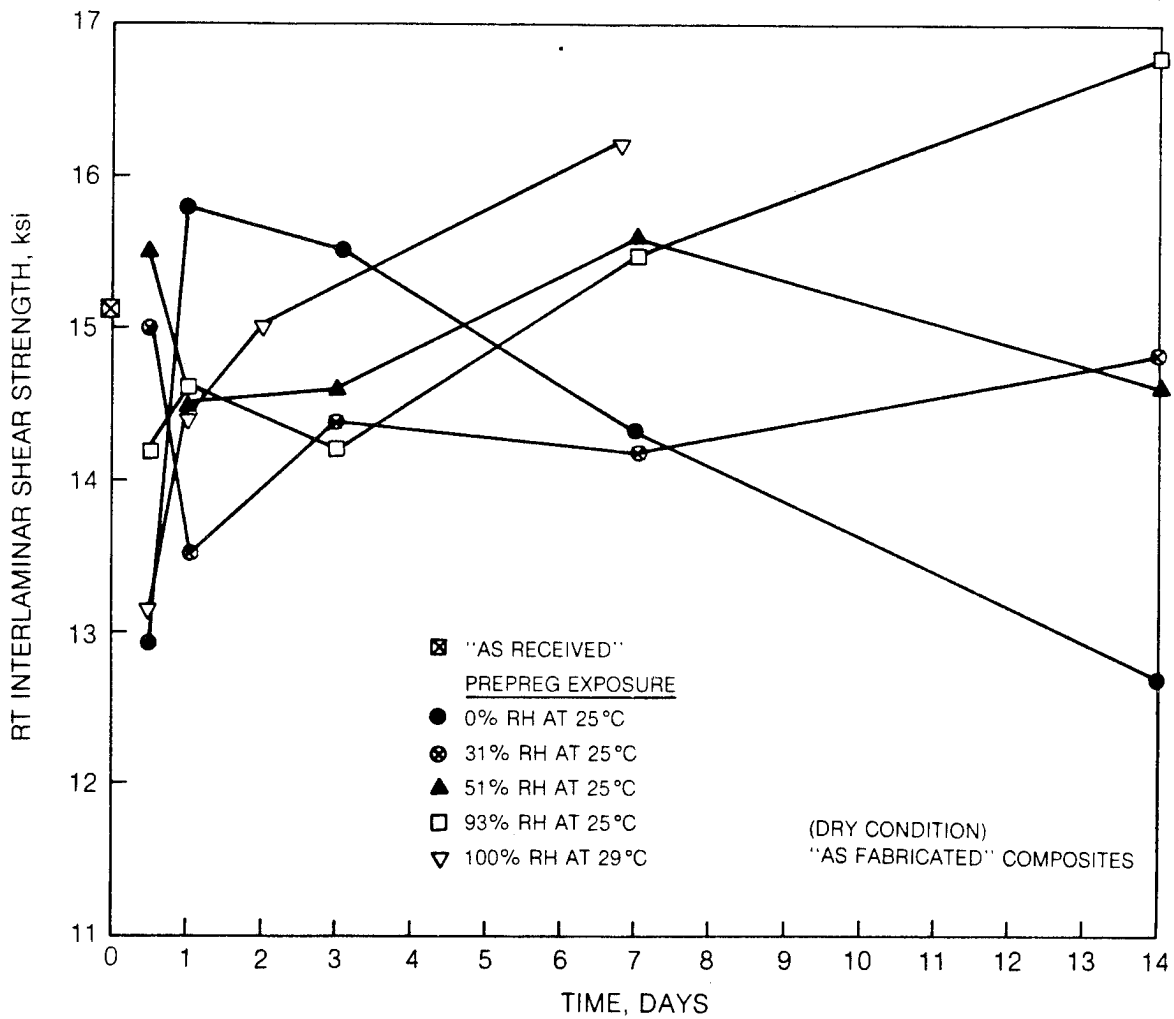


c) NAV-35 PREPREG HUMID AGED
168 hrs, 31% RH 200µm

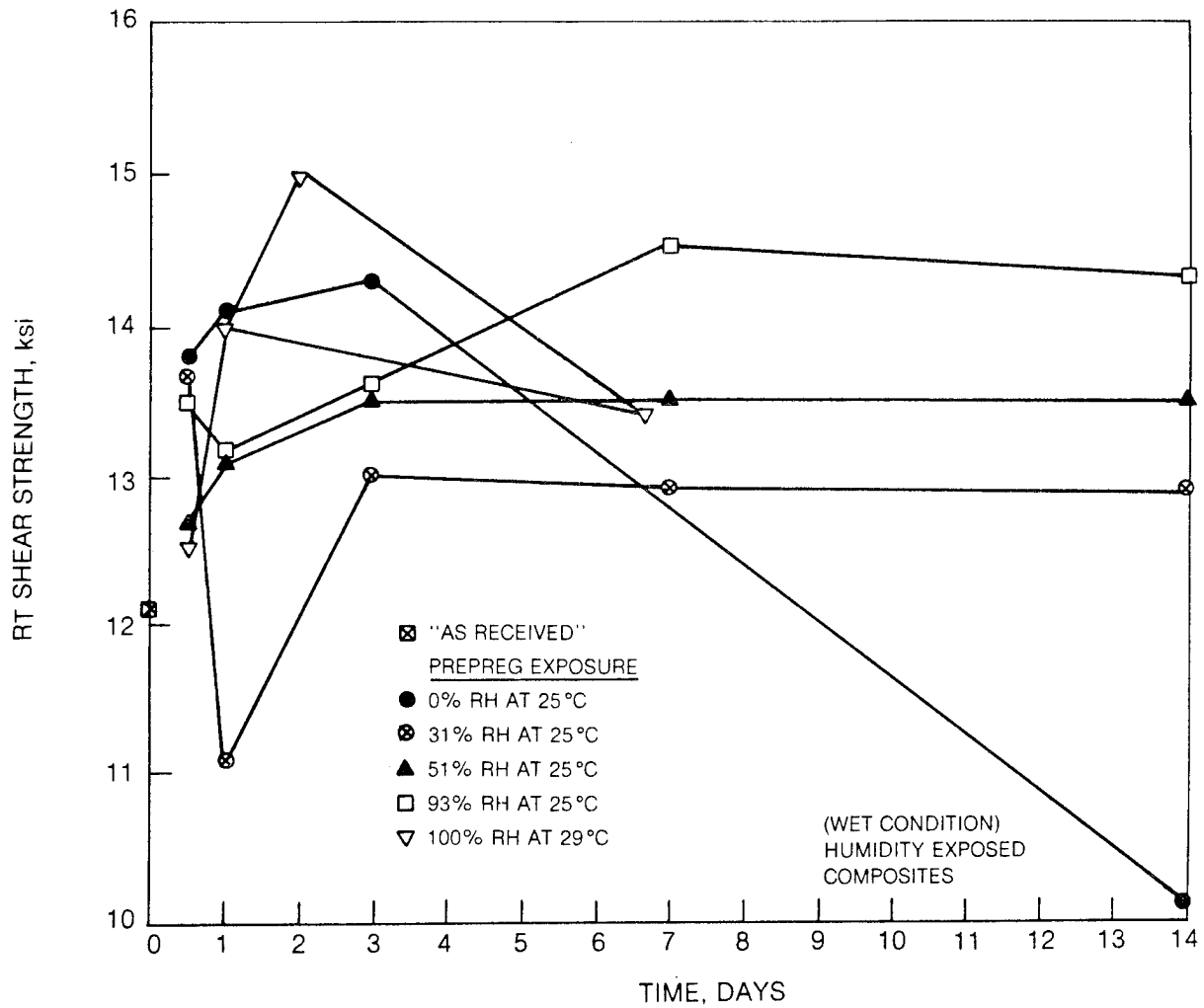


d) NAV-30 PREPREG HUMID AGED
168 hrs, 93% RH 200µm

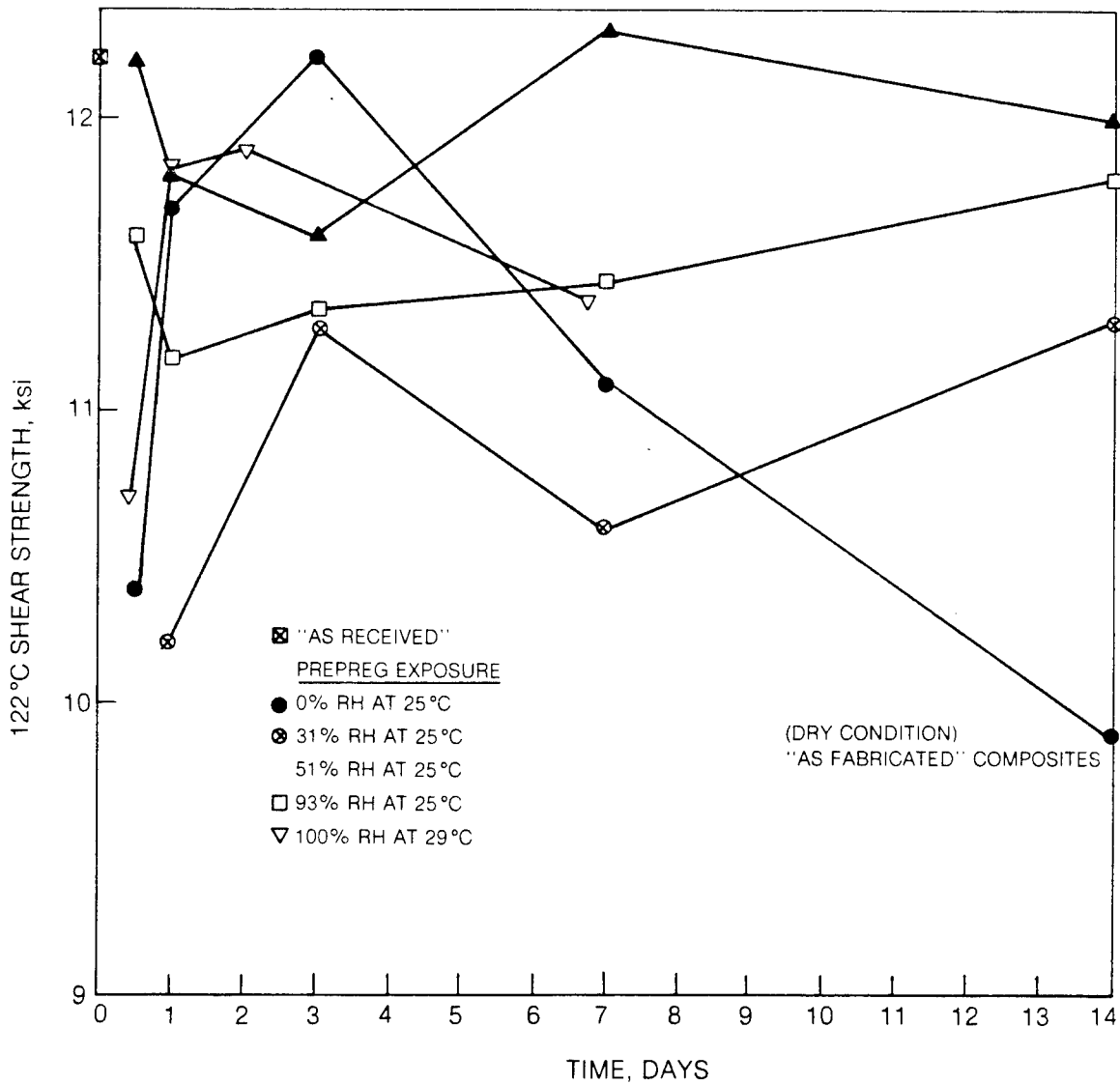
EFFECTS OF HUMID AGING OF AS/3501-6 PREPREG ON COMPOSITE SHEAR STRENGTH



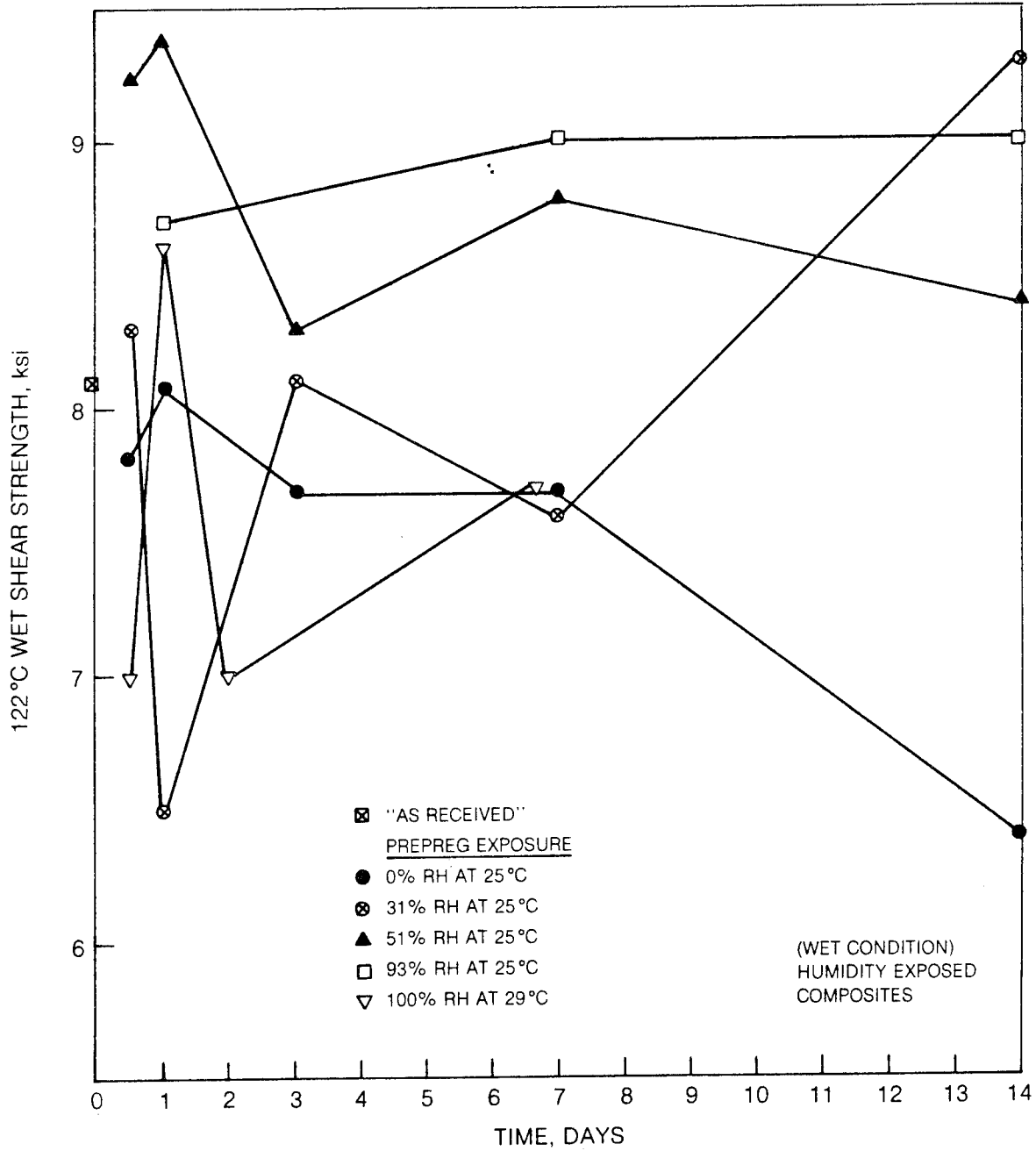
EFFECTS OF HUMID AGING OF AS/3501-6 PREPREG ON COMPOSITE SHEAR STRENGTH



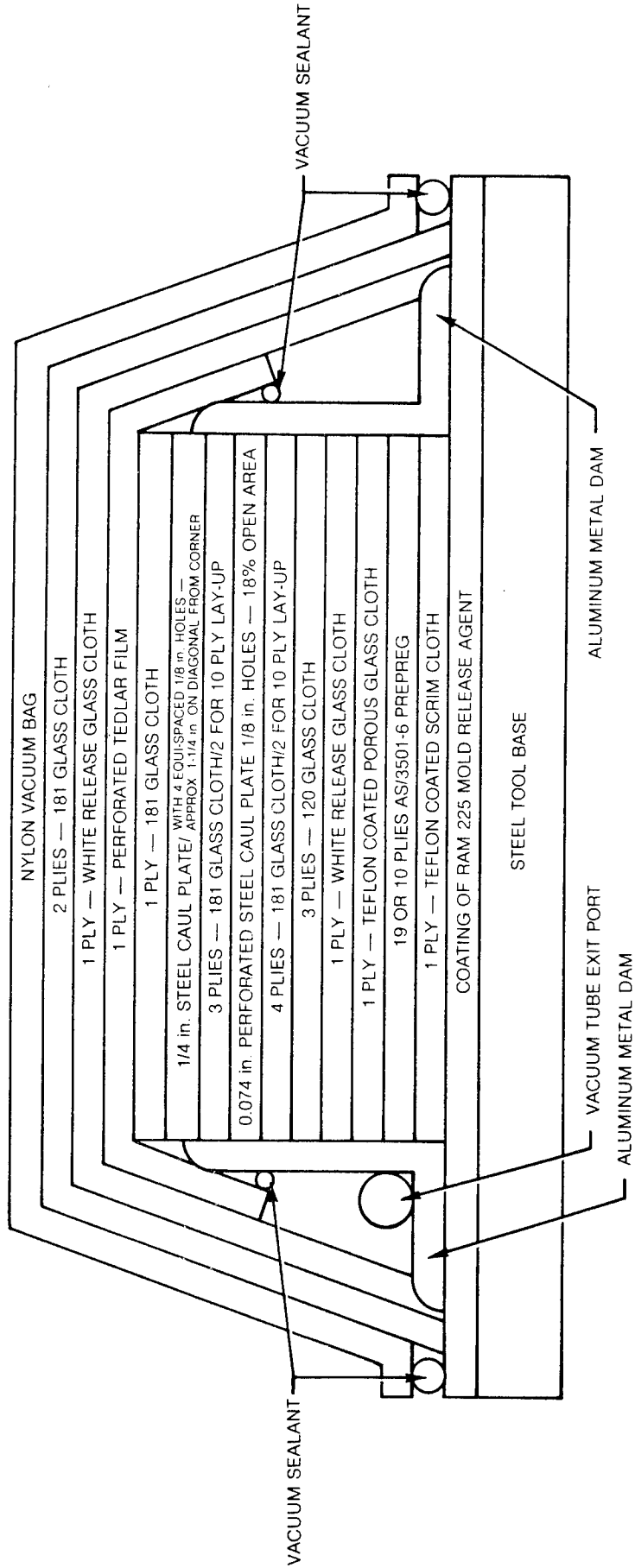
EFFECTS OF HUMID AGING OF AS/3501-6 PREPREG ON COMPOSITE SHEAR STRENGTH



EFFECTS OF HUMID AGING OF AS/3501-6 PREPREG ON COMPOSITE SHEAR STRENGTH



AUTOClave LAY-UP FOR AS/3501-6 PREPREG



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