

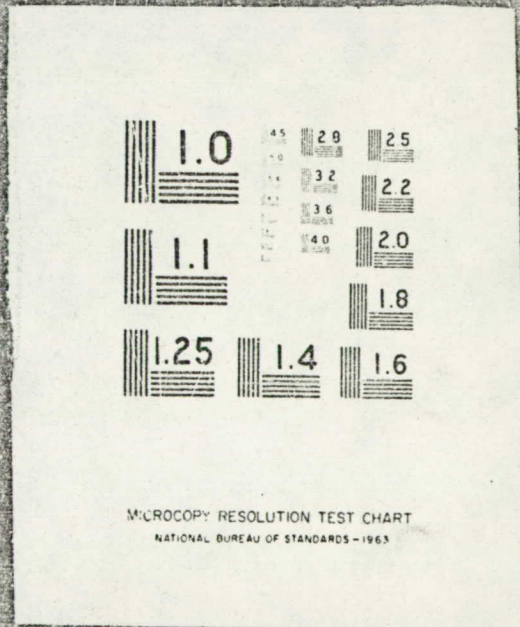
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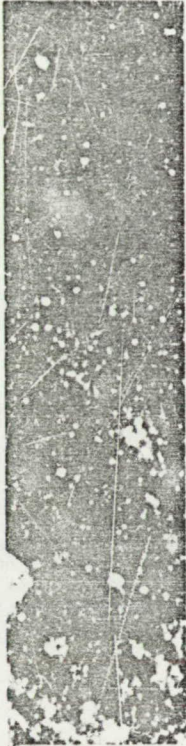
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— FINAL REPORT —

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# TESTING OF DIELECTRIC MATERIALS AND APPLICATION TECHNIQUES IN ELECTRONIC PACKAGING

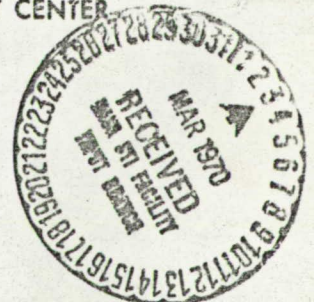
FINAL REPORT



Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
GEORGE C. MARSHALL SPACE FLIGHT CENTER

Under  
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GENERAL ELECTRIC Co.

SPACE SYSTEMS ORG.

DOCUMENT No. 69SD4371  
22 DECEMBER 1969

- FINAL REPORT -  
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TECHNIQUES IN ELECTRONIC PACKAGING

PREPARED FOR

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GEORGE C. MARSHALL SPACE FLIGHT CENTER  
MARSHALL SPACE FLIGHT CENTER, ALABAMA 35812

UNDER

CONTRACT No. NAS 8-21333

GENERAL  ELECTRIC

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## ABSTRACT

The results of a test program in extended temperature ranges of mechanical, physical and electrical properties for ten dielectric materials between  $-125^{\circ}\text{C}$  and  $+125^{\circ}\text{C}$  are given. In addition, the adequacies of these materials in the extended temperature range in specialized applications in vacuum, ultraviolet, and vibration environments are reported.

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SECTION 1  
INTRODUCTION

Test data on dielectric materials such as polyurethanes, epoxies, silicones, and urethane foams used in electronic packaging are generally available at or near room temperature. Reported herein are the results of a program designed to generate physical, electrical, and mechanical property data in an extended temperature range, -125 to +125°C. Complete testing was accomplished in accordance with the Marshall Space Flight Center Specifications listed below for the ten materials shown in Table 1-1.

APPLICABLE MFSC SPECIFICATIONS

<u>Specification Number</u>	<u>Issue</u>
202A	May 12, 1965
222B	October 19, 1965
276A	February 17, 1965
377A	November 16, 1966
379A	September 13, 1965
393A	January 17, 1966
411	March 1, 1966
418	February 15, 1966

In addition, the following tests were performed to establish the adequacy of the various materials in specialized applications at the extended temperature range:

1. Vibration testing on each material after it had been applied to its applicable MFSC procedure.
2. Light transmission effects of outgassing products from each material when deposited on simulated optical systems of spacecraft in the presence of ultraviolet light.
3. Ultraviolet weathering resistance.
4. Thermal conductivity and expansion.

Table 1-1. List of Materials Tested

Material	Manufacturer	MSFC Specification
2850 GT and Catalyst 9 (4%)	Emerson and Cuming Canton, Massachusetts	222 Type III
2651 and Catalyst 9 (8%)	Emerson and Cuming Canton, Massachusetts	222 Type V
1690 and Catalyst 9 (11%)	Emerson and Cuming Canton, Massachusetts	222 Type IV
Eccofoam FPH and Catalyst 12-2H	Emerson and Cuming Canton, Massachusetts	418 Type II
PC 22	Hysol Corporation Olean, New York	393 Type I
PR-1538 (amber)	Products Research Corp. 410 Jersey Avenue Gloucester City, New Jersey	393 Type I
PR-1535	Products Research Corp. 410 Jersey Avenue Gloucester City, New Jersey	202 Type III
794	Coast Pro-Seal Mfg. Co. Los Angeles, California	202 Type III
Sylgard 182	Dow Corning Midland, Michigan	379 Type III
GE-RTV 602	General Electric Company Waterford, New York	379 Type III

A test matrix of the mechanical and physical properties determined in this study is shown in Table 1-2. To ensure that the materials tested were representative of those contemplated for prime hardware applications, each was subjected to material acceptance tests outlined by its applicable specification. The results are outlined in Table 1-3. In all cases the materials tested met or exceeded specification limits.

Table 1-2. Mechanical and Physical Properties Test Matrix

Test	MSFC SPEC →	Material				
		Elastomeric Potting Compounds 202A	Epoxy Insulation 222B	Silicone Potting 379A	Elastomeric Conformal Coating 383A	Polyurethane Foam 418
Moisture Resistance (Electrical)		**o	**o	**o	**o	
Dielectric Constant		**o	**o	**o	**o	**o
Dissipation Factor		**o	**o	**o	**o	**o
Dielectric Strength		**o	**o	**o	**o	
Volume Resistivity		**o	**o	**o	**o	
Surface Resistivity		**o	**o	**o	**o	
Arc Resistance		**o	**o	**o	**o	
Dielectric Withstand Voltage		**o	**o	**o	**o	
Insulation Resistance		**o	**o	**o	**o	
Vibration(I)		**o	**o	**o	**o	**o
Viscosity (24°C) Fresh Mixed		.	.	.	. 45°C	
Viscosity (24°C) Fresh Thawed		.	.	.	.	
Specific Gravity		.	.	.	.	
Non-Volatile Content		.	(Volatile Loss)	.	.	
Bulk Density		.	.	.	.	.
Low Temperature Flex. (-55°C)	**			**		
Fungus	.	.	.	.	.	.
Ozone Resistance	**o		**o	**o		
Temperature Resistance	.		**o (Thermal Shock)	**o		**o
Moisture Absorption		.	.	.	.	.
Brittle Point		.	.	.	.	.
Flame Resistance		.	.	.	.	.
Aging Stability and Volume Change		.	.	.	.	**o
Tear Strength	**o			**o	**o	
Tensile Strength	**o			**o	**o	
Elongation	**o			**o	**o	
Shrinkage	.	.	.	.	.	.
Hardness After Full Cure	**o		**o	**o	**o	
Compression Set	**o			**o	**o	
Modulus of Elasticity in Tension	**o		**o	**o	**o	
Compressive Strength						**o
Stress-Strain						**o
Impact Resistance			**o			
Adhesion Bond Strength (Metal)	**o				**o (and epoxy fiber glass)	
Adhesion Bond Strength (PVC)	**o					
Adhesion Bond Strength (Neoprene)	**o					
Peel Strength			**o	**o		
Thermal Conductivity		**o	**o	**o		**o
Coefficient of Thermal Expansion		**o	**o	**o		**o
UV Radiation/Weathering Resistance	.	.	.	.	.	.
Light Transmission	.	.	.	.	.	.

(1) Test conducted from -125 to +125°C at 50°C increments

LEGEND

- Test at Normal Conditions
- o Test at +125°C
- \* Test at -125°C

Table 1-3. Results of Acceptance Tests

Material	Applicable #SFC Specifications	Shrinkage (%)		Viscosity (Poises)		Specific Gravity		Non-Volatile Content (%)	
		Requirements	Results	Requirements	Results	Requirements	Results	Requirements	Results
2850 GT & Catalyst	222 Type III	2 max	<2.0	---	---	2.4 max	2.30	99 min	99.89
2651 & Catalyst	222 Type V	3 max	<3.0	---	---	1.7 max	1.60	79 min	99.93
1090 & Catalyst	222 Type IV	2 max	<2.0	---	---	0.9 max	0.79	99 min	99.88
Eccofoum FPH & Catalyst 12-2H*	418 Type II	---	---	---	---	---	---	---	---
PC 22	393 Type I	3 max	2.49	100-450 100 max	156 29	1.2 max	1.10	99 min	99.97
PR-1538	393 Type I	3 max	2.69	250 max 100 max	139 28.8	1.2 max	1.08	99 min	99.93
FR-1535	202 Type III	3 max	2.65	100-300	200	1.1 max	1.09	99.9 min	99.98
794	202 Type III	3 max	2.65	100-300	204	1.1 max	1.08	99 min	99.96
Sylgard 182	379 Type III	1.6 max	0.56	150 max	30	0.99-1.08	1.05	99 min	99.84
RTV 602	379 Type III	1.6 max	0.55	150 max	50	0.99-1.08	1.00	99 min	99.0

\* Bulk density requirement for Eccofoum FPH was 2 to 3 cubic feet. Test Results were 2.65 cubic feet.

SECTION 2  
THERMAL PROPERTIES

2.1 COEFFICIENT OF THERMAL EXPANSION

2.1.1 EQUIPMENT

A quartz tube dilatometer similar to that described in ASTM D 696-44 was used with the exception that a Linear Variable Differential Transformer (LVDT) was used to sense thermal strain rather than a dial guage.

2.1.2 TEST PROCEDURE

Specimens, in a cylindrical geometry 3/8 inch by 2 inches long, were prepared from the following materials:

1. Stycast 2850
2. Stycast 2651
3. Stycast 1090
4. RTV 602
5. Sylgard 182

A hole 1/32-inch in diameter by 1/8-inch deep was drilled in each specimen in a radial direction approximately at the center point of the longitudinal axis. A copper-constantan thermocouple was inserted and held in place with Ducseal. The specimen was placed in the quartz tube of the dilatometer and the temperature was lowered to  $-125^{\circ}\text{C}$ . Data was collected at appropriate points as the temperature was raised to  $+125^{\circ}\text{C}$ . Data was obtained for three test specimens of each material.

### 2.1.3 TEST RESULTS

Test result data is plotted on curves in Figures 2-1 through 2-5.

## 2.2 THERMAL CONDUCTIVITY

### 2.2.1 EQUIPMENT AND TEST METHOD

The TC-100 Thermal Conductivity Comparator (Figure 2-6) is designed in accordance with ASTM Specification C-518. It makes steady-state measurements on insulative materials in the range of -250 to +600°F. The comparator's special advantage is the convenient sample size of a square plaque 2-1/2 inches on edge, nominally 1/4-inch thick, and its ability to determine a wider range of thermal conductivities than the guarded hot plate method. The principle of operation is based upon comparing the temperature drop across a specimen with that across each of two samples of known thermal conductivity. Armco Iron, Pyrocera 9606, fused silica, and Pyrex 7740 standards are used, depending on the thermal conductivity of the unknown specimen.

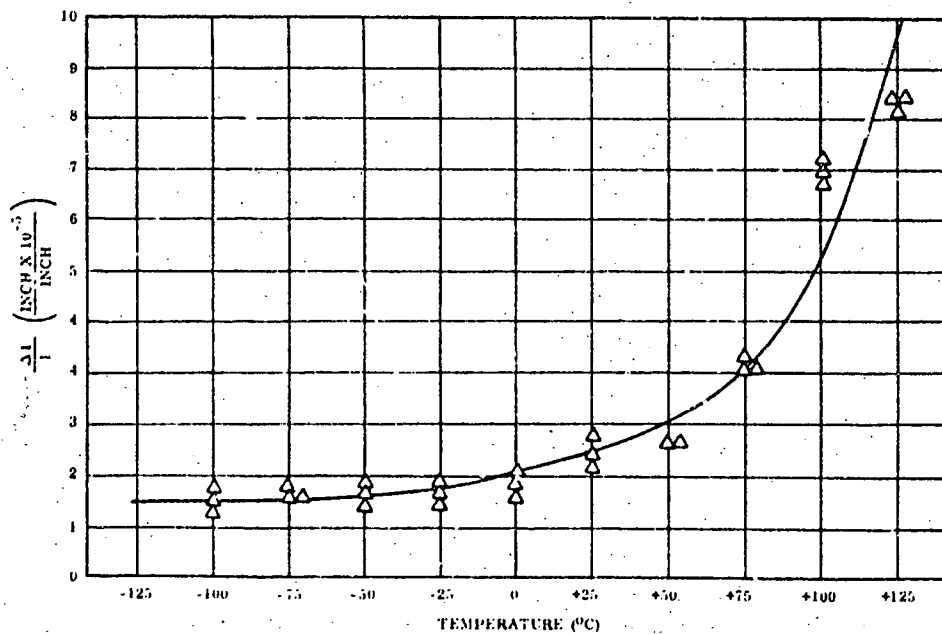


Figure 2-1. Coefficient of Thermal Expansion - Stycast 2850

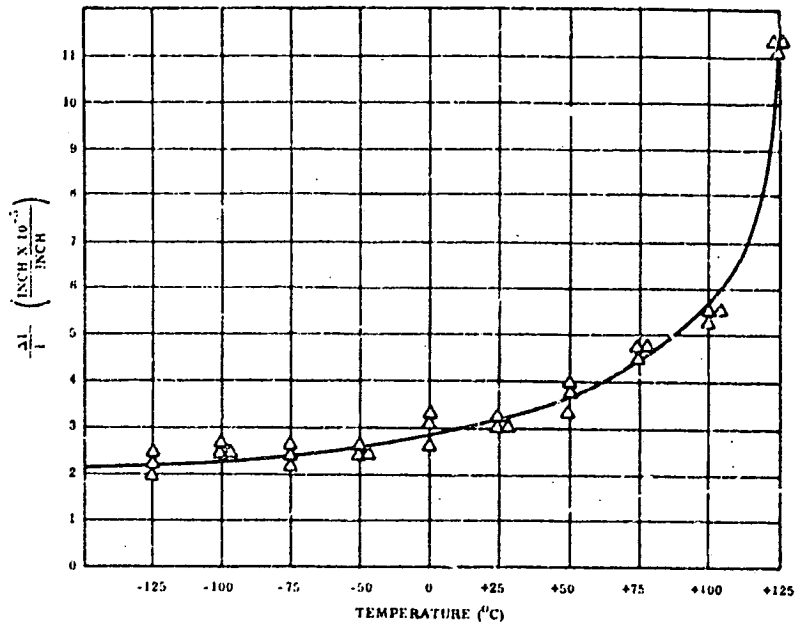


Figure 2-2. Coefficient of Thermal Expansion - Stycast 2651

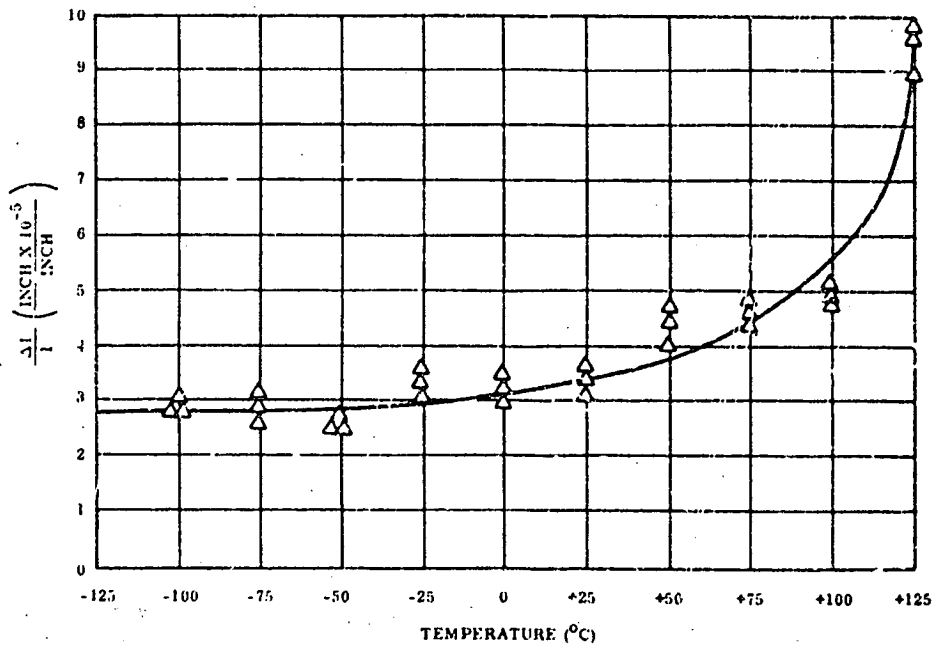


Figure 2-3. Coefficient of Thermal Expansion - Stycast 1090

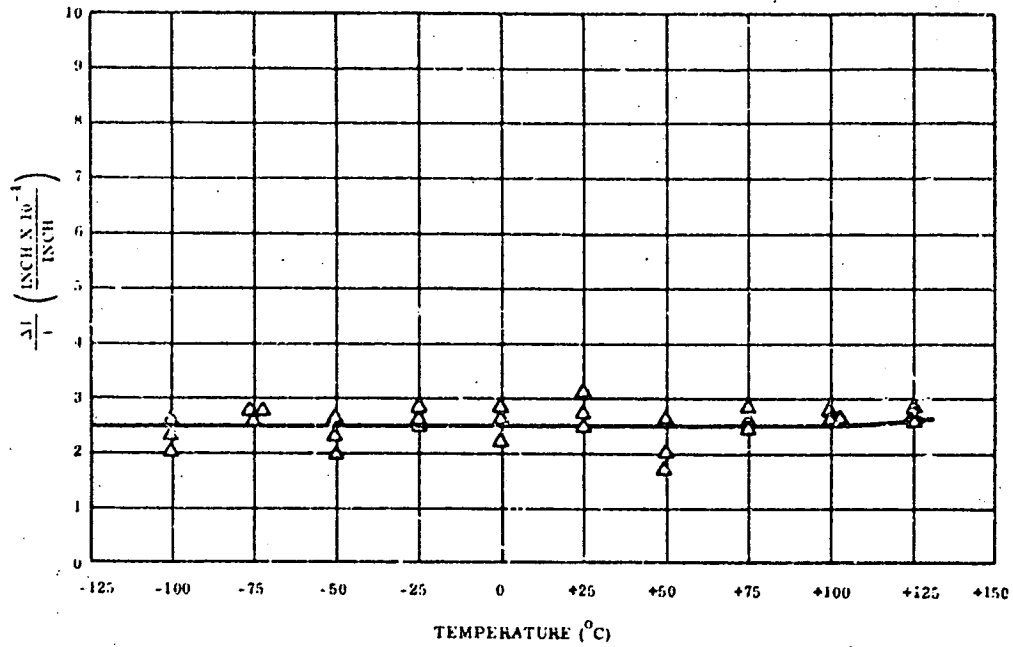


Figure 2-4. Coefficient of Thermal Expansion - Sylgard 182

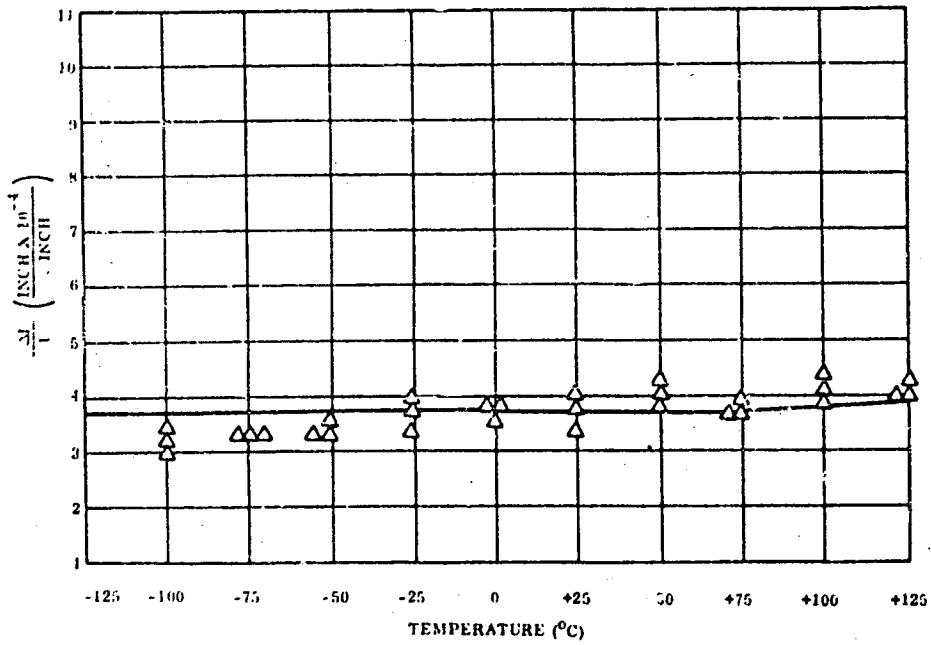


Figure 2-5. Coefficient of Thermal Expansion - RTV 602

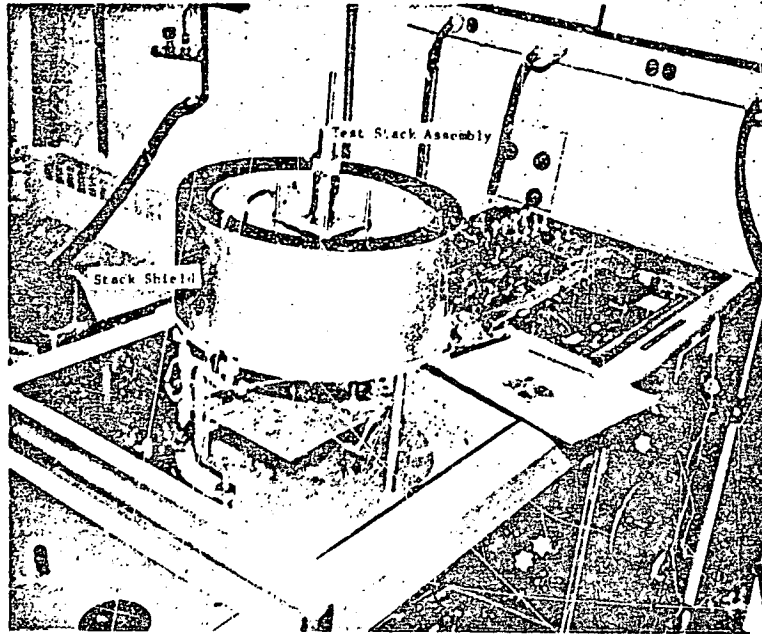


Figure 2-6. TC-1000 Thermal Conductivity Comparator

The apparatus consists of a power supply for heater power, a water-cooled heat sink, and a test stack assembly that includes two identical heat meters between which the specimen is sandwiched (Figure 2-7). Thus, the test section consists of a stack containing a heater, heat meter, specimen, a second heat meter, and finally, a heat sink at the bottom of the stack. During a test, the heat flows from the heater assembly through the stack to the heat sink. Surface temperatures are measured on each heat meter and specimen.

The temperature-dependent thermal conductivity coefficients of the specimen are determined by substituting recorded temperature gradients, heat meter thermal conductivity coefficients, and specimen and heat meter thickness into the following equation:

$$k_s = k_m \frac{T_m}{T_s} \frac{X_s}{X_m}$$

where

- $K_s, K_m$  = the specimen and heat meter thermal conductivities, respectively  
 $X_s, X_m$  = the specimen and heat meter thicknesses, respectively  
 $T_m$  = the average temperature drop across the heat meters  
 $T_s$  = the specimen temperature drop

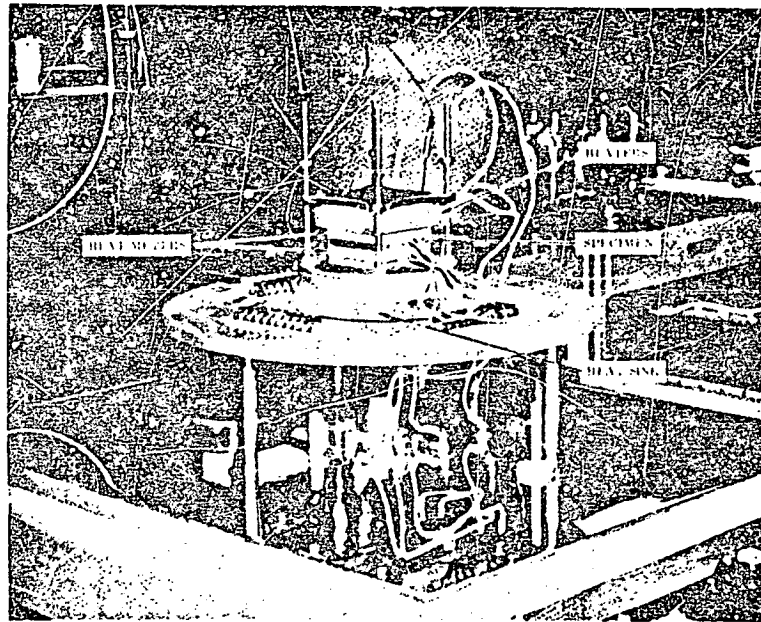


Figure 2-7. Comparator Test Stack Assembly

Measurements at the lower temperatures are obtained by circulating liquid nitrogen through the heat sink. The higher temperatures are obtained by use of an electrically-heated sink that raises the general temperature level of the composite stack.

### 2.2.2 TEST RESULTS

The thermal conductivities of the materials covered by MSFC Specifications 22C, 379B, and 418 are shown graphically in Figure 2-8.

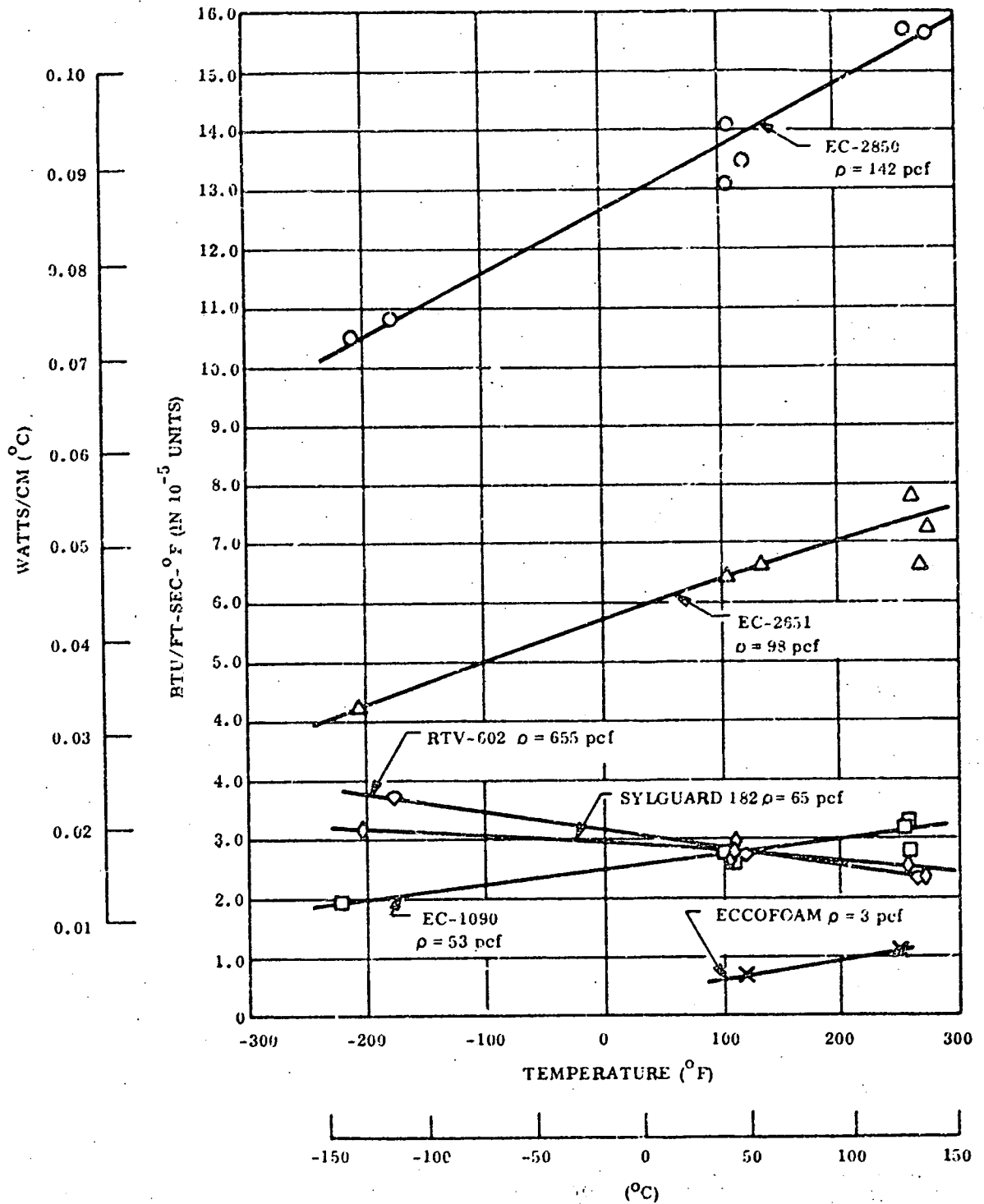


Figure 2-8. Thermal Conductivity of Potting Compounds

SECTION 3  
ELECTRICAL PROPERTIES

3.1 ELECTRICAL PROPERTIES TESTING

The electrical properties of the materials were determined in accordance with applicable MSFC, ASTM or federal test methods. Where extreme conditions lying outside the normal range covered by these procedures were necessary, the equipment and procedures were modified to conform with the best laboratory practices available. A summary of the data is given in Table 3-1.

3.2 DIELECTRIC CONSTANT AND DISSIPATION FACTOR TESTS

Dielectric constant and dissipation factor were determined in accordance with the applicable specification and ASTM D-150. Measurements were made at a frequency of 1 MHz on a Wayne Kerr Type B601 Capacitance Bridge powered by a General Radio Type 1339A Oscillator and modulated by a 100 Hz signal. Bridge balance was achieved from the 1000 Hz audible signal fed to a Hallicrafters Model S120 Receiver tuned to 1 MHz.

Dielectric constant was calculated from the specimen capacitance indicated by the bridge, the average specimen thickness (line readings) and the electrode area (after applying the proper edge correction for capacitance). Dissipation factor was calculated from the ac resistance, the measuring frequency and the capacitance indicated by the bridge. The overall accuracy for these measurements is  $\pm 3$  percent.

In general, the dielectric constant and dissipation factor of materials covered by the 202 and 393 specifications were proportional to temperature. Materials covered by the 222 specification exhibited very little change in dielectric constant and dissipation factor over the entire temperature excursion. However, the silicone materials covered by the 379 specification were inversely proportional to temperature in their dielectric constant, while maintaining a reasonably constant value for the dissipation factor. The extremely low density of the foam material covered by MSFC 418 precluded any reliable measurements. The specimen-electrode contact is so minimal that the bridge does not "see" any material at all.

### 3.3 VOLUME AND SURFACE RESISTIVITY

These properties were measured in accordance with ASTM D-257. All materials exhibited a decrease in volume and surface resistivity with an increase in temperature. Silicone materials were the least affected by thermal stress.

### 3.4 DIELECTRIC STRENGTH TESTS

This property was measured in accordance with ASTM D-149-64. The voltage source was a GE Cat. No. 153x355 Test Set having a variable output rating of 0 to 50 kv, 60 Hz and 2 kva. The waveshape conforms with paragraph 5.1 of ASTM D-149. The voltage control is a motor-driven adjustable autotransformer that produces a rate voltage rise variable from 300 to 3000 volts/second and capable of producing the 500 volts/second rate-of-rise as specified in the MSFC specifications.

One problem that arose in measuring dielectric strength at  $-125^{\circ}\text{C}$  was the inability to achieve breakdown of the material using 50 mil specimens. At that time, the test was also being performed in air and because of the higher breakdown voltages involved, arcing and flashover occurred. It was felt that a reduction in specimen thickness would lower the breakdown voltage to a level at which no arcing or flashover occurred prior to breakdown. New test specimens 15 to 20 mils thick were prepared; however, the breakdown voltage still exceeded the flashover voltage. A new test fixture was fabricated containing three sets of electrodes in a plexiglas enclosure. The specimens were placed between the electrodes and submerged in GE No. 10C Transil Oil. Breakdown of the material was thus possible, for although the oil was solid at  $-125^{\circ}\text{C}$ , the material represented the weakest insulation between the electrodes. Arcing and flashover were also eliminated by this technique in addition to increasing the number of specimens that could be tested during one temperature excursion. PR-1535, Pro-Seal 794, Stycasts 2850, 2651, and 1090, Sylgard 182 and PR-1538 all demonstrated a reduction in dielectric strength with increasing temperature. RTV 662 and PC 22, however, showed an increase in dielectric strength at  $+125^{\circ}\text{C}$ . These materials may undergo a post-cure reaction at this temperature, which increases their dielectric strength. It is quite feasible that this parameter could be used to determine the life expectancy of potting compounds and other types of insulation by utilizing accelerated aging techniques developed within GE.

Properties	Sylgard 182 (MSFC 379 Type III)	RTV 602 (MSFC 379 Type III)	PC 22 (MSFC 393 Type I)
<b>DIELECTRIC CONSTANT</b>			
ε -125°C	2.83 2.86 2.83 <u>AVG</u> 2.84	2.95 3.04 3.02 <u>AVG</u> 3.00	2.54 2.61 2.77 <u>AVG</u> 2.61
+ 25°C	2.53 2.44 2.50 2.49	2.54 2.54 2.54 2.54	4.03 3.98 3.98 3.98
+125°C	2.08 2.00 2.02 2.03	2.08 2.09 2.06 2.07	3.26 3.86 3.71 3.71
<b>DISSIPATION FACTOR</b>			
ε -125°C	0.0016 0.0016 0.0016 <u>AVG</u> 0.0016	0.0016 0.0016 0.0030 <u>AVG</u> 0.0021	0.0018 0.0018 0.0017 <u>AVG</u> 0.0018
+ 25°C	0.0023 0.0024 0.0024 0.0024	0.0022 0.0025 0.0022 0.0023	0.071 0.078 0.076 0.076
+125°C	0.0022 0.0026 0.0024 0.0024	0.0022 0.0024 0.0021 0.0022	0.048 0.057 0.054 0.054
<b>VOLUME RESISTIVITY (OHM-CM)</b>			
ε -125°C	1.3x10 <sup>16</sup> 6.5x10 <sup>15</sup> 6.7x10 <sup>15</sup> <u>AVG</u> 8.7x10 <sup>15</sup>	1.8x10 <sup>16</sup> 7.1x10 <sup>15</sup> 7.1x10 <sup>15</sup> <u>AVG</u> 1.0x10 <sup>16</sup>	1.9x10 <sup>16</sup> 1.8x10 <sup>16</sup> 1.8x10 <sup>16</sup> <u>AVG</u> 1.8x10 <sup>16</sup>
+ 25°C	2.3x10 <sup>15</sup> 2.7x10 <sup>15</sup> 2.5x10 <sup>15</sup> 2.5x10 <sup>15</sup>	5.3x10 <sup>13</sup> 3.1x10 <sup>13</sup> 3.6x10 <sup>13</sup> 4.0x10 <sup>13</sup>	2.5x10 <sup>13</sup> 2.7x10 <sup>13</sup> 3.2x10 <sup>13</sup> 3.2x10 <sup>13</sup>
+125°C	3.7x10 <sup>13</sup> 2.3x10 <sup>13</sup> 2.1x10 <sup>13</sup> 2.7x10 <sup>13</sup>	2.8x10 <sup>12</sup> 1.6x10 <sup>12</sup> 1.8x10 <sup>12</sup> 2.1x10 <sup>12</sup>	1.4x10 <sup>10</sup> 1.2x10 <sup>10</sup> 9.4x10 <sup>9</sup> 9.4x10 <sup>9</sup>
<b>SURFACE RESISTIVITY (OHM)</b>			
ε -125°C			3.0x10 <sup>19</sup> 2.1x10 <sup>14</sup> 2.8x10 <sup>14</sup> <u>AVG</u> 2.8x10 <sup>14</sup>
+ 25°C			7.0x10 <sup>14</sup> 9.5x10 <sup>14</sup> 1.2x10 <sup>15</sup> 1.2x10 <sup>15</sup>
+125°C			2.8x10 <sup>12</sup> 1.7x10 <sup>12</sup> 2.1x10 <sup>12</sup> 2.1x10 <sup>12</sup>
<b>DIELECTRIC STRENGTH (volts per mil)</b>			
ε -125°C	1250 940 1330 1120 1060 <u>AVG</u> 1140	930 1160 690 780 650 <u>AVG</u> 842	1830 1670 1420 1460 1560
+ 25°C	478 740 500 750 470 587	480 423 440 500 423 453	645 593 600 480 510
+125°C	650 630 500 535 280 520	705 795 765 655 830 750	737 690 685 725 737
<b>DIELECTRIC WITHSTAND VOLTAGE-1000 volts for one minute</b>			
ε -125°C			N.B.* N.B. N.B. <u>AVG</u> N.B.
+ 25°C			N.B. N.B. N.B. <u>AVG</u> N.B.
+125°C			N.B. N.B. N.B. <u>AVG</u> N.B.
<b>INSULATION RESISTANCE (megohms)</b>			
ε -125°C			1.7x10 <sup>8</sup> 2.5x10 <sup>8</sup> 1.0x10 <sup>8</sup> <u>AVG</u> 1.7x10 <sup>8</sup>
+ 25°C	2.5x10 <sup>8</sup> 1.7x10 <sup>8</sup> 1.7x10 <sup>8</sup> 2.0x10 <sup>8</sup>		2.6x10 <sup>5</sup> 2.4x10 <sup>5</sup> 1.9x10 <sup>5</sup> 1.9x10 <sup>5</sup>
+125°C			190 150 170 170
<b>MOISTURE RESISTANCE (megohms)</b>			
ε -125°C			4.2x10 <sup>8</sup> 1.7x10 <sup>8</sup> 4.5x10 <sup>8</sup> <u>AVG</u> 3.5x10 <sup>8</sup>
+ 25°C			2.1x10 <sup>4</sup> 2.4x10 <sup>4</sup> 1.9x10 <sup>4</sup> 1.9x10 <sup>4</sup>
+125°C			69 72 53 53

FOLDOUT FRAME

Type I)	PR-1538 (MSFC 393 Type I)	PR 1535 (MSFC 202 Type III)	PRO-SEAL 794 (MSFC 202 Type III)	E C-2850 (MSFC Type III)
AVG .77 2.64	2.57 2.59 2.56 2.58	2.68 2.54 2.64 2.62	2.57 2.56 2.56 2.56	4.66 4.46 4.52 4.54
.98 3.99	4.07 3.97 4.00 4.01	4.73 4.91 4.93 4.86	4.62 4.62 4.63 4.62	5.20 5.00 4.91 5.05
.71 3.61	4.37 3.76 3.90 4.01	4.77 4.47 4.65 4.63	4.49 4.18 4.31 4.32	4.98 5.10 4.90 5.07
AVG .0017 0.0018	0.009 0.009 0.009 0.009	0.0095 0.0098 0.0120 0.0104	0.012 0.009 0.009 0.010	0.008 0.007 0.007 0.007
.076 0.075	0.076 0.075 0.078 0.076	0.075 0.077 0.076 0.076	0.062 0.062 0.063 0.062	0.020 0.020 0.019 0.02
.054 0.053	0.054 0.047 0.046 0.049	0.039 0.032 0.033 0.034	0.037 0.035 0.036 0.036	0.023 0.025 0.025 0.02
AVG .8x10 <sup>16</sup> 1.8x10 <sup>16</sup>	1.8x10 <sup>16</sup> 1.2x10 <sup>16</sup> 1.8x10 <sup>16</sup> 1.6x10 <sup>16</sup>	3.7x10 <sup>16</sup> 3.6x10 <sup>16</sup> 2.2x10 <sup>16</sup> 3.2x10 <sup>16</sup>	1.2x10 <sup>16</sup> 9.0x10 <sup>15</sup> 1.2x10 <sup>16</sup> 1.1x10 <sup>16</sup>	1.7x10 <sup>16</sup> 1.9x10 <sup>16</sup> 1.8x10 <sup>16</sup> 1.8x10 <sup>16</sup>
.2x10 <sup>13</sup> 2.8x10 <sup>13</sup>	6.9x10 <sup>12</sup> 7.1x10 <sup>12</sup> 6.9x10 <sup>12</sup> 7.0x10 <sup>12</sup>	1.1x10 <sup>13</sup> 2.4x10 <sup>12</sup> 2.6x10 <sup>12</sup> 5.3x10 <sup>12</sup>	7.7x10 <sup>12</sup> 7.1x10 <sup>12</sup> 6.3x10 <sup>12</sup> 1.1x10 <sup>16</sup>	1.2x10 <sup>15</sup> 1.4x10 <sup>15</sup> 1.3x10 <sup>15</sup> 1.3x10 <sup>15</sup>
.4x10 <sup>9</sup> 1.2x10 <sup>10</sup>	6.9x10 <sup>9</sup> 9.8x10 <sup>9</sup> 7.9x10 <sup>9</sup> 8.2x10 <sup>9</sup>	1.0x10 <sup>10</sup> 9.6x10 <sup>9</sup> 9.8x10 <sup>9</sup> 9.8x10 <sup>9</sup>	1.9x10 <sup>10</sup> 1.5x10 <sup>10</sup> 1.9x10 <sup>10</sup> 1.7x10 <sup>10</sup>	6.2x10 <sup>10</sup> 3.5x10 <sup>10</sup> 4.0x10 <sup>10</sup> 4.6x10 <sup>10</sup>
AVG .8x10 <sup>14</sup> 2.6x10 <sup>14</sup>	6.2x10 <sup>14</sup> 6.2x10 <sup>14</sup> 1.2x10 <sup>15</sup> 8.1x10 <sup>14</sup>	1.2x10 <sup>14</sup> 5.0x10 <sup>13</sup> 8.3x10 <sup>13</sup> 8.4x10 <sup>13</sup>	6.2x10 <sup>14</sup> 1.2x10 <sup>15</sup> 8.2x10 <sup>14</sup> 8.8x10 <sup>14</sup>	8.3x10 <sup>15</sup> 1.2x10 <sup>16</sup> 1.2x10 <sup>16</sup> 1.1x10 <sup>16</sup>
.2x10 <sup>15</sup> 9.5x10 <sup>14</sup>	5.2x10 <sup>13</sup> 2.8x10 <sup>14</sup> 2.5x10 <sup>13</sup> 1.2x10 <sup>14</sup>	5.5x10 <sup>12</sup> 1.2x10 <sup>13</sup> 1.2x10 <sup>13</sup> 9.8x10 <sup>12</sup>	4.5x10 <sup>13</sup> 1.2x10 <sup>14</sup> 1.2x10 <sup>14</sup> 9.5x10 <sup>13</sup>	5.5x10 <sup>14</sup> 6.2x10 <sup>14</sup> 4.2x10 <sup>14</sup> 5.3x10 <sup>14</sup>
.1x10 <sup>12</sup> 2.2x10 <sup>12</sup>	5.7x10 <sup>11</sup> 3.1x10 <sup>11</sup> 3.9x10 <sup>11</sup> 4.2x10 <sup>11</sup>	1.2x10 <sup>12</sup> 1.9x10 <sup>11</sup> 2.5x10 <sup>11</sup> 5.4x10 <sup>11</sup>	3.4x10 <sup>12</sup> 8.9x10 <sup>12</sup> 4.5x10 <sup>12</sup> 5.6x10 <sup>12</sup>	2.2x10 <sup>12</sup> 1.7x10 <sup>12</sup> 2.1x10 <sup>12</sup> 2.0x10 <sup>12</sup>
AVG 160 1560 1588	1210 1350 1460 1500 1310 1370	1820 1610 1470 1650 1600 1648	1740 1870 825 1170 900 1260	630 455 515 775 715
180 510 565	528 580 534 521 544 542	540 492 500 573 491 520	570 580 568 572 561 570	533 620 521 471 365
725 737 715	500 585 500 525 445 511	310 355 382 362 330 348	352 374 265 143 176 262	212 328 446 304 234
AVG .B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.
.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.
.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.	N.B. N.B. N.B. N.B.
AVG 1.0x10 <sup>8</sup> 1.7x10 <sup>8</sup>	1.0x10 <sup>9</sup> 2.5x10 <sup>9</sup> 1.2x10 <sup>9</sup> 1.6x10 <sup>9</sup>	1.0x10 <sup>9</sup> 8.3x10 <sup>8</sup> 8.3x10 <sup>8</sup> 8.8x10 <sup>8</sup>	1.0x10 <sup>9</sup> 1.0x10 <sup>9</sup> 1.0x10 <sup>9</sup> 1.0x10 <sup>9</sup>	3.3x10 <sup>8</sup> 3.3x10 <sup>8</sup> 1.2x10 <sup>8</sup> 2.6x10 <sup>8</sup>
.9x10 <sup>5</sup> 2.3x10 <sup>5</sup>	1.2x10 <sup>4</sup> 2.5x10 <sup>4</sup> 2.1x10 <sup>4</sup> 1.9x10 <sup>4</sup>	2.9x10 <sup>4</sup> 1.9x10 <sup>4</sup> 3.1x10 <sup>4</sup> 2.6x10 <sup>4</sup>	6.2x10 <sup>4</sup> 7.1x10 <sup>4</sup> 5.9x10 <sup>4</sup> 6.4x10 <sup>4</sup>	5.0x10 <sup>7</sup> 4.2x10 <sup>7</sup> 5.0x10 <sup>7</sup> 4.6x10 <sup>7</sup>
70 170	120 50 60 76	89 130 89 103	76 100 76 84	670 2000 670 1113
AVG 5x10 <sup>8</sup> 3.4x10 <sup>8</sup>	3.1x10 <sup>8</sup> 4.5x10 <sup>8</sup> 1.9x10 <sup>8</sup> 3.2x10 <sup>8</sup>	3.3x10 <sup>8</sup> 3.3x10 <sup>8</sup> 5.0x10 <sup>8</sup> 3.8x10 <sup>8</sup>	3.3x10 <sup>8</sup> 5.0x10 <sup>8</sup> 5.0x10 <sup>8</sup> 4.4x10 <sup>8</sup>	5.0x10 <sup>6</sup> 2.5x10 <sup>6</sup> 2.5x10 <sup>6</sup> 3.3x10 <sup>6</sup>
.9x10 <sup>4</sup> 2.1x10 <sup>4</sup>	860 1000 860 906	1.1x10 <sup>3</sup> 6.4x10 <sup>2</sup> 8.9x10 <sup>2</sup> 870	680 620 620 640	3.3x10 <sup>7</sup> 5.0x10 <sup>7</sup> 5.0x10 <sup>6</sup> 2.9x10 <sup>6</sup>
3 65	25 20 20 22	83 120 100 101	38 36 50 41	2100 550 680 1100

FOLDOUT FRAME 2

Table 3-1. Summary of Electrical Property Testing

AL 794 (MSFC 202 Type III)				E C-2650 (MSFC Type III)				E C-2651 (MSFC Type IV)				E C 1090 (MSFC Type V)			
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
2.56	2.56	2.56		4.66	4.46	4.52	4.54	3.42	3.38	3.45	3.42	2.31	2.31	2.32	2.31
4.62	4.63	4.62		5.23	5.00	4.91	5.05	3.86	3.35	3.89	3.90	2.59	2.62	2.63	2.62
4.18	4.31	4.32		4.98	5.19	4.90	5.07	3.73	4.01	4.01	3.92	2.62	2.59	2.76	2.66
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
0.009	0.009	0.010		0.008	0.007	0.007	0.007	0.009	0.008	0.008	0.008	0.011	0.012	0.011	0.011
0.062	0.063	0.062		0.020	0.020	0.019	0.020	0.033	0.034	0.033	0.033	0.028	0.026	0.026	0.027
0.035	0.036	0.036		0.023	0.025	0.025	0.025	0.038	0.044	0.048	0.043	0.054	0.052	0.055	0.054
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
$9.0 \times 10^{15}$	$1.2 \times 10^{16}$	$1.1 \times 10^{16}$		$1.7 \times 10^{16}$	$1.9 \times 10^{16}$	$1.8 \times 10^{16}$	$1.9 \times 10^{16}$	$1.8 \times 10^{16}$	$1.4 \times 10^{16}$	$1.2 \times 10^{16}$	$1.5 \times 10^{16}$	$1.2 \times 10^{16}$	$1.8 \times 10^{16}$	$1.7 \times 10^{16}$	$1.8 \times 10^{16}$
$7.1 \times 10^{12}$	$6.3 \times 10^{12}$	$1.1 \times 10^{16}$		$1.2 \times 10^{15}$	$1.4 \times 10^{15}$	$1.3 \times 10^{15}$	$1.3 \times 10^{15}$	$3.2 \times 10^{14}$	$3.5 \times 10^{14}$	$3.1 \times 10^{14}$	$3.3 \times 10^{14}$	$1.8 \times 10^{14}$	$1.5 \times 10^{14}$	$1.9 \times 10^{14}$	$1.7 \times 10^{14}$
$1.5 \times 10^{10}$	$1.9 \times 10^{10}$	$1.7 \times 10^{10}$		$6.2 \times 10^{10}$	$3.5 \times 10^{10}$	$4.0 \times 10^{10}$	$4.6 \times 10^{10}$	$1.8 \times 10^{10}$	$2.6 \times 10^{10}$	$1.3 \times 10^{10}$	$1.9 \times 10^{10}$	$3.9 \times 10^{10}$	$2.4 \times 10^{10}$	$1.8 \times 10^{10}$	$2.7 \times 10^{10}$
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
$1.2 \times 10^{15}$	$8.2 \times 10^{14}$	$8.8 \times 10^{14}$		$8.3 \times 10^{15}$	$1.2 \times 10^{16}$	$1.2 \times 10^{16}$	$1.1 \times 10^{16}$	$1.2 \times 10^{16}$	$1.6 \times 10^{16}$	$2.2 \times 10^{15}$	$1.1 \times 10^{16}$	$2.5 \times 10^{15}$	$2.3 \times 10^{15}$	$1.8 \times 10^{15}$	$2.2 \times 10^{15}$
$1.2 \times 10^{14}$	$1.2 \times 10^{14}$	$9.5 \times 10^{13}$		$5.5 \times 10^{14}$	$6.2 \times 10^{14}$	$4.2 \times 10^{14}$	$5.3 \times 10^{14}$	$6.2 \times 10^{15}$	$1.2 \times 10^{16}$	$5.0 \times 10^{15}$	$7.7 \times 10^{15}$	$4.2 \times 10^{14}$	$6.2 \times 10^{14}$	$3.6 \times 10^{14}$	$4.6 \times 10^{14}$
$8.9 \times 10^{12}$	$4.5 \times 10^{12}$	$5.6 \times 10^{12}$		$2.2 \times 10^{12}$	$1.7 \times 10^{12}$	$2.1 \times 10^{12}$	$2.0 \times 10^{12}$	$1.6 \times 10^{11}$	$1.2 \times 10^{13}$	$3.1 \times 10^{11}$	$4.1 \times 10^{12}$	$3.1 \times 10^{12}$	$1.8 \times 10^{12}$	$2.1 \times 10^{12}$	$1.4 \times 10^{12}$
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
170	825	1170	900 1260	680	455	515	775 715 630	1165	1210	1105	1300 1200 1195	900	1000	710	850 850 800
180	568	572	561 570	533	620	521	471 365 502	1050	1165	1075	765 925 996	450	452	395	450 375 424
174	265	143	176 262	212	328	446	304 234 305	334	250	300	270 284 287	150	137	157	173 137 150
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
N.B.	N.B.	N.B.		N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.
N.B.	N.B.	N.B.		N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.
N.B.	N.B.	N.B.		N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.	N.B.
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
$1.0 \times 10^9$	$1.0 \times 10^9$	$1.0 \times 10^9$		$3.3 \times 10^8$	$3.3 \times 10^8$	$1.2 \times 10^8$	$2.6 \times 10^8$	$3.3 \times 10^7$	$1.0 \times 10^8$	$2.5 \times 10^6$	$1.3 \times 10^8$	$5.0 \times 10^8$	$5.0 \times 10^8$	$2.5 \times 10^8$	$4.1 \times 10^8$
$7.1 \times 10^4$	$5.9 \times 10^4$	$6.4 \times 10^4$		$5.0 \times 10^7$	$4.2 \times 10^7$	$5.0 \times 10^7$	$4.6 \times 10^7$	$3.8 \times 10^7$	$3.3 \times 10^7$	$4.2 \times 10^7$	$3.7 \times 10^7$	$6.2 \times 10^6$	$8.3 \times 10^6$	$8.3 \times 10^6$	$7.6 \times 10^6$
100	76	84		670	2000	670	1113	330	450	360	390	106	147	111	121
<u>AVG</u>				<u>AVG</u>				<u>AVG</u>				<u>AVG</u>			
$5.0 \times 10^8$	$5.0 \times 10^8$	$4.4 \times 10^8$		$5.0 \times 10^8$	$2.5 \times 10^8$	$2.5 \times 10^8$	$3.3 \times 10^8$	$5.0 \times 10^8$	$2.8 \times 10^8$	$1.2 \times 10^8$	$3.0 \times 10^8$	$3.3 \times 10^8$	$5.0 \times 10^8$	$2.5 \times 10^8$	$3.6 \times 10^8$
620	620	640		$3.3 \times 10^7$	$5.0 \times 10^7$	$5.0 \times 10^6$	$2.9 \times 10^7$	$3.3 \times 10^7$	$4.2 \times 10^7$	$3.1 \times 10^7$	$3.5 \times 10^7$	$3.3 \times 10^4$	$5.3 \times 10^3$	$5.6 \times 10^3$	$1.5 \times 10^4$
36	50	41		2100	550	680	1100	500	38	312	397	110	84	100	98

Although the frequency of the voltage used in these tests was limited to 60 Hz, it is known that the frequency will exert a strong influence on the dielectric strength of a material. Future evaluations on potting compounds and insulators should include dielectric strength testing at radio frequencies and higher.

### 3.5 DIELECTRIC WITHSTAND VOLTAGE TEST

Tests were performed in accordance with the applicable MSFC specification. No breakdown occurred in any specimen of all the materials. This test is rather insignificant because it is unlikely that a breakdown would occur unless a gross defect existed, i. e., an air pocket containing trapped moisture or contaminant that would be conductive. These types of defects would be evident in the insulation resistance test.

### 3.6 INSULATION RESISTANCE AND MOISTURE RESISTANCE TEST

Testing was performed in accordance with the applicable MSFC specifications. This test provides a good indication of the product's susceptibility to high humidity environments.

All the materials tested except the Stycasts showed appreciable degradation in insulation resistance after the humidity cycles (moisture resistance). The degradation was slight at  $-125^{\circ}\text{C}$ , where the resistance dropped about a half decade. At  $+25^{\circ}\text{C}$ , the resistance dropped approximately two decades and at  $+125^{\circ}\text{C}$  was about 50 percent of the original value.

### 3.7 ARC RESISTANCE

#### 3.7.1 TEST SPECIMENS

Samples 0.050 inch thick of the following materials were tested:

1. PR-1538
2. PR-1535
3. PC 22
4. Pro-Seal 794

5. Stycast 1090
6. Stycast 2651
7. Stycast 2850GT

### 3.7.2 TEST METHOD

ASTM D-495 with tungsten rod electrodes was used. The equipment used meets specified requirements.

Room temperature tests were made in a room controlled at  $23 \pm 1^{\circ}\text{C}$  and  $50 \pm 2\%$  RH. Tests at  $-125^{\circ}$  and  $+125^{\circ}\text{C}$  were made in a Missimers box, and controlled to better than  $\pm 5^{\circ}\text{C}$ . Cold nitrogen gas from a liquid nitrogen tank was used for the cold temperature tests. A current of dry gas was used to keep the surface of the test specimen from accumulating frost. However, it was necessary to turn off the gas circulating fan while the tests were being made, since the air currents distributed the arcs. At high temperatures, electrical heating units were used. The tests were viewed through a glass window in the front of the box. Care was used to achieve temperature stability in the test specimen before starting the tests.

### 3.7.3 TEST RESULTS

The complete test results for the seven types of specimens are shown in Table 3-2. For some of the materials, at least two types of failure end-points could be achieved - one in which the arc largely disappeared in the melting and flaming material, and one in which a conducting carbonaceous path developed. According to ASTM, it is the first failure that should be recorded. However, in some cases the time to form the conducting path has also been recorded. Brief comments have been added to describe some of the accompanying phenomena. Individual values are listed in order of magnitude and not in the chronological order in which the results were obtained. The average value has been calculated.

### 3.7.4 DISCUSSION OF THE DATA

It should be recognized that the D-495 test is conducted as a series of 60-second tests with a sharp increase in the severity of the test after each step. Hence, a failure at 123 seconds

Table 3-2. D-495 Arc Resistance, Tungsten Rod Electrodes

Material	Temperature (°C)	1st Failure (seconds)	Conductive Path (seconds)	Comments	Material	Temperature (°C)	1st Failure (seconds)	Conductive Path (seconds)	Comments						
PR-1538	23	124	270	Continuous flaming occurs at 123 seconds with a conductive path becoming established after 270 seconds. Time to conductive path will vary with thickness and melting pattern. Melting starts with the first arc and builds up to a large area (G/2 inch dia. hole) when the conductive path forms. Depolymerization of the material occurs during the test.  Same as above with slightly more melting in the earlier parts of the test.	Similar to PR-1538	-125	245	320	Similar to PR-1538						
		123					244								
	123	243													
	122	243													
	122	242													
	122	242													
	123	243													
	Avg.	Avg.													
	PR-1535	+125	122				270			Same as above. Burning occurs at 234 seconds	Same as above	+125	47	Same as 1st	Just prior to the formation of the conductive path, the area between the electrodes usually becomes enveloped in flames.
			122										45		
122		44													
122		44													
122		41													
122		36													
122		26													
122		40													
Avg.		Avg.													
PR-1535		-125	330	350	Same as above. Burning occurs at 234 seconds	Same as above	-125	170	Same				Same as above		
	328		165												
	330	150													
	302	147													
	282	147													
	267	147													
	253	138													
	242	302													
	242	320													
	Avg.	Avg.													
PR-1535	25	124	220	Similar to PR-1538	Stycast 2651	+23	136	Same as 1st failure	Similar to Stycast 1090						
		123					134								
	123	133													
	122	126													
	122	124													
	123	123													
	122	129													
	Avg.	Avg.													
	PR-1535	+125	122				183			Similar to PR-1538	Stycast 1090	+125	130	Same	Similar to Stycast 1090
			122										130		
122		129													
121		129													
121		129													
121		129													
121		127													
Avg.		Avg.													
PR-1535		-125	304	344	Similar to PR-1538	Stycast 1090	-125	127	Same				Similar to Stycast 1090		
			304					127							

PC 22	AVG. + 23	247 242 241 241 255	268 > 360 366	Similar to PR-1538	AVG. -125	129 255 249 244 243 242 233 215 203 170 228	Same as 1st failure	Continuous flame occurs between electrodes at 241 seconds until conductive path forms. (If path has not already been formed.)
	AVG. +125	126 124 123 123 122 122	230	Similar to PR-1538	AVG. + 23	197 195 189 188 188 165 183 172 161 155 181	Same	Similar to Stycast 1090
	AVG. -125	123 122 121 122	181 238	Similar to PR-1538	AVG.	160 160 150 150 140 152	Same	Similar to Stycast 1090
Pro-Seal 704	AVG. + 23	350 280 265 247 243 243 271	> 360	Similar to PR-1538	AVG. -125	372* 342 309 303 302 298	Same	Similar to Stycast 1090
	AVG. +125	125 123 123 122 122 125	267	Similar to PR-1538	AVG.	268 254 244 242 285*	Same	

\* The value of 372 is not included in the average because it was obtained by extending the test with the 30 ma arc for 12 seconds.

means that failure occurred quickly during the third step. In consequence, it is difficult to make a very meaningful comparison of the results.

Nevertheless, it is apparent that the first four materials in Table 3-2 are similar. The results for the Stycast materials differ to a significant degree. The Stycast materials failed upon formation of a conducting path. The other materials failed by what has been termed a "flame" failure along with considerable melting and apparent depolymerization.

At the low temperature, all of the values are higher than at room temperature, but the type of failure for a given material ultimately is about the same. It seems likely that the cold specimen is able to absorb more energy before failure takes place. It is possible that a very thin layer of ice may have formed on the test specimens, but if present, the ice was not visible.

At higher temperatures the results for the first four materials in the tabulation are about the same as at room temperature, although the tendency to melt under the influence of the arc increased. The higher temperature did adversely affect the results with the Stycast materials.

Some comment should be made about the significance of the ASTM Arc Resistance Test as related to the proposed use of the materials. The test makes use of a high voltage-low current arc above a dry surface. It does not indicate the performances to be expected if high current arcs are involved in service or if wet and contaminated conditions prevail. Moreover, only an order of magnitude for comparison should be made. For example, the materials described in this report are all better than conventional molded or laminated phenolics which usually have values below 15 seconds and, except for Stycast 2850, are all poorer than a melamine-glass laminate which normally fails at about 185 seconds.

Many factors that affect the actual performance of materials may not be reflected in the D-495 test. For example, if the arc is confined (as at the interface between two insulating surfaces) or is held close to a surface, a conductive path type failure may occur much more easily than is indicated by the conventional D-495 test. Since the D-495 method is such a qualitative test, it is doubtful that the results at low and high temperatures have much significance and it is not recommended that such tests be made.

Table 3-3. Arc Resistance at 13<sup>o</sup>C, D-495 Stainless Steel Strip Electrodes

Material	1st Failure (seconds)	Comments
PR-1538	121	Flame failure during the first 60 seconds seems to be critical with spacing - at slightly more than 1/4 inch, flame occurs at each tip; at a 1/4-inch spacing, a nearly but not quite continuous flame forms between the electrodes. At slightly less than a 1/4-inch spacing, a continuous flame forms between the electrode.
PC 22	122	
PR-1535	122	
Pro-Seal 794	80	
Stycast 1090	12	A conductive path started to form with the first arc but was not completed until the time shown.
	12	
	8	
Stycast 2651	48	A conductive path started at approximately 15 seconds and was complete at the time shown.
	40	
	24	
Stycast 2850	130	A track tried to start at 14, 22, and 456 seconds but did not.
	127	A conductive path formed rapidly.
	127	A conductive path formed rapidly.
	30	A track started at 20 seconds.
Test with Quartz Plate and Flat Stainless Steel Electrodes (Confined Arc)		
Stycast 2850	10	Conductive path
	8	
	6	
Pro-Seal 794	6	Conductive path
	4	
Pr-1538	16	Conductive path
	14	
	12	

### 3.7.5 VARIATIONS IN ARC RESISTANCE TEST METHOD

As mentioned in the discussion of the data above, test parameters may very markedly affect test results. This situation is recognized in ASTM D-495 where stainless steel strip electrodes are included as an alternative to the tungsten rod electrodes that are normally used. With strip electrodes, the arc is held close to the surface of the test specimen and the test results may differ from those obtained with the rod electrode.

The arc may be even further confined if a quartz glass plate is placed over the test specimen with the stainless steel strip acting as a spacer. A 500-gram load is used to hold plate and specimen together. This method has just been developed by the General Electric Company in order to show the marked effect of a confined arc.

#### 3.7.5.1 Test Results

Test results at 23°C using the two test modifications are given in Table 3-3. Only a sufficient number of tests were made to indicate the trend of the results.

#### 3.7.5.2 Discussion of the Data

The test results shown in Table 3-4 use "average" values even though in some cases only one value was available. Consequently, emphasis should be placed primarily on comparison and not so much on the values by themselves.

Table 3-4. Average Arc Resistance-Seconds

Material	Tungsten Rod Electrodes	Stainless Steel Strip Electrodes	Strip Electrodes Confined Arc
PR-1538	123	121	14
PR-1535	123	123	
PC 22	123	122	
Pro-Seal 794	123	80	5
Stycast 1090	40	11	
Stycast 2651	129	37	
Stycast 2850	181	104	8

It is immediately obvious that the arc resistance of the materials tested in different ways do not have the same order of comparison. With the confined arc, the performance of all material is poor and a conducting track was obtained in every case rather than a flame failure. The comparison between results for tungsten rod and stainless steel strip electrodes is good for the PR-1538, PR-1535 and the PC 22 materials and poor for the others. Obviously, the different materials are affected in different ways by the variation in the parameters of the test.

It can be concluded that the ASTM 495 Arc Resistance Test may not provide a good guide for the selection of materials even when the other limitations such as contamination and higher currents are not considered. At best the ASTM 495 test can provide only a very general guide and in some situations may actually be misleading.

SECTION 4  
MECHANICAL PROPERTIES

4.1 TENSILE STRENGTH - ELONGATION - TENSILE MODULUS

Mechanical property determinations of tensile strength, elongation and tensile modulus were made at  $-125^{\circ}\text{C}$ , room temperature (RT), and  $125^{\circ}\text{C}$ . Test procedures become extremely complicated by the following:

1. The material types considered here have an intrinsic elongation specification minimum varying from 100 to 500 percent. If a standard length specimen (as defined in the appropriate test standard) is used, the testing machine quickly "runs out" of capability, particularly when an environmental chamber is in place. In addition, recovery following rupture places an uncertainty upon what is true elongation, the total extension at rupture, or a final elongation between gauge marks. The problem is further complicated because permanent gauge marks cannot be placed on some of the materials used; in some cases the entire specimen (dog-bone) elongated during the test, not just the reduced section.
2. In using butt-faced grips, extreme difficulty was found in holding specimens during the test. Too low a pressure would allow the specimen to creep out of the grip during loading, and too heavy a pressure resulted in cutting the specimen at the grip face edge with premature failure.
3. Modulus of elasticity is normally determined using an extensometer. While an incremental extensometer useful to extensions of 1000 percent is available, this type of equipment can be used only at or near room temperature. Extensometers covering the temperature range required in this study are not available and other means of determining modulus were required.

To solve these problems, the following procedures were used:

1. A micro-tensile specimen, 0.876-inch long with a 0.187-inch width, was used for tensile strength and elongation determinations of plastic materials; however, this specimen configuration is not suitable for modulus of elasticity determination (ASTMD 1708-59T).\*

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\* 1/2-inch gauge length is needed for determination of percent elongation.

2. Special elastomeric grips, which have a unique gripping system that virtually eliminates slipping, were procured for these tests. Following discussions with Instron Corporation and General Electric R&D Center, Schenectady, the following procedures were established.

Tensile strength and elongation were determined using micro-tensile specimens held with butt-face grips. The elongation was determined as the total extension at fracture, since the specimen, as gripped, is almost all reduced specimen. Separate modulus specimens were 1/2-inch wide, gripped in the straight section and tested to fracture.

#### 4.1.1 TEST METHODS

Tangent modulus was calculated for low temperature determinations while secant modulus is reported for room temperature and elevated temperature determinations according to ASTM 638. Since the stress/strain curves do not generally show proportion of stress to strain at room and elevated temperature, secant modulus is then the appropriate property to report at these temperatures. The following procedure was used to calculate tangent and secant modulus.

For determination of modulus at  $-125^{\circ}\text{C}$ , the initial tangent modulus was calculated by drawing the slope tangent to the initial portion of the load versus deflection curve. Cold temperature specimens indicated a proportional stress versus strain relationship that allowed a tangent line to be constructed. The RT and  $125^{\circ}\text{C}$  specimens, because of their elastic properties, did not exhibit an initial proportional stress versus strain curve. By use of the secant modulus constructed at a 20 percent strain increment, a representative modulus could be calculated. For the RT specimens an incremental extensometer was used for recording percent strain. However, for  $+125$  and  $-125^{\circ}\text{C}$  the movement of the testing machine crosshead was used for determination of strain.

A Missjmers chamber was used for elevated or reduced temperature testing with the specimen being held for at least 30 minutes at temperature before testing. Temperature was monitored by means of a thermocouple placed in promixity to the specimen.

#### 4.1.2 DISCUSSION OF THE DATA

Tables 4-1 through 4-6 give the tensile, elongation and modulus values for PR1535, Pro-Seal 794, PC22, PR1538, Sylgard 182 and RTV 602, respectively.

For elastomeric potting compounds PR1535 and Pro-Seal 294, cooling below room temperature drastically increases tensile strength and modulus and decreases elongation. Heating to 125°C decreases tensile strength and elongation of PR1535, decreases tensile strength and increases elongation of Pro-Seal 294, but has very little effect on modulus values. Room temperature tensile strength and elongation meet the MSFC 202 requirement.

Tables 4-3 and 4-4 show the properties of elastomeric conformal coatings PC22 and PR1538. Again, cooling drastically increases tensile strength and modulus and reduces elongation. Heating to 125°C decreases tensile strength of both materials and the elongation of PR1538, but has only a minor effect on elongation of PC22, and modulus of either material. In general, RT properties do not meet the requirements of MSFC 393A.

Tables 4-5 and 4-6 show the data collected for Sylgard 182 and RTV 602. Neither of these materials meet RT tensile or elongation requirements of MSFC 379B. Cooling to -125°C increases tensile strength, modulus and elongation of Sylgard 182 while the RTV 602 tensile strength and modulus are increased and elongation decreased upon cooling. Heating to 125°C almost destroys the tensile strength of Sylgard, increases elongation and has no effect upon its modulus. Heating of RTV 602 to 125°C has no effect on tensile strength, but increases elongation and reduces modulus.

#### 4.2 LOW TEMPERATURE FLEXIBILITY

The low temperature flexibility test was performed on PR-1535, Pro-Seal 798, Sylgard 182 and RTV 602 per the applicable provisions of the MSFC specifications. The low temperature flexibility jig specified in MSFC 379 was used also for the materials covered by MSFC 202.

Table 4-1. Mechanical Properties of PR-1535

Test Temperature (°C)	Tensile Properties				Modulus			Remarks
	Spec No.	Spec Size (1)	Tensile Strength (psi) (3)	Elongation (%) (3)	Spec Size (2)	Result (ksi)	Crosshead Speed (in./min)	
-125	1	MT	17500	20	Straight	192	0.05	Initial tangent modulus Initial tangent modulus Initial tangent modulus
	2	MT	17300	20	Straight	240	0.05	
	3	MT	17700	20	Straight	210	0.05	
	AVG	-	17500	20	AVG	216	-	
RT	1	MT	3630	450	Straight	1.6	20	Secant modulus Secant modulus Secant modulus
	2	MT	2990	474	Straight	1.64	20	
	3	MT	3930	470	Straight	1.72	20	
	AVG	-	3520	465	AVG	1.65	-	
125	1	MT	520	380	Straight	1.3	20	Secant modulus Secant modulus Secant modulus
	2	MT	580	400	Straight	1.5	20	
	3	MT	560	420	Straight	1.5	20	
	AVG	-	550	400	AVG	1.43	-	

- NOTES: (1) Micro-tensile specimen: 0.5-inch gauge length - crosshead speed 2 in./min.  
(2) Reduced section specimen gripped on straight portion, 2 inch gauge length.  
(3) MSFC 202 room temperature requirements - tensile strength - 2560 psi min., elongation - 300% min.

Table 4-2. Mechanical Properties of Pro-Seal 794

Test Temperature (°C)	Tensile Properties				Modulus			Remarks
	Spec No.	Spec Size (1)	Tensile Strength (psi) (3)	Elongation (%) (3)	Spec Size (2)	Result (ksi)	Crosshead Speed (in./min)	
-125	1	MT	11100	18	Straight	255	0.05	Initial tangent modulus
	2	MT	15380	20	Straight	255	0.05	Initial tangent modulus
	3	MT	15170	20	Straight	290	0.05	Initial tangent modulus
	AVG	-	13680	19	AVG	267	-	
RT	1	MT	2740	590	1	1.36	20	Secant modulus
	2	MT	2860	580	2	1.32	20	Secant modulus
	3	MT	2650	570	3	1.48	20	Secant modulus
	AVG	-	2750	580	AVG	1.39	-	
	1	MT	780	640	1	0.96	20	Secant modulus
	2	MT	660	560	2	1.4	20	Secant modulus
125	3	MT	779	700	3	1.3	20	Secant modulus
	AVG	-	740	633	AVG	1.2	-	

NOTES: (1) Micro-tensile specimen: 0.5 inch gauge length - crosshead speed 2 in./min.  
 (2) Reduced section specimen gripped on straight portion, 2 inch gauge length.  
 (3) MSFC 202 room temperature requirements - tensile strength - 2560 psi; min., elongation - 300% min.

Table 4-3. Mechanical Properties of PC 22

Test Temperature (°C)	Tensile Properties				Modulus			Remarks
	Spec No.	Spec Size	Tensile Strength (psi)	Elongation (%)	Spec Size	Result (ksi)	Crosshead Speed (in./min)	
-125	1	MT	10900	10	Straight	290	0.05	Initial tangent modulus Initial tangent modulus Initial tangent modulus
	2	MT	12700	10	Straight	320	0.05	
	3	MT	13200	10	Straight	256	0.05	
	AVG	-	12300	10	AVG	286	-	
RT	1	MT	1280	420	Straight	1.64	20	Secant modulus Secant modulus Secant modulus
	2	MT	1270	450	Straight	1.76	20	
	3	MT	1260	430	Straight	1.72	20	
	AVG	-	1270	430	AVG	1.7	-	
125	1	MT	320	440	Straight	.92	20	Secant modulus Secant modulus Secant modulus
	2	MT	380	440	Straight	1.2	20	
	3	MT	350	460	Straight	1.1	20	
	AVG	-	360	450	AVG	1.1	-	

NOTES: (1) Micro-tensile specimen: 0.5 inch gauge length - crosshead speed 2 in./min.  
 (2) Reduced section specimen gripped on straight portion, 2 inch gauge length.  
 (3) MSFC 393A room temperature requirements - tensile strength - 1400 psi min., elongation - 500% min.

Table 4-4. Mechanical Properties of PR-1538

Test Temperature (°C)	Tensile Properties				Modulus			Remarks
	Spec No.	Spec (1) Size	Tensile Strength (psi)	Elongation (%) (3)	Spec (2) Size	Result (ksi)	Crosshead Speed (in./min)	
-125	1	MT	13100	10	Straight	250	0.05	Initial tangent modulus Initial tangent modulus Initial tangent modulus
	2	MT	10500	8	Straight	210	0.05	
	3	MT	11000	10	Straight	275	0.05	
	AVG	-	11500	9	AVG	250	-	
RT	1	MT	1330	670	Straight	1.42	20	Secant modulus Secant modulus Secant modulus
	2	MT	1290	720	Straight	1.28	20	
	3	MT	1350	750	Straight	1.41	20	
	AVG	-	1320	713	AVG	1.37	-	
125	1	MT	310	130	Straight	0.88	20	Secant modulus Secant modulus Secant modulus
	2	MT	320	160	Straight	0.64	20	
	3	MT	320	140	Straight	0.61	20	
	AVG	-	320	140	AVG	0.72	-	

NOTES: (1) Micro-tensile specimen: 0.5 inch gauge length - crosshead speed 2 in./min.

(2) Reduced section specimen gripped on straight portion, 2 inch gauge length.

(3) MSFC 393A room temperature requirements - tensile strength - 1460 psi min., elongation - 500% min.

Table 4-5. Mechanical Properties of Sylgard 182

Test Temperature (°C)	Tensile Properties				Modulus			Remarks
	Spec No.	Spec (1) Size	Tensile (2) Strength (psi)	Elongation (3) (%)	Spec (2) Size	Result (psi)	Crosshead Speed (in./min)	
-125	1	MT	3300	220	Straight	48	0.05	Initial tangent modulus
	2	MT	2600	220	Straight	56	0.05	Initial tangent modulus
	3	MT	2900	230	Straight	-	-	Initial tangent modulus
	AVG	-	2900	220	AVG	52	-	
RT	1	MT	290	74	Straight	0.22	20	Secant modulus
	2	MT	260	80	Straight	0.21	20	Secant modulus
	3	MT	290	80	Straight	0.22	20	Secant modulus
	AVG	-	280	78	AVG	0.22	-	
125	1	MT	27	170	Straight	0.18	20	Secant modulus
	2	MT	15	110	Straight	0.22	20	Secant modulus
	3	MT	34	130	Straight	0.20	20	Secant modulus
	4	MT	18	80	Straight			Secant modulus
	AVG	-	23	120	AVG	0.20	-	

NOTES: (1) Micro-tensile specimen: 0.5 inch gauge length - crosshead speed 0.5 in./min.  
 (2) Reduced section specimen gripped on straight portion, 2 inch gauge length.  
 (3) MSFC 329B room temperature requirements - tensile strength 350 psi min., elongation - 100% min.

Table 4-6. Mechanical Properties of RTV 602

Test Temperature (°C)	Tensile Properties				Modulus			Remarks
	Spec No.	Spec (1) Size	Tensile Strength (psi)	Elongation (%) (3)	Spec (2) Size	Result (ksi)	Crosshead Speed (in./min)	
-125	1	Straight (2)	3300	10	Straight	48	0.05	Initial tangent modulus
	2	Straight	2000	5	Straight	48	0.05	Initial tangent modulus
	3	Straight			Straight	44	0.05	Initial tangent modulus
	AVG		2600	8	AVG	47	-	
RT	1	MT (1)	40	80	Straight	0.048	20	Secant modulus
	2	MT	40	90	Straight	0.052	20	Secant modulus
	3	MT	40	70	Straight	0.056	20	Secant modulus
	AVG	-	40	80	AVG	0.052	-	
125	1	MT	43	130	Straight	0.032	20	Secant modulus
	2	MT	43	120	Straight	0.032	20	Secant modulus
	3	MT	43	120	Straight	0.032	20	Secant modulus
	AVG	-	43	120	AVG	0.032	-	

NOTE: (1) Micro-tensile specimen: 0.5 inch gauge length - crosshead speed 0.5 in./min.  
 (2) Reduced section specimen gripped on straight portion, 2 inch gauge length.  
 (3) MSFC 320B room temperature requirements - tensile strength - 350 psi min., elongation 100% min.

The test fixture and coated specimens were placed in a Missimers Chamber and held a minimum of four hours at temperature. The specimens were then placed in the test fixture slot, bent by hand over the radius, removed and examined.

There was no blistering, cracking, or separation from the aluminum test strip for any of the materials at either ambient or  $-55^{\circ}\text{C}$ .

#### 4.3 PEEL STRENGTH

Peel strength of silicone potting materials Sylgard 182 and RTV 602, MSFC specification 379A, Type III, was determined according to Method 1041.1 of FTMS 175. The test consists of a 180-degree peel test of a flexible member bonded to a piece of flexible or rigid material. In this test, the flexible member chosen was cotton duck and the other member was 0.064-inch thick aluminum alloy.

For high and low temperature, the tests were conducted using a Missimers Chamber. A thermocouple was placed in close proximity to the grips and the specimens were held a minimum of 15 minutes at temperature before testing.

Table 4-7 shows the peel strength of the two materials. Heating from RT to  $125^{\circ}\text{C}$  reduces peel strength to about one-third of room temperature value. RTV 602 did not attain RT specification requirement, but Sylgard has a peel strength at  $125^{\circ}\text{C}$  equal to its RT requirement.

No peel strength determinations could be made at  $-125^{\circ}\text{C}$ ; the duck broke at the bond line and no peel was initiated.

Table 4-7. Peel Strength of Silicone Potting Compounds-Pounds/Inch

	-125°C	RT	125°C	RT Spec. Required Minimum
RTV 602	No peel <sup>(1)</sup>	1.32	.45	3
Sylgard 182	No peel <sup>(1)</sup>	10.0	3.2	3

Note <sup>(1)</sup> At -125°C, the silicone had embrittled the duck and it broke with no peel being initiated.

#### 4.4 TEAR RESISTANCE TESTS

Tear resistance was determined on the two elastomeric potting compounds and the two elastomeric conformal coating materials according to the test standard method for Tear Resistance of Vulcanized Rubber, ASTM D624-54. Specimens were cut from 0.125-inch thick cast slabs using die C, which produces an unnicked 90-degree angle specimen, 4 inches long. Tests at -125 and 125°C were performed in a Missimers Chamber. Temperature within the chamber was obtained from a thermocouple placed in close proximity to the specimen; specimens were held a minimum of 15 minutes in the chamber at temperature before testing at a crosshead speed of 20 in./min. Tear resistance test data is listed in Table 4-8.

Room temperature tear strength of MSFC 202 materials is only slightly better than specification minimum (250 lb/in.) but the 393 materials exceeded specification minimum of 125 lb/in.

At 125°C, the 393 materials lost approximately 85 percent of RT tear strength while the 202 materials lost 60 percent of RT tear strength. Tear strength at -125°C increases in an erratic manner.

Table 4-8. Tear Resistance of Elastomeric Materials - Pounds/Inch Avg.

Material	-125°C	RT	125°C	RT Spec. Required Minimum
PR-1535	347	380	83	250
Pro-Seal 794	488	259	103	250
PR-1538	546	183	26	125
PC 22	484	196	29	125

#### 4.5 COMPRESSION SET

Compression set was determined on two elastomeric and two conformal coating compounds according to ASTM D395-61, Method B (constant deflection). The deflection employed was 25 percent and the specimens tested at other than RT were allowed to return to RT before final measurements were made. The compression set is the percentage of original deflection that is not recovered. Deflection set data is given in Table 4-9. For all materials, RT compression set meets applicable MSFC specification requirements.

Obviously, these materials cannot be used at 125°C since they compress to the deflection employed and do not recover when cooled; i. e., a compression set of over 100 percent was measured. The reason for this phenomenon is that the material is compressed to the limit of the spacer blocks; upon removal and cooling to room temperature the specimens contract and the final thickness results in a compression set measurement that was greater than the original deflection.

Table 4-9. Compression Set Data (%)

Material	-125°C	RT	70°C	125°C
PR-1535	2.8	9.8	13.9	103
Pro-Seal 794	3.0	16.9	21.5	108
PR-1538	1.5	23.0	32.0	118
PC 22	1.0	22.8	31.1	114

4.6 ISOD IMPACT STRENGTH

Impact resistance of the three Emerson and Cummings materials was determined in accordance with the procedure outlined in ASTM D-256-56, Impact Resistance of Plastic and Electrical Insulating Materials. The only deviation from this procedure was that only three specimens were tested at each condition instead of the five specified.

The impact test technique used was the Cantilever Beam (ISOD Type) test. All specimens (Figure 4-1) were prepared and dimensioned according to the recommended procedure and size. The materials tested were dielectric materials that fall under specification MSFC 222B, which specifically references ASTM D-256. The materials were Emerson and Cummings EC-2850, EC-2651 and EC-1090. Tests were performed at -125°C (-193°F), 24°C (75°F) and +125°C (257°F) on each of the three materials.

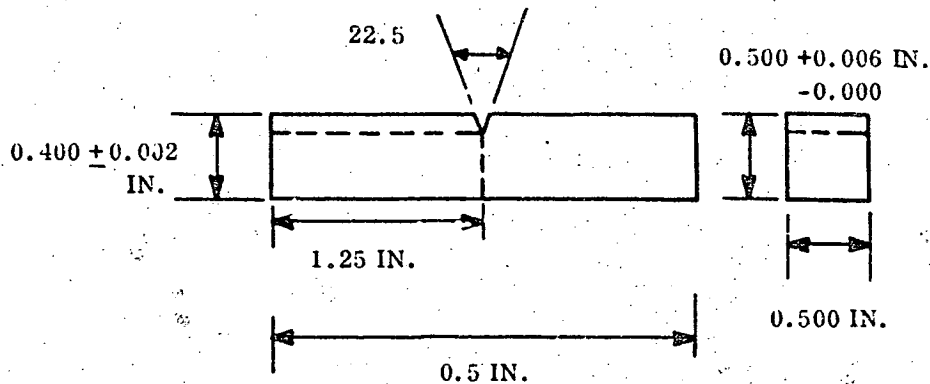


Figure 4-1. ISOD Impact Test Specimen

Results are presented in Table 4-10 for each test and material. Note that the difference between the ISOD Impact Strength and the Net ISOD Impact Strength is the Toss Factor (energy). The Toss Factor is the amount of energy per inch of notch required to throw the broken end of the specimen. Net ISOD Impact Strength is the total amount of energy per inch of notch required to deform the specimen, initiate fracture and propagate the fracture across the specimen. Mathematically, the Net ISOD Impact Strength is equal to the total measured energy loss during the test, per inch of notch, minus the sum of the Toss Factor and the friction loss factor.

Table 4-11 summarizes the data from Table 4-10 to show the effect of temperature of impact strength. Unfortunately, the data is quite inconclusive because of the scatter obtained when using only three specimens. One point to note is the very large increase in impact resistance for EC-2850 when going from RT to 125°C. Although some scatter exists, the data seems conclusive enough to indicate that this is a real effect. For EC-1090 and EC-2651 the scatter in the data makes any conclusion almost impossible. Finally, it should be noted that the two specimens of EC-2651 (EC-2651-OD1 and EC-2651-OD2) that were cut at 90 degrees from the rest of the EC-2651 specimens, showed a marked increase in impact strength (Table 4-10). This is indicative of preferential orientation within the material and should be investigated in more detail.

#### 4.7 ADHESIVE BOND STRENGTH

Adhesive bond strength (ABS) was determined as follows:

1. Elastomeric conformal coating (MSFC 393)
  - a. Cotton duck bonded to aluminum using the coating (metal).
  - b. Cotton duck bonded to fiberglass using the coating (fiberglass).
2. Elastomeric potting compound (MSFC 202)
  - a. Cotton duck bonded to aluminum with the compound (metal).

Table 4-10. ISOD Impact Test Results for Materials of Less Than 0.5 Ft-Lb per In. of Notch

Sample Designation	Test Temperature (°F)	ISOD Impact Strength (Ft-Lb/in. of Notch)	TOSS Factor (Energy) (Ft-Lb/in. of Notch)	Net ISOD Impact Strength (Ft-Lb/in. of Notch)
EC-1090-5	75	0.204	0.104	0.100
EC-1090-10	75	0.194	0.096	0.098
EC-1090-15	75	0.214	0.124	0.090
Mean	-	0.204	0.108	0.096
EC-2850-1	75	0.320	0.196	0.124
EC-2850-4	75	0.344	0.234	0.110
EC-2850-6	75	0.360	0.198	0.162
Mean	-	0.341	0.209	0.132
EC-2651-3	75	0.354	0.168	0.186
EC-2651-7	75	0.382	0.188	0.194
EC-2651-13	75	0.374	0.168	0.206
Mean	-	0.370	0.175	0.195
** EC-2651-OD1	75	0.440	0.174	0.266
** EC-2651-OD2	75	0.434	0.178	0.256
Mean	-	0.437	0.176	0.261
EC-1090-4	-193	0.162	0.108	0.054
EC-1090-9	-193	0.190	0.108	0.082
EC-1090-14	-193	0.162	0.088	0.072
Mean	-	0.171	0.101	0.069
EC-2850-5	-193	0.360	0.252	0.108
EC-2850-11	-193	0.376	0.204	0.136
EC-2850-16	-193	0.380	0.208	0.172
Mean	-	0.372	0.221	0.139
EC-2651-5	-193	0.354	0.184	0.170
EC-2651-10	-193	0.420	0.180	0.240
EC-2651-15	-193	0.346	0.178	0.168
Mean	-	0.373	0.181	0.193
EC-1090-3	257	0.200	0.100	0.100
EC-1090-2	257	0.176	0.094	0.082
EC-1090-13	257	0.198	0.084	0.114
Mean	-	0.191	0.093	0.099
EC-2850-3	257	0.766	0.204	0.562
EC-2850-8	257	0.676	0.193	0.474
EC-2850-17	257	0.680	0.202	0.478
Mean	-	0.705	0.201	0.505
EC-2651-2	257	0.338	0.185	0.152
EC-2651-9	257	0.346	0.143	0.198
EC-2651-14	257	0.346	0.172	0.174
Mean	-	0.343	0.169	0.175

\* Friction loss factor was 0.080 ft-lb/in. of notch for all of the above tests.  
 \*\* Specimens cut at 90-degrees to the rest of the specimens of this material.

- b. Neoprene bonded to a 1/4-inch thick slab of the compound (neoprene) contained in an aluminum plate.
- c. PVC bonded to a 1/4-inch thick slab of the compound (PVC) contained in an aluminum plate. (The plate referred to was backed by a piece of aluminum sheet.)

Where required by the specification and sample preparation description, primers recommended by the materials manufacturer were used in assembling the various samples. After assembly and cure, individual specimen setups, etc., were prepared from the samples as required by the specification.

The 180-degree pull tests were made using an Instron Tester at a crosshead speed of 2 in. / min. For tests at 125 and -125°C, a Missimers Chamber was used. Temperature was monitored by a thermocouple in close proximity to the sample. Average load from the load curve was estimated for each test and the average of these tests reported. Table 4-12 shows the adhesive bond strengths determined.

Table 4-11. Summary of ISOD Impact Strengths

Material	Temperature, °C (°F)	Net ISOD Impact Strength (Ft-Lb/In. of Notch)
EC-1090	-125 (-193)	0.069
	24 ( 75)	0.096
	+125 ( 257)	0.099
EC-2850	-125 (-193)	0.139
	24 ( 75)	0.132
	+125 ( 257)	0.505
EC-2651	-125 (-193)	0.193
	24 ( 75)	0.195
	+125 ( 275)	0.175

Table 4-12. Summary of Adhesive Bond Strength Data-Lb/In.

Bond Material	Combination	-125°C	RT	125°C	RT Requirement	Remarks
PC 22	Duck-Metal	N. T. (1)	2.9	1.9	15	
PC 22	Duck-Fiberglass	0.9 (2)	10.7	3.9	15	RT reached 15 lb/in.
PR-1538	Duck-Metal	N. T. (1)	19.9	7.3	15	
PR-1538	Duck-Fiberglass	9.7 (2)	12.2	0.65	15	1 RT sample avg. 18.5 lb/in.
PR-794	Duck-Metal	N. T. (1)	2	3.2	15	Duck broke at -125°C
PR-794	PVC-slab	5 (3)	58	.7	15	PVC broke at low temperature
PR-794	Neoprene-slab	N. T. (4)	15.2	N. T. (5)	15	
PR-1535	Duck-Metal	N. T. (1)	17.2	3.8	15	
PR-1535	PVC-slab	12 (3)	2.8	1.1	15	
PR-1535	Neoprene-slab	N. T. (4)	12.3	N. T. (5)	15	Load to break fillet at RT approximately 55 lb/in.

- Notes: (1) - Duck embrittled and broke, no test  
 (2) - Duck broke  
 (3) - PVC tore at bond line  
 (4) - Neoprene broke in brittle fashion - no test  
 (5) - Neoprene split and tore, no test

It is obvious at once that the 180-degree peel tests at  $-125^{\circ}\text{C}$  are of little or no value because both the coated duck and the neoprene embrittled and snapped at very low, non-measurable loads. In the no-test category of Table 4-12, as the sample was loaded it broke faster than the recorder response capability (calibrated for low loads) and no measure of breaking strength could be recorded. In any case, peeling was seldom initiated at  $-125^{\circ}\text{C}$  and the indicated loads are those at which the adherent failed.

Where satisfactory peeling occurred, the elevated temperature ABS was lower than RT ABS in all but one case. The data that shows the ABS to be higher for Pro-Seal 794 at  $125^{\circ}\text{C}$  than at RT is anomalous. Peak loads encountered at RT in no case approached the average ABS at  $125^{\circ}\text{C}$ .

Four of the ten combinations had an ABS meeting the RT requirement of the specification. Two other combinations had peak values that equalled or exceeded the specification requirement while another (PR-1535/Neoprene) required 55 lb/in. to break the initial bond.

SECTION 5  
PHYSICAL PROPERTIES

5.1 THERMAL SHOCK

Thermal shock was performed on EC-1090, EC-2651, and EC-2850 using specimens prepared according to paragraph 4.8.17 of MSFC specification 222C. Specimens were molded to the size specified by MIL-I-16323 and a 1-inch long by 0.5-inch hexagonal aluminum bar was molded in the specimen for expansion. Shock temperature range was 194<sup>o</sup>F to that of liquid nitrogen. Each material was cycled five times. The specimens were placed pin side down on an aluminum spider suspended in a standard cabinet for 30 minutes, removed, plunged into LN<sub>2</sub> for 10 minutes, removed and examined. After examination, the cycle was repeated.

EC-1090 and EC-2651 appeared to be cracked at the end of cycle 1 and were definitely cracked by the end of the second cycle. Heavy cracking occurred on the specimens after five cycles (Figure 5-1). At the end of the fourth cycle, the specimen of EC-2850 showed a crack-like indication. At the end of cycle 5, microscopic examination (25X) showed that the cracks were actually wrinkles analogous to a drying paint skin and are not cracks (Figure 5-1).

Conclusions

Under five thermal shocks varying from 198<sup>o</sup>F to liquid nitrogen temperature, EC-1090 and EC-2651 failed by cracking; EC-2850 developed no cracks but did develop crack-like indications that were actually skin ripples.

## 5.2 FLAME RESISTANCE OF ECCOFOAM FPH

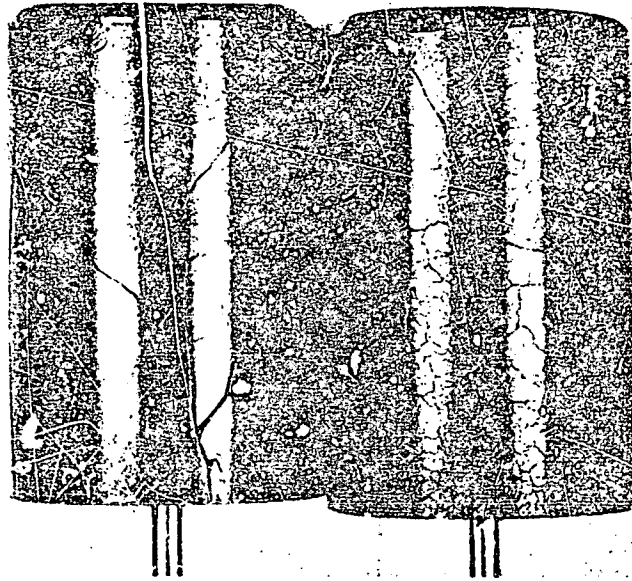
This test was performed according to ASTM-D-1692 except that the test samples were 2 by 4 by 1/2 inch instead of the specified 2 by 6 inches. Upon application of the Bunsen burner flame, all samples ignited and continued to burn completely upon removal of the flame. This material is classed "burning by this test" whereas specification MSFC 418 requires a flame resistant foam.

## 5.3 HARDNESS AFTER FULL CURE

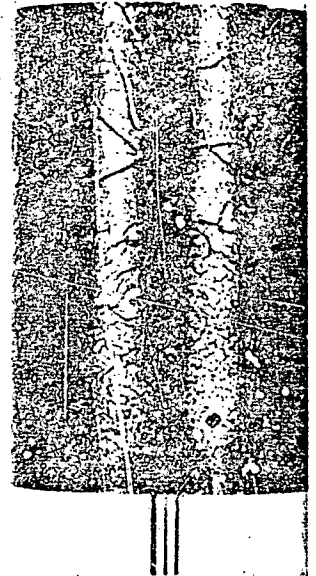
Hardness after full cure at 125 and -125°C per FTMS 406 or 601 could not be obtained. The absolute temperature limits for the Shore instrument are -45°C to 52°C and in no case is it permissible to test material at temperatures outside these limits without damaging the equipment. Therefore, hardness determinations at -45°C, RT, and 52°C were made after soaking the specimens for 2 hours at temperature and the test instrument for 1 hour before testing.

Test materials were placed on an 1/2-inch thick aluminum plate in a Standard cabinet and subjected to the appropriate heating or cooling cycle. When necessary, material was piled up, as allowed by the standard, to give a thickness of at least 1/4 inch. Five readings were made on each material within the cabinet and a short period (10 minutes) of reheating or cooling was allowed before the next set of hardness determinations was made. At the low temperature, frost was scraped from the sample using a doctor's blade before u.e. determinations were made.

Table 5-1 lists the hardness values obtained. Elastomeric potting compounds (PR-1535 and Pro-Seal 794) and conformal coatings (PC 22 and PR-1538) increase in hardness as the temperature is lowered, while the silicone potting materials (Sylgard 182 and RTV 602) soften as the temperature decreases. In addition, RTV 602 did not meet the specification requirement for hardness.

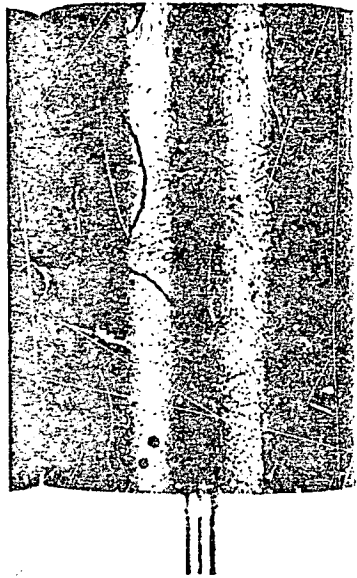


(a) EC-2651

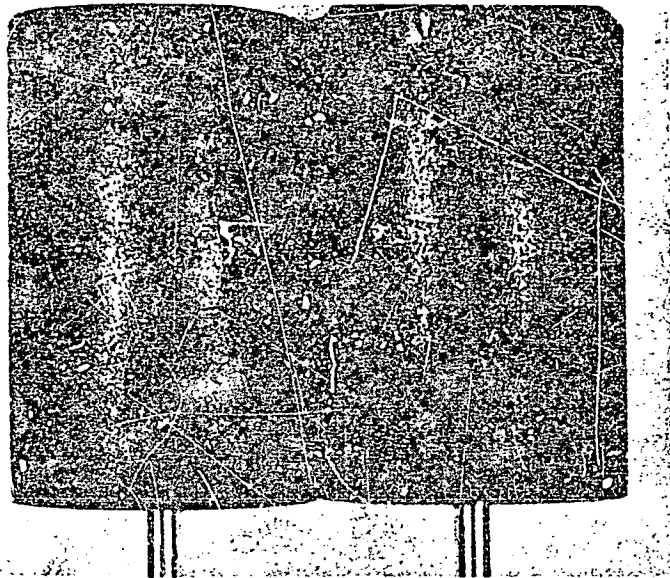


(b)

FOLDOUT FRAME |



EC-1090



(c) EC-2850 (Material is not cracked - indications are surface ripples)

Figure 5-1. Thermal Shock Cracking After Five Cycles from 198 to -320°F

Table 5-1. Shore -A Hardness After Full Cure

Material	Specification Requirements, RT	Temperature (°C)		
		-45	RT	52
PR-1535	76-99	94	86	88
Pro-Seal 794	76-99	96	83	84
PC 22	70-90	93	79	79
PR-1538	70-90	99	77	74
Sylgard 182*	35 minimum	39	45	47
RTV 602*	35 minimum	19	21	23

Values are average of 5 determinations

\*Piled up, 3 thicknesses

#### 5.4 MOISTURE ABSORPTION

Moisture absorption of six materials was determined according to the requirements of their applicable specifications. These differences are described in the following paragraphs.

##### 5.4.1 TEST METHOD - MSFC 329A (SYLGARD 182 AND RTV 602)

Specimens 2 inches in diameter x 0.125-inch thick were conditioned by drying 24 hours at 50°C, cooled 24 hours at 23°C in a dessicator, and weighed. The specimens were immersed in distilled water at 23°C for 168 hours, removed, wiped dry with a cloth and weighed. Percent absorption was calculated as wet weight minus conditioned weight divided by wet weight.

##### 5.4.2 TEST METHOD - MSFC 418 (ECCOFOAM FPH)

Eight 1-cubic inch samples were cut from a large block of foam material. Zero absorption weight was determined by immersing each specimen under a 2-inch head

of distilled water at 23°C for 10 seconds, drained 10 seconds and weighed. The material was then reimmersed for 24 hours, removed, drained 10 seconds and weighed. Percent absorption was calculated on the basis of final weight minus zero weight divided by final weight.

#### 5.4.3 TEST METHOD - MSFC 222C (EC-1090, EC-2651 AND EC-2850)

These materials were tested according to Procedure A of ASTM-D-570. Specimens 2 inches in diameter by 1/8-inch thick were dried in an oven at 50°C for 24 hours, cooled and weighed. The specimens were immersed in distilled water at 23°C for 24 hours, wiped dry and weighed. Moisture absorption was calculated as

$$\frac{\text{Wet Weight} - \text{Conditioned Weight}}{\text{Conditioned Weight}} \times 100$$

#### 5.4.4 TEST RESULTS

Table 5-2 presents the moisture absorption values collected. EC-1090 has a value only slightly above the specification maximum (0.51 percent versus 0.5 maximum) while Eccofoam has a moisture absorption value varying from 1.8 to 3.4 percent versus a specification maximum of 1.4 percent. All other materials are within or fall below the specification maximum.

Table 5-2. Summary of Moisture Absorption Data (%)

MSFC Specification	222C			379		418
	1090	2651	2850	Sylgard 182	RTV 602	Eccofoam
Sample 1	0.49	0.09	0.06	0.03	0.45	3.0
2	0.51	0.09	0.06	0.03	0.47	3.4
3	0.53	0.10	0.06	0.04	-	1.8
Average	0.51	0.09	0.06	0.03	0.46	2.7
Specification Limit, Maximum	0.50	0.50	0.50	0.50	0.50	1.4

### 5.5 TEMPERATURE RESISTANCE, ECCOFOAM

This property was determined according to Procedure 2, Method 6011 of FTMS 406. The object of the test is to obtain weight and shape changes in plastic materials under various conditions of use, while Procedure 2 is designed to reveal poorly cured parts by developing cracks in these parts. Two-inch cubes cut from 4-inch cubes were measured, weighed, heated 72 hours at 60°C, cooled in a dessicator, remeasured and reweighed, and visually examined. Results of the test showed that:

1. There was no change in surface outline or general appearance and no cracks developed.
2. There was no change in the dimensions of the material.
3. Weight loss of the samples during this test were:

<u>Sample No.</u>	<u>Loss (%)</u>
1	1.33
2	1.35
3	1.35
4	1.36
Average	1.35

### 5.6 AGING STABILITY AND VOLUME CHANGE, COMPRESSIVE STRENGTH AND STRESS/STRAIN FOR ECCOFOAM FPH

#### 5.6.1 TEST METHODS

Property data was obtained from nominal 2 by 2 by 1-inch blocks cut from 4 by 4 by 4 inch cubes of the material. Specimens with the 1-inch direction both perpendicular and parallel to the direction of foam rise were prepared.

Compressive strength and stress/strain were determined by compressing the blocks at a crosshead speed of 0.1 in./min., determining the compressive stress at 5 percent intervals

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of strain and plotting the stress/strain curve. Compressive strength was obtained as the stress of 10 percent compression of original thickness.

Volume change was determined by calculating volume, exposing the material to 95 percent RH at 49°C for two weeks (in a closed system of glycerine-water of proper concentration held at constant temperature). After this conditioning, the volume was recalculated and percent change determined. The stress/strain diagram of these conditioned specimens was determined.

Compression tests were run on an Instron testing machine using a compression cage. A Missimers Chamber was used for high and low temperature ranges. The conditioned specimens were randomly chosen for the compression tests.

#### 5.6.2 VOLUME CHANGE

Table 5-3 gives the volume change associated with relative humidity conditioning. In general, specimens cut with the 1-inch direction perpendicular to the direction of rise had a small volume loss, averaging 0.35 percent while those with the 1-inch direction parallel to the rise direction "grew." Some of these parallel specimens had a configuration change in that the center portion of the specimen actually bowed, decreasing the effective dimension. These specimens were omitted from average, giving a mean growth of 0.48 percent.

#### 5.6.3 COMPRESSIVE STRENGTH

Compressive strength was taken as the average compressive stress at 10 percent deflection. Table 5-4 gives the compressive strength data collected. At room temperature, the relative humidity conditioned (aged) material in the parallel direction and the raw material in the perpendicular direction meets the strength requirement of MSFC Specification 418 based on the straight line function of density. The compressive strength at low temperature is increased by the aging, but at elevated temperature this property is virtually the same for all test parameters.

Table 5-3. Volume Change of Eccofoam FPII

Specimen No.	1-Inch Direction	Volume Change (%)	Specimen No.	1-Inch Direction	Volume Change (%)
1	Perpendicular	-0.16	10	Parallel	+0.51
2	Perpendicular	-0.52	11	Parallel	+1.59
3	Perpendicular	-0.52	12	Parallel	+0.55
4	Perpendicular	+0.51	13	Parallel	-0.67
5	Perpendicular	-0.25	14	Parallel	+0.25
6	Perpendicular	-0.46	15	Parallel	-5.5*
7	Perpendicular	-0.24	16	Parallel	+0.66
8	Perpendicular	-0.51	17	Parallel	-8.1
9	Perpendicular	-0.56	18	Parallel	-2.16
Average		-0.35			+0.48

\*Bowed specimen, not included in average

1-Inch direction either perpendicular to, or parallel to direction of rise of foam

Table 5-4. Compressive Strength of Eccofoam FPII

1-Inch Direction	Condition	Compressive Strength (psi)		
		Temperature (°C)		
		-125	RT	125
Parallel	Raw	31	49	21
Parallel	RII aged	136	78	21
Perpendicular	Raw	28	70	30
Perpendicular	RII aged	37	42	21

#### 5.6.4 STRESS/STRAIN

Stress/strain curves are shown in Figures 5-2 through 5-7, where "raw" and relative humidity (RH) conditioned (aged) curves are on the same plot. In all cases, the perpendicular direction shows a higher stress per percent of strain for the raw material than does the aged material, while in the parallel direction, the aged material has higher compressive stress per percent of strain.

At  $-125^{\circ}\text{C}$ , the material crumbles under the initial load. Substantial deformation then occurs with little load increase until about the 60 percent deformation level, after which the load increases. In most cases, the specimen was removed from test as a powder.

At room temperature, the curves are more nearly parallel than at  $-125^{\circ}\text{C}$  and a gradual increase in stress per unit of strain occurs. The stress increases more rapidly after strains of 50-55 percent.

At  $125^{\circ}\text{C}$ , the compressive stresses are lower and the curves are roughly parallel at lower strains but begin to diverge at higher strains. The material is definitely softer, with peak loads at 80 percent strain approaching 150 psi for perpendicularly cut material and 100 psi for the material cut in the parallel direction. In addition to the lower strength level associated with  $125^{\circ}\text{C}$  for this material, scorching of the specimen edges was observed at this temperature.

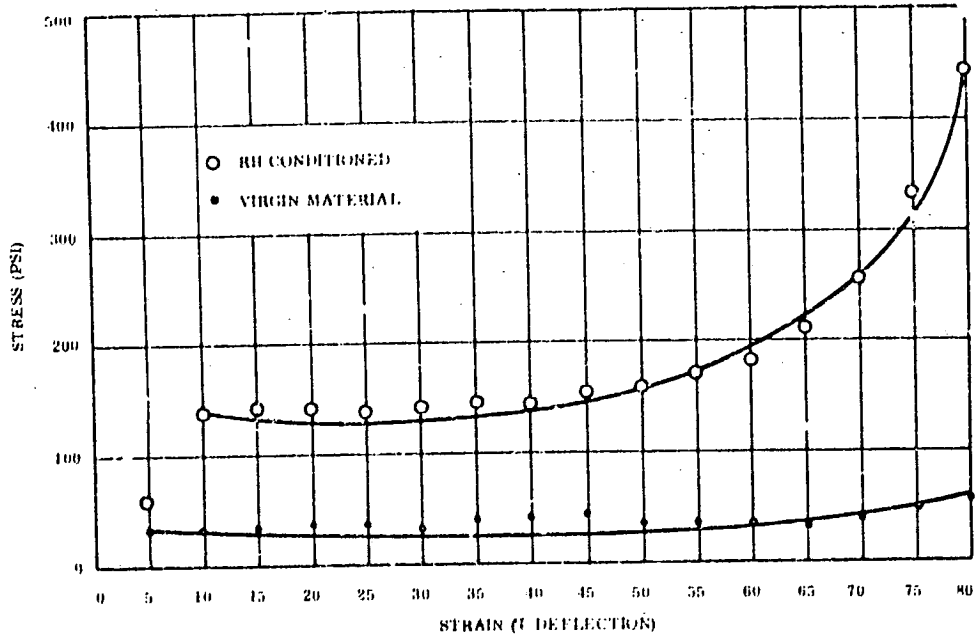


Figure 5-2. Stress/Strain Relationship Eccof foam FPII at  $-125^{\circ}\text{C}$   
Parallel to Foam Rise Direction

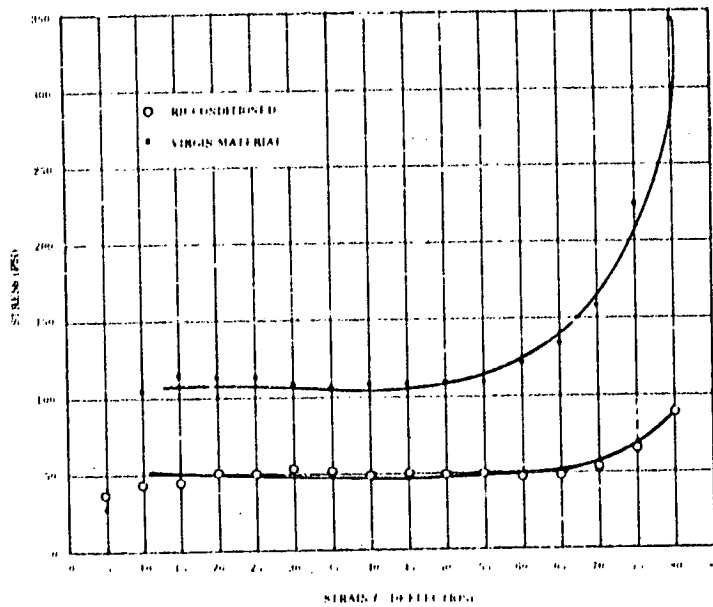


Figure 5-3. Stress/Strain Relationship, Eccof foam FPII at  $-125^{\circ}\text{C}$   
Perpendicular to Foam Rise Direction

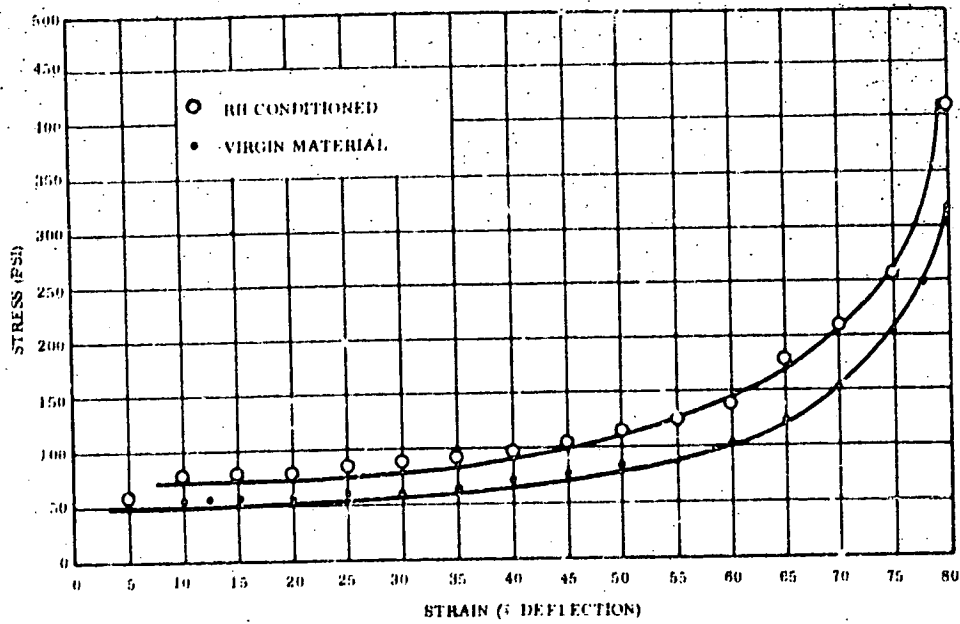


Figure 5-4. Stress/Strain Relationship, Eccofoam FPH at Room Temperature Parallel to Foam Rise Direction

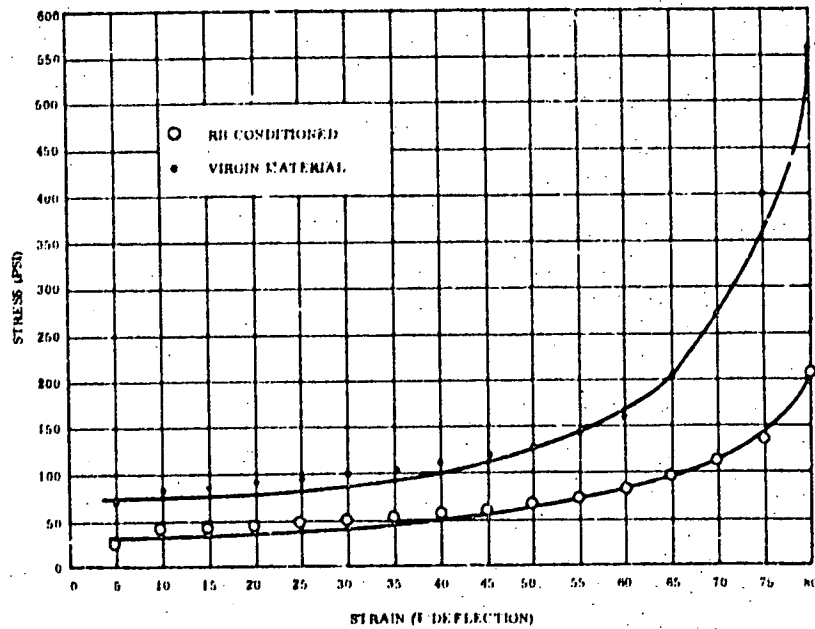


Figure 5-5. Stress/Strain Relationship, Eccofoam FPH at Room Temperature Perpendicular to Foam Rise Direction

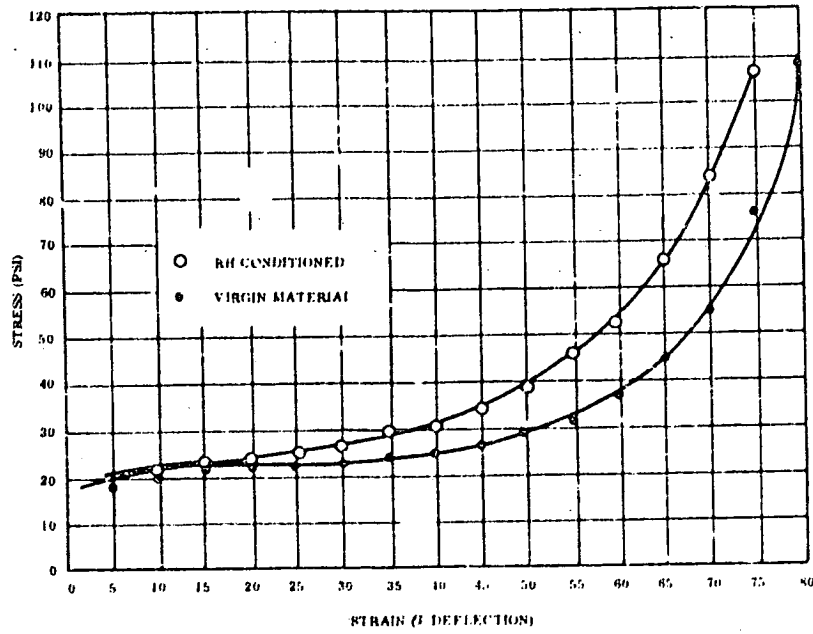


Figure 5-6. Stress/Strain Relationship, Eccofoam FPH at 125°C Parallel to Foam Rise Direction

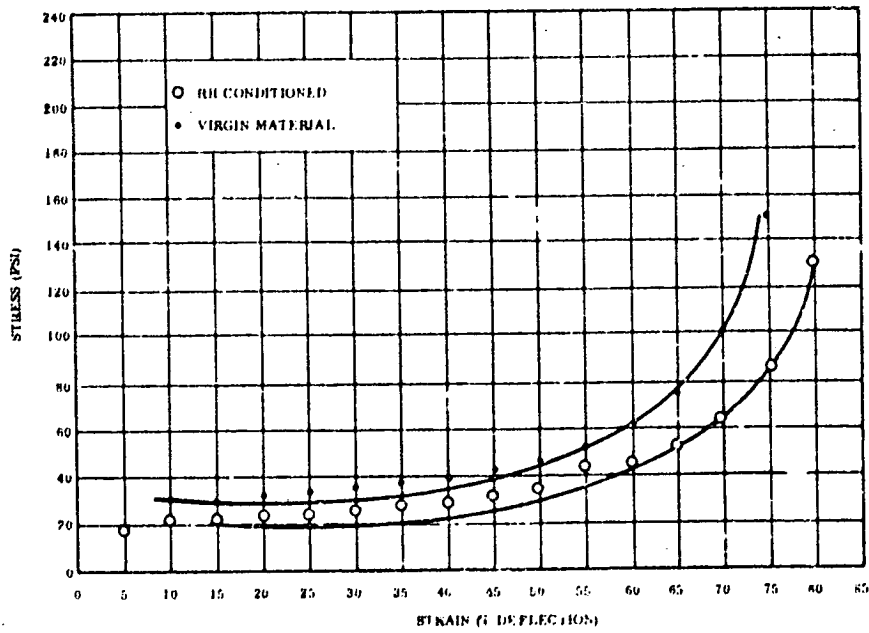


Figure 5-7. Stress/Strain Relationship, Eccofoam FPH at 125°C Perpendicular to Foam Rise Direction

SECTION 6  
ENVIRONMENTAL TESTS

6.1 OZONE RESISTANCE

6.1.1 TEST METHOD

Ozone resistance tests were performed on five specimens each of the four elastomeric materials listed below:

1. Sylgard 182
2. RTV 602
3. PR-535
4. Pro-Seal 794

An atmosphere of 50 parts per 100 million of ozone in air was used for all tests. Three separate tests were run for 7-day durations at temperatures of 125, 25 and  $-100^{\circ}\text{C}$ . (Note: testing at  $-125^{\circ}\text{C}$  was not attempted because the condensation temperature of  $\text{O}_3$  is reported as  $-112^{\circ}\text{C}$ .) Specimens were stretched to an elongation of 25 percent prior to placing them in the Ozonometer. All specimens were examined for signs of cracking or deterioration after each 7-day test with a 7-power magnifier as specified by the appropriate MSFC specification (i.e., 202A and 379A).

6.1.2 TEST RESULTS

Although no cracking was observed in any of the specimens, some material deterioration occurred particularly during the room temperature and  $125^{\circ}\text{C}$  tests. The results obtained are described in Table 6-1.

Table 6-1. Results of Ozone Resistance Tests

Material	Description of Samples After 7-day Exposures to Ozone at Temperature		
	As Received	-100° C	Room Temperature (25° C)
Sylgard 182	Clear, translucent, soft feel, tends to break on pulling apart.	No cracking, no color change, but samples broke upon stretching.	No cracking, no color change, but samples broke upon stretching.
RTV 602	Clear, translucent, soft feel, tends to break upon pulling apart.	No cracking, no color change, but samples broke upon stretching.	No cracking, no color change, but samples broke upon stretching.
Pro-Seal 794	Light amber in color, tough feel, hard to pull apart.	No cracking, no color change.	No cracking, slight darkening of exposed area.
PR-1535	Light amber in color, tough feel, hard to pull apart.	No cracking, no color change.	No cracking, slight darkening of exposed area.
			+125° C
			Slight yellowing of exposed area. No cracking, some loss of elasticity. Specimens broke upon stretching.
			Noticeable yellowing of exposed area. No cracking, some loss of elasticity. Specimens broke upon stretching.
			Extensive darkening to a dark reddish brown color. Some loss of elasticity, no cracking.
			Extensive darkening to a dark reddish brown color. Some loss of elasticity, no cracking.

## 6.2 FUNGUS RESISTANCE

### 6.2.1 TEST PROCEDURE

The requirements of MIL-E-5272C were used to conduct this test. The mixed spore suspension below was employed.

<u>Group</u>	<u>Organism</u>	<u>American Type Culture Collection Number</u>
I	Myrothexium Verrucaria	9095
II	Aspergillus Niger	6275
III	Aspergillus Flavus	10836
IV	Penicillium Citrinum	9849

The materials tested were nine of the materials being studied for this program, the exception being Eccofoam FPII. Test materials were sectioned to appropriate size and cleaned using a 70 percent isopropyl alcohol and 30 percent water blend. These samples were allowed sufficient time to dry before being bagged in separate envelopes. A sample of each candidate material was suspended on a stainless steel wire and another sample placed within the petri dishes containing the hardened culture media. Control specimens of filter paper, cork and leather were also placed on the culture media to verify the use of viable spores. Both the suspended samples and petri dish specimens were inoculated using a spray technique.

The test and control specimens were placed in a test chamber maintained at  $86 \pm 3.6^{\circ}\text{F}$  ( $30 \pm 2^{\circ}\text{C}$ ) and  $95 \pm 5$  percent relative humidity. A fungus growth on the control specimens after 3 days verified that viable spores were employed. The test materials were evaluated after 14-day and 28-day exposures. The following arbitrary numbering system was used to distinguish between no-growth and variations-in-growth accumulation.

<u>Number</u>	<u>Degree of Growth</u>
0	None
1	Light
2	Medium
3	Heavy
4	Copious

### 6.2.2 TEST RESULTS

The results of this test are given in Table 6-2.

Table 6-2. Results of Fungus Resistance Tests

Material	Degree of Growth (arbitrary scale 0-4)	Results per MIL-E-5272C
EC-2850	0	No growth
EC-2651	0	No growth
EC-1090	0	No growth
PC 22	0	No growth
PR-1538	1	Light growth
PR-1535	1	Light growth
Pre-Seal 794	2	Medium growth
Sylgard 182	0	No growth
RTV 602	0	No growth

### 6.3 ULTRAVIOLET RADIATION/WEATHERING TESTING

All ten materials included in this program were exposed to a one year equivalent sea level ultraviolet radiation (between 0.29 and 0.4 microns) and a high relative humidity condition at both 220° F and -90° F. The ultraviolet radiation dose was established by integrating the air mass and irradiance tables so that the maximum possible ultraviolet exposure attainable in one year on earth was simulated\*.

#### 6.3.1 TEST PROCEDURE

The ten materials were irradiated under a General Electric H23KX (H1500-A23) lamp. A two-dimensional parabolic reflector was used to collimate the light beam. Specimens were positioned on two plates under the collimator. The hot plate was operated at a temperature of 220 ± 20° F and the cold plate was maintained at a temperature of -90 ± 15° F. Liquid nitrogen was used to regulate temperature. Specimens were positioned as shown in Figure 6-1.

Total test time was 413 hours. Figure 6-2 is a photograph of the test setup after 100 hours; Figures 6-3 and 6-4 show the setup at the end of the test. Table 6-3 and Figures 6-5 and 6-6 give the spectral distribution data of the H23KX lamp used in this test.

#### 6.3.2 TEST RESULTS

As can be seen in Figure 6-2, the cold side of the test was continuously covered with frost. Attempts to prevent frost build-up by passing a stream of dry nitrogen over the specimens failed. Figure 6-2 also indicates that the tops of Eccofoam specimens were badly scorched in both the cold and hot sides of the test. This is attributed to the high insulative qualities of the foam. Because frost covered the bulk of the cold side samples, post-test evaluation of the materials was not made.

The Emerson and Cummings materials on the hot side were bleached almost white. The elastomeric conformal coating materials, potting materials and the silicone potting

\*Handbook of Geophysics, Revised Edition, 1960, The McMillan Co., New York, N. Y.

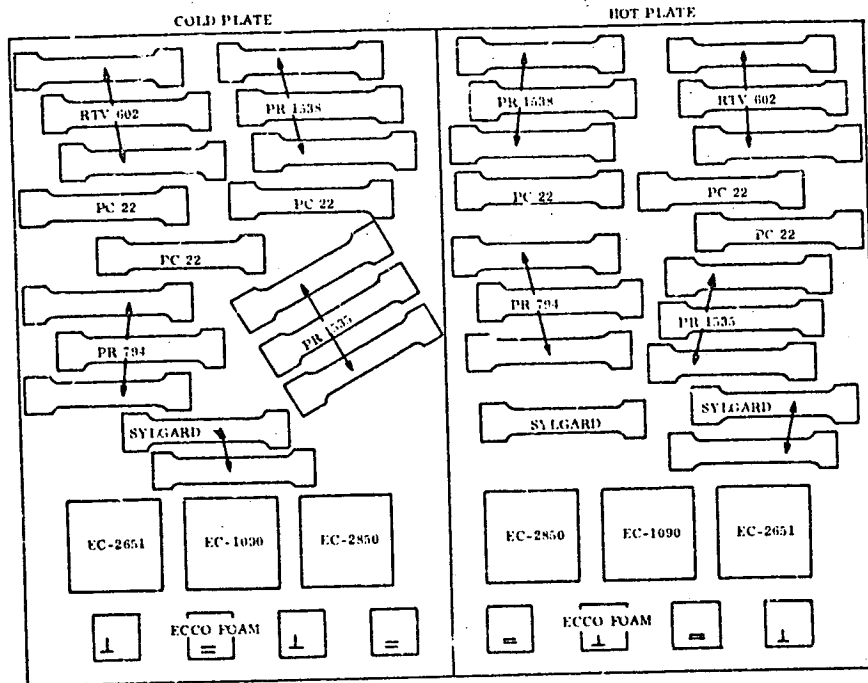


Figure 6-1. Specimen Locations

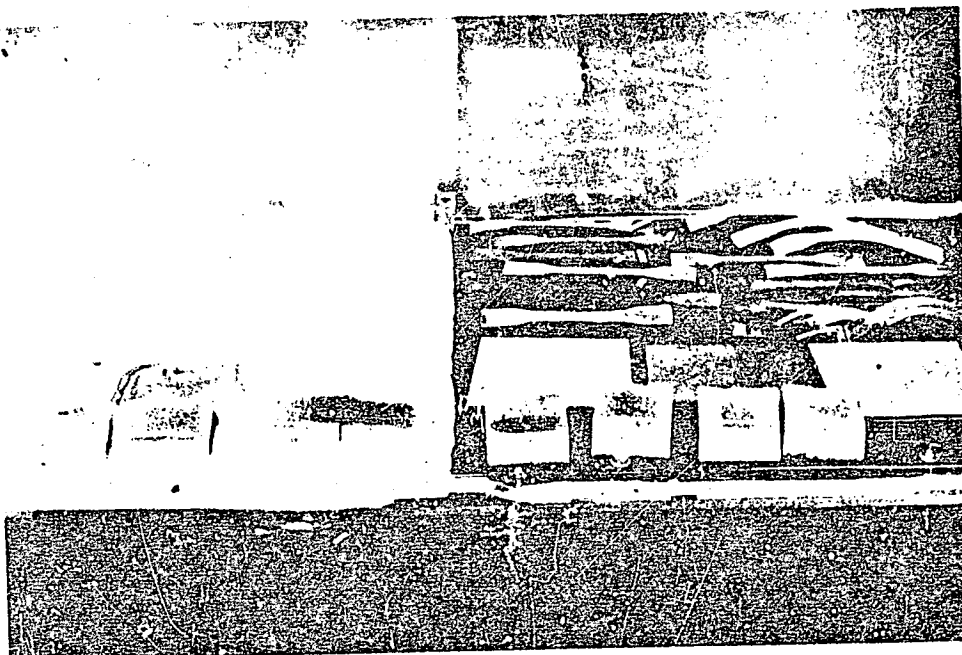


Figure 6-2. Samples After 100 Hours

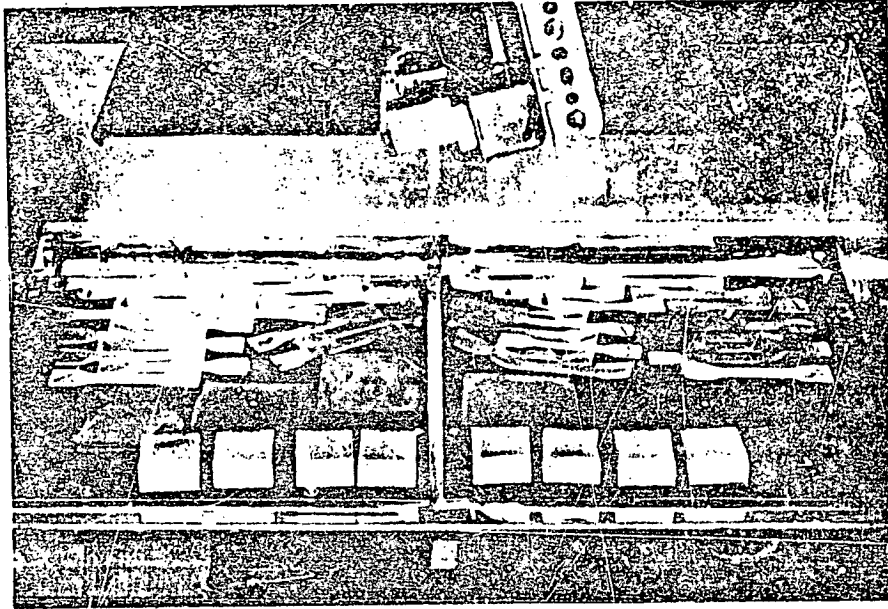


Figure 6-3. Samples After 412 Hours, View 1



Figure 6-4. Samples After 412 Hours, View 2

Table 6-3. Watts Radiated at Various Wavelength Bands

Wavelength Band (Angstroms)	Principal Lines	H1500-A23	
2200 - 2300	2259	18.017	Far Ultraviolet
2300 - 2400	2358	22.940	
2400 - 2500	2482	21.823	
2500 - 2600	2537	78.154	
2600 - 2700	2652	39.027	
2700 - 2800	----	9.003	
2800 - 2900	2804 - 2894	27.556	Middle Ultraviolet
2900 - 3000	2967	21.822	
3000 - 3100	3022	39.584	
3100 - 3200	3132	67.954	
3200 - 3300	----	8.069	Near Ultraviolet
3300 - 3400	3341	13.346	
3400 - 3500	----	5.633	
3500 - 3600	----	4.619	
3600 - 3700	3650	104.697	
3700 - 3800	----	3.451	
3800 - 4000		5.532	
4000 - 4100	4047	25.122	Visible
4100 - 4300	----	4.263	
4300 - 4400	4358	46.791	
4400 - 5400	----	13.194	
5400 - 5500	5461	52.374	
5500 - 5700	----	3.908	
5700 - 5800	5780	61.407	
5800 - 7600	----	14.209	
Region	Units	H1500-A23	
Far Ultraviolet	Watts E-Vitons	188.994 7,827,109	
Middle Ultraviolet	Watts E-Vitons	156.916 4,684,472	
Near Ultraviolet	Watts E-Vitons	139.815 21,415	
Visible	Watts Lumens	226.800 61,000	

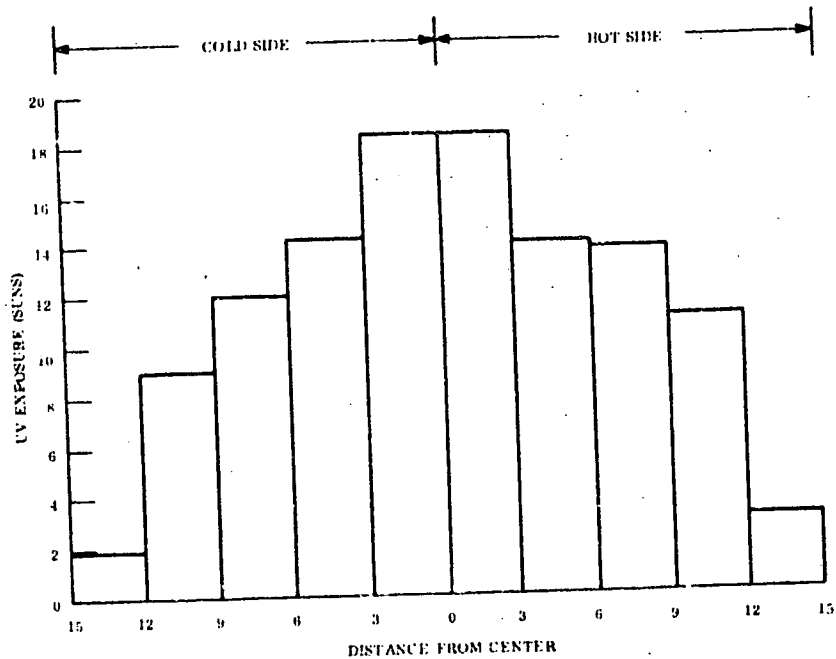


Figure 6-5. GE - H23KX Lamp, UV Exposure Versus Distance

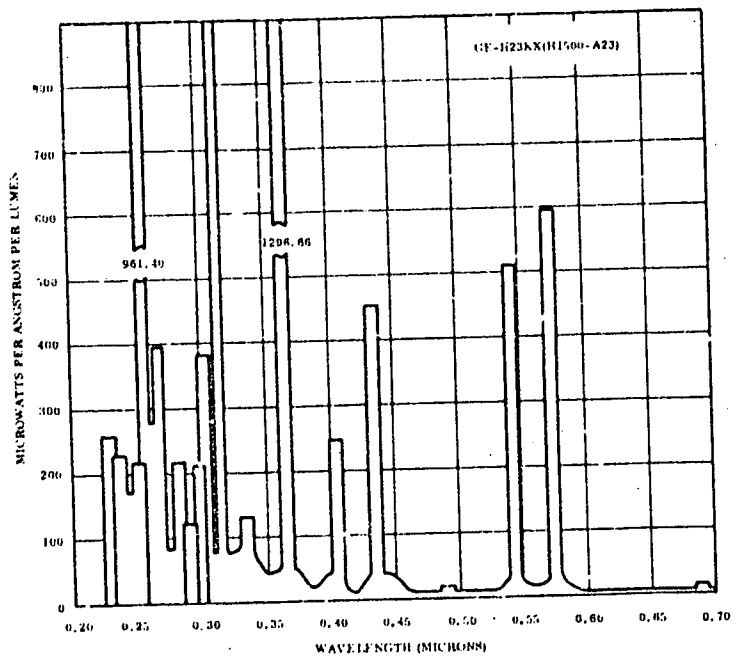


Figure 6-6. GE - H23KX Lamp, Spectral Distribution Data

materials were embrittled. In addition, these materials were all warped and several specimens fractured during the post-test bagging operation. The conditions of the hot side test resulted in catastrophic mechanical damage to these materials.

#### 6.4 VIBRATION TESTING

##### 6.4.1 TEST METHOD

All ten materials included in this study were subjected to a vibration range of 0 to 100 g's, 5 to 2000 Hz (sine only) in the temperature range -125 to 125°C. All specimens, except the Eccofoam material, were fabricated by casting a one-inch cube of material around a 1/4-20 hex-head bolt. The Eccofoam specimens were also one-inch cubes with the exception that a 5/8 inch hole was drilled through the center and the hex-head bolt placed through the hole. All ten specimen types were then attached to a tapped aluminum test fixture plate by means of the bolt. A total of three test plates were constructed. The tests were conducted in a specially constructed environmental chamber mounted on an MB C-150 vibration system.

##### 6.4.2 TEST PROCEDURE

The three test plates, each holding one specimen of each material, received the combinations of temperature-vibration shown in the test matrix of Table 6-4. The vibration scheme is shown in Figure 6-7.

The following notation is used in Table 6-4:

- |    |   |   |                 |
|----|---|---|-----------------|
| I  | - | Vibration Run No. 1 - all runs are at 2 oct/min |                 |
|    |   | 5 - 27 Hz                                       | 0.8 inches D.A. |
|    |   | 27 - 2000 Hz                                    | 30 g's          |
| II | - | Vibration Run No. 2:                            |                 |
|    |   | 5 - 28 Hz                                       | 0.8 inches D.A. |
|    |   | 28 - 58 Hz                                      | 70 l.p.s.       |

III - Vibration Run No. 3:

5	-	28 Hz	0.8 inches D.A.
28	-	88 Hz	70 l.p.s.
88	-	2000 Hz	100 g's

IV - Vibration Run No. 4:

A resonant frequency ( $f_c$ ) was determined by sweeping at 2 g's from 20 to 2000 Hz; then dwell at that frequency ( $f_c$ ) at 10 g's for 5 minutes.

Table 6-4. Combination of Temperature - Vibration to Which Specimens are Tested; Order of Testing Indicated by Numbers 1 Through 25

Test No.	Temp (°C)	Vib. Level 2 oct/ min	I		II		III		IV	
			1	2	3	4	5	6	7	8
A	Start	1	2	3	4	5				
	+25		a	a	a	a				
B		6	7	8	9					
	-25		a	a	a					
B		10	11	12	13					
	-75		b	b	b					
C		14	15	16	17					
	-125		b	b	b					
C		18	19	20	21					
	+75		c	c	c					
C	Finish	22	23	24	25					
	+125		c	c	c					

\* NOTE:

- A - Test No. 1: for Plate a
- B - Test No. 2: for Plate b
- C - Test No. 3: for Plate c

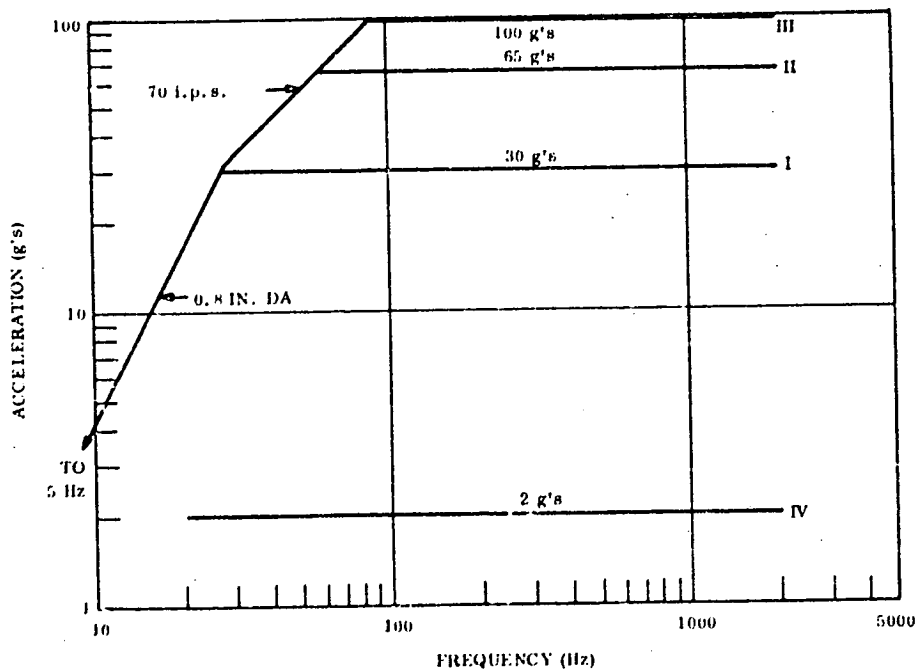


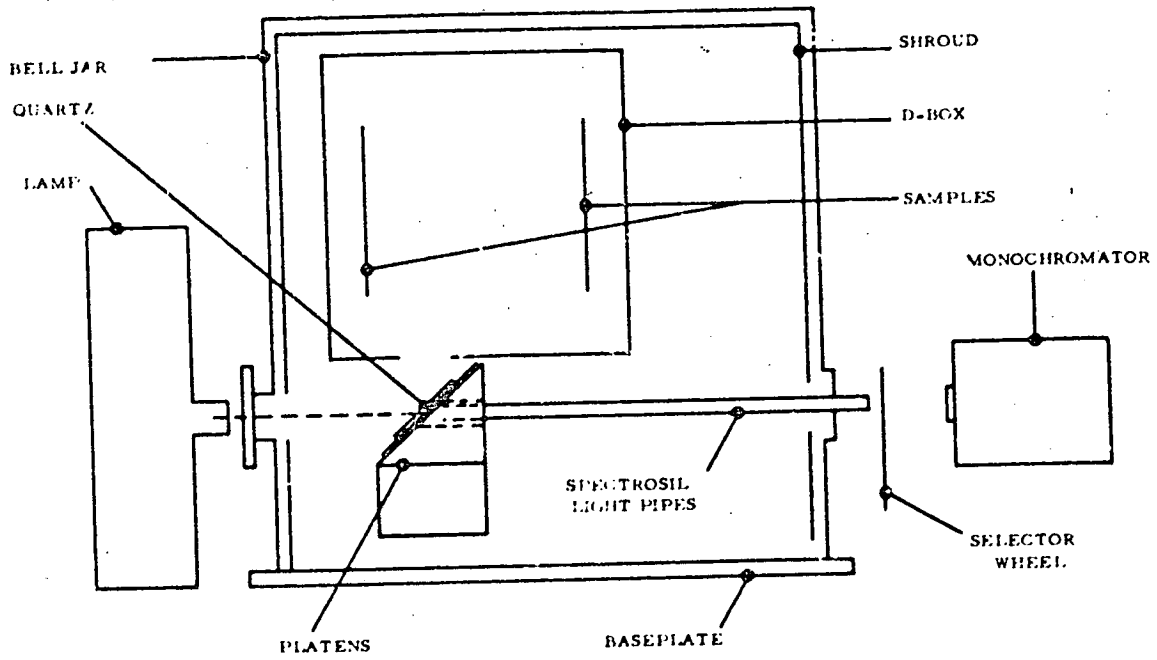
Figure 6-7. Acceleration as a Function of Frequency for Vibration Runs I, II, III and IV

## SECTION 7 LIGHT TRANSMISSION TESTS

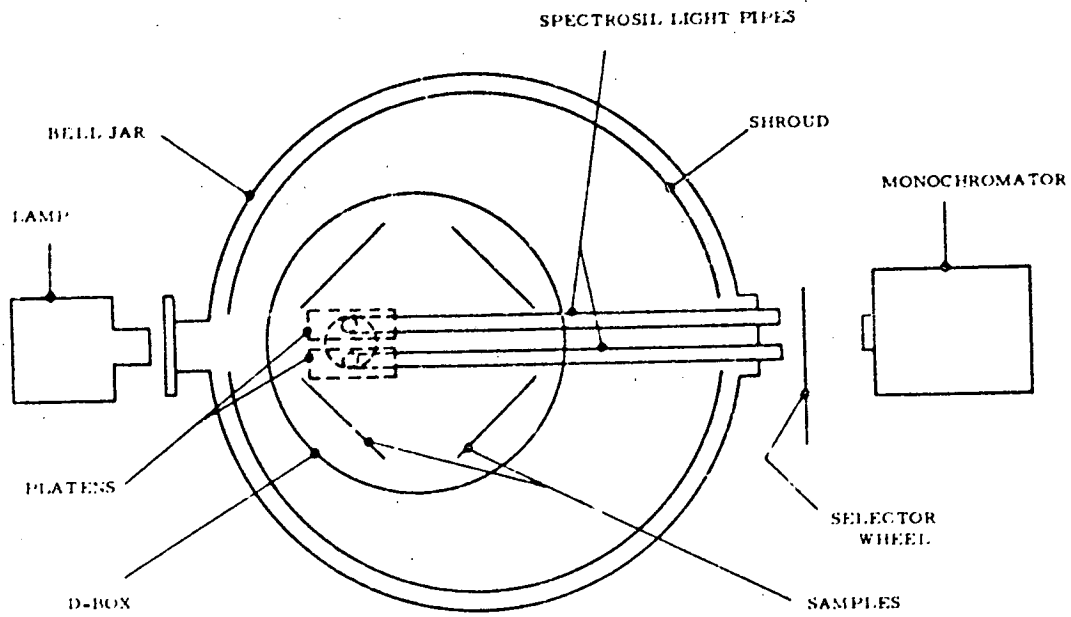
### 7.1 EXPERIMENTAL TECHNIQUES

The purpose of these tests was to establish the change in light transmission that would result from the combined effects of UV radiation and outgassing from selected materials. The test configuration used to establish this is shown in Figures 7-1a and b. Four samples, nominally 4-inch squares about 0.050-inch thick, plus one 2-inch by 4-inch by 0.050-inch sample were placed in the D-box. Sample temperature was maintained with the infrared lamps in the double-walled D-box shown in Figures 7-2a and b. These lamps were controlled with an on/off controller sensing a thermocouple attached to the 2- by 4-inch sample. The sample temperature was held constant at a nominal 120°C.

There are two platens located directly beneath the only hole in the D-box, as shown in Figures 7-3a and b. These platens are inclined at 45 degrees to the bottom of the D-box and at 45 degrees to the incident UV irradiation. The quartz slides on which the outgassed particles were incident were bonded to an aluminum frame which was fastened to these platens. Platen temperatures were individually controlled with an on/off controller regulating the heat flow from the platens to a liquid nitrogen heat sink. The controllers sensed thermocouples imbedded in the platens and correlated to the surface temperature of the quartz slides in a calibration test with the UV irradiation incident on the quartz slides. One of the platens has a movable flap that can cover it or be removed from outside the vacuum chamber. By covering one platen for the first six hours at temperature, the effect of vacuum treating the material was established simultaneously with the untreated material. This is obtained by noting the change in light transmission of each platen. Total test duration is 102 hours; therefore, platen No. 1 (which is never covered) shows the change in light transmission caused by the virgin material for 102 hours, while platen No. 2 (which was covered the first six hours the sample was at temperature) shows the change in transmission caused by the material during 96 hours.

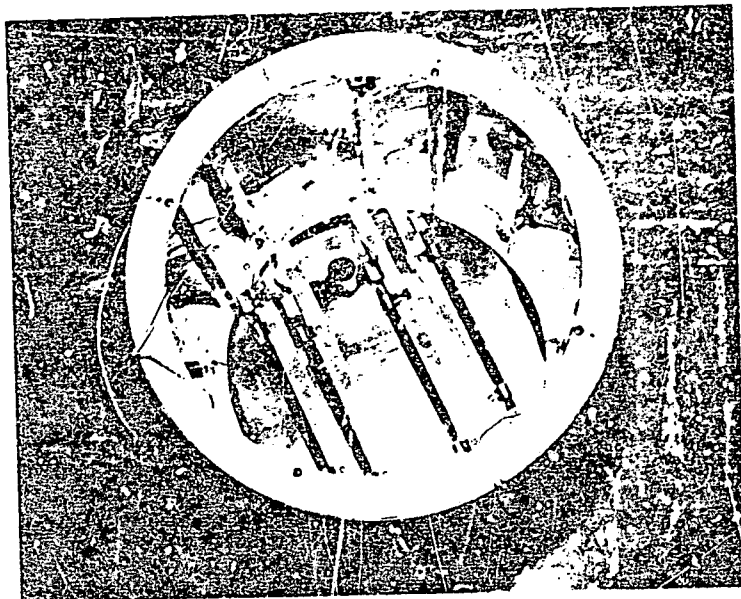


a. Side View

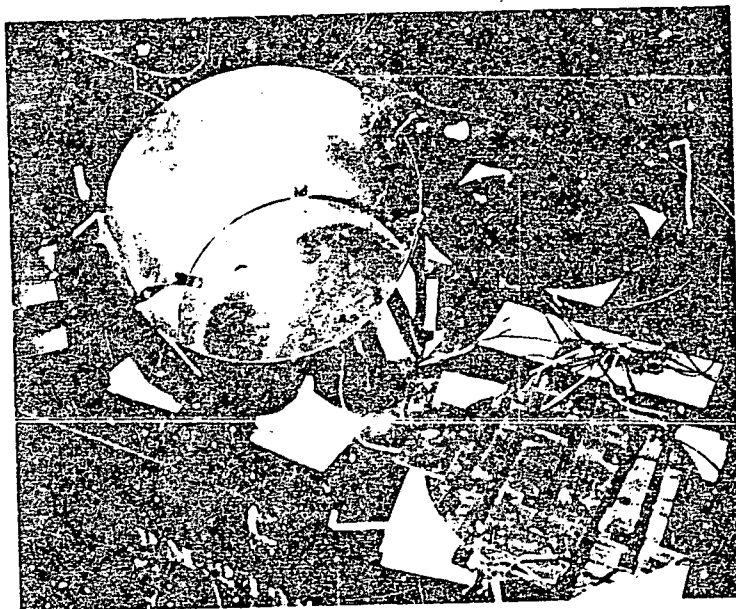


b. Top View

Figure 7-1. Experimental Configuration

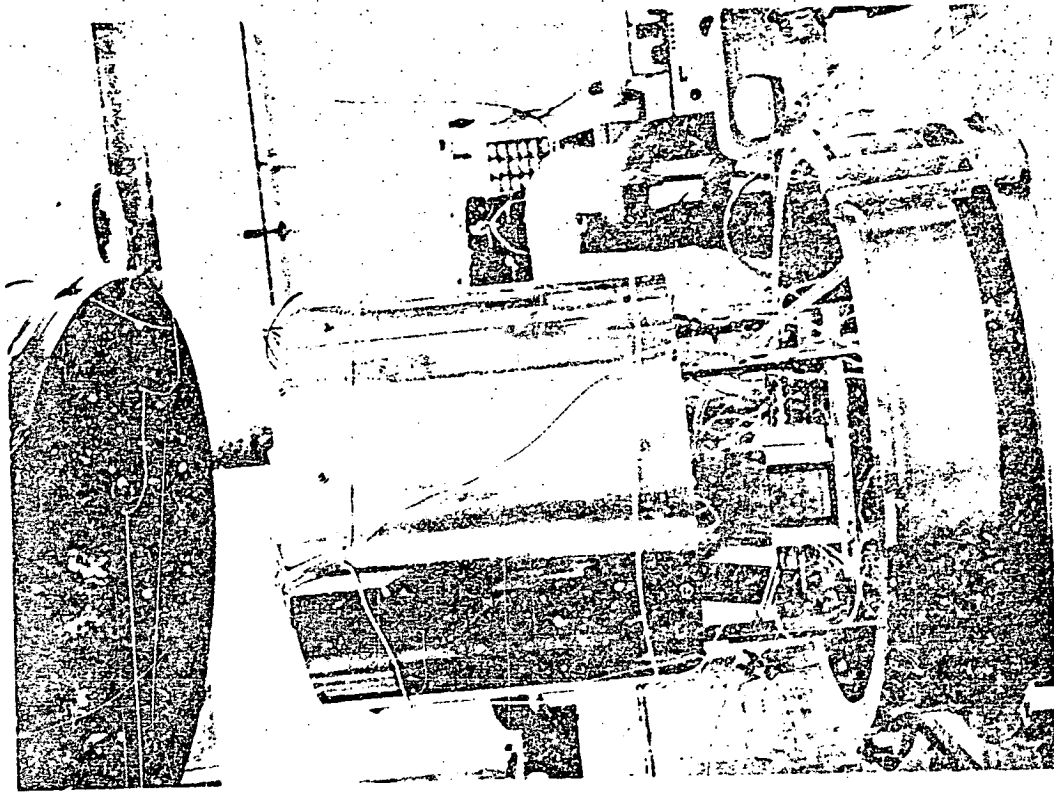


a.

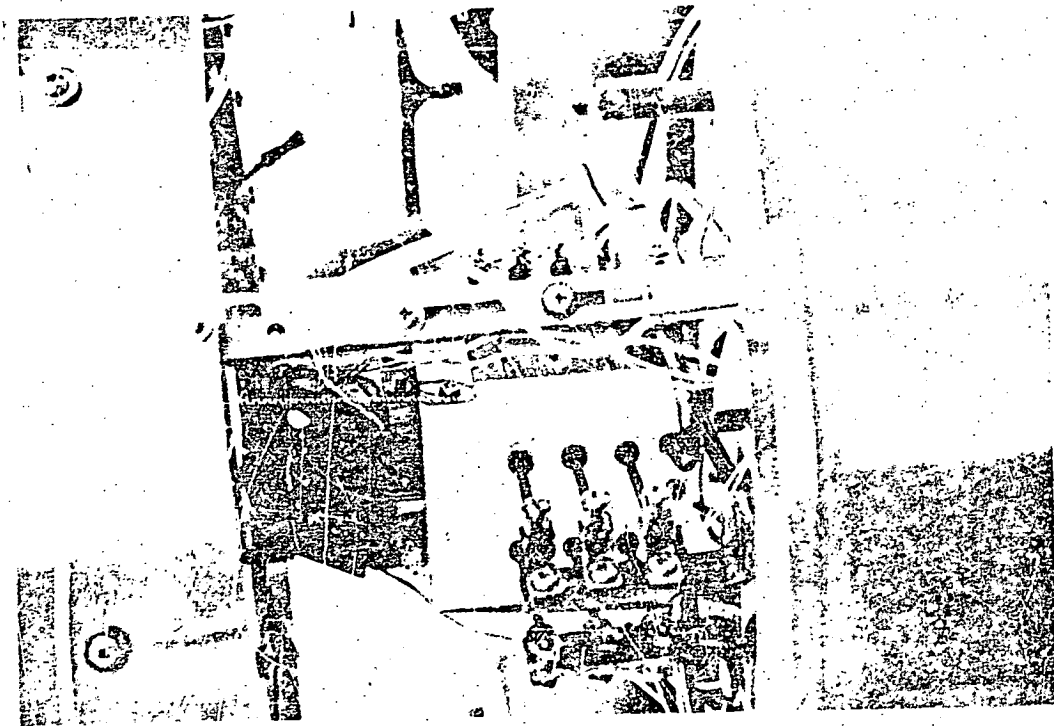


b.

Figure 7-2. Double-Walled D-Box



b.



a.

Figure 7-3. D-Box Showing Platen Locations

The average UV irradiance on the quartz slides was 5 EUVS\*. The UV radiation source used was a 2.5 kev HgXe short arc lamp, whose typical spectral distribution at the sample plane is as shown in Figure 7-4. The UV irradiation entered the vacuum chamber through a sapphire window. The UV irradiation source was used simultaneously for the light source of the quartz slide transmission measurements.

Transmission measurements were performed before, during, and after the test. The measurements performed during the test were at the single wavelength, 0.35 microns, while the before and after measurements were performed from 0.3 to 4.2 microns. All transmission measurements performed were relative change, plotted as percent decrease from the original value. These measurements were performed by comparing the light transmitted by the quartz slide to the light emitted by the source. The light was directed from the source and the quartz slides by light pipes which were put through a selector wheel and into a Perkin-Elmer Model 99 Monochromator. The measurements from 2 to 4.25 microns were performed on a Perkin-Elmer Model 457 Infrared Spectrophotometer.

Figure 7-3b shows the reference light pipe in the background and the quartz slide light pipes in the central region of the photograph. Figures 7-5a and b show the details of the selector wheel and the monochromator with its input optics.

Comparative measurements at 0.35 microns were performed automatically during the test at time intervals of 15 to 30 minutes. The selector wheel in Figures 7-5a and b automatically cycle at pre-established intervals. The holes in this selector wheel allow the zero (background reading), the transmission through the quartz slide on platen No. 2, the lamp output, and the transmission through the quartz slide on platen No. 1 to be measured sequentially within 3 minutes of each other. This sequence is repeated for each measurement.

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\*EUVS is the equivalent ultraviolet runs below 0.4 microns. An intensity of one EUVS is when the integrated irradiance below 0.4 microns incident on the sample plane is the same as the integrated irradiance of the sun below 0.4 microns outside the earth's atmosphere and at a distance equal to the radius of the mean earth orbit about the sun.

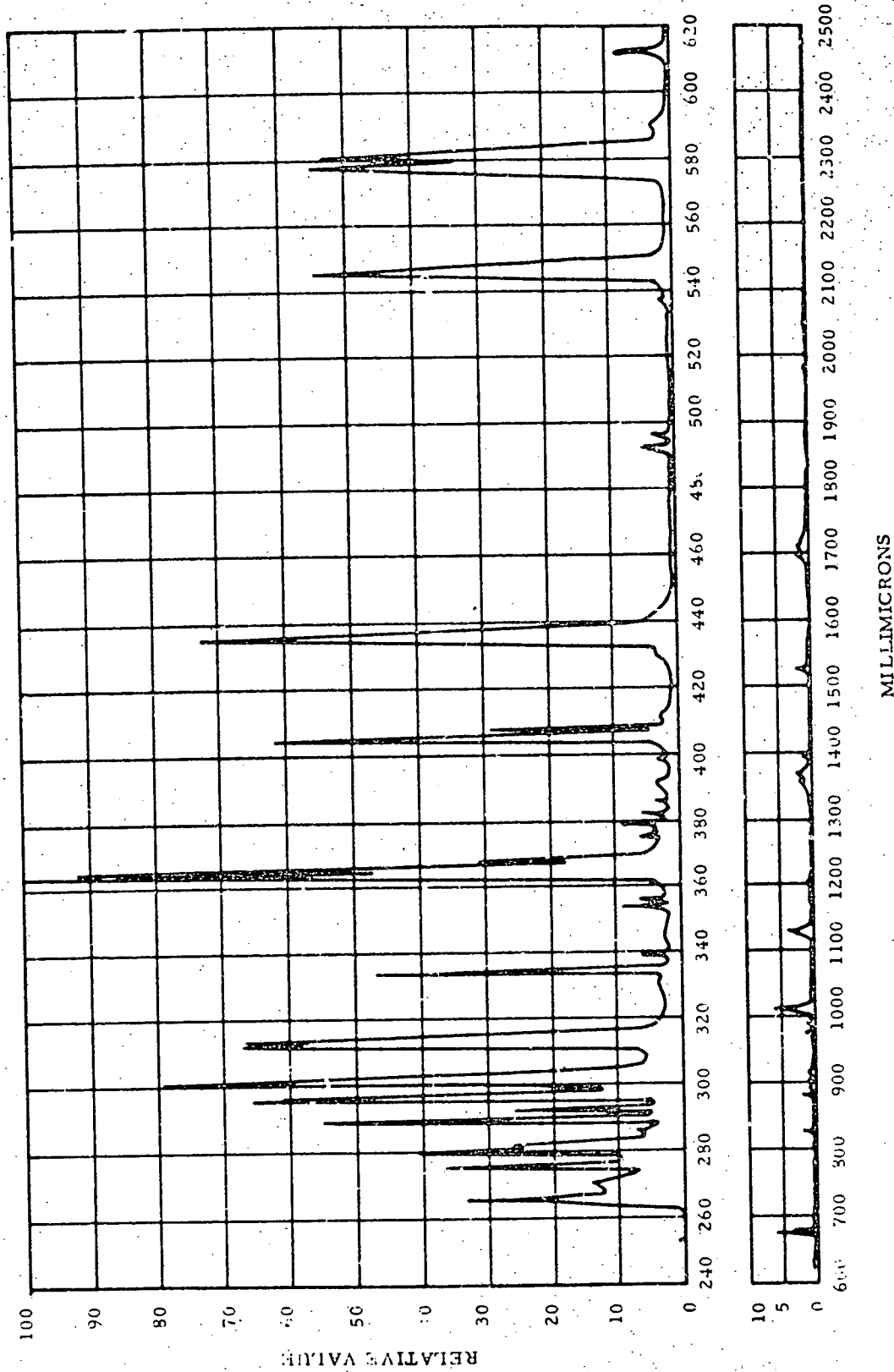
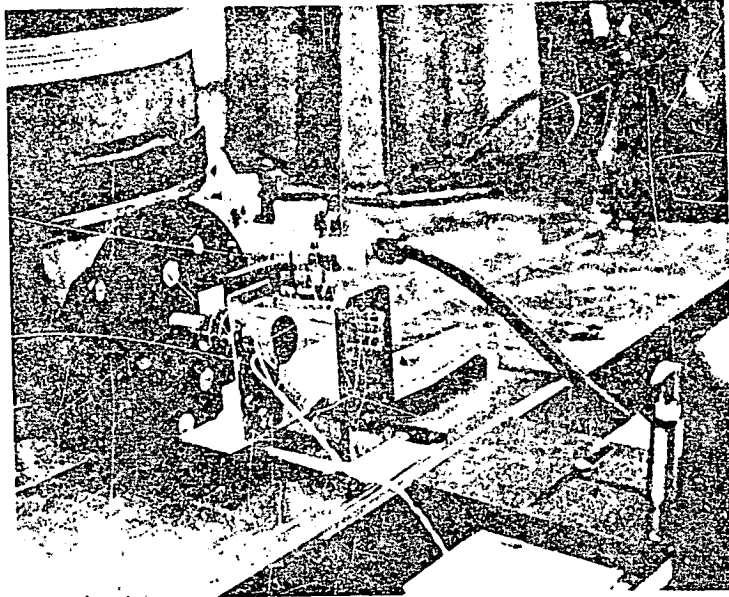
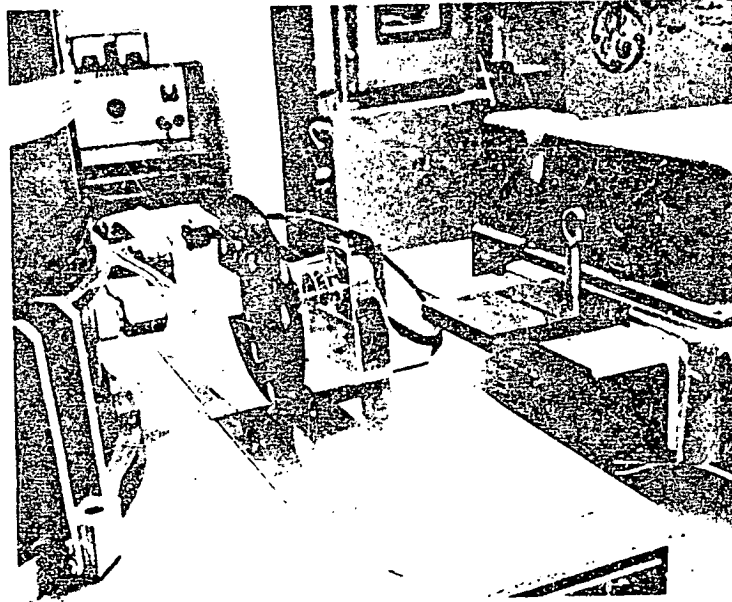


Figure 7-4. HgXe Lamp Spectral Distribution



a.



b.

Figure 7-5. Selector Wheel and Monochromator with Input Optics

The relative transmission of the quartz slide on platen i,  $T_{i \text{ rel}}$ , is then computed as

$$T_{i \text{ rel}} = \frac{V_i - V_z}{V_R - V_z} \quad (7-1)$$

where

$V_i$  is the monochromator signal output when the light transmitted through the quartz slide on platen i is incident on it,

$V_z$  is the zero or background signal from the monochromator, and

$V_R$  is the monochromator signal when the light from the UV source is incident on its detector.

The percent decrease in transmission, as a function of time,  $T_i(t)$ , or at the beginning and end of the test, is then computed as

$$T_i(t) = \frac{T_{i \text{ rel}}^{(0)} - T_{i \text{ rel}}^{(t)}}{T_{i \text{ rel}}^{(0)}} \times 100 \quad (7-2)$$

where

$T_i(t)$  is the percent decrease in transmission at the quartz slide on platen i at time t.

$T_{i \text{ rel}}^{(0)}$  and  $T_{i \text{ rel}}^{(t)}$  is the computation of Equation 7-1 at the beginning of the test (time,  $t = 0$ ) and at time ( $t = t$ ), respectively.

The factor of 100 is to put this in the percent change in transmission. Although this has been described for the measurements made in situ at 0.35 microns as a function of time during the test, it also applies for measurements made as a function of wavelength before and after the test.

In addition to the change in light transmission, the bulk weight loss of the samples exposed to 120°C for 102 hours in vacuum was also established. \* Bulk weight loss is the total weight loss of the samples less that which is moisture and gases. Bulk weight loss, therefore, represents the maximum amount of condensables outgassed from the material. This value is computed from the before test and after test stabilized weights of the samples in a constant relative humidity and temperature environment.

## 7.2 EXPERIMENTAL PROCEDURES

The samples were prepared according to the applicable Marshall specifications and pre-conditioned in a 46 percent relative humidity, 21°C environment. The five samples described earlier were held in the D-box with stainless steel wire. The D-box was then placed so that the hole in its bottom was centered over the two platens. Transmission measurements were made on the quartz slides from 0.30 to 4.25 microns before and after being screwed to the copper platens. The transmission measurements at 0.35 microns made with the selector wheel system were repeated at 15 minute intervals both before evacuation of the system and during the first 30 hours of test. At approximately 30 hours, the interval was changed to 30 minutes. The UV radiation source was stabilized before transmission measurements were begun and remained on until the end of the test. The flap on platen No. 2 was closed before the chamber was evacuated.

In order to prevent contamination from backstreaming of diffusion pump fluid, the following technique was used. While the chamber was being evacuated to about 50 microns, the high vacuum gate valve between the diffusion pump and the chamber remained closed and the anti-migration chevron liquid nitrogen trap between the diffusion pump and the gate valve was cooled (the diffusion pump always remained on). After the chamber was at 50 microns, the gate valve was opened. At shutdown, the gate valve was closed before the liquid nitrogen trap was allowed to warm up. Using this technique the worst periods for backstreaming (start-up and shutdown) were completely avoided. Figure 7-6 shows the environmental facility used in this test.

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\* It should be noted that this was not contractually required.

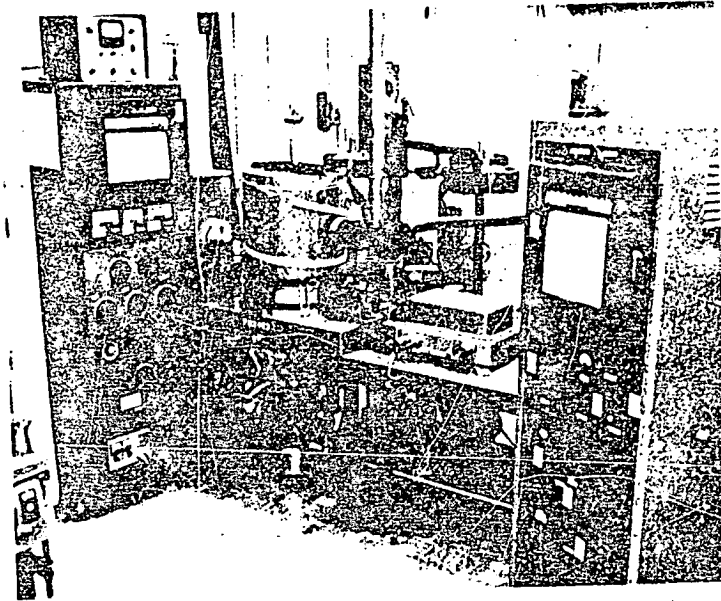


Figure 7-6. Environmental Facility used in Light Transmission Tests

After the chamber was in the low  $10^{-6}$  Torr range, the quartz slide on platen No. 1 was cooled to  $0^{\circ}\text{C}$ . During this time, platen No. 2 was maintained at about  $30^{\circ}\text{C}$ . Heat was applied to the D-box after the platens had stabilized thermally. The test time was considered to begin when the samples in the D-box reached  $120^{\circ}\text{C}$ . After 6 hours under these conditions, the quartz slide on platen No. 2 was brought to  $0^{\circ}\text{C}$  and the flap was opened. The environmental parameters were then maintained constant for an additional 96 hours. At this time, transmission measurements were made as a function of wavelength and the test was shut down. The shutdown procedure was designed to eliminate all mass transfer between the various surfaces in the system. This was accomplished by using the following technique:

1. First:
  - a. UV radiation source was shut down.
  - b. High vacuum gate valve was closed.
  - c. Heat to the D-box was shut off.
  - d. Liquid nitrogen to the chamber shroud was shut off.

2. After the shroud warmed up to  $-112^{\circ}\text{C}$ :
  - a. Chamber was backfilled to 1000 microns with dry nitrogen.
  - b. Quartz slides was heated to  $20^{\circ}\text{C}$ .
3. The chamber was brought to atmospheric pressure with dry nitrogen when the following conditions were obtained:
  - a. Shroud and platens were at  $20 \pm 5^{\circ}\text{C}$ .
  - b. D-box was  $80^{\circ}\text{C}$  or lower.

The quartz slides were then removed and weighed immediately. \* Transmission measurements were made from 0.30 to 4.25 microns as soon as possible. The samples were removed from the D-box and put into the 46 percent RH,  $24^{\circ}\text{C}$  environment for post-conditioning. They were weighed periodically in this environment until their weight stabilized.

In addition to actual sample tests, both full and abbreviated control tests were performed. A full control test is identical to a sample test except that no samples are placed in the D-box and the flap on platen No. 2 remains open. An abbreviated control test is the same as a full control test except its duration is shorter, usually from 24 to 48 hours.

The entire system was cleaned with appropriate solvents after all tests. In addition, thermal vacuum bake-outs of the system were performed as necessary. Full control tests were conducted at the beginning and end of the program with abbreviated control tests performed after any significant condensates were formed.

### 7.3 RESULTS

The results of the light transmission test are presented in three parts:

1. Figure 7-Xa is a composite plot of the platen temperatures, sample temperature, and chamber pressure, all as a function of time for the entire test sequence.

---

\* The quartz slides were also weighed immediately before test.

2. Figure 7-Xb is a plot of the relative percent transmission at 0.35 microns as a function of the time during the test.\*
3. Figure 7-Xc is a plot of the percent decrease in transmission as a function of wavelength. These values are based on transmission measurements made before and after environmental exposure.

Table 7-1 lists the specific figure number for each test sequence, and Table 7-2 gives the bulk weight loss of the materials resulting from environmental exposure.

Table 7-1. Index of Light Transmission Test Results

Test Sequence	Figure Number
EC-2850	7-7a, b, c
EC-1090	7-8a, b, c
EC-2651	7-9a, b, c
Eccofoam FPH	7-10a, b, c
PC 22	7-11a, b, c
PF-1535	7-12a, b, c
PR-1538	7-13a, b, c
Pro-Seal 794	7-14a, b, c
SYLGARD 182	7-15a, b, c
RTV-602	7-16a, b, c
Full Control Test	7-17a, b, c

\*Relative percent transmission is normalized to 100 percent at the beginning of the test.

Table 7-2. Bulk Weight Loss \*

Material	Weight Loss (%)
EC-2850	0.00
EC-1090	0.00
EC-2651	0.00
Eccofoam FPH	0.56
PC 22	0.81
PR-1535	1.1
PR-1538	0.41
Pro-Seal 794	0.66
SYLGARD 182	1.4
RTV-602	1.8

\*Loss due to environmental exposure of 102 hours at 120°C at a pressure in the low  $10^{-6}$  Torr range.

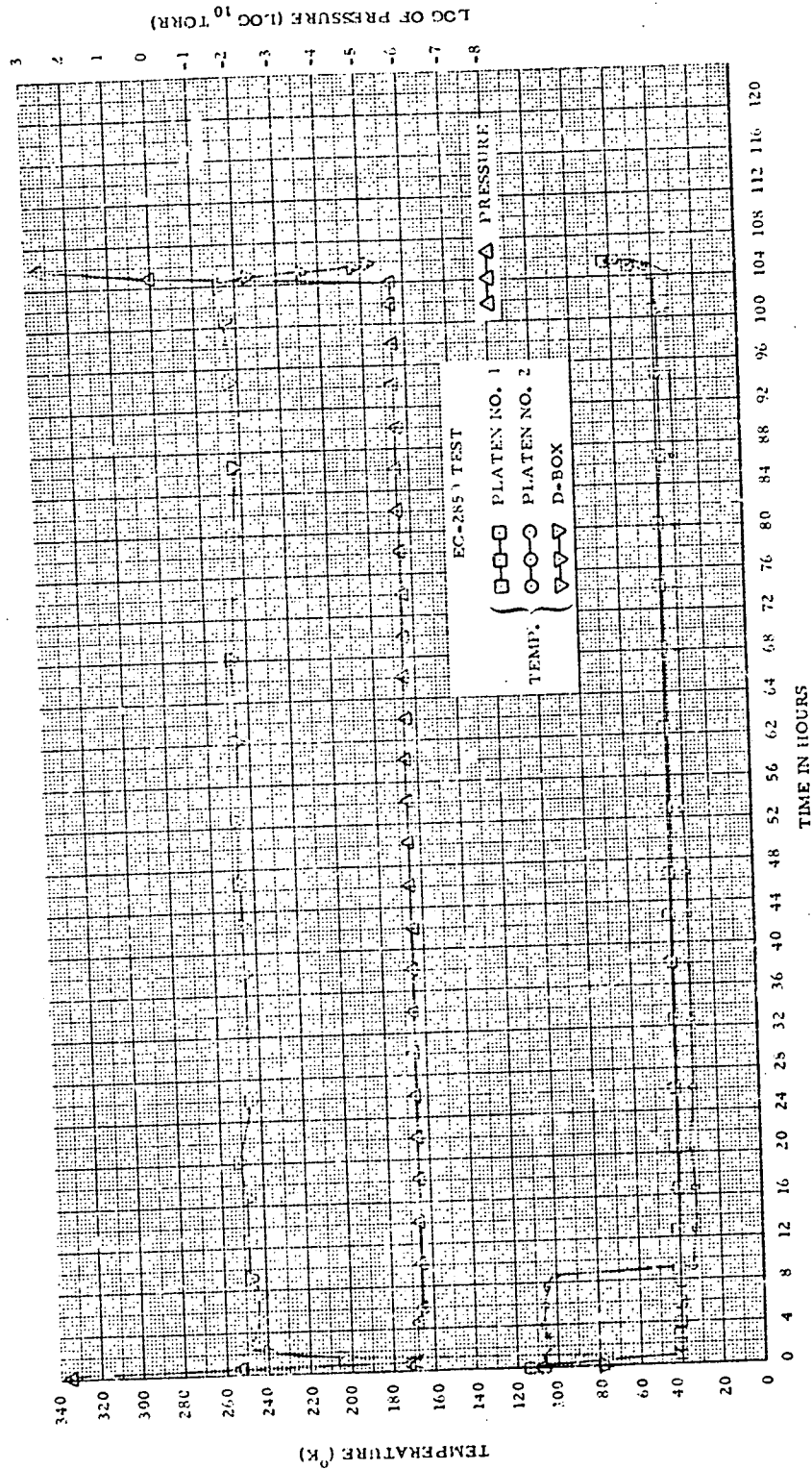


Figure 7-7a. EC-2850 Test (Sheet 1 of 3)

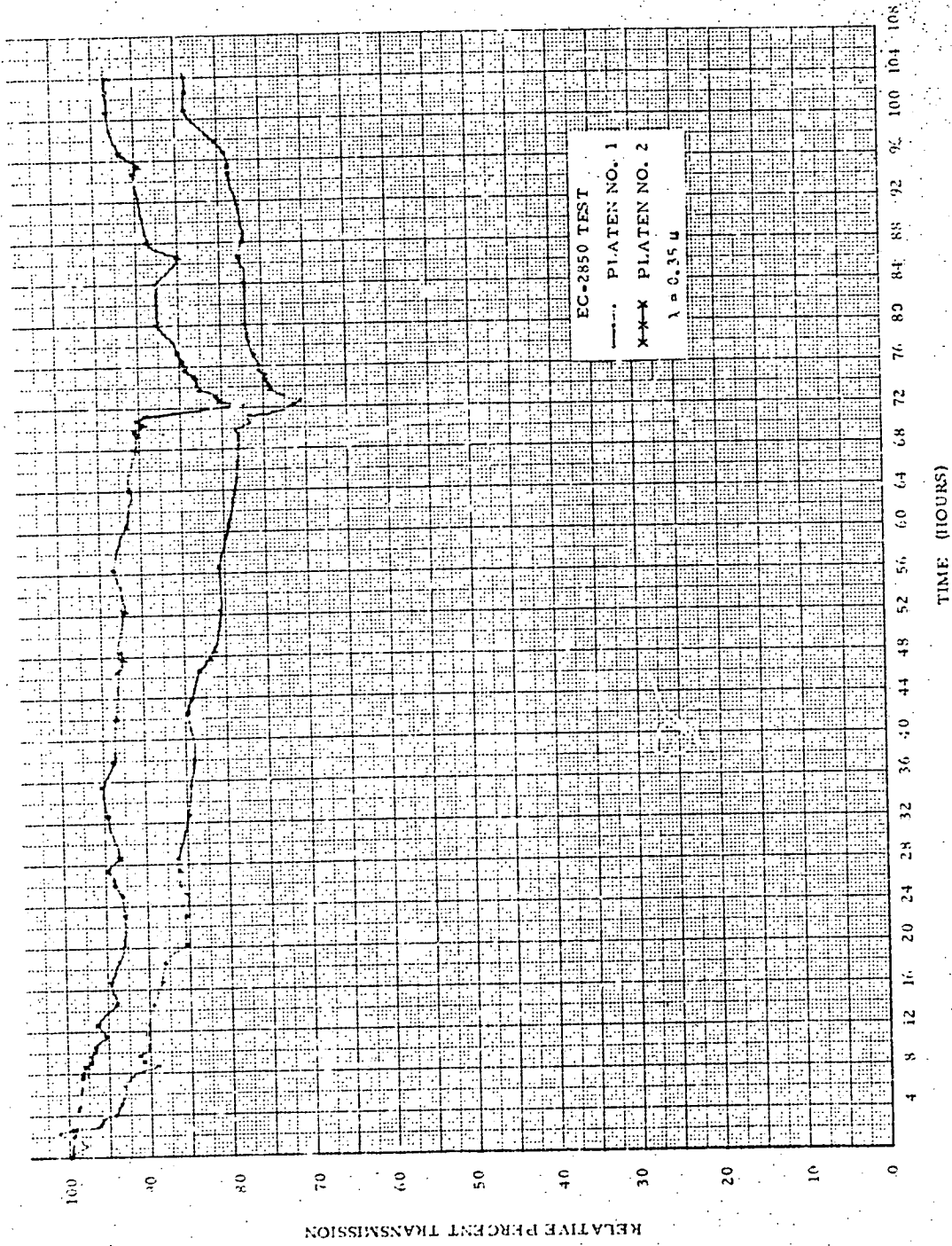


Figure 7-7b. EC-2850 Test (Sheet 2 of 3)

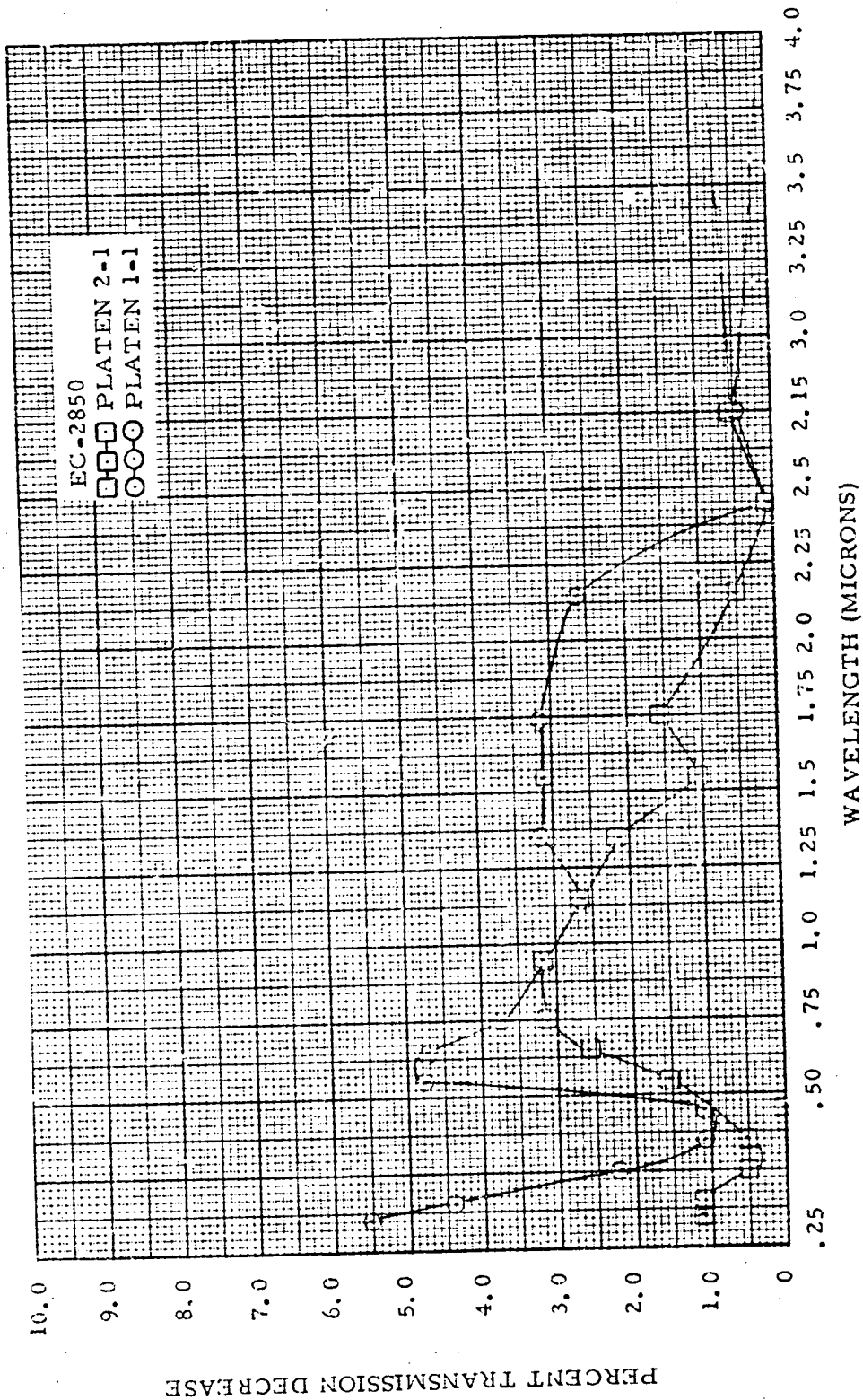


Figure 7-7c. EC-2850 Test (Sheet 3 of 3)

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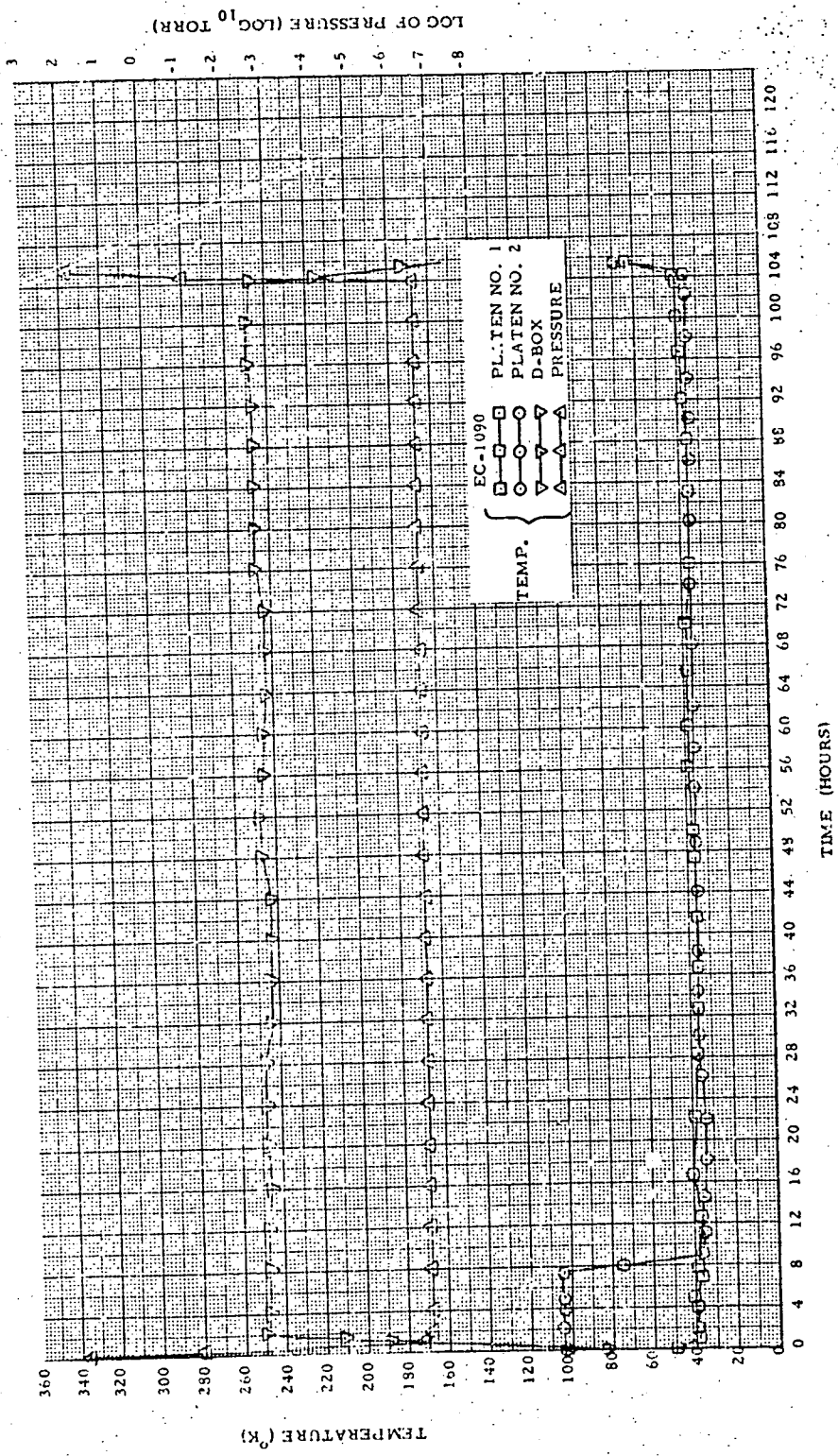


Figure 7-8a. EC-1090 Test (Sheet 1 of 3)

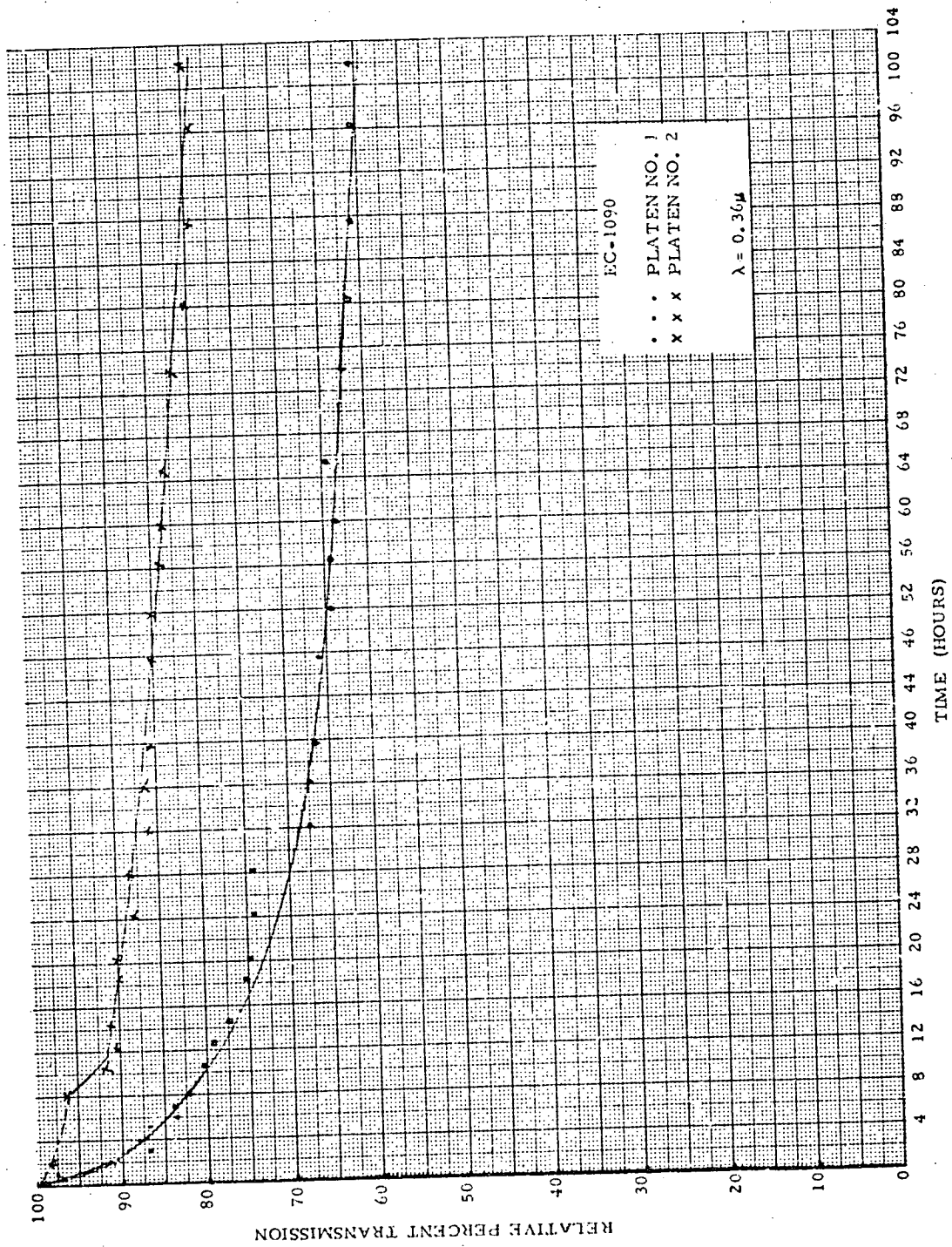


Figure 7-8b. EC-1090 Test (Sheet 2 of 3)

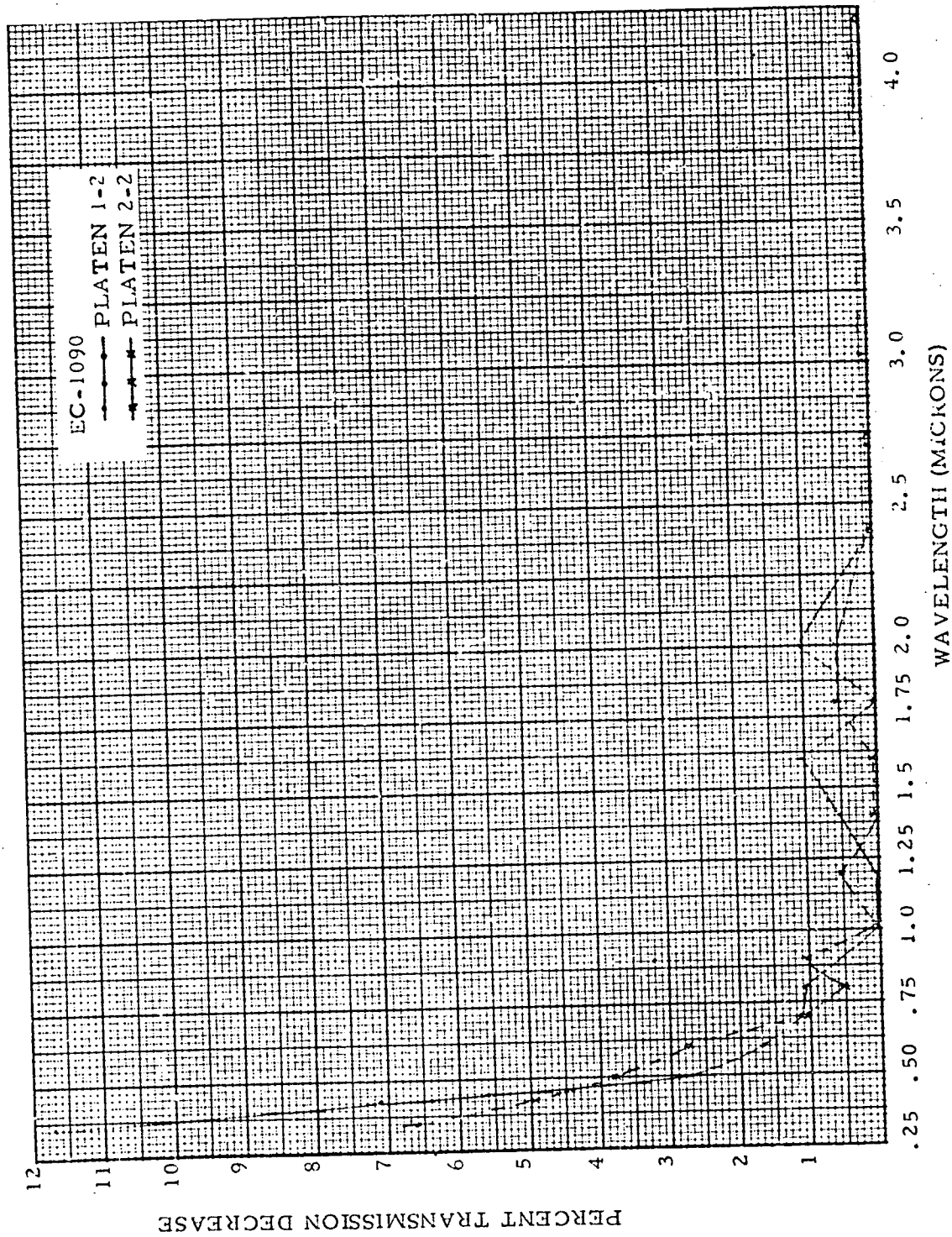


Figure 7-8c. EX-1090 Test (Sheet 3 of 3)

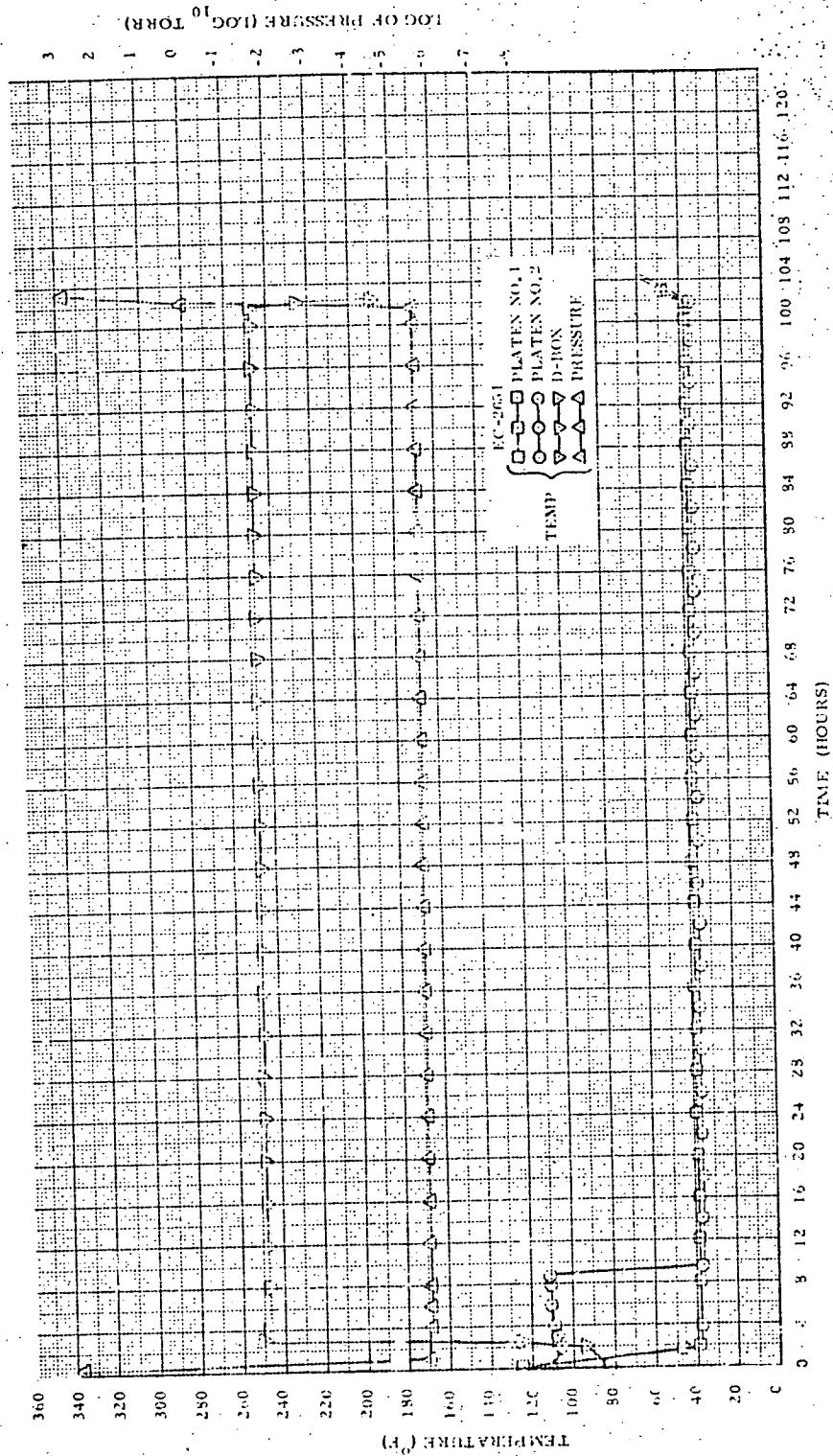


Figure 7-9a. EC-2651 Test (Sheet 1 of 3)

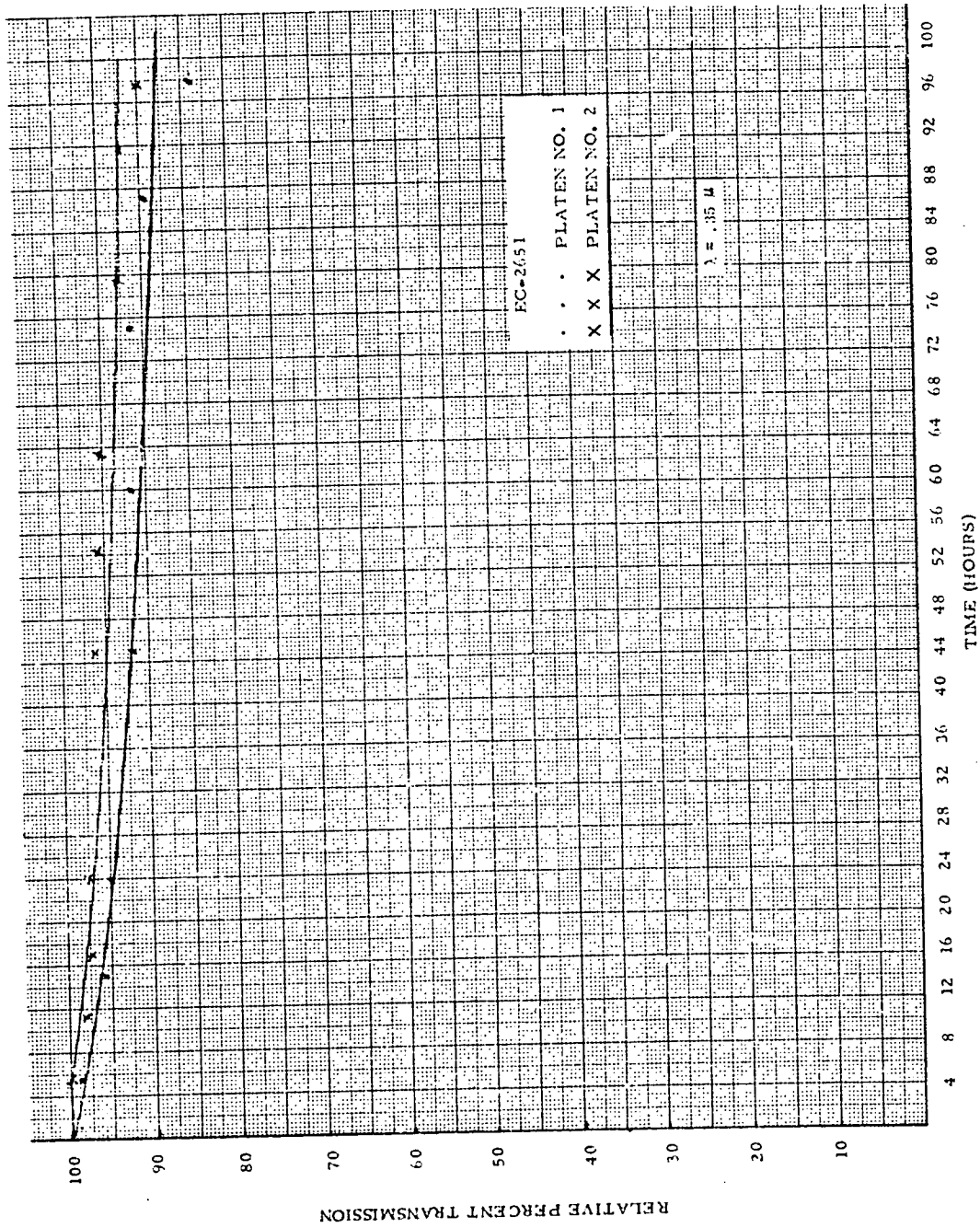


Figure 7-9b. EC-2651 Test (Sheet 2 of 3)

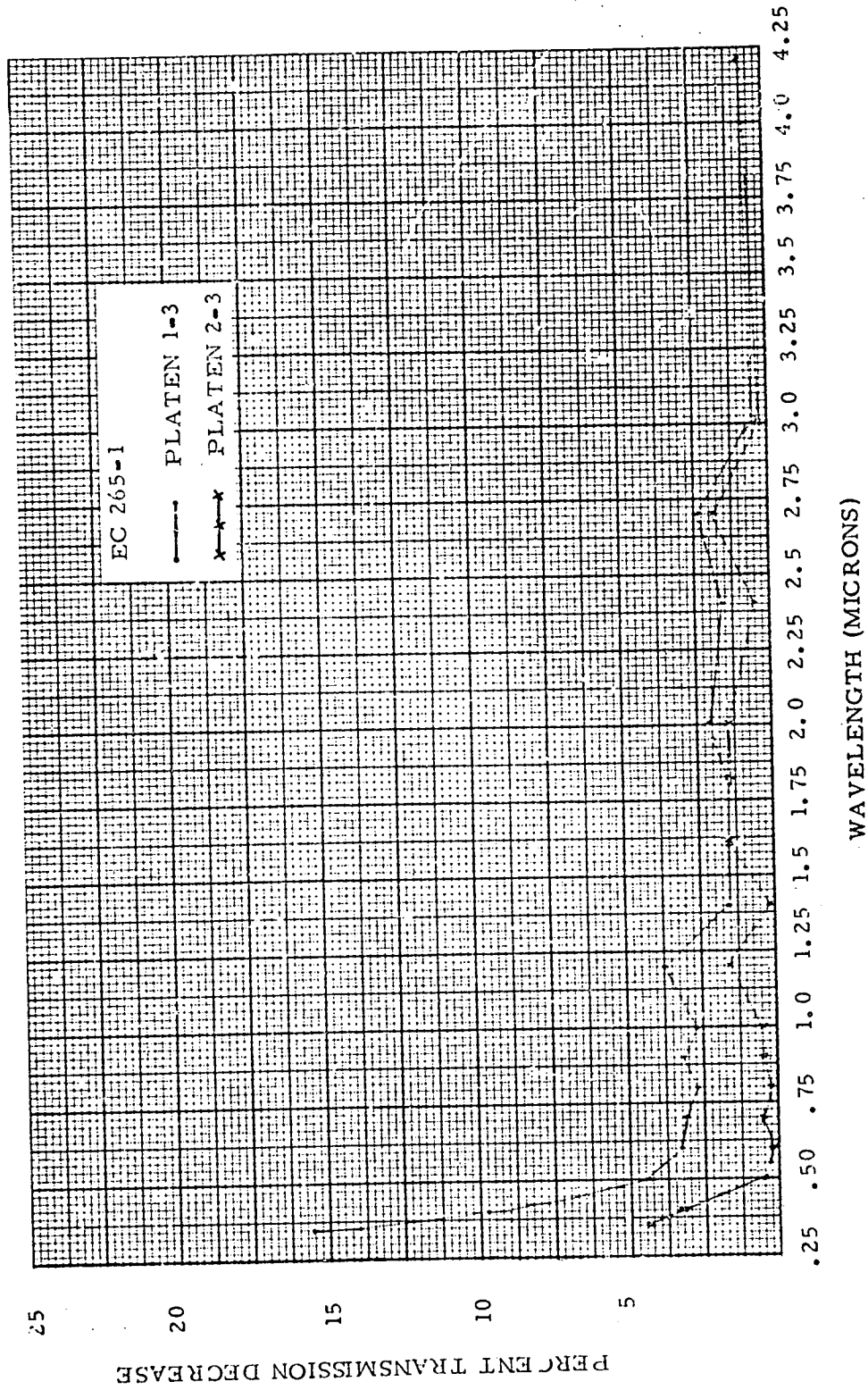


Figure 7-9c. EC-2651 Test (Sheet 3 of 3)

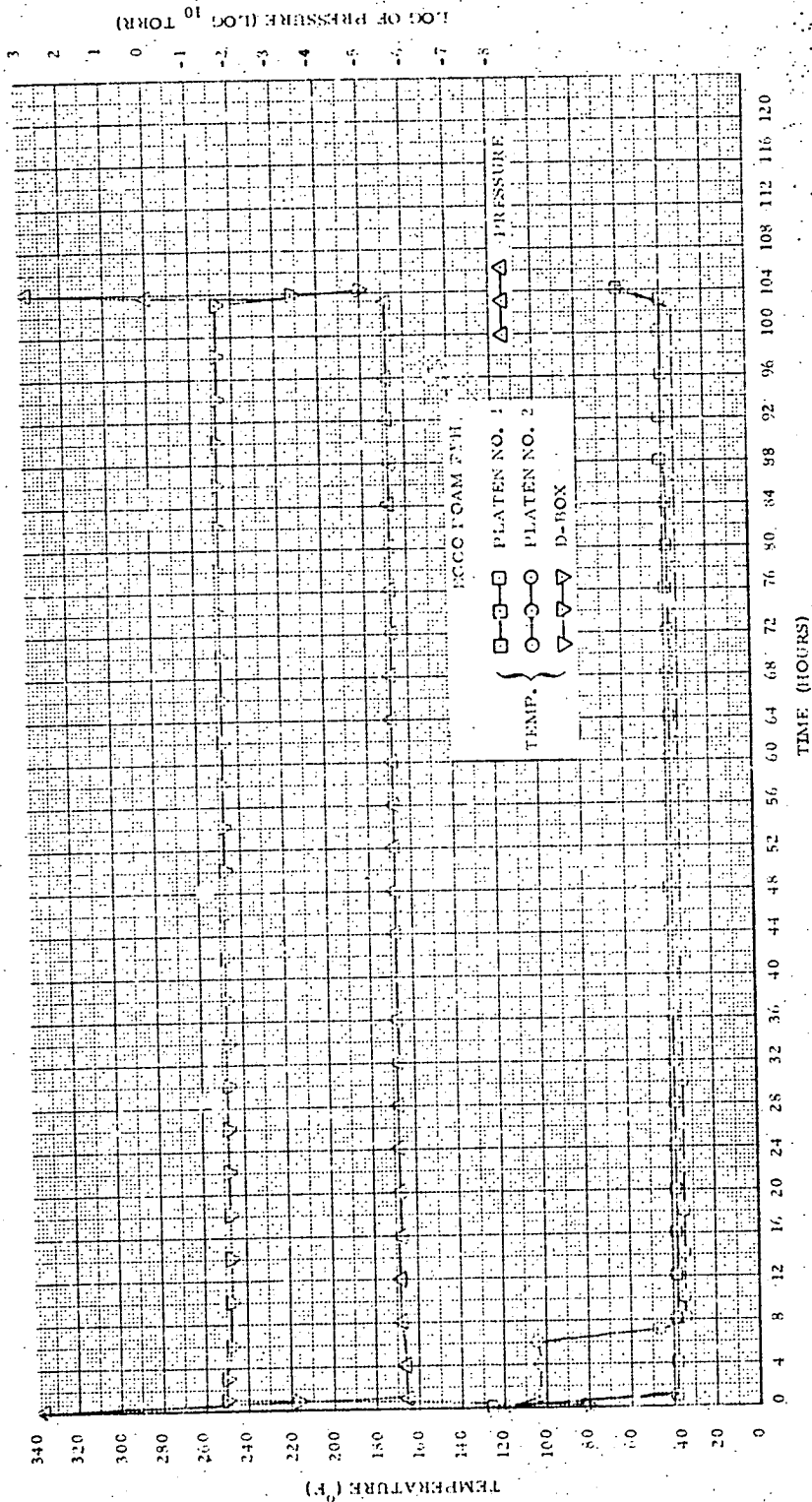


Figure 7-10a. Eccof foam FPH Test (Sheet 1 of 3)

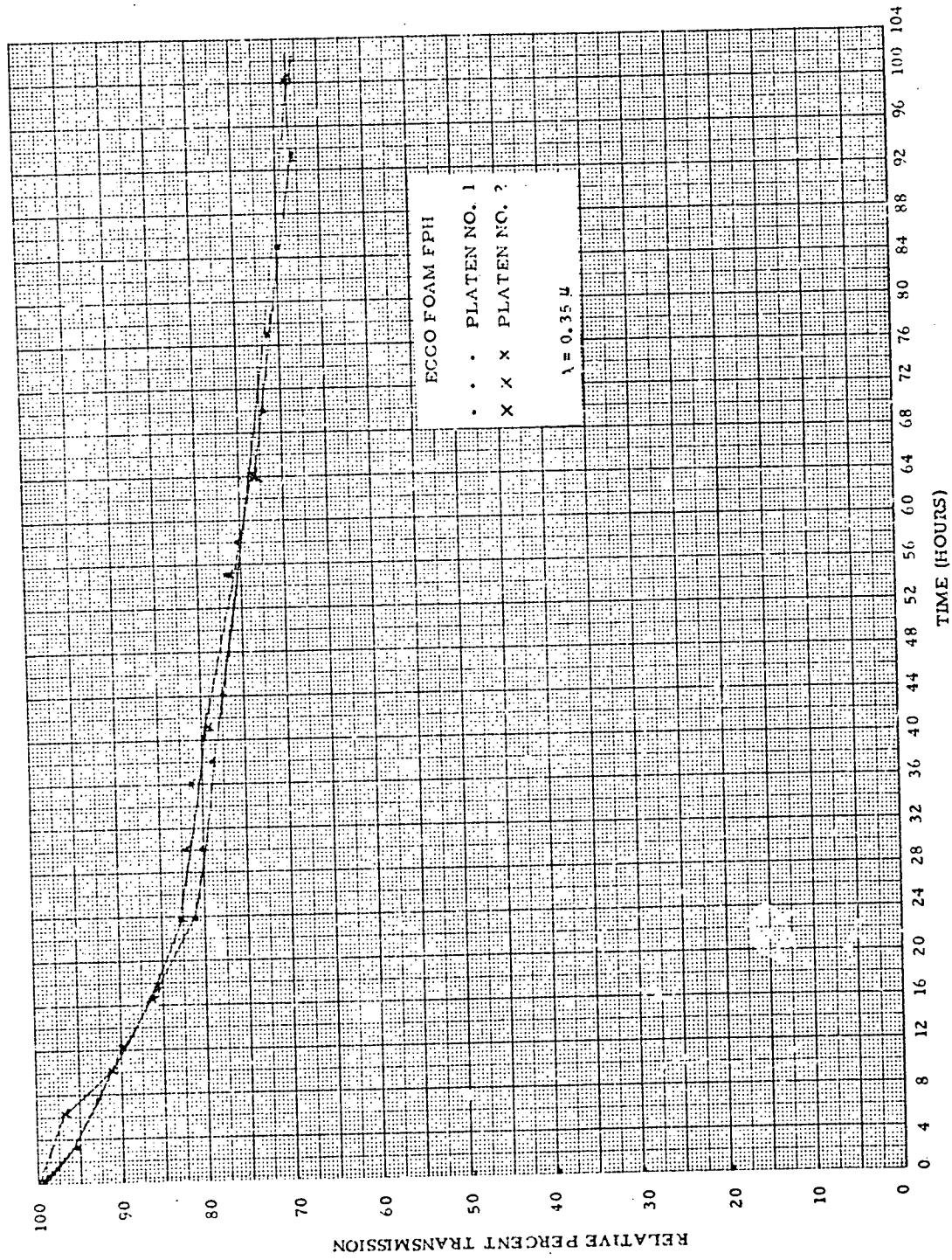


Figure 7-10b. Eccofoam FPH Test (Sheet 2 of 3)

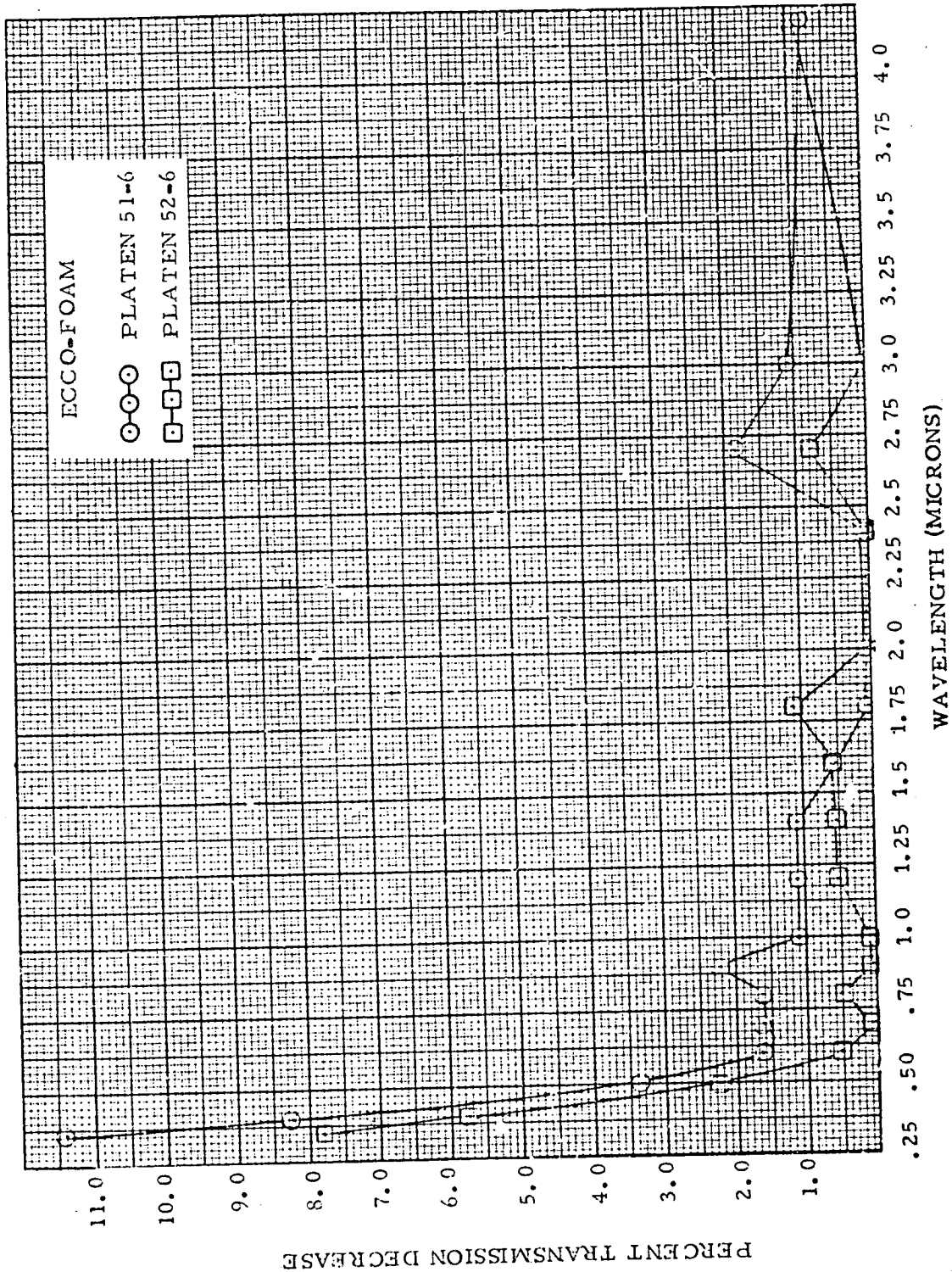


Figure 7-10c. Eccof-foam FPH Test (Sheet 3 of 3)

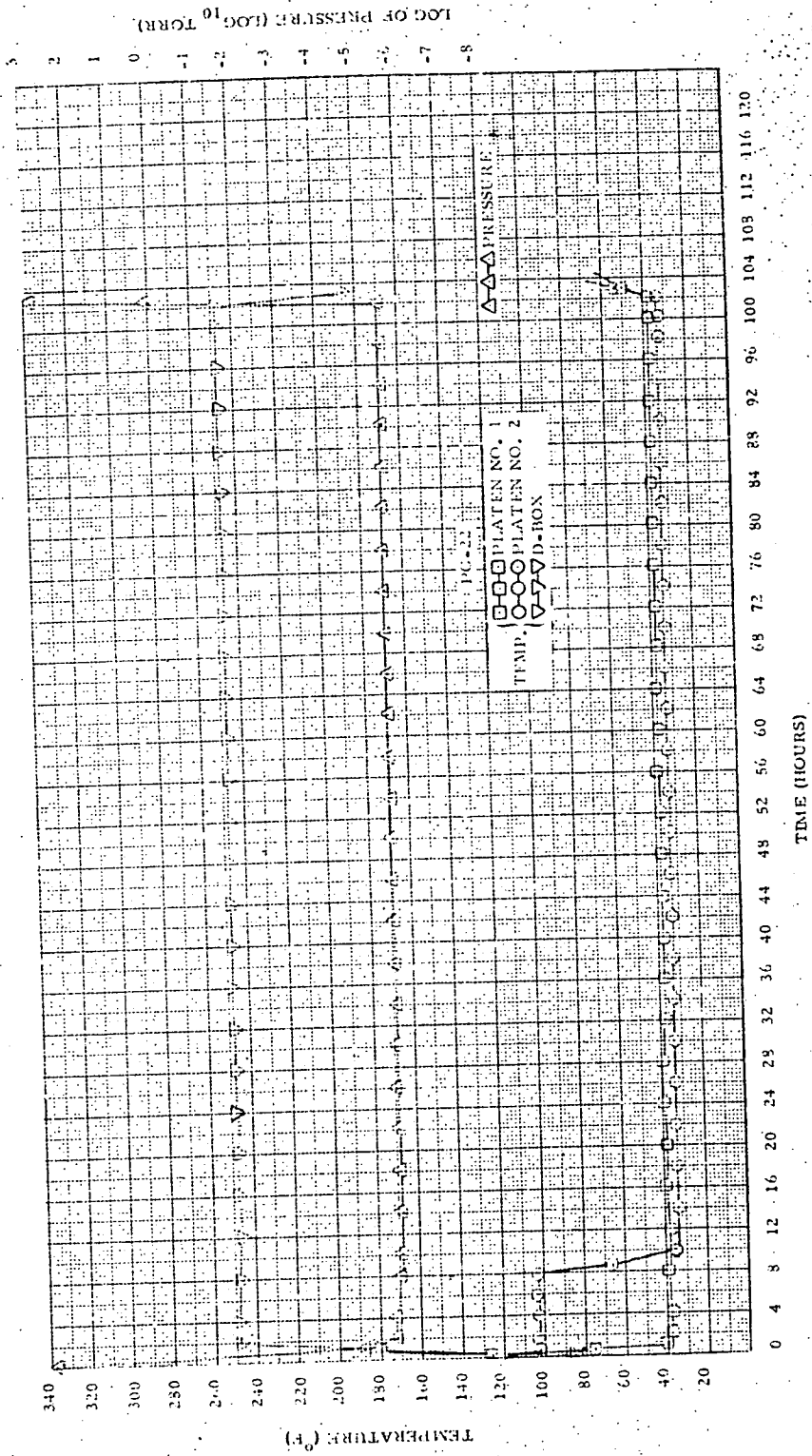


Figure 7-11a. PC 22 Test (Sheet 1 of 3)

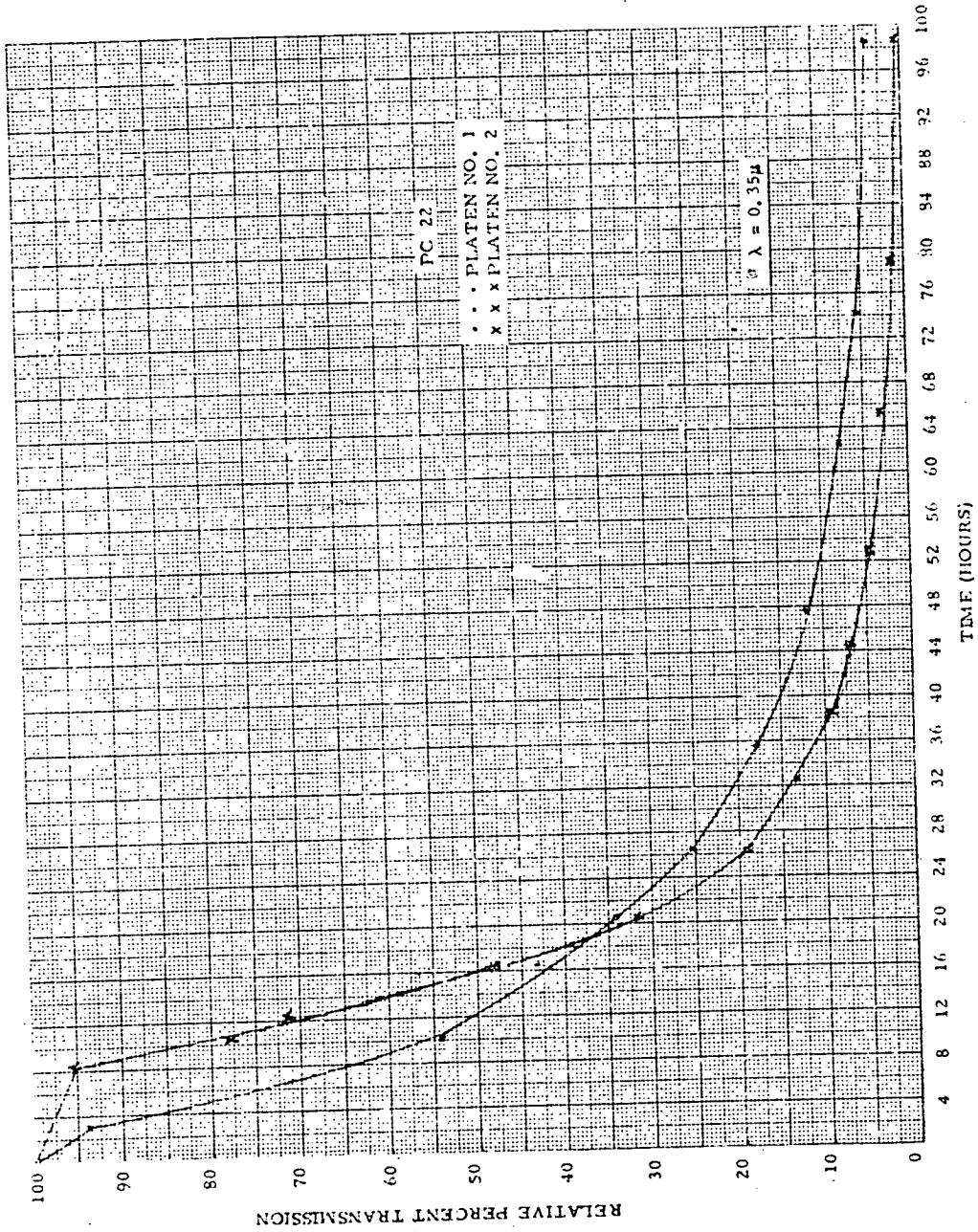


Figure 7-11b. PC 22 Test (Sheet 2 of 3)

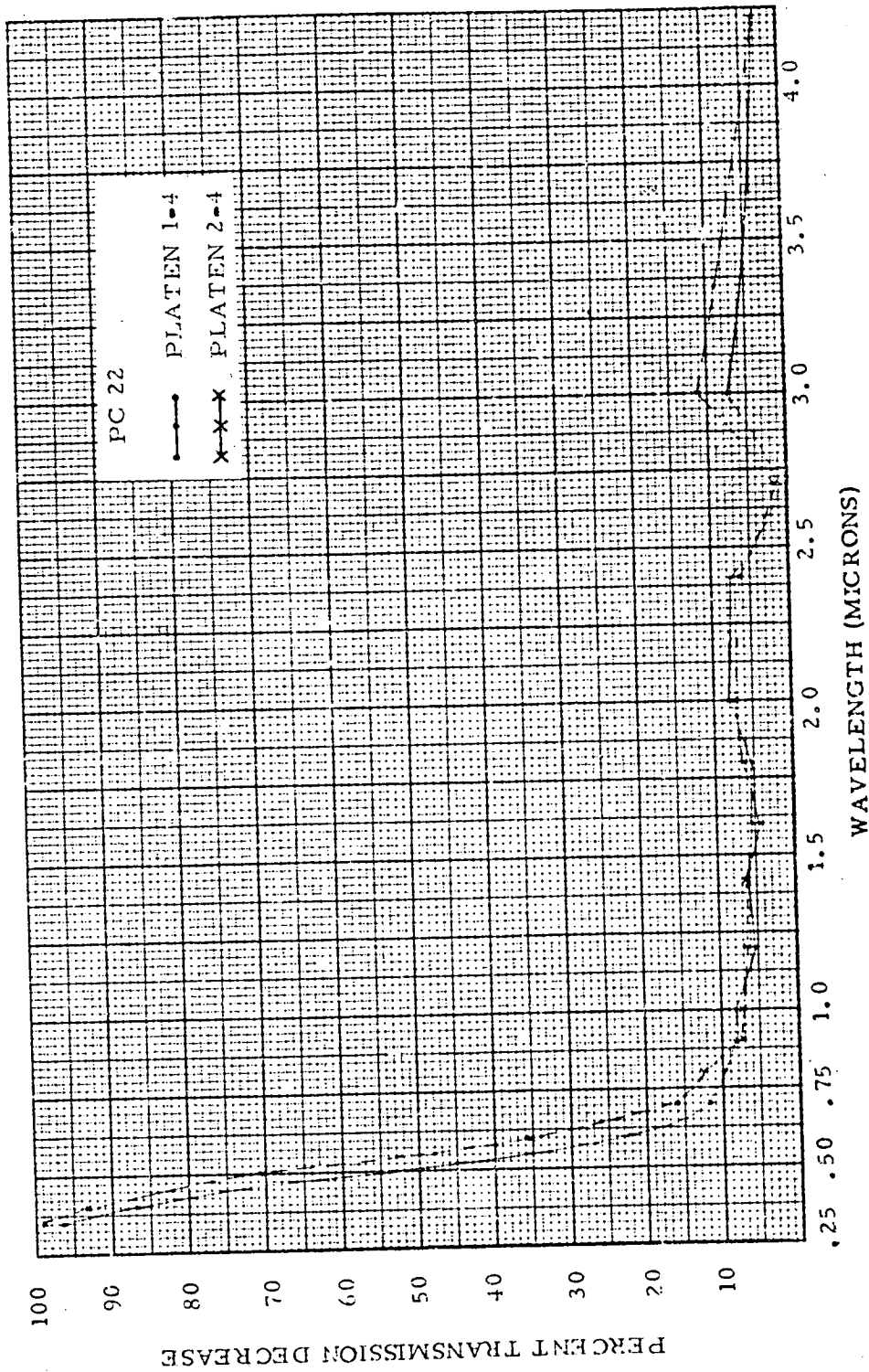


Figure 7-11c. PC 22 Test (Sheet 3 of 3)

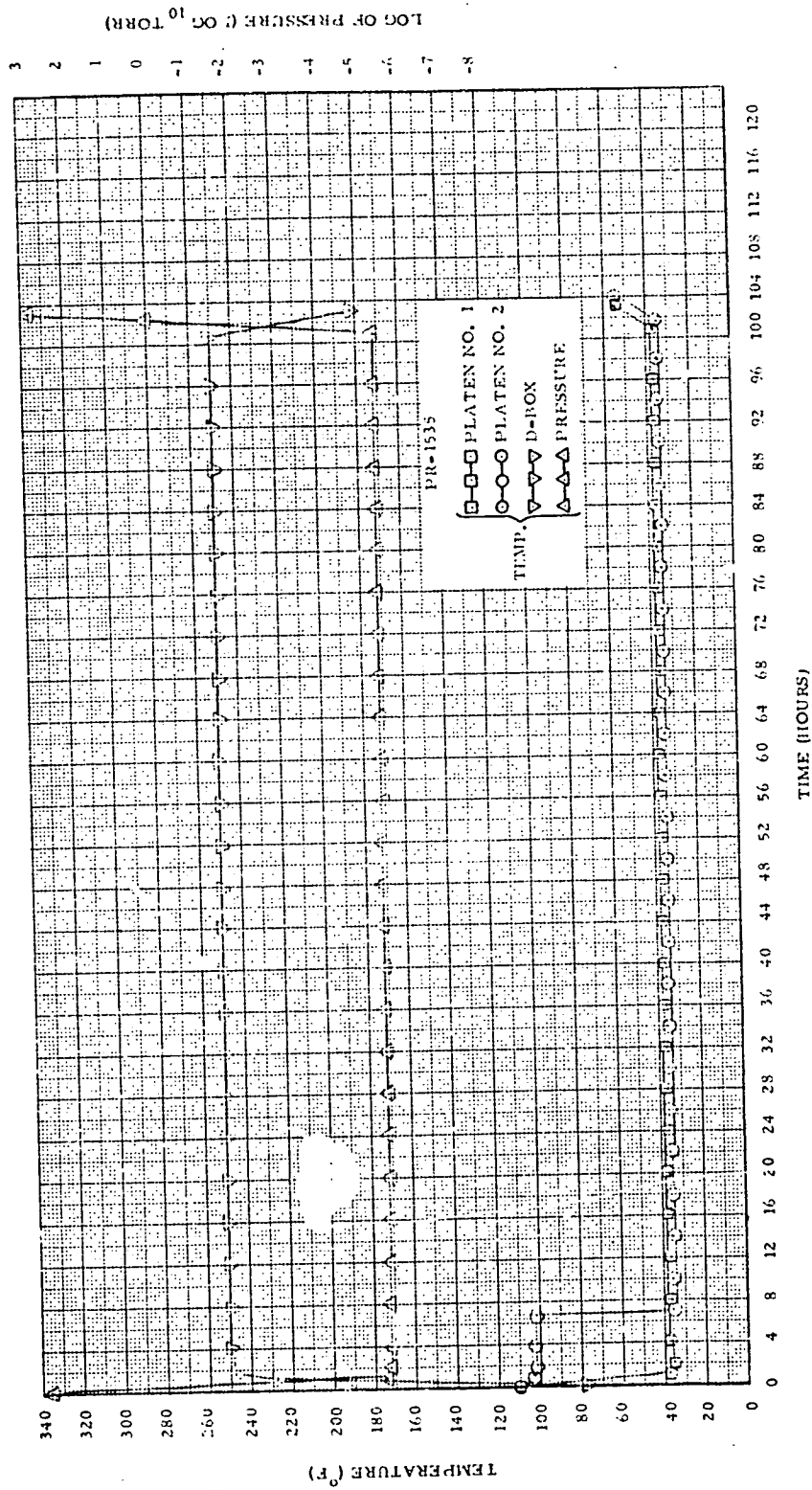


Figure 7-12a. PR-1535 Test (Sheet 1 of 3)

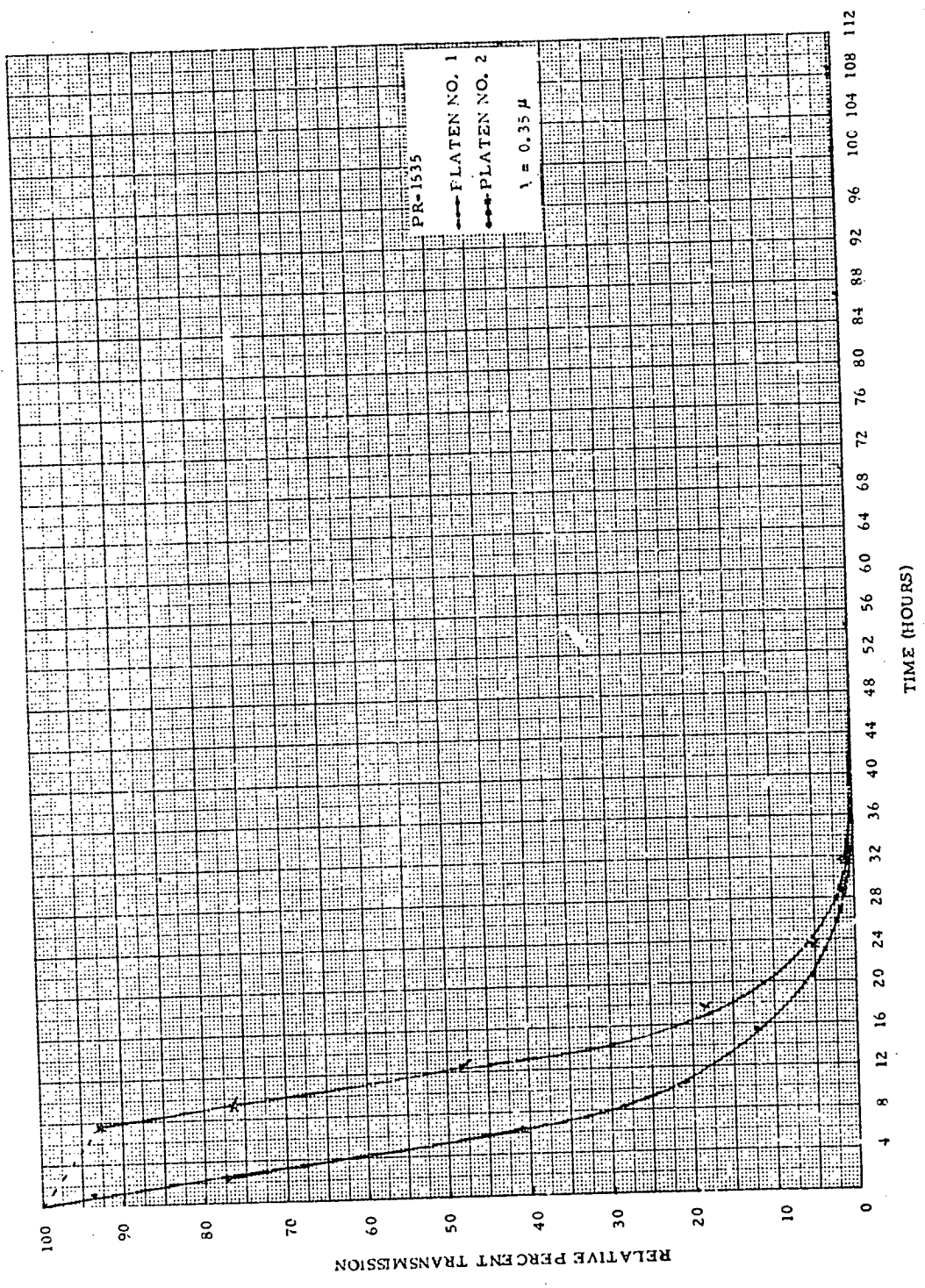


Figure 7-12b. PR-1535 Test (Sheet 2 of 3)

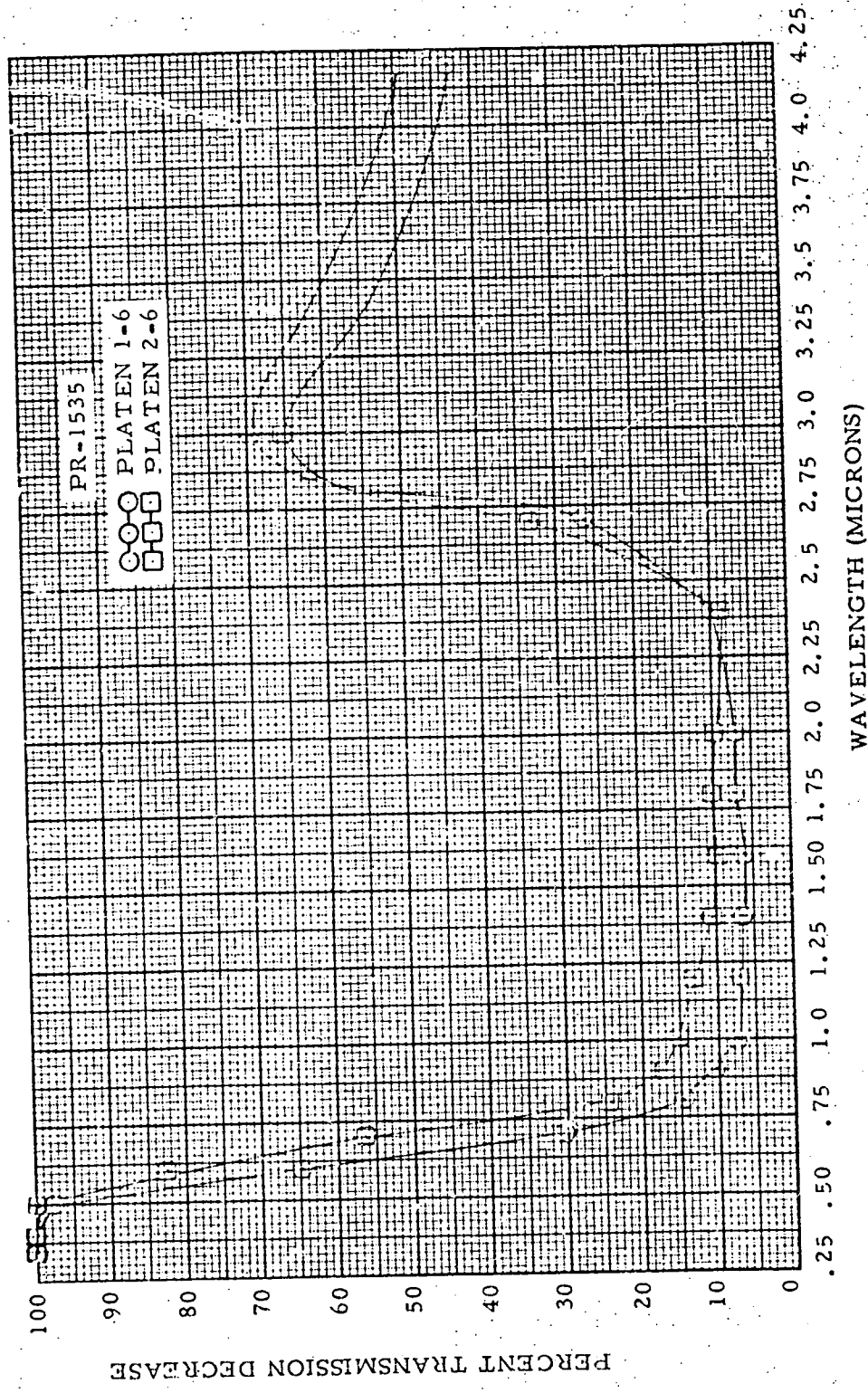


Figure 7-12c. PR-1535 Test (Sheet 3 of 3)

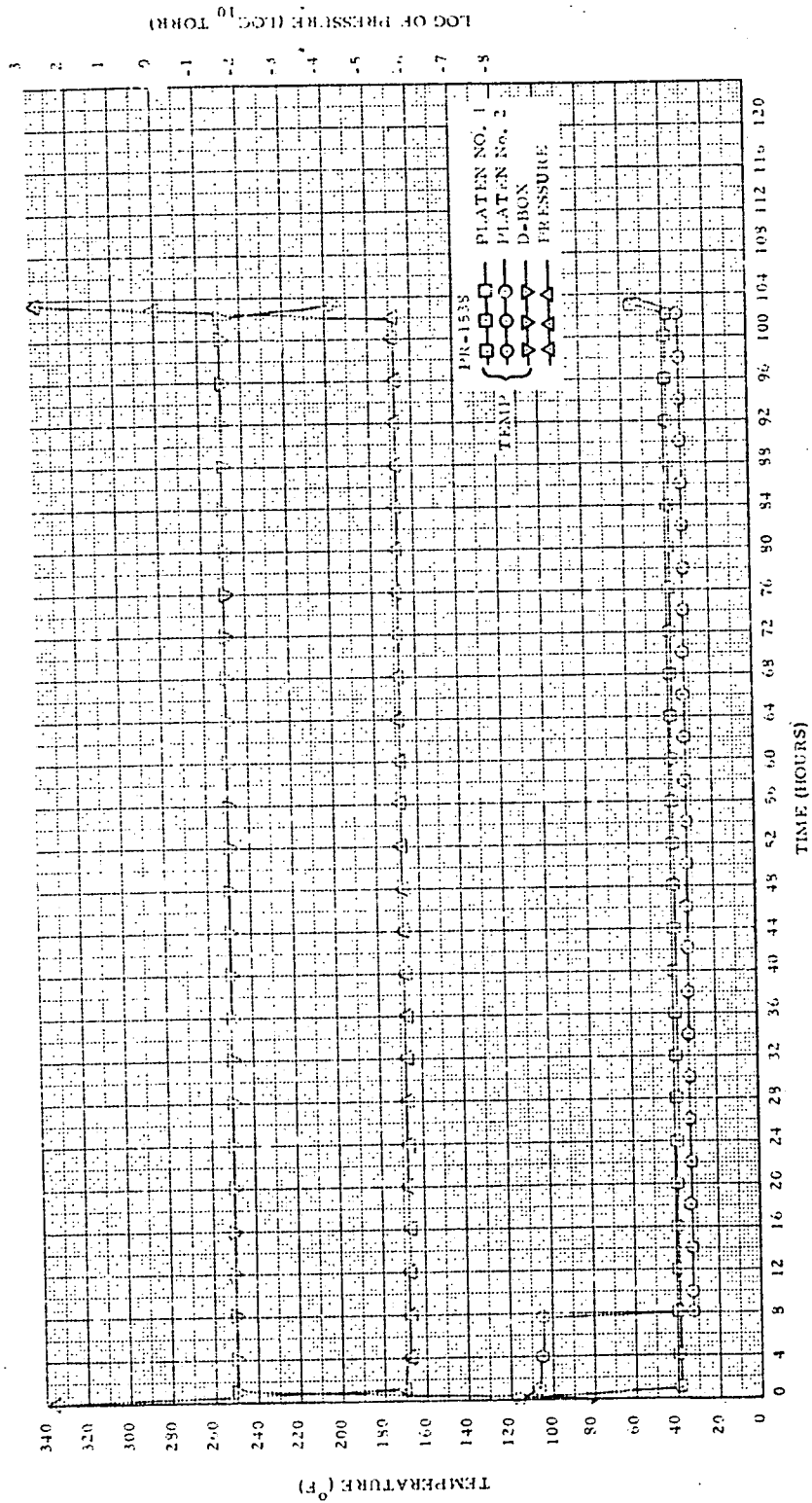


Figure 7-13a. PR-1538 Test (Sheet 1 of 3)

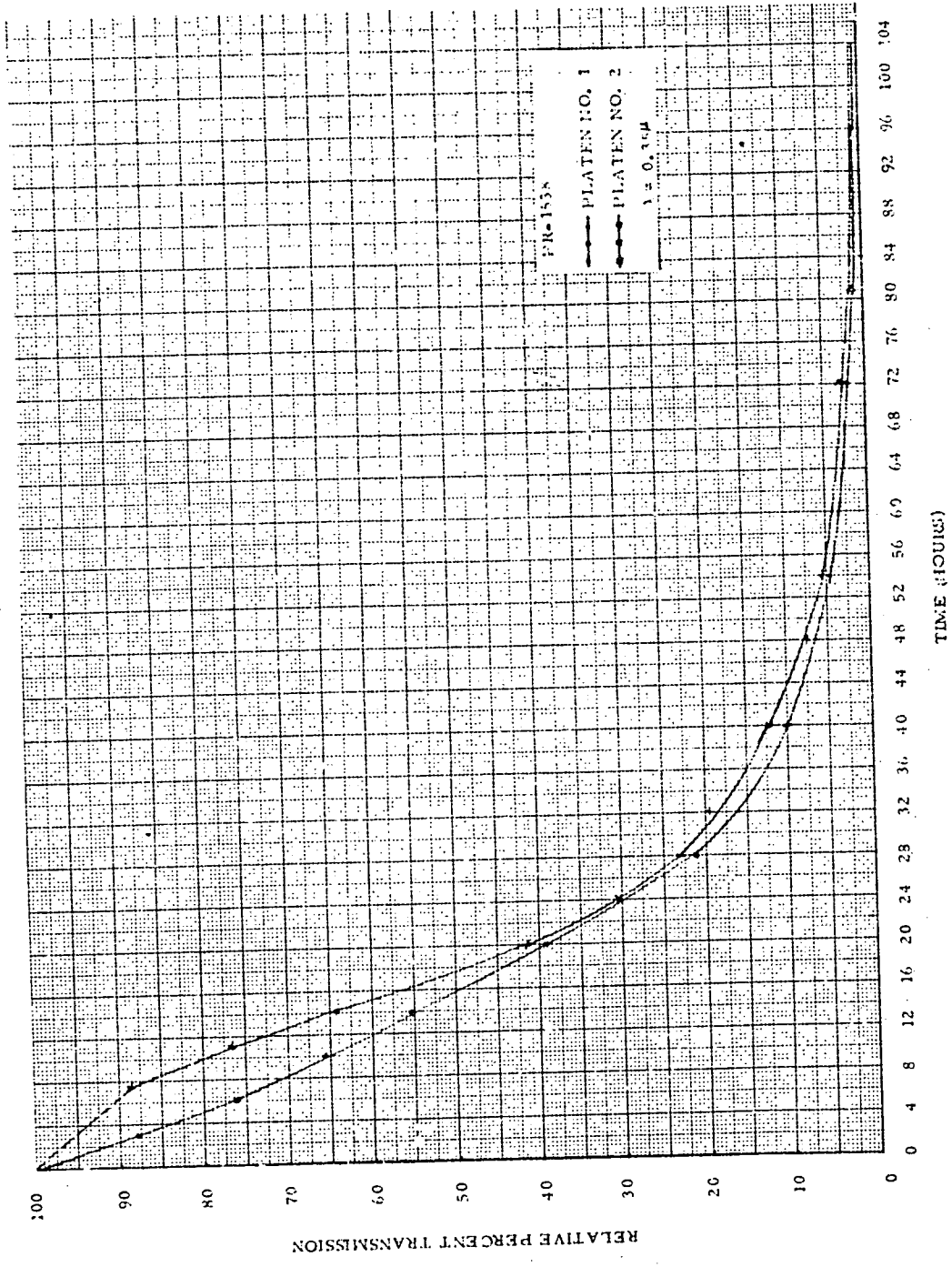


Figure 7-13b. PR-1538 Test (Sheet 2 of 3)

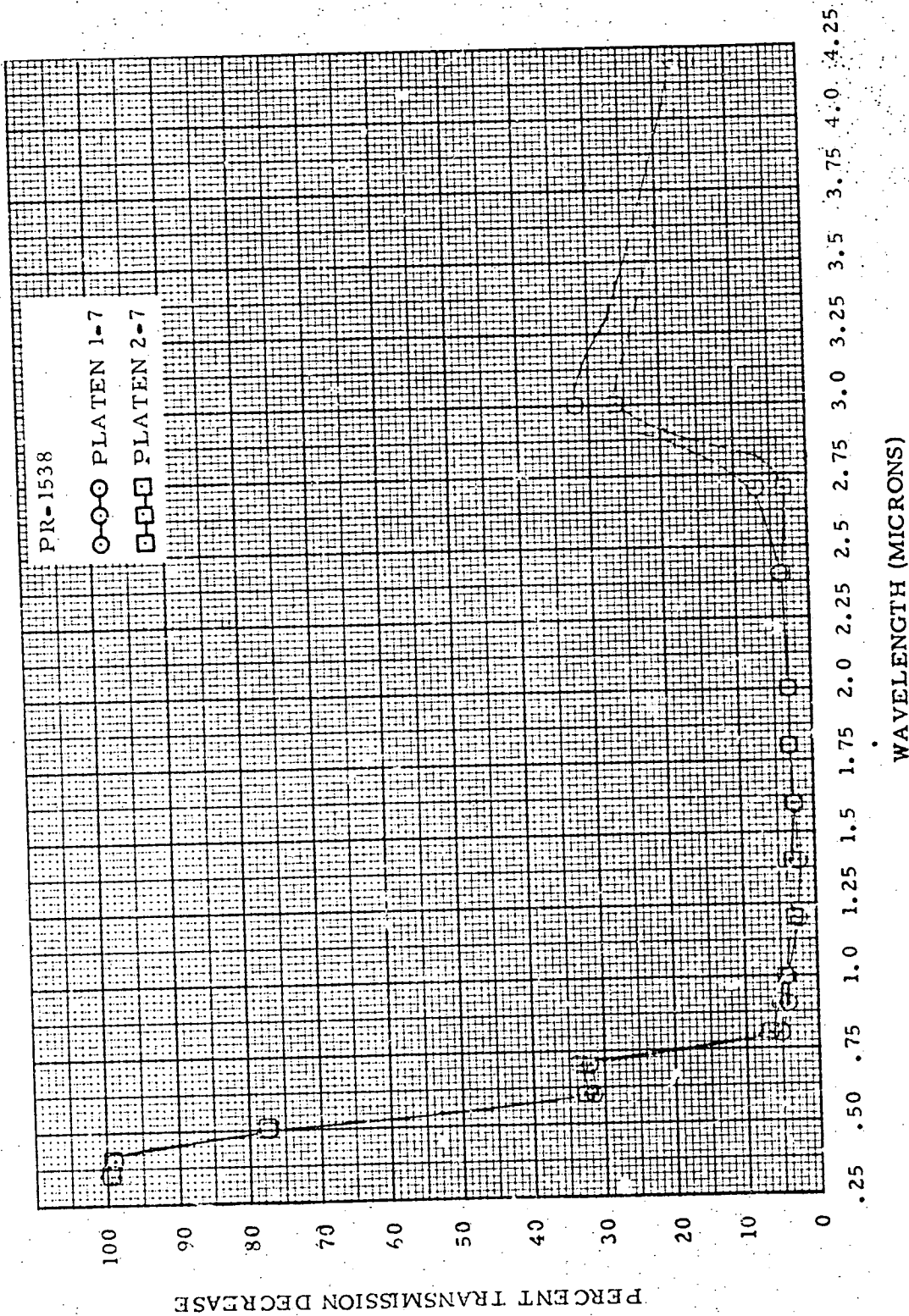


Figure 7-13c. PR-1538 Test (Sheet 3 of 3)

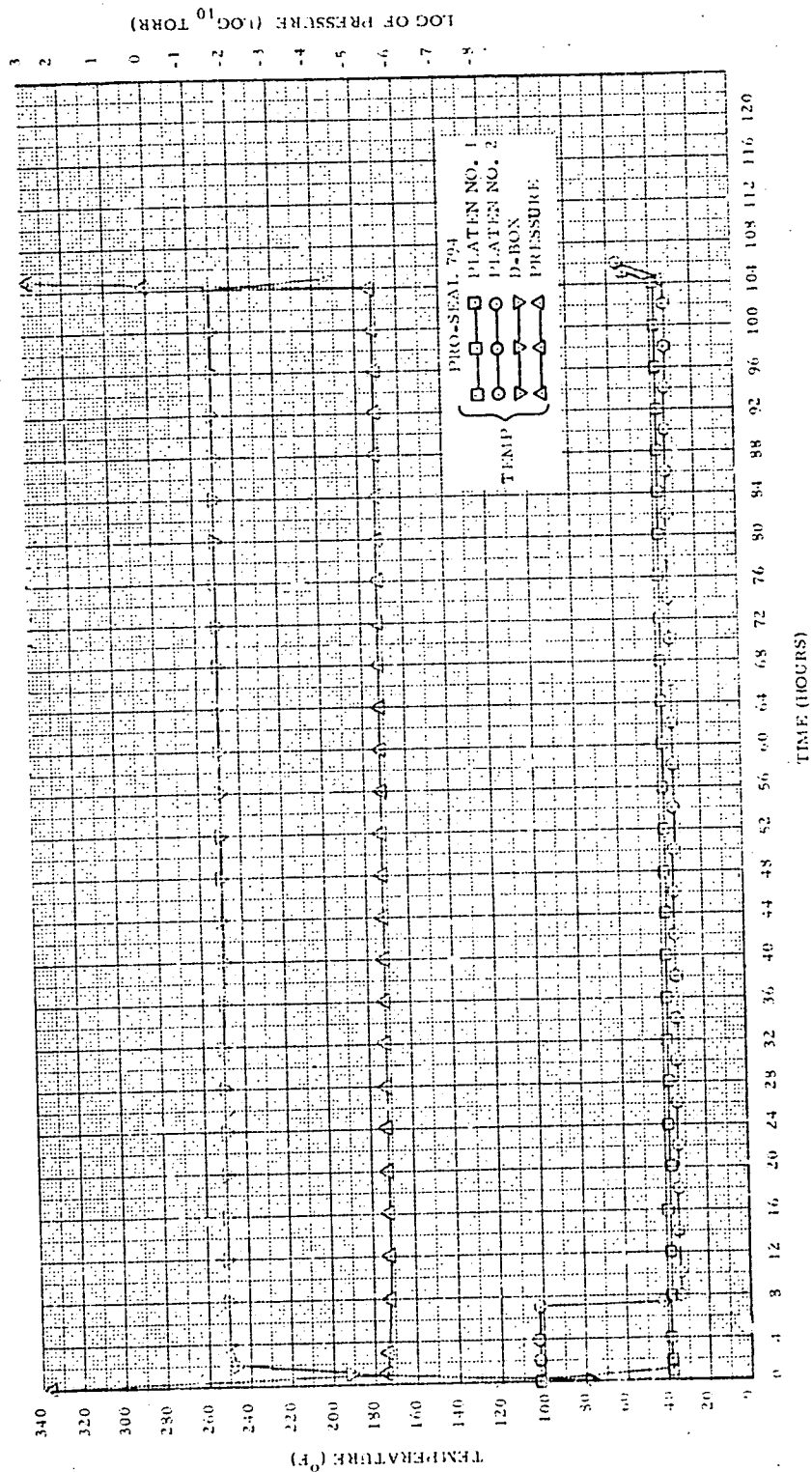


Figure 7-14a. Pro-Seal 794 Test (Sheet 1 of 3)

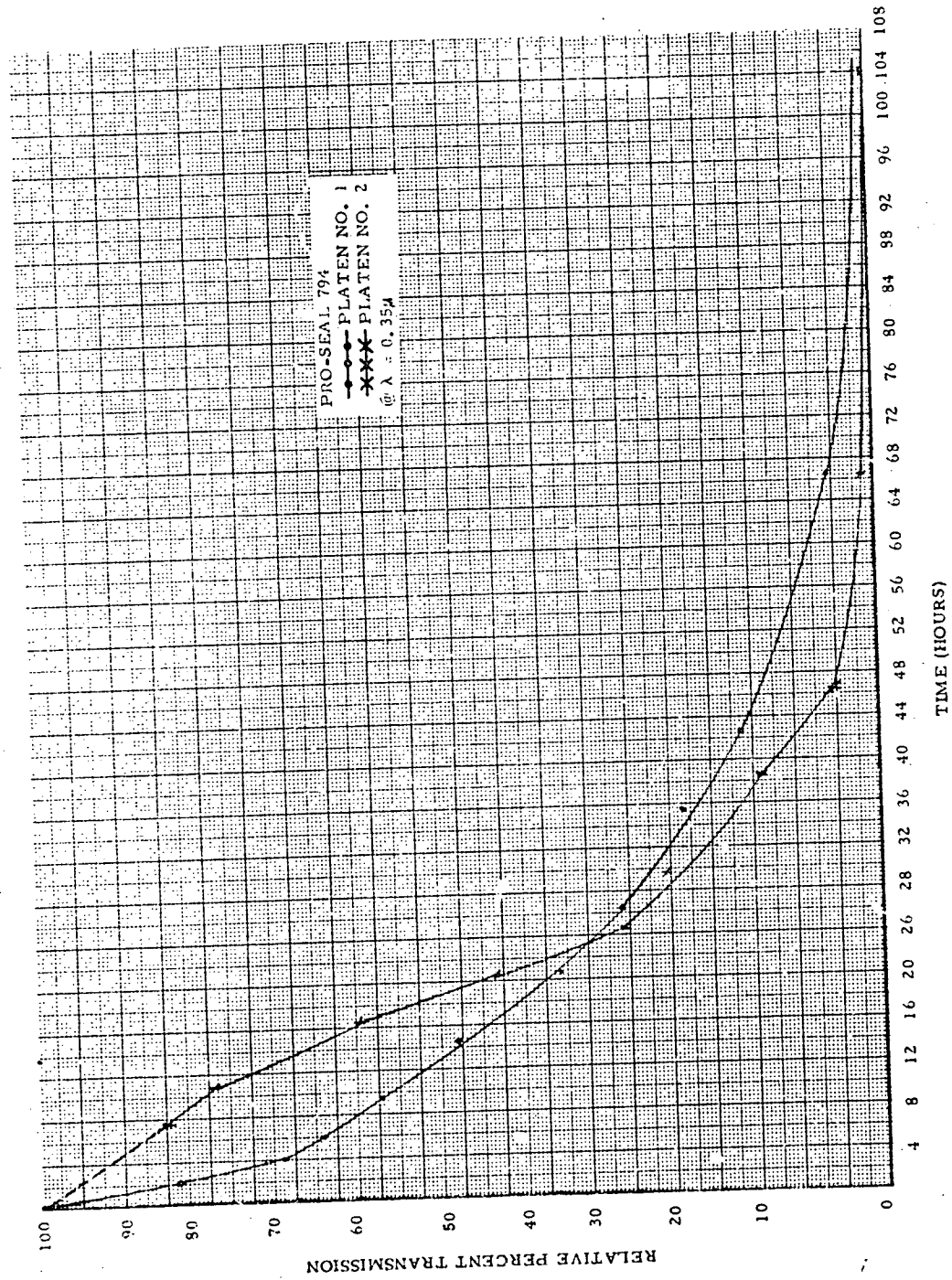
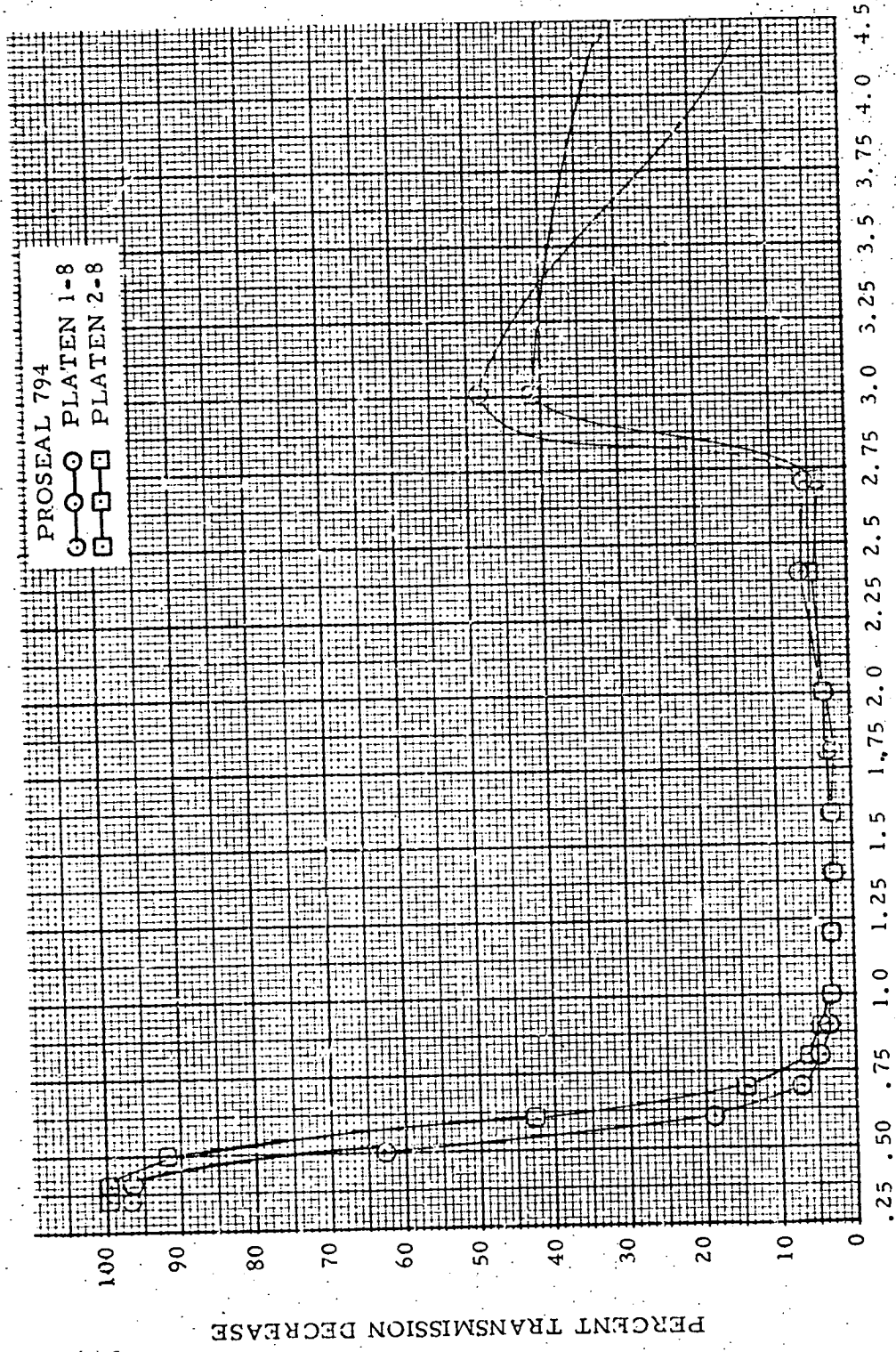


Figure 7-14b. Pro-Seal 794 Test (Sheet 2 of 3)



WAVELENGTH (MICRONS)

Figure 7-14c. Pro-Seal 794 Test (Sheet 3 of 3)

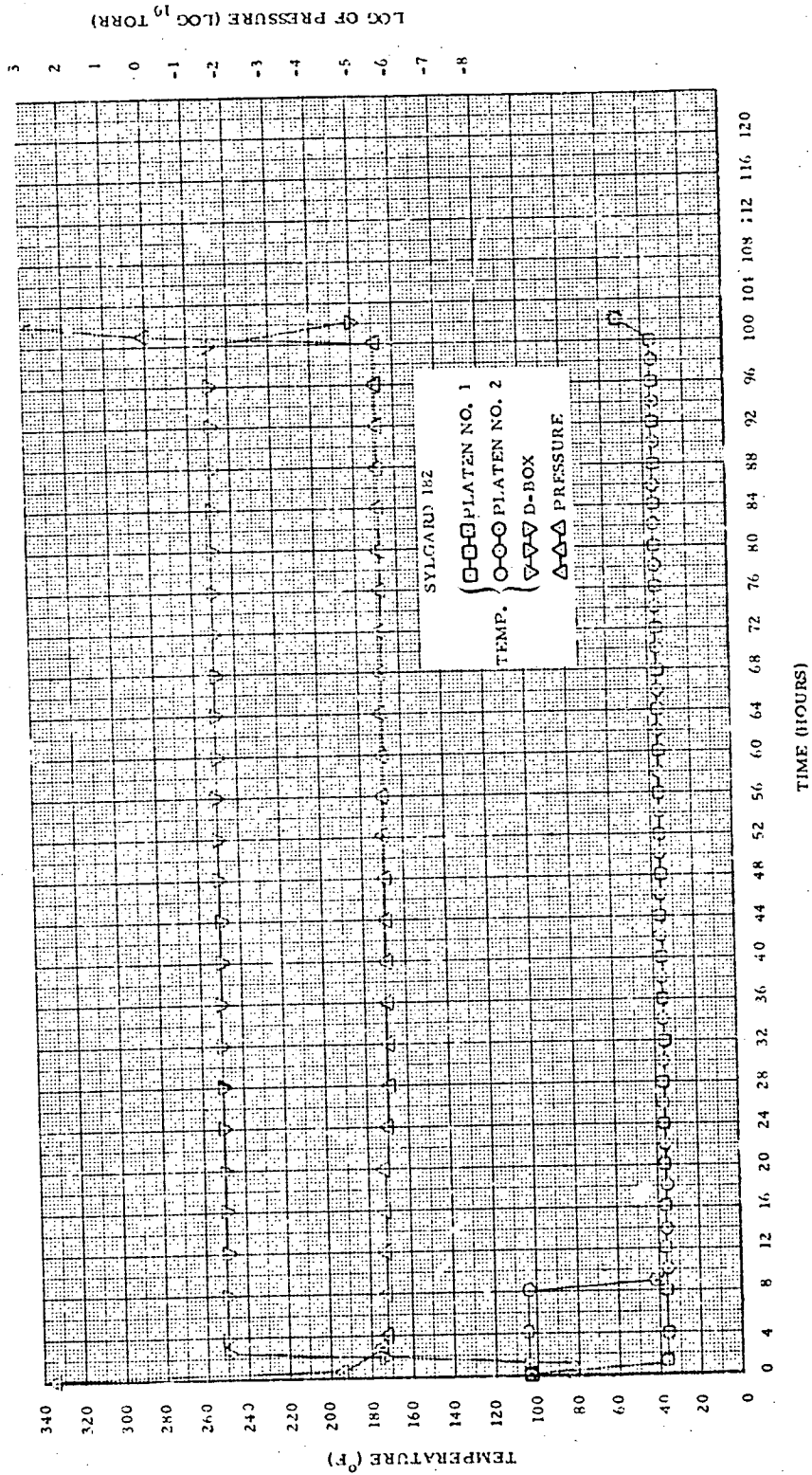


Figure 7-15a. Sylgard 182 Test (Sheet 1 of 3)

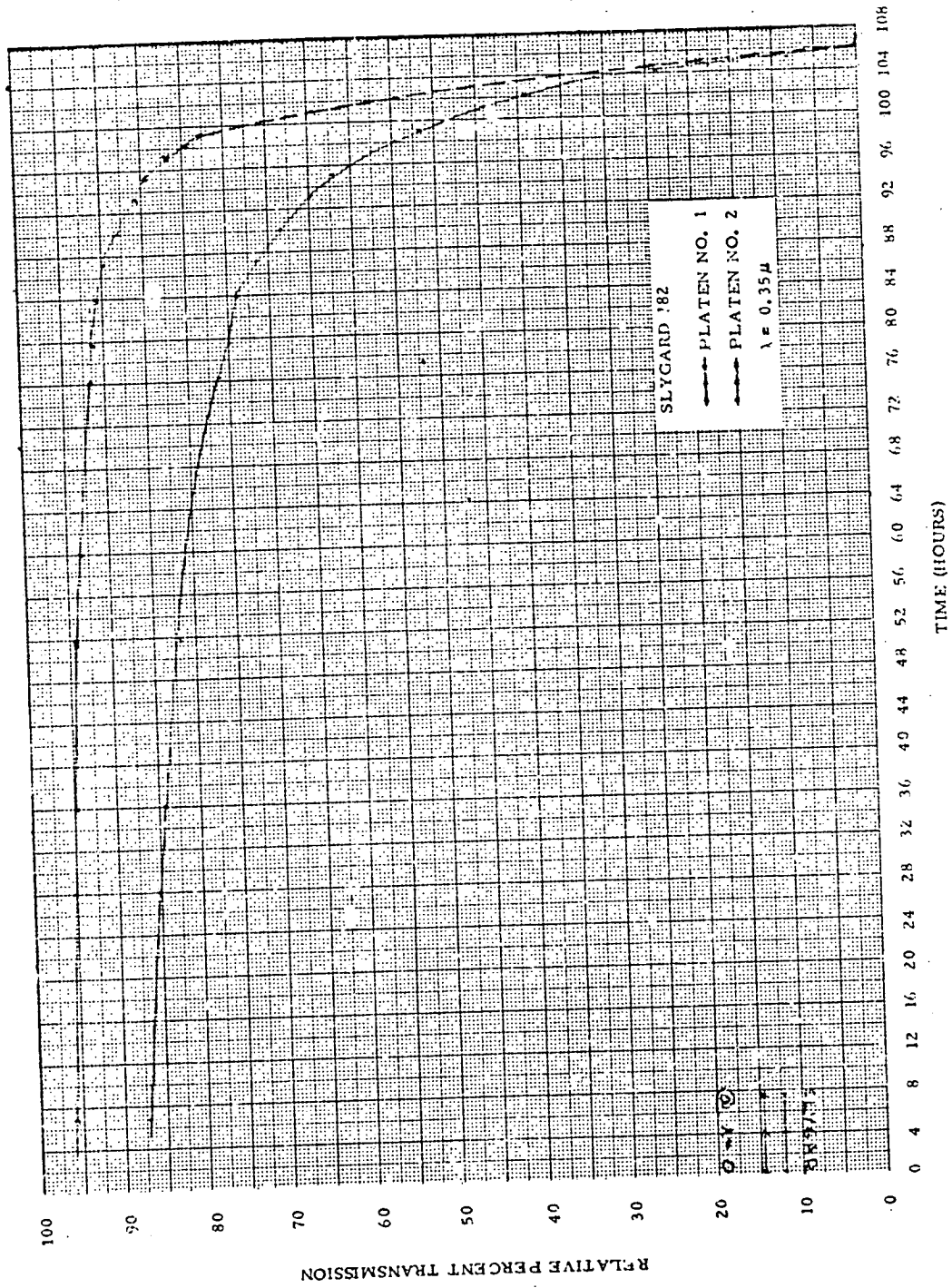


Figure 7-15b. Sylgard 182 Test (Sheet 2 of 3)

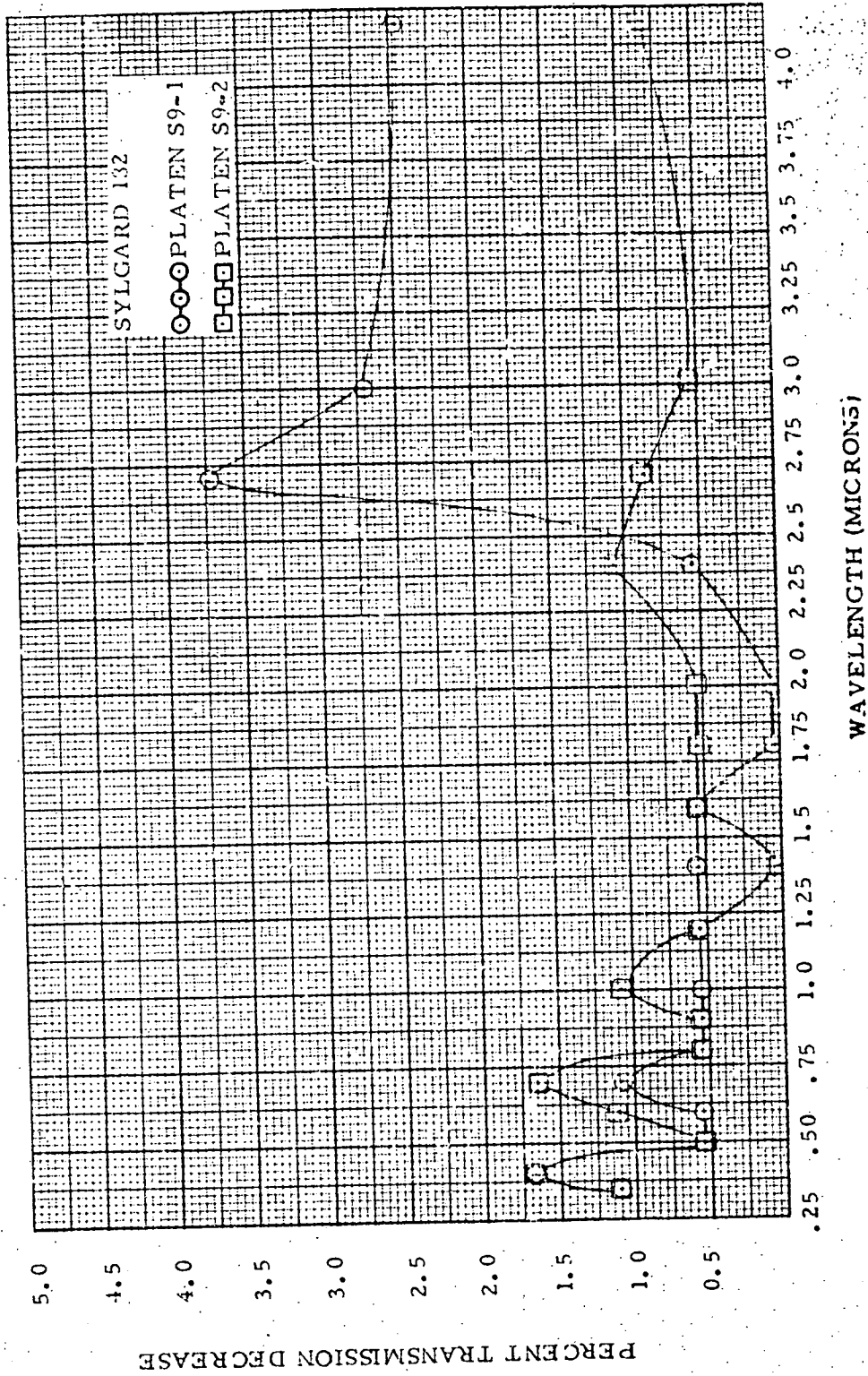


Figure 7-15c. Sylgard 162 Test (Sheet 3 of 3)

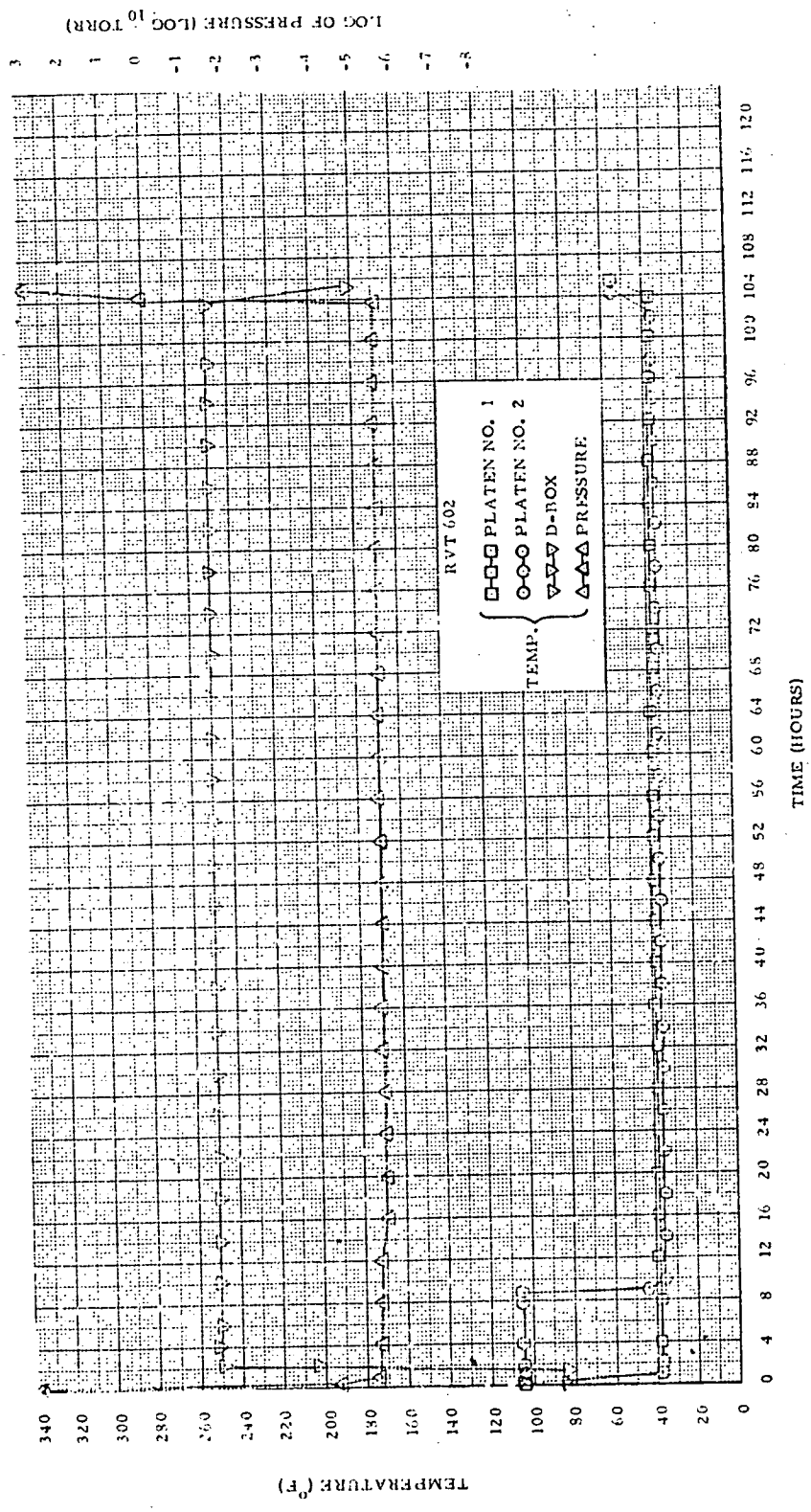


Figure 7-16a. RTV 602 Test (Sheet 1 of 3)

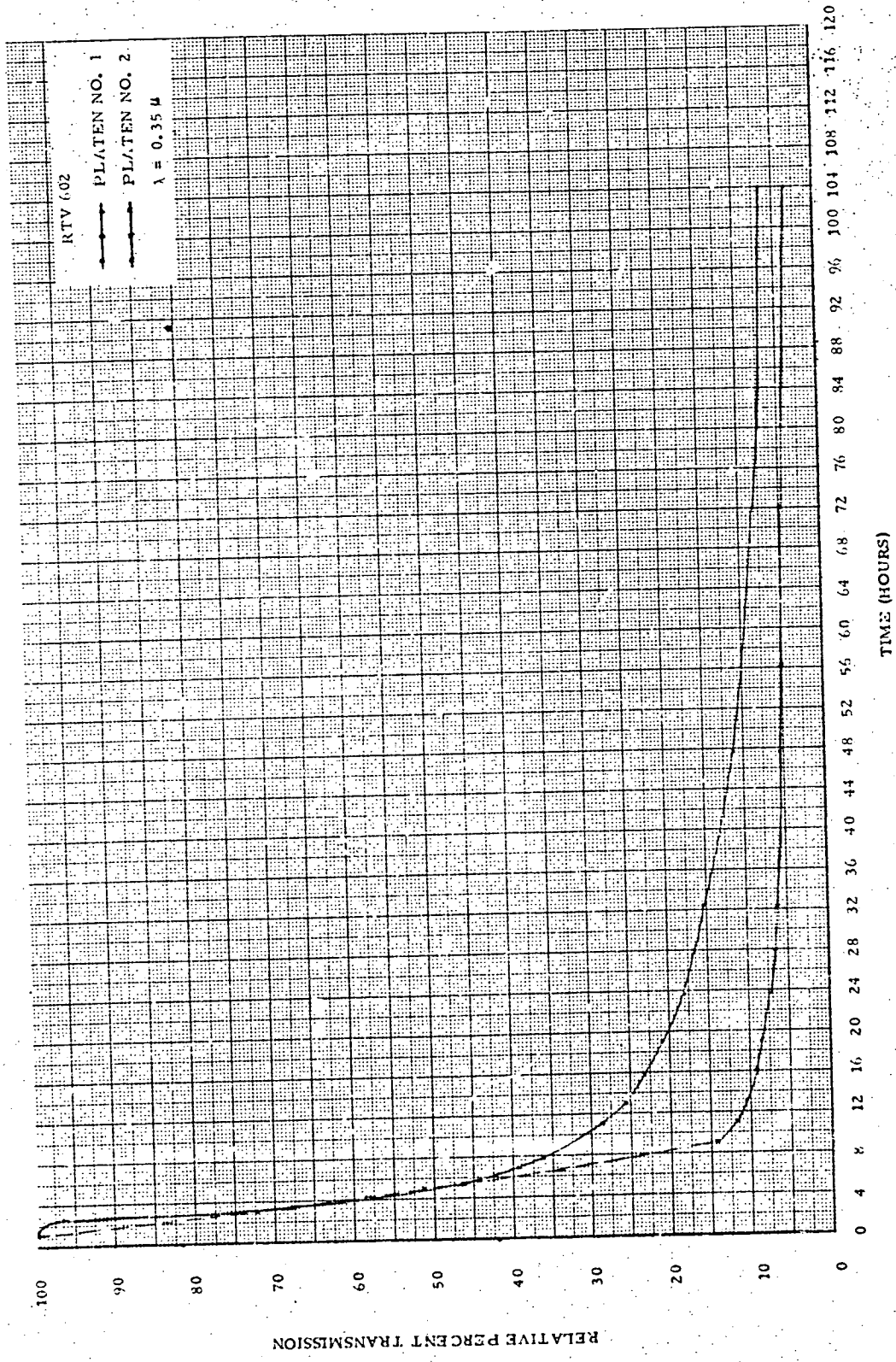


Figure 7-16b. RTV 602 Test (Sheet 2 of 3)

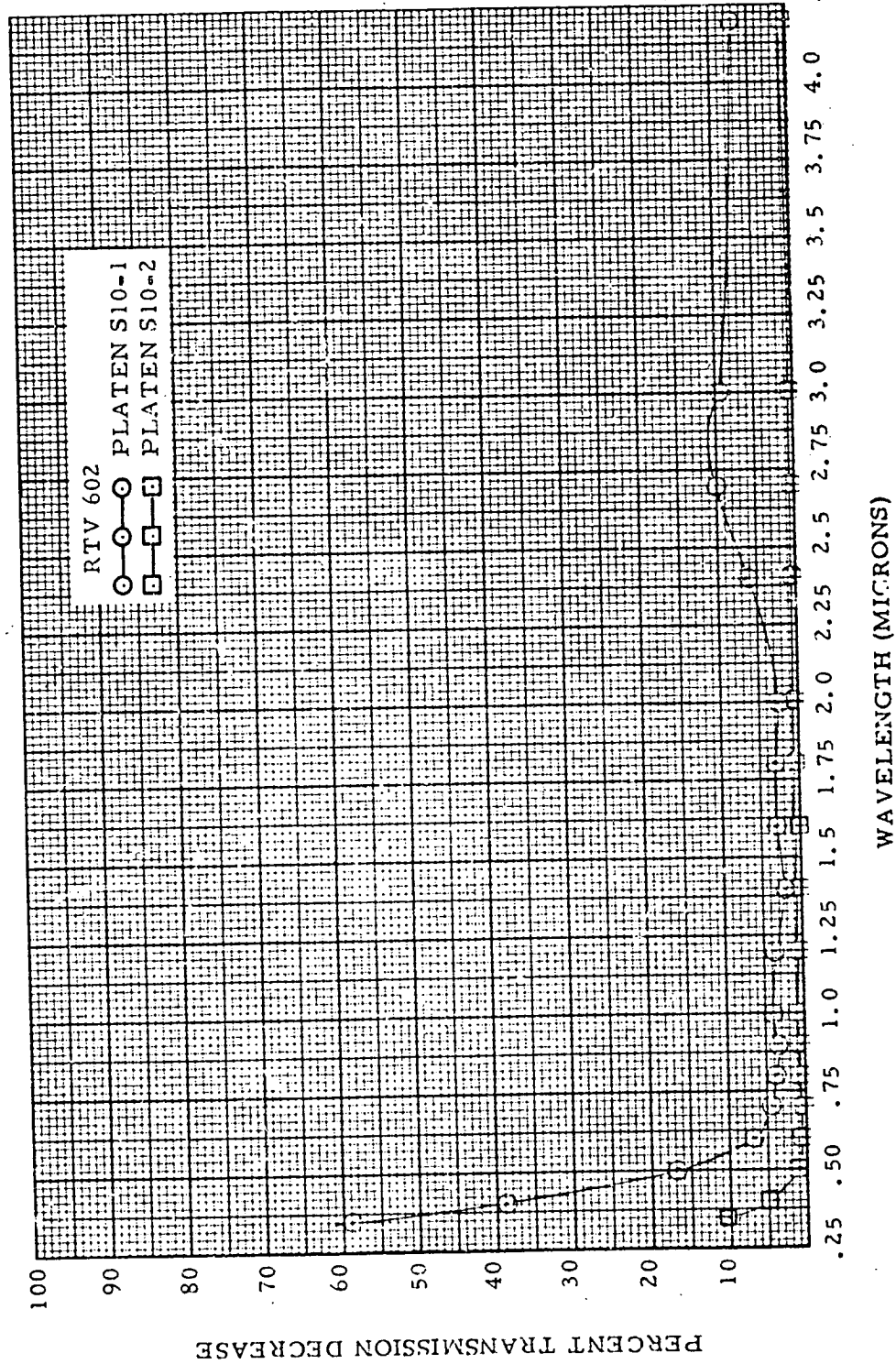


Figure 7-16c. RTV 602 Test (Sheet 3 of 3)

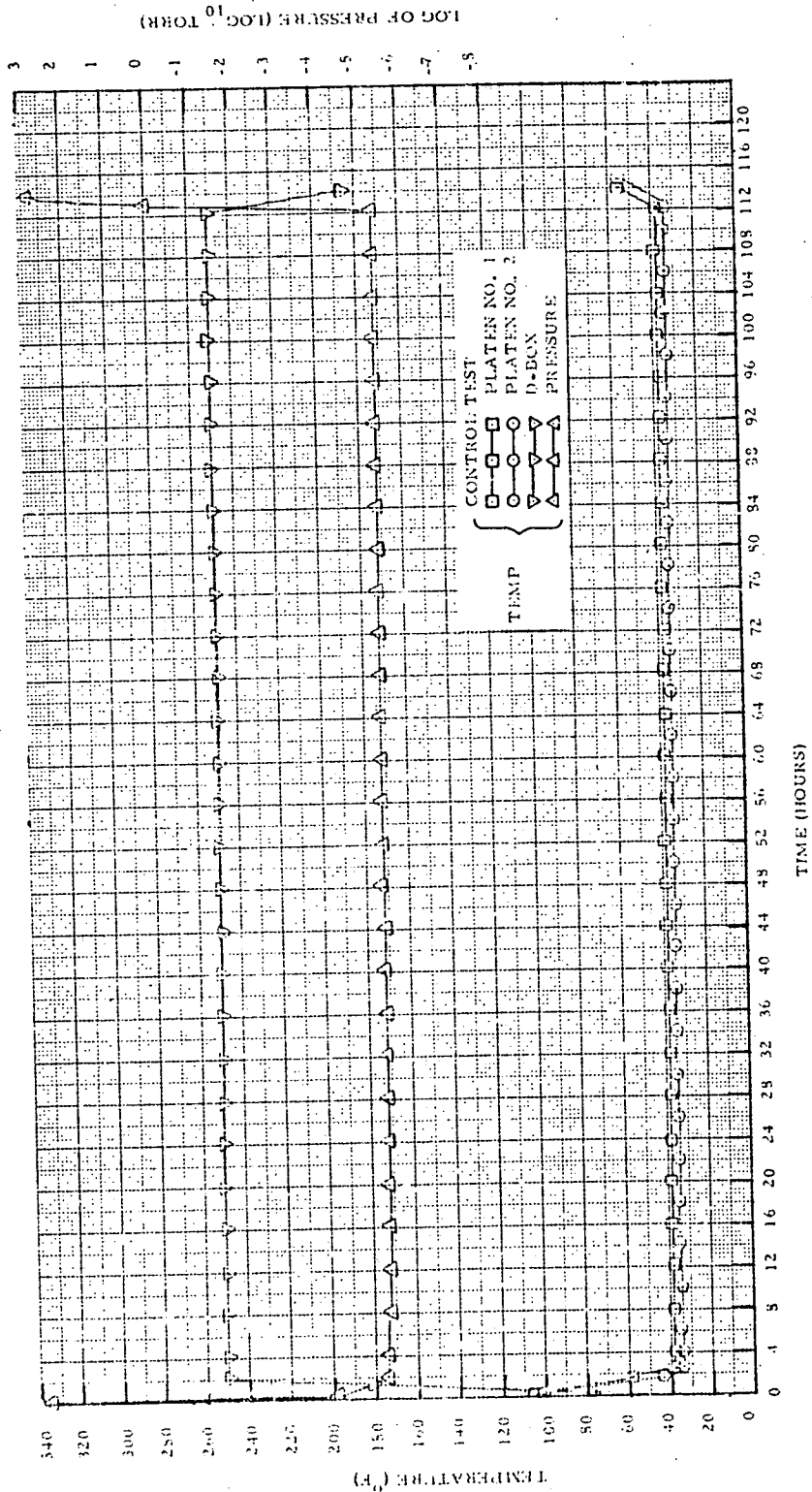


Figure 7-17a. Control Test (Sheet 1 of 3)

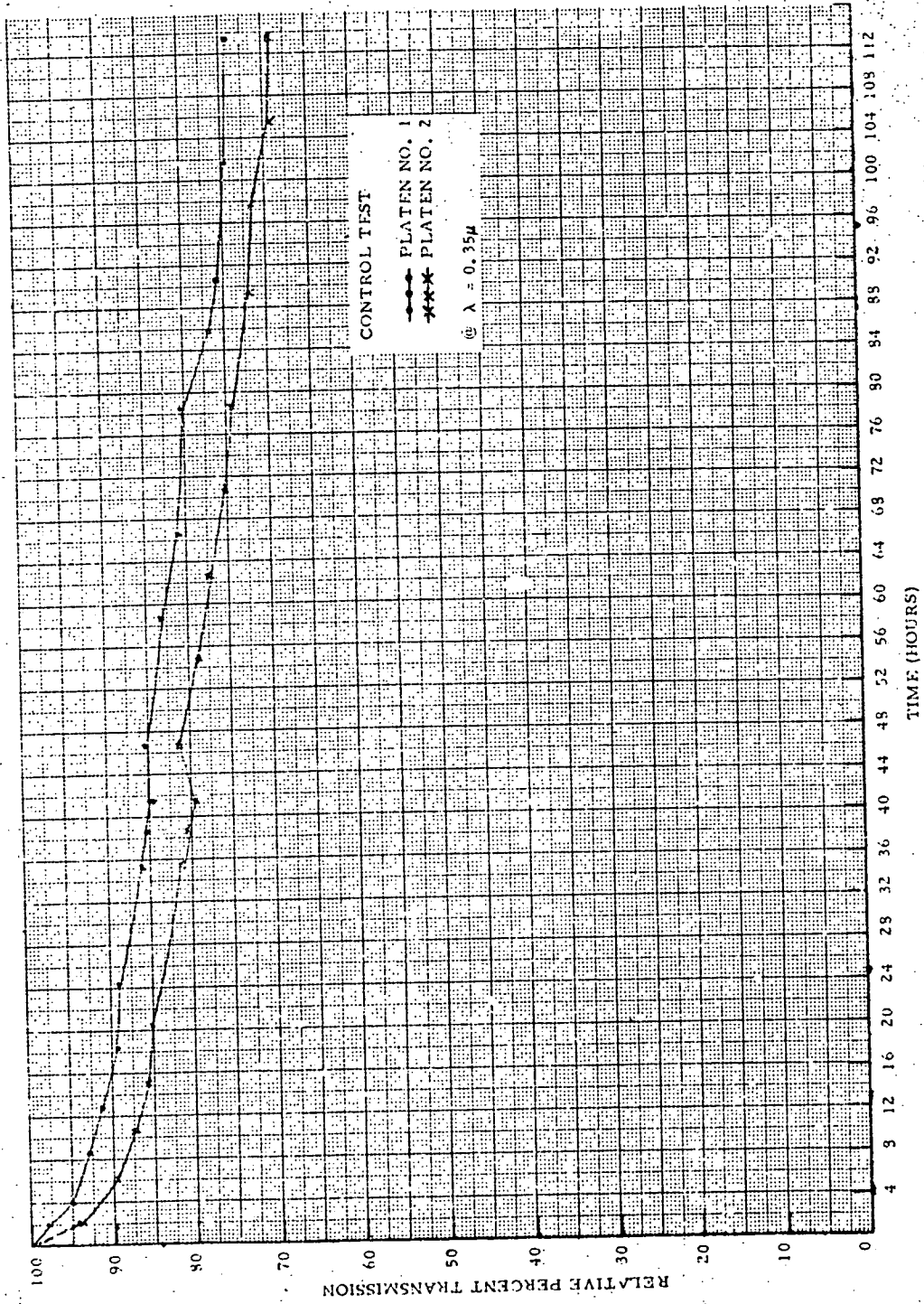


Figure 7-17b. Control Test (Sheet 2 of 3)

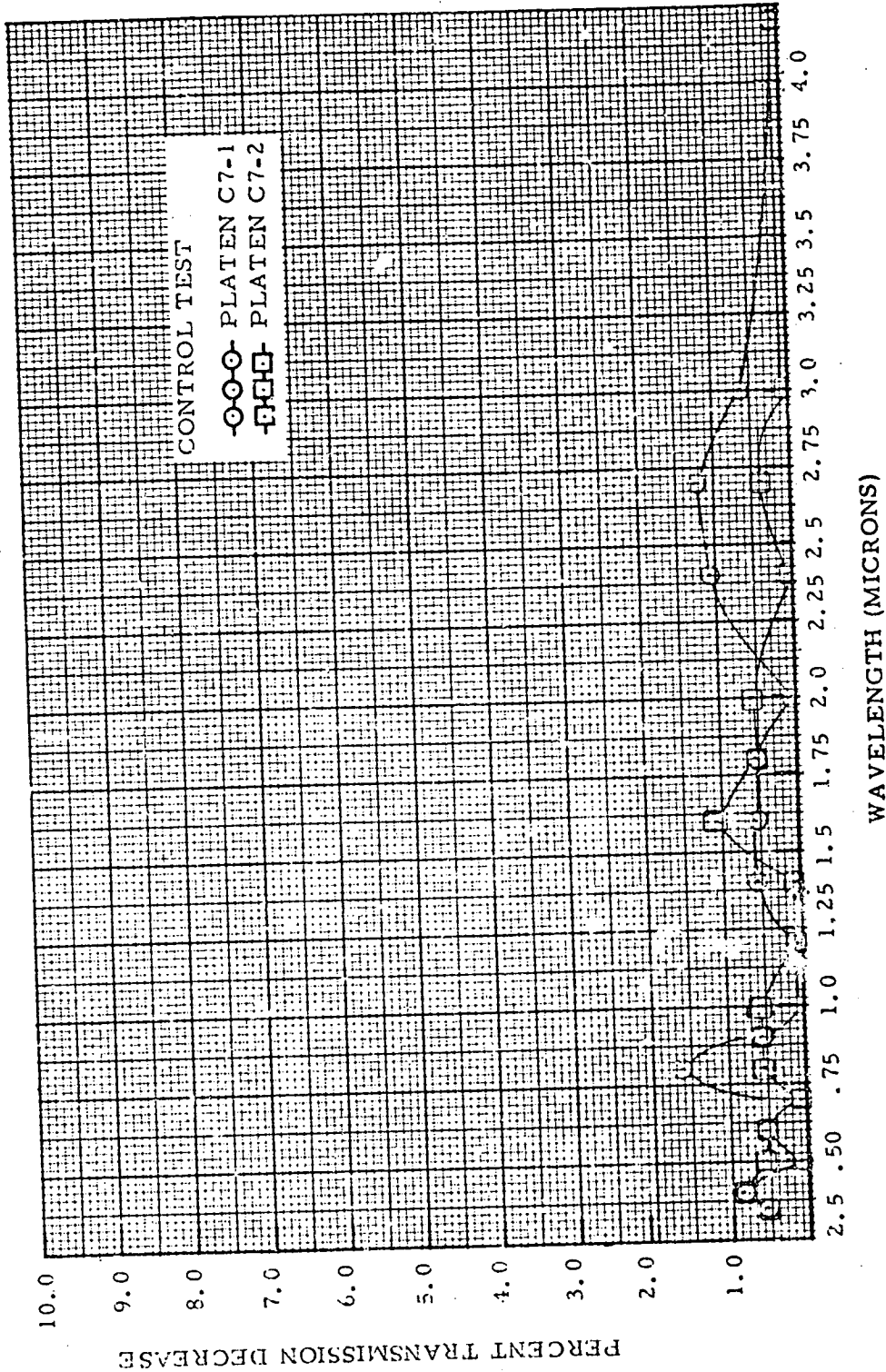


Figure 7-17c. Control Test (Sheet 3 of 3)

#### 7.4 DISCUSSION OF RESULTS

Differences noted between the in situ transmission plots and the before and after measurements probably result from one or more of the following:

1. Contamination of the sapphire UV port.
2. Difference of incidence angle on the quartz slide in the two measurements.
3. Changes in film property when brought from vacuum to air.

The epoxies exhibited only small decreases in light transmission in the UV and visible regions of the spectrum and were the best materials tested. EC-2850 was the worst of the epoxies based on its higher transmission loss in the near infrared. The 6-hour vacuum treatment had little effect on EC-1096, while it showed some improvement for EC-2651 and EC-2850. At this time it should be noted that the ordinate scale of the spectral transmission loss is in general different for each material.

The polyurethanes were the worst of the materials tested; however, 120°C is much higher than the normal use temperature for these materials in space hardware. The apparent "instant" decrease in the transmission of platen No. 2 when the flap is removed is probably due to photopolymerization on the window of particles reflected from the flap that was covering platen No. 2. All polyurethanes except PC 22 exhibited large decreases in transmission at about 3 microns. This is most likely due to hydrogen-carbon bond absorption. The 6-hour vacuum treatment did not result in any significant improvement for any of the polyurethanes.

Eccofoam FPH exhibited a spectral transmission loss similar to the epoxies but somewhat greater in magnitude. The 6-hour vacuum treatment resulted in a smaller light transmission loss in the near UV and the visible.

Both silicone rubbers produced heavy visible condensates compared to their corresponding transmission degradation. The Sylgard 182 appeared to result in less degradation than the RTV 602, based on before and after measurements. The large and apparent "instantaneous" transmissions decrease on platen No. 2 when the flap was opened and the large difference in transmission loss between the in situ and the before and after measurements is due to photopolymerization on the sapphire UV window. This was proven after the test by transmission measurements performed on the sapphire window before and after it was cleaned. The 6-hour vacuum treatment resulted in a large improvement in the light transmission for the RTV 602 in the near UV, visible, and at about 3 microns, whereas it resulted in a significant improvement only at about 3 microns for the Sylgard 182.

The percent bulk weight loss for the epoxies was essentially zero, in agreement with their corresponding low transmission loss. The Eccofoam has a large bulk weight loss compared to its low transmission loss and the small amount of visible condensate. This was in contrast to the silicone rubbers, which exhibited large visible condensates in spite of their low transmission losses and large bulk weight losses. The polyurethanes all exhibited large weight losses and there was no obvious relationship between that and their corresponding light transmission losses when considered as a group.

#### 7.5 CONCLUSIONS AND RECOMMENDATIONS

Based on light transmission loss and bulk weight loss the epoxies were the best generic type of material tested and were followed closely by the foam. The polyurethanes were very bad from both points of view and should not be considered for use at 120<sup>o</sup>C. The silicone rubbers exhibited high bulk weight losses but low transmission loss with the exception of untreated RTV 602. Because of their high sensitivity to photopolymerization one should not consider this type of silicone rubber for use near critical surfaces exposed to UV.\* Although the light transmission loss was low for the silicone rubbers, these measurements provide no information concerning the resolution change that could occur due to scattering. It is recommended that the space-grade silicone rubbers be investigated for use where they offer significant advantages over other types of materials.

\* Space-grade silicone rubbers are now available from both Dow Corning and General Electric.  
\*\* The silicone rubbers were an exception with only the untreated RTV 602 causing appreciable transmission loss in the near UV.

There was only a limited correlation between bulk weight loss and light transmission loss. Materials with a near zero bulk weight loss produced very little transmission loss whereas materials with significant bulk weight losses may or may not have produced appreciable light transmission losses. This is not surprising since:

1. All condensates do not have the same absorption coefficients.
2. All outgassing fragments do not have the same sticking coefficient under the conditions tested.
3. The bulk weight loss is the theoretical maximum amount of condensables which a material releases.

The spectral regions which exhibited the major transmission losses were generally the near UV\* and the infrared at about 3 microns. This was not surprising as most organics (hydrocarbons) absorb strongly in the UV and infrared. Strong absorption bands should be expected in the remainder of the infrared at least to 15 microns. Large transmission losses should also be expected throughout the UV, clear into the soft X-ray region. There was no general trend observed on the effect of the 6-hour thermal-vacuum treatment on transmission loss.

Some additional items which should be considered in future studies of this type include: new materials, multiple sample and condenser temperatures, different condenser surfaces, condensation identification, synergistic radiation effects, basic condensation parameters, and other optical property measurements.

It should be noted that much more than programs of this type are required to solve the optical contamination problem. We believe that a Quantitative Systems Condensation Analysis (QSDA) is the best technique to eliminate this problem. QSDA will eliminate condensation problems through minor materials, process, and design changes which are suggested from a total system analysis.

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