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QUALIFICATION OF A NEW NAPO SOURCE AND ERL-510 CURING AGENT FOR--ETC(U)
JUL 79 J A THOMPSON
MANCP-419(79)

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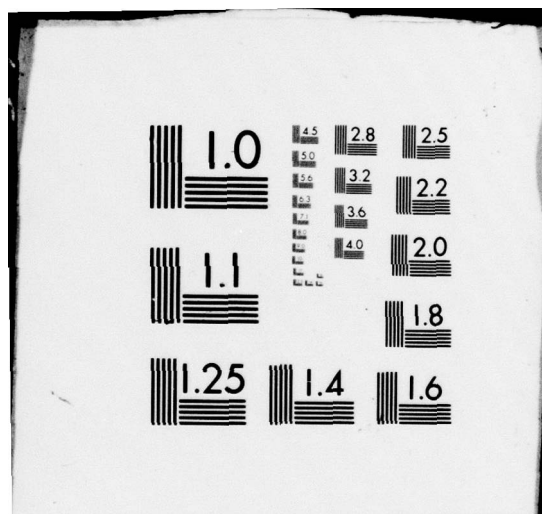
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LEVEL II

QUALIFICATION OF A NEW MAPO
SOURCE AND ERL-510 CURING AGENT
FOR MINUTEMAN STAGE 1 UF-2121 LINER

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QUALIFICATION OF A NEW MAPO
SOURCE AND ERL-510 CURING AGENT
FOR MINUTEMAN STAGE 1 UF-2121 LINER

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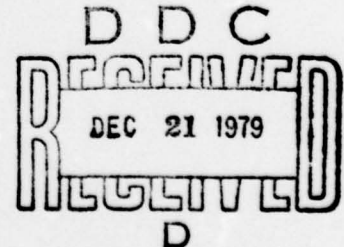
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ABSTRACT

Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required.

Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.

This report includes the test results for the first, second, third, fourth and fifth time testing of the control and special specimens at Ogden ALC.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 REV A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

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INTRODUCTION

A. PURPOSE:

Quality assurance testing of specimens prepared from the new source of MAPO to assure that liner material for First Stage Minuteman Motors will perform as predicted.

B. BACKGROUND:

*Tris [1-(2 methyl) aziridinyl] phosphine oxide (MAPO) is used as a curing agent in the Minuteman Stage One UF-2121 liner. MAPO was produced by Immont Chemical (Immont) and shipped to Arsynco Incorporated (Arsynco) for purification and marketing. Immont sold the production rights for MAPO to Arsynco and terminated the production of raw MAPO in 1972.

Since MAPO is a critical ingredient in UF-2121 liner formulation, the source change for the manufacture of MAPO was considered a serious change. Therefore, it was necessary for Thiokol to conduct qualification testing on liner material using MAPO manufactured by Arsynco before it could be considered acceptable for use in Minuteman Stage I UF-2121 liner.

ERLA-500 was the qualified epoxy curing agent used with MAPO in the UF2121 liner. Union Carbide terminated their process for ERLA-500. ERLA-510 used in similar liners (i.e. UF-2137) was substituted for ERLA-500 and qualified with MAPO from the new source.

The test conditions and test methods are shown in Table I.

*TWR-7857 Rev A Report, J. W. Rabern

Qualification testing was performed by Thiokol and reported in TWR-7857 Rev A. In addition, specimens were prepared by Thiokol from the new vendor's material and from the old source material for a "follow on" test program. These specimens were then transferred to Ogden ALC for a continuing surveillance test program designed to cover a ten year span. The material from the old source will be used as the control samples.

The ten year sampling plan is shown below. Those specimens identified for the fifth year were tested at this test period. The types of specimens are Disc (steel/liner/steel), Cup (steel/liner/TP-H1011), and Peel (broadcloth/liner/TP-H1011). For the disc specimen, the adhesion between the liner and steel is the critical factor. For the cup specimen, the adhesion between the propellant and the liner is critical. For the peel specimen the propellant to liner peel strength when pulled at 180° is critical.

TEN YEAR CONTINGENCY AGING SAMPLE CODING

Age (yr)	Storage Temp (°F)	Disc (Sample Nr)		Cup (Sample Nr)		Peel (Sample Nr)	
		Control*	Special**	Control*	Special**	Control*	Special**
1	75	1 - 6	181-186	61-66	241-246	121-126	301-306
2	75	7 -12	187-192	67-72	247-252	127-132	307-312
3	75	13-18	193-198	73-78	253-258	133-138	313-318
4	75	19-24	199-204	79-84	259-264	239-144	319-324
5	75	25-30	205-210	85-90	265-270	145-150	325-330
6	75	31-36	211-216	91-96	271-276	151-156	331-336
7	75	37-42	217-222	97-102	277-282	157-162	337-342
8	75	43-48	223-228	103-108	283-288	163-168	343-348
9	75	49-54	229-234	109-114	289-294	169-174	349-354
10	75	55-60	235-240	115-120	295-300	175-180	355-360

* Liner mix A73-11846 - control or old MAPO source material

** Liner mix A73-11810 - Experimental MAPO

TABLE I

Test Conditions and Methods

<u>Group</u>	<u>Test</u>	<u>Condition</u>	<u>Config- uration</u>	G085 Spec Code	Spec Per Cond	Total Number of Spec	Test Method
Bond in Tension Disc	Tensile Adhesion OI#127-3	CHS 0.5 in/ min, Chart 5.0 in/min, 500 lbs full scale load 77°F ± 2°	Discs	TV	Control 6 Special 6	12	A
Bond in Tension Cup	Tensile Adhesion OI#127-3	CHS 0.5 in/ min, Chart 5.0 in/min, 200 lbs full scale load 77°F ± 2°	Cup	TC	Control 6 Special 6	12	A
180° Peel Specimens	Tensile Peel OI#127-3	CHS 10 in/min 77°F ± 2° Chart 5 in/ min 20 lbs full scale load	Peel	TE	Control 6 Special 6	12	B

TEST CONDITIONS

A. Testing of tensile adhesion specimens was performed using an Instron testing instrument. Properties measured were maximum stress to the nearest pound and failure mode.

Steel disc specimens require a stress of about 240 psi. The recommended initial full scale load is 500 pounds. This instrument setting should be changed to another setting if the first reading goes off scale on the high side. If 500 psi is exceeded, then change the reading to 1000 psi full scale.

Cup adhesion specimens are tested with a stress of 200 lbs per sq inch; the recommended full scale load is 500 lbs.

B. Testing of 180° peel samples was performed using an Instron testing instrument. The physical property of the material to be determined was the average peel strength to the nearest pound per inch.

NOTE: Thiokol's procedure for Testing and Laboratory Mixing of UF-2121 Liner. SLP 400, 28 April 71.

STATISTICAL ANALYSIS

UF-2121 liner material is being tested under a ten year program to determine whether or not differences exist between liner materials manufactured from two separate sources of curing agent (MAPO). Test specimens were manufactured in two groups; control, using original source curing agent, and special, using new source curing agent. The test specimens for these two groups are of three kinds; disc, cup, and peel. For each specimen type within each test group the sample test size is six. Laboratory testing for five test periods or five years has been accomplished. Test data for the years 1975, 1976, 1977, 1978 and 1979 are in Tables 2 thru 6 and columns are summarized using means and standard deviations.

To determine any differences in data pertaining to the two MAPO sources, statistical analysis should take into consideration the effect of differing test periods or aging. Two factor analysis of variance was employed to analyze effects pertaining to MAPO source and test period, see Tables 7, 8 and 9. Hypotheses were made that MAPO source has no effect on the data and also that the test period has no effect. If a hypothesis fails, then it is termed significant. Analysis of variance indicates significant test period differences in data pertaining to each of the three specimen types while the MAPO source is only significant in the cup data, see Table 10. A significant statement for interaction is given when the trend from control to special data in one test period is opposite the trend found in another test period.

With five test periods accomplished, regression plots (Figures 1 - 6) were made to determine whether slope differences existed between control and special test data. No differences between slopes were found. The regression model $Y = a + bx$, using individual data points, was employed to evaluate test results. The variance about the least squares trend line is used to compute a tolerance interval such that at the 90% confidence level 90% of the sample distribution falls within this interval. This tolerance interval is extrapolated 24 months past the age point pertaining to the oldest motor tested. The statistical significance of the slope of the trend line is evaluated for each regression plot. If significant, it is an indication that a change over time is occurring.

A statistically significant change in regression does not necessarily mean that the indicated change is significant from an engineering viewpoint.

TEST RESULTS

The 1979 test data and the mean for the respective control and special data is shown in Table 6. In addition, for a convenient comparison, the 1975, 1976, 1977 and 1978 test data are included in Tables 2, 3, 4 and 5 respectively.

A statistical analysis was made on the 1979 data and the two factor analysis of variance are shown in Tables 7, 8 and 9 for the disc, cup and peel specimen data. Significance at the five percent significance level pertaining to analysis of variance tables is shown in Table 10.

DISC:

No significant difference is shown for variance in MAPO source with a statistically significant difference shown for test period and interaction analysis (Table 9). For the year 1979, the mean of the control and special data is 16.78 and 16.86 kg/sq cm respectively.

The minimum specification requirement according to TWR-7857 Rev A is 12.30 kg/sq cm minimum. As seen in Table 5, MANCP's data is well above the minimum.

Regression curves for the control and special specimen data shows a statistically significant decrease (Figures 1 and 2). The slopes of the respective curves show no statistically significant difference.

The failure mode for the control and special specimens was 100% cohesive in the liner.

CUP:

There is a statistically significant difference in MAPO source variance analysis and test period analysis and no statistical significance for

interaction analysis (Table 9).

According to TWR-7857 Rev A Report, the minimum requirement is 4.92 kg/sq cm. The data means are 9.64 kg/sq cm for the control and 9.40 kg/sq cm for the special specimens.

Regression curves for the control and special specimen data shows a statistically significant decrease (Figures 3 and 4). The slopes of the respective curves show no statistically significant difference.

The failure mode for the control specimens for 1979 was 100% cohesive in the propellant. ~~The special~~ specimens were 100% cohesive in the propellant for four specimens, 97% cohesive in the propellant and 3% adhesive propellant to liner for one specimen, and for the remaining specimen 98% cohesive in the propellant with 2% adhesive propellant to liner.

PEEL:

There is no significant difference in MAPO source control and special specimen data. A statistically significant difference is shown for test period and A and B interaction (Table 9).

Thiokol reported (TWR-7857 Rev A) 0.679 and 0.732 *kg/L cm respectively for the control and special specimen mean data at age six months. This compares with 1979 data of 0.830 and 0.780 respectively for control and special mean data.

The regression curves for the control and special data show no statistically significant change (Figures 5 and 6). The slopes of the respective curves show no statistically significant difference.

The mode of failure was 100% liner to propellant bond.

* Kilograms per linear centimeter

CONCLUSIONS

Based on this analysis, there is no significant difference between MAPO sources pertaining to data for disc and peel specimens. There is a statistically significant difference between the control and special MAPO source test data for cup specimens. This may be caused by an increase in the cohesive failure of the propellant as seen in the failure mode.

For the test period analysis, a statistically significant difference is shown in the test data for disc, cup and peel specimens.

The interaction analyses show a statistically significant difference for disc and peel data with no significant difference for the cup data.

In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 Rev A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

RECOMMENDATIONS

It is recommended that the testing plan be continued to assure long range capability of the liner produced from new source material.

TABLE 2
TEST DATA SUMMARY
JULY 1975

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/L-Cm	Nr	Kg/L-Cm
1	17.085	181	17.225	61		241	11.601	121	0.6786	301	0.6786
2	17.507	182	17.858	62	11.812	242	11.601	122	0.6786	302	0.6965
3	17.225	183	17.015	63	11.741	243	11.531	123	0.7143	303	0.6965
4	17.929	184	16.944	64	12.163	244	11.671	124	0.7500	304	0.6965
5	17.366	185	17.436	65	12.234	245	11.390	125	0.7679	305	0.7143
6	17.296	186	19.054	66	11.882	246	11.390	126	0.7858	306	0.6965
$\bar{Y}$	17.401		17.589		11.966		11.531		0.7292		0.6965
S	0.2943		0.7899		0.2191		0.1176		0.0458		0.0113

TABLE 3
TEST DATA SUMMARY
MAY 1976

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/L-Cm	Nr	Kg/L-Cm
7	15.116	187	14.483	67		247	13.288	127	0.9643	307	1.0536
8	16.311	188	14.905	68	13.710	248	13.710	128	0.9286	308	1.0358
9	15.397	189	14.483	69	13.640	249	13.640	129	0.9286	309	1.0179
10	15.960	190	14.765	70	13.007	250	13.077	130	1.0179	310	1.0358
11	15.819	191	15.468	71	13.148	251	13.359	131	1.1072	311	1.0536
12	14.554	192	14.765	72	13.499	252	13.499	132	1.0001	312	1.0358
$\bar{Y}$	15.526		14.812		13.401		13.429		0.9911		1.0388
S	0.6356		0.3633		0.3088		0.2354		0.0675		0.0134

NOTE: Kg/L-Cm = Kilograms per linear centimeter. Also, for the peel test the results are in average peel.

TABLE 4

TEST DATA SUMMARY
APRIL 1977

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/L-Cm	Nr	Kg/L-Cm
13	17.155	193	17.436	73	9.281	253	9.140	133	0.7322	313	0.7858
14	16.522	194	16.874	74	9.281	254	9.070	134	0.7143	314	0.7858
15	16.874	195	16.944	75	8.999	255	8.999	135	0.6786	315	0.7858
16	17.015	196	17.366	76	9.281	256	9.140	136	0.7500	316	0.7143
17	16.874	197	17.015	77	9.492	257	8.367	137	0.7500	317	0.7500
18	16.874	198	17.015	78	9.281	258	8.789	138	0.6429	318	0.7143
$\bar{X}$	16.886		17.108		9.269		8.918		0.7113		0.7560
S	0.2107		0.2337		0.1570		0.2994		0.0429		0.0352

TABLE 5

TEST DATA SUMMARY
JUNE 1978

DISC				CUP				PEEL			
Control		Special		Control		Special		Control		Special	
Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/Cm ²	Nr	Kg/L-Cm	Nr	Kg/L-Cm
19	14.906	199	14.554	79	10.898	259	10.406	139	0.9109	319	1.0180
20	14.624	200	14.695	80	10.968	260	10.476	140	0.9823	320	1.0359
21	14.695	201	14.343	81	10.617	261	10.616	141	0.9466	321	1.0180
22	14.906	202	14.343	82	10.125	262	10.125	142	1.0002	322	1.0359
23	14.343	203	14.624	83	10.406	263	10.687	143	1.0716	324	1.0537
24	15.187	204	14.414	84	10.476	264	10.125	144	0.9466	326	0.9287
$\bar{X}$	14.777		14.496		10.582		10.406		0.9764		1.0150
S	0.2898		0.1503		0.3166		0.2391		0.0561		0.0444

TEST DATA SUMMARY
11 June 1979

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TABLE 7

TWO FACTOR ANALYSIS OF VARIANCE
FOR
DISC DATA, FACTOR A = MAPD SOURCE (CONTROL AND SPECIAL), FACTOR B = TEST PERIOD

SOURCE	DF	SS	MS	F
FACTOR A	1	0.15251017E+00	0.15251017E+00	0.87337160E+00
FACTOR B	4	1.74451229E+02	0.18612807E+02	0.10650893E+03
A X B INTERACTION	4	0.18911092E+01	0.47282229E+00	0.27076855E+01
ERROR	50	0.87311143E+01	0.17462229E+00	
TOTAL	55	1.85226141E+02		

TABLE 8

TWO FACTOR ANALYSIS OF VARIANCE
FOR
CUP DATA, FACTOR A = HAPD SOURCE (CONTROL AND SPECIAL), FACTOR B = TEST PERIOD

SOURCE	DF	SS	MS	F
FACTOR A	1	0.699977E+00	0.699977E+00	0.12650295E+02
FACTOR B	4	0.1255665E+03	0.31417E+02	0.5677041E+00
A X B INTERACTION	4	0.4305757E+00	0.1076439E+00	0.1945367E+01
ERROR	40	0.2213315E+01	0.5532867E-01	
TOTAL	49	0.1250124E+03		

TABLE 9

TWO FACTOR ANALYSIS OF VARIANCE
FOR
PEEL DATA, FACTOR A = MAPO SOURCE (CONTROL AND SPECIAL), FACTOR B = TEST PERIOD

SOURCE	DF	SS	MS	F
FACTOR A	1	0.1361360E-02	0.1361360E-02	0.5385081E+00
FACTOR B	4	0.9951702E+00	0.2482925E+00	0.9821612E+02
A X B INTERACTION	4	0.2661336E-01	0.6703341E-02	0.2651615E+01
ERROR	50	0.1264010E+00	0.2528021E-02	
TOTAL	55	0.1147746E+01		

TABLE 10

SIGNIFICANCES AT THE FIVE PERCENT SIGNIFICANCE LEVEL
PERTAINING TO ANALYSIS OF VARIANCE TABLES 7, 8 and 9

<u>Test</u>	<u>MAPO Source (A)</u>	<u>Test Period (B)</u>	<u>A x B Interaction</u>
Disc	NS	S	S
Cup	S	S	NS
Peel	NS	S	S

NS = Not significant

S = Significant

$Y = (17.023727) + (-0.016447) * X$
 F = 2.183369
 R = -0.268955
 C = 1.477623
 N = 30
 STORAGE COND= AMBIENT
 TEST COND = 77 DEG F AMB RH
 UF-2121 LINER
 SIGNIFICANCE OF F = NOT SIGNIFICANT
 SIGNIFICANCE OF R = NOT SIGNIFICANT
 SIGNIFICANCE OF C = NOT SIGNIFICANT
 DEGREES OF FREEDOM= 28
 OY = 1.046818
 SB = 0.011131
 SF = 1.026092

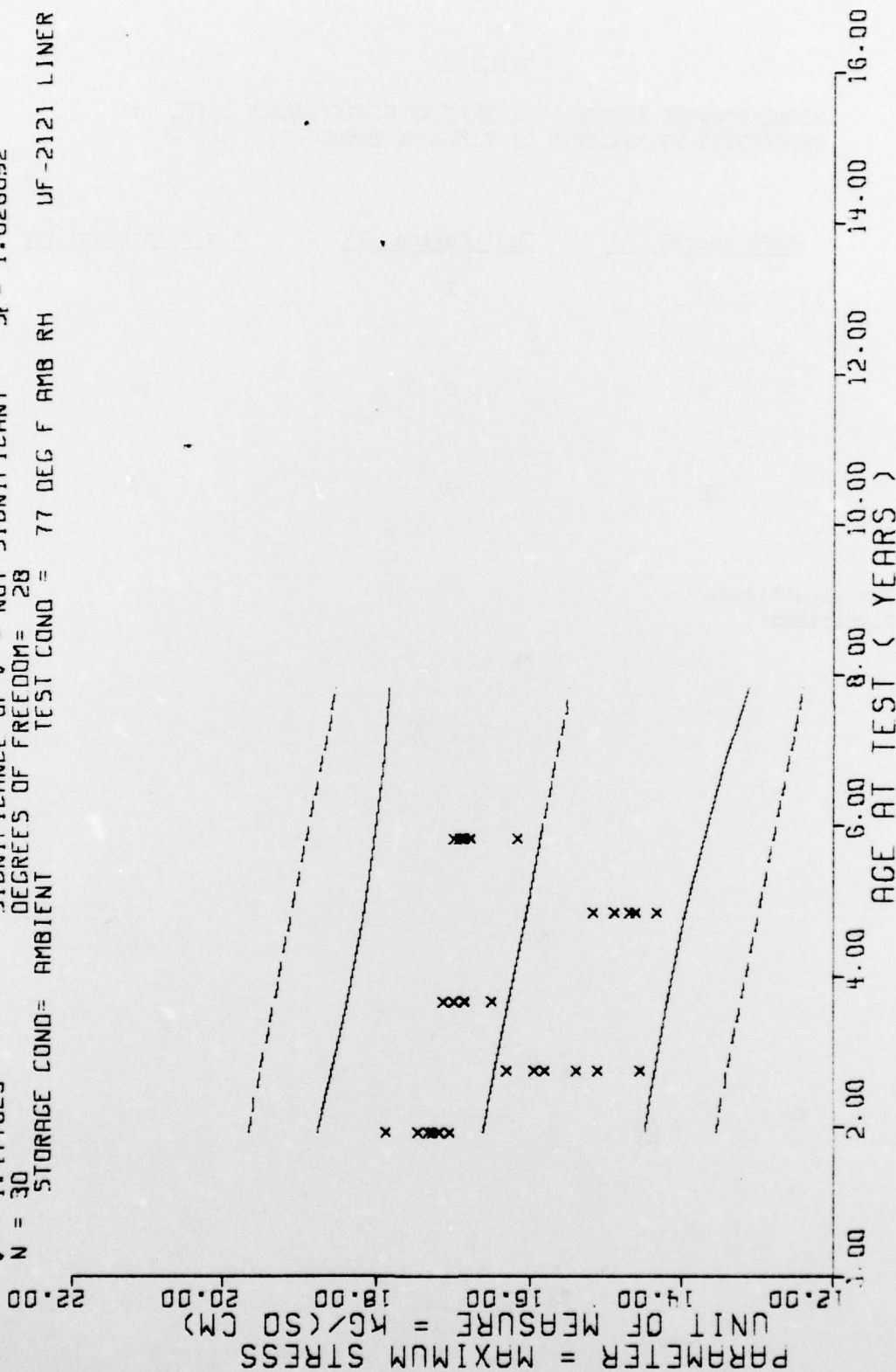
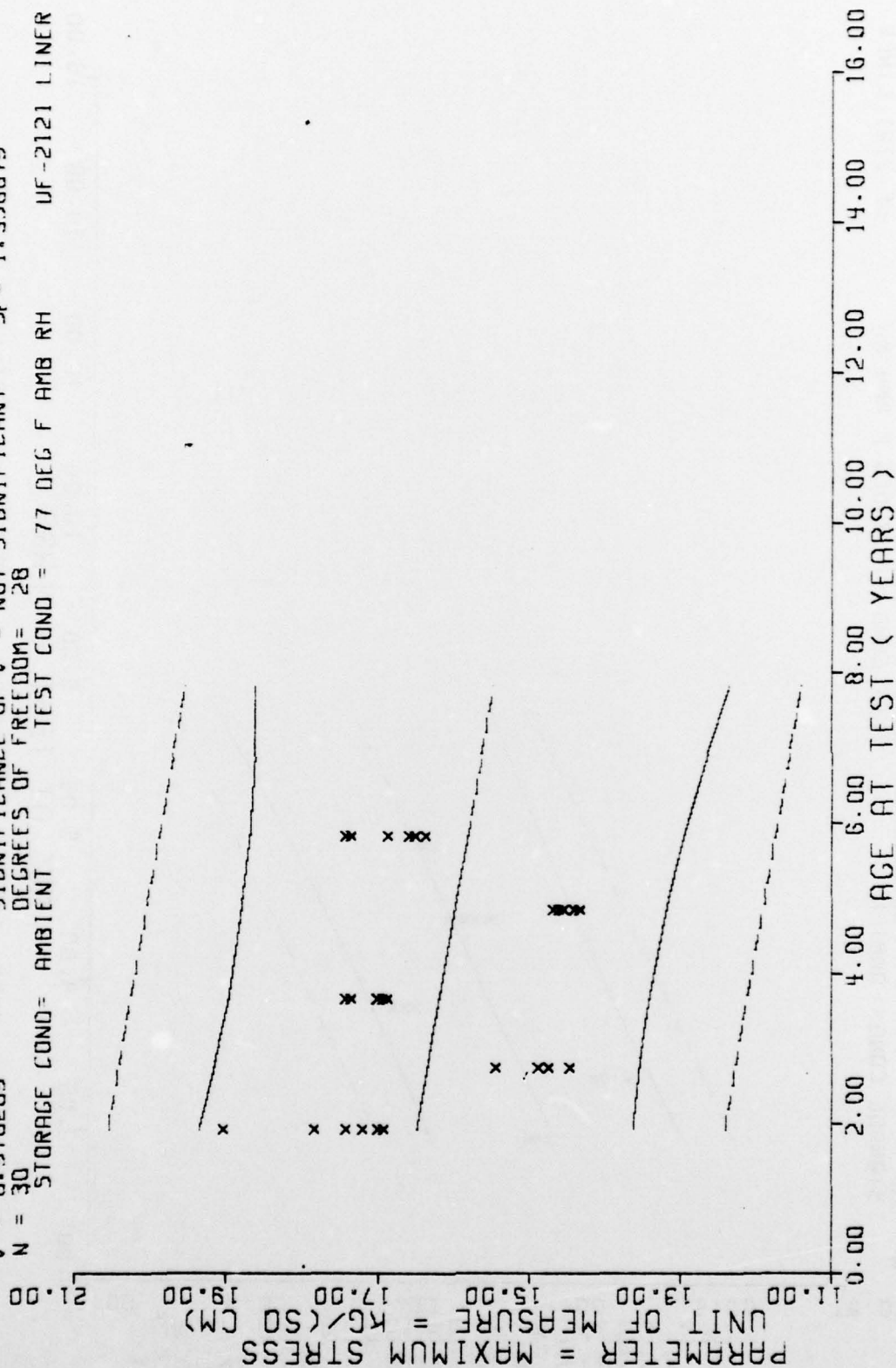


Figure 1

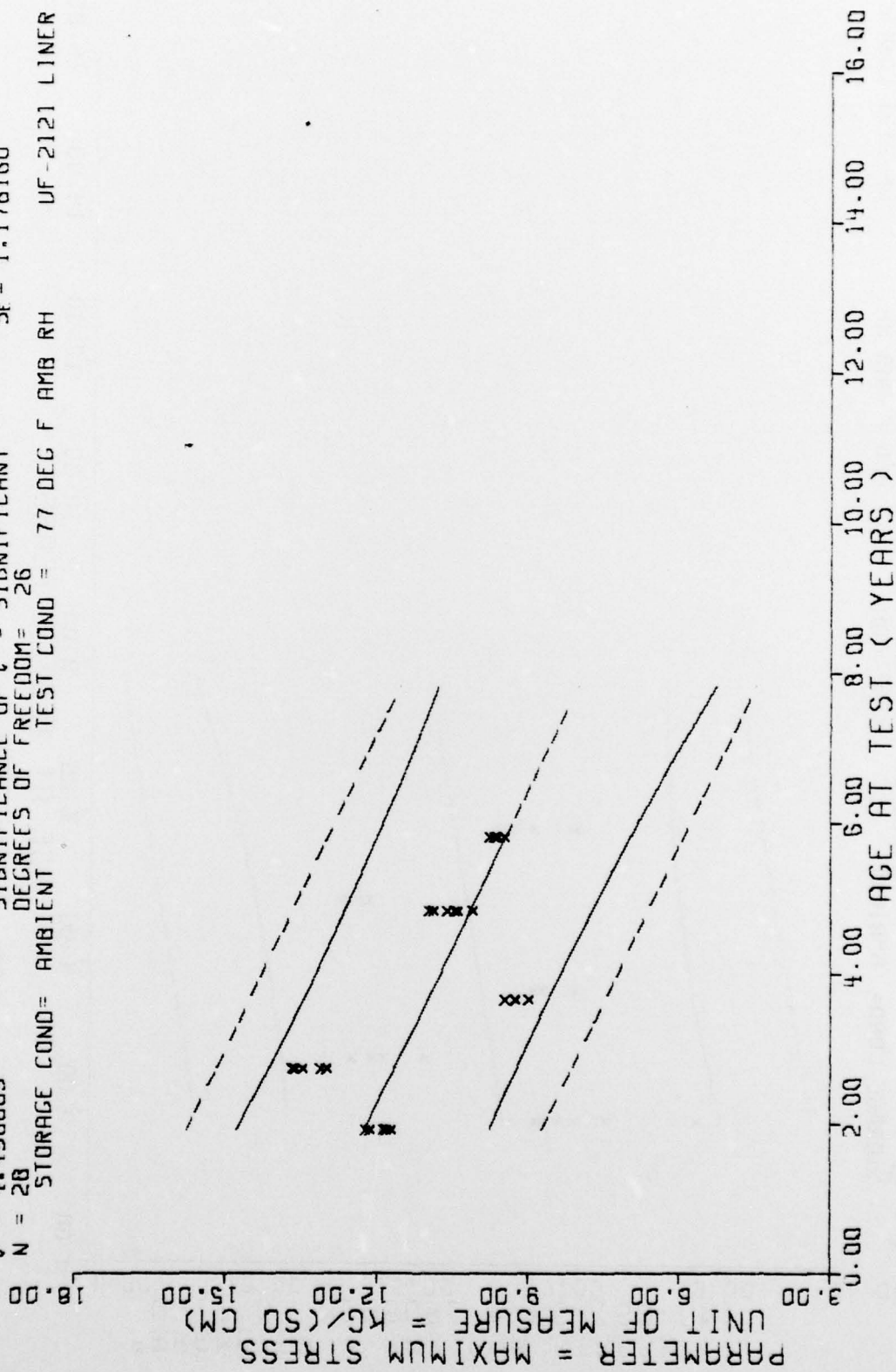
$F = 0.956893$
 $R = -0.181784$
 $\chi^2 = 0.978209$
 $N = 30$
 STORAGE COND = AMBIENT
 $Y = (16.829414) + (-0.014396) * X$
 SIGNIFICANCE OF F = NOT SIGNIFICANT
 SIGNIFICANCE OF R = NOT SIGNIFICANT
 SIGNIFICANCE OF χ^2 = NOT SIGNIFICANT
 DEGREES OF FREEDOM = 28
 TEST COND = 77 DEG F AMB RH
 UF-2121 LINER



MAPQ. SPECIAL DISC (STEEL/LINER/STEEL) TEST NR 5

Figure 2

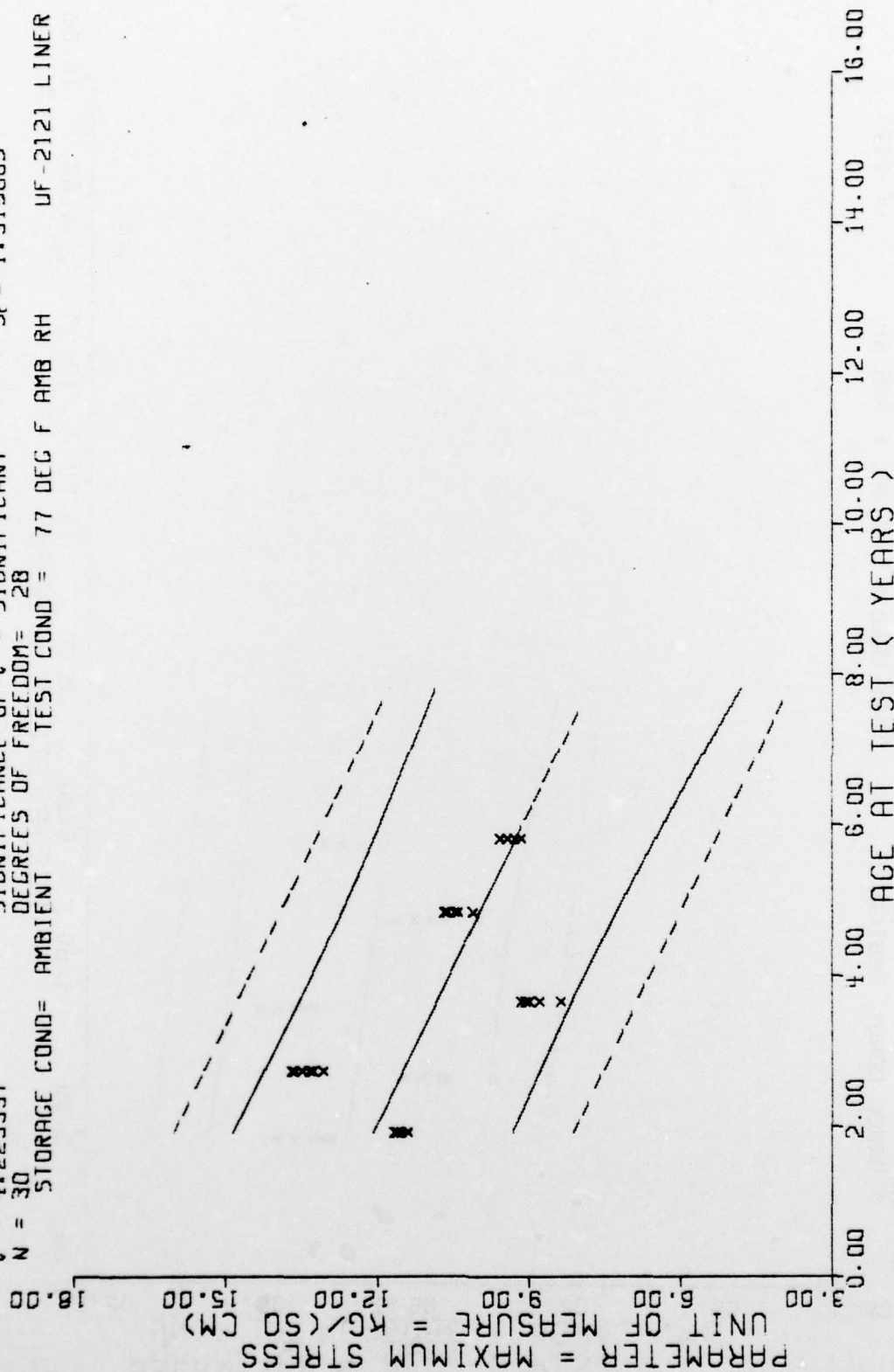
F = 20.222015
 R = -0.661436
 U = 4.496689
 N = 28
 STORAGE COND = AMBIENT
 TEST COND = 77 DEG F AMB RH
 UF-2121 LINER
 $Y = (13.662364) + (-0.060044) * X$
 SIGNIFICANCE OF F = SIGNIFICANT
 SIGNIFICANCE OF R = SIGNIFICANT
 SIGNIFICANCE OF U = SIGNIFICANT
 DEGREES OF FREEDOM = 26
 OY = 1.541511
 SB = 0.013352
 SE = 1.178160



MAPQ. CONTROL CUP (STEEL/LINER/STEEL) TEST NR 5

Figure 3

F = 17.836578
 R = -0.623806
 U = 4.223337
 N = 30
 STORAGE COND= AMBIENT
 TEST COND = 77 DEG F AMB RH
 UF-2121 LINER
 Y = (13.490608) + (-0.060432) * X
 SIGNIFICANCE OF F = SIGNIFICANT
 SIGNIFICANCE OF R = SIGNIFICANT
 SIGNIFICANCE OF U = SIGNIFICANT
 DEGREES OF FREEDOM= 28
 Qr = 1.658339
 Sr = 0.014309
 Sf = 1.319065



MAPQ. SPECIAL CUP (STEEL/LINER/STEEL) TEST NR 5

Figure 4

	Y = (0.774814) + (0.001599) * X		
R = 1.311509	SIGNIFICANCE OF F = NOT SIGNIFICANT	OY = 0.129379	
F = 0.211527	SIGNIFICANCE OF R = NOT SIGNIFICANT	SB = 0.001396	
t = 1.145211	SIGNIFICANCE OF t = NOT SIGNIFICANT	SE = 0.128690	
N = 30	DEGREES OF FREEDOM = 28		
STORAGE COND= AMBIENT	TEST COND = 77 DEG F AMB RH		UF-2121

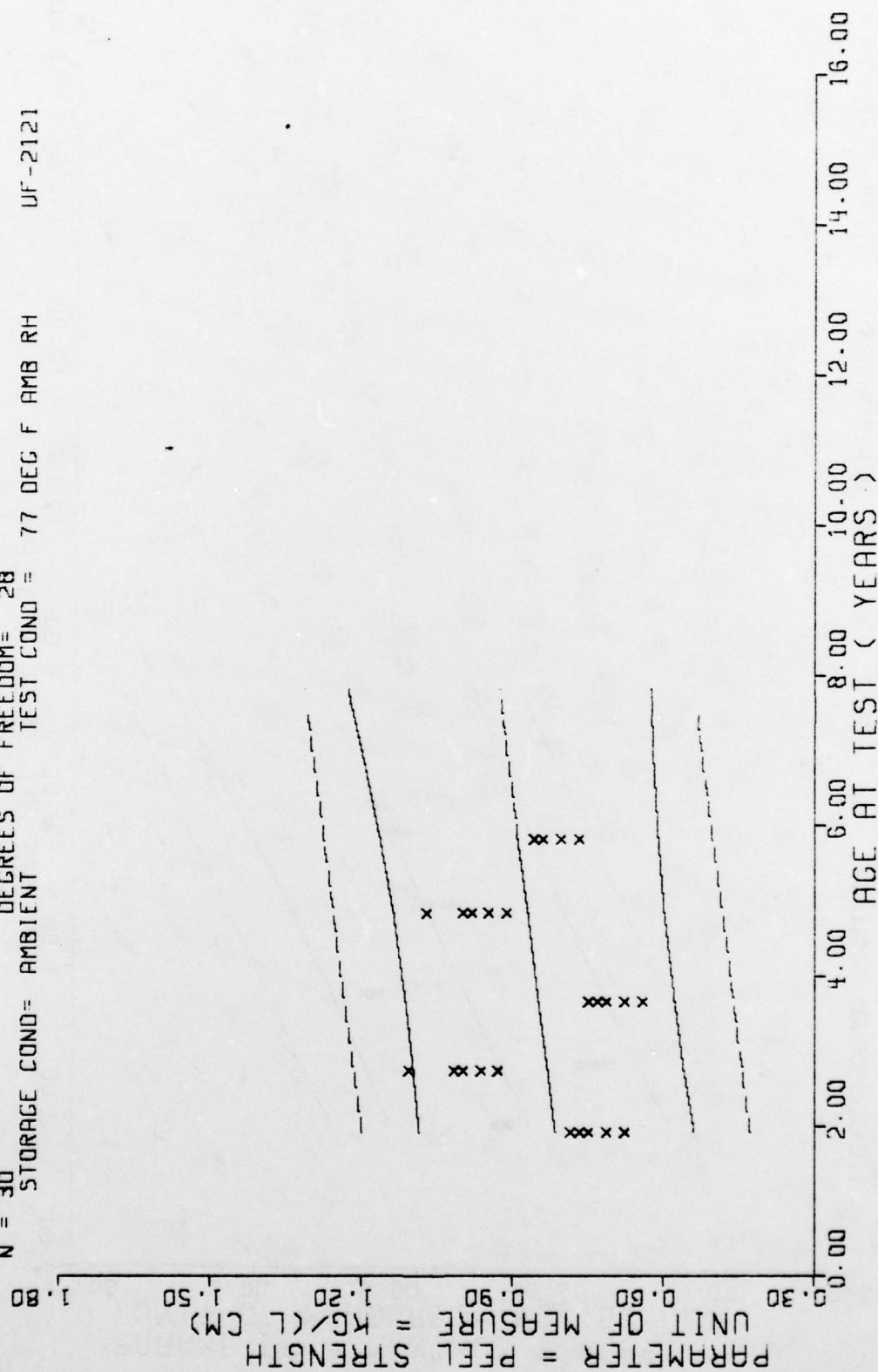
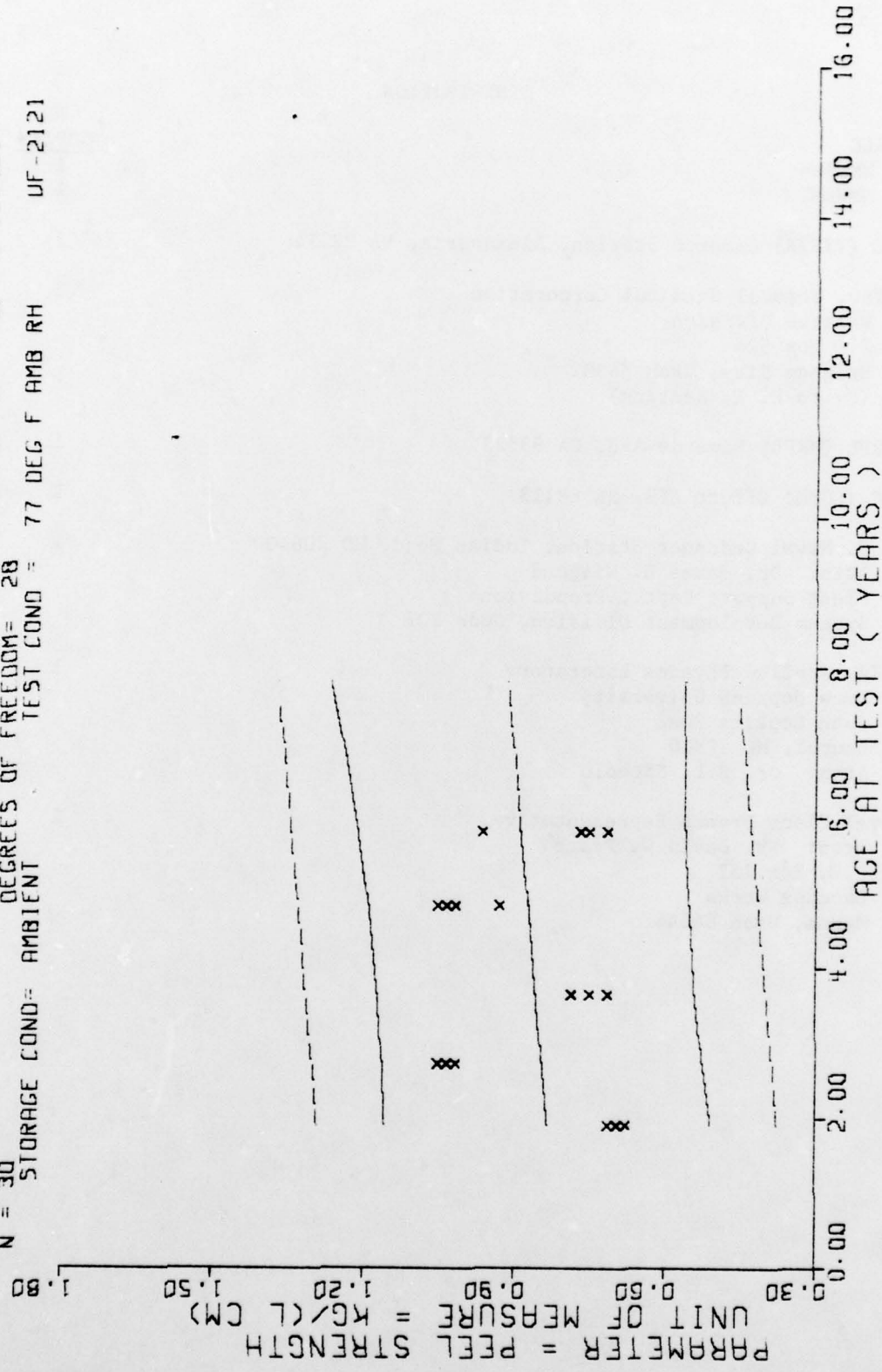


Figure 5

$Y = (0.806282) + (0.001117) * X$
 F = 0.456752 SIGNIFICANCE OF F = NOT SIGNIFICANT $Q_1 = 0.150970$
 R = 0.126691 SIGNIFICANCE OF R = NOT SIGNIFICANT $S_B = 0.001653$
 L = 0.675834 SIGNIFICANCE OF L = NOT SIGNIFICANT $S_F = 0.152404$
 N = 30 DEGREES OF FREEDOM = 28
 STORAGE COND = AMBIENT TEST COND = 77 DEG F AMB RH UF-2121



MAPQ. SPECIAL PEEL (STEEL/LINER/STEEL) TEST NR 5

Figure 6

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18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Minuteman MAPO UF-2121 Liner		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Thiokol Chemical Corporation/Wasatch Division uses MAPO in the production of UF-2121 liner. Thiokol changed MAPO vendors and, therefore, qualification of the new source MAPO was required. Thiokol prepared specimens from the new source and also specimens from the original source which are to be used as the control material in the 10 year surveillance testing program. The specimens were transferred to Ogden ALC for testing and reporting of the data obtained.		

✓ This report includes the test results for the first, second, third, fourth, and fifth time testing of the control and special specimens at Ogden ALC. In all instances, the mean data for the control and special specimens are well above the minimum requirements found in TWR-7857 A, Thiokol specimen data. Therefore, the capability of the liner from the new source material is expected to perform satisfactorily.

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