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A STUDY AND EVALUATION OF KEL-F ELASTOMER

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FOREWORD

This report was prepared by the Organic Materials Branch and was initiated under Project No. 7340, "Rubber Plastic and Composite Materials," Task No. 73405, "Compounding of Elastomers," formerly RDO No. 617-12, "Compounding of Elastomers," and was initiated under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. R. E. Headrick acting as project engineer.

Acknowledgement is made to The M. W. Kellogg Company for their "First Report on A New Fluorocarbon Rubber" and "Supplement Number 1" to this report which supplied a starting point for the subsequent work covered herein.

Many of the compounds tested were not developed or intended by the manufacturers for the conditions to which they have been subjected. Any failure or poor performance of a material is therefore not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

This report covers work conducted from August 1954 to January 1955.

ABSTRACT

The bulk of the work reported herein is a summary of experimental compounding while trying to compound for low compression set and good chemical resistance.

Initial compounding of Kel-F Elastomer revealed the most promising compounds for low compression set and chemical resistance were those cured with benzoyl peroxide. The compound having the lowest set in this effort was 266-62-1. This compound has a set of 40 percent when compressed 30 percent at 250°F for 70 hours.

Immersion tests in experimental hydraulic fluids composed of silicate esters or "silicone oils" indicate that Kel-F Elastomer may prove useful for aircraft hydraulic system applications up to 400°F. Tests also indicate that this elastomer when properly compounded has exceptional resistance to potential rocket fuels such as fuming nitric acid and may prove useful for hose, seals, protective clothing and other items for contact with these fluids.

Of particular importance to the rubber compounder is the discovery that prolonged milling is required during compounding to obtain uniform physical properties from the compounded elastomer. Variation in milling time can change final physical properties of the cured elastomer as much as 100 percent, at least on the polymer produced to date.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
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INTRODUCTION

Chemical resistant elastomers have been the subject of much investigation during the past few years. The problem of finding elastomers that will withstand the damaging effects of corrosive chemicals, hydraulic fluids and lubricants has been and is of much concern to the Air Force, aircraft and lubricant manufacturers, and a host of other interested agencies. The problem of rubber seals that will withstand higher operating temperatures is becoming increasingly important. Occasionally a new elastomer is developed that shows promise of solving some of these problems. This report concerns such a material, Kel-F Elastomer, developed by the M. W. Kellogg Company from work originally sponsored by the Office of the Quartermaster General US Army. It is reported to be a fluorocarbon material which contains more than 50% fluorine by weight.

SECTION I

MILLING AND TESTING PROCEDURES

The compounding was done on a standard 3" x 12" two roll rubber mill. For the initial breakdown no heat is needed nor is cooling water needed. Kel-F Elastomer mills very easily.

The fluids used in the immersion tests were oils, conforming to MIL-L-7808 a diester type, MLO-8200 and Monsanto's OS-45 silicate ester type fluids and a simulated fuel mixture of 70 percent isooctane and 30 percent toluene by volume, conforming to Specification MIL-H-3136.

The brittle point tests were run in accordance with ASTM Specification D-746-52 T, except the thickness of the test specimen used for this test was .040 inches.

The temperature retraction tests were run according to the method described in ASTM Specification D-599-40 T. The T. R. curves were drawn for retraction from 0 to 60 percent.

All compounds were molded in a "Preco" electrically heated press at approximately 20,000 pounds ram pressure. A 2" x 3" x .040" mold was used for all sample sheets.

The compression set tests were run according to ASTM Specification D-395-52 T, Method B, except the temperature was 250°F unless otherwise noted.

Permeability tests were conducted in an H tube with the Kel-F Elastomer membrane between the two halves of the H tube. The membranes were from 8 to 10 mils thick. Water was placed on one side of the H tube and RFNA (15% oxides) on the other. At various time intervals the pH of the water side was recorded. The grams of RFNA which passed to the water side were calculated from the pH change.

SECTION II

COMPOUNDING AND VULCANIZATION

Compounding of Kel-F Elastomer was accomplished using benzoyl peroxide, triethylene tetramine (TETA) and methylene bis (4 phenyl isocyanate) (MDI) as the curing agents. (See Tables 1, 2, and 7) The peroxide cures produced compounds having the best compression set, tensile strength, elongation, and heat aging characteristics. Table 5 indicates the ratio of benzoyl peroxide used does not have very much effect on the final properties; however, in thick sections 3 phr of benzoyl peroxide may cause blowing unless the compound is very well milled; 1 1/2 phr is recommended for thick sections. The benzoyl peroxide used for this evaluation was C. P. granular with 10 percent water added. Paste type benzoyl peroxide was tried in several compounds witho t success. (See Table 6)

The stabilizer and activator used were dibasic lead phosphate (Dyphos) and zinc oxide, respectively. Each of these was reduced to 3 phr without a harmful effect on the aging properties. The gum may be cured by using benzoyl peroxide alone, but the milling must be very thorough until the benzoyl peroxide is very finely dispersed. (See Tables 6 and 12)

Several white fillers were found to have a reinforcing effect; however, the silica fillers, besides being the most chemical resistant, also produced compounds with the highest tensile strength. Tensile strengths of 5000 psi have been recorded for silica filled compounds. (See Tables 8 and 9)

Carbon black was used as a filler for isocyanate cures but the reinforcing effect was very poor. (See Table 10)

Thorough milling is a must. Compound 266-62-1 was cured after varying periods of milling. The tensile strength increased 300 percent by extending the milling time 20 minutes beyond what appeared to be a well dispersed compound. (See Table 4)

Post curing by oven aging at 300°F of compound 266-33-1 increased the tensile strength 230 percent and increased the elongation 40 percent. The original press cure for this compound was at 300°F for 60 minutes. Actually the original press curing temperature is not important to obtain the optimum physical properties, but the post cure is. The temperature and period of the post cure can be adjusted with the press cure to give the optimum properties within limits. The lower the press cure temperature, the better the mold flow.

A cure temperature of 250°F produces a well formed molding. Post curing is also recommended for isocyanate cured compounds. (See Table 3 and Figure 1)

SECTION III

COMPOUNDING FOR LOW COMPRESSION SET

The best compression set for the compounds in Tables 1, 2, 3 and 4 is 51 percent for a peroxide cure, the lower loading levels having the lower sets. Compound 266-62-1 with all the filler removed and the activator and stabilizer reduced to a minimum had a compression set of 43.5 percent when run at 250°F and 40 percent set when run at 212°F. The compression set of amine and isocyanate cures was very poor, nearly all specimens crushed in the jig. (See Table 7)

Mercurous oxide when added in an attempt to improve the set had little effect.

T-butyl peroxide, sometimes used to cure silicone compounds for low set, was used to replace benzoyl peroxide in compound 266-62-1. The set was 59 percent as compared to 43.5 percent for the benzoyl peroxide cure. (See Table 13)

"O" rings fabricated from Kel-F Elastomer have been subjected to a modified crush test required by current Hydraulic "O" ring Spec. MIL-P-5516A. The test consisted of giving the "O" ring a half twist and subjecting the distorted "O" ring to a compression of 10,000 psi for 30 seconds in a 250°F press. The "O" ring did not fail in crush but did in plastic flow. The rings tested were unaged rings but rings aged for 24 hours at 300°F also failed in plastic flow. Broken pieces of a dumbbell which had been aged for one week in an oven at 300°F were given a similar crush test. The results were the same, plastic flow. So it appears that even at the highest state of cure there is not enough cross linkage to prevent plastic flow when using benzoyl peroxide as the curing agent. At least this may account for the poor compression set.

SECTION IV

FLUID IMMERSION EVALUATION

The fluids used for these evaluations are those of current interest to the Air Force. The tests as run were as follows:

(a) 70 hours immersion in MLO-8200 experimental hydraulic oil at 400°F and three hours at 550°F.

(b) 24 to 168 hours immersion in OS-45 experimental hydraulic oil at 400°F.

(c) 70 hours immersion in MIL-L-7808. A jet engine oil at 350°F.

(d) 70 hours immersion in 70-30 isooctane-toluene fuel at room temperature.

The results of the tests run in MLO-8200 oil were promising. The higher loaded compounds containing Hi Sil, Hi Sil x 303, and Hi Sil LM-3 (silicone oil coated) fillers faired very well, losing little of their original physical properties. The best result obtained was with a Hi Sil x 303 loaded compound cured with benzoyl peroxide, compound 266-73-1.

Compound 266-73-3 plasticized with a monochlorotrifluoroethylene oil aged very well in MLO-8200 fluid. The amine cured compound 266-63-1 when aged in MLO-8200 fluid at 400°F became very brittle and the amine-isocyanate cured compound 266-63-2 was only fair with slight cracking. (See Table 14)

Two compounds, a gum stock and a loaded compound, were immersed in MLO-8200 fluid for three hours at 550°F. The effect of the high temperature was very detrimental to both compounds.

Compound 266-97-1 was aged at 400°F in OS-45 fluid from 24 to 168 hours. At 168 hours the specimens still retained 1300 psi tensile strength, 240 percent elongation, volume change of +12 percent, and a Shore A hardness of 67. The surface condition of the specimen was excellent. (See Figure 2)

Immersion tests in a MIL-L-7808 type fluid showed that the diester type fluids are very damaging to Kel-F Elastomer compounds. Volume swells were about 100 percent and the retained tensile strength was only 30 percent of the original after 70 hours at 350°F. All samples became very sticky and had apparently started to dissolve. (See Table 14)

The 70-30 fuel immersion test of the benzoyl peroxide cured compounds resulted in a volume swell range from 23 percent to 30 percent and a retained tensile strength of about 50 percent or better of the original after 70 hours aging at room temperature. Compound 266-97-1 aged for 168 hours at room temperature had a volume swell of 25 percent and a tensile strength of 1560 psi, 38 percent of the original.

SECTION V

NITRIC ACID RESISTANCE

Compounds of Kel-F Elastomer after 70 hours immersion in RFNA (15% NO_2) had an approximate volume swell of 25 percent and 70 percent loss of tensile strength. Compound 266-73-2 containing Hi Sil LM-3 filler produced the best results. The appearance of these compounds after the immersion was excellent. Better results might have been obtained if larger, standard size dumbbell test specimens had been used. The dumbbell test specimens used for all the immersion tests were .040" thick and 1/8" wide.

Buna N compounds will completely disintegrate in this acid in two hours. A typical butyl compound immersed in the above acid for two hours was severely attacked.

Addition of Kel-F resin products to the elastomer shows some sign of improving the acid resistance. Also the acid permeability of these compounds is very much lowered by adding Kel-F resin products. Contrary to the immersion results silica filled compounds have very poor permeability resistance. (See Table 18 and Figure 4)

SECTION VI

LOW TEMPERATURE PROPERTIES

Brittle points were run on several compounds ranging from a gum stock to a well loaded compound. The brittle points ranged between -58°F to -43°F. The gum stock had the lowest brittle point, -58°F. (See Table 16)

Temperature - Retraction tests were conducted and the data were plotted for three compounds. The extrapolated freezing points were about +26°F. The gum stock, as in the brittle point test, had the lowest freezing point. (See Table 16 and Figure 3)

No compounding attempts were made to improve the low temperature properties. None of the compounds tested were plasticized.

SECTION VII

CONCLUSIONS

The optimum properties of Kel-F Elastomer are obtained by using benzoyl peroxide as the curing agent. The peroxide cure has the greater chemical resistance, better heat stability, higher physical properties including lower compression set, than the other types of cures tested.

Compounded Kel-F Elastomer has excellent resistance to RFNA, superior to any elastomer tested at WADC to date. The physical properties deteriorate gradually; however, the surface condition remains very good. The volume swell is not excessive. These compounds when properly fabricated should be useful where resistance to RFNA is important.

The results of the immersion tests in silicate ester type fluids indicate Kel-F Elastomer should be useful in these fluids up to 400 °F.

The brittle point of Kel-F Elastomer is approximately -50 °F which is fairly good but the T. R. curve indicates that at 32 °F the rubber like characteristics begin to disappear rapidly.

Results of tests conducted at room temperature in 70-30 type fuel were very satisfactory and because Kel-F Elastomer has excellent high temperature stability its use in contact with fuels at higher temperature may be possible.

APPENDIX I

TABLES AND FIGURES

TABLE I

BASIC COMPOUNDS

	257-41-2	257-41-3	257-42-1	266-33-1
KEL-F ELASTOMER	100	100	100	100
BENZOYL PEROXIDE		3	3	3
MDI	5			
DYPHOS		10	10	10
ZINC OXIDE		10	10	10
HI SIL	20	20		
HI SIL x 303			15	
60 MINUTES PRESS CURE AT 300°F				
100% MODULUS (psi)	739	518	393	187
TENSILE STRENGTH (psi)	1120	2420	2790	1365
% ELONGATION	1100	515	485	520
% PERMANENT SET AT BREAK	100	26	20	11
SHORE A HARDNESS	80	74	76	63
% COMPRESSION SET (70 HRS. AT 250°F)	100	76	90	90
OVEN AGED 24 HRS. AT 300°F				
MODULUS	747	594	544	256
TENSILE STRENGTH	1835	3360	4215	1975
ELONGATION	590	510	455	510
PERMANENT SET	16	23	14	7
HARDNESS	83	76	74	64
% COMPRESSION SET	88	80	70	67

TABLE II

EXPERIMENTAL VULCANIZING RECIPES

	266-7-1	2	3	4	5	6	7	8
KEL-F ELASTOMER	100	100	100	100	100	100	100	100
FINE SILICA	15	15						
MDI	5							
BENZOYL PEROXIDE		3						1.5
ZINC OXIDE			10	10	10	10	10	10
"808"			1					
METHYL ZIMATE				1				
METHYL TUADS					1			
N-22						1		
DPG							1	
DYPHOS								10
HI SIL								20

60 MINUTE PRESS CURE AT 300°F

	266-7-1	2	3	4	5	6	7	8
100% MODULUS (psi)	NO CURE	340	NO CURE	NO CURE	NO CURE	NO CURE	NO CURE	475
TENSILE STRENGTH (psi)		980						1135
% ELONGATION		560						825
% PERMANENT SET AT BREAK		32						52
SHORE A HARDNESS		77						79

TABLE III

EFFECT OF OVEN AGING AT 300°F

	266-33-1
KEL-F ELASTOMER	100
ZINC OXIDE	10
DYPHOS	10
BENZOYL PEROXIDE	3

60 MINUTES PRESS CURE AT 300°F

	Original Properties	Oven Aging		
		24 hrs.	72 hrs.	168 hrs.
100% MODULUS (psi)	245	255	280	235
TENSILE STRENGTH (psi)	1290	1810	2170	2940
% ELONGATION	440	470	510	630
% PERMANENT SET AT BREAK	8	7	5	11
SHORE A HARDNESS	67	72	72	
% COMPRESSION SET (70 HOURS AT 250°F)	90	68	66	64

TABLE IV

EFFECT OF MILLING TIME

	266-62-1
KEL-F ELASTOMER	100
ZINC OXIDE	3
DYPHOS	3
BENZOYL PEROXIDE	3

All compounds cured 60 minutes at 260°F - oven cured 20 hours at 300°F.

	Tensile Strength (psi)	% Elongation
All ingredients milled in and several passes made through a tight mill.	780	280
Five extra minutes milling on a tight mill	1370	460
Fifteen extra minutes milling on a tight mill	2175	460

TABLE V

EFFECT OF BENZOYL PEROXIDE

	266-5-1	266-5-2	266-5-3	266-5-4	266-5-5
KEL-F ELASTOMER	100	100	100	100	100
DYPHOS	10	10	10	10	5
ZINC OXIDE	10	10	10	10	5
BENZOYL PEROXIDE	2	4	5		3
CUMENE HYDROPEROXIDE				3	
Cures 60 minutes at 300°F 24 Hrs. Oven Cure at 300°F.					
100% MODULUS (psi)	220	255	255	DID NOT CURE	235
TENSILE STRENGTH (psi)	1690	1395	1990		1390
% ELONGATION	500	455	460		550
% PERMANENT SET	13	10	9		12
SHORE A HARDNESS	67	69	69		63
% COMPRESSION SET AT 250°F	55	56	60		51

TABLE VI

BENZOYL PEROXIDE CURES WITHOUT ACTIVATORS

	266-18-1	2	3	4	5	6	7
KEL-F ELASTOMER	100	100	100	100	100	100	100
BENZOYL PEROXIDE	3	4	5				2
TETA				4			
LUPERCO AGE (50%)					1	2	
100% MODULUS		185	147				
TENSILE STRENGTH (psi)	630	748	619	1340	212	219	633
% ELONGATION		640	530	520			
% PERMANENT SET AT BREAK		7	7	10			
SHORE A HARDNESS	68	68	67	66			

Note - Compounds 1, 2, 3, 5, 6 and 7 press cured 60 minutes at 300°F plus a 24 hour oven cure at 300°F. Compound 4 press cured 60 minutes at 260°F plus a 3 hour oven cure at 300°F.

TABLE VII

TETRAMINE AND ISOCYANATE CURES

266-17-	1	2	3	4
KEL-F ELASTOMER	100	100	100	100
TETA	1.5	1.5	1.5	1.5
ZINC OXIDE	5	5	5	5
HI SIL x 303		20		20
MDI			5	5
100% MODULUS (psi)	198	816	245	807
TENSILE STRENGTH (psi)	1633	2025	1803	1505
% ELONGATION	782	650	610	250
% PERMANENT SET AT BREAK	12		8	10
SHORE A HARDNESS	67	90	72	89
% COMPRESSION SET (72 hrs. at 250°F)	93	98	99	95

Note - Press Cures 60 minutes at 300°F - Oven Cures 24 hours at 300°F.

TABLE VIII

EFFECT OF VARIOUS FILLERS ON BENZOYL PEROXIDE CURES

266-9-	1	2	3	4	5
KEL-F ELASTOMER	100	100	100	100	100
ZINC OXIDE	10	10	10	10	10
DYPHOS	10	10	10	10	10
BENZOYL PEROXIDE	3	3	3	3	3
HI SIL x 303	20				
SILENE SF		20			
MAGNESIUM CARBONATE			20		
KALVAN				20	
ELC MAGNESIA					20
60 MINUTES PRESS CURE AT 300°F AND 24 HRS. OVEN AGING AT 300°F.					
100% MODULUS (psi)	536	506	535	312	496
TENSILE STRENGTH (psi)	2695	1780	1745	1447	1745
% ELONGATION	460	564	488	448	604
% PERMANENT SET AT BREAK	18	22	14	13	30
SHORE A HARDNESS	87	80	74	74	77
% COMPRESSION SET (70 hrs. at 250°F)	73	82	78	80	72

TABLE VIII EFFECT OF VARIOUS FILLERS ON BENZOYL PEROXIDE CURES (cont'd)

168 HOURS OVEN AGING AT 300°F

266-9-	1	2	3	4	5
100% MODULUS (psi)	719	546	721	489	555
TENSILE STRENGTH (psi)	3760	2675	2607	3210	3380
% ELONGATION	395	446	434	463	530
% PERMANENT SET AT BREAK	10	21	16	15	22

TABLE IX

EFFECT OF VARIOUS FILLERS ON ISOCYANATE CURES

266-13-	1	2	3	4	5	6
KEL-F ELASTOMER	100	100	100	100	100	100
MDI	5	5	5	5	5	5
HI SIL x 303		20				
SILENE EF			20			
MAGNESIUM CARBONATE				20		
KALVAN					20	
ELC MAGNESIA						20
PURE CURE 60 MINUTES AT 300°F - 24 HRS. OVEN CURE AT 300°F						
100% MODULUS (psi)		797	793		506	588
TENSILE STRENGTH (psi)		1850	1495		1847	1950
% ELONGATION		438	525		500	794
% PERMANENT SET AT BREAK	NO CURE	28	25		21	61
SHORE A HARDNESS		84	82	POOR CURE	77	78
% COMPRESSION SET (70 hours at 250°F)		100	90		100	100
70 HOURS OVEN CURE AT 300°F						
100% MODULUS (psi)		647	606	558	424	620
TENSILE STRENGTH (psi)		1205	1090	990	1100	1490
ELONGATION		730	755	700	752	647

TABLE IX EFFECT OF VARIOUS FILLERS ON ISOCYANATE CURES (cont'd)

	266-13-1	2	3	4	5	6
% PERMANENT SET AT BREAK	NO CURE	65	75	20	38	39
SHORE A HARDNESS		84	81	76	76	76
% COMPRESSION SET (168 Hrs. at 250°F)		84.2	84.8	93.8	88.3	85.8

TABLE X

CARBON BLACK FILLED COMPOUNDS

266-15-	1	2	3	4	5	6	7	8	9	10
KEL-F ELASTOMER	100	100	100	100	100	100	100	100	100	100
MDI	5	8	10	5	8	10	5	10	5	5
F.T. BLACK	10	10	10	20	20	20			10	20
ZINC OXIDE							5	5	5	5
PRESS CURE 60 MINUTES AT 300°F - OVEN CURE 24 HRS. AT 300°F										
100% MODULUS (psi)	540	479	445	534	654	748	261	395	364	400
TENSILE STRENGTH (psi)	1447	1145	1372	1285	1357	1285	1270	1365	1253	1240
% ELONGATION	651	956	465	631	1027	533	550	726	780	576
% PERMANENT SET AT BREAK	28	31	9	25	47	19	15	27	24	17
SHORE A HARDNESS	72	76	77	78	80	81	67	75	79	78
% COMPRESSION SET * (70 hrs. at 250°F)	100	100	100	100	100	100	100	100	93.5	100

*All Samples crushed

TABLE XI

KEL-F RESIN FILLED COMPOUNDS

7343 -9-	a	b	c	d	e	f
KEL-F ELASTOMER	100	100	100	100	100	100
ZINC OXIDE	3	3	3	3	3	3
DYPHOS	3	3	3	3	3	3
BENZOYL PEROXIDE	3	3	3	3	3	3
KEL-F No. 200 RESIN	10	20	10			10
HI SIL LM-3			15	15		10
KEL-F No. 200 Wax					15	

PRESS CURE 60 MINUTES AT 250°F - OVEN AGED 64 HRS. AT 350°F

	a	b	c	d	e	f
TENSILE STRENGTH (psi)	1200	2640	2650	4610	1330	
% ELONGATION	420	570	455	490	480	
% PERMANENT SET AT BREAK	18	45	30	20	6	
SHORE A HARDNESS	66	69	75	71	60	

Note - Compounds for acid permeability tests see Table XVIII, and Figure 4.

TABLE XII

COMPRESSION SET STUDIES

	266-37-1	266-37-2	266-18-1	266-37-3	266-37-4	266-5-5
KEL-F ELASTOMER	100	100	100	100	100	100
BENZOYL PEROXIDE	1	2	3	3	3	3
ZINC OXIDE				5		5
DYPHOS					5	5

PRESS CURE 60 MINUTES AT 300°F - OVEN CURE 24 HRS. AT 300°F

	266-37-1	266-37-2	266-18-1	266-37-3	266-37-4	266-5-5
100% MODULUS (psi)	NO CURE	NO CURE	196	142	195	225
TENSILE STRENGTH (psi)			786	630	1870	1450
% ELONGATION			700	400	550	500
% PERMANENT SET AT BREAK			20	0	5	3
SHORE A HARDNESS			67			
% COMPRESSION SET (70 hrs. at 250°F)			60	47	52	51

TABLE XIII

EFFECT OF MERCURIC OXIDE ON COMPRESSION SET

Compound 266-	85-1	85-2	85-3	85-4	83-1	85-5	62-1	85-6	85-7	85-8
KEL-F ELASTOMER	100	100	100	100	100	100	100	100	100	100
DYPHOS	1	1	1.5	1.5	2	2	3	3	3	3
ZINC OXIDE	1	1	1.5	1.5	2	2	3	3	3	3
BENZOYL PEROXIDE	1	1	1.5	1.5	2	2	3	3	3	3
MERCURIC OXIDE		1.5		1.5		1.5		1.5	2	3
PRESS CURE 60 MINUTES AT 300°F - OVEN AGED 24 HRS. AT 300°F										
% COMPRESSION SET (70 hrs. at 250°F)	62	62	63	47	48	45	43	46	48	46
TENSILE STRENGTH (psi)	1200	1350	1430	1280	1440	1460	1510	1660	1710	2110

TABLE XIV

70 Hour Fluid Immersion Tests

PRESS CURE 60 MINUTES AT 300°F - OVEN AGED 24 HOURS AT 300°F

Compounds 266-	5-5	31-1	37-3	37-4	50-1	50-3	50-4	62-1	62-2	62-3	62-4	63-1	63-2	73-1	73-2	73-3	97-1
KEL-F ELASTOMER	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
ZINC OXIDE	5	10	5		5	5	10	3	3	5	10	5	5	5	5	5	3
DYPHOS	5	10		5		5	10	3	3	5	10			5	5	5	3
BENZOYL PEROXIDE	3	3	3	3	3	3	3	3	3	3	3			3	3	3	3
TETA												4	1.5				
MDI													5				
HI SIL x 303					10	10	10		20	30	30			15		15	15
HI SIL LM-3															15		
MONOCHLORO-TRIFLUOROETHYLENE OIL																10	
ORIGINAL PROPERTIES																	
100% MODULUS (psi)	229	259	228	245	377	350	340	216	574	837	812	234	319				
TENSILE STRENGTH (psi)	1713	2585	1490	1870	2355	3760	3750	1510	3820	3540	3525	1665	965	4630	4410	2990	4100
% ELONGATION	440	460	430	450	390	481	525	480	480	470	450	644	620	430	430	470	530
% PERMANENT SET AT BREAK	4	8	5	5	9	23	14	6	18	16	27	7	13				

TABLE XIV 70 HOUR FLUID IMMERSION TESTS (cont'd)

Compounds 266-	5-5	33-1	37-3	37-4	50-1	50-3	50-4	62-1	62-2	62-3	62-4	63-1	63-2	73-1	73-2	73-3	97-1
SHORE A HARDNESS	72	72	81	74	81	72	75	73	78	84	85	72	75	73	72	76	71
% COMPRESSION SET (70 HRS. AT 250°F)	51	69	47	52		66	64	44	81	84	80						
MLO-8200 FLUID AT 400°F																	
100% MODULUS (psi)	263	262	195	270	582	435	553	229	762	1425	1385		482	780	590	740	
TENSILE STRENGTH (psi)	998	974	562	560	1233	2433	2475	1145	2225	2990	3380	2063	665	2030	1800	1500	
% ELONGATION	343	340	280	320	220	410	320	418	275	217	226	7	180	230	250	200	
% PERMANENT SET AT BREAK	6	6	5	10	7	8	8	8	9	14	13		4	9	11	7	
SHORE A HARDNESS	66	69	67	73	79	73	75	70	85	94	91	100+	75	77	72	78	
% VOLUME CHANGE (70 HRS. AT 400°F)	6.4	7.1	11.4	8.8	8.5	7.7	6.9	7.8	7.8	10.5	7.4	7.0	7.5	6.8	10.2	7.6	
CONDITION*	SC	SC	SC	SC	SC	G	G	G	G	G	G	B	SC	G	G	G	

* SC - Slight Cracking
G - Good
B - Brittle

TABLE XIV 70 HOUR IMMERSION TESTS (cont'd)

COMPOUNDS 266-	5-5	33-1	37-3	37-4	50-1	50-3	50-4	62-1	62-2	62-3	62-4	63-1	63-2	73-1	73-2	73-3	97-1
MIL-L-7808 Fluid at 350°F																	
100% MODULUS (psi)	106	95	97	106	116	116	102										
TENSILE STRENGTH (psi)	395	229	446	441	863	677	554										
% ELONGATION	465	550	575	450	640	565	510										
% PERMANENT SET AT BREAK	14	34	21	16	28	22	17										
SHORE A HARDNESS	35	32	38	38	38	39	38										
% VOLUME CHANGE	108	108	112	102	107	113	105										

NOTE: All samples had started to deteriorate and were very sticky.

COMPOUNDS 266-	5-5	33-1	37-3	37-4	50-1	50-3	50-4	62-1	62-2	62-3	62-4	63-1	63-2	73-1	73-2	73-3	97-1
70-30 FUEL AT ROOM TEMPERATURE																	
100% MODULUS (psi)	210	232	202	184	227	259	289	220	333	457	457	233	255				
TENSILE STRENGTH (psi)	811	881	836	666	1392	1895	1610	768	1968	2180	2265	1278	449				1560
% ELONGATION	450	465	450	440	445	475	430	425	500	510	480	560	1125				770
% PERMANENT SET AT BREAK	7	6	8	7	8	11	7	6	19	32	25	19	68				
SHORE A HARDNESS	66	66	66	68	70	74	70	72	73	78	77	72	71				51
% VOLUME CHANGE	30	32	28	30	32	27	24	30	27	24	25	26	28				25

TABLE XIV 70 HOUR IMMERSION TESTS (cont'd.)

IMMERSION IN RED FUMING NITRIC ACID AT ROOM TEMPERATURE *

Compounds 266-	5-5	33-1	37-3	37-4	50-1	50-3	50-4	62-1	62-2	62-3	62-4	63-1	63-2	73-1	73-2	73-3	97-1
100% MODULUS (psi)	128	153	155	164	150	178	183										
TENSILE STRENGTH (psi)	293	354	489	590	520	560	580							970	1380	220	
% ELONGATION	620	520	520	460	495	480	490							640	420	1000+	
% PERMANENT SET AT BREAK	18	12	15	12	17	21	26										
SHORE A HARDNESS	60	59	61	62	66	67	68							63	63	60	
% VOLUME INCREASE	41	41	27	42	30	27	22							15	21	17	

*Surface conditions were excellent after the test.

TABLE XV

IMMERSION TESTS IN OS-45 FLUID

	266-97-1	266-37-2
KEL-F ELASTOMER	100	100
ZINC OXIDE	3	
DYPHOS	3	
BENZOYL PEROXIDE	3	2
HI SIL x 303	15	
ORIGINAL PROPERTIES		
TENSILE STRENGTH (psi)	4100	1080
% ELONGATION	530	500
SHORE A HARDNESS	71	60
IMMERSED 24 HOURS AT 400°F		
TENSILE STRENGTH (psi)	3600	
% ELONGATION	590	
% VOLUME INCREASE	8.6	
SHORE A HARDNESS		
IMMERSED 72 HOURS AT 400°F		
TENSILE STRENGTH (psi)	2130	
% ELONGATION	390	
% VOLUME INCREASE	11.6	
SHORE A HARDNESS	63	

TABLE XV IMMERSION TESTS IN OS-45 FLUID (cont'd)

	266-97-1	266-37-2
IMMERSED 168 HOURS AT 400°F		
TENSILE STRENGTH (psi)	1330	
% ELONGATION	240	
% VOLUME INCREASE	12.7	
SHORE A HARDNESS	67	
IMMERSED 3 HOURS AT 550°F*		
TENSILE STRENGTH	1000	610
% ELONGATION	25	30
% VOLUME INCREASE	3.6	-7.0
SHORE A HARDNESS	95	91

NOTE - See Figure 3

* Surfaces became very hard and brittle, broke on bending.

TABLE XVI

LOW TEMPERATURE DATA

Brittle Point Data °F

Compound	Passed	Failed
266-50-3	-45	-49
266-50-4	-40	-45
266-62-2	-45	-50
266-62-4	-45	-50
266-37-3	-57.5	-59

TABLE XVII

TEMPERATURE - RETRACTION DATA (°F)

% Retraction	266-37-2	266-62-1	266-78-1
3	+20		
5		+26	+23
8	+27		
10		+30	+26
13	+30		
20	+31	+32	+30
27	+32		
30		+33	+32
33	+33		
40	+34	+35	+33
47	+34		
50		+36	+36
53	+35		
60	+35		
63	+36		

TABLE XVIII

RED FUMING NITRIC ACID H - CELL PERMEABILITY OF KEL-F ELASTOMER COMPOUNDS

Gm/m^2 Penetration of Test Specimens *

Time, Hours		5	3	7	25	32	95 1/2	120	144	169
266-62-1	0	0	0	0	0	3	250	End		
266-62-1	0	0	0	0	0	0	230	End		
7343-9-a	0	0	0	0	0	0	0	24	60	110
7343-9-d	0	0	0	0	.9	26	320	End		
7343-9-e	0	0	0	0	0	0	75	270	End	
7343-9-f	0	0	0	0	.9	11	390	End		

Note - See Table II for Formulas and Physical Properties.

* Calculations made from pH data. Test Specimens 8 to 10 mils thick.

APPENDIX II

TRADEMARK COMPOUNDING MATERIALS

Code or Trade Name	Material	Manufacturer
KEL-F ELASTOMER	Fluorocarbon Copolymer	The M. W. Kellogg Co.
DYPHOS	Dibasic lead Phosphate	The National Lead Co.
HI SIL	Fine Silica	Columbia Southern Chemical Corp.
HI SIL x 303	Super Fine Silica	Columbia Southern Chemical Corp.
HI SIL IM-3	Silicone coated Fine Silica	Columbia Southern Chemical Corp.
SILENE EF	Calcium Silicate	Columbia Southern Chemical Corp.
KALVAN	Ultra fine calcium carbonate	R. T. Vanderbilt Co.
ACCELERATOR "808"	Condensation product of butyraldehyde and aniline	E. I. du Pont de Nemours & Co., Inc.
NA-22	2 mercaptoimidazeline	E. I. du Pont de Nemours & Co., Inc.
MDI	Methylene bis (4 phenyl isocyanate)	E. I. du Pont de Nemours & Co., Inc.
DPG	Diphenylguanidine	E. I. du Pont de Nemours & Co., Inc.
FT Black	Fine Thermal Carbon Black	------------------------
TETA	Triethylene tetramine	United Carbide & Carbon Corp.
KEL-F No. 300 POWDER	Monochlorotrifluoroethylene polymer	The M. W. Kellogg Co.
KEL-F No. 200 RESIN	Fluorocarbon Copolymer	The M. W. Kellogg Co.
KEL-F No. 200 WAX		The M. W. Kellogg Co.
LUPERCO AGE	50% Benzoyl Peroxide 50% Silicone Oil	Novadel-Agene Corp.

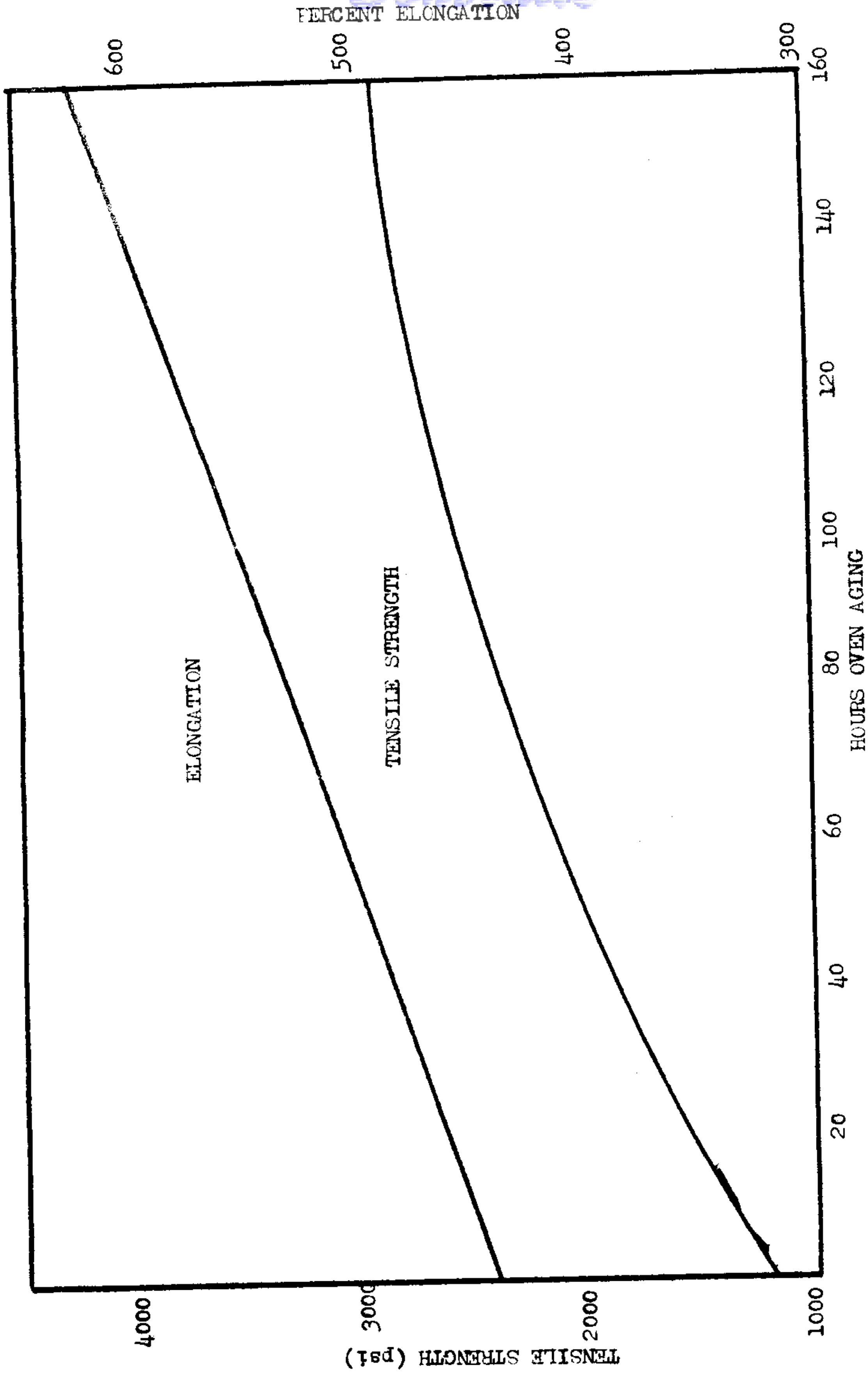

FIGURE 1
EFFECT OF OVEN AGING AT 300 °F
COMPOUND 266-33-1

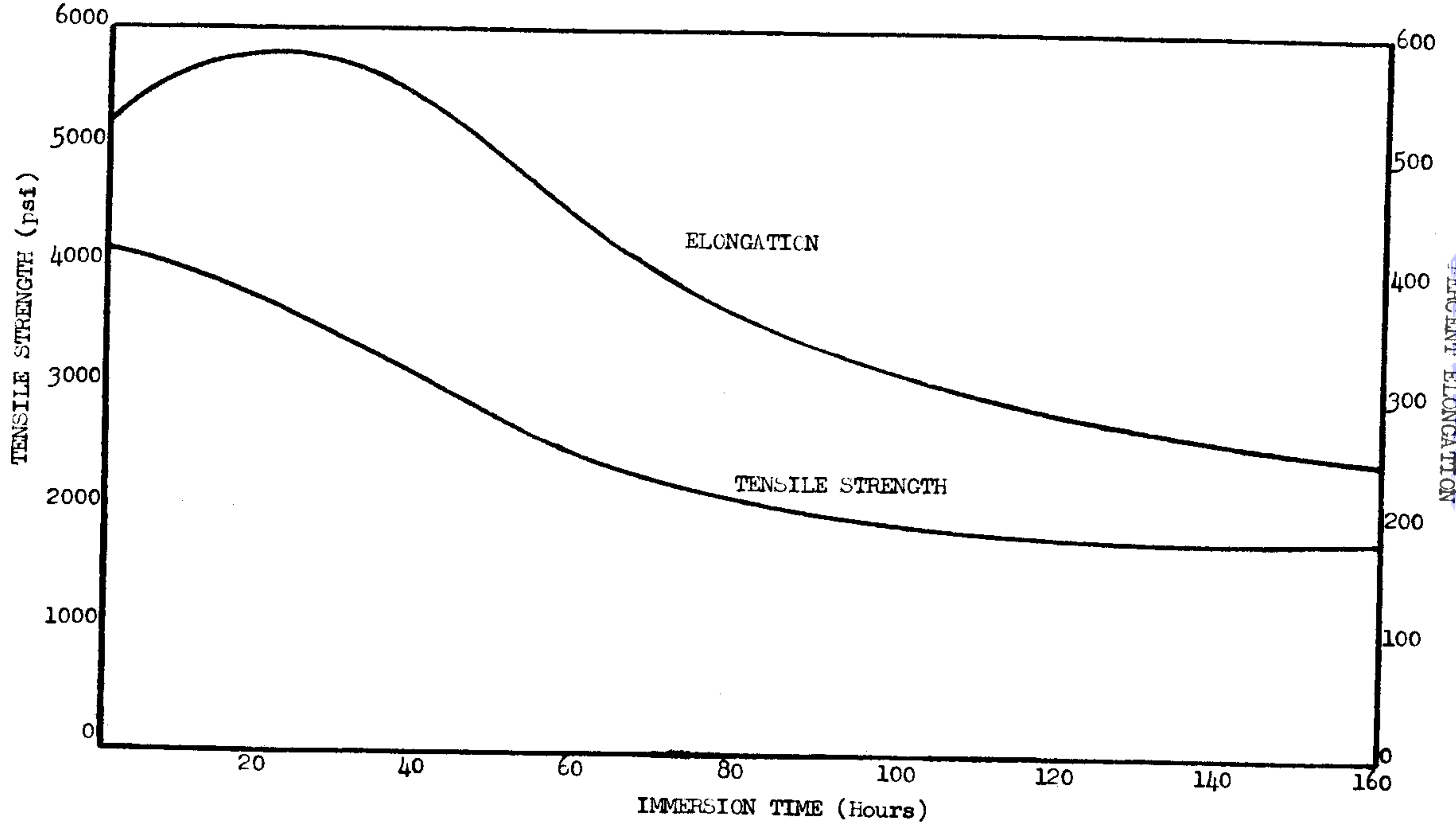

FIGURE 2

COMPOUND 266-97-1 IMMERSED
IN OS-45 FLUID AT 400° F

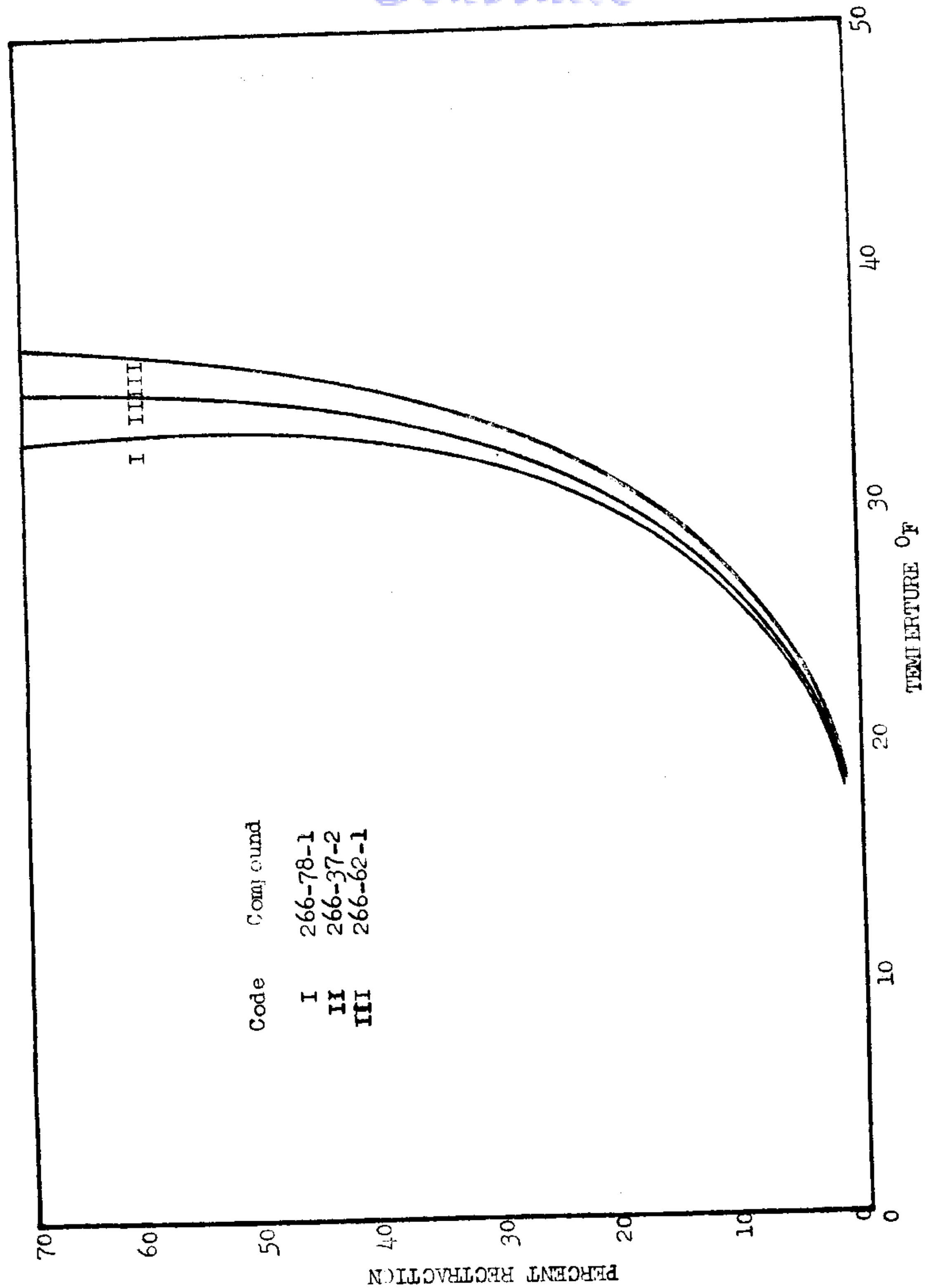

FIGURE 3
TEMPERATURE - RETRACTION CURVES

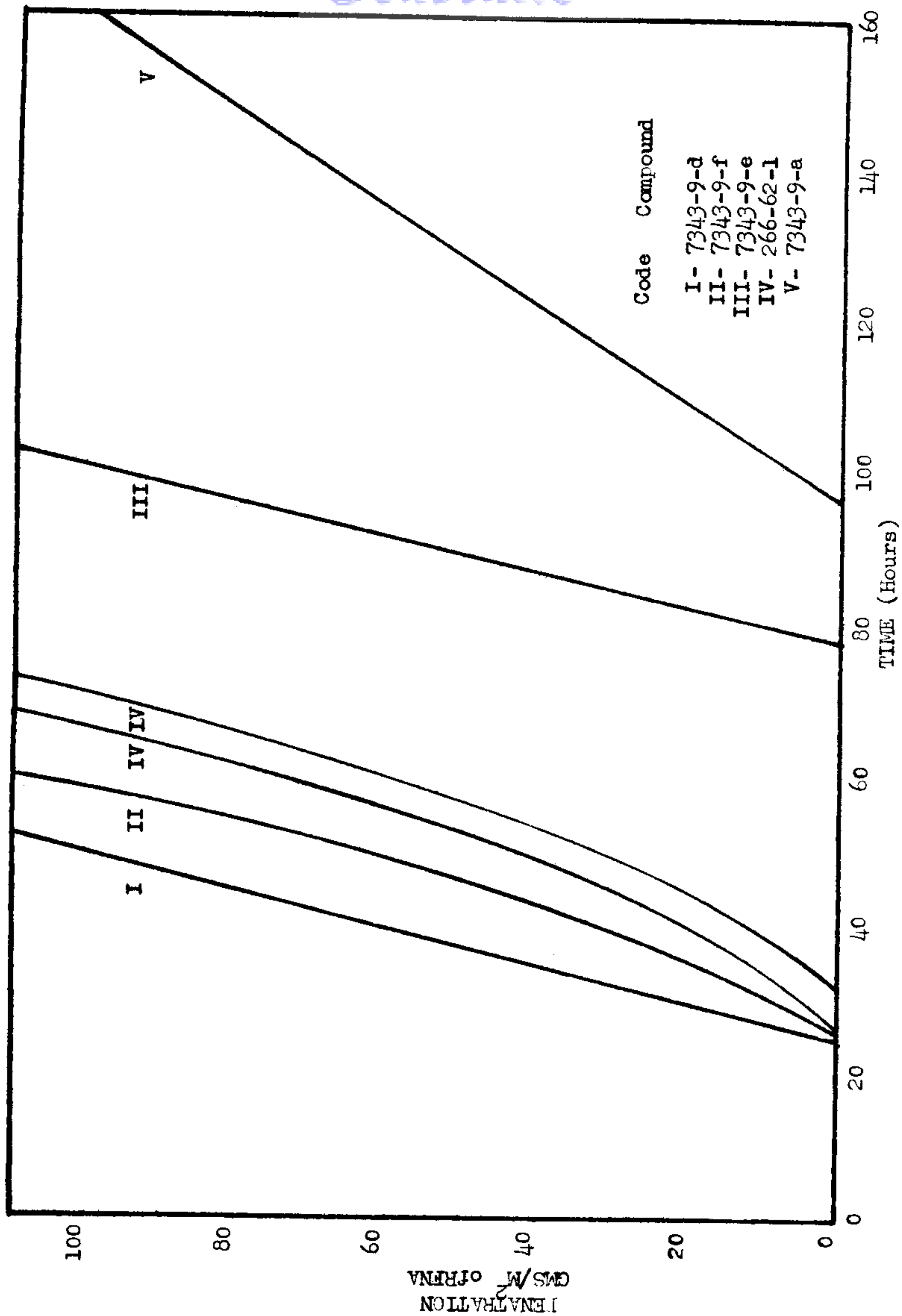

FIGURE 4
PERMEABILITY TO RFNA