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TECHNICAL REPORT	
PERFORMANCE OF URETHANE VULCANIZATES IN ENVIRONMENTS OF HIGH HUMIDITY	
By Frank B. Testroet	
Department of the Army Project No. 1-H-O-24401-A-110	
AMC Code No. 5025.11.842	
Report No. 63-2808	Copy No. _____
IEL No. 1-9-100-2	Date 30 August 1963

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PERFORMANCE OF URETHANE VULCANIZATES
IN ENVIRONMENTS OF HIGH HUMIDITY

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30 August 1963

DA Project No. 1-H-O-24401-A-110

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Rock Island Arsenal
Rock Island, Illinois

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ABSTRACT

Polyester urethane elastomeric end items have exhibited severe breakdown after service periods of as little as one to two years. The rubber becomes tar-like within an unreasonably brief period of service and is rendered unfit for further use. Duplication of this rubber failure under laboratory conditions was accomplished by exposing specimens over water at 158°F and at room temperature. Two modes of degradation appear to occur simultaneously and independently of one another; i.e., hydrolysis or chain scission of specimens exposed at 158°F and microorganism attack of the specimen at room temperature accompanied by hydrolysis. A polycarbodiimide and pentachlorophenol demonstrate their ability to retard chain scission and microorganism attack, respectively. Unfortunately, pentachlorophenol interferes somewhat with the efficiency of the polycarbodiimide when the two materials are used in admixture.

Polyether urethane vulcanizates, containing no special additives, are not adversely affected by environments of high humidity.

RECOMMENDATIONS

It is recommended that, for the present, the use of polyester urethane elastomers be discontinued.

Polyester urethane A and D vulcanizates containing PCD and pentachlorophenol, individually and in admixture, should be subjected to outdoor aging tests at Fort Amador to determine the efficacy of these additives in prolonging the life of this rubber.

It is recommended that the search for methods to improve the performance of polyester urethanes in environments of high humidity be continued.

**PERFORMANCE OF URETHANE VULCANIZATES
IN ENVIRONMENTS OF HIGH HUMIDITY**

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PERFORMANCE OF URETHANE VULCANIZATES IN ENVIRONMENTS OF HIGH HUMIDITY

OBJECT

Attempt to detect and eliminate the cause for the extreme deterioration of polyester urethane end items in field service.

Determine the compatibility of various elastomeric vulcanizates with the environment at the Canal Zone and localities of similar climate.

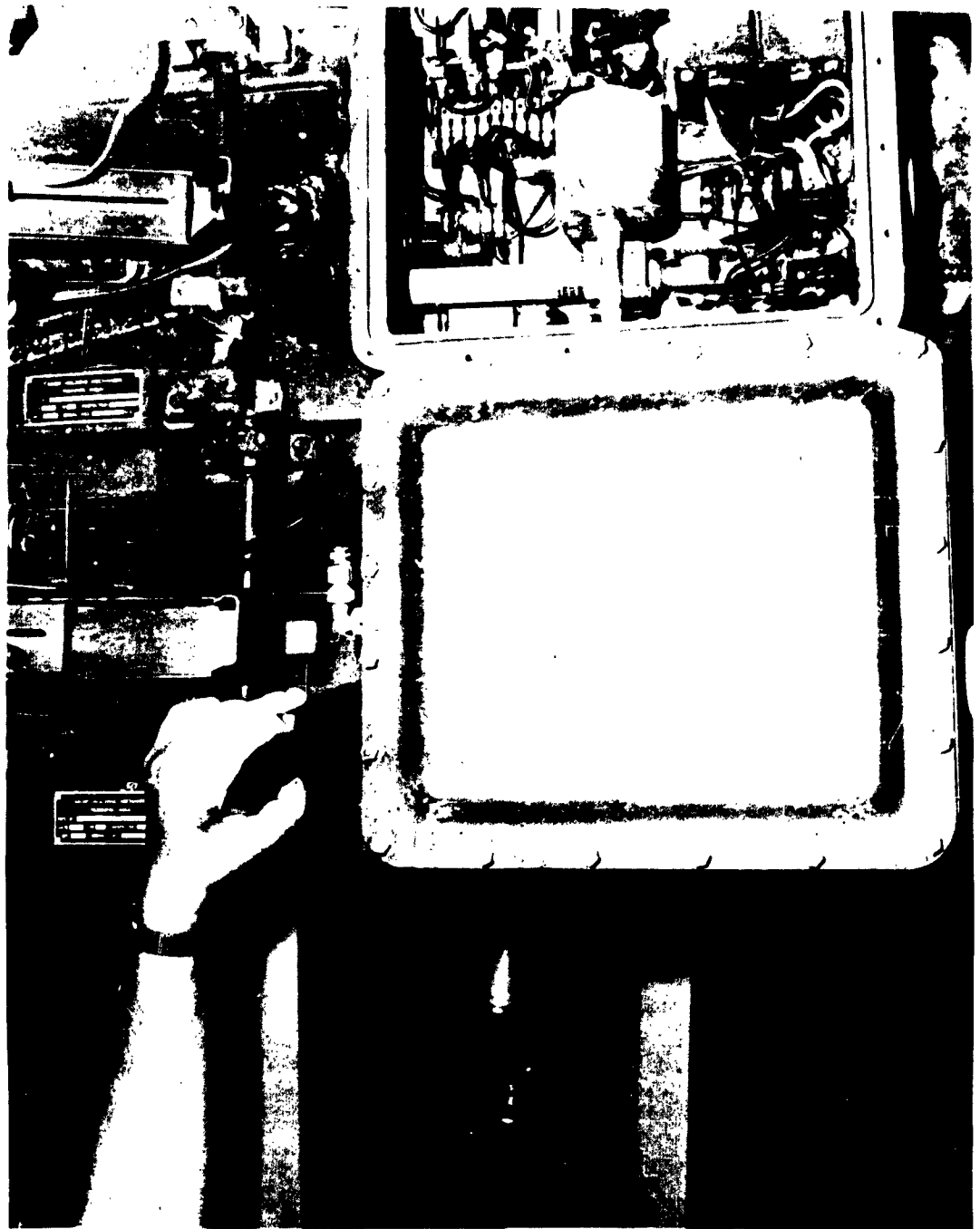
INTRODUCTION

Unsatisfactory equipment reports concerning Bellows, Rubber, for Target, Holding, Mechanism, Trainfire, FSN 6920-585-2640, were received from the Canal Zone and Fort Knox, Kentucky. It was reported that subject bellows were severely degraded during normal field use as integral components of trainfire mechanisms (an electric motor driven mechanical device used to "pop-up" silhouette targets for small arms training). The rubber became very soft, tacky and unfit for further use within a short period of exposure on the trainfire ranges. Figure 1 shows the unsatisfactory condition of bellows mounted on trainfire mechanisms as taken from a firing range at Fort Knox, Kentucky. Ultimate softening and collapse of the bellows, which appear as partially melted tar, is illustrated in Figure 2.

Production of bellows used for this application have been made of polychloroprene, which does not show the softening, since January 1961.

Analysis of the collapsed bellows revealed that they were fabricated from polyester urethane A elastomer*. This elastomer was utilized for this application because of the rubber's durability, as indicated by its inherent resistance to degradation caused by heat, oxygen, ozone and a variety of fluids as well as excellent resistance to impact fracture at extremely low temperatures and excellent physical strength.¹ Furthermore, as will be shown later in this report, good stability of a vulcanizate based on polyester urethane A was demonstrated during three years indoor and outdoor storage tests at Rock Island Arsenal, Illinois. Therefore, the Canal Zone's climate, which is described as tropical with consistently high but not extreme temperatures, high humidity and abundant

* For trade names see Code Sheet at end of report.



BELLOWS FAILURE INCURRED DURING
FIELD SERVICE AT FORT KNOX, KENTUCKY



TAR-LIKE APPEARANCE OF BELLOWS
AFTER REMOVAL FROM TRAINFIRE MECHANISMS

rainfall, and the more moderate climate of Fort Knox, Kentucky, did not appear to be the sole factor for the extremely short service life of the bellows.

No extraordinary circumstances to account for the poor performance of the bellows in field use were apparent. It is to be noted, however, that because the bellows were mounted in an airtight position they were expanded during the heat of the day and returned to an unstressed position during the cooler nights. The trainfire mechanisms were, at times, submerged during and after heavy rainfall.

Available technical information and consultation with the manufacturer of the elastomer involved, failed to provide a solution for the problem. Accordingly, a program was initiated to ascertain (1) the reason for the excessive deterioration of the rubber, (2) a means of eliminating or retarding this deterioration without altering desirable physical properties of the vulcanizate and (3) the suitability of other elastomers such as SBR, nitrile, polybutadiene or polychloroprene for use in environments such as found in the Canal Zone.

PROCEDURE

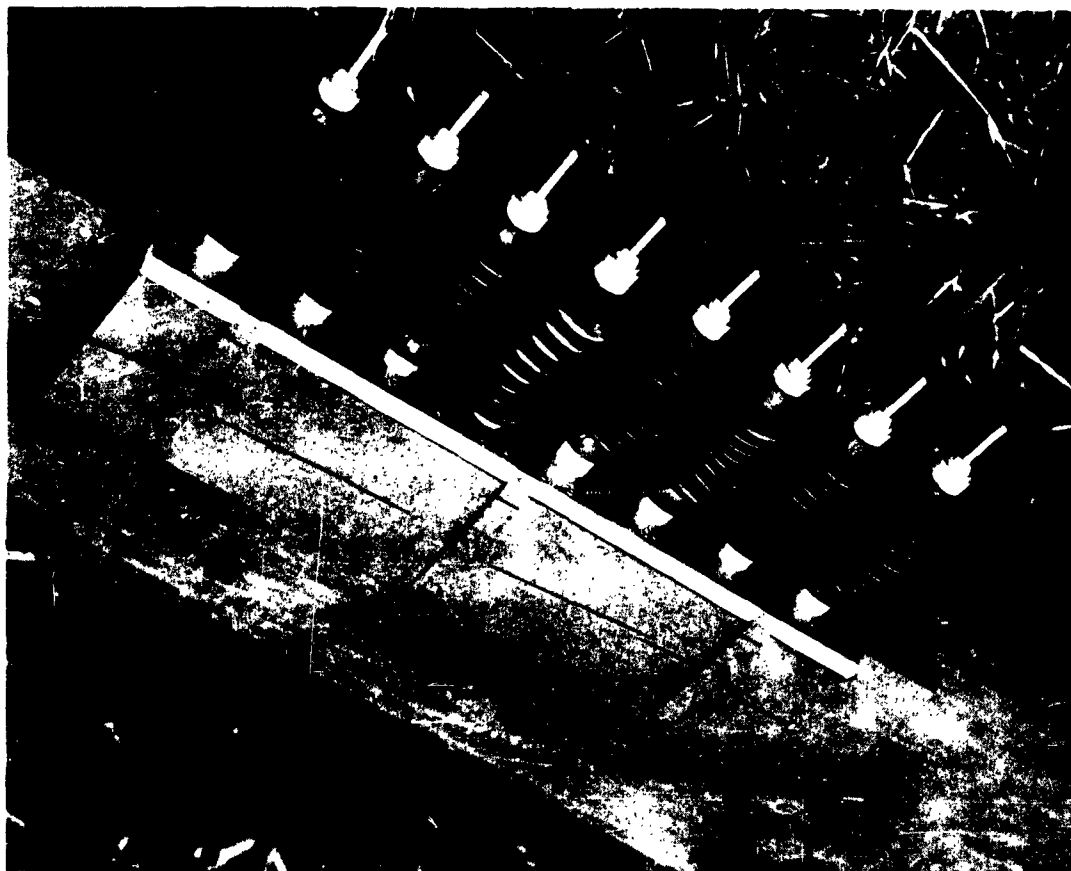
In the initial phase of this investigation, controlled outdoor aging tests were conducted in an attempt to determine the conditions required to produce the reported rubber failure. Bellows and test pads were prepared from materials listed in Table I. Polyester urethane A compounds are included, one of which (Z60F5), was utilized in fabricating the bellows found to be unsatisfactory during field service. Two compounds based on polyester urethane D, a modification of polyester urethane A, were formulated for testing and comparison with its predecessor. One each of the bellows were affixed to test fixtures in such a manner as to simulate emplacement of the bellows, in a static position, on a trainfire mechanism. Test fixtures were exposed at Fort Amador, Canal Zone, to an environment similar to that encountered by the trainfire mechanisms reported in the aforementioned unsatisfactory equipment report. Fort Amador was chosen over Fort Knox as the site for the outdoor aging tests because of its somewhat more severe climate and the availability of facilities for environmental exposure.

A test fixture is shown in Figure 3 and the general test area may be seen in Figure 4. Observations of periodic visual examinations of the bellows were recorded by personnel at Fort Amador and furnished this Arsenal in monthly progress reports.

TABLE I
FORMULATIONS OF EXPERIMENTAL BELLOWS FOR ENVIRONMENTAL EXPOSURE
AT FORT AMADOR, CANAL ZONE

COMPOUNDING INGREDIENTS	P a r t s B y W e i g h t									
	<u>N75EF</u>	<u>Z60CF8</u>	<u>Z60F5</u>	<u>Z10SC1F2</u>	<u>S64B</u>	<u>S64K</u>	<u>N117EF3</u>	<u>S64</u>	<u>Z60D4</u>	<u>Z60D4N</u>
Polychloroprene Elastomer	100									
Polyester Urethane A		100	100							
Carboxy modified polybutadiene				100						
Carboxylic elastomer					100					
SEB 1500						100	100	100	100	100
Polyester Urethane D										
Zinc Oxide	5			3	3	3	5	3		
Stearic Acid		0.2	0.2	2	2	2	2	2	0.2	0.2
Diphenyl-para-phenylenediamine/ phenyl beta naphthalamine mixture	3			1	1	1	1	1		
Polymerized trimethyldihydroquinoline					1	1				
Phenyl beta naphthalamine	3				1	1				
Wax	4									
Magnesium Oxide	1									
2-mercaptimidazole										
Tetramethyl thiuram monosulfide										
Sulfur				1.75	1.75	1.75	1.5	1.75	4	4
Dicumyl peroxide, 40% active		3	4							
N-cyclohexyl-2-benzothiazole sulfenamide				1	1	1				
N,N'-di-3(5 methyl heptyl)-para- phenylenediamine					3	*				1
Mercaptobenzothiazole										
Trioctylphosphate	35						15			
Dioctyl sebacate		5								
Silicon dioxide										
SEF Black				20						
FFB Black		5	30		50	50	30	50	25	25
SAF Black	40									
Toluene 2,4 diisocyanate on calcium carbonate									4	2
Compression molded, min. 60°	30 & 307	30 & 320	30 & 320	30 & 307	30 & 307	30 & 307	30 & 307	30 & 307	30 & 320**	30 & 320**

* Vulcanizate dipped twice in 50/50 mixture of N,N'-di-3(5 methyl heptyl)-para-phenylenediamine/acetone
** Post cured 24 hours @ 300°F



TEST FIXTURE EXPOSED AT FORT AMADOR, CANAL ZONE



TEST AREA FOR ENVIRONMENTAL EXPOSURE
OF RUBBER BELLOWS AT FORT AMADOR

Physical properties of the bellows compounds, as well as all other compounds reported herein, were measured in accordance with ASTM² procedures where applicable.

Test pads of polyurethane A (Z60, See Table II for formula) were subjected to three year outdoor and indoor shelf aging test at Rock Island, Illinois. Pads stored outdoors were placed in a test rack facing south at a forty-five degree angle to the horizontal. These pads encountered temperatures ranging from -10°F to 100°F, accompanied by periods of high humidity, rainfall and, occasionally, the pads were covered with snow. Shelf aged pads were wrapped in polyethylene and stored in the absence of light. Physical properties of the stored pads were measured after 3, 6, 12, 24 and 36 months storage.

Laboratory tests were improvised with the intention of inducing degradation of rubber specimens similar to that encountered during field service of urethane bellows. These tests consisted of exposing stressed specimens over water in a fixture in such a manner as to cause 20 percent extension of the rubber or by looping strips measuring 2.75" x 0.250 x 0.075" and securing the ends with tape. Dumbbell specimens used to determine any change in physical properties were not under stress. Peroxide cured polyester urethanes A, B, C and D and polyester urethane B vulcanizates employing a sulfur cure system were exposed to the high humidity environments. Likewise, polyether urethane A utilizing peroxide and sulfur cures and an isocyanate cured polyether urethane B were exposed. Formulations of these vulcanizates are listed in Table II.

Bent loop specimens of polyester urethane A were suspended over water in sealed containers; appropriate precautions were taken, in one instance, to insure that the rubber was exposed under sterile conditions whereas the contents of a second container was exposed under nonsterile conditions. These containers, along with one that was neither sealed or covered, were placed in a cabinet maintained at a temperature range of 85 to 95°F at 86 ± 2 percent relative humidity.

Polycarbodiimide (PCD), a material of the type $R-N=C=N-R'$, has been reported to improve the hydrolytic stability of polyester based resins used with glass fiber.³ The performance of PCD in polyester and polyether urethane elastomeric vulcanizates was evaluated by exposing vulcanizates containing 4 pph rubber PCD to high humidity environments. Stress-strain properties of the vulcanizates aged over water at 158°F were measured after 3, 7, 10, 14 days and intervals of 7 days thereafter. Stressed specimens in

TABLE II
POLYESTER AND POLYETHER URETHANE COMPOUND FORMULATIONS

	Z129	Z129G	Z129GM17	Z129GM18	Z129GM19	Z129M18	Z60	Z121	Z121G	Z121C	Z121CG	Z76CDF	Z124F	Z124FG	Z51	Z51G	Z51C	Z51CG	Z46E
Polyester urethane A	100	100	100	100	100	100	100	100	100	100	100								
Polyester urethane B												100							
Polyester urethane C													100						
Polyester urethane D														100					
Polyether urethane A															100				
Polyether urethane B																100			
Polycarbodiimide (PCD)																			100
Stearic acid	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	4			0.25	0.2	0.2		4		4	
Dicumyl peroxide, 40% active	5	5	5	5	5	5	4			3	3	4		5			2.5	2.5	
Sulfur								2	2						1.5	1.5			
2-Mercaptobenzothiazole								2	2						1	1			
Benzothiazyl disulfide								4	4						3	3			
4,4'-methylene-O-tolylisocyanate																			5
MBTS: Zinc chloride complex																			
Toluene diisocyanate on calcium carbonate													4	4					
Activator A								1	1										
Cadmium stearate								0.5	0.5										
Pentachlorophenol			1	2	3	2													
FEF carbon black	35	35	35	35	35	35	25												
SAF carbon black								30	30	30	30		15	15					
BAF carbon black												20							
Cure time & temp., min. @ 0°.	30 @ 320	30 @ 320	30 @ 320	30 @ 320	30 @ 320	30 @ 320	30 @ 320	20 @ 307	20 @ 307	15 @ 307	15 @ 307	30 @ 320	30 @ 320	30 @ 320	30 @ 290	45 @ 290	45 @ 310	45 @ 310	30 @ 275
Post cure time and temp., 0°.													24 hr/ 300	24 hr/ 300					

the room temperature test were inspected periodically to detect any surface degradation or softening of the rubber.

Polyester urethane A vulcanizates, with and without PCD, were exposed over and immersed in water at room temperature and 158°F in an additional test of the stabilizing efficiency of PCD.

Curatives other than the dicumyl peroxide usually utilized with Polyester urethane A were evaluated in search of a possible improved resistance to the noted degradation. Crosslinking of this elastomer by exposure to 35 megarads gamma radiation was also attempted. Formulations and physical properties of compositions containing experimental curatives may be found in Table III.

Additives, other than the previously mentioned PCD, evaluated as potential stabilizers for polyester urethane compound Z129, are listed in Table IV.

Easy processing channel (EPC) and medium thermal (MT) carbon blacks were substituted for the fast extrusion furnace (FEF) black in order to ascertain any effect exerted by the pH of the filler; the pH values for EPC, MT and FEF are approximately 5, 7 and 9, respectively. Two aluminum oxide fillers were evaluated for comparison with carbon black in polyester urethane A vulcanizates. In addition, vulcanizates containing no carbon black were also prepared for testing. These gum stocks were cured with recrystallized dicumyl peroxide and dicumyl peroxide contained on calcium carbonate. Both curatives were used in order to ascertain the effect of calcium carbonate on initiating deterioration of the rubber.

RESULTS

Environmental exposure of the bellows at Fort Amador was terminated after 14 months at which time the test fixtures were returned to Rock Island Arsenal for examination. Figures 5, 6 and 7 depict the bellows upon completion of the environmental exposure test. It will be noted that the polyester urethane A bellows, located at stations 2 and 3 of Figure 5, are severely degraded whereas polyester urethane D bellows located at stations 2, 3 and 4 of Figure 7 are in good condition with the exception of ozone cracks on the collars.

Visual observations made at the test site are listed in Table V. These observations indicate that the polyester urethane A exhibited signs of deterioration similar to that of bellows which degraded during field use. Polyester

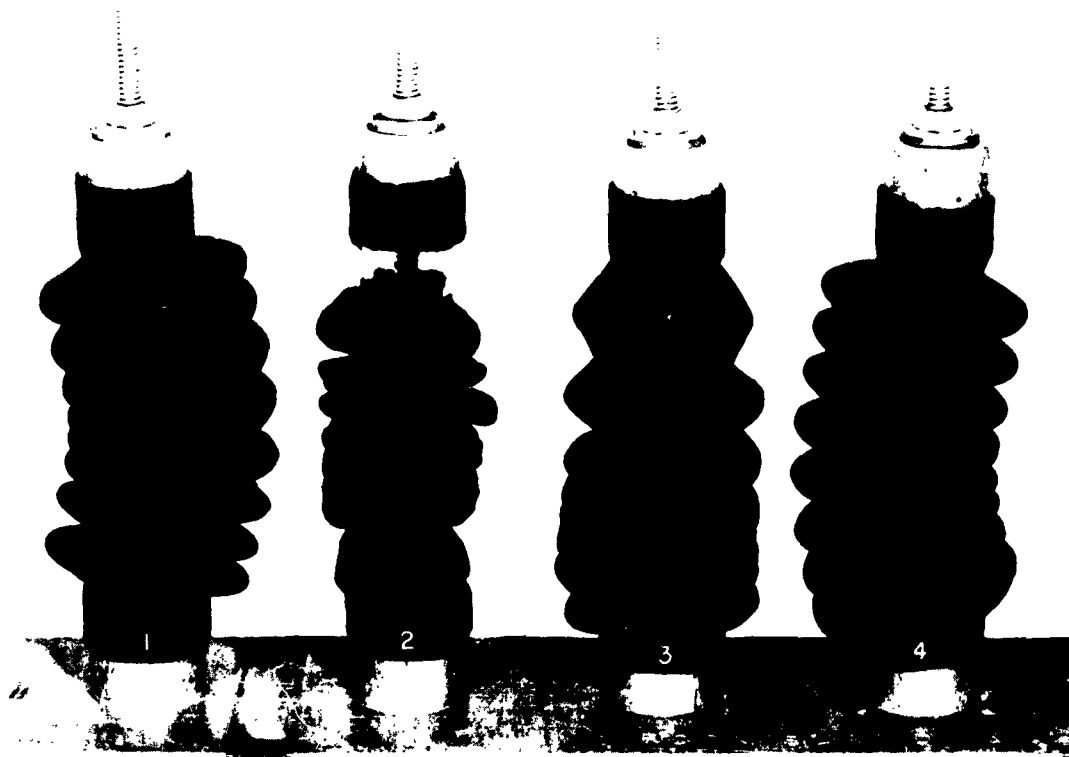
TABLE III
FORMULATIONS AND PHYSICAL PROPERTIES OF POLYESTER URETHANE A
UTILIZING EXPERIMENTAL CURE SYSTEMS

COMPOUNDING INGREDIENTS	P a r t s B y W e i g h t											
	Z129	Z129C	Z129C1	Z129C2	Z129C3	Z129C4	Z129C5	Z129C6	Z129C7	Z129C8	Z129C9	Z129C5
Polyester urethane A	100	100	100	100	100	100	100	100	100	100	100	100
Stearic acid	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
FEF carbon black	35	35	35	35	35	35	35	35	35	35	35	35
Dicumyl peroxide	5										5	
Sulfur												0.32
Di-tertiary butyl peroxide	3											
Poly-methylol polyphenyl isocyanate												
2,5-di-tertiary butyl peroxyl 2,5-di-methylbenzene												
Toluene 2,4-diisocyanate on calcium carbamate												
Epithallene 1,6-diisocyanate												
Malogenated phenol formaldehyde resin												
Phenol formaldehyde resin A plus brominated polyisobutylene/isoprene												
Phenol formaldehyde resin B plus chlorosulfonated polyethylene												
Cure Conditions												
Tensile strength, psi.	4490	1780	POOR	3800	DID	DID	1600	DID	DID	DID	EXTR-VERY	
Modulus @ 300% E, psi.	3150	310	CURE	2090	NOT	NOT	740	NOT	NOT	NOT	LY	
Elongation, %	450	1100		560	CURE	CURE	525	CURE	CURE	CURE	POOROUS	
Hardness, Shore A	71	53		68			56					
3 Days/Over Water/158°F.:												
Tensile strength, psi.	3350	860		2990			TOO				SOFT	
Modulus @ 300% E, psi.	2175	1200		1180			GUMMY				AND	
Elongation, %	515	47		665			TO				GUMMY	
Hardness, Shore A	65			63			TEST					
7 Days/Over Water/158°F.:												
Tensile strength, psi.	2990	TOO		2130								
Modulus @ 300% E, psi.	1360	GUMMY		520								
Elongation, %	685	TO		920								
Hardness, Shore A	60	TEST		52								
14 Days/Over Water/158°F.:												
Tensile strength, psi.	970			1030								
Modulus @ 300% E, psi.	150			1200								
Elongation, %	1020			40								
Hardness, Shore A	33											

All compounds compression molded 30' @ 320°F except Z129C5 which was exposed to 35 megarads gamma radiation.

TABLE IV
ADDITIVES EVALUATED AS POTENTIAL STABILIZERS
FOR POLYESTER URETHANE A VULCANIZATES

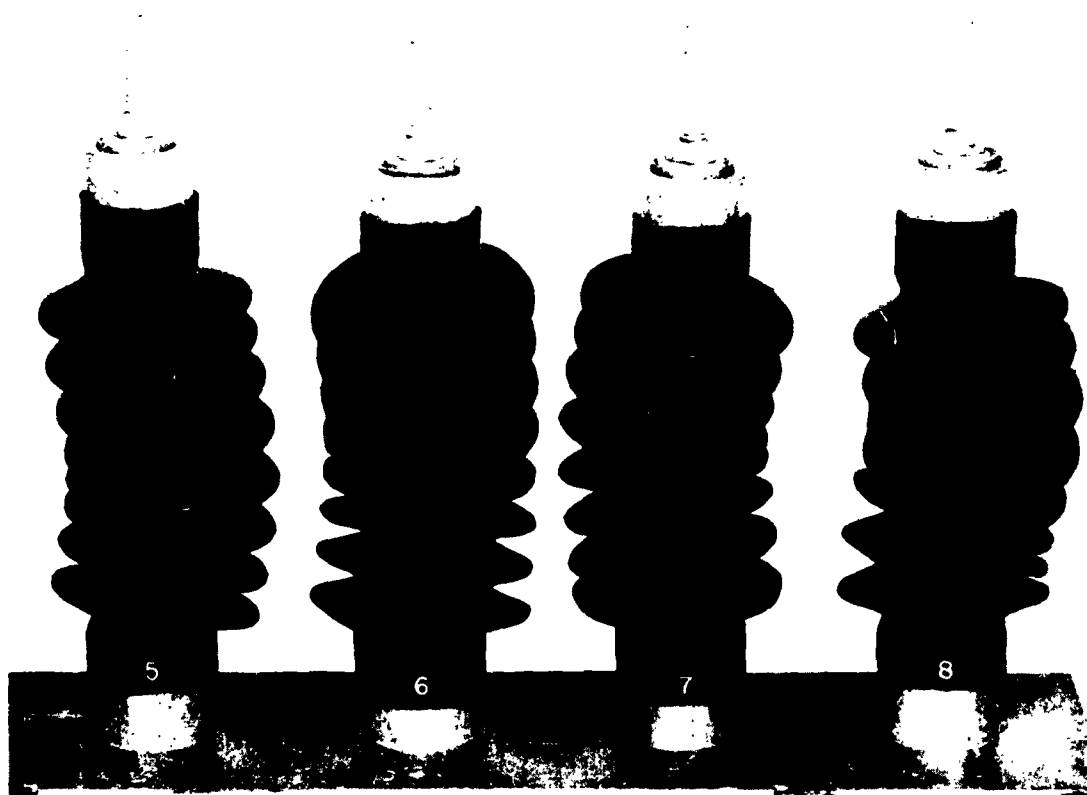
<u>CHEMICAL ADDITIVE</u>	<u>PPH RUBBER</u>
Cadmium/barium fatty acid soaps chelator mixture	5
Copper 8 hydroxy quinolinolate	0.5 & 1
2,2 diphenyl-1-picryl hydrazine	4
2,2 diphenyl-1-picryl hydrazyl	3
2,6 di-tert-butyl-4 methyl phenol	1.5
2,2'methylenebis 4 chlorophenol	1 & 1.5
2,2'methylenebis-(3,4,6 trichlorophenol)	1 & 1.5
O-phenyl phenol	1
P-aminophenol	1
Pentachlorophenol	0.5 & 2
Pentachlorophenol plus PCD	0.5, 1, 2 & 3 4
Phthalic anhydride	1.5
Propylene glycol	1
Sodium-O-phenyl phenate	1
Tetramethyl thiuram disulfide	1
Trichloromethyl thiotetrahydrophthalamide	1
Triethylene glycol	1
Zinc dibutyldithiocarbamate	0.5, 1 & 2



APPEARANCE OF BELLOWS AFTER FOURTEEN MONTHS
ENVIRONMENTAL EXPOSURE AT FORT AMADOR

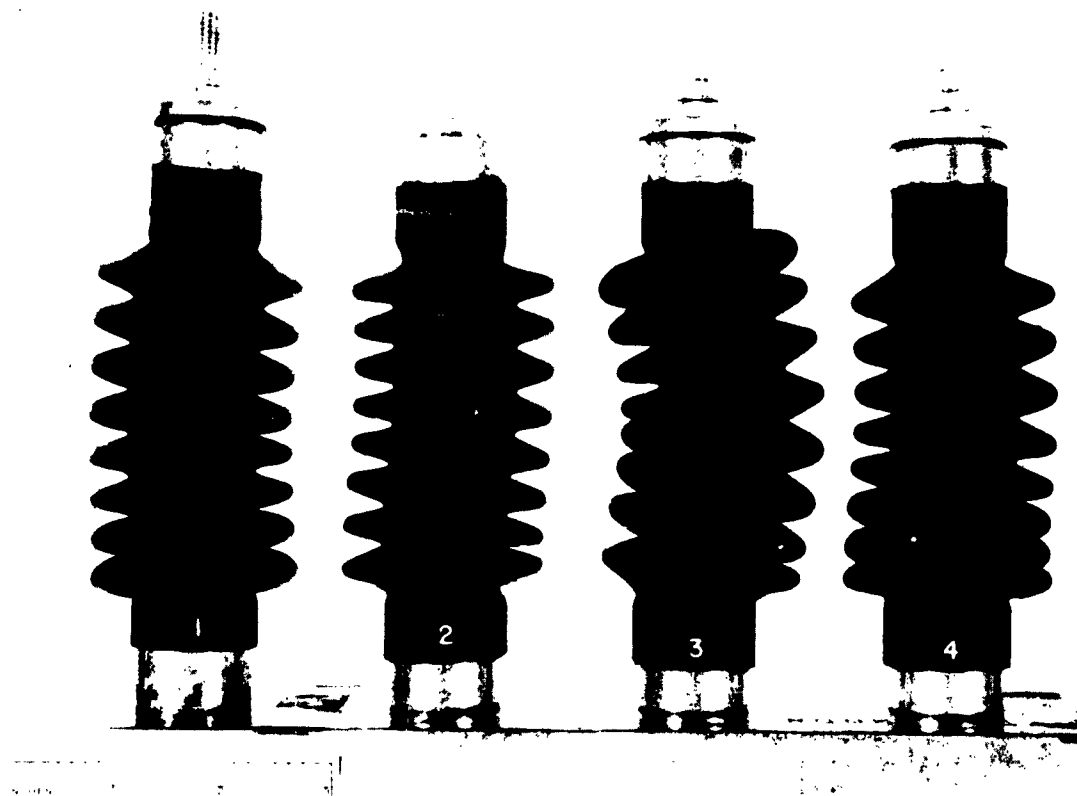
1. Polychloroprene, M75EF
2. Polyester Urethane A, Z60CF8
3. Polyester Urethane A, Z60F5
4. Carboxy Modified Polybutadiene, Z103C1F2

Negative No.
11-070-4035/ORD-62



APPEARANCE OF BELLOWS AFTER FOURTEEN MONTHS
ENVIRONMENTAL EXPOSURE AT FORT AMADOR

- 5. SBR with Antiozonant, S64B
- 6. SBR Antiozonant Applied by Dip Coating, X64X
- 7. Carboxylic Elastomer, N117EF3
- 8. SBR No Antiozonant (S64)



APPEARANCE OF BELLOWS AFTER FOURTEEN MONTHS
ENVIRONMENTAL EXPOSURE AT PANAMA

1. Chloroprene, M75EF, Airtight.
2. Polyester Urethane D, Z60D4, Not Airtight.
3. Polyester Urethane D, Z60D4, Airtight.
4. Polyester Urethane D with Fungicide, Z60D4M, Airtight.

Negative No.
11-070-4448/ORD-63

[illegible]

urethane D bellows displayed cracking on both collars but were neither softened nor tacky. Bellows prepared from other elastomers (SBR, NBR, BR and CR) remained in good condition. However, as was anticipated, the uninhibited non-ozone resistant rubbers did display ozone cracking.

The physical property values of the test bellows compounds are contained in Table VI. Examination of the stress-strain data reveals that the vulcanizates were properly cured.

Table VII furnishes data showing good physical property retention by the polyester urethane A vulcanizates during both outdoor and indoor storage for three years at Rock Island, Illinois. It will be noted that the vulcanizate may be showing a tendency towards reversion but the rubber would not be considered unserviceable even after three years storage. There was no evidence of surface tack or other surface degradation of these pads. Scrutiny of data concerning elongation, hardness and strain appears to show that indoor storage conditions are more detrimental to the vulcanizate than is outdoor exposure.

Polyester urethane A vulcanizate's poor resistance to moist heat is vividly illustrated by Figure 8 which shows a specimen as it appeared originally and after 14 and 17 days exposure over water at 158°F. Excessive surface tack and loss of dimensional integrity is exhibited after 14 days; after 17 days the tar-like appearance of the specimen closely resembles that of the bellows pictured in Figure 2.

Data furnished in Table VIII discloses that polyester urethane A, B and C vulcanizates are susceptible to degradation caused by hot, humid conditions regardless of cure system employed; i.e., sulfur or peroxide. These vulcanizates are severely degraded within 14 days exposure at 158°F and are unsatisfactory for any useful purpose. Polyester urethane D, however, shows good property retention for as long as 35 days (test in progress). It is also shown that polyether urethane vulcanizates display a decline in stress-strain properties during exposure over water at 158°F but they are not as severely degraded as the polyester urethane vulcanizates. The polyether vulcanizates would not necessarily be considered unfit for use in surroundings of high humidity.

Polycarbodiimide (PCD), when used at 4 pph rubber, has demonstrated its ability to retard degradation of polyester urethane A but appears to have little advantageous effect in polyester urethane B vulcanizates employing either sulfur or peroxide cure system. Polyester urethane A containing

TABLE VI
PHYSICAL PROPERTIES OF EXPERIMENTAL BELLOWS COMPOUNDS

PROPERTY MEASURED	N75KF	Z60CF8	Z60F5	Z103C1F2	S64B	S64X	N117F3	S64	Z60D4	Z60D4M
Tensile strength, psi.	2380	3630	3890	1200	1890	Same	2870	2200	2960	2860
Modulus @ 300% E, psi.	1540	330	1600	390	-	as	2200	-	2550	1950
Elongation, %	385	730	635	550	300	Com-	410	275	320	400
Hardness, Shore A	48	50	63	53	61	pound	75	67	72	70
Compression Set, Method B, 70 Hrs./212°F, %	22	47	54	73	19	S64	55	15	44	46
Ozone Resistance, Bent Loop, time to first crack 25 ppbm. @ 100°F										
50 ppbm. @ 100°F										
ASTM D1043, Torsional Stiffness, Temperature @ which Young's Modulus equals 10,000 psi., °F.	OK 30 Days* OK 30 Days*	OK 30 Days* OK 30 Days*	OK 30 Days* OK 30 Days*	OK 30 Days* OK 30 Days*	OK 30 Days* OK 30 Days*		2 Hrs. 2 Hrs. 2 Hrs. 2 Hrs.	OK 30 Days*	OK 30 Days*	OK 30 Days*
ASTM D746, Solenoid Activated, 2-1/2 min. exposure @ -67°F.	-73	-40	-37	-67	-36		+1	-35	-32	-32
70 Hrs./212°F/Air Oven	Pass	Pass	Pass	Pass	Failed		Pass	Failed	Pass	Failed
Tensile strength, psi.	1990	4150	3700	1270	1830		3970	1960	3050	2520
Modulus @ 300% E, psi.	1590	360	1515	1030			-	-	2670	1930
Elongation, %	345	745	615	350	265		270	215	325	360
Hardness, Shore A	50	53	65	59	65		80	70	73	71

* Test discontinued after 30 days exposure.

TABLE VII
PHYSICAL PROPERTIES OF POLYESTER URETHANE A VULCANIZATE DURING
THREE YEARS INDOOR AND OUTDOOR STORAGE AT ROCK ISLAND, ILLINOIS

COMPOUND NUMBER Z60	INDOOR SHELF STORAGE					
	ORIGINAL	3 MONTHS	6 MONTHS	12 MONTHS	24 MONTHS 36 MONTHS	
Properties Measured:						
Tensile strength, psi.	4090	3630	3620	3640	3360	2990
Modulus @ 300% E, psi.	1500	1520	1560	1400	1140	820
Elongation, %	600	590	630	630	700	780
Hardness, Shore A	64	65	65	64	60	58
Strain, % E @ 400 psi.	163	161	156	165	186	227
Flexibility, D1043, °F	-40	-40	-40	-41	-40	-40
Brittleness, D746, °F	Below -80	Below -80	Below -80	Below -80	Below -80	Fails -80
OUTDOOR STORAGE						
Tensile strength, psi.	3810	4000	3760	3460	3260	2760
Modulus @ 300% E, psi.	1440	1300	1600	1360	1000	1040
Elongation, %	620	630	600	610	630	680
Hardness, Shore A	65	65	66	66	63	64
Strain, % E @ 400 psi.	163	156	144	136	192	211
Flexibility, D1043, °F	-40	-40	-40	-39	-32	-34
Brittleness, D746 °F	Below -80	Below -80	Below -80	Below -80	Failed -80	Failed -80



POLYESTER URETHANE VULCANIZATE Z129
BEFORE AND AFTER EXPOSURE OVER WATER AT 158°F

	A Original	B After 14 Days Exp.	C After 17 Days Exp.
Tensile strength, psi.	4490	970	Too soft
Modulus @ 300% E, psi.	3150	150	and gummy
Elongation, %	450	1020	too test.
Hardness, Shore A	71	33	

TABLE VIII
STRESS-STRAIN PROPERTIES OF URETHANE VULCANIZATES EXPOSED OVER WATER @ 158°F

	ORIGINAL	EXPOSURE TIME IN DAYS												
		3	7	10	14	16	21	28	35	42	49	56	63	70
Z120 Polyester Urethane A														
Tensile Strength, psi.		3350	2890	2360	970	Gummy								
Modulus @ 300% E, psi.		2170	1360	960	150	No								
Elongation, %		515	685	720	1020	Test								
Hardness, Shore A		71	65	60	51	33								
Z129G Polyester Urethane A plus PCD 4 pph Rubber														
Tensile Strength, psi.		3910	3570	3720	3070		2960	2740	2430	2170	Gummy			
Modulus @ 300% E, psi.		2920	2600	2530	2000		1870	1650	1180	890	No			
Elongation, %		465	435	440	525		520	530	590	630	Test			
Hardness, Shore A		72	68	69	70	67	66	62	61	59				
Z121 Polyester Urethane B Buflur Cure														
Tensile Strength, psi.		2790	1650	730	110									
Modulus @ 300% E, psi.		1740	980	330										
Elongation, %		460	460	465	485									
Hardness, Shore A		80	56	60	52	34								
Z121G Polyester Urethane B plus PCD 4 pph Rubber														
Tensile Strength, psi.		2330	1700	1180	800		100							
Modulus @ 300% E, psi.		1830	1010	720	430		400							
Elongation, %		585	570	530	500		400							
Hardness, Shore A		80	61	62	55	42	40							
Z121C Polyester Urethane B Peroxide Cure														
Tensile Strength, psi.		3300	2700	2250	1640		660							
Modulus @ 300% E, psi.		245	240	245	240		215							
Elongation, %		64	64	56	52		45							
Hardness, Shore A		73												
Z121CG Polyester Urethane B plus PCD 4 pph Rubber														
Tensile Strength, psi.		3680	3130	3070	3450		2790	1740						
Modulus @ 300% E, psi.		300	275	265	320		260	260						
Elongation, %		71	66	66	62		60	56						
Hardness, Shore A														
Z76CWF Polyester Urethane C Peroxide Cure														
Tensile Strength, psi.		2900			1910		700							
Modulus @ 300% E, psi.		1360			930		625							
Elongation, %		410	530		570									
Hardness, Shore A		67	66		46									

	EXPOSURE	TIME	IN	DAYS	
7	10	14	16	21	28
2	10	14	16	21	28
3	10	14	16	21	28
4	10	14	16	21	28
5	10	14	16	21	28
6	10	14	16	21	28
7	10	14	16	21	28
8	10	14	16	21	28
9	10	14	16	21	28
10	10	14	16	21	28
11	10	14	16	21	28
12	10	14	16	21	28
13	10	14	16	21	28
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64	10	14	16	21	28
65	10	14	16	21	28
66	10	14	16	21	28
67	10	14	16	21	28
68	10	14	16	21	28
69	10	14	16	21	28
70	10	14	16	21	28
71	10	14	16	21	28
72	10	14	16	21	28
73	10	14	16	21	28
74	10	14			

[illegible]

PCD displays good, although diminishing, physical property retention for 42 days over water at 158°F but is abruptly rendered soft, tacky and unsuitable for practical testing during the next seven days exposure. A bent loop of polyester urethane A, containing PCD, exposed over water at 158°F for 42 days, is illustrated in Figure 9 for comparison with an uninhibited specimen after 14 days in test. Physical property retention of polyether urethane vulcanizates utilizing a sulfur cure is not significantly affected by PCD whereas the peroxide cured vulcanizate displays impaired aging resistance with the presence of PCD.

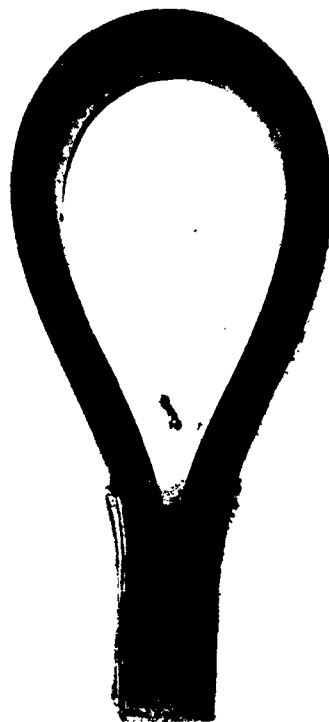
Stressed polyester urethane vulcanizates employing a peroxide curative displayed cracks within 28 days after initiation of tests conducted at room temperature. Typical cracking of the specimens is represented by Figure 10. Continued exposure eventually causes complete separation of the stressed specimens. Examination of cracked specimens under 20X magnification reveals the presence, in some instances, of microorganisms on the rubber.

Cracking was not manifest in either sulfur vulcanized polyester urethane or any of the polyether urethane vulcanizates even after eight months of testing. A summary of rubbers tested and their resistance to cracking is presented in Table IX.

Polyester urethane A specimens contained in sealed, sterile and nonsterile test tubes exhibited no sign of cracking during four months conditioning over water, whereas specimens contained in an open tube with access to circulating air, were cracked.

Test data concerning immersion of polyester urethane A vulcanizates in water at 158°F indicate that a significant improvement of physical property retention is achieved by the use of PCD. Room temperature water immersion tests show that the PCD inhibited vulcanizate displays somewhat better property retention than does the control. However, dumbbell specimens containing PCD were cracked within 21 days exposure over water at room temperature whereas specimens containing no PCD remain crack-free for as long as 35 days. Substantiating data is provided in Table X.

Vulcanizates of polyester urethane A utilizing experimental curatives displayed no better resistance to the deleterious effects of high humidity than did the commonly used dicumyl peroxide cured vulcanizate. The use of recrystallized dicumyl peroxide is of no advantage in this respect.

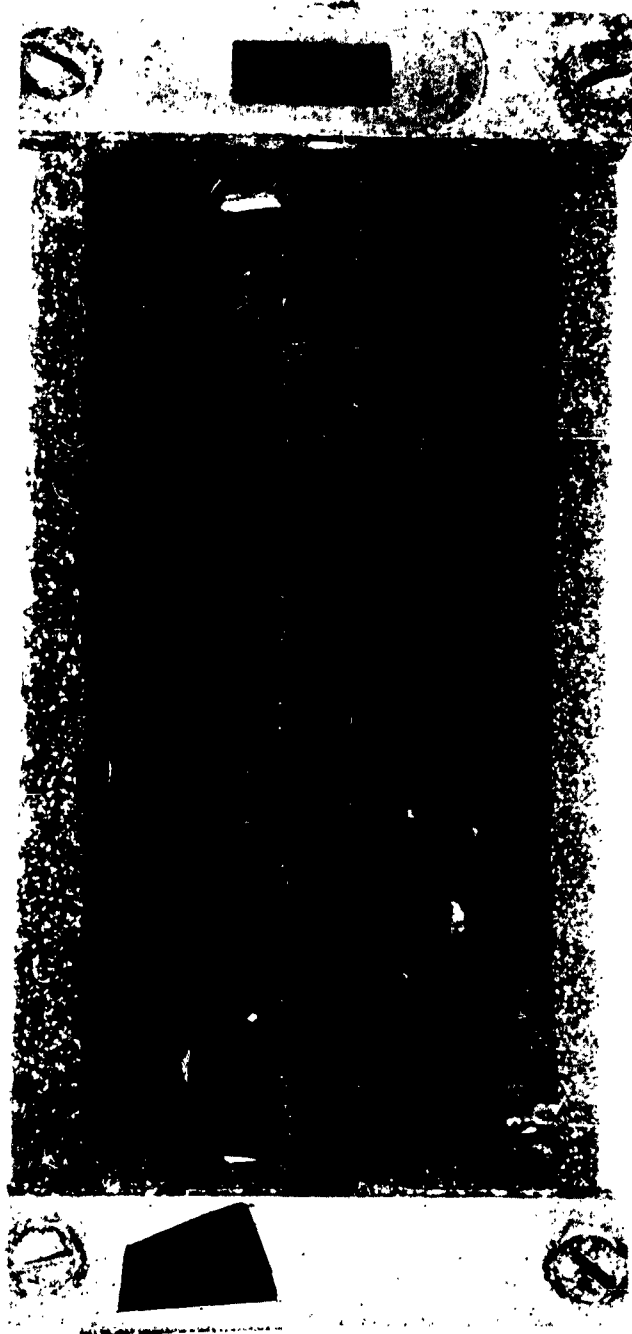


Z129G



Z129

APPEARANCE OF POLYESTER URETHANE A VULCANIZATE
Z129G AFTER 42 DAYS OVER WATER AT 158°F COMPARED WITH
UNINHIBITED VULCANIZATE Z129 AFTER 14 DAYS IN TEST



APPEARANCE OF POLYESTER URETHANE A VULCANIZATE Z60F5
AFTER 42 DAYS OVER WATER AT ROOM TEMPERATURE

TABLE IX
OBSERVATIONS CONCERNING URETHANE VULCANIZATES EXPOSED OVER
WATER AT ROOM TEMPERATURE

<u>COMPOUNDS TESTED</u>	<u>TIME TO FIRST CRACK</u>	<u>CURE SYSTEM</u>	<u>SPECIAL ADDITIVES</u>
Polyether Urethanes			
Z46E	OK 8 months; test continues	Isocyanate	None
Z51	"	Sulfur	"
Z51G	"	Sulfur	PCD 4 pph rubber
Z51C	"	Peroxide	None
Z51CG	"	Peroxide	PCD 4 pph rubber
Polyester Urethanes			
Z60F5	28 days	Peroxide	None
Z129G	21 days	"	PCD 4 pph rubber
Z129GM20	11 days	"	PCD 4 pph rubber plus Pentachlorophenol 0.5 pph rubber
Z129GM17	OK 80 days, test continues	"	PCD 4 pph rubber plus Pentachlorophenol 1 pph rubber
Z129GM18	OK 80 days, test continues	"	PCD 4 pph rubber plus Pentachlorophenol 2 pph rubber
Z129GM19	45 days	"	PCD 4 pph rubber plus Pentachlorophenol 3 pph rubber
Z129M20	OK 30 days, test continues	"	Pentachlorophenol 0.5 pph rubber
Z60D4	28 days	"	None
Z121	OK 8 months; test continues	Sulfur	"
Z121C	28 days	Peroxide	"
Z76CDF	28 days	Peroxide	"
Z124F	37 days	Peroxide	"

TABLE X
PHYSICAL PROPERTIES OF POLYESTER URETHANE A VULCANIZATES
IMMERSED IN WATER AT 158°F AND ROOM TEMPERATURE

	IMMERSED IN WATER AT 158°F				
	ORIG.	7 DAYS	14 DAYS	21 DAYS	28 DAYS 35 DAYS 42 DAYS 84 DAYS
Z129					
Tensile strength, psi.	4490	2550	740		
Modulus @ 300% E, psi.	3150	1350			
Elongation, %	450	650	1050		
Hardness, Shore A	71	58	28		
Volume change, %		7	13		
Z129G (4 Parts PCD)					
Tensile strength, psi.	4270	3300	3010		
Modulus @ 300% E, psi.	2820	2520	2080		
Elongation, %	485	380	450		
Hardness, Shore A	71	68	66		
Volume change, %		4	5		
OVER WATER @ ROOM TEMPERATURE					
Z129					
Tensile strength, psi.	4490	4510	4290	4000	4000 4120 560*
Modulus @ 300% E, psi.	3150	3230	3320	3450	3850 3490
Elongation, %	450	400	410	380	400 395
Hardness, Shore A	71	70	71	71	73 71 69
Z129G (4 Parts PCD)					
Tensile strength, psi.	4270		4340	640*	1960*
Modulus @ 300% E, psi.	2820		3010		
Elongation, %	485		410	110	200
Hardness, Shore A	71		71	71	71
IMMERSED IN WATER @ ROOM TEMPERATURE					
Z129					
Tensile strength, psi.	4490	4220	4370	4200	4330
Modulus @ 300% E, psi.	3150	3040	3190	3460	3290
Elongation, %	450	400	405	400	400
Hardness, Shore A	71	70	72	71	73
Z129G (4 Parts PCD)					
Tensile strength, psi.	4270		4540	4300	4750
Modulus @ 300% E, psi.	2820		3080	3100	3350
Elongation, %	485		420	400	440
Hardness, Shore A	71		71	71	72

* Specimens (unstressed) display cracking.

Likewise, gum compounds and EPC, MT, FEF or aluminum oxide reinforced vulcanizates are equally susceptible to degradation.

Pentachlorophenol has been found to be the only additive capable of inhibiting cracking of polyester urethane A vulcanizates conditioned under stress over water at room temperature. Vulcanizates containing 4 pph rubber PCD in admixture with 1 or 2 pph rubber pentachlorophenol have remained crack-free for 80 days (test continues) whereas a similar vulcanizate containing no special additive is cracked withing 28 days. Other vulcanizates containing the same admixture, where the pentachlorophenol is used at 0.5 or 3 pph rubber, display cracks sooner (14 days) than the control which contains no special additive.

It is to be noted from data presented in Table XI that pentachlorophenol has a somewhat adverse effect on original stress-strain properties of the vulcanizates and, in addition, has an even greater adverse effect on the stability of rubber exposed over water at 158°F.

DISCUSSION

High humidity had not heretofore been considered a significant factor in the life expectancy or degradation of rubber items. In fact, previous experience has shown that, in general, humidity was of no consequence in the aging of rubber.⁴ However, this investigation has shown that the service life of polyester urethane items is drastically shortened by high humidity.

Observations of test bellows exposed at Fort Amador revealed that polyester urethane A bellows display cracking and softening similar to that produced by both of the previously described laboratory tests. It appears that two separate modes of degradation are occurring simultaneously and independently of one another. This is demonstrated by rubber specimens cracking in the room temperature test prior to softening whereas specimens exposed in the 158°F test become soft and tar-like without cracking.

Cracking of the rubber under laboratory conditions may have been induced by airborne aerobic microorganisms or enzymes, since it has been shown that specimens do not crack when immersed in water or are conditioned over sterile or nonsterile water with a lack of circulating air. Aerobic microorganisms are dependent upon an adequate supply of air for respiration and thrive in the presence of high humidity. Specimens exposed at 158°F would not and do not crack since this temperature is sufficient to prevent microorganism germination. Furthermore, polyester urethane B vulcanizate

TABLE XI

**PHYSICAL PROPERTIES OF URETHANE VULCANIZATES EXPOSED
OVER WATER AT 158°F**

<u>Zl29 - Control</u>	<u>EXPOSURE TIME IN DAYS</u>					
	<u>0</u>	<u>3</u>	<u>7</u>	<u>14</u>	<u>21</u>	<u>28</u>
Tensile, psi.	4490	3350	2890	970	Too	
Modulus @ 300% E, psi.	3150	2170	1360	150	Gummy	
Elongation, %	450	515	685	1020	To	
Hardness, Shore A	71	65	60	33	Test	
<u>Zl29G - PCD 4 pph Rubber</u>						
Tensile, psi.	4220	3910	3570	3070	2960	2740
Modulus @ 300% E, psi.	3070	2520	2600	2000	1870	1650
Elongation, %	425	465	435	525	520	530
Hardness, Shore A	72	68	69	67	66	62
<u>Zl29M20 Pentachlorophenol 0.5 pph Rubber</u>						
Tensile, psi.	3480	3390	2360	Too		
Modulus @ 300% E, psi.	1560	1900	880	Gummy		
Elongation, %	540	510	675	To		
Hardness, Shore A	66	65	43	Test		
<u>Zl29GM20 Pentachlorophenol 0.5 pph Rubber plus PCD 4 pph Rubber</u>						
Tensile, psi.	3690	3750	3450	3130	2530	2140
Modulus @ 300% E, psi.	2090	2540	1760	1540	810	500
Elongation, %	565	440	620	625	815	895
Hardness, Shore A	69	70	67	68	57	53
<u>Zl29GM17 Pentachlorophenol 1 pph Rubber plus PCD 4 pph Rubber</u>						
Tensile, psi.	3730	2700	2550	2650	1640	Too
Modulus @ 300% E, psi.	2110	1080	580	1010	350	Gummy
Elongation, %	575	720	805	750	1000	To
Hardness, Shore A	70	62	62	62	53	Test
<u>Zl29M18 Pentachlorophenol 2 pph Rubber</u>						
Tensile, psi.	3180	2300	840	Too		
Modulus @ 300% E, psi.	1400	620		Gummy		
Elongation, %	720	960	1300	To		
Hardness, Shore A	64	58	41	Test		
<u>Zl29GM18 Pentachlorophenol 2 pph Rubber plus PCD 4 pph Rubber</u>						
Tensile, psi.	2980	2670	2140	1980	Too	
Modulus @ 300% E, psi.	1230	800	510	530	Gummy	
Elongation, %	730	845	925	955	To	
Hardness, Shore A	67	61	53	55	Test	
<u>Zl29GM19 Pentachlorophenol 3 pph Rubber plus PCD 4 pph Rubber</u>						
Tensile, psi.	3490	2680	1980	1050	Too	
Modulus @ 300% E, psi.	1520	910	420		Gummy	
Elongation, %	670	820	1100	1250	To	
Hardness, Shore A	68	60	54	44	Test	

with a sulfur cure system displays resistance to cracking but its peroxide cured counterpart does crack in the room temperature test. However, the same vulcanizates are both degraded over water at 158°F. The biocidal action of sulfur, as demonstrated in this instance, is well known and has been extensively exploited to this end. Unfortunately, sulfur, even 0.32 pph rubber, is incompatible with the required peroxide cure system of polyester urethane A compounds.

The independent stabilizing efficiency of PCD and pentachlorophenol (the former retarding chain scission or hydrolysis at elevated temperature and the latter preventing cracking) tends to substantiate that two separate forms of deterioration are taking place.

Although PCD fails to eliminate hydrolysis of polyester urethane A in laboratory tests, this material may prove satisfactory in stabilizing end items for field service where temperature and humidity will not be as severe as in accelerated tests.

The apparent interference of pentachlorophenol with PCD's stabilizing action points out the need for an efficient biocidal agent that will function properly without producing undesirable side effects in polyester urethane vulcanizates.

This investigation has shown that polyester urethane D possesses good hydrolytic stability in both outdoor and laboratory tests. Polyester urethane D vulcanizate's usefulness cannot be completely assessed until the cause and elimination of cracking during outdoor exposure is determined.

Properly compounded SBR, nitrile, polybutadiene and chloroprene vulcanizates are suitable for use in environments such as the Canal Zone.

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3. Evaluation		3. Evaluation	3. Evaluation
4. Properties - General		4. Properties - General	4. Properties - General
Polyester urethane elastomeric end items have ex- hibited severe breakdown after service periods of as little as one to two years. The rubber becomes tar-like within an unreasonably brief period of service and is rendered unfit for further use. Duplication of this rubber failure under laboratory conditions was accomplished by exposing specimens over water at 150°F and at room temperature. Two modes of degradation appear to occur simultaneously and independently of one another: i.e., hydrolysis or chain scission of specimens exposed at 150°F and microorganism attack of the specimens at room tem- perature accompanied by hydrolysis. A polycarbo- nate and pentachlorophenol demonstrate their dimide and pentachlorophenol demonstrate their (Cont.) over		DISTRIBUTION: Copies obtainable from DOC	
AD Rock Island Arsenal Laboratory, Rock Island, Illinois PERFORMANCE OF URETHANE VULCANIZATES IN ENVIRON- MENTS OF HIGH HUMIDITY, by F. B. Testroet		UNCLASSIFIED	
1. Elastomers		1. Elastomers	1. Elastomers
2. Polyurethane		2. Polyurethane	2. Polyurethane
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Polyether urethane vulcanizates, containing no special additives, are not adversely affected by environments of high humidity.

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