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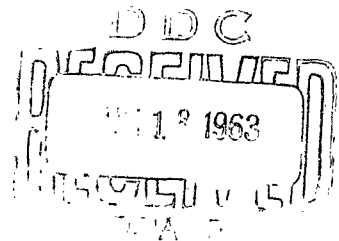
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TECHNICAL REPORT

SYNTHESIS AND EVALUATION OF POLYURETHANE ELASTOMERS

By

W. M. Veroeven

NO OTS

Department of the Army Project No. 1-D-S-21801-D-262

AMC Code No. 5521.12.255

Report No. 63-1242 Copy No. _____

IEL No. 1-8-107-3 Date 17 April 1963

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By

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Laboratory Director

17 April 1963

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Rock Island Arsenal
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ABSTRACT

Commercially available castable, millable and/or injection moldable polyurethane elastomers were evaluated to determine their characteristics and potential use for Army applications.

Experimental polyurethanes were synthesized using various polyols, polyamines and stabilizing agents in an effort to improve the heat stability in the range 149 to 204°C.

It was shown that retention of strength at elevated temperature can be improved by increased functionality in the system, but only at the expense of the elastic characteristics of the material.

The increased crosslinking of the millable gums offers the most promise for heat resistant polyurethanes.

RECOMMENDATIONS

It is recommended that the following be considered for future R&D work in polyurethanes.

1. Conduct compounding studies with various millable gum urethanes (both sulfur and peroxide vulcanizable) and blends with other polymers. Determine the effects of fillers, curatives and additives on the thermal stability and hysteresis of these compounds.

2. Investigate additives for improving the high temperature and hydrolytic stability of polyurethanes.

SYNTHESIS AND EVALUATION OF POLYURETHANE ELASTOMERS

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SYNTHESIS AND EVALUATION OF POLYURETHANE ELASTOMERS

OBJECT

To synthesize new heat stable urethanes and to study ways for compounding commercial urethanes to improve their heat stability. To evaluate commercially available castable and injection moldable polyurethanes for potential use in Military applications.

INTRODUCTION

Polyurethane elastomers are well known for their excellent strength, resistance to abrasion, ozone and oil and ability to withstand low temperature impact. These properties and previous work at this Arsenal⁽¹⁾ suggested the further investigation of urethanes for Army application. The present report is the result of such investigations.

Interest for improving urethane properties at elevated temperatures was prompted by previous work done by Detroit Arsenal^(2,3,4); and, Dayco Corporation⁽⁵⁾ in conjunction with Detroit Arsenal, on the use of polyurethanes for tracked vehicle pads. This work indicated that although urethanes exhibit excellent tensile and abrasion properties at room temperature, their use in tracked vehicle pads appears to be hindered by a reduction of these properties at the elevated temperatures attained at the surface of the pads during vehicle operation. Thus, further investigation of newly developed heat stable experimental polyurethanes prepared at this Arsenal and by Dayco Corporation was undertaken. Attempts were made to improve tensile strength properties at elevated temperatures in commercially available urethanes by screening various polyfunctional crosslinkers in castable polyurethanes. Various heat stabilizers were evaluated in a millable gum urethane in an attempt to improve resistance to thermal degradation.

Commercially available castable and injection moldable urethanes were evaluated to determine processing characteristics and properties obtainable.

PROCEDURE

Hydroxy terminated polyesters were prepared by the addition polymerization of epsilon-caprolactone initiated with d-sorbitol or the condensation polymerization of isophthaloyl chloride with a diol. Fumaric acid was used to introduce unsaturation.

Preparation of experimental polyesters and polyurethanes was carried out in a 500 ml. resin kettle fitted with a thermometer, stirrer, gas inlet and outlet. Temperature was controlled with a heating mantle and a Variac. Standard laboratory procedures were followed in carrying out the reactions.

All commercial compounds discussed were prepared following the manufacturers recommendations. Any changes made in these procedures have been noted in the text or tables.

Specimens were tested in accordance with ASTM procedures⁽⁶⁾ unless otherwise noted. Tensiles at elevated temperatures were determined on specimens conditioned at the test temperature for six minutes before being tested.

RESULTS AND DISCUSSION

Formulations and physical properties obtained on experimental urethanes are summarized in Table I. The Z130 formulation was found to exhibit the best properties of all the experimental urethanes evaluated. The usefulness of such a urethane, however, would be doubtful because of its low initial tensile strength. Its use would also be hampered by a very short pot life of about one minute after crosslinker addition. The pot life problem was overcome (sufficiently to permit casting of test pads) by lowering the prepolymer temperature prior to addition of the crosslinker.

Formulation Z130D5C3F3 in Table I was evaluated in an attempt to crosslink the urethane gum, through the unsaturation present, by means of a conventional sulfur curing system. A possible explanation for the lack of cure might be found in a report by Briggs, Erickson and Fuller⁽⁷⁾ in which they state that esters are rapidly hydrolyzed by zinc and magnesium oxides.

Formulations and physical properties for commercial urethanes compounded for heat stability are summarized in Table II. Tensile values exhibited at ambient and elevated temperatures by compound Z130D9C6F5 showed the most promise of all the compounds evaluated.

Table III lists heat stabilizers previously screened in SBR at this Arsenal^(8,9) and found to have varying degrees of effectiveness in this polymer. Evaluation was made by incorporating 5 parts of the stabilizer into a basic urethane compound comprised of 100 parts of Polymer H, 25 parts of MAF carbon black, 4 parts of 40% active dicumyl peroxide and 0.2 parts of stearic acid. Test pads were cured 30 minutes at 160°C. Many of the stabilizer compounds were found to retard or destroy the peroxide cure. None of the compounds listed in Table III improved the heat aging stability of the basic urethane compound after aging 21 days at 149°C in an air oven.

TABLE I

PREPARATION AND PHYSICAL PROPERTIES FOR EXPERIMENTAL POLYURETHANES

	Z130	Z130D	Z130D1	Z130D2C	Z130D3C1F	Z130D4C2F2	Z130D5C3F3
Polyester of epsilon-caprolactone - 207.9; d-sorbitol - 8.19; zinc borate 0.21	100	100	100				
Polyester of isophthaloyl chloride - 50.8; p-phenylenedithoxydiethanol - 4.95; 1,10-decanediol - 44.55; o-dichlorobenzene - 6.52				100	100		
Polyester of isophthaloyl chloride - 40.6; fumaric acid - 5.8; p-phenylenedithoxydiethanol - 4.95; 1,10-decanediol - 44.55; hydroquinone 0.1; o-dichlorobenzene - 6.52						100	
1,5-naphthalene diisocyanate	35						
P,p'-diphenylmethane diisocyanate		41.7	29.5	8	4	10	10
Toluene-2,4-diisocyanate			10.4				
1,4-butanediol	10.4	10.4		3.7	10	7	
4,4' methylene bis (2 chloroaniline)						Trace	1
Dicumyl peroxide (40%)							5
Stearic acid							1.5
Zinc oxide							1
Sulfur							1.5
Benzothiazyl disulfide							1
Tetramethyl thiuram disulfide							1.5
HAF Carbon Black					50	10	40
EPC Carbon Black							30 min. @153°C
Cure	16 hrs. @110°C	16 hrs. @110°C	16 hrs. @110°C	3 hrs. @110°C	2 hrs. @160°C	2 hrs. @153°C	
Post cure	14 days @25°C & 50% rel- ative humidity	14 days @25°C & 50% rel- ative humidity	14 days @25°C & 50% rel- ative humidity				
Tensile at 25°C	2620	1270	880	160*			
Hardness, Shore A	87	74	56	31	Resisted melting after 24 hrs. @249°C in an air oven	270	Failed to cure
Tensile at 100°C	1900	330	290	-		54	
Tensile at 149°C	980	**	260	-		-	
Tensile at 204°C	490	**	190	-		-	

*Pulled on an Instron tester using 1/4 inch wide specimens and crosshead speed of 20 inches/min.

**Properties too poor to permit testing.

TABLE II

**FORMULATIONS AND PHYSICAL PROPERTIES FOR COMMERCIAL
URETHANES COMPOUNDED FOR HEAT STABILITY**

	(1) Z130D6C4	Z130D7C5F4	(2) Z130D8C4	Z130D9C6F5	Z130D10C7	Z130D10C8	Z130D10C9	Z130D10C10
Polymer A (Injection moldable and millable polyester urethane)**	100							
Polymer B (Millable polyester urethane)		100						
Polymer C (Injection moldable and millable polyester urethane)			100					
Polymer E (Castable polyether urethane)					100	100	100	100
Polymer H (Millable polyester urethane)				100				
FEF Carbon black				25				
RPC Carbon black		20		0.2				
Stearic acid								
Barium stearate	1	1	1			1.12		
1,4-Butanediol								
1,2,6-Hexametriol							1.12	1.12
Trimethanolpropane								8.3
4,4' Methylene bis (2 chloroaniline)					11	8.3		
Di-cumyl peroxide (40%)								
Di-cumyl peroxide (95%)		3		4				
Polymethylene polyphenyl isocyanate	10		10					
Cure	60 min. @177°C	45 min. @153°C	60 min. @177°C	30 min. @160°C	5 hrs. @110°C	5 hrs. @110°C	5 hrs. @110°C	5 hrs. @110°C
Post cure	7 days @24°C & 50% rel- ative humidity		7 days @24°C & 50% rel- ative humidity		14 days @24°C & 50% rel- ative humidity	14 days @24°C & 50% rel- ative humidity	14 days @24°C & 50% rel- ative humidity	14 days @24°C & 50% rel- ative humidity
Tensile, psi at 25°C	2370	5050	3530	4000	2630	3170	1600	2220
Tensile, psi at 100°C	300	1420	320	1780	1320	1330	830	1190
Tensile, psi at 149°C	210	480	*	1160	850	650	640	530
Tensile, psi at 204°C	100	200	*	450	S.M.	S.M.	S.M.	S.M.
Hardness, Shore A	78	52	79	62	84	89	79	91

(1) Mill rolls heated with hot water during milling process.

(2) Mill rolls heated with steam during milling process.

* Properties too poor to permit testing.

** This and other coded materials are identified in Code Sheet at end of report.

S.M., Specimens Melt.

TABLE II (Cont.)

	Z130D10C11	Z130D10C12	Z130D10C13	Z130D10C14	Z130D10C15	Z130D10C16	Z130D10C17
Polymer E (Castable polyether urethane)	100	100	100	100	100	100	100
Polymer F, (Castable urethane)							
1,2,6-Hexanetriol			4.47	4.47			
Trimethanolpropane							
1,2,3 Propanetriol	0.767				3.07		
N,N,N',N'-tetrakis (2-hydroxy propyl) ethylenediamine						7.30	
Hydroxyethyl cellulose		10					
4,4' Methylene bis (2 chloroaniline)	8.3	9.4					29
Citric acid		0.040					
Cure	5 hrs. @110°C	3 hrs. @100°C	24 hrs. @110°C	24 hrs. @110°C	24 hrs. @110°C	24 hrs. @110°C	60 min. @100°C
Post cure	14 days @24°C & 50% relative humidity	14 days @24°C & 50% relative humidity	14 days @24°C & 50% relative humidity	14 days @24°C & 50% relative humidity	14 days @24°C & 50% relative humidity	14 days @24°C & 50% relative humidity	14 days @24°C & 50% relative humidity
Tensile, psi at 25°C	3680	2160	340	560	1500	250	6620
Tensile, psi at 100°C	1330	1100	270	370	1040	250	3730
Tensile, psi at 149°C	610	630	300	250	300	*	1790
Tensile, psi at 204°C	S.M.	S.M.	230	290	S.M.	*	*
Hardness, Shore A	92	94	57	57	58	58	-
Hardness, Shore D	-	-	-	-	-	-	68

TABLE III

HEAT STABILIZERS EVALUATED IN A MILLABLE GUM URETHANE

Cadmium-barium soap chelator mixture

Calcium ricinoleate-barium stearate-ethylenediamine
tetraacetic acid mixture

Barium stearate-calcium myristate mixture

Pentaerythritol ester of saturated fatty acids

Resin modified polyester

Ferric oxide

Metallic soap

Arsenic pentoxide

Stannous butyl benzoate

Polymeric secondary amine from p-phenylenediamine and
p-dichlorobenzene

Phenyl didecylphosphate

Sec-amine from resorcinol m-phenylenediamine

Barium/cadmium salt ferrocenoylpropionic acid

Nickel chelate of 8-hydroxyquinoline

Urethane materials listed in Table IV were evaluated to obtain a background of knowledge on processing characteristics and properties obtainable with injection moldable and castable urethanes. From the data listed it can be seen that the injection moldable and castable urethanes in general display the excellent room temperature tensile properties usually associated with polyurethanes. These polymers also display good solvent resistance except in polar type brake fluid where degradation is quite severe. Compression set is high as is frequently the case with urethanes.

TABLE IV

**FORMULATIONS AND PHYSICAL PROPERTIES FOR COMMERCIAL
CASTABLE AND INJECTION MOLDABLE URETHANES**

	Polymer A - 100 Barium stearate - 1 Compression molded	Polymer B - 100 EPC Carbon Black - 20 Barium stearate - 1 Dicumyl peroxide (95%) - 3 Compression molded	Polymer C - 100 Barium stearate - 1 Compression molded	Polymer D - 100 Injection molded	Polymer E - 100 4,4' methylene bis (2 chloro- aniline) - 11	Polymer F - 100 4,4' methylene bis (2 chloro- aniline) - 29	Polymer G - 100 1,4-Butanediol - 6.4	"One Shot" urethane prepared and supplied by manufacturer
ORIGINAL								
Tensile, psi	5170	5660	6760	3870	5470	8890	7350	2230
100% M, psi	590	290	1570	1275	1150	3810	610	1210
200% M, psi	740	-	2090	1770	-	6120	890	1540
300% M, psi	910	2190	2960	2420	2010	8890	1260	1810
% Elongation	665	500	540	480	500	300	640	405
Hardness								
Shore A	75	62	84	90	94	95	78	91
Shore D						68		
Compression Set, %								
22 hrs. @70°C	-	29	-	-	25*	-	26*	-
70 hrs. @100°C	-	82	-	-	-	-	-	106
94 hrs. @-55°C	-	-	-	-	-	-	-	102
Change in Volume, % and Hardness (ΔH)								
ASTM #3 Oil	+5	+8	+3	-	+18	+7	+4	+12
70 hrs. @100°C	ΔH(-11)	ΔH(-10)	ΔH(0)	-	ΔH(-7)	ΔH(-7)	ΔH(-6)	-
Di Octyl Sebacate	+3	+4	+1	-	+17	+7	+1	-
70 hrs. @100°C	ΔH(-10)	ΔH(-7)	ΔH(0)	-	ΔH(-5)	ΔH(-5)	ΔH(-5)	-
Brake Fluid	Disinte-	Disinte-	Disinte-	-	-	Disinte-	Disinte-	-
70 hrs. @100°C	grated	grated	grated			grated	grated	
ASTM Reference	+14	+16	-	-	+29	+16	+11	-
Fuel B	ΔH(0)	ΔH(-3)	-	-	ΔH(-5)	ΔH(-7)	ΔH(-5)	-
7 days @24°C						(-13)		
Air Oven Aging								
70 hrs. @100°C								
Tensile, psi					5070			1920
100% M, psi					1220			1240
200% M, psi					1630			1460
300% M, psi					2110			1590
% Elongation					460			405
Hardness, Shore A					90			89
Low temperature brittleness	-	-	-	-	-	-	-	5 Break
ASTM D746 @-55°C								

ΔH - Change in Shore A hardness.

* - Obtained on laminated specimen.

Although these observations are quite general and based upon properties summarized in Table IV, they should act as a guide and not as a limiting factor of properties available. Various grades and classes of injection moldable and castable urethanes are available to provide a wide latitude in properties. With certain castable urethanes, a wide range of properties may be obtained by the type of chemical reagents selected and the manner in which they are formulated. Proper selection and compounding can, therefore, often produce an injection moldable or castable urethane having physical properties desired for specific end-use.

CONCLUSIONS

Polyurethanes based upon d-sorbitol/epsilon-caprolactone polyester exhibit fair tensile properties at 204°C. Tensile strength at room temperature in these urethanes; however, is one-half to one-third that normally associated with urethanes. Castable pot life of such a resin would create a problem in any attempted practical applications.

Tensile at elevated temperatures can be improved in castable urethanes by the use of triols and higher functionality polyols. However, original physical properties are destroyed to such a great extent that their use would be of questionable value in an application such as track pads.

Millable gum urethanes appear to offer the greatest potential in the immediate future for good properties at room and elevated temperatures.

Castable and injection moldable urethanes offer a wide range of properties, applications and advantages for Army use.

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<u>Chemical Name or Description</u>	<u>Trade Name or Common Name</u>	<u>Source</u>
Polym̄er A (Injection moldable & millable polyester urethane)	Estane 5740X1	B.F. Goodrich Chemical Co.
Polymer B (Millable polyester urethane)	Estane 5740X2	"
Polymer C (Injection moldable & millable polyester urethane)	Estane 5740X7	"
Polymer D (Injection moldable polyester urethane)	Texin 192A	Mobay Chemical Company
Polymer E (Castable polyether urethane)	Adiprene L-100	E.I. Dupont de Nemours & Co.
Polymer F (Castable urethane)	Adiprene LD-315	"
Polymer G (Castable polyester urethane)	Multrathane F66	Mobay Chemical Company
Dicumyl Peroxide (95%)	Recrystallized Dicumyl Peroxide	Hercules Powder Company
4,4' Methylene bis (2 chloroaniline)	Moca	E.I. Dupont de Nemours & Co.
EPC Carbon Black	Kosmobile 77	United Carbon Co., Inc.
Brake Fluid	Wagner Brake Fluid	Wagner Electric Company

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CODE SHEET (Cont.)

<u>Chemical Name or Description</u>	<u>Trade Name or Common Name</u>	<u>Source</u>
Polymer H (Mellable polyester urethane)	Genthane S	General Tire & Rubber Co.
FEF Carbon Black	Philblack A	Phillips Chem. Co.
Dicumyl Peroxide (40%)	DiCup 40C	Hercules Powder Co.
Metallic Soap	Thermolite 12	General Electric
Cadmium-Barium Soap/Chelator mixture	Ferro 1825	Ferro Chemical Co.
Pentaerythritol ester of saturated fatty acids	Hercolube A	Hercules Powder Co.
Rosin modified polyester	Neolyn 40	Hercules Powder Co.
N-methyl-N,4-dini- trosoaniline	Elastopar	Monsanto Chemical Company
Hydroxyethyl Cellulose	Cellosize WP-09	Union Carbide Chemical Co.
N,N,N',N'-tetrakis (2-hydroxypropyl) ethylenediamine	Quadrol	Wyandotte Chem. Corp.
Polymethylene poly- phenyl isocyanate	Papi	Carwin Company
HAF Carbon Black	Philblack "0"	Phillips Chem. Co.
Benzothiazyl Disulfide	Altax	R.T. Vanderbilt Co.
Tetramethyl thiuram disulfide	Methyl tuads	R.T. Vanderbilt Co.

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