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DEVELOPMENT OF LOW VISCOSITY ALKANE-BASED URETHANE FOR CONNECTOR POTTING APPLICATIONS

By Donald E. Morris

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16. ABSTRACT

Two series of saturated hydrocarbon-based urethanes were prepared with isophorone diisocyanate and one series with methyl bis (4-cyclohexyl isocyanate). The urethanes with molecular weights as great as 2500 had viscosities low enough and a working life long enough to be used in potting, molding, and coating applications. Specimens were prepared and mechanical properties such as hardness, tensile strength elongation, and tear strength were determined. Thermomechanical properties (glass transition and expansion coefficient) and thermogravimetric properties were determined.

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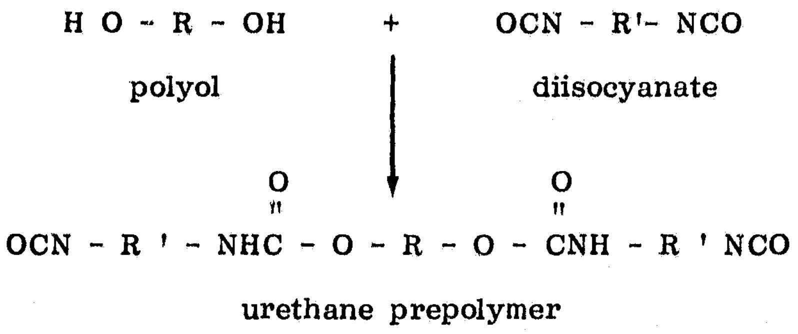
DEVELOPMENT OF LOW VISCOSITY ALKANE-BASED URETHANE FOR CONNECTOR POTTING APPLICATIONS

I. INTRODUCTION

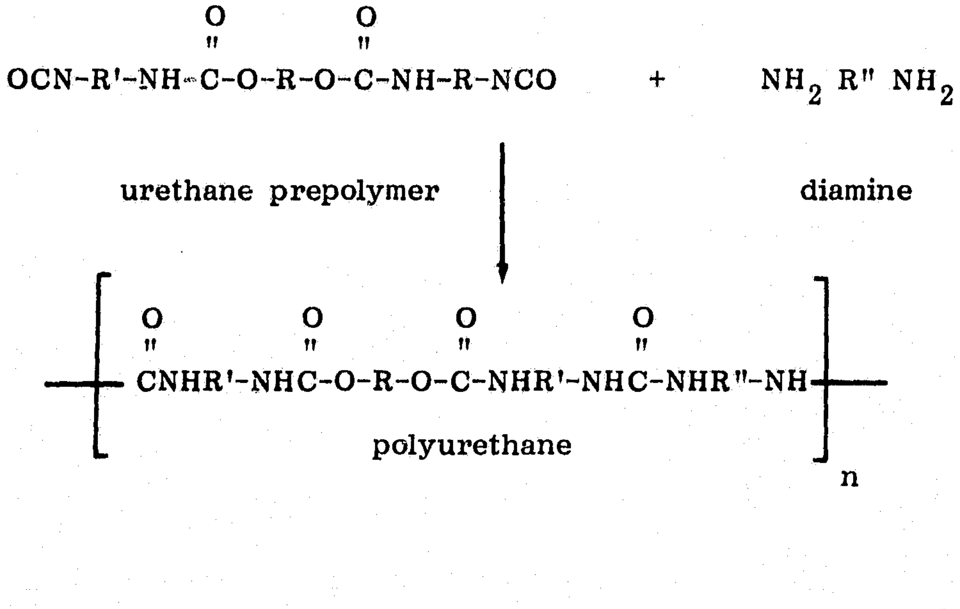
It was established in a previous Discretionary Fund task that alkane-based urethanes are hydrolytically and oxidatively stable and have excellent dielectric properties. However, the urethanes prepared from most commercially available alkane materials are very viscous liquids or semi-solids which cannot be easily used in potting applications. To take advantage of the inherently good properties of this type of urethane, this study has been directed toward the development of alkane-based urethanes with viscosities low enough to permit easy mixing with catalyst at room temperature, followed by out-gassing, and molding.

Polyurethanes are made essentially of three basic components: difunctional polyols, diisocyanates, and chain extenders. It is the interaction of these three components which produces the polyurethane:



$$\underset{\text{polyol}}{\mathrm{HO-R-OH}} + \underset{\text{diisocyanate}}{\mathrm{OCN-R'-NCO}} \longrightarrow \underset{\text{urethane prepolymer}}{\mathrm{OCN-R'-NH\overset{O}{\overset{\|}{C}}-O-R-O-\overset{O}{\overset{\|}{C}}NH-R'NCO}}$$

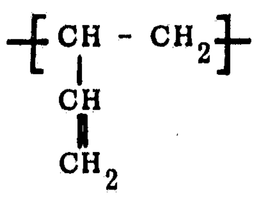
The urethane is cured with chain extenders which in this study, consisted of a diamine.



$$\underset{\text{urethane prepolymer}}{\mathrm{OCN-R'-NH-\overset{O}{\overset{\|}{C}}-O-R-O-\overset{O}{\overset{\|}{C}}-NH-R-NCO}} + \underset{\text{diamine}}{\mathrm{NH_2R''NH_2}} \longrightarrow \underset{\text{polyurethane}}{\left[\mathrm{\overset{O}{\overset{\|}{C}}NHR'-NH\overset{O}{\overset{\|}{C}}-O-R-O-\overset{O}{\overset{\|}{C}}-NHR'-NH\overset{O}{\overset{\|}{C}}-NHR''-NH}\right]_n}$$

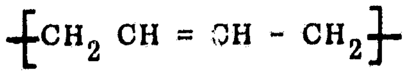
Each of these components can influence the viscosity of the reaction mixture. Pot life, or time interval before the material is too viscous to use, will depend upon the relative reactivity of the components, the molecular weight, and the molecular structure.

Most of the alkane-based urethanes used in this study were prepared by the hydrogenation of hydroxy-terminated polybutadiene. The high viscosity of the resulting urethane is due to crystallinity and to the high molecular weight of the polybutadiene used. The preparation of hydroxy-terminated polybutadiene by anionic techniques lead to both 1,2- and 1,4- additions of butadiene:



$-\!\!\left[CH(CH{=}CH_2) - CH_2\right]\!\!-$

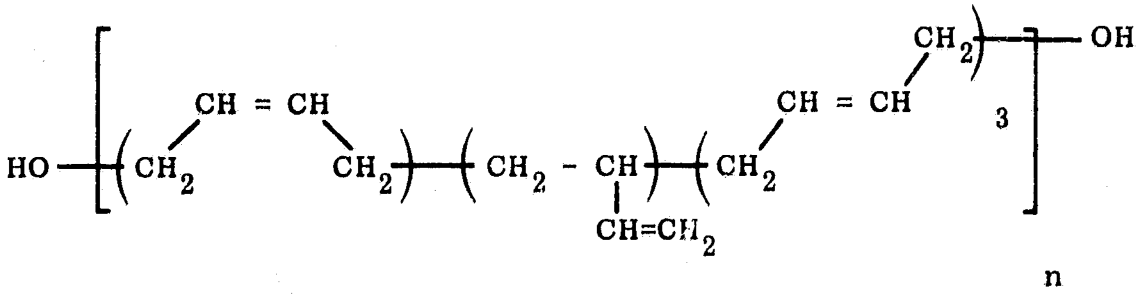
1,2- addition



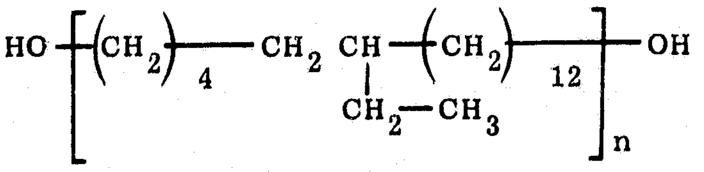
$-\!\!\left[CH_2\ CH = CH - CH_2\right]\!\!-$

1,4- addition

One typical commercially available hydroxy-terminated polybutadiene is:



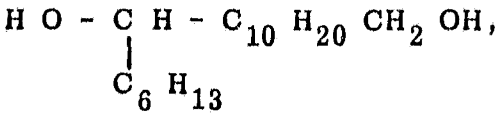
The predominant configuration is trans-1,4 (60 percent) with approximately 20 percent each cis-1,4 and vinyl-1,2. Hydrogenation yields



$$HO-\left[(CH_2)_4 - CH_2\ CH(CH_2-CH_3) - (CH_2)_{12}\right]_n - OH \ .$$

Hydrogenation of the 1,4-chain leads to polyethylene, a highly crystalline polymer, whereas, hydrogenation of the 1,2-chain would yield poly-1-butene, presumably atactic, and hence non crystalline. A study of methods for preparing polybutadiene with a high 1,2-addition content was not within the scope of this study. However, the range of usable molecular weights found by this study could undoubtably be extended by the use of highly branched polyols to disrupt the chain regularity favoring crystallinity and thus lower the polymer viscosity. As an example, a urethane prepared from 1,12-dodecanediol, molecular weight 202, had an initial viscosity greater than 20,000 poise; while one prepared from a hydrogenated polybutadiene diol, molecular weight 1350, had an initial viscosity of 380 poise.

Seven alkane-based polyols, varying in molecular weight from approximately 300 to 4000, were used for preparing polyurethanes to be used in this study. With one exception, the polyols were prepared by the hydrogenation of hydroxy-terminated polybutadiene. Hydrogenated ricinoleic acid,



$$HO-\underset{\displaystyle C_6H_{13}}{\underset{|}{CH}}-C_{10}H_{20}CH_2OH,$$

was used as the lowest molecular weight diol.

Diisocyanates are the second group of components used in the manufacture of polyurethanes. These chemicals end-cap the polyols and supply the active sites necessary for cross-linking or chain extension. The most commonly used and least expensive diisocyanate is 2,4-toluene diisocyanate (TDI). However, in this study, two aliphatic diisocyanates, isophorone dissocyanate and methylene bis(4-cyclohexyl-isocyanate), were used to evaluate their effect on pot life as compared to the aromatic TDI-type diisocyanate.

The final component of polyurethanes is the chain-extender or cross-linker. This component allows the urethane prepolymer to reach its final molecular weight and converts it via crosslinks to a thermoset material. The chain extenders fall into two categories, diols and amines. Both types were used in this study. The most common diamine type is 4,4' methylene-bis(2-chloroaniline), known under the DuPont trade name MOCA. This curing agent adds toughness to the urethane; but it was not used in this study because it is a carcinogen suspect. Polacure 740M, trimethylene glycol di-p-aminobenzoate made by Polaroid Corporation, was used. 1,4-Butanediol was used in one series of urethanes.

Three series of urethanes with molecular weights from 300 to 4000 were prepared, molded, and cured into configurations for the evaulation of physical properties. The cured urethanes were tested for hardness, tensile and tear strength, percent elongation, and specific gravity. These properties were compared to that required by MSFC-SPEC-515, which is the controlling specification for urethane potting and molding material. Thermogravimetric and thermomechanical analyses were performed to further characterize these materials.

II. URETHANE PREPARATION

A. Hydrogenation of Polybutadiene

The polybutadiene diol was hydrogenated using a Parr 3921, shaker type, hydrogenation apparatus. A 200 W glass fabric heating mantle was used for heating the reaction bottle. The temperature of the reaction was automatically controlled with a Parr 3931 temperature controller and a stainless sheathed thermistor probe in the reaction bottle.

One hundred grams of hydroxy-terminated polybutadiene was dissolved in 1 liter of toluene in a 2-liter reaction bottle. Approximately 1.0 gram of Palladium (5%) on activated carbon was added and the bottle was placed in the hydrogenation apparatus. The bottle was evacuated and refilled with nitrogen. This procedure was repeated three times. The vessel was evacuated and refilled with hydrogen twice. The apparatus was pressurized to 50 psig and the material reacted at 75°C. The reaction was allowed to proceed until a minimum of 90 percent of the theoretical amount of hydrogen was absorbed. The catalyst was then removed by Soxhlet extraction of the reaction mixture. The toluene was evaporated isolating the hydrogenated polybutadiene diol.

Figures 1 and 2 show the infrared spectrum of polybutadiene before and after hydrogenation. The disappearance of the absorption bands in the 910 cm^{-1} to 1000 cm^{-1} region, generally associated with cis, trans, and 1,2 vinyl units, indicates the polybutadiene has been hydrogenated. The groups were hydrogenated preferentially. The vinyl group was hydrogenated the most rapidly and the trans-1,4 the least. Some samples showed some residual unsaturated trans units. However, according to the amount of hydrogen absorbed, all samples were hydrogenated to 90 percent or greater.

B. Alkane Diols

The alkane diols used in this study were primarily hydrogenated, hydroxy-terminated polybutadienes (HTPBD). Two of these materials under the trade name

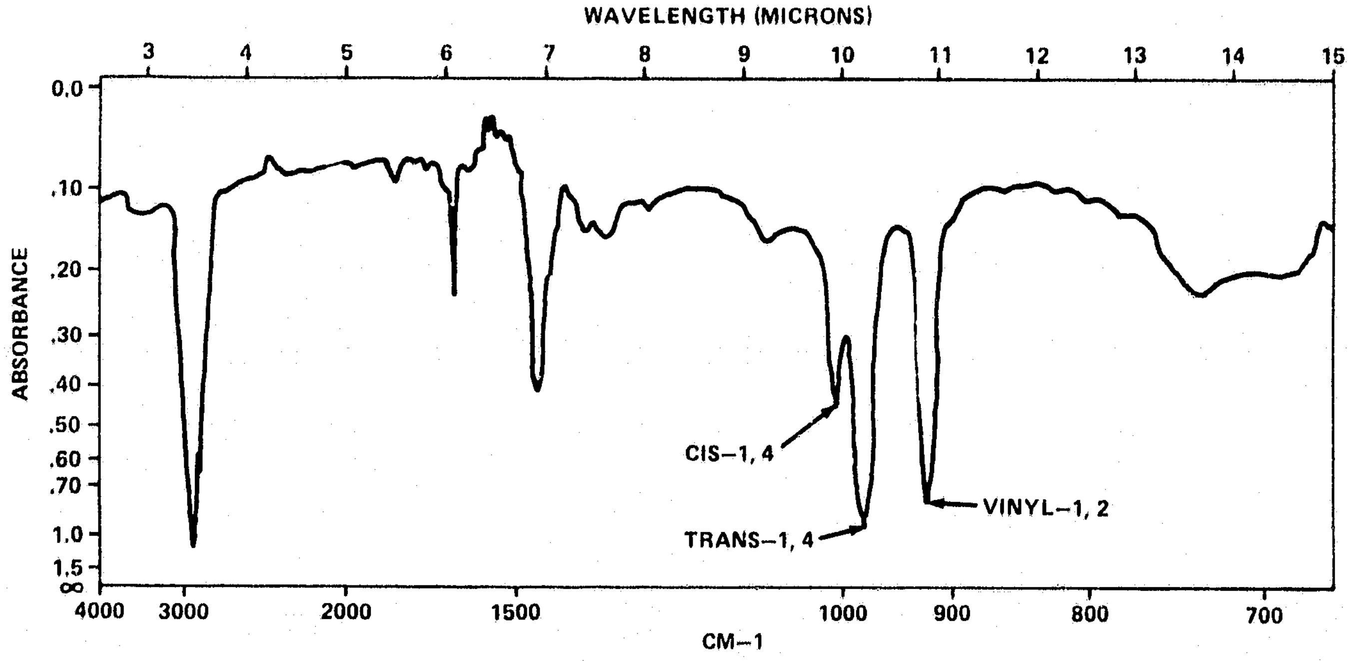


Figure 1. Infrared spectrum of hydroxy-terminated polybutadiene.

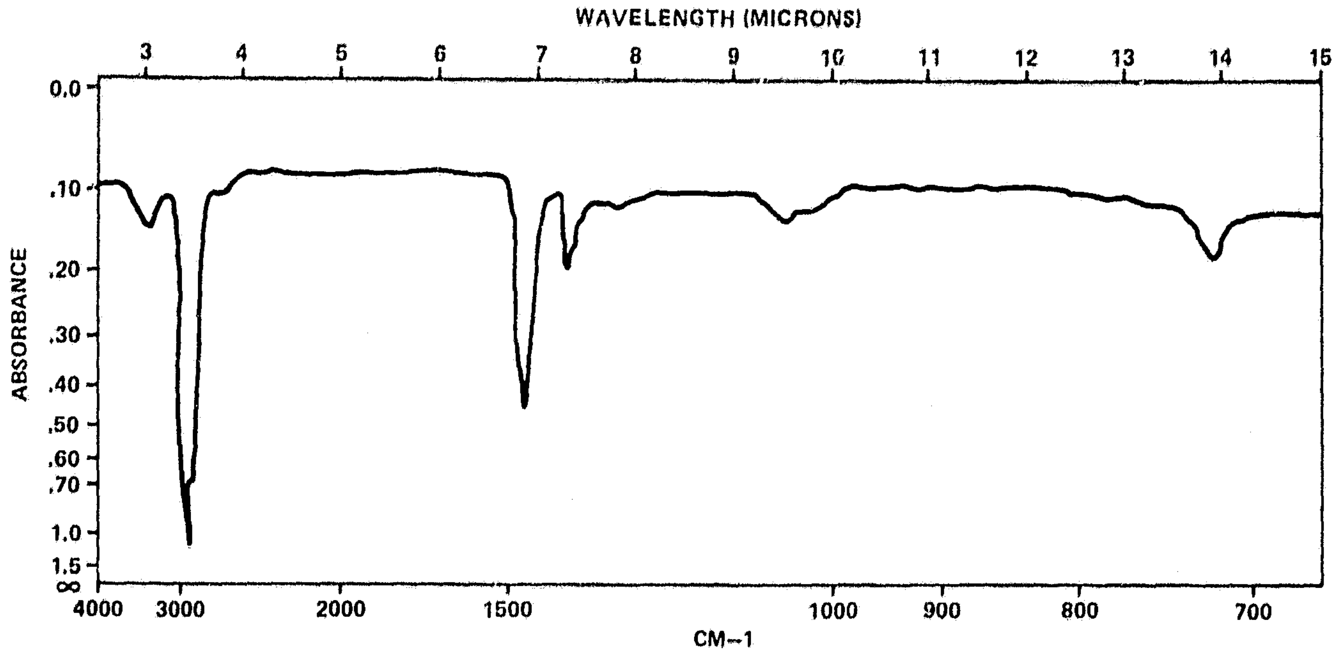


Figure 2. Infrared spectrum of hydrogenated, hydroxy-terminated polybutadiene.

Telagen-S were obtained from General Tire and Rubber Company. Hydroxy-terminated polybutadienes ranging in molecular weight from 450 to 3350 were obtained from Polyscience, LTD and Arco Chemical Company. This range would include and exceed the molecular weight required in the preparation of polyurethanes for connector-potting applications.

Table 1 lists the diols used in this study. Molecular weights for those diols not furnished by the distributor were determined using gel permeation chromatographic (GPC) techniques. The chromatography studies were carried out on a Waters Associates liquid chromatograph equipped with a Model 6000 A solvent delivery system, Model 440 absorbance detector, and R 401 differential refractometer.

Hydroxyl functionality was calculated from hydroxyl number and number-average molecular weight. Hydroxyl number is the milligrams of OH expressed as potassium hydroxide per gram of sample. The hydroxyl equivalent weight is 1000 x molecular weight of potassium hydroxide/hydroxyl number or

$$\text{Hydroxyl equivalent weight} = \frac{56{,}100}{\text{Hydroxyl Number}}$$

Functionality of the diol is the number-average molecular weight/hydroxyl equivalent weight or

$$\text{Functionality} = \frac{M_n \times \text{Hydroxyl Number}}{56{,}100}$$

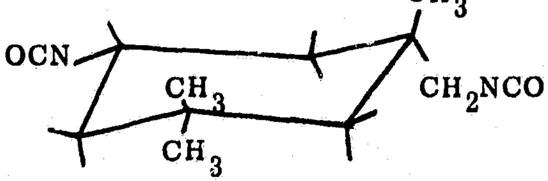
A functionality of two indicates two hydroxyl groups per molecule. Monofunctional molecules would prevent chain extension and result in a lower molecular weight of the cured polymer. The functionality of the diols used in this study was very close to two considering variations due to experimental error, with the exception of HTPBD 1350 and 2000 as indicated in Table 1. Mechanical properties of urethanes prepared from these diols would be suspect.

TABLE 1. CHARACTERIZATION OF DIOLS

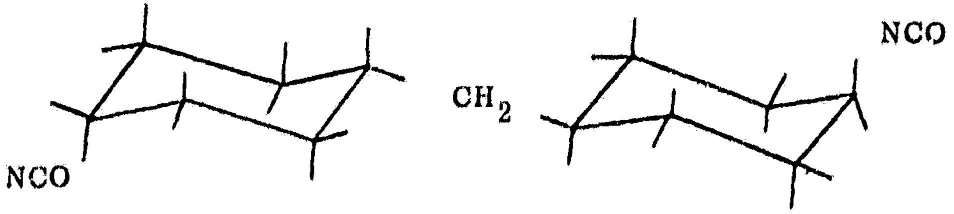
Alkane Diol	Molecular Weight Number Average, $\bar{M}_n$	Functionality
1,12-Dodecane diol	202	2.0
Hydrogenated Ricinoleic Acid	286	2.0
HTPBD 450	450	2.0
HTPBD 1350	1350	1.6
Telagen-S A595-88	1660	2.1
HTPBD 2000	2000	1.4
HTPBD 2800	2800	2.2
HTPBD 3500	3350	2.3
Telagen-S 185M	3960	1.9

C. Diisocyanates

Two aliphatic diisocyanates, isophorone diisocyanate and methyl bis-(4-cyclohexylisocyanate), were studied in this project. Isophorone diisocyanate or 3-isocyanate-methyl-3,5,5-trimethyl-cyclohexylisocyanate was obtained from Thorson Chemical Corporation. The structure is



Methyl-bis(4-cyclohexylisocyanate) was obtained from Mobay Chemical Corporation under the trade name Desmodur W. The structure is



Each diisocyanate was used for preparing a series of urethanes with the diols in Table 1. The diisocyanates react much more slowly with the diols than does TDI. Viscosity increases slowly resulting in a urethane with lower viscosity and a longer working life. Higher temperatures are required for complete cure. Because of lower reactivity some of these urethanes can be processed at elevated temperatures to take advantage of the resulting lower viscosities. A urethane prepared from 1,12-dodecanediol with a viscosity greater than 20,000 poise at room temperature was processed satisfactorily at elevated temperature. Viscosity decreases rapidly with increase in temperature. Some unique urethanes probably could be prepared by reacting these aliphatic diisocyanates with phenol-type materials.

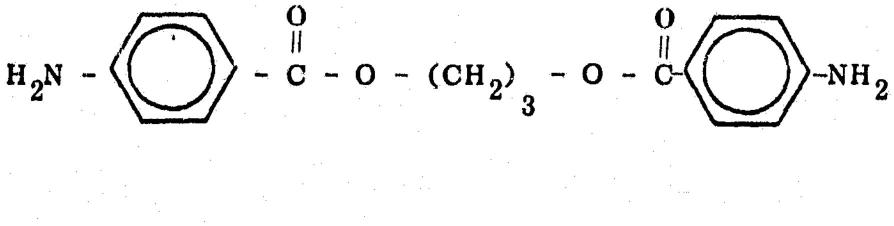
Relative reactivity of catalyzed urethanes prepared from the two aliphatic diisocyanates and a commercial urethane of the TDI-type is illustrated in Figure 3. MSFC-SPEC-515, the specification for molding and potting material, requires that the application life of freshly mixed urethane (time for viscosity to exceed 1000 poise) should be at least 1 hr. The two experimental urethanes easily meet this requirement. The comparison of the application life of the three urethanes is:

Commercial urethane	1 to 2 hr
Isophorone urethane	2 to 3 hr
Desmodur W urethane	4 to 5 hr

However, the initial viscosity and the cure time of the two experimental diisocyanates exceed that required by MSFC-SPEC-515. Urethanes prepared from Desmodur W and hydrogenated, hydroxy-terminated polybutadienes of molecular weights up to 2500 (Fig. 4) are usable as potting and modling materials. Those prepared from isophorone diisocyanate are more viscous but are usable to about 2000 molecular weight. However, the urethanes have a high initial viscosity and a long cure time. These disadvantages must be weighed against the materials' excellent dielectric properties and moisture resistance.

D. Curing Agents

The urethanes were cured with trimethylene glycol di-p-aminobenzoate



$$H_2N-C_6H_4-\overset{O}{\overset{\|}{C}}-O-(CH_2)_3-O-\overset{O}{\overset{\|}{C}}-C_6H_4-NH_2$$

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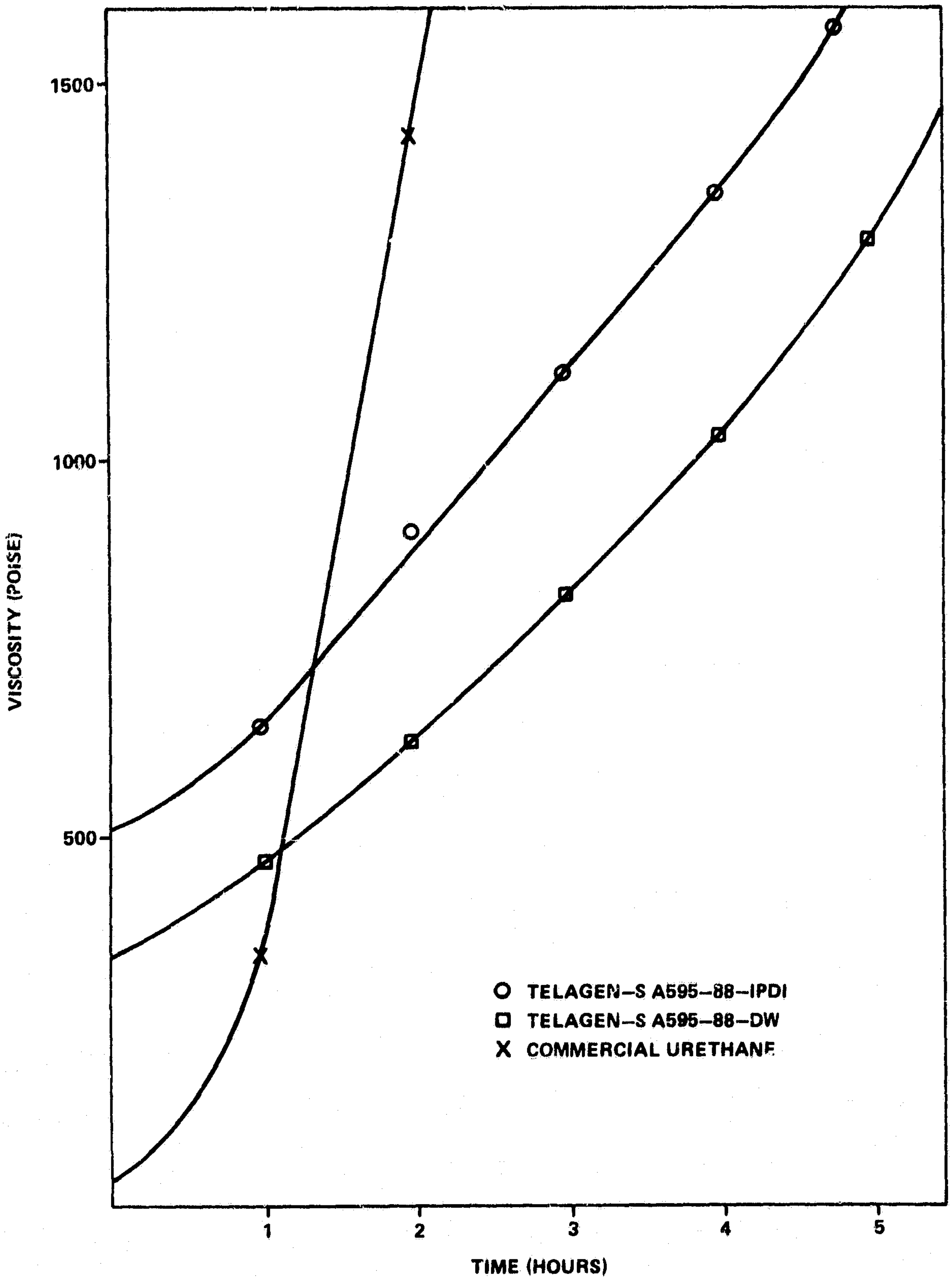

Figure 3. Viscosity of catalyzed urethanes versus time.

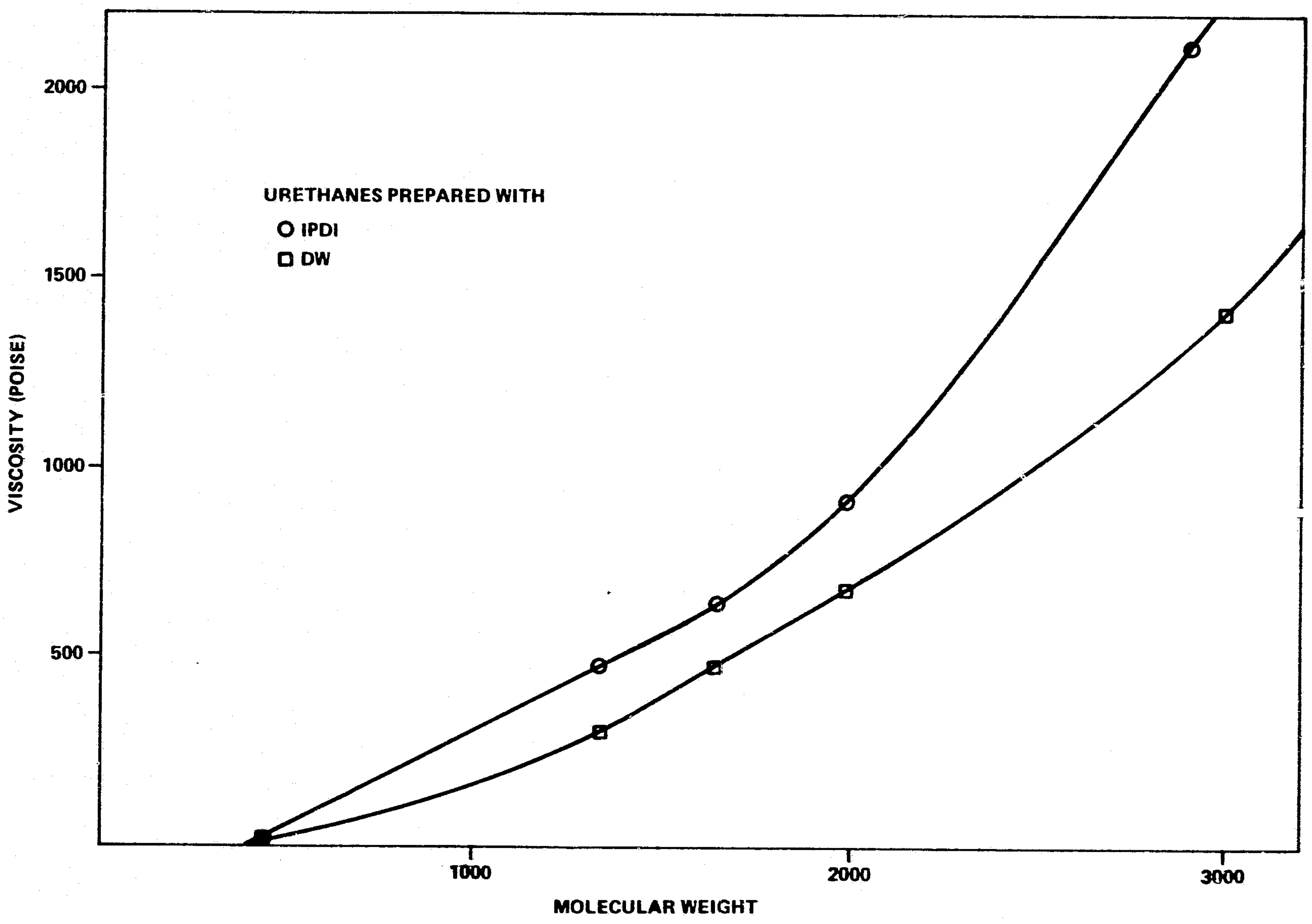

Figure 4. Viscosity of alkane-based urethanes catalyzed for one hour versus molecular weight of diol.

sold by Polaroid Corporation under the trade name Polacure 740M. Three series of urethanes were prepared with alkane diols listed in Table 2. One series was prepared with isophorone diisocyanate (IPDI) and one with Desmodur W (DW). These urethanes were cured with the Polacure 740M. One additional series was prepared with the alkane diols and isophorone diisocyanate and cured with a mixture of Polacure 740M and 1,4 butanediol to evaluate the diol as a chain extender.

TABLE 2. COMPOSITION OF ALKANE-BASED URETHANES

Alkane Diol	Diisocyanate	Weight Diisocyanate g/100g Diol	Curing Agents g/100g Diol	
			Polacure 740M	1,4 Butanediol
Hydrogenated Ricinoleic Acid	IPDI	86.1	4.5	2.1
HTPBD 450	IPDI	64.5	4.2	3.8
HTPBD 1350	IPDI	33.0	4.2	4.3
Telagen-S A595-88	IPDI	26.8	4.0	3.1
HTPBD 2000	IPDI	22.2	3.8	2.3
HTPBD 2800	IPDI	15.8	3.6	1.1
HTPBD 3500	IPDI	13.2	3.6	0.6
Telagen-S 185M	IPDI	11.2	3.5	0.3
1,12 Dodecandiol	IPDI	117.4	5.4	-
Hydrogenated Ricinoleic Acid	IPDI	80.4	4.2	-
HTPBD 450	IPDI	54.4	3.4	-
HTPBD 1350	IPDI	20.0	2.5	-
Telagen-S A595-88	IPDI	16.9	2.5	-
HTPBD 2000	IPDI	14.7	2.5	-
HTPBD 2800	IPDI	11.7	2.7	-
HTPBD 3500	IPDI	10.6	2.9	-
Telagen-S 185M	IPDI	10.0	3.1	-
1,12 Dodecandiol	DW	139.8	6.1	-
Hydrogenated Ricinoleic Acid	DW	95.8	4.7	-
HTPBD 450	DW	64.8	3.8	-
HTPBD 1350	DW	23.9	2.7	-
Telagen-S A595-88	DW	20.2	2.6	-
HTPBD 2000	DW	17.5	2.7	-
HTPBD 2800	DW	14.0	2.8	-
HTPBD 3500	DW	12.8	3.0	-
Telagen-S 185M	DW	12.0	3.2	-

E. Preparation of Test Specimens

The urethanes were prepared in a one step reaction with the alkane diol, diisocyanate, and chain extenders in the ratios shown in Table 2. Polacure 740M was dissolved by heating with the alkane diol. The diisocyanate was added to the mixture, stirred thoroughly, and then degassed. The reaction mixture was poured into molds for sheet material. The material was cured in air at 70°C for 12 hr and 120°C for

18 hr. Specimens for tensile strength, elongation, tear strength, and thermal tests were cut from the cured sheets of the elastomer for evaluation of mechanical properties.

III. URETHANE EVALUATION

A. Hardness

Hardness was measured with a Shore Durometer, Type A-2, with the exception of the urethanes prepared from 1,12-dodecanediol. Hardness for these urethanes was measured with Shore Durometer, Type D. The dodecanediol-urethane has a relatively short flexible chain and a very regular structure resulting in a highly crystalline and hard urethane. These urethanes are not elastomeric and are not suitable for potting applications. MSFC-SPEC-515 permits a hardness range of 60 to 90 Shore A hardness for potting applications. Most of the urethanes prepared in this study, as shown in Table 3, fall in this range.

B. Tensile Strength and Elongation

Tensile strength and elongation testing was in accordance with ASTM D 638-77. This method covers the determination of the tensile properties of plastics in the form of standard dumbell-shaped test specimens. The specimens were cut from cast sheets of the cured urethane. The values obtained for the experimental urethanes are shown in Tables 4 and 5. Urethanes prepared with Desmodur W have higher tensile strength

TABLE 3. HARDNESS OF CURED URETHANES

	Diisocyanate		
Alkane Diol	Desmodur W	Isophorone Diisocyanate	Isophorone[a] Diisocyanate
1,12-Dodecane Diol	78 D	78 D	-
Hydrogenated Ricinoleic Acid	96	95	95
HTPBD 450	77	83	89
HTPBD 1350	67	63	76
Telagen-S A595-88	64	60	74
HTPBD 2000	64	62	73
HTPBD 2800	64	61	72
HTPBD 3500	64	63	60
Telagen-S 185M	57	60	60

a. 1,4 Butanediol added as chain extender as shown in Table 2.

TABLE 4. TENSILE STRENGTH AT BREAK, PSI

Alkane Diol	Diisocyanate		
	Desmodur W	Isophorone Diisocyanate	Isophorone[a] Diisocyanate
1,12-Dodecane Diol	7510	8099	-
Hydrogenated Ricinoleic Acid	2971	1600	2576
HTPBD 450	629	516	892
HTPBD 1350	320	294	339
Telagen-S A595-88	729	523	729
HTPBD 2000	383	358	390
HTPBD 2800	656	405	806
HTPBD 3500	445	408	458

a. 1,4 Butanediol added as chain extender as shown in Table 2.

TABLE 5. ELONGATION AT BREAK, PERCENT

Alkane Diol	Diisocyanate		
	Desmodur W	Isophorone Diisocyanate	Isophorone[a] Diisocyanate
1,12-Dodecane Diol	70	90	-
Hydrogenated Ricinoleic Acid	142	172	98
HTPBD 450	167	172	182
HTPBD 1350	192	243	197
Telagen-S A595-88	437	525	275
HTPBD 2000	74	83	142
HTPBD 2800	125	175	170
HTPBD 3500	192	197	155

a. 1,4 Butanediol added as chain extender as shown in Table 2.

and lower percent elongation than those prepared with isophorone diisocyanate. The tensile strength of the isophorone diiscoyanate material was increased by the addition of 1,4 butanediol as a chain extender. A study directed toward cure optimization would probably result in urethanes with better mechanical properties but such a study is beyond the scope of this task.

MSFC-SPEC-515 requires a minimum 1500 psi tensile strength and 200 percent elongation for potting applications. The urethanes prepared from 1,12 dodecanediol has a very high tensile strength, but the material is not elastomeric, as indicated by its low percent elongation, and is not suitable for potting applications. The low values for tensile strength obtained for HTPBD 1350 and 2000 are probably due to their low functionality. Most of the experimental resins are usable as potting material. However, the alkane-based urethanes, in general are less elastomeric, more plastic, and not as tough as the ether-based urethane.

C. Tear Strength

Tear strength was determined in accordance with ASTM D624-54. The test specimens were cut with a Model C die from cast sheets of the cured urethane. Urethanes prepared with Desmodur W (Table 6) have slightly greater tear strength than those prepared with isophorone diisocyanate. The use of 1,4 butanediol with the isophorone diisocyanate as a chain extends improves the tear strength slightly. Hydroxy-terminated polybutadiene 2800, with a low functionality, formed urethanes with low tear strengths for all three formulations. MSFC-SPEC-515 requires a minimum tear strength of 175 lb/in. Most of these experimental urethanes do not meet these requirements. In general, the alkane-based urethanes are not as resistant to tear as are the ether-based materials.

TABLE 6. TEAR STRENGTH, LBS/IN

Alkane Diol	Diisocyanate		
	Desmodur W	Isophorone Diisocyanate	Isophorone[a] Diisocyanate
1,12-Dodecane Diol	1099	1096	-
Hydrogenated Ricinoleic Acid	419	126	434
HTPBD 450	72	63	83
HTPBD 1350	69	65	69
Telagen-S A595-88	112	93	119
HTPBD 2000	62	52	58
HTPBD 2800	109	97	126
HTPBD 3500	97	95	100

a. 1,4 Butanediol added as chain extender as shown in Table 2.

D. Glass Transition and Expansion Coefficient

Glass transitions and expansion coefficients for the experimental urethanes were determined with DuPont 943 Thermomechanical Analyzer in conjunction with the Dupont 1090 Thermal Analysis/Data System. Figure 5, a typical thermomechanical analysis of an alkane-based urethane, shows the glass transition (T_g) of the material as well as coefficient of expansion (α) below and above T_g. Table 7 lists the glass transition temperatures for the experimental urethanes. Table 8 lists the expansion coefficients. The coefficient of linear thermal expansion is a very important parameter for materials used for coating circuit boards. It is possible for a thick coating with a high expansion coefficient to break a solder joint. MSFC-SPEC-507, specification for conformal coating material for printed circuit boards, requires a maximum coefficient of expansion of 175μm/m °C from -55 to 100°C. As shown in Table 8, the expansion coefficient for a urethane below T_g is much less than that above T_g. This temperature range spans the glass transition range of most urethanes and includes two distinct rates of expansion. As a result the expansion rate over the specified range would be an average or resultant vector for the above T_g and below T_g rates. This is illustrated in Figure 5. The expansion coefficient is 75μm/m °C below T_g and 338μm/m °C above T_g. It is 214μm/m °C between -55 and 100°C. The urethanes with the higher glass transition temperatures give the better expansion coefficients in the required range. In general, the alkane-based urethanes have expansion coefficients higher than is desirable for coating applications.

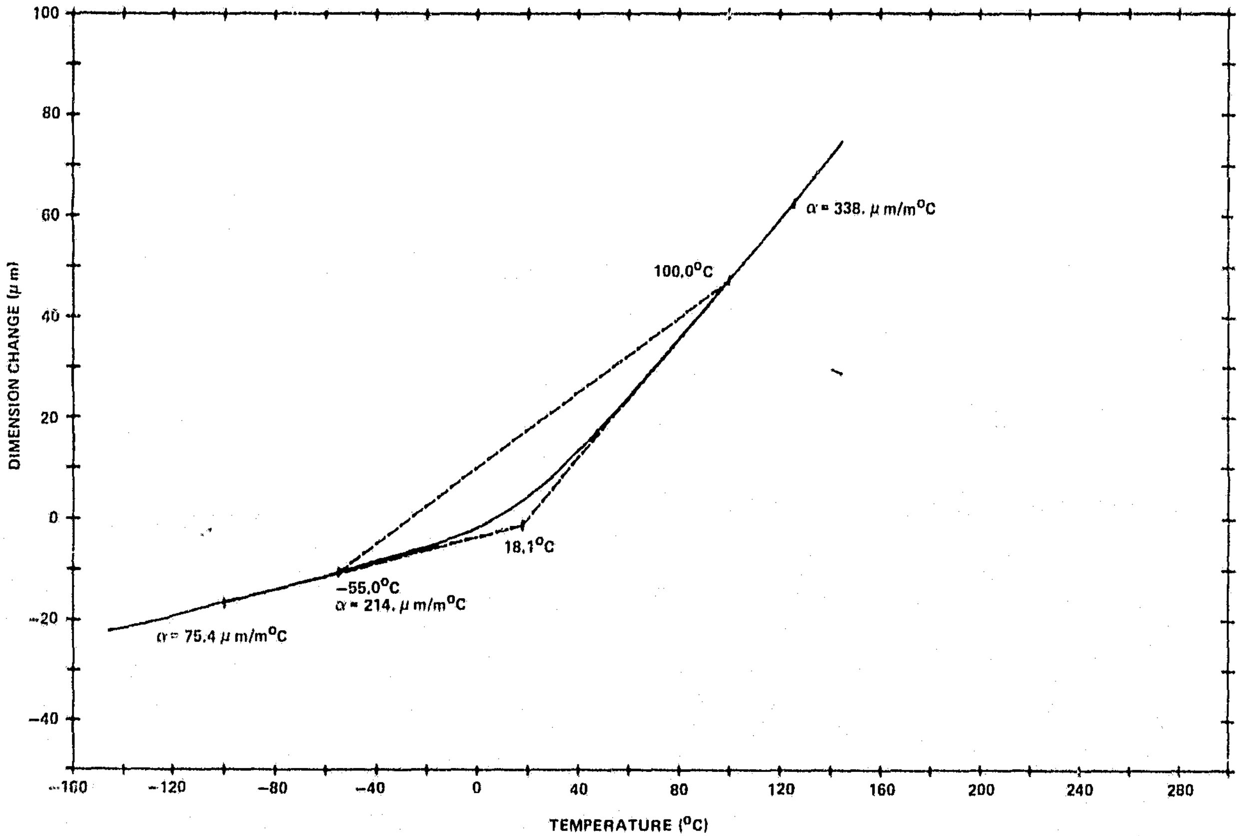

Figure 5. Thermomechancial analysis of alkane-based urethane.

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TABLE 7. GLASS TRANSITION, °C

Alkane Diol	Diisocyanate Desmodur W	 Isophorone Diisocyanate	 Isophorone[a] Diisocyanate
1,12-Dodecane Diol	50	51	-
Hydrogenated Ricinoleic Acid	49	42	54
HTPBD 450	17	15	19
Telagen-S A595-88	-20	-16	-25
HTPBD 2800	-9	-18	-17
HTPBD 3500	-24	-19	-34

a. 1,4 Butanediol added as chain extender as shown in Table 2.

TABLE 8. EXPANSION COEFFICIENT, μm/m °C

	Diisocyanate					
	Desmodur W		Isophorone Diisocyanate		Isophorone[a] Diisocyanate	
Alkane Diol	Below T_g	Above T_g	Below T_g	Above T_g	Below T_g	Above T_g
1,12-Dodecane Diol	83	192	68	332	-	-
Hydrogenated Ricinoleic Acid	118	207	136	232	111	246
HTPBD 450	132	263	129	305	75	337
Telagen-S A595-88	108	309	87	452	94	292
HTPBD 2800	139	405	115	325	112	330
HTPBD 3500	112	382	125	411	113	413

A. 1,4 Butanediol added as chain extender as shown in Table 2.

E. Thermogravimetric Analysis

Decomposition temperatures and weight loss for the alkane-base urethane were determined with a DuPont 951 Thermogravimetric Analyzer in conjunction with a DuPont 1090 Thermal Analysis/Data System. Table 9 summarizes the thermogravimetric analyses of the experimental alkane-based urethanes. The urethanes were heated to 600°C at a rate of 20 deg/min in gaseous nitrogen. Decomposition was almost complete for all samples. The temperature of onset of decomposition, the inflection point, and percent weight loss are shown in Table 9 for the three series of urethanes. The thermal stability of the experimental urethanes compare favorably with other urethane types shown in Figure 6. There is very little difference among the urethane types with the exception of the polybutadiene-based urethane. This urethane has a lower on-set temperature and appears to have two transition temperatures. The thermal stability of the three series of urethanes prepared in this study are very similar. Figure 7 compares three alkane-based urethanes prepared from hydrogenated ricinoleic acid (HRA). One urethane is prepared from ricinolic acid and Desmodur W (DW-HRA), one from the acid and isophorone diisocyanate (IPDI-HRA), and one with 1,4 butanediol added as a chain extender (IPDI-HRABA). The urethane prepared with Desmodur W appears to have the best thermal properties. The two prepared with isophorone diisocyanate have very similar properties. In general, the thermal properties of the alkane-based urethanes are comparable to other type urethanes.

IV. CONCLUSIONS

Alkane-based urethanes were prepared by reaction of both isophorone diisocyanate and methyl-bis(4-cyclohexylisocyanate) with hydrogenated, hydroxy-termianted polybutadiene. Urethanes with molecular weights up to 2500 had viscosities low enough to be used in potting and molding applications. Since the reactivity of the alkane diisocyanates was less than that of their aromatic diisocyanate counterparts, the working life of the experimental urethanes was longer than that of the average commercial urethane. However, the initial viscosity was higher. The higher molecular weight alkane-based urethanes were not suitable for potting applications. A comparison of the properties of the experimental urethanes with MSFC-SPEC 515 requirements is shown in Table 10.

The experimental urethanes could be prepared in the required hardness range, 60 to 90 Shore A. However, none of the urethanes met all of the requirements relating to hardness, tensile strength, elongation, and tear strength. The alkane-based urethanes did not have as high tensile strength, percent elongation, and resistance to tear as ether or ester-based urethanes. The experimental urethanes were less elastomeric, more plastic, and not as tough as ether-based material.

The thermal stability of the experimental urethanes was as good or better than ether, ester, and polybutadiene-based urethanes. The coefficient of linear thermal expansion for the material was higher than desirable for printed circuit board coatings.

The alkane-based urethane can be used for potting and molding applications. Up to 2500 molecular weight, viscosity is low enough and working life is long enough for this usage. Dielectric properties and hydrolytic stability are excellent. However, mechanical properties are not as good as those of ether and ester-based urethanes.

TABLE 9. THERMOGRAVIMETRIC ANALYSIS

Alkane Diol	Diisocyanate								
	Desmodur W			Isophorone Diisocyanate			Isophorone[a.] Diisocyanate		
	Onset, °C	Inflection Point, °C	Weight Loss Percent	Onset, °C	Inflection Point, °C	Weight Loss, Percent	Onset, °C	Inflection Point, °C	Weight Loss, Percent
1.12-Dodecane Diol	359	393	95	356	387	97	–	–	–
Hydrogenated Ricinoleic Acid	342	464	98	328	375	99	334	424	98
HTPBD 450	317	480	96	325	457	93	326	484	96
HTPBD 1350	342	446	97	340	422	97	335	420	98
Telagen-S A595-88	359	453	97	350	468	99	339	468	99
HTPBD 2800	325	463	94	350	460	96	313	487	96
HTPBD 3500	375	482	99	380	464	96	325	479	96

a. 1,4 Butanediol added as chain extender as shown in Table 2.

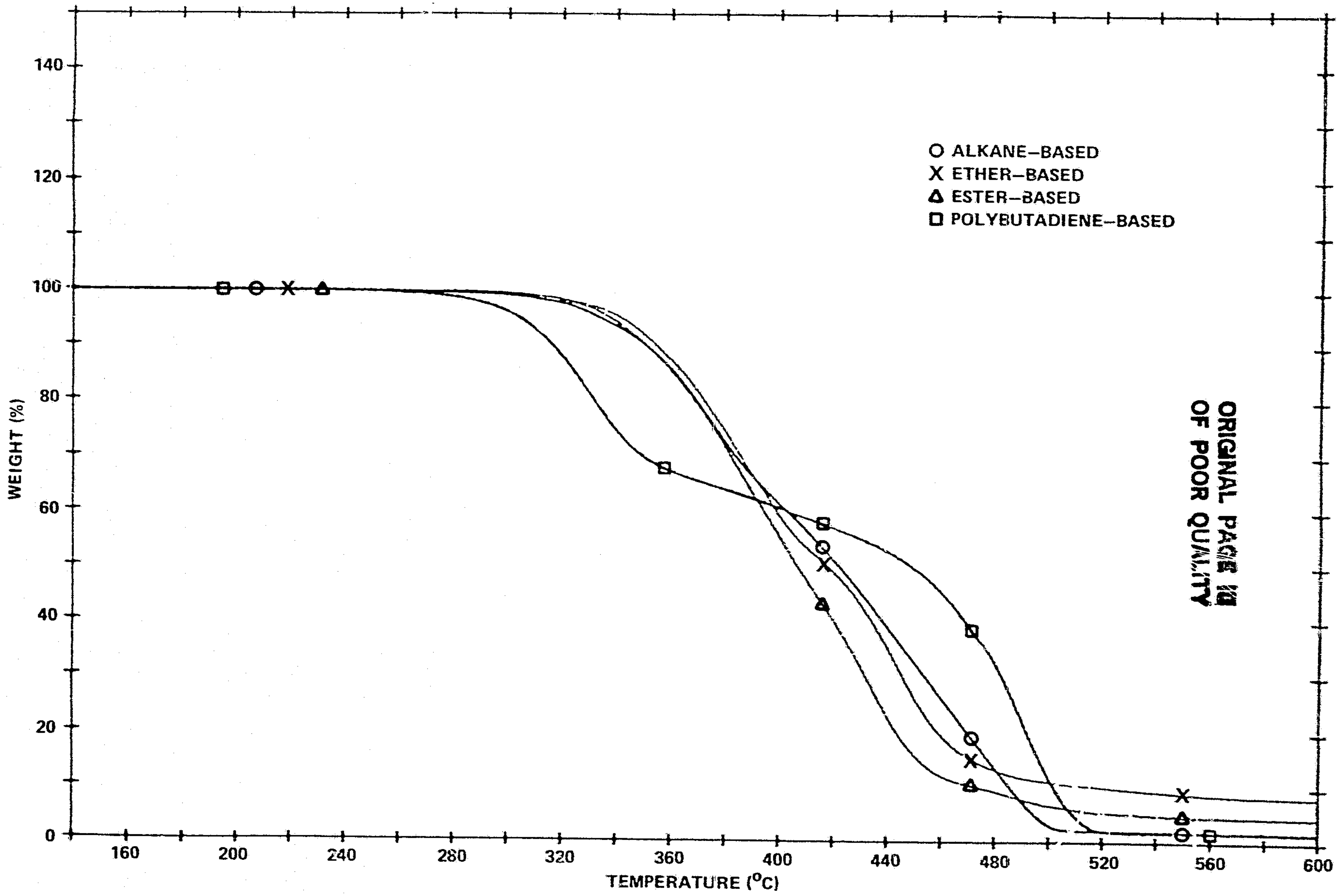

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Figure 6. Weight loss of urethanes versus temperature.

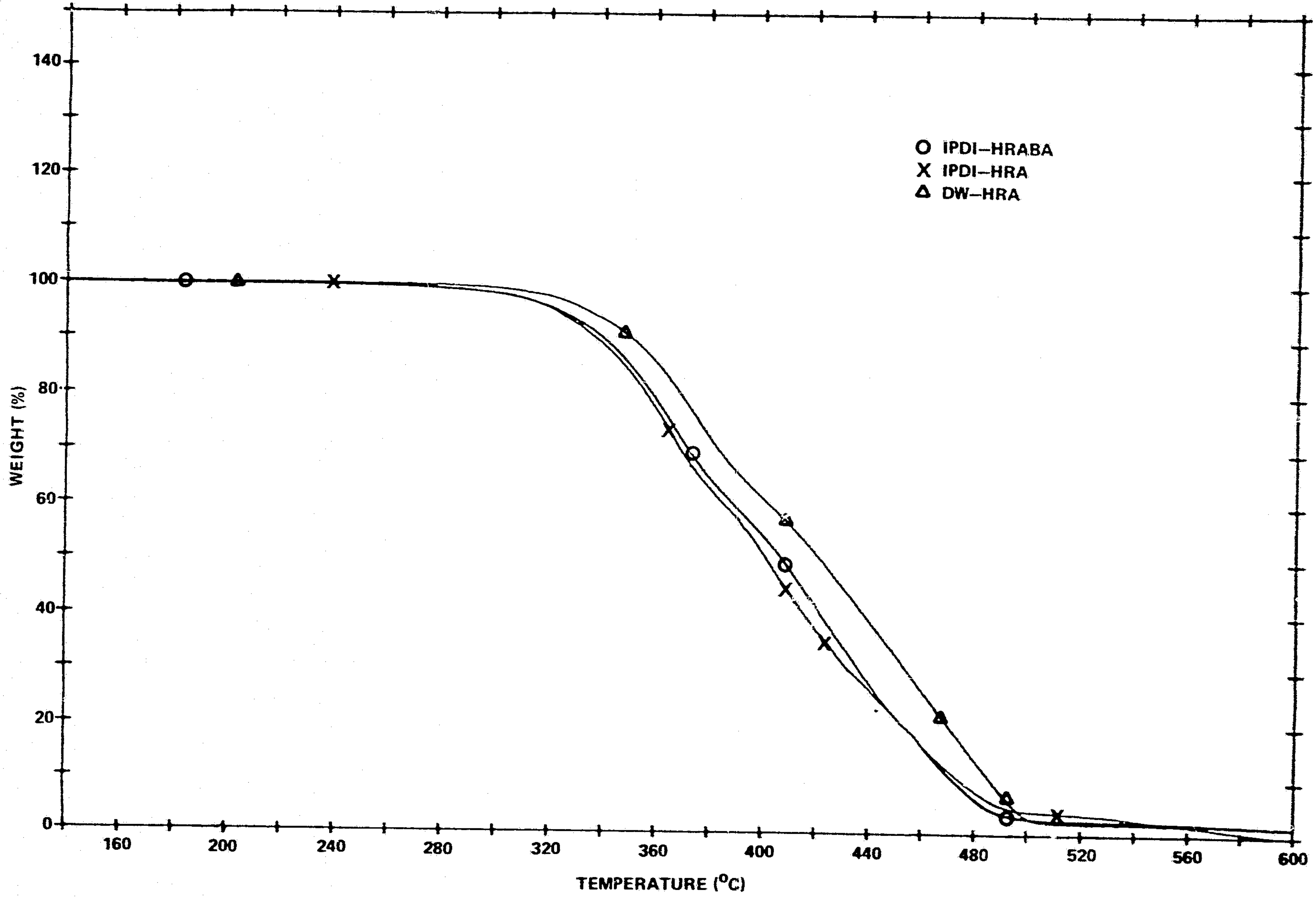

Figure 7. Weight loss of alkane-based urethanes versus temperature.

TABLE 10. COMPARISON OF PROPERTIES OF EXPERIMENTAL URETHANES WITH REQUIREMENTS OF SPECIFICATIONS FOR URETHANE POTTING MATERIAL

Properties	MSFC-SPEC 515[a]	Experimental Alkane-Based Urethanes
Hardness, Shore A	60-90	57-96
Tensile Strength, psi	1500 min	400-3000
Tear Strength, lb/in	175 min	52-434
Elongation, percent	200 min	74-525
Viscosity, Initial at 25°C, poise	450 max	20-500
Application Life At 25°C, hours	1 min	2-10
Coefficient of Thermal Expansion, -55°C to +100°C, in/in/°C	175×10^{-6} max	$162\text{-}324 \times 10^{-6}$

[a] Specification for urethane potting and molding elastomers.

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