Relationship of Molecular Structure and Resin Performance for a Series of Diglycidyl Ethers

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INTRODUCTION

Since the commercialization of epoxy resins, numerous bisphenols have been investigated as possible starting materials. Many of these evaluations, however, have been performed on reaction products of mixed composition. Thus, although valuable from the standpoint of specific resins, the results were not always definitive, and correlations between performance and structure were frequently of indeterminate value. In order to obtain valid correlations, a few pure diglycidyl ethers of the following basic structure were examined.

$$CH_2-CH-CH_2-O-CH_2-O-CH_2-CH-CH_2$$

These materials were then compared with the diglycidyl ether of the bisphenol of acetone (BPA or bisphenol A) for which R_1 and R_2 are CH_3 , a constituent of most commercial epoxy resins. The diglycidyl ethers selected for examination were arbitrarily restricted to those derived from aliphatic ketones. These include an unsymmetrical homologous straight-chain series, methyl ethyl, methyl amyl, methyl nonyl, and methyl heptadecyl ketones; a symmetrical straight-chain ketone, diheptadecyl; and a branched-chain ketone, methyl isobutyl.

PROCEDURES AND DISCUSSION

The ketones used in this program were, with one exception, commercially available products. The exception was diheptadecyl ketone, which was prepared by the pyrolysis of magnesium stearate following the method of Curtis, Dobson, and Hatt.¹

All of the bisphenols were synthesized from the ketones by heating five moles of phenol with one of ketone. The only variations in the syntheses were those of temperature and reaction time shown in Table I. The reaction time variations were dictated by yield considerations, but the temperature variations were required either because of poor solubility of the solid ketones in the phenol or crystallization of the reaction mixture at some time after the initiation of the reaction. This crystallization is presumed to be due to the formation of one or more phenol-bisphenol adducts. Adduct formation between bisphenol A and phenol has been known for some time. Also, the formation of 1:1 phenol-bisphenol adducts with methyl nonyl and methyl heptadecyl bisphenols has been conclusively shown and was utilized in their purification. It is believed that adduct formation was general for the series studied.

After removal of unreacted phenol and ketone, all of the bisphenols were purified to better than 99% purity. The bisphenol analyses are given in Table II.

Prior to discussion of the diglycidyl ethers, it is perhaps pertinent to mention some qualitative relationships between structure and ketone reactivity. The reactivity of the ketones is quite dependent on variations in the alkyl groups. As would be expected, methyl ketones proved more reactive than

TABLE IPreparation of the Bisphenols

-		-			
Ketone	Reaction tempera- ture, °C.	Reaction time, hr.	Crude yield, wt%*		
Methyl ethyl	55	8	94		
Methyl isobutyl	55	24	70		
Methyl amyl	60	24	97		
Methyl nonyl	55	18	96		
Methyl heptadecyl	70	18	95		
Diheptadecyl	100	114	34ь		

• Weight of stripped product/theoretical weight of bisphenol.

^b Yield was measured after removal of unreacted ketone by precipitation from acetone and subsequent stripping to remove acetone.

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Bisphenol	Molecul	ar weight	Phenolic OH, equil./100 g. ^b		Melting point (u	Phlenoic				
	Founds	Theory	Found	Theory	Found	Literature	impurities			
Methyl ethyl	241	242	0.829	0.826	126.5-127.8	124 ^d	None			
Methyl isobutyl	270	270	0.741	0.741	154.1-155.0	153 ^d	None			
Methyl amyl	273	284	0.711	0.704	104.5 - 105.8		Trace, unidentified			
Methyl nonyl	335	340	0.593	0.588	74.8-75.8	Liquid ^e	None			
Methyl heptadecyl	463	452	0.445	0.442	91.5-92.0	80-81.5f	None			
Diheptadecvl	653	676	0.310	0.296	111.0-111.5	77-78 ^r	Trace, unidentified			

TABI	EI	[
Bisphenol	Ana	lvses

* Ebulliometric.

^b By potentiometric titration with tetra-*n*-butylammonium hydroxide,⁵

 $^{\circ}$ Paper chromatographic examination with water and CCl₄ as solvents and developed with *p*-nitrobenzene-diazonium fluoroborate.⁴

^d Data of Reid and Wilson.²

• Data of Erich and Bodnar.4

^f Data of Morriss et al.³

	Epoxide,	eq./100 g.	Purity based on wt. per epoxide.	a-Glycol.	Phenolic OH.	Total Cl, wt. %	Viscosity (25°C.), poises
Diglycidyl ether	Found	Theory	%	eq./100 g.	eq./100 g.		
Methyl methyl	0.581	0.588	98	Q.000	0.000	0.05	43
Methyl ethyl	0.518	0.565	84	0.000	0.002	0.05	137
Methyl isobutyl	0.513	0.524	96	0.002	0.001	0.12	165
Methyl amyl	0.492	0.505	95	0.002	0.001	0.13	86
Methyl nonyl	0.431	0.442	95	0.002	0.002	0.12	31
Methyl heptadecyl	0.344	0.355	94	0.002	0.003	0.10	23
Diheptadecyl	0.247	0.254	95	0.001	0.005	0.21	35

TABLE III Diglycidyl Ether Analyses

higher homologs, and branched-chain ketones are less reactive than straight-chain compounds. An example, pertinent in both cases, is provided by methyl heptadecyl ketone. This material, intermediate in molecular weight between methyl isobutyl ketone and diheptadecyl ketone, is more reactive than either. Further examples of the effect of chain branching were noted in the failure of diisopropyl or diisobutyl ketones to yield bisphenols. Although the Fisher-Taylor-Hirschfelder models of these bisphenols can be made, both are very hindered and do not possess free rotation about the central atom.

All of the bisphenols prepared are white, crystalline solids of widely different melting points and solubilities. Methyl ethyl² and methyl isobutyl² bisphenols have solubilities similar to BPA. Methyl nonyl, methyl amyl, and methyl heptadecyl³ bisphenols are much more soluble in the usual organic solvents and are listed in order of decreasing solubility. The diheptadecyl³ bisphenol is moderately soluble in both hydrocarbons and alcohols. Other properties of these compounds as well as preparation and purification details may be found in the Experimental Section.

The use of purified bisphenols simplified the preparation of the pure diglycidyl ethers. All of the bisphenols were smoothly converted to their glycidyl ethers by a standard procedure using epichlorohydrin, details of which are given in the Experimental Section. The diglycidyl ethers were then isolated by molecular distillation. Approximately equal molar yields of the desired 2:1 component were isolated from each preparation, indicating little or no interference with the phenolic function by substituents on the central carbon atom.

Analyses of the diglycidyl ethers indicated that, with the exception of the methyl ethyl derivative, purities were very high based on weight per epoxide values. The lower purity of the methyl ethyl compound probably arose as the result of partial thermal decomposition during an initial flash distillation



Fig. 1. Diglycidyl ether viscosity vs. the number of carbon atoms in the alkyl group.

prior to the molecular distillation. The analyses of the diglycidyl ethers are given in Table III.

Several interesting correlations can be drawn from the viscosity data obtained at 25°C. Variations of diglycidyl ether viscosity with structural change are shown in Figure 1. For the relatively long, straight-chain isomers of the methyl series, i.e., the methyl amyl, methyl nonyl, and methyl heptadecyl ethers, an increase in chain length results in a decrease in viscosity (from 86 to 31 to 23 poises, respectively). It may also be noted that this change is not linear. From amyl to nonyl, a four-carbon increase in chain length, a viscosity lowering of 55 poises occurs; whereas from nonyl to heptadecyl, an eight-carbon increase, the viscosity lowering is only 8 poises. Thus, once a chain length of about nine carbons is obtained, further increase in chain length has a much less marked effect on viscosity. It might be speculated that the decrease in viscosity with an increase in chain length is a result either of a reduction in intermolecular association



Fig. 2. Fisher-Taylor-Hirschfelder models of (left) methyl isobutyl bisphenol and (right) methyl heptadecyl bisphenol.

caused by the bulky substituent or the formation of a more nearly spherical shape occasioned by coiling of the longer alkyl chains around the rest of the molecule. The plausibility of this latter explanation may be seen by examination of Figure 2, a picture of the Fisher-Taylor-Hirschfelder models of methyl isobutyl and methyl heptadecyl bisphenols. The long heptadecyl chain can be coiled around the axis of the molecule to give a spherical configuration which can not be duplicated by the tridentate molecule derived from methyl isobutyl bisphenol.

In the case of the two symmetrical ethers, a large increase in chain length resulted only in a minor viscosity change. The viscosity of the diheptadecyl compound (35 poises) is very close to that of a typical sample of the diglycidyl ether of bisphenol A (43 poises).

As pointed out above, branching of the alkyl chain increases viscosity. A comparison of the viscosity of the straight chain n-amyl derivative (86 poises) with that of the branched chain isobutyl derivative (165 poises) shows a much greater variation than would be expected solely on the basis of a two-carbon atom change.

Finally, with the notable exception of the diglycidyl ether of bisphenol A, there appears to be a gross relationship between bisphenol melting point and diglycidyl ether viscosity. As the bisphenol melting point decreases, so does the diglycidyl ether viscosity.

All of the diglycidyl ethers of this series were cured with an amine-type and an anhydride-type

Diglycidyl ether	Heat di temperat	stortion ture, °C.	Notche impact re ftlb./in.	ed Izod esistance, of notch	Rockwell hardness, M scale		% Weight loss or aging, curing agent				
	Zª	PAb	Z	PA	Z	PA	24 hr. at 500°F.	at 500°F.			
	Methyl methyl	164	146	0.98	0.61	107	111	2	23		
	Methyl ethyl	139	129	0.93	0.73	110	109	2	23		
	Methyl isobutyl	154	135	0.89	0.65	105	104	2	29		
	Methyl amyl	137	125	0.79	0.63	97	101	2	24		
	Methyl nonyl	105	98	0.74	0.61	81	88	2	23		
	Methyl heptadecyl	76	72	0.72	0.52	28	37	2	18		
	Diheptadecyl	54	50	0.41	0.70	-98	18	3	17		

 TABLE IV

 Performance Tests on Cured Diglycidyl Ethers

^a Curing agent Z.

^b Phthalic anhydride.

curing agent. The amine agent was a partial reaction product of m-phenylenediamine, methylene dianiline, and phenyl glycidyl ether. The anhydride cures were effected with phthalic anhydride. These curing agents were chosen because they represent two different methods of cure and because both are routinely used for the curing of commercial epoxy resins.

The cured resin series was evaluated for heat distortion temperature, Rockwell hardness, chemical resistance, and high temperature resistance. The results are given in Table IV.

Excellent correspondence was obtained between the heat distortion temperatures of the amine-cured and anhydride-cured resins, as shown in Figure 3. The variations between the performance of diglycidyl ethers must therefore be the result of changes in length and configuration of the chains and not the result of different modes of cure. Three effects may be noted. An increase in straight chain length results in a decrease in heat distortion temperature, and branched chain substituents increase heat distortion temperature. It was previously noted that there appeared to be a relationship between bisphenol melting point and diglycidyl ether viscosity. It appears that a similar relationship holds for bisphenol melting point and diglycidyl ether heat distortion temperature, if only the short chain ethers are considered. These relationships are not quantitative but are nonetheless pertinent; see Figure 4.



Fig. 3. Heat distortion temperatures of cured resins vs. the number of carbon atoms in straight alkyl chains.

With the exception of the diheptadecyl diglycidyl ether, the amine-cured and anhydride-cured resins exhibited very similar Rockwell hardnesses. The diheptadecyl compound was much softer when amine cured than when anhydride cured. This relationship can be seen in Figure 5.

When aged at 500°F. the cured ethers exhibited a curious behavior. At the end of 24 hr., weight loss was found to be greater for longer chain length. However, after seven days the trend was reversed, and the weight loss was less for the methyl hepta-

decyl and diheptadecyl than for any other ethers.

The chemical resistance tests were performed on 0.5 in. $\times 0.5$ in. blocks immersed in 50 ml. of solvent. The blocks were weighed, and the gain or loss of weight noted at 24 hr. and 7 days at ambient temperature and again after 24 hr. at reflux. The reagents were water, 10 wt.-% sodium hydroxide, 30 wt.-% sulfuric acid, and acetone. The results are given in Table V. In general, the long chain members show better resistance to aqueous base and acid than the shorter chain members, probably a re-



Fig. 4. Bisphenol melting point vs. heat distortion temperature for (O) amine-cured and (Δ) anhydride-cured lower molecular weight diglycidyl ethers: (1) acetone-derived; (2) methyl isobutyl ketone-derived; (3) methyl ethyl ketone-derived; (4) methyl amyl ketone-derived; (5) methyl nonyl ketone-derived.



Fig. 5. Rockwell hardness of cured resins vs. the number of carbon atoms in straight alkyl chains.

							Weig	ht chang	e, %					
		Water			30% Sulfuric acid			10% S	10% Sodium hydroxide			Acetone		
	Cur-	Ambient temp.		Reflux.	Ambient temp.		Reflux	Ambient temp.		. Reflux.	Ambient temp.		Beflux.	
Diglycidyl ether	agent	24 hr.	7 days	24 hr.	24 hr.	7 days	24 hr.	24 hr.	7 days	24 hr.	24 hr	7 days	24 hr.	
Methyl methyl	Z PA	0.08	0.23	1.05	0.07	0.20	1.58	0.07	0.19	1.04	0.21	0.82	1.91	
Methyl ethyl	Z PA	0.07	0.17	0.87	0.05	0.13	1.29	0.05	0.12 0.09	0.83	0.28	1.55	4.11	
Methyl isobutyl	Z	0.07	0.21	0.90	0.06	0.15	1.06	0.07	0.17	0.84	0.75	2.55 2.96	4.74	
Methyl amyl	Z PA	0.08	0.20	0.91	0.05	0.10	1.12	0.06	0.17	0.85 -0.07	1.43	4.49 4.83	7.88	
Methyl nonyl		0.07	0.15	0.92	0.07	0.10	1.27	0.05	0.13	0.85	3.70	10.79 10.46	17.11 Fractured	
Methyl heptadecy		0.08	0.10	0.91	0.02	0.10	1.12	0.06	0.13	0.32 0.79	5.52	14.29	21.28	
Diheptadecyl	Z PA	0.02 0.08 0.00	0.07 0.16 0.02	$0.01 \\ 0.72 \\ 0.55$	0.01 0.04 -0.01	0.04 0.09 -0.01	0.48 0.66 0.23	0.01 0.04 -0.01	0.03 0.11 -0.02	-0.57 0.61 -0.52	7.64 7.08	12.00 16.66 15.03	22.49 24.37	

TABLE V. Chemical Resistance of Cured Resins

sult of the greater hydrophobic character of the long chains. With acetone, the reverse was true; the amount of acetone absorbed increased with increasing chain length. No effect attributable to chain branching was noted. The acetone absorption after 24 hr. is shown in Figure 6.

EXPERIMENTAL

1. Preparation of Diheptadecyl Ketone

Stearic acid (44 g.) and magnesium oxide (190 g.) were placed in a one-liter flask equipped with a con-

denser maintained at 100–150 °C. by steam. The flask and contents were heated to 340 °C. Molten stearic acid (240 g.) was added in 10-g. portions at 15 min. intervals. After the addition was complete, heating was continued until the total reaction time was 10 hr. The product was cooled, boiled with one liter of 4N sulfuric acid to remove magnesium salts, washed with water, and then boiled with one liter of 5% sodium hydroxide to remove unreacted fatty acid. It was then successively washed with one-liter portions of boiling water until the washings were clear. The ketone was allowed to solidify and



Fig. 6. Acetone absorption of cured resins after 24 hr. immersion at 25°C. vs. the number of carbon atoms in the straight alkyl chains.

was then recrystallized from acetone as glistening white flakes, m.p. 88-89°C., yield 91-95%.

2. Preparation of the Bisphenols

The phenol and ketone (5:1 molar ratio of phenol:ketone) were charged to a resin kettle equipped with a thermometer, stirrer, reflux condenser, and a gas inlet tube immersed in the reactants. The mercaptan auxiliary catalyst, 1.0 wt.-% of a 1:1 (w/w) mixture of methyl and lauryl mercaptans, was added and the mixture stirred and brought to temperature. Anhydrous hydrogen chloride was then bubbled through the mixture, removed from the top of the condenser, and passed through a column of mercury sufficient to maintain a back pressure of 1 lb. The gas was then passed through water and vented to the hood. At the conclusion of the reaction period the mixture was vacuum-stripped to 180°C. at less than 1 mm. Hg pressure, steam-distilled until no odor of phenol was detectable in the distillate, and again vacuum-stripped.

3. Purification of the Crude Bisphenols

Purification of the bisphenols was carried out after removal of the excess phenol by distillation and steam distillation.

Methyl Ethyl Bisphenol. The crude product was crystallized from chlorobenzene, recrystallized twice from benzene, and washed twice with boiling water. It was then twice recrystallized from benzenehexane, washed with hexane, and dried at 100°C. for 16 hr. at 1 mm. Hg.

Methyl Isobutyl Bisphenol. The bisphenol was recrystallized from benzene five times. It was then packed into a 3×24 in. cylindrical tube fitted with a sintered glass filter at the bottom and a steam inlet at the top. Steam was passed into the top of the tube and water removed from the bottom. After 24 hr. the bisphenol was removed and twice recrystallized from aqueous methanol and dried at 100°C. for 16 hr. at 1 mm. Hg.

Methyl Amyl Bisphenol. The stripped product was recrystallized three times from benzene and twice from benzene-hexane. It was then distilled under 60μ Hg pressure. The fraction boiling at 220– 240°C. was thoroughly washed with warm water and then steam-distilled under house vacuum. The mixture was then cooled, the water decanted off, and the bisphenol recrystallized twice from benzene and dried at 100°C. for 16 hr. at 1 mm. Hg.

Methyl Nonyl Bisphenol. The resinous bisphenol was melted with an equimolar amount of phenol. The adduct crystallized on cooling. It was broken up and slurried with hexane in a Waring Blendor and filtered. This process was repeated until the crystals were no longer sticky. The material was then recrystallized from hexane containing a little benzene. The phenol was removed by vacuum distillation and steam distillation. The bisphenol was crystallized twice from a chlorobenzene-hexane (60-40 wt.-%) mixture and dried at 50 °C. for 16 hr. at 1 mm. Hg.

Methyl Heptadecyl Bisphenol. The procedure outlined above for methyl nonyl bisphenol was duplicated except for the final crystallizations. This material was crystallized from chlorobenzene and twice recrystallized from acetone and dried at 75°C. for 16 hr. at 1 mm. Hg.

Diheptadecyl Bisphenol. The mixture of bisphenol and unreacted ketone was dissolved in the minimum amount of boiling acetone and then cooled to 0°C. The precipitated ketone was filtered and washed with acetone at 0°C. The washings and filtrate were combined and vacuum stripped of acetone. The bisphenol was then recrystallized twice from hexane and three times from 95% ethanol. It was then dried at 75°C. for 16 hr. at 1 mm. Hg.

4. Preparation of the Glycidyl Ethers

Reactants used were bisphenol (1 mole), epichlorohydrin (10 moles), acetone (equivalent to epichlorohydrin by weight), water (6 wt.-% of the first three reactants), and sodium hydroxide (2.2 moles as a 20 wt.-% aqueous solution). The first four reactants were charged to a suitable vessel equipped with a mechanical stirrer, a reflux condenser, and a dropping funnel containing the caustic solution. The mixture was rapidly stirred and heated to reflux. The base solution was then added in units of 1/6 of the total volume at 15 min. intervals. After the fifth addition and 15 min. reflux period, the mixture was allowed to separate into two phases and the aqueous layer drawn off and discarded. The stirring and refluxing were restarted and the last base addition made. Refluxing was continued 15 minutes and the aqueous phase removal repeated. The resin solution was then stripped to 170°C. at 5μ Hg pressure.

5. Distillation of the Glycidyl Ethers

The methyl heptadecyl compound was distilled in a Consolidated Vacuum Corporation centrifugal still; all of the other diglycidyl ethers were distilled in an A. F. Smith wiper-type molecular still.

6. Viscosity Determinations

The viscosities of all of the pure diglycidyl ethers were measured in Ubbelohde tubes at 25°C.

7. Curing Conditions

The amine curing agent was added to the diglycidyl ethers at concentrations based on the epoxide equivalent of the ether. The formula followed was: grams of curing agent per 100 g. of diglycidyl ether = 4000/WPE (weight per epoxide). The mixture was then centrifuged to remove bubbles and poured into a mold. The cure schedule was 2 hr. at 80°C. followed by 1 hr. at 200°C.

The anhydride curing agent was added to the epoxide at a level of one mole of phthalic anhydride per equivalent of epoxide, with 0.1 phr benzyldimethylamine as promoter. The cure schedule was 2.8 hr. at 115°C. followed by 20 hr. at 150°C.

CONCLUSIONS

Seven bisphenols covering a range of molecular weight and structural features were synthesized and smoothly converted to their diglycidyl ethers. The viscosities of the diglycidyl ethers were found to be quite dependent on the size and configuration of the central hydrocarbon group. The diglycidyl ethers which could assume spherical configurations showed generally low viscosities, whereas one with a tridentate shape possessed a high viscosity.

All of the ethers were cured with an amine and with an anhydride curing agent. The cured resins were then compared, with respect to thermal, mechanical, and chemical resistance properties, to the diglycidyl ether of bisphenol A. The ethers derived from methyl ethyl and methyl isobutyl ketone gave cured products that were comparable in performance properties to the acetone derived ether, except for a slightly lower heat distortion temperature. As the chain length increased to the amyl and nonvl derivatives, the performance properties were adversely affected. In the case of the two long chain derivatives, the methyl heptadecyl and diheptadecyl compounds, a marked improvement in resistance toward aqueous acid and alkali was realized, but at the expense of lowered performance in all other properties.

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Synopsis

An investigation of the relationship between molecular structure and polymer performance has been made for a series of six diglycidyl ethers. The resins evaluated were those based on the bisphenols derived from methyl ethyl, methyl isobutyl, methyl n-amyl, methyl n-nonyl, methyln-heptadecyl and di-n-heptadecyl ketones. The first three members of this series gave performances close to those of the diglycidyl ether of p, p'-bisphenol acetone. The long chain members of the series showed poorer over-all performance but did exhibit significant hydrophobic character in the cured specimens. The cured diglycidyl ethers showed variation attributed to both length and configuration of the alkyl chain. An increase in length of straight chain resulted in lowered heat distortion temperature, hardness, shear strength, and organic solvent resistance. Branching in the alkyl chain imparts improved performance characteristics.

Résumé

On a étudié la relation qui existe entre le structure moléculaire et la capacité polymérique d'une série de six éthers diglycidiques. Les résines testées sont des dérivés biphénoliques de la méthyl-éthyl, la méthyl-isobutyl, la méthylheptadécyl, et a di-n-heptadécylacétone. Les trois premiers éléments de cette série possèdent des capacités qui se rapprochant de l'éther diglycidique de la p-p'-bisphenol-acétone. Les éléments de cette série qui ont des longues chaînes se caractérisent par une capacité totale moindre, mais les échantillons vulcanisés possèdent un caractère hydrophobe prononcée. On observe chez les éthers diglycidiques étudiés une variation qui peut être attribuée aussi bien á la longueur qu'á la configuration de la chaîne alcoyle. Un allongement de la chaîne rectiligne provoque un abaissement de température de distortion thermique, une rigidité et une résistance á la force de cisaillement et aux solvants organiques. Une ramifiication de la chaîne alcoylée provoque une amélioration des caractéristiques de ces capacités.

Zusammenfassung

An einier Reihe von sechs Diglycidyläthern wurde eine Untersuchung des Zusammenhangs zwischen Molekülstruktur und Gebrauchseigenschaften der Polymeren ausgeführt. Es wurden Harze auf Grundlage der von Methyläthyl-, Methylisobutyl-, Methyl-n-amyl-; Methyl-n-nonyl-, Methyln-heptadecyl- und Di-n-heptadecylketon abgeleiteten Bisphenole herangezogen. Die ersten drei Glieder dieser Reihe erweisen sich in ihren Eigenschaften dem Diglycidyläther von p.p'-Bisphenolaceton ähnlich. Die langkettigen Glieder der Reihe zeigten schlechtere Gesamteigenschaften, wiesen aber als gehärtete Proben einen charakteristischen, hydrophoben Charakter auf. Die gehärteten Diglycidyläther liessen sowohl einen Einfluss der Länge als auch der Konfiguration der Alkylkette erkennen. Erhöhung der Länge der unverzweigten Kette ergibt eine niedrigere Wärmeformbeständigkeit, Härte, Scherfestigkeit und Beständigkeit gegen organische Lösungsmittel. Verzweigung in der Alkylkette verleiht eine bessere Gebrauchscharakteristik.

Received October 5, 1961 Revised November 13, 1961