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Interfacial Polycondensation. VIII. Application to A—B-Type Monomers*

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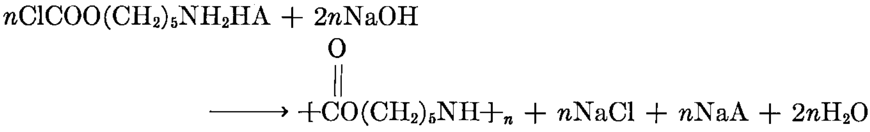
INTRODUCTION

The first of this series of papers[1] described the use of interfacial polycondensation for the laboratory preparation of polyamides, polyurethanes, polysulfonamides, polyureas, and polyphenyl esters. These condensations utilized a fast irreversible reaction between two difunctional starting materials, A—A and B—B, to give the polymer —A—AB—B—. In this paper, the application of interfacial polycondensation to the preparation of polyamides and polyurethanes from monomers containing both functional groups in a single molecule, i.e., from A—B type monomers, is discussed.

More specifically, for example, preparation of a polyamide by interfacial polycondensation involved the reaction at an interface of a diacid chloride in an organic solvent with a diamine plus an acid acceptor in an aqueous phase. It was possible to extend this method to the preparation of polyamides and polyurethanes from aminoacid chlorides and aminoalkyl chloroformates, respectively, by employing the simple expedient of blocking the amino groups by salt formation until the polymerization reaction was ready to begin. Evidently, polyureas and polysulfonamides from A—B monomers could have been synthesized by utilizing the same strategy. In carrying out the condensation, the monomer as the salt was dissolved in an organic solvent and added to a stirred aqueous caustic solution. Either hydrochloric or *p*-toluenesulfonic acid was used to form the salt. Since both acids are strong, dissociation is minimized; *p*-toluenesulfonic acid salts appeared to be more soluble in organic solvents. At least two moles of caustic per mole of monomer was required, for example:



$$n\mathrm{ClCOO(CH_2)_5NH_2HA} + 2n\mathrm{NaOH} \longrightarrow \left[\overset{\displaystyle \mathrm{O}}{\overset{\|}{\mathrm{C}}}\mathrm{O(CH_2)_5NH}\right]_n + n\mathrm{NaCl} + n\mathrm{NaA} + 2n\mathrm{H_2O}$$

This method has been anticipated to some extent by previous authors. Fischer,[2] many years ago, had prepared aminoacid chloride hydrochlorides,

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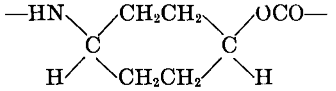
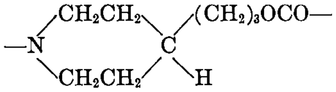
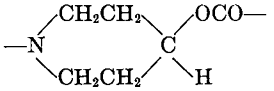
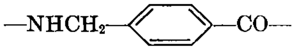
and Frankel, Liwschitz, and Zilkha[3] recently used these compounds to prepare low molecular weight polypeptides of DP 10–20 by heating the monomers *in vacuo* or by adding trimethylamine to *N*,*N*-dimethylformamide solutions of the monomers. The only previous use of an interface for this type of polymerization involved the synthesis of the polyurethane in the above equation from 5-aminopentyl chloroformate salts.[4]

Scope

As in the previous papers of this series, a few polymers were made which had also been made by melt or ring-opening methods. These included poly-ϵ-caproamide and poly-ω-undecanoamide. Others were high-melting types not capable of being prepared by melt polymerization (see Table I).

TABLE I

Polymers from A—B Type Monomers

Monomer	Polymer unit	Yield, %	η_{inh}[a]	T_m, °C.[b]
A. Polyurethanes				
(III)	$—NH(CH_2)_5OCO—$	83	0.94	155
(VII)		93	0.08	>335
(IX)		98	0.81	amorphous
(XI)		62	1.25	270
B. Polyamides				
(XVI)	$—NH(CH_2)_5CO—$	20	0.98	210
(XVIII)	$—NH(CH_2)_{10}CO—$	60	0.36	188
(XX)		78	0.15	~300

[a] Viscosity in this paper is expressed as inherent viscosity ($\eta_{inh} = [\ln \eta_{rel}]/c$) at a concentration of 0.5 g. polymer/100 ml. solution. The solvent is *m*-cresol and the temperature 30°C. unless otherwise designated.

[b] The melting points listed are the temperatures at which disappearance of birefringence occurred on a Kofler micro hot stage.

DISCUSSION

Method and Mechanism

Interfacial polycondensation in the A—B monomer system differs as to method and mechanism from the corresponding A—A plus B—B system due to three principal factors: (*1*) the A—B monomer is a salt; (*2*) both complementary functional groups are in the same molecule; (*3*) both functional groups are present in the organic phase.

The fact that the monomer is a *salt* creates certain problems. In the first place, it is difficult to purify on a large scale since it must be recrystallized from a nonreactive dry solvent such as benzene, chloroform, or dichloromethane. Some of the monomers are so insoluble in nonreactive solvents that they cannot be purified in this way, e.g., ε-aminocaproyl chloride hydrochloride (XVI). Furthermore, the monomers are sensitive to moisture and heat so that they must be kept dry and cold, or be recrystallized shortly before use. In carrying out interfacial polycondensation, insoluble monomers can usually be polymerized as a suspension in the organic phase. With all monomers, an additional complication arises in that the monomer will partition between the aqueous and organic phases. That portion of the material which dissolves in the aqueous phase will be rapidly hydrolyzed. This difficulty was overcome to a great extent by using high concentrations of inorganic salt, e.g., potassium carbonate or alkali, in the aqueous phase to "salt" the monomer into the organic phase. With very water-soluble salts, such as 4-aminobutyl chloroformate *p*-toluenesulfonate, or the lower molecular weight aminoacid chloride hydrochlorides, such as those derived from glycine or α- or β-alanine, even this method was ineffective for preventing extensive hydrolysis. Thus, 4-aminobutyl chloroformate *p*-toluenesulfonate salt gave a small amount of polymer in the organic phase, but most of the product was an oligomer of DP 4 or 5, which appeared to have been formed in the aqueous phase. Similarly, even ε-aminocaproyl chloride hydrochloride gave only low yields of high polymer. In this case, it is to be noted that the polymer formed in the organic phase was high polymer, even though the aqueous phase may have contained a very low molecular weight oligomer. A single organic phase containing an excess of a tertiary amine as an acid acceptor has been used by Frankel et al.[3] to avoid the hydrolytic problem. This was tried in this work also, with 5-aminopentyl chloroformate *p*-toluenesulfonate salt. Low molecular weight polymers were formed in both cases.[3]

Factors (2) and (3) above are interrelated and will be discussed as a unit. First of all, since both functional groups are in the same molecule, we do not have to adjust concentration, solvents, and partition equilibria to get a balance of reactants at the interface.[5] There will, it is true, be a partition of the monomer between the organic and aqueous phase, but the main effect of this will be to lower yield, as noted above. Thus, solvents such as chloroform, benzene, dichloromethane, and nitrobenzene all gave high molecular weight polyurethane polymer from 5-aminopentyl chloroformate *p*-toluenesulfonate salt. The second point to be noted is that all amino groups in these monomers are blocked by salt formation; therefore, the salt must diffuse to the interface to be unblocked by the inorganic base. This should give a much slower reaction than in the corresponding A—A plus B—B case. In beaker experiments[5] (both phases present in a beaker, not stirred), a polymeric film was formed at the interface in the A—B system, but it was not possible to remove it continuously. This showed that polycondensation is much slower than in the A—A plus B—B system, and/or that the reaction terminates when only very thin weak films are formed. It is probable that both factors must be taken into account. Thus, the acid by-product of condensation can be taken up by a second molecule of diamine in the A—A, B—B system, leading to a faster reaction, and also reaction at a greater distance from the interface (giving a thicker film) than in the A—B system. In order to facilitate the A—B type polycondensation, therefore, it was necessary to provide a maximum amount of interfacial surface and to use concentrated solutions of the monomer in the organic phase. This was accomplished by the use of vigorous stirring and of detergents such as sodium lauryl sulfate.

The type of products formed by hydrolysis of an A—B monomer is the next point of discussion. An aminoalcohol is formed in the case of the polyurethanes and an aminoacid (as the alkali metal salt) in the case of the polyamides. Both of these materials are chain terminators and will tend to be "salted" into the organic phase. For example, it was found that the addition of sodium ϵ-aminocaproate to the aqueous solution in the polymerization of ϵ-aminocaproyl chloride hydrochloride lowered the inherent viscosity of the polymer produced. Thus, it is necessary to carefully adjust the concentration of alkali to secure maximum molecular weight. Furthermore, the aminoalcohol and the aminoacid, respectively, are likely impurities in the monomer and, therefore, it is necessary to purify monomers very carefully in order to obtain high molecular weight polymer. In this connection, it is probable that the aminoalcohol will be a worse offender than the aminoacid since the latter will tend to form a zwitterion salt and to be partitioned mainly into the aqueous phase. Thus, it was found that the aminoacid chloride hydrochlorides gave high polymer without careful purification, whereas the aminoalkyl chloroformate salts had to be carefully recrystallized before use.

Lastly, temperatures in the range of 0–30°C. were found to be optimum for preparing polymers. Higher temperatures resulted in decreased yield and inherent viscosity of the polymers.

Yield and Molecular Weight

To summarize, yield depended mainly on the use of a monomer salt which was relatively insoluble in water and the use of the right concentration of inorganic acid acceptor in the aqueous phase to "salt" out the monomer. A low temperature minimized hydrolysis. Other factors were less important.

To obtain high molecular weight polymers, it was necessary to use pure reactants (especially in the case of polyurethanes), a temperature in the range of 0–30°C., and vigorous agitation. In addition, another factor was found to be important. It was stressed in a previous paper[5] that a fast reaction is necessary to build up to a high molecular weight before precipitation of the polymer occurred. In the monomers we have been discussing, reaction is slower because we are dealing with a monomer which is a salt. This means that very insoluble polymers, such as those derived from ring-containing compounds like (VII) and (XX), precipitate before high molecular weight is reached. Hence, polymers have to be soluble or somewhat swollen to obtain high molecular weight.

Intermediates

Most of the salts and the monomers described in this paper (see Experimental) have not been reported previously.

Polymers

The polyurethanes derived from the chloroformic ester salts of 5-aminopentanol, 3-(γ-piperidyl)propanol, and 4-hydroxypiperidine were all of high enough molecular weight to yield either drawable fibers or a rubberlike material. The polymer based on 4-hydroxypiperidine was amorphous as formed. It was cast as a transparent film from a mixture of chloroform and methanol. These films were boiled in water and then drawn 470% at 140° on a heated cylinder to yield a crystalline, highly oriented material with a crystalline melting point of 270°C. This is slightly higher than that for the related A—A plus B—B polyurethane, poly(ethylene piperazinedicarboxylate),[1] T_m 245°C., which has a higher concentration of functional groups but does not have as stiff a chain. The introduction of three methylene groups, however, in the polyurethane based on 3-(γ-piperidyl)propanol causes a remarkable change in properties, this polymer being an amorphous, almost rubberlike, material with a softening temperature of 110°C.

EXPERIMENTAL

Polyurethane from 5-Aminopentyl Chloroformate Salts

Materials

Pentanolamine (I) was obtained by reductive amination of hydroxyvaleraldehyde,[6] b.p. 95°C./6 mm., 56% yield. The *p*-toluenesulfonate salt (II) was formed from equimolar amounts of (I) and *p*-toluenesulfonic acid monohydrate in ethyl acetate solution. It was recrystallized from an ethyl acetate-alcohol mixture ($^{90}/_{10}$), m.p. 111°C., 51% yield.

ANAL.: Calc. for $C_{12}H_{21}NO_4S$: C 52.4%, H 7.70%, N 5.09%. Found: C 52.4%, H 7.61%, N 5.04%.

5-Aminopentyl chloroformate p-*toluenesulfonate salt* (III) was obtained by stirring a suspension of 10 g. of (II) in 100 g. of phosgene for 0.5 hr. under reflux, after which 50 g. of chloroform was added and the solution stirred for an additional half hour. The phosgene was evaporated, leaving a clear solution. Evaporation of the chloroform left (III) as white needles which were recrystallized from dry benzene.

ANAL.: Calc. for $C_{13}H_{20}ClNO_5S$: C 46.2%, H 6.0%, N 4.2%, Cl 10.5%. Found: C 46.0%, H 5.8%, N 4.4%, Cl 10.8%.

5-Aminopentyl chloroformate hydrochloride (IV). Anhydrous hydrogen chloride and (I) (10 g.) at 0°C. gave pentanolamine hydrochloride. Reaction of the latter with a mixture of 10 ml. of dichloromethane and 50 ml. of phosgene under reflux for 7 hr. gave a clear solution of (IV). This product was precipitated from dichloromethane solution by petroleum ether as a white, fluffy material in 50% yield. It was soluble also in tetramethylene cyclic sulfone.

ANAL.: Calc. for $C_6H_{13}Cl_2NO_2$: C 35.7%, H 6.5%, N 6.94%, Cl 35.1%. Found: C 35.6%, H 6.5%, N 7.35%, Cl 35.7%.

Polymerization

(*1*) A solution of 1 g. of recrystallized (III) in 5 ml. of purified chloroform[7] was added to 5 ml. of stirred 4*M* potassium carbonate solution. This mixture was stirred vigorously and maintained at a temperature of 23–24°C. A white precipitate of polymer formed almost immediately upon the addition of the chloroform solution. After 0.5 hr. the precipitated polymer was filtered, washed with water until free of salts, and dried. An 83% yield of a fiber-forming polymer with an inherent viscosity of 0.94 was isolated (see footnote to Table I).

ANAL.: Calc. for $C_6H_{11}NO_2$: C 55.8%, H 8.6%, N 10.8%. Found: C 55.6%, H 8.3%, N 10.7%.

(*2*) A solution of 3.6 g. of (IV) in 8 ml. of dichloromethane was added to excess stirred 20% aqueous sodium hydroxide at room temperature to give a 74% yield of polymer with an inherent viscosity of 1.13.

(*3*) Excess triethylamine was added to a solution of (IV) in tetramethylene cyclic sulfone. After 1 hr. at room temperature, a 42% yield of the same polymer with an inherent viscosity of 0.19 was isolated.

Polyurethane from 4-Aminobutyl Chloroformate *p*-Toluenesulfonate Salt (V)

Materials

Butanolamine p-*toluenesulfonate salt* (VI) was prepared from equimolar amounts of the amine and acid in ethyl acetate solution. The salt was precipitated, isolated by filtration, and recrystallized from an ethyl acetate–ethyl alcohol mixture (90/10), m.p. 77–78°C., 58% yield.

ANAL.: Calc. for $C_{11}H_{19}NO_4S$: C 50.5%, H 7.3%, N 5.36%. Found: C 50.7%, H 7.2%, N 5.31%.

4-Aminobutyl chloroformate p-*toluenesulfonate salt* (V) was obtained by the reaction of a suspension of 10 g. of (VI) in 100 g. of phosgene, which was essentially complete in 1.5 hr. The product was completely soluble in chloroform and was recrystallized from benzene, m.p. 127–128°C., 83% yield.

ANAL.: Calc. for $C_{12}H_{18}ClNO_5S$: C 44.5%, H 5.6%, N 4.3%, Cl 10.9%. Found: C 44.6%, H 5.4%, N 4.4%, Cl 10.5%.

Polymerization

Several polymerizations were carried out of 10% solutions of (V) in purified chloroform.[7] These solutions were added to 200 ml. of vigorously stirred 4*M* potassium carbonate maintained at 0°C. A small quantity (3%) of white, presumably polymeric, product, m.p. 190–196°C., was obtained. Analyses were low due to contamination with inorganic material. Apparently most of the monomer had been converted to a low molecular weight water-soluble polyurethane, since the major product was a liquid with a neutral equivalent of 400, roughly corresponding to a DP of 4.

Polyurethane from 4-Aminocyclohexyl Chloroformate *p*-Toluenesulfonate Salt (VII)

Materials

4-Aminocyclohexanol was obtained in 61% yield by hydrogenation of recrystallized (from methanol) *p*-aminophenol (Eastman-Kodak Co.) over Raney nickel by the method of Senderens and Aboulenc,[8] b.p. 93–96°C./0.5 mm. The *p*-toluenesulfonate salt (VIII) formed when concentrated solutions of the acid and the amine in alcohol were mixed. Addition of ether precipitated the salt, m.p. 190–230°C. The large range in melting point probably resulted from the presence of a mixture of the *cis* and *trans* isomers. At times the salt separated as a liquid phase and it was difficult to induce crystallization.

Anal.: Calc. for $C_{13}H_{21}NO_4S$: C 54.4%, H 7.36%, N 4.87%. Found: C 54.3%, H 7.09%, N 4.83%.

4-Aminocyclohexyl chloroformate p*-toluenesulfonate salt* (VII). A suspension of 10 g. of VIII in 100 g. of phosgene containing 0.5 cc. of triethylamine gave a product (VII) after 3–4 hr. which was completely soluble in chloroform. On recrystallization from chloroform-benzene mixtures it melted at 128–130°C.

Anal.: Calc. for $C_{14}H_{20}ClNO_5S$: C 48.1%, H 5.8%, N 4.01%, Cl 10.1%. Found: C 48.3%, H 5.8%, N 4.30%, Cl 9.7%.

Polymerization

A 10–20% chloroform solution of (VII) was added quickly to excess stirred 4*M* potassium carbonate at 25°C. After 30 min. a 93% yield of polymer with an inherent viscosity of 0.08 (in H_2SO_4) was isolated. This polymer was crystalline and melted at >335°C. In other experiments, yields of polymer varied from 38 to 100%, but the inherent viscosities were never greater than 0.10. Some samples were soluble in *m*-cresol, others only in H_2SO_4.

Polyurethane from 3-(γ-Piperidyl)propyl Chloroformate *p*-Toluenesulfonate Salt (IX)

Materials

3-(γ-Piperidyl)propanol p*-toluenesulfonate salt* (X). Redistilled (b.p. 164°C./14 mm.) 3-(γ-pyridyl)propanol (Eastman-Kodak Co.) was reduced in 84% yield to 3-(γ-piperidyl)-propanol. b.p. 125–130°C./0.5 mm. by the method of Burtner and Brown,[9] except that only water was used as a solvent. The latter compound was converted to the *p*-toluenesulfonate salt (X) in ethyl alcohol. The salt was precipitated by addition of ether and crystallized from an ethyl acetate-ethyl alcohol mixture (95/5), m.p. 116–118°C., 81% yield.

ANAL.: Calc. for $C_{15}H_{25}NO_4S$: N 4.44%. Found: N 4.20%.

3-(γ-Piperidyl)propyl chloroformate p*-toluenesulfonate salt* (IX). The reaction of (X) with phosgene was complete in 3 hr. The product (IX) was crystallized from benzene, m.p. 118–119°C. The analyses differed from the theoretical, yet high polymer was produced.

ANAL.: Calc. for $C_{16}H_{24}ClNO_5S$: C 50.8%, H 6.4%, N 3.7%, Cl 9.4%. Found: C 49.6%, H 6.4%, N 4.1%, Cl 10.6%.

Polymerization

(IX) was polymerized by adding a solution of 1 g. of monomer in 5 ml. of dichloromethane to 200 ml. of vigorously stirred 1*M* sodium carbonate solution maintained at 6–7°C. After 1 hr. the polymer was filtered, washed thoroughly, and dried. A yield of 98% of polymer with an inherent viscosity of 0.81 was obtained. The polymer was a rubberlike, amorphous material which was soluble in a methanol-chloroform mixture (15/85), *m*-cresol, and formic acid. Solvent and thermal treatments failed to crystallize the polymer.

Salt Polyurethane from γ-Piperidyl Chloroformate *p*-Toluenesulfonate Salt (XI)

Materials

4-Hydroxypiperidine (XIII). Pyridine was converted to γ-piperidyl pyridinium dichloride by the procedure of Koenigs and Greiner[10] in 57% yield. The latter compound was hydrolyzed by the procedure of Leis and Curran[11] by refluxing a 40% aqueous solution for 80–100 hr. to give a 66% yield of 4-hydroxypyridine (XII), b.p. 182°C./1.5 mm., m.p. 151–152°C. Hydrogenation of 20 g. of (XII) in 18 g. of water over 7.5 g. of ruthenium (5%) on carbon at 140°C. and 1700 psi hydrogen pressure for 3 hr. gave 4-hydroxypiperidine (XIII). The yield was 57% of material, b.p. 99°C./15 mm., m.p. 88–91°C. Attempts to reduce (XII) in ethyl alcohol by the method of Campbell et al.[12] were unsuccessful, for in the presence of ruthenium on carbon in alcohol a 39% yield of *N*-ethyl-4-hydroxypiperidine (XIV), b.p.

99–101°C./14 mm., was obtained instead of the expected 4-hydroxypiperidine. (XIV) formed a crystalline phenylurethane, m.p. 130.5–131°C. (literature value[13] 125–128). The identity of (XIV) was further established by elementary analysis and by its infrared spectrum.

ANAL.: Calc. for $C_7H_{15}NO$: C 65.1%, H 11.53%. Found: C 65.1%, H 11.51%.

4-Hydroxypiperidine p-*toluenesulfonate salt* (XV) was obtained in ethyl acetate solution. The salt precipitated as an oil which slowly crystallized at 0°C., m.p. 109–111°C., 97% yield.

ANAL.: Calc. for $C_{12}H_{19}NO_4S$: N 5.12%. Found: N 4.91%.

γ-Piperidyl chloroformate p-*toluenesulfonate salt* (XI) was obtained after 4–6 hr. reaction of a suspension of (XV) in phosgene. The product was crystallized from mixtures of benzene and cyclohexane (75/25) to give white needles, m.p. 117–120°C., 40% yield.

ANAL.: Calc. for $C_{13}H_{18}ClNO_5S$: C 46.5%, H 5.40%, N 4.17%, Cl 10.56%. Found: C 46.0%, H 5.45%, N 4.07%, Cl 10.47%.

Polymerization

A 14% solution of the monomer (XI) in dichloromethane was added to a stirred 1*M* sodium carbonate solution containing 0.67% sodium lauryl sulfate at 4–5°C. After 1 hr. the precipitated polymer was filtered, washed, and dried to give a 62% yield of polymer of inherent viscosity 1.25.

Polyamide from ε-Aminocaproyl Chloride Hydrochloride (XVI)

Materials

ε-Caprolactam was hydrolyzed to ε-aminocaproic acid hydrochloride (XVII) in 85–96% yield; 5 g. (XVII) was mixed with 5 ml. (2.4 equivalents) of thionyl chloride in a 50-ml. flask fitted with a drying tube. After 15 min. a clear solution had formed. This solution was heated to 50–60°C. for 1–2 hr., and a deep red color formed. Excess thionyl chloride was removed *in vacuo.* A reddish-brown solid (XVI) remained which could be used directly for polymerization. Thoroughly washing with carbon tetrachloride, ether, and petroleum ether, in turn, yielded a white product which analyzed for 36.4–37.9% chlorine compared with a theoretical value of 38.1%. The compound was sparingly soluble in all inert solvents suitable for use in interfacial polycondensation.

Polymerization

Crude (XVI), 6.6 g., was suspended in 90 ml. ethylene chloride (in which it was slightly soluble); the suspension was added to a stirred solution of 15 g. of potassium carbonate in saturated sodium chloride solution at room temperature. A yield of 20% of poly-ε-caproamide of inherent viscosity 0.98 was obtained.

Polyamide from 11-Aminoundecanoyl Chloride Hydrochloride (XVIII)

Materials

11-Aminoundecanoic acid was prepared from 11-bromoundecanoic acid (Aldrich Chemical Co.) according to an example in the patent literature[14] in 22% yield, m.p. 180–182°C. (decomposition). The aminoacid hydrochloride (XIX) was obtained by mixing 1 g. of the amino acid with 1 ml. of concentrated hydrochloric acid in 4 ml. of water. Precipitation by addition of 50 ml. of acetone gave a 75% yield of (XIX).

ANAL.: Calc. for $C_{11}H_{24}ClNO_2$: Cl 14.9%. Found: Cl 14.9%.

11-Aminoundecanoyl chloride hydrochloride (XVIII) was prepared from (XIX) by the thionyl chloride method described above. The final product was very soluble in ethylene chloride and in tetramethylene cyclic sulfone.

Polymerization

A solution of 0.75 g. of (XVIII) in 3.5 ml. of ethylene chloride was added to a stirred 5% sodium hydroxide solution at room temperature. After 1 hr. a polymer with an inherent viscosity of 0.36 was isolated by filtration in 60% yield.

Polyamide from *p*-(Aminomethyl)benzoyl Chloride Hydrochloride (XX)

Materials

p-*(Aminomethyl)benzoyl chloride hydrochloride* (XX) was prepared from *p*-tolunitrile (Eastman-Kodak Co.) by a literature method[15] in an overall yield of 9%. The only solvent found was diethyl phosphite in which XX was only slightly soluble.

ANAL.: Calc. for $C_8H_9Cl_2NO$: Cl 34.4%. Found: Cl 34.6%, 34.9%.

Polymerization

A suspension of 1 g. of (XIX) in 10 ml. of diethyl phosphite was added to excess stirred 10% sodium hydroxide solution at room temperature. A 78% yield of polymer with an approximate melting point of 300°C. and an inherent viscosity of 0.15 was isolated.

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Synopsis

The application of interfacial polycondensation to the preparation of several polyurethanes and polyamides from A—B type monomers is described. The monomers are salts of aminoalkyl chloroformates and aminoacid chlorides, respectively. Differences in method and mechanism from the A—A plus B—B type interfacial polycondensation are discussed. Several new ring-containing polymers were synthesized.

Résumé

L'application de la polycondensation interfaciale à la préparation de plusieurs polyuréthannes et polyamides au départ de monomères du type A—B a été décrite. Les monomères sont des sels de chloroformiates d'aminoalcoyle et des chlorures d'acides aminés, respectivement. Les différences de méthode et de mécanisme comparées à la polycondensation interfaciale du type A—A plus B—B sont soumises à discussion. On a synthétisé de nombreux polymères contenant des cycles nouveaux.

Zusammenfassung

Es wird die Anwendung der Grenzflächenpolykondensation zur Darstellung mehrerer Polyurethane und Polyamide aus Monomeren vom A—B-Typ beschrieben. Die Monomeren sind Salze von Aminlalkylchlorformiaten bzw. Aminosäurechloriden. Die Unterschiede gegenüber dem Verfahren und dem Mechanismus bei der Grenzflächenpolykondensation vom A—A- plus B—B-Typus werden diskutiert. Einige neue Polymere, die Ringe enthalten, werden synthetisiert.

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