

and then was covered with aqueous acetone. The solid was collected and dissolved in hot acetic acid. An equal volume of water was added to the solution to precipitate any unreacted xanthone. The neutral material was removed by filtration and the filtrate was then made alkaline. The product which separated was dissolved in chloroform. The solution was evaporated *in vacuo* to leave an oil which was dissolved in absolute alcohol. The alcohol solution was

treated with dry hydrogen chloride whereupon the desired xanthone hydrochloride separated. After crystallization from absolute ethanol there was obtained 4.4 g. of the desired salt in a state of satisfactory purity, m.p. 178.5–181° (cor.).

The xanthones reported in Table III were prepared by the above method or some minor modification thereof.

RENSSELAER, NEW YORK

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF THE UNIVERSITY OF DELAWARE]

The Effect of Alkali on Linear Polyurethans¹

BY ELIZABETH DYER AND GEORGE W. BARTELS, JR.²

RECEIVED SEPTEMBER 3, 1953

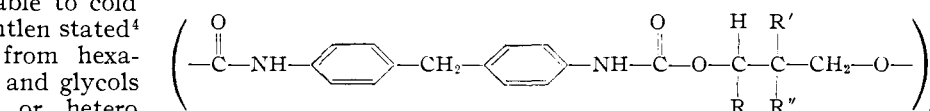
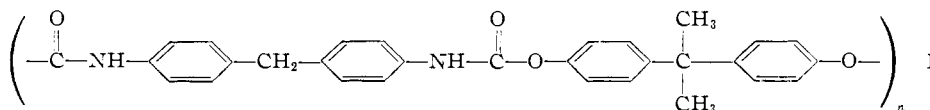
New polyurethans have been prepared by the reaction of methylene-bis-(4-phenyl isocyanate) and hexamethylene diisocyanate with various branched 1,3-diols and with two dihydric phenols. The polyurethans made from aliphatic diols were unaffected by 1% sodium hydroxide at 50°, whereas the polyurethans from phenols were attacked. Quantitative determinations were made of the hydrolysis products, which included carbon dioxide, the diamine, the phenol and a polyurea. The polyurea was shown to result from the action of the diamine on unchanged polyurethan.

The behavior of polyurethans toward alkali has been mentioned, but not described in detail. The fibrous polyurethan obtained by the action of hexamethylene diisocyanate on 1,4-butanediol (Igamid U) was reported³ to be stable to cold and hot alkalis. Höchtlen stated⁴ that polymers made from hexamethylene diisocyanate and glycols containing side chains or hetero atoms had less stability to alkali than Igamid U. Bayer mentioned⁵ the high alkali resistance of molded plastics prepared from high melting diisocyanates (such as di-*p*-xylylene methane) and polyhydroxy compounds. The cross-linked polyester-polyurethans known as Vulcollans were said⁶ to have a somewhat limited resistance to alkali; a sample was completely decomposed after boiling for 12 hours in contact with 15% sodium hydroxide.

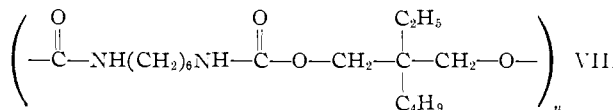
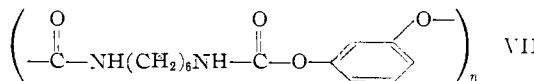
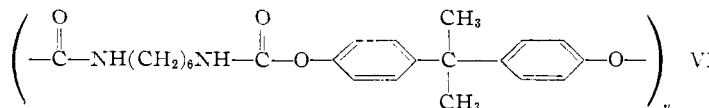
In the present investigation eight linear polyurethans were prepared, which represent the four types obtainable from (a) aromatic diisocyanates and dihydroxy phenols, (b) aromatic diisocyanates and aliphatic diols, (c) aliphatic diisocyanates and dihydroxy phenols and (d) aliphatic diisocyanates and aliphatic diols.

These polyurethans were obtained from the interaction of the diisocyanate and the dihydroxy compound either in solution^{5,7} using triethylamine as catalyst or in a melt^{3,8} with no solvent and no catalyst. Polymers I, II and III were prepared by the solution method in yields of 73–95%, while the

others were obtained by bulk polymerization in nearly quantitative yields. The chief advantage of



II, R = H, R' = CH₃, R'' = CH₃
 III, R = H, R' = C₂H₅, R'' = C₂H₅
 IV, R = H, R' = C₂H₅, R'' = *n*-C₄H₉
 V, R = *n*-C₃H₇, R' = H, R'' = C₂H₅



the bulk method was the avoidance of products which differed from the desired polyurethans in having higher nitrogen contents, and decreased solubility and film-forming characteristics. It is probable that these products contained a homopolymer of the diisocyanate, since methylene-bis-(4-phenyl isocyanate) was shown to form an insoluble product in solvents containing triethylamine. The absence of such homopolymers in the polyurethans II–IX was shown by the fact that these substances were completely soluble in acetone, a non-solvent for the isocyanate homopolymer.

The properties of the polyurethans are given in Table I. The polymers were rather brittle in nature, formed films and frequently fibers, but had no

- (1) From the Ph.D. dissertation of George W. Bartels, Jr., University of Delaware, Sept., 1953.
- (2) Armstrong Cork Company Research Fellow, 1951–1953.
- (3) J. DeBell, W. Goggin and W. Gloor, "German Plastics Practice," DeBell and Richardson, Cambridge, Mass., 1946, p. 305.
- (4) A. Höchtlen, *Kunststoffe*, **42**, 303 (1952).
- (5) O. Bayer, *Angew. Chem.*, **59**, 257 (1947).
- (6) O. Bayer, E. Müller, S. Petersen, H. F. Piepenbrink and E. Windemuth, *ibid.*, **62**, 65 (1950).
- (7) C. S. Marvel and J. H. Johnson, *THIS JOURNAL*, **72**, 1874 (1950).
- (8) J. H. Brewster, *ibid.*, **73**, 368 (1951).

TABLE I
 LINEAR POLYURETHANS FROM 1,3-DIOLS AND PHENOLS

Iso-cyanate	Reactants		Properties of polymers					Analyses, %		Nitrogen ^e		
		Dihydroxy cpd.	Physical states ^a	Softening and dec. temp., °C	Solu- bility ^b	$[\eta]$ ^c at 25°	Carbon ^d Calcd.	Carbon ^d Found	Hydrogen Calcd.	Hydrogen Found	Calcd.	Found
I	MDI ^f	Bisphenol A ^g	Powder. Brittle films and fibers	S ^h 190-193 Dec. 260	C, D, N						5.85	6.10
II	MDI	2,2-Dimethyl-1,3-propanediol	Powder. Brittle films and fibers	S 180-190 Dec. 280	A, C, D, N	25 ⁱ	67.77	68.14	6.26	6.74		
III	MDI	2,2-Diethyl-1,3-propanediol	Light, fluffy ppt. Strong, pliable films	S 150 Dec. 235	A, C, D, T	33 ⁱ	69.07	69.21	6.85	7.01	7.33	7.49
IV	MDI	2-n-Butyl-2-ethyl-1,3-propanediol	Powder. Weak films and fibers	S ^j 110-120, 160 Dec. 240	A, C, D, T	49 ^k 28 ^l	70.21	70.13 ⁱ	7.36	7.69 ⁱ	6.83	6.74 ^k 6.83 ^l
V	MDI	2-Ethyl-1,3-hexanediol	Powder. Films, not fibers	S 140 Dec. 210	A, C, D, T		69.66	69.18	7.12	7.92	7.07	6.99
VI	HDI ^m	Bisphenol A	Powder. Weak fibers and films	S 130 Dec. 230	A, C, D	25 ⁱ	69.66	69.37	7.12	7.37	7.07	6.82
VII	HDI	Resorcinol	Powder. Poor films, no fibers	S 150 Dec. 220	A, C, D	20 ⁱ	60.42	60.73	6.52	6.76	10.07	10.07
VIII	HDI	2-n-Butyl-2-ethyl-1,3-propanediol	Powder. Soft, pliable films	S 60 Dec. 250	A, C, D	82 ^k 42 ^l	62.16	61.78	9.82	10.22	8.53	8.52

^a After reprecipitation, except for I and II, which were powders as isolated. ^b A, acetone; C, *m*-cresol; D, dimethylformamide; N, nitrobenzene; T, tetrahydrofuran. ^c Limiting viscosity numbers in *m*-cresol except for II, done in dimethylformamide. ^d Analyses for carbon and hydrogen by the microanalytical laboratories of Dr. Carl Tiedcke and Dr. A. Elek. ^e Semi-micro Dumas. ^f Methylene-bis-(4-phenyl isocyanate). ^g 2,2-Bis-(*p*-hydroxyphenyl)propane. ^h S = softens, dec. = darkens and decomposes. ⁱ Unfractionated material. ^j Slight softening at the lower range, viscous flow at the second temperature. ^k First fraction. ^l Fifth fraction. ^m 1,6-Hexamethylene diisocyanate. ⁿ Slightly soluble in acetone.

elastic properties. An increase in the aliphatic character of the molecule or an increase in the irregularity of the chains generally produced polymers having lower softening temperatures, greater pliability of films and greater solubility. These effects are in accord with previous observations on polyurethans^{8,9} and other polymers.¹⁰

The resistance of the polyurethans to alkali was studied by immersion of weighed samples in 1% sodium hydroxide at 50° for varying periods. Data on the weight and limiting viscosity of the recovered products are given in Table II. It is apparent that the polyurethans prepared from aliphatic diols were unaffected (regardless of the nature of the isocyanate component) but that the polyurethans prepared from dihydric phenols were more or less decomposed under these conditions. Further evidence of the resistance to alkali of the polyurethans from aliphatic diols was obtained from the infrared

absorption spectra of films of polymers IV and VIII. There was no detectable change in the spectra after treatment with 1% sodium hydroxide at 50° for 6 days. The difference in reactivity between the two classes of polyurethans is parallel to the known difference¹¹ in the rates of alkaline hydrolysis of alkyl and phenolic esters of simple carboxylic acids.

The solid product which remained after hydrolysis of the phenolic polyurethan VII was found to be about 50% gel and the portion soluble in *m*-cresol possessed a much higher intrinsic viscosity than the original material. The infrared absorption spectrum of the residual solid was practically identical with that of known analyzed polyhexamethyleneurea.¹² The amide band at 5.9 μ and the N-substituted amide band at 6.5 μ of the polyurethan had entirely disappeared, and new bands, characteristic of the polyurea, were present at 6.2 and 6.4 μ . Additional properties which indicated that the material was largely polyhexamethyleneurea were its low solubility and its high softening temperature, (245-250°).

In addition to the polyhexamethyleneurea, resorcinol, hexamethylenediamine and carbon dioxide (present as sodium carbonate) were shown to be products of the hydrolysis. Resorcinol was separated and identified as the dibenzoate and the diamine was isolated and identified as the dibenzamide.

The hydrolysis of the phenolic polyurethans VI and VII was studied quantitatively; the results are summarized in Table III. In both cases the amount of carbon dioxide liberated was of the same magnitude and the solid material remaining was polyhexamethyleneurea. The fact that Bisphenol-A was separated in theoretical amounts from the hydrolysis of VI while resorcinol could not be simi-

 TABLE II
 STABILITY OF POLYURETHANS TOWARD 1% SODIUM HYDROXIDE AT 50°

Poly-mer no.	Polyurethan from	Me- dium	Time, hr.	Wt. re- cov., %	$[\eta]$ ^a creov. Original	ma- terial
III	MDI and 2,2-diethyl-1,3-propanediol	Base	24	98.6	33	34
		Base	64	98.8		32
		Base	121	98.7		..
		H ₂ O	121	98.7		32
VIII	HDI and 2-n-butyl-2-ethyl-1,3-propanediol	Base	144	99.8	82	85
		Base	552	92.8		
		Base	552	93.4		
		H ₂ O	552	98.6		
I	MDI and bisphenol A ^g	Base	122	38.1	20	106 ^c
		Base	122	26.1		..
		Base	122	26.1		..
		H ₂ O	122	100.0		..

^a Limiting viscosity number in *m*-cresol at 25°. ^b Carried out at room temperature. ^c Soluble portion.

(9) T. Lieser and G. Nischk, *Ann.*, **569**, 59 (1950).

(10) R. Hill and E. E. Walker, *J. Polymer Sci.*, **3**, 609 (1948).

(11) L. P. Hammett, "Physical Organic Chemistry," McGraw-Hill Book Co., New York, N. Y., 1940, p. 211.

(12) Supplied through the courtesy of Dr. M. R. Lytton and Mr. E. A. Wielicki, American Viscose Corporation.

TABLE III

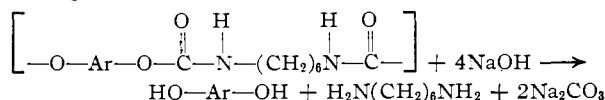
Polyurethan from	QUANTITATIVE DATA ON PRODUCTS OF HYDROLYSIS ^a OF PHENOLIC POLYURETHANS						
	Wt. used, g. ^b	Wt. CO ₂ formed	Phenol formed G.	%	Amine formed, g.	Polyurea found, g.	Material balance wt. % ^c N, % ^d
HDI and resorcinol	2.000	0.405	0.159	0.811	... 89.6
HDI and bisphenol	2.851	.390	1.617	98.7	0	1.101	104 95.5
HDI and bisphenol	2.851	.382	1.621	98.9	0	1.099	104 ..

^a Tests were carried out at 90° in 1% NaOH for 6 days. ^b In all cases 0.0072 mole. ^c The weight per cent. could not be computed for the polyurethan of HDI and resorcinol because of the lack of reliable data on recovered resorcinol. ^d The N per cent. was computed by comparing the nitrogen content of the products (based on actual analysis of the polyurea) with the nitrogen content of the polyurethan.

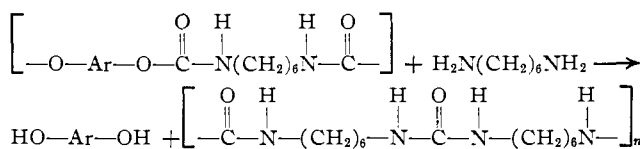
larly isolated in the case of VII was probably due to the instability of resorcinol to oxidation.

There was no evidence of free hexamethylenediamine in the filtrates from either of two duplicate hydrolyses of VI. This is in accord with the fact that almost the theoretical amount of nitrogen present in the original polymer was present in the final solid, polyhexamethyleneurea.

These results may be explained by assuming that free hexamethylenediamine is formed by the action of sodium hydroxide solution on the urethan linkage



The free diamine is then capable of reacting with urethan linkages by a nucleophilic displacement at the carbonyl carbon atom with subsequent formation of the polyhexamethyleneurea and dihydroxy compound.



This reaction would be similar to that reported¹³ for the production of polyureas from diurethans and primary or secondary diamines.

The validity of the proposed aminolysis of the polyurethan linkage by free diamine in aqueous solution was substantiated by the isolation of Bisphenol A and polyhexamethyleneurea from the reaction of an aqueous solution of hexamethylenediamine with polymer VI.

Acknowledgment.—The authors are indebted to the Armstrong Cork Company for a fellowship in support of this investigation, to Drs. E. J. Pieper, L. H. Dunlap and F. W. Breuer for helpful discussions, and to Dr. H. C. Beachell and Miss M. J. Lucas for infrared absorption measurements and interpretations.

Experimental

Materials.—The diisocyanates were purified by distillation, and their purity determined by the piperidine method¹⁴: methylene-bis-(4-phenyl isocyanate),¹⁵ b.p. 184° at 3 mm., purity 99.4%; 1,6-hexamethylene diisocyanate (Bios Laboratories), b.p. 99–101° at 1 mm., purity 99.1%.

The 2,2-dimethyl-1,3-propanediol (Matheson) melted at

(13) E. Martin, U. S. Patent 2,181,663 (Nov. 28, 1939).

(14) E. Dyer, H. A. Taylor, S. J. Mason and J. Samson, *THIS JOURNAL*, **71**, 4107 (1949).

(15) We are indebted to the Monsanto Chemical Company for samples of this material.

127° after recrystallization from benzene. The other three diols¹⁶ were purified by distillation: 2,2-diethyl-1,3-propanediol, b.p. 115° at 6 mm., f.p. 62°; 2-ethyl-1,3-hexanediol, b.p. 95.5° at 2 mm., *n*_D²⁰ 1.4510; 2-butyl-2-ethyl-1,3-propanediol, b.p. 122° at 10 mm., f.p. 41°. Resorcinol, recrystallized from benzene, melted at 110°, and 2,2-bis-(*p*-hydroxyphenyl)-propane (Bisphenol A)¹⁷ melted at 151–152° after recrystallization from dilute acetic acid.

Chlorobenzene and *o*-dichlorobenzene were anhydrous distilled materials, b.p. 132° and 181–182°, respectively. The dimethylformamide¹⁸ boiled at 153° after distillation. The redistilled *m*-cresol had *n*_D²⁰ 1.5393. Acetone¹⁹ and methanol²⁰ were specially purified. Tetrahydrofuran was freed from peroxides.²¹

Preparation of Polyurethans. A. Solution Polymerization.

—A procedure similar to that described by Marvel and Johnson⁷ was used, with the addition of three drops of triethylamine for the reaction of 0.03 mole of dihydroxy compound and 0.03 mole of diisocyanate. The amount of solvent needed was 200 to 300 ml. for the dihydroxy compound and 100 ml. for the diisocyanate. For preparation of polymer I the solvent was benzene, for preparation of polymers II and III it was an 80:20 chlorobenzene-*o*-dichlorobenzene mixture. Purification of polymer I was done by solution in dimethylformamide and precipitation in methanol, of polymer III by solution in tetrahydrofuran and precipitation with water, of polymer II by partial evaporation of a nitrobenzene solution.

Polymer III was prepared equally well by bulk polymerization. When it was learned that polymer IV could not be obtained in pure condition by solution polymerization because of the interference of the side-reaction giving isocyanate homopolymer, all further reactions were done by the bulk method.

B. Bulk Polymerization.—The procedure below for preparing polymer VIII is typical of that used for polymers IV–VIII. The reaction vessel was a 30-ml. lipless beaker with a stopper carrying a thermometer, stirrer, dropping funnel and gas inlet and outlet tubes. The diol (0.03 mole + 0.5% excess) was melted in the reaction vessel in an atmosphere of dry, oxygen-free nitrogen. The diisocyanate (0.03 mole) was added slowly during stirring. As the reaction progressed and the melt became more viscous, the temperature was slowly raised to 180°. When the mixture became too viscous to stir, the stirrer was removed and heating continued for a short time. The mixture was cooled, dissolved in pure acetone, and treated with a small amount of anhydrous methanol to ensure complete reaction of the isocyanate groups. After standing overnight the polymer was precipitated by adding the solution dropwise to violently stirred water.

Polymers IV and V were precipitated from acetone into methanol, VI and VII from dimethylformamide into water.

Viscosity Determinations.—To find the limiting viscosity numbers²² a modified Ubbelohde viscosimeter²³ was used. Readings were taken at five concentrations, varying from

(16) Kindly furnished by the Carbide and Carbon Chemicals Company.

(17) Kindly supplied by the Dow Chemical Company.

(18) Kindly supplied by E. I. du Pont de Nemours and Company.

(19) J. B. Conant and W. R. Kirner, *THIS JOURNAL*, **46**, 232 (1924).

(20) L. F. Fieser, "Experiments in Organic Chemistry," 2nd Ed., D. C. Heath and Co., Boston, 1941, p. 360.

(21) Electrochemicals Department, E. I. du Pont de Nemours and Co., New Products Bulletin No. 4, Tetrahydrofuran, 1949, p. 8.

(22) Report on Nomenclature of the International Union of Pure and Applied Chemistry, *J. Polymer Sci.*, **8**, 270 (1952).

(23) W. E. Davis and J. H. Elliott, *J. Colloid Sci.*, **4**, 313 (1949).

0.004 to 0.0008 g. per ml. of *m*-cresol or dimethylformamide.

In some cases it was necessary to fractionate the polymers in order to obtain straight line viscosity relationships. Polyurethans IV and V were fractionally precipitated using an acetone-methanol system. Polyurethan VIII was similarly handled with an acetone-*n*-hexane mixture. Nitrogen analysis of the first and fifth fractions of IV indicated that the fractions were the same in chemical composition. Viscosity determinations of the first and fifth fractions of IV and VIII indicated that the fractionations had been effective in narrowing the molecular weight distribution of the material.

Infrared Analysis.—The infrared absorption spectra were taken on a Baird Associates, Inc., Model B infrared recording spectrophotometer. When it was not possible to form films of the material, as was the case with polyhexamethyleneurea, Nujol mulls of finely divided polymer were used.

Homopolymer²⁴ of Methylene-bis-(4-phenyl Isocyanate).—When solutions of this diisocyanate in anhydrous and peroxide-free ether, dioxane, tetrahydrofuran or chlorobenzene were treated with a few drops of triethylamide, a white precipitate appeared in a few minutes at room temperature. The copper chelate of acetylacetone also catalyzed the formation of this substance. In tetrahydrofuran with no catalyst the product started to appear after about 30 hours. The substance was not soluble in common solvents except dimethylformamide. For analysis a sample was prepared in ether and washed thoroughly with benzene to remove monomer. The polymer softened and slowly discolored above 205–210°, with no sharp decomposition point.

Anal. Calcd. for $(C_{15}H_{10}O_2N_2)_x$: N, 11.20. Found: N (Dumas), 11.16. No attempt was made to determine molecular weight.

Hydrolysis of the Phenolic Polyurethans VI and VII.—To 0.0027 mole of the respective polyurethan contained in a 500-ml., three-neck, round-bottom flask equipped with a thermometer and reflux condenser and protected from atmospheric carbon dioxide with an ascarite tube were added exactly 200 ml. of 0.2192 *N* sodium hydroxide solution and a small amount of Duponol ME as wetting agent. After standing six days at 90° the mixture was cooled and the solution separated from the solid material by vacuum filtration through a weighed sintered glass funnel. The solid material was washed thoroughly with water and dried to constant weight. The substance softened at 245–250° and became fluid at 260–270°. It was partly soluble in *m*-cresol. The

same substance was obtained by hydrolysis of both VI and VII.

Anal. Calcd. for $(C_7H_{14}N_2O)_x$: C, 59.13; H, 9.92; N, 19.70. Found: C, 59.90; H, 9.86; N, 18.17.

Although the analytical results indicate that this substance was not homogeneous, the infrared absorption spectra were practically identical with that of an authentic specimen¹² of polyhexamethyleneurea. Hydrolysis product of VI or VII: $\lambda_{\max}^{\text{mineral oil}}$ 2.34(w), 3.01(s), 6.17(s), 6.38(m), 7.99(m), 8.25(w), 9.25(w), 13.83(m). Known polyhexamethyleneurea: $\lambda_{\max}^{\text{mineral oil}}$ 2.33(w), 3.05(s), 6.19(s), 6.43(m), 8.02(m), 8.30(w), 9.25(w), 13.83(m). Polyurethan VI: $\lambda_{\max}^{\text{mineral oil}}$ 2.35(w), 3.01(w), 4.32(m), 5.89(s), 8.27(s), 8.52(m), 9.84(w), 13.84(s).

Aliquot portions (20 ml.) of the hydrolysis filtrate after removal of the polyurea were titrated with standard 0.1 *N* hydrochloric acid using a Beckman model M pH meter with glass electrode.

The equivalence points were found from the differential plot of $\Delta pH/\Delta v$ against *v* of acid. Control experiments with known mixtures of the diamine, sodium carbonate and sodium hydroxide showed that the amount of carbonate and diamine could be determined by a single titration with an accuracy of 1%. The amount of carbonate was also found by liberation of carbon dioxide and absorption in a weighed ascarite tube.

The amount of free dihydroxy compound present was found by continuous extraction of an acidified aliquot sample and evaporation of the ether extract. In the case of resorcinol this method gave an unreasonably high value, while an attempted iodimetric titration gave low values.

Aminolysis of VI.—To a 0.500-g. sample of VI in a 500-ml., three-neck, round-bottom flask equipped with a thermometer and reflux condenser was added 100 ml. of an aqueous solution of 1,6-hexamethylenediamine (0.293 g. diamine/100 ml. of water). After heating at 90° for six days the solid material (0.460 g.) was separated from the solution by filtration through a weighed sintered glass funnel. Bisphenol A (0.0381 g.) was isolated from a 20-ml. aliquot of the filtrate by continuous ether extraction.

The polymeric substance softened at 245–250°, and was soluble only in *m*-cresol, whereas the original polyurethan melted at 130° and was soluble in acetone. The infrared absorption spectrum showed the prominent urea bands at 6.2 and 6.4 μ , characteristic of polyhexamethyleneurea and had only a trace of the bands given by the original polyurethan.

NEWARK, DELAWARE

(24) A review of work on isocyanate homopolymers is given by J. H. Saunders and R. J. Slocombe, *Chem. Revs.*, **43**, 203 (1948).

[CONTRIBUTION FROM THE RESEARCH DEPARTMENT, CIBA PHARMACEUTICAL PRODUCTS, INC.]

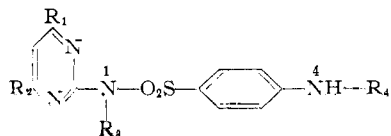
The Acetylation of Some Sulfapyrimidines

By J. B. ZIEGLER AND A. C. SHABICA

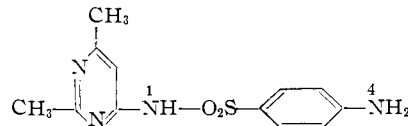
RECEIVED JULY 21, 1953

It has been shown that acetylation with excess acetic anhydride in pyridine affords monoacetyl derivatives of sulfadiazine, sulfamerazine and Elkosan, but gives a diacetyl derivative of sulfamethazine. However, acetylation of sulfamethazine with excess acetic anhydride in glacial acetic acid yields a monoacetyl derivative. A mechanism is suggested to account for these facts.

During the course of a study of the solubility of a number of substituted sulfonamides, to be reported elsewhere, it was necessary to synthesize the N⁴-monoacetyl derivatives of sulfadiazine (Ia), sulfamerazine (Ib), sulfamethazine (Ic) and Elkosin



Ia, $R_1 = R_2 = R_3 = R_4 = H$;
Ib, $R_1 = CH_3, R_2 = R_3 = R_4 = H$;
Ic, $R_1 = R_2 = CH_3, R_3 = R_4 = H$



II

Id, $R_1 = R_2 = CH_3, R_3 = H, R_4 = CO-CH_3$;
Ie, $R_1 = R_2 = CH_3, R_3 = R_4 = CO-CH_3$

(II). A general procedure was devised which involved heating the free sulfonamide with an excess of acetic anhydride in pyridine solution. This method yielded the desired N⁴-monoacetyl derivatives with all sulfapyrimidines with the