tillation of the oil which had separated. The oily layer from the steam distillate was identical in infrared absorption characteristics with the tertiary amine XII, R = CH_3.

Nitrosation of the Lactam V with Nitrogen Tetroxide.—Five grams of lactam dissolved in 25 ml. of glacial acetic acid was chilled to 0° and saturated with dry nitrogen tetroxide. After standing one hour at room temperature the solution was poured on 100 g. of ice. A yellow solution resulted which turned red-brown when made basic with 50% sodium hydroxide. The basic solution was extracted with ether and the aqueous portion was set aside. In the best run, evaporation of the ether extract left about 0.25 g. of white, crystalline solid melting at 162–164° after recrystallization from ethyl acetate. The solid had $[\alpha]^{26}D$ +288 ± 5° (*l* 0.5, *c* 4, 95% ethanol), and is assigned structure XIV; spectrum, λ_{max}^{mull} 3.12, 3.22, 5.94, 6.21 μ.

Anal. Calcd. for $C_{10}H_{16}N_2O_2$: C, 61.20; H, 8.22. Found: C, 61.33; H, 8.33.

The aqueous portion from the extraction was acidified with concentrated sulfuric and extracted with ether. The extract was evaporated on a steam-bath to remove ether, then under reduced pressure to remove acetic acid. A black tar remained that slowly crystallized to yield 0.75 g. of white needles melting at 155–156° after recrystallization from ethanol–water; $[\alpha]^{26}D$ −104 ± 5° (*l* 0.5, *c* 4, 95% ethanol). This compound is assigned structure XV; spectra: λ_{max}^{alc} 261 mμ (ε 9200), 280 mμ (ε 8150); λ_{max}^{mull} 3.12, 4.52, 5.92, 6.12 μ.

Anal. Calcd. for $C_{10}H_{14}N_2O$: C, 67.38; H, 7.92; N, 15.72. Found: C, 67.71; H, 7.88; N, 15.55.

When the nitrile XV (0.20 g.) was warmed for 15 minutes with 10 ml. at 10% sodium hydroxide a colorless solution was obtained. Acidification of the hydrolysis mixture caused an oil to separate and when this oil was dissolved in dilute sodium bicarbonate solution, carbon dioxide and hydrogen cyanide, detected by the formation of prussian blue, were evolved.

STANFORD, CALIF.

[CONTRIBUTION FROM THE RESEARCH CENTER, HERCULES POWDER CO.]

Synthesis of Poly-β-alanine from Acrylamide. A Novel Synthesis of β-Alanine[1]

BY DAVID S. BRESLOW, GEORGE E. HULSE AND ALBERT S. MATLACK

RECEIVED MARCH 15, 1957

Poly-β-alanine has been prepared by the base-catalyzed polymerization of acrylamide. By fractionation samples with weight-average molecular weights as high as about 80,000 have been prepared. Acid hydrolysis of the polymer constitutes a simple preparation of β-alanine in yields of 90% starting with acrylamide. Substituted acrylamides and analogous sulfonamides yield polymers of much lower molecular weight. An unusual mechanism for the polymerization is proposed.

Poly-β-alanine has been prepared previously by the elimination of HX from a β-alanine derivative, $H_2NCH_2CH_2COX$, where X is NH_2,[2] OC_2H_5,[3] Cl[4] or OH,[5] or by the elimination of thiophenol and carbon dioxide from N-carbothiophenyl-β-alanine.[6] It is questionable whether any of these methods give high molecular weight polymers.[7] Poly-β-alanine has now been prepared by the base-catalyzed polymerization of acrylamide, a readily available compound.

Preparation of Polymers.[9]—Poly-β-alanine formed a mixture of water-soluble and water-insoluble polymers when acrylamide, usually in an inert diluent, was contacted with a strong base in the presence of an inhibitor for radical-induced polymerization. The technique of polymerization was similar in most cases, typical examples being given in the Experimental section.

The effects of certain variables on the polymerization are shown in Table I.

(1) Presented at the 131st Meeting of the American Chemical Society, Miami, Florida, April, 1957.

(2) A. P. N. Franchimont and H. Friedman, *Rec. trav. chim.*, **25**, 80 (1906).

(3) E. Abderhalden and F. Reich, *Z. physiol. Chem.*, **178**, 169 (1928).

(4) M. Frankel, Y. Liwschitz and A. Zilkha, *Experientia*, **9**, 179 (1953); THIS JOURNAL, **76**, 2814 (1954).

(5) J. S. Chirtel and A. M. Mark, U. S. Patent 2,691,643 (1954).

(6) J. Noguchi and T. Hayakawa, THIS JOURNAL, **76**, 2846 (1954).

(7) Noguchi and Hayakawa reported a molecular weight of 43,500 for their sample of poly-β-alanine. However, their intrinsic viscosity of 0.063 in formic acid is remarkably low for this molecular weight. Recently Weymouth[8] has shown that several polypeptides made by this procedure have relatively low molecular weights, of the order of 1000–5000.

(8) F. J. Weymouth, *Chem. & Ind.*, Brit. Inds. Fair Rev., April, 1956, R34.

(9) D. S. Breslow, British Patent 736,461 (1955); U. S. Patent 2,749,331 (1956).

The highest molecular weight water-insoluble polymer was obtained in pyridine. The largest amount of water-insoluble polymer was formed in the absence of solvent. Phenyl-β-naphthylamine was superior to hydroquinone as a radical inhibitor, presumably because hydroquinone acted as a chain terminator for basic polymerization. It was possible to carry out small-scale preparations without an inhibitor, but in larger runs the presence of an inhibitor was advantageous. Sodium *t*-butoxide in *t*-butyl alcohol appeared to be the best catalyst; apparently the alcohol did not terminate polymer chains. Dilution lowered the yield and molecular weight of water-insoluble polymer somewhat.

Properties of Poly-β-alanine.—Each polymerization yielded a spectrum of products with fractions soluble in pyridine, fractions soluble in water and fractions soluble only in solvents such as formic acid being obtained. The pyridine-soluble material consisted largely of monomer and dimer, the latter analyzing reasonably well for $CH_2{=}CH{-}CONHCH_2CH_2CONH_2$. This polymerized much like acrylamide itself on addition of base.

Both the water-soluble and the water-insoluble polymers were highly crystalline, melting at about 325° and about 340°, respectively, with considerable decomposition. Both gave the same well-defined X-ray diffraction pattern, the water-insoluble polymer being somewhat more crystalline than the water-soluble. The lattice spacings of 4.6, 3.9 and 3.55 Å. had relative intensities of 60, 10 and 100, respectively, for the water-insoluble polymer.

The water-insoluble polymer showed typical polyamide solubility. Thus, it was soluble in for-

TABLE I

THE BASE-CATALYZED POLYMERIZATION OF ACRYLAMIDE[a]

Solvent	Catalyst	Inhibitor[b]	Temp., °C.	Yield, % Sol.	Insol.	Specific viscosity[c] Sol.	Insol.
t-Butanol	*t*-C_4H_9ONa[d]	HQ	83	39	8	0.307(P)	
						.202(W)	
Acetonitrile	CH_3ONa[e]	HQ	82	67	19	.150(A)	0.193(A)
Benzene	CH_3ONa[f]	HQ	80	48		.171(W)	
Dioxane	*t*-C_4H_9ONa[d]	HQ	100	28	13	.222(P)	0.293(A)
						.210(A)	
Quinoline	*t*-C_4H_9ONa[d]	HQ	200		55		0.234(A)
							.151(F)
Pyridine	*t*-C_4H_9ONa[d]	HQ	100	28	39	.196(P)	.316(P)
Pyridine	*t*-C_4H_9ONa[d]	DBHQ	100	70	0		
Pyridine	*t*-C_4H_9ONa[d]	PBNA	100	36	48		1.03(A)
							0.658(F)
Pyridine	*t*-C_4H_9ONa[d]	Q	100	69	17		0.91(A)
Pyridine	*t*-C_4H_9ONa[d]	None	100	48	29		1.03(A)
Dioxane	Na[f]	PBNA	100	30	4		0.88(A)
Pyridine	$NaNH_2$[g]	PBNA	100	44	12		.63(F)
None	*t*-C_4H_9ONa[h]	PBNA	[k]		65		.242(F)
Pyridine[i]	*t*-C_4H_9ONa[d]	PBNA	100		47		.636(F)
Pyridine[j]	*t*-C_4H_9ONa[d]	PBNA	100		21		.567(F)

[a] Runs were made with 10 g. of acrylamide in 100 ml. of solvent for 18 hr. [b] 0.1% hydroquinone (HQ), 0.2% phenyl-β-naphthylamine (PBNA), 0.2% di-*t*-butylhydroquinone (DBHQ) and 0.2% quinone (Q) were used. [c] Determined as 1% solutions in phenol at 100° (P) or at 25° in 90% formic acid (F), anhydrous formic acid (A) or water (W). [d] 0.46 g. [e] 0.5 g. [f] 0.1 g. [g] 0.2 g. [h] 0.1 g. of sodium in 10 ml. of *t*-butyl alcohol, then solvent removed *in vacuo*. [i] 40 ml. [j] 300 ml. [k] Uncontrolled reaction.

mic acid, dichloroacetic acid, phenol containing 11% water and concentrated aqueous salt solutions. It was also soluble in phenol at 100°, in boiling ethylene glycol and in boiling glycerol. Viscosities were higher in anhydrous formic acid than in 90% formic acid; this behavior has been observed with other polyamides.[10]

Poly-β-alanine has been prepared with weight-average molecular weights of about 80,000 (by light scattering in 90% formic acid). The molecular weight of the water-insoluble polymer could be raised by solution and reprecipitation. Thus, a sample of molecular weight 58,000, corresponding to a 1% specific viscosity of 0.500 and an intrinsic viscosity of 0.416 (both in 90% formic acid), after solution in formic acid and precipitation by water, had a molecular weight of 76,000, corresponding to a specific viscosity of 0.639.

Structure of Poly-β-alanine and the Mechanism of Polymerization.—Acrylamide might polymerize with base in a manner analogous to that of methacrylonitrile[11]; the product would then be polyacrylamide. That the polymer was essentially linear poly-β-alanine was proved by hydrolysis of the polymer with 50% sulfuric acid. This constitutes a convenient synthesis of β-alanine from acrylamide, a readily available starting material, in essentially one step, since it is only necessary to heat acrylamide with a base and hydrolyze the reaction mixture. Yields of about 90% based on acrylamide can be obtained.[12]

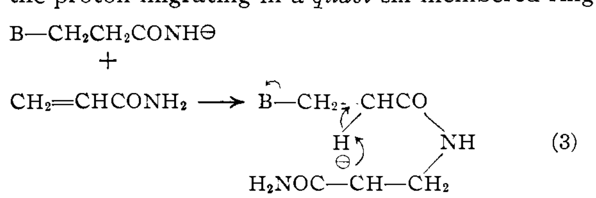
The presence of an olefinic double bond as an end group in poly-β-alanine was shown by both hydrogenation and polarographic reduction. However, in the absence of data on the number-average molecular weight of these polymers, the presence of some alkoxy end groups cannot be ruled out entirely. The presence of a $-CONH_2$ end group was shown by carrying out a hydrolysis with hydrochloric acid and determining the ammonium chloride formed.

Two alternative modes of initiation must be considered for the base-catalyzed polymerization of acrylamide.

$$CH_2{=}CHCONH_2 + B^- \rightleftharpoons CH_2{=}CHCONH^{\ominus} + BH \quad (1)$$

$$CH_2{=}CHCONH_2 + B^- \rightleftharpoons B{-}CH_2\overset{\ominus}{C}HCONH_2 \rightleftharpoons B{-}CH_2CH_2CONH^{\ominus} \quad (2)$$

Equation 1 is preferred as the major source of initiation for several reasons. One is the olefinic end group found in the polymer. More important, however, is the isolation of the unsaturated dimer from the reaction mixture. If initiation were by equation 2, $B\text{-}CH_2CH_2CONH_2$ should be present and none was found. It must be admitted, however, that the unsaturated dimer could be formed from equation 2 by a scheme such as the following, the proton migrating in a *quasi* six-membered ring.



(3)

Two possible methods of growth must be considered, too. One would involve a continuation of the process depicted in the first part of reaction sequence 3, the anion transferring its charge from carbon to nitrogen as shown in equation 2, and

(10) J. R. Schaefgen and C. F. Trivisonno, THIS JOURNAL, **73**, 4580 (1951).

(11) R. G. Beaman, *ibid.*, **70**, 3115 (1948).

(12) A. S. Matlack, U. S. Patent 2,672,480 (1954).

TABLE II

BASE-CATALYZED POLYMERIZATION OF OTHER MONOMERS[a]

Monomer	Solvent	Inhibitor[b]	Yield, %[c]	% N in end group	Specific viscosity[d]	Physical properties, m.p., °C.
Methacrylamide[e]	*t*-Butanol	HQ	9	0.28		
Methacrylamide[e]	Dioxane	HQ	36	0.81	0.100(A)	257 d.
Methacrylamide[f]	None	PBNA	39		.092(A)	
Crotonamide[g]	Pyridine	HQ	69	1.32	.072(F)	
Crotonamide[f]	None	PBNA	55		.066(F)	>300 d.
3,4,5,6-Tetrahydrobenzamide[h]	None	PBNA	100	4.28		Brown resin
Methylenebisacrylamide[i]	Pyridine	PBNA	100			Insol., infus.
Ethylenesulfonamide[f]	None	PBNA	85		.079(D)	Soft. 100–160
2-Propene-1-sulfonamide[j]	None	PBNA	39		.060(D)	Soft. 130
Ethylenesulfonanilide[k]	None	PBNA	57		.022(D)[k]	270 d.
p-Acrylamidobenzenesulfonamide[l]	Pyridine	PBNA	44		.038(D, 100°)	255–300
Acrolein oxime[m]	Dioxane	PBNA	25		.06(F)	>300 d.

[a] Polymerization of 10 g. of monomer in 100 ml. of solvent, 0.1 g. of sodium as catalyst, overnight at 100°, except as noted. [b] 0.01 g. of hydroquinone (HQ) or 0.02 g. of phenyl-β-naphthylamine (PBNA). [c] Of water insoluble-polymer. [d] Of 1% solutions in anhydrous formic acid (A), 90% formic acid (F) or dimethylformamide (D) at 25°. [e] Five days at 100°. [f] Exothermic reaction allowed to subside. [g] Two days at 100°. [h] Mixture heated gradually to 220°, then cooled; it was not fractionated. [i] Sodium methoxide catalyst (5%). [j] Heated 6 hr., purified by ether extraction. [k] Sodium catalyst (5%), polymer extracted with hot ethanol, viscosity of 0.5% solution at 150°. [l] 25% solution. [m] Polymer extracted with hot acetone.

growth continuing on the amide part of the chain. This could be initiated by the anion shown in equation 1 in exactly the same manner. Chain termination would involve reaction of the growing chain with BH, regenerating the base B^-, or with any other active hydrogen compound present. This mechanism differs from the usually accepted mechanism for the base-catalyzed polymerization of acrylates[11] only in the necessity for a proton migrating from nitrogen to carbon for each addition. A second possibility exists, however, for the initiating species shown in equation 1. This involves essentially a chain transfer reaction in each growth step, as shown in reaction sequence 4.

$$CH_2{=}CHCONH^{\ominus} + CH_2{=}CHCONH_2 \longrightarrow CH_2{=}CHCONHCH_2\overset{\ominus}{C}HCONH_2 \quad (4)$$

$$CH_2{=}CHCONHCH_2\overset{\ominus}{C}HCONH_2 + CH_2{=}CHCONH_2 \longrightarrow CH_2{=}CHCONHCH_2CH_2CONH_2$$

$$+ CH_2{=}CHCONH^{\ominus} \longrightarrow CH_2{=}CHCONHCH_2\overset{\ominus}{C}HCO(NHCH_2CH_2CO)_nNH_2$$

Although the sequence has been depicted with the growing anion chain abstracting a proton from acrylamide, it could abstract a proton from any acidic species present, such as the unsubstituted amide end group of an already formed polymer chain[13]; the chain could grow, therefore, from either end. This unusual polymerization is in essence an addition polymerization which should have all the attributes of a condensation polymerization[14]; the polymerization should be relatively slow and the molecular weight should increase with conversion. Our preference for this mode of polymerization is at present based on only one observation. It would appear to us highly unlikely that, in any usual type of polymerization, one could isolate both a dimer and a high molecular weight polymer from the same reaction mixture. If chain transfer were so prevalent as to form a dimer, a high molecular weight polymer should not be found. Inasmuch as both were isolated, the molecular weight distribution must be unusually broad, and it seems that at least the major amount of polymer must be formed by this chain-transfer route.

Other Polymers.—The polymerization of several other monomers containing both an active hydrogen and an activated double bond is summarized in Table II. Any substituent in the acrylamide molecule decreased both the rate and degree of polymerization, a substituent on the nitrogen showing the greatest effect, on the β-carbon next and on the α-carbon least. The sulfonamides appeared to give lower molecular weight polymers than their carbon analogs. A factor influencing the molecular weight of all these polymers may be the inherent reversibility of addition reactions of α,β-unsaturated systems.

Experimental[15]

Materials.—Acrylamide (American Cyanamid Co.) was sublimed *in vacuo* before use. Methacrylamide was obtained from Rohm and Haas Co. The following compounds were prepared by described procedures: crotonamide,[16] 3,4,5,6-tetrahydrobenzamide,[17] 2-propene-1-sulfonamide,[18] ethylenesulfonanilide[19] and acrolein oxime.[20] The synthesis of ethylenesulfonamide will be reported elsewhere.

To prepare *p*-acrylamidobenzenesulfonamide a solution of 5.0 g. of sulfanilamide hydrochloride (Matheson Co.) in 70 ml. of water at 50° was treated with 4.0 g. of acrylic anhydride[21] and a solution of 2.05 g. of sodium acetate in 12 ml. of water. The mixture was stirred for 20 minutes at 65°, cooled to 0° and the product collected by filtration; the yield was quantitative. After two recrystallizations from aqueous ethanol (containing a trace of phenyl-β-naphthyl-

(13) Since alkyl groups are electron-donating, the hydrogen on a terminal amide group should be more acidic than one on a substituted amide group in the chain. If this were not so, a highly branched polymer would result. N-Substituted acrylamides have been found to polymerize much less readily under these conditions.

(14) Flory would actually classify this as a condensation polymerization (P. J. Flory, "Principles of Polymer Chemistry," Cornell University Press, Ithaca, N. Y., 1953, pp. 37–40).

(15) Analyses were carried out by the Analytical and Physical Chemistry Divisions of the Research Center.

(16) R. Stoermer and H. S. Stockmann, *Ber.*, **47**, 1786 (1914).

(17) A. Einhorn, *ibid.*, **26**, 451 (1893).

(18) A. Lambert and J. D. Rose, *J. Chem. Soc.*, 46 (1949).

(19) A. A. Goldberg, *ibid.*, 465 (1945).

(20) K. H. W. Tuerck and H. J. Lichtenstein, U. S. Patent 2,417,024 (1947).

(21) W. Reppe, *et al.*, *Ann.*, **582**, 1 (1953).

amine) at 60°, colorless needles melting at 215° were obtained.

Anal. Calcd. for $C_9H_{10}SO_3N_2$: C, 47.77; H, 4.46. Found: C, 47.70; H, 4.55.

Polymerization of Acrylamide in Pyridine.—Acrylamide (10 g.) was added with stirring at 100° to a solution of 0.02 g. of phenyl-β-naphthylamine in 100 ml. of pyridine (dried over barium oxide). When the acrylamide had dissolved, a solution of 0.1 g. of sodium in 10 ml. of *t*-butyl alcohol was added. Polymer began to form on the walls in 4 minutes. After heating for 16 hr., the polymer was removed by filtration, extracted with boiling water for an hr. and dried *in vacuo* at 80° (4.8 g.). After neutralization with acetic acid, the aqueous extract was evaporated to dryness. The residue (3.6 g.) was purified by pouring an aqueous solution into methanol; 2.6 g. of polymer was isolated.

After the addition of phenyl-β-naphthylamine (0.02 g.) the mother liquor was evaporated to dryness *in vacuo*. On sublimation of the residue at 0.1 mm., acrylamide (0.4 g., m.p. 81–83°) was obtained, leaving behind 1.2 g. of solid.

Anal. Calcd. for $C_6H_{10}O_2N_2$: H_2 absorption, 1.42; NH_3, 12.0. Found: H_2 absorption, 1.18; NH_3, 12.5.

Application of the above polymerization procedure to this dimer fraction converted it into a mixture of polymer fractions similar to the one described.

Bulk Polymerization of Acrylamide.—To a solution of 0.4 g. of sodium in 15 ml. of *t*-butyl alcohol was added 40 g. of acrylamide and 0.04 g. of hydroquinone. The alcohol was removed *in vacuo* at 40°, and the residue was cautiously heated (while being stirred with a thermometer) to form a homogeneous melt. In a short time the temperature began to rise rapidly as a vigorous exothermic reaction set in. After polymerization was complete (three minutes), the solid foam was extracted with boiling water for 1 hr., yielding 26 g. of water-insoluble polymer with a specific viscosity of 0.23 (1% in anhydrous formic acid).

β-Alanine.—A solution of 30 g. of crude polymer prepared by bulk polymerization (before water extraction) in 120 ml. of 50% sulfuric acid was refluxed for 4 hr. The cooled solution was diluted with water and adjusted to *p*H 7 with hot aqueous barium hydroxide. After removal of the barium sulfate by filtration, the filtrate was evaporated to dryness. The residue (34 g., 90.5% yield based on acrylamide) melted at 191–193°. One recrystallization from aqueous methanol raised the melting point to 195–196°; a mixed melting point with authentic β-alanine showed no depression. The compound was further identified by conversion to the β-naphthalenesulfonamide derivative, m.p. 136–137° (lit.[22] m.p. 135.5–136.5°).

Polymerization of 2-Propene-1-sulfonamide.—A mixture of 2-propene-1-sulfonamide (1.30 g.), phenyl-β-naphthylamine (0.01 g.) and sodium (0.03 g.) was heated overnight at 100°. The product, a brittle resin, was extracted with 2% acetic acid, taken up in acetone and poured into ether. After drying overnight *in vacuo* the polymer weighed 0.50 g.

(22) H. H. Weinstock, H. K. Mitchell, E. F. Pratt and R. J. Williams, THIS JOURNAL, **61**, 1421 (1939).

WILMINGTON, DELAWARE

[CONTRIBUTION FROM THE RESEARCH DIVISION, RIKER LABORATORIES, INC.]

Alkaloids of *Rauwolfia Canescens* Linn. IV.[1] The Structure of Pseudoreserpine

BY M. W. KLOHS, F. KELLER, R. E. WILLIAMS AND G. W. KUSSEROW

RECEIVED MARCH 20, 1957

The isolation and characterization of pseudoreserpine ($C_{32}H_{38}O_9N_2$), a new alkaloid possessing hypotensive and sedative activity, is reported. On methanolysis pseudoreserpine yielded methyl 3,4,5-trimethoxybenzoate and a new alkamine designated methyl pseudoreserpate, which has been shown to be methyl 17-nor-reserpate by conversion to the 17,18-diacetyl derivative of methyl 17-nor-3-isoreserpate and through the conversion of reserpine to methyl pseudoreserpate.

A continuation of our investigation of the crude reserpine fraction obtained from the roots of *Rauwolfia canescens* Linn. has resulted in the isolation of **pseudoreserpine,** a new reserpine-like alkaloid, possessing hypotensive and sedative activity.

Further development of the chromatogram which had previously yielded deserpidine and reserpine[2] afforded an additional fraction from which crystalline pseudoreserpine was obtained. As a test for homogeneity, this compound was submitted to a 24-plate countercurrent distribution between 5% acetic acid and chloroform–methylchloroform (60:40) and gave a single peak which corresponded well with the theoretical curve for a single substance.

The analyses of pseudoreserpine and its derivatives, as well as equivalent weight determinations, were in accord with the empirical formula $C_{32}H_{38}O_9N_2$. The infrared spectrum of pseudoreserpine showed certain similarities to the spectrum of reserpine in exhibiting ester carbonyl absorption at 5.86 μ and a band at 6.13 μ, attributed to polarization of an indole nucleus by a methoxyl group in the 6-position.[3] The region of >NH, –OH absorption showed two discrete bands at 2.81 and 2.90 μ, one of which could be attributed to an indole >NH, and the other to the possible presence of a hydroxyl group. The existence of the latter group was further demonstrated by the formation of an O-acetyl derivative. The ultraviolet spectrum of pseudoreserpine was identical to that of reserpine and indicated that it also contained the 3,4,5-trimethoxybenzoyl and 6-methoxyindole chromophores.

On treatment with sodium methoxide, pseudoreserpine yielded methyl 3,4,5-trimethoxybenzoate and a new alkamine, methyl pseudoreserpate. The analysis of this new base was in agreement with the empirical formula $C_{22}H_{28}O_5N_2$ and showed the presence of two methoxyl groups. The infrared spectrum of methyl pseudoreserpate revealed a single strong broad band in the >NH, –OH region (2.95 μ) indicative of hydrogen bonding, ester carbonyl absorption at 5.74 μ, and the 6-methoxyindole band at 6.10 μ. The ultraviolet spectrum was identical to that of methyl reserpate, the alkamine of reserpine.

Lithium aluminum hydride reduction of pseudoreserpine afforded the corresponding triol which

(1) A preliminary report of this investigation appeared in a previous communication; *cf.* M. W. Klohs, F. Keller, R. E. Williams and G. W. Kusserow, *Chemistry & Industry*, 187 (1956).

(2) M. W. Klohs, F. Keller, R. E. Williams and G. W. Kusserow, THIS JOURNAL, **77**, 4084 (1955).

(3) N. Neuss, H. E. Boaz and J. W. Forbes, *ibid.*, **76**, 2463 (1954).