mother liquors (yield 88%). The infrared spectrum[10] of the material showed no carbonyl absorption but did indicate strong NH/OH absorption. Analytical data established the product to be 3-anilino-3-phenyl-1-propanol.

A similar reduction using ether as a solvent in place of tetrahydrofuran also gave the amino alcohol, yield 64%.

Anal. Calcd. for $C_{15}H_{17}NO$: C, 79.26; H, 7.54; N, 6.15. Found: C, 79.51, 79.70; H, 7.37, 7.30; N, 6.24, 6.00.

A portion of the amino alcohol was refluxed in benzene for two hours with excess phenyl isocyanate. The benzene was distilled and the solid recrystallized from toluene. The phenylurea-phenylurethan of II melted at 166–167.5°.

Anal. Calcd. for $C_{29}H_{27}N_3O_3$: C, 74.82; H, 5.84; N, 9.02. Found: C, 74.85, 74.85; H, 5.79, 5.65; N, 9.14, 9.31.

Lithium Aluminum Hydride Reduction of β-Phenyl-β-Anilinopropionic Acid.—To a solution of 1.14 g. (0.03 mole) of lithium aluminum hydride in 150 ml. of dry ether was added 7.78 g. of β-phenyl-β-anilinopropionic acid[7] in 140 ml. of tetrahydrofuran and a vigorous exothermic reaction observed. After standing at room temperature for 18 hours the mixture was hydrolyzed with wet ether and 10% aqueous sodium hydroxide. The ether tetrahydrofuran layer was decanted, washed with water and concentrated. The oil remaining did not crystallize so it was taken into toluene, washed with alkali to remove unreduced acid, dried, concentrated to a small volume and cooled. The crystals which separated were recrystallized from toluene and melted at 87–88°. The melting point of this product when mixed with the material from the lactam reduction was not depressed.

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[10] We wish to thank Dr. J. L. Johnson and associates of the Upjohn Department of Physics for the infrared data and Mr. W. A. Struck and associates of the Upjohn Analytical Chemistry Laboratory for the analytical data.

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DEPARTMENT OF CHEMISTRY
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The Preparation of Crystalline *dl*-Pantothenamide

BY MAYNETTE VERNSTEN, W. C. BRAATEN AND M. B. MOORE

RECEIVED MAY 29, 1954

Previous attempts in these laboratories to produce a solid form of pantothenamide led to the discovery of pantothenamide–calcium chloride double salt.[1] Although Wieland, *et al.*,[2] recently have described the synthesis of oily *d*-pantothenamide by another method, it was found here that the addition of pure β-alanine amide to *dl*-pantolactone provides a method of obtaining pure solid *dl*-pantothenamide. Optical crystallographic and X-ray diffraction characteristics proved the crystalline nature of this solid.

Experimental[3]

β-Alanine Amide.—β-Alanine amide hydrochloride (1.54 g., 0.023 mole) prepared from cyanoacetamide by the method of Carlson[4] was slurried in water with the hydroxyl form of IRA 400 resin[5] to remove the chloride ion. The resulting solution was lyophilized to yield the β-alanine amide free base which is hygroscopic and rapidly absorbs carbon dioxide from the air.

***dl*-Pantothenamide.**—β-Alanine amide (0.7 g., 0.0079 mole) and *dl*-pantolactone (1.03 g., 0.0079 mole) were mixed in a flask protected by a soda lime drying tube and then warmed in hot water until the two solids liquefied. After standing at room temperature for three days the clear colorless oil was dissolved in 50 ml. of absolute alcohol, filtered through sintered glass and dried *in vacuo* for two days at 50°. The resulting gummy residue was stirred several times with dry ether and it then crystallized, m.p. 97–100°, 1.5 g. (88%). A sample of this material was dried *in vacuo* at 56°, m.p. 106–108°.

Anal. Calcd. for $C_9H_{18}N_2O_4$: C, 49.52; H, 8.31; N, 12.83. Found: C, 49.07; H, 8.32; N, 12.99.

Acknowledgment.—The authors wish to express their appreciation to M. Freifelder for the reduction of the cyanoacetamide, to E. A. Shelberg and his staff for the microanalyses, and to the Physical Chemistry Department for the physical measurements.

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[1] M. B. Moore, U. S. Patent 2,369,839 (1945).

[2] T. Wieland, E. A. Miller and G. Dieckelmann, *Ber.*, **85**, 1035 (1952).

[3] Melting points are corrected.

[4] G. H. Carlson, U. S. Patent 2,354,909 (1944).

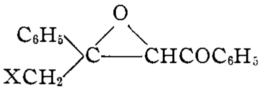
[5] Rohm and Haas Company.

cis- and *trans*-γ-Halo-α,β-epoxy Ketones. Reactions with Aniline

BY HARRY H. WASSERMAN AND JOYCE B. BROUS[1]

RECEIVED JUNE 7, 1954

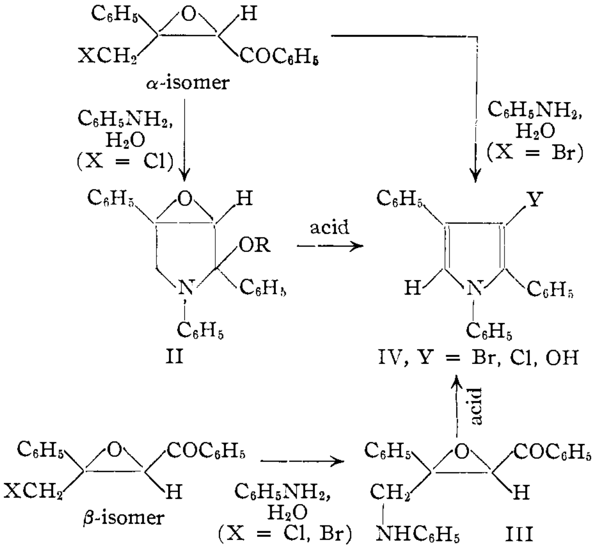
In previous articles[2,3] the reactions of various organic bases with the isomeric γ-halo-α,β-epoxy ketones of general structure I were described. Based on these reactions, conclusions were drawn with respect to the configurations of these so-called halodiphenacyls, namely, that the α-isomers have a *trans* arrangement of phenyl and benzoyl groupings and the β-isomers, the *cis* configuration.



I, X = Cl, Br

The reactions with aniline–water mixtures in the β-series, as summarized in Chart I, led only to a monoanilino derivative III, which could be converted to a pyrrole IV only by a second-stage treat-

CHART I



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[1] Abstracted from a dissertation by J. B. Brous presented to the faculty of Yale University in partial fulfillment of the requirements for the degree of Doctor of Philosophy, 1954.

[2] H. H. Wasserman and J. B. Brous, *J. Org. Chem.*, **19**, 515 (1954).

[3] C. L. Stevens and V. J. Traynelis, *ibid.*, **19**, 533 (1954),