

plus sodamide) or of  $C_6H_5-CHLi-COONa^4$  to Schiff bases. (b) Addition of ethyl phenylacetate to Schiff bases in the presence of aluminum chloride, followed by alkaline hydrolysis.<sup>5-7</sup>

We wish to report that a simple mixture of phenylacetic acid and *N*-benzylidenemethylamine, after being heated at about 100°, affords I, Ar =  $C_6H_5$ , R =  $CH_3$ . No catalyst is necessary. A discussion of possible reaction mechanisms is postponed until we have determined the stereochemical nature (DL-erythro, DL-threo, or some mixture of these) of products formed by the various methods. Only malonic acid has previously been reported capable of adding without catalyst to a carbon-nitrogen double bond to give a beta amino acid.<sup>8</sup>

## EXPERIMENTAL

*2,3-Diphenyl-3-methylaminopropanoic acid.* Equimolecular quantities of phenylacetic acid and *N*-benzylidenemethylamine<sup>9,10</sup> are heated at 100° for 2 hr. in a flask equipped with a calcium chloride drying tube. The reaction mixture, which is almost completely solid at the end of the heating period, is cooled and treated with a 0.1M NaOH solution. Any alkali-insoluble material is removed by filtration. A stream of carbon dioxide gas is then passed through the alkali solution until the  $\beta$ -aminoacid precipitates. After being filtered, washed with water, and dried it melts at 200°.

*Anal.* Calcd. for  $C_{16}H_{17}NO_2$ : C, 75.27; H, 6.71; N, 5.49. Found: C, 74.81; H, 6.85; N, 5.30.

Yields are variable but may reach approximately 75%. Polymerization reactions of the imine, which appear to reduce the yield, may possibly be minimized by the use of an inert diluent.

The  $\beta$ -aminoacid dissolves in very dilute hydrochloric acid. When concentrated hydrochloric acid is added to such a solution, then the hydrochloride of the  $\beta$ -aminoacid precipitates. This substance is easily water soluble but only slightly soluble in fairly concentrated (3M or higher) hydrochloric acid. It melts sharply, but with decomposition, anywhere between 194.5° and 198°, depending on the rate of heating.

*Anal.* Calcd. for  $C_{16}H_{18}ClNO_2$ : C, 65.86; H, 6.22; Cl, 12.15; N, 4.80. Found: C, 65.89; H, 6.34; Cl, 11.99; N, 4.69.

A sample of  $\beta$ -aminoacid hydrochloride prepared by Mollov according to method (b) was reported to melt with decomposition at 190–191°. The free  $\beta$ -aminoacid was not described by Mollov.

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## Levulinic Acid. II.<sup>1</sup> Some Derivatives of 2-Aminoethyl Levulinate

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In connection with other studies a series of esters of levulinic acid were required; most of these have been described previously. Attempts to prepare 2-aminoethyl levulinate by azeotropic esterification of a mixture of levulinic acid and ethanolamine only yielded a viscous gum which could not be distilled; hydrogenation of the same mixture gives 1-(ethan-2-ol)-5-methyl-2-pyrrolid-one.<sup>2,3</sup> Using the azeotropic method, levulinic acid was successfully esterified with 2-dimethylaminoethanol, 2-diethylaminoethanol, and *N*-2-hydroxyethylphthalimide.

EXPERIMENTAL<sup>4</sup>

*2-Dimethylaminoethyl levulinate.* Levulinic acid (116 g., 1.0 mole), 2-dimethylaminoethanol (89 g., 1.0 mole), and benzene (100 ml.) were refluxed in a flask fitted with a Dean and Stark adaptor; water (18 ml.) separated in 24 hrs. The reaction product was washed with water, sodium bicarbonate, and dried. After removal of the solvent the ester had b.p. 156–158°/30 mm.;  $n_D^{23}$  1.4395; yield 61 g. (33%).

*Anal.* Calcd. for  $C_9H_{17}O_3N$ : C, 57.73; H, 9.15; N, 7.49. Found: C, 57.32; H, 9.08; N, 6.90%.

*2-Diethylaminoethyl levulinate.* This ester was prepared in a similar manner and had b.p. 172–176°/30 mm.,  $n_D^{27}$  1.4435; yield 46%.

*Anal.* Calcd. for  $C_{11}H_{21}O_3N$ : C, 61.37; H, 9.83; N, 6.51. Found: C, 61.54; H, 9.45; N, 6.32%.

*2-Phthalimidoethyl levulinate.* *N*-2-hydroxyethyl phthalimide<sup>6</sup> (95.5 g., 0.5 mole.) and levulinic acid (58 g., 0.5 mole.) in benzene (100 ml.) were refluxed for 48 hrs. in a flask fitted with a Dean and Stark adaptor; water (9.5 ml.) separated. After cooling the reaction mixture was washed twice with 2N sodium carbonate; at this stage the product crystallized and was collected by filtration. The dried product was recrystallized from ethanol m.p. 90–93°; yield 89 g. (62%).

*Anal.* Calcd. for  $C_{15}H_{15}O_5N$ : C, 62.28; H, 5.23; N, 4.84. Found: C, 62.58; H, 5.35; N, 4.91%.

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## Monomeric and Polymeric Compositions from Carbethoxymethyl Isocyanate<sup>1</sup>

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Recent work in the field of synthetic polypeptides has provided improved techniques for the

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