acetonitrile with various *t*-aminoethyl chlorides, using sodium amide or lithium amide as described.[13-16]

Method B.—The basic amides were prepared by 90% sulfuric acid hydrolysis of the nitriles obtained by method A.[14,16]

Method C.—A mixture of 0.17 mole of the amidone-type amide and 1 mole of thionyl chloride was prepared in an ice-bath. No reaction occurred. This mixture was heated on a steam-bath for one to three hours, made strongly alkaline with sodium hydroxide and extracted with ether. The extracts were dried over potassium carbonate, and the ether removed by evaporation. The residual basic materials were purified by crystallization or fractionation *in vacuo* followed by salt formation as indicated in the table.

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Lithium Aluminum Hydride Reduction of Diethyl 2,6-Dimethyl-3,4-pyridinedicarboxylate

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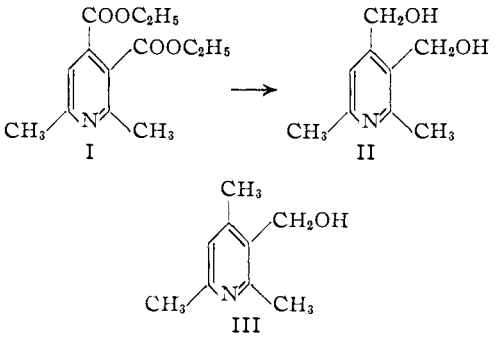
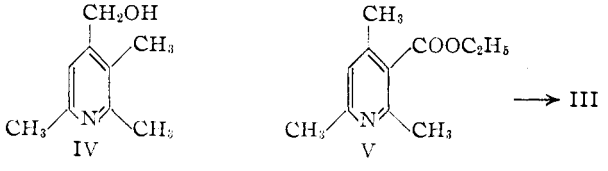
In a previous paper[1] it was shown that reduction of diethyl 2,6-dimethyl-3,4-pyridinedicarboxylate (I) with lithium aluminum hydride gave either the corresponding glycol (II) or a monohydric alcohol ($C_9H_{13}NO$) depending on the conditions used. The



latter product was formulated as III by analogy with the known reductive cleavage of pyridoxine to desoxypyridoxine,[2] and this alternative was favored in a review by Rudinger, Ferles and Protiva.[3] However, an unequivocal proof of structure was lacking. Subsequently Gaylord[4] suggested that the alternative formulation IV was preferred over III by analogy with the course of a number of other hydrogenolysis reactions effected by lithium aluminum hydride. In order to resolve this question we have now synthesized III by reduction of the ester V.[5]



The alcohol III so obtained, m.p. 87–88.5°, was not identical with the isomer, m.p. 127–128°, derived from I. Since the structure of III was established by its derivation from V, the monohydric alcohol obtained from I must be formulated as IV and not III. The 4-hydroxymethyl isomer IV was also obtained when the glycol II was subjected to catalytic hydrogenolysis in the presence of palladium catalyst. It is evident, therefore, that both chemical and catalytic reduction result in cleavage of the hydroxymethyl group in the 3-position, and the conclusion of Gaylord[4] appears to be correct.

(1) R. G. Jones and E. C. Kornfeld, THIS JOURNAL, **73**, 107 (1951).
(2) S. Harris, *ibid.*, **62**, 3203 (1940).
(3) J. Rudinger, M. Ferles and M. Protiva, *Chem. Listy*, **45**, 309 (1951).
(4) N. G. Gaylord, *Experientia*, **10**, 166 (1954).
(5) R. Michael, *Ann.*, **225**, 121 (1884); A. Hantzsch, *ibid.*, **215**, 42 (1882).

Experimental[6]

2,4,6-Trimethyl-3-hydroxymethylpyridine (III).—A solution of 1.6 g. of lithium aluminum hydride in 100 ml. of dry ether was stirred in an ice-bath, and to it was added dropwise during about 30 minutes a mixture of 8.0 g. of ethyl 2,4,6-trimethyl-3-pyridinecarboxylate and 100 ml. of ether. Stirring was continued for one-half hour at room temperature, after which the reaction mixture was treated cautiously with 3 ml. of water and 50 ml. of methanol. The suspension was saturated with carbon dioxide, filtered, and the solid was extracted twice with 50-ml. portions of hot methanol. The combined filtrates were evaporated to dryness, and the residue was taken up in chloroform. The chloroform solution was filtered, and the solvent was distilled. The residue was taken up in acetone, and the solution was filtered and then treated with dry hydrogen chloride. The salt which separated was filtered (2.1 g.) and recrystallized from a mixture of methanol and acetone, m.p. 168–170°.

Anal. Calcd. for $C_9H_{13}NO \cdot HCl$: C, 57.60; H, 7.52; N, 7.46; Cl, 18.89. Found: C, 58.09; H, 7.65; N, 7.62; Cl, 18.71.

The salt was dissolved in a little water, and the solution was treated with excess 50% aqueous sodium hydroxide. The oily product was extracted with chloroform; the extract was dried over magnesium sulfate, and the solvent was distilled. The hydroxymethyl compound was crystallized from acetone, m.p. 87.0–88.5°.

Anal. Calcd. for $C_9H_{13}NO$: C, 71.49; H, 8.67; N, 9.26. Found: C, 71.27; H, 8.66; N, 9.21.

The infrared spectrum in chloroform solution was different from that of the isomer IV, and the dissociation constant in water (pK'_a = 7.10) also differed from that of IV (pK'_a = 7.30).

2,3,6-Trimethyl-4-hydroxymethylpyridine (IV) by Hydrogenolysis of 2,6-Dimethyl-3,4-di-(hydroxymethyl)-pyridine.[1]—The glycol (1.0 g.) was hydrogenated for three hours at 50 pounds per square inch pressure in 50 ml. of glacial acetic acid using 1.0 g. of 5% palladium-on-carbon catalyst. The catalyst was filtered, and the filtrate was concentrated to dryness *in vacuo*. The residue was dissolved in water, the excess sodium hydroxide was added. The product was extracted with three 20-ml. portions of chloroform, and the extracts were dried over magnesium sulfate and concentrated. The product was crystallized from acetone; yield 0.35 g. (39%), m.p. 127–128°. A mixture melting point with a sample obtained by lithium aluminum hydride reduction of the diester[1] I was not depressed.

(6) Melting points are uncorrected.

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Potential Anti-viral Agents. I. N,N-Dimethyl-N'-isopropyl-N'-(2-nitroisobutyl)-ethylenediamine Hydrochloride

BY FREDERICK LEONARD AND FLOYD E. ANDERSON

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In recent years, increasing attention has been focused on the anti-viral and anti-rickettsial properties of a variety of nitro compounds. Chloram-