Anal.[9] (On compound prepared in first experiment). Calc'd for $C_{27}H_{26}Si$: Si, 7.43; C, 85.65; H, 6.92. Found: Si, 7.66, 7.61; C, 86.61, 85.91; H, 7.16, 7.24.

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1-Phenyl-4-methyl-6-pyridazone-3-carboxylic-Acid Derivatives

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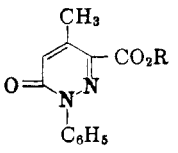
1-Phenyl-4-methyl-6-pyridazone-3-carboxylic acid (Ia), which has been made available for the first time by the rearrangement of γ-keto-β-



I

a, R = H
b, R = CH_3
c, R = C_2H_5
d, R = $N(C_2H_5)_2$
e, R = NHC_6H_5

methylglutaconic anhydride phenylhydrazone,[2] has been converted to its methyl ester (Ib), ethyl ester (Ic), N,N-diethylamide (Id), and anilide (Ie). The preparation and properties of these derivatives are recorded in this Note. All were prepared from the acid chloride which was in turn prepared from the acid.

EXPERIMENTAL

1-Phenyl-4-methyl-6-pyridazone-3-carboxylic acid chloride. One gram of 1-phenyl-4-methyl-6-pyridazone-3-carboxylic acid was warmed one hour with 15 ml. of thionyl chloride. The yellow residue remaining after removal of the excess thionyl chloride under a vacuum was recrystallized from carbon tetrachloride to give 1.1 g. of white needles melting at 135°. This chloride was used in the following reactions.

Methyl 1-phenyl-4-methyl-6-pyridazone-3-carboxylate (Ib). The acid chloride (0.5 g.) was refluxed one hour with absolute methanol. Evaporation of the solvent left a solid which was recrystallized from petroleum ether to give 0.4 g. (76.3%) of the methyl ester as white needles, m.p. 125°.

Anal. Calc'd for $C_{13}H_{12}N_2O_3$: N, 11.47. Found: N, 11.16.

Ethyl 1-phenyl-4-methyl-6-pyridazone-3-carboxylate (Ic). The acid chloride (0.5 g.) was refluxed 2.5 hours with 15 ml. of absolute ethanol. Dilution with water precipitated a solid which was recrystallized from ethyl acetate to give 0.5 g. (83.5%) of the ethyl ester as white needles, m.p. 102°.

Anal. Calc'd for $C_{14}H_{14}N_2O_3$: N, 10.85. Found: N, 10.65.

N,N-Diethyl-1-phenyl-4-methyl-6-pyridazone-3-carboxamide (Id). To a solution of 0.5 g. of the acid chloride in 25 ml. of hot toluene was added 2 ml. of diethylamine. After 0.5 hour at reflux the mixture was filtered hot to separate the amine hydrochloride and was cooled. Addition of 25 ml. of petroleum ether precipitated a crude product. On recrystallization from petroleum ether 0.4 g. (66%) of the N,N-diethylamide, m.p. 54°, was obtained.

Anal. Calc'd for $C_{16}H_{21}N_3O_3$: N, 13.85. Found: N, 13.75.

1-Phenyl-4-methyl-6-pyridazone-3-carboxanilide (Ie). To a solution of 0.5 g. of the acid chloride in 25 ml. of hot toluene was added 2 ml. of freshly distilled aniline. After one hour at reflux, the mixture was filtered hot to separate the aniline hydrochloride. On cooling the crude product precipitated. Recrystallization from a mixture of equal parts of toluene and petroleum ether gave 0.4 g. (70%) of the anilide as white needles, m.p. 259°.

Anal. Calc'd for $C_{18}H_{15}N_3O_2$: N, 13.76. Found: N, 13.89.

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A Wayward Prevost Oxidation

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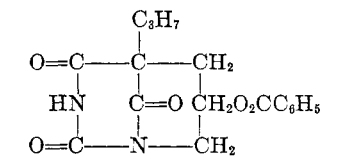
In connection with our study of central nervous system depressants, we were interested in some derivatives of 5-isopropyl-5-(2′,3′-dihydroxypropyl)barbituric acid. A likely approach to the dibenzoate of this compound appeared to be through a Prevost[1] oxidation of the commercially available 5-isopropyl-5-allylbarbituric acid using the silver benzoate-iodine complex. The product we obtained, however, did not analyze for the expected compound. It gave a blue color with alcoholic cobalt nitrate and potassium hydroxide [Mohrschulz[2] test for barbituric acid], but unlike this test for the starting material, the color disappeared with continued addition of the alcoholic potassium hydroxide. Rather the analysis corresponded to the formula



This structure can be explained by the action of the silver benzoate-iodine complex to give 5-

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(2) Wiley and Jarboe, Jr., *J. Am. Chem. Soc.*, **77**, 403 (1955).

(1) Prevost, *Compt. rend.*, **196**, 1129 (1933); **197**, 1661 (1933); Prevost and Lutz, *Compt. rend.*, **198**, 2264 (1934); Prevost and Wiemann, *Compt. rend.*, **204**, 700, 989 (1937).

(2) Mohrschulz, *Munchen med. Wochschr.*, **81**, 672 (1934).