carbon. The barium carbonate derived from C-20 of the biosynthesized cortisol-C^{14} contained 48,000 d/m/mM.

On the basis of the scheme postulated by Woodward and Bloch[5] one would expect ten radioactive carbons to be incorporated into the first 21 carbons of cholesterol from acetate-1-C^{14}. If one assumes that corticosteroids are biosynthesized from acetate-1-C^{14} through cholesterol then the arrangement of all carbons and the number of C^{14} atoms in corticosteroids would be expected to be the same as that found in the first 21 carbons of cholesterol. Our findings demonstrate the absence of radioactivity in carbons 21 of both cortisol-C^{14} and corticosterone-C^{14}. By our method of counting we can say that if radioactivity was present in C-21 of cortisol the value must be less than 8.1% of that found in C-20. The count of 48,000 d/m/mM. found in C-20 of cortisol-C^{14} is in reasonable agreement with the calculated value of 52,500 d/m/mM. Our findings demonstrate that the arrangement of methyl and carboxyl carbons in the corticoid side chain is identical to that of carbons 20 and 21 of cholesterol.

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The Preparation of 3,4-Bis(2-furyl)-1,2,5-oxadiazole[1,2]

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INTRODUCTION

A compound, later identified as 3,4-bis(2-furyl)-1,2,5-oxadiazole, precipitated gradually from urea solutions of α-furildioxime prepared in connection with a study of the effect of urea on the solubility of water-soluble *vic*-dioximes. 3,4-Bis(2-furyl)-1,2,5-oxadiazole was characterized by Tsumaki and Yamaguchi[3] who originally obtained it as a product of the pyrolysis of bis(α-furildioximato-N,N′)-nickel(II).

3,4-Bis(2-furyl)-1,2,5-oxadiazole has been prepared in 58% yield by treating α-furildioxime with urea solution. This compound has been prepared also from both the α- and γ-furildioxime by modifying the conditions of a sealed tube reaction described by Auwers and Meyer[4] for the preparation of diphenylfurazan.

The effect of urea on the aqueous solubility of 2,3-butanedionedioxime, 1,2 - diaminoethanedionedioxime, 3-methyl-, 4-methyl-, 3-ethyl-, 4-isopropyl-, and 4-*tert*-amyl-1,2-cyclohexanedionedioxime, 1,2-cycloheptanedionedioxime, 1,2-cyclohexanedionedioxime and α-benzildioxime was also studied. This catalytic dehydration by urea does not seem to be a general reaction since none of the above aliphatic, alicyclic or aromatic *vic*-dioximes were converted to the corresponding 1,2,5-oxadiazoles by urea under the conditions found most satisfactory for preparing 3,4-bis(2-furyl)-1,2,5-oxadiazole.

EXPERIMENTAL WORK

From α-furildioxime. α-Furildioxime, 1.2556 g. (0.0057 mole) was treated with 25 ml. of 50% (w/w) urea solution, and the mixture was warmed to 80°. The solution soon became discolored and cloudy, and an oily phase appeared. Heating was continued until the aqueous phase became clear. As the mixture cooled, the 3,4-bis(2-furyl)-1,2,5-oxadiazole solidified and was collected by filtration. This material was dissolved in the minimum amount of methanol and was precipitated by the addition of 150 ml. of distilled water. The yield of 3,4-bis(2-furyl)-1,2,5-oxadiazole, m.p. 61–62°, was 0.6602 g. (58%).

α-Furildioxime, 1.3625 g. (0.006 mole) and water, 2.5 g. (0.14 mole), were sealed in a 200-ml. container constructed from borosilicate tubing ($1^3/_4$-inch O.D., $^3/_{32}$-inch wall thickness). The container then was placed in a 12-inch section of 2-inch iron pipe closed at both ends with pipe caps. The container was heated in an oven at 175° for 8 hours. Upon cooling, the container was opened, and the contents were rinsed out with methanol. The methanolic solution of the product was heated, filtered, and concentrated by evaporation to a volume of 5 ml. Crystallization of the 3,4-bis(2-furyl)-1,2,5-oxadiazole was effected by the addition, with swirling, of 150 ml. of distilled water. The yield was 0.1515 g. (12%).

From γ-furildioxime. 3,4-Bis(2-furyl)-1,2,5-oxadiazole was obtained by heating γ-furildioxime with water in a sealed container for 12 hours in an oven at 165°. The amount of water necessary to produce a pressure of 25 atmospheres was calculated from the simple gas law. The product was purified by the previously described procedure. The yield from this isomer of the dioxime was 20.5%.

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[1] No. XIX in a series on "Chemistry of the *vic*-Dioximes." Previous paper in this series is No. XVIII, *J. Org. Chem.*, **21**, 547 (1956).

[2] Contribution No. 430; work was performed in the Ames Laboratory of the U. S. Atomic Energy Commission.

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Phenylboronates of Pentoses and 6-Deoxyhexoses

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Although it is known that boric acid and boric acid derivatives react with polyhydric alcohols and with sugars, stable products have not been isolated from the reducing sugars.[2-4] Kuivila and coworkers[4] have described the phenylboronates, a new

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