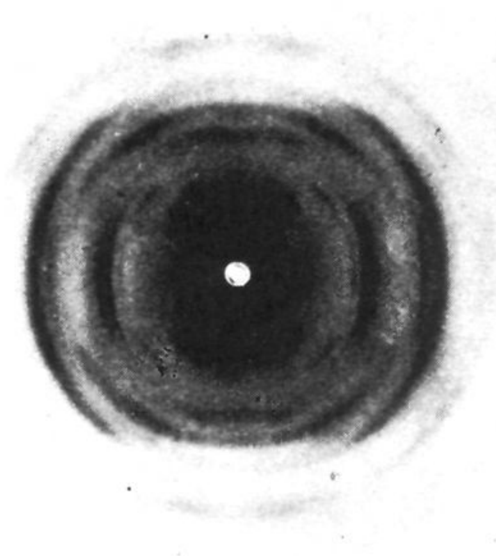

Fig. 1.—X-Ray diffraction photograph of a sodium pectate fiber: CuKα radiation; fiber axis vertical; camera distance 5.0 cm.

values the identity period in the fiber direction can be determined to be approximately 13.1 Å. This value lies between the maximum value (10.4 Å.), observed when there are two pyranose rings in the identity period, as in cellulose and chitin, and the value (15.2 Å.) observed for certain cellulose derivatives[2] when there are three pyranose rings in the identity period.

The symmetry of the galacturonide chain in pectin evidently approximates that of a threefold screw axis but with the angle between the plane of the pyranose rings and the fiber axis somewhat larger than occurs in cellulose and its derivatives. The threefold screw axis is also suggested by the fact that the sodium pectate fiber diagram can be indexed on a hexagonal lattice. The true unit cell can only be pseudohexagonal, however, for reasons that will be discussed elsewhere.

A detailed analysis of the fiber diagram will be published in the near future.

[2] J. Gunderman, *Z. physik. Chem.*, **37B**, 387 (1937).

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RECEIVED APRIL 16, 1945

THE SYNTHESIS OF 3,4-DIAMINOCARBETHOXYFURAN

Sir:

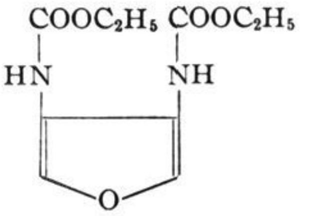
The recent interest in 3,4-diaminocarbethoxyfurans[1] prompts us to record a synthesis of the parent compound of this series (I).

Furan-3,4-dicarboxylic acid was prepared according to the literature.[2] Treatment of the diacid with phosphorus pentachloride in benzene readily gave the diacid chloride in 85% yield. This crystallized from benzene in colorless needles (m. p. 76°; calcd. for $C_6H_2O_3Cl_2$: Cl, 36.7. Found: Cl, 36.5). Hydrolysis of this acid chloride gave a diacid which did not depress the melting point of authentic furan-3,4-dicarboxylic acid. Heating with methyl alcohol gave a dimethyl ester identical with that prepared from the diacid and diazomethane.

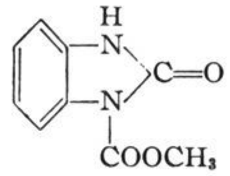
Concentrated ammonium hydroxide immediately gave the corresponding diamide (m. p. 262° [dec.]; calcd. for $C_6H_6O_3N_2$: N, 18.18. Found: N, 18.30). It is interesting to note that the success of this method of preparation of the 3,4-diamide is in striking contrast to the great difficulty experienced in attempting to make some similar compounds from the dicarboxylic esters.[3,4]

Reaction of the diacid chloride with sodium azide in cold aqueous acetone gave the corresponding diazide in almost quantitative yield. This compound is stable when moist, but explodes on rubbing when dry. Heating the diazide with ethyl alcohol gave the desired 3,4-diaminocarbethoxyfuran (I) in good yield. It formed small, colorless crystals from ligroin, m. p. 166–167° (calcd. for $C_{16}H_{14}O_5N_2$: N, 11.6. Found: N, 11.4).[5]

It is of interest that the corresponding reaction in the benzene series gives not the diaminocarbethoxy compound, but (II), the cyclized benzimidazole derivative.[6] This is another example of the difficulty of fusing a five-membered ring in the 3,4-position of a furan nucleus.



(I)



(II)

The work reported in this Communication had to be discontinued almost two years ago.

DEPARTMENT OF CHEMISTRY
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RECEIVED APRIL 12, 1945

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[1] Hofmann, THIS JOURNAL, **67**, 694 (1945).

[2] Reichstein, Grüssner, Schindler and Hardmeier, *Helv. Chim. Acta*, **16**, 276 (1933).

[3] Seka, *Ber.*, **57**, 1864 (1924).

[4] Bilton and Linstead, *J. Chem. Soc.*, 922 (1937).

[5] Analysis kindly performed by Paul Kletzke.

[6] Lindemann and Schultheis, *Ann.*, **464**, 237 (1928)