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# The Progessive and Step-Wise Decarboxylation of Furan-Tetracarboxlic Acid

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# THE PROGRESSIVE AND STEP-WISE DECARBOXYLATION OF FURAN-TETRACARBOXYLIC ACID

HENRY GILMAN, HAROLD OATFIELD AND
WILLARD H. KIRKPATRICK

Tetra-ethyl furantetracarboxylate is readily prepared by the condensation of oxalacetic ester. The corresponding furantetracarboxylic acid can be progressively decarboxylated. Two of the interesting products of decarboxylation are 3, 4-furandicarboxylic acid and 3-furoic acid. The 3, 4-furandicarboxylic acid is interesting not only for purposes of absolute orientation (it completes the series of furandicarboxylic acids) but also because it gives unequivocal evidence for the constitution of a series of 2-nitrofurans. It now appears that this method of stripping carboxyl groups is the best now available for the synthesis of 3-furoic acid, which otherwise is prepared with considerable difficulty.

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# THE PREPARATION OF DIBROMOAMINE AND ITS REACTION WITH GRIGNARD REAGENTS

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Dibromoamine was prepared by passing dry ammonia into a cold ethereal solution of bromine. The reaction may be represented by the equation:

$$3\ NH_3 + 2\ Br_2 \rightarrow NHBr_2 + 2\ NH_4Br$$

A study of the decomposition rates of the dibromoamine solution at 0° and –72° shows that the product decomposes very rapidly at 0°, but it is relatively stable at the lower temperature.

Dibromoamine reacts with Grignard reagents to produce primary amines, secondary amines, ammonia, and nitrogen. The percentage yields of these products obtained in two typical reactions were as follows: for n-butyl magnesium chloride; n-butylamine 7.8 per cent, di-n-butylamine 2.2 per cent, ammonia 79.0 per cent, nitrogen 5.9 per cent; for benzyl magnesium chloride; benzylamine 29.6 per cent, dibenzylamine 5.5 per cent, ammonia 42.8 per cent, nitrogen 4.7 per cent.

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