acetic acid removed in this manner from the intermediate compound, which may or may not be a product of ring scission, can also be removed by means of other bases like dimethylaniline. The nitrofurfural is readily obtained, if necessary, by hydrolysis[1] of the crude diacetate.

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CONTRIBUTION FROM THE
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A Note on the Preparation of Glycine.—The classical method of preparing glycine through the interaction of chloro-acetic acid and ammonia with the subsequent formation of the copper or lead salt has been abandoned by Clarke and Taylor[1] in favor of a method involving the hydrolysis of methylene-amino-acetonitrile. The starting material is fairly expensive and the yield of 31–37% is not high. Robertson,[2] after a study of the velocity and course of the reaction between chloro-acetic acid and ammonia, recommends the use of a large excess of ammonia. When the molecular ratio of ammonia to chloro-acetic acid is 60:1, the actual concentration of glycine in solution is increased to 86%. He eliminates the ammonium chloride by means of silver oxide and obtains a final yield of 50% of pure glycine.

It was thought that a correspondingly large yield of pure glycine could be obtained by making use of this high ratio of ammonia to chloro-acetic acid, and crystallizing the glycine directly from the concentrated reaction mixture in the presence of pyridine according to the method of Clarke and Taylor or in the presence of aniline as suggested by Benedict,[3] thus eliminating the time and expense involved in the use of silver oxide to remove the ammonium chloride. The attempt proved successful and the following method was worked out.

Two moles of chloro-acetic acid (189 g.) is dissolved in 8 liters of ammonium hydroxide (sp. gr. 0.90) in a 12-liter flask and allowed to stand at room temperature for forty-eight hours. The excess of ammonia is distilled off and recovered. The mixture is concentrated, *in vacuo* if preferred, until precipitation of ammonium chloride begins. The salt is dissolved by warming on the steam-bath with the addition of the smallest possible volume of water. If the solution is not clear, it is filtered by suction. The filtrate and washings should occupy a volume of about 500 cc. A

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[1] Clarke and Taylor, "Organic Syntheses," John Wiley and Sons, Inc., New York, 1925, Vol. IV, p. 31.

[2] Robertson, THIS JOURNAL, 49, 2889 (1927).

[3] Benedict, *ibid.*, 51, 2277 (1929).

mixture of 800 cc. of methyl alcohol and 140 cc. of pyridine is stirred in. Crystallization of the glycine begins at once. After standing overnight, the glycine is filtered off, suspended in methyl alcohol, filtered and washed with methyl alcohol. A yield of 96 g. or 64% is obtained. A further yield of 2 to 3 g. may be secured from the combined mother liquor and washings on standing. If an attempt is made to crystallize the glycine from a warm solution of much greater concentration than the one suggested, the product will be contaminated with considerable ammonium chloride. This may be almost completely removed by washing with methyl alcohol. The glycine is recrystallized by dissolving in 300 cc. of water with warming. In order to remove the last traces of ammonia, 6 g. of permutit is added and after thorough stirring the mixture is filtered through a charcoal mat. The solution with washings should occupy about 400 cc. and should be crystal clear; 800 cc. of methyl alcohol is stirred in and the mixture is allowed to stand overnight until crystallization is complete. The glycine is filtered off and washed with methyl alcohol. The yield is 81 g. or 54%. The product is free from the chloride ion and from ammonia, as shown by testing with Nessler solution. It melts at from 225–230° (corr.), and shows the theoretical percentage of nitrogen and amino nitrogen. An equivalent amount of aniline may be substituted for the pyridine if desired in the first crystallization but the product carries a slight yellow color. This is completely removed on recrystallization.

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o-Phenetylurea.—On account of its industrial importance, dulcin has received considerable attention while the corresponding ortho derivative has been slighted. The writer finds but one literature reference in a paper by Pierron,[1] who prepared this compound from *o*-ethoxyphenylcyanamine as a means of identification of the latter. He quotes a melting point of 206°, which appears to be too high. It seemed worth while to make the compound principally in order to compare its properties with those of dulcin derivatives now in preparation.

A 5-g. portion of *o*-phenetidine is treated with 40 cc. of water and 2.5 cc. of concentrated hydrochloric acid. A solution of 2.25 g. of potassium cyanate in 20 cc. of water is added in small portions with a thorough shaking between additions. Precipitation is complete in about ten minutes. After filtration the solid is washed with dilute ammonium hydroxide and then with ether. It is at once recrystallized from hot dilute ethanol to

[1] Pierron, *Ann. chim. phys.*, [7] **15**, 145 (1908).