

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 permittit 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.

CONTRIBUTION FROM THE  
CHEMICAL LABORATORY OF  
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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,<sup>1</sup> 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

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

which sufficient ammonium hydroxide is added to give a decided odor. The crystals are washed with ether. When dry the purification is completed by a recrystallization from hot benzene; yield, 2 g.; white microscopic needles. The compound shows a slight shrinkage at about  $139^{\circ}$  and melts at  $142\text{--}143^{\circ}$ . It is tasteless and odorless. Concentrated sulfuric acid gives a colorless solution which upon heating becomes a faint straw color.

*Anal.* Calcd. for  $C_9H_{12}O_2N_2$ : C, 60.0; H, 6.66; N, 15.56. Found: C, 60.10; H, 6.72; N, 15.64.

The compound is soluble in hot water, ethanol, amyl alcohol and hot benzene. It is very slightly soluble in ether.

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## COMMUNICATIONS TO THE EDITOR

### DR. C. S. HUDSON'S VIEWS ON THE RELATIONSHIP OF STRUCTURE TO THE OPTICAL ROTATIONS OF SUGARS

*Sir:*

Dr. C. S. Hudson has not utilized the means which were open to him to test the validity of his views by direct chemical experiments. The basis on which he develops his argument is the presumed existence, which his statistical methods enable him to detect, of a new form of mannose (calculated  $[\alpha]_D + 77^{\circ}$ ) in derivatives of 4-glucosido-mannose, obtainable from cellobiose through cellobial. If this foundation for his scheme fails, then the entire superstructure of rival formulas which he has raised upon it must collapse.

A survey of his two recent papers [THIS JOURNAL, 52, 1680, 1707 (1930)] has led me to select for this critical test an experimental method which he has tacitly approved: he has accepted and utilized the observation of Fischer and Armstrong that  $\beta$ -methylmaltoside gives rise by enzyme hydrolysis to  $\beta$ -methylglucoside without ring change. Implicit in Dr. Hudson's scheme, therefore, is the expectation that 4-glucosido- $\alpha$ -methylmannoside will yield by enzyme cleavage his hypothetical  $\alpha$ -methylmannoside ( $[\alpha]_D + 125^{\circ}$ ), inasmuch as this is the glycoside of the unknown form of mannose to which he has assigned the 1,5-ring.

With my colleague Dr. E. L. Hirst and other co-workers (R. J. W. Reynolds, H. R. L. Streight, H. A. Thomas, J. I. Webb and Miss M. Plant) I have prepared and investigated the chemical behavior of both 4-glucosido- $\alpha$ -methylmannoside and 4-galactosido- $\alpha$ -methylmannoside to which the 1,4-ring cannot apply since the 4-position in the mannose residue is occupied by the biiose link. Both these substances are hydrolyzed by