colorless crystals, m.p. 150.2–152.1°. *Anal.* Calcd. for $C_9H_8N_4$: C, 62.78; H, 4.68; N, 32.54. Found: C, 62.82; H, 4.68; N, 32.47.

2-Pyrimidyl-2'-thiazolylamine.—The sodium salt of 2-aminopyrimidine (22.18 g., 0.189 mole) and 2-chlorothiazole (22.58 g., 0.189 mole) were heated in an oil-bath at 95° for 2.75 hours when it became necessary to cool the reaction mixture in order to moderate an exothermic reaction which had developed; the reaction mixture was then heated for an additional 6 hours at 95°, cooled, suspended in 50 ml. of water and steam distilled (6 g. of 2-chlorothiazole was recovered). The residue from the steam distillation was filtered, washed with cold water and dried at 100°; yield 3.1 g. (9.5%), m.p. 209–211.8°. Cream colored crystals were obtained upon recrystallization from ethanol, m.p. 211–212.1°. Vacuum sublimation under 6 mm. pressure and recrystallization from ethanol gave colorless crystals, m.p. 212.1–212.6°. *Anal.* Calcd. for $C_7H_6N_4S$: C, 47.18; H, 3.39; N, 31.44; S, 17.99. Found: C, 47.22; H, 3.44; N, 31.44; S, 17.89.

2-Anilinopyrimidine.[10]—A mixture of aniline (3.26 g., 0.035 mole) and sodium amide (1.37 g., 0.035 mole) in 35 ml. of dry benzene was heated and stirred for 3.25 hours in an oil-bath at 92° under an atmosphere of nitrogen. 2-Chloropyrimidine (4 g., 0.035 mole) in 20 ml. of anhydrous benzene was added to the cooled reaction mixture. After the addition, this mixture was heated and stirred at gentle reflux for 4.5 hours, cooled and then steam distilled. The distillate was acidified with concentrated hydrochloric acid and shaken to extract the amine from the benzene portion of the distillate. A brown colored solid was precipitated from the acidic solution upon adjusting it to a *p*H 10; yield 2.7 g. Solution of this substance in boiling water and filtration from insoluble impurities produce 1.3 g. (22%) of colorless needles, m.p. 114.3–115.2°.

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[10] T. B. Johnson and F. W. Heyl, *Am. Chem. J.*, **38**, 244 (1907), prepared this compound by the reduction of 4-chloro-2-anilinopyrimidine, m.p. 116°.

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The Hydrolysis of Glucose-4-phosphate[1]

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The mechanism by which galactose may be derived from glucose in biological systems is one which holds considerable interest. Robinson[3] suggested that D-glucose-4-phosphate, if present in nature, might be hydrolyzed at the C–O– bond at carbon number four with a resultant Walden inversion. This mechanism is referred to repeatedly, but it has never been tested directly. Cohn[4] has shown, using O^{18}, that hydrolysis of glucose-1-phosphate may occur at the C–O– bond or the –O–P bond depending on the catalyst used.

Experimental

The disodium and barium salts of D-glucose-4-phosphoric acid were prepared in this Laboratory.[5]

Acid Hydrolysis.—To 28 mg. of barium D-glucose-4-phosphate was added excess (2 ml. of 0.1 *N*) sulfuric acid. After barium sulfate was removed by centrifugation, the solution was maintained at 100° for 30 hours. The hydrolysis mixture was concentrated, extracted with pyridine[6] and chromatographed[7] (descending) on Whatman No. 1 paper, using *s*-collidine–water as a solvent. The spray used was that recommended by Trevelyan.[8] Well defined glucose spots were obtained, but no galactose spot was discernible. There was no difficulty in differentiating the spots due to the glucose and galactose standards.

Hydrolysis at *p*H 7.—A solution of 21.4 mg. of the disodium salt of glucose-4-phosphoric acid in 2.0 ml. of water was found to be at *p*H 7.2. After 42 hours heating at 100° in a stoppered tube 50% of the compound was hydrolyzed as evidenced by analysis for inorganic phosphate.[9] The solution was chromatographed as above and identical results were obtained.

Acid Phosphatase Action.—The enzyme used was obtained by ammonium sulfate precipitation of potato press juice.[10] A sample of 20.6 mg. of disodium glucose-4-phosphate was dissolved in 2.0 ml. of water. To each volume of this solution used was added an equal volume of molar acetate buffer, *p*H 5.2, and an equal volume of purified phosphatase solution. Liberation of inorganic phosphate indicated 90% hydrolysis after 12 hours incubation at 37°. A chromatogram of the solution showed only a glucose spot.

Alkaline Phosphatase Action.—Essentially identical experiments were performed using Armour intestinal phosphatase as a catalyst at a *p*H of 8.0. Neither chromatography nor the colorimetric method of Dische,[11] *et al.*, indicated the presence of galactose in the hydrolysis mixtures.

Conclusion.—The above results do not suggest the idea that Walden inversion occurs during the hydrolysis of sugar phosphates and in this they agree with the work of Cohn.[4] The mechanism of cleavage will be further investigated with O^{18}.

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[1] Work performed under Contract N6onr-218, Office of Naval Research.

[2] To whom inquiries concerning this article should be addressed.

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The Reaction between Diazonium Fluoborates and Antimony Trichloride in Organic Solvents

BY G. O. DOAK, LEON D. FREEDMAN AND STELLA M. EFLAND

The present paper describes the reaction between antimony trichloride and diazonium fluoborates and is a continuation of our study of the reaction between the halides of certain elements and diazonium fluoborates in organic solvents. Under conditions similar to those employed with arsenic trichloride,[1] a mixture of arylstibonic and diarylstibinic acids was obtained. A number of attempts were made to separate the mixture of primary and secondary acids. Fractional crystallization of various derivatives of these acids was used, and it was found possible to obtain the pure secondary acids in low yields. However, analyses of the primary acids and m.ps. of the corresponding pyridinium chloroantimonates[2] indicated that the primary acids were invariably contaminated with small amounts of secondary acids.

The total yield as well as the ratio between the yields of the two acids varied with both the solvent

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[1] G. O. Doak and L. D. Freedman, THIS JOURNAL, **73**, 5658 (1951).

[2] *Cf.* G. O. Doak and H. G. Steinman, *ibid.*, **68** 1987 (1946).