A complete study of the applicability of the synthetic method outlined above is in progress.

THE BAKER LABORATORY OF CHEMISTRY

CORNELL UNIVERSITY ITHACA, NEW YORK RECEIVED JANUARY 20, 1947

## THE CRYSTALLINE OCTAACETATE OF $6-\alpha$ -D-GLUCOPYRANOSIDO- $\beta$ -D-GLUCOSE

Sir:

The polysaccharide dextran ( $[\alpha]^{20}D + 180^{\circ};$ c 1, water) produced by Leuconostoc dextranicum was hydrolyzed at room temperature in 2% solution in 30% hydrochloric acid to approximately two-thirds completion ( $[\alpha]^{20}$ D of solution + 105°). After removal of inorganic ions by successive treatment with lead carbonate, hydrogen sulfide, sodium bicarbonate and Amberlite resins (IR-100 and IR-4), the D-glucose was removed by yeast fermentation and the sirup obtained on solvent removal was acetylated with hot acetic anhydride and sodium acetate. The resultant mixture of sugar acetates was chromatographed<sup>1</sup> on Magnesol-Celite employing benzene-ethanol development under such conditions that any monosaccharide present would be removed from the column. The material from the lowest zone was rechromatographed in similar fashion and the lowest zone material again obtained was crystallized (elongated prisms) from ethanol; m. p. 143–144°,  $[\alpha]^{25}D + 97^{\circ}$  (c 2.7, chloroform).

Anal. Calcd. for  $C_{12}H_{14}O_{11}(CH_3CO)_8$ : C, 49.56; H, 5.63; CH<sub>3</sub>CO, 11.79 cc. of 0.1 N sodium hydroxide per 100 mg.; mol. wt., 678.6. Found: C, 49.74; H, 5.67; CH<sub>3</sub>CO, 11.86 cc.; mol. wt. (Rast), 680.

Since methylation studies<sup>2,3</sup> have demonstrated that the polysaccharide employed in this work is built up of  $\alpha$ -D-glucopyranose units linked in the 1,6-position, it follows that the disaccharide isolated must be  $6-\alpha$ -D-glucopyranosido- $\beta$ -D-glucose octaacetate. Its determined rotation is in agreement with the value predictable by application of the isorotation rules of Hudson. The same acetvlation and chromatographic procedure was applied to the sirup soluble in 80% ethanol that was obtained essentially according to the procedure of Örtenblad and Myrbäck<sup>4</sup> from the enzymic hydrolyzate of amylopectin (waxy maize) after removal of most of the maltose and D-glucose by yeast fermentation. In this case the lowest zone on the chromatogram was composed of  $\beta$ -maltose octaacetate (m.p. 159-160°, mixed melting point 159–160° unchanged;  $[\alpha]^{25}D + 62^{\circ}, c 1.1,$  chloroform) and from the zone immediately above this

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there was obtained by acetone elution and crystallization from ethanol, a substance crystallizing in tufts of fine needles; m. p. 134–136°, mixed melting point with  $6 \cdot \alpha$ -D-glucopyranosido- $\beta$ -Dglucose octaacetate 120–125°,  $[\alpha]^{24}D + 86°$  (*c* 1.6, chloroform). This material is the hendecaacetate of a trisaccharide and its structure is under further investigation.

Anal. Calcd. for  $C_{18}H_{21}O_{16}(CH_3CO)_{11}$ : C, 49.69; H, 5.63; mol. wt., 966.8. Found: C, 49.46; H, 557; mol. wt. (Rast), 971.

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## THE ANTIBACTERIAL ACTIVITY OF *p*-AMINOBENZENEPHOSPHONOUS ACID

Sir:

It has been shown by Kuhn, Möller and Wendt<sup>1</sup> that phosphanilic acid  $(p-H_2NC_6H_4PO(OH)_2)$  has a slight antibacterial action, similar in nature to that of the sulfonamides in being antagonized by p-aminobenzoic acid. Proceeding on the assumption that the strength of the sulfonamide–enzyme complex is increased with decreasing acidity,<sup>2</sup> we have prepared p-aminobenzenephosphonous acid,  $p-H_2NC_6H_4P(OH)_2$ , by the procedure

$$C_{6}H_{3}Br + PCl_{3} \xrightarrow{AlCl_{3}} p - BrC_{6}H_{4}PCl_{2} \xrightarrow{H_{2}O} p - BrC_{6}H_{4}P(OH)_{2} \quad (1)$$

$$p - BrC_{6}H_{4}P(OH)_{2} + NH_{4}OH \xrightarrow{Cu_{2}O} p - H_{2}NC_{6}H_{4}P(OH)_{2} \quad (2)$$

The first step, the preparation of p-bromobenzenephosphonous acid, has been carried out previously.<sup>3</sup> The second step, the synthesis of the amino compound, is similar to that used by Bauer<sup>4</sup> to prepare phosphanilic acid.

The product obtained has these properties: m.p. 169°; equivalent weight, calculated 157.1, found 158.0; analysis, calculated P 19.74%, found P 19.66%; solubility in water at 0° about 5%; pK, 3.68.

The activity of p-aminobenzenephosphonous acid was tested against *E. coli* and found to be slightly less than that of sulfanilamide. This antibacterial action was antagonized by p-aminobenzoic acid at concentrations approximately equal to those necessary to counteract sulfanilamide. Further tests on this substance and related compounds are in progress.

Department	$\mathbf{OF}$	CHEMISTRY
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Northwestern University I. M. Klotz Evanston, Illinois R. T. Morrison Received January 16, 1947

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<sup>(4)</sup> H. Baner, This JOURNAL, 63, 2137 (1941).