and the reaction vessel is evacuated continuously in the initial stages of the reaction by means of a rotary pump. Towards the end of the reaction, a diffusion pump is coupled to the system as well. Completion of the reaction is accompanied by a marked improvement in the vacuum obtainable.

The period of heating required appears to depend on the precise nature of the charcoal and varies from 1 to 2 h. No advantage seems to be gained by crushing the charcoal, and large pieces appear to react just as rapidly as a coarse powder.

Further treatment of the sample was identical to that used by BARKER and conversion was found to be better than 95%, thus eliminating any possibility of fractionation. As an additional check on the method, the count of a randomly chosen sample was compared with that of an identical sample prepared by the BARKER method and found to be 13.54 ± 0.06 as opposed to 13.64 ± 0.07. Using this new method it is possible to get a sample of pure dry acetylene into the counter in less than 5 h. Since the apparatus is essentially the same as that used by BARKER, samples other than charcoal, such as wood, shell, or bone, as well as the N.B.S. oxalic acid standard, can still be prepared without any inconvenience by the method of BARKER[4].

Résumé. On décrit ici une nouvelle méthode pour convertir, dans un four d'acier à 800°C, le charbon de bois directement en carbure de lithium produisant de l'acétylène pour déterminer le carbone radioactif. La méthode offre une notable économie de temps pour la préparation des échantillons et le produit de conversion atteint le 95% ou davantage.

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[4] I would like to acknowledge the help of Miss E. A. HEGGARTY, who has carried out most of the work of preparing and counting the samples.

Crystalline β-D-Ribofuranosyl-β-D-ribofuranoside: A New Diriboside Synthesis

ZAMENHOF et al.[1] have proposed that the type specific substance of *Hemophilous influenzae* b is a polyribophosphate polymer whose ribose units are present as disaccharides with the structure β-D-ribofuranosyl-β-D-ribofuranoside (III). While a chemical synthesis[2] of this substance *via* a Koenigs–Knorr reaction gave a well characterized hexabenzoate derivative II, hydrolysis provided the free sugar only as a non-crystalline substance.

In connection with another project in these laboratories, it was of interest to learn whether 1-*o*-acetyl 2,3,5-tribenzoylribofuranoside (I)[3], when treated in benzene with boron trifluoride etherate could yield any C_1-phenylated ribose[4].

While none of this substance was isolated, a new crystalline product, m.p. 143–144°, $[\alpha]_D + 35.2°$, C = 0.475 (chloroform) could be obtained[5]. Analysis and further investigation suggested that this substance was identical with the hexabenzoate II and comparison with the reported physical constants upheld this view[6].

Hydrolysis of II was effected with fresh sodium methoxide in anhydrous methanol. Following evaporation, the residue was dissolved in water and percolated through Dowex-50-pyridinium salt to yield III, crystallized from absolute ethanol m.p. 158–160°, $[\alpha]_D - 102°$, C = 0.47 (water), $C_{10}H_{18}O_9$, Found: C, 42.74; H, 6.38; O, 50.71. Direct comparison of this crystalline substance with the amorphous natural product[1] by means of paper chromatography showed identical mobilities[7].

Examination of the n.m.r. spectra of II and III has allowed an unambiguous decision in favour of the β,β′-configuration previously suggested[2] on the basis of rotational data. In deuterium oxide for III and deuterochloroform for II, the anomeric protons appear in sharp singlets, δ 5.68 for III and δ 5.73 for II[8], thereby demonstrating that the coupling constants with the adjacent protons on C_2 and C_2' have the value J = 0. Reference to the Karplus equation and modification thereof[9] indicates that coupling constants with J values equal to 0 occur only when the angle between the protons in question is in the neighborhood of 90°. Since molecular models of II and III show that such angles may be achieved only when the anomeric protons are in the α-configuration (i.e trans to those at C_2 and C_2'), it follows that the sugars must be joined by β,β′ linkages[10].

Zusammenfassung. Behandlung von 1-*o*-Acetyl-2,3,5-tri-*o*-benzoyl-β-D-ribofuranosid mit Bortrifluorid-ätherat gibt ein Diribosid-hexabenzoat, welches zu reinem, kristallinem β-D-Ribofuranosyl-β-D-ribofuranosid hydrolysiert werden kann.

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[2] E. ROSENBERG and S. ZAMENHOF, J. biol. Chem. *237*, 1040 (1962).
[3] E. F. RECONDO and H. RINDERKNECHT, Helv. chim. Acta *42*, 1171 (1959).
[4] Friedel-Crafts alkylations employing sugar halides and anomeric acetates have been reported. See, W. A. BONNER, Advanc. carbohyd. Chem. *6*, 251 (1951).
[5] Later, it was shown that the use of methylene chloride instead of benzene provided more reproducible yields.
[6] For a mineral acid catalyzed dipentose synthesis employing trimethyllyxofuranose, see H. G. BOTTS, E. L. HIRST, and J. A. B. SMITH, J. chem. Soc. *1930*, 658.
[7] We are grateful to Dr. ZAMENHOF for advising us of his results prior to publication and for making the direct comparison reported in solvent systems A and C (see ref. [1]).
[8] The spectra were recorded on a Varian A-60 spectrometer. Tetramethylsilane was used as a standard externally for III and internally for II.
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[10] The author is indebted to Dr. A. WALSER for discussions regarding the n.m.r. spectra.
[11] On leave at the Eidg. Technische Hochschule, Zürich (Switzerland).