

values at 10 and 25 mA cm<sup>-2</sup> but  $D_H$  apparently depends on the applied current, presumably because of a dependence on the bulk concentration of hydrogen in the metal (ca. 10<sup>-6</sup> g-atom cm<sup>-3</sup>). Gileadi, Fullenwider, and Bockris (6) observed an increasing  $D_H$  in successive permeations through platinum. Their suggestion of hydrogen concentrated in regions of strain being less free to diffuse may apply here also. However, the formula  $PbH_{0.00002}$  differs by orders of magnitude from Wells and Roberts'  $PbH_{0.19}$  achieved by exposure of evaporated lead films to atomic hydrogen (7). Hence the lead is unlikely to have become saturated in the present work.

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1. D. P. SMITH. Hydrogen in metals, Univ. of Chicago Press. Chicago, Ill. 1948. p. 286.
2. H. R. HEATH. Brit. J. Appl. Phys. 3, 13 (1952).
3. H. B. WAHLIN and V. O. NAUMANN. J. Appl. Phys. 24, 42 (1953).
4. M. A. V. DEVANATHAN and Z. O. J. STACHURSKI. Proc. Roy. Soc. London, Ser. A, 270, 90 (1962).
5. J. MCBREEN, L. NANIS, and W. BECK. J. Electrochem. Soc. 113, 1218 (1966).
6. E. GILEADI, M. A. FULLENWIDER, and J. O'M. BOCKRIS. J. Electrochem. Soc. 113, 926 (1966).
7. B. R. WELLS and M. W. ROBERTS. Proc. Chem. Soc. 173 (1964).

## Total synthesis of DL-glucose

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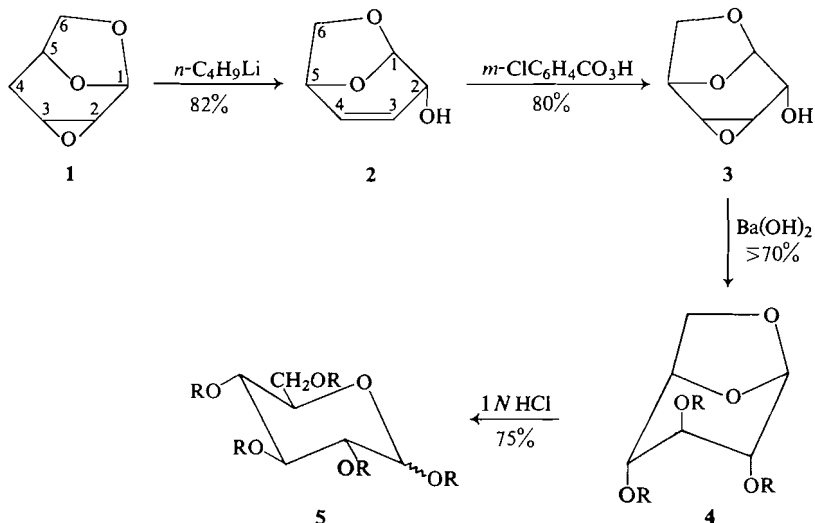
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The stereoselective synthesis of DL-glucose has been accomplished in 34% overall yield starting from 1,6:2,3-dianhydro-4-deoxy-β-DL-ribo-hexopyranose (1), a compound obtainable from the Diels-Alder condensation of acrolein.

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The compound 1,6:2,3-dianhydro-4-deoxy-β-DL-ribo-hexopyranose (1), obtained from acrolein dimer according to published directions (1), was converted into 1,6-anhydro-3,4-dideoxy-β-DL-erythro-hex-3-enopyranose (2) with *n*-butyl-

lithium at room temperature following the known reaction of oxiranes with the organolithium reagent (2). Treatment of 2 with *m*-chloroperoxybenzoic acid in methylene chloride at room temperature for 24 h gave the expected 1,6:3,4-



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dianhydro- $\beta$ -DL-*allo*-hexopyranose (3). The reaction with 10% aqueous barium hydroxide at 80° for 16 h under nitrogen (3) converted 3 into 1,6-anhydro- $\beta$ -DL-*gluco*-hexopyranose (4, R = H), which yielded the triacetate 4 (R = CH<sub>3</sub>CO) with pyridine and acetic anhydride. Hydrolysis of 4 (R = H) with 1 *N* aqueous hydrochloric acid gave  $\alpha,\beta$ -DL-glucose (5, R = H) as a solid which shrinks at 44° but melts at 113–115°. <sup>2</sup> Acetic anhydride and pyridine converted 5 (R = H) into its pentacetate 5 (R = CH<sub>3</sub>CO) which was an approximately 1:1 mixture of the  $\alpha$  and  $\beta$  anomers.

Elemental analyses all agreed very well with the calculated values. The infrared (i.r.) and proton magnetic resonance (p.m.r.) spectra of

all of these compounds supported the assigned structures. Furthermore the i.r. and p.m.r. spectra of compounds 4 (R = CH<sub>3</sub>CO) and 5 (R = H and CH<sub>3</sub>CO) were identical with those of authentic 2,3,4-tri-*O*-acetyl-1,6-anhydro- $\beta$ -D-glucopyranose,  $\alpha,\beta$ -D-glucose, and  $\alpha,\beta$ -D-glucose pentacetate respectively.

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1. F. SWEET and R. K. BROWN. *Can. J. Chem.* **46**, 2289 (1968).
2. H. NOZAKI, T. MORI, and R. NOYORI. *Tetrahedron*, **22**, 1207 (1966).
3. G. CHARALAMBOUS and E. PERCIVAL. *J. Chem. Soc.* 2443 (1954).
4. M. L. WOLFROM and B. H. WOOD. *J. Amer. Chem. Soc.* **71**, 3175 (1949).

<sup>2</sup>Reference 4 reports the melting point of a 1:1 mixture of D- and L-glucose as 112–113.5°.