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Received January 7, 1974.

Molecular Structure of Methyl 6-*O*-Acetyl-β-D-glucopyranoside

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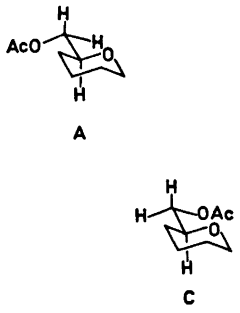
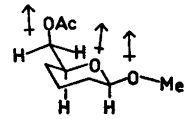
In our attempts to correlate the circular dichroism of glycoside acetates with molecular geometry, we find that the single, negative CD band observed for methyl 6-*O*-acetyl-β-D-glucopyranoside is best explained by assuming that rotamer C predominates in ethanol solution.

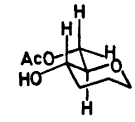


This contrasts with the results obtained for the corresponding α-anomer, for which a double CD band was observed, and which led to the suggestion that both rotamers B and C contributed to the observed CD. We suggested that for the β-anomer an unfavourable dipolar interaction for rotamer B would lead to the presence of



rotamer C only. Since this interaction is absent in the corresponding α-anomer, both rotamers B and C can be present.[1] Lemieux and coworkers have suggested, on the basis of NMR studies, as well as from considerations of optical rotation, that for D-*erythro*-hexopyranosides, rotamer C predominates over A and B.[2,3]

We have previously reported an X-ray crystallographic study on methyl 6-*O*-acetyl-β-D-galactopyranoside.[4] This showed that in the crystalline state rotamer A predominated. In the D-*gluco* series, however, this rotamer is unimportant, due to an unfavourable 1,3 interaction.



The present study, summarized in Fig. 1 and Table 1 establishes that for methyl 6-*O*-acetyl-β-D-glucopyranoside the conformation in the crystalline state corresponds to rotamer C, in agreement with the interpretation of the results from the CD investigation. The compound crystallized in space group $P2_1$, $a = 10.201$, $b = 7.239$, $c = 7.863$, $Z = 2$. The X-ray data were obtained on a Philips PW 1100 computer-controlled single-crystal diffractometer with graphite monochromatized Cu$K\alpha$ radiation. The phase

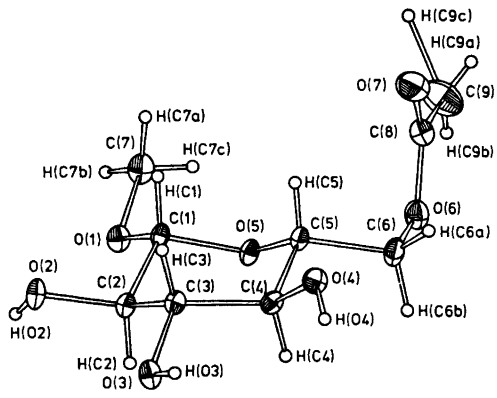


Fig. 1. Molecular structure of methyl 6-*O*-acetyl-β-D-glucopyranoside.

Table 1. Intramolecular nonhydrogen bond distances (Å) and angles(°). Estimated standard deviations are given in parentheses.

C(1)–C(2)	1.522(4)	C(1)–C(2)–C(3)	108.2(3)
C(2)–C(3)	1.522(5)	C(2)–C(3)–C(4)	112.4(3)
C(3)–C(4)	1.514(5)	C(3)–C(4)–C(5)	111.7(3)
C(4)–C(5)	1.534(4)	C(4)–C(5)–O(5)	109.4(3)
C(5)–C(6)	1.522(6)	C(5)–O(5)–C(1)	110.8(2)
C(8)–C(9)	1.538(8)	O(5)–C(1)–C(2)	107.9(3)
C(1)–O(1)	1.384(4)	C(4)–C(5)–C(6)	110.8(3)
C(7)–O(1)	1.428(5)	O(5)–C(5)–C(6)	107.0(3)
C(2)–O(2)	1.425(5)	C(5)–C(6)–O(6)	113.2(3)
C(3)–O(3)	1.423(4)	C(1)–O(1)–C(7)	112.4(3)
C(4)–O(4)	1.421(4)	C(6)–O(6)–C(8)	115.9(4)
C(1)–O(5)	1.429(4)	O(6)–C(8)–C(9)	111.5(4)
C(5)–O(5)	1.429(4)	O(6)–C(8)–O(7)	126.2(5)
C(6)–O(6)	1.423(5)	C(9)–C(8)–O(7)	122.3(5)
C(8)–O(6)	1.295(5)	O(1)–C(1)–O(5)	106.9(3)
C(8)–O(7)	1.184(6)	O(1)–C(1)–C(2)	109.4(3)
		C(1)–C(2)–O(2)	111.2(3)
		C(3)–C(2)–O(2)	108.1(3)
		C(2)–C(3)–O(3)	107.8(3)
		C(4)–C(3)–O(3)	110.2(3)
		C(3)–C(4)–O(4)	110.6(3)
		C(5)–C(4)–O(4)	108.0(3)

determinations were carried out by a computerized application of direct methods using the weighted phase-sum formula described by Norrestam.[6] Several cycles of full-matrix least-squares refinement (anisotropic nonhydrogen and fixed isotropic hydrogen temperature parameters) gave an R-value of 0.046. The molecular structure is shown in Fig. 1. Intramolecular distances and angles are listed in Table 1. Full details of the X-ray diffraction investigation will be published elsewhere.[7]

Acknowledgements. We are indebted to Professor Peder Kierkegaard and to Professor Bengt Lindberg for their interest. The work received financial support from Riksbankens Jubileumsfond, Hierta-Retzius Stipendiefond and Statens Naturvetenskapliga Forskningsråd.

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Received December 21, 1973.

Synthesis of Asterosterol, a Novel C_{26} Marine Sterol

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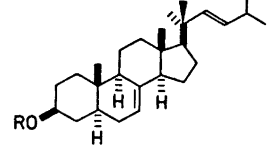
Recently Kobayashi *et al.*[1,2] suggested structure *1a* for a new marine C_{26} sterol, asterosterol, isolated from several asteroids [3] and stated [1] that they had synthesized a 22-*cis* and -*trans* mixture of 24-*nor*-cholesta-7,22-dien-3β-ol, resistant to separation. Through the investigation of the sterol components of the marine sponge *Halicondria panicea* we have now found the same sterol present as a minor component.



1a : R = H
1b : R = Ac

Due to the uncertainties associated with the stereochemistry at C-20 as well as the biogenetic novelty of the sidechain structure a final proof