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Structure of Methyl 3-*C*-(Acetoxymethyl)-1,4-di-*O*-acetyl-α-*erythro*-pentofuranoside

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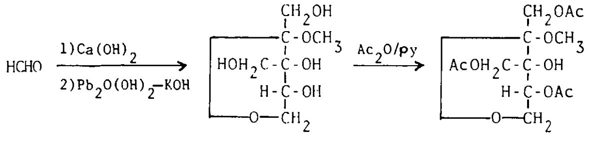
Abstract. $C_{13}H_{20}O_9$, $M_r = 320{\cdot}3$, orthorhombic, *Pbca*, $a = 21{\cdot}614\,(7)$, $b = 17{\cdot}239\,(5)$, $c = 8{\cdot}518\,(4)$ Å, $V = 3174$ Å^3, $Z = 8$, $D_x = 1{\cdot}34$ g cm^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}5418$ Å, $\mu = 9{\cdot}4$ cm^{-1}, $F(000) = 1360$, room temperature, $R = 0{\cdot}076$ for 1668 observed reflections. The five-membered ring is puckered at C(3), and its conformation is *endo*. The hydroxyl and methoxy groups occupy quasi-axial positions. There is an intermolecular hydrogen bond of 3·022 (6) Å between the hydroxyl group and one of the acetyl groups of a molecule related by the *c*-glide plane.

Introduction. The reaction of formaldehyde in the presence of base catalysts such as $Ca(OH)_2$, known as the formose reaction, gives a complex mixture of saccharides (Mizuno & Weiss, 1974). The reaction mechanism and the effect of reaction conditions on the selective formation of products have been studied extensively (Shigemasa, Nagae, Sakazawa, Nakashima & Matsuura, 1978; Shigemasa, Akagi, Waki & Nakashima, 1981). Addition of $Pb_2O(OH)_2$ and adjustment of the pH of the reaction mixture to 10·0 at the end of the induction period resulted in selective synthesis of 3-*C*-(hydroxymethyl)pentofuranose (1) (Shigemasa, Hamada, Hirabayashi, Waki, Nakashima, Harada, Takeda & Suzuki, 1981). The product was isolated as the triacetate (2) by silica-gel chromatography. Although the structures of (1) and (2) were examined by spectroscopic and chemical means, ambiguity concerning their configurations remained. The crystallographic analysis of (2) was carried out to establish the configuration of the molecule.



Experimental. Colourless prisms of (2) (from benzene/cyclohexane solution), 0·1 × 0·15 × 0·3 mm. Rigaku diffractometer (Katsube, 1982), Ni-filtered Cu $K\alpha$ radiation. Cell parameters determined by least squares from 2θ values measured for 18 reflections with $14{\cdot}9 < \theta < 15{\cdot}6°$. Integrated intensities recorded by θ–2θ scan, scan speed 4° min^{-1} (θ), scan range $1{\cdot}0° + 0{\cdot}2° \tan\theta$, 5 s stationary background counts. $2\theta_{\max} = 120°$ (h: 0→24, k: 0→19, l: 0→9). Two reference reflections monitored every 100 reflections showed no significant variation in intensity during data collection. 1960 independent reflections measured, 1668

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reflections with $I > 2\sigma(I)$ used for refinement. No absorption correction. Five strongest reflections corrected for extinction ($g = 4{\cdot}2 \times 10^{-7}$). Structure solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971). All H atoms located on a difference map. Block-diagonal least-squares refinement (Ashida, 1973) with anisotropic temperature factors for C and O atoms and isotropic ones for H atoms converged at $R = 0{\cdot}076$, $wR = 0{\cdot}089$. Max. Δ/σ 0·78 for non-H atoms. Max. $\Delta\rho$ 0·36 e Å^{-3}. $\sum w(|F_o| - |F_c|)^2$ minimized with weight $w = 1{\cdot}0$ for $0 < F_o \leq 32$ and $w = [1{\cdot}0 + 0{\cdot}05(F_o - 32)]^{-1}$ for $F_o > 32$. Relatively high R value reflected rather poor quality of intensity data due to small size of the crystal. Scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on HITAC M-150 at the Tottori University Computing Center.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i \sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
C(1)	0·7558 (2)	0·6143 (3)	0·1280 (8)	4·6
C(2)	0·6901 (2)	0·5957 (3)	0·0799 (7)	4·1
C(3)	0·6380 (2)	0·6292 (3)	0·1855 (6)	3·3
C(4)	0·5874 (2)	0·5687 (3)	0·1508 (6)	3·9
C(5)	0·6240 (3)	0·4923 (3)	0·1444 (9)	5·2
C(6)	0·6227 (2)	0·7123 (3)	0·1405 (7)	4·2
C(7)	0·4839 (3)	0·5757 (4)	0·2459 (8)	5·3
C(8)	0·4436 (3)	0·5621 (4)	0·3853 (9)	6·8
C(9)	0·5423 (2)	0·8017 (3)	0·1926 (7)	4·6
C(10)	0·4832 (3)	0·8115 (4)	0·2820 (9)	7·2
C(11)	0·8093 (2)	0·7206 (3)	0·0087 (7)	4·8
C(12)	0·8139 (3)	0·8067 (4)	0·0088 (9)	6·1
C(13)	0·7048 (3)	0·5760 (4)	−0·1976 (8)	6·5
O(1)	0·6856 (2)	0·5143 (2)	0·1010 (4)	4·8
O(2)	0·5430 (1)	0·5626 (2)	0·2769 (4)	4·2
O(3)	0·6577 (2)	0·6240 (2)	0·3441 (4)	4·2
O(4)	0·6748 (2)	0·6183 (2)	−0·0735 (4)	4·4
O(5)	0·4669 (2)	0·5994 (4)	0·1203 (6)	9·1
O(6)	0·5668 (2)	0·7318 (2)	0·2269 (5)	4·3
O(7)	0·5649 (2)	0·8459 (2)	0·1025 (6)	6·3
O(8)	0·7661 (2)	0·6961 (2)	0·1105 (5)	5·0
O(9)	0·8389 (2)	0·6775 (3)	−0·0728 (6)	7·5

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·514 (9)	C(1)—O(8)	1·435 (8)
C(2)—C(3)	1·552 (8)	C(2)—O(1)	1·418 (8)
C(2)—O(4)	1·402 (7)	C(3)—C(4)	1·539 (8)
C(3)—C(6)	1·519 (8)	C(3)—O(3)	1·419 (6)
C(4)—C(5)	1·537 (9)	C(4)—O(2)	1·444 (7)
C(5)—O(1)	1·433 (9)	C(6)—O(6)	1·455 (7)
C(7)—C(8)	1·492 (10)	C(7)—O(2)	1·325 (7)
C(7)—O(5)	1·203 (9)	C(9)—C(10)	1·496 (10)
C(9)—O(6)	1·347 (7)	C(9)—O(7)	1·187 (8)
C(11)—C(12)	1·488 (10)	C(11)—O(8)	1·343 (8)
C(11)—O(9)	1·202 (8)	C(13)—O(4)	1·437 (8)

C(2)—C(1)—O(8)	108·9 (5)	C(1)—C(2)—C(3)	116·4 (5)
C(1)—C(2)—O(1)	103·8 (5)	C(1)—C(2)—O(4)	114·5 (5)
C(3)—C(2)—O(1)	104·1 (5)	C(3)—C(2)—O(4)	105·5 (5)
O(1)—C(2)—O(4)	112·1 (5)	C(2)—C(3)—C(4)	98·8 (4)
C(2)—C(3)—C(6)	111·2 (5)	C(2)—C(3)—O(3)	108·2 (4)
C(4)—C(3)—C(6)	115·8 (5)	C(4)—C(3)—O(3)	110·7 (4)
C(6)—C(3)—O(3)	111·4 (4)	C(3)—C(4)—C(5)	102·8 (5)
C(3)—C(4)—O(2)	112·3 (4)	C(5)—C(4)—O(2)	107·7 (5)
C(4)—C(5)—O(1)	105·1 (5)	C(3)—C(6)—O(6)	105·7 (5)
C(8)—C(7)—O(2)	112·2 (6)	C(8)—C(7)—O(5)	125·7 (7)
O(2)—C(7)—O(5)	122·1 (6)	C(10)—C(9)—O(6)	109·1 (5)
C(10)—C(9)—O(7)	127·4 (6)	O(6)—C(9)—O(7)	123·5 (6)
C(12)—C(11)—O(8)	111·1 (6)	C(12)—C(11)—O(9)	125·6 (6)
O(8)—C(11)—O(9)	123·2 (6)	C(2)—O(1)—C(5)	111·0 (5)
C(4)—O(2)—C(7)	118·7 (4)	C(2)—O(4)—C(13)	116·0 (5)
C(6)—O(6)—C(9)	115·1 (4)	C(1)—O(8)—C(11)	118·9 (5)

Discussion. The atomic parameters are listed in Table 1.* The bond lengths and angles are given in Table 2. Fig. 1 shows a perspective view drawn by *DCMS*-3 (Takenaka, 1977) and atom numbering used in this paper. The present crystallographic analysis has established the structure of (2) as methyl 3-*C*-(acetoxymethyl)-1,4-di-*O*-acetyl-α-*erythro*-pentofuranoside. The five-membered ring is puckered at C(3); C(3) is displaced by 0·66 Å from the mean plane of the other four atoms. The deviations of the four atoms from the plane are ±0·002 Å. Since C(3) and C(1) deviate to the same side of the plane, the pucker is *endo*. This conformation is one of the conformations frequently observed in nucleosides and nucleotides (Altona & Sundaralingam, 1972). The hydroxyl and methoxy groups occupy quasi-axial positions. There is an intermolecular hydrogen bond between the hydroxyl group and one of the acetyl groups of a molecule related by the *c*-glide plane. The distances O(3)···O(7) and H[O(3)]···O(7) are 3·022 (6) and 1·98 (7) Å respectively, and the angle O(3)—H[O(3)]···O(7) is 154 (5)°.

We thank Shigeo Fujii and Mikio Hirabayashi for technical assistance.

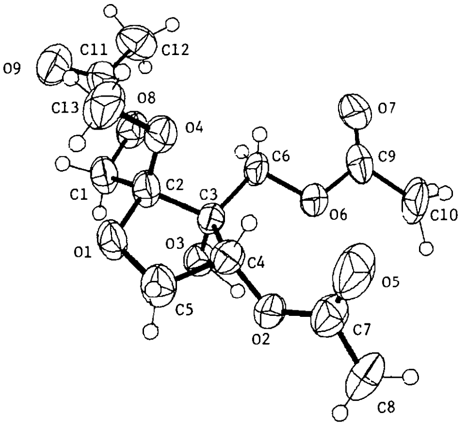

Fig. 1. A perspective view and atom numbering of (2).

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* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42417 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Carbocyclic Oxapenam Analogue

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Abstract. 7-*exo*-Succinimido-2-oxabicyclo[3.2.0]heptan-6-*exo*-ol, $C_{10}H_{13}NO_4$, $M_r = 211.2$, orthorhombic, $P2_12_12_1$, $a = 9.748\,(2)$, $b = 9.918\,(3)$, $c = 10.136\,(2)$ Å, $V = 980$ Å^3, $Z = 4$, $D_x = 1.43$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1.5418$ Å, $\mu = 0.947$ mm^{-1}, $F(000) = 448$, $T = 293$ K, $R = 0.0685$ for 894 unique observed reflections. The cyclobutanone and furan rings of the oxaheptan-6-*exo*-ol moiety make an angle of 118.2 (7)° with normal bond lengths and angles but with an unexpected stereochemistry. The OH group forms a hydrogen bond to N with a length of 3.06 (1) Å.

Introduction. This determination is the third of a series of structure determinations of carbocyclic analogues of penicillin derivatives in which the β-lactam ring is replaced by cyclobutanone (Sheldrick, Akrigg, Page & Agathocleous, 1985). In the previous structures the substituents on the cyclobutanone ring all had the *cis* configuration.

Experimental. Material prepared by M. I. Page and D. Agathocleous (Agathocleous, Buckwell, Proctor & Page, 1985), crystallized from ethyl acetate. Tabular crystal, 0.42 × 0.22 × 0.11 mm. Enraf–Nonius CAD-4*F* diffractometer, Cu $K\alpha$ radiation. No correction for absorption. $2\theta_{\max} = 140°$, $\pm hk\pm l$; 2824 reflections measured, 1087 considered unobserved $[F < 3\sigma(F)]$. Check reflection 403, average count 324.5, calculated σ (of the distribution) = 17.2 (5.3%). Cell dimensions from θ measurements of 24 reflections in the range $42 \le 2\theta \le 68°$. Data merged using *SHELX*76 (Sheldrick, 1976) giving 894 unique reflections, $R_{\rm int} = 0.05$, index range h 0 to 11, k 0 to 12, l 0 to 12. *MULTAN*80 (Main *et al.*, 1980) used to solve structure, by direct methods. Least-squares refinement with *SHELX*76; positional parameters of all atoms and anisotropic thermal parameters for non-H atoms refined; $\sum w(\Delta F)^2$ minimized with $w = 1/[\sigma^2(F) + 0.000968F^2]$. H atoms from difference Fourier syntheses, included in refinement with fixed $U_{\rm iso}$. In final cycle max. $\Delta/\sigma = 0.107$ (non-H) and 0.168 (H), average = 0.029. $\Delta\rho$ in final difference Fourier map within +0.44 and −0.23 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). $R = 0.0685$, $wR = 0.0759$ for 894 observed reflections. Figures drawn with *PLUTO*78 (Motherwell & Clegg, 1978).

Discussion. The molecule and numbering scheme are shown in Fig. 1 and a stereoview of the cell packing in Fig. 2. Atom coordinates and equivalent isotropic temperature factors are in Table 1* and bond lengths and angles in Table 2.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters, torsion angles and best planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42455 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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