

Related literature. The synthesis of the title compound and its conformational analysis in solution were reported by Takeda, Akimoto & Kyogoku (1982).

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Structure of 1,6-Anhydro-4-carboxymethyl-2,4-dideoxy-2-fluoro- β -D-*allo*-hexopyranose- γ -lactone

BY PHILIP J. COX*

School of Pharmacy, Robert Gordon's Institute of Technology, Schoolhill, Aberdeen AB9 1FR, Scotland

AND GEORGE A. SIM

Chemistry Department, University of Glasgow, Glasgow G12 8QQ, Scotland

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Abstract. C₈H₉FO₄, $M_r = 188.16$, orthorhombic, $P2_12_12_1$, $a = 7.5071$ (10), $b = 8.9402$ (8), $c = 11.7375$ (20) Å, $V = 787.8$ Å³, $Z = 4$, $D_x = 1.57$ Mg m⁻³, Cu $K\alpha$, $\lambda = 1.5418$ Å, $\mu = 1.119$ mm⁻¹, $F(000) = 392$, room temperature, $R = 0.039$ for 1159 observed reflexions. The anhydro and lactone rings are on opposite sides of the pyranose ring such that the molecule has an overall chair conformation. The dioxolane and lactone rings adopt envelope conformations with O(2) and C(4) respectively at the flap. By comparison with cyclohexane, O(2) shows enhanced puckering and C(3) is flattened. The torsion angles about the bonds to O(2) are $ca |76^\circ|$ and those about bonds to C(3) are $ca |36^\circ|$. Therefore, the F–C(2)–C(3)–O(3) torsion angle is positive, 37.4 (2)°, but considerably less than 60° . Intramolecular non-bonded distances of interest with respect to NMR long-range coupling are F...C(4) 3.129 (3), F...C(7) 3.096 (3), F...H(7A) 2.755 (3) Å and F...H(7B) 4.084 (3) Å.

Experimental. Colourless crystal, dimensions 0.10 × 0.20 × 0.10 mm. Enraf-Nonius CAD-4 diffractometer, graphite monochromator, Cu $K\alpha$ radiation. Cell dimensions from setting angles of 25 independent reflexions with $\theta \approx 22^\circ$. 885 unique intensities measured, with $2\theta < 70^\circ$, as θ – 2θ scans. Range of hkl 0–9, 0–10, 0–14, 811 independent reflexions with $I > 2.5\sigma(I)$. Two reference reflexions monitored period-

ically showed no significant variation in intensity. No absorption correction. Structure was determined with *MITHRIL* (Gilmore, 1984) and H atoms located in difference Fourier maps with *SHELX* (Sheldrick, 1976). Full-matrix least-squares calculations on F with anisotropic thermal parameters for C, F and O atoms and isotropic thermal parameters for H atoms converged at R 0.039, wR 0.042, $\Delta/\sigma < 0.03$, $w = 49.3/\sigma^2(F_o)$. Final $\Delta\rho$ max. 0.07, min. 0.14 e Å⁻³. Scattering factors from *International Tables for X-ray Crystallography* (1974). Molecular diagrams and geometries were generated by the *GX* package (Mallinson & Muir, 1985) and all computer programs were run on a Honeywell CP6 mainframe. Atomic coordinates

Table 1. Fractional atomic coordinates ($\times 10^4$) with *e.s.d.*'s in parentheses and equivalent values of the anisotropic temperature factor coefficients ($\text{Å}^2 \times 10^3$)

	x	y	z	U_{eq}
F	–0.9183 (2)	–0.4847 (2)	–0.7736 (1)	0.073
O(1)	–1.2063 (2)	–0.5111 (3)	–0.5319 (1)	0.066
O(2)	–0.9890 (3)	–0.6677 (2)	–0.5943 (2)	0.063
O(3)	–0.7552 (2)	–0.2816 (2)	–0.6524 (2)	0.055
O(4)	–0.4725 (2)	–0.3205 (3)	–0.6959 (1)	0.077
C(1)	–1.1153 (3)	–0.5611 (3)	–0.6308 (2)	0.061
C(2)	–1.0204 (3)	–0.4301 (3)	–0.6834 (2)	0.055
C(3)	–0.8986 (3)	–0.3588 (2)	–0.5961 (2)	0.044
C(4)	–0.8069 (3)	–0.4704 (2)	–0.5170 (2)	0.043
C(5)	–0.9258 (3)	–0.6025 (2)	–0.4899 (2)	0.049
C(6)	–1.1008 (3)	–0.5545 (3)	–0.4355 (2)	0.053
C(7)	–0.6361 (3)	–0.5043 (3)	–0.5818 (3)	0.056
C(8)	–0.6045 (3)	–0.3649 (3)	–0.6497 (2)	0.053

$$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33}).$$

* Director, Molecular Structures Unit, Robert Gordon's Institute of Technology.

Table 2. Bond lengths (Å) and valency angles (°) with e.s.d.'s in parentheses

F—C(2)	1.396 (3)	O(1)—C(1)	1.419 (4)
O(1)—C(6)	1.435 (3)	O(2)—C(1)	1.411 (4)
O(2)—C(5)	1.438 (3)	O(3)—C(3)	1.439 (3)
O(3)—C(8)	1.355 (3)	O(4)—C(8)	1.198 (3)
C(1)—C(2)	1.503 (4)	C(2)—C(3)	1.514 (4)
C(3)—C(4)	1.527 (4)	C(4)—C(5)	1.515 (4)
C(4)—C(7)	1.521 (4)	C(5)—C(6)	1.522 (4)
C(7)—C(8)	1.498 (4)		
C(1)—O(1)—C(6)	107.1 (2)	C(1)—O(2)—C(5)	101.9 (2)
C(3)—O(3)—C(8)	110.5 (2)	O(1)—C(1)—O(2)	106.8 (3)
O(1)—C(1)—C(2)	108.6 (3)	O(2)—C(1)—C(2)	109.4 (2)
F—C(2)—C(1)	107.4 (3)	F—C(2)—C(3)	109.2 (2)
C(1)—C(2)—C(3)	109.6 (2)	O(3)—C(3)—C(2)	110.0 (2)
O(3)—C(3)—C(4)	104.8 (2)	C(2)—C(3)—C(4)	114.1 (2)
C(3)—C(4)—C(5)	111.8 (2)	C(3)—C(4)—C(7)	101.9 (2)
C(5)—C(4)—C(7)	116.5 (2)	O(2)—C(5)—C(4)	109.3 (2)
O(2)—C(5)—C(6)	100.8 (2)	C(4)—C(5)—C(6)	112.1 (2)
O(1)—C(6)—C(5)	102.8 (2)	C(4)—C(7)—C(8)	103.5 (2)
O(3)—C(8)—O(4)	119.9 (3)	O(3)—C(8)—C(7)	109.7 (2)
O(4)—C(8)—C(7)	130.4 (3)		

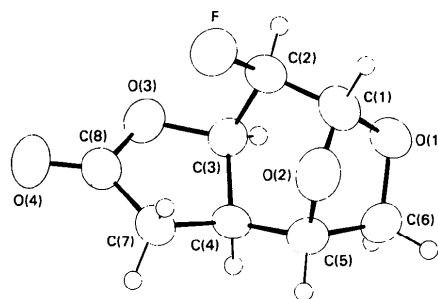


Fig. 1. The atomic arrangement.

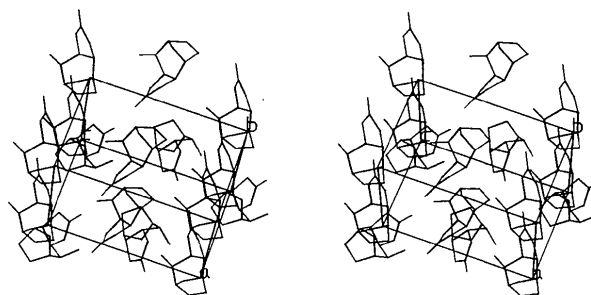


Fig. 2. Crystal packing diagram.

are listed in Table 1,* bond lengths and valency angles in Table 2. The atomic arrangement and crystal packing are shown in Figs. 1 and 2.

Related literature. Tricyclic lactones have been used as synthetic intermediates en route to thromboxanes (Kelly & Roberts, 1980). A detailed NMR study of the title compound (I) has shown interesting long-range couplings between F and H(7A), H(7B) and between F and C(4), C(7) (Roberts, 1986). We undertook the X-ray study to obtain accurate geometrical details of the molecule with particular reference to the F environment.

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, selected mean planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44586 (10 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 Novel, Macrocyclic Schiff Base

BY PARIMAL K. BHARADWAJ, JOSEPH A. POTENZA AND HARVEY J. SCHUGAR

Department of Chemistry, Rutgers, The State University of New Jersey, New Brunswick, New Jersey 08903, USA

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Abstract. (*E,E*)-1,2,3,4,4 α ,12 β -Hexahydro-7,7,10,10-tetramethyl-7H,10H-8,9-dithia-5,12-diazabenzocyclodecene, C₁₄H₂₄N₂S₂, *M_r* = 284.49, monoclinic, *P*2₁/*n*, *a* = 8.962 (1), *b* = 19.356 (3), *c* = 10.178 (1) Å,

β = 114.49 (1)°, *V* = 1606.7 (7) Å³, *Z* = 4, *D_m* = 1.21 (1), *D_x* = 1.176 Mg m⁻³, μ (Mo *K*α, λ = 0.71073 Å) = 0.31 mm⁻¹, *F*(000) = 616, *T* = 296 (1) K, *R_F* = 0.031 for 2066 reflections. The mole-