SHORT STRUCTURAL PAPERS

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A Cyclitol Penta-acetate: (±)-2-Acetoxymethyl-1,3,4,6-tetra-*O*-acetylepiinositol

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Abstract. A cyclitol penta-acetate, $C_{17}H_{24}O_{12}$, was obtained as an intermediary product during a novel synthesis of cyclitol ring systems. Crystals grown from ethanol solution are monoclinic, space group $P2_1/c$, with $a=17{\cdot}04(1)$, $b=5{\cdot}635(4)$, $c=21{\cdot}99(2)$ Å, $\beta=108{\cdot}26(6)°$, $Z=4$, $D_{calc}=1{\cdot}392$, and $D_{obs}=1{\cdot}37$ g cm^{-3}. This structural analysis established the chemical configuration of the compound.

Introduction. The structure analysis was undertaken to establish the chemical configuration of this cyclitol system. Earlier chemical and spectroscopic (infrared and n.m.r.) studies yielded information insufficient to establish the configuration at the tertiary carbon atom C(2), or to determine whether acetyl migration from C(5) to C(4) had occurred during synthesis (Kiely & Cantrell, 1972).

Experimental. All crystals that we examined were twinned and produced Weissenberg photographs in which the spots appeared as doublets. The systematic absences are $h0l$ when l is odd and $0k0$ when k is odd. The intensities were measured by using a Picker FACS-diffractometer (Nickel-filtered Cu $K\alpha$ radiation, scintillation counter, θ–2θ scanning method). About half the intensity data were collected from one crystal, and

Table 1. *Final heavy-atom parameters and their standard deviations*

All values have been multiplied by 10^4.
The temperature parameters shown are coefficients in the expression $T=\exp\,(-\beta_{11}h^2-\beta_{22}k^2-\beta_{33}l^2-2\beta_{12}hk-2\beta_{13}hl-2\beta_{23}kl)$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	7358 (3)	5156 (10)	2039 (3)	39 (3)	114 (19)	20 (2)	−6 (7)	8 (2)	4 (5)
C(2)	7178 (3)	5476 (9)	1326 (2)	29 (3)	148 (20)	20 (2)	−11 (7)	5 (2)	5 (5)
C(3)	7587 (3)	3460 (9)	1070 (2)	27 (3)	163 (20)	17 (2)	4 (7)	2 (2)	−4 (5)
C(4)	8492 (3)	3158 (9)	1429 (2)	29 (3)	177 (21)	17 (2)	5 (7)	11 (2)	−1 (5)
C(5)	8690 (3)	3015 (10)	2156 (3)	26 (3)	241 (23)	21 (2)	7 (8)	4 (2)	9 (6)
C(6)	8284 (3)	5099 (11)	2381 (2)	34 (3)	255 (24)	14 (2)	−33 (8)	4 (2)	12 (5)
O(1)	8404 (2)	4890 (7)	3060 (2)	44 (2)	250 (15)	14 (1)	−14 (5)	6 (1)	1 (4)
C(7)	8710 (4)	6799 (13)	3419 (3)	55 (4)	317 (29)	15 (2)	−26 (10)	5 (2)	−1 (6)
O(2)	8934 (4)	8530 (9)	3226 (2)	143 (4)	418 (23)	22 (1)	−135 (9)	12 (2)	−10 (5)
C(8)	8703 (4)	6495 (12)	4104 (3)	48 (3)	507 (33)	20 (2)	−5 (10)	9 (2)	−17 (7)
O(3)	7009 (2)	7121 (7)	2282 (2)	47 (2)	205 (15)	23 (1)	7 (5)	18 (1)	0 (4)
C(9)	6409 (4)	6667 (14)	2553 (3)	53 (4)	372 (33)	34 (2)	6 (11)	24 (3)	−17 (8)
O(4)	6163 (4)	4783 (10)	2606 (3)	102 (4)	398 (25)	101 (3)	−34 (9)	78 (3)	−9 (8)
C(10)	6070 (4)	8929 (13)	2753 (3)	92 (5)	435 (33)	48 (3)	54 (11)	43 (3)	−25 (8)
O(5)	7518 (2)	7732 (6)	1247 (2)	48 (2)	132 (13)	19 (1)	−1 (5)	10 (1)	12 (3)
C(11)	6262 (4)	5666 (11)	964 (3)	37 (3)	282 (27)	23 (2)	9 (8)	3 (2)	5 (6)
O(6)	5870 (3)	3383 (8)	1014 (2)	22 (2)	358 (21)	33 (1)	11 (6)	1 (1)	−4 (5)
C(12)	5082 (5)	3447 (15)	861 (4)	59 (5)	318 (34)	38 (3)	8 (12)	14 (3)	−16 (8)
O(7)	4657 (3)	5207 (10)	692 (3)	38 (3)	461 (25)	75 (3)	21 (7)	9 (2)	−15 (7)
C(13)	4713 (5)	1065 (15)	902 (4)	48 (4)	420 (37)	82 (4)	−19 (11)	16 (3)	−27 (11)
O(8)	7462 (2)	4027 (7)	407 (2)	42 (2)	201 (15)	14 (1)	9 (5)	5 (1)	6 (3)
C(14)	7374 (3)	2233 (12)	−20 (3)	30 (3)	288 (27)	18 (2)	−1 (8)	5 (2)	13 (6)
O(9)	7390 (3)	192 (7)	115 (2)	89 (3)	198 (16)	21 (1)	−24 (7)	18 (2)	0 (4)
C(15)	7253 (4)	3222 (12)	−675 (3)	54 (4)	388 (29)	20 (2)	16 (9)	7 (2)	24 (6)
O(10)	8971 (2)	5094 (6)	1312 (2)	31 (2)	218 (14)	16 (1)	−13 (5)	8 (1)	2 (3)
C(16)	9320 (4)	4898 (12)	834 (3)	29 (3)	292 (25)	19 (2)	−9 (8)	7 (2)	11 (6)
O(11)	9227 (3)	3195 (8)	492 (2)	53 (2)	332 (19)	27 (1)	−27 (6)	19 (1)	−23 (4)
C(17)	9775 (4)	7096 (11)	793 (3)	52 (3)	344 (28)	20 (2)	−20 (9)	12 (2)	2 (6)
O(12)	9541 (2)	2870 (7)	2475 (2)	27 (2)	390 (19)	21 (1)	19 (5)	2 (1)	18 (4)

the rest of the intensity data were collected from a second crystal; the two crystals had approximate dimensions $0{\cdot}3\times0{\cdot}1\times0{\cdot}05$ mm. Both crystals produced reflections that appeared as double peaks with identical 2θ values, but the χ and φ values of the peaks differed by 0·3–0·8°. Because of considerable overlap it was impossible to completely separate the two peaks; therefore, a composite orientation matrix for use in intensity measurements was calculated by simultaneously refining the angular settings of both peaks from six medium-angle reflections. The settings that were calculated by this method resulted in scans that included contributions from the two peaks of the reflections. Three standard reflections (006, 402, and 112) were monitored at regular intervals while measurements were being made. For both crystals these reflections exhibited a 5% reduction of intensity during the collection of intensity data. Cell parameters were determined by a least-squares refinement of 2θ values for 12 low-angle reflections (Cu $K\beta$, $\lambda=1{\cdot}3922$ Å, 23 ± 2°C) that were measured with the diffractometer. Intensity measurements were made for the 3298 independent reflections with $2\theta\le127°$. The scanning speed was 1° min^{-1}, and a 20 sec background measurement was performed at each terminus of the scans. The intensities were assigned variances, $\sigma^2(I)$, according to the statistics of the scan and background counts, plus a correctional term $(0{\cdot}03S)^2$, S being the scan counts. The standard re-

Table 2. *Hydrogen-atom parameters and their standard deviations*

The values of the coordinates have been multiplied by 10^3.

	x	y	z	B (Å)^2
H(C1)	716 (3)	374 (8)	212 (2)	1·0 (1·1)
H(C3)	728 (3)	217 (9)	108 (2)	3·6 (1·5)
H(C4)	873 (2)	179 (8)	128 (2)	1·2 (1·0)
H(C5)	801 (3)	167 (9)	224 (2)	3·3 (1·4)
H(C6)	852 (3)	638 (9)	231 (2)	1·9 (1·4)
H(C8)	809 (4)	654 (14)	405 (3)	8·5 (2·5)
H(C8)′	917 (5)	722 (19)	437 (4)	10·9 (3·5)
H(C8)″	888 (5)	509 (15)	424 (4)	10·8 (3·3)
H(C10)	659 (5)	972 (17)	294 (4)	17·6 (2·9)
H(C10)′	578 (6)	869 (22)	292 (5)	16·0 (4·8)
H(C10)″	608 (9)	1014 (25)	253 (6)	21·7 (7·0)
H(C11)	609 (3)	596 (11)	050 (3)	5·8 (1·7)
H(C11′)	603 (3)	681 (11)	122 (2)	5·4 (1·7)
H(C13)	416 (5)	116 (14)	72 (3)	8·8 (2·7)
H(C13)′	494 (8)	41 (25)	140 (6)	24·4 (7·1)
H(C13)″	487 (5)	5 (17)	62 (4)	13·0 (3·7)
H(C15)	772 (3)	329 (12)	−71 (3)	9·7 (1·8)
H(C15)′	698 (5)	468 (16)	−73 (4)	11·2 (3·1)
H(C15)″	681 (5)	243 (16)	−100 (4)	11·4 (3·3)
H(C17)	1012 (4)	723 (13)	115 (3)	6·6 (2·4)
H(C17)′	943 (4)	862 (14)	70 (3)	9·8 (2·5)
H(C17)″	1001 (3)	683 (11)	43 (2)	6·0 (1·6)
H(O5)	745 (3)	806 (11)	87 (2)	8·0 (1·6)
H(O12)	981 (5)	416 (15)	252 (4)	9·0 (3·0)

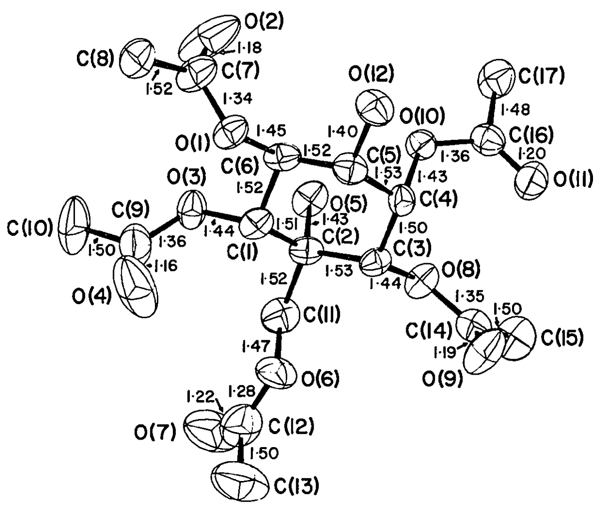

Fig. 1. A thermal ellipsoid plot (Johnson, 1965), at the 50% probability level, showing the numbering system and the bond lengths. Hydrogen atoms are not shown. The estimated standard deviations in bond lengths are about 0·005 Å.

Table 3. *Bond angles involving only non-hydrogen atoms. The estimated standard deviations are about* 0·4°

C(2)—C(1)—O(3)	108·8°
C(2)—C(1)—C(6)	110·9
O(3)—C(1)—C(6)	108·6
C(1)—C(2)—C(3)	109·0
C(1)—C(2)—C(11)	113·1
C(1)—C(2)—O(5)	105·9
C(3)—C(2)—O(5)	111·6
C(3)—C(2)—C(11)	111·9
O(5)—C(2)—C(11)	105·3
C(2)—C(3)—C(4)	113·7
C(2)—C(3)—O(8)	105·5
C(4)—C(3)—O(8)	111·0
C(3)—C(4)—C(5)	114·2
C(3)—C(4)—O(10)	111·6
C(5)—C(4)—O(10)	106·0
C(4)—C(5)—C(6)	108·9
C(4)—C(5)—O(12)	112·5
C(6)—C(5)—O(12)	112·8
C(1)—C(6)—C(5)	111·3
C(1)—C(6)—O(1)	107·6
C(5)—C(6)—O(1)	109·9
O(1)—C(7)—O(2)	124·3
O(1)—C(7)—C(8)	111·8
C(8)—C(7)—O(2)	123·9
O(3)—C(9)—O(4)	124·2
O(3)—C(9)—C(10)	110·6
C(10)—C(9)—O(4)	125·3
C(2)—C(11)-O(6)	108·4
O(6)—C(12)-O(7)	125·4
O(6)—C(12)-C(13)	112·7
C(13)-C(12)-O(7)	121·9
O(8)—C(14)-O(9)	124·3
O(8)—C(14)-C(15)	109·9
C(15)-C(14)-O(9)	125·9
O(10)-C(16)-O(11)	123·0
O(10)-C(16)-C(17)	110·1
C(17)-C(16)-O(11)	126·9
C(6)—O(1)—C(7)	116·5
C(1)—O(3)—C(9)	118·3
C(11)-O(6)—C(12)	114·9
C(3)—O(8)—C(14)	118·9
C(4)—O(10)-C(16)	119·1

flections were used to scale the sets of intensity data from the two crystals. To compensate for the decrease in the intensities of the standard reflections during data collection, the intensities and their standard deviations were scaled by a least-squares procedure similar to that described by Ibers (1969). A trial structure, including all 29 heavy atoms, was obtained by direct methods with the computer program *MULTAN* (Germain, Main & Woolfson, 1971). The trial structure was refined by full-matrix least-squares methods; the quantity minimized was $\sum w(F_o^2-F_c^2/k^2)^2$, with k as a scale factor and the weight w equal to $1/\sigma^2(F_o^2)$. All hydrogen atoms were located in difference Fourier maps that were calculated during the later stages of refinement. In addition to the positional parameters, anisotropic temperature parameters for heavy atoms, isotropic temperature factors for hydrogen atoms, and an isotropic extinction coefficient were refined. All of the 3298 reflections were considered observable and were included in the refinement with their calculated weights. The final R_1 index $[\sum||F_o|-|F_c||/\sum|F_o|]$ for all reflections is 0·22, the R_2 index $[\sum|F_o^2-F_c^2|/\sum F_o^2]$ is 0·11, and goodness-of-fit is 1·10. If only those 1904 reflections with $I>\sigma(I)$ are considered, $R_1=0{\cdot}13$, $R_2=0{\cdot}09$ and goodness-of-fit $=1{\cdot}42$. A final difference Fourier map showed no peaks or troughs with magnitudes exceeding 0·8 e Å^{-3}.

Results. Table 1 gives the heavy-atom parameters and Table 2 lists the hydrogen atom parameters. A table of observed and calculated structure factors is available.* The tertiary hydroxyl group at C(2) is in the axial position, and an acetyl group is at the C(4) position. Fig. 1 depicts the molecular conformation, thermal ellipsoids and bond lengths between heavy atoms. Bond angles involving only non-hydrogen atoms are given in Table 3. The C–H bond lengths have an average value of 0·93 Å and range from 0·75 to 1·10 Å. The two O–H bond lengths are 0·85 and 0·83 Å.

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* This table has been deposited with the National Lending Library, England as Supplementary Publication No. SUP 30073 (16 pp.). Copies may be obtained from the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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4-(4-Chloro-α,α,α-trifluoro-*m*-tolyl)-1-[4, 4-bis-(*p*-fluorophenyl)butyl]-4-piperidinol (Penfluridol)

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Abstract. Orthorhombic, $P2_12_12_1$, $a=16{\cdot}141$ (9), $b=9{\cdot}559$ (5), $c=16{\cdot}794$ (10) Å, 25°C, $C_{28}H_{27}NOF_5Cl$, $M=523{\cdot}96$, $Z=4$, $D_x=1{\cdot}337$ g cm^{-3}.

Introduction. Penfluridol is the longest-acting neuroleptic known today. Transparent needle-like crystals were obtained by slow evaporation from a (90:10) mixture of n-hexane and isopropyl alcohol.

Experimental. Lattice parameters were obtained by least-squares refinement of the setting angles of twelve reflexions. Weissenberg photographs showed absences characteristic of the space group $P2_12_12_1$.

Intensity data were collected on a Picker four-circle card-controlled diffractometer. The relevant data are given in Table 1.

Table 1. *Experimental data*

Crystal dimensions: 0·30 × 0·30 × 0·25 mm
Source Cu $K\bar{\alpha}$; $\lambda=1{\cdot}5418$ Å; Ni filter; ω–2θ scan; $\Delta2\theta=\pm1°$; $\theta_{max}=57{\cdot}5°$
Confidence level: 2·0
Total number of independent reflexions: 2039
Total observed: 1617

The quality of the data was rather poor as the crystal was highly mosaic and decomposed under irradiation. A gradual loss of intensity which reached 10%