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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, $C_{10}H_{13}NO_4$, $M_r = 211{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}748$ (2), $b = 9{\cdot}918$ (3), $c = 10{\cdot}136$ (2) Å, $V = 980$ Å^3, $Z = 4$, $D_x = 1{\cdot}43$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}947$ mm^{-1}, $F(000) = 448$, $T = 293$ K, $R = 0{\cdot}0685$ 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, $R_{int} = 0{\cdot}05$, 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 $w = 1/[\sigma^2(F) + 0{\cdot}000968F^2]$. H atoms from difference Fourier syntheses, included in refinement with fixed U_{iso}. In final cycle max. $\Delta/\sigma = 0{\cdot}107$ (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). $R = 0{\cdot}0685$, $wR = 0{\cdot}0759$ 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.

* 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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One intermolecular hydrogen bond is formed by the OH group [O(5)···N(1′)(2−x, $-\frac{1}{2}+y$, $\frac{1}{2}-z$) = 3·065 (7) Å]. The fused rings make an angle of 118·2 (7)°, slightly larger than the previous values.

The synthesis of this compound was intended to produce the *endo* isomer by a formally disallowed concerted cycloaddition of a ketene to an alkene. This method of formation of a cyclobutanone is thought to involve a $\pi 2s + \pi 2a$ process and is usually stereospecific with a strong preference for the *endo* isomer (Brady & Hoff, 1970). Selectivity is determined by the larger lobe of the HOMO of the alkene overlapping the larger lobe of the LUMO of the ketene (Brook, Harrison & Duke, 1970). The O attached to the alkene will raise the energy and amplitude of the HOMO. Either this must be responsible for the change in stereochemistry or the mechanism has become stepwise and the thermodynamically most stable product is formed.

We thank the University of Leeds Computing Service for the provision of facilities.

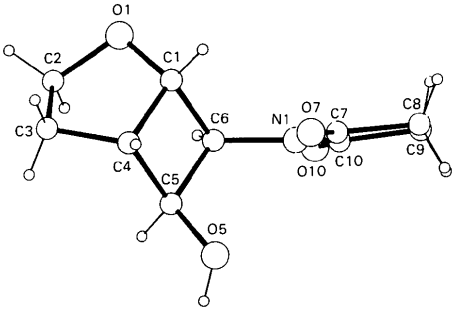

Fig. 1. Diagram of the molecule showing numbering scheme.

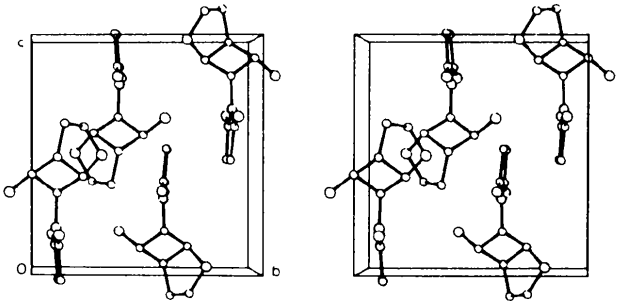

Fig. 2. Stereoscopic view along the a axis showing the cell packing.

Table 1. *Atom coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² × 10⁴)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
N(1)	9586 (5)	1067 (5)	1954 (4)	216
C(1)	10412 (8)	2221 (5)	4085 (5)	258
C(2)	9527 (10)	2284 (8)	6159 (7)	495
C(3)	10781 (8)	1439 (8)	6297 (6)	402
C(4)	11170 (5)	1131 (6)	4875 (5)	249
C(5)	10334 (7)	11 (5)	4173 (5)	272
C(6)	9537 (5)	1117 (6)	3390 (4)	219
C(7)	10759 (5)	1180 (6)	1227 (5)	241
C(8)	10375 (7)	1272 (7)	−215 (5)	326
C(9)	8833 (7)	1174 (8)	−236 (6)	357
C(10)	8396 (6)	1026 (6)	1191 (6)	272
O(1)	9690 (5)	3054 (4)	4963 (4)	401
O(5)	11078 (6)	−939 (4)	3436 (4)	508
O(7)	11903 (4)	1192 (6)	1684 (4)	394
O(10)	7258 (4)	922 (6)	1622 (5)	491

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

C(1)–C(6)	1·556 (8)	N(1)–C(6)	1·457 (6)
C(1)–C(4)	1·535 (8)	N(1)–C(7)	1·366 (7)
C(2)–C(3)	1·489 (10)	N(1)–C(10)	1·395 (6)
C(3)–C(4)	1·521 (8)	O(1)–C(1)	1·404 (7)
C(4)–C(5)	1·550 (8)	O(1)–C(2)	1·442 (8)
C(5)–C(6)	1·561 (7)	C(5)–O(5)	1·404 (7)
C(7)–C(8)	1·512 (7)	C(7)–O(7)	1·207 (7)
C(8)–C(9)	1·507 (8)	C(10)–O(10)	1·197 (7)
C(9)–C(10)	1·515 (8)		
O(1)–C(1)–C(4)	109·0 (4)	C(1)–C(6)–N(1)	117·3 (5)
O(1)–C(1)–C(6)	115·2 (6)	C(6)–N(1)–C(7)	124·3 (5)
C(4)–C(1)–C(6)	90·3 (4)	C(6)–N(1)–C(10)	121·8 (5)
O(1)–C(2)–C(3)	106·7 (6)	C(7)–N(1)–C(10)	113·5 (4)
C(2)–C(3)–C(4)	103·2 (5)	N(1)–C(7)–C(8)	108·6 (5)
C(3)–C(4)–C(5)	116·6 (5)	N(1)–C(7)–O(7)	124·5 (5)
C(3)–C(4)–C(1)	103·5 (5)	O(7)–C(7)–C(8)	126·8 (5)
C(1)–C(4)–C(5)	90·7 (4)	C(7)–C(8)–C(9)	104·9 (5)
C(4)–C(5)–C(6)	89·5 (4)	C(8)–C(9)–C(10)	105·9 (5)
C(4)–C(5)–O(5)	117·0 (6)	C(9)–C(10)–N(1)	107·0 (5)
O(5)–C(5)–C(6)	117·3 (4)	C(9)–C(10)–O(10)	128·1 (5)
C(5)–C(6)–C(1)	89·5 (4)	O(10)–C(10)–N(1)	124·8 (6)
C(5)–C(6)–N(1)	117·9 (5)	C(1)–O(1)–C(2)	106·1 (5)

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