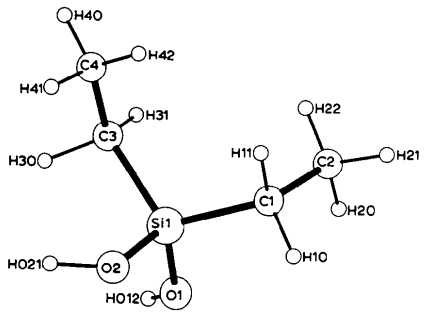

Fig. 1. Diagram of the molecule showing numbering scheme.

The structure differs from that deduced by Kakudo & Watase (1952) (in which the space group was assumed to be $P2_1/a$). Their suggested unusual 'opposed dipole' arrangement of OH groups does not occur and the hydrogen bonds have normal lengths (2·679 to 2·727 Å). There are two equally populated schemes of hydrogen bonding as listed in Table 2. In addition, although the *ac* projection may be described in terms of a centred orthorhombic cell, the quoted relationship between structure factors is only valid when $k = 0$.*

It has been suggested that other silanediol structures were analogous to that of the title compound and therefore contained an unusual type of hydrogen bonding. It now appears that these assumptions are unnecessary.

* See deposition footnote.

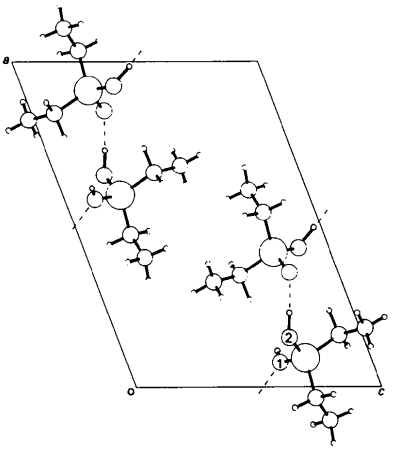

Fig. 2. *b*-axis-projection packing diagram (*PLUTO*78, Motherwell & Clegg, 1978).

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Structure of a Carbocyclic Analogue of Penicillin

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Abstract. Methyl 4-(6-*endo*-hydroxybicyclo[3.2.0]-hept-2-en-7-*endo*-ylamino)-4-oxobutyrate (6*R**,7*S**), $C_{12}H_{17}NO_4$, $M_r = 239{\cdot}27$, monoclinic, $P2_1/c$, $a = 11{\cdot}594$ (1), $b = 6{\cdot}138$ (1), $c = 17{\cdot}512$ (1) Å, $\beta = 102{\cdot}35$ (1)°, $V = 1217{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}31$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}725$ mm^{-1}, $F(000) = 512$, $T = 293$ K, $R = 0{\cdot}077$ for 1714 unique observed reflections. The cyclobutanone and cyclopentene rings of the bicyclohept-2-en-6-one moiety make an angle of 116·5 (6)°, the bond lengths and angles are normal. Both the NH and OH groups form hydrogen bonds with lengths of 3·35 (1) and 2·74 (1) Å respectively, linking molecules in the *b*-axis direction.

Introduction. This determination forms one of a series of structure determinations of carbocyclic analogues of penicillin in which the β-lactam ring is replaced by a cyclobutanone. One structure of such a carbocyclic analogue of penicillin has been published (Sheldrick, Akrigg, Page & Agathocleous, 1984).

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Experimental. Material prepared by M. I. Page and crystallized from ethanol/water. Tabular crystal, 0·17 × 0·23 × 0·30 mm. Enraf–Nonius CAD-4*F* diffractometer. No correction for absorption. $2\theta_{max} = 140°$, $\pm hk\pm l$; 5048 reflections measured. Check reflection 008: average count 838, calculated σ (of the distribution) = 57 (6·8%). Cell dimensions from θ measurements of 34 reflections. Data merged using *SHELX*76 (Sheldrick, 1976) giving 1714 unique reflections, index range h −14 to 13, k 0 to 6, l 0 to 21; 1976 considered unobserved [$F_o < 3\sigma(F_o)$], merging $R_{int} = 0{\cdot}042$. *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 $w = 1/[\sigma^2(F) + 0{\cdot}008383F^2]$. H atoms from difference Fourier syntheses. In final cycle max. $\Delta/\sigma = 0{\cdot}047$ (C) and 0·168 (H), average = 0·014. $\Delta\rho$ in final difference Fourier map within +0·27 and −0·23 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). $R = 0{\cdot}0774$, $wR = 0{\cdot}1186$ for 1714 observed reflections.

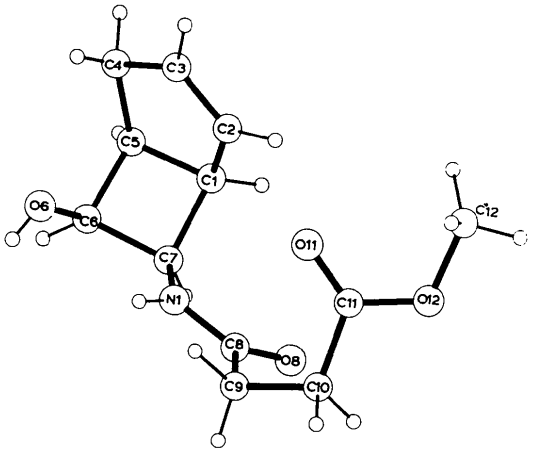

Fig. 1. Diagram of the molecule showing numbering scheme.

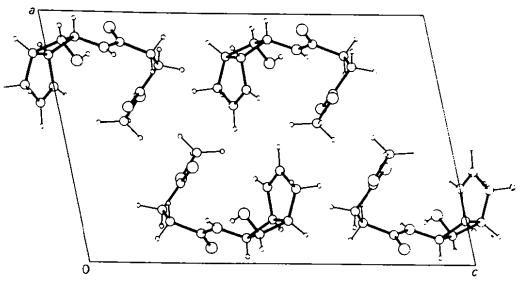

Fig. 2. *b*-axis-projection packing diagram (*PLUTO*78, Motherwell & Clegg, 1978).

Discussion. The molecule and numbering scheme are shown in Fig. 1, and a diagram 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.

Hydrogen bonds are formed by the NH group [N(1)···O(12′)(x,y−1,z) = 3·346 (7) Å, intermolecularly and N(1)···O(6) = 2·749 Å, intramolecularly, H(N1) lying 2·60 (4) Å from O(12′) and 2·47 (4) Å from O(6)] and the OH group [O(6)···O(8′)(x,y−1,z) = 2·738 (7) Å] connecting molecules in the *b*-axis direction.

The fused rings make an angle of 116·5 (7)° with each other, compared with 117·1 (10)° in an analogous

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, torsion angles and best planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42081 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates* (×10^4) *and* U_{eq} (Å^2 × 10^4)

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

	x	y	z	U_{eq}
C(1)	1763 (3)	−47 (7)	9971 (2)	401
C(2)	3052 (4)	260 (8)	10005 (2)	482
C(3)	3699 (4)	−1277 (9)	10430 (3)	576
C(4)	2991 (4)	−2920 (9)	10749 (2)	567
C(5)	1716 (3)	−2305 (6)	10373 (2)	426
C(6)	1181 (3)	−3310 (7)	9556 (2)	418
C(7)	1029 (3)	−990 (6)	9199 (2)	359
C(8)	1200 (3)	1036 (6)	8044 (2)	349
C(9)	1657 (4)	1128 (7)	7297 (2)	433
C(10)	2110 (4)	3379 (8)	7159 (2)	463
C(11)	3179 (3)	4000 (7)	7776 (2)	403
C(12)	4398 (5)	6897 (10)	8364 (4)	673
O(6)	1975 (3)	−4605 (5)	9262 (2)	609
O(8)	627 (3)	2569 (5)	8233 (2)	532
O(11)	3810 (3)	2728 (5)	8175 (2)	655
O(12)	3359 (3)	6153 (5)	7812 (2)	508
N(1)	1420 (3)	−734 (5)	8481 (2)	369

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

C(1)–C(2)	1·494 (6)	C(7)–N(1)	1·435 (4)
C(2)–C(3)	1·329 (6)	N(1)–C(8)	1·321 (5)
C(3)–C(4)	1·483 (7)	C(8)–C(9)	1·515 (5)
C(4)–C(5)	1·531 (6)	C(8)–O(8)	1·237 (4)
C(5)–C(6)	1·561 (5)	C(9)–C(10)	1·516 (5)
C(5)–C(1)	1·561 (5)	C(10)–C(11)	1·507 (5)
C(6)–C(7)	1·550 (5)	C(11)–O(11)	1·190 (5)
C(6)–O(6)	1·395 (5)	C(11)–O(12)	1·337 (5)
C(7)–C(1)	1·547 (5)	O(12)–C(12)	1·447 (6)
C(2)–C(1)–C(5)	103·0 (3)	C(7)–N(1)–C(8)	122·6 (3)
C(2)–C(1)–C(7)	116·1 (3)	C(6)–C(7)–N(1)	115·1 (3)
C(5)–C(1)–C(7)	89·8 (3)	C(1)–C(7)–C(6)	89·9 (3)
C(1)–C(2)–C(3)	111·9 (4)	N(1)–C(8)–O(8)	121·3 (3)
C(2)–C(3)–C(4)	113·7 (4)	N(1)–C(8)–C(9)	118·1 (3)
C(3)–C(4)–C(5)	103·4 (3)	C(9)–C(8)–O(8)	120·7 (3)
C(4)–C(5)–C(6)	117·0 (3)	C(8)–C(9)–C(10)	111·6 (3)
C(4)–C(5)–C(1)	106·8 (3)	C(9)–C(10)–C(11)	112·1 (3)
C(1)–C(5)–C(6)	89·0 (3)	C(10)–C(11)–O(11)	124·3 (4)
C(5)–C(6)–O(6)	113·5 (3)	C(10)–C(11)–O(12)	112·3 (3)
C(5)–C(6)–C(7)	89·7 (3)	O(11)–C(11)–O(12)	123·3 (4)
C(7)–C(6)–O(6)	113·7 (3)	C(11)–O(12)–C(12)	116·3 (4)
C(1)–C(7)–N(1)	120·5 (3)		

compound (Sheldrick *et al.*, 1984). The amide group is planar as shown by the torsion angles C(7)–N(1)–C(8)–C(9) = 179·9 (3) and C(7)–N(1)–C(8)–O(8) = −0·7 (5)° and the carbonyl group nearly so with C(10)–C(11)–O(12)–C(12) = −177·3 (4) and O(11)–C(11)–O(12)–C(12) = 1·2 (6)°. The chain twists around C(8)–C(9), torsion angle N(1)–C(8)–C(9)–C(10) = −141·0 (4)°, allowing the H atoms of the CH_2 groups to adopt a staggered conformation, and curving the chain around to form a compact shape rather than an extended form.

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

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Structure of a Ten-Membered Macrocyclic Diester of (+)-Retronecine, $C_{12}H_{15}NO_4$: a Pyrrolizidine Alkaloid Analogue

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Abstract. (+)-7-Methyl-2,3,5,7a-tetrahydro-1*H*-pyrrolizine-1,8-diyl succinate, $M_r = 237·1$, orthorhombic, $P2_12_12_1$, $a = 7·934$ (1), $b = 10·786$ (3), $c = 13·246$ (2) Å, $V = 1133·5$ Å^3, $Z = 4$, $D_m = 1·41$, $D_x = 1·39$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 1·12$ cm^{-1}, $F(000) = 504$, $T = 291$ K, final $R = 0·043$ for 1429 unique reflections. The conformation of the pyrrolizidine moiety in this novel ten-membered macrocyclic pyrrolizidine alkaloid analogue is similar to those found in 11- and 12-membered alkaloids. The ester-group carbonyls lie on either side of the macrocycle in a nearly antiparallel orientation. This conformation is similar to those observed in 12-membered pyrrolizidine alkaloids containing retronecine, whereas most 11-membered pyrrolizidine alkaloids have been found to possess ester carbonyl groups that are synparallel.

Introduction. Interest in pyrrolizidine alkaloids (PA's) stems from their widespread occurrence and their important biological activity, which includes hepatotoxicity. The most toxic PA's are macrocyclic diesters of (+)-retronecine, which occur with 11- and 12-membered ring sizes. Naturally occurring 13-membered diesters and 14-membered macrocyclic triesters of (+)-retronecine are also known (Robins, 1982). At present PA's containing ten-membered rings are unknown. Recently, a range of ten-membered macrocyclic diesters of (+)-retronecine was synthesized (Burton & Robins, 1985) and here we present the crystal structure of the parent compound, (+)-7,9-*O,O*-(succinyl)retronecine.

Experimental. Colourless plate-shaped crystals grown from hexane, crystal *ca* 0·4 × 0·4 × 0·1 mm used in data collection, CAD-4 diffractometer. Preliminary Weissenberg photographs indicated crystals to be orthorhombic, $P2_12_12_1$. D_m by flotation. 1589 independent intensities, θ limit 28°, $\omega/2\theta$ scan. 2 standard intensities used to monitor variations in intensity data; <3% variation observed. Least-squares technique based on 25 reflections, $\theta > 12°$, used to refine lattice parameters. No absorption correction. h 0–10, k 0–14, l 0–17. Structure solution by direct methods with *MITHRIL* (Gilmore, 1984). Full-matrix least-squares refinement on F of coordinates and anisotropic thermal parameters for all non-hydrogen atoms converged to R and wR of 0·043 and 0·058 with $w = (1/\sigma_F^2)$. Hydrogen-atom parameters included in final least squares, but not refined. 1429 reflections, $I \geq 2·5\sigma_I$, used. $\Delta_{max}/\sigma = 0·10$; max. and min. heights in final difference Fourier synthesis = 0·15 and −0·2 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using Glasgow *GX* package (Mallinson & Muir, 1985).

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