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Structure of L-1-Benzyl-4-hydroxymethyl-2-azetidinone

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Abstract. $C_{11}H_{13}NO_2$, $M_r = 191{\cdot}22$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}183\,(1)$, $b = 6{\cdot}364\,(1)$, $c = 21{\cdot}79\,(1)$ Å, $V = 996{\cdot}07\,(1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}25$, $D_x = 1{\cdot}275$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 5{\cdot}2$ cm^{-1}, $F(000) = 408$, $T = 298$ K. Final $R = 0{\cdot}045$ for 1164 observed reflections. In the β-lactam system, the amide group is almost planar with bond distances 1·334 (4) and 1·220 (4) Å for C—N and C═O, respectively. These structural results are consistent with those of other biologically inactive β-lactams.

Introduction. In those β-lactams having antibiotic activity, Sweet & Dahl (1970) have pointed out that the lactam N atom has pyramidal character. Increased activity was also correlated with decreased electron delocalization in the amide group as shown by lengthening of the C—N bond and shortening of the C═O bond. The crystal-structure determination now reported for L-1-benzyl-4-hydroxymethyl-2-azetidinone (Fig. 1) shows molecular features for this biologically inactive β-lactam which help to confirm the Sweet & Dahl hypothesis.

Experimental. A sample of the title compound was provided by C. H. Lee. Colorless rectangular crystals were grown from dichloromethane solution. The crystal density was measured by flotation in benzene/carbon tetrachloride. A crystal measuring 0·3 × 0·3 × 0·5 mm was used for data collection with an Enraf–Nonius CAD-4 diffractometer using Nb-filtered Mo $K\alpha$ radiation. Unit-cell parameters were determined from 25 reflections having $30 < 2\theta < 40°$. Intensities were measured for 1720 reflections with $(\sin\theta)/\lambda < 0{\cdot}70$ Å^{-1}, indices in the range $0 \le h \le 10$, $0 \le k \le 8$ and $0 \le l \le 30$ using $\omega/2\theta$ scans with scan width $\Delta\omega = (0{\cdot}7 + 0{\cdot}35\tan\theta)°$. Three monitor reflections showed deviations less than 3% from their average intensities. No absorption corrections were applied. There were 1164 reflections with $|F_o| > 3\sigma(F_o)$ which were used in the structure refinement.

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Table 1. *Final positional parameters of non-*H *atoms* ($\times 10^4$) *and isotropic mean-square amplitudes of thermal vibration* ($\text{Å}^2 \times 10^3$)

	x	y	z	U_{eq}
N1	4379 (3)	3952 (4)	1637 (1)	40 (1)
C2	3910 (4)	1966 (5)	1691 (1)	40 (1)
C3	1873 (5)	2349 (6)	1559 (2)	49 (1)
C4	2450 (4)	4690 (6)	1529 (1)	47 (1)
C5	1705 (5)	6116 (7)	2018 (2)	55 (1)
O6	1731 (4)	5171 (5)	2604 (1)	59 (1)
C7	6144 (4)	5049 (6)	1679 (1)	42 (1)
C8	7117 (4)	5285 (5)	1070 (1)	34 (1)
C9	7273 (5)	3585 (5)	0675 (1)	46 (1)
C10	8195 (5)	3788 (6)	0117 (2)	58 (1)
C11	8960 (5)	5677 (6)	−0050 (2)	59 (1)
C12	8843 (6)	7366 (7)	0342 (2)	59 (1)
C13	7917 (5)	7172 (5)	0900 (2)	45 (1)
O14	4847 (3)	0367 (4)	1801 (1)	55 (1)

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

N1—C2	1·337 (4)	N1—C4	1·482 (4)
N1—C7	1·450 (4)	C2—C3	1·515 (5)
C2═O14	1·222 (4)	C3—C4	1·547 (5)
C4—C5	1·498 (5)	C5—O6	1·411 (5)
C7—C8	1·507 (4)	C8—C9	1·387 (4)
C8—C13	1·382 (5)	C9—C10	1·390 (4)
C10—C11	1·371 (5)	C11—C12	1·375 (6)
C12—C13	1·391 (6)		
C3—C2—N1	93·2 (2)	C3—C4—N1	86·5 (2)
C4—N1—C2	94·8 (2)	C4—C3—C2	85·4 (3)
C5—C4—N1	114·3 (3)	C5—C4—C3	117·2 (3)
O6—C5—C4	112·4 (3)	C7—N1—C2	132·7 (2)
C7—N1—C4	132·5 (2)	C8—C7—C1	113·4 (2)
C9—C8—C7	120·4 (3)	C10—C9—C8	120·6 (3)
C11—C10—C9	120·3 (4)	C12—C11—C10	119·7 (4)
C12—C13—C8	120·7 (3)	C13—C8—C7	121·0 (3)
C13—C8—C9	118·6 (3)	C13—C12—C11	120·2 (4)
O14═C2—N1	131·4 (3)	O14═C2—C3	135·5 (3)
C4—N1—C2—C3	2·5 (3)	C2—C3—C4—C5	−113·5 (3)
C4—N1—C2═O14	−178·1 (3)	N1—C4—C5—O6	−57·9 (4)
C7—N1—C2—C3	−178·4 (3)	C3—C4—C5—O6	41·2 (4)
C7—N1—C2═O14	1·0 (5)	N1—C7—C8—C9	−46·4 (4)
C2—N1—C4—C3	−2·5 (2)	N1—C7—C8—C13	135·4 (3)
C2—N1—C4—C5	115·9 (3)	C7—C8—C9—C10	−179·0 (3)
C7—N1—C4—C3	178·4 (3)	C13—C8—C9—C10	−0·8 (5)
C7—N1—C4—C5	−63·2 (4)	C7—C8—C13—C12	178·8 (3)
C2—N1—C7—C8	91·7 (4)	C9—C8—C13—C12	0·6 (5)
C4—N1—C7—C8	−89·5 (4)	C8—C9—C10—C11	−0·1 (5)
N1—C2—C3—C4	−2·4 (2)	C9—C10—C11—C12	1·2 (6)
O14═C2—C3—C4	178·3 (4)	C10—C11—C12—C13	−1·4 (6)
C2—C3—C4—N1	2·2 (2)	C11—C12—C13—C8	0·5 (6)

The crystal structure was determined by direct methods using *SHELX*86 (Sheldrick, 1986) and refined by full-matrix least squares using the *SHELX*76 system (Sheldrick, 1976). Atomic scattering factors were from Cromer & Waber (1974) except for H (Stewart, Davidson & Simpson, 1965). The residual minimized was $\sum w\Delta^2$ with $\Delta = |F_o| - |F_c|$ and $w = 1/[\sigma^2(F_o) + 0{\cdot}001F_o^2]$. All H atoms were located in a difference Fourier map. The structure parameters consisted of a scale factor, positional parameters for all atoms, and thermal parameters which were anisotropic for non-H atoms and isotropic for H atoms. Final values with e.s.d.'s are given in Table 1.* In the final cycle of refinement the largest parameter change was 2σ. The agreement indices were $R(F) = 0{\cdot}045$ and $wR(F) = 0{\cdot}047$. In a Fourier difference synthesis, the maximum and minimum residual electron densities were 0·68 and $-0{\cdot}58$ e Å^{-3}. Molecular bond lengths, bond angles and torsion angles are shown in Table 2.

Discussion. The absolute configuration of the molecule shown in Fig. 1 is derived from the starting material of L-aspartic acid. It has not been confirmed by X-ray anomalous scattering.

The benzene rings are planar within experimental error, and the four lactam ring atoms are nearly coplanar. The dihedral angle between these planes is 106°. The average C—C bond length in the benzene ring is 1·383 Å. The lactam ring atom N1 is displaced by only 0·007 (3) Å from the plane of the three atoms to which it is chemically bonded and therefore has no significant pyramidal character.

As further indications of the planarity of the amide group, the torsion angles C7—N1—C2—O14 and C4—N1—C2—O14 are 1·0 (5) and −178·1 (3)° respectively. The effects of electron delocalization can be seen in the short N1—C2 bond length of 1·337 (4) Å which is indicative of considerable double-bond character. These values are similar to the corresponding N—C bond lengths in the crystal

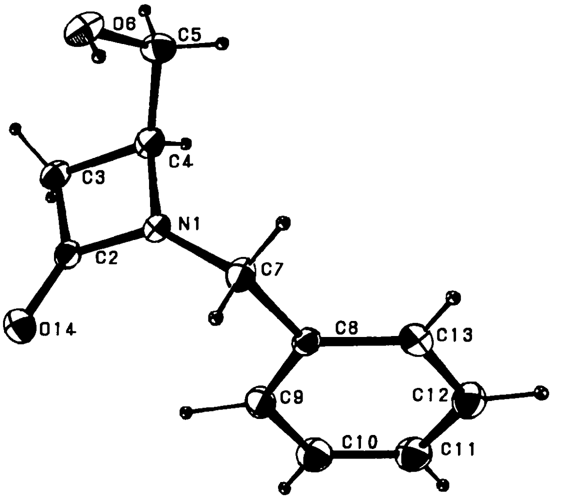

Fig. 1. Molecular structure and atom-numbering scheme in L-1-benzyl-4-hydroxymethyl-2-azetidinone. Ellipsoids drawn at 20% probability.

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* Tables of anisotropic thermal parameters, H-atom parameters, distances and angles involving H atoms, molecular least-squares planes and the observed and calculated structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52472 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

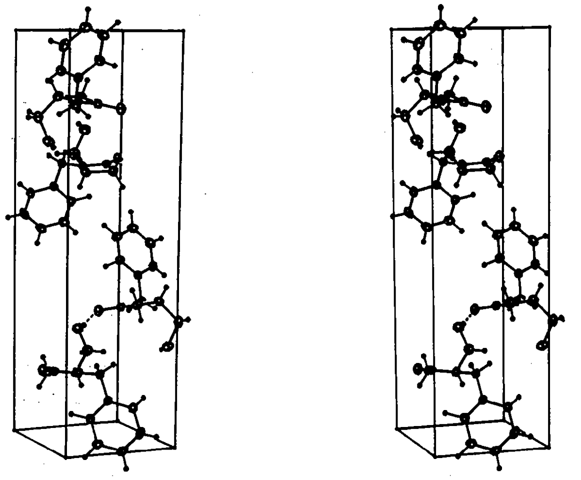

Fig. 2. Stereoscopic drawing of the crystal packing. The dashed line indicates the hydrogen bond.

structure of a closely related azetidone derivative (1·342 Å; Lee, Cho, Kim, Shin, Ruble & Craven, 1990) and in the crystal structure of a Δ^2cephalosperin (1·339 Å; Sweet & Dahl, 1970), all three compounds inactive as antibiotics. In contrast, C—N bond lengths of 1·392 (4) Å are reported for an active penicillin derivative by Domiano, Nardelli, Blasmo, Macchia & Macchia (1979). Thus the results from the present structure determination are consistent with the structure–activity relationship proposed by Sweet & Dahl (1970).

As can be seen in Fig. 1, the molecule adopts a conformation with O6 lying over the face of the β-lactam ring. This gives rise to unfavorable intramolecular repulsions which could be avoided in the isolated molecule by rotations of ±120° about the C4—C5 bond. In the crystal structure, the N1—C4—C5—O6 group with torsion angle −58° (Table 2) has the *gauche* configuration. The twisted configuration, which is found to be preferred for the N—C—C—O system in a variety of molecules, has been attributed to favorable intramolecular Coulombic interactions (Sundaralingam, 1972). The hydroxyl O6 is brought into proximity with the lactam carbonyl C2, along a pathway suitable for nucleophilic attack (Bürgeil, Dunitz & Shefter, 1974). However, the C···O distance (3·27 Å) is not unusually short and the carbonyl C atom is found to be coplanar within experimental error with the three atoms to which it is covalently bonded. In the crystal structure (Fig. 2), the hydroxyl group forms an intermolecular hydrogen bond with the carbonyl oxygen, O6—H6···O14 having O6···O14 distance 2·78 Å and O—H···O angle 169 (4)°.

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Structure of Tetramethylammonium Bromide: a Redetermination

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Abstract. $C_4H_{12}N^+.Br^-$, $M_r = 154·0$, tetragonal, $P4/nmm$, $a = b = 7·708$ (1), $c = 5·498$ (1) Å, $V = 326·6$ Å^3, $Z = 2$, $D_x = 1·566$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71069 Å, $\mu = 61·1$ cm^{-1}, $F(000) = 156$, $T = 293$ K, $R = 0·055$ for all 298 unique reflections. In this redetermination in a higher symmetry, the structure

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