bonds is shown in Fig. 3. Additionally, some short intramolecular contacts [O(1)···C(10) = 2·779 (2), O(1)···H(C10) = 2·44 (2) Å] are observed in isomer (II) which can be most probably ascribed to steric constraints in the molecule.

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Structure of 4-Triphenylmethylthio-2-azetidinone

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Abstract. $C_{22}H_{19}NOS$, $M_r = 345{\cdot}5$, monoclinic, $C2/c$, $a = 27{\cdot}592$ (2), $b = 8{\cdot}021$ (1), $c = 16{\cdot}359$ (1) Å, $\beta = 96{\cdot}89$ (1)°, $V = 3594{\cdot}4$ (7) Å^3, $Z = 8$, $D_m = 1{\cdot}28$ (1), $D_x = 1{\cdot}28$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 16{\cdot}14$ cm^{-1}, $F(000) = 1456$, room temperature, $R = 0{\cdot}033$ for 2793 reflections with $|F_o| > 3\sigma(F)$. The isolated β-lactam ring is planar to within 0·006 Å and the dimensions of the ring are similar to those found in penicillins and cephalosporins.

Introduction. The structures of β-lactam compounds with bicyclic skeletons such as penicillins and cephalosporins have been well investigated, but only a little is known about the structure of monocyclic β-lactams, especially of *N*-aryl-substituted monocyclic β-lactams whose β-lactam rings have been found to be flat (Fujiwara, Varley & van der Veen, 1977; Kartha & Ambady, 1973). It is known that aldol-type reactions between the α-position H and the β-lactam carbonyl give stereochemically different structures for bicyclic and monocyclic compounds (Yoshida, Hayashi, Takeda, Oida & Ohki, 1981; DiNinno, Beattie & Christensen, 1977), which suggests that there are reaction intermediates with different conformations for each structure.

The present work attempts to reveal details of the monocyclic β-lactam structure as part of a study of the stereochemistry in the aldol reaction of the β-lactam ring.

Experimental. The synthesized material was crystallized from dichloromethane/*n*-hexane solution. D_m by flotation in KI/H_2O. Diffraction intensities were measured from a colorless prismatic crystal 0·20 × 0·14 × 0·44 mm, in the ω–2θ scan mode with variable scan width to a maximum $2\theta = 125°$ on a Rigaku-AFC diffractometer using Ni-filtered Cu $K\alpha$ radiation. Lattice parameters and orientation matrix refined with 20 reflections in range $21 < \theta < 23°$ by least-squares method. 2934 unique reflections meas-

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}].$$

	x	y	z	B_{eq}(Å^2)
N(1)	0·42066 (5)	0·1367 (2)	0·56838 (8)	4·24 (6)
C(2)	0·44377 (6)	−0·0077 (2)	0·55169 (10)	3·98 (7)
C(3)	0·46458 (6)	−0·0288 (2)	0·64114 (11)	4·09 (7)
C(4)	0·43700 (5)	0·1340 (2)	0·65708 (9)	3·28 (6)
O(5)	0·44549 (5)	−0·0831 (2)	0·48832 (8)	5·82 (7)
S(6)	0·39179 (1)	0·09839 (4)	0·72609 (2)	2·87 (2)
C(7)	0·36646 (5)	0·3113 (2)	0·73946 (8)	2·68 (5)
C(8)	0·35442 (5)	0·3800 (2)	0·65215 (8)	2·86 (5)
C(9)	0·38348 (5)	0·5013 (2)	0·62080 (9)	3·32 (6)
C(10)	0·37506 (6)	0·5500 (2)	0·53830 (10)	4·10 (7)
C(11)	0·33778 (6)	0·4784 (2)	0·48654 (10)	4·41 (7)
C(12)	0·30921 (6)	0·3575 (2)	0·51660 (9)	4·35 (7)
C(13)	0·31729 (5)	0·3084 (2)	0·59858 (9)	3·67 (6)
C(14)	0·32187 (5)	0·2807 (2)	0·78602 (8)	2·77 (5)
C(15)	0·27866 (5)	0·3692 (2)	0·76712 (9)	3·68 (7)
C(16)	0·23993 (6)	0·3459 (2)	0·81385 (11)	4·42 (7)
C(17)	0·24400 (6)	0·2367 (2)	0·87848 (11)	4·58 (8)
C(18)	0·28692 (7)	0·1503 (2)	0·89835 (10)	4·36 (7)
C(19)	0·32580 (6)	0·1721 (2)	0·85300 (9)	3·46 (6)
C(20)	0·40140 (5)	0·4255 (2)	0·79464 (8)	2·73 (5)
C(21)	0·38881 (6)	0·5932 (2)	0·80283 (10)	3·57 (6)
C(22)	0·41839 (7)	0·6993 (2)	0·85415 (10)	4·31 (7)
C(23)	0·46087 (6)	0·6413 (2)	0·89794 (10)	4·23 (7)
C(24)	0·47314 (5)	0·4761 (2)	0·89169 (10)	4·19 (7)
C(25)	0·44392 (5)	0·3684 (2)	0·84058 (9)	3·41 (6)

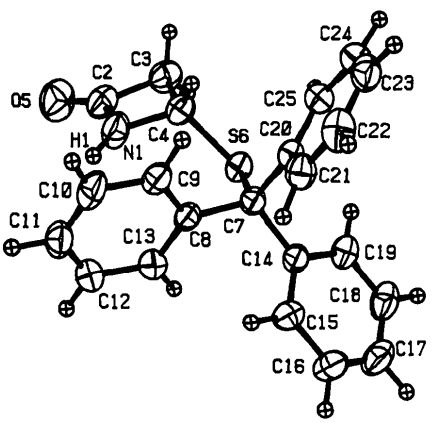

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecular structure with atom-numbering scheme. H atoms are shown with an arbitrary thermal factor. The thermal ellipsoids correspond to 50% probability levels of atomic displacement.

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

N(1)—C(2)	1·366 (2)	C(11)—C(12)	1·377 (3)
N(1)—C(4)	1·467 (2)	C(12)—C(13)	1·390 (3)
C(2)—C(3)	1·516 (2)	C(14)—C(15)	1·390 (2)
C(2)—O(5)	1·206 (2)	C(14)—C(19)	1·394 (2)
C(3)—C(4)	1·550 (2)	C(15)—C(16)	1·399 (3)
C(4)—S(6)	1·803 (2)	C(16)—C(17)	1·367 (3)
S(6)—C(7)	1·869 (1)	C(17)—C(18)	1·377 (3)
C(7)—C(8)	1·530 (2)	C(18)—C(19)	1·387 (2)
C(7)—C(14)	1·542 (2)	C(20)—C(21)	1·400 (2)
C(7)—C(20)	1·541 (2)	C(20)—C(25)	1·393 (2)
C(8)—C(9)	1·397 (2)	C(21)—C(22)	1·390 (2)
C(8)—C(13)	1·390 (2)	C(22)—C(23)	1·379 (3)
C(9)—C(10)	1·397 (2)	C(23)—C(24)	1·376 (3)
C(10)—C(11)	1·377 (3)	C(24)—C(25)	1·391 (3)
C(4)—S(6)—C(7)	103·0 (1)	C(20)—C(21)—C(22)	120·8 (2)
S(6)—C(4)—N(1)	117·9 (1)	S(6)—C(4)—C(3)	111·2 (1)
N(1)—C(4)—C(3)	86·8 (1)	C(7)—C(8)—C(9)	121·6 (1)
C(7)—C(8)—C(13)	120·0 (1)	C(9)—C(8)—C(13)	118·0 (1)
S(6)—C(7)—C(8)	105·2 (1)	S(6)—C(7)—C(20)	113·3 (1)
S(6)—C(7)—C(14)	104·1 (1)	C(8)—C(7)—C(20)	112·5 (1)
C(8)—C(7)—C(14)	114·9 (1)	C(20)—C(7)—C(14)	106·6 (1)
C(21)—C(20)—C(7)	118·9 (1)	C(21)—C(20)—C(25)	117·9 (1)
C(7)—C(20)—C(25)	123·2 (1)	C(7)—C(14)—C(15)	121·5 (1)
C(7)—C(14)—C(19)	119·7 (1)	C(15)—C(14)—C(19)	118·6 (1)
C(20)—C(25)—C(24)	120·6 (2)	O(5)—C(2)—N(1)	131·7 (2)
O(5)—C(2)—C(3)	136·4 (2)	N(1)—C(2)—C(3)	91·9 (1)
C(4)—N(1)—C(2)	95·2 (1)	C(8)—C(9)—C(10)	120·9 (1)
C(14)—C(15)—C(16)	120·1 (2)	C(14)—C(19)—C(18)	120·5 (2)
C(22)—C(23)—C(24)	119·2 (2)	C(18)—C(17)—C(16)	119·6 (2)
C(21)—C(22)—C(23)	120·5 (2)	C(25)—C(24)—C(23)	120·9 (2)
C(19)—C(18)—C(17)	120·6 (2)	C(11)—C(12)—C(13)	120·8 (2)
C(12)—C(11)—C(10)	119·5 (2)	C(15)—C(16)—C(17)	120·7 (2)
C(8)—C(13)—C(12)	120·8 (2)	C(4)—C(3)—C(2)	86·1 (1)
C(9)—C(10)—C(11)	120·2 (2)		

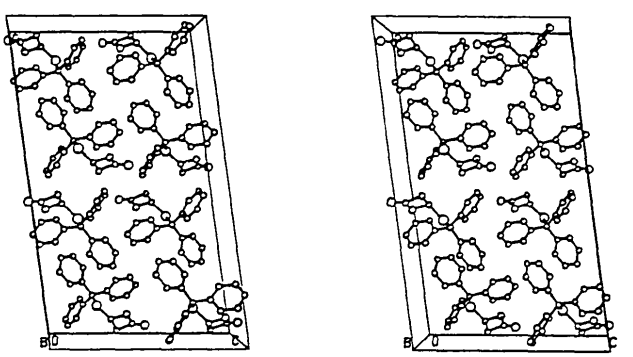

Fig. 2. Stereoscopic *PLUTO* (Motherwell, 1978) drawing of packing in the unit cell.

ured in index range $-31 \le h \le 31$, $0 \le k \le 9$, $0 \le l \le 18$. 2793 reflections with $|F_o| > 3\sigma(F)$. Three standard reflections ($\bar{1}0,0,6$, $\bar{3}18$, $12,\bar{2},0$) were monitored every 100 reflections. Variation of the standards 1%. No absorption or extinction corrections were made.

The structure was solved by direct-phase determination using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978): a phase set of the highest figure-of-merit 2·65 and residual value 17·62 gave all non-H atoms. The structure was refined on $|F_o| > 3\sigma(F)$ of $[(\sin\theta)/\lambda]_{max} = 0{\cdot}575$ Å^{-1} by blocked-diagonal-matrix least squares. H atoms located in difference maps were included at calculated positions. All the non-H atoms were refined with anisotropic thermal parameters and the H atoms with isotropic parameters. Finally, the non-H atoms were refined by full-matrix least-squares method. Function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = [\sigma^2(F) - 0{\cdot}0089|F_o| + 0{\cdot}0014|F_o|^2]^{-1}$, $R = 0{\cdot}033$, $wR = 0{\cdot}056$, $S = 2{\cdot}1$, maximum final shift-to-e.s.d. ratio 0·10 for anistropic B_{23} of N(11), and maximum and minimum electron density in final

difference map 0·27 and −0·46 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1983). Local unpublished programs on NEC PC-9801 personal computers for calculations.

Discussion. The atomic parameters are given in Table 1.* Bond distances and angles in Table 2. The molecular structure with atom-numbering scheme is shown in Fig. 1. A stereoscopic view of the packing in Fig. 2.

Without the fused five- or six-membered ring found in the penicillin or cephalosporin derivatives, the β-lactam ring in the title compound has a different conformation, though the general features are similar to those of penicillins or cephalosporins; the bond lengths of the ring decrease from 1·550 (2) to 1·366 (2) Å in the order C(3)—C(4), C(2)—C(3), N(1)—C(4), N(1)—C(2). The lengths are in good agreement with those of penicillin derivatives and cephalosporins.

Differences from penicillins and cephalosporins are observed in the angles around C(4) and N(1). The angle S(6)—C(4)—N(1) = 117·9 (1)° is significantly greater than the corresponding averaged angle 104·5 (16)° from 21 penicillins or 110·5° from two cephalosporins with *R* factors less than 10% obtained through a search of the Cambridge Structural Database (Allen, Bellard, Brice, Cartwright, Doubleday, Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979). The angle C(4)—N(1)—H(1) = 132 (1)° is also greater than the averaged angle 117 (1)° of the penicillins and 127° of the cephalosporins.

Unlike the atoms in the β-lactam rings of the penicillins or the cephalosporins, the atoms in the isolated β-lactam ring lie within only 0·006 Å from a mean plane through the ring atoms, which is as planar as the phenyl rings in the compound, whose maximum atom deviation is 0·009$_5$ Å. The mean-plane displacements of the β-lactam ring atoms of the penicillins and the cephalosporins are greater by a factor of ten.

* Lists of structure factors, anisotropic temperature factors, H-atom parameters, geometrical data concerning H atoms and best planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52035 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of 2,2′-[1,2-Ethanediylbis(oxy)]bis(benzenemethanol)

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Abstract. $C_{16}H_{18}O_4$, $M_r = 274{\cdot}31$, orthorhombic, $a = 13{\cdot}760\ (9)$, $b = 11{\cdot}732\ (7)$, $c = 9{\cdot}084\ (5)$ Å, $U = 1466{\cdot}4\ (15)$ Å^3, $D_x = 1{\cdot}243$ g cm^{-3}, $Z = 4$, space group *Pbcn* (D_{2h}^{14}) [from systematic absences $0kl$, $k = 2n + 1$; $h0l$, $l = 2n + 1$; $hk0$, $h + k = 2n + 1$], Mo $K\alpha$ radiation ($\bar{\lambda} = 0{\cdot}71069$ Å), μ(Mo $K\alpha$) = 0·83 cm^{-1}, $F(000) = 584$, final $R = 0{\cdot}0400$ for 897 observed reflections, 91 parameters. Molecules possess C_2 symmetry and are hydrogen bonded in pairs of infinite chains through the alcoholic functions.

Introduction. One strategy for the synthesis of reduced oxaazamacrocycles involves the generation of

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