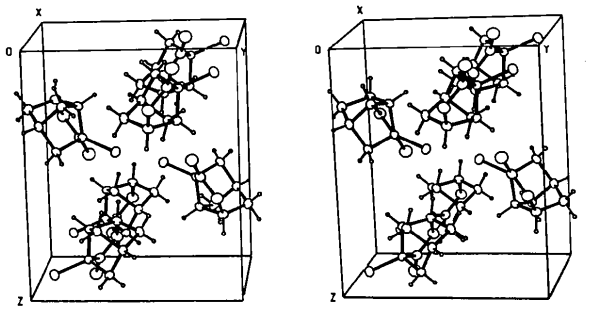

Fig. 2. Stereoview of the crystal packing in the unit cell for (2).

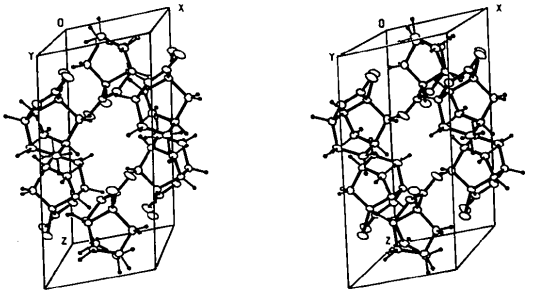

Fig. 3. Stereoview of the crystal packing in the unit cell for (4).

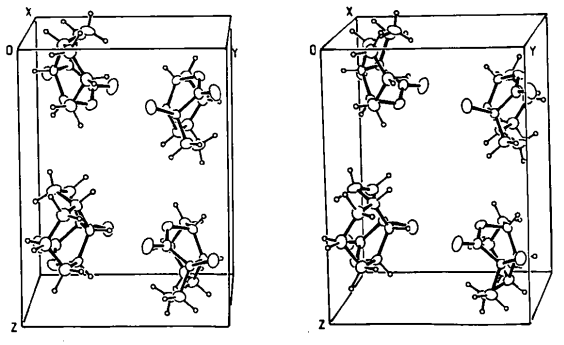

Fig. 4. Stereoview of the crystal packing in the unit cell for (6).

significantly shorter than the corresponding bond in (2) and (4), 1·511 (1) and 1·514 (3) Å, respectively. The average C_{sp^3}–C_{sp^3} bond lengths in (2) [1·531 (1), range 1·522 (1)–1·544 (1) Å] and (4) [1·529 (3), range 1·509 (3)–1·543 (3) Å] agree very well while those in (6) [1·512 (4), range 1·493 (4)–1·542 (4) Å] are slightly shorter than those in the other two structures.

The endocyclic bond angles in the five-membered rings in all three structures are significantly smaller than their expected values. Ring *A* in (2) exhibits significantly smaller angles than the other two structures, *e.g.* angles C(1)–O(1)–C(4), O(1)–C(1)–C(2) and C(2)–C(3)–C(4) in the three structures are, respectively, 110·15 (5), 107·58 (6) and 97·40 (5)° in (2), 111·6 (2), 109·4 (2) and 101·6 (2)° in (4) and 111·3 (2), 110·5 (2) and 103·6 (2)° in (6). With the exception of C(3)–C(8)–C(7) [100·9 (2)°] in ring *B* in (4), the angles are in the range 109·5 (2)–114·2 (2)°. The angles in ring *B* of fused lactone (6) are larger [range 110·5 (3)–116·9 (2)°] than those observed in ring *B* in bridged lactone (4). The crystals of the three compounds consist of discrete molecules separated by normal van der Waals distances (Figs. 2–4).

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Structure of the Sodium Salt of Penicillanic Acid

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Abstract. $Na^+.C_8H_{10}NO_3S^-$, $M_r = 223·2$, orthorhombic, $P2_12_12_1$, $a = 10·640$ (16), $b = 15·093$ (25), $c = 5·982$ (26) Å, $V = 960·6$ Å^3, $Z = 4$, $D_x = 1·54$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·54178$ Å, $\mu = 26·86$ cm^{-1}, $F(000) = 460·0$, $T = 293$ K, $R = 0·04$ for 522 observed reflexions. The conformation of this penicillin is *C*3, with the α-CH_3 in pseudo-equatorial, and the β-CH_3 and the C(3) substituent in pseudo-axial positions. This geometry is

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different from that reported for the corresponding sulfone penicillin. Bond distances and angles agree with those of other penicillin derivatives.

Table 1. *Final atomic coordinates* ($\times 10^4$) *and thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j \mathbf{a}_i^* . \mathbf{a}_j^* a_i a_j U_{ij}$$

	x	y	z	B_{eq}
S(1)	6037 (2)	8751 (1)	9427 (4)	3·01
C(2)	6129 (8)	8760 (5)	6336 (14)	2·09
C(3)	4897 (7)	8287 (4)	5572 (15)	1·55
N(4)	4682 (6)	7590 (4)	7225 (11)	1·68
C(5)	4956 (8)	7810 (5)	9596 (13)	2·14
C(6)	5478 (9)	6852 (5)	9896 (18)	3·00
C(7)	5227 (8)	6756 (5)	7325 (17)	2·32
O(8)	5408 (6)	6209 (3)	5922 (11)	3·13
C(9)	6188 (8)	9715 (5)	5554 (17)	2·85
C(10)	7268 (8)	8237 (6)	5591 (18)	3·24
C(11)	3772 (7)	8905 (5)	5369 (15)	2·04
O(12)	3514 (6)	9193 (4)	3450 (10)	2·82
O(13)	3186 (5)	9102 (3)	7123 (9)	2·43
Na(14)	3399 (3)	10001 (2)	10213 (5)	1·87

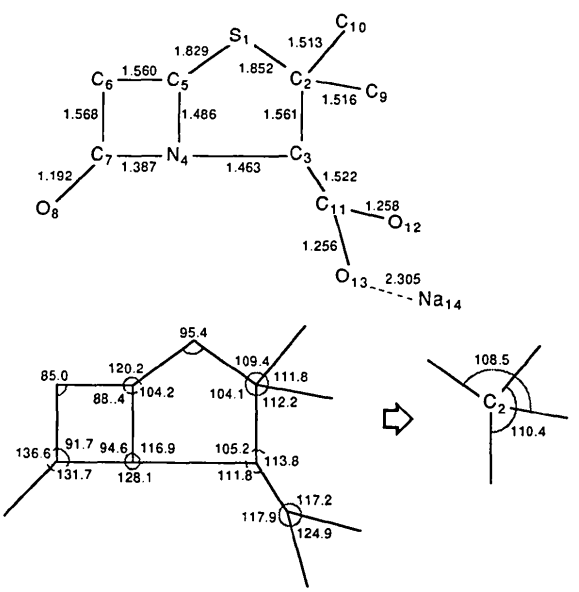

Fig. 1. Atom numbering, bond lengths (Å) and valence angles (°) (max. e.s.d.'s 0·014 Å and 0·9°).

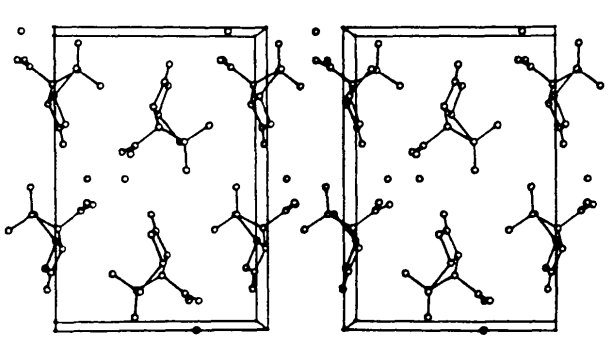

Fig. 2. Stereoview of the molecular conformation and crystal packing.

Introduction. Penicillins form one of the most important of the antibiotic families. Their antibacterial activities are limited by the growing resistance of pathogenic bacteria. This resistance proceeds from β-lactamase enzymes which transform antibiotics into biologically inactive molecules. A great deal of effort is being devoted to finding resistant antibiotics or inhibitors of β-lactamases. The resistance of some penicillins to β-lactamase enzymes is generally attributed to the nature of the C(6) side chain and to the conformation of the penam moiety (Blanpain & Durant, 1976, 1977; Blanpain, Laurent & Durant, 1977).

The particular feature of the penicillanic acid molecule is the absence of this C(6) side chain and a low affinity for the TEM β-lactamase enzymes; moreover, the affinity of the corresponding *S*-oxide molecule for the same enzyme is raised (Labia, private communication).

Experimental. Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate–methanol–butanol solution. Colourless prismatic crystal 0·22 × 0·03 × 0·02 mm for all X-ray measurements. Enraf–Nonius CAD-4 diffractometer. Lattice parameters from least-squares refinement of 25 well centred reflexions. No absorption or extinction correction. No intensity variation of standard reflexion. $2 \le 2\theta \le 65°$. 976 independent reflexions measured ($0 \le h \le 12$, $0 \le k \le 17$, $0 \le l \le 9$), 522 observed [$I \ge 2{\cdot}5\sigma(I)$]. Structure solved by direct methods (*SHELX*76; Sheldrick, 1976).

All non-H atoms found in the best *E* map. Full-matrix least-squares refinement on *F* using *SHELX*76. Eight H atoms located on a difference-Fourier map. H(53) and H(63) calculated using *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Anisotropic temperature factors (U_{ij}) for all non-H atoms and isotropic ones for H atoms. $R = 0{\cdot}04$, $wR = 0{\cdot}04$, $w = 1{\cdot}0/[\sigma^2(F)+0{\cdot}0001F^2]$, max. $(\Delta/\sigma) = -0{\cdot}216$ [U_{23} of C(9)], $S = 1{\cdot}01$, max. and min. heights in final difference-Fourier synthesis 0·29 and −0·31 e Å^{-3}. Scattering factors from *SHELX*76 (Na^+ from *International Tables for X-ray Crystallography*, 1974). Structural analysis by *XRAY*76 (Stewart *et al.*, 1976).

Discussion. Atomic parameters are given in Table 1.* Fig. 1 shows the atom numbering and bond lengths (Å) and angles (°). The observed values for the β-lactam and thiazolidine rings agree with those of other

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44562 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

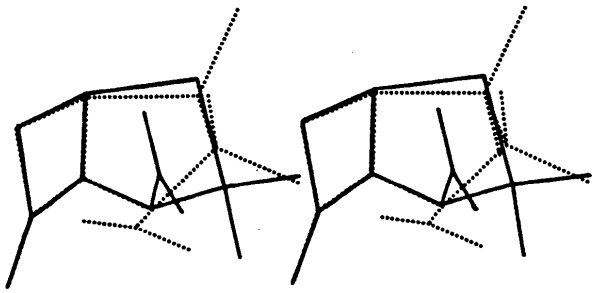

Fig. 3. Stereoscopic view of rigid fitting between penicillanic acid (full lines) and penicillanic acid 1,1-dioxide (dotted lines) by superposition of N(4), C(5), C(6), C(7) and O(8).

Table 2. *The* Na(14) *environment distances* (Å)

O(8)(x,y,z)···Na(14(x,y,z)	2·30 (1)
O(12)(x,y,z)···Na(14)($x,y,z-1$)	2·29 (1)
O(13)(x,y,z)···Na(14)($-x+1,y-\frac{1}{2},-z+\frac{1}{2}$)	2·32 (1)

penicillin derivatives (Blanpain, Nagy, Laurent & Durant, 1980). The stereochemistry of the molecule can be seen from Fig. 2. The thiazolidine ring adopts a *C*3 conformation with α-CH_3 in pseudo-equatorial and β-CH_3 and the C(3) substituent in pseudo-axial positions. This geometry is opposite to that reported for the corresponding *S*-oxide compound, penicillanic acid 1,1-dioxide, which crystallizes in an *S*1 conformation (Brenner & Knowles, 1981) with α-CH_3 in pseudo-axial and β-CH_3 and the C(3) substituent in pseudo-equatorial positions.

A rigid fitting (Lejeune, Michel & Vercauteren, 1986) of both molecules, obtained by superposition of N(4), C(5), C(6), C(7) and O(8) atoms, is shown in Fig. 3. N(4) of the β-lactam ring lies 0·38 Å from the plane of C(3), C(5) and C(7) for both molecules. However, the dihedral angle between β-lactam and thiazolidine rings is 64° for the *S*-oxide, as for oxacillin (Blanpain & Durant, 1977) and penicillin G (Dexter & Van de Veen, 1978), and 49° for the title compound.

The crystal packing is shown in Fig. 2; the Na(14) environment is summarized in Table 2.

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Structure of 9-(2-Fluorobenzyl)-6-methylamino-9*H*-purine Hydrochloride, a Novel Anticonvulsant

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Abstract. $C_{13}H_{13}FN_5^+.Cl^-$, $M_r = 293·73$, monoclinic, $P2_1/n$, $a = 13·538$ (7), $b = 7·274$ (9), $c = 15·175$ (7) Å, $\beta = 116·06$ (3)°, $V = 1342·4$ (1) Å^3, $Z = 4$, $D_m = 1·45$ (2), $D_x = 1·45$ g cm^{-3}, $\mu = 24·89$ cm^{-1}, $F(000) = 608$, room temperature, $R = 0·065$ for 2217 observed reflections. Molecules are linked by Cl···N hydrogen bonds. The phenyl groups form spiraling stacks along **b**, perpendicular stacking separation $b/2 =$

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