Acta Cryst. (1988). C**44**, 1327–1328

Structure of Pivaloyloxymethyl (3*S*,5*R*)-Penicillanate 1,1-Dioxide

BY GRACIELA PUNTE AND BLAS E. RIVERO

Laboratorio de Cristalografía, Departamento de Física, Universidad Nacional de La Plata, CC 67, 1900 *La Plata, Argentina*

AND PEDRO M. ALZARI

Immunologie Structurale, Institut Pasteur, 25 *rue du Dr Roux,* 75724 *Paris CEDEX* 15, *France*

(*Received* 28 *September* 1987; *accepted* 24 *March* 1988)

Abstract. $C_{14}H_{21}NO_7S$, $M_r = 347{\cdot}4$, monoclinic, $P2_1$, unique axis c, $a = 10{\cdot}688$ (4), $b = 12{\cdot}068$ (5), $c = 7{\cdot}078$ (6) Å, $\gamma = 70{\cdot}95$ (2)°, $V = 864{\cdot}$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}337$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 1{\cdot}76$ cm^{-1}, $F(000) = 368$, room temperature, final $R = 0{\cdot}05$ for 1297 independent reflections. The thiazolidine ring adopts the conformation with α-CH_3 axial, β-CH_3 equatorial and α-COO*R* axial, S(1) being 0·85 (1) Å out of the C(2)C(3)N(4)C(5) mean plane. The β-lactam ring is slightly puckered with C(5) 0·11 (1) Å out of the N(4)C(6)C(7) plane. The N atom shows the characteristic pyramidal geometry of penam derivatives; the distance to the plane defined by its bonded atoms is 0·42 (1) Å.

Experimental. Crystallized by slow evaporation from methanol, transparent crystal plate, 0·4 × 0·15 × 0·15 mm, D_m not measured, automated four-circle Huber diffractometer, graphite-monochromated Mo $K\alpha$, ω step scan, scan width 1°, min. scan speed 0·48° min^{-1}, max. scan speed: 4·76° min^{-1}, lattice parameters from setting angles of 20 centred reflections with $7 < 2\theta < 30°$, space group $P2_1$, unique axis c, Lorentz–polarization corrections, no absorption correction, data collected to $2\theta = 40°$, index range: $-12 \le h \le 12$, $-1 \le k \le 14$, $0 \le l \le 8$; three reflections monitored each 90 min showed an overall relative instability of 0·022; 1829 reflections measured, 1614 unique, $R_{sym} = 0{\cdot}029$, of which 1297 with $I \ge 3\sigma(I)$ considered observed. Structure solved by integrated Patterson and direct methods (Egert & Sheldrick, 1985) and difference Fourier synthesis, fragment used: penam

Table 1. *Fractional positional parameters and equivalent isotropic thermal parameters* (*Hamilton,* 1959) *with e.s.d.'s in parentheses*

	x	y	z	B_{eq}(Å^2)
S(1)	1·0328 (1)	0·1757 (1)	0·7316	6·17 (8)
C(2)	0·8734 (5)	0·2026 (5)	0·611 (1)	5·1 (3)
C(3)	0·8095 (4)	0·3366 (4)	0·6511 (8)	3·4 (2)
N(4)	0·9171 (3)	0·3855 (3)	0·6346 (7)	3·8 (2)
C(5)	1·0545 (5)	0·3058 (4)	0·637 (1)	4·5 (2)
C(6)	1·0884 (5)	0·3898 (6)	0·779 (1)	6·1 (3)
C(7)	0·9409 (6)	0·4574 (6)	0·772 (1)	6·1 (3)
O(8)	0·8650 (5)	0·5380 (5)	0·851 (1)	10·6 (3)
C(9)	0·8972 (8)	0·1775 (6)	0·400 (1)	7·7 (4)
C(10)	0·8001 (7)	0·1287 (6)	0·708 (2)	8·9 (4)
C(11)	0·6952 (5)	0·3944 (4)	0·517 (1)	3·8 (2)
O(12)	0·7022 (4)	0·4443 (4)	0·3750 (8)	5·3 (2)
O(13)	0·5859 (3)	0·3793 (3)	0·5882 (6)	3·9 (1)
C(14)	0·4660 (5)	0·4304 (5)	0·481 (1)	4·7 (3)
O(15)	0·4651 (3)	0·3569 (3)	0·3250 (6)	4·2 (2)
C(16)	0·4216 (5)	0·2653 (5)	0·363 (1)	4·4 (3)
O(17)	0·3853 (5)	0·2482 (5)	0·5162 (8)	6·8 (3)
C(18)	0·4306 (5)	0·1877 (5)	0·1920 (9)	4·5 (2)
C(19)	0·4294 (9)	0·2489 (8)	0·005 (1)	7·5 (5)
C(20)	0·3093 (7)	0·1448 (7)	0·199 (1)	7·5 (4)
C(21)	0·5558 (7)	0·0860 (7)	0·216 (2)	8·2 (4)
O(22)	1·0074 (5)	0·1921 (6)	0·930 (1)	9·1 (3)
O(23)	1·1281 (4)	0·0718 (4)	0·663 (1)	9·9 (3)

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

S(1)–O(22)	1·431 (7)	S(1)–O(23)	1·418 (6)
S(1)–C(2)	1·838 (7)	S(1)–C(5)	1·790 (5)
C(2)–C(3)	1·562 (7)	C(2)–C(9)	1·53 (1)
C(2)–C(10)	1·53 (1)	C(3)–N(4)	1·460 (6)
C(3)–C(11)	1·525 (8)	N(4)–C(5)	1·470 (6)
N(4)–C(7)	1·381 (9)	C(5)–C(6)	1·55 (1)
C(6)–C(7)	1·522 (9)	C(7)–O(8)	1·18 (1)
C(11)–O(12)	1·186 (8)	C(11)–O(13)	1·339 (7)
O(13)–C(14)	1·443 (7)	C(14)–O(15)	1·418 (8)
O(15)–C(16)	1·359 (7)	C(16)–O(17)	1·192 (9)
C(16)–C(18)	1·514 (9)	C(18)–C(19)	1·51 (1)
C(18)–C(20)	1·55 (1)	C(18)–C(21)	1·50 (1)
C(3)–H(3)	1·066 (5)	C(5)–H(5)	1·135 (7)

O(22)–S(1)–O(23)	120·5 (4)	N(4)–C(5)–C(6)	88·7 (4)
O(22)–S(1)–C(2)	107·9 (3)	C(5)–C(6)–C(7)	84·6 (5)
O(22)–S(1)–C(5)	108·6 (3)	N(4)–C(7)–C(6)	93·3 (5)
O(23)–S(1)–C(2)	110·8 (4)	N(4)–C(7)–O(8)	129·1 (7)
O(23)–S(1)–C(5)	112·7 (3)	C(6)–C(7)–O(8)	137·5 (7)
C(2)–S(1)–C(5)	92·9 (3)	C(3)–C(11)–O(12)	125·8 (5)
S(1)–C(2)–C(3)	99·5 (4)	C(3)–C(11)–O(13)	108·0 (5)
S(1)–C(2)–C(9)	109·5 (5)	O(12)–C(11)–O(13)	126·2 (6)
S(1)–C(2)–C(10)	108·2 (5)	C(11)–O(13)–C(14)	116·0 (4)
C(3)–C(2)–C(9)	111·8 (6)	O(13)–C(14)–O(15)	109·2 (5)
C(3)–C(2)–C(10)	112·8 (6)	C(14)–O(15)–C(16)	115·4 (5)
C(9)–C(2)–C(10)	114·0 (6)	O(15)–C(16)–O(17)	122·6 (6)
C(2)–C(3)–N(4)	105·5 (4)	O(15)–C(16)–C(18)	112·0 (5)
C(2)–C(3)–C(11)	112·0 (5)	O(17)–C(16)–C(18)	125·3 (6)
N(4)–C(3)–C(11)	112·7 (4)	C(16)–C(18)–C(19)	114·1 (6)
C(3)–N(4)–C(5)	119·1 (4)	C(16)–C(18)–C(20)	106·9 (5)
C(3)–N(4)–C(7)	122·0 (5)	C(16)–C(18)–C(21)	105·9 (6)
C(5)–N(4)–C(7)	93·0 (4)	C(19)–C(18)–C(20)	108·5 (6)
S(1)–C(5)–N(4)	100·9 (4)	C(19)–C(18)–C(21)	111·1 (6)
S(1)–C(5)–C(6)	117·1 (4)	C(20)–C(18)–C(21)	110·2 (6)

0108-2701/88/071327-02$03.00

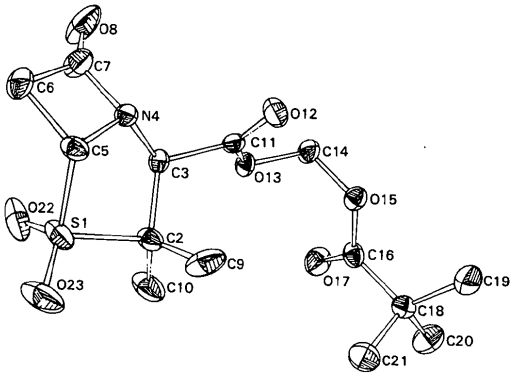

Fig. 1. *ORTEP* (Johnson, 1965) thermal-ellipsoid (20%) plot, showing atomic labelling. Hydrogens omitted.

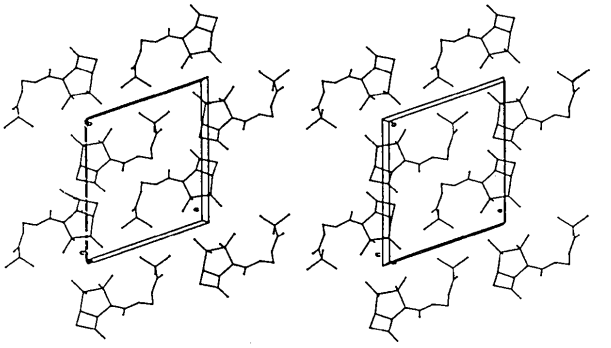

Fig. 2. Stereoview of the molecular packing along **c**.

nucleus taken from Alzari, Ronco, Rivero & Punte (1986), refined by full-matrix least squares based on F's, weighting scheme $w=1/[\sigma^2(F)+0{\cdot}00344F^2]$, H atoms (parameters not refined) positioned from ΔF synthesis when possible, the others being stereochemically determined, with isotropic thermal parameters as those of bonded C atoms. Isotropic secondary extinction of the form $F'=F\ (1-CF^2/\sin\theta)$, applied on F_c, refined value of $C=1{\cdot}82\times10^{-7}$. Final agreement factors $R=0{\cdot}050$, $wR=0{\cdot}054$ for 208 parameters refined, max. and min. heights in final difference map: 0·21 and $-0{\cdot}18$ e Å^{-3}, $(\Delta/\sigma)_{max}=0{\cdot}06$. Scattering factors from *International Tables for X-ray Crystallography* (1974), computer program used for refinement: *SHELX*76 (Sheldrick, 1976).

Fractional atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are presented in Table 1;* bond lengths and angles are listed in Table 2. Atom labelling is shown in Fig. 1; packing diagram in Fig. 2.

Related literature. This work is part of a systematic study on the influence of 1,6-substituents on the conformation of the molecular nucleus of penam derivatives (Punte, Rivero & Alzari, 1986, and references therein).

We thank Dr O. Mascaretti for the synthesis of the title compound and CONICET (Argentina) for financial support.

* Lists of observed and calculated structure factors, anisotropic thermal parameters for non-hydrogen atoms, positional and isotropic thermal parameters for hydrogen atoms and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44893 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ALZARI, P. M., RONCO, A. E., RIVERO, B. E. & PUNTE, G. (1986). *Acta Cryst.* C**42**, 1032–1034.

EGERT, E. & SHELDRICK, G. M. (1985). *PATSEE*. Fragment search by integrated Patterson and direct methods. Univ. of Göttingen, Federal Republic of Germany.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1965) *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

PUNTE, G., RIVERO, B. E. & ALZARI, P. M. (1986). *Acta Cryst.* C**42**, 1037–1038.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

International Union of Crystallography

Acta Cryst. (1988). C**44**, 1328

Microfiche Version of *Acta Crystallographica* and *Journal of Applied Crystallography*

All back volumes of both journals are now available on microfiche, including Volumes 2 and 3 of *Acta Crystallographica* which have been out of print for many years.

Orders may be placed direct with the publisher (Munksgaard International Publishers Ltd, 35 Nørre Søgade, PO Box 2148, DK-1016 Copenhagen K, Denmark) or with Polycrystal Book Service, from whom prices may also be obtained.