$C_{22}H_{18}O_2N_2$

C7—C6—C11	118.8 (5)	C27—C26—C31	119.0 (5)
C6—C7—C8	119.6 (6)	C26—C27—C28	120.1 (6)
C7—C8—C9	120.9 (6)	C27—C28—C29	119.8 (6)
C8—C9—C10	120.2 (7)	C28—C29—C30	120.4 (6)
C9—C10—C11	119.9 (7)	C29—C30—C31	120.4 (6)
C6—C11—C10	120.6 (6)	C26—C31—C30	120.3 (5)

Symmetry codes: (i) $-x, -y, 1 - z$; (ii) $-x, -y, -z$.

The title structure was solved by direct methods and refined by full-matrix least-squares calculations. H atoms were placed at geometrically idealized positions with C—H bond lengths of 0.96 Å and isotropic displacement parameters fixed at 0.06 Å^2.

Data collection: MXC diffractometer system (MacScience, 1989). Cell refinement: MXC diffractometer system (MacScience, 1989). Data reduction: *CRYSTAN* (Gilmore & Brown, 1988). Program(s) used to solve structure: *CRYSTAN*. Program(s) used to refine structure: *CRYSTAN*. Molecular graphics: *ORTEP* (Johnson, 1965) in *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1045). Services for accessing these data are described at the back of the journal.

References

Gilmore, C. J. & Brown, S. R. (1988). *Acta Cryst.* A44, 1018–1021.
Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Klingsberg, E. (1954). *Chem. Rev.* 54, 59–77.
Kollenz, G., Penn, G., Theuer, R., Fabian, W. M. F., Abd El-Nabi, H. A., Zhang, X., Peters, K., Peters, E.-M. & von Schnering, H. G. (1996). *Tetrahedron*, 52, 5427–5440.
MacScience (1989). *Operating Manual of the MXC Four-Circle Diffractometer.* MacScience Co. Ltd, Yokohama, Japan.

Two Diastereoisomers of 2-(Benzenesulfonyl)-5-benzoyl-1-oxo-3-phenyl-2,5-diazaspiro[3.4]octan-7-yl Acetate

Alessandra Forni

CNR-Centro per lo Studio delle Relazioni fra Struttura e Reattività Chimica, Via Golgi 19, I-20133 Milano, Italy. E-mail: aforni@rs6.csrsrc.mi.cnr.it

Abstract

The structures of two diastereoisomers of the title compound, $C_{27}H_{24}N_2O_6S$, were determined by single-crystal diffractometry. The two molecules are very similar as far as bond distances and angles are concerned, but more substantial differences are observed in some torsion angles.

Comment

Two diastereoisomers of the title compound were analysed by single-crystal diffractometry, that is, the 3S,4S,7R, (I), and 3R,4S,7R, (II), isomers. Both compounds were synthesized starting from the natural product 4-hydroxy-L-proline (La Rosa & Dalla Croce, 1998), hence the stereochemistry at the C7 atom is known. The X-ray analysis of these compounds was strongly suggested by the difficulty in assigning the relative configurations to the other two asymmetric centres (C3 and C4) by NMR spectroscopy. A complete knowledge of the stereochemistry of β-lactams is required in order to study the biological activity of this class of compounds.

(I)   (II)
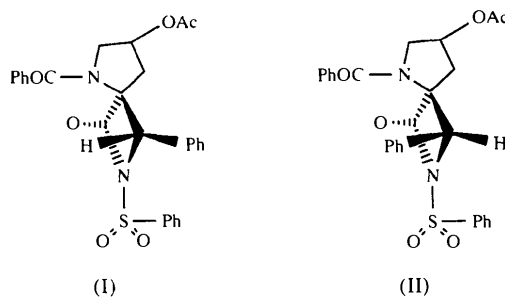
The geometries of the two molecules are very similar as far as bond distances and angles are concerned, the greatest differences being 0.042 (6) Å in the distances (C1—N2) and 5.3 (4)° in the angles (C3—N2—S22).

The geometric parameters of the two molecules (Figs. 1 and 2) are not very different from the values found in the literature for other β-lactam derivatives. We only mention that the bonds involving the N atom in the four-membered ring of compound (II) are slightly more elongated [C1—N2 1.422 (5) and N2—C3 1.516 (5) Å (Table 4) compared with C_{sp^2}—N 1.385 (19) and N—C_{sp^3} 1.464 (12) Å reported for β-lactams in *International Tables for Crystallography* (1995, Vol. C)]. This indicates, in compound (II), a reduced conjugation of the N2 atom with the C1=O21 group, and it is accompanied by some degree of pyramidalization of N2 which is out of the plane formed by C1, C3 and S22 by 0.344 (4) Å [sum of angles at N2 is 344.4 (5)°]. In compound (I), this distance decreases to 0.159 (2) Å and the angles at N2 sum to 356.5 (3)°. In this case, the four-membered ring deviates from planarity, having an average torsion angle of 5.5 (1)° and a maximum deviation from the least-squares plane of the ring of 0.046 (3) Å for C1, while in compound (II), the ring is nearly planar, the average torsion angle being 0.6 (1)°. The O atom (O21) of the β-lactam is also in the least-squares plane of the ring, the atom–plane distances being 0.071 (2) Å for (I) and 0.128 (4) Å for (II).

The four-membered ring is nearly perpendicular to the least-squares plane of the five-membered ring, the two planes forming a dihedral angle of 86 (1)° in (I) and 82 (1)° in (II). The puckering analysis (Cremer & Pople,
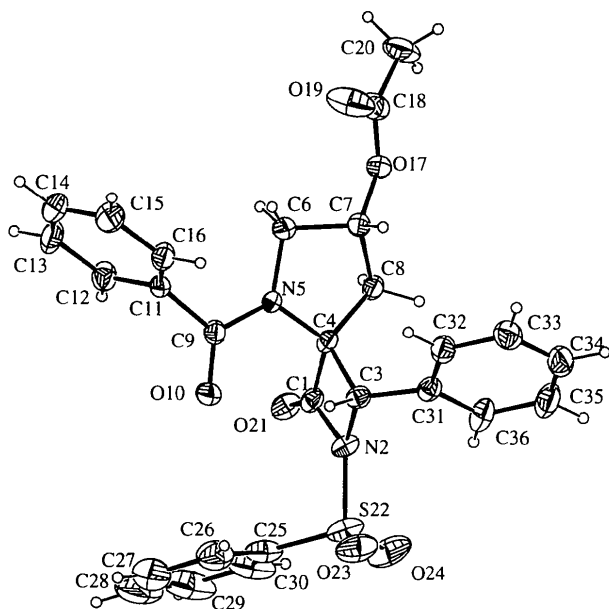
Fig. 1. *ORTEP*II plot (Johnson, 1976) of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
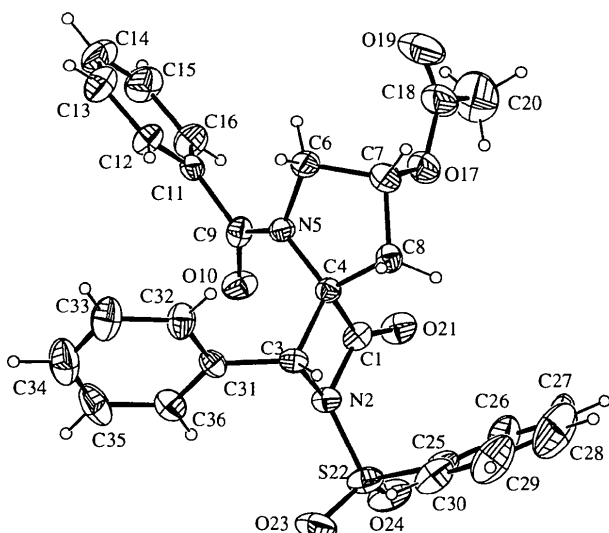

Fig. 2. *ORTEP*II plot (Johnson, 1976) of (II) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

different configuration at the C3 centre and the different packing of the crystals. The most obvious difference is the orientation of the benzenesulfonyl group with respect to the spiro group, in order to reduce the steric hindrance with the phenyl group bonded to the asymmetric C3 centre; the torsion angle of the C3—N2—S22—C25 sequence is 119.7 (3)° for (I) and −64.9 (4)° for (II). Also, the orientation of the C9=O10 bond is strongly influenced by the stereochemistry at C3; it is coplanar with the five-membered ring in (I), while in (II), it points away from the least-squares plane of this ring in order to reduce the interaction with the phenyl group bonded to C3 [O10–plane distance 1.181 (3) Å]. Another relevant difference is found in the junction of the acetoxy group at the C7 centre, the N5—C6—C7—O17 torsion angle being 148.8 (2)° for (I) and 75.2 (4)° for (II). These values correspond to a distance between O17 and the plane of the ring of 0.443 (2) Å for (I) and 1.735 (4) Å for (II). In this case, the different conformation is due only to the intermolecular interactions, because the molecular environment is nearly unchanged in both cases. In fact, both O atoms of this group are involved in weak C—H···O intermolecular hydrogen bonds. Several other weak C—H···O bonds are found in both compounds due to their large conformational flexibility.

Experimental

Crystals of the title compounds were obtained with extreme difficulty by precipitation from ethyl acetate for (I) and from acetone for (II). The best crystals were needle-shaped and were separate from a common aggregation centre.

Compound (I)

Crystal data

$C_{27}H_{24}N_2O_6S$	Mo $K\alpha$ radiation
$M_r = 504.54$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 29
$P2_12_12_1$	reflections
$a = 10.032 (2)$ Å	$\theta = 2.22–9.40°$
$b = 10.787 (3)$ Å	$\mu = 0.176$ mm^{-1}
$c = 22.947 (4)$ Å	$T = 293 (2)$ K
$V = 2483.2 (10)$ Å^3	Needle
$Z = 4$	$0.40 \times 0.15 \times 0.15$ mm
$D_x = 1.350$ Mg m^{-3}	Colourless
D_m not measured	

Data collection

Siemens *P*4 diffractometer	$\theta_{max} = 24.99°$
$\omega/2\theta$ scans	$h = -11 \rightarrow 11$
Absorption correction: none	$k = -1 \rightarrow 11$
7295 measured reflections	$l = -25 \rightarrow 27$
3461 independent reflections	3 standard reflections
2589 reflections with	every 197 reflections
$I > 2\sigma(I)$	intensity decay: 5.12%
$R_{int} = 0.032$	

1975) of the five-membered ring gives the parameters $Q = 0.362 (3)$ Å and $\varphi = 302.6 (4)°$ for (I), and $Q = 0.367 (5)$ Å and $\varphi = 100.7 (7)°$ for (II), Q being the puckering amplitude and φ the phase angle. These values correspond to a nearly twisted conformation for (I) and an intermediate twisted-envelope conformation for (II).

More relevant differences between the diastereoisomers are observed in the torsion angles, due to both the

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.167$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{min} = -0.183$ e Å^{-3}
$wR(F^2) = 0.066$	Extinction correction: none
$S = 0.991$	Scattering factors from
3461 reflections	International Tables for
325 parameters	Crystallography (Vol. C)
H atoms not refined	Absolute structure: Flack
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2]$	(1983)
where $P = (F_o^2 + 2F_c^2)/3$	Flack parameter $= -0.09$ (9)
$(\Delta/\sigma)_{max} = 0.002$	

Table 1. Selected bond lengths (Å) for (I)

C1—O21	1.196(3)	C4—N5	1.449(3)
C1—N2	1.380(3)	C4—C8	1.521(3)
C1—C4	1.538(4)	N5—C6	1.466(3)
N2—C3	1.497(3)	C6—C7	1.521(3)
C3—C4	1.589(4)	C7—C8	1.514(3)

Compound (II)

Crystal data

$C_{27}H_{24}N_2O_6S$	Mo $K\alpha$ radiation
$M_r = 504.54$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 27
$P2_12_12_1$	reflections
$a = 6.237$ (1) Å	$\theta = 2.35–7.51°$
$b = 18.928$ (2) Å	$\mu = 0.173$ mm^{-1}
$c = 21.321$ (3) Å	$T = 293$ (2) K
$V = 2517.0$ (6) Å^3	Needle
$Z = 4$	$0.52 \times 0.22 \times 0.10$ mm
$D_x = 1.331$ Mg m^{-3}	Colourless
D_m not measured	

Data collection

Siemens $P4$ diffractometer	$\theta_{max} = 23.00°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 6$
Absorption correction: none	$k = 0 \rightarrow 20$
3978 measured reflections	$l = -23 \rightarrow 23$
3492 independent reflections	3 standard reflections
2256 reflections with	every 97 reflections
$I > 2\sigma(I)$	intensity decay: $<4\%$
$R_{int} = 0.025$	

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.176$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.052$	$\Delta\rho_{min} = -0.161$ e Å^{-3}
$wR(F^2) = 0.083$	Extinction correction: none
$S = 1.089$	Scattering factors from
3492 reflections	International Tables for
325 parameters	Crystallography (Vol. C)
H atoms not refined	Absolute structure: Flack
$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2]$	(1983)
where $P = (F_o^2 + 2F_c^2)/3$	Flack parameter $= 0.09$ (14)
$(\Delta/\sigma)_{max} = 0.007$	

Table 2. Selected bond lengths (Å) for (II)

C1—O21	1.185(5)	C4—N5	1.447(5)
C1—N2	1.422(5)	C4—C8	1.538(5)
C1—C4	1.535(6)	N5—C6	1.472(5)
N2—C3	1.516(5)	C6—C7	1.511(5)
C3—C4	1.571(5)	C7—C8	1.513(5)

Because of the bad shape of the crystals, the data collections were limited in 2θ. However, the reduced thermal motion allowed the determination of both structures with sufficient accuracy.

For both compounds, data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL93* and *PARSTCIF* (Nardelli, 1991). Molecular parameters were calculated using *PARST* (Nardelli, 1983, 1995).

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Fait, J. (1991). *XSCANS Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
La Rosa, C. & Dalla Croce, P. (1998). In preparation.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Nardelli, M. (1991). *PARSTCIF. Program for Creating a CIF from the Output of PARST.* University of Parma, Italy.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

8-Ammonioquinolinium Dichloride

Yaw Kai Yan, Ngoh Khang Goh and Lian Ee Khoo

Division of Chemistry, National Institute of Education, Nanyang Technological University, 469 Bukit Timah Road, Singapore 259756, Republic of Singapore. E-mail: yanyk@nievax.nie.ac.sg

Abstract

The crystals of $C_9H_{10}N_2^{2+}.2Cl^-$ contain 8-ammonioquinolinium cations, which are stacked along the c axis, and chloride anions, which occupy the channels between the stacks. There is extensive N—H···Cl hydrogen bonding between the cations and anions, as well as weak anion–cation Cl···H—C_{sp^2} interactions.