

Diphenylmethyl 1-oxo-1-penicillanate

Guo-Yi Bai,^{a*} Hong-Wei Peng,^a
Xin-Ying Qin,^a Yue-Cheng
Zhang^b and Tao Zeng^b^aCollege of Chemistry and Environmental
Science, Hebei University, Hebei 071002,
People's Republic of China, and ^bSchool of
Chemical Engineering and Technology, Tianjin
University, Tianjin 300072, People's Republic
of China

Correspondence e-mail: baiguoyi@hotmail.com

Key indicators

Single-crystal X-ray study
 $T = 113\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.038
 wR factor = 0.086
Data-to-parameter ratio = 19.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{21}\text{H}_{21}\text{NO}_4\text{S}$, is the key intermediate in
the synthesis of tazobactam. The crystal structure is stabilized
by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.Received 20 August 2006
Accepted 30 August 2006

Comment

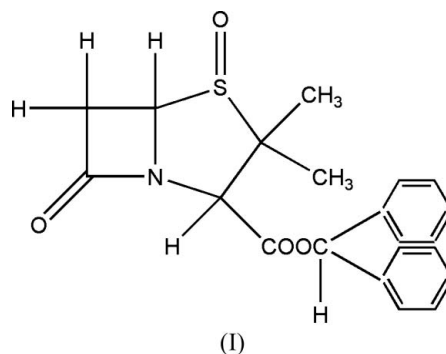
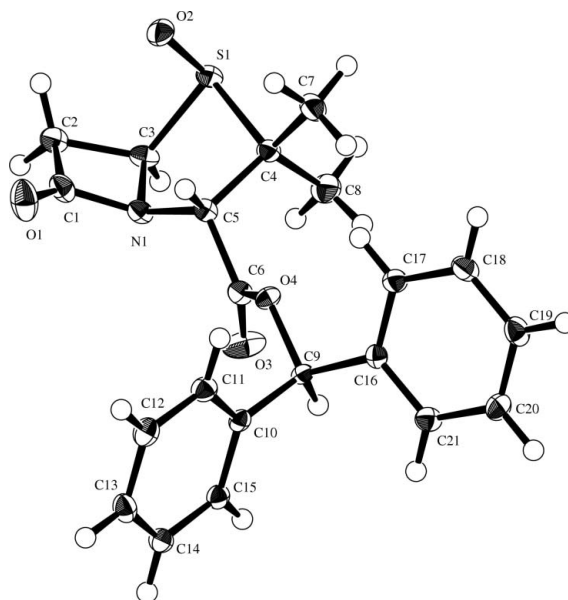
Tazobactam is a widely used β -lactamase inhibitor (Bai *et al.*,
2001). The title compound, (I), is the key intermediate for the
synthesis of tazobactam and its structure is reported here
(Fig. 1).All bond lengths and angles in (I) are within normal ranges
(Allen *et al.*, 1987). The crystal structure is stabilized by weak
 $\text{C}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds (Fig. 2 and Table 1).

Figure 1
The molecular structure of (I), showing the atom-numbering scheme and
30% probability displacement ellipsoids.

The five-membered thiazolidine ring (N1, C3, S1, C4 and C5) adopts an envelope conformation (Spek, 2003), with an r.m.s. deviation from the mean plane through atoms N1, C3, C4 and C5 of 0.019 Å and the S1 atom 0.830 (2) Å from that plane. The four-membered azetidine ring is in a distorted planar conformation, with an r.m.s. deviation from the plane through atoms N1, C1, C2 and C3 of 0.059 Å. The dihedral angle between the N1/C3/C4/C5 and N1/C1/C2/C3 planes is 42.29 (9)°. The two phenyl rings of the diphenylmethyl group are inclined at an angle of 77.13 (5)°.

Experimental

The title compound was prepared according to the procedure of Bai *et al.* (2001), using chiral (+)-6-aminopenicillanic acid as the starting material. Colourless single crystals of (I) were grown by slow evaporation of an acetone solution.

Crystal data

$C_{21}H_{21}NO_4S$	$Z = 4$
$M_r = 383.45$	$D_x = 1.382 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_1$	Mo $K\alpha$ radiation
$a = 7.9978 (14) \text{ \AA}$	$\mu = 0.20 \text{ mm}^{-1}$
$b = 9.5959 (17) \text{ \AA}$	$T = 113 (2) \text{ K}$
$c = 24.020 (5) \text{ \AA}$	Block, colorless
$V = 1843.4 (6) \text{ \AA}^3$	$0.24 \times 0.22 \times 0.10 \text{ mm}$

Data collection

Rigaku Saturn diffractometer	16269 measured reflections
ω scans	4730 independent reflections
Absorption correction: numerical (NUMABS; Higashi, 1999)	4400 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.940$, $T_{\max} = 0.980$	$R_{\text{int}} = 0.044$
	$\theta_{\text{max}} = 28.7^\circ$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
$wR(F^2) = 0.086$	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
$S = 1.06$	Extinction correction: <i>SHELXL97</i>
4730 reflections	Extinction coefficient: 0.0277 (16)
248 parameters	Absolute structure: Flack (1983), 2021 Friedel pairs
H-atom parameters constrained	Flack parameter: $-0.05 (6)$
$w = 1/[\sigma^2(F_o^2) + (0.0396P)^2 + 0.2494P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C8-H8C\cdots O1^i$	0.98	2.44	3.320 (2)	150
$C15-H15\cdots O2^{ii}$	0.95	2.40	3.160 (2)	137
$C21-H21\cdots O2^{ii}$	0.95	2.48	3.323 (2)	149

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

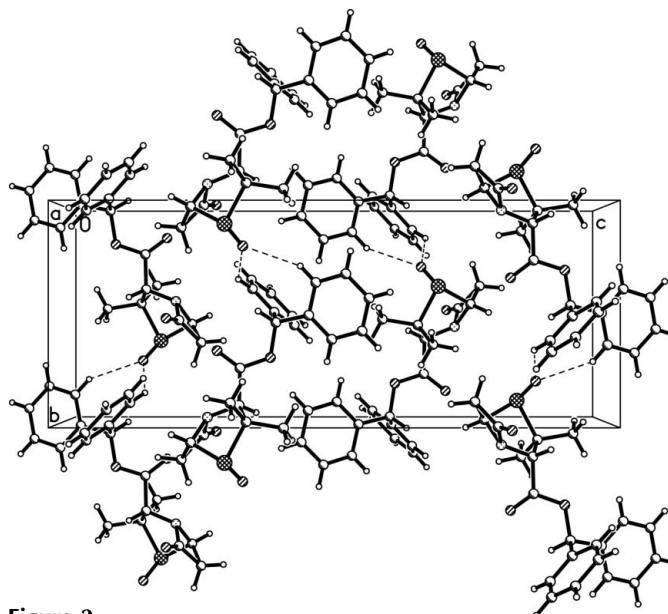


Figure 2

Packing diagram for (I), with C—H...O hydrogen bonds drawn as dashed lines.

All H atoms were refined using a riding model, with C—H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic, C—H = 1.00 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH, C—H = 0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH₂ and C—H = 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for CH₃ H atoms.

Data collection: *CrystalClear* (Rigaku/MSK, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSK, 2005) and *PLATON* (Spek, 2003).

Financial support by the Science Project of the Hebei Education Department (grant No. 2005350) and the Science Foundation of Hebei University (grant No. 2005046) is gratefully acknowledged.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bai, G. Y., Chen, L. G., Li, Y. & Cao, L. (2001). *Fine Chem.* **18**, 634–637.
- Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MSK (2005). *CrystalStructure* (Version 3.7.0) and *CrystalClear* (Version 1.36). Rigaku/MSK Inc., The Woodlands, Texas, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.