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Benzhydryl 2-(3-benzyl-7-oxo-4-thia-2,6-diazabicyclo[3.2.0]hept-2-en-6-yl)-3-morpholinobut-2-enoate

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Key indicators

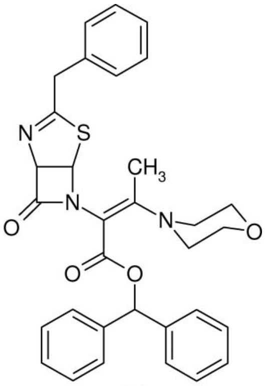
Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.081
Data-to-parameter ratio = 18.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{32}H_{31}N_3O_4S$, is an important intermediate in the synthesis of cephem compounds, which can be converted into broad-spectrum cephalosporin antibiotics. Intermolecular C—H···O and C—H···S contacts are observed in the crystal structure.

Comment

The title compound, (I) (Fig. 1), is used in the preparation of 3-hydroxy-7-β-phenylacetylaminocephem-4-carboxylic acid diphenylmethyl ester (Nakanokoshi *et al.*, 1998), which is an important intermediate for manufacturing Ceftizoxime, Ceftibuten (Hamashima *et al.*, 1987) and other broad-spectrum antibiotics (Yoshida *et al.*, 1999; Lee *et al.*, 2000). In the solid state, the C29/C30/C31/C32/N3/O4 ring adopts a chair conformation, with atoms O4 and N3 displaced by 0.660 (1) and 0.632 (1)Å, respectively, on opposite sides of the mean plane defined by C29–C32. The dihedral angle between the two phenyl rings attached to C14 is 79.0 (1)°.



(I)

Intermolecular C—H···O and C—H···S contacts are observed in the crystal structure (Table 1).

Experimental

Triethylamine (0.8 g, 8 mmol) and methanesulfonyl chloride (0.6 g, 5 mmol) were added to a mixture of diphenylmethyl 2-(3-benzyl-6-oxo-2-thia-4,7-diazabicyclo[3.2.0]hept-3-en-7-yl)-3-hydroxy-2-butenoate (2.0 g, 4 mmol) and dichloromethane (25 ml) and stirred for 2 h at 253 K. Morpholine (0.43 g, 5 mmol) was then added to the reaction mixture, and stirring was continued for 3 h at 263 K. Water (20 ml) was then added, and the organic layer was separated, dried over anhydrous magnesium sulfate and concentrated under reduced pressure. Purification of the resulting residue by chromatography over silica gel (eluting with ethyl acetate and petroleum, 2:1 v/v) gave 1.6 g of compound (I). Crystallization of (I) (1.0 g) from methanol (15 ml) gave 0.7 g of colourless crystals.

Crystal data

$C_{32}H_{31}N_3O_4S$
$M_r = 553.66$
Orthorhombic, $P2_12_12_1$
$a = 8.648$ (3) Å
$b = 12.278$ (4) Å
$c = 26.246$ (8) Å
$V = 2786.6$ (15) Å^3
$Z = 4$
$D_x = 1.320$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.16$ mm^{-1}
$T = 113$ (2) K
Block, colourless
0.24 × 0.20 × 0.18 mm

Data collection

Rigaku Saturn CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min} = 0.963$, $T_{\max} = 0.972$
25624 measured reflections
6676 independent reflections
6023 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.051$
$\theta_{\max} = 27.9°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.081$
$S = 1.03$
6676 reflections
364 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0078 (6)
Absolute structure: Flack (1983), 3304 Friedel pairs
Flack parameter: −0.01 (6)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2$^{\rm i}$	1.00	2.54	3.444 (2)	150
C16—H16···O2$^{\rm i}$	0.95	2.48	3.350 (2)	152
C23—H23···S1$^{\rm ii}$	0.95	2.74	3.543 (2)	142

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-1, y, z$.

H atoms were placed in calculated positions, with C—H = 0.95 (aromatic H), 0.98 (CH_3), 0.99 (CH_2) or 1.00 Å (CH), and constrained to ride on their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}({\rm C})$ for the methyl group. The methyl group was allowed to rotate about its local threefold axis.

Data collection: *CrystalClear* (Rigaku, 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/MSC, 2005).

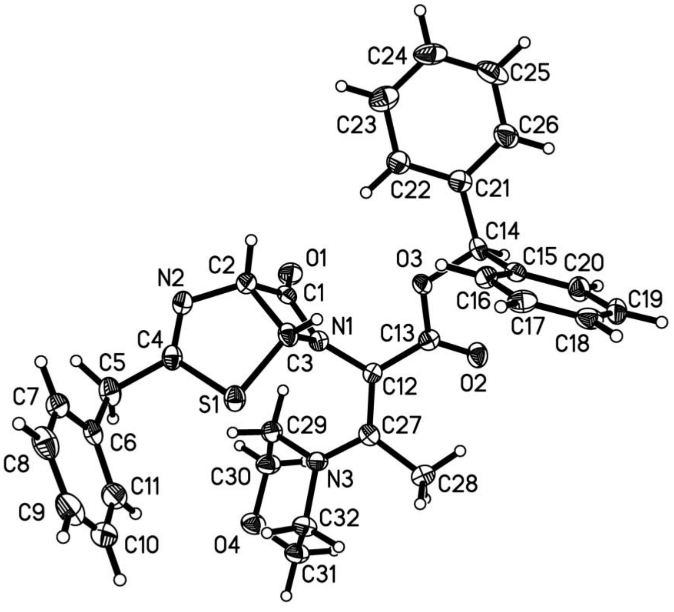


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radius.

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