organic papers

1,3,3,4,4-Pentaphenyl-β-lactam

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In the title compound, $C_{33}H_{25}NO$, the β-lactam is planar in the two crystallographically independent molecules in the asymmetric unit. The phenyl ring attached at the N atom is coplanar with the β-lactam, while the orientations of the two phenyl rings attached at each of the two C atoms of the β-lactam are determined by the Csp^3 nature of these atoms.

Comment

Photoinduced electron transfer (PET) reactions of nitro-aromatic compounds with alkynes and alkenes have been of research interest (Scheinbaum, 1964; D'Auria *et al.*, 1996). Recently, we have focused on PET reactions of nitrobenzene derivatives with 1,2-diphenylacetylene (Tian *et al.*, 2002). Among the synthesized products are benzenamine derivatives whose structures have been reported previously (Usman *et al.*, 2002*a*,*b*). We report here the crystal structure of the title compound, (I), which was isolated from the PET reaction of nitrobenzene with 1,2-diphenylacetylene.

(I)
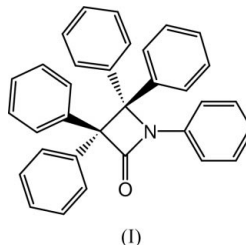
The asymmetric unit of (I) contains two crystallographically independent molecules, *A* and *B*, which are related by a local pseudo-rotation axis. The bond lengths and angles in molecules *A* and *B* (Fig. 1) agree with each other. The values are within normal ranges (Allen *et al.*, 1987), except for the bond lengths and angles exocyclic to and those involving the β-lactam moiety (C1/C2/C3/N1) (Table 1). The C—C and C—N bond lengths are significantly larger than those of the corresponding typical values, while the angles C1—N1—C3 and N1—C1—C2 are also significantly larger than those of the C1—C2—C3 and N1—C3—C2. These differences are due to the interactions between the attached bulky substituents.

The β-lactam is nearly planar, with maximum deviations of 0.029 (4) Å and 0.017 (4) Å from their mean plane in molecules *A* and *B*, respectively, while the dihedral angle between the N1/C1/C2 and N1/C2/C3 planes is 173.8 (4)° in molecule *A* and 176.3 (4)° molecule *B*. This planarity is mainly due to the sp^2 states of atoms C1 and N1. Atom O1 lies close to the β-lactam plane, with deviations of 0.083 (3) and 0.057 (3) Å in molecules *A* and *B*, respectively.
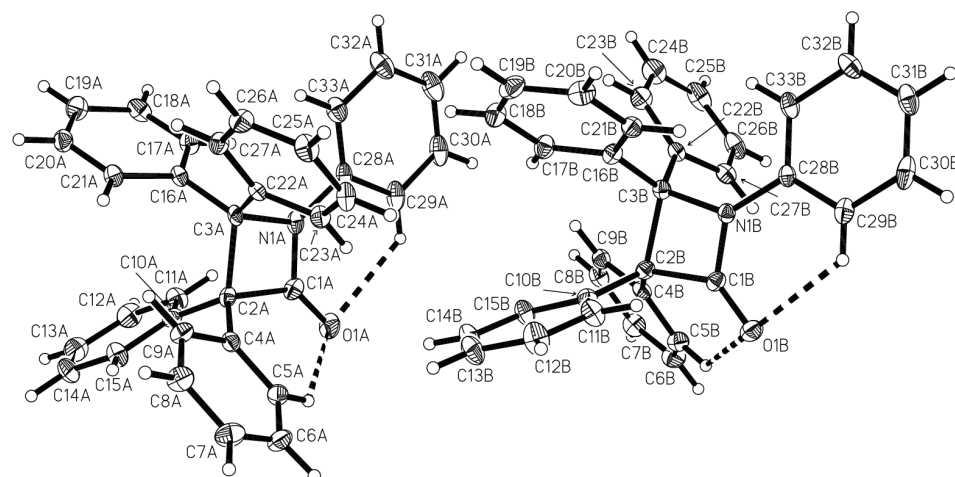
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Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. C—H⋯O interactions are shown as dashed lines.

The phenyl ring (C28–C33) attached at N1 is almost coplanar with the β-lactam. The dihedral angle between these two rings is 3.2 (3)° in molecule A and 9.8 (3)° in molecule B.

The orientations of the two phenyl rings attached at each of C2 and C3 are determined by the sp^3 states of these two C atoms. Except for the C1—C2—C3 and N1—C3—C2 angles, the average bond angles subtended at atoms C2 and C3 are 114.0 and 113.5° in molecules A and B. The torsion angles C4—C2—C3—C22 and C10—C2—C3—C16 are 9.8 (4) and 7.7 (4)° in molecule A, and 4.4 (4) and 4.9 (4)° in molecule B, indicating an eclipsed configuration of atoms C2 and C3.

In molecule A, phenyl rings C4–C9 and C10–C15, attached at C2, make dihedral angles of 51.0 (3) and 78.9 (3)°, respectively, with the β-lactam. These angles for phenyl rings C16–C21 and C28–C33, attached at C3, are 61.5 (3) and 81.1 (3)°, respectively. In molecule B, the corresponding angles are comparable [53.9 (3), 76.8 (3), 59.9 (3) and 86.6 (3)°, respectively].

In molecules A and B (Fig. 1), atom O1 is involved in two intramolecular C—H⋯O interactions (C5—H5⋯O1 and C29—H29⋯O1), forming O1—C1—C2—C4—C5—H5 and O1—C1—N1—C28—C29—H29 six-membered rings. In the crystal structure, symmetry-related pairs of A and B molecules form molecular dimers (Fig. 2) by intermolecular C6—H6⋯O1 interactions (Table 2 and Fig. 2).

Experimental

The title compound was isolated from the PET reaction of nitrobenzene in the presence of an excess amount of 1,2-diphenylacetylene in benzene solution, by column chromatography on silica gel. Single crystals suitable for X-ray crystallographic measurement were prepared by slow evaporation of the solvent from a petroleum ether–ethyl acetate solution (4:1 v/v).

Crystal data

$C_{33}H_{25}NO$	$Z = 4$
$M_r = 451.54$	$D_x = 1.249$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.3827$ (1) Å	Cell parameters from 8086
$b = 14.5395$ (2) Å	reflections
$c = 17.3675$ (1) Å	$\theta = 2.5$–28.3°
$\alpha = 105.145$ (1)°	$\mu = 0.07$ mm^{-1}
$\beta = 102.943$ (1)°	$T = 183$ (2) K
$\gamma = 98.9522$ (5)°	Block, colorless
$V = 2401.68$ (4) Å^3	$0.50 \times 0.48 \times 0.40$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	8200 independent reflections
	4864 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.083$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 25.0°$
	$h = -11 \rightarrow 12$
$T_{min} = 0.903$, $T_{max} = 0.964$	$k = -17 \rightarrow 17$
11565 measured reflections	$l = -16 \rightarrow 20$

Refinement

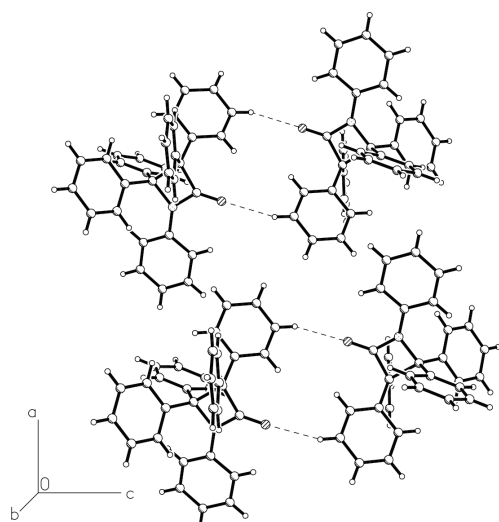|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1362P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.266$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.96$	$\Delta\rho_{max} = 0.68$ e Å^{-3}
8200 reflections	$\Delta\rho_{min} = -0.64$ e Å^{-3}
632 parameters	Extinction correction: *SHELXL*97
H-atom parameters constrained	Extinction coefficient: 0.030 (4)

Figure 2
Packing diagram of the title compound, viewed down the b axis, showing the dimer formations.

Table 1
Selected geometric parameters (Å, °).

N1A—C1A	1.355 (5)	N1B—C1B	1.367 (5)
N1A—C28A	1.440 (5)	N1B—C28B	1.425 (4)
N1A—C3A	1.500 (4)	N1B—C3B	1.491 (4)
C1A—C2A	1.546 (5)	C1B—C2B	1.551 (5)
C2A—C10A	1.523 (5)	C2B—C10B	1.518 (5)
C2A—C4A	1.529 (5)	C2B—C4B	1.528 (5)
C2A—C3A	1.647 (5)	C2B—C3B	1.652 (5)
C3A—C16A	1.508 (5)	C3B—C16B	1.519 (5)
C3A—C22A	1.540 (5)	C3B—C22B	1.538 (5)
C1A—N1A—C3A	96.7 (3)	C1B—N1B—C3B	96.5 (3)
N1A—C1A—C2A	94.2 (3)	N1B—C1B—C2B	94.2 (3)
C1A—C2A—C3A	83.8 (3)	C1B—C2B—C3B	83.5 (3)
N1A—C3A—C2A	85.0 (2)	N1B—C3B—C2B	85.7 (2)
C10A—C2A—C4A	112.7 (3)	C10B—C2B—C4B	113.3 (3)
C10A—C2A—C1A	111.2 (3)	C10B—C2B—C1B	112.4 (3)
C4A—C2A—C1A	115.3 (3)	C4B—C2B—C1B	114.2 (3)
C10A—C2A—C3A	115.5 (3)	C10B—C2B—C3B	114.4 (3)
C4A—C2A—C3A	115.4 (3)	C4B—C2B—C3B	115.8 (3)
N1A—C3A—C16A	114.2 (3)	N1B—C3B—C16B	113.5 (3)
N1A—C3A—C22A	109.5 (3)	N1B—C3B—C22B	111.1 (3)
C16A—C3A—C22A	115.4 (3)	C16B—C3B—C22B	115.1 (3)
C16A—C3A—C2A	113.6 (3)	C16B—C3B—C2B	113.5 (3)
C22A—C3A—C2A	115.4 (3)	C22B—C3B—C2B	114.5 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5A—H5A···O1A	0.93	2.53	3.191 (5)	128
C5B—H5B···O1B	0.93	2.52	3.162 (5)	127
C6A—H6A···O1A[i]	0.93	2.54	3.432 (6)	160
C6B—H6B···O1B[ii]	0.93	2.60	3.303 (6)	133
C29A—H29A···O1A	0.93	2.45	3.067 (6)	124
C29B—H29B···O1B	0.93	2.47	3.070 (6)	122

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $1 - x, -y, 1 - z$.

The H atoms were fixed geometrically and were treated as riding atoms on the parent C atoms, with C_{ar}—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). Owing to a large fraction of weak data at higher angles, the 2θ maximum was limited to 50°.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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