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3-Phenoxy-1,4-diphenylazetidin-2-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.114
Data-to-parameter ratio = 16.9

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

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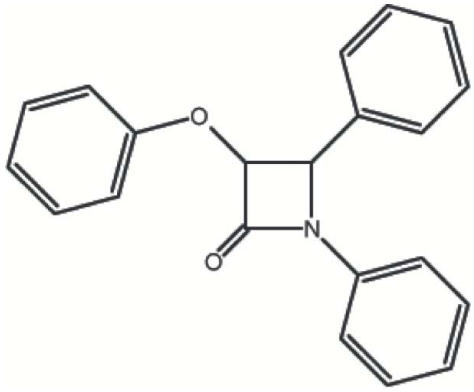
In the structure of the title compound, $C_{21}H_{17}NO_2$, the four-membered β-lactam ring is nearly planar, with long C—C distances of 1.526 (3) and 1.567 (3) Å. The angles between the planes of the three phenyl rings and this group are 81.12 (8), 3.02 (8) and 49.88 (7)°. The dihedral angles between the planes of the phenyl rings are 83.72 (7), 68.03 (7) and 47.06 (6)°. The crystal structure is stabilized by inter- and intramolecular C—H···O hydrogen-bond interactions.

Comment

The development of new synthetic methods for the efficient construction of biologically active compounds is an important field in organic chemistry (Alcaide *et al.*, 2001). The synthesis of diversely substituted monocyclic β-lactams has been of considerable interest to the synthetic community in the past few decades (Jarrahpour *et al.*, 2004*a*,*b*; Jarrahpour & Jahaniani, 2005; Gomez-Gallego *et al.*, 2000). Because of recent developments using β-lactams as synthons for several biologically active compounds, research on this topic has gained tremendous attention (Neu, 1992; Davies, 1994; Rosenblum *et al.*, 1998).



(I)

Some of the synthetic monocyclic β-lactams display interesting biological activities, such as inhibition of prostate-specific antigens (Adlington *et al.*, 1997), thrombin (Han *et al.*, 1995), tryptase (Bisacchi *et al.*, 2004; Sutton *et al.*, 2004; Qian *et al.*, 2002), human cytomegalovirus protein (Borthwick *et al.*, 1998), cholesterol absorption (Burnett, 2004; Clader, 2004) human leukocyte elastase (Cainelli *et al.*, 2003), cysteine protease (Zhou *et al.*, 2003), porcine pancereatic elastease (PPE) (Gerard *et al.*, 2004), anticancer activities (Banik *et al.*, 2003), anti HIV-1 protease (Sperka *et al.*, 2005) and antibacterial activity (Turos *et al.*, 2002; Coates *et al.*, 2003). In

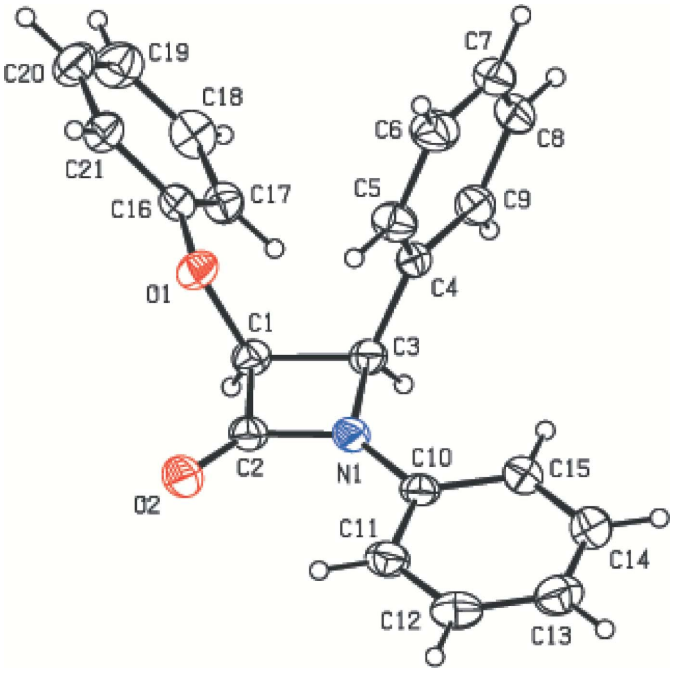


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids

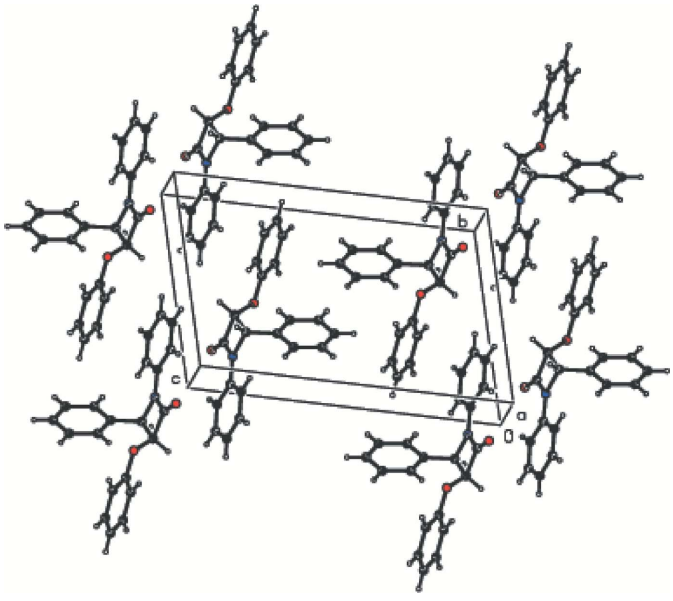


Figure 2
A view of the packing of (I), approximately along the *a* axis.

addition to their diverse current uses as pharmaceuticals, β-lactams are of interest as synthetic building blocks (Kuznetsova *et al.*, 2004; Alcaide & Almendros, 2001; Palomo *et al.*, 2000; Ojima, 1995). In this study, the molecular structure of 3-phenoxy-1,4-diphenylazetidin-2-one, (I), has been determined and is reported here.

The molecular structure of (I) is shown in Fig. 1. The four-membered β-lactam ring of (I) is nearly planar, with a maximum deviation of 0.022 (2) Å for carbonyl atom C2. The bond lengths in the lactam ring are comparable with those found in previous similar studies (Ercan *et al.*, 1996*a*,*b*; Kabak *et al.*, 1999). The N1—C2 bond, conjugated with the carbonyl group, is shorter than the N1—C3 and N1—C10 bonds (Table 1), and these results seem to be in agreement with those found in similar molecules (Ercan *et al.*, 1996*a*,*b*; Ülkü *et al.*, 1997). However, the C2═O2 bond is slightly longer than those found in the literature [1.198 (12) Å; Allen *et al.*, 1987].

In the present work, the C2—C1 and C1—C3 bond lengths [1.526 (3) and 1.567 (3) Å, respectively] are comparable with those reported by Kabak *et al.* (1999) [1.543 (7) and 1.571 (6) Å], but are slightly longer than those reported by Ercan *et al.* (1996*a*,*b*) and Ülkü *et al.* (1997) [1.536 (5), 1.55 (2), 1.535 (5), 1.558 (4), 1.60 (2) and 1.566 (5) Å]. The *A*/*B*, *A*/*C* and *B*/*C* dihedral angles between the phenyl rings *A* (C4–C9), *B* (C10–C15) and *C* (C16–C21) are 83.72 (7), 68.03 (7) and 47.06 (6)°, respectively. The angles between the planes of the *A*, *B* and *C* phenyl rings and the four-membered β-lactam ring are 81.12 (8), 3.02 (8) and 49.88 (7)°, respectively. The sum of the bond angles about N1 is 360.09°.

Fig. 2 shows the packing of (I), approximately along the *a* axis. The crystal structure of (I) is stabilized by inter- and intramolecular C—H···O hydrogen-bonding interactions (Table 2).

Experimental

A solution of phenoxyacetyl chloride (0.35 ml, 0.43 g, 2.53 mmol) in CH_2Cl_2 (15 ml) was added slowly to a solution of benzalaniline (0.63 g, 3.50 mmol) and triethylamine (1.38 ml, 0.99 g, 10.00 mmol) in CH_2Cl_2 (15 ml) at 258 K. The reaction mixture was then allowed to warm to room temperature and was stirred overnight. It was then washed with water (2 × 20 ml), saturated sodium bicarbonate solution (15 ml) and brine (15 ml), and dried (Na_2SO_4). The organic solvent was evaporated to give the crude monocyclic β-lactam, which was purified by column chromatography over silica gel. The title compound was recrystallized from dichloromethane to give colourless prismatic stick-shaped crystals. The IR spectrum showed the characteristic absorption of β-lactam carbonyl at 1755.0 cm^{-1}. The ^1H NMR spectrum showed a multiplet for aromatic protons at 6.69–7.67 p.p.m. CHCO appeared as a doublet at 5.58 p.p.m (J = 4.87 Hz) and the CHN proton at 5.41 (J = 4.87 Hz). The ^{13}C NMR spectrum showed C═O at 167.01 p.p.m.

Crystal data

$C_{21}H_{17}NO_2$
M_r = 315.36
Triclinic, $P\overline{1}$
a = 6.0985 (9) Å
b = 9.5020 (16) Å
c = 15.318 (2) Å
α = 74.815 (12)°
β = 87.578 (12)°
γ = 73.355 (12)°
V = 820.2 (2) Å^3
Z = 2
D_x = 1.277 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14524 reflections
θ = 2.3–28.1°
μ = 0.08 mm^{-1}
T = 296 K
Prismatic stick, colourless
0.80 × 0.38 × 0.09 mm

Data collection

Stoe IPDS-II diffractometer	R_{int} = 0.084
ω scans	θ_{max} = 27.6°
Absorption correction: none	$h = -7 \rightarrow 7$
9578 measured reflections	$k = -12 \rightarrow 12$
3689 independent reflections	$l = -19 \rightarrow 19$
2112 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.114	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
S = 0.93	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
3689 reflections	Extinction correction: *SHELXL97*
218 parameters	Extinction coefficient: 0.019 (4)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.407 (2)	N1—C2	1.371 (2)
O1—C16	1.379 (2)	N1—C3	1.483 (2)
O2—C2	1.215 (2)	N1—C10	1.406 (3)
C1—O1—C16	118.13 (14)	N1—C2—C1	92.04 (14)
C2—N1—C3	95.40 (15)	N1—C3—C1	86.30 (13)
C2—N1—C10	134.15 (15)	N1—C3—C4	115.89 (15)
C3—N1—C10	130.44 (15)	N1—C10—C11	121.12 (18)
O1—C1—C2	111.31 (15)	N1—C10—C15	119.42 (18)
O1—C1—C3	118.39 (14)	O1—C16—C17	124.68 (16)
O2—C2—N1	132.67 (18)	O1—C16—C21	114.99 (16)
O2—C2—C1	135.29 (18)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.98	2.45	3.389 (2)	160
C9—H9···O1^{i}	0.93	2.57	3.455 (2)	160
C11—H11···O2	0.93	2.58	3.180 (3)	123

Symmetry code: (i) $x-1, y, z$.

H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.93 and 0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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