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4′-[(2-Butyl-4-oxo-1,3-diazaspiro[4.4]-non-1-en-3-yl)methyl]-1,1′-biphenyl-2-carbonitrile 0.25-hydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.118
Data-to-parameter ratio = 12.3

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

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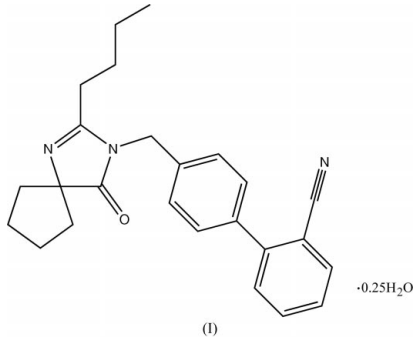
In the title compound, $C_{25}H_{27}N_3O\cdot0.25H_2O$, the minimum and maximum C—C bond lengths in the cyclopentane ring are 1.444 (3) and 1.489 (3) Å, respectively. In the crystal structure, molecules are linked by weak intermolecular C—H···O hydrogen bonds.

Comment

The title compound, (I), is an important intermediate in the synthesis of 2-alkyl or 2-alkoxy-3-[[2′-(1*H*-tetrazol-5-yl)[1,1′-biphenyl]-4-yl]methyl]-1,3-diazaspiro[4,4]non-1-en-4-ones (Caron *et al.*, 1997; Spinale, 1996; Bernhart *et al.*, 1993), and its salts are useful as antagonists of the peptide hormone angiotensin II. Angiotensin II is a potent vasopressor and the biologically active product of the renin–angiotensin system. Renin acts on the angiotensinogen of the plasma to produce angiotenson I, which is converted to angiotensin II by the action of the angiotensin I converting enzyme. The final compounds inhibit the action of angiotensin II on its receptors and thus prevent an increase in blood pressure produced by the hormone-receptor interaction. These compounds are therefore useful in the treatment of hypertension and heart failure (Duncia *et al.*, 1991).



(I)

The asymmetric unit of (I) consists of one molecule of 4′-[(2-butyl-4-oxo-1,3-diazaspiro[4.4]non-1-en-3-yl)methyl]-1,1′-biphenyl- 2-carbonitrile and one quarter molecule of solvent water. The water molecule lies close to a crystallographic inversion center. In the cyclopentane ring, the minimum C—C bond length is 1.444 (3) Å and the maximum C—C bond length is 1.489 (3) Å; the average distance for the cyclopentane C—C bond [1.463 (9)°] in (I) is slightly shorter than that of the average cyclopentane ring C—C bond [1.483 (9)°] in 1-aminocyclopentanecarboxamide (Song *et al.*, 2004). In the biphenyl portion, the torsion angles C4—C5—C8—C9 and C6—C5—C8—C13 for the link between the two benzene rings [−54.7 (2) and −50.0 (3)°, respectively] in (I) are similar

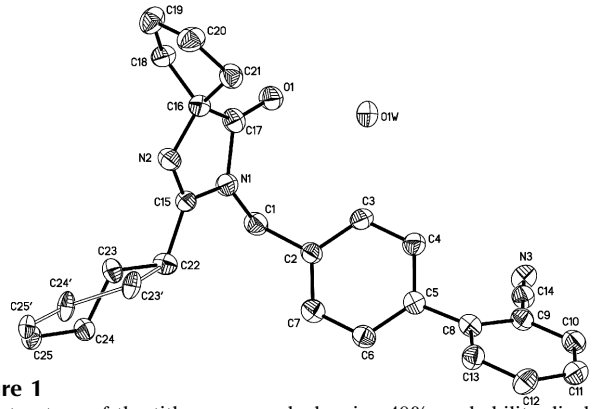


Figure 1
The structure of the title compound, showing 40% probability displacement ellipsoids and the atom-numbering scheme. Both disordered components are shown. H atoms have been omitted.

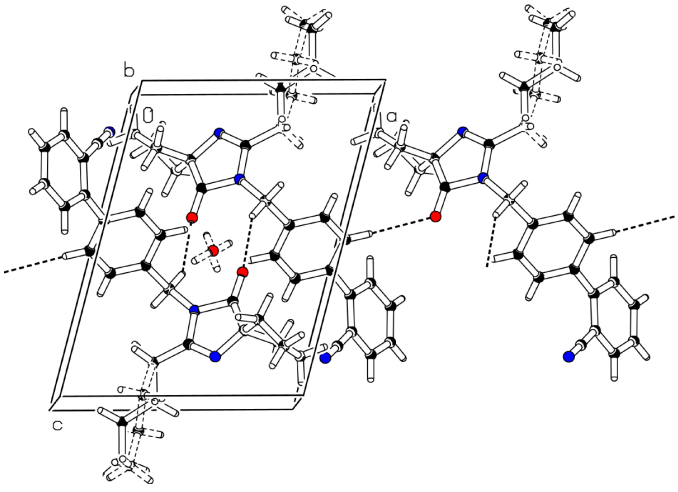


Figure 2
A packing diagram (Spek, 2003) of the title compound, viewed along the *b* axis. Intermolecular C—H···O interactions are shown as dashed lines. Color codes: red O, blue N and black C.

to those in 4′-(bromomethyl)-1,1′-biphenyl-2-carbonitrile [−48.6 (7)°, and −48.3 (7)°, respectively; Song *et al.*, 2003]. The alkyl group C23—C24—C25 is disordered over two sites with relative occupancies 0.83 (1):0.17 (1). In the title molecule, the remaining bond lengths and angles are not unusual. Weak C—H···O interactions are the principal intermolecular forces (see Fig. 2 and Table 2).

Experimental

A mixture of 2-butyl-1,3-diazaspiro[4.4]nonan-4-one hydrochloride (1.16 g, 5.0 mmol), 4′-(bromomethyl)[1,1′-biphenyl]-2-carbonitrile (1.36 g, 5.0 mmol), a 75% aqueous solution of methyltributylammonium chloride (0.12 ml, 0.375 mmol), toluene (15.0 ml) and a 50% aqueous solution of sodium hydroxide (10.0 ml) was stirred vigorously at room temperature for 3.5 h. The reaction mixture was then diluted with water (15 ml) and the two phases separated; the organic phase was extracted twice with water (10 ml) and then evaporated under vacuum, giving the title compound (yield 1.65 g, 85.7%). Crystals were obtained from a solution in ethyl acetate after two weeks at room temperature.

Crystal data

$C_{25}H_{27}N_3O\cdot0.25H_2O$	$Z = 2$
$M_r = 390.00$	$D_x = 1.201$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.1419$ (13) Å	Cell parameters from 831 reflections
$b = 10.0656$ (14) Å	$\theta = 2.5$–$21.4°$
$c = 12.1721$ (17) Å	$\mu = 0.08$ mm^{-1}
$\alpha = 90.438$ (3)°	$T = 293$ (2) K
$\beta = 105.110$ (3)°	Prism, colorless
$\gamma = 94.126$ (3)°	0.32 × 0.28 × 0.20 mm
$V = 1078.1$ (3) Å^3	

Data collection

Bruker SMART APEX CCD diffractometer	3713 independent reflections
φ and ω scans	3033 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$R_{int} = 0.013$
$T_{min} = 0.97$, $T_{max} = 0.98$	$\theta_{max} = 25.0°$
5382 measured reflections	$h = -9 \rightarrow 10$
	$k = -11 \rightarrow 11$
	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.10$ e Å^{-3}
3713 reflections	$\Delta\rho_{min} = -0.10$ e Å^{-3}
301 parameters	

Table 1
Selected geometric parameters (Å, °).

C16—C18	1.455 (2)	C18—C19	1.489 (3)
C16—C21	1.462 (2)	C19—C20	1.444 (3)
C16—C17	1.466 (2)	C20—C21	1.465 (3)
C4—C5—C8—C9	−54.7 (2)	C6—C5—C8—C13	−50.0 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1^{i}	0.97	2.57	3.474 (2)	154
C6—H6···O1^{ii}	0.93	2.57	3.477 (2)	164
C18—H18B···O1	0.97	2.59	2.994 (2)	105

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

All H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.85 Å for O—H and 0.93–0.97 Å for C—H. Isotropic displacement parameters were set at 1.2 and 1.5 (for methyl) times U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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