2-(1H-Benzotriazol-1-yl)-1-phenyl-2-(1H-1,2,4-triazol-1-yl)ethanone cyclohexane hemisolvate

Jun Wan, Chun-Li Li, Xue-Mei Li, Liang-Zhong Xu and Shu-Sheng Zhang*

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China

Correspondence e-mail: shushzhang@126.com

In the title compound, $C_{16}H_{12}N_6O\cdot0.5C_6H_{12}$, the disordered molecule of cyclohexane lies on a centre of symmetry. In the crystal structure, molecules are linked into double chains along the b axis by intermolecular C—H$\cdots$N interactions. The packing is further stabilized by C—H$\cdots\pi$ and π–π interactions.

Comment

We have recently reported the structure of 2-(1H-benzotriazol-1-yl)-1-(2,4-dichlorophenyl)-2-(1H-1,2,4-triazol-1-yl)-ethanone, (II) (Wan *et al.*, 2005). In our ongoing search for new triazole compounds with higher bioactivity, the title compound, (I), was synthesized and we report its structure here.
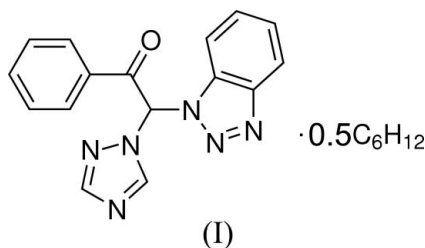
The bond lengths and angles in (I) compare well with those in the related compound, (II). The benzotriazole moiety is essentially planar, with a dihedral angle of 0.5 (1)° between the rings C9–C14 and N1–N3/C9/C14. The mean plane of the benzotriazole group makes an angle of 70.2 (1)° with the N4–N6/C15/C16 ring. The cyclohexane molecule lies on a centre of symmetry and atom C17 is disordered over two positions, with refined site occupancies of 0.44 (1) for atom C17A and 0.56 (1) for atom C17B.

In the crystal structure of (I), molecules are linked into double chains by intermolecular C8—H8A$\cdots$N5 and C16—H16A$\cdots$N3 interactions (Fig. 2, Table 1). The packing is further stabilized by C—H$\cdots\pi$ and π–π interactions involving the benzene rings: $Cg3\cdots Cg3(-x, -y, -1 - z) = 3.755$ (2) Å, where $Cg3$ is the centroid of the C1–C6 ring.

Experimental

Bromine (3.2 g, 0.02 mol, 50 ml) was added dropwise to a solution of 1-phenyl-2-(1H-1,2,4-triazol-1-yl)ethanone (3.7 g, 0.02 mol) and sodium acetate (1.6 g, 0.02 mol) in acetic acid (50 ml). The reaction was maintained for about 2–3 h until the mixture turned colourless. Water (50 ml) and chloroform (20 ml) were then added. The organic layer was washed successively with saturated sodium bicarbonate solution and brine, dried over anhydrous magnesium sulfate and the
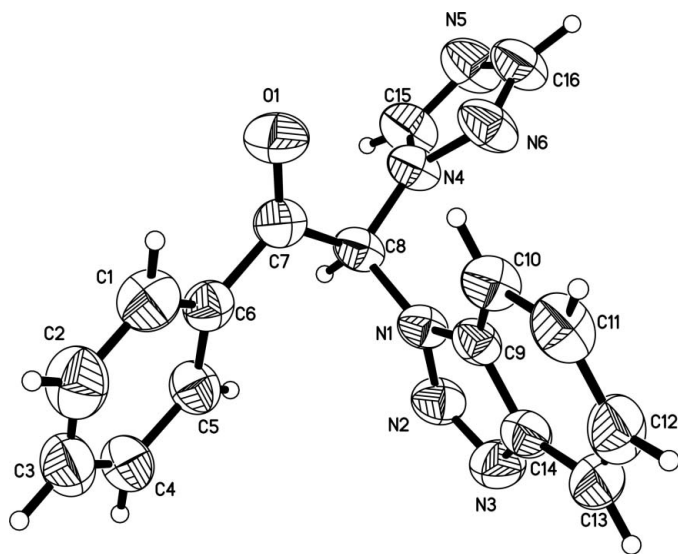
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The cyclohexane solvent molecule has been omitted.
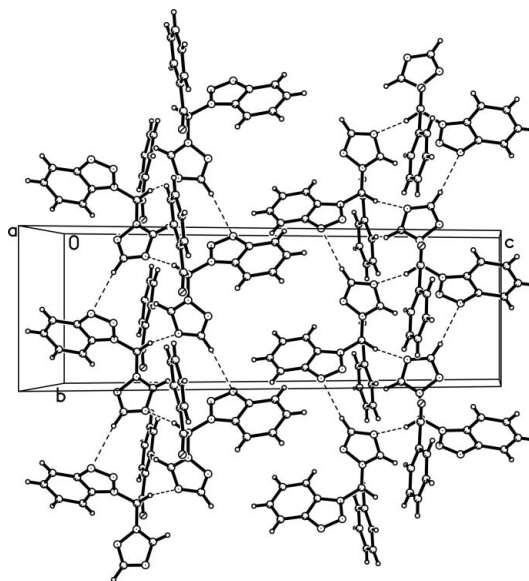

Figure 2
A packing diagram, viewed down the *a* axis. Hydrogen bonds are indicated by dashed lines. The cyclohexane solvent molecule has been omitted.

chloroform solution filtered. It was cooled with ice–water, and then an acetone solution (10 ml) of benzotriazole (2.4 g, 0.02 mol) and triethylamine (2.8 ml, 0.02 mol) were added with stirring. The mixture was stirred at room temperature for about 2 h. The solution was then filtered, concentrated and purified by flash column chromatography (silica gel, petroleum ether–ethyl acetate, 3:1 *v/v*) to afford the title compound. Single crystals of (I) suitable for X-ray measurements were obtained by slow evaporation of an ethyl acetate–cyclohexane (3:1 *v/v*) solution at room temperature over a period of one week.

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

$Cg3$ is the centroid of the C1–C6 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$A\cdots$N5[i]	0.98	2.48	3.399 (2)	155
C16—H16$A\cdots$N3[ii]	0.93	2.57	3.489 (2)	172
C12—H12$A\cdots$Cg3[iii]	0.93	2.84	3.726	151

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

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å, and with U_{iso}(H) = 1.2 U_{eq}(C). The distances C19—C17A and C19—C17B were restrained to be equal to within 0.002 Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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, 2003).

References

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Wan, J., Li, C.-L., Li, X.-M., Zhang, S.-S. & Ouyang, P.-K. (2005). *Acta Cryst.* E**61**, o3232–o3233.

Crystal data

C$_{16}$H$_{12}$N$_6$O·0.5C$_6$H$_{12}$
M_r = 346.39
Monoclinic, $P2_1/c$
a = 8.9187 (12) Å
b = 8.3101 (11) Å
c = 25.367 (3) Å
β = 108.718 (4)°
V = 1780.6 (4) Å^3
Z = 4

D_x = 1.292 Mg m^{-3}
Mo Kα radiation
Cell parameters from 3808 reflections
θ = 2.4–25.5°
μ = 0.09 mm^{-1}
T = 293 (2) K
Plate, colourless
0.30 × 0.20 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.975, T_{max} = 0.992
9620 measured reflections

3479 independent reflections
2832 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 26.0°
$h = -11 \rightarrow 9$
$k = -9 \rightarrow 10$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.149
S = 0.94
3479 reflections
246 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0928P)^2 + 0.5045P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = -0.28 e Å^{-3}