Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Benzyl 3,5-bis(1*H*-benzotriazol-1-ylmethyl)phenyl ether

S. Selvanayagam,[a]
D. Velmurugan,[a]*
K. Ravikumar,[b]
M. Dhanasekaran[c] and
P. Rajakumar[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]Department of Organic Chemistry, University of Madras, Guindy campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.130
Data-to-parameter ratio = 16.6

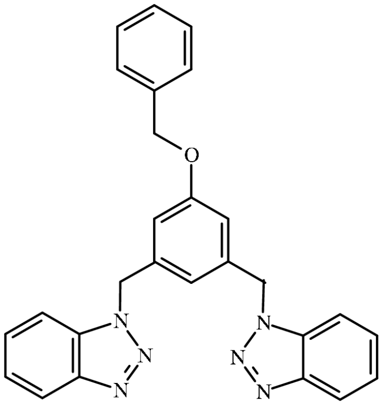
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 October 2004
Accepted 25 October 2004
Online 30 October 2004

In the title molecule, $C_{27}H_{22}N_6O$, the dihedral angle between the two benzotriazole moieties is 18.2 (1)° and those between the central benzene ring and the attached benzotriazole moieties are 74.7 (1) and 88.3 (1)°. The molecular packing in the crystal structure is stabilized by intramolecular C—H···N hydrogen bonds and intermolecular C—H···O interactions.

Comment

Benzotriazole and its derivatives comprise an important class of corrosion inhibitors, typically used as trace additives in industrial chemical mixtures, such as coolants, cutting fluids and hydraulic fluid (Pillard *et al.*, 2001). These derivatives are used as inhibitors of Acanthamoeba castellanii (Kopanska *et al.*, 2004), UV-absorbing agents (Joo & Simon, 1974) and ligands for some serotonin and dopamine receptor subtypes (Boido *et al.*, 2001). These derivatives are active with respect to plant growth (Sparatore, La Rotonda, Paglietti *et al.*, 1978; Sparatore, La Rotonda, Ramundo *et al.*, 1978). Recent studies have shown that benzotriazole derivatives are a major component of aircraft deicing fluids and are responsible for toxicity to bacteria (Pillard *et al.*, 2001; Gruden *et al.*, 2001). In this paper, we report the structure of the title compound, (I).



(I)

The molecular structure of (I) and atom-numbering scheme are shown in Fig. 1. The bond lengths and angles observed in the two benzotriazole ring systems agree with each other and are comparable with those reported in other benzotriazole compounds (Fayos & Garcia-Blanco, 1972; Lopez De Lerma *et al.*, 1973; Bosch *et al.*, 1983; Selvanayagam *et al.*, 2002). The bond lengths in benzene rings *A* and *B* (Fig. 1) are comparable to the reported mean value of 1.384 (13) Å (Allen *et al.*, 1987).

The exocyclic angles around atoms N1 and N4 show considerable asymmetry, with the C—N—C angles [129.2 (1)

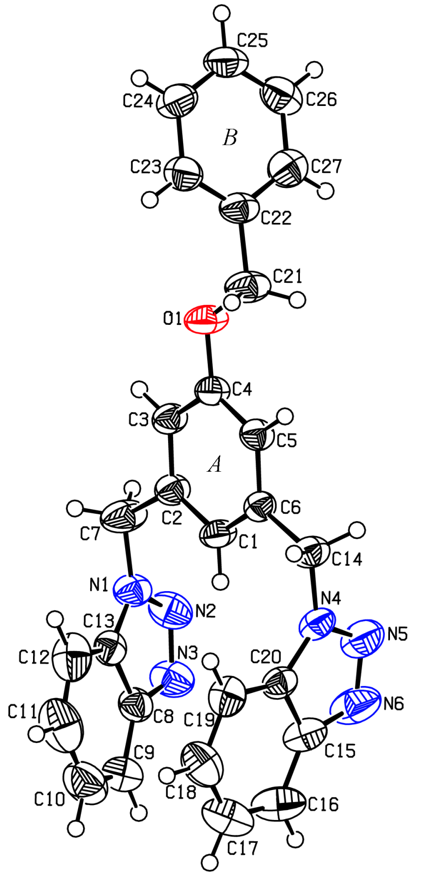


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

and 129.4 (1)°] being much wider than the C—N—N angles [120.6 (1) and 120.2 (1)°]. This asymmetry has been observed in earlier reported structures (Peeters *et al.*, 1993; Fayos & Garcia-Blanco, 1972; Selvanayagam *et al.*, 2002).

The torsion angles N4—C14—C6—C1 [7.4 (2)°] and N1—C7—C2—C3 [176.3 (2)°] indicate the conformations of the two benzotriazole rings attached to benzene ring *A*, *i.e.* they adopt a *syn–anti* conformation with respect to one another. The conformation between benzene rings *A* and *B* is +*anti*-le C22—C21—O1—C4 of 172.6 (1)°.

One of the benzotriazole ring systems (N1/N2/N3/C8–C13) is planar within 0.024 (2) Å, while the other is planar within 0.009 (2) Å. The dihedral angle between these two planes is 18.2 (1)°. The dihedral angle between benzene ring *A* and the N1-benzotriazole plane is 74.7 (1)°; that between benzene ring *A* and the N4-benzotriazole is 88.3 (1)°. Benzene rings *A* and *B* are oriented at an angle of 82.6 (1)° with respect to one another.

In addition to van der Waals interactions, intramolecular C—H⋯N hydrogen bonds influence the conformation of the molecules and the crystal structure is stabilized by intermolecular C—H⋯O interactions connect molecules in sheets parallel to the *ab* plane (Fig. 2 and Table 2).

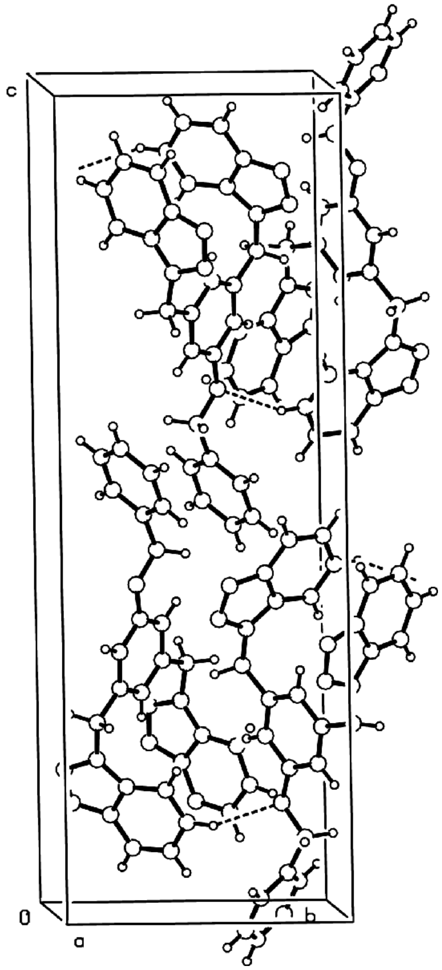


Figure 2
The molecular packing of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

Experimental

To a solution of benzotriazole (0.02 mol) in acetonitrile (50 ml), NaOH solution (10 ml, 25%) was added and the resulting solution was stirred for 10 min. 1,3-Bis(bromomethyl)-5-benzoyloxybenzene (0.01 mol) in acetonitrile (10 ml) was then added in one portion and the solution was stirred for 28 h at room temperature. After completion of the reaction, the reaction mixture was evaporated in a vacuum, extracted with $CHCl_3$, washed with brine and dried over $MgSO_4$. The solvent was evaporated in a vacuum. The crude product was purified by column chromatography on neutral alumina, using ethyl acetate–hexane (1:4) as eluant. The compound was recrystallized from an ethyl acetate–hexane mixture (1:1).

Crystal data

$C_{27}H_{22}N_6O$
$M_r = 446.51$
Monoclinic, $P2_1/c$
$a = 8.5494$ (9) Å
$b = 9.5609$ (10) Å
$c = 27.627$ (3) Å
$\beta = 98.780$ (2)°
$V = 2231.8$ (4) Å^3
$Z = 4$

$D_x = 1.329$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4863 reflections
$\theta = 2.3$–27.1°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.28 × 0.16 × 0.14 mm

o2166 S. Selvanayagam *et al.* • $C_{27}H_{22}N_6O$ *Acta Cryst.* (2004). E**60**, o2165–o2167

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
13153 measured reflections
5095 independent reflections
3847 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 28.0^\circ$
$h = -7 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -35 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.130$
$S = 1.02$
5095 reflections
307 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.3501P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—C7	1.517 (2)	C21—O1	1.423 (2)
C4—O1	1.3624 (15)	C21—C22	1.502 (2)
C6—C14	1.515 (2)	N1—N2	1.350 (2)
C7—N1	1.4450 (18)	N2—N3	1.296 (2)
C8—N3	1.371 (2)	N4—N5	1.351 (2)
C14—N4	1.453 (2)	N5—N6	1.304 (2)
C15—N6	1.369 (2)		
N2—N1—C7	120.6 (1)	N5—N4—C14	120.2 (1)
C13—N1—C7	129.2 (1)	C20—N4—C14	129.4 (1)
C3—C2—C7—N1	176.3 (2)	C22—C21—O1—C4	172.6 (1)
C1—C6—C14—N4	7.4 (2)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1$\cdots$N1	0.93	2.54	2.876 (2)	102
C1—H1$\cdots$N4	0.93	2.51	2.854 (2)	102
C11—H11$\cdots$O1$^{\rm i}$	0.93	2.46	3.327 (2)	155

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

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with aromatic C—H distances of 0.93 Å and methylene C—H distances of 0.97 Å, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

S*S* thanks the Council of Scientific and Industrial Research (CSIR) for providing a Senior Research Fellowship. DV thanks the Department of Biotechnology (DBT) and the University Grants Commission (UGC) for providing support under major research projects.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Boido, A., Boido, C. C. & Sparatore, F. (2001). *Farmaco*, **56**, 263–275.
Bosch, R., Jung, G. & Winter, W. (1983). *Acta Cryst.* C**39**, 1089–1092.
Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Fayos, J. & Garcia-Blanco, S. (1972). *Acta Cryst.* B**28**, 2863–2868.
Gruden, C. L., Dow, S. M. & Hernandez, M. T. (2001). *Water Environ. Res.* **73**, 72–79.
Joo, I. & Simon, N. (1974). *Arch. Dermatol. Forsch.* **249**, 13–19.
Kopanska, K., Najda, A., Zebrowska, J., Chomicz, L., Piekarczyk, J., Myjak, P. & Bretner, M. (2004). *Bioorg. Med. Chem.* **12**, 2617–2624.
Lopez De Lerma, J., Martinez-Carrera, S. & Garcia-Blanco, S. (1973). *Acta Cryst.* B**29**, 537–541.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Peeters, O. M., Schuerman, G. S., Blaton, N. M. & De Ranter, C. J. (1993). *Acta Cryst.* C**49**, 1958–1961.
Pillard, D. A., Cornell, J. S., Dufresne, D. L. & Hernandez, M. T. (2001). *Water Res.* **35**, 557–560.
Selvanayagam, S., Rajakannan, V., Velmurugan, D., Dhanasekaran, M., Rajakumar, P. & Kim, M.-J. (2002). *Acta Cryst.* E**58**, o1190–o1192.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sparatore, F., La Rotonda, M. I., Paglietti, G., Ramundo, E., Silipo, C. & Vittoria, A. (1978). *Farmaco* (*Sci.*), **33**, 901–923.
Sparatore, F., La Rotonda, M. I., Ramundo, E., Silipo, C. & Vittoria, A. (1978). *Farmaco* (*Sci*,), **33**, 924–944.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Acta Cryst. (2004). E**60**, o2165–o2167 S. Selvanayagam *et al.* • $C_{27}H_{22}N_6O$ **o2167**