Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# 2,2′-Bis(1-ethylbenzimidazol-2-yl)biphenyl

**Robert T. Stibrany, Harvey J. Schugar and Joseph A. Potenza***

Department of Chemistry and Chemical Biology, Rutgers, The State University of New Jersey, 610 Taylor Road, Piscataway, New Jersey 08854-8087, USA

Correspondence e-mail: potenza@rutchem.rutgers.edu

Received 31 March 2003
Accepted 7 April 2003
Online 23 April 2003

The title compound, $C_{30}H_{26}N_4$, adopts a folded, twisted clamshell geometry in the solid state in which the ethyl groups are *trans*, and in which the imidazole fragments of the benzimidazole units are nearly eclipsed. Distances between imidazole fragments indicate extensive intramolecular $\pi$–$\pi$ interactions. The molecules pack in columns along $b$ with little, if any, intermolecular $\pi$–$\pi$ interactions. In contrast, the *n*-propyl analog, 2,2′-bis(1-propylbenzimidazol-2-yl)]biphenyl, adopts an extended conformation with little, if any, $\pi$–$\pi$ interactions of any kind.

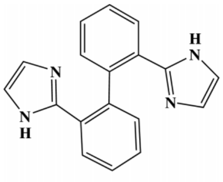
**Key indicators**

Single-crystal X-ray study
$T$ = 294 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.060
$wR$ factor = 0.174
Data-to-parameter ratio = 10.2

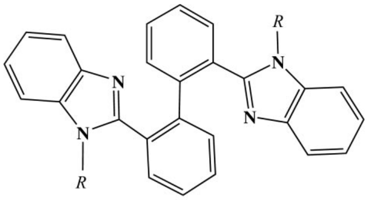
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

Bis(imidazole)biphenyl and bis(benzimidazole)biphenyl species such as (I) and (II), with three torsional degrees of freedom, have been used as ligands to study electron self-exchange (Knapp *et al.*, 1990; Xie *et al.*, 1999), and to enforce near-tetrahedral coordination in transition metal complexes (Stibrany *et al.*, 2003). They have also found use in catalysis (Stibrany, Matturo *et al.*, 2002) and have been shown to act as proton sponges (Stibrany, Schugar & Potenza, 2002).



(I)



(II*a*) *R* = ethyl
(II*b*) *R* = *n*-propyl

The title compound, (II*a*) (Fig. 1), adopts a compact twisted clamshell conformation with the ethyl groups *trans*. Both the substituted phenyl (ph) and benzimidazole (bz) fragments are planar. Fragment planarity, coupled with the ph/ph [63.10 (8)°] and ph/bz [54.47 (6) and 48.29 (6)°] dihedral angles, give the molecule a pseudo-twofold axis passing through the midpoint of the C21—C31 bond and between the bz fragments. The shortest distances between C and N atoms of the nearly eclipsed imidazole fragments range from 3.167 (3) Å for C12···C42 to 3.673 (3) Å for N11···C43, indicative of intramolecular $\pi$–$\pi$ interactions. In type (II) species, the molecular shape correlates with the ph/ph dihedral angle. Thus, the

DOI: 10.1107/S1600536803007852

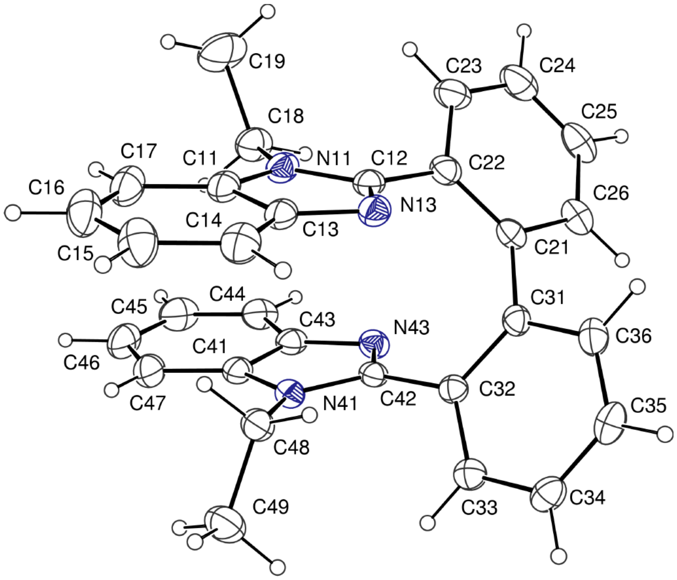
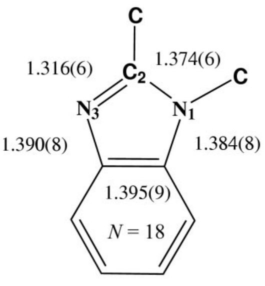
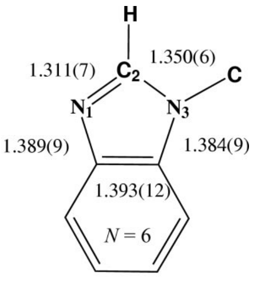


**Figure 1**
The molecular structure of the title compound, showing 25% probability displacement ellipsoids.

proton sponge II$b$H$^+$ (Stibrany, Schugar & Potenza, 2002) adopts a more open structure than (II$a$), consistent with its larger ph/ph dihedral angle [88.6 (6)°], while molecules of (II$b$), with ph/ph dihedral angles of 126.71 (1) and 129.93 (1)° for two unique molecules, exhibit an open *trans* structure with little evidence of intra- or intermolecular $\pi$–$\pi$ interactions (Stibrany *et al.*, 2003).



(III)



(IV)

Metric parameters in the benzimidazole fragments of (II$a$) agree well with those reported for 18 neutral benzimidazole structures (III) having carbon substituents at the N1 and C2 positions [Cambridge Structural Database, Version 5.24 (Allen, 2002); refcodes: DIKFEI, HIXXAX, HOHHUH, JELROH, LEBGOO, MEVTAI, MEVTEM, NUBCOC, QAXKAB, QIBPEW, QIFPOK, SAGDIN, UBIPUQ, XITZOP, YAXDAC and ZENSOA]. Benzimidazoles unsubstituted at the C2 position, (IV), show equivalent distances for the imidazole fragments, except for the the C2—N(amine) distances which are significantly shorter in the unsubstituted compounds (Allen, 2002; refcodes: CAXKUH, MIFVUS, NEBFUV, SEMWAI, SIGVIG and VENSAI). We have not explored the extent to which this difference arises from steric and/or electronic effects.

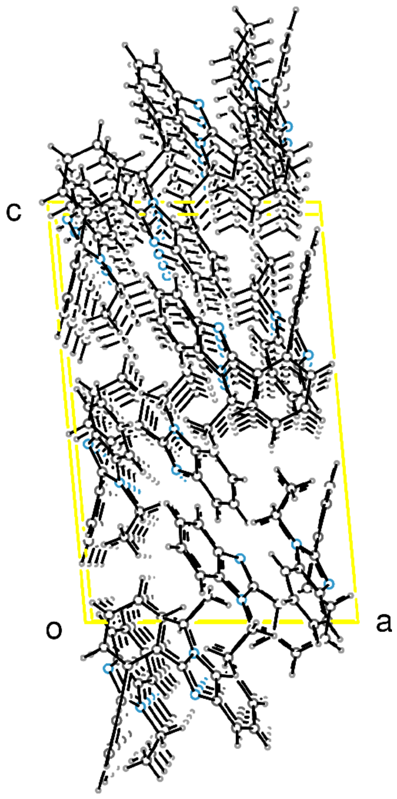


**Figure 2**
View of the unit cell along $b$. H atoms are omitted for clarity.

Molecules of (II$a$) stack along $b$ in columns (Fig. 2) related to each other by the centers of symmetry, glide planes and screw axes of the space group.

## Experimental

The title compound, (II$a$), was obtained by condensation of phenylenediamine with diphenic acid, followed by alkylation with ethyl iodide. Details of the preparation will be published elsewhere (Stibrany *et al.*, 2003). Crystals were obtained by slow evaporation of a solution of (II$a$) in a mixture of ethanol and triethyl orthoformate. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.60 (*d, J* = 7.0 Hz, 2H), 7.41 (*d, J* = 6.5 Hz, 2H), 7.30 (*m*, 6H), 7.23 (*m*, 6H), 3.67 (*brd s*, 4H), 1.19 (*t, J* = 7.0 Hz, 6H). $^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 152.5 143.4, 141.1, 134.5, 131.5, 130.7, 130.0, 129.4, 127.41, 122.2, 121.8, 120.0, 110.0, 38.9, 14.8.

*Crystal data*

$C_{30}H_{26}N_4$
$M_r$ = 442.55
Monoclinic, $P2_1/c$
$a$ = 12.0156 (6) Å
$b$ = 10.7001 (6) Å
$c$ = 18.6280 (9) Å
$\beta$ = 95.177 (4)°
$V$ = 2385.2 (2) Å$^3$
$Z$ = 4
$D_x$ = 1.232 Mg m$^{-3}$
$D_m$ = 1.19 (1) Mg m$^{-3}$
$D_m$ measured by flotation in carbon tetrachloride/cyclohexane
Mo $K\alpha$ radiation
Cell parameters from 821 reflections
$\theta$ = 2.2–23.1°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 294 (1) K
Block, colorless
0.38 × 0.36 × 0.26 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
$T_{\min} = 0.869$, $T_{\max} = 1.000$
16281 measured reflections
4179 independent reflections
3375 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 25.0°$
$h = -13 \rightarrow 14$
$k = -12 \rightarrow 12$
$l = -22 \rightarrow 22$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.174$
$S = 1.00$
4179 reflections
411 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1086P)^2 + 0.4514P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.13$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å).

| | | | |
|---|---|---|---|
| N11—C12 | 1.383 (3) | N43—C43 | 1.387 (3) |
| N41—C42 | 1.385 (3) | N11—C11 | 1.387 (3) |
| N13—C12 | 1.314 (3) | N41—C41 | 1.385 (3) |
| N43—C42 | 1.312 (3) | C11—C13 | 1.395 (3) |
| N13—C13 | 1.387 (3) | C41—C43 | 1.393 (3) |

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *ORTEP*32 (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

## References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (2000). *SHELXTL*(Version6.10), *SAINT-Plus* (Version 6.02) and *SMART-WNT*/2000 (Version 5.622). Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Knapp, S., Keenan, T. P., Zhang, X., Fikar, R., Potenza, J. A., & Schugar, H. J. (1990). *J. Am. Chem. Soc.* **112**, 3452–3464.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Stibrany, R. T., Matturo, M. G., Zushma, S. & Patil, A. O. (2002). US Patent 6 501 000.
Stibrany, R. T., Schugar, H. J. & Potenza, J. A. (2002). *Acta Cryst.* E**58**, o1142–o1144.
Stibrany, R. T., Schugar, H. J. & Potenza, J. A. (2003). *Inorg. Chem.* In preparation.
Xie, B., Elder, T., Wilson, L. J. & Stanbury, D. M. (1999). *Inorg. Chem.* **38**, 12–19.