# organic compounds

# 2,2′-(Ethane-1,2-diyl)bis(1H-benzimida-zole)

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The complete molecule of the title compound, $C_{16}H_{14}N_4$, is generated by crystallographic inversion symmetry. In the crystal, molecules are linked by N—H⋯N hydrogen bonds, generating (001) sheets. Weak aromatic $\pi$–$\pi$ stacking interactions [centroid–centroid distances = 3.7383 (13) and 3.7935 (14) Å] are also observed.

## Related literature

For background to metal-organic frameworks, see: van Albada *et al.* (2007). For the synthesis, see: Wang & Joulli (1957).
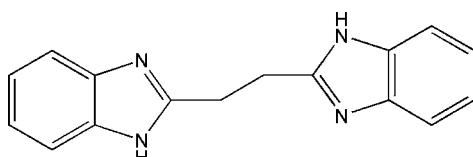
## Experimental

### Crystal data

$C_{16}H_{14}N_4$
$M_r = 262.31$

Orthorhombic, $Pbca$
$a = 8.4295$ (18) Å
$b = 9.924$ (2) Å
$c = 15.351$ (4) Å
$V = 1284.2$ (5) Å$^3$

$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.08$ mm$^{-1}$
$T = 296$ K
$0.32 \times 0.25 \times 0.19$ mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.975$, $T_{max} = 0.984$

10702 measured reflections
1475 independent reflections
966 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.122$
$S = 1.02$
1475 reflections

91 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ e Å$^{-3}$

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

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N2—H2B⋯N1i | 0.86 | 2.04 | 2.8568 (18) | 159 |

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6688).

## References

Albada, G. A. van, Mutikainen, I., Turpeinen, U. & Reedijk, J. (2007). *J. Chem. Cryst.* **37**, 489–496.
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Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, L. L.-Y. & Joulli, M. M. (1957). *J. Am. Chem. Soc.* **79**, 5706–5708.

# 2,2′-(Ethane-1,2-diyl)bis(1*H*-benzimidazole)

## Xu Chen, Xiao-Yong Wu and Guo-Liang Zhao

## Comment

We present a polyaromatic compoud (1) that contains multiple functional groups that can develop a series of metal-organic frameworks with potential applications (e.g. van Albada *et al.* (2007)). The molecular struture of (1), shown in Fig.1, consists of two symmetrical benzimidazole rings. The two benzimidazolyl rings are nearly parallel, witha dihedral angle of 2.645 (6)° between them. Molecules are linked *via* a network of hydrogen bonds (N2—H2B···N1; Table 2). $\pi$–$\pi$ stacking interactions are observed between nearly parallel benzimidazolyl benzene rings. The centroid-to-centroid distance between C1—C6 benzene rings is 3.7379 Å, while between C1A—C6A benzene rings it is 3.7944 Å (the symmetry operation: -*x* + 1,-*y*,-*z*). The hydrogen bonds and $\pi$–$\pi$ weak non-covalent interactions lend stability to the structure. The stacking plot of this compound was shown in Fig. 2.

## Experimental

*o*-Phenylenediamine (1.081 g, 10 mmol) and succinic acid (0.590 g, 5 mmol) were refluxed for 4 h in 30 ml of 10% hydrochloric acid solution. The reaction mixture was cooled to room temperature, the precipitation that formed was filtered and then recrystallized from water. The colourless crystals were obtained from water after a week (Wang *et al.*, 1957).

## Refinement

The H atoms bonded to C and N atoms were positioned geometrically and refined using a riding model [aliphatic C—H =0.96 Å ($U_{iso}$(H) = 1.5$U_{eq}$(C)), aromatic C—H = 0.93 Å ($U_{iso}$(H) = 1.2$U_{eq}$(C)) and N—H = 0.86 Å with $U_{iso}$(H) = 1.2$U_{eq}$(N).

## Computing details

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT* (Bruker, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).
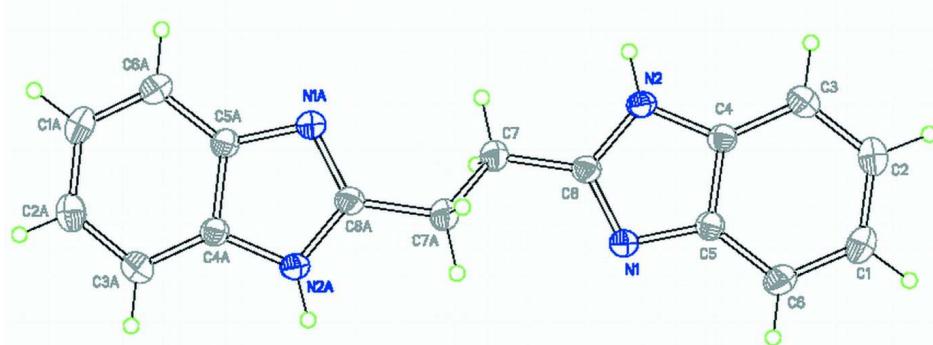
**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. Atoms with suffix A are at the symmetry position (1–x, 1–y, –z).
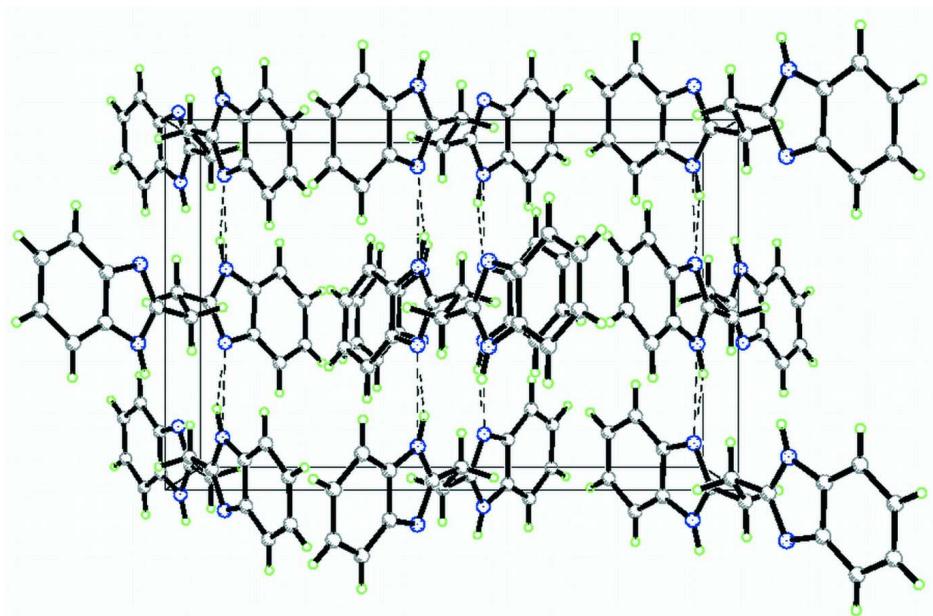


**Figure 2**

The stacking plot of the title compound, showing H-bond interactions (dashed lines) and $\pi$–$\pi$ stacking interactions.

**2,2′-(Ethane-1,2-diyl)bis(1H-benzimidazole)**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{14}N_4$ | $F(000) = 552$ |
| $M_r = 262.31$ | $D_x = 1.357$ Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ac 2ab | $\theta = 2.7$–27.6° |
| $a = 8.4295$ (18) Å | $\mu = 0.08$ mm$^{-1}$ |
| $b = 9.924$ (2) Å | $T = 296$ K |
| $c = 15.351$ (4) Å | Block, colourless |
| $V = 1284.2$ (5) Å$^3$ | $0.32 \times 0.25 \times 0.19$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 10702 measured reflections |
| Radiation source: fine-focus sealed tube | 1475 independent reflections |
| Graphite monochromator | 966 reflections with $I > 2\sigma(I)$ |
| phi and $\omega$ scans | $R_{int} = 0.052$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $\theta_{max} = 27.6°$, $\theta_{min} = 2.7°$ |
| | $h = -10 \rightarrow 10$ |
| | $k = -12 \rightarrow 12$ |
| $T_{min} = 0.975$, $T_{max} = 0.984$ | $l = -19 \rightarrow 16$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.045$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.122$ | |
| $S = 1.02$ | H-atom parameters constrained |
| 1475 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.2274P]$ |
| 91 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 0 restraints | $(\Delta/\sigma)_{max} < 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.15$ e Å⁻³ |
| | $\Delta\rho_{min} = -0.21$ e Å⁻³ |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| N2 | 0.18376 (15) | 0.39276 (12) | 0.06131 (8) | 0.0378 (4) |
| H2B | 0.1935 | 0.3084 | 0.0498 | 0.045* |
| N1 | 0.23667 (16) | 0.61293 (12) | 0.06218 (9) | 0.0402 (4) |
| C5 | 0.09915 (18) | 0.58969 (15) | 0.11024 (10) | 0.0357 (4) |
| C8 | 0.28216 (19) | 0.49246 (14) | 0.03497 (11) | 0.0375 (4) |
| C4 | 0.06495 (18) | 0.45137 (15) | 0.11012 (10) | 0.0354 (4) |
| C7 | 0.42738 (19) | 0.46475 (17) | −0.01726 (12) | 0.0452 (4) |
| H7A | 0.4092 | 0.4932 | −0.0769 | 0.054* |
| H7B | 0.4467 | 0.3684 | −0.0178 | 0.054* |
| C3 | −0.0631 (2) | 0.39827 (18) | 0.15503 (11) | 0.0475 (5) |
| H3A | −0.0860 | 0.3066 | 0.1537 | 0.057* |
| C6 | 0.0040 (2) | 0.67805 (16) | 0.15747 (11) | 0.0461 (5) |
| H6A | 0.0247 | 0.7701 | 0.1581 | 0.055* |
| C2 | −0.1552 (2) | 0.48740 (18) | 0.20187 (12) | 0.0524 (5) |
| H2A | −0.2416 | 0.4551 | 0.2333 | 0.063* |
| C1 | −0.1215 (2) | 0.62512 (18) | 0.20317 (12) | 0.0520 (5) |
| H1A | −0.1856 | 0.6824 | 0.2357 | 0.062* |

*Atomic displacement parameters (Å²)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N2 | 0.0346 (8) | 0.0281 (6) | 0.0506 (8) | 0.0003 (5) | 0.0010 (6) | −0.0046 (6) |
| N1 | 0.0373 (8) | 0.0317 (7) | 0.0515 (8) | −0.0013 (5) | 0.0053 (6) | −0.0039 (6) |
| C5 | 0.0323 (9) | 0.0347 (8) | 0.0402 (9) | 0.0015 (7) | −0.0014 (7) | 0.0005 (6) |
| C8 | 0.0328 (9) | 0.0329 (8) | 0.0469 (9) | −0.0014 (6) | −0.0024 (7) | −0.0044 (7) |
| C4 | 0.0326 (9) | 0.0330 (8) | 0.0407 (9) | 0.0013 (6) | −0.0027 (7) | −0.0005 (6) |
| C7 | 0.0343 (9) | 0.0450 (9) | 0.0563 (11) | −0.0019 (7) | 0.0050 (8) | −0.0102 (8) |
| C3 | 0.0458 (11) | 0.0432 (9) | 0.0537 (10) | −0.0071 (8) | 0.0034 (9) | 0.0023 (8) |
| C6 | 0.0473 (11) | 0.0371 (9) | 0.0541 (11) | 0.0069 (8) | 0.0020 (9) | −0.0036 (7) |
| C2 | 0.0435 (11) | 0.0626 (12) | 0.0512 (11) | −0.0013 (9) | 0.0105 (9) | 0.0027 (9) |
| C1 | 0.0458 (11) | 0.0571 (11) | 0.0529 (11) | 0.0130 (9) | 0.0083 (9) | −0.0057 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N2—C8 | 1.3530 (19) | C7—H7A | 0.9700 |
| N2—C4 | 1.3795 (19) | C7—H7B | 0.9700 |
| N2—H2B | 0.8600 | C3—C2 | 1.379 (2) |
| N1—C8 | 1.3233 (18) | C3—H3A | 0.9300 |
| N1—C5 | 1.393 (2) | C6—C1 | 1.374 (2) |
| C5—C6 | 1.392 (2) | C6—H6A | 0.9300 |
| C5—C4 | 1.403 (2) | C2—C1 | 1.396 (2) |
| C8—C7 | 1.489 (2) | C2—H2A | 0.9300 |
| C4—C3 | 1.385 (2) | C1—H1A | 0.9300 |
| C7—C7$^i$ | 1.506 (3) | | |
| | | | |
| C8—N2—C4 | 107.40 (12) | C8—C7—H7B | 109.0 |
| C8—N2—H2B | 126.3 | C7$^i$—C7—H7B | 109.0 |
| C4—N2—H2B | 126.3 | H7A—C7—H7B | 107.8 |
| C8—N1—C5 | 104.99 (12) | C2—C3—C4 | 117.01 (16) |
| C6—C5—N1 | 130.61 (14) | C2—C3—H3A | 121.5 |
| C6—C5—C4 | 119.91 (15) | C4—C3—H3A | 121.5 |
| N1—C5—C4 | 109.41 (13) | C1—C6—C5 | 117.96 (15) |
| N1—C8—N2 | 112.88 (14) | C1—C6—H6A | 121.0 |
| N1—C8—C7 | 125.11 (14) | C5—C6—H6A | 121.0 |
| N2—C8—C7 | 122.00 (13) | C3—C2—C1 | 121.40 (17) |
| N2—C4—C3 | 132.51 (15) | C3—C2—H2A | 119.3 |
| N2—C4—C5 | 105.32 (13) | C1—C2—H2A | 119.3 |
| C3—C4—C5 | 122.14 (15) | C6—C1—C2 | 121.57 (16) |
| C8—C7—C7$^i$ | 113.14 (16) | C6—C1—H1A | 119.2 |
| C8—C7—H7A | 109.0 | C2—C1—H1A | 119.2 |
| C7$^i$—C7—H7A | 109.0 | | |
| | | | |
| C8—N1—C5—C6 | 176.72 (17) | N1—C5—C4—C3 | 178.13 (14) |
| C8—N1—C5—C4 | −0.20 (18) | N1—C8—C7—C7$^i$ | 46.1 (3) |
| C5—N1—C8—N2 | 0.41 (18) | N2—C8—C7—C7$^i$ | −132.3 (2) |
| C5—N1—C8—C7 | −178.13 (15) | N2—C4—C3—C2 | 176.40 (16) |
| C4—N2—C8—N1 | −0.47 (19) | C5—C4—C3—C2 | −1.2 (2) |
| C4—N2—C8—C7 | 178.12 (14) | N1—C5—C6—C1 | −176.40 (16) |

| | | | |
|---|---|---|---|
| C8—N2—C4—C3 | −177.62 (17) | C4—C5—C6—C1 | 0.2 (2) |
| C8—N2—C4—C5 | 0.31 (17) | C4—C3—C2—C1 | 0.6 (3) |
| C6—C5—C4—N2 | −177.37 (14) | C5—C6—C1—C2 | −0.9 (3) |
| N1—C5—C4—N2 | −0.07 (17) | C3—C2—C1—C6 | 0.4 (3) |
| C6—C5—C4—C3 | 0.8 (2) | | |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*B*···N1ii | 0.86 | 2.04 | 2.8568 (18) | 159 |

Symmetry code: (ii) −*x*+1/2, *y*−1/2, *z*.