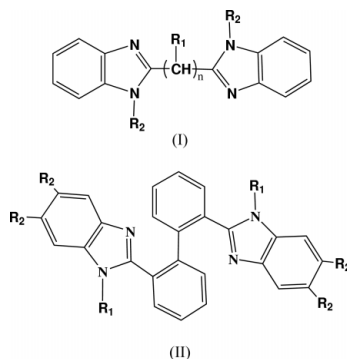


**(*S,S*)-1,2-Bis(1-methylbenzimidazol-2-yl)-
1',2'-bis(methoxy)ethane****Robert T. Stibrany, Harvey J.
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Taylor Road, Piscataway, New Jersey 08854,
USACorrespondence e-mail:
potenza@rutchem.rutgers.edu**Key indicators**Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.048
 wR factor = 0.120
Data-to-parameter ratio = 11.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{20}\text{H}_{22}\text{N}_4\text{O}_2$, exhibits twofold site symmetry and adopts a stepped or kinked conformation in the solid state, with the approximately planar *N*-methyl benzimidazole fragments corresponding to the run and the 1',2'-bis(methoxy)ethane fragment to the rise. When viewed along the *b* axis, the molecules stack in a herringbone pattern as layers along *a*. Intermolecular distances suggest that the molecules are held in the lattice largely by $\text{C}-\text{H}\cdots\pi$ interactions with little, if any, $\pi\cdots\pi$ bonding. Examination of the displacement parameters suggests that the molecules librate about axes approximately parallel to the *a* axis and passing near the center of mass of the molecules. Bond distances in the imidazole group lie within one s.u. of the average reported for 19 structures containing comparable benzimidazole fragments.

Comment

Bis(2-benzimidazol-2-yl) species bridged by alkyl (I), aryl (II) or other groups are versatile species that have attracted considerable interest. For example, compounds related to (I) have shown antiviral or antifungal activity (Roderick, 1971; Del Poeta *et al.*, 1998) and have been used in selective ion-exchange resins (van Berkel *et al.*, 1995), while biaryl-bridged bis(imidazole) and bis(benzimidazole) species of type (II) have been used in catalysis (Stibrany, Matturo *et al.*, 2002), as proton sponges (Stibrany, Schugar & Potenza, 2002), in electron self-exchange studies (Knapp *et al.*, 1990; Xie *et al.*, 1999) and as ligands to enforce near-tetrahedral coordination in transition metal complexes (Stibrany *et al.*, 2003*b*). Introduction of stereogenic centers on the bridging group, as in the title compound [(I), $R_1 = \text{methoxy}$, $R_2 = \text{methyl}$, $n = 2$], expands the spectroscopic reach of these species and affords the possibility of using them in applications such as stereoselective catalysis in which chirality is critical.



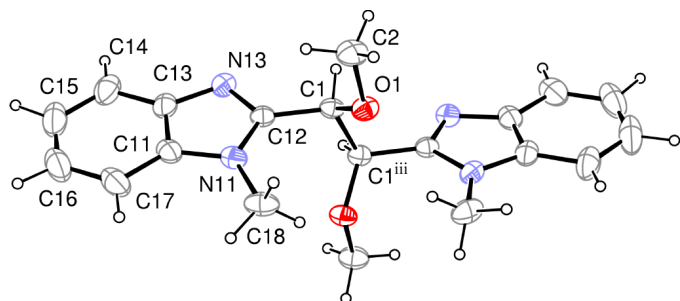


Figure 1
Molecular structure of the title compound, showing 25% probability displacement ellipsoids.

Molecules of the title compound, $C_{20}H_{22}N_4O_2$, exhibit twofold symmetry (site 4b in space group $C222_1$) with the diad axis passing through the midpoint of the C1—C1ⁱⁱⁱ bond [Fig. 1; symmetry code: (iii) $-x, y, \frac{1}{2} - z$]. When viewed in profile (Fig. 2a), they have a stepped or kinked conformation, with the two approximately planar *N*-methyl benzimidazole fragments corresponding to the run and the 1',2'-bis(methoxy)-ethane fragment to the rise. Atoms C1 and C1ⁱⁱⁱ are stereogenic centers whose configurations are *S*, as determined by the starting material in the synthetic procedure, l(+)-tartaric acid. These atoms yield a vector skewed with respect to the benzimidazole plane, as evidenced by the torsion angles N11—C12—C1—C1ⁱⁱⁱ and N13—C12—C1—C1ⁱⁱⁱ [$85.2(2)^\circ$ and $-94.8(2)^\circ$, respectively], an orientation stabilized, in part, by intramolecular C18—H18a \cdots O1ⁱⁱⁱ hydrogen bonds. Metric parameters of the benzimidazole fragments agree well with those reported for related compounds. In particular, bond distances within the imidazole fragment lie within one s.u. of the average of those reported for 19 neutral benzimidazole structures having carbon substituents at the N11 and C12 positions (Stibrany *et al.*, 2003a). As expected for an *N*-alkyl substituted benzimidazole, the C12—N13(imine) distance is substantially shorter than the C12—N11(amine) distance.

When viewed along *b* (Fig. 2a), the molecules stack in a herringbone pattern, as layers along *a*. Intermolecular distances suggest that the molecules are held in the lattice largely by C—H \cdots π interactions with little, if any, $\pi\cdots\pi$ bonding. Examination of the displacement parameters suggests that the molecules librate about axes approximately parallel to the *a* axis and passing near the center of mass of the molecules (Fig. 2b).

Experimental

(*S,S*)-1,2-Bis(1-methylbenzimidazol-2-yl)-1',2'-bis(methoxy)ethane was prepared as described previously (Isele *et al.*, 2002), starting with l(+)-tartaric acid. The title compound was obtained by alkylation of the diol with methyl iodide, using a procedure described in Stibrany *et al.* (2003b). Crystals were obtained by slow evaporation of an acetonitrile solution of the product. 1H NMR (400 MHz, $CDCl_3$): δ = 7.64(*dm*, *J* = 6.9 Hz, 2H), 7.21(*m*, 6H), 5.44(*s*, 2H), 3.84(*s*, 6H), 3.44(*s*, 6H). ^{13}C NMR (400 MHz, $CDCl_3$): δ = 150.1 141.8, 135.6, 123.2, 120.3, 118.6, 109.9, 78.4, 57.1, 29.9. mp, 419–420 K.

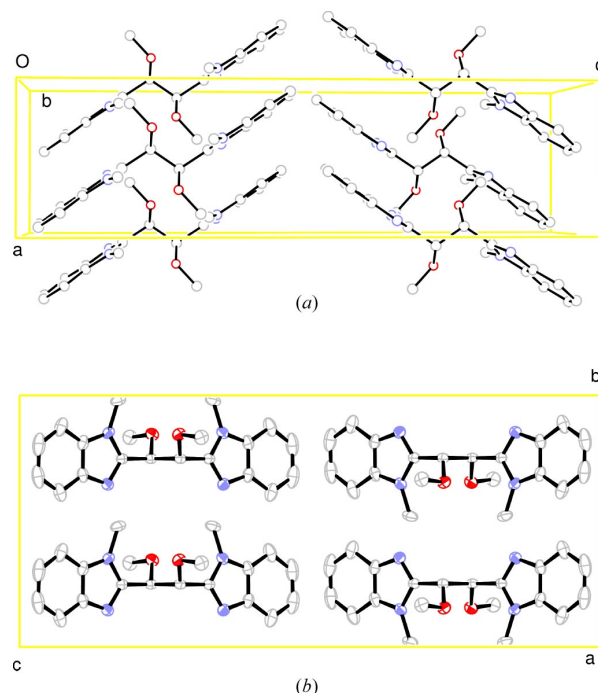


Figure 2
(a) View of the unit cell along the *b* axis and (b) projection of the structure along the *a* axis, showing 25% probability displacement ellipsoids. H atoms are omitted for clarity.

Crystal data

$C_{20}H_{22}N_4O_2$
 $M_r = 350.41$
Orthorhombic, $C222_1$
 $a = 6.7980(6) \text{ \AA}$
 $b = 10.9084(9) \text{ \AA}$
 $c = 25.331(2) \text{ \AA}$
 $V = 1878.4(3) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.239 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 664 reflections
 $\theta = 3.5\text{--}22.8^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 296(1) \text{ K}$
Plate, colorless
 $0.50 \times 0.45 \times 0.03 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Blessing, 1995)
 $T_{\min} = 0.684, T_{\max} = 0.998$
3653 measured reflections

1607 independent reflections
1228 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\max} = 25.1^\circ$
 $h = -7 \rightarrow 8$
 $k = -12 \rightarrow 12$
 $l = -30 \rightarrow 29$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.120$
 $S = 1.00$
1607 reflections
140 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.3025P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.12 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA).

N13—C12	1.310 (4)	N11—C11	1.381 (4)
N13—C13	1.387 (4)	C11—C13	1.400 (4)
N11—C12	1.371 (4)		

The absolute configuration of the title compound was determined from that of the starting material, as outlined in the experimental section. In the absence of significant anomalous scattering effects, Friedel pairs were merged and the absolute configuration could not be confirmed by the crystallographic experiment.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996), *ORTEP32* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 2000).

References

- Berkel, P. M. van, Dijkstra, D. J., Driessen, W. L., Reedijk, J. & Sherrington, D. C. (1995). *React. Funct. Polymer*, **28**, 39–54.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruker (2000). *SHELXTL* (Version 6.10), *SAINTE-Plus* (Version 6.02) and *SMART-WNT2000* (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.
- Del Poeta, M., Schell, W. A., Dykstra, C. C., Jones, S., Tidwell, R. R., Czarny, A., Bajic, M., Bajic, M., Kumar, A., Boykin, D. & Perfect, J. R. (1998). *Antimicrob. Agents Chemother.* **42**, 2495–2502.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Isele, K., Broughton, V., Matthews, C. J., Williams, A. F., Bernardinelli, G., Franz, P. & Decurtins, S. (2002). *J. Chem. Soc. Dalton Trans.* pp. 3899–3905.
- Knapp, S., Keenan, T. P., Zhang, X., Fikar, R., Potenza, J. A. & Schugar, H. J. (1990). *J. Am. Chem. Soc.* **112**, 3452–3464.
- Roderick, W. R. (1971). DE Patent 2 063 856.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Stibrany, R. T., Matturo, M. G., Zushma, S. & Patel, A. O. (2002). US Patent 6 501 000.
- Stibrany, R. T., Schugar, H. J. & Potenza, J. A. (2002). *Acta Cryst.* **E58**, o1142–o1144.
- Stibrany, R. T., Schugar, H. J. & Potenza, J. A. (2003a). *Acta Cryst.* **E59**, o693–o695.
- Stibrany, R. T., Schugar, H. J. & Potenza, J. A. (2003b). *Inorg. Chem.* Submitted.
- Xie, B., Elder, T., Wilson, L. J. & Stanbury, D. M. (1999). *Inorg. Chem.* **38**, 12–19.