Affinement

Affinement à partir des F^2
$R[F^2 > 2\sigma(F^2)] = 0{,}052$
$wR(F^2) = 0{,}130$
$S = 1{,}058$
1735 réflexions
167 paramètres
Tous les paramètres des atomes d'hydrogène affinés
$w = 1/[\sigma^2(F_o^2) + (0{,}0614P)^2 + 1{,}0456P]$
où $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0{,}326$ e Å^{-3}
$\Delta\rho_{\min} = -0{,}296$ e Å^{-3}
Pas de correction d'extinction
Facteurs de diffusion des *International Tables for Crystallography* (Tome C)

Tableau 1. *Paramètres géométriques (Å, °)*

P1—O2	1,485 (3)	O1—HO1	0,69 (4)
P1—O3	1,491 (3)	N1—H1N1	0,92 (4)
P1—O1	1,548 (3)	N1—H2N1	0,87 (4)
P1—O4	1,597 (2)	N1—H3N1	1,02 (4)
O2—P1—O3	116,4 (2)	O3—P1—O4	103,7 (1)
O2—P1—O1	110,9 (2)	O1—P1—O4	105,1 (2)
O3—P1—O1	111,0 (2)	P1^{i}—O4—P1	133,8 (2)
O2—P1—O4	108,8 (1)		

Code de symétrie: (i) $-x, y, \frac{1}{2} - z$.

Tableau 2. *Distances et liaisons hydrogène (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—HO1···O2^{i}	0,69 (4)	1,91 (4)	2,587 (4)	166 (5)
N1—H1N1···O3^{ii}	0,92 (4)	1,84 (4)	2,763 (4)	175 (3)
N1—H2N1···O3	0,87 (4)	1,88 (4)	2,738 (4)	166 (3)
N1—H3N1···O2^{iii}	1,02 (4)	1,71 (4)	2,733 (4)	175 (3)

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

La largeur de balayage est $(1 + 0{,}35\text{tg}\theta)°$. Les intensités ont été corrigées des facteurs de Lorentz–polarisation. La structure a été résolue par les méthodes directes (*SHELXS*86; Sheldrick, 1990) puis affinée par la méthode des moindres carrés (*SHELXL*93; Sheldrick, 1993).

Collection des données: *CAD*-4 *EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992). Affinement des paramètres de la maille: *CAD*-4 *EXPRESS*. Réduction des données: *MolEN* (Fair, 1990). Programme(s) pour la solution de la structure: *SHELXS*86 (Sheldrick, 1990). Programme(s) pour l'affinement de la structure: *SHELXL*93 (Sheldrick, 1993). Graphisme moléculaire: *ZORTEP* (Zsolnai & Pritzkow, 1994). Logiciel utilisé pour préparer le matériel pour publication: *SHELXL*93.

Des documents complémentaires concernant cette structure peuvent être obtenus à partir des archives électroniques de l'UICr (Référence: DU1178). Les processus d'accès à ces archives sont donnés au dos de la couverture.

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Acta Cryst. (1999). C**55**, 476–478

1-Hydroxyethyl-2,2′-biimidazole

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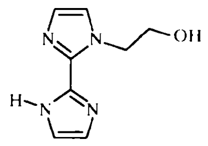
(*Received 24 August 1998; accepted 14 December 1998*)

Abstract

The two imidazole rings in the title compound, $C_8H_{10}N_4O$, are almost coplanar, having an angle of 2.3 (2)° about the C—C bond connecting the rings. The conformation adopted is such that the nitrogen substituents (-H and $-CH_2CH_2OH$) are in a *trans* orientation. The molecules are connected by intermolecular O—H···O and N—H···N hydrogen bonds to yield an arrangement of pairs of molecules reminiscent of side-by-side stacks of crinkled ribbon.

Comment

The structure determination of 1-hydroxyethyl-2,2′-biimidazole, (I), was undertaken as part of a series of studies of substituted biimidazoles (Secondo, Barnett *et al.*, 1996; Secondo *et al.*, 1997; Barnett *et al.*, 1997). The geometry of the title molecule, the first mono-substituted derivative in the series, is illustrated in Fig. 1, and assumes a *trans* conformation as seen in nearly all others in the series. The 2,2′-biimidazole rings are nearly coplanar, as the dihedral angle of the two five-membered rings is 2.3 (2)° about the central C1—C4 bond and all ten non-H atoms lie within 0.02 Å of their least-squares plane. The C—C and C—N bond lengths and angles in the biimidazole skeleton are generally in agreement with the corresponding bond



(I)

Acta Crystallographica Section C
ISSN 0108-2701 © 1999

lengths observed in the other disubstituted biimidazoles referenced above, and in the unsubstituted 2,2′-biimidazole rings (Cromer *et al.*, 1987).

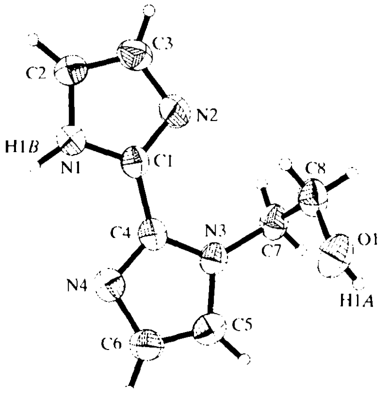


Fig. 1. View of the title molecule, showing the labeling of the non-H and significant H atoms. Displacement ellipsoids are shown at 50% probability levels; H atoms are drawn as small spheres of arbitrary radii.

Double-bond character is indicated for C1—N2 and C4—N4, as their distances [average of 1.325 (2) Å] are significantly shorter than the C1—N1 and C4—N3 distances [average of 1.357 (2) Å], respectively. All but one of the corresponding distances and internal ring angles (through the pseudo-inversion center at the midpoint of the C1—C4 bond) differ by less than 3σ. The C4—N3 distance is $>5\sigma$ longer than the corresponding C1—N1 distance, and this is likely to be due to the difference in electron-withdrawing strength of the $-CH_2CH_2OH$ group on N3 *versus* the H atom on N1.

All five internal ring angles and all but one of the ring distances are within $< 3\sigma$ of the average of the distances and angles in the six rings of five different previously described disubstituted biimidazoles (Secondo, Barnett *et al.*, 1996; Secondo *et al.*, 1997; Barnett *et al.*, 1997). However, he C1—N1 distance [1.351 (2) Å] in the title compound is $\sim 8\sigma$ below the average (1.368 Å) for these same systems. This is also likely to be due to the presence of only H1*B* rather than a non-H group.

The N3–C7–C8 plane is inclined at 81.9 (1)° with respect to the imidazole ring to which it is attached, while the value of the C4—N3—C7—C8 torsion angle is 79.2 (2)°, which is in general agreement with those of similar compounds not having conjugated substituents (Secondo, Barnett *et al.*, 1996; Secondo *et al.*, 1997; Barnett *et al.*, 1997). Systems with conjugated substituents [–C(O)Me and $-CHCH_2$] are essentially coplanar with the imidazole (Secondo, Hester *et al.*, 1996; Sokal *et al.*, 1992). The O1 atom in the title compound is out of the N3–C7–C8 plane by 1.121 (4) Å [N3—C7—C8—O1 = 58.3 (2)°] and out of the ten-atom biimidazole plane by 2.364 (3) Å.

Pairs of molecules are connected by N—H···N hydrogen bonds (Table 2) about inversion centers, and these dimers are linked by O—H···O hydrogen bonds *via* a *c*-glide, creating an 'accordion-fold' arrangement of the molecular pairs.

Experimental

Absolute EtOH (150 ml), 2,2′-biimidazole (8.0 g, 60 mmol) and 3 *M* NaOH (200 ml) were heated to 333 K for about 20 min, and then 2-chloroethanol (6.0 g, 75 mmol) in absolute EtOH (50 ml) was added over a 1 h period. After 10 h, the solution was evaporated on a rotary evaporator to approximately 100 ml. Light-yellow needles of (I) began to form after the solution had cooled to room temperature. The crystals were then filtered and dried in a vacuum oven at 333 K. The purity of the title compound was verified by elemental analysis: actual C 53.99, H 5.40, N 31.71%; theoretical C 53.93, H 5.62, N 31.46%. Recrystallization was by slow evaporation from MeOH.

Crystal data

$C_8H_{10}N_4O$
$M_r = 178.20$
Monoclinic
$P2_1/c$
$a = 11.401\ (1)$ Å
$b = 14.698\ (2)$ Å
$c = 5.0903\ (6)$ Å
$\beta = 94.530\ (9)°$
$V = 850.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.392$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 5.3–20.1°$
$\mu = 0.098$ mm^{-1}
$T = 293\ (2)$ K
Parallelepiped cut from large rod
0.39 × 0.35 × 0.21 mm
Colorless

Data collection

Siemens/Bruker *P*3 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1793 measured reflections
1493 independent reflections
1102 reflections with $I > 2\sigma(I)$
$R_{int} = 0.009$

$\theta_{max} = 25.05°$
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 0$
$l = 0 \rightarrow 6$
3 standard reflections every 50 reflections intensity decay: average of 0.95% in $\sigma(I)$ values

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.113$
$S = 1.012$
1484 reflections
120 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.2152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.183$ e Å^{-3}
$\Delta\rho_{min} = -0.135$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.047 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C1	1.351 (2)	N4—C4	1.327 (2)
N1—C2	1.366 (2)	N4—C6	1.373 (2)
N2—C1	1.322 (2)	C1—C4	1.447 (3)
N2—C3	1.372 (3)	C2—C3	1.349 (3)
N3—C4	1.363 (2)	C5—C6	1.347 (3)
N3—C5	1.370 (2)		
C1—N1—C2	107.0 (2)	C3—C2—N1	106.0 (2)
C1—N2—C3	104.7 (2)	C2—C3—N2	110.7 (2)
C4—N3—C5	106.9 (2)	N4—C4—N3	111.1 (2)
C4—N4—C6	105.1 (2)	N4—C4—C1	124.0 (2)
N2—C1—N1	111.6 (2)	N3—C4—C1	124.9 (2)
N2—C1—C4	126.6 (2)	C6—C5—N3	106.3 (2)
N1—C1—C4	121.8 (2)	C5—C6—N4	110.7 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···N4^{i}	0.90	2.04	2.891 (2)	157
O1—H1*A*···O1^{ii}	0.82	1.95	2.768 (2)	175

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

H atoms were treated as riding, with C—H 0.96, N—H 0.90 and O—H 0.82 Å. Atoms H1*A* and H1*B* were first located in a difference map, then placed in ideal positions.

Data collection: *P3/P4-PC Diffractometer Program* (Siemens, 1991*a*). Cell refinement: *P3/P4-PC Diffractometer Program*. Data reduction: *XDISK* (Siemens, 1991*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXTL/PC* and *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1514). Services for accessing these data are described at the back of the journal.

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