

Fig. 1. ORTEP diagram (Johnson, 1976) illustrating numbering scheme. Non-H ellipsoids at 50% probability level.

Related literature. Crystal structures containing the bicyclo[3.2.1] framework have been reported by Van Meerssche, Germain & Declercq (1979); Murthy, Venkatesan, Reddy & Kasturi (1982); Mehta, Rao, Suri, Cameron & Chan (1980); Duc, Fetizon, Hanna,

Olesker, Pascard & Prange (1980); Yonemitsu, Nakai, Kanaoka, Karle & Witkop (1970).

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References

- CREMER, D. & POPL, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 DUC, D. K. M., FETIZON, M., HANNA, I., OLESKER, A., PASCARD, C. & PRANGE, T. (1980). *J. Chem. Soc. Chem. Commun.* pp. 1209–1210.
 GRAY, B. D. (1983). PhD Thesis. Univ. of Dundee, Scotland.
 JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
 METHA, G., RAO, K. S., SURI, S. C., CAMERON, T. S. & CHAN, C. (1980). *J. Chem. Soc. Chem. Commun.* pp. 650–652.
 MURTHY, P. S., VENKATESAN, K., REDDY, S. M. & KASTURI, T. R. (1982). *Acta Cryst.* **B38**, 2055–2057.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge.
 VAN MEERSSCHE, M., GERMAIN, G. & DELCERCQ, J.-P. (1979). *Acta Cryst.* **B35**, 491–493.
 YONEMITSU, O., NAKAI, H., KANAOKA, Y., KARLE, I. L. & WITKOP, B. (1970). *J. Am. Chem. Soc.* **92**, 5691–5700.

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Structure of 2,2'-Biimidazole*

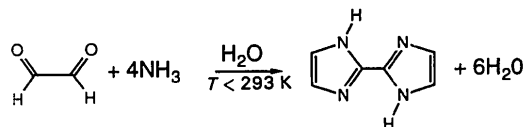
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Abstract. $C_6H_6N_4$, $M_r = 134.14$, monoclinic, $P2_1/c$, $a = 5.067$ (2), $b = 10.786$ (3), $c = 11.490$ (3) Å, $\beta = 102.58$ (3)°, $V = 612.88$ Å³, $Z = 4$, $D_x = 1.453$ g cm⁻³, $\lambda(\text{Mo K}\alpha_1) = 0.70926$ Å, $\mu = 0.92$ cm⁻¹, $F(000) = 280$, room temperature, final $R = 0.054$ for 578 observed reflections [$I > 3\sigma(I)$] out of 806 independent reflections. There are ribbons of hydrogen-bonded molecules parallel to the a axis and the length of this axis is equal to the width of the molecule. Each molecule is joined to its neighbors by two hydrogen bonds on each side. The hydrogen-bond distances are $N(1)\cdots N(2) = 2.865$ (5) and $N(3)\cdots N(4) = 2.879$ (8) Å. This rigid arrangement accounts for the low solubility of 2,2'-biimidazole in anything but an acid solution. The five atoms in each ring are coplanar within 0.002 Å but the two rings are rotated 4.6° about the central C—C bond.

Experimental. 2,2'-Biimidazole prepared by a modification of a method reported by Debus (1958) and by Fiesemann, Hendrickson & Stucky (1978). Anhy-



drous ammonia was bubbled through a 20% solution of glyoxal, carefully keeping the solution temperature below 293 K. After 3–4 h a copious light-tan precipitate was obtained. Precipitate collected by vacuum filtration and washed with water and acetone. Crystallization from water or 0.1% NaOH (1 g l⁻¹) gives a white material. Crystals for the X-ray structure determination obtained by slow cooling of an aqueous solution. Long needle parallel to a axis, dimensions 0.06 × 0.08 × 1.3 mm. CAD-4 diffractometer, θ –2 θ scans. θ -scan range (1.2 + 0.34 tan θ)°. Scan speed

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1.8–5.5° min⁻¹. Background first and last 1/6 of scan range. Graphite-monochromated Mo K α radiation. Unit cell, 25 reflections, $7 < \theta < 20^\circ$. No absorption correction. $(\sin \theta)/\lambda_{\max} = 0.540 \text{ \AA}^{-1}$. Index ranges $-5 \leq h \leq 5$, $-11 \leq k \leq 11$, $-12 \leq l \leq 12$, 3222 reflections measured, equivalent reflections averaged $wR_F = 0.022$, no significant variation for standard reflections 045, 024. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). Least-squares refinement minimized $\sum w(\Delta F)^2$, $w = [\sigma_c^2(F) + 0.015F^2]^{-1}$, $\sigma_c^2(F)$ based on counting statistics. Scale factor, isotropic type-II extinction parameter [mosaic block size = $4.7(7) \times 10^{-5} \text{ mm}$ (Larson, 1970)], positional parameters, anisotropic thermal parameters for C, N and isotropic thermal parameters for H refined. $R = 0.054$, $wR = 0.067$, $S = 3.9$. Poor quality refinement probably caused by systematic errors resulting from extreme length of crystals, which could not be cut without shattering. Final ΔF Fourier synthesis $-0.24 \leq \Delta \rho < 0.24 \text{ e \AA}^{-3}$. Final max. $\Delta \xi_i/\sigma \xi_i = 0.01$. Scattering factors f , f' , f'' from *International Tables for X-ray Crystallography* (1974). Calculations on a CDC-7600 using the Los Alamos Crystal Structure System developed primarily by A. C. Larson.* The

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43814 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

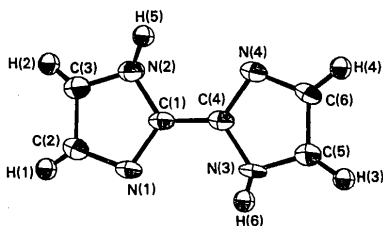


Fig. 1. ORTEP drawing of the molecule. Thermal ellipsoids are 50% probability. H atoms were given equal values of $B = 2.0 \text{ \AA}^2$ for clarity.

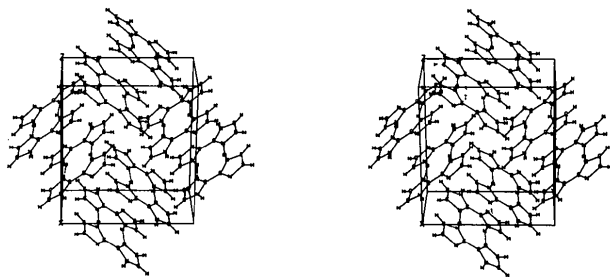


Fig. 2. Stereodrawing of the structure. Hydrogen bonds are shown as dotted lines. H atoms are in idealized positions. The view direction is approximately along a^* .

Table 1. Positional ($\times 10^4$ for C, N; $\times 10^3$ for H) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^2$)

	$U_{eq} = \frac{1}{3} \sum U_{ii}$			
	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
N(1)	-0397 (7)	0190 (4)	3238 (3)	3.8 (4)
N(2)	3937 (8)	0061 (4)	3199 (4)	3.8 (4)
N(3)	0777 (9)	2287 (4)	4910 (4)	3.8 (5)
N(4)	5031 (7)	2274 (4)	4760 (4)	4.2 (5)
C(1)	2052 (8)	0682 (5)	3639 (4)	3.1 (5)
C(2)	-0001 (10)	-0764 (6)	2524 (5)	4.5 (6)
C(3)	2685 (10)	-0851 (5)	2493 (5)	4.4 (6)
C(4)	2644 (9)	1722 (4)	4415 (4)	3.3 (5)
C(5)	1990 (9)	3240 (5)	5589 (5)	4.5 (6)
C(6)	4606 (10)	3223 (6)	5491 (5)	4.9 (7)
H(1)	-117 (10)	-127 (5)	216 (5)	7 (2)
H(2)	347 (9)	-142 (4)	212 (4)	5 (2)
H(3)	129 (9)	387 (4)	608 (4)	5 (1)
H(4)	599 (8)	377 (4)	589 (4)	3 (1)
H(5)	547 (15)	045 (7)	317 (6)	14 (3)
H(6)	-062 (10)	219 (4)	482 (4)	4 (2)

Table 2. Bond distances (\AA) and angles ($^\circ$)

C(1)–N(1)	1.336 (13)	N(1)–C(1)–N(2)	110.7 (4)
C(4)–N(4)	1.329 (12)	N(4)–C(4)–N(3)	110.3 (4)
C(1)–N(2)	1.351 (11)	N(1)–C(1)–C(4)	125.6 (4)
C(4)–N(3)	1.351 (13)	N(4)–C(4)–C(1)	126.5 (4)
C(2)–N(1)	1.358 (7)	N(2)–C(1)–C(4)	123.7 (4)
C(6)–N(4)	1.371 (7)	N(3)–C(4)–C(1)	123.3 (4)
C(3)–N(2)	1.344 (12)	N(1)–C(2)–C(3)	110.2 (5)
C(5)–N(3)	1.355 (12)	N(4)–C(6)–C(5)	110.4 (5)
C(2)–C(3)	1.373 (7)	N(1)–C(2)–H(1)	127.5 (35)
C(5)–C(6)	1.354 (7)	N(4)–C(6)–H(4)	122.8 (23)
C(2)–H(1)	0.85 (5)	H(1)–C(2)–C(3)	122.2 (35)
C(6)–H(4)	0.94 (4)	H(4)–C(6)–C(5)	126.8 (24)
C(3)–H(2)	0.89 (4)	C(2)–C(3)–N(2)	105.9 (5)
C(5)–H(3)	1.00 (4)	C(6)–C(5)–N(3)	105.7 (5)
N(2)–H(5)	0.89 (7)	C(2)–C(3)–H(2)	127.7 (30)
N(3)–H(6)	0.70 (5)	C(6)–C(5)–H(3)	122.0 (26)
C(1)–C(4)	1.423 (8)	N(2)–C(3)–H(2)	126.4 (30)
		N(3)–C(5)–H(3)	132.2 (26)
		C(1)–N(1)–N(2)	105.1 (4)
		C(4)–N(4)–C(6)	105.3 (4)
		C(1)–N(2)–C(3)	108.1 (4)
		C(4)–N(3)–C(5)	108.3 (4)
		C(1)–N(2)–H(5)	118.0 (49)
		C(4)–N(3)–H(6)	130.2 (41)
		H(5)–N(2)–C(3)	128.9 (48)
		H(6)–N(3)–C(5)	121.2 (41)

Hydrogen bonds

N(2)–N(1)	2.865 (5)	N(2)–H(5)···N(1)	143 (7)
N(3)–N(4)	2.879 (8)	N(3)–H(6)···N(4)	165 (5)
H(5)···N(1)	2.10 (8)		
H(6)···N(4)	2.19 (5)		

molecule with the atom-number sequence used is shown in Fig. 1. Final parameters are given in Table 1. Bond lengths and angles, none of which is unusual, are given in Table 2. The molecule has an approximate, non-crystallographic twofold axis and bonds and angles so related are adjacent in Table 2. The H-atom positions have large standard deviations so that distances and angles involving H are not particularly accurate. A stereoview of the structure is shown in Fig. 2.

Related literature. Kaiser, Saillant, Butler & Rasmussen (1976*a,b*) discuss the preparation and structures of Rh and Ir complexes of biimidazole.

References

- DEBUS, H. (1858). *Liebigs Ann. Chem.* **107**, 199–208.
 FIESELMANN, B. F., HENDRICKSON, D. N. & STUCKY, G. D. (1978). *Inorg. Chem.* **17**, 2078–2084.
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
 KAISER, S. W., SAILLANT, R. B., BUTLER, W. M. & RASMUSSEN, P. G. (1976*a*). *Inorg. Chem.* **15**, 2681–2687.
 KAISER, S. W., SAILLANT, R. B., BUTLER, W. M. & RASMUSSEN, P. G. (1976*b*). *Inorg. Chem.* **15**, 2688–2694.
 LARSON, A. C. (1970). In *Crystallographic Computing*, p. 291. Copenhagen: Munksgaard.

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Stereochemical Studies of Oligomers. XXIII.* 2,5-Hexanediyl Bis(*o*-chlorobenzoate)

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Abstract. $C_{20}H_{20}Cl_2O_4$, $M_r = 395.3$, monoclinic, $P2_1/c$, $a = 11.501(2)$, $b = 8.203(3)$, $c = 11.010(3)$ Å, $\beta = 112.80(5)^\circ$, $V = 957.6(6)$ Å³, $Z = 2$, $D_x = 1.37$ g cm⁻³, $Mo K\alpha$, $\lambda = 1.5418$ Å, $\mu = 32.84$ cm⁻¹, $F(000) = 412$, room temperature, $R = 0.036$ for 1207 unique observed reflections. The conformation of the chain which is *trans* in the central part [O(2)–C(8)–C(9)–C(9') = 173.0(2), C(8)–C(9)–C(9')–C(8') = 180°] is far from this arrangement approaching the carboxylic moieties [C(7)–O(2)–C(8)–C(9) = 154.3(2)°]. The carboxylic group is tilted by 46.5(1)° with respect to the phenyl-ring plane. The methyl groups, like the halogens, are *trans* to each other.

Experimental. Prismatic specimen approximately $0.4 \times 0.6 \times 0.9$ mm obtained by slow evaporation of a benzene/petroleum ether mixture at room temperature.

Intensity data collected at room temperature on a Siemens AED single-crystal diffractometer equipped with a General Automation Jumbo 220 computer with Ni-filtered $Cu K\alpha$ radiation. Lattice parameters from least-squares fit with 29 reflections up to $\theta_{max} = 28.35^\circ$ automatically centered on the diffractometer. The intensities evaluated from a modified version (Belletti, Uguzzoli, Cantoni & Pasquinelli, 1979) of the Lehmann

& Larsen (1974) method. One check reflection, monitored every 50 counts, with only random deviations. Lp correction, absorption ignored.

1986 reflections measured, 1821 unique, $R_{int} = 0.016$, of which 1207 with $I \geq 2\sigma(I)$ used in the refinement, $3 \leq \theta \leq 70^\circ$, $-14 \leq h \leq 12$, $0 \leq k \leq 10$, $0 \leq l \leq 12$. The structure was solved by direct methods with *SHELX76* (Sheldrick, 1976). Refinement by full-matrix least squares using *SHELX* with anisotropic temperature factors for all non-H atoms. H atoms, found in a ΔF map, isotropic. $\sum w(\Delta F)^2$ minimized, 158 refined parameters, final $R = 0.036$ and $wR = 0.039$, $w = 1.0/(\sigma^2 F + 0.00272 F^2)$, $(\Delta/\sigma)_{max}$ was 0.23 for heavy atoms and $(\Delta/\sigma)_{max} = 0.20$ e Å⁻³.

All the calculations were performed on a Gould SEL 32/77 computer. Atomic scattering factors were taken from *SHELX*.

The positional parameters with equivalent values of the anisotropic temperature factors are given in Table 1. In Table 2 bond lengths and angles are listed. Fig. 1 shows the molecule and the numbering scheme.†

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43874 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Part XXII: Bocelli & Grenier-Loustalot (1987).