Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (II)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N1	0.35661 (7)	0.43268 (6)	0.13688 (10)	0.0462 (3)
C2	0.32267 (8)	0.49537 (8)	0.10684 (11)	0.0436 (3)
C3	0.36508 (8)	0.57259 (8)	0.08945 (11)	0.0440 (3)
N4	0.45564 (7)	0.57210 (7)	0.11698 (10)	0.0473 (3)
C5	0.48912 (8)	0.50968 (8)	0.14906 (11)	0.0450 (3)
C6	0.44772 (8)	0.43169 (8)	0.16126 (11)	0.0448 (3)
O21	0.23921 (6)	0.49959 (5)	0.08198 (9)	0.0550 (3)
C22	0.19135 (10)	0.42947 (9)	0.0962 (2)	0.0670 (5)
O51	0.57249 (6)	0.50568 (6)	0.17541 (9)	0.0587 (3)
C52	0.61902 (10)	0.57731 (11)	0.17297 (14)	0.0692 (5)
C1′	0.32039 (8)	0.63625 (7)	0.15598 (11)	0.0431 (3)
C2′	0.25669 (9)	0.67887 (8)	0.10408 (13)	0.0534 (4)
C3′	0.21616 (10)	0.73733 (9)	0.1638 (2)	0.0647 (4)
C4′	0.23830 (10)	0.75341 (9)	0.2765 (2)	0.0673 (4)
C5′	0.30127 (11)	0.71126 (9)	0.32934 (14)	0.0633 (4)
C6′	0.34231 (9)	0.65298 (8)	0.26967 (12)	0.0523 (3)
C1″	0.46220 (7)	0.39571 (8)	0.27945 (11)	0.0441 (3)
C2″	0.50493 (10)	0.32704 (9)	0.29123 (15)	0.0619 (4)
C3″	0.51408 (12)	0.29264 (11)	0.3988 (2)	0.0768 (5)
C4″	0.48210 (11)	0.32742 (11)	0.4955 (2)	0.0695 (5)
C5″	0.44001 (11)	0.39596 (11)	0.48541 (14)	0.0695 (5)
C6″	0.42981 (10)	0.42958 (9)	0.37787 (13)	0.0594 (4)

Table 3. *Selected geometric parameters (Å, °)*

Compound (I)		Compound (II)	
N1—C2^i	1.263 (2)	N1—C2	1.261 (2)
		N4—C5	1.261 (2)
N1—C3	1.464 (2)	N1—C6	1.462 (2)
		N4—C3	1.461 (2)
C2—C3	1.508 (2)	C2—C3	1.512 (2)
		C5—C6	1.510 (2)
C2—O21	1.354 (2)	C2—O21	1.347 (2)
		C5—O51	1.349 (2)
O21—C22	1.431 (2)	O21—C22	1.442 (2)
		O51—C52	1.444 (2)
C2^i—N1—C6	117.69 (13)	C2—N1—C6	118.61 (11)
		C5—N4—C3	118.49 (11)
N1^i—C2—C3	128.58 (14)	N1—C2—C3	127.95 (12)
		N4—C5—C6	128.17 (12)
N1—C3—C2	113.63 (12)	N4—C3—C2	113.41 (11)
		N1—C6—C5	113.28 (11)
N1^i—C2—O21	121.52 (14)	N1—C2—O21	121.25 (12)
		N4—C5—O51	121.14 (12)
O21—C2—C3	109.90 (13)	O21—C2—C3	110.77 (11)
		O51—C5—C6	110.67 (12)
C2—O21—C22	117.13 (13)	C2—O21—C22	116.11 (11)
		C5—O51—C52	116.42 (12)
N1^i—C2—O21—C22	−0.3 (2)	N1—C2—O21—C22	2.3 (2)
		N4—C5—O51—C52	4.4 (2)

Symmetry code: (i) $\frac{3}{2} - x, \frac{3}{2} - y, 1 - z$.

H atoms, revealed in a difference synthesis, were refined with fixed individual displacement parameters [$U(H) = 1.5U_{eq}(C_{methyl})$ or $1.2U_{eq}(C_{other})$] using a riding model with aromatic C—H = 0.93, tertiary C—H = 0.98 and methyl C—H = 0.96 Å. The methyl groups were also allowed to rotate about their local threefold axes.

For both compounds, data collection: *DIF*4 (Stoe & Cie, 1988*a*); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1988*b*); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1094). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Stoe & Cie (1988*a*). *DIF4. Diffractometer Control Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988*b*). *REDU4. Data Reduction Program.* Version 6.2. Stoe & Cie, Darmstadt, Germany.

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2,2′-Bi(1*H*-imidazolium) Dichloride

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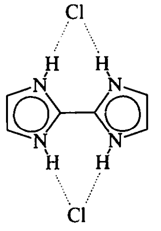
Abstract

The basic structural unit in the title compound, 2,2′-bi(1*H*-imidazol-3-ium) dichloride, $C_6H_8N_4^{2+}.2Cl^-$, consists of a planar diprotonated biimidazole moiety and two Cl^- ions each connected to the dication *via* a pair of

N—H···Cl bonds. The cation occupies a site of crystallographic 2/*m* symmetry with the twofold axis running through the connecting C2—C2′ bond and the middle of the ring C3—C3′ bonds. Contacts of Cl^- ions with H atoms in adjacent units generate infinite layers in the (210) planes.

Comment

A drawing of the title molecule (I) is shown in Fig. 1 with the numbering scheme. The asymmetric unit corresponds to one fourth of a diprotonated biimidazole cation ($biimH_4^{2+}$) and one half of a Cl^- anion. The cation sits on a site of 2/*m* symmetry, C2 being located on the twofold axis. The imidazole rings are planar to within 1σ (0.002 Å) and the two imidazolyl rings are coplanar by symmetry. Protonation induces large variations in the geometry of $biimH_2$ (Cromer, Ryan & Storm, 1987): the ring angle at the protonation site is increased by 3.7° and the adjacent angles are reduced by 2.7 and 3.1° while the more remote angles undergo small increases (1.4 and 0.7°) keeping the ring planar. A similar effect is noted for protonation of anionic tetranitrobiimidazolate (Cromer & Storm, 1990*a*) to the neutral form (Cromer & Storm, 1990*b*). Bond lengths probably undergo small variations upon protonation, but the relatively high standard deviations for $biimH_2$ preclude conclusions in this case.



(I)

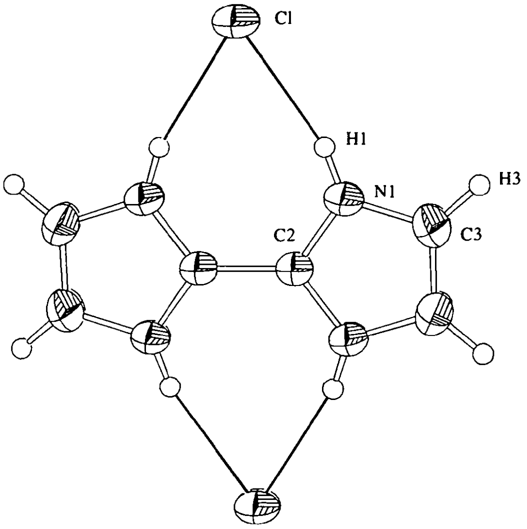


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the molecule. Ellipsoids correspond to 50% probability. H atoms are drawn as spheres of arbitrary size.

Two Cl^- anions in a mirror plane are in close contact with the $biimH_4^{2+}$ cation. Each anion forms hydrogen bonds with two N—H groups [N1···Cl 3.067 (2), H···Cl 2.28 (3) Å, N—H···Cl 165 (2)°]. The Cl^- ions are displaced by 0.042 (6) Å from the plane of the biimidazole rings. They also establish non-bonded contacts of *ca* 2.69 Å with H3 atoms in adjacent cations, thereby creating slightly puckered sheets in the (210) planes (Fig. 2).

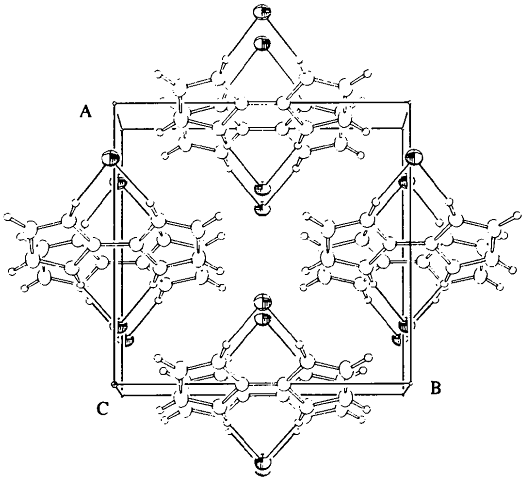


Fig. 2. *ORTEP*II (Johnson, 1976) drawing of the unit cell down the *b* axis. Ellipsoids correspond to 30% probability. H atoms are drawn as spheres of arbitrary size.

Experimental

2,2′-Biimidazole was prepared as described by Cromer, Ryan & Storm (1987) and the dihydrochloride was first isolated as a reaction by-product during work on rhenium(V)-oxo chemistry with biimidazole. The crystal used for the X-ray work was isolated from a 1:1 acetone:HCl 1*M* mixture.

Crystal data

$C_6H_8N_4^{2+}.2Cl^-$
$M_r = 207.06$
Monoclinic
$C2/m$
$a = 9.500 (2)$ Å
$b = 10.031 (3)$ Å
$c = 4.910 (2)$ Å
$\beta = 97.40 (2)°$
$V = 464.0 (3)$ Å^3
$Z = 2$
$D_x = 1.482$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 20.00–25.00°$
$\mu = 6.05$ mm^{-1}
$T = 293 (2)$ K
Plate
0.31 × 0.21 × 0.09 mm
Light yellow

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: by integration from crystal shape
$T_{min} = 0.24$, $T_{max} = 0.61$
1502 measured reflections
467 independent reflections
440 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.053$
$\theta_{max} = 69.80°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -5 \rightarrow 5$
6 standard reflections
frequency: 30 min
intensity decay: 0.7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.109$
$S = 1.12$
467 reflections
39 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0845P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.03$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0043 (18)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl	0.23662 (7)	0	0.50616 (13)	0.0467 (4)
N1	0.4255 (2)	−0.1499 (2)	0.1468 (3)	0.0397 (5)
H1	0.373 (3)	−0.124 (2)	0.253 (5)	0.045 (6)
C2	1/2	−0.0719 (2)	0	0.0339 (6)
C3	0.4536 (3)	−0.2799 (2)	0.0916 (5)	0.0508 (6)
H3	0.415 (3)	−0.351 (4)	0.182 (5)	0.068 (9)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.328 (2)	C2—C2^i	1.442 (5)
N1—C3	1.366 (3)	C3—C3^{ii}	1.338 (5)
N1—H1	0.81 (3)	C3—H3	0.94 (4)
C2—N1—C3	108.9 (2)	N1—C2—C2^i	126.1 (1)
C2—N1—H1	125 (2)	C3^{ii}—C3—N1	107.2 (1)
C3—N1—H1	126 (2)	C3^{ii}—C3—H3	131 (2)
N1^{ii}—C2—N1	107.8 (2)	N1—C3—H3	122 (2)

Symmetry codes: (i) $1 - x, -y, -z$; (ii) $1 - x, y, -z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NRC Crystallographic Computer Programs* (Ahmed, Hall, Pippy & Huber, 1973). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976; Gabe, Le Page, Charland, Lee & White, 1989). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1258). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,2-[3,4-Dihydro-2-(4-methoxyphenyl)-2*H*-pyrrolo][60]fullerene Carbon Disulfide

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Abstract

The title fullerene, $C_{69}H_9NO.CS_2$, is a low-symmetry derivative of C_{60} and does not show the known disorder of the unsubstituted fullerene molecule. Contrary to spectroscopic investigations, the C_s symmetry of the molecule is no longer present in the crystal. The two bridging atoms, C1 and C2, 'stand out' of the C_{60} surface.

Comment

Disorder plays a crucial role when investigating solid structures of fullerenes, and a number of static and