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2,2′-Ethylenediisoquinolinium dibromide dihydrate

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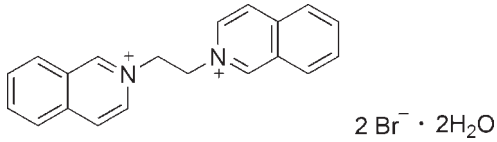
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.078; data-to-parameter ratio = 18.0.

In the title compound, $C_{20}H_{18}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$, the complete dication is generated by a crystallographic centre of symmetry. In the crystal, O—H···Br, C—H···Br and C—H···O hydrogen bonds and π–π stacking [shortest centroid–centroid separation = 3.657 (2) Å] help to establish the packing.

Related literature

For background to supramolecular chemistry related to the title compound, see: Loeb & Wisner (1998); Li (2007). For related structures, see: Li *et al.* (2008); Xu *et al.* (2007); Fan *et al.* (2007).



Experimental

Crystal data

$C_{20}H_{18}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$
M_r = 482.22
Triclinic, $P\overline{1}$
a = 7.5203 (15) Å
b = 8.0749 (16) Å
c = 9.2059 (18) Å
α = 110.34 (3)°
β = 106.96 (3)°
γ = 97.26 (3)°
V = 484.9 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 4.20 mm^{-1}
T = 113 K
0.18 × 0.16 × 0.14 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.519, T_{max} = 0.591
3994 measured reflections
2262 independent reflections
1800 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.078
S = 1.07
2262 reflections
126 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···Br1^{i}	0.90 (4)	2.41 (4)	3.308 (3)	176 (3)
O1—H1B···Br1^{ii}	0.81 (5)	2.51 (5)	3.313 (3)	178 (5)
C1—H1···Br1^{iii}	0.95	2.84	3.593 (3)	137
C9—H9···Br1^{iv}	0.95	2.81	3.691 (3)	154
C10—H10B···Br1^{iv}	0.99	2.87	3.683 (3)	140
C3—H3···O1^{v}	0.95	2.57	3.396 (4)	145
C4—H4···O1^{vi}	0.95	2.54	3.380 (4)	147
C10—H10A···O1^{iii}	0.99	2.27	3.214 (4)	158

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z$; (iii) $x, y+1, z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $-x+1, -y+2, -z+1$; (vi) $x-1, y+1, z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5199).

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supporting information

Acta Cryst. (2009). E**65**, o2966 [https://doi.org/10.1107/S1600536809045036]

2,2′-Ethylenediisoquinolinium dibromide dihydrate

Jiang-Sheng Li and Peng-Yu Li

S1. Comment

As part of our ongoing studies of analogs of 1,2-bis(pyridinium) ethane dications (Li *et al.*, 2008), we synthesized a new dication 1,2-bis(isoquinolinium)ethane. Herein, its crystal structure is reported.

The molecular structure of (I) is shown in Fig. 1. The molecule has a centre of symmetry at the mid-point of the C10—C10A bond. The two isoquinoline rings are parallel to each other. The $N^{+}\cdots N^{+}$ distance in the title compound is 3.7609 (8) Å, similar to the value previously reported (*ca* 3.75 Å) in the 1,2-bis(pyridinium)ethane dication (Loeb & Wisner, 1998). The crystal structure is stabilized by a series of intermolecular hydrogen bonds (Table 1). The hydrate tends to form an extensive network in the crystal by the aid of Br anions and water molecules. Also, the title cation were stacked *via* π-π interactions between isoquinolinium rings.

S2. Experimental

The title compound was obtained according to the method of Loeb and Wisner (1998). Light yellow blocks of (I) were grown from its aqueous solution.

S3. Refinement

The water H atoms were positioned geometrically to acheive a reasonable hydrogen-bonding scheme. The other H atoms were positioned geometrically, with C—H = 0.95 Å for aromatic H and 0.99 Å for methyl H, and were constrained to ride on their parent atoms, with U_{iso}(H) =1.2U_{eq}(C).

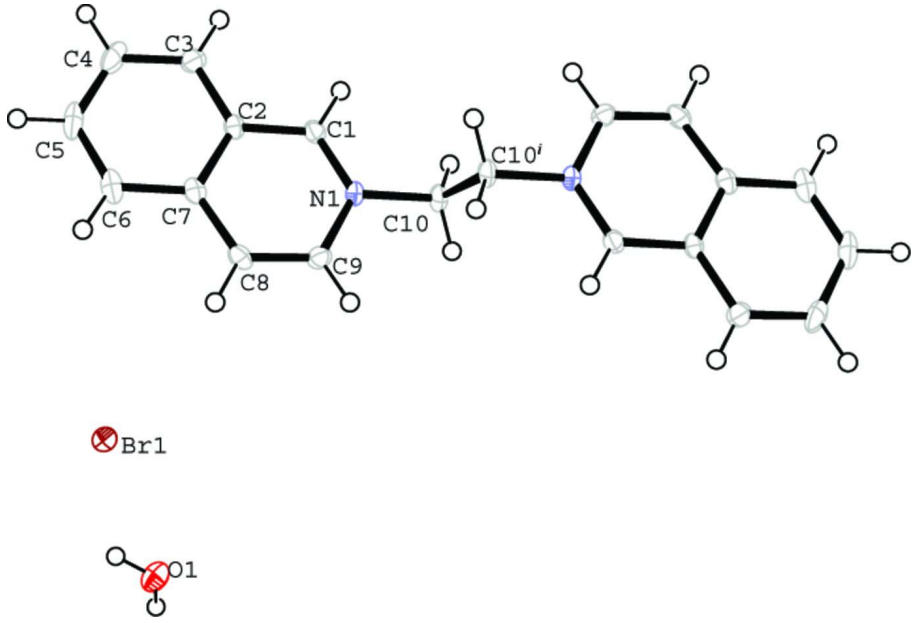

Figure 1

The molecular structure of (I) showing 50% probability displacement displacement ellipsoids. [Symmetry codes: (i) 1 - *x*, 2 - *y*, 2 - *z*.]

2,2′-Ethylenediisoquinolinium dibromide dihydrate

Crystal data

$C_{20}H_{18}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$
$M_r = 482.22$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.5203\ (15)$ Å
$b = 8.0749\ (16)$ Å
$c = 9.2059\ (18)$ Å
$\alpha = 110.34\ (3)°$
$\beta = 106.96\ (3)°$
$\gamma = 97.26\ (3)°$
$V = 484.9\ (2)$ Å^3
$Z = 1$
$F(000) = 242$
$D_x = 1.651$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1667 reflections
$\theta = 2.5–27.9°$
$\mu = 4.20$ mm^{-1}
$T = 113$ K
Block, light yellow
0.18 × 0.16 × 0.14 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.519$, $T_{max} = 0.591$
3994 measured reflections
2262 independent reflections
1800 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.5°$
$h = -8\rightarrow 9$
$k = -8\rightarrow 10$
$l = -11\rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.078$
$S = 1.07$
2262 reflections
126 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.30313 (4)	0.40880 (4)	0.11468 (3)	0.02152 (11)
N1	0.4507 (3)	0.9986 (3)	0.7872 (2)	0.0144 (5)
C1	0.3923 (4)	1.1342 (4)	0.7547 (3)	0.0158 (5)
H1	0.4226	1.2506	0.8429	0.019*
C2	0.2857 (4)	1.1081 (4)	0.5912 (3)	0.0148 (5)
C3	0.2230 (4)	1.2538 (4)	0.5580 (3)	0.0211 (6)
H3	0.2509	1.3701	0.6457	0.025*
C4	0.1210 (4)	1.2239 (4)	0.3969 (4)	0.0263 (7)
H4	0.0762	1.3196	0.3727	0.032*
C5	0.0829 (4)	1.0516 (5)	0.2675 (3)	0.0260 (7)
H5	0.0148	1.0340	0.1564	0.031*
C6	0.1411 (4)	0.9094 (4)	0.2972 (3)	0.0226 (6)
H6	0.1120	0.7941	0.2078	0.027*
C7	0.2446 (4)	0.9341 (4)	0.4610 (3)	0.0159 (5)
C8	0.3097 (4)	0.7935 (4)	0.5028 (3)	0.0182 (6)
H8	0.2827	0.6753	0.4180	0.022*
C9	0.4100 (4)	0.8262 (4)	0.6625 (3)	0.0174 (6)
H9	0.4525	0.7310	0.6893	0.021*
C10	0.5660 (4)	1.0297 (4)	0.9599 (3)	0.0173 (6)
H10A	0.6328	1.1608	1.0248	0.021*
H10B	0.6643	0.9589	0.9589	0.021*
O1	0.8807 (4)	0.4163 (3)	0.1546 (3)	0.0267 (5)
H1A	0.994 (5)	0.408 (4)	0.142 (4)	0.022 (9)*
H1B	0.838 (6)	0.458 (6)	0.088 (5)	0.067 (15)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02605 (18)	0.02344 (16)	0.01838 (16)	0.01248 (12)	0.00851 (12)	0.00973 (12)
N1	0.0142 (11)	0.0168 (11)	0.0109 (10)	0.0027 (9)	0.0026 (9)	0.0062 (9)
C1	0.0194 (14)	0.0150 (13)	0.0136 (12)	0.0039 (11)	0.0073 (11)	0.0056 (10)
C2	0.0160 (13)	0.0176 (13)	0.0138 (12)	0.0027 (11)	0.0079 (11)	0.0083 (11)
C3	0.0245 (15)	0.0259 (15)	0.0236 (14)	0.0127 (13)	0.0146 (12)	0.0152 (13)
C4	0.0251 (16)	0.0406 (19)	0.0315 (16)	0.0139 (14)	0.0149 (14)	0.0297 (15)
C5	0.0162 (15)	0.0469 (19)	0.0175 (14)	0.0038 (14)	0.0034 (12)	0.0197 (14)
C6	0.0177 (15)	0.0316 (16)	0.0142 (13)	−0.0020 (13)	0.0041 (12)	0.0085 (12)
C7	0.0128 (13)	0.0211 (14)	0.0148 (12)	0.0016 (11)	0.0073 (11)	0.0074 (11)
C8	0.0210 (15)	0.0142 (13)	0.0145 (12)	0.0013 (11)	0.0060 (11)	0.0014 (11)
C9	0.0183 (14)	0.0150 (13)	0.0188 (13)	0.0052 (11)	0.0057 (11)	0.0073 (11)
C10	0.0177 (14)	0.0186 (14)	0.0109 (12)	0.0006 (11)	0.0004 (11)	0.0059 (11)
O1	0.0271 (13)	0.0288 (12)	0.0299 (11)	0.0089 (10)	0.0099 (10)	0.0182 (10)

Geometric parameters (Å, º)

N1—C1	1.324 (3)	C5—H5	0.9500
N1—C9	1.387 (3)	C6—C7	1.410 (4)
N1—C10	1.486 (3)	C6—H6	0.9500
C1—C2	1.409 (3)	C7—C8	1.417 (4)
C1—H1	0.9500	C8—C9	1.354 (4)
C2—C3	1.416 (4)	C8—H8	0.9500
C2—C7	1.418 (4)	C9—H9	0.9500
C3—C4	1.374 (4)	C10—C10^{i}	1.521 (5)
C3—H3	0.9500	C10—H10A	0.9900
C4—C5	1.408 (4)	C10—H10B	0.9900
C4—H4	0.9500	O1—H1A	0.90 (4)
C5—C6	1.364 (4)	O1—H1B	0.81 (5)
C1—N1—C9	121.6 (2)	C5—C6—H6	120.1
C1—N1—C10	120.1 (2)	C7—C6—H6	120.1
C9—N1—C10	118.3 (2)	C6—C7—C8	123.4 (3)
N1—C1—C2	120.9 (2)	C6—C7—C2	118.5 (3)
N1—C1—H1	119.5	C8—C7—C2	118.1 (2)
C2—C1—H1	119.5	C9—C8—C7	120.6 (2)
C1—C2—C3	120.5 (2)	C9—C8—H8	119.7
C1—C2—C7	118.6 (2)	C7—C8—H8	119.7
C3—C2—C7	120.9 (2)	C8—C9—N1	120.1 (3)
C4—C3—C2	118.9 (3)	C8—C9—H9	119.9
C4—C3—H3	120.6	N1—C9—H9	119.9
C2—C3—H3	120.6	N1—C10—C10^{i}	109.4 (3)
C3—C4—C5	120.1 (3)	N1—C10—H10A	109.8
C3—C4—H4	119.9	C10^{i}—C10—H10A	109.8
C5—C4—H4	119.9	N1—C10—H10B	109.8
C6—C5—C4	121.8 (3)	C10^{i}—C10—H10B	109.8

C6—C5—H5	119.1	H10A—C10—H10B	108.2
C4—C5—H5	119.1	H1A—O1—H1B	99 (4)
C5—C6—C7	119.8 (3)		

C9—N1—C1—C2	0.0 (4)	C1—C2—C7—C6	−178.8 (2)
C10—N1—C1—C2	178.9 (2)	C3—C2—C7—C6	0.5 (4)
N1—C1—C2—C3	179.6 (3)	C1—C2—C7—C8	1.3 (4)
N1—C1—C2—C7	−1.0 (4)	C3—C2—C7—C8	−179.3 (3)
C1—C2—C3—C4	179.4 (3)	C6—C7—C8—C9	179.4 (3)
C7—C2—C3—C4	0.0 (4)	C2—C7—C8—C9	−0.7 (4)
C2—C3—C4—C5	−1.0 (4)	C7—C8—C9—N1	−0.2 (4)
C3—C4—C5—C6	1.4 (5)	C1—N1—C9—C8	0.6 (4)
C4—C5—C6—C7	−0.9 (4)	C10—N1—C9—C8	−178.2 (3)
C5—C6—C7—C8	179.7 (3)	C1—N1—C10—C10^{i}	96.5 (3)
C5—C6—C7—C2	−0.1 (4)	C9—N1—C10—C10^{i}	−84.7 (4)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···Br1^{ii}	0.90 (4)	2.41 (4)	3.308 (3)	176 (3)
O1—H1*B*···Br1^{iii}	0.81 (5)	2.51 (5)	3.313 (3)	178 (5)
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Symmetry codes: (ii) *x*+1, *y*, *z*; (iii) −*x*+1, −*y*+1, −*z*; (iv) *x*, *y*+1, *z*+1; (v) −*x*+1, −*y*+1, −*z*+1; (vi) −*x*+1, −*y*+2, −*z*+1; (vii) *x*−1, *y*+1, *z*.