Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Octane-1,8-diyldipyridinium dibromide dihydrate

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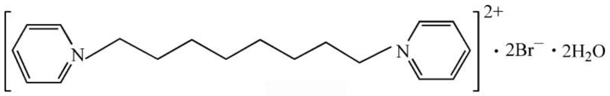
Received 23 November 2007; accepted 28 November 2007

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.077; data-to-parameter ratio = 23.0.

The asymmetric unit of the title compound, $C_{18}H_{26}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$, consists of one-half of the organic cation, one Br^- anion and one water molecule. The organic cation is situated on a centre of inversion. The dihedral angle between the pyridine ring and the plane of the central linkage is 59.3 (1)°. The cations, anions and water molecules are linked *via* O—H···Br, C—H···Br and C—H···O hydrogen bonds, forming a three-dimensional framework.

Related literature

For general background, see: Day *et al.* (2000, 2002); Freeman *et al.* (1981); Kim *et al.* (2000).



Experimental

Crystal data

$C_{18}H_{26}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$
M_r = 466.26
Orthorhombic, *Pbca*
a = 9.8329 (7) Å
b = 13.3000 (11) Å
c = 16.5688 (13) Å
V = 2166.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.75 mm^{-1}
T = 273 (2) K
0.32 × 0.24 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.380, T_{max} = 0.536 (expected range = 0.347–0.490)
21410 measured reflections
2503 independent reflections
1842 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.077
S = 1.01
2503 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WB···Br1	0.81	2.55	3.353 (2)	173
O1W—H1WA···Br1^{i}	0.84	2.57	3.392 (2)	169
C1—H1···Br1	0.93	2.82	3.596 (2)	141
C2—H2···O1W^{ii}	0.93	2.48	3.271 (3)	143
C5—H5···Br1^{iii}	0.93	2.67	3.588 (2)	168

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We acknowledge the support of the National Natural Science Foundation of China (No. 20662003) and the Foundation of the Governor of Guizhou Province, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2531).

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supplementary materials

Acta Cryst. (2008). E**64**, o183 [**doi:10.1107/S1600536807063970**]

Octane-1,8-diyldipyridinium dibromide dihydrate

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Comment

As part of our ongoing investigation on bipyridyl compounds, we present here the crystal structure of the title compound, (I), which can develop strong intermolecular interactions with cucurbit[*n*]urils (CB[*n*]) (Freeman *et al.*, 1981; Day *et al.*, 2000, 2002; Kim *et al.*, 2000).

The asymmetric unit of compound (I) (Fig. 1) consists of one-half of the organic cation, one Br^- anion and one lattice water molecule. The organic cation is situated on a centre of inversion which coincides with the midpoint of the C9—C9^i bond [symmetry code: (i) $1 - x$, $1 - y$, $-z$]. The two pyridine rings are parallel by virtue of the centre of symmetry. The dihedral angle between the pyridine ring and the central C6—C9/C6^i—C9^i chain is 59.3 (1)°. The cations, anions and water molecules are linked *via* O—H···Br, C—H···Br and C—H···O hydrogen bonds (Table 1) forming a three-dimensional framework.

Experimental

A solution of 1,8-dibromine-octane (2.72 g, 0.01 mol) was added to a stirred solution of pyridine (1.98 g, 0.025 mol) in 1,4-dioxane (50 ml) at 383 K for 5 h. After cooling to room temperature, the mixture was filtered. The solid product was dissolved in 80 ml water, and then set aside for four weeks to obtain colourless crystals of the title compound.

Refinement

Water H atoms were located in a difference Fourier map and refined as riding in their as-found positions relative to the parent O atom, with $U_{iso}(H) = 1.2U_{eq}(O)$. All other H atoms were placed in calculated positions and refined as riding, with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme for the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level. unlabelled atoms are related to labelled atoms by the symmetry operation ($1 - x$, $1 - y$, $-z$). Symmetry related bromide ion and water molecule are not shown.

Octane-1,8-diyldipyridinium dibromide dihydrate

Crystal data

$C_{18}H_{26}N_2^{2+}\cdot 2(Br^-)\cdot 2H_2O$	$F_{000} = 952$
$M_r = 466.26$	$D_x = 1.429$ Mg m^{-3}

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

a = 9.8329 (7) Å

b = 13.3000 (11) Å

c = 16.5688 (13) Å

V = 2166.8 (3) Å^3

Z = 4

Mo $K\alpha$ radiation
λ = 0.71073 Å

Cell parameters from 21410 reflections

θ = 2.5–27.6°

μ = 3.75 mm^{-1}

T = 273 (2) K

Prism, colourless

0.32 × 0.24 × 0.19 mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 273(2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

T_{min} = 0.380, T_{max} = 0.536

21410 measured reflections

2503 independent reflections

1842 reflections with $I > 2\sigma(I)$

R_{int} = 0.034

θ_{max} = 27.6°

θ_{min} = 2.5°

$h = -12 \rightarrow 12$

$k = -17 \rightarrow 17$

$l = -15 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.029

$wR(F^2)$ = 0.077

S = 1.01

2503 reflections

109 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.8723P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}}$ = 0.001

$\Delta\rho_{\text{max}}$ = 0.25 e Å^{-3}

$\Delta\rho_{\text{min}}$ = −0.39 e Å^{-3}

Extinction correction: none

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 > 2sigma(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	0.28891 (2)	0.652327 (19)	0.328729 (15)	0.05280 (11)
N1	0.44180 (18)	0.35645 (13)	0.32104 (10)	0.0376 (4)
C1	0.3916 (3)	0.40288 (17)	0.38614 (14)	0.0507 (6)
H1	0.3246	0.4517	0.3801	0.061*
C2	0.4380 (3)	0.3791 (2)	0.46131 (16)	0.0642 (8)
H2	0.4014	0.4105	0.5065	0.077*
C3	0.5389 (3)	0.3088 (2)	0.46995 (16)	0.0660 (8)
H3	0.5725	0.2931	0.5209	0.079*
C4	0.5895 (3)	0.2622 (2)	0.40288 (15)	0.0588 (7)
H4	0.6582	0.2146	0.4077	0.071*
C5	0.5382 (2)	0.28617 (18)	0.32886 (13)	0.0475 (6)
H5	0.5707	0.2532	0.2832	0.057*
C6	0.3864 (2)	0.37965 (18)	0.23978 (13)	0.0455 (5)
H6A	0.3137	0.4285	0.2453	0.055*
H6B	0.3476	0.3189	0.2170	0.055*
C7	0.4916 (2)	0.42052 (18)	0.18213 (12)	0.0432 (5)
H7A	0.5633	0.3712	0.1749	0.052*
H7B	0.5320	0.4808	0.2048	0.052*
C8	0.4286 (2)	0.44515 (19)	0.10078 (13)	0.0444 (5)
H8A	0.3865	0.3850	0.0791	0.053*
H8B	0.3577	0.4950	0.1084	0.053*
C9	0.5310 (2)	0.48488 (17)	0.04014 (12)	0.0408 (5)
H9A	0.5766	0.5428	0.0633	0.049*
H9B	0.5992	0.4335	0.0306	0.049*
O1W	0.1270 (2)	0.6061 (2)	0.15455 (12)	0.0973 (8)
H1WA	0.0442	0.6131	0.1647	0.117*
H1WB	0.1689	0.6117	0.1965	0.117*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04958 (16)	0.05876 (17)	0.05006 (17)	0.00012 (12)	−0.00083 (11)	0.01642 (11)
N1	0.0424 (10)	0.0428 (10)	0.0277 (9)	−0.0036 (8)	0.0058 (7)	0.0044 (7)
C1	0.0627 (14)	0.0464 (13)	0.0429 (14)	0.0058 (12)	0.0161 (12)	0.0025 (11)
C2	0.094 (2)	0.0637 (16)	0.0345 (14)	−0.0017 (16)	0.0104 (14)	−0.0069 (12)
C3	0.081 (2)	0.0830 (19)	0.0341 (14)	−0.0090 (17)	−0.0084 (14)	0.0108 (14)
C4	0.0601 (15)	0.0672 (17)	0.0492 (15)	0.0092 (13)	−0.0038 (13)	0.0141 (13)
C5	0.0528 (14)	0.0527 (14)	0.0368 (13)	0.0072 (11)	0.0061 (11)	0.0008 (11)
C6	0.0437 (12)	0.0601 (13)	0.0326 (12)	−0.0035 (11)	−0.0004 (10)	0.0098 (11)
C7	0.0447 (12)	0.0539 (13)	0.0311 (12)	−0.0047 (11)	0.0010 (10)	0.0064 (10)
C8	0.0437 (12)	0.0590 (14)	0.0305 (11)	−0.0035 (10)	−0.0010 (10)	0.0069 (10)
C9	0.0402 (11)	0.0540 (13)	0.0281 (11)	−0.0067 (10)	−0.0031 (9)	0.0036 (10)
O1W	0.0625 (12)	0.177 (3)	0.0519 (12)	0.0086 (16)	−0.0006 (10)	−0.0161 (14)

Geometric parameters (Å, °)

N1—C1	1.337 (3)	C6—H6A	0.97
N1—C5	1.337 (3)	C6—H6B	0.97
N1—C6	1.485 (3)	C7—C8	1.520 (3)
C1—C2	1.364 (4)	C7—H7A	0.97
C1—H1	0.93	C7—H7B	0.97
C2—C3	1.370 (4)	C8—C9	1.518 (3)
C2—H2	0.93	C8—H8A	0.97
C3—C4	1.366 (4)	C8—H8B	0.97
C3—H3	0.93	C9—C9^{i}	1.518 (4)
C4—C5	1.364 (3)	C9—H9A	0.97
C4—H4	0.93	C9—H9B	0.97
C5—H5	0.93	O1W—H1WA	0.84
C6—C7	1.509 (3)	O1W—H1WB	0.81
C1—N1—C5	120.42 (19)	C7—C6—H6B	108.9
C1—N1—C6	120.0 (2)	H6A—C6—H6B	107.7
C5—N1—C6	119.53 (18)	C6—C7—C8	111.07 (19)
N1—C1—C2	120.4 (2)	C6—C7—H7A	109.4
N1—C1—H1	119.8	C8—C7—H7A	109.4
C2—C1—H1	119.8	C6—C7—H7B	109.4
C1—C2—C3	119.7 (2)	C8—C7—H7B	109.4
C1—C2—H2	120.1	H7A—C7—H7B	108.0
C3—C2—H2	120.1	C9—C8—C7	113.04 (18)
C4—C3—C2	119.2 (2)	C9—C8—H8A	109.0
C4—C3—H3	120.4	C7—C8—H8A	109.0
C2—C3—H3	120.4	C9—C8—H8B	109.0
C5—C4—C3	119.4 (2)	C7—C8—H8B	109.0
C5—C4—H4	120.3	H8A—C8—H8B	107.8
C3—C4—H4	120.3	C9^{i}—C9—C8	113.9 (2)
N1—C5—C4	120.8 (2)	C9^{i}—C9—H9A	108.8
N1—C5—H5	119.6	C8—C9—H9A	108.8
C4—C5—H5	119.6	C9^{i}—C9—H9B	108.8
N1—C6—C7	113.42 (17)	C8—C9—H9B	108.8
N1—C6—H6A	108.9	H9A—C9—H9B	107.7
C7—C6—H6A	108.9	H1WA—O1W—H1WB	108.2
N1—C6—H6B	108.9		
C5—N1—C1—C2	0.0 (3)	C3—C4—C5—N1	1.8 (4)
C6—N1—C1—C2	177.4 (2)	C1—N1—C6—C7	120.1 (2)
N1—C1—C2—C3	1.5 (4)	C5—N1—C6—C7	−62.5 (3)
C1—C2—C3—C4	−1.3 (4)	N1—C6—C7—C8	−178.7 (2)
C2—C3—C4—C5	−0.3 (4)	C6—C7—C8—C9	−179.0 (2)
C1—N1—C5—C4	−1.6 (3)	C7—C8—C9—C9^{i}	−177.0 (2)
C6—N1—C5—C4	−179.0 (2)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1WB···Br1	0.81	2.55	3.353 (2)	173
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C2—H2···O1W^{iii}	0.93	2.48	3.271 (3)	143
C5—H5···Br1^{iv}	0.93	2.67	3.588 (2)	168

Symmetry codes: (ii) *x*−1/2, *y*, −*z*+1/2; (iii) −*x*+1/2, −*y*+1, *z*+1/2; (iv) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1