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2,2′-Dimethylbutane-1,4-diyldipyridinium dibromide dihydrate

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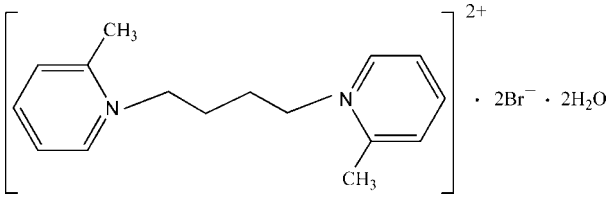
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 17.2.

In the crystal structure of the title compound, $C_{16}H_{22}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$, the organic cation exhibits an extended zigzag structure. The Br^- anions link with the solvent water molecules by O—H···Br hydrogen bonding. π–π stacking is observed between nearly parallel pyridine rings of adjacent molecules, the centroid-to-centroid distance between these rings being 3.887 (6) Å.

Related literature

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



Experimental

Crystal data

$C_{16}H_{22}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$
M_r = 438.21
Orthorhombic, $P2_12_12_1$
a = 7.3604 (12) Å
b = 9.4157 (14) Å
c = 28.6294 (19) Å
V = 1984.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.09 mm^{-1}
T = 293 (2) K
0.23 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.425, T_{max} = 0.545
13083 measured reflections
3436 independent reflections
2997 reflections with i > 2$\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.079
S = 1.08
3436 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}
Absolute structure: Flack (1983), 1399 Friedel pairs
Flack parameter: −0.002 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1WA···Br1	0.95	2.43	3.384 (3)	176
O1—H1WB···Br1^{i}	0.91	2.64	3.406 (4)	143
O2—H2WA···Br2	0.96	2.42	3.358 (4)	164
O2—H2WB···Br2^{ii}	0.99	2.41	3.344 (4)	159

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2371).

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supplementary materials

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2,2'-Dimethylbutane-1,4-diyldipyridinium dibromide dihydrate

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Comment

As part of our ongoing investigation on bibenzene compounds, we present a compound containing multiple functional groups that can develop strong intermolecular interactions with cucurbit[*n*]urils (CB[*n*]) (Freeman *et al.*, 1981; Day & Arnold, 2000; Day *et al.*, 2002; Kim *et al.*, 2000).

The crystal structure of the title compound (Fig. 1) consists of organic cations, Br^- anions and lattice water molecules. The two pyridine rings of organic cations are nearly parallel, the dihedral angle being 4.58 (13)°. The O—H···Br hydrogen bonds are observed between Br^- and the water molecule (Table 1). The highly ordered linear formation was aggregated by intermolecular π-π stacking between neighboring pyridine rings, the centroid-to-centroid distance of N1-pyridine ring to N2-pyridine ring is 3.887 (6) Å (symmetry codes: *x*,-1 + *y*,*z*).

Experimental

A solution of 1,4-dibromine-butane (2.16 g, 0.01 mol) was added to a stirred solution of 2-methylpyridine (2.33 g, 0.025 mol) in 1,4-dioxane (50 ml) at 110°C 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 three weeks to obtain colorless crystals.

Refinement

Water H atoms were located in a difference Fourier map and refined as riding in their as-found positions relative to O atoms 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, N—H = 0.86 Å, and U_{iso}(H) = 1.2–1.5 U_{eq}(C,*N*).

Figures

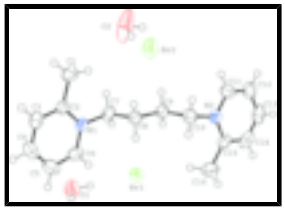


Fig. 1. The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,2'-Dimethylbutane-1,4-diyldipyridinium dibromide dihydrate

Crystal data

$C_{16}H_{22}N_2^{2+}\cdot 2Br^-\cdot 2H_2O$ F_{000} = 888

M_r = 438.21	D_x = 1.467 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 3436 reflections
a = 7.3604 (12) Å	θ = 1.4–25.0º
b = 9.4157 (14) Å	μ = 4.09 mm^{-1}
c = 28.6294 (19) Å	T = 293 (2) K
V = 1984.1 (5) Å^3	Prism, colorless
Z = 4	0.23 × 0.18 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3436 independent reflections
Radiation source: fine-focus sealed tube	2997 reflections with i > 2σ(I)
Monochromator: graphite	R_{int} = 0.035
T = 293(2) K	θ_{max} = 25.0º
φ and ω scan	θ_{min} = 1.4º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	h = −8→8
T_{min} = 0.425, T_{max} = 0.545	k = −11→10
13083 measured reflections	l = −34→34

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 0.1829P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.079	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.08	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
3436 reflections	$\Delta\rho_{min}$ = −0.49 e Å^{-3}
200 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1399 Friedels Pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.002 (13)

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 calculat-

ing *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}
C1	0.3499 (7)	0.3486 (5)	0.46614 (13)	0.0585 (12)
H1A	0.3618	0.4496	0.4696	0.088*
H1B	0.4662	0.3045	0.4708	0.088*
H1C	0.2655	0.3132	0.4889	0.088*
C2	0.2813 (5)	0.3147 (4)	0.41772 (12)	0.0380 (9)
C3	0.2511 (6)	0.1763 (4)	0.40263 (15)	0.0470 (10)
H3	0.2707	0.1013	0.4231	0.056*
C4	0.1930 (6)	0.1487 (5)	0.35815 (16)	0.0528 (11)
H4	0.1759	0.0556	0.3483	0.063*
C5	0.1599 (6)	0.2610 (5)	0.32796 (16)	0.0563 (12)
H5	0.1194	0.2440	0.2977	0.068*
C6	0.1871 (6)	0.3955 (5)	0.34302 (13)	0.0486 (11)
H6	0.1650	0.4707	0.3228	0.058*
C7	0.2788 (6)	0.5759 (4)	0.39991 (15)	0.0442 (10)
H7A	0.2652	0.5884	0.4334	0.053*
H7B	0.1890	0.6349	0.3845	0.053*
C8	0.4688 (5)	0.6224 (4)	0.38512 (13)	0.0401 (9)
H8A	0.4897	0.5950	0.3529	0.048*
H8B	0.5584	0.5746	0.4044	0.048*
C9	0.4917 (5)	0.7830 (4)	0.38998 (13)	0.0398 (9)
H9A	0.4731	0.8107	0.4223	0.048*
H9B	0.4016	0.8312	0.3710	0.048*
C10	0.6838 (6)	0.8269 (4)	0.37417 (14)	0.0435 (10)
H10A	0.7717	0.7962	0.3973	0.052*
H10B	0.7126	0.7801	0.3449	0.052*
C11	0.7408 (6)	1.0636 (5)	0.40605 (14)	0.0495 (11)
H11	0.7641	1.0185	0.4343	0.059*
C12	0.7511 (6)	1.2078 (5)	0.40395 (17)	0.0564 (12)
H12	0.7805	1.2606	0.4303	0.068*
C13	0.7174 (6)	1.2733 (4)	0.36221 (19)	0.0590 (13)
H13	0.7256	1.3716	0.3600	0.071*
C14	0.6711 (6)	1.1947 (5)	0.32338 (16)	0.0563 (12)
H14	0.6470	1.2405	0.2953	0.068*
C15	0.6601 (5)	1.0457 (4)	0.32589 (13)	0.0418 (9)
C16	0.6124 (7)	0.9537 (5)	0.28430 (13)	0.0645 (12)
H16A	0.7151	0.8959	0.2761	0.097*
H16B	0.5806	1.0131	0.2583	0.097*
H16C	0.5114	0.8938	0.2921	0.097*
N1	0.2468 (4)	0.4236 (3)	0.38756 (10)	0.0364 (7)
N2	0.6973 (4)	0.9831 (3)	0.36796 (10)	0.0381 (7)
O1	−0.2299 (5)	0.5469 (4)	0.28463 (13)	0.0811 (11)
H1WA	−0.1382	0.6180	0.2861	0.097*

H1WB	−0.1451	0.4919	0.2701	0.097*
O2	0.4977 (7)	0.6342 (4)	0.53256 (19)	0.130 (2)
H2WA	0.5903	0.6036	0.5111	0.156*
H2WB	0.4962	0.7385	0.5297	0.156*
Br1	0.10716 (6)	0.79001 (4)	0.292950 (14)	0.05101 (13)
Br2	0.87524 (7)	0.52517 (5)	0.477339 (16)	0.06024 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.092 (4)	0.044 (2)	0.039 (2)	−0.002 (2)	−0.008 (2)	0.0094 (19)
C2	0.043 (2)	0.034 (2)	0.037 (2)	−0.0037 (17)	0.0040 (16)	0.0050 (17)
C3	0.051 (2)	0.033 (2)	0.057 (3)	0.0013 (18)	0.003 (2)	0.006 (2)
C4	0.050 (3)	0.043 (2)	0.065 (3)	−0.008 (2)	−0.002 (2)	−0.016 (2)
C5	0.051 (3)	0.068 (3)	0.050 (3)	−0.005 (2)	−0.007 (2)	−0.014 (2)
C6	0.051 (3)	0.060 (3)	0.034 (2)	0.002 (2)	−0.0045 (18)	0.008 (2)
C7	0.055 (3)	0.028 (2)	0.049 (2)	0.0055 (18)	0.012 (2)	0.0043 (18)
C8	0.054 (2)	0.028 (2)	0.039 (2)	0.0021 (18)	0.0041 (18)	−0.0012 (18)
C9	0.049 (2)	0.034 (2)	0.037 (2)	0.0022 (18)	0.0049 (18)	−0.0003 (18)
C10	0.050 (2)	0.033 (2)	0.047 (2)	0.0024 (18)	−0.0026 (19)	−0.0017 (18)
C11	0.057 (3)	0.051 (3)	0.041 (2)	−0.004 (2)	−0.006 (2)	−0.006 (2)
C12	0.066 (3)	0.037 (3)	0.067 (3)	−0.011 (2)	0.006 (2)	−0.006 (2)
C13	0.055 (3)	0.027 (2)	0.094 (4)	−0.001 (2)	0.022 (3)	0.003 (3)
C14	0.054 (3)	0.055 (3)	0.060 (3)	0.008 (2)	0.018 (2)	0.022 (2)
C15	0.036 (2)	0.048 (2)	0.041 (2)	−0.0006 (18)	0.0075 (17)	0.0087 (19)
C16	0.067 (3)	0.086 (3)	0.040 (2)	−0.006 (3)	−0.002 (2)	0.006 (2)
N1	0.0419 (18)	0.0311 (17)	0.0361 (17)	−0.0011 (13)	0.0052 (14)	0.0039 (14)
N2	0.0415 (17)	0.0353 (17)	0.0375 (17)	−0.0034 (14)	0.0021 (14)	0.0011 (15)
O1	0.088 (2)	0.063 (2)	0.092 (3)	−0.0154 (19)	0.028 (2)	−0.016 (2)
O2	0.147 (4)	0.048 (2)	0.196 (5)	−0.004 (2)	0.107 (4)	−0.001 (3)
Br1	0.0583 (2)	0.0496 (2)	0.0451 (2)	−0.0059 (2)	−0.0037 (2)	0.00105 (19)
Br2	0.0632 (3)	0.0460 (2)	0.0715 (3)	−0.0087 (2)	0.0086 (3)	0.0003 (2)

Geometric parameters (Å, °)

C1—C2	1.510 (5)	C9—H9B	0.9700
C1—H1A	0.9600	C10—N2	1.485 (5)
C1—H1B	0.9600	C10—H10A	0.9700
C1—H1C	0.9600	C10—H10B	0.9700
C2—N1	1.364 (5)	C11—C12	1.360 (6)
C2—C3	1.390 (6)	C11—N2	1.366 (5)
C3—C4	1.368 (6)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.368 (7)
C4—C5	1.387 (6)	C12—H12	0.9300
C4—H4	0.9300	C13—C14	1.378 (7)
C5—C6	1.353 (6)	C13—H13	0.9300
C5—H5	0.9300	C14—C15	1.407 (6)
C6—N1	1.374 (5)	C14—H14	0.9300
C6—H6	0.9300	C15—N2	1.368 (5)

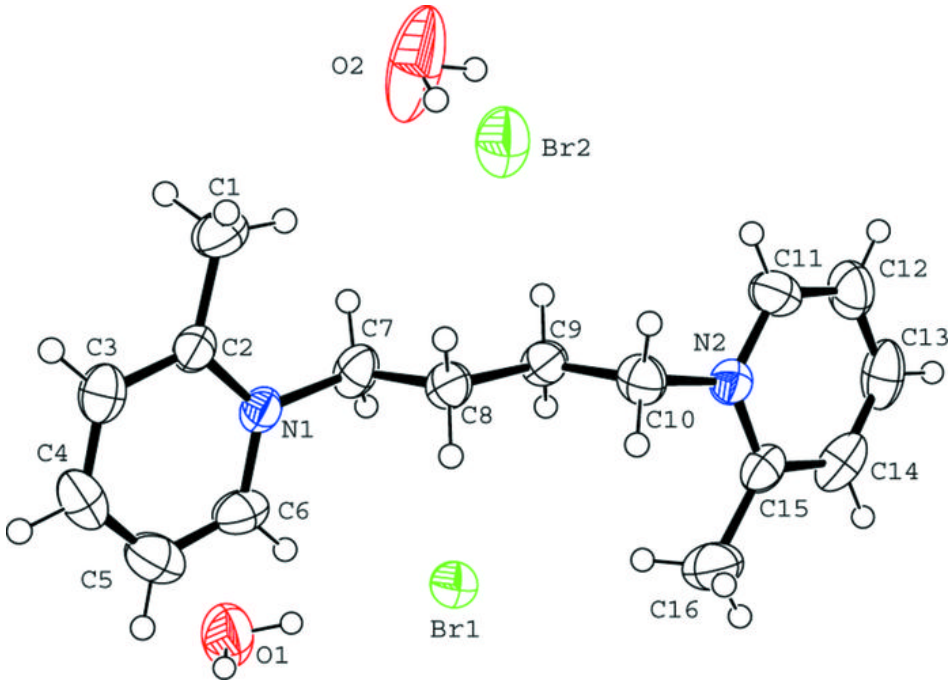
C7—N1	1.496 (5)	C15—C16	1.513 (6)
C7—C8	1.526 (5)	C16—H16A	0.9600
C7—H7A	0.9700	C16—H16B	0.9600
C7—H7B	0.9700	C16—H16C	0.9600
C8—C9	1.528 (5)	O1—H1WA	0.9515
C8—H8A	0.9700	O1—H1WB	0.9117
C8—H8B	0.9700	O2—H2WA	0.9619
C9—C10	1.541 (6)	O2—H2WB	0.9857
C9—H9A	0.9700		
C2—C1—H1A	109.5	C10—C9—H9B	109.7
C2—C1—H1B	109.5	H9A—C9—H9B	108.2
H1A—C1—H1B	109.5	N2—C10—C9	111.2 (3)
C2—C1—H1C	109.5	N2—C10—H10A	109.4
H1A—C1—H1C	109.5	C9—C10—H10A	109.4
H1B—C1—H1C	109.5	N2—C10—H10B	109.4
N1—C2—C3	118.5 (3)	C9—C10—H10B	109.4
N1—C2—C1	119.0 (3)	H10A—C10—H10B	108.0
C3—C2—C1	122.5 (4)	C12—C11—N2	122.1 (4)
C4—C3—C2	121.2 (4)	C12—C11—H11	119.0
C4—C3—H3	119.4	N2—C11—H11	119.0
C2—C3—H3	119.4	C11—C12—C13	118.6 (4)
C3—C4—C5	119.3 (4)	C11—C12—H12	120.7
C3—C4—H4	120.3	C13—C12—H12	120.7
C5—C4—H4	120.3	C12—C13—C14	120.5 (4)
C6—C5—C4	119.3 (4)	C12—C13—H13	119.7
C6—C5—H5	120.4	C14—C13—H13	119.7
C4—C5—H5	120.4	C13—C14—C15	120.5 (4)
C5—C6—N1	121.5 (4)	C13—C14—H14	119.7
C5—C6—H6	119.2	C15—C14—H14	119.7
N1—C6—H6	119.2	N2—C15—C14	117.6 (4)
N1—C7—C8	110.7 (3)	N2—C15—C16	119.5 (4)
N1—C7—H7A	109.5	C14—C15—C16	122.9 (4)
C8—C7—H7A	109.5	C15—C16—H16A	109.5
N1—C7—H7B	109.5	C15—C16—H16B	109.5
C8—C7—H7B	109.5	H16A—C16—H16B	109.5
H7A—C7—H7B	108.1	C15—C16—H16C	109.5
C7—C8—C9	111.1 (3)	H16A—C16—H16C	109.5
C7—C8—H8A	109.4	H16B—C16—H16C	109.5
C9—C8—H8A	109.4	C2—N1—C6	120.1 (3)
C7—C8—H8B	109.4	C2—N1—C7	122.8 (3)
C9—C8—H8B	109.4	C6—N1—C7	117.0 (3)
H8A—C8—H8B	108.0	C11—N2—C15	120.7 (3)
C8—C9—C10	109.8 (3)	C11—N2—C10	118.0 (3)
C8—C9—H9A	109.7	C15—N2—C10	121.2 (3)
C10—C9—H9A	109.7	H1WA—O1—H1WB	86.2
C8—C9—H9B	109.7	H2WA—O2—H2WB	104.7

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1WA···Br1	0.95	2.43	3.384 (3)	176
O1—H1WB···Br1^{i}	0.91	2.64	3.406 (4)	143
O2—H2WA···Br2	0.96	2.42	3.358 (4)	164
O2—H2WB···Br2^{ii}	0.99	2.41	3.344 (4)	159

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) *x*−1/2, −*y*+3/2, −*z*+1.

Fig. 1


O2
Br2
C1
C11
C12
C3
C2
C7
C9
N2
C13
C8
C10
N1
C4
C14
C6
C15
C5
Br1
C16
O1