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Bis[4-(dimethylamino)pyridinium] tetrabromidocadmate(II) monohydrate

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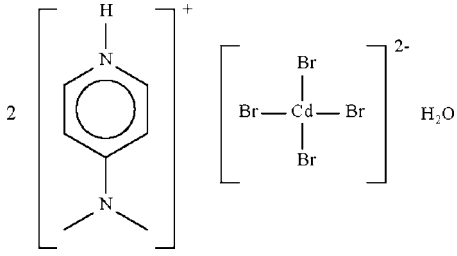
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.011 Å; R factor = 0.037; wR factor = 0.131; data-to-parameter ratio = 16.8.

The Cd atom in the hydrated title salt, $(C_7H_{11}N_2)_2[CdBr_4]\cdot H_2O$, exists in an approximately tetrahedral coordination geometry, with Br—Cd—Br angles in the range 105.52 (3)–111.50 (3)°. The cation, anion and water molecule interact by O—H···Br, N—H···Br and N—H···O hydrogen bonds, forming a linear chain structure running along the *a* axis.

Related literature

For other tetrahedral ammonium tetrabromidocadmates, see: Altermatt *et al.* (1979); Battaglia *et al.* (1991); Casals *et al.* (1987); Geselle & Fuess (1994); Ishihara *et al.* (1998); Krishnan *et al.* (1991); Sato *et al.* (1986); Waskowska (1994).



Experimental

Crystal data

$(C_7H_{11}N_2)_2[CdBr_4]\cdot H_2O$
M_r = 696.41
Triclinic, $P\overline{1}$
a = 7.8918 (2) Å
b = 8.1197 (2) Å
c = 17.2719 (4) Å
α = 95.481 (1)°
β = 99.747 (1)°
γ = 96.489 (1)°
V = 1076.35 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 8.45 mm^{-1}
T = 100 K
0.25 × 0.25 × 0.25 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.258, T_{max} = 0.431
(expected range = 0.072–0.121)
6014 measured reflections
3711 independent reflections
3255 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.131
S = 1.07
3711 reflections
221 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.53 e Å^{-3}
$\Delta\rho_{min}$ = −1.65 e Å^{-3}

Table 1
Selected bond angles (°).

Br1—Cd1—Br2	109.23 (3)	Br2—Cd1—Br3	105.52 (3)
Br1—Cd1—Br3	109.83 (3)	Br2—Cd1—Br4	110.72 (3)
Br4—Cd1—Br1	111.50 (3)	Br3—Cd1—Br4	109.87 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H11···Br1	0.84	2.83	3.351 (7)	122
O1—H12···Br3^{i}	0.84	2.67	3.511 (7)	172
N1—H1···Br2	0.88	2.56	3.373 (7)	154
N3—H3···O1	0.88	2.36	3.011 (10)	131

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2619).

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supporting information

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Bis[4-(dimethylamino)pyridinium] tetrabromidocadmate(II) monohydrate

Kong Mun Lo and Seik Weng Ng

S1. Experimental

Cadmium chloride (0.5 g, 2 mmol) dissolved in water (10 ml) and 4-dimethylaminopyridine hydrobromide perbromide (0.8 g, 2 mmol) dissolved in ethanol (80 ml) were mixed and the mixture heated for 1 h. Slow evaporation of the filtrate gave colorless crystals.

S2. Refinement

Carbon- and nitrogen-bound H atoms were placed in calculated positions (C—H 0.95 to 0.98 Å, N—H 0.88 Å) and were included in the refinement in the riding model approximation, with *U*(H) fixed at 1.2–1.5*U*(C). The water H atoms were placed in chemically sensible positions on the basis of hydrogen-bonding interactions, but were not refined.

The final difference Fourier map had a large peak at 1.3 Å from H5 and a deep hole at 0.7 Å from Br1.

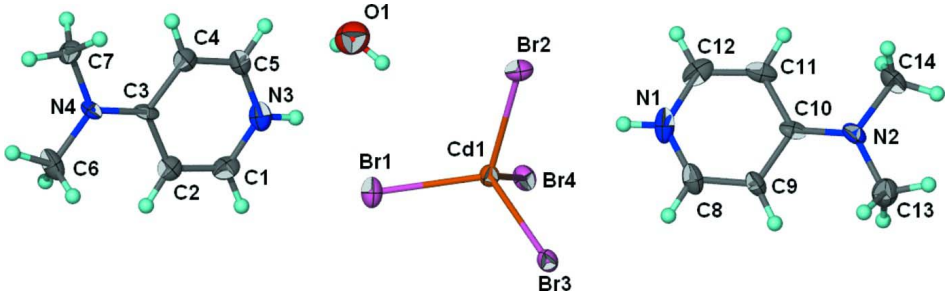

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $(C_7H_{11}N_2)_2(CdBr_4).H_2O$ at the 70% probability level. H atoms are drawn as spheres of arbitrary radius.

Bis[4-(dimethylamino)pyridinium] tetrabromidocadmate(II) monohydrate

Crystal data

$(C_7H_{11}N_2)_2[CdBr_4]\cdot H_2O$
$M_r = 696.41$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.8918$ (2) Å
$b = 8.1197$ (2) Å
$c = 17.2719$ (4) Å
$\alpha = 95.481$ (1)°
$\beta = 99.747$ (1)°
$\gamma = 96.489$ (1)°
$V = 1076.35$ (5) Å^3
$Z = 2$
$F(000) = 664$
$D_x = 2.149$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3763 reflections
$\theta = 2.4$–$28.2°$
$\mu = 8.45$ mm^{-1}
$T = 100$ K
Irregular block, colourless
0.25 × 0.25 × 0.25 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.258, T_{max} = 0.431
6014 measured reflections
3711 independent reflections
3255 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°, θ_{min} = 2.4°
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.131
S = 1.07
3711 reflections
221 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 6.748P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.53 e Å^{-3}
$\Delta\rho_{min}$ = −1.65 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.56389 (6)	0.31451 (6)	0.25658 (3)	0.01616 (17)
Br1	0.43625 (10)	0.44170 (10)	0.37244 (5)	0.0256 (2)
Br2	0.53082 (10)	0.50457 (9)	0.14316 (5)	0.0237 (2)
Br3	0.89498 (9)	0.31530 (10)	0.30001 (4)	0.0205 (2)
Br4	0.41821 (9)	0.01732 (9)	0.20678 (4)	0.0209 (2)
O1	0.1769 (9)	0.6316 (8)	0.2453 (4)	0.0438 (17)
H11	0.2666	0.5854	0.2431	0.066*
H12	0.1005	0.5627	0.2575	0.066*
N1	0.8619 (9)	0.3422 (9)	0.0770 (4)	0.0280 (16)
H1	0.7974	0.3812	0.1095	0.034*
N2	1.1657 (8)	0.1572 (8)	−0.0724 (4)	0.0187 (13)
N3	0.2927 (8)	0.8098 (8)	0.4100 (4)	0.0233 (14)
H3	0.3098	0.7253	0.3777	0.028*
N4	0.1981 (8)	1.1958 (7)	0.5626 (3)	0.0150 (12)
C1	0.3353 (10)	0.8102 (9)	0.4877 (5)	0.0210 (16)
H1A	0.3895	0.7197	0.5070	0.025*
C2	0.3061 (9)	0.9320 (9)	0.5412 (5)	0.0199 (16)
H2	0.3352	0.9246	0.5963	0.024*
C3	0.2300 (9)	1.0730 (9)	0.5130 (4)	0.0143 (14)
C4	0.1916 (9)	1.0680 (9)	0.4308 (5)	0.0179 (15)
H4	0.1414	1.1579	0.4089	0.022*
C5	0.2228 (9)	0.9411 (9)	0.3813 (4)	0.0197 (15)
H5	0.1955	0.9438	0.3258	0.024*
C6	0.2363 (11)	1.1943 (10)	0.6469 (5)	0.0258 (18)
H6A	0.1801	1.0901	0.6608	0.039*

H6B	0.1928	1.2893	0.6729	0.039*
H6C	0.3620	1.2025	0.6645	0.039*
C7	0.1264 (10)	1.3411 (9)	0.5317 (5)	0.0195 (15)
H7A	0.2057	1.3956	0.5012	0.029*
H7B	0.1122	1.4203	0.5759	0.029*
H7C	0.0134	1.3043	0.4975	0.029*
C8	1.0058 (10)	0.2831 (10)	0.1052 (5)	0.0249 (17)
H8	1.0381	0.2845	0.1608	0.030*
C9	1.1099 (10)	0.2201 (10)	0.0575 (4)	0.0199 (15)
H9	1.2124	0.1779	0.0800	0.024*
C10	1.0651 (9)	0.2175 (8)	−0.0254 (4)	0.0163 (15)
C11	0.9084 (10)	0.2811 (9)	−0.0549 (5)	0.0221 (17)
H11A	0.8709	0.2804	−0.1102	0.026*
C12	0.8130 (10)	0.3430 (9)	−0.0026 (5)	0.0245 (17)
H12A	0.7100	0.3877	−0.0220	0.029*
C13	1.3311 (10)	0.1038 (11)	−0.0401 (5)	0.0274 (18)
H13A	1.3938	0.1870	0.0031	0.041*
H13B	1.4006	0.0925	−0.0818	0.041*
H13C	1.3100	−0.0040	−0.0199	0.041*
C14	1.1133 (11)	0.1373 (10)	−0.1580 (4)	0.0242 (17)
H14A	1.1968	0.0788	−0.1822	0.036*
H14B	1.1100	0.2474	−0.1766	0.036*
H14C	0.9979	0.0723	−0.1728	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0151 (3)	0.0170 (3)	0.0153 (3)	0.0015 (2)	0.0019 (2)	−0.0014 (2)
Br1	0.0261 (4)	0.0246 (4)	0.0270 (5)	0.0027 (3)	0.0108 (3)	−0.0031 (3)
Br2	0.0229 (4)	0.0236 (4)	0.0244 (4)	0.0049 (3)	0.0000 (3)	0.0067 (3)
Br3	0.0158 (4)	0.0304 (4)	0.0148 (4)	0.0038 (3)	0.0021 (3)	0.0002 (3)
Br4	0.0239 (4)	0.0184 (4)	0.0187 (4)	−0.0008 (3)	0.0038 (3)	−0.0013 (3)
O1	0.049 (4)	0.037 (4)	0.044 (4)	0.010 (3)	0.004 (3)	0.000 (3)
N1	0.025 (4)	0.027 (4)	0.034 (4)	−0.001 (3)	0.019 (3)	−0.005 (3)
N2	0.020 (3)	0.026 (3)	0.011 (3)	0.002 (3)	0.004 (2)	0.007 (3)
N3	0.024 (3)	0.024 (3)	0.023 (4)	0.000 (3)	0.010 (3)	0.000 (3)
N4	0.019 (3)	0.017 (3)	0.009 (3)	0.003 (2)	0.001 (2)	0.004 (2)
C1	0.022 (4)	0.016 (4)	0.028 (4)	0.004 (3)	0.007 (3)	0.013 (3)
C2	0.016 (3)	0.019 (4)	0.024 (4)	−0.002 (3)	0.003 (3)	0.003 (3)
C3	0.012 (3)	0.017 (3)	0.013 (4)	−0.002 (3)	−0.001 (3)	0.012 (3)
C4	0.011 (3)	0.020 (4)	0.022 (4)	−0.001 (3)	0.002 (3)	0.003 (3)
C5	0.021 (4)	0.023 (4)	0.015 (4)	0.000 (3)	0.007 (3)	0.001 (3)
C6	0.031 (4)	0.027 (4)	0.017 (4)	−0.008 (3)	0.010 (3)	−0.006 (3)
C7	0.023 (4)	0.013 (3)	0.023 (4)	0.006 (3)	0.003 (3)	−0.001 (3)
C8	0.026 (4)	0.026 (4)	0.022 (4)	−0.002 (3)	0.009 (3)	−0.007 (3)
C9	0.022 (4)	0.025 (4)	0.012 (4)	0.000 (3)	0.005 (3)	−0.001 (3)
C10	0.019 (4)	0.011 (3)	0.016 (4)	−0.006 (3)	−0.004 (3)	0.008 (3)
C11	0.020 (4)	0.021 (4)	0.020 (4)	−0.010 (3)	−0.005 (3)	0.009 (3)

C12	0.020 (4)	0.015 (4)	0.037 (5)	0.000 (3)	0.003 (3)	0.005 (3)
C13	0.022 (4)	0.028 (4)	0.031 (5)	0.002 (3)	0.003 (3)	0.001 (4)
C14	0.031 (4)	0.028 (4)	0.012 (4)	0.001 (3)	0.003 (3)	0.001 (3)

Geometric parameters (Å, º)

Cd1—Br1	2.5706 (9)	C4—C5	1.347 (11)
Cd1—Br2	2.6044 (9)	C4—H4	0.9500
Cd1—Br3	2.5926 (8)	C5—H5	0.9500
Cd1—Br4	2.5546 (8)	C6—H6A	0.9800
O1—H11	0.8418	C6—H6B	0.9800
O1—H12	0.8427	C6—H6C	0.9800
N1—C8	1.318 (11)	C7—H7A	0.9800
N1—C12	1.364 (11)	C7—H7B	0.9800
N1—H1	0.8800	C7—H7C	0.9800
N2—C10	1.326 (10)	C8—C9	1.364 (11)
N2—C14	1.455 (10)	C8—H8	0.9500
N2—C13	1.460 (10)	C9—C10	1.414 (10)
N3—C1	1.327 (10)	C9—H9	0.9500
N3—C5	1.352 (10)	C10—C11	1.427 (11)
N3—H3	0.8800	C11—C12	1.367 (12)
N4—C3	1.325 (10)	C11—H11A	0.9500
N4—C6	1.438 (10)	C12—H12A	0.9500
N4—C7	1.472 (9)	C13—H13A	0.9800
C1—C2	1.355 (11)	C13—H13B	0.9800
C1—H1A	0.9500	C13—H13C	0.9800
C2—C3	1.439 (10)	C14—H14A	0.9800
C2—H2	0.9500	C14—H14B	0.9800
C3—C4	1.396 (10)	C14—H14C	0.9800
Br1—Cd1—Br2	109.23 (3)	N4—C6—H6C	109.5
Br1—Cd1—Br3	109.83 (3)	H6A—C6—H6C	109.5
Br4—Cd1—Br1	111.50 (3)	H6B—C6—H6C	109.5
Br2—Cd1—Br3	105.52 (3)	N4—C7—H7A	109.5
Br2—Cd1—Br4	110.72 (3)	N4—C7—H7B	109.5
Br3—Cd1—Br4	109.87 (3)	H7A—C7—H7B	109.5
H11—O1—H12	108.9	N4—C7—H7C	109.5
C8—N1—C12	120.1 (7)	H7A—C7—H7C	109.5
C8—N1—H1	120.0	H7B—C7—H7C	109.5
C12—N1—H1	120.0	N1—C8—C9	122.4 (8)
C10—N2—C14	121.3 (6)	N1—C8—H8	118.8
C10—N2—C13	121.3 (6)	C9—C8—H8	118.8
C14—N2—C13	117.4 (6)	C8—C9—C10	119.8 (7)
C1—N3—C5	119.1 (7)	C8—C9—H9	120.1
C1—N3—H3	120.5	C10—C9—H9	120.1
C5—N3—H3	120.5	N2—C10—C9	120.4 (7)
C3—N4—C6	121.8 (6)	N2—C10—C11	122.7 (7)
C3—N4—C7	120.0 (6)	C9—C10—C11	116.9 (7)

C6—N4—C7	118.2 (6)	C12—C11—C10	119.1 (7)
N3—C1—C2	123.8 (7)	C12—C11—H11A	120.4
N3—C1—H1A	118.1	C10—C11—H11A	120.4
C2—C1—H1A	118.1	N1—C12—C11	121.6 (7)
C1—C2—C3	118.8 (7)	N1—C12—H12A	119.2
C1—C2—H2	120.6	C11—C12—H12A	119.2
C3—C2—H2	120.6	N2—C13—H13A	109.5
N4—C3—C4	123.6 (6)	N2—C13—H13B	109.5
N4—C3—C2	121.4 (6)	H13A—C13—H13B	109.5
C4—C3—C2	114.9 (7)	N2—C13—H13C	109.5
C5—C4—C3	122.9 (7)	H13A—C13—H13C	109.5
C5—C4—H4	118.6	H13B—C13—H13C	109.5
C3—C4—H4	118.6	N2—C14—H14A	109.5
C4—C5—N3	120.5 (7)	N2—C14—H14B	109.5
C4—C5—H5	119.8	H14A—C14—H14B	109.5
N3—C5—H5	119.8	N2—C14—H14C	109.5
N4—C6—H6A	109.5	H14A—C14—H14C	109.5
N4—C6—H6B	109.5	H14B—C14—H14C	109.5
H6A—C6—H6B	109.5		

C5—N3—C1—C2	3.0 (11)	C12—N1—C8—C9	−0.5 (12)
N3—C1—C2—C3	−2.3 (11)	N1—C8—C9—C10	0.4 (12)
C6—N4—C3—C4	178.6 (7)	C14—N2—C10—C9	173.5 (7)
C7—N4—C3—C4	−3.1 (10)	C13—N2—C10—C9	−4.7 (10)
C6—N4—C3—C2	−0.5 (10)	C14—N2—C10—C11	−6.1 (10)
C7—N4—C3—C2	177.7 (6)	C13—N2—C10—C11	175.7 (7)
C1—C2—C3—N4	179.9 (7)	C8—C9—C10—N2	179.7 (7)
C1—C2—C3—C4	0.6 (10)	C8—C9—C10—C11	−0.7 (10)
N4—C3—C4—C5	−179.1 (7)	N2—C10—C11—C12	−179.2 (7)
C2—C3—C4—C5	0.1 (10)	C9—C10—C11—C12	1.2 (10)
C3—C4—C5—N3	0.6 (11)	C8—N1—C12—C11	1.1 (11)
C1—N3—C5—C4	−2.1 (11)	C10—C11—C12—N1	−1.4 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···Br1	0.84	2.83	3.351 (7)	122
O1—H12···Br3^{i}	0.84	2.67	3.511 (7)	172
N1—H1···Br2	0.88	2.56	3.373 (7)	154
N3—H3···O1	0.88	2.36	3.011 (10)	131

Symmetry code: (i) *x*−1, *y*, *z*.