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1,4-Diazoniabicyclo[2.2.2]octane tetrabromidocadmate(II) monohydrate

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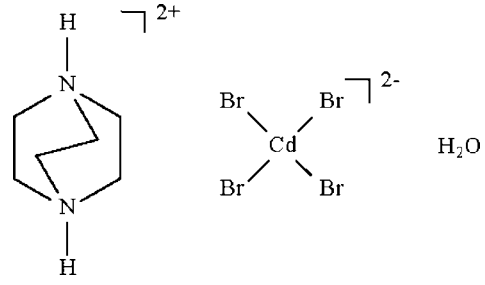
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.023 Å; R factor = 0.057; wR factor = 0.187; data-to-parameter ratio = 19.1.

The metal atom in the anion of the title salt, $(C_6H_{14}N_2)[CdBr_4]\cdot H_2O$, shows a slightly distorted tetrahedral coordination. The water molecule is involved in three hydrogen bonds, *viz*. one N—H···O and two O—H···Br, and an N—H···Br interaction consolidates the three-dimensional network.

Related literature

For other ammonium tetrabromidocadmates, see: Al-Far & Ali (2008); Battaglia *et al.* (1991); Chen *et al.* (2006); Geselle & Fuess (1994); Hatano *et al.* (2008); Ishihara *et al.* (2002, 2006); Ravikumar *et al.* (1995); Waskowska (1994); Zhang & Fang (2005).



Experimental

Crystal data

$(C_6H_{14}N_2)[CdBr_4]\cdot H_2O$
M_r = 564.25
Orthorhombic, $P2_12_12_1$
a = 8.6323 (1) Å
b = 11.8736 (2) Å
c = 13.5619 (2) Å
V = 1390.05 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 13.04 mm^{-1}
T = 296 K
0.30 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.111, T_{max} = 0.562
(expected range = 0.103–0.521)
10779 measured reflections
2451 independent reflections
2267 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.187
S = 1.36
2451 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.21 e Å^{-3}
$\Delta\rho_{min}$ = −2.09 e Å^{-3}
Absolute structure: Flack (1983), 1021 Friedel pairs
Flack parameter: 0.14 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1w—H11···Br4^{i}	0.84	2.72	3.15 (3)	113
O1w—H12···Br1^{ii}	0.84	2.83	3.65 (3)	167
N1—H1···Br1	0.86	2.92	3.568 (11)	134
N2—H2···O1w	0.86	2.04	2.80 (2)	146

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2504).

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supporting information

Acta Cryst. (2009). E**65**, m973 [doi:10.1107/S160053680902813X]

1,4-Diazoniabicyclo[2.2.2]octane tetrabromidocadmate(II) monohydrate

Kong Mun Lo and Seik Weng Ng

S1. Experimental

Triethylenediammonium dibromide was prepared from the reaction of triethylenediamine (1 g, 1.68 mmol) with bromine (1:2) in the presence of excess hydrobromic acid. To this was added cadmium chloride hemipentahydrate (0.38 g, 1.68 mmol) in ethanol (50 ml). The mixture was heated for an hour. The filtered solution when allow to evaporate slowly yielded colorless crystals.

S2. Refinement

C- and N-bound H atoms were placed at calculated positions (C–H 0.97Å and N–H 0.86 Å) and were treated as riding on their parent atoms with U(H) set to 1.2U_{eq}(C, N). The water-bound 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 peak 0.2 Å from Cd1 and a hole 0.4 Å from Br4.

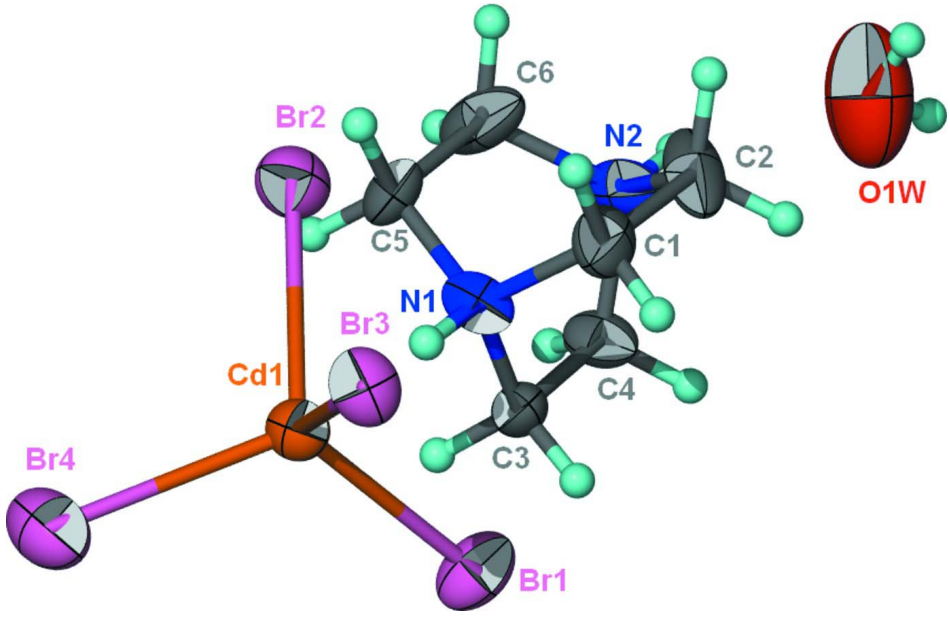

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[C_6H_{14}N_2][CdBr_4]\cdot H_2O$ at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

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Crystal data

$(C_6H_{14}N_2)[CdBr_4]\cdot H_2O$
$M_r = 564.25$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.6323$ (1) Å
$b = 11.8736$ (2) Å
$c = 13.5619$ (2) Å
$V = 1390.05$ (4) Å^3
$Z = 4$
$F(000) = 1048$
$D_x = 2.696$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6547 reflections
$\theta = 2.8$–$28.2°$
$\mu = 13.04$ mm^{-1}
$T = 296$ K
Block, colorless
0.30 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.111$, $T_{max} = 0.562$
10779 measured reflections
2451 independent reflections
2267 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.187$
$S = 1.36$
2451 reflections
128 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
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 2.21$ e Å^{-3}
$\Delta\rho_{min} = -2.09$ e Å^{-3}
Absolute structure: Flack (1983), 1021 Friedel pairs
Absolute structure parameter: 0.14 (3)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cd1	0.25482 (11)	0.47127 (8)	1.00019 (6)	0.0410 (3)
Br1	0.3099 (2)	0.28309 (13)	0.91553 (12)	0.0596 (5)
Br2	0.4870 (2)	0.59802 (15)	0.95680 (13)	0.0579 (5)
Br3	0.2467 (2)	0.46741 (14)	1.19150 (10)	0.0552 (4)
Br4	0.0032 (2)	0.56945 (19)	0.95508 (17)	0.0741 (6)
O1W	1.151 (3)	0.265 (2)	0.6666 (18)	0.144 (9)
H11	1.1965	0.2150	0.6336	0.216*
H12	1.2016	0.2746	0.7189	0.216*
N1	0.6148 (13)	0.4081 (10)	0.7757 (7)	0.040 (3)
H1	0.5272	0.4215	0.8034	0.048*
N2	0.8660 (14)	0.3708 (12)	0.6978 (13)	0.063 (4)
H2	0.9523	0.3577	0.6682	0.076*

C1	0.725 (2)	0.3684 (14)	0.8530 (12)	0.056 (4)
H1A	0.6860	0.3008	0.8846	0.067*
H1B	0.7396	0.4260	0.9029	0.067*
C2	0.882 (2)	0.3435 (18)	0.7975 (16)	0.086 (8)
H2A	0.9647	0.3877	0.8264	0.104*
H2B	0.9083	0.2645	0.8042	0.104*
C3	0.5912 (16)	0.3214 (11)	0.7016 (12)	0.045 (3)
H3A	0.5121	0.3449	0.6551	0.054*
H3B	0.5577	0.2519	0.7326	0.054*
C4	0.7409 (17)	0.3035 (13)	0.6494 (9)	0.047 (3)
H4A	0.7308	0.3262	0.5810	0.056*
H4B	0.7679	0.2242	0.6510	0.056*
C5	0.6697 (19)	0.5139 (11)	0.7291 (11)	0.044 (3)
H5A	0.6781	0.5729	0.7783	0.052*
H5B	0.5968	0.5382	0.6790	0.052*
C6	0.824 (2)	0.4924 (13)	0.6836 (17)	0.066 (5)
H6A	0.9016	0.5400	0.7143	0.079*
H6B	0.8206	0.5100	0.6138	0.079*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0398 (5)	0.0445 (6)	0.0387 (5)	0.0007 (4)	0.0034 (5)	−0.0059 (4)
Br1	0.0813 (11)	0.0441 (8)	0.0535 (8)	0.0002 (8)	0.0191 (7)	−0.0081 (7)
Br2	0.0570 (9)	0.0650 (10)	0.0515 (8)	−0.0032 (8)	−0.0029 (7)	0.0044 (7)
Br3	0.0546 (8)	0.0682 (10)	0.0428 (7)	0.0061 (9)	0.0040 (7)	0.0035 (6)
Br4	0.0606 (11)	0.0862 (13)	0.0756 (12)	0.0076 (10)	−0.0051 (9)	−0.0244 (10)
O1W	0.150 (18)	0.153 (19)	0.129 (16)	0.073 (17)	0.022 (14)	0.012 (15)
N1	0.037 (5)	0.063 (7)	0.020 (5)	−0.003 (5)	0.003 (4)	−0.009 (5)
N2	0.029 (6)	0.057 (8)	0.104 (12)	−0.002 (5)	0.019 (7)	−0.038 (8)
C1	0.067 (11)	0.051 (9)	0.051 (8)	0.005 (8)	−0.023 (8)	0.003 (7)
C2	0.089 (14)	0.071 (12)	0.099 (15)	0.039 (11)	−0.064 (13)	−0.051 (11)
C3	0.036 (6)	0.031 (7)	0.069 (9)	0.000 (5)	−0.002 (7)	0.000 (6)
C4	0.045 (7)	0.056 (8)	0.040 (6)	−0.008 (7)	−0.005 (7)	−0.014 (6)
C5	0.059 (9)	0.026 (6)	0.045 (7)	0.002 (6)	0.007 (7)	0.000 (6)
C6	0.066 (11)	0.040 (9)	0.091 (12)	−0.011 (7)	0.035 (10)	−0.017 (8)

Geometric parameters (Å, º)

Cd1—Br4	2.540 (2)	C1—H1A	0.9700
Cd1—Br1	2.557 (2)	C1—H1B	0.9700
Cd1—Br2	2.574 (2)	C2—H2A	0.9700
Cd1—Br3	2.596 (2)	C2—H2B	0.9700
O1W—H11	0.84	C3—C4	1.49 (2)
O1W—H12	0.84	C3—H3A	0.9700
N1—C3	1.453 (18)	C3—H3B	0.9700
N1—C5	1.484 (18)	C4—H4A	0.9700
N1—C1	1.491 (18)	C4—H4B	0.9700

N1—H1	0.8600	C5—C6	1.49 (2)
N2—C2	1.40 (3)	C5—H5A	0.9700
N2—C4	1.495 (18)	C5—H5B	0.9700
N2—C6	1.50 (2)	C6—H6A	0.9700
N2—H2	0.8600	C6—H6B	0.9700
C1—C2	1.58 (3)		

Br4—Cd1—Br1	116.87 (7)	N2—C2—H2B	109.8
Br4—Cd1—Br2	110.01 (7)	C1—C2—H2B	109.8
Br1—Cd1—Br2	105.29 (7)	H2A—C2—H2B	108.2
Br4—Cd1—Br3	103.05 (7)	N1—C3—C4	107.9 (11)
Br1—Cd1—Br3	116.00 (7)	N1—C3—H3A	110.1
Br2—Cd1—Br3	105.05 (6)	C4—C3—H3A	110.1
H11—O1W—H12	107.7	N1—C3—H3B	110.1
C3—N1—C5	110.5 (10)	C4—C3—H3B	110.1
C3—N1—C1	110.6 (12)	H3A—C3—H3B	108.4
C5—N1—C1	111.3 (12)	C3—C4—N2	110.0 (11)
C3—N1—H1	108.1	C3—C4—H4A	109.7
C5—N1—H1	108.1	N2—C4—H4A	109.7
C1—N1—H1	108.1	C3—C4—H4B	109.7
C2—N2—C4	111.8 (15)	N2—C4—H4B	109.7
C2—N2—C6	111.8 (15)	H4A—C4—H4B	108.2
C4—N2—C6	106.5 (14)	N1—C5—C6	108.5 (11)
C2—N2—H2	108.9	N1—C5—H5A	110.0
C4—N2—H2	108.9	C6—C5—H5A	110.0
C6—N2—H2	108.9	N1—C5—H5B	110.0
N1—C1—C2	105.7 (13)	C6—C5—H5B	110.0
N1—C1—H1A	110.6	H5A—C5—H5B	108.4
C2—C1—H1A	110.6	C5—C6—N2	109.1 (12)
N1—C1—H1B	110.6	C5—C6—H6A	109.9
C2—C1—H1B	110.6	N2—C6—H6A	109.9
H1A—C1—H1B	108.7	C5—C6—H6B	109.9
N2—C2—C1	109.5 (13)	N2—C6—H6B	109.9
N2—C2—H2A	109.8	H6A—C6—H6B	108.3
C1—C2—H2A	109.8		

C3—N1—C1—C2	60.9 (16)	C2—N2—C4—C3	57.5 (17)
C5—N1—C1—C2	−62.3 (16)	C6—N2—C4—C3	−64.8 (17)
C4—N2—C2—C1	−60.2 (17)	C3—N1—C5—C6	−63.7 (16)
C6—N2—C2—C1	59.0 (19)	C1—N1—C5—C6	59.6 (16)
N1—C1—C2—N2	2 (2)	N1—C5—C6—N2	2 (2)
C5—N1—C3—C4	58.4 (15)	C2—N2—C6—C5	−63 (2)
C1—N1—C3—C4	−65.3 (15)	C4—N2—C6—C5	59.1 (19)
N1—C3—C4—N2	6.0 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1w—H11···Br4^{i}	0.84	2.72	3.15 (3)	113
O1w—H12···Br1^{ii}	0.84	2.83	3.65 (3)	167
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