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1,1′-(Propane-1,3-diyl)dipyridinium dibromidodichloridocadmate(II)

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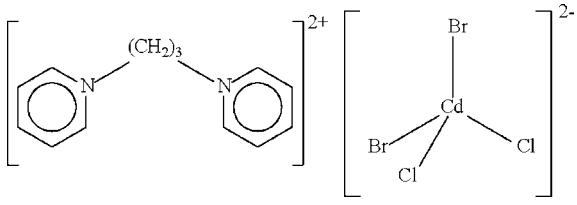
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.012 Å; disorder in main residue; R factor = 0.037; wR factor = 0.121; data-to-parameter ratio = 16.0.

The Cd atom in the title compound, $(C_{13}H_{16}N_2)[CdBr_2Cl_2]$, is coordinated by four halogen atoms in a tetrahedral geometry. The four halogen atoms are each disordered between Br and Cl, with the Br:Cl ratios being approximately 0.5:0.5, 0.6:0.4, 0.4:0.6 and 0.8:0.2.

Related literature

For related literature, see: Kao & Chen (2004); Liu *et al.* (2007).



Experimental

Crystal data

$(C_{13}H_{16}N_2)[CdBr_2Cl_2]$
M_r = 554.51
Monoclinic, $P2_1/c$
a = 8.1159 (6) Å
b = 15.058 (1) Å
c = 15.103 (1) Å
β = 95.023 (1)°
V = 1838.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.32 mm^{-1}
T = 295 (2) K
0.15 × 0.10 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.409, T_{max} = 0.632
10838 measured reflections
3220 independent reflections
2287 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.121
S = 1.08
3220 reflections
201 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.63 e Å^{-3}

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2142).

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supplementary materials

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1,1'-(Propane-1,3-diyl)dipyridinium dibromidodichloridocadmate(II)

X.-C. Liu, J. Shi, Y.-Y. Niu, N. Xu and S. W. Ng

Comment

The preceding paper reports the structure of the tetrahedral dibromidodichloridocadmate(II), which has been isolated as the 1,2-ethanedipyridinium salt (Liu *et al.*, 2007). The structure of propyl-1,3-di(4-pyridinium) tetrabromidocadmate(II) has been reported in the non-centrosymmetric space group *Cc* (Kao & Chen, 2004). The title compound, $[C_{13}H_{16}N_2][CdBr_2Cl_2]$ is not, however, isostructural. The cadmium atom in the centrosymmetric structure is coordinated by four halogen atoms in a tetrahedral geometry; the halogen atoms are disordered (Fig. 1). The cations and anions do not have signification interactions with each other.

Experimental

The salt was synthesized from the reaction of 1,3-propanedipyridinium dibromide (0.036 g, 0.1 mmol) in methanol (5 ml) and cadmium dichloride (0.037 g, 0.2 mmol) in DMF (10 ml). The mixture was set aside for the formation of colourless crystals in 30% yield after several days.

Refinement

The halogens lie in general positions; initial attempts to refine the structure with either four chlorines or four bromines gave unacceptably high *R*-indices (and large peaks/holes). The four halogen atoms were then refined as four (Br+Cl) mixtures; one attempt allowed the mixtures to have the same displacement parameters as well as to share the same site. A second attempt had the components having only the same displacement parameters. The second led to a formulation consisting of approximately 2.25 Br and 1.75 C l atoms for the anion. A special restraint was used that fixed the total number of Br and Cl atoms as exactly 2.25 Br and 1.75 C l. In the best disorder model, the site occupancy factors for the (Br+Cl) mixtures refined to 0.486 (2):0.514 (2), 0.561 (2):0.439 (2), 0.431 (2):0.569 (2) and 0.771 (2):0.229 (2).

The compound is $[C_{13}H_{16}N_2]$ $[CdBr_{2.25}Cl_{1.75}]$ but because it has nearly equal numbers of bromine and chlorine atoms, it is regarded as $[C_{13}H_{16}N_2]$ $[CdBr_2Cl_2]$ for the purpose of naming the compound. The CH&N elemental percentages are in fair agreement with the $[C_{13}H_{16}N_2]$ $[CdBr_{2.25}Cl_{1.75}]$ formulation.

The disorder in the anion did not affect the cation. Carbon-bound H atoms were positioned geometrically (C—H 0.93 and 0.97 Å), and were included in the refinement in the riding model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

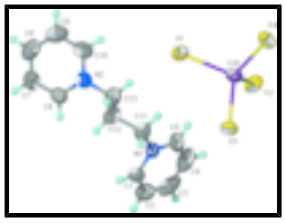

Fig. 1. The structure of $[C_{13}H_{16}N_2][CdBr_2Cl_2]$, with displacement ellipsoids drawn at the 50% probability level. The bromine and chlorine atoms are disordered; the figure depicts the anion as a $[CdX_4]^{2-}$ species. Hydrogen atoms are drawn as spheres of arbitrary radius.

Propane-1,3-diyldipyridinium dibromidodichloridocadmate(II)

Crystal data

$(C_{13}H_{16}N_2)[CdBr_2Cl_2]$	F_{000} = 1058
M_r = 554.51	D_x = 2.003 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2441 reflections
a = 8.1159 (6) Å	θ = 2.7–21.3°
b = 15.058 (1) Å	μ = 6.32 mm^{-1}
c = 15.103 (1) Å	T = 295 (2) K
β = 95.023 (1)°	Block, colourless
V = 1838.6 (2) Å^3	0.15 × 0.10 × 0.08 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	3220 independent reflections
Radiation source: fine-focus sealed tube	2287 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 298(2) K	θ_{max} = 25.0°
φ and ω scans	θ_{min} = 1.9°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −9→9
T_{min} = 0.409, T_{max} = 0.632	k = −17→17
10838 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H-atom parameters constrained
$wR(F^2)$ = 0.121	$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001

3220 reflections	$\Delta\rho_{max}$ = 0.82 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.63 e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cd1	0.25358 (6)	0.20461 (3)	0.28984 (3)	0.0586 (2)	
Br1	0.4650 (9)	0.3290 (3)	0.2721 (6)	0.0757 (10)	0.4863 (18)
Cl1	0.485 (2)	0.3078 (9)	0.2754 (14)	0.0757 (10)	0.5137 (18)
Br2	0.3988 (7)	0.0714 (3)	0.3716 (4)	0.0712 (8)	0.561 (2)
Cl2	0.416 (2)	0.0906 (10)	0.3671 (15)	0.0712 (8)	0.439 (2)
Br3	0.1618 (15)	0.1511 (12)	0.1337 (11)	0.0651 (15)	0.431 (2)
Cl3	0.135 (3)	0.145 (2)	0.139 (2)	0.0651 (15)	0.569 (2)
Br4	0.0147 (3)	0.27032 (12)	0.36647 (16)	0.0712 (5)	0.771 (2)
Cl4	−0.011 (3)	0.2488 (14)	0.3558 (16)	0.0712 (5)	0.229 (2)
N1	0.6152 (5)	0.1612 (3)	0.0464 (3)	0.0528 (12)	
C1	0.6960 (9)	0.1278 (5)	−0.0181 (5)	0.0695 (18)	
H1	0.7119	0.1625	−0.0676	0.083*	
C2	0.7553 (10)	0.0450 (6)	−0.0135 (6)	0.095 (3)	
H2	0.8148	0.0240	−0.0590	0.114*	
C3	0.7317 (11)	−0.0074 (6)	0.0538 (8)	0.098 (3)	
H3	0.7691	−0.0658	0.0551	0.118*	
C4	0.6496 (10)	0.0269 (6)	0.1227 (7)	0.096 (3)	
H4	0.6339	−0.0077	0.1723	0.115*	
C5	0.5921 (8)	0.1118 (5)	0.1169 (5)	0.073 (2)	
H5	0.5362	0.1354	0.1628	0.088*	
N2	0.9403 (6)	0.3797 (3)	0.1317 (3)	0.0568 (13)	
C6	1.0594 (8)	0.3774 (5)	0.0759 (4)	0.0649 (17)	
H6	1.0619	0.3315	0.0347	0.078*	
C7	1.1779 (9)	0.4426 (6)	0.0793 (6)	0.084 (2)	
H7	1.2612	0.4416	0.0409	0.101*	
C8	1.1712 (9)	0.5093 (6)	0.1404 (6)	0.087 (2)	
H8	1.2503	0.5541	0.1435	0.104*	
C9	1.0487 (11)	0.5103 (6)	0.1970 (5)	0.087 (2)	
H9	1.0438	0.5555	0.2386	0.105*	
C10	0.9341 (9)	0.4443 (5)	0.1915 (5)	0.0731 (19)	
H10	0.8505	0.4443	0.2299	0.088*	
C11	0.5538 (8)	0.2530 (4)	0.0405 (5)	0.0660 (18)	
H11A	0.4859	0.2640	0.0891	0.079*	
H11B	0.4847	0.2604	−0.0147	0.079*	
C12	0.6934 (8)	0.3208 (4)	0.0443 (5)	0.0657 (18)	
H12A	0.7527	0.3149	−0.0084	0.079*	
H12B	0.6470	0.3801	0.0446	0.079*	
C13	0.8126 (8)	0.3086 (5)	0.1259 (5)	0.073 (2)	
H13A	0.7524	0.3100	0.1786	0.087*	

H13B	0.8660	0.2511	0.1235	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0570 (3)	0.0650 (3)	0.0539 (3)	0.0085 (2)	0.0050 (2)	−0.0107 (2)
Br1	0.077 (2)	0.062 (3)	0.0900 (11)	−0.0072 (19)	0.0218 (13)	−0.022 (2)
Cl1	0.077 (2)	0.062 (3)	0.0900 (11)	−0.0072 (19)	0.0218 (13)	−0.022 (2)
Br2	0.0635 (15)	0.056 (2)	0.0904 (11)	0.0047 (14)	−0.0153 (10)	0.0029 (17)
Cl2	0.0635 (15)	0.056 (2)	0.0904 (11)	0.0047 (14)	−0.0153 (10)	0.0029 (17)
Br3	0.067 (4)	0.072 (3)	0.056 (2)	0.003 (3)	0.004 (2)	−0.0156 (17)
Cl3	0.067 (4)	0.072 (3)	0.056 (2)	0.003 (3)	0.004 (2)	−0.0156 (17)
Br4	0.0762 (11)	0.0748 (13)	0.0660 (9)	0.0202 (8)	0.0252 (7)	0.0008 (8)
Cl4	0.0762 (11)	0.0748 (13)	0.0660 (9)	0.0202 (8)	0.0252 (7)	0.0008 (8)
N1	0.051 (3)	0.056 (3)	0.051 (3)	−0.005 (2)	0.001 (2)	0.006 (2)
C1	0.085 (5)	0.064 (5)	0.062 (4)	−0.008 (4)	0.018 (4)	−0.010 (4)
C2	0.099 (7)	0.077 (6)	0.111 (7)	−0.005 (5)	0.025 (5)	−0.025 (5)
C3	0.083 (6)	0.056 (5)	0.150 (9)	−0.001 (4)	−0.025 (6)	−0.015 (6)
C4	0.092 (6)	0.076 (6)	0.112 (7)	−0.019 (5)	−0.028 (5)	0.043 (6)
C5	0.060 (4)	0.092 (6)	0.069 (5)	−0.013 (4)	0.012 (3)	0.009 (4)
N2	0.052 (3)	0.060 (3)	0.057 (3)	0.010 (3)	−0.001 (2)	0.005 (3)
C6	0.060 (4)	0.071 (5)	0.065 (4)	0.007 (4)	0.012 (3)	−0.006 (3)
C7	0.076 (5)	0.094 (6)	0.085 (6)	−0.001 (5)	0.020 (4)	0.009 (5)
C8	0.068 (5)	0.075 (5)	0.114 (7)	−0.008 (4)	−0.014 (5)	0.011 (5)
C9	0.090 (6)	0.075 (6)	0.091 (6)	0.017 (5)	−0.018 (5)	−0.018 (5)
C10	0.065 (4)	0.088 (5)	0.065 (4)	0.020 (4)	0.001 (3)	−0.006 (4)
C11	0.057 (4)	0.063 (4)	0.076 (5)	0.002 (3)	−0.004 (3)	0.009 (4)
C12	0.072 (4)	0.052 (4)	0.070 (4)	0.004 (3)	−0.011 (3)	0.005 (3)
C13	0.062 (4)	0.073 (5)	0.081 (5)	−0.006 (4)	−0.002 (4)	0.015 (4)

Geometric parameters (Å, °)

Cd1—Cl2	2.404 (19)	N2—C10	1.331 (8)
Cd1—Cl1	2.46 (2)	N2—C6	1.338 (8)
Cd1—Br3	2.540 (17)	N2—C13	1.488 (8)
Cd1—Br4	2.544 (2)	C6—C7	1.373 (10)
Cd1—Cl4	2.53 (2)	C6—H6	0.9300
Cd1—Cl3	2.55 (3)	C7—C8	1.368 (11)
Cd1—Br1	2.570 (8)	C7—H7	0.9300
Cd1—Br2	2.585 (5)	C8—C9	1.366 (11)
N1—C1	1.320 (8)	C8—H8	0.9300
N1—C5	1.327 (8)	C9—C10	1.358 (10)
N1—C11	1.469 (8)	C9—H9	0.9300
C1—C2	1.336 (11)	C10—H10	0.9300
C1—H1	0.9300	C11—C12	1.522 (9)
C2—C3	1.314 (12)	C11—H11A	0.9700
C2—H2	0.9300	C11—H11B	0.9700
C3—C4	1.383 (12)	C12—C13	1.511 (9)
C3—H3	0.9300	C12—H12A	0.9700

C4—C5	1.362 (11)	C12—H12B	0.9700
C4—H4	0.9300	C13—H13A	0.9700
C5—H5	0.9300	C13—H13B	0.9700
Cl2—Cd1—Cl1	95.9 (5)	C5—C4—H4	120.4
Cl2—Cd1—Br3	109.1 (7)	C3—C4—H4	120.4
Cl1—Cd1—Br3	106.4 (6)	N1—C5—C4	120.4 (7)
Cl2—Cd1—Br4	117.7 (6)	N1—C5—H5	119.8
Cl1—Cd1—Br4	114.4 (3)	C4—C5—H5	119.8
Br3—Cd1—Br4	111.9 (3)	C10—N2—C6	121.3 (6)
Cl2—Cd1—Cl4	116.4 (8)	C10—N2—C13	119.9 (6)
Cl1—Cd1—Cl4	123.3 (6)	C6—N2—C13	118.8 (6)
Br3—Cd1—Cl4	105.0 (6)	N2—C6—C7	119.9 (7)
Br4—Cd1—Cl4	9.3 (5)	N2—C6—H6	120.0
Cl2—Cd1—Cl3	109.2 (9)	C7—C6—H6	120.0
Cl1—Cd1—Cl3	112.1 (8)	C8—C7—C6	118.8 (7)
Br3—Cd1—Cl3	5.9 (7)	C8—C7—H7	120.6
Br4—Cd1—Cl3	107.3 (6)	C6—C7—H7	120.6
Cl4—Cd1—Cl3	100.1 (7)	C7—C8—C9	120.3 (8)
Cl2—Cd1—Br1	103.2 (4)	C7—C8—H8	119.9
Cl1—Cd1—Br1	7.9 (4)	C9—C8—H8	119.9
Br3—Cd1—Br1	106.2 (4)	C10—C9—C8	119.0 (8)
Br4—Cd1—Br1	107.84 (13)	C10—C9—H9	120.5
Cl4—Cd1—Br1	116.5 (5)	C8—C9—H9	120.5
Cl3—Cd1—Br1	111.6 (6)	N2—C10—C9	120.6 (7)
Cl2—Cd1—Br2	6.4 (5)	N2—C10—H10	119.7
Cl1—Cd1—Br2	102.2 (4)	C9—C10—H10	119.7
Br3—Cd1—Br2	106.4 (4)	N1—C11—C12	112.4 (5)
Br4—Cd1—Br2	114.72 (16)	N1—C11—H11A	109.1
Cl4—Cd1—Br2	112.5 (6)	C12—C11—H11A	109.1
Cl3—Cd1—Br2	105.8 (8)	N1—C11—H11B	109.1
Br1—Cd1—Br2	109.55 (17)	C12—C11—H11B	109.1
C1—N1—C5	119.6 (6)	H11A—C11—H11B	107.9
C1—N1—C11	120.1 (5)	C13—C12—C11	111.8 (6)
C5—N1—C11	120.3 (6)	C13—C12—H12A	109.3
N1—C1—C2	121.1 (7)	C11—C12—H12A	109.3
N1—C1—H1	119.4	C13—C12—H12B	109.3
C2—C1—H1	119.4	C11—C12—H12B	109.3
C3—C2—C1	121.7 (8)	H12A—C12—H12B	107.9
C3—C2—H2	119.2	N2—C13—C12	110.7 (6)
C1—C2—H2	119.2	N2—C13—H13A	109.5
C2—C3—C4	118.0 (8)	C12—C13—H13A	109.5
C2—C3—H3	121.0	N2—C13—H13B	109.5
C4—C3—H3	121.0	C12—C13—H13B	109.5
C5—C4—C3	119.2 (8)	H13A—C13—H13B	108.1
C5—N1—C1—C2	−0.2 (10)	C6—C7—C8—C9	0.2 (12)
C11—N1—C1—C2	178.9 (7)	C7—C8—C9—C10	0.0 (12)
N1—C1—C2—C3	2.1 (13)	C6—N2—C10—C9	0.4 (10)
C1—C2—C3—C4	−3.0 (13)	C13—N2—C10—C9	−179.2 (6)

C2—C3—C4—C5	2.1 (12)	C8—C9—C10—N2	−0.3 (11)
C1—N1—C5—C4	−0.7 (9)	C1—N1—C11—C12	−66.1 (7)
C11—N1—C5—C4	−179.7 (6)	C5—N1—C11—C12	112.9 (7)
C3—C4—C5—N1	−0.3 (11)	N1—C11—C12—C13	−54.8 (8)
C10—N2—C6—C7	−0.2 (10)	C10—N2—C13—C12	106.4 (7)
C13—N2—C6—C7	179.4 (6)	C6—N2—C13—C12	−73.3 (8)
N2—C6—C7—C8	−0.1 (11)	C11—C12—C13—N2	−175.6 (5)

Fig. 1