

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1,1,4,4-Tetramethylpiperazinium tetraiodocadmate(II)

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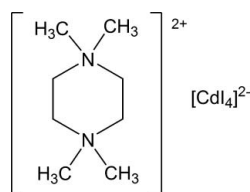
Received 29 September 2007; accepted 12 October 2007

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.033; wR factor = 0.083; data-to-parameter ratio = 29.8.

The title compound, $(\text{C}_8\text{H}_{20}\text{N}_2)[\text{CdI}_4]$, contains isolated 1,1,4,4-tetramethylpiperazinium cations and tetrahedral tetraiodocadmate(II) anions, both of which lie on crystallographic twofold rotation axes. The cations and anions lie in layers parallel to the (101) planes.

Related literature

For the structures of the related compounds, $(\text{C}_8\text{H}_{20}\text{N}_2)[\text{FeCl}_5]$ and $(\text{C}_4\text{H}_{12}\text{N}_2)[\text{CdI}_4]$, see James *et al.* (1995) and Ishihara *et al.* (2002), respectively.



Experimental

Crystal data

$(\text{C}_8\text{H}_{20}\text{N}_2)[\text{CdI}_4]$
 $M_r = 764.26$

Monoclinic, $P2_1/n$
 $a = 9.777$ (4) Å

$b = 7.772$ (4) Å
 $c = 12.184$ (6) Å
 $\beta = 96.326$ (6)°
 $V = 920.2$ (8) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 7.88$ mm⁻¹
 $T = 298$ (2) K
 $0.28 \times 0.14 \times 0.08$ mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.277$, $T_{\max} = 0.533$

5322 measured reflections
2118 independent reflections
1869 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.083$
 $S = 1.05$
2118 reflections

71 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.30$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.51$ e Å⁻³

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Siemens, 1994) and DIAMOND (Brandenburg, 2004); software used to prepare material for publication: SHELXTL.

The authors thank the Project of Fujian Science and Technology Committee (grant No. 2006 F5067) for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2243).

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supplementary materials

Acta Cryst. (2007). E63, m2747 [doi:10.1107/S1600536807050180]

1,1,4,4-Tetramethylpiperazinium tetraiodidocadmiate(II)

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Comment

The title compound (Me₄pip)[CdI₄] (where pip denotes piperazine) is composed of isolated tetrahedral [CdI₄]²⁻ anions and 1,1,4,4-tetra-methylpiperazinium (Me₄pip)²⁺ cations (Fig. 1), both of which lie on crystallographic twofold rotation axes. There does not appear to be any unusual distortion of the metal-halide distances due to the presence of the (Me₄pip)²⁺ cations: the coordination environment of Cd^{II} involves Cd—I bond distances of 2.7664 (10) and 2.7721 (11) Å, with I—Cd—I bond angles ranging between 106.30 (5) and 114.69 (2)°. The cations and anions lie in layers parallel to the (101) planes (Fig. 2), with C—H···I contacts formed between them.

Experimental

Crystals of the title compound were prepared by slow evaporation of a solution containing (Me₄pip)I₂ (0.796 g, 2 mmol) and CdI₂ (1.465 g, 4 mmol) in deionized water (20 ml), acidified by 10 drops of 1M HI. The resulting yellow crystals were obtained in approximately 68% yield (based on Cd).

Refinement

H atoms were positioned geometrically, with C—H = 0.97 or 0.96 Å and were refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. The methyl groups were allowed to rotate about their local threefold axes.

Figures

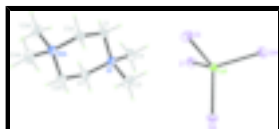


Fig. 1. The molecular structure of the title compound showing displacement ellipsoids at the 35% probability level for non-H atoms.

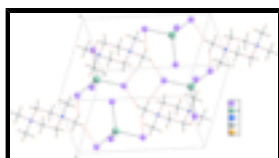


Fig. 2. Packing diagram for the title compound. The dashed lines denote intermolecular C—H···I contacts.

1,1,4,4-Tetramethylpiperazinium tetraiodidocadmium(II)

Crystal data

(C₈H₂₀N₂)[CdI₄]

$F_{000} = 684$

supplementary materials

$M_r = 764.26$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1ac$

$a = 9.777 (4) \text{ \AA}$

$b = 7.772 (4) \text{ \AA}$

$c = 12.184 (6) \text{ \AA}$

$\beta = 96.326 (6)^\circ$

$V = 920.2 (8) \text{ \AA}^3$

$Z = 2$

$D_x = 2.758 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5559 reflections

$\theta = 2.5\text{--}28.3^\circ$

$\mu = 7.88 \text{ mm}^{-1}$

$T = 298 (2) \text{ K}$

Block, yellow

$0.28 \times 0.14 \times 0.08 \text{ mm}$

Data collection

Siemens SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2) \text{ K}$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.277$, $T_{\max} = 0.533$

5322 measured reflections

2118 independent reflections

1869 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 28.3^\circ$

$\theta_{\min} = 2.5^\circ$

$h = -10 \rightarrow 12$

$k = -6 \rightarrow 10$

$l = -13 \rightarrow 15$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.083$

$S = 1.05$

2118 reflections

71 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

$$w = 1/[\sigma^2(F_o^2) + (0.0397P)^2 + 1.5636P]$$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.30 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -1.50 \text{ e \AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cd1	0.2500	0.00204 (7)	0.7500	0.03049 (14)
I1	0.02271 (3)	-0.21007 (5)	0.71028 (3)	0.04278 (13)
I2	0.20422 (4)	0.21594 (5)	0.92431 (3)	0.04632 (14)
N1	0.2645 (4)	0.7067 (5)	0.1294 (3)	0.0285 (8)
C1	0.2223 (5)	0.8634 (6)	0.1897 (4)	0.0314 (10)
H1A	0.1227	0.8692	0.1834	0.038*
H1B	0.2551	0.9653	0.1548	0.038*
C2	0.2221 (5)	0.5506 (6)	0.1900 (4)	0.0330 (10)
H2A	0.2543	0.4484	0.1550	0.040*
H2B	0.1224	0.5456	0.1841	0.040*
C3	0.4172 (5)	0.7061 (8)	0.1194 (5)	0.0440 (14)
H3A	0.4671	0.7024	0.1918	0.066*
H3B	0.4414	0.8085	0.0820	0.066*
H3C	0.4400	0.6069	0.0781	0.066*
C4	0.1892 (6)	0.7085 (8)	0.0150 (4)	0.0413 (12)
H4A	0.0918	0.7050	0.0195	0.062*
H4B	0.2158	0.6100	-0.0252	0.062*
H4C	0.2119	0.8116	-0.0225	0.062*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0308 (2)	0.0302 (3)	0.0304 (3)	0.000	0.00283 (18)	0.000
I1	0.0360 (2)	0.0388 (2)	0.0531 (2)	-0.00967 (14)	0.00302 (16)	-0.00510 (15)
I2	0.0492 (2)	0.0452 (3)	0.0447 (2)	0.00182 (15)	0.00580 (16)	-0.01700 (16)
N1	0.0324 (19)	0.028 (2)	0.026 (2)	-0.0005 (16)	0.0062 (16)	0.0007 (16)
C1	0.039 (2)	0.022 (2)	0.034 (3)	0.0015 (19)	0.0040 (19)	0.001 (2)
C2	0.040 (2)	0.024 (2)	0.035 (3)	-0.0044 (19)	0.008 (2)	-0.002 (2)
C3	0.032 (3)	0.059 (4)	0.044 (3)	-0.001 (2)	0.013 (2)	-0.004 (3)
C4	0.049 (3)	0.047 (3)	0.028 (3)	0.000 (2)	0.001 (2)	0.002 (2)

Geometric parameters (\AA , $^\circ$)

Cd1—I1	2.7664 (10)	C1—H1B	0.970
Cd1—I1 ⁱ	2.7664 (10)	C2—C2 ⁱⁱ	1.504 (10)
Cd1—I2	2.7721 (11)	C2—H2A	0.970
Cd1—I2 ⁱ	2.7721 (11)	C2—H2B	0.970
N1—C2	1.502 (6)	C3—H3A	0.960
N1—C1	1.503 (6)	C3—H3B	0.960
N1—C4	1.504 (6)	C3—H3C	0.960
N1—C3	1.511 (6)	C4—H4A	0.960
C1—C1 ⁱⁱ	1.509 (9)	C4—H4B	0.960

supplementary materials

C1—H1A	0.970	C4—H4C	0.960
I1—Cd1—I1 ⁱ	106.84 (4)	N1—C2—C2 ⁱⁱ	112.8 (3)
I1—Cd1—I2	107.28 (2)	N1—C2—H2A	109.0
I1 ⁱ —Cd1—I2	114.69 (2)	C2 ⁱⁱ —C2—H2A	109.0
I1—Cd1—I2 ⁱ	114.69 (2)	N1—C2—H2B	109.0
I1 ⁱ —Cd1—I2 ⁱ	107.28 (2)	C2 ⁱⁱ —C2—H2B	109.0
I2—Cd1—I2 ⁱ	106.30 (5)	H2A—C2—H2B	107.8
C2—N1—C1	108.0 (4)	N1—C3—H3A	109.5
C2—N1—C4	109.2 (4)	N1—C3—H3B	109.5
C1—N1—C4	108.3 (4)	H3A—C3—H3B	109.5
C2—N1—C3	111.5 (4)	N1—C3—H3C	109.5
C1—N1—C3	111.6 (4)	H3A—C3—H3C	109.5
C4—N1—C3	108.2 (4)	H3B—C3—H3C	109.5
N1—C1—C1 ⁱⁱ	112.8 (3)	N1—C4—H4A	109.5
N1—C1—H1A	109.0	N1—C4—H4B	109.5
C1 ⁱⁱ —C1—H1A	109.0	H4A—C4—H4B	109.5
N1—C1—H1B	109.0	N1—C4—H4C	109.5
C1 ⁱⁱ —C1—H1B	109.0	H4A—C4—H4C	109.5
H1A—C1—H1B	107.8	H4B—C4—H4C	109.5
C2—N1—C1—C1 ⁱⁱ	54.5 (6)	C1—N1—C2—C2 ⁱⁱ	-54.8 (6)
C4—N1—C1—C1 ⁱⁱ	172.6 (5)	C4—N1—C2—C2 ⁱⁱ	-172.3 (5)
C3—N1—C1—C1 ⁱⁱ	-68.4 (6)	C3—N1—C2—C2 ⁱⁱ	68.2 (6)

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

Fig. 1

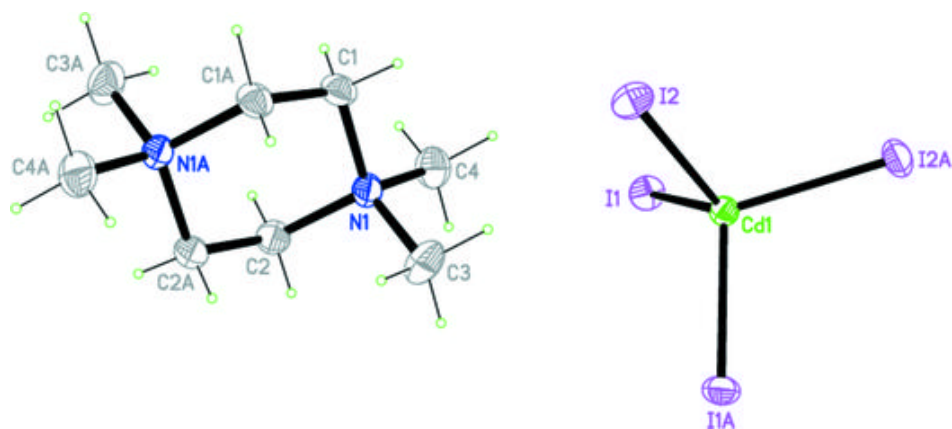


Fig. 2

