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catena-Poly[[iodidocadmium(II)]-di-μ-iodido-[iodidocadmium(II)]-bis(μ-4-methylpyridine *N*-oxide)]

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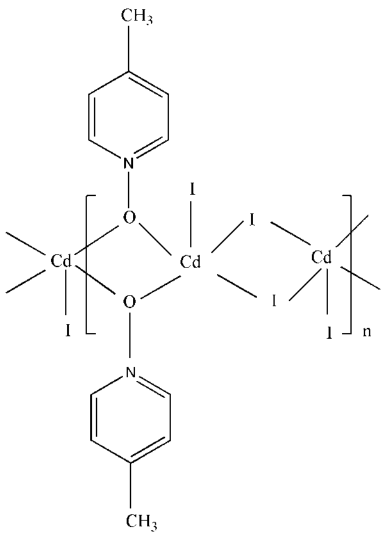
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.030; wR factor = 0.070; data-to-parameter ratio = 21.0.

In the title complex, $[CdI_2(C_6H_7NO)]_n$, the Cd^{II} ion exhibits a trigonal–bipyramidal coordination geometry [Cd—O = 2.272 (4) Å and Cd—I = 2.7054 (7)–2.8049 (8) Å], with iodide anions and 4-methylpyridine *N*-oxide ligands acting as bridging groups to form a one-dimensional chain structure.

Related literature

In most reported crystal structures the Cd^{II} ion has coordination number six (Shi *et al.*, 2006), while those with coordination number five are rarer (Reger *et al.*, 2002). The title structure is similar to that of $\{[Cd(\mu\text{-}I)(\mu\text{-}ONC_5H_5)]I\}_n$ (where ONC_5H_5 is pyridine *N*-oxide) (Sawitzki & Schnering, 1974).

For related literature, see: Pecaut *et al.* (1993); Selvasekarapandian *et al.* (1997).



Experimental

Crystal data

$[CdI_2(C_6H_7NO)]$
M_r = 475.33
Triclinic, $P\bar{1}$
a = 7.0584 (16) Å
b = 8.4025 (19) Å
c = 9.875 (2) Å
α = 102.633 (3)°
β = 96.050 (3)°
γ = 103.510 (3)°
V = 548.1 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 7.58 mm^{-1}
T = 293 (2) K
0.09 × 0.08 × 0.07 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.549, T_{max} = 0.619
3046 measured reflections
2147 independent reflections
1839 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.070
S = 1.02
2147 reflections
102 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.85 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cd1—O1	2.272 (4)	Cd1—I1	2.8049 (8)
Cd1—I2	2.7054 (7)		

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2387).

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supplementary materials

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catena-Poly[[iodidocadmium(II)]-di-μ-iodido-[iodidocadmium(II)]-bis(μ-4-methylpyridine *N*-oxide)]

S.-G. Zhang and J.-M. Shi

Comment

Complexes containing Cd(II) are very common and useful, and they have played an important role in functional materials, especially in the area of optics (Pecaut *et al.*, 1993; Selvasekarapandian *et al.*, 1997). In published work most Cd^{II} ions assume octahedron geometry with six coordinated atoms (Shi *et al.*, 2006), and Cd(II) complexes with coordination number five are limited (Reger *et al.*, 2002). We report here a novel one-dimensional Cd(II) complex (I) with five coordinated atoms.

The asymmetric unit and symmetry-related fragments of (I) are shown in Fig. 1. Atom Cd1 is in a distorted trigonal-bipyramidal CdO_2I_3 coordination geometry (Table 1). Atom I1 acts as a bridging ligand coordinated two Cd^{II} ions with a separation of 3.9511 (11) Å, which creates a four-membered ring with the four atoms strictly coplanar by virtue of the crystallographic inversion center which is in the middle of the four-membered ring. A neutral 4-methylpyridine N-oxide group also acts as bridging ligand with a pair of Cd^{II} ions having a separation of 3.8705 (11) Å also leading to the formation of a four-membered ring with the four atoms strictly coplanar by virtue of the crystallographic inversion center which is also at the middle of this four-membered ring. The dihedral angle of the two planes is 56.99 °. The two kinds of four-membered rings connect alternately, leading to the formation of a one-dimensional zigzag chain along the *a* axis as shown in Fig. 2. There is one weak interchain C—H···I interaction [H6A···I1(x, $-1 + y$, $-1 + z$) = 3.24 Å; C6—H6A···I1(x, $-1 + y$, $-1 + z$) = 160°. The chain structure in (I) is very similar to that of the complex $\{[Cd(\mu\text{-}I)(\mu\text{-}ONC_5H_5)]I\}_n$ (where ONC_5H_5 is pyridine N-oxide) (Sawitzki & Schnering, 1974), but the crystal system and space groups of the two crystals are different and probably attributed to the minor difference of the methyl substitution.

Experimental

$Cd(ClO_4)_2\cdot 6H_2O$ (0.2724 g, 0.649 mmol), 4-methylpyridine N-oxide (0.0793 g, 0.727 mmol) and NaI (0.2417 g, 1.61 mmol) were dissolved in 10 ml H_2O, respectively, and then the three solutions were mixed together. The colorless single crystals (I) were obtained after allowing the mixed solution to stand at room temperature for two weeks.

Refinement

The H atoms were placed in calculated positions and refined as riding, with C—H = 0.93 Å, U_{iso}(H) = 1.2_{eq}(C) for pyridine ring; C—H = 0.96 Å, U_{iso}(H) = 1.5_{eq}(C) for the methyl group.

Figures

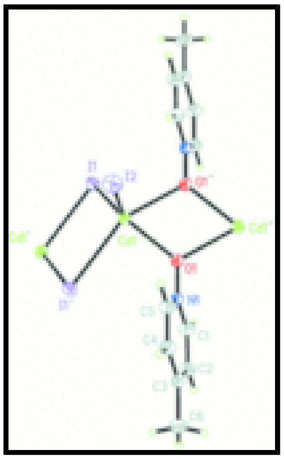

Fig. 1. Part of the structure of (I) showing the atom numbering scheme with thermal ellipsoids drawn at the 30% probability level. [Symmetry codes: (i) −*x*, −*y* + 2, −*z* + 2; (ii) −*x* + 1, −*y* + 2, −*z* + 2].

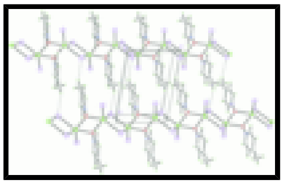

Fig. 2. Part of the crystal structure of (I). Dashed lines indicate weak C—H···I interactions.

catena-Poly[[iodidocadmium(II)]-di-μ-iodido-[iodidocadmium(II)]-bis(μ- 4-methylpyridine N-oxide)]

Crystal data

$[CdI_2(C_6H_7NO)]$

$M_r = 475.33$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

$a = 7.0584\ (16)$ Å

$b = 8.4025\ (19)$ Å

$c = 9.875\ (2)$ Å

$\alpha = 102.633\ (3)°$

$\beta = 96.050\ (3)°$

$\gamma = 103.510\ (3)°$

$V = 548.1\ (2)$ Å^3

$Z = 2$

$F_{000} = 424$

$D_x = 2.880$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å

Cell parameters from 1365 reflections

$\theta = 2.6–26.8°$

$\mu = 7.58$ mm^{-1}

$T = 293\ (2)$ K

Block, colorless

0.09 × 0.08 × 0.07 mm

Data collection

Bruker SMART APEX CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

φ and ω scans

Absorption correction: multi-scan

2147 independent reflections

1839 reflections with $I > 2\sigma(I)$

$R_{int} = 0.017$

$\theta_{max} = 26.2°$

$\theta_{min} = 2.1°$

$h = -8 \rightarrow 8$

(SADABS; Sheldrick, 1996)	
T_{min} = 0.549, T_{max} = 0.619	$k = -8\rightarrow10$
3046 measured reflections	$l = -12\rightarrow10$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.070$	$(\Delta/\sigma)_{max} = 0.037$
$S = 1.02$	$\Delta\rho_{max} = 0.85$ e Å^{-3}
2147 reflections	$\Delta\rho_{min} = -0.89$ e Å^{-3}
102 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0063 (5)
Secondary atom site location: difference Fourier map	

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 $F^2 > \sigma(F^2)$ is used only for calculating 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}
Cd1	0.25296 (6)	0.96122 (5)	1.07178 (4)	0.03312 (14)
I1	0.12264 (5)	1.25617 (5)	1.10555 (4)	0.03973 (14)
I2	0.31135 (7)	0.81337 (6)	1.28439 (4)	0.05230 (16)
C6	0.1724 (11)	0.2298 (9)	0.5226 (7)	0.0588 (19)
H6A	0.1267	0.2389	0.4302	0.088*
H6B	0.0671	0.1605	0.5548	0.088*
H6C	0.2813	0.1795	0.5192	0.088*
N1	0.3549 (6)	0.7153 (6)	0.8070 (5)	0.0322 (10)
C4	0.2630 (9)	0.4203 (8)	0.7651 (6)	0.0419 (14)
H4	0.2387	0.3249	0.8005	0.050*
O1	0.4163 (6)	0.8687 (5)	0.8971 (4)	0.0409 (10)
C1	0.3308 (9)	0.7026 (8)	0.6688 (6)	0.0438 (14)
H1	0.3538	0.7997	0.6357	0.053*

C5	0.3229 (9)	0.5765 (8)	0.8557 (6)	0.0419 (14)
H5	0.3418	0.5869	0.9522	0.050*
C2	0.2719 (9)	0.5459 (9)	0.5757 (6)	0.0475 (16)
H2	0.2549	0.5384	0.4796	0.057*
C3	0.2378 (9)	0.4012 (8)	0.6209 (6)	0.0397 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0310 (2)	0.0323 (2)	0.0372 (2)	0.01002 (18)	0.00636 (17)	0.00883 (17)
I1	0.0335 (2)	0.0305 (2)	0.0513 (3)	0.00987 (16)	−0.00021 (17)	0.00418 (17)
I2	0.0631 (3)	0.0504 (3)	0.0487 (3)	0.0166 (2)	0.0047 (2)	0.0239 (2)
C6	0.070 (5)	0.042 (4)	0.051 (4)	0.004 (4)	0.014 (4)	−0.005 (3)
N1	0.030 (2)	0.025 (2)	0.037 (3)	0.0046 (19)	0.0080 (19)	0.001 (2)
C4	0.050 (4)	0.029 (3)	0.050 (4)	0.013 (3)	0.011 (3)	0.011 (3)
O1	0.035 (2)	0.033 (2)	0.047 (2)	0.0046 (17)	0.0123 (18)	−0.0046 (18)
C1	0.053 (4)	0.038 (4)	0.041 (3)	0.010 (3)	0.008 (3)	0.013 (3)
C5	0.046 (4)	0.043 (4)	0.037 (3)	0.012 (3)	0.006 (3)	0.010 (3)
C2	0.052 (4)	0.049 (4)	0.038 (3)	0.010 (3)	0.005 (3)	0.010 (3)
C3	0.039 (3)	0.039 (4)	0.037 (3)	0.007 (3)	0.011 (3)	0.005 (3)

Geometric parameters (Å, °)

Cd1—O1	2.272 (4)	N1—C5	1.337 (8)
Cd1—O1^{i}	2.384 (4)	N1—O1	1.340 (5)
Cd1—I2	2.7054 (7)	C4—C5	1.360 (8)
Cd1—I1	2.8049 (8)	C4—C3	1.385 (8)
Cd1—I1^{ii}	2.9650 (7)	C4—H4	0.9300
I1—Cd1^{ii}	2.9650 (7)	O1—Cd1^{i}	2.384 (4)
C6—C3	1.487 (8)	C1—C2	1.374 (9)
C6—H6A	0.9600	C1—H1	0.9300
C6—H6B	0.9600	C5—H5	0.9300
C6—H6C	0.9600	C2—C3	1.364 (9)
N1—C1	1.335 (7)	C2—H2	0.9300
O1—Cd1—O1^{i}	67.55 (15)	C5—N1—O1	120.0 (5)
O1—Cd1—I2	109.61 (11)	C5—C4—C3	121.0 (6)
O1^{i}—Cd1—I2	96.51 (11)	C5—C4—H4	119.5
O1—Cd1—I1	124.55 (11)	C3—C4—H4	119.5
O1^{i}—Cd1—I1	88.89 (10)	N1—O1—Cd1	123.9 (3)
I2—Cd1—I1	122.90 (2)	N1—O1—Cd1^{i}	121.8 (3)
O1—Cd1—I1^{ii}	88.83 (10)	Cd1—O1—Cd1^{i}	112.45 (15)
O1^{i}—Cd1—I1^{ii}	152.48 (9)	N1—C1—C2	119.9 (6)
I2—Cd1—I1^{ii}	104.94 (2)	N1—C1—H1	120.1
I1—Cd1—I1^{ii}	93.61 (2)	C2—C1—H1	120.1
Cd1—I1—Cd1^{ii}	86.39 (2)	N1—C5—C4	120.5 (5)
C3—C6—H6A	109.5	N1—C5—H5	119.8

C3—C6—H6B	109.5	C4—C5—H5	119.8
H6A—C6—H6B	109.5	C3—C2—C1	121.5 (6)
C3—C6—H6C	109.5	C3—C2—H2	119.3
H6A—C6—H6C	109.5	C1—C2—H2	119.3
H6B—C6—H6C	109.5	C2—C3—C4	116.7 (6)
C1—N1—C5	120.5 (5)	C2—C3—C6	122.7 (6)
C1—N1—O1	119.4 (5)	C4—C3—C6	120.6 (6)

O1—Cd1—I1—Cd1^{ii}	91.06 (12)	I2—Cd1—O1—Cd1^{i}	−89.03 (17)
O1^{i}—Cd1—I1—Cd1^{ii}	152.57 (10)	I1—Cd1—O1—Cd1^{i}	71.94 (19)
I2—Cd1—I1—Cd1^{ii}	−110.39 (3)	I1^{ii}—Cd1—O1—Cd1^{i}	165.52 (16)
I1^{ii}—Cd1—I1—Cd1^{ii}	0.0	C5—N1—C1—C2	0.3 (9)
C1—N1—O1—Cd1	125.4 (5)	O1—N1—C1—C2	178.6 (5)
C5—N1—O1—Cd1	−56.2 (6)	C1—N1—C5—C4	−0.8 (9)
C1—N1—O1—Cd1^{i}	−71.6 (6)	O1—N1—C5—C4	−179.2 (5)
C5—N1—O1—Cd1^{i}	106.8 (5)	C3—C4—C5—N1	1.3 (10)
O1^{i}—Cd1—O1—N1	164.4 (5)	N1—C1—C2—C3	−0.3 (10)
I2—Cd1—O1—N1	75.4 (4)	C1—C2—C3—C4	0.8 (10)
I1—Cd1—O1—N1	−123.6 (4)	C1—C2—C3—C6	179.5 (6)
I1^{ii}—Cd1—O1—N1	−30.1 (4)	C5—C4—C3—C2	−1.3 (9)
O1^{i}—Cd1—O1—Cd1^{i}	0.0	C5—C4—C3—C6	179.9 (6)

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

Fig. 1

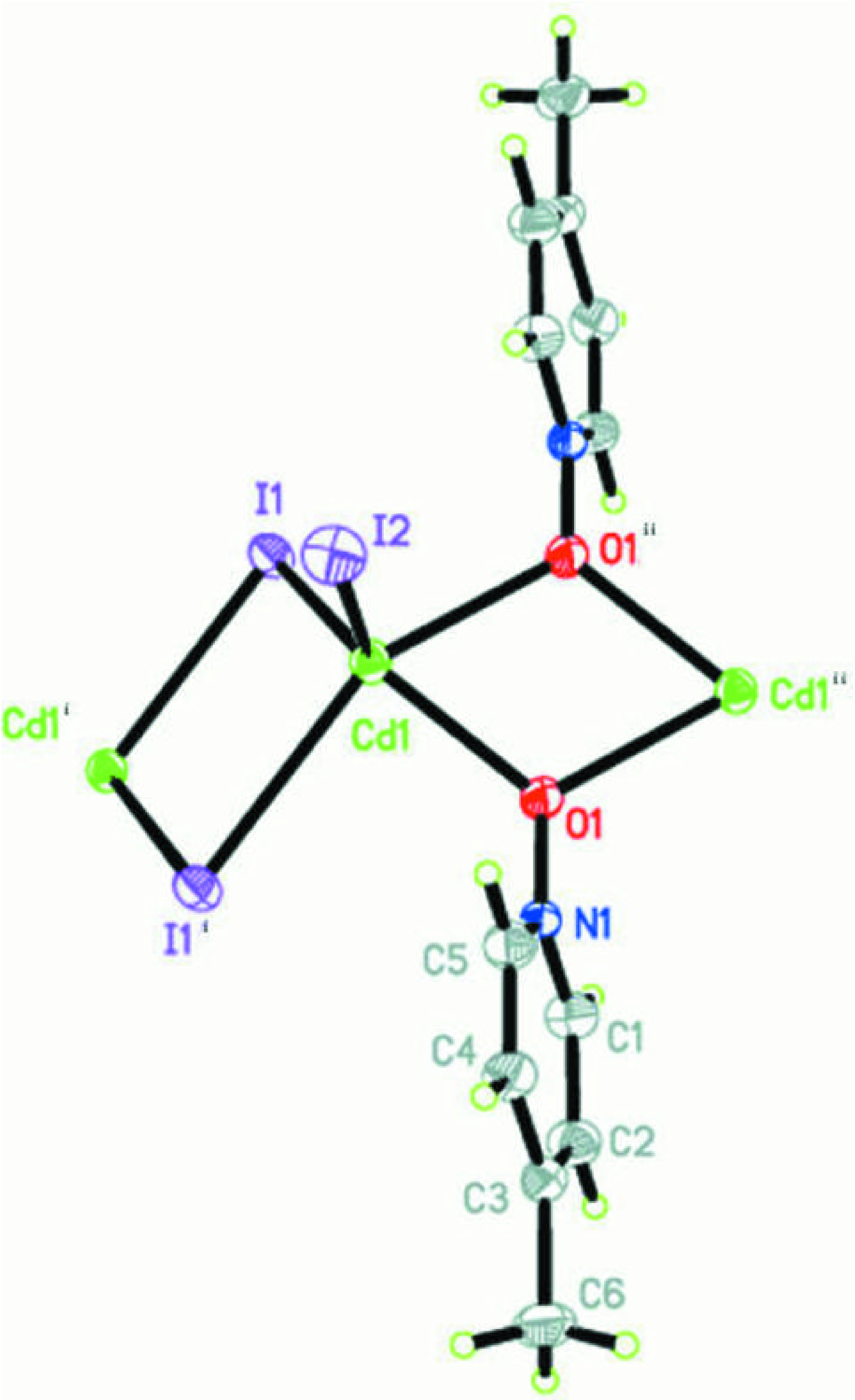

Fig. 2