Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

catena-Poly[[dipyridinecadmium(II)]-μ-5-amino-2,4,6-triiodoisophthalato]

Yang Zou

Chemistry Department, Zhejiang Sci-Tech University, Hangzhou, 310018, People's Republic of China
Correspondence e-mail: zouyang@zstu.edu.cn

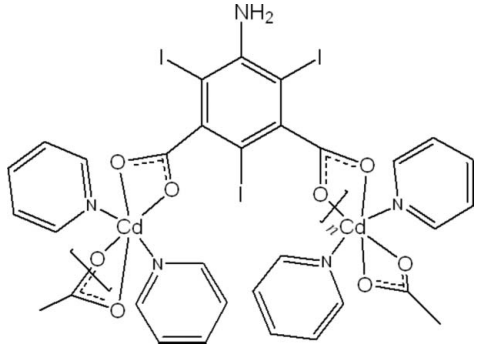
Received 26 September 2010; accepted 3 October 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.014 Å; R factor = 0.038; wR factor = 0.089; data-to-parameter ratio = 20.3.

The hydrothermal reaction of cadmium(II) nitrate with 5-amino-2,4,6-triiodoisophthalic acid and pyridine in DMF solution leads to the formation of the title compound, $[Cd(C_8H_2I_3NO_4)(C_5H_5N)_2]_n$. The structure contains a four-coordinate Cd^{2+} ion in a distorted tetrahedral geometry, which lies on a crystallographic twofold rotation axis. The Cd^{2+} ion is bonded to two N atoms from two pyridine ligands and two carboxylate O atoms from two 5-amino-2,4,6-triiodoisophthalate anions. The Cd—O distances are 2.429 (5) and 2.305 (5) Å and the Cd—N distance is 2.236 (8) Å. The two carboxylate groups of individual 5-amino-2,4,6-triiodoisophthalate anions act as a bridge to the Cd^{2+} atoms. leading to a chain structure along the c axis.

Related literature

For the isotypic Hg complex, see: Zhang *et al.* (2008). For the structure of 5-amino-2,4,6-triiodoisophthalic acid monohydrate, see: Beck & Sheldrick (2008). For the structures of related metal complexes, see: Dai *et al.* (2008)·For the use of triiodinated aromatic compounds in radiology, see: Estep *et al.* (2000).



Experimental

Crystal data

$[Cd(C_8H_2I_3NO_4)(C_5H_5N)_2]$
M_r = 827.41
Tetragonal, $P4_12_12$
a = 11.824 (3) Å
c = 15.841 (9) Å
V = 2214.7 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 5.20 mm^{-1}
T = 293 K
0.25 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker 2003)
T_{min} = 0.357, T_{max} = 0.423
14248 measured reflections
2714 independent reflections
1949 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.089
S = 1.02
2714 reflections
134 parameters
60 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}
Absolute structure: Flack (1983), 1096 Friedel pairs
Flack parameter: −0.04 (6)

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The author thanks the Natural Science Foundation of Zhejiang Province, China (No. Y4080342) and the Science Foundation of Zhejiang Sci-Tech University (No. 0813622-Y) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2296).

References

Beck, T. & Sheldrick, G. M. (2008). *Acta Cryst.* E**64**, o1286.
Brandenburg, K. (2000). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2003). *SADABS*, *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin,USA.
Dai, F., He, H. & Sun, D. (2008). *J. Am. Chem. Soc.* **130**, 14064–14065.
Estep, K. G., Josef, K. A., Bacon, E. R., Illig, C. R., Toner, J. L., Mishra, D., Blazak, W. F., Miller, D. M., Johnson, D. K., Allen, J. M., Spencer, A. & Wilson, S. A. (2000). *J. Med. Chem.* **43**, 1940–1948.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Zhang, Y., Zhao, J., Tang, G. & Jiang, Z. (2008). *Acta Cryst.* E**64**, m1324.

supporting information

Acta Cryst. (2010). E**66**, m1371 [https://doi.org/10.1107/S1600536810039498]

catena-Poly[[dipyridinecadmium(II)]-μ-5-amino-2,4,6-triiodoisophthalato]

Yang Zou

S1. Comment

5-Amino-2,4,6-triiodoisophthalic acid (ATIA), is the precursor and core structure of triiodinated contrast media used in radiology (Estep *et al.*, 2000). The crystal structure of this compound was reported recently (Beck *et al.*, 2008), however, there are very few studies that have been reported on the structural characterization of its metal complexes (Dai *et al.*, 2008; Zhang *et al.*, 2008). Here we report the synthesis and crystal structure of the title complex *catena*-[bis(pyridine)-μ-5-amino-2,4,6-triiodoisophthalic acid-O,*O*-cadmium(II)].

In the title complex the central cadmium ion is coordinated by two nitrogen atoms from two pyridine ligands and two oxygen atoms from different ATIA ligands in a tetrahedral geometry. The bond lengths are 2.236 (8)Å for Cd1—N2; 2.305 (5) Å for Cd1—O2 and 2.429 (5) Å for Cd1—O1. Both carboxylate groups of ATIA ligand are deprotonated during the reaction, and the whole ligand acts as a bridging linker to connect two cadmium ions. Thus, the [Cd(pyr)$_2$] units are infinitely connected by ATIA ligands along the *c* axis to give rise to a one-dimensional chain structure.

S2. Experimental

5-amino-2,4,6-triiodoisophthalic acid (0.5 mmol) was dissolved in 10 ml DMA, in which Cd(NO$_3$)$_2$(0.5 mmol) and 20 μl pyridine were added in. The mixture was sealed in a Pyrex tube and heated at 358 K for 3 d. After cooling to room temperature, light yellow block crystals were obtained.

S3. Refinement

All H atoms were positioned geometrically and constrained as riding atoms, with C—H distance of 0.93 Å and U_{iso}(H) set to 1.2 U_{eq}(C) of the parent atom.

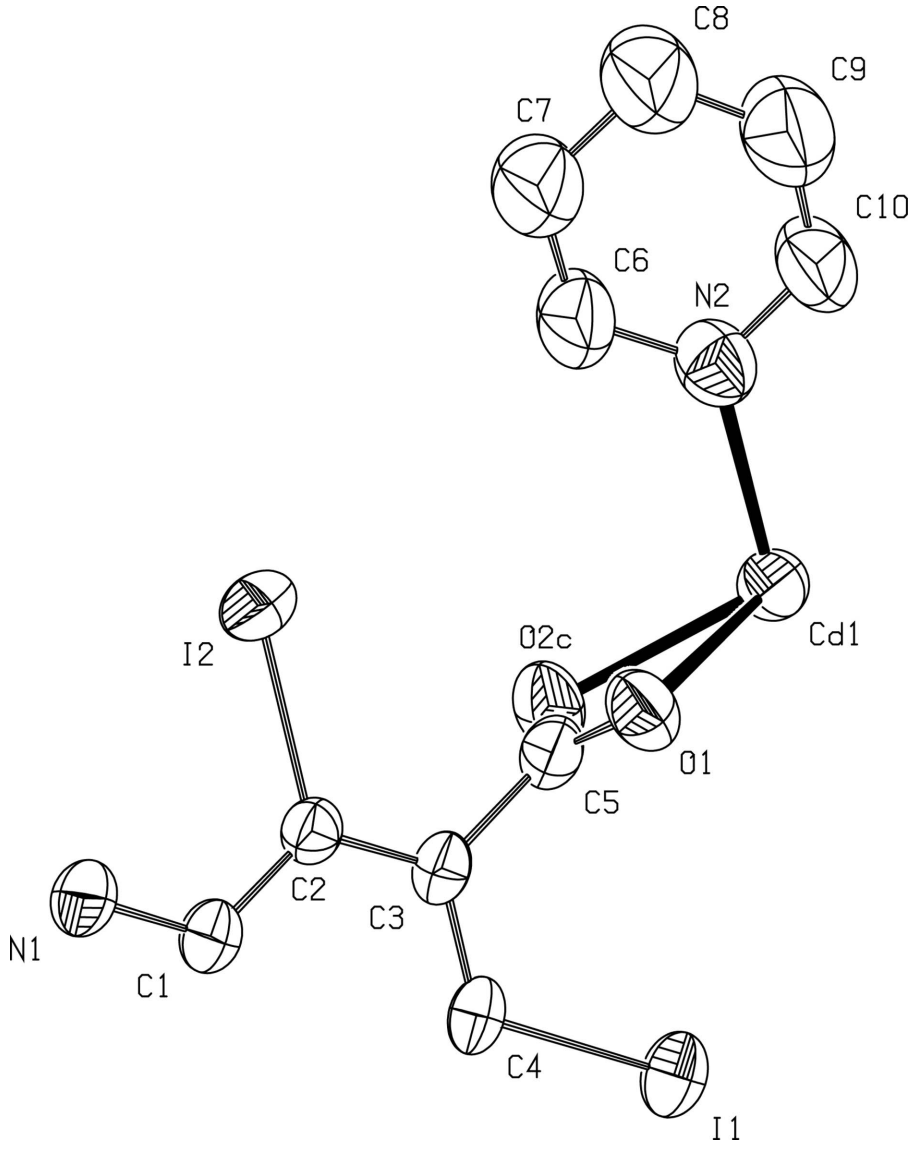


Figure 1

ORTEP plot of the title complex with atom numbering scheme. Thermal ellipsoids are drawn at 40% probability level.

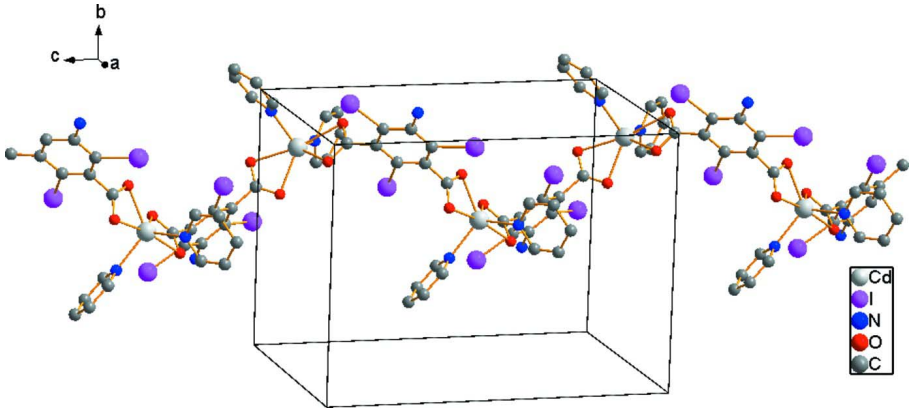


Figure 2

A section of the infinite [Cd(ATIA)(pyr)$_2$]n chain along the *c* axis.

catena-Poly[[dipyridinecadmium(II)]-μ-5-amino-2,4,6- triiodoisophthalato]

Crystal data

$[Cd(C_8H_2I_3NO_4)(C_5H_5N)_2]$
M_r = 827.41
Tetragonal, $P4_12_12$
Hall symbol: P 4abw 2nw
a = 11.824 (3) Å
c = 15.841 (9) Å
V = 2214.7 (15) Å^3
Z = 4
$F(000)$ = 1520
D_x = 2.482 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1949 reflections
θ = 2.2–28.2°
μ = 5.20 mm^{-1}
T = 293 K
Block, light yellow
0.25 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: none pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker 2003)
T_{min} = 0.357, T_{max} = 0.423
14248 measured reflections
2714 independent reflections
1949 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 28.2°, θ_{min} = 2.2°
h = −15→15
k = −14→15
l = −17→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.089
S = 1.02
2714 reflections
134 parameters
60 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
$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 2.974P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}
Absolute structure: Flack (1983), 1096 Friedel pairs
Absolute structure parameter: −0.04 (6)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cd1	0.62771 (5)	0.37229 (5)	0.2500	0.0572 (2)	
I1	0.51631 (4)	0.51631 (4)	0.0000	0.0630 (2)	
I2	0.28105 (5)	0.12921 (6)	0.17206 (4)	0.0802 (2)	
N1	0.1416 (5)	0.1416 (5)	0.0000	0.064 (2)	
H1A	0.1357	0.0961	0.0421	0.077*	0.50
H1B	0.0961	0.1357	−0.0421	0.077*	0.50
N2	0.6179 (7)	0.2214 (7)	0.3348 (5)	0.0771 (19)	
O1	0.5507 (5)	0.2646 (5)	0.1328 (3)	0.0668 (15)	
O2	0.6079 (5)	0.5525 (4)	0.3035 (3)	0.0684 (16)	
C1	0.2221 (6)	0.2221 (6)	0.0000	0.051 (2)	
C2	0.2982 (6)	0.2357 (6)	0.0666 (4)	0.0462 (16)	
C3	0.3809 (6)	0.3171 (6)	0.0673 (4)	0.0479 (17)	
C4	0.3908 (5)	0.3908 (5)	0.0000	0.043 (2)	
C5	0.4652 (7)	0.3247 (7)	0.1375 (5)	0.0540 (19)	
C6	0.5209 (10)	0.1691 (10)	0.3519 (9)	0.113 (3)	
H6	0.4560	0.1946	0.3248	0.135*	
C7	0.5108 (12)	0.0835 (12)	0.4049 (10)	0.134 (4)	
H7	0.4419	0.0464	0.4114	0.161*	
C8	0.6063 (12)	0.0494 (13)	0.4514 (11)	0.147 (4)	
H8	0.6026	−0.0039	0.4944	0.177*	
C9	0.7027 (12)	0.0999 (13)	0.4288 (10)	0.143 (4)	
H9	0.7701	0.0745	0.4523	0.172*	
C10	0.7060 (10)	0.1827 (10)	0.3754 (8)	0.113 (4)	
H10	0.7755	0.2169	0.3654	0.135*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.0617 (3)	0.0617 (3)	0.0482 (4)	−0.0120 (4)	−0.0049 (3)	−0.0049 (3)
I1	0.0559 (3)	0.0559 (3)	0.0773 (5)	−0.0148 (3)	0.0062 (3)	−0.0062 (3)
I2	0.0755 (4)	0.0957 (5)	0.0695 (3)	−0.0276 (4)	−0.0027 (3)	0.0291 (3)
N1	0.060 (4)	0.060 (4)	0.071 (5)	−0.027 (5)	−0.007 (4)	0.007 (4)
N2	0.075 (5)	0.073 (5)	0.083 (5)	−0.004 (4)	−0.013 (4)	−0.005 (4)
O1	0.054 (3)	0.091 (4)	0.056 (3)	0.008 (3)	−0.006 (3)	−0.012 (3)
O2	0.091 (4)	0.056 (3)	0.058 (3)	−0.003 (3)	−0.026 (3)	−0.005 (2)
C1	0.047 (3)	0.047 (3)	0.058 (5)	−0.009 (5)	0.003 (4)	−0.003 (4)

C2	0.041 (4)	0.050 (4)	0.048 (4)	−0.001 (3)	0.005 (3)	0.001 (3)
C3	0.040 (4)	0.052 (4)	0.052 (4)	−0.006 (3)	0.009 (3)	−0.005 (3)
C4	0.036 (3)	0.036 (3)	0.058 (5)	0.000 (4)	0.013 (3)	−0.013 (3)
C5	0.048 (5)	0.061 (5)	0.053 (4)	−0.011 (4)	0.006 (4)	−0.003 (4)
C6	0.077 (6)	0.105 (7)	0.156 (9)	−0.028 (6)	−0.026 (6)	0.040 (6)
C7	0.102 (7)	0.127 (8)	0.173 (9)	−0.027 (7)	−0.027 (7)	0.054 (7)
C8	0.105 (8)	0.142 (8)	0.196 (9)	−0.010 (7)	−0.031 (8)	0.062 (8)
C9	0.108 (8)	0.129 (8)	0.192 (9)	−0.026 (7)	−0.043 (8)	0.046 (8)
C10	0.075 (6)	0.102 (7)	0.161 (9)	−0.008 (6)	−0.038 (6)	0.048 (6)

Geometric parameters (Å, º)

Cd1—N2	2.236 (8)	C1—C2	1.395 (9)
Cd1—N2^{i}	2.236 (8)	C1—C2^{ii}	1.395 (9)
Cd1—O2^{i}	2.305 (5)	C2—C3	1.373 (9)
Cd1—O2	2.305 (5)	C3—C4	1.382 (8)
Cd1—O1^{i}	2.429 (5)	C3—C5	1.495 (11)
Cd1—O1	2.429 (5)	C4—C3^{ii}	1.382 (8)
Cd1—C5^{i}	2.681 (8)	C5—O2^{i}	1.246 (9)
Cd1—C5	2.681 (8)	C6—C7	1.321 (16)
I1—C4	2.098 (8)	C6—H6	0.9300
I2—C2	2.102 (7)	C7—C8	1.407 (17)
N1—C1	1.346 (12)	C7—H7	0.9300
N1—H1A	0.8600	C8—C9	1.336 (17)
N1—H1B	0.8600	C8—H8	0.9300
N2—C10	1.307 (12)	C9—C10	1.293 (16)
N2—C6	1.331 (13)	C9—H9	0.9300
O1—C5	1.239 (10)	C10—H10	0.9300
O2—C5^{i}	1.246 (9)		
N2—Cd1—N2^{i}	116.3 (4)	C5^{i}—O2—Cd1	93.2 (5)
N2—Cd1—O2^{i}	104.7 (3)	N1—C1—C2	122.5 (4)
N2^{i}—Cd1—O2^{i}	120.8 (2)	N1—C1—C2^{ii}	122.5 (4)
N2—Cd1—O2	120.8 (2)	C2—C1—C2^{ii}	115.0 (9)
N2^{i}—Cd1—O2	104.7 (3)	C3—C2—C1	123.1 (7)
O2^{i}—Cd1—O2	87.0 (3)	C3—C2—I2	118.8 (5)
N2—Cd1—O1^{i}	82.4 (2)	C1—C2—I2	118.0 (5)
N2^{i}—Cd1—O1^{i}	91.2 (2)	C2—C3—C4	119.8 (7)
O2^{i}—Cd1—O1^{i}	136.7 (2)	C2—C3—C5	121.6 (7)
O2—Cd1—O1^{i}	54.96 (18)	C4—C3—C5	118.6 (6)
N2—Cd1—O1	91.2 (2)	C3^{ii}—C4—C3	119.2 (8)
N2^{i}—Cd1—O1	82.4 (2)	C3^{ii}—C4—I1	120.4 (4)
O2^{i}—Cd1—O1	54.96 (18)	C3—C4—I1	120.4 (4)
O2—Cd1—O1	136.7 (2)	O1—C5—O2^{i}	123.3 (7)
O1^{i}—Cd1—O1	167.9 (3)	O1—C5—C3	117.7 (7)
N2—Cd1—C5^{i}	100.6 (3)	O2^{i}—C5—C3	119.0 (7)
N2^{i}—Cd1—C5^{i}	101.2 (3)	O1—C5—Cd1	64.8 (4)
O2^{i}—Cd1—C5^{i}	111.4 (2)	O2^{i}—C5—Cd1	59.1 (4)

O2—Cd1—C5^{i}	27.7 (2)	C3—C5—Cd1	169.9 (5)
O1^{i}—Cd1—C5^{i}	27.5 (2)	C7—C6—N2	124.3 (13)
O1—Cd1—C5^{i}	164.3 (3)	C7—C6—H6	117.8
N2—Cd1—C5	101.2 (3)	N2—C6—H6	117.8
N2^{i}—Cd1—C5	100.6 (3)	C6—C7—C8	118.7 (14)
O2^{i}—Cd1—C5	27.7 (2)	C6—C7—H7	120.7
O2—Cd1—C5	111.4 (2)	C8—C7—H7	120.7
O1^{i}—Cd1—C5	164.3 (3)	C9—C8—C7	114.6 (15)
O1—Cd1—C5	27.5 (2)	C9—C8—H8	122.7
C5^{i}—Cd1—C5	138.0 (4)	C7—C8—H8	122.7
C1—N1—H1A	120.0	C10—C9—C8	122.6 (15)
C1—N1—H1B	120.0	C10—C9—H9	118.7
H1A—N1—H1B	120.0	C8—C9—H9	118.7
C10—N2—C6	115.1 (9)	C9—C10—N2	124.2 (12)
C10—N2—Cd1	122.3 (7)	C9—C10—H10	117.9
C6—N2—Cd1	122.5 (7)	N2—C10—H10	117.9
C5—O1—Cd1	87.7 (5)		

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