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Crystal structure of diiodido-bis(phenanthridine-κ*N*)cadmium(II), $CdI_2(C_{13}H_9N)_2$, $C_{26}H_{18}CdI_2N_2$

Guo-Liang Zhang, Yi-Long Zheng, Juan Qiao*, Li-Duo Wang and Yong Qiu

Key Laboratory of Organic Optoelectronics and Molecular Engineering of Ministry of Education, Department of Chemistry, Tsinghua University, Beijing 100084, P. R. China

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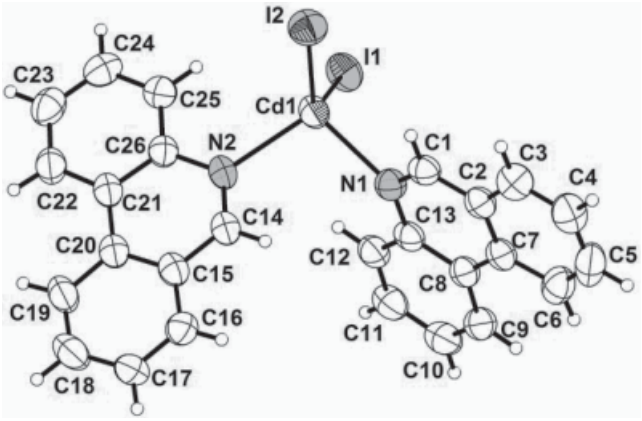

Abstract

$C_{26}H_{18}CdI_2N_2$, monoclinic, $P2_1/c$ (no. 14), a = 16.805(4) Å, b = 10.693(3) Å, c = 14.737(3) Å, β = 115.24(2)°, V = 2395.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.0428, $wR_{ref}(F^2)$ = 0.1033, T = 295 K.

Table 1. Data collection and handling.

Crystal:	colourless prisms, size 0.1×0.4×0.6 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	35.05 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5551, 4446
$N(param)_{refined}$:	280
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3722
Programs:	SHELX [12]

Source of material

The title compound was synthesized through the coordination reaction between CdI_2 and phenanthridine at 1:2 molar ratio in refluxed anhydrous ethanol solution. A mixture of CdI_2 (1.06 mmol) and phenanthridine (2.12 mmol) was dissolved in ethanol (50 ml) solution. After the mixture was stirred for 2h while being heated at reflux, the reaction solution was filtered immediately and the filtrate was cooled to room temperature to give white power (yield: 0.64 g, 82.5 %). A single crystal suitable for X-ray crystallography was obtained by recrystallization from ethanol solution. **Elemental analysis** found: C, 42.94 %; H, 2.55 %; N, 3.87 %; calcd. for $C_{26}H_{18}CdI_2N_2$ (724.6): C, 43.09 %; H, 2.50 %; N, 3.87 %.

Experimental details

The highest and lowest difference electron peaks are 1.386 and –0.578 e·Å^3, respectively.

Discussion

Organic-inorganic hybrids materials and coordination polymers based on metal halide and organic N-donor ligands have been widely studied because of their intriguing architectures and various potential applications [1-6]. For group 12 (IIB) metal halides and pyridine-type ligands, the reported hybrid materials generally have a formal composition $[MX_2(L)_2]$, where M represents a divalent metal cation (Zn, Cd, Hg), X a halogen ligand (Cl, Br, I), L a pyridine derivative such as pyridine, pyrazine or bipyridine, etc. [7-10]. In most cases, zinc (II) derivatives form mononuclear tetrahedral complexes, whereas the higher homologue cadmium(II) yields chain polymers [7-10]. Different from the chain polymers of most reported CdX_2L_2, the title compound is a mononuclear tetrahedral complex. The Cd(II) atom is four-coordinated in a distorted tetrahedral configuration by two N atoms from two phenanthridine and two iodido ligands, which is very similar to the corresponding zinc(II) complex, $ZnCl_2(C_{13}H_9N)_2$ [11]. The angle at the central metal subtended by the nitrogen atoms of the phenanthridine ligands is 102.27(17)°. The bulky phenanthridine rings exclude mirror symmetry and the dihedral angle between them is 69.32°. There are moderate π-π contacts with the shortest distance to be 3.36(5) Å between phenanthridine rings of adjacent molecules.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.5891	0.8563	0.1760	0.060
H(3A)	4*e*	0.4552	0.9301	0.0370	0.080
H(4A)	4*e*	0.3197	0.8866	–0.0913	0.086
H(5A)	4*e*	0.2680	0.6846	–0.1191	0.092
H(6A)	4*e*	0.3478	0.5228	–0.0211	0.081
H(9A)	4*e*	0.4339	0.3761	0.0775	0.073
H(10A)	4*e*	0.5260	0.2310	0.1865	0.082
H(11A)	4*e*	0.6585	0.2916	0.3138	0.078
H(12A)	4*e*	0.6988	0.4985	0.3327	0.069
H(14A)	4*e*	0.7832	0.6162	0.2181	0.060
H(16A)	4*e*	0.8251	0.5124	0.0998	0.071
H(17A)	4*e*	0.9307	0.4157	0.0633	0.082
H(18A)	4*e*	1.0768	0.4094	0.1861	0.091
H(19A)	4*e*	1.1141	0.4974	0.3399	0.078
H(22A)	4*e*	1.1441	0.5881	0.4838	0.076
H(23A)	4*e*	1.1676	0.6788	0.6353	0.082
H(24A)	4*e*	1.0526	0.7685	0.6578	0.081
H(25A)	4*e*	0.9118	0.7640	0.5287	0.069

* Correspondence author (e-mail: qjuan@mail.tsinghua.edu.cn)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd(1)	4*e*	0.73815(3)	0.76298(4)	0.36835(3)	0.0451(2)	0.0463(2)	0.0566(3)	0.0038(2)	0.0254(2)	0.0015(2)
I(1)	4*e*	0.71643(3)	0.67947(4)	0.53137(3)	0.0638(3)	0.0649(3)	0.0608(3)	0.0071(2)	0.0349(2)	0.0084(2)
I(2)	4*e*	0.74558(3)	1.01110(4)	0.33560(4)	0.0723(3)	0.0452(2)	0.0753(3)	–0.0009(2)	0.0327(2)	0.0023(2)
N(1)	4*e*	0.6200(3)	0.6899(5)	0.2280(4)	0.048(3)	0.047(3)	0.051(3)	–0.002(2)	0.025(2)	–0.002(2)
N(2)	4*e*	0.8554(3)	0.6686(4)	0.3541(4)	0.039(2)	0.048(3)	0.052(3)	0.004(2)	0.024(2)	0.004(2)
C(1)	4*e*	0.5693(4)	0.7740(6)	0.1665(5)	0.057(4)	0.048(3)	0.054(3)	–0.003(3)	0.032(3)	–0.003(3)
C(2)	4*e*	0.4860(4)	0.7496(6)	0.0858(5)	0.055(4)	0.060(4)	0.050(3)	–0.001(3)	0.033(3)	–0.002(3)
C(3)	4*e*	0.4345(5)	0.8484(8)	0.0253(5)	0.071(5)	0.068(4)	0.064(4)	0.001(4)	0.032(4)	0.005(4)
C(4)	4*e*	0.3537(5)	0.8224(8)	–0.0507(6)	0.066(5)	0.088(6)	0.060(4)	0.010(4)	0.027(4)	0.009(4)
C(5)	4*e*	0.3229(5)	0.7008(9)	–0.0670(6)	0.048(4)	0.120(7)	0.061(4)	–0.005(4)	0.021(3)	0.000(5)
C(6)	4*e*	0.3707(5)	0.6034(8)	–0.0087(5)	0.059(4)	0.085(5)	0.062(4)	–0.012(4)	0.029(4)	–0.005(4)
C(7)	4*e*	0.4552(4)	0.6254(6)	0.0705(5)	0.053(3)	0.064(4)	0.057(4)	–0.006(3)	0.039(3)	–0.006(3)
C(8)	4*e*	0.5099(4)	0.5307(6)	0.1362(5)	0.051(3)	0.058(4)	0.057(4)	–0.008(3)	0.036(3)	–0.008(3)
C(9)	4*e*	0.4871(5)	0.4021(7)	0.1281(6)	0.060(4)	0.065(4)	0.071(4)	–0.015(3)	0.042(4)	–0.013(4)
C(10)	4*e*	0.5423(5)	0.3148(7)	0.1936(6)	0.083(5)	0.049(4)	0.099(6)	–0.009(4)	0.063(5)	–0.008(4)
C(11)	4*e*	0.6216(5)	0.3509(7)	0.2699(6)	0.071(5)	0.053(4)	0.083(5)	0.007(3)	0.045(4)	0.004(4)
C(12)	4*e*	0.6458(5)	0.4747(6)	0.2806(5)	0.056(4)	0.056(4)	0.071(4)	0.001(3)	0.036(3)	–0.005(3)
C(13)	4*e*	0.5926(4)	0.5648(6)	0.2152(5)	0.054(3)	0.050(3)	0.060(4)	–0.003(3)	0.039(3)	–0.004(3)
C(14)	4*e*	0.8408(4)	0.6165(6)	0.2675(5)	0.046(3)	0.050(3)	0.058(4)	0.004(3)	0.025(3)	0.007(3)
C(15)	4*e*	0.9059(4)	0.5613(6)	0.2443(5)	0.051(3)	0.047(3)	0.062(4)	0.004(3)	0.029(3)	0.007(3)
C(16)	4*e*	0.8830(5)	0.5084(6)	0.1482(5)	0.056(4)	0.061(4)	0.062(4)	–0.007(3)	0.027(3)	–0.006(3)
C(17)	4*e*	0.9457(5)	0.4515(7)	0.1260(6)	0.068(5)	0.066(4)	0.081(5)	–0.001(4)	0.041(4)	–0.019(4)
C(18)	4*e*	1.0337(5)	0.4480(8)	0.2002(7)	0.077(5)	0.068(5)	0.098(6)	0.014(4)	0.053(5)	–0.004(4)
C(19)	4*e*	1.0559(5)	0.5007(7)	0.2923(6)	0.053(4)	0.064(4)	0.086(5)	0.009(3)	0.037(4)	0.002(4)
C(20)	4*e*	0.9927(4)	0.5598(6)	0.3168(5)	0.047(3)	0.042(3)	0.062(4)	0.002(3)	0.025(3)	0.009(3)
C(21)	4*e*	1.0127(4)	0.6162(5)	0.4136(5)	0.045(3)	0.042(3)	0.062(4)	0.003(2)	0.025(3)	0.010(3)
C(22)	4*e*	1.0968(4)	0.6221(7)	0.4924(6)	0.048(4)	0.059(4)	0.080(5)	0.002(3)	0.025(3)	0.001(4)
C(23)	4*e*	1.1113(5)	0.6776(7)	0.5834(6)	0.058(4)	0.059(4)	0.072(5)	–0.001(3)	0.011(4)	0.006(4)
C(24)	4*e*	1.0426(5)	0.7308(7)	0.5970(5)	0.073(5)	0.063(4)	0.056(4)	–0.007(4)	0.018(4)	–0.006(3)
C(25)	4*e*	0.9581(5)	0.7278(6)	0.5195(5)	0.058(4)	0.056(4)	0.059(4)	0.001(3)	0.024(3)	–0.003(3)
C(26)	4*e*	0.9425(4)	0.6717(5)	0.4292(5)	0.046(3)	0.039(3)	0.057(4)	0.002(2)	0.021(3)	0.006(3)

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