Crystal structure of tri-(1,4-bis(imidazol-1-yl)butane)-cadmium(II) dihexafluorophosphate, $\{[Cd(bimb)_3](PF_6)_2\}_n$, $C_{30}H_{42}CdF_{12}N_{12}P_2$

Xia Zhu*, Ying Guo, Jian-Gang Li and Kun Yang

Science College, Civil Aviation University of China, Tianjin 300300, P. R. China

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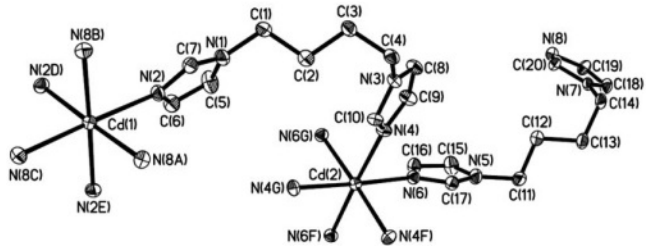

Abstract

$C_{30}H_{42}CdF_{12}N_{12}P_2$, trigonal, *P*3 (no. 143), a = 13.318(2) Å, c = 13.278(2) Å, V = 2039.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.0353, $wR_{ref}(F^2)$ = 0.0954, T = 193 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.30×0.35×0.36 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	7.08 cm^{-1}
Diffractometer, scan mode:	Rigaku Mercury CCD, ω
$2\theta_{max}$:	50.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20052, 4966
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4605
$N(param)_{refined}$:	355
Programs:	SHELX [14]

Source of material

A 20 ml H_2O solution of $Cd(NO_3)_2{\cdot}4H_2O$ (0.154 g, 0.5 mmol) and KPF_6 (0.184 g, 1.0 mmol) was added to one leg of an "H-shaped" tube, and a 20 ml MeOH solution of *bimb* (1,4-bis(imidazol-1-yl)butane, 0.285 g, 1.5 mmol) was added to the other leg of the tube. After three weeks, the well-shaped colourless single crystals were obtained. Yield: 57%. **Elemental analysis**, found (%): C, 36.86; H, 4.24; N, 17.16; calcd. for $C_{30}H_{42}CdF_{12}N_{12}P_2$: C, 37.03; H, 4.35; N, 17.28%. **IR** data (cm^{-1}): 3157*w*, 1520*m*, 1466*w*, 1405*w*, 1273*w*, 1235*m*, 1088*m*, 934*w*, 841*vs*, 749*m*, 664*w*, 556*w*.

Experimental details

Two of the four crystallographic independant PF_6^- counter anions suffer from a disorder.

Discussion

In recent years, the design and synthesis of metal-organic frameworks (MOFs) with well-regulated architectures have caused increasing attention as they can be carried out as functional materials with potential applications in such fields as nonlinear optics, magnetism, molecular separation, catalysis, luminescence, and gas sorption [1-6]. The ligands and metal centers both are the keys to the design and construction of metal-organic frameworks (MOFs) with fascinating topology and physicochemical properties [7,8]. Flexible ligands are employed in the construction of metal-organic frameworks (MOFs) with variety of architectures and topologies because flexible ligands can adopt different conformations according to the geometric needs of the different metal ions [9,10]. The long flexible ligand 1,4-bis(imidazol-1-yl)butane (*bimb*) can adopt various conformations with respect to the relative orientations of the CH_2 groups. Metal-organic frameworks (MOFs) containing *bimb* ligand exhibit a variety of architectures [11,12]. In this paper, we report on a new coordination polymer $\{[Cd(bimb)_3](PF_6)_2\}_n$ based on the flexible ligand 1,4-bis(imidazol-1-yl)butane (*bimb*) and the cadmium metal center. There are two crystallographically independent Cd(II) atoms. Both Cd(1) and Cd(2) atoms are located on the threefold axis and each of them is coordinated by six imidazole nitrogen atoms from six *bimb* ligands. There are two crystallographically independent *bimb* ligands. All *bimb* ligands exhibit the *anti-anti-gauche* conformation. The torsion angles C1–C4 and C11–C14 of the butane chains of two independent *bimb* ligands are 175.4(4) and –174.6(4)°, respectively. The dihedral angles between two imidazole ring planes are 23.7° for N1/N2/C5-C7 and N3/N4/C8-C10 and 23.0° for N5/N6/C15-C17 and N7/N8/C18/C20 imidazole ring planes. The butane chains C1-C4 and C11-C14 are both well coplanar with the mean deviation from the plane 0.0282 and 0.0330 Å, respectively. The planes of butane chains C1-C4 and C11-C14 acutely inclined, by 65.9 and 63.3°, to N1/N2/C5-C7 and N3/N4/C8-C10, 66.6 and 63.7° to N5/N6/C15-C17 and N7/N8/C18/C20 imidazole ring planes. The Cd–N bond lengths are in the ranges of 2.340(3)-2.374(3) Å and the N–Cd–N bond angles are in the range of 87.78(13)-91.32(12)°. Each *bimb* ligand bridges two Cd(II) atoms and each Cd(II) atom links six Cd(II) atoms via six *bimb* ligands along six directions, resulting the three-dimensional network. This three-dimensional network is completely different from the related threefold interpenetrating three-dimensional network of $\{[Cd(bimb)_3](ClO_4)_2\}_n$ [13]. The Cd–Cd separations separation by *bimb* ligands are 10.160 and 10.157 Å, which is longer than the corresponding Cd–Cd separation (9.0819(2) Å) for $[Cd(bimb)_2(NCS)_2]_n$ in which *bimb* ligands show the *gauche-anti-gauche* conformation [13] and obviously shorter than 14.1666(8)-14.6212(9) Å for $\{[Cd(bimb)_3](ClO_4)_2\}_n$ [13] in which *bimb* ligands show the completely anti (*anti-anti-anti*)conformation.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	3*d*	0.3387	0.4875	0.8072	0.058
H(1B)	3*d*	0.3198	0.5005	0.6898	0.058
H(2A)	3*d*	0.4625	0.4475	0.6513	0.054
H(2B)	3*d*	0.4847	0.4438	0.7695	0.054

* Correspondence author (e-mail: xzhu@cauc.edu.cn)

Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(3A)	3*d*	0.5289	0.6483	0.6602	0.049
H(3B)	3*d*	0.5428	0.6468	0.7799	0.049
H(4A)	3*d*	0.7246	0.7215	0.7126	0.043
H(4B)	3*d*	0.6888	0.5960	0.7557	0.043
H(5A)	3*d*	0.1946	0.2933	0.8769	0.041
H(6A)	3*d*	0.1218	0.0900	0.6404	0.048
H(7A)	3*d*	0.2652	0.2866	0.5866	0.063
H(8A)	3*d*	0.6387	0.4309	0.6221	0.040
H(9A)	3*d*	0.7772	0.6309	0.3839	0.049
H(10A)	3*d*	0.7646	0.7522	0.5169	0.047
H(11A)	3*d*	1.1671	0.6520	0.1896	0.055
H(11B)	3*d*	1.1542	0.6707	0.3071	0.055

Table 2. continued.

Atom	Site	x	y	z	U_{iso}
H(12A)	3*d*	1.1110	0.8185	0.2694	0.053
H(12B)	3*d*	1.1141	0.7963	0.1512	0.053
H(13A)	3*d*	1.3145	0.8759	0.2786	0.051
H(13B)	3*d*	1.3151	0.8634	0.1588	0.051
H(14A)	3*d*	1.2624	1.0216	0.2567	0.044
H(14B)	3*d*	1.3883	1.0584	0.2138	0.044
H(15A)	3*d*	0.9603	0.5276	0.3771	0.041
H(16A)	3*d*	0.7571	0.4547	0.1401	0.050
H(17A)	3*d*	0.9538	0.5982	0.0866	0.060
H(18A)	3*d*	1.0980	0.9728	0.1226	0.038
H(19A)	3*d*	1.2979	1.1114	–0.1158	0.047
H(20A)	3*d*	1.4198	1.1003	0.0175	0.049

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd(1)	1*a*		0	0	0.88423(3)	0.0275(2)	0.0275(2)	0.0238(3)	0.01377(9)	0	0
Cd(2)	1*c*		$\frac{2}{3}$	$\frac{1}{3}$	0.38409(3)	0.0277(2)	0.0277(2)	0.0236(3)	0.01383(9)	0	0
P(1)	1*a*		1	1	0.3758(2)	0.0384(6)	0.0384(6)	0.023(1)	0.0192(3)	0	0
P(2)	1*c*		$\frac{2}{3}$	$\frac{1}{3}$	0.8763(2)	0.0370(6)	0.0370(6)	0.024(1)	0.0185(3)	0	0
P(3)	1*b*		$\frac{1}{3}$	$\frac{2}{3}$	0.9765(2)	0.0556(8)	0.0556(8)	0.028(1)	0.0278(4)	0	0
P(4)	1*b*		$\frac{1}{3}$	$\frac{2}{3}$	0.4763(2)	0.0549(8)	0.0549(8)	0.032(1)	0.0275(4)	0	0
F(1)	3*d*		0.9515(6)	0.8925(6)	0.3083(6)	0.139(4)	0.169(5)	0.194(6)	0.096(4)	–0.107(5)	–0.132(5)
F(2)	3*d*		0.8840(5)	0.9362(6)	0.4306(5)	0.100(4)	0.172(6)	0.201(7)	0.056(4)	0.099(5)	0.100(6)
F(3)	3*d*		0.7797(5)	0.3834(6)	0.9374(6)	0.101(4)	0.154(5)	0.203(7)	0.046(4)	–0.101(5)	–0.006(5)
F(4)	3*d*		0.7263(6)	0.4428(6)	0.8119(6)	0.124(4)	0.165(6)	0.211(7)	0.064(4)	0.028(4)	0.140(5)
F(5)	3*d*	0.67	0.243(2)	0.5429(8)	0.9718(5)	0.177(9)	0.068(4)	0.053(4)	–0.042(5)	0.016(6)	0.004(3)
F(6)	1*b*		$\frac{1}{3}$	$\frac{2}{3}$	1.0975(4)	0.072(2)	0.072(2)	0.028(2)	0.036(1)	0	0
F(7)	1*b*		$\frac{1}{3}$	$\frac{2}{3}$	0.8594(4)	0.098(3)	0.098(3)	0.014(3)	0.049(1)	0	0
F(8)	3*d*	0.67	0.2046(7)	0.628(2)	0.9833(5)	0.071(5)	0.41(2)	0.041(4)	0.087(9)	–0.003(3)	0.030(9)
F(9)	1*b*		$\frac{1}{3}$	$\frac{2}{3}$	0.5980(4)	0.069(2)	0.069(2)	0.030(2)	0.0347(9)	0	0
F(10)	1*b*		$\frac{1}{3}$	$\frac{2}{3}$	0.3583(4)	0.089(3)	0.089(3)	0.011(3)	0.044(1)	0	0
F(11)	3*d*	0.67	0.216(1)	0.668(2)	0.4845(5)	0.167(9)	0.45(2)	0.049(4)	0.24(1)	–0.004(5)	–0.007(8)
F(12)	3*d*	0.67	0.338(2)	0.781(1)	0.4704(5)	0.38(2)	0.124(6)	0.058(4)	0.19(1)	–0.040(8)	–0.021(5)
N(1)	3*d*		0.2559(3)	0.3339(3)	0.7338(3)	0.038(2)	0.031(2)	0.050(2)	0.014(2)	0.013(2)	0.006(2)
N(2)	3*d*		0.1277(3)	0.1590(3)	0.7815(3)	0.030(2)	0.029(2)	0.037(2)	0.011(2)	0.004(2)	0.008(2)
N(3)	3*d*		0.6962(3)	0.6043(3)	0.6042(3)	0.035(2)	0.024(2)	0.030(2)	0.012(2)	–0.000(2)	–0.000(1)
N(4)	3*d*		0.7046(3)	0.4946(3)	0.4844(3)	0.043(2)	0.031(2)	0.029(2)	0.021(2)	0.001(2)	–0.001(1)
N(5)	3*d*		1.0011(3)	0.5896(3)	0.2333(3)	0.031(2)	0.039(2)	0.053(2)	0.014(2)	0.006(2)	0.011(2)
N(6)	3*d*		0.8242(3)	0.4609(3)	0.2817(3)	0.031(2)	0.032(2)	0.033(2)	0.014(2)	0.004(1)	0.001(2)
N(7)	3*d*		1.2709(3)	1.0294(3)	0.1046(3)	0.025(2)	0.027(2)	0.031(2)	0.009(1)	–0.004(1)	–0.002(1)
N(8)	3*d*		1.1598(3)	1.0363(3)	–0.0164(3)	0.032(2)	0.038(2)	0.029(2)	0.016(2)	–0.001(1)	–0.000(2)
C(1)	3*d*		0.3408(4)	0.4582(4)	0.7392(4)	0.041(2)	0.031(2)	0.073(3)	0.018(2)	0.015(2)	0.006(2)
C(2)	3*d*		0.4616(4)	0.4815(4)	0.7174(4)	0.040(2)	0.037(2)	0.056(3)	0.018(2)	–0.003(2)	–0.005(2)
C(3)	3*d*		0.5487(4)	0.6119(3)	0.7159(4)	0.037(2)	0.030(2)	0.050(3)	0.013(2)	0.005(2)	–0.007(2)
C(4)	3*d*		0.6715(3)	0.6374(4)	0.7026(3)	0.041(2)	0.028(2)	0.034(2)	0.014(2)	–0.001(2)	–0.010(2)
C(5)	3*d*		0.1926(3)	0.2664(3)	0.8103(3)	0.035(2)	0.035(2)	0.031(2)	0.017(2)	0.007(2)	0.002(2)
C(6)	3*d*		0.1542(5)	0.1571(5)	0.6819(4)	0.044(3)	0.041(3)	0.031(3)	0.019(2)	0.011(2)	0.002(2)
C(7)	3*d*		0.2325(5)	0.2639(5)	0.6520(4)	0.052(3)	0.051(3)	0.033(2)	0.010(2)	0.008(2)	–0.002(2)
C(8)	3*d*		0.6729(5)	0.4962(4)	0.5786(4)	0.042(3)	0.028(2)	0.034(3)	0.022(2)	0.000(2)	0.001(2)
C(9)	3*d*		0.7475(4)	0.6052(4)	0.4497(3)	0.058(3)	0.037(2)	0.028(2)	0.024(2)	0.000(2)	0.006(2)
C(10)	3*d*		0.7415(4)	0.6724(4)	0.5223(4)	0.048(3)	0.027(2)	0.041(2)	0.016(2)	0.005(2)	0.008(2)
C(11)	3*d*		1.1251(4)	0.6731(4)	0.2390(4)	0.033(2)	0.040(2)	0.065(3)	0.019(2)	0.006(2)	0.013(2)
C(12)	3*d*		1.1484(4)	0.7954(4)	0.2171(4)	0.034(2)	0.041(2)	0.055(3)	0.017(2)	–0.004(2)	–0.002(2)
C(13)	3*d*		1.2791(3)	0.8827(4)	0.2152(4)	0.028(2)	0.040(2)	0.052(3)	0.011(2)	–0.007(2)	0.008(2)
C(14)	3*d*		1.3043(4)	1.0052(3)	0.2035(3)	0.027(2)	0.038(2)	0.036(2)	0.010(2)	–0.010(2)	–0.001(2)
C(15)	3*d*		0.9333(3)	0.5259(3)	0.3106(3)	0.033(2)	0.035(2)	0.033(2)	0.016(2)	0.002(2)	0.007(2)
C(16)	3*d*		0.8239(5)	0.4873(5)	0.1821(4)	0.035(3)	0.044(3)	0.036(3)	0.013(2)	0.002(2)	0.014(2)
C(17)	3*d*		0.9312(5)	0.5657(5)	0.1521(4)	0.049(3)	0.053(3)	0.033(2)	0.013(2)	0.000(2)	0.009(2)
C(18)	3*d*		1.1630(4)	1.0062(4)	0.0786(4)	0.025(2)	0.038(3)	0.034(3)	0.017(2)	0.002(2)	0.002(2)
C(19)	3*d*		1.2719(4)	1.0811(4)	–0.0502(3)	0.032(2)	0.059(3)	0.032(2)	0.026(2)	0.006(2)	0.001(2)
C(20)	3*d*		1.3397(4)	1.0761(4)	0.0226(4)	0.027(2)	0.047(3)	0.045(3)	0.016(2)	0.008(2)	–0.001(2)

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