Crystal structure of bis(11-fluoro-dipyrido[3,2-*a*:2′,3′-*c*]phenazine)-adpiatocadmium(II), $Cd_2(C_{18}H_9N_4F)_2(C_6H_8O_4)_2$

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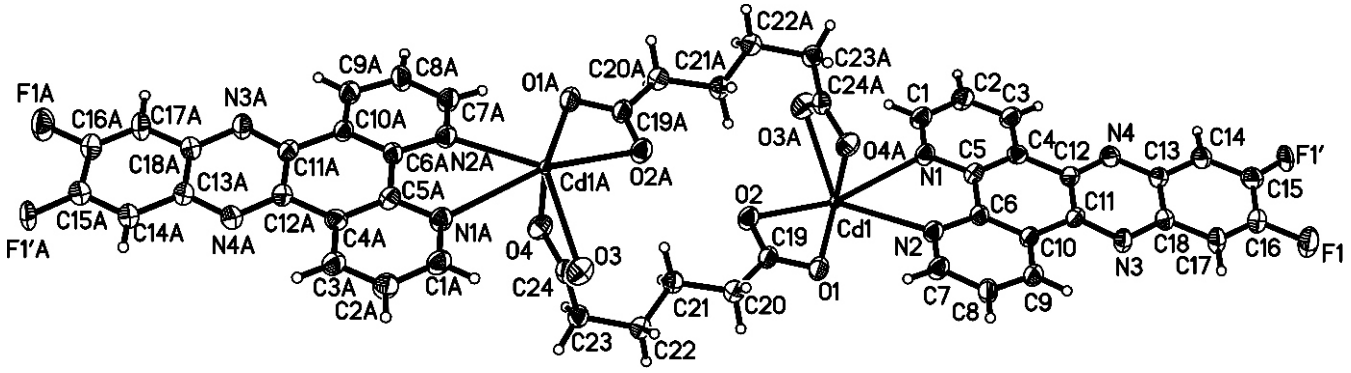

Abstract

$C_{48}H_{34}Cd_2F_2N_8O_8$, triclinic, $P\bar{1}$ (no. 2), a = 8.882(2) Å, b = 9.831(2) Å, c = 12.965(2) Å, α = 81.616(2)°, β = 80.166(2)°, γ = 73.902(2)°, V = 1065.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.059, $wR_{ref}(F^2)$ = 0.197, T = 293 K.

Source of material

The pH value of a mixture of $CdCl_2 \cdot 2.5H_2O$ (0.5 mmol), adipic acid (H_2adp, 0.5 mmol) and 11-fluoro-dipyrido[3,2-*a*:2',3'-c]phenazine (L, 0.5 mmol) in 12 mL distilled water was adjusted between 5 and 6 by addition of triethylamine. The resultant solution was heated at 450 K in a Teflon-lined stainless steel autoclave for five days. The reaction system was then slowly cooled to room temperature. Pale yellow crystals of the title compound suitable for single crystal X-ray diffraction analysis were collected from the final reaction system by filtration, washed several times with distilled water and dried in air at ambient temperature (33 % yield based on Cd(II)).

Experimental details

All H atoms were positioned geometrically d(C—H) = 0.93 Å or 0.97 Å and treated as riding with U_{iso}(H) = 1.2 U_{eq}(C). The disordered F atoms were refined using two split sites F1 and F1' with a total occupancy of 1. The hydrogen atoms attached to C15 and C15A were not generated geometrically because of the disordered F atoms.

Discussion

So far, the coordination chemistry of the rigid aromatic polycarboxylates, such as benzene-1,4-dicarboxylate, benzene-1,3,5-tricarboxylate, and benzene-1,2,4,5-tetracarboxylate, has been widely studied [1]. In contrast to rigid carboxylates, the coordination complexes constructed from flexible carboxylates are relatively underinvestigated.

the central Cd(II) atom is six-coordinated by two nitrogen atoms from one L ligand, and four carboxylate oxygen atoms from two different adp anions in a distorted octahedral environment. The Cd—O and Cd—N bond lengths are in the normal ranges. Two Cd(II) atoms are bridged by two adp anions to form a dimer with d(Cd···Cd) = 9.138 Å.

Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.18 × 0.27 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.74 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD, φ/ω
$2\theta_{max}$:	50.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5522, 3709
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3414
$N(param)_{refined}$:	316
Programs:	SHELXS-97, SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.7225	1.1620	0.5901	0.120
H(2)	2*i*	0.9053	1.2662	0.6339	0.127
H(3)	2*i*	0.8760	1.3278	0.8034	0.119
H(7)	2*i*	0.1385	1.0480	0.9062	0.115
H(8)	2*i*	0.0909	1.1056	1.0752	0.125
H(9)	2*i*	0.2615	1.2025	1.1369	0.111
H(14)	2*i*	0.8853	1.5085	1.0755	0.130
H(17)	2*i*	0.4891	1.3808	1.3171	0.122
H(20A)	2*i*	–0.1017	0.9009	0.7123	0.099
H(20B)	2*i*	0.0085	0.7531	0.7460	0.099

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(21A)	2*i*	0.0678	0.7129	0.5635	0.099
H(21B)	2*i*	–0.0673	0.8508	0.5418	0.099
H(22A)	2*i*	–0.2492	0.7404	0.6586	0.109
H(22B)	2*i*	–0.1126	0.6004	0.6668	0.109
H(23A)	2*i*	–0.1002	0.5916	0.4808	0.101
H(23B)	2*i*	–0.2600	0.5711	0.5457	0.101

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}
C(1)	2*i*		0.711(2)	1.186(2)	0.658(1)	0.10(1)	0.12(1)	0.070(8)	–0.04(1)	0.004(8)	–0.013(8)
C(2)	2*i*		0.822(2)	1.250(2)	0.684(1)	0.09(1)	0.15(2)	0.09(1)	–0.05(1)	0.004(8)	–0.01(1)
C(3)	2*i*		0.804(2)	1.287(2)	0.784(1)	0.10(1)	0.13(1)	0.075(9)	–0.05(1)	–0.009(8)	–0.003(8)
C(4)	2*i*		0.676(2)	1.263(2)	0.856(1)	0.077(8)	0.089(8)	0.083(8)	–0.034(7)	–0.011(6)	0.001(7)
C(5)	2*i*		0.577(2)	1.194(2)	0.826(1)	0.078(8)	0.092(9)	0.066(7)	–0.019(7)	–0.009(6)	–0.011(6)
C(6)	2*i*		0.437(2)	1.166(1)	0.901(1)	0.077(7)	0.087(8)	0.075(8)	–0.032(6)	–0.013(6)	–0.010(6)
C(7)	2*i*		0.210(2)	1.085(2)	0.930(1)	0.090(9)	0.14(1)	0.072(8)	–0.049(9)	0.000(7)	–0.015(8)
C(8)	2*i*		0.180(2)	1.121(2)	1.031(1)	0.12(1)	0.13(1)	0.09(1)	–0.07(1)	0.008(8)	–0.039(9)
C(9)	2*i*		0.282(2)	1.179(2)	1.068(1)	0.11(1)	0.11(1)	0.068(8)	–0.045(9)	0.001(7)	–0.020(7)
C(10)	2*i*		0.416(2)	1.201(2)	1.003(1)	0.079(8)	0.096(9)	0.062(7)	–0.033(7)	–0.007(6)	–0.010(6)
C(11)	2*i*		0.517(2)	1.272(2)	1.037(1)	0.091(9)	0.092(9)	0.060(7)	–0.027(7)	–0.011(6)	–0.007(6)
C(12)	2*i*		0.650(2)	1.303(2)	0.965(1)	0.086(8)	0.088(8)	0.077(8)	–0.032(7)	–0.023(7)	–0.005(6)
C(13)	2*i*		0.711(2)	1.410(2)	1.092(1)	0.11(1)	0.089(9)	0.079(9)	–0.034(8)	–0.028(8)	–0.007(7)
C(14)	2*i*		0.803(2)	1.484(2)	1.123(1)	0.14(1)	0.11(1)	0.10(1)	–0.06(1)	–0.04(1)	–0.001(9)
C(15)	2*i*		0.773(3)	1.525(2)	1.226(2)	0.15(2)	0.11(1)	0.11(1)	–0.06(1)	–0.05(1)	0.01(1)
C(16)	2*i*		0.644(2)	1.484(2)	1.296(1)	0.13(1)	0.11(1)	0.09(1)	–0.04(1)	–0.023(9)	–0.024(9)
C(17)	2*i*		0.565(2)	1.411(2)	1.267(1)	0.11(1)	0.12(1)	0.10(1)	–0.04(1)	–0.018(9)	–0.042(9)
C(18)	2*i*		0.585(2)	1.373(2)	1.164(1)	0.093(9)	0.089(9)	0.086(9)	–0.033(7)	–0.019(7)	–0.013(7)
C(19)	2*i*		0.124(2)	0.894(2)	0.681(1)	0.097(9)	0.11(1)	0.065(8)	–0.036(8)	–0.009(7)	–0.017(7)
C(20)	2*i*		–0.005(2)	0.830(2)	0.690(1)	0.074(8)	0.095(9)	0.081(8)	–0.022(7)	–0.009(6)	–0.012(7)
C(21)	2*i*		–0.032(2)	0.772(2)	0.594(1)	0.077(8)	0.10(1)	0.077(8)	–0.029(7)	–0.006(6)	–0.017(7)
C(22)	2*i*		–0.154(2)	0.685(2)	0.621(1)	0.10(1)	0.10(1)	0.079(8)	–0.036(8)	–0.022(7)	–0.005(7)
C(23)	2*i*		–0.196(2)	0.639(2)	0.523(1)	0.088(9)	0.084(8)	0.082(8)	–0.018(7)	–0.028(7)	–0.008(7)
C(24)	2*i*		–0.283(2)	0.759(2)	0.461(1)	0.073(7)	0.099(9)	0.079(8)	–0.031(7)	0.000(6)	–0.031(7)
N(1)	2*i*		0.590(1)	1.158(1)	0.7267(9)	0.084(7)	0.107(9)	0.072(7)	–0.029(6)	0.000(6)	–0.019(6)
N(2)	2*i*		0.341(1)	1.102(1)	0.8642(9)	0.090(7)	0.112(9)	0.072(7)	–0.044(7)	–0.005(6)	–0.012(6)
N(3)	2*i*		0.492(2)	1.301(1)	1.138(1)	0.098(8)	0.108(9)	0.081(8)	–0.035(7)	–0.014(6)	–0.028(6)
N(4)	2*i*		0.740(2)	1.374(1)	0.991(1)	0.097(8)	0.097(8)	0.093(8)	–0.039(7)	–0.024(6)	–0.005(6)
O(1)	2*i*		0.160(1)	0.929(1)	0.7640(8)	0.121(8)	0.131(8)	0.074(6)	–0.054(7)	–0.012(5)	–0.021(5)
O(2)	2*i*		0.211(1)	0.911(1)	0.5914(8)	0.100(7)	0.147(9)	0.073(6)	–0.061(7)	–0.002(5)	–0.016(6)
O(3)	2*i*		–0.412(1)	0.846(1)	0.4995(9)	0.092(7)	0.127(9)	0.094(7)	–0.001(6)	0.008(6)	–0.026(6)
O(4)	2*i*		–0.227(1)	0.789(1)	0.3620(8)	0.093(7)	0.121(8)	0.078(6)	–0.019(6)	0.005(5)	–0.017(5)
F(1)	2*i*	0.75	0.624(2)	1.520(2)	1.397(1)	0.16(1)	0.19(2)	0.11(1)	–0.07(1)	–0.004(9)	–0.06(1)
Cd(1)	2*i*		0.40778(5)	0.99155(5)	0.67527(3)	0.0318(3)	0.0404(4)	0.0258(3)	–0.0120(2)	–0.0028(2)	–0.0075(2)
F(1')	2*i*	0.25	0.896(5)	1.576(4)	1.242(3)	0.18(4)	0.12(3)	0.12(3)	–0.07(3)	–0.06(3)	–0.03(2)

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References

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