Crystal structure of [2-(2-chloro-6-fluorophenyl)-1*H*-imidazo[4,5-*f*][1,10]phenanthroline]-fumaratocadmium(II) hemihydrate, $Cd_2(C_{19}H_{10}N_4FCl)_2(C_4H_2O_4)_2 \cdot 0.5H_2O$

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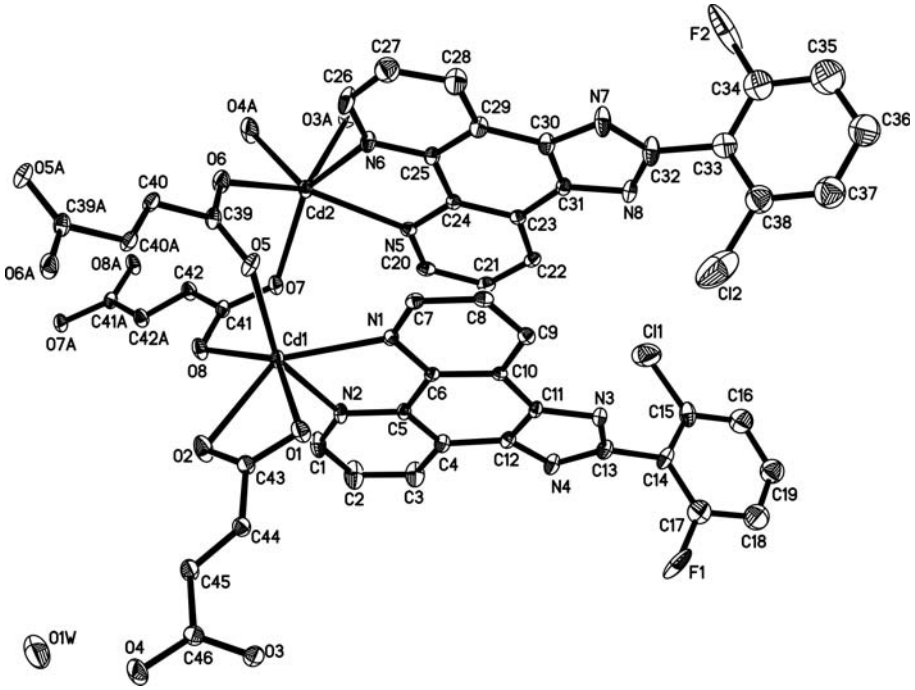

Abstract

$C_{92}H_{50}Cd_4Cl_4F_4N_{16}O_{17}$, monoclinic, $P12_1/c1$ (no. 14), a = 12.2949(8) Å, b = 30.399(1) Å, c = 13.1049(9) Å, β = 109.963(7)°, V = 4603.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.052, $wR_{ref}(F^2)$ = 0.164, T = 293 K.

Source of material

The pH value of a mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.5 mmol), fumaric acid (0.5 mmol) and 2-(2-chloro-6-fluoro-phenyl)-1*H*-imidazo[4,5-*f*][1,10]phenanthroline (L, 1 mmol) in 10 mL distilled water was adjusted between 5 and 6 by addition of triethylamine. The resultant solution was heated at 460 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 (19 % yield based on Cd(II)).

Experimental details

All H atoms were positioned geometrically d(N—H) = 0.86 Å and d(C—H) = 0.93 Å and treated as riding with U_{iso}(H) = 1.2 U_{eq}(carrier). The hydrogen atoms of O1W were not located from the difference Fourier map.

Discussion

The design and synthesis of coordination polymers are currently attracting considerable attention in view of their interesting structural topologies and properties [1]. In recent years, the coordination chemistry of 1,10-phenanthroline derivatives has received intense interest [1].

In the title crystal structure, each Cd(II) atom is six-coordinated by two nitrogen atoms from one L ligand, and four carboxylate oxygen atoms from three different fumarate ligands in a distorted octahedral manner with d(Cd—N) = 2.306 - 2.411 Å and d(Cd—O) = 2.224 - 2.500 Å. The fumarate ligands connect neighboring Cd(II) atoms to generate a two-dimensional layer structure. The L ligands are located on both sides of the layers.

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Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.20 × 0.24 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	11.12 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20600, 9393
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5942
$N(param)_{refined}$:	610
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)	4*e*	1.0560	0.0961	0.2338	0.071
H(2)	4*e*	1.2139	0.1423	0.2791	0.092
H(3)	4*e*	1.1854	0.2168	0.2559	0.077
H(7)	4*e*	0.5481	0.1593	0.0204	0.041
H(8)	4*e*	0.4954	0.2325	0.0021	0.043
H(9)	4*e*	0.6364	0.2865	0.0544	0.039
H(16)	4*e*	1.0838	0.4406	–0.0140	0.105
H(18)	4*e*	1.1234	0.4663	0.2897	0.116
H(19)	4*e*	1.1318	0.4857	0.1290	0.105
H(20)	4*e*	0.9302	0.1653	–0.0830	0.045
H(21)	4*e*	0.9698	0.2395	–0.0729	0.046
H(22)	4*e*	0.8181	0.2891	–0.1301	0.040
H(26)	4*e*	0.4340	0.0875	–0.2666	0.078
H(27)	4*e*	0.2712	0.1288	–0.3102	0.087
H(28)	4*e*	0.2831	0.2044	–0.3109	0.082
H(35)	4*e*	0.2639	0.4368	–0.4484	0.172
H(36)	4*e*	0.2771	0.4777	–0.2952	0.140
H(37)	4*e*	0.3755	0.4497	–0.1238	0.136
H(40)	4*e*	0.4891	–0.0170	–0.1000	0.051
H(42)	4*e*	0.9681	0.0226	–0.0956	0.050
H(44)	4*e*	0.7089	0.1185	0.4575	0.049
H(45)	4*e*	0.7961	0.0367	0.5156	0.061
H(3A)	4*e*	0.8192	0.3365	0.1147	0.042
H(8A)	4*e*	0.6288	0.3340	–0.1975	0.051

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)	4*e*		1.0434(6)	0.1262(2)	0.2226(6)	0.051(4)	0.036(3)	0.096(6)	0.014(3)	0.032(4)	0.014(4)
C(2)	4*e*		1.1395(6)	0.1538(2)	0.2511(8)	0.035(4)	0.058(5)	0.134(8)	0.013(3)	0.025(4)	0.012(5)
C(3)	4*e*		1.1227(5)	0.1976(2)	0.2372(7)	0.033(3)	0.053(4)	0.103(6)	0.001(3)	0.020(4)	0.006(4)
C(4)	4*e*		1.0104(5)	0.2135(2)	0.1946(5)	0.035(3)	0.035(3)	0.048(4)	0.000(2)	0.018(3)	–0.003(3)
C(5)	4*e*		0.9177(4)	0.1836(2)	0.1652(4)	0.038(3)	0.026(3)	0.034(3)	0.001(2)	0.019(3)	0.001(2)
C(6)	4*e*		0.7985(4)	0.1989(2)	0.1176(4)	0.039(3)	0.030(3)	0.021(3)	–0.002(2)	0.016(2)	–0.002(2)
C(7)	4*e*		0.6055(4)	0.1807(2)	0.0423(4)	0.029(3)	0.039(3)	0.033(3)	–0.006(2)	0.011(2)	–0.003(2)
C(8)	4*e*		0.5732(5)	0.2248(2)	0.0308(4)	0.031(3)	0.048(3)	0.029(3)	0.001(2)	0.012(2)	–0.002(3)
C(9)	4*e*		0.6567(4)	0.2569(2)	0.0620(4)	0.037(3)	0.038(3)	0.028(3)	0.005(2)	0.017(2)	0.002(2)
C(10)	4*e*		0.7729(4)	0.2442(2)	0.1055(4)	0.034(3)	0.030(3)	0.019(2)	–0.001(2)	0.016(2)	0.000(2)
C(11)	4*e*		0.8695(5)	0.2735(2)	0.1383(4)	0.042(3)	0.023(3)	0.034(3)	–0.003(2)	0.022(3)	–0.002(2)
C(12)	4*e*		0.9816(5)	0.2591(2)	0.1766(5)	0.036(3)	0.032(3)	0.040(3)	–0.004(2)	0.017(3)	–0.005(2)
C(13)	4*e*		0.9914(5)	0.3285(2)	0.1669(5)	0.041(3)	0.037(3)	0.057(4)	–0.005(3)	0.021(3)	–0.007(3)
C(14)	4*e*		1.0308(5)	0.3741(2)	0.1583(6)	0.046(4)	0.036(3)	0.075(5)	–0.009(3)	0.027(4)	–0.020(3)
C(15)	4*e*		1.0391(6)	0.3891(2)	0.0633(7)	0.056(4)	0.040(4)	0.091(6)	0.000(3)	0.036(4)	0.013(4)
C(16)	4*e*		1.0781(7)	0.4312(3)	0.0516(8)	0.087(3)	0.088(3)	0.088(3)	–0.001(1)	0.031(1)	0.001(1)
C(17)	4*e*		1.0623(8)	0.4023(3)	0.2458(8)	0.082(3)	0.081(3)	0.082(3)	–0.001(1)	0.028(1)	–0.001(1)
C(18)	4*e*		1.1019(8)	0.4464(3)	0.2323(8)	0.097(3)	0.095(3)	0.097(3)	–0.001(1)	0.033(1)	–0.002(1)
C(19)	4*e*		1.1066(8)	0.4574(3)	0.1369(8)	0.088(3)	0.086(3)	0.089(3)	–0.001(1)	0.030(1)	0.000(1)
C(20)	4*e*		0.8689(5)	0.1850(2)	–0.1074(4)	0.037(3)	0.052(4)	0.025(3)	0.005(3)	0.013(3)	–0.002(3)
C(21)	4*e*		0.8937(5)	0.2297(2)	–0.1003(5)	0.040(3)	0.048(3)	0.030(3)	–0.010(3)	0.018(3)	0.000(3)
C(22)	4*e*		0.8037(5)	0.2590(2)	–0.1346(4)	0.040(3)	0.036(3)	0.028(3)	–0.010(2)	0.018(3)	–0.005(2)
C(23)	4*e*		0.6905(4)	0.2433(2)	–0.1763(4)	0.036(3)	0.035(3)	0.023(3)	0.002(2)	0.015(2)	–0.001(2)
C(24)	4*e*		0.6730(4)	0.1975(2)	–0.1827(4)	0.035(3)	0.029(3)	0.025(3)	0.000(2)	0.017(2)	0.002(2)
C(25)	4*e*		0.5567(5)	0.1785(2)	–0.2239(5)	0.037(3)	0.031(3)	0.040(3)	0.000(2)	0.018(3)	0.000(2)
C(26)	4*e*		0.4414(6)	0.1180(2)	–0.2622(7)	0.049(4)	0.031(3)	0.115(7)	–0.008(3)	0.028(4)	–0.009(4)
C(27)	4*e*		0.3430(7)	0.1425(3)	–0.2902(7)	0.072(2)	0.072(2)	0.075(2)	–0.001(1)	0.024(1)	0.000(1)
C(28)	4*e*		0.3498(6)	0.1873(2)	–0.2889(7)	0.037(4)	0.058(4)	0.105(7)	–0.002(3)	0.019(4)	0.001(4)
C(29)	4*e*		0.4590(5)	0.2067(2)	–0.2538(5)	0.038(3)	0.040(3)	0.060(4)	–0.001(3)	0.020(3)	–0.003(3)
C(30)	4*e*		0.4785(5)	0.2528(2)	–0.2476(5)	0.038(3)	0.035(3)	0.059(4)	0.004(2)	0.025(3)	0.001(3)
C(31)	4*e*		0.5880(5)	0.2699(2)	–0.2117(4)	0.045(3)	0.028(3)	0.034(3)	–0.001(2)	0.021(3)	0.001(2)
C(32)	4*e*		0.4600(6)	0.3218(2)	–0.2542(7)	0.055(4)	0.036(4)	0.110(6)	0.007(3)	0.048(4)	0.004(4)
C(33)	4*e*		0.4135(5)	0.3674(2)	–0.2660(6)	0.073(2)	0.074(2)	0.075(2)	–0.000(1)	0.027(1)	0.001(1)
C(34)	4*e*		0.3545(6)	0.3842(2)	–0.3687(5)	0.108(3)	0.108(3)	0.109(3)	0.001(1)	0.038(2)	0.001(1)
C(35)	4*e*		0.3034(6)	0.4255(2)	–0.3797(5)	0.143(5)	0.143(5)	0.143(5)	0.000(1)	0.050(2)	0.001(1)
C(36)	4*e*		0.3113(6)	0.4501(2)	–0.2879(7)	0.116(4)	0.116(4)	0.117(4)	–0.001(1)	0.041(2)	0.000(1)
C(37)	4*e*		0.3702(6)	0.4333(2)	–0.1852(5)	0.113(4)	0.113(4)	0.115(4)	–0.000(1)	0.039(2)	–0.001(1)
C(38)	4*e*		0.4213(5)	0.3919(2)	–0.1742(4)	0.091(3)	0.091(3)	0.092(3)	–0.001(1)	0.031(1)	–0.000(1)
C(39)	4*e*		0.5872(5)	0.0411(2)	–0.0542(5)	0.045(3)	0.032(3)	0.056(4)	–0.006(3)	0.027(3)	0.003(3)
C(40)	4*e*		0.5146(5)	0.0031(2)	–0.0432(5)	0.056(4)	0.032(3)	0.043(4)	–0.016(3)	0.023(3)	–0.010(3)
C(41)	4*e*		0.9002(5)	0.0461(2)	0.0210(5)	0.038(3)	0.025(3)	0.043(3)	0.008(2)	0.016(3)	–0.003(2)
C(42)	4*e*		0.9692(5)	0.0167(2)	–0.0256(5)	0.056(4)	0.035(3)	0.042(4)	0.010(3)	0.027(3)	0.006(3)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(43)	4*e*		0.7537(5)	0.0872(2)	0.3374(5)	0.053(4)	0.037(3)	0.037(3)	0.008(3)	0.023(3)	0.002(3)
C(44)	4*e*		0.7403(5)	0.0920(2)	0.4453(5)	0.048(4)	0.036(3)	0.039(3)	0.007(3)	0.018(3)	0.001(3)
C(45)	4*e*		0.7666(6)	0.0641(2)	0.5242(5)	0.083(5)	0.035(3)	0.045(4)	0.012(3)	0.038(4)	0.002(3)
C(46)	4*e*		0.7505(5)	0.0750(2)	0.6283(5)	0.056(4)	0.046(4)	0.039(4)	–0.002(3)	0.027(3)	0.001(3)
N(1)	4*e*		0.7142(4)	0.1681(1)	0.0831(3)	0.038(3)	0.027(2)	0.027(2)	–0.005(2)	0.014(2)	–0.001(2)
N(2)	4*e*		0.9364(4)	0.1399(1)	0.1809(4)	0.046(3)	0.025(2)	0.053(3)	0.007(2)	0.024(2)	0.003(2)
N(3)	4*e*		0.8762(4)	0.3183(1)	0.1341(4)	0.036(3)	0.027(2)	0.047(3)	0.000(2)	0.019(2)	–0.001(2)
N(4)	4*e*		1.0580(4)	0.2941(2)	0.1944(5)	0.043(3)	0.034(3)	0.068(4)	–0.004(2)	0.022(3)	–0.008(3)
N(5)	4*e*		0.7629(4)	0.1690(1)	–0.1468(3)	0.038(3)	0.036(2)	0.024(2)	–0.001(2)	0.013(2)	0.000(2)
N(6)	4*e*		0.5454(4)	0.1345(1)	–0.2297(4)	0.037(3)	0.029(2)	0.061(3)	–0.005(2)	0.022(2)	0.002(2)
N(7)	4*e*		0.3984(4)	0.2859(2)	–0.2726(6)	0.044(3)	0.033(3)	0.126(6)	0.008(2)	0.039(3)	0.010(3)
N(8)	4*e*		0.5748(4)	0.3145(1)	–0.2158(4)	0.049(3)	0.026(2)	0.063(4)	–0.001(2)	0.034(3)	–0.002(2)
O(1)	4*e*		0.7175(4)	0.1193(1)	0.2735(3)	0.077(3)	0.047(3)	0.044(3)	0.020(2)	0.038(2)	0.011(2)
O(2)	4*e*		0.7990(5)	0.0563(2)	0.3104(4)	0.135(5)	0.047(3)	0.064(3)	0.040(3)	0.061(3)	0.014(2)
O(1W)	4*e*	0.50	0.901(1)	–0.0262(4)	0.673(1)	0.16(1)	0.084(9)	0.12(1)	0.020(9)	0.04(1)	0.035(8)
O(3)	4*e*		0.7336(4)	0.1138(1)	0.6493(3)	0.066(3)	0.044(2)	0.041(2)	–0.009(2)	0.034(2)	–0.005(2)
O(4)	4*e*		0.7571(5)	0.0454(2)	0.6954(4)	0.143(5)	0.051(3)	0.058(3)	0.003(3)	0.064(3)	0.015(2)
O(5)	4*e*		0.6122(4)	0.0703(1)	0.0150(4)	0.055(3)	0.035(2)	0.068(3)	–0.015(2)	0.029(2)	–0.016(2)
O(6)	4*e*		0.6152(4)	0.0399(1)	–0.1368(4)	0.083(3)	0.038(2)	0.054(3)	–0.012(2)	0.043(3)	0.001(2)
O(7)	4*e*		0.8627(3)	0.0812(1)	–0.0302(3)	0.050(2)	0.029(2)	0.050(3)	0.009(2)	0.019(2)	0.006(2)
O(8)	4*e*		0.8868(4)	0.0366(1)	0.1078(3)	0.080(3)	0.036(2)	0.047(3)	0.016(2)	0.042(2)	0.012(2)
Cd(1)	4*e*		0.77801(4)	0.09382(1)	0.13487(3)	0.0479(3)	0.0244(2)	0.0354(3)	0.0019(2)	0.0250(2)	–0.0010(2)
Cd(2)	4*e*		0.71391(4)	0.09397(1)	–0.18179(3)	0.0489(3)	0.0267(2)	0.0349(3)	0.0016(2)	0.0234(2)	0.0029(2)
Cl(1)	4*e*		1.0128(3)	0.3548(1)	–0.0389(2)	0.148(2)	0.120(2)	0.077(2)	–0.049(2)	0.029(2)	0.005(2)
Cl(2)	4*e*		0.4697(3)	0.3696(2)	–0.0495(4)	0.112(3)	0.212(4)	0.233(5)	0.008(3)	0.032(3)	–0.129(4)
F(1)	4*e*		1.0600(6)	0.3884(2)	0.3493(4)	0.233(7)	0.073(3)	0.109(4)	–0.081(4)	0.134(5)	–0.060(3)
F(2)	4*e*		0.3396(7)	0.3598(2)	–0.4652(7)	0.232(8)	0.209(8)	0.204(7)	0.162(7)	0.164(7)	0.162(7)

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