Crystal structure of (2-(2-chloro-6-fluorophenyl)-1*H*-imidazo[4,5-*f*][1,10]-phenanthroline)(2-(2-chloro-6-fluorophenyl)imidazo[4,5-*f*][1,10]phenanthrolin-1-ide)-dicadmium(II)-fumarate] tetrahydrate, $[Cd(C_{19}H_{10}ClFN_4)(C_{19}H_9ClFN_4)(C_2HO_2)]_2{\cdot}4H_2O$, $C_{80}H_{44}Cd_2Cl_4F_4N_{16}O_8$

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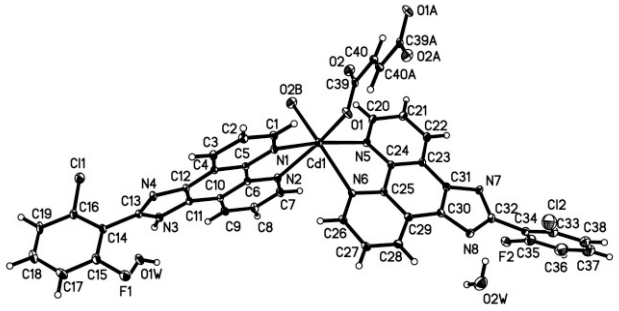

Abstract

$C_{80}H_{44}Cd_2Cl_4F_4N_{16}O_6$, triclinic, $P\bar{1}$ (no. 2), a = 8.316(3) Å, b = 14.081(5) Å, c = 15.541(6) Å, α = 96.045(7)°, β = 94.660(7)°, γ = 91.155(6)°, V = 1803.0 Å^3, Z = 1, $R_{gt}(F)$ = 0.0635, $wR_{ref}(F^2)$ = 0.1718, T = 293 K.

Table 1. Data collection and handling.

Crystal:	pale yellow blocks, size 0.17×0.2×0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.21 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD area detector, φ and ω
$2\theta_{max}$:	52.7°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10255, 6988
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4786
$N(param)_{refined}$:	502
Programs:	SHELXS-97 [5]

Source of material

A mixture of $CdCl_2{\cdot}2H_2O$ (0.5 mmol), H_2fum (0.5 mmol) and HL (0.5 mmol) was suspended in 8 ml of deionized water. The pH value of the mixture was adjusted to about 5-6 by adding triethylamine. The resultant solution was heated at 463 K in a Teflon-lined stainless steel autoclave for 6 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. Yield: 11% based on Cd(II).

Experimental details

All H atoms were positioned geometrically (C–H = 0.93 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier). The water H atoms were located in a difference Fourier map, and were refined with distance restraints of O–H = 0.85±0.01 Å; their temperature factors were tied to those of parent atoms by a factor of 1.5.

Discussion

The design and synthesis of coordination polymers have captured much attention in recent decades not only because of their intriguing variety of topological structural features but also due to their potential applications in some major scientific fields, such as magnetism, chirality, catalysis, and non-linear optical materials [1-3]. In this regard, 1,10-phenanthroline (phen) and its derivatives have been used to build novel supramolecular architectures through its aromatic π-π interactions [1,2]. In this work, we present a new Cd^{II} coordination polymer, $[Cd(HL)(L)(fu)_{0.5}]{\cdot}2H_2O$ (H_2fu = fumaric acid and HL = 2-(2-chloro-6-fluoro- phenyl)-1*H*-imidazo[4,5-f][1,10]phenanthroline).

As shown in the figure , the central Cd^{II} atom is six-coordinated by four nitrogen atoms from one L ligand and one HL ligand, and two carboxylate oxygen atoms from two different fu anions in a distorted octahedral environment. The Cd–O and Cd–N distances are in the normal ranges [4]. The fu ligands bridge neighboring Cd^{II} atoms to yield an one-dimensional chain. The L and HL ligands are located on both sides of the one-dimensional chains. Furthermore, the strong π-π interactions among the L or HL ligands of neighboring chains result in a two-dimensional supramolecular structure.

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.7580	0.2713	0.4207	0.048
H(2)	2*i*	0.7889	0.1122	0.4427	0.056
H(3)	2*i*	0.5704	0.0080	0.4163	0.051
H(7)	2*i*	0.1155	0.4279	0.2591	0.058
H(8)	2*i*	–0.1161	0.3369	0.2273	0.060
H(9)	2*i*	–0.1238	0.1797	0.2546	0.053
H(17)	2*i*	0.0660	–0.3755	0.1988	0.082
H(18)	2*i*	–0.0979	–0.4288	0.2992	0.082
H(19)	2*i*	–0.1795	–0.3252	0.4068	0.077
H(20)	2*i*	0.3743	0.6391	0.4328	0.059
H(21)	2*i*	0.4124	0.7994	0.4079	0.062
H(22)	2*i*	0.5199	0.8319	0.2831	0.061
H(26)	2*i*	0.5431	0.2944	0.1751	0.054
H(27)	2*i*	0.6438	0.3100	0.0436	0.061
H(28)	2*i*	0.7028	0.4580	0.0087	0.060
H(36)	2*i*	0.6132	1.0003	–0.1215	0.145

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(37)	2*i*	0.8806	1.0213	–0.1496	0.146
H(38)	2*i*	1.0758	0.9237	–0.0940	0.138
H(40)	2*i*	0.9595	0.5725	0.5581	0.053
H(3A)	2*i*	–0.0823	0.0034	0.2827	0.052
HW(11)	2*i*	–0.3184	–0.0752	0.1785	0.101
HW(12)	2*i*	–0.2947	0.0209	0.1918	0.101
HW(22)	2*i*	0.9540	0.5694	–0.0373	0.166
HW(21)	2*i*	0.8139	0.5400	–0.0836	0.166

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}
C(1)	2*i*	0.6694(8)	0.2292(4)	0.4088(4)	0.034(4)	0.038(4)	0.047(4)	–0.003(3)	–0.002(3)	0.006(3)
C(2)	2*i*	0.6890(8)	0.1333(5)	0.4225(4)	0.038(4)	0.043(4)	0.059(4)	0.006(3)	–0.003(3)	0.007(3)
C(3)	2*i*	0.5595(8)	0.0715(5)	0.4060(4)	0.047(4)	0.030(3)	0.053(4)	0.005(3)	0.002(3)	0.013(3)
C(4)	2*i*	0.4097(7)	0.1030(4)	0.3736(4)	0.037(3)	0.029(3)	0.036(3)	–0.002(3)	0.000(3)	0.005(2)
C(5)	2*i*	0.4002(7)	0.2003(4)	0.3603(4)	0.033(3)	0.034(3)	0.032(3)	–0.003(3)	0.002(3)	0.005(2)
C(6)	2*i*	0.2472(8)	0.2366(4)	0.3243(4)	0.039(4)	0.026(3)	0.040(3)	0.004(3)	0.003(3)	0.006(2)
C(7)	2*i*	0.1148(9)	0.3648(5)	0.2718(5)	0.051(4)	0.027(3)	0.067(5)	0.003(3)	–0.010(4)	0.014(3)
C(8)	2*i*	–0.0243(8)	0.3100(5)	0.2518(5)	0.033(4)	0.045(4)	0.071(5)	0.003(3)	–0.013(3)	0.019(3)
C(9)	2*i*	–0.0292(8)	0.2166(5)	0.2677(4)	0.036(4)	0.042(4)	0.053(4)	–0.004(3)	–0.006(3)	0.004(3)
C(10)	2*i*	0.1110(7)	0.1768(4)	0.3040(4)	0.037(4)	0.032(3)	0.035(3)	–0.005(3)	0.004(3)	0.006(2)
C(11)	2*i*	0.1251(8)	0.0787(4)	0.3200(4)	0.040(4)	0.031(3)	0.040(3)	–0.005(3)	0.005(3)	0.004(3)
C(12)	2*i*	0.2691(8)	0.0435(4)	0.3522(4)	0.044(4)	0.025(3)	0.042(3)	–0.004(3)	0.004(3)	0.006(3)
C(13)	2*i*	0.1008(8)	–0.0725(4)	0.3268(4)	0.053(4)	0.031(3)	0.040(4)	–0.008(3)	0.004(3)	0.006(3)
C(14)	2*i*	0.0333(9)	–0.1720(5)	0.3179(4)	0.052(4)	0.030(3)	0.052(4)	–0.005(3)	–0.005(3)	0.009(3)
C(15)	2*i*	0.077(1)	–0.2374(5)	0.2521(5)	0.059(5)	0.046(4)	0.068(5)	–0.006(4)	0.000(4)	0.010(4)
C(16)	2*i*	–0.0670(8)	–0.2079(5)	0.3749(4)	0.043(4)	0.043(4)	0.056(4)	–0.006(3)	–0.001(3)	0.014(3)
C(17)	2*i*	0.032(1)	–0.3334(6)	0.2434(6)	0.067(6)	0.046(5)	0.083(6)	0.009(4)	–0.014(5)	–0.012(4)
C(18)	2*i*	–0.066(1)	–0.3648(6)	0.3033(6)	0.056(5)	0.040(5)	0.108(7)	–0.007(4)	–0.008(5)	0.018(5)
C(19)	2*i*	–0.114(1)	–0.3033(6)	0.3671(6)	0.058(5)	0.044(5)	0.093(6)	–0.017(4)	–0.002(5)	0.030(4)
C(20)	2*i*	0.4192(9)	0.6527(5)	0.3825(4)	0.066(5)	0.040(4)	0.047(4)	0.004(3)	0.023(4)	0.010(3)
C(21)	2*i*	0.441(1)	0.7502(5)	0.3678(5)	0.070(5)	0.038(4)	0.050(4)	0.009(4)	0.017(4)	0.004(3)
C(22)	2*i*	0.5043(9)	0.7689(5)	0.2942(5)	0.068(5)	0.027(3)	0.060(4)	0.002(3)	0.015(4)	0.009(3)
C(23)	2*i*	0.5464(8)	0.6953(4)	0.2348(4)	0.044(4)	0.035(4)	0.043(4)	–0.001(3)	0.005(3)	0.007(3)
C(24)	2*i*	0.5241(7)	0.5999(4)	0.2534(4)	0.035(3)	0.032(3)	0.042(4)	0.000(3)	0.001(3)	0.007(3)
C(25)	2*i*	0.5657(7)	0.5195(4)	0.1935(4)	0.034(3)	0.034(3)	0.042(4)	–0.002(3)	–0.002(3)	0.006(3)
C(26)	2*i*	0.5653(8)	0.3552(5)	0.1602(4)	0.051(4)	0.028(3)	0.055(4)	0.001(3)	–0.004(3)	0.003(3)
C(27)	2*i*	0.6261(9)	0.3637(5)	0.0815(5)	0.062(5)	0.031(4)	0.056(4)	0.000(3)	0.001(4)	–0.007(3)
C(28)	2*i*	0.6588(9)	0.4516(5)	0.0610(4)	0.050(4)	0.051(4)	0.047(4)	–0.003(3)	0.005(3)	–0.005(3)
C(29)	2*i*	0.6296(8)	0.5340(5)	0.1150(4)	0.039(4)	0.039(4)	0.046(4)	0.001(3)	–0.002(3)	0.003(3)
C(30)	2*i*	0.6580(8)	0.6307(5)	0.0982(4)	0.041(4)	0.038(4)	0.047(4)	–0.002(3)	0.004(3)	0.002(3)
C(31)	2*i*	0.6194(9)	0.7059(5)	0.1553(4)	0.052(4)	0.035(4)	0.046(4)	–0.002(3)	–0.002(3)	0.010(3)
C(32)	2*i*	0.7217(9)	0.7590(5)	0.0442(5)	0.060(5)	0.043(4)	0.053(4)	–0.008(3)	0.008(4)	0.013(3)
C(33)	2*i*	0.9253(8)	0.8442(5)	–0.0327(4)	0.12(1)	0.084(7)	0.068(6)	–0.030(7)	0.024(6)	–0.005(5)
C(34)	2*i*	0.7651(9)	0.8316(4)	–0.0160(3)	0.114(8)	0.055(5)	0.045(4)	–0.033(5)	0.023(5)	–0.007(4)
C(35)	2*i*	0.6481(8)	0.8901(5)	–0.0492(4)	0.146(7)	0.052(5)	0.065(5)	0.004(5)	0.000(5)	0.023(4)
C(36)	2*i*	0.691(1)	0.9612(4)	–0.0993(4)	0.186(9)	0.082(6)	0.096(7)	0.011(7)	0.011(7)	0.014(5)
C(37)	2*i*	0.852(1)	0.9738(4)	–0.1161(4)	0.183(9)	0.095(7)	0.085(6)	–0.020(7)	0.032(7)	–0.006(6)
C(38)	2*i*	0.9686(9)	0.9153(6)	–0.0828(4)	0.156(8)	0.108(7)	0.079(6)	–0.047(7)	0.047(6)	–0.019(5)
C(39)	2*i*	0.7684(8)	0.5054(5)	0.4775(4)	0.039(4)	0.033(4)	0.053(4)	–0.008(3)	–0.006(3)	0.018(3)
C(40)	2*i*	0.9389(8)	0.5235(5)	0.5133(4)	0.044(4)	0.040(4)	0.047(4)	–0.003(3)	–0.001(3)	0.004(3)
N(1)	2*i*	0.5285(6)	0.2626(4)	0.3792(3)	0.031(3)	0.033(3)	0.047(3)	–0.003(2)	0.000(2)	0.010(2)
N(2)	2*i*	0.2494(7)	0.3307(4)	0.3088(3)	0.042(3)	0.031(3)	0.053(3)	0.001(2)	–0.002(3)	0.013(2)
N(3)	2*i*	0.0167(6)	0.0029(4)	0.3034(3)	0.043(3)	0.037(3)	0.050(3)	–0.006(2)	–0.005(3)	0.006(2)
N(4)	2*i*	0.2528(7)	–0.0529(3)	0.3576(3)	0.043(3)	0.025(3)	0.056(3)	–0.001(2)	0.004(3)	0.007(2)
N(5)	2*i*	0.4600(7)	0.5813(4)	0.3278(3)	0.044(3)	0.035(3)	0.042(3)	0.003(2)	0.002(3)	0.010(2)
N(6)	2*i*	0.5370(6)	0.4305(4)	0.2159(3)	0.041(3)	0.032(3)	0.042(3)	–0.004(2)	–0.005(2)	0.005(2)
N(7)	2*i*	0.6620(8)	0.7900(4)	0.1210(3)	0.064(4)	0.042(3)	0.042(3)	–0.004(3)	0.009(3)	0.012(3)
N(8)	2*i*	0.7247(7)	0.6649(4)	0.0273(3)	0.060(4)	0.046(4)	0.044(3)	–0.002(3)	0.012(3)	0.007(3)
O(1)	2*i*	0.7421(6)	0.4491(3)	0.4103(4)	0.043(3)	0.041(3)	0.085(4)	–0.005(2)	–0.018(3)	–0.003(3)
O(2)	2*i*	0.6616(6)	0.5502(4)	0.5148(4)	0.038(3)	0.098(5)	0.071(4)	0.005(3)	0.014(3)	0.020(3)
O(1W)	2*i*	–0.2859(6)	–0.0274(4)	0.2136(3)	0.064(4)	0.050(3)	0.083(4)	–0.015(3)	–0.012(3)	0.004(3)
O(2W)	2*i*	0.916(1)	0.5401(5)	–0.0858(4)	0.121(6)	0.115(6)	0.101(5)	0.008(5)	0.040(5)	0.015(4)
F(1)	2*i*	0.1726(7)	–0.2053(3)	0.1932(3)	0.106(4)	0.067(3)	0.068(3)	–0.014(3)	0.026(3)	–0.007(2)
F(2)	2*i*	0.4873(6)	0.8795(3)	–0.0396(3)	0.074(3)	0.031(2)	0.075(3)	0.014(2)	–0.008(2)	0.005(2)
Cl(1)	2*i*	–0.1278(3)	–0.1308(2)	0.4596(1)	0.076(2)	0.071(1)	0.069(1)	–0.007(1)	0.018(1)	0.010(1)
Cl(2)	2*i*	1.0595(5)	0.7807(3)	0.0017(3)	0.102(3)	0.176(4)	0.154(3)	–0.008(3)	0.022(2)	0.003(3)
Cd(1)	2*i*	0.47762(6)	0.42439(3)	0.36337(3)	0.0348(3)	0.0286(3)	0.0494(3)	–0.0040(2)	–0.0013(2)	0.0093(2)

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