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Crystal structure of [2-(2-chloro-6-fluorophenyl)-1*H*-imidazo[4,5-*f*]-[1,10]phenanthroline]-(1,3-benzenedicarboxylato)cadmium(II) hydrate, $Cd_2(C_{19}H_{10}N_4FCl)_2(C_8H_4O_4)_2 \cdot 0.25H_2O$

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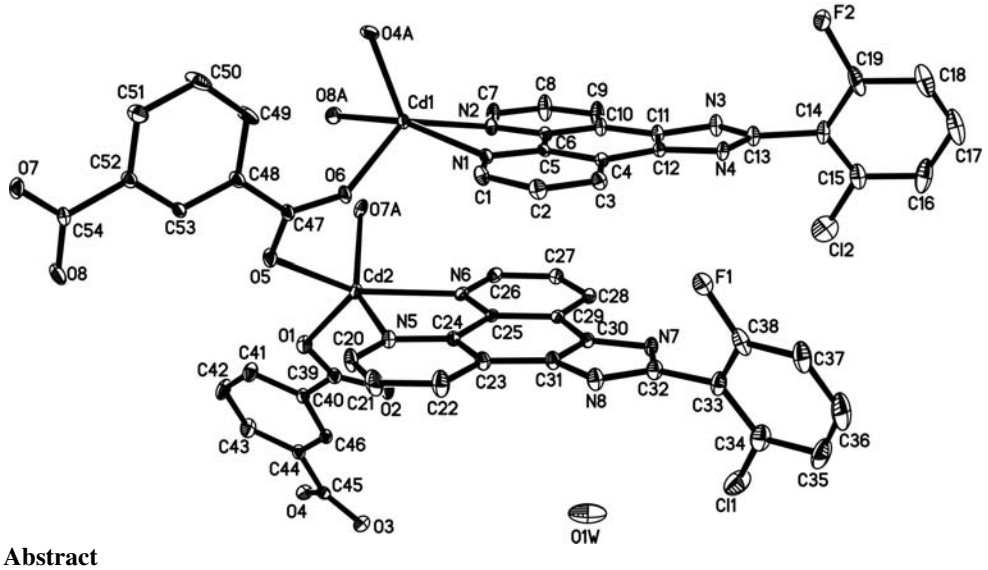

Abstract

$C_{54}H_{28.50}Cd_2Cl_2F_2N_8O_{8.25}$, monoclinic, $P2_1/n$ (no. 14), a = 17.704(5) Å, b = 11.517(3) Å, c = 26.168(7) Å, β = 105.041(4)°, V = 5152.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.147, T = 293 K.

Source of material

The pH value of a mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.5 mmol), 1,3-benzenedicarboxylic acid (1,3-H_2bdc, 0.5 mmol) and 2-(2-chloro-6-fluorophenyl)-1*H*-imidazo[4,5-*f*][1,10]phenanthroline (L, 0.5 mmol) in 12 mL distilled water was adjusted to 5 - 6 by addition of triethylamine. The resultant solution was heated at 460 K in a Teflon-lined stainless steel autoclave for four 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 (25% yield based on Cd).

Experimental details

All H atoms were positioned geometrically with d(N—H) = 0.86 Å, d(C—H) = 0.93 Å and refined as riding, with U_{iso}(H) = 1.2 U_{eq}(C,N). The hydrogen on water were not located.

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Discussion

In recent years, the design and synthesis of coordination polymers based on 1,10-phenanthroline derivatives have received intense interest of chemists because of their interesting structural topologies and properties [1].

In the title crystal structure, each Cd(II) atom is five-coordinated by two nitrogen atoms from one L ligand, and three carboxylate oxygen atoms from three different 1,3-bdc ligands. Neighboring Cd(II) atoms are linked by the 1,3-bdc ligands to a layer. The L ligands are located on both sides of the layers. In addition, the hydrogen bonds N4–H4···O3 (d(N4–H4···O3) = 2.760(6) Å, ∠N4–H4···O3 = 168.7°) and N7–H7A···O2 (d(N7–H7A···O2) = 2.688(6) Å, ∠N7–H7A···O2 = 158.4°) stabilize the layer structure.

Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.21 × 0.24 × 0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	10.00 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD, φ/ω
$2\theta_{max}$:	52.32°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28190, 1016
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6948
$N(param)_{refined}$:	694
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*	0.4654	–0.1663	0.2584	0.061
H(2)	4*e*	0.3691	–0.2882	0.2716	0.064
H(3)	4*e*	0.2637	–0.2147	0.2973	0.055
H(7)	4*e*	0.4765	0.3608	0.3086	0.071
H(8)	4*e*	0.3840	0.4556	0.3410	0.093
H(9)	4*e*	0.2785	0.3547	0.3521	0.088
H(16)	4*e*	–0.0304	0.0392	0.4466	0.160
H(17)	4*e*	–0.1378	0.0210	0.3771	0.179
H(18)	4*e*	–0.1269	0.0254	0.2947	0.153
H(20)	4*e*	0.7148	–0.2343	0.3709	0.080
H(21)	4*e*	0.6621	–0.4161	0.3653	0.105
H(22)	4*e*	0.5441	–0.4455	0.3835	0.093
H(26)	4*e*	0.5459	0.2049	0.4252	0.054
H(27)	4*e*	0.4333	0.1987	0.4520	0.051

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(28)	4*e*	0.3693	0.0243	0.4525	0.050
H(35)	4*e*	0.2023	–0.5451	0.5198	0.136
H(36)	4*e*	0.1213	–0.6186	0.4419	0.144
H(37)	4*e*	0.1364	–0.5670	0.3632	0.109
H(41)	4*e*	0.8938	0.2260	0.4530	0.078
H(42)	4*e*	1.0002	0.3299	0.4990	0.094
H(43)	4*e*	1.0176	0.3662	0.5877	0.081
H(46)	4*e*	0.8273	0.1792	0.5863	0.048
H(49)	4*e*	0.5878	–0.0239	0.1804	0.117
H(50)	4*e*	0.6162	–0.0906	0.1033	0.152
H(51)	4*e*	0.7351	–0.1827	0.1087	0.103
H(53)	4*e*	0.7927	–0.1461	0.2657	0.050
H(4)	4*e*	0.1528	–0.0953	0.3226	0.052
H(7A)	4*e*	0.3166	–0.1843	0.4491	0.052

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*		0.4243(3)	–0.1350(5)	0.2700(2)	0.039(3)	0.049(3)	0.066(4)	0.005(3)	0.017(3)	–0.010(3)
C(2)	4*e*		0.3657(3)	–0.2089(5)	0.2774(2)	0.054(4)	0.035(3)	0.073(4)	0.004(3)	0.019(3)	–0.006(3)
C(3)	4*e*		0.3037(3)	–0.1661(4)	0.2931(2)	0.040(3)	0.037(3)	0.061(3)	–0.003(2)	0.012(3)	–0.003(2)
C(4)	4*e*		0.3017(3)	–0.0464(4)	0.3027(2)	0.033(3)	0.036(3)	0.038(3)	–0.001(2)	0.009(2)	–0.002(2)
C(5)	4*e*		0.3643(3)	0.0229(4)	0.2962(2)	0.026(2)	0.036(3)	0.034(2)	0.005(2)	0.005(2)	0.002(2)
C(6)	4*e*		0.3675(3)	0.1460(4)	0.3086(2)	0.028(3)	0.040(3)	0.039(3)	–0.002(2)	0.007(2)	–0.005(2)
C(7)	4*e*		0.4341(4)	0.3187(5)	0.3137(3)	0.049(4)	0.053(4)	0.085(4)	–0.022(3)	0.034(3)	–0.014(3)
C(8)	4*e*		0.3784(4)	0.3769(5)	0.3328(3)	0.064(4)	0.044(3)	0.142(7)	–0.017(3)	0.059(5)	–0.028(4)
C(9)	4*e*		0.3163(4)	0.3174(5)	0.3392(3)	0.060(4)	0.053(4)	0.125(6)	–0.012(3)	0.054(4)	–0.033(4)
C(10)	4*e*		0.3084(3)	0.1995(4)	0.3265(2)	0.036(3)	0.039(3)	0.060(3)	–0.005(2)	0.023(3)	–0.012(2)
C(11)	4*e*		0.2435(3)	0.1279(4)	0.3310(2)	0.034(3)	0.044(3)	0.061(3)	–0.005(2)	0.022(3)	–0.010(3)
C(12)	4*e*		0.2404(3)	0.0126(4)	0.3195(2)	0.030(3)	0.039(3)	0.042(3)	–0.004(2)	0.011(2)	0.000(2)
C(13)	4*e*		0.1359(3)	0.0675(5)	0.3414(2)	0.039(3)	0.047(3)	0.071(4)	–0.006(3)	0.026(3)	–0.008(3)
C(14)	4*e*		0.0577(4)	0.0588(5)	0.3521(3)	0.048(4)	0.055(4)	0.097(5)	–0.007(3)	0.042(4)	–0.010(3)
C(15)	4*e*		0.0469(4)	0.0610(7)	0.4016(4)	0.057(4)	0.076(5)	0.123(7)	–0.005(4)	0.039(5)	–0.016(5)
C(16)	4*e*		–0.0258(7)	0.0428(9)	0.4120(5)	0.124(9)	0.127(9)	0.19(1)	–0.015(8)	0.12(1)	–0.031(8)
C(17)	4*e*		–0.0887(6)	0.031(1)	0.3710(7)	0.057(6)	0.17(1)	0.24(2)	–0.007(7)	0.058(9)	–0.05(1)
C(18)	4*e*		–0.0818(6)	0.032(1)	0.3223(6)	0.057(6)	0.14(1)	0.18(1)	–0.021(6)	0.028(7)	–0.049(9)
C(19)	4*e*		–0.0092(4)	0.0432(7)	0.3097(5)	0.038(4)	0.083(6)	0.19(1)	–0.024(4)	0.017(5)	–0.013(6)
C(20)	4*e*		0.6664(4)	–0.2443(6)	0.3780(3)	0.051(4)	0.057(4)	0.104(5)	0.007(3)	0.044(4)	–0.009(4)
C(21)	4*e*		0.6352(5)	–0.3538(7)	0.3748(3)	0.075(5)	0.070(5)	0.132(7)	0.028(4)	0.053(5)	–0.010(5)
C(22)	4*e*		0.5653(4)	–0.3713(6)	0.3855(3)	0.071(5)	0.049(4)	0.128(6)	–0.003(3)	0.054(5)	–0.016(4)
C(23)	4*e*		0.5252(3)	–0.2771(5)	0.3996(2)	0.043(3)	0.044(3)	0.058(3)	0.001(3)	0.018(3)	–0.007(3)
C(24)	4*e*		0.5604(3)	–0.1675(4)	0.4013(2)	0.035(3)	0.041(3)	0.042(3)	0.001(2)	0.013(2)	0.001(2)
C(25)	4*e*		0.5215(3)	–0.0653(4)	0.4149(2)	0.034(3)	0.038(3)	0.025(2)	–0.001(2)	0.008(2)	–0.002(2)
C(26)	4*e*		0.5223(3)	0.1332(5)	0.4266(2)	0.053(3)	0.039(3)	0.044(3)	–0.010(3)	0.015(3)	–0.001(2)
C(27)	4*e*		0.4539(3)	0.1306(4)	0.4421(2)	0.045(3)	0.037(3)	0.048(3)	–0.001(2)	0.015(3)	–0.004(2)
C(28)	4*e*		0.4163(3)	0.0273(4)	0.4429(2)	0.042(3)	0.043(3)	0.041(3)	0.000(3)	0.014(2)	–0.003(2)
C(29)	4*e*		0.4501(3)	–0.0740(4)	0.4290(2)	0.034(3)	0.037(3)	0.027(2)	–0.006(2)	0.005(2)	–0.004(2)
C(30)	4*e*		0.4174(3)	–0.1884(4)	0.4277(2)	0.034(3)	0.041(3)	0.041(3)	–0.006(2)	0.007(2)	–0.001(2)
C(31)	4*e*		0.4517(3)	–0.2849(4)	0.4126(2)	0.040(3)	0.037(3)	0.062(3)	–0.003(3)	0.018(3)	–0.004(3)
C(32)	4*e*		0.3458(4)	–0.3412(5)	0.4284(2)	0.049(3)	0.041(3)	0.064(4)	–0.011(3)	0.018(3)	–0.008(3)
C(33)	4*e*		0.2803(4)	–0.4162(5)	0.4336(3)	0.062(4)	0.040(3)	0.104(5)	–0.005(3)	0.049(4)	–0.003(3)
C(34)	4*e*		0.2687(5)	–0.4471(6)	0.4831(3)	0.086(6)	0.060(4)	0.111(6)	–0.021(4)	0.040(5)	–0.015(4)
C(35)	4*e*		0.2093(6)	–0.5238(7)	0.4871(5)	0.120(8)	0.072(5)	0.18(1)	–0.023(6)	0.091(8)	0.019(6)
C(36)	4*e*		0.1610(5)	–0.5669(8)	0.4402(6)	0.075(6)	0.071(6)	0.23(1)	–0.038(5)	0.065(8)	–0.044(7)
C(37)	4*e*		0.1700(5)	–0.5365(7)	0.3936(4)	0.059(5)	0.068(5)	0.154(9)	–0.017(4)	0.043(6)	–0.029(5)
C(38)	4*e*		0.2272(4)	–0.4614(5)	0.3889(3)	0.039(3)	0.038(3)	0.127(6)	–0.003(3)	0.016(4)	–0.030(4)
C(39)	4*e*		0.7798(3)	0.1216(5)	0.4842(2)	0.039(3)	0.059(4)	0.043(3)	0.000(3)	0.009(3)	–0.006(3)
C(40)	4*e*		0.8488(3)	0.1901(5)	0.5147(2)	0.038(3)	0.058(3)	0.036(3)	–0.002(3)	0.010(2)	–0.008(2)
C(41)	4*e*		0.9015(4)	0.2375(6)	0.4892(2)	0.058(4)	0.093(5)	0.048(3)	–0.019(4)	0.021(3)	–0.019(3)
C(42)	4*e*		0.9647(4)	0.3008(7)	0.5164(3)	0.061(4)	0.121(6)	0.058(4)	–0.042(4)	0.027(3)	–0.013(4)
C(43)	4*e*		0.9756(4)	0.3213(6)	0.5695(2)	0.049(4)	0.092(5)	0.058(4)	–0.028(4)	0.010(3)	–0.021(3)
C(44)	4*e*		0.9243(3)	0.2754(5)	0.5961(2)	0.036(3)	0.054(3)	0.043(3)	–0.003(3)	0.009(2)	–0.005(2)
C(45)	4*e*		0.9370(3)	0.2983(5)	0.6542(2)	0.036(3)	0.045(3)	0.042(3)	–0.002(3)	0.001(2)	–0.002(2)
C(46)	4*e*		0.8617(3)	0.2103(4)	0.5685(2)	0.032(3)	0.046(3)	0.041(3)	–0.006(2)	0.009(2)	–0.005(2)
C(47)	4*e*		0.6684(3)	–0.0385(5)	0.2805(2)	0.036(3)	0.049(3)	0.045(3)	0.010(3)	0.015(3)	–0.005(2)
C(48)	4*e*		0.6874(3)	–0.0813(5)	0.2311(2)	0.040(3)	0.062(3)	0.042(3)	0.017(3)	0.006(3)	–0.009(3)

Table 3. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(49)	4*e*		0.6346(4)	–0.0626(8)	0.1822(2)	0.076(5)	0.156(7)	0.046(4)	0.080(5)	–0.009(4)	–0.023(4)
C(50)	4*e*		0.6517(5)	–0.1018(9)	0.1360(3)	0.116(7)	0.21(1)	0.038(4)	0.120(7)	–0.011(4)	–0.028(5)
C(51)	4*e*		0.7226(4)	–0.1582(7)	0.1394(2)	0.089(5)	0.126(6)	0.040(3)	0.064(5)	0.012(4)	–0.012(4)
C(52)	4*e*		0.7743(3)	–0.1777(5)	0.1880(2)	0.044(3)	0.059(4)	0.043(3)	0.019(3)	0.010(3)	–0.005(3)
C(53)	4*e*		0.7566(3)	–0.1363(4)	0.2332(2)	0.033(3)	0.055(3)	0.037(3)	0.012(3)	0.007(2)	–0.004(2)
C(54)	4*e*		0.8500(3)	–0.2415(5)	0.1919(2)	0.036(3)	0.052(3)	0.056(3)	0.009(3)	0.021(3)	–0.003(3)
N(1)	4*e*		0.4235(2)	–0.0210(4)	0.2790(2)	0.030(2)	0.046(3)	0.041(2)	0.004(2)	0.010(2)	–0.001(2)
N(2)	4*e*		0.4303(2)	0.2076(4)	0.3024(2)	0.033(2)	0.040(2)	0.052(2)	–0.009(2)	0.014(2)	–0.009(2)
N(3)	4*e*		0.1767(3)	0.1638(4)	0.3445(2)	0.043(3)	0.049(3)	0.087(4)	–0.008(2)	0.034(3)	–0.015(3)
N(4)	4*e*		0.1707(2)	–0.0256(4)	0.3267(2)	0.037(2)	0.040(2)	0.060(3)	–0.010(2)	0.024(2)	–0.007(2)
N(5)	4*e*		0.6311(3)	–0.1543(4)	0.3904(2)	0.040(3)	0.053(3)	0.055(3)	0.000(2)	0.020(2)	–0.003(2)
N(6)	4*e*		0.5567(2)	0.0392(4)	0.4135(2)	0.040(2)	0.046(3)	0.032(2)	–0.008(2)	0.010(2)	0.002(2)
N(7)	4*e*		0.3498(3)	–0.2257(3)	0.4384(2)	0.040(2)	0.036(2)	0.059(3)	–0.009(2)	0.023(2)	–0.010(2)
N(8)	4*e*		0.4058(3)	–0.3812(4)	0.4127(2)	0.049(3)	0.039(3)	0.085(4)	–0.011(2)	0.026(3)	–0.012(2)
O(1)	4*e*		0.7808(2)	0.0905(4)	0.4377(1)	0.048(2)	0.092(3)	0.038(2)	–0.010(2)	0.009(2)	–0.019(2)
O(2)	4*e*		0.7249(2)	0.0974(4)	0.5042(2)	0.040(2)	0.077(3)	0.061(2)	–0.014(2)	0.020(2)	–0.029(2)
O(1W)	4*e*	0.25	0.465(2)	–0.598(1)	0.4674(8)	0.18(3)	0.04(1)	0.08(1)	0.00(1)	–0.02(2)	–0.002(9)
O(3)	4*e*		0.9033(2)	0.2382(3)	0.6812(1)	0.063(3)	0.057(2)	0.045(2)	–0.020(2)	0.013(2)	–0.008(2)
O(4)	4*e*		0.9823(2)	0.3822(3)	0.6728(1)	0.054(3)	0.059(2)	0.043(2)	–0.021(2)	0.001(2)	–0.007(2)
O(5)	4*e*		0.7242(2)	–0.0239(4)	0.3202(1)	0.039(2)	0.088(3)	0.040(2)	0.001(2)	0.013(2)	–0.016(2)
O(6)	4*e*		0.5986(2)	–0.0218(3)	0.2810(1)	0.036(2)	0.064(2)	0.057(2)	0.015(2)	0.020(2)	–0.003(2)
O(7)	4*e*		0.8628(2)	–0.2844(3)	0.1502(2)	0.058(3)	0.055(2)	0.064(2)	0.014(2)	0.036(2)	–0.004(2)
O(8)	4*e*		0.8968(2)	–0.2507(4)	0.2367(2)	0.040(2)	0.094(3)	0.062(3)	0.028(2)	0.006(2)	–0.016(2)
Cd(1)	4*e*		0.50912(2)	0.12090(3)	0.25901(1)	0.0267(2)	0.0504(2)	0.0415(2)	–0.0013(2)	0.0121(2)	0.0000(2)
Cd(2)	4*e*		0.67039(2)	0.03767(3)	0.38554(1)	0.0347(2)	0.0544(3)	0.0371(2)	–0.0080(2)	0.0143(2)	–0.0050(2)
Cl(1)	4*e*		0.3285(2)	–0.3874(2)	0.5383(1)	0.147(2)	0.110(2)	0.099(2)	–0.042(2)	0.036(2)	0.012(1)
Cl(2)	4*e*		0.1175(2)	0.0892(3)	0.4509(1)	0.154(3)	0.187(3)	0.147(3)	0.006(3)	0.048(2)	0.032(2)
F(1)	4*e*		0.2336(2)	–0.4315(3)	0.3388(2)	0.057(2)	0.048(2)	0.082(3)	–0.004(2)	0.015(2)	–0.006(2)
F(2)	4*e*		–0.0072(2)	0.0571(3)	0.2513(1)	0.058(2)	0.094(3)	0.049(2)	–0.030(2)	0.016(2)	–0.025(2)

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