Crystal structure of [(2-(2-chloro-6-fluorophenyl)-1*H*-imidazo-[4,5-*f*][1,10]phenanthroline]-(benzene-1,4-dicarboxylato)lead(II), $Pb(C_{19}H_{10}N_4FCl)_2(C_8H_4O_4)$

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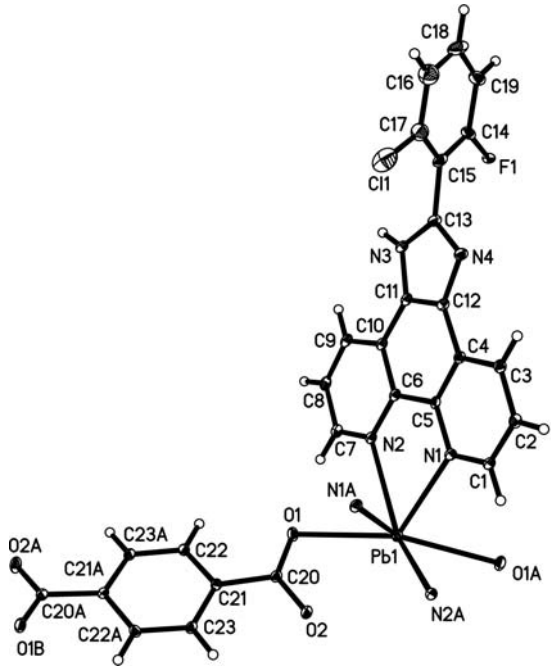
Received January 31, 2011, accepted and available on-line February 2, 2011; CCDC no. 1267/3384



Abstract

$C_{46}H_{24}Cl_2F_2N_8O_4Pb$, monoclinic, *C*12/*c*1 (no. 15), a = 11.975(2) Å, b = 17.585(3) Å, c = 19.587(3) Å, β = 106.981(2)°, V = 3945.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.104, T = 293 K.

Source of material

The pH value of a mixture of $Pb(NO_3)_2$ (0.5 mmol), benzene-1,4-dicarboxylic acid (1,4-H_2bdc, 0.5 mmol) and 2-(2-chloro-6-fluorophenyl)-1*H*-imidazo[4,5-*f*][1,10]phenanthroline (L, 1 mmol) in 13 mL distilled water was adjusted to between 5 and 6 by addition of triethylamine. The resultant solution was heated at 458 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 (yield 31 % based on Pb).

Experimental details

All H atoms were positioned geometrically with d(N—H) = 0.86 Å, d(C—H) = 0.93 Å and treated as riding with U_{iso}(H) = 1.2 U_{eq}(C,N).

Discussion

It is well-known that 1,10-phenanthroline (phen) has been widely used to construct supramolecular architectures due to its excellent coordinating ability and large conjugated system [1].

In the title complex, each Pb(II) cation is coordinated by four nitrogen atoms from two different L ligands, and two carboxylate oxygen atoms from two different 1,4-bdc ligands. The 1,4-bdc ligands connect two neighboring Pb(II) cations to form a chain. The L ligands are located on both sides of the one-dimensional chains. In addition, the N3–H3A···O2 hydrogen bond (d(N3–H3A···O2) = 2.723(6) Å, ∠N3–H3A···O2 = 161.4°) further stabilizes the chain structure.

Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.15 × 0.18 × 0.27 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	44.80 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD, φ/ω
$2\theta_{max}$:	52.1°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10896, 3890
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3386
$N(param)_{refined}$:	273
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)	8*f*	0.3560	0.1999	0.8117	0.053
H(2)	8*f*	0.2774	0.0798	0.7977	0.056
H(3)	8*f*	0.2423	0.0193	0.6886	0.047
H(7)	8*f*	0.5255	0.3651	0.5774	0.051
H(8)	8*f*	0.4714	0.3266	0.4594	0.052
H(9)	8*f*	0.3768	0.2133	0.4277	0.049
H(22)	8*f*	0.8438	0.3891	0.6472	0.044
H(23)	8*f*	0.9687	0.3942	0.8607	0.046
H(3A)	8*f*	0.2798	0.0790	0.4145	0.048
H(16)	8*f*	0.2459	–0.2392	0.4115	0.124
H(18)	8*f*	0.0676	–0.2349	0.3235	0.166
H(19)	8*f*	–0.0376	–0.1222	0.3011	0.137

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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)	8*f*	0.3442(6)	0.1756(4)	0.7680(3)	0.050(4)	0.057(4)	0.031(3)	–0.013(3)	0.022(3)	–0.009(3)
C(2)	8*f*	0.2956(6)	0.1036(4)	0.7599(3)	0.050(3)	0.065(4)	0.028(3)	–0.010(3)	0.018(3)	0.004(3)
C(3)	8*f*	0.2748(5)	0.0677(3)	0.6953(3)	0.039(3)	0.043(3)	0.036(3)	–0.002(3)	0.014(2)	0.004(2)
C(4)	8*f*	0.3031(5)	0.1051(3)	0.6394(3)	0.035(3)	0.042(3)	0.026(3)	0.001(2)	0.010(2)	0.001(2)
C(5)	8*f*	0.3556(5)	0.1770(3)	0.6528(3)	0.025(2)	0.049(4)	0.027(3)	0.005(2)	0.010(2)	0.003(2)
C(6)	8*f*	0.3899(5)	0.2180(3)	0.5967(3)	0.031(3)	0.040(3)	0.028(3)	0.010(2)	0.009(2)	0.003(2)
C(7)	8*f*	0.4817(6)	0.3209(3)	0.5647(3)	0.044(3)	0.046(4)	0.042(3)	0.003(3)	0.019(3)	0.003(3)
C(8)	8*f*	0.4520(5)	0.2968(4)	0.4935(3)	0.050(4)	0.045(4)	0.043(4)	0.003(3)	0.028(3)	0.010(3)
C(9)	8*f*	0.3953(5)	0.2304(4)	0.4746(3)	0.048(3)	0.052(4)	0.029(3)	0.011(3)	0.019(3)	0.005(3)
C(10)	8*f*	0.3642(5)	0.1868(3)	0.5273(3)	0.035(3)	0.040(3)	0.029(3)	0.008(2)	0.012(2)	0.004(2)
C(11)	8*f*	0.3083(5)	0.1140(3)	0.5152(3)	0.038(3)	0.043(3)	0.029(3)	0.006(2)	0.014(2)	0.002(2)
C(12)	8*f*	0.2812(5)	0.0739(3)	0.5691(3)	0.038(3)	0.036(3)	0.032(3)	0.006(2)	0.011(2)	0.001(2)
C(13)	8*f*	0.2287(5)	0.0045(4)	0.4765(3)	0.052(3)	0.041(3)	0.035(3)	–0.001(3)	0.015(3)	–0.008(3)
C(20)	8*f*	0.7620(5)	0.3790(3)	0.7595(3)	0.037(3)	0.040(3)	0.031(3)	0.001(2)	0.013(2)	0.001(2)
C(21)	8*f*	0.8852(5)	0.3871(3)	0.7550(3)	0.034(3)	0.036(3)	0.031(3)	0.000(2)	0.014(2)	0.003(2)
C(22)	8*f*	0.9063(5)	0.3890(3)	0.6887(3)	0.029(3)	0.051(4)	0.029(3)	0.004(2)	0.005(2)	–0.001(2)
C(23)	8*f*	0.9811(5)	0.3907(4)	0.8161(3)	0.038(3)	0.056(4)	0.024(3)	0.001(3)	0.014(2)	0.001(2)
N(1)	8*f*	0.3746(4)	0.2117(3)	0.7173(2)	0.040(3)	0.044(3)	0.028(2)	–0.004(2)	0.013(2)	–0.003(2)
N(2)	8*f*	0.4501(4)	0.2834(3)	0.6146(2)	0.036(2)	0.040(3)	0.031(2)	0.004(2)	0.013(2)	0.002(2)
N(3)	8*f*	0.2741(4)	0.0689(3)	0.4563(2)	0.049(3)	0.048(3)	0.026(2)	0.001(2)	0.016(2)	–0.003(2)
N(4)	8*f*	0.2316(4)	0.0054(3)	0.5442(3)	0.055(3)	0.039(3)	0.036(3)	–0.005(2)	0.018(2)	–0.005(2)
O(1)	8*f*	0.6848(4)	0.3636(3)	0.7039(2)	0.033(2)	0.083(3)	0.034(2)	–0.009(2)	0.013(2)	0.000(2)
O(2)	8*f*	0.7437(4)	0.3860(3)	0.8191(2)	0.040(2)	0.088(4)	0.038(2)	–0.008(2)	0.020(2)	–0.014(2)
Pb(1)	4*e*	½	0.33451(2)	¾	0.0326(2)	0.0395(2)	0.0286(2)	0	0.0125(1)	0
F(1)	8*f*	0.0081(2)	0.0068(2)	0.3598(2)	0.023(1)	0.031(2)	0.039(2)	0.014(1)	–0.007(1)	–0.007(1)
Cl(1)	8*f*	0.3660(3)	–0.1359(2)	0.4971(2)	0.112(2)	0.147(3)	0.099(2)	0.062(2)	0.006(2)	0.001(2)
C(14)	8*f*	0.0776(4)	–0.0591(3)	0.3756(3)	0.088(6)	0.116(7)	0.046(4)	–0.065(6)	0.038(4)	–0.044(4)
C(15)	8*f*	0.1844(4)	–0.0617(3)	0.4283(2)	0.084(5)	0.047(4)	0.045(4)	–0.005(3)	0.040(4)	–0.006(3)
C(17)	8*f*	0.2474(5)	–0.1292(3)	0.4418(3)	0.077(2)	0.076(2)	0.076(2)	0.000(1)	0.024(1)	–0.001(1)
C(16)	8*f*	0.2037(7)	–0.1941(3)	0.4025(4)	0.105(4)	0.103(4)	0.104(4)	–0.000(1)	0.032(1)	–0.000(1)
C(18)	8*f*	0.0969(7)	–0.1915(3)	0.3498(4)	0.26(2)	0.093(7)	0.14(1)	–0.090(9)	0.17(1)	–0.064(7)
C(19)	8*f*	0.0339(5)	–0.1240(4)	0.3364(3)	0.132(9)	0.15(1)	0.081(7)	–0.093(9)	0.069(7)	–0.074(7)

Acknowledgments. The authors thank the Key Laboratory of Preparation and Applications of Environmental Friendly Materials and Institute Foundation of Siping City (grant no. 2009011) for supporting this work.

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