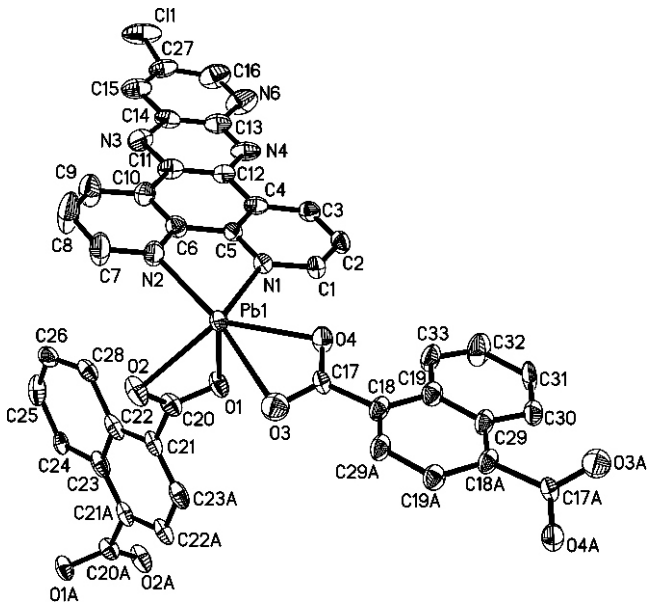
Crystal structure of dipyrido[7,6-*a*:6′,7′-*c*]-3-chloropyrido[2,3-*b*]-quinoxaline)-(naphthalene-1,4-dicarboxylato)lead(II), $Pb(C_{17}H_8N_4Cl)(C_{12}H_6O_4)$

Ji-Ku Wang[*,I,II], Hao Chen[I,II] and Hong-Bin Xu[III]

[I] Jilin Normal University, College of Chemistry, Siping 136000, P. R. China
[II] Key Laboratory of Preparation and Applications of Environment-Friendly Materials, Jilin Normal University, Ministry of Education, P. R. China
[III] China Science and Technology Exchange Center, Ministry of Science and Technology, Beijing 100045, P. R. China

Received February 2, 2011, accepted and available on-line June 17, 2011; CCDC no. 1267/3386



Abstract

$C_{29}H_{14}ClN_4O_4Pb$, triclinic, $P\bar{1}$ (no. 2), a = 10.310(5) Å, b = 10.521(5) Å, c = 12.168(5) Å, α = 80.808(5)°, β = 81.973(5)°, γ = 79.638(5)°, V = 1273.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.138, T = 293 K.

Source of material

The pH value of a mixture of $Pb(NO_3)_2$ (0.5 mmol), naphthalene-1,4-dicarboxylatic acid (1,4-H_2ndc, 0.5 mmol) and dipyrido[7,6-*a*:6′,7′-*c*]-3-chloropyrido[2,3-*b*]quinoxaline (L, 0.5 mmol) in 10 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 seven 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 27 % based on Pb).

Experimental details

All H atoms were positioned geometrically d(C—H) = 0.93 Å and refined as riding with U_{iso}(H) = 1.2 U_{eq}(C). The naphthalene-1,4-dicarboxylate ligand is disordered over two positions in the fused-ring portion. The hydrogen atoms attached to C29' and C19' were not generated geometrically because of the disordered fused-ring portion.

Discussion

The current interest in coordination polymer frameworks not only stems from their potential applictions in microelectronics, nonlinear optics, porous materials, and catalysis, but also from their intriguing variety of topologies and entanglement motifs [1]. Recently, 1,10-phenanthroline (phen) derivatives have received intense interests in their coordination chemistry [2].

In the title complex, each Pb(II) atom is six-coordinated by two nitrogen atoms from one L ligand, and four carboxylate oxygen atoms from two different 1,4-ndc^{-2} ligands. The 1,4-ndc^{-2} ligands bridge neighboring Pb(II) atoms to form one-dimensional chains. The Pb···Pb distance bridged by 1,4-ndc^{-2} is about 11.72 Å. The L ligands are located on both sides of the one-dimensional chains. Further, the strong π-π interactions between the L ligands and the 1,4-ndc ligands of neighboring chains result in a 2D supramolecular structure [2].

Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.16 × 0.18 × 0.21 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	67.77 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD, φ/ω
$2\theta_{max}$:	50.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6666, 4456
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3850
$N(param)_{refined}$:	385
Programs:	SHELXS-97, SHELXL-97 [3]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	2*i*		0.4369	0.1788	0.0667	0.076
H(2)	2*i*		0.2571	0.2967	–0.0148	0.084
H(3)	2*i*		0.2754	0.3507	–0.2082	0.081
H(7)	2*i*		0.9847	0.0053	–0.2029	0.148
H(8)	2*i*		1.0046	0.0208	–0.3967	0.199
H(9)	2*i*		0.8537	0.1665	–0.4937	0.141
H(15)	2*i*		0.6070	0.3570	–0.7457	0.125
H(16)	2*i*		0.2259	0.5258	–0.7085	0.156
H(24)	2*i*	0.50	1.1326	0.6950	–0.2363	0.064
H(25)	2*i*	0.50	1.1339	0.5951	–0.3994	0.105

* Correspondence author (e-mail: wangjikujlnu@yahoo.com.cn)

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(26)	2*i*	0.50	1.0581	0.4092	–0.3882	0.095
H(28)	2*i*	0.50	0.9689	0.3052	–0.2077	0.081
H(30)	2*i*	0.50	0.1927	0.1185	0.6115	0.096

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(31)	2*i*	0.50	0.0792	0.2302	0.4642	0.106
H(32)	2*i*	0.50	0.1833	0.2397	0.2819	0.139
H(33)	2*i*	0.50	0.4009	0.1376	0.2470	0.099

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*		0.446(1)	0.201(1)	–0.0108(9)	0.051(6)	0.070(7)	0.065(6)	–0.007(5)	–0.001(5)	–0.005(5)
C(2)	2*i*		0.336(1)	0.272(1)	–0.059(1)	0.044(6)	0.078(7)	0.087(8)	–0.004(5)	–0.004(5)	–0.014(6)
C(3)	2*i*		0.347(1)	0.303(1)	–0.1731(9)	0.060(6)	0.067(7)	0.076(7)	–0.001(5)	–0.020(5)	–0.013(5)
C(4)	2*i*		0.467(1)	0.264(1)	–0.2362(9)	0.082(7)	0.048(5)	0.068(6)	–0.014(5)	–0.025(5)	–0.006(4)
C(5)	2*i*		0.5742(9)	0.1939(9)	–0.1805(8)	0.051(5)	0.056(5)	0.058(5)	–0.012(4)	–0.006(4)	–0.009(4)
C(6)	2*i*		0.697(1)	0.154(1)	–0.2434(9)	0.063(7)	0.090(8)	0.061(6)	–0.016(6)	–0.015(5)	–0.002(5)
C(7)	2*i*		0.913(2)	0.045(2)	–0.241(1)	0.068(9)	0.19(2)	0.09(1)	0.01(1)	0.008(7)	–0.01(1)
C(8)	2*i*		0.929(2)	0.063(3)	–0.359(1)	0.10(1)	0.27(3)	0.08(1)	0.04(2)	0.017(9)	0.00(1)
C(9)	2*i*		0.836(2)	0.140(2)	–0.417(1)	0.076(9)	0.18(2)	0.079(9)	0.01(1)	–0.009(7)	0.01(1)
C(10)	2*i*		0.714(1)	0.180(1)	–0.361(1)	0.082(8)	0.094(9)	0.067(7)	–0.024(7)	0.005(6)	–0.008(6)
C(11)	2*i*		0.603(1)	0.255(1)	–0.4170(9)	0.11(1)	0.062(6)	0.056(6)	–0.023(6)	–0.028(6)	0.010(5)
C(12)	2*i*		0.482(1)	0.296(1)	–0.3573(9)	0.076(7)	0.058(6)	0.068(6)	–0.008(5)	–0.021(6)	–0.011(5)
C(13)	2*i*		0.393(2)	0.387(1)	–0.517(1)	0.11(1)	0.075(8)	0.083(9)	–0.012(8)	–0.042(8)	–0.009(6)
C(14)	2*i*		0.510(1)	0.353(1)	–0.578(1)	0.098(9)	0.063(7)	0.070(7)	–0.024(6)	–0.042(7)	0.008(5)
C(15)	2*i*		0.527(2)	0.381(1)	–0.702(1)	0.16(2)	0.063(8)	0.10(1)	–0.042(9)	–0.04(1)	–0.004(7)
C(16)	2*i*		0.299(2)	0.480(2)	–0.674(2)	0.16(2)	0.12(1)	0.11(1)	0.01(1)	–0.07(1)	–0.02(1)
C(17)	2*i*		0.6393(9)	0.027(1)	0.2729(8)	0.038(5)	0.061(6)	0.062(6)	–0.013(4)	0.001(4)	0.003(4)
C(18)	2*i*		0.566(1)	0.012(1)	0.3909(8)	0.083(7)	0.055(6)	0.051(5)	–0.022(5)	0.017(5)	–0.006(4)
C(20)	2*i*		0.900(1)	0.2545(9)	0.022(1)	0.055(6)	0.043(5)	0.091(8)	–0.011(5)	–0.010(5)	0.000(5)
C(21)	2*i*		0.9527(9)	0.382(1)	0.008(1)	0.033(5)	0.049(5)	0.108(9)	–0.008(4)	0.001(5)	–0.002(5)
C(22)	2*i*		1.003(1)	0.439(1)	–0.100(1)	0.036(5)	0.051(6)	0.13(1)	–0.008(4)	–0.005(6)	–0.007(6)
C(23)	2*i*		1.052(1)	0.559(1)	–0.106(1)	0.039(5)	0.047(6)	0.13(1)	0.002(4)	–0.005(6)	0.000(6)
C(24)	2*i*	0.50	1.101(2)	0.616(2)	–0.227(2)	0.039(9)	0.040(9)	0.07(1)	–0.002(7)	0.000(8)	0.010(8)
C(25)	2*i*	0.50	1.101(2)	0.555(2)	–0.330(2)	0.07(2)	0.06(1)	0.11(2)	0.01(1)	0.02(1)	0.02(1)
C(26)	2*i*	0.50	1.057(2)	0.447(3)	–0.324(2)	0.05(1)	0.08(2)	0.11(2)	0.00(1)	–0.02(1)	–0.04(2)
C(27)	2*i*		0.417(2)	0.443(1)	–0.744(1)	0.15(1)	0.073(8)	0.063(7)	–0.014(9)	–0.047(8)	–0.008(6)
C(28)	2*i*	0.50	1.003(2)	0.382(2)	–0.211(2)	0.04(1)	0.06(1)	0.10(2)	–0.004(9)	–0.02(1)	–0.00(1)
C(19)	2*i*		0.4272(8)	0.0668(8)	0.4083(6)	0.073(7)	0.064(6)	0.062(6)	–0.019(5)	0.005(5)	–0.003(5)
C(29)	2*i*		0.3649(9)	0.0611(9)	0.5175(6)	0.076(7)	0.067(7)	0.057(6)	–0.025(6)	0.019(5)	–0.006(5)
C(30)	2*i*	0.50	0.2345(9)	0.122(1)	0.5384(8)	0.07(2)	0.12(2)	0.05(1)	–0.03(1)	0.01(1)	–0.00(1)
C(31)	2*i*	0.50	0.1664(9)	0.189(2)	0.450(1)	0.04(1)	0.13(2)	0.08(2)	–0.02(1)	0.03(1)	0.01(1)
C(32)	2*i*	0.50	0.229(1)	0.195(2)	0.341(1)	0.11(2)	0.15(3)	0.06(2)	–0.02(2)	0.00(2)	0.03(2)
C(33)	2*i*	0.50	0.359(1)	0.134(1)	0.3200(6)	0.08(2)	0.11(2)	0.05(1)	–0.02(1)	0.01(1)	–0.00(1)
N(1)	2*i*		0.5634(7)	0.1630(7)	–0.0676(6)	0.051(4)	0.052(4)	0.057(5)	–0.009(4)	–0.008(3)	–0.002(3)
N(2)	2*i*		0.798(1)	0.082(1)	–0.1843(8)	0.062(6)	0.127(9)	0.064(6)	–0.003(6)	–0.003(5)	–0.016(6)
N(3)	2*i*		0.620(1)	0.284(1)	–0.5325(9)	0.120(9)	0.083(7)	0.077(7)	–0.033(7)	–0.005(6)	–0.011(5)
N(4)	2*i*		0.380(1)	0.361(1)	–0.4073(9)	0.124(9)	0.069(6)	0.091(7)	–0.007(6)	–0.060(7)	–0.010(5)
N(6)	2*i*		0.284(2)	0.454(1)	–0.566(1)	0.15(1)	0.14(1)	0.097(9)	0.03(1)	–0.071(9)	–0.021(8)
O(1)	2*i*		0.7784(7)	0.2542(7)	0.0471(7)	0.049(4)	0.058(4)	0.109(6)	–0.018(3)	0.007(4)	–0.005(4)
O(2)	2*i*		0.9787(8)	0.1549(7)	0.0017(9)	0.054(4)	0.052(4)	0.171(9)	–0.008(4)	–0.002(5)	–0.015(5)
O(3)	2*i*		0.761(1)	0.020(1)	0.2604(9)	0.099(3)	0.101(3)	0.099(3)	–0.016(1)	–0.011(1)	–0.014(1)
O(4)	2*i*		0.5772(7)	0.0533(8)	0.1913(6)	0.056(4)	0.096(6)	0.061(4)	–0.004(4)	–0.005(3)	0.006(4)
Cl(1)	2*i*		0.3995(7)	0.4896(4)	–0.8800(4)	0.271(7)	0.107(3)	0.110(3)	–0.055(4)	–0.113(4)	0.017(2)
Pb(1)	2*i*		0.76337(3)	0.03798(3)	0.03963(3)	0.0457(2)	0.0493(2)	0.0546(2)	–0.0115(2)	–0.0002(2)	–0.0011(1)

Acknowledgment. The authors thank the Key Laboratory of Preparation and Applications of Environment-Friendly Materials for supporting this work.

References

1. Wang, X.-L.; Bi, Y.-F.; Lin, H.-Y.; Liu, G.-C.: Three Novel Cd(II) Metal-Organic Frameworks Constructed from Mixed Ligands of Dipyrido[3,2-*d*:2′,3′-*f*]quinoxaline and Benzene-dicarboxylate: From a 1-D Ribbon, 2-D Layered Network, to a 3-D Architecture. Cryst. Growth Des. **7** (2007) 1086-1091.
2. Kong, Z.-G.; Wang, M.; Ma, X.-Y.; Wang, Q.-W.: *catena*-Poly[[aqua(11-chloropyrido[2′,3′:2,3]pyrimidino[5,6-*f*][1,10]phenanthroline)cadmium(II)]-benzene-1,4-dicarboxylato]: an inclined interpenetrating (6,3) network. Acta Crystallogr. **C65** (2009) m472-m474.
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.