Crystal structure of (1,10-phenanthroline-*N*,*N*′)bis(2-fluorobenzoato)lead(II), $Pb(FC_6H_4COO)_2(C_{12}H_8N_2)$

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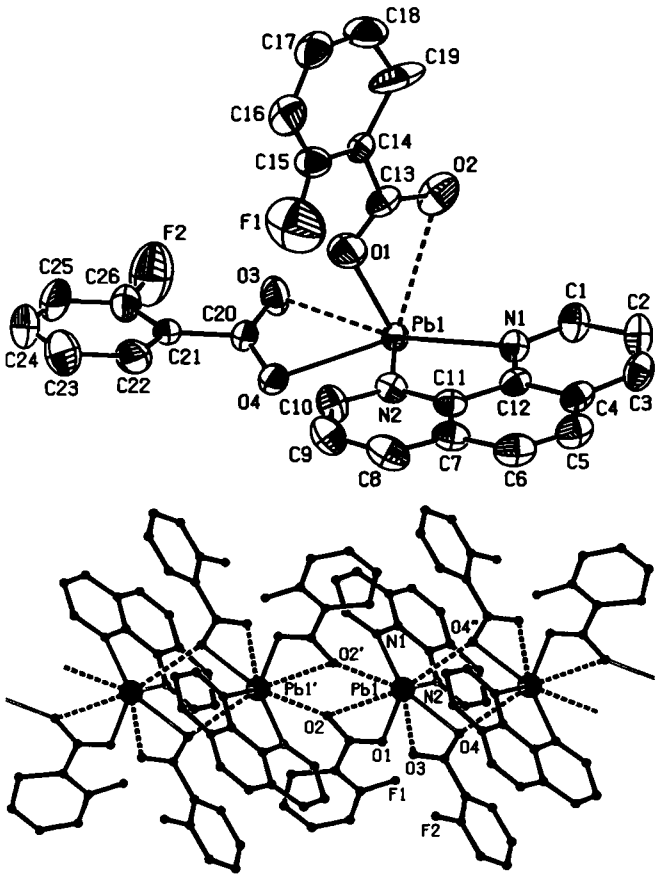

Abstract

$C_{26}H_{16}F_2N_2O_4Pb$, triclinic, $P\bar{1}$ (no. 2), $a = 9.431(2)$ Å, $b = 10.950(2)$ Å, $c = 11.609(2)$ Å, $\alpha = 80.90(3)°$, $\beta = 84.41(3)°$, $\gamma = 73.36(3)°$, $V = 1132.5$ Å^3, $Z = 2$, $R_{gt}(F) = 0.035$, $wR_{ref}(F^2) = 0.098$, $T = 293$ K.

Source of material

Freshly prepared $PbCO_3$ (0.13 g, 0.49 mmol), 1,10-phenanthroline (phen · H_2O) (0.10 g, 0.51 mmol), 2-fluoro-benzoic acid (0.08 g, 0.52 mmol), 12 mL CH_3OH/H_2O (1:2 *v/v*) were mixed and stirred for ca. 3 h. Subsequently, the resulting suspension was heated in a 23 ml Teflon-lined stainless steel autoclave at 373 K for 7 day. After the autoclave was cooled to room temperature, the solid was filtered off. The resulting yellow filtrate was allowed to stand at room temperature and slow evaporation for a week afforded yellowish block crystals.

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Discussion

The crystal structure of the title compound is similar to the structure of $[Pb(C_7H_4O_2I)(C_{12}H_8N_2)_2](C_7H_4O_2I) \cdot 2H_2O$ [1]. Within the $[Pb(C_7H_4O_2F)_2(C_{12}H_8N_2)]$ complex molecule (figure, top), each Pb atom is coordinated by two N atoms from one bidentate chelating *o*-phenanthroline ligand and two O atoms from two *o*-fluoro-benzoic acid anion ligands, to complete an PbN_2O_2 non-regular coordination polyhedron with *d*(Pb—N) = 2.567(5) Å and 2.665(5) Å, *d*(Pb—O) = 2.311(5) Å and 2.509(4) Å. The $[Pb(phen)(C_7H_4O_2F)_2]$ molecules form 1 D chains along the *a* axis via Pb···O interactions with *d*(Pb···O3) = 2.847 Å, *d*(Pb···O4″) = 2.953 Å, *d*(Pb···O2′) = 3.000 Å and *d*(Pb···O2) = 3.019 Å (figure, bottom). The chains are connected to each other via weak hydrogen bonds between the 2-fluorobenzoic acid anion F and *o*-phenanthroline H atoms with *d*(C–H···F) = 3.11(4) Å – 3.29(5) Å, ∠C–H···F = 129.0(6)° – 154.1(9)°. Through the week Pb···O and hydrogen bonds interactions the compound is interlinked to form the 3D network.

Table 1. Data collection and handling.

Crystal:	yellowish block, size 0.12 × 0.31 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	75.02 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD II, φ/ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13657, 5570
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4800
$N(param)_{refined}$:	317
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	2*i*	0.5337	0.3239	0.6786	0.065
H(2)	2*i*	0.6473	0.1270	0.7914	0.065
H(3)	2*i*	0.5785	–0.0501	0.7778	0.065
H(5)	2*i*	0.4255	–0.1569	0.6821	0.065
H(6)	2*i*	0.2626	–0.1466	0.5510	0.065
H(8)	2*i*	0.0947	–0.0159	0.3942	0.065
H(9)	2*i*	0.0003	0.1808	0.2816	0.065
H(10)	2*i*	0.0608	0.3599	0.3191	0.065
H(16)	2*i*	0.5109	0.2976	–0.0552	0.065
H(17)	2*i*	0.7537	0.3011	–0.0966	0.065
H(18)	2*i*	0.8839	0.3491	0.0386	0.065
H(19)	2*i*	0.7778	0.4146	0.2327	0.065
H(22)	2*i*	–0.0977	0.5927	0.1806	0.065
H(23)	2*i*	–0.2340	0.6982	0.0234	0.065
H(24)	2*i*	–0.2644	0.9024	–0.0396	0.065
H(25)	2*i*	–0.1632	1.0303	0.0615	0.065

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}
Pb(1)	2*i*	0.24095(2)	0.48169(2)	0.49051(1)	0.0482(1)	0.0434(1)	0.0456(1)	−0.01010(7)	−0.00476(8)	−0.00632(7)
N(1)	2*i*	0.3982(5)	0.2592(4)	0.6013(4)	0.059(3)	0.045(2)	0.054(2)	−0.009(2)	−0.006(2)	−0.010(2)
N(2)	2*i*	0.1927(5)	0.2759(4)	0.4443(4)	0.053(2)	0.051(2)	0.062(3)	−0.013(2)	0.000(2)	−0.018(2)
O(1)	2*i*	0.3519(6)	0.4565(6)	0.3061(4)	0.078(3)	0.140(5)	0.069(3)	−0.026(3)	0.011(2)	−0.027(3)
O(2)	2*i*	0.5547(7)	0.4243(6)	0.3853(4)	0.150(5)	0.106(4)	0.055(3)	−0.055(4)	−0.003(3)	−0.028(3)
O(3)	2*i*	0.1349(6)	0.7263(5)	0.3545(5)	0.085(3)	0.079(3)	0.098(4)	−0.039(3)	−0.046(3)	0.019(3)
O(4)	2*i*	0.0178(4)	0.5750(4)	0.3708(3)	0.062(2)	0.057(2)	0.057(2)	−0.015(2)	−0.003(2)	−0.001(2)
F(1)	2*i*	0.373(1)	0.329(1)	0.1325(8)	0.184(8)	0.26(1)	0.187(8)	−0.091(7)	−0.013(6)	−0.096(7)
F(2)	2*i*	−0.003(1)	0.9441(7)	0.2208(7)	0.24(1)	0.101(5)	0.167(7)	−0.045(5)	−0.083(7)	0.006(4)
C(1)	2*i*	0.5045(8)	0.2509(6)	0.6730(6)	0.086(4)	0.050(3)	0.069(4)	−0.009(3)	−0.017(3)	−0.011(3)
C(2)	2*i*	0.5741(9)	0.1313(7)	0.7414(6)	0.093(5)	0.066(4)	0.066(4)	0.001(3)	−0.031(4)	−0.012(3)
C(3)	2*i*	0.5337(9)	0.0273(7)	0.7330(7)	0.088(5)	0.052(3)	0.066(4)	−0.001(3)	−0.010(3)	0.001(3)
C(4)	2*i*	0.4246(7)	0.0314(5)	0.6579(5)	0.073(4)	0.043(3)	0.061(3)	−0.011(2)	0.010(3)	−0.004(2)
C(5)	2*i*	0.3832(9)	−0.0780(6)	0.6386(7)	0.094(5)	0.042(3)	0.091(5)	−0.020(3)	0.005(4)	0.001(3)
C(6)	2*i*	0.2863(9)	−0.0723(6)	0.5609(8)	0.087(5)	0.048(3)	0.102(6)	−0.030(3)	0.015(4)	−0.017(3)
C(7)	2*i*	0.2181(7)	0.0472(6)	0.4921(6)	0.061(3)	0.053(3)	0.084(4)	−0.023(2)	0.011(3)	−0.021(3)
C(8)	2*i*	0.1209(8)	0.0564(8)	0.4067(8)	0.071(4)	0.082(5)	0.116(6)	−0.035(4)	−0.002(4)	−0.047(4)
C(9)	2*i*	0.0635(8)	0.1736(7)	0.3405(7)	0.073(4)	0.078(4)	0.097(5)	−0.021(3)	−0.020(4)	−0.033(4)
C(10)	2*i*	0.1015(7)	0.2810(6)	0.3631(6)	0.068(4)	0.061(3)	0.082(4)	−0.009(3)	−0.022(3)	−0.018(3)
C(11)	2*i*	0.2524(6)	0.1607(5)	0.5082(5)	0.050(3)	0.047(3)	0.061(3)	−0.013(2)	0.009(2)	−0.016(2)
C(12)	2*i*	0.3597(6)	0.1518(5)	0.5919(4)	0.055(3)	0.043(2)	0.050(3)	−0.010(2)	0.009(2)	−0.010(2)
C(13)	2*i*	0.4846(7)	0.4238(5)	0.3006(5)	0.080(4)	0.038(2)	0.054(3)	−0.018(2)	0.011(3)	−0.008(2)
C(14)	2*i*	0.5659(6)	0.3869(4)	0.1883(4)	0.061(3)	0.041(2)	0.044(2)	−0.011(2)	−0.002(2)	−0.000(2)
C(15)	2*i*	0.4986(8)	0.3498(7)	0.1049(6)	0.069(4)	0.083(4)	0.063(4)	−0.030(3)	0.007(3)	−0.021(3)
C(16)	2*i*	0.563(1)	0.3190(8)	−0.0012(6)	0.109(6)	0.110(6)	0.054(4)	−0.027(5)	−0.002(4)	−0.032(4)
C(17)	2*i*	0.707(1)	0.321(1)	−0.0250(7)	0.096(6)	0.105(6)	0.059(4)	−0.005(5)	0.019(4)	−0.011(4)
C(18)	2*i*	0.7850(9)	0.353(1)	0.0590(9)	0.068(5)	0.114(7)	0.112(7)	−0.015(4)	0.034(5)	−0.010(5)
C(19)	2*i*	0.727(1)	0.3923(8)	0.178(1)	0.127(7)	0.084(5)	0.161(9)	0.010(5)	0.105(7)	0.033(6)
C(20)	2*i*	0.0421(6)	0.6803(6)	0.3233(5)	0.055(3)	0.058(3)	0.050(3)	−0.011(2)	−0.007(2)	0.004(2)
C(21)	2*i*	−0.0467(6)	0.7503(6)	0.2197(5)	0.042(2)	0.073(3)	0.045(3)	−0.015(2)	0.000(2)	0.001(2)
C(22)	2*i*	−0.1100(9)	0.680(1)	0.1580(7)	0.085(5)	0.18(1)	0.072(5)	−0.070(6)	−0.018(4)	−0.002(5)
C(23)	2*i*	−0.191(1)	0.745(2)	0.063(1)	0.128(9)	0.25(2)	0.099(8)	−0.09(1)	−0.059(7)	0.011(9)
C(24)	2*i*	−0.210(1)	0.865(2)	0.026(1)	0.109(8)	0.27(2)	0.097(8)	−0.05(1)	−0.052(6)	0.07(1)
C(25)	2*i*	−0.149(1)	0.943(1)	0.084(1)	0.101(7)	0.15(1)	0.113(8)	0.002(7)	−0.017(6)	0.074(8)
C(26)	2*i*	−0.0666(9)	0.8754(8)	0.1772(7)	0.080(4)	0.084(5)	0.080(5)	−0.022(4)	−0.022(4)	0.012(4)

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