Crystal structure of (2,2′-bipyridine-*N*,*N*′)bis(2-fluorobenzoato)lead(II), $Pb(C_7H_4O_2F)_2(C_{10}H_8N_2)$

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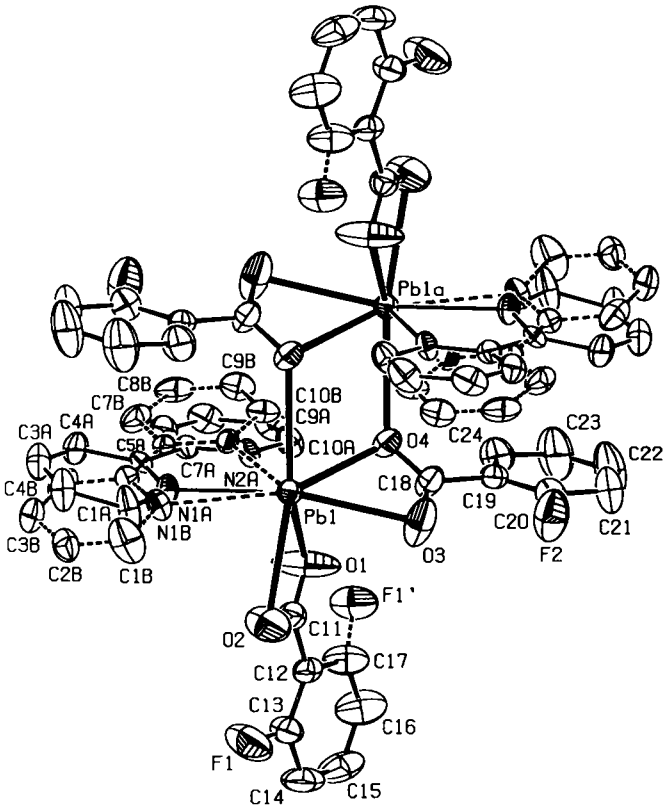

Abstract

$C_{24}H_{16}F_2N_2O_4Pb$, triclinic, $P\bar{1}$ (no. 2), a = 9.494(2) Å, b = 10.231(2) Å, c = 11.996(2) Å, α = 82.84(3)°, β = 85.53(3)°, γ = 70.25(3)°, V = 1087.3 Å^3, Z = 2, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.063, T = 293 K.

Source of material

Freshly prepared $PbCO_3$ (0.12 g, 0.42 mmol), 2,2′-bipyridine (0.04 g, 0.26 mmol), 2-fluorobenzoic acid (0.04 g, 0.29 mmol), 15 mL CH_3OH/H_2O (1:2, *v*/*v*) were mixed and stirred for ca. 1.5 h. Subsequently, the resulting suspension was heated in a 23 ml Teflon-lined stainless steel autoclave at 423 K for 7 day. After the autoclave was cooled to room temperature, the solid was filtered off. The resulting yellowish filtrate was allowed to stand at room temperature and slow evaporation for a month afforded yellowish block-shaped crystals.

Experimental details

The bipyridine molecule and one fluorobenzoato unit were found to be disordered. While both parts were resolved without the use of geometrical restraints, the occupancy of bpy only was refined.

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Discussion

The title compound has a crystal structure being similar to the crystal structure of $[Pb(FC_6H_4COO)_2(C_{12}H_8N_2)]$ [1]. Within the $[Pb(C_7H_4O_2F)_2(C_{10}H_8N_2)]$ molecules, each Pb atom is coordinated by two N atoms from one bidentate chelating 2,2′-bipyridine ligands and two O atoms from two *o*-fluorobenzoic acid anion ligands, to complete a significantly distorted PbN_2O_2 polyhedron with *d*(Pb—N) = 2.521(6) Å – 2.637(6) Å, *d*(Pb—O) = 2.313(7) Å – 2.515(5) Å, the [Pb(2,2′-bpy)-$(C_7H_4O_2F)_2$] molecules form 1D chains along the *a* axis via weak Pb—O and Pb···Pb interactions with *d*(Pb—O2) = 2.835 Å, *d*(Pb—O3) = 2.807 Å, *d*(Pb—O2′) = 3.173 Å, *d*(Pb—O4″) = 2.898 Å, *d*(Pb···F1′) = 3.526 Å, *d*(Pb···Pb) = 4.491 Å. The chains are connected to each other via weak intermolecular hydrogen bonds between the 2-fluorobenzoic acid anions O atoms, F atoms and 2,2′-bipyridine H atoms with *d*(C8···O3^{i}) = 3.399 Å, *d*(C9···F2^{i}) = 3.269 Å, *d*(C23···F1^{ii}) = 3.230 Å, ∠C8–H8···O3^{i} = 167.2°, ∠C9–H9···F2^{i} = 136.4°, ∠C23–H23···F1^{ii} = 133.7° (i: *x*,1+*y*,*z*; ii: 2–*x*,1–*y*,2–*z*). Through the week Pb—O, Pb···Pb and hydrogen bond interactions the molecules are interlinked to form the 3D network.

Table 1. Data collection and handling.

Crystal:	yellowish block, size 0.13 × 0.25 × 0.33 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	78.09 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10774, 4932
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4285
$N(param)_{refined}$:	411
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(1A)	2*i*	0.52	0.4830	0.6905	0.3075	0.092
H(2A)	2*i*	0.52	0.4229	0.8731	0.1546	0.102
H(3A)	2*i*	0.52	0.4725	1.0737	0.1692	0.098
H(4A)	2*i*	0.52	0.6006	1.0907	0.3164	0.090
H(7A)	2*i*	0.52	0.6949	1.0967	0.4710	0.073
H(8A)	2*i*	0.52	0.8291	1.0917	0.6244	0.087
H(9A)	2*i*	0.52	0.9469	0.8780	0.7252	0.093
H(10A)	2*i*	0.52	0.9160	0.6824	0.6765	0.076
H(1B)	2*i*	0.48	0.4333	0.6632	0.3188	0.123
H(2B)	2*i*	0.48	0.2852	0.8494	0.2183	0.089
H(3B)	2*i*	0.48	0.3276	1.0578	0.1987	0.102
H(4B)	2*i*	0.48	0.5042	1.0860	0.3018	0.090
H(7B)	2*i*	0.48	0.6689	1.1057	0.4038	0.081

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(8B)	2i	0.48	0.8530	1.1093	0.5191	0.085
H(9B)	2i	0.48	0.9941	0.9068	0.6154	0.079
H(10B)	2i	0.48	0.9560	0.7030	0.5944	0.067
H(13)	2i	0.25	0.2295	0.6528	0.7481	0.100
H(14)	2i		0.1055	0.6922	0.9218	0.134
H(15)	2i		0.2362	0.7061	1.0706	0.139

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(16)	2i		0.4809	0.6917	1.0461	0.148
H(17)	2i	0.75	0.6022	0.6478	0.8764	0.115
H(21)	2i		1.1786	0.0012	0.9472	0.140
H(22)	2i		1.2608	0.1456	1.0356	0.191
H(23)	2i		1.2047	0.3762	0.9732	0.172
H(24)	2i		1.0780	0.4644	0.8077	0.111

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}
Pb(1)	2i		0.75171(2)	0.54284(2)	0.50248(2)	0.0450(1)	0.0369(1)	0.0516(1)	−0.01242(7)	−0.00011(7)	−0.00612(8)
O(1)	2i		0.6368(8)	0.620(1)	0.6709(6)	0.119(5)	0.266(9)	0.168(6)	−0.129(6)	0.091(5)	−0.150(7)
O(2)	2i		0.4620(7)	0.584(1)	0.6053(5)	0.100(4)	0.28(1)	0.056(3)	−0.044(5)	0.000(3)	−0.022(4)
O(3)	2i		0.8516(8)	0.3068(7)	0.6594(6)	0.181(6)	0.140(5)	0.152(5)	−0.129(5)	−0.111(5)	0.079(4)
O(4)	2i		0.9762(5)	0.4546(4)	0.6239(3)	0.078(3)	0.066(2)	0.055(2)	−0.037(2)	−0.004(2)	0.003(2)
F(1)	2i	0.75(1)	0.2053(8)	0.656(1)	0.7258(7)	0.074(5)	0.23(1)	0.139(7)	−0.039(5)	−0.013(4)	−0.031(6)
F(1')	2i	0.25	0.628(2)	0.672(3)	0.864(1)	0.10(1)	0.20(2)	0.08(1)	−0.09(1)	−0.005(9)	−0.04(1)
F(2)	2i		1.0244(8)	0.0663(6)	0.7797(5)	0.193(6)	0.099(4)	0.147(5)	−0.064(4)	−0.068(4)	0.022(3)
N(1A)	2i	0.52(1)	0.609(2)	0.772(2)	0.386(1)	0.057(8)	0.041(7)	0.040(5)	−0.012(5)	−0.023(6)	−0.005(4)
C(1A)	2i	0.52	0.517(2)	0.766(1)	0.305(2)	0.09(1)	0.039(6)	0.10(1)	−0.001(6)	−0.08(1)	−0.021(6)
C(2A)	2i	0.52	0.473(2)	0.881(2)	0.216(1)	0.11(1)	0.058(8)	0.073(9)	−0.001(8)	−0.036(9)	−0.004(7)
C(3A)	2i	0.52	0.505(2)	0.998(2)	0.224(2)	0.09(1)	0.060(8)	0.07(1)	−0.007(7)	−0.021(9)	0.016(8)
C(4A)	2i	0.52	0.583(2)	1.008(1)	0.310(1)	0.08(1)	0.043(6)	0.09(1)	−0.016(7)	−0.005(9)	0.015(6)
C(5A)	2i	0.52	0.637(2)	0.891(2)	0.391(2)	0.03(1)	0.039(6)	0.05(1)	−0.011(8)	0.003(7)	−0.010(7)
N(2A)	2i	0.52	0.790(2)	0.765(2)	0.544(1)	0.045(6)	0.032(5)	0.062(8)	−0.008(4)	−0.013(8)	−0.002(8)
C(6A)	2i	0.52	0.729(2)	0.890(3)	0.486(2)	0.040(7)	0.047(9)	0.05(1)	−0.010(6)	0.001(8)	−0.01(1)
C(7A)	2i	0.52	0.741(2)	1.013(1)	0.514(1)	0.062(8)	0.038(6)	0.08(1)	−0.013(5)	−0.004(8)	−0.005(7)
C(8A)	2i	0.52	0.821(1)	1.010(1)	0.604(2)	0.062(8)	0.047(6)	0.12(1)	−0.028(6)	0.006(8)	−0.032(8)
C(9A)	2i	0.52	0.889(2)	0.884(1)	0.665(2)	0.065(8)	0.061(7)	0.11(1)	−0.017(6)	−0.020(8)	−0.026(8)
C(10A)	2i	0.52	0.871(1)	0.767(1)	0.634(1)	0.061(7)	0.045(6)	0.09(1)	−0.014(5)	−0.022(7)	−0.009(7)
N(1B)	2i	0.48	0.565(1)	0.763(1)	0.371(1)	0.044(8)	0.032(5)	0.07(1)	−0.007(5)	−0.026(7)	−0.014(5)
C(1B)	2i	0.48	0.449(1)	0.749(1)	0.314(1)	0.10(2)	0.09(1)	0.14(2)	−0.03(1)	−0.06(1)	−0.04(1)
C(2B)	2i	0.48	0.364(2)	0.857(2)	0.255(1)	0.07(1)	0.074(9)	0.067(9)	−0.008(8)	−0.021(8)	0.000(7)
C(3B)	2i	0.48	0.386(2)	0.983(2)	0.246(1)	0.09(1)	0.09(1)	0.056(8)	−0.02(1)	−0.020(9)	0.023(8)
C(4B)	2i	0.48	0.493(2)	0.999(2)	0.304(2)	0.10(2)	0.058(8)	0.06(1)	−0.03(1)	0.01(1)	0.004(8)
C(5B)	2i	0.48	0.586(2)	0.884(3)	0.368(2)	0.05(1)	0.046(9)	0.046(9)	−0.02(1)	0.000(8)	−0.004(7)
N(2B)	2i	0.48	0.790(2)	0.779(2)	0.493(1)	0.045(6)	0.038(7)	0.045(7)	−0.016(5)	−0.001(7)	0.002(8)
C(6B)	2i	0.48	0.703(2)	0.898(2)	0.438(2)	0.04(1)	0.039(7)	0.05(1)	−0.013(8)	0.008(9)	−0.00(1)
C(7B)	2i	0.48	0.727(2)	1.024(2)	0.445(2)	0.08(1)	0.043(7)	0.08(1)	−0.023(6)	0.012(9)	−0.001(8)
C(8B)	2i	0.48	0.837(2)	1.026(2)	0.513(2)	0.07(1)	0.065(9)	0.10(1)	−0.049(8)	0.023(9)	−0.030(9)
C(9B)	2i	0.48	0.920(2)	0.907(2)	0.569(1)	0.061(8)	0.075(9)	0.08(1)	−0.039(7)	0.000(7)	−0.025(8)
C(10B)	2i	0.48	0.896(2)	0.785(1)	0.557(1)	0.049(7)	0.061(7)	0.064(8)	−0.024(6)	0.013(6)	−0.022(7)
C(11)	2i		0.5133(7)	0.6153(5)	0.6802(6)	0.064(4)	0.041(3)	0.075(4)	−0.016(2)	0.020(3)	−0.012(3)
C(12)	2i		0.4285(6)	0.6447(5)	0.7921(5)	0.061(3)	0.036(2)	0.076(4)	−0.020(2)	0.014(3)	−0.009(3)
C(13)	2i		0.2821(8)	0.6590(7)	0.8084(7)	0.056(4)	0.076(4)	0.109(6)	−0.017(3)	0.012(4)	−0.002(4)
C(14)	2i		0.207(1)	0.6830(9)	0.913(1)	0.075(5)	0.090(6)	0.162(9)	−0.028(4)	0.060(6)	−0.020(6)
C(15)	2i		0.284(1)	0.6924(9)	1.0003(9)	0.147(9)	0.087(5)	0.120(8)	−0.052(6)	0.076(7)	−0.041(6)
C(16)	2i		0.430(1)	0.682(1)	0.9864(8)	0.143(9)	0.153(9)	0.099(6)	−0.074(7)	0.048(6)	−0.071(6)
C(17)	2i		0.501(1)	0.6570(9)	0.8845(7)	0.094(6)	0.112(6)	0.103(6)	−0.056(5)	0.034(5)	−0.057(5)
C(18)	2i		0.9484(8)	0.3523(7)	0.6813(5)	0.088(4)	0.077(4)	0.057(3)	−0.046(4)	−0.018(3)	0.015(3)
C(19)	2i		1.0414(6)	0.2880(6)	0.7820(5)	0.046(3)	0.073(4)	0.051(3)	−0.019(3)	0.000(2)	−0.001(3)
C(20)	2i		1.0723(8)	0.1518(8)	0.8266(6)	0.070(4)	0.077(4)	0.082(5)	−0.020(3)	−0.010(3)	−0.008(4)
C(21)	2i		1.156(1)	0.095(1)	0.9205(8)	0.123(7)	0.085(6)	0.116(7)	0.001(5)	−0.049(6)	0.014(5)
C(22)	2i		1.204(1)	0.181(1)	0.972(1)	0.17(1)	0.14(1)	0.15(1)	−0.011(8)	−0.107(9)	0.006(8)
C(23)	2i		1.174(1)	0.319(1)	0.9341(9)	0.17(1)	0.136(9)	0.132(8)	−0.038(8)	−0.090(8)	−0.010(7)
C(24)	2i		1.0948(8)	0.372(1)	0.8365(6)	0.087(5)	0.117(6)	0.077(5)	−0.036(5)	−0.026(4)	−0.006(4)

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