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Crystal structure of (2-(4-fluorophenyl)-1*H*-imidazo[4,5-*f*][1,10]-phenanthroline)-(5-nitro-1,3-benzenedicarboxylato)lead(II) sesquihydrate, $Pb(C_{19}H_{11}N_4F)_2(C_8H_3NO_6) \cdot 1.5H_2O$

Xiu-Yan Wang*[,I,II], Shuai Ma[I,II] and Yu He[I,II]

[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, Siping 136000, P. R. China

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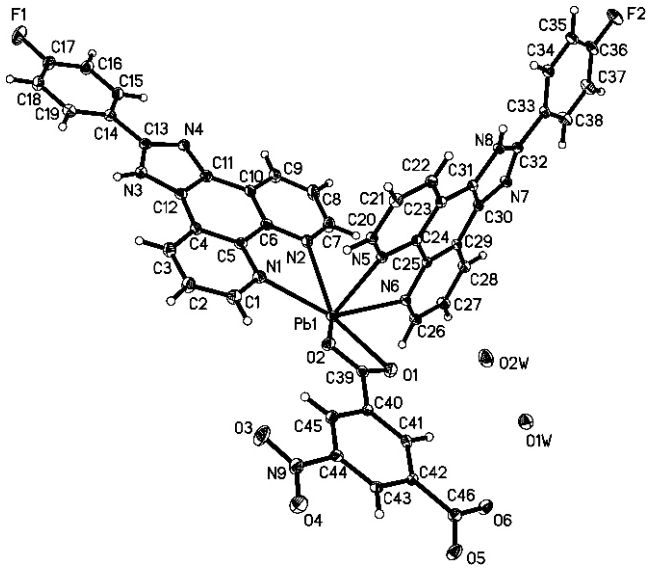

Abstract

$C_{92}H_{56}F_4N_{18}O_{15}Pb_2$, monoclinic, *P*12/*c*1 (no. 13), $a = 18.290(1)$ Å, $b = 10.6611(6)$ Å, $c = 20.872(1)$ Å, $\beta = 100.122(1)°$, $V = 4006.6$ Å^3, $Z = 2$, $R_{gt(F)} = 0.039$, $wR_{ref(F^2)} = 0.096$, $T = 293$ K.

Source of material

The pH value of a mixture of $Pb(NO_3)_2$ (0.5 mmol), 5-nitro-1,3-benzenedicarboxylic acid (1,3-H_2bdc, 0.5 mmol) and 2-(4-fluorophenyl)-1*H*-imidazo[4,5-*f*][1,10]phenanthroline (L, 1 mmol) in 13 mL distilled water was adjusted between 5 and 6 by addition of triethylamine. The resultant solution was heated at 462 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 by filtration, washed several times with distilled water and dried in air at ambient temperature (28 % yield 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). The hydrogen atoms of O1W and O2W were not located from the difference Fourier maps.

* Correspondence author (e-mail: wangxiuyan2001@yahoo.com.cn)

Discussion

1,10-Phenanthroline (phen) is a common ligand in the field of coordination complex due to its strong ability to coordinate the metal atoms. There are many reports on the coordination compounds constructed from phen or its derivatives [1].

In the title crystal structure, the central Pb(II) atom is six-coordinated by four nitrogen atoms from two different ligands, and two carboxylate oxygen atoms from one 1,3-bdc anion in a distorted octahedral manner. The Pb—O and Pb—N bond lengths are in the normal ranges. In addition, the hydrogen bonds N3–H3A···O2W (d(N3–H3A···O2W) = 2.864(7) Å, ∠N3–H3A···O2W = 172.0°) and N8–H8A···O5 (d(N8–H8A···O5) = 2.727(6) Å, ∠N8–H8A···O5 = 161.9°) stabilize the molecular packing.

Table 1. Data collection and handling.

Crystal:	pale yellow block, size 0.18 × 0.23 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	42.90 cm^{-1}
Diffractometer, scan mode:	Bruker APEX CCD, φ/ω
$2\theta_{max}$:	50.12°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20302, 7070
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5631
$N(param)_{refined}$:	591
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*g*	0.5585	0.4277	–0.0707	0.064
H(2)	4*g*	0.5313	0.5474	–0.1636	0.065
H(3)	4*g*	0.6104	0.7074	–0.1810	0.059
H(7)	4*g*	0.8533	0.4791	0.1312	0.070
H(8)	4*g*	0.9432	0.6289	0.1223	0.075
H(9)	4*g*	0.9218	0.7719	0.0391	0.065
H(15)	4*g*	0.9349	1.0367	–0.1130	0.060
H(16)	4*g*	0.9781	1.2036	–0.1681	0.071
H(18)	4*g*	0.7864	1.2147	–0.2926	0.073
H(19)	4*g*	0.7421	1.0475	–0.2388	0.067
H(20)	4*g*	0.5738	0.5349	0.0911	0.047
H(21)	4*g*	0.5485	0.6751	0.1671	0.051
H(22)	4*g*	0.6264	0.6889	0.2674	0.048
H(26)	4*g*	0.8244	0.1345	0.1479	0.054
H(27)	4*g*	0.9183	0.1391	0.2368	0.056
H(28)	4*g*	0.9172	0.2937	0.3148	0.053
H(34)	4*g*	0.7986	0.8243	0.4452	0.049

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(35)	4*g*	0.8616	0.9312	0.5344	0.057
H(37)	4*g*	1.0063	0.6483	0.5721	0.071
H(38)	4*g*	0.9451	0.5422	0.4816	0.067
H(41)	4*g*	0.5040	0.0075	0.1185	0.043
H(43)	4*g*	0.3023	–0.0450	0.0114	0.042
H(45)	4*g*	0.4297	0.2397	–0.0303	0.047
H(3A)	4*g*	0.7107	0.8734	–0.1846	0.051
H(8A)	4*g*	0.7341	0.6915	0.3666	0.039

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)	4*g*	0.5917(3)	0.4903(6)	–0.0777(3)	0.039(4)	0.062(4)	0.058(4)	–0.018(3)	0.003(3)	0.004(3)
C(2)	4*g*	0.5744(4)	0.5628(6)	–0.1337(3)	0.042(4)	0.067(4)	0.050(4)	–0.010(3)	–0.004(3)	0.011(3)
C(3)	4*g*	0.6214(4)	0.6568(6)	–0.1442(3)	0.042(4)	0.058(4)	0.046(4)	–0.001(3)	0.004(3)	0.002(3)
C(4)	4*g*	0.6865(3)	0.6761(5)	–0.0990(3)	0.041(3)	0.046(3)	0.032(3)	–0.006(3)	0.007(3)	–0.003(3)
C(5)	4*g*	0.7013(3)	0.5956(5)	–0.0433(3)	0.035(3)	0.038(3)	0.043(3)	–0.007(3)	0.008(3)	–0.003(3)
C(6)	4*g*	0.7703(3)	0.6080(5)	0.0036(3)	0.046(4)	0.043(3)	0.038(3)	–0.011(3)	0.008(3)	–0.006(3)
C(7)	4*g*	0.8448(4)	0.5351(7)	0.0965(3)	0.053(4)	0.078(5)	0.040(3)	–0.025(4)	–0.003(3)	0.012(3)
C(8)	4*g*	0.8995(4)	0.6263(7)	0.0919(3)	0.054(4)	0.086(5)	0.043(4)	–0.033(4)	–0.003(3)	0.003(4)
C(9)	4*g*	0.8870(4)	0.7097(6)	0.0425(3)	0.051(4)	0.067(4)	0.043(4)	–0.028(3)	0.006(3)	–0.002(3)
C(10)	4*g*	0.8218(4)	0.7023(5)	–0.0033(3)	0.050(4)	0.047(3)	0.035(3)	–0.016(3)	0.006(3)	–0.004(3)
C(11)	4*g*	0.8047(3)	0.7840(6)	–0.0592(3)	0.044(4)	0.045(3)	0.044(3)	–0.011(3)	0.010(3)	–0.005(3)
C(12)	4*g*	0.7405(3)	0.7698(5)	–0.1041(3)	0.040(3)	0.038(3)	0.040(3)	–0.002(3)	0.011(3)	–0.005(3)
C(13)	4*g*	0.8082(4)	0.9230(5)	–0.1317(3)	0.050(4)	0.038(3)	0.042(3)	–0.009(3)	0.017(3)	–0.003(3)
C(14)	4*g*	0.8323(4)	1.0285(5)	–0.1691(3)	0.056(4)	0.040(3)	0.041(3)	0.000(3)	0.013(3)	–0.003(3)
C(15)	4*g*	0.9045(4)	1.0730(6)	–0.1486(3)	0.055(4)	0.050(3)	0.043(3)	–0.014(3)	0.001(3)	0.005(3)
C(16)	4*g*	0.9306(4)	1.1716(6)	–0.1816(3)	0.063(5)	0.057(4)	0.059(4)	–0.013(4)	0.013(4)	0.010(3)
C(17)	4*g*	0.8856(5)	1.2199(6)	–0.2337(3)	0.084(6)	0.050(4)	0.050(4)	0.000(4)	0.031(4)	0.009(3)
C(18)	4*g*	0.8156(5)	1.1783(6)	–0.2564(3)	0.082(6)	0.056(4)	0.043(4)	0.008(4)	0.010(4)	0.009(3)
C(19)	4*g*	0.7893(4)	1.0796(6)	–0.2238(3)	0.066(5)	0.052(4)	0.048(4)	–0.005(3)	0.006(4)	–0.005(3)
C(20)	4*g*	0.6052(3)	0.5379(5)	0.1313(3)	0.038(3)	0.046(3)	0.032(3)	–0.011(3)	–0.002(3)	0.002(3)
C(21)	4*g*	0.5899(3)	0.6233(6)	0.1767(3)	0.029(3)	0.048(3)	0.051(4)	0.002(3)	0.003(3)	0.003(3)
C(22)	4*g*	0.6361(3)	0.6319(5)	0.2362(3)	0.040(3)	0.039(3)	0.040(3)	0.003(3)	0.003(3)	0.002(3)
C(23)	4*g*	0.6983(3)	0.5521(5)	0.2487(2)	0.029(3)	0.035(3)	0.032(3)	–0.002(2)	0.005(2)	0.000(2)
C(24)	4*g*	0.7083(3)	0.4626(5)	0.2004(2)	0.028(3)	0.033(3)	0.029(3)	–0.006(2)	0.004(2)	0.001(2)
C(25)	4*g*	0.7686(3)	0.3732(5)	0.2126(2)	0.036(3)	0.033(3)	0.029(3)	–0.003(2)	0.006(2)	0.003(2)
C(26)	4*g*	0.8244(4)	0.1985(5)	0.1782(3)	0.057(4)	0.039(3)	0.042(3)	0.004(3)	0.016(3)	–0.013(3)
C(27)	4*g*	0.8809(4)	0.1991(5)	0.2320(3)	0.046(4)	0.044(3)	0.050(4)	0.010(3)	0.010(3)	–0.006(3)
C(28)	4*g*	0.8802(3)	0.2907(5)	0.2780(3)	0.042(4)	0.047(3)	0.043(3)	0.007(3)	0.004(3)	–0.004(3)
C(29)	4*g*	0.8226(3)	0.3800(5)	0.2689(2)	0.034(3)	0.035(3)	0.029(3)	0.001(2)	0.005(2)	–0.003(2)
C(30)	4*g*	0.8146(3)	0.4770(5)	0.3149(2)	0.034(3)	0.041(3)	0.025(3)	0.005(3)	–0.002(2)	0.001(2)
C(31)	4*g*	0.7538(3)	0.5549(5)	0.3060(2)	0.042(3)	0.033(3)	0.027(3)	0.001(2)	0.006(3)	0.000(2)
C(32)	4*g*	0.8304(3)	0.6027(5)	0.3957(2)	0.039(3)	0.033(3)	0.032(3)	0.004(3)	0.003(3)	–0.001(2)
C(33)	4*g*	0.8646(3)	0.6715(5)	0.4544(2)	0.036(3)	0.044(3)	0.029(3)	0.003(3)	0.002(2)	–0.002(2)
C(34)	4*g*	0.8403(3)	0.7885(5)	0.4706(3)	0.039(3)	0.047(3)	0.036(3)	0.008(3)	0.002(3)	0.000(3)
C(35)	4*g*	0.8774(4)	0.8521(6)	0.5239(3)	0.053(4)	0.047(3)	0.043(3)	–0.003(3)	0.012(3)	–0.013(3)
C(36)	4*g*	0.9380(4)	0.7972(6)	0.5613(3)	0.046(4)	0.071(4)	0.032(3)	–0.010(3)	0.000(3)	–0.020(3)
C(37)	4*g*	0.9644(4)	0.6829(6)	0.5465(3)	0.046(4)	0.080(5)	0.045(4)	0.021(4)	–0.009(3)	–0.015(3)
C(38)	4*g*	0.9276(4)	0.6199(6)	0.4926(3)	0.055(4)	0.060(4)	0.046(4)	0.023(3)	–0.007(3)	–0.013(3)
C(39)	4*g*	0.5503(3)	0.2096(5)	0.0605(3)	0.045(4)	0.047(3)	0.034(3)	–0.015(3)	0.016(3)	–0.009(3)
C(40)	4*g*	0.4785(3)	0.1368(5)	0.0471(2)	0.041(3)	0.033(3)	0.028(3)	–0.008(2)	0.012(3)	–0.001(2)
C(41)	4*g*	0.4668(3)	0.0317(5)	0.0844(2)	0.035(3)	0.043(3)	0.027(3)	0.001(3)	0.002(2)	0.000(2)
C(42)	4*g*	0.4018(3)	–0.0368(5)	0.0722(2)	0.038(3)	0.032(3)	0.031(3)	–0.006(2)	0.006(3)	0.003(2)
C(43)	4*g*	0.3468(3)	–0.0009(5)	0.0206(2)	0.032(3)	0.036(3)	0.037(3)	–0.008(2)	0.003(3)	–0.003(2)
C(44)	4*g*	0.3593(3)	0.1003(5)	–0.0163(3)	0.040(3)	0.035(3)	0.035(3)	–0.005(3)	–0.002(3)	–0.002(2)
C(45)	4*g*	0.4235(3)	0.1709(5)	–0.0044(3)	0.043(3)	0.038(3)	0.038(3)	–0.001(3)	0.011(3)	0.003(3)
C(46)	4*g*	0.3901(3)	–0.1533(5)	0.1113(3)	0.036(3)	0.043(3)	0.043(3)	–0.004(3)	–0.002(3)	0.002(3)
N(1)	4*g*	0.6525(3)	0.5052(4)	–0.0339(2)	0.036(3)	0.047(3)	0.047(3)	–0.011(2)	0.006(2)	–0.002(2)
N(2)	4*g*	0.7818(3)	0.5249(5)	0.0539(2)	0.045(3)	0.057(3)	0.034(3)	–0.018(3)	0.005(2)	0.000(2)
N(3)	4*g*	0.7435(3)	0.8596(4)	–0.1504(2)	0.047(3)	0.040(3)	0.040(3)	–0.004(2)	0.007(2)	0.002(2)
N(4)	4*g*	0.8468(3)	0.8810(4)	–0.0767(2)	0.050(3)	0.040(2)	0.040(3)	–0.016(2)	0.010(3)	–0.002(2)
N(5)	4*g*	0.6623(2)	0.4600(4)	0.1420(2)	0.029(2)	0.036(2)	0.032(2)	–0.004(2)	0.002(2)	–0.001(2)
N(6)	4*g*	0.7708(3)	0.2818(4)	0.1671(2)	0.045(3)	0.036(2)	0.032(2)	–0.005(2)	0.010(2)	–0.005(2)
N(7)	4*g*	0.8634(3)	0.5067(4)	0.3713(2)	0.040(3)	0.041(3)	0.034(2)	0.008(2)	–0.001(2)	–0.005(2)
N(8)	4*g*	0.7639(2)	0.6336(4)	0.3586(2)	0.033(3)	0.031(2)	0.033(2)	0.006(2)	0.003(2)	–0.004(2)
N(9)	4*g*	0.3005(3)	0.1321(5)	–0.0719(3)	0.052(4)	0.058(3)	0.047(3)	–0.008(3)	–0.007(3)	0.012(3)
O(1)	4*g*	0.6020(2)	0.1700(4)	0.1023(2)	0.040(3)	0.063(3)	0.047(2)	–0.018(2)	–0.002(2)	0.008(2)
O(2)	4*g*	0.5552(2)	0.3066(3)	0.0270(2)	0.042(2)	0.045(2)	0.051(2)	–0.015(2)	0.007(2)	0.004(2)
O(1W)	2*f*	½	–0.0202(6)	¼	0.068(4)	0.060(4)	0.049(3)	0	0.002(3)	0

Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(3)	4*g*	0.3076(3)	0.2289(5)	–0.1021(3)	0.078(4)	0.087(4)	0.078(4)	–0.030(3)	–0.027(3)	0.046(3)
O(2W)	4*g*	0.6278(3)	0.1173(5)	0.2384(2)	0.069(4)	0.107(4)	0.048(3)	–0.019(3)	–0.003(3)	–0.004(3)
O(4)	4*g*	0.2467(3)	0.0657(5)	–0.0844(2)	0.056(3)	0.079(3)	0.072(3)	–0.027(3)	–0.025(3)	0.020(3)
O(5)	4*g*	0.3308(2)	–0.2117(4)	0.0916(2)	0.045(3)	0.061(3)	0.062(3)	–0.022(2)	–0.007(2)	0.021(2)
O(6)	4*g*	0.4389(3)	–0.1832(4)	0.1577(2)	0.054(3)	0.056(3)	0.054(3)	–0.017(2)	–0.015(2)	0.019(2)
F(1)	4*g*	0.9106(3)	1.3208(4)	–0.2650(2)	0.107(4)	0.084(3)	0.087(3)	–0.007(3)	0.040(3)	0.040(2)
F(2)	4*g*	0.9733(2)	0.8590(4)	0.6147(2)	0.054(2)	0.094(3)	0.049(2)	–0.009(2)	–0.006(2)	–0.032(2)
Pb(1)	4*g*	0.69111(1)	0.32580(2)	0.05121(1)	0.0374(1)	0.0411(1)	0.0367(1)	–0.0080(1)	0.0049(1)	–0.0036(1)

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References

1. 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-κ^2N^4,N^5)cadmium(II)]-benzene-1,4-dicarboxylato-$\kappa^3O^1,O^{1'}:O^4$]: an inclined interpenetrating (6,3) network. Acta Crystallogr. **C65** (2009) m472-m474.
2. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.