Crystal structure of aquabis(1,10-phenanthroline)- μ -(1,1'-biphenyl-2,2'-dicarboxylato)dinitratodilead(II), $\text{Pb}_2(\text{C}_{12}\text{H}_8\text{N}_2)_2(\text{C}_{14}\text{H}_8\text{O}_4)(\text{H}_2\text{O})(\text{NO}_3)_2$

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0.36 g) and water (20 mL) in a 30 mL Teflon-lined stainless-steel reactor. The solution was heated to 430 K for three days. After the reaction system was slowly cooled to room temperature, then the yellow block crystals were collected and washed with distilled water (yield 65 %).

Discussion

Lead(II) is capable of exhibiting variable coordination with or without stereochemically active lone pair of electrons [1]. The absence of crystal field stabilization energy effects also allows the Pb(II) cations to adopt different coordinations not restricted to octahedral, tetrahedral or square planar [2]. Of these, a number of polymeric Pb(II) compounds have been structurally characterized [3–5]. Carboxylate anions possess versatile binding modes to metal cations [6], therefore, metal complexes with carboxylate anions [7,8] have been extensively studied, including several 4,4'-biphenyldicarboxylate complexes [9,10]. However, the number of complexes constructed from 2,2'-biphenyldicarboxylate anions and metal cations are still limited [11]. Thus, we have selected the Pb-bpdc-phen (bpdc = 1,1'-biphenyl-2,2'-dicarboxylate dianion and phen = 1,10-phenanthroline) system to extend this area of research and present here the crystal structure of the title compound, $[\text{Pb}_2(\text{bpdc})(\text{phen})_2(\text{NO}_3)_2(\text{H}_2\text{O})]_n$ (figure, top).

There are two different kinds of independent Pb centres bridged by bpdc ligands to form a novel *catena* chain (figure, bottom). The Pb2 centre has a deformed trigonal-bipyramidal environment completed by two O atoms belonging to two carboxylate of a bpdc ligand, two N atoms from a phen molecule and one nitrate O atom. The Pb1 centre is irregularly six-coordinated by four carboxylate O atoms from two bpdc dianions and two N atoms from a phen ligand. All Pb—O_{carboxylate} bond lengths are within the range of 2.555(4) Å – 2.700(4) Å, being typical for such distances [2]. The Pb—N bond lengths of 2.512(5) Å – 2.609(6) Å are also in the range of Pb—N_{phen} bond distances observed for related structures (2.540(4) Å – 2.599(3) Å [12,13]). The phen ligand and two carboxylate groups of different bpdc dianions are in chelating and two carboxylate O atoms from two bpdc dianions are in bridging modes.

Abstract

$\text{C}_{38}\text{H}_{26}\text{N}_6\text{O}_{11}\text{Pb}_2$, monoclinic, $P12_1/c1$ (no. 14), $a = 10.8980(7)$ Å, $b = 23.449(2)$ Å, $c = 14.814(1)$ Å, $\beta = 110.57(3)^\circ$, $V = 3544.3$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.029$, $wR_{\text{ref}}(F^2) = 0.070$, $T = 298$ K.

Source of material

The title compound was synthesized with the hydrothermal method from a mixture of 2,2'-biphenyldicarboxylate (2 mmol, 0.48 g), $\text{Pb}(\text{NO}_3)_2$ (1 mmol, 0.66 g), 1,10-phenanthroline (2 mmol,

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.08 × 0.20 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	95.62 cm ⁻¹
Diffractometer, scan mode:	Bruker APEX CCD, 600 frames, $\Delta\omega = 0.3^\circ$
$2\theta_{\text{max}}$:	50.5°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	18805, 6376
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 5124
$N(\text{param})_{\text{refined}}$:	515
Programs:	SHELX-97 [14], ORTEP-II [15]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(3)	4e	0.5877	0.3529	0.7286	0.061
H(4)	4e	0.7225	0.3530	0.8879	0.069
H(5)	4e	0.6312	0.3614	1.0075	0.062
H(6)	4e	0.4087	0.3686	0.9665	0.059
H(10)	4e	-0.0492	0.3206	0.8066	0.061
H(11)	4e	-0.1382	0.4034	0.7278	0.073
H(12)	4e	-0.0098	0.4675	0.6860	0.074
H(13)	4e	0.2116	0.4522	0.7299	0.059
H(15)	4e	0.4533	0.2463	0.5472	0.056
H(16)	4e	0.6769	0.2336	0.6068	0.065
H(17)	4e	0.8107	0.3057	0.5904	0.061
H(19)	4e	0.8329	0.4026	0.5252	0.069
H(20)	4e	0.7392	0.4812	0.4414	0.067
H(22)	4e	0.5346	0.5316	0.3390	0.068

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(23)	4e	0.3097	0.5334	0.2705	0.070
H(24)	4e	0.1896	0.4600	0.3027	0.060
H(27)	4e	0.4347	0.2445	0.7821	0.049
H(28)	4e	0.6541	0.2213	0.8295	0.060
H(29)	4e	0.7162	0.1370	0.7760	0.067
H(31)	4e	0.6498	0.0457	0.6845	0.074
H(32)	4e	0.4912	-0.0111	0.5871	0.079
H(34)	4e	0.2525	-0.0235	0.4805	0.089
H(35)	4e	0.0445	0.0133	0.4181	0.087
H(36)	4e	-0.0008	0.0981	0.4805	0.075
O(11)	4e	-0.022(1)	0.5605(7)	0.570(1)	0.233(5)
H(11A)	4e	-0.03(1)	0.542(6)	0.518(6)	0.280
H(11B)	4e	0.055(8)	0.566(7)	0.621(7)	0.280

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Pb(1)	4e	0.20278(2)	0.338966(9)	0.43820(2)	0.0379(1)	0.0370(1)	0.0408(1)	-0.00133(9)	0.0138(1)	0.00301(9)
Pb(2)	4e	0.13434(2)	0.215179(9)	0.62939(2)	0.0357(1)	0.0395(1)	0.0492(2)	0.00148(9)	0.0102(1)	0.0080(1)
N(1)	4e	0.4495(4)	0.3207(2)	0.4893(3)	0.040(3)	0.037(3)	0.040(3)	-0.004(2)	0.013(2)	-0.002(2)
N(2)	4e	0.3355(5)	0.4141(2)	0.3837(3)	0.044(3)	0.041(3)	0.043(3)	0.000(2)	0.016(3)	0.002(2)
N(3)	4e	0.3656(4)	0.1789(2)	0.6990(3)	0.038(3)	0.035(3)	0.036(3)	-0.001(2)	0.014(2)	0.001(2)
N(4)	4e	0.1761(5)	0.1153(2)	0.5679(3)	0.060(4)	0.047(3)	0.038(3)	-0.016(3)	0.019(3)	-0.003(2)
N(5)	4e	-0.0431(6)	0.1228(3)	0.6881(5)	0.056(4)	0.058(4)	0.075(5)	-0.007(3)	0.022(4)	-0.003(3)
N(6)	4e	0.9298(6)	0.2762(3)	0.4339(5)	0.037(4)	0.097(5)	0.070(5)	0.008(3)	0.021(4)	0.020(4)
O(1)	4e	0.3151(5)	0.3962(2)	0.5920(3)	0.088(4)	0.042(2)	0.039(2)	-0.015(2)	0.015(2)	-0.001(2)
O(2)	4e	0.2799(4)	0.3065(2)	0.6196(3)	0.055(3)	0.038(2)	0.041(2)	-0.016(2)	0.014(2)	-0.006(2)
O(3)	4e	0.2178(4)	0.2432(2)	0.8148(3)	0.048(2)	0.032(2)	0.048(2)	0.006(2)	0.020(2)	0.000(2)
O(4)	4e	0.1977(4)	0.2721(2)	0.9509(3)	0.041(2)	0.038(2)	0.042(2)	0.006(2)	0.012(2)	0.003(2)
O(5)	4e	0.0730(4)	0.1377(2)	0.7315(3)	0.048(3)	0.063(3)	0.054(3)	-0.010(2)	0.016(2)	0.000(2)
O(6)	4e	-0.1033(6)	0.1486(3)	0.6158(5)	0.066(4)	0.162(7)	0.106(5)	-0.004(4)	-0.020(4)	0.046(5)
O(7)	4e	-0.0891(6)	0.0829(3)	0.7181(5)	0.109(5)	0.077(4)	0.165(6)	-0.048(4)	0.064(5)	0.001(4)
O(8)	4e	0.8975(5)	0.2291(3)	0.4529(4)	0.069(4)	0.101(4)	0.096(4)	-0.023(3)	0.034(3)	0.006(3)
O(9)	4e	0.9109(6)	0.2909(3)	0.3506(4)	0.067(4)	0.164(6)	0.077(4)	0.022(4)	0.013(4)	0.049(4)
O(10)	4e	0.9882(5)	0.3093(2)	0.5027(4)	0.057(3)	0.078(4)	0.096(4)	0.007(3)	0.037(3)	0.011(3)
C(1)	4e	0.3296(6)	0.3546(2)	0.6472(4)	0.041(3)	0.041(3)	0.040(3)	-0.005(3)	0.020(3)	-0.007(3)
C(2)	4e	0.4153(6)	0.3605(2)	0.7512(4)	0.045(4)	0.028(3)	0.047(4)	-0.008(2)	0.015(3)	-0.001(2)
C(3)	4e	0.5516(6)	0.3560(2)	0.7767(5)	0.050(4)	0.045(4)	0.058(4)	-0.010(3)	0.020(4)	-0.007(3)
C(4)	4e	0.6322(6)	0.3562(3)	0.8715(5)	0.042(4)	0.048(4)	0.072(5)	-0.003(3)	0.008(4)	0.001(3)
C(5)	4e	0.5775(6)	0.3612(3)	0.9429(5)	0.050(4)	0.047(4)	0.045(4)	-0.004(3)	-0.002(3)	0.004(3)
C(6)	4e	0.4441(6)	0.3659(2)	0.9179(4)	0.053(4)	0.051(4)	0.038(4)	0.003(3)	0.010(3)	-0.003(3)
C(7)	4e	0.3603(5)	0.3667(2)	0.8223(4)	0.042(3)	0.027(3)	0.036(3)	-0.001(2)	0.008(3)	0.001(2)
C(8)	4e	0.2169(5)	0.3755(2)	0.7955(4)	0.041(3)	0.033(3)	0.033(3)	0.003(2)	0.012(3)	-0.003(2)
C(9)	4e	0.1360(6)	0.3367(2)	0.8180(4)	0.039(3)	0.038(3)	0.031(3)	0.002(3)	0.006(3)	-0.003(2)
C(10)	4e	0.0043(6)	0.3471(3)	0.7918(5)	0.046(4)	0.051(4)	0.053(4)	0.006(3)	0.015(3)	-0.001(3)
C(11)	4e	-0.0490(7)	0.3963(3)	0.7439(5)	0.044(4)	0.064(5)	0.061(5)	0.020(3)	0.002(4)	-0.002(4)
C(12)	4e	0.0274(7)	0.4347(3)	0.7200(5)	0.063(5)	0.046(4)	0.060(5)	0.026(3)	0.002(4)	0.013(3)
C(13)	4e	0.1595(6)	0.4253(2)	0.7456(4)	0.062(4)	0.040(3)	0.041(4)	0.000(3)	0.014(3)	0.000(3)
C(14)	4e	0.1897(5)	0.2808(2)	0.8655(4)	0.024(3)	0.034(3)	0.048(4)	0.002(2)	0.010(3)	0.001(3)
C(15)	4e	0.5064(6)	0.2749(2)	0.5377(4)	0.054(4)	0.037(3)	0.049(4)	-0.001(3)	0.018(3)	0.001(3)
C(16)	4e	0.6412(6)	0.2672(3)	0.5751(5)	0.048(4)	0.053(4)	0.055(4)	0.011(3)	0.008(3)	0.004(3)
C(17)	4e	0.7200(6)	0.3097(3)	0.5645(4)	0.036(4)	0.063(4)	0.048(4)	0.001(3)	0.009(3)	-0.005(3)
C(18)	4e	0.6646(6)	0.3591(3)	0.5149(4)	0.043(4)	0.051(4)	0.039(3)	-0.011(3)	0.013(3)	-0.012(3)
C(19)	4e	0.7420(6)	0.4052(3)	0.4996(5)	0.038(4)	0.081(5)	0.052(4)	-0.024(4)	0.013(3)	-0.008(4)
C(20)	4e	0.6868(6)	0.4518(3)	0.4497(5)	0.052(4)	0.057(4)	0.064(5)	-0.021(3)	0.026(4)	-0.004(3)
C(21)	4e	0.5480(6)	0.4565(2)	0.4092(4)	0.049(4)	0.045(3)	0.040(4)	-0.015(3)	0.022(3)	-0.007(3)
C(22)	4e	0.4853(7)	0.5020(3)	0.3506(5)	0.071(5)	0.045(4)	0.065(5)	-0.020(3)	0.036(4)	-0.011(3)
C(23)	4e	0.3520(7)	0.5033(3)	0.3101(5)	0.071(5)	0.043(4)	0.072(5)	-0.003(3)	0.037(4)	0.000(3)
C(24)	4e	0.2805(6)	0.4586(2)	0.3294(5)	0.045(4)	0.041(4)	0.065(4)	0.001(3)	0.023(4)	0.001(3)
C(25)	4e	0.4672(6)	0.4123(2)	0.4220(4)	0.039(4)	0.044(3)	0.037(3)	-0.006(3)	0.014(3)	-0.007(3)
C(26)	4e	0.5285(5)	0.3631(2)	0.4777(4)	0.037(3)	0.047(3)	0.030(3)	-0.006(3)	0.015(3)	-0.004(2)
C(27)	4e	0.4588(6)	0.2110(2)	0.7589(4)	0.043(4)	0.041(3)	0.040(3)	-0.001(3)	0.016(3)	0.002(3)
C(28)	4e	0.5914(6)	0.1971(3)	0.7887(4)	0.039(4)	0.063(4)	0.044(4)	-0.003(3)	0.008(3)	0.011(3)
C(29)	4e	0.6282(6)	0.1470(3)	0.7571(5)	0.044(4)	0.069(5)	0.059(4)	0.015(3)	0.023(4)	0.018(3)
C(30)	4e	0.5318(6)	0.1115(3)	0.6963(4)	0.052(4)	0.049(4)	0.042(4)	0.011(3)	0.022(3)	0.011(3)

Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(31)	4e	0.5629(8)	0.0577(3)	0.6641(5)	0.083(5)	0.050(4)	0.064(5)	0.020(4)	0.040(4)	0.006(3)
C(32)	4e	0.4693(8)	0.0243(3)	0.6053(5)	0.103(6)	0.044(4)	0.070(5)	0.016(4)	0.055(5)	-0.002(3)
C(33)	4e	0.3359(8)	0.0426(3)	0.5703(4)	0.089(5)	0.043(4)	0.044(4)	-0.010(4)	0.036(4)	0.001(3)
C(34)	4e	0.234(1)	0.0118(3)	0.5016(5)	0.129(8)	0.048(4)	0.063(5)	-0.029(5)	0.056(6)	-0.014(4)
C(35)	4e	0.1103(9)	0.0327(3)	0.4658(5)	0.101(7)	0.066(5)	0.060(5)	-0.039(5)	0.039(5)	-0.012(4)
C(36)	4e	0.0848(7)	0.0845(3)	0.5027(5)	0.066(5)	0.072(5)	0.054(4)	-0.029(4)	0.026(4)	-0.003(4)
C(37)	4e	0.3011(6)	0.0952(2)	0.6015(4)	0.059(4)	0.039(3)	0.035(3)	-0.003(3)	0.022(3)	0.003(3)
C(38)	4e	0.4012(6)	0.1294(2)	0.6674(4)	0.056(4)	0.034(3)	0.037(3)	0.002(3)	0.024(3)	0.007(2)

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