# Synthesis and crystal structure of a novel lead coordination polymer, $[[Pb_3(C_8H_3O_7S)_2(C_3H_7NO)_{2.5}(H_2O)]\cdot 1.5H_2O]_n$, $C_{47}H_{57}N_5O_{38}Pb_6S_4$

Hua-Yi Zhao, Xue-Juan Zhang and Xin-Hua Li*

College of Chemistry and Materials Engineering, Wenzhou University, Wenzhou 325035, Zhejiang Province, P. R. China

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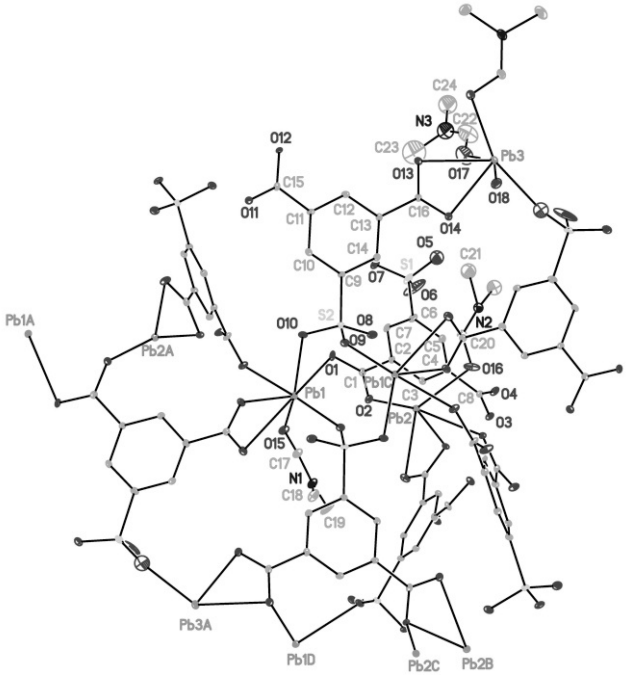


**Abstract**

$C_{47}H_{57}N_5O_{38}Pb_6S_4$, tetragonal, $I4_1/a$ (no. 88), $a = 20.142(1)$ Å, $c = 33.803(2)$ Å, $V = 13714.2$ Å$^3$, $Z = 8$, $R_{gt}(F) = 0.0365$, $wR_{ref}(F^2) = 0.1004$, $T = 298$ K.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colourless blocks, size 0.15×0.18×0.23 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 149.01 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX CCD, $\varphi$ and $\omega$ |
| $2\theta_{max}$: | 50.98° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 37396, 6397 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 5358 |
| $N(param)_{refined}$: | 469 |
| Programs: | SHELXS-97 [18] |

## Source of material

An aqueous mixture (10 ml) of Lead(II) nitrate (0.0331 g, 0.1 mmol), 5-sulfoisophthalic acid sodium salt (0.0268 g, 0.1 mmol) was added dropwise to a solution of dimethylformamide (DMF) (10 ml) containing 2,2'-dithiosalicylic acid (0.0921 g, 0.3 mmol) at room temperature. The reaction mixture was filtered and the filtrate was left to stand for about four weeks until colorless single crystals were obtained (yield 32%, based on Pb). Colourless blockshaped crystals of (I) suitable for X-ray diffraction were collected by filtration, washed with water and ethanol, and finally dried in air.

* Correspondence author (e-mail: lixinhua01@126.com)

Anal. calcd. (%) for $C_{47}H_{57}N_5O_{38}Pb_6S_4$:
H:2.15; C:21.13; N:2.62. Found: H:2.18; C:21.21; N:2.68.

## Experimental details

The uncoordinated water H atoms were refined subject to the restraint O–H = 0.82(5) Å. The other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of C–H = 0.93 Å and $U_{iso}(H) = 1.2\ U_{eq}(C)$ for Csp$^2$, C–H = 0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for Csp$^3$, and O–H = 0.82 Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

## Discussion

In recent years, the studies on the synthesis of metal-organic coordinations from metals and organic ligands have been extensively developed for their crystallographic diversity and potential applications in catalysis, nonlinear optics, magnetism, electronics, catalysis, sorption, and molecular recognition [1]. So far, various structures with interesting compositions and topologies have been produced through judicious choice of ligand and metal precursor geometry [2]. The largest class of coordination polymers are those containing carboxylate ligands and until now a variety of hybrid materials with different dimensions has been designed with the use of carboxylate groups [3]. The carboxylate group has rich coordination modes, and usually binds to a metal ion forming discrete, one-, two- and three-dimensional coordination polymers. Also lead(II) has a large radius, flexible coordination environment and various stereochemical activities, and provides unique opportunities for formation of unusual network topologies with interesting properties. Lead(II) contains the 6s$^2$ lone-pair, and the absence of crystal field stabilization energy, which can cause distortion in coordination sphere [4-9], allows for variable coordination numbers. A number of polymeric Pb(II) compounds have been structurally characterized, and frequently discussed considering the stereo-chemical activity of valence shell electron lone pairs [10-16]. In this paper, we chose the sip (5-sulfo- isophthalate) anion and the Pb(II) cation to construct a coordination polymer. Our purpose was to investigate the coordination modes of carboxylate groups in lead(II) coordination polymers, and to prepare multi-dimensional coordination networks. Hence we report here the synthesis and crystal structure of a new lead (II) coordination polymer, namely $[[Pb_3(sip)_2(DMF)_{2.5}(H_2O)]\cdot 1.5H_2O]_n$.

In the compound, there are three independent Pb(II) ions, and the coordination geometry of each Pb atom is of great difference. Three Pb(II) ions are bridged by two sip ligands forming a trimer subunit. Pb1, Pb2 and Pb3 have different coordination number, and because of the absence of crystal field-stabilization energy effects, their coordination geometries do not restricted to a polyhedron, such as octahedral, tetrahedral or square planar [17].

Atom Pb1 has a coordination number of seven, made up of seven

O atoms from three carboxylate groups, two sulfonate groups from five sip anions and one DMF molecule. The two sulfonic groups adopt the same bidentate coordination mode, bridging two Pb(II) cations by two different O atoms. The DMF molecule also adopts a bidentate coordination mode, but it bridges two Pb(II) cations by the same one O atom, different from the two sulfonate groups. The coordination modes of the three carboxylate groups are different, one is chelating, one is bidentately bridging two Pb(II) cations by two different O atoms, and the third one adopts a tridentate mode, chelating a Pb cation by two carboxylate O atoms and one of O atom also coordinates to another Pb cation.

Pb2 is five-coordinated, consisting of four carboxylate oxygen atoms from three different sips, and one oxygen atom from a DMF molecule. Just as for Pb1, carboxylate oxygen atoms adopt a tridentate mode and bidentate mode, and the DMF molecule also adopts a bidentate coordination mode. One of the four carboxylate oxygen atoms comes from a bidentate carboxylate group, another one comes from a tridentate carboxylate group, and the last two carboxylate oxygen atoms come from a different tridentate carboxylate group.

Pb3 is six-coordinated by two chelating carboxylate O atoms from a tridentate carboxylate group of a sip, a $\mu_2$-bridging DMF molecule, and three mono-coordinated O atoms from a DMF ligand, a water molecule and a monodentate sulfonic group. There are two kinds of DMF ligands, one is bidentate and the other is monodentate. The sulfonate group in Pb3 also adopts a monodentate coordinated mode, different from the findings for Pb1.

The coordination fashions of sip ligands are also different, and there are two kinds of sip ligands, one serves as $\mu_6$-bridging and the other is $\mu_4$-bridging. The Pb(II) cations bridged by sip ligands form a complex 3D network structure. There are also O–H···O hydrogen bonds, which connect the coordination polymer and uncoordinated water molecules and help to stabilize the crystal structure.

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

| Atom | Site | Occ. | *x* | *y* | *z* | $U_{iso}$ |
|---|---|---|---|---|---|---|
| H(18E) | 16*f* | | –0.317(5) | 0.917(5) | 0.164(3) | 0.02(3) |
| H(18D) | 16*f* | | –0.316(8) | 0.959(7) | 0.184(4) | 0.06(6) |
| H(19E) | 16*f* | | 0.6932 | 0.9200 | 0.0787 | 0.111 |
| H(19G) | 16*f* | | 0.6474 | 0.9186 | 0.1094 | 0.111 |
| H(20A) | 16*f* | 0.5 | 0.1641 | 0.7847 | 0.2571 | 0.146 |
| H(20B) | 16*f* | 0.5 | 0.1404 | 0.8439 | 0.2726 | 0.146 |
| H(3) | 16*f* | | 0.1418 | 0.7539 | 0.0259 | 0.024 |
| H(5) | 16*f* | | 0.2084 | 0.6637 | 0.1243 | 0.038 |
| H(7) | 16*f* | | 0.2091 | 0.8619 | 0.1180 | 0.041 |
| H(10) | 16*f* | | 0.0428 | 1.1161 | 0.1176 | 0.023 |
| H(12) | 16*f* | | –0.0796 | 1.0848 | 0.2096 | 0.027 |
| H(14) | 16*f* | | –0.0896 | 0.9663 | 0.1144 | 0.025 |
| H(17) | 16*f* | | 0.3380 | 0.9046 | –0.0303 | 0.051 |
| H(18A) | 16*f* | | 0.1781 | 0.9074 | –0.0776 | 0.094 |
| H(18B) | 16*f* | | 0.2143 | 0.9008 | –0.1185 | 0.094 |
| H(18C) | 16*f* | | 0.2191 | 0.9672 | –0.0945 | 0.094 |
| H(19A) | 16*f* | | 0.3542 | 0.8351 | –0.0799 | 0.150 |
| H(19B) | 16*f* | | 0.3151 | 0.8482 | –0.1192 | 0.150 |
| H(19C) | 16*f* | | 0.2862 | 0.7994 | –0.0874 | 0.150 |
| H(20E) | 16*f* | 0.5 | –0.0106 | 0.8280 | 0.0823 | 0.055 |
| H(21A) | 16*f* | | 0.0077 | 0.8396 | 0.1432 | 0.406 |
| H(21B) | 16*f* | | –0.0282 | 0.7859 | 0.1692 | 0.406 |
| H(21C) | 16*f* | | –0.0655 | 0.8186 | 0.1334 | 0.406 |
| H(22) | 16*f* | | –0.1108 | 0.8361 | 0.3003 | 0.179 |
| H(23A) | 16*f* | | 0.0404 | 0.8778 | 0.2507 | 0.433 |
| H(23B) | 16*f* | | 0.0556 | 0.9258 | 0.2861 | 0.433 |
| H(23C) | 16*f* | | –0.0012 | 0.9430 | 0.2560 | 0.433 |
| H(24A) | 16*f* | | 0.0166 | 0.8967 | 0.3469 | 0.271 |
| H(24B) | 16*f* | | 0.0492 | 0.8335 | 0.3280 | 0.271 |
| H(24C) | 16*f* | | –0.0211 | 0.8286 | 0.3477 | 0.271 |

**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) | 16*f* | | 0.15621(2) | 1.01263(2) | 0.000692(9) | 0.0216(2) | 0.0166(2) | 0.0220(2) | –0.0001(1) | –0.0032(1) | 0.0020(1) |
| Pb(2) | 16*f* | | 0.00888(2) | 0.84739(2) | –0.003790(9) | 0.0226(2) | 0.0185(2) | 0.0194(2) | 0.0019(1) | 0.0015(1) | –0.0003(1) |
| Pb(3) | 16*f* | | –0.22184(2) | 0.88364(2) | 0.22880(1) | 0.0462(3) | 0.0261(2) | 0.0333(2) | –0.0078(2) | 0.0163(2) | –0.0020(2) |
| S(1) | 16*f* | | 0.2401(2) | 0.7663(1) | 0.17601(9) | 0.143(4) | 0.024(2) | 0.034(2) | –0.016(2) | –0.049(2) | 0.005(1) |
| S(2) | 16*f* | | 0.0049(1) | 1.0154(1) | 0.06207(6) | 0.024(1) | 0.026(1) | 0.014(1) | –0.0007(9) | 0.0015(8) | –0.0034(8) |
| O(1) | 16*f* | | 0.1865(3) | 0.9325(3) | 0.0584(2) | 0.046(4) | 0.014(3) | 0.039(4) | –0.010(3) | –0.020(3) | 0.010(3) |
| O(2) | 16*f* | | 0.1315(3) | 0.8766(3) | 0.0129(2) | 0.039(4) | 0.018(3) | 0.028(3) | 0.002(3) | –0.013(3) | 0.001(3) |
| O(3) | 16*f* | | 0.1191(3) | 0.6292(3) | 0.0242(2) | 0.036(4) | 0.020(3) | 0.027(3) | 0.008(3) | –0.011(3) | –0.006(3) |
| O(4) | 16*f* | | 0.1888(4) | 0.5815(3) | 0.0648(2) | 0.062(5) | 0.014(3) | 0.038(4) | 0.011(3) | –0.020(3) | –0.003(3) |
| O(5) | 16*f* | | 0.1909(7) | 0.7381(7) | 0.1995(4) | 0.115(4) | 0.110(4) | 0.108(4) | –0.005(2) | –0.003(2) | 0.001(2) |
| O(6) | 16*f* | | 0.293(1) | 0.7217(6) | 0.1785(4) | 0.37(2) | 0.093(9) | 0.12(1) | 0.12(1) | –0.19(1) | –0.056(8) |
| O(7) | 16*f* | | 0.2548(5) | 0.8330(4) | 0.1851(2) | 0.104(7) | 0.031(4) | 0.032(4) | –0.014(4) | –0.025(4) | –0.003(3) |
| O(8) | 16*f* | | 0.0184(4) | 0.9457(3) | 0.0633(2) | 0.054(5) | 0.031(4) | 0.028(4) | 0.010(3) | 0.006(3) | –0.004(3) |
| O(9) | 16*f* | | –0.0503(3) | 1.0316(3) | 0.0358(2) | 0.028(4) | 0.041(4) | 0.020(3) | –0.007(3) | –0.007(3) | 0.000(3) |
| O(10) | 16*f* | | 0.0624(3) | 1.0566(3) | 0.0522(2) | 0.025(3) | 0.046(4) | 0.021(3) | –0.006(3) | 0.009(3) | –0.009(3) |
| O(11) | 16*f* | | 0.0731(3) | 1.1880(3) | 0.1789(2) | 0.029(4) | 0.038(4) | 0.031(4) | –0.016(3) | 0.010(3) | –0.010(3) |
| O(12) | 16*f* | | –0.0061(3) | 1.1800(3) | 0.2231(2) | 0.027(3) | 0.015(3) | 0.014(3) | –0.001(2) | 0.002(2) | –0.004(2) |
| O(13) | 16*f* | | –0.1734(3) | 1.0007(3) | 0.2160(2) | 0.031(4) | 0.030(4) | 0.032(4) | –0.007(3) | 0.015(3) | –0.005(3) |
| O(14) | 16*f* | | –0.1547(4) | 0.9200(3) | 0.1729(2) | 0.052(5) | 0.033(4) | 0.037(4) | –0.021(3) | 0.024(3) | –0.013(3) |
| O(15) | 16*f* | | 0.2722(3) | 0.9681(4) | –0.0220(2) | 0.032(4) | 0.049(5) | 0.045(4) | 0.007(3) | –0.004(3) | –0.017(4) |
| O(16) | 8*e* | | 0 | ¾ | 0.0532(3) | 0.053(7) | 0.12(1) | 0.023(6) | 0.043(7) | 0 | 0 |
| O(17) | 16*f* | | –0.1047(7) | 0.8741(9) | 0.2534(5) | 0.09(1) | 0.19(2) | 0.13(1) | –0.00(1) | –0.032(9) | 0.05(1) |
| O(18) | 16*f* | | –0.3101(5) | 0.9270(5) | 0.1851(3) | 0.059(6) | 0.042(6) | 0.051(6) | 0.000(5) | 0.002(4) | –0.018(5) |
| O(19) | 16*f* | | 0.6885(4) | 0.9202(5) | 0.1037(2) | 0.059(6) | 0.115(8) | 0.049(5) | 0.032(5) | 0.011(4) | 0.005(5) |
| O(20) | 16*f* | 0.5 | 0.134(1) | 0.8143(9) | 0.2551(6) | 0.16(2) | 0.05(1) | 0.09(2) | 0.03(1) | –0.03(2) | –0.01(1) |
| N(1) | 16*f* | | 0.2756(5) | 0.8951(4) | –0.0724(3) | 0.046(6) | 0.038(5) | 0.059(6) | 0.003(4) | 0.004(5) | –0.023(5) |
| N(2) | 8*e* | | 0 | ¾ | 0.1176(4) | 0.047(4) | 0.047(4) | 0.046(4) | –0.001(2) | 0 | 0 |
| N(3) | 16*f* | | –0.023(1) | 0.871(1) | 0.2943(6) | 0.144(6) | 0.144(6) | 0.144(6) | –0.000(2) | 0.000(2) | 0.000(2) |
| C(1) | 16*f* | | 0.1630(4) | 0.8799(4) | 0.0451(2) | 0.018(4) | 0.019(5) | 0.027(5) | 0.002(3) | –0.006(4) | 0.003(4) |

**Table 3.** continued.

| Atom | Site | Occ. | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| C(2) | 16*f* | | 0.1731(4) | 0.8172(4) | 0.0684(2) | 0.029(5) | 0.020(5) | 0.021(4) | –0.005(4) | –0.008(4) | 0.005(3) |
| C(3) | 16*f* | | 0.1594(4) | 0.7559(4) | 0.0513(2) | 0.019(4) | 0.020(5) | 0.021(4) | 0.000(3) | –0.006(3) | 0.006(3) |
| C(4) | 16*f* | | 0.1719(4) | 0.6977(4) | 0.0720(3) | 0.029(5) | 0.016(4) | 0.025(5) | 0.005(4) | –0.005(4) | –0.003(3) |
| C(5) | 16*f* | | 0.1984(5) | 0.7022(5) | 0.1103(3) | 0.050(6) | 0.019(5) | 0.026(5) | –0.005(4) | –0.013(4) | 0.010(4) |
| C(6) | 16*f* | | 0.2095(6) | 0.7634(5) | 0.1271(3) | 0.061(7) | 0.020(5) | 0.027(5) | 0.007(5) | –0.021(5) | 0.000(4) |
| C(7) | 16*f* | | 0.1991(5) | 0.8210(5) | 0.1067(3) | 0.054(7) | 0.021(5) | 0.027(5) | –0.011(5) | –0.018(4) | 0.000(4) |
| C(8) | 16*f* | | 0.1591(4) | 0.6314(4) | 0.0526(2) | 0.025(5) | 0.022(5) | 0.021(4) | –0.001(4) | –0.001(4) | –0.001(3) |
| C(9) | 16*f* | | –0.0208(4) | 1.0393(4) | 0.1099(2) | 0.025(5) | 0.019(4) | 0.012(4) | 0.001(4) | –0.002(3) | 0.000(3) |
| C(10) | 16*f* | | 0.0087(4) | 1.0923(4) | 0.1294(2) | 0.018(4) | 0.022(5) | 0.018(4) | 0.000(4) | –0.001(3) | 0.006(3) |
| C(11) | 16*f* | | –0.0136(4) | 1.1092(4) | 0.1671(2) | 0.022(5) | 0.025(5) | 0.012(4) | 0.000(4) | –0.004(3) | –0.002(3) |
| C(12) | 16*f* | | –0.0645(4) | 1.0731(4) | 0.1846(2) | 0.023(5) | 0.025(5) | 0.018(4) | 0.002(4) | 0.003(3) | –0.004(3) |
| C(13) | 16*f* | | –0.0932(4) | 1.0189(4) | 0.1648(2) | 0.015(4) | 0.019(4) | 0.025(5) | –0.003(3) | 0.000(3) | 0.000(3) |
| C(14) | 16*f* | | –0.0711(4) | 1.0023(4) | 0.1276(2) | 0.021(5) | 0.020(4) | 0.021(4) | –0.002(4) | –0.005(3) | –0.001(3) |
| C(15) | 16*f* | | 0.0199(4) | 1.1633(4) | 0.1905(2) | 0.016(4) | 0.013(4) | 0.028(5) | 0.001(3) | –0.002(3) | –0.001(3) |
| C(16) | 16*f* | | –0.1447(4) | 0.9768(4) | 0.1851(3) | 0.021(5) | 0.021(5) | 0.034(5) | –0.005(4) | 0.015(4) | –0.008(4) |
| C(17) | 16*f* | | 0.2985(5) | 0.9217(5) | –0.0404(3) | 0.036(6) | 0.040(6) | 0.051(7) | 0.006(5) | –0.001(5) | –0.021(5) |
| C(18) | 16*f* | | 0.2168(7) | 0.9197(6) | –0.0925(4) | 0.068(9) | 0.044(8) | 0.074(9) | 0.004(7) | –0.029(7) | –0.010(6) |
| C(19) | 16*f* | | 0.3109(8) | 0.8397(8) | –0.0914(5) | 0.08(1) | 0.09(1) | 0.14(2) | 0.011(9) | –0.01(1) | –0.09(1) |
| C(20) | 16*f* | 0.5 | –0.005(1) | 0.7807(8) | 0.0832(3) | 0.045(6) | 0.046(6) | 0.046(6) | 0.000(2) | 0.000(2) | 0.002(2) |
| C(21) | 16*f* | | –0.023(2) | 0.803(1) | 0.1429(8) | 0.27(2) | 0.27(2) | 0.27(2) | 0.000(2) | 0.000(2) | –0.001(2) |
| C(22) | 16*f* | | –0.083(1) | 0.859(2) | 0.2829(8) | 0.06(1) | 0.24(3) | 0.14(2) | 0.03(2) | –0.00(1) | 0.02(2) |
| C(23) | 16*f* | | 0.021(2) | 0.908(2) | 0.270(1) | 0.29(2) | 0.29(2) | 0.29(2) | 0.000(2) | 0.000(2) | 0.000(2) |
| C(24) | 16*f* | | 0.008(1) | 0.856(1) | 0.3325(8) | 0.181(9) | 0.181(9) | 0.180(9) | 0.000(2) | –0.001(2) | 0.000(2) |

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