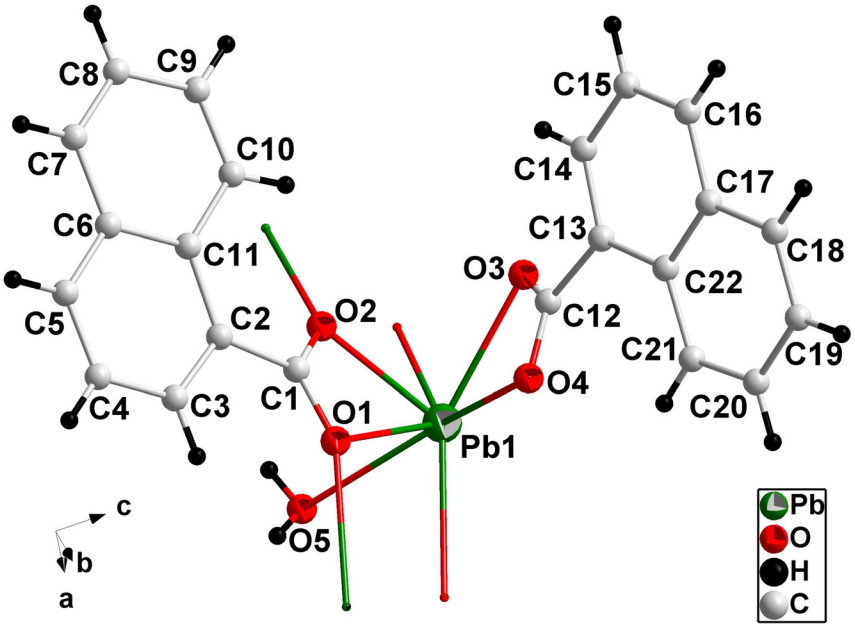
Jiahong Li, Zheyu Deng, Biao Li, Jiaqi Ou and Xiaoming Zhu*

The crystal structure of *catena*–poly [aqua(1-naphthoato-κ^2O,O')-(μ-1-naphthoato-$\kappa^4O:O,O':O'$)lead(II)], $C_{22}H_{16}O_5Pb$



https://doi.org/10.1515/ncrs-2023-0032
Received January 19, 2023; accepted February 6, 2023;
published online February 22, 2023

Abstract

$C_{22}H_{16}O_5Pb$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.3395(8) Å, b = 10.7768(11) Å, c = 23.034(3) Å, V = 1821.9(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0327, w$R_{ref}(F^2)$ = 0.0735, T = 296(2) K.

CCDC no.: 1409879

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size	0.24 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	9.29 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9946, 3545, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3423
$N(param)_{refined}$:	255
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

1 Source of material

All chemicals were purchased from commercial sources and used as received. In a typical experiment 2 mmol 1-naphthoic acid and 1 mmol lead acetate were added in CH_3OH (50 mL) and refluxed with agitating for 11 h. Then, the reaction solution was filtered. Finally, the title crystal was precipitated by controlling solvent volatilization.

***Corresponding author: Xiaoming Zhu**, School of Chemistry and Materials Science, Hengyang Normal University, Hengyang, Hunan 421008, China, E-mail: zxmhnhy@hynu.edu.cn. https://orcid.org/0000-0003-4086-4674

Jiahong Li, Zheyu Deng, Biao Li and Jiaqi Ou, School of Chemistry and Materials Science, Hengyang Normal University, Hengyang, Hunan 421008, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.73035 (4)	0.80457 (3)	0.03859 (2)	0.02730 (12)
O1	0.8849 (9)	0.6349 (7)	−0.0172 (3)	0.0349 (17)
O2	0.5902 (9)	0.6350 (6)	−0.0105 (3)	0.0363 (17)
O3	0.5682 (9)	0.6904 (9)	0.1218 (3)	0.0489 (18)
O4	0.8631 (9)	0.6604 (7)	0.1136 (3)	0.0429 (19)
O5	0.7246 (11)	0.8886 (7)	−0.0714 (3)	0.0540 (19)
H5A	0.807426	0.857132	−0.088028	0.065*
H5B	0.608026	0.882350	−0.086578	0.065*
C1	0.7344 (11)	0.5847 (8)	−0.0281 (3)	0.0246 (17)
C2	0.7265 (14)	0.4679 (7)	−0.0627 (4)	0.0283 (18)
C3	0.8715 (14)	0.4408 (9)	−0.0975 (5)	0.038 (2)
H3	0.973597	0.491835	−0.095887	0.046*
C4	0.8731 (17)	0.3401 (11)	−0.1350 (5)	0.052 (3)
H4	0.974594	0.324197	−0.158033	0.063*
C5	0.7264 (18)	0.2660 (9)	−0.1378 (5)	0.049 (3)
H5	0.726359	0.199977	−0.163848	0.058*
C6	0.5732 (14)	0.2854 (9)	−0.1026 (5)	0.037 (2)
C7	0.4220 (17)	0.2047 (11)	−0.1052 (5)	0.055 (3)
H7	0.422440	0.139519	−0.131649	0.066*
C8	0.2777 (19)	0.2204 (10)	−0.0703 (6)	0.061 (3)
H8	0.178946	0.166566	−0.072784	0.074*
C9	0.2754 (15)	0.3184 (9)	−0.0297 (5)	0.046 (2)
H9	0.175260	0.329211	−0.005481	0.056*
C10	0.4197 (13)	0.3972 (9)	−0.0260 (4)	0.033 (2)
H10	0.417855	0.460002	0.001665	0.040*
C11	0.5726 (13)	0.3865 (8)	−0.0631 (4)	0.030 (2)
C12	0.7147 (14)	0.6400 (9)	0.1386 (4)	0.033 (2)
C13	0.7076 (14)	0.5518 (8)	0.1896 (4)	0.033 (2)
C14	0.5507 (16)	0.4857 (10)	0.1966 (5)	0.045 (3)
H14	0.452933	0.499507	0.171710	0.054*
C15	0.5354 (17)	0.3962 (10)	0.2412 (6)	0.050 (3)
H15	0.429416	0.349459	0.244642	0.060*
C16	0.6722 (17)	0.3786 (11)	0.2785 (5)	0.047 (3)
H16	0.659402	0.320919	0.308289	0.057*
C17	0.8351 (15)	0.4461 (8)	0.2734 (4)	0.036 (2)
C18	0.9783 (18)	0.4313 (10)	0.3134 (5)	0.049 (3)
H18	0.965086	0.373330	0.342974	0.059*
C19	1.1332 (18)	0.4973 (12)	0.3106 (6)	0.057 (3)
H19	1.225637	0.484550	0.337564	0.068*
C20	1.1533 (16)	0.5850 (12)	0.2668 (5)	0.051 (3)
H20	1.259481	0.631894	0.264884	0.061*
C21	1.0203 (15)	0.6033 (10)	0.2268 (5)	0.041 (3)
H21	1.038048	0.661518	0.197568	0.049*
C22	0.8547 (15)	0.5354 (9)	0.2286 (4)	0.034 (2)

2 Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors U_{iso}(H) = 1.2 or 1.5 U_{eq} (parent C-atom). The absolute structure was established by refinement of the Flack parameter (−0.018(16) from 1350 selected quotients) using Parsons' method [5].

3 Comment

In recent years, rational designs and syntheses of coordination polymer of lead have attracted wide attention, owing to their rich structural aesthetics and functionalities [6–9]. Aromatic organic carboxylic acids are widely used as ligands to construct inorganic and organic hybrid materials [10–11]. The different coordination modes make the expected structures much more robust. Deprotonated carboxylate groups can form hydrogen bonds to participate in supermolecular self-assembly with coordination bonds as acceptors [12]. So, the synthesis and crystal structure of the title compound are of great significance to studying the structure and function.

Single-crystal structure analysis reveals that the title compound crystallizes in the orthorhombic space group $P2_12_12_1$ and exhibits a chain structure (see the Figure). The Pb(II) ion is seven-coordinated by six carboxylate oxygen atoms from four different 1-naphthalene carboxyl and one water molecule. The Pb–O bond lengths and O–Pb–O angles, all of which are within the range of those observed for other analogical Pb complexes [13, 14], are ranging from 2.382(7) to 2.796(7) Å and 51.1(2)° to 149.2(2)°, respectively.

Intramolecular and intermolecular hydrogen bonds exist in the crystal structure of the title compound. In an infinite chain, the oxygen atom O5 provides one intramolecular hydrogen bond to O4′ and O3″ (O5···O4′ = 2.874(1) Å; O5···O3″ = 2.903(1) Å; ′ = $x - 0.5, -y + 1.5, -z$; ″ = $x + 0.5, -y + 1.5, -z$).

It should not be unmentioned that the water free analogous structure has been already reported [15].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Hunan Provincial Natural Science Foundation of China (No. 2021JJ40010), and College Students Innovation and Entrepreneurship Training Program of Hunan Province General Project (No. cxcy2022011), Science and Technology Plan Project of Hengyang City (No. 202002042081).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. *SAINT*, *APEX2* and *SADABS*; Bruker AXS Inc.: Madison, WI, USA, 2012.
2. Sheldrick G. M. SHELXTL – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with Shelxl. *Acta Crystallogr* 2015, *C71*, 3–8.
4. Brandenburg K. *DIAMOND. Visual Crystal Structure Information System. Ver. 4.0*; Crystal Impact: Bonn, Germany, 2015.
5. Parsons S., Flack H. D., Wagner T. Use of intensity quotients and differences in absolute structure refinement. *Acta Crystallogr.* 2013, *B69*, 249–259.
6. Martínez-Casado F. J., Ramos-Riesco M., Rodríguez-Cheda J. A., Cucinotta F., Matesanz E., Miletto I., Gianotti E., Marchese L., Mateěj Z. Unraveling the decomposition process of lead(II) acetate: anhydrous polymorphs, hydrates, and byproducts and room temperature phosphorescence. *Inorg. Chem.* 2016, *55*, 8576–8586.
7. Kowalik M., Masternak J., Kazimierczuk K., Kupcewicz B., Khavryuchenkod O. V., Barszcza B. Exploring thiophene-2-acetate and thiophene-3-acetate binding modes towards the molecular, supramolecular structures and photoluminescence properties of Pb(II) polymers. *CrystEngComm* 2020, *22*, 7025–7035.
8. Chen Y., Hu C. L., Fang Z., Li Y. L., Mao J. G. From $Pb(H_2C_3N_3O_3)(OH)$ to $Pb(H_2C_3N_3O_3)F$: homovalent anion substitution-induced band gap enlargement and birefringence enhancement. *Inorg. Chem.* 2022, *61*, 1778–1786.
9. Kamakura Y., Chinapang P., Masaoka S., Saeki A., Ogasawara K., Nishitani S. R., Yoshikawa H., Katayama T., Tamai N., Sugimoto K., Tanaka D. Semiconductive nature of lead-based metal-organic frameworks with three-dimensionally extended sulfur secondary building units. *J. Am. Chem. Soc.* 2020, *142*, 27–32.
10. Fu F., Chen S.-P., Ren Y.-X., Gao S.-L. Single crystal structures and fluorescence property of a series of lanthanide coordination polymers incorporating isophthalate. *Acta Chim. Sinica* 2008, *66*, 1663–1668.
11. Yang J., Miao J., Dai J., Yang L., Zhang L.-N. Syntheses, crystal structures and properties of two lead(II) complexes with para-substituted benzoic acid and 2,2′-bipyridine co-ligand. *Chinese J. Inorg. Chem.* 2010, *26*, 1992–2000.
12. Shen W., Hu W.-J., Wu X.-Y., Zhao G.-L. Syntheses, structures and DNA interaction of Zn(II) and Pb(II) complexes based on imidazo-phenanthrolin-phenoxy acetic acid. *Chinese J. Inorg. Chem.* 2016, *32*, 1101–1110.
13. Sarma R., Jubaraj B., Baruah J. B. Solvent coordination in changing dimensionality of lead benzoate coordination polymers. *Inorg. Chim. Acta* 2009, *362*, 4977–4984.
14. Xue J.-L., Tian C.-H. Lead(II) coordination polymer with 4-chlorophenoxyacetate as ligand: structure, luminescence and biological bctivity. *Chinese J. Inorg. Chem.* 2014, *30*, 907–912.
15. Hammer A. C., Jia X., Zeller M., Coughlin E. J., Zhang W., Oertel C. M. Ligand geometry directs the packing and symmetry of one-dimensional helical motifs in lead oxide naphthoates and biphenylcarboxylates. *CrystEngComm* 2020, *22*, 6465–6477.