Available online at www.sciencedirect.com

SCIENCE DIRECT®

Journal of Solid State Chemistry 177 (2004) 922–927

JOURNAL OF SOLID STATE CHEMISTRY

http://elsevier.com/locate/jssc

Syntheses, characterizations and crystal structures of two new lead(II) amino and carboxylate–sulfonates with a layered and a pillared layered structure

Yan-Ping Yuan, Jiang-Gao Mao,* and Jun-Ling Song

State Key Laboratory of Structural Chemistry, Fujian Institute for Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

Received 24 July 2003; received in revised form 17 September 2003; accepted 24 September 2003

Abstract

Reactions of lead(II) acetate with *m*-aminobenzenesulfonic acid (HL^1) and 5-sulfoisophthalic acid (H_3L^2) afforded two new lead(II) sulfonates, $Pb(L^1)_2$ **1** and $Pb_2(L^2)(\mu_3\text{-OH})(H_2O)$ **2**. In compound **1**, the lead(II) ion is eight-coordinated by two sulfonate groups bidentately, two sulfonate groups unidentately and two amino groups from six ligands. Each L^1 ligand is tetradentate and bridges with three Pb(II) ions. The interconnection of the Pb(II) ions via bridging sulfonate ligands resulted in ⟨100⟩ and ⟨200⟩ layers. In compound **2**, one Pb(II) ion is six-coordinated by a carboxylate group bidentately, by two carboxylate groups unidentately, by a sulfonate oxygen atom and by an OH anion, whereas the other one is six-coordinated by a bidentate chelating carboxylate group, two μ_3-OH anions, a sulfonate oxygen atom and an aqua ligand. The interconnection of irregular PbO_6 polyhedra via carboxylate–sulfonate ligands resulted in the formation of a pillared layered structure with the 2D layer being formed; the lead(II) ions, hydroxyl groups, carboxylate and sulfonate groups and the benzene ring as the pillar agent.

Keywords: Crystal structure; Layered compound; Lead(II) sulfonates; Hydrothermal synthesis; Inorganic–organic hybrids

1. Introduction

Mixed inorganic–organic hybrids with layered structures or porous open framework structures are of current research interest due to their potential applications in chemical separations and catalysis [1,2]. Much of the studies in this area has involved organically functionalized clays, metal carboxylates and metal phosphonates [3–5]. Similar to phosphonic acids [5], organo-sulfonic acids have been reported to form various types of layered or pillared layered compounds with metal ions, such as silver(I) ion [6], alkali and alkaline earth metal ions [7–8], transition metal ions as well as lanthanide(III) ions [9–11]. Analogous to that in the chemistry of metal phosphonates [5], various functional groups such as amine, hydroxyl and carboxylate groups can be attached to the organo-sulfonic acids to build new inorganic–organic hybrid materials [7–11]. Reports on metal carboxylate–sulfonates are still rare. 4-Carboxybenzenesulfonic acid has been reported to form a layered compound with potassium(I) ion [7a], a 1D coordination polymer with Cd(II) ion [9e], and 3D pillared layered networks with Eu(III) and Gd(III) ions when benzene-1,4-disulfonate is also added in the reactions [10d]. Two copper(II) complexes with a 1D chain structure and three isomorphous layered lanthanide complexes with 5-sulfoisophthalic acid ($HO_3SC_6H_3$-1,3-$(CO_2H)_2$) have also been reported recently [11]. A number of metal complexes with sulfanilic acid or metanilic acid have been reported [12,13]. As exemplified by lead(II) phosphonates [14], the lead(II) ion usually shows a different coordination chemistry from those of other divalent transition metal ions due to the presence of the lone pair electrons, hence it is of interest to study the crystal structures of lead(II) sulfonates. Structurally characterized lead(II) sulfonates including lead(II) trifluoromethanesulfonates are still rare [15,16]. Reactions of lead(II) acetate with metanilic acid (HL^1) and 5-sulfoisophthalic acid ($HO_3SC_6H_3$-1,3-$(CO_2H)_2$, H_3L^2) afforded two new lead(II) sulfonates, $Pb(L^1)_2$ **1** with a layered structure and $Pb_2(L^2)$

---

*Corresponding author. Fax: +86-591-371-4946.
E-mail address: mjg@ms.fjirsm.ac.cn (J.-G. Mao).

0022-4596/$ - see front matter © 2003 Elsevier Inc. All rights reserved.
doi:10.1016/j.jssc.2003.09.030

(μ_3-OH)(H_2O) **2** with a pillared layered structure. Herein we report their syntheses, characterizations and crystal structures.

2. Experimental

All chemicals and solvents were of reagent grade and used as received. Elemental analyses were performed on a German Elementary Vario EL III instrument. IR spectra were recorded on a Magna 750 FT–IR spectrometer photometer as KBr pellets in the range 4000–400 cm^{-1}. Thermogravimetric analyses were carried out with a NETZSCH STA 449C unit, at a heating rate of 15°C/min under a nitrogen atmosphere from 30°C to 800°C. X-ray powder diffraction patterns were collected on a Philips XPERT PRO diffractometer with Cu$K\alpha$ radiation in the 2θ range from 5° to 70°.

2.1. Synthesis of Pb(L^1)$_2$ 1

About 1.0 mmol of *m*-aminobenzenesulfonic acid and 1.0 mmol of lead(II) acetate were dissolved in 10 mL deionized water. Then, ethanol was allowed to diffuse into the resultant solution slowly. The initial and final pH values of the solution are 5.0 and 6.0, respectively. After 1 week, colorless prismatic crystals of compound **1** were collected in a ca yield of 35%. Elemental analysis for compound **1**: C, 26.50%; H, 1.93%; N, 5.15%; S, 11.47%. Calcd: C, 26.11%; H, 2.18%; N, 5.08%; S, 11.60%.

2.2. Synthesis of Pb_2(L^2)(μ_3-OH)(H_2O) 2

Compound **2** was synthesized by hydrothermal reaction. A mixture of 1.5 mmol of lead(II) acetate, 1.0 mmol of 5-sulfoisophthalic acid monosodium salt and 10 mL of H_2O was sealed in Teflon-lined stainless steel autoclaves and heated at 180°C for 4 days. The initial and final pH values of the solution are 6.0 and 7.0, respectively. Colorless brick-shaped crystals of compound **2** were obtained in a yield of 45.7%. Elemental analysis for compound **2**: C, 13.51%; H, 0.89%; S, 4.78%. Calcd: C, 13.86%; H, 0.87%; S, 4.62%.

2.3. X-ray crystallography

Single crystals for compounds **1** and **2** were mounted on a Siemens Smart CCD diffractometer with graphite-monochromated Mo$K\alpha$ radiation ($\lambda = 0.71073$ Å). Intensity data were collected by the narrow frame method at 293 K. The two data sets were corrected for Lorentz and polarization factors as well as for empirical absorption correction by ψ-scan technique. The space groups for compounds **1** and **2** are determined uniquely to be $P2/c$ and $P\bar{1}$, respectively. Both structures were solved by the direct methods and refined by full-matrix least-squares on F^2 by SHELX-97 [17]. Hydrogen atoms except those for the aqua ligand in compound **2** were located at geometrically calculated positions. The residual extremes of the final refinements are meaningless and are within 1.0 Å of the lead(II) atoms. Crystallographic data and refinement parameters were

Table 1
Summary of crystal data and structure refinements for compounds **1** and **2**

Compound	1	2
Molecular formula	$C_{12}H_{12}N_2O_6PbS_2$	$C_8H_6O_9Pb_2S$
Formula weight	551.55	692.57
Space group	$P2/c$	$P\bar{1}$
A (Å)	18.7022(6)	6.9412(2)
B (Å)	5.3044(2)	8.3316(4)
c (Å)	16.3434(6)	10.8530(5)
α (deg.)	90.0	90.749(2)
β (deg.)	114.985(2)	107.803(2)
γ (deg.)	90.0	91.078(2)
V (Å^3)	1469.60(9)	597.36(4)
Z	4	2
D_c (g/cm^3)	2.493	3.850
μ (Mo$K\alpha$) (mm^{-1})	11.797	28.365
F(000)	1040	612
Reflections, total	4339	3095
Reflections, independent	2567 ($R_{\text{int}} = 0.0858$)	2064 ($R_{\text{int}} = 0.0761$)
Goodness-of-fit (GOF)	1.137	1.098
R_1/wR_2[$I > 2\sigma(I)$]	0.0546/0.1459	0.0554/0.1386
R_1/wR_2 (all data)	0.0717/0.1629	0.0739/0.1533

[a] $R1 = \sum|F_o| - |F_c\sum|F_o|$,
$$wR2 = \left\{\sum w[(F_o)^2 - (F_c)^2]^2 / \sum w[(F_o)^2]^2\right\}^{1/2}.$$

Table 2
Selected bond lengths (Å) and angles (deg.) for compounds **1** and **2**

Compound 1			
Pb(1)–O(12)	2.576(9)	Pb(1)–O(12)#1	2.576(9)
Pb(1)–O(13)#2	2.677(10)	Pb(1)–O(13)#3	2.677(10)
Pb(1)–N(1)#4	2.714(13)	Pb(1)–N(1)#5	2.714(13)
Pb(1)–O(11)#2	2.803(10)	Pb(1)–O(11)#3	2.803(10)
Pb(2)–O(23)#6	2.565(8)	Pb(2)–O(23)#7	2.565(8)
Pb(2)–O(22)#8	2.705(10)	Pb(2)–O(22)	2.705(10)
Pb(2)–N(2)#9	2.727(12)	Pb(2)–N(2)#4	2.727(12)
Pb(2)–O(21)	2.759(10)	Pb(2)–O(21)#8	2.759(10)
Compound 2			
Pb(1)–O(5)#1	2.351(13)	Pb(1)–O(4)	2.416(18)
Pb(1)–O(2)#1	2.501(15)	Pb(1)–O(3)	2.70(2)
Pb(1)–O(13)#2	2.734(17)	Pb(1)–O(3)#3	2.74(2)
Pb(2)–O(5)	2.330(14)	Pb(2)–O(5)#4	2.511(13)
Pb(2)–O(2)	2.657(15)	Pb(2)–O(1W)	2.685(18)
Pb(2)–O(1)	2.710(17)	Pb(2)–O(12)#5	2.713(17)

Symmetry transformations used to generate equivalent atoms: for compound **1**: #1 $-x+1, y, z+1/2$; #2 $-x+1, y+1, -z+1/2$; #3 $x, y+1, z$; #4 $x, -y+1, z+1/2$; #5 $-x+1, -y+1, -z$; #6 $x, y-1, z$; #7 $-x, y-1, -z+3/2$; #8 $-x, y, -z+3/2$; #9 $-x, -y+1, -z+1$. For compound **2**: #1 $-x, -y, -z+1$; #2 $x, y+1, z$; #3 $-x, -y+1, -z+1$; #4 $-x, -y-1, -z$; #5 $-x+1, -y-1, -z+1$; #6 $x, y-1, z$.

summarized in Table 1. Important bond distances are listed in Table 2.

Crystallographic data (excluding structure factors) for the two structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC 215977 and 215978. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: (44) 1223 336-033; e-mail: deposit@ccdc.cam.ac.uk).

3. Results and discussion

The structure of compound **1** contains two lead(II) ions located at positions with a two-fold symmetry (Pb1: 0, 0.2854(1), 3/4 and Pb2: 1/2, 0.7724(1), 1/4) and two L^1 ligands. Pb1$(L^1)_2$ and Pb2$(L^1)_2$ fragments are similar to each other, so only the structure of Pb1$(L^1)_2$ fragment is shown in Fig. 1. Both Pb1 and Pb2 are eight-coordinated by six oxygen atoms from four sulfonate groups and two amine groups from six L^1 ligands. The coordination geometry around the Pb(II) ion can be described as a distorted square antiprism. Two of the four sulfonate groups are in a bidentate chelating fashion whereas the other two in a unidentate fashion. The Pb–O11a and Pb–O11b bond distances of 2.803(10) Å are significantly longer than those of remaining Pb–O distances, which are in the range 2.565(8)–2.759(10) Å. The Pb–N distances range from 2.714(13) to 2.727(12) Å. These distances are comparable to those reported in lead(II) sulfonates [15,16]. Each L^1 ligand acts as a tetradentate metal linker, chelates with a Pb(II) ion and bridges with two other Pb(II) ions.

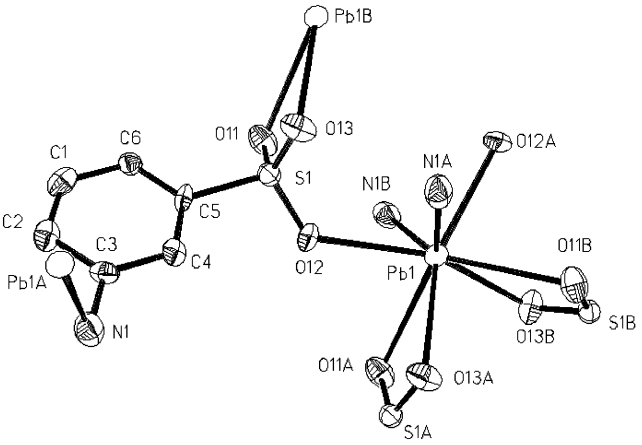

Fig. 1. ORTEP drawing showing a Pb1$(L^1)_2$ fragment in compound **1**. The thermal ellipsoids are drawn at 50% probability.

The interconnection of the Pb(II) ions through bridging L^1 ligands resulted in the formation of ⟨100⟩ and ⟨200⟩ lead sulfonate layers. These two different layers have a similar structure (Figs. 2 and 3). The ⟨100⟩ layer is formed by Pb1(L^1) fragments and the ⟨200⟩ layer by Pb2(L^1) fragments. Within the 2D layer, there are two types of rings. One is the eight-membered ring formed by two Pb(II) ions and two bridging sulfonate groups, and the other type is the 14-membered ring formed by two lead(II) ions and two bridging L^1 ligands (Pb–(O–S–C–C–C–N)$_2$–Pb) (Fig. 2). The latter rings form open tunnels along *b*-axis (Fig. 3). The benzene rings of the ligands are orientated toward the interlayer space. The shortest distance between two neighboring benzene ring centers is 5.26 Å, therefore it is expected that there is no $\pi-\pi$ interaction between these benzene rings. The interlayer distance is estimated to be about 9.4 Å (1/2*a*) and the thickness of the layer is about 7.2 Å, we expect that there is not much free space between the two neighboring layers.

As shown in Fig. 4, compound **2** is composed of two lead(II) ions, one 5-sulfoisothphalate anion, one

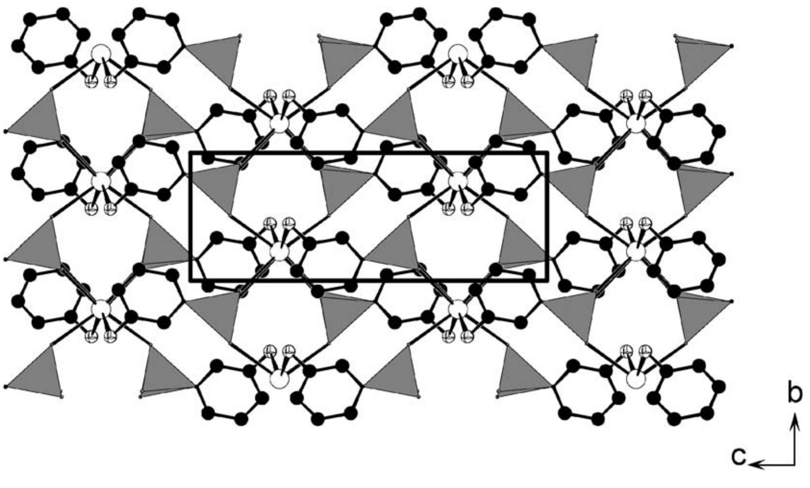

Fig. 2. A ⟨100⟩ layer formed by Pb1(L^1) fragments in compound **1**. C–SO$_3$ tetrahedra are shaded in gray. The Pb, N and C atoms are represented by open, octanded and black circles, respectively.

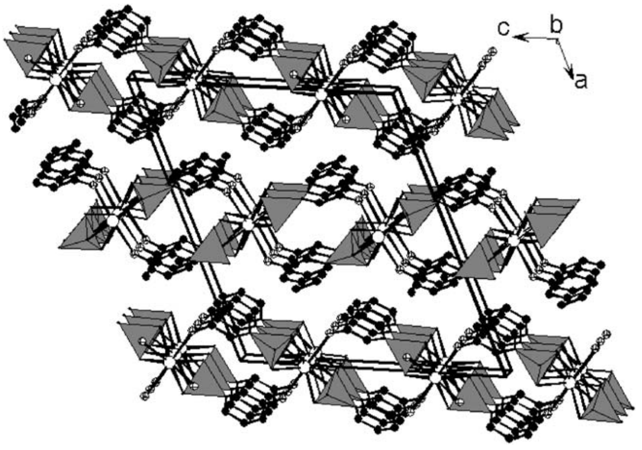

Fig. 3. View of the structure of compound **1** down *b*-axis. C–SO_3 tetrahedra are shaded in gray. The Pb, N, C and O atoms are represented by open, octanded, black and crossed circles, respectively.

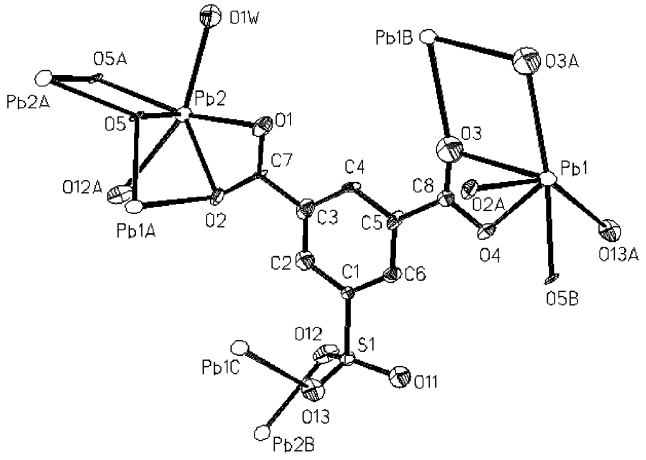

Fig. 4. ORTEP drawing showing the coordination geometries around the lead(II) ions as well as the coordination mode of the carboxylate–sulfonate ligand in compound **2**. The thermal ellipsoids are drawn at 50% probability.

hydroxyl anion and an aqua ligand. The hydroxyl anion formed due to the high pH value (about 7.0) for the reaction. Pb1 is six-coordinated by a carboxylate group bidentately, by two carboxylate groups unidentately, by a sulfonate oxygen atom and by a OH anion, whereas Pb2 is six-coordinated by a carboxylate group in a bidentate fashion and a sulfate oxygen atom from two L^2 ligands, two hydroxyl anions as well as an aqua ligand. The coordination geometries around the lead(II) ions can be described as a ψ-PbO_6 pentagonal bipyramid with the seventh coordination site occupied by the lone pair of the Pb(II) ion. Pb–O distances range 2.33(1)–2.74(2) Å, comparable to those in compound **1** and those reported in lead(II) sulfonates and carboxylates [15,16,18]. The sulfonate group of L^2 ligand is bidentate and bridges with two Pb(II) ions. The third sulfonate oxygen atom remains non-coordinated. Both carboxylate groups of the L^2 ligand are tridentate chelating and bridging. The hydroxyl anion is a μ_3 metal linker, bridging with three lead(II) ions. Each pair of Pb(II) ions is bridged by a pair of oxygen atoms and forms three types of four membered rings (Pb1–O3–O3a–Pb1b, Pb2–O5–O5a–Pb2a, Pb2–O5–O2–Pb1a) (Fig. 4).

The interconnection of the lead(II) ions through bridging hydroxyl group as well as the carboxylate and sulfonate groups of L^2 ligands resulted in a ⟨020⟩ 2D lead(II) carboxylate–sulfonate hybrid layer (Fig. 5). Such layers are interconnected by the benzene rings of the ligands to form a pillared layered structure (Fig. 6). Neighboring benzene rings are further interconnected via weak $\pi-\pi$ interaction. The distance between two such benzene ring centers (symmetry code: $1-x$, $-y$, $1-z$) is only 3.667 Å and these rings are parallel to each other. Small pores are formed; however, such pores are of no practical use due to a too small size (less than

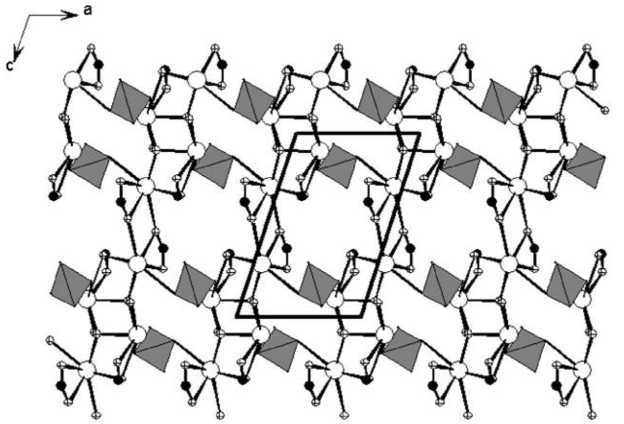

Fig. 5. A ⟨020⟩ 2D lead(II) carboxylate–sulfonate layer in compound **2**. C–SO_3 tetrahedra are shaded in gray. The Pb, C and O atoms are represented by open, black and crossed circles, respectively.

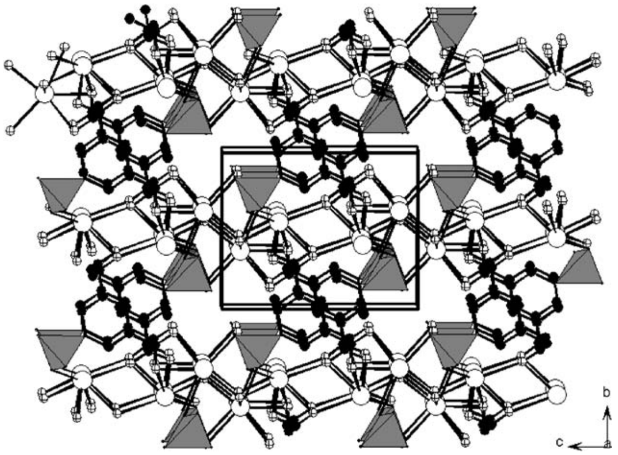

Fig. 6. View of the structure of compound **2** down *a*-axis. C–SO_3 tetrahedra are shaded in gray. The Pb, C and O atoms are represented by open, black and crossed circles, respectively.

6.0 Å^2). The aqua ligand also forms a hydrogen bond with non-coordinated sulfonate oxygen (O11, symmetry code: $1-x$, $-y$, $1-z$) and the O1w⋯O11 separation is 2.88(2) Å.

IR spectra of *m*-aminobenzenesulfonic acid and its lead(II) complex were measured. Compared with *m*-aminobenzenesulfonic acid, the symmetric vibration absorption band of the NH_2 group at 3350 cm^{-1} of the free ligand has been split into two bands at 3347 and 3284 cm^{-1} in compound **1**. The asymmetric vibration absorption band of the sulfonate group at 1200 cm^{-1} of the free ligand has been split into two bands at 1205 and 1180 cm^{-1} in compound **1**, indicating that both amino and sulfonate groups were involved in the coordination to the lead ions. Absorption bands at 634 and 615 cm^{-1} are characteristic bands for ν_{S-O} of the sulfonate group. IR spectra of compound **2** were also measured. The bands at 3550 and 3496 cm^{-1} can be assigned to the O–H stretching vibrations of the hydroxyl anion and the lattice water, respectively. The two strong bands at 1598 and 1546 cm^{-1} correspond to the antisymmetric stretching bands of the carboxylate groups. The symmetric stretching band of the carboxylate group appeared around 1429 cm^{-1}. The strong absorption band at 622 cm^{-1} is due to the ν_{S-O} stretching.

XRD powder patterns for both compounds were collected to check the purity of the bulk samples. Results indicate that the experimental data are in good agreement with the simulated XRD powder patterns based on single crystal data, hence both compounds were obtained as a mono-phase.

TGA analyses revealed that compound **1** is stable up to 250°C. The weight loss from 250°C to 800°C corresponds to the pyrolysis of organic groups. The final product is $Pb(SO_3)_2$. The total weight loss of 34.2% is in good agreement with the calculated value (33.4%). TGA curves of compound **2** show two main steps of weight losses. Compound **2** is stable up to 240°C. The weight loss of 3.4% occurred from 240°C to 280°C corresponds to the release of the aqua ligand (calculated value, 2.6%). Such an unusual high temperature needed for the removal of the aqua ligand is due to its hydrogen bonding with the non-coordinated sulfonate oxygen, which has been discussed earlier. The second weight loss appeared from 420°C to 800°C is the burning of the organic groups. The final product is a mixture of $PbSO_4$ and PbO in a 1:1 molar ratio. The total weight loss of 27.1% is slightly larger than the calculated value of 24.0%.

4. Conclusions

Reactions of lead(II) acetate with *m*-aminobenzenesulfonic acid (HL^1) and 5-sulfoisophthalic acid (H_3L^2) afforded two new lead(II) sulfonates, $Pb(L^1)_2$ **1** and $Pb(L^2)(\mu_3\text{-OH})(H_2O)$ **2**. Compound **1** has a layered structure, whereas the structure of compound **2** is pillared layered. In both compounds, the functional amine and carboxylate groups attached to the sulfonate ligand are involved in the metal coordination. Furthermore, the sulfonate group in compound **1** acts as tridentate metal linker whereas the one in compound **2** is bidentate.

Acknowledgments

This work was supported by the Innovative Project and the Introduction of Overseas Elitists Program by Chinese Academy of Sciences, and the Scientific Research Foundation for the Returned Overseas Chinese Scholars, State Education Ministry. We thank Prof. Abraham Clearfield for his great help and valuable suggestions.

References

[1] A.K. Cheetham, G. Férey, T. Loiseau, Angew. Chem. Int. Ed. 38 (1999) 3268 (and references therein).
[2] F. Pinnavaia, Science 220 (1983) 365.
[3] E. Rightor, M. Tzou, T. Pinnavaia, J. Catal. 130 (1991) 29.
[4] (a) O.M. Yaghi, M. O'Keeffe, N.W. Ockwig, H.K. Chae, M. Eddaoudi, J. Kim, Nature 423 (2003) 705;
  (b) M. Eddaoudi, J. Kim, N. Rosi, D. Vodak, J. Wachter, M. O'Keefe, O.M. Yaghi, Science 295 (2002) 469;
  (c) M. Eddaoudi, D.B. Moler, H.L. Li, B.L. Chen, T.M. Reineke, M. O'keeffe, O.M. Yaghi, Accounts Chem. Res. 34 (2001) 319.
[5] (a) E. Stein, A. Clearfield, M.A. Subramanian, Solid State Ionics 83 (1996) 113;
  (b) G. Alberti, U. Costantino, in: J.M. Lehn (Ed.), Comprehensive Supramolecular Chemistry, Pergamon-Elsevier Science Ltd., London, 1996, p. 1;
  (c) A. Clearfield, Curr. Opin. Solid State Mater. Sci. 1 (1996) 268;
  (d) A. Clearfield, Metal phosphonate chemistry, in: K.D. Karlin (Ed.), Progress in Inorganic Chemistry, vol. 47, Wiley, New York, 1998, pp. 371–510 (and references therein).
[6] (a) G.K.H. Shimizu, G.D. Enright, C.I. Ratcliffe, K.F. Preston, J.L. Reid, J.A. Ripmeester, Chem. Commun. (1999) 1485;
  (b) G. Smith, B.A. Cloutt, D.E. Lynch, K.A. Byriel, C.H.L. Kennard, Inorg. Chem. 37 (1998) 3236;
  (c) A.P. Côté, M.J. Ferguson, K.A. Khan, G.D. Enright, A.D. Kulynych, S.A. Dalrymple, G.K.H. Shimizu, Inorg. Chem. 41 (2002) 287;
  G.K.H. Shimizu, G.D. Enright, C.I. Ratcliffe, G.S. Rego, J.L. Reid, J.A. Ripmeester, Chem. Mater. 10 (1998) 3282.
[7] (a) B.J. Gunderman, P.J. Squattrito, Inorg. Chem. 33 (1994) 2924;
  (b) B.J. Gunderman, I.D. Kabell, P.J. Squattrito, S.N. Dubey, Inorg. Chim. Acta 258 (1997) 237;
  (c) B.J. Gunderman, P.J. Squattrito, Inorg. Chem. 34 (1995) 2399.
[8] (a) A.P. Côté, G.K.H. Shimizu, Chem. Commun. (2001) 251;
  (b) S.A. Dalrymple, G.K.H. Shimizu, Chem. Eur. J. 8 (2002) 3010;
  (c) J.O. Yu, A.P. Côté, G.D. Enright, G.K.H. Shimizu, Inorg. Chem. 40 (2001) 582.

[9] (a) C.-H. Chen, J.-W. Cai, C.-Z. Liao, X.-L. Feng, X.-M. Chen, S.W. Ng, Inorg. Chem. 41 (2002) 4967;
(b) J.-W. Cai, C.-H. Chen, C.-Z. Liao, J.-H. Yao, X.-P. Hu, X.-M. Chen, J. Chem. Soc. Dalton Trans. (2001) 1137;
(c) F. Tisato, F. Refosco, G. Bandoli, G. Pilloni, B. Corain, Inorg. Chem. 40 (2001) 1394;
(d) R.K. Henderson, E. Bouwman, A.L. Spek, J. Reedijk, Inorg. Chem. 36 (1997) 4616;
(e) R.-X. Yuan, R.-G. Xiong, Y.-L. Xie, X.-Z. You, S.-M. Peng, G.-H. Lee, Inorg. Chem. Commun. 4 (2001) 384.

[10] (a) A. Drljaca, M.J. Hardie, J.A. Johnson, C.-L. Raston, H.R. Webb, Chem. Commun. (1999) 1135;
(b) G.W. Orr, L.J. Barbour, J.L. Atwood, Science 285 (1999) 1049;
(c) M.J. Hardie, J.A. Johnson, C.L. Raston, H.R. Webb, Chem. Commun. (2000) 849;
(d) R.-G. Xiong, J. Zhang, Z.-F. Chen, X.-Z. You, C.-M. Che, J. Chem. Soc. Dalton Trans. (2001) 780;
(e) J.W. Steed, J.P. Johnson, C.L. Barness, R.K. Juneja, J.L. Atwood, S. Reilly, R.L. Hollis, P.H. Smith, D.L. Clark, J. Am. Chem. Soc. 117 (1995) 11426.

[11] (a) D. Sun, R. Cao, Y.-Q. Sun, X. Li, W. Bi, M.-C. Hong, Y.-J. Zhao, Eur. J. Inorg. Chem. (2003) 94;
(b) Z. Wang, M. Ströbele, K.-L. Zhang, H.-J. Meyer, X.-Z. You, Z. Yu, Inorg. Chem. Commun. 5 (2002) 230.

[12] C.H. Kim, S.G. Lee, Acta Crystallogr. C 58 (2002) 421;
B.J. Gunderman, S.N. Dubey, P.J. Squattrito, Acta Crystallogr. C 53 (1997) 17;
B.J. Gunderman, P.J. Squattrito, S.N. Dubey, Acta Crystallogr. C 52 (1996) 1131;
V. Shakeri, S. Haussuhl, Z. Kristallogr. 198 (1992) 165;
V. Shakeri, S. Haussuhl, Z. Kristallogr. 198 (1992) 167.

[13] K. Brodersen, R. Beck, Z. Inorg. Allg. Chem. 553 (1987) 35.

[14] S. Ayyappan, G.D. de Delgado, A.K. Cheetham, G. Ferey, C.N.R. Rao, J. Chem. Soc. Dalton Trans. (1999) 2905;
N. Stock, Solid State Sci. 4 (2002) 1089;
J.-G. Mao, Z.-K. Wang, A. Clearfield, Inorg. Chem. 41 (2002) 6106;
J.-G. Mao, Z.-K. Wang, A. Clearfield, J. Chem. Soc. Dalton Trans. (2002) 4457.

[15] S. Larsen, F.M. Nicolaisen, A.M. Schonemann, Acta Chem. Scand. 46 (1992) 1135;
A.T. Yordanov, O.A. Gansow, M.W. Brechbiel, L.M. Rogers, R.D. Rogers, Polyhedron 18 (1999) 1055;
F. Huber, J. Remer, M. Schurmann, Acta Crystallogr. C 50 (1994) 1913.

[16] G.M. de Lima, D.J. Duncalf, S.P. Constantine, Main Group Metal Chem. 23 (2000) 307;
A.M. Garcia, D.M. Bassani, J.-M. Lehn, G. Baum, D. Fenske, Chem. Eur. J. 5 (1999) 1234.

[17] G.M. Sheldrick, SHELXTL, Crystallographic Software Package, SHELXTL, Version 5.1, Bruker-AXS, Madison, WI, 1998.

[18] M.R.St J. Foreman, T. Gelbrich, M.B. Hursthouse, M.J. Plater, Inorg. Chem. Acta 3 (2000) 234.