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Syntheses, characterizations and crystal structures of two lead(II) phosphonate–sulfonate hybrid materials

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Abstract

Hydrothermal reactions of lead(II) acetate with 5-sulfoisophthalic acid monosodium salt (NaH_2BTS) and *N*-(phosphonomethyl)-*N*-methylglycine, $MeN(CH_2CO_2H)(CH_2PO_3H_2)$ (H_3L^1), or a new aminodiphosphonic acid, 3-Pyridyl-$CH_2N(CH_2PO_3H_2)_2$ (H_4L^2), afforded two novel lead(II) phosphonate–sulfonate hybrids, namely, $Pb_3[L^1][BTS][H_2O]\cdot H_2O$ **1** and $Pb_2[HL^3][BTS]\cdot H_2O$ **2** (H_2L^3 = 3-Pyridyl-$CH_2(Me)N(CH_2PO_3H_2)$). H_2L^3 was formed as a result of the decomposition of one phosphonate group in H_4L^2 during the reaction. Compound **1** crystallizes in the triclinic space group $P\bar{1}$ with $a = 9.9148(4)$ Å, $b = 10.4382(4)$ Å, $c = 10.6926(2)$ Å, $\alpha = 96.495(2)^\circ$, $\beta = 110.599(2)^\circ$, $\gamma = 98.433(2)^\circ$, $V = 1008.31(6)$ Å^3, and $Z = 2$. The structure of compound **1** features a 3D network built from the interconnection of hexanuclear $Pb_6(L^1)_2$ units and 1D double chains of lead(II) carboxylate–sulfonate. Compound **2** crystallizes in the monoclinic space group $P2_1/c$ with $a = 9.5403(7)$ Å, $b = 11.6170(8)$ Å, $c = 19.7351(15)$ Å, $\beta = 97.918(2)^\circ$, $V = 2166.4(3)$ Å^3, and $Z = 4$. Compound **2** has a 3D network structure built by the cross-linkage of 1D double chains of lead(II) phosphonates and 2D layers of lead(II) carboxylate–sulfonate.

Keywords: Metal phosphonates; Sulfonates; Hydrothermal synthesis; Open framework; Crystal structures

1. Introduction

In the past few years, many research efforts have been devoted to the chemistry of metal phosphonates due to their potential applications in the areas of catalysis, ion exchange, proton conductivity, intercalation chemistry, photochemistry, and materials chemistry [1]. Metal phosphonates also exhibit a variety of open frameworks such as layered and open-framework structures. Materials with open-framework structures are promising candidates for hybrid composite materials in electro-optical and sensing applications [2]. In the chemistry of metal phosphonates, the use of bifunctional or multifunctional anionic units, such as diphosphonates, aminophosphonates or phosphonocarboxylates has led to a number of new materials with open-framework structures [3–7]. The strategy of attaching functional groups such as carboxylic acids, crown ethers and amines to the phosphonic acids has been also proven to be an effective route for the synthesis of metal phosphonates with open-framework architectures [8]. A 3D open-framework tin(II) phosphonopropionate oxalate and a layered tin(II) methylphosphonate oxalate have been reported by the Cheetham group [9a,9b] and a zinc(II) compound of phosphonopropionic acid and 1,3,5-benzene-tricarboxylic acid (H_3BTC) was isolated; however, the tricarboxylate moiety remains non-coordinated and is also severely disordered [2b]. Two lead(II) compounds of di- or tri-phosphononic acid and H_3BTC have reported in our group, in which the tricarboxylate moiety is involved in metal coordination [9c]. We deem that sulfonic acids should be also capable to form metal sulfonate–phosphonate hybrids. As an expansion of our previous work, we selected one carboxylic-phosphonic acid, *N*-(phosphonomethyl)-*N*-methylglycine, $MeN(CH_2CO_2H)(CH_2PO_3H_2)$ (H_3L^1) or a new amino diphosphonic acid, 3-Pyridyl-$CH_2N(CH_2PO_3H_2)_2$ (H_4L^2) and 5-sulfoisophthalic acid monosodium salt (NaH_2BTS)

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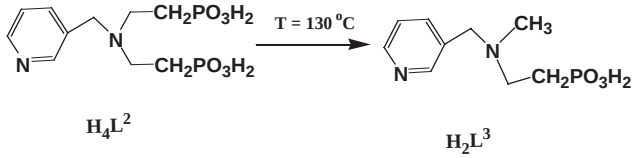

Scheme 1. Formation of H_2L^3 by decomposition of H_4L^2.

as the second metal linker. Hydrothermal reactions of the above two phosphonate ligands with lead(II) acetate and NaH_2BTS afforded two novel 3D lead(II) phosphonates, namely, $Pb_3[L^1][BTS][H_2O]\cdot H_2O$ **1** and $Pb_2[HL^3][BTS]\cdot H_2O$ **2** (H_2L^3 = 3-Pyridyl-CH_2(Me)N($CH_2PO_3H_2$)). H_2L^3 was formed as a result of the decomposition of one phosphonate group in H_4L^2 under reaction temperature (Scheme 1). Herein, we report their syntheses, characterizations and crystal structures.

2. Experimental section

MeN(CH_2CO_2H)($CH_2PO_3H_2$) (H_3L^1), and 3-Pyridyl-CH_2N($CH_2PO_3H_2$)$_2$ (H_4L^2) were prepared by a Mannich-type reaction according to procedures described previously [8d,9c,10]. All other chemicals were obtained from commercial sources and used without further purification. Elemental analyses were performed on a Vario EL III elemental analyzer. Thermogravimetric analyses were carried out with a TGA/SBTA851 unit, at a heating rate of 15°C/min under a nitrogen atmosphere. IR spectra were recorded on a Magna 750 FT–IR spectrometer photometer as KBr pellets in the 4000−400 cm^{-1}. X-ray powder diffraction (XRD) patterns (Cu$K\alpha$) were collected in a sealed glass capillary on a Philips XPERT-MPD diffractometer.

2.1. Synthesis of $Pb_3[L^1][BTS][H_2O]\cdot H_2O$ 1

Compound **1** was prepared by hydrothermal reaction of lead(II) acetate trihydrate (1.0 mmol, 0.38 g), H_3L^1 (1.0 mmol, 0.18 g) and NaH_2BTS (1.0 mmol, 0.26 g) in a 1:1:1 molar ratio in 10.0 mL of deionized water. The mixture was sealed in autoclave equipped with a Teflon liner (25 mL) and then heated at 130°C for 4 days. The initial and final pH values are 4 and 5, respectively. Colorless crystals of **1** were recovered in a 65.8% yield (based on Pb). Elemental analysis for compound **1**, $C_{12}H_{14}NO_{14}PSPb_3$: C, 13.33%; H, 1.31%; N, 1.30%. Calcd: C, 13.26%; H, 1.14%; N, 1.3%. IR (KBr, cm^{-1}): 3442m, 3068w, 1593s, 1531vs, 1435m, 1394w, 1360vs, 1315w, 1200m, 1115m, 1045m, 997m, 974w, 924w, 779m, 721m, 721m, 673w, 623s, 580w, 519w, 442m.

2.2. Synthesis of $Pb_2[HL^3][BTS]\cdot H_2O$ 2

Compound **2** was prepared by hydrothermal reaction of lead(II) acetate trihydrate (0.75 mmol, 0.29 g), H_4L^2 (0.25 mmol, 0.08 g) and NaH_2BTS (0.5 mmol, 0.14 g) in a 3:1:2 molar ratio in 10.0 mL of deionized water. The resultant mixture was sealed in an autoclave equipped with a Teflon liner (25 mL) and then heated at 130°C for 5 days. The pH value of the resultant solution was adjusted to approximately 4 by slow addition of NaOH. Colorless crystals of **2** were recovered in 63.6% yield (based on Pb). Elemental analysis for compound **2**, $C_{16}H_{17}N_2O_{11}PSPb_2$: C, 21.57%; H, 1.89%; N, 3.14%. Calcd: C, 21.60%; H, 1.81%; N, 3.15%. IR (KBr, cm^{-1}): 3419m, 3059m, 1599s, 1543vs, 1479w, 1431m, 1360vs, 1309w, 1230m, 1196w, 1169w, 1115s, 1082m, 1034s, 960m, 939m, 868w, 845w, 781m, 766w, 723m, 683w, 623m, 567s, 521w, 442w.

2.3. Crystallography

Single crystals of **1** and **2** were mounted on a Siemens Smart CCD diffractometer equipped with a graphite-monochromated Mo$K\alpha$ radiation ($\lambda = 0.71073$ Å). Intensity data were collected by the narrow frame method at 293 K. Both data sets were corrected for Lorentz and polarization as well as for absorption by the SADABS program [11]. Both structures were solved by direct methods and refined by full-matrix least-squares fitting on F^2 by SHELX-97 [11]. All non-hydrogen atoms, except for O(1), C(2), C(14) C(15) and C(17) atoms in **1** and C(3), C(6), C(15) and C(17) atoms in **2**, were refined with anisotropic thermal parameters. All hydrogen atoms except those of the water molecules were located at geometrically calculated positions. Final cycle of refinements reveals featureless residual peak and hole of 2.62 e Å^3 (1.17 Å from O3) and −4.07 e Å^3 (0.96 Å from Pb3) for compound **1**; and 1.93 e Å^3 (1.05 Å from Pb1) and −2.28 e Å^3 (1.19 Å from Pb1) for compound **2**, respectively. Crystallographic data and structural refinements are summarized in Table 1. Important bond distances and angles 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 Centre as supplementary publication nos. CCDC 228761 and 228762. 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** features a complicated 3D network. As shown in Fig. 1, there are three unique

Table 1
Crystal data and structure refinements for **1** and **2**[a]

Compound	**1**	**2**
Formula	$C_{12}H_{14}NO_{14}PSPb_3$	$C_{16}H_{17}N_2O_{11}PSPb_2$
F_w	1080.84	890.73
Space group	$P\bar{1}$	$P2_1/c$
a (Å)	9.9148(4)	9.5403(7)
b (Å)	10.4382(4)	11.6170(8)
c (Å)	10.6926(2)	19.7351(15)
α (deg)	96.495(2)	90.0
β (deg)	110.599(2)	97.918(2)
γ (deg)	98.433(2)	90.0
V (Å^3)	1008.31(6)	2166.4(3)
Z	2	4
D_{calcd} (g cm^{-3})	3.560	2.731
μ (mm^{-1}), Mo$K\alpha$ (mm^{-1})	25.243	15.756
F (000)	964	1640
Reflections collected	5144	6530
Independent reflections	3477 ($R_{int} = 0.0381$)	3806 ($R_{int} = 0.0608$)
Observed reflections $[I > 2\sigma(I)]$	3243	2536
Goodness-of-fit (GOF)	1.096	1.118
R_1, wR_2 ($I > 2\sigma(I)$)[a]	0.0408/0.1090	0.0734/0.1270
R_1, wR_2 (all data)	0.0444/0.1133	0.1288/0.1531

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$, w$R_2 = \{\sum w[(F_o)^2 - (F_c)^2]^2/\sum w[(F_o)^2]^2\}^{1/2}$.

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

Compound **1**

Pb(1)–O(11)#1	2.387(8)	Pb(2)–O(6)#3	2.625(9)
Pb(1)–O(3)#2	2.518(8)	Pb(2)–O(4)#2	2.628(8)
Pb(1)–O(2)	2.639(8)	Pb(2)–O(7)	2.672(10)
Pb(1)–O(1W)	2.694(9)	Pb(3)–O(13)#1	2.359(7)
Pb(1)–O(4)#2	2.721(8)	Pb(3)–O(12)	2.363(8)
Pb(2)–O(1)	2.415(9)	Pb(3)–O(2)	2.554(8)
Pb(2)–O(5)#3	2.463(9)	Pb(3)–N(1)	2.637(9)
O(11)#1–Pb(1)–O(3)#2	72.5(3)	O(1)–Pb(2)–O(6)#3	120.6(3)
O(11)#1–Pb(1)–O(2)	81.8(3)	O(5)#3–Pb(2)–O(6)#3	50.6(3)
O(3)#2–Pb(1)–O(2)	145.5(3)	O(1)–Pb(2)–O(4)#2	76.5(3)
O(11)#1–Pb(1)–O(1W)	74.0(3)	O(5)#3–Pb(2)–O(4)#2	82.4(3)
O(3)#2–Pb(1)–O(1W)	84.0(3)	O(6)#3–Pb(2)–O(4)#2	83.5(3)
O(2)–Pb(1)–O(1W)	110.9(3)	O(1)–Pb(2)–O(7)	89.6(3)
O(11)#1–Pb(1)–O(4)#2	71.2(3)	O(5)#3–Pb(2)–O(7)	92.6(4)
O(3)#2–Pb(1)–O(4)#2	49.7(2)	O(6)#3–Pb(2)–O(7)	103.5(3)
O(2)–Pb(1)–O(4)#2	100.7(2)	O(4)#2–Pb(2)–O(7)	166.1(3)
O(1W)–Pb(1)–O(4)#2	128.2(3)	O(13)#1–Pb(3)–O(12)	80.4(3)
O(1)–Pb(2)–O(5)#3	71.5(3)	O(13)#1–Pb(3)–O(2)	73.1(3)
O(13)#1–Pb(3)–N(1)	114.2(3)	O(12)–Pb(3)–O(2)	111.3(3)
O(12)–Pb(3)–N(1)	72.1(3)	O(2)–Pb(3)–N(1)	64.2(3)
O(11)#1–Pb(1)–O(3)#2	72.5(3)	O(1)–Pb(2)–O(6)#3	120.6(3)
O(11)#1–Pb(1)–O(2)	81.8(3)	O(5)#3–Pb(2)–O(6)#3	50.6(3)
O(3)#2–Pb(1)–O(2)	145.5(3)	O(1)–Pb(2)–O(4)#2	76.5(3)

Compound **2**

Pb(1)–O(12)	2.318(16)	Pb(2)–O(4)	2.619(19)
Pb(1)–O(2)	2.537(16)	Pb(2)–O(13)#4	2.358(15)
Pb(1)–N(2)#1	2.61(2)	Pb(2)–O(11)#5	2.375(14)
Pb(1)–O(4)#2	2.705(16)	Pb(2)–O(3)	2.498(18)
Pb(1)–O(13)#3	2.731(16)	Pb(2)–O(21)#6	2.769(19)
Pb(1)–O(1)	2.785(17)		
O(12)–Pb(1)–O(2)	76.8(5)	N(2)#1–Pb(1)–O(13)#3	147.5(6)
O(12)–Pb(1)–N(2)#1	76.7(6)	O(4)#2–Pb(1)–O(13)#3	113.1(5)
O(2)–Pb(1)–N(2)#1	80.0(6)	O(13)#4–Pb(2)–O(11)#5	83.8(5)
O(12)–Pb(1)–O(4)#2	77.9(6)	O(13)#4–Pb(2)–O(3)	95.0(7)
O(2)–Pb(1)–O(4)#2	153.6(6)	O(11)#5–Pb(2)–O(3)	120.2(6)
N(2)#1–Pb(1)–O(4)#2	87.2(6)	O(13)#4–Pb(2)–O(4)	99.8(6)
O(12)–Pb(1)–O(13)#3	82.9(5)	O(11)#5–Pb(2)–O(4)	70.2(5)
O(2)–Pb(1)–O(13)#3	70.8(5)		

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

Pb(II) ions in the asymmetric unit of compound **1**. Pb1 is 5-coordinated by one carboxylate and one phosphonate oxygens from two L^1 ligands, two carboxylate oxygen atoms from a BTS anion and an aqua ligand. Pb2 is 5-coordinated by one carboxylate oxygen atom from one L^1 ligand, three carboxylate oxygen atoms from two BTS anions, and one sulfonate oxygen atom from the third BTS anion. The coordination geometry of Pb1 and Pb2 can be described as a ψ-PbO_5 octahedron with the lone pair occupying the sixth coordination site. Pb3 is 4-coordinated by a chelating L^1 ligand in a tridentate fashion (O2, N1 and O12), and one phosphonate oxygen O13 from another L^1 ligand. Its coordination geometry can be described as a ψ-PbO_3N square pyramid with the lone pair occupying the fifth coordination site. This type of coordination geometry has been reported in other lead phosphonates [9]. The Pb–O distances range from 2.387(8) to 2.721(8) Å, comparable to those reported for other lead(II) phosphonates and lead(II) carboxylates [9,12–14]. The phosphonate ligand anion is heptadentate, it chelates one Pb(II) ion tridentately and bridges four other Pb(II) ions. O2 is bidentate whereas the remaining coordination atoms are unidentate. The BTS^{3-} anion is pentadentate. Each carboxylate group chelates with a lead(II) ion bidentately and the sulfonate group is unidentate (O7). O8 and O9 of the sulfonate group remain non-coordinated. Based on charge balance, P–O, S–O and C–O distances as well as their coordination mode, both L^1 and BTS should be 3-, which means that both of them have been deprotonated completely.

The interconnection of the Pb(II) ions via chelating and bridging L^1 ligands leads to the formation of $Pb_6(L^1)_2$ unit (Fig. 2a), whereas the interconnection of the Pb(II) ions via bridging BTS anions resulted in a 1D double chain along the c-axis (Fig. 2b). The cross-linkage of the above two building blocks leads to a complicated 3D network (Fig. 3). The lattice water molecule is located in the cavity of the structure, it is hydrogen bonded to the aqua ligand (O(1w)···O(2w) 2.748(14) Å).

Compound **2** was obtained by hydrothermal reaction of lead(II) acetate with NaH_2BTS and the aminodiphosphonic acid, 3-pyridyl-$CH_2NH(CH_2PO_3H_2)_2$ (H_4L^2). During the reaction, one phosphonate group of H_4L^2

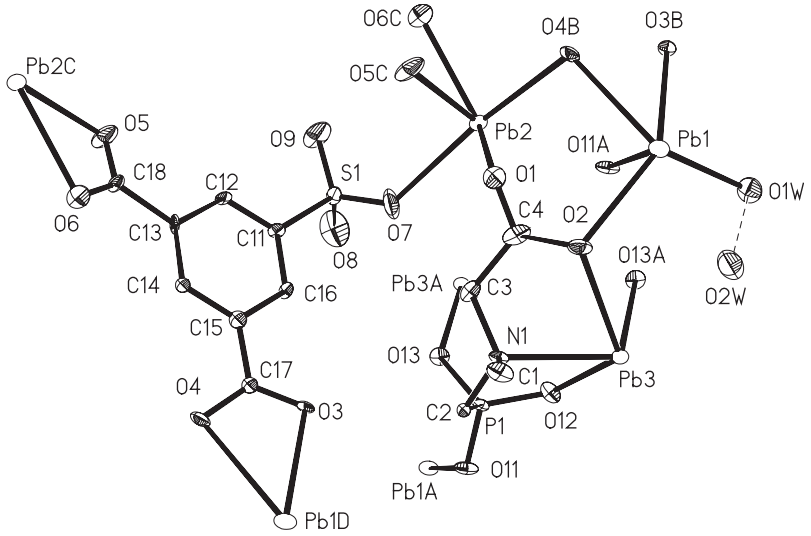

Fig. 1. ORTEP representation of the selected unit of compound **1**. The thermal ellipsoids are drawn at 50% probability. The dotted line represents the hydrogen bond.

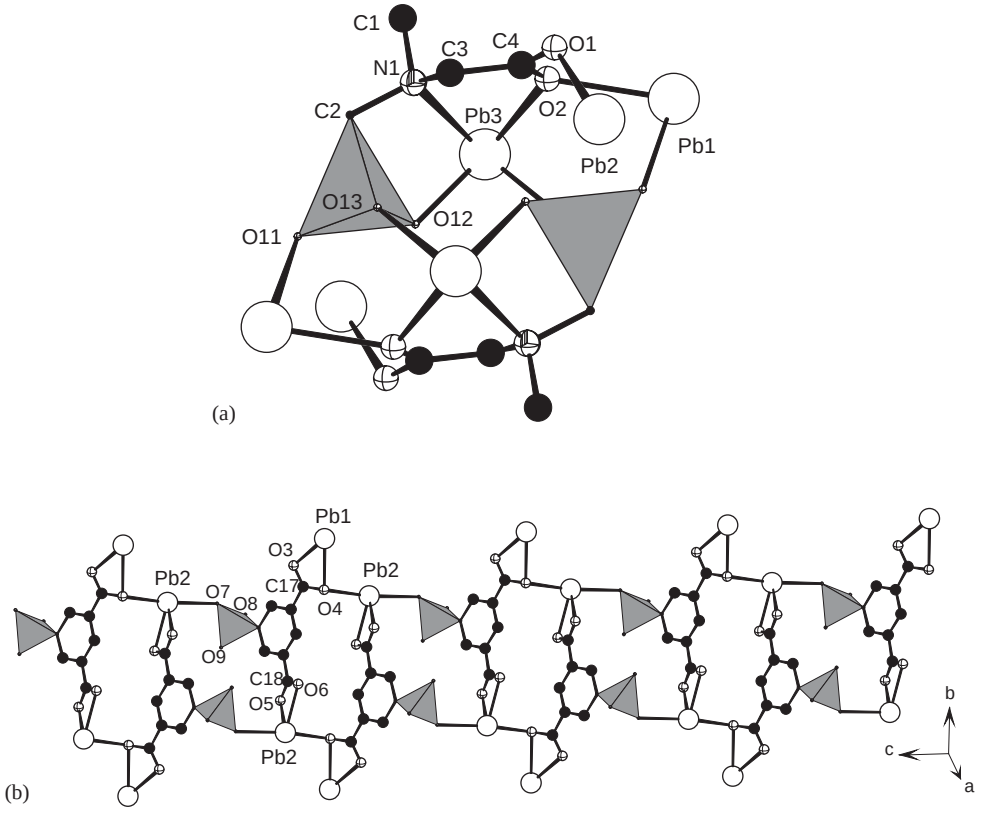

Fig. 2. (a) A $Pb_6(L^1)_2$ hexanuclear phosphonate unit; (b) A 1D double chain of lead(II) carboxylate–sulfonate in compound **1**. The CSO_3 and CPO_3 tetrahedra are shaded in gray. Pb, C, O and N atoms are drawn as open, black, crossed and octanded circles, respectively.

was decomposed to give a new monophosphonate ligand H_2L^3. The structure of **2** features a 3D network. As shown in Fig. 4, there are two unique Pb(II) ions in the asymmetric unit of compound **2**. Pb1 is 6-coordinated by two phosphonate oxygens and one pyridyl nitrogen atom of three HL^3 ligands, as well as three carboxylate oxygens from two BTS anions. Pb2 is 5-coordinated by two phosphonate oxygens of two HL^3 ligands, and two carboxylate oxygens and one sulfonate oxygen from two BTS anions. The Pb1 has a ψ-PbO_5 octahedral geometry in which the sixth coordination site is occupied by the lone pair electrons. The coordination

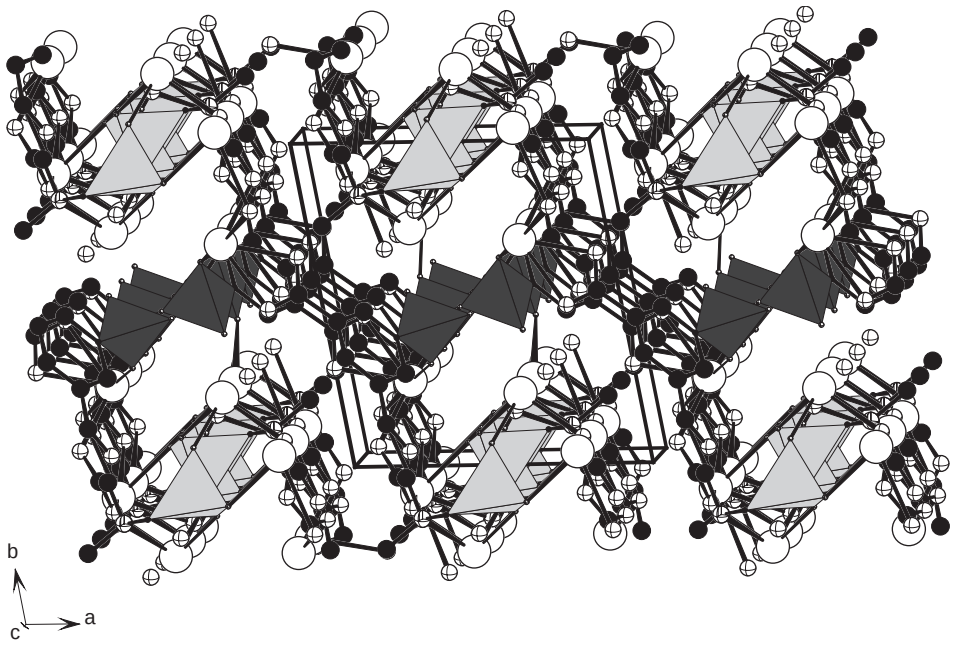

Fig. 3. View of the structure of **1** down the *c*-axis. The CSO_3 and CPO_3 tetrahedra are shaded in dark and light gray, respectively. Pb, C, O and N atoms are drawn as open, black, crossed and octanded circles, respectively.

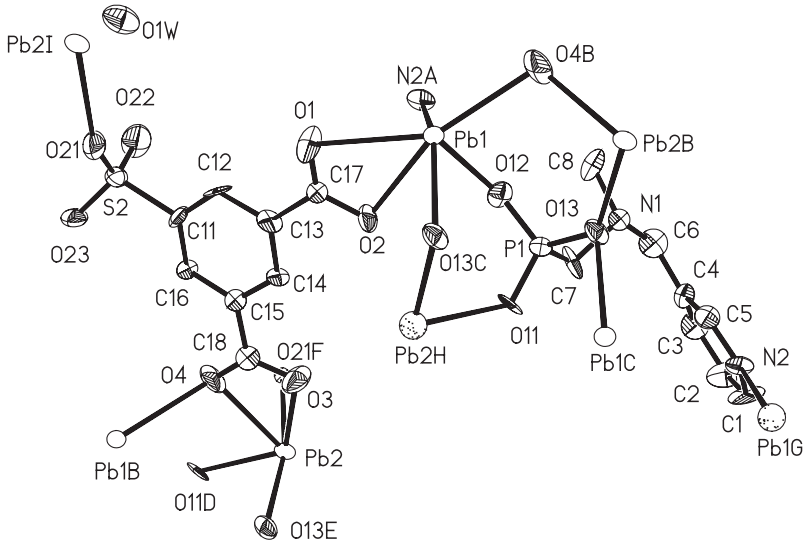

Fig. 4. ORTEP representation of the selected unit of compound **2**. The thermal ellipsoids are drawn at 50% probability.

geometry of Pb2 is a severely distorted PbO_5N octahedron, the distortion was caused by the lone pair electrons. The Pb–O distances range from 2.318(16) to 2.785(17) Å, which are comparable to those in **1** and other lead(II) phosphonates [9,12–14].

Based on the requirement of charge balance as well as P–O, C–O and S–O distances, the HL^3 phosphonate anion is −1 and the sulfonate-carboxylate BTS ligand is −3 in charge. The amino group N1 of the phosphonate ligand is protonated. The phosphonate anion is pentadentate and bridges with five Pb(II) ions. O13 of the phosphonate group is μ_2-bridging, whereas O11 and O12 are unidentate. It is interesting to note that the pyridyl N atom is also involved in the metal coordination. The BTS^{3-} anion is hexadentate and bridges with four Pb(II) ions. One carboxylate group (O1–C17–O2) is bidentate chelating, whereas the other one composed of O3–C18–O4 is μ_3 chelating and bridging. The sulfonate BTS ligand is unidentate (O21). O22 and O23 remain non-coordinated.

The interconnection of Pb1 and Pb2 atoms by the phosphonate groups resulted in a $Pb_4(HL^3)_2$ unit, and such neighboring units are bridged by the pyridine groups to form a 1D double chain along the *a*-axis

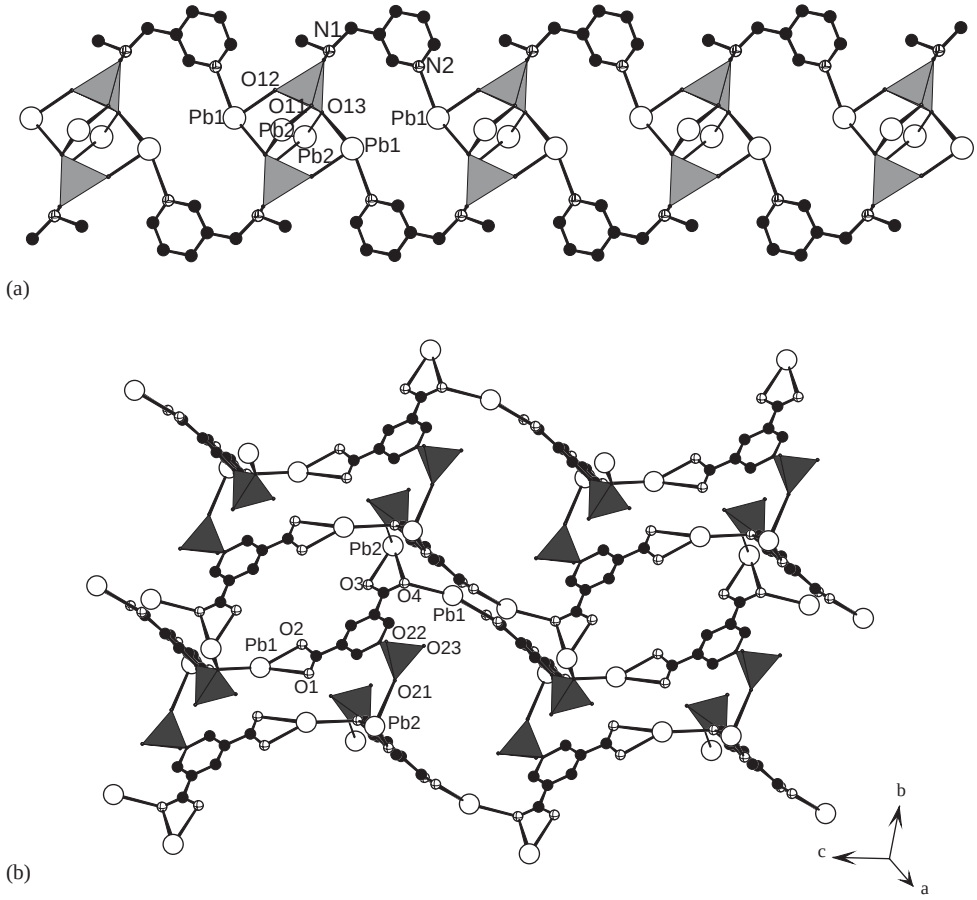

Fig. 5. (a) A 1D double chain of lead(II) phosphonate; (b) A 2D layer of lead(II) carboxylate–sulfonate in compound **2**. The CSO_3 and CPO_3 tetrahedra are shaded in dark and light gray, respectively. Pb, C, O and N atoms are drawn as open, black, crossed and octanded circles, respectively.

(Fig. 5a). The interconnection of the Pb(II) ions by bridging and chelating BTS anions leads to a 2D layer (Fig. 5b). The cross-linkage of the above two building units resulted in a 3D network with large cavities (Fig. 6). The size of the cavity is estimated to be $12.0 \times 16.0\,\text{Å}^2$ based on crystal structure. The pyridyl rings and two water molecules are located at these micropores. The lattice water molecule O1W is hydrogen bonded with the non-coordinated sulfonate oxygen O22 with an O···O separation of 2.914(20) Å. The protonated N1 atom forms a hydrogen bond with the other non-coordination sulfonate oxygen (O23). The N1···O23 (symmetry code: $x+1,\ 1/2-y,\ -1/2+z$) separation is 2.912(25) Å.

The XRD powder patterns for compounds **1** and **2** were collected on a Philips X'Pert-MPD diffractometer using graphite-monochromated Cu$K\alpha$ radiation in the angular range of $2\theta = 5 - 70°$. The measured powder patterns match with the ones simulated from their single crystal structure data, thus both compounds were obtained as a single phase.

The IR spectra of both compounds **1** and **2** were measured. The broadband at 3442 and 3068 cm^{-1} for compound **1** can be assigned to the O–H stretching vibrations, such bands appeared at 3419 and 3059 cm^{-1} for compound **2**. The asymmetric vibration absorption band of the sulfonate group appeared at 1200 cm^{-1} in compound **1** and at 1196 cm^{-1} in compound **2**. Absorption band around 623 cm^{-1} is characteristic band for υ_{S-O} of the sulfonate group. The two strong bands at 1593 and 1531 cm^{-1} for compound **1** or 1599 and 1543 cm^{-1} for compound **2** correspond to the antisymmetric stretching bands of the carboxylate groups. The symmetric stretching bands of the carboxylate group appeared around 1435 and 1431 cm^{-1}, respectively for compounds **1** and **2**. The bands from 900–1100 cm^{-1} are due to the stretching vibrations of the tetrahedral CPO_3 group.

The TGA diagram of **1** shows three main steps of the weight losses (Fig. 7). The first step is loss of one lattice water molecules, which started at 78°C and was completed at 243°C, the observed weight loss of 1.68% is in good agreement with the calculated value (1.69%). The second step covering a temperature range of 356–530°C corresponds to the burning of BTS and phosphonate ligands. The third step overlapped with the second one and continued up to 1000°C, during which the compound was further decomposed. The final product is a mixture of $Pb_2P_2O_7$ and PbO based on

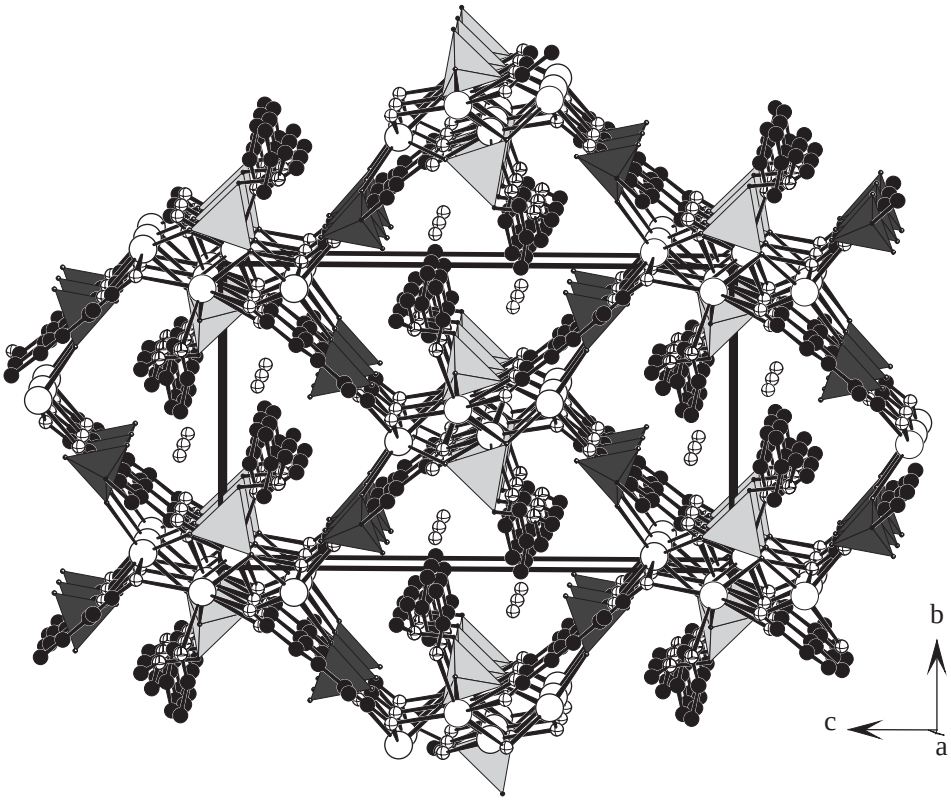

Fig. 6. View of the structure of compound **2** down the *a*-axis. The CSO_3 and CPO_3 tetrahedra are shaded in dark and light gray, respectively. Pb, C, O and N atoms are drawn as open, black, crossed and octanded circles, respectively.

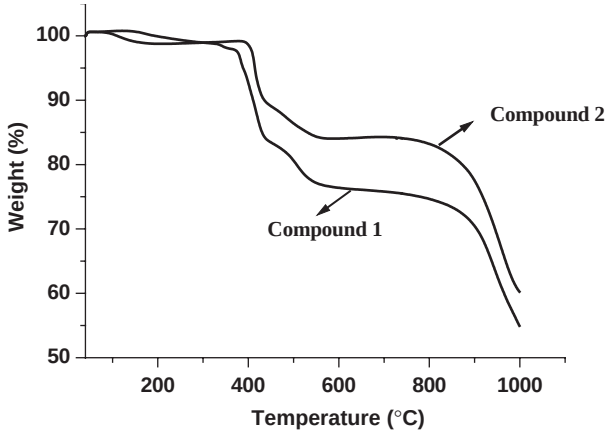

Fig. 7. TGA curves of compounds **1** and **2**.

the XRD powder diffraction. The total weight loss is 45.0%. The TGA curves of **2** exhibit also three steps of weight losses (Fig. 7). The first step is release of the partial lattice water molecules, which started at 92°C and was completed at 295°C, the observed weight loss of 1.3% is slightly less than the theoretical one (2.02%). The second weight loss starting at 392°C and continued up to 550°C, which corresponds to the burning of BTS and phosphonate ligands. The third step covers a temperature range of 760–1000°C, corresponding to the further decomposition of the compound. The final product is a mixture of PbO and $Pb_2P_2O_7$ based on XRD powder diffraction. The total weight loss is of 40.1% (Fig. 7).

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