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Transformation of mixed anionic coordination polymers of lead

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ABSTRACT

The coordination polymerization from lead(II) nitrate on reaction with 4-nitrobenzoic acid and pyridine *N*-oxide at room temperature passes through stepwise ligand substitution reaction. An intermediate polymer $[Pb(NB)(PyO)_2(NO_3)]_n$ (where NB = 4-nitrobenzoate, PyO = Pyridine *N*-oxide) is formed to give the final polymer $[Pb(NB)_2(PyO)]_n$. A hydrated mononuclear complex $[Pb(NB)_2(PyO)(H_2O)]$ is also formed if rigorous anhydrous condition is not maintained. The reaction is extended to 4,4′-bipyridyl *N*-oxide (BPNO), which initially gives a coordination polymer $[Pb_2(NO_3)(NB)_3(BPNO)_2]_n$ which gets converted to another coordination polymer $[Pb(NB)_2(BPNO)_2]_n$. All these complexes are structurally characterized.

1. Introduction

The field of hybrid inorganic–organic framework materials specially coordination polymers has emerged as a subject of intensive research not only for their fascinating topologies but also for the promise they have offered as storage materials, catalyst, and so on [1–10]. General strategy during syntheses of such hybrid materials involves reaction of a ligand with appropriate coordination modes, and a metal ion that can act as a multidimensional hub to get a thermodynamically stable polymer [1–13]. A good number of reports regarding coordination polymers of lead with carboxylate ligand are available in the literature [14–17]. Recently, it has been shown that both hemi and hollow directed coordination spheres can be generated in lead carboxylates [18–19]. Presence of underlying s orbital of lead results in interesting coordination behavior owing to the inert pair effect [18–21]. Numbers of coordination polymers are prepared through solvothermal reactions and their transformations to another coordination polymer are reported [22–27]. Most of these transformations are driven by thermal or photochemical means and are basically solid state transformations [22–27]. Unfortunately, intermediate species formed during such reactions are often not isolated. Alternatively, solution chemistry can still be very useful for syntheses of coordination polymers, since, transient complexes formed during the reaction can sometimes be isolated [28–29]. Moreover, there is need for systematic study, especially in synthesizing mixed anionic coordination polymers and in transformation of a coordination back-bone of one coordination polymer to another through chemical reactions. Reactions carried out in solid state, followed by crystallization can also be useful in describing inter conversion of different supramolecular isomers or polymorphs which may eventually show path for controlled and selective syntheses [30]. With the objective of understanding possible transformation among polymeric species, we have studied the transformation of coordination polymers of lead derived from 4-nitrobenzoate and aromatic *N*-oxides in solution. Here we report the transformation of mixed anionic polymeric species containing pyridine *N*-oxide either to supramolecular self assembly or to homo anionic coordination polymer. In the case of 4,4′-bipyridyl *N*-oxide (BPNO) complex, inter conversion of one mixed anionic 2-D polymeric species to a homo anionic 2-D polymer is reported.

2. Experimental

Synthesis of complexes $[Pb(NB)(PyO)(NO_3)]_n$ **(1)**, $[Pb(NB)_2$–$(PyO)(H_2O)]$ (**2**) and $[Pb(NB)_2(PyO)]_n$ (**3**) $[Pb_2(NO_3)(NB)_3(BPNO)_2]_n$ (**4**), $[Pb(NB)_2(BPNO)_2]_n$ **(5)**:

To a solution of 4-nitrobenzoic acid (2 mmol, 0.334 g) in methanol (20 ml) methanolic solution of NaOH (2 mmol, 0.080 g, 5 ml) was added. After stirring this reaction mixture for about 30 min $Pb(NO_3)_2$ (1 mmol, 0.331 g) was added and stirred for another 30 min and then PyO (1 mmol, 0.095 g) was added. A small amount of toluene was added. The clear solution was then kept for crystallization and good quality colorless crystals of **1** were obtained after about 24 h. IR (KBr, cm^{-1}): 1573 (m), 1555 (m), 1470 (s), 1385 (s), 1350 (s), 1216 (m), 832 (m). ^1H NMR (DMSO-d_6, ppm): 8.29 (d, 2H, J = 8.8 Hz), 8.21 (d, 2H, *J* = 7.2 Hz), 8.11 (d, 2H, *J* = 8.8 Hz), 7.41 (m, 2H,), 7.34 (t, 1H, *J* = 7.6 Hz). Elemental *Anal.* Calc. for $C_{17}H_{14}N_4O_9Pb$: C, 32.61; H, 2.24. Found: C, 32.65; H, 2.26%.

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The complex **1** along with the mother liquor was kept and from this crystals of **2** were obtained after 2 days. Isolated IR (KBr, cm^{-1}): 1615 (m), 1578 (s), 1509 (m), 1384 (s), 1351 (s), 1220 (m), 831 (m). ^{1}H NMR (DMSO-d_6, ppm): 8.22 (m, 4H), 8.09 (d, 2H, J = 8.4 Hz), 7.41 (m, 2H), 7.33 (t, 1H, J = 7.6 Hz). Elemental *Anal.* Calc. for $C_{19}H_{15}N_3O_{10}Pb$: C, 35.05, H, 2.31. Found: C, 35.12, H, 2.02%.

Crystals of the complex **3** were obtained when **2** was dissolved in methanol and crystallized. Isolated yield: 93%. IR (KBr, cm^{-1}): 1621 (m), 1583 (s), 1510 (m), 1474 (m), 1384 (s), 1350 (s), 1227 (m), 832 (m). ^{1}H NMR (DMSO-d_6, ppm): 8.22 (m, 4H), 8.11 (d, 2H, J = 8.4 Hz), 7.41 (m, 2H), 7.33 (t, 1H, J = 7.6 Hz). Elemental *Anal.* Calc. for $C_{19}H_{13}N_3O_9Pb$: C, 35.93, H, 2.05. Found: C, 35.91; H, 2.11%.

For the synthesis of **4;** 4-nitrobenzoic acid (2 mmol, 0.334 g) and sodium hydroxide (2 mmol, 0.080 g, 5 ml) was dissolved in methanol (20 ml). After stirring this reaction mixture for about 30 min lead nitrate (1 mmol, 0.331 g) and 4,4′-bipyridine *N*-oxide (0.188 g, 1 mmol) was added and stirred for another 30 min. Yellow colored precipitate appeared which was dissolved in DMSO. The clear solution was then kept for crystallization and good quality colorless crystals of **4** were obtained after about 8 h. Isolated yield 48%. IR (KBr, cm^{-1}): 1619 (m), 1563 (s), 1519 (m), 1470 (m), 1384 (s), 1342 (s), 1214 (m), 833 (m). Elemental *Anal.* Calc. for $C_{31}H_{20}N_6O_{17}Pb_2$: C, 32.01 H, 1.73. Found: C, 32.15; H, 2.06%.

The complex **4** along with the mother liquor was kept and from this crystals of **5** were obtained after 24 h. Isolated yield: 65%. IR (KBr, cm^{-1}): 1619 (m), 1580 (s), 1473 (m), 1389 (m), 1348 (s), 1231 (m), 840 (m). Elemental *Anal.* Calc. for $C_{24}H_{16}N_4O_{10}Pb$: C, 39.61 H, 2.24. Found: C, 39.45; H, 2.31%.

The X-ray diffraction data were collected at 296 K with Mo Kα radiation (λ = 0.71073 Å) using a Bruker Nonius SMART CCD diffractometer equipped with a graphite monochromator. SMART software was used for data collection and also for indexing the reflections and determining the unit cell parameters; the collected data were integrated using SAINT software. The structures were solved by direct methods and refined by full-matrix least-squares calculations using SHELXTL software. All the non-H atoms were refined in the anisotropic approximation. The crystallographic parameters of the compounds studied are given in Table 1.

The elemental analyses of the complexes were carried out with pure crystals and satisfactory results are obtained, however, from bulk materials we could not get satisfactory data.

3. Results and discussion

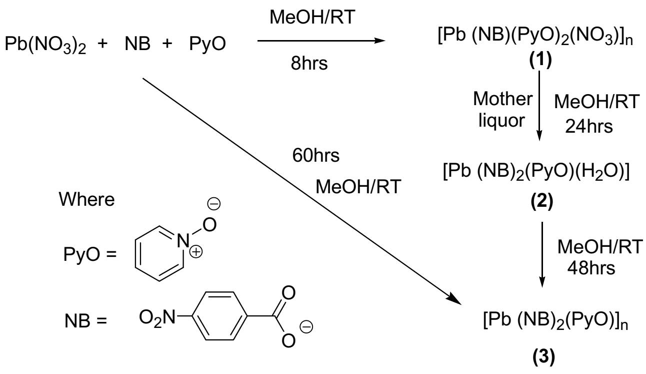
We have observed that the reaction of lead(II) nitrate with 4-nitrobenzoate and pyridine *N*-oxide in methanol initially gives a mixed anionic coordination polymer **1** having composition $[Pb(NB)(PyO)_2(NO_3)]_n$ (where NB = 4-nitrobenzoate, PyO = Pyridine *N*-oxide). This compound on keeping dissolved in the mother liquor for 24 h gets converted to a mononuclear complex **2** having composition $[Pb(NB)_2(PyO)(H_2O)]$. Finally, the complex **2** slowly gets converted to a new coordination polymer **3** $[Pb(NB)_2(PyO)]_n$ on stirring in methanol solution. The reaction is presented in Scheme 1.

The complex **1** has its characteristic IR absorptions at 1555 cm^{-1} due to the metal bound chelating nitrate group and at 1216 cm^{-1} due to pyridine *N*-oxide, the carbonyl frequency appears at 1573 cm^{-1}. In the ^{1}H NMR spectra, the complex has the A_2B_2 pattern for the 4-nitrobenzoate group at 8.1 ppm and 8.3 ppm, in addition to the aromatic peaks of pyridine *N*-oxide.

Fig. 1b shows the structure of the coordination polymer **1**. Each of the metal centers in **1** is octa coordinated in which 4-nitrobenzoate ligands satisfy three of the coordination sites and are in bidentate bridging mode. The nitrate groups in the polymer are chelating and each lead is associated with three different *N*-oxide oxygen two bridging and the other mono-dentate. The Pb–O distance for the bridging *N*-oxide is 2.746 Å whereas that for the mono-dentate *N*-oxide is 2.44 Å. The Pb–O distances for the nitrate ligand are Pb1–O1, 2.82 Å; Pb1–O2, 2.57 Å. A careful look on the coordination polymer shows that it comprises mononuclear building blocks, which are of hemi directed geometry (Fig. 1a), and on self-assembling such hemi directed units result in the coordination polymer **1** (Fig. 1b) with hollow directed lead centers.

Table 1
Crystallographic parameters for complexes **1–5**

Compound number	**1**	**2**	**3**	**4**	**5**
Formulae	$C_{17}H_{14}N_4O_9Pb$	$C_{19}H_{15}N_3O_{10}Pb$	$C_{19}H_{13}N_3O_9Pb$	$C_{31}H_{20}N_6O_{17}Pb_2$	$C_{24}H_{16}N_4O_{10}Pb$
Formula weight	625.51	652.53	634.51	1162.91	727.60
Crystal system	monoclinic	triclinic	monoclinic	monoclinic	monoclinic
Space group	*P*2(1)/*n*	$P\bar{1}$	*P*2(1)/*c*	*P*2(1)/*c*	*P*2(1)/*c*
a (Å)	12.1032(5)	6.8980(3)	10.62940(10)	17.6692(13)	13.061(2)
b (Å)	7.5460(3)	11.7165(5)	7.71560(10)	8.1098(6)	7.4870(11)
c (Å)	21.6511(8)	13.6190(7)	24.6416(3)	24.3357(17)	26.345(4)
α (°)	90.00	72.189(3)	90.00	90.00	90.00
β (°)	94.402(2)	83.549(3)	98.6340(10)	100.471(4)	108.057(10)
γ (°)	90.00	84.304(3)	90.00	90.00	90.00
V (Å^3)	1971.58(13)	1038.85(8)	1998.01(4)	3429.1(4)	2449.3(6)
Z	4	2	4	4	4
Density (Mg m^{-3})	2.107	2.080	2.109	2.253	1.973
Absorption coefficient (mm^{-1})	8.617	8.184	8.504	9.896	6.955
F(000)	1192	620	1208	2192	1400
Total number of reflections	19740	9628	26282	31952	25184
Reflections, $I > 2\sigma(I)$	4855	3757	4903	8458	5996
Maximum 2θ (°)	28.30	25.50	28.37	28.33	28.45
Ranges (*h*, *k*, *l*)	$-16 \geqslant h \geqslant 16$	$-5 \geqslant h \geqslant 8$	$-13 \geqslant h \geqslant 13$	$-21 \geqslant h \geqslant 23$	$-17 \geqslant h \geqslant 17$
	$-9 \geqslant k \geqslant 10$	$-14 \geqslant k \geqslant 14$	$-10 \geqslant k \geqslant 10$	$-10 \geqslant k \geqslant 10$	$-9 \geqslant k \geqslant 9$
	$-28 \geqslant l \geqslant 28$	$-16 \geqslant l \geqslant 16$	$-31 \geqslant l \geqslant 32$	$-31 \geqslant l \geqslant 32$	$-30 \geqslant l \geqslant 35$
Complete to 2θ (%)	99.2	96.9	97.9	99.0	97.0
Data/restraints/parameters	4855/0/280	3757/0/298	4903/0/289	8458/0/505	5996/0/352
Goodness-of-fit (F^2)	1.039	1.056	1.036	1.070	0.990
R indices [$I > 2\sigma(I)$]	0.0190	0.0508	0.0197	0.0260	0.0463
R indices (all data)	0.0275	0.0732	0.0275	0.0426	0.0826



Scheme 1.

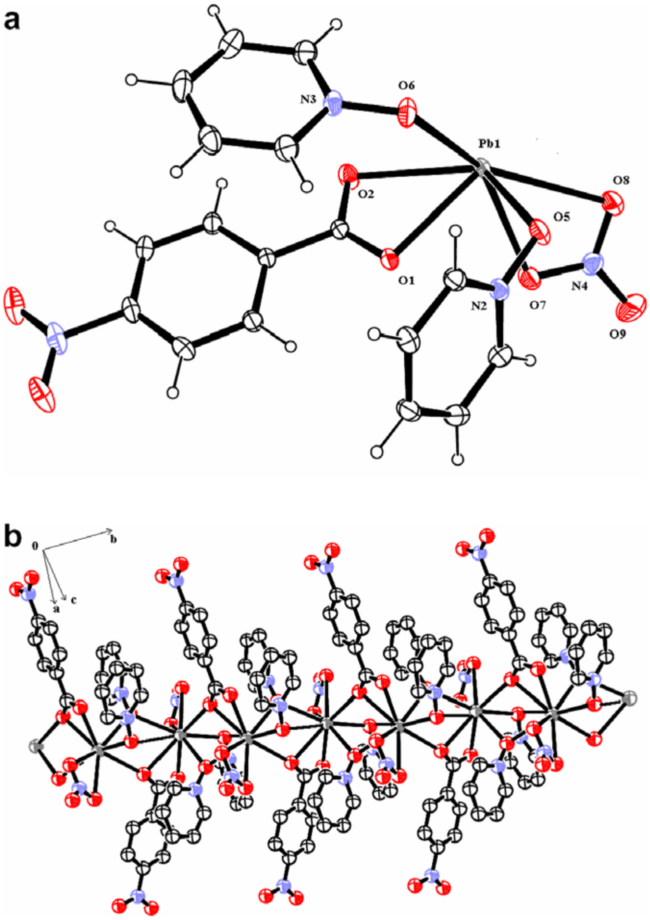


Fig. 1. (a) Smallest building block of the coordination polymer $[Pb(NB)(PyO)_2(NO_3)]_n$ (**1**) (b) the coordination polymer.

The coordination polymer **1** degrades under solution state and gets converted to the mononuclear complex **2**. The degradation process involves the removal of the nitrate group and incorporation of one 4-nitrobenzoate group. The complex **2** has hexa coordinated hemi directed lead center with two bidentate carboxylates, one PyO and one aqua ligand co-ordinated to the metal center. The aqua ligand in the complex is derived from the residual water in methanol. When dry methanol was used instead of ordinary methanol, complex **2** could not be isolated and complex **3** was found to be formed directly. From the crystal structure of the complex it is seen that the Pb–O bonds to the 4-nitrobenzoate groups in **2** (Pb1–O1, 2.78 Å; Pb1–O2, 2.37 Å; Pb1–O5, 2.38 Å; Pb1–O6, 2.77 Å) are much stronger than those in **1** (Pb1–O1, 2.46 Å; Pb1–O2, 2.72 Å). This may be one of the various factors responsible for the degradation of the coordination polymer as it leads to formation of much stronger bonds. Also the Pb1–O9 distance for the *N*-oxide ligand here is 2.44 Å. The complex self assembles through weak O2···H–O10 (*d*D–H 0.818 Å; *d*D···A, 2.781 Å; ∠D–H···A, 161.58°), O6···H–O10 (*d*D–H 0.833 Å; *d*D···A, 2.792.Å; ∠D–H···A, 141.42°) interactions and π···π interactions between the aromatic rings of 4-nitrobenzoate ligand (Fig. 2). The Pb1···O10 distance is 2.612 Å. The aromatic rings of the 4-nitro benzoate groups are stacked parallel with the nitro-groups slightly off-set with a distance of separation between C2 and C9 of two rings is 3.392 Å (Fig. 2) which is well within the limit for having center to center π···π stacking interactions.

It is interesting to note that the lead coordination polymers are used for reduction of nitrate as well as nitration of aromatic substrates [31]. However, in our case we could not obtain any nitrated product from the reaction of resorcinol with the complex **1**; what we could obtain is the complex **2** and the solution showed the test for presence of nitrate anion in solution. The coordination polymer **1** also reacts with sodium salt of 4-nitrobenzoic acid to give **3**. The coordination polymer **3** is also formed on reaction of lead(II) nitrate with sodium salt of 4-nitrobenzoic acid and pyridine *N*-oxide for a longer reaction time. We have observed that the complex **2** is unstable and slowly gets converted to the coordination polymer **3** by losing the water molecule in the presence or absence of sodium 4-nitrobenzoate.This suggests both ligand disproportionation as well as ligand exchange process to be possible for interconversion of **2** to **3**. However, we have not been successful in characterizing any other product formed from ligand disproportionation of **3**. The complex **2** is thus a side product in this reaction and apparently can be considered to be an isolable reaction intermediate. The complex **3** has IR absorptions at 1227 cm^{-1} due to pyridine *N*-oxide and 1621 cm^{-1}, 1583 cm^{-1} due to the two carboxylate ligands. The complex **3** has a seven coordinated geometry around lead ions and remains as one dimensional coordination polymer. Six of these coordinations come from the 4-nitrobenzoate groups

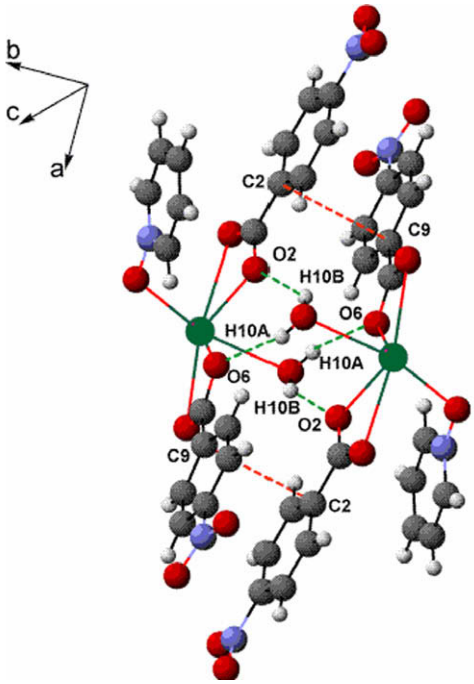
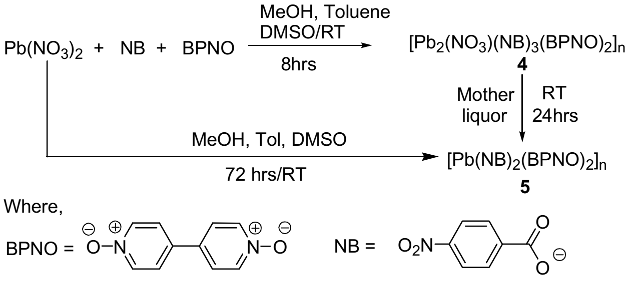


Fig. 2. The dimeric assembly of $[Pb(NB)_2(PyO)(H_2O)]$ showing intermolecular H-bonding interaction and π···π stacking interactions in (**2**).

while the seventh coordination is from a monodentate PyO ligand. It is the tendency towards expansion of coordination number of the metal that acts as the driving force towards the elimination of the aqua ligand and results in the polymerization of **2** to give **3**. The bond lengths of the Pb–O bonds in **3** are in the range of 2.40–2.83 Å. The structure of the complex with selected bond distances is shown in Fig. 3. The ^{1}H NMR spectra of the three complexes recorded in DMSO-*d*6 clearly indicates the presence of pyridine *N*-oxides and 4-nitrobenzoate groups (please refer to Supplementary material). The powder X-ray diffraction of the three complexes **1–3** are recorded (please refer to Supplementary material) and compared with the simulated pattern. It is found that in addition to desired peaks there are additional peaks in each case. Careful look at the powder pattern of **2** and **3**, shows that there is some amount of each of them in both the XRD patterns. This happens due to incomplete transformation of **2** to **3**.

Further support to these results comes from the fact that similar reactions of lead nitrate, 4-nitrobenzoate with 4,4′-bipyridyl *N*-oxide initially gives a mixed anionic two dimensional coordination polymer **4** which gets converted to another 2-D coordination polymer **5** in the reaction mixture with time. This conversion is shown in Scheme 2.

Both the complexes are characterized by single crystal XRD and also by other conventional spectroscopic techniques. The complex **4** has the composition $[Pb_2(NO_3)(NB)_3(BPNO)_2]_n$ and is featured by two crystallographically distinct Pb centers. One of these lead centers (Pb1) is octa co-ordinated (three from carboxylate oxygen, three from nitrate ligands and rest are from BPNO ligands) with a hollow directed geometry and contains tridentate nitrate ligands along with carboxylate and μ-oxo containing BPNO ligands. The other lead center (Pb2) has hexa coordinated hemi directed geometry. Four of this hexa coordination come from carboxylate oxygens and rest are from BPNO ligands. Pb1 is connected to another Pb1 through the tridentate nitrate groups while it is connected to Pb2 via *N*-oxide oxygens as well as carboxylate oxygen. It is interesting to note here that among all the Pb–O bonds in **4** the ones to the nitrate ligand viz. Pb1–O29 (2.91 Å), Pb1–O30 (2.87 Å), Pb1–O31(2.82 Å) are the weaker. Bond lengths of all other bonds lies in the range 2.47–2.65 Å. The complex **4** has IR absorptions at 1519 cm^{-1} characteristic of the nitrate ligand. The peaks at 1619 cm^{-1} and 1563 cm^{-1} are due to the carboxylate ligand and at 1214 cm^{-1} is due to the N–O stretching of BPNO ligand. It has a two dimensional network structure growing along *b* and *c* crystallographic axes as shown in Fig. 4.



Scheme 2.

It has been observed that the complex **4** is unstable in the reaction mixture and on leaving dissolved in the mother liquor for 24 h gives a new two dimensional coordination polymer **5** having composition $[Pb(NB)_2(BPNO)_2]_n$ with the removal of the weakly bound nitrate ligands. This time the complex lacks any IR absorptions around 1520 cm^{-1} which was present in **4** due to the nitrate ligand. The peaks at 1619 cm^{-1} and 1580 cm^{-1} are due to the carboxylate ligand and at 1231 cm^{-1} due to the BPNO ligand. Each of the lead centers in **5** has a hemi directed hepta coordinated geometry, four of which are from two chelating carboxylate ligands and rest are from bridging as well as monodentate BPNO ligands. Here the Pb–O bond lengths lie in the range 2.48–2.77 Å. In this case also the formation of comparatively stronger Pb–O (carboxylate) forces the dissociation of the Pb–O (nitrate) bonds thereby forming the homo anionic coordination polymer from the mixed anionic one. The dimensionality of this 2-D coordination polymer extends along the *b* and *c* crystallographic axes as shown in Fig. 5. The ^{1}H NMR spectra of both these complexes are recorded in DMSO-*d*6 and shows the presence of 4-nitrobenzoate group as well as the BPNO ligand (please refer to Supplementary material).

In both these reaction schemes it is observed that mixed anionic coordination polymer containing 4-nitrobenzoate as well as nitrate is formed initially with the excess 4-nitrobenzoate remaining in reaction mixture. These mixed anionic complexes are unstable in the mother liquor and gets converted to polymeric species containing only 4-nitrobenzoate as anionic ligand through substitution of the nitrate ligand with a 4-nitrobenzoate. There are examples of lead complexes derived from *N*-oxide of nictonic acid and they also lead to coordination polymers of lead and in that case the ligand itself has *N*-oxide and carboxylic acids, however, our system is different in a sense that we are being able to manipulate the stoichiometry of the reactant to control the reactions which are not possible when two different types of coordinating sites are bound to rigid system [14].

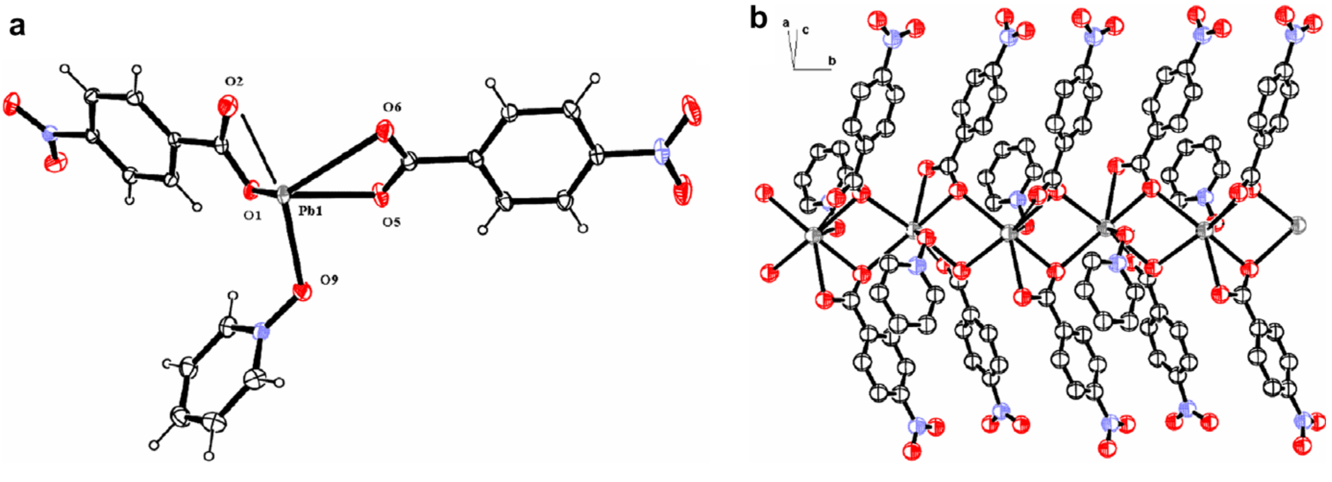


Fig. 3. (a) The building block of coordination polymer $[Pb(NB)_2(PyO)]_n$ (**3**) (b) the polymeric structure.

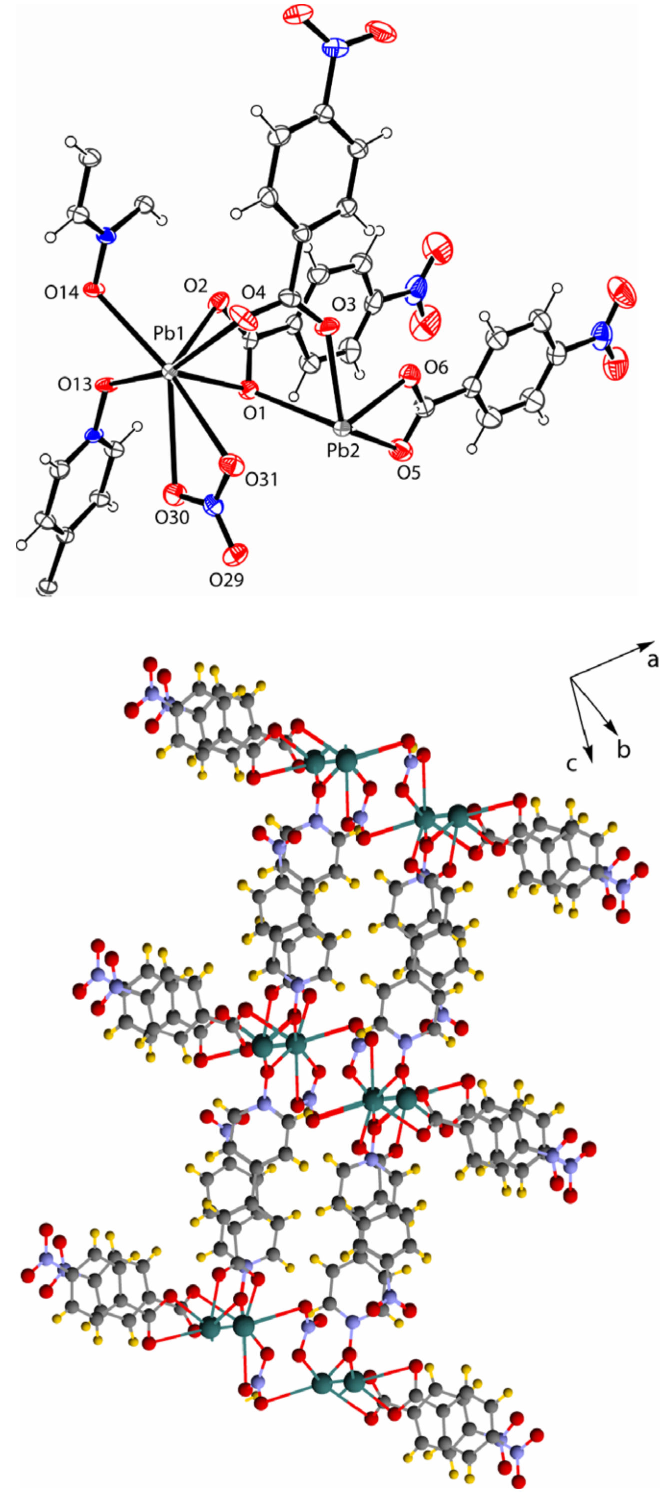


Fig. 4. (a) The building block of coordination polymer $[Pb_2(NO_3)(NB)_2(BPNO)_2]_n$ (b) 2-D polymeric structure.

In conclusion we have isolated and characterized mixed anionic coordination polymers in the reaction of lead(II) nitrate with benzoate and *N*-oxide ligands that are intermediates formed during the reactions. In addition to this, the conversion of a mixed anionic coordination polymer to homo anionic coordination polymer and also formation of a self-assembled mono-nuclear coordination complex from mixed anionic coordination polymer through hydration is demonstrated.

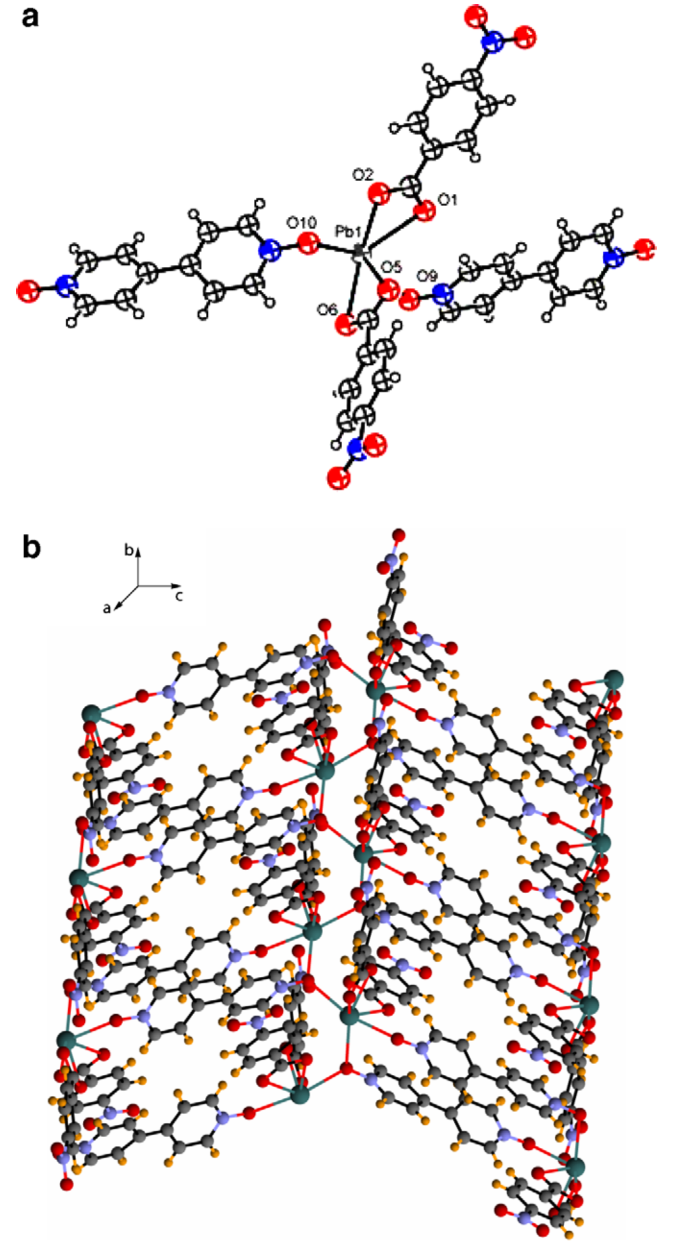


Fig. 5. (a) The building block of coordination polymer $[Pb(NB)_2(BPNO)_2]_n$ (b) 2-D polymeric structure.

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Appendix A. Supplementary material

CCDC 673855, 673856, 673857, 684603 and 684604 contain the supplementary crystallographic data for **1**, **2**, **3**, **4** and **5**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. The CIF files for the five complexes and the ^{1}H NMR spectra of the complexes **1**–**5** and the powder X-ray diffraction pattern of **1**–**3** are available. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2008.07.018.

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