Polyhedron 29 (2010) 2985–2990

Contents lists available at ScienceDirect

Polyhedron

journal homepage: www.elsevier.com/locate/poly

Synthesis, structural characterization and properties of one-dimensional coordination polymers of cobalt(II)- and nickel(II)-phosphonate complexes with 2,2′-bipyridine as a secondary ligand component: Observation of both *cis* and *trans* conformations of a diphosphonic acid

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A R T I C L E I N F O

Article history:
Received 6 May 2010
Accepted 7 August 2010
Available online 18 August 2010

Keywords:
Transition metal phosphonates
Hydrothermal synthesis
p-Xylylenediphosphonic acid
Crystal structure
Supramolecular interactions

A B S T R A C T

Two new isomorphous cobalt and nickel phosphonates $[M^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$, M = Co (compound **1**), M = Ni (compound **2**) were hydrothermally synthesized from *p*-xylylenediphosphonic acid (LH_4) and the corresponding metal salts with 2,2′-bipyridine as secondary ligand component. Both the compounds **1** and **2** are characterized by routine elemental analyses, IR-, electronic-spectral analyses, thermogravimetric studies and unambiguously characterized by single crystal X-ray crystallography. The structures were refined in monoclinic space group *C*2/*c*. The crystal structure consists of 1D $[M(2,2'\text{-bipy})_2LH_4]^{2+}$ chains and $[LH_2]^{2-}$ anions. The flexibility of non-rigid ligand *p*-xylylenediphosphonic acid (LH_4) tends to adopt a rare *cis* conformation in the crystal structure to meet the coordination requirement of the metal center from the usual *trans* conformation. The hydrogen bonding in the crystal structure leads to cylindrical tubes that extend via *p*-xylylenediphosphonic acid resulting in a 2D supramolecular sheet throughout the crystal. Compounds **1** and **2** are additionally characterized by thermogravimetric studies.

1. Introduction

Metal organophosphonates are among the earliest and most extensively studied examples of inorganic–organic hybrid materials. Numerous compounds of di-, tri-, tetra-, penta- and hexa-valent metals with a variety of organophosphonic ligands have been studied because of their catalytic, ion exchange, sensor and nonlinear optical properties [1–6]. Among metal organophosphonates, metal-organodiphosphonates are very common in recent literatures [7–9]. The prototypical metal-organodiphosphonate have general formula $M_x[O_3P(\text{organic spacer})PO_3]_y$. Its structural modification can be achieved by introducing secondary linkers [10–14]. Metal phosphonates, based on *p*-xylylenediphosphonic acid, are widely studied with a range of transition metals and lanthanides [15–18]. Majority of these structures consist of typical $\{M_xO_3P\}$ layers separated by organic spacers. *p*-Xylylenediphosphonic acid is a good example of flexible ditopic ligand in which ($-CH_2-P-$) bond can adopt *cis*/*trans* conformation with respect to benzene ring as shown in Scheme 1. The flexibility of ligand is necessary for structures to show some interesting properties like molecular switching derived from conformational change, adaptive recognition properties, organic molecular clips, etc. [19], and also the flexibility of ligand gives insight information of self assembly process. The final structures based on flexible ligands are prone to several synthetic conditions. It is also important to note that rotations along single bond leads to the loss of control in designing. These factors make the synthesis of coordination polymers based on such flexible ligands a challenging task [20].

We report here two metal-organodiphosphonate coordination polymers **1** and **2** that are based on *p*-xylylenediphosphonic acid as a primary linker and 2,2′-bipyridine (2,2′-bipy) as a secondary ligand component.

$[M^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$, M = Co (compound **1**), M = Ni (compound **2**).
$LH_4 = (HO)_2O{=}PCH_2C_6H_4CH_2P{=}O(OH)_2$.
$LH_2{}^{2-} = (HO)(O^-)O{=}PCH_2C_6H_4CH_2P{=}O(O^-)(OH)$.

In compounds **1** and **2**, the neutral ligand LH_4 acts as the linker in *cis* conformation via its coordination to the metal ion M^{2+} through P=O resulting in $[M^{II}(2,2'\text{-bipy})_2LH_4]^{2+}$ as a di-positive cation, which, in turn, is stabilized by the deprotonated anion $LH_2{}^{2-}$ in *trans* conformation. To our knowledge, this is the first instance of a metal-*p*-xylylenediphosphonate compound that is isolated with a secondary ligand component (here it is 2,2′-bipyridine) in its both *cis* and *trans* conformations. This is a unique system in the sense that, the same *p*-xylylenediphosphonic acid acts as coordinating

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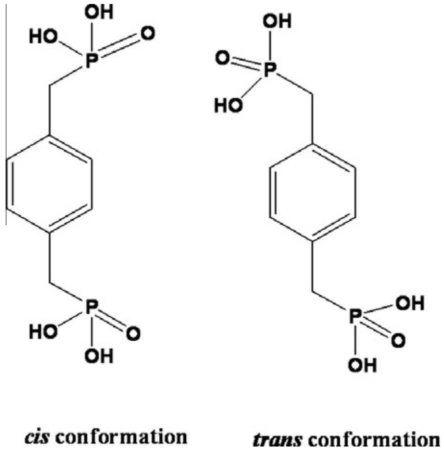
doi:10.1016/j.poly.2010.08.008



Scheme 1.

ligand in its neutral form (LH_4) and as the anion in its ionic form ($LH_2{}^{2-}$) in the ion pair compounds **1** and **2**.

2. Experimental

2.1. General considerations

All reactions were performed in a 23 mL Teflon lined autoclave vessel (Thermocon, India). The starting materials $Na_2MoO_4{\cdot}2H_2O$, $CoSO_4{\cdot}7H_2O$, $NiSO_4{\cdot}7H_2O$, 2,2′-bipyridine, hydrofluoric acid were obtained from Finar, Loba Chem. *p*-Xylylenediphosphonic acid was prepared according to the procedure reported in the literature [21].

2.2. Synthesis of compound 1

A mixture of $Na_2MoO_4{\cdot}2H_2O$ (0.038 g, 0.158 mmol), 2,2′-bipyridine (0.034 g, 0.221 mmol), $H_2PO_3(C_8H_8)PO_3H_2$ (0.115 g, 0.436 mmol), $CoSO_4{\cdot}7H_2O$ (0.123 g, 0.440 mmol), H_2O (10 g, 555 mmol), and concentrated HF (0.125 g) in mole ratio 1:1.3:2.75:2.78:3512 were stirred under air atmosphere for few hours. The solution was transferred into teflon-lined stainless steel vessel (25 mL), sealed and heated up to 200 °C for 96 h. By slow cooling of the reaction mixture to room temperature over 2 days red block crystals were obtained. Yield: (0.18 g, 45%). CHN analysis: *Anal.* Calc. for $C_{36}H_{38}N_4P_4O_{12}Co$: C, 47.96; H, 4.24; N, 6.21. Found: C, 48.12; H, 4.19; N, 6.29%. IR (KBr pellet) (υ/cm^{-1}) for **1**: 3425, 3088, 2744, 2648, 2334, 1658, 1597, 1512, 1469, 1440, 1255, 1141, 1097, 979, 927, 810, 771, 733, 684, 567, and 507.

2.3. Synthesis of compound 2

The same synthetic procedure was used to synthesize **2** as that for **1** except $NiSO_4{\cdot}7H_2O$ was used instead of $CoSO_4{\cdot}7H_2O$. Yield: (0.10 g, 25%). CHN analysis: *Anal.* Calc. for $C_{36}H_{38}N_4P_4O_{12}Ni$: C, 47.97; H, 4.24; N, 6.21. Found: C, 47.88; H, 4.28; N, 6.15%. IR (KBr pellet) (υ/cm^{-1}) for **2**: 3425, 3097, 3003, 2627, 2361, 1649, 1599, 1510, 1469, 1440, 1255, 1141, 1093, 978, 927, 771, 736, 684, 569, and 511.

2.4. X-ray crystallography

The data were collected at 298(2) K on a Bruker SMART APEX CCD area detector system [λ(Mo Kα) = 0.71073 Å], graphite monochromator, 2400 frames were recorded with an ω scan width of 0.3°, each for 10 s, crystal–detector distance 60 mm, collimator 0.5 mm. The data were reduced by using SAINTPLUS [22] and a multi-scan absorption correction using SADABS [22] was performed. Structure solution and refinement were done using programs of SHELX-97 [23]. All nonhydrogen atoms were refined anisotropically. Hydrogen atoms on the C atoms of the *p*-xylylenediphosphonic acid were introduced on calculated positions and were included in the refinement riding on their respective parent atoms. All the hydrogen atoms of the hydroxyl groups were identified by the Fourier electron density and refined freely, except H6a of O6 atom.

3. Results and discussion

Compounds **1** and **2** have been synthesized by a direct one pot reaction of MSO_4 (M = Co^{2+} and Ni^{2+}) with phosphonic acid, 2,2′-bipyridine and sodium molybdate under hydrothermal conditions in the presence of hydrofluoric acid. Compounds **1** and **2** were obtained as well-formed blocks. Both compounds are non-hygroscopic and stable in air and insoluble in common organic solvents and in water. Sodium molybdate, used in this synthesis, is not the part of the isolated compounds **1** and **2**. We believe that sodium molybdate plays an important role in this synthesis because the crystals of compounds **1** and **2** cannot be obtained, if we do not use sodium molybdate in the relevant synthesis.

Both compounds **1** and **2** that crystallize in a monoclinic *C*2/*c* space group are isomorphous. The structural details for compound **2** are described herewith. The basic crystal data with refinement parameters of both the compounds are provided with Table 1, and selected bond distances and angles are provided with Table 2. The thermal ellipsoidal plot of the molecular structure of compound **2** is shown in Fig. 1. As shown in Fig. 1, the nickel ion is in NiO_2N_4 octahedron constructed by four nitrogen atoms from two bipyridine molecules and two oxygen atoms from two phosphonic acids. The phosphonic acid is coordinated to the metal atoms with P═O groups on both sides which is in good accordance with the P–O bond length of 1.476 Å and thereby the charge is balanced by the uncoordinated phosphonic acid which is deprotanated from LH_4 to $LH_2{}^{2-}$. In the crystal structure, an extended nickel coordination complex {NiO_2N_4} (chainlike) polymer is formed by the use of LH_4 (*p*-xylylenediphosphonic acid) as a linker. Along the chain, the nickel(II) ion has a coordination number of six (6) and is in the center of a distorted octahedron formed by four nitrogen donors from two bipyridine ligands and two terminal oxygen atoms (P═O) from two different linkers. The formation of chain is schematically shown in Scheme 2. *p*-Xylylenediphosphonic acid

Table 1
Crystallographic data for $[M^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$, M = Co(**1**), Ni(**2**).

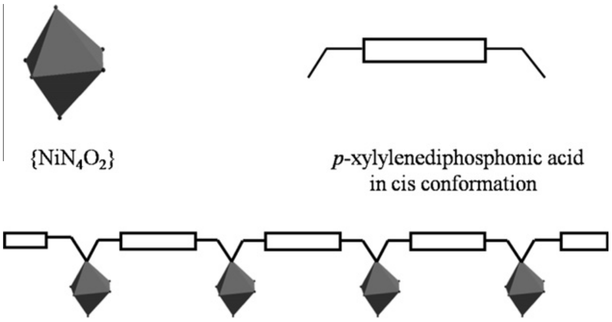
	1	**2**
Formula	$C_{36}H_{38}N_4O_{12}P_4Co$	$C_{36}H_{38}N_4O_{12}P_4Ni$
Formula weight	901.51	901.29
Crystal system	Monoclinic	Monoclinic
Space group	*C*2/*c*	*C*2/*c*
a (Å)	18.113(3)	18.051(4)
b (Å)	24.451(3)	24.356(4)
c (Å)	8.778(13)	8.776(16)
α (°)	90.00	90.00
β (°)	102.996(3)	103.119(4)
γ (°)	90.00	90.00
Z	4	4
ρ_{cal} (Mg m^{-3})	1.581	1.593
Goodness-of-fit on F^2	1.093	1.128
R_1 (F_0^2) [$I > 2\sigma(I)$]	0.0357	0.0532
wR_2 (F_0^2) [$I > 2\sigma(I)$]	0.0832	0.1155
R_1 (F_0^2) (all data)	0.0403	0.0617
wR_2 (F_0^2) (all data)	0.0855	0.1199
Largest difference in peak and hole (e $Å^{-3}$)	0.317 and −0.250	0.861 and −0.336

Table 2
Selected bond lengths (Å) and bond angles (°) of compounds **1** and **2**.

Distances	Bond lengths	Angles	Bond angles
*[Co^{II}(2,2′-bipy)$_2$$LH_4$]$_n$[$LH_2$]$_n$ (**1**)*			
Co(1)–N(1)	2.145(19)	N(1)–Co(1)–N(1)$^{\#1}$	169.18(11)
Co(1)–N(2)	2.140(2)	O(3)$^{\#1}$–Co(1)–N(2)	164.41(7)
Co(1)–O(3)	2.045(17)	O(3)–Co(1)–N(1)	98.79(7)
P(1)–O(1)	1.543(18)	N(2)–Co(1)–N(2)$^{\#1}$	92.49(11)
P(1)–O(2)	1.554(19)	N(1)$^{\#1}$–Co(1)–N(2)$^{\#1}$	76.01(8)
P(1)–O(3)	1.478(17)	O(3)$^{\#1}$–Co(1)–O(3)	96.16(11)
P(2)–O(4)	1.513(17)	O(3)$^{\#1}$–Co(1)–N(1)	88.46(7)
P(2)–O(5)	1.500(17)	N(1)$^{\#1}$–Co(1)–N(2)	96.39(11)
P(2)–O(6)	1.552(2)		
*[Ni^{II}(2,2′-bipy)$_2$$LH_4$]$_n$[$LH_2$]$_n$ (**2**)*			
Ni(1)–N(1)$^{\#2}$	2.083(3)	N(1)–Ni(1)–N(1)$^{\#2}$	171.01(16)
Ni(1)–N(2)$^{\#2}$	2.087(3)	O(3)$^{\#2}$–Ni(1)–N(2)	166.43(11)
Ni(1)–O(3)	2.040(3)	O(3)–Ni(1)–N(1)	97.76(11)
P(1)–O(1)	1.549(3)	N(2)–Ni(1)–N(2)$^{\#2}$	94.61(16)
P(1)–O(2)	1.555(3)	N(1)$^{\#2}$–Ni(1)–N(2)$^{\#2}$	78.04(11)
P(1)–O(3)	1.475(3)	O(3)$^{\#2}$–Ni(1)–O(3)	93.67(17)
P(2)–O(4)	1.516(2)	O(3)$^{\#2}$–Ni(1)–N(1)	88.40(11)
P(2)–O(5)	1.498(3)	N(1)$^{\#2}$–Ni(1)–N(2)	95.79(11)
P(2)–O(6)	1.556(4)		

Symmetry transformations used to generate equivalent atoms: #1: −x, y, −z + 3/2, #2: −x, y, −z + 1/2.

(in its neutral form) adopts a *cis* conformation by coordinating with the transition metal ion and *trans* conformation in its anionic form (uncoordinated) as shown in Scheme 1. By introducing a secondary ligand component (2,2′-bipyridine in the present studies) blocks four coordination sites, i.e. three equatorial and one apical positions of metal octahedron, leaving one equatorial and one apical sites available for coordination with LH_4. In order to meet this coordination requirement (i.e. to satisfy the coordination sites: one equatorial and one apical sites) a flexible non-rigid ligand (*p*-xylylenediphosphonic acid in the present case) has to adopt a rare *cis* conformation. This conformational freedom of LH_4 allows the existence of both *cis* and *trans* conformations in the same crystal structure, which may not be observed/possible for the similar rigid system like $((HO)_2O{=}P{-}C_6H_4{-}P{=}O(OH)_2$. The existence of *p*-xylylenediphosphonic acid in its *cis* conformation in the present study is first paradigm of its kind; the literatures related to crystal structures of *p*-xylylenediphosphonic acid-associated compounds [15–18] do not say anything about its *cis* conformation. The arrangement of coordinated neutral LH_4 in its *cis* conformation and uncoordinated anionic LH_2^{2-} in its *trans* conformation in the packing diagram is shown in Fig. 2. It is worth mentioning that in the crystal structure, both the neutral and anionic phosphonate species contain crystallographically-imposed symmetry.



Scheme 2.

As shown in Scheme 2, the linker (*p*-xylylenediphosphonic acid) is attached to the two different metal centers on its both ends *via* its terminal oxygen atoms (P═O) and thereby extends the structure in one-dimensional running parallel to the crystallographic *c* axis. The structure of compound **1** has not been shown here, be-

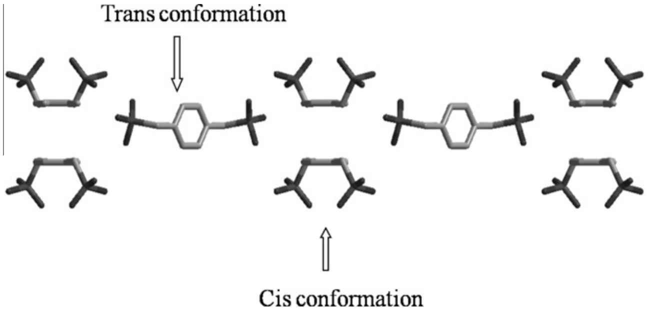


Fig. 2. Arrangement of *cis* and *trans* conformations of *p*-xylylenediphosphonic acid in the packing diagram in the crystal structure of compound **2**.

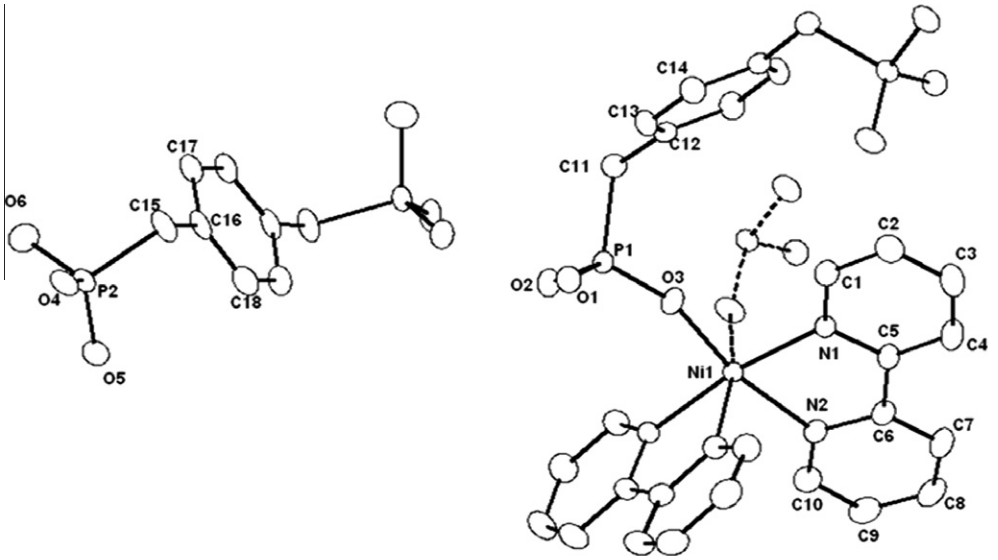


Fig. 1. Thermal ellipsoidal plot of the molecular structure of compound [Ni^{II}(2,2′-bipy)$_2$$LH_4$]$_n$[$LH_2$]$_n$ (**2**); hydrogen atoms are not shown for clarity (30% probability). $LH_4 = (HO)_2O{=}PCH_2C_6H_4CH_2P{=}O(OH)_2$, *cis* conformation; $LH_2^{2-} = (HO)(O^-)O{=}PCH_2C_6H_4CH_2P{=}O(O^-)(OH)$, *trans* conformation.

Table 3
Phosphorus–oxygen bond lengths in compound **2**.

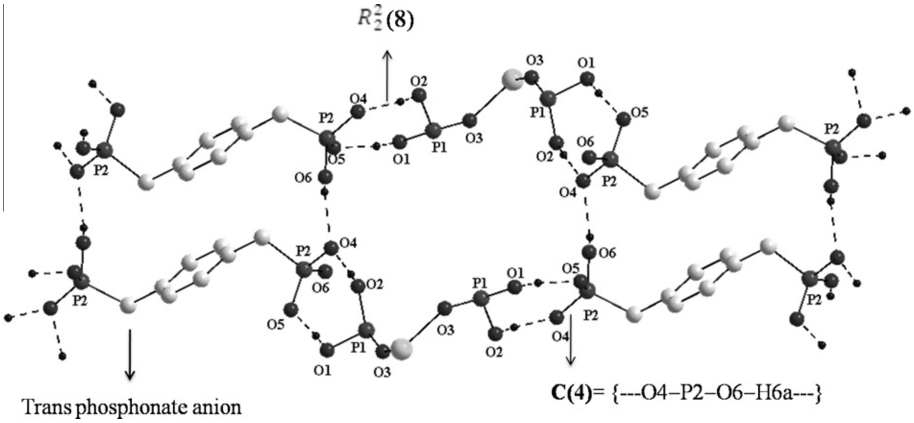
Coordinated *cis* neutral phosphonic acid LH_4 (in Å)	Uncoordinated *trans* ionic phosphonic acid LH_2^{2-} (in Å)
P1–O1 (P–OH)	P2–O4 (P–O)
1.549(3)	1.516(2)
P1–O2 (P–OH)	P2–O5 (P═O)
1.555(3)	1.498(3)
P1–O3 (P═O)	P2–O6 (P–OH)
1.475(3)	1.556(4)

cause both compounds **1** and **2** are isomorphous and crystallize in *C2/c* space group.

In both the complexes **1** and **2**, the metals are present in +2 oxidation state. As mentioned above, the phosphonic acid is coordinated to the metal atoms with P═O group on both sides which is in good accordance with the P–O bond length of 1.476 Å (Table 3) and thereby the charge of overall complex is +2, which is counter-balanced by the deprotonated phosphonic acid (LH_2). So the resulting ion paired compounds can be termed as clathrated complexes [24]. The fact, that the coordinated *cis* *p*-xylylenediphosphonic acid is neutral and the uncoordinated *trans* *p*-xylylenediphosphonic acid is ionic, is supported by the observed phosphorous–oxygen bond lengths present in compound **2** as described in Table 3.

From Table 3, it is clearly evident that neutral phosphonic acid is coordinated to metal and the charge is balanced by the deprotonated phosphonic acid which is uncoordinated. The assignment of P–O bonds according to the bond lengths (Table 3) are in good accordance with the relevant reported values [25]. Use of secondary ligand component, 2,2′-bipyridine blocks the four coordination sites of the metal center, thereby preventing the spatial expansion into 3D as reported for *p*-xylylenediphosphonic acid-associated compound in the literature [15]. Thus the careful selection of the secondary ligand components allows us to direct the dimensionality of the coordination polymers.

In the crystal structure, there are four protons, located on P–O bonds, resulting in four P–OH groups (two in the coordinated *cis* LH_4 and two in uncoordinated *trans* LH_2) and thereby an extended hydrogen bonding situation is expected. The oxygen atoms (O1 and O2) of the metal coordinated *cis* phosphonic acid are strongly hydrogen bonded to oxygen atoms of uncoordinated *trans* phosphonic acid (O4 and O5) with in a distance of 2.49 and 2.52 Å, respectively. The values are in good accordance for strong hydrogen bonding. The oxygen atom (O6) of the *trans* phosphonic acid is strongly hydrogen bonded to another *trans* phosphonic acid oxygen atom O4 with in a distance of 2.57 Å and so on resulting in a one-dimensional chain like arrangement that runs parallel to the crystallographic *c* axis with notation C(4) {—O4–P2–O6–H6a—}. Each metal center of a coordination polymer (for example chain



Scheme 3. Hydrogen bonding situation involving both *cis* and *trans* *p*-xylylenediphosphonic acid and *p*-xylylenediphosphonate anion, respectively: 2,2′-bipyridine and *cis* *p*-xylylene moiety are not shown for clarity, dotted lines indicate hydrogen bonding, $R_2^2(8)$, C(4) are the graph set notations for the hydrogen bondings.

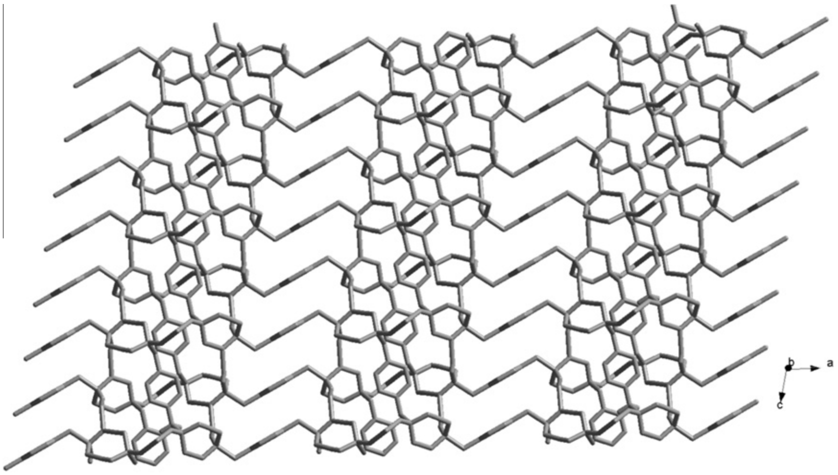


Fig. 3. The molecular packing diagram of $[Ni^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$ (**2**), characterized by hydrogen bonding when viewed down to the crystallographic *b* axis; 2,2′-bipyridine moieties are removed for clarity.

Table 4
Hydrogen bonding table for compounds **1** and **2**.

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
*$[Co^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$ (**1**)*				
O(1)–H(1A)···O(5)^{i}	0.96(4)	1.52(5)	2.482(2)	176(4)
O(2)–H(2A)···O(4)^{i}	0.79(3)	1.75(3)	2.535(3)	174(3)
O(6)–H(6A)···O(4)^{ii}	0.80(4)	1.78(4)	2.580(3)	171(4)
*$[Ni^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$ (**2**)*				
O(1)–H(1A)···O(5)^{iii}	0.94(7)	1.57(7)	2.494(4)	169(7)
O(2)–H(2A)···O(4)^{iii}	0.80(6)	1.73(6)	2.528(4)	173(6)
O(6)–H(6A)···O(4)^{iv}	0.82	1.80	2.572(4)	155.2

Symmetry transformations used to generate equivalent atoms: (i) $-x+1$, y, $-z+1.5$; (ii) x, $-y+1$, $z+0.5$; (iii) $-x+1$, y, $-z+0.5$; and (iv) x, $-y+1$, $z+0.5$.

A) is hydrogen bonded to its two adjacent *trans* phosphonate anions (LH_2) from opposite sides involving two hydrogen bonds (O(1)–H···O(4) and O(2)–H···O(5)) to form a rings of notation $R_2^2(8)$. Each of these LH_2 phosphonate anions, in turn, is hydrogen bonded to another *trans* LH_2 phosphonate anion by O(6)–H···O(4) hydrogen bond[C(4)] (Scheme 3). Each of these second generation LH_2 is again hydrogen bonded to a second coordination polymer (for example chain B) by using O(1)–H···O(4) and O(2)–H···O(5) hydrogen bonds to form a rings with notation $R_2^2(8)$. These chains A and B constitute a cylinder-like arrangement with the assistance of four *trans* LH_2 components Scheme 3. These cylindrical arrangements are, consecutively, linked with another set of cylindrical arrangements as the linker is ($-CH_2-C_6H_4-CH_2-$) as shown in Fig. 3. The relevant hydrogen bonding distances and angles with symmetry operations of both the compounds are listed in Table 4.

4. Spectroscopy

The IR spectra of compounds **1** and **2** exhibit characteristic bands centered at 2627 and 2361 cm^{-1}, that are related to O–H vibrations of {P–OH} moiety and are always present in the IR spectra of any phosphonate-compounds that contain a {P–OH} group [26]. The relatively weak bands centered at 3097 cm^{-1} are due to aromatic {CH} moieties. The bands at 1649 and 1599 cm^{-1} are of typical C=C stretching mode of the benzene ring. The strong and intense bands at 1141, 1093 and 978 cm^{-1} are due to the P–O stretching vibrations [24].

Compounds **1** and **2** are additionally characterized by solid state electronic absorption spectroscopy. The relevant diffuse reflectance spectra are shown in Fig. 4. Both compounds have almost identical electronic absorption spectral features except there is a well defined d–d band for compound **1**. The room temperature magnetic moment data of compound **1** is consistent with a high spin Co(II) complex with three unpaired electrons.

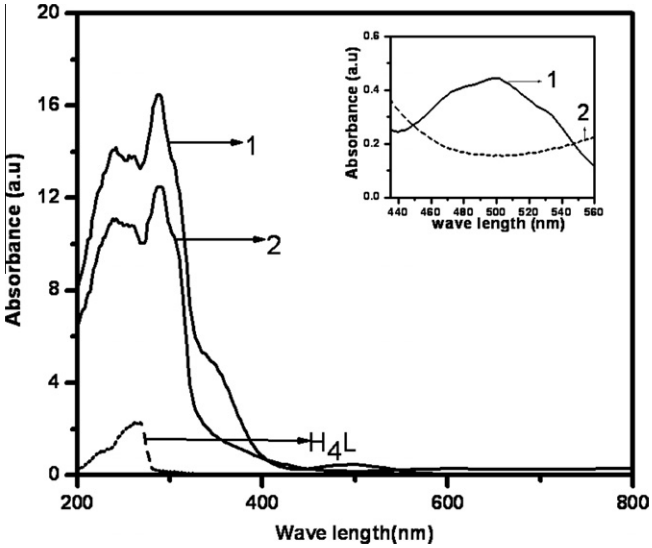


Fig. 4. Diffuse reflectance electronic spectra for compounds **1** and **2**.

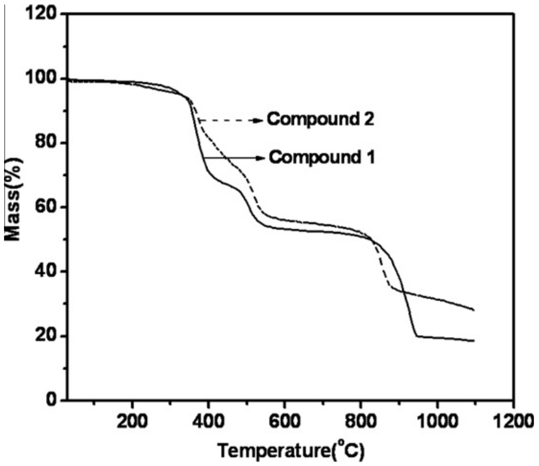


Fig. 5. Thermogravimetric curves for compounds **1** and **2**.

5. Thermal properties

The TGA curve of compound $[Ni^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$ (**2**) exhibits a thermal stability up to 320 °C. There are several weight loses in the region from 320 °C to 900 °C which are attributed to condensation of the hydroxyl groups in both the phosphonate ligands and combustion of bipyridine and organic ligands with the observed weight loss of 60% (theoretical values of 61.3%). The final end product is supposed to be $Ni(PO_3)_2$ with a formula weight of 216.0(23%) but the observed weight remained is 333.0(37%); this increase in weight may be due to the retention of some P_2O_5 at this temperature as shown by the continuous weight loss from 900 °C to 1100 °C. Compound $[Co^{II}(2,2'\text{-bipy})_2LH_4]_n[LH_2]_n$ (**1**) also exhibits a comparable TG curve that shows thermal stability up to 315 °C. The TGA curve shows similar weight loses as shown in the TG curve of compound **2**. The final end product is supposed to be $Co(PO_3)_2$ with a formula weight of 216.0(23%) but the observed weight remained is 180.2(20%); this may be due to some loss of P_2O_5. Both TG curves are shown in Fig. 5.

6. Conclusion

In conclusion, for the first time, we report two new isomorphous one-dimensional metal phosphonates (M = Co, Ni) in which *p*-xylylenediphosphonic acid adopts both *cis* and *trans* conformation and 2,2′-bipyridine as secondary ligand component. The use of secondary ligand component prevents the spatial expansion into 3D by blocking the coordination sites of the metal center. The conformational freedom of the flexible non-rigid *p*-xylylenediphosphonic acid allows tending to be in *cis* conformation to meet the coordination geometry of the metal center, as some of the coordination sites are already blocked by the secondary ligand components. This is the rare example in which *p*-xylylenediphosphonic acid adopts *cis* conformation during its coordination. This system proves to be a good example to investigate self assembly process and provides more information of the directional synthesis of the targeted coordination polymers. Multiple hydrogen bonding interactions between the 1D polymeric chains result in the formation of 2D network consisting of cylindrical channels.

Acknowledgements

We thank Department of Science and Technology, Government of India, for financial support (Project No. SR/SI/IC-23/2007). The National X-ray Diffractometer facility at University of Hyderabad by the Department of Science and Technology, Government of India, is gratefully acknowledged. We are grateful to UGC, New Delhi, for providing infrastructure facility at University of Hyderabad under UPE grant. B.K.T. and R.K. thank UGC, CSIR, New Delhi, for their fellowships.

Appendix A. Supplementary data

CCDC 748112 and 748113 contain the supplementary crystallographic data for compounds **1** and **2**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.poly.2010.08.008.

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