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$[Co(en)_3][In_3(H_2PO_4)_6(HPO_4)_3]\cdot H_2O$: A new layered indium phosphate templated by cobalt complex

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Abstract

A new layered indium phosphate $[Co(en)_3][In_3(H_2PO_4)_6(HPO_4)_3]\cdot H_2O$ (**1**) has been synthesized solvothermally by using a racemic mix of chiral metal complex $Co(en)_3Cl_3$ as a template. Its structure is determined by single-crystal X-ray diffraction analysis and further characterized by X-ray powder diffraction, ICP, NMR and TG analyses. The inorganic layer is built up by alternation of In-centred octahedra (InO_6) and P-centered tetrahedra ($PO_3(OH)$, $PO_2(OH)_2$, $PO_2(=O)(OH)$ and $PO(=O)(OH)_2$) forming a 4.12-net. The metal complex cations locate in the interlayer region and interact with the host network through H-bonds. It is the first indium phosphate compound templated by a transition-metal complex and is isostructural with GaPO–CJ14. Crystal data: **1**, monoclinic, space group $P2_1/m$ (No. 11), $a = 9.1700(18)$ Å, $b = 22.6923(5)$ Å, $c = 9.9116(2)$ Å, $\beta = 107.87(3)°$, $Z = 4$, $R_{1[I>2\sigma(I)]} = 0.0287$ and $wR_{2(\text{all data})} = 0.0939$.

Keywords: Layer; Indium phosphate; Synthesis; Structure; Metal complex

1. Introduction

Over the past few decades, employing various alkali metal ions or organic amines as a structure-directing agent, a large number of open-framework metal phosphates have been synthesized under hydrothermal/solvothermal conditions, because of their potential applications in catalysis, adsorption and separation, as well as in advanced materials. These open-framework metal phosphates include aluminophosphates [1–4], gallium phosphates [5–8], indium phosphates [9–12], zinc phosphates [13–17], cobalt phosphates [18,19], titanium phosphates [20,21], and iron phosphates [22,23], etc.

Among these open framework metal phosphates, there are a small number of metal phosphates that are templated by transition metal complexes. Such compounds are known as a few aluminophosphates [24–29], gallium phosphates $[d\text{-}Co(en)_3][H_3Ga_2P_4O_{16}]$ [30], and $[Co(en)_3][Ga_3(H_2PO_4)_6(HPO_4)_3]$ [31], boron phosphate $[Co(en)_3][B_2P_3O_{11}(OH)_2]$ [32], a few zinc phosphates [33–35], and vanadium borophosphate $[Co(en)_3]_2[V_3P_3BO_{19}][H_2PO_4]\cdot 4H_2O$ [36]. They are templated by an optically pure or a racemic mix of metal complex, such as $Co(en)_3^{3+}$, $Co(tn)_3^{3+}$, $Co(dien)_2^{3+}$, $Ir(en)_3^{3+}$, Ir- or Co-(1, 2-diaminocyclohexane)$_3^{3+}$. Interestingly, the rigid octahedrally coordinated metal amine complex has a chiral feature. Our recent studies have shown that there exists a stereo-specific correspondence between the host network and the guess metal complexes and H-bonds are its origin [35]. This phenomenon has important implications concerning the imprinting of the chirality of the templating agents into the host framework.

Compared to the large number of aluminophosphates and gallium phosphates there are a few reports on the indium phosphates. The indium phosphates exhibit interesting framework structures, such as 3-D open framework $[en]_3[In_5(PO_4)_4F_3]\cdot 2H_2O$ [9] containing 16-membered ring, $[NH_3(CH_2)_2NH_2(CH_2)_2NH_3][NH_2(CH_2)_2NH_2(CH_2)_2NH_2][In_{6.8}(H_2O)_2(PO_4)_4(HPO_4)_4F_8]\cdot 2H_2O$ [10] containing 16-membered ring channels. 2-D layers $(Hen)[In(PO_4)F]$ [9],

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$[In_4(4\text{-}4'bipy)_3(HPO_4)_4] \cdot 4H_2O$ [11] and $[C_5H_5NH][In(HPO_4)(H_2PO_4)_2]$ [12].

In this work, employing a racemic mix of chiral metal complex $Co(en)_3Cl_3$ as a template, a new layered indium phosphate $[Co(en)_3][In_3(H_2PO_4)_6(HPO_4)_3] \cdot H_2O$ (**1**) has been synthesized. It is the first indium phosphate templated by the metal complex and is isostructural with $[Co(en)_3][Ga_3(H_2PO_4)_6(HPO_4)_3]$ (GaPO-CJ14) [31].

2. Experimental section

Compound **1** was prepared by a solvothermal reaction of a mixture of $In(OH)_3$, H_3PO_4, $Co(en)_3Cl_3$ and 2-BuOH. In a typical synthesis, 0.169 g indium hydroxide (99.9 wt%) was first dissolved in 10 mL of 2-butanol, and then 0.272 mL of phosphoric acid (85 wt% in water) was added with stirring. Finally, 0.190 g $Co(en)_3Cl_3$ was added to the above reaction mixture. A gel was formed and stirred for 1 h until it was homogeneous, and then was sealed in a Teflon-lined stainless steel autoclave and heated at 130°C for 6 d under static conditions. The orange plate-shaped single crystals were separated by sonication and further washed by distilled water and then air-dried.

X-ray powder diffraction (XRD) data were collected on a Siemens D5005 diffractometer with Cu$K\alpha$ radiation ($\lambda = 1.5418$ Å).

Inductively coupled plasma (ICP) analysis was performed on a Perkin–Elmer Optima 3300DV spectrometer. A Perkin–Elmer TGA 7 unit was used to carry out the thermogravimetric analysis (TGA) in air with a heating rate of 10°C/min.

The magic-angle-spinning (MAS) NMR spectra were recorded on a Varian Infinity Plus-400 spectrometer. The ^{31}P NMR spectrum was taken at 161.9 MHz with a spinning rate of 11 kHz. Chemical shifts were referenced to 85% H_3PO_4. The recycle delay time was 60 s.

A suitable single crystal with dimensions $0.2 \times 0.08 \times 0.08$ mm^3 was selected for single-crystal X-ray diffraction analysis. The intensity data were collected on a Rigaku R-AXIS RAPID IP diffractomer by oscillation scans using graphite-monochromated Mo$K\alpha$ radiation ($\lambda = 0.71073$ Å). Cell refinement and data reduction were accomplished with the RAPID AUTO program. The structure was solved in the space group $P2_1/m$ by the direct methods and refined on F^2 by full-matrix least squares using SHELXTL97 [37]. No adsorption correction was applied. The heaviest atoms In, Co and P were easily located. O, C and N atoms were subsequently located in the difference Fourier maps and H atoms attached to the terminal P–O groups and to the metal complex cations were placed geometrically and refined using a riding model. H atoms associated with water molecules are not added. It was noted that part of the C and N atoms of the $Co(en)_3^{3+}$ cation were positionally disordered. The low–temperature X-ray study had been tried at 80 K before, but there is no improvement for the disorder of C and N atoms in the structure. All non-hydrogen atoms were refined anisotropically. Experimental details for the structure determination are presented in Table 1. The selected bond distances and bond angles are presented in Table 2. CCDC 231898 contains the supplementary crystallogtaphic data for this paper.

Table 1
Crystal data and structure refinement for compound **1**

Empirical formula	$C_6H_{41}Co_1In_3N_6O_{37}P_9$
Formula weight	1470.39
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, $P2_1/m$
Unit cell dimensions	$a = 9.1700(18)$ Å $\alpha = 90°$ $b = 22.692(5)$ Å $\beta = 107.87(3)°$ $c = 9.912(2)$ Å $\gamma = 90°$
Volume	1963.0(7) Å^3
Z, Calculated density	2, 2.934 Mg/m^3
Absorption coefficient	7.213 mm^{-1}
$F(000)$	1582
Crystal size	$0.2 \times 0.08 \times 0.08$ mm^3
Theta range for data collection	1.79–27.48°
Limiting indices	$0 \leqslant h \leqslant 11$, $0 \leqslant k \leqslant 29$, $-12 \leqslant l \leqslant 12$
Reflections collected/unique	4300/4300 [R(int) = 0.0000]
Completeness to $\theta = 27.48$	93.3%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4300/0/319
Goodness-of-fit on F^2	0.885
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0285$, w$R_2 = 0.0667$
R indices (all data)	$R_1 = 0.0653$, w$R_2 = 0.0932$
Largest diff. peak and hole	1.125 and -0.668 e Å^{-3}

3. Results and discussion

The orange single crystals of **1** can be crystallized from reaction gel with molar composition 1.0 $In(OH)_3$: 4.0 H_3PO_4: 0.5 $Co(en)_3Cl_3$: 66 2-BuOH at 130°C. Using other solvents such as water, ethylene glycol instead of 2-butanol, this compound cannot be obtained under above experimental conditions.

Fig. 1 shows the experimental and simulated X-ray powder diffraction patterns of $[Co(en)_3][In_3(H_2PO_4)_6(HPO_4)_3] \cdot H_2O$, which are in agreement with each other, suggesting the phase purity of the as-synthesized product.

Inductively coupled plasma (ICP) analysis for **1** gives the contents of In as 23.31 wt% (calcd 23.42 wt%), P as 18.47 wt% (calcd 18.96 wt%), and Co as 3.92 wt% (calcd 4.01 wt%). Elemental analysis indicates that the sample contains 5.82, 2.68 and 5.61 wt% of C, H and N, respectively. These are in agreement with those calculated values of 5.90, 2.79 and 5.71 wt% of C, H, and N, respectively, on the basis of the empirical formula

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

In(1)–O(6)	2.103(4)	In(1)–O(12)	2.105(5)
In(1)–O(11)	2.121(4)	In(1)–O(8)	2.144(4)
In(1)–O(4)	2.154(5)	In(1)–O(7)	2.163(4)
In(2)–O(18)	2.088(4)	In(2)–O(18)#	22.088(4)
In(2)–O(19)	2.142(4)	In(2)–O(19)#	22.142(4)
In(2)–O(15)	2.152(5)	In(2)–O(15)#	22.152(5)
P(1)–O(6)	1.504(4)	P(1)–O(7)#	31.524(4)
P(1)–O(19)#	41.526(4)	P(1)–O(5)	1.585(5)
P(2)–O(2)	1.491(5)	P(2)–O(4)	1.499(4)
P(2)–O(1)	1.570(5)	P(2)–O(3)	1.573(5)
P(3)–O(10)	1.507(7)	P(3)–O(8)	1.510(4)
P(3)–O(8)#5	1.510(4)	P(3)–O(9)	1.576(7)
P(4)–O(11)	1.499(4)	P(4)–O(15)	1.502(5)
P(4)–O(13)	1.568(5)	P(4)–O(14)	1.578(5)
P(5)–O(12)	1.498(4)	P(5)–O(18)#	21.507(4)
P(5)–O(16)	1.562(5)	P(5)–O(17)	1.565(5)
P(2)–O(4)–In(1)	131.3(3)	P(1)–O(6)–In(1)	147.4(3)
P(1)#3–O(7)–In(1)	142.6(3)	P(3)–O(8)–In(1)	131.2(2)
P(4)–O(11)–In(1)	134.2(3)	P(5)–O(12)–In(1)	136.3(3)
P(4)–O(15)–In(2)	137.0(3)	P(5)#2–O(18)–In(2)	139.4(3)
P(1)#6–O(19)–In(2)	133.4(3)		

H-Bonds

D–H···A	*d*(*D*···A)	<(DHA)
O(1)–H(1)···O(19)#4	2.631(7)	151.6
O(5)–H(5)···N(2)#3	2.924(12)	169.2
O(9)–H(9)···O(11)	3.025(5)	146.0
O(14)–H(14)···O(1W)	2.176(6)	142.3
O(1)–H(9)···O(1W)	2.250(4)	105.9

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

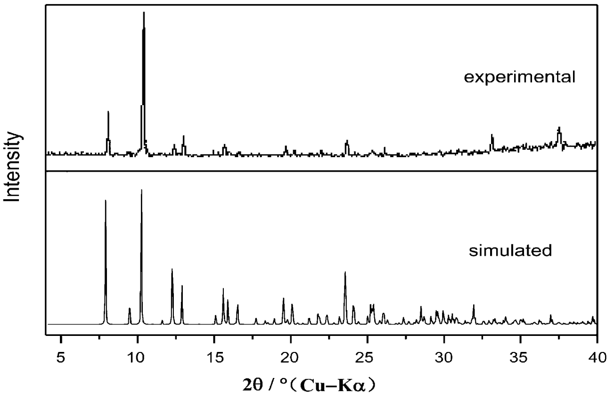

Fig. 1. The experimental and simulated X-ray power diffraction patterns for **1**.

$[Co(en)_3][In_3(H_2PO_4)_6(HPO_4)_3]\cdot H_2O$ given by single-crystal structure analysis.

Thermogravimetric analysis (Fig. 2) shows a weight loss of 1.28 wt% occurring at ca. 210°C, which is attributed to the loss of H_2O (calcd 1.22 wt%) in the product, The weight loss of 12.19 wt% around 300°C, is attributed to the decomposition of the metal complexes (calcd 12.24 wt%). The as-made product changes to amorphous phase after heated at 500°C and further transforms to dense phase $CoIn_3P_4O_{16}$ after heated at 800°C.

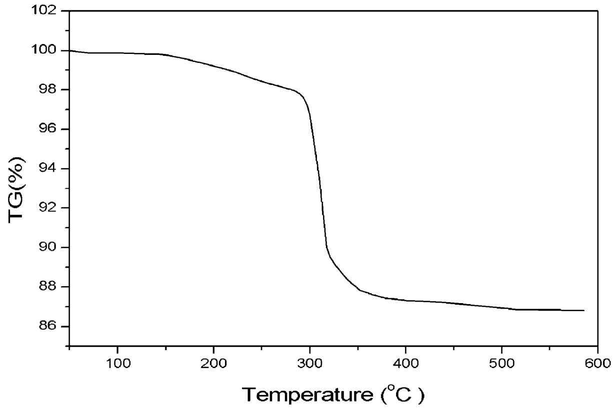

Fig. 2. Thermogravimetric curve of **1**.

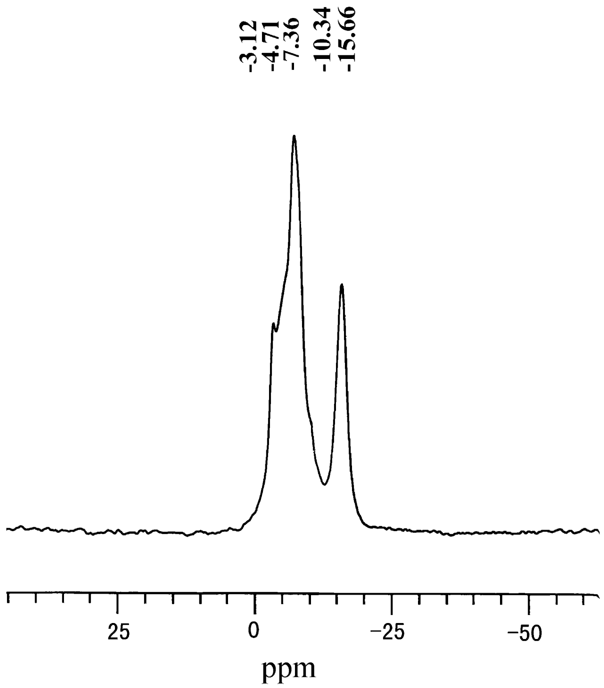

Fig. 3. The ^{31}P NMR spectrum of **1**.

As seen in Fig. 3, the ^{31}P NMR spectrum shows three sharp resonance lines at 3.33, 7.16 and 15.79 ppm, respectively. The signal at 7.16 ppm with two shoulders at 6.93 and 10.42 ppm, respectively indicates that it is the superposition of three lines with close chemical shifts. The five ^{31}P signals are in agreement with the five crystallographically distinct P sites in the structure and are characteristic for the tetrahedral phosphate sites.

The structure of **1** consists of macroanionic $[In_3(H_2PO_4)_6(HPO_4)_3]^{3-}$ sheets. The charge neutrality is achieved by the $Co(en)_3^{3+}$ cations located in the interlayer region. The asymmetric unit, as seen in Fig. 4,

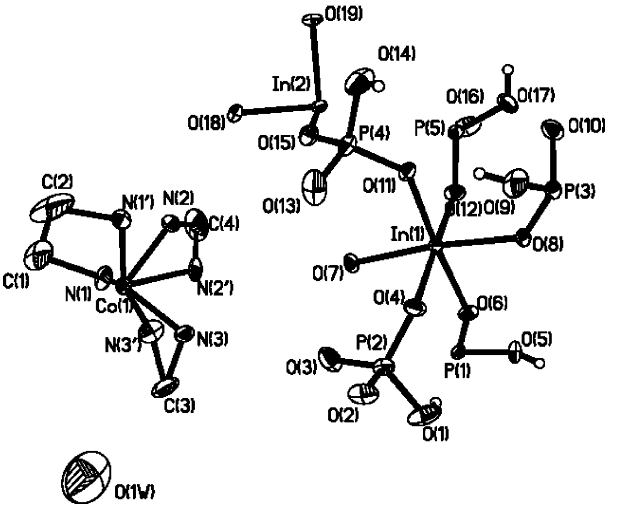

Fig. 4. Thermal ellipsoid plot (50%) showing atom in **1**.

contains two crystallographically distinct In atoms. Each In atom is coordinated to six oxygen atoms. In(1) atom lies on a general position. The In(1)O_6 unit is slightly distorted with the In–O distances in the range of 2.088(4)–2.153(5) Å. The O–In–O bond angle for two oxygens at the *trans* positions are in the range of 173.03(17)–177.82(16)°. In(2) atom lies on an inversion center, and the In(2)O_6 unit has a regular octahedral geometry with the In–O bond lengths in the range of 2.103(4)–2.163(4) Å. The O–In–O angles for two oxygens at the *trans* positions is 180°. Of the five distinct tetrahedrally coordinated P atoms, P(3) lies on a mirror plane, while other P atoms are on a general position. P(1) makes three P–O–In links and has one terminal P–OH bond. P(2) makes only one P–O–In link and possesses two terminal P–OH bonds and one terminal P$=$O bond (1.491(5) Å). P(3) makes two P–O–In links, has one terminal P–OH bond and one terminal P$=$O bond (1.508(7) Å). P(4) and P(5) each makes two P–O–In links and possesses two terminal P–OH bonds. The P–O_{bridging} distances are in the ranges of 1.499(5)–1.526(5) Å, while the longer P–OH bonds are in the range of 1.562 (5)–1.585(5) Å.

Each asymmetric unit also contains one crystallographically distinct Co atom lying on a mirror plane. $\mathrm{Co(en)}_3^{3+}$ is either the delta or the rambda enantiomer of a chiral identity, As a consequence, the two isomers randomly occupy the lattice positions. The disorder of the C and N atoms in the complex is similar to those in $[\mathrm{Co(en)_3}][\mathrm{Ga_3(H_2PO_4)_6(HPO_4)_3}]$ (GaPO-CJ14) [31] and $\mathrm{Co(en)_3[AlP_2O_8]} \cdot x\mathrm{H_2O}$ [29].

The strict alternation of In-centred octahedra ($\mathrm{InO_6}$) and P-centred tetrahedra ($\mathrm{PO_3(OH)}$, $\mathrm{PO_2(OH)_2}$, $\mathrm{PO_2(=O)(OH)}$ and $\mathrm{PO(=O)(OH)_2}$) forms a 2-D sheet parallel to the *bc* plane containing 4- and 12-membered rings (Fig. 5a). The 4.12-net sheet is a new type of layered structure in indium phosphate family. It is helpful to view this framwork as being built up from a chain composed of In-centered octahedra and P-centered tetrahedral, as shown in Fig. 5b. A series of such chains interconnected with each other via P(3) tetrahedra forms the 2D layered structure. As with GaPO–CJ14, there exist two centrosymmetrically related [3.3.3] propellane-like chiral motifs sharing a common In(1) atom at the inversion center (Fig. 5c).

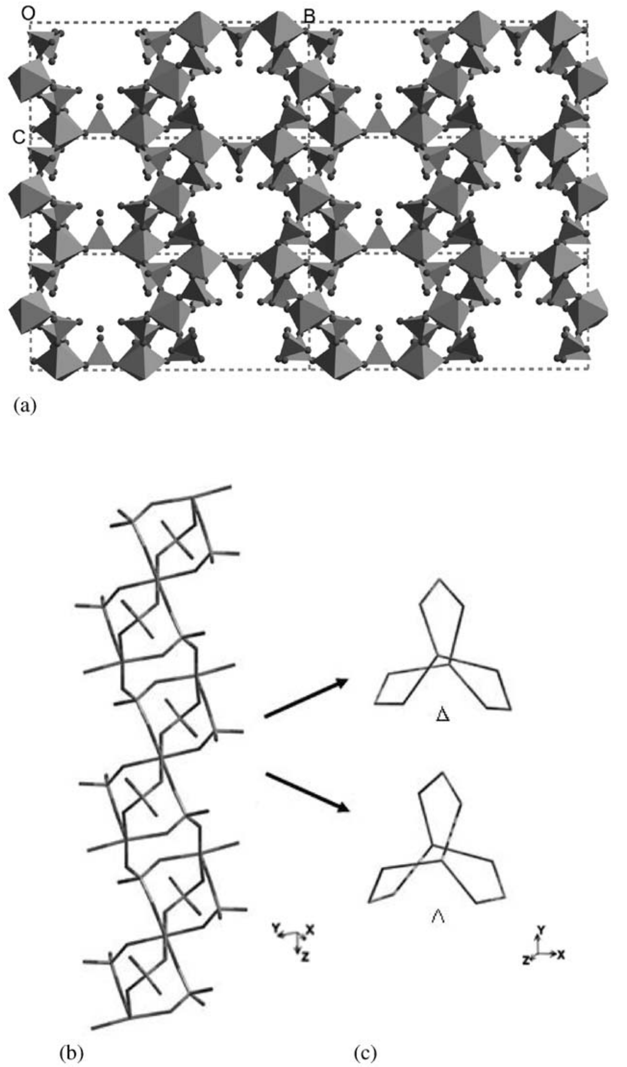

Fig. 5. (a) The 4.12-net sheet structure parallel to the *bc* plane in **1**; (b) a chain made up of solely four-membered rings along the *c*-axis; (c) two centrosymmetrically related [3.3.3] propellane-like chiral motifs existing in Δ and Λ configurations, respectively.

As seen in Fig. 6, the inorganic sheets stack in an AAAA sequence along the [100] direction, with the metal complex cations and H_2O residing in the interlayer region. The complex cations interact with the terminal OH groups in the inorganic network through H-bonds with the N⋯O separation of 2.926(12) Å. There are H-bonds between the bridging

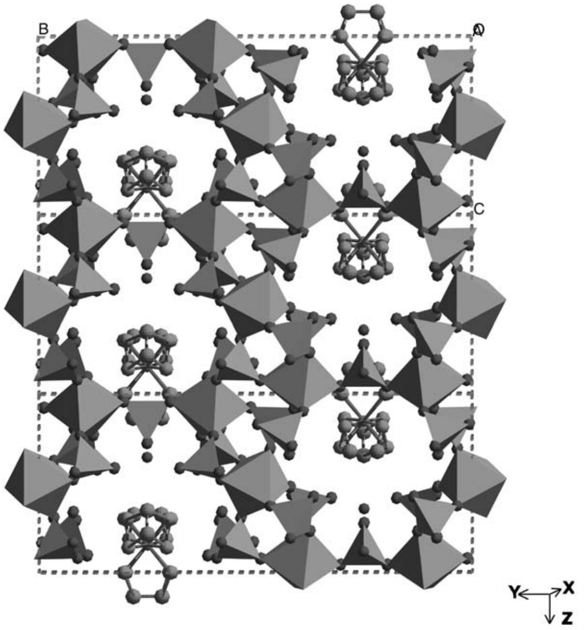

Fig. 6. Packing of the inorganic sheets in an AAAA sequence along the [100] direction.

oxygens and the terminal OH groups inside the inorganic network with the O⋯O separation of 2.629(7)–3.025(5) Å. Table 2 summarizes the H-bond information. There also exist H-bonds between the H_2O molecules and the OH groups, template. The H-bond distances are in the range of 2.539–2.715 Å as measured by using Cerius2 software package [38].

Very interestingly, it is found that [3.3.3] propellane-like chiral motif has been found in several cobalt complex-templated metal phosphates including layered aluminophosphates $[Co(en)_3][Al_3P_4O_{16}] \cdot 3H_2O$ [24] and *trans*-$Co(dien)_2 \cdot Al_3P_4O_{16} \cdot 3H_2O$ [26], and layered zinc phosphate $[Co(en)_3]_2 \cdot [Zn_6P_8O_{32}H_8]$ [33], and layered gallium phosphate $[Co(en)_3][Ga_3(H_2PO_4)_6(HPO_4)_3]$ [32]. Recently, two new gallium phosphates $[Co(en)_3][Ga_3P_4O_{16}] \cdot 3H_2O$ and *trans*-$[Co(dien)_2][Ga_3P_4O_{16}] \cdot 3H_2O$ whose structures are isostructural to the layered aluminophosphates $[Co(en)_3][Al_3P_4O_{16}] \cdot 3H_2O$ and *trans*-$Co(dien)_2 \cdot Al_3P_4O_{16} \cdot 3H_2O$, respectively, are also featured by such a chiral structural motif. Structurally, it appears that the symmetry and configuration of the [3.3.3]propellane-like structural motif is in a good match with that of the octaherally coordinated chiral cobalt complex. This further demonstrates that there exists a stereo-specific correspondence between the host network and the guest metal complexes.

4. Conclusions

A new 2-D layered indium phosphate $[Co(en)_3][In_3(H_2PO_4)_6(HPO_4)_3] \cdot H_2O$ has been prepared solvothermally. It is the first indium phosphate compound templated by a transition-metal complex and is isostructural with GaPO–CJ14. The structure is characteristic of a structural unit composed of a pair of enantiomers of chiral [3.3.3] propellane-like structural motifs. The guest metal complexes and H_2O located in the interlayer regions interact with the host network through H-bonds. The chiral [3.3.3] propellane-like structural motif has been commonly encountered in cobalt complex-templated metal phosphates. Structurally, it is in a good match with that of the octaherally coordinated chiral cobalt complex, indicating that there exists a stereo-specific correspondence between the host network and the guess metal complexes.

Acknowledgments

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