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Crystal structures of five transition-metal 1,4-butylenediphosphonates

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Abstract

The structures of five transition-metal 1,4-butylenediphosphonates: $[\{Co(H_2O)_6\}\ \{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]$ (**1**), $[\{Ni(H_2O)_6\}\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]$ (**2**), $[\{Cu(H_2O)_2\}\{HO_3P(CH_2)_4PO_3H\}]_n$ (**3**), $[Zn\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]_n$ (**4**) and $[\{Cu_2(H_2O)_2\}\{O_3P(CH_2)_4PO_3\}\cdot H_2O]_n$ (**5**) have been determined by single crystal X-ray diffraction. The structures are diverse, including zero-dimensional, chain-like and three-dimensional framework types.

Keywords: Crystal structures; Transition-metal; 1,4-Butylenediphosphonates

1. Introduction

From 1970s onward much research has been concentrated on metal organophosphonates due to their potential or practical applications in ion exchange [1], diagnostic agents [2,3] and optics [4–7]. Particularly, numerous porous materials have been produced using organodiphosphonate ligands ($-O_3PRPO_3-$, R = organic units) with variable lengths and shapes of the organic units [8–33]. Recently, Clearfield et al. [34–37] used nitrilotri(methylphosphonic), *N*-(phosphonomethyl)imniodiacetic and *N*,*N*-bis(phosphonomethyl)aminoacetic acids to produce some novel compounds. Metal alkylenediphosphonate ($-O_3P(CH_2)_nPO_3-$, $n = 1-3$) have been extensively studied in previous work [9,11–16,18–21,26,27,29]. However, among metal 1,4-butylenediphosphonates only the structure of $Cu_2[(O_3PC_4H_8PO_3)(H_2O)_2]\cdot 2H_2O$ has been determined by X-ray powder diffraction [30]. Herein, we report single crystal structures of five transition-metal 1,4-butylenediphosphonates: zero-dimensional $[\{Co(H_2O)_6\}\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]$ (**1**) and $[\{Ni(H_2O)_6\}\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]$ (**2**), stepped chain $[\{Cu(H_2O)_2\}\{HO_3P(CH_2)_4PO_3H\}]_n$ (**3**) and three-dimensional frameworks $[Zn\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]_n$ (**4**) and $[\{Cu_2(H_2O)_2\}\{O_3P(CH_2)_4PO_3\}\cdot H_2O]_n$ (**5**).

2. Experimental

2.1. Materials and synthesis

1,4-Butylene-diphosphonic acid was prepared according to the reported method [38,39]. Other chemicals were reagent grade quality and obtained from commercial sources without further purification. The elements were analyzed with Vario EL III element analyzer and UV-1100 spectrophotometer. Infrared spectra were obtained on a Nicolet Magna 750 FT-IR spectrometer.

2.1.1. $[\{Co(H_2O)_6\}\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]$ (1)

0.2095 g (0.7453 mmol) $CoSO_4\cdot 7H_2O$ and 0.1036 g (0.4751 mmol) 1,4-butylenediphosphonic acid were dissolved in 10 ml distilled water. A small amount of precipitate appeared after the pH of the solution adjusted to 3.21 with 1.000 mol l^{-1} NaOH. Dimethylsulfoxide (2 ml) was added to the solution with vigorous stirring. Then the solution was filtered into a 25-ml beaker and left at ambient temperature. After 70 days, red block crystals were obtained in poor yield.

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2.1.2. $[\{Ni(H_2O)_6\}\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]$ (**2**)

0.1731 g (0.7283 mmol) $NiCl_2\cdot 6H_2O$ and 0.1041 g (0.4773 mmol) 1,4-butylenediphosphonic acid were dissolved in 10 ml distilled water. A small amount of green precipitate appeared after the pH of the solution was adjusted to 4.31 with 1.000 mol l^{-1} NaOH. The mixture was stirred homogeneously after 2 ml DMSO was added to the solution. Green cuboid crystals were obtained after the solution was filtered into a 25-ml beaker and left at ambient temperature for 40 days. The green crystals were filtered, dried at ambient temperature and yielded 36% based on 1,4-butylenediphosphonic acid. *Anal*. Calc. for **2**: C 11.47, H 6.26, Ni 14.01%; Found: C 11.64, H 5.45, Ni 13.59%. IR (KBr pellet, cm^{-1}): 3550vs, 2964m, 2376m, 2322w, 1672m, 1462m, 1414m, 1340m, 1304m, 1205s, 1182s, 1109s, 1090s, 1043s, 931s, 766m, 723m, 548s, 490m, 471m.

2.1.3. $[\{Cu(H_2O)_2\}\{HO_3P(CH_2)_4PO_3H\}]_n$ (**3**)

0.3418 g (2.005 mmol) $CuCl_2\cdot 2H_2O$ and 0.218 g (1.00 mmol) 1,4-butylenediphosphonic acid were dissolved in 10 ml distilled water. A small amount of blue precipitate appeared after the pH of the solution was adjusted to 1.75 with 1.000 mol l^{-1} NaOH. Blue flake-like crystals were obtained after the solution was filtered into a test tube, layered with 2-ml isopropyl alcohol and left at ambient temperature for 110 days. The blue crystals were filtered, washed in turn with distilled water, ethanol and aether, dried at ambient temperature and yielded 10% based on 1,4-butylenediphosphonic acid. *Anal*. Calc. for 3: C 15.22, H 4.470, Cu 20.1%; Found: C 15.28, H 3.735, Cu 18.6%. IR (KBr pellet, cm^{-1}): 3321m, 2960m, 2939m, 2902m, 2872m, 2370m, 2283m, 2139w, 1624m, 1448m, 1404m, 1325m, 1300m, 1196vs, 1101s, 1078s, 1011s, 949s, 779m, 731m, 557s, 494m, 444m, 405m.

Thermogravimetric analysis (TGA) was performed on 10.7778 mg of a polycrystalline sample of compound **3** from ambient temperature to 1100 °C. The TGA data show that three obvious weight loss stages: (1) 100–164 °C, weight loss 11.3% possibly corresponding to removing two moles of coordinating water molecules per formula unit (Calc. 11.4%). (2) 200–536 °C, weight loss 21.7%. (3) 679–1000 °C, weight loss 27.0%.

2.1.4. $[Zn\{HO_3P(CH_2)_4PO_3H\}\cdot 2H_2O]_n$ (**4**)

1.1717 g (4.0749 mmol) $ZnSO_4\cdot 7H_2O$ was dissolved in 20.0 ml of 0.1000 mol l^{-1} 1,4-butylenediphosphonic acid solution in a beaker. The pH of the solution was slowly adjusted to 2.00 with 1.000 mol l^{-1} NaOH. Then 0.100 g (1.67 mmol) urea was added to the solution and dissolved. Colorless block single crystals were obtained after the solution filtered into a 50-ml flask and kept at 55 °C for 24 h. The crystals were filtered, washed with distilled water, dried at ambient temperature and yielded 18% based on 1,4-butylenediphosphonic acid.

The crystals can also be obtained by the following procedure. 0.1016 g (0.7455 mmol) $ZnCl_2$, 0.0522 g (0.0239 mmol) 1,4-butylenediphosphonic acid and a small amount of 1 M HCl were mixed in 20.0 ml distilled water. Five-milliliter DMSO was added to the solution. After the solution was stirred homogeneously, a small amount of white precipitate appeared when the pH of the solution was adjusted to 2.84 with 1.000 mol l^{-1} NaOH. Colorless block-like crystals appeared after the solution was filtered into a 50-ml flask and kept in the ambient environment for 6 months. *Anal*. Calc. for **4**: C 15.13, H 4.445, Zn 20.59%; Found: C 15.25, H 4.513, Zn 21.68%. IR (KBr pellet, cm^{-1}): 3452s, 3373s, 3255m, 2966w, 2947w, 2914w, 2879w, 2380m, 1792m, 1647w, 1460m, 1410m, 1333m, 1275w, 1201s, 1178m, 1132vs, 1092s, 1053vs, 1009m, 951vs, 775s, 721m, 552s, 465m, 409m.

TGA was performed on 11.0211 mg of a polycrystalline sample of compound **4** from ambient temperature to 700 °C. The TGA data show that three obvious weight loss stages: (1) 70–137 °C, weight loss 11.0% possibly corresponding to removing two moles of free water molecules per formula unit (Calc. 11.3%). (2) 241–391 °C, weight loss 5.7%. (3) 500–600 °C, weight loss 11.7%.

2.1.5. $[\{Cu_2(H_2O)_2\}\{O_3P(CH_2)_4PO_3\}\cdot H_2O]_n$ (**5**)

0.3421 g (2.007 mmol) $CuCl_2\cdot 2H_2O$ and 0.2185 g (1.002 mmol) 1,4-butylenediphosphonic acid were dissolved in 10 ml distilled water. Then 5 ml DMSO was added to the solution. A small amount of blue precipitate appeared after the pH of the solution was adjusted to 2.62 with 1.000 mol l^{-1} NaOH. Green flake-like crystals were obtained after the solution was filtered into a 50-ml beaker and stood at ambient temperature for 40 days. The green crystals were filtered, washed with distilled water, dried at ambient temperature and yielded 6.9% based on 1,4-butylenediphosphonic acid. *Anal*. Calc. for **5**: C 12.16, H 3.571%; Found: C 12.11, H 3.363%.

2.2. X-ray crystallography

X-ray data on single crystals were collected at 293 ± 2 K on a Siemens SMART-CCD diffractometer using graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). Data were reduced and absorption corrected with SMART and SADABS software, respectively. The structures were solved by direct methods and refined by full-matrix least-squares techniques on F^2 using SHELXTL-97 [40]. All non-hydrogen atoms of these compounds were treated anisotropically. All hydrogen atoms of these compounds were from difference Fourier map and fixed isotropic thermal parameter.

2.2.1. Crystal data for *1*

$C_4H_{26}CoO_{14}P_2$, $M = 419.12$, triclinic, $P\bar{1}$, $a = 6.3931(7)$, $b = 11.0800(11)$, $c = 11.9285(12)$ Å, $\alpha = 80.347(2)°$, $\beta = 81.880(2)°$, $\gamma = 79.308(2)°$, $V = 813.23(15)$ Å^3, $Z = 2$, Density (Calc.) $= 1.712$ g cm^{-3}, $T = 293(2)$ K, μ(Mo Kα) $= 1.315$ mm^{-1}. Data were collected on a single crystal with dimensions $0.36 \times 0.32 \times 0.08$ mm. 4186 reflections were measured in the range of $1.74° \leq \theta \leq 25.03°$, 2826 independent reflections ($R_{int} = 0.0301$). Final $R_1 = 0.0499$ for 2349 observed reflections [$I > 2\sigma(I)$] and $wR_2 = 0.1207$.

2.2.2. Crystal data for *2*

$C_4H_{26}NiO_{14}P_2$, $M = 418.90$, triclinic, $P\bar{1}$, $a = 6.3924(6)$, $b = 11.0324(11)$, $c = 11.8857(12)$ Å, $\alpha = 80.1470(10)°$, $\beta = 81.5170(10)°$, $\gamma = 79.03°$, $V = 805.03(14)$ Å^3, $Z = 2$, Density (Calc.) $= 1.728$ g cm^{-3}, $T = 293(2)$ K, μ(Mo Kα) $= 1.468$ mm^{-1}. Data were collected on a single crystal with dimensions $0.44 \times 0.42 \times 0.32$ mm. 4170 reflections were measured in the range of $1.75° \leq \theta \leq 25.10°$, 2789 independent reflections ($R_{int} = 0.0246$). Final $R_1 = 0.0447$ for 2582 observed reflections [$I > 2\sigma(I)$] and $wR_2 = 0.1161$.

2.2.3. Crystal data for *3*

$C_4H_{14}CuO_8P_2$, $M = 315.63$, triclinic, $P\bar{1}$, $a = 4.6067(5)$, $b = 5.5119(6)$, $c = 10.6260(12)$ Å, $\alpha = 102.892(3)°$, $\beta = 94.792(2)°$, $\gamma = 101.704(2)°$, $V = 255.19(15)$ Å^3, $Z = 1$, Density (Calc.) $= 2.054$ g cm^{-3}, $T = 293(2)$ K, μ(Mo Kα) $= 2.475$ mm^{-1}. Data were collected on a single crystal with dimensions $0.36 \times 0.14 \times 0.06$ mm. 1303 reflections were measured in the range of $1.98° \leq \theta \leq 25.03°$, 887 independent reflections ($R_{int} = 0.0230$). Final $R_1 = 0.0468$ for 748 observed reflections [$I > 2\sigma(I)$] and $wR_2 = 0.1223$.

2.2.4. Crystal data for *4*

$C_4H_{14}ZnO_8P_2$, $M = 317.46$, monoclinic, $C2/c$, $a = 18.545(2)$, $b = 8.2688(11)$, $c = 8.2977(11)$ Å, $\beta = 114.159(2)°$, $V = 1161.0(3)°$ Å^3, $Z = 4$, Density (Calc.) $= 1.816$ g cm^{-3}, $T = 293(2)$ K, μ(Mo Kα) $= 2.410$ mm^{-1}. Data were collected on a single crystal with dimensions $0.28 \times 0.16 \times 0.12$ mm. 1866 reflections were measured in the range of $2.41° \leq \theta \leq 25.05°$, 1018 independent reflections ($R_{int} = 0.0237$). Final $R_1 = 0.0337$ for 855 observed reflections [$I > 2\sigma(I)$] and $wR_2 = 0.0867$.

2.2.5. Crystal data for *5*

$C_4H_{14}Cu_2O_9P_2$, $M = 395.17$, monoclinic, $C2/m$, $a = 20.036(4)$, $b = 6.6708(14)$, $c = 4.3999(10)$ Å, $\beta = 91.251(4)°$, $V = 587.9(2)$ Å^3, $Z = 2$, Density (Calc.) $= 2.232$ g cm^{-3}, $T = 293(2)$ K, μ(Mo Kα) $= 3.921$ mm^{-1}. Data were collected on a single crystal with dimensions $0.20 \times 0.10 \times 0.03$ mm. 943 reflections were measured in the range of $2.03° \leq \theta \leq 25.05°$, 568 independent reflections ($R_{int} = 0.0359$). Final $R_1 = 0.0661$ for 447 observed reflections [$I > 2\sigma(I)$] and $wR_2 = 0.1799$.

3. Results and discussion

The structure of **1** is a zero-dimensional complex (Fig. 1(a)). The octahedral coordination sites of the Co(II) atom are occupied by six oxygen atoms from six different water molecules with Co–O from 2.058(4) to 2.148(4) Å. There are many hydrogen bonds linking

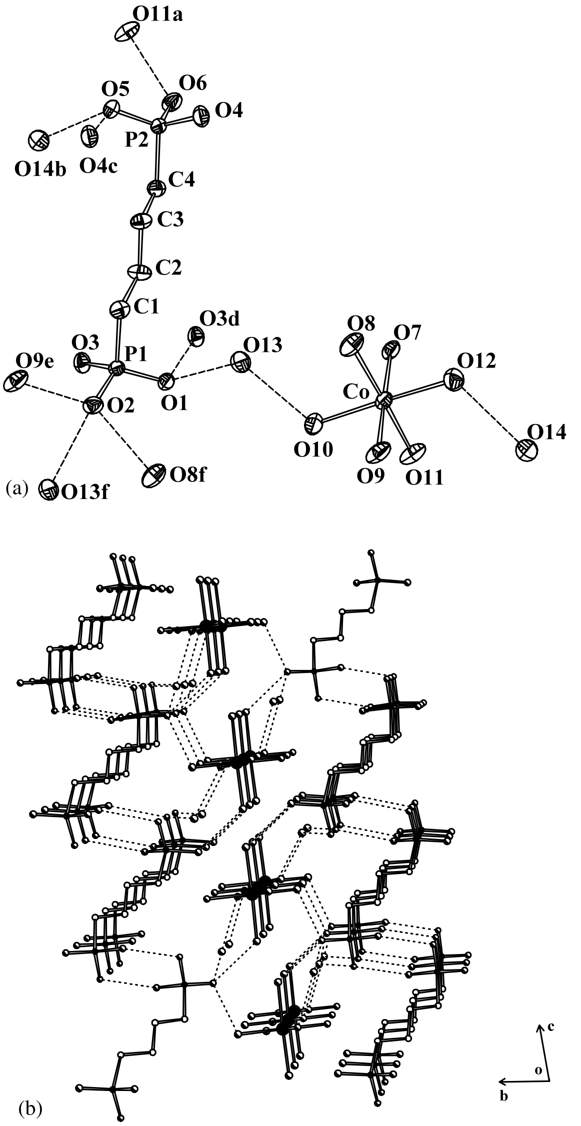


Fig. 1. (a) ORTEP of **1** shows coordination geometries of cobalt and phosphorus (ellipsoids at 50% probability). The dashed lines represent hydrogen bonds. Hydrogen atoms are omitted for clarity. Symmetry code: a: $x, 1+y, -1+z$; b: $1+x, 1+y, -1+z$; c: $1-x, -y, -1-z$; d: $2-x, -y, -z$; e: $2-x, -1-y, -z$; f: $1+x, y, z$. (b) Packing view of **1**. The dotted lines represent hydrogen bonds. The hydrogen atoms are omitted for clarity. Big black dots: Co; Small black dots: P; part filled circle: O; circle: C.

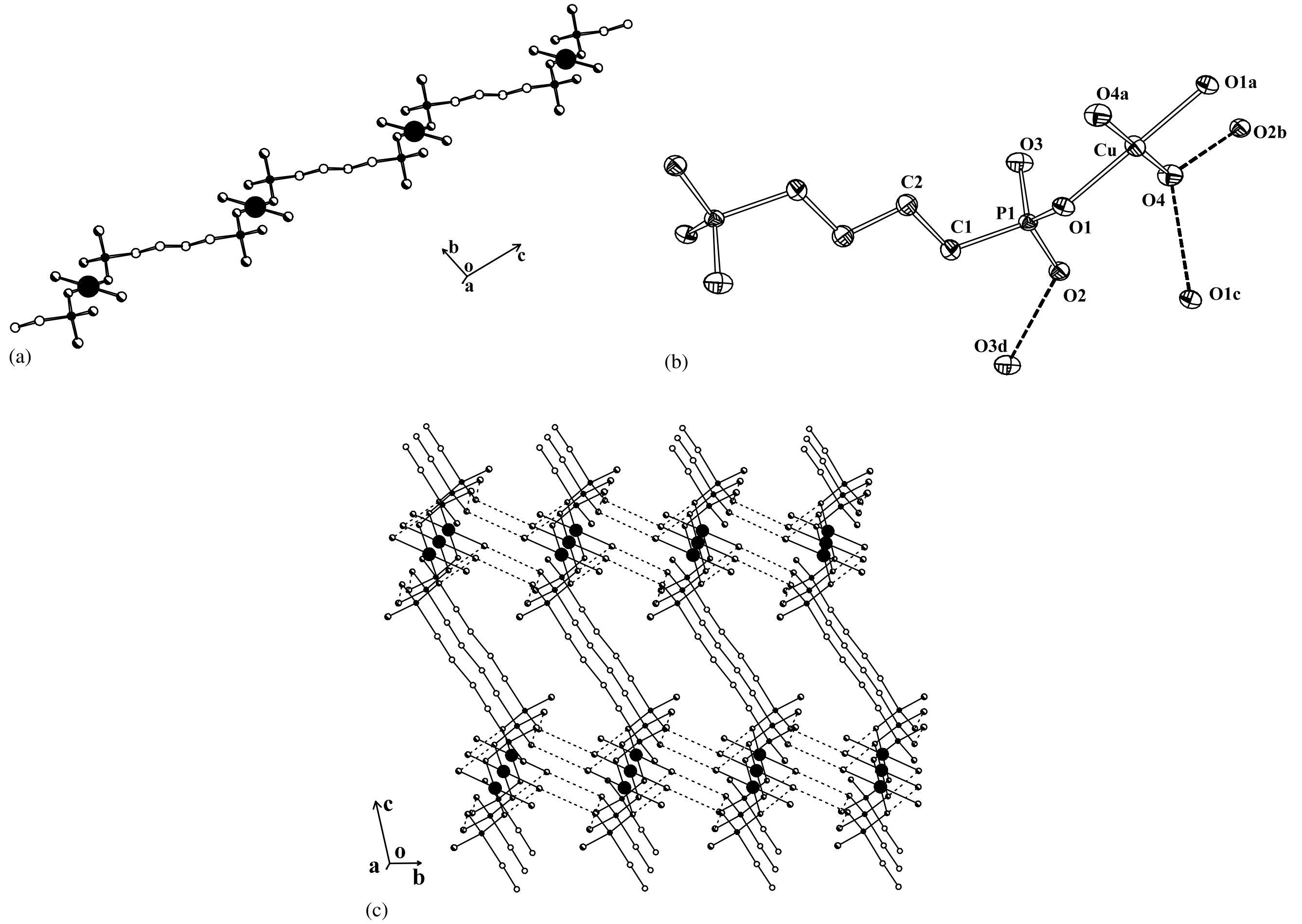
CoO_6 octahedra, 1,4-butylenediphosphonate ligands and free water molecules (Fig. 1(b)).

Compound **2** is isomorphous with **1**. The Ni (II) atom is surrounded by aqua ligands with Ni–O from 2.030(4) to 2.110(4) Å.

The structure of **3** is a stepped chain built up from CuO_4 squares, PCO_3 tetrahedra and butylene bridging units (Fig. 2(a)). As shown in Fig. 2(b), the Cu(II) atom is coordinated by two equivalent oxygen atoms from 1,4-butylenediphosphonate ligands (Cu–O1, 1.957(3) Å) and aqua ligands (Cu–O4, 1.940(3) Å). The hydrogen bonds (O4···O2b = 2.714, O4···O1c = 2.783 and O2···O3d = 2.644 Å) link adjacent chains to form a channel-like framework (Fig. 2(c)).

The structure of **4** is a three-dimensional framework constructed from ZnO_4 tetrahedra, PCO_3 tetrahedra and butylene bridging units (Fig. 3(a)). The Zn(II) center is in a slightly distorted tetrahedral coordination geometry, which is defined by oxygen atoms from two pairs of equivalent 1,4-butylenediphosphonate ligands (Zn–O1, 1.930(3) Å; Zn–O2b, 1.946(2) Å) (Fig. 3(b)). A strong hydrogen bond exists between O_3 and free water molecule O_4 ($O_3\cdots O_4 = 2.550$ Å). Each terminal PO_3 group of 1,4-butylenediphosphonate ligand bonds to two different Zn(II) atoms with its O_1 and O_2 oxygen atoms (Fig. 3(c)). As a result, an 8-membered ring $\{Zn_2P_2O_4\}$ is formed from two adjacent Zn(II) atoms, four oxygen atoms and two phosphorus atoms. The 8-membered rings $\{Zn_2P_2O_4\}$ are linked alternatively through Zn(II) atoms along the *c* axis to form a chain. The chains are linked by butylenes bridging units along the *b* axis to form a channel-like framework. The channel-like structure is distinctly different from those of $Zn(HO_3PC_{12}H_{10}PO_3H)$ [14] and $Zn(HO_3PC_3H_6PO_3H)$ [16], which show double chains.

The structure of **5** has been established previously by X-ray powder diffraction [30]. Herein, we confirm the structure by X-ray single crystal data (Fig. 4). The measured Cu–O bond lengths are 1.942(6), 2.044(5) and 2.412(10) Å based on X-ray single crystal data, while



(a)

(b)

(c)

Fig. 2. (a) View of the stepped chain of **3**. Hydrogen atoms are omitted for clarity. Big black dots: Cu; Small black dots: P; part filled circle: O; circle: C. (b) ORTEP view of **3** shows coordination geometries of copper and phosphorus (ellipsoids at 50% probability). Hydrogen atoms are omitted for clarity. The dashed single lines represent hydrogen bonds. Symmetry code: a: $-x$, $-y$, $-z$; b: $-1-x$, $-1-y$, $-z$; c: $-1-x$, $-y$, $-z$.; d: $-1+x$, y, z. (c) Packing view of **3** shows the channel-like framework. The dashed lines represent the hydrogen bonds. Hydrogen atoms are omitted for clarity. Big black dots: Cu; Small black dots: P; part filled circle: O; circle: C.

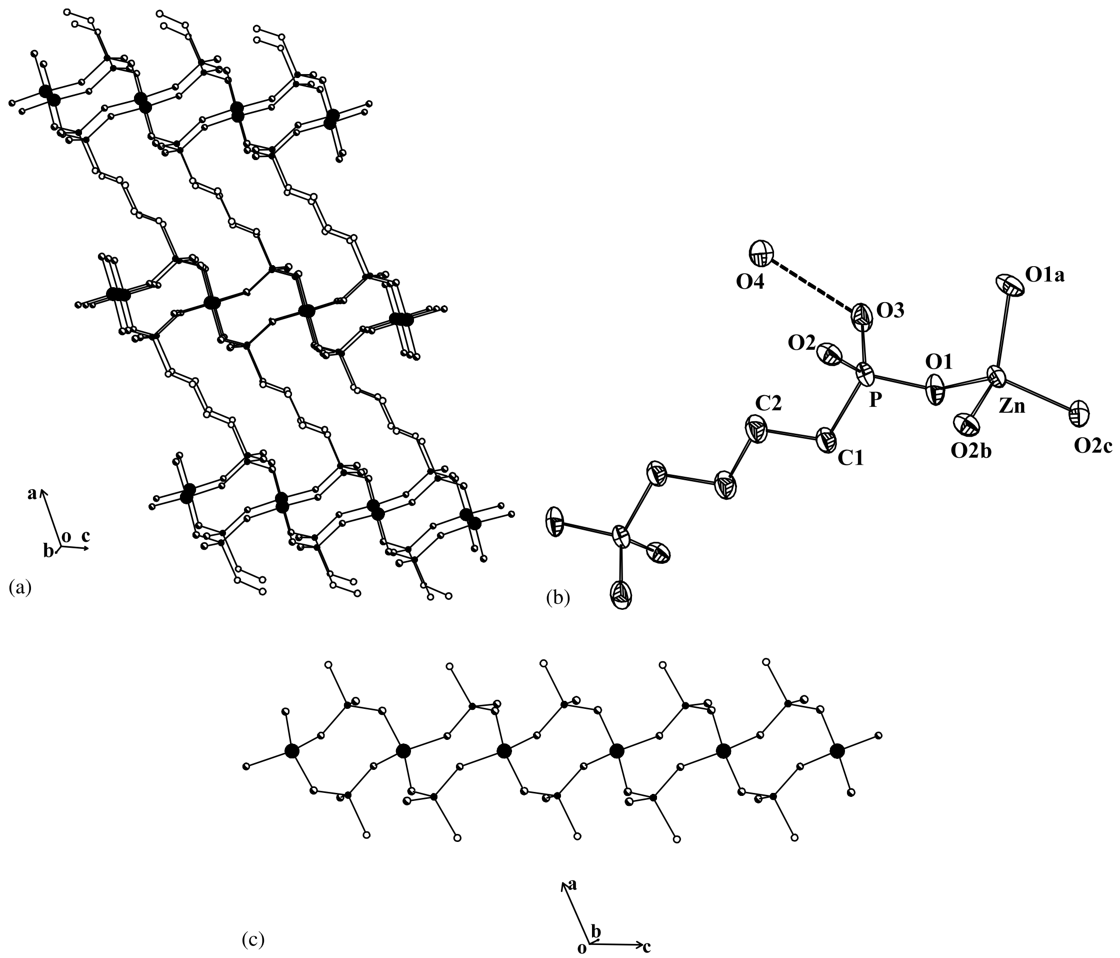


Fig. 3. (a) View of the framework of **4**. Hydrogen atoms and free water molecules are omitted for clarity. Big black dots: Zn; Small black dots: P; part filled circle: O; circle: C. (b) ORTEP view of 4 shows the coordination geometries of zinc and phosphorus (ellipsoids at 50% probability). Hydrogen atoms are omitted for clarity. The dashed single line represents hydrogen bond. Symmetry code: a: $-x, y, -1/2-z$; b: $x, -y, -1/2+z$; c: $-x, -y, -z$. (c): Perspective view of the zinc–oxygen chain of compound **4**. Hydrogen atoms are omitted for clarity. Big black dots: Zn; Small black dots: P; part filled circle: O; circle: C.

they are 1.962(3), 2.048(3) and 2.293(10) or 2.635(8) Å based on X-ray powder data [30]. In addition, the coordinated water molecules is disorder based on X-ray powder data, while it is not disorder based on X-ray single crystal data.

There are four interesting aspects in the structures of these compounds: (1) 1,4-Butylenediphosphonic acid is partly deprotonated in **1**–**4**, but in **5** it is entirely deprotonated; (2) The ratio of metal: 1,4-butylenediphosphonic acid is 1:1 in compound **1**–**4**, while it is 2:1 in compound **5**. (3) There are aqua ligands coordinated to Co, Ni and Cu in **1**–**3** and **5**, while no water molecule is bonded to the Zn(II) center in **4**; (4) Hydrogen bonds play an important role in the structures of these compounds.

In conclusion, we have synthesized suitable single crystals of five transition-metal 1,4-butylenediphosphonates and determined their structures by X-ray single crystal diffraction. The structures are diverse, including

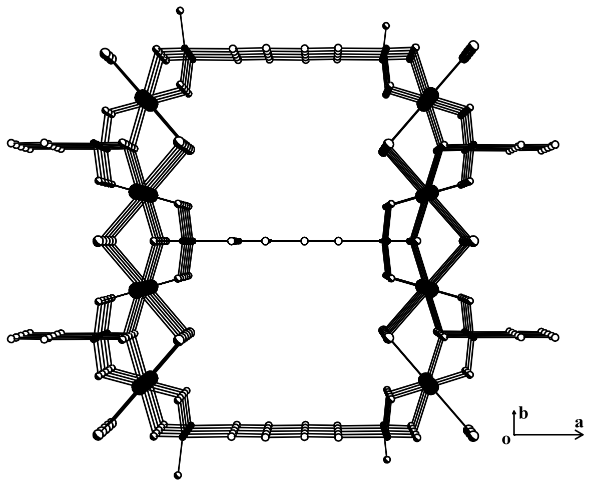


Fig. 4. Packing view of the framework of **5**. The free molecules and hydrogen atoms are omitted for clarity. Big black dots: Cu; Small black dots: P; part filled circle: O; circle: C.

zero-dimensional, chain-like and three-dimensional framework types.

4. Supplementary material

Crystallorgraphic data for these compounds have been deposited with Cambridge Crystallographic Data Center, CCDC No. 200208, 200209, 200210, 200212 and 200213 for compound **1**–**5**, respectively. The supplementary crystallographic data for this paper 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: +44-1223-336033; or e-mail: deposit@ccdc.cam.ac.uk).

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References

[1] A. Clearfield, in: M. Abe, T. Kataoka, T. Susuki (Eds.), New Developments in Ion Exchange Materials, Kodansha Ltd, Tokyo, 1991.
[2] K. Libsonm, E. Deutsch, B.L. Barnett, J. Am. Chem. Soc. 102 (1980) 2476.
[3] S.S. Jurisson, J.J. Benedict, R.C. Elder, R. Whittle, E. Deutsch, Inorg. Chem. 22 (1983) 1332.
[4] G. Cao, H.G. Hong, T.E. Mallouk, Acc. Chem. Res. 25 (1992) 420.
[5] J.C. Horne, G.J. Blanchard, J. Am. Chem. Soc. 110 (1996) 12788.
[6] J.C. Horne, Y. Huang, G.Y. Liu, G.J. Blanchard, J. Am. Chem. Soc. 121 (1999) 4419.
[7] J.C. Horne, G.J. Blanchard, J. Am. Chem. Soc. 121 (1999) 4427.
[8] M.B. Dines, P.M. Digiacomo, Inorg. Chem. 20 (1981) 92.
[9] G. Alberti, U. Costantino, F. Marmottini, R. Vivani, P. Zappelli, Angew. Chem., Int. Ed. 32 (1993) 1357.
[10] G. Alberti, F. Marmottini, S.M. Mascarós, R. Vivani, Angew. Chem., Int. Ed. 33 (1994) 1594.
[11] V. Soghomonian, Q. Chen, R.C. Haushalter, J. Zubieta, Angew. Chem., Int. Ed. 34 (1995) 223.
[12] V. Soghomonian, R. Diaz, R.C. Haushalter, C.J. O'Connor, J. Zubieta, Inorg. Chem. 34 (1995) 4460.
[13] D.M. Poojary, B.L. Zhang, P. Bellinghausen, A. Clearfield, Inorg. Chem. 35 (1996) 4942.
[14] D.M. Poojary, B.L. Zhang, P. Bellinghausen, A. Clearfield, Inorg. Chem. 35 (1996) 5254.
[15] G.H. Bonavia, R.C. Haushalter, C.J. O'Connor, J. Zubieta, Inorg. Chem. 35 (1996) 5603.
[16] D.M. Poojary, B.L. Zhang, A. Clearfield, J. Am. Chem. Soc. 119 (1997) 12550.
[17] D.L. Lohse, S.C. Sevov, Angew. Chem., Int. Ed. 36 (1997) 1619.
[18] G. Bonavia, R.C. Haushalter, S.C. Lu, C.J. O'Connor, J. Zubieta, J. Solid State Chem. 132 (1997) 144.
[19] D. Riou, C. Serre, G. Férey, J. Solid State Chem. 141 (1998) 89.
[20] D. Riou, G. Férey, J. Mater. Chem. 8 (1998) 2733.
[21] F. Serpaaggi, G. Férey, J. Mater. Chem. 8 (1998) 2749.
[22] L.M. Zheng, C.Y. Duan, X.R. Ye, L.Y. Zhang, C. Wang, X.Q. Xin, J. Chem. Soc., Dalton Trans. (1998) 905.
[23] B.L. Zhang, D.M. Poojary, A. Clearfield, Inorg. Chem. 37 (1998) 1844.
[24] L.M. Zhang, H.H. Song, C.H. Lin, S.L. Wang, Z. Hu, Z. Yu, X.Q. Xin, Inorg. Chem. 38 (1999) 4618.
[25] L.M. Zheng, H.H. Song, C.Y. Duan, X.Q. Xin, Inorg. Chem. 38 (1999) 5061.
[26] A. Distler, D.L. Lohse, S.C. Sevov, J. Chem. Soc., Dalton Trans. (1999) 1805.
[27] D. Riou, C. Serre, J. Provost, G. Férey, J. Solid State Chem. 155 (2000) 238.
[28] A. Clearfield, D.M. Poojary, B.L. Zhang, B.Y. Zhao, A.D. Kovacs, Chem. Mater. 12 (2000) 2745.
[29] C. Serre, G. Férey, Inorg. Chem. 40 (2001) 5350.
[30] D.I. Arnold, X. Ouyang, A. Clearfield, Chem. Mater. 14 (2002) 2020.
[31] O.R. Evans, D.R. Manke, W.B. Lin, Chem. Mater. 14 (2002) 3866.
[32] H. Jankovics, M. Daskalakis, C.P. Raptopoulou, A. Terzis, V. Tangoulis, J. Giapintzakis, T. Kiss, A. Salifoglou, Inorg. Chem. 41 (2002) 3366.
[33] L.M. Zhang, P. Yin, X.Q. Xin, Inorg. Chem. 41 (2002) 4084.
[34] C.V.K. Sharma, A. Clearfield, J. Am. Chem. Soc. 123 (2001) 2885.
[35] J.G. Mao, A. Clearfield, Inorg. Chem. 41 (2002) 2319.
[36] A. Cabeza, X. Ouyang, C.V.K. Sharma, M.A.G. Aranda, S. Bruque, A. Clearfield, Inorg. Chem. 41 (2002) 2325.
[37] J.G. Mao, Z. Wang, A. Clearfield, New J. Chem. 26 (2002) 1010.
[38] A.K. Bhattacharya, G. Thyagarajan, Chem. Rev. 81 (1981) 415.
[39] K. Moedritzer, R.R. Irani, J. Inorg. Nucl. Chem. 22 (1961) 297.
[40] G.M. Sheldrick, SHELXT-97, Program for Crystal Structure Refinement, University of Göttingen, Germany, 1997.