

基于 2,5-二甲基苯-1,4-二亚甲基二膦酸的钴、镍同构化合物的水热合成及晶体结构

张爱江* 陈长娟

(黄淮学院化学化工系, 驻马店 463000)

摘要: 在水热条件下, 利用 2,5-二甲基苯-1,4-二亚甲基二膦酸(H_4L)与 $CoCl_2 \cdot 6H_2O$ 或 $NiSO_4 \cdot 6H_2O$ 反应得到 2 个同构的过渡金属有机膦酸化合物, $[Co(H_2O)_4(H_2L)]_n$ (**1**), $[Ni(H_2O)_4(H_2L)]_n$ (**2**), 并用元素分析、红外光谱、粉末及单晶 X-射线等方法对其进行了表征。晶体结构分析表明: 化合物 **1** 和 **2** 都属于三斜晶系, 空间群为 $P\bar{1}$, 化合物 **1** 的晶胞参数为 $a=0.497\ 0(2)\text{ nm}$, $b=0.711\ 3(3)\text{ nm}$, $c=1.177\ 8(5)\text{ nm}$, $\alpha=97.779(7)^\circ$, $\beta=92.103(7)^\circ$, $\gamma=107.217(6)^\circ$, $V=0.3927(3)\text{ nm}^3$, $Z=2$; 化合物 **2** 的晶胞参数为 $a=0.494\ 35(19)\text{ nm}$, $b=0.708\ 9(3)\text{ nm}$, $c=1.172\ 6(5)\text{ nm}$, $\alpha=97.919(6)^\circ$, $\beta=92.130(6)^\circ$, $\gamma=107.441(5)^\circ$, $V=0.387\ 0(3)\text{ nm}^3$, $Z=2$ 。金属离子采取了八面体构型, 6 个配位氧原子分别来自 2 个反式构型的 H_2L 配体和 4 个配位水分子。每 1 个金属离子与 2 个反式构型的 H_2L 配体配位形成了一维线型链状结构。这 2 个化合物最终通过 $O-H\cdots O$ 氢键作用形成了三维结构。此外, 对 2 个化合物的热稳定性也进行了研究。

关键词: 钴(II); 镍(II); 2,5-二甲基苯-1,4-二亚甲基二膦酸; 水热合成; 晶体结构

中图分类号: O614.81+2; O614.81+3

文献标识码: A

文章编号: 1001-4861(2014)07-1712-07

DOI: 10.11862/CJIC.2014.221

Isostructural Transition Metal (Co, Ni) Phosphonates of 2,5-Dimethylbenzene-1,4-diylbis (methylene)diphosphonic Acid: Hydrothermal Synthesis and Crystal Structures

ZHANG Ai-Jiang* CHEN Chang-Juan

(Department of Chemistry and Chemical Engineering, huanghuai university, zhumadian, Henan 463000, China)

Abstract: Two new isostructural cobalt and nickel phosphonates $[M(H_2O)_4(H_2L)]_n$, $M=Co$ (compound **1**), $M=Ni$ (compound **2**) have been hydrothermally synthesized from 2,5-dimethylbenzene-1,4-diylbis (methylene)diphosphonic acid (H_4L) and the corresponding metal salts. And the compounds **1** and **2** have been characterized by routine elemental analyses, IR, powder and single-crystal X-ray diffraction methods. Both compounds crystallize in triclinic space group $P\bar{1}$ with $a=0.497\ 0(2)\text{ nm}$, $b=0.711\ 3(3)\text{ nm}$, $c=1.177\ 8(5)\text{ nm}$, $\alpha=97.779(7)^\circ$, $\beta=92.103(7)^\circ$, $\gamma=107.217(6)^\circ$, $V=0.3927(3)\text{ nm}^3$ and $Z=2$ for **1**, $a=0.494\ 35(19)\text{ nm}$, $b=0.708\ 9(3)\text{ nm}$, $c=1.172\ 6(5)\text{ nm}$, $\alpha=97.919(6)^\circ$, $\beta=92.130(6)^\circ$, $\gamma=107.441(5)^\circ$, $V=0.387\ 0(3)\text{ nm}^3$ and $Z=2$ for **2**. Metal ion have all octahedral geometry coordinated to six oxygen atom from two *trans*- H_2L ligands and four coordinated water molecules. Each metal ion is coordinated to two *trans*- H_2L ligands thereby generating an infinite one-dimensional (1D) linear chain. Finally, all the compounds show three-dimensional (3D) network through $O-H\cdots O$ hydrogen bonds. Thermal stability of compounds **1** and **2** have also been investigated. CCDC: 934867, **1**; 935051, **2**.

Key words: cobalt(II); nickel(II); 2,5-dimethylbenzene-1,4-diylbis(methylene)diphosphonic acid; hydrothermal synthesis; crystal structure

收稿日期: 2013-12-08。收修改稿日期: 2014-02-10。

国家自然科学基金(No.21301064); 河南省基础与前沿技术研究(No.132300410413)资助项目。

*通讯联系人。E-mail: zaj315@163.com; 会员登记号: S06N5325M1404。

Metal phosphonates are very attractive in the field of organic-inorganic hybrid materials due to their unusual structures and properties which may lead to potential applications in the fields of catalysis^[1], magnetism^[2-3], optics^[4], gas storage and separation^[5], ion exchange^[6], corrosion inhibition^[7], and so on^[8]. Therefore, the rational design and assembly of novel metal phosphonates with the intriguing diversity of the architectures and properties has become a particularly important subject^[9]. So far, a great number of compounds with interesting architectures and possible functionalities have been reported by rational designing phosphonate ligands with different functional groups such as pyridyl^[10], carboxylic^[11], hydroxyl^[12], amino^[13], macrocyclic^[14] groups etc^[15]. However, it is a great challenge to control structures of metal phosphate with desired properties because many factors affect the result, such as central metal, geometry of the phosphonic acids, solvents, temperature, pH value, and so on^[16]. The most effective approach to overcome this problem is a appropriate choice of the well-designed phosphates bridging ligands.

Recently, the diphosphonic acids, $\text{H}_2\text{O}_3\text{P-R-PO}_3\text{H}_2$ (R=alkyl or aryl group), have been proved to be very useful ligands for the synthesis of metal phosphonates with new structure types in which the organic part plays a controllable spacer role and the two inorganic- PO_3 groups chelate with metal ions to form one-, two-, and three-dimensional structures^[17-21]. In order to extend our investigation in this field, we chose 2,5-dimethylbenzene-1,4-diylbis (methylene) diphosphonic acid (H_4L) as the organic bridging ligand, based on the following considerations: (i) H_4L is a flexible ligand that not only includes rigid phenyl group but also bearing a flexible $-\text{OCH}_2-$ spacer, which allows more flexibility and may result in rich topologies, including coordination modes, packing fashions. (ii) The plenty of O atoms and OH groups of H_4L can serve as hydrogen bond acceptors as well as donors to construct supramolecular frameworks.

Taking inspiration from the aforementioned points, Herein we report on their syntheses, crystal structures, thermal stability of two new cobalt and

nickel phosphonates based on 2,5-dimethylbenzene-1,4-diylbis(methylene)diphosphonic acid.

1 Experimental

1.1 Materials and instruments

All chemicals were of reagent grade quality obtained from commercial sources and used without further purification. Solvents were purified according to the standard methods. Elemental analyses (C and H) were performed on an Elementar Vario MICRO Elemental Analyzer at the analysis of huanghuai university. Infrared spectra were recorded on a Bruker Tensog 27 FTIR spectrophotometer in the $400\sim 4\,000\text{ cm}^{-1}$ region by using KBr pellets. Solution ^1H NMR spectra were recorded on a Bruker AVANCE-III NMR (600 MHz). Power X-ray diffraction (PXRD) measurements of the compounds were recorded on a Bruker D8 Advanced X-ray diffraction using $\text{Cu K}\alpha$ radiation ($\lambda=0.154\,18\text{ nm}$), and the X-ray tube was operated at 40 kV and 40 mA. The thermogravimetric analysis was carried out on a SDT Q600 thermal analyzer at a heating rate of $10\text{ }^\circ\text{C}\cdot\text{min}^{-1}$ from 25 to $1\,000\text{ }^\circ\text{C}$ under a nitrogen atmosphere.

1.2 Synthesis of ligand

The H_4L was synthesized by reacting 1,4-bis (bromomethyl)-2,5-dimethylbenzene^[22] with triethylphosphite and followed by refluxing the obtained oil with conc. hydrochloric acid according to the literature methods^[23]. ^1H NMR (600 MHz, $\text{DMSO-}d_6$, ppm): δ 6.96 (s, 2H, Ar-H); 2.99 (d, 2H, $J=18\text{ Hz}$, Ar- CH_2); 2.96 (d, 2H, $J=18\text{ Hz}$, Ar- CH_2); 2.90 (d, 2H, $J=18\text{ Hz}$, Ar- CH_2); 2.87 (d, 2H, $J=18\text{ Hz}$, Ar- CH_2); 2.25 (s, 6H, Ar- CH_3). Anal. Calcd. for $\text{C}_{10}\text{H}_{16}\text{O}_6\text{P}_2$ (%): C, 40.83; H, 5.48. Found (%): C, 41.04; H, 5.05. IR (KBr pellet, cm^{-1}): 3 431(b), 2 981(m), 2 916(w), 2 271(m), 1 639 (m), 1 510(m), 1 462(m), 1 408(m), 1 387(m), 1 257 (m), 1 216(m), 1 162(m), 1 127(m), 1 052(m), 1 012 (m), 995(m), 954(m), 899(w), 852(w), 761(w), 737(w), 601(w).

1.3 Synthesis of compounds

1.3.1 Synthesis of $[\text{Co}(\text{H}_2\text{O})_4(\text{H}_2\text{L})]_n$ (1)

A 0.147 g portion (0.50 mmol) of H_4L was mixed with 0.119 g of $\text{CoCl}_2\cdot 6\text{H}_2\text{O}$ (0.50 mmol) in 15 mL of

deionized distilled water, and the mixture was heated in Teflon-lined steel vessel (Volume ~25 mL) at 160 °C for 3 d. The vessel was cooled to room temperature, and X-ray-diffraction-quality pink single crystals were collected from the Teflon liner by filtration in 71% yield (based on Co). Anal. Calcd. for $C_{10}H_{22}O_{10}P_2Co$ (%): C, 28.40; H, 5.24. Found(%): C, 28.75; H, 5.35. IR (KBr pellet, cm^{-1}): 3 532(s), 3 242(s, b), 2 915(w), 2 275(b), 1 654(m), 1 507(m), 1 461(w), 1 413(m), 1 393(w), 1 249(m), 1 207(s), 1 184(s), 1 110(m), 1 016(s), 930(s), 755(m), 703(m), 604(s), 505(s), 464(w).

1.3.2 Synthesis of $[Ni(H_2O)_4(H_2L)]_n$ (**2**)

Same method was employed to synthesize compound **2**, the only difference is the displacement of $CoCl_2 \cdot 6H_2O$ with $NiSO_4 \cdot 6H_2O$. The X-ray-diffraction-quality greenish single crystals were collected from the Teflon liner by filtration in 52% yield (based on Ni). Anal. Calcd. for $C_{10}H_{22}O_{10}P_2Ni$ (%): C, 28.40;

H, 5.24. Found(%): C, 28.65; H, 5.30. IR (KBr pellet, cm^{-1}): 3 527(s), 3 200(s, b), 2 917(w), 2 274(b), 1 659(m), 1 507(m), 1 459(w), 1 413(m), 1 393(w), 1 252(s), 1 186(s), 1 107(m), 1 019(s), 928(s), 755(m), 704(m), 604(m), 507(s), 466(w).

1.4 Crystal structure determination

Single-crystal X-ray diffraction measurements of compounds **1**, and **2** were carried out on a Bruker SMARTAPEXII CCD diffractometer (Mo $K\alpha$ radiation, $\lambda=0.071\ 073$ nm) at 295(2) and 273(2) K using ω -scan technique. SAINT was used for integration of intensity of reflections and scaling^[24]. Semiempirical absorption corrections were carried out with the program SADABS^[25]. Crystal structures were solved by direct methods using SHELXS^[26]. Subsequent difference Fourier analyses and least squares refinement with SHELXL-97^[27] allowed for the location of the atom positions. In the final step of the crystal structure

Table 1 Crystal data and structure refinements for **1** and **2**

Compound	1	2
Empirical formula	$C_5H_{11}O_5PCo_{0.5}$	$C_5H_{11}O_5PNi_{0.5}$
Temperature / K	295(2)	273(2)
Wavelength / nm	0.071 073	0.071 073
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a / nm	0.497 0(2)	0.494 35(19)
b / nm	0.711 3(3)	0.708 9(3)
c / nm	1.177 8(5)	1.172 6(5)
α / (°)	97.779(7)	97.919(6)
β / (°)	92.103(7)	92.130(6)
γ / (°)	107.217(6)	107.441(5)
Volume / nm ³	0.392 7(3)	0.387 0(3)
Z	2	2
μ / mm ⁻¹	1.345	1.510
θ range for the data collection / (°)	1.75 to 24.98	1.76 to 26.19
Index ranges	$-5 \leq h \leq 5, -8 \leq k \leq 8, -13 \leq l \leq 13$	$-6 \leq h \leq 6, -8 \leq k \leq 8, -14 \leq l \leq 13$
Reflection collected	2 788	2 847
Independent reflections	1 365	1 513
R_{int}	0.029 6	0.030 4
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	1 365 / 0 / 107	1 513 / 0 / 107
GOF	1.604	1.106
$R_1(F)$, $wR_2(F^2)$ ($I > 2\sigma(I)$)	0.053 8, 0.141 2	0.042 2, 0.104 9
$R_1(F)$, $wR_2(F^2)$ (all data)	0.061 6, 0.147 2	0.053 8, 0.113 8
Largest diff. peak and hole / ($e \cdot nm^{-3}$)	1 536 and -381	494 and -460

Table 2 Selected bond lengths (nm) and angles (°) for **1** and **2**

1					
Co1-O2W	0.206 4(3)	Co1-O2	0.208 4(3)	Co1-O1W	0.214 2(3)
O2W-Co1-O2WC	180.00(13)	O2W-Co1-O2C	87.39(13)	O2WC-Co1-O2C	92.61(13)
O2C-Co1-O2	180.000(1)	O2W-Co1-O1W	88.94(12)	O2WC-Co1-O1W	91.06(12)
O2C-Co1-O1W	87.46(13)	O2-Co1-O1W	92.54(13)		
2					
Ni1-O2W	0.202 7(3)	Ni1-O2	0.205 9(3)	Ni1-O1W	0.208 4(3)
O2W-Ni1-O2WC	180.00(13)	O2W-Ni1-O2C	87.23(11)	O2WC-Ni1-O2C	92.77(11)
O2C-Ni1-O2	180.000(10)	O2W-Ni1-O1W	89.81(11)	O2WC-Ni1-O1W	90.19(11)
O2C-Ni1-O1W	87.81(11)	O2-Ni1-O1W	92.19(11)		

Symmetry code: C: 2-x, -y, 2-z.

refinement hydrogen atoms of idealized-CH₂ and -CH₃ groups were added and treated with their riding atom mode, their isotropic displacement factor was chosen as 1.2 and 1.5 times the preceding carbon atom, respectively. The hydrogen atoms on the water molecules of **1** and **2** were found in the electron density map and refined by riding. Details of the crystal parameters, data collection and refinements for **1** and **2** are summarized in Table 1, and selected bond lengths and angles with estimated standard deviations are listed in Table 2.

CCDC: 934867, **1**; 935051, **2**.

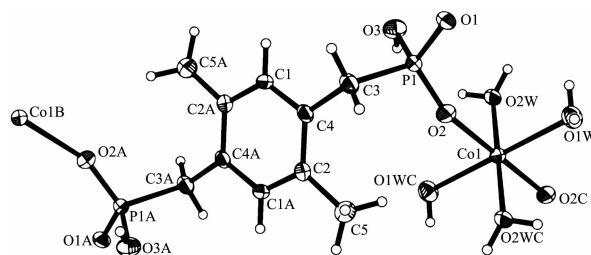
2 Result and discussion

2.1 Crystal structure description of compounds **1** and **2**

Single-crystal X-ray structure analysis revealed that the compounds **1** and **2** are isostructural. Therefore, compound **1** is taken as an example to present and discuss the structure in detail. The compound **1** crystallizes in triclinic system with $P\bar{1}$ space group. The asymmetry unit of **1** (Fig.1) consists of a half of Co(II) ion, a half of partial deprotonated diphosphonate ligand(H₂L), and two coordination water molecules. The Co(II) ion is six-coordinated in an octahedral coordination environment by two phosphonate oxygen atoms (O2, O2C) from two different H₂L ligands and four oxygen atoms (O1W, O2W, O1WC O2WC) from four water molecules. The bond angles of O2W-Co1-O1WC, O2W-Co1-O1WC, O1WC-Co1-

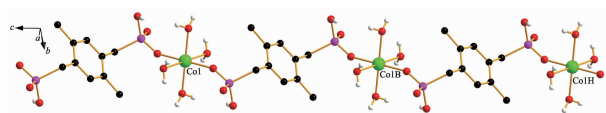
O2WC, O2WC-Co1-O1W are add up to equal to 360°, show that O1W, O2W, O1WC and O2WC atoms are in the equatorial position. Moreover, the bond angles of O2-Co1-O2C, O2W-Co1-O2WC, O1W-Co1-O1WC are also equal to 180°, further indicating that the geometries around each metal center are all octahedral. The Co-O bonds lengths and O-Co-O bond angles fall in the range from 0.206 4(3) to 0.214 2(3) nm and 87.39(13)° to 180.00(13)°, respectively, comparable to those reported for other cobalt (II) phosphonates^[28-29] (see Table 2). In compound **1**, the diphosphonate ligand (H₂L) is doubly deprotonated and adopts a trans configuration. Adjacent Co(II) center are bridged by H₂L ligand to generate an infinite one-dimensional linear chain structure along the *c*-axis through Co-O coordination interactions, with Co...Co separation of 1.177 8 nm (Fig.2).

As expected, significant O-H...O hydrogen bonding interactions exist in the compound **1**. Although



Symmetry code: A: 2-x, -y, 1-z; B: +x, +y, -1+z; C: 2-x, -y, 2-z

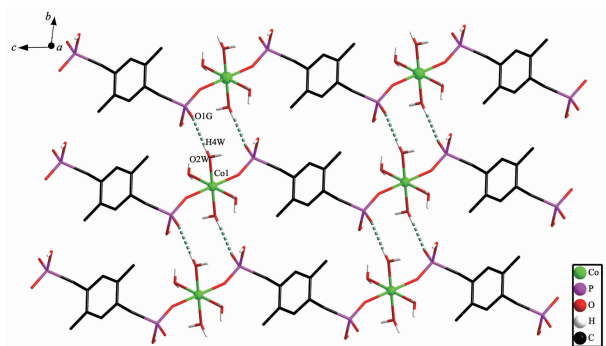
Fig.1 Coordination environment of Co(II) ion in **1** with the thermal ellipsoid at the 50% probability level



Symmetry code: B: +x; +y; -1+z; H: x, y, -2+z

Fig.2 View of the 1D linear chain for **1**

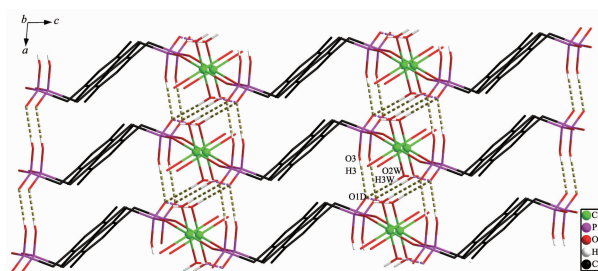
hydrogen bonds are typically weak interactions, they play an important role in crystal packing^[30]. First, the 1D linear chains form two-dimensional (2D) layers in the *bc* plane by the O—H...O hydrogen bonding between the uncoordinated phosphonate groups O atoms and the O atoms of coordinated water molecular with the O2W...O1G distance of 0.268 21(4) nm and O2W—H4W...O1G angle of 174° (Fig.3). Furthermore, these 2D layers are therefore assembled together to build a three-dimensional network via intermolecular



Symmetry codes: G: 2-x, 1-y, 2-z

Fig.3 2D layer structure in **1** connected by O—H...O hydrogen bonds indicated by dashed lines

hydrogen bonds (O3—H3W...O1D; O2W—H3W...O1D) in the other direction (Fig.4). The length and angles of the hydrogen bonds for **1** and **2** are listed in Table 3.



Symmetry codes: D: 1+x, y, z

Fig.4 3D network of **1** linked by O—H...O hydrogen bonds indicated by dashed lines

2.2 IR spectrum

In the spectra, the broad bands centered around 3 216 and 2 274 cm^{-1} can be attributed to the asymmetric and symmetric O—H stretching vibrations of the water molecules and the protonated P—O—H groups. As expected, the characteristic vibrations of the CPO_3 groups were observed for the ligand and the two compounds in the range of 1 260~900 cm^{-1} . The band around 1 250 cm^{-1} can be assigned to the P—C stretching vibration. Similarly, both the asymmetric stretching vibrations, $\nu_{\text{as}}(\text{PO}_3)$, (**1**: 1 184, 1 110; **2**: 1 186, 1 107; and symmetric stretching vibrations, $\nu_{\text{s}}(\text{PO}_3)$, (**1**: 1 016, 930; **2**: 1 019, 928;) of compounds

Table 3 Parameters of hydrogen bonds for **1** and **2**

D—H...A	$d(\text{D—H}) / \text{nm}$	$d(\text{H...A}) / \text{nm}$	$d(\text{D...A}) / \text{nm}$	$\angle \text{DHA} / (^\circ)$
1				
O2W—H4W...O1G	0.083	0.186	0.268 21(4)	174
O2W—H3W...O1D	0.082	0.190	0.269 51(4)	162
O3—H3...O1D	0.082	0.194	0.272 3(4)	159
O1W—H1W...O2WF	0.082	0.223	0.287 2(5)	135
O1W—H1W...O2E	0.082	0.229	0.299 4(5)	144
O1W—H2W...O3G	0.080	0.229	0.304 41(5)	158
2				
O2W—H4W...O1G	0.082	0.187	0.268 7(4)	174
O2W—H3W...O1D	0.082	0.189	0.267 9(4)	161
O3—H3...O1D	0.082	0.194	0.270 4(4)	155
O1W—H1W...O2WF	0.082	0.222	0.285 8(4)	135
O1W—H1W...O2E	0.082	0.228	0.298 7(4)	144
O1W—H2W...O3G	0.079	0.230	0.304 1(4)	157

Symmetry codes: D: 1+x, y, z; E: 1-x, -y, 2-z; F: x-1, y, z; G: 2-x, 1-y, 2-z.

1 and **2** are found shifting to lower values in comparison to the free ligand (ν_{as} : 1 257, 1 216, 1 127; ν_{s} : 1 052, 995, 954)^[31-32], indicating changes in the vibrational status of the ligand upon complexation.

2.3 Thermogravimetric analyses and PXRD result

The thermal stability of compounds **1** and **2** were examined by thermogravimetric analyses (TGA) in the 30 ~1 000 °C range. Thermograms show similar profiles and are provided as Fig.5. The compounds are thermally stable up to 100 °C with the first weight loss between 101 and 200 °C (16.93% for **1**, 16.31% for **2**), corresponding to the release of two coordinated water molecules (Calcd. 17.03% for **1**, 17.03% for **2**). Then a gradual and continuing decomposition about (45.23% for **1**, 44.91% for **2**) occurred. At the end of the measurement (1 000°C) they were still not finished. They could be attributed to the partial thermal decomposition of the organic components in the compounds.

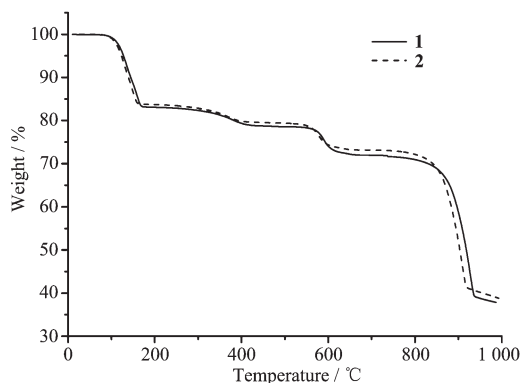


Fig.5 Thermogravimetric analyses (TGA) Curves of **1** and **2**

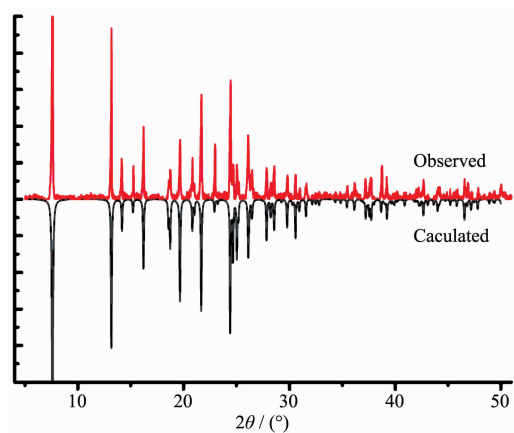


Fig.6 Observed and calculated X-ray powder diffraction patterns of **1**

In an attempt to confirm the homogeneity of the material synthesized under the hydrothermal condition, PXRD measurements were carried out for **1** and **2** at room temperature. The experimental and simulated PXRD patterns for **1** and **2** are shown in Fig.6 and Fig. 7, which demonstrate that the diffraction peaks of simulated and experimental patterns match well. The results indicate that the bulk products obtained are homogeneous in nature.

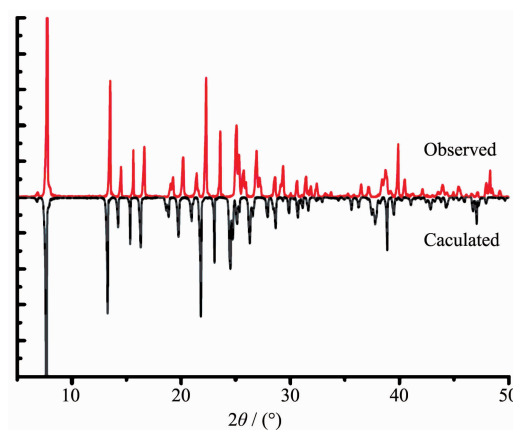


Fig.7 Observed and calculated X-ray powder diffraction patterns of **2**

References:

- [1] Kirumakki S, Samarajeewa S, Harwell R, et al. *Chem. Commun.*, **2008**:5556-5558
- [2] Bao S S, Liao Y, Zheng L M, et al. *Angew. Chem. Int. Ed.*, **2011**,**50**:5504-5508
- [3] YANG Xiao-Jing(杨晓婧), BAO Song-Song(鲍松松), ZHENG Li-Min(郑丽敏). *Chinese J. Inorg. Chem.*(无机化学学报), **2013**,**29**(3):621-627
- [4] Mao J G. *Coord. Chem. Rev.*, **2007**,**251**:1493-1520
- [5] Zhai F P, Zheng Q S, Zhou Y M, et al. *CrystEngComm*, **2013**,**15**:2040-2043
- [6] Plabst M, McCusker L B, Bein J. *J. Am. Chem. Soc.*, **2009**, **131**:18112-18118
- [7] Demadis K D, Papadaki M, Císařová I. *ACS Appl. Mater. Interfaces*, **2010**,**2**:1814-1816
- [8] Shimizu G K H, Vaidhyanathan R, Taylor J M. *Chem. Soc. Rev.*, **2009**,**38**:1430-1449
- [9] Gagnon K J, Perry H P, Clearfield A. *Chem. Rev.*, **2012**, **112**:1034-1045
- [10] Yang T H, Liao Y, Zheng L M, et al. *Chem. Commun.*, **2009**:5556-5558
- [11] Chu W, Sun Z G, Jiao C Q, et al. *Dalton Trans.*, **2013**,**42**:

- 8009-8017
- [12]Hix G B, Turner A, Kariuki, et al. *J. Mater. Chem.*, **2012**, **12**:3220-3227
- [13]Zhou T H, Yi F Y, Mao J G, et al. *Inorg. Chem.*, **2010**,**49**: 905-915
- [14]Ngo H L, Lin W B. *J. Am. Chem. Soc.*, **2002**,**124**:14298-14299
- [15]Du Z Y, Wen H R, Liu C M, et al. *Cryst. Growth Des.*, **2010**,**10**:3721-3726
- [16]Ouellette W, Yu M H, Zubieta J, et al. *Inorg. Chem.*, **2006**, **45**:3224-3239
- [17]Vilela S M F, Mendes R F, Paz F A A, et al. *Cryst. Growth Des.*, **2013**,**13**:543-560
- [18]Tripuramallu B K, Mukherjee S, Das S K. *Cryst. Growth Des.*, **2012**,**12**:5579-5597
- [19]Bialek M J, Janczak J, Zoń J. *CrystEngComm*, **2013**,**15**:390-399
- [20]Rueff J M, Perez O, Pautrat A, et al. *Inorg. Chem.*, **2012**,**51**: 10251-10261
- [21]Zoń J, Kong D Y, Cagnon K, et al. *Dalton Trans.*, **2010**,**39**: 11008-11018
- [22]Granzhan A, Ihmels H, Mikhlin K. *Eur. J. Org. Chem.*, **2005**,**19**:4098-4108
- [23]Murugavel R, Singh M P. *New J. Chem.*, **2010**,**34**:1846-1854
- [24]SAINT, Version 6.45, Bruker Analytical X-ray Systems Inc., **2003**.
- [25]Sheldrick G M. *SADABS*, Version 2.10, Bruker AXS Inc., Madison, Wisconsin, USA, **2003**.
- [26]Sheldrick G M. *SHELXS-97*, Program for the Solution of Crystal Structures, University of Göttingen, Germany, **1997**.
- [27]Sheldrick G M. *SHELXTL*, Crystallographic Software Package, *SHELXTL*, Version 5.1, Bruker-AXS, Madison, Wisconsin, USA, **1998**.
- [28]Wang P F, Duan Y, Song Y, et al. *Chem. Eur. J.*, **2011**,**17**: 3579-3583
- [29]Perry H P, Gagon K J, Law J, et al. *Dalton Trans.*, **2012**,**41**: 3985-3994
- [30]SU Cheng-Yong(苏成勇), PAN Mei(潘梅). *Supramolecular Coordination Chemistry Structures and Functions*(配位超分子结构化学基础与进展). Beijing: Science Press, **2010**:43-53
- [31]Tang S F, Song J L, Mao J G, et al. *Cryst. Growth Des.*, **2007**,**7**:360-366
- [32]Ayi A A, Kinnibrough T L, Clearfield A. *Dalton Trans.*, **2011**, **40**:12648-12650