

$$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.3136P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

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Table 1. Selected bond lengths (Å)

Cu—O1	1.9206 (14)	O9—C6	1.252 (2)
Cu—O3	1.9351 (13)	O10—C7	1.253 (2)
Cu—O4	1.9585 (13)	O11—C7	1.2414 (19)
Cu—O2	1.9749 (13)	C1—C5	1.441 (2)
Cu—O5	2.3763 (15)	C1—C2	1.444 (2)
Cu—O6	2.6746 (16)	C2—C3	1.346 (2)
O6—C1	1.2471 (19)	C3—C6	1.517 (2)
O7—C3	1.3485 (17)	C4—C5	1.341 (2)
O7—C4	1.3524 (17)	C4—C7	1.512 (2)
O8—C6	1.240 (2)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O12—H12A...O8 <sup>i</sup>	0.925 (16)	1.853 (16)	2.776 (2)	175 (3)
O12—H12B...O5 <sup>ii</sup>	0.970 (16)	1.963 (18)	2.851 (2)	151 (2)
O1—H1A...O10 <sup>iii</sup>	0.76 (3)	1.85 (3)	2.597 (2)	173 (3)
O1—H1B...O12	0.76 (4)	1.94 (4)	2.693 (2)	171 (4)
O2—H2A...O8 <sup>iv</sup>	0.81 (4)	1.93 (4)	2.7343 (19)	176 (4)
O2—H2B...O11 <sup>iii</sup>	0.77 (3)	1.94 (3)	2.7137 (18)	179 (3)
O3—H3A...O6 <sup>v</sup>	0.75 (3)	1.95 (3)	2.6812 (19)	166 (3)
O3—H3B...O9 <sup>v</sup>	0.80 (4)	1.82 (4)	2.6105 (18)	169 (4)
O4—H4A...O9 <sup>vi</sup>	0.82 (4)	1.87 (4)	2.691 (2)	174 (3)
O4—H4B...O10 <sup>vi</sup>	0.72 (3)	1.99 (3)	2.7122 (19)	178 (3)
O5—H5A...O11 <sup>vii</sup>	0.84 (3)	1.95 (3)	2.7835 (19)	171 (3)
O5—H5B...O12 <sup>viii</sup>	0.62 (5)	2.20 (5)	2.813 (2)	168 (6)

Symmetry codes: (i)  $-1-x, -y, 1-z$ ; (ii)  $x-1, y, z$ ; (iii)  $-1-x, 1-y, -z$ ; (iv)  $-1-x, 1-y, 1-z$ ; (v)  $-x, -y, 1-z$ ; (vi)  $1+x, y-1, z$ ; (vii)  $1+x, y, z$ ; (viii)  $-x, -y, -z$ .

In the final calculations, restraints were applied to the parameters of the two O12 water molecule H atoms (O—H 0.97 Å and H—O—H 109.5°), but other H atoms were refined freely.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD-4 EXPRESS*. Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP-3* (Farrugia, 1997). Software used to prepare material for publication: *CIFWRITE* (Muir & Pauson, 1996).

We wish to thank the EPSRC for an equipment grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1282). Services for accessing these data are described at the back of the journal.

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## catena-Poly[[[bis(1,3-diaminopropane-*N,N'*)nickel(II)]- $\mu$ -(4,4'-bipyridine-*N:N'*)]diperchlorate]

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(Received 15 April 1998; accepted 2 September 1998)

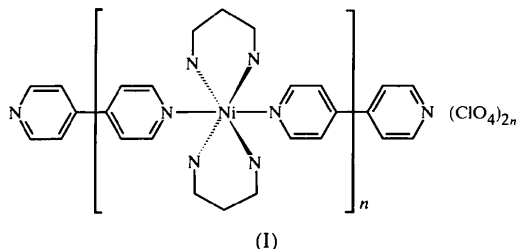
## Abstract

In the structure of the title polymer,  $\{[\text{Ni}(\text{C}_3\text{H}_8\text{N}_2)_2(\text{C}_{10}\text{H}_8\text{N}_2)](\text{ClO}_4)_2\}_n$ , the metal atom is in a slightly distorted octahedral NiN<sub>6</sub> geometry surrounded by four N atoms of two 1,3-diaminopropane ligands in equatorial positions [2.131 (2) and 2.145 (2) Å] and two N atoms from 4,4'-bipyridine ligands in axial sites [2.165 (3) and 2.205 (3) Å]. The 4,4'-bipyridine ligands bridge neighbouring Ni<sup>II</sup> atoms to form polymeric chains in the lattice.

## Comment

Coordination polymers using 4,4'-bipyridine as bridging ligands have been of special interest in recent years. These compounds may exhibit interesting topological structures (Gable *et al.*, 1990; MacGillovray *et al.*, 1994), and clathrates of the cavity structures have possible applications in catalysis and separation (Fujita *et al.*, 1994). Dinuclear and polynuclear metal complexes with 4,4'-bipyridine bridges are also of magnetic interest (Julve *et al.*, 1987; Slangan *et al.*, 1993; Xu & Su, 1990). We have recently prepared some interesting one- and two-dimensional coordination polymers using 4,4'-bipyridine as bridging ligands (Chen *et al.*, 1996; Tong, Cai *et al.*, 1998; Tong, Chen *et al.*, 1998; Tong, Ye *et al.*, 1998). We describe herein the crystal structure of another one-dimensional coordination polymer using

4,4'-bipyridine as a bridging ligand, namely, *catenapoly*[[[bis(1,3-diaminopropane-*N,N'*)nickel(II)]- $\mu$ -(4,4'-bipyridine-*N:N'*)] diperchlorate], (I).



The complex consists of discrete cationic polymeric bis(1,3-diaminopropane)(4,4'-bipyridine)nickel(II) chains and perchlorate anions. The Ni<sup>II</sup> atom, located on a twofold crystallographic axis, is octahedrally coordinated, being surrounded by two N atoms from two 4,4'-bipyridine ligands and by two N atoms each from two 1,3-diaminopropane ligands; the most distorted angle is N3—Ni1—N2 at 92.76(5)°. The Ni—N bond lengths are similar to those [2.104(2) and 2.149(2) Å] found in the cobalt(II) and zinc(II) complexes of 4,4'-bipyridine (Tong, Cai *et al.*, 1998; Tong, Ye *et al.*, 1998), but shorter than those [2.35(2) Å] found in a nickel(II) complex of 4,4'-bipyridine (Stephens & Vagg, 1980). The 4,4'-bipyridine ligands bridge the neighbouring Ni<sup>II</sup> atoms into linear [Ni(4,4'-bipyridine)(1,3-diaminopropane)<sub>2</sub>]<sub>n</sub> chains [N1—Ni1—N2<sup>i</sup> = 180°; symmetry code: (i)  $x, y-1, z$ ] running along the *b* axis, with the adjacent Ni<sup>II</sup> atoms separated by 11.493(6) Å. The pyridyl rings in each 4,4'-bipyridine ligand are not coplanar and the dihedral angle between them is 23.7(1)°, which is commonly seen when the 4,4'-bipyridine ligand has no significant intermolecular interactions with other molecules (Chen *et al.*, 1996; Tong, Cai *et al.*, 1998; Tong, Chen *et al.*, 1998; Tong, Ye *et al.*, 1998).

Finally, it is noteworthy that hydrogen bonding between the amine and perchlorate groups may play a role in consolidating the structure. Each amine N atom donates two weak hydrogen bonds to two perchlorate O

atoms [N3...O4<sup>iv</sup> 3.042(4) and N3...O1<sup>v</sup> 3.118(4), and N4...O1<sup>vi</sup> 3.230(4) and N4...O2<sup>vii</sup> 3.183(4) Å; symmetry codes: (iv)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$ ; (v)  $x, -y, z + \frac{1}{2}$ ; (vi)  $-x, -y, -z$ ; (vii)  $x - \frac{1}{2}, y + \frac{1}{2}, z$ ]. These hydrogen bonds extend the polymeric chains into two-dimensional networks, as shown in Fig. 2.

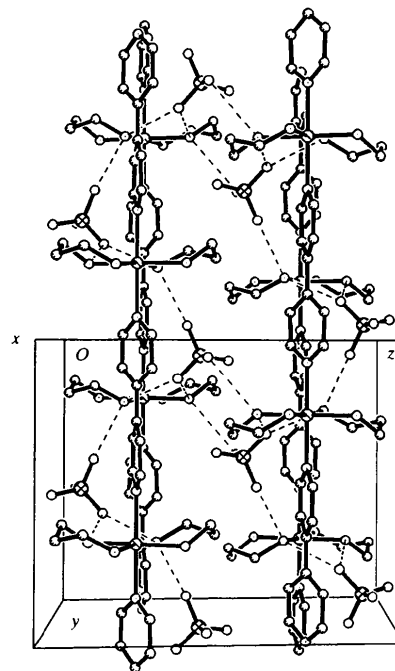


Fig. 2. The crystal packing of (I).

## Experimental

1,3-Diaminopropane (2.0 mmol) was dissolved in water (2 ml) and the solution mixed with an aqueous solution containing Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (1.0 mmol). The mixture was heated to 333 K and stirred briefly for 5 min. An ethanolic solution of 4,4'-bipyridine (1.0 mmol) was then added, followed by an aqueous solution of NaClO<sub>4</sub> (1.0 mmol). Purple block-shaped crystals were deposited after a few days.

### Crystal data

[Ni(C<sub>3</sub>H<sub>10</sub>N<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)]-(ClO<sub>4</sub>)<sub>2</sub>

*M<sub>r</sub>* = 562.05

Monoclinic

*C*2/*c*

*a* = 14.348(6) Å

*b* = 11.493(6) Å

*c* = 13.936(6) Å

β = 95.43(4)°

*V* = 2287.8(18) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.632 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 7–16°

μ = 1.137 mm<sup>-1</sup>

*T* = 293(2) K

Polyhedral

0.60 × 0.58 × 0.40 mm

Purple

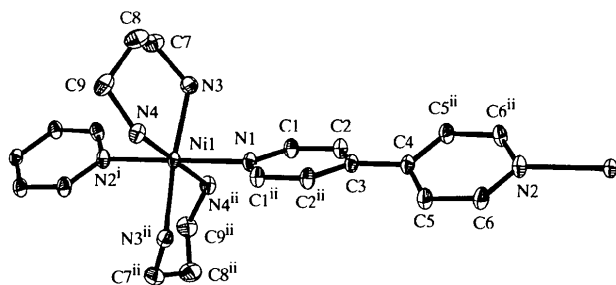


Fig. 1. ORTEP-3 (Farrugia, 1997) plot (35% probability displacement ellipsoids) of a fragment of the coordination polymer. Symmetry codes are as in Table 2.

**Data collection**

Siemens R3m diffractometer  
 $\omega$  scans  
 Absorption correction:  
 $\psi$  scan (North *et al.*,  
 1968)  
 $T_{\min} = 0.52$ ,  $T_{\max} = 0.64$   
 2722 measured reflections  
 2628 independent reflections  
 2236 reflections with  
 $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$   
 $\theta_{\text{max}} = 27.54^\circ$   
 $h = 0 \rightarrow 18$   
 $k = 0 \rightarrow 14$   
 $l = -18 \rightarrow 17$   
 2 standard reflections  
 every 120 reflections  
 intensity decay: 1.0%

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.035$   
 $wR(F^2) = 0.091$   
 $S = 1.07$   
 2625 reflections  
 152 parameters  
 H atoms riding  
 $w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 2.9507P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.42 \text{ e } \text{Å}^{-3}$   
 Extinction correction: none  
 Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U^{ij}a'_i a'_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
Ni1	0	0.22794 (3)	1/4	0.02252 (12)
N1	0	0.4198 (2)	1/4	0.0277 (5)
N2	0	1.0395 (2)	1/4	0.0292 (6)
C1	0.0709 (2)	0.4813 (2)	0.2948 (2)	0.0292 (5)
C2	0.0735 (2)	0.6016 (2)	0.2968 (2)	0.0298 (5)
C3	0	0.6650 (2)	1/4	0.0256 (6)
C4	0	0.7944 (2)	1/4	0.0267 (6)
C5	-0.0828 (2)	0.8575 (2)	0.2369 (2)	0.0313 (5)
C6	-0.0794 (2)	0.9782 (2)	0.2358 (2)	0.0321 (5)
N3	0.13429 (14)	0.2369 (2)	0.19467 (14)	0.0319 (4)
N4	-0.06264 (14)	0.2286 (2)	0.10513 (13)	0.0317 (4)
C7	0.1539 (2)	0.1753 (2)	0.1048 (2)	0.0422 (6)
C8	0.0814 (2)	0.2036 (3)	0.0214 (2)	0.0487 (7)
C9	-0.0168 (2)	0.1595 (2)	0.0323 (2)	0.0432 (6)
Cl1	0.30158 (4)	-0.05766 (5)	-0.06716 (4)	0.0389 (2)
O1	0.2729 (2)	-0.1459 (2)	-0.1360 (2)	0.0670 (6)
O2	0.40058 (15)	-0.0623 (2)	-0.0436 (2)	0.0651 (6)
O3	0.2552 (2)	-0.0769 (3)	0.0175 (2)	0.0799 (8)
O4	0.2762 (2)	0.0530 (2)	-0.1075 (2)	0.0861 (9)

Table 2. Selected geometric parameters ( $\text{Å}$ ,  $^\circ$ )

Ni1—N4	2.131 (2)	Ni1—N2 <sup>i</sup>	2.165 (3)
Ni1—N3	2.145 (2)	Ni1—N1	2.205 (3)
N4—Ni1—N3	88.39 (8)	C1—N1—Ni1	121.76 (13)
N4 <sup>ii</sup> —Ni1—N3	91.59 (8)	C6—N2—Ni1 <sup>iii</sup>	121.78 (13)
N4—Ni1—N1	89.79 (5)	C7—N3—Ni1	121.8 (2)
N3—Ni1—N1	87.24 (5)	C9—N4—Ni1	118.0 (2)

Symmetry codes: (i)  $x, y - 1, z$ ; (ii)  $-x, y, \frac{1}{2} - z$ ; (iii)  $x, 1 + y, z$ .

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP-3* (Farrugia, 1997). Software used to prepare material for publication: *SHELXL93*.

We acknowledge financial support by the NSFC (29625102). We are also indebted to the Chemistry

Department of The Chinese University of Hong Kong for donation of the R3m diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1408). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 182–186

## Tricarbonyl[1,1,1-tris(dimethylphosphinomethyl)ethane]chromium(0), -molybdenum(0) and -tungsten(0) complexes

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(Received 6 May 1998; accepted 28 September 1998)

**Abstract**

The structures of chromium(0)–, molybdenum(0)– and tungsten(0)–tricarbonyl complexes containing the tripodal tridentate phosphine ligand 1,1,1-tris(dimethyl-