

**catena-Poly[bis[(1,10-phenanthroline)nickel(II)]- $\mu$ -3,6-dicarboxycyclohexane-1,2,4,5-tetracarboxylato]**

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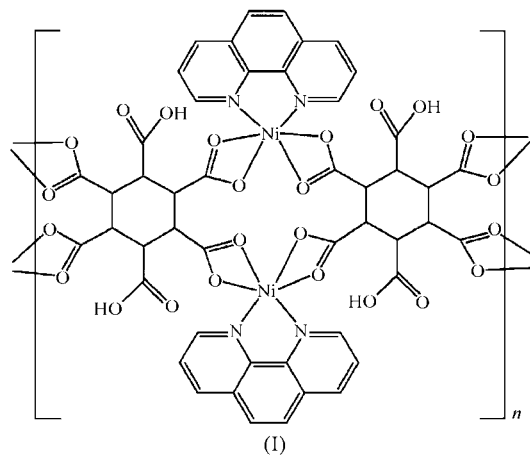
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In the title compound,  $[\text{Ni}_2(\text{C}_{12}\text{H}_8\text{O}_{12})(\text{C}_{12}\text{H}_8\text{N}_2)_2]_n$ , the 3,6-dicarboxycyclohexane-1,2,4,5-tetracarboxylate ( $\text{H}_2\text{chhc}^{4-}$ ) anion has crystallographically imposed  $C_2$  symmetry and bridges the six-coordinate Ni atoms to generate polymeric  $\infty[\text{Ni}_2(\text{H}_2\text{chhc})_{2/2}(\text{C}_{12}\text{H}_8\text{N}_2)_2]$  chains extending in the [010] direction. The coordination polymer chains are linked into a three-dimensional framework by  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

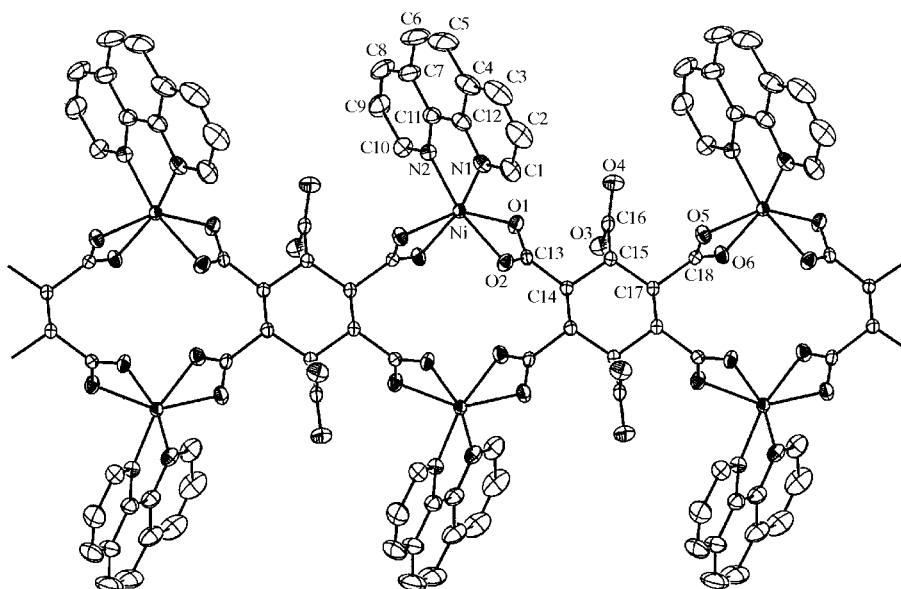
**Comment**

In recent years, metal-organic coordination polymers have been attracting more and more attention owing to their potential application in many fields (Moulton & Zaworotko,

2001; MacGillivray *et al.*, 1998). It is well known that aromatic carboxylic acids, such as terephthalic acid (Chen *et al.*, 2003), benzene-1,3,5-tricarboxylic acid (Chui *et al.*, 1999), benzene-1,2,4,5-tetracarboxylic acid (Murugavel *et al.*, 2002) and mellitic acid (Ranganathan *et al.*, 2001), exhibit a variety

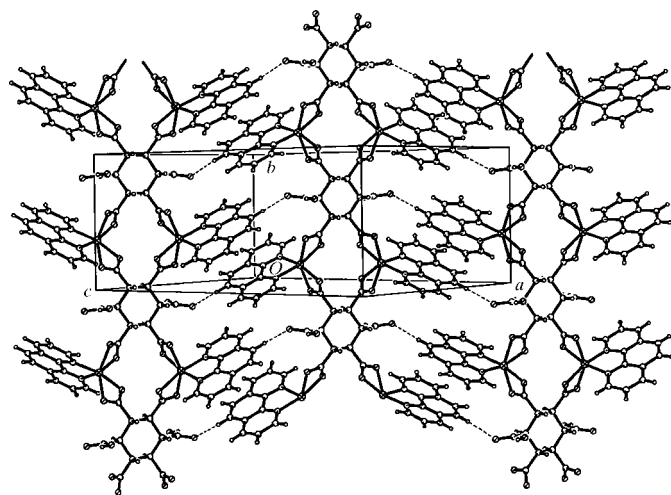


of bridging modes and coordination fashions and have been widely used to construct coordination polymers with specific network topologies and interesting properties. Because of their remarkable conformational flexibility, the aliphatic carboxylic acids have also aroused much attention (Zheng *et al.*, 2004; Zheng & Kong, 2002). Comparably few naphthenic polycarboxylic acids have been explored as ligands for the construction of coordination polymers (Bi *et al.*, 2003; Myunghyun *et al.*, 2003; Kil & Myunghyun, 2001). In the present contribution, we report a new nickel coordination polymer,  $[\text{Ni}_2(\text{H}_2\text{chhc})(\text{C}_{12}\text{H}_8\text{N}_2)_2]$ , (I), resulting from self-assembly of  $\text{Ni}^{2+}$  ions, phenanthroline (phen) and a tetra-anion derived from cyclohexane-1,2,3,4,5,6-hexacarboxylic acid ( $\text{H}_6\text{chhc}$ ).

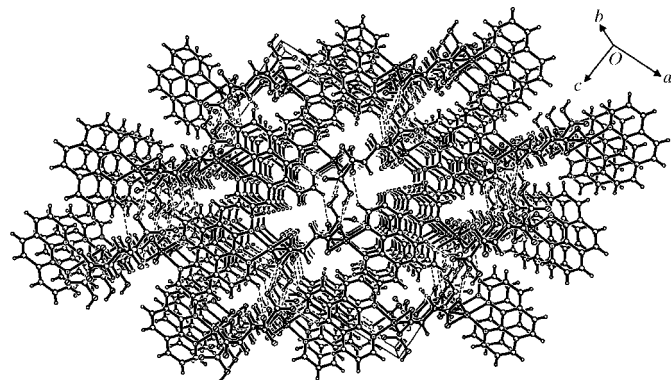
**Figure 1**

A fragment of the one-dimensional  $\infty[\text{Ni}_2(\text{H}_2\text{chhc})_{2/2}(\text{phen})_2]$  polymeric chain. Displacement ellipsoids are drawn at the 45% probability level. H atoms have been omitted.

The asymmetric unit of the title compound consists of one Ni<sup>2+</sup> cation, one phen ligand and one-half of a H<sub>2</sub>chhc<sup>4-</sup> anion lying across a twofold rotation axis. The Ni atoms are each in a distorted octahedral environment defined by four O atoms of two carboxylate groups from different H<sub>2</sub>chhc<sup>4-</sup> anions and two N atoms of one phen ligand. The Ni–N bond distances are 2.050 (2) and 2.056 (2) Å, and the Ni–O bond distances range from 2.084 (2) to 2.118 (2) Å. Obviously, such bonding values fall in the normal region (Zheng *et al.*, 2002). The H<sub>2</sub>chhc<sup>4-</sup> anions display a stable chair conformation with the two opposite carboxyl groups and four carboxylate groups oriented equatorially, similar to those reported in the literature (Bi *et al.*, 2003; Myunghyun *et al.*, 2003; Kil & Myunghyun, 2001). Each carboxylate group of the H<sub>2</sub>chhc<sup>4-</sup> anion chelates one Ni atom; as a result, the H<sub>2</sub>chhc<sup>4-</sup> anions are each coordinated to four [Ni(phen)]<sup>2+</sup> units, leading to polymeric chains formulated as  $[\text{Ni}_2(\text{H}_2\text{chhc})_{2/2}(\text{C}_{12}\text{H}_8\text{N}_2)_2]_n$  running along the [010] direction with the phen ligands *exo*-orientated, as shown in Fig. 1. On the basis of the interchain C–H···O hydrogen bonds (Table 1), these chains are assembled into layers parallel to (101) (Fig. 2). The layers are



**Figure 2**  
The two-dimensional supramolecular layer in (I), parallel to (101). Hydrogen bonds are indicated by dashed lines.



**Figure 3**  
The packing of (I). Hydrogen bonds are indicated by dashed lines.

further connected to form a three-dimensional framework (Fig. 3) *via* strong interlayer O–H···O hydrogen bonds and weak interchain C–H···O hydrogen bonds. The H<sub>2</sub>chhc<sup>4-</sup> anions exhibit normal C–O bond distances (Kil & Myunghyun, 2001).

### Experimental

The precipitate Ni(OH)<sub>2</sub>·xH<sub>2</sub>O was deposited from dropwise addition of 1 M NaOH (3.0 ml) to a stirred aqueous solution of NiCl<sub>2</sub>·6H<sub>2</sub>O (0.24 g, 1.0 mmol, in 7.0 ml H<sub>2</sub>O), and was separated by centrifugation and washed with distilled water several times until no detectable Cl<sup>-</sup> anions remained in the supernatant. Subsequently, the precipitate was added to a stirred solution of H<sub>6</sub>chhc (0.17 g, 0.50 mmol) and 1,10-phenanthroline monohydrate (0.20 g, 1.0 mmol) in water (20.0 ml). The resulting mixture was stirred for *ca* 30 min and then filtered. The green filtrate was allowed to stand at room temperature. Slow evaporation for several days afforded green plate-like crystals (yield *ca* 20%, based on the initial NiCl<sub>2</sub>·6H<sub>2</sub>O input).

#### Crystal data

[Ni<sub>2</sub>(C<sub>12</sub>H<sub>8</sub>O<sub>12</sub>)(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]  
M<sub>r</sub> = 411.01  
Monoclinic, C2/c  
a = 22.018 (4) Å  
b = 8.9600 (18) Å  
c = 16.371 (3) Å  
β = 92.49 (3)°  
V = 3226.6 (11) Å<sup>3</sup>

Z = 8  
D<sub>x</sub> = 1.692 Mg m<sup>-3</sup>  
Mo Kα radiation  
μ = 1.24 mm<sup>-1</sup>  
T = 298 (2) K  
Plate, green  
0.31 × 0.22 × 0.11 mm

#### Data collection

Bruker P4 diffractometer  
ω/2θ scans  
Absorption correction: ψ scan  
(North *et al.*, 1968)  
T<sub>min</sub> = 0.727, T<sub>max</sub> = 0.872  
4519 measured reflections  
3715 independent reflections

2959 reflections with I > 2σ(I)  
R<sub>int</sub> = 0.030  
θ<sub>max</sub> = 27.5°  
3 standard reflections  
every 97 reflections  
intensity decay: none

#### Refinement

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.036  
wR(F<sup>2</sup>) = 0.087  
S = 1.01  
3715 reflections  
250 parameters  
H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.0381P)<sup>2</sup> + 3.2579P]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> = 0.001  
Δρ<sub>max</sub> = 0.34 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.29 e Å<sup>-3</sup>  
Extinction correction: SHELXL97  
Extinction coefficient: 0.38

**Table 1**  
Hydrogen-bond geometry (Å, °).

D–H···A	D–H	H···A	D···A	D–H···A
C6–H6···O4 <sup>i</sup>	0.93	2.48	3.210 (4)	136
O3–H3A···O1 <sup>ii</sup>	0.86 (4)	1.82 (3)	2.628 (2)	155 (4)
C10–H10···O6 <sup>ii</sup>	0.93	2.42	3.139 (3)	134

Symmetry codes: (i)  $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 2$ ; (ii)  $-x + 2, -y, -z + 2$ .

Compound (I) is monoclinic; space group C2/c was assumed and confirmed by the analysis. H atoms attached to C atoms were placed in calculated positions and refined using a riding model [C–H = 0.93 or 0.98 Å, and U<sub>iso</sub>(H) = 1.2U<sub>eq</sub>(C)], while the H atom on atom O3 was observed in a difference Fourier map and was refined freely [O–H = 0.86 (4) Å].

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3041). Services for accessing these data are described at the back of the journal.

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