metal-organic papers

# Poly[nickel(II)-*μ*-4,4′-bipyridine-*μ*-terephthalato]

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4,4′-Bipyridine(terephthalato)nickel(II), [Ni($C_8H_4O_4$)($C_{10}H_8$-N_2)], exists as poly[μ_2-terephthalato-μ_4-terephthalato-bis-[(μ_2-4,4′-bipyridine)nickel(II)]]. Two independent terephthalato groups both occupy positions of $2/m$ symmetry; one of these groups acts as a μ_4 bridge and coordinates four different Ni atoms, one with each of its O atoms. The other one acts as a μ_2-bridge and serves as a bidentate chelate for two neighboring metal atoms. The Ni atom lies in a special position of m site symmetry.

Comment

4,4′-Bipyridine(terephthalato)nickel(II), [Ni($C_8H_4O_4$)($C_{10}H_8$-N_2)], exists as poly[μ_2-terephthalato-μ_4-terephthalato-bis-[(μ_2-4,4′-bipyridine)nickel(II)]]. Two independent terephthalato groups both occupy positions of $2/m$ symmetry; one of these groups acts as a μ_4 bridge and coordinates four different Ni atoms, one with each of its O atoms. The other one acts as a μ_2-bridge and serves as a bidentate chelate for two neighboring metal atoms (Fig. 1). Consequently, the terephthalate–nickel framework forms infinite two-dimensional layers parallel to the *ab* plane of the crystal. The layers are linked into a three-dimensional network through the μ_2-bridging 4,4′-bipyridine ligands occupying a special position across the mirror plane. The compound is isostructural with the published cobalt analog, whose detailed description (Tao *et al.*, 2000) also applies to the title compound.

(I)
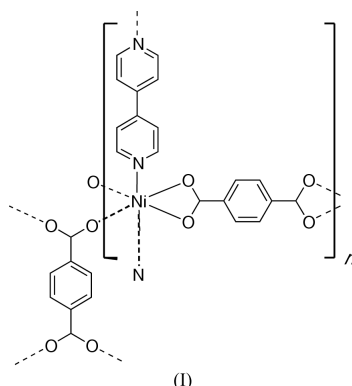
Experimental

The compound was synthesized hydrothermally from nickel nitrate hexahydrate (0.29 g, 1 mmol), terephthalic acid (0.17 g, 1 mmol), 4,4′-bipyridine dihydrochloride (0.23 g, 1 mmol) and sodium hydroxide (0.16 g, 4 mmol) in water (18 ml). The mixture was placed in a 20 ml Teflon-lined stainless-steel vessel, which was heated at 493 K for 100 h. The vessel was cooled to room temperature at a rate of 6 K h^{-1}. The product was isolated in 25% yield.

Crystal data

[Ni(C$_8$H$_4$O$_4$)(C$_{10}$H$_8$N$_2$)]
$M_r = 379.01$
Monoclinic, $C2/m$
$a = 16.610\ (3)$ Å
$b = 10.222\ (2)$ Å
$c = 11.237\ (2)$ Å
$\beta = 119.22\ (3)°$
$V = 1665.1\ (5)$ Å^3
$Z = 4$

$D_x = 1.512$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25
 reflections
$\theta = 14.0–16.0°$
$\mu = 1.19$ mm^{-1}
$T = 298\ (2)$ K
Block, green
$0.18 \times 0.18 \times 0.11$ mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
ω scans
Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{min} = 0.696$, $T_{max} = 0.877$
1790 measured reflections
1728 independent reflections
1312 reflections with $I > 2\sigma(I)$

$R_{int} = 0.038$
$\theta_{max} = 26.0°$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 12$
$l = -13 \rightarrow 12$
3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.135$
$S = 1.03$
1728 reflections
128 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2$
 $+ 1.5719P]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.999 (3)	Ni1—N1	2.076 (5)
Ni1—O2	2.149 (3)		
O1—Ni1—O1^i	111.4 (2)	O2—Ni1—O2^i	61.3 (2)
O1—Ni1—O2	154.7 (1)	O2—Ni1—N1	89.5 (2)
O1—Ni1—O2^i	93.5 (1)	O2—Ni1—N2^{ii}	87.3 (2)
O1—Ni1—N1	93.4 (1)	N1—Ni1—N2^{ii}	176.3 (2)
O1—Ni1—N2^{ii}	88.6 (1)		

Symmetry codes: (i) $x, 1 - y, z$; (ii) $x, y, 1 + z$.

The aromatic portion of one of the terephthalate groups (at the Wyckoff 2*d* site of 2/*m* symmetry), made up of atoms C5, C6 and C7, showed large displacement parameters for atoms C6 and C7. H atoms were positioned geometrically (C—H = 0.93 Å) and included in the subsequent refinement in the riding motion approximation with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1988); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms, 1997);
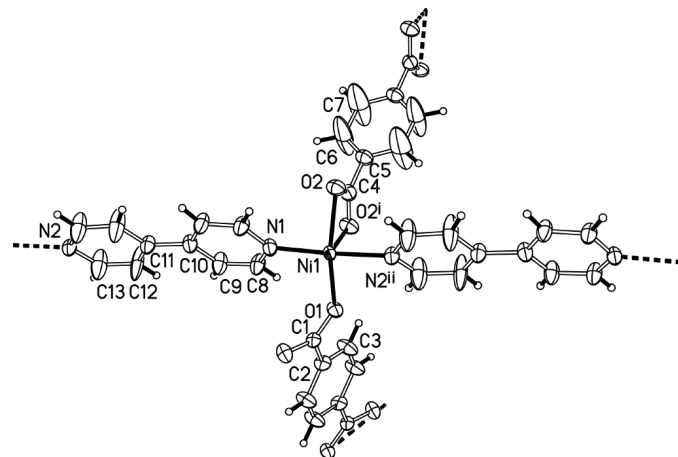

Figure 1
*ORTEP*II (Johnson, 1976) plot of a fragment of the structure of (I), with ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry codes: (i) $x, 1 - y, z$; (ii) $x, y, 1 + z$.]

program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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