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Aqua(4,4-bipyridine)phthalatonickel(II) trihydrate

Shi-Yao Yang,[a] La-Sheng Long,[a] Rong-Bin Huang,[a] Lan-Sun Zheng[a] and Seik Weng Ng[b]*

[a]State Key Laboratory for Physical Chemistry of Solid Surfaces, Xiamen University, Xiamen 361005, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

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Key indicators

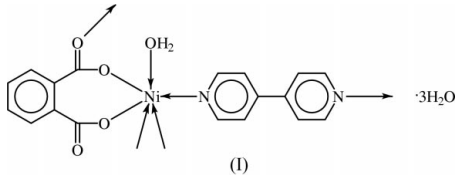
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.010 Å
R factor = 0.060
wR factor = 0.153
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The $(H_2O)(C_{10}H_8N_2)Ni(C_8H_4O_4)$ portion of the title compound, $[Ni(C_8H_4O_4)(C_{10}H_8N_2)(H_2O)]\cdot 3H_2O$, adopts a layer architecture in which the dianionic entity chelates to the Ni atom [Ni—O = 2.053 (5) and 2.056 (4) Å, and O—Ni—O = 91.5 (2)°]. The two chelating O atoms, a water molecule [Ni—O = 2.083 (5) Å], the carboxyl O atom from an adjacent dianion, and the N atoms of two 4,4′-bipyridine heterocycles comprise the octahedron around the Ni atom. Adjacent layers are linked into a network motif by hydrogen bonds.

Comment

In our studies on complexes of transition metal benzenedicarboxylates, we have documented the 4,4′-bipyridine adduct of nickel(II) terephthalate (Yang *et al.*, 2003). The use of phthalic anhydride as the reagent in place of terephthalic acid in the synthesis afforded the analogous nickel phthalate, but the compound crystallizes with four water molecules, one of which is coordinated to the Ni atom (Fig. 1 and Table 1). In the $(H_2O)(C_{10}H_8N_2)Ni(C_8H_4O_4)$ unit, the phthalate dianion chelates to the Ni atom [Ni—O = 2.053 (5) and 2.056 (4) Å, and O—Ni—O = 91.5 (2)°]. The two chelating O atoms, a water molecule [Ni—O = 2.083 (5) Å], the carboxyl O atom from an adjacent dianion, and the N atoms of two 4,4′-bipyridine heterocycles comprise the octahedron around the Ni atom. The dianion links adjacent Ni atoms into a chain, and adjacent chains are held together through the heterocyclic spacer into layers (Fig. 2). Adjacent layers are linked into a network motif by hydrogen bonds (Table 2). The dianion is only monodentate in the 2,2′-bipyridine adduct of nickel phthalate, which exists as a triaqua monohydrate (Poleti *et al.*, 1990).



(I)

Experimental

The compound was synthesized hydrothermally from nickel nitrate hexahydrate (0.29 g, 1 mmol), phthalic anhydride (0.16 g, 1 mmol), 4,4′-bipyridine (0.16 g, 1 mmol) and sodium hydroxide (0.08 g, 2 mmol) in water (18 ml). The mixture was placed in a 20 ml Teflon-lined stainless-steel vessel, which was heated to 443 K for 120 h. The vessel was cooled to room temperature at 5 K h^{-1}. The compound separated from the solution as fine crystals.

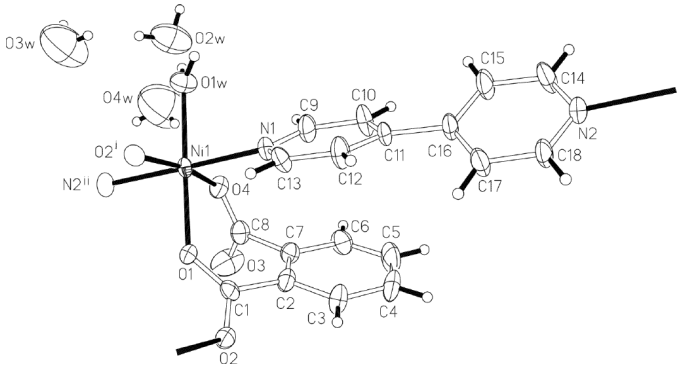


Figure 1
*ORTEP*II (Johnson, 1976) plot of a fragment of the structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii. [Symmetry codes: (i) x, $1-y$, z; (ii) x, y, $1+z$.]

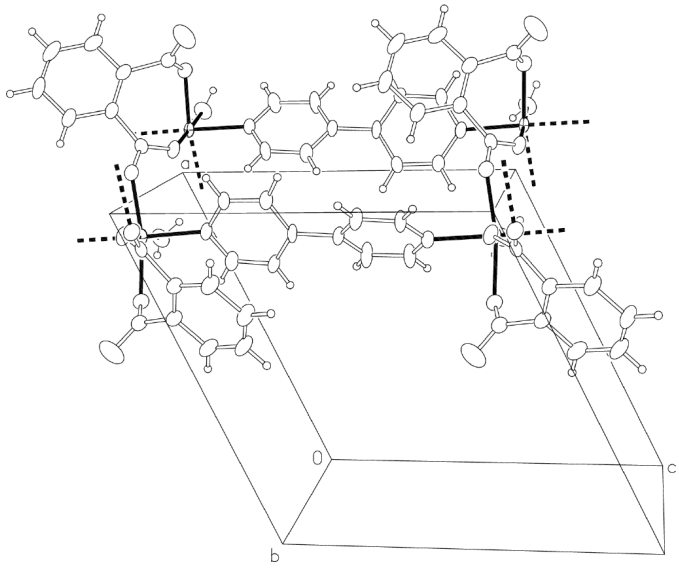


Figure 2
Layer structure of the title compound. The uncoordinated water molecules are not shown.

Crystal data

$[Ni(C_8H_4O_4)(C_{10}H_8N_2)(H_2O)]\cdot 3H_2O$	$D_x = 1.578$ Mg m^{-3}
$M_r = 451.07$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 25 reflections
$a = 11.134$ (2) Å	$\theta = 12.0$–$14.0°$
$b = 8.379$ (2) Å	$\mu = 1.07$ mm^{-1}
$c = 11.265$ (2) Å	$T = 298$ (2) K
$\beta = 115.39$ (3)°	Plate, green
$V = 949.4$ (3) Å^3	0.32 × 0.29 × 0.06 mm
$Z = 2$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.059$
ω scans	$\theta_{max} = 30.0°$
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 15$
$T_{min} = 0.455$, $T_{max} = 0.939$	$k = 0 \rightarrow 11$
3070 measured reflections	$l = -15 \rightarrow 14$
2936 independent reflections	2 standard reflections
2171 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0777P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.060$	$(\Delta/\sigma)_{max} = 0.03$
$wR(F^2) = 0.153$	$\Delta\rho_{max} = 0.62$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.64$ e Å^{-3}
2936 reflections	Absolute structure: Flack & Schwarzenbach (1988)
262 parameters	Flack parameter = 0.00 (3)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.053 (5)	Ni1—O1*w*	2.083 (5)
Ni1—O2^{i}	2.065 (4)	Ni1—N1	2.093 (4)
Ni1—O4	2.056 (4)	Ni1—N2^{ii}	2.112 (4)
O1—Ni1—O2^{i}	84.3 (2)	O2^{i}—Ni1—N2^{ii}	85.0 (2)
O1—Ni1—O4	91.5 (2)	O4—Ni1—O1*w*	88.4 (2)
O1—Ni1—O1*w*	178.9 (2)	O4—Ni1—N1	96.0 (2)
O1—Ni1—N1	91.7 (2)	O4—Ni1—N2^{ii}	85.5 (2)
O1—Ni1—N2^{ii}	88.5 (3)	O1*w*—Ni1—N1	87.2 (2)
O2^{i}—Ni1—O4	169.7 (2)	O1*w*—Ni1—N2^{ii}	92.6 (3)
O2^{i}—Ni1—O1*w*	96.1 (2)	N1—Ni1—N2^{ii}	178.5 (2)
O2^{i}—Ni1—N1	93.6 (2)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*w*—H1*w*2···O2*w*	0.85	1.95	2.778 (9)	162
O1*w*—H1*w*1···O2^{iii}	0.86	2.01	2.807 (7)	154
O2*w*—H2*w*2···O3^{iii}	0.87	2.09	2.88 (1)	151
O3*w*—H3*w*1···O4*w*^{iv}	0.88	1.80	2.64 (2)	158
O4*w*—H4*w*1···O2*w*	0.89	2.40	2.73 (2)	102
O4*w*—H4*w*1···O3*w*	0.89	2.38	3.14 (2)	142

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

H atoms were positioned geometrically (C—H = 0.93 Å) and were included in the subsequent refinement in the riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$. The water H atoms were found by using the *HYDROGEN* (Nardelli, 1999) option in the *WinGX* suite (Farrugia, 1999). These were not refined; their displacement parameters were set at 0.05 Å^2.

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