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Polymeric tetraaqua(1,2,4,5-benzenetetracarboxylato)(pyrazine)dinickel(II) dihydrate

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

[a]Department of Chemistry, 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

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.065
wR factor = 0.142
Data-to-parameter ratio = 13.2

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

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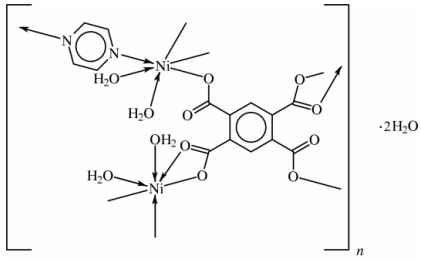
The Ni atom in the title compound, $\{[Ni_2(C_{10}H_2O_8)(C_4H_4N_2)(H_2O)_4]\cdot 2H_2O\}_n$, exists in an octahedral NO_5Ni coordination environment that is defined by the two O atoms of a chelating carboxyl group, the O atom of a monodentate carboxyl group belonging to another carboxylato tetraanion, two water molecules and the N atom of the *N*-heterocycle. The tetracarboxylato anion and the pyrazine molecule are each located on a center of inversion. The bonding pattern leads to the formation of a layer structure; the layers are linked into a three-dimensional network by extensive hydrogen bonds involving the water molecules and the O atoms of the anionic unit.

Comment

A number of adducts of nickel benzenepolycarboxylates with *N*-heterocycles have been reported (Li *et al.*, 2003; Yuan *et al.*, 2001). To this number has been added the 4,4′-bipyridine adduct of nickel terephthalate (Yang *et al.*, 2003*a*) and of nickel phthalate (Yang *et al.*, 2003*b*). The present pyrazine adduct, (I), of dinickel 1,2,4,5-benzenetetracarboxylate exists as the dihydrate (Fig. 1).



(I)

The Ni atom in (I) exists in an octahedral NO_5Ni environment that is defined by the two O atoms of a chelating $-CO_2$ group, the O atom of a monodentate CO_2 group of another tetraanion, two water molecules and the N atom of pyrazine (Table 1). The tetracarboxylate anion and the pyrazine molecule are each located on a center of inversion. The bonding pattern leads to the formation of layers; the layers are linked into a three-dimensional network by hydrogen bonds involving the water molecules and the O atoms of the anionic unit (Table 2).

Experimental

1,2,4,5-Benzenetetracarboxylic acid anhydride (pyromellitic anhydride, 0.22 g, 1 mmol) was dissolved in water (15 ml) containing sodium hydroxide (0.16 g, 4 mmol). Nickel dinitrate hexahydrate

(0.58 g, 2 mmol) and pyrazine (0.16 g, 2 mmol) dissolved in water (3 ml) were then added. The mixture was placed in a 20 ml Teflon-lined stainless-steel bomb. The bomb was heated at 453 K for 100 h. Crystals separated from the solution when the bomb was cooled down at a rate of 5 K h^{-1}.

Crystal data

$[Ni_2(C_{10}H_2O_8)(C_4H_4N_2)(H_2O)_4]\cdot 2H_2O$
$M_r = 555.72$
Triclinic, $P\overline{1}$
$a = 7.2005$ (5) Å
$b = 8.0322$ (5) Å
$c = 9.3735$ (6) Å
$\alpha = 96.175$ (1)°
$\beta = 101.872$ (1)°
$\gamma = 116.245$ (1)°
$V = 463.50$ (5) Å^3
$Z = 1$
$D_x = 1.991$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2804 reflections
$\theta = 2.3$–28.3°
$\mu = 2.12$ mm^{-1}
$T = 298$ (2) K
Irregular block, green
0.14 × 0.13 × 0.04 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.616$, $T_{max} = 0.919$
5415 measured reflections
2152 independent reflections
2055 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 28.3°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.142$
$S = 1.25$
2152 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.259P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.95$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.126 (3)	Ni1—O1*w*	2.049 (3)
Ni1—O2	2.123 (3)	Ni1—O2*w*	2.025 (4)
Ni1—O3^{i}	2.014 (3)	Ni1—N1	2.068 (4)
O1—Ni1—O2	61.7 (1)	O2—Ni1—N1	85.8 (1)
O1—Ni1—O3^{i}	84.9 (1)	O3^{i}—Ni1—O1*w*	93.2 (1)
O1—Ni1—O1*w*	103.2 (1)	O3^{i}—Ni1—O2*w*	89.8 (1)
O1—Ni1—O2*w*	162.8 (1)	O3^{i}—Ni1—N1	173.6 (1)
O1—Ni1—N1	91.5 (1)	O1*w*—Ni1—O2*w*	93.4 (1)
O2—Ni1—O3^{i}	87.8 (1)	O1*w*—Ni1—N1	92.8 (1)
O2—Ni1—O1*w*	164.8 (1)	O2*w*—Ni1—N1	92.2 (2)
O2—Ni1—O2*w*	101.8 (1)		

Symmetry code: (i) $1-x, 1-y, 2-z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*2···O1^{ii}	0.85 (1)	1.87 (3)	2.660 (5)	155 (5)
O1*w*—H1*w*1···O4^{i}	0.84 (1)	1.82 (2)	2.621 (5)	157 (5)
O2*w*—H2*w*2···O1*w*^{iii}	0.85 (1)	1.96 (1)	2.807 (5)	176 (6)
O2*w*—H2*w*1···O3*w*^{iv}	0.85 (1)	1.90 (1)	2.744 (5)	176 (6)
O3*w*—H3*w*1···O2	0.85 (1)	2.26 (3)	3.047 (5)	155 (6)
O3*w*—H3*w*2···O3^{v}	0.85 (1)	2.02 (1)	2.866 (5)	174 (7)

Symmetry codes: (i) $1-x, 1-y, 2-z$; (ii) $1-x, 1-y, 1-z$; (iii) $1-x, 2-y, 1-z$; (iv) $x-1, y, z$; (v) $1+x, 1+y, z$.

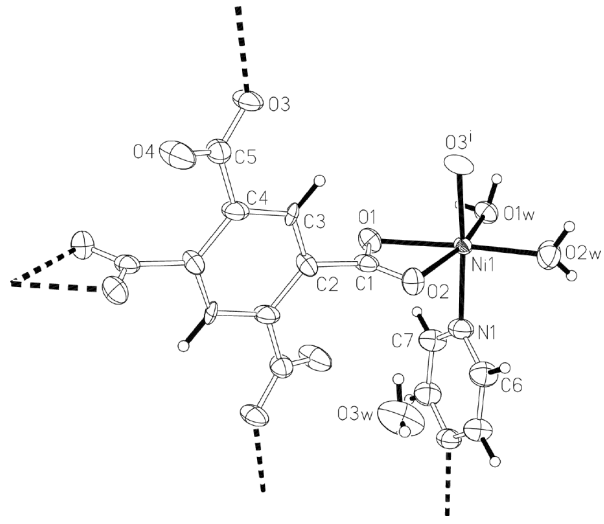


Figure 1
*ORTEP*II (Johnson, 1976) plot of a segment of the title structure, with displacement ellipsoids at the 75% probability level. [Symmetry code: (i) $1-x, 1-y, 2-z$.]

The use of too small a value for the θ-dependent absorption correction in *SADABS* (Sheldrick, 1996) led to non-positive definite displacement parameters for several atoms, and a compromise value of 0.5 was used, for which parameters of only one atom (C3) were non-positive definite. The displacement parameter of this atom was restrained to be close to isotropic. The aromatic H atoms were positioned geometrically (C—H = 0.93 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C). The water H atoms were located and refined with restraints of O—H = 0.85 (1) Å, H···H = 1.39 (1) Å and U_{iso}(H) = 1.2U_{eq}(O).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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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