

Pentaaqua(pyridine-3,5-dicarboxylato- $\kappa N$ )-nickel(II) dihydrateXiang-Pei Zhang,<sup>a</sup> Guang Yang<sup>a</sup>  
and Seik Weng Ng<sup>b\*</sup><sup>a</sup>Department of Chemistry, Zhengzhou University, Zhengzhou 450052, People's Republic of China, and <sup>b</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

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

## Key indicators

Single-crystal X-ray study  
 $T = 295$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.022  
 $wR$  factor = 0.066  
Data-to-parameter ratio = 9.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

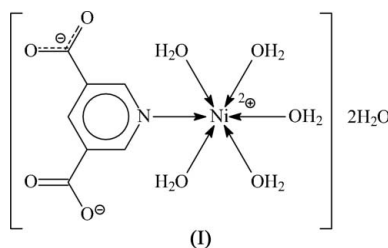
In the title compound,  $[\text{Ni}(\text{C}_7\text{H}_3\text{NO}_4)(\text{H}_2\text{O})_5]\cdot 2\text{H}_2\text{O}$ , the pyridine-3,5-dicarboxylate dianion bonds through its N atom to Ni, the octahedral geometry of the latter being completed by five water molecules. Adjacent complex molecules are linked through the two non-coordinated water molecules, forming a three-dimensional, tightly held, hydrogen-bonded network.

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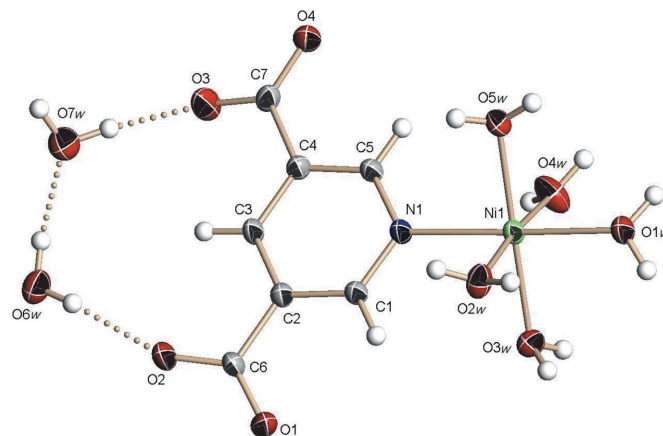
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## Comment

Two metal derivatives of 3,5-pyridinedicarboxylic acid have been crystallographically characterized, namely the diaqua-copper(II) complex (Lu & Schauss, 2001) and the isostructural cobalt(II) analog (Whitfield *et al.*, 2001). In these compounds, the dianion binds to the metal through both the pyridyl N atom and the carboxylate O atoms, leading to the formation of a layer motif. The title nickel derivative, (I), exists as a pentaaqua dihydrate in which the dianion binds to the metal only through its N atom (Table 1, Fig. 1).



Of the two negatively charged  $-\text{CO}_2$  units in the ligand, one (C7) displays statistically indistinguishable C–O distances (implying that the charge is delocalized). However, the



**Figure 1**  
View of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Dashed lines indicate hydrogen bonds.

different C–O distances in the other (C8) imply a localized-charge carboxylate unit. In this unit, the O atom of the shorter C–O bond accepts two O–H···O hydrogen bonds whereas the O atom of the longer C–O bond accepts three (Table 2). Overall, the hydrogen bonding in (I) gives rise to the formation of a tightly held, three-dimensional network.

Experimental

An aqueous solution (5 ml) of nickel(II) chloride hexahydrate (12 mg, 0.05 mmol) was mixed with a warm ethanol solution (5 ml) of 3,5-pyridinedicarboxylic acid (8 mg, 0.05 mmol). The solution was set aside for several days and light-green crystals of (I) were obtained in about 20% yield.

Crystal data

[Ni(C<sub>7</sub>H<sub>3</sub>NO<sub>4</sub>)(H<sub>2</sub>O)<sub>5</sub>].2H<sub>2</sub>O  
*M<sub>r</sub>* = 349.93  
 Orthorhombic, *Pna*2<sub>1</sub>  
*a* = 15.743 (1) Å  
*b* = 11.697 (1) Å  
*c* = 6.995 (1) Å  
*V* = 1288.0 (3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.805 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 923 reflections  
 $\theta$  = 3.1–27.5°  
 $\mu$  = 1.57 mm<sup>-1</sup>  
*T* = 295 (2) K  
 Block, green  
 0.18 × 0.09 × 0.07 mm

Data collection

Bruker SMART 1000 CCD diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.778, *T<sub>max</sub>* = 0.898  
 7706 measured reflections

2296 independent reflections  
 2157 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.018  
 $\theta_{max}$  = 27.0°  
*h* = -18 → 19  
*k* = -14 → 14  
*l* = -5 → 8

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.022  
*wR* (*F*<sup>2</sup>) = 0.066  
*S* = 1.06  
 2296 reflections  
 238 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.0644P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.25 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.50 \text{ e \AA}^{-3}$   
 Absolute structure: Flack (1983),  
 797 Friedel pairs  
 Flack parameter: 0.03 (1)

Table 1

Selected bond lengths (Å).

Ni1–O1 <sub>w</sub>	2.073 (2)	Ni1–N1	2.101 (2)
Ni1–O2 <sub>w</sub>	2.042 (2)	C6–O1	1.247 (2)
Ni1–O3 <sub>w</sub>	2.102 (2)	C6–O2	1.250 (2)
Ni1–O4 <sub>w</sub>	2.016 (2)	C7–O3	1.238 (2)
Ni1–O5 <sub>w</sub>	2.077 (2)	C7–O4	1.263 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1 <sub>w</sub> –H1 <sub>w</sub> 1···O1 <sup>i</sup>	0.85 (1)	1.96 (1)	2.807 (2)	172 (4)
O1 <sub>w</sub> –H1 <sub>w</sub> 2···O4 <sup>ii</sup>	0.85 (1)	2.01 (1)	2.851 (2)	174 (4)
O2 <sub>w</sub> –H2 <sub>w</sub> 1···O4 <sup>iii</sup>	0.85 (1)	1.88 (1)	2.717 (2)	165 (3)
O2 <sub>w</sub> –H2 <sub>w</sub> 2···O7 <sub>w</sub> <sup>iv</sup>	0.84 (1)	1.96 (1)	2.766 (3)	161 (2)
O3 <sub>w</sub> –H3 <sub>w</sub> 1···O2 <sup>i</sup>	0.86 (1)	1.80 (1)	2.657 (2)	174 (2)
O3 <sub>w</sub> –H3 <sub>w</sub> 2···O4 <sup>v</sup>	0.86 (1)	2.12 (2)	2.902 (3)	150 (2)
O4 <sub>w</sub> –H4 <sub>w</sub> 1···O3 <sup>ii</sup>	0.84 (1)	1.89 (1)	2.697 (2)	160 (3)
O4 <sub>w</sub> –H4 <sub>w</sub> 2···O6 <sub>w</sub> <sup>vi</sup>	0.83 (1)	1.91 (1)	2.733 (3)	168 (2)
O5 <sub>w</sub> –H5 <sub>w</sub> 1···O1 <sup>vii</sup>	0.85 (1)	1.88 (1)	2.722 (2)	171 (3)
O5 <sub>w</sub> –H5 <sub>w</sub> 2···O6 <sub>w</sub> <sup>iv</sup>	0.84 (1)	1.90 (2)	2.711 (3)	160 (3)
O6 <sub>w</sub> –H6 <sub>w</sub> 1···O2	0.83 (1)	1.91 (2)	2.687 (2)	157 (3)
O6 <sub>w</sub> –H6 <sub>w</sub> 2···O7 <sub>w</sub>	0.84 (1)	1.87 (1)	2.702 (2)	171 (4)
O7 <sub>w</sub> –H7 <sub>w</sub> 1···O3	0.85 (1)	1.82 (1)	2.660 (2)	174 (3)
O7 <sub>w</sub> –H7 <sub>w</sub> 2···O3 <sub>w</sub> <sup>iv</sup>	0.85 (1)	2.31 (2)	2.982 (3)	136 (2)

Symmetry codes: (i)  $x + \frac{1}{2}, -y + \frac{5}{2}, z$ ; (ii)  $x + \frac{1}{2}, -y + \frac{3}{2}, z$ ; (iii)  $-x + \frac{3}{2}, y + \frac{1}{2}, z - \frac{1}{2}$ ; (iv)  $-x + 1, -y + 2, z - \frac{1}{2}$ ; (v)  $-x + \frac{3}{2}, y + \frac{1}{2}, z + \frac{1}{2}$ ; (vi)  $-x + 1, -y + 2, z + \frac{1}{2}$ ; (vii)  $-x + \frac{3}{2}, y - \frac{1}{2}, z - \frac{1}{2}$ .

The arrangement of the complex molecules is pseudo-centrosymmetric. However, the non-coordinated water molecules cannot be successfully modelled in a higher symmetry space group. The carbon-bound H atoms were placed at calculated positions (C–H = 0.93 Å) and refined as riding, with *U<sub>iso</sub>*(H) = 1.2*U<sub>eq</sub>*(C). The oxygen-bound H atoms were located in difference maps and were refined with distance restraints of O–H = 0.85 (1) Å and H···H = 1.39 (1) Å; their *U<sub>iso</sub>* values were freely refined.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97*.

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