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Key indicators

Single-crystal X-ray study
 $T = 294\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$
Disorder in solvent or counterion
 R factor = 0.068
 wR factor = 0.157
Data-to-parameter ratio = 12.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Triaqua(4-hydroxypyridine-2,6-dicarboxylato)-
nickel(II) sesquihydrate

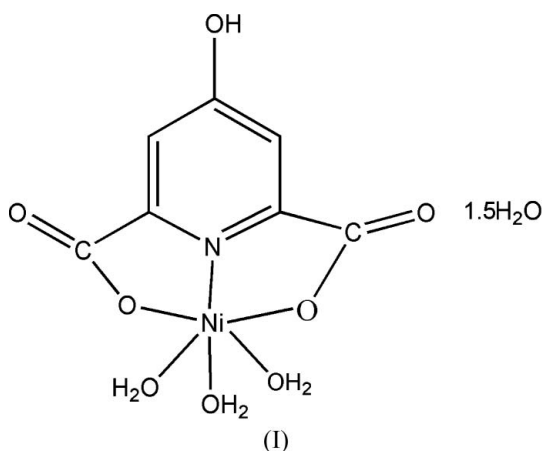
The title compound, $[\text{Ni}(\text{chedam})(\text{H}_2\text{O})_3] \cdot 1.5\text{H}_2\text{O}$ [H_2chedam = 4-hydroxypyridine-2,6-dicarboxylic acid, $\text{C}_7\text{H}_5\text{NO}_5$], consists of one nickel cation, one chedam ligand, three coordinated water molecules and 1.5 uncoordinated water molecules. The nickel(II) cation is six-coordinate, with a distorted octahedral geometry.

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Comment

The carboxylate group is an important class of ligand in inorganic and bioinorganic chemistry. Metal complexes containing carboxylic acids are well known and the publication of many structurally characterized examples of this class of compound has demonstrated the versatility of the carboxylate group as an inner-sphere ligand (Mehrotra & Bohra, 1983). However, the coordination chemistry of 4-hydroxypyridine-2,6-dicarboxylic acid (H_2chedam) as one of many dicarboxylic acids is far less developed and, indeed, structural information for this class of complex is relatively scarce (Gao *et al.*, 2006).



The asymmetric unit of the title complex, (I), is composed of one nickel(II) cation, one chedam ligand, three aqua ligands and 1.5 uncoordinated water molecules, one water molecule being disordered over a twofold rotation axis (Fig. 1). The nickel(II) cation is six-coordinate with a distorted octahedral geometry. The equatorial sites of the nickel cation(II) are occupied by one O atom from one aqua ligand, and two O atoms and one N atom from one chelating ligand. The axial sites are occupied by two aqua ligands. The three coordinated water molecules and the hydroxy atom from the ligand generate intermolecular hydrogen bonds, resulting in a three-dimensional supramolecular network (Fig. 2 and Table 1).

Experimental

[Ni(chedam)(H₂O)₃].1.5H₂O was prepared under hydrothermal conditions. [Ni(SO₄)₂].6H₂O (0.2 mmol) and H₂chedam (0.2 mmol) were placed in a 25 ml reaction vessel. The reaction vessel was then sealed and subsequently placed in an oven for 140 h at 433 K. Prism-shaped crystals of (I) were obtained and washed with ethanol.

Crystal data

[Ni(C₇H₃NO₅)(H₂O)₃].1.5H₂O
M_r = 320.89
 Monoclinic, *C*2/*c*
a = 14.879 (2) Å
b = 6.9505 (14) Å
c = 22.567 (3) Å
 β = 90.474 (9)°
V = 2333.6 (7) Å³
Z = 8
D_x = 1.827 Mg m⁻³
 Mo *K*α radiation
 μ = 1.71 mm⁻¹
T = 294 (2) K
 Prism, green
 0.22 × 0.18 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.698, *T_{max}* = 0.821
 5364 measured reflections
 2069 independent reflections
 1700 reflections with *I* > 2σ(*I*)
R_{int} = 0.052
 θ_{max} = 25.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.068
wR(*F*²) = 0.157
S = 1.23
 2069 reflections
 172 parameters
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0372P)^2 + 28.3869P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.61 e Å⁻³
 Δρ_{min} = -0.71 e Å⁻³

Table 1

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>
O5–H5...O1 ⁱ	0.85	1.82	2.670 (6)	174
O6–H6A...O2 ⁱⁱ	0.85	1.94	2.738 (6)	156
O7–H7A...O2 ⁱⁱⁱ	0.85	2.25	2.878 (6)	131
O7–H7A...O5 ^{iv}	0.85	2.40	3.121 (6)	143
O8–H8A...O3 ^v	0.85	1.94	2.784 (6)	173

Symmetry codes: (i) *x* – ½, *y* – ½, *z*; (ii) –*x* + 1, –*y*, –*z* + 1; (iii) –*x* + 1, –*y* + 1, –*z* + 1; (iv) –*x* + ½, –*y* + ½, –*z* + 1; (v) –*x* + 1, *y*, –*z* + ½.

All H atoms were positioned geometrically and treated as riding atoms with C–H = 0.93 Å, O–H = 0.85 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C,O).

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; 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.

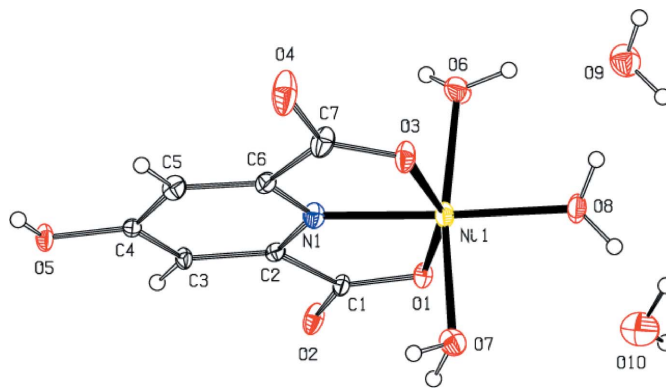


Figure 1

The asymmetric unit and atom-labelling scheme of (I). Displacement ellipsoids are drawn at the 30% probability level.

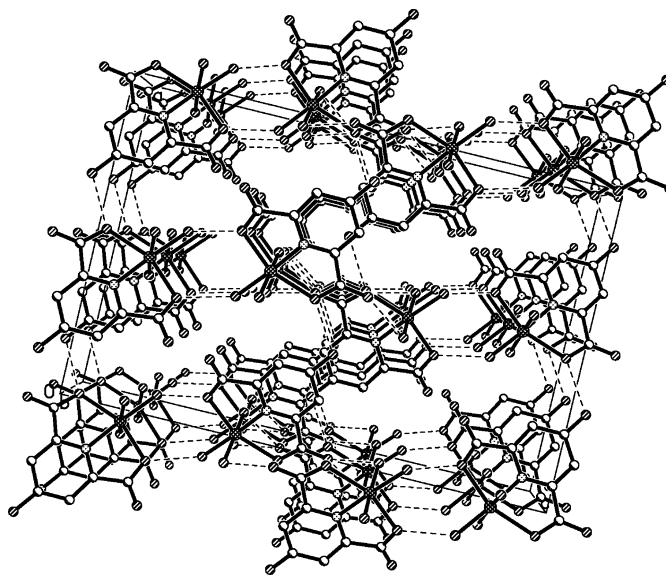


Figure 2

The three-dimensional supramolecular structure of (I). Dashed lines indicate hydrogen bonds and H atoms have been omitted.

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