Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis{3-[(*E*)-2-(aminocarbonyl)hydrazonomethyl]pyridine-κ*N*}tetraaquanickel(II) diacetate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.077
Data-to-parameter ratio = 13.8

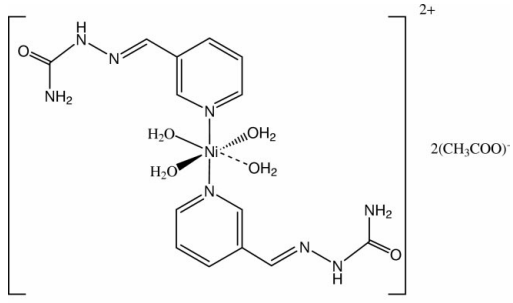
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 May 2004
Accepted 21 May 2004
Online 29 May 2004

The title compound, $[Ni(C_{14}H_{16}N_8O_2)_2(H_2O)_4](CH_3COO)_2$, is isostructural with the published Co^{II} complex. The Ni atom occupies an inversion centre and is octahedrally coordinated by four aqua ligands and two molecules of the Schiff base.

Comment

The title compound, (I) (Fig. 1), is isostructural with the cobalt(II) complex (Chen *et al.*, 2004), and it consists of discrete $[Ni(H\text{-}Pysc)_2(H_2O)_4]^{2+}$ cations and acetate counterions. The Ni atom lies on an inversion centre and has a slightly distorted octahedral coordination formed by two monodentate H-Pysc and four aqua ligands (Table 1). The two neutral semicarbazone ligands are planar and only utilize their pyridyl N atoms for metal coordination. The packing is stabilized by an extensive three-dimensional network (Fig. 2 and Table 2) of hydrogen bonds (O—H···O and N—H···O) involving the aqua ligands, Schiff bases and acetate counterions.



(I)

Experimental

The Schiff base ligand pyridine-3-carbaldehyde semicarbazone (H-Pysc) was synthesized by the condensation of semicarbazide with pyridine-3-carbaldehyde in a 1:1 molar ratio in methanol at 333 K for 1 h. $Ni(CH_3COO)_2.4H_2O$ (0.2 mmol) and H-Pysc (0.4 mmol) were placed in a thick Pyrex tube (*ca* 20 cm long). After addition of ethanol (1.5 ml) and water (0.5 ml), the tube was cooled with liquid N_2, evacuated under vacuum and sealed with a torch. The tube was heated at 348 K for 2 d to yield green block-shaped crystals of (I) in about 50% yield.

Crystal data

$[Ni(C_{14}H_{16}N_8O_2)_2(H_2O)_4](C_2H_3O_2)_2$
M_r = 577.21
Triclinic, $P\overline{1}$
a = 7.8498 (19) Å
b = 8.836 (2) Å
c = 10.2711 (17) Å
α = 65.822 (15)°
β = 69.678 (16)°
γ = 82.14 (2)°
V = 609.4 (2) Å^3
Z = 1
D_x = 1.573 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2857 reflections
θ = 3.3–27.5°
μ = 0.87 mm^{-1}
T = 193 (2) K
Block, green
0.56 × 0.21 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.643, T_{max} = 0.918
6785 measured reflections
2731 independent reflections
2567 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.077
S = 1.12
2731 reflections
198 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0234P)^2 + 0.4761P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O2	2.0689 (15)	Ni1—N1	2.1098 (17)
Ni1—O3	2.0770 (15)		
O2—Ni1—O3	90.14 (6)	O3—Ni1—N1	89.83 (6)
O2—Ni1—O3^{i}	89.86 (6)	O3—Ni1—N1^{i}	90.17 (6)
O2—Ni1—N1	90.34 (6)	C6—N2—N3	116.60 (17)
O2—Ni1—N1^{i}	89.66 (6)	N2—N3—C7	119.62 (17)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2b···O5^{ii}	0.84 (3)	1.98 (3)	2.789 (2)	160 (3)
N3—H3b···O4^{iii}	0.79 (2)	2.27 (2)	2.966 (2)	147 (2)
O3—H3c···O4	0.85 (3)	1.81 (3)	2.647 (2)	166 (3)
N4—H4a···O5	0.86 (3)	2.33 (3)	3.089 (3)	147 (2)

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

H atoms bound to C atoms were positioned geometrically and included in the refinement in the riding-model approximation [C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}]. Water H atoms were located in difference maps and refined, subject to an O—H distance restraint of 0.85 (1) Å. H atoms bound to N atoms were located in a difference map and refined subject to an N—H distance restaint of 0.86 (1) Å.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear* (Rigaku, 1999); data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXTL*.

The authors thank the Youth Science Foundation of Guangxi, the Natural Science Foundation of Guangxi, and the Project of One Hundred Persons Plan of Guangxi Universities of the People's Republic of China, as well as the Teaching and Research Award Program for Outstanding Young Teachers in Higher Education Institutions of the Chinese Ministry of Education.

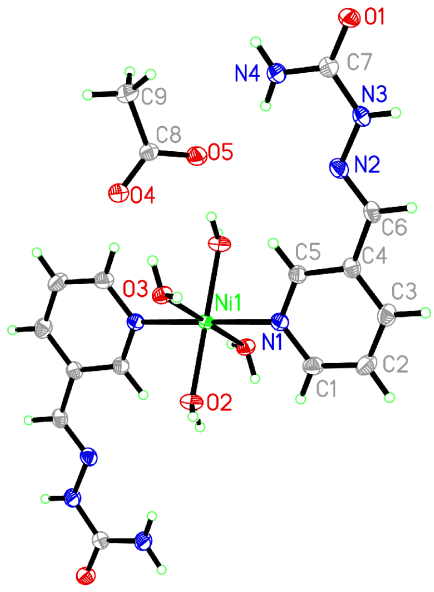


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-labelling scheme. H atoms are represented by small spheres of arbitrary radii.

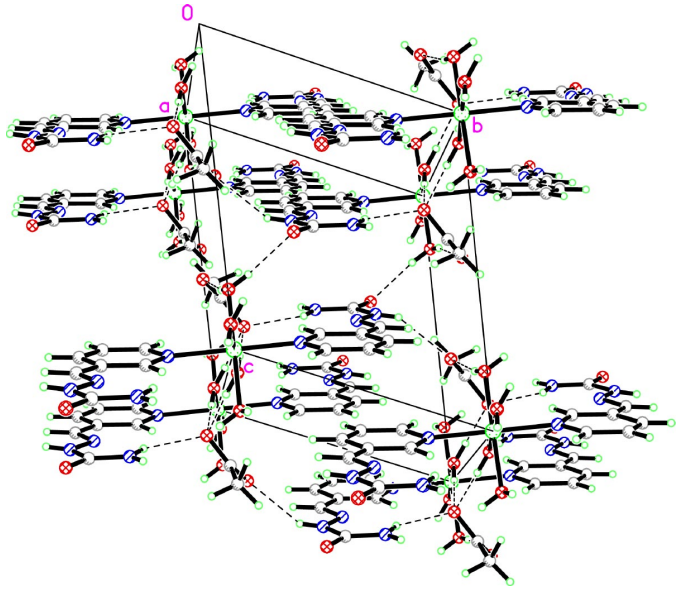


Figure 2
Packing diagram of (I), with hydrogen bonds indicated by dashed lines.

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