

trans-Diaquabis(2-pyridylethanol-*N,O*)-nickel(II) disaccharinateSevim Hamamci,^a Veysel T. Yilmaz^{a*} and Carsten Thöne^b^aDepartment of Chemistry, Faculty of Arts and Science, Ondokuz Mayıs University, 55139 Kurupelit Samsun, Turkey, and ^bInstitut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany

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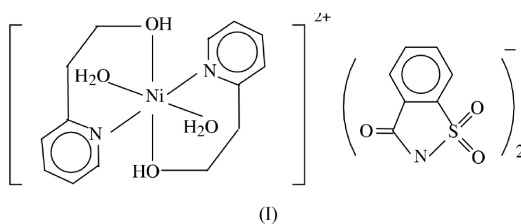
Key indicators

Single-crystal X-ray study
 $T = 133\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.032
 wR factor = 0.096
Data-to-parameter ratio = 19.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title complex, $[\text{Ni}(\text{pyet})_2(\text{H}_2\text{O})_2](\text{sac})_2$ (sac^- is the saccharinate anion, $\text{C}_7\text{H}_4\text{NO}_3\text{S}^-$, and pyet is 2-pyridylethanol, $\text{C}_7\text{H}_9\text{NO}$), is built of discrete sac^- and $[\text{Ni}(\text{H}_2\text{O})_2(\text{pyet})_2]^{2+}$ ions. The Co^{2+} ion sits on a centre of symmetry and exhibits a distorted octahedral geometry, formed by two bidentate pyet and two water molecules. The crystal structure is stabilized by hydrogen-bonding and weak aromatic π - π -stacking interactions between the planar sac^- ions, forming a three-dimensional network.

Comment

The structure of the title complex, (I), is shown in Fig. 1. The nickel(II) complex is isostructural with the corresponding cobalt(II) complex and the chemical unit consists of a complex cation, $[\text{Ni}(\text{H}_2\text{O})_2(\text{pyet})_2]^{2+}$, and two saccharinate (sac^-) anions. The crystal structure is centrosymmetric and the Ni^{II} ion, located at the centre of symmetry, is coordinated by a pair of neutral 2-pyridylethanol (pyet) ligands and two water molecules, resulting in distorted octahedral geometry. The sac^- ions are not in the primary coordination sphere, being separate counter-ions. Two pyet ligands are chelated to the Ni^{II} ion through the N and hydroxyl O atoms, forming two symmetrically-related six-membered chelate rings. The two pyet ligands form the equatorial plane of the coordination octahedron, while two water molecules occupy the axial positions with a *trans* configuration.



The crystal packing is shown in Fig. 2. Packing of molecules in (I) is very similar to that in the corresponding cobalt(II) complex (Yilmaz *et al.*, 2002). The discrete cations and anions are linked by hydrogen bonds, as well as by π - π interactions. The parallel stacking of pairs of the sac^- ions results in weak π - π interactions ($> 4.0\text{ \AA}$; the planes are defined by atoms C11-C16/S1/N1/C21/O21 and Ni/N2/C31-C36 for sac^- and pyet , respectively). The H atoms of the water molecules (O4) and hydroxyl group (O3) of pyet form $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds with the sulfonyl (O1) and carbonyl (O21) O atoms of neighboring sac^- anions. Additionally, two weak $\text{C}-\text{H}\cdots\text{O}$ interactions between H atoms of the py ring and the carbonyl and sulfonyl O atoms of the adjacent sac^- ions occur. The ions are held together by hydrogen bonds and weak π - π interactions, creating a three-dimensional network.

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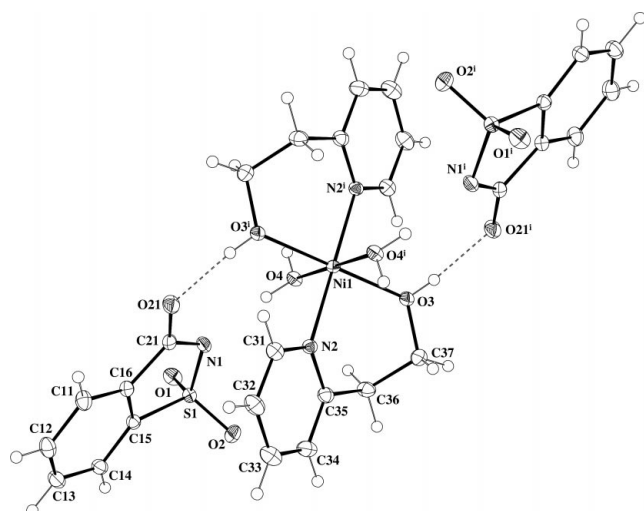


Figure 1
A molecular view of $[\text{Ni}(\text{H}_2\text{O})_2(\text{pyet})_2](\text{sac})_2$ (40% displacement ellipsoids). [Symmetry code: (i) $-x, 1-y, -z$.]

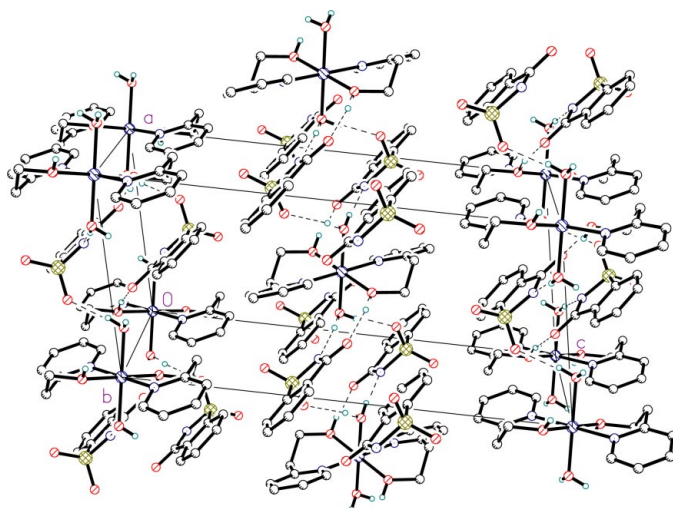


Figure 2
Packing diagram of $[\text{Ni}(\text{H}_2\text{O})_2(\text{pyet})_2](\text{sac})_2$, showing the hydrogen-bonding scheme.

Experimental

Previously prepared $[\text{Ni}(\text{sac})_2(\text{H}_2\text{O})_4] \cdot 2\text{H}_2\text{O}$ (1.0 mmol, 0.53 g; Haider *et al.*, 1985) was dissolved in methanol (30 ml) at 303 K with stirring. Pyet (0.25 g, 2.0 mmol) was then added to the solution dropwise. The resulting solution was allowed to evaporate slowly for crystallization at room temperature. Suitable single crystals obtained within a week were collected by suction filtration, washed with acetone and dried in air.

Crystal data

$[\text{Ni}(\text{C}_7\text{H}_9\text{NO})_2(\text{H}_2\text{O})_2] \cdot (\text{C}_7\text{H}_4\text{NO}_3\text{S})_2$
 $M_r = 705.39$
 Monoclinic, $P2_1/n$
 $a = 8.8009$ (6) Å
 $b = 8.7664$ (6) Å
 $c = 19.0840$ (14) Å
 $\beta = 100.262$ (3)°
 $V = 1448.82$ (18) Å³
 $Z = 2$

$D_x = 1.617$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5714 reflections
 $\theta = 2.2\text{--}30.5^\circ$
 $\mu = 0.88$ mm⁻¹
 $T = 133$ (2) K
 Irregular, pale blue
 $0.24 \times 0.15 \times 0.13$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
 ω and φ scans
 Absorption correction: multi-scan (SADABS; Bruker, 1998)
 $T_{\min} = 0.826$, $T_{\max} = 0.928$
 25 119 measured reflections

4249 independent reflections
 3604 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\max} = 30.0^\circ$
 $h = -12 \rightarrow 12$
 $k = -12 \rightarrow 12$
 $l = -26 \rightarrow 26$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.096$
 $S = 1.05$
 4249 reflections
 217 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.2009P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.04$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ni—O3	2.0645 (11)	Ni—O4	2.0851 (11)
Ni—N2	2.0729 (12)		
O3—Ni—N2 ⁱ	89.48 (5)	O3—Ni—O4	90.95 (5)
O3—Ni—N2	90.52 (5)	N2 ⁱ —Ni—O4	87.28 (5)
O3 ⁱ —Ni—O4	89.05 (5)	N2—Ni—O4	92.72 (5)

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
O3—H3 ⁱ ⋯O21 ⁱ	0.81 (3)	1.85 (3)	2.6515 (16)	172 (3)
O4—H4A ⁱⁱ ⋯O1 ⁱⁱ	0.76 (2)	2.07 (2)	2.8325 (17)	175 (2)
C32—H32 ⁱⁱⁱ ⋯O21 ⁱⁱⁱ	0.95	2.64	3.357 (2)	133
C33—H33 ^{iv} ⋯O2 ^{iv}	0.95	2.45	3.342 (2)	156

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

H atoms of the hydroxyl group and water molecules were refined freely, while H atoms bonded to carbon were included using a riding model, starting from calculated positions. The maximum electron-density peak was located 0.99 Å from C32.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: XP (Siemens, 1994); software used to prepare material for publication: SHELXL97.

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