

Bis(triethanolamine-*N,O,O'*)nickel(II) bis(saccharinate)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.048
wR factor = 0.143
Data-to-parameter ratio = 16.0

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

The structure of the title compound, $[\text{Ni}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_7\text{H}_4\text{NO}_3\text{S})_2$, consists of a cationic $[\text{Ni}(\text{TEA})_2]^{2+}$ and two anionic $(\text{SAC})^-$ moieties (TEA is triethanolamine and SAC is the saccharinate anion). Two TEA ligands coordinate to the Ni^{II} ion through four O atoms and two N atoms acting as tridentate ligands (*N,O,O'*) in a distorted octahedral environment, with the bond distances [$d(\text{Ni}-\text{O}) = 2.078(2)$ and $2.082(2) \text{ \AA}$, and $d(\text{Ni}-\text{N}) = 2.108(2) \text{ \AA}$]. SAC anions participate in intermolecular hydrogen bonding with the hydroxyl H atoms of the TEA ligands to form a three-dimensional network.

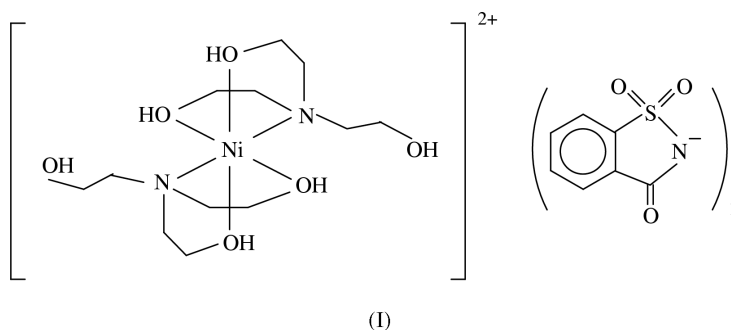
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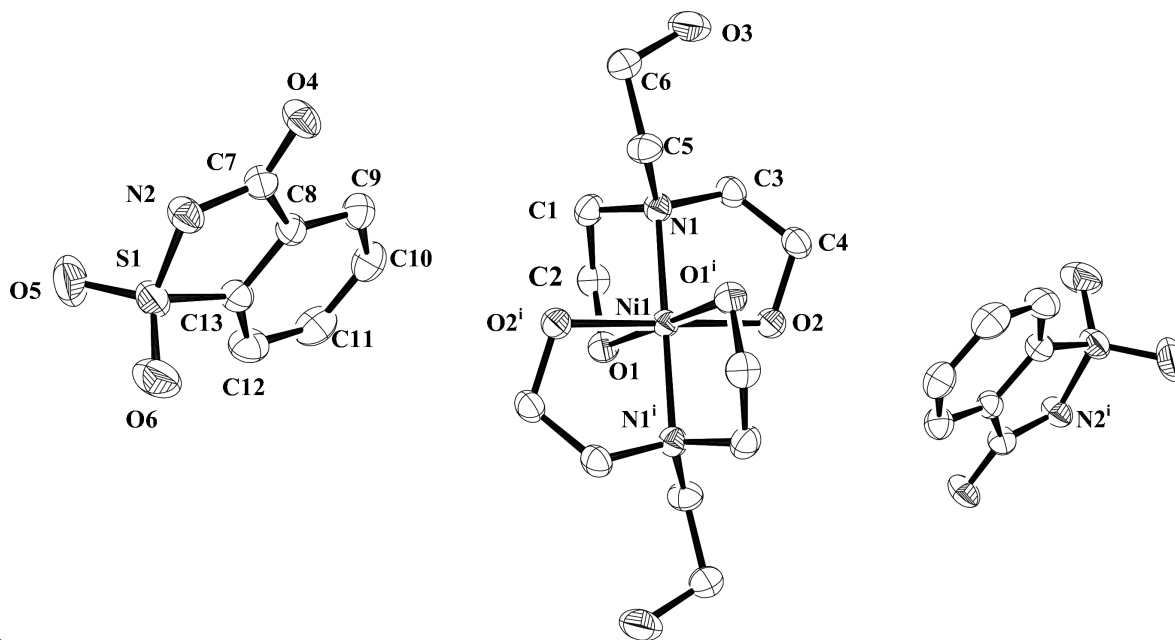
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Comment

In the preparation of metal complexes of ethanolamines with the saccharinate anion (SAC) as a co-ligand, the title complex, (I), was obtained by the reaction of hexaaquabis(saccharinato)nickel(II) with triethanolamine (TEA). The reaction resulted in the replacement of all the ligands around the Ni^{II} ion by the bulky TEA ligands. The title complex contains a cationic $[\text{Ni}(\text{TEA})_2]^{2+}$ and two anionic $(\text{SAC})^-$ moieties.



The cation resides on a crystallographic inversion centre and is formed by the Ni^{II} sitting at the centre and binding two TEA ligands which results in a six coordinate environment around the metal. Each TEA acts as a tridentate ligand through its N and two hydroxyl O atoms, one hydroxyl O atom of the ethanolic groups being non-coordinated (Fig. 1). The Ni–O distances are in the range 2.078(2)–2.082(2) Å and the Ni–N distances are 2.108(2) Å, while the bond angles range from 82.12(9) to 97.88(9)° (Table 1). Both bond distances and angles are comparable with those observed for the Ni complex of TEA with chloride (Icbudak *et al.*, 1995) and acetate (Krabbes *et al.*, 2000). The SAC ligand is essentially planar and intramolecular bond lengths are virtually identical to those found in the free SAC molecule (Okaya, 1969), $[\text{Mn}(\text{phen})_2(\text{H}_2\text{O})_2](\text{SAC})_2 \cdot \text{H}_2\text{O}$ (Jianmin *et al.*, 1993) and

**Figure 1**

The structure of the title complex showing the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x, -y, -z$].

ammonium saccharinate (Ng, 1998). SAC ions are linked by hydrogen bonds to the hydroxyl groups of the TEA ligands in the complex cation. The coordinated hydroxyl groups of the TEA ligands form $O-H \cdots N$ and $O-H \cdots O$ hydrogen bonds with the SAC anions, while the non-coordinated hydroxyl groups form $O-H \cdots O$ hydrogen bonds with the anions. In addition, weak $C-H \cdots O$ hydrogen bonds are observed between the aromatic C9 and C11 atoms of the SAC moiety and the O atoms of neighbouring SAC anions, as well as the non-coordinated hydroxyl group of the TEA ligand (Table 2).

Experimental

Previously prepared $[\text{Ni}(\text{SAC})_2(\text{H}_2\text{O})_4] \cdot 2\text{H}_2\text{O}$ (1.06 g, 2.0 mmol) was dissolved in 50 ml of ethanol at 343 K with stirring and cooled to room temperature. Then TEA (0.60 g, 4.0 mmol) was added to the solution dropwise. The resulting blue solution was left at room temperature until evaporation resulted in the formation of pale blue crystals suitable for X-ray diffraction analysis.

Crystal data

$[\text{Ni}(\text{C}_6\text{H}_{15}\text{NO}_3)_2](\text{C}_7\text{H}_4\text{NO}_3\text{S})_2$
 $M_r = 721.43$
 Monoclinic, $P2_1/c$
 $a = 7.3714$ (4) Å
 $b = 12.9097$ (8) Å
 $c = 15.7403$ (9) Å
 $\beta = 97.732$ (1)°
 $V = 1484.27$ (15) Å³
 $Z = 2$

$D_x = 1.614$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3145 reflections
 $\theta = 2.6$ – 27.5 °
 $\mu = 0.87$ mm⁻¹
 $T = 295$ (2) K
 Diamond, pale blue
 $0.31 \times 0.30 \times 0.11$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1999)
 $T_{\min} = 0.759$, $T_{\max} = 0.909$
 9255 measured reflections

3417 independent reflections
 2396 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$
 $\theta_{\text{max}} = 27.6$ °
 $h = -6 \rightarrow 9$
 $k = -16 \rightarrow 16$
 $l = -15 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.143$
 $S = 0.99$
 3417 reflections
 214 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0907P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.48$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.42$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ni1–O1	2.078 (2)	N1–C3	1.499 (4)
Ni1–O2	2.082 (2)	S1–O6	1.430 (3)
Ni1–N1	2.108 (2)	S1–O5	1.433 (3)
O1–C2	1.435 (4)	S1–N2	1.621 (3)
O2–C4	1.442 (4)	S1–C13	1.761 (3)
O3–C6	1.403 (4)	N2–C7	1.336 (4)
N1–C5	1.483 (4)	O4–C7	1.248 (4)
N1–C1	1.495 (4)		
O1–Ni1–O2 ⁱ	88.44 (9)	O1 ⁱ –Ni1–N1	97.88 (9)
O1 ⁱ –Ni1–O2 ⁱ	91.56 (9)	O2 ⁱ –Ni1–N1	97.07 (9)
O1–Ni1–N1	82.12 (9)	O2–Ni1–N1	82.93 (9)

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

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1–H1O \cdots N2 ⁱ	0.79 (4)	1.93 (4)	2.692 (3)	161 (4)
O2–H2O \cdots O4 ⁱ	0.84 (4)	1.89 (4)	2.734 (3)	178 (4)
O3–H3O \cdots O4 ⁱⁱ	0.77 (6)	2.06 (6)	2.823 (3)	173 (6)
C9–H9 \cdots O3 ⁱⁱⁱ	0.93	2.58	3.394 (4)	147
C11–H11 \cdots O6 ^{iv}	0.93	2.50	3.352 (4)	153

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

The hydroxyl H atoms were located from a difference map and their coordinates were refined with fixed individual displacement parameters [$U(\text{H}) = 1.5U_{\text{eq}}(\text{O})$]. Other H atoms were introduced at idealized positions and were allowed to ride on the parent atom.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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