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Bis(3-ethoxy-4-hydroxybenzaldehyde thiosemicarbazonato-κ^2S,N)nickel(II) monohydrate

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Key indicators

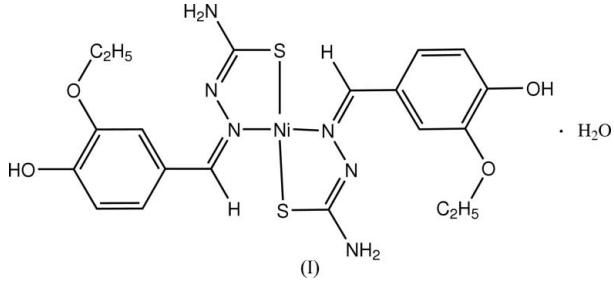
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
H-atom completeness 93%
Disorder in solvent or counterion
R factor = 0.053
wR factor = 0.142
Data-to-parameter ratio = 12.8

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

In the crystal structure of the title compound, $[Ni(C_{10}H_{12}N_3O_2S)_2]\cdot H_2O$, the Ni^{II} atom is located on an inversion center and is chelated by two hydroxyethoxybenzaldehyde thiosemicarbazonate anions in a square-planar geometry. The five-membered chelate ring assumes an envelope conformation, with the Ni atom lying at the flap position. The uncoordinated water molecule is disordered over another inversion center and is hydrogen bonded to the Ni^{II} complex.

Comment

Recently, much attention has been focused on thiosemicarbazone and its complexes because of their biological activities and chemical versatility (Marisa *et al.*, 1998; Shen *et al.*, 1998; Mao *et al.*, 1999). We present here the structure of the title Ni^{II} complex, (I), which incorporates hydroxyethoxybenzaldehyde thiosemicarbazonate (hetc) anions.



(I)

The molecular structure of (I) is shown in Fig. l. The Ni^{II} atom is located on an inversion center and is chelated by two hydroxyethoxybenzaldehyde thiosemicarbazonate (hetc) anions in a square-planar geometry. The five-membered chelate ring assumes an envelope conformation, with atom Ni1 lying at the flap position, displaced by 0.433 (5) Å from the mean plane formed by the other four atoms. The Ni—S and Ni—N bond distances (Table 1) agree with those found in an Ni^{II} complex with a thiosemicarbazone ligand (Marisa *et al.*, 2001).

The uncoordinated water molecule is disordered over an inversion center and is hydrogen bonded, as an acceptor, to the hydroxy group (Table 2). Intermolecular N—H···O hydrogen bonding stabilizes the crystal structure.

Experimental

4-Hydroxy-3-ethoxybenzaldehyde and thiosemicarbazide were dissolved in a water/ethanol solution (1:1) in 1:1 molar ratio. The solution was refluxed for 2 h. The solid product was separated and recrystallized from an ethanol solution to give pink crystals of H_2hetc.

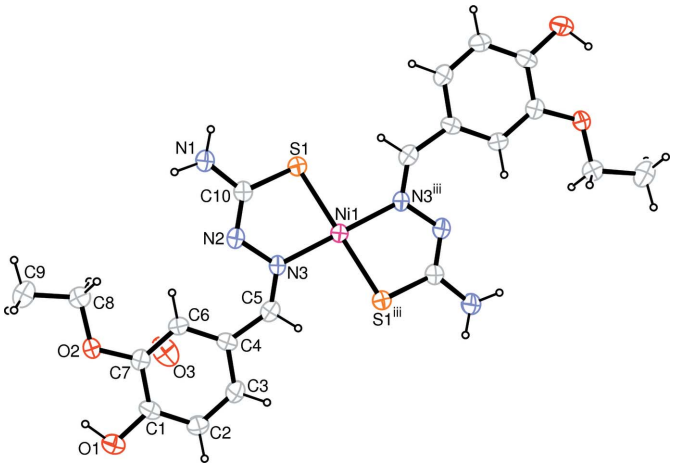


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids (arbitrary spheres for H atoms). [Symmetry code: (iii) $-x$, $2-y$, $-z$.]

A methanol solution (10 ml) of H_2hetc (0.2 mmol) was mixed with a tetrahydrofuran solution (10 ml) of $Ni(CH_3COO)_2{\cdot}2H_2O$ (0.2 mmol). The mixture was stirred at room temperature for 5 h. Single crystals of (I) were obtained after two weeks.

Crystal data

$[Ni(C_{10}H_{12}N_3O_2S)_2]{\cdot}H_2O$
M_r = 553.30
Triclinic, $P\overline{1}$
a = 5.794 (3) Å
b = 10.222 (4) Å
c = 10.227 (5) Å
α = 75.100 (7)°
β = 87.398 (6)°
γ = 81.956 (6)°
V = 579.6 (5) Å^3
Z = 1
D_x = 1.585 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 728 reflections
θ = 2.5–21.7°
μ = 1.06 mm^{-1}
T = 294 (2) K
Prism, red
0.27 × 0.13 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.763, T_{max} = 0.957
2904 measured reflections
2015 independent reflections
1593 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.1°
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 9$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.142
S = 0.97
2015 reflections
158 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.082P)^2 + 0.1304P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—S1	2.1712 (12)	N2—C10	1.282 (5)
Ni1—N3	1.916 (3)	N2—N3	1.402 (4)
S1—C10	1.723 (4)	N3—C5	1.279 (5)
N1—C10	1.350 (5)		
N3—Ni1—S1	85.53 (10)	C10—S1—Ni1	95.26 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.87	2.30	3.124 (5)	156
O1—H1C···O2	0.94	2.06	2.678 (5)	121
O1—H1C···O3^{ii}	0.94	2.05	2.896 (4)	148

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

H atoms of the uncoordinated water molecule are disordered and were not located. Amino and hydroxy H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.2U_{eq}(N,O)$. Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and refined as riding, with $U_{iso}(H) = 1.5U_{eq}(C)$ and the torsion angles refined to fit the electron density. Other H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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