

Acetatodiaqua[3-(salicyloylhydrazono)butan-2-one oximato]nickel(II) ethanol solvate

You Song, Yan Xu, Dun-Ru Zhu and Xiao-Zeng You*

Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China
Correspondence e-mail: xraychem@nju.edu.cn

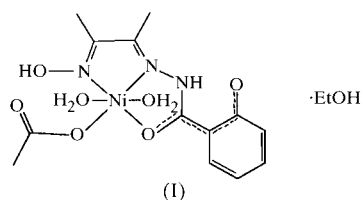
Received 10 September 1999

Accepted 6 January 2000

In the title compound, $[\text{Ni}(\text{C}_{11}\text{H}_{12}\text{N}_3\text{O}_3)(\text{CH}_3\text{CO}_2)(\text{H}_2\text{O})_2] \cdot \text{C}_2\text{H}_5\text{OH}$, the coordination geometry of the Ni^{II} atom is a distorted octahedron, with one carbonyl O and two imino N atoms of the hydrazone ligand, together with an acetate O atom, comprising the basal plane, and the two water O atoms occupying axial positions.

Comment

Salicylhydroxamic acid and analogous derivatives, because of their nature as polydentate ligands, may function as cation recognition agents to form metallacrown ether compounds (Gibney *et al.*, 1994; Psomas *et al.*, 1998), but salicylic hydrazide, with a similar structure, does not exhibit this function. Compared with salicylic hydrazide, salicyloyl hydrazone Schiff bases have additional donor atoms, whose presence introduces a wider range of properties (Kwak *et al.*, 1998) as new types of inorganic host molecules. We report here the crystal structure of a new nickel(II)–salicyloylhydrazone complex, (I), derived from salicylic hydrazide.



The coordination geometry of the Ni^{II} atom in (I) is a distorted octahedron. The two water molecules occupy axial positions, while the carbonyl O5 and the imino N1 and N2 atoms of the hydrazone ligand, together with the acetate O1 atom, comprise the basal plane (mean deviation from the plane of 0.0084 Å). The basal plane is stabilized by intramolecular O4–H4A···O2 hydrogen bonds, in which O4 is from the oxime and O2 from the acetate. Atom O6 of the hydrazone ligand and atom O3 of the ethanol solvate molecule do not form coordination bonds but can both link two water

molecules from two neighbouring molecules of the complex by strong intermolecular hydrogen bonds of the form O1Wⁱ–H2ⁱ···O6, O2Wⁱⁱ–H3ⁱⁱ···O6, O1W–H1···O3 and O2Wⁱⁱⁱ–H4ⁱⁱⁱ···O3; atom O6 has additional intramolecular hydrogen bonding with N3 (N3–H3A···O6) [symmetry codes: (i) $-x - 2, -y, -z$; (ii) $-x - 3, -y, -z$; (iii) $x + 1, y, z$]. Thus, two rows of complex molecules are connected by these hydrogen bonds to form polymeric chains, which are then connected by O3–H6A···O2^{iv} hydrogen bonds to yield a two-dimensional layer structure (see Fig. 2) [symmetry code: (iv) $-x - 1, 1 - y, 1 - z$].

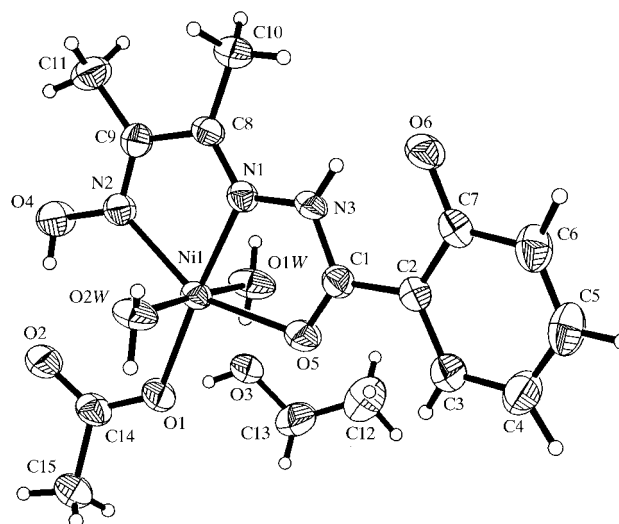


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are shown as spheres of arbitrary radii.

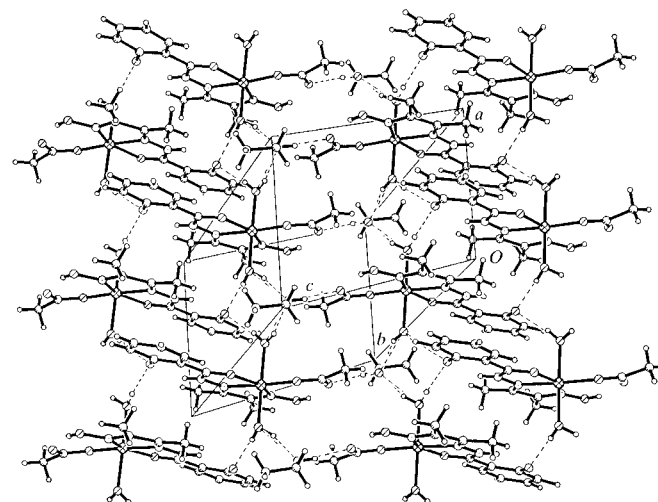


Figure 2
Packing diagram for (I).

Experimental

Compound (I) was synthesized by the reaction of a 1:1 molar ratio of diacetyl monoxime salicyloylhydrazone and nickel(II) acetate tetrahydrate in ethanol at room temperature. Green single crystals of (I)

suitable for X-ray diffraction were obtained by evaporating the solution in air for several weeks.

Crystal data

[Ni(C₁₁H₁₂N₃O₃)(C₂H₃O₂)(H₂O)₂].C₂H₆O
M_r = 434.09
 Triclinic, *P* $\bar{1}$
a = 7.4120 (15) Å
b = 11.505 (2) Å
c = 13.255 (3) Å
 α = 103.13 (3)°
 β = 103.97 (3)°
 γ = 106.80 (3)°
V = 995.0 (3) Å³
Z = 2
D_x = 1.449 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 1.56–6.68°
 μ = 1.020 mm⁻¹
T = 293 (2) K
 Prism, green
 0.40 × 0.20 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 2θ/ω scans
 Absorption correction: empirical via ψ scan (North *et al.*, 1968)
T_{min} = 0.654, *T_{max}* = 0.947
 3784 measured reflections
 3486 independent reflections
 2472 reflections with *I* > 2σ(*I*)
R_{int} = 0.022
 θ_{\max} = 24.98°
h = 0 → 8
k = -13 → 13
l = -15 → 15
 3 standard reflections every 97 reflections
 intensity decay: none

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.047
wR(*F*²) = 0.134
S = 1.000
 3484 reflections
 244 parameters
 H-atom parameters constrained
w = 1/[σ²(*F_o*²) + (0.0842*P*)²]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.63 e Å⁻³
 Δρ_{min} = -0.59 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ni1—O1	1.990 (3)	Ni1—O1W	2.040 (3)
Ni1—N1	2.012 (3)	Ni1—N2	2.100 (3)
Ni1—O2W	2.037 (3)	Ni1—O5	2.148 (3)
O2W—Ni1—O1W	177.10 (11)	O1—Ni1—O5	98.23 (11)
O1—Ni1—N2	109.91 (12)	N1—Ni1—O5	76.94 (12)
N1—Ni1—N2	74.92 (13)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O4—H4A...O2	2.576 (5)	163
O1W ⁱ —H2 ⁱ ...O6	2.688 (5)	164
O2W ⁱⁱ —H3 ⁱⁱ ...O6	2.658 (5)	172
O1W—H1...O3	2.754 (4)	166
O2W ⁱⁱⁱ —H4 ⁱⁱⁱ ...O3	2.773 (5)	158
O3—H6A...O2 ^{iv}	2.648 (4)	173
N3—H3A...O6	2.522 (3)	135

Symmetry codes: (i) -2 - *x*, -*y*, -*z*; (ii) -3 - *x*, -*y*, -*z*; (iii) 1 + *x*, *y*, *z*; (iv) -1 - *x*, 1 - *y*, 1 - *z*.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990a); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990b); software used to prepare material for publication: *SHELXL93*.

The work is supported by the National Science Foundation of China and the Analysis and Testing Found of Nanjing University. The authors would like to thank Hua-Qing Wang for help in the structure determination.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1121). Services for accessing these data are described at the back of the journal.

References

- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
 Gibney, B. R., Kessissoglou, D. P., Kampf, J. W. & Pecoraro, V. L. (1994). *Inorg. Chem.* **33**, 4840–4849.
 Kwak, B., Rhee, H., Park, S. & Lah, M. S. (1998). *Inorg. Chem.* **37**, 3599–3602.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Psomas, G., Dendrinou-Samara, C., Alexiou, M., Tsohos, A., Raptopoulou, C. P., Terzis, A. & Kessissoglou, D. P. (1998). *Inorg. Chem.* **37**, 6556–6557.
 Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1990b). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1993). *SHELXL93*. University of Göttingen, Germany.