

Triaqua[*N*-(5-formylsalicylidene)glycinato(2-)- $\kappa^3$ O,*N*,O']nickel(II) monohydrateXu-Hui Liu,<sup>a</sup> Jin-Hua Cai,<sup>a\*</sup>  
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## Key indicators

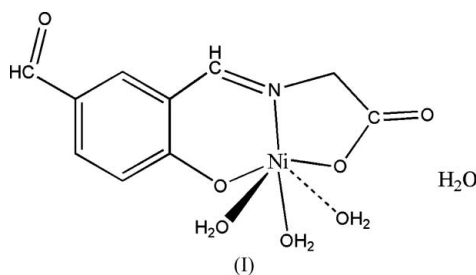
Single-crystal X-ray study  
*T* = 293 K  
Mean  $\sigma$ (C–C) = 0.004 Å  
*R* factor = 0.038  
*wR* factor = 0.092  
Data-to-parameter ratio = 13.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title complex,  $[\text{Ni}(\text{C}_{10}\text{H}_7\text{NO}_4)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}$ , a mononuclear nickel(II) compound, the  $\text{Ni}^{\text{II}}$  atom is six-coordinated in an octahedral geometry by the imine N, the phenolate O and a carboxyl O atom from the tridentate Schiff base ligand, and by three O atoms from three coordinated water molecules. In the crystal structure, the molecules are linked through intermolecular O–H...O hydrogen bonds, forming a three-dimensional framework.

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## Comment

Metal complexes with Schiff bases as ligands have played an important part in the development of inorganic chemistry as a whole. The research field dealing with Schiff base metal complexes has expanded enormously, as they have attracted much attention because of their catalytic activity in some industrial (Wu *et al.*, 2001) and biochemical processes (Kono & Fridovich, 1983; May *et al.*, 2004) and some polymerization reactions (Dutta & Das, 1988). Transition metal complexes of salicylaldehyde–amino acid Schiff bases are found to behave analogously to those of pyridoxal–amino acid Schiff bases (Snell *et al.*, 1963). However, complexes of the 5-formylsalicylaldehyde derivative have seldom been reported. We thus focused our attention on the assembly of transition metal ions with flexible ligands, and report here a new nickel(II) compound, (I).



The title compound is a mononuclear nickel(II) complex (Fig. 1) consisting of an [*N*-5-formylsalicylidene-glycinato-*N,O,O*]-triqua- $\text{Ni}^{\text{II}}$  unit and a solvent water molecule. The  $\text{Ni}^{\text{II}}$  atom is coordinated by the imine N, the phenolate O and one carboxyl O atom of the tridentate ligand Schiff base. The octahedral geometry is completed by the three O atoms of the coordinated water molecules. The equatorial plane is formed by four O atoms (O1, O5, O3, O7), while the axial sites are occupied by O6 and N1. The Ni–O and Ni–N bond lengths (Table 1) are comparable to the corresponding values observed in other Schiff base nickel(II) complexes (Cui *et al.*, 1993; Shova *et al.*, 2000).

It is interesting to note that, since 5-formylsalicylaldehyde has two aldehyde groups, a double Schiff base ligand should form from the reaction of glycine and 5-formylsalicylaldehyde. However, Fig. 1 shows that there is only one imine group in the complex. This is because, in the case of the imine *ortho* to the hydroxyl, the imino N, hydroxy O and Ni atoms can form a six-membered chelate ring stabilizing the imine group, while the other imine is unable to do this. As a result, the C=N double bond is broken down by attack by water.

In the crystal structure, the molecules are linked through intermolecular O—H...O hydrogen bonds (Table 2), forming a three-dimensional network structure.

### Experimental

All reagents were of commercially available grade and were used without further purification. 5-Formylsalicylaldehyde (0.1 mmol, 0.134 g), glycine (0.2 mmol, 0.15 g) and KOH (0.2 mmol, 0.112 g) were dissolved in aqueous methanol (20 ml). The mixture was stirred at room temperature for 10 min to give a clear yellow solution. To this solution was added an aqueous solution (10 ml) of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (0.2 mmol, 0.608 g), with stirring. The mixture was stirred and refluxed at 323 K for 6 h, then cooled to room temperature. After filtration, the filtrate was left to stand at room temperature. Green crystals suitable for X-ray diffraction analysis were obtained in a yield of 45%. Analysis found (%): C 35.70, H 4.52, N 4.19; C<sub>10</sub>H<sub>15</sub>NNiO<sub>8</sub> requires (%): C 35.75, H 4.50, N 4.17%.

#### Crystal data

[Ni(C <sub>10</sub> H <sub>7</sub> NO <sub>4</sub> )(H <sub>2</sub> O) <sub>3</sub> ]-H <sub>2</sub> O	Z = 8
<i>M<sub>r</sub></i> = 335.92	<i>D<sub>x</sub></i> = 1.684 Mg m <sup>-3</sup>
Orthorhombic, <i>Pbca</i>	Mo <i>K</i> α radiation
<i>a</i> = 11.158 (4) Å	<i>μ</i> = 1.50 mm <sup>-1</sup>
<i>b</i> = 7.674 (3) Å	<i>T</i> = 293 (2) K
<i>c</i> = 30.950 (12) Å	Block, green
<i>V</i> = 2650.3 (18) Å <sup>3</sup>	0.15 × 0.10 × 0.08 mm

#### Data collection

Bruker SMART CCD area-detector diffractometer	12108 measured reflections
φ and ω scans	2886 independent reflections
Absorption correction: multi-scan ( <i>SADABS</i> ; Sheldrick, 1996)	2241 reflections with <i>I</i> > 2σ( <i>I</i> )
<i>T<sub>min</sub></i> = 0.806, <i>T<sub>max</sub></i> = 0.889	<i>R<sub>int</sub></i> = 0.057
	<i>θ<sub>max</sub></i> = 27.0°

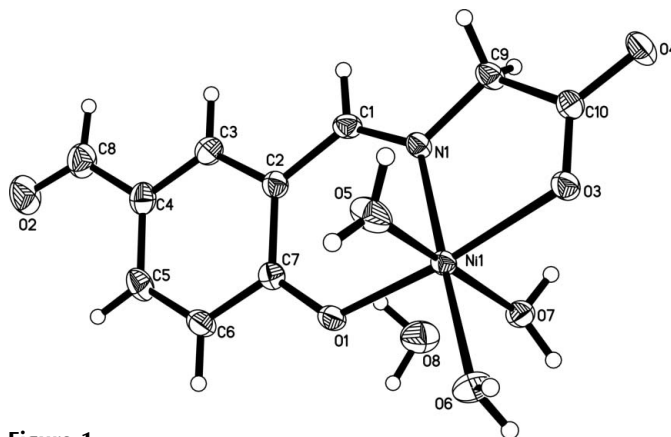
#### Refinement

Refinement on <i>F</i> <sup>2</sup>	H atoms treated by a mixture of independent and constrained refinement
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.038	<i>w</i> = 1/[σ <sup>2</sup> ( <i>F<sub>o</sub></i> <sup>2</sup> ) + (0.0494 <i>P</i> ) <sup>2</sup> ]
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.092	where <i>P</i> = ( <i>F<sub>o</sub></i> <sup>2</sup> + 2 <i>F<sub>c</sub></i> <sup>2</sup> )/3
<i>S</i> = 1.01	(Δ/σ) <sub>max</sub> = 0.002
2886 reflections	Δρ <sub>max</sub> = 0.43 e Å <sup>-3</sup>
213 parameters	Δρ <sub>min</sub> = -0.37 e Å <sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Ni1—Ni1	2.002 (2)	Ni1—O3	2.0889 (18)
Ni1—O1	2.0092 (18)		
N1—Ni1—O1	90.79 (8)	O1—Ni1—O3	171.27 (7)
N1—Ni1—O3	80.87 (8)		



**Figure 1**

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O8—H8A...O5 <sup>i</sup>	0.89 (2)	2.00 (5)	2.668 (4)	131 (6)
O7—H7B...O4 <sup>ii</sup>	0.831 (17)	1.923 (18)	2.745 (3)	170 (3)
O5—H5A...O8 <sup>iii</sup>	0.857 (17)	1.824 (18)	2.668 (4)	168 (3)
O8—H8B...O2 <sup>iv</sup>	0.86 (2)	2.02 (4)	2.774 (4)	146 (7)
O5—H5B...O1 <sup>v</sup>	0.823 (17)	1.922 (18)	2.737 (3)	170 (3)
O6—H6A...O4 <sup>vi</sup>	0.857 (17)	1.819 (19)	2.659 (3)	166 (3)
O6—H6B...O4 <sup>vii</sup>	0.851 (18)	2.15 (2)	2.965 (3)	160 (3)
O7—H7A...O3 <sup>vi</sup>	0.846 (17)	1.916 (18)	2.760 (3)	175 (3)

Symmetry codes: (i) *x*, *y* + 1, *z*; (ii)  $-x + \frac{3}{2}$ ,  $y + \frac{1}{2}$ , *z*; (iii) *x*, *y* - 1, *z*; (iv)  $-x + 1$ ,  $-y + 1$ ,  $-z + 1$ ; (v)  $-x + \frac{1}{2}$ ,  $y - \frac{1}{2}$ , *z*; (vi)  $-x + 1$ ,  $y + \frac{1}{2}$ ,  $-z + \frac{1}{2}$ ; (vii)  $x - \frac{1}{2}$ ,  $-y$ ,  $-z + \frac{1}{2}$ .

The coordinated and uncoordinated water H atoms were located in a difference Fourier map and refined isotropically, with the O—H distances restrained to 0.82 (3) and 0.89 (2) Å, and refined as riding in their as-found relative positions. The C-bound H atoms were placed in calculated positions, with C—H(aryl) = 0.93 Å and C—H(methylene) = 0.97 Å, and refined using a riding model, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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### References

- Bruker (1998). *SMART* (Version 5.628) and *SAINTE* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
- Cui, X. G., Sun, H. J., Liu, D. X., Li, X. Y. & Li, S. L. (1993). *Acta Chim. Sin* **51**, 346–351.
- Dutta, R. L. & Das, B. R. (1988). *J. Sci. Ind. Res.* **47**, 547–551.
- Kono, Y. & Fridovich, I. (1983). *J. Biol. Chem.* **258**, 6015–6020.
- May, J. P., Ting, R., Lermer, L., Thomas, J. M., Roupioz, Y. & Perrin, D. M. (2004). *J. Am. Chem. Soc.* **126**, 4145–4156.

- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Shova, S., Novitski, G., Gdanets, M., Simonov, Yu. A. & Turte, K. (2000). *Zh. Neorg. Khim.* **45**, 1313–1317. (In Russian.)
- Snell, E. E., Fasella, P. M., Braunstein, A. & Rossi-Fanelli, A. (1963). *Chemical and Biological Aspects of Pyridoxal Catalysis*. New York: Macmillan.
- Wu, Z. Y., Xu, D. J. & Feng, Z. X. (2001). *Polyhedron*, **20**, 281–284.