

***trans*-Diaquabis{2-[3-(dimethylamino)prop-2-enoyl]-pyridine}nickel(II) bis(tetrafluoroborate) dimethylformamide tetrasolvate****Ze-Qun Yan**

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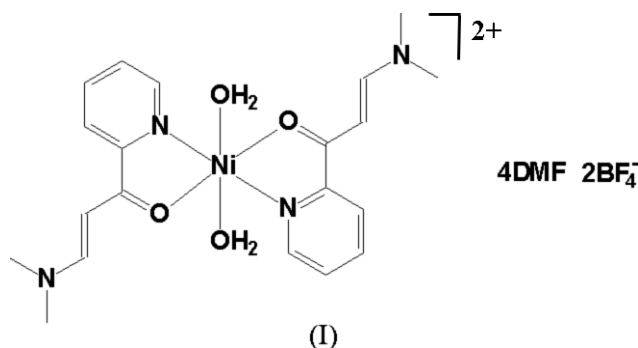
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**Key indicators**Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.009$  Å  
Disorder in solvent or counterion  
 $R$  factor = 0.074  
 $wR$  factor = 0.200  
Data-to-parameter ratio = 11.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $[\text{Ni}(\text{C}_{10}\text{H}_{12}\text{N}_2\text{O})_2(\text{H}_2\text{O})_2](\text{BF}_4)_2 \cdot 4\text{C}_3\text{H}_7\text{NO}$ , was synthesized by the reaction of 2-[3-(dimethylamino)prop-2-enoyl]pyridine and  $\text{Ni}(\text{BF}_4)_2$  in dimethylformamide. It has been characterized by elemental analysis and X-ray diffraction techniques. The crystal structure reveals that the  $\text{Ni}^{\text{II}}$  atom, on an inversion center, has an  $\text{NiN}_2\text{O}_4$  octahedral coordination environment with two carbonyl O atoms and two pyridyl N atoms in the equatorial plane and two water molecules in the axial positions. 2-[3-(Dimethylamino)prop-2-enoyl]pyridine chelates to the Ni atom through the O and pyridine N atoms. Tetrafluoroborate counter-anions and dimethylformamide solvent molecules are not coordinated to the central metal ion.

**Comment**

The synthesis and design of supramolecular compounds with special geometric and topological character have attracted much attention in recent decades, because they display recognition, transformation and self-assembly properties (Zhu *et al.*, 2001; Braga, 2000). We have synthesized and characterized an  $\text{Ni}^{\text{II}}$  complex, (I), with the ligand 2-[3-(dimethylamino)prop-2-enoyl]pyridine (*L*).



The crystal structure analysis reveals that (I) is a monomeric  $\text{Ni}^{\text{II}}$  complex (Fig. 1). The coordination geometry around the  $\text{Ni}^{\text{II}}$  atom, which lies on an inversion center, is octahedral with two carbonyl O atoms and two pyridyl N atoms from *L* in the equatorial plane and two water molecules in the axial positions. The Ni—O, Ni—OW and Ni—N distances are 2.028 (3), 2.097 (4) and 2.038 (4) Å, respectively, which fall into the normal ranges. Ligand *L* chelates to the Ni atom through the O and pyridine N atoms, and the tetrafluoroborate counter-anions and dimethylformamide solvent molecules are not coordinated to the central metal ion.

Experimental

Complex (I) was prepared by reacting a dimethylformamide (DMF, 15 ml) solution of nickel(II) bis(tetrafluoroborate) with 2-[3-(dimethylamino)prop-2-enyl]pyridine in a 1:2 ratio. The mixture was stirred at room temperature for 1 h. Green block-shaped crystals were recovered by filtration and dried in air. The yield was ca 45% (based on Ni). Analysis calculated for C<sub>32</sub>H<sub>56</sub>B<sub>2</sub>F<sub>8</sub>N<sub>8</sub>NiO<sub>8</sub>: C 42.09, H 6.19, N 12.28%; found: C 42.34, H 6.28, N 12.51%.

Crystal data

[Ni(C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>](BF<sub>4</sub>)<sub>2</sub>·4C<sub>3</sub>H<sub>7</sub>NO  
*M<sub>r</sub>* = 913.18  
 Triclinic, *P* $\bar{1}$   
*a* = 10.036 (3) Å  
*b* = 10.384 (3) Å  
*c* = 13.384 (3) Å  
 $\alpha$  = 107.744 (5)°  
 $\beta$  = 93.331 (5)°  
 $\gamma$  = 118.732 (5)°  
*V* = 1129.4 (5) Å<sup>3</sup>  
*Z* = 1  
*D<sub>x</sub>* = 1.343 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 652 reflections  
 $\theta$  = 3.2–23.0°  
 $\mu$  = 0.52 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, green  
 0.25 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.743, *T<sub>max</sub>* = 0.902  
 5657 measured reflections  
 3835 independent reflections  
 2265 reflections with *I* > 2 *s*(*I*)  
*R<sub>int</sub>* = 0.040  
 $\theta_{max}$  = 25.1°  
*h* = -11 → 11  
*k* = -12 → 12  
*l* = -14 → 15

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.074  
*wR* (*F*<sup>2</sup>) = 0.201  
*S* = 0.98  
 3835 reflections  
 324 parameters  
 H atoms treated by a mixture of independent and constrained refinement  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.1115*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.84 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.54 e Å<sup>-3</sup>

Table 1 Selected geometric parameters (Å, °).

Ni1—O1	2.028 (3)	Ni1—O2	2.097 (4)
Ni1—N1	2.038 (4)		
O1—Ni1—O1 <sup>i</sup>	180	N1—Ni1—O2 <sup>i</sup>	90.02 (17)
O1—Ni1—N1	80.00 (16)	O1—Ni1—O2	89.73 (16)
O1 <sup>i</sup> —Ni1—N1	100.00 (16)	N1—Ni1—O2	89.98 (17)
N1—Ni1—N1 <sup>i</sup>	180	O2 <sup>i</sup> —Ni1—O2	180
O1—Ni1—O2 <sup>i</sup>	90.27 (16)		

Symmetry code: (i) -x, 2 - y, 2 - z.

C-bound H atoms were placed at calculated positions and allowed to ride on their parent atoms [C—H = 0.93 Å and *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C)]. H atoms of the water molecules were located in a

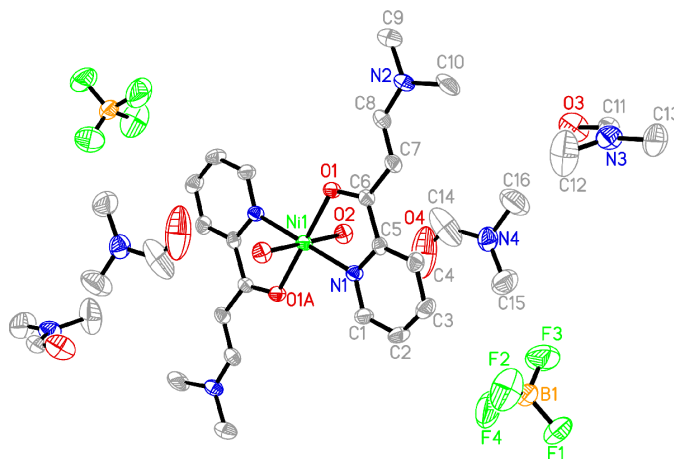


Figure 1 View of the title compound, showing ellipsoids at the 30%. Suffix A corresponds to symmetry code (i) in Table 1. Minor disorder components and H atoms are omitted.

difference map and were refined isotropically, with O—H distances restrained to 0.85 (1) Å. One of the solvent molecules is disordered and the occupancies of the two components C11, C12, C13, O3 and C11', C12', C13', O3' refined to 0.64 (2) and 0.36 (2). Atoms F3 and F4 of the tetrafluoroborate counter-anion are also disordered and the occupancies refined to 0.52 (4) and 0.48 (4).

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

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