

## metal-organic papers

Acta Crystallographica Section E  
 Structure Reports  
 Online

ISSN 1600-5368

John Bacsa,\* Hanhua Zhao and  
 Kim R. Dunbar

Department of Chemistry, Texas A&M  
 University, PO Box 30012, College Station,  
 Texas 77842-3012, USA

Correspondence e-mail:  
 jbacsa@mail.chem.tamu.edu

## Key indicators

Single-crystal X-ray study  
 T = 110 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
 R factor = 0.036  
 wR factor = 0.080  
 Data-to-parameter ratio = 11.7

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

## Bis(pyridin-2-ylmethanolato- $\kappa^2N,O$ )bis-(trifluoroacetato)nickel(II)

The  $\text{Ni}^{\text{II}}$  atom in the title compound,  $[\text{Ni}(\text{C}_2\text{F}_3\text{O}_2)_2(\text{C}_6\text{H}_7\text{NO})_2]$ , is located on an inversion centre and is in an irregular coordination environment. Two trifluoroacetate ions and two pyridin-2-ylmethanol molecules coordinate to the  $\text{Ni}^{\text{II}}$  atom. The pyridin-2-ylmethanol ligand coordinates *via* its pyridyl N atom and its methanol O atom, resulting in a five-membered chelate ring. The ring is puckered in an envelope conformation, with the methanol O atom as flap. The trifluoroacetate ions coordinate *via* one O atom only. The second O atom accepts a strong hydrogen bond from the methanol hydroxy group.

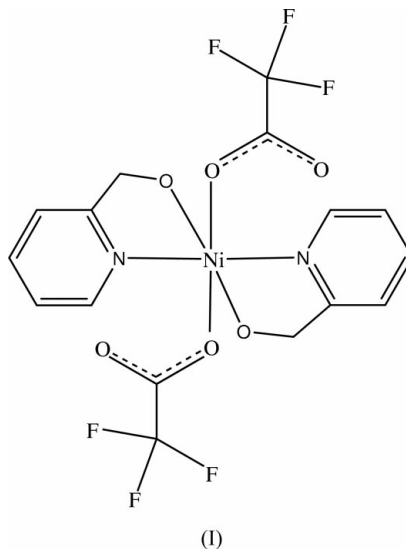
Received 21 June 2004

Accepted 28 June 2004

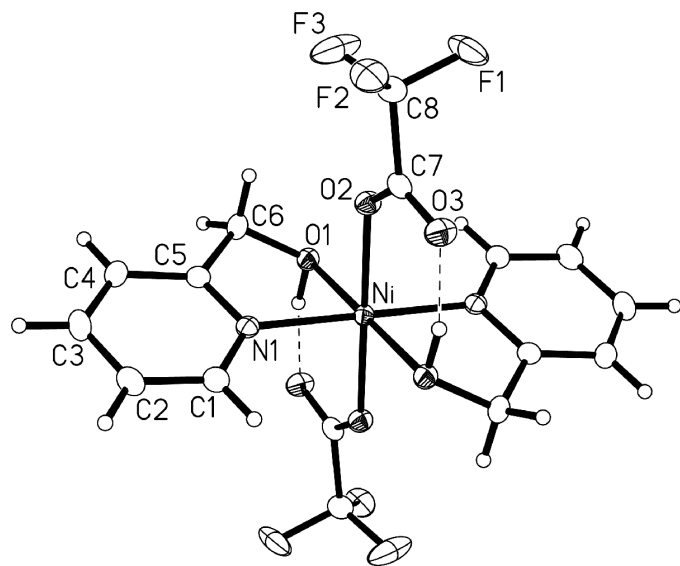
Online 9 July 2004

## Comment

The molecule of the title compound, (I), is neutral, and consists of an  $\text{Ni}^{\text{II}}$  atom coordinated by two pyridin-2-ylmethanol (pym) molecules and two trifluoroacetate (tfa) ions (Fig. 1). The  $\text{Ni}^{\text{II}}$  atom is located on an inversion centre and there is one crystallographically unique pym ligand and one unique tfa ion. The pym ligand coordinates to  $\text{Ni}^{\text{II}}$  *via* its pyridyl N atom and its methanol O atom, resulting in a five-membered chelate ring. This ring is puckered in an envelope configuration, with O1 as flap (Evans & Boeyens, 1989).



The C—O bond distances [1.242 (3) and 1.247 (3)  $\text{\AA}$ ] are intermediate between single and double bonds (bond order 1.5). The trifluoroacetate ion coordinates to  $\text{Ni}^{\text{II}}$  *via* one O atom only. The negative charge on the second O atom is partially stabilized since this O atom accepts a strong intramolecular hydrogen bond from the methanol hydroxy group. By treating this hydrogen bond as part of the bonding framework, the carboxylate ion is bidentate and, together with


**Figure 1**

View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as circles of arbitrary radius and hydrogen bonds are shown as dashed lines.

the intramolecular hydrogen bond, forms a distorted, nearly planar six-membered ring. The coordination environment about the Ni<sup>II</sup> atom deviates substantially from ideal octahedral geometry due to the conformational preferences of the bidentate pym ligand. Other mononuclear divalent first-row transition metal complexes ( $M = \text{Mn, Fe, Co, Ni, Cu, and Zn}$ ) of pym and the saccharinate anion have been characterized by X-ray crystallography (Yilmaz *et al.*, 2002). The structure of a mononuclear Cu<sup>II</sup> complex of pym and the salicylate anion has also been published (Hoang *et al.*, 1992). The metal atoms in these complexes are also six-coordinate, and the pym ligands bidentate (binding through the N and O atoms). Each of these complexes is also stabilized by a strong intramolecular hydrogen bond, but in these cases either the carbonyl O or N atoms of the saccharinate or salicylate anions are the acceptors.

## Experimental

A solution of pyridin-2-ylmethanol (0.54 g, 5.0 mmol) in tetrahydrofuran (15 ml) was added to a suspension of NiBr<sub>2</sub> (0.62 g, 0.53 mmol) and NaO<sub>2</sub>CCF<sub>3</sub> (0.62 g, 4.5 mmol) in tetrahydrofuran (15 ml) with stirring. The color of the suspension changed from light green to blue after adding pyridin-2-ylmethanol. The suspension was refluxed for 4 h and filtered at room temperature. Blue crystals were obtained after the blue filtrate was evaporated slowly under an N<sub>2</sub> atmosphere for 3 d.

### Crystal data

[Ni(C<sub>2</sub>F<sub>3</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>7</sub>NO)<sub>2</sub>]  
 $M_r = 503.00$

Monoclinic,  $C2/c$   
 $a = 12.539$  (3) Å  
 $b = 11.113$  (2) Å  
 $c = 13.315$  (3) Å  
 $\beta = 92.23$  (3)°  
 $V = 1854.0$  (7) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.802$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 2173 reflections  
 $\theta = 2.4\text{--}27.3^\circ$   
 $\mu = 1.15$  mm<sup>-1</sup>  
 $T = 110$  (2) K  
 Prism, blue  
 $0.29 \times 0.26 \times 0.13$  mm

### Data collection

Bruker SMART 1K CCD diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 2003)  
 $T_{\min} = 0.733$ ,  $T_{\max} = 0.865$   
 4350 measured reflections

2012 independent reflections  
 1769 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$   
 $\theta_{\text{max}} = 27.2^\circ$   
 $h = -16 \rightarrow 14$   
 $k = -11 \rightarrow 14$   
 $l = -12 \rightarrow 17$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.036$   
 $wR(F^2) = 0.080$   
 $S = 1.09$   
 2012 reflections  
 172 parameters  
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.022P)^2 + 5.385P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.35$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.38$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Ni—N1	2.041 (2)	C2—C3	1.391 (4)
Ni—O1	2.070 (2)	C3—C4	1.388 (4)
Ni—O2	2.077 (2)	C4—C5	1.383 (3)
C1—N1	1.347 (3)	C5—C6	1.508 (3)
C1—C2	1.377 (3)	C7—C8	1.549 (3)
O1—C6	1.437 (3)	C8—F3	1.322 (3)
N1—C5	1.346 (3)	C8—F2	1.333 (3)
O2—C7	1.247 (3)	C8—F1	1.342 (3)
O3—C7	1.242 (3)		
N1—Ni—N1 <sup>i</sup>	180	C5—C4—C3	119.0 (2)
N1—Ni—O1	80.50 (7)	N1—C5—C4	121.8 (2)
N1 <sup>i</sup> —Ni—O1	99.50 (7)	N1—C5—C6	115.6 (2)
O1—Ni—O1 <sup>i</sup>	180	C4—C5—C6	122.6 (2)
N1—Ni—O2 <sup>i</sup>	92.26 (7)	O1—C6—C5	111.0 (2)
O1—Ni—O2 <sup>i</sup>	93.50 (6)	O3—C7—O2	129.9 (2)
O1—Ni—O2	86.51 (6)	O3—C7—C8	114.8 (2)
N1—Ni—O2	87.74 (7)	O2—C7—C8	115.2 (2)
O2 <sup>i</sup> —Ni—O2	180	F3—C8—F2	106.9 (2)
N1—C1—C2	122.0 (2)	F3—C8—F1	108.2 (2)
C5—N1—C1	119.2 (2)	F2—C8—F1	106.0 (2)
C5—N1—Ni	113.8 (2)	F3—C8—C7	113.4 (2)
C1—N1—Ni	127.0 (2)	F2—C8—C7	112.2 (2)
C1—C2—C3	119.0 (2)	F1—C8—C7	109.8 (2)
C4—C3—C2	119.0 (2)		

Symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$ .

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
O1—H7 <sup>i</sup> ···O3 <sup>i</sup>	0.981 (10)	1.670 (13)	2.628 (2)	164 (3)

Symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$ .

All the H atoms were located in difference electron-density maps and refined isotropically.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: X-SEED (Barbour, 2001) and SHELXTL (Bruker, 2000); software used to prepare material for publication: PLATON (Spek, 2003).

We gratefully acknowledge the service provided by Dr Joseph Reibenspies and the X-ray facility at Texas A&M University. We thank the National Science Foundation for PI and NIRT grants (CHE-9906583 and DMR-0103455) and for

equipment grants for the CCD X-ray equipment (CHE-9807975). We thank the Department of Energy for a PI grant (DE-FG03-02ER45999). Support from the Welch Foundation (A-1449) and from a Telecommunications and Informatics Task Force (TITF) Grant from Texas A&M University is also gratefully acknowledged.

### References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.

- Bruker (2000). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2001). *SMART* (Version 5.625) and *SAINTE* (Version 6.34). Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Evans, D. G. & Boeyens, J. C. A. (1989). *Acta Cryst.* **B45**, 581–590.
- Hoang, N. N., Valach, F., Dunaj-Jurco, M. & Melnik, M. (1992). *Acta Cryst.* **C48**, 443–445.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Yilmaz, V. T., Guney, S., Andac, O. & Harrison, W. T. A. (2002). *Polyhedron*, **21**, 2393–2402.