

Yin-Qiu Liu,^a Xi-Rui Zeng,^{b*}
Han-Mao Kuang,^b Hua-Long
Chen^b and Qiu-Yan Luo^b^aGanNan Normal College, JiangXi Province Key Laboratory of Coordination Chemistry, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China, and ^bCollege of Chemistry and Chemical Engineering, JiangXi Province Key Laboratory of Coordination Chemistry, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China

Correspondence e-mail: zengxirui@jgsu.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.030
 wR factor = 0.079
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Diacetato[2,6-bis(2-pyridylamino)pyridine]nickel(II)

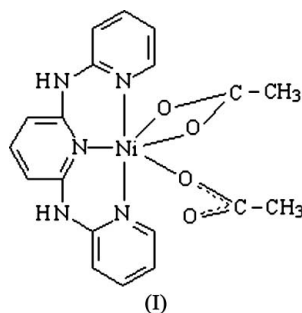
The Ni atom in the title complex, $[\text{Ni}(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_{15}\text{H}_{13}\text{N}_5)]$ or $[\text{Ni}(\text{CH}_3\text{COO})_2(\text{tpdaH}_2)]$ (tpdaH₂ is tripyridyldiamine), has a distorted octahedral coordination geometry formed by the tridentate tpdaH₂ ligand and two acetate groups. The tpdaH₂ ligand is *mer*-coordinated, with the N atom of the central pyridine ring in the equatorial position and the N atoms of the peripheral pyridine rings in the axial positions. The remaining three equatorial positions are occupied by the O atoms of two acetate anions. The H atoms of both NH groups of the tpdaH₂ ligand are involved in strong hydrogen bonds with O atoms of adjacent complex molecules to form a three-dimensional network.

Received 22 February 2006

Accepted 24 March 2006

Comment

Transition metal complexes with polypyridylamine ligands, possessing diverse structures and special optical and electromagnetic properties (Xu *et al.*, 2004), have aroused great interest among researchers. The tripyridyldiamine ligand usually exhibits donor as well as acceptor properties and is frequently used as a chelating ligand (Jing *et al.*, 2000). In recent years great efforts have been devoted to synthesizing and characterizing metal chain complexes which can be used to study metal–metal interactions (Yang *et al.*, 1997; Cotton *et al.*, 1998). In addition, metal chain complexes with potential application as new nanomaterials, such as molecular metal wires, have attracted much attention and have been extensively investigated (Peng *et al.*, 2000). A series of polynuclear metal chain complexes has been successfully synthesized and characterized (Sheu *et al.*, 1996; Shieh *et al.*, 1997; Chang *et al.*, 1999). In our ongoing research aimed at the synthesis of new multinuclear transition metal complexes, we attempted to synthesize complexes featuring Ni metal chains by reaction of the nickel(II) ion with tripyridyldiamine (tpdaH₂), but the only product we obtained was the mononuclear title complex, (I), the structure of which is reported here.



Atom Ni1 in the title complex has a distorted octahedral coordination geometry provided by the tridentate tpdaH₂

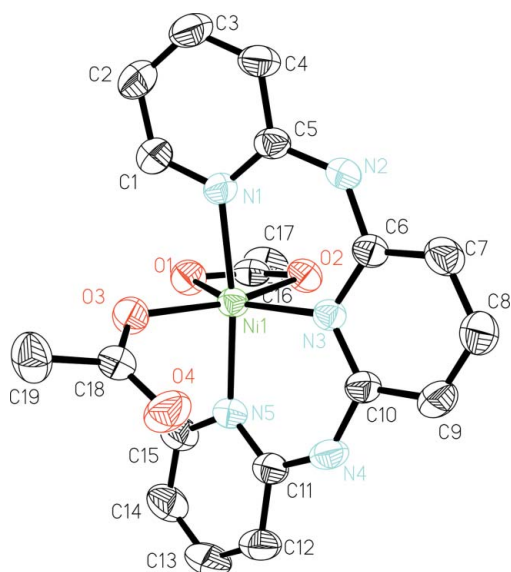


Figure 1
A view of (I), showing 50% probability displacement ellipsoids. H atoms have been omitted.

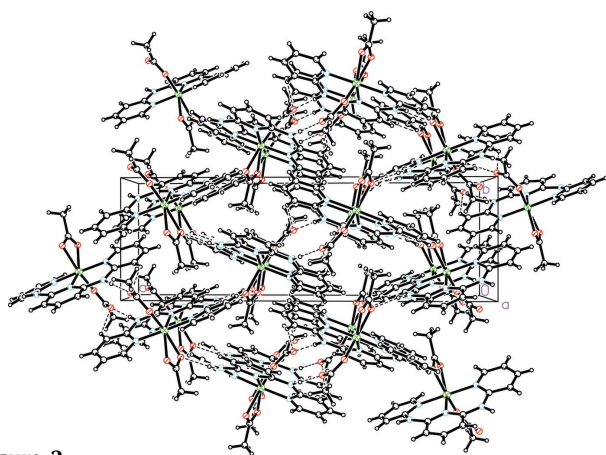


Figure 2
Packing diagram of (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

ligand and two acetate anions (Fig. 1). The tpdah2 ligand is *mer*-coordinated, with the peripheral N1 and N5 atoms in the axial positions and the central N3 atom equatorial. The remaining equatorial positions are occupied by the O atoms of two acetate anions. Selected geometric parameters are listed in Table 1. Atoms O1, O2, O3 and N3 in the equatorial plane of the octahedron are approximately coplanar with the central Ni atom, the maximum deviation from the least-squares plane through all five atoms being 0.0367 (5) Å for atom O2. The three pyridine rings of the tpdah2 ligand are not coplanar. The dihedral angles between the planes of the central pyridine ring and two peripheral rings are 13.0 (4) and 21.4 (9)°. The molecular configuration of the title complex is similar to that of [2,6-bis(2-pyridylamino)pyridine]dinitratocadmium monohydrate (Fang *et al.*, 2005).

In the crystal structure of (I), the molecules are linked by strong intermolecular hydrogen bonds involving both amine groups of tpdah2 and the O atoms of adjacent acetate groups,

and by weak intermolecular C—H···O hydrogen interactions (Table 2), resulting in the formation of a three-dimensional network (Fig. 2).

Experimental

Tripyridyldiamine (0.05 g) and nickel acetate (0.18 g) were added to a solution of dry ethanol (10 ml) and acetonitrile (12 ml). The mixture was heated at 333 K for 5 h under reflux with stirring. The resulting solution was then filtered off and diethyl ether was added. Single crystals suitable for X-ray diffraction analysis formed after a week by slow evaporation of the solvent (40% yield).

Crystal data

[Ni(C₂H₃O₂)₂(C₁₅H₁₃N₅)]
M_r = 440.10
 Monoclinic, *P*2₁/*c*
a = 9.6135 (4) Å
b = 7.9969 (3) Å
c = 24.4047 (9) Å
 β = 93.942 (1)°
V = 1871.75 (13) Å³
Z = 4

D_x = 1.562 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 8134 reflections
 θ = 2.1–29.1°
 μ = 1.08 mm⁻¹
T = 273 (2) K
 Block, blue
 0.30 × 0.17 × 0.10 mm

Data collection

Bruker APEX-II area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.80, *T_{max}* = 0.89
 21140 measured reflections

4296 independent reflections
 3478 reflections with *I* > 2σ(*I*)
R_{int} = 0.033
 θ_{\max} = 27.5°
h = -12 → 12
k = -10 → 10
l = -31 → 31

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.030
wR (*F*²) = 0.079
S = 1.04
 4296 reflections
 264 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.399P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.27 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1—O3	2.0350 (13)	Ni1—N3	2.0737 (14)
Ni1—N1	2.0467 (15)	Ni1—O2	2.1535 (13)
Ni1—N5	2.0485 (15)	Ni1—O1	2.2394 (14)
O3—Ni1—N1	84.06 (6)	O3—Ni1—N3	110.87 (6)
O3—Ni1—N5	89.75 (6)	N1—Ni1—N3	92.34 (6)
N1—Ni1—N5	172.37 (6)	N5—Ni1—N3	94.03 (6)

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>
N2—H2A···O2 ⁱ	0.86	2.07	2.9139 (19)	167
N4—H4A···O4 ⁱⁱ	0.86	1.92	2.777 (2)	174
C12—H12···O4 ⁱⁱ	0.93	2.48	3.212 (3)	136
C15—H15···O1	0.93	2.34	2.949 (2)	123

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x, -y + 1, -z + 1$.

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C–H = 0.93–0.96 Å, N–H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},\text{N})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2*.

We gratefully acknowledge the financial support of the Natural Science Foundation of JiangXi Province (grant Nos. 0320024 and 0520036)

References

Bruker (2004). *APEX2* (Version 1.22) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

- Chang, H.-C., Li, J.-T., Wang, C.-C., Lin, T.-W., Lee, H.-C., Lee, G.-H. & Peng, S.-M. (1999). *Eur. J. Inorg. Chem.* pp. 1243–1251.
- Cotton, F. A., Daniels, L. M., Murillo, C. A. & Wang, X. (1998). *Chem. Commun.* pp. 39–40.
- Fang, X.-N., Li, X.-F. & Zeng, X.-R. (2005). *Acta Cryst.* **E61**, m1123–m1125.
- Jing, B.-W., Wu, T., Zhang, M.-W. & Shen, T. (2000). *Chem. J. Chin. Univ.* **21**, 395–400.
- Peng, S.-M., Wang, C.-C., Jang, Y.-L., Chen, Y.-H., Li, F.-Y., Mou, C.-Y. & Leung, M.-K. (2000). *J. Magn. Magn. Mater.* **209**, 80–83.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheu, J. T., Liu, T. W. & Peng, S. M. (1996). *Chem. Commun.* pp. 315–316.
- Shieh, S.-J., Chou, C.-C., Lee, G.-H., Wang, C.-C. & Peng, S.-M. (1997). *Angew. Chem. Int. Ed. Engl.* **36**, 56–58.
- Xu, C., Qiao, H.-B., Mao, H.-Y., Zhang, H.-Y., Wu, Q.-A., Liu, H.-L. & Zhu, Y. (2004). *J. Zheng Zhou Univ.* **36**, 67–70. (In Chinese.)
- Yang, M. H., Lin, T. W., Chou, C. C., Lee, H. C., Chang, H. C., Lee, G. H., Leung, M. K. & Peng, S. M. (1997). *Chem. Commun.* pp. 39–40.