Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Bis(dimethylformamide-2*κO*)dipyridine-1*κN*,3*κN*-bis(μ-2′-salicylbenzohydrazidato)-1κ^3*O*,*N*,*O*′:-2κ^2*N*,*O*″;2κ^2*N*,*O*″:3κ^3*O*,*N*,*O*′-trinickel(II) dimethylformamide disolvate hemihydrate

Ming-Xing Yang and Shen Lin*

College of Chemistry and Material Science, Fujian Normal University, Fuzhou, Fujian 350007, People's Republic of China

Correspondence e-mail: shenlin@fjnu.edu.cn

Received 29 March 2005
Accepted 3 May 2005
Online 14 May 2005

Key indicators

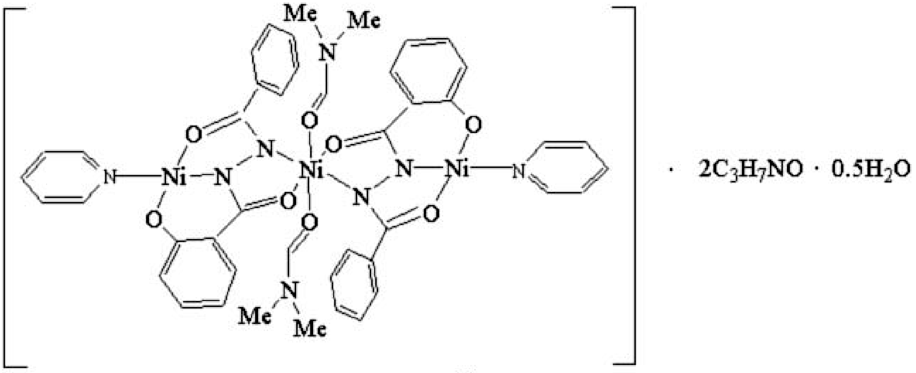
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.047
wR factor = 0.137
Data-to-parameter ratio = 17.9

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

In the trinuclear nickel(II) complex of the title compound, $[Ni_3(C_{30}H_{20}N_4O_6)_2(C_5H_5N)_2(C_2H_6NO)_2]\cdot 2C_3H_7NO\cdot 0.5H_2O$, the central Ni^{2+} ion is located on an inversion centre. This Ni^{2+} ion has a distorted octahedral coordination geometry involving two N and two O atoms from the 2′-salicylbenzohydrazidate ligands in the equatorial plane and two N atoms from two dimethylformamide molecules in the axial positions. The inversion-related outer Ni^{2+} ions, separated by 9.3038 (7) Å, have square-planar coordination environments.

Comment

N-Alkylsalicylhydrazides have been actively investigated as multifunctional ligands which can chelate as well as bridge metal ions to give polynuclear, metallamacroyclic and one-dimensional complexes (Kwak *et al.*, 1998, 2000; Kim *et al.*, 2001; Lin *et al.*, 2002, 2002*a*, 2002*b*, 2004; Liu *et al.*, 2001; Yang *et al.*, 2003; Li *et al.*, 2005), which have potential applications in chemically modified electrodes, anion-selective separation agents, magnetic materials and bioinorganic chemistry (Liu *et al.*, 2001; Alexiou *et al.*, 2003). In these complexes, the triply deprotonated *N*-acylsalicylhydrazides may bridge the neighboring metal ions through their hydrazide N—N group, forming polynuclear complexes. We report here the synthesis and crystal structure of a new linear trinuclear nickel(II) complex, (I), with 2′-salicylbenzohydrazide, in which the central Ni atom adopts an axially elongated octahedral geometry, whereas the two outer Ni atoms on the two sides have square-planar coordination environments.



(I)

In the trinuclear nickel(II) complex of the title compound, the central atom Ni2 (Fig. 1) has an axially elongated octahedral coordination of Ni(ON)(ON)(O)(O) type. The two carbonyl O atoms and the two hydrazide N atoms make up the equatorial plane, with Ni2—O2 and Ni2—N2 distances of

2.023 (2) and 2.101 (2) Å, respectively, while the two O atoms of the dimethylformamide molecules occupy the axial positions at longer distances [Ni2—O4 = 2.137 (2) Å]. The square-planar coordination of the outer Ni atoms (Ni1 and Ni1*A* in Fig. 1) is formed by the hydrazine N1, carbonyl O3, phenolate O1 and pyridine N3 atoms. The interatomic distance between the symmetry-related atoms Ni1 and Ni1*A* is 9.3038 (7) Å, which is longer than the Ni···Ni distances [9.2030 (8)–9.1876 (9) Å] in the similar structure of bis[μ-(*N*-butylsalicylhydrazidate)(pyridine)nickel(II)]bispyridinenickel(II) (Yang *et al.*, 2003).

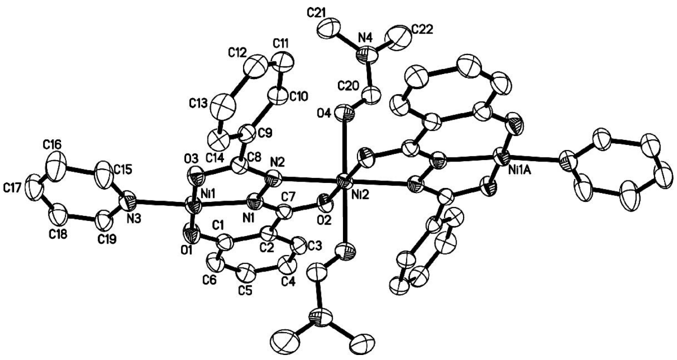


Figure 1

View of the title compound with 30% probability displacement ellipsoids. H atoms and solvent molecules have been omitted for clarity. Symmetry code (which applies also to unlabelled atoms): (A) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$.]

Experimental

2′-Salicylbenzohydrazide (H_2L) was synthesized according to the method of Liu *et al.* (2001). Five drops of pyridine were added to a mixture of $Ni(OAc)_2\cdot 4H_2O$ (0.25 mmol) in methanol (5 ml) and H_2L (0.25 mmol) in dimethylformamide (5 ml). The resulting red solution was stirred for 10 min and then filtered. After standing for 20 d, red block-shaped crystals were separated from the filtrate.

Crystal data

$[Ni_3(C_{30}H_{20}N_4O_6)_2(C_5H_5N)_2(C_2H_6NO)_2]\cdot 2C_3H_7NO\cdot 0.5H_2O$
$M_r = 1142.19$
Monoclinic, $C2/c$
$a = 29.4926$ (15) Å
$b = 10.1678$ (3) Å
$c = 19.7293$ (3) Å
$\beta = 115.389$ (2)°
$V = 5344.9$ (3) Å^3
$Z = 4$
$D_x = 1.419$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5983 reflections
$\theta = 1.5$–27.5°
$\mu = 1.11$ mm^{-1}
$T = 293$ (2) K
Block, red
0.56 × 0.46 × 0.38 mm

Data collection

Rigaku Weissenberg IP diffractometer with 18 kW rotating anode
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.517$, $T_{max} = 0.66$
5983 measured reflections
5983 independent reflections
4085 reflections with $I > 2\sigma(I)$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 38$
$k = 0 \rightarrow 13$
$l = -25 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.137$
$S = 0.98$
5983 reflections
334 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

The C-bound H atoms were positioned geometrically. Atom HO6 of the water molecule was located in a difference Fourier map. H atoms were refined as riding, with C—H = 0.93–0.97 Å, O—H = 0.81 Å and $U_{iso} = 1.2$–$1.5U_{eq}$(parent atom). The occupancies of O6 (water) and HO6 were fixed at 0.5; the water molecule lies on a twofold rotation axis.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1999); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *CrystalStucture* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL/PC*.

This work was supported by the Natural Science Foundation of Fujian Province, China (No.2002J001), and the Foundation for University Key Teachers of the Ministry of Education.

References

Alexiou, M., Tsivikas, I., Dendrinou-Samara, C., Pantazaki, A. A., Trikalitis, P., Lalioti, N., Kyriakidis, D. A. & Kessissoglou, D. P. (2003). *J. Inorg. Biochem.* **93**, 256–264.

Kim, I., Kwak, B. & Lah, M. S. (2001). *Inorg. Chim. Acta*, **317**, 12–20.

Kwak, B., Rhee, H. & Lah, M. S. (2000). *Polyhedron*, **19**, 1985–1994.

Kwak, B., Rhee, H., Park, S. & Lah, M. S. (1998). *Inorg. Chem.* **37**, 3599–3602.

Li, B., Han, D. D., Cheng, G. Z. & Ji, Z, P. (2005). *Inorg. Chem. Commun.* **8**, 216–218.

Lin, S., Liu, S. X., Chen, Z., Lin B. Z. & Gao, S. (2004). *Inorg. Chem.* **43**, 2222–2224.

Lin, S., Liu, S. X., Huang J. Q. & Lin, C. C. (2002). *J. Chem. Soc. Dalton Trans.* pp. 1595–1601.

Lin, S., Liu, S. X. & Lin, B. Z. (2002*a*). *Inorg. Chim. Acta*, **328**, 69–73.

Lin, S., Liu, S. X. & Lin, B. Z. (2002*b*). *Chin. J. Inorg. Chem.* **8**, 1205–1210.

Liu, S. X., Lin, S., Lin, B. Z., Lin, C. C. & Huang, J. Q. (2001). *Angew. Chem. Int. Ed.* **40**, 1084–1087.

Molecular Structure Corporation (1999). *TEXSAN*. Version 1.10. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2002). *Crystal Structure*. Version 3.00. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1998). *SHELXTL/PC*. Bruker AXS Inc., Madison, Wisconsin, USA.

Yang, M. X., Lin, S., Chen, L. J. & Liu, S. X. (2003). *Chin. J. Inorg. Chem.* **19**, 433–436.