

{6,6'-Dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilo-methylidyne)]diphenolato}nickel(II) chloroform solvate**Yu-Ye Yu**Normal College, Jinhua University, Jinhua,
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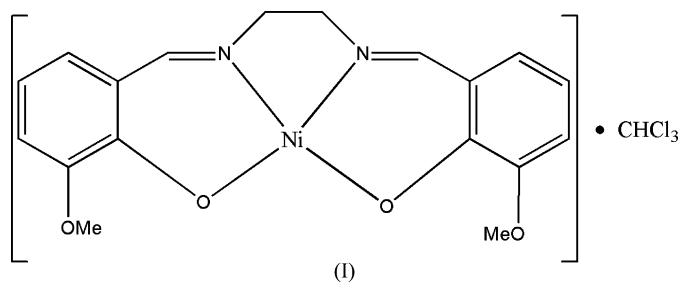
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Key indicatorsSingle-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.041
 wR factor = 0.116
Data-to-parameter ratio = 18.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $[\text{Ni}(\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_4)] \cdot \text{CHCl}_3$, has been synthesized by the solid-state reaction of $\text{Ni}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ and the Schiff base ligand *N,N'*-ethylene-bis(3-methoxy-salicylaldehyde) [abbreviated as $\text{H}_2(3\text{-MeO-salen})$]. The Ni atom adopts a slightly deformed square-planar coordination geometry in which the 3-MeO-salen ligand acts as a *cis*- N_2O_2 donor.

Received 8 February 2006
Accepted 22 March 2006**Comment**

Over the past decades, much attention has been focused on the chemistry of multidentate Schiff base ligands and their complexes, not only because of their ability to coordinate metal ions, but also due to their widespread use in the fields of conducting and magnetic materials, dyes, non-linear optics, catalysis, analytical chemistry, biochemical research and agriculture (Fujita *et al.*, 1994; Choudhary *et al.*, 1999; Santos *et al.*, 2000; Feng 2003). A considerable number of complexes with multidentate Schiff bases containing *O,N* donor atoms have been studied (Clarke *et al.*, 1998; Marchetti *et al.*, 1999). In this paper, we describe the structure of the title compound, (I), which was prepared by a solid-state reaction at room temperature.



X-ray diffraction analysis shows that complex (I) consists of a neutral unit, $[\text{Ni}(3\text{-MeO-salen})]$, and a chloroform molecule. As shown in Fig. 1, the coordination geometry around the Ni atom is roughly square planar, with two N and two O atoms in a *cis*-configuration. The N1/N2/O1/O3 atoms are slightly distorted in a tetrahedral fashion, with a maximum deviation of 0.03 Å from the mean plane of the four atoms. The coordination of (I) is more nearly planar than those of compounds with substituents in the imine bridge (Santos *et al.*, 2000; Azevedo *et al.*, 1999).

It is worth noting that a similar compound, $[\text{Ni}(3\text{-MeO-salen})] \cdot \text{H}_2\text{O}$, (II), has been synthesized in a reaction in solution (Liu *et al.*, 1993). The related bond lengths and bond angles are comparable to those in (I). The successful synthesis of the complex in (I) reveals that the solid-state reaction is an

effective synthetic method for Schiff base complexes and affords us a new strategy to obtain coordination compounds.

Experimental

The Schiff base ligand $H_2(3\text{-MeO-salen})$ was prepared according to a literature method (Na *et al.*, 1988) and was recrystallized from absolute ethanol. $Ni(OAc)_2 \cdot 4H_2O$ (0.2488 g, 1 mmol) and $H_2(3\text{-MeO-salen})$ (0.3283 g, 1 mmol) were weighed accurately and mixed carefully in an agate mortar. The mixture was ground for 20 min and placed in air at room temperature for 24 h to ensure completeness of the reaction. After further grinding at room temperature, the mixture was washed with alcohol (95%) and air-dried (yield 90%). Anal. Calcd. (%) for $C_{18}H_{20}N_2NiO_5$: C, 53.64; H, 5.00; N, 6.95; Ni, 14.56. Found (%): C, 53.28; H, 4.93; N, 6.77; Ni, 14.31. The product (0.2015 g, 0.5 mmol) was dissolved in chloroform (20 ml) and brown single crystals of the title complex suitable for X-ray diffraction were obtained by slow evaporation of the solvent after 5 d (yield 70%; m.p. 436–437 K). Analysis calculated for $C_{19}H_{19}Cl_3N_2NiO_4$: C 45.24, H 3.80, N 5.56, Ni 11.64%; found: C 45.35, H 3.76, N 5.42, Ni 11.89%.

Crystal data

$[Ni(C_{18}H_{18}N_2O_4)] \cdot CHCl_3$	$D_x = 1.603 \text{ Mg m}^{-3}$
$M_r = 504.42$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 16249 reflections
$a = 11.381(2) \text{ \AA}$	$\theta = 3.1\text{--}27.5^\circ$
$b = 13.842(3) \text{ \AA}$	$\mu = 1.34 \text{ mm}^{-1}$
$c = 13.268(3) \text{ \AA}$	$T = 296(2) \text{ K}$
$\beta = 91.43(3)^\circ$	Prism, brown
$V = 2089.5(8) \text{ \AA}^3$	$0.31 \times 0.21 \times 0.19 \text{ mm}$
$Z = 4$	

Data collection

Rigaku RAXIS-RAPID diffractometer	4767 independent reflections
ω scans	3682 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan <i>SADABS</i> (Sheldrick, 1996)	$R_{int} = 0.031$
$T_{min} = 0.509$, $T_{max} = 0.775$	$\theta_{max} = 27.5^\circ$
19480 measured reflections	$h = -14 \rightarrow 14$
	$k = -17 \rightarrow 17$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 0.4559P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.116$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.10$	$\Delta\rho_{max} = 0.93 \text{ e \AA}^{-3}$
4767 reflections	$\Delta\rho_{min} = -0.71 \text{ e \AA}^{-3}$
265 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0030 (5)

Table 1

Selected geometric parameters (\AA , $^\circ$).

Ni1—O1	1.8392 (17)	Ni1—N1	1.846 (2)
Ni1—N2	1.843 (2)	Ni1—O3	1.8458 (17)
O1—Ni1—N2	178.65 (8)	O1—Ni1—O3	84.18 (7)
O1—Ni1—N1	94.75 (8)	N2—Ni1—O3	94.48 (9)
N2—Ni1—N1	86.60 (9)	N1—Ni1—O3	178.85 (9)

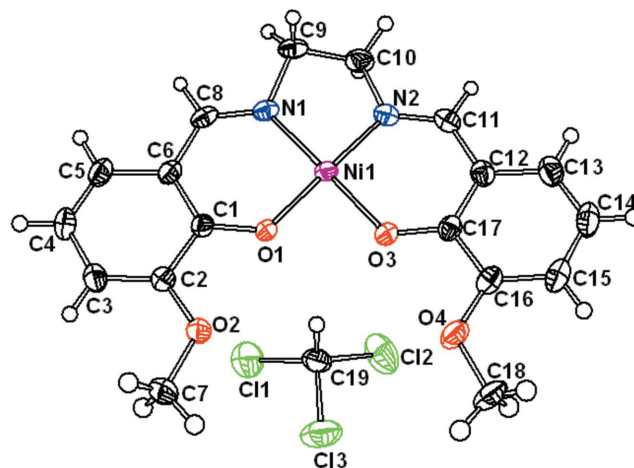


Figure 1

The asymmetric unit of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

H atoms were positioned geometrically and refined as riding, with $C-H = 0.96$ (methyl), 0.97 (other aliphatic) and 0.93 \AA (aromatic), and with $U_{iso}(H)$ set at 1.2 (1.5 for methyl) times $U_{eq}(C)$. The methyl groups were allowed to rotate to fit the electron density.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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