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Di-μ-chloro-bis({2-[(2-hydroxyethyl)iminomethyl]-phenolato-κ^3N,O,O'}nickel(II)) methanol solvate

Kristof Van Hecke,[a] Peter Nockemann,[b] Koen Binnemans[b] and Luc Van Meervelt[a]*

[a]Biomolecular Architecture, Katholieke Universiteit Leuven, Department of Chemistry, Celestijnenlaan 200F, B-3001 Leuven (Heverlee), Belgium, and [b]Laboratory of Coordination Chemistry, Katholieke Universiteit Leuven, Department of Chemistry, Celestijnenlaan 200F, B-3001 Leuven (Heverlee), Belgium

Correspondence e-mail:
luc.vanmeervelt@chem.kuleuven.be

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The title compound, $[Ni_2Cl_2(C_9H_{10}NO_2)_2]\cdot CH_3OH$, is a dinuclear unit built up by two nickel(II) complexes, bridged by two Cl atoms. The coordination geometry around each Ni^{II} atom can be considered as distorted square-pyramidal, with the tridendate chelate Schiff base ligands coordinating in a *trans* conformation through their imine N atom and phenoxy and alkoxy O atoms.

Key indicators

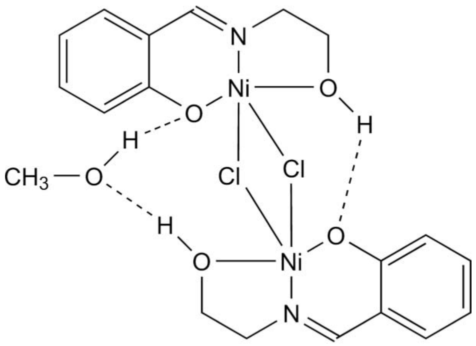
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.009 Å
R factor = 0.041
wR factor = 0.104
Data-to-parameter ratio = 8.6

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

Comment

Ligands derived from the Schiff base proligand 2-[(2-hydroxyethyl)-iminomethyl]phenol (H_2sae or salicylidene-2-ethanolamine) have been incorporated into a number of polynuclear transition metal complexes. After deprotonation, H_2sae yields the potentially tridentate Hsae$^-$ or sae^{2-} anions, which are able to bind in both a chelating and a bridging manner.

Hsae$^-$ or sae^{2-} ligands have previously been incorporated into a number of transition metal complexes, involving Cu^{II}, Ni^{II}, Fe^{III}, Mn^{II}, Mn^{III} and Zn^{II} cations (Maniukiewicz & Bukowska-Strzyzewska, 1994, 1996; Boskovic *et al.*, 2002, 2003, 2004; Hoshino *et al.*, 2003; Chumakov *et al.*, 1989, 2004; Dey *et al.*, 2002*b*; Dey, Rao, Saarenketo *et al.*, 2002), including tetranuclear Cu^{II}, Ni^{II} and Fe^{II} clusters with cubane structures (Chumakov *et al.*, 1990; Boskovic *et al.*, 2002; Hoshino *et al.*, 2002; Dey *et al.*, 2002*a*; Oshio *et al.*, 2004; Sieber *et al.*, 2005). Even a one-dimensional chain-like Mn^{III} complex was found (Zhang *et al.*, 2005). Some of these polynuclear complexes of paramagnetic metals can be of interest because of the potential for these spin clusters to act as single-molecule magnets (SSMs) (Boskovic *et al.*, 2003; Oshio *et al.*, 2004; Sieber *et al.*, 2005). Against this background, we present here the structure of the title compound, (I).



(I)

The structure of compound (I), shows isomorphism with the Cu^{II} complex [Cambridge Structural Database, Version 5.28;

Allen, 2002; reference code ZUWHEE (Maniukiewicz & Bukowska-Strzyzewska, 1996)]. The structure is dimeric, with a distorted square-pyramidal geometry at each Ni^{II} centre (Fig. 1). Each Ni^{II} atom is five-coordinated by a tridentate Shiff base ligand, coordinating through their imine N atom and phenoxy and alkoxy O atoms, and further coordinated by two Cl atoms. Atoms Cl1 and Cl2 bridge atoms Ni1 and Ni2: the Ni1—Cl1, Ni1—Cl2, Ni2—Cl1 and Ni2—Cl2 distances are 2.2809 (15), 2.6704 (16), 2.6703 (15) and 2.2681 (15) Å, respectively. Atom Cl1 is almost basal with respect to the Ni1 complex and apical to the Ni2 complex, and *vice versa* for atom Cl2. The Ni1···Ni2 separation in the dimer is 3.3975 (11) Å.

At one side of the dimer, the two Schiff base ligands are connected through a hydrogen bond between atoms H4*A* (on O4) and O1 (Table 1). Due to this hydrogen bond, the two ligands on opposite sides are too far away from each other for further interaction. As a consequence, a MeOH solvent molecule is inserted to optimize the interactions at this site. A further hydrogen bond is formed between atoms H5*A* (on methanol O5) and O3 (Table 1).

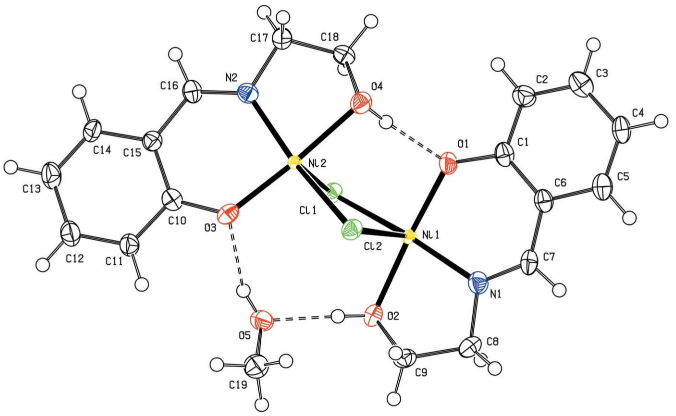


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme and with 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines and H atoms are represented as small spheres of arbitrary radii.

Experimental

The ligand H_2Sae was synthesized by reacting salicylaldehyde (12.2 g, 10 mmol) with ethanolamine (6.1 g, 10 mmol) in a 1:1 molar ratio in methanol (50 ml) under reflux for 1 h. The solvent was evaporated under reduced pressure to give a yellow oil. $[Ni(C_9H_{10}NO_2)Cl]_2$ was synthesized by adding a solution of $NiCl_2\cdot6H_2O$ (0.183 g, 1 mmol) in methanol (20 ml) to a stirred solution of H_2Sae (0.164 g, 1 mmol) in methanol (20 ml). The mixture was stirred at 323 K for 1 h. After the mixture had cooled down to room temperature, it was placed overnight in a refrigerator at 277 K. Green crystals of (I) were precipitated, collected by filtration, washed with cold methanol and chloroform, and dried in a desiccator.

Crystal data

$[Ni_2Cl_2(C_9H_{10}NO_2)_2]\cdot CH_4O$
$M_r = 548.68$
Orthorhombic, $P2_12_12_1$
$a = 9.6183$ (4) Å
$b = 11.7082$ (5) Å
$c = 19.6458$ (8) Å
$V = 2212.37$ (16) Å^3
$Z = 4$
$D_x = 1.647$ Mg m^{-3}
Cu $K\alpha$ radiation
$\mu = 4.63$ mm^{-1}
$T = 100$ (2) K
Plate, green
0.3 × 0.3 × 0.15 mm

Data collection

Bruker SMART 6000 diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.332$, $T_{max} = 0.498$
17341 measured reflections
2421 independent reflections
2119 reflections with $I > 2\sigma(I)$
$R_{int} = 0.076$
$\theta_{max} = 71.3°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.104$
$S = 1.10$
2421 reflections
280 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 0.2378P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.83$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}
Absolute structure: (Flack, 1983), with 1767 Friedel pairs
Flack parameter: 0.49 (4)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O5	0.93 (8)	1.68 (8)	2.591 (6)	167 (7)
O4—H4*A*···O1	0.90 (8)	1.75 (8)	2.637 (6)	169 (7)
O5—H5*A*···O3	0.82	1.98	2.758 (6)	160

H atoms were refined in riding mode, with constrained *X*—H distances depending on both the chemical environment and the temperature, and with $U_{iso}(H)$ fixed at 1.2 times U_{eq} of the parent atom or 1.5 times U_{eq} for methyl groups. Atoms H2*A* and H4*A* were located in a difference Fourier map and their positions were further refined unrestrained [O—H = 0.90 (3)–0.93 (3) Å], with $U_{iso}(H)$ fixed at 1.5 times U_{eq} of the parent atom. The value of the Flack (1983) parameter indicates inversion twinning.

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

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