Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2-(dimethylamino)ethanol-κ^2N,O](pentane-2,4-dionato-κ^2O,O')nickel(II) chloride

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.137
Data-to-parameter ratio = 19.9

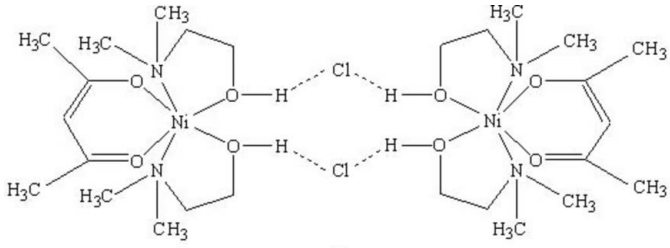
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The Ni atom in the title complex, $[Ni(C_5H_7O_2)(C_4H_{11}NO)_2]Cl$, is in a distorted octahedral coordination environment. Cations are linked into centrosymmetric dimers *via* O—H···Cl hydrogen bonds involving the OH groups of the 2-(dimethylamino)ethanol ligands and the Cl^- anions.

Received 30 June 2005
Accepted 9 September 2005
Online 17 September 2005

Comment

The title compound, (I), is a synthetic precursor for the possible deposition of nickel oxide thin films through aerosol-assisted chemical vapour deposition (AACVD). The molecular structure of complex (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1.



(I)

The complex has a distorted octahedral geometry around the Ni^{II} atom and contains two bidentate chelating dimethylaminoethanol groups and a bidentate acetylacetonate group. The N atoms are in mutually *trans* positions, with an N2—Ni1—N1 angle of 171.43 (10)°. The Ni1—N2 bond length of 2.139 (3) Å is significantly shorter than that of 2.166 (3) Å for Ni1—N1. The Ni—O1, Ni—O2 and Ni—O3 bonds are very similar to the analogous bonds in the related compound $[Ni(acac)_2(dmaeH)]$ (acac is acetylacetonate and dmaeH is dimethylaminoethanol; Williams *et al.*, 2001). Not surprisingly,

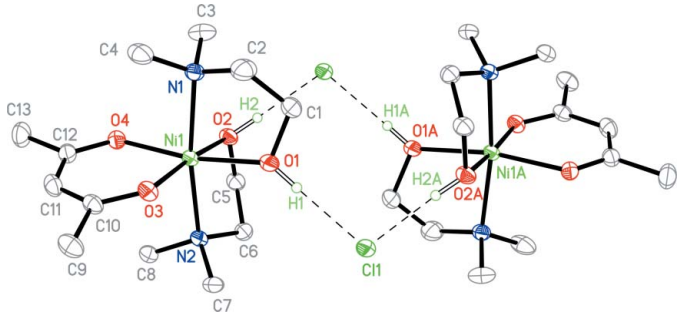


Figure 1
The hydrogen-bonded (dashed lines) dimer of the title compound, showing 30% displacement ellipsoids. Atoms labelled with the suffix A are related by the symmetry operator $(2-x, 1-y, 1-z)$.

the Ni—O bonds of the coordinated dmaeH groups are longer [2.080 (2) and 2.106 (2) Å] than the Ni—O(acac) bonds [2.014 (2) and 2.015 (2) Å]. The *cis* O—Ni—O and O—Ni—N bond angles in (I) are close to the ideal octahedral value of 90°, lying in the range 89.07 (9)–93.84 (9)°, with the exception of the bite angles of the chelating dmaeH groups [80.30 (10) and 81.10 (9)°], and 97.08 (10)° for N2—Ni1—O4. Distortions of the *trans* O—Ni—O angles from the ideal 180° are also evident [169.95 (9)–172.31 (10)°].

In the crystal structure, molecules are linked *via* O—H···Cl hydrogen bonds to form centrosymmetric dimers involving the O—H groups of the dmaeH ligands and the Cl^- anions [H1—Cl 2.15 (4) and H2—Cl1^{i} 2.18 (4) Å, and O1—H1···Cl1 172 (4) and O2—H2···Cl1^{i} 161 (6)°; symmetry code: (i) $2 - x$, $1 - y$, $1 - z$].

Experimental

Bis(2,4-pentanedionato)nickel(II), [Ni(acac)$_2$] (0.5 g, 1.95 mmol), was reacted with dimethylaminoethanol (dmaeH; 0.391 ml, 3.9 mmol) in the presence of methoxytin(II) chloride (0.7 g, 3.9 mmol), [ClSnOCH$_3$] in toluene under argon. The resulting product was recrystallized from tetrahydrofuran at 263 K to give crystals of [Ni(acac)(dmaeH)$_2$]Cl, (I).

Crystal data

$[Ni(C_5H_7O_2)(C_4H_{11}NO)_2]Cl$	D_x = 1.375 Mg m^{-3}
M_r = 371.54	Mo $K\alpha$ radiation
Monoclinic, $P2_1/a$	Cell parameters from 25977 reflections
a = 13.6400 (3) Å	θ = 2.9–27.5°
b = 8.7900 (3) Å	μ = 1.25 mm^{-1}
c = 15.2310 (5) Å	T = 150 (2) K
β = 100.6970 (10)°	Block, colourless
V = 1794.40 (9) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker Nonius KappaCCD area-detector diffractometer	4059 independent reflections
ω scans	3191 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (Blessing, 1995)	R_{int} = 0.093
T_{min} = 0.706, T_{max} = 0.886	θ_{max} = 27.5°
27270 measured reflections	$h = -17 \rightarrow 17$
	$k = -11 \rightarrow 11$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 1.8862P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.137	$\Delta\rho_{max}$ = 1.00 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.86 e Å^{-3}
4059 reflections	
204 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	2.080 (2)	Ni1—N1	2.166 (3)
Ni1—O2	2.106 (2)	Ni1—N2	2.139 (3)
Ni1—O3	2.014 (2)	O1—H1	0.86 (4)
Ni1—O4	2.015 (2)	O2—H2	0.86 (2)
N1—Ni1—N2	171.43 (10)	N2—Ni1—O2	81.10 (9)
O1—Ni1—O4	169.95 (9)	N2—Ni1—O3	91.48 (10)
O2—Ni1—O3	172.31 (9)	N2—Ni1—O4	97.08 (10)
N1—Ni1—O1	80.30 (10)	O1—Ni1—O2	90.96 (10)
N1—Ni1—O2	93.78 (9)	O1—Ni1—O3	91.42 (10)
N1—Ni1—O3	93.84 (9)	O2—Ni1—O4	89.07 (9)
N1—Ni1—O4	89.66 (10)	O3—Ni1—O4	89.86 (9)
N2—Ni1—O1	92.86 (10)		

H atoms on O atoms were located in a difference map and refined isotropically. C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.98–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms. The highest peak is located 0.96 Å from atom C2 and 1.61 Å from atom N1.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinski & Minor, 1997); data reduction: *DENZO* (Otwinski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank the Pakistan Science Foundation, Islamabad 45320, Pakistan, for funding [contract/grant No. PSF/R&D/C-QU/Chem(218)].

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