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{2-[(2-Hydroxy-5-methylphenyl)iminomethyl]-4,6-dinitrophenolato(2–)-*O*,*N*,*O*′}tris(3-methylpyridine-*N*)nickel(II)

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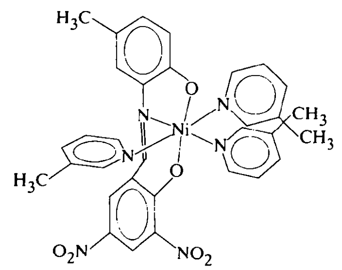
Abstract

In the title compound, [Ni($C_{14}H_9N_3O_6$)(C_6H_7N)$_3$], the coordination polyhedron around the Ni^{II} atom is a slightly distorted octahedron, with one N atom of the tridentate ligand and three N atoms of three monodentate ligands forming the equatorial plane. The apical positions are occupied by the two phenolic O atoms of the tridentate ligand. The Ni—O distances are 2.013 (2) and 2.008 (2) Å, and the Ni—N distances range from 2.043 (2) to 2.168 (3) Å. The *cis* bond angles at Ni^{II} range from 82.75 (9) to 96.2 (1)°. There are intramolecular, as well as intermolecular, hydrogen bonds in the structure.

Comment

The behaviour of tridentate ONO or ONS Schiff base ligands in the presence of monodentate ligands has been studied previously with four-coordinate nickel(II). In such complexes, the coordination of nickel is square planar or distorted square planar (Atakol *et al.*, 1996; Ülkü *et al.*, 1996; Tahir *et al.*, 1996; Ercan *et al.*, 1996). The title complex, (I), was studied to determine the effect on coordination when the tridentate ligand has nitro substituents.



(I)

As can be seen from Fig. 1, if a nitro-substituted tridentate Schiff base ligand is present in the reaction, the coordination around Ni^{II} becomes sixfold. We attribute this to the inductive effects of the nitro groups, whereby the ability of the tridentate ligands to donate electron density through their O and N atoms is reduced; the increase in the coordination number of the Ni^{II} centre offsets this potential electron deficit. The equatorial plane of the slightly distorted octahedral environment around Ni^{II} involves the coordination of one N atom of the tridentate 2-[(2-hydroxy-5-methylphenyl)iminomethyl]-4,6-dinitrophenolate ligand and three N atoms of three monodentate 3-methylpyridine ligands. The two phenolic O atoms of the tridentate ligand occupy the apical positions. The equatorial plane is defined by atoms N3, N4, N5 and N6 [maximum deviation 0.045 (2) Å], and Ni^{II} is located only 0.0022 (4) Å from this plane. The Ni—O bond lengths are practically equal [2.008 (2) and 2.013 (2) Å]. The Ni—N distances in the equatorial plane range from 2.043 (2) to 2.168 (3) Å. Among the *cis* bond angles, O6—Ni—N3 [82.75 (9)°] has the greatest deviation from 90°.

A comparison of the Ni—N and Ni—O bond lengths of the title complex with those of a nickel complex of 3,5-dinitrosalicylaldehyde [Ni—N 2.059 (2)–2.084 (3) and Ni—O 2.072 (2) Å; Tahir *et al.*, 1997] shows that they have similar values.

The dihedral angle between the planes of the two chelating moieties (O1, C1, C6, C7, N3 and N3, C8,

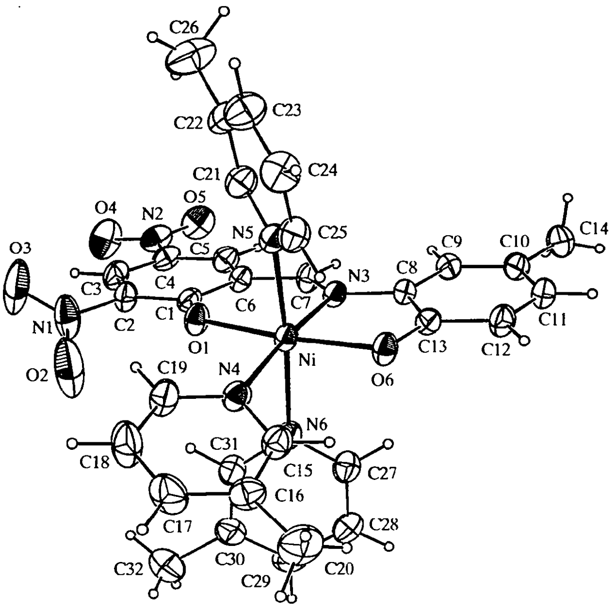


Fig. 1. Plot of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level; H atoms are shown as small circles of arbitrary radii.

C13, O6) has a value of 6.9 (8)°. The dihedral angles between the equatorial plane and the three monodentate rings, *i.e.* N4 and C15–C19, N5 and C21–C25, and N6 and C27–C31, are 75.7 (1), 127.8 (1) and 62.1 (1)°, respectively.

Four intramolecular and three intermolecular C—H···O hydrogen bonds (Table 2) stabilize the structure.

Experimental

3,5-Dinitrosalicylaldehyde (0.212 g, 1 mmol) and 2-amino-4-methylphenol (0.123 g, 1 mmol) were dissolved in MeCN (50 ml). This solution was heated to boiling point and 3-methylpyridine (1.5 ml) was added. The resulting solution was mixed with a solution of $Ni(CH_3COO)_2.4H_2O$ (0.25 g, 1 mmol) in hot MeOH (30 ml) and set aside for 1 d at room temperature. The precipitated crystals were dried in air and were suitable for X-ray data collection.

Crystal data

$[Ni(C_{14}H_9N_3O_6)(C_6H_7N)_3]$
$M_r = 653.343$
Triclinic
$P\overline{1}$
$a = 10.309\ (2)$ Å
$b = 11.451\ (2)$ Å
$c = 13.361\ (3)$ Å
$\alpha = 96.706\ (2)°$
$\beta = 93.109\ (3)°$
$\gamma = 105.188\ (2)°$
$V = 1505.9\ (5)$ Å^3
$Z = 2$
$D_x = 1.436$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 11.43–20.76°$
$\mu = 0.697$ mm^{-1}
$T = 295$ K
Prism
0.40 × 0.15 × 0.12 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
$T_{min} = 0.90$, $T_{max} = 0.92$
5844 measured reflections
5549 independent reflections

4131 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.46°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 0$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 120 min
intensity decay: <1.0%

Refinement

Refinement on F
$R = 0.042$
$wR = 0.047$
$S = 0.83$
4131 reflections
406 parameters
H atoms: see below
$w = 1/[\sigma(F^2) + (0.02F)^2 + 1.0]$; $w = 0$ if $F^2 < 2\sigma(F^2)$

$(\Delta/\sigma)_{max} = 0.0002$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.10$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Ni—O1	2.013 (2)	Ni—N4	2.097 (3)
Ni—O6	2.008 (2)	Ni—N5	2.146 (3)
Ni—N3	2.043 (2)	Ni—N6	2.168 (3)
O1—Ni—O6	172.61 (9)	O6—Ni—N6	92.3 (1)
O1—Ni—N3	89.96 (9)	N3—Ni—N4	174.0 (1)
O1—Ni—N4	92.58 (9)	N3—Ni—N5	96.2 (1)
O1—Ni—N5	88.9 (1)	N3—Ni—N6	90.4 (1)
O1—Ni—N6	89.0 (1)	N4—Ni—N5	89.2 (1)
O6—Ni—N3	82.75 (9)	N4—Ni—N6	84.2 (1)
O6—Ni—N4	94.80 (9)	N5—Ni—N6	173.04 (9)
O6—Ni—N5	90.6 (1)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15···O6	0.95	2.47	3.070 (4)	121
C17—H17···O3^{i}	0.95	2.52	3.393 (6)	153
C19—H19···O1	0.95	2.53	3.076 (5)	117
C27—H27···O6	0.95	2.57	3.099 (5)	115
C28—H28···O3^{ii}	0.95	2.57	3.310 (5)	135
C31—H31···O1	0.95	2.54	3.076 (4)	116
C26—H261···O4^{iii}	1.07	2.42	3.426 (6)	157

Symmetry codes: (i) $-x, -y, -z$; (ii) $x, 1 + y, z$; (iii) $1 - x, -y, 1 - z$.

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically at 0.95 Å from their parent atoms and the H atoms of C14, C20, C26 and C32 were refined for a few cycles. Finally, a riding model was adopted for all H atoms, with $U_{eq}(H) = 1.3U_{eq}(C)$.

Data collection and cell parameters: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction, program used to solve structure, program used to refine structure, molecular graphics and software used to prepare material for publication: *MolEN* (Fair, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1157). Services for accessing these data are described at the back of the journal.

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Bis(2,6-di-*tert*-butyl-4-methylphenolato-*O*)-(η^5-methylcyclopentadienyl)(tetrahydrofuran-*O*)ytterbium

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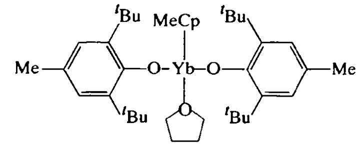
Abstract

The crystals of the title complex, [Yb($C_{15}H_{23}O$)$_2$(C_6H_7)(C_4H_8O)], are monoclinic (space group $P2_1/c$). The molecule has pseudo-tetrahedral geometry. The central metal atom is coordinated by one methylcyclopentadienyl (MeCp) ligand and three O atoms, with a Yb···Cp(centroid) distance of 2.355 (5) Å and Yb—O(Ar) distances of 2.040 (4) and 2.078 (4) Å.

Comment

In recent years, there has been considerable interest in developing aryloxo auxiliary ligands for the preparation of organometallic complexes of lanthanide elements (Deacon *et al.*, 1991; Zhou *et al.*, 1992; Evans *et al.*, 1996). In the course of our work on the syntheses of homo- and heteroaryloxo lanthanide complexes (Qi *et al.*, 1994, 1995), the title complex, (I), was isolated from the reaction of anhydrous $YbCl_3$ with ArONa in the molar ratio 1:2, followed by reaction with an equivalent of MeCpNa.



(I)

The molecule structure of (I) is shown in Fig. 1; it appears that the C8 ^{t}Bu substituent is affected by rotational disorder about the C2—C8 bond, resulting in exceptionally high displacement parameters for the C9, C10 and C11 atoms.

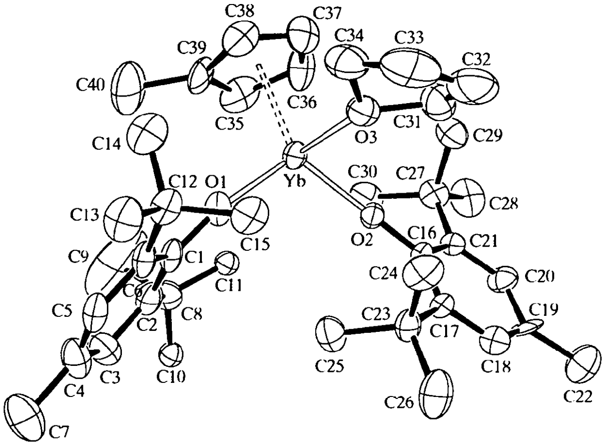


Fig. 1. The molecular structure of (I) showing 40% probability displacement ellipsoids. H atoms have been omitted for clarity.

The title complex consists of one Yb atom, two aryloxo ligands, one methylcyclopentadienyl group and one tetrahydrofuran (thf) ligand; the coordination number of the Yb atom is six. The complex can be considered as a quasi-four-coordinate complex. The Yb—C distances range from 2.598 (6) to 2.689 (7) Å, the longest being that involving the C atom bonded to the methyl group. The Yb···Cp(centroid) distance is 2.355 (5) Å. The average Yb—O(Ar) distance is 2.059 (4) Å and the Yb—O(thf) distance is 2.317 (4) Å. Interestingly, the Yb···H14 distance is quite short (2.46 Å); it seems that this close contact is a result of agostic interaction between the Yb and H14 atoms.

The angles O1—Yb—O2, O1—Yb···Cp(centroid) and O2—Yb···Cp(centroid) are 107.8 (1), 112.9 (9) and