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

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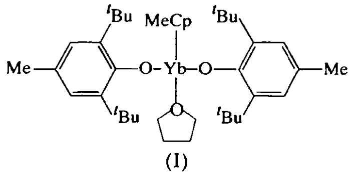
Abstract

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 ᵗBu substituent is affected by rotational disorder about the C2—C8 bond, resulting in exceptionally high displacement parameters for the C9, C10 and C11 atoms.

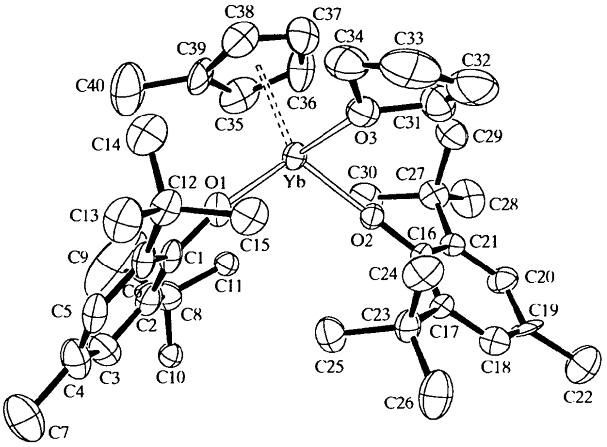


Fig. 1. The molecular structure of (I) showing 40% probability displacement ellipsoids. H atoms have been omitted for clarity.

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

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133.6 (9)°, respectively. The Yb—O1—C1 and Yb—O2—C16 angles are 168.0 (4) and 178.8 (4)°, respectively. These angles are different from those in the related compounds $[(C_5Me_5)Ce(OC_6H_3{}^t\!Bu_2)_2]$ (Heeres *et al.*, 1989) and $[(C_5Me_5)Y(OC_6H_3{}^t\!Bu_2)_2]$ (Schaverien *et al.*, 1991), which each display an agostic interaction between the metal and a ^{t}Bu methyl group of one of the aryloxide ligands.

Experimental

The organolanthanide complex described in this paper is very sensitive to air and moisture. Consequently, all manipulations were carried out under argon by Schlenk techniques. Crystals were obtained from tetrahydrofuran–toluene at 268 K. A suitable crystal was sealed in a thin-walled glass capillary for crystal structure determination.

Crystal data

$[Yb(C_{15}H_{23}O)_2(C_6H_7)(C_4H_8O)]$
$M_r = 762.96$
Monoclinic
$P2_1/c$
$a = 12.206\,(5)$ Å
$b = 15.259\,(3)$ Å
$c = 20.718\,(8)$ Å
$\beta = 100.51\,(3)°$
$V = 3794\,(2)$ Å^3
$Z = 4$
$D_x = 1.336$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 23 reflections
$\theta = 11.9$–$13.2°$
$\mu = 2.498$ mm^{-1}
$T = 293$ K
Prismatic
0.45 × 0.20 × 0.20 mm
Red

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (Walker & Stuart, 1983)
$T_{min} = 0.484$, $T_{max} = 0.607$
4951 measured reflections
4671 independent reflections

3666 reflections with $F > 2\sigma(F)$
$R_{int} = 0.078$
$\theta_{max} = 22.51°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -22 \rightarrow 15$
3 standard reflections every 200 reflections intensity decay: 0.34%

Refinement

Refinement on F
$R = 0.029$
$wR = 0.039$
$S = 1.34$
3666 reflections
398 parameters
H atoms not refined
$w = 1/\sigma^2(F_o) = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max} = 0.011$

$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 0.665
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Yb—O(1)	2.040 (4)	Yb—C(39)	2.689 (7)
Yb—O(2)	2.078 (4)	Yb···Cp(centroid)	2.355 (5)
Yb—O(3)	2.317 (4)	O(1)—C(1)	1.361 (7)
Yb—C(35)	2.623 (7)	O(2)—C(16)	1.350 (7)
Yb—C(36)	2.598 (6)	O(3)—C(31)	1.464 (8)
Yb—C(37)	2.616 (6)	O(3)—C(34)	1.463 (8)
Yb—C(38)	2.662 (6)		
O(1)—Yb—O(2)	107.8 (1)	O(2)—Yb—C(39)	151.5 (2)
O(1)—Yb—O(3)	108.9 (2)	O(2)—Yb···Cp(centroid)	133.6 (9)
O(1)—Yb—C(35)	104.9 (2)	O(3)—Yb—C(35)	126.1 (2)
O(1)—Yb—C(36)	135.6 (2)	O(3)—Yb—C(36)	100.2 (2)
O(1)—Yb—C(37)	131.2 (2)	O(3)—Yb—C(37)	74.9 (2)
O(1)—Yb—C(38)	100.7 (2)	O(3)—Yb—C(38)	82.2 (2)
O(1)—Yb—C(39)	86.8 (2)	O(3)—Yb—C(39)	112.1 (2)
O(1)—Yb···Cp(centroid)	112.9 (9)	O(3)—Yb···Cp(centroid)	99.6 (5)
O(2)—Yb—O(3)	86.7 (1)	Yb—O(1)—C(1)	168.0 (4)
O(2)—Yb—C(35)	120.8 (2)	Yb—O(2)—C(16)	178.8 (4)
O(2)—Yb—C(36)	106.7 (2)	Yb—O(3)—C(31)	121.5 (4)
O(2)—Yb—C(37)	121.0 (2)	Yb—O(3)—C(34)	130.3 (4)
O(2)—Yb—C(38)	151.4 (2)		

The value $\theta_{max} = 22.51°$ was governed by disorder and the quality of the crystal. An empirical absorption correction using the program *DIFABS* (Walker & Stuart, 1983) was applied, which, in the absence of ψ-scan measurements, at the time seemed appropriate for such an unstable crystal sealed in a capillary. The structure was solved by direct methods with *SAPI*91 (Fan, 1991) and expanded using Fourier techniques (Beurskens *et al.*, 1992). *TEXSAN* (Molecular Structure Corporation, 1989) was used to refine the structure. The non-H atoms were refined anisotropically. H atoms were included but not refined.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1292). Services for accessing these data are described at the back of the journal.

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Co-Crystallized (Macrocyclic tetraamine)-bis(thiocyanato-*N*)nickel(II) Complexes

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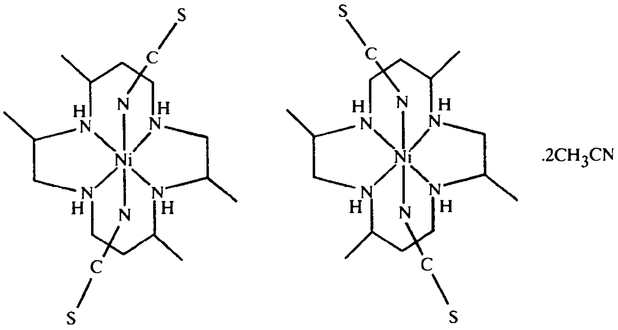
Abstract

In (2,7,9,14-tetramethyl-1,4,8,11-tetraazacyclotetradecane-κ^4N)bis(thiocyanato-*N*)nickel(II) (2,5,9,12-tetramethyl-1,4,8,11-tetraazacyclotetradecane-κ^4N)bis(thiocyanato-*N*)nickel(II) diacetonitrile solvate, $[NiL^1(NCS)_2][NiL^2(NCS)_2].2CH_3CN$, where L^1 and L^2 are isomeric tetraazacyclotetradecane ligands ($C_{14}H_{32}N_4$), each of the co-crystallized complex molecules has an NiN_4 square plane involving the four N atoms of the secondary amine. In each of the two isomeric complexes, the two thiocyanate ligands coordinate axially through their N atoms.

Comment

In the structure of $[NiL^1(NCS)_2][NiL^2(NCS)_2].2CH_3CN$, (I), shown in Fig. 1, the ligand skeleton of each complex contains two *gauche* five-membered chelate rings and two six-membered chelate rings in chair conformations.



(I)

The Ni^{II} ion in each complex occupies a crystallographic inversion centre and therefore the methyl

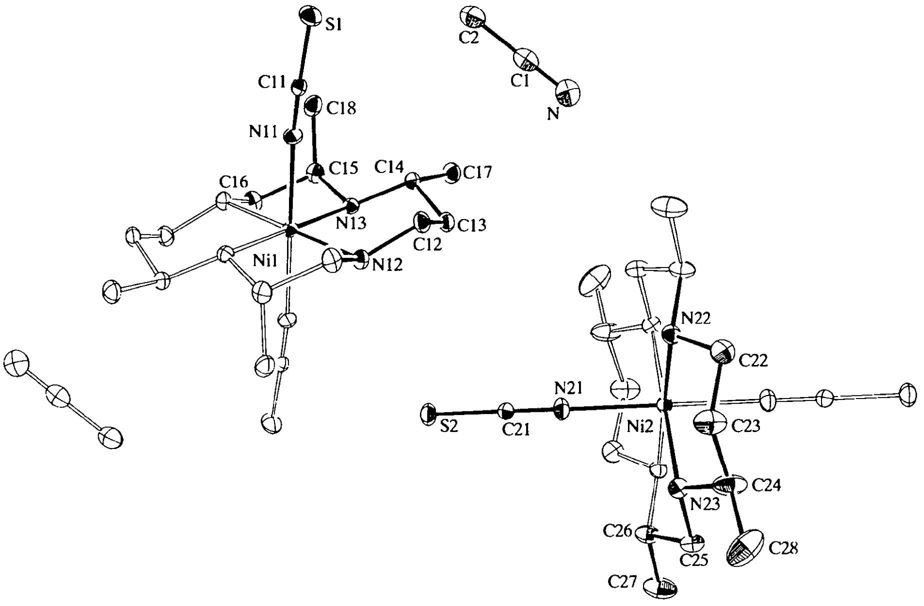


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound showing the atom-labelling scheme. The asymmetric unit is indicated by filled bonds. The displacement ellipsoids are drawn at the 40% probability level and H atoms have been omitted for clarity.