

$(\eta^5\text{-}tert\text{-Butylcyclopentadienyl})\text{chloro}[N,N\text{-}(2,6\text{-diisopropylphenyl})\text{pentane-}2,4\text{-diiminato}]ytterbium(\text{III})$ 

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## Key indicators

Single-crystal X-ray study  
 $T = 193\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$   
 $R$  factor = 0.025  
 $wR$  factor = 0.059  
Data-to-parameter ratio = 16.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title complex,  $[\text{Yb}(\text{C}_9\text{H}_{12})(\text{C}_{26}\text{H}_{42}\text{N}_2)\text{Cl}]$  or  $\{[(2,6\text{-Pr}^i_2\text{C}_6\text{H}_3)\text{NC}(\text{CH}_3)]_2\text{CH}\}(\text{tBuC}_5\text{H}_4)\text{YbCl}$ , exists as a monomeric molecule; the six-membered chelate ring adopts a boat conformation. The Yb atom is six-coordinate and the shape of the molecule approximates to a three-legged piano stool.

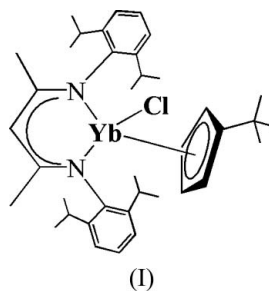
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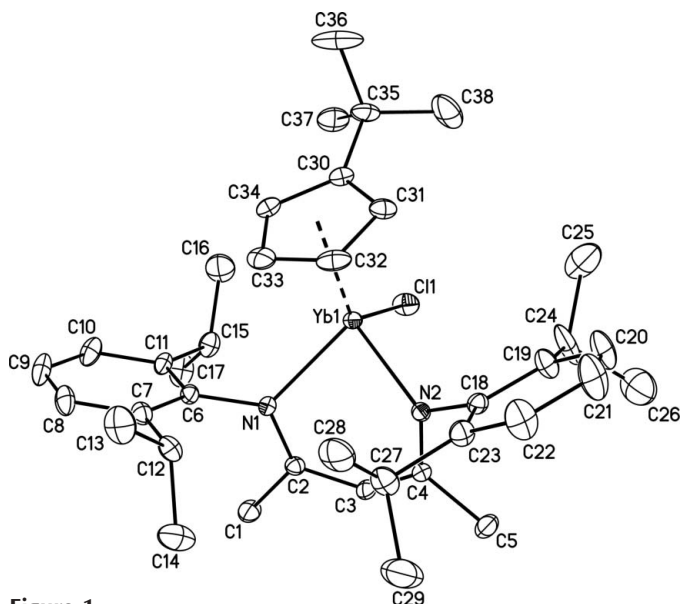
## Comment

The use of  $\beta$ -diketiminates as supporting ligands in both main group and transition metal coordination chemistry has been reviewed (Bourget-Merle *et al.*, 2002). On the other hand, the use of  $\beta$ -diketiminates as ancillary ligands in the synthesis of lanthanide complexes still remains relatively poorly explored (Eisenstein *et al.*, 2003; Neculai *et al.*, 2003; Lauterwasser *et al.*, 2004; Yao *et al.*, 2002, 2003). Recently, we investigated the synthesis and reactivity of lanthanide systems, and have ascertained that the  $\{[(2,6\text{-Pr}^i_2\text{C}_6\text{H}_3)\text{NC}(\text{CH}_3)]_2\text{CH}\}$  anion is ideal for the synthesis of mixed-ligand lanthanide complexes (Yao *et al.*, 2002). In the present study, we describe the product of the reaction of  $\{[(2,6\text{-Pr}^i_2\text{C}_6\text{H}_3)\text{NC}(\text{CH}_3)]_2\text{CH}\}\text{YbCl}_2$  with  $(\text{tBuC}_5\text{H}_4)\text{Na}$  in a 1:1 molar ratio in tetrahydrofuran, *viz.* the title compound,  $\{[(2,6\text{-Pr}^i_2\text{C}_6\text{H}_3)\text{NC}(\text{CH}_3)]_2\text{CH}\}(\text{tBuC}_5\text{H}_4\text{-}4)\text{YbCl}$ , (I).



Compound (I) exists as an unsolvated monomer. The central metal atom is coordinated by one  $\text{tBuC}_5\text{H}_4$  ring, two N atoms from the  $\beta$ -diketimate ligand, and one Cl atom, the coordination giving the central metal a formal coordination number of six. The coordination geometry about the ytterbium center approximates to a three-legged piano stool, with the two N atoms of the chelating  $\beta$ -diketimate anion and a Cl atom constituting the 'legs' of the molecule. The backbone of the ligand (N1, C2, C3, C4 and N2) and the Yb atom form a six-membered ring with a boat conformation.

The  $\beta$ -diketimate ligand is symmetrically coordinated to the Yb atom [ $\text{Yb}-\text{N} = 2.241$  (2) and  $2.246$  (2)  $\text{\AA}$ ]; the  $\text{Yb}-\text{N}$  distances are similar to those reported in the literature (Bourget-Merle *et al.*, 2002). The average  $\text{Yb}-\text{C}(\text{ring})$  distance is  $2.597$  (3)  $\text{\AA}$ . The bond dimensions of the molecule are comparable with those found in  $\{[(2,6\text{-Pr}^i_2\text{C}_6\text{H}_3)\text{NC}(\text{CH}_3)]_2\text{CH}\}\text{YbCl}_2$ .



**Figure 1**  
The molecule of (I), showing 25% probability displacement ellipsoids. H atoms have been omitted for clarity.

$\text{NC}(\text{CH}_3)_2\text{CH}(\text{MeC}_5\text{H}_4)\text{YbCl}$  (Yao *et al.*, 2002). The Yb—C2 and Yb—C4 distances are comparable with those [2.986 (6) and 3.180 (9) Å] found in  $\eta^2\text{-C}_5\text{H}_5$ -bridged  $(\text{C}_5\text{Me}_5)_2\text{Sm}(\mu\text{-C}_5\text{H}_5)\text{Sm}(\text{C}_5\text{Me}_5)_2$  (Evans & Ulibarri, 1987), as well as those [2.814 (4)–3.148 (6) Å] found in  $\eta^6, \eta^1$ -chelating  $[\text{Yb}(\text{Odpp})_3]_2$  (Odpp is 2,6-diphenylphenolate) (Deacon *et al.*, 1990). The complex may be considered to involve  $\pi$  coordination of the  $\beta$ -diketiminato ligand. The bond distances C2—C3, C3—C4, N1—C2 and N2—C4 fall between single- and double-bond values (Allen *et al.*, 1987), and they indicate significant electron delocalization within the  $\pi$ -system. The compound exhibits IR bands at 1551 and 1524  $\text{cm}^{-1}$ , which implies partial C=N double-bond character (Richeson *et al.*, 1987).

## Experimental

The title complex is moderately sensitive to air and moisture. Consequently, all manipulations were carried out under pure argon using Schlenk techniques. Complex (I) was obtained from the reaction of  $[\{(2,6\text{-Pr}^i_2\text{C}_6\text{H}_3)\text{NC}(\text{CH}_3)_2\text{CH}\}\text{YbCl}_2]$  with one equivalent of  $(\text{tBuC}_5\text{H}_4)\text{Na}$  in tetrahydrofuran (THF), followed by crystallization from a toluene–THF solution at 263 K. A crystal suitable for analysis was sealed in a thin-walled glass capillary. Analysis calculated for  $\text{C}_{38}\text{H}_{54}\text{ClN}_2\text{Yb}$ : C 61.07, H 7.30, N 3.75, Yb 23.15; found: C 60.86, H 7.22, N 3.73, Yb 23.05. IR (KBr pellet,  $\text{cm}^{-1}$ ): 3061 (*m*), 2962 (*m*), 2868 (*m*), 1592 (*m*), 1551 (*s*), 1524 (*s*), 1460 (*m*), 1435 (*s*), 1365 (*s*), 1155 (*m*), 1022 (*m*), 934 (*m*), 836 (*m*), 787 (*s*), 756 (*m*).

### Crystal data

$[\text{Yb}(\text{C}_9\text{H}_{12})(\text{C}_{26}\text{H}_{42}\text{N}_2)\text{Cl}]$	$Z = 2$
$M_r = 747.32$	$D_x = 1.369 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 10.858 (2) \text{ \AA}$	Cell parameters from 8023 reflections
$b = 11.803 (2) \text{ \AA}$	$\theta = 3.0\text{--}25.3^\circ$
$c = 15.378 (3) \text{ \AA}$	$\mu = 2.68 \text{ mm}^{-1}$
$\alpha = 97.688 (3)^\circ$	$T = 193 (2) \text{ K}$
$\beta = 104.337 (4)^\circ$	Block, orange
$\gamma = 104.019 (4)^\circ$	$0.34 \times 0.26 \times 0.11 \text{ mm}$
$V = 1812.8 (6) \text{ \AA}^3$	

### Data collection

Rigaku Mercury diffractometer	6255 reflections with $I > 2\sigma(I)$
$\omega$ scans	$R_{\text{int}} = 0.024$
Absorption correction: multi-scan (Jacobson, 1998)	$\theta_{\text{max}} = 25.4^\circ$
$T_{\text{min}} = 0.463$ , $T_{\text{max}} = 0.757$	$h = -13 \rightarrow 12$
18042 measured reflections	$k = -14 \rightarrow 12$
6585 independent reflections	$l = -18 \rightarrow 18$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0254P)^2 + 1.8363P]$
$R[F^2 > 2\sigma(F^2)] = 0.025$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.059$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 1.09$	$\Delta\rho_{\text{max}} = 1.36 \text{ e \AA}^{-3}$
6585 reflections	$\Delta\rho_{\text{min}} = -0.73 \text{ e \AA}^{-3}$
393 parameters	H-atom parameters constrained

**Table 1**

Selected geometric parameters (Å, °).

Yb1—N1	2.241 (2)	Yb1—C30	2.662 (3)
Yb1—N2	2.246 (2)	Yb1—C2	3.028 (3)
Yb1—Cl1	2.4927 (9)	Yb1—C4	3.030 (3)
Yb1—C32	2.554 (3)	N1—C2	1.342 (4)
Yb1—C33	2.557 (3)	N2—C4	1.332 (4)
Yb1—C31	2.606 (3)	C2—C3	1.403 (4)
Yb1—C34	2.607 (3)	C3—C4	1.407 (4)
N1—Yb1—N2	85.73 (9)	C2—N1—Yb1	112.93 (19)
N1—Yb1—Cl1	105.01 (7)	C4—N2—Yb1	113.20 (19)
N2—Yb1—Cl1	105.05 (7)	C2—C3—C4	131.2 (3)

H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2$  or 1.5 times  $U_{\text{eq}}(\text{C})$ . The highest electron-density peak is 0.97 Å from atom H24.

Data collection: *CRYSTALCLEAR* (Rigaku, 1999); cell refinement: *CRYSTALCLEAR*; data reduction: *CrystalStructure* (Rigaku/MSK, 2000–2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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