APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2005; **19**: 398–399
Published online in Wiley InterScience (www.interscience.wiley.com). DOI:10.1002/aoc.855

Crystallographic report

Crystal structure of a homoleptic lanthanide guanidinate complex: $[Ph_2NC(NCy)_2]_3Yb{\cdot}2PhCH_3$

Liying Zhou[1], Yingming Yao[1], Yong Zhang[1], Hongting Sheng[1], Mingqiang Xue[1] and Qi Shen[1,2]*

[1]Department of Chemistry and Chemical Engineering, Suzhou University, Suzhou 215006, People's Republic of China
[2]State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, People's Republic of China

Received 19 September 2004; Accepted 12 October 2004

The title complex, $[Ph_2NC(NCy)_2]_3Yb{\cdot}2PhCH_3$ is a monomer with a six-coordinate ytterbium center ligated by six nitrogen atoms of three chelating bidentate guanidinate ligands. The coordination geometry around the lanthanide ion is best described as a distorted trigonal prism. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: synthesis; crystal structure; organolanthanide; guanidinate ligand

COMMENT

The title complex is a monomer with a six-coordinate ytterbium center ligated by six nitrogen atoms of three chelating bidentate guanidinate ligands, and the coordination geometry around the lanthanide ion is best described as a distorted trigonal prism. As shown in Fig. 1, the Yb–N bond lengths, varying from 2.362(6) to 2.387(9) Å, are slightly longer than the values reported for $\{CyNC[N(SiMe_3)_2]NCy\}_2YbN(SiMe_3)_2$ (2.301(15) to 2.329(13) Å)[1] and $[(Me_3Si)_2NC(N^iPr)_2]_2Yb(\mu\text{-}Cl)_2Li(THF)_2$ (2.295(3) to 2.332(3) Å).[2] The C–N distances within the chelating guanidinate ligands are approximately equivalent and significantly shorter than the C–N single bond distances, indicating that the π-electrons in the present complexes are delocalized within the N–C–N fragment. Bond angles of N(1)–Yb(1)–N(2), N(4)–Yb(1)–N(5) and N(7)–Yb(1)–N(8) are 57.2(3)°, 57.0(2)° and 57.7(2)° respectively, which are comparable to the corresponding angles in $[CyNC(Ph)NCy]_3Yb \cdot 2THF$ (58.1(2)° and 57.98(17)°).[3]

*Correspondence to: Qi Shen, Department of Chemistry and Chemical Engineering, Suzhou University, Suzhou 215006, People's Republic of China.
E-mail: qshen@suda.edu.cn
Contract/grant sponsor: Chinese National Natural Science Foundation; Contract/grant number: 20272040.

EXPERIMENTAL

Synthesis of the title complex

A Schlenk flask was charged with $[Li(THF)_4][Yb(NPh_2)_4]$[4] (4.56 g, 4.0 mmol) in 30 ml tetrahydrofuran (THF) and a stirring bar. Then, *N*,*N*′- dicyclohexylcarbodiimide (2.47 g, 12.0 mmol) in 20 ml THF was added slowly to it. The color of the solution slowly changed to yellow. The solution was then stirred for another 48 h and evaporated to dryness in vacuum. The residue was extracted with toluene and LiCl was removed by centrifugation. The solution was concentrated and stored at the room temperature for crystallization. The title complex was obtained as yellow crystals (4.62 g, 78%). M.p. 287–289 °C (dec.). ^{1}H NMR (400 MHz, C_6D_6, 25 °C): δ = 6.88–7.40 (m, 42 H, Ph); 3.16–3.98 (m, 6 H, unique H-Cy); 0.34–2.15 (br, 60 H, C_6H_{10}). Anal. Found: C, 71.96; H, 8.05; N, 8.79; Yb, 10.73. Calc. for $C_{89}H_{112}N_9Yb$ (1480.96): C, 72.18; H, 7.62; N, 8.51; Yb, 11.69%. IR (KBr pellet, cm^{-1}): 3430 (s), 2928 (s), 2855 (m), 1640 (s), 1590 (m), 1493 (m), 1451 (w), 1308 (w), 1235 (w), 1154 (w), 1076 (w), 1030 (w), 891 (w), 752 (m), 694 (m).

Crystallography

Intensity data for the title compound were collected at 193.1 K on a Rigaku Mercury CCD area detector for a yellow prism crystal with dimensions of 0.41 × 0.59 × 0.20 mm^3. Crystallographic data: $C_{89}H_{112}N_9Yb$, M = 1480.96, monoclinic, $P2_1$ (#4), a = 14.436(3), b = 22.005(3), c = 14.192(3) Å, β = 117.712(7), V = 3991.2(14) $Å^3$, D_c = 1.232 g cm^{-3}, Z = 2; 82 036 data collected, 17 053 unique data ($3.2 \le \theta \le 27.5°$), 13 188 observed data with ($I > 3.00\sigma(I)$). R_{obs} = 0.055, wR = 0.142 (all data), ρ_{max} = 6.80 e^- $Å^{-3}$, ρ_{min} = −3.70 e^- $Å^{-3}$. Programs used: CrystalClear, SIR97, DIRDIF99, CrystalStructure 3.5.1 and CRYSTALS Issue 10. CCDC number: 228 392.

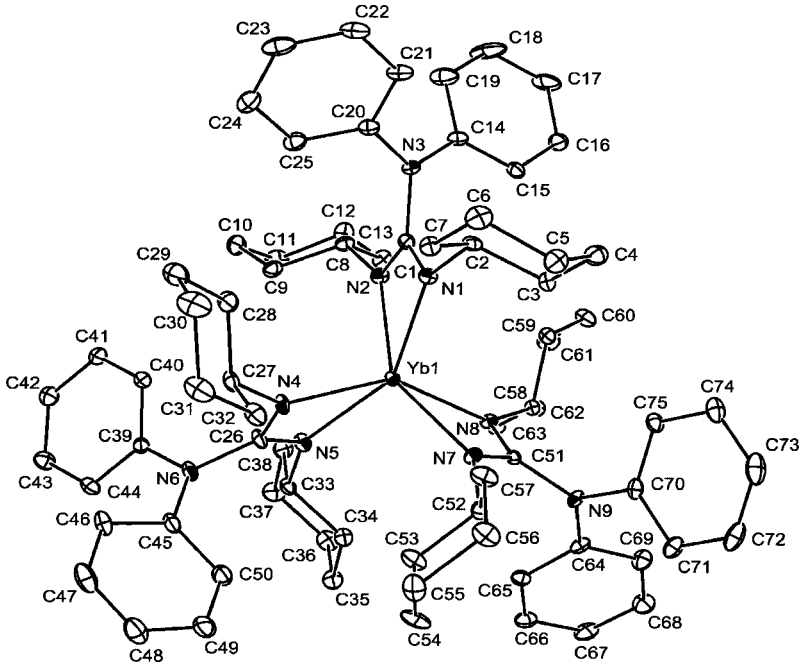

Figure 1. Molecular structure of the title complex. Key geometric parameters: Yb(1)–N(1) 2.364(8), Yb(1)–N(2) 2.378(9), Yb(1)–N(4) 2.381(9), Yb(1)–N(5) 2.387(9), Yb(1)–N(7) 2.364(6), Yb(1)–N(8) 2.362(6), Yb(1)–C(1) 2.77(1), Yb(1)–C(26) 2.79(1), Yb(1)–C(51) 2.794(6), N(1)–C(1) 1.31(1), N(2)–C(1) 1.35(1), N(4)–C(26) 1.34(1), N(5)–C(26) 1.33(1), N(7)–C(51) 1.358(9), N(8)–C(51) 1.35(1) Å; N(1)–Yb(1)–N(2) 57.2(3), N(4)–Yb(1)–N(5) 57.0(2), N(7)–Yb(1)–N(8) 57.7(2)°.

Acknowledgements

We are indebted to the Chinese National Natural Science Foundation (20272040) for financial support.

REFERENCES

1. Zhou YL, Yap GPA, Richeson DS. *Organometallics* 1998; **17**: 4387.
2. Luo YJ, Yao YM, Shen Q, Yu KB, Weng LH. *Eur. J. Inorg. Chem.* 2003; 318.
3. Luo YJ, Yao YM, Shen Q. *J. Organometal. Chem.* 2002; **662**: 144.
4. Yao YM, Lu XH, Shen Q, Yu KB. *J. Chem. Cryst.* 2004; **34**(4): 275.