

Supporting Information for:
Rare-earth metal complexes having an unusual
indolyl-1,2-dianion through C-H activation with a novel
 $\eta^1:(\mu_2-\eta^1:\eta^1)$ bonding with metals

Xiancui Zhu,^a Shuangliu Zhou,^a Shaowu Wang,^{*,a,b} Yun Wei,^a Lijun Zhang,^a Fenhua Wang,^a
Shaoyin Wang,^{a,c} and Zhijun Feng^a

^a Anhui Laboratory of Molecule-Based Materials, Institute of Organic Chemistry, School of Chemistry and Materials Science, Anhui Normal University, Wuhu, Anhui 241000, P. R. China. Fax: +86-553-3883517; Tel: +86-553-5910015; E-mail: swwang@mail.ahnu.edu.cn

^b State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Shanghai 200032, P. R. China. E-mail: swwang@mail.ahnu.edu.cn

^c Department of Chemistry, Wannan Medical College, Wuhu, Anhui 241003, P. R. China

Table of Contents

1. General Information.....	02
2. Preparations of 1– 7.....	02
3. Molecular Structures of the Complexes.....	05
4. X-ray crystallography and Crystallographic Data.....	07

General Information. All syntheses and manipulations of air- and moisture-sensitive materials were performed under dry argon and oxygen-free atmosphere using standard Schlenk techniques or a glovebox. All solvents were refluxed and distilled over sodium benzophenone ketyl under argon prior to use unless otherwise noted. $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{RE}^{\text{III}}(\mu\text{-Cl})\text{Li}(\text{THF})_3$ (RE = Y, Yb),¹ 3-(^tBu-N=CH)C₈H₅NH,² 3-(^tBu-NHCH₂)C₈H₅NH,³ (2,6-ⁱPr₂C₆H₃)NHC(H)=N(2,6-ⁱPr₂C₆H₃)⁴ were prepared according to literature methods. Elemental analyses data were obtained on a Perkin-Elmer 2400 Series II elemental analyzer. ¹H NMR and ¹³C NMR spectra for analyses of compounds were recorded on a Bruker AV-300 NMR spectrometer (300 MHz for ¹H; 75.0 MHz for ¹³C in C₆D₆ for lanthanide complexes. Chemical shifts (δ) were reported in ppm. *J* values are reported in Hz. IR spectra were recorded on a Shimadzu FTIR-8400s spectrometer (KBr pellet).

Preparation of

$\{[\eta^1:(\mu_2-\eta^1):\eta^1]:\eta^1\text{-3-(t-BuN=CH)C}_8\text{H}_4\text{N}]\text{Y}_2(\mu_2\text{-Cl})_2(\text{THF})[\text{N}(\text{SiMe}_3)_2](\eta^1:\eta^1\text{-}[\mu\text{-}\eta^5:\eta^2\text{-3-(t-BuN=CH)C}_8\text{H}_5\text{N}]_2\text{Li})\}_2$ (**1**)

To a toluene (10.0 mL) solution of 3-(^tBu-N=CH)C₈H₅NH (0.41 g, 2.03 mmol) was added a toluene (20.0 mL) solution of $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{Y}^{\text{III}}(\mu\text{-Cl})\text{Li}(\text{THF})_3$ (1.12 g, 1.35 mmol) at room temperature. After the reaction mixture was heated to reflux for 24 h, the color of the solution was gradually changed from white to redish green. The solvent was evaporated under reduced pressure. The residue was extracted with *n*-hexane (2×10 mL). The combined extractions were concentrated to about 10.0 mL. The light yellow crystals were obtained at 0 °C for several days (0.39 g, 53 % yield). m.p. 201 °C (dec.). Anal. Calc. for C₄₉H₇₀Cl₂LiN₇OSi₂Y₂·2C₆H₁₄: C, 58.27; H, 7.86; N, 7.80. Found: C, 58.05; H, 7.93; N, 7.85. δ_{H} (300 MHz, C₆D₆, ppm): 8.59 (s, 3H, N=CH), 8.34 (d, 2H, *J* = 8.01 Hz), 7.98 (s, 3H), 7.62 (m, 3H), 7.33 (m, 3H), 7.10 (m, 3H), 3.31 (s, 4H, -CH₂O), 1.40 (s, 4H, -CH₂), 0.94 (s, 27H, C(CH₃)₃), 0.61 (s, 9H, Si(CH₃)₃), 0.09 (s, 9H, Si(CH₃)₃). δ_{C} (75 MHz, C₆D₆, ppm): 157.4 (N=CH), 139.8, 129.0, 126.3, 125.4, 121.9, 121.6, 118.5, 115.5, 56.2 (C(CH₃)₃), 29.6 (C(CH₃)₃), 5.1, 2.3 (SiMe₃). IR (KBr pellet, cm⁻¹): 2926w, 2866w, 1627s, 1531s, 1456s, 1381m, 1354w, 1236s, 1205s, 1138s, 1116s, 933m, 829w, 742w, 574w, 420w.

Preparation of

$\{[\eta^1:(\mu_2-\eta^1):\eta^1]:\eta^1\text{-3-(t-BuN=CH)C}_8\text{H}_4\text{N}]\text{Yb}_2(\mu_2\text{-Cl})_2(\text{THF})[\text{N}(\text{SiMe}_3)_2](\eta^1:\eta^1\text{-}[\mu\text{-}\eta^5:\eta^2\text{-3-(t-BuN=CH)C}_8\text{H}_5\text{N}]_2\text{Li})\}_2$ (**2**)

This compound was isolated as red crystals in 58% yield by treatment of $[(\text{Me}_3\text{Si})_2\text{N}]_3\text{Yb}^{\text{III}}(\mu\text{-Cl})\text{Li}(\text{THF})_3$ (1.34 g, 1.47 mmol) with 3-(^tBu-N=CH)C₈H₅NH (0.44 g, 2.20 mmol) following procedures similar to those used for the preparation of **1**. m.p. 185 °C (dec.). Anal. Calc. for C₄₉H₇₀Cl₂LiN₇OSi₂Yb₂·2C₆H₁₄: C, 51.39; H, 6.93; N, 6.88. Found: C, 51.12; H, 7.38; N, 7.15. IR (KBr pellet, cm⁻¹): 2966m, 2868w, 1629s, 1531s, 1454m, 1359w, 1236m, 1205m, 1116s, 1010s, 933s, 837w, 742s, 570w, 422w.

Preparation of $\{\eta^4:\eta^2:\eta^1\text{-3-(t-BuN=CH)C}_8\text{H}_5\text{N}]\text{Li}_2[\text{N}(\text{SiMe}_3)_2]\}_2$ (**3**)

To a toluene (10.0 mL) solution of 3-(^tBu-N=CH)C₈H₅NH (0.49 g, 2.47 mmol) was added a toluene (20.0 mL) solution of LiN(SiMe₃)₂ (0.83 g, 4.94 mmol) at room temperature. After the reaction mixture was stirred at reflux for 24 h, the solvent was evaporated under reduced pressure. The residue was extracted with *n*-hexane (10 mL). The colorless crystals were obtained upon

standing the solution at room temperature for 12 h (0.64 g, 69%). m.p. 208 °C. Anal. Calc. for $C_{38}H_{66}Li_4N_6Si_4$: C, 61.09; H, 8.90; N, 11.25. Found: C, 61.15; H, 8.86; N, 11.17. δ_H (300 MHz, C_6D_6 , ppm): 8.62 (s, 1H, N=CH), 8.23 (s, 1H), 8.09 (d, 1H, $J = 7.83$ Hz), 7.81 (d, 1H, $J = 7.44$ Hz), 7.41 (d, 1H, $J = 7.41$ Hz), 7.31 (d, 1H, $J = 7.20$ Hz), 1.03 (s, 9H, $C(CH_3)_3$), 0.09 (s, 9H, $Si(CH_3)_3$), 0.01 (s, 9H, $Si(CH_3)_3$). δ_C (75.0 MHz, C_6D_6 , ppm): 158.0 (N=CH), 149.0, 137.0, 129.2, 122.2, 121.1, 118.2, 117.8, 112.1, 56.3 ($C(CH_3)_3$), 29.7 ($C(CH_3)_3$), 4.8, 2.3 ($SiMe_3$). IR (KBr pellet, cm^{-1}): 2966m, 2349m, 1629s, 1581m, 1531s, 1456s, 1381m, 1359w, 1236m, 1205m, 1138s, 1066m, 1010s, 933m, 829w, 605w, 574w.

Preparation of $[\mu\{-\{\eta^1:\eta^1:\eta^1:\eta^1\text{-3-(t-BuNHCH}_2\text{)Ind}\}_2Li\}Y[N(SiMe_3)_2]_2]$ (4)

This compound was isolated as white crystals in 45% yield by treatment of $[(Me_3Si)_2N]_3Y^{III}(\mu\text{-Cl})Li(THF)_3$ (0.89 g, 1.07 mmol) with 3-(^tBu-NHCH₂)C₈H₅NH (0.33 g, 1.61 mmol) following procedures similar to those used for the preparation of **1**. m.p. 212 °C (dec.). Anal. Calc. for $C_{38}H_{70}LiN_6Si_4Y \cdot 2THF$: C, 57.35; H, 9.00; N, 8.72. Found: C, 57.26; H, 8.54; N, 8.81. δ_H (300 MHz, C_6D_6 , ppm): 7.79 (m, 2H), 7.35-6.90 (m, 8H), 3.62 (d, 4H, $J = 7.62$ Hz, -CH₂), 1.18 (s, 18H, $C(CH_3)_3$), 0.22 (s, 18H, $Si(CH_3)_3$), 0.14 (s, 18H, $Si(CH_3)_3$). δ_C (75 MHz, C_6D_6 , ppm): 145.2, 134.1, 121.4, 120.8, 118.1, 117.6, 110.2, 109.9, 45.7 ($C(CH_3)_3$), 29.4 (-CH₂), 21.9 ($C(CH_3)_3$), 1.1, 0.15 ($SiMe_3$). IR (KBr pellet, cm^{-1}): 2962m, 2385s, 2349s, 1456m, 1354m, 1336m, 1261w, 1091m, 1020w, 864m, 800w, 740w.

Preparation of $[\mu\{-\{\eta^1:\eta^1:\eta^1:\eta^1\text{-3-(t-BuNHCH}_2\text{)Ind}\}_2Li\}Yb[N(SiMe_3)_2]_2]$ (5)

This compound was isolated as pale yellow crystals in 50% yield by treatment of $[(Me_3Si)_2N]_3Yb^{III}(\mu\text{-Cl})Li(THF)_3$ (1.21 g, 1.33 mmol) with 3-(^tBu-NHCH₂)C₈H₅NH (0.40 g, 1.99 mmol) following procedures similar to those used for the preparation of **1**. m.p. 205 °C (dec.). Anal. Calc. for $C_{38}H_{70}LiN_6Si_4Yb \cdot 2THF$: C, 52.74; H, 8.27; N, 8.02. Found: C, 53.05; H, 7.93; N, 7.85. IR (KBr pellet, cm^{-1}): 2962m, 2904s, 2349s, 1456m, 1363m, 1261w, 1182m, 1095w, 1018w, 933m, 800m, 738w.

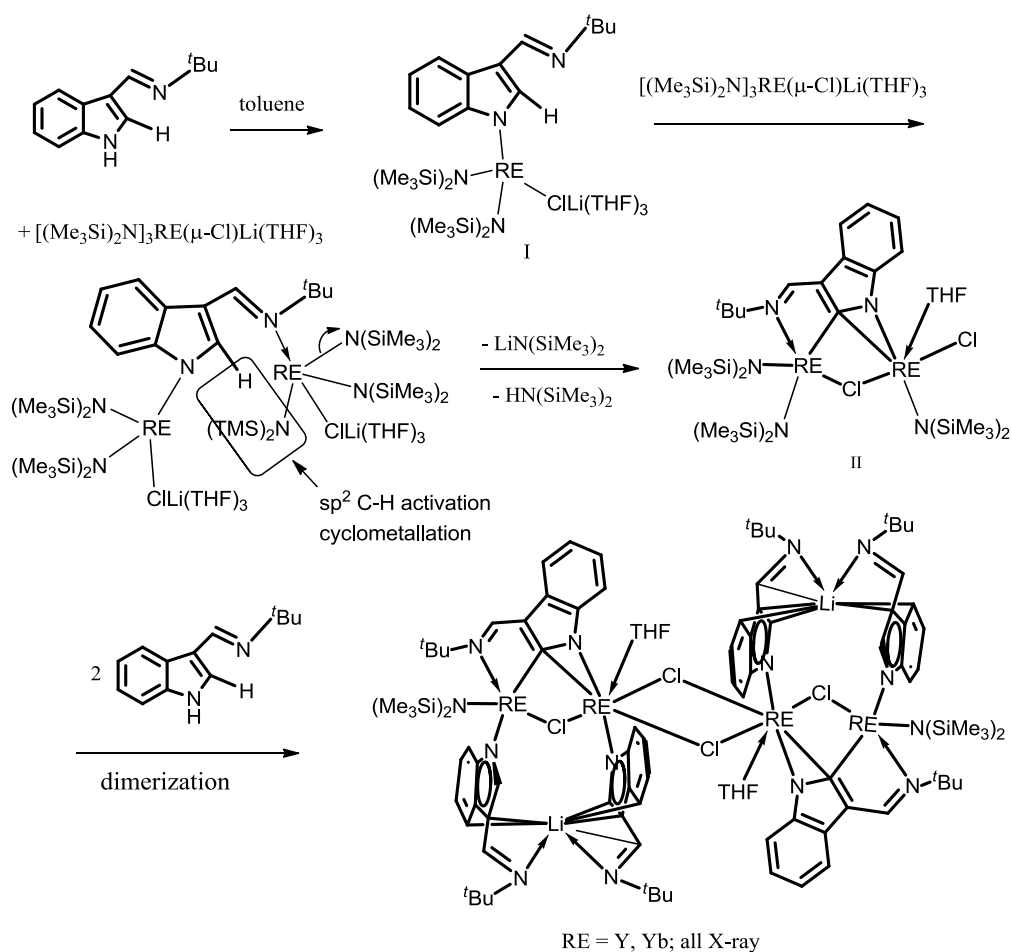
Preparation of $\{[3\text{-}(t\text{-BuN=CH)C}_8\text{H}_5\text{N}]_2Y[(2,6\text{-}^i\text{Pr}_2\text{C}_6\text{H}_3\text{N})_2(\text{CH})](\text{THF})_2\}$ (6)

To a toluene (10.0 mL) solution of **1** (0.58 g, 0.26 mmol) was added a toluene (10.0 mL) solution of (2,6-ⁱPr₂C₆H₃)NHC(H)=N(2,6-ⁱPr₂C₆H₃) (0.10 g, 0.26 mmol) at room temperature. The reaction mixture was heated to reflux for 12 h. Then the solvent was evaporated under reduced pressure. The residue was extracted with 10 mL *n*-hexane. The colorless crystals were obtained at 0 °C for several days (0.13 g, 45 % yield). m.p. 158-159 °C. Anal. Calc. for $C_{59}H_{81}N_6O_2Y \cdot C_7H_8$: C, 72.90; H, 8.25; N, 7.73. Found: C, 72.99; H, 8.28; N, 7.79. ¹H NMR (300 MHz, C_6D_6 , ppm): δ 9.08 (s, 1H, NCHN), 8.91 (d, 2H, $J = 6.96$ Hz), 8.53 (s, 2H, N=CH), 8.02 (d, 2H, $J = 7.17$ Hz), 7.47-7.02 (m, 12H), 3.95-3.90 (m, 4H, -CH(CH₃)₂), 3.11 (s, 8H, -CH₂O), 1.56-1.49 (m, 24H), 1.42 (s, 8H, -CH₂), 1.26-1.24 (m, 18H). ¹³C NMR (75 MHz, C_6D_6 , ppm): δ 171.8 (NCHN), 147.8 (N=CH), 144.6, 142.5, 133.6, 128.6, 124.5, 122.5, 122.2, 120.8, 120.2, 119.0, 115.7, 112.8, 70.2, 55.3, 30.7, 29.3, 27.5, 25.1, 23.5, 22.2, 21.8, 13.1. IR (KBr pellet, cm^{-1}): 1664s, 1625m, 1585w, 1529m, 1456s, 1354w, 1286m, 1236m, 1118w, 1066s, 798m, 742w.

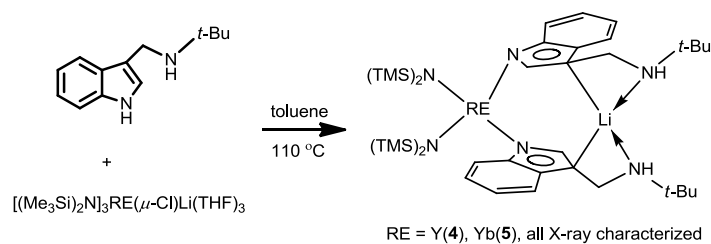
Preparation of $\{[3\text{-}(t\text{-BuN=CH)C}_8\text{H}_5\text{N}]_2Yb[(2,6\text{-}^i\text{Pr}_2\text{C}_6\text{H}_3\text{N})_2(\text{CH})](\text{THF})_2\}$ (7)

This compound was isolated as red crystals in 39% yield by treatment of **2** (0.74 g, 0.29 mmol) with (2,6-ⁱPr₂C₆H₃)NHC(H)=N(2,6-ⁱPr₂C₆H₃) (0.11 g, 0.29 mmol) following procedures similar to

those used for the preparation of **6**. m.p. 147-149 °C. Anal. Calc. for $C_{59}H_{81}N_6O_2Yb$: C, 66.65; H, 7.56; N, 7.79. Found: C, 66.78; H, 7.75; N, 7.88. IR (KBr pellet, cm^{-1}): 1664s, 1626m, 1529m, 1454s, 1354w, 1286m, 1236m, 1138m, 1058w, 933w, 798m, 742m, 574w.



Scheme S1. Proposed Pathway for the formation of complexes **1** and **2**



Scheme S2 Reactions of 3-(*t*-butylaminomethylene)indole with rare-earth metal amides

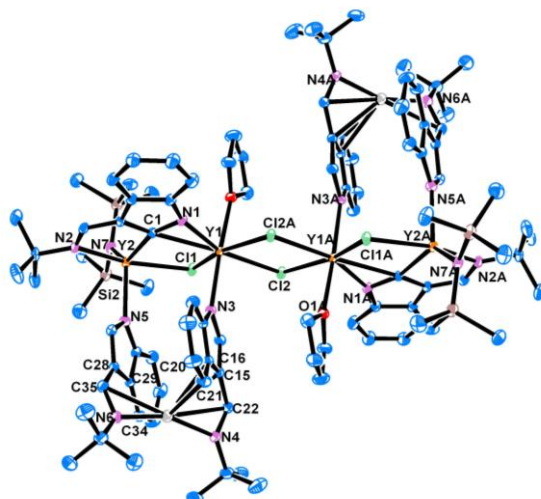


Fig. S1. Structure of complex **1**. Hydrogen atoms were omitted for clarity. Selected bond distances (Å) and angles (°): Y(1)-N(1) 2.216(7), Y(1)-N(3) 2.344(7), Y(1)-O(1) 2.354(6), Y(1)-Cl(1) 2.662(2), Y(1)-C(1) 2.665(8), Y(1)-Cl(2) 2.741(2), Y(2)-N(7) 2.238(7), Y(2)-N(5) 2.305(7), Y(2)-N(2) 2.385(7), Y(2)-C(1) 2.551(8), Y(2)-Cl(1), 2.670(2), Li(1)-N(6) 1.99(2), Li(1)-N(4) 2.01(2), Li(1)-C(22) 2.53(2), Li(1)-C(15) 2.55(2), Li(1)-C(16) 2.56(2), Li(1)-C(21) 2.60(2), Li(1)-C(35) 2.61(2), Li(1)-C(34) 2.67(2), Li(1)-C(29) 2.74(2), Li(1)-C(28), 2.76(2), Y(1)-Cl(1)-Y(2) 92.01(7), N(3)-Y(1)-Cl(2) 89.51(18), N(7)-Y(2)-N(5) 119.3(3), N(5)-Y(2)-Cl(1) 90.1(2).

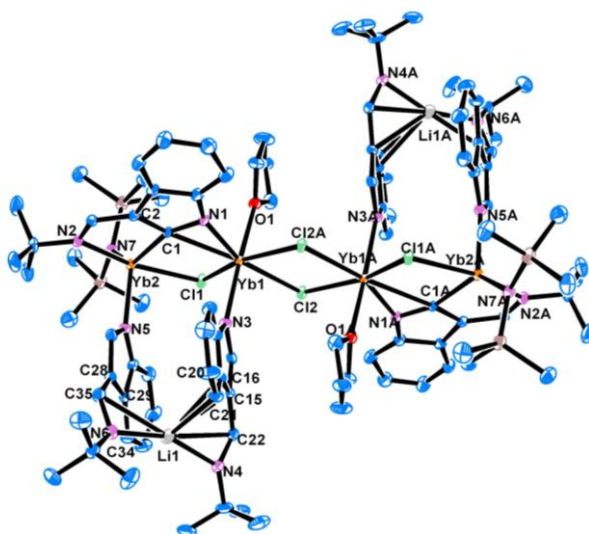


Fig. S2. Structure of complex **2**. Hydrogen atoms were omitted for clarity. Selected bond distances (Å) and angles (°): Yb(1)-N(1) 2.182(9), Yb(1)-N(3) 2.303(10), Yb(1)-O(1) 2.345(9), Yb(1)-Cl(1) 2.632(3), Yb(1)-C(1) 2.654(10), Yb(1)-Cl(2) 2.702(3), Yb(2)-N(7) 2.201(9), Yb(2)-N(5) 2.267(10), Yb(2)-N(2) 2.371(9), Yb(2)-C(1) 2.506(12), Yb(2)-Cl(1), 2.641(3), Li(1)-N(6) 2.00(3), Li(1)-N(4) 2.00(3), Li(1)-C(22) 2.50(3), Li(1)-C(15) 2.59(3), Li(1)-C(16) 2.55(3), Li(1)-C(21) 2.67(4), Li(1)-C(35) 2.63(3), Li(1)-C(34) 2.69(4), Li(1)-C(29) 2.73(4), Li(1)-C(28), 2.78(3), Yb(1)-Cl(1)-Yb(2) 92.42(9), N(3)-Yb(1)-Cl(2) 89.1(3), N(7)-Yb(2)-N(5) 119.4(4), N(5)-Yb(2)-Cl(1) 90.4(3).

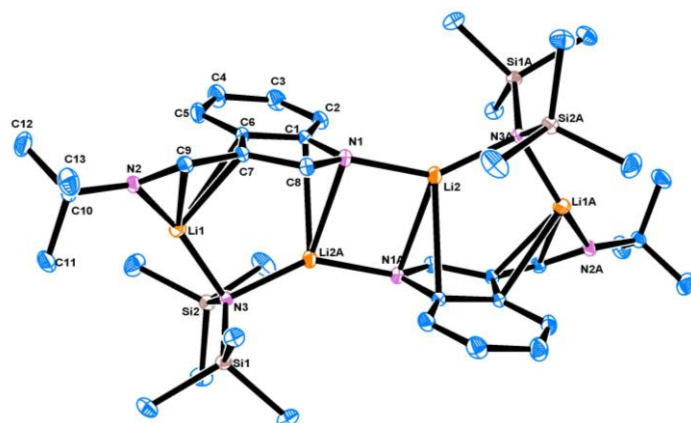


Fig. S3. Structure of complex **3**. Hydrogen atoms were omitted for clarity. Selected bond distances (Å) and angles (°): Li(1)-N(3) 1.962(4), Li(1)-N(2) 2.007(4), Li(1)-C(9) 2.498(5), Li(1)-C(7) 2.458(5), Li(1)-C(6) 2.464(4), N(3)-Li(2A) 2.028(4), N(1)-Li(2A) 2.598(5), C(1)-Li(2A) 2.669(5), N(2)-C(9) 1.273(3), N(3)-Li(1)-N(2) 147.8(2), N(3A)-Li(2)-N(1A) 126.42(19).

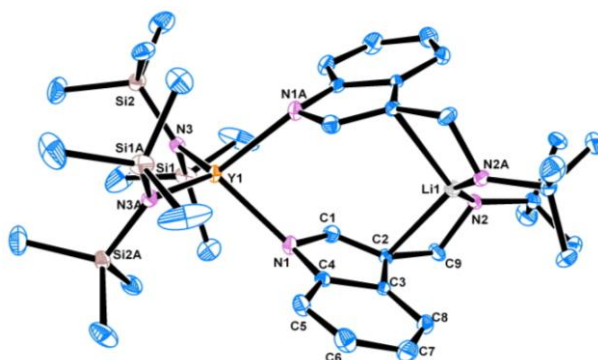


Fig. S4. Structure of complex **4**. Hydrogen atoms were omitted for clarity. Selected bond distances (Å) and angles (°): Y(1)-N(3) 2.236(3), Y(1)-N(1) 2.306(4), Li(1)-N(2) 2.061(5), Li(1)-C(2) 2.524(7), N(2)-C(9) 1.479(5), N(1A)-Y(1)-N(1) 83.73(17), N(3A)-Y(1)-N(3) 120.14(16), N(2)-Li(1)-C(2A) 137.8(3), N(2)-Li(1)-N(2A) 149.3(6).

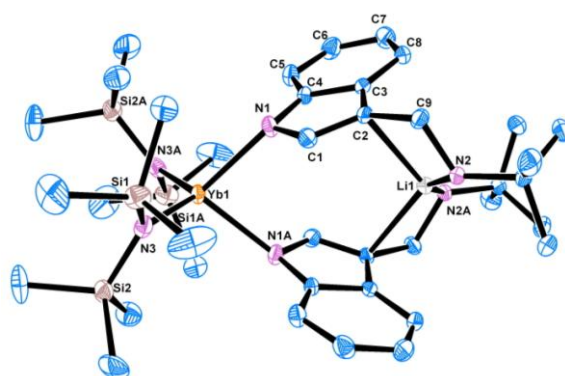


Fig. S5. Structure of complex **5**. Hydrogen atoms were omitted for clarity. Selected bond distances (Å) and angles (°): Y(1)-N(3) 2.205(7), Yb(1)-N(1) 2.275(7), N(2)-Li(1) 2.069(8), C(2)-Li(1) 2.499(13), N(2)-C(9) 1.492(10), N(1A)-Yb(1)-N(1) 83.5(4), N(3A)-Yb(1)-N(3) 121.7(4), N(2)-Li(1)-C(2A) 138.0(4), N(2)-Li(1)-N(2A) 147.7(11).

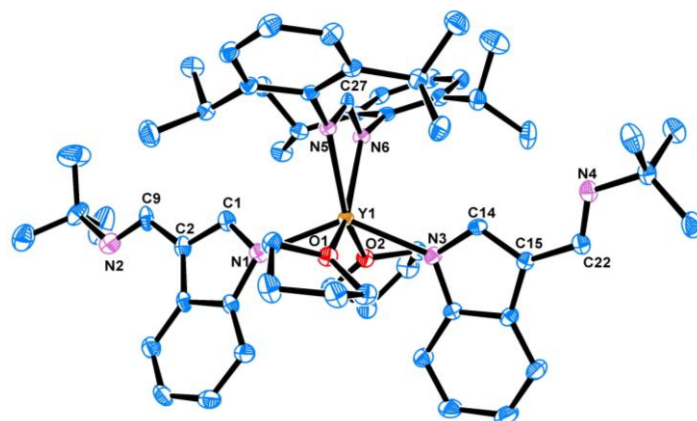


Fig. S6. Structure of complex **6**. Hydrogen atoms and solvated toluene molecule were omitted for clarity. Selected bond distances (Å) and angles (°): Y(1)-N(1) 2.288(2), Y(1)-N(3) 2.2843(19), Y(1)-N(5) 2.3682(18), Y(1)-N(6) 2.3580(18), Y(1)-O(1) 2.3540(16), Y(1)-O(2) 2.3589(16), Y(1)-C(27) 2.774(2), N(3)-Y(1)-N(1) 136.48(7), O(1)-Yb(1)-O(2) 134.04(6), N(6)-Y(1)-N(5) 57.43(6).

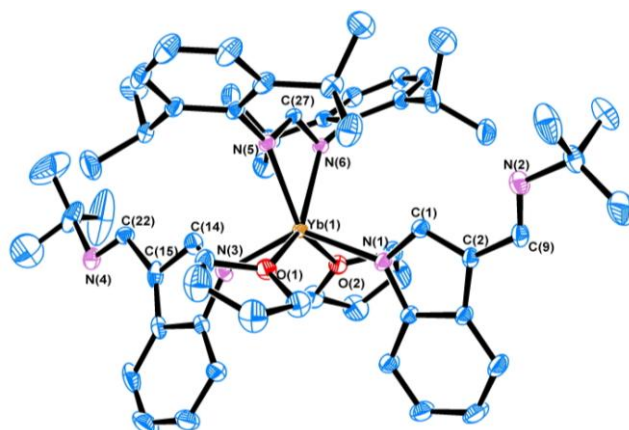


Fig. S7. Structure of complex **7**. Hydrogen atoms were omitted for clarity. Selected bond distances (Å) and angles (°): Yb(1)-N(1) 2.249(3), Yb(1)-N(3) 2.250(3), Yb(1)-N(5) 2.336(2), Yb(1)-N(6) 2.339(2), Yb(1)-O(1) 2.331(2), Yb(1)-O(2) 2.339(2), Yb(1)-C(27) 2.730(3), N(3)-Yb(1)-N(1) 134.38(10), O(1)-Yb(1)-O(2) 135.89(9), N(6)-Yb(1)-N(5) 57.59(8).

Crystal Structure Determinations. A suitable crystal of complexes **1-7** was each mounted in a sealed capillary. Diffraction was performed on a Bruker SMART CCD area detector diffractometer using graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å). An empirical absorption correction was applied using the SADABS program.⁵ All structures were solved by direct methods, completed by subsequent difference Fourier syntheses, and refined anisotropically for all non-hydrogen atoms by full-matrix least-squares calculations on F^2 using the SHELXTL program package.⁶ All hydrogen atoms were refined using a riding model. Crystal data and details of the data collection are given in Table 1.

References

1. (a) S. L. Zhou, S. W. Wang, G. S. Yang, X. Y. Liu, E. H. Sheng, K. H. Zhang, L. Cheng, Z. X.

- Huang, *Polyhedron.*, 2003, **22**, 1019-1024; (b) E. H. Sheng, S. W. Wang, G. S. Yang, S. L. Zhou, K. H. Zhang, L. Cheng, Z. X. Huang, *Organometallics*, 2003, **22**, 684–692.
 2. K. Kloc, E. Kubicz, J. Mlochowski and L. Syper, *Synthesis*, 1987, 1084-1087.
 3. El-Saved M. Afsah, *J. Chem. Soc., Perkin. Trans.*, 1984, 1929-1932.
 4. K. E. Krahulic, G. D. Enright, M. Parvez and R. Roesler, *J. Am. Chem. Soc.*, 2005, **127**, 4142-4143.
 5. G. M. Sheldrick, SADABS: Program for Empirical Absorption Correction of Area Detector Data, University of Göttingen, Germany, 1996.
 6. G. M. Sheldrick, SHELXTL 5.10 for Windows NT: Structure Determination Software Programs, Bruker Analytical X-ray Systems, Inc., Madison, WI, 1997.

Table 1. Crystallographic Data for Complexes 1-7

Compound	1	2	3	4	5	6	7
formula	C ₄₉ H ₇₀ Cl ₂ LiN ₇ OSi ₂ Y ₂	C ₄₉ H ₇₀ Cl ₂ LiN ₇ OSi ₂ Yb ₂	C ₃₈ H ₆₆ Li ₄ N ₆ Si ₄	C ₃₈ H ₇₀ LiN ₆ Si ₄ Y	C ₃₈ H ₇₀ LiN ₆ Si ₄ Yb	C ₅₉ H ₈₁ N ₆ O ₂ Y·C ₇ H ₈	C ₅₉ H ₈₁ N ₆ O ₂ Yb
formula wt	1084.96	1253.22	747.09	819.21	903.34	1087.34	1079.34
cryst syst	Triclinic	Triclinic	Triclinic	Monoclinic	Monoclinic	Triclinic	Triclinic
space group	<i>P</i> -1	<i>P</i> -1	<i>P</i> -1	<i>P</i> 2/ <i>c</i>	<i>P</i> 2/ <i>c</i>	<i>P</i> -1	<i>P</i> -1
<i>a</i> (Å)	15.0187(13)	15.0250(14)	9.1116(8)	12.431(2)	12.454(3)	11.8630(12)	11.9972(8)
<i>b</i> (Å)	16.0047(14)	15.9524(15)	11.2374(10)	15.176(3)	15.076(3)	13.3410(14)	13.3074(9)
<i>c</i> (Å)	16.6926(14)	16.6905(16)	12.0801(11)	15.3466(17)	15.519(3)	20.967(2)	21.3606(15)
α (°)	84.1590(10)	84.3230(10)	97.2570(10)	90	90	106.190(6)	105.7870(10)
β (°)	64.9450(10)	65.1050(10)	100.0590(10)	124.166(9)	124.822(13)	102.549(13)	102.5530(10)
γ (°)	78.9000(10)	78.8840(10)	101.9070(10)	90	90	91.116(7)	92.6190(10)
Volume (Å ³)	3565.9(5)	3560.0(6)	1174.63(18)	2395.5(7)	2392.0(9)	3099.2(5)	3183.2(4)
Z	2	2	1	2	2	2	2
<i>D</i> _{calcd} (g cm ⁻³)	1.010	1.169	1.056	1.136	1.254	1.165	1.126
μ (mm ⁻¹)	1.759	2.750	0.157	1.348	2.085	0.987	1.509
F (000)	1128	1252	404	876	938	1164	1126
θ range [°]	1.30 to 27.54	1.30 to 27.58	1.74 to 25.99	1.98 to 27.52	1.99 to 27.64	1.63 to 27.94	1.63 to 27.79
Limiting indices	-19<= <i>h</i> <=19	-19<= <i>h</i> <=19	-11<= <i>h</i> <=10	-15<= <i>h</i> <=16	-15<= <i>h</i> <=16	-15<= <i>h</i> <=15	-15<= <i>h</i> <=15
	-20<= <i>k</i> <=20	-20<= <i>k</i> <=20	-13<= <i>k</i> <=13	-17<= <i>k</i> <=19	-18<= <i>k</i> <=19	-17<= <i>k</i> <=17	-17<= <i>k</i> <=16
	-21<= <i>l</i> <=21	-21<= <i>l</i> <=18	-14<= <i>l</i> <=14	-19<= <i>l</i> <=19	-20<= <i>l</i> <=20	-27<= <i>l</i> <=27	0<= <i>l</i> <=28
Reflections	31129 / 16071	30653 / 15993	9138 / 4549	20090 / 5483	19741 / 5505	40064 / 14725	14509 / 14509
collected/ unique	[<i>R</i> (int) = 0.0543]	[<i>R</i> (int) = 0.0516]	[<i>R</i> (int) = 0.0211]	[<i>R</i> (int) = 0.1383]	[<i>R</i> (int) = 0.0814]	[<i>R</i> (int) = 0.0506]	[<i>R</i> (int) = 0.0000]
Data/ restraints/ parameters	16071 / 48 / 592	15993 / 24 / 592	4549 / 0 / 244	5483 / 0 / 240	5505 / 0 / 236	14725 / 0 / 676	14509 / 46 / 646
GOF	1.014	1.029	1.048	1.087	1.009	1.161	0.983
Final <i>R</i> indices	<i>R</i> _I = 0.0888	<i>R</i> _I = 0.0655	<i>R</i> _I = 0.0434	<i>R</i> _I = 0.0609	<i>R</i> _I = 0.0684	<i>R</i> _I = 0.0501	<i>R</i> _I = 0.0326
[<i>I</i> > 2σ(<i>I</i>)]	w <i>R</i> ₂ = 0.2847	w <i>R</i> ₂ = 0.2046	w <i>R</i> ₂ = 0.1204	w <i>R</i> ₂ = 0.0833	w <i>R</i> ₂ = 0.1418	w <i>R</i> ₂ = 0.1142	w <i>R</i> ₂ = 0.0855
<i>R</i> indices	<i>R</i> _I = 0.1914	<i>R</i> _I = 0.1450	<i>R</i> _I = 0.0617	<i>R</i> _I = 0.1963	<i>R</i> _I = 0.1321	<i>R</i> _I = 0.0634	<i>R</i> _I = 0.0410
(all data)	w <i>R</i> ₂ = 0.3462	w <i>R</i> ₂ = 0.2519	w <i>R</i> ₂ = 0.1340	w <i>R</i> ₂ = 0.1036	w <i>R</i> ₂ = 0.1726	w <i>R</i> ₂ = 0.1180	w <i>R</i> ₂ = 0.0892
largest diff peak/hole (e Å ⁻³)	1.854 and -0.468	1.773 and -0.998	0.260 and -0.195	0.410 and -0.372	2.063 and -1.036	0.697 and -0.841	0.691 and -0.682