Electronic supplementary materials *Mendeleev Commun.*, 2025, **35**, 31–34

Synthesis and structure of trinuclear yttrium(III) dihydrido cluster stabilized by bulky amidinate ligand

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Experimental section

All operations were carried out under an argon atmosphere using Schlenk techniques or in nitrogen filled glovebox. THF and toluene were purified by distillation from sodium/benzophenone ketyl and degassed thoroughly. Hexane was dried by distillation from sodium/triglyme and benzophenone ketyl prior to use. Lanthanide metal analysis was carried out by complexonometric titration.[S1] NMR spectra were recorded using a Bruker 300 MHz spectrometer. Chemical shifts for ^{1}H, ^{13}C{^{1}H} in the NMR spectra were referenced internally to the residual solvent resonances and are reported relative to TMS. The C, H, N elemental analyses were performed in the microanalytical laboratory of the G. A. Razuvaev Institute of Organometallic Chemistry.

Characterization of (Amd^{tBu})Y(CH$_2$SiMe$_3$)$_2$(THF) (1) by single crystal X-Ray analysis. Complex **1** was synthesized according to previously published procedure.[S2] Single-crystal samples of **1** were obtained by slowly concentrating its solution in a THF/hexane mixture (1:5 v/v) at room temperature.

Synthesis of [(Amd^{tBu})Y(μ-H)$_2$]$_3$(THF)$_2$ (2). To a solution of **1** (0.490 g, 0.65 mmol) in a THF/hexane mixture (1:4, 15 mL) was added PhSiH$_3$ (0.165 g, 1.5 mol; 2.3 equiv.) at room temperature, and the reaction mixture was left at ambient temperature for 12 hours. The solvents were removed in vacuum, the solid residue was dried for 1 hour and then dissolved in a fresh portion of THF/hexane (1:4, 15 mL). Concentrating the resulting solution at room temperature led to the formation of colorless crystals of complex **2**. The mother liquor was decanted, the crystals were washed with hexane and dried in vacuum for 30 minutes. Complex **2** was isolated in 59% yield (0.235 g, 0.13 mmol). $C_{95}H_{151}N_6O_2Y_3\cdot 2(C_6H_{14})$ (1848.32 g/mol). Found

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[S1] S. J. Lyle and M. M. Rahman, *Talanta*, 1963, **10**, 1177; https://doi.org/10.1016/0039-9140(63)80170-8.

[S2] A. V. Karpov, A. S. Shavyrin, A. V. Cherkasov, G. K. Fukin and A. A. Trifonov, *Organometallics*, 2012, **31**, 5349; https://doi.org/10.1021/om300377h.

(%): C, 69.18; H, 9.38; N, 4.80; Y, 14.92. Calculated (%): C, 69.53; H, 9.76; N, 4.55; Y, 14.43. ^{1}H NMR spectrum (300 MHz, toluene-d_8, 293 K) (δ, ppm): 1.08–1.60 (m, 99H, $C\underline{H}_3$ tBu and iPr), 1.69 (br s, 8H, β-$C\underline{H}_2$ THF) , 3.38 (br s, 8H, α-$C\underline{H}_2$ THF), 3.61 (m, 8H, $C\underline{H}$ iPr), 3.72 (m, 4H, $C\underline{H}$ iPr), 5.92 (quadr, $^1J_{YH}$ = 16.5 Hz, 6H, Y−$\underline{H}$) , 6.74–7.08 (m, 18H, $C\underline{H}$ $C_6H_3iPr_2$). $^{13}C\{^1H\}$ NMR spectrum (75 MHz, toluene-d_8, 293 K) (δ, ppm): 21.8 (s, $\underline{C}H_3$ iPr), 22.8 (s, $\underline{C}H_3$ iPr), 22.9 (s, $\underline{C}H_3$ iPr), 23.7 (s, $\underline{C}H_3$ iPr), 24.6 (s, β-$\underline{C}H_2$ THF), 28.4 (s, $\underline{C}H$ iPr), 29.9 (s, $\underline{C}H$ iPr), 30.5 (s, $\underline{C}H_3$ tBu), 31.6 (s, $\underline{C}H_3$ tBu), 44.2 (s, $\underline{C}$ tBu), 44.5 (s, $\underline{C}$ tBu), 67.2 (s, α-$\underline{C}H_2$ THF), 123.0 (s, $\underline{C}H$ $C_6H_3iPr_2$), 123.5 (s, $\underline{C}H$ $C_6H_3iPr_2$), 123.8 (s, $\underline{C}H$ $C_6H_3iPr_2$), 124.0 (s, $\underline{C}H$ $C_6H_3iPr_2$), 142.2 (s, $\underline{C}$ $C_6H_3iPr_2$), 142.5 (s, $\underline{C}$ $C_6H_3iPr_2$), 143.2 (s, $\underline{C}$ $C_6H_3iPr_2$), 144.1 (s, $\underline{C}$ $C_6H_3iPr_2$), 178.9 (s, $N\underline{C}N$).

General procedure for hydrosilylation reactions. To a solution of complex **2** (10 μmol) in C_6D_6 (0.6 mL) were added $PhSiH_3$ (0.5 mmol) and alkene (0.5 mmol). The reaction course was controlled by ^{1}H NMR spectroscopy. Conversion and selectivity were determined by integrating the remaining substrates and the newly formed hydrosilylation products in the ^{1}H, $^{13}C\{^1H\}$ and $^{29}Si\{^1H\}$ NMR spectra according to previously known data.

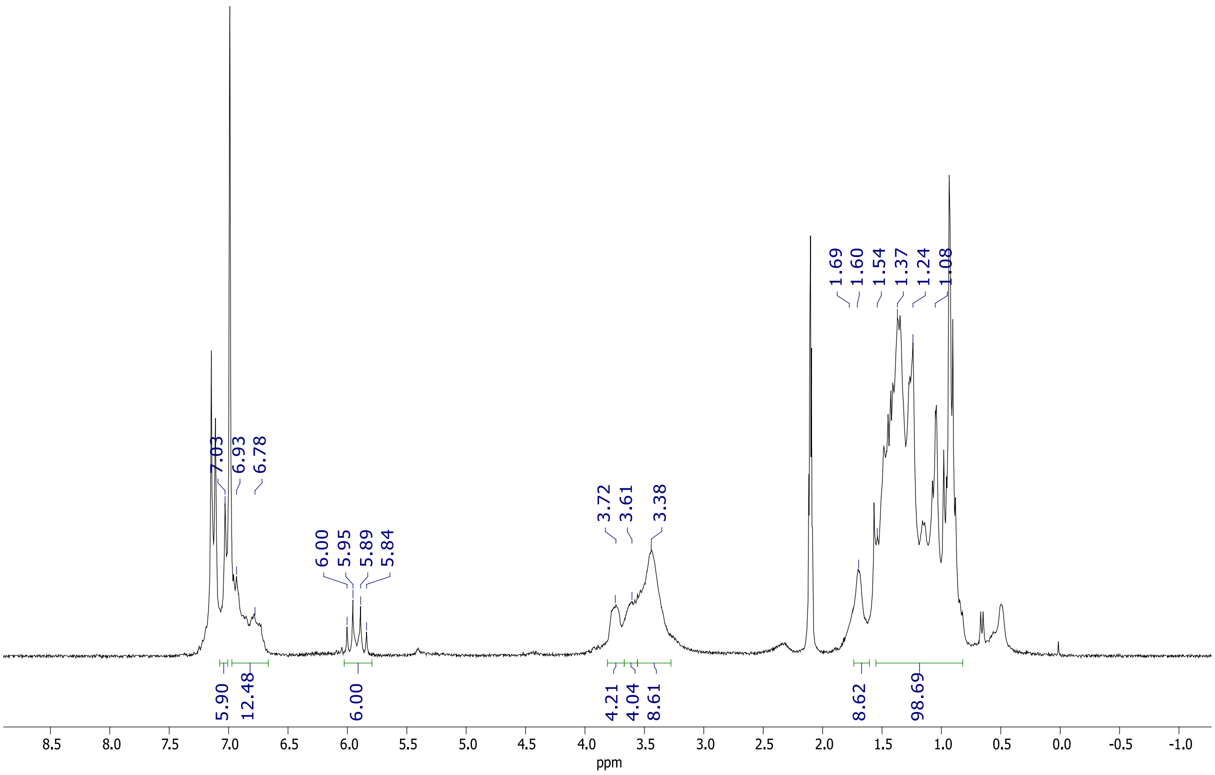

Figure S1. ^{1}H NMR spectrum of $[(Amd^{tBu})Y(\mu\text{-}H)_2]_3(THF)_2$ (**2**) (300 MHz, toluene-d_8, 293 K)

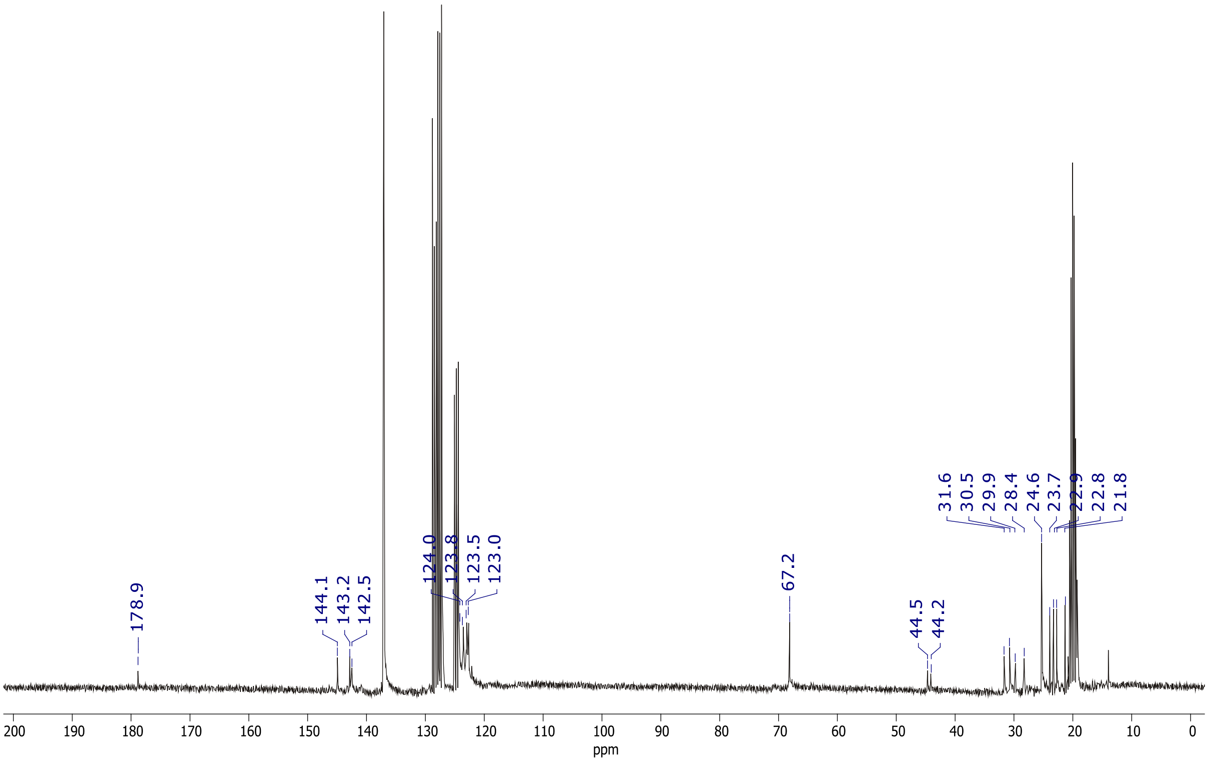

Figure S2. $^{13}C\{^{1}H\}$ NMR spectrum of $[(Amd^{tBu})Y(\mu\text{-}H)_2]_3(THF)_2$ (**2**) (75 MHz, toluene-d_8, 293 K).