The Synthesis and Structure of Bis- and Tris(amino)-Functionalised Cyclopentadienylyttrium Derivatives

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The reaction of YCl_3 with 2 or 3 equiv. of $NaC_5H_4CH([CH_2]_2)_2NMe$ in toluene affords $[\{Y[\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe]_2(\mu\text{-}Cl)\}_2]$ (**1**) and $[Y\{\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe\}_3]$ (**2**), respectively, in high yield; the three amino-functionalised cyclopentadienyl units of **2** are fluxional at room temperature, giving us a rare example of stabilisation of an yttrium centre without the use of extraneous ligands.

Introduction

Amino-functionalised cyclopentadienyl derivatives have been used to great effect in complexes of the d-block elements.[1,2] As part of our ongoing studies investigating the early transition metal systems for metal−dihydrogen bonding we decided, given previous successes, to concentrate on the (*N*-methylpiperid-4-yl)cyclopentadienyl ligand.[3−6] In this paper we present a number of new yttrium chloride complexes, one of which shows particularly interesting fluxional properties and provides a rare example of stabilisation of a cyclopentadienyl centre without the use of extraneous ligands.

Results and Discussion

As part of our ongoing work concerning the formation of new bis(cyclopentadienyl)yttrium hydride derivatives we report the synthesis and structure of $[\{Y[\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe]_2(\mu\text{-}Cl)\}_2]$ (**1**) and $[Y\{\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe\}_3]$ (**2**); the latter derivative is an intriguing fluxional tris(cyclopentadienyl) compound and provides the first example of a homoleptic base-stabilised tris(cyclopentadienyl)yttrium complex. The reaction of YCl_3 with 2 equiv. of $NaC_5H_4CH([CH_2]_2)_2NMe$ in THF at 45 °C afforded **1** in high yield (71%) (Scheme 1) which was characterised by ^1H, $^{13}C\{^1H\}$, and ^1H-^{13}C correlation NMR spectroscopy. The reaction of YCl_3 with 3 equiv. of $NaC_5H_4CH([CH_2]_2)_2NMe$ in THF at 50 °C similarly afforded **2** in high yield (85%) which was also formed when the reaction was carried out in toluene.

Crystallisation of **1** from a concentrated toluene solution at −30 °C afforded colourless prisms. The molecular structure, determined from a single-crystal X-ray diffraction study, revealed **1** to be dimeric with an inversion centre at the intersection of the Y(1)−Y(1)′ and Cl(1)−Cl(1)′ vectors and a *metallocene dichloride type* ligand geometry about the Y atoms (Figure 1) while the *N*-methylpiperid-4-yl moieties, in chair conformation, point to opposite sides of the *metallocene* geometry to minimise steric crowding. A similar bonding arrangement has been previously reported for $[\{Y(\eta\text{-}C_5H_4SiMe_3)_2(\mu\text{-}Cl)\}_2]$,[7,8] $[\{Y(\eta\text{-}C_5H_4[C_6H_9\text{-}2\text{-}i\text{Pr-}5\text{-}Me])_2(\mu\text{-}Cl)\}_2]$,[9] $[\{Y(\eta\text{-}C_5H_5)_2(\mu\text{-}Cl)\}_2]$,[10] and $[\{Y(\eta\text{-}C_5H_4PPh_2)_2(\mu\text{-}Cl)\}_2]$.[11] The Y(1)−Cl(1) and Y(1)−Cl(1)′ bond lengths of 2.6850(7) and 2.6984(7) Å are similar to other reported chloro-bridged dimeric bis(cyclopentadienyl)yttrium derivatives. The average cyclopentadienyl ring C−C bond lengths and angles are unexceptional [1.412(4) Å, 108.0(3)°].

Despite the relatively high coordination number, monomeric tris(cyclopentadienyl)Ln complexes are coordinatively unsaturated. In the absence of solvents, these molecules form oligomeric structures in the solid state.[12,13] With the addition of donor ligands or coordinating solvents molecules of the type $LnCp_3L$ can be formed, e.g., $[Y(\eta\text{-}C_5H_5)_3(THF)]$[14] and $[Y(\eta\text{-}C_5H_5)_2(\eta\text{-}C_5H_4CH_2CH_2OMe)]$.[15] Crystallisation of the extremely air- and mositure-sensitive compound **2** from a concentrated toluene solution at −30 °C afforded pale yellow prisms. The molecular structure was determined from a single-crystal X-ray diffraction study (Figure 2). The ligand geometry about the Y atom is slightly distorted from trigonal-planar, with

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$$\underset{\textbf{1}}{[\{Y(\eta\text{-}C_5H_4CH[CH_2]_4NMe)_2(\mu\text{-}Cl)\}_2]} \xleftarrow{(i)} YCl_3 \xrightarrow{(ii)} \underset{\textbf{2}}{[Y\{\eta\text{-}C_5H_4CH(CH_2)_4NMe\}_3]}$$

Scheme 1. Reagents and conditions: (*i*) 2 $NaC_5H_4CH([CH_2]_2)_2NMe$, THF, 45 °C, 12 h; (*ii*) 3 $NaC_5H_4CH([CH_2]_2)_2NMe$, THF or toluene, 50 °C, 12 h

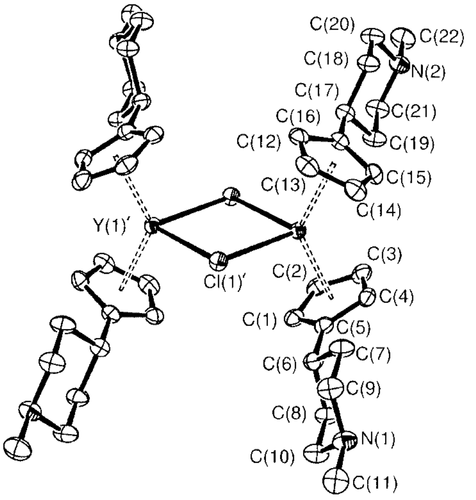

Figure 1. The molecular structure of **1** and atom numbering scheme with selected bond lengths [Å] and angles [°]; estimated standard deviations in parentheses: Y(1)−C(1) 2.652(3), Y(1)−C(12) 2.653(3), Y(1)−C(2) 2.612(3), Y(1)−C(13) 2.619(3), Y(1)−C(3) 2.589(3), Y(1)−C(14) 2.591(3), Y(1)−C(4) 2.618(3), Y(1)−C(15) 2.617(3), Y(1)−C(5) 2.667(3), Y(1)−C(16) 2.673(3), Y(1)−Cl(1) 2.6984(7), Y(1)−Cl(1)′ 2.6850(7); C(2)−Y(1)−C(1) 30.99(10), Y(1)−Cl(1)−Y(1)′ 96.30(2), C(4)−Y(1)−C(12) 132.04(10), C(10)−N(1)−C(11) 111.0(3), C(21)−N(2)−C(22) 110.0(3), C(9)−N(1)−C(11) 109.8(3)

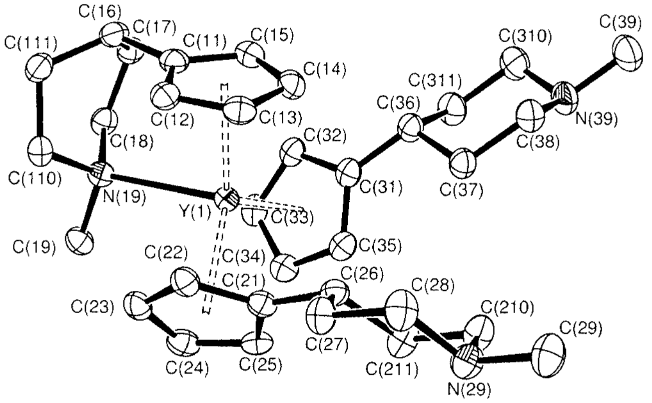

Figure 2. The molecular structure of **2** and atom numbering scheme with selected bond lengths [Å] and angles [°]; estimated standard deviations in parentheses: Y(1)−C(11) 2.723(2), Y(1)−C(14) 2.693(2), Y(1)−C(15) 2.680(2), Y(1)−C(34) 2.690(2), Y(1)−C(33) 2.712(2), Y(1)−C(35) 2.724(2), Y(1)−C(21) 2.732(2), Y(1)−C(22) 2.750(2), Y(1)−C(23) 2.738(2), Y(1)−C(24) 2.734(2), Y(1)−C(25) 2.743(2), Y(1)−N(19) 2.6954(19); C(11)−Y(1)−C(21) 122.32(7), C(26)−C(21)−Y(1) 119.12(15), C(19)−N(19)−Y(1) 107.14(14), C(19)−N(19)−C(18) 107.35(18), C(19)−N(19)−C(110) 107.27(19), C(18)−N(19)−C(110) 106.44(18), C(19)−N(19)−Y(1) 107.14(14), C(18)−N(19)−Y(1) 116.34(14), C(110)−N(19)−Y(1) 111.88(14)

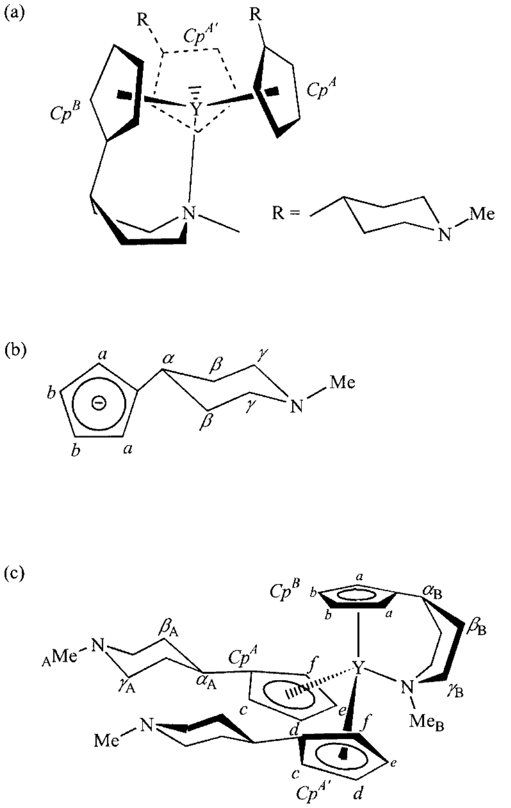

Figure 3. (a) Ligand geometry about the yttrium centre of **2**; (b) $C_5H_4CH([CH_2]_2)_2NMe$ ligand; (c) molecular structure of **2** at −60 °C

the angles between the centroids of the cyclopentadienyl substituents, M−Y(1)−M, of ca. 116−118°.

Two of the *N*-methylpiperid-4-yl rings adopt a chair conformation and point away from the metal centre [Cp^A and $Cp^{A'}$, see (a) in Figure 3], whereas the third *N*-methylpiperid-4-yl ring (Cp^B) is bound to the metal centre through the d_{z^2} metal orbital.[16] A similar bonding arrangement has been previously reported for [2-(dimethylamino)ethyl]cyclopentadienyl derivatives [Ln(η-$C_5H_4CH_2CH_2NMe_2)_3$] (Ln = La, Nd).[17] The boat confomation of the Cp^B ring confirms the bound nature of the N moiety. The Y(1)−N(19) bond length of 2.6954(19) Å is somewhat longer than the distance of 2.501(4) Å reported for [Y(η-C_5Me_5)(η-$C_5H_4CHC_6H_4CH_2NMe_2$)Cl][18] and **2** could be viewed as a strained 10-coordinate compound. The Y−C(Cp) bond lengths are similar to previously reported tris(cyclopentadienyl)Y derivatives.[12,14,15] The average cyclopentadienyl ring C−C bond lengths and angles are unexceptional [1.409(3) Å, 108.0(2)°].

Compound **2** was initially characterised by ^1H NMR spectroscopy; the room-temperature ^1H NMR spectrum (300.13 MHz, C_7D_8) displayed broad resonance signals, which sharpened upon warming to 80 °C to give the time-averaged ^1H NMR spectrum typical for the $C_5H_4CH([CH_2]_2)_2NMe$ ligand [see (b) in Figure 3]. Compound **2** was characterised at this elevated temperature by

^{1}H, ^{13}C{^{1}H}, and ^{1}H-^{13}C correlation NMR spectroscopy. Cooling of **2** from room temperature to −60 °C afforded multiple resonance signals in the ^{1}H NMR spectrum, indicating the formation of a static structure with one of the pendant arms bound to the yttrium centre [see (c) in Figure 3]. This structural arrangement was also investigated by low-temperature ^{13}C{^{1}H}, DEPT-135, INEPT (Figure 4), and ^{1}H-^{13}C correlation NMR spectroscopy. The ^{13}C{^{1}H} NMR resonance assignments for the *N*-methylpiperid-4-yl function at both 80 and −60 °C are summarised in Table 1.

The ^{13}C{^{1}H} NMR spectrum of **2** at −60 °C in the *N*-methylpiperid-4-yl region ($\delta \approx 20-60$ ppm) displays 9 distinct resonances due to slow structural isomer interconversion on the NMR time-scale. Two resonance signals are observed for the γ-CH$_2$ environment at δ = 56.5 and 53.1 ppm in the approximate ratio 2:1. Warming sees a gradual broadening and coalescence at ca. 20 °C eventually giving rise to one ^{13}C resonance at δ = 55.6 ppm. Two ^{13}C resonances are also observed for the Me environments (δ = 46.9 and 50.8 ppm coalescing at ca. 20 °C and giving one resonance at 50 °C at δ = 47.2 ppm) and the α-CH environment (δ = 25.1 and 36.7 ppm coalescing at ca. 30 °C giving one resonance at δ = 34.0 ppm). The β-CH$_2$ environments give rise at −60 °C, however, to three distinct resonances (δ = 25.0, 34.2, and 34.9 ppm), indicating a further decrease in the symmetry of the molecular fragment, due to close approach of the *N*-methylpiperid-4-yl arms at the β-position. A similar phenomenon has been previously observed in the

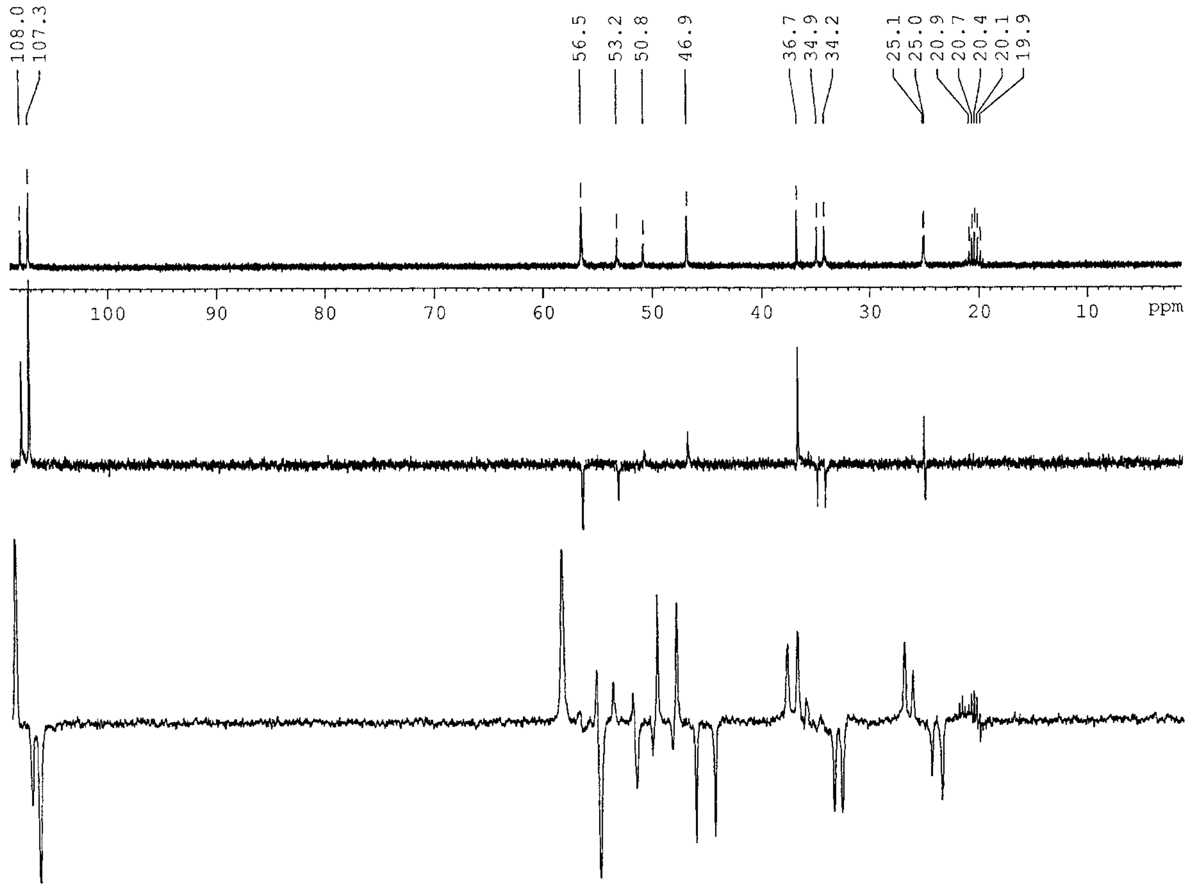

Figure 4. (a) ^{13}C{^{1}H}, (b) DEPT 135, and (c) INEPT NMR spectra of **2** at −60 °C

Table 1. ^{13}C{^{1}H} NMR resonance assignments for the *N*-methylpiperid-4-yl function

Compound	Assignment			
	β-CH$_2$	α-CH	Me	γ-CH$_2$
NaC$_5$H$_4$CH([CH$_2$]$_2$)$_2$NMe (C$_4$D$_8$O, room temp.)	36.7	38.4	47.4	58.3
1 (C$_4$D$_8$O, room temp.)	34.5	36.8	47.0	57.1
2 (C$_7$D$_8$, 80 °C)	32.0	34.0	47.2	55.6
2 (C$_7$D$_8$, −60 °C)	25.0 (β$_B$), 34.2 (β$_A$), 34.9 (β$_A$)	25.1 (α$_B$), 36.7 (α$_A$)	46.9 (Me$_A$), 50.8 (Me$_B$)	53.2 (γ$_B$), 56.5 (γ$_A$)

cyclohexyl derivative $[\mathrm{Fe}(\eta\text{-}\mathrm{C_5H_2}\text{-}1,2,4\text{-}\{\mathrm{CH}([\mathrm{CH_2}]_2)_2\mathrm{CH_2}\}_3)_2]$.[19] The ^{1}H NMR spectrum of **1** at room temperature exhibits resonances typical for the $\mathrm{C_5H_4CH([CH_2]_2)_2NMe}$ ligand. Comparison of the ^{13}C{^{1}H} resonance values of **2** with those of **1** at both −60 and 80 °C (Table 1) provides evidence for non-coordination of the *N*-methylpiperid-4-yl side arm to the $\mathrm{Y^{3+}}$ centre. Compound **1** was found to be only sparingly soluble in toluene, petroleum ether, and dichloromethane solvents. The ^{1}H and ^{13}C{^{1}H} NMR spectral assignments, obtained in $\mathrm{C_4D_8O}$ with resulting cleavage of the bridging Y−Cl bonds,[20] are thus of the THF adduct $\mathrm{Y\{\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe\}_2Cl(THF)}$.

In conclusion the following features of **1** and **2** are noteworthy. (*i*) The presence of the pendant arm functionality on **1** provides a Lewis base for protonation in close proximity to the metal centre. (*ii*) Compound **2** also reveals the potential advantage of the strained *N*-methylpiperid-4-yl-functionalised cyclopentadienyl ligand in the stabilisation and reaction of related yttrium hydride derivatives as it may point towards, yet only weakly bind to, the $\mathrm{Y^{3+}}$ metal centre. (*iii*) Compound **2** is the first example of a homoleptic base-stabilised tris(cyclopentadienyl)yttrium derivative.

Experimental Section

$[\{\mathrm{Y[\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe]_2(\mu\text{-}Cl)}\}_2]$ (1): To a Schlenk tube charged with a slurry of $\mathrm{YCl_3}$ (0.61 g, 3.1 mmol) in THF (80 mL) was added a THF slurry (60 mL) of $\mathrm{NaC_5H_4CH([CH_2]_2)_2NMe}$ (1.15 g, 6.2 mmol) at room temperature. The reaction mixture was warmed to 45 °C and stirred for 12 h to afford an off-white slurry. The THF was removed in vacuo and the resulting beige solid extracted with toluene (2 × 50 mL). The extracts were combined and the solvents were removed in vacuo to afford **1** as a white solid (0.99 g, 1.10 mmol, 71%). $\mathrm{C_{44}H_{64}Cl_2N_4Y_2}$ (897.7): calcd. C 58.87, H 7.19, N 6.24; found C 55.0, H 7.2, N 3.7 (after many attempts, this was the best microanalysis found). ^{1}H NMR (300.13 MHz, $\mathrm{C_4D_8O}$, room temp.): δ = 1.48 (m, 2 H, γ), 1.69 (br. s, THF), 1.87 (m, 4 H, β), 2.16 (s, 3 H, Me), 2.49 (tt, $J_{H,H}$ = 3.68, 11.76 Hz, 1 H, α), 2.76 (m, 2 H, γ), 3.53 (br. s, THF), 5.91 (br. s, 4 H, Cp) ppm. ^{13}C{^{1}H} NMR (75.47 MHz, $\mathrm{C_4D_8O}$, room temp.): δ = 34.5 (β, $^1J_{CH}$ = 129 Hz), 36.8 (α, $^1J_{CH}$ = 131 Hz), 47.0 (Me, $^1J_{CH}$ = 132 Hz), 57.1 (γ, $^1J_{CH}$ = 132 Hz), 110.1 (Cp, $^1J_{CH}$ = 167 Hz), 110.2 (Cp, $^1J_{CH}$ = 167 Hz) ppm.

$[\mathrm{Y\{\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe\}_3}]$ (2): To a Schlenk tube charged with a slurry of $\mathrm{YCl_3}$ (0.99 g, 5.1 mmol) in THF (80 mL) was added a THF slurry (50 mL) of $\mathrm{NaC_5H_4CH([CH_2]_2)_2NMe}$ (2.80 g, 15.2 mmol) dropwise at room temperature. An immediate colour change of white to pale yellow was observed. The reaction mixture was warmed to 50 °C and stirred for 12 h to afford a brown slurry. The THF was removed in vacuo and the resulting sticky brown solid extracted with toluene (3 × 30 mL). The toluene was removed in vacuo to afford **2** as a white solid (2.48 g, 4.3 mmol, 85%). $\mathrm{C_{33}H_{48}N_3Y}$ (575.7): calcd. C 68.9, H 8.3, N 7.3; found C 68.45, H 8.2, N 6.9. ^{1}H NMR (300.13 MHz, $\mathrm{C_7D_8}$, room temp.): δ = 1.98 (br. s, 6 H, β, γ), 2.21 (br. s, 3 H, Me), 2.82 (br. s, 1 H, α), 2.90 (br. s, 2 H, γ), 6.01 (br. s, 4 H, Cp) ppm; (300.13 MHz, $\mathrm{C_7D_8}$, 80 °C): δ = 1.77 (m, 4 H, β), 1.90 (m, 2 H, γ), 2.15 (s, 3 H, Me), 2.56 (m, 1 H, α), 2.80 (m, 2 H, γ), 5.79 (m, 2 H, Cp), 5.82 (m, 2 H, Cp) ppm; (300.13 MHz, $\mathrm{C_7D_8}$, −60 °C): δ = 1.27 (br. s, γ_B), 1.29 (br. s, β_B), 1.79 (s, $\mathrm{Me_B}$, β_B), 1.84 (m, β_A), 1.95 (m, γ_A), 2.04 (m, β_A), 2.09 (m, β_A), 2.28 (s, $\mathrm{Me_A}$), 2.49 (br. s, γ_B), 2.73 (br. s, α_A, α_B), 2.95 (bt, γ_A), 5.47 (s, Cp), 5.50 (s, Cp), 5.87 (s, Cp), 5.95 (s, Cp), 6.03 (s, Cp), 6.18 (s, Cp) ppm. ^{13}C{^{1}H} NMR (75.47 MHz, $\mathrm{C_7D_8}$, −60 °C): δ = 25.0 (β_B, $^1J_{CH}$ = 130 Hz), 25.1 (α_B, $^1J_{CH}$ = 132 Hz), 34.2 (β_A, $^1J_{CH}$ = 127 Hz), 34.9 (β_A, $^1J_{CH}$ = 128 Hz), 36.7 (α_A, $^1J_{CH}$ = 135 Hz), 46.9 ($\mathrm{Me_A}$, $^1J_{CH}$ = 132 Hz), 50.8 ($\mathrm{Me_B}$, $^1J_{CH}$ = 137 Hz), 53.2 (γ_B, $^1J_{CH}$ = 139 Hz), 56.5 (γ_A, $^1J_{CH}$ = 132 Hz), 107.3 (Cp, $^1J_{CH}$ = 167 Hz), 108.0 (Cp, $^1J_{CH}$ = 167 Hz), 110.8 (Cp, $^1J_{CH}$ = 166 Hz), 111.6 (Cp, $^1J_{CH}$ = 164 Hz), 112.4 (Cp, $^1J_{CH}$ = 166 Hz) ppm; (75.47 MHz, $\mathrm{C_7D_8}$, 80 °C): δ = 32.0 (β), 34.0 (α), 47.2 (Me), 55.6 (γ), 109.2 ($\mathrm{Cp_{a,b}}$), 111.4 ($\mathrm{Cp_{a,b}}$) 129.6 (Cp-C) ppm.

X-ray Structure Determination for 1 and 2

Crystal Data for $[\{\mathrm{Y[\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe]_2(\mu\text{-}Cl)}\}_2]\cdot\mathrm{C_7H_8}$ ($\mathbf{1\cdot C_7H_8}$): Crystallographic measurements were made with a Nonius Kappa CCD area diffractometer using (Mo-K_α) radiation, λ = 0.71073 Å. The structure was solved by direct methods and refined on F^2 by the full-matrix least-squares procedure. T = 150(2) K. Specimen 0.53 × 0.46 × 0.22 mm, $\mathrm{C_{51}H_{72}Cl_2N_4Y_2}$, M = 989.85, monoclinic, spacegroup $P2_1/c$, a = 16.1070(3), b = 21.8000(3), c = 7.02100(10) Å, β = 97.6950(10)°, V = 2443.10(7) Å^3, $D_{\text{calcd.}}$ = 1.346 Mg/m^3, Z = 2. For reflections with 0.998° ≤ θ ≤ 27.48°, $R(F)$ = 0.0442 for 4810 observed reflections [$I > 2\sigma(I)$] and $wR(F^2)$ = 0.1389 for 5576 independent reflections [R(int) = 0.0571]. A molecule of toluene was found which showed a symmetry-related disorder over 2 sites, which was modelled using a rigid body refinement.

Crystal Data for $[\mathrm{Y\{\eta\text{-}C_5H_4CH([CH_2]_2)_2NMe\}_3}]$ (2): Crystallographic measurements were made with a Nonius Kappa CCD area diffractometer using (Mo-K_α) radiation, λ = 0.71073 Å. The structure was solved by direct methods and refined on F^2 by the full-matrix least-squares procedure. T = 150(2) K. Specimen 0.36 × 0.28 × 0.22 mm, $\mathrm{C_{33}H_{48}N_3Y}$, M = 575.65, monoclinic, spacegroup $C2/c$, a = 36.9061(6), b = 12.5796(2), c = 13.2611(2) Å, β = 108.9720(10)°, V = 5822.21(16) Å^3, $D_{\text{calcd.}}$ = 1.313 Mg/m^3, Z = 8. For reflections with 1.72° ≤ θ ≤ 26°, $R(F)$ = 0.0332 for 4803 observed reflections [$I > 2\sigma(I)$] and $wR(F^2)$ = 0.085 for 5701 independent reflections [R(int) = 0.0445].

Acknowledgments

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