Electronic Supplementary Material (ESI) for Dalton Transactions.

Atom-economic access to cationic magnesium complexes

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SUPPORTING INFORMATION

Experimental

General experimental

THF, Et_2O and hexane were distilled over sodium benzophenone ketyl immediately prior to use. Pyrrole was distilled over CaH_2 and stored over 4 Å molecular sieves prior to use. ^{Dipp}NacNac(H) was synthesised according to a literature method.[1] Diarylmagnesium complexes were prepared by oxidative addition of Mg to the parent arylhalide and then shifting of the Schlenk equilibrium via 1,4-dioxane precipitation of magnesium dihalide. *n*Bu_2Mg and *n*BuLi solutions, and $GaCl_3$ were purchased directly from Sigma-Aldrich and used as received. $AlCl_3$ was purchased from Sigma-Aldrich and sublimed prior to use. NMR spectra were recorded on a Bruker AVANCE 400 NMR spectrometer, operating at 400.13 MHz for ^{1}H, 100.62 MHz for ^{13}C, and 104.2 MHz for ^{27}Al. All ^{13}C spectra were proton decoupled. ^{1}H and ^{13}C spectra were referenced to the appropriate solvent resonances. Crystallographic data were collected on Oxford Diffraction/Rigaku instruments with Mo or Cu Kα radiation. Structures were solved using direct methods, and refined on F^2 against all independent reflections by the full-matrix least-squares method using *SHELXL* [2] or *OLEX-2* programs.[3]

Synthesis of (^{Dipp}NacNac)MgPh·THF

$MgPh_2$·4THF (1.3 g, 2.9 mmol) was added to a solution of ^{Dipp}NacNac(H) (1.31 g, 3.0 mmol) in 15 mL of THF. The resulting solution was refluxed for two hours, after cooling down the reaction mixture the volume of THF was reduced until a precipitate appeared. Redissolving the precipitate and slow cooling afforded a crop of crystals. Yield: 1.3 g (76 %).

^{1}H NMR (400 MHz, 298K, C_6D_6): δ = 7.21 (m, 8H, aromatics), 7.12 (m, 3H, aromatics), 4.86 (s, 1H, C*H*, NacNac), 3.54 (m, 4H, THF), 3.30 (broad s, 4H, C*H*, *i*Pr), 1.72 (s, 6H, CH_3, NacNac), 1.23 (d, 12H, CH_3, *i*Pr), 1.19 (m, 4H, THF), 1.13 (d, 12H, CH_3, *i*Pr); **^{13}C NMR (100.6 MHz, 298K, C_6D_6):** δ = 168.4 (*C*-CH_3, NacNac), 165.2 ($C_{quaternary}$, Ph), 145.8 ($C_{quaternary}$, Ar*), 142.8 (*C*H, Ar*), 140.8 (*C*H, Ph), 126.3 (*C*H, Ph), 125.4 (*C*H, Ar*), 125.0 (*C*H, Ph), 124.0 (*C*H, Ar*), 94.5 (*C*H, NacNac), 70.1 (THF), 28.3 (*C*H, *i*Pr), 25.2 (THF), 25.0 (CH_3, *i*Pr), 24.4 (CH_3, *i*Pr), 24.0 (C−CH_3, NacNac).

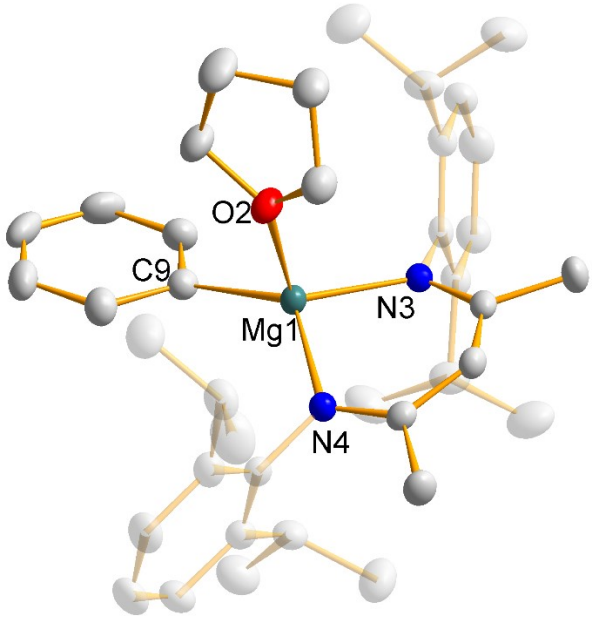

Figure S1 Molecular structure of (^{Dipp}NacNac)MgPh·THF with hydrogen atoms omitted for clarity and with thermal ellipsoids drawn at 50% probability.

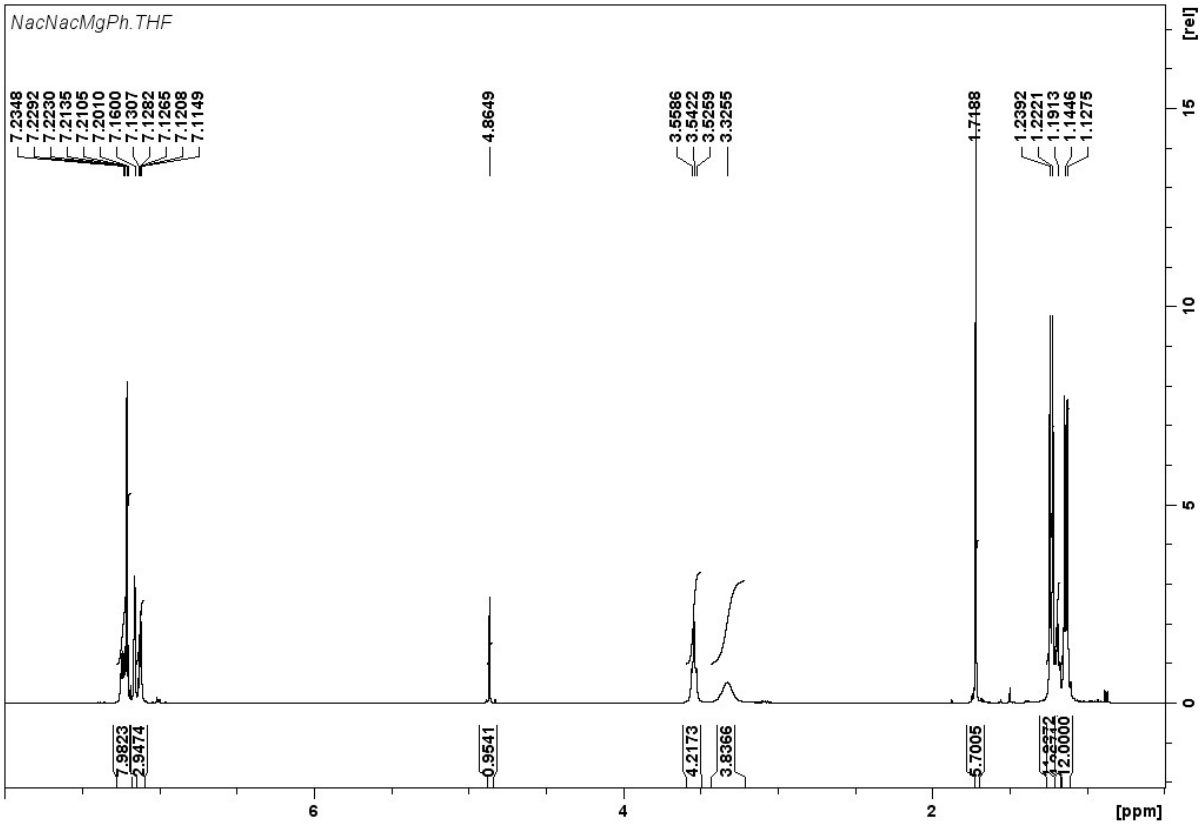

Figure S2 ^{1}H NMR spectrum of (^{Dipp}NacNac)MgPh·THF in C_6D_6

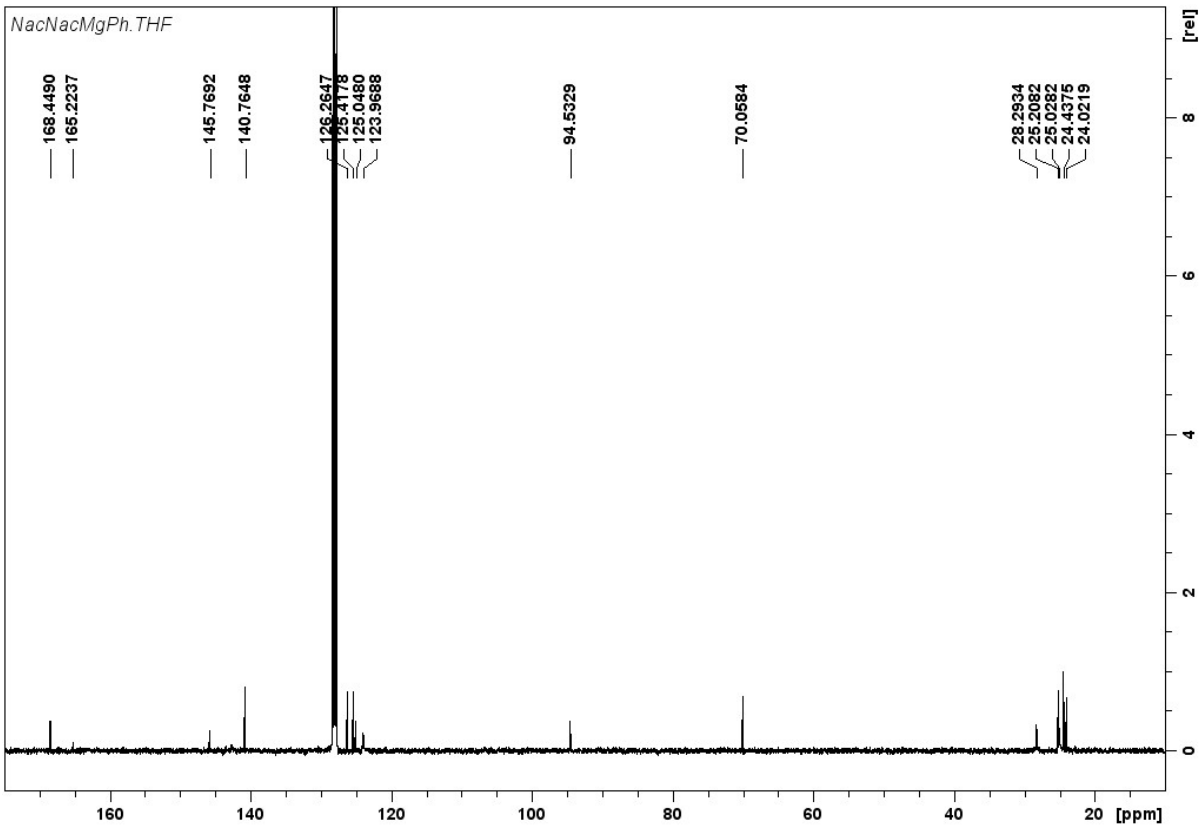

Figure S3 ^{13}C NMR spectrum of (^{Dipp}NacNac)MgPh·THF in C_6D_6

Rational synthesis of $[Mg{\cdot}6THF]^{2+}$ $2[AlPh_4]^-$ (**1a**)

A solution of $AlPh_3{\cdot}OEt_2$ (0.664 g, 2.0 mmol) in THF was added to a solution of $MgPh_2{\cdot}4THF$ (0.467 g, 1.0 mmol) in THF. The addition had to be made slowly such that the Al solution lies on top of the Mg solution, slowly diffusing overnight and forming large colourless crystals. Yield: 1.082 g (96%). Integration of ^1H NMR spectrum suggests one equivalent of THF was removed under vacuum drying of crystals.

^1H NMR (400 MHz, 298K, d_6-DMSO): δ = 7.57 (m, 16H, *ortho*-CH), 7.03 (m, 24H, *meta*- and *para*-CH), 3.60 (m, 20H, THF), 1.76 (m, 20H, THF); **^{13}C NMR (100.6 MHz, 298K, d_6-DMSO):** δ = 138.4 ($C_{quaternary}$, Ph), 125.8 (2 x *C*H, Ph), 124.5 (*C*H, Ph), 67.0 (THF), 25.1 (THF); **^{27}Al NMR 104.2 MHz, 298K, d_6-DMSO):** δ = 132.6 (s).

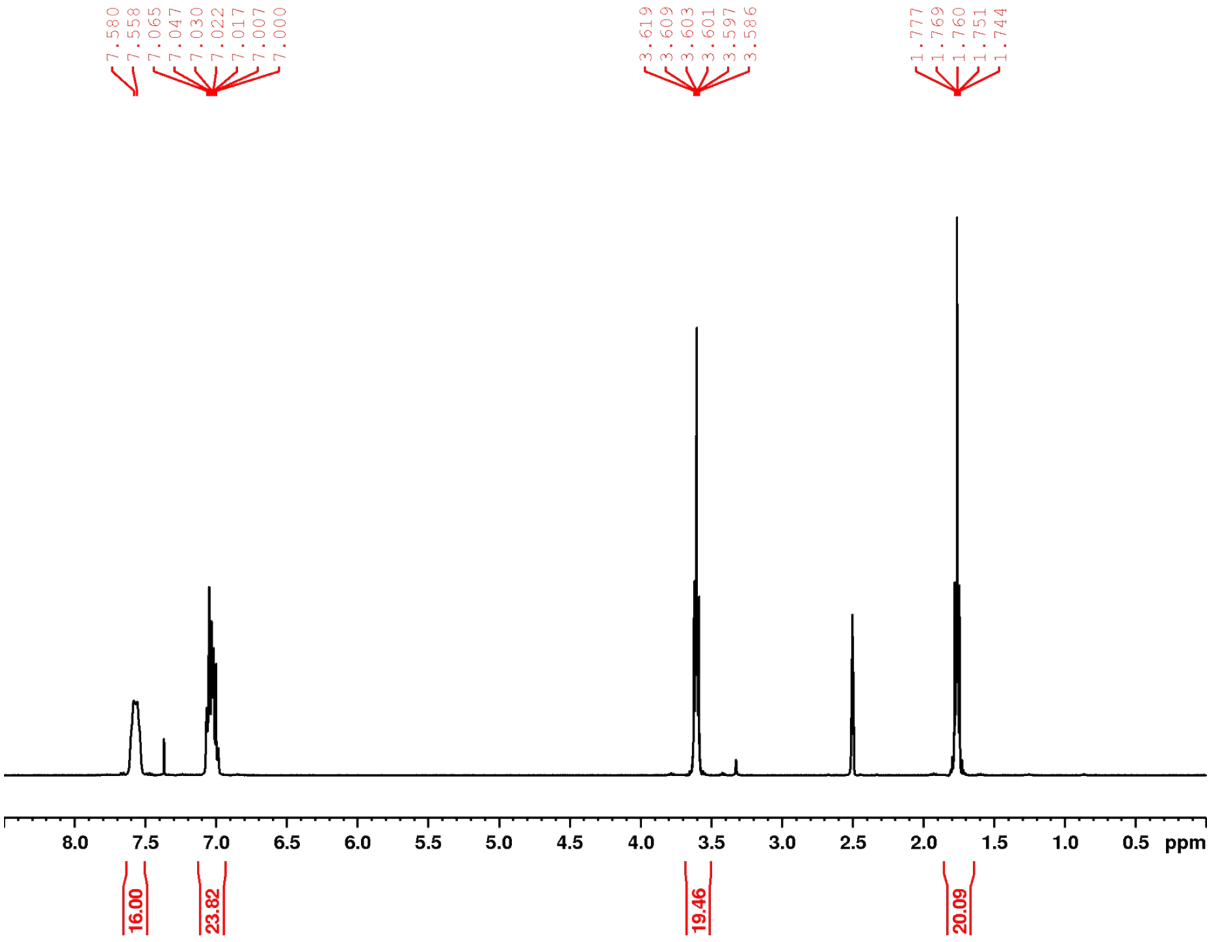

Figure S4 ^1H NMR spectrum of $[Mg{\cdot}6THF]^{2+}$ $2[AlPh_4]^-$ (**1a**) in d_6-DMSO

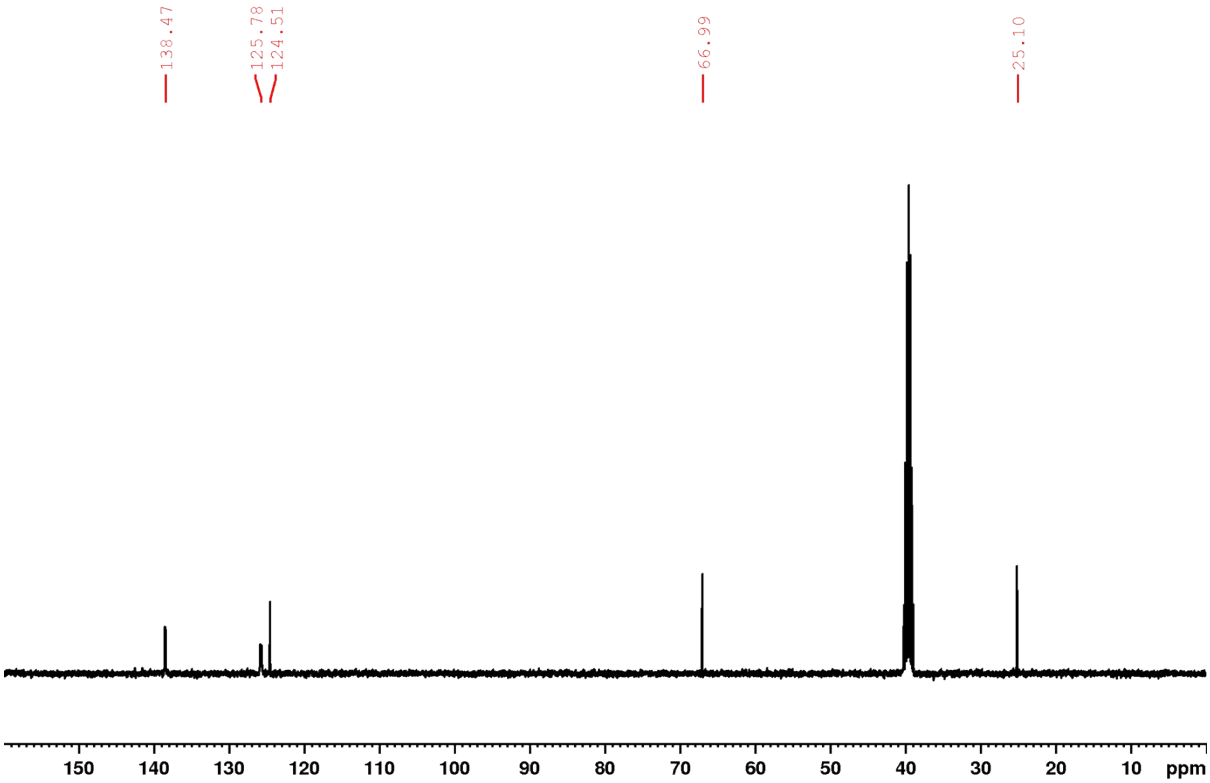

Figure S5 ^{13}C NMR spectrum of $[Mg{\cdot}6THF]^{2+}$ $2[AlPh_4]^-$ (**1a**) in d_6-DMSO

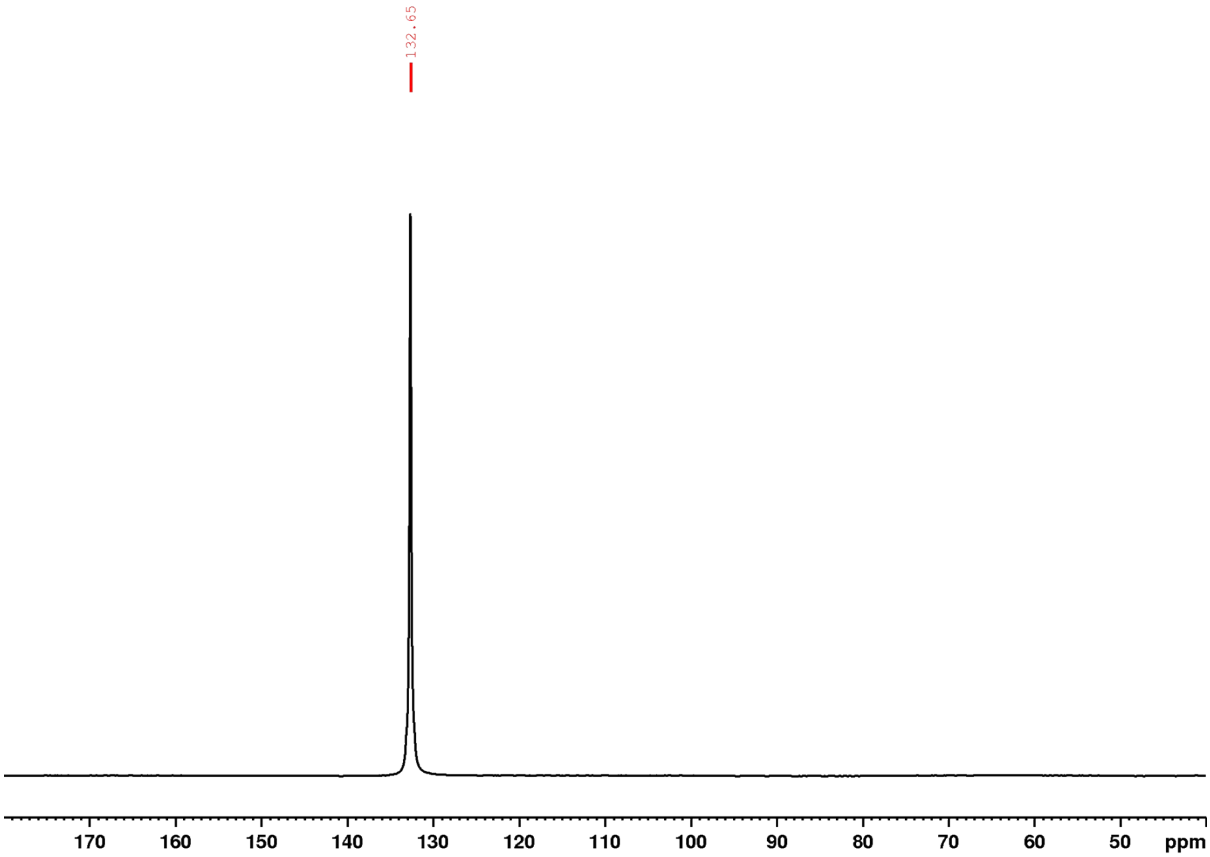

Figure S6 ^{27}Al NMR spectrum of $[Mg{\cdot}6THF]^{2+}$ $2[AlPh_4]^-$ (**1a**) in d_6-DMSO

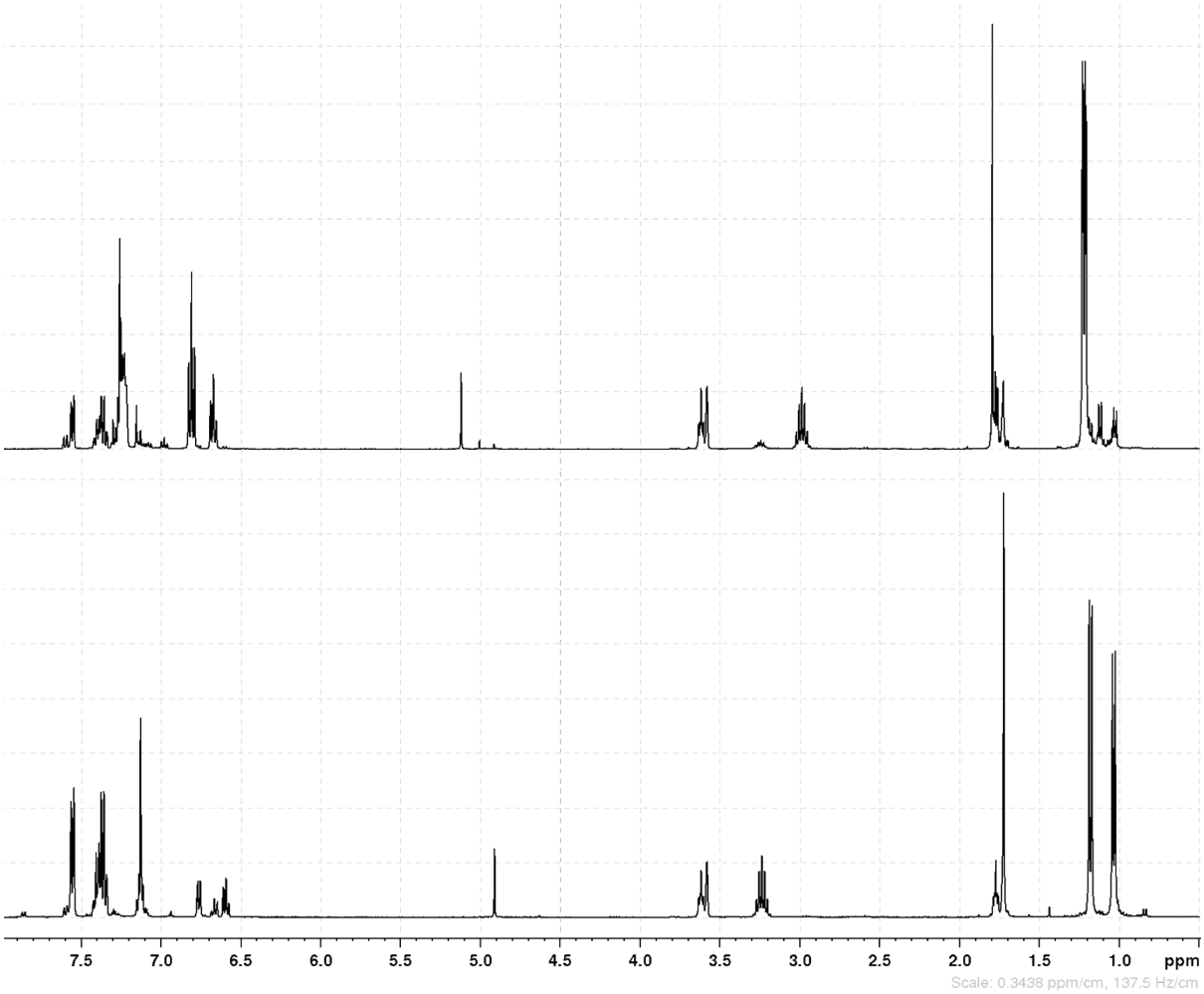

Figure S7 ^{1}H NMR spectrum of (^{Dipp}NacNac)MgPh·THF and BPh_3 in d_8-THF after 5 minutes (bottom) and 8 days at 60^{o}C (top)

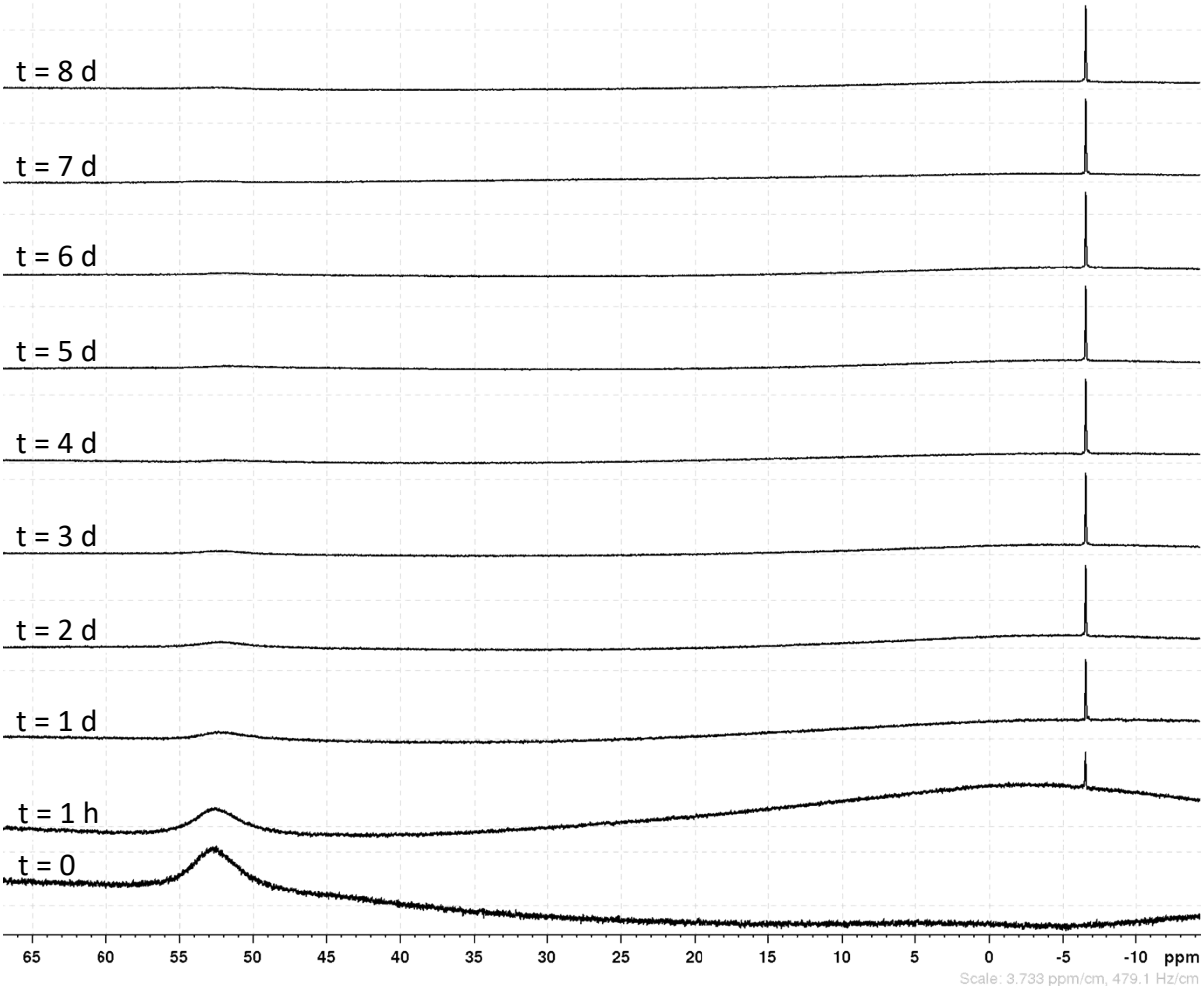

Figure S8 ^{11}B NMR spectrum of NMR scale reaction of (^{Dipp}NacNac)MgPh·THF and BPh_3 in d_8-THF

Synthesis of $GaPh_3{\cdot}OEt_2$

PhLi (0.5 g, 6.0 mmol) in 15 mL of Et_2O was added to a solution of $GaCl_3$ (0.35 g, 2.0 mmol) in 10 mL of Et_2O to form a white precipitate and the reaction mixture was stirred for 1 hour at room temperature. After filtration of the precipitate the remaining colourless solution was concentrated in vacuo until a solid appears. This was redissolved with gentle heating and a crop of colourless crystals was obtained by slow cooling. Yield: 0.53 g (71%).

^{1}H NMR (400 MHz, 298K, C_6D_6): δ = 7.94 (d, 6H, *ortho*-CH), 7.38 (t, 6H, *meta*-CH), 7.31 (t, 3H, *para*-CH), 3.29 (q, 4H, CH_2, Et_2O), 0.56 (t, 6H, CH_3, Et_2O); **^{13}C NMR (100.6 MHz, 298K, C_6D_6):** δ = 147.0 ($C_{quaternary}$, Ph), 137.9 (*C*H, Ph), 128.4 (*C*H, Ph), 128.1 (*C*H, Ph), 65.8 (Et_2O), 13.6 (Et_2O).

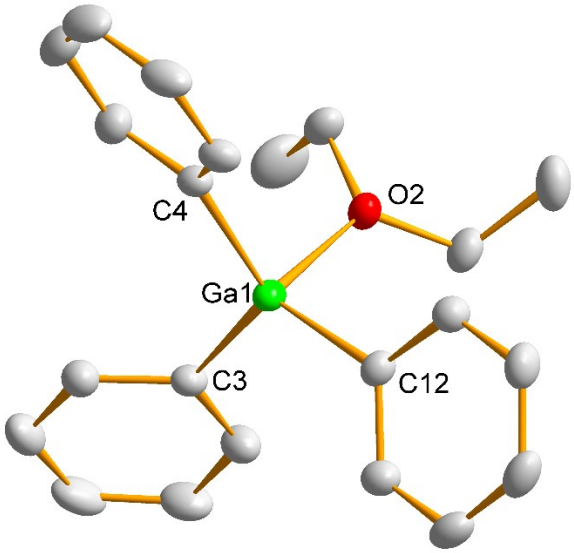

Figure S9 Molecular structure of $GaPh_3 \cdot OEt_2$ with hydrogen atoms omitted for clarity and with thermal ellipsoids drawn at 50% probability.

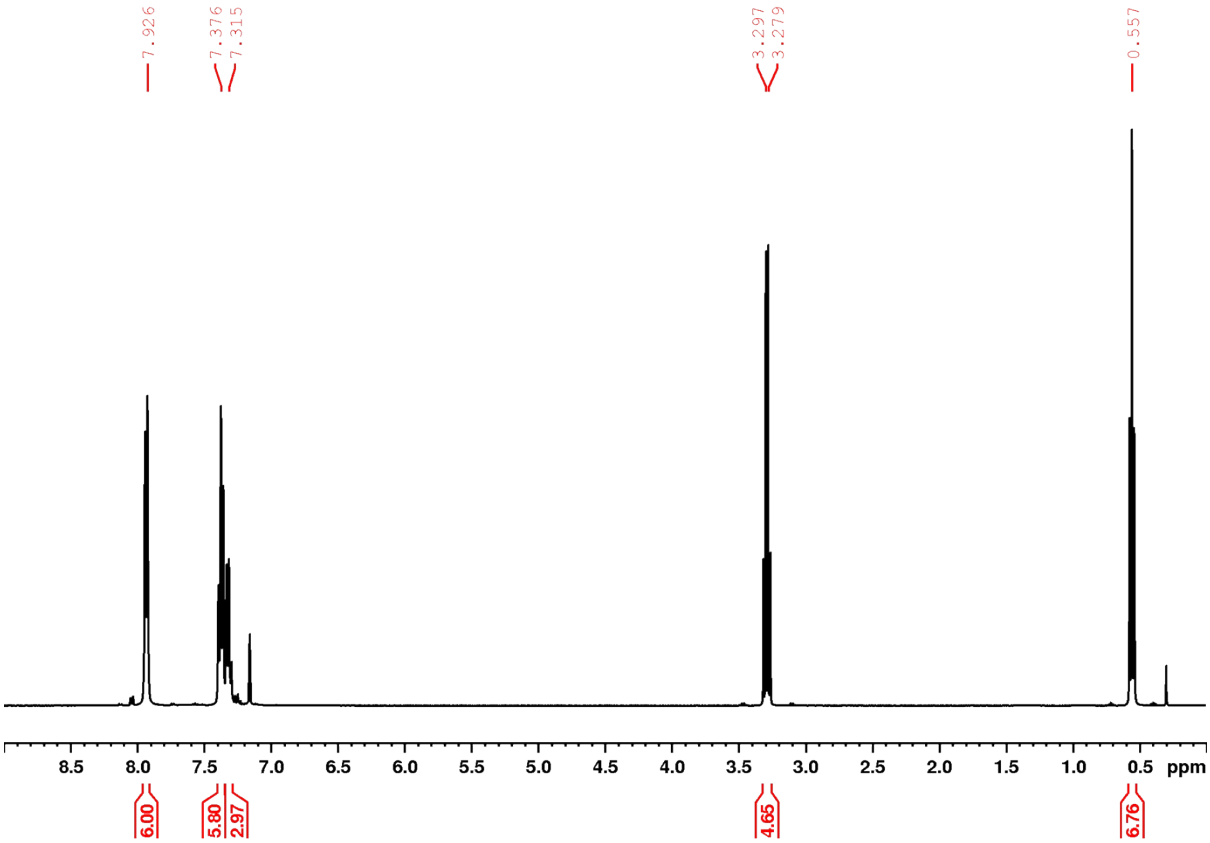

Figure S10 ^1H NMR spectrum of $GaPh_3 \cdot OEt_2$ in C_6D_6

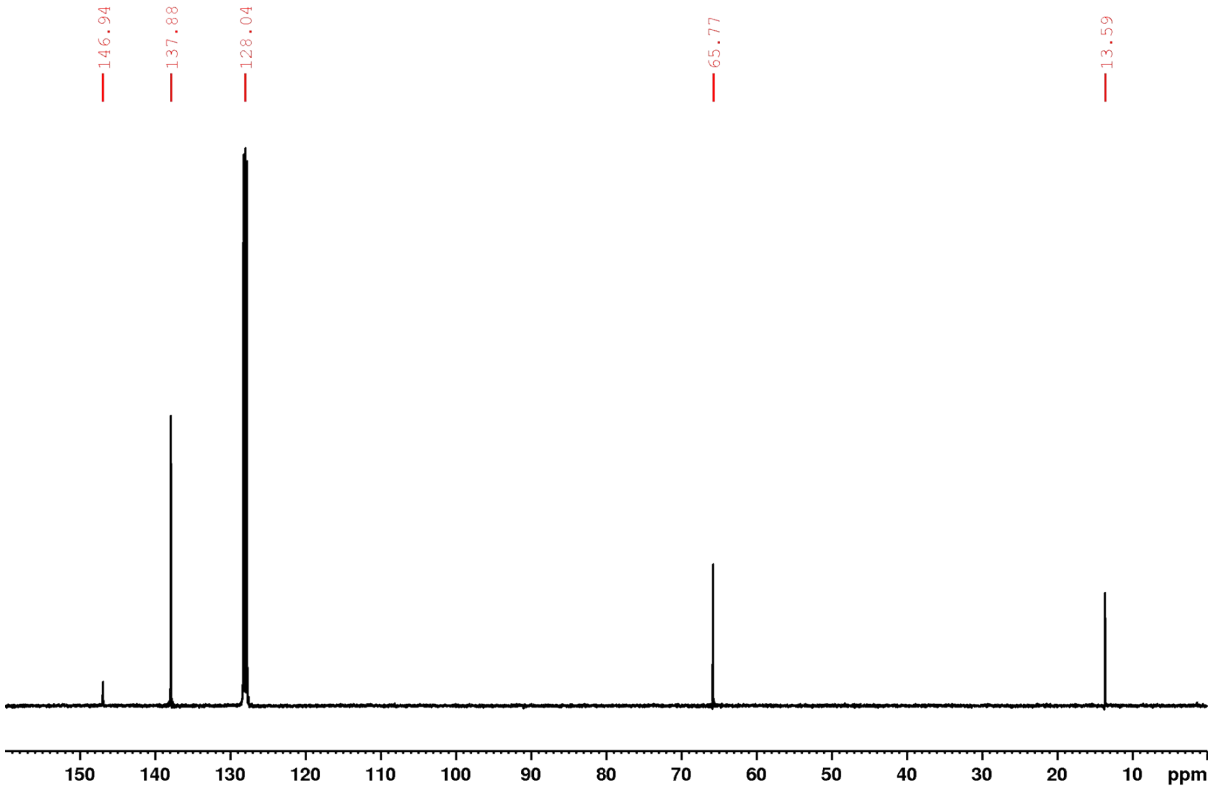

Figure S11 ^{13}C NMR spectrum of $GaPh_3 \cdot OEt_2$ in C_6D_6

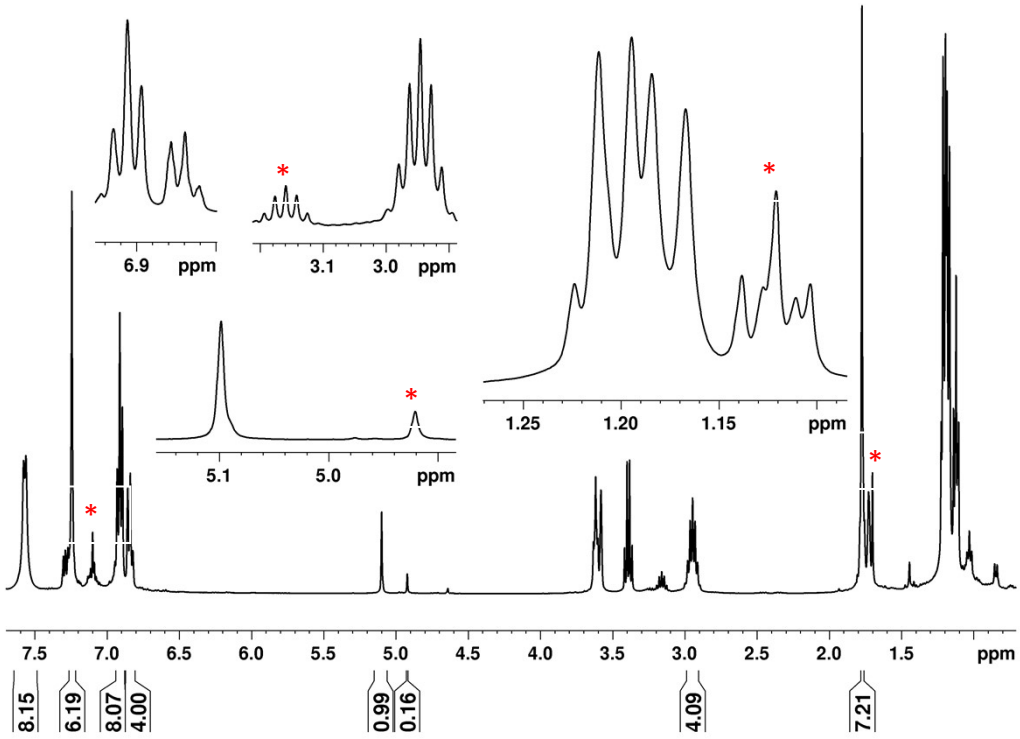

Figure S12 ^{1}H NMR spectrum of (^{Dipp}NacNac)MgPh·THF and $GaPh_3$ in d_8-THF after 15 minutes. There is a small amount of (^{Dipp}NacNac)MgPh·THF present on account of the difficulty of accurately weighing both reagents for an NMR scale reaction, these are marked with a *

Synthesis of (^{Dipp}NacNac)Mg(*p*-Tol)·THF

Mg(*p*-Tol)$_2$·0.1THF·0.2Dioxane (0.464 g, 2 mmol) was added to a solution of ^{Dipp}NacNac(H) (0.84 g, 2.0 mmol) in 10 mL of THF. The resulting solution was refluxed for two hours, after cooling down the reaction mixture the volume of THF was reduced and hexane was layered on top. Colourless crystals were obtained by cooling of this solution to -36°C. Yield: 0.28 g (46 %).

^{1}H NMR (MHz 400, 298 K, d_8-THF): δ = 7.12 (m, 6H, *m/p*-C*H*, ^{Dipp}Ar), 6.64 (d, 2H, *o*-C*H*, Tolyl), 6.44 (d, 2H, *m*-C*H*, Tolyl), 4.90 (s, 1H, C*H*, NacNac), 3.61 (m, 4H, THF), 3.23 (sept, 4H, C*H*(CH_3)$_2$, *i*Pr), 1.98 (s, 3H, Ph-CH_3), 1.77 (m, 4H, THF), 1.71 (s, 6H, C-CH_3), 1.17 (d, 12H, CH(CH_3)$_2$, *i*Pr), 1.03 (d, 12H, CH(CH_3)$_2$, *i*Pr) **^{13}C NMR (MHz 100.6, 298 K, d_8-THF):** δ = 168.7 (*C*-CH_3, NacNac), 160.6 ($C_{quaternary}$, Tolyl), 146.1 ($C_{quaternary}$, Ar*), 142.9 (*C*H, Ar*), 140.8 (*C*H, Tolyl), 128.6 ($C_{quaternary}$, Tolyl), 126.6 (*C*H, Ar*), 125.2 (*C*H, Tolyl), 123.9 (*C*H, Ar*), 94.6 (*C*H, NacNac), 67.9 (THF), 28.5 (*C*H, *i*Pr), 26.1 (THF), 25.0 (*C*H_3, *i*Pr), 24.2 (*C*H_3, *i*Pr), 23.9 (*C*–CH_3, NacNac), 21.3 (Ph-*C*H_3).

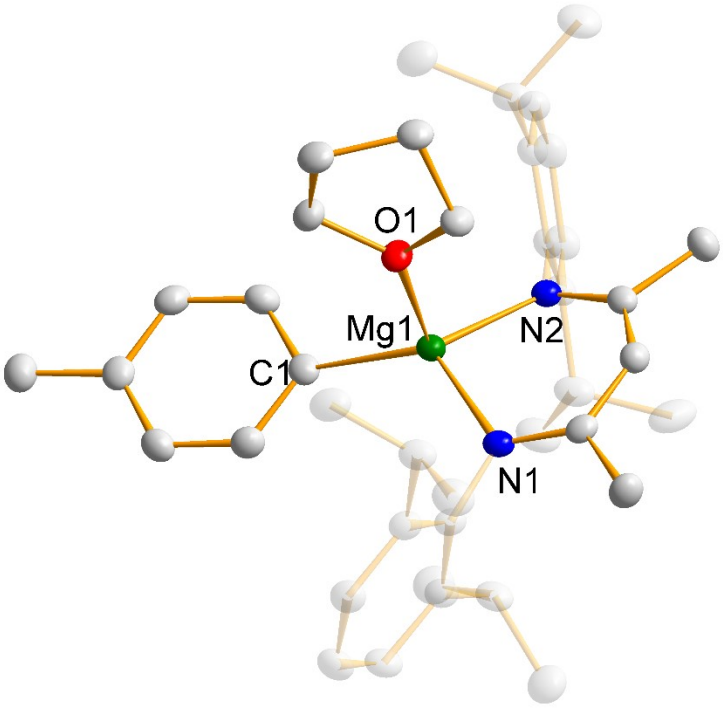

Figure S13 Molecular structure of (^{Dipp}NacNac)Mg(*p*-Tol)·THF with hydrogen atoms omitted for clarity and with thermal ellipsoids drawn at 50% probability.

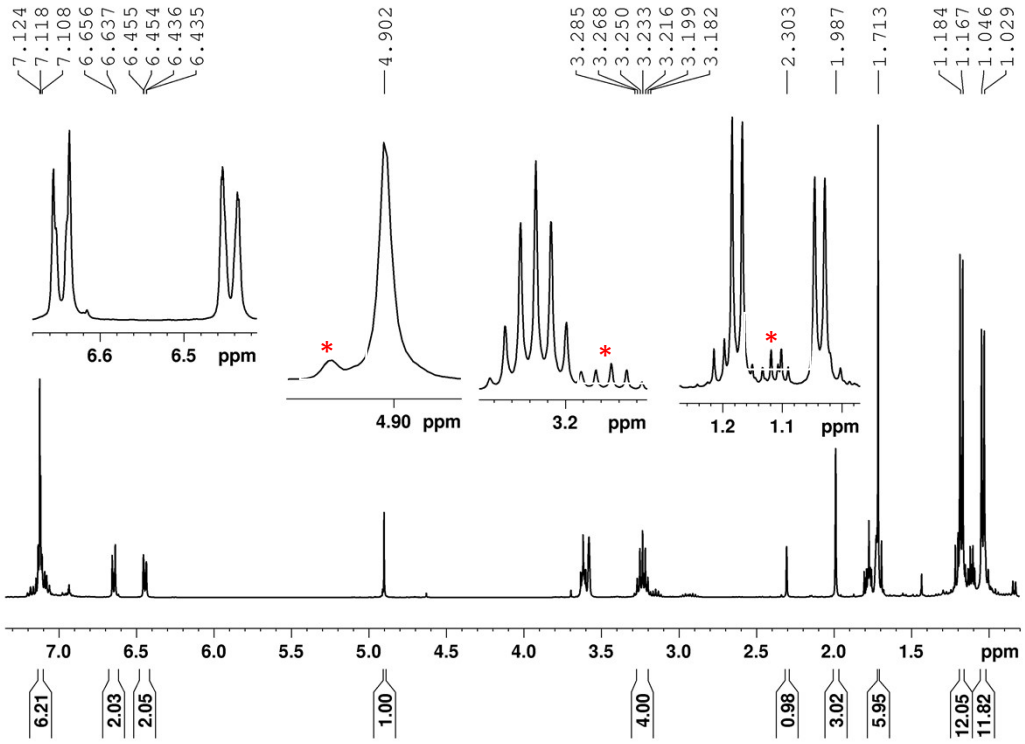

Figure S14 ^{1}H NMR spectrum of (^{Dipp}NacNac)Mg(*p*-Tol)·THF in d_8-THF. A small amount of NacNac containing impurity is denoted with *

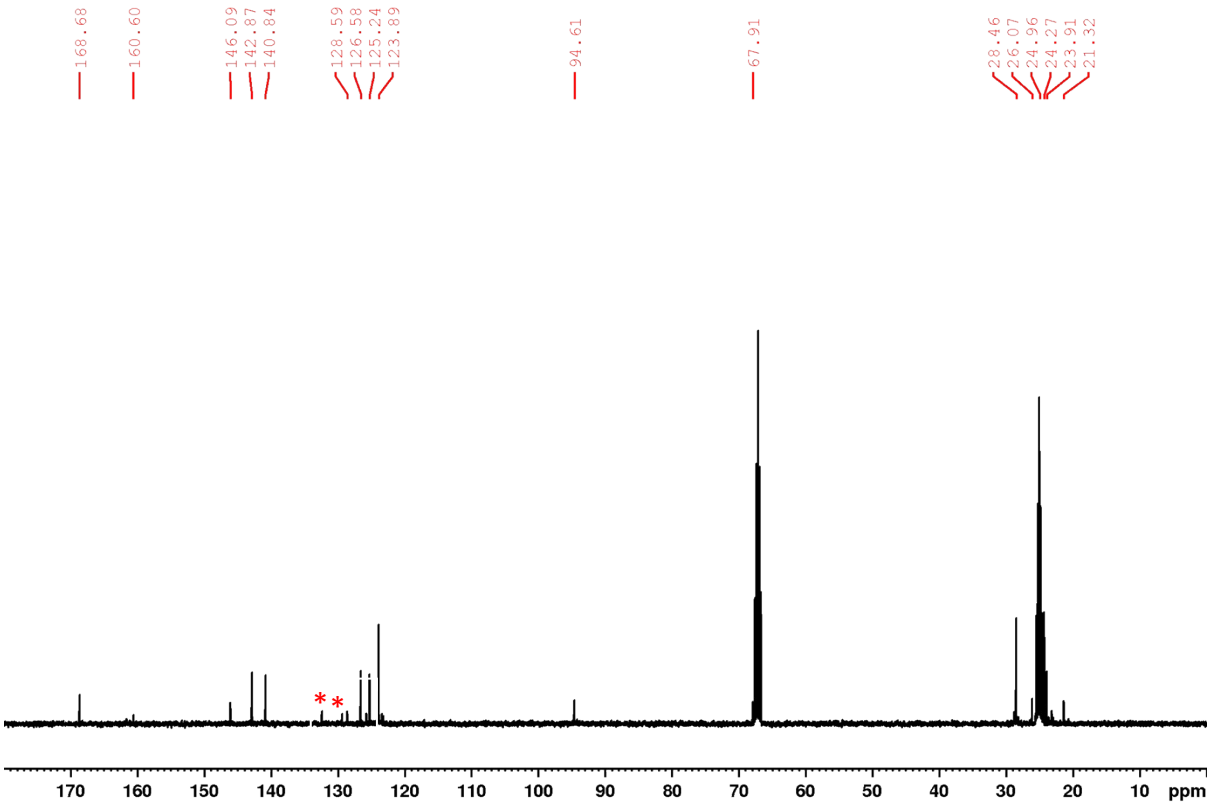

Figure S15 ^{13}C NMR spectrum of (^{Dipp}NacNac)Mg(*p*-Tol)·THF in d_8-THF. A small amount of NacNac containing impurity is denoted with *

Synthesis of Al(*p*-Tol)$_3$·OEt$_2$

(*p*-Tol)Li (0.88 g, 9.0 mmol, synthesised by metal-halogen exchange of *n*BuLi with *p*-iodotoluene) in 20 mL of Et_2O was added to a solution of $AlCl_3$ (0.39 g, 3.0 mmol) in 20 mL of Et_2O to form a white precipitate and the reaction mixture was stirred overnight at room temperature. After filtration of the precipitate the remaining colourless solution was concentrated in vacuo until a solid appears. This was redissolved with gentle heating and a crop of colourless crystals was obtained by cooling to -36°C. Yield: 0.53 g (47%).

^{1}H NMR (400 MHz, 298 K, d_8-THF): δ = 7.61 (d, 6H, *o*-C*H,* Ar), 7.04 (d, 6H, *m*-C*H,* Ar), 3.38 (q, 4H, OEt_2), 2.27 (s, 9H, Ph-CH_3), 1.11 (t, 6H, OEt_2); **^{13}C NMR (100.6 MHz, 298 K, d_8-THF):** δ = 144.5 (*ipso*-*C,* Ar), 138.8 (*o*-*C*H, Ar), 136.7 (*p*-*C*H, Ar), 128.2 (*m*-*C*H, Ar), 66.1 (O-CH_2-CH_3, Et_2O), 21.4 (Ph-CH_3), 15.4 (O-CH_2-CH_3, Et_2O) **^{27}Al NMR (104.2 MHz, 298K, d_8-THF):** δ = 70.2 (s).

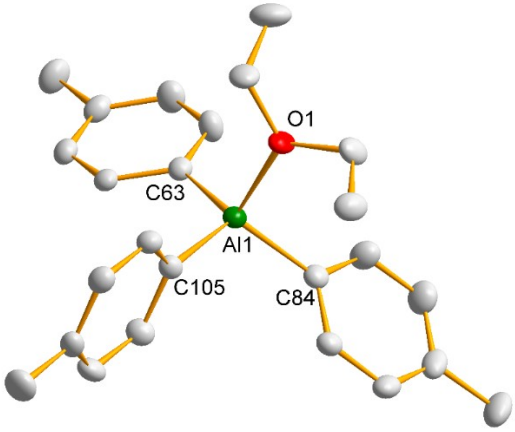

Figure S16 Molecular structure of one of the four independent molecules of Al(*p*-Tol)$_3$·OEt$_2$ with hydrogen atoms omitted for clarity and thermal ellipsoids drawn at 50% probability.

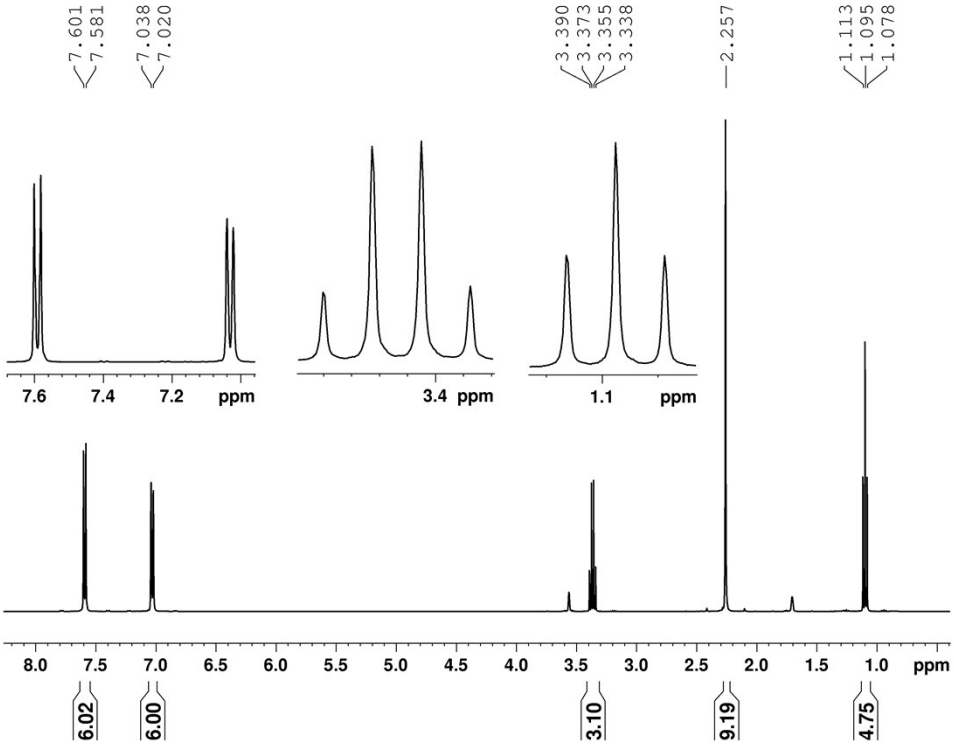

Figure S17 ^{1}H NMR spectrum of Al(*p*-Tol)$_3$·OEt$_2$ in d_8-THF

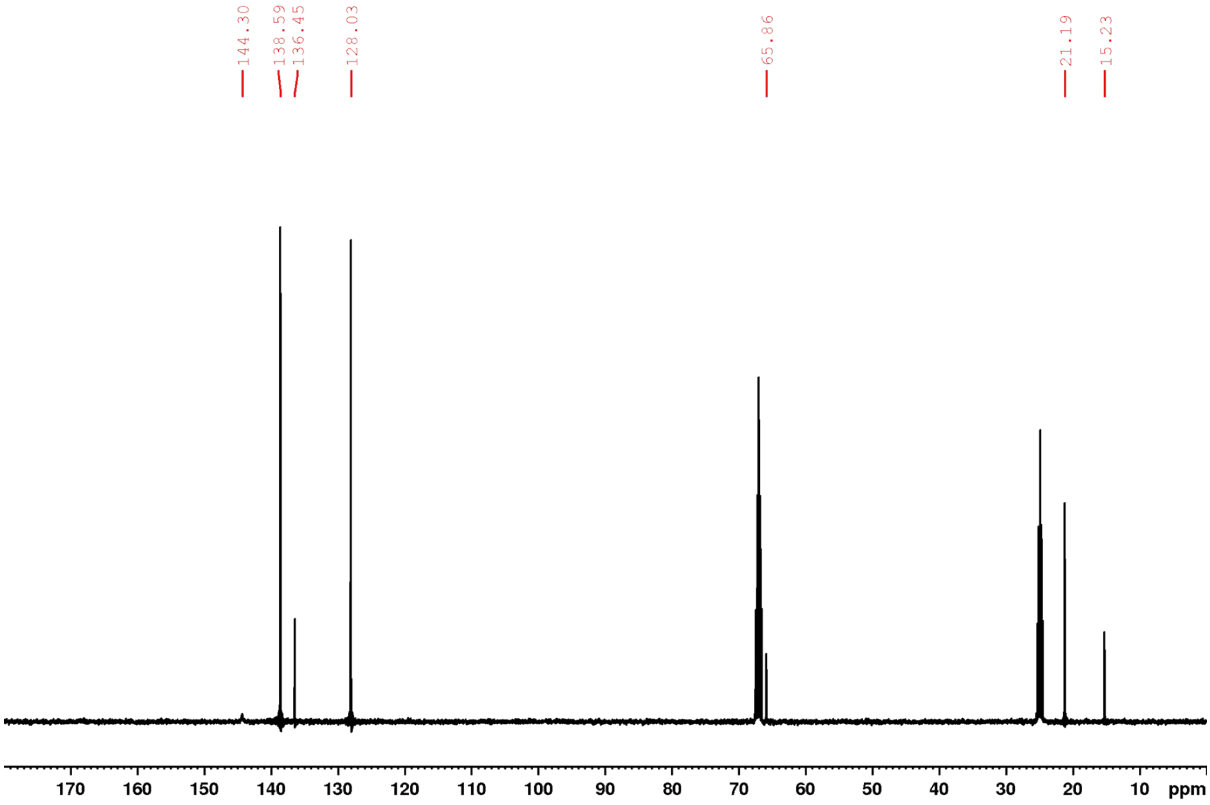

Figure S18 ^{13}C NMR spectrum of Al(*p*-Tol)$_3$·OEt$_2$ in d$_8$-THF

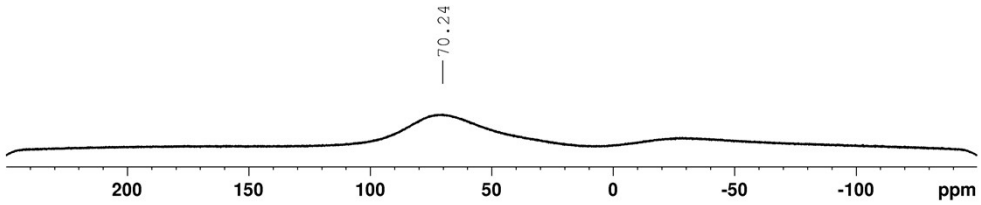

Figure S19 ^{27}Al NMR spectrum of Al(*p*-Tol)$_3$·OEt$_2$ in d$_8$-THF

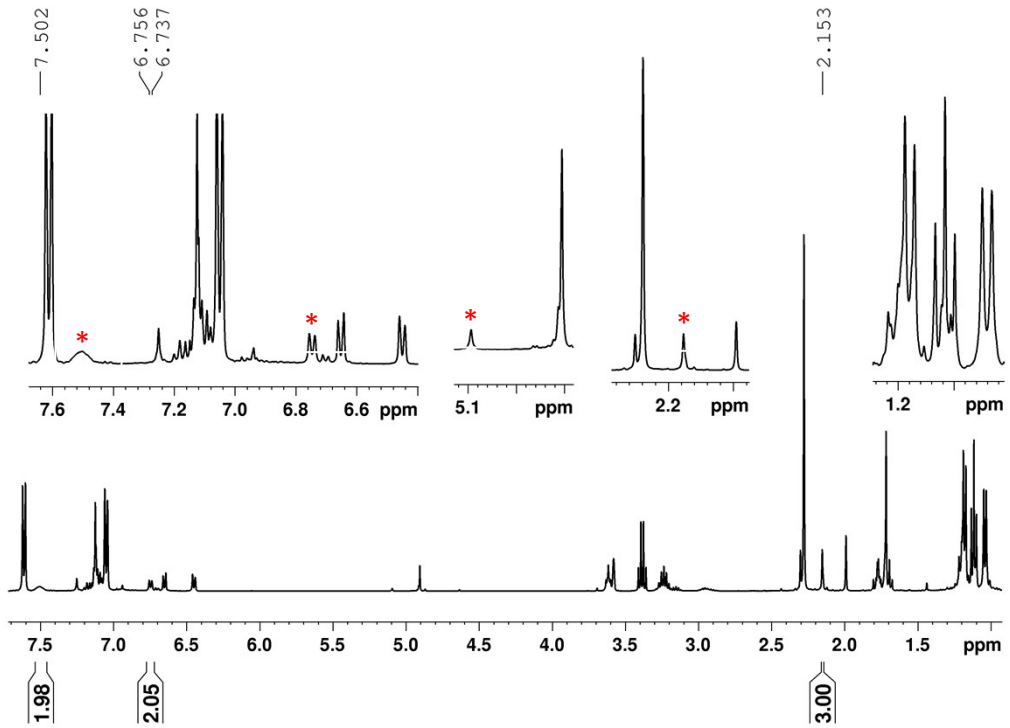

Figure S20 ^{1}H NMR spectrum of (^{Dipp}NacNac)Mg(*p*-Tol)·THF and Al(*p*-Tol)$_3$·OEt$_2$ in d$_8$-THF after 56 hours. New resonances corresponding to [(^{Dipp}NacNac)Mg·2THF]$^+$ [Al(*p*-Tol)$_4$]$^-$ (**4**) are highlighted with *

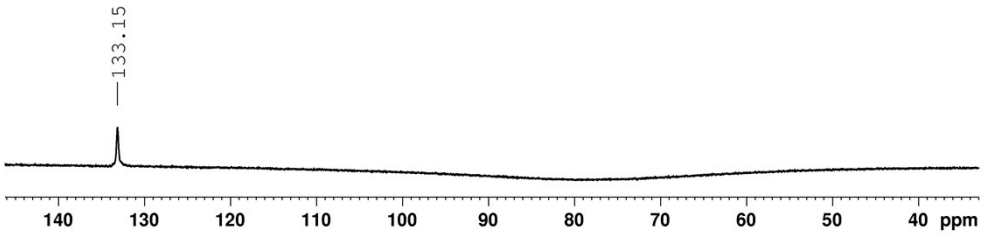

Figure S21 ^{27}Al NMR spectrum of (^{Dipp}NacNac)Mg(*p*-Tol)·THF and Al(*p*-Tol)$_3$·OEt$_2$ in d$_8$-THF after 56 hours

Synthesis of (^{Dipp}NacNac)Mg(pyr)·THF

*n*Bu$_2$Mg (5 mL, 5.0 mmol, 1 M in heptanes) was added to a solution of ^{Dipp}NacNac(H) (2.08 g, 5.0 mmol) in 15 mL of THF. After leaving the reaction mixture to stir at room temperature for 1.5 h pyrrole (0.35 mL, 5.0 mmol) was added to the solution and an exothermic reaction occurred. Within 1 h a solid was obtained, redissolving it by gently heating and slowly cooling at room temperature afforded colourless crystals. Yield: 2.350 g (80%).

^{1}H NMR (400 MHz, 298K, C$_6$D$_6$): δ = 7.17 (broad m obscured by solvent signal, 6H, C*H*, Ar*), 6.60 (t, 2H, J = 1.73Hz, NC*H*, pyrrolyl), 6.48 (t, 2H, J = 1.66 Hz, β-C*H,* pyrrolyl), 4.87 (s, 1H, C*H*, NacNac), 3.37 (broad s, 2H, C*H*, *i*Pr), 3.29 (t, 4H, J = 6.67 Hz, THF), 3.16 (broad s, 2H, C*H*, *i*Pr), 1.70 (s, 6H, C-C*H*$_3$, NacNac), 1.20 (d, 12H, J = 6.74 Hz, C*H*$_3$, *i*Pr), 1.10 (d, 12H, J = 6.80 Hz, C*H*$_3$, *i*Pr), 1.05 (t, 4H, J = 6.75 Hz, THF); **^{13}C NMR (100.6 MHz, 298K, C$_6$D$_6$):** δ = 169.4 (*C*-CH$_3$, NacNac), 145.0 (*C*$_{quarternary}$, Ar*), 125.8 (*C*H, Ar*), 125.7 (N*C*H, pyrrolyl), 124.7 (*C*H, Ar*), 123.4 (*C*$_{quarternary}$,

Ar*), 108.3 (β-*C*H, pyrrolyl), 94.5 (*C*H, NacNac), 69.9 (THF), 28.3 (*C*H, *i*Pr), 25.1 (*C*H_3, *i*Pr), 24.6 (THF), 24.5 (*C*H_3, *i*Pr), 24.0 (C-*C*H_3, NacNac).

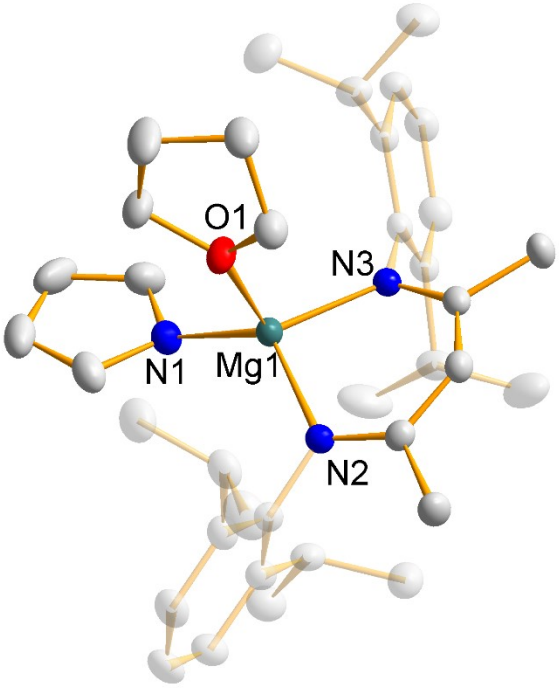

Figure S22 Molecular structure of ([Dipp]NacNac)Mg(pyr)·THF with hydrogen atoms omitted for clarity and with thermal ellipsoids drawn at 50% probability.

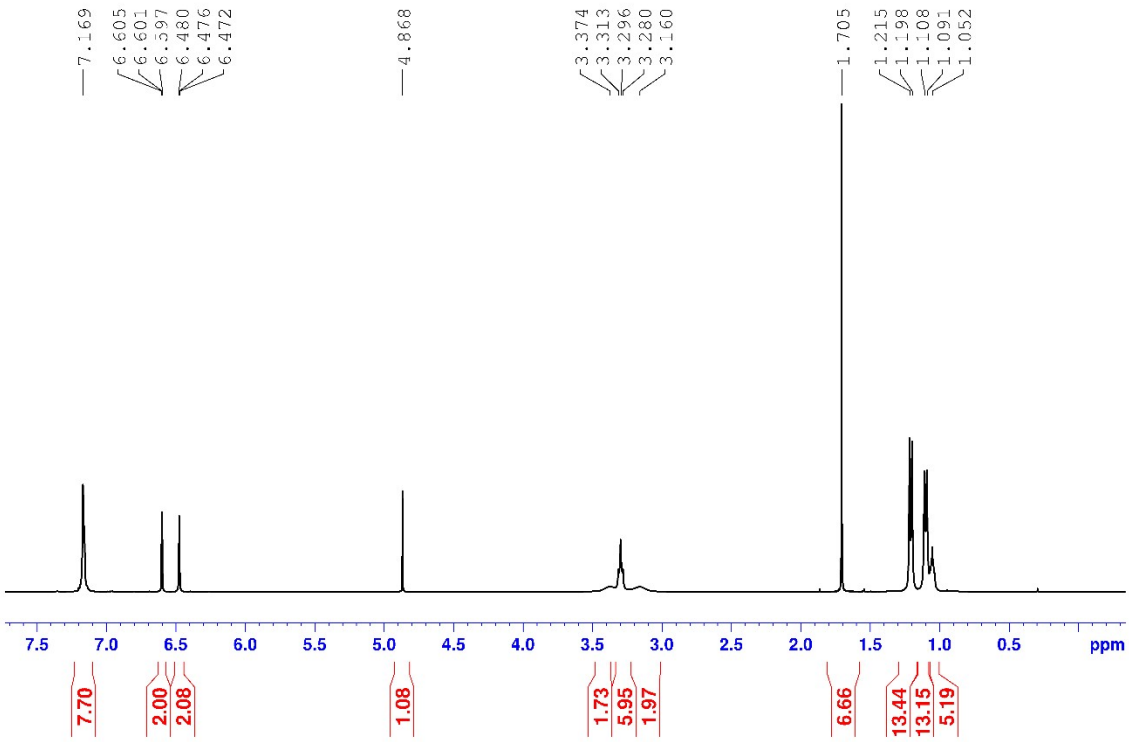

Figure S23 ^{1}H NMR spectrum of ([Dipp]NacNac)Mg(pyr)·THF in C_6D_6

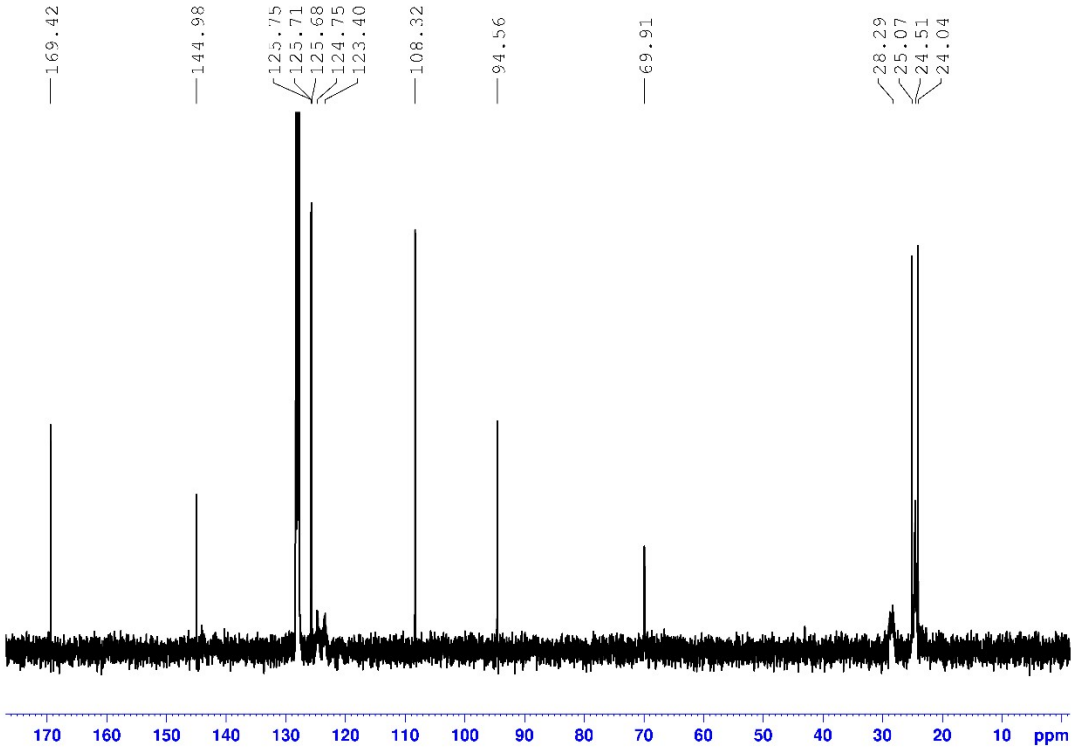

Figure S24 ^{13}C NMR spectrum of (^{Dipp}NacNac)Mg(pyr)·THF in C_6D_6

Synthesis of $Al(pyr)_3{\cdot}OEt_2$

^{n}BuLi (3.75 mL, 6.0 mmol, 1.6 M in hexane) was added to a solution of pyrrole (0.42 mL, 6.0 mmol) in 15 mL of diethyl ether to form a yellow precipitate and the reaction mixture was stirred for 30 min at room temperature. $AlCl_3$ (0.266 g, 2.0 mmol) was added to the reaction mixture at 0 °C and stirred at room temperature for 3h to form two layers, one dark brown layer with a white precipitate and one colourless. The diethyl ether was then removed in vacuo and replaced by 30 mL of toluene to give a brown solution with a white precipitate. The solid was filtered off and the remaining solution concentrated. Crystalline blocks grow from a toluene:hexane mixture with a drop of Et_2O at −18 °C. Yield: 0.386 g (65%).

^{1}H NMR (400 MHz, 298K, C_6D_6): δ = 7.00 (t, 6H, J = 1.84 Hz, NC*H*, pyrrole), 6.64 (t, 6H, J = 1.85 Hz, β-C*H*, pyrrole), 3.11 (q, 4H, J = 7.08 Hz, CH_2, Et_2O), 0.34 (t, 6H, J = 7.07 Hz, CH_3, Et_2O); **^{13}C NMR (100.6 MHz, 298K, C_6D_6):** δ = 125.2 (N*C*H, pyrrolyl), 111.8 (β-*C*H; pyrrolyl), 70.0 (Et_2O), 12.8 (Et_2O).

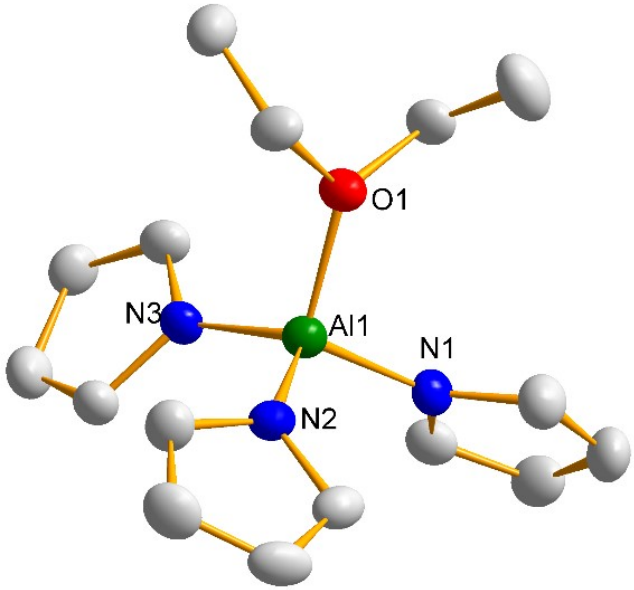

Figure S25 Molecular structure of $Al(pyr)_3 \cdot OEt_2$ with hydrogen atoms omitted for clarity and with thermal ellipsoids drawn at 50% probability.

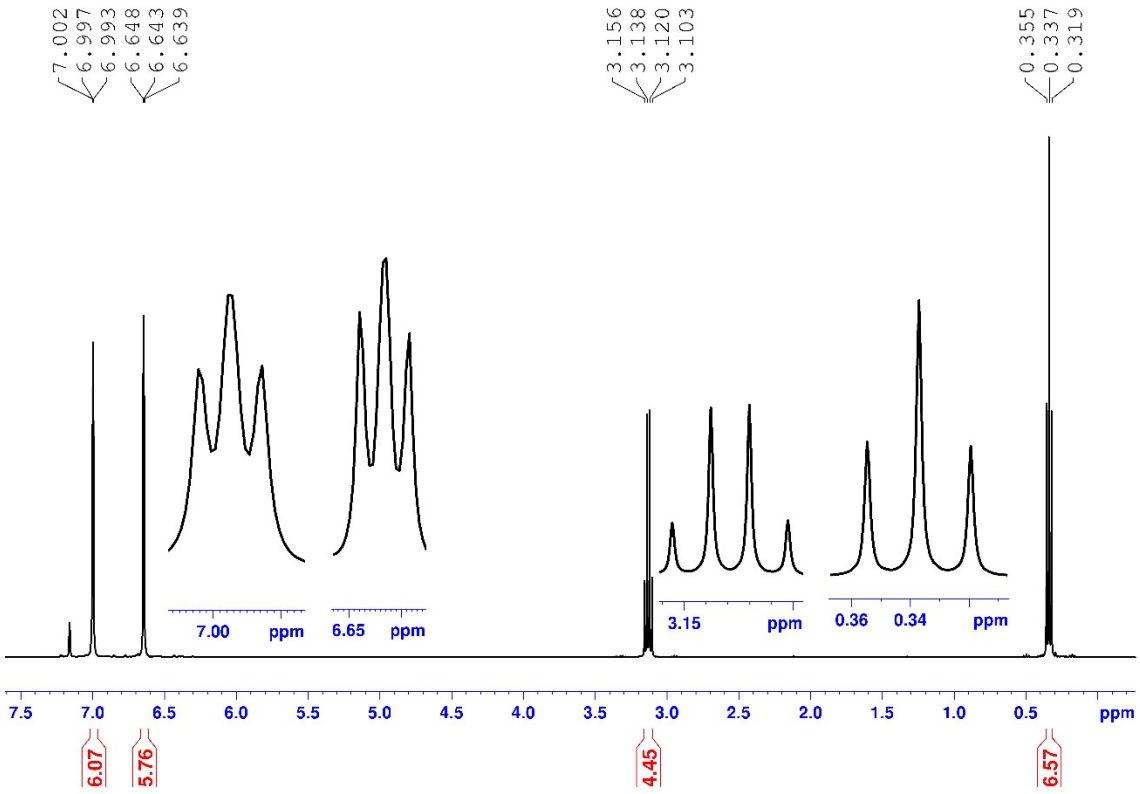

Figure S26 ^1H NMR spectrum of $Al(pyr)_3 \cdot OEt_2$ in C_6D_6

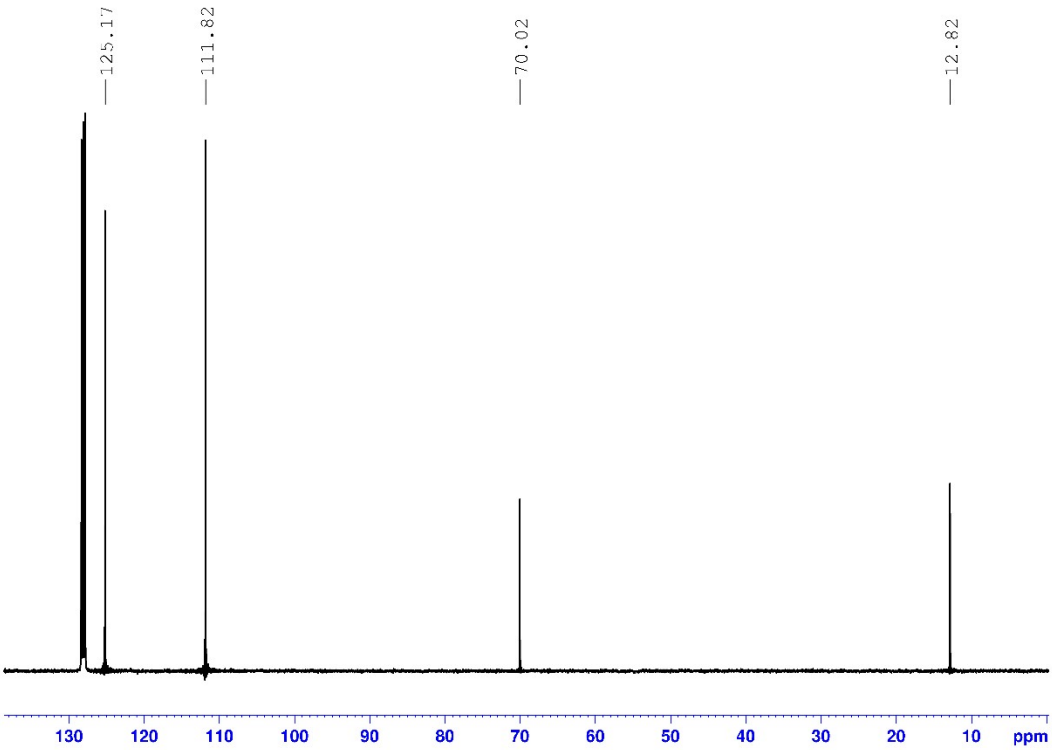

Figure S27 ^{13}C NMR spectrum of Al(pyr)$_3$·OEt$_2$ in C_6D_6

Synthesis of [(^{Dipp}NacNac)Mg·2THF]$^+$ [Al(pyr)$_4$]$^-$ (**5**)

A solution of Al(pyr)$_3$·OEt$_2$ (0.84g, 2.8 mmol) in THF was added to a solution of (NacNac)Mg(pyr)·THF (1.64 g, 2.8 mmol) in 10 mL of THF. The reaction mixture was stirred for 2 h at room temperature. The solvent was removed *in vacuo* and 15 mL of hexane was added to form a slightly hazy solution. After filtration the filtrate was placed at −20 °C to form colourless crystals. Crystalline yield 1.25 g (50%).

^{1}H NMR (400 MHz, 298K, C_6D_6): δ = 7.24 (broad s, 8H, NC*H*, pyrrolyl), 7.15 (obscured by solvent signal, 4H, C*H*, Ar*), 7.06 (d, 2H, J = 7.40 Hz, C*H*, Ar*), 6.50 (broad s, 8H, β-C*H*, pyrrolyl), 4.70 (s, 1H, C*H*, NacNac), 3.12 (broad s, 8H, THF), 2.72 (sept, 4H, J = 6.07 Hz, C*H*, *i*Pr), 1.48 (s, 6H, C-C*H*$_3$, NacNac), 1.19 (broad, 8H, THF), 1.08 (d, 12H, J = 6.84 Hz, C*H*$_3$ *i*Pr), 1.01 (d, 12H, J = 6.35 Hz, C*H*$_3$ *i*Pr); **^{13}C NMR (100.6 MHz, 298K, C_6D_6):** δ = 171.4 (*C*-CH$_3$, NacNac), 143.1 (*C*$_{quaternary}$, Ar*), 142.0 (*C*H, Ar*), 126.9 (*C*$_{quaternary}$, Ar*), 126.1 (N*C*H, pyrrolyl), 124.6 (*C*H, Ar*), 109.3 (β-*C*H, pyrrolyl), 95.3 (*C*H, NacNac), 71.3 (THF), 28.4 (*C*H, *i*Pr), 25.3 (THF), 25.0 (*C*H$_3$, *i*Pr), 24.3 (C-*C*H$_3$, NacNac), 24.1 (*C*H$_3$, *i*Pr). **^{27}Al NMR (MHz 104.2, 298K, C_6D_6):** δ = 98.1 (s).

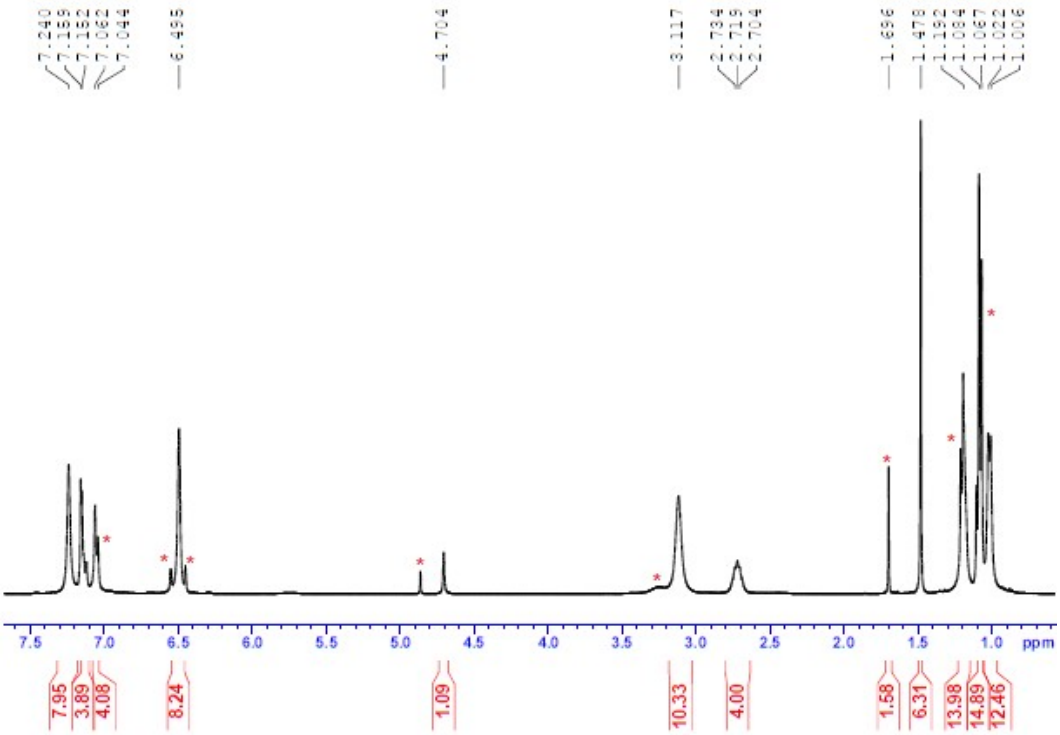

Figure S28 ^{1}H NMR spectrum of [(^{Dipp}NacNac)Mg·2THF]$^{+}$ [Al(pyr)$_4$]$^{-}$ (**5**) in C_6D_6 with (^{Dipp}NacNac)Mg(pyr)·THF highlighted with *

6

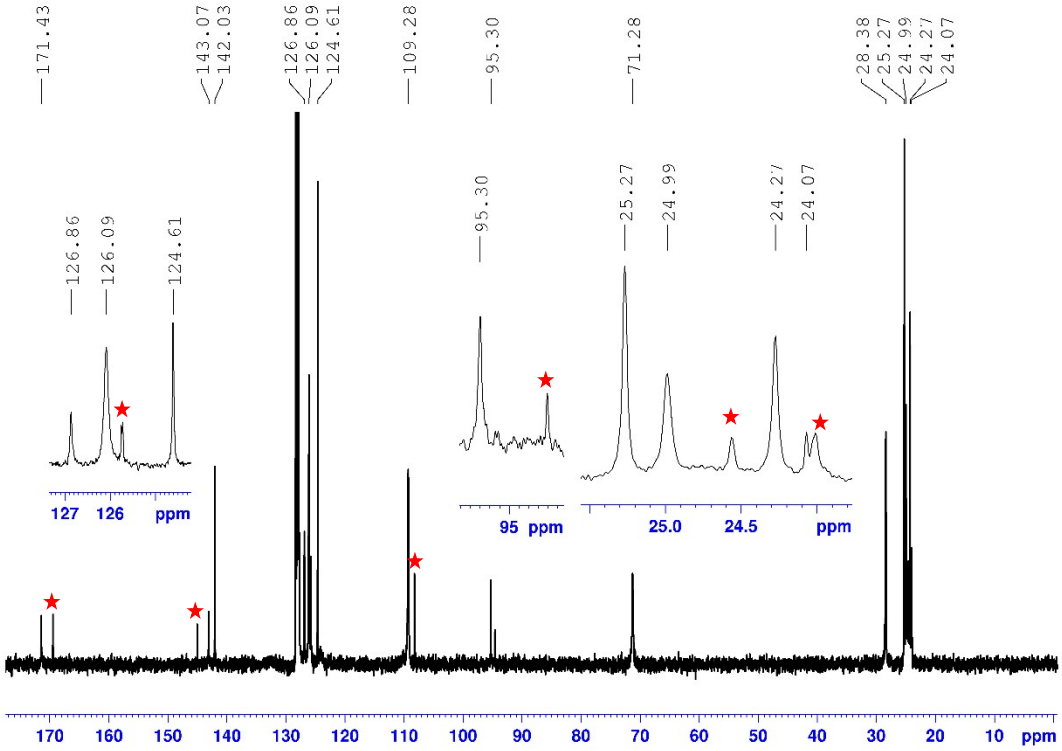

Figure S29 ^{13}C NMR spectrum of [(^{Dipp}NacNac)Mg·2THF]$^{+}$ [Al(pyr)$_4$]$^{-}$ (**5**) in C_6D_6 with (^{Dipp}NacNac)Mg(pyr)·THF highlighted with *

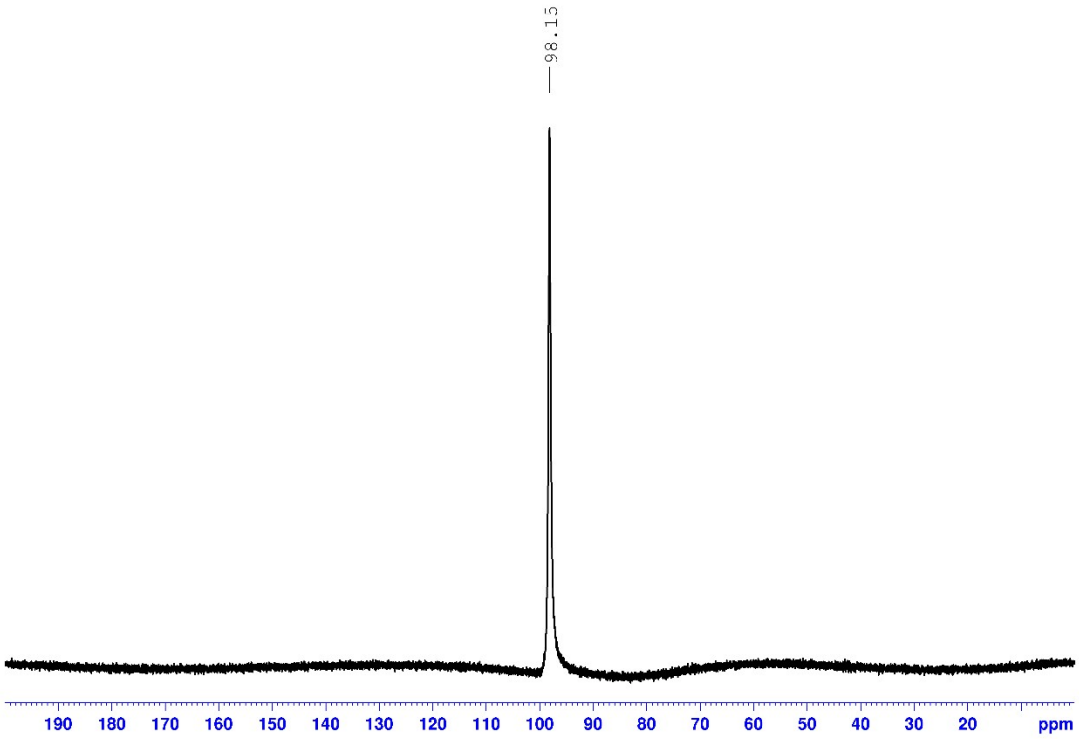

Figure S30 ^{27}Al NMR spectrum of [(^{Dipp}NacNac)Mg·2THF]$^{+}$ [Al(pyr)$_4$]$^{-}$ (**5**) in C_6D_6

Table S1 Selected crystallographic and refinement parameters

	(^{Dipp}NacNac)MgPh·THF	**1a**	$GaPh_3 \cdot OEt_2$	(^{Dipp}NacNac)Mg(*p*-Tol)·THF	Al(*p*-Tol)$_3$·OEt_2	(^{Dipp}NacNac)Mg(pyr)·THF	Al(pyr)$_3$·OEt_2	**5**
Empirical formula	$C_{39}H_{54}MgN_2O$	$C_{72}H_{88}Al_2MgO_6$	$C_{22}H_{25}GaO$	$C_{40}H_{56}MgN_2O$	$C_{25}H_{31}AlO$	$C_{37}H_{53}MgN_3O$	$C_{16}H_{22}AlN_3O$	$C_{53}H_{73}AlMgN_6O_2$
MW	591.15	1127.69	375.14	605.17	374.48	580.13	299.34	877.46
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic	Orthorhombic	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$	$P2_1/c$	P-1	$Pna2_1$	$P2_1/n$	$P2_1/c$	C2/c
Temp. (K)	153(2)	123(2)	123(2)	100(2)	100(2)	123(2)	138(2)	123(2)
a (Å)	10.7108(2)	18.7709(5)	12.2499(2)	9.2045(2)	14.2675(1)	10.8131(1)	11.0633(14)	38.2698(12)
b (Å)	19.4049(3)	13.0019(3)	7.4542(1)	12.8235(3)	13.0762(1)	19.0170(3)	7.7845(10)	9.7287(3)
c (Å)	17.7729(3)	25.8666(7)	21.2085(3)	15.2783(3)	47.5036(2)	17.3003(2)	19.436(3)	27.2433(8)
α (°)	90	90	90	97.717(2)	90	90	90	90
β (°)	106.699(2)	91.115(2)	100.416(1)	91.981(2)	90	105.924(1)	101.884(12)	92.726(3)
γ (°)	90	90	90	94.629(2)	90	90	90	90
V (Å^3)	3538.17(11)	6311.7(3)	1904.70(5)	1779.31(7)	8862.49(10)	3420.99(8)	1638.0(4)	10131.6(5)
Z	4	4	4	2	16	4	4	8
λ (Å)	0.71073	1.54184	1.54184	1.54184	1.54184	1.54184	0.71073	1.54184
2Θmax (°)	57.782	146.794	146.290	146.466	146.252	146.148	53.994	139.998
Reflns. collected	53508	35631	12719	53061	292014	28308	16818	33647
Unique Reflns.	8703	12426	3757	7114	17727	6765	3515	9585
R_{int}	0.0324	0.0473	0.0324	0.0446	0.0717	0.0228	0.0820	0.0712
GooF	1.030	1.007	1.072	1.079	1.059	1.028	1.050	1.070
R[I>2σ(I)]	0.0437	0.0524	0.0281	0.0428	0.0391	0.0417	0.0589	0.0523
ωR2	0.1009	0.1328	0.0767	0.1189	0.1085	0.1148	0.1198	0.1603
CCDC	2073561	2073562	2288826	2288828	2288827	2073563	2073564	2073565

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[2] G.M. Sheldrick, *Acta Crystallogr. Sect. A Found. Crystallogr.*, **2008**, *64*, 112-122.
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