

Supplementary Information for

From Alkaline Earth to Coinage Metal Carboranyls

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Synthetic Details

General Considerations

All manipulations were carried out using standard Schlenk line and glovebox techniques under an inert atmosphere of argon. NMR experiments were conducted in J-Young tap NMR tubes prepared in a glovebox. NMR spectra were recorded on a Bruker BioSpin GmbH spectrometer operating at 400.13 MHz (^1H) and 100.62 MHz (^{13}C) or on an Agilent ProPulse spectrometer operating at 500.06 MHz (^1H), 125.75 MHz (^{13}C) and 160.44 MHz (^{11}B). Elemental analyses were performed at Elemental Microanalysis Ltd., Okehampton, Devon, UK. Solvents were dried by passage through a commercially available solvent purification system and stored under argon in ampoules over 4 Å molecular sieves. C_6D_6 was purchased from Merck, dried over potassium before distilling and storage over molecular sieves. $[(\text{BDI})\text{MgBu}]$ ($\text{BDI} = \text{HC}\{(\text{Me})\text{CN}-2,6\text{-}i\text{-Pr}_2\text{C}_6\text{H}_3\}_2$),¹ $[(\text{BDI})\text{CaH}]_2$ and the $\text{N,N}'$ -diisopropyl-4,5-dimethyl-2-ylidene (NHC^{iPr}) copper, silver and gold(I) chlorides were synthesised according to literature procedures.²⁻⁴

Synthesis of $[(\text{BDI})\text{Mg}(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (1)

A J. Young's NMR tube was charged with $[(\text{BDI})\text{Mg}n\text{Bu}]$ (50 mg, 0.1 mmol), *ortho*-carborane (14.4 mg, 0.1 mmol) and C_6D_6 (0.6 cm³). The sample was heated at 100 °C for 16 hours before removing all volatiles under reduced pressure. The crude solid was recrystallised from a hexane-toluene mixture, affording colourless crystals of $[(\text{BDI})\text{Mg}(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (1). Yield: 23 mg, 47%.

^1H NMR (C_6D_6): $\delta = 7.11\text{-}6.99$ (m, ^{Dipp}ArH, 6H), 4.83 (s, HCCN, 1H), 3.40-1.70 (br m, BH, 10H), 2.87 (hept, ³ $J_{\text{HH}} = 6.9$ Hz, $\text{HC}(\text{CH}_3)_2$, 4H), 2.41 (s, HCcarborane, 1H), 1.57 (s, NCCH_3 , 6H), 1.21 (d, ³ $J_{\text{HH}} = 6.9$ Hz, $\text{HC}(\text{CH}_3)_2$, 12H), 1.05 (d, ³ $J_{\text{HH}} = 6.8$ Hz, $\text{HC}(\text{CH}_3)_2$, 12H).

$^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6): $\delta = 171.0$ (NCCH_3), 142.9 (*i*- C_6H_3), 141.3 (*o*- C_6H_3), 126.8 (*p*- C_6H_3), 124.7 (*m*- C_6H_3), 95.7 (HCCN), 60.2 (HCcarborane), 29.4 ($\text{HC}(\text{CH}_3)_2$), 24.3 ($\text{HC}(\text{CH}_3)_2$), 23.5 ($\text{HC}(\text{CH}_3)_2$), 23.4 (NCCH_3).

^{11}B NMR (C_6D_6) $\delta = -0.62$ (m), -5.91 (d, $J = 150.4$ Hz), $-8.64\text{-}15.54$ (m).

Anal. Calc. for $\text{C}_{31}\text{H}_{52}\text{N}_2\text{B}_{10}\text{Mg}_1$: C, 63.63; H, 8.96; N, 4.79. Found: C, 63.39; H, 9.00; N, 4.96.

Synthesis of $[(\text{BDI})\text{Ca}(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (2)

A J. Young's NMR tube was charged with $[(\text{BDI})\text{CaH}]_2$ (30 mg, 0.06 mmol), *ortho*-carborane (9.2 mg, 0.06 mmol) and C_6D_6 (0.6 cm³), resulting in effervescence. After *ca.* 20 hours at ambient temperature

the volatiles were removed under reduced pressure affording a colourless solid which could be recrystallised from a saturated hexane solution at affording [(BDI)Ca(*o*-C₂B₁₀H₁₁)] (**2**). Yield: 17.4 mg, 45%.

¹H NMR (C₆D₆): δ = 7.14-7.08 (m, ^{Dipp}ArH, 6H), 4.52 (s, HCCN, 1H), 3.80-1.80 (br m, BH, 10H), 3.03 (s, HCcarborane, 1H), 2.75 (hept, ³J_{HH} = 5.9 Hz, HC(CH₃)₂, 4H), 1.46 (s, NCCH₃, 6H), 1.27 (d, ³J_{HH} = 6.9 Hz, HC(CH₃)₂, 12H), 1.12 (d, ³J_{HH} = 6.6 Hz, HC(CH₃)₂, 12H).

¹³C{¹H} NMR (C₆D₆): δ = 166.8 (NCCH₃), 146.7 (*i*-C₆H₃), 141.2 (*o*-C₆H₃), 125.2 (*p*-C₆H₃), 124.3 (*m*-C₆H₃), 88.9 (HCCN), 61.8 (HCcarborane), 28.7 (HC(CH₃)₂), 25.4 (HC(CH₃)₂), 25.15 (NCCH₃).

¹¹B NMR (C₆D₆) δ = -0.56 (d, *J* = 140.1 Hz), -5.43 (d, *J* = 146.6 Hz), -10.37 (d, *J* = 153.1 Hz), -12.56 (d, *J* = 171.2 Hz).

Anal. Calc. for C₃₁H₅₂N₂B₁₀Ca₁: C, 61.96; H, 8.72; N, 4.66. Found: C, 61.76; H, 8.61; N, 4.38

Synthesis of [(BDI)Ca(THF)(*o*-C₂B₁₀H₁₁)] (**3**)

A J. Young's NMR tube was charged with [(BDI)Ca(*o*-C₂B₁₀H₁₁)] (**2**, 17.4 mg, 0.3 mmol) and THF (*ca.* 0.6 cm³). After 2 hours the volatiles were removed under reduced pressure affording a colourless solid which could be recrystallised from a toluene solution at -35 °C to affording [(BDI)Ca(THF)(*o*-C₂B₁₀H₁₁)] (**3**). Yield: 17 mg, 85%.

¹H NMR (C₆D₆): δ = 7.13-7.05 (m, ^{Dipp}ArH, 6H), 4.70 (s, HCCN, 1H), 3.47 (br, OCH₂CH₂, 4H), 3.37-2.33 (br m, BH, 10H), 2.99 (br s, HC(CH₃)₂, 4H), 2.54 (s, HCcarborane, 1H), 1.60 (s, NCCH₃, 6H), 1.22-1.18 (overlapping m, OCH₂CH₂ and HC(CH₃)₂, 16 H), 1.16 (d, ³J_{HH} = 6.83 Hz, HC(CH₃)₂, 12 H).

¹³C{¹H} NMR (C₆D₆): δ = 166.6 (NCCH₃), 144.8 (*i*-C₆H₃), 141.3 (*o*-C₆H₃), 125.4 (*p*-C₆H₃), 124.2 (*m*-C₆H₃), 92.5 (HCCN), 69.3 (OCH₂CH₂), 28.7 (HC(CH₃)₂), 25.3 (HC(CH₃)₂), 24.5 (NCCH₃), 24.3 (OCH₂CH₂).

¹¹B NMR (C₆D₆) δ = -1.01 d (147.1 Hz), -5.86 (d, *J* = 147.1 Hz), -9.7 - -14.30 (m).

General Synthetic Protocol for [(NHC^{iPr})M(*o*-C₂B₁₀H₁₁)] [M = Cu (**4**), Ag (**5**), Au (**6**)]

[(BDI)Mg(*o*-C₂B₁₀H₁₁)] was generated *in-situ* from [(BDI)MgⁿBu] (25 mg, 0.05 mmol) and *ortho*-carborane (7.22 mg, 0.05 mmol) in C₆D₆ (0.6 cm³), as described above (*vide supra*), and subsequently reacted with 1 equivalent of NHC^{iPr}MCl (0.05 mmol). Colourless crystals of [(BDI)MgCl]₂ instantly precipitated from the solution. After filtration the volatiles were removed under reduced pressure and

the resulting crude solid was washed with hexane to afford the respective [(NHC^{iPr})M(*o*-*o*-C₂B₁₀H₁₁)] (M = Cu (**4**), Au (**6**)). Yield Cu: 8.4 mg, 43%; Yield Au: 11.5 mg, 44%.

Although the formation of NHC^{iPr}Ag(*o*-C₂B₁₀H₁₁) was evident allowing its crystallographic characterised, constant precipitation of Ag metal was observed and therefore a sufficiently clean sample for spectroscopic characterisation could not be obtained.

[(NHC^{iPr})Cu(*o*-C₂B₁₀H₁₁)] (**4**)

¹H NMR (C₆D₆): δ = 3.80-2.32 (br m, BH, 10H), 3.50 (hept, ³J_{HH} = 6.9 Hz, HC(CH₃)₂, 2H), 3.18 (s, HCcarborane, 1H), 1.27 (s, CH₃, 6H), 1.20 (d, ³J_{HH} = 6.9 Hz, HC(CH₃)₂, 12H).

¹³C{¹H} NMR (C₆D₆): δ = 169.0 (NHC^{iPr}-C), 123.2 (NHC^{iPr}-ArC), 61.7 (HCcarborane), 49.0 (CH(CH₃)₂), 24.8 (NHC^{iPr}-C(CH₃)₂), 8.3 (NHC^{iPr}-CH₃).

¹¹B NMR (C₆D₆) δ = -1.10 (d, J = 137.3 Hz), -6.57 (d, J = 151.2 Hz), -9.45 (d, J = 151.2 Hz), -11.7 (t, J = 141.9 Hz).

Anal. Calc. for C₁₃H₃₁N₂B₁₀Cu₁: C, 40.34; H, 8.07; N, 7.24. Found: C, 39.73; H, 7.11; N, 7.05.

[(NHC^{iPr})Au(*o*-Carborane)] (**6**)

¹H NMR (C₆D₆): δ = 3.99 (hept, ³J_{HH} = 6.9 Hz, HC(CH₃)₂, 2H), 3.80-2.32 (br m, BH, 10H), 3.37 (s, HCcarborane, 1H), 1.38 (s, CH₃, 6H), 1.32 (d, ³J_{HH} = 6.9 Hz, HC(CH₃)₂, 12H).

¹³C{¹H} NMR (C₆D₆): δ = 175.7 (NHC^{iPr}-C), 123.5 (NHC^{iPr}-ArC), 63.1 (HCcarborane), 50.2 (CH(CH₃)₂), 23.2 (NHC^{iPr}-C(CH₃)₂), 8.6 (NHC^{iPr}-CH₃).

¹¹B NMR (C₆D₆) δ = -1.43 (d, J = 139.2 Hz), -7.28 (d, J = 152.8 Hz), -8.95 (d, J = 161.2 Hz), -11.92 (m).

Anal. Calc. for C₁₃H₃₁N₂B₁₀Cu₁: C, 30.00; H, 6.00; N, 5.38. Found: C, 30.92; H, 5.37; N, 4.96.

NMR Spectra

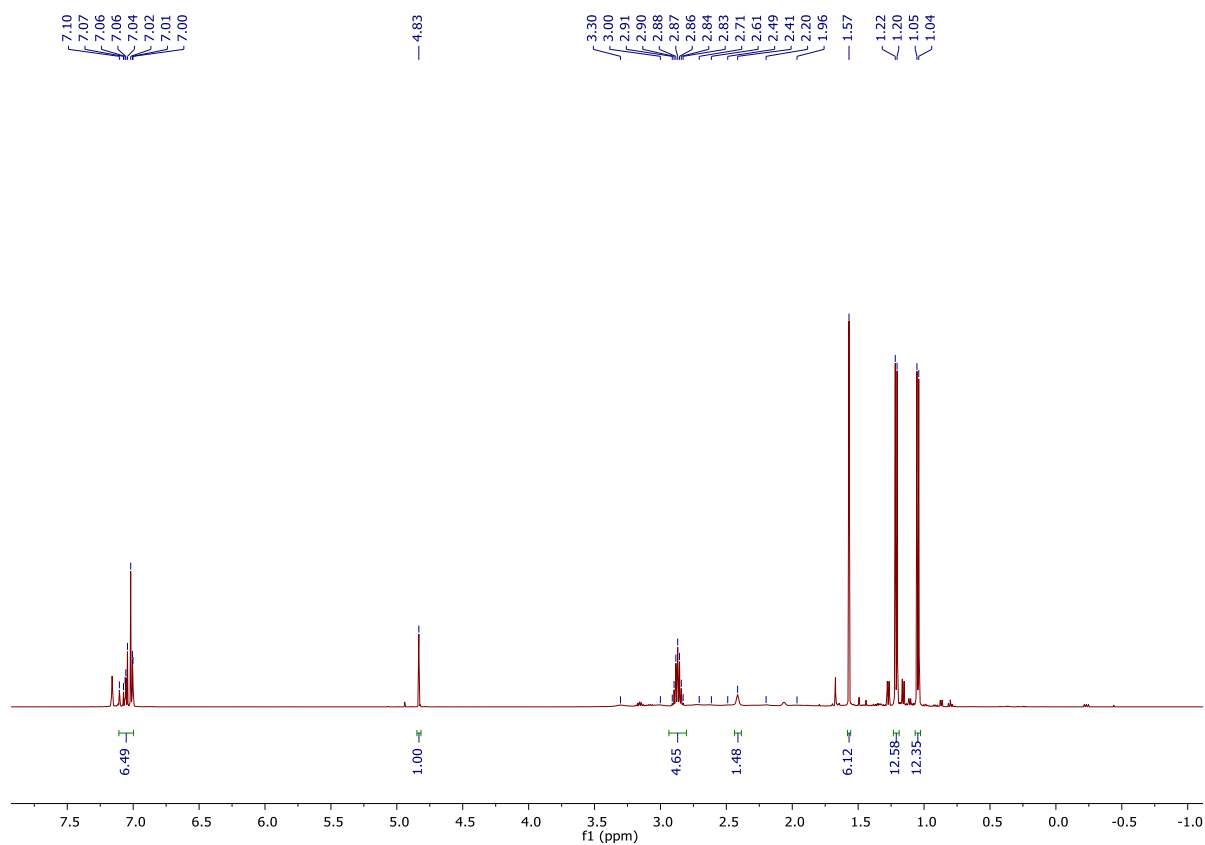


Figure S1. ^1H NMR Spectrum (C_6D_6 , 298 K, 500.06 MHz) for $[(\text{BDI})\text{Mg}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (1).

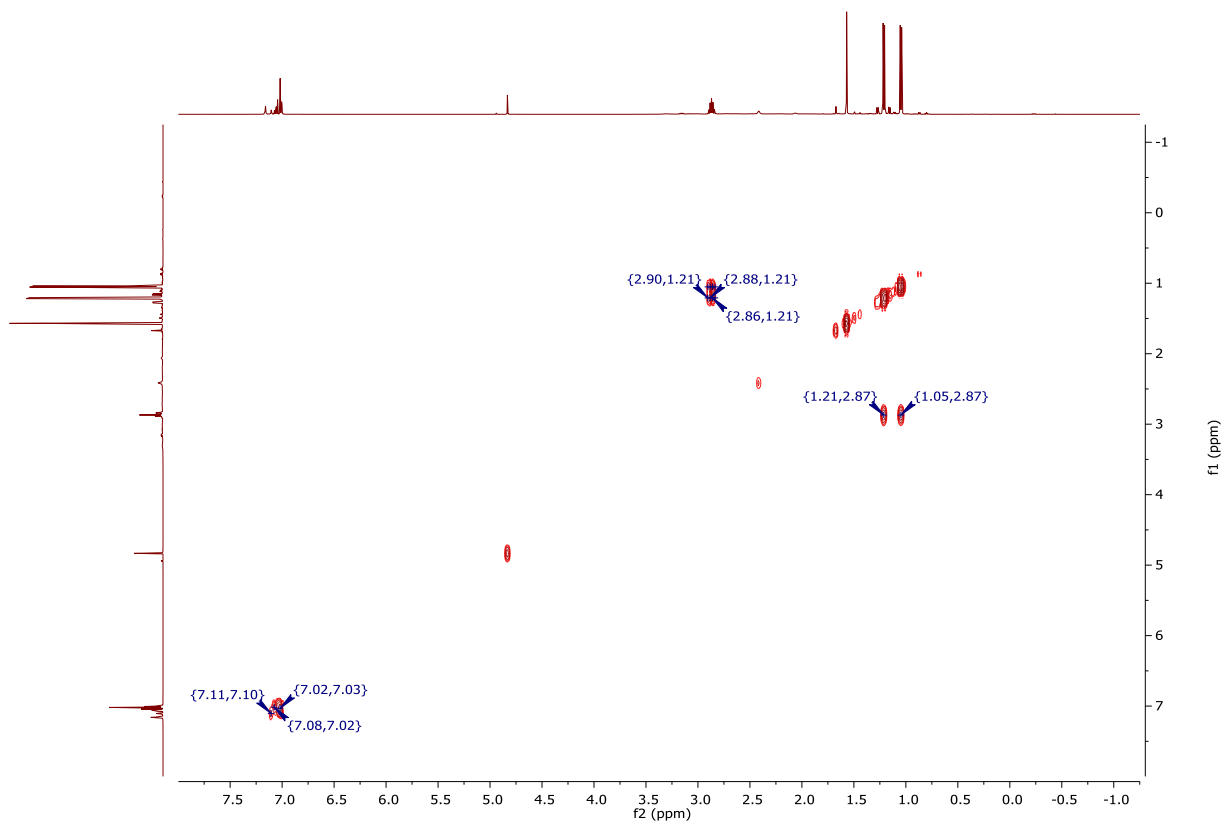


Figure S2. ^1H - ^1H COSY trace (C_6D_6 , 298 K, 500.06, 500.06 MHz) for $[(\text{BDI})\text{Mg}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (1).

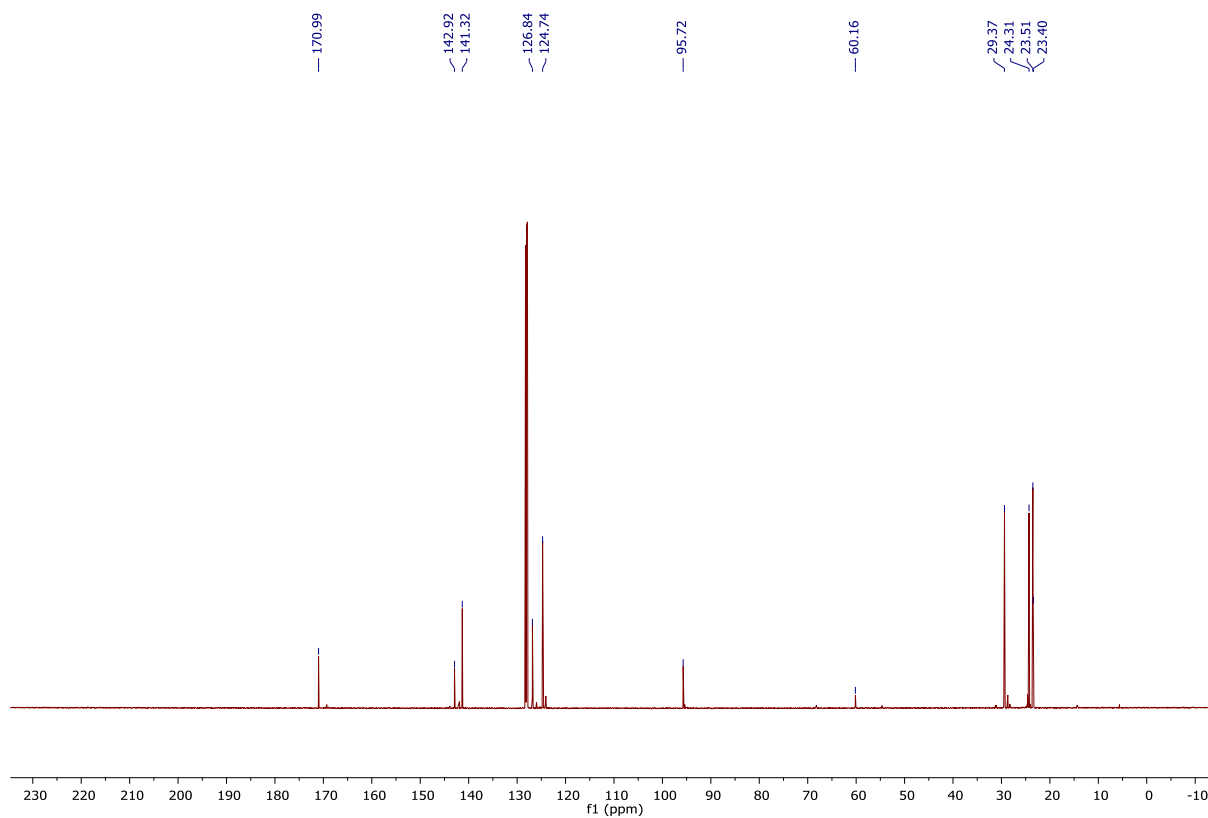


Figure S3. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 125.75 MHz) for $[(\text{BDI})\text{Mg}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (1).

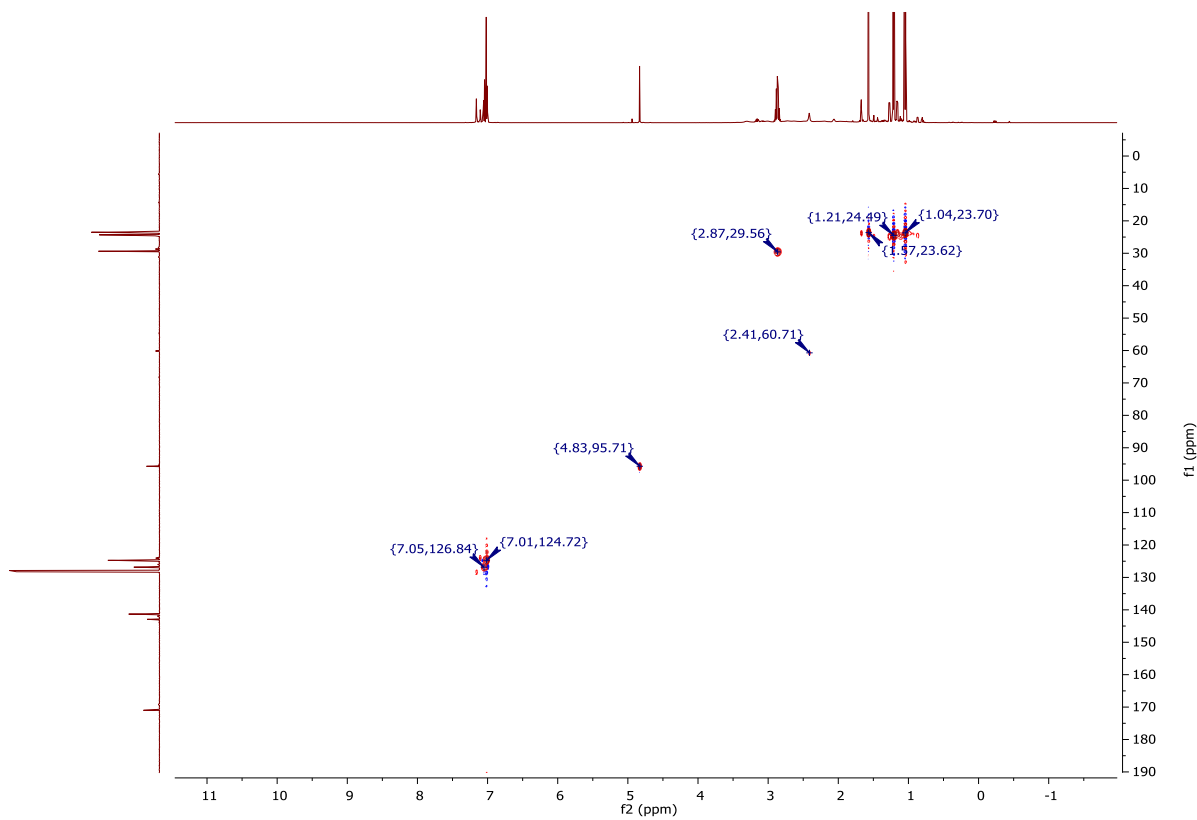
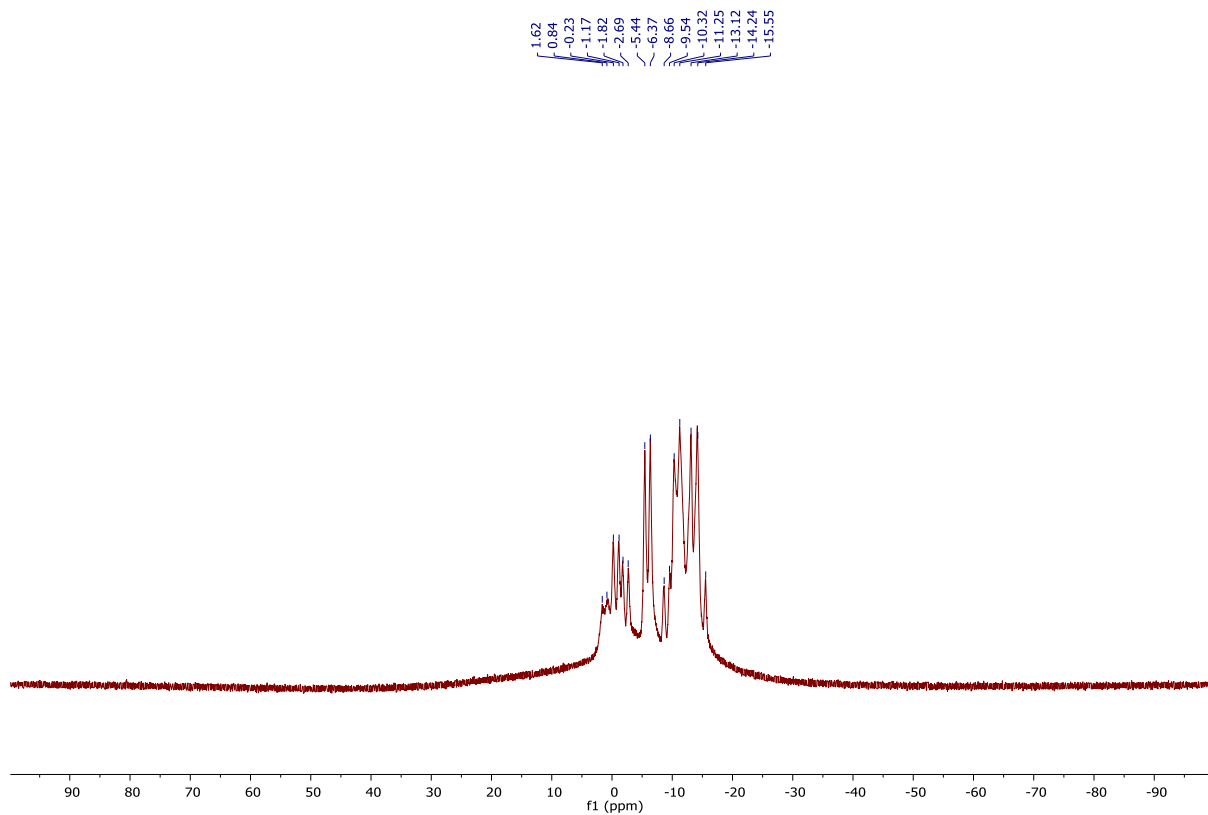
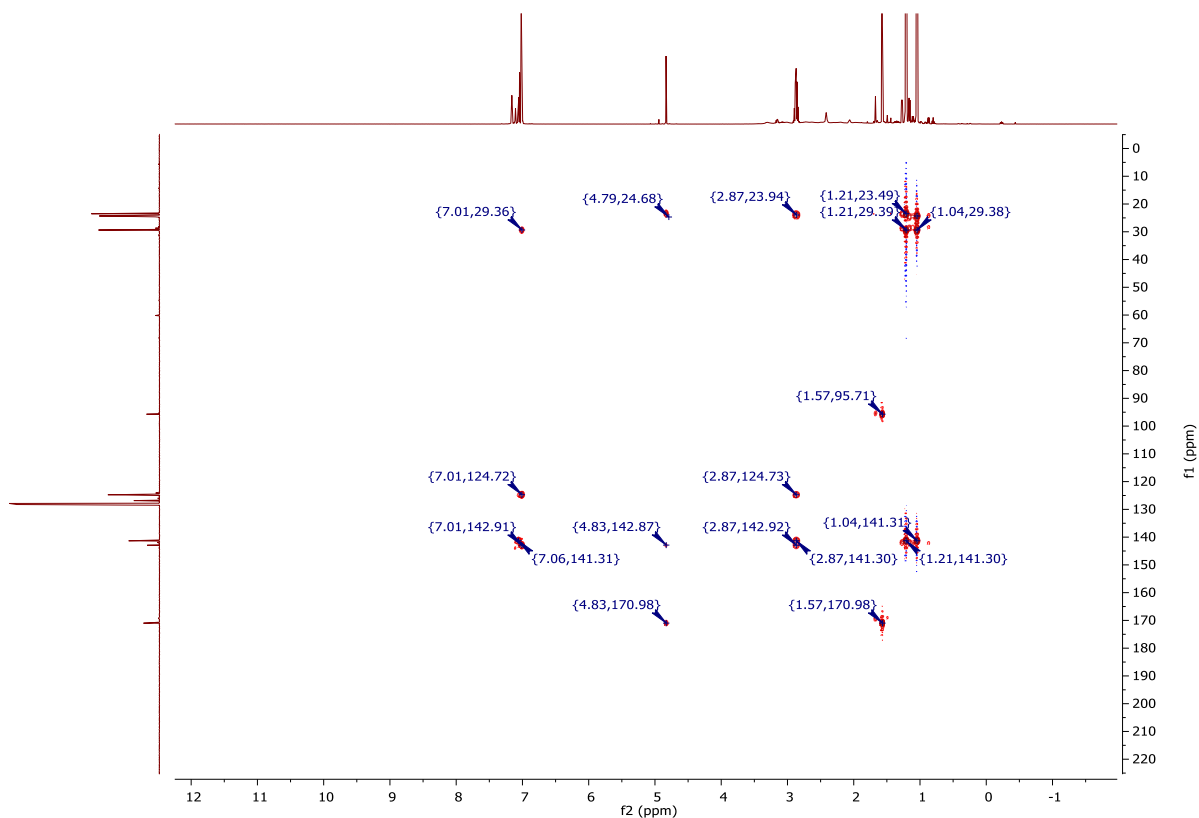


Figure S4. $^1\text{H}-^{13}\text{C}$ HSQC trace (C_6D_6 , 298 K, 500.06, 125.75 MHz) for $[(\text{BDI})\text{Mg}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (1).



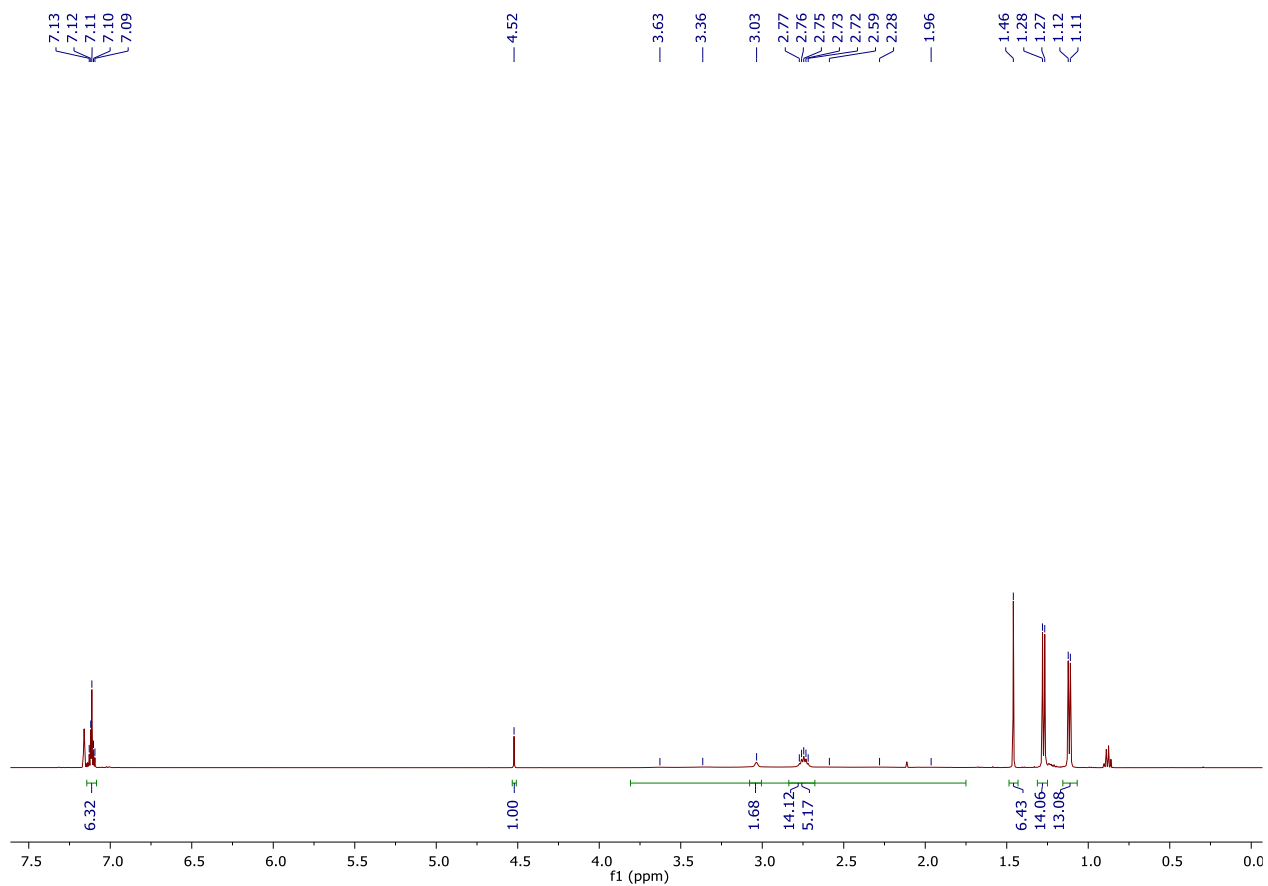


Figure S7. ^1H NMR Spectrum (C_6D_6 , 298 K, 500.06 MHz) for $[(\text{BDI})\text{Ca}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (2).

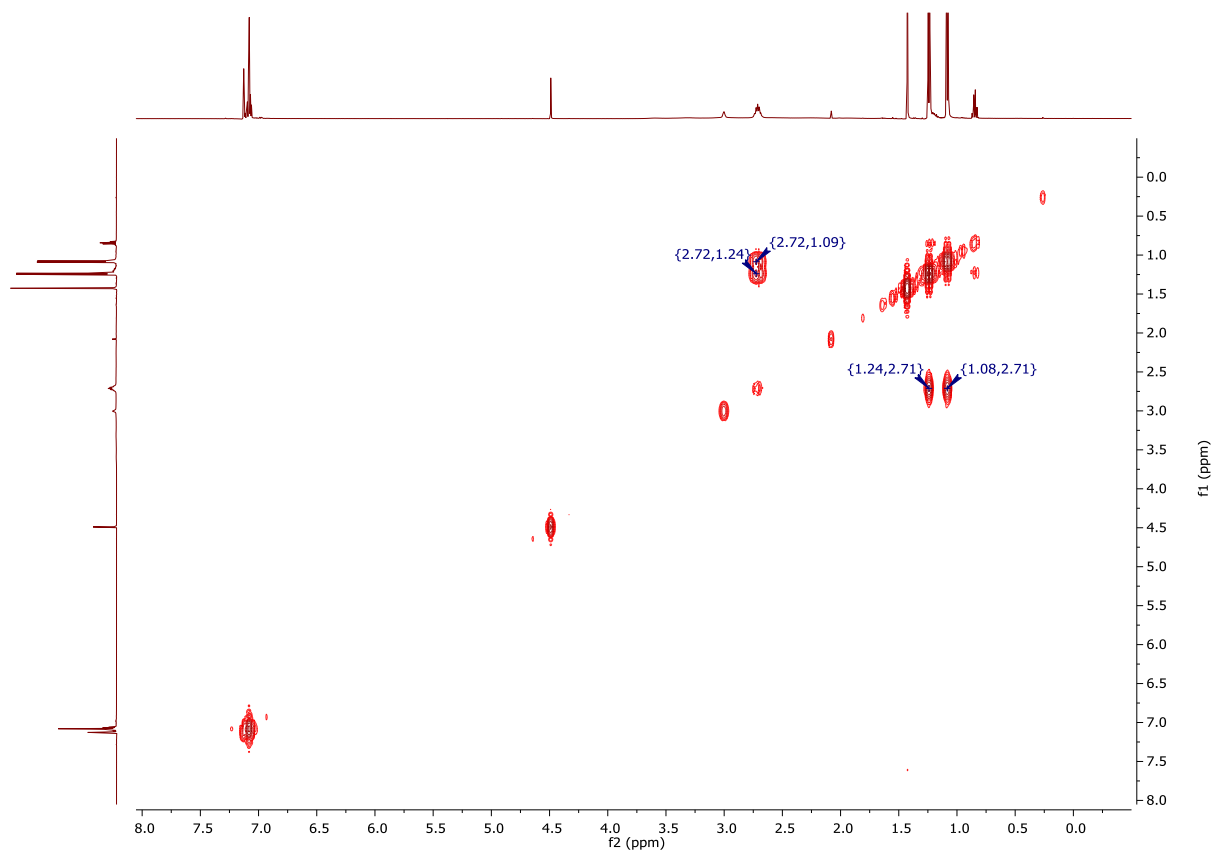


Figure S8. $^1\text{H}-^1\text{H}$ COSY trace (C_6D_6 , 298 K, 500.06, 500.06 MHz) for $[(\text{BDI})\text{Ca}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (2).

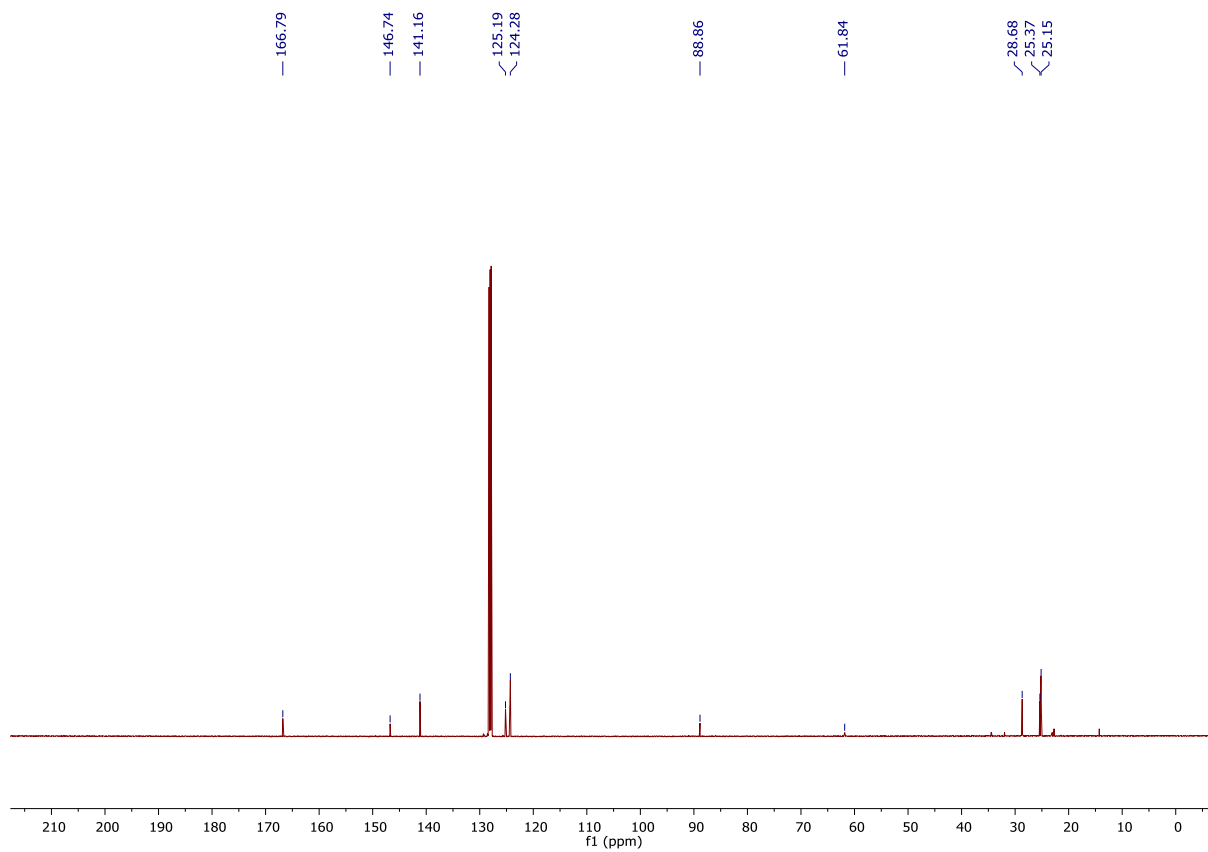


Figure S9. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 125.75 MHz) for $[(\text{BDI})\text{Ca}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (3).

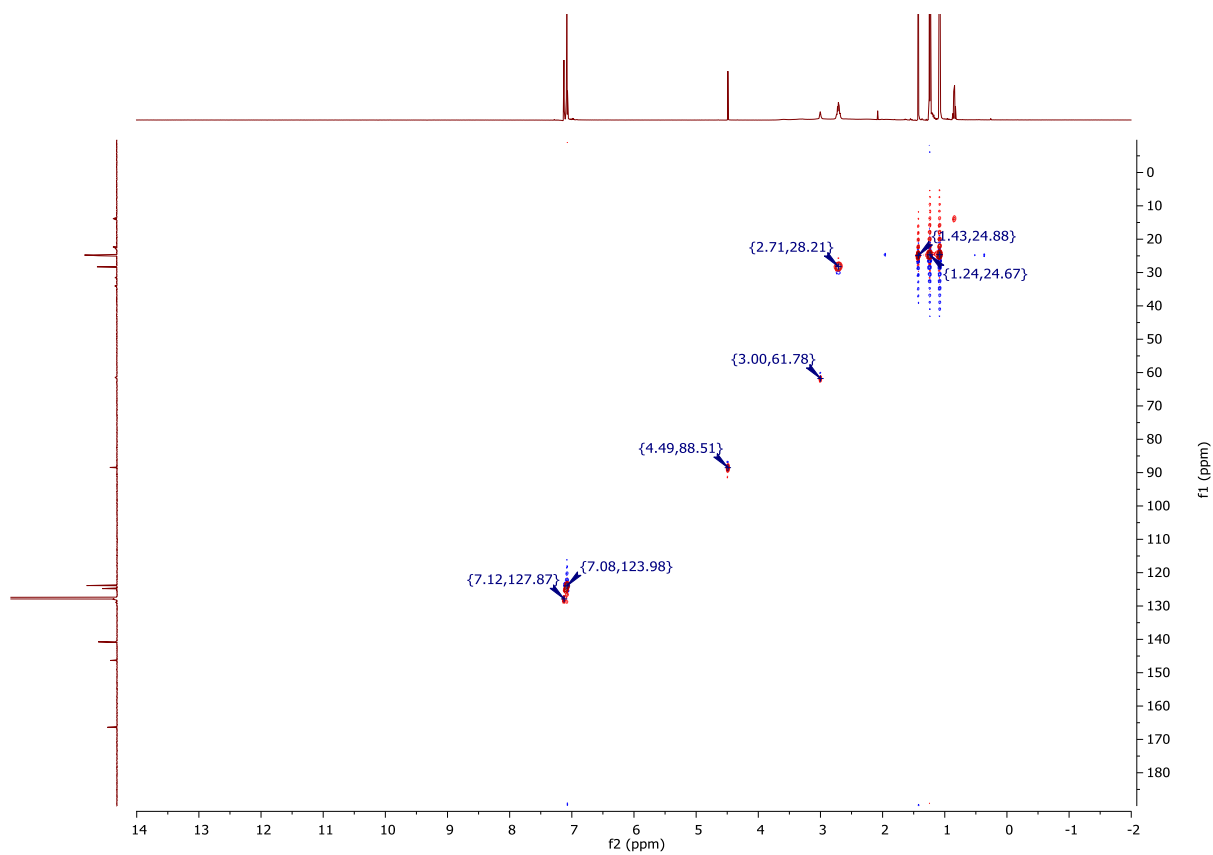


Figure S10. $^1\text{H}-^{13}\text{C}$ HSQC trace (C_6D_6 , 298 K, 500.06, 125.75 MHz) for $[(\text{BDI})\text{Ca}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (2).

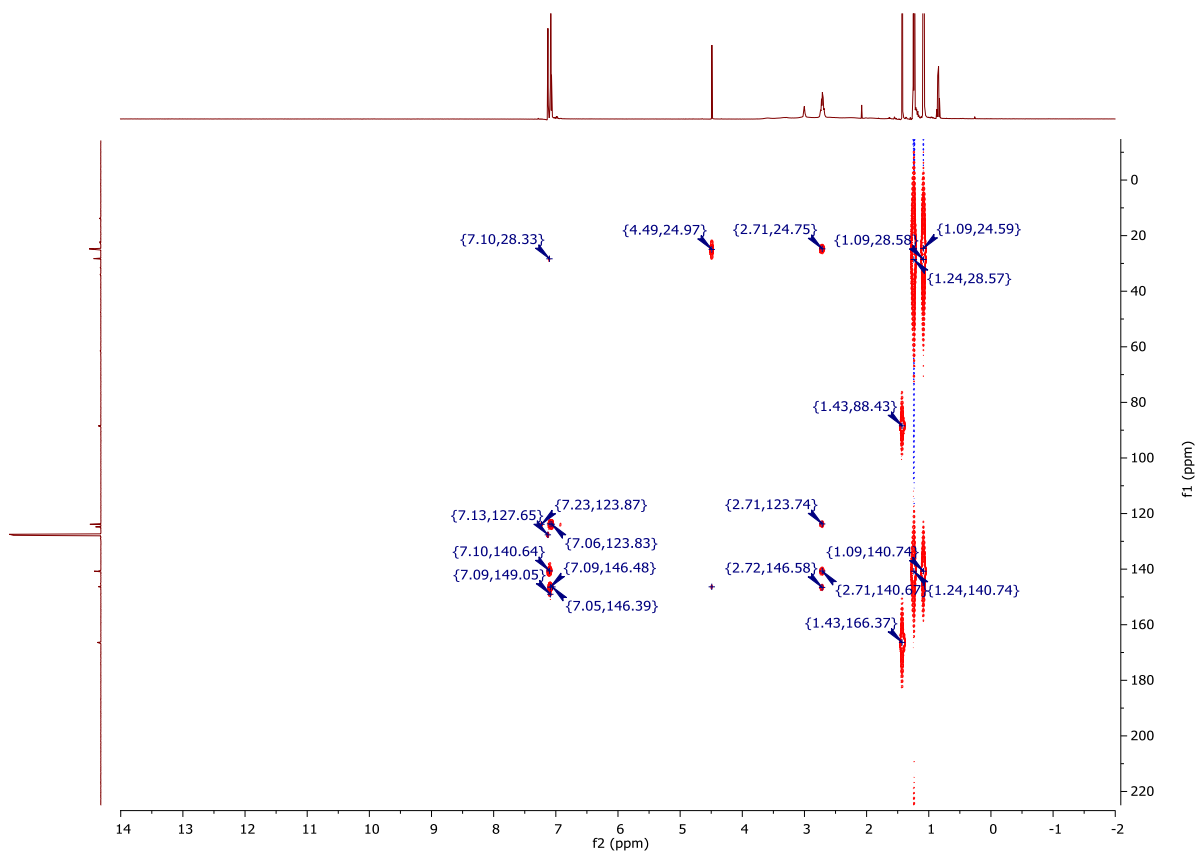


Figure S11. ^1H - ^{13}C HMBC trace (C_6D_6 , 298 K, 500.06, 125.75 MHz) for $[(\text{BDI})\text{Ca}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})](2)$.

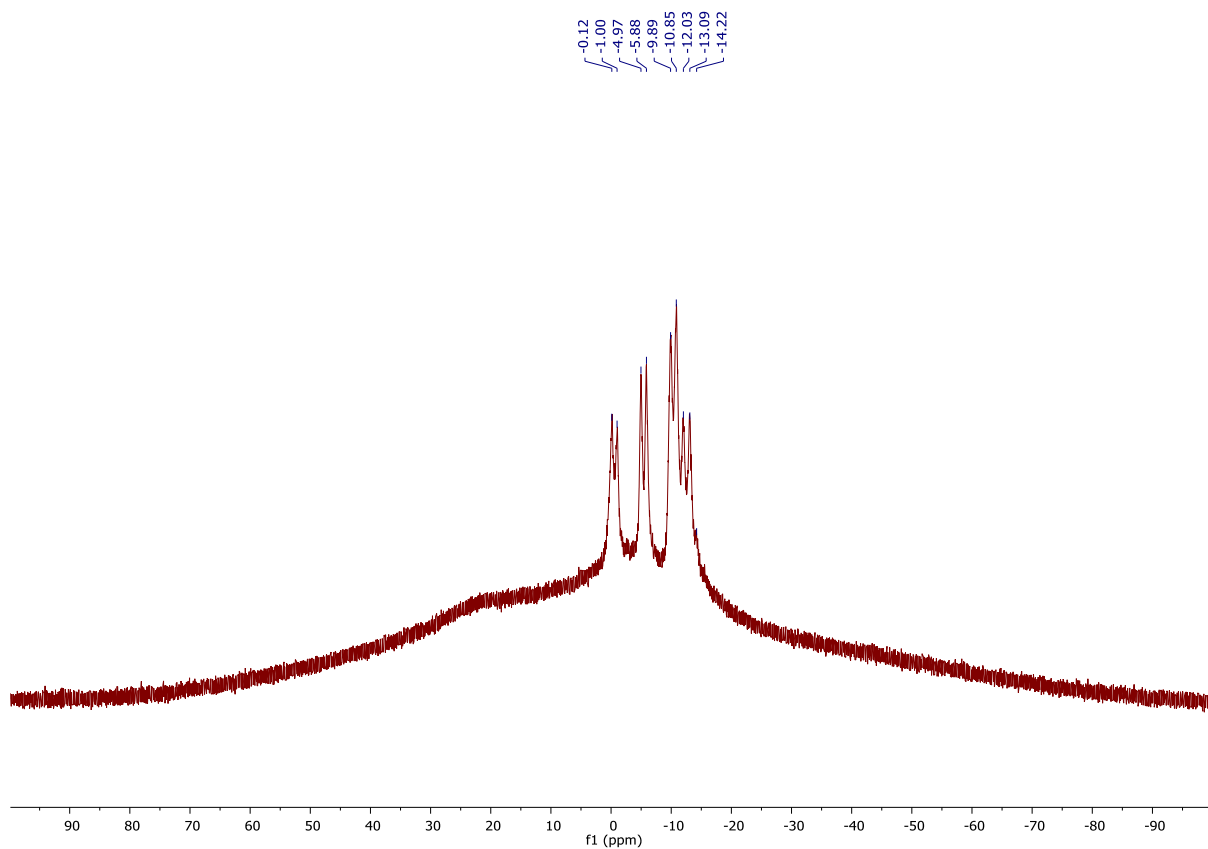


Figure S12. $^{11}\text{B}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 160.44 MHz) for $[(\text{BDI})\text{Ca}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})](2)$.

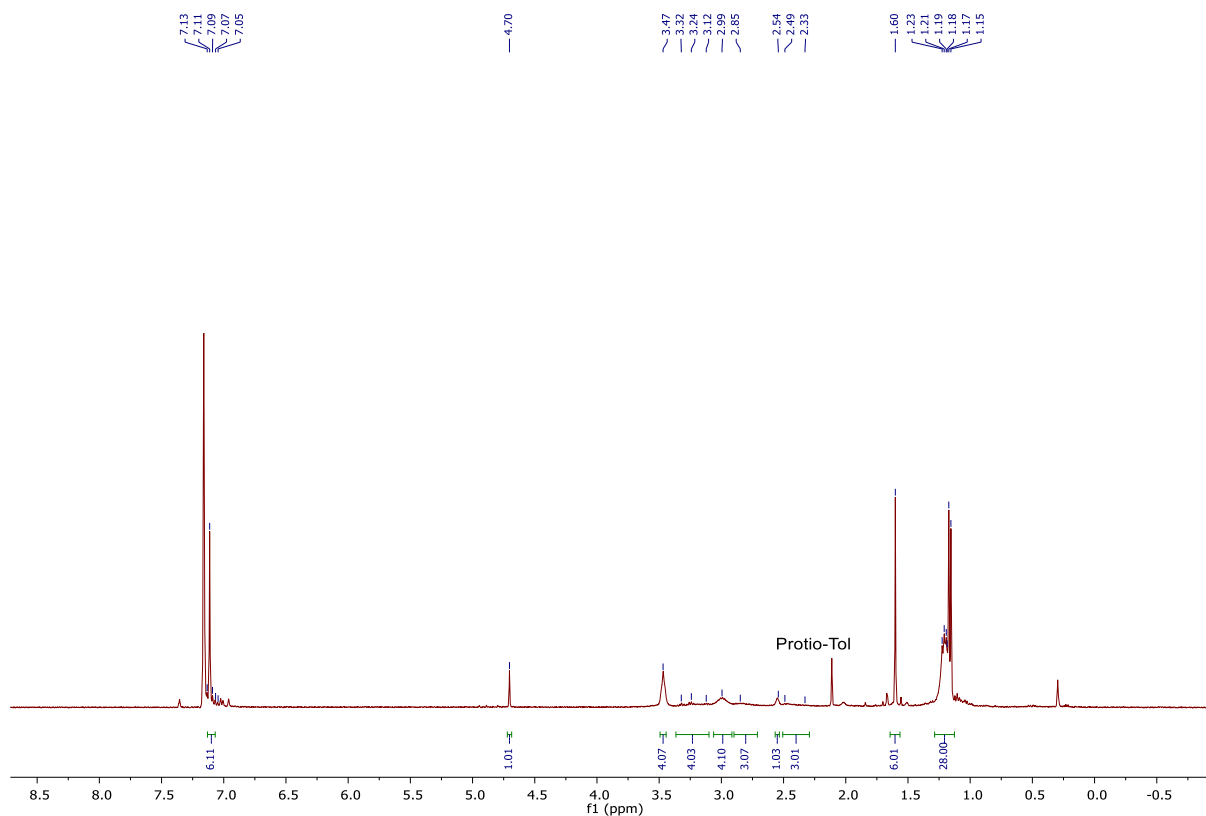


Figure S13. ^1H NMR Spectrum (C_6D_6 , 298 K, 500.06 MHz) for $[(\text{BDI})\text{Ca}(\text{THF})(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (3).

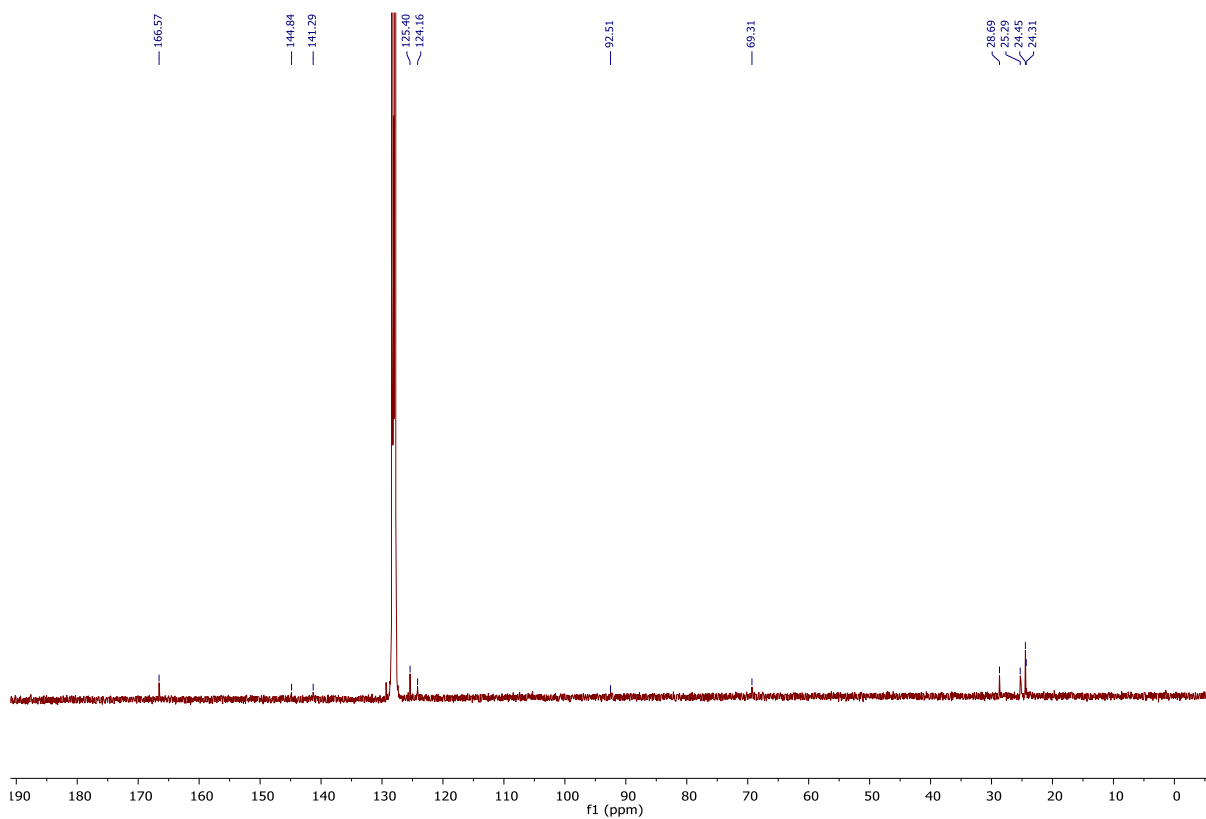


Figure S14. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 125.75 MHz) for $[(\text{BDI})\text{Ca}(\text{THF})(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (3).

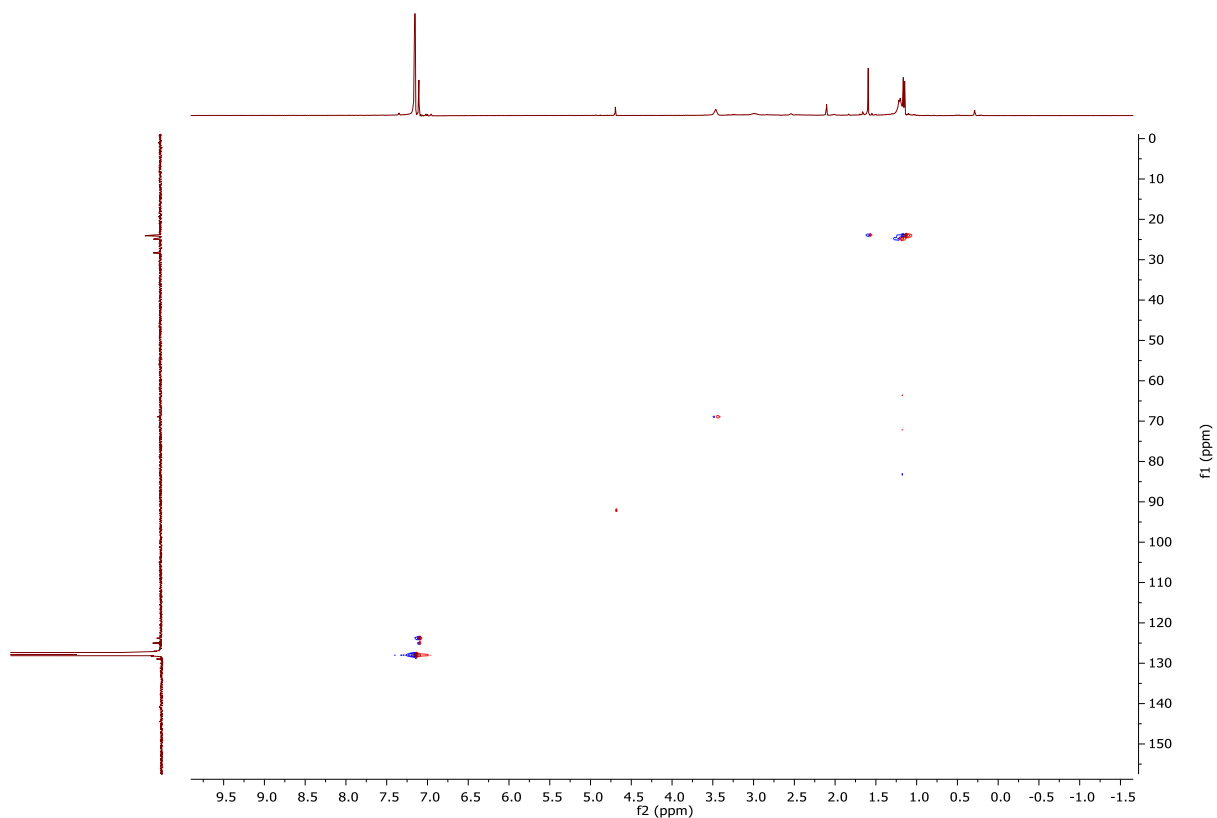


Figure S15. ^1H - ^{13}C HSQC trace (C_6D_6 , 298 K, 500.06, 125.75 MHz) for $[(\text{BDI})\text{Ca}(\text{THF})(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (3).

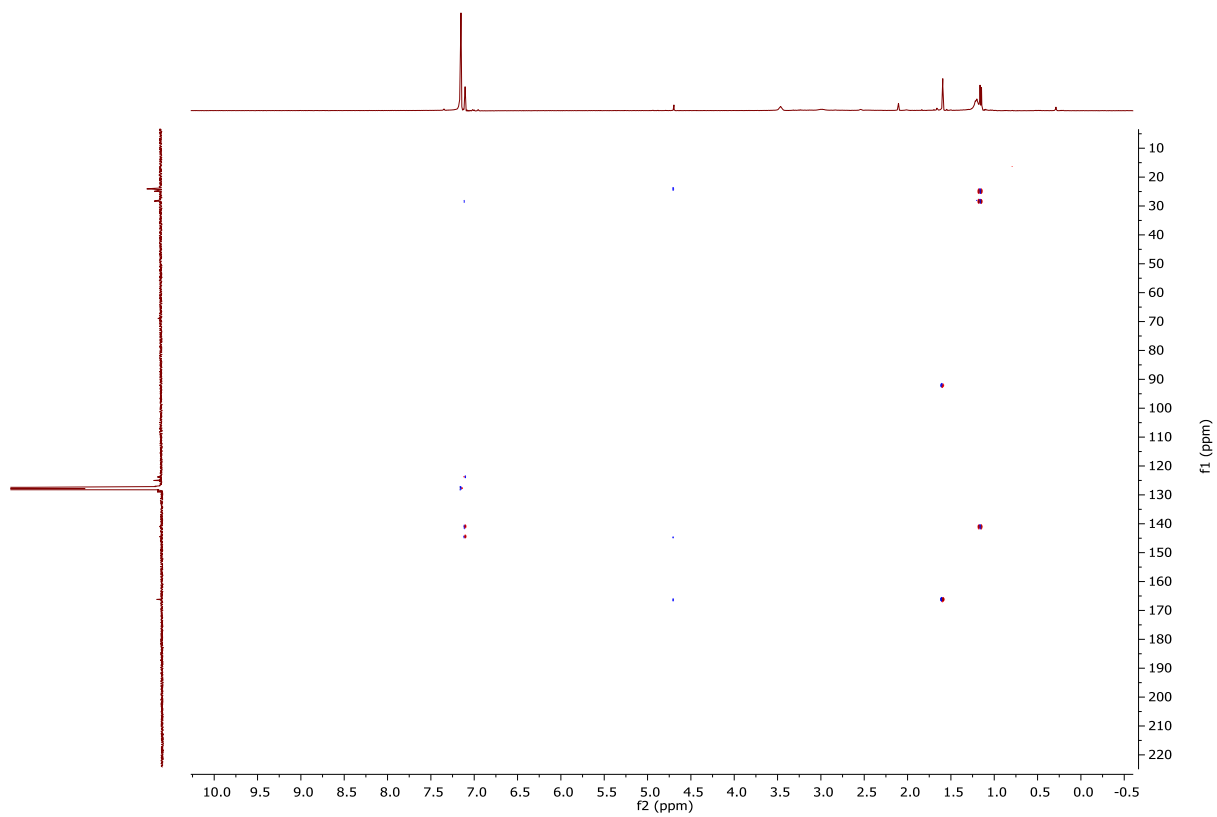


Figure S16. ^1H - ^{13}C HMBC trace (C_6D_6 , 298 K, 500.06, 125.75 MHz) for $[(\text{BDI})\text{Ca}(\text{THF})(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (3).

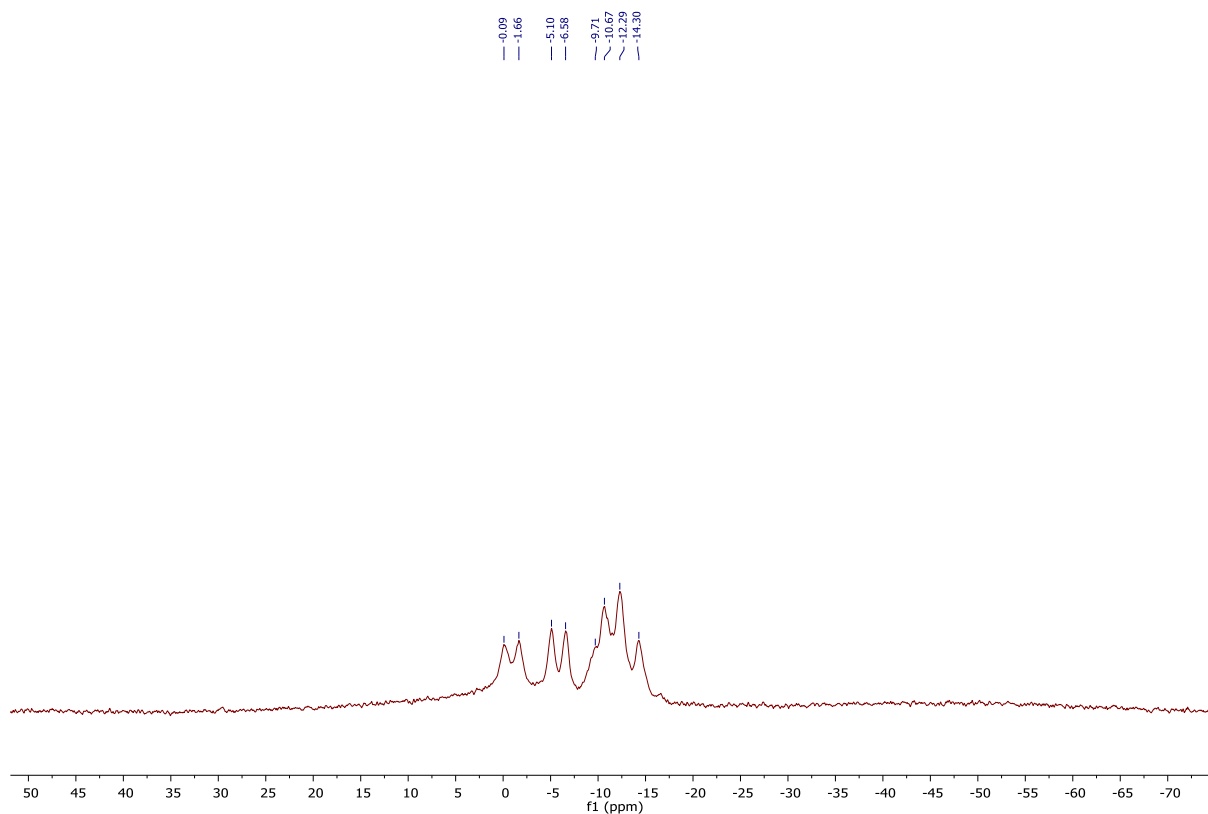


Figure S17. $^{11}\text{B}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 160.44 MHz) for $[(\text{BDI})\text{Ca}(\text{THF})(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (3).

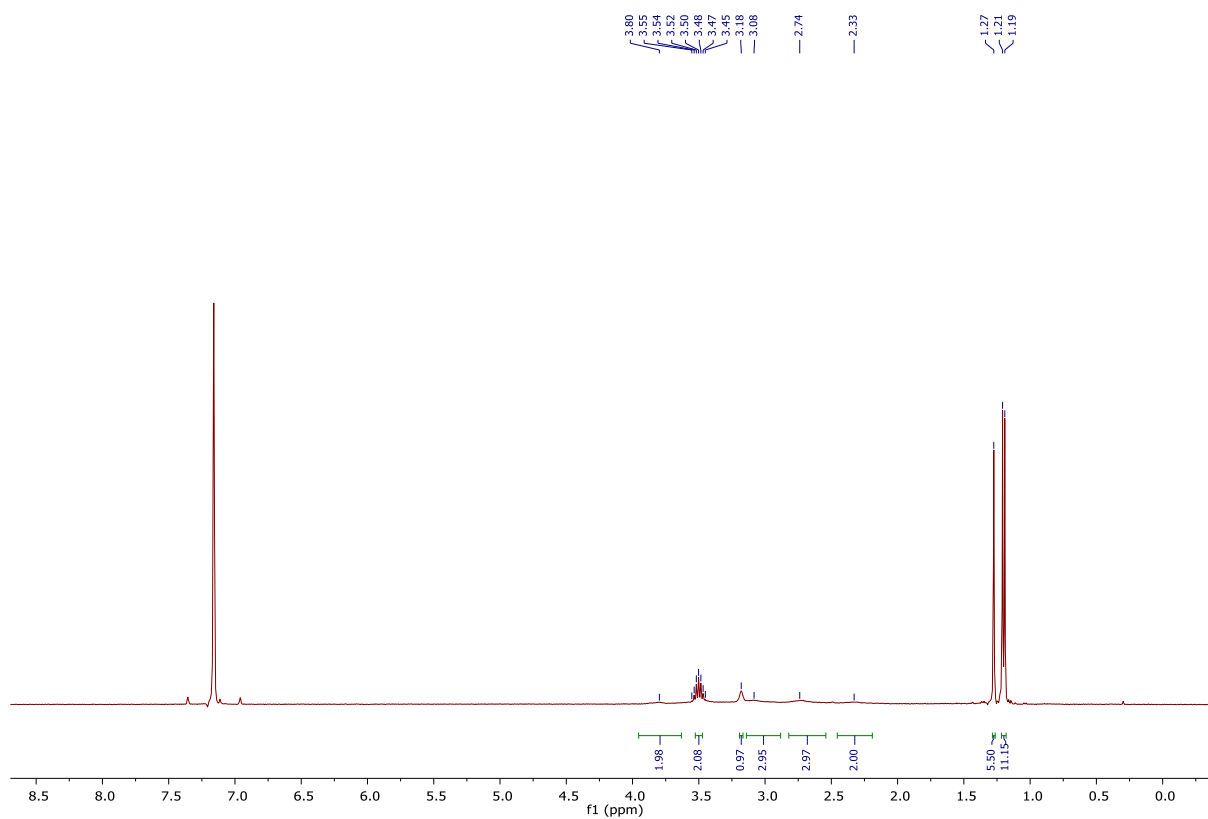


Figure S18. ^1H NMR Spectrum (C_6D_6 , 298 K, 400.13 MHz) for $[(\text{NHC}^{\text{Pr}})\text{Cu}(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (4).

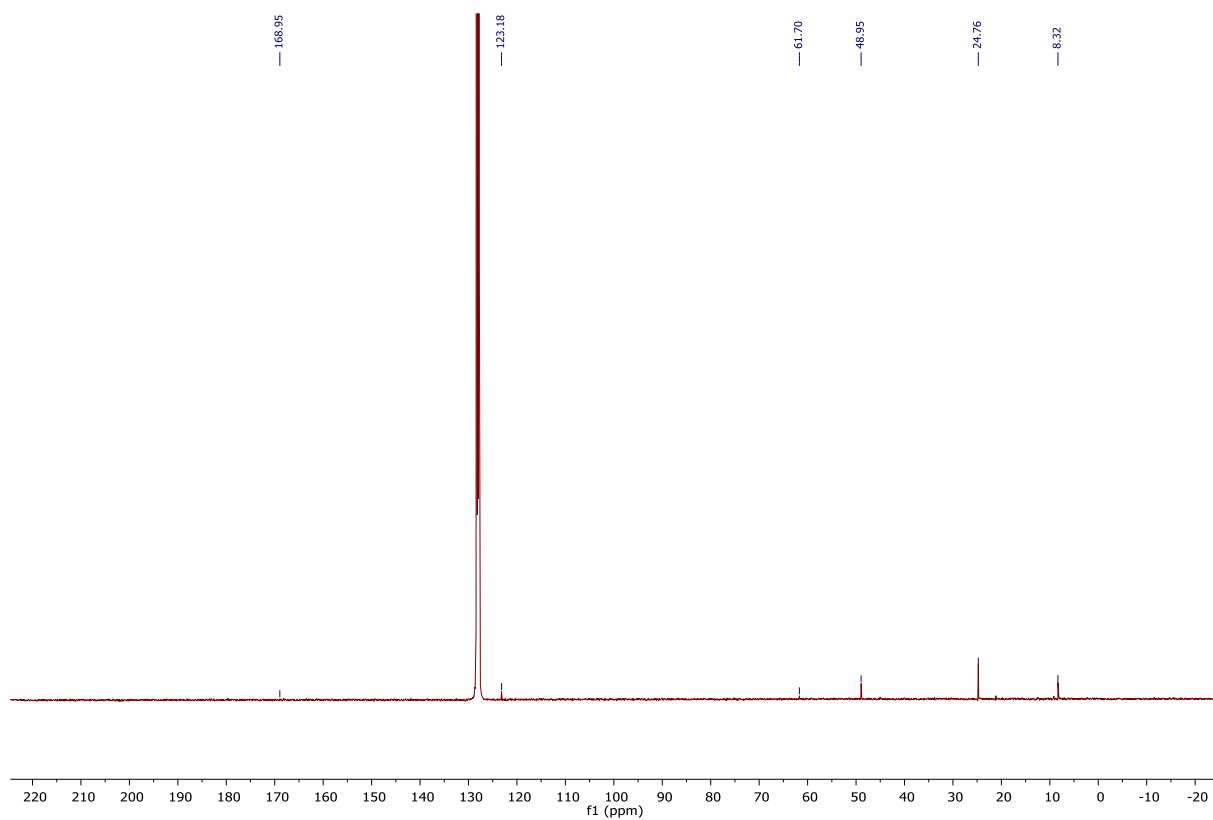


Figure S19. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 100.62 MHz) for $[(\text{NHC}^{\text{iPr}})\text{Cu}(\text{o-C}_2\text{B}_{10}\text{H}_{11})]$ (4).

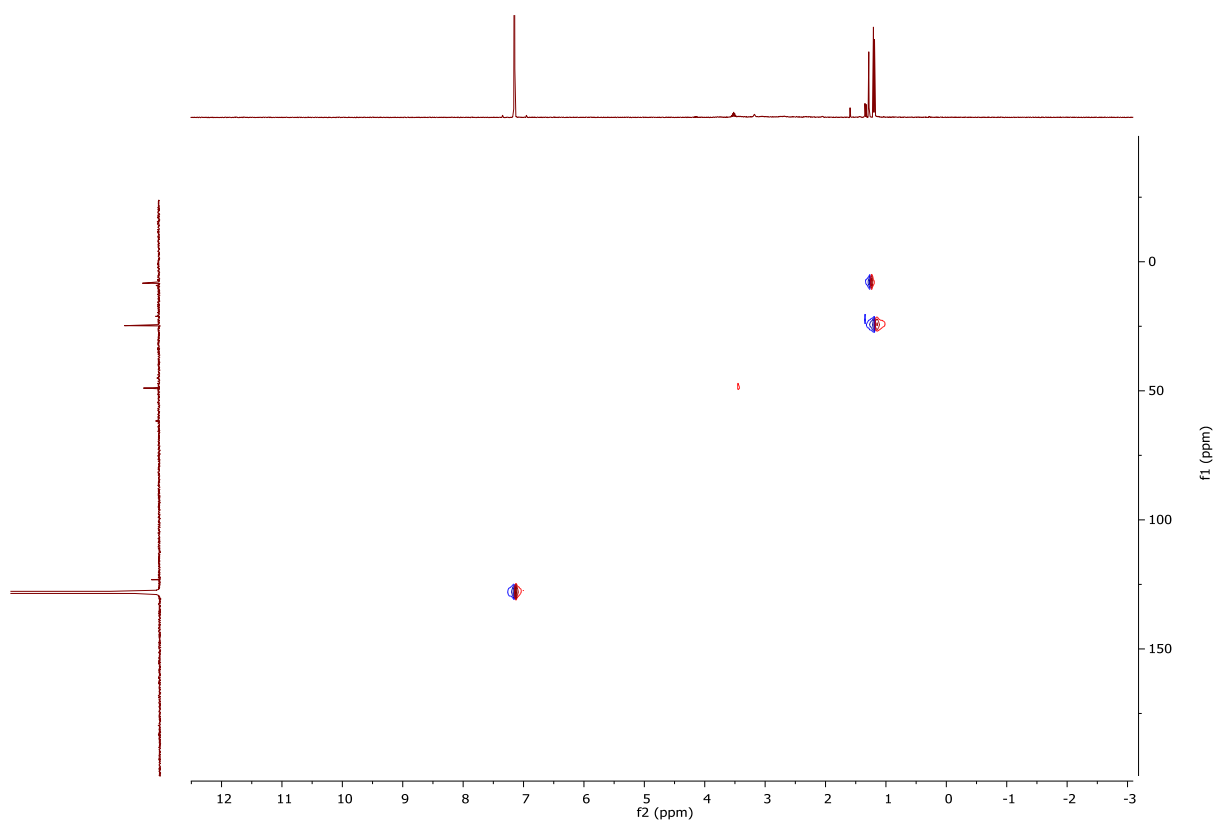


Figure S20. $^1\text{H}-^{13}\text{C}$ HSQC trace (C_6D_6 , 298 K, 400.13, 100.62 MHz) for $[(\text{NHC}^{\text{iPr}})\text{Cu}(\text{o-C}_2\text{B}_{10}\text{H}_{11})]$ (4).

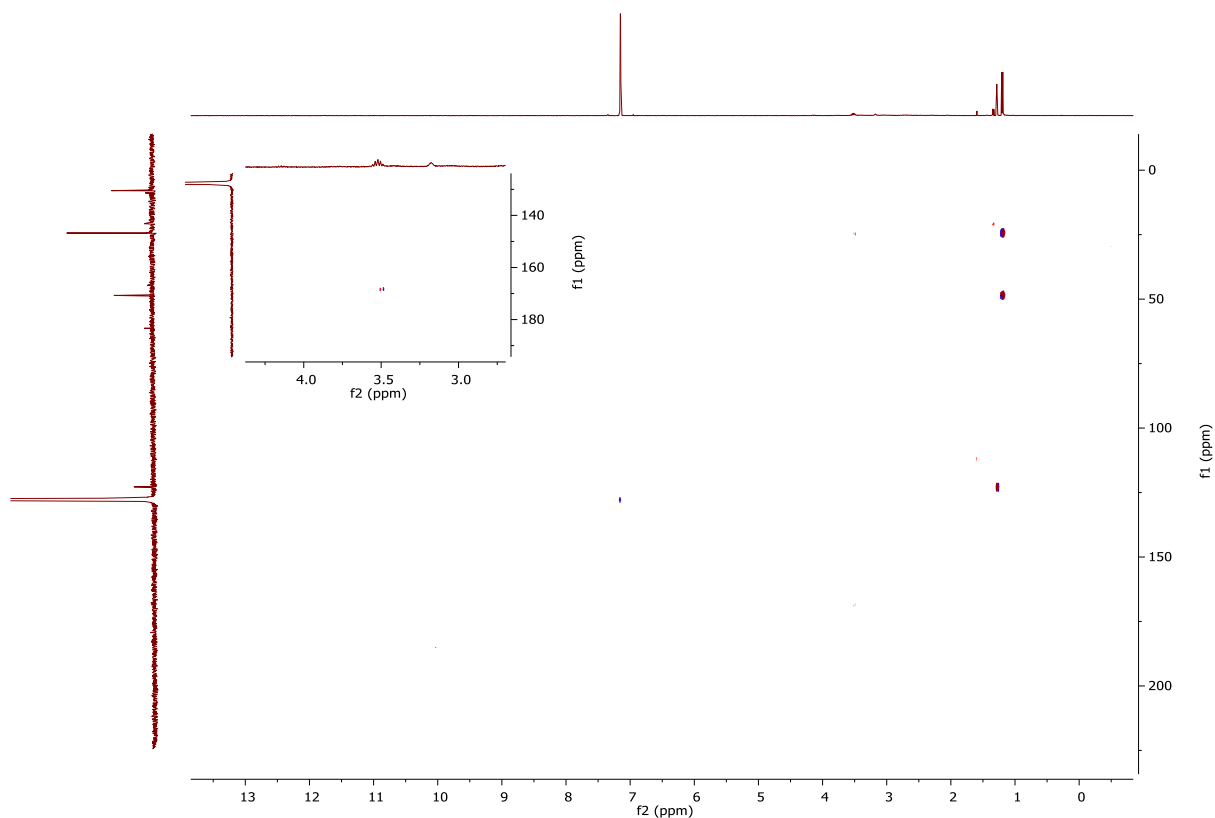


Figure S21. ^1H - ^{13}C HMBC trace (C_6D_6 , 298 K, 400.13, 100.62 MHz) for $[(\text{NHC}^{\text{iPr}})\text{Cu}(\text{o-C}_2\text{B}_{10}\text{H}_{11})]$ (**4**).

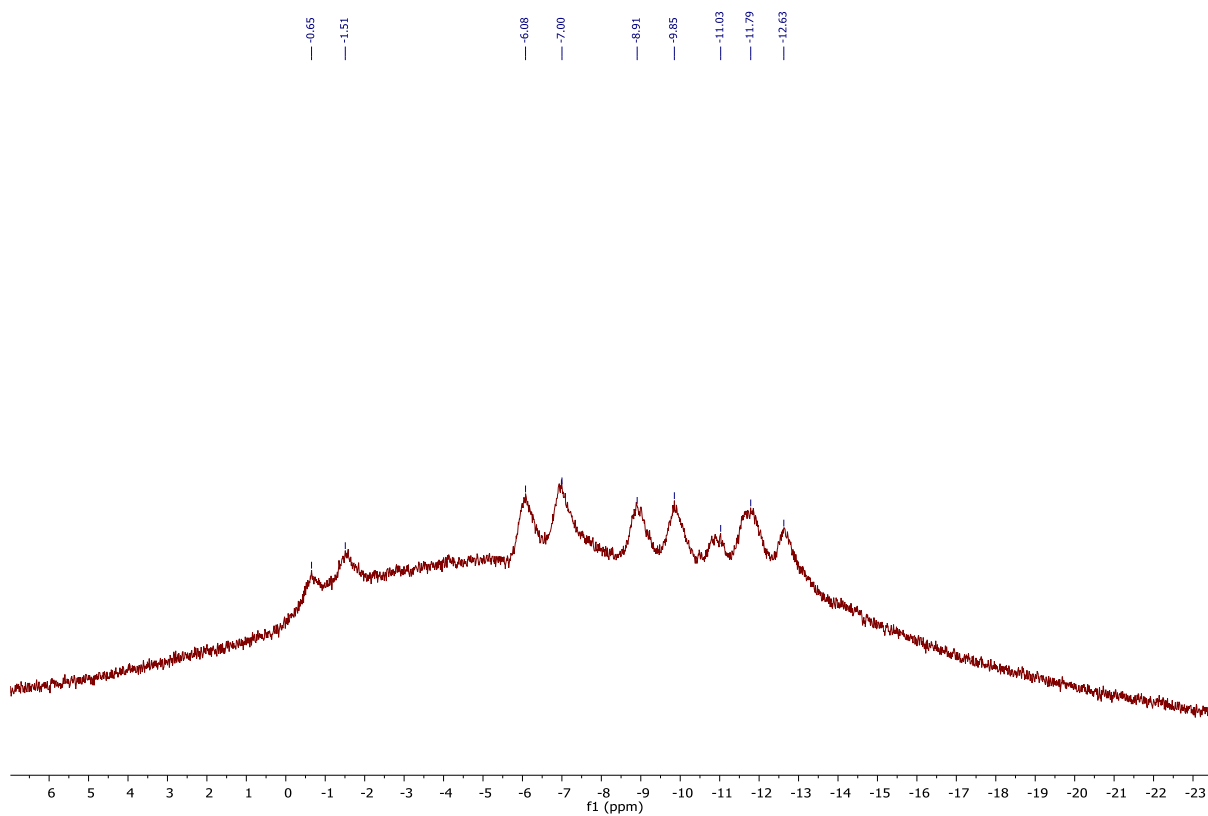


Figure S22. $^{11}\text{B}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 160.44 MHz) for $[(\text{NHC}^{\text{iPr}})\text{Cu}(\text{o-C}_2\text{B}_{10}\text{H}_{11})]$ (**4**).

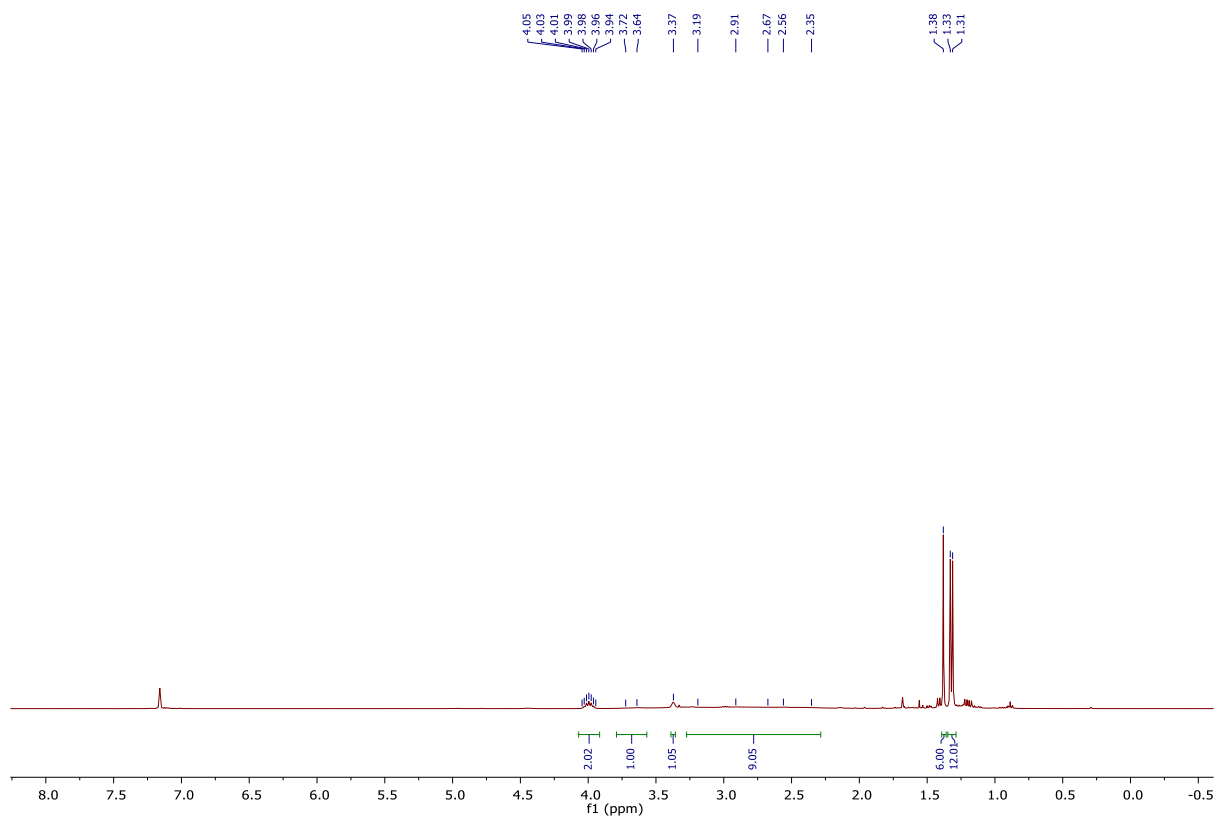


Figure S23. ^1H NMR Spectrum (C_6D_6 , 298 K, 400.13 MHz) for $[(\text{NHC}^{\text{iPr}})\text{Au}(\text{o-C}_2\text{B}_{10}\text{H}_{11})]$ (5).

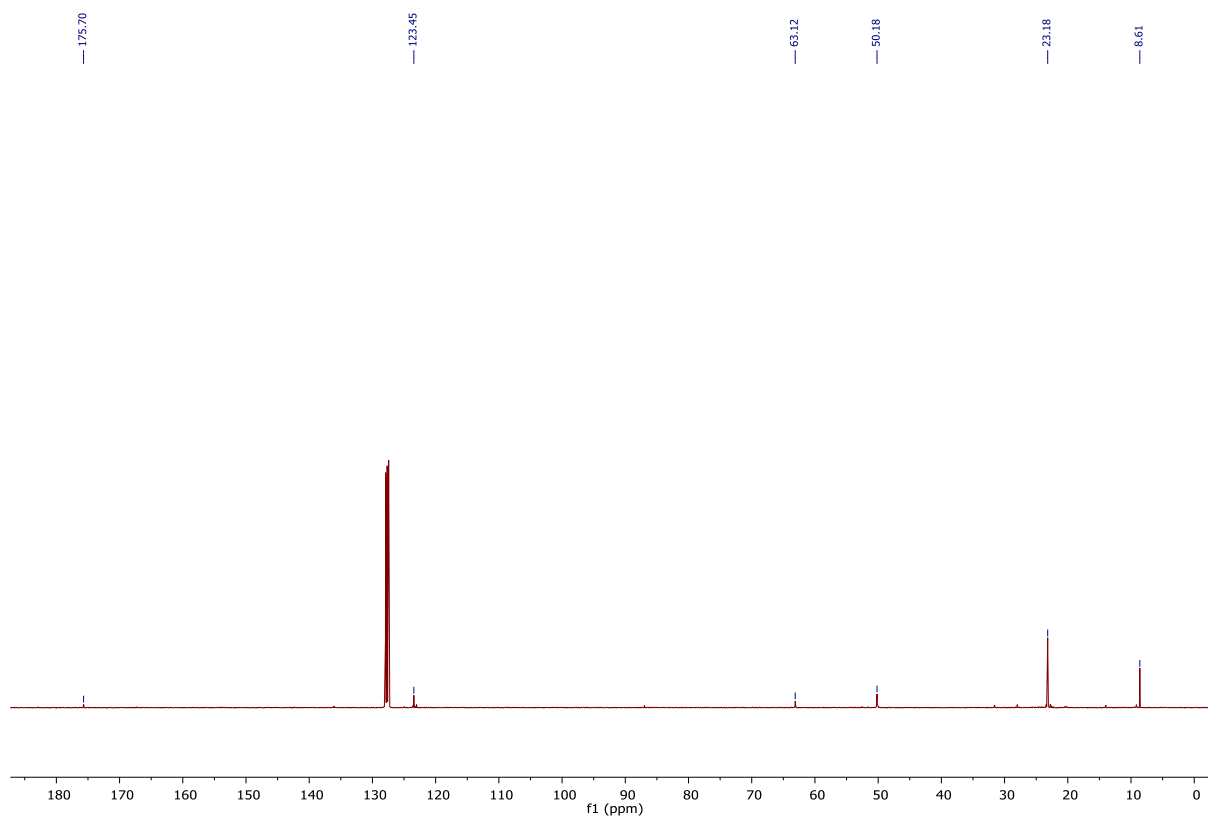


Figure S24. $^{13}\text{C}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 100.62 MHz) for $[(\text{NHC}^{\text{iPr}})\text{Au}(\text{o-C}_2\text{B}_{10}\text{H}_{11})]$ (5).

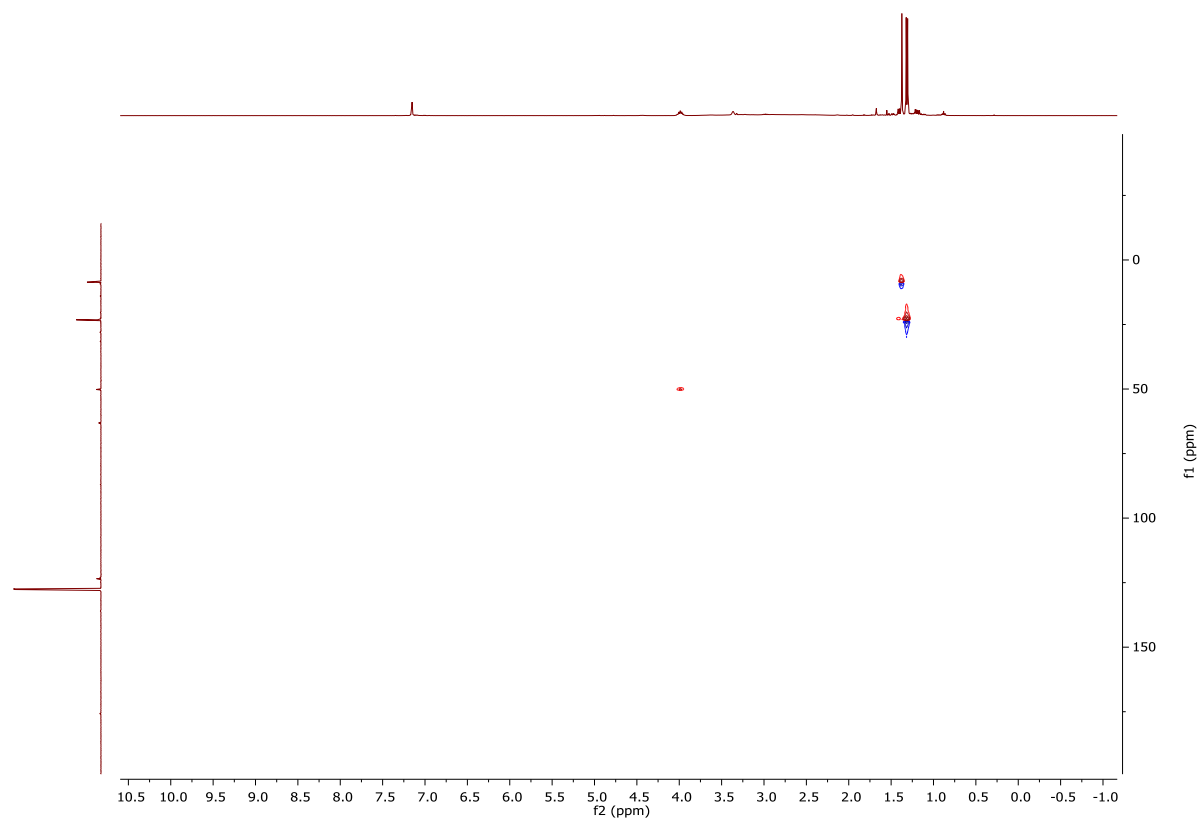


Figure S25. ^1H - ^{13}C HSQC trace (C_6D_6 , 298 K, 400.13, 100.62 MHz) for $[(\text{NHC}^{\text{tPr}})\text{Au}(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (**5**).

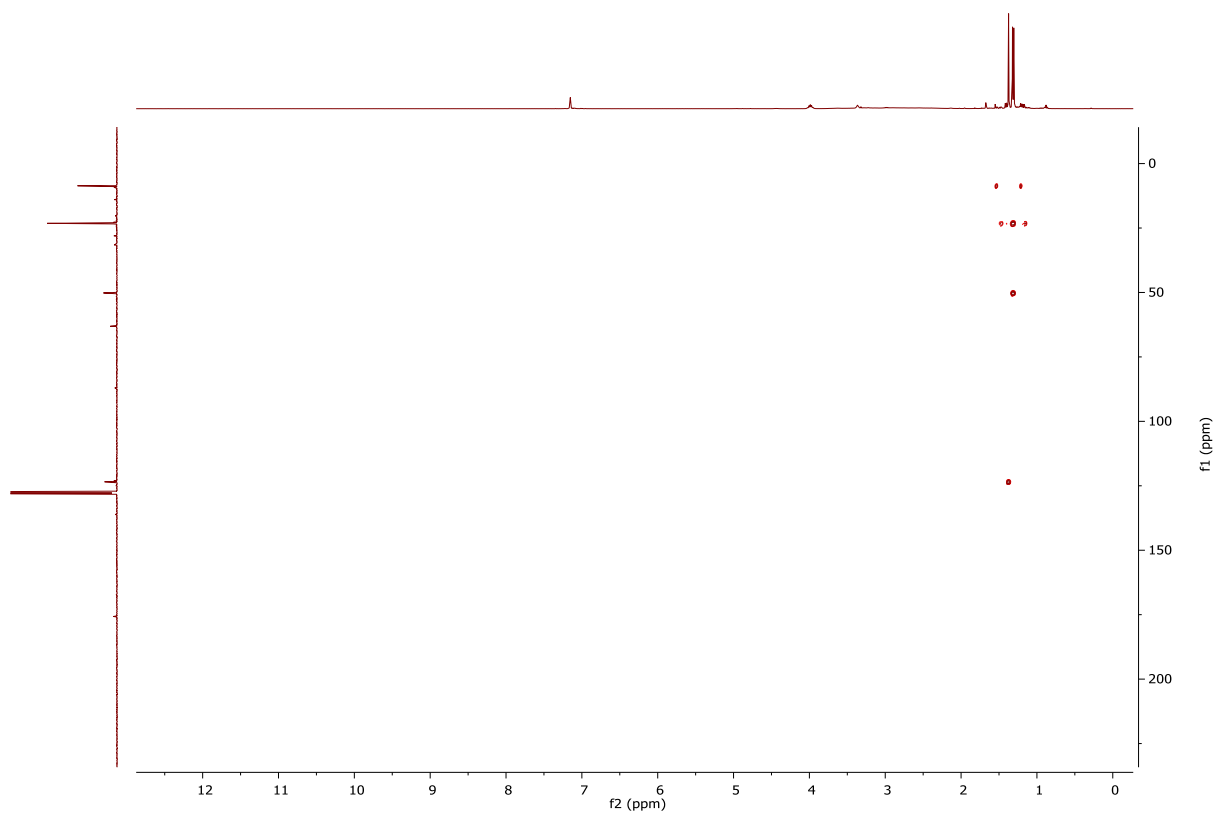


Figure S26. ^1H - ^{13}C HMBC trace (C_6D_6 , 298 K, 400.13, 100.62 MHz) for $[(\text{NHC}^{\text{tPr}})\text{Au}(o\text{-C}_2\text{B}_{10}\text{H}_{11})]$ (**5**).

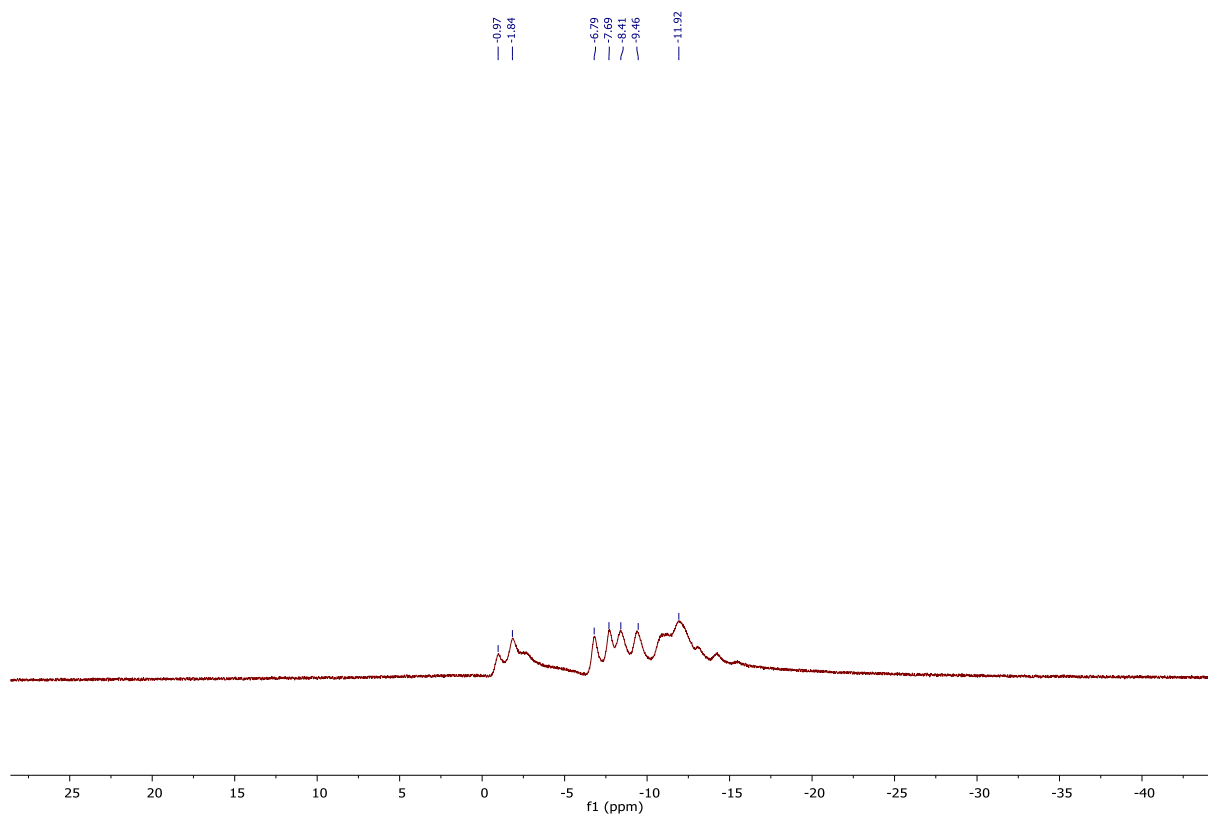


Figure S27. $^{11}\text{B}\{^1\text{H}\}$ NMR Spectrum (C_6D_6 , 298 K, 160.44 MHz) for $[(\text{NHC}^i\text{Pr})\text{Au}(\text{o}-\text{C}_2\text{B}_{10}\text{H}_{11})]$ (5).

Crystallographic Details

Single Crystal X-ray diffraction data were collected on a SuperNova, EosS2 diffractometer using CuK α ($\lambda = 1.54184 \text{ \AA}$) radiation, except for **3** which were collected on a Nonius Kappa CCD diffractometer using MoK α ($\lambda = 0.71073 \text{ \AA}$) radiation. In each case, the crystals were maintained at 150 K during data collection. Using Olex2,⁵ the structures were solved with the olex2.solve⁶ structure solution program or ShelXT and refined with the ShelXL⁷ refinement package using Least-Squares minimisation.

The hydrogen atoms pertaining to the methyl group closest to the magnesium centre (C15) were located in the structure of **1**. The ensuing refinement included C-H and H...H distance restraints.

The asymmetric unit in **2** equates to a monomer containing one calcium centre. In the gross structure, this gives rise to 1-D polymers which propagate along *c*, by virtue of the associated 2₁ screw parallel to this axis. The hydrogens attached to B6, B7 and B10 were located and refined at a distance of 1.12 \AA from the relevant parent atoms. Data completeness for this structure was hampered by a small number of poor frame-sets late in the data collection which were omitted to avoid introducing errors into the dataset as a whole. The sample was very small in size, and the R_{int} value reflects this to a degree. While the R_1 and wR_2 values are compellingly unremarkable, given our observations with the raw data, we present this model as a solid-state characterisation and definitive spatial arrangement of the atoms, rather than presenting any over-interpretation of the associated metric data.

50:50 disorder was modelled for C32-C35, in the structure of **3**, with the inclusion of appropriate distance and ADP restraints (to assist convergence).

The asymmetric unit in **5** equates to half of a molecule. The remainder is generated by a crystallographic mirror on which Ag1, B3 and B6 plus all carbons barring C5 and C9 are located. The hydrogens attached to C6 and C7 were located and refined at a distance of 0.98 \AA from the parent atoms. Two of these hydrogens (H6a and H7a) are also coincident with the mirror plane, as are the hydrogen atoms attached to C4, C8, C13, B3 and B6. The C13 bound hydrogen was also located and refined as per those attached to C6 and C7. The larger than expected difference map maxima lie at chemically insignificant distances from the silver centre. These artefacts reflect the presence of a small passenger on the main sample that gives rise to approximately 6% of the diffraction. Efforts to address this during integration served to degrade the data and, hence, a twin integration was abandoned.

Table S1: Crystal Data and Structural Refinement for the Alkaline Earth Compounds **1** – **3**.

	1	2	3
Empirical formula	C ₃₁ H ₅₂ B ₁₀ MgN ₂	C ₃₁ H ₅₂ B ₁₀ CaN ₂	C ₃₅ H ₆₀ B ₁₀ CaN ₂ O
Formula weight	585.15	600.92	673.03
Crystal system	triclinic	orthorhombic	Monoclinic
Space group	<i>P</i> -1	<i>Pca</i> 2 ₁	<i>P</i> 2 ₁ / <i>a</i>
<i>a</i> / Å	10.4591(2)	21.5052(8)	17.2140(2)
<i>b</i> / Å	10.8125(3)	11.9353(3)	13.0880(2)
<i>c</i> / Å	15.8411(5)	13.8858(5)	18.4650(3)
α / °	81.172(2)	90	90
β / °	89.603(2)	90	103.181(1)
γ / °	88.360(2)	90	90
<i>U</i> / Å ³	1769.50(8)	3564.1(2)	4050.51(10)
<i>Z</i>	2	4	4
ρ_{calc} / g cm ⁻³	1.098	1.120	1.104
μ / mm ⁻¹	0.587	1.670	0.184
<i>F</i> (000)	628.0	1288.0	1448.0
Crystal size/ mm ³	0.185 × 0.151 × 0.087	0.092 × 0.076 × 0.050	0.1 × 0.05 × 0.025
2 Θ range for data collection/°	5.646 to 146.296	7.406 to 140.954	7.402 to 54.968
Index ranges	-12 ≤ <i>h</i> ≤ 10 -13 ≤ <i>k</i> ≤ 13 -19 ≤ <i>l</i> ≤ 19	-26 ≤ <i>h</i> ≤ 25 -14 ≤ <i>k</i> ≤ 8 -16 ≤ <i>l</i> ≤ 16	-22 ≤ <i>h</i> ≤ 22 -16 ≤ <i>k</i> ≤ 16 -23 ≤ <i>l</i> ≤ 20
Reflections collected	20253	21906	76630
Independent reflections, <i>R</i> _{int}	7070, 0.0303	5999, 0.0950	9215, 0.0494
Data/restraints/parameters	7070/6/458	5999/4/420	9215/109/537
Goodness-of-fit on <i>F</i> ²	1.040	1.013	1.200
Final <i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> ≥ 2 σ (<i>I</i>)]	0.0489, 0.1314	0.0599, 0.1435	0.0867, 0.1932
Final <i>R</i> ₁ , <i>wR</i> ₂ [all data]	0.0532, 0.1366	0.0757, 0.1620	0.0978, 0.1986
Largest diff. peak/hole/ e Å ⁻³	0.32/-0.41	0.53/-0.49	0.29/-0.39
Flack parameter	-	0.492(17)	-

Table S2: Crystal Data and Structural Refinement for the Group 11 Compounds **4** – **6**.

	4	5	6
Empirical formula	C ₁₃ H ₃₁ B ₁₀ CuN ₂	C ₁₃ H ₃₁ AgB ₁₀ N ₂	C ₁₃ H ₃₁ AuB ₁₀ N ₂
Formula weight	387.04	431.37	520.7
Crystal system	orthorhombic	monoclinic	orthorhombic
Space group	<i>Pbca</i>	<i>P2₁/m</i>	<i>Pbca</i>
<i>a</i> / Å	17.6117(1)	9.1557(3)	13.3382(1)
<i>b</i> / Å	13.2950(1)	9.8409(4)	17.8016(1)
<i>c</i> / Å	17.8699(1)	11.6235(3)	17.8489(1)
α / °	90	90	90
β / °	90	91.091(3)	90
γ / °	90	90	90
<i>U</i> / Å ³	4184.19(5)	1047.09(6)	4238.07(5)
<i>Z</i>	8	2	8
ρ_{calc} / g cm ⁻³	1.229	1.368	1.631
μ / mm ⁻¹	1.419	7.669	12.996
<i>F</i> (000)	1616.0	440.0	2016.0
Crystal size/ mm ³	0.268 × 0.154 × 0.14	0.121 × 0.041 × 0.03	0.34 × 0.194 × 0.134
2 Θ range for data collection/°	9.694 to 146.45	7.608 to 146.824	9.654 to 146.564
Index ranges	-21 ≤ <i>h</i> ≤ 21 -16 ≤ <i>k</i> ≤ 16 -19 ≤ <i>l</i> ≤ 22	-11 ≤ <i>h</i> ≤ 11 -9 ≤ <i>k</i> ≤ 12 -14 ≤ <i>l</i> ≤ 14	-16 ≤ <i>h</i> ≤ 16 -22 ≤ <i>k</i> ≤ 21 -22 ≤ <i>l</i> ≤ 14
Reflections collected	53231	10376	54313
Independent reflections, <i>R</i> _{int}	4194, 0.0222	2230, 0.0494	4248, 0.0459
Data/restraints/parameters	4194/0/241	2230/5/158	4248/0/242
Goodness-of-fit on <i>F</i> ²	1.069	1.085	1.296
Final <i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> ≥ 2 σ (<i>I</i>)]	0.0283, 0.0805	0.0410, 0.1052	0.0208, 0.0541
Final <i>R</i> ₁ , <i>wR</i> ₂ [all data]	0.0288, 0.0809	0.0453, 0.1080	0.0208, 0.0541
Largest diff. peak/hole/ e Å ⁻³	0.28/-0.32	1.75/-1.74	0.91/-0.51

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