

Supplementary Information for

Dial-a-Base Mechanochemical Synthesis of N-Heterocyclic Carbene Copper Complexes

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General Considerations and Starting Materials

All reactions involving air- and moisture-sensitive compounds were carried out under argon atmosphere using standard Schlenk line and glovebox techniques. NMR experiments using air-sensitive compounds were conducted in J. Young's tap NMR tubes prepared and sealed in a glovebox under argon. Hexane was purified using an MBraun Solvent Purification System and stored over 4 Å molecular sieves. C₆D₆ was dried over a potassium mirror prior to vacuum transfer into a sealed ampoule and storage in the glove box under argon. All reactions were performed in a Retsch PM100 ball mill, in a 25 mL zirconia jar 1/3 filled with 0.5 mm diameter zirconia balls (80 g), and sealed with a Retsch Safety Clamp. All NMR data were acquired at 298 K on an Agilent ProPulse instrument for ¹H (500 MHz), ¹³C (126 MHz), ¹⁹F (470 MHz) and ¹¹B (160 MHz). ¹H and ¹³C NMR spectra were referenced using residual solvent resonances. Data was processed using MestReNova software. Elemental analyses were performed by Elemental Microanalysis Ltd., Okehampton, Devon, U.K. Starting materials were purchased from standard suppliers and used without further purification unless otherwise stated. [IPr]HCl,¹ [6-Dipp]HBr,² [6-Dipp]HCl,³ and [7-Dipp]HBr² were synthesised according to literature conditions. Phenyl lithium was synthesised according to literature conditions,⁴ dried *in vacuo* and stored in the glovebox.

General Procedure A: for the Mechanochemical Synthesis of (NHC)CuX

[NHC]HX (1 g, *ca.* 2 mmol) was placed in a milling jar with CuCl (1 eq., *ca.* 2 mmol), alongside milling balls and toluene (10 mL). The resultant mixture was sealed inside the glovebox, removed, and milled at 500 rpm for 10 minutes. The mixture was returned to the glovebox and KX was added (3 eq.). The jar was resealed, removed again, and milled at 500 rpm for 3 x 30 minutes. The resultant slurry was transferred to a frit and extracted with toluene (20 mL), before removal of all volatiles *in vacuo*. The resultant powder was collected and analysed by NMR. Material for elemental analysis was purified via recrystallisation; hexane was diffused into a toluene solution of (NHC)CuX and the resultant material collected, dried *in vacuo* and analysed by CHN.

General Procedure B: for Mechanochemical Salt Metathesis to Synthesise (6-Dipp)CuX

(6-Dipp)CuCl (1 g, *ca.* 2 mmol) was placed in a milling jar with K₂CO₃ (3 eq., *ca.* 6 mmol), and HX (1.1 eq., *ca.* 2.2 mmol) alongside milling balls and toluene (10 mL). The resultant mixture was sealed inside the glovebox, removed, and milled at 500 rpm for 3 x 30 minutes. The resultant slurry was transferred to a frit and extracted with toluene (20 mL), before removal of all volatiles *in vacuo*. The resultant powder was isolated and analysed with no further purification unless otherwise stated.

(IPr)CuOtBu, 1

Synthesised via general procedure A with [IPr]HCl (1.00 g, 2.35 mmol), CuCl (1 eq., 0.23 g, 2.35 mmol), and KOtBu (3 eq., 0.79 g, 7.06 mmol). Isolated as a white powder (0.80 g, 65 %). Spectroscopic data was consistent with previous reports.⁵

(6-Dipp)CuOtBu, 2

Synthesised via general procedure A with [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol) and KOtBu (3 eq., 0.69 g, 6.18 mmol). Isolated as a white powder (0.93 g, 83 %). Spectroscopic data was consistent with previous reports.⁶

(7-Dipp)CuOtBu, 3

Synthesised via general procedure A with [7-Dipp]HBr (1.00g, 2.00 mmol), CuCl (1 eq., 0.20 g, 2.00 mmol), and KOtBu (3 eq., 0.67 g, 6.00 mmol). Isolated as a white powder (0.76 g, 57 %). Spectroscopic data was consistent with previous reports.³

(6-Dipp)CuOMe, 4

Synthesised via general procedure A with [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol), and NaOMe (3 eq., 0.33 g, 6.18 mmol). Isolated as a white powder (0.51 g, 50 %). Spectroscopic data was consistent with previous reports.⁷

(6-Dipp)CuN(SiMe₃)₂, 5

Synthesised via general procedure A with [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol) and KN(SiMe₃)₂ (2 eq., 0.82 g, 4.11 mmol) over 16 hours (8 x 30 minutes). Reactions using 3 eq. of KN(SiMe₃)₂ provided material contaminated with KN(SiMe₃)₂. Isolated as a white powder (1.04 g, 80 %). ¹H NMR (500 MHz, C₆D₆) δ 7.29 – 7.18 (m, 2H, ArH), 7.12 (m, 4H, ArH), 3.09 (hept, *J* = 6.9 Hz, 4H, iPr CH), 2.74 (t, *J* = 5.9 Hz, 4H, NCH₂CH₂), 1.55 (d, *J* = 6.8 Hz, 12H, iPr CH₃), 1.49 (t, *J* = 5.6 Hz, 2H, NCH₂CH₂), 1.16 (d, *J* = 7.0 Hz, 12H, iPr CH₃), -0.03 (s, 17H, N(SiMe₃)₂). ¹³C NMR (126 MHz, C₆D₆) δ 204.4 (NCN), 145.4 (Ar C), 142.9 (Ar C), 129.5 (Ar C), 125.5 (Ar C), 47.7 (NCH₂CH₂), 28.9 (iPr CH), 25.1 (iPr CH₃), 24.8 (iPr CH₃), 20.94 (NCH₂CH₂), 6.8 (N(SiMe₃)₂). ²⁹Si NMR (99 MHz, C₆D₆) δ -9.4. Anal. calc. for C₃₄H₅₈CuN₃Si₂ C, 64.97; H, 9.30; N, 6.69 %. Found: C, 64.79; H, 9.18; N, 6.53 %.

(6-Dipp)CuCl, 6

Synthesised via general procedure A with [6-Dipp]HCl (1.00 g, 2.27 mmol), CuCl (1 eq., 0.22 g, 2.27 mmol) and K₂CO₃ (3 eq., 0.94 g, 6.80 mmol) over 16 hours (8 x 30 minutes). Isolated as a white powder (0.67 g, 58 %). Spectroscopic data was consistent with previous reports.⁸

(6-Dipp)CuMe, 7

Synthesised via general procedure A with [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol), and MeLi (3 eq., 3.86 mL, 1.6 molar solution in diethyl ether, 6.18 mmol). Isolated as a white powder (0.91 g, 91 %). ¹H NMR (500 MHz, C₆D₆) δ 7.21 – 7.17 (m, 2H, ArH), 7.09 (m, 4H, ArH), 3.11 (hept, *J* = 6.8 Hz, 4H, iPr CH), 2.73 (t, *J* = 5.8 Hz, 4H, NCH₂CH₂), 1.53 (d, *J* = 6.8 Hz, 14H, NCH₂CH₂ + iPr CH₃), 1.22 (d, *J* = 7.0 Hz, 12H, iPr CH₃), -0.92 (s, 3H, CuCH₃). ¹³C NMR (126 MHz, C₆D₆) δ 204.7 (NCN), 145.8 (Ar C), 142.1 (Ar C), 129.1 (Ar C), 124.5 (Ar C), 46.1 (NCH₂CH₂), 28.9 (iPr CH), 25.0 (iPr CH₃), 24.7 (iPr CH₃), 20.6 (NCH₂CH₂), -14.0 (CuCH₃). Anal. calc. for C₂₉H₄₃CuN₂ C, 72.08; H, 8.97; N, 5.80 %. Found: C, 71.99; H, 9.12; N, 5.75 %.

(6-Dipp)CuBu, 8

Synthesised via general procedure A with [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol), and BuLi (3 eq., 2.47 mL, 2.5 molar solution in hexanes, 6.18 mmol). Isolated as a white powder (0.86 g, 79 %). ¹H NMR (500 MHz, C₆D₆) δ 7.24 – 7.17 (m, 2H, ArH), 7.10 (m, 4H, ArH), 3.09 (hept, *J* = 6.9 Hz, 4H, iPr CH), 2.74 – 2.68 (m, 4H, NCH₂CH₂), 1.63 (m, 2H, Bu CH₂), 1.53 (d, *J* = 6.8 Hz, 14H, NCH₂CH₂ + iPr CH₃), 1.22 (d, *J* = 7.0 Hz, 12H, iPr CH₃), 1.13 – 1.03 (m, 2H, Bu CH₂), 0.97 (t, *J* = 7.2 Hz, 3H, Bu CH₃), 0.14 (t, *J* = 7.3 Hz, 2H, Bu CH₂). ¹³C NMR (126 MHz, C₆D₆) δ 204.4 (NCN), 145.4 (Ar C), 141.8 (Ar C), 128.7 (Ar C), 124.2 (Ar C), 45.7 (NCH₂CH₂), 33.2 (Bu CH₂), 30.5 (Bu CH₂), 28.56 (iPr CH), 24.6 (iPr CH₃), 24.3 (iPr CH₃), 20.2 (NCH₂CH₂), 14.5 (Bu CH₃), 10.2 (Bu CH₂). Anal. calc. for C₃₂H₄₉CuN₂ C, 73.17; H, 9.40; N, 5.33 %. Found: C, 71.92; H, 9.37; N, 5.30 %. Best of 3 attempts, correlates well with partial oxidation.

(6-Dipp)CutBu, 9

Synthesised via general procedure A with [6-Dipp] [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol), and tBuLi (3 eq., 3.63 mL, 1.7 molar solution in pentane, 6.18 mmol). Isolated as a white powder (0.87 g, 80 %). ¹H NMR (500 MHz, C₆D₆) δ 7.21 (m, 2H, ArH), 7.10 (m, 4H, ArH), 3.04 (hept, *J* = 6.8 Hz, 4H, iPr CH), 2.72 (t, *J* = 5.9 Hz, 4H, NCH₂CH₂), 1.52 (d, *J* = 6.8 Hz, 14H, NCH₂CH₂ + iPr CH₃), 1.21 (d, *J* = 7.0 Hz, 12H, iPr CH₃), 1.00 (s, 9H, C(CH₃)₃). ¹³C NMR (126 MHz, C₆D₆) δ 205.1 (NCN), 145.8 (Ar C), 142.2 (Ar C), 129.1 (Ar C), 124.6 (Ar C), 45.8 (NCH₂CH₂), 35.3 (C(CH₃)₃), 29.0 (iPr CH), 24.8 (iPr CH₃), 24.8 (iPr CH₃), 20.4 (NCH₂CH₂), 14.4 (C(CH₃)₃). Anal. calc. C₃₂H₄₉CuN₂ C, 73.17; H, 9.40; N, 5.33 %. Found: C, 72.98; H, 9.41; N, 5.27 %.

(6-Dipp)CuPh, 10

Synthesised via general procedure A with [6-Dipp]HBr (1.00 g, 2.06 mmol), CuCl (1 eq., 0.20 g, 2.06 mmol), and PhLi (3 eq., 0.52 g, 6.18 mmol). Isolated as a white powder (0.94 g, 82 %). ¹H NMR (500 MHz, C₆D₆) δ 7.25 (t, *J* = 7.6 Hz, 4H, ArH), 7.18 (d, *J* = 7.3 Hz, 2H, ArH), 7.12 (m, 4H, ArH), 7.10 – 7.04 (m, 1H, ArH), 3.08 (hept, *J* = 7.0 Hz, 4H, iPr CH), 2.74 (t, *J* = 5.9 Hz, 4H, NCH₂CH₂), 1.54 – 1.44 (m, 14H, NCH₂CH₂ + iPr CH₃), 1.20 (d, *J* = 6.8 Hz, 12H, iPr CH₃). ¹³C NMR (126 MHz, C₆D₆) δ 205.2 (NCN), 166.2 (Ar C), 145.9 (Ar C), 142.0 (Ar C), 140.6 (Ar C), 129.3 (Ar C), 126.1 (Ar C), 124.8 (Ar C), 124.1 (Ar C), 45.9 (NCH₂CH₂), 29.0 (iPr CH), 25.2 (iPr CH₃), 24.7 (iPr CH₃), 20.40 (NCH₂CH₂). Anal. calc. for C₃₄H₄₅CuN₂ C, 74.89; H, 8.32; N, 5.14 %. Found: C, 74.08; H, 8.38; N, 5.12 %.

(6-Dipp)CuCCPh, 11

Synthesised via general procedure B with [6-Dipp]CuCl (1.00 g, 1.99 mmol), K₂CO₃ (3 eq., 0.82 g, 5.96 mmol), and PhCCH (1.1 eq., 0.22 g, 2.18 mmol). Isolated as a white powder (0.91 g, 81 %). Spectroscopic data was consistent with previous reports.⁹

(6-Dipp)CuOCH(CF₃)₂, 12

Synthesised via general procedure B with [6-Dipp]CuCl (1.00 g, 1.99 mmol), K₂CO₃ (3 eq., 0.82 g, 5.96 mmol), and HOCH(CF₃)₂ (1.1 eq., 0.37 g, 2.18 mmol). Isolated as a white powder (0.91 g, 73 %). ¹H NMR (400 MHz, C₆D₆) δ 7.16 (m, 2H, ArH), 7.04 (m, 4H, ArH), 4.20 (hept, *J* = 6.5 Hz, 1H, OCH(CF₃)₂), 2.96 (hept, *J* = 6.9 Hz, 4H, iPr CH), 2.67 (t, *J* = 5.8 Hz, 4H, NCH₂CH₂), 1.49 – 1.37 (m, 14H, NCH₂CH₂ + iPr CH₃), 1.15 (d, *J* = 6.9 Hz, 12H, iPr CH₃). ¹³C NMR (126 MHz, C₆D₆) δ 202.1 (NCN), 145.5 (Ar C), 142.1 (Ar C), 129.6 (Ar C), 126.0 (br., OCH(CF₃)₂), 125.0 (Ar C), 123.7 (br., OCH(CF₃)₂), 76.2 (hept, *J* = 29.6 Hz, OCH(CF₃)₂), 46.0 (NCH₂CH₂), 28.9 (iPr CH), 24.7 (iPr CH₃), 24.6 (iPr CH₃), 20.10 (NCH₂CH₂). ¹⁹F NMR (376 MHz, C₆D₆) δ -76.9 (d, *J* = 6.4 Hz). Anal. calc. for C₃₁H₄₁CuF₆N₂O C, 58.62; H, 6.51; N, 4.41 %. Found: C, 58.78; H, 6.64; N, 4.43 %.

X-ray Crystallography

7, **8**, and **10** were collected on a SuperNova, Dual, Cu at home/near, EosS2 diffractometer with Cu source ($\lambda = 1.54184$). **9** was collected on a New Xcalibur, EosS2 diffractometer with Mo source ($\lambda = 0.71073$). Crystals were maintained at 150 K during collection. Using Olex2,¹⁰ the structure was solved with the SHELXT structure solution program using Intrinsic Phasing and refined with the SHELXL refinement package using Least Squares minimisation.¹¹

Half of a molecule constitutes the asymmetric unit in the structure of **7**. Cu1, C1, C16, C3, H3a and H3b are coincident with a crystallographic mirror plane which generates the remainder of the complex.

A satisfactory refinement was achieved for **9** once 55:45 disorder was accounted for in relation to C3 and 70:30 disorder for the methyl groups pertaining to the tert-butyl ligand. Distance and ADP restraints were employed, on merit, in disordered regions to assist convergence.

The asymmetric unit in the structure of **10** comprises half of a molecule of the copper complex and a region of solvent. Cu1, C1, C16 and C19 are co-located with a crystallographic 2-fold rotation axis, which serves to generate the remainder of the molecule. C3 is necessarily disordered about this 2-fold axis and its associated hydrogen atoms, plus the four (half-occupancy) hydrogens attached to C2, were readily located and refined with distance restraints but free U_{iso} values. C10-C12 were also treated for 55:45 disorder, with the inclusion of appropriate distance and ADP restraints. The solvent sits in channels along the *c*-axis and is both diffuse and disordered. It was treated using the masking algorithm available in Olex-2 and an allowance made, in the formula as presented, for 1/8 molecules of pentane per asymmetric unit.

Table S1. Crystal data and structure refinement details.

	7	8	9	10
Identification code				
Empirical formula	C ₂₉ H ₄₃ CuN ₂	C ₃₂ H ₄₉ N ₂ Cu	C ₃₂ H ₄₉ N ₂ Cu	C _{35.25} H ₄₈ CuN ₂
Formula weight	483.19	525.27	525.27	563.29
Temperature/K	150.00(10)	150.00(10)	149.9(6)	150.00(10)
Crystal system	orthorhombic	orthorhombic	monoclinic	trigonal
Space group	Pnma	P2 ₁ 2 ₁ 2 ₁	P2 ₁ /n	R-3c
a/Å	15.2570(1)	10.8840(1)	10.9361(6)	27.8759(4)
b/Å	21.1082(1)	16.2724(1)	15.2910(6)	27.8759(4)
c/Å	8.5870(1)	17.2809(1)	18.7521(8)	22.0267(4)
α/°	90	90	90	90
β/°	90	90	100.920(4)	90
γ/°	90	90	90	120
Volume/Å ³	2765.42(4)	3060.60(4)	3079.0(2)	14823.1(5)
Z	4	4	4	18
ρ _{calc} /cm ³	1.161	1.140	1.133	1.136
μ/mm ⁻¹	1.222	1.140	0.730	1.095
F(000)	1040.0	1136.0	1136.0	5445.0
Crystal size/mm ³	0.19 × 0.099 × 0.053	0.148 × 0.104 × 0.094	0.426 × 0.304 × 0.237	0.16 × 0.14 × 0.08
Radiation	Cu Kα (λ = 1.54184)	Cu Kα (λ = 1.54184)	Mo Kα (λ = 0.71073)	Cu Kα (λ = 1.54184)
2θ range for data collection/°	8.378 to 144.988	7.462 to 144.65	6.542 to 59.294	8.826 to 146.002
Index ranges	-16 ≤ h ≤ 18, -26 ≤ k ≤ 25, -9 ≤ l ≤ 10	-13 ≤ h ≤ 13, -19 ≤ k ≤ 20, -20 ≤ l ≤ 21	-15 ≤ h ≤ 14, -20 ≤ k ≤ 20, -26 ≤ l ≤ 24	-34 ≤ h ≤ 34, -34 ≤ k ≤ 33, -22 ≤ l ≤ 26
Reflections collected	27151	38970	27835	42986
Independent reflections	2822 [R _{int} = 0.0290, R _{sigma} = 0.0133]	6058 [R _{int} = 0.0318, R _{sigma} = 0.0183]	8130 [R _{int} = 0.0323, R _{sigma} = 0.0392]	3271 [R _{int} = 0.0864, R _{sigma} = 0.0287]
Data/restraints/parameters	2822/0/156	6058/0/325	8130/78/366	3271/80/231
Goodness-of-fit on F ²	1.068	1.063	1.022	1.036
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0274, wR ₂ = 0.0754	R ₁ = 0.0234, wR ₂ = 0.0636	R ₁ = 0.0438, wR ₂ = 0.1025	R ₁ = 0.0479, wR ₂ = 0.1334
Final R indexes [all data]	R ₁ = 0.0288, wR ₂ = 0.0764	R ₁ = 0.0243, wR ₂ = 0.0643	R ₁ = 0.0700, wR ₂ = 0.1158	R ₁ = 0.0588, wR ₂ = 0.1422
Largest diff. peak/hole / e Å ⁻³	0.31/-0.32	0.23/-0.14	0.52/-0.42	0.30/-0.29

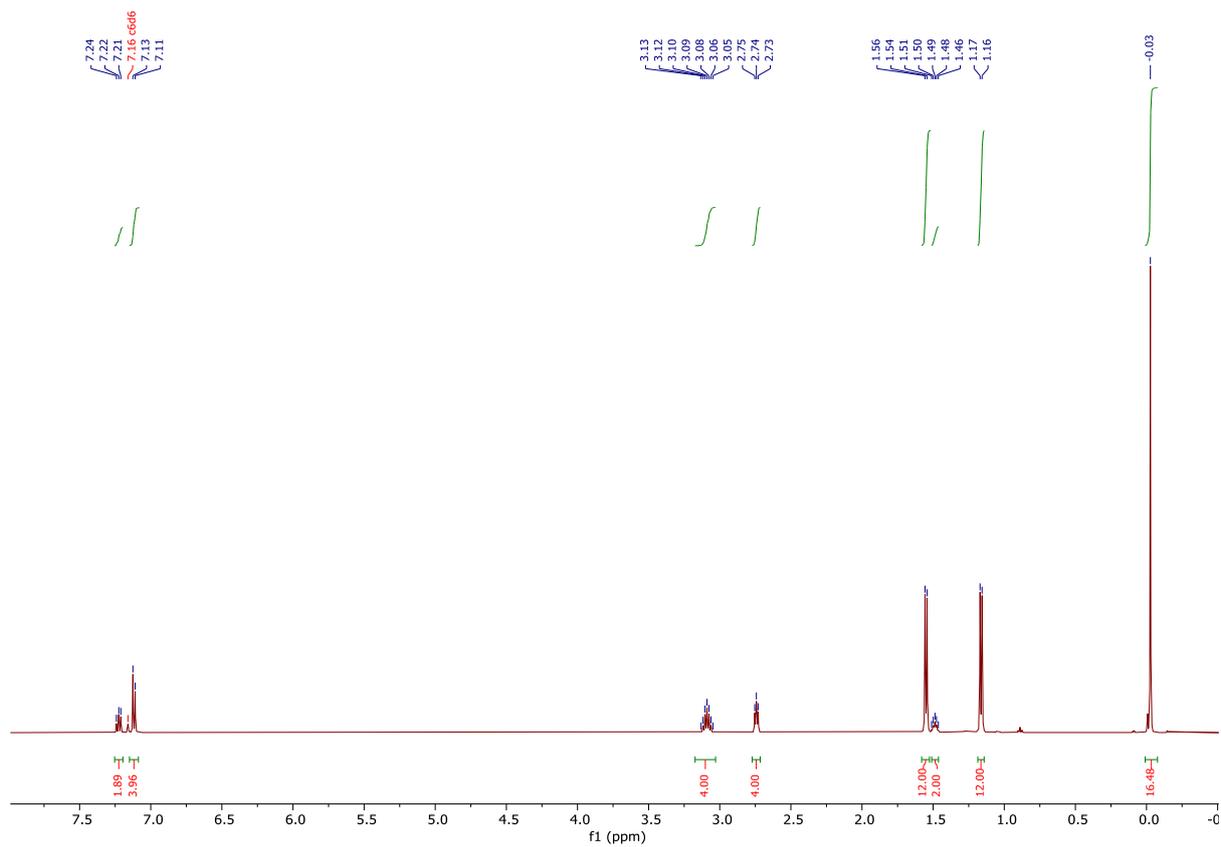


Figure S1. ^1H NMR spectrum (500 MHz, C_6D_6) of **(6-Dipp)CuN(SiMe₃)₂, 5**.

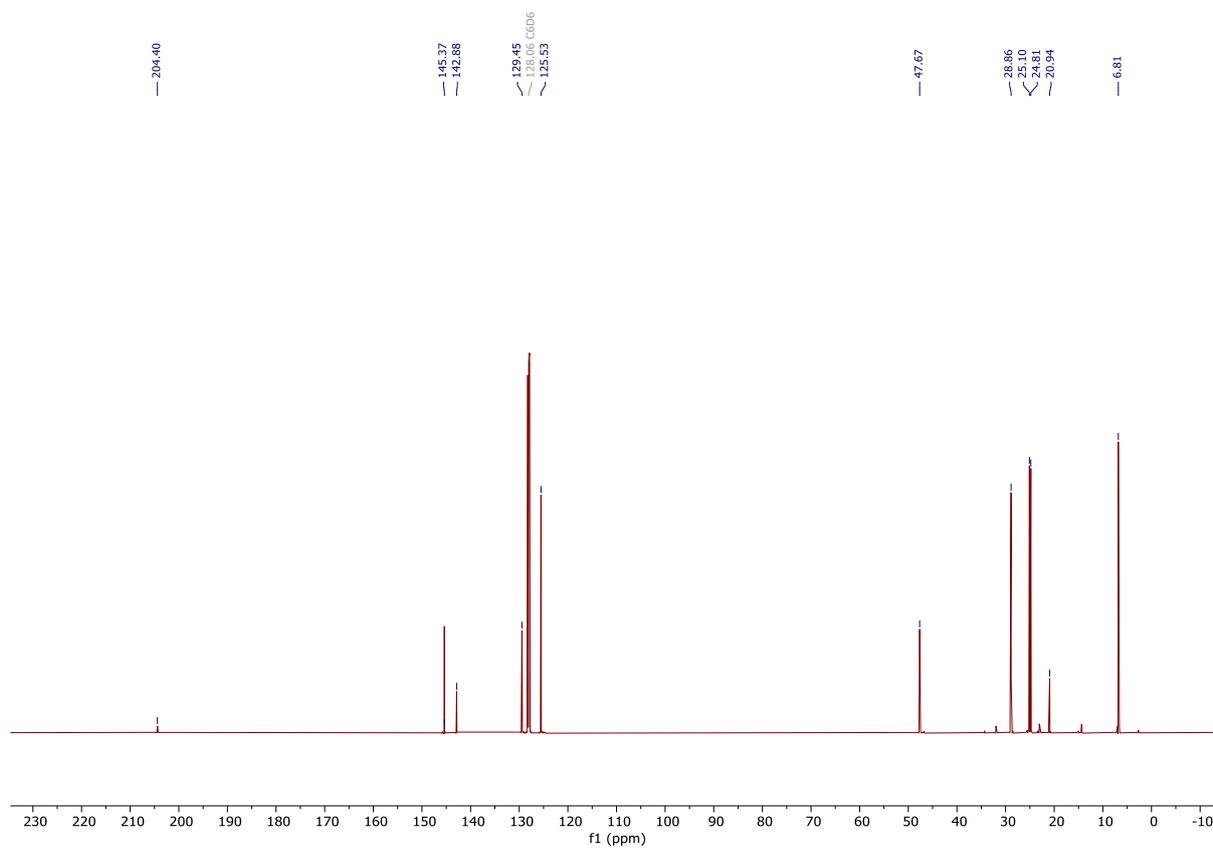


Figure S2. ^{13}C NMR spectrum (126 MHz, C_6D_6) of **(6-Dipp)CuN(SiMe₃)₂, 5**.

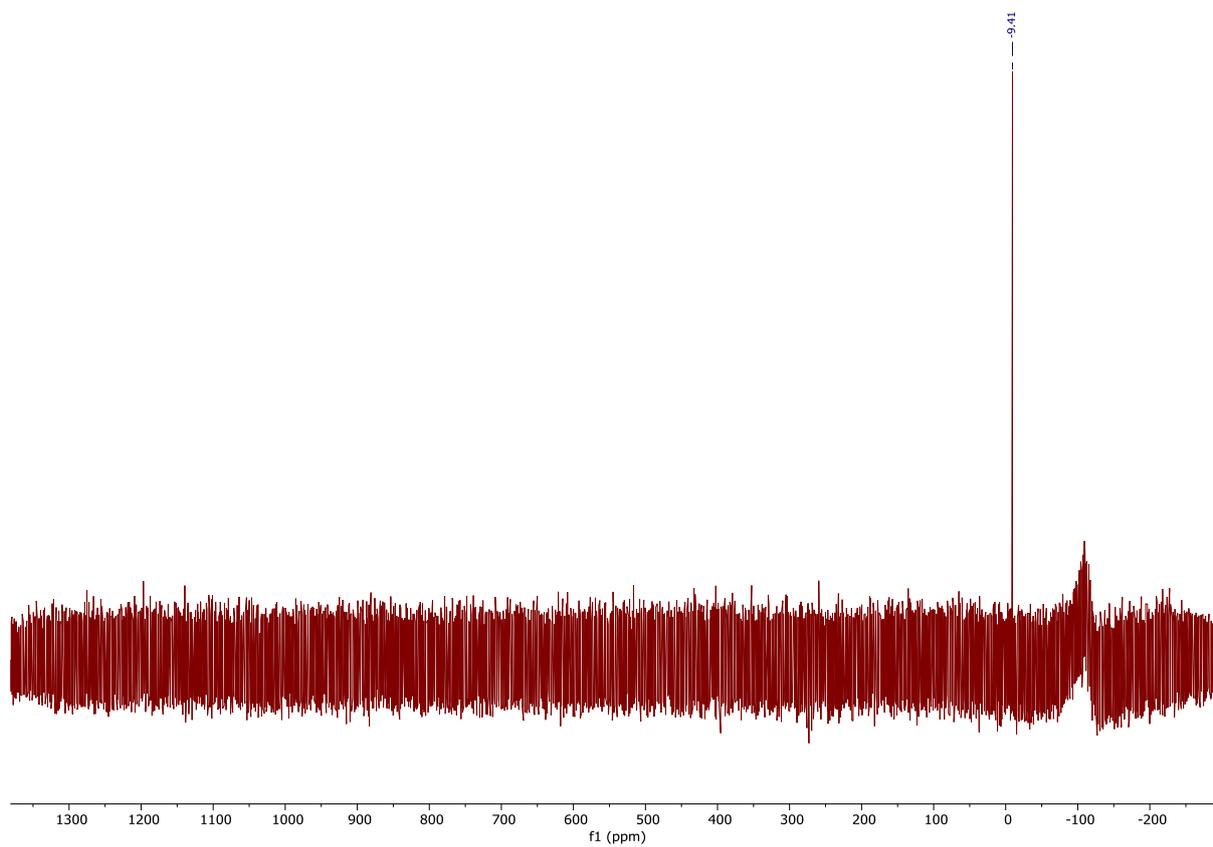


Figure S3. ^{29}Si NMR spectrum (99 MHz, C_6D_6) of **(6-Dipp)CuN(SiMe₃)₂, 5**.

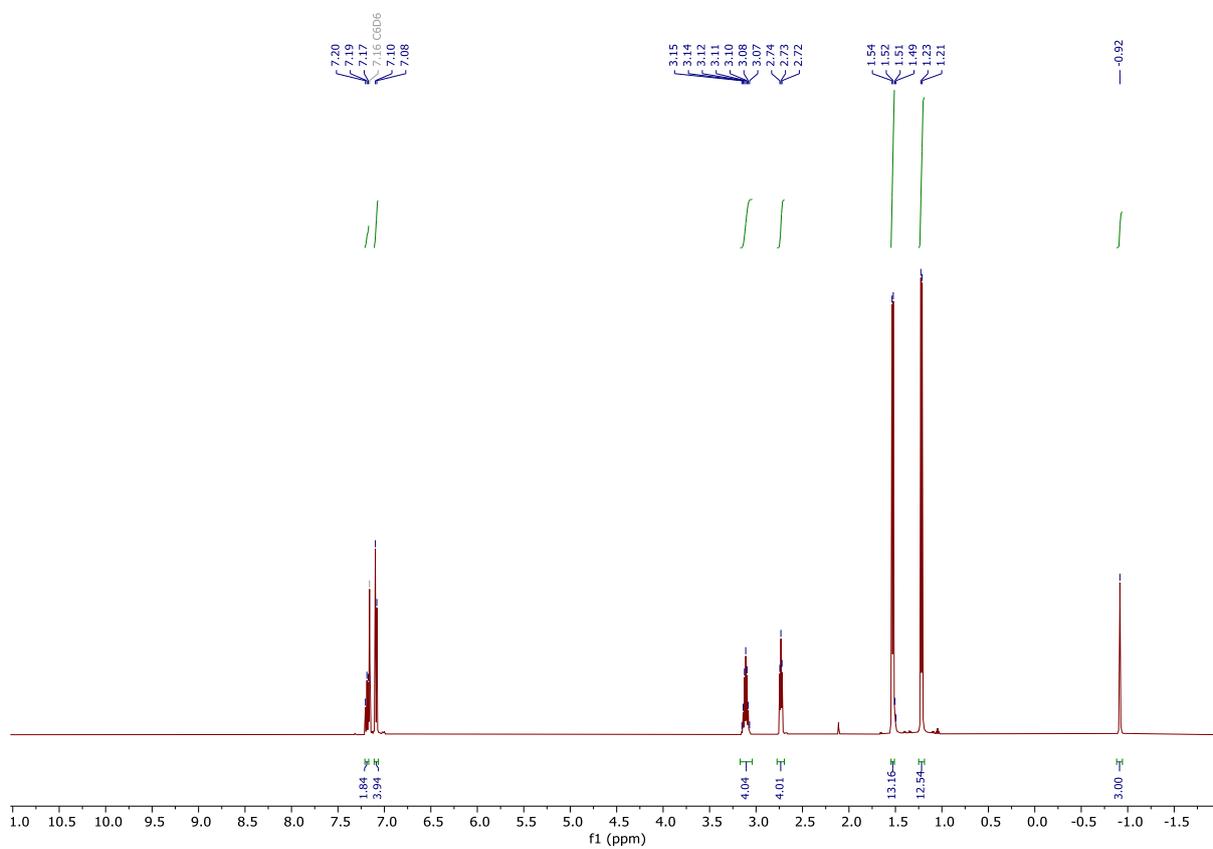


Figure S4. ^1H NMR spectrum (500 MHz, C_6D_6) of **(6-Dipp)CuMe, 7**.

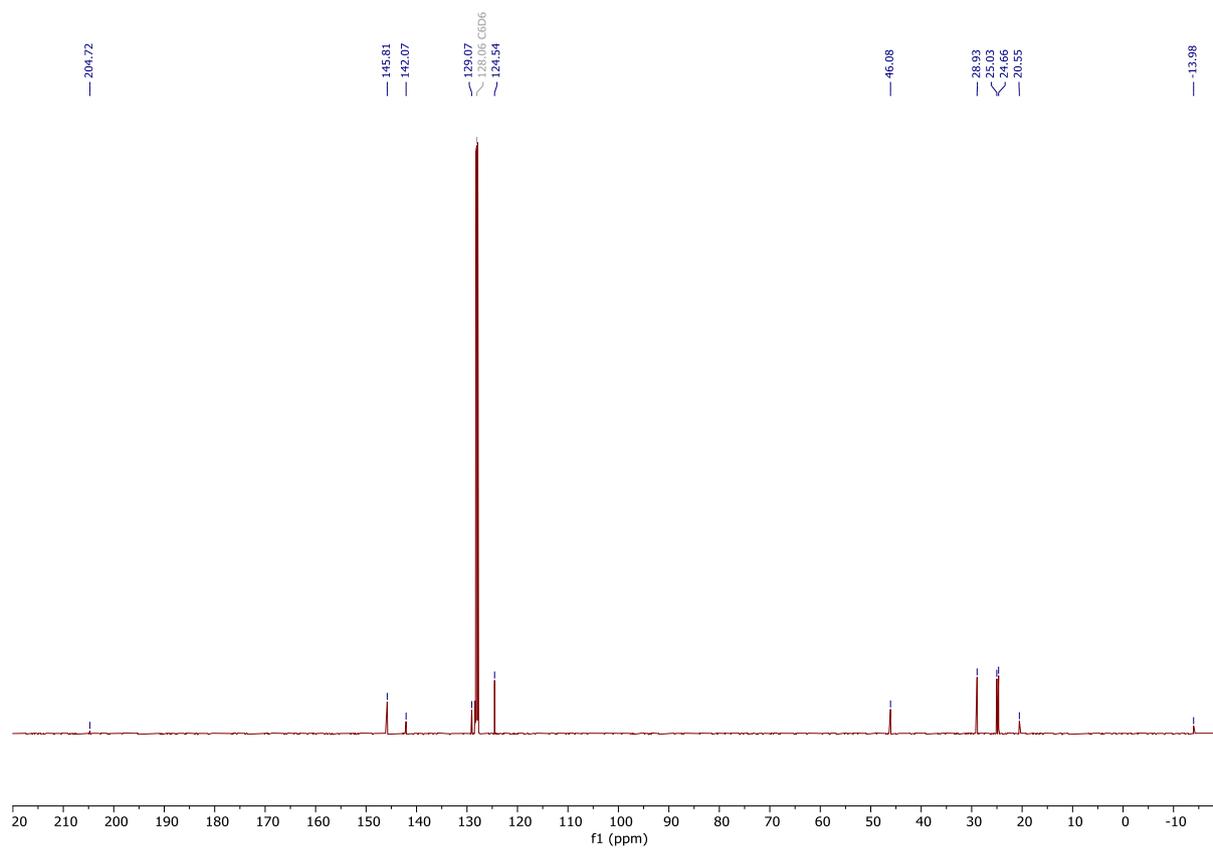


Figure S5. ¹³C NMR spectrum (126 MHz, C₆D₆) of **(6-Dipp)CuMe, 7**.

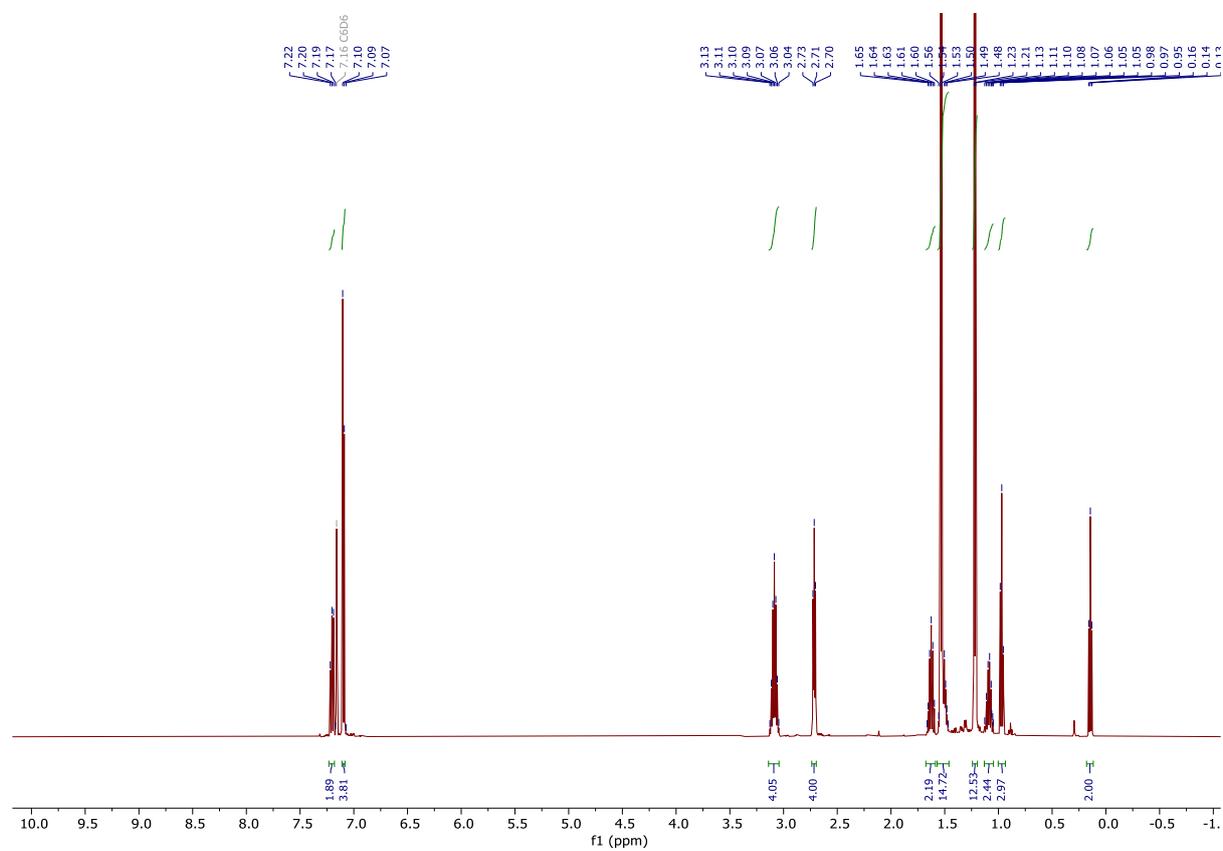


Figure S6. ¹H NMR spectrum (500 MHz, C₆D₆) of **(6-Dipp)CuBu, 8**.

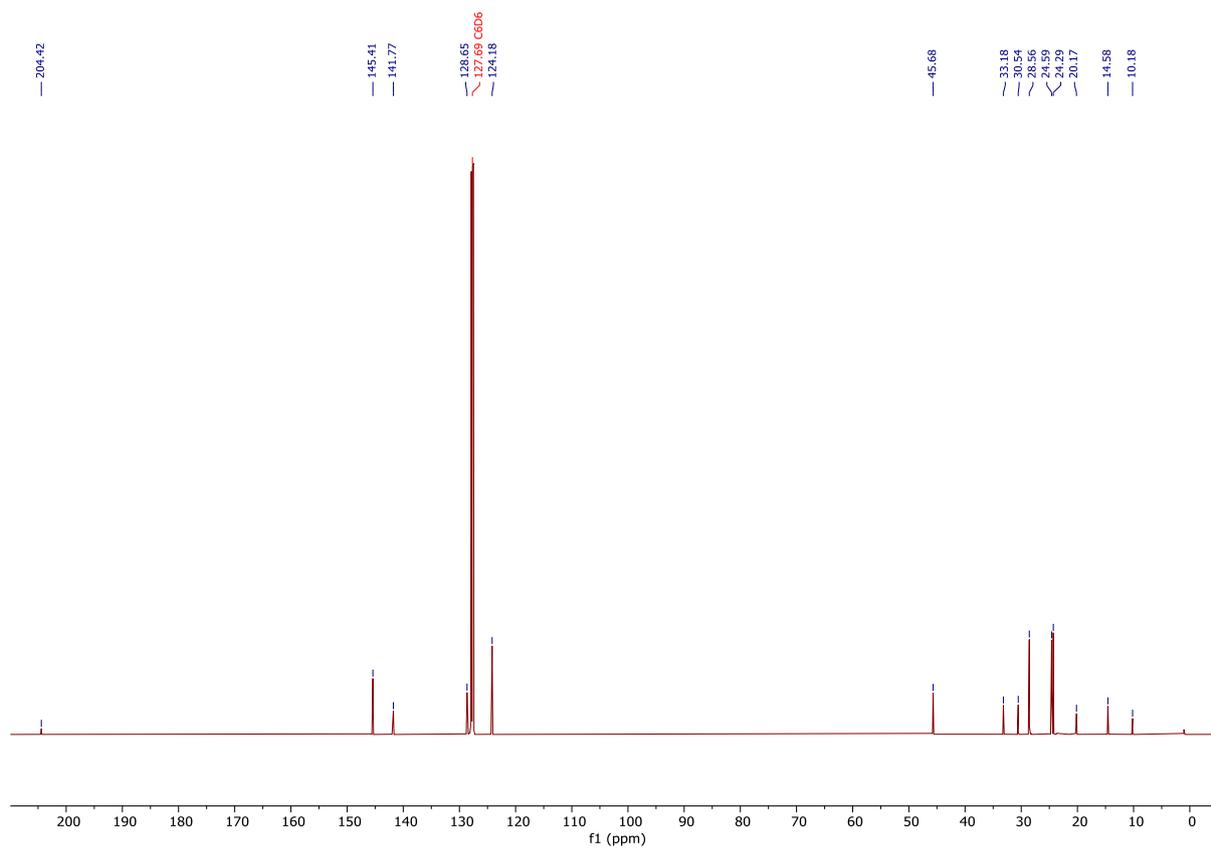


Figure S7. ^{13}C NMR spectrum (126 MHz, C_6D_6) of **(6-Dipp)CuBu, 8**.

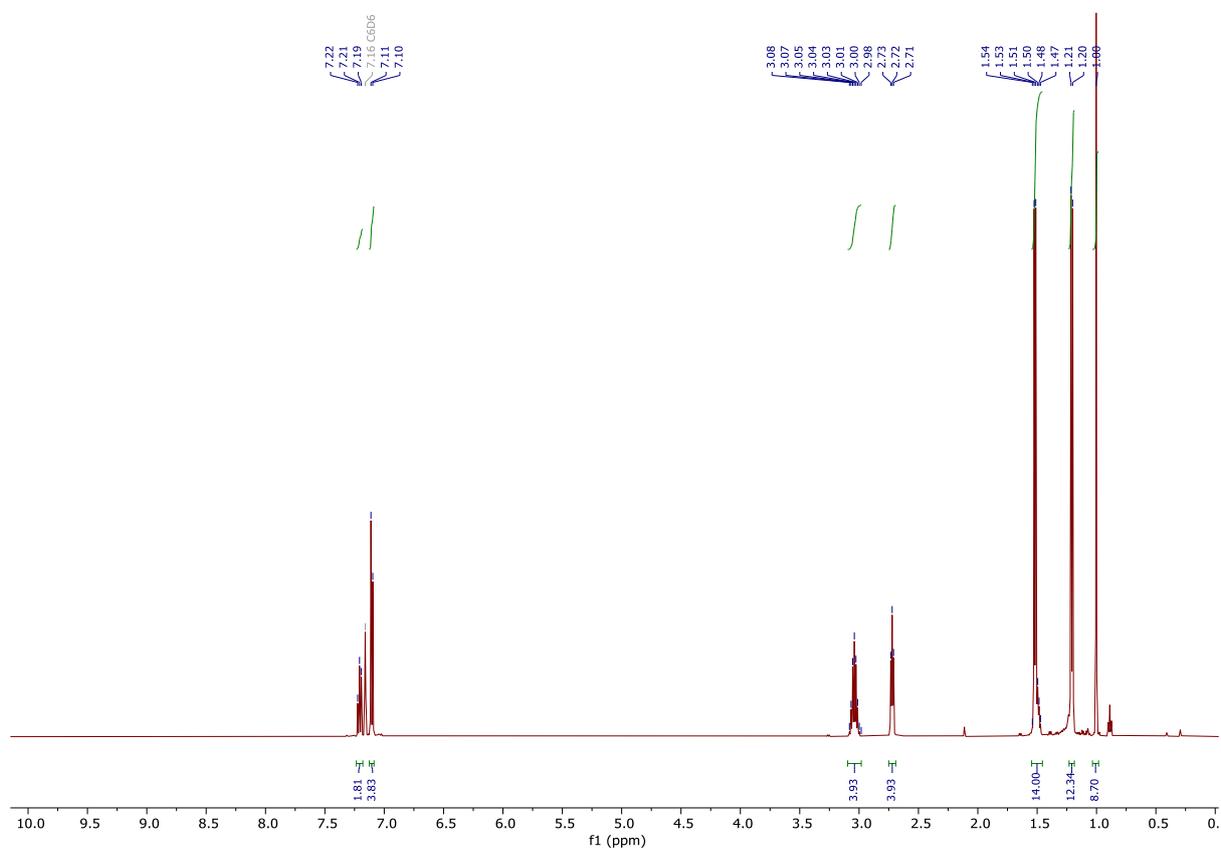


Figure S8. ^1H NMR spectrum (500 MHz, C_6D_6) of **(6-Dipp)CutBu, 9**.

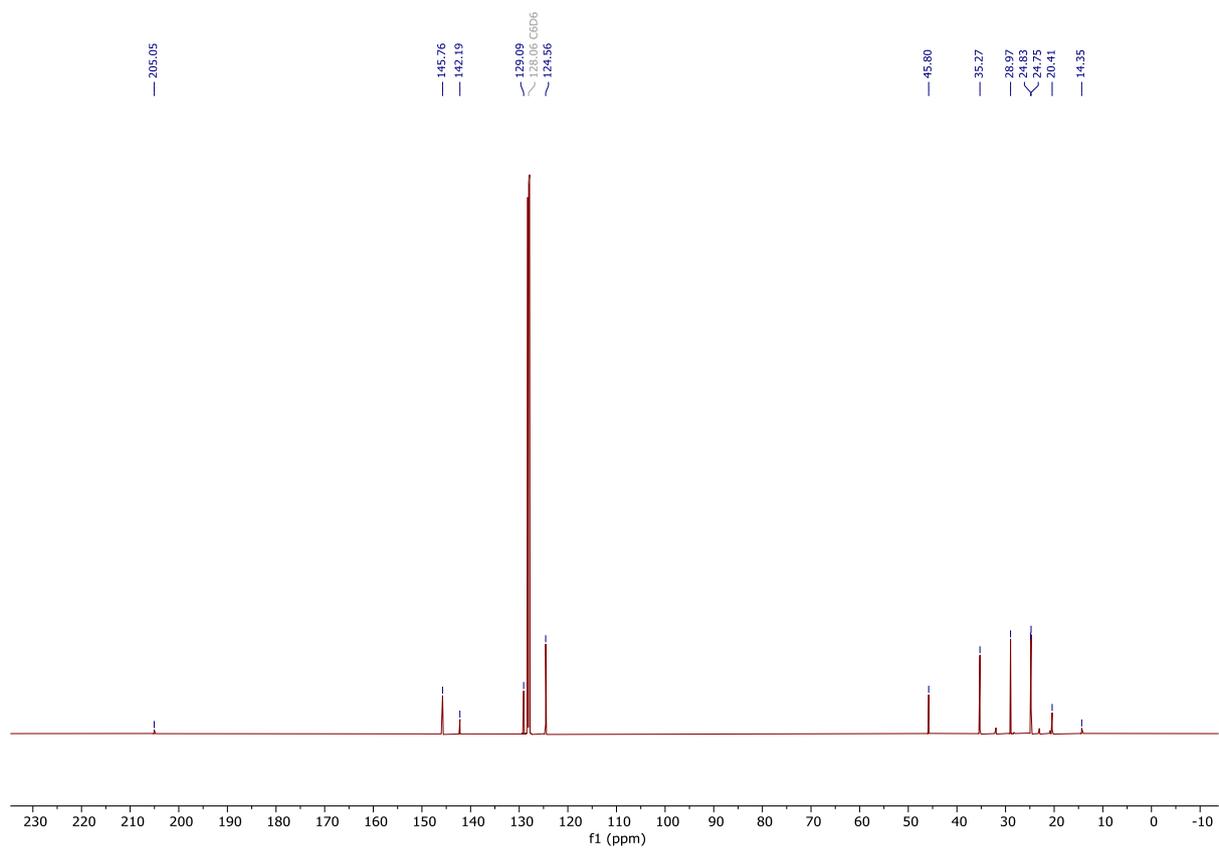


Figure S9. ^{13}C NMR spectrum (126 MHz, C_6D_6) of **(6-Dipp)CutBu, 9**.

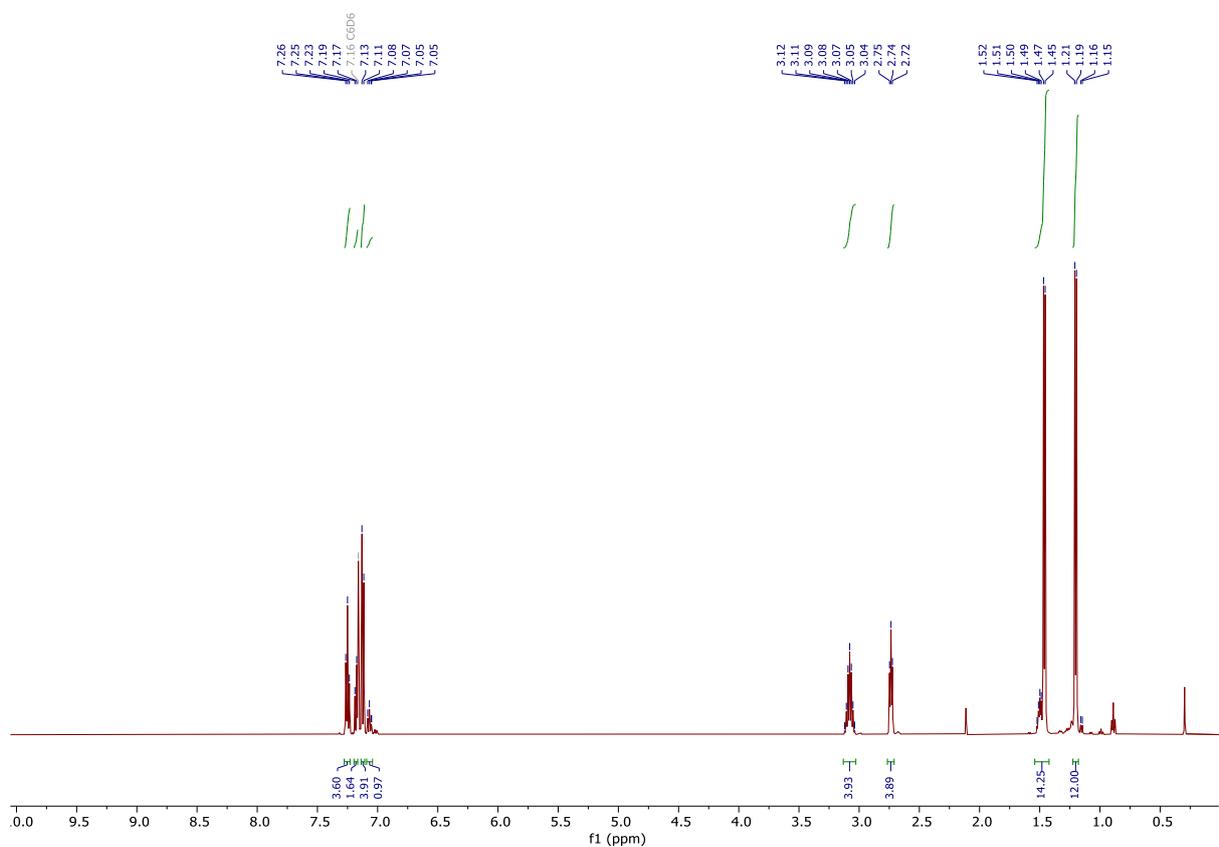


Figure S10. ^1H NMR spectrum (500 MHz, C_6D_6) of **(6-Dipp)CuPh, 10**.

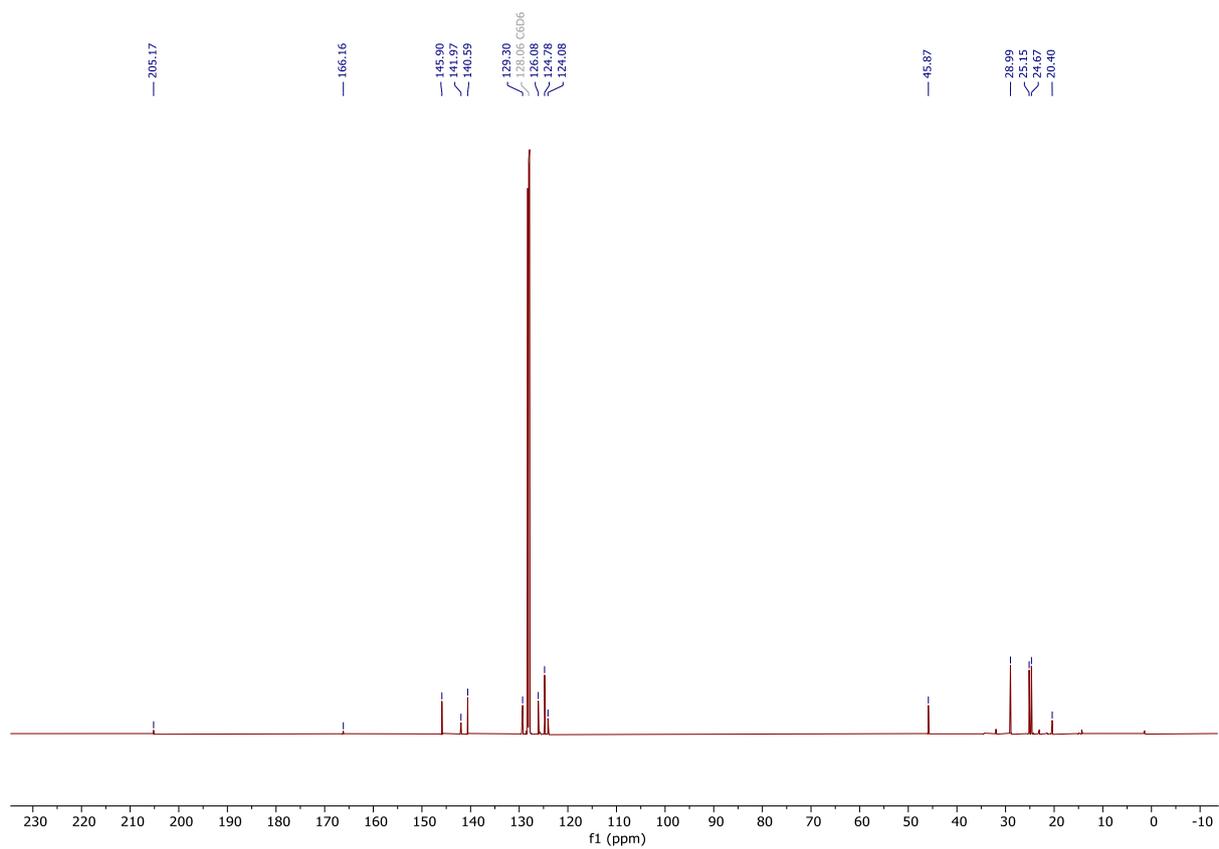


Figure S11. ^{13}C NMR spectrum (126 MHz, C_6D_6) of **(6-Dipp)CuPh, 10**.

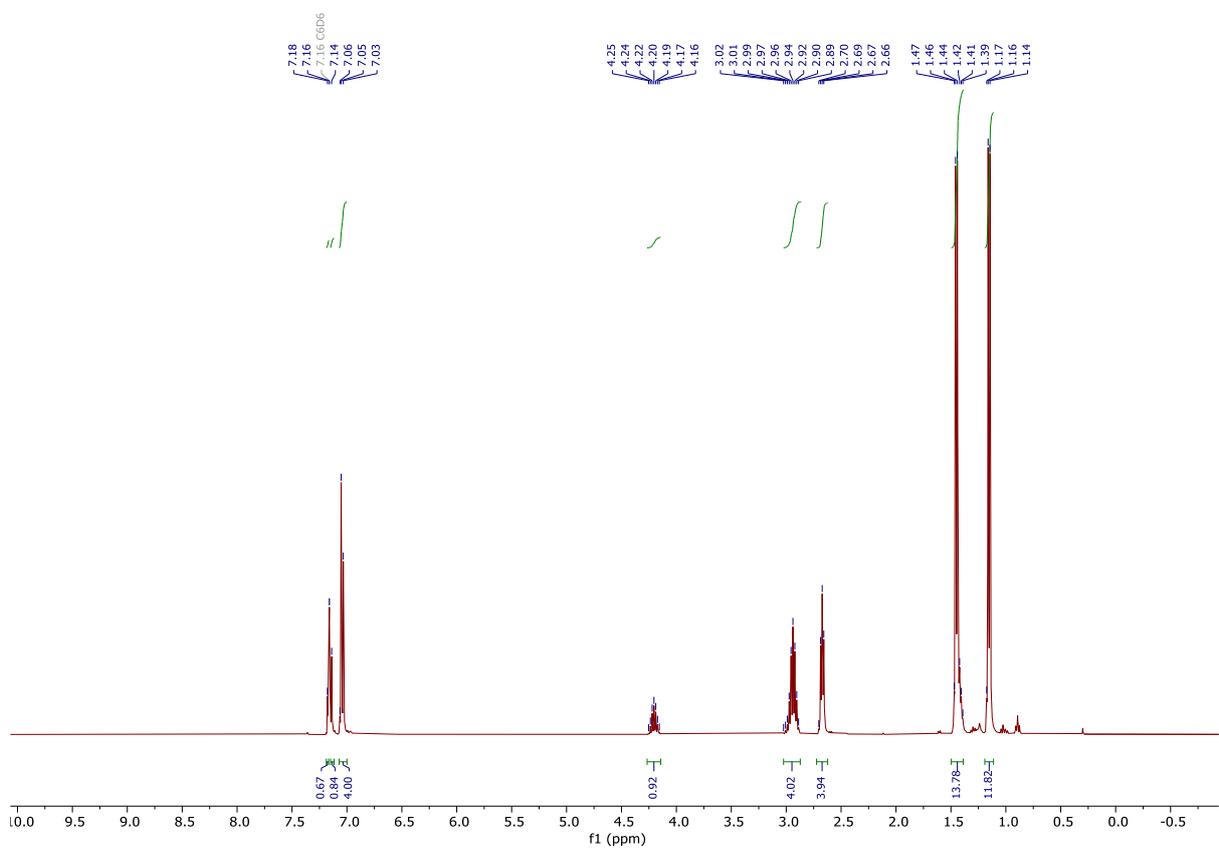


Figure S12. ^1H NMR spectrum (400 MHz, C_6D_6) of **(6-Dipp)CuOCH(CF₃)₂, 12**.

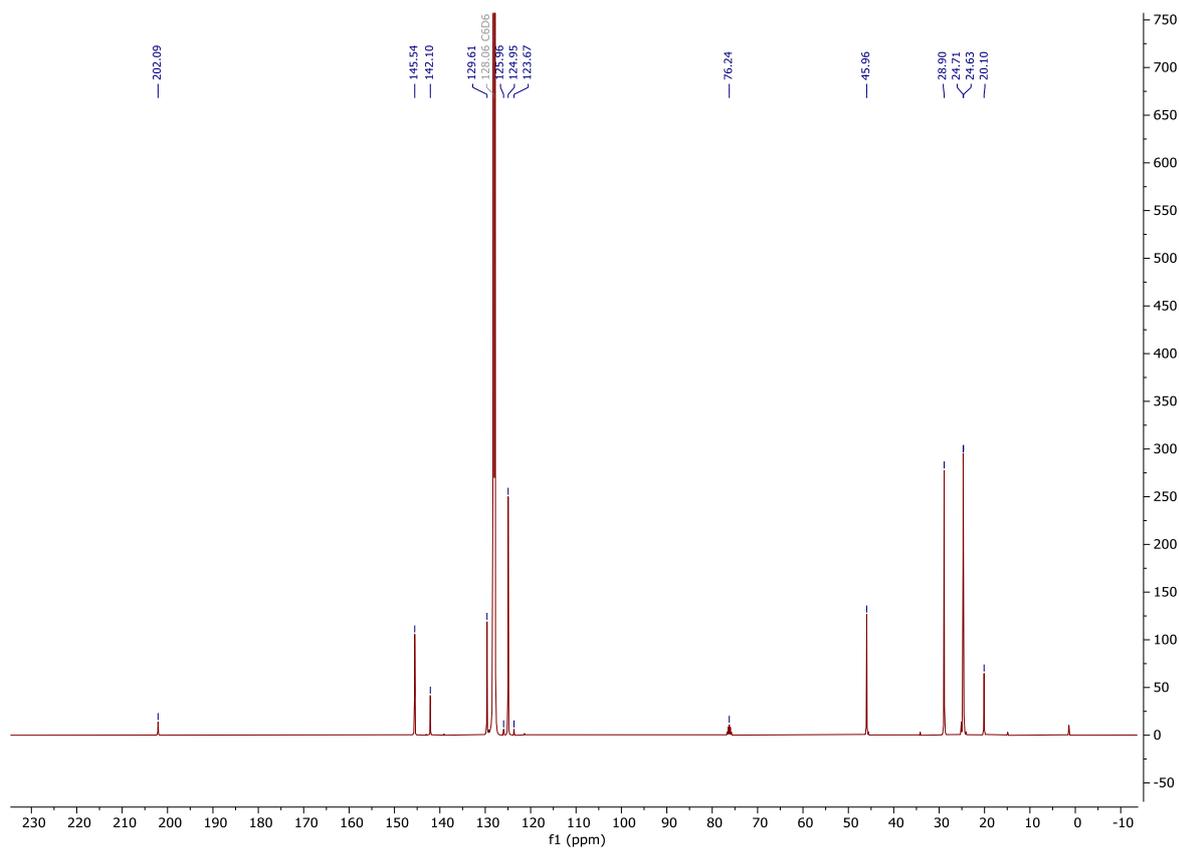


Figure S13. ^{13}C NMR spectrum (126 MHz, C_6D_6) of **(6-Dipp)CuOCH(CF₃)₂, 12**.

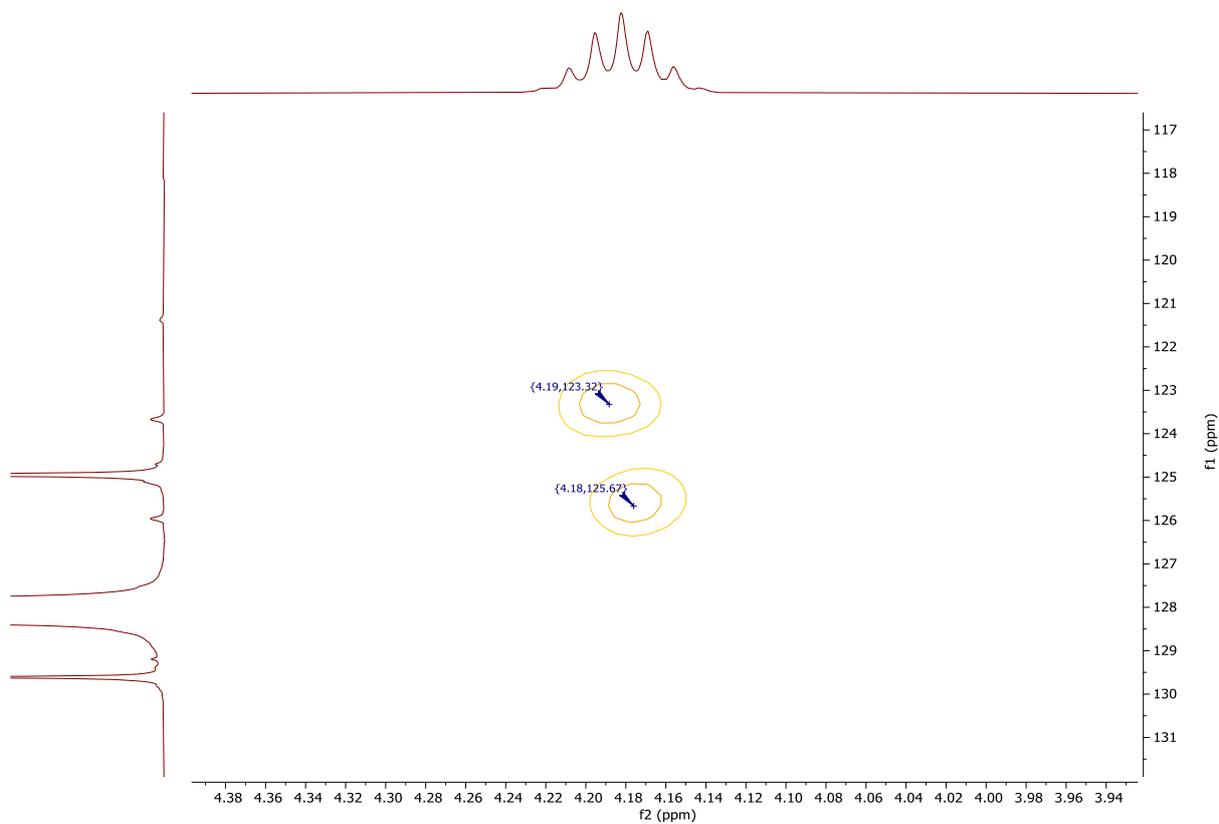


Figure S14. Inset of ^1H - ^{13}C NMR HMBC of **(6-Dipp)CuOCH(CF₃)₂, 12**.

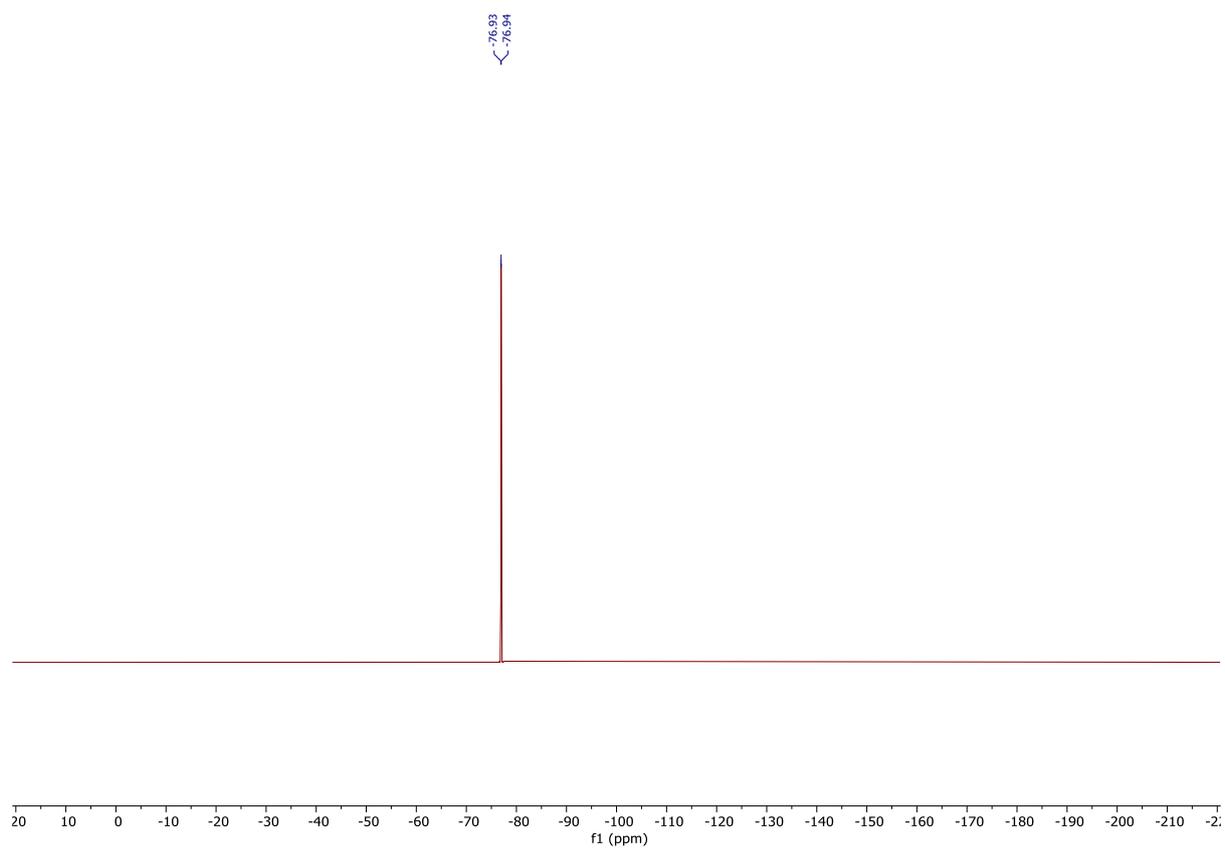


Figure S15. ^{19}F NMR spectrum (376 MHz, C_6D_6) of **(6-Dipp)CuOCH(CF₃)₂, 12**.

References

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