SUPPORTING INFORMATION

for

Metalation Behavior of a Bis-Saturated NHC Ligand with a Flexible *m*-Xylyl Linker

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I. Spectroscopic Data

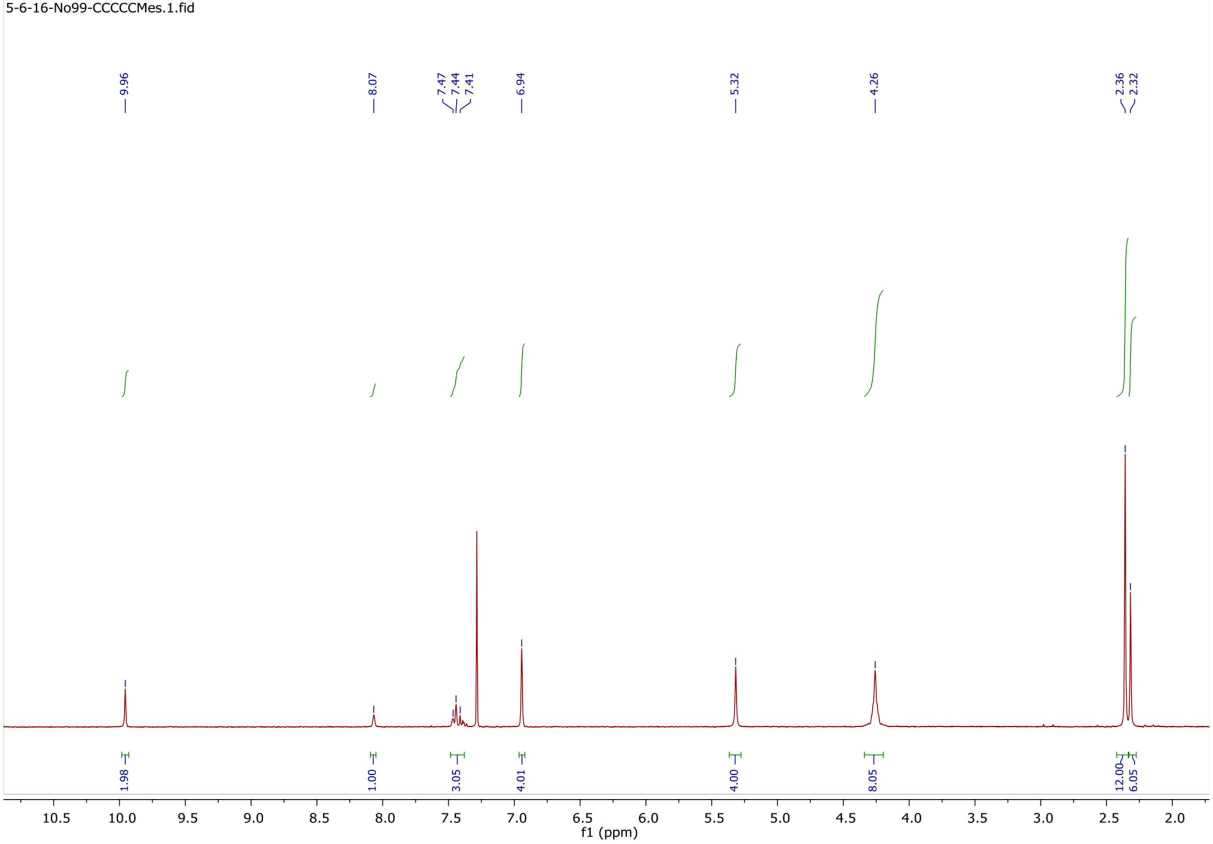

Figure S1. The ^{1}H NMR spectrum of compound **[4a][Br]**.

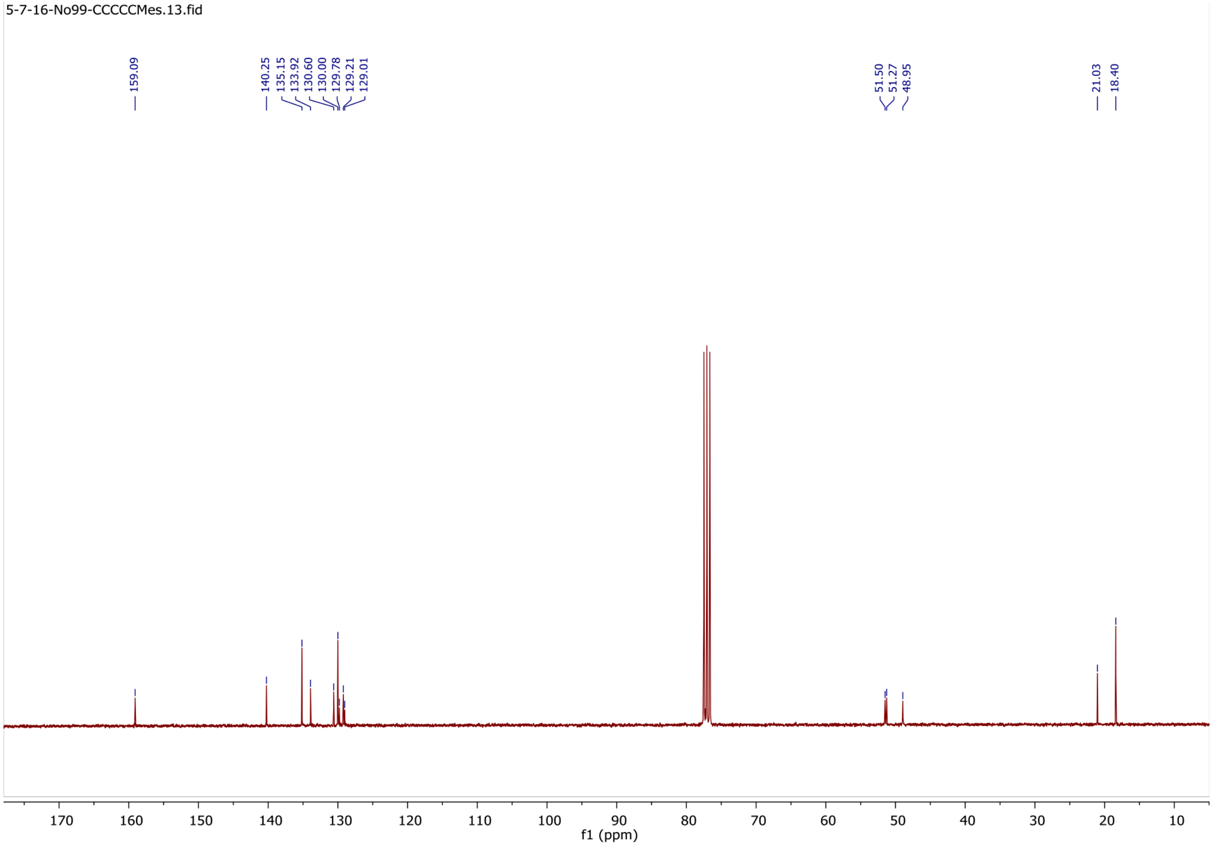

Figure S2. The ^{13}C NMR spectrum of compound **[4a][Br]**.

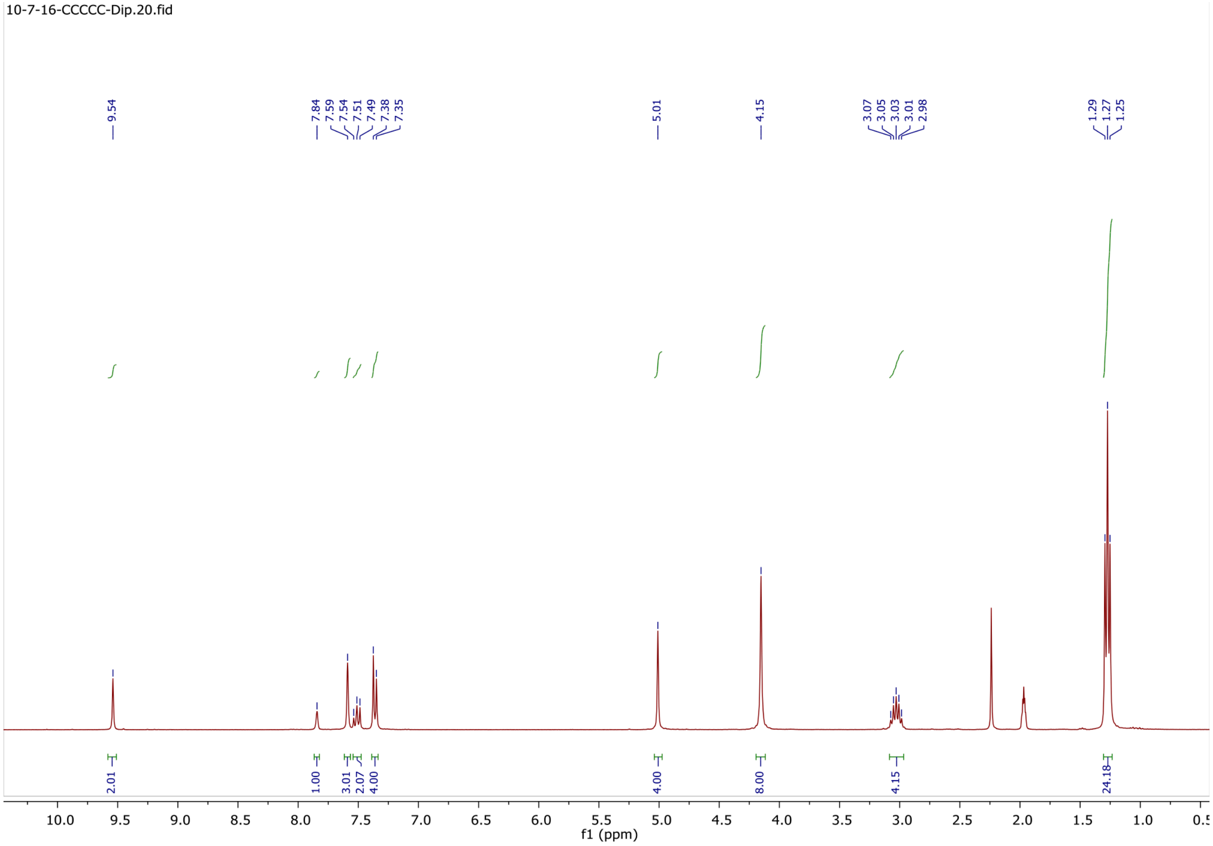

Figure S3. The 1H NMR spectrum of compound [**4b[Br]**.

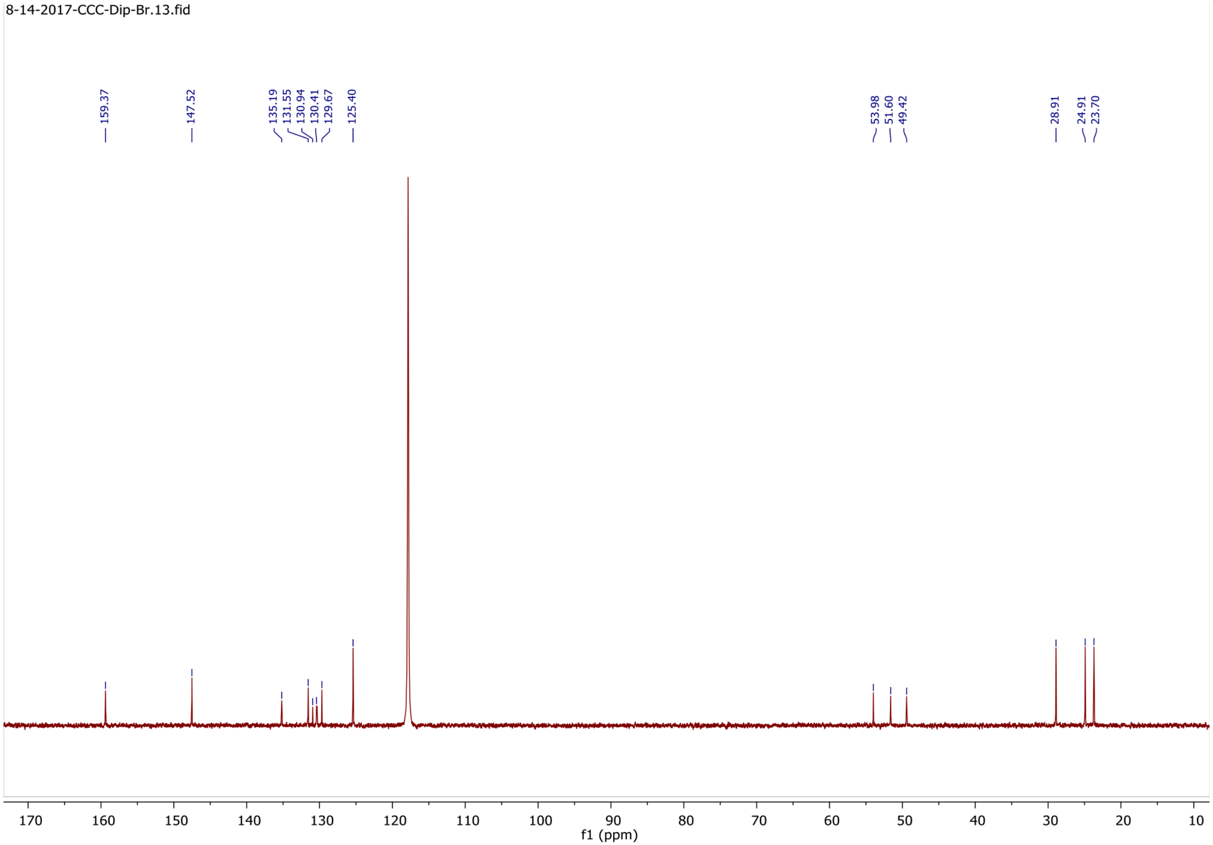

Figure S4. The ^{13}C NMR spectrum of compound [**4b[Br]**.

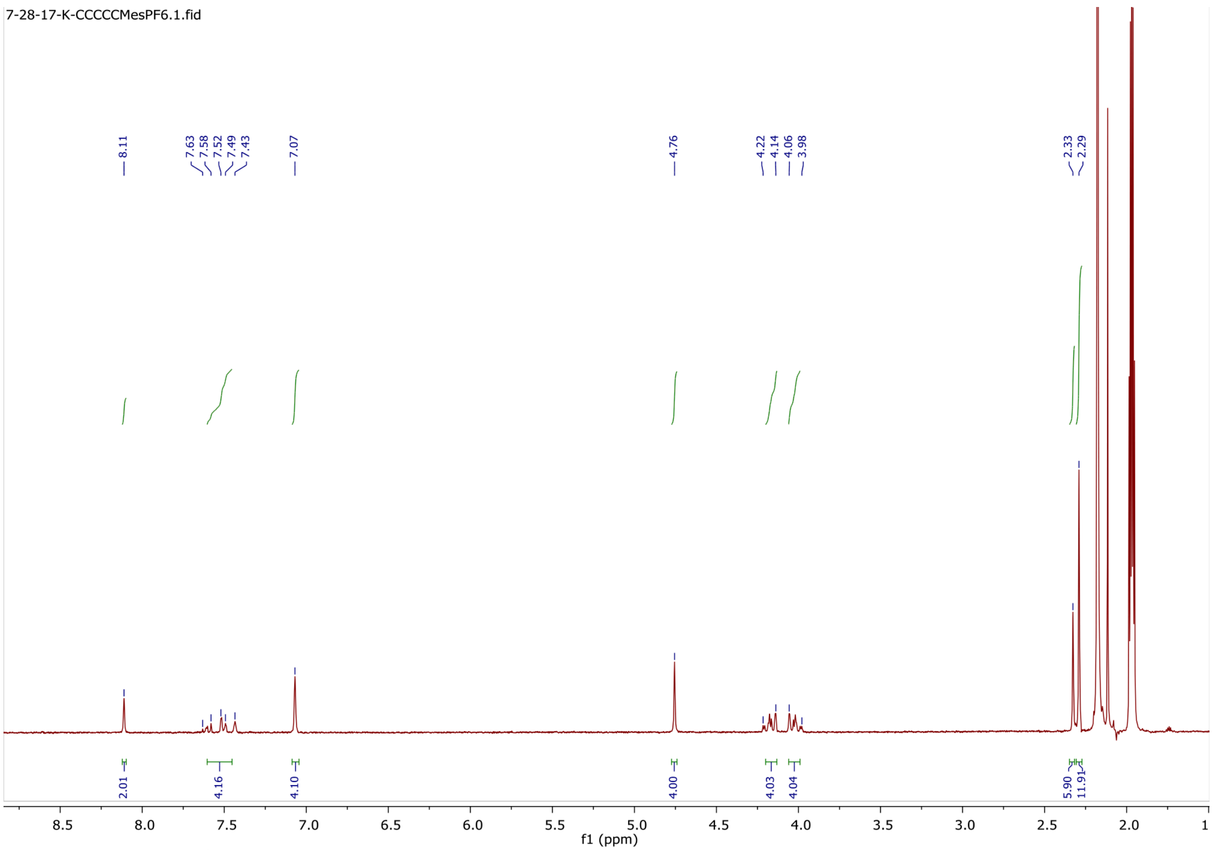

Figure S5. The ^{1}H NMR spectrum of compound **[4a][PF$_6$]**.

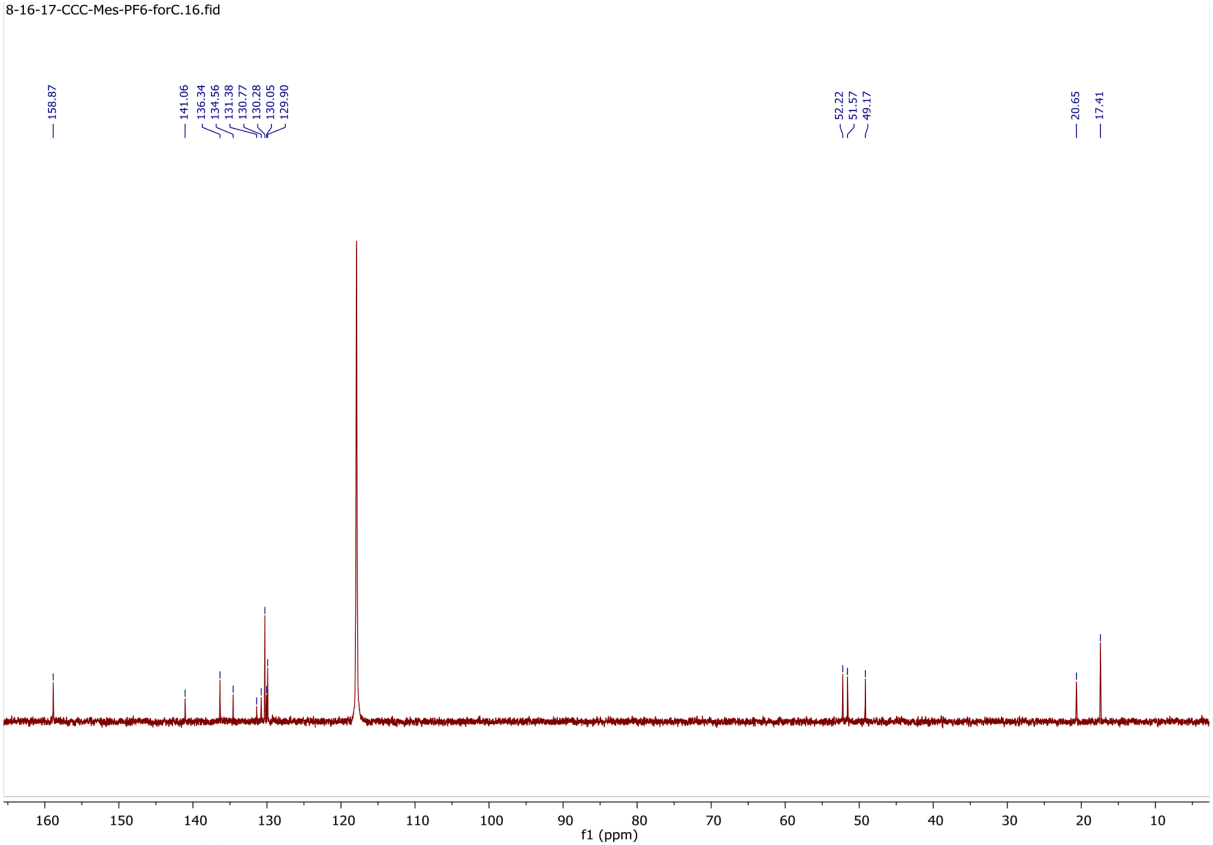

Figure S6. The ^{13}C NMR spectrum of compound **[4a][PF$_6$]**

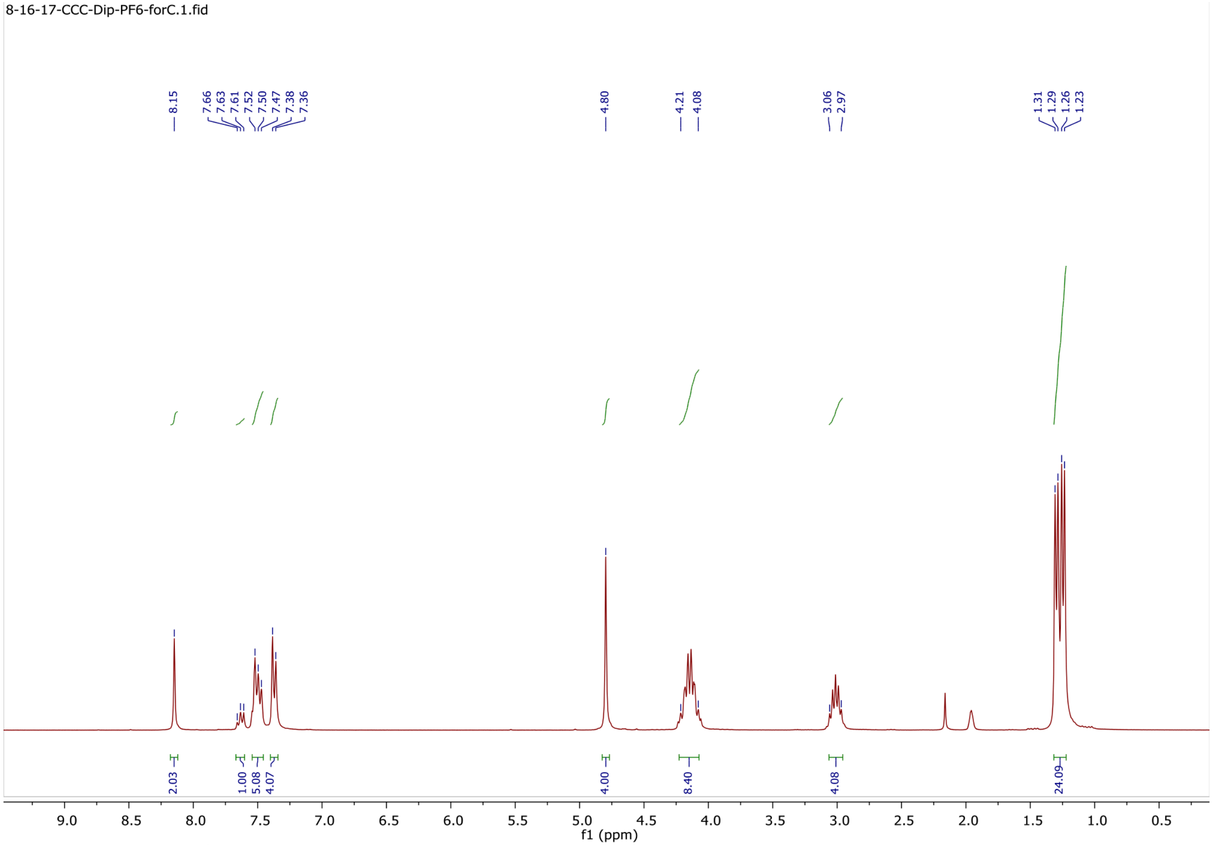

Figure S7. The ^{1}H NMR spectrum of compound **[4b][PF$_6$]**.

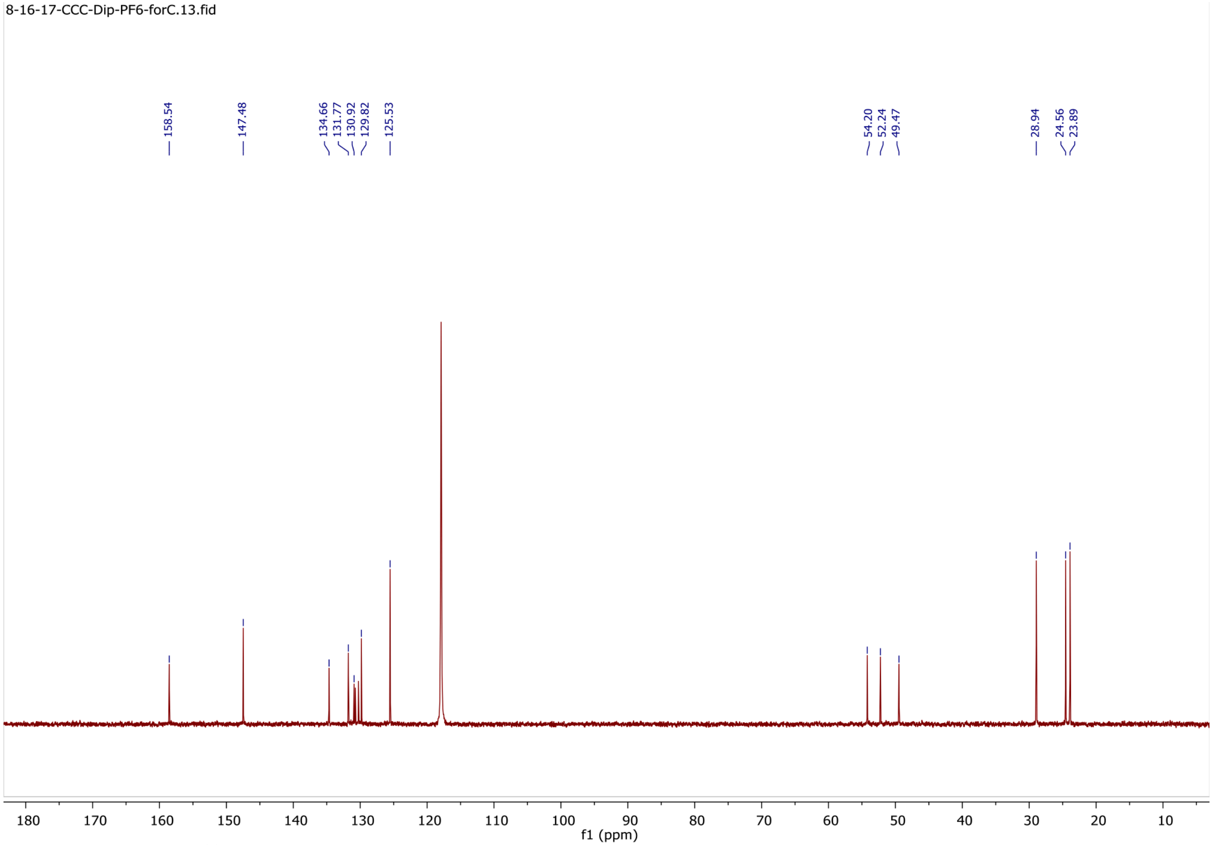

Figure S8. The ^{13}C NMR spectrum of compound **[4b][PF$_6$].**

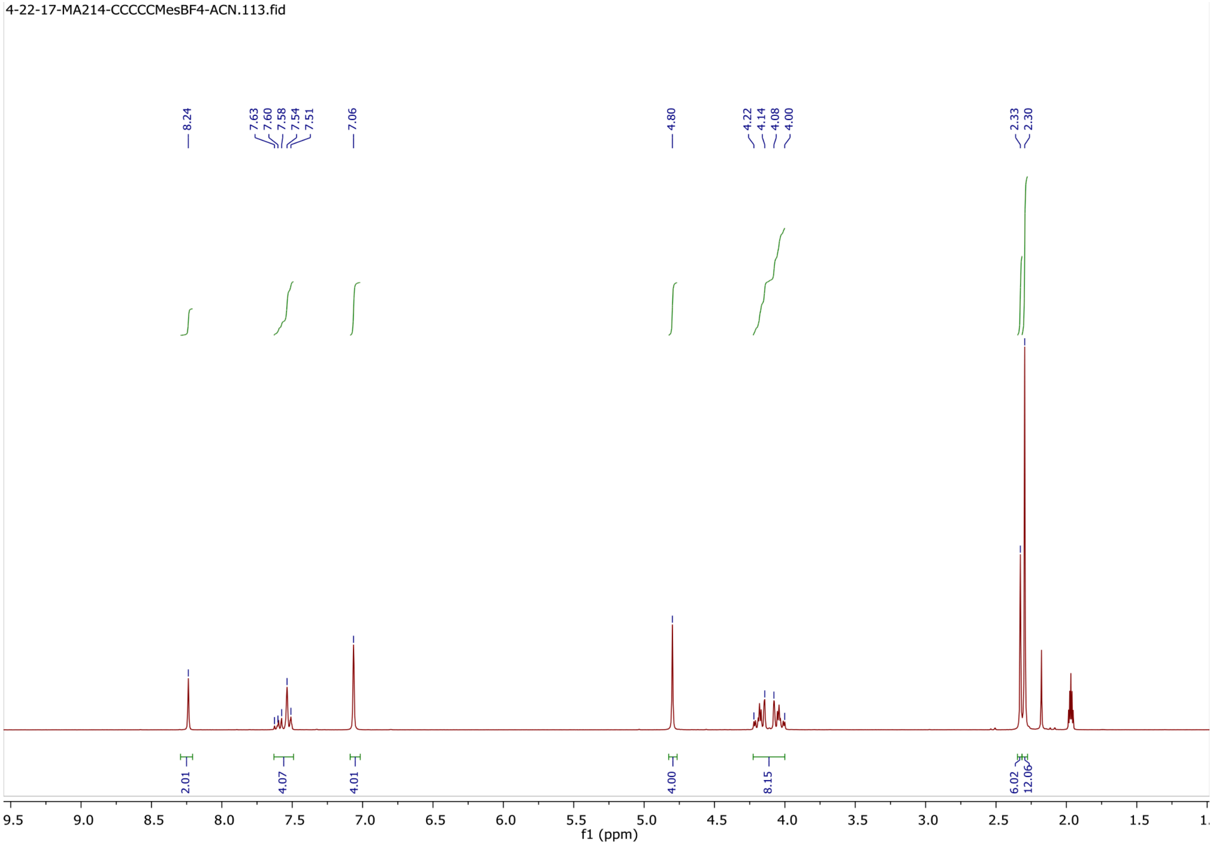

Figure S9. The ^{1}H NMR spectrum of compound **[4a][BF$_4$]**.

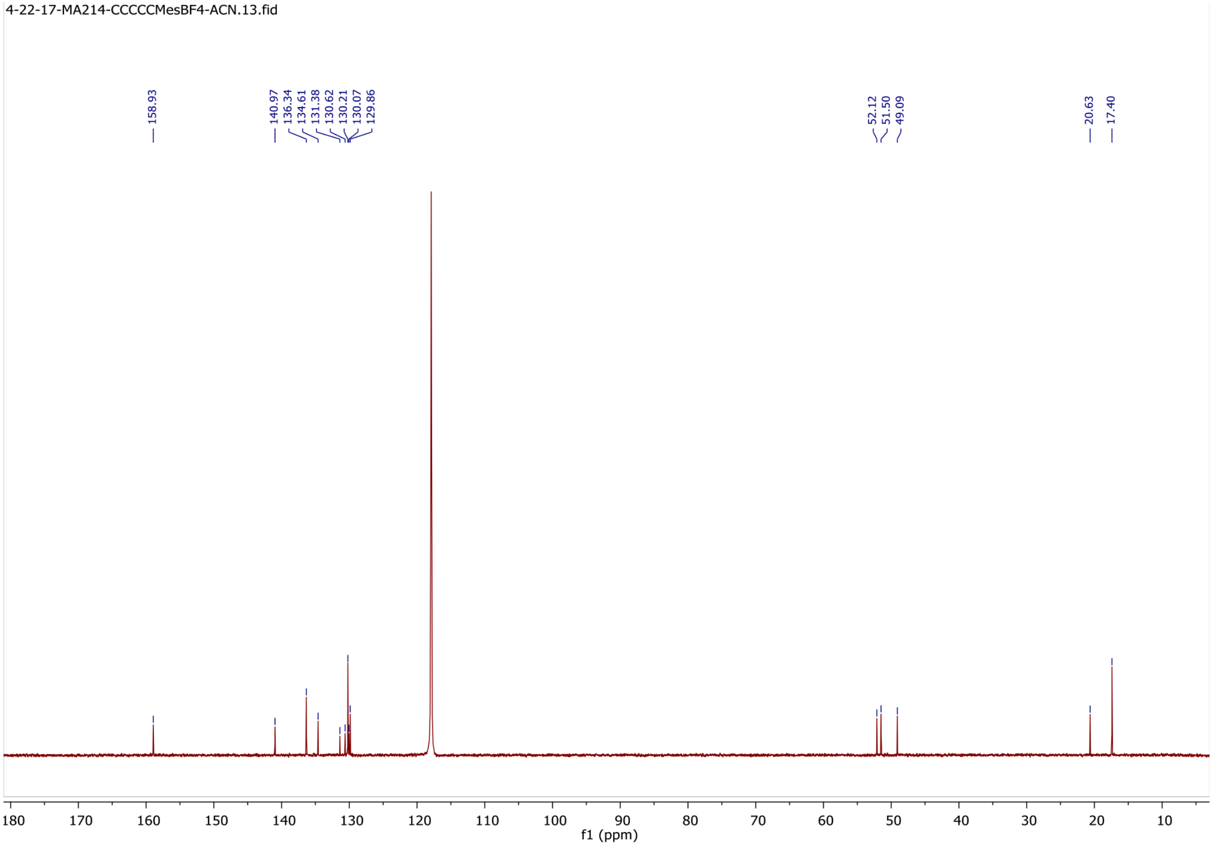

Figure S10. The ^{13}C NMR spectrum of compound **[4a][BF$_4$]**.

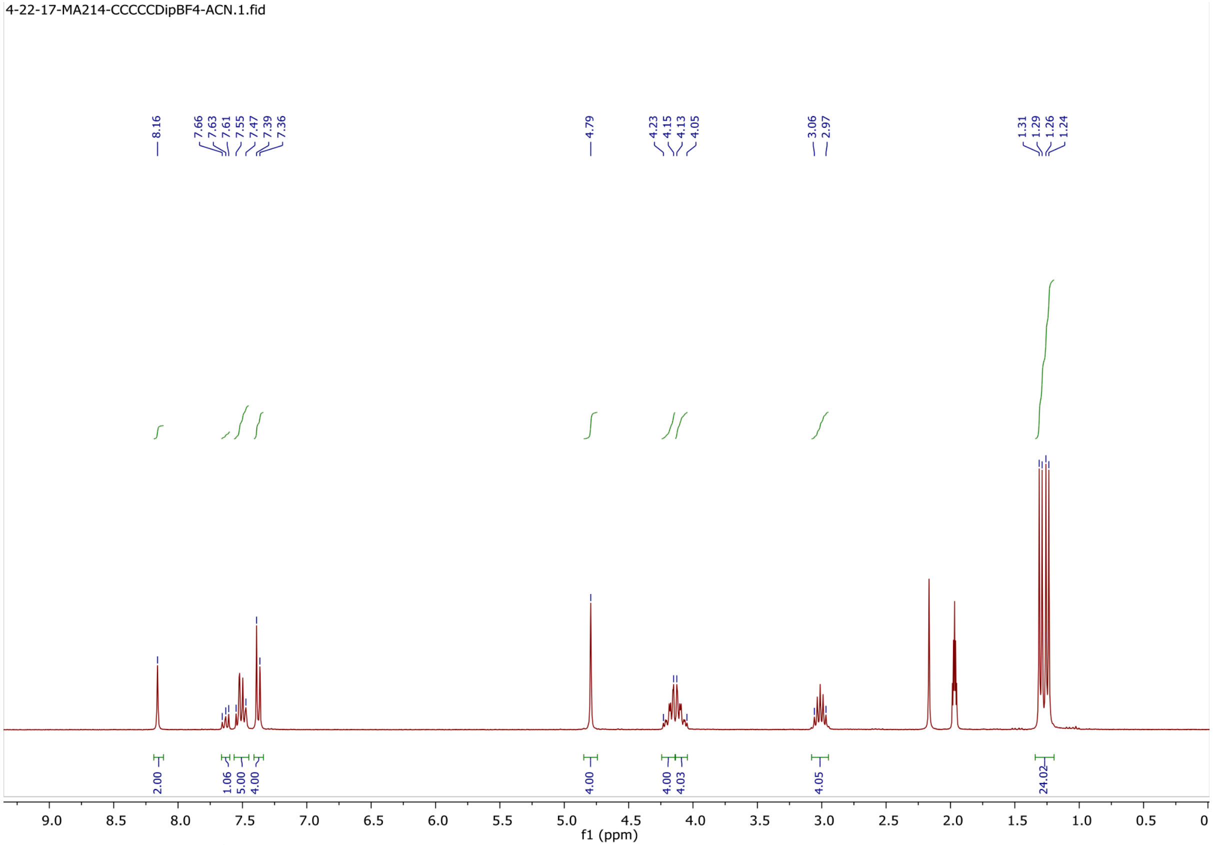

Figure S11. The ^{1}H NMR spectrum of compound **[4b][BF$_4$]**.

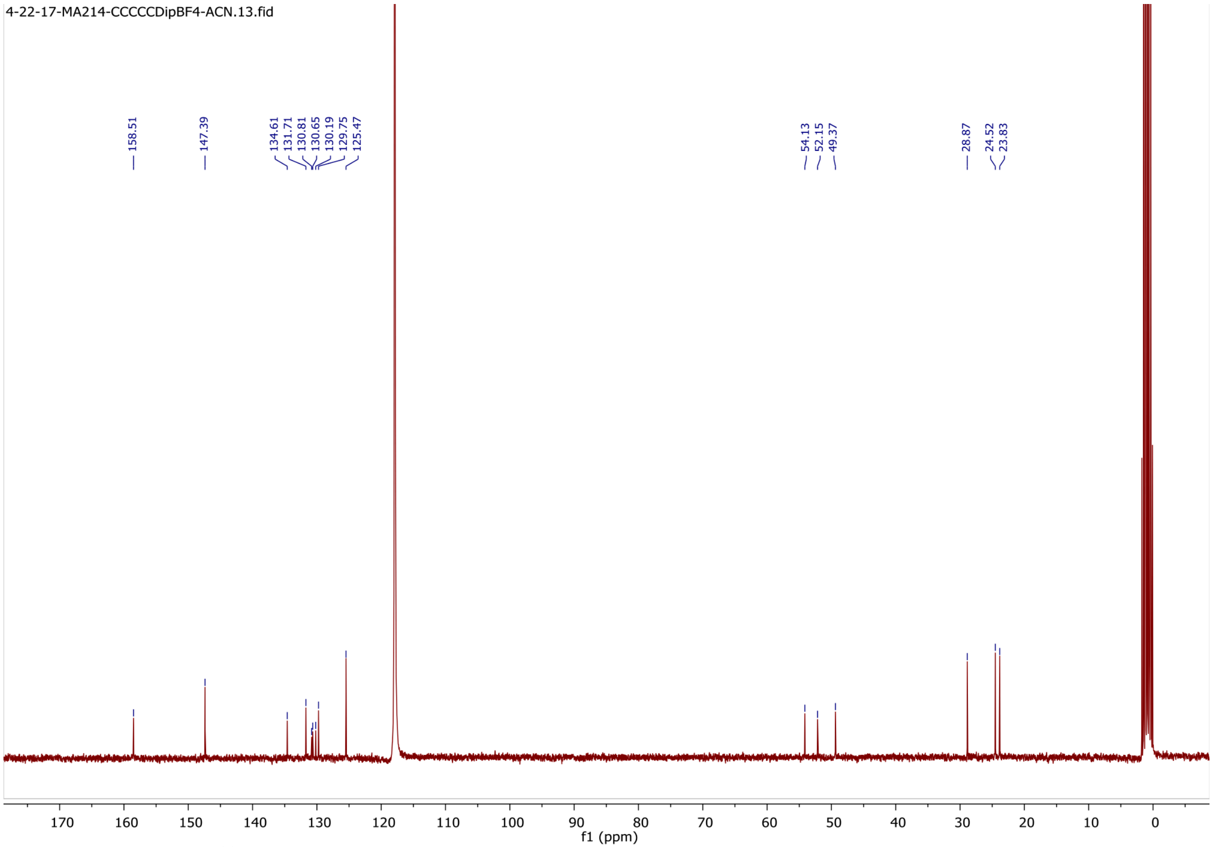

Figure S12. The ^{13}C NMR spectrum of compound **[4b][BF$_4$]**.

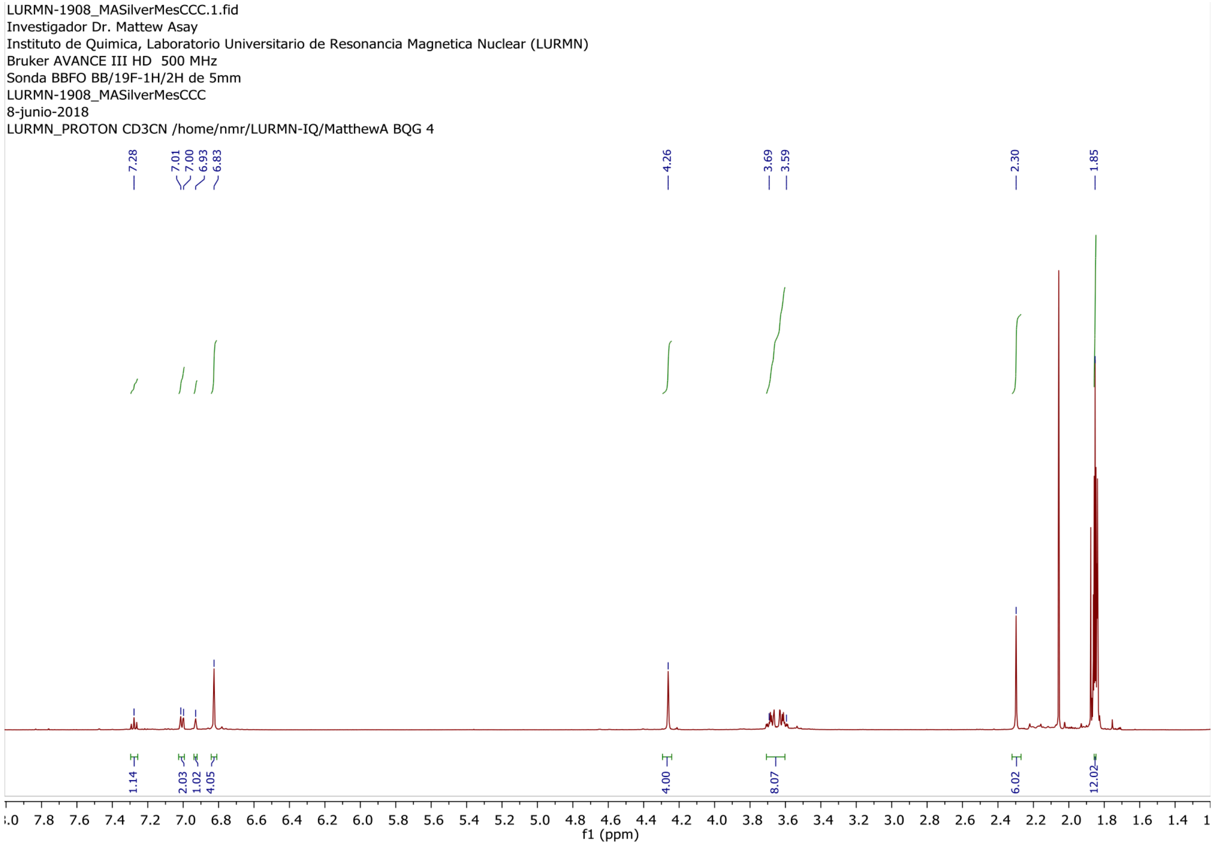

Figure S13. The ^{1}H NMR spectrum of compound **[5a][PF$_6$]**.

Salt **[4a][PF$_6$]** (0.105 g, 0.126 mmol), Ag_2O (0.044 g, 0.189 mmol) in acetonitrile (8 mL) gave **[5a][PF$_6$]** as a white solid. Yield: 0.090 g, (97%). m.p. = 115.1–115.4°C (Decomp.). ^{1}H NMR (500 MHz, Acetonitrile-*d$_3$*, 25°C) δ 1.85 (s, 12H, C*H*$_3$ *o-mesityl*), 2.30 (s, 6H, C*H*$_3$ *p-mesityl*), 3.59 – 3.72 (m, 8H, C*H*$_2$ *imid*), 4.26 (s, 4H, C*H*$_2$ *linker*), 6.83 (s, 4H, C*H m-mesityl*), 6.93 (s, 1H, C*H 1-phenyl*) 7.01 (d, $^{3}J_{\mathrm{HH}}$ = 7.7 Hz, 2H, C*H 3,5-phenyl*), 7.28 (t, $^{3}J_{\mathrm{HH}}$ = 7.6 Hz, 1H, C*H 4-phenyl*). ^{13}C[^{1}H] NMR (126 MHz, Acetonitrile-*d$_3$*, 25°C) δ 17.47 (*C*H$_3$ *o-mesityl*), 20.79 (*C*H$_3$ *p-mesityl*), 50.27 (*C*H$_2$ *imid*), 51.74 (*C*H$_2$ *imid*), 53.28 (*C*H$_2$ *linker*), 126.00 (*C*H *4-phenyl*), 126.33 (*C*H *3,5-phenyl*), 129.93 (*C*H *m-mesityl*), 135.98 (*C*H *1-phenyl*), 136.35 (*C o-mesityl*), 138.23 (*C p-mesityl*), 138.94 (*C ipso-mesityl*), 206.6 (dd, $^{1}J_{\mathrm{CAg}}$ = 194 Hz, $^{1}J_{\mathrm{CAg}}$ = 169 Hz, *C* carbene). FAB^{+} (Acetonitrile) m/z: [M-PF$_6$]$^{+}$ = 585.2158 (calc'd = 585.2147).

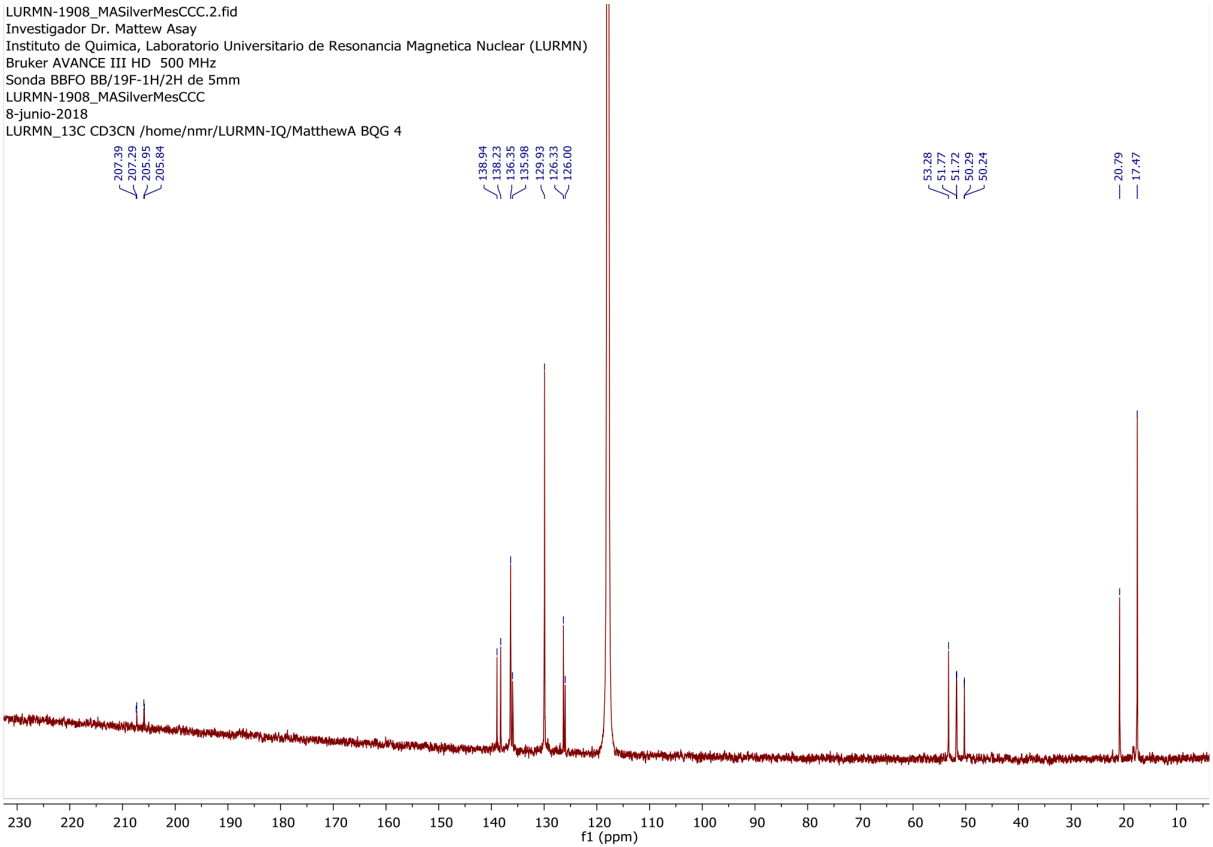

Figure S14. The ^{13}C NMR spectrum of compound **[5a][PF$_6$]**.

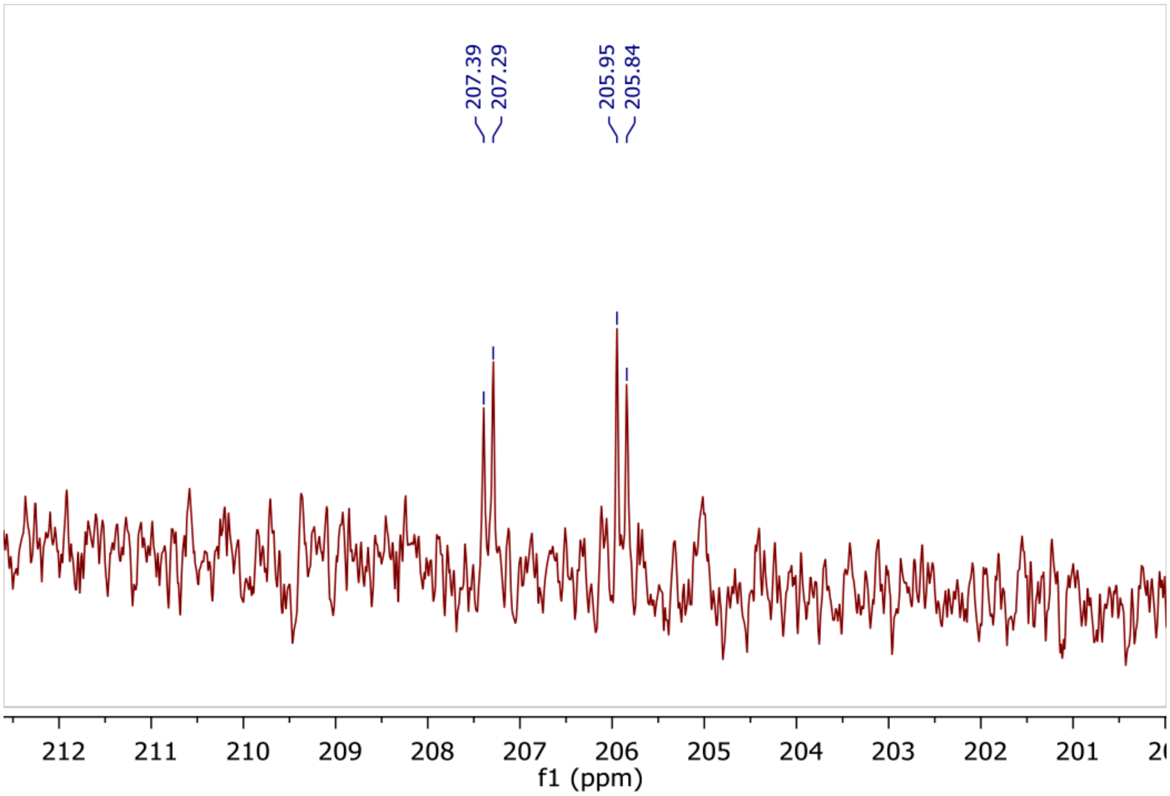

Figure S15. Expanded view of the ^{13}C NMR spectrum of compound **[5a][PF$_6$]** showing the C$_{\text{carbene}}$-Ag coupling to both ^{107}Ag and ^{109}Ag.

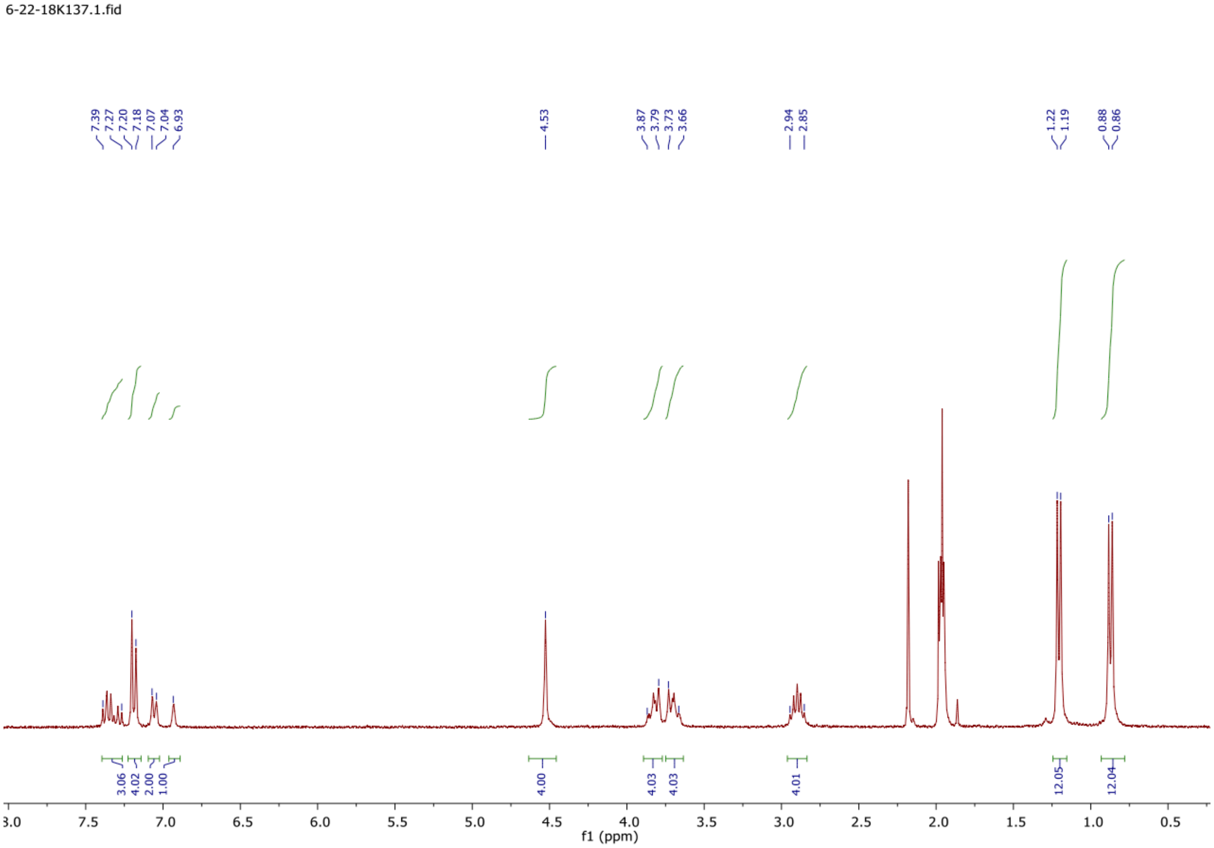

Figure S16. The ^{1}H NMR spectrum of compound **[5b][PF$_6$]**.

Salt **[4b][PF$_6$]** (0.110 g, 0.128 mmol), Ag_2O (0.045 g, 0.193 mmol) in acetonitrile (8 mL) gave **[5b][PF$_6$]** as a white solid. Yield: 0.105 g, (95%).m.p. = 122.4–122.7°C (decomp.). ^{1}H NMR (300 MHz, Acetonitrile-d_3, 25°C): δ 0.87 (d, $^3J_{HH}$ = 6.8 Hz, 12H, CH_3 *iPr*), 1.210 (d, $^3J_{HH}$ = 6.8 Hz, 12H, CH_3 *iPr*), 2.90 (m, 4H, C*H* *iPr*), 3.66–3.73 (m, 4H, CH_2 *imid*), 3.79–3.87 (m, 4H, CH_2 *imid*), 4.53 (s, 4H, CH_2 *linker*), 6.93 (s, 1H, C*H* *1-phenyl*), 7.06 (d, *J* = 8.7 Hz. 2H, C*H* *3,5-phenyl*), 7.19 (d, $^3J_{HH}$ = 7.8 Hz, 4H, C*H* *m-Dipp*), 7.27–7.39 (m, 3H, C*H* *p-Dipp* and C*H* *4-phenyl*). ^{13}C[^{1}H] NMR (76 MHz, Acetonitrile-d_3, 25°C) δ 23.42 (*C*H_3 *iPr*), 25.45 (*C*H_3 *iPr*), 28.55 (*C*H *iPr*), 49.52 (*C*H_2 *imid*), 53.65 (*C*H_2 *linker*), 54.95 (*C*H_2 *imid*), 124.75 (*C*H *4-phenyl*), 125.05 (*C*H *m-Dipp*), 125.96 (*C*H *3,5-phenyl*), 130.07 (*C*H *1-phenyl*), 130.40 (*C* *2,6-phenyl*), 135.09 (*C*H *p-Dipp*), 137.38 (*C* *o-Dipp*), 147.45 (*C* *ipso-Dipp*), 205.60 (dd, $^1J_{CAg}$ = 171 Hz, $^1J_{CAg}$ = 197 Hz, *C* *carbene*). FAB$^+$ m/z [M-PF$_6$]$^+$ = 671.3078 (calc’d = 671.3081).

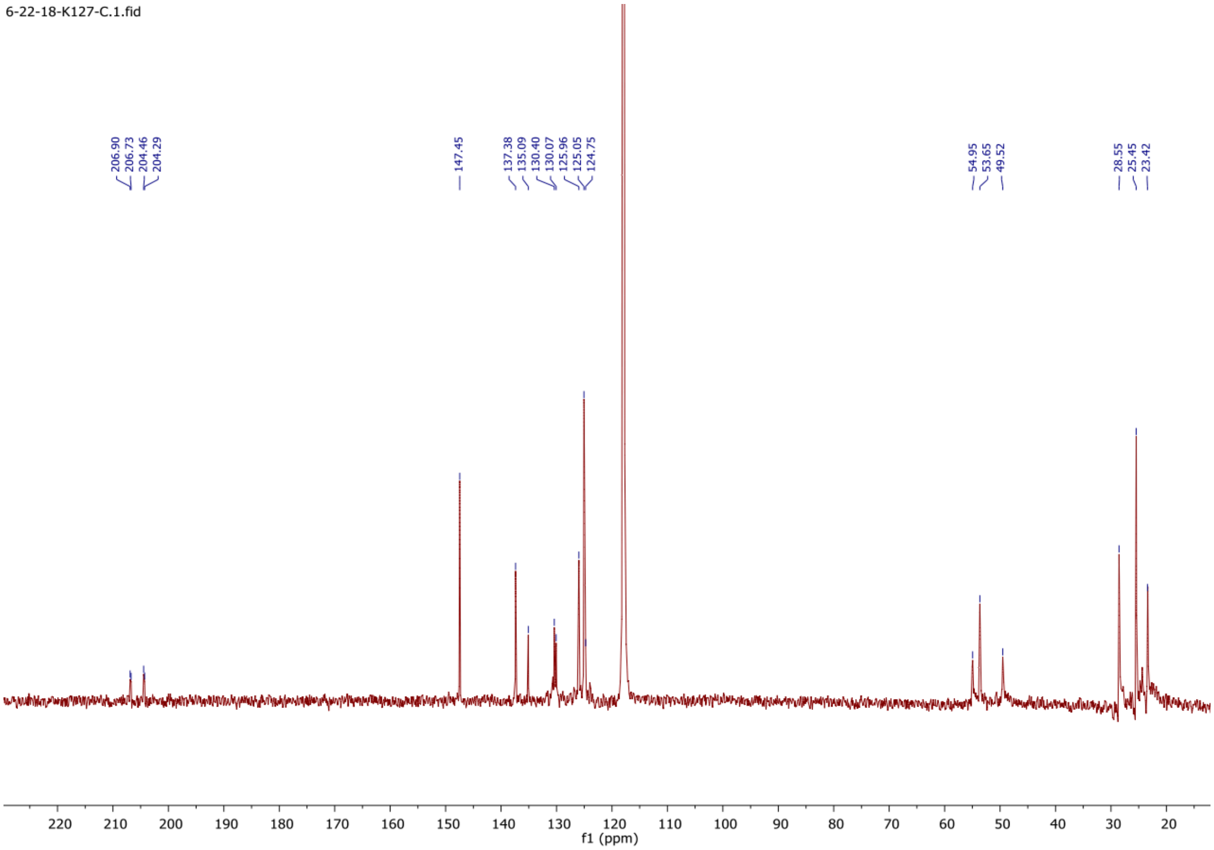

Figure S17. The ^{13}C NMR spectrum of compound **[5b][PF$_6$]**.

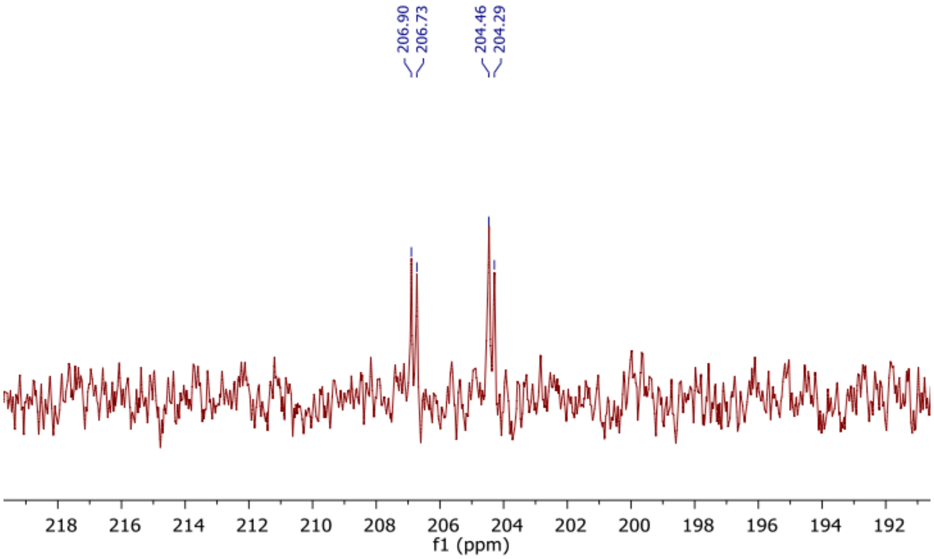

Figure S18. Expanded view of the ^{13}C NMR spectrum of compound **[5b][PF$_6$]** showing the C$_{carbene}$-Ag coupling to both ^{107}Ag and ^{109}Ag.

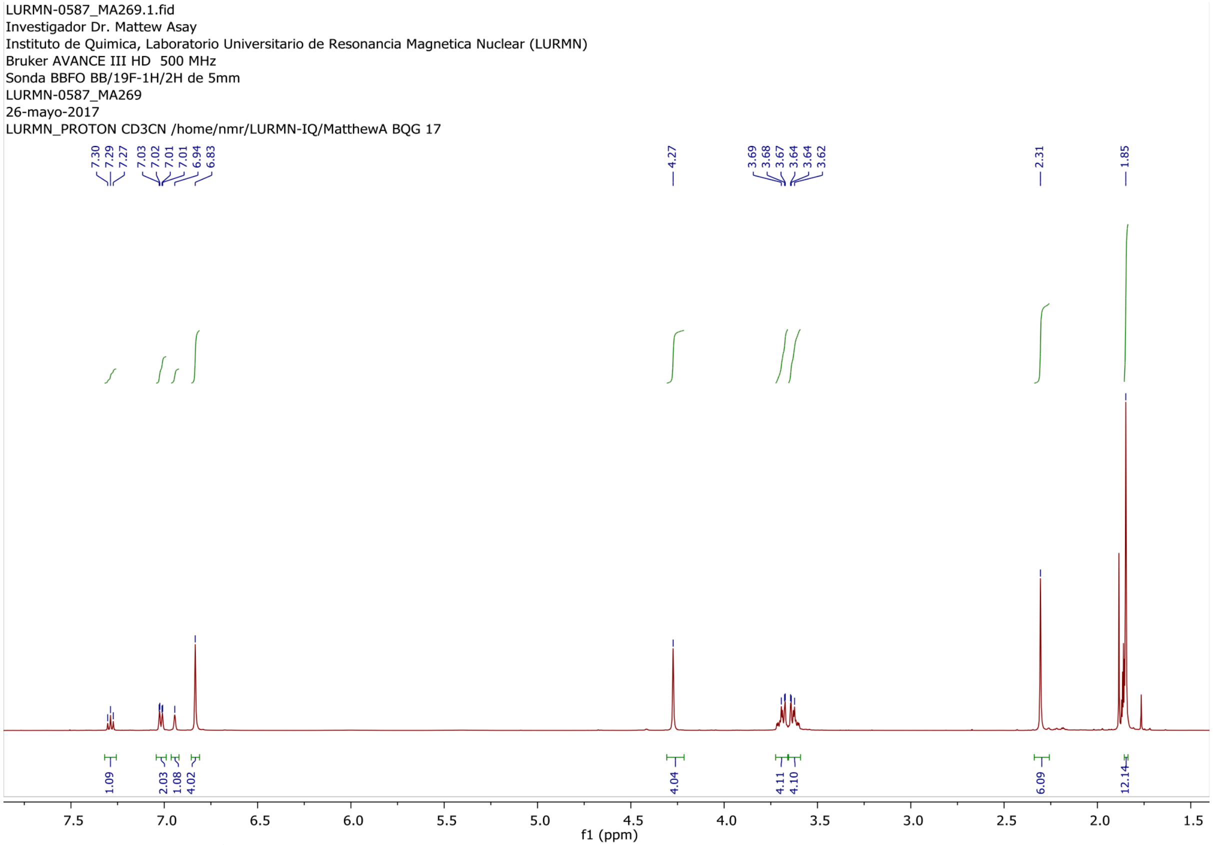

Figure S19. The ^{1}H NMR spectrum of compound **[5a][BF$_4$]**.

Salt **[4a][BF$_4$]** (0.126 g, 0.193 mmol), Ag_2O (0.068 g, 0.289 mmol) in acetonitrile (8 mL) gave **[5a][BF$_4$]** as a white solid. Yield: 0.125 g, (96%). m.p. = 112.0–112.6°C (Decomp.). ^{1}H NMR (500 MHz, Acetonitrile-*d$_3$*, 25°C) δ 1.85 (s, 12H, C*H*$_3$ *o-mesityl*), 2.31 (s, 6H, C*H*$_3$ *p-mesityl*), 3.59 – 3.64 (m, 4H, C*H*$_2$ *imid*), 3.67–3.72 (m, 4H, C*H*$_2$ *imid*), 4.27 (s, 4H, C*H*$_2$ *linker*), 6.83 (s, 4H, C*H m-mesityl*), 6.94 (s, 1H, C*H 1-phenyl*) 7.02 (d, $^{3}J_{HH}$ = 7.7 Hz, 2H, C*H 3,5-phenyl*), 7.29 (t, $^{3}J_{HH}$ = 7.6 Hz, 1H, C*H 4-phenyl*). ^{13}C[^{1}H] NMR (126 MHz, Acetonitrile-*d$_3$*, 25°C) δ 17.47 (*C*H$_3$ *o-mesityl*), 20.80 (*C*H$_3$ *p-mesityl*), 50.27 (*C*H$_2$ *imid*), 51.74 (*C*H$_2$ *imid*), 53.28 (*C*H$_2$ *linker*), 126.01 (*C*H *4-phenyl*), 126.33 (*C*H *3,5-phenyl*), 129.92 (*C*H *m-mesityl*), 135.98 (*C*H *1-phenyl*), 136.35 (*C o-mesityl*), 138.23 (*C p-mesityl*), 138.92 (*C ipso-mesityl*), 206.6 (dd, $^{1}J_{CAg}$ = 194 Hz, $^{1}J_{CAg}$ = 169 Hz, *C* carbene). FAB^{+} (Acetonitrile) m/z: [M-BF$_4$]$^{+}$ = 585.2147 (calc'd = 585.2147).

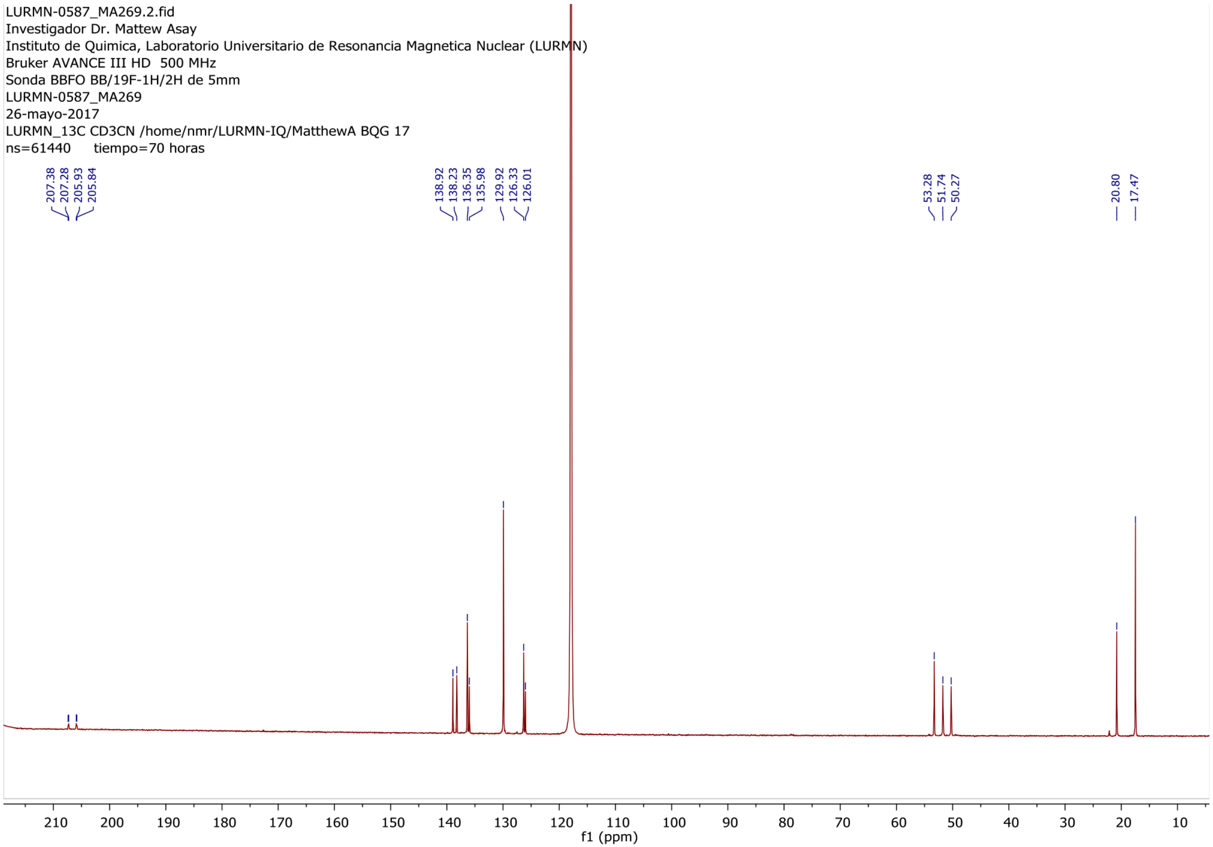

Figure S20. The ^{13}C NMR spectrum of compound **[5a][BF$_4$]**

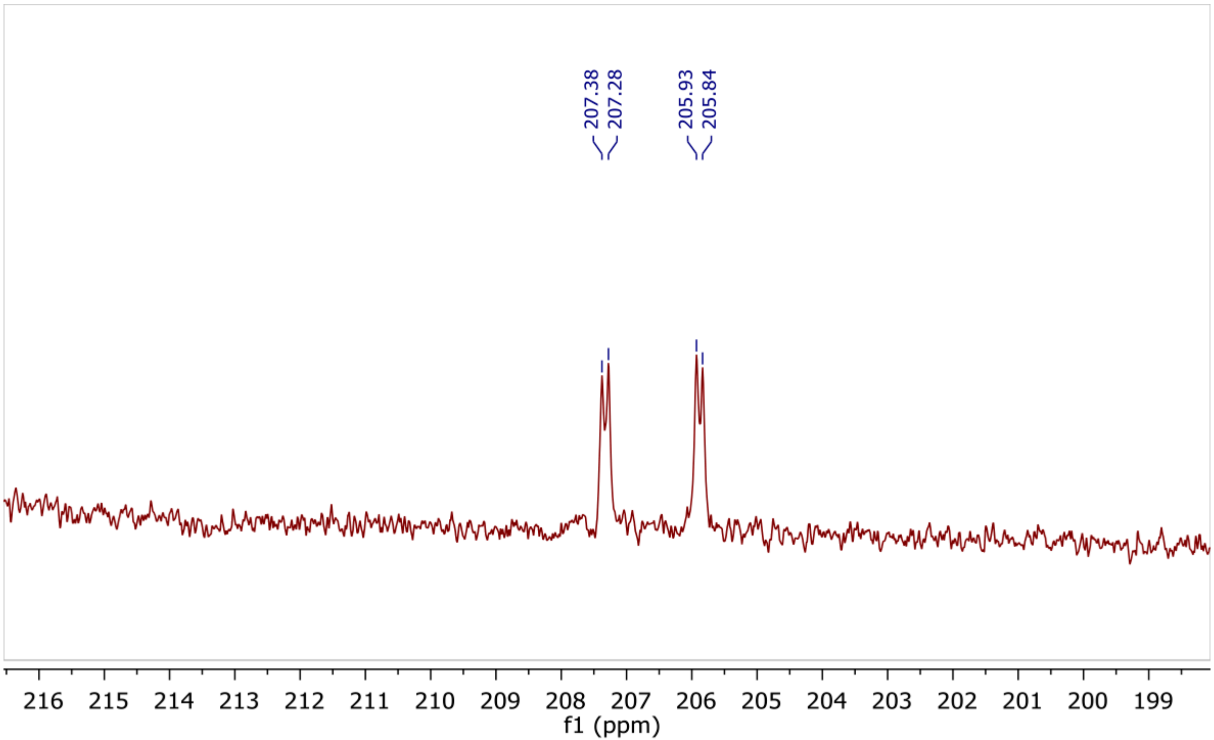

Figure S21. Expanded view of the ^{13}C NMR spectrum of compound **[5a][BF$_4$]** to show the C$_{carbene}$- Ag coupling to both ^{107}Ag and ^{109}Ag.

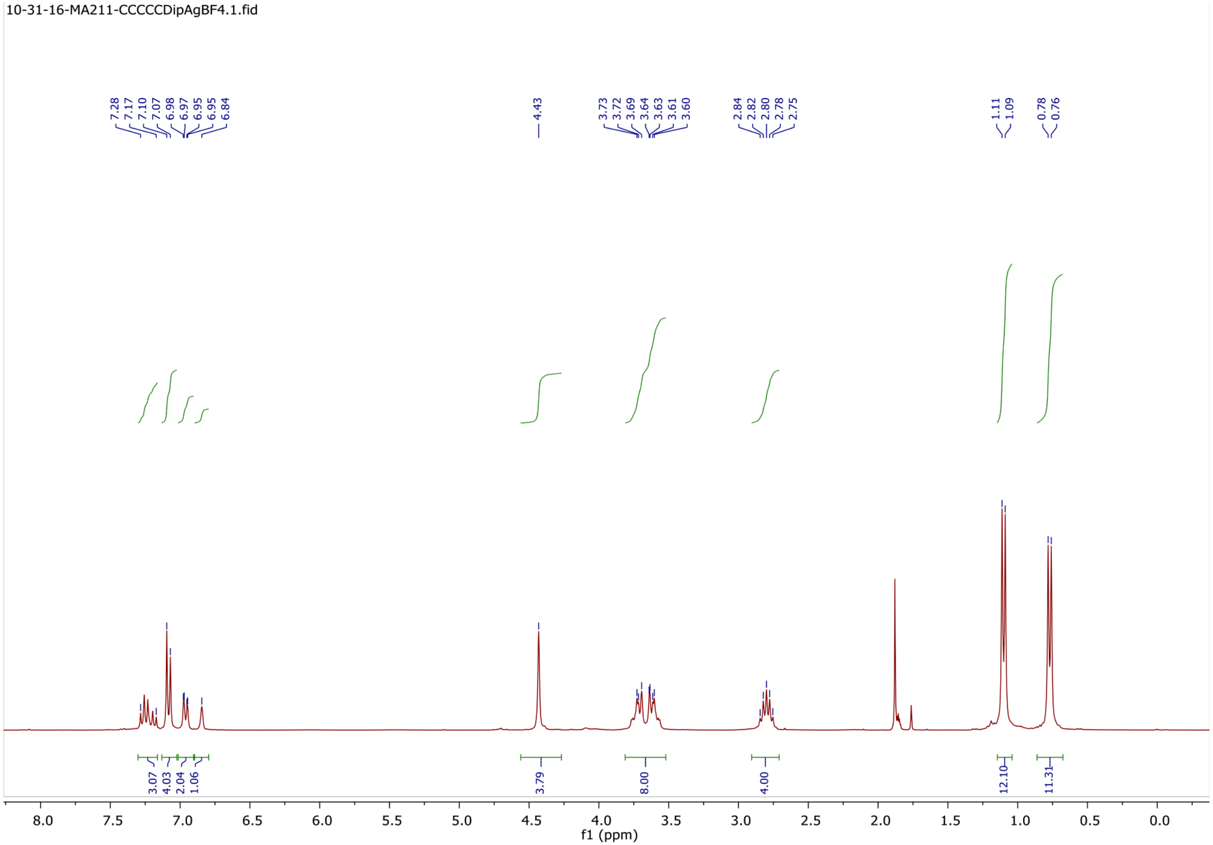

Figure S22. The ^{1}H NMR spectrum of compound **[5b][BF$_4$]**.

Salt **[4b][BF$_4$]** (0.300g, 0.461 mmol), Ag$_2$O (0.141g, 0.608 mmol) in acetonitrile (8 mL) gave **[5b][BF$_4$]** as a white solid. Yield: 0.287 g, (93%). m.p. = 118.3–118.5°C (decomp.). ^{1}H NMR (300 MHz, Acetonitrile-d_3, 25°C): δ 0.77 (d, $^3J_{\mathrm{HH}}$ =6.8 Hz, 12H, CH_3 *iPr*) 1.10 (d, $^3J_{\mathrm{HH}}$ = 6.8 Hz, 12H, CH_3 *iPr*), 2.78-2.82 (m, 4H, C*H iPr*), 3.60–3.64 (m, 4H, CH_2 *imid*), 3.69–3.73 (m, 4H, CH_2 *imid*), 4.43 (s, 4H, CH_2 *linker*), 6.84 (s, 1H,C*H 1-phenyl*) 6.96 (d, $^3J_{\mathrm{HH}}$ = 7.5 Hz, 2H, C*H 3,5-phenyl*), 7.08 (d, $^3J_{\mathrm{HH}}$ = 7.7 Hz, 4H, C*H m-Dipp*), 7.17–7.28 (m, 3H, C*H p-Dipp* and C*H 4-phenyl*). ^{13}C[^{1}H] NMR (76 MHz, Acetonitrile-d_3, 25°C) δ 23.45 (*C*H$_3$ *iPr*), 25.47 (*C*H$_3$ *iPr*), 28.56 (*C*H *iPr*), 49.58 (*C*H$_2$ *imid*), 53.66 (*C*H$_2$ *linker*), 55.00 (*C*H$_2$ *imid*), 124.80 (*C*H *4-phenyl*), 125.03 (*C*H *m-Dipp*), 125.96 (*C*H *3,5-phenyl*), 130.06 (*C*H *1-phenyl*), 130.37 (*C 2,6-phenyl*), 135.12 (*C*H *p-Dipp*), 137.40 (*C o-Dipp*), 147.47 (*C ipso-Dipp*), 205.68 (dd, $^1J_{\mathrm{CAg}}$ = 171 Hz, $^1J_{\mathrm{CAg}}$ = 197 Hz, *C carbene*). FAB$^+$ m/z [M-BF4]$^+$ = 671.3081 (calc'd = 671.3083).

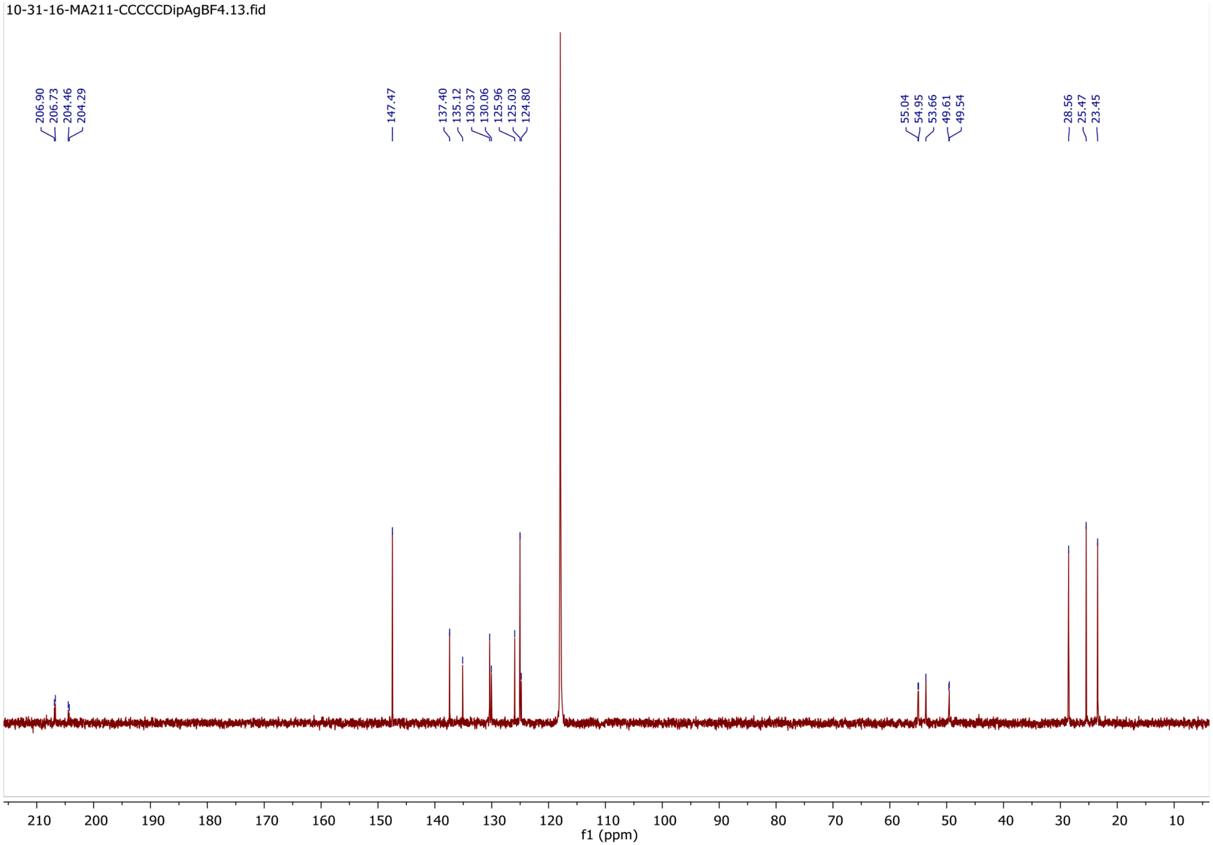

Figure S23. The ^{13}C NMR spectrum of compound **[5b][BF$_4$]**.

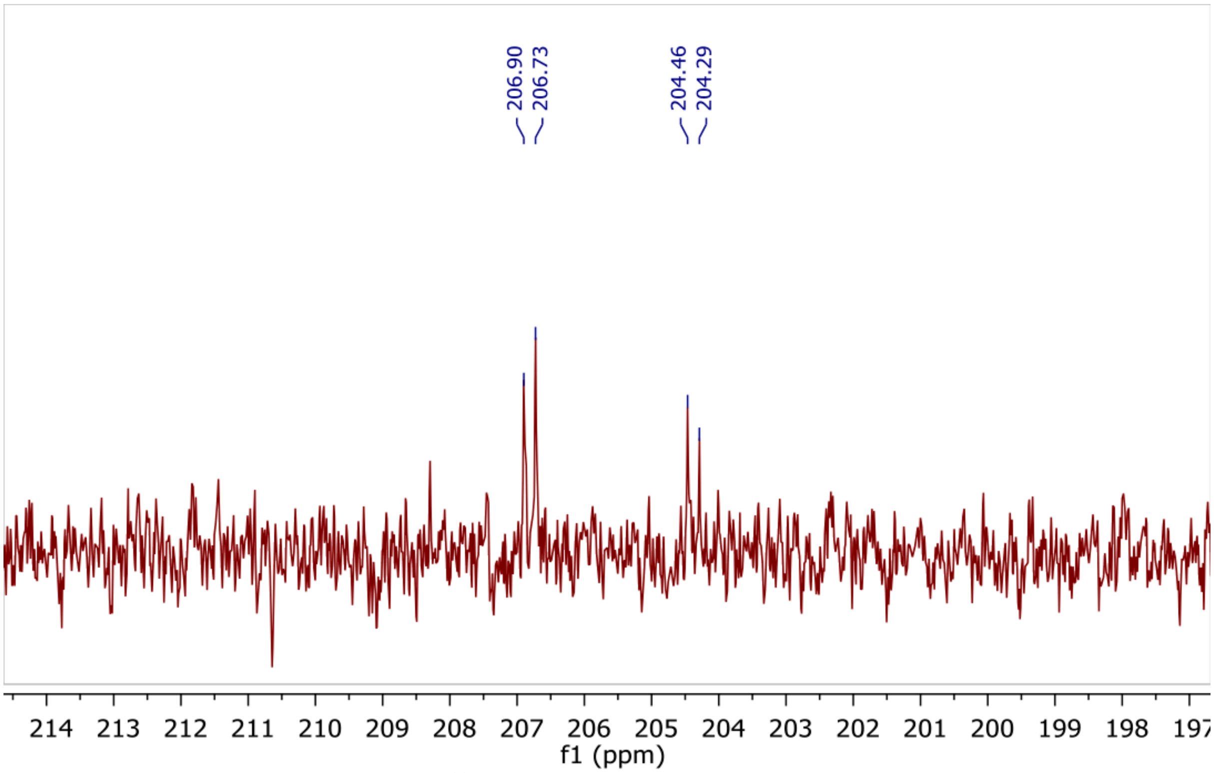

Figure S24. Expanded view of the ^{13}C NMR spectrum of compound **[5b][BF$_4$]** showing the C$_{\text{carbene}}$-Ag coupling to both ^{107}Ag and ^{109}Ag.

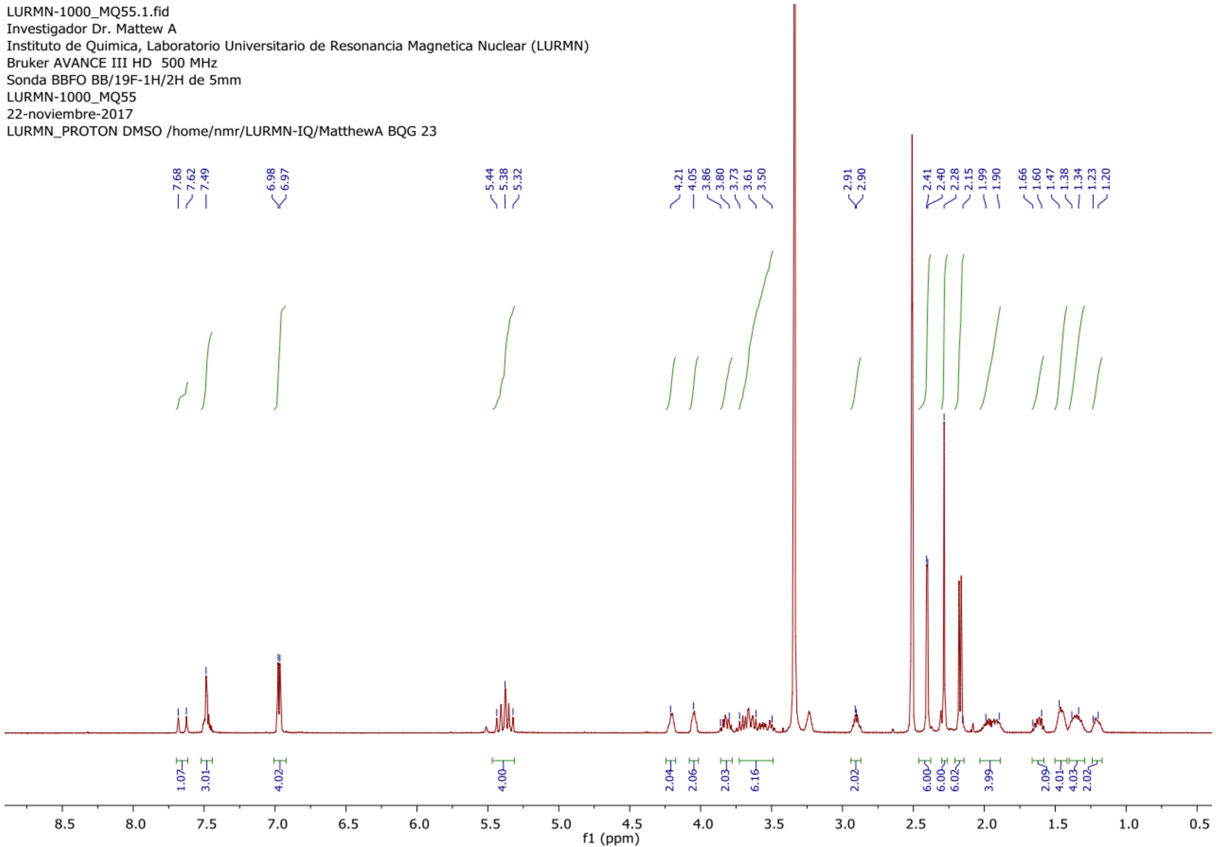

Figure S25. The ^{1}H NMR spectrum of compound **6**.

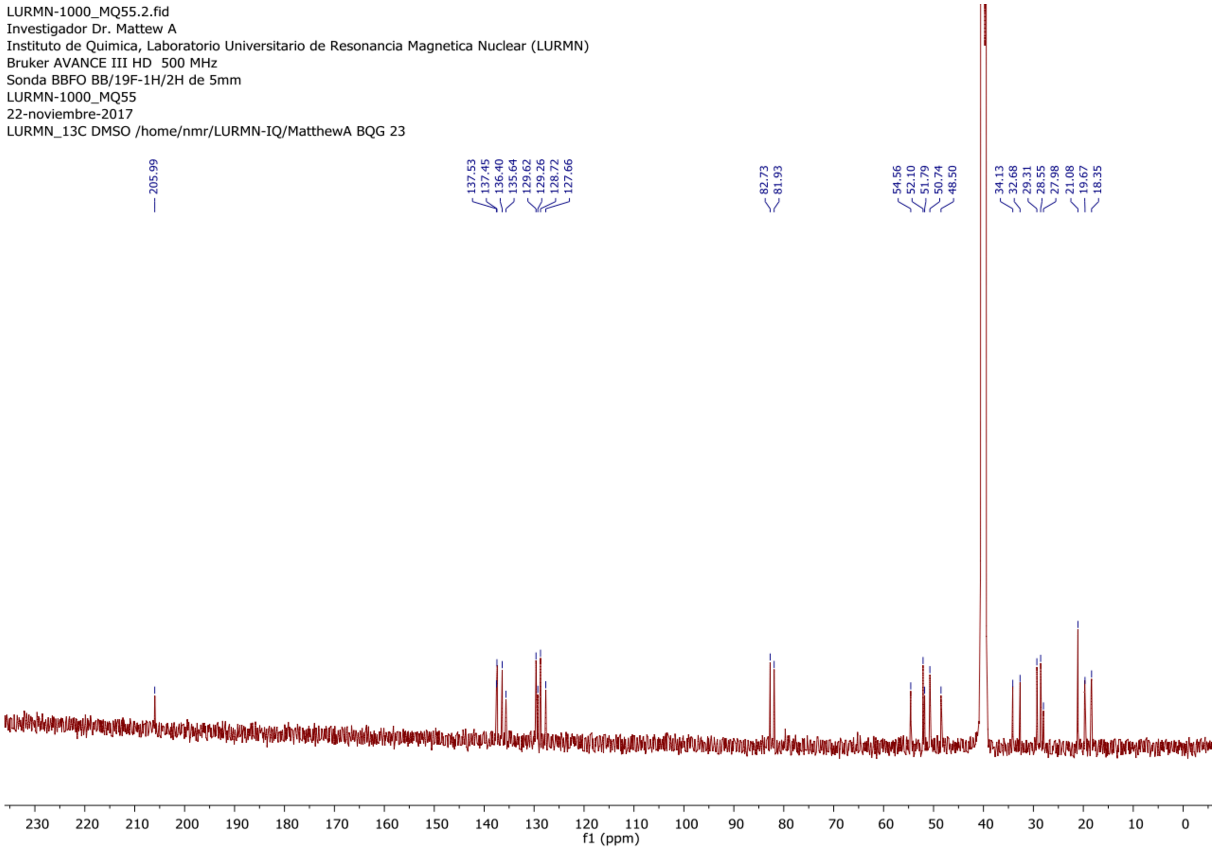

Figure S26. The ^{13}C NMR spectrum of Diiridium complex **6**.

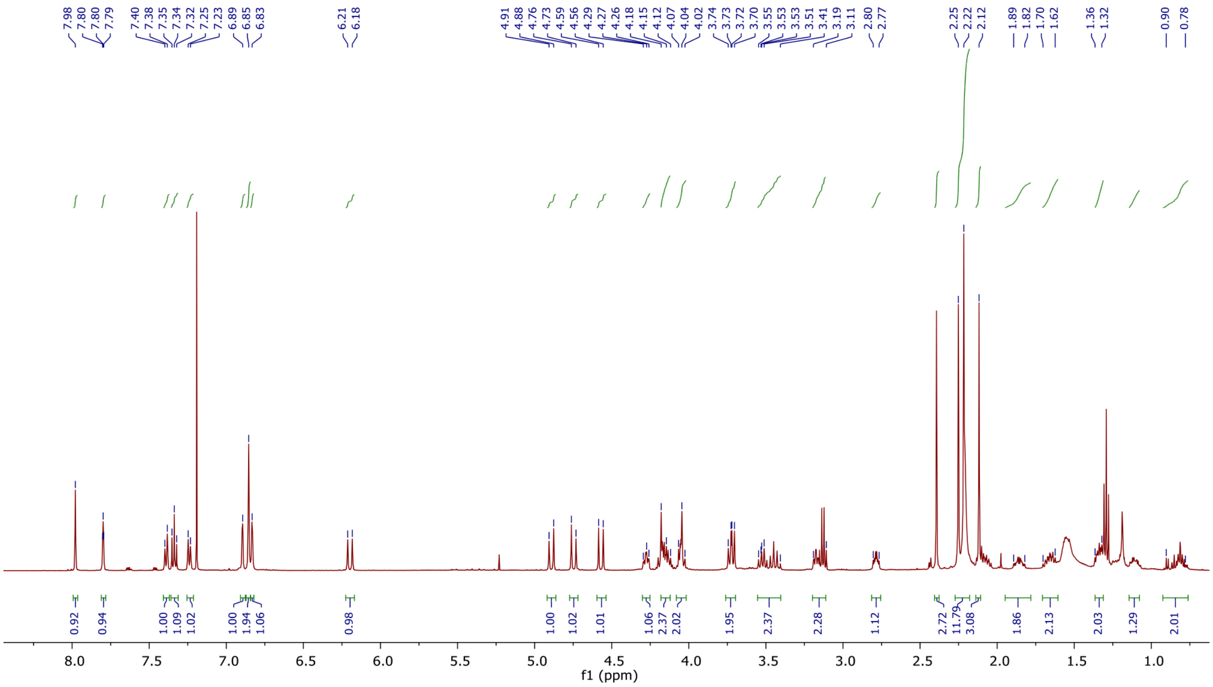

Figure S27. The ^{1}H NMR spectrum of compound **7**.

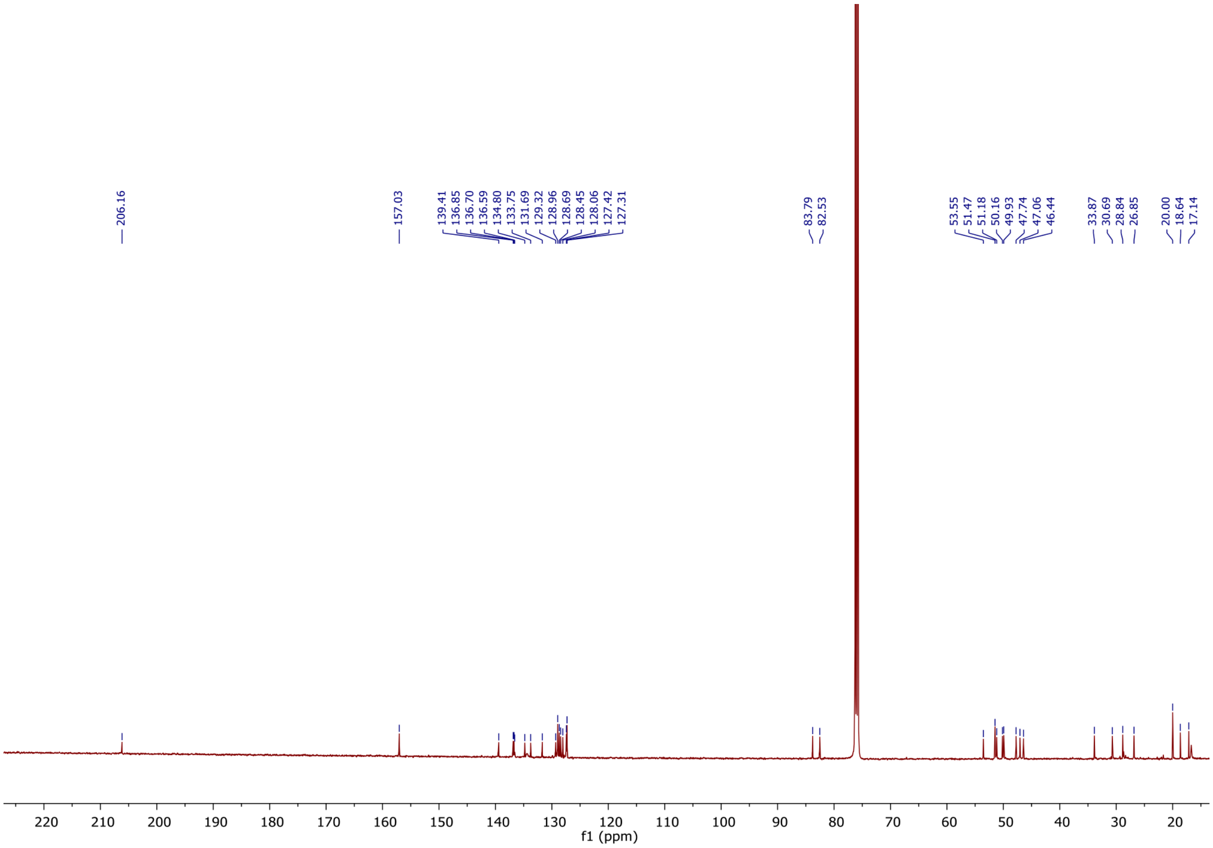

Figure S28. The ^{13}C NMR spectrum of Iridium complex **7**.

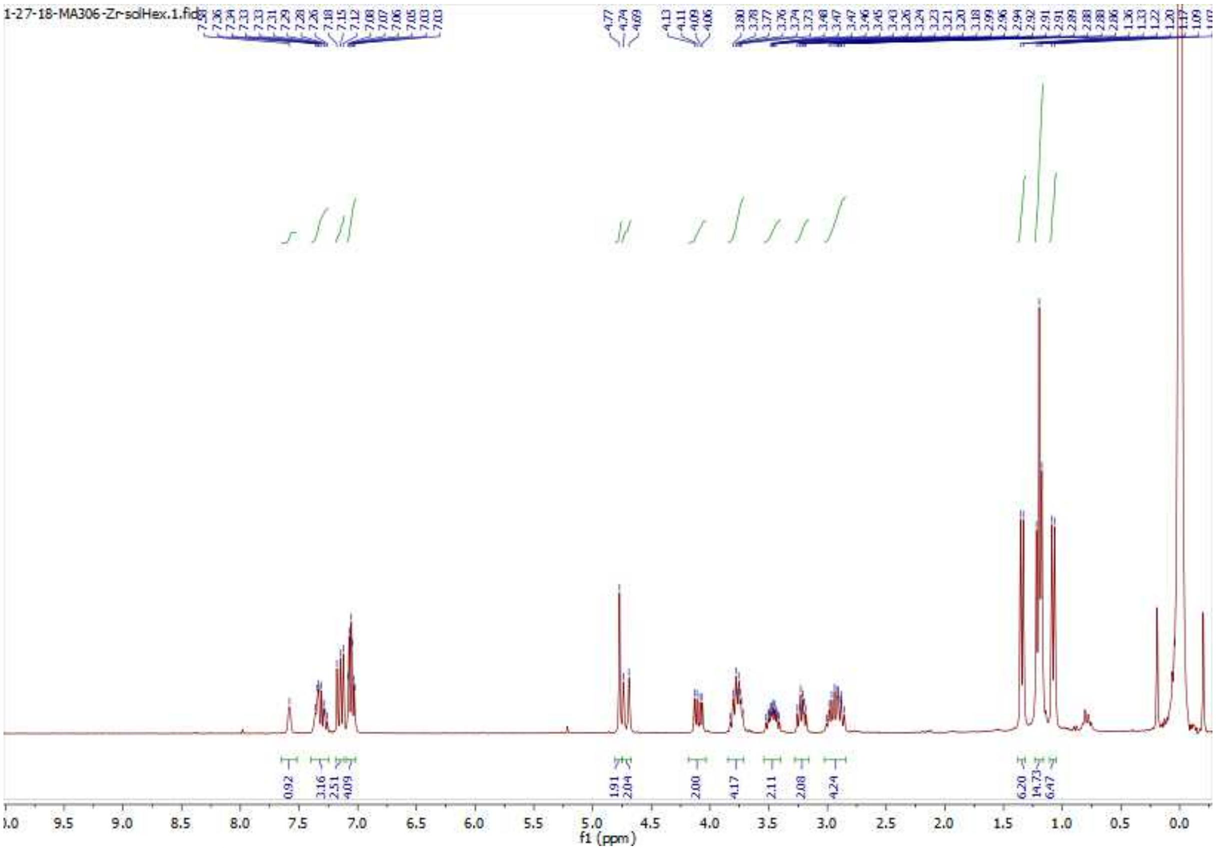

Figure S29. The ^{1}H NMR spectrum of chloroform adduct **8**.

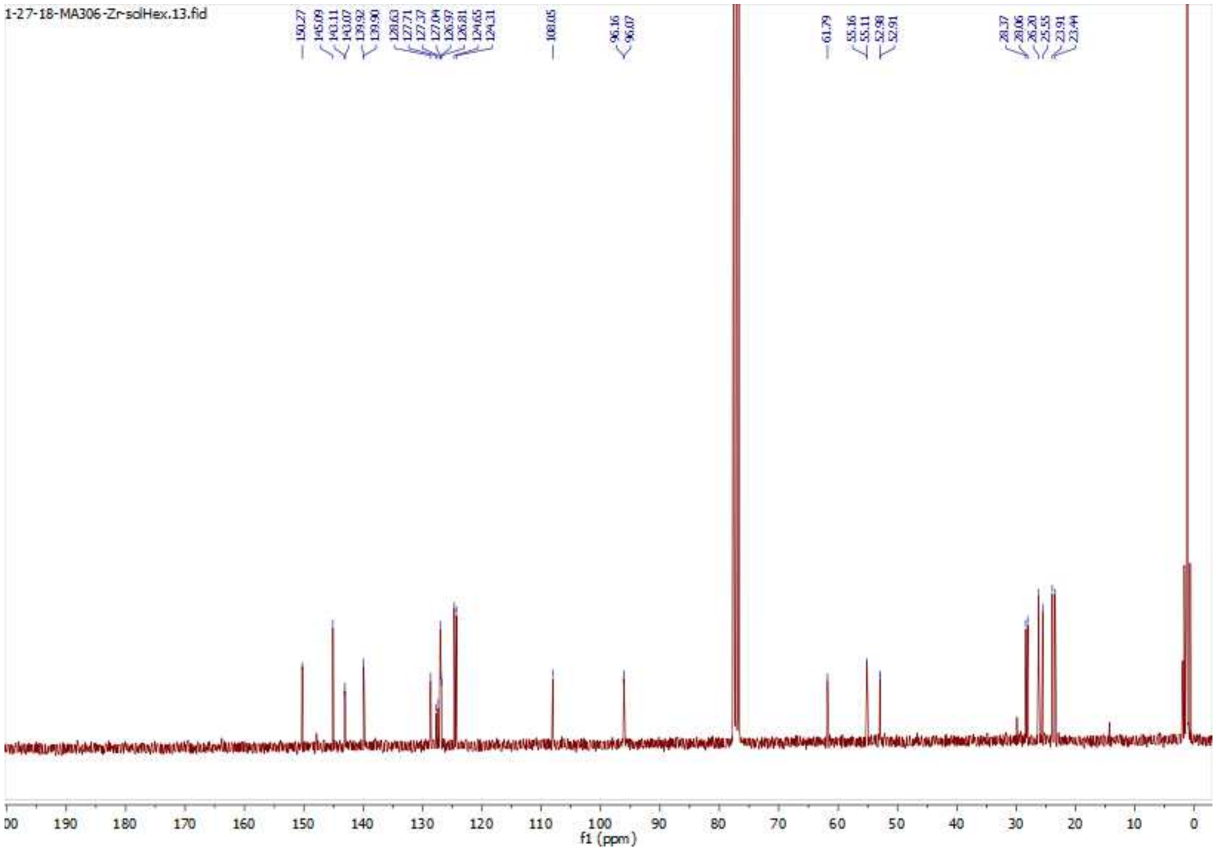

Figure S30. The ^{13}C NMR spectrum of chloroform adduct **8**.

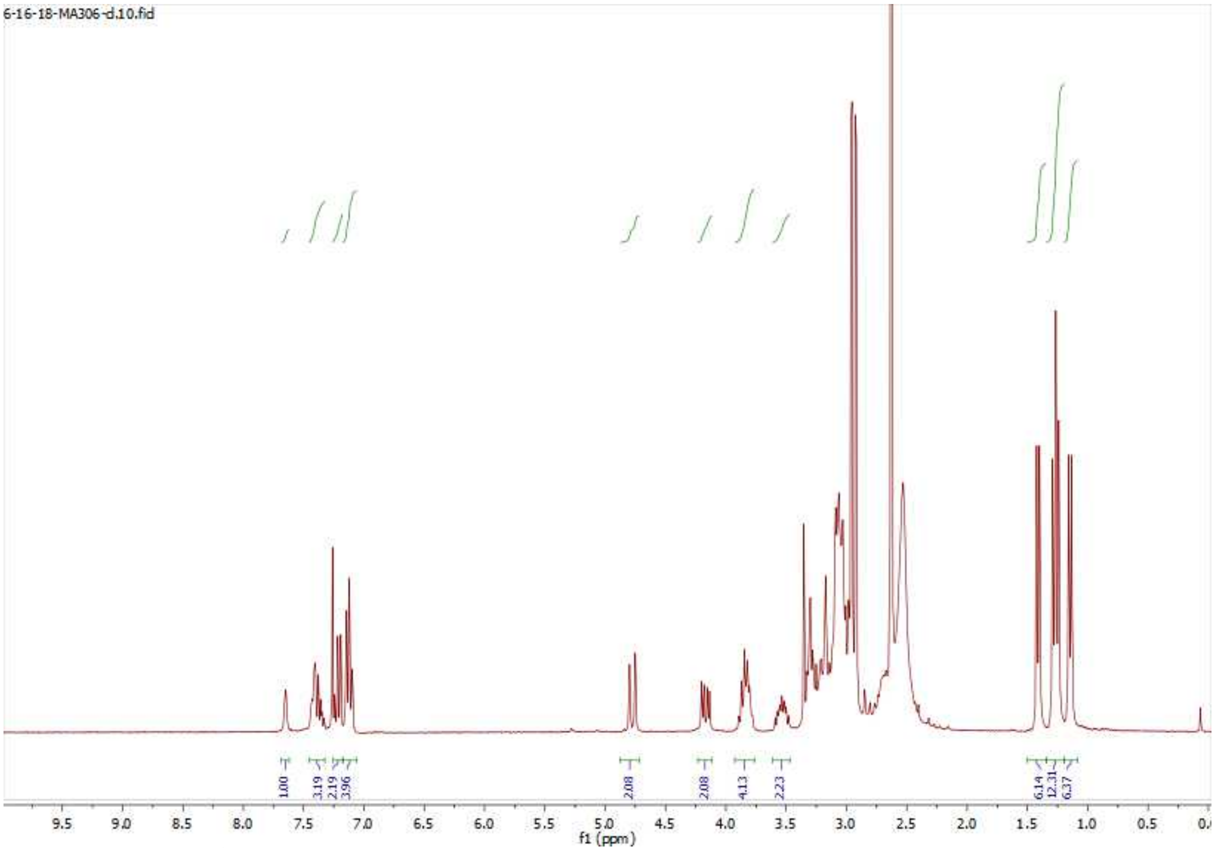

Figure S31. The ^{1}H NMR spectrum of reaction mixture of chloroform adduct **8-d_2**.

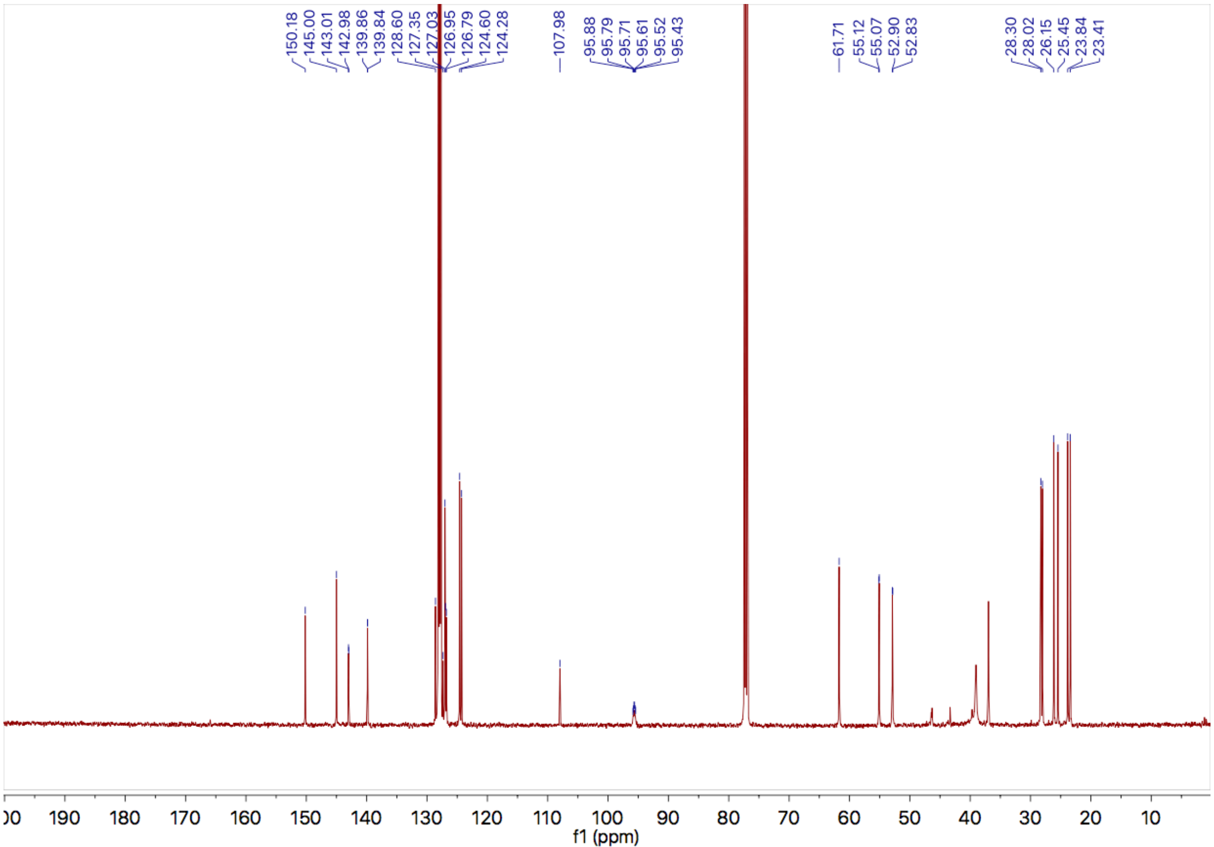

Figure S32. The ^{13}C NMR spectrum of chloroform adduct **8-*d*$_2$**.

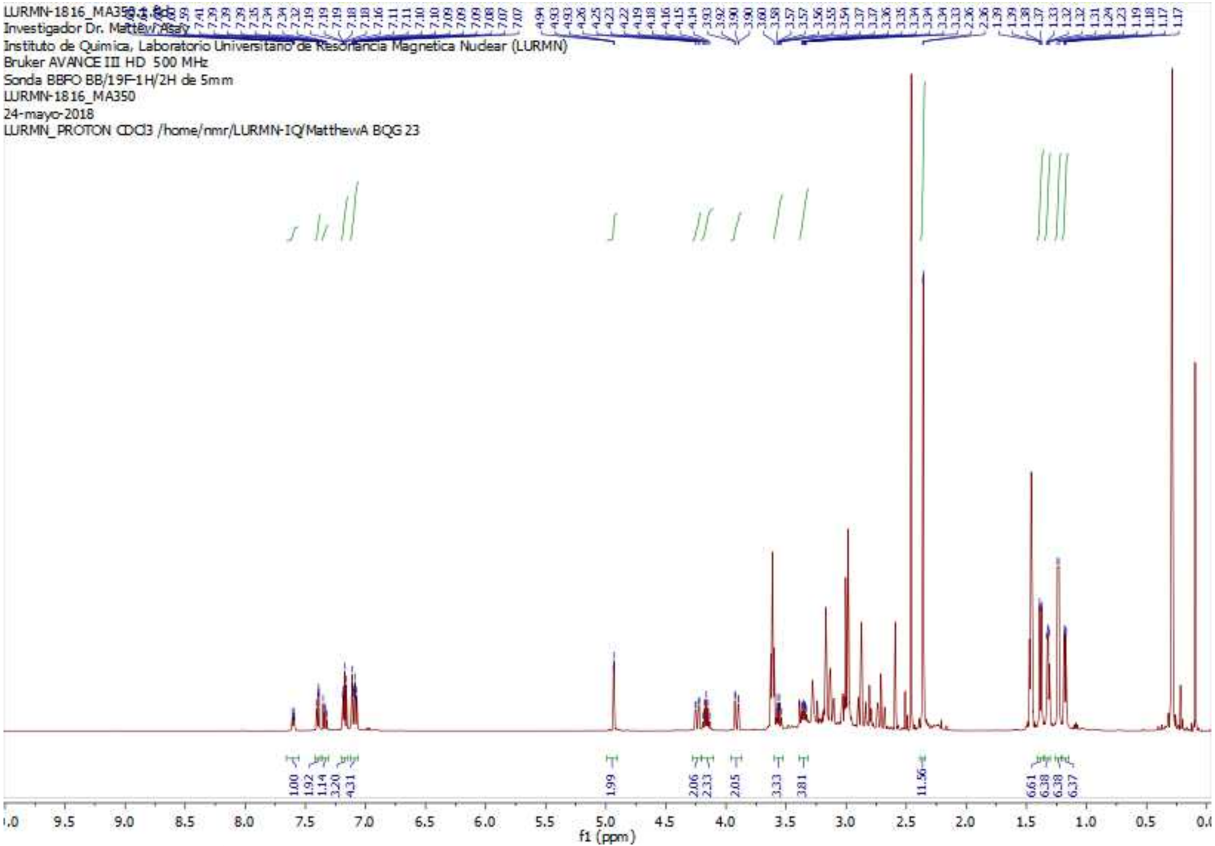

Figure S33. The ^{1}H NMR spectrum of reaction mixture of amide adduct **9** from the reaction in THF.

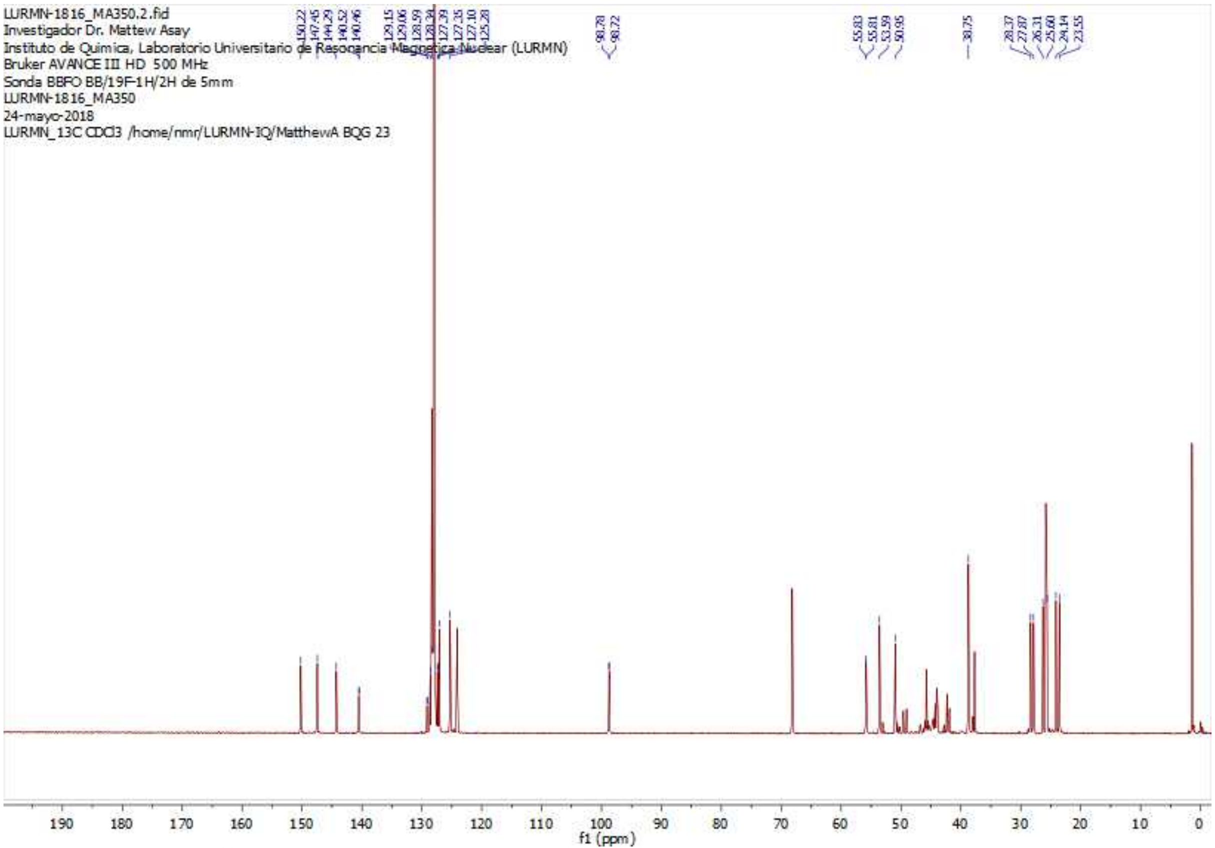

Figure S34. The ^{13}C NMR spectrum of reaction mixture of amide adduct **9** performed in THF.

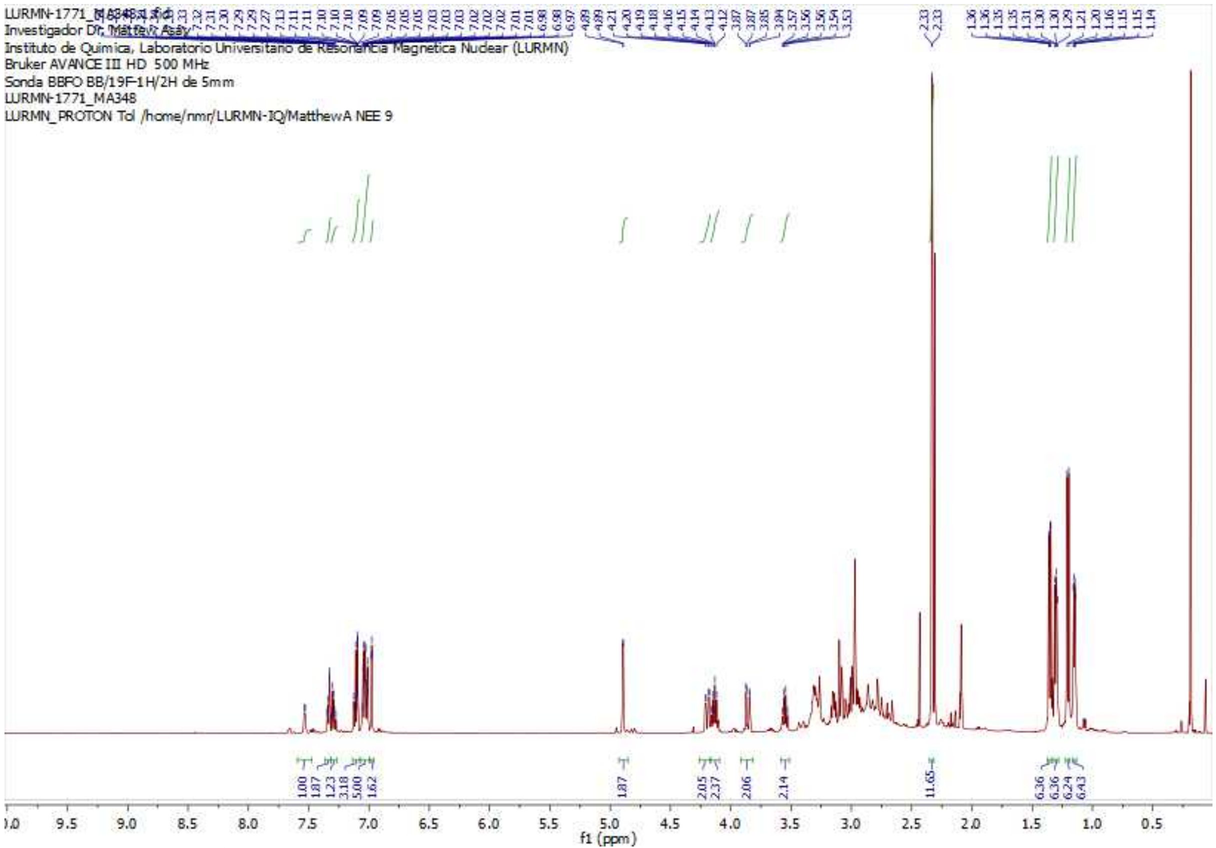

Figure S35. The ^{1}H NMR spectrum of reaction mixture of amide adduct **9** from the reaction in toluene-d_8.

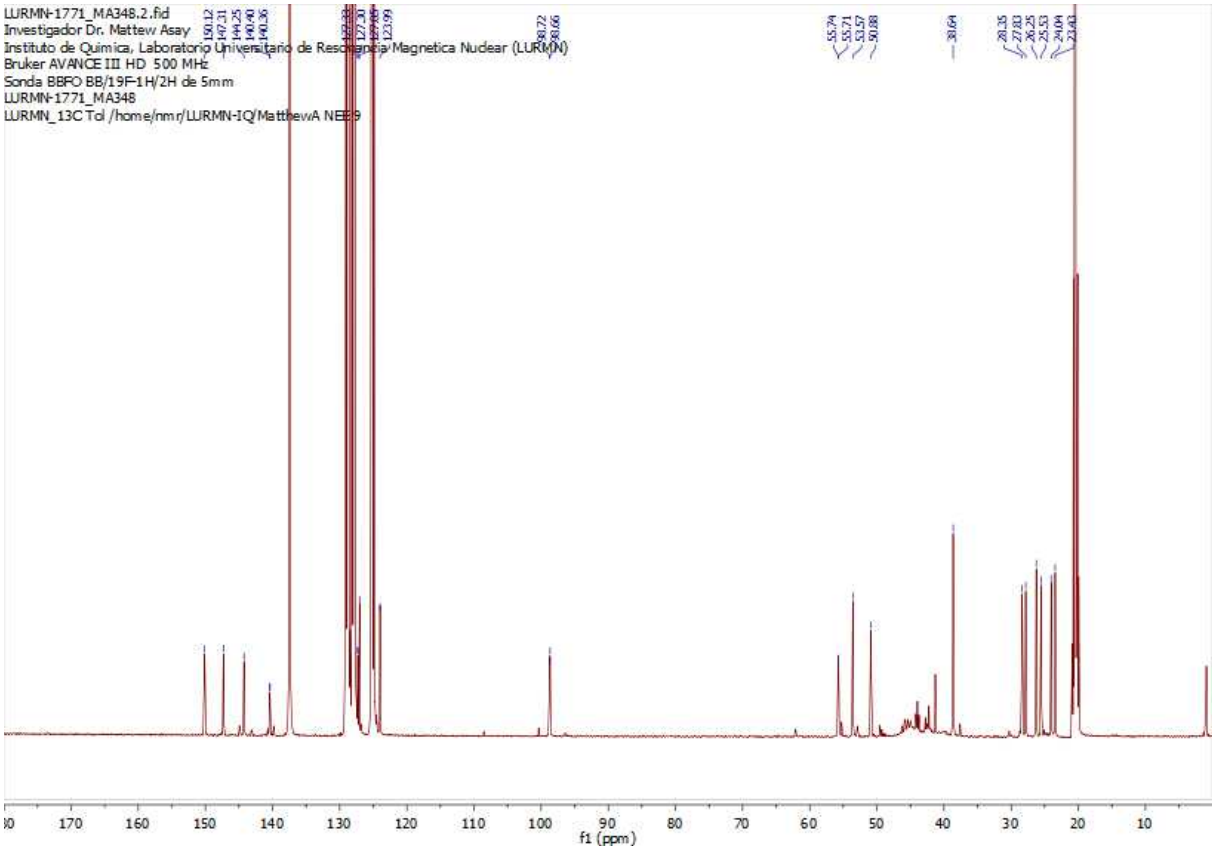

Figure S36. The ^{13}C NMR spectrum of reaction mixture of amide adduct **9** performed in toluene-*d*$_8$.

II. X-ray Data

Data for **[4a][Br]** (**028ASM17_0m**) was collected on a Bruker APEX II CCD Diffractometer at 150K, using Mo-Kα radiation (k = 0.71073 Å), from an Incoatec ImuS source and multilayer optic monochromators.[3] Frames were collected using omega scans and integrated with SAINT.[4] Multi-scan absorption correction (SADABS) was applied.[4] Suitable crystals were coated with hydrocarbon oil (Paratone), picked up with a glass fiber, and mounted in the cold nitrogen stream of the diffractometer. The structures were solved using intrinsic phasing (SHELXT)[5] and refined by full-matrix least-squares on F^2[6] using the ShelXle GUI.[7] The hydrogen atoms of the C–H bonds were placed in idealized positions and refined with U_{iso} tied to the parent atom.

Compound **[4a][Br]** crystalized in the monoclinic $P2_1/c$ space group as clear blocks. One of water molecule presents positional disorder in two positons with occupation refined in a 60/40 ratio. The full crystallographic information file (.CIF) has been deposited at the CCDC and can be obtain free of charge using the CCDC identifier 1851164.

Table S1. Crystal and structure refinement data for **[4a][Br]**.

Identification code	mo_028ASM17_0m	
Empirical formula	$C_{32} H_{44} Br_2 N_4 O_2$	
Formula weight	676.53	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 7.91973(9) Å	α = 90°.
	b = 37.0804(5) Å	β = 106.6518(5)°.
	c = 11.65888(13) Å	γ = 90°.
Volume	3280.24(7) Å^3	
Z	4	
Density (calculated)	1.370 Mg/m^3	
Absorption coefficient	2.505 mm^{-1}	

F(000)	1400
Crystal size	0.317 x 0.210 x 0.184 mm^3
Theta range for data collection	1.904 to 27.446°.
Index ranges	-10<=h<=10, -47<=k<=47, -15<=l<=15
Reflections collected	45779
Independent reflections	7503 [R(int) = 0.0295]
Completeness to theta = 25.242°	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7503 / 17 / 395
Goodness-of-fit on F^2	1.034
Final R indices [I>2sigma(I)]	R1 = 0.0266, wR2 = 0.0611
R indices (all data)	R1 = 0.0314, wR2 = 0.0627
Extinction coefficient	n/a
Largest diff. peak and hole	0.601 and -0.439 e.$Å^{-3}$

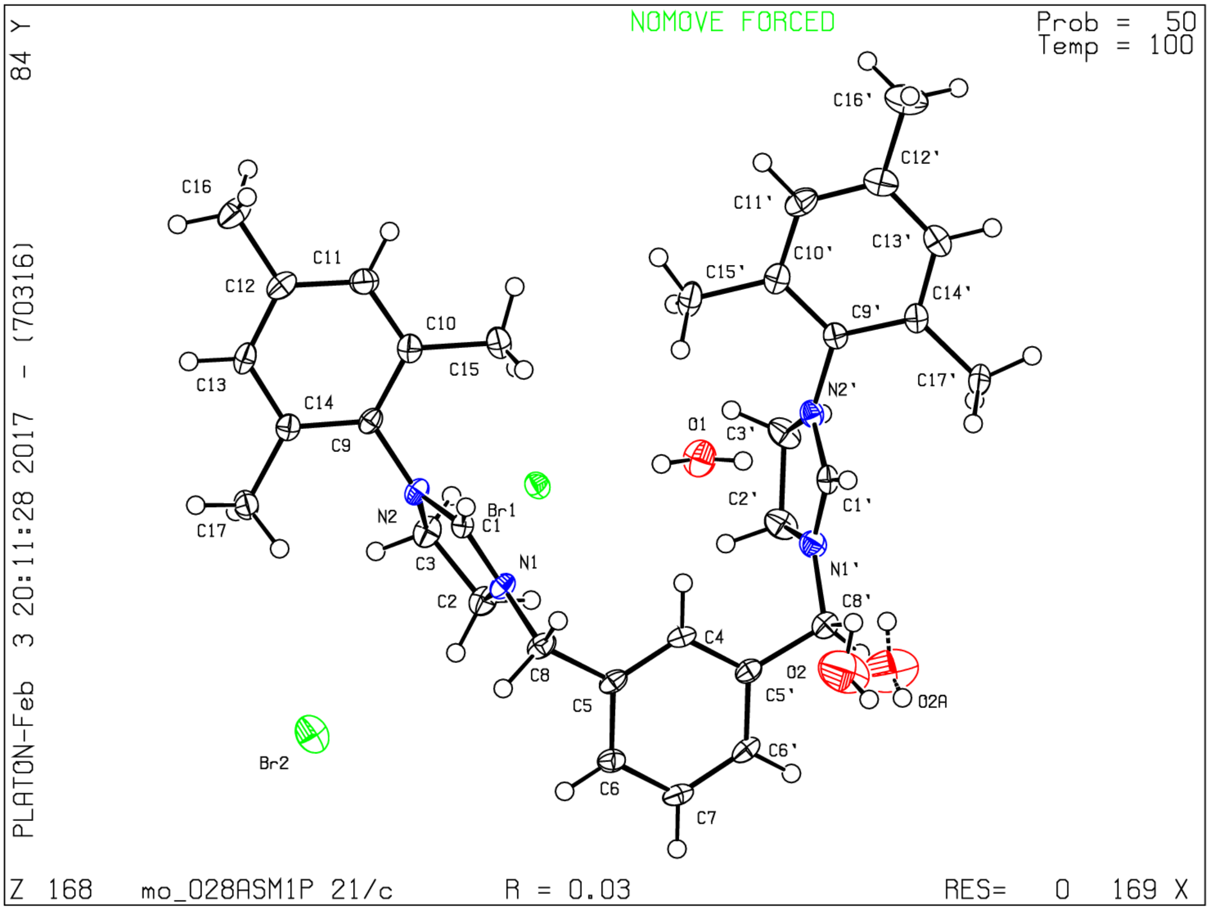

Figure S37. X-ray crystal structure of **[4a][Br]**.

Data for **[4b][Br]** (**twin5**) was collected on a Bruker APEX II CCD Diffractometer at 100K, using Mo-Kα radiation (k = 0.71073 Å), from an Incoatec ImuS sources and multilayer optic monochromators.[3] Frames were collected using omega scans and integrated with SAINT.[4] Multi-scan absorption correction (SADABS) was applied.[4] Suitable crystals were coated with hydrocarbon oil (Parabar), picked up with a nylon loop, and mounted in the cold nitrogen stream of the diffractometer. The structures were solved using intrinsic phasing (SHELXT)[5] and refined by full-matrix least-squares on F^2[6] using the shelXle GUI.[7] The hydrogen atoms of the C–H bonds were placed in idealized positions and were refined with U_{iso} tied to the parent atom.

Compound **[4b][Br]** crystalized in the triclinic P-1 space group. One molecule of water and one bromide anion present positional disorder, related to each other, in two positons with occupation refined in a 84/16 ratio, which was solved using geometry (SAME and DFIX) and Uij restraints (SIMU and RIGU) implemented in SHELXL[4] and the occupancy was refined using free variables. The full crystallographic information file (.CIF) has been deposited at the CCDC and can be obtain free of charge using the CCDC identifier 1851165.

Table S2. Crystal data and structure refinement for **[4b][Br]**.

Identification code	twin5	
Empirical formula	$C_{38} H_{57.70} Br_2 N_4 O_{2.84}$	
Formula weight	775.89	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.2559(2) Å	α = 98.4942(11)°.
	b = 10.8530(2) Å	β = 90.4235(10)°.
	c = 20.0395(5) Å	γ = 101.4568(9)°.
Volume	1949.90(7) Å^3	
Z	2	
Density (calculated)	1.321 Mg/m^3	
Absorption coefficient	2.117 mm^{-1}	
F(000)	813	
Crystal size	0.268 x 0.262 x 0.090 mm^3	
Theta range for data collection	1.937 to 27.532°.	
Index ranges	-12<=h<=12, -14<=k<=13, 0<=l<=26	
Reflections collected	8891	
Independent reflections	8891	

Completeness to theta = 25.242°	99.2 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8891 / 563 / 565
Goodness-of-fit on F^2	1.125
Final R indices [I>2sigma(I)]	R1 = 0.0387, wR2 = 0.1068
R indices (all data)	R1 = 0.0429, wR2 = 0.1086
Extinction coefficient	n/a
Largest diff. peak and hole	1.135 and -0.579 e.$Å^{-3}$

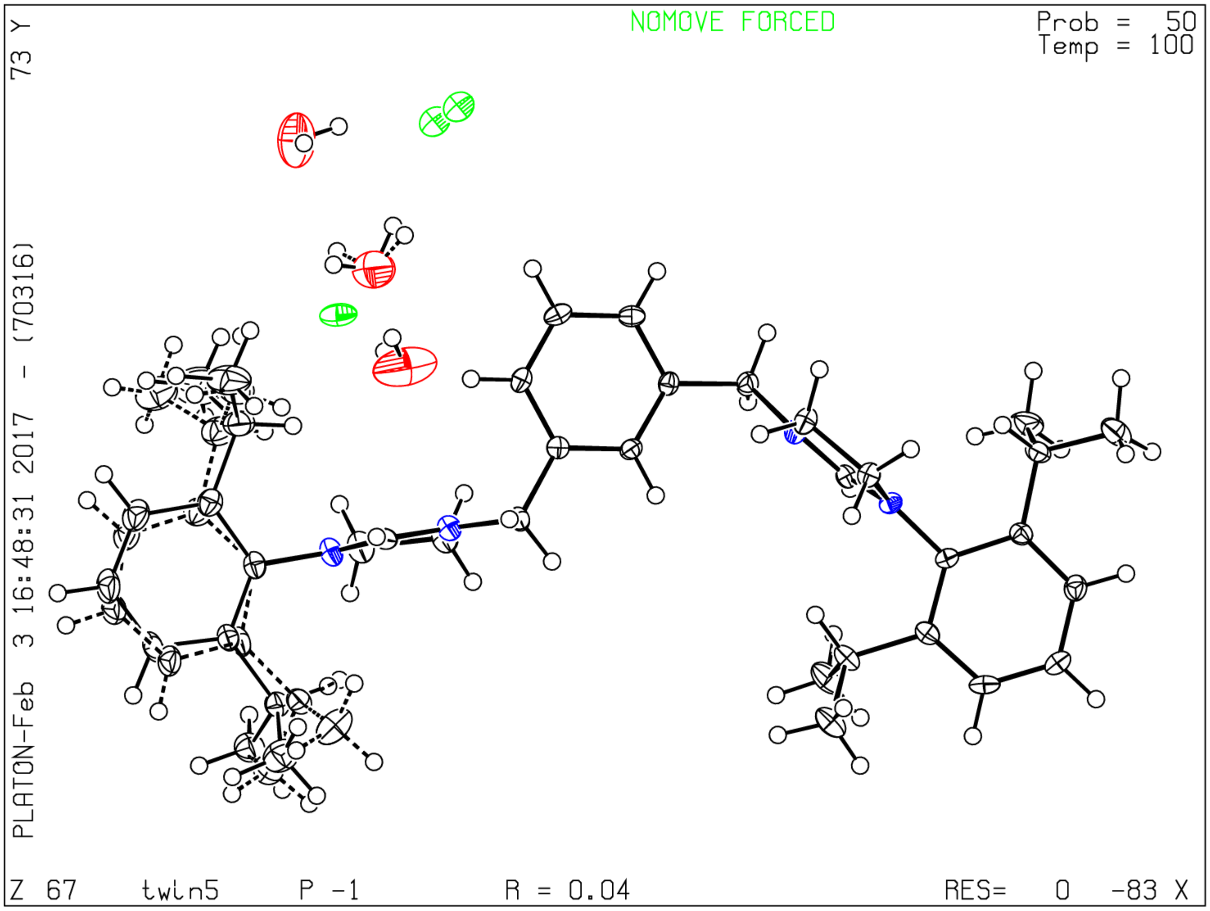

Figure S38. X-ray crystal structure of **[4b][Br]**.

Data for **[5b][PF$_6$]** (**083ASM17**) was collected on a Bruker APEX II CCD Diffractometer at 100K, using Cu-Kα radiation (k = 1.54178 Å), from an Incoatec ImuS sources and multilayer optic monochromators.[3] Frames were collected using omega scans and integrated with SAINT.[4] Multi-scan absorption correction (SADABS) was applied.[4] Suitable crystals were coated with hydrocarbon oil (Parabar), picked up with a nylon loop, and mounted in the cold nitrogen stream of the diffractometer. The structures were solved using intrinsic phasing (SHELXT)[5] and refined by full-matrix least-squares on F2 [6] using the shelXle GUI.[7] The hydrogen atoms of the C–H bonds were placed in idealized positions and were refined with U_{iso} tied to the parent atom.

Compound **[5b][PF$_6$]** crystalized as blocks in the triclinic P-1 space group. In the crystal of **[5b][PF$_6$]** one of the acetonitrile molecule presents positional disorder in special position (inversion center) that was modeled in two different positions with occupation refined at a 80/20 ratio. The global occupation was fixed at 0.5. The full crystallographic information file (.CIF) has been deposited at the CCDC and can be obtain free of charge using the CCDC identifier 1851166.

Table S3. Crystal data and structure refinement for **[5b][PF$_6$]**

Identification code	083AMS17_0m	
Empirical formula	$C_{86} H_{115} Ag_2 F_{12} N_{13} P_2$	
Formula weight	1836.58	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 11.8752(2) Å	a= 94.7901(5)°.
	b = 12.8426(2) Å	b= 99.9807(5)°.
	c = 15.1054(2) Å	g = 100.9198(5)°.
Volume	2211.58(6) Å^3	
Z	1	
Density (calculated)	1.379 Mg/m^3	
Absorption coefficient	0.555 mm^{-1}	
F(000)	954	
Crystal size	0.241 x 0.194 x 0.179 mm^3	
Theta range for data collection	1.780 to 27.446°.	
Index ranges	-15<=h<=15, -16<=k<=16, -19<=l<=19	
Reflections collected	73017	
Independent reflections	10113 [R(int) = 0.0212]	

Completeness to theta = 25.242°	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10113 / 87 / 572
Goodness-of-fit on F^2	1.038
Final R indices [I>2sigma(I)]	R1 = 0.0195, wR2 = 0.0505
R indices (all data)	R1 = 0.0204, wR2 = 0.0512
Extinction coefficient	n/a
Largest diff. peak and hole	0.477 and -0.319 e.Å^{-3}

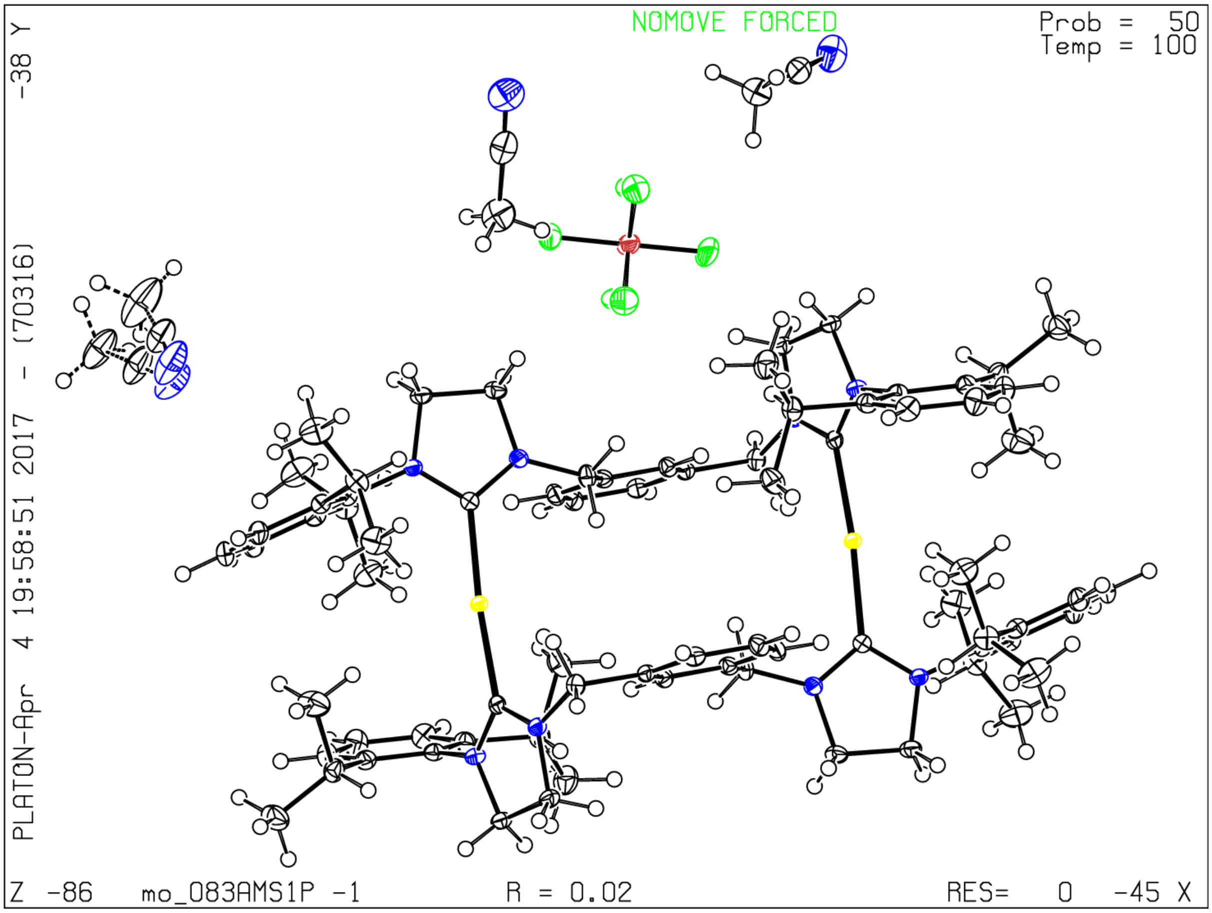

Figure S39. X-ray crystal structure of **[5b][PF$_6$]**.

Data for **6** (**163ASM18_2C2c**) was collected on a Bruker APEX II CCD Diffractometer at 100K, using Cu-Kα radiation (k = 1.54178 Å), from an Incoatec ImuS sources and multilayer optic monochromators.[3] Frames were collected using omega scans and integrated with SAINT.[4] Multi-scan absorption correction (SADABS) was applied.[4] Suitable crystals were coated with hydrocarbon oil (Parabar), picked up with a nylon loop, and mounted in the cold nitrogen stream of the diffractometer. The structures were solved using intrinsic phasing (SHELXT)[5] and refined by full-matrix least-squares on F2 [6] using the shelXle GUI.[7] The hydrogen atoms of the C–H bonds were placed in idealized positions and were refined with U_{iso} tied to the parent atom.

Compound **6** crystalized as orange blocks in the monoclinic C2/c space group. In the crystal of **6** one of the acetonitrile molecule is present. The full crystallographic information file (.CIF) has been deposited at the CCDC and can be obtain free of charge using the CCDC identifier 1851163.

Table S4. Crystal data and structure refinement for **6**

Identification code	163ASM18_2C2c	
Empirical formula	C50 H65 Cl2 Ir2 N5	
Formula weight	1191.37	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 28.1877(7) Å	α = 90°.
	b = 14.2540(3) Å	β = 91.0523(11)°.
	c = 11.8552(3) Å	γ = 90°.
Volume	4762.5(2) Å^3	
Z	4	
Density (calculated)	1.662 Mg/m^3	
Absorption coefficient	11.984 mm^{-1}	
F(000)	2352	
Crystal size	0.137 x 0.117 x 0.115 mm^3	
Theta range for data collection	3.475 to 71.835°.	
Index ranges	-34<=h<=34, -17<=k<=17, -14<=l<=14	
Reflections collected	41917	
Independent reflections	4606 [R(int) = 0.0295]	

Completeness to theta = 67.679°	99.3 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4606 / 24 / 285
Goodness-of-fit on F^2	1.108
Final R indices [I>2sigma(I)]	R1 = 0.0201, wR2 = 0.0525
R indices (all data)	R1 = 0.0203, wR2 = 0.0526
Extinction coefficient	n/a
Largest diff. peak and hole	1.352 and -0.678 e.Å^{-3}

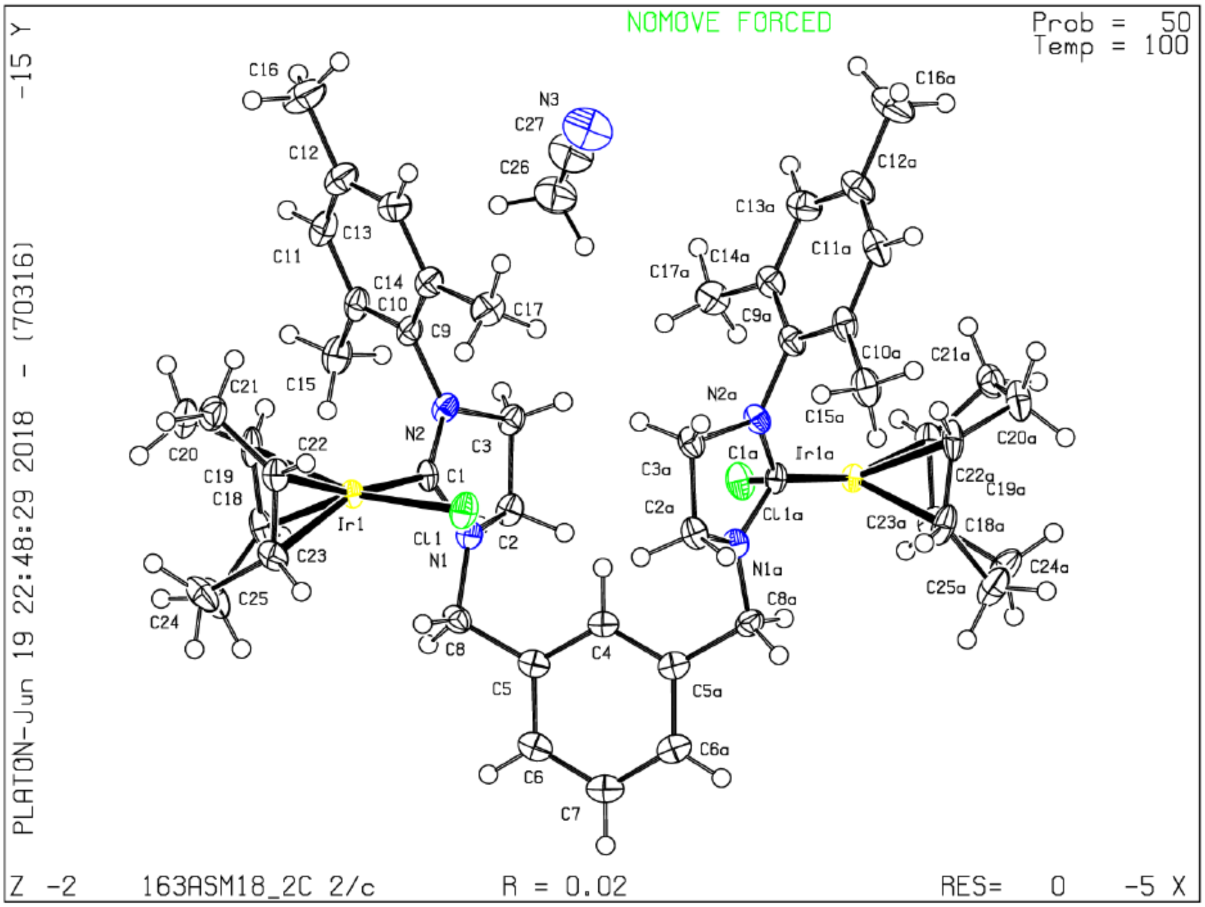

Figure S40. X-ray crystal structure of **6**

III. References

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