

Electronic Supplementary Information

Iridium-Catalyzed Regioselective B(3,6)-Dialkenylation or B(4)-Alkenylation of *o*-Carboranes via B–H Activation and 1,2-Carbon Migration of Alkynes

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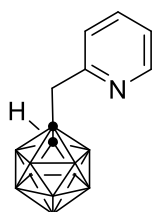
General Procedures.

All reactions were carried out in oven-dried glassware under an atmosphere of dry N₂ with the rigid exclusion of air and moisture using standard Schlenk techniques or in a glovebox. Diethyl ether and toluene were purified by solvent purification system prior to use. 1-(2-Picolyl)-*o*-carborane (**1**)¹, alkynes (**2**)² and 1-(2-pyridinyl)-2-methyl-*o*-carborane (**7**)³ were prepared according to literature procedures. All other chemicals were purchased from either Aldrich or J&K Chemical Co. and used as received unless otherwise specified. ¹H NMR spectra were recorded on a Bruker/Agilent/Varian 400 spectrometer at 400 MHz. ¹³C{¹H}, ¹¹B and ¹⁹F NMR spectra were recorded on a Bruker/Agilent/Varian 400 spectrometer at 101, 128 and 376 MHz, respectively. All signals were reported in ppm unit with references to the residual solvent resonances of the deuterated solvents for proton and carbon chemical shifts, to external BF₃·OEt₂ (0.00) for boron chemical shifts and to external CFCl₃ (0.00) for fluorine chemical shifts. Mass spectra were obtained on a Thermo Finnigan MAT 95 XL spectrometer. The melting points of solid compounds were determined by the melting point apparatus (Shanghai INESA Physico-Optical Instrument Co., LTD).

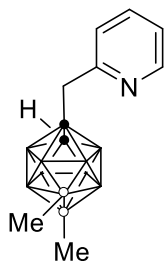
A Representative Procedure for the Preparation of Starting Materials 1a, 1b, 5a-

f. *o*-Carborane/1-substituted-*o*-carborane (10.0 mmol) was dissolved in Et₂O (20 mL) and cooled to 0 °C, to which was slowly added ⁿBuLi (11.0 mmol, 2.5 M in hexane, 4.4 mL). The resulting solution was stirred for 2 h at 0 °C. Then 2-picolyl chloride was added at 0 °C. The resulting solution was stirred at 40 °C overnight. After hydrolysis

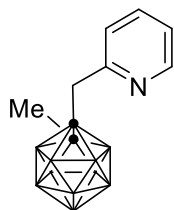
with water (20 mL) and extraction with diethyl ether (20 mL x 3), the ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to flash column chromatography on silica gel (200-300 mesh) using *n*-hexane and ethyl acetate (4/1 in v/v) as eluent to give the products **1/5**.



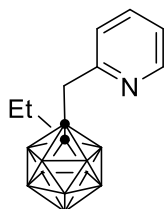
1a: White solid. Yield: 87%. Mp: 107.9-109.6 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.55 (d, $J = 4.0$ Hz, 1H), 7.70 (td, $J = 7.6, 2.0$ Hz, 1H), 7.25 (m, 1H), 7.17 (d, $J = 7.6$ Hz, 1H) (aromatic CH), 4.09 (s, 1H) (cage CH), 3.65 (s, 2H) (CH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 101 MHz): δ 155.2, 149.9, 137.4, 124.6, 123.2 (aromatic C), 73.4, 58.5 (cage C), 45.1 (CH_2). ^{11}B NMR (CDCl_3 , 128 MHz): δ -1.9 (d, $J = 148.5$ Hz, 1B), -5.4 (d, $J = 148.5$ Hz, 1B), -9.7 (d, $J = 145.9$ Hz, 2B), -10.8 (d, $J = 142.1$ Hz, 2B), -11.9 (d, $J = 105.0$ Hz, 2B), -12.8 (d, $J = 157.4$ Hz, 2B). HRMS (DART) Calcd for $\text{C}_8\text{H}_{18}^{10}\text{B}_2^{11}\text{B}_8\text{N}^+$ [$\text{M}+\text{H}^+$]: 236.2437, Found: 236.2437.



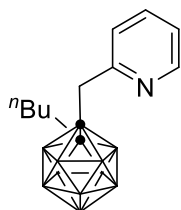
1b: White solid. Yield: 81%. Mp: 79.8-81.6 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.54 (d, $J = 5.2$ Hz, 1H), 7.66 (td, $J = 7.6, 2.0$ Hz, 1H), 7.22 (m, 1H), 7.15 (d, $J = 8.0$ Hz, 1H) (aromatic CH), 3.85 (s, 1H) (cage CH), 3.63 (s, 2H) (CH_2), 0.19 (s, 3H), 0.12 (s, 3H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 101 MHz): δ 155.5, 149.8, 137.2, 124.5, 123.0 (aromatic C), 66.3, 51.7 (cage C), 44.4 (CH_2), the $\text{B}_{\text{cage}}\text{-C}$ was not observed. ^{11}B NMR (CDCl_3 , 128 MHz): δ 8.0 (s, 1B), 4.5 (s, 1B), -7.7 (d, $J = 147.2$ Hz, 2B), -10.9 (d, $J = 158.7$ Hz, 2B), -12.9 (d, $J = 153.6$ Hz, 4B). HRMS (DART) Calcd for $\text{C}_{10}\text{H}_{22}^{10}\text{B}_2^{11}\text{B}_8\text{N}^+$ [$\text{M}+\text{H}^+$]: 264.2750, Found: 264.2750.



5a: White solid. Yield: 80%. Mp: 65.4-66.5 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.55 (d, $J = 5.2$ Hz, 1H), 7.69 (td, $J = 7.6, 1.6$ Hz, 1H), 7.25 (m, 2H) (aromatic CH), 3.68 (s, 2H) (CH_2), 2.26 (s, 3H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 101 MHz): δ 155.4, 149.5, 136.9, 125.2, 123.1 (aromatic C), 77.4, 75.3 (cage C), 43.7 (CH_2), 23.8 (CH_3). ^{11}B NMR (CDCl_3 , 128 MHz): δ -4.0 (d, $J = 184.3$ Hz, 1B), -5.6 (d, $J = 174.1$ Hz, 1B), -9.4 (d, $J = 107.5$ Hz, 4B), -10.3 (d, $J = 142.1$ Hz, 4B). HRMS (DART) Calcd for $\text{C}_9\text{H}_{20}^{10}\text{B}_2^{11}\text{B}_8\text{N}^+$ [$\text{M}+\text{H}^+$]: 250.2593, Found: 250.2593.

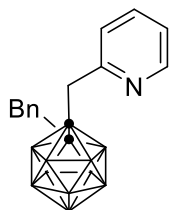


5b: Yellow oil. Yield: 74%. ^1H NMR (400 MHz, CDCl_3): δ 8.55 (d, $J = 3.6$ Hz, 1H), 7.69 (td, $J = 7.6, 1.6$ Hz, 1H), 7.24 (m, 2H) (aromatic CH), 3.66 (s, 2H) (CH_2Py), 2.57 (q, $J = 7.6$ Hz, 2H) (CH_2CH_3), 1.20 (t, $J = 7.6$ Hz, 3H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 101 MHz): δ 155.4, 149.5, 136.8, 125.2, 123.0 (aromatic C), 81.3, 78.4 (cage C), 43.3 (CH_2Py), 29.0 (CH_2CH_3), 14.3 (CH_2CH_3). ^{11}B NMR (CDCl_3 , 128 MHz): δ -4.8 (d, $J = 149.8$ Hz, 2B), -10.3 (m, 6B), -11.0 (m, 2B). HRMS (DART) Calcd for $\text{C}_{10}\text{H}_{22}^{10}\text{B}_2^{11}\text{B}_8\text{N}^+$ [$\text{M}+\text{H}^+$]: 264.2750, Found: 264.2750.



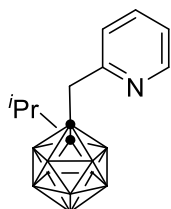
5c: White solid. Yield: 89%. Mp: 61.9-62.3 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.55 (d, $J = 4.4$ Hz, 1H), 7.68 (td, $J = 7.6, 1.6$ Hz, 1H), 7.24 (m, 2H) (aromatic CH), 3.66 (s, 2H) (CH_2Py), 2.48 (m, 2H), 1.55 (m, 2H), 1.38 (m, 2H) (alkyl CH_2), 0.96 (t, $J = 7.2$ Hz, 3H) (CH_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 101 MHz): δ 155.3, 149.5, 136.7, 125.1, 122.9 (aromatic C), 80.5, 78.4 (cage C), 43.3 (CH_2Py), 35.1, 31.9, 22.5 (alkyl CH_2), 13.8 (CH_3). ^{11}B NMR (CDCl_3 , 128 MHz): δ -4.7 (d, $J = 147.2$ Hz, 2B), -10.3 (d, $J = 129.3$ Hz, 8B). HRMS (DART) Calcd for

$C_{12}H_{26}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 292.3063, Found: 292.3063.



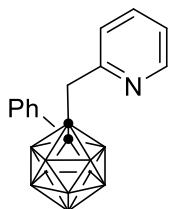
5d: White solid. Yield: 73%. Mp: 95.0-97.4 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.62 (d, J = 4.8 Hz, 1H), 7.74 (td, J = 7.6, 2.0 Hz, 1H), 7.32 (m, 5H), 7.27 (m, 2H) (aromatic CH), 3.92 (s, 2H), 3.88 (s, 2H) (CH_2).

$^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 155.6, 149.5, 137.0, 135.8, 130.5, 128.6, 128.0, 125.3, 123.2 (aromatic C), 79.9, 78.9 (cage C), 43.8, 40.9 (CH_2). ^{11}B NMR ($CDCl_3$, 128 MHz): δ -4.4 (d, J = 144.6 Hz, 2B), -10.0 (d, J = 125.4 Hz, 8B). HRMS (DART) Calcd for $C_{15}H_{24}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 326.2906, Found: 326.2905.



5e: White solid. Yield: 84%. Mp: 67.1-68.0 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.54 (d, J = 4.4 Hz, 1H), 7.68 (td, J = 8.0, 2.0 Hz, 1H), 7.24 (m, 2H) (aromatic CH), 3.67 (s, 2H) (CH_2), 2.93 (m, 1H) ($CH(CH_3)_2$),

1.25 (d, J = 6.8 Hz, 6H) ($CH(CH_3)_2$). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 155.4, 149.4, 136.8, 125.3, 123.0 (aromatic C), 86.7, 80.8 (cage C), 43.3 (CH_2), 31.3 ($CH(CH_3)_2$), 24.6 ($CH(CH_3)_2$). ^{11}B NMR ($CDCl_3$, 128 MHz): δ -3.9 (d, J = 139.5 Hz, 1B), -5.0 (d, J = 135.7 Hz, 1B), -9.8 (d, J = 135.7 Hz, 2B), -10.8 (d, J = 131.8 Hz, 4B), -11.7 (d, J = 139.5 Hz, 2B). HRMS (DART) Calcd for $C_{11}H_{24}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 278.2906, Found: 278.2906.



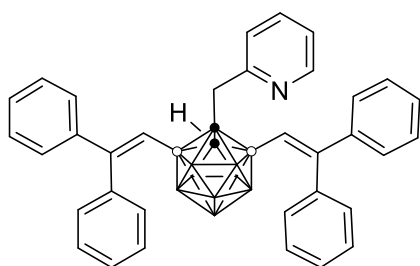
5f: White solid. Yield: 91%. Mp: 102.6-104.2 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.54 (d, J = 5.2 Hz, 1H), 7.75 (m, 2H), 7.58 (td, J = 7.6, 1.6 Hz, 1H), 7.46 (m, 3H), 7.18 (m, 1H), 6.88 (d, J = 7.6 Hz, 1H)

(aromatic CH), 3.23 (s, 2H) (CH_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 155.1, 149.7, 136.6, 131.6, 130.9, 130.8, 129.2, 124.8, 122.8 (aromatic C), 83.7, 80.7 (cage C), 43.2

(CH₂). ¹¹B NMR (CDCl₃, 128 MHz): δ -3.4 (d, *J* = 145.9 Hz, 2B), -10.0 (d, *J* = 115.2 Hz, 4B), -10.8 (d, *J* = 113.9 Hz, 4B). HRMS (DART) Calcd for C₁₄H₂₂¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 312.2750, Found: 312.2750.

General procedure for the synthesis of 4 or 6.

An oven-dried Schlenk flask equipped with a stir bar was charged with 1-(2-picolyl)-*o*-carborane (**1/5**) (0.2 mmol), alkyne (**2**) (0.5 mmol), [Cp*IrCl₂]₂ (4.0 mg, 0.005 mmol), AgNTf₂ (7.7 mg, 0.02 mmol) and HOAc (6.0 mg, 0.1 mmol), followed by dry toluene (3 mL). The flask was closed under an atmosphere of nitrogen and stirred at 130 °C for 12 h. After the addition of water (5 mL) and extraction with diethyl ether (5 mL x 3), the ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to flash column chromatography on silica gel (200-300 mesh) using *n*-hexane and ethyl acetate (10/1 in v/v) as eluent to give product **4/6**.



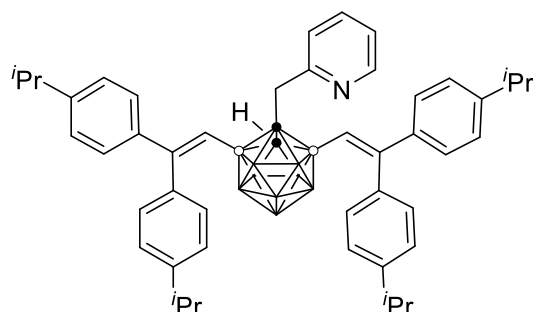
4a: White solid. Yield: 90%. Mp: 185.9-186.4 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.66 (d, *J* = 4.8 Hz, 1H), 7.51 (td, *J* = 7.6, 2.0 Hz, 1H), 7.39 (m, 6H), 7.25 (m, 6H), 7.20 (m, 9H), 7.09 (d, *J* = 8.0 Hz, 1H)

(aromatic CH), 6.26 (s, 2H) (alkenyl CH), 3.73 (s, 1H) (cage CH), 3.63 (s, 2H) (CH₂).

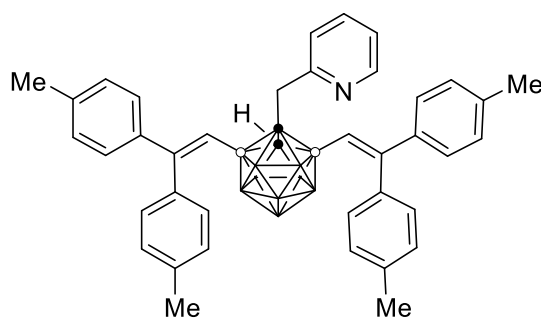
¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 156.8, 155.7, 149.2, 142.6, 140.3, 136.6, 129.9, 128.3, 128.2, 128.1, 127.8, 127.2, 125.3, 122.6 (alkenyl and aromatic C), 74.1 (cage C), 62.0 (cage CH), 41.5 (CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz):

δ -4.5 (m, 4B), -10.6 (m, 6B). HRMS (DART) Calcd for $C_{36}H_{38}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 593.3992, Found: 593.3981.



4b: White solid. Yield: 91%. Mp: 102.3-104.0 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.62 (d, J = 4.8 Hz, 1H), 7.49 (td, J = 7.6, 1.6 Hz, 1H), 7.23 (d, J = 8.0 Hz, 4H), 7.16 (m, 13H), 7.02 (d, J = 7.6 Hz, 1H)

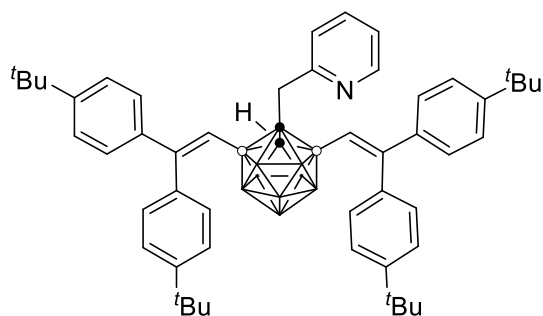
(aromatic CH), 6.15 (s, 2H) (alkenyl CH), 3.56 (s, 2H) (CH_2), 3.26 (s, 1H) (cage CH), 2.98 (m, 2H), 2.88 (m, 2H) ($CH(CH_3)_2$), 1.30 (d, J = 6.8 Hz, 12H), 1.23 (d, J = 6.8 Hz, 12H) (CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 156.7, 155.8, 149.2, 149.0, 148.6, 140.5, 137.8, 136.4, 130.0, 127.3, 126.2, 126.1, 125.3, 122.5 (alkenyl and aromatic C), 74.3 (cage C), 62.3 (cage CH), 41.5 (CH_2), 34.0, 33.9 ($CH(CH_3)_2$), 24.3, 24.2, 24.1, 24.0 (CH_3), the $B_{cage}-C$ was not observed. ^{11}B NMR ($CDCl_3$, 128 MHz): δ -4.5 (m, 4B), -10.7 (m, 6B). HRMS (DART) Calcd for $C_{48}H_{62}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 760.5880, Found: 760.5867.



4c: White solid. Yield: 95%. Mp: 101.3-103.4 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.63 (d, J = 4.8 Hz, 1H), 7.50 (td, J = 7.6, 1.6 Hz, 1H), 7.18 (m, 5H), 7.06 (m, 13H) (aromatic CH), 6.19 (s, 2H) (alkenyl CH),

3.64 (s, 1H) (cage CH), 3.59 (s, 2H) (CH_2), 2.41 (s, 6H), 2.32 (s, 6H) (CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 156.7, 155.8, 149.2, 140.3, 138.0, 137.5, 137.4, 136.5,

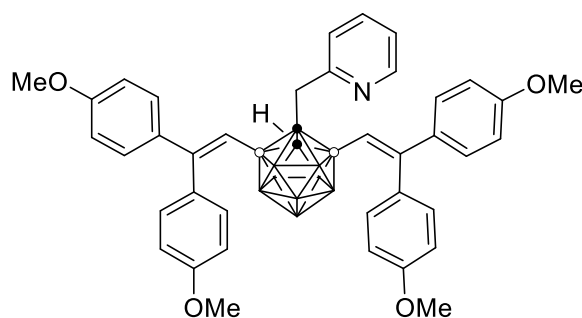
129.9, 128.9, 127.2, 125.3, 122.5 (alkenyl and aromatic C), 74.1 (cage C), 62.3 (cage CH), 41.5 (CH₂), 21.5, 21.3 (CH₃), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.5 (m, 4B), -10.7 (m, 6B). HRMS (DART) Calcd for C₄₀H₄₆¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 648.4628, Found: 648.4623.



4d: Yellow solid. Yield: 92%. Mp: 157.7-

159.1 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.62 (d, *J* = 4.4 Hz, 1H), 7.49 (td, *J* = 7.6, 1.6 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 4H), 7.29 (d, *J* = 8.0 Hz, 4H), 7.18 (m, 9H), 7.01 (d,

J = 7.6 Hz, 1H) (aromatic CH), 6.14 (s, 2H) (alkenyl CH), 3.56 (s, 2H) (CH₂), 3.09 (s, 1H) (cage CH), 1.37 (s, 18H), 1.31 (s, 18H) (CH₃). ¹³C {¹H} NMR (CDCl₃, 101 MHz): δ 156.5, 155.8, 151.2, 151.0, 149.3, 140.0, 137.4, 136.3, 129.7, 127.0, 125.3, 125.1, 124.9, 122.5 (alkenyl and aromatic C), 74.3 (cage C), 62.3 (cage CH), 41.5 (CH₂), 34.8, 34.7 (C(CH₃)₃), 31.6, 31.4 (C(CH₃)₃), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.8 (m, 4B) -10.7 (m, 6B). HRMS (DART) Calcd for C₅₂H₇₀¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 816.6506, Found: 816.6499.

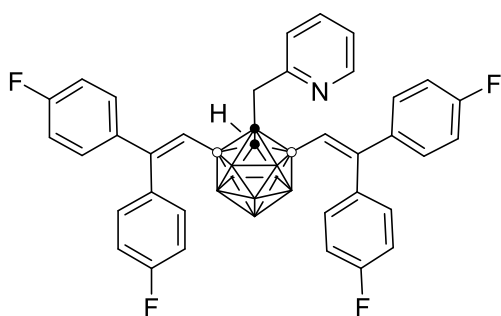


4e: Yellow solid. Yield: 90%. Mp:

88.9-89.1 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.63 (d, *J* = 4.0 Hz, 1H), 7.51 (td, *J* = 8.4, 2.0 Hz, 1H), 7.19 (m, 1H), 7.10 (m, 9H), 6.91 (d, *J* = 8.4 Hz,

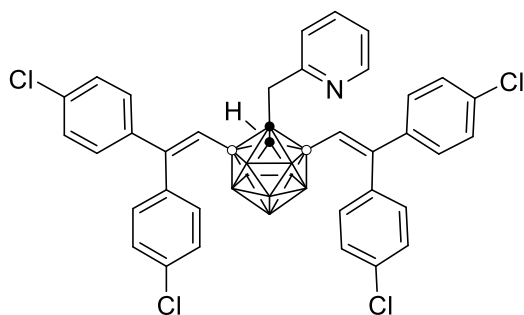
4H), 6.77 (d, *J* = 8.4 Hz, 4H) (aromatic CH), 6.14 (s, 2H) (alkenyl CH), 3.86 (s, 6H),

3.79 (s, 6H) (OCH₃), 3.62 (s, 1H) (cage CH), 3.60 (s, 2H) (CH₂). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 159.7, 159.2, 156.0, 155.8, 149.2, 136.5, 135.8, 132.9, 131.1, 128.6, 125.3, 122.5, 113.6, 113.5 (alkenyl and aromatic C), 74.1 (cage C), 62.1 (cage CH), 55.4, 55.4 (OCH₃), 41.5 (CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.3 (m, 4B), -10.7 (m, 6B). HRMS (DART) Calcd for C₄₀H₄₆¹⁰B₂¹¹B₈NO₄⁺ [M+H⁺]: 712.4425, Found: 712.4409.



4f: White solid. Yield: 75%. Mp: 89.3-91.1 °C.

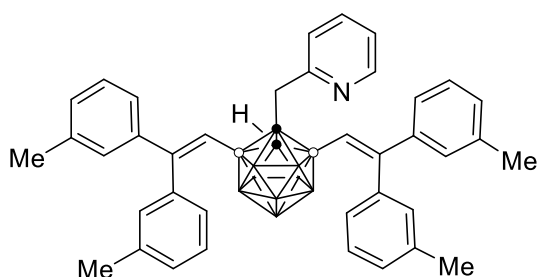
¹H NMR (400 MHz, CDCl₃): δ 8.66 (d, *J* = 4.8 Hz, 1H), 7.50 (td, *J* = 7.6, 1.6 Hz, 1H), 7.22 (m, 1H), 7.15 (m, 4H), 7.07 (m, 9H), 6.92 (t, *J* = 8.8 Hz, 4H) (aromatic CH), 6.13 (s, 2H) (alkenyl CH), 4.18 (s, 1H) (cage CH), 3.63 (s, 2H) (CH₂). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 162.8 (d, ¹*J*_{CF} = 249.5 Hz), 162.6 (d, ¹*J*_{CF} = 248.5 Hz), 155.7, 155.1, 149.2, 138.7 (d, ⁴*J*_{CF} = 3.0 Hz), 136.9, 136.0 (d, ⁴*J*_{CF} = 4.0 Hz), 131.6 (d, ³*J*_{CF} = 8.1 Hz), 128.9 (d, ³*J*_{CF} = 9.1 Hz), 125.3, 122.8, 115.4 (d, ²*J*_{CF} = 21.2 Hz), 115.1 (d, ²*J*_{CF} = 21.2 Hz) (alkenyl and aromatic C), 73.9 (cage C), 61.8 (cage CH), 41.5 (CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.6 (m, 4B), -10.3 (m, 6B). ¹⁹F NMR (CDCl₃, 376 MHz): δ -113.8 (m, 4F). HRMS (DART) Calcd for C₃₆H₃₄¹⁰B₂¹¹B₈F₄N⁺ [M+H⁺]: 664.3625, Found: 664.3623.



4g: White solid. Yield: 62%. Mp: 215.0-

215.8 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.66 (d, *J* = 4.8 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 4H), 7.21 (m, 5H),

7.12 (d, $J = 8.0$ Hz, 4H), 7.04 (d, $J = 7.6$ Hz, 1H), 6.98 (d, $J = 8.0$ Hz, 4H) (aromatic CH), 6.15 (s, 2H) (alkenyl CH), 4.38 (s, 1H) (cage CH), 3.63 (s, 2H) (CH_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 155.6, 154.8, 149.2, 140.7 138.2, 137.1, 134.3, 134.1, 131.3, 128.7, 128.4, 125.2, 122.8 (alkenyl and aromatic C), 73.8 (cage C), 61.8 (cage CH), 41.4 (CH_2), the $B_{cage}-C$ was not observed. ^{11}B NMR ($CDCl_3$, 128 MHz): δ -4.6 (m, 4B), -10.4 (m, 6B). HRMS (DART) Calcd for $C_{36}H_{34}^{10}B_2^{11}B_8Cl_4N^+$ $[M+H^+]$: 730.2414, Found: 730.2412.



4h: White solid. Yield: 84%. Mp: 127.4-

129.1 °C. 1H NMR (400 MHz, $CDCl_3$): δ

8.63 (d, $J = 4.4$ Hz, 1H), 7.50 (td, $J = 7.6$,

2.0 Hz, 1H), 7.22 (m, 2H), 7.15 (m, 5H),

7.04 (m, 7H), 6.96 (m, 4H) (aromatic CH), 6.14 (s, 2H) (alkenyl CH), 3.58 (s, 2H)

(CH_2), 3.53 (s, 1H) (cage CH), 2.35 (s, 6H), 2.29 (s, 6H) (CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$,

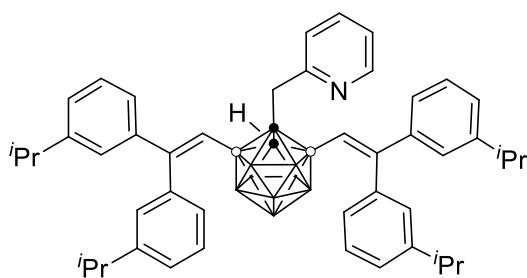
101 MHz): δ 157.2, 155.9, 149.2, 142.8, 140.2, 137.7, 137.7, 136.6, 130.9, 128.9, 128.4,

128.1, 128.0, 127.8, 126.9, 125.3, 124.6, 122.5 (alkenyl and aromatic C), 74.3 (cage C),

62.3 (cage CH), 41.4 (CH_2), 21.6, 21.6 (CH_3), the $B_{cage}-C$ was not observed. ^{11}B NMR

($CDCl_3$, 128 MHz): δ -4.5 (m, 4B), -10.5 (m, 6B). HRMS (DART) Calcd for

$C_{40}H_{46}^{10}B_2^{11}B_8N^+$ $[M+H^+]$: 648.4628, Found: 648.4619.



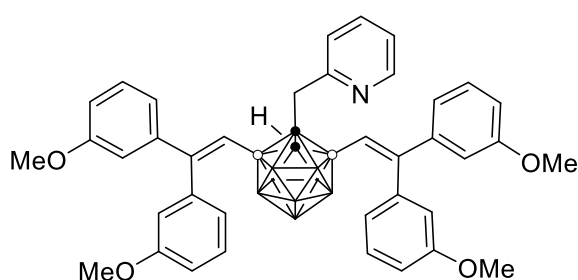
4i: Yellow solid. Yield: 95%. Mp: 95.8-

96.7 °C. 1H NMR (400 MHz, $CDCl_3$): δ

8.68 (d, $J = 4.8$ Hz, 1H), 7.53 (t, $J = 7.6$

Hz, 1H), 7.31 (td, $J = 7.6$, 1.6 Hz, 2H),

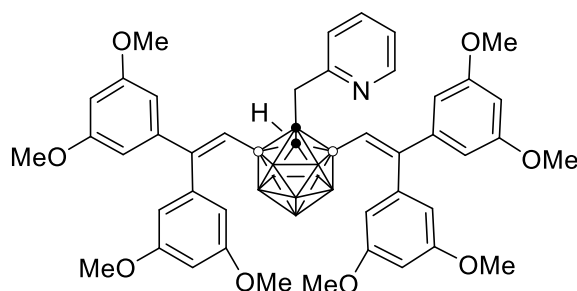
7.20 (m, 11H), 7.10 (d, $J = 8.0$ Hz, 1H), 7.01 (d, $J = 7.6$ Hz, 4H) (aromatic CH), 6.25 (d, $J = 2.0$ Hz, 2H) (alkenyl CH), 3.64 (s, 2H) (CH_2), 3.57 (s, 1H) (cage CH), 2.91 (m, 4H) ($CH(CH_3)_2$), 1.29 (d, $J = 6.8$ Hz, 12H), 1.25 (d, $J = 6.8$ Hz, 12H) ($CH(CH_3)_2$). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 157.4, 155.9, 149.2, 148.8, 148.7, 142.7, 140.3, 136.5, 128.5, 128.1, 128.1, 127.1, 126.1, 125.8, 125.3, 125.2, 125.1, 122.5 (alkenyl and aromatic C), 74.2 (cage C), 62.2 (cage CH), 41.5 (CH_2), 34.3 ($CH(CH_3)_2$), 24.2, 24.1, 24.1 ($CH(CH_3)_2$), the $B_{cage}-C$ was not observed. ^{11}B NMR ($CDCl_3$, 128 MHz): δ -4.8 (m, 4B), -10.8 (m, 6B). HRMS (DART) Calcd for $C_{48}H_{62}^{10}B_2^{11}B_8N^+$ [$M+H^+$]: 760.5880, Found: 760.5865.



4j: Yellow solid. Yield: 91%. Mp:

123.9-125.6 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.65 (d, $J = 4.8$ Hz, 1H), 7.51 (td, $J = 7.6, 2.0$ Hz, 1H), 7.29 (m,

2H), 7.19 (m, 3H), 7.08 (d, $J = 7.6$ Hz, 1H), 6.92 (d, $J = 6.8$ Hz, 2H), 6.79 (m, 10H) (aromatic CH), 6.24 (s, 2H) (alkenyl CH), 3.82 (s, 6H) (OCH_3), 3.79 (s, 1H) (cage CH), 3.76 (s, 6H) (OCH_3), 3.65 (s, 2H) (CH_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 159.5, 159.5, 156.5, 155.6, 149.1, 143.9, 141.4, 136.7, 129.3, 129.1, 125.4, 122.7, 122.2, 119.8, 115.6, 113.6, 113.2 (alkenyl and aromatic C), 74.1 (cage C), 62.2 (cage CH), 55.4, 55.3 (OCH_3), 41.5 (CH_2), the $B_{cage}-C$ was not observed. ^{11}B NMR ($CDCl_3$, 128 MHz): δ -5.0 (m, 4B), -10.8 (m, 6B). HRMS (DART) Calcd for $C_{40}H_{46}^{10}B_2^{11}B_8NO_4^+$ [$M+H^+$]: 713.4418, Found: 713.4415.



4k: White solid. Yield: 77%. Mp:

153.4-153.9 °C. ^1H NMR (400 MHz,

CDCl_3): δ 8.64 (d, $J = 4.8$ Hz, 1H),

7.53 (td, $J = 8.0, 2.0$ Hz, 1H), 7.20 (m,

1H), 7.10 (d, $J = 8.0$ Hz, 1H), 6.49 (s,

2H), 6.43 (s, 8H), 6.40 (s, 2H) (aromatic CH), 6.21 (s, 2H) (alkenyl CH), 3.92 (s, 1H)

(cage CH), 3.80 (s, 12H), 3.75 (s, 12H) (CH_3), 3.69 (s, 2H) (CH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR

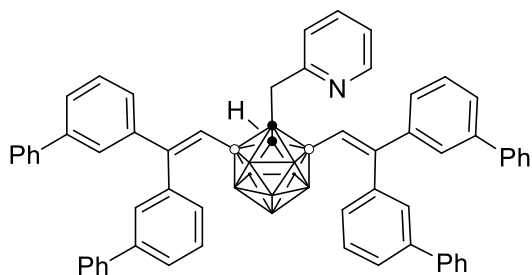
(CDCl_3 , 101 MHz): δ 160.6, 160.5, 156.5, 155.7, 149.0, 144.4, 141.8, 136.6, 125.3,

122.6, 108.2, 105.7, 100.0, 99.8 (alkenyl and aromatic C), 74.3 (cage C), 62.3 (cage

CH), 55.5, 55.4 (OCH_3), 41.4 (CH_2), the $\text{B}_{\text{cage}}\text{-C}$ was not observed. ^{11}B NMR (CDCl_3 ,

192 MHz): δ -5.0 (m, 4B), -10.8 (m, 6B). HRMS (DART) Calcd for

$\text{C}_{44}\text{H}_{54}^{10}\text{B}_2^{11}\text{B}_8\text{NO}_8^+$ [$\text{M}+\text{H}^+$]: 832.4847, Found: 832.4834.



4l: Yellow solid. Yield: 73%. Mp: 128.3-

129.7 °C. ^1H NMR (400 MHz, CDCl_3): δ

8.57 (d, $J = 3.6$ Hz, 1H), 7.59 (m, 6H),

7.48 (m, 10H), 7.42 (m, 9H), 7.33 (m, 8H),

7.21 (d, $J = 7.2$ Hz, 2H), 7.12 (d, $J = 7.6$ Hz, 2H), 7.03 (m, 2H) (aromatic CH), 6.34 (s,

2H) (alkenyl CH), 4.05 (s, 1H) (cage CH), 3.69 (s, 2H) (CH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 ,

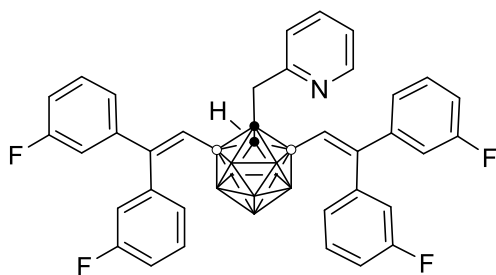
101 MHz): δ 157.0, 155.7, 149.2, 143.1, 141.2, 141.1, 140.9, 140.6, 136.7, 128.9, 128.8,

128.8, 128.7, 127.5, 127.3, 127.3, 127.0, 126.7, 126.5, 125.8, 125.3, 122.6 (alkenyl and

aromatic C), 74.3 (cage C), 62.3 (cage CH), 41.5 (CH_2), the $\text{B}_{\text{cage}}\text{-C}$ was not observed.

^{11}B NMR (CDCl_3 , 128 MHz): δ -4.8 (m, 4B), -10.1 (m, 6B). HRMS (DART) Calcd for

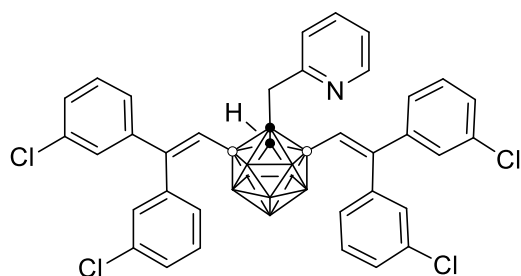
$C_{60}H_{54}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 897.5258, Found: 897.5246.



4m: Yellow solid. Yield: 62%. Mp: 129.1-

131.4 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.72 (d, J = 4.8 Hz, 1H), 7.51 (m, 1H), 7.36 (m, 2H), 7.20 (m, 3H), 7.09 (m, 3H), 7.00

(d, J = 7.6 Hz, 2H), 6.92 (m, 4H), 6.82 (m, 4H) (aromatic CH), 6.19 (s, 2H) (alkenyl CH), 4.47 (s, 1H) (cage CH), 3.67 (s, 2H) (CH_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 162.8 (d, $^1J_{CF}$ = 246.4 Hz), 162.3 (d, $^1J_{CF}$ = 248.5 Hz), 155.5, 154.6, 149.3, 144.3 (d, $^3J_{CF}$ = 7.1 Hz), 141.7 (d, $^3J_{CF}$ = 7.1 Hz), 137.1, 130.0 (d, $^3J_{CF}$ = 9.1 Hz), 129.7 (d, $^3J_{CF}$ = 8.1 Hz), 125.9 (d, $^4J_{CF}$ = 3.0 Hz), 125.3, 123.0, 122.8 (d, $^4J_{CF}$ = 3.0 Hz), 117.0 (d, $^2J_{CF}$ = 21.2 Hz), 115.2 (d, $^2J_{CF}$ = 21.2 Hz), 115.1 (d, $^2J_{CF}$ = 20.8 Hz), 113.8 (d, $^2J_{CF}$ = 22.2 Hz) (alkenyl and aromatic C), 73.9 (cage C), 61.7 (cage CH), 41.5 (CH_2), the $B_{cage}-C$ was not observed. ^{11}B NMR ($CDCl_3$, 128 MHz): δ -4.9 (m, 4B), -10.0 (m, 6B). ^{19}F NMR ($CDCl_3$, 376 MHz): δ -112.7 (m, 2F), -113.3 (m, 2F). HRMS (DART) Calcd for $C_{36}H_{34}^{10}B_2^{11}B_8F_4N^+$ $[M+H]^+$: 664.3625, Found: 664.3609.

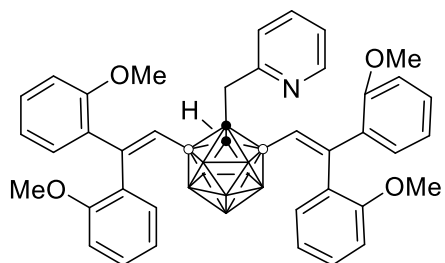


4n: Yellow solid. Yield: 58%. Mp: 137.7-

139.1 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.76 (d, J = 4.8 Hz, 1H), 7.57 (td, J = 7.6, 2.0 Hz, 1H), 7.35 (m, 5H), 7.15 (m, 11H),

6.91 (d, J = 8.0 Hz, 2H) (aromatic CH), 6.16 (s, 2H) (alkenyl CH), 4.53 (s, 1H) (cage CH), 3.66 (s, 2H) (CH_2). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 155.4, 154.5, 149.4, 143.8, 141.2, 137.3, 134.4, 134.3, 130.0, 129.7, 129.5, 128.3, 128.3, 127.0, 125.5, 125.2,

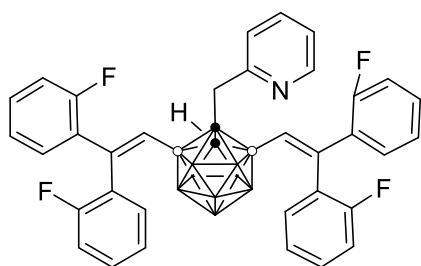
123.1 (alkenyl and aromatic C), 74.1 (cage C), 61.8 (cage CH), 41.4 (CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.7 (m, 4B), -10.2 (m, 6B). HRMS (DART) Calcd for C₃₆H₃₄¹⁰B₂¹¹B₈Cl₄N⁺ [M+H⁺]: 730.2414, Found: 730.2410.



4o: Yellow solid. Yield: 94%. Mp: 96.8-98.5 °C.

¹H NMR (400 MHz, CDCl₃): δ 8.54 (d, *J* = 4.8 Hz, 1H), 7.52 (td, *J* = 8.0, 1.6 Hz, 1H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.17 (m, 9H), 6.85 (m, 8H)

(aromatic CH), 6.22 (s, 2H) (alkenyl CH), 3.82 (s, 2H) (CH₂), 3.67 (s, *J* = 2.0 Hz, 12H) (CH₃), 3.11 (s, 1H) (cage CH). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 157.0, 157.0, 156.2, 151.5, 149.2, 135.9, 133.4, 131.9, 130.5, 130.4, 128.9, 128.5, 125.7, 122.3, 120.5, 120.0, 111.8, 110.8 (alkenyl and aromatic C), 74.6 (cage C), 62.5 (cage CH), 55.7, 55.5 (OCH₃), 40.3 (CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.7 (m, 4B), -11.4 (m, 6B). HRMS (DART) Calcd for C₄₀H₄₆¹⁰B₂¹¹B₈NO₄⁺ [M+H⁺]: 713.4418, Found: 713.4411.

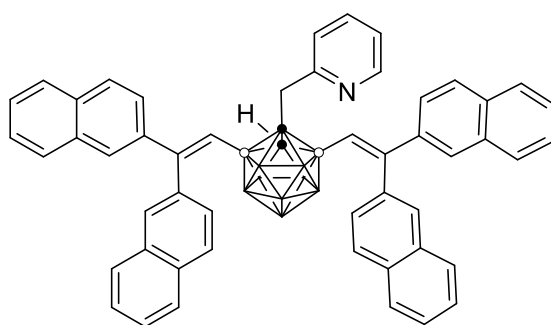


4p: Yellow solid. Yield: 60%. Mp: 145.7-146.3

°C. ¹H NMR (400 MHz, CDCl₃): δ 8.58 (d, *J* = 5.2 Hz, 1H), 7.47 (td, *J* = 7.6, 1.6 Hz, 1H), 7.34 (m, 4H), 7.21 (m, 3H), 7.15 (m, 3H), 7.02 (m, 8H)

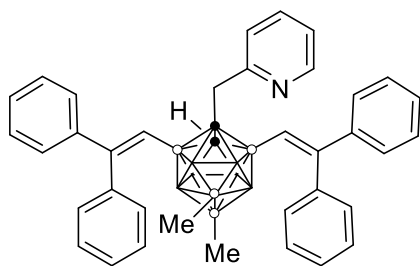
(aromatic CH), 6.25 (s, 2H) (alkenyl CH), 4.36 (s, 1H) (cage CH), 3.81 (s, 2H) (CH₂). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 161.1 (d, ²*J*_{CF} = 18.3 Hz), 158.7 (d, ²*J*_{CF} = 23.3 Hz), 155.5, 149.0, 146.2, 136.5, 132.5 (m), 129.3 (d, ¹*J*_{CF} = 274.1 Hz), 129.2 (d, ¹*J*_{CF} = 279.1 Hz), 130.3 (d, ⁴*J*_{CF} = 3.0 Hz), 130.0 (d, ³*J*_{CF} = 8.1 Hz), 129.5 (d, ³*J*_{CF} = 8.1 Hz),

127.9 (d, $^2J_{\text{CF}} = 17.2$ Hz), 125.6, 124.0 (d, $^3J_{\text{CF}} = 13.5$ Hz), 123.8 (d, $^3J_{\text{CF}} = 13.3$ Hz), 122.5, 116.1 (d, $^2J_{\text{CF}} = 23.2$ Hz), 115.5 (d, $^2J_{\text{CF}} = 22.2$ Hz) (alkenyl and aromatic C), 74.3 (cage C), 62.0 (cage CH), 40.9 (CH_2), the $\text{B}_{\text{cage}}\text{-C}$ was not observed. ^{11}B NMR (CDCl_3 , 128 MHz): δ -4.8 (m, 4B), -10.6 (m, 6B). ^{19}F NMR (CDCl_3 , 376 MHz): δ -113.9 (m, 2F), -114.5 (m, 2F). HRMS (DART) Calcd for $\text{C}_{36}\text{H}_{34}^{10}\text{B}_2^{11}\text{B}_8\text{F}_4\text{N}^+$ [$\text{M}+\text{H}^+$]: 664.3625, Found: 664.3609.



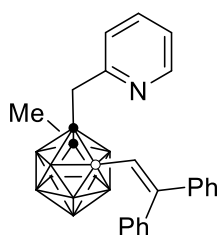
4q: Yellow solid. Yield: 60%. Mp: 132.6-133.4 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.79 (d, $J = 5.2$ Hz, 1H), 7.90 (m, 2H), 7.82 (m, 9H), 7.72 (d, $J = 8.4$ Hz, 2H), 7.64 (d, $J = 7.2$ Hz, 3H),

7.54 (m, 4H), 7.41 (m, 9H), 7.29 (m, 1H), 7.12 (d, $J = 7.6$ Hz, 1H) (aromatic CH), 6.39 (s, 2H) (alkenyl CH), 4.17 (s, 1H) (cage CH), 3.76 (s, 2H) (CH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 126 MHz): δ 157.0, 140.0, 137.6, 133.2, 133.1, 133.1, 132.9, 129.5, 128.6, 128.3, 128.0, 127.9, 127.7, 127.6, 127.3, 126.5, 126.4, 124.9, 122.7 (alkenyl and aromatic C), 74.3 (cage C), 62.3 (cage CH), 41.6 (CH_2), the $\text{B}_{\text{cage}}\text{-C}$ was not observed. ^{11}B NMR (CDCl_3 , 128 MHz): δ -4.3 (m, 4B), -10.4 (m, 6B). HRMS (ESI) Calcd for $\text{C}_{52}\text{H}_{46}^{10}\text{B}_2^{11}\text{B}_8\text{N}^+$ [$\text{M}+\text{H}^+$]: 792.4628, Found: 792.4625.

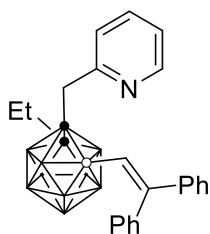


4r: White solid. Yield: 98%. Mp: 183.5-183.9 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.69 (d, $J = 5.2$ Hz, 1H), 7.54 (td, $J = 7.6, 2.0$ Hz, 1H), 7.42 (m, 6H), 7.29 (m, 9H), 7.24 (m, 6H), 7.13 (d, $J = 7.6$ Hz, 1H)

(aromatic *CH*), 6.33 (s, 2H) (alkenyl *CH*), 3.66 (s, 2H) (*CH*₂), 3.37 (s, 1H) (cage *CH*), 0.08 (s, 3H), 0.01 (s, 3H) (*CH*₃). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 156.2, 156.0, 149.2, 142.8, 140.4, 136.5, 130.0, 128.2, 128.2, 128.0, 127.7, 127.2, 125.3, 122.5 (alkenyl and aromatic *C*), 67.1, 55.6 (cage *C*), 40.8 (*CH*₂), the B_{cage}-*C* was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ 5.4 (s, 2B), -8.2 (m, 4B), -11.4 (m, 4B). HRMS (DART) Calcd for C₃₈H₄₂¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 620.4315, Found: 620.4299.

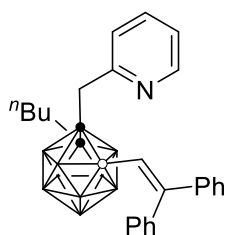


6a: White solid. Yield: 99%. Mp: 171.3-172.2 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.52 (d, *J* = 4.8 Hz, 1H), 7.65 (td, *J* = 7.6, 2.0 Hz, 1H), 7.37 (m, 5H), 7.29 (m, 5H), 7.21 (m, 2H) (aromatic *CH*), 6.46 (s, 1H) (alkenyl *CH*), 3.96 (d, *J* = 15.2 Hz, 1H), 3.49 (d, *J* = 15.2 Hz, 1H) (*CH*₂), 2.19 (s, 3H) (*CH*₃). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 156.0, 156.0, 149.3, 143.3, 140.7, 136.7, 130.4, 128.3, 128.1, 128.0, 127.6, 127.2, 125.3, 122.9 (alkenyl and aromatic *C*), 77.4, 75.7 (cage *C*), 41.3 (*CH*₂), 24.1 (*CH*₃), the B_{cage}-*C* was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -2.6 (s, 1B), -3.9 (d, *J* = 122.9 Hz, 1B), -4.8 (d, *J* = 142.1 Hz, 1B), -10.9 (m, 7B). HRMS (DART) Calcd for C₂₃H₃₀¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 428.3376, Found: 428.3368.

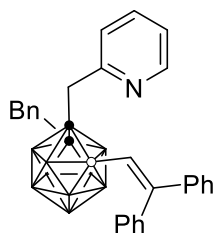


6b: White solid. Yield: 98%. Mp: 146.3-146.7 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.52 (d, *J* = 4.8 Hz, 1H), 7.64 (td, *J* = 7.6, 2.0 Hz, 1H), 7.38 (m, 5H), 7.29 (m, 5H), 7.20 (m, 2H) (aromatic *CH*), 6.47 (s, 1H) (alkenyl *CH*), 3.94 (d, *J* = 15.6 Hz, 1H), 3.50 (d, *J* = 15.2 Hz, 1H) (*CH*₂Py), 2.56 (m, 2H) (*CH*₂*CH*₃), 1.10 (t, *J* = 7.6 Hz, 3H) (*CH*₂*CH*₃). ¹³C{¹H} NMR (CDCl₃, 101 MHz): δ 156.0, 155.9, 149.3, 143.3, 140.7, 136.6, 130.4, 128.3,

128.1, 127.9, 127.6, 127.2, 125.4, 122.9 (alkenyl and aromatic C), 81.7, 79.1 (cage C), 41.0 (CH₂Py), 29.0 (CH₂CH₃), 14.1 (CH₂CH₃), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -2.8 (s, 1B), -4.1 (d, *J* = 148.5 Hz, 2B), -9.4 (m, 2B), -11.1 (m, 5B). HRMS (DART) Calcd for C₂₄H₃₂¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 442.3532, Found: 442.3524.

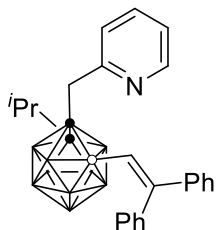


6c: White solid. Yield: 99%. Mp: 90.0-91.3 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.52 (d, *J* = 4.8 Hz, 1H), 7.64 (td, *J* = 7.6, 2.0 Hz, 1H), 7.38 (m, 5H), 7.30 (m, 5H), 7.20 (m, 2H) (aromatic CH), 6.47 (s, 1H) (alkenyl CH), 3.94 (d, *J* = 15.2 Hz, 1H), 3.53 (d, *J* = 15.2 Hz, 1H) (CH₂Py), 2.56 (m, 1H), 2.41 (m, 1H), 1.39 (m, 4H) (alkyl CH₂), 0.93 (t, *J* = 7.2 Hz, 3H) (alkyl CH₃). ¹³C {¹H} NMR (CDCl₃, 101 MHz): δ 156.0, 155.8, 149.2, 143.3, 140.7, 136.5, 130.3, 128.3, 128.1, 127.9, 127.5, 127.2, 125.3, 122.8 (alkenyl and aromatic C), 80.9, 79.2 (cage C), 41.1 (CH₂Py), 35.1, 31.8, 22.5 (alkyl CH₂), 13.9 (alkyl CH₃), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -4.5 (m, 3B), -9.5 (m, 2B), 11.2 (m, 5B). HRMS (DART) Calcd for C₂₆H₃₆¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 470.3845, Found: 470.3835.

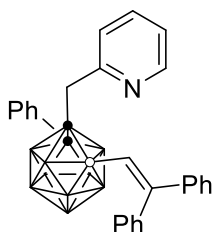


6d: White solid. Yield: 98%. Mp: 145.5-146.2 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.60 (d, *J* = 5.2 Hz, 1H), 7.70 (td, *J* = 7.6, 2.0 Hz, 1H), 7.40 (m, 5H), 7.30 (m, 9H), 7.20 (m, 3H) (aromatic CH), 6.48 (s, 1H) (alkenyl CH), 4.09 (d, *J* = 10.8 Hz, 1H), 4.05 (d, *J* = 10.8 Hz, 1H), 3.77 (d, *J* = 14.0 Hz, 2H) (CH₂). ¹³C {¹H} NMR (CDCl₃, 101 MHz): δ 156.2, 156.1, 149.2, 143.3, 140.6, 136.8, 136.2, 130.4, 130.3, 128.5, 128.3, 128.2, 128.0, 127.7, 127.6, 127.2, 125.4, 123.0 (alkenyl and aromatic C), 80.2, 79.7 (cage C), 41.3, 40.7

(CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -3.7 (m, 3B), -8.9 (m, 2B), -10.9 (m, 5B). HRMS (DART) Calcd for C₂₉H₃₄¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 504.3689, Found: 504.3679.



6e: White solid. Yield: 95%. Mp: 129.0-129.7 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.50 (d, *J* = 4.8 Hz, 1H), 7.63 (td, *J* = 7.6, 1.6 Hz, 1H), 7.38 (m, 5H), 7.30 (m, 5H), 7.19 (m, 2H) (aromatic CH), 6.44 (s, 1H) (alkenyl CH), 3.94 (d, *J* = 15.6 Hz, 1H), 3.53 (d, *J* = 15.2 Hz, 1H) (CH₂), 3.08 (m, 1H) (CH(CH₃)₂), 1.15 (d, *J* = 6.8 Hz, 3H), 1.07 (d, *J* = 6.8 Hz, 3H) (CH(CH₃)₂). ¹³C {¹H} NMR (CDCl₃, 101 MHz): δ 155.9, 155.8, 148.9, 143.3, 140.7, 136.9, 130.4, 128.3, 128.1, 128.0, 127.6, 127.2, 125.6, 123.0 (alkenyl and aromatic C), 87.3, 81.6 (cage C), 40.8 (CH₂), 31.0 (CH(CH₃)₂), 24.4, 24.3 (CH(CH₃)₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -3.0 (m, 2B), -5.0 (d, *J* = 204.8 Hz, 1B), -8.9 (d, *J* = 157.4 Hz, 1B), -10.7 (m, 3B), -12.6 (m, 3B). HRMS (DART) Calcd for C₂₅H₃₄¹⁰B₂¹¹B₈N⁺ [M+H⁺]: 456.3689, Found: 456.3679.

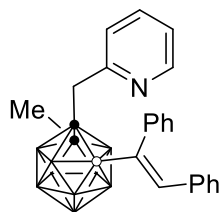


6f: White solid. Yield: 95%. Mp: 149.7-150.9 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.50 (d, *J* = 4.0 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.49 (m, 2H), 7.40 (m, 7H), 7.29 (m, 5H), 7.13 (t, *J* = 6.4 Hz, 1H), 6.74 (d, *J* = 8.0 Hz, 1H) (aromatic CH), 6.60 (s, 1H) (alkenyl CH), 3.70 (d, *J* = 14.8 Hz, 1H), 2.97 (d, *J* = 14.8 Hz, 1H) (CH₂). ¹³C {¹H} NMR (CDCl₃, 101 MHz): δ 155.4, 155.3, 149.5, 143.3, 140.8, 136.1, 131.6, 131.1, 130.8, 130.4, 128.9, 128.2, 128.1, 127.7, 127.4, 127.2, 125.0, 122.6 (alkenyl and aromatic C), 84.1, 80.8 (cage C), 40.8 (CH₂), the B_{cage}-C was not observed. ¹¹B NMR (CDCl₃, 128 MHz): δ -

2.5 (m, 4B), -10.3 (m, 6B). HRMS (DART) Calcd for $C_{28}H_{32}^{10}B_2^{11}B_8N^+$ $[M+H]^+$: 490.3532, Found: 490.3525.

Synthesis of 8.

An oven-dried Schlenk flask equipped with a stir bar was charged with 1-(2-picolyl)-2-methyl-*o*-carborane (**5a**) (49.8 mg, 0.2 mmol), diphenylacetylene (**2a**) (89.0 mg, 0.5 mmol), $[Ir(COD)Cl]_2$ (3.4 mg, 0.005 mmol), $AgNTf_2$ (3.9 mg, 0.01 mmol) and HOAc (6.0 mg, 0.1 mmol), followed by dry toluene (3 mL). The flask was closed under an atmosphere of nitrogen and stirred at 130 °C for 12 h. After the addition of water (5 mL) and extraction with diethyl ether (5 mL x 3), the ether solutions were combined and concentrated to dryness in vacuo. The residue was subjected to flash column chromatography on silica gel (200-300 mesh) using *n*-hexane and ethyl acetate (10/1 in v/v) as eluent to give product **8** as a white solid (46.1 mg, 54%).



8: Mp: 138.5-139.1 °C. 1H NMR (400 MHz, $CDCl_3$): δ 8.51 (d, J = 4.8 Hz, 1H), 7.58 (td, J = 7.6, 2.0 Hz, 1H), 7.38 (m, 2H), 7.31 (m, 3H), 7.19 (m, 5H), 7.03 (m, 2H), 6.80 (d, J = 7.6 Hz, 1H) (alkenyl CH and aromatic CH), 4.04 (d, J = 15.2 Hz, 1H), 3.52 (d, J = 15.6 Hz, 1H) (CH_2), 2.33 (s, 3H) (CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 101 MHz): δ 156.1, 149.2, 143.3, 142.3, 137.5, 136.7, 129.8, 129.0, 128.8, 128.0, 127.3, 126.5, 125.0, 122.8 (alkenyl and aromatic C), 77.6, 76.8 (cage C), 40.5 (CH_2), 23.9 (CH_3), the $B_{cage}-C$ was not observed. ^{11}B NMR ($CDCl_3$, 128 MHz): δ 0.86 (s, 1B), -3.1 (d, J = 151.0 Hz, 1B), -4.5 (d, J = 152.3 Hz, 1B), -10.9 (m, 7B). HRMS (DART) Calcd for $C_{23}H_{30}^{10}B_2^{11}B_8N^+$

[M+H⁺]: 428.3374, Found: 428.3376.

X-ray Structure Determination. The data of **4a** (180 K), **6e** (173 K) and **8** (193 K) were collected at different temperatures on a Bruker APEX DUO diffractometer. An empirical absorption correction was applied using the SADABS program.⁴ All structures were solved by direct methods and subsequent Fourier difference techniques and refined anisotropically for all non-hydrogen atoms by full-matrix least-squares on F^2 using the SHELXTL program package.⁵ All hydrogen atoms were geometrically fixed using the riding model. Crystal data and details of data collection and structure refinements were given in Table S1.

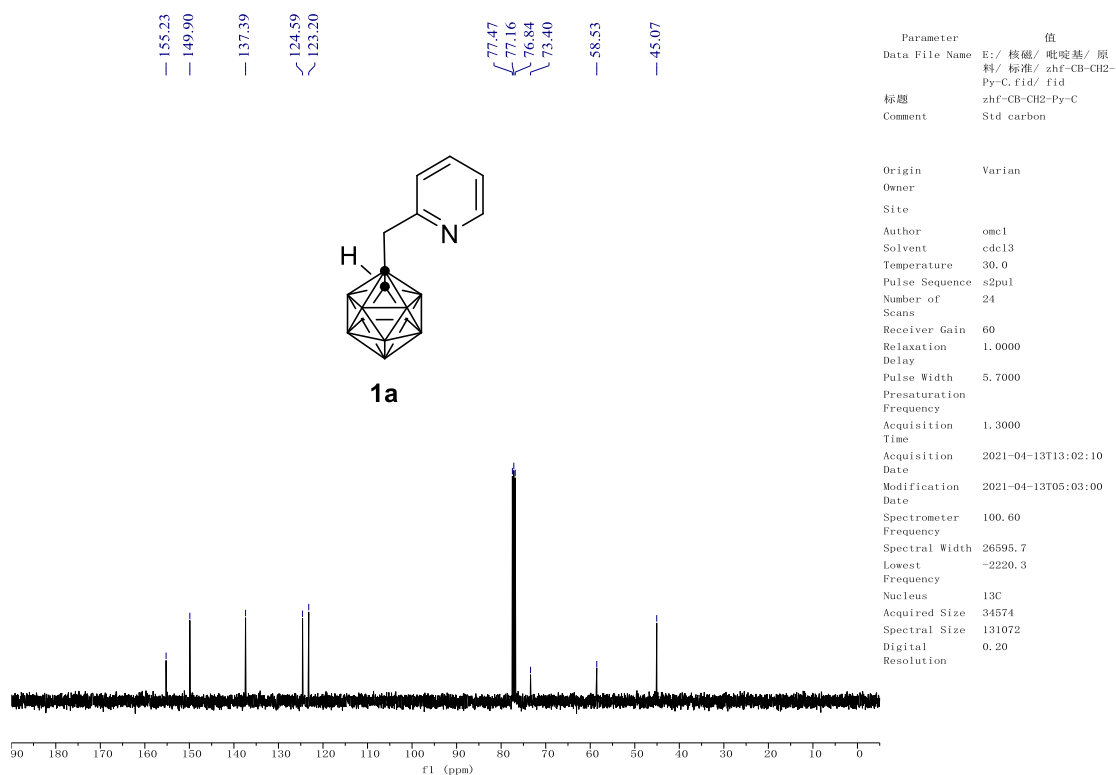
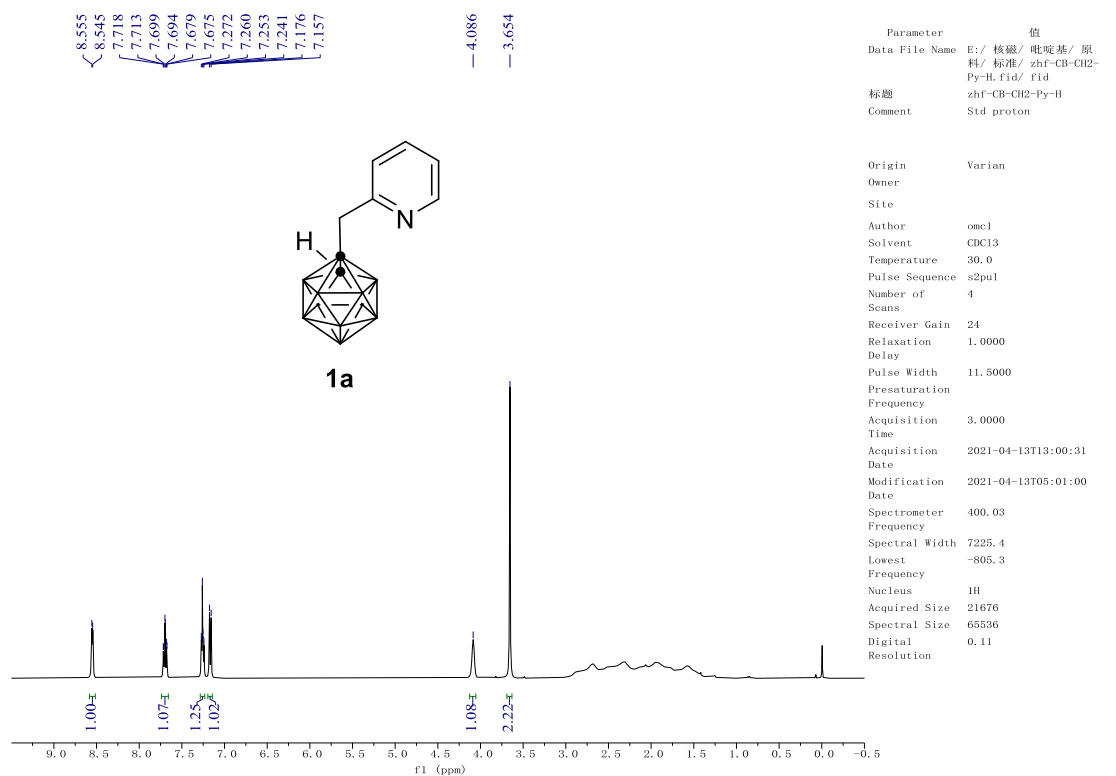
CCDC 2215404-2215406 (**4a**, **6e** and **8**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

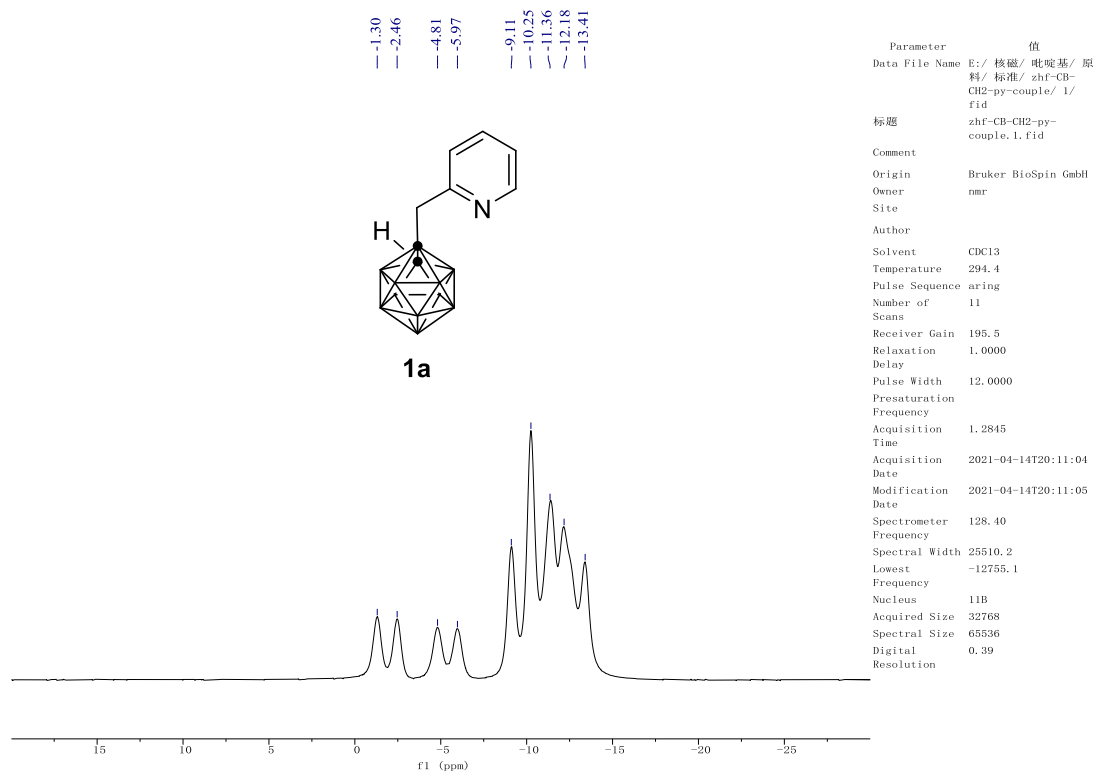
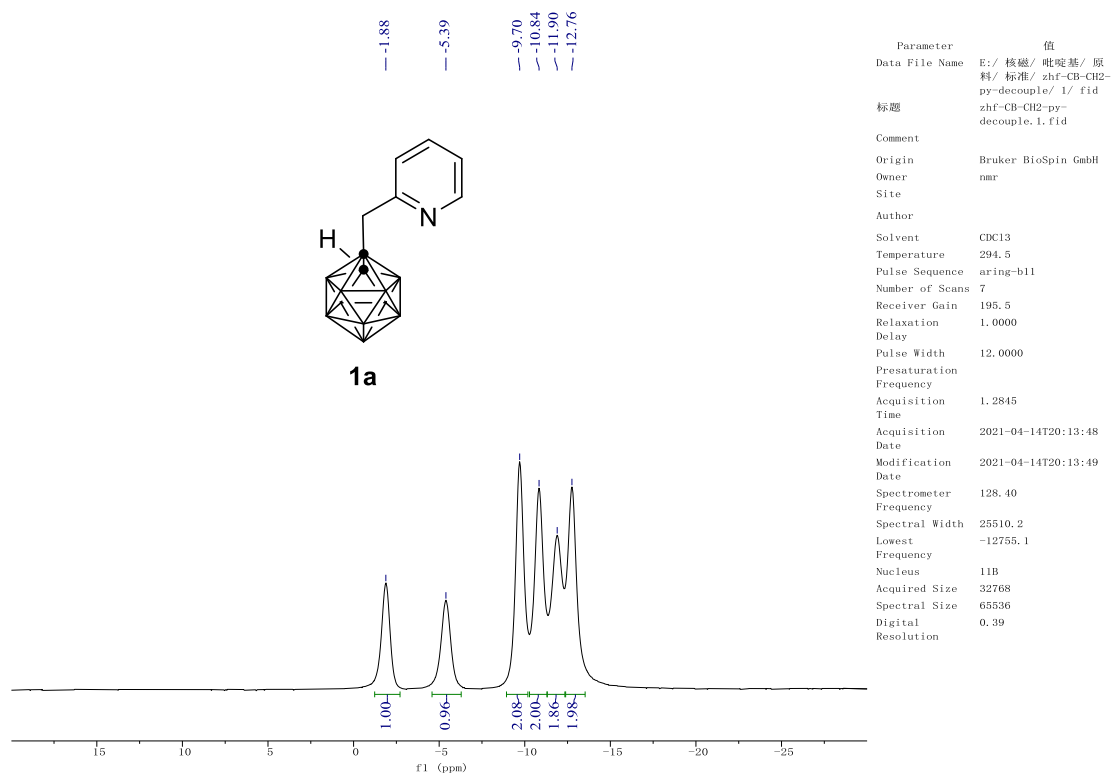
Table S1. Crystal Data and Summary of Data Collection and Refinements.

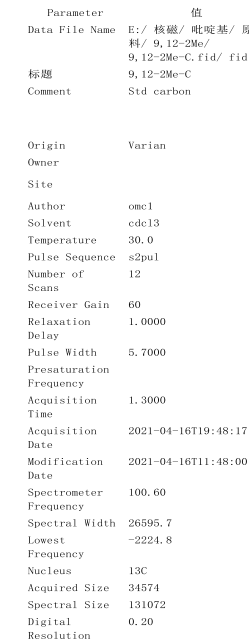
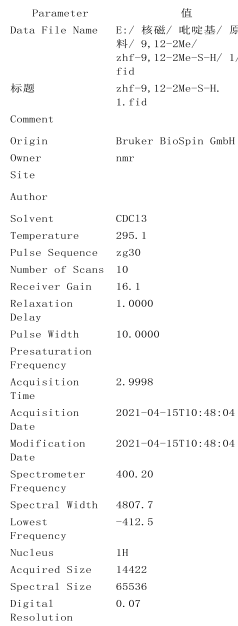
compound	4a	6e	8
formula	C ₃₆ H ₃₇ B ₁₀ N	C ₂₅ H ₃₃ B ₁₀ N	C ₂₃ H ₂₉ B ₁₀ N
crystal size (mm)	0.12 x 0.05 x 0.03	0.17 x 0.15 x 0.12	0.05 x 0.03 x 0.02
fw	591.76	455.62	427.57
crystal system	Triclinic	Monoclinic	Triclinic
space group	<i>P</i> -1	<i>P</i> 2 ₁ /c	<i>P</i> -1
<i>a</i> , Å	10.327(1)	10.718(1)	8.172(1)
<i>b</i> , Å	10.604(1)	18.015(1)	11.935(1)
<i>c</i> , Å	16.150(1)	13.762(1)	13.559(1)
α , deg	85.701(1)	90	108.242(5)
β , deg	81.211(1)	92.277(2)	94.126(5)
γ , deg	68.369(1)	90	99.211(5)
<i>V</i> , Å ³	1624.3(1)	2655.1(1)	1229.3(2)
<i>Z</i>	2	8	2
<i>D</i> _{calcd} , Mg/m ³	1.210	1.140	1.155
radiation (λ) Å	1.34139	1.34139	1.34139
2 θ range, deg	8.8 to 110.0	7.2 to 109.9	6.0 to 110.4
μ , mm ⁻¹	0.306	0.278	0.281
<i>F</i> (000)	620	960	448
no. of obsd reflns	6105	5039	4656
no. of params refnd	424	327	308
goodness of fit	1.045	1.042	1.072
R1	0.0509	0.0517	0.0636
wR2	0.1315	0.1296	0.1661

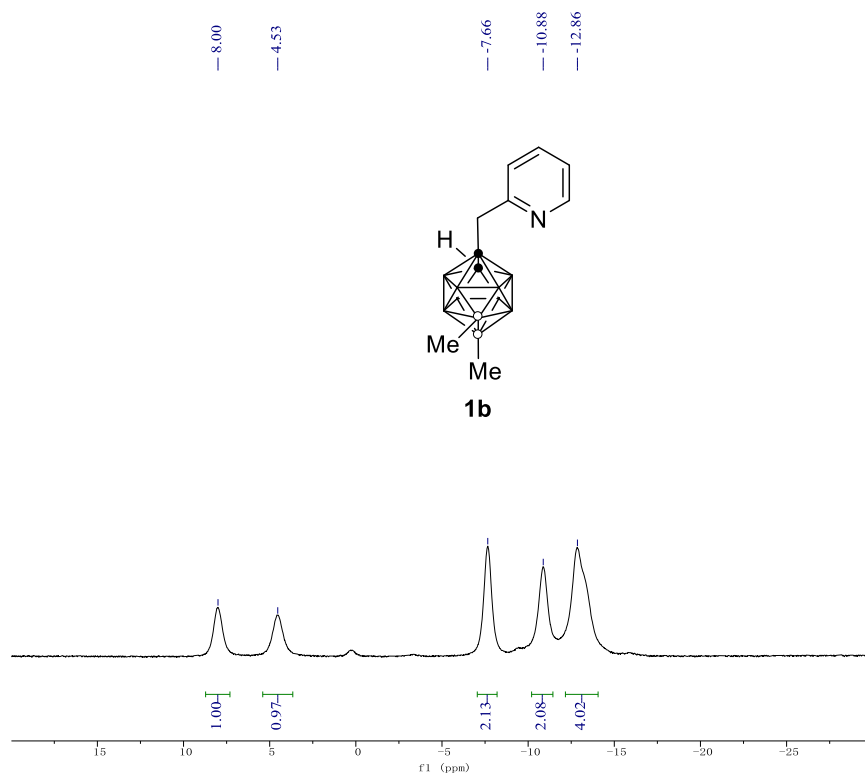
References

- 1 E. S. Alekseyeva, A. S. Batsanov, L. A. Boyd, M. A. Fox, T. G. Hibbert, J. A. K. Howard, J. A. H. MacBride, A. Mackinnon and K. Wade, *Dalton Trans.* **2003**, 475-482.
- 2 K. Park, G. Bae, J. Moon, J. Choe, K. H. Song and S. Lee, *J. Org. Chem.* **2010**, 75, 6244-6251.
- 3 J. C. Axtell, K. O. Kirlikovali, P. I. Djurovich, D. Jung, V. T. Nguyen, B. Munekiyo, A. T. Royappa, A. L. Rheingold and A. M. Spokoyny, *J. Am. Chem. Soc.* **2016**, 138, 15758-15765.
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- 5 G. M. Sheldrick, SHELXTL 5.10 for Windows NT: Structure Determination Software Programs. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, **1997**.

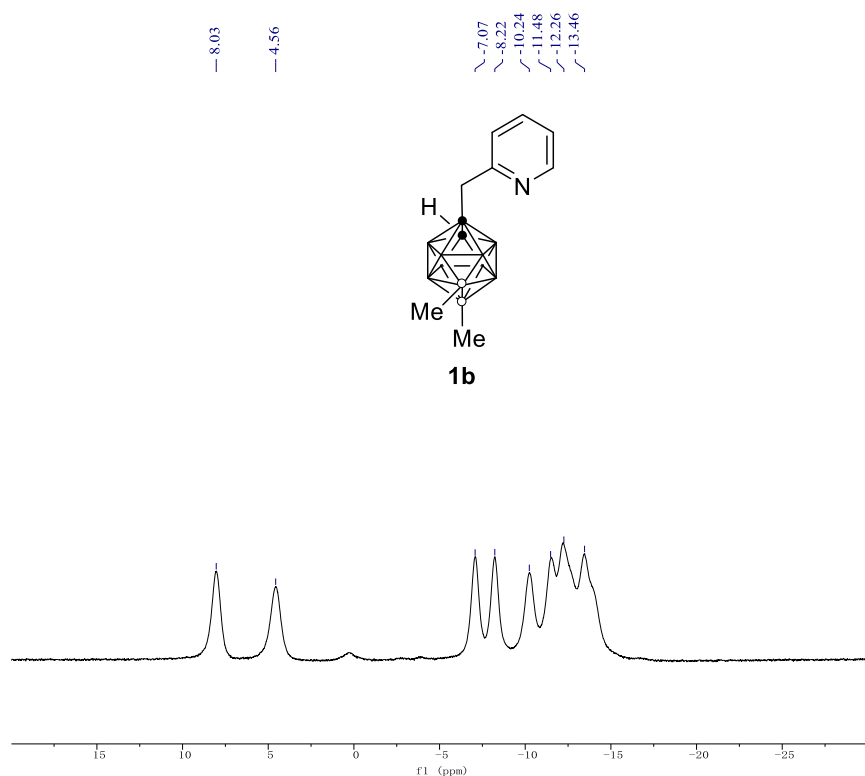




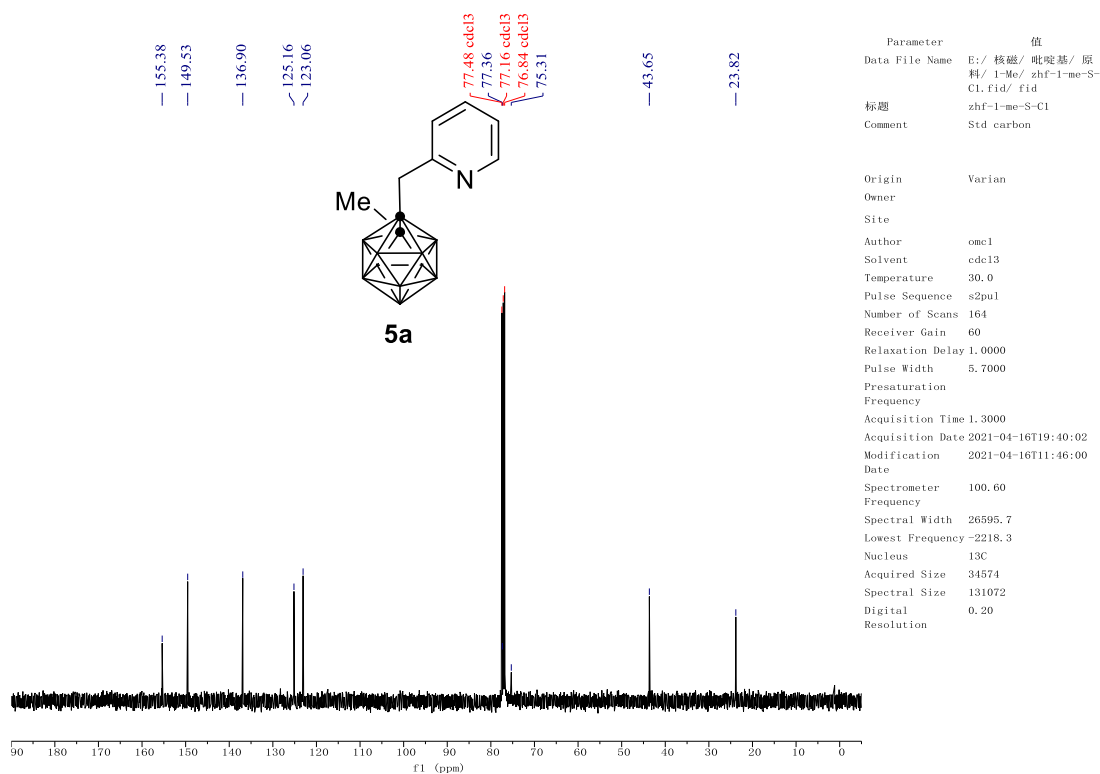
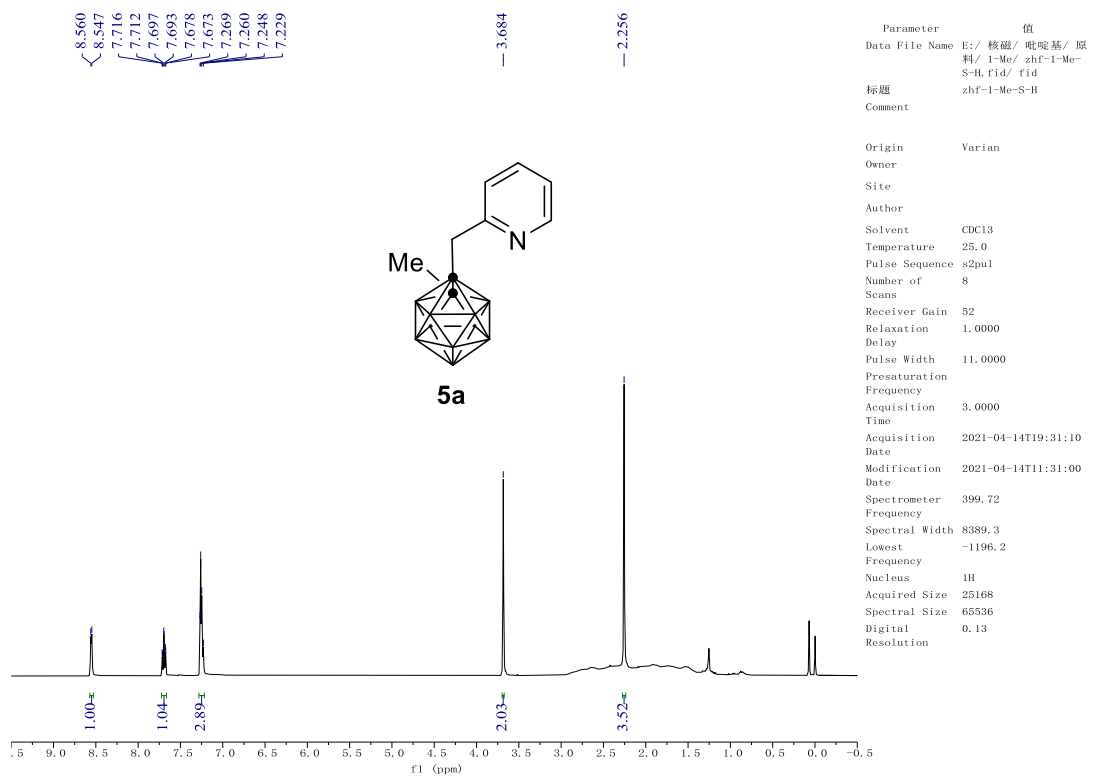


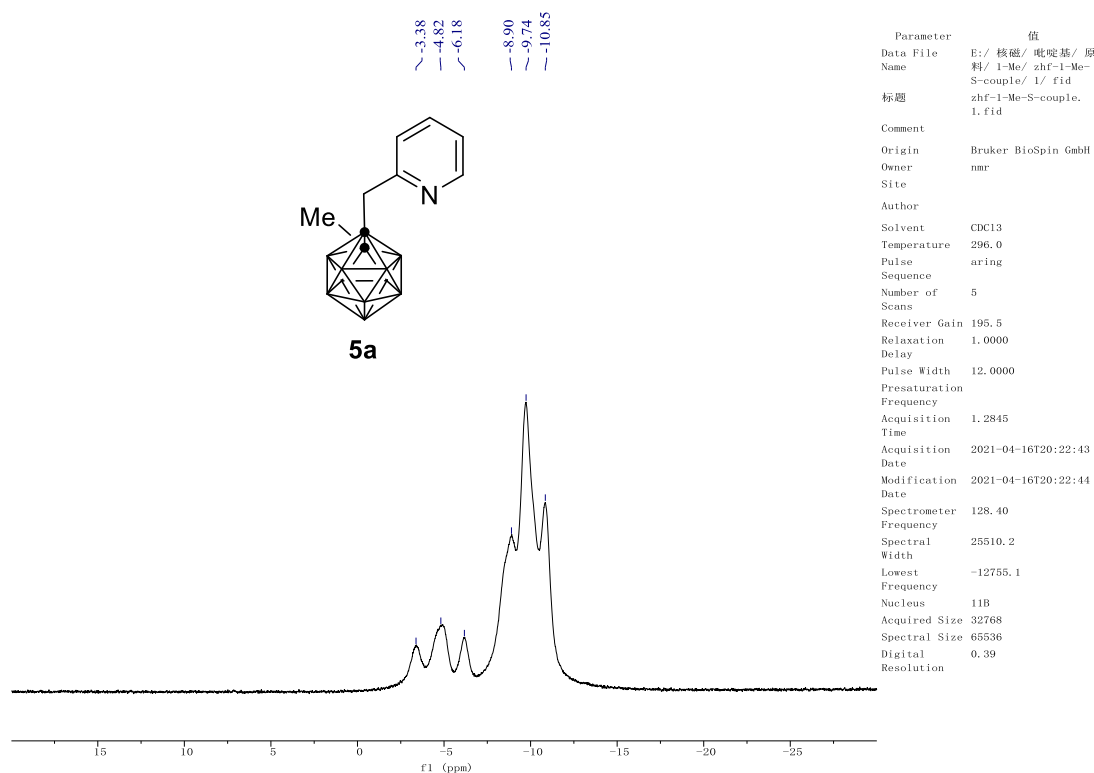
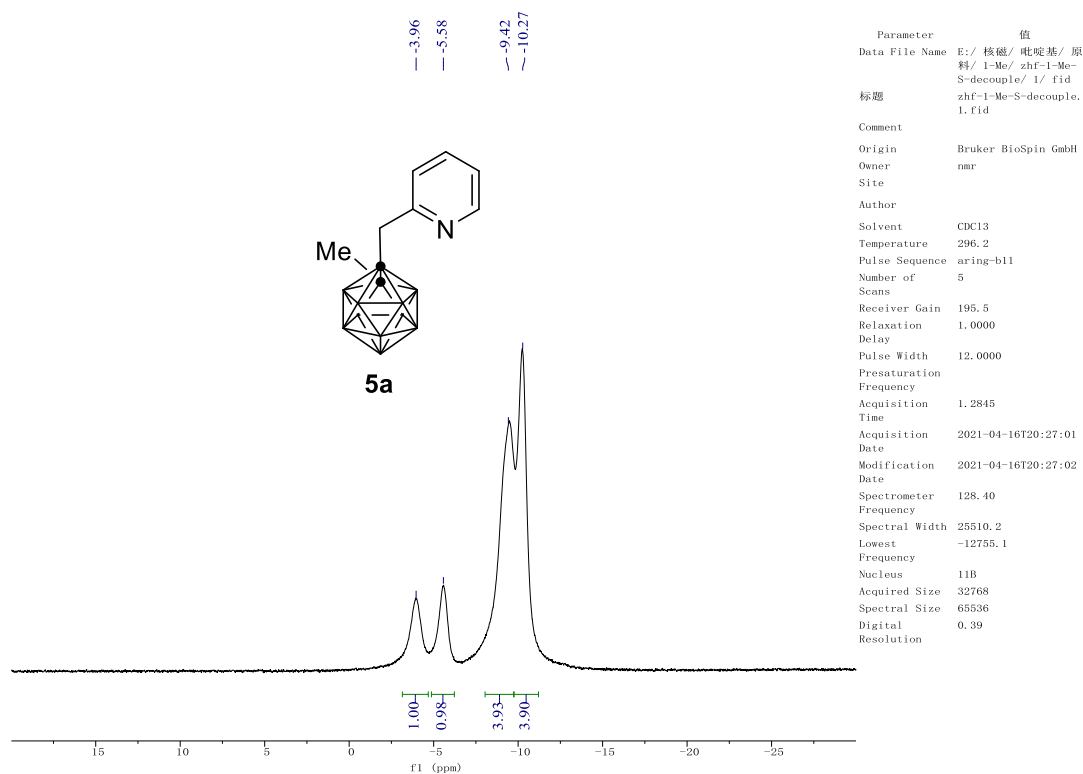


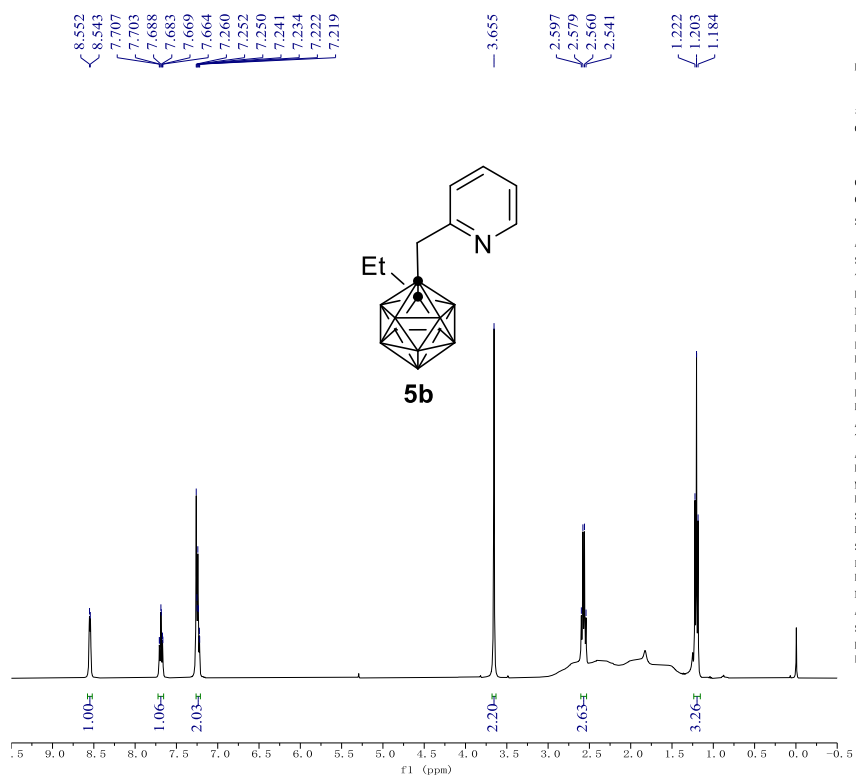
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Author	
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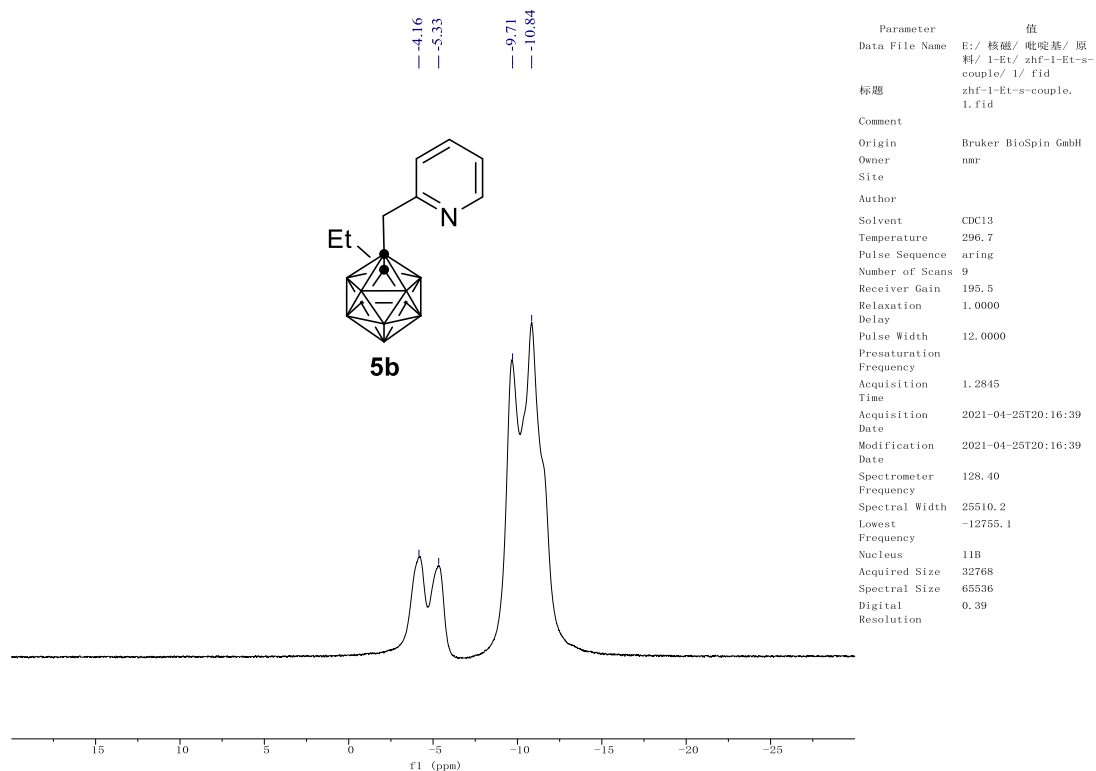
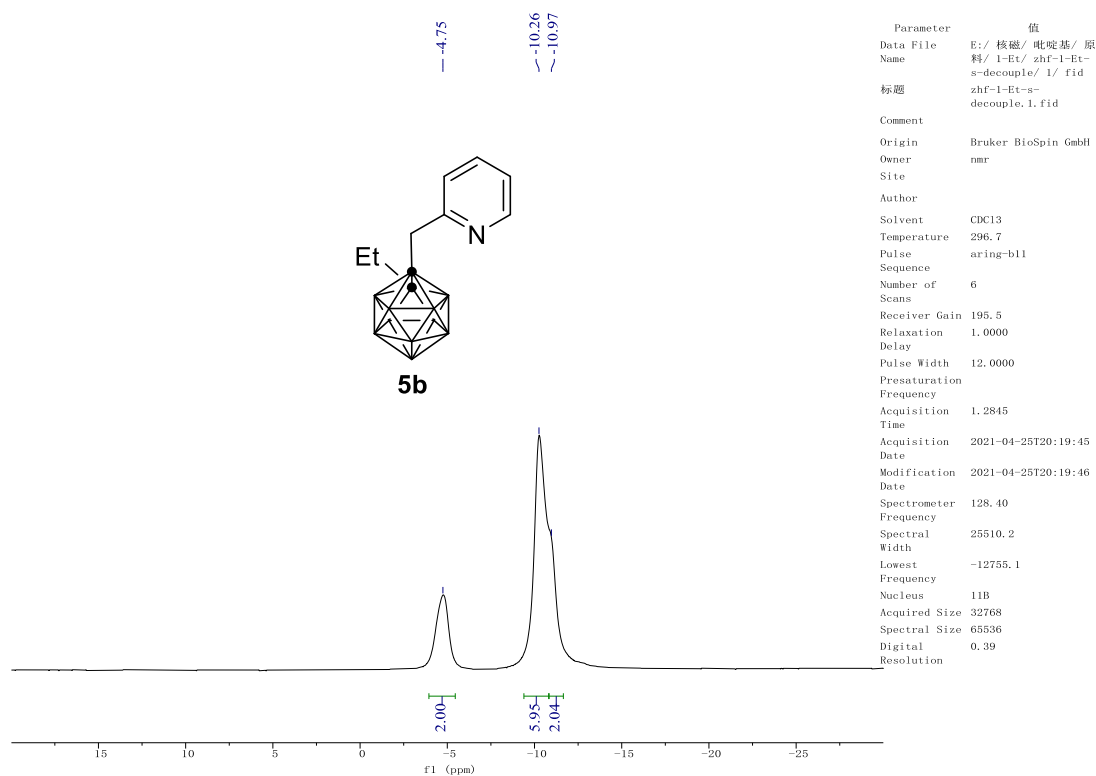


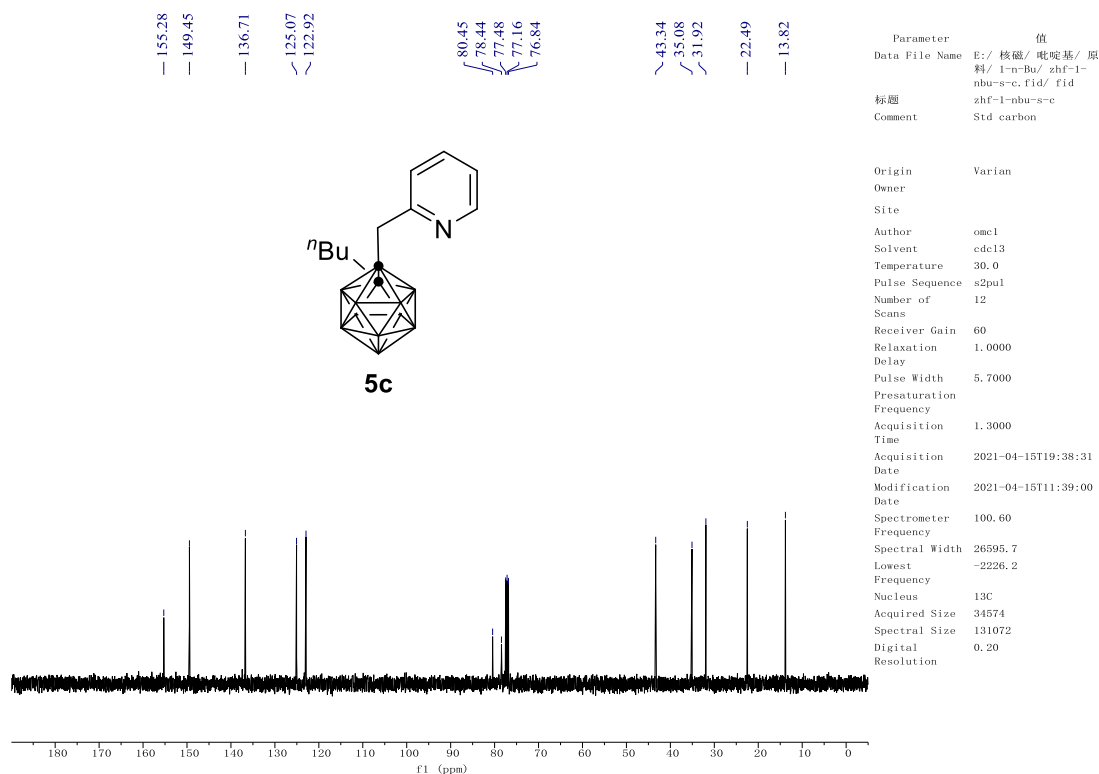
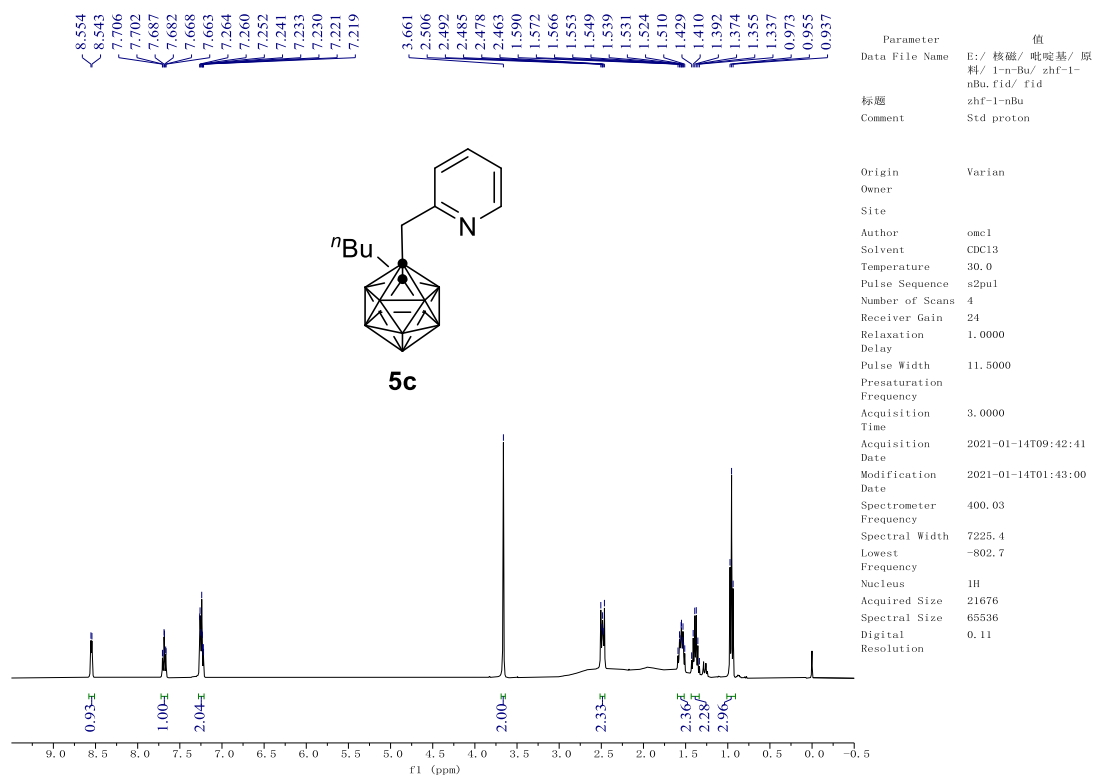
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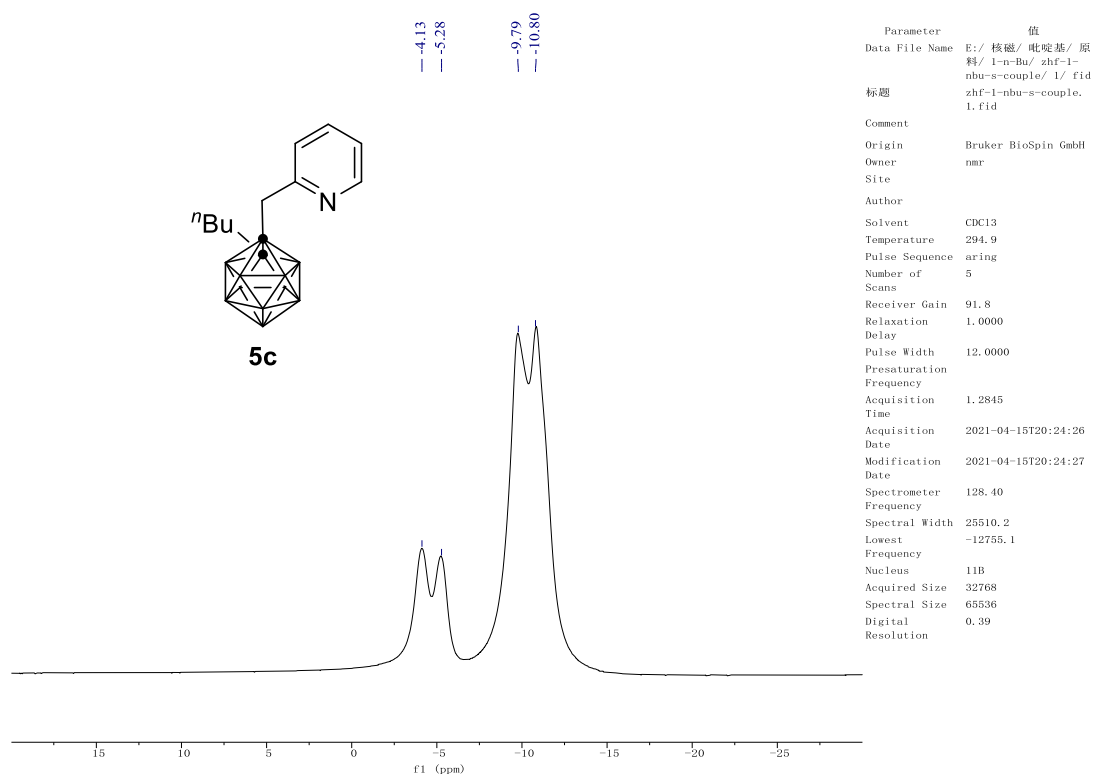
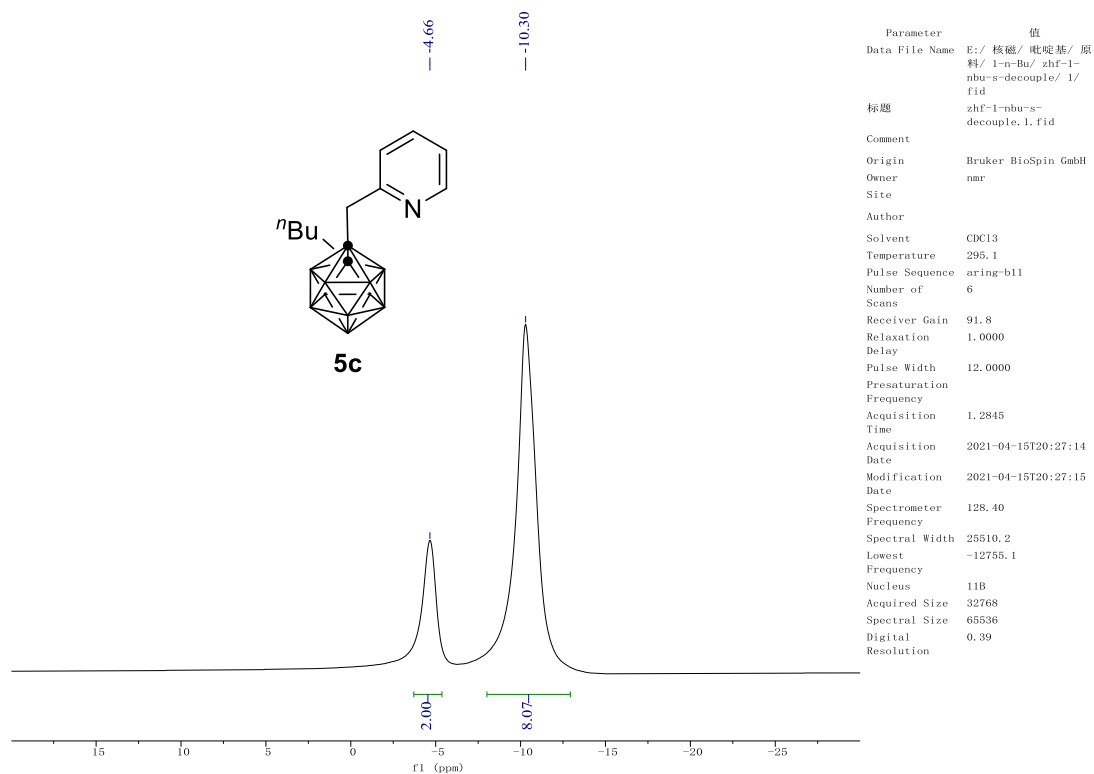


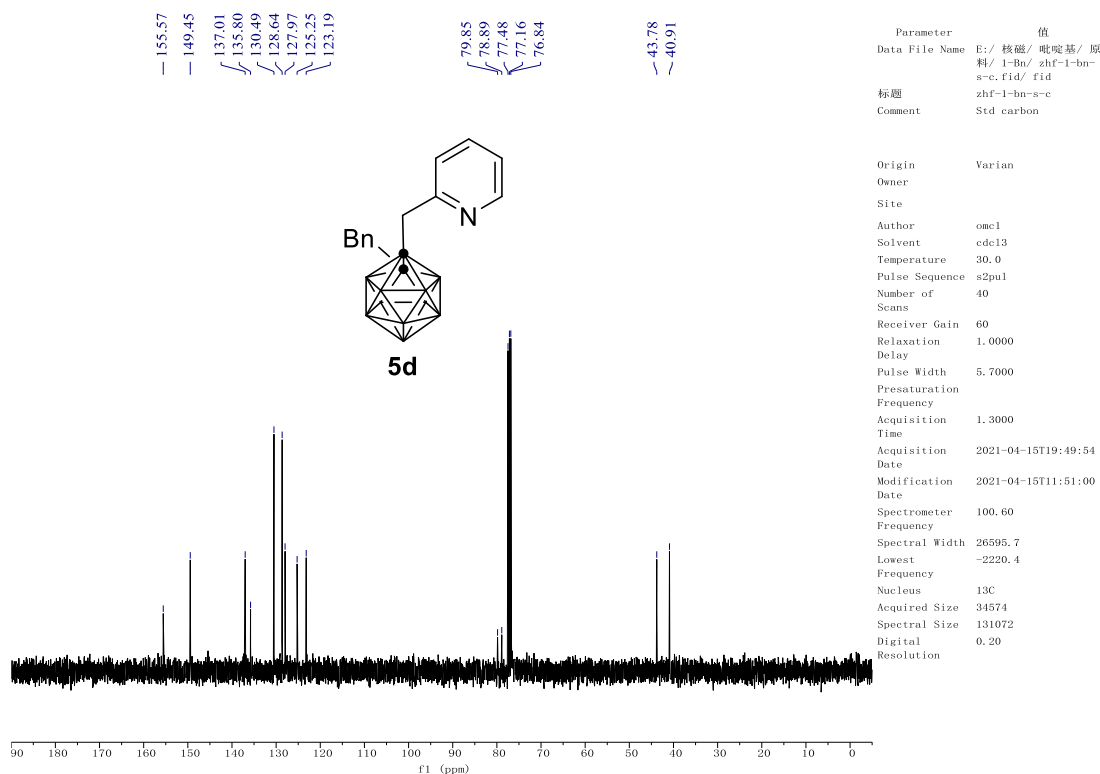
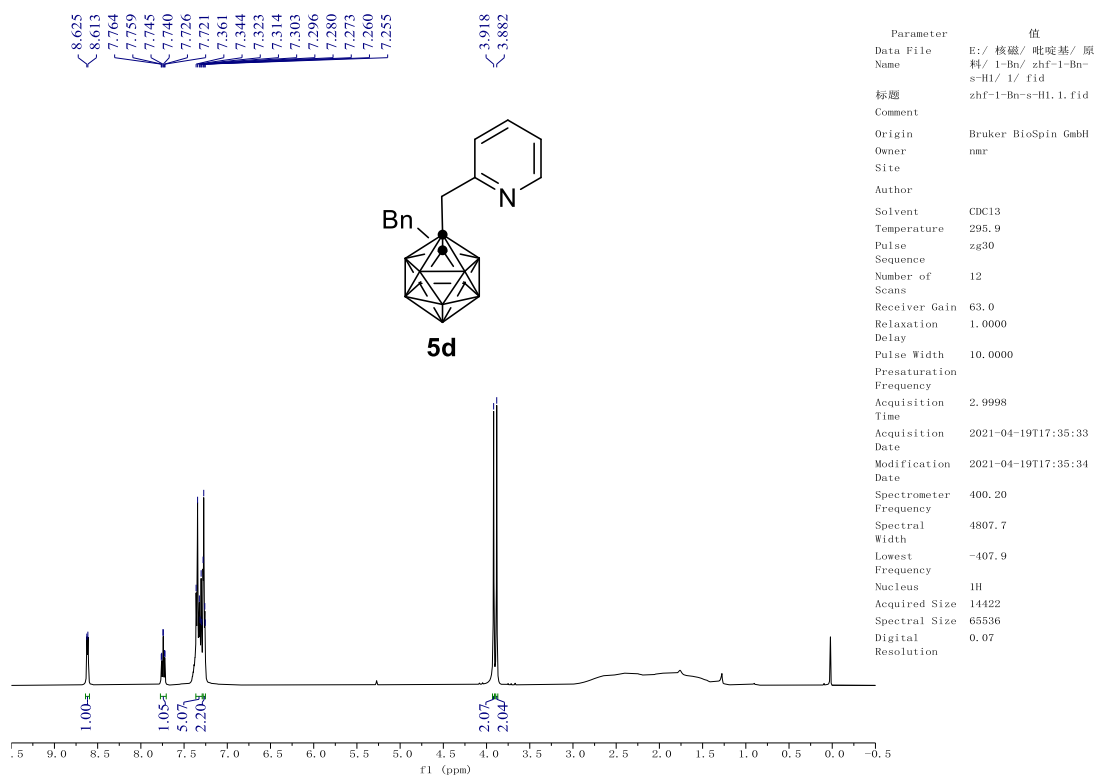


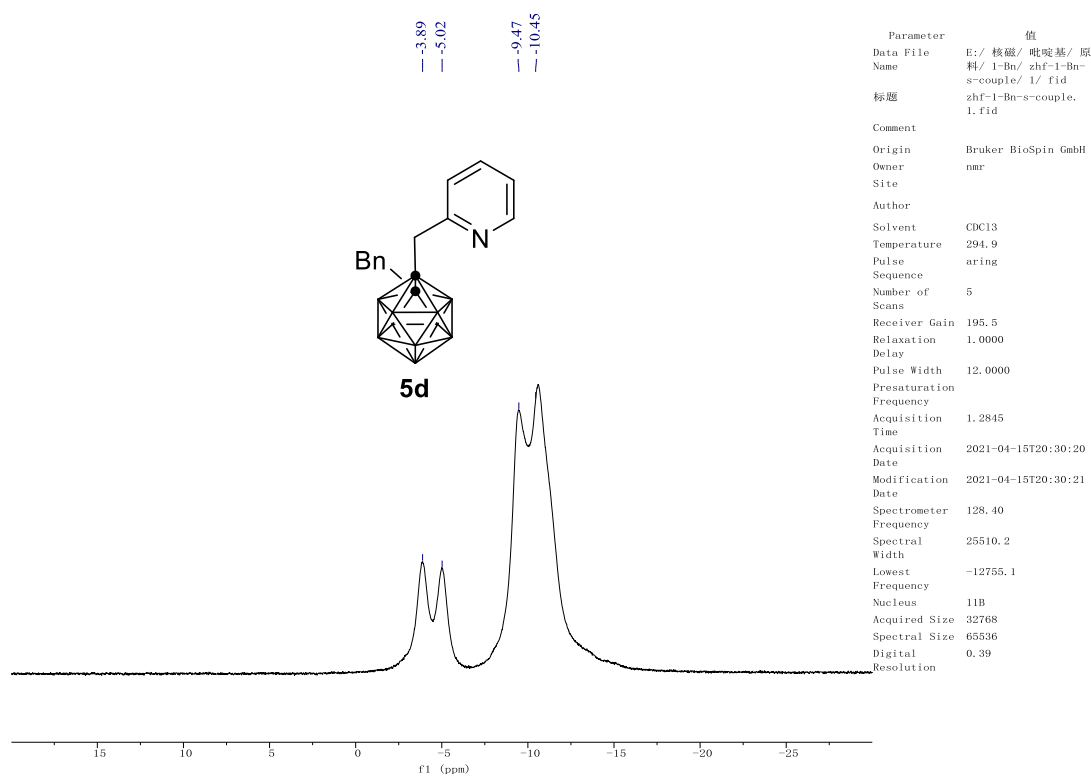
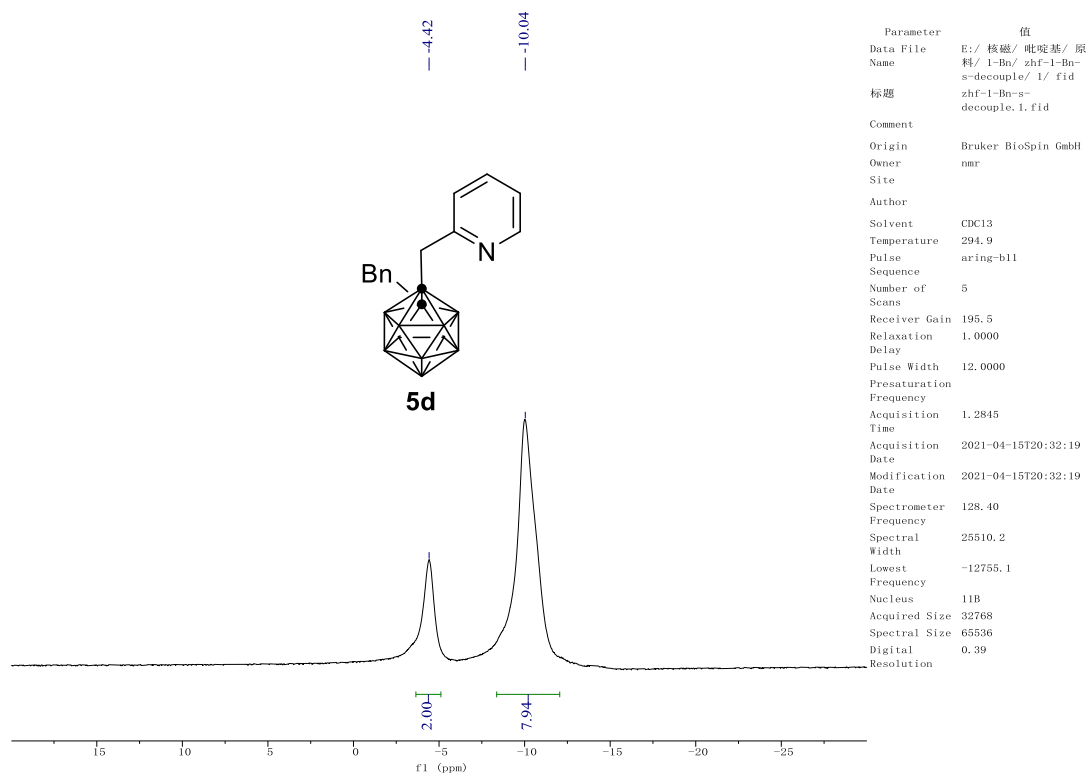


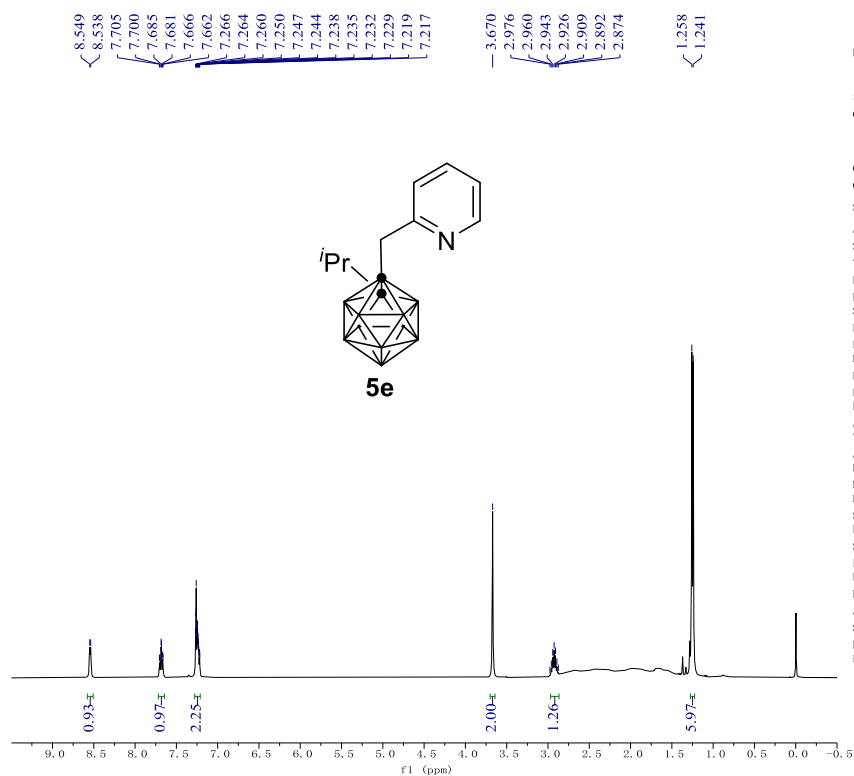






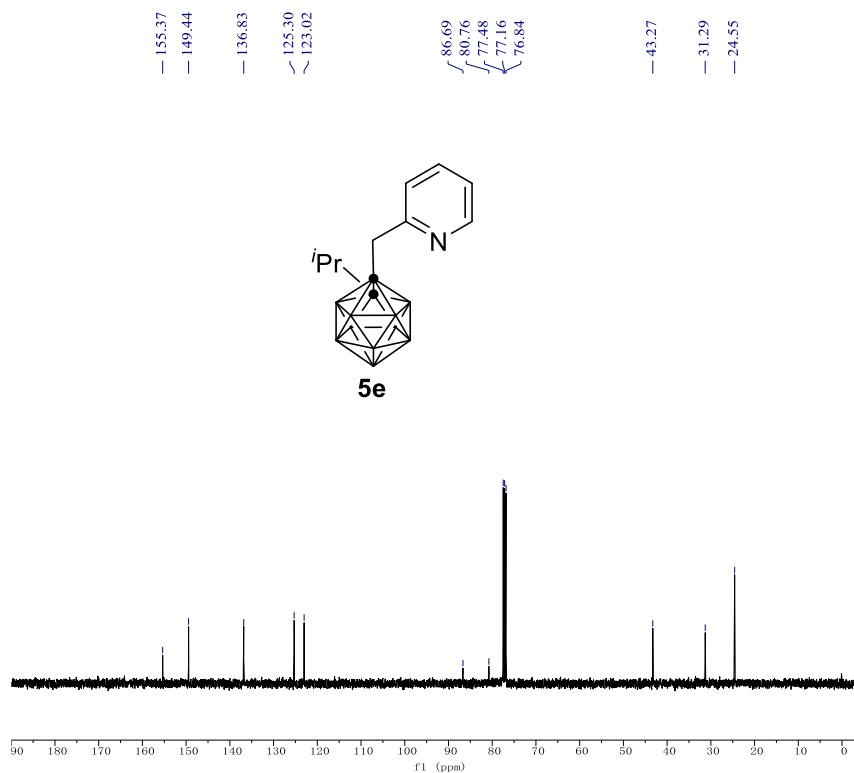






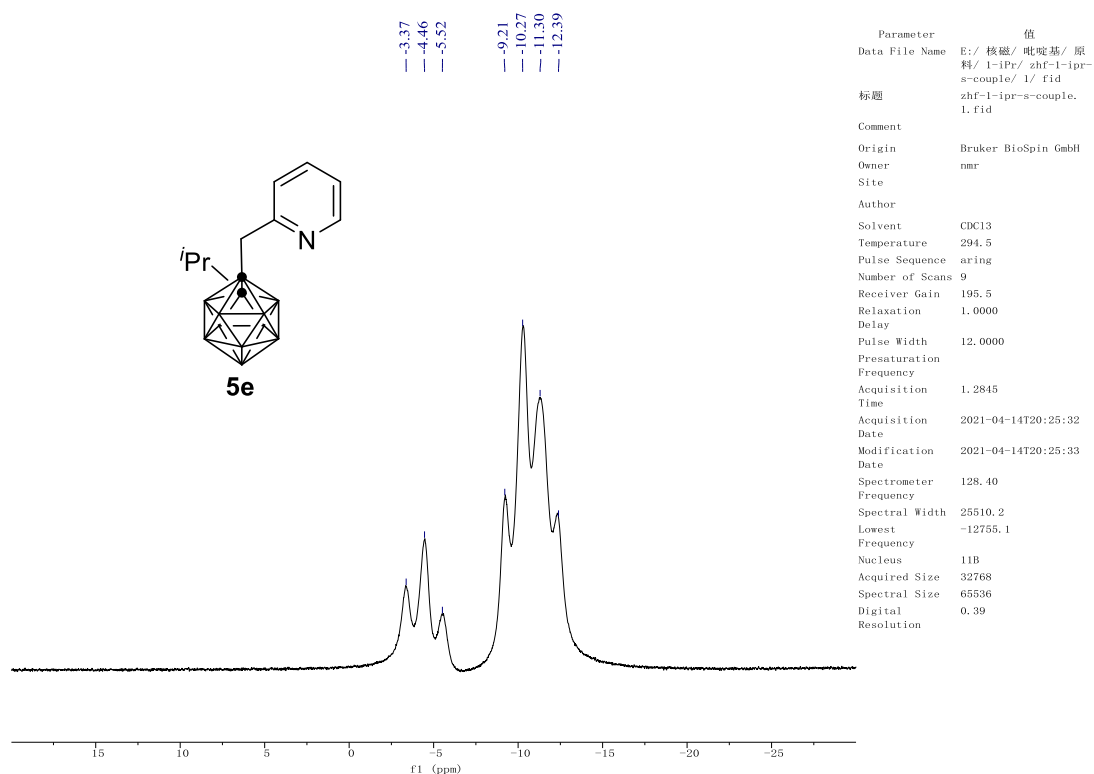
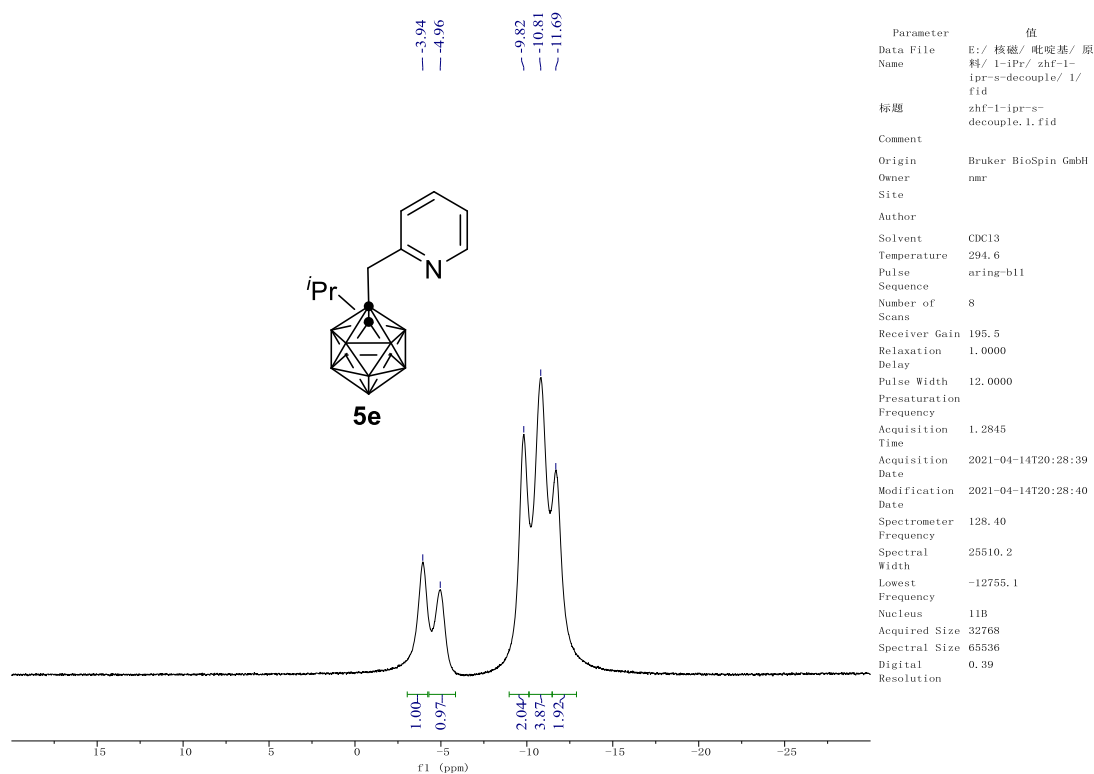
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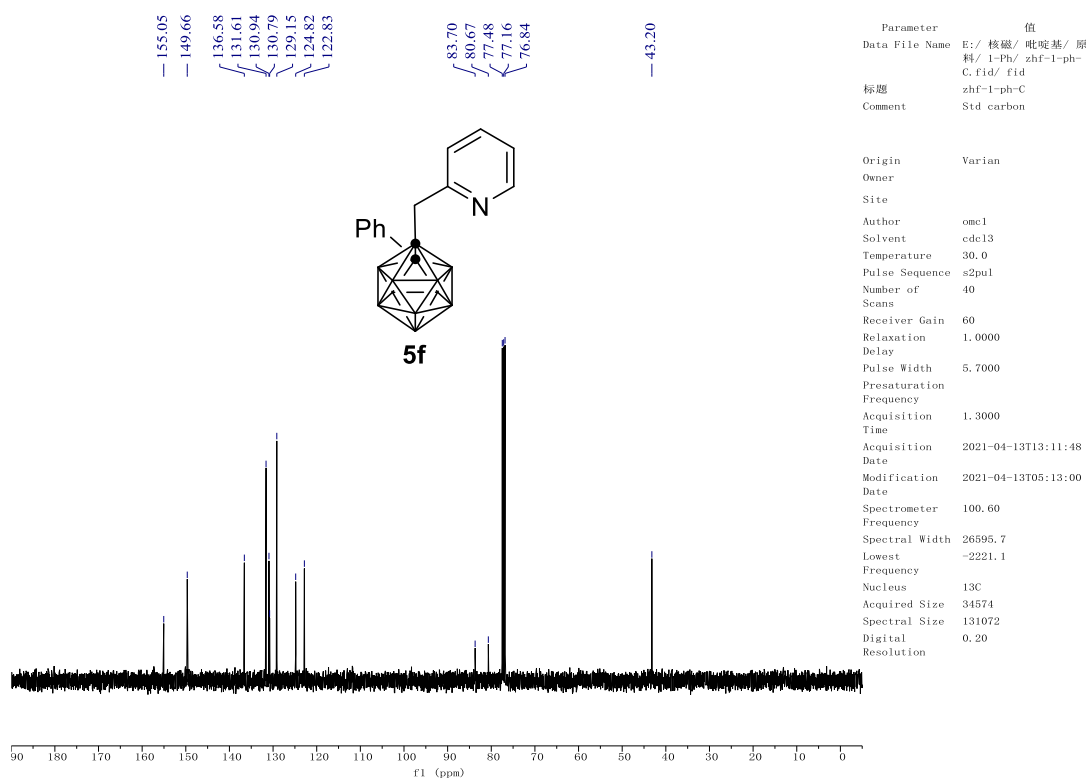
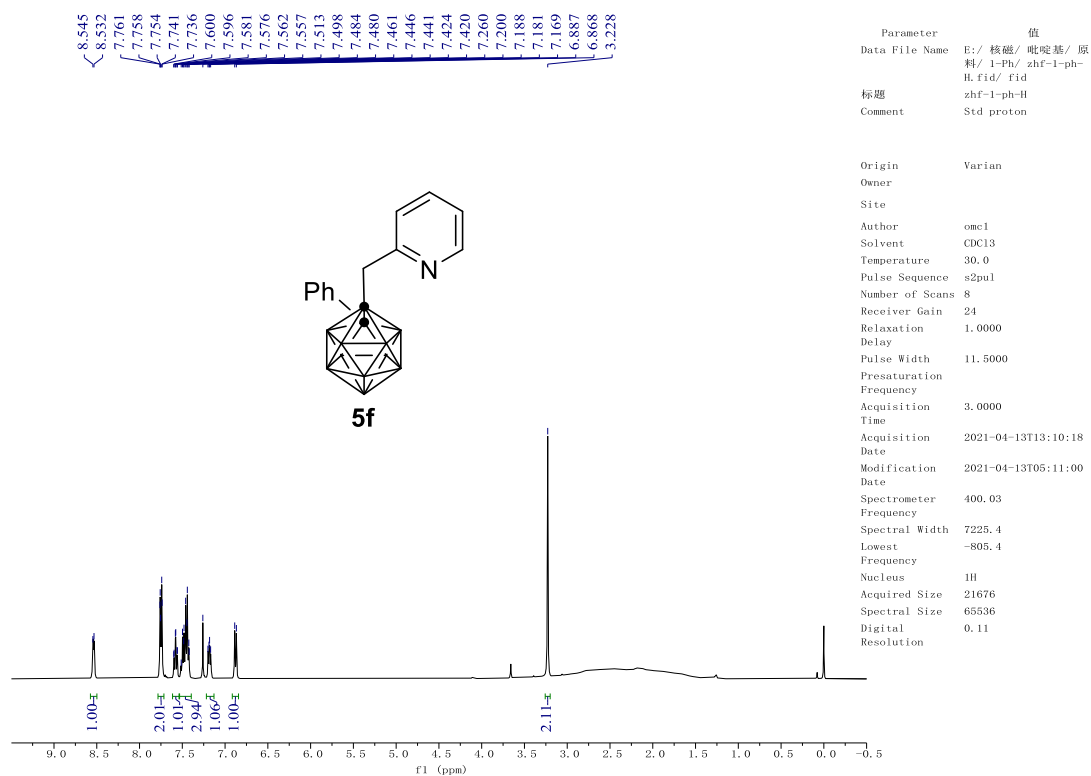
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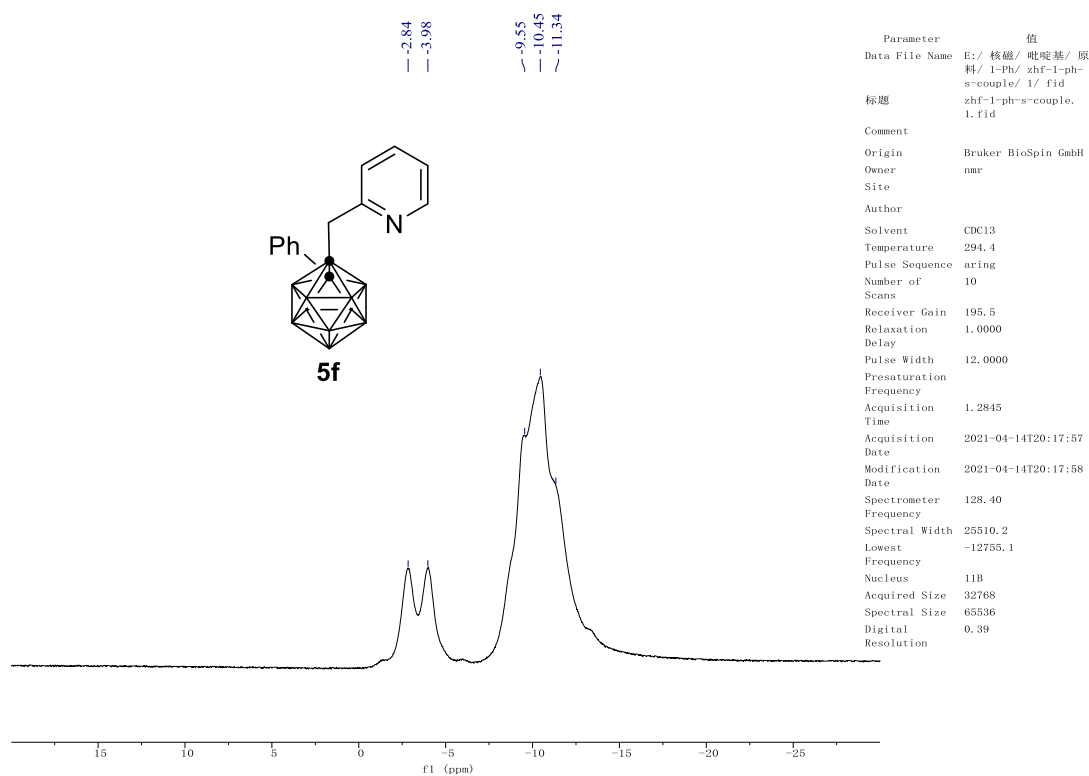
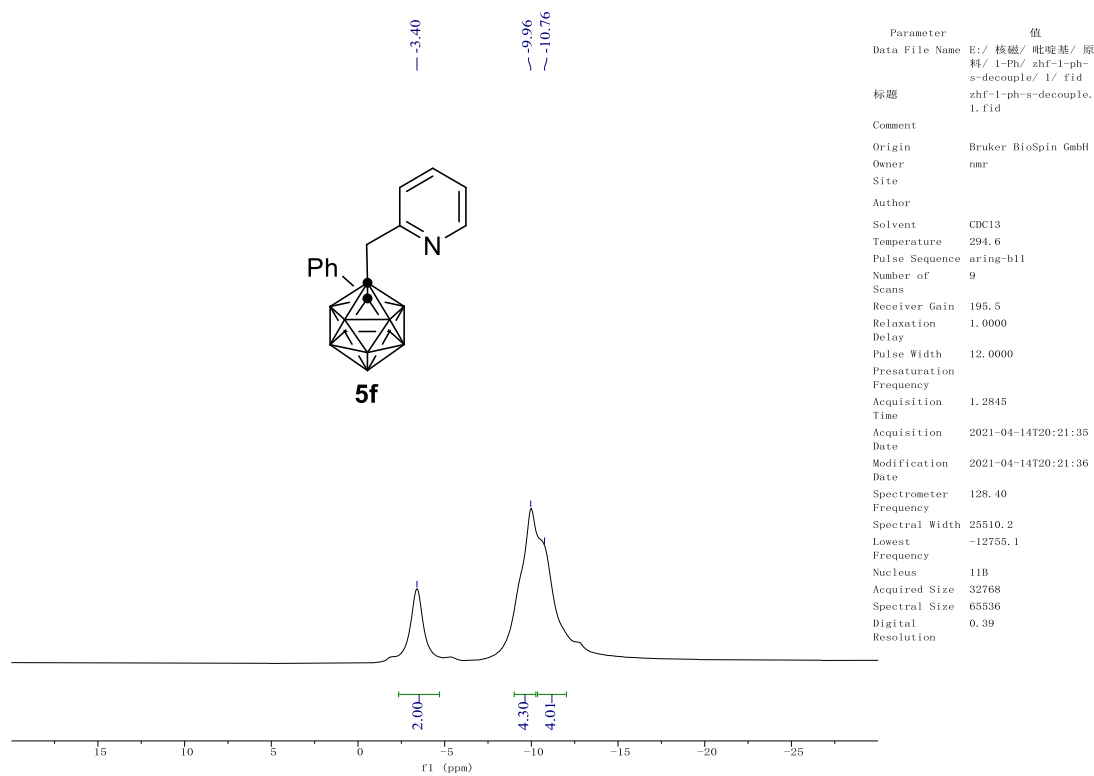


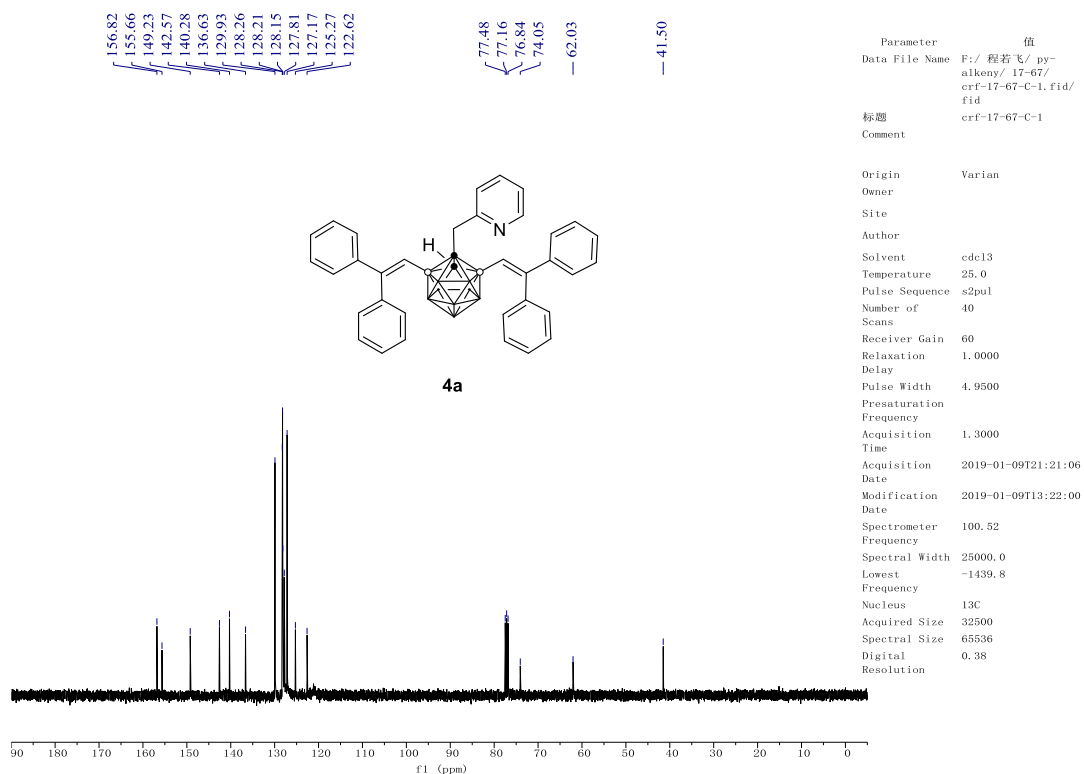
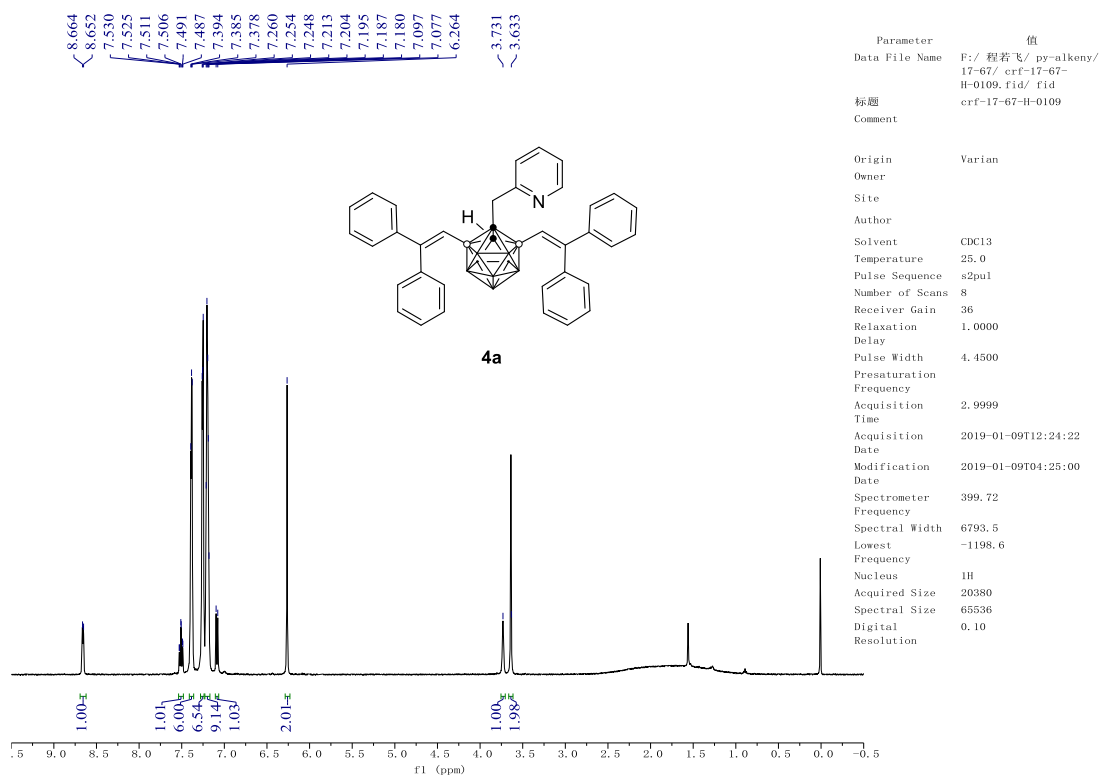
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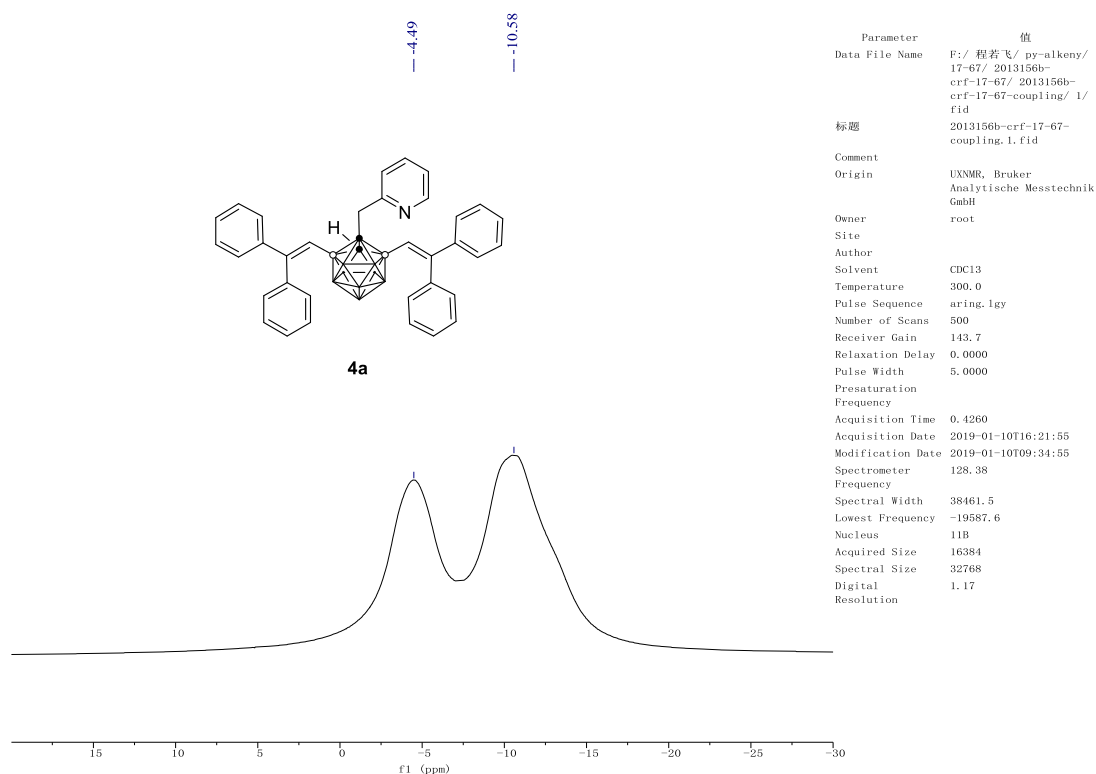
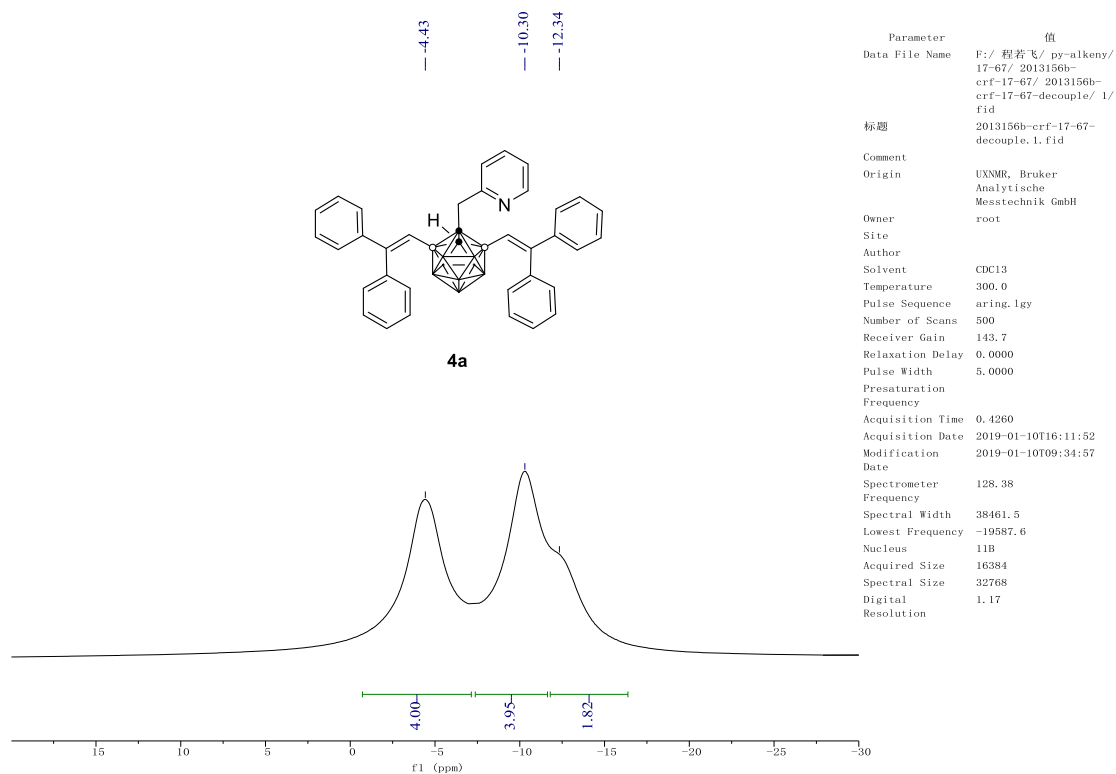
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Resolution

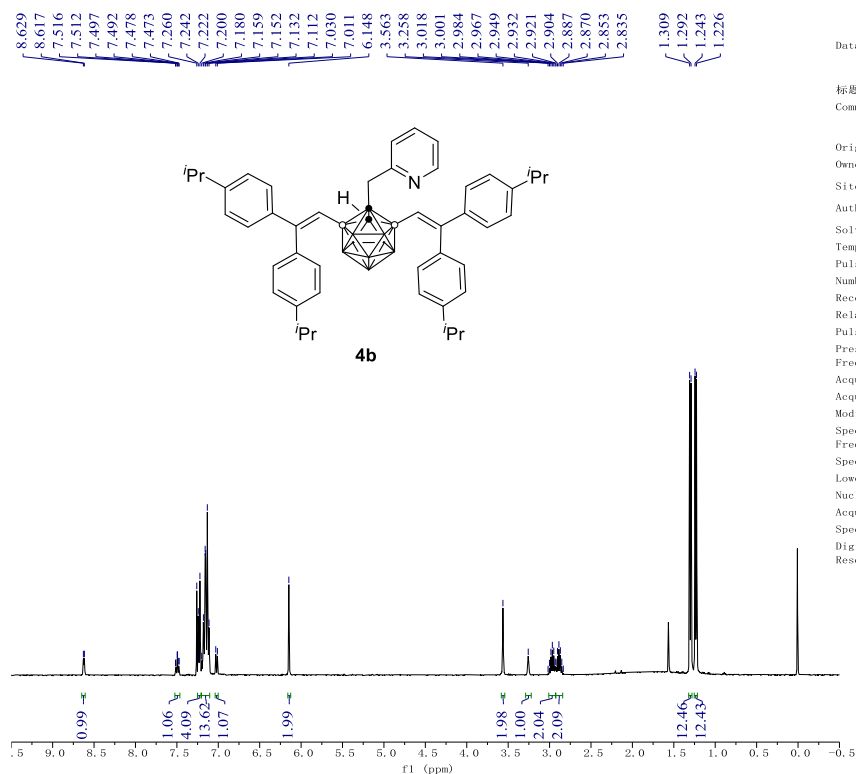




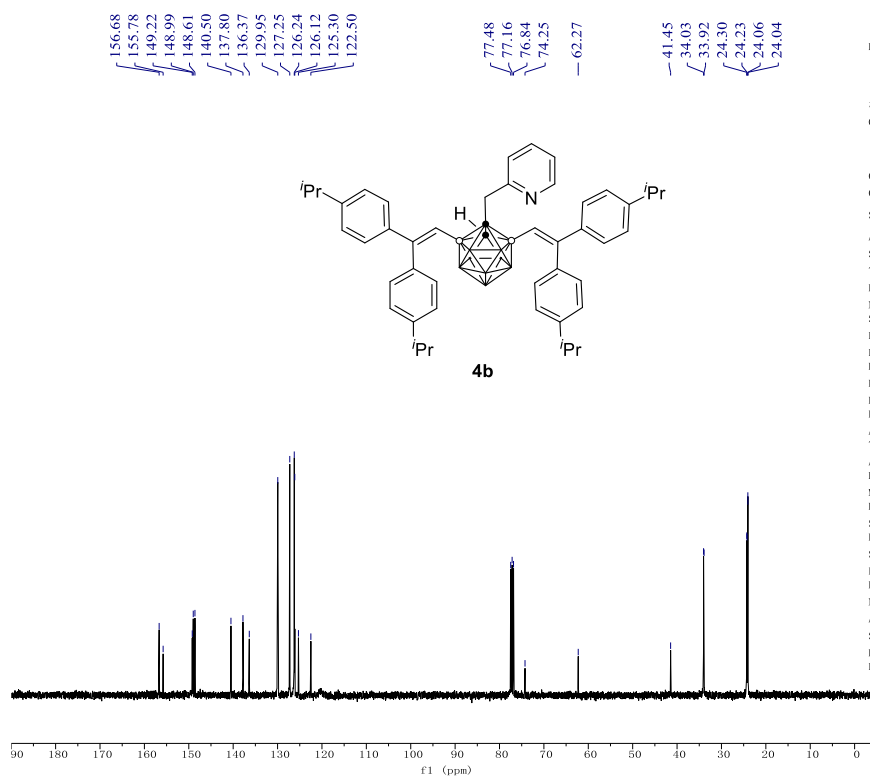




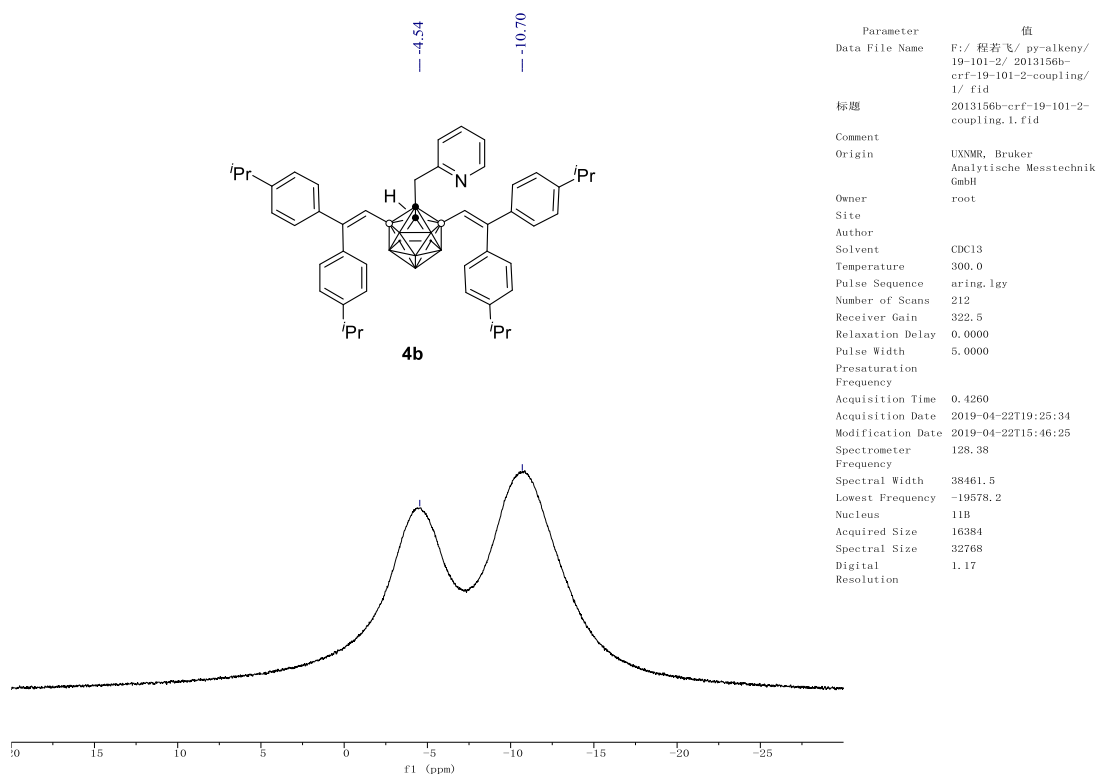
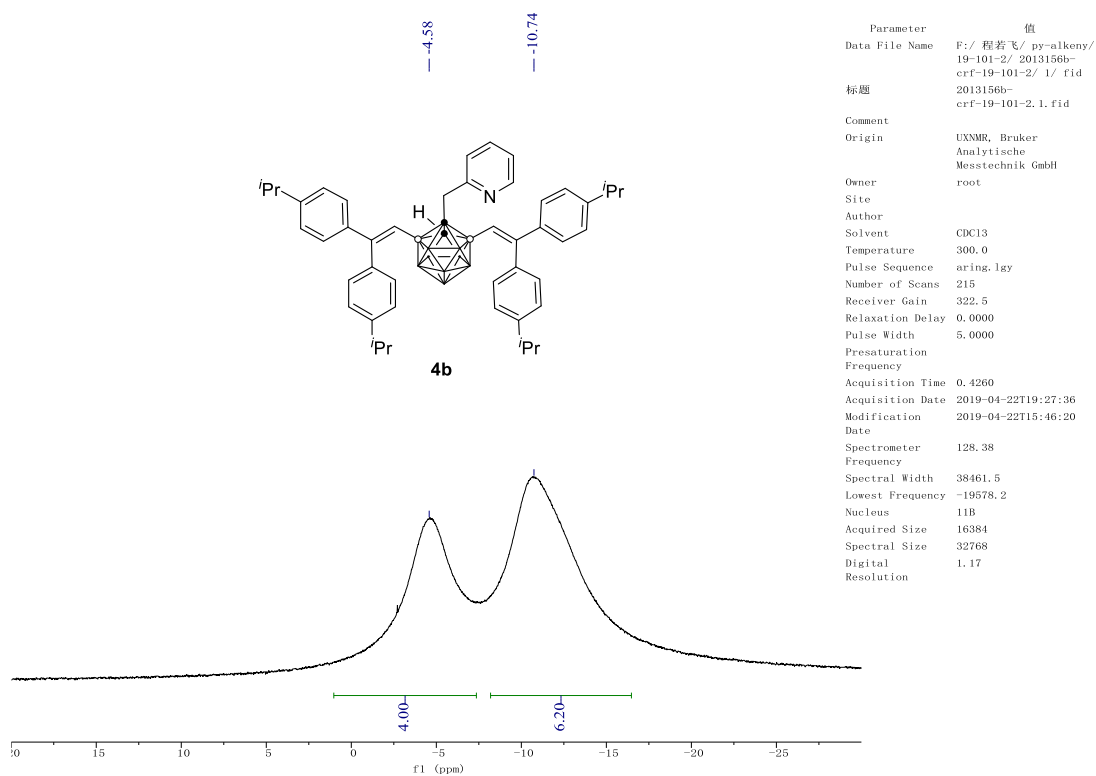


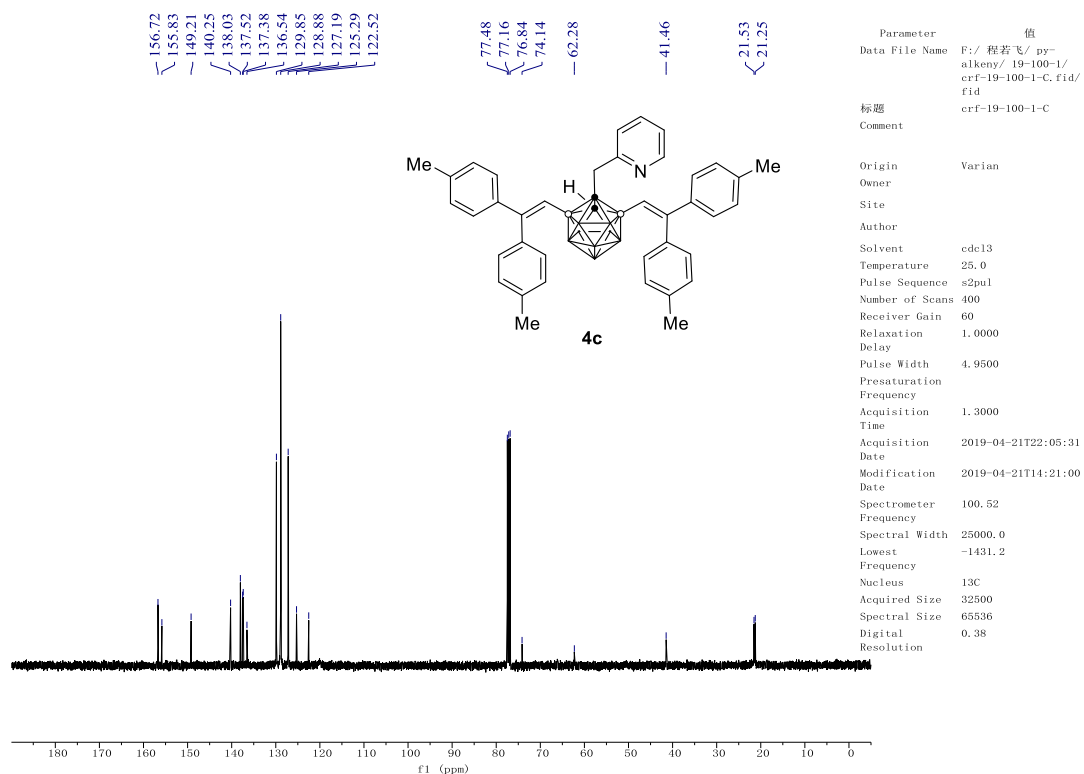
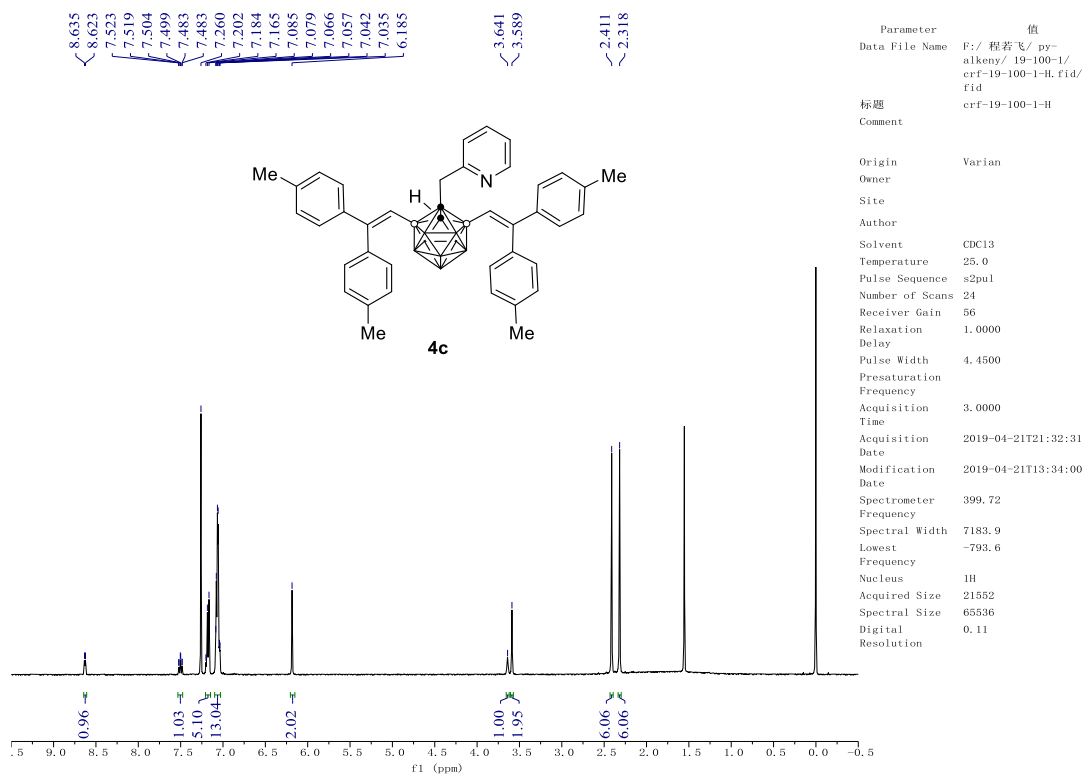


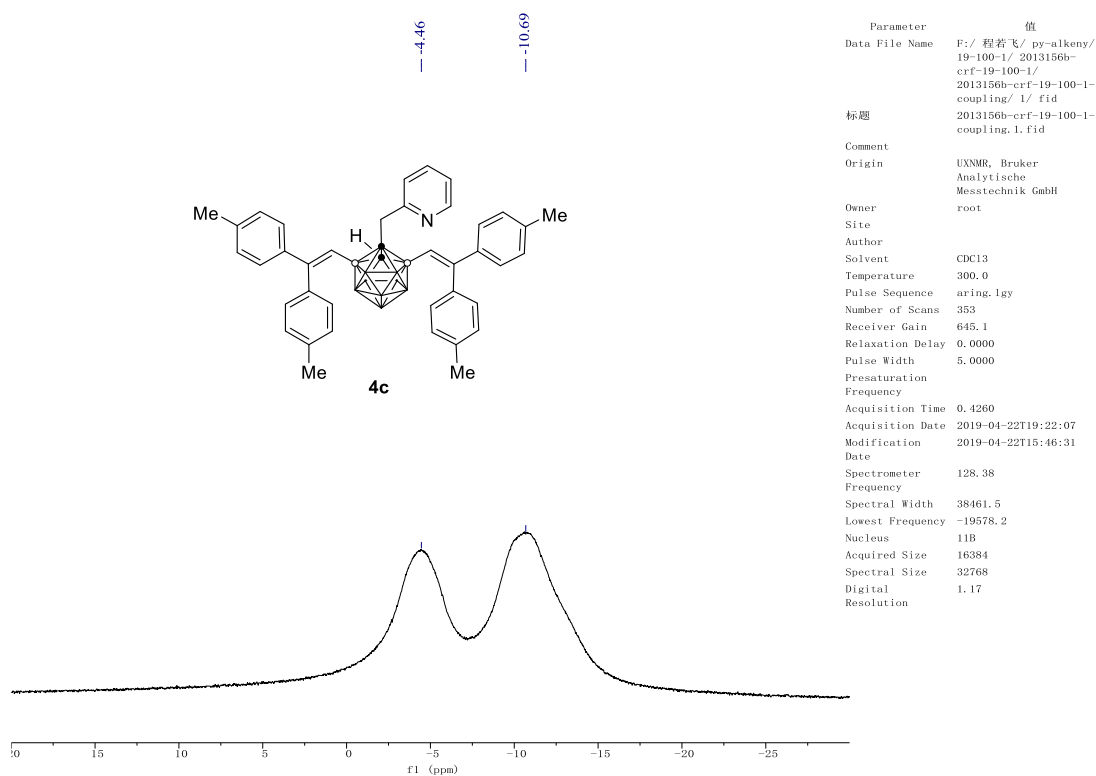
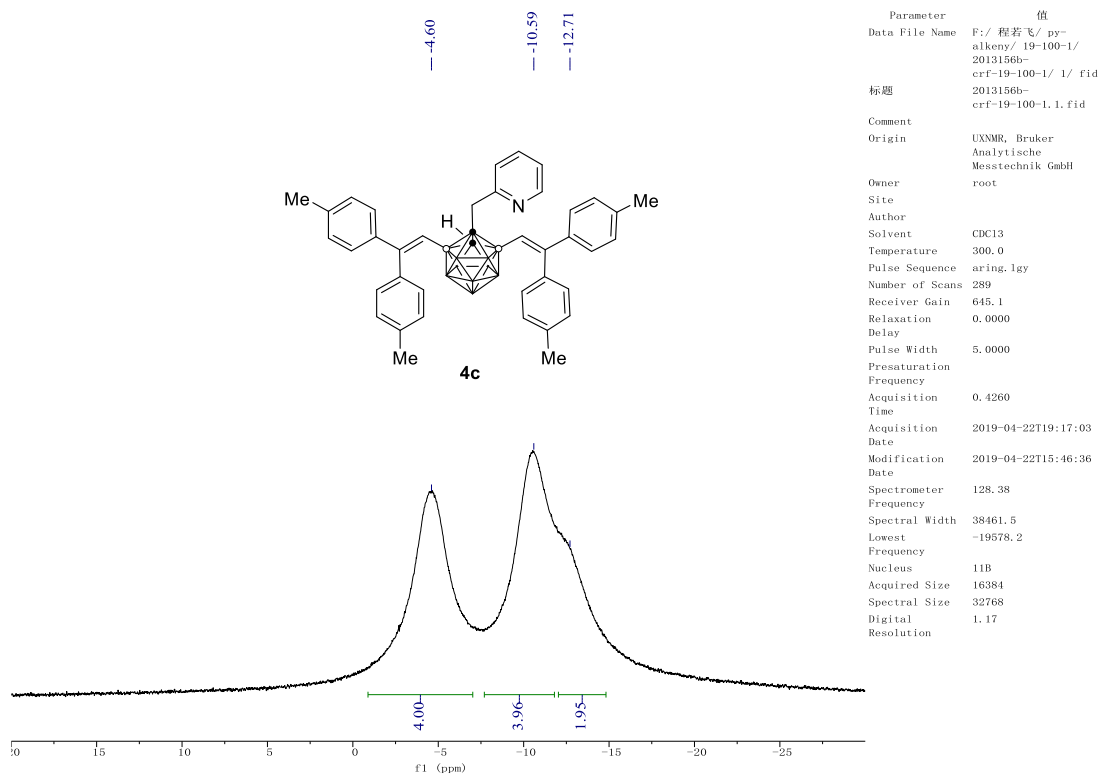
Parameter	值
Data File Name	F:/ 程若飞/ py-alkeny/ 19-101-2/ crf-19-101-2- H.fid/ fid
标题	crf-19-101-2-H
Comment	
Origin	Varian
Owner	
Site	
Author	
Solvent	CDCl ₃
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	4
Receiver Gain	44
Relaxation Delay	1.0000
Pulse Width	4.4500
Presaturation	
Frequency	
Acquisition Time	3.0000
Acquisition Date	2019-04-21T21:35:30
Modification Date	2019-04-21T13:36:00
Spectrometer	399.72
Frequency	
Spectral Width	7183.9
Lowest Frequency	-793.7
Nucleus	¹ H
Acquired Size	21552
Spectral Size	65536
Digital	0.11
Resolution	

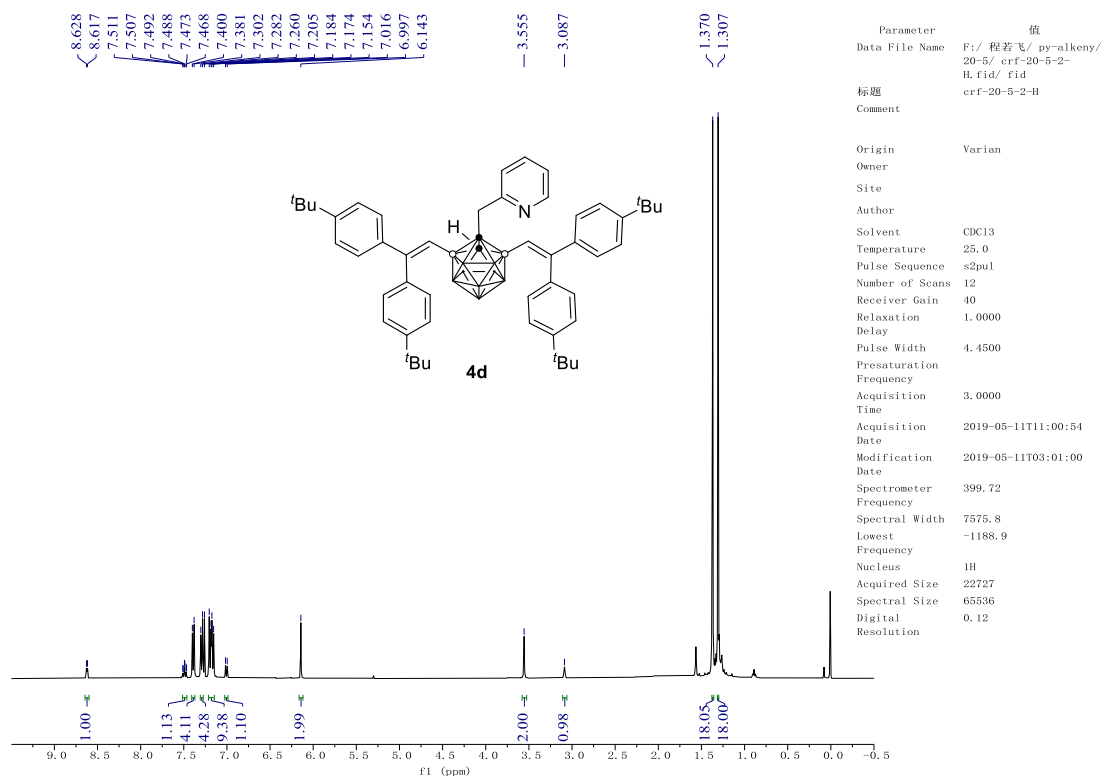


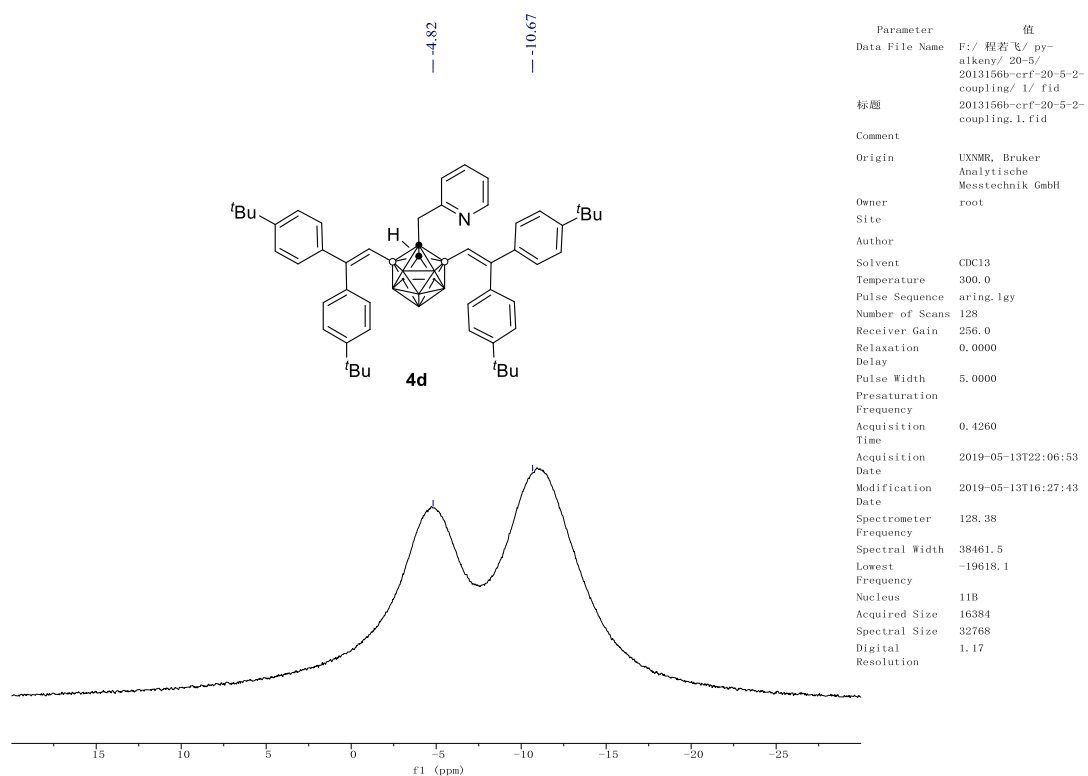
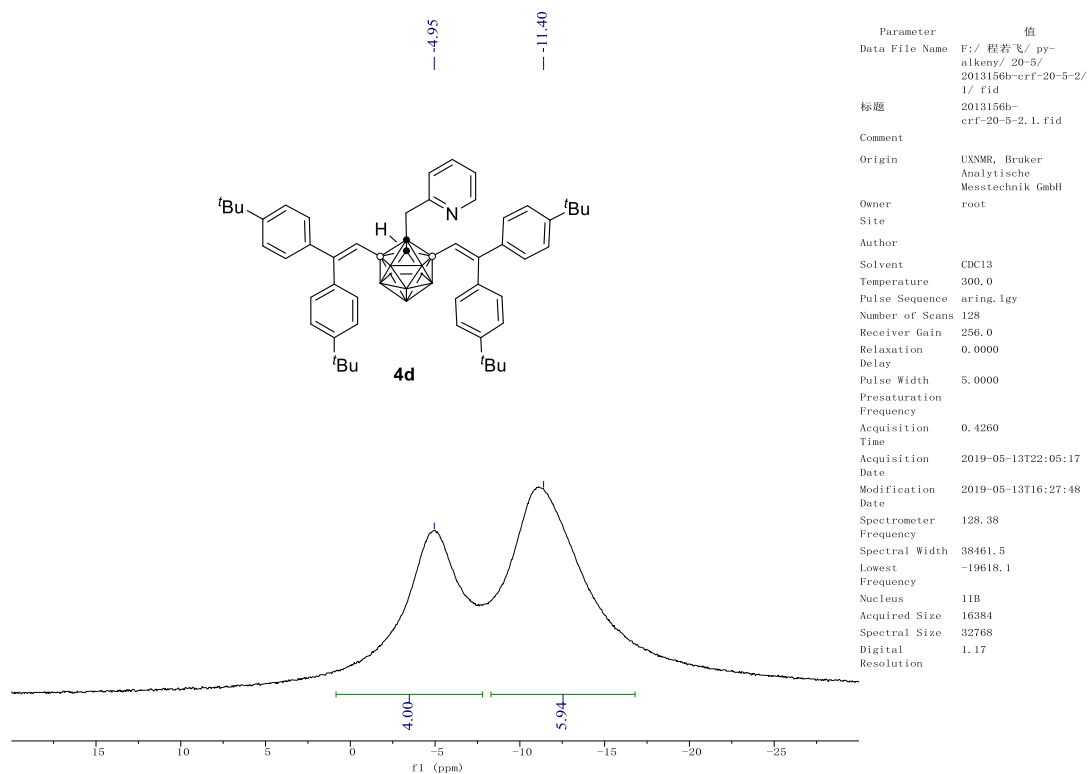
Parameter	值
Data File Name	F:/ 程若飞/ py-alkeny/ 19-101-2/ crf-19-101-2-C.fid/ fid
标题	crf-19-101-2-C
Comment	Std carbon
Origin	Varian
Owner	
Site	
Author	omc1
Solvent	cdcl3
Temperature	20.0
Pulse Sequence	s2pul
Number of Scans	84
Receiver Gain	60
Relaxation Delay	1.0000
Pulse Width	5.7000
Presaturation	
Frequency	
Acquisition Time	1.3000
Acquisition Date	2019-04-21T19:55:02
Modification Date	2019-04-21T11:58:00
Spectrometer	100.60
Frequency	
Spectral Width	24509.8
Lowest Frequency	-1681.6
Nucleus	¹³ C
Acquired Size	31863
Spectral Size	65536
Digital	0.37
Resolution	

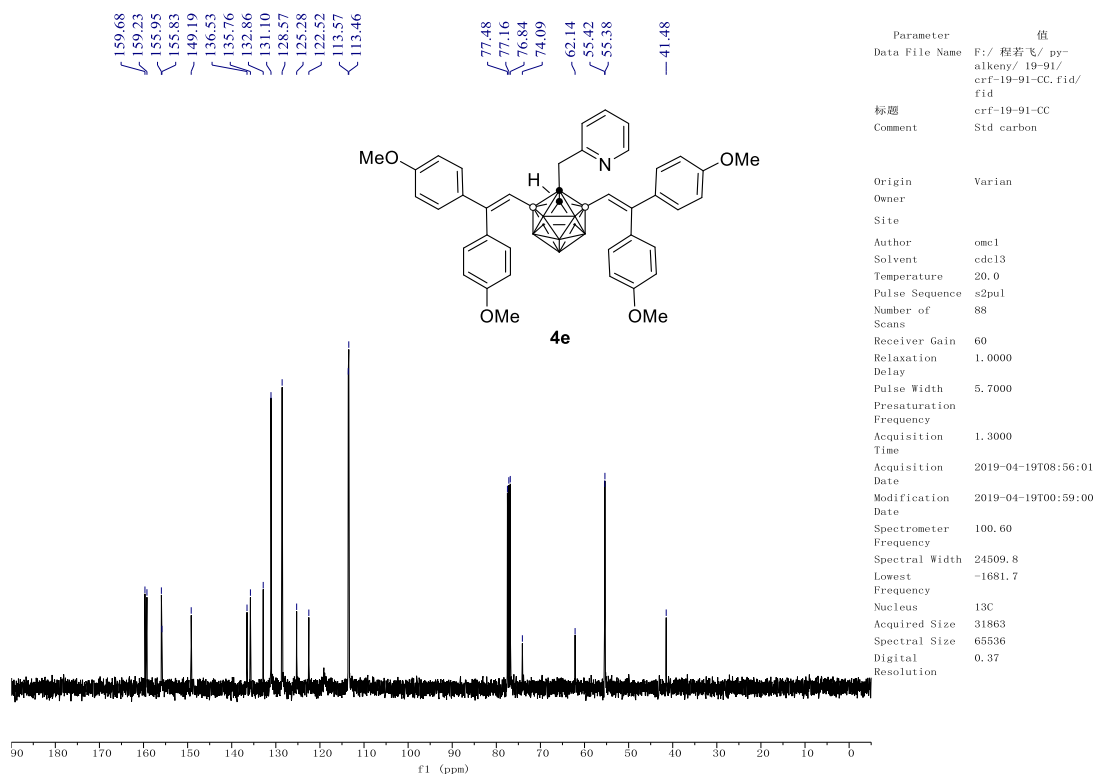
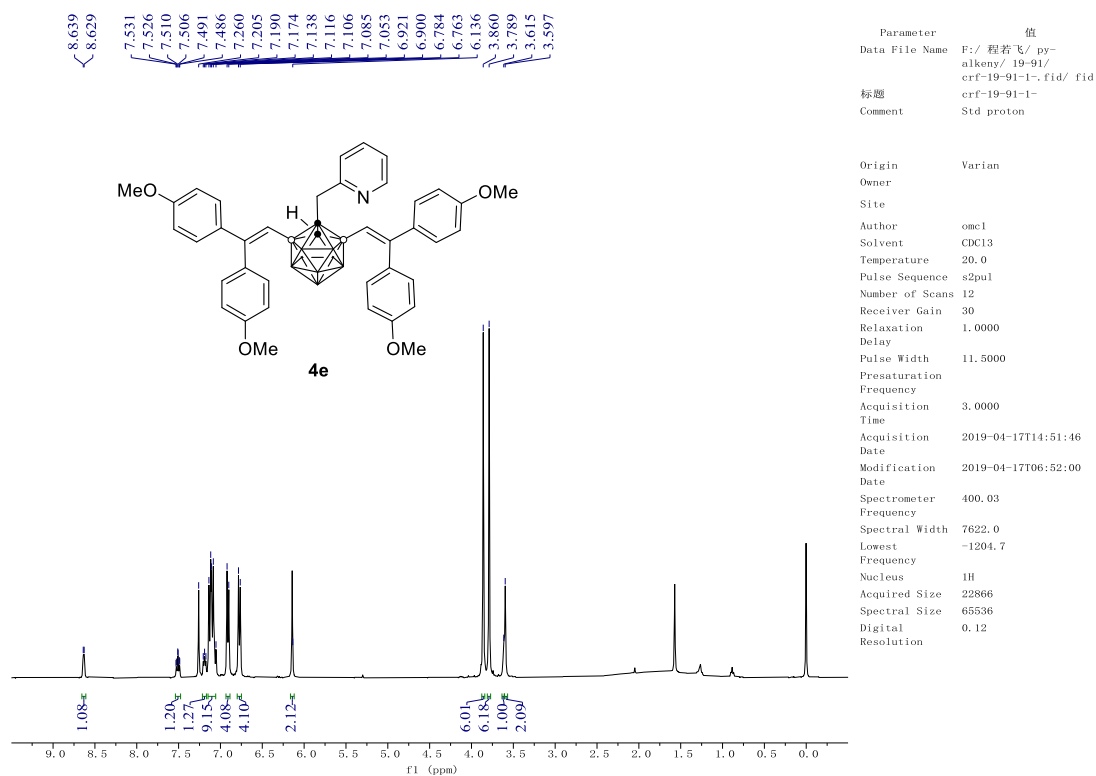


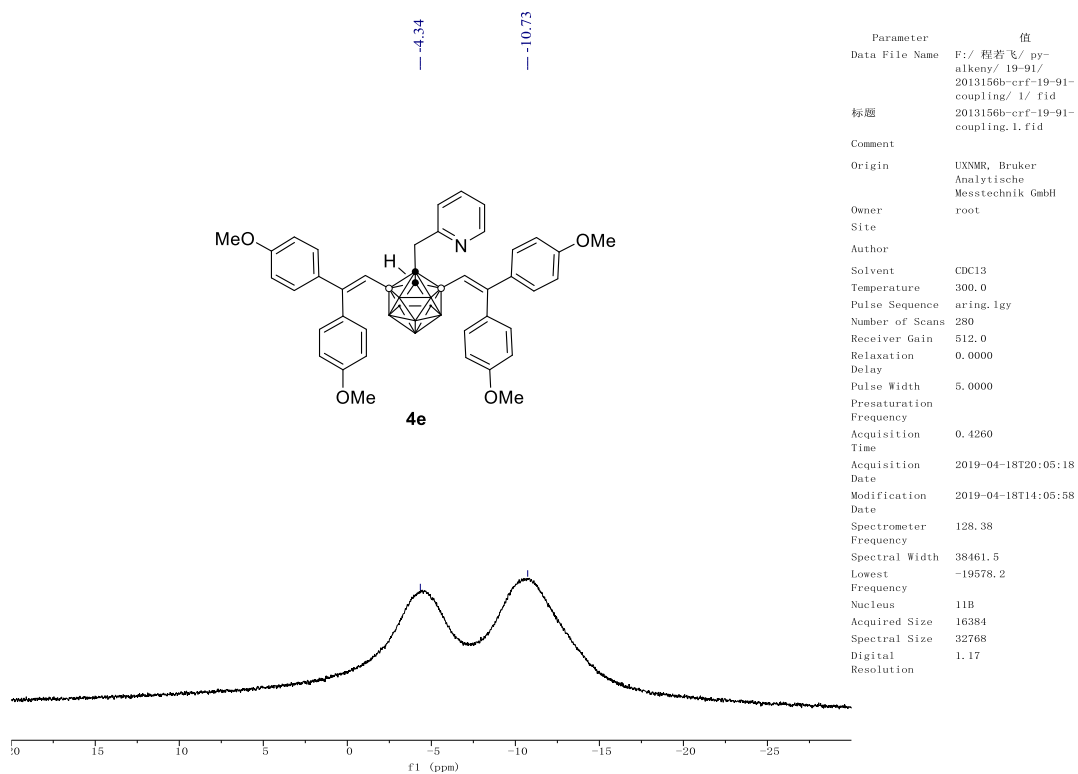
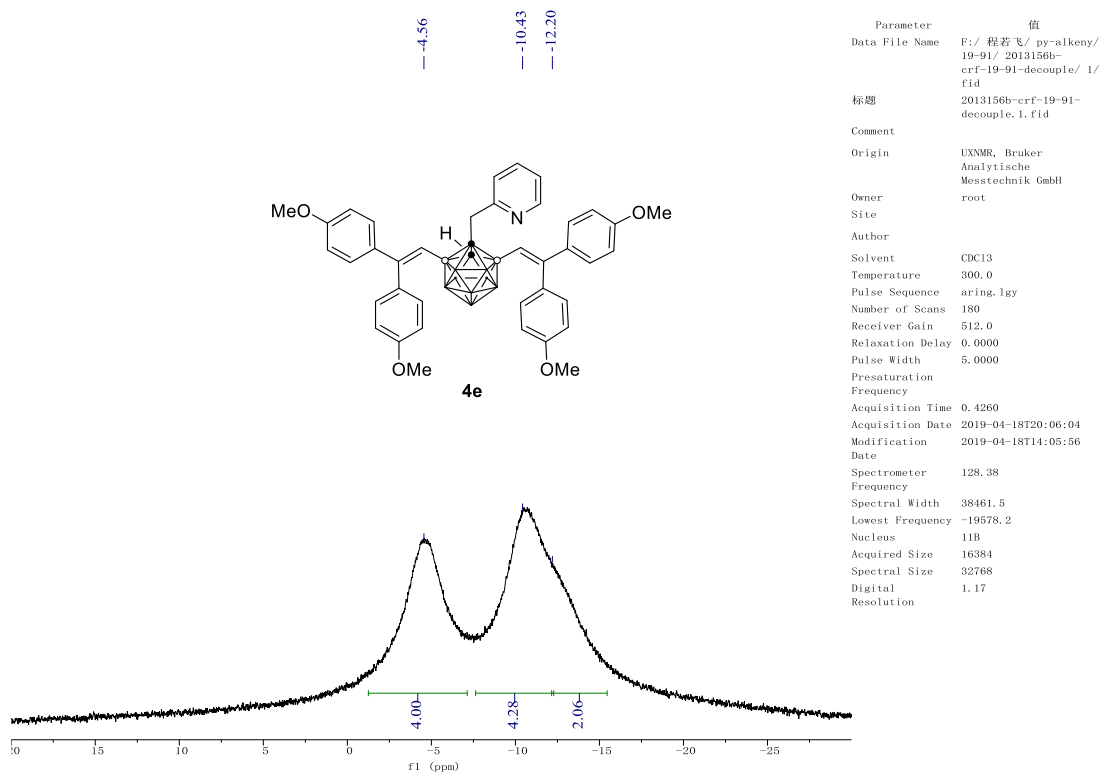


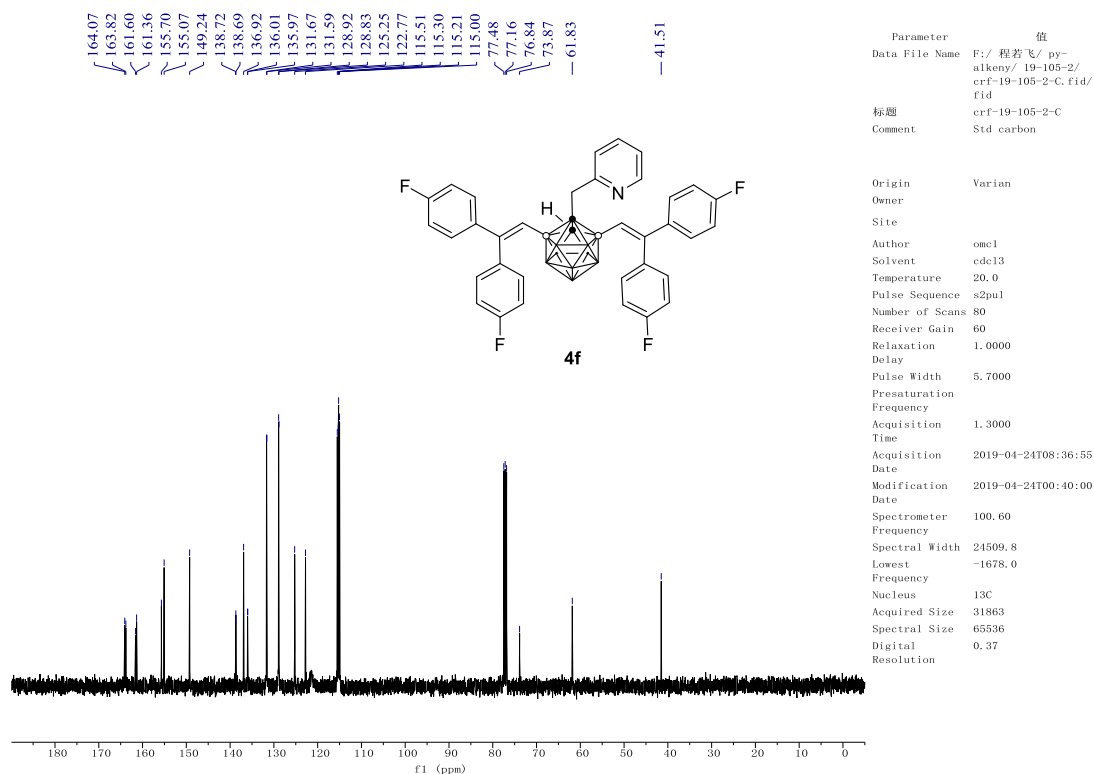
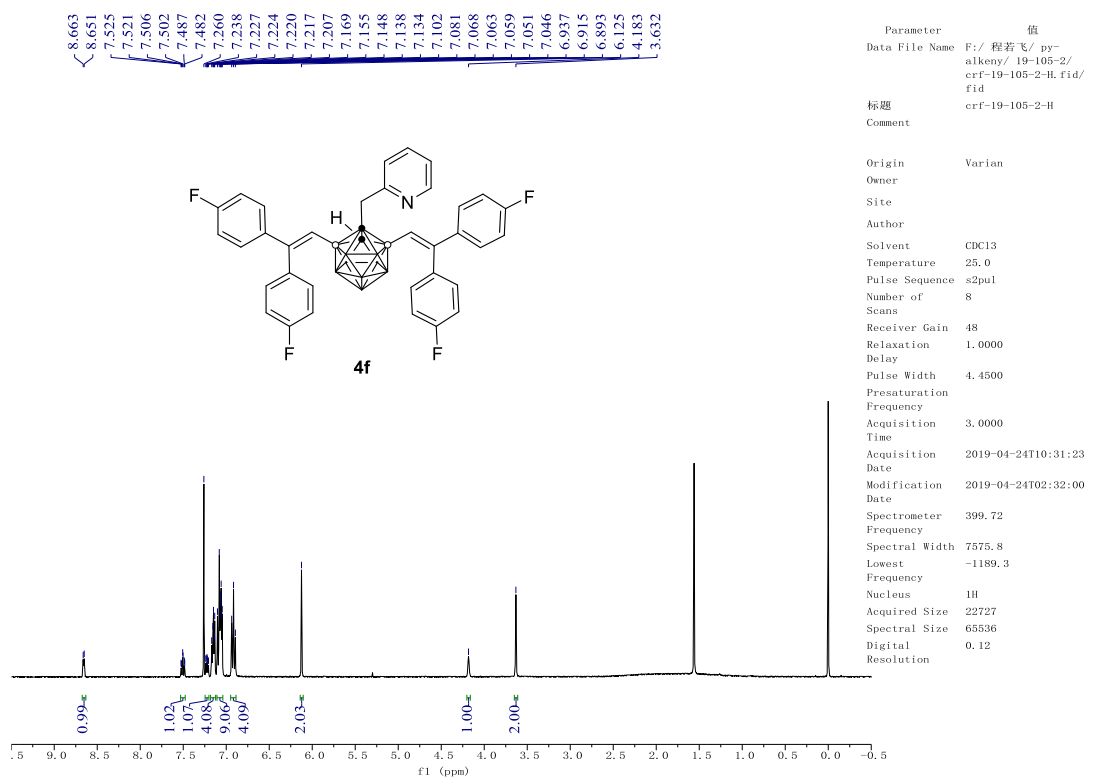


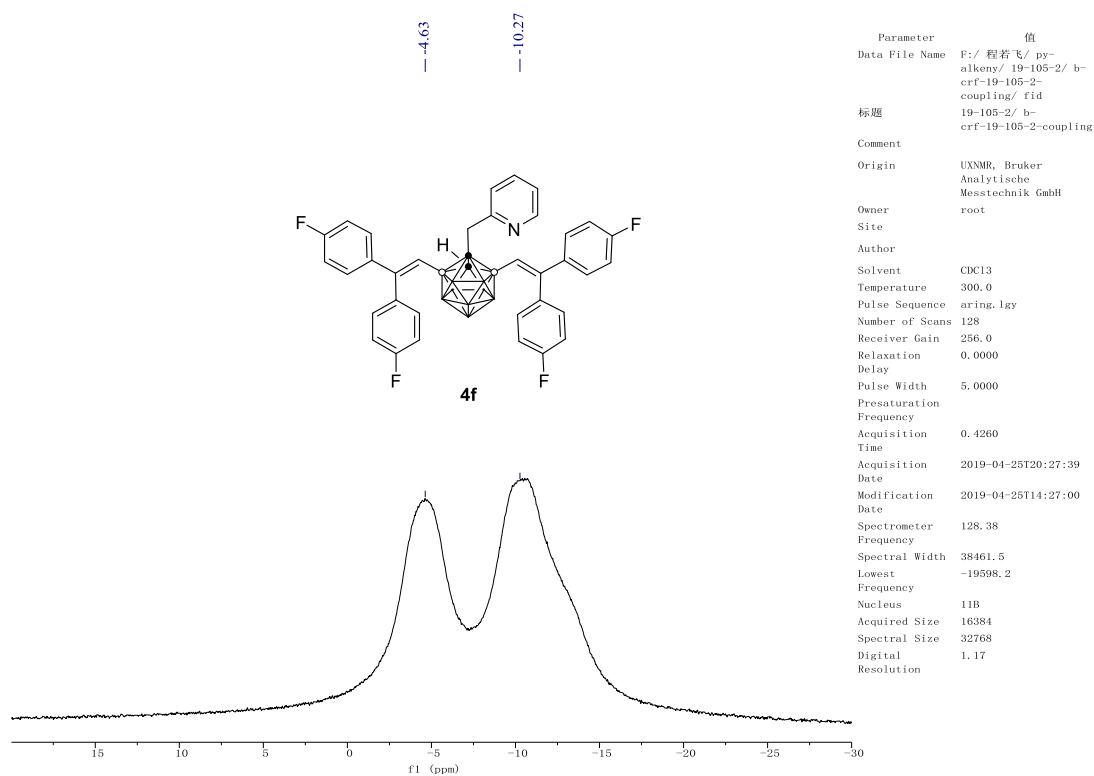
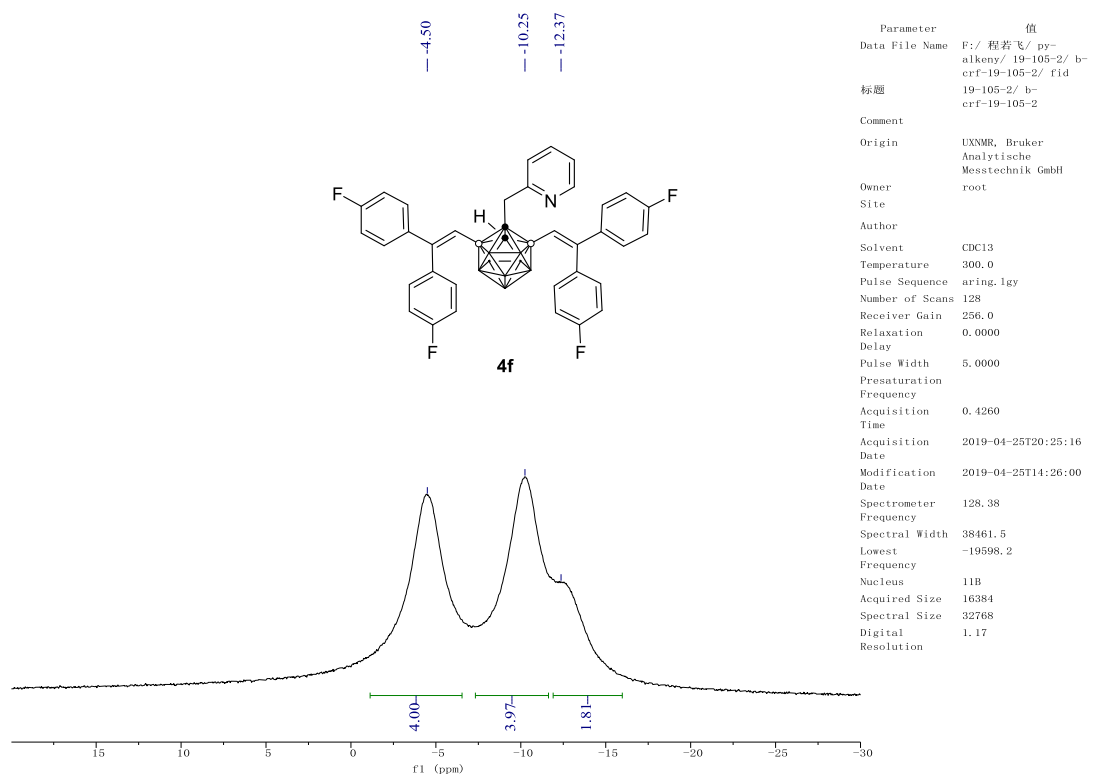


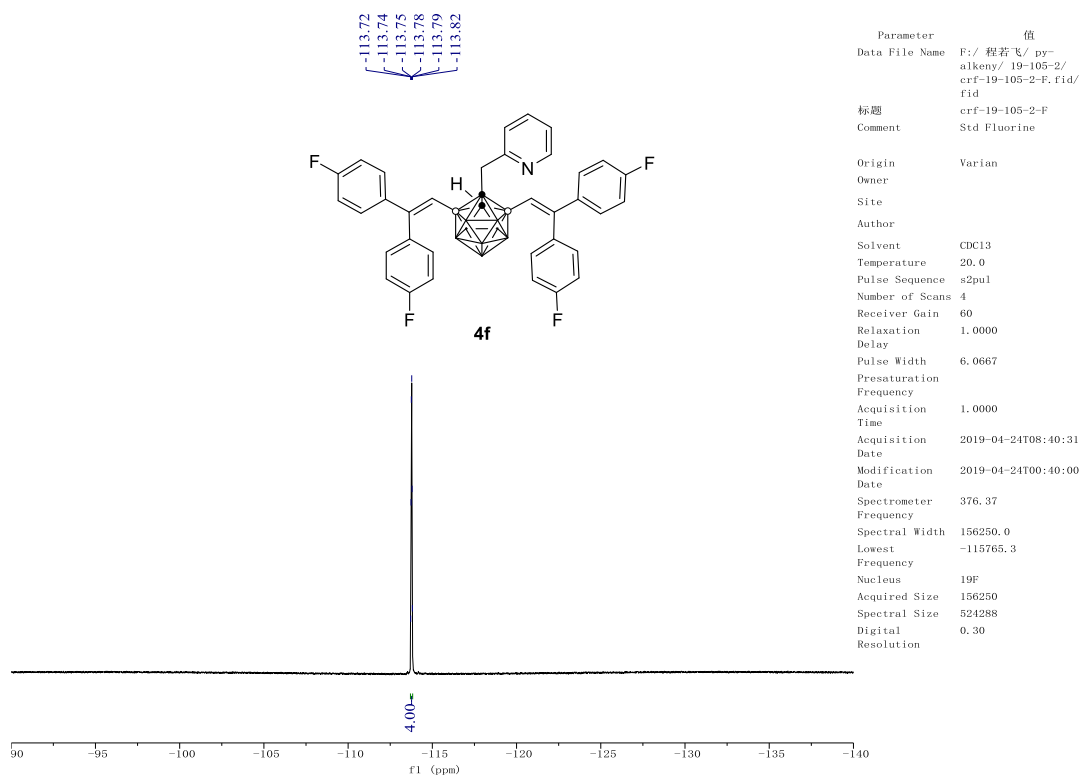


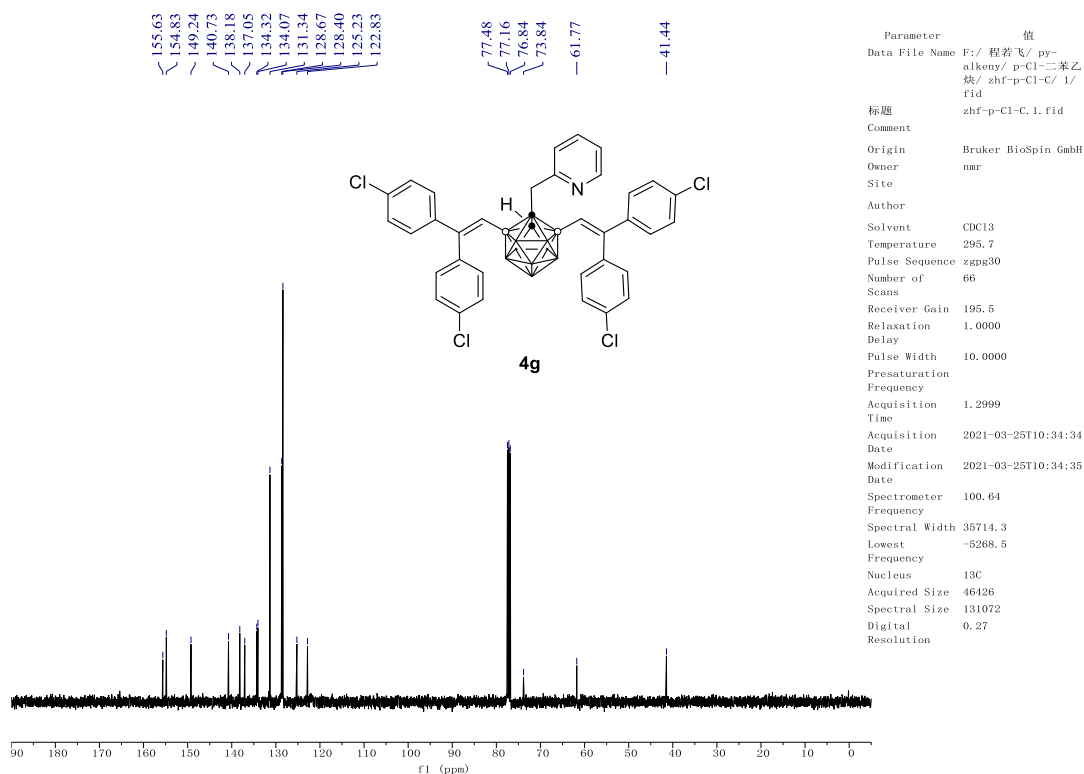
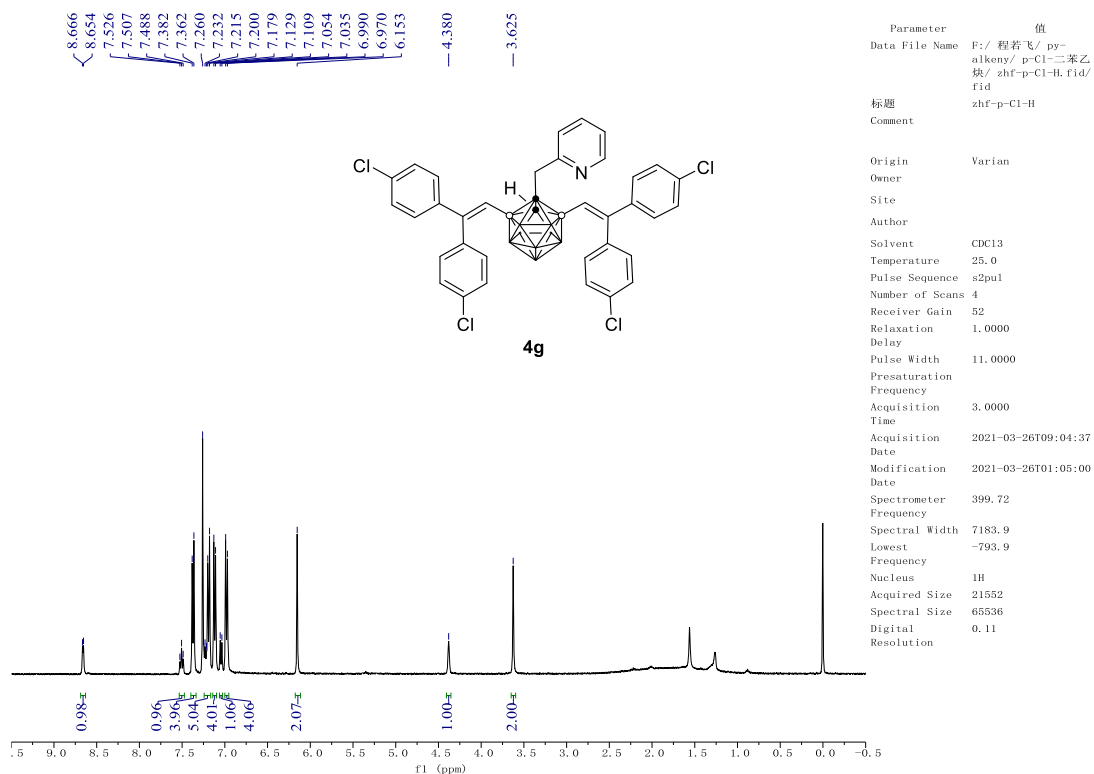


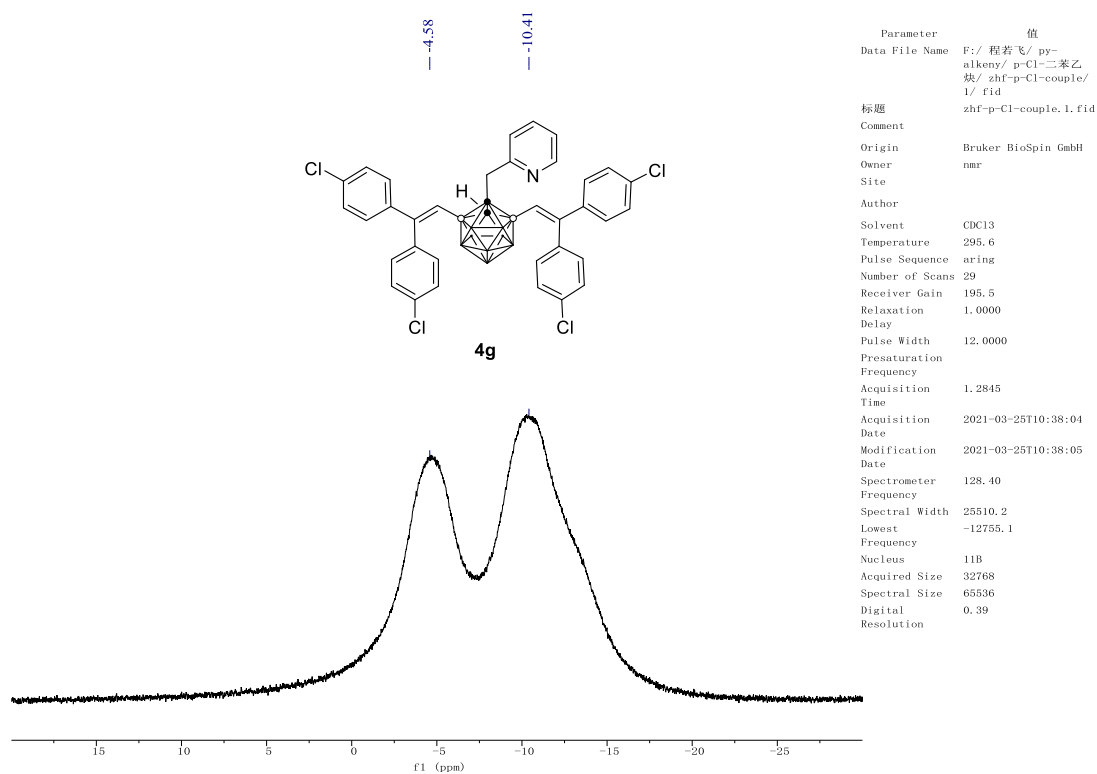
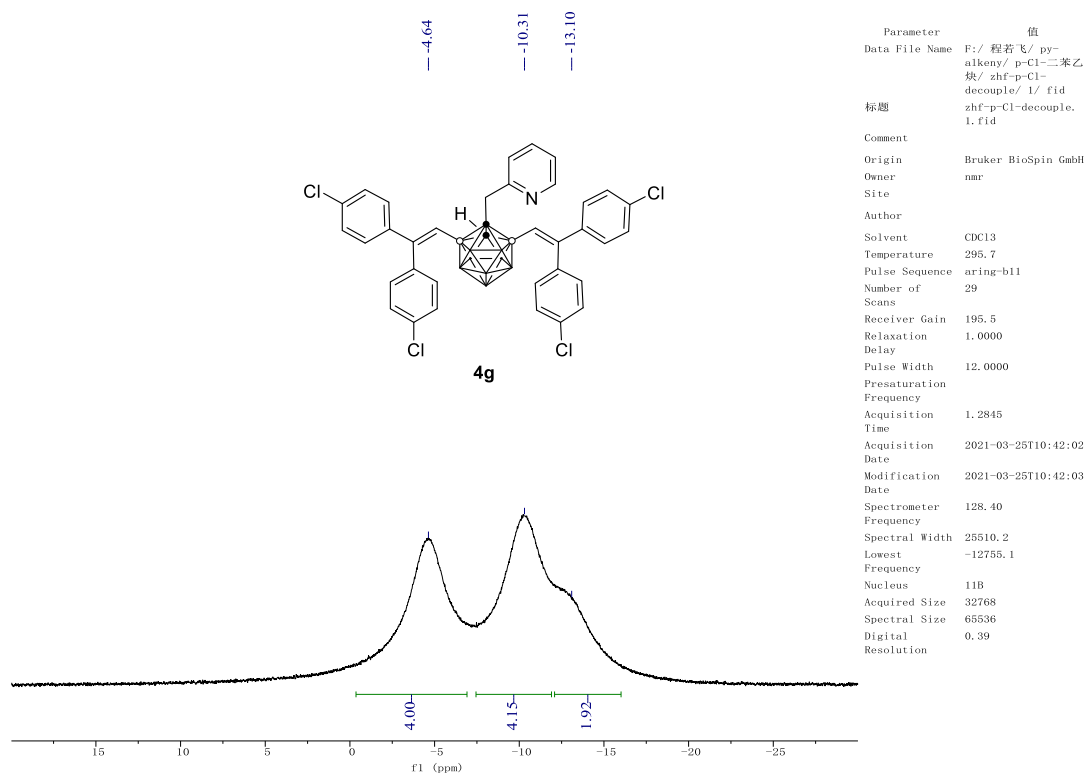


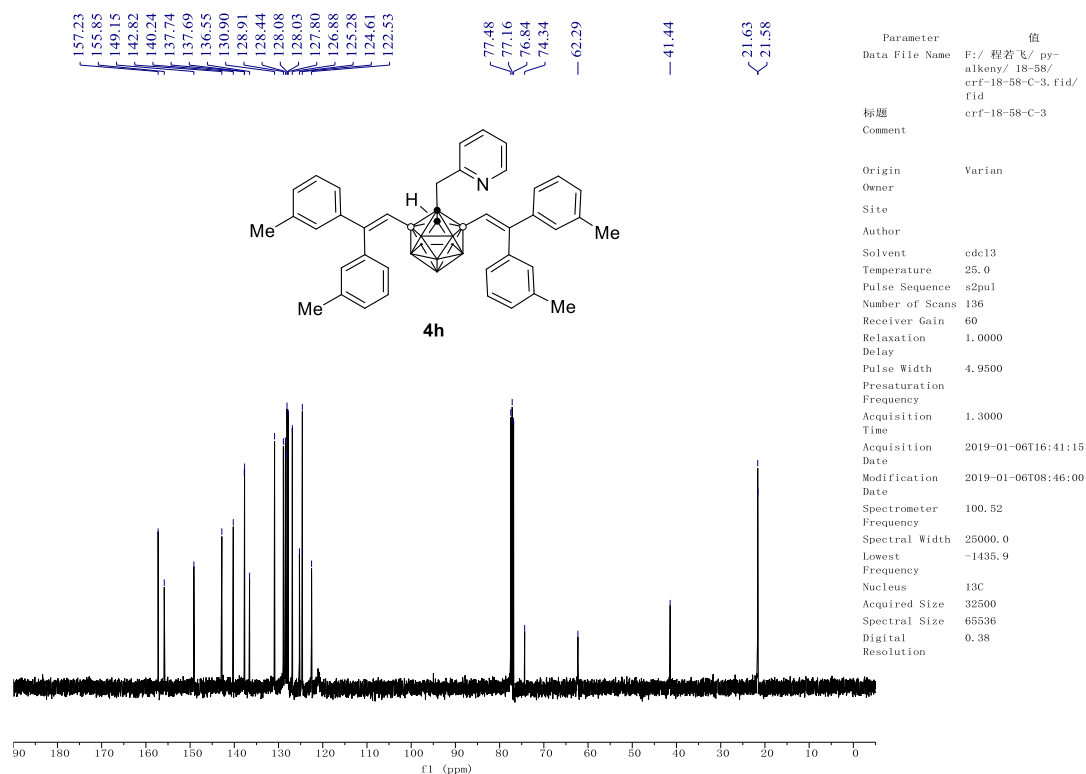
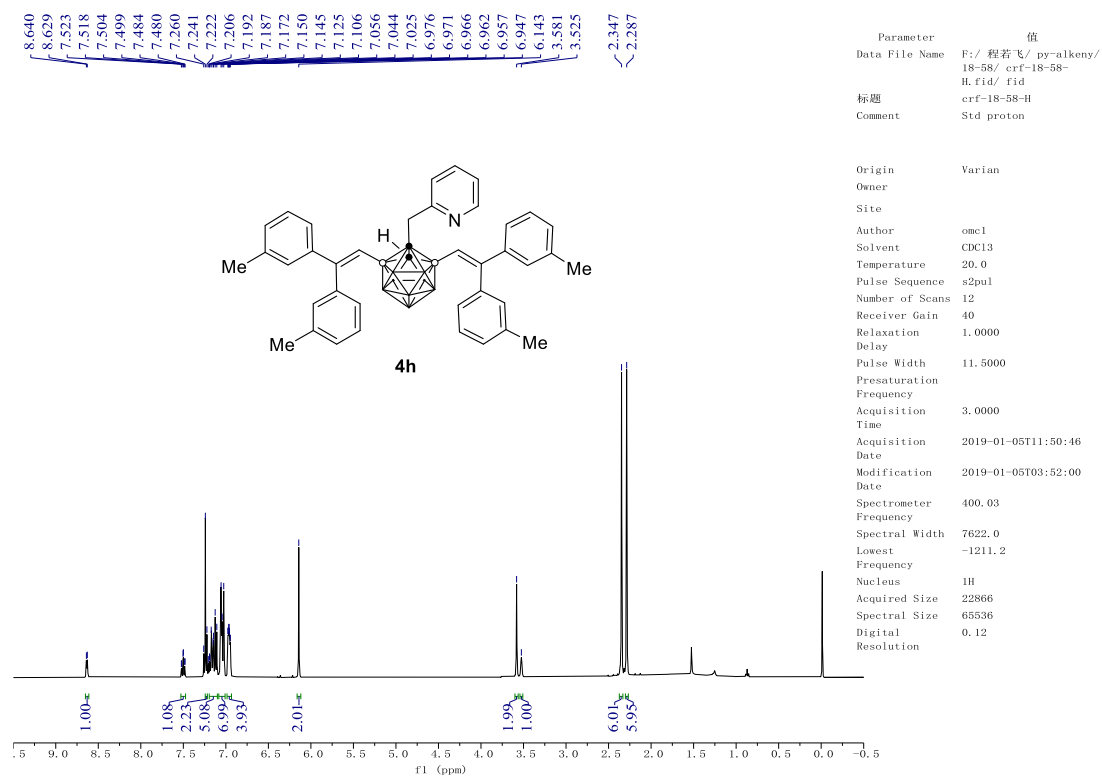


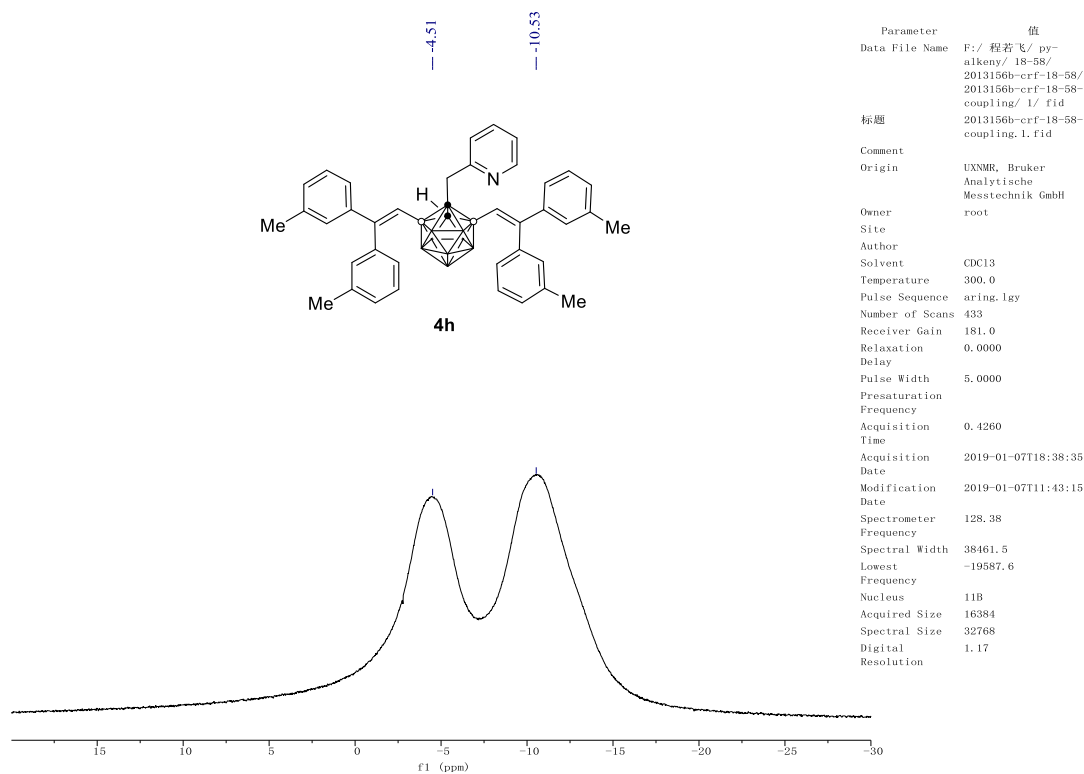
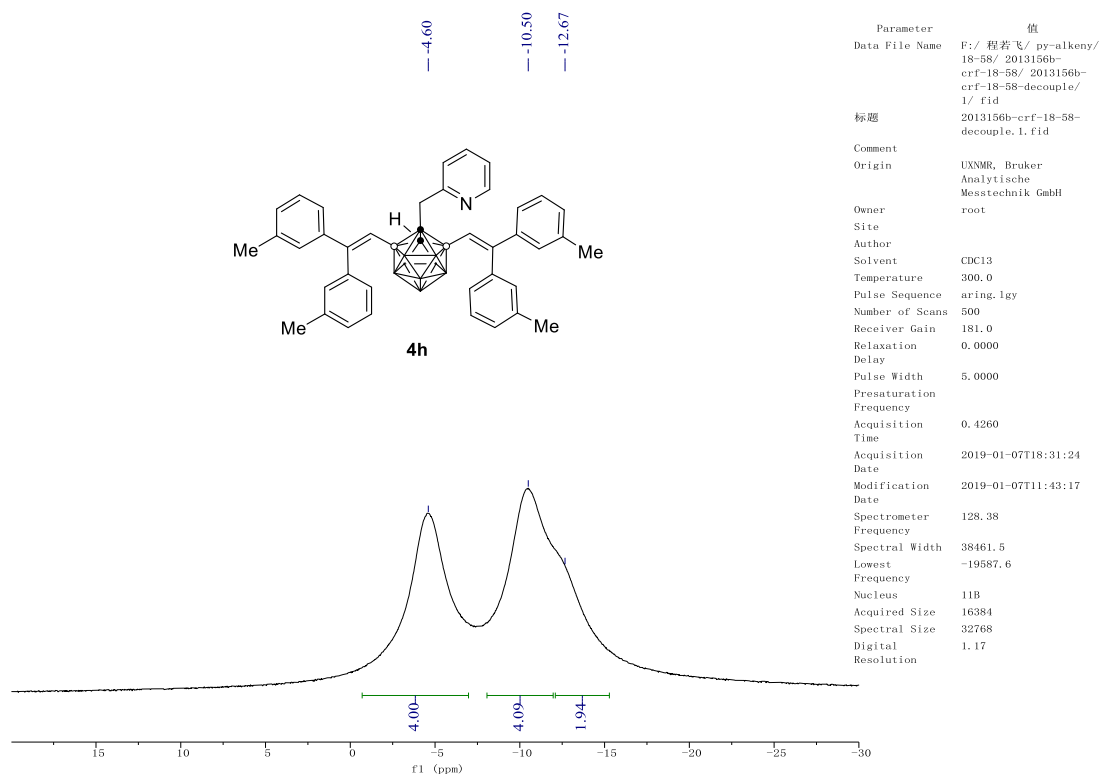


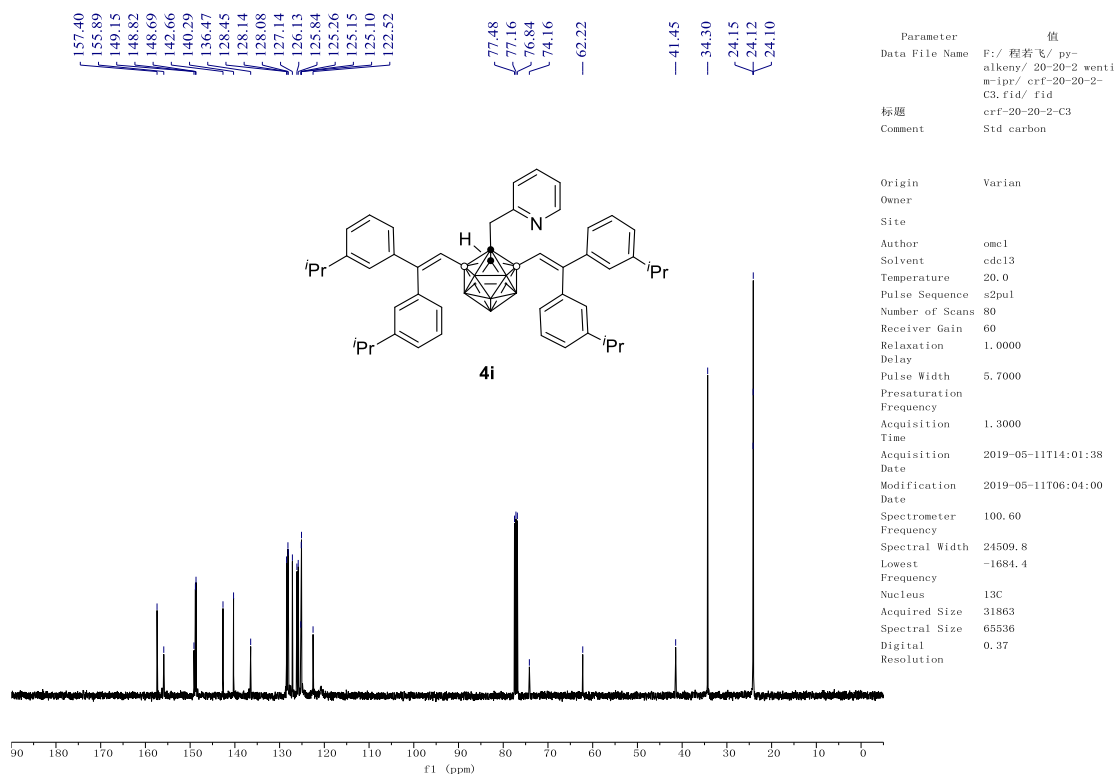
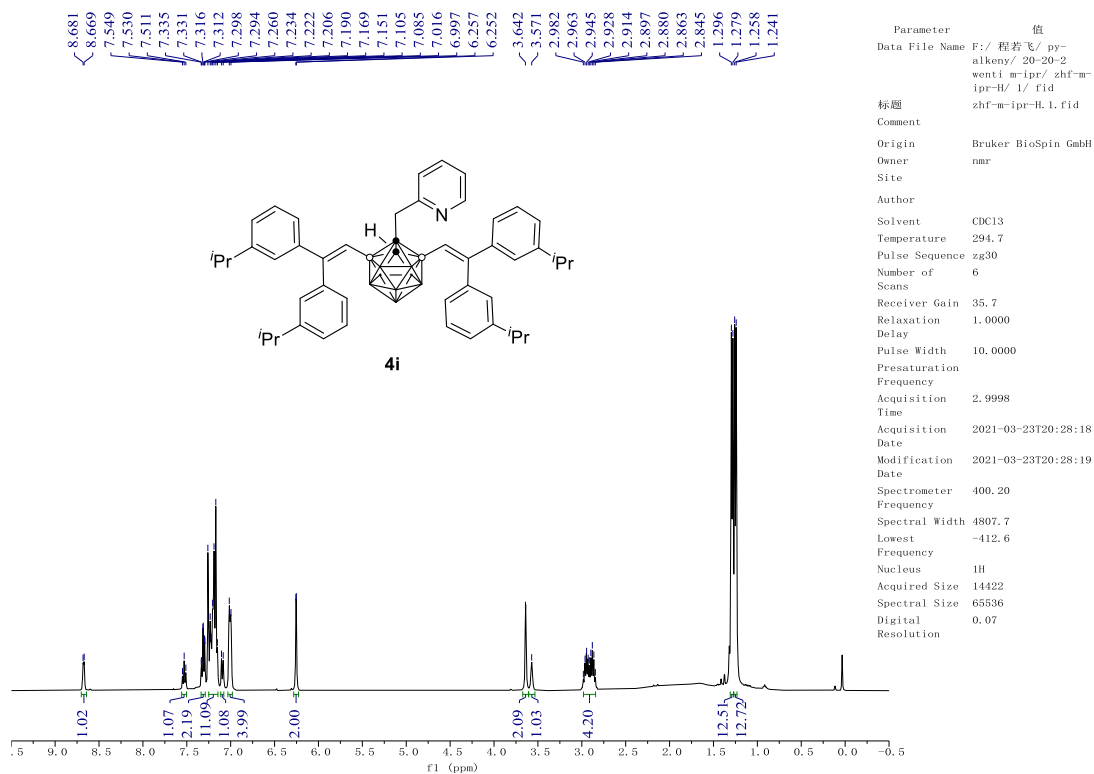


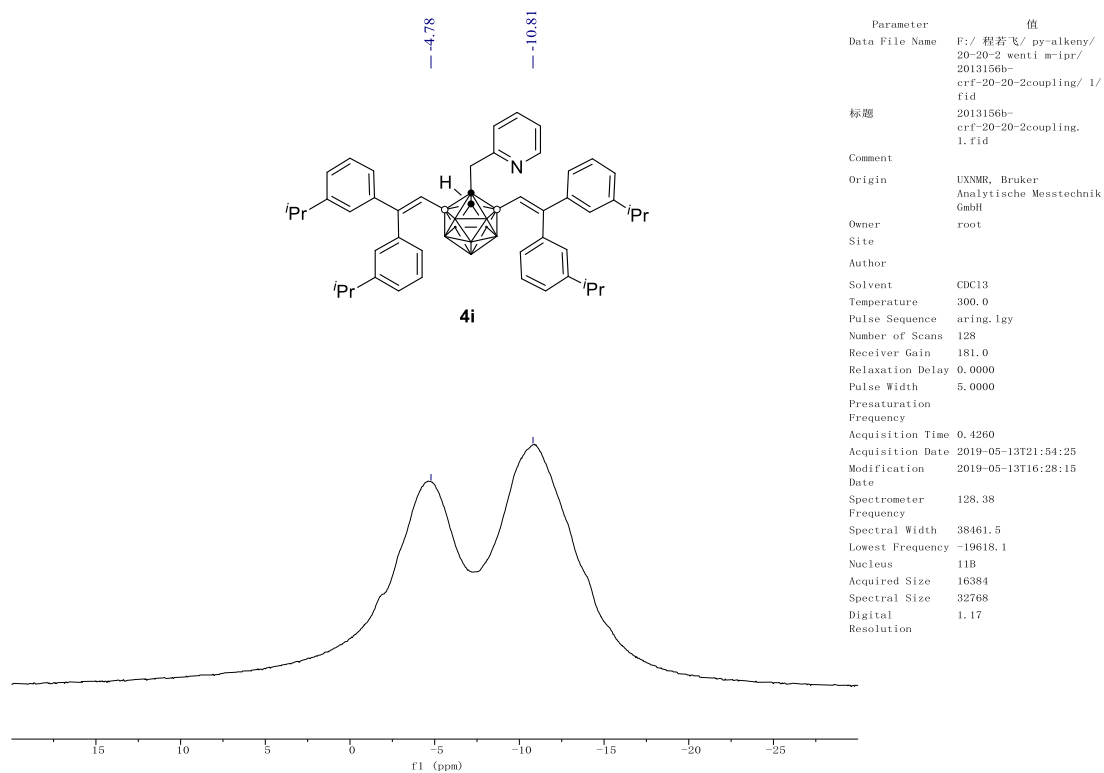
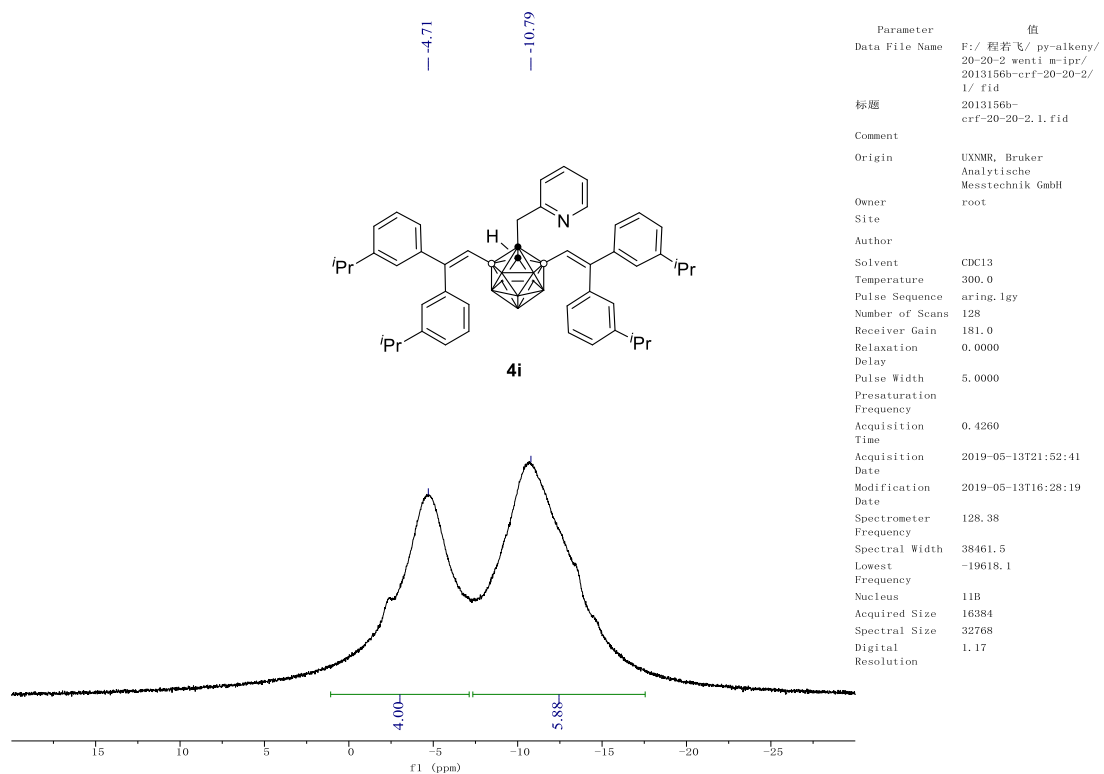


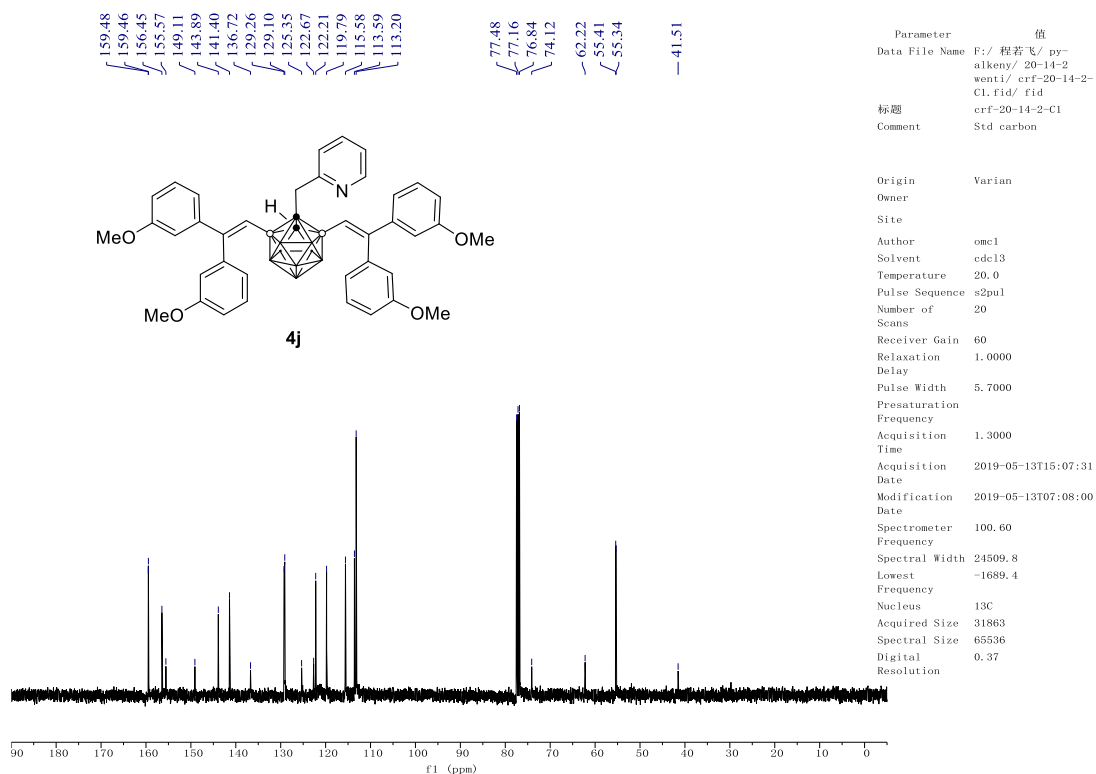
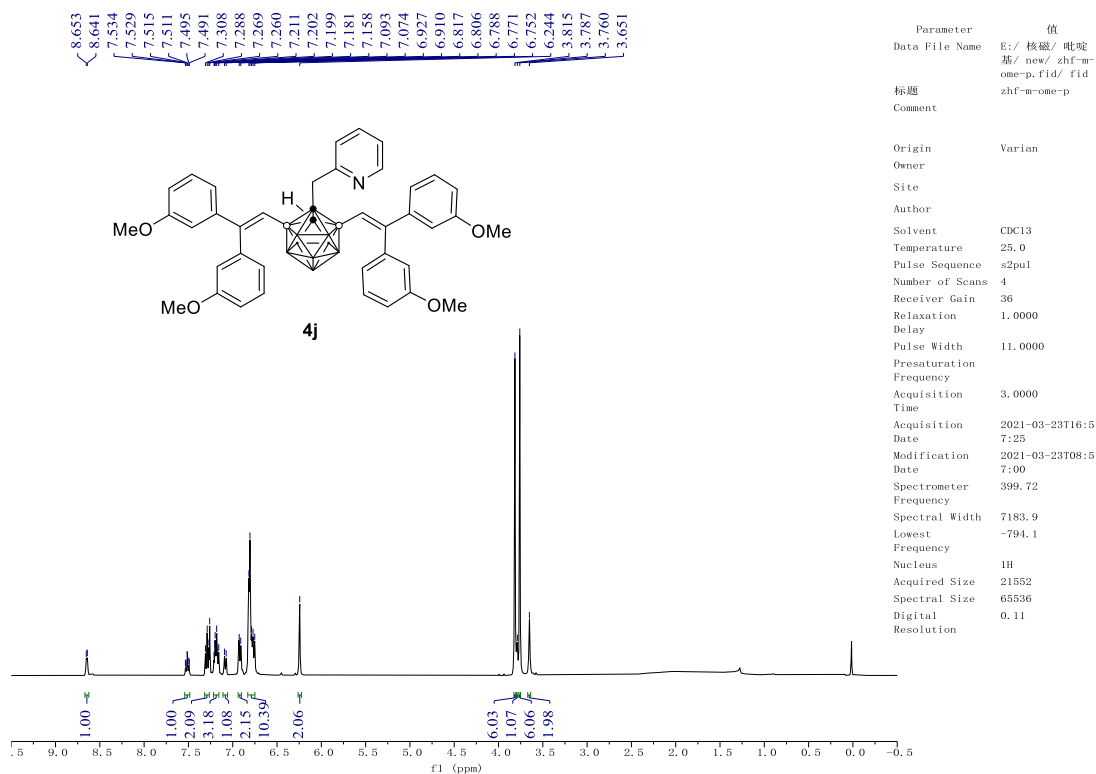


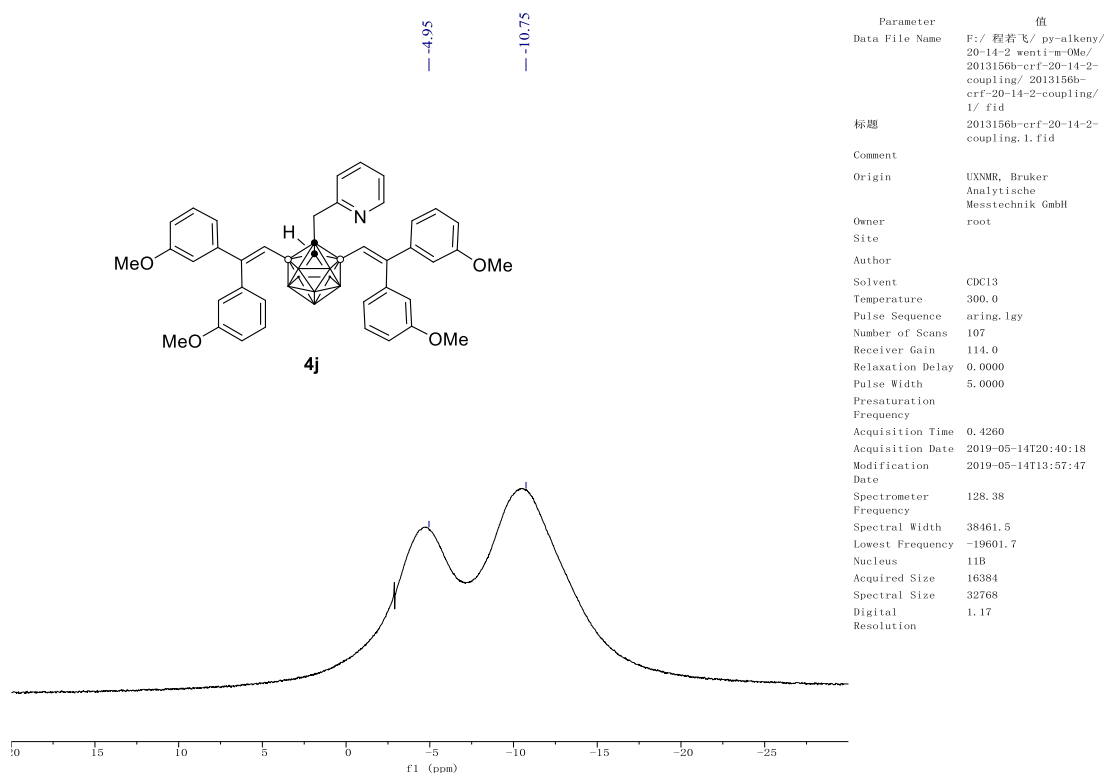
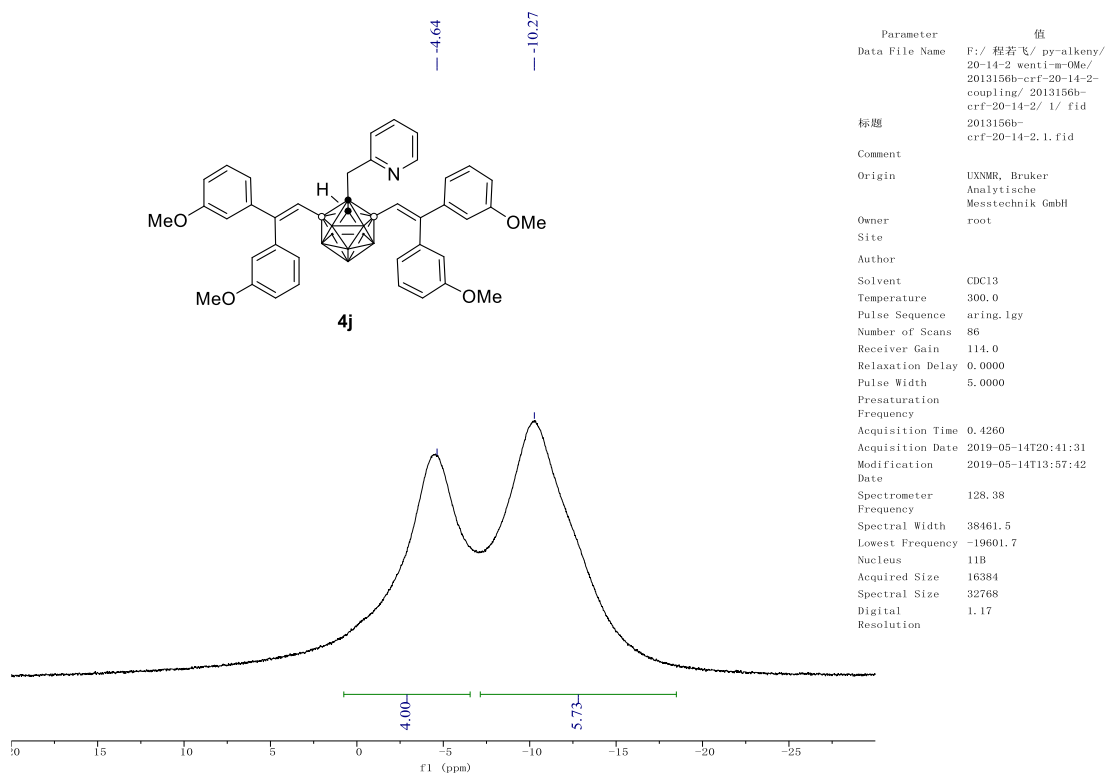


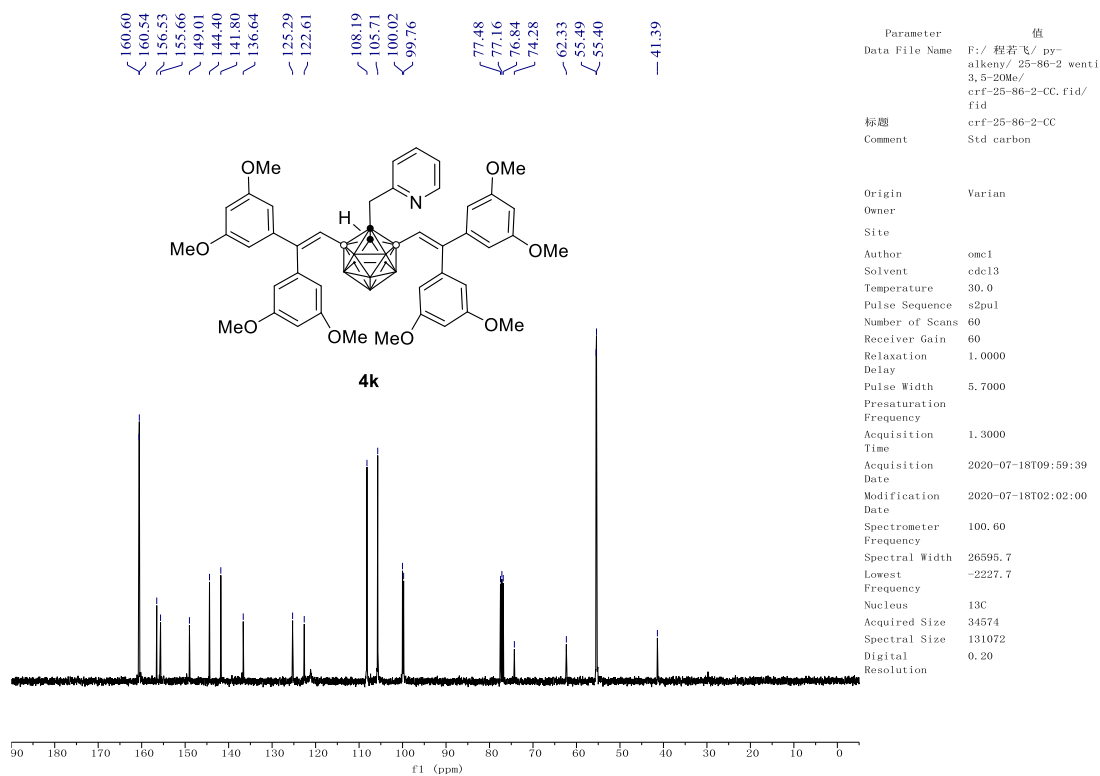
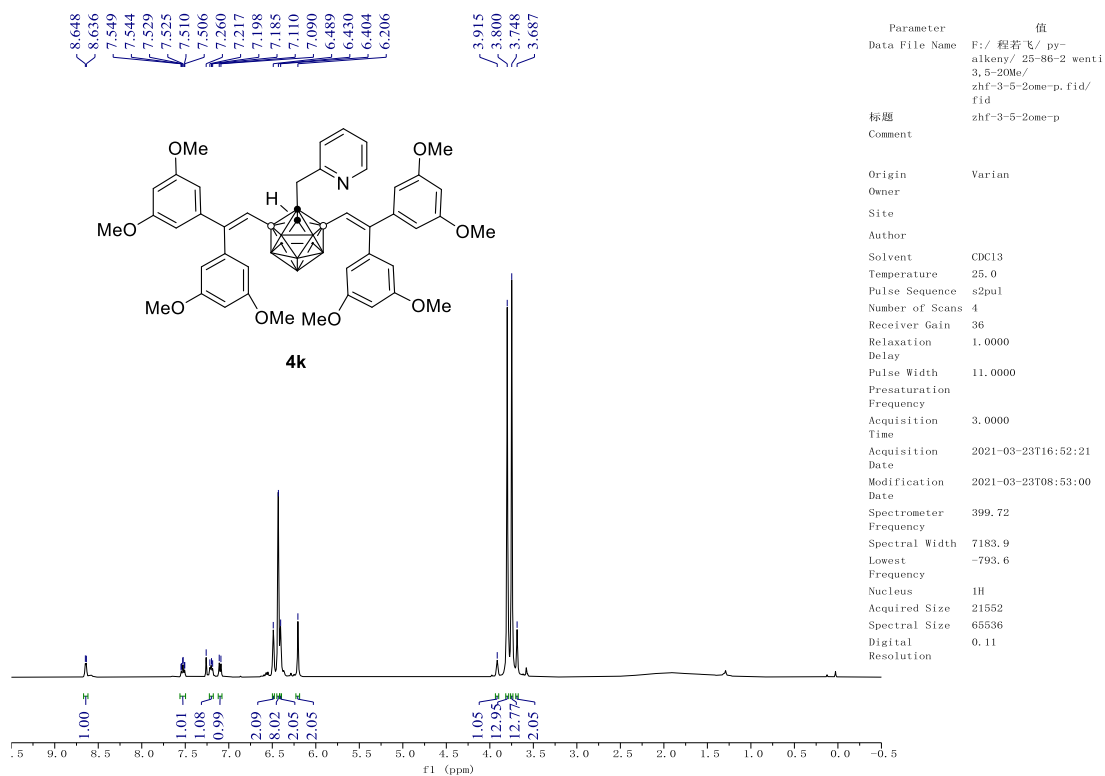


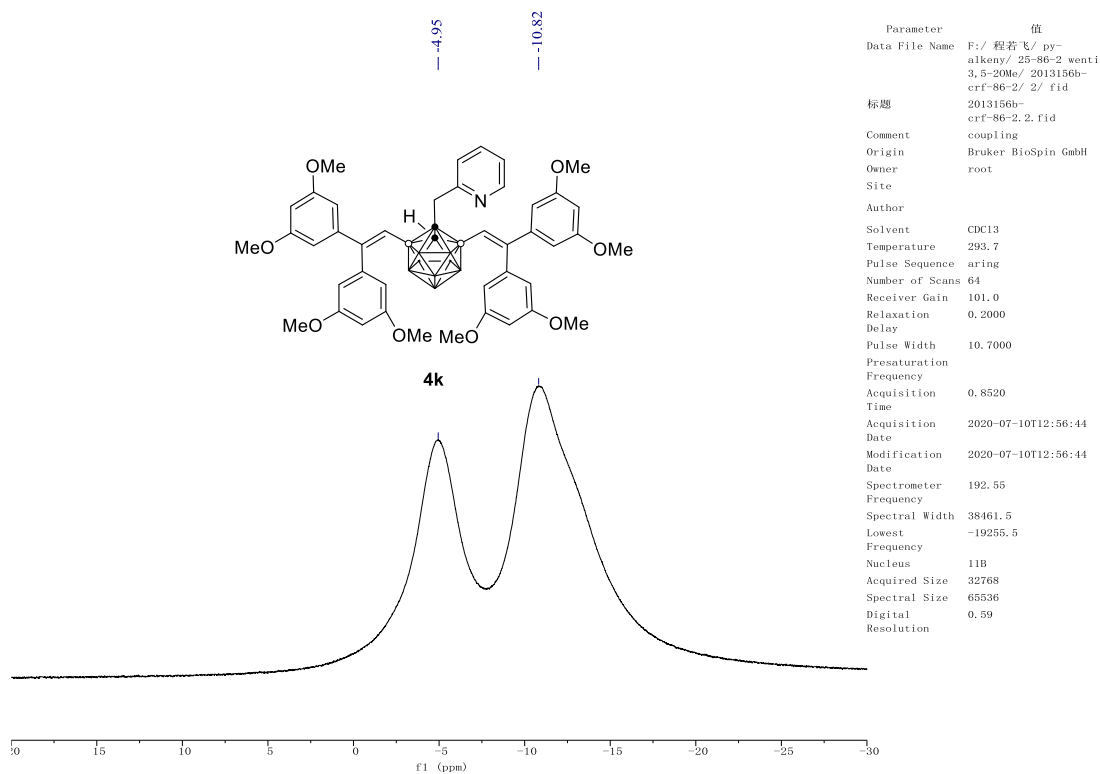
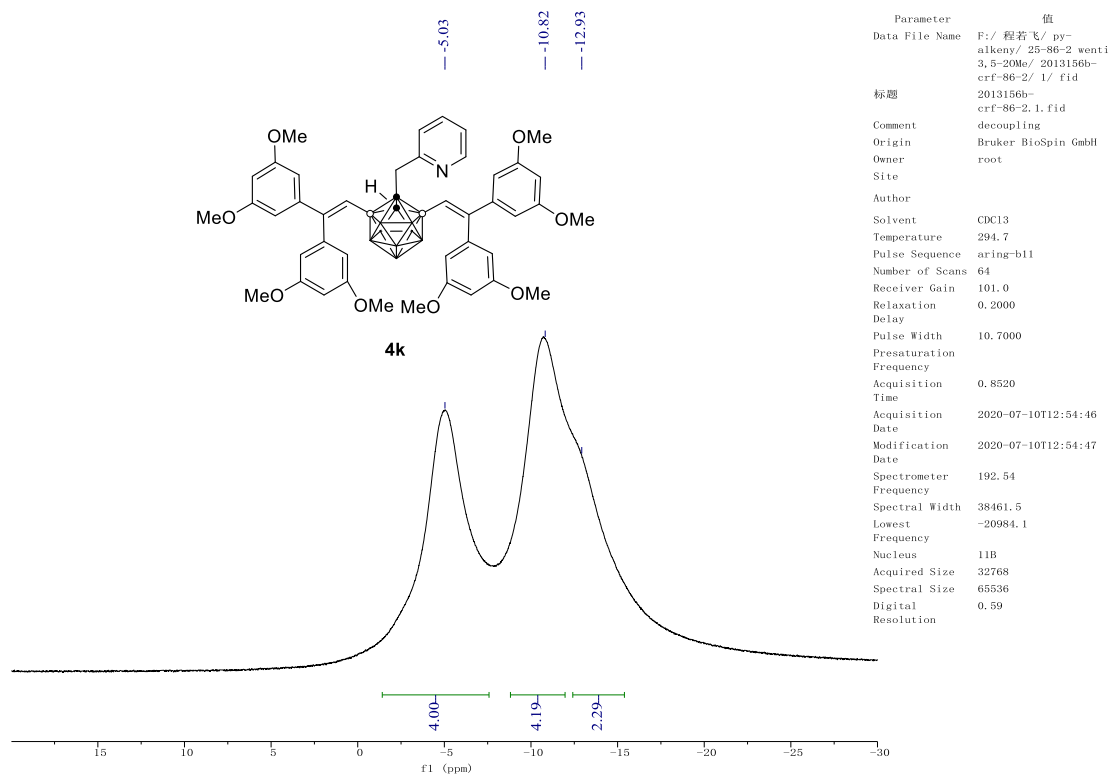


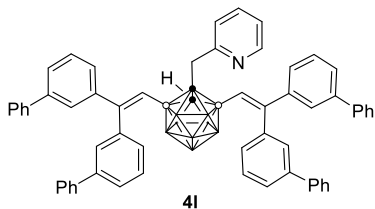
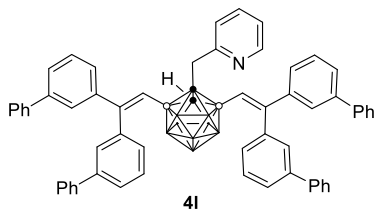


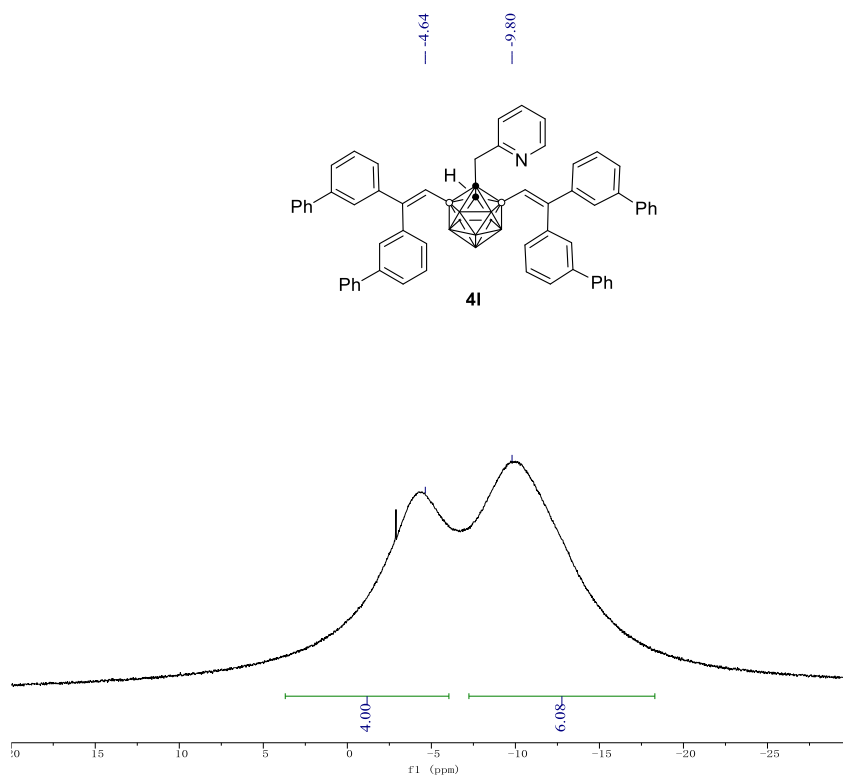




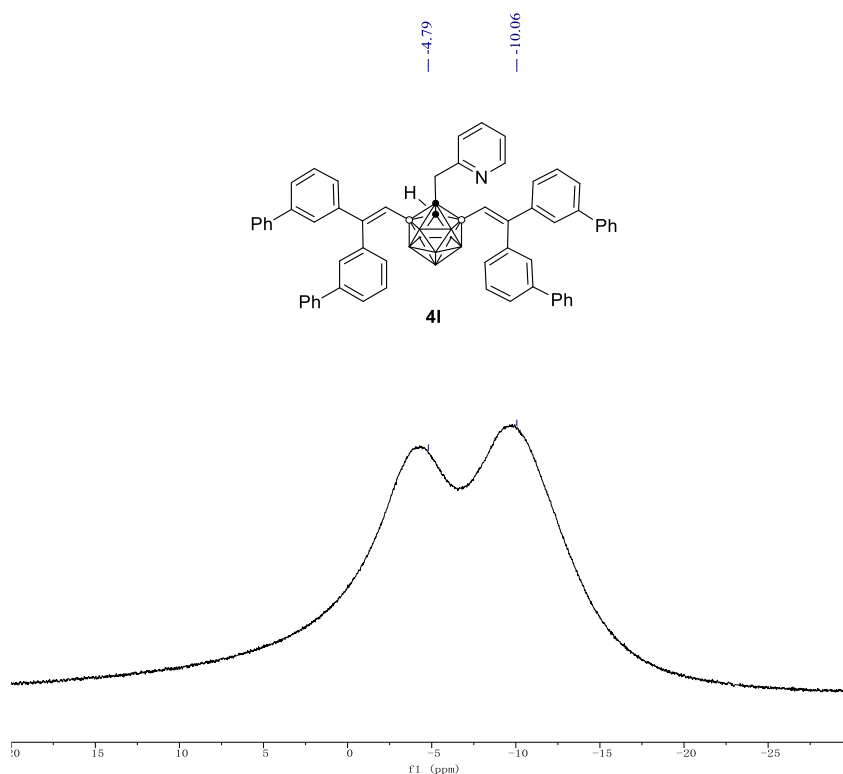




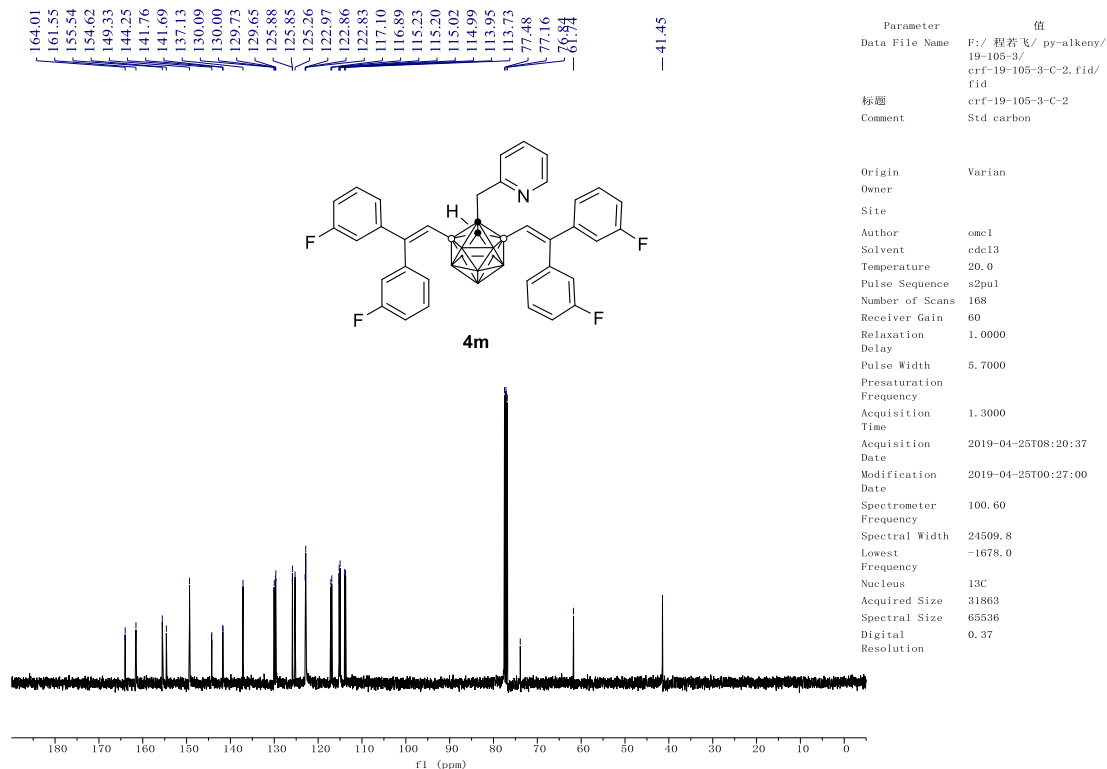
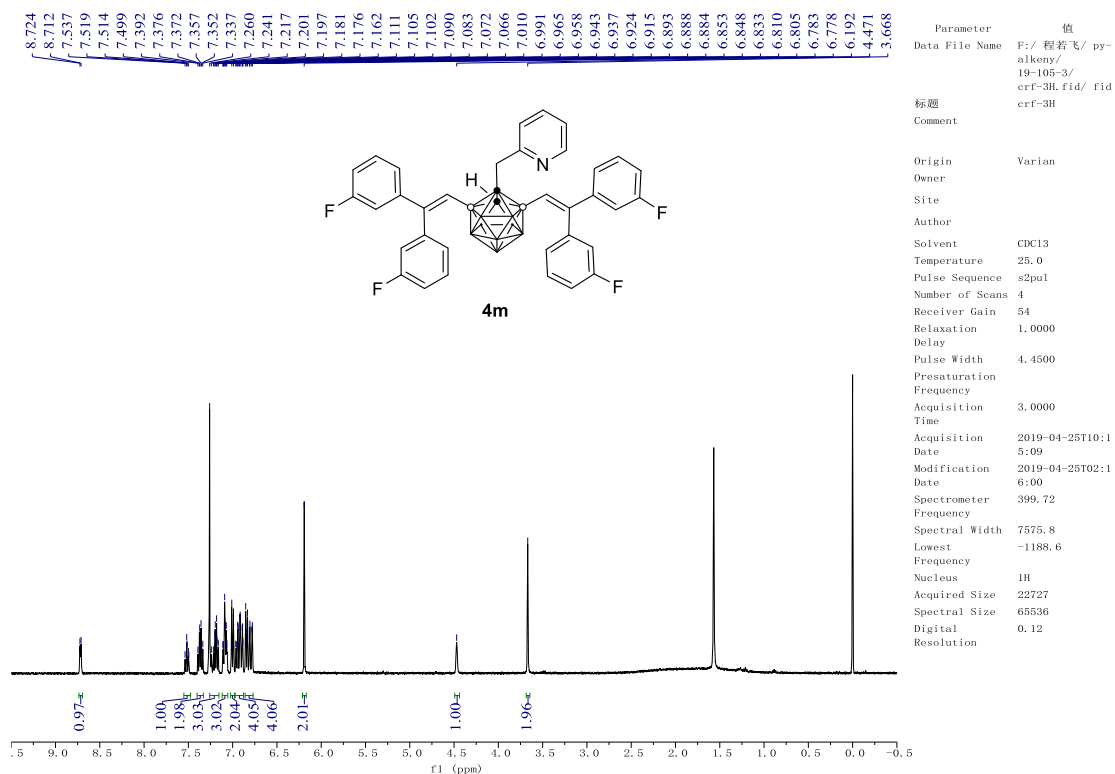


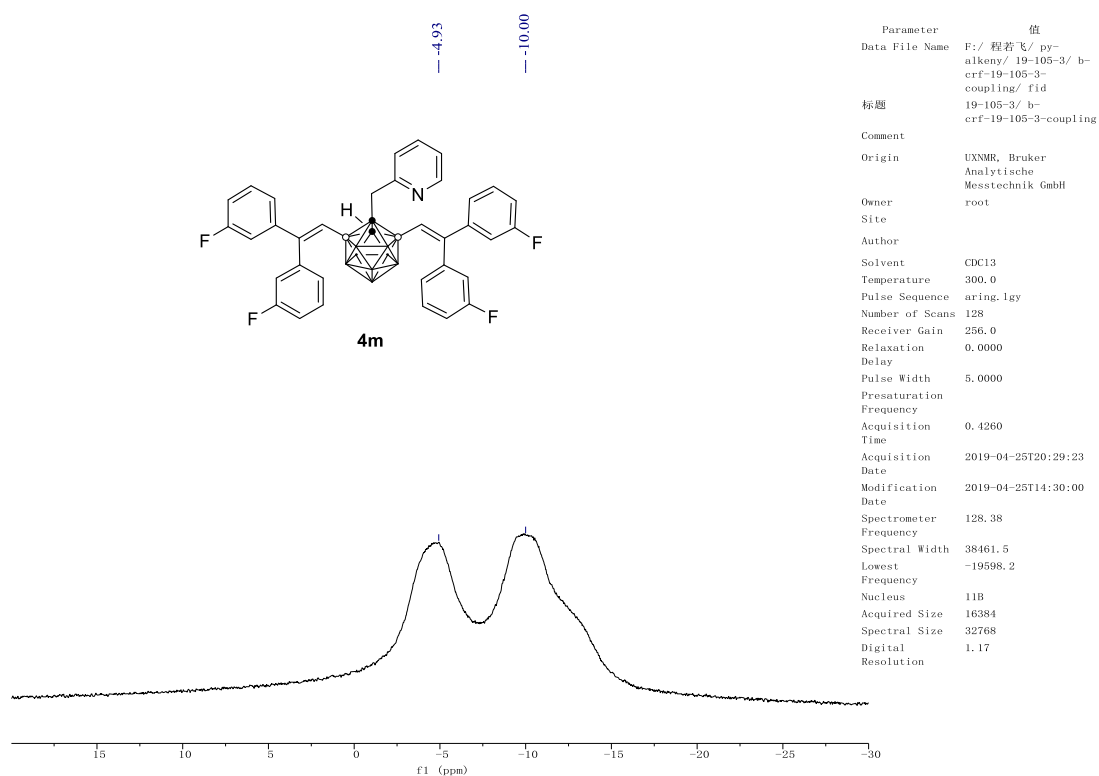
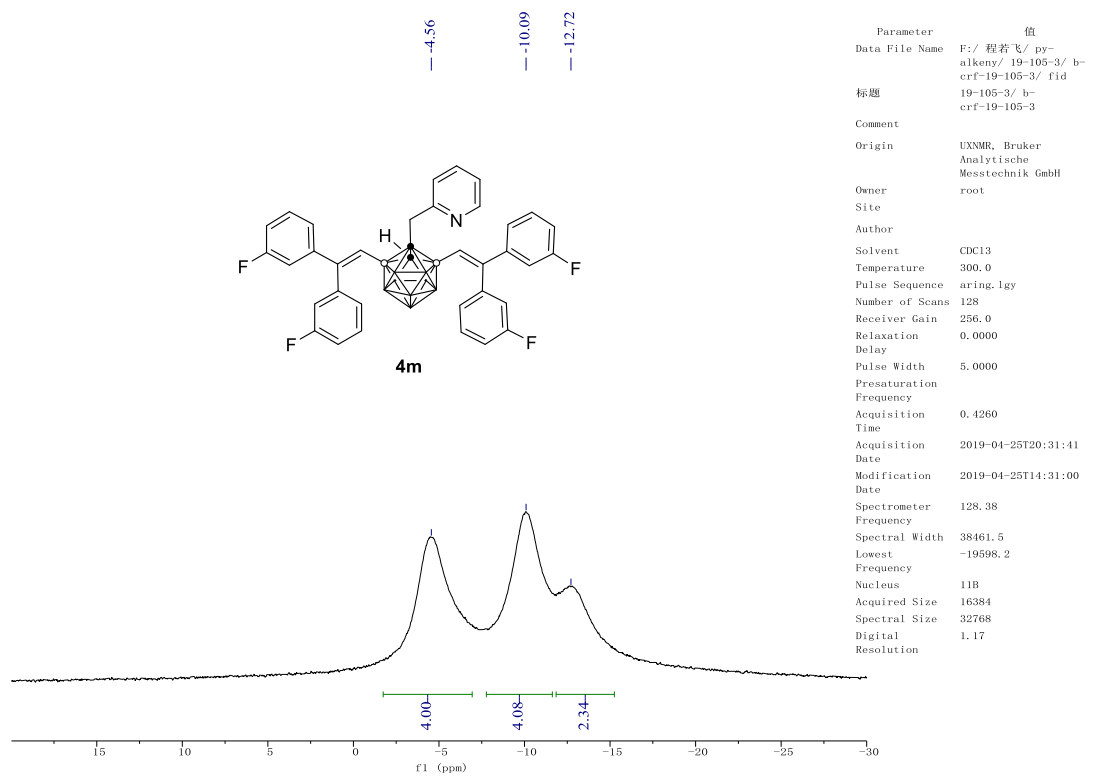


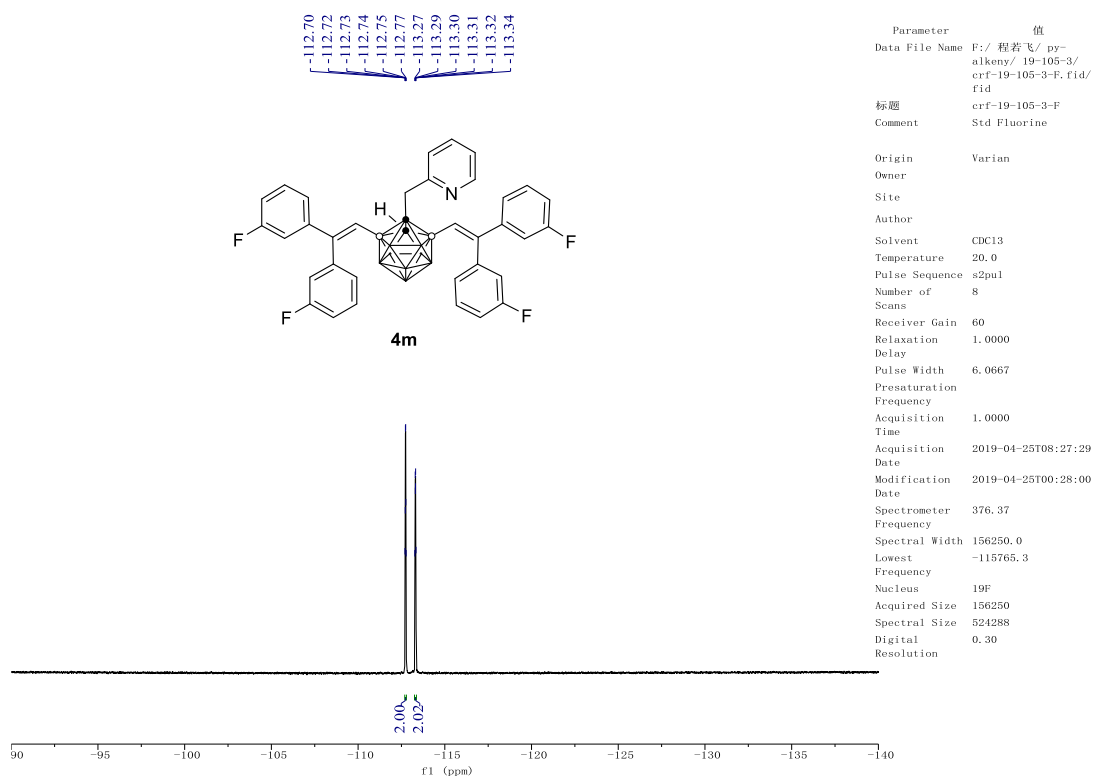
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标题	2013156b- cr-f-20-15-1.1.fid
Comment	
Origin	UXNMR, Bruker Analytische Messtechnik GmbH
Owner	root
Site	
Author	
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	aring.lgy
Number of Scans	117
Receiver Gain	114.0
Relaxation Delay	0.0000
Pulse Width	5.0000
Presaturation	
Frequency	
Acquisition Time	0.4260
Acquisition Date	2019-05-14T20:36:19
Modification	2019-05-14T13:57:56
Date	
Spectrometer	128.38
Frequency	
Spectral Width	38461.5
Lowest Frequency	-19601.7
Nucleus	11B
Acquired Size	16384
Spectral Size	32768
Digital	1.17
Resolution	

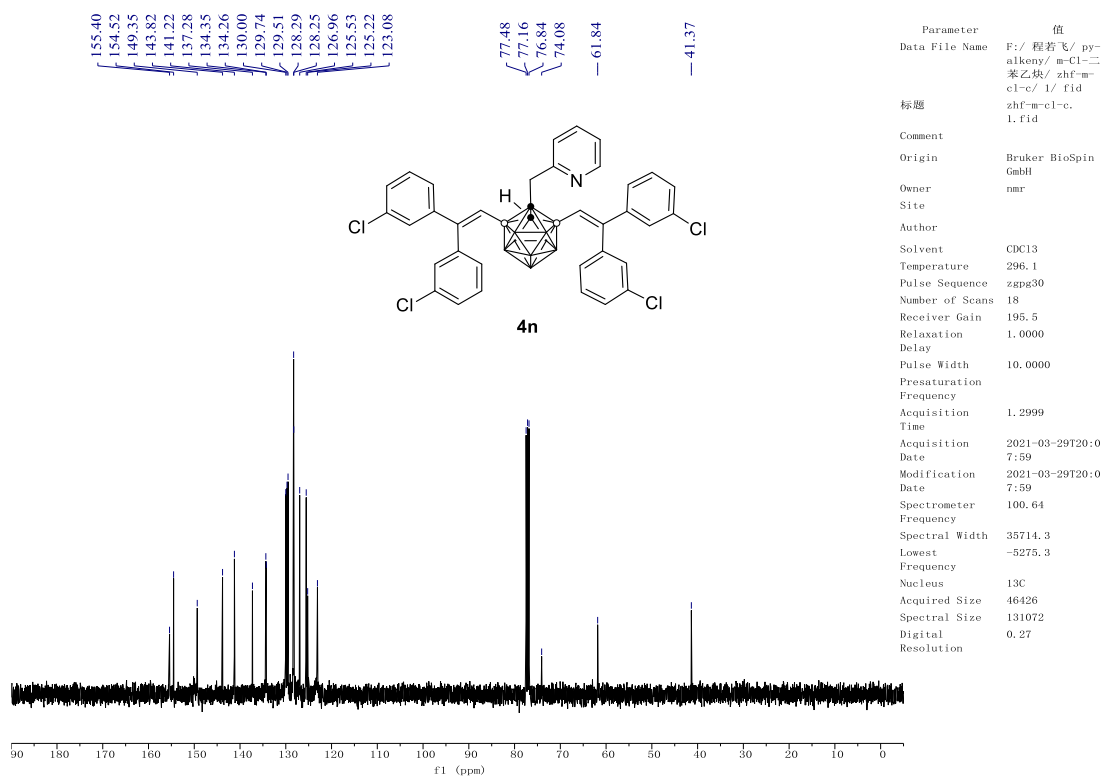
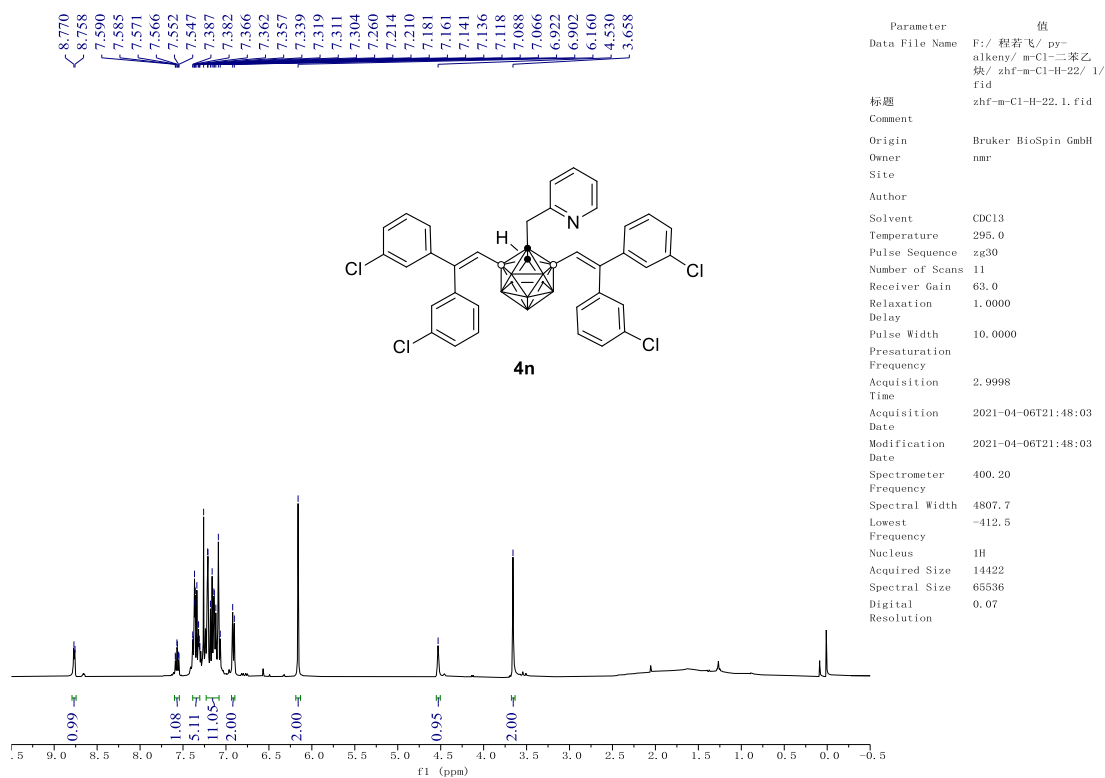


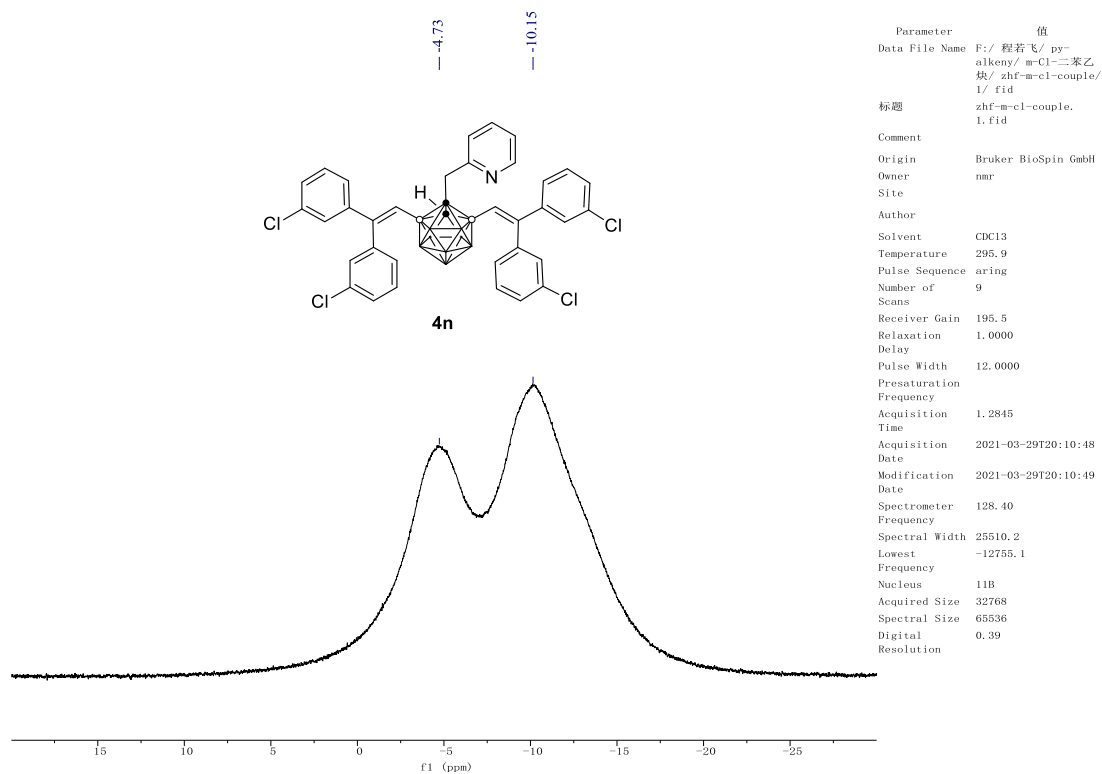
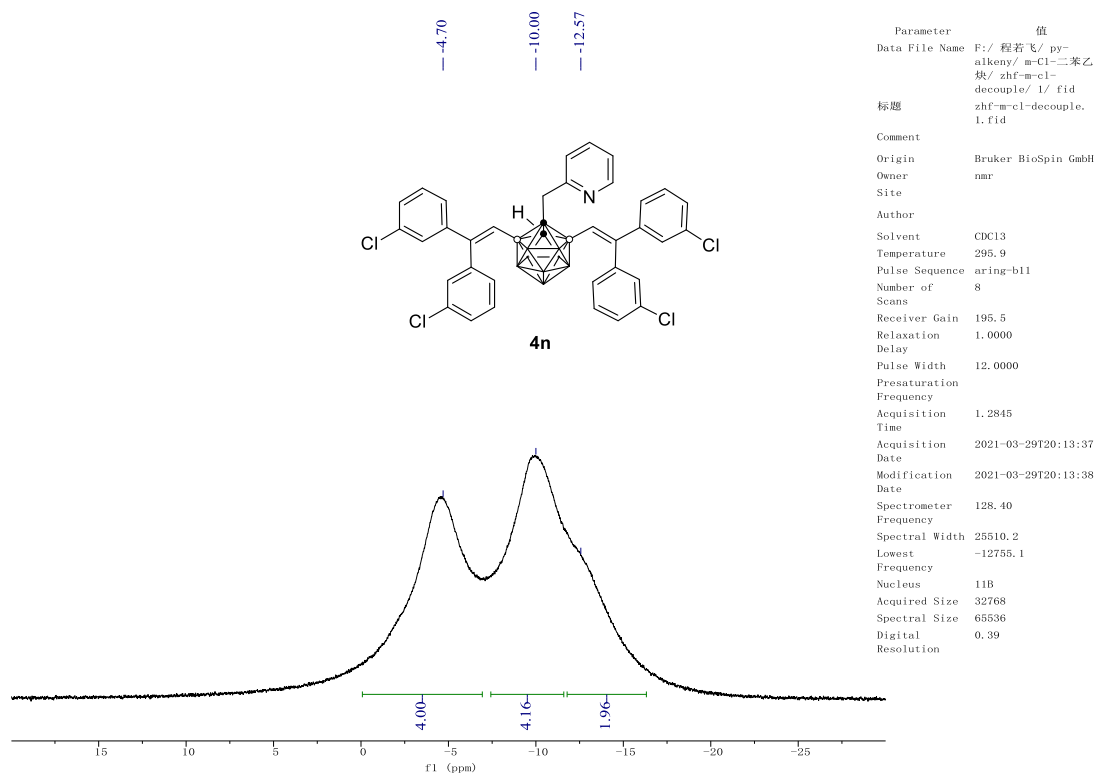
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Data File Name	F:/ 程若飞/ py-alkeny/ 20-15-1 wenti m-Ph/ 2013156b-cr-f-20-15-1- coupling/ 1/ fid
标题	2013156b-cr-f-20-15-1- coupling.1.fid
Comment	
Origin	UXNMR, Bruker Analytische Messtechnik GmbH
Owner	root
Site	
Author	
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	aring.lgy
Number of Scans	132
Receiver Gain	114.0
Relaxation Delay	0.0000
Pulse Width	5.0000
Presaturation	
Frequency	
Acquisition Time	0.4260
Acquisition Date	2019-05-14T20:37:34
Modification Date	2019-05-14T13:57:51
Spectrometer	128.38
Frequency	
Spectral Width	38461.5
Lowest Frequency	-19601.7
Nucleus	11B
Acquired Size	16384
Spectral Size	32768
Digital	1.17
Resolution	

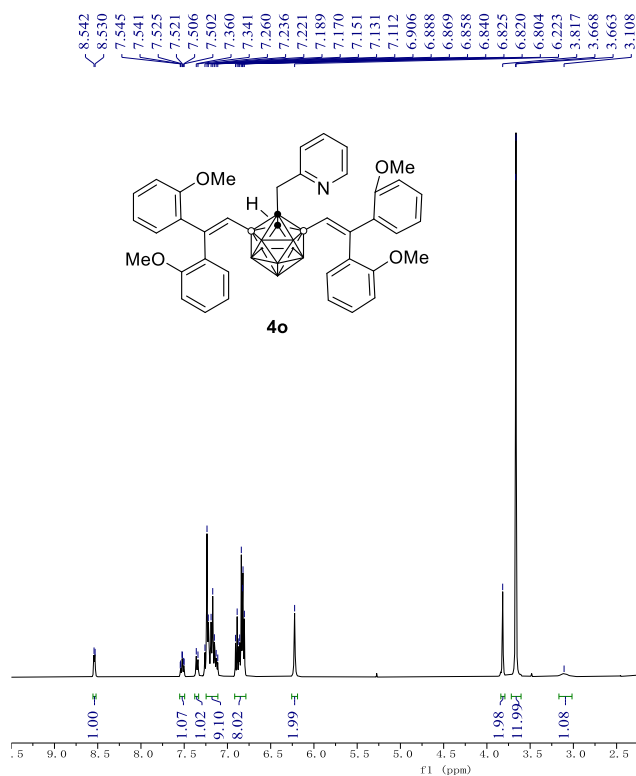






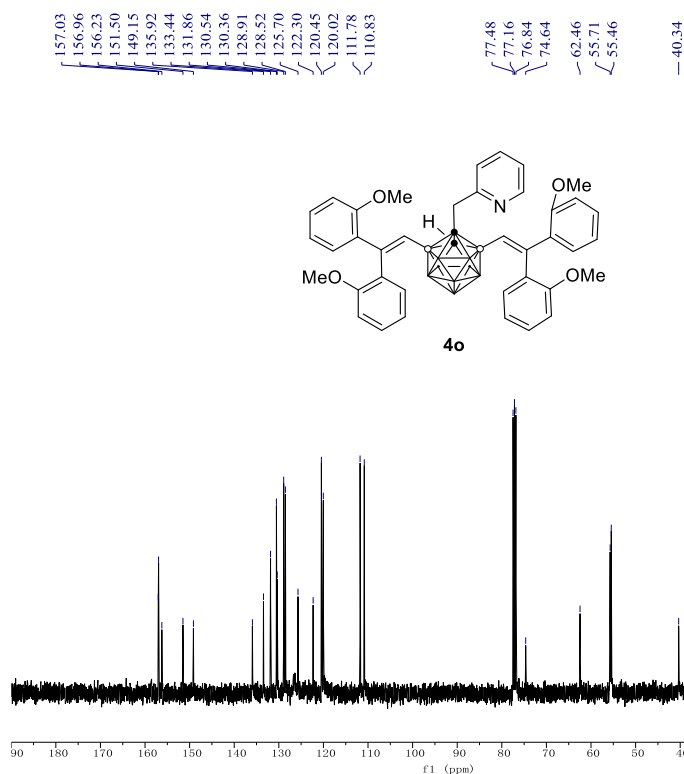






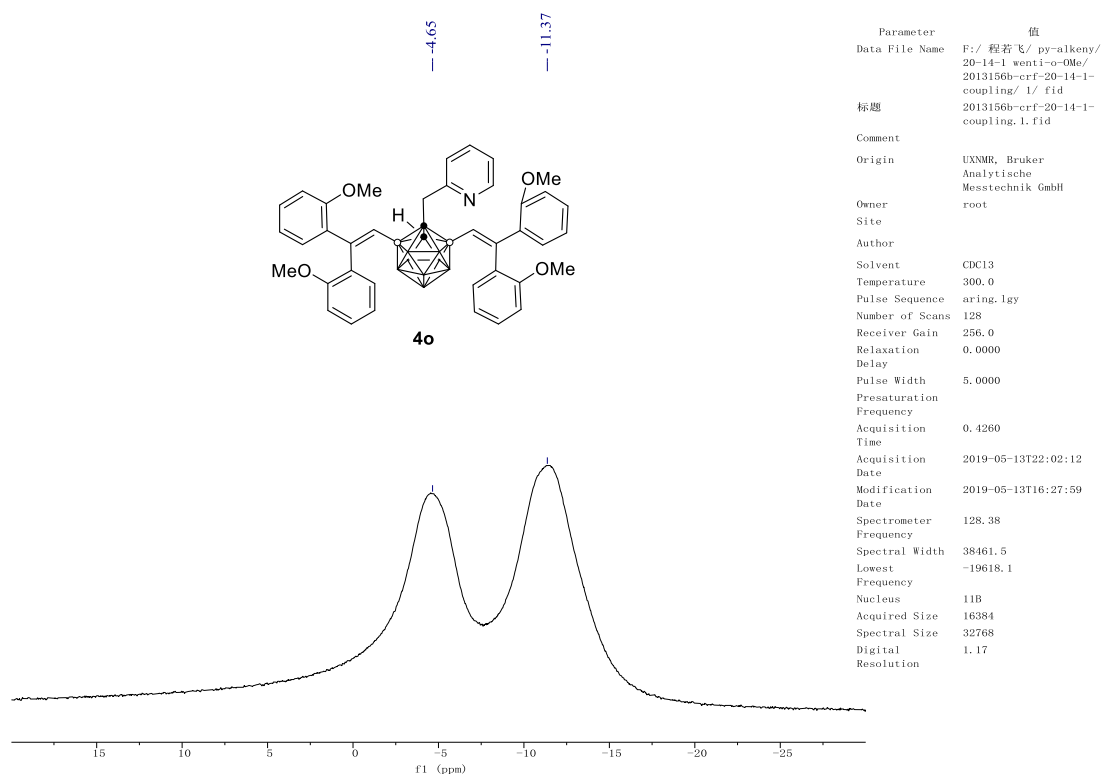
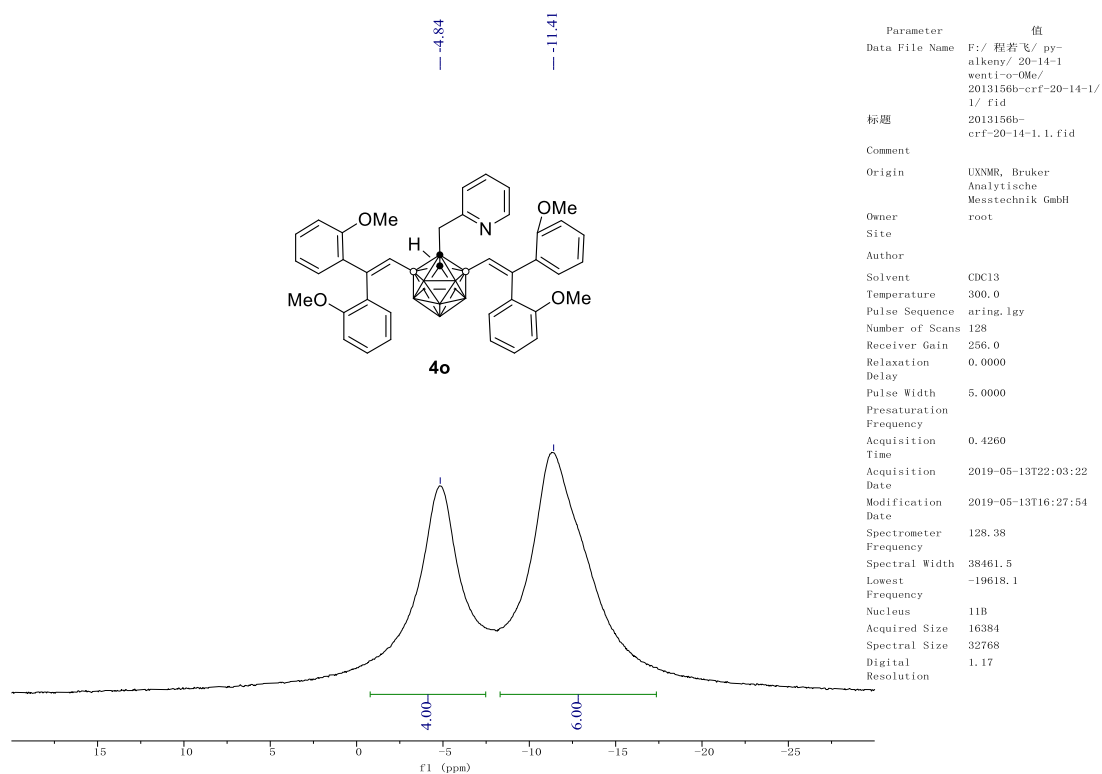
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Data File Name F:/ 程若飞/ py-alkeny/ 20-14-1 wenti-o-OMe/ crf-20-14-1- CDC13-0511.fid/ fid
标题 crf-20-14-1-CDC13-0511
Comment Std proton

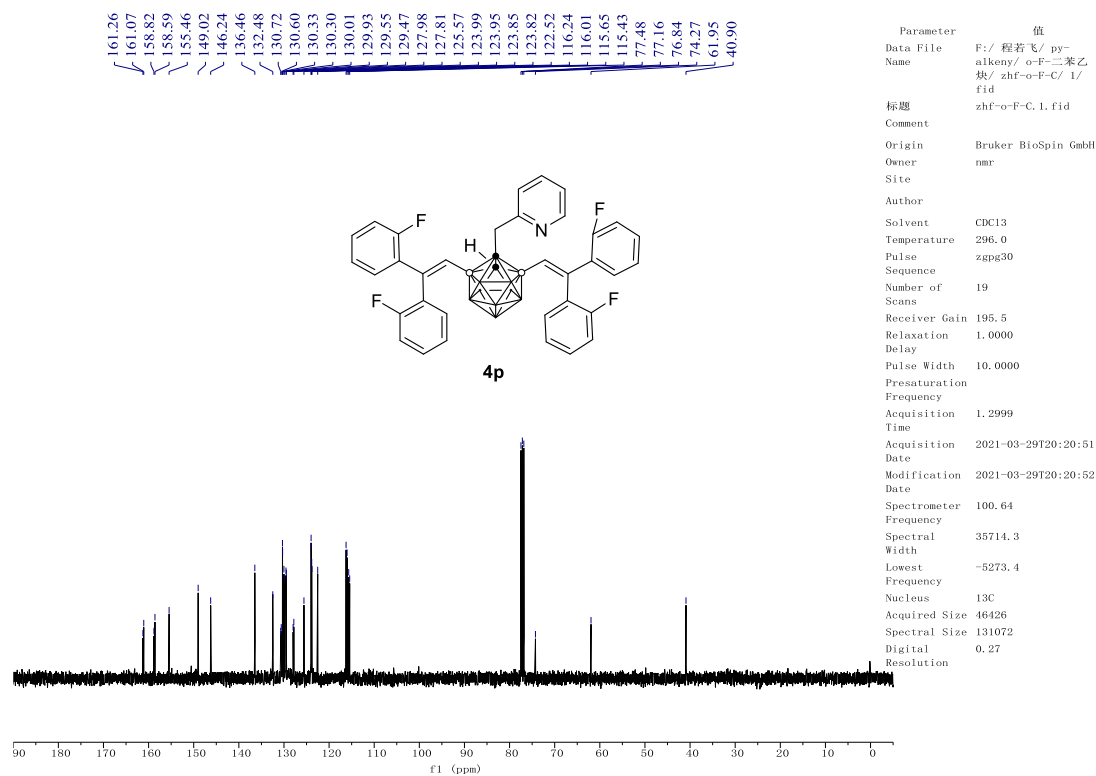
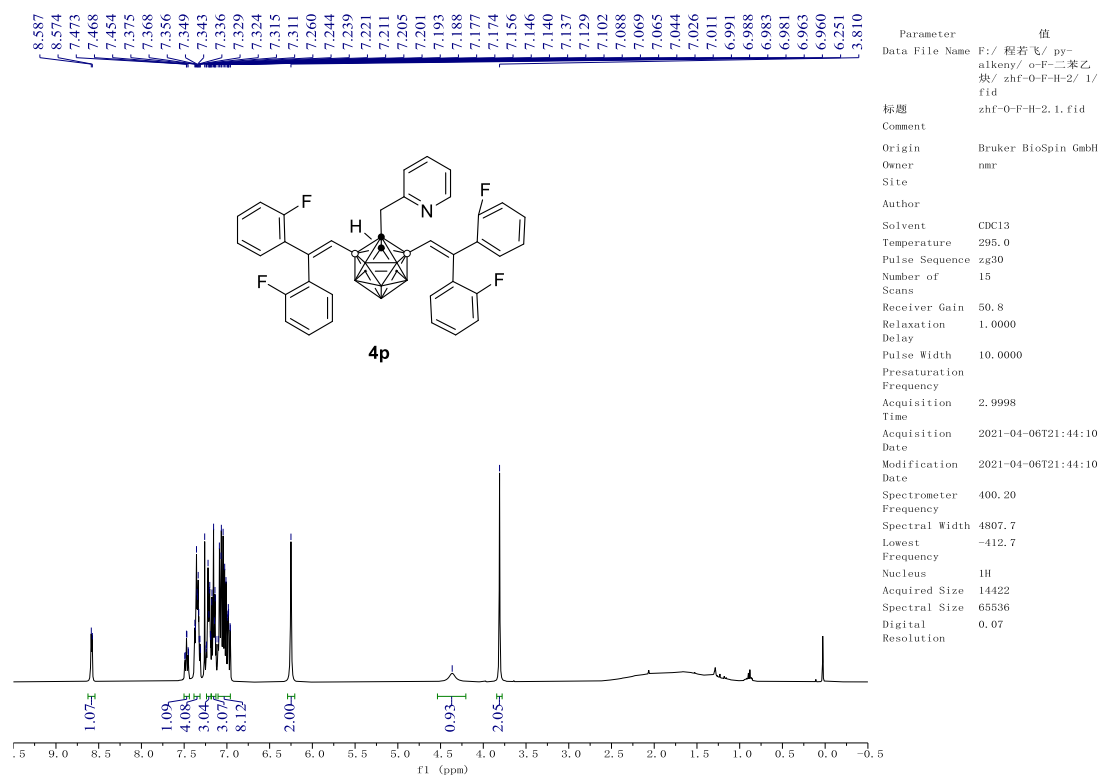
Origin Varian
Owner
Site
Author omc1
Solvent CDCl3
Temperature 20.0
Pulse Sequence s2pul
Number of Scans 24
Receiver Gain 38
Relaxation Delay 1.0000
Pulse Width 11.5000
Presaturation
Frequency
Acquisition Time 3.0000
Acquisition Date 2019-05-11T13:52:58
Modification 2019-05-11T05:55:00
Date
Spectrometer 400.03
Frequency
Spectral Width 8445.9
Lowest Frequency -2024.9
Nucleus 1H
Acquired Size 25338
Spectral Size 65536
Digital 0.13
Resolution

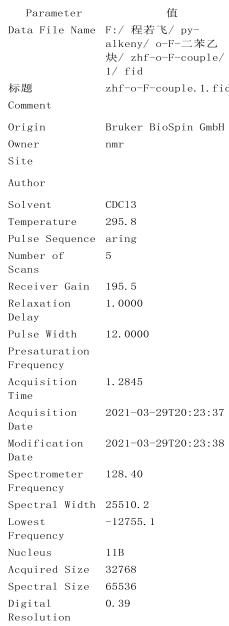
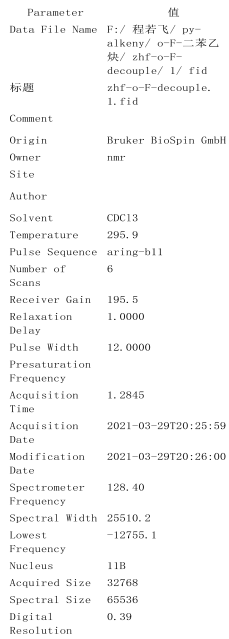


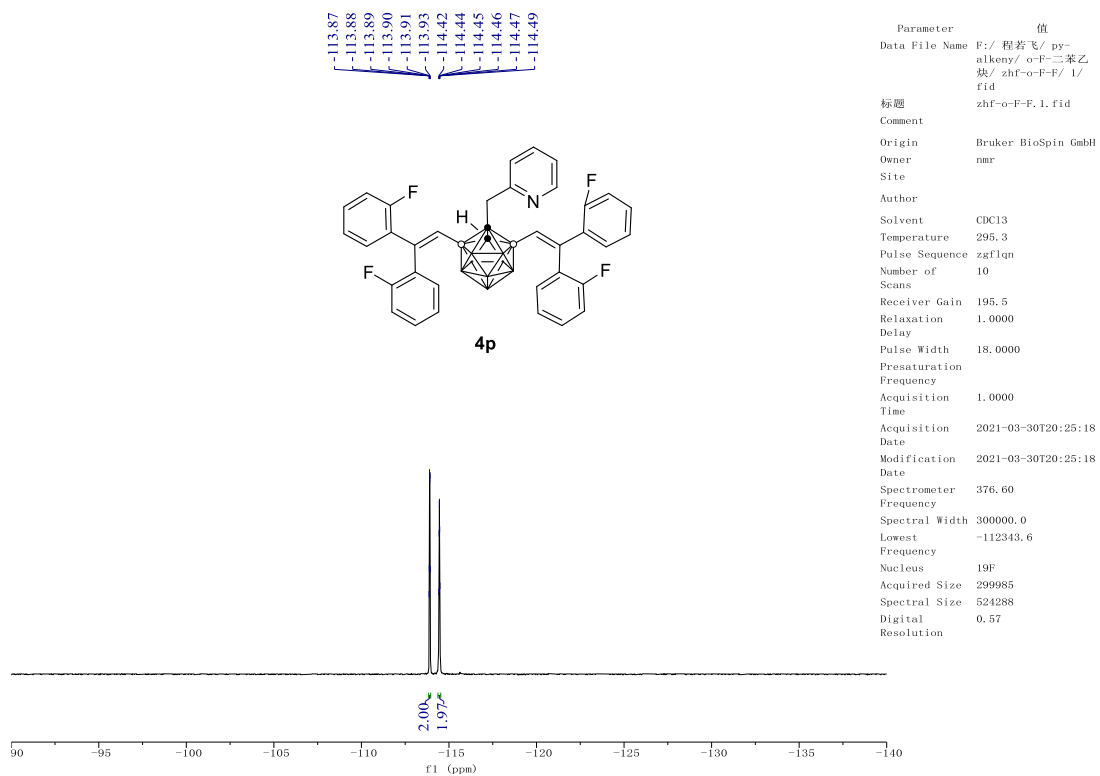
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Comment Std carbon

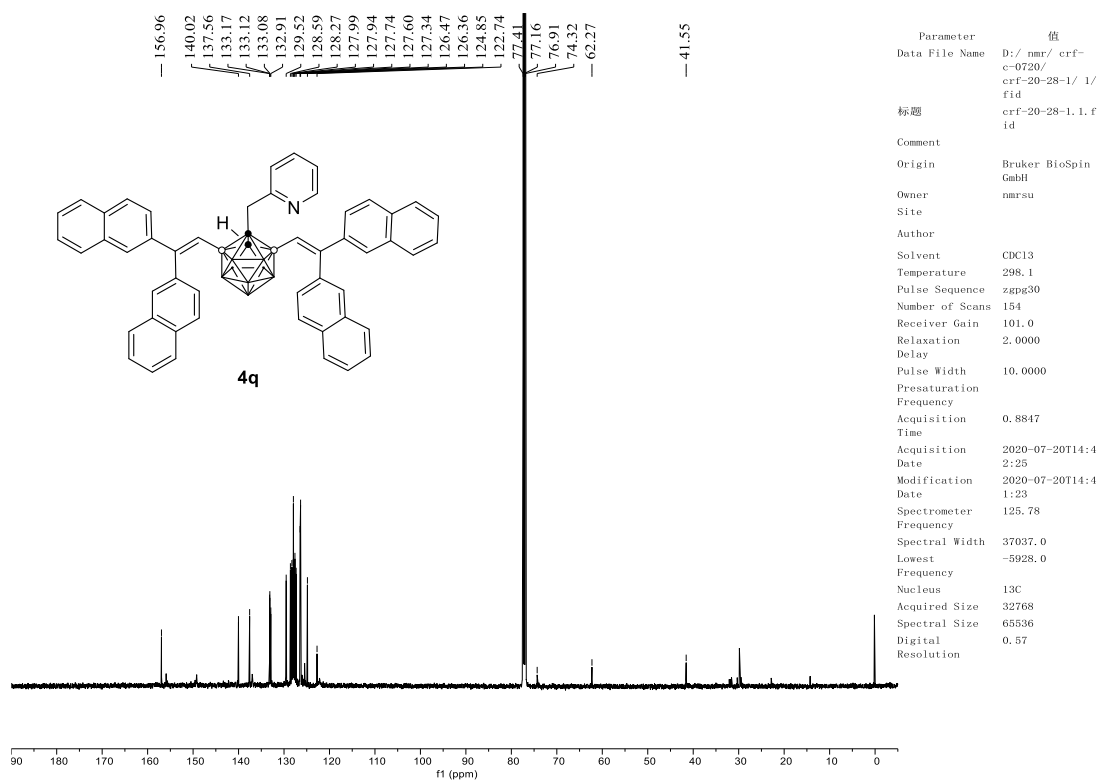
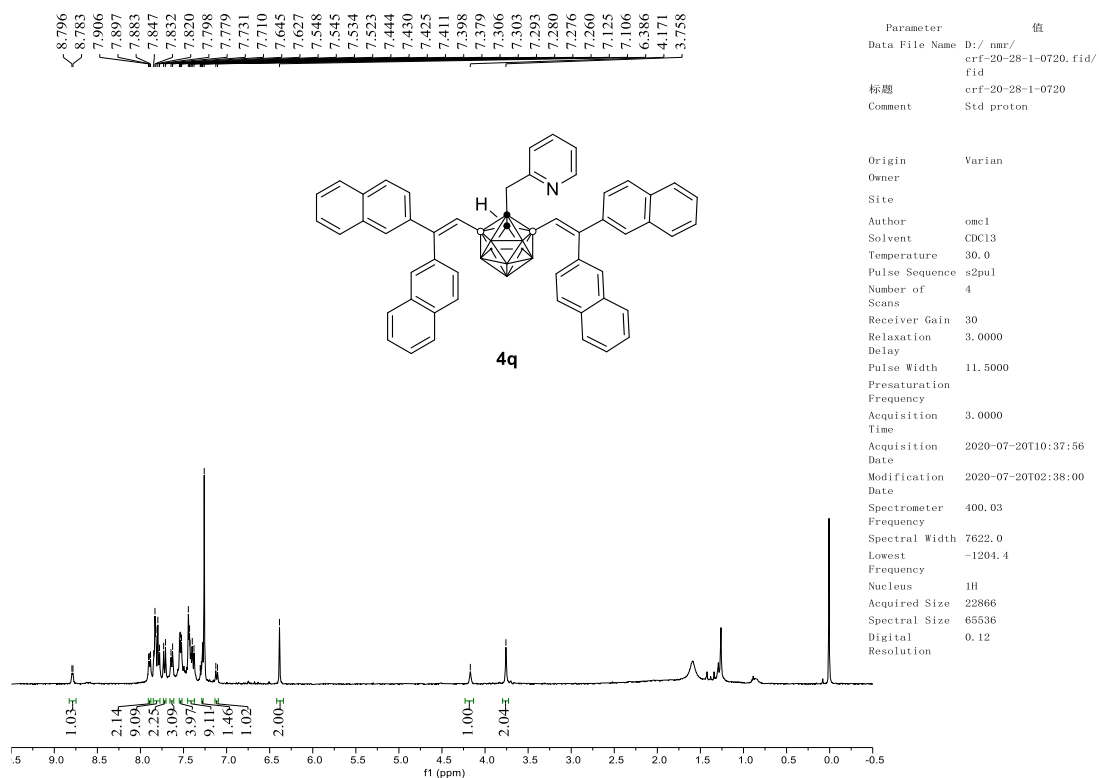
Origin Varian
Owner
Site
Author omc1
Solvent cdcl3
Temperature 20.0
Pulse Sequence s2pul
Number of Scans 84
Receiver Gain 60
Relaxation Delay 1.0000
Pulse Width 5.7000
Presaturation
Frequency
Acquisition Time 1.3000
Acquisition Date 2019-05-11T13:56:28
Modification 2019-05-11T05:59:00
Date
Spectrometer 100.60
Frequency
Spectral Width 24509.8
Lowest Frequency -1682.0
Nucleus 13C
Acquired Size 31863
Spectral Size 65536
Digital 0.37
Resolution

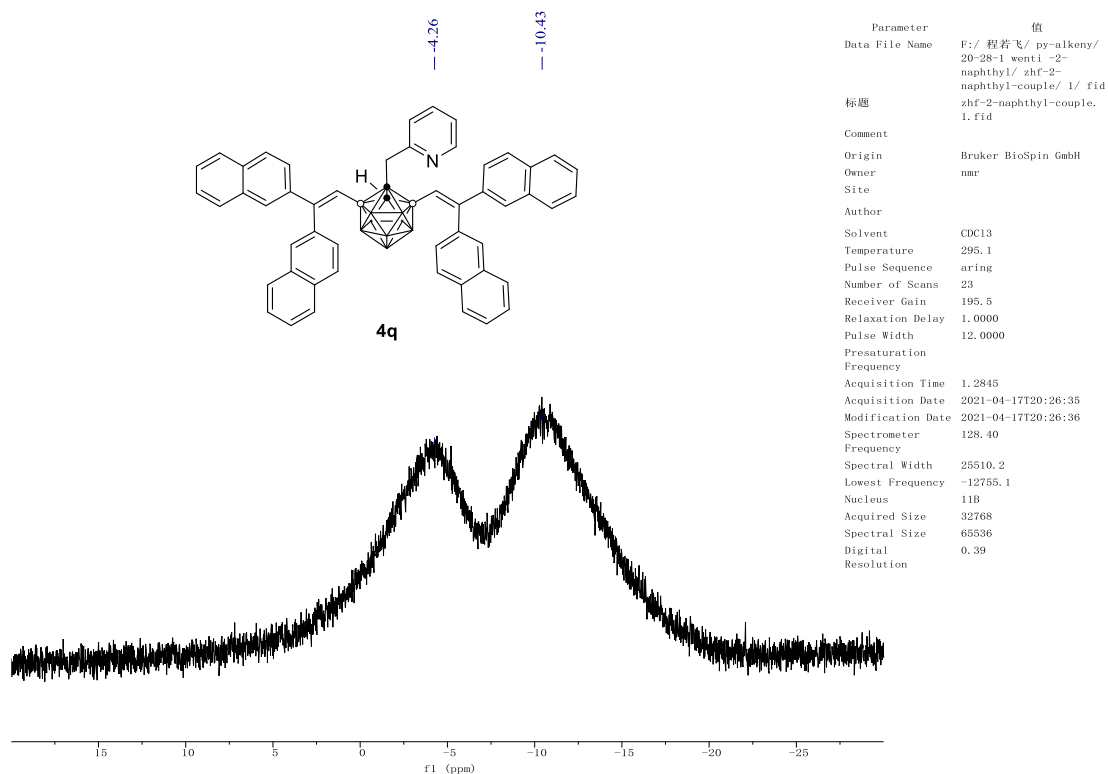
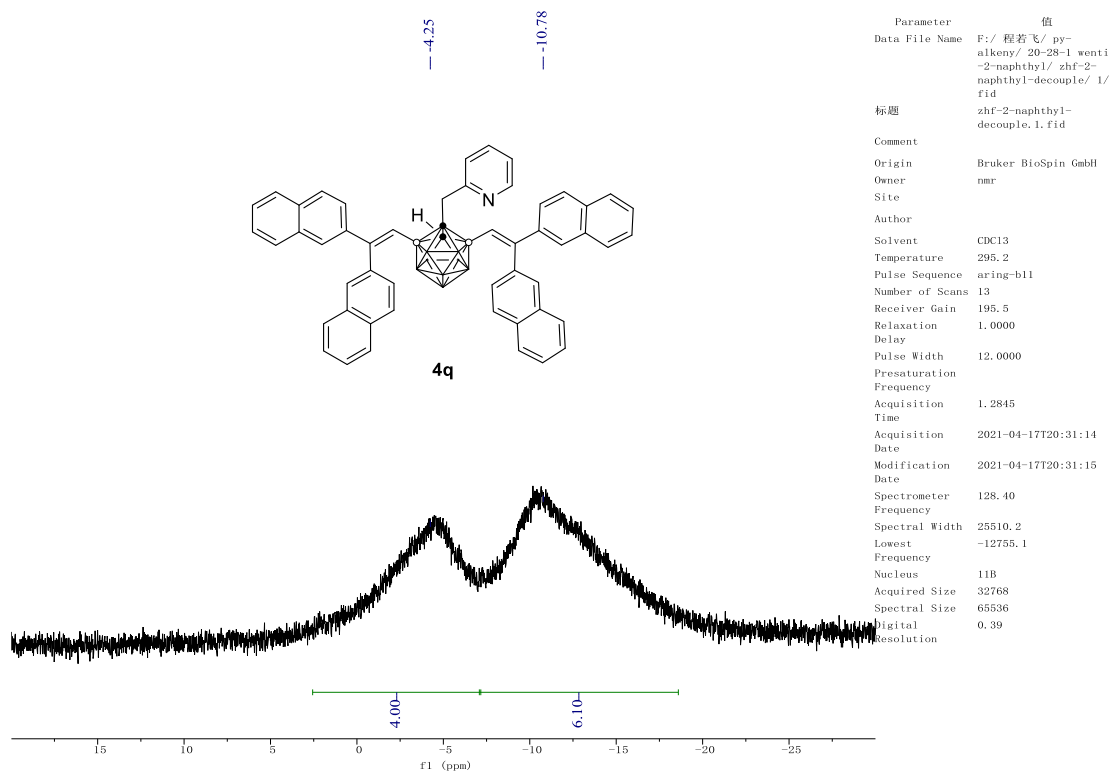


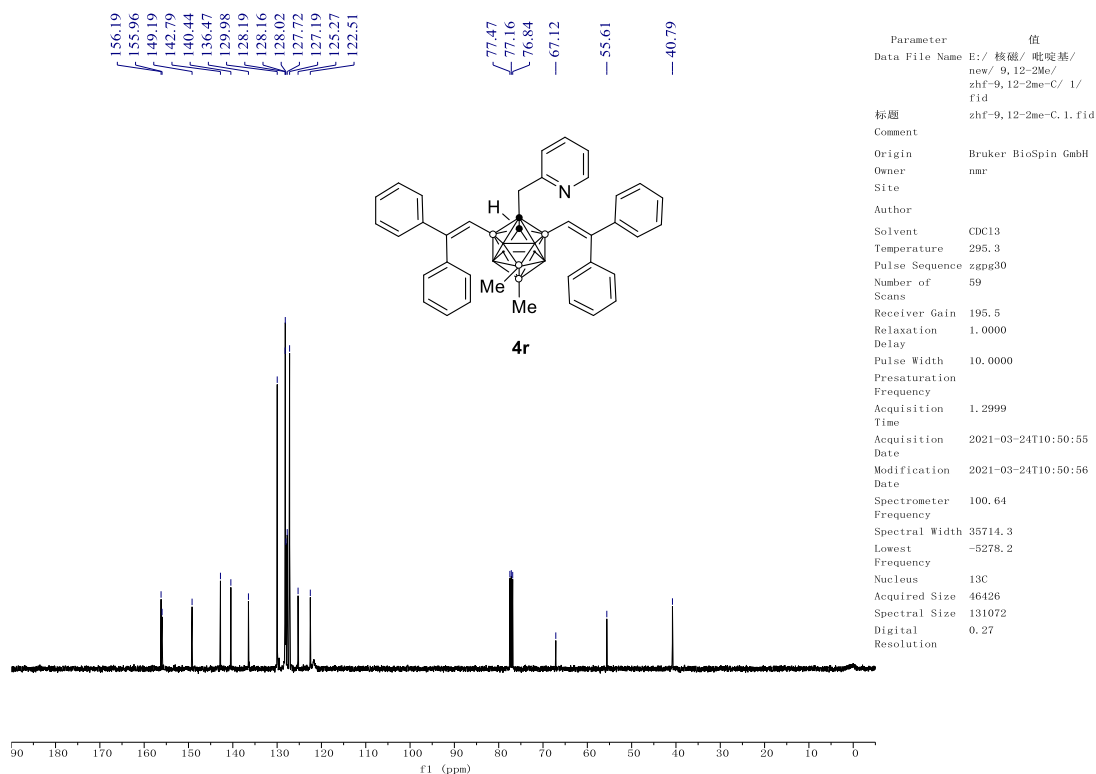
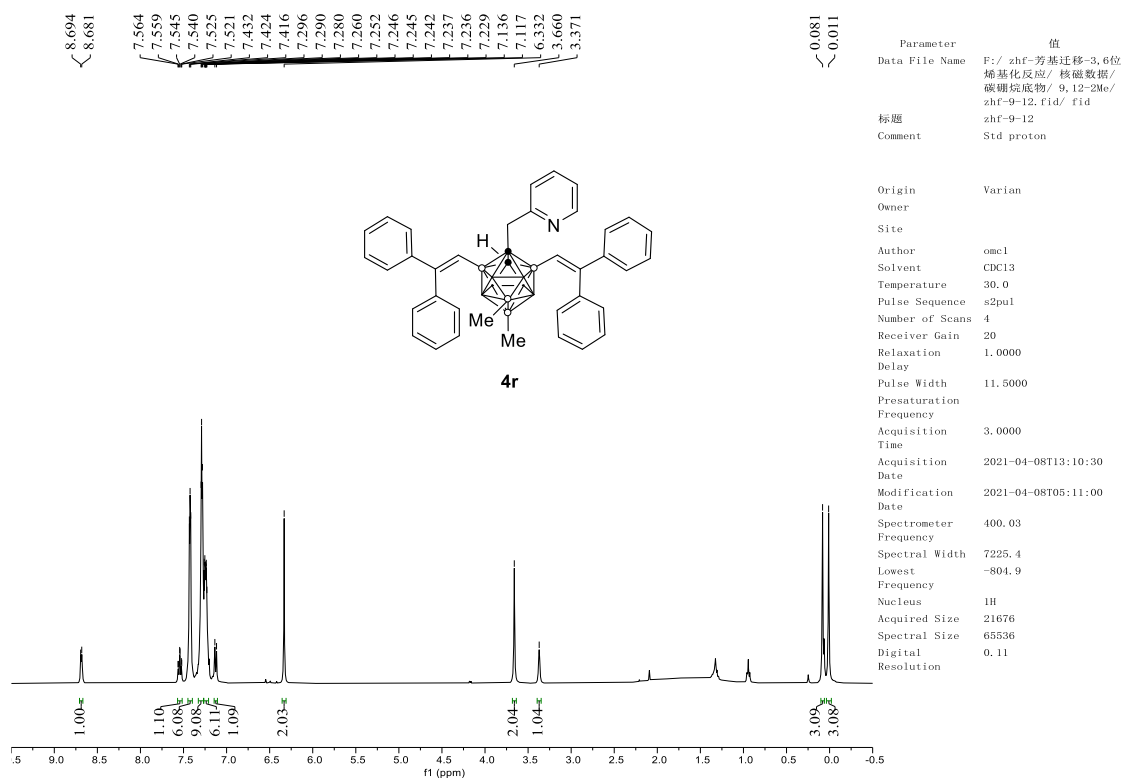


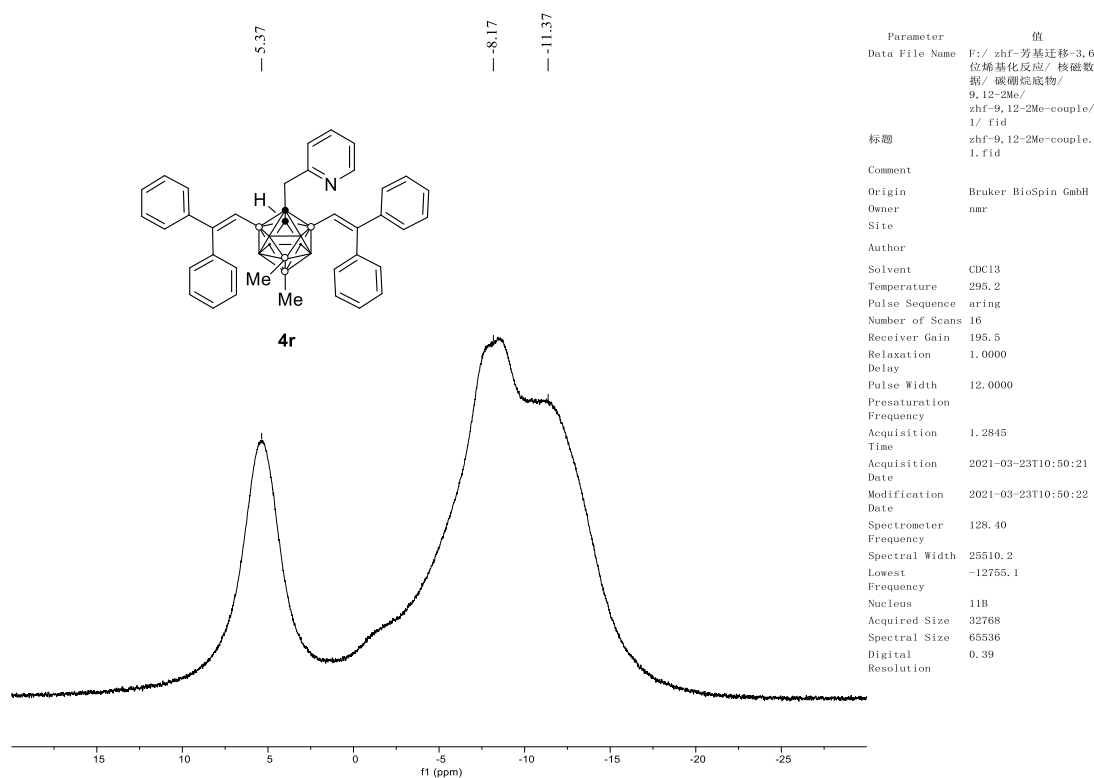
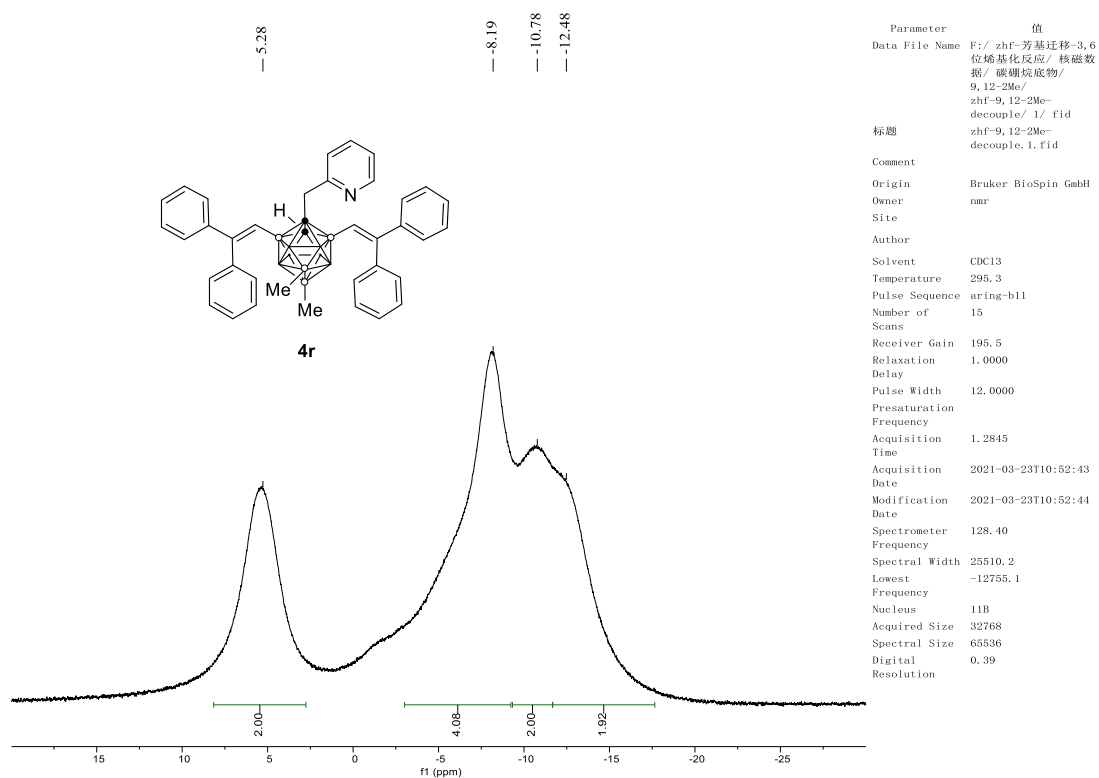


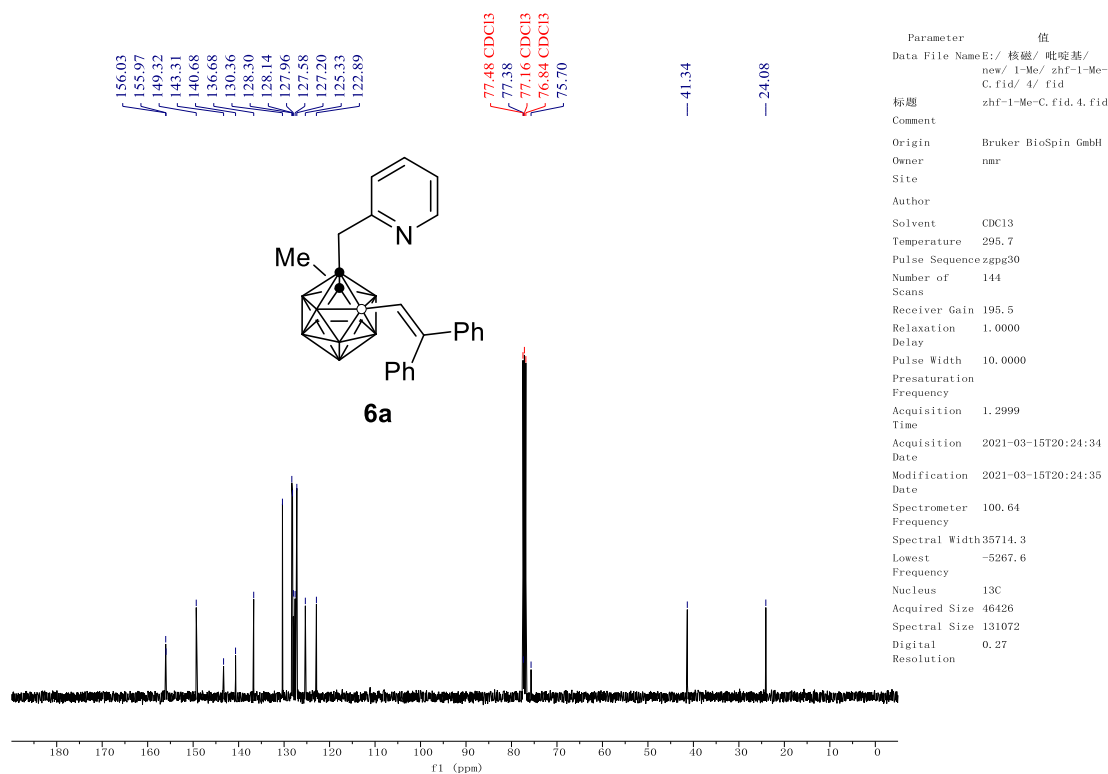
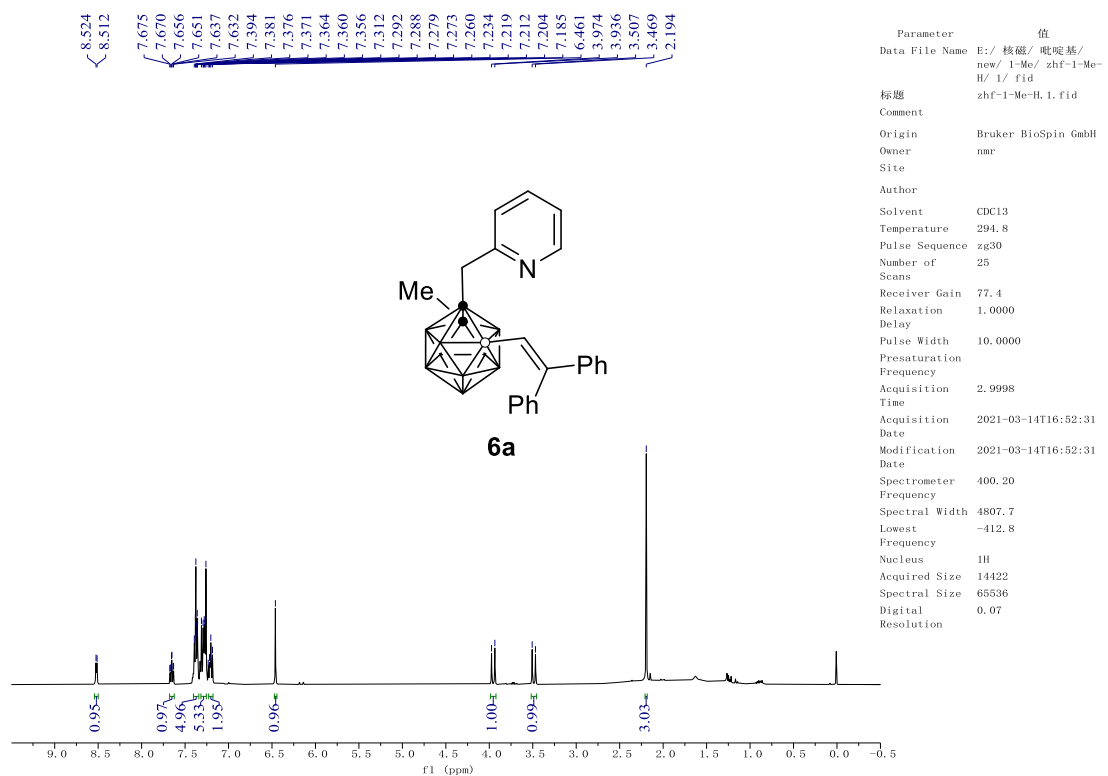


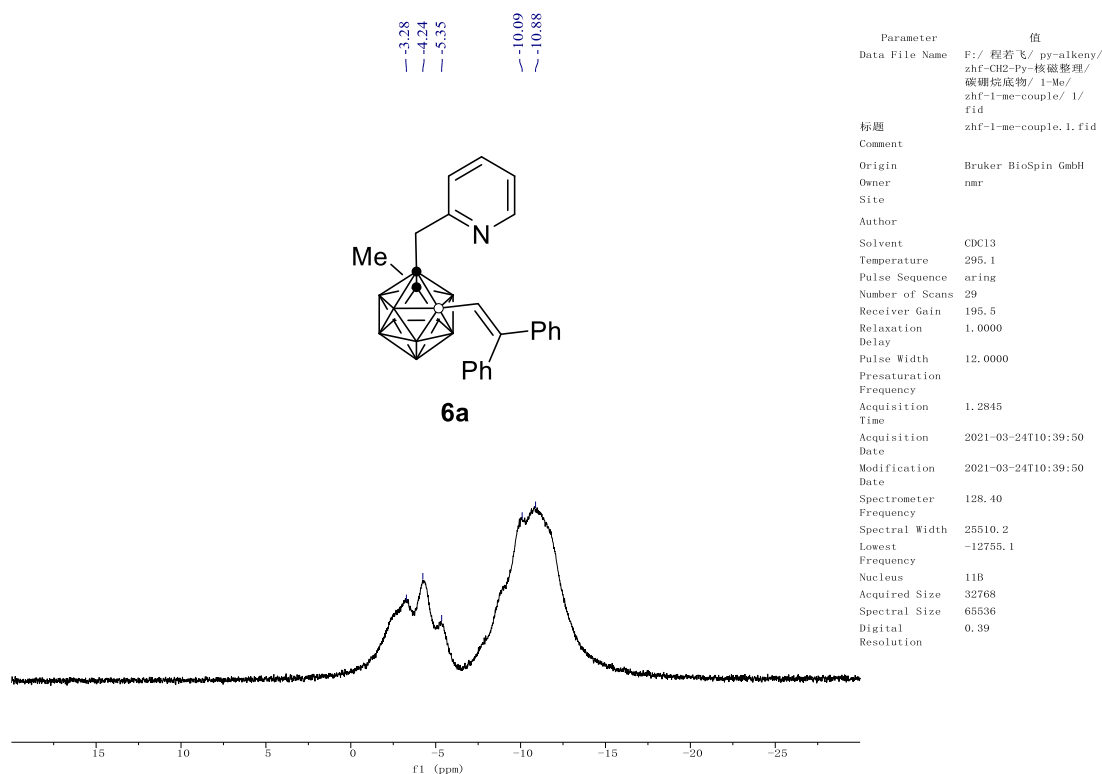
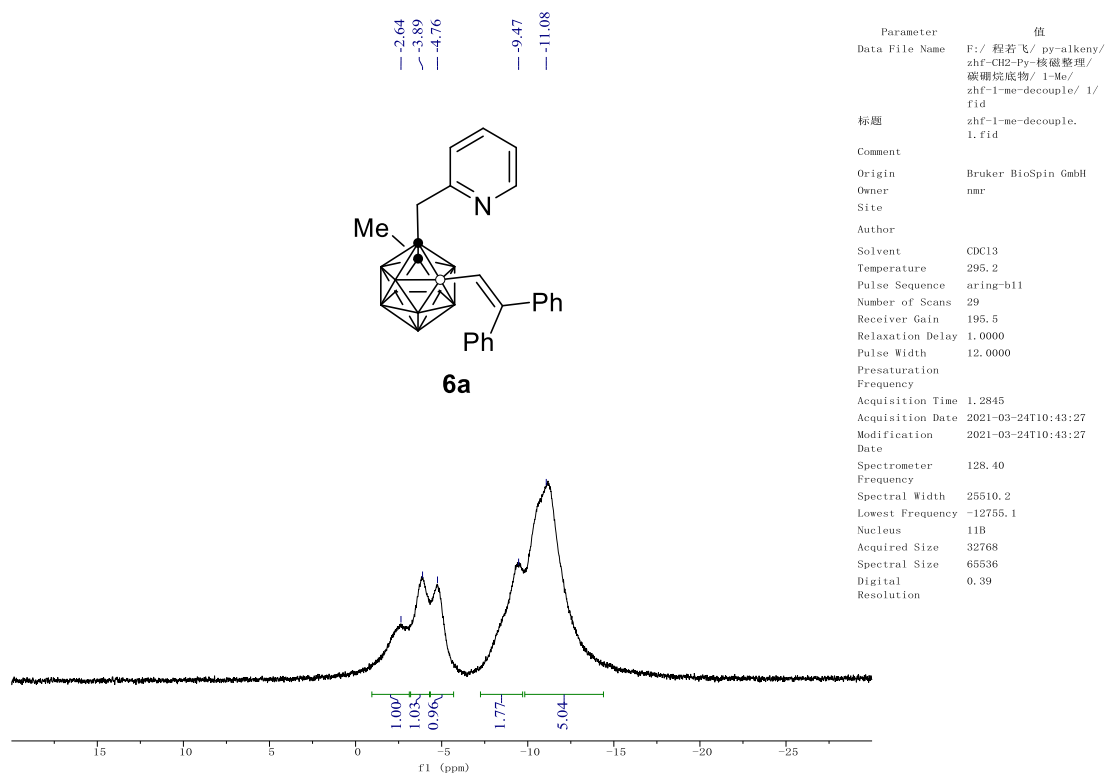


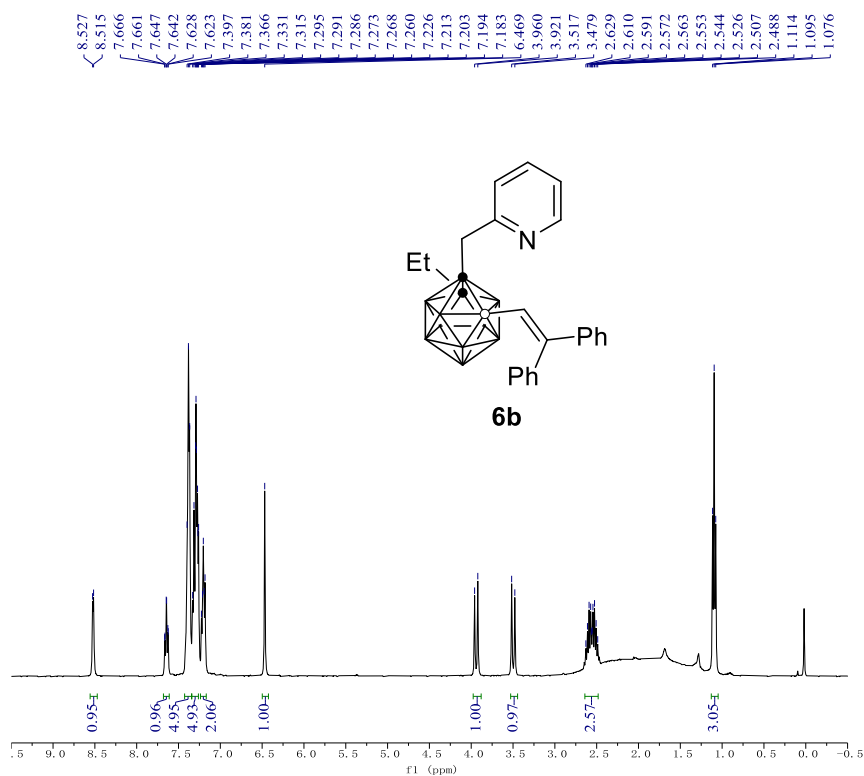




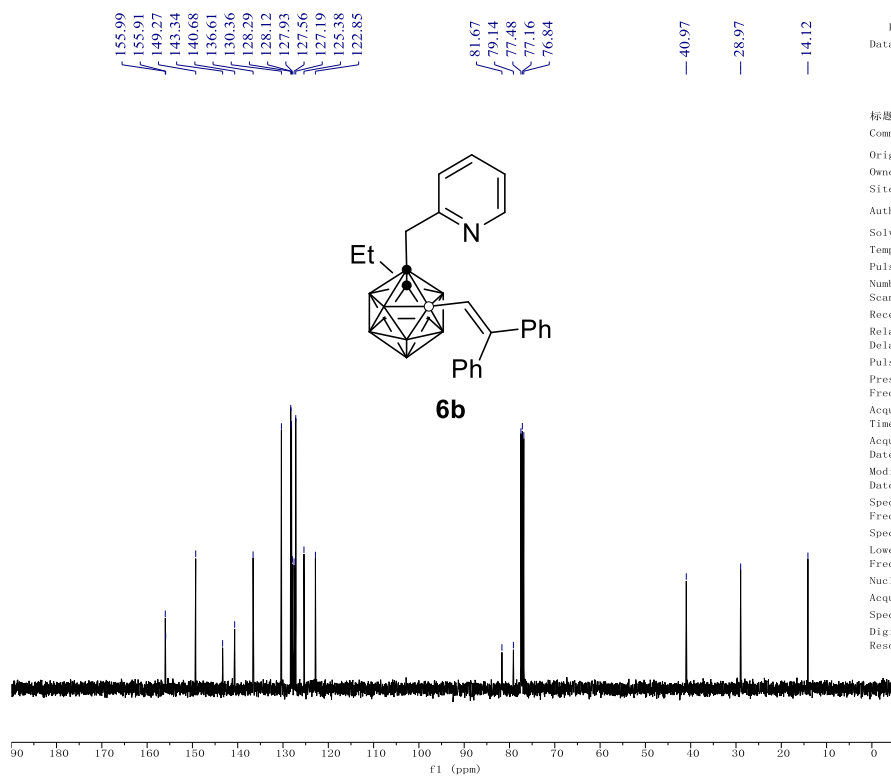




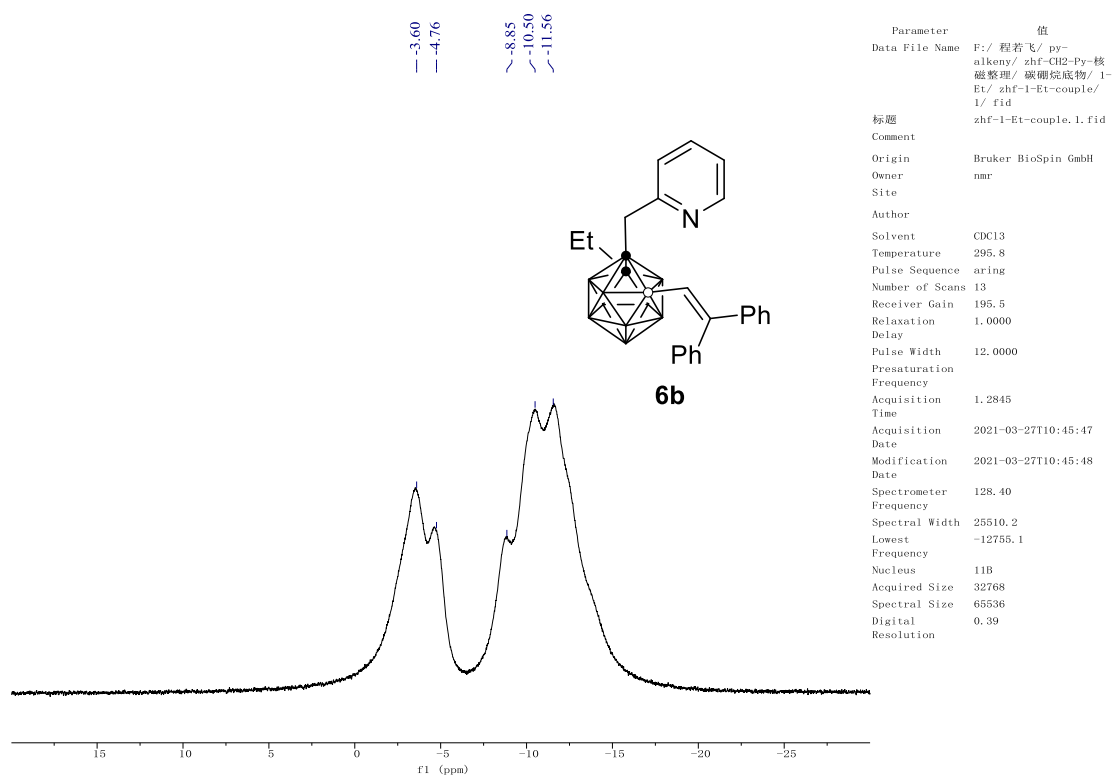
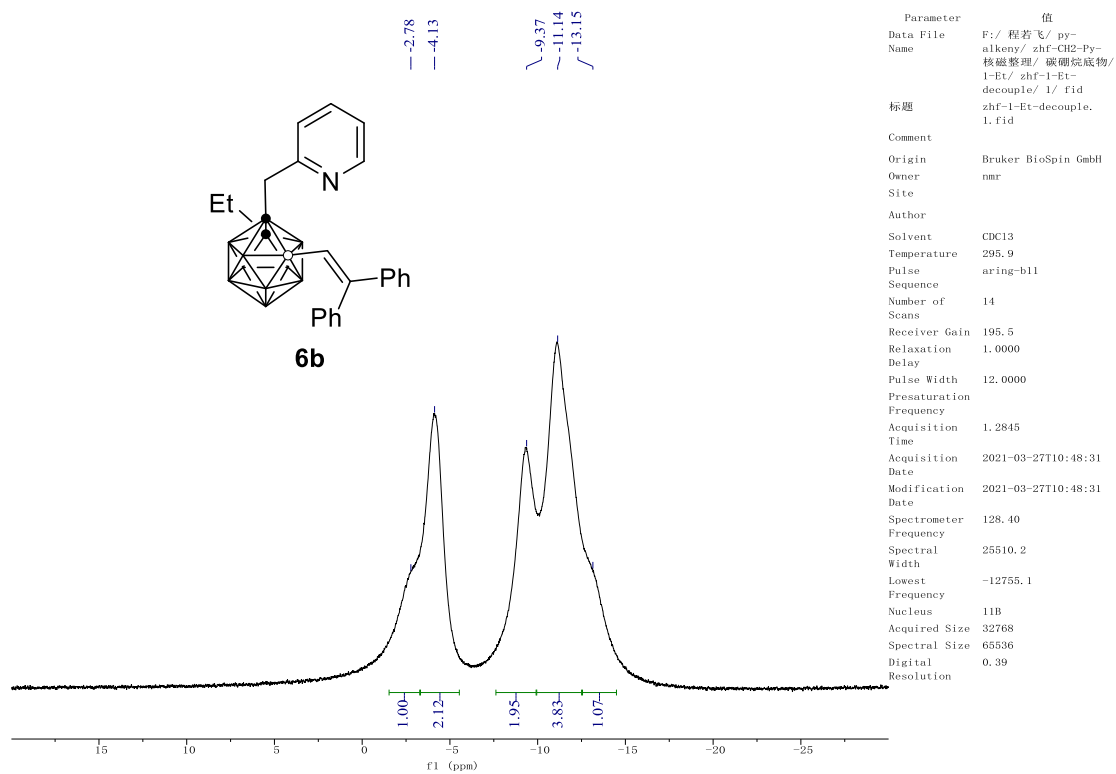


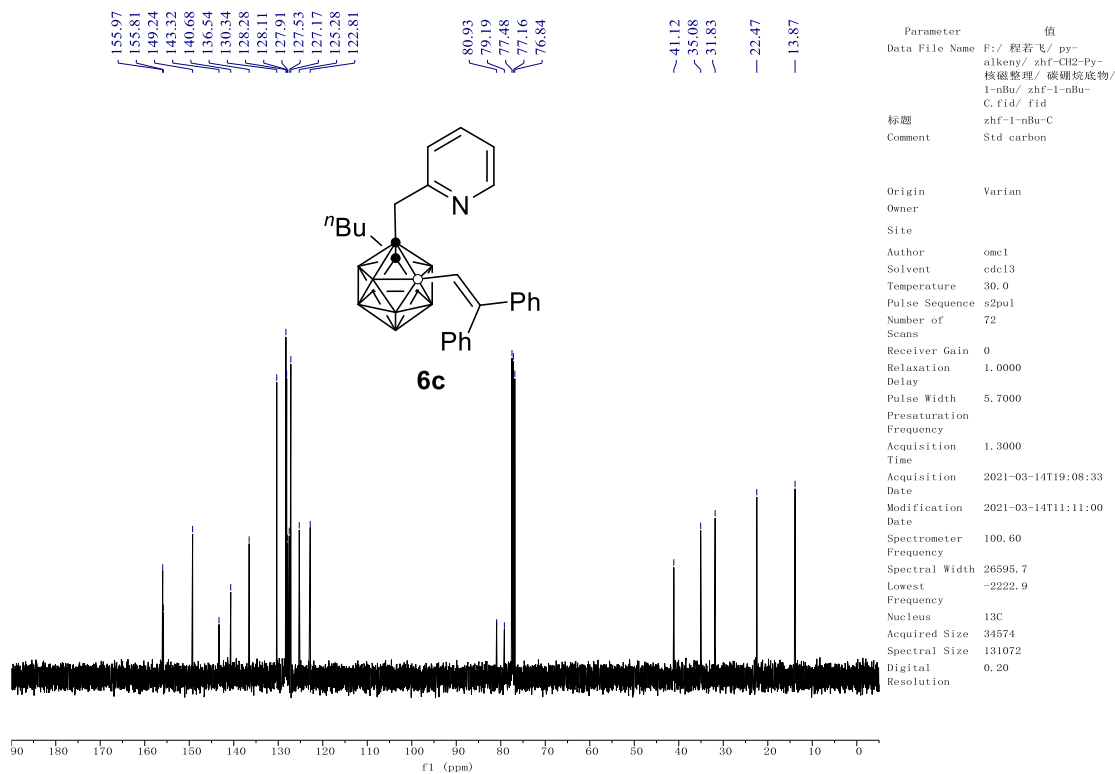
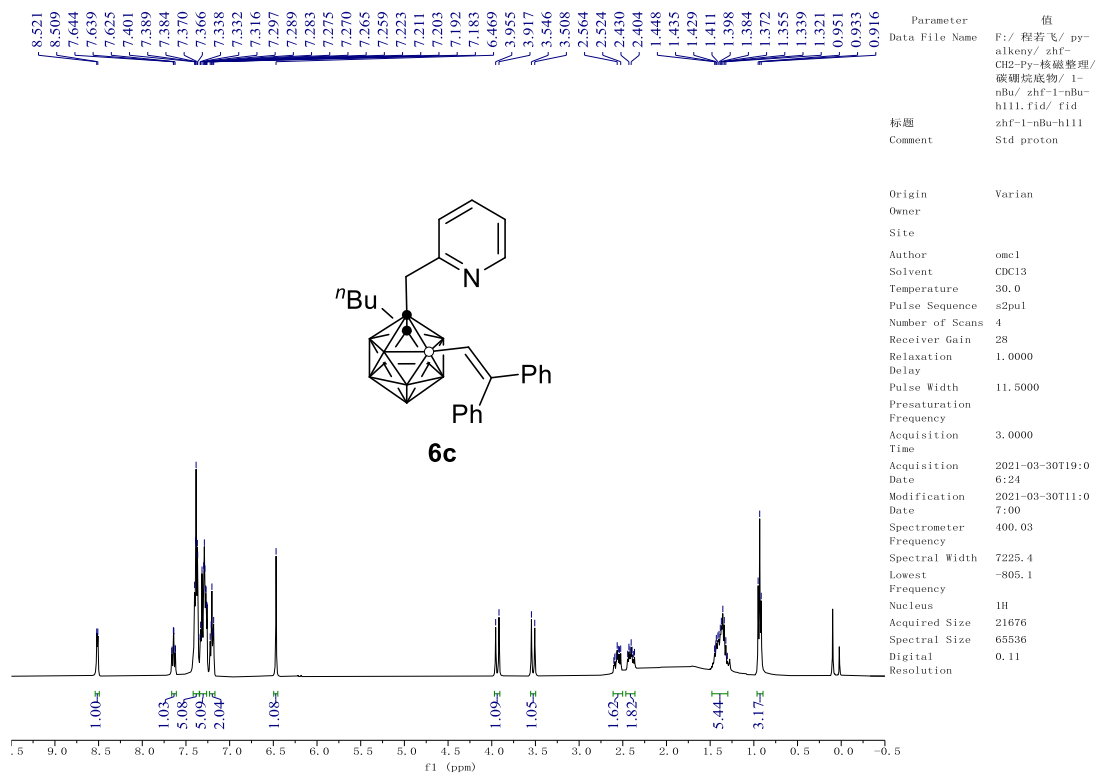


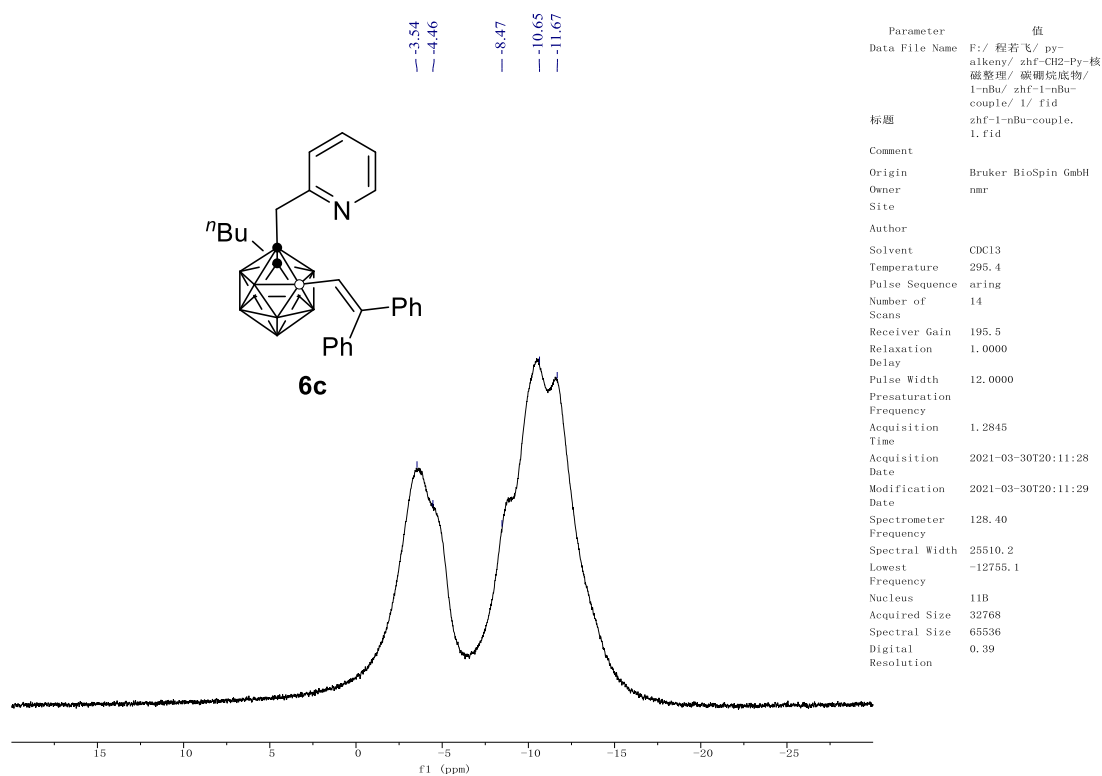
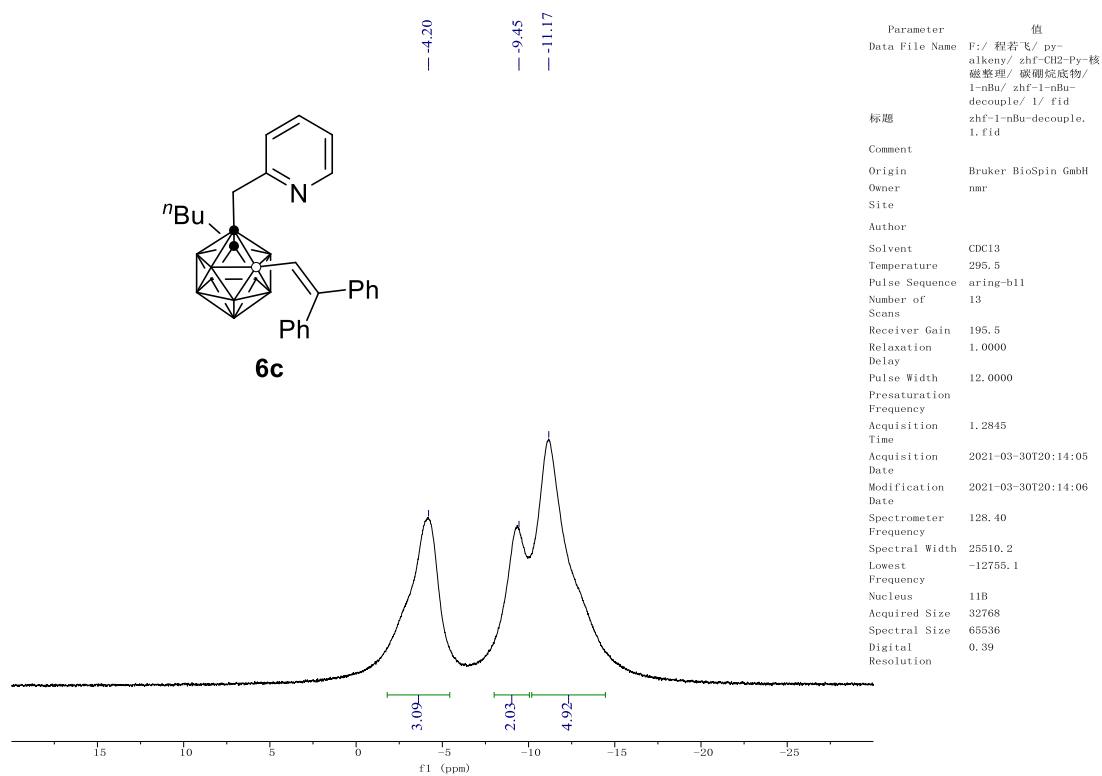
Parameter	值
Data File Name	F:/ 程若飞/ py-alkeny/ zhf-CH2-Py-核磁整理/ 碳硼烷底物/ 1-Et/ zhf-1-Et-H2.fid/ fid
标题	zhf-1-Et-H2
Comment	
Origin	Varian
Owner	
Site	
Author	
Solvent	CDCl3
Temperature	25.0
Pulse Sequence	s2pul
Number of Scans	4
Receiver Gain	48
Relaxation	1.0000
Delay	
Pulse Width	11.0000
Presaturation	
Frequency	
Acquisition	3.0000
Time	
Acquisition	2021-03-27T09:08:40
Date	
Modification	2021-03-27T01:09:00
Date	
Spectrometer	399.72
Frequency	
Spectral Width	7183.9
Lowest	-793.5
Frequency	
Nucleus	1H
Acquired Size	21552
Spectral Size	65536
Digital	0.11
Resolution	

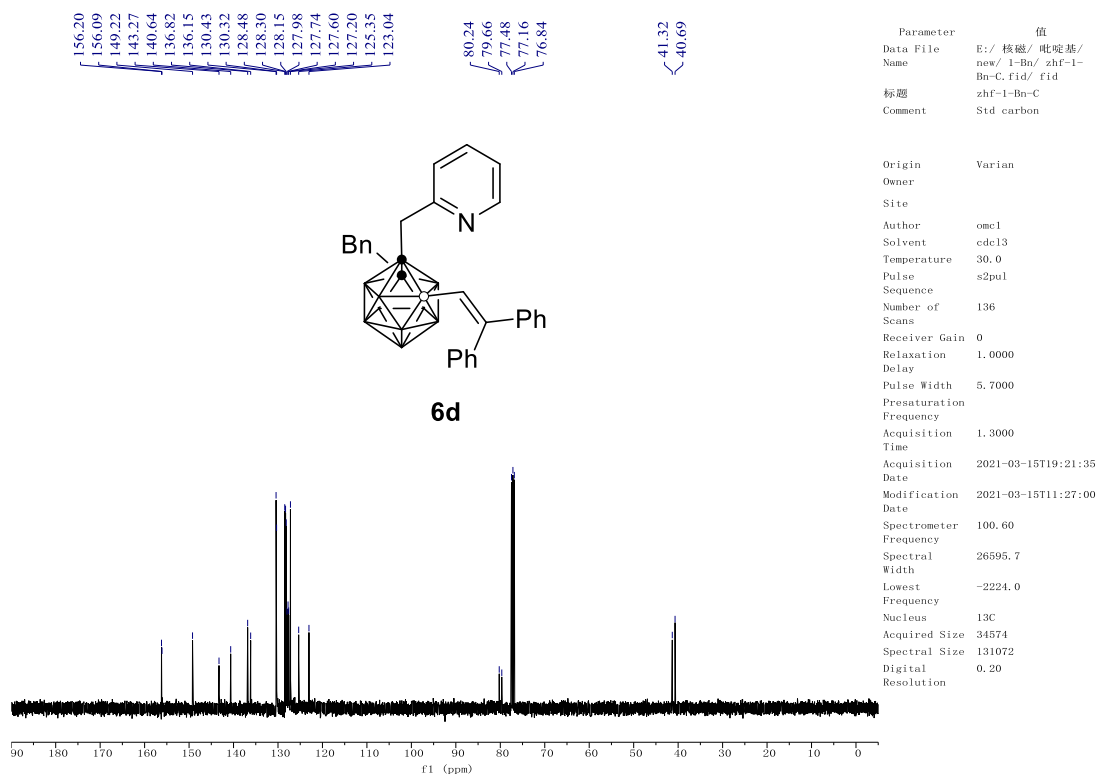
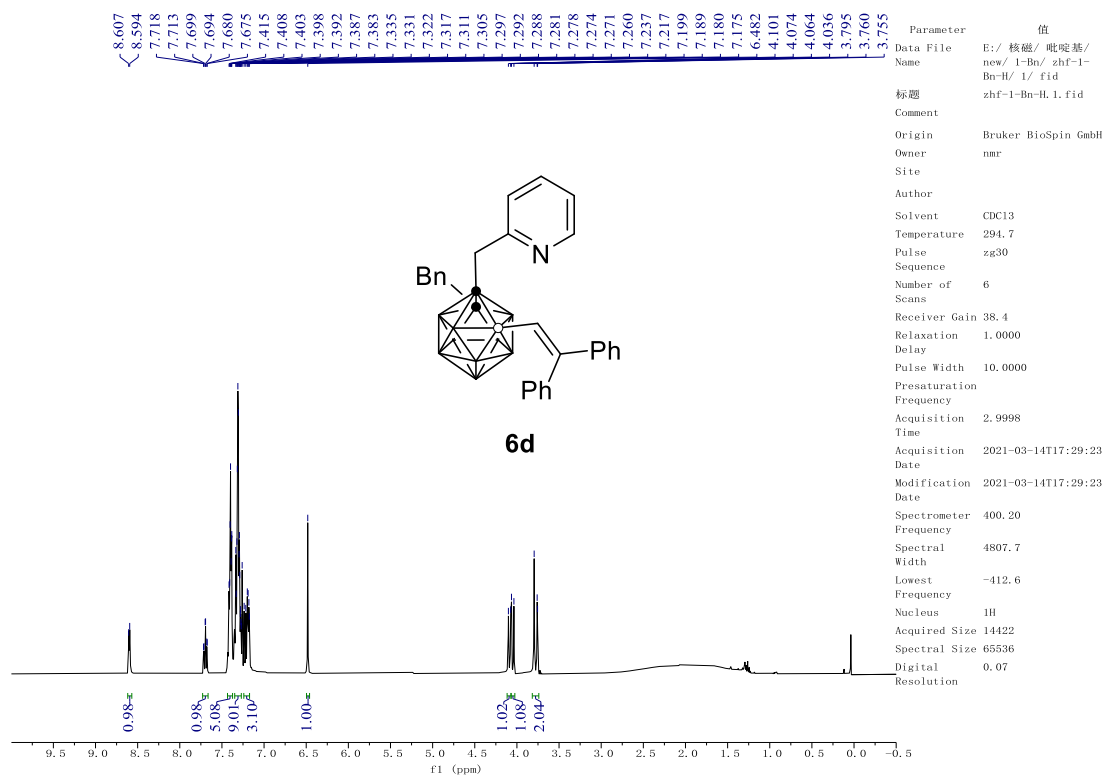


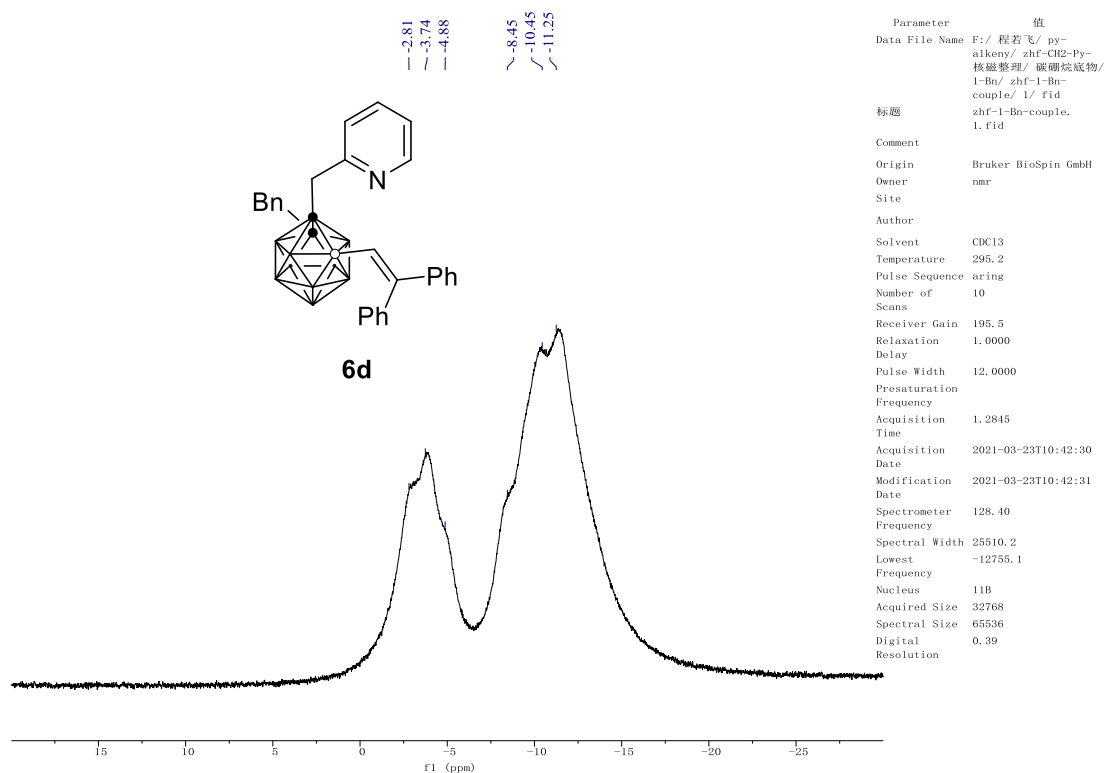
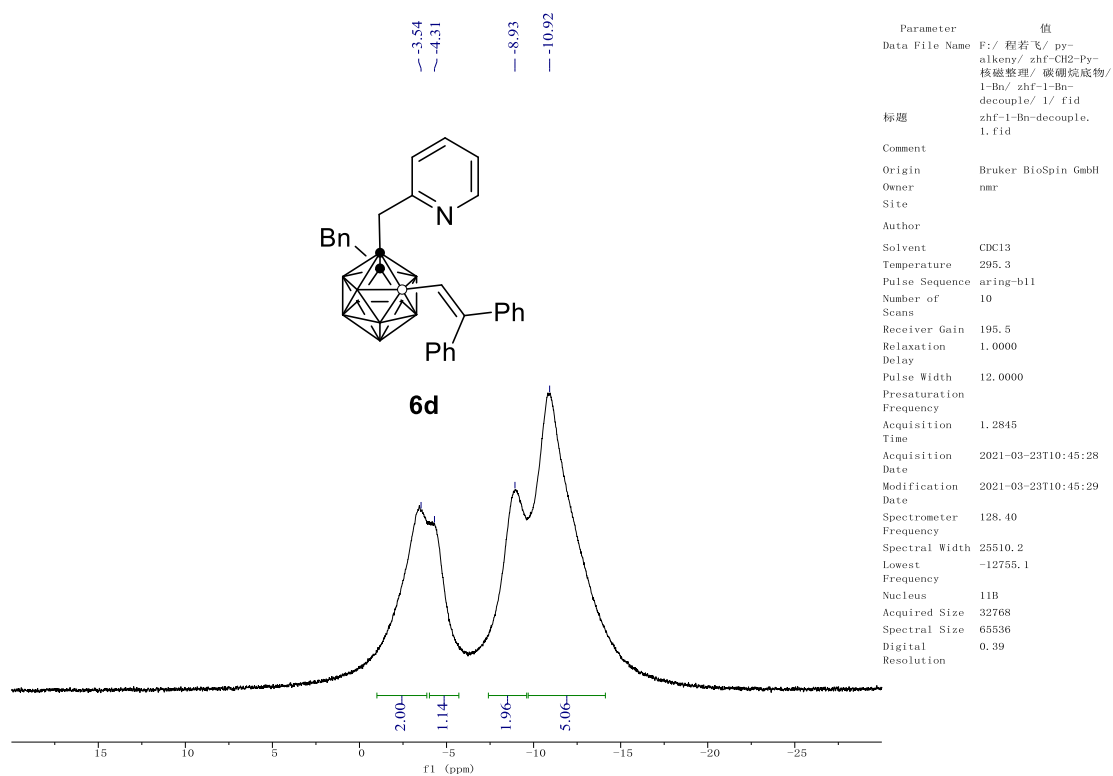
Parameter	值
Data File Name	F:/ 程若飞/ py-alkeny/ zhf-CH2-Py-核磁整理/ 碳硼烷底物/ 1-Et/ zhf-1-Et-C.1.fid
标题	zhf-1-Et-C.1.fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl3
Temperature	295.9
Pulse Sequence	zgpg30
Number of Scans	32
Receiver Gain	195.5
Relaxation	1.0000
Delay	
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition	0.9999
Time	
Acquisition	2021-03-27T10:42:40
Date	
Modification	2021-03-27T10:42:41
Date	
Spectrometer	100.64
Frequency	
Spectral Width	26041.7
Lowest	-1942.6
Frequency	
Nucleus	13C
Acquired Size	26040
Spectral Size	65536
Digital	0.40
Resolution	

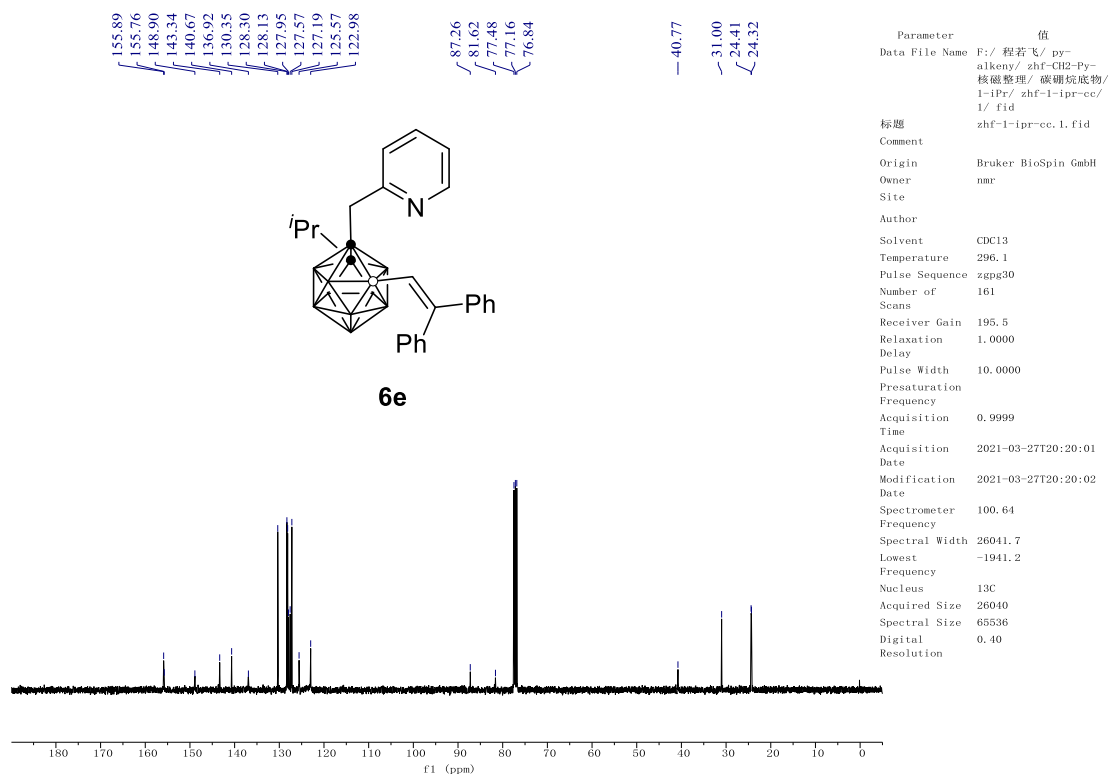
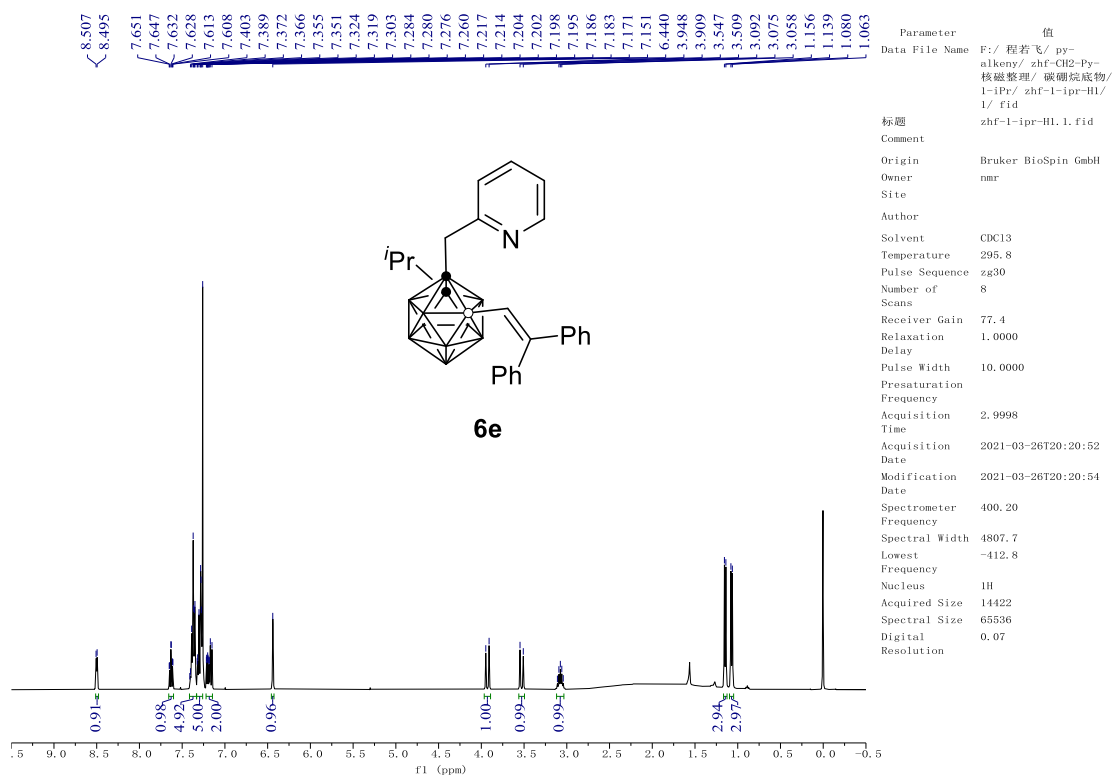


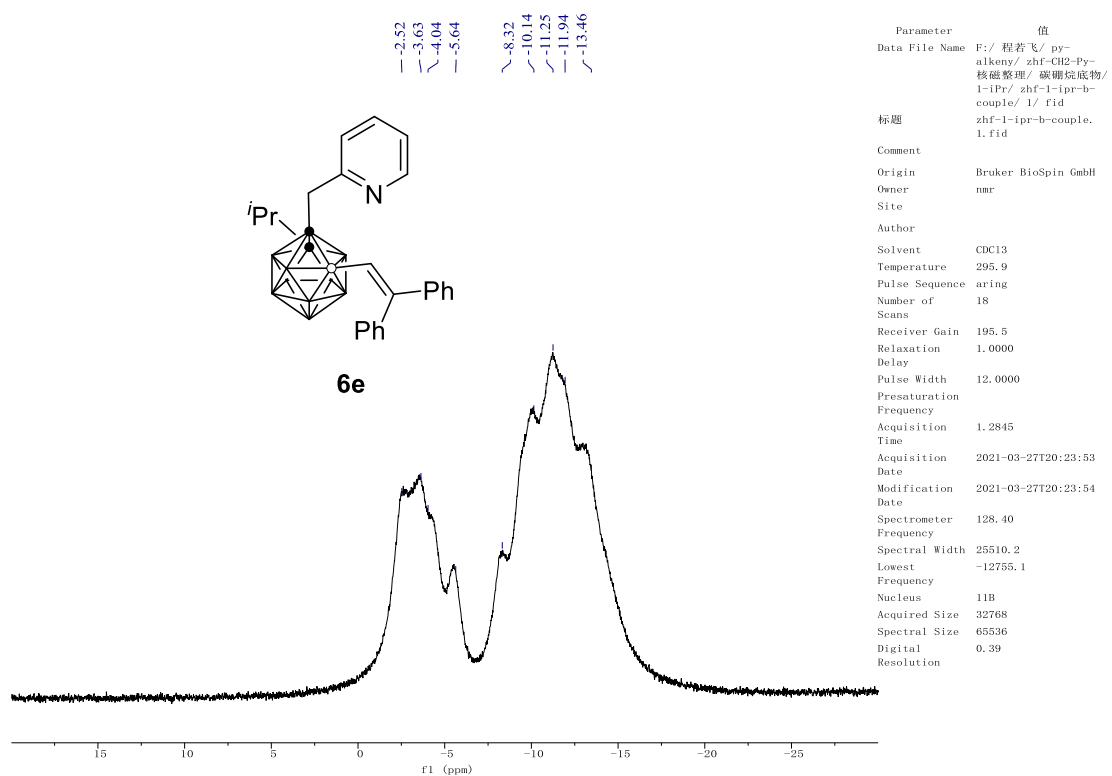
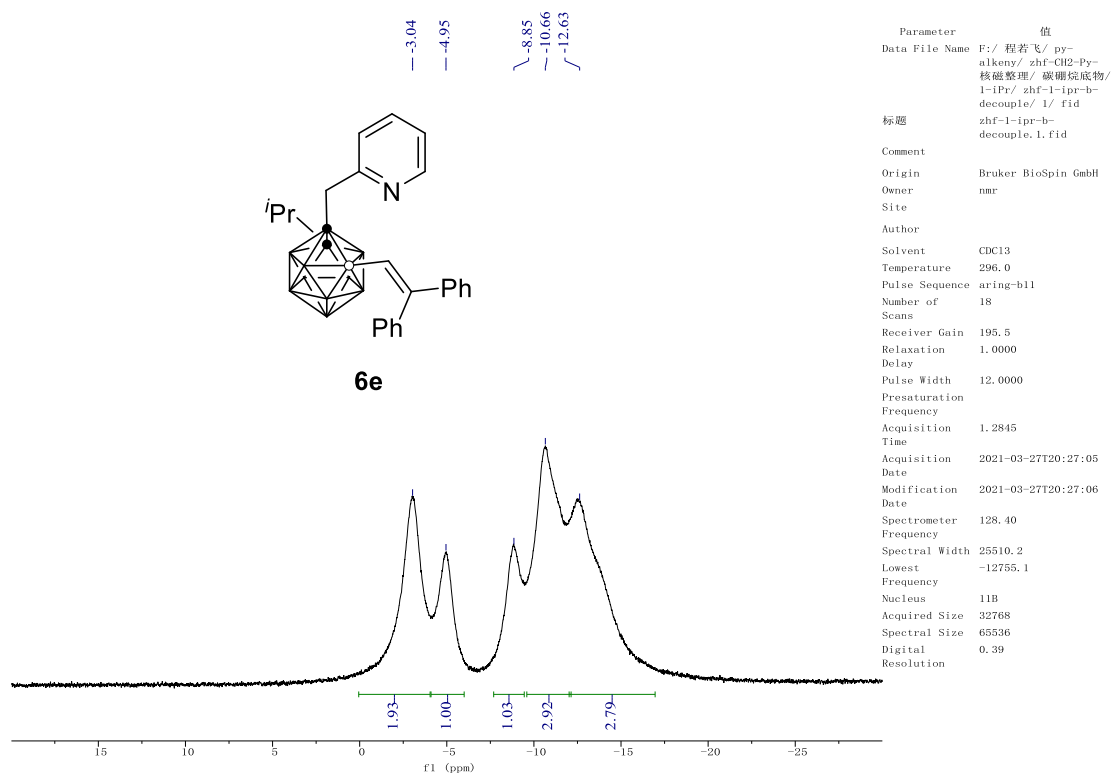


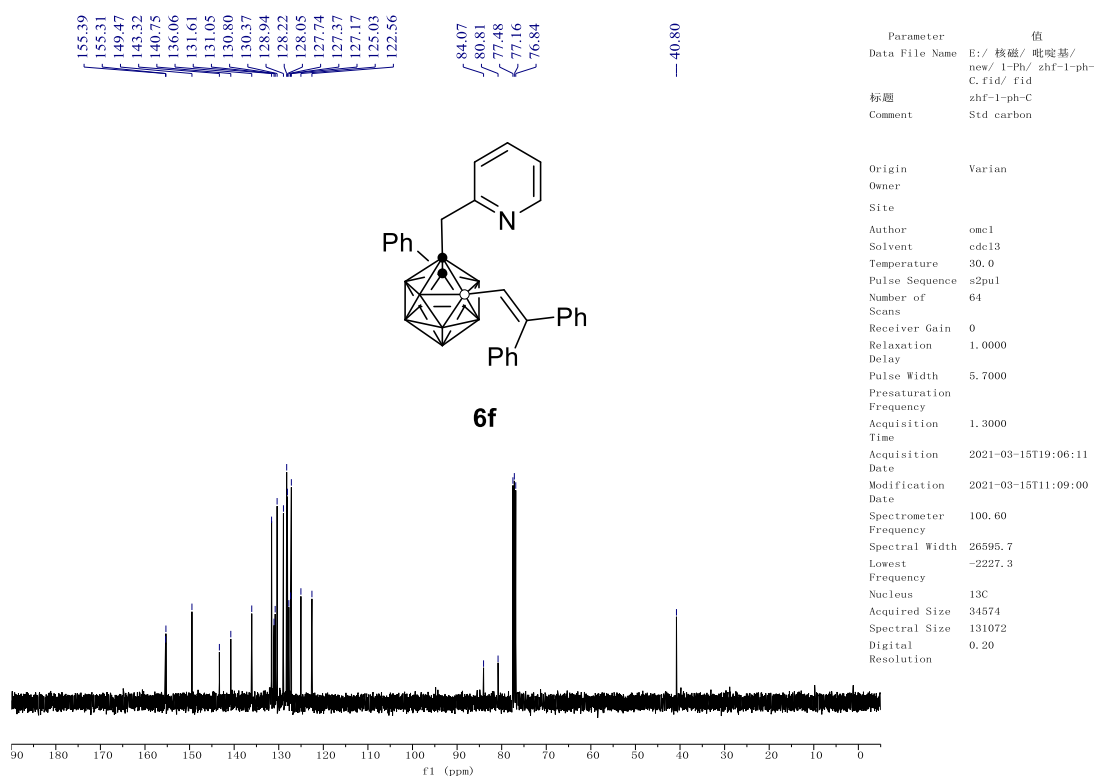
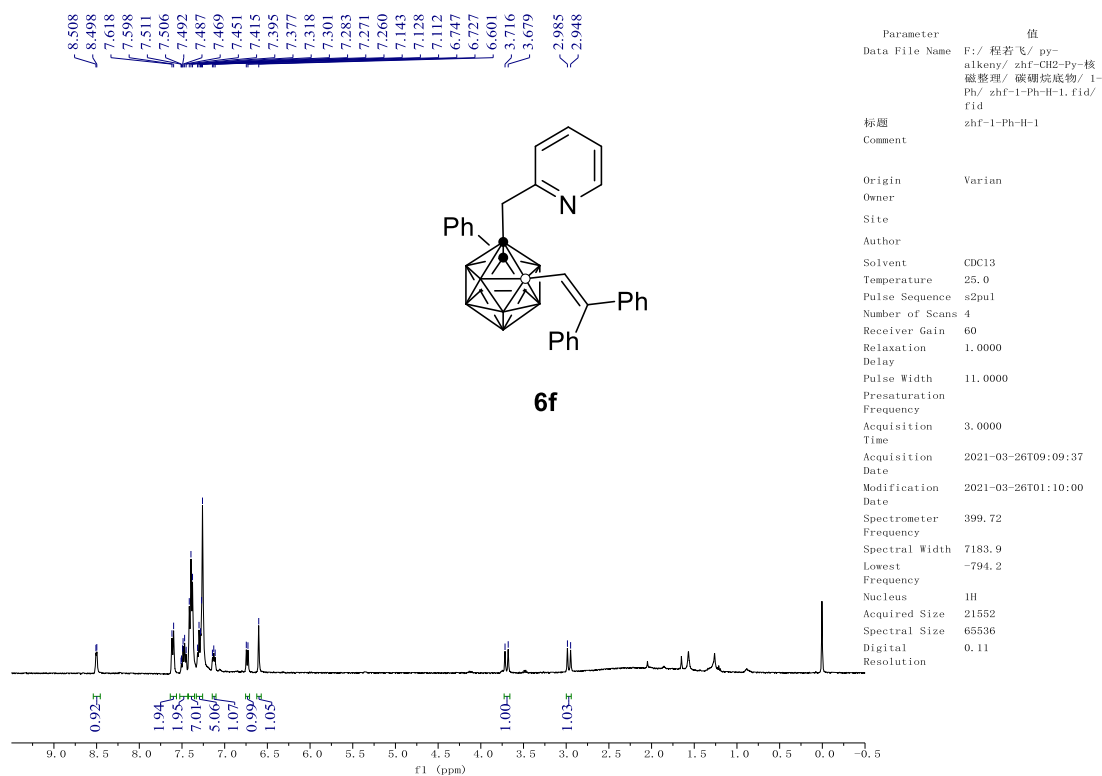


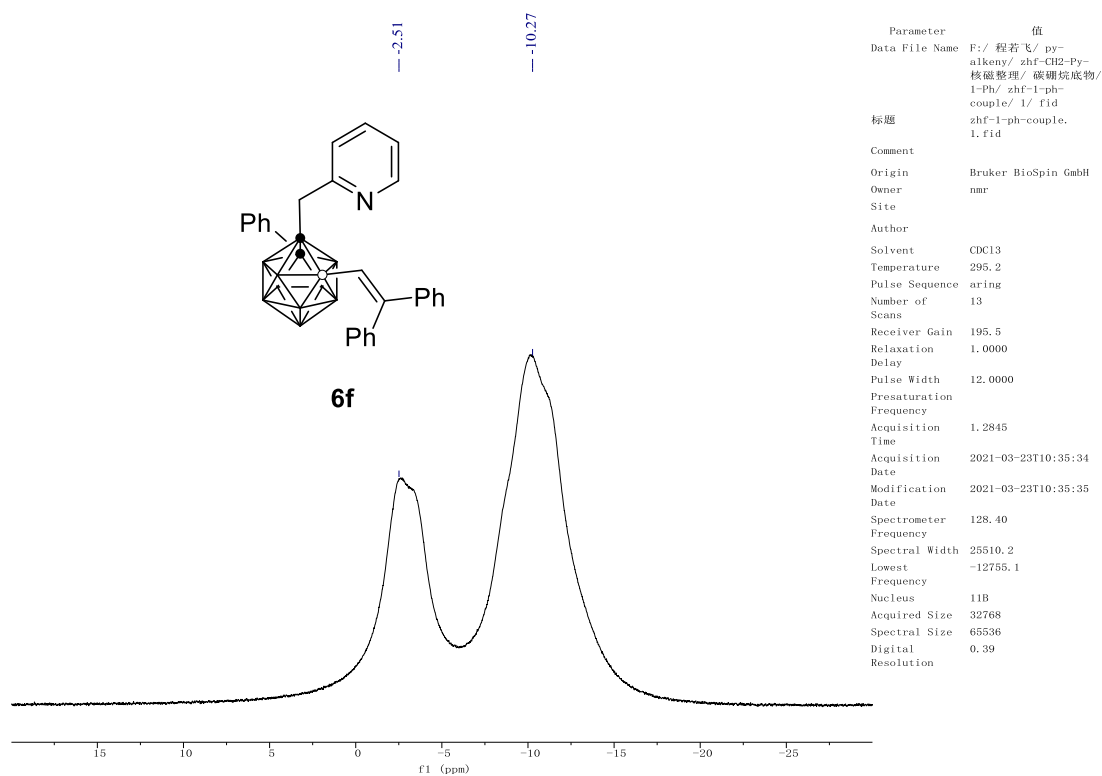
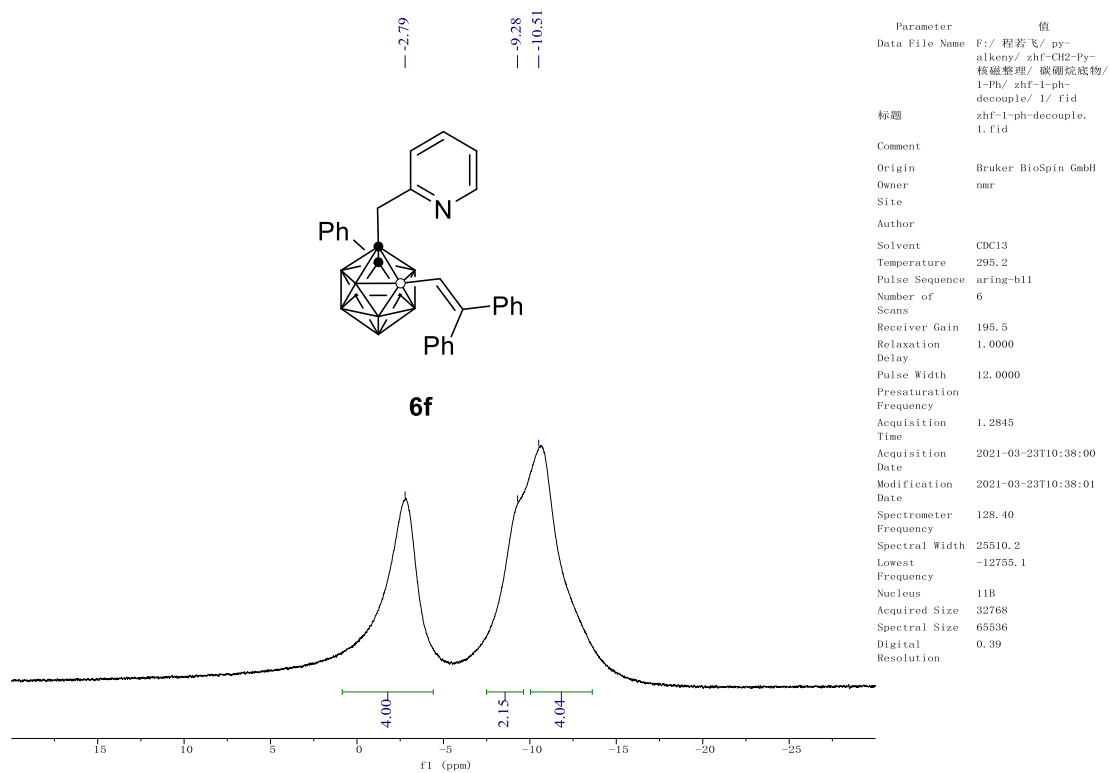


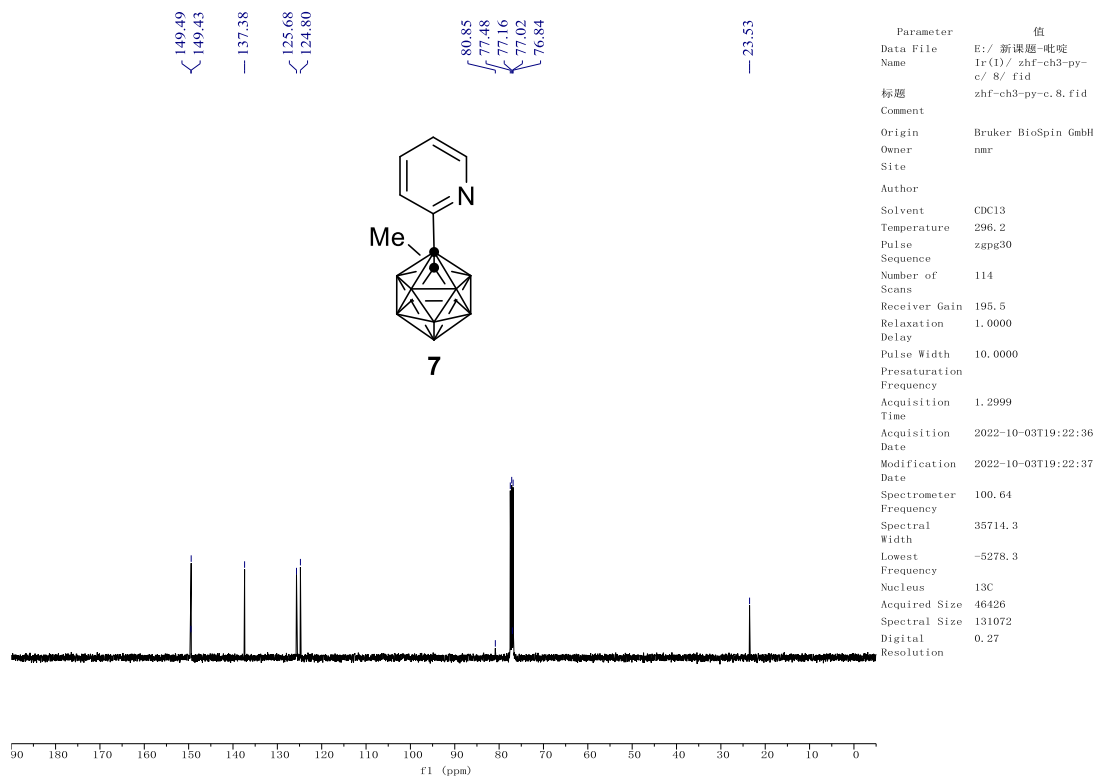
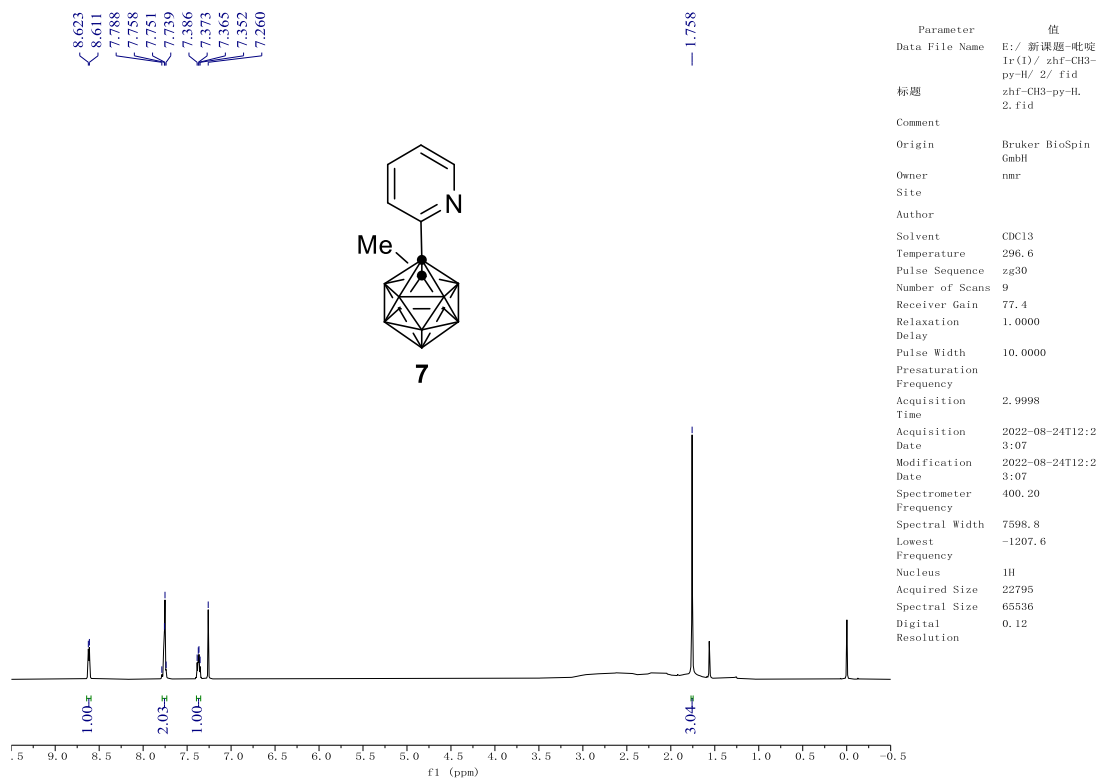


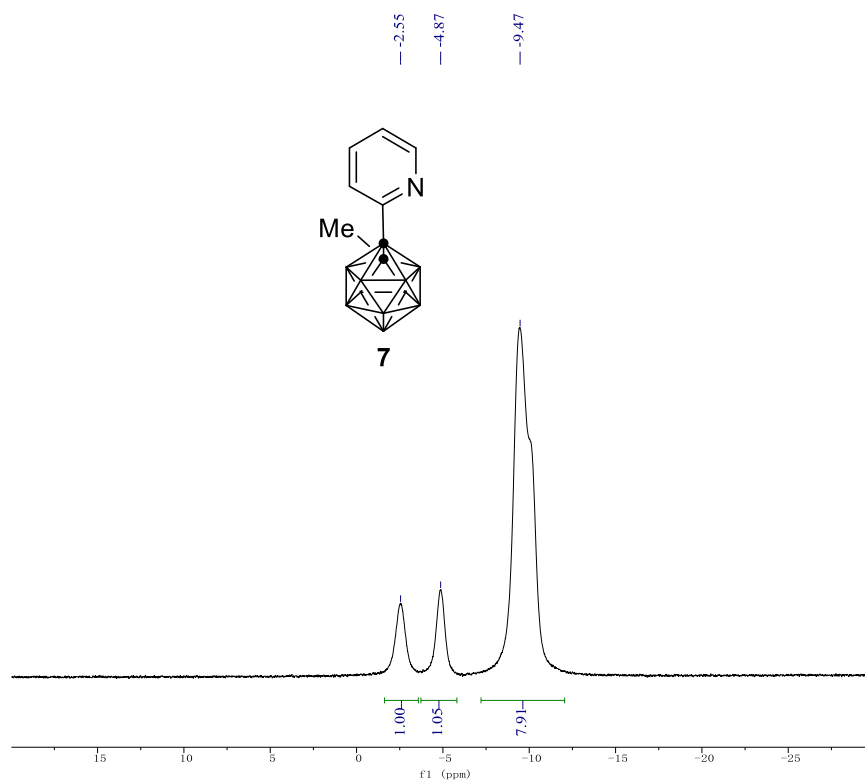




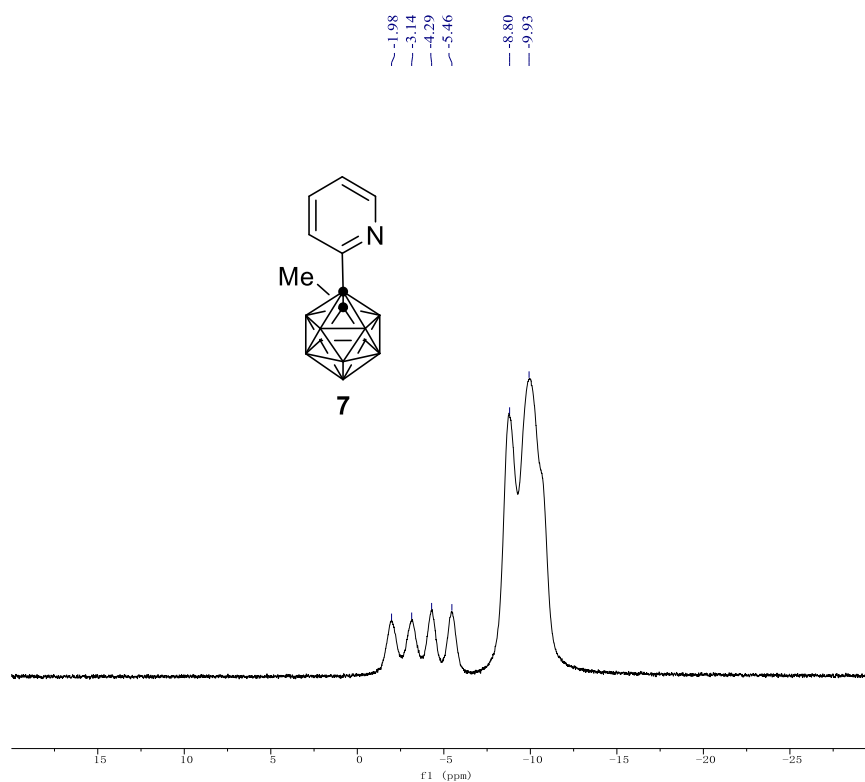




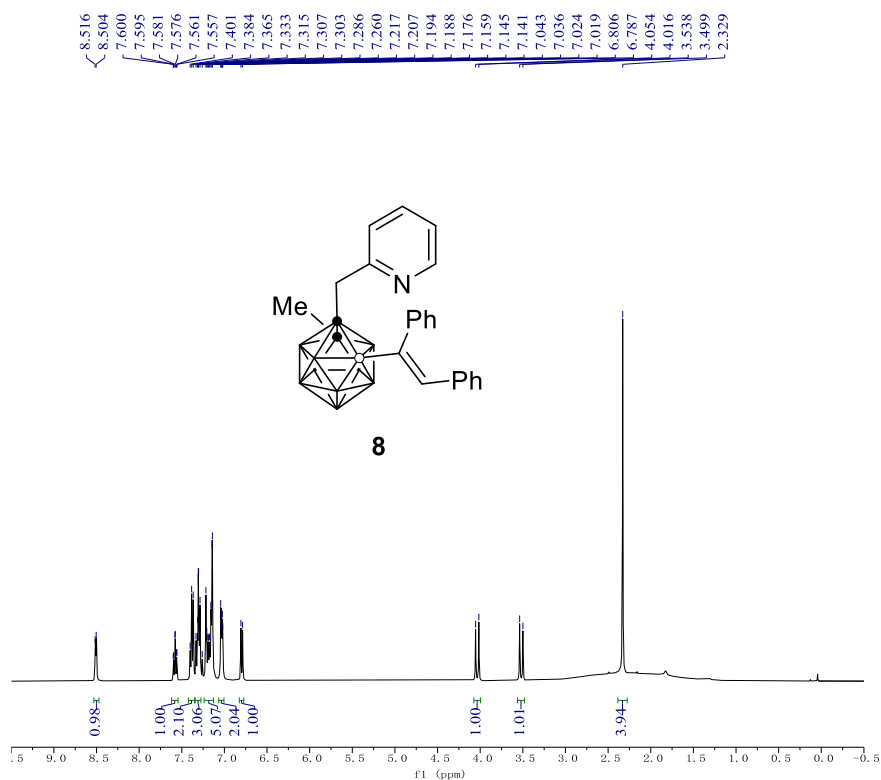




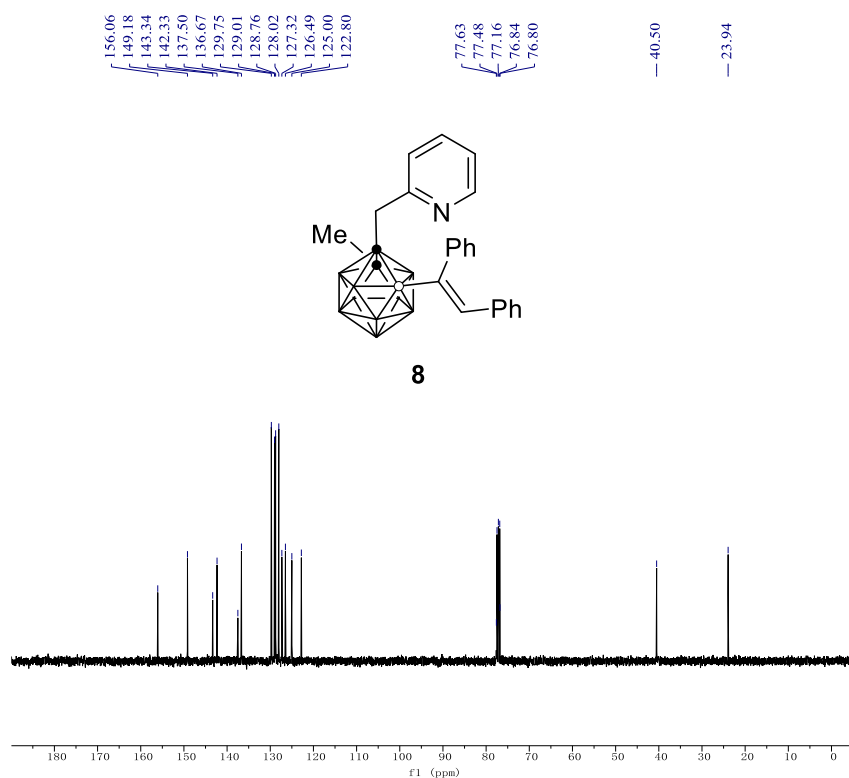
Parameter	值
Data File Name	E:/ 新课题-吡啶
	Ir(1)/ zhF-CH3-py-B/
	16/ fid
标题	zhf-CH3-py-B. 16. fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl3
Temperature	297.0
Pulse Sequence	aring-b11
Number of Scans	6
Receiver Gain	195.5
Relaxation	1.0000
Delay	
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-08-26T19:13:48
Modification Date	2022-08-26T19:13:49
Spectrometer	128.40
Frequency	
Spectral Width	25510.2
Lowest	-12755.1
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536
Digital	0.39
Resolution	



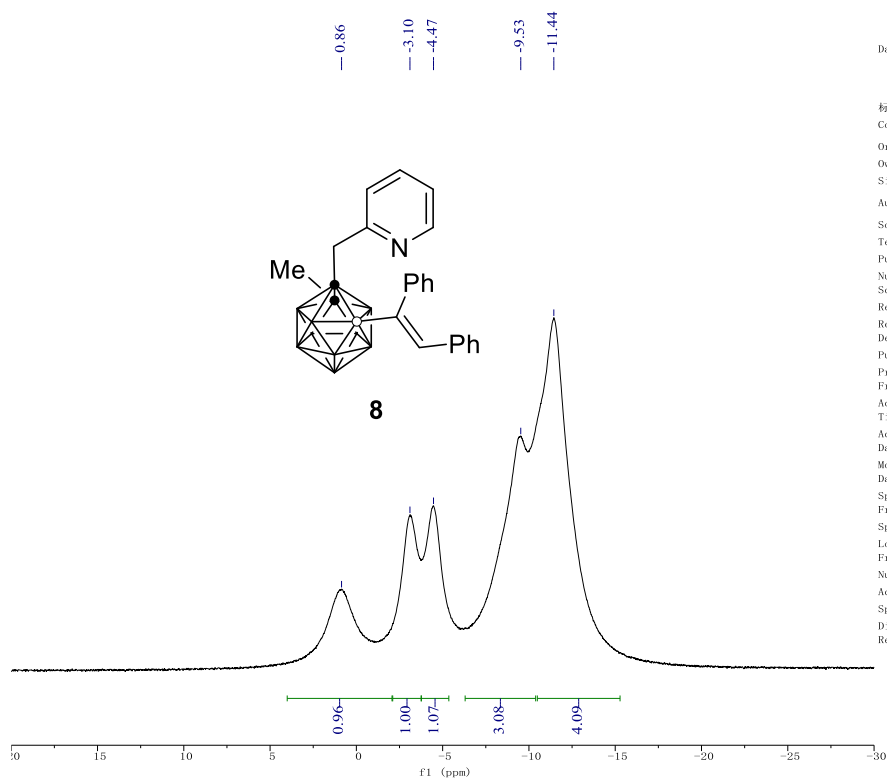
Parameter	值
Data File Name	E:/ 新课题-吡啶
	Ir(1)/ zhF-CH3-py-
	no-B/ 15/ fid
标题	zhf-CH3-py-no-B.
	15. fid
Comment	
Origin	Bruker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl3
Temperature	297.0
Pulse Sequence	aring
Number of Scans	5
Receiver Gain	195.5
Relaxation	1.0000
Delay	
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition Time	1.2845
Acquisition Date	2022-08-26T19:13:11
Modification Date	2022-08-26T19:13:12
Spectrometer	128.40
Frequency	
Spectral Width	25510.2
Lowest	-12755.1
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536
Digital	0.39
Resolution	



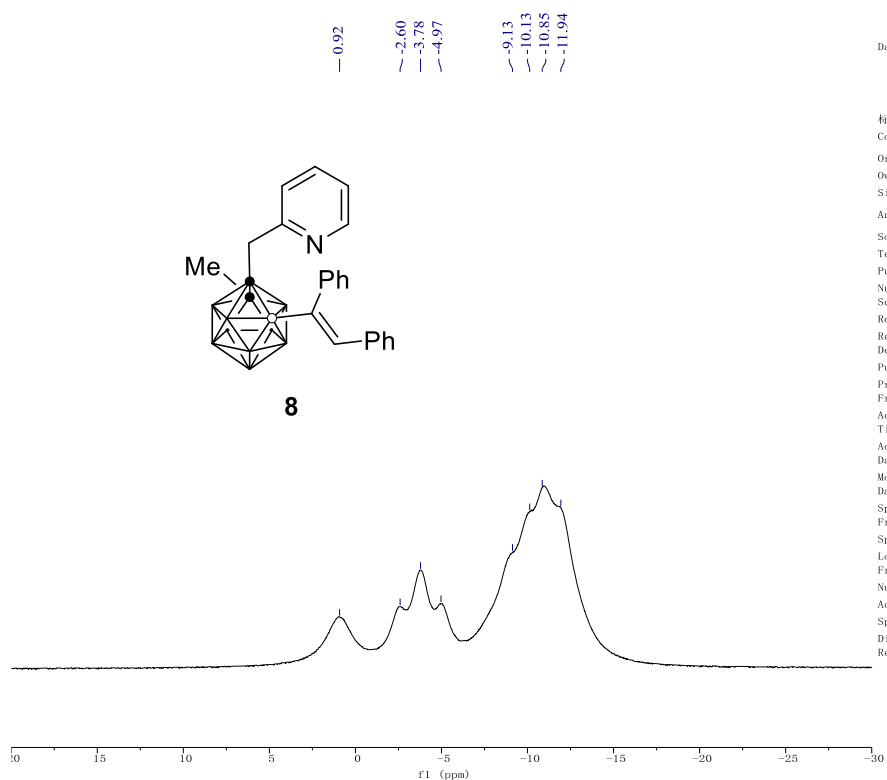
Parameter	值
Data File Name	F:/ zhf-芳基迁移-3,6位 烯基化反应-Work-1/ 核磁数据/ 8038/ zhf-8038-H/ 1/ fid
标题	zhf-8038-H, 1. fid
Comment	
Origin	Broker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl3
Temperature	296.3
Pulse Sequence	zg30
Number of Scans	1
Receiver Gain	27.2
Relaxation Delay	1.0000
Pulse Width	10.0000
Presaturation Frequency	
Acquisition Time	2.9998
Acquisition Date	2022-07-10T21:17:43
Modification Date	2022-07-10T21:17:43
Spectrometer Frequency	400.20
Spectral Width	4807.7
Lowest Frequency	-412.3
Nucleus	¹ H
Acquired Size	14422
Spectral Size	65536
Digital Resolution	0.07



Parameter	值
Data File Name	F:/ zhf-芳基迁移-3,6位 烯基化反应-Work-1/ 核磁数据/ 8038/ zhf-8038-C/ 8/ fid
标题	zhf-8038-C, 8. fid
Comment	
Origin	Broker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl3
Temperature	296.6
Pulse Sequence	zgpg30
Number of Scans	17
Receiver Gain	195.5
Relaxation Delay	1.0000
Pulse Width	10.0000
Presaturation Frequency	
Acquisition Time	1.2999
Acquisition Date	2022-07-11T19:11:28
Modification Date	2022-07-11T19:11:29
Spectrometer Frequency	100.64
Spectral Width	35714.3
Lowest Frequency	-5274.3
Nucleus	¹³ C
Acquired Size	46426
Spectral Size	131072
Digital Resolution	0.27



Parameter	值
Data File Name	F:/ zhf-芳基迁移-3,6
	位烯基化反应-Work-1/
	核磁数据/ 8038/
标题	zhf-8038-B. 9. fid
Comment	
Origin	Brucker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl ₃
Temperature	296.5
Pulse Sequence	aring-bll
Number of	6
Scans	
Receiver Gain	195.5
Relaxation	1.0000
Delay	
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition	1.2845
Time	
Acquisition	2022-07-11T19:13:02
Date	
Modification	2022-07-11T19:13:03
Date	
Spectrometer	128.40
Frequency	
Spectral Width	25510.2
Lowest	-12755.1
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536
Digital	0.39
Resolution	



Parameter	值
Data File Name	F:/ zhf-芳基迁移-3,6
	位烯基化反应-Work-1/
	核磁数据/ 8038/
	zhf-8038-no-B. 10/
	fid
标题	zhf-8038-no-B. 10. fid
Comment	
Origin	Brucker BioSpin GmbH
Owner	nmr
Site	
Author	
Solvent	CDCl ₃
Temperature	296.5
Pulse Sequence	aring
Number of	6
Scans	
Receiver Gain	195.5
Relaxation	1.0000
Delay	
Pulse Width	10.0000
Presaturation	
Frequency	
Acquisition	1.2845
Time	
Acquisition	2022-07-11T19:13:41
Date	
Modification	2022-07-11T19:13:42
Date	
Spectrometer	128.40
Frequency	
Spectral Width	25510.2
Lowest	-12755.1
Frequency	
Nucleus	11B
Acquired Size	32768
Spectral Size	65536
Digital	0.39
Resolution	