

Supporting Information

Direct Synthesis of Dialkylaryl-vinylsilane Derivatives: Metathesis of Dialkylaryl-iso-propenylsilane and Its Application to Tetracyclic Silacycle Dye Synthesis

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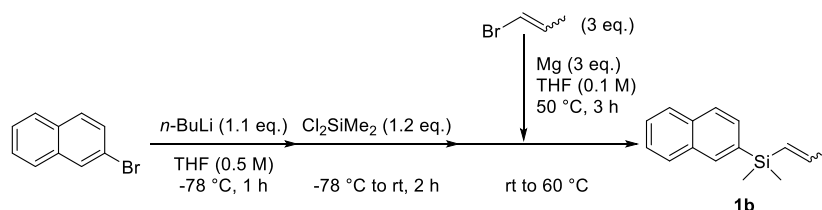
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General

¹H-NMR spectra were recorded in CDCl₃ at 25 °C unless otherwise noted, at 300, 400 or 500 MHz, with TMS as an internal standard. ¹³C-NMR spectra were recorded in CDCl₃ at 25 °C unless otherwise noted, at 300, 400 or 500MHz with TMS or CDCl₃ as an internal standard. ¹⁹F NMR spectra were recorded in CDCl₃ at 25 °C unless otherwise noted, at 470 MHz, with C₆F₆ as an internal standard

1. Synthesis of Cross metathesis substrate **1**

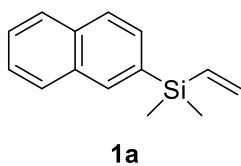


To a solution of 2-bromonaphthalene (5.0 mmol, 1.035 g) in THF (0.5 M) was added dropwise 2.76 M *n*-BuLi (1.1 eq.) in *n*-hexane at -78 °C. After the mixture was stirred at -78 °C for 1 h, Me₂SiCl₂ (1.2 eq.) was added dropwise to the mixture. The reaction mixture was warmed to room temperature over 2 h. We call this reaction mixture, solution α .

On the other hand, to a solution of magnesium (3.0 eq.) in THF (0.2 M) was added dropwise a solution of 1-bromopropene in THF (0.2 M) at 50 °C for 3 h. We call this reaction mixture, solution β .

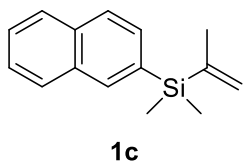
To a solution α was dropwisely added solution β at room temperature and heated to 50 °C for 3 h. After that, the reaction was quenched by the addition of 1M HCl aq. to the reaction mixture. The organic compound was extracted with CHCl₃. The organic layer was dried over Na₂SO₄, and the solvent was evaporated. The residue was subjected to column chromatography (only *n*-hexane) on silica gel 60N to give compound **1b** (a colorless oil, E/Z = 7/2: 0.550 mmol, 124.6 mg, 11%).

¹H-NMR (CDCl₃, 400 MHz) δ : 8.02 (1H, s), 7.86-7.81 (3H, m), 7.64-7.62 (1H, m), 7.49-7.46 (2H, m), 6.58 (1H, ddd, *J* = 14.0, 6.8, 1.8 Hz), 5.73 (1H, d, *J* = 14.0 Hz), 1.73 (3H, dd, *J* = 6.8, 1.8 Hz), 0.43 (6H, s). ¹³C-NMR (CDCl₃, 125 MHz) δ : 144.45, 136.83, 134.49, 133.79, 133.07, 130.37, 129.34, 128.18, 127.83, 127.01, 126.36, 125.96, 22.89, -2.29. HRMS (MALDI-TOF) calcd for C₁₅H₁₈NaSi: 249.1070 ([M + Na]⁺), found 249.1074 ([M + Na]⁺).



1a (a yellow oil, quant, 1.06 g, 5.00 mmol) was prepared from 2-bromonaphthalene (1.04 mg, 0.500 mmol) by the same procedure with **1b** using dimethylvinylchlorosilane instead of dimethyldichlorosilane and Grignard reagent.

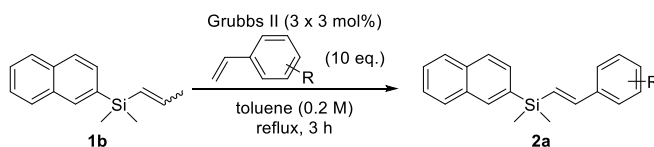
¹H-NMR (CDCl₃, 300 MHz) δ : 8.04 (1H, s), 7.89-7.82 (3H, m), 7.62 (1H, dd, *J* = 8.0, 1.1 Hz), 7.52-7.48 (2H, m), 6.39 (1H, dd, *J* = 20.2, 14.7 Hz), 6.12 (1H, dd, *J* = 14.7, 3.7 Hz), 5.83 (1H, dd, *J* = 20.2, 3.7 Hz), 0.46 (6H, s). ¹³C-NMR (75 MHz, CHCl₃) δ : 137.98, 135.90, 134.54, 133.76, 133.14, 132.97, 130.18, 128.12, 127.77, 127.04, 126.39, 125.97, -2.79. HRMS (ESI) calcd for C₁₄H₁₆Si: 213.1100 ([M+H]⁺), found 213.1092 ([M+H]⁺).



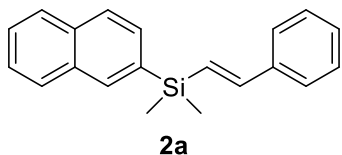
1c (a colorless oil, 43%, 0.486 g, 0.215 mmol) was prepared from 2-bromonaphthalene (1.04 mg, 0.500 mmol) by the same procedure with **1b** using 2-bromopropene instead of 1-bromopropene.

$^1\text{H-NMR}$ (CDCl_3 , 500 MHz) δ : 8.12 (1H, s), 7.94-7.91 (3H, m), 7.69 (1H, dd, $J = 8.0, 1.1$ Hz), 7.57-7.56 (2H, m), 5.83 (1H, s), 5.50 (1H, s), 0.55 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 146.10, 135.56, 134.62, 133.81, 133.02, 130.28, 128.14, 127.79, 127.03, 126.82, 126.38, 125.94, 22.70, -3.35 HRMS (ESI) calcd for $\text{C}_{15}\text{H}_{18}\text{NaSi}$: 249.1070 ($[\text{M}+\text{Na}]^+$) found 249.1066 ($[\text{M}+\text{Na}]^+$)

2. Cross metathesis of compound **1b** (general procedure A)

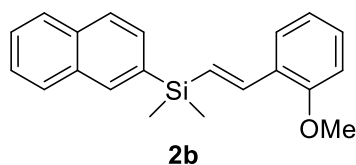


To a solution of **1b** (1.0 mmol) in toluene (0.2 M) was added Grubbs II (3 x 3 mol%, every hour) and the mixture was refluxed for 3 h. The solvent was evaporated and the residue was subjected to column chromatography on neutral flash silica gel 60N to give **2a**.



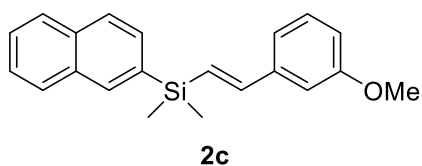
2a (a colorless oil, 85%, 24.5 mg, 0.850 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and styrene (104.2 mg, 1.00 mmol) by a general procedure A.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 8.07 (1H, s), 7.86-7.82 (3H, m), 7.65 (1H, dd, $J = 8.5, 1.2$ Hz), 7.50-7.46 (4H, m), 7.37-7.32 (2H, m), 7.29-7.26 (1H, m), 6.99 (1H, d, $J = 19.5$ Hz), 6.66 (1H, d, $J = 19.5$ Hz), 0.52 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 145.5, 138.2, 136.1, 134.6, 133.8, 133.0, 130.3, 128.6, 128.3, 128.1, 127.8, 127.1, 126.61, 126.58, 126.4, 126.00, -2.4. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{20}\text{Si}$: 311.1232 ($[\text{M}+\text{Na}]^+$) found 311.1221 ($[\text{M}+\text{Na}]^+$)



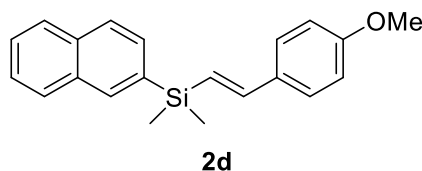
2d (a colorless oil, 97%, 30.8 mg, 0.967 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and 2-vinyanisole (134.2 mg, 1.00 mmol) by a general procedure A.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.91 (1H, s), 7.69-7.64 (3H, m), 7.49 (1H, dd, $J = 8.1, 1.1$ Hz), 7.42 (1H, dd, $J = 7.8, 1.8$ Hz), 7.32-7.29 (3H, m), 7.08-7.05 (1H, m), 6.79 (1H, d, $J = 7.8$ Hz), 6.70 (1H, dd, $J = 8.1, 0.9$ Hz), 6.47 (1H, d, $J = 19.2$ Hz), 3.66 (3H, s), 0.36 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 156.8, 139.9, 136.6, 134.7, 133.8, 133.1, 130.4, 129.4, 128.2, 127.8, 127.4, 127.4, 127.1, 126.5, 126.4, 126.0, 120.7, 111.1, 55.6, -2.2 HRMS (MALDI-TOF) calcd for $\text{C}_{21}\text{H}_{22}\text{ONaSi}$: 341.1332 ($[\text{M}+\text{Na}]^+$), found 341.1329 ($[\text{M}+\text{Na}]^+$).



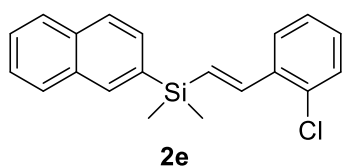
2c (a colorless oil, 84%, 26.8 mg, 0.842 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and 3-vinyanisole (134.2 mg, 1.00 mmol) by a general procedure A.

$^1\text{H-NMR}$ (CDCl_3 , 400MHz) δ : 8.08 (1H, s), 7.88-7.84 (3H, m), 7.66 (1H, dd, $J = 8.2, 0.9$ Hz), 7.52-7.49 (2H, m), 7.27 (1H, dd, $J = 8.2, 8.2$ Hz), 7.08 (1H, d, $J = 8.0$ Hz), 7.02 (1H, dd, $J = 2.1, 2.1$ Hz), 6.97 (1H, d, $J = 19.0$ Hz), 6.84 (1H, dt, $J = 8.0, 1.4$ Hz), 6.66 (1H, d, $J = 19.0$ Hz), 3.83 (3H, s), 0.54 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 156.0, 145.4, 139.7, 136.0, 134.7, 133.8, 133.0, 130.3, 129.6, 128.2, 127.8, 127.5, 127.1, 126.5, 126.0, 119.4, 114.3, 111.4, 55.3, -2.4 HRMS (MALDI-TOF) calcd for $\text{C}_{21}\text{H}_{22}\text{OSi}$: 318.1434 ($[\text{M}+\text{H}]^+$), found 318.1435 ($[\text{M}+\text{H}]^+$).



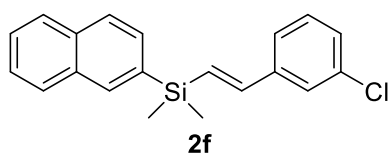
2d (a colorless oil, 90%, 28.7 mg, 0.901 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-vinyanisole (134.2 mg, 1.00 mmol) by a general procedure A.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 8.09 (1H, s), 7.89-7.84 (3H, m), 7.67 (1H, dd, $J = 8.2, 0.9$ Hz), 7.52-7.49 (2H, m), 7.45-7.42 (2H, m), 6.96 (1H, d, $J = 19.2$ Hz), 6.91-6.87 (2H, m), 6.51 (1H, d, $J = 19.2$ Hz), 3.83 (3H, s), 0.53 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 159.8, 145.0, 136.4, 134.6, 133.8, 133.0, 131.2, 130.4, 128.2, 127.9, 127.8, 127.1, 126.4, 126.0, 124.2, 114.0, 55.4, -2.2. HRMS (MALDI-TOF) calcd for $\text{C}_{21}\text{H}_{22}\text{OSi}$: 318.1434 ($[\text{M}+\text{H}]^+$), found 318.1432 ($[\text{M}+\text{H}]^+$).



2e (a colorless oil, 52%, 16.9 mg, 0.0523 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-chlorostyrene (138.6 mg, 1.00 mmol) by a general procedure A.

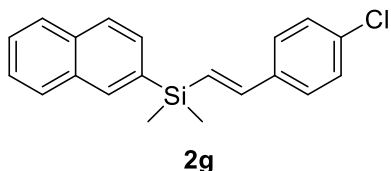
$^1\text{H-NMR}$ (CDCl_3 , 500 MHz) δ : 8.09 (1H, s), 7.88-7.83 (3H, m), 7.68-7.65 (2H, m), 7.51-7.49 (2H, m), 7.45 (1H, d, $J = 18.9$ Hz), 7.37 (1H, d, $J = 7.8$ Hz), 7.25 (1H, dd, $J = 7.8, 7.8$ Hz), 7.22-7.19 (1H, m), 6.67 (1H, d, $J = 18.9$ Hz), 0.56 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 141.3, 136.3, 135.8, 134.6, 133.8, 133.3, 133.0, 130.8, 130.2, 129.8, 129.1, 128.2, 127.8, 127.2, 126.9, 126.5, 126.3, 126.0, -2.4 HRMS (MALDI-TOF) calcd for $\text{C}_{20}\text{H}_{19}\text{NaSiCl}$: 345.0837 ($[\text{M}+\text{Na}]^+$), found 345.0823 ($[\text{M}+\text{Na}]^+$).



2f (a colorless oil, 63%, 20.2 mg, 0.0626 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-chlorostyrene (138.6 mg, 1.00 mmol) by a general procedure A.

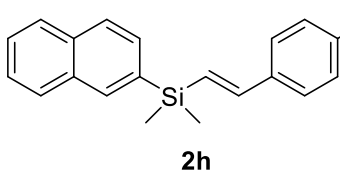
$^1\text{H-NMR}$ (CDCl_3 , 500 MHz) δ : 8.06 (1H, s), 7.85-7.84 (3H, m), 7.64-7.62 (1H, m), 7.51-7.49 (2H, m), 7.47-7.45 (1H, m), 7.32-7.31 (1H, m), 7.28-7.22 (2H, m), 6.90

(1H, d, $J = 19.5$ Hz), 6.68 (1H, d, $J = 18.9$ Hz), 0.53 (6H, s). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 144.0, 140.1, 135.6, 134.7, 133.8, 133.0, 130.2, 129.8, 129.2, 128.15, 128.13, 127.8, 127.2, 126.5, 126.1, 124.9, -2.5. HRMS (MALDI-TOF) calcd for $\text{C}_{20}\text{H}_{19}\text{SiCl}$: 322.0939 ($[\text{M}+\text{H}]^+$), found 322.0934 ($[\text{M}+\text{H}]^+$).



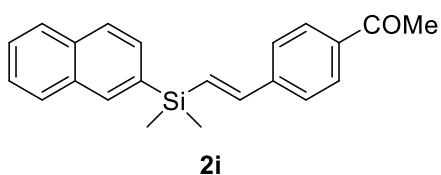
2g (a colorless oil, 69%, 22.3 mg, 0.0691 mmol) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-chlorostyrene (138.6 mg, 1.00 mmol) by a general procedure A.

^1H -NMR (CDCl_3 , 400 MHz) δ : 8.08 (1H, s), 7.89-7.84 (3H, m), 7.65 (1H, d, $J = 7.9$ Hz), 7.52-7.50 (2H, m), 7.40 (2H, d, $J = 8.6$ Hz), 7.31 (2H, d, $J = 8.6$ Hz), 6.93 (1H, d, $J = 19.2$ Hz), 6.64 (1H, d, $J = 19.2$ Hz), 0.54 (6H, s). ^{13}C -NMR (CDCl_3 , 100 MHz) δ : 144.1, 136.7, 135.7, 134.6, 133.9, 133.8, 133.0, 130.2, 128.8, 128.14, 128.09, 127.80, 127.76, 127.2, 126.5, 126.1, -2.4. HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{19}\text{ClSi}$: 345.0842 ($[\text{M}+\text{Na}]^+$), found 345.0833 ($[\text{M}+\text{Na}]^+$).



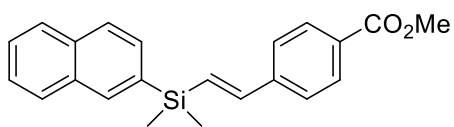
2h (a colorless oil, 88%, 30.5 mg) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-acetoxystyrene (162.2 mg, 1.00 mmol) by a general procedure A.

^1H -NMR (CDCl_3 , 400 MHz) δ : 8.06 (1H, s), 7.85-7.84 (3H, m), 7.64 (1H, d, $J = 8.0$ Hz), 7.49-7.48 (4H, m), 7.07 (2H, d, $J = 8.7$ Hz), 6.96 (1H, d, $J = 19.0$ Hz), 6.61 (1H, d, $J = 19.0$ Hz), 2.30 (3H, s), 0.52 (6H, s). ^{13}C -NMR (CDCl_3 , 100 MHz) δ : 169.51, 150.55, 144.42, 136.04, 135.91, 134.63, 133.79, 132.99, 130.25, 128.14, 127.78, 127.58, 127.46, 127.11, 126.45, 126.01, 121.72, 21.23, -2.39. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{22}\text{NaO}_2\text{Si}$: 369.1281 ($[\text{M}+\text{Na}]^+$), found 369.1281 ($[\text{M}+\text{Na}]^+$).



2i (a pale yellow oil, 43%, 14.2 mg) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-acetylstyrene (146.2 mg, 1.00 mmol) by a general procedure A.

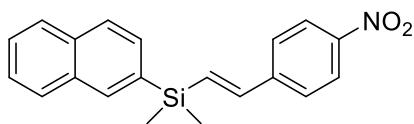
^1H -NMR (CDCl_3 , 400 MHz) δ : 8.06 (1H, s), 7.94-7.91 (2H, m), 7.87-7.83 (3H, m), 7.63 (1H, d, $J = 8.4$ Hz), 7.54-7.48 (4H, m), 7.00 (1H, d, $J = 19.0$ Hz), 6.81 (1H, d, $J = 19.0$ Hz), 2.60 (3H, s), 0.53 (6H, s). ^{13}C -NMR (CDCl_3 , 100 MHz) δ : 197.76, 144.21, 142.54, 136.51, 135.42, 134.69, 133.85, 133.00, 131.24, 130.17, 128.82, 128.14, 127.81, 127.21, 126.69, 126.57, 126.10, 26.75, -2.50. HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{22}\text{NaOSi}$: 353.1332 ($[\text{M}+\text{Na}]^+$), found 353.1331 ($[\text{M}+\text{Na}]^+$).



2j

2j (a pale yellow oil, 51%, 17.7 mg) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-(Methoxycarbonyl)styrene (162.2 mg, 1.00 mmol) by a general procedure A.

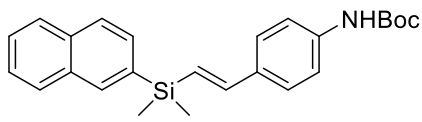
$^1\text{H-NMR}$ (CDCl_3 , 500 MHz) δ : 8.07 (1H, s), 8.02-8.00 (2H, m), 7.86-7.85 (3H, m), 7.64 (1H, d, $J = 8.2$ Hz), 7.52-7.49 (4H, m), 7.00 (1H, d, $J = 18.9$ Hz), 6.80 (1H, d, $J = 18.9$ Hz), 3.92 (3H, s), 0.54 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 166.97, 144.35, 142.40, 135.47, 134.68, 133.85, 133.00, 130.87, 130.18, 129.99, 129.54, 128.15, 127.81, 127.20, 126.55, 126.50, 126.08, 52.18, -2.50 HRMS (ESI) calcd for $\text{C}_{22}\text{H}_{22}\text{NaO}_2\text{Si}$: 369.1276 ($[\text{M}+\text{Na}]^+$) found 369.1278 ($[\text{M}+\text{Na}]^+$)



2k

2k (a pale yellow oil, 35%, 11.7 mg) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-nitrostyrene (162.2 mg, 1.00 mmol) by a general procedure A.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 8.21-8.18 (2H, m), 8.06 (1H, s), 7.86-7.84 (3H, m), 7.63 (1H, dd, $J = 8.3, 0.9$ Hz), 7.58-7.56 (2H, m), 7.52-7.49 (2H, m), 7.00 (1H, d, $J = 18.8$ Hz), 6.87 (1H, d, $J = 18.8$ Hz), 0.55 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 147.32, 144.19, 142.96, 134.90, 134.74, 133.94, 133.90, 133.00, 130.06, 128.14, 127.83, 127.33, 127.16, 126.68, 126.18, 124.06, -2.61 HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{19}\text{NNaO}_2\text{Si}$: 356.1072 ($[\text{M}+\text{Na}]^+$) found 356.1075 ($[\text{M}+\text{Na}]^+$)

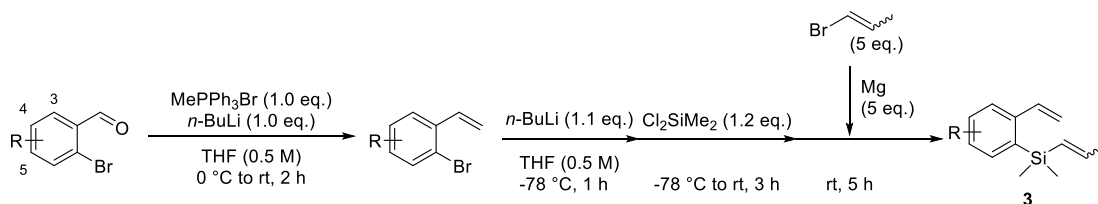


2l

2l (a pale yellow oil, 59%, 23.8 mg) was prepared from **1b** (22.6 mg, 0.100 mmol) and 4-(*tert*-butoxycarbonylamino)styrene (219.3 mg, 1.00 mmol) by a general procedure A

$^1\text{H-NMR}$ (CDCl_3 , 500 MHz) δ : 8.06 (1H, s), 7.84-7.83 (3H, m), 7.64 (1H, d, $J = 8.3$ Hz), 7.50-7.47 (2H, m), 7.40 (2H, d, $J = 8.6$ Hz), 7.33 (2H, d, $J = 8.6$ Hz), 6.92 (1H, d, $J = 19.2$ Hz), 6.53 (1H, d, $J = 19.2$ Hz), 6.53 (1H, br s), 0.51 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 152.62, 144.88, 138.37, 136.22, 134.61, 133.79, 133.21, 133.00, 130.32, 128.15, 127.78, 127.34, 127.06, 126.39, 125.96, 125.22, 118.33, 80.74, 28.41, -2.33 HRMS (ESI) calcd for $\text{C}_{25}\text{H}_{29}\text{NNaO}_2\text{Si}$: 426.1865 ($[\text{M}+\text{Na}]^+$) found 426.1850 ($[\text{M}+\text{Na}]^+$)

3. Synthesis of ring-closing metathesis substrate **3** (general procedure **B**)

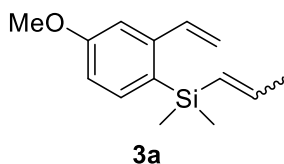


To a solution of MePPh₃Br (1.0 eq.) in THF (0.5 M) was dropwise 2.76 M solution of *n*-BuLi (1.0 eq.) in *n*-hexane at 0 °C to room temperature over a period of 2 h. After that, a solution of an *o*-bromobenzaldehyde derivative (1.0 eq.) was added to the reaction mixture at 0 °C and warmed to room temperature. After stirring for 2 h, the reaction was stopped by the addition of 1 M HCl aq. to the reaction mixture. The organic compound was extracted with CHCl₃. The organic layer was dried over Na₂SO₄, and the solvent was evaporated. The residue was subjected to column chromatography (only *n*-hexane) on silica gel 60N to give an *o*-bromostyrene derivative.

To a solution of the *o*-bromostyrene derivative in THF (0.5 M) was added dropwise 2.76 M solution of *n*-BuLi (1.1 eq.) in *n*-hexane at -78 °C. After the mixture was stirred at -78 °C for 1 h, the mixture was added dropwise to a solution of Me₂SiCl₂ (1.2 eq.) in THF (2 M). The reaction mixture was warmed to room temperature over 2 h. We call this reaction mixture, solution α .

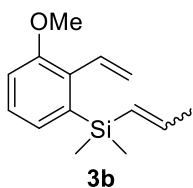
On the other hand, to a mixture of magnesium (3.0 eq.) in THF (0.2 M) was added dropwise a solution of 1-bromopropene in THF (0.2 M) at 50 °C for 3 h. We call this reaction mixture, solution β .

To the solution α was dropwisely added the solution β at room temperature and the reaction mixture was heated to 50 °C for 3 h. After that, the reaction was stopped by the addition of 1M HCl aq. to the reaction mixture. The organic compound was extracted with CHCl₃. The organic layer was dried over Na₂SO₄, and the solvent was evaporated. The residue was subjected to column chromatography (only *n*-hexane) on silica gel to give compound **3**.



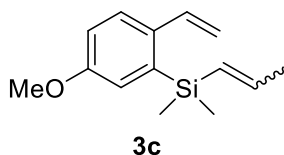
3a (a colorless oil, *E/Z* = 4/1, 64%, 743 mg, 3.20 mmol) was prepared from 2-bromo-5-methoxybenzaldehyde (1.08 g, 5.00 mmol) by a general procedure **B**.

¹H-NMR (CDCl₃, 400 MHz) δ : 7.48 (1H, d, *J* = 7.9 Hz), 7.11 (1H, dd, *J* = 2.4, 2.4 Hz), 7.05 (1H, dd, *J* = 17.1, 11.0 Hz), 6.82 (1H, dd, *J* = 7.9, 2.4 Hz), 6.48 (1H, dq, *J* = 14.0, 7.0 Hz), 5.70 (1H, dq, *J* = 14.0, 1.5 Hz), 5.63 (1H, dd, *J* = 17.1, 1.2 Hz), 5.25 (1H, dd, *J* = 10.4, 1.2 Hz), 3.84 (3H, s), 1.66 (3H, dd, *J* = 7.0, 1.5 Hz), 0.40 (6H, s). ¹³C-NMR (CDCl₃, 100 MHz) δ : 160.7, 145.4, 144.4, 138.1, 136.1, 130.2, 129.1, 114.9, 112.6, 110.7, 55.1, 19.1, 0.0. HRMS (ESI) calcd for C₁₄H₂₀OSi: 255.1181 ([M+Na]⁺) found 255.1170 ([M+Na]⁺)



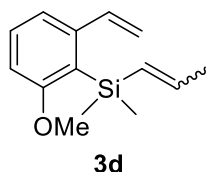
3b (a colorless oil, $E/Z = 18/1$, a colorless oil, 23%, 268 mg, 1.15 mmol) was prepared from 2-bromo-6-methoxybenzaldehyde (1.08 g, 5.00 mmol) by a general procedure **B**.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.22 (1H, dd, $J = 9.0, 7.3$ Hz), 7.15 (1H, dd, $J = 7.3, 1.2$ Hz), 6.94 (1H, dd, $J = 9.0, 1.2$ Hz), 6.89 (1H, dd, $J = 17.7, 11.6$ Hz), 6.11 (1H, dq, $J = 18.3, 6.1$ Hz), 5.84 (1H, dd, $J = 18.3, 1.8$ Hz), 5.77 (1H, dd, $J = 17.7, 2.4$ Hz), 5.47 (1H, dd, $J = 11.6, 2.4$ Hz), 3.84 (3H, s), 1.84 (3H, dd, $J = 6.1, 1.8$ Hz), 0.36 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 157.5, 143.4, 139.9, 134.8, 132.6, 130.4, 127.5, 127.3, 119.6, 112.1, 55.4, 22.8, -0.7. HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{20}\text{OSi}$: 255.1181 ($[\text{M}+\text{Na}]^+$) found 255.1174 ($[\text{M}+\text{Na}]^+$)



3c (a colorless oil, $E/Z = 1.8/1$, 46%, 533 mg, 2.29 mmol) was prepared from 2-bromo-4-methoxybenzaldehyde (1.08 g, 5.00 mmol) by a general procedure **B**.

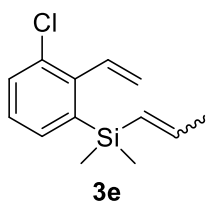
$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.55 (1H, d, $J = 8.5$ Hz), 7.12 (1H, d, $J = 2.7$ Hz), 7.01 (1H, dd, $J = 17.4, 11.0$ Hz), 6.90 (1H, dd, $J = 8.5, 2.7$ Hz), 6.51 (1H, dq, $J = 13.8, 6.8$ Hz), 5.71 (1H, dd, $J = 13.8, 1.2$ Hz), 5.54 (1H, dd, $J = 17.4, 1.2$ Hz), 5.14 (1H, dd, $J = 11.0, 1.2$ Hz), 3.8 (3H, s), 1.68 (3H, dd, $J = 6.8, 1.6$ Hz), 0.44-0.38 (6H, m). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 158.6, 144.9, 139.6, 137.7, 136.5, 128.7, 126.5, 120.4, 114.4, 112.8, 55.4, 19.4, -0.1. HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{20}\text{OSi}$: 233.1362 ($[\text{M}+\text{H}]^+$) found 233.1358 ($[\text{M}+\text{H}]^+$)



3d (a colorless oil, $E/Z = 11/5$, 40%, 158 mg, 0.679 mmol) was prepared from 2-bromo-3-methoxybenzaldehyde (538 mg, 2.50 mmol) by a general procedure **B** (2-bromo-3-methoxybenzaldehyde was synthesized from 2-bromo-3-hydroxybenzaldehyde by method of previous report¹).

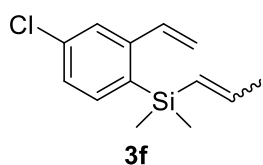
$^1\text{H-NMR}$ (CDCl_3 , 500 MHz) δ : 7.31 (1H, dd, $J = 8.0, 7.6$ Hz), 7.15 (1H, d, $J = 7.6$ Hz), 7.11 (1H, dd, $J = 16.0, 10.7$ Hz), 6.77 (1H, d, $J = 8.0$ Hz), 6.09 (1H, dq, $J = 18.3, 6.1$ Hz), 5.94 (1H, d, $J = 18.3$ Hz), 5.53 (1H, dd, $J = 16.0, 1.5$ Hz), 5.20 (1H, dd, $J = 10.7, 1.5$ Hz), 3.79 (3H, s), 1.84 (3H, dd, $J = 6.1, 1.6$ Hz), 0.42 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 125 MHz) δ : 164.79, 146.29, 141.51, 139.80, 132.33, 131.45, 130.53, 125.00, 119.55, 115.02, 109.25, 55.28, 22.69, 0.90. HRMS (APCI) calcd for $\text{C}_{14}\text{H}_{20}\text{OSi}$: 233.1356 ($[\text{M}+\text{H}]^+$) found 233.1352 ($[\text{M}+\text{H}]^+$)

Ref. 1) Guoqing Zhao, Guangqing Xu, Chao Qian and Wenjun Tan *J. Am. Chem. Soc.* **2017**, *139*, 3360-3363



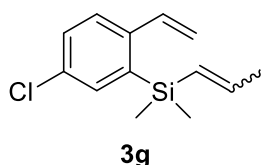
3e (a colorless oil, *E/Z* = 12/1, 15%, 178 mg, 0.753 mmol) was prepared from 2-bromo-6-chlorobenzaldehyde (1.10 g, 5.00 mmol) by a general procedure **B**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.43 (1H, d, *J* = 7.3 Hz), 7.38 (1H, d, *J* = 8.0 Hz), 7.17 (1H, dd, *J* = 8.0, 7.3 Hz), 6.85 (1H, dd, *J* = 17.1, 11.5 Hz), 6.07 (1H, dq, *J* = 18.3, 6.1 Hz), 5.80 (1H, dq, *J* = 18.3, 1.3 Hz), 5.56 (1H, dd, *J* = 11.5, 1.3 Hz), 5.45 (1H, dd, *J* = 17.1, 1.3 Hz), 1.84 (3H, dd, *J* = 6.1, 1.3 Hz), 0.33 (6H, s). ¹³C NMR (CDCl₃, 100 MHz) δ: 143.7, 140.5, 135.9, 133.6, 133.1, 130.4, 130.4, 130.2, 127.5, 121.5, 22.8, -0.7. HRMS (ESI) calcd for C₁₃H₁₇ClSi: 259.0686 ([M+Na]⁺) found 259.0685 ([M+Na]⁺)



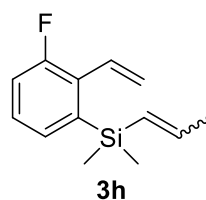
3f (a colorless oil, *E/Z* = 1/1, 21%, 250 mg, 1.05 mmol) was prepared from 2-bromo-5-chlorobenzaldehyde (1.10 g, 5.00 mmol) by a general procedure **B**.

¹H-NMR (CDCl₃) δ: 7.53 (1H, d, *J* = 2.3 Hz), 7.48 (1H, d, *J* = 8.0 Hz), 7.22 (1H, dd, *J* = 8.0, 2.3 Hz), 7.05 (1H, dd, *J* = 8.0, 8.0 Hz), 6.50 (1H, dq, *J* = 14.0, 6.7 Hz), 5.69 (1H, dq, *J* = 14.0, 1.4 Hz), 5.66 (1H, dd, *J* = 4.0, 1.3 Hz), 5.31 (1H, d, *J* = 4.0 Hz), 1.65 (3H, dd, *J* = 6.7, 1.4 Hz), 0.36 (6H, s). ¹³C-NMR (CDCl₃, 125 MHz) δ: 145.5, 145.1, 137.1, 136.3, 136.0, 135.8, 128.2, 126.8, 125.2, 116.0, 22.8, -0.3. HRMS (ESI) calcd for C₁₃H₁₇ClSi: 259.0686 ([M+Na]⁺) found 259.0681 ([M+Na]⁺)



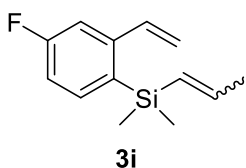
3g (a colorless oil, only *E*, 20%, 234mg, 0.99 mmol) was prepared from 2-bromo-4-chlorobenzaldehyde (1.10 g, 5.00 mmol) by a general procedure **B**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.51 (1H, d, *J* = 1.8 Hz), 7.41 (1H, d, *J* = 7.9 Hz), 7.20 (1H, dd, *J* = 7.9, 1.8 Hz), 7.02 (1H, dd, *J* = 17.4, 11.0 Hz), 6.12 (1H, dq, *J* = 18.3, 6.1 Hz), 5.81 (1H, dq, *J* = 18.3, 1.8 Hz), 5.63 (1H, d, *J* = 17.4 Hz), 5.29 (1H, d, *J* = 11.0 Hz), 1.85 (3H, dd, *J* = 6.1, 1.8 Hz), 0.34 (6H, s). ¹³C-NMR (CDCl₃, 100 MHz) δ: 145.7, 144.4, 137.2, 136.3, 135.8, 135.5, 129.3, 126.8, 125.2, 116.0, 22.8, -1.3. HRMS (ESI) calcd for C₁₃H₁₇ClSi: 259.0686 ([M+Na]⁺) found 259.0675 ([M+Na]⁺)



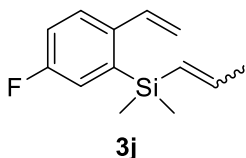
3h (a colorless oil, *E/Z* = 1.8/1, 34%, 378 mg, 1.72 mmol) was prepared from 2-bromo-6-fluorobenzaldehyde (1.01 g, 5.00 mmol) by a general procedure **B**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.30-7.28 (1H, m), 7.19-7.17 (1H, m), 7.10-7.05 (1H, m), 6.80 (1H, dd, *J* = 18.0, 11.9 Hz), 6.16-6.10 (1H, m), 5.85-5.77 (2H, m), 5.55-5.52 (1H, m), 1.85 (3H, dd, *J* = 6.4, 1.5 Hz), 0.37 (6H, s). ¹³C-NMR (CDCl₃, 100 MHz) δ: 161.1 (*J* = 251 Hz), 144.5 (*J* = 60 Hz), 132.1, 130.6 (*J* = 20 Hz), 130.6 (*J* = 7 Hz), 129.5, 128.4, 127.8 (*J* = 3 Hz), 120.6 (*J* = 11 Hz), 117.1 (*J* = 23 Hz), 22.8, -1.1. ¹⁹F-NMR (CDCl₃, 376 MHz) δ: -110.2 (m) HRMS (ESI) calcd for C₁₃H₁₇FSi: 243.0981 ([M+Na]⁺) found 243.0971 ([M+Na]⁺)



3i (a colorless oil, *E/Z* = 20/1, 49%, 542 mg, 2.46 mmol) was prepared from 2-bromo-5-fluorobenzaldehyde (1.01 g, 5.00 mmol) by a general procedure **B**.

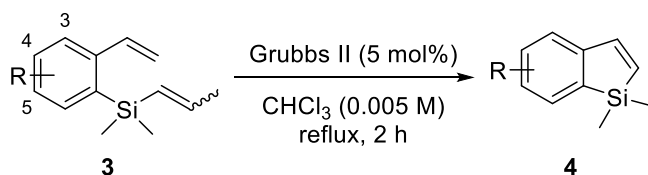
¹H-NMR (CDCl₃, 400 MHz) δ: 7.29 (1H, d, *J* = 7.6 Hz), 7.21-7.17 (1H, m), 7.07 (1H, dd, *J* = 11.6, 7.6 Hz), 6.80 (1H, dd, *J* = 17.8, 12.3 Hz), 6.13 (1H, dq, *J* = 12.0, 6.1 Hz), 5.81 (2H, dd, *J* = 17.8, 12.3 Hz), 5.53 (1H, dq, *J* = 12.0, 1.8 Hz), 1.85 (3H, dd, *J* = 6.1, 1.8 Hz), 0.39 (6H, s). ¹³C-NMR (CDCl₃, 100 MHz) δ: 162.3 (*J* = 250 Hz), 144.2, 141.1, 132.1, 130.6 (*J* = 20 Hz), 130.6 (*J* = 7 Hz), 129.5, 127.8 (*J* = 8 Hz), 120.6 (*J* = 11 Hz), 117.1 (*J* = 23 Hz), 22.8, -1.1 ¹⁹F-NMR (CDCl₃, 376 MHz) δ: -110.2 (m). HRMS (ESI) calcd for C₁₃H₁₇FSi: 243.0981 ([M+Na]⁺) found 243.0973 ([M+Na]⁺)



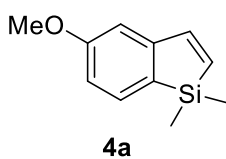
3j (a colorless oil, *E/Z* = 4/1, 49%, 542 mg, 2.46 mmol) was prepared from 2-bromo-4-fluorobenzaldehyde (1.01 g, 5.00 mmol) by a general procedure **B**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.73-7.71 (1H, m), 7.44-7.36 (1H, m), 7.26-7.19 (2H, m), 6.34 (1H, dq, *J* = 18.3, 6.1 Hz), 6.02 (1H, dd, *J* = 18.3, 1.5 Hz), 5.75 (1H, d, *J* = 18.3 Hz), 5.42 (1H, d, *J* = 11.0 Hz), 2.06 (3H, dd, *J* = 6.1, 1.5 Hz), 0.56 (6H, d, *J* = 3.7 Hz). ¹³C-NMR (CDCl₃, 100 MHz) δ: 162.0 (*J* = 247 Hz), 144.6, 140.2 (*J* = 4 Hz), 137.2, 129.1, 127.9, 127.0 (*J* = 7 Hz), 121.1 (*J* = 19 Hz), 116.2 (*J* = 21 Hz), 114.5, 22.8, -1.5 ¹⁹F-NMR (CDCl₃, 376 MHz) δ: -110.0 (m). HRMS (ESI) calcd for C₁₃H₁₇FSi: 243.0981 ([M+Na]⁺) found 243.0973 ([M+Na]⁺)

4. Ring-closing metathesis of **3** (general procedure **C**)



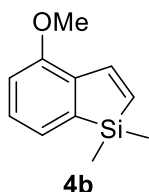
To a solution of **14** in CHCl₃ (0.005 M) was added Grubbs II (5 mol%) and the mixture was refluxed for 2 h. The solvent was evaporated and the residue was subjected to column chromatography on neutral flash silica gel 60N to give **15**.



4a (a colorless oil, quant, 19.0 mg, 0.0998 mmol) was prepared from **3a** (23.2 mg, 0.100 mmol) by a general procedure **C**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.47 (1H, d, *J* = 7.8 Hz), 7.32 (1H, d, *J* = 10.1 Hz), 6.88 (1H, s), 6.80 (1H, dd, *J* = 7.8, 1.0 Hz), 6.36 (1H, dd, *J* = 10.1, 1.0 Hz),

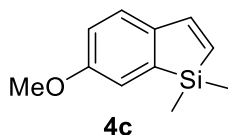
3.84 (3H, s), 0.35 (6H, s). ^{13}C -NMR (CDCl_3 , 100 MHz) δ : 161.6, 151.4, 148.8, 134.2, 132.7, 129.0, 111.9, 110.9, 55.3, -3.7. HRMS (ESI) calcd for $\text{C}_{11}\text{H}_{14}\text{OSi}$: 213.0712 ($[\text{M}+\text{Na}]^+$) found 213.0703 ($[\text{M}+\text{Na}]^+$)



4b (a colorless oil, 35%, 6.6 mg, 0.0348 mmol) was prepared from **3b** (23.2 mg, 0.100 mmol) by a general procedure C.

^1H -NMR (CDCl_3 , 500 MHz) δ : 7.67 (1H, d, $J = 10.3$ Hz), 7.22 (1H, dd, $J = 8.0, 6.9$ Hz), 7.12 (1H, d, $J = 6.9$ Hz), 6.87 (1H, d, $J = 8.0$ Hz), 6.19 (1H, d, $J = 10.3$ Hz), 3.85 (3H, s), 0.31 (6H, s). ^{13}C NMR (CDCl_3 , 100 MHz) δ : 154.31, 143.93, 140.55, 137.02,

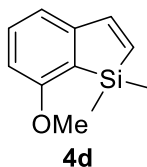
130.66, 128.51, 123.88, 112.49, 55.43, -3.96. HRMS (APCI) calcd for $\text{C}_{11}\text{H}_{14}\text{OSi}$: 191.0887 ($[\text{M}+\text{H}]^+$) found 191.0885 ($[\text{M}+\text{H}]^+$)



4c (a colorless oil, quant, 19.0 mg, 0.100 mmol) was prepared from **3c** (23.2 mg, 0.100 mmol) by a general procedure C.

^1H -NMR (CDCl_3 , 400 MHz) δ : 7.32 (1H, d, $J = 10.3$ Hz), 7.19 (1H, d, $J = 8.1$ Hz), 7.12 (1H, d, $J = 2.4$ Hz), 6.83 (1H, dd, $J = 8.1, 2.4$ Hz), 6.12 (1H, d, $J =$

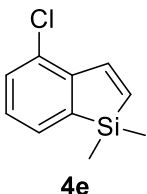
10.3 Hz), 3.84 (3H, s), 0.33 (6H, s). ^{13}C NMR (CDCl_3 , 100 MHz) δ : 159.1, 148.8, 142.2, 140.8, 129.7, 124.8, 118.4, 113.8, 55.5, -3.7. HRMS (APCI) calcd for $\text{C}_{11}\text{H}_{14}\text{OSi}$: 191.0887 ($[\text{M}+\text{H}]^+$) found 191.0883 ($[\text{M}+\text{H}]^+$)



4d (a colorless oil, 63%, 14.6 mg, 0.0626 mmol) was prepared from **3d** (23.3 mg, 0.100 mmol) by a general procedure C.

^1H -NMR (CDCl_3 , 500 MHz) δ : 7.28 (1H, dd, $J = 7.5, 7.5$ Hz), 7.23 (1H, d, $J = 10.8$ Hz), 6.86 (1H, d, $J = 7.5$ Hz), 6.70 (1H, d, $J = 7.5$ Hz), 6.22 (1H, d, $J = 10.8$ Hz), 3.79 (3H, s), 0.32 (6H, s). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 163.15, 151.04, 148.31, 133.44,

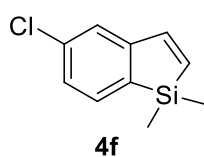
132.00, 117.49, 109.41, 55.37, -4.41. HRMS (APCI) calcd for $\text{C}_{11}\text{H}_{14}\text{OSi}$: 191.0887 ($[\text{M}+\text{H}]^+$) found 191.0883 ($[\text{M}+\text{H}]^+$)



4e (a colorless oil, 25%, 4.9 mg, 0.0252 mmol) was prepared from **3e** (23.7 mg, 0.100 mmol) by a general procedure C.

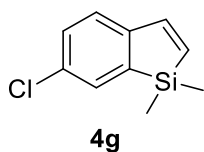
^1H -NMR (CDCl_3 , 300 MHz) δ : 7.65 (1H, d, $J = 10.6$ Hz), 7.37 (1H, d, $J = 6.9$ Hz), 7.29 (1H, dd, $J = 7.8, 0.9$ Hz), 7.14 (1H, dd, $J = 7.8, 6.9$ Hz), 6.38 (1H, d, $J = 10.6$ Hz), 0.32 (7H, s). ^{13}C -NMR (CDCl_3 , 100 MHz) δ : 145.76, 145.68, 141.34, 134.12, 130.41,

130.15, 129.75, 128.37, -4.08. HRMS (APCI) calcd for $\text{C}_{10}\text{H}_{11}\text{ClSi}$: 195.0391 ($[\text{M}+\text{H}]^+$) found 195.0387 ($[\text{M}+\text{H}]^+$)



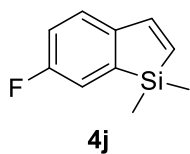
4f (a colorless oil, 62%, 12.1 mg, 0.0621 mmol) was prepared from **3f** (23.7 mg, 0.100 mmol) by a general procedure C.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.42 (1H, d, $J = 7.3$ Hz), 7.26 (1H, d, $J = 10.4$ Hz), 7.23 (1H, d, $J = 2.3$ Hz), 7.19 (1H, dd, $J = 7.3, 2.3$ Hz), 6.35 (1H, d, $J = 10.4$ Hz), 0.32 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 151.1, 148.0, 136.5, 135.9, 134.6, 132.6, 126.7, 124.3, -4.1. HRMS (ESI) calcd for $\text{C}_{10}\text{H}_{11}\text{ClSi}$: 195.0397 ($[\text{M}+\text{H}]^+$) found 195.0391 ($[\text{M}+\text{H}]^+$)



4g (a colorless oil, 80%, 15.6 mg, 0.0801 mmol) was prepared from **3g** (23.7 mg, 0.100 mmol) by a general procedure C.

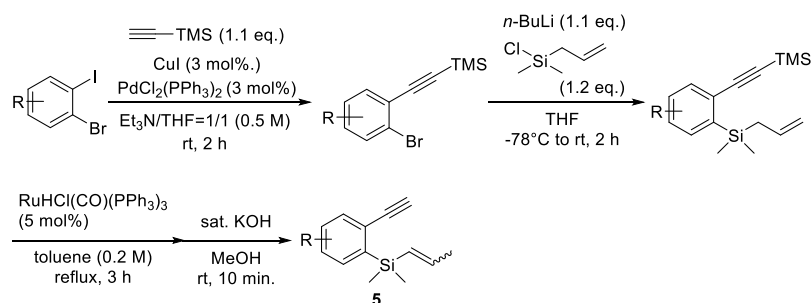
$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.42 (1H, d, $J = 2.1$ Hz), 7.25 (1H, d, $J = 10.3$ Hz), 7.23 (1H, dd, $J = 8.0, 2.1$ Hz), 7.11 (1H, d, $J = 8.0$ Hz), 6.23 (1H, d, $J = 10.3$ Hz), 0.29 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 148.3, 147.5, 141.1, 133.2, 132.9, 131.8, 129.5, 125.1, -4.1. LRMS (EI) calcd for $\text{C}_{10}\text{H}_{11}\text{ClSi}$: 217.0216 ($[\text{M}+\text{Na}]^+$) found 217.0211 ($[\text{M}+\text{Na}]^+$)



4j (a colorless oil, 42%, 7.4 mg, 0.0415 mmol) was prepared from **3j** (20.2 mg, 0.100 mmol) by a general procedure C.

$^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.29 (1H, d, $J = 10.5$ Hz), 7.19 (1H, dd, $J = 7.8, 0.9$ Hz), 7.19 (1H, dd, $J = 8.6, 7.8$ Hz), 6.97 (1H, ddd, $J = 8.6, 6.4, 0.9$ Hz), 6.21 (1H, d, $J = 10.5$ Hz), 0.31 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 162.6 ($J = 246$ Hz), 148.3, 145.1, 141.7, 131.8, 125.1, 118.8 ($J = 20$ Hz), 115.9 ($J = 22$ Hz), -4.0. $^{19}\text{F-NMR}$ (CDCl_3 , 376 MHz) δ : -111.6 (m). HRMS (APCI) calcd for $\text{C}_{10}\text{H}_{11}\text{FSi}$: 179.0687 ($[\text{M}+\text{H}]^+$) found 179.0684 ($[\text{M}+\text{H}]^+$)

5. Synthesis of enyne metathesis substrate **5** (general procedure D)

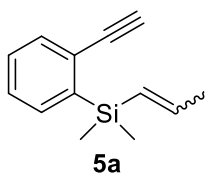


To a solution of $\text{PdCl}_2(\text{PPh}_3)_2$ (3 mol %), CuI (3 mol%) and an *o*-bromoiodobenzene derivative (1.0 eq.) in $\text{Et}_3\text{N}/\text{THF}=1/1$ (0.5 M) was added trimethylsilylacetylene (1.1 eq.) at room temperature. After the mixture was stirred at room temperature for 2 h, the mixture was filtrated through celite cake and the solvent was evaporated under reduced pressure. The residue was subjected to column

chromatography on neutral flash silica gel 60N to give an *o*-bromotrimethylsilylacetylene derivative.

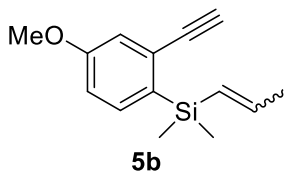
To a solution of compound *o*-bromotrimethylsilylacetylene derivative in THF (0.5 M) was added dropwise 2.76 M *n*-BuLi in *n*-hexane (1.1 eq.) at -78 °C. After the mixture was stirred at -78 °C for 1 h, allylchlorodimethylsilane (1.2 eq.) was added dropwise to the mixture. The reaction mixture was stirred at -78 °C for 1 h, and warmed to room temperature. The reaction was stopped with the addition of 1M HCl aq. to the reaction mixture and organic compound was extracted with CHCl₃. The organic layer was dried over Na₂SO₄, and the solvent was evaporated. The residue was subjected to column chromatography on neutral flash silica gel 60N to give compound *o*-trimethylsilylacetylene-dimethylallylsilylbenzene derivative.

To a solution of compound *o*-trimethylsilylacetylenedimethylallylsilylbenzene derivative in toluene (0.2 M) was added RuHCl(CO)(PPh₃)₃ (5 mol%) and the mixture was refluxed for 3 h. After cooled to room temperature, saturated KOH in MeOH was added to the reaction mixture and stirred for 10 minutes. The reaction was stopped with 1M HCl aq. and organic compound was extracted with CHCl₃. The organic layer was dried over Na₂SO₄, and the solvent was evaporated. The residue was subjected to column chromatography on neutral flash silica gel 60N to give compound **5**.



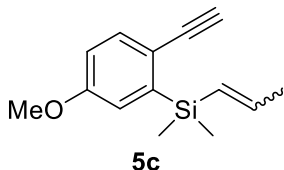
5a (a colorless oil, *E/Z* = 10/1, 40%, 397 mg, 1.98 mmol) was prepared from *o*-bromiodobenzene (1.41 g, 5.00 mmol) by a general procedure **D**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.55-7.54 (2H, m), 7.33-7.33 (2H, m), 6.19 (1H, dq, *J* = 20.0, 6.4 Hz), 5.99 (1H, d, *J* = 20.0 Hz), 3.24 (1H, s), 1.90 (3H, d, *J* = 6.4 Hz), 0.46 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ: 147.1, 145.2, 137.6, 136.4, 131.9, 131.8, 131.0, 130.4, 88.3, 83.3, 25.8, 0.6. HRMS (EI) calcd for C₁₃H₁₆Si: 223.0919 ([M+Na]⁺) found 223.0912 ([M+Na]⁺)



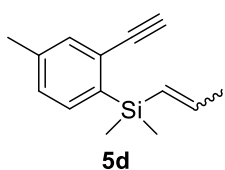
12b (a colorless oil, *E/Z* = 10/1, 32%, 327 mg, 1.42 mmol) was prepared from 3-bromo-2-iodoanisole (1.37 g, 4.39 mmol) by a general procedure **D**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.49 (1H, dd, *J* = 8.5, 5.3 Hz), 7.17 (1H, dd, *J* = 8.8, 2.7 Hz), 6.97 (1H, ddd, *J* = 8.8, 8.5, 2.7 Hz), 6.18 (1H, dq, *J* = 18.3, 6.2 Hz), 5.90 (1H, dq, *J* = 18.3, 1.6 Hz), 3.80 (3H, s), 3.17 (1H, s), 1.87 (3H, dd, *J* = 6.2, 1.6 Hz), 0.43 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ: 163.0, 146.8, 139.2, 136.3, 132.4, 131.8, 121.6, 117.6, 88.2, 83.2, 58.2, 25.9, 0.9. HRMS (ESI) calcd for C₁₄H₁₈OSi: 231.1205 ([M+H]⁺) found 231.1197 ([M+H]⁺)



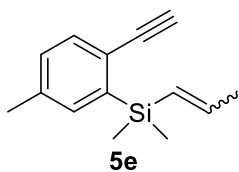
5c (a yellow oil, *E/Z* = 10/1, 44%, 505 mg, 2.19 mmol) was prepared from 2-bromo-3-iodoanisole (1.56 g, 5.00 mmol) by a general procedure **D**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.47 (1H, d, *J* = 8.2 Hz), 7.03 (1H, d, *J* = 2.7 Hz), 6.81 (1H, dd, *J* = 8.2, 2.7 Hz), 6.18 (1H, dq, *J* = 18.4, 6.1 Hz), 5.94 (1H, dq, *J* = 18.4, 1.2 Hz), 3.81 (3H, s), 3.13 (1H, s), 1.87 (3H, dd, *J* = 6.1, 1.2 Hz), 0.43 (6H, s). ¹³C-NMR (CHCl₃, 100 MHz) δ: 159.0, 144.3, 144.2, 135.1, 128.7, 120.9, 119.4, 113.4, 85.2, 78.8, 55.2, 22.8, -2.5. HRMS (ESI) calcd for C₁₄H₁₈OSi: 231.1205 ([M+H]⁺) found 231.1198 ([M+H]⁺)



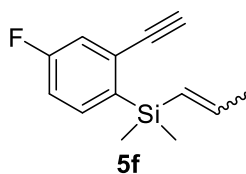
5d (a pale yellow oil, *E/Z* = 10/1, 44%, 476 mg, 2.22 mmol) was prepared from 3-bromo-4-iodoluene (1.57 g, 5.00 mmol) by a general procedure **D**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.42 (1H, d, *J* = 7.3 Hz), 7.39 (1H, s), 7.16 (1H, d, *J* = 7.3 Hz), 6.20 (1H, dq, *J* = 19.1, 6.1 Hz), 5.98 (1H, d, *J* = 19.1 Hz), 3.21 (1H, s), 2.34 (3H, s), 1.90 (3H, d, *J* = 6.0 Hz), 0.46 (6H, s). ¹³C-NMR (CHCl₃, 100 MHz) δ: 143.9, 138.7, 138.6, 134.8, 134.1, 129.2, 129.0, 127.4, 85.4, 80.1, 22.9, 21.2, -2.3. HRMS (ESI) calcd for C₁₄H₁₈Si: 237.1075 ([M+Na]⁺) found 237.1068 ([M+Na]⁺)



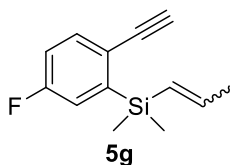
5e (a yellow oil, *E/Z* = 10/1, 52%, 560 mg, 2.61 mmol) was prepared from 2-bromo-3-iodoluene (1.57 g, 5.00 mmol) by a general procedure **D**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.44 (1H, d, *J* = 7.8 Hz), 7.31 (1H, d, *J* = 1.4 Hz), 7.13 (1H, dd, *J* = 7.8, 1.4 Hz), 6.19 (1H, dq, *J* = 18.3, 6.0 Hz), 5.97 (1H, dq, *J* = 18.3, 1.6 Hz), 3.18 (1H, s), 2.35 (3H, s), 1.89 (3H, dd, *J* = 6.0, 1.6 Hz), 0.45 (6H, s). ¹³C-NMR (CHCl₃, 100 MHz) δ: 143.9, 141.9, 137.8, 135.3, 133.4, 129.6, 129.0, 124.4, 85.4, 79.6, 22.8, 21.7, -2.4. HRMS (ESI) calcd for C₁₄H₁₈Si: 241.0825 ([M+Na]⁺) found 241.0818 ([M+Na]⁺)



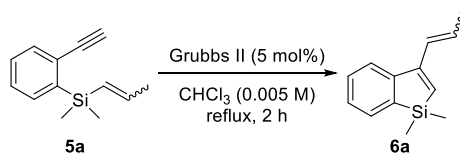
5f (a pale yellow oil, *E/Z* = 10/1, 17%, 186 mg, 0.851 mmol) was prepared from 2-bromo-5-fluoroiodobenzene (1.50 g, 5.00 mmol) by a general procedure **D**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.47 (1H, dd, *J* = 8.2, 6.4 Hz), 7.22 (1H, dd, *J* = 9.6, 2.3 Hz), 7.02 (1H, ddd, *J* = 8.2, 8.2, 2.3 Hz), 6.18 (1H, dq, *J* = 19.2, 6.0 Hz), 5.92 (1H, dd, *J* = 19.2, 1.8 Hz), 3.26 (1H, s), 1.88 (3H, dd, *J* = 6.0, 1.8 Hz), 0.44 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ: 162.0 (*J* = 248 Hz), 144.3, 137.9 (*J* = 4 Hz), 136.7 (*J* = 9 Hz), 129.4 (*J* = 8 Hz), 128.7, 120.2 (*J* = 22 Hz), 115.4 (*J* = 19 Hz), 84.1, 81.2, 22.8, -2.4. ¹⁹F-NMR (CDCl₃, 283 MHz) δ: -108.1 (m). HRMS (ESI) calcd for C₁₃H₁₅FSi: 241.0825 ([M+Na]⁺) found 241.0819 ([M+Na]⁺)



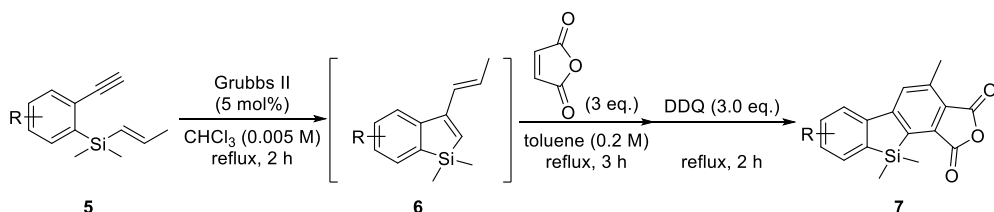
5g (a pale yellow oil, $E/Z = 3/1$, 13%, 143 mg, 0.655 mmol) was prepared from 2-bromo-4-fluoriodobenzene (1.50 g, 5.00 mmol) by a general procedure **D**. $^1\text{H-NMR}$ (CDCl_3 , 300 MHz) δ : 7.49 (1H, dd, $J = 8.6, 5.3$ Hz), 7.17 (1H, dd, $J = 8.6, 2.8$ Hz), 7.00-6.94 (1H, m), 6.19 (1H, dq, $J = 18.4, 6.2$ Hz), 5.91 (1H, dq, $J = 18.4, 1.6$ Hz), 3.18 (1H, s), 1.88 (3H, dd, $J = 6.2, 1.6$ Hz), 0.43 (6H, s). $^{13}\text{C-NMR}$ (100 MHz, CHCl_3) δ : 161.9 ($J = 250$ Hz), 145.8 ($J = 5$ Hz), 144.8, 135.5 ($J = 8$ Hz), 128.0, 123.1, 121.4 ($J = 20$ Hz), 115.7 ($J = 22$ Hz), 84.2, 80.0, 22.7, -2.8. $^{19}\text{F-NMR}$ (CDCl_3 , 283 MHz) δ : -107.7 (m). HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{15}\text{FSi}$: 241.0825 ($[\text{M}+\text{Na}]^+$) found 241.0818 ($[\text{M}+\text{Na}]^+$)

6. Synthesis of enyne metathesis compound **6a**



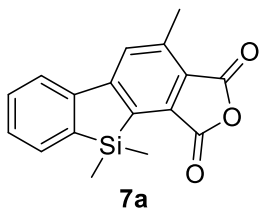
To a solution of **5a** (20.0 mg, 0.100 mmol) in CHCl_3 (2 mL, 0.05 M) was added Grubbs II (4.2 mg, 5 mol%) and the mixture was refluxed for 2 h. The solvent was evaporated and the residue was subjected to column chromatography on neutral flash silica gel 60N to give **6a** (a pale yellow oil, 60%, 12.1 mg, 0.0604 mmol, $E/Z = 4/1$). $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ : 7.53 (1H, dd, $J = 6.6, 1.2$ Hz), 7.45 (1H, d, $J = 7.3$ Hz), 7.35 (1H, ddd, $J = 7.8, 7.3, 1.2$ Hz), 7.24 (1H, dd, $J = 7.8, 6.6$ Hz), 6.54 (1H, dq, $J = 15.6, 1.2$ Hz), 6.31 (1H, dq, $J = 15.6, 6.5$ Hz), 6.21 (1H, s), 1.91 (3H, dd, $J = 6.5, 1.2$ Hz), 0.30 (6H, s). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ : 156.34, 148.95, 140.18, 131.81, 130.11, 129.60, 127.19, 126.81, 125.15, 121.47, 18.82, -3.79. HRMS (APCI) calcd for $\text{C}_{13}\text{H}_{16}\text{Si}$: 201.1094 ($[\text{M}+\text{H}]^+$) found 201.1090 ($[\text{M}+\text{H}]^+$)

7. Synthesis of polycyclic compound **7** (general procedure **E**)



To a solution of **5** in CHCl_3 (0.005 M) was added Grubbs II (5 mol%) and the mixture was reflux for 2 h. After the solvent was evaporated, the residue was dissolved in toluene (0.2 M). Then maleic anhydride (3.0 eq.) was added to the mixture and the mixture was reflux for 3 h. 2,3-dichloro-5,6-dicyano-*p*-benzoquinone (3.0 eq.) was added to the reaction mixture and the mixture was reflux for 3 h. The solvent was evaporated and the residue was subjected to column chromatography on neutral

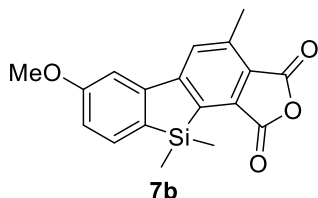
flash silica gel 60N to give **7**.



7a (a white solid, 52%, 15.3 mg, 0.0520 mmol) was prepared from **5a** (20.0 mg, 0.100 mmol) by a general procedure **E**.

¹H-NMR (CDCl₃, 400 MHz) δ : 7.98 (1H, s), 7.92 (1H, d, J = 7.9 Hz), 7.73 (1H, d, J = 7.0 Hz), 7.52 (1H, dd, J = 7.9, 7.0 Hz), 7.43 (1H, dd, J = 7.0, 7.0 Hz), 2.78 (3H, s), 0.56 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ : 164.7, 163.8, 156.3, 145.0, 142.6, 141.4, 136.9, 136.6, 133.5, 130.7, 129.8, 128.7, 126.5,

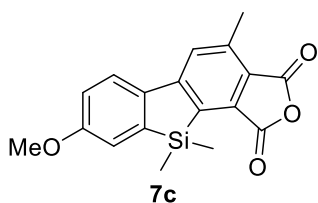
122.4, 18.4, -4.3 HRMS (MALDI-TOF) calcd for C₁₇H₁₅O₃Si: 295.0785 ([M+H]⁺), found 295.0785 ([M+H]⁺). m.p. >250 °C (recrystallized from CHCl₃, a white column).



7b (a yellow solid, 35%, 15.3 mg, 0.0520 mmol) was prepared from **5b** (23.0 mg, 0.100 mmol) by a general procedure **E**.

¹H-NMR (CDCl₃, 400 MHz) δ : 7.92 (1H, s), 7.64 (1H, d, J = 7.8 Hz), 7.42 (1H, d, J = 2.3 Hz), 6.99 (1H, dd, J = 7.8, 2.3 Hz), 3.92 (3H, s), 2.78 (3H, s), 0.53 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ : 164.6, 163.7,

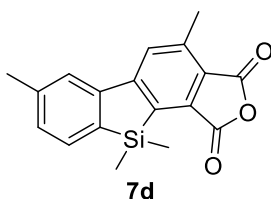
162.1, 155.9, 146.9, 142.4, 138.0, 136.4, 134.5, 132.0, 128.6, 126.5, 115.4, 108.4, 55.4, 18.3, -4.1 HRMS (MALDI-TOF) calcd for C₁₈H₁₆O₄Si: 324.0812 ([M+H]⁺), found 324.0805 ([M+H]⁺). m.p. 241.0-242.0 °C (recrystallized from CHCl₃, a pale yellow column).



7c (an orange solid, 48%, 15.7 mg, 0.0484 mmol) was prepared from **5c** (23.0 mg, 0.100 mmol) by general procedure **E**.

¹H-NMR (CDCl₃, 500 MHz) δ : 7.85 (1H, s), 7.84 (1H, d, J = 8.6 Hz), 7.22 (1H, d, J = 2.6 Hz), 7.02 (1H, dd, J = 8.6, 2.6 Hz), 3.90 (3H, s), 0.55 (6H, s). ¹³C-NMR (125 MHz, CHCl₃) δ : 164.8, 163.8, 161.1,

156.3, 143.6, 142.6, 137.6, 136.5, 135.9, 127.6, 125.2, 123.8, 118.2, 116.2, 55.5, 18.3, -4.4 HRMS (MALDI-TOF) calcd for C₁₈H₁₆O₄Si: 324.0812 ([M+H]⁺), found 324.0820 ([M+H]⁺). m.p. >250 °C (recrystallized from CHCl₃, an orange needle).

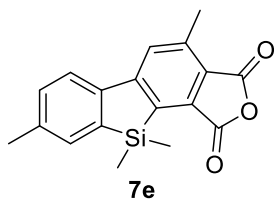


7d (an orange solid, 24%, 7.3 mg, 0.0237 mmol) was prepared from **5d** (21.8 mg, 0.100 mmol) by a general procedure **E**.

¹H-NMR (CDCl₃, 400 MHz) δ : 7.98 (1H, s), 7.75 (1H, s), 7.62 (1H, d, J = 7.3 Hz), 7.26 (1H, d, J = 7.3 Hz), 2.78 (3H, s), 2.47 (3H, s), 0.53 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ : 164.7, 163.8, 163.8, 162.2, 156.0, 156.0,

147.0, 142.5, 138.1, 136.5, 134.6, 132.1, 128.7, 126.6, 115.5, 108.5, 55.5, 18.4, -4.0. HRMS (MALDI-TOF) calcd for C₁₈H₁₇O₃Si: 309.0942 ([M+H]⁺), found 309.0940 ([M +

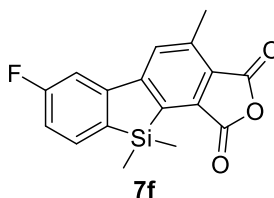
H]⁺). m.p. 249.0-250.0 °C (recrystallized from CHCl₃, a yellow column).



7e (an orange solid, 21%, 6.5 mg, 0.0211 mmol) was prepared from **5e** (21.8 mg, 0.100 mmol) by a general procedure **E**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.93 (1H, s), 7.80 (1H, d, *J* = 7.8 Hz), 7.54 (1H, d, *J* = 1.4 Hz), 7.33 (1H, dd, *J* = 7.8, 1.4 Hz), 2.77 (3H, s), 2.43 (3H, s), 0.54 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ: 164.7, 163.8, 156.4, 142.5,

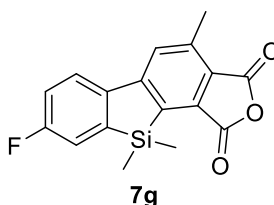
142.4, 141.4, 139.9, 136.5, 136.5, 134.1, 131.4, 128.2, 126.0, 122.2, 21.5, 18.3, -4.4. HRMS (MALDI-TOF) calcd for C₁₈H₁₆O₃Si: 308.0863 ([M+H]⁺), found 308.0863 ([M+H]⁺). m.p. >250 °C (recrystallized from CHCl₃, a yellow needle).



7f (a white solid, 39%, 12.2 mg, 0.0391 mmol) was prepared from **5f** (21.8 mg, 0.100 mmol) by a general procedure **E**.

¹H-NMR (CDCl₃, 500 MHz) δ: 7.91 (1H, s), 7.68 (1H, dd, *J* = 7.9, 5.5 Hz), 7.58 (1H, dd, *J* = 10.5, 2.3 Hz), 7.14 (1H, ddd, *J* = 9.0, 7.9, 2.3 Hz), 2.79 (3H, s), 0.55 (6H, s). ¹³C-NMR (125 MHz, CHCl₃) δ: 165.1 (*J* = 247 Hz),

164.4, 163.5, 154.9, 147.6 (*J* = 8 Hz), 142.7, 137.5, 136.5, 135.4, 135.0 (*J* = 8 Hz), 128.9, 126.9, 116.8 (*J* = 20 Hz), 109.7 (*J* = 21 Hz), 18.3, -4.4. ¹⁹F-NMR (376 MHz, CHCl₃) δ: -105.1 (ddd, *J* = 10.5, 9.0, 5.5). HRMS (MALDI-TOF) calcd for C₁₇H₁₄O₃FSi: 313.0691 ([M+H]⁺), found 313.0689 ([M+H]⁺). m.p. 249.0-250.0 °C (recrystallized from CHCl₃, a white column).

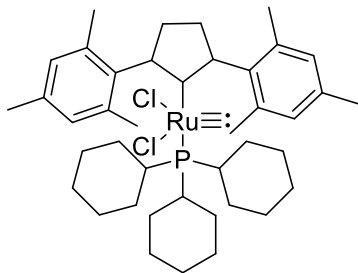


7g (a white solid, 38%, 11.8 mg, 0.0379 mmol) was prepared from **5g** (21.8 mg, 0.100 mmol) by a general procedure **E**.

¹H-NMR (CDCl₃, 400 MHz) δ: 7.91 (1H, s), 7.89 (1H, dd, *J* = 8.7, 4.6 Hz), 7.39 (1H, dd, *J* = 7.6, 2.5 Hz), 7.19 (1H, ddd, *J* = 8.7, 8.7, 2.5 Hz), 2.78 (3H, s), 0.56 (6H, s). ¹³C-NMR (100 MHz, CHCl₃) δ: 164.3 (*J* = 201 Hz),

164.6, 163.6, 155.3, 144.5 (*J* = 5 Hz), 142.8, 140.8, 136.6, 136.2, 128.4, 126.1, 124.1 (*J* = 6 Hz), 120.0 (*J* = 16 Hz), 117.6 (*J* = 18 Hz), 18.3, -4.5. ¹⁹F-NMR (376 MHz, CHCl₃) δ: -106.5 (ddd, *J* = 8.7, 7.6, 4.6 Hz). HRMS (MALDI-TOF) calcd for C₁₇H₁₄O₃FSi: 313.0691 ([M + H]⁺), found 313.0700 ([M + H]⁺). m.p. >250 °C (recrystallized from CHCl₃, a yellow column).

8. Synthesis of carbide complex

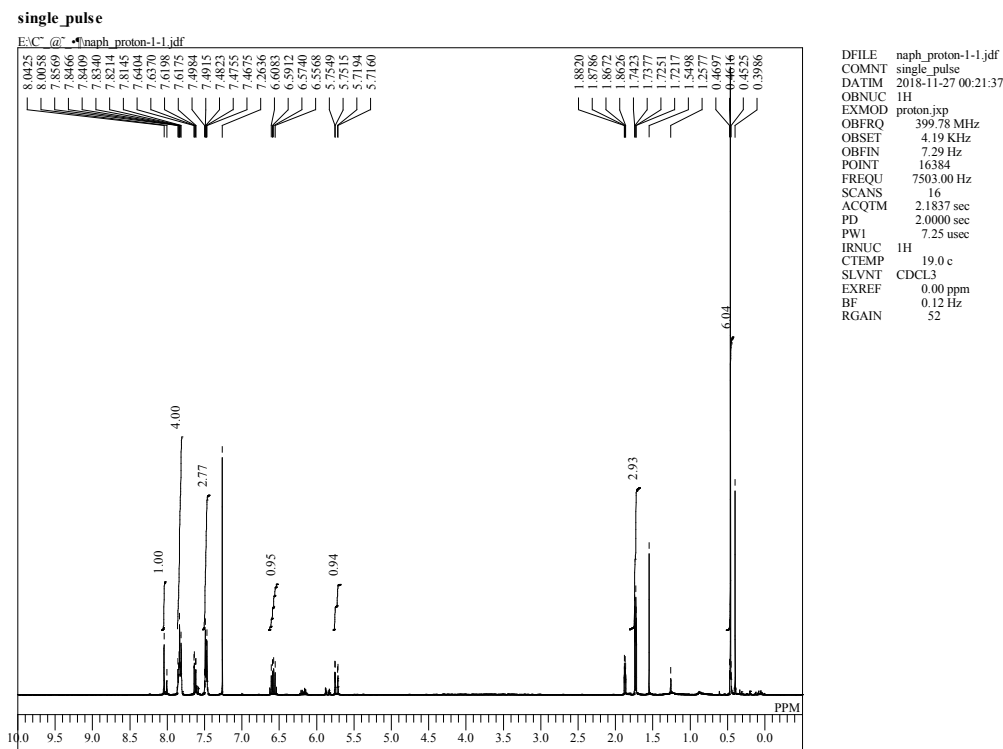


To a solution of **1b** (63.7 mg, 0.300 mmol) in CHCl_3 (2 mL, 0.05 M) was added Grubbs II (84.9 mg, 0.100 mmol) and the mixture was heated for 2 h at 45 °C. The solvent was evaporated and the residue was subjected to column chromatography (*n*-hexane/AcOEt = 20/1) on neutral flash silica gel 60N to give carbide complex (an orange solid, 34%, 26.4 mg, 0.0343 mmol).

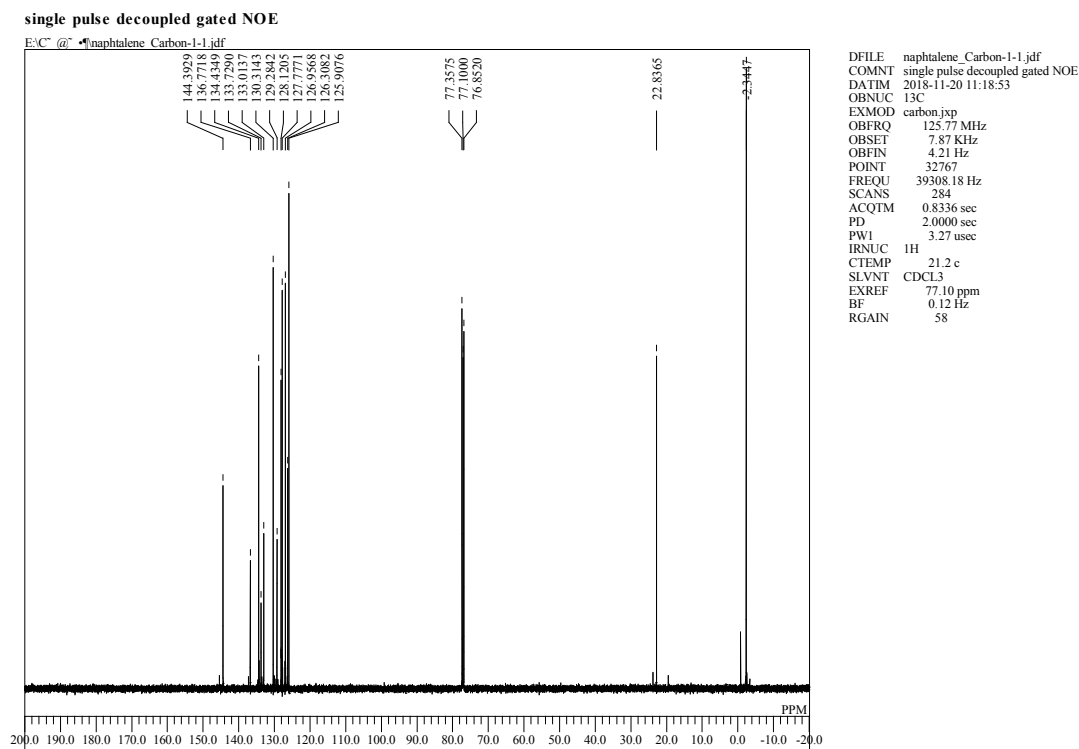
^1H -NMR (CDCl_3 , 400 MHz) δ : 6.95 (2H, s), 6.89 (2H, s), 4.10-4.04 (4H, m), 2.54 (6H, s), 2.49 (6H, s), 2.34-2.29 (3H, m), 2.29 (3H, s), 2.24 (3H, s), 1.88-1.86 (6H, m), 1.67-1.60 (6H, m), 1.19-1.12 (12H, m).

9. NMR chart

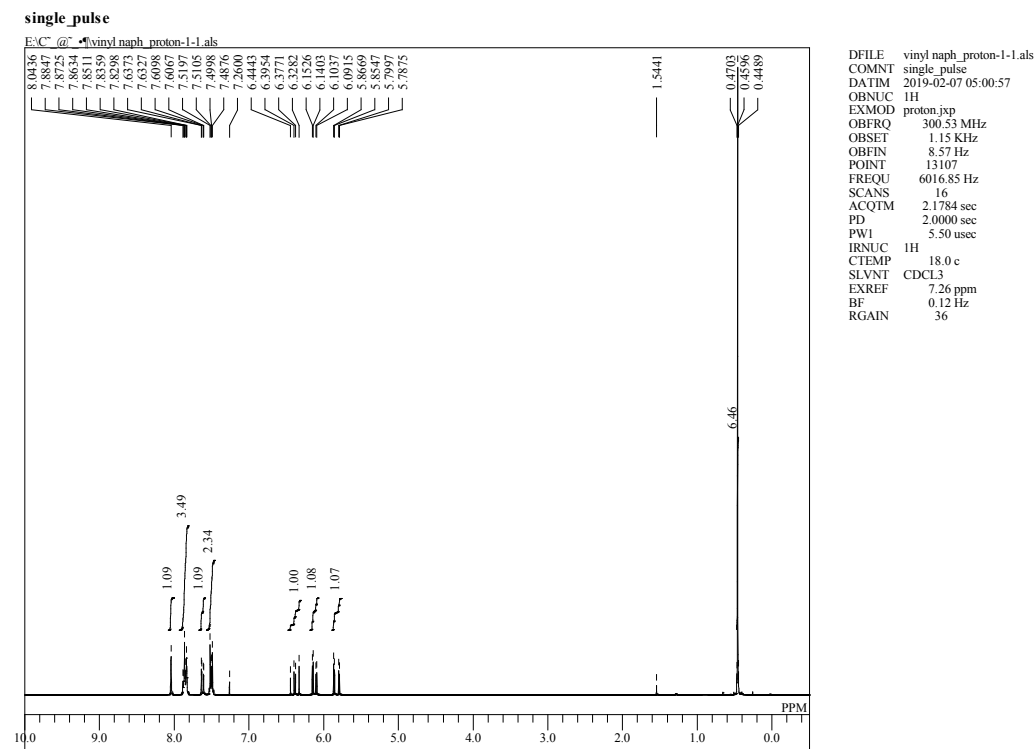
1b



1b

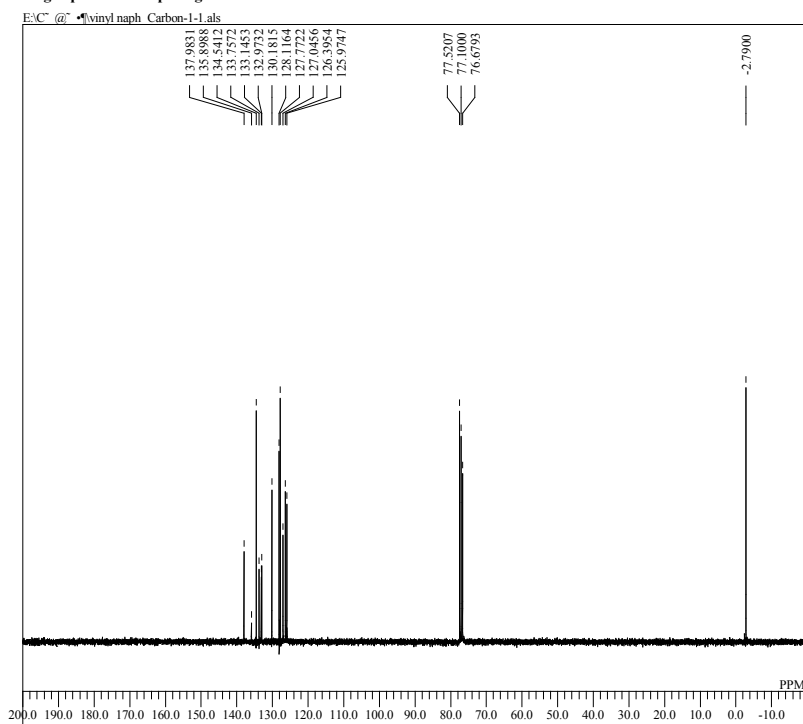


1a



1a

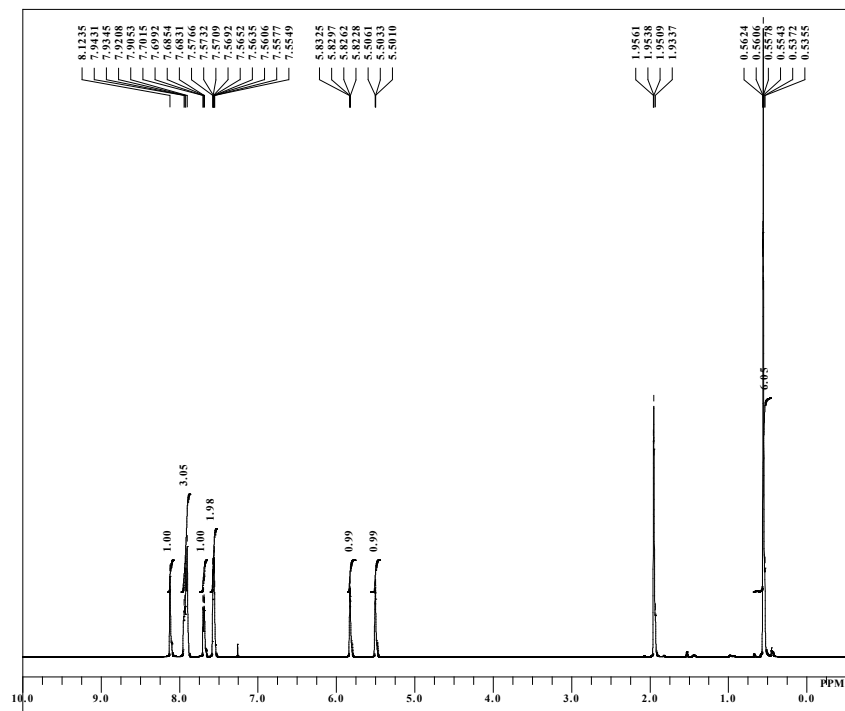
single pulse decoupled gated NOE



DFILE vinyl naph Carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2019-02-07 08:09:12
OBNUC 13C
EXMOD carbon.jsp
OBFREQ 75.57 MHz
OBSET 5.79 KHz
OBFIN 1.08 Hz
POINT 13107
FREQU 18939.39 Hz
SCANS 1024
ACQTM 0.6921 sec
PD 2.0000 sec
PW1 3.73 usec
IRNUC 1H
CTEMP 18.2 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

1c

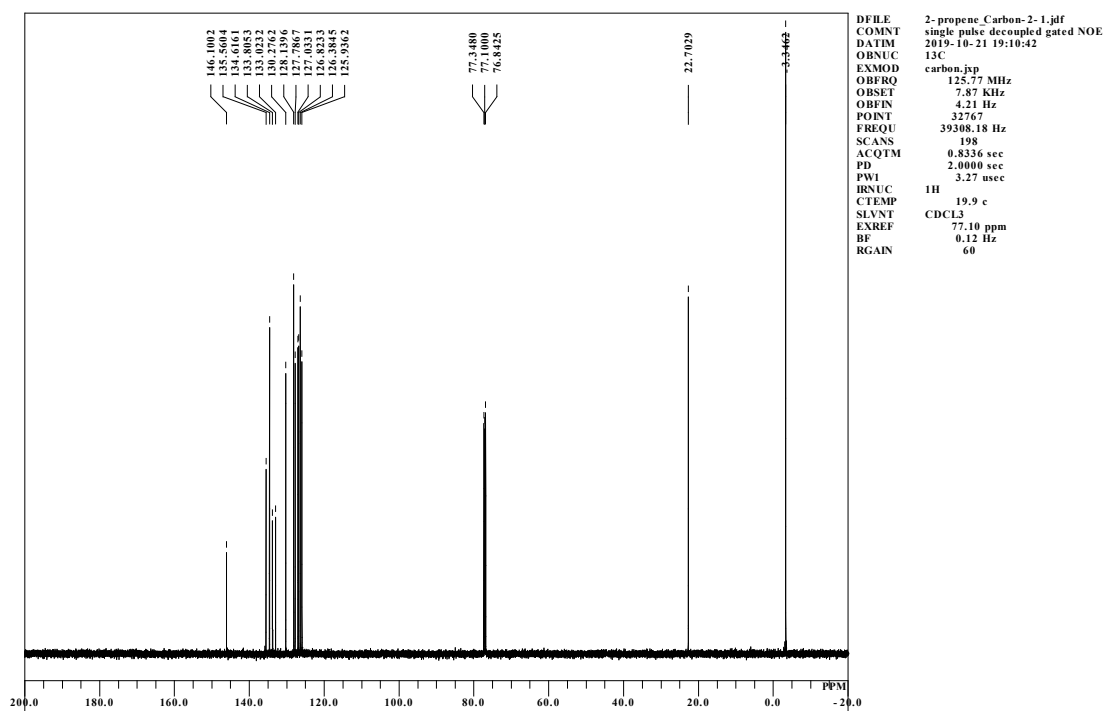
single_pulse



DFILE 2-propene_proton-2-1.jdf
COMNT single_pulse
DATIM 2019-10-21 19:07:07
OBNUC 1H
EXMOD proton.jsp
OBFREQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32767
FREQU 9384.38 Hz
SCANS 16
ACQTM 3.4918 sec
PD 2.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 24

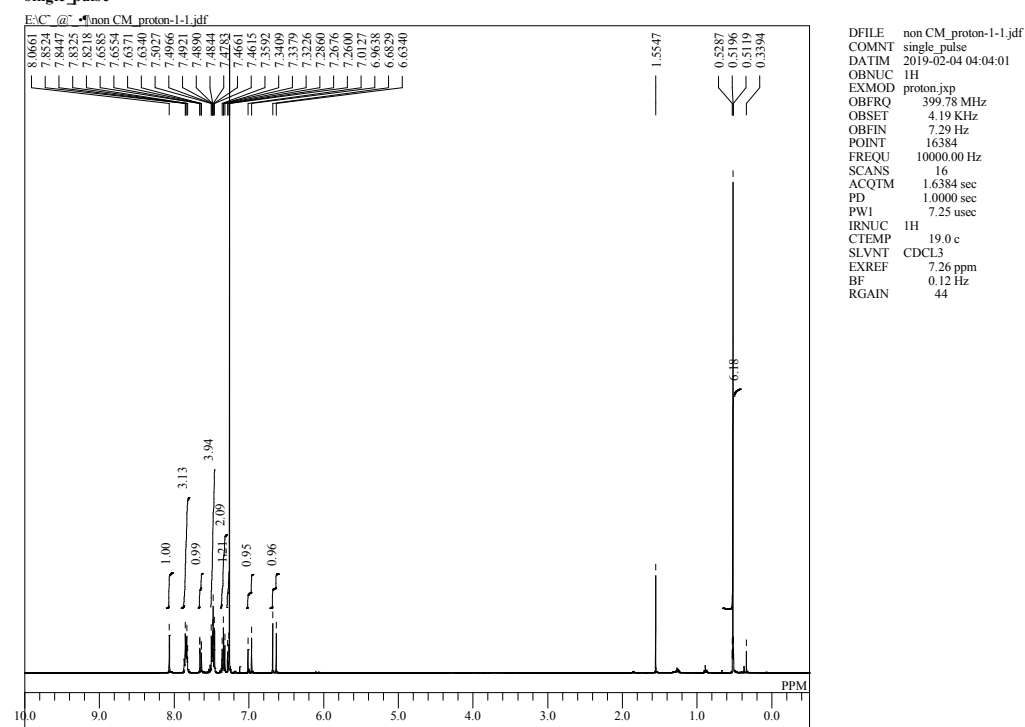
1c

single pulse decoupled gated NOE

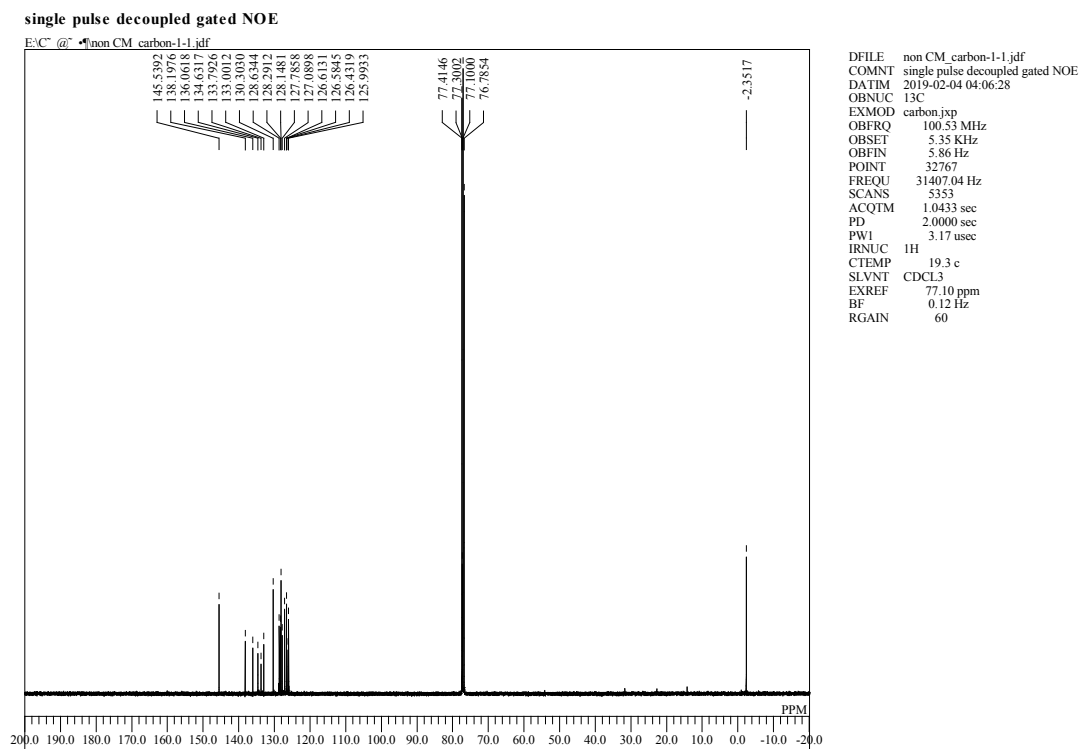


2a

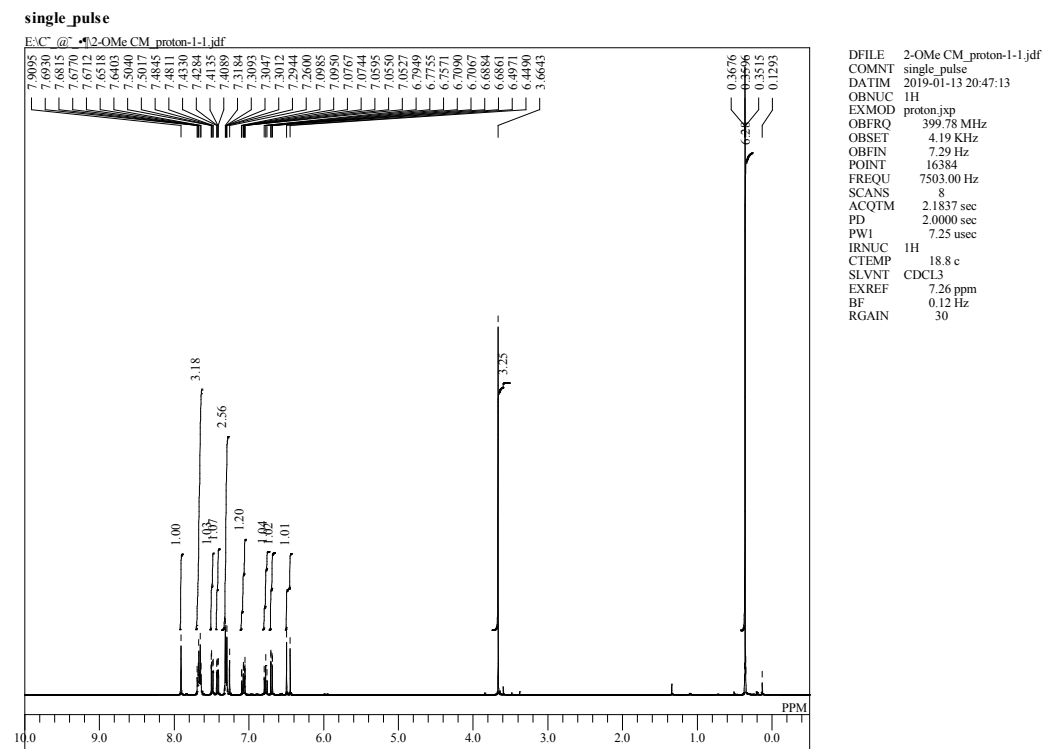
single_pulse



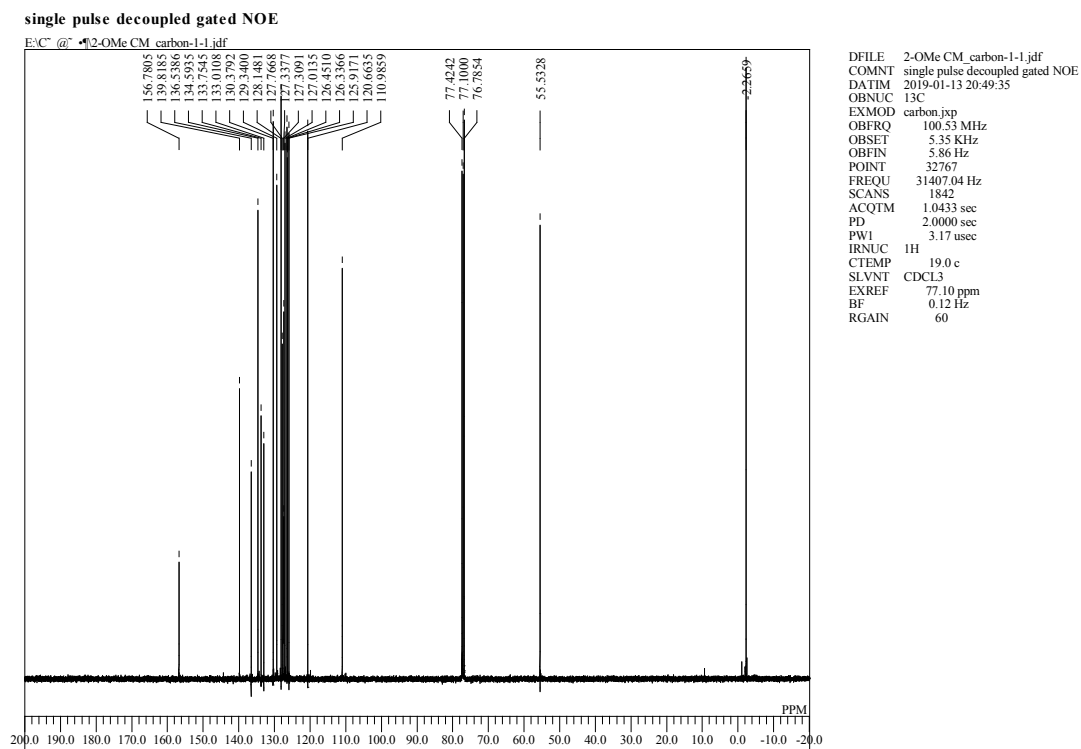
2a



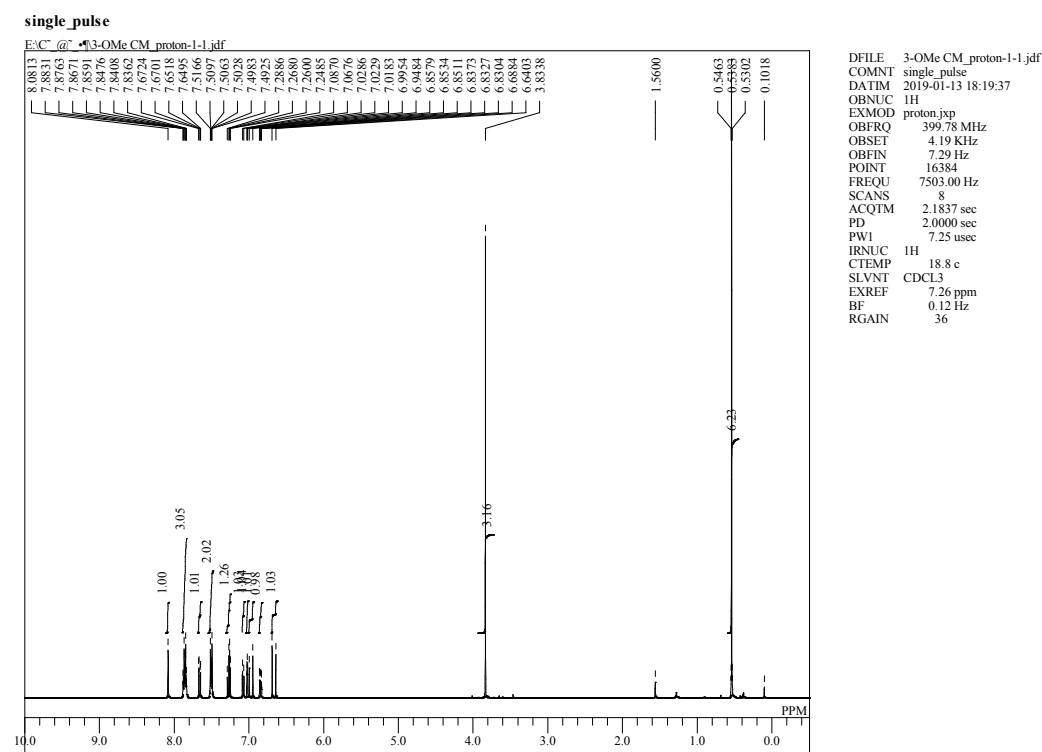
2b



2b

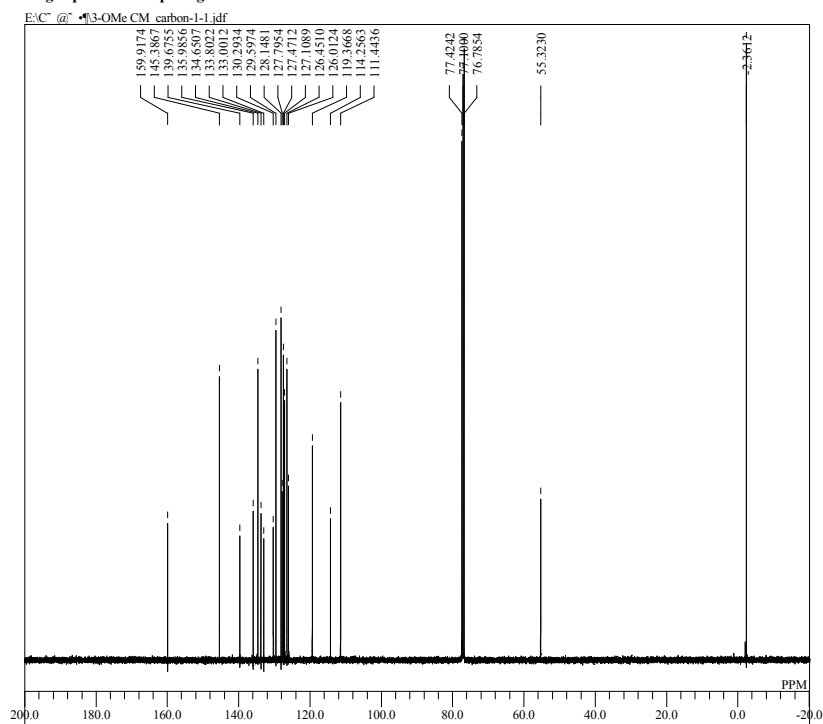


2c



2c

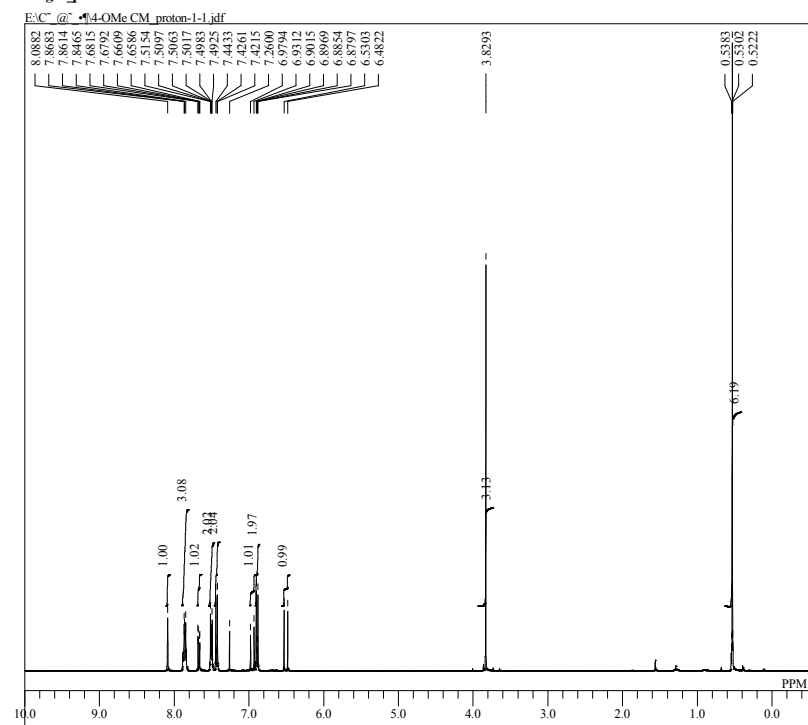
single pulse decoupled gated NOE



DFILE 3-OMe CM_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-13 18:22:01
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 1486
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

2d

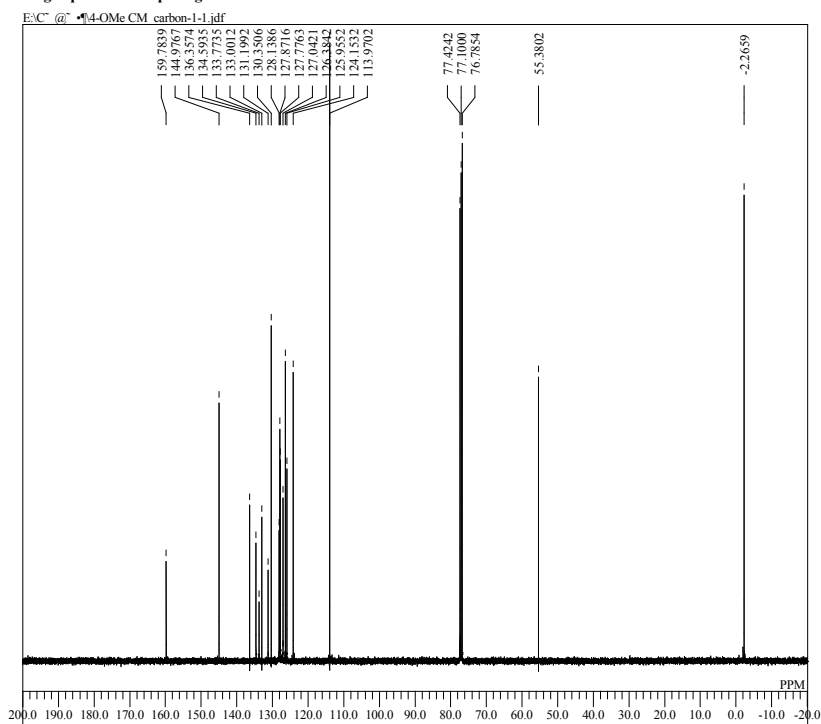
single pulse



DFILE 4-OMe CM_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-01-13 19:43:00
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 8
 ACQTM 2.1837 sec
 PD 2.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 34

2d

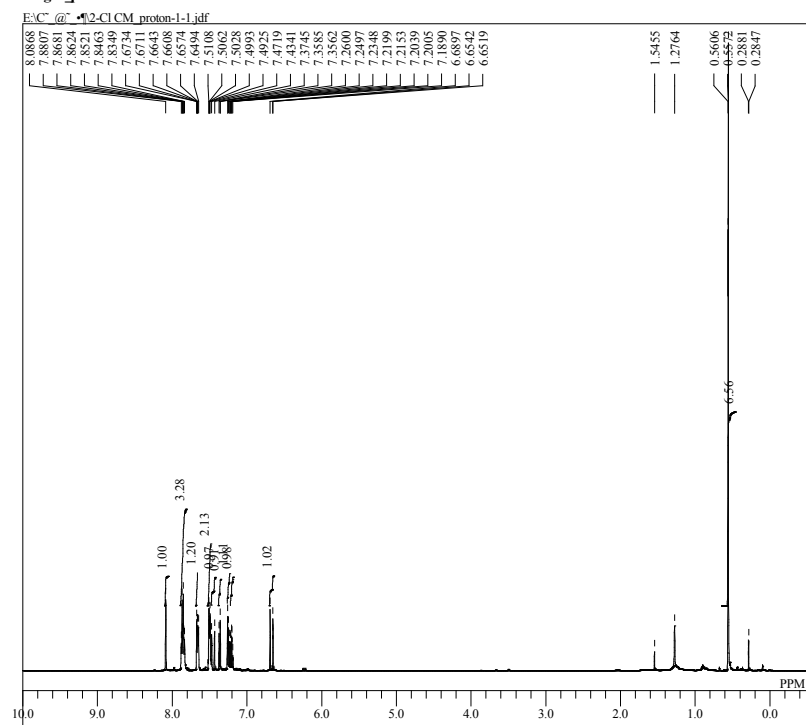
single pulse decoupled gated NOE



DFILE 4-Ome CM_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-13 19:45:21
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.33 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 1112
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC ¹H
 CTEMP 19.2 c
 SLVNT CDCL₃
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

2e

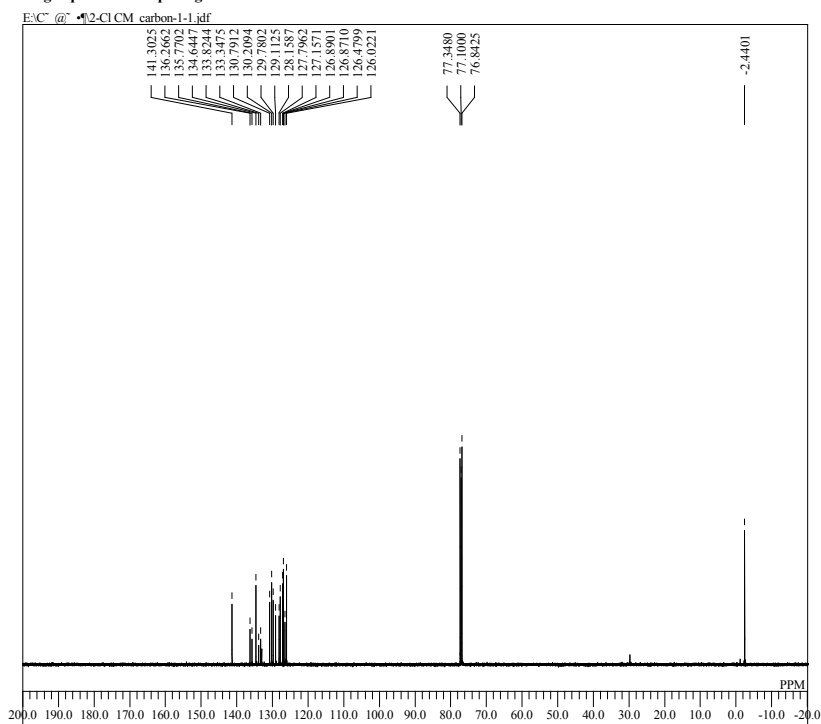
single pulse



DFILE 2-Cl CM_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-01-23 23:53:09
 OBNUC ¹H
 EXMOD proton.jxp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 16
 ACQTM 1.7459 sec
 PD 1.0000 sec
 PW1 6.50 usec
 IRNUC ¹H
 CTEMP 20.1 c
 SLVNT CDCL₃
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

2e

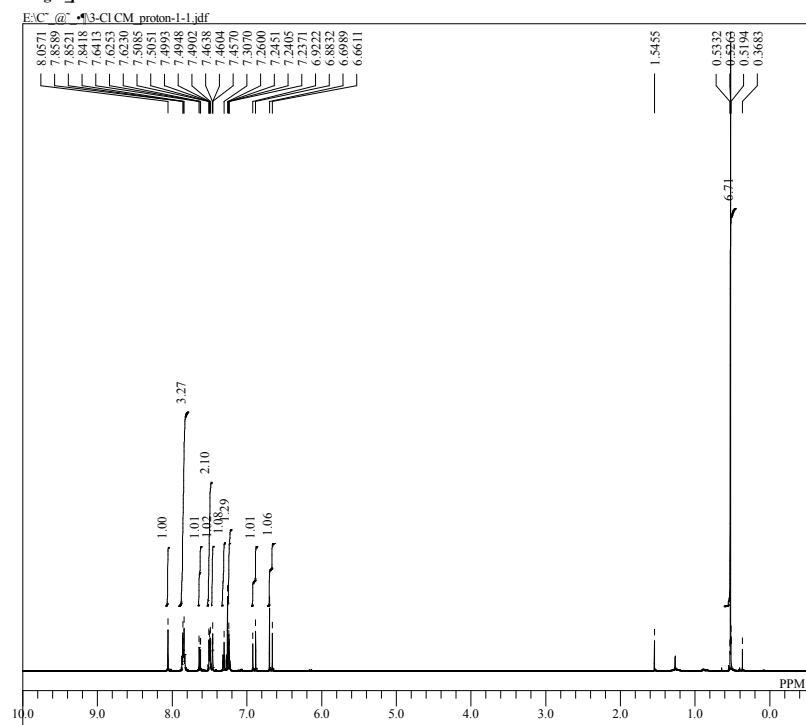
single pulse decoupled gated NOE



DFILE 2-Cl CM carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-23 23:56:22
 OBNUC ¹³C
 EXMOD carbon.jxp
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32767
 FREQU 39308.18 Hz
 SCANS 748
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.27 usec
 IRNUC ¹H
 CTEMP 20.4 c
 SLVNT CDCL₃
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

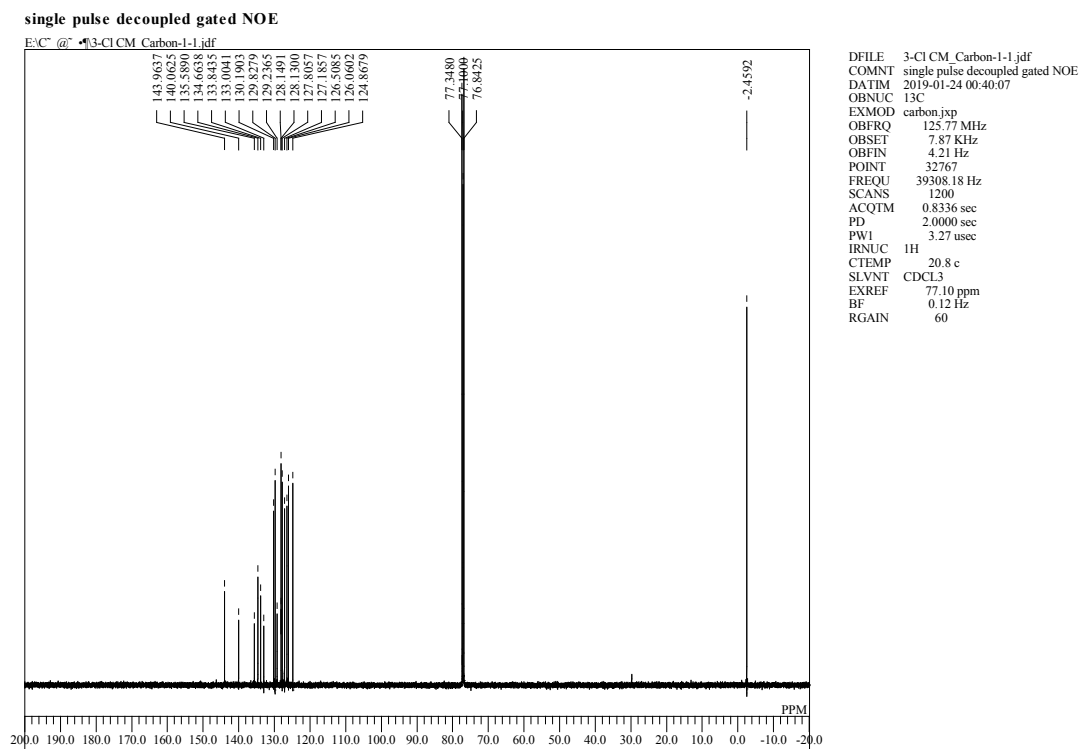
2f

single pulse

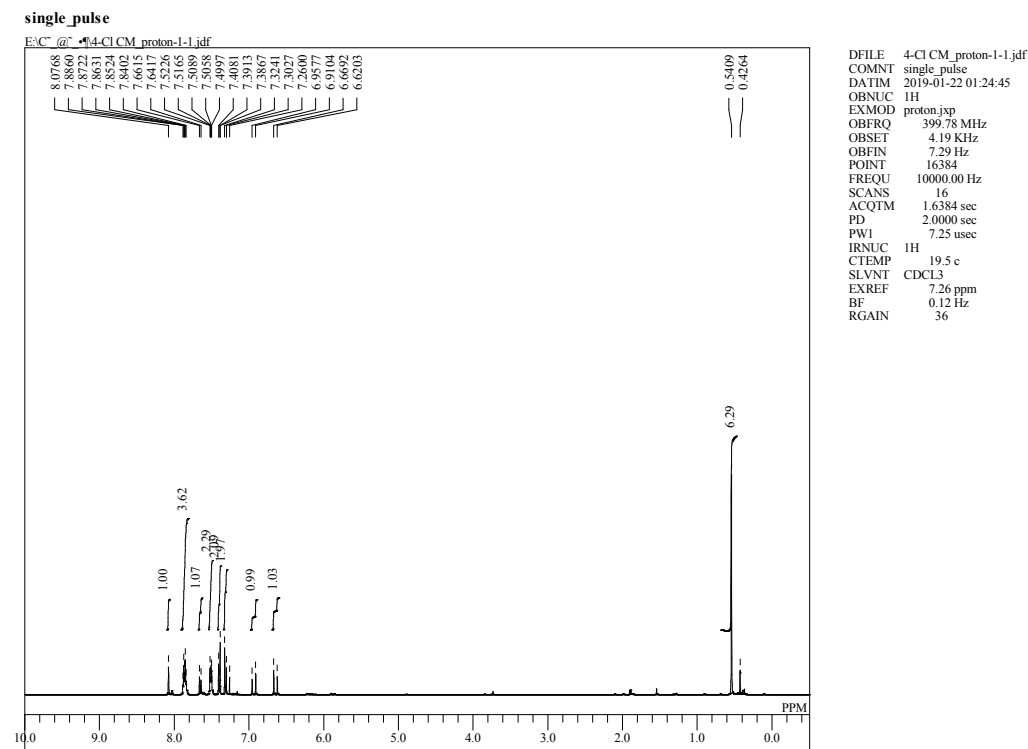


DFILE 3-Cl CM proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-01-24 00:37:04
 OBNUC ¹H
 EXMOD proton.jxp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 16
 ACQTM 1.7459 sec
 PD 1.0000 sec
 PW1 6.50 usec
 IRNUC ¹H
 CTEMP 20.5 c
 SLVNT CDCL₃
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 42

2f

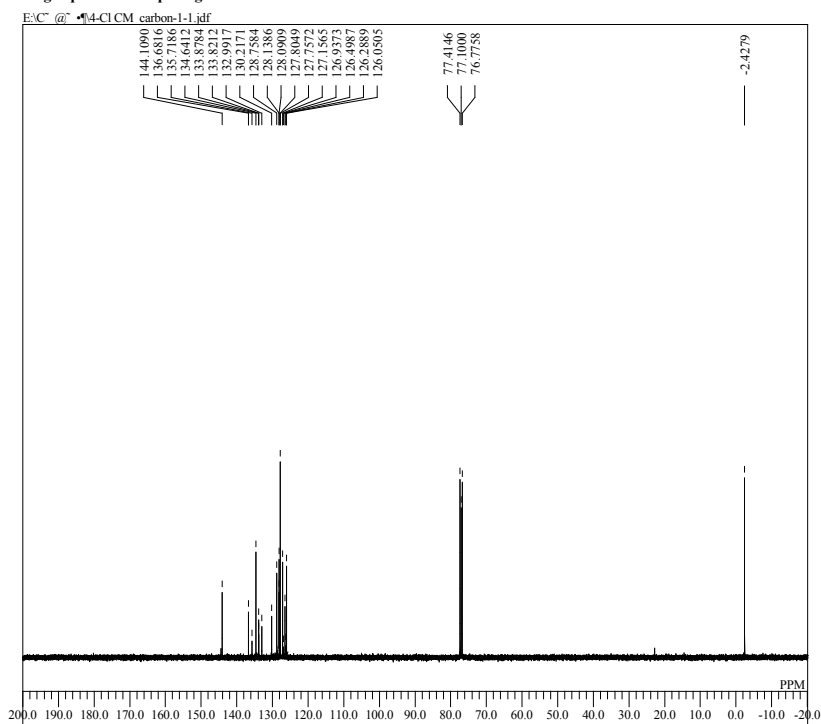


2g



2g

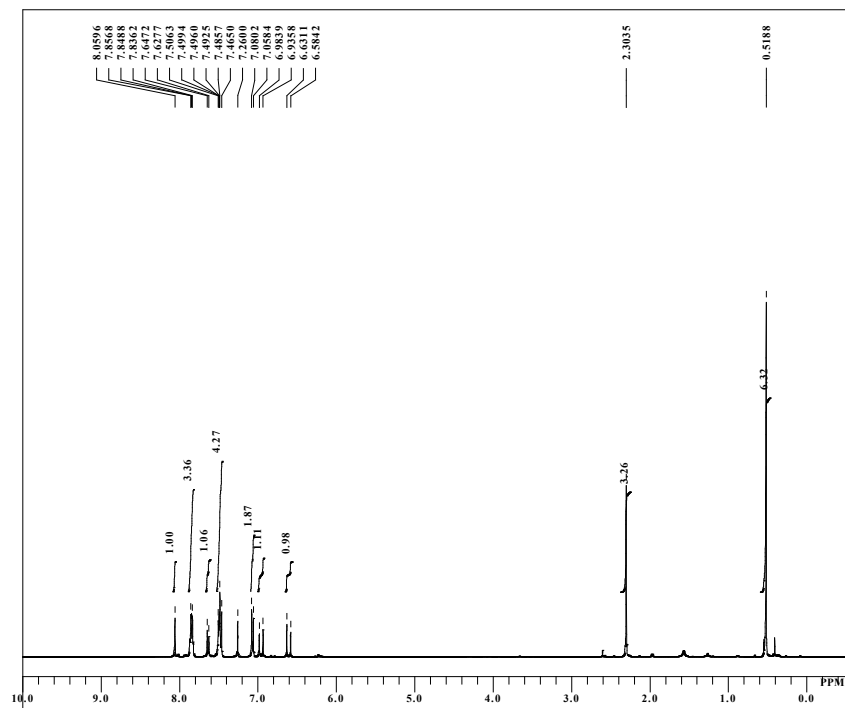
single pulse decoupled gated NOE



DFILE 4-Cl CM carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2019-01-22 01:27:30
OBNUC 13C
EXMOD carbon.jxp
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 193
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

2h

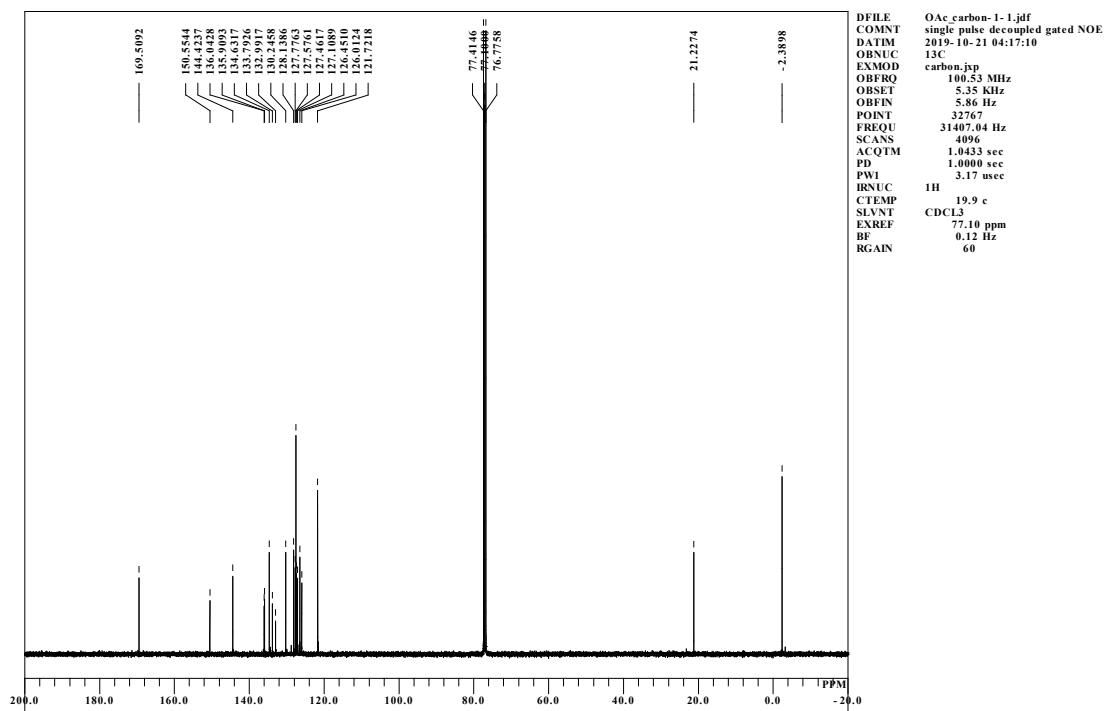
single_pulse



DFILE OAc_proton-1-1.jdf
COMNT single_pulse
DATIM 2019-10-21 00:33:29
OBNUC 1H
EXMOD proton.jxp
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 40

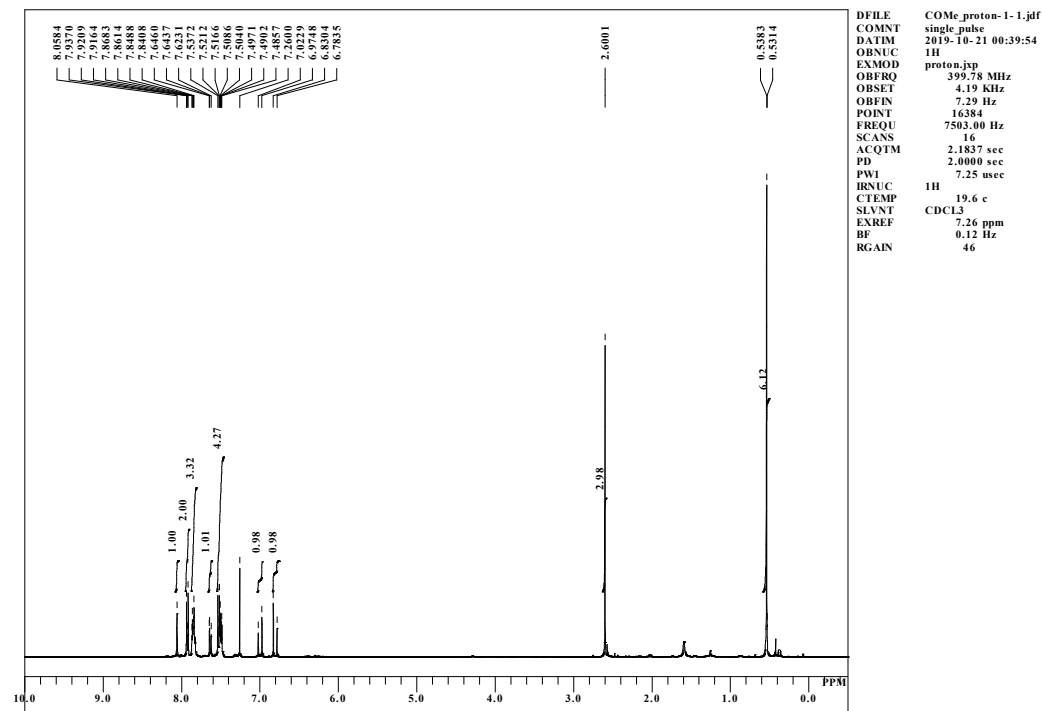
2h

single pulse decoupled gated NOE



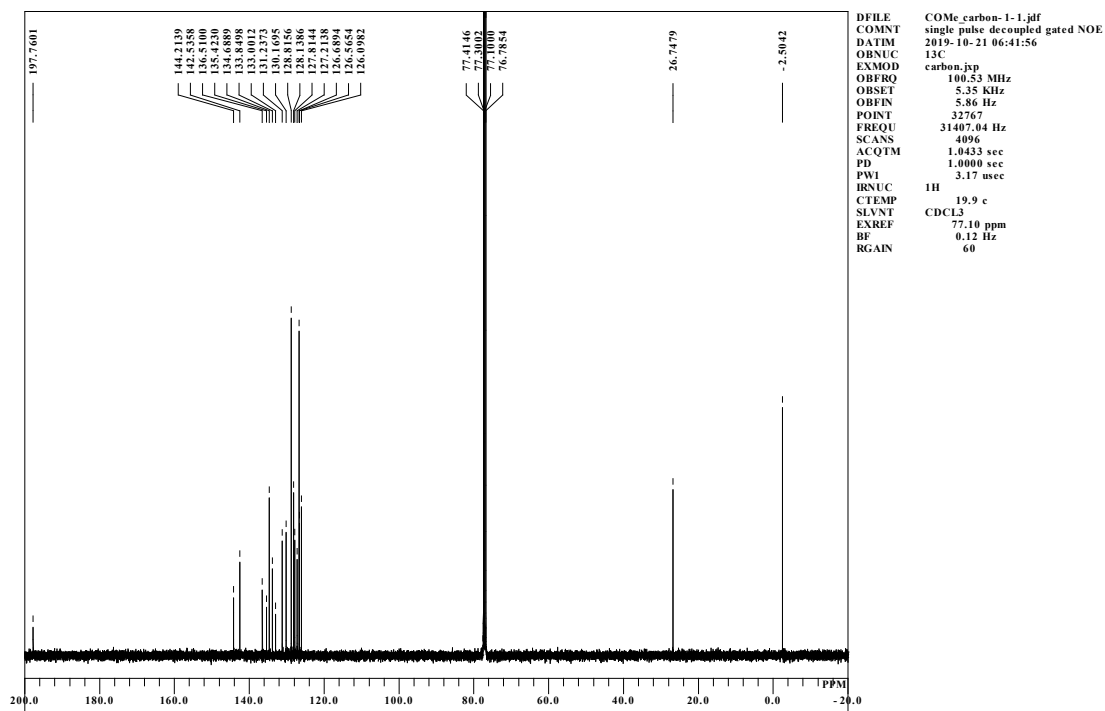
2i

single pulse



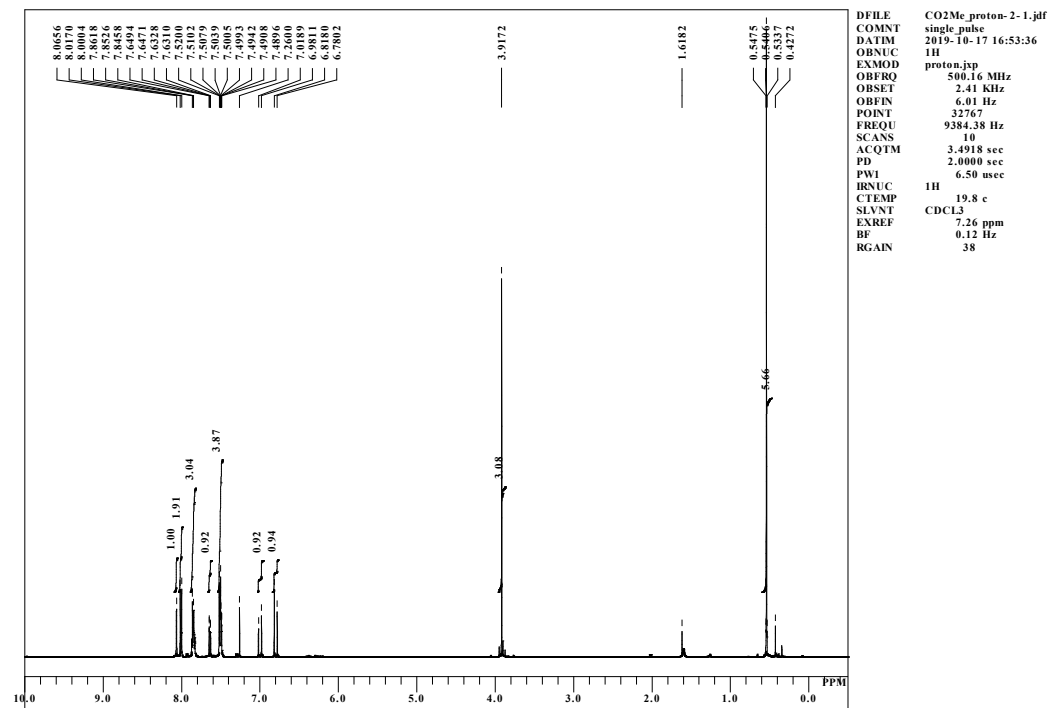
2i

single pulse decoupled gated NOE



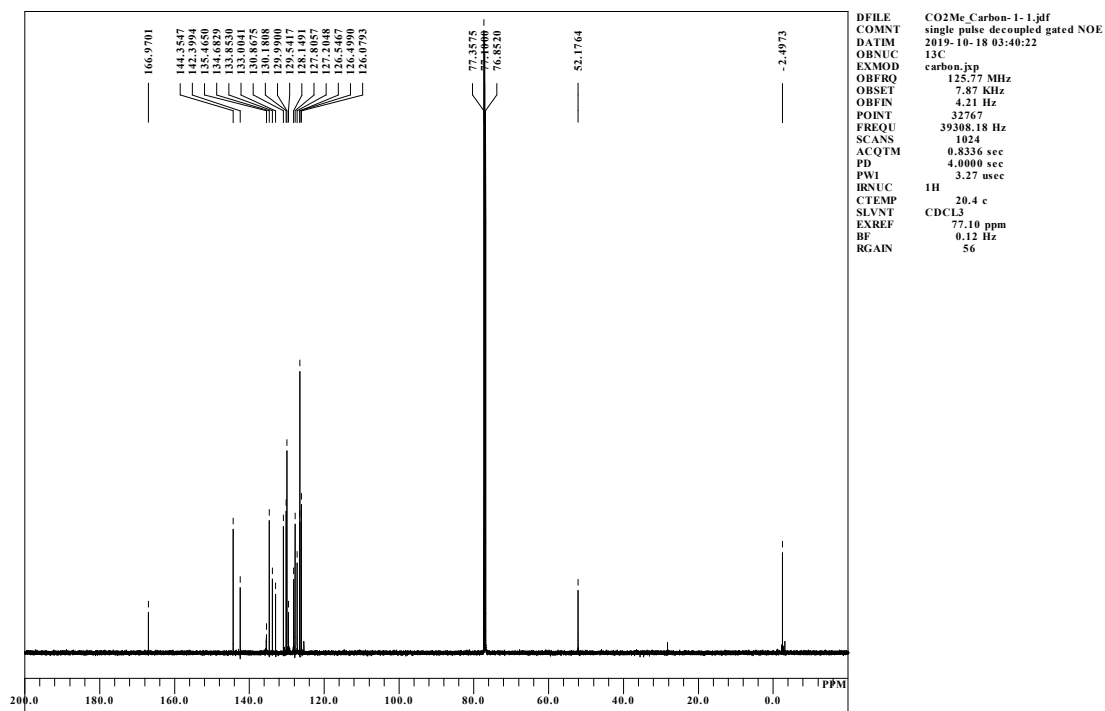
2j

single_pulse



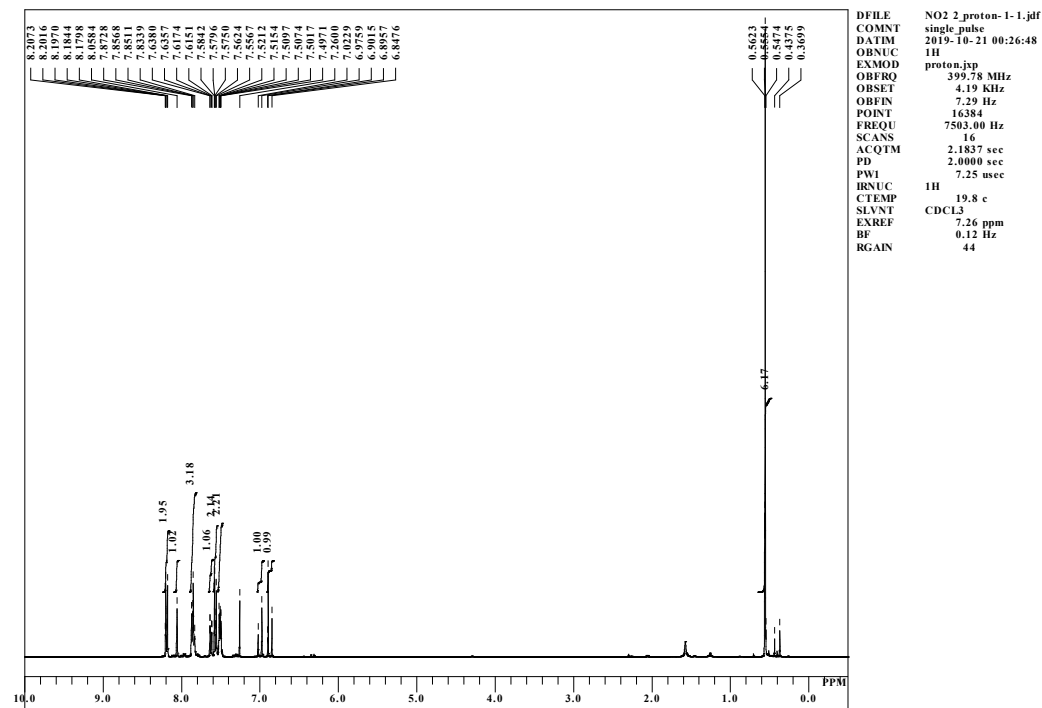
2j

single pulse decoupled gated NOE



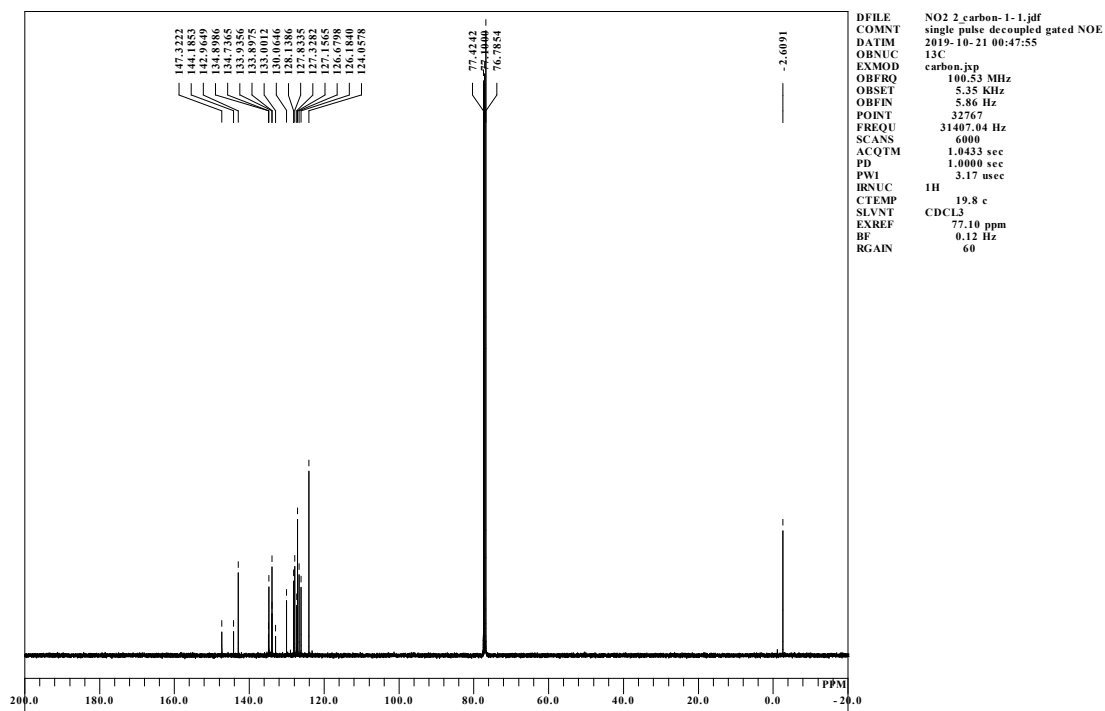
2k

single_pulse



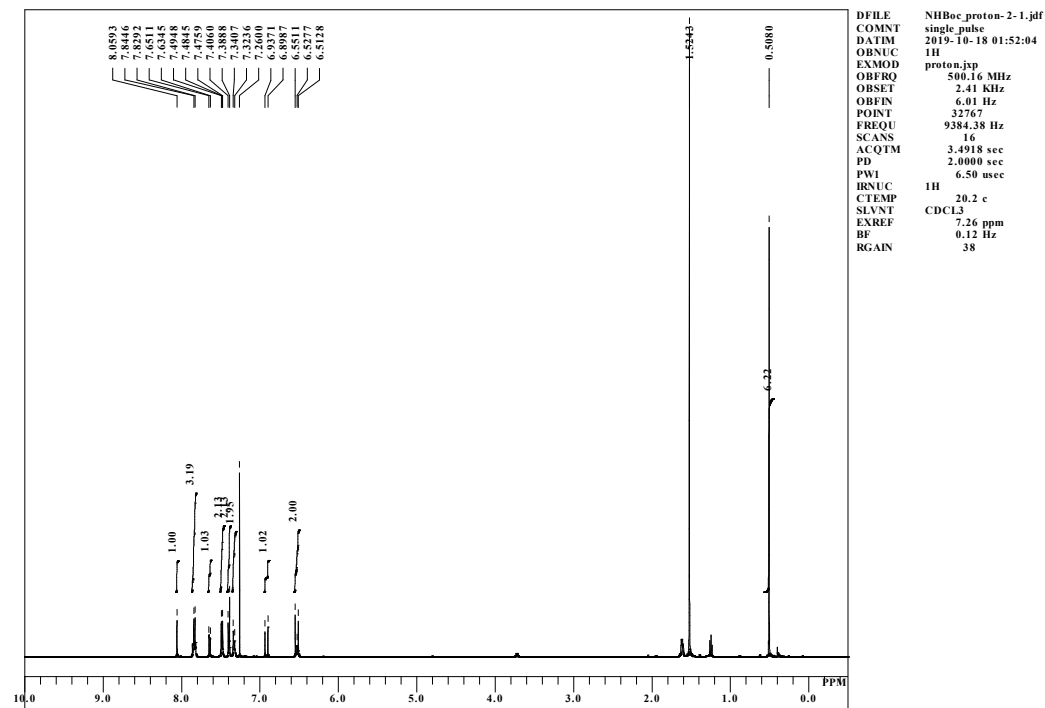
2k

single pulse decoupled gated NOE



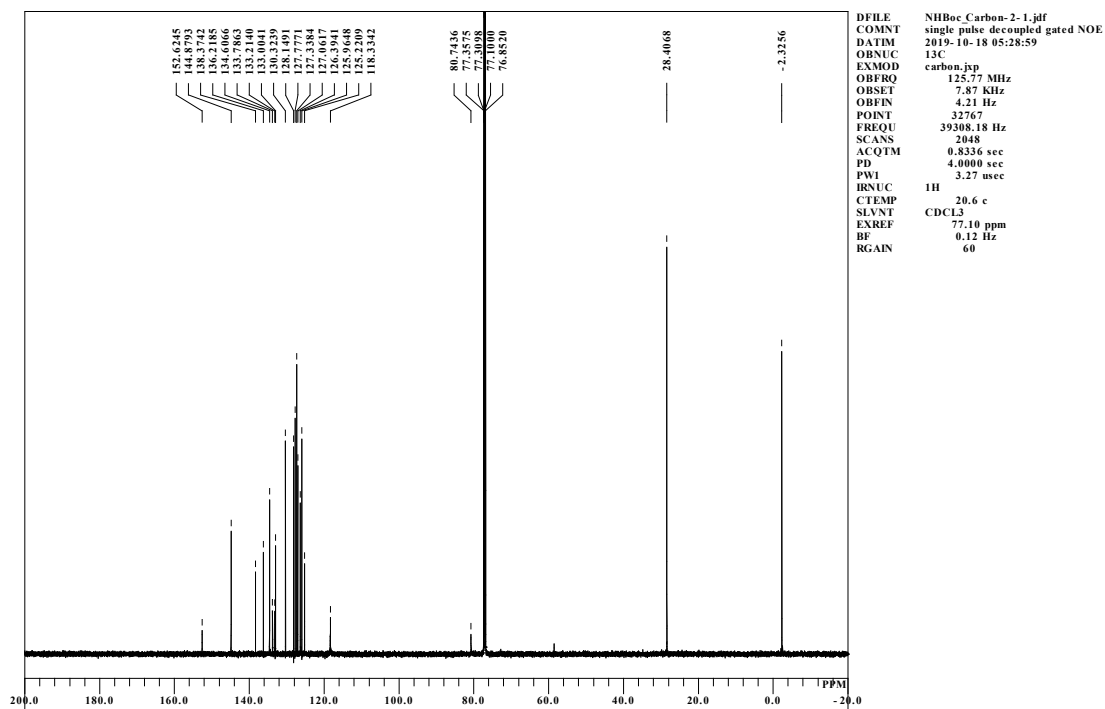
2l

single_pulse



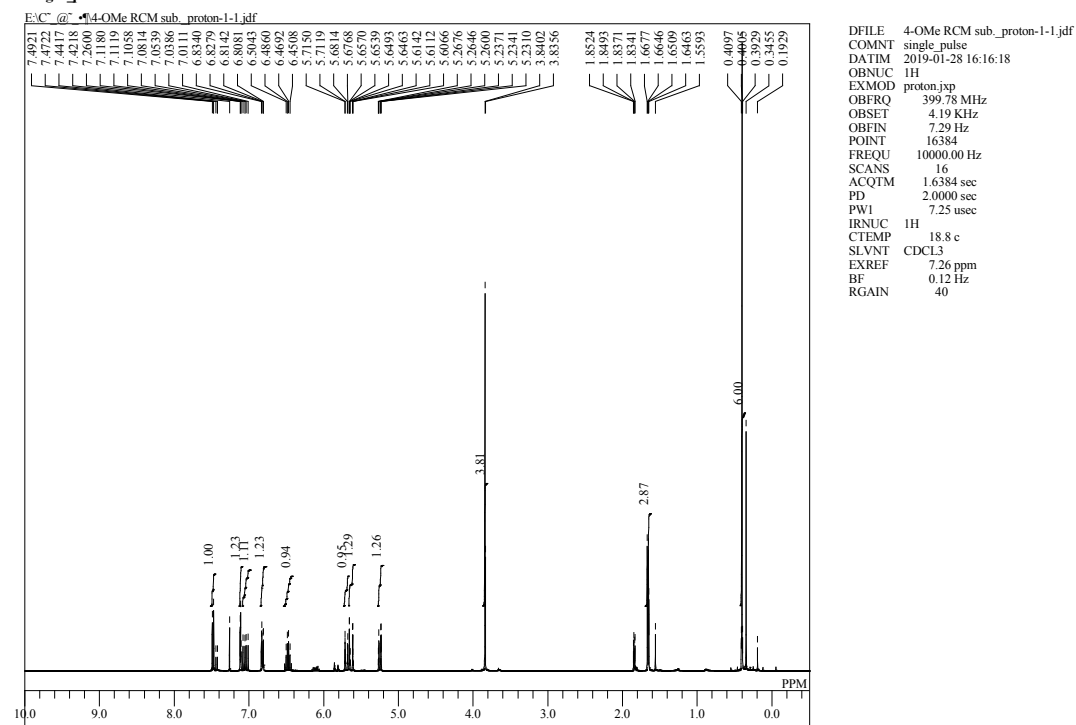
21

single pulse decoupled gated NOE



3a

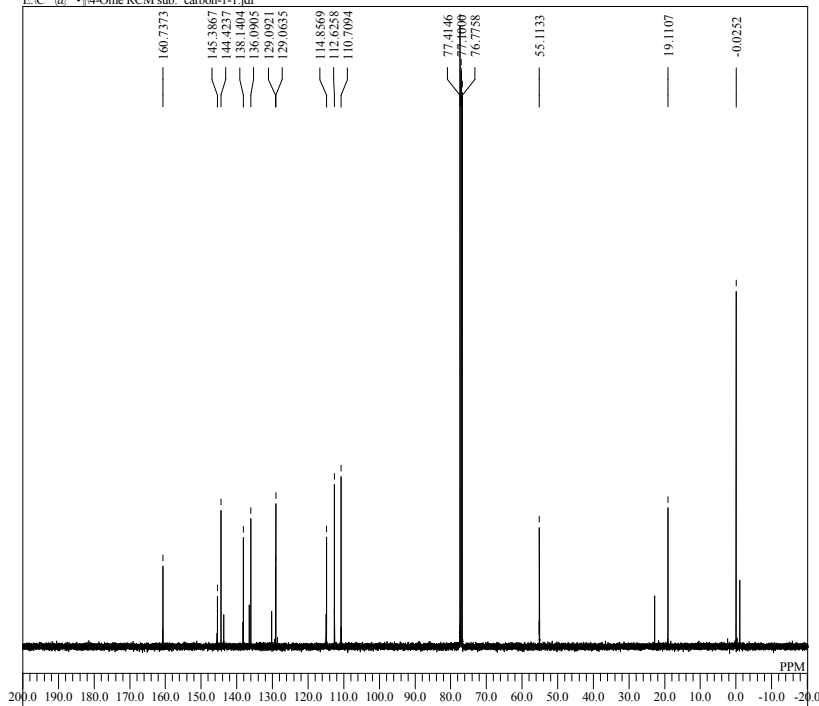
single_pulse



3a

single pulse decoupled gated NOE

E: C¹³ @ 4-Ome RCM sub. carbon-1-1.jdf

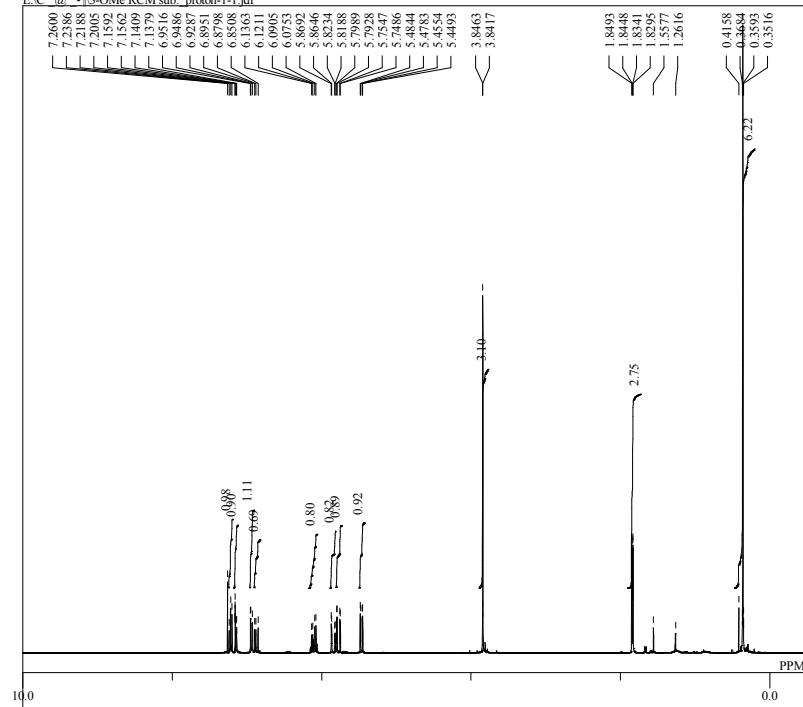


DFILE 4-Ome RCM sub. carbon-1-1.jdf
COMINT single pulse decoupled gated NOE
DATIM 2019-01-29 01:07:35
OBNUC 13C
EXMOD carbon.jpg
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 1304
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

3b

single_pulse

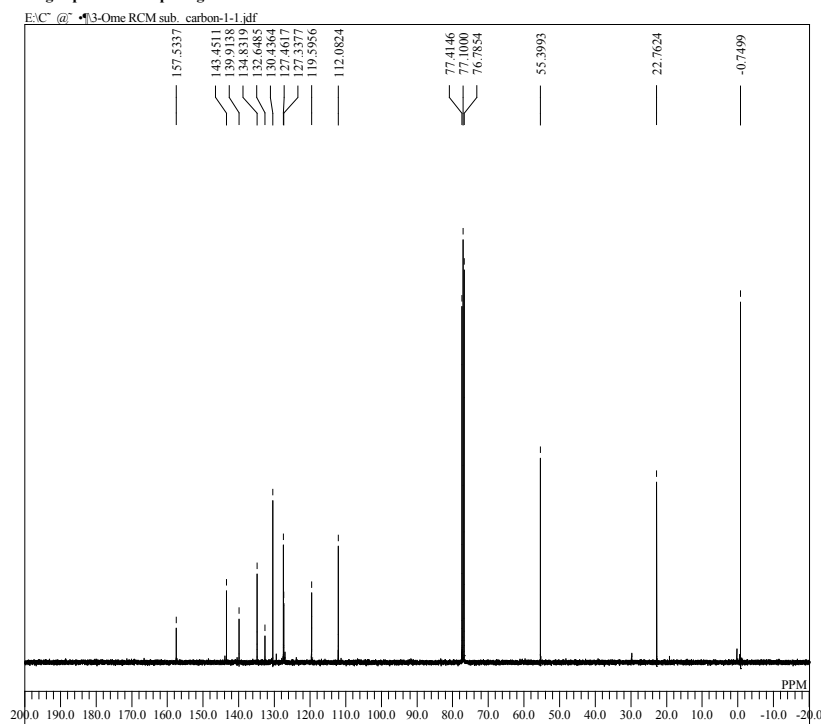
E: C¹³ @ 3-Ome RCM sub. proton-1-1.jdf



DFILE 3-Ome RCM sub. proton-1-1.jdf
COMINT single_pulse
DATIM 2019-01-28 16:24:59
OBNUC 1H
EXMOD proton.jpg
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

3b

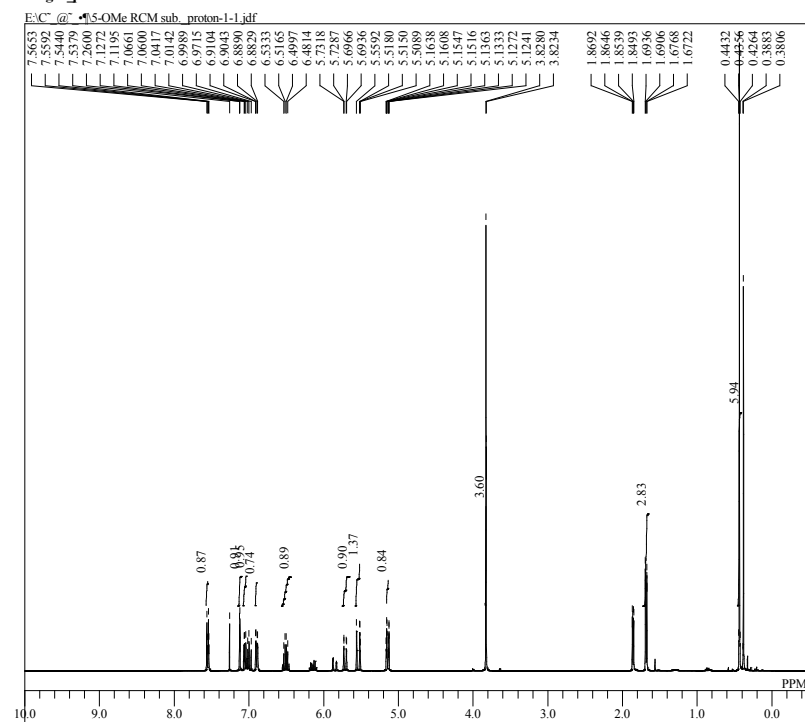
single pulse decoupled gated NOE



DFILE 3-Ome RCM sub_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-28 23:37:30
 OBNUC 13C
 EXMOD carbon.jpg
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 1600
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

3c

single pulse

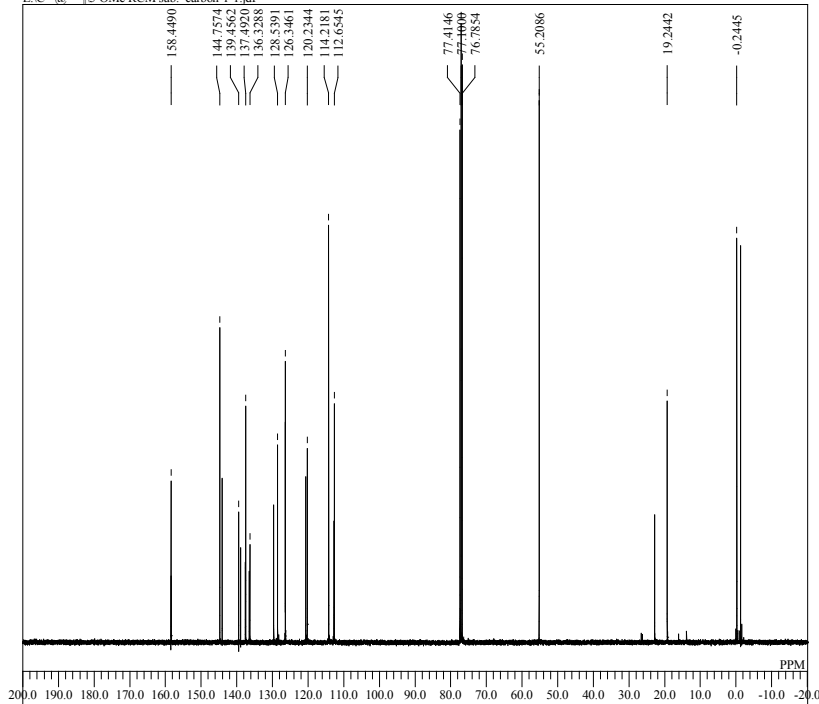


DFILE 5-Ome RCM sub_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-02-04 23:41:18
 OBNUC 1H
 EXMOD proton.jpg
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.0 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 32

3c

single pulse decoupled gated NOE

E:\C\ @\ 5-OMe RCM sub_ carbon-1-1.jdf

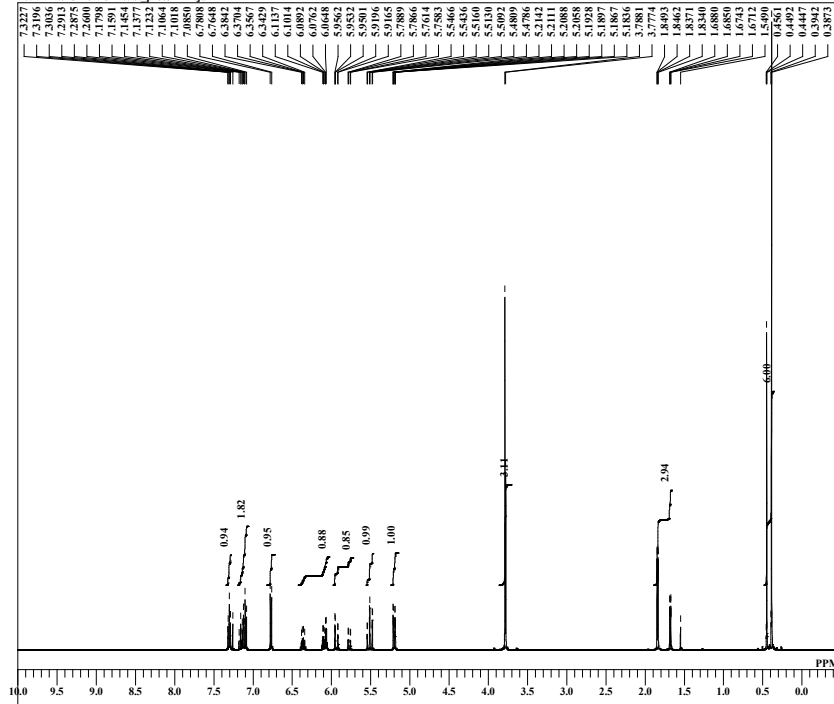


DFILE 5-OMe RCM sub_ carbon-1-1.jdf
COMINT single pulse decoupled gated NOE
DATIM 2019-02-04 23:43:58
OBNUC 13C
EXMOD carbon.jpg
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 2892
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

3d

single pulse

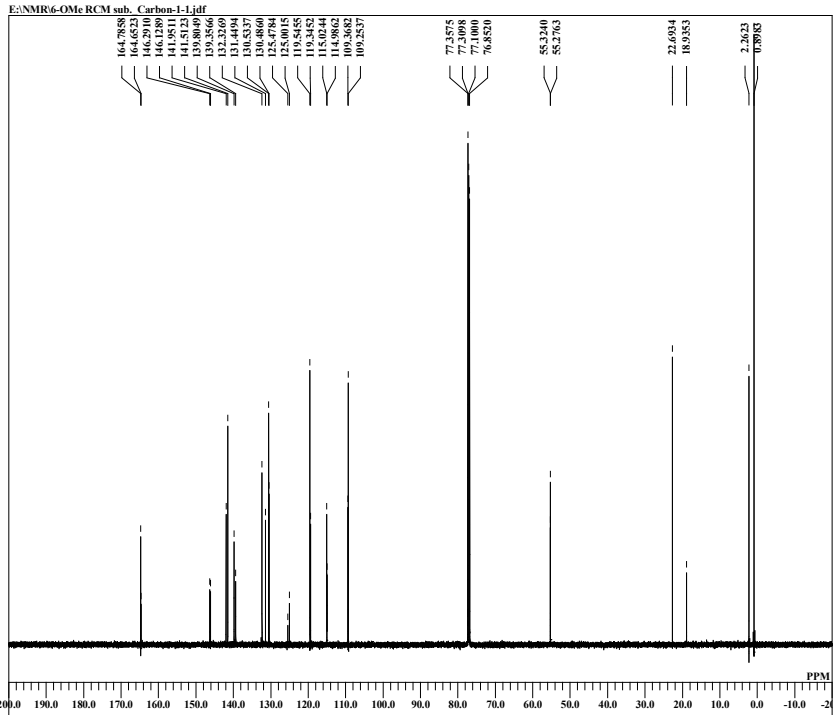
E:\NMR\6-OMe RCM sub_ proton-1-1.jdf



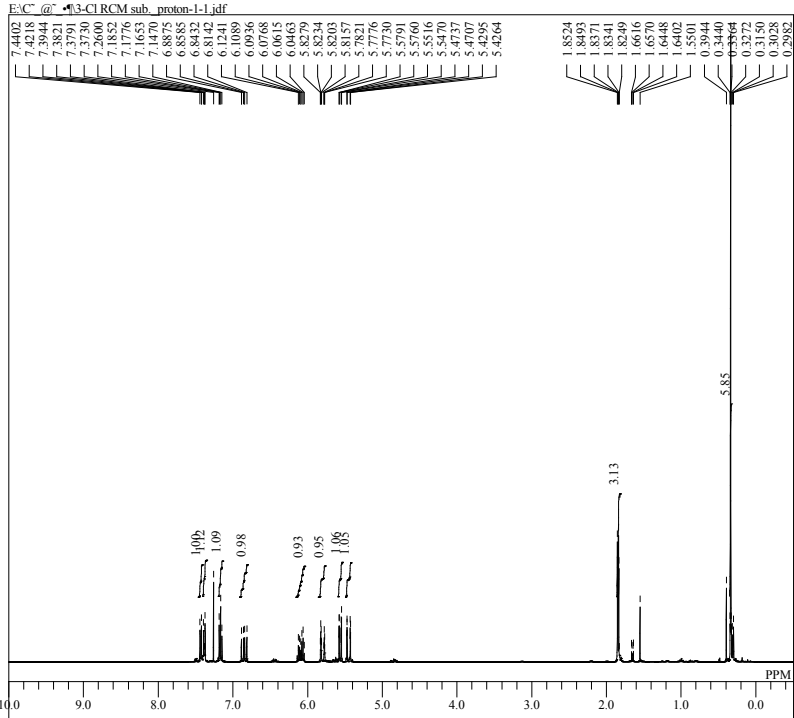
DFILE 6-OMe RCM sub_ proton-1-1.jdf
COMINT single_pulse
DATIM 2019-06-17 12:15:41
OBNUC 1H
EXMOD proton.jpg
OBFREQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 32767
FREQU 12525.05 Hz
SCANS 16
ACQTM 2.6162 sec
PD 1.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 34

3d

single pulse decoupled gated NOE



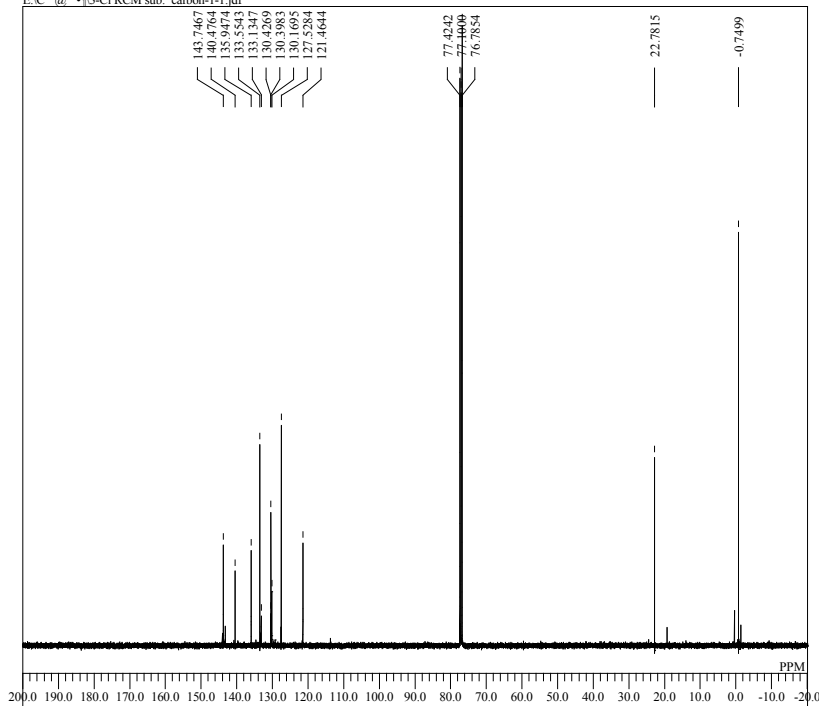
3e

single_pulse

3e

single pulse decoupled gated NOE

F1C' @ 13C-Cl RCM sub_carbon-1-1.jdf

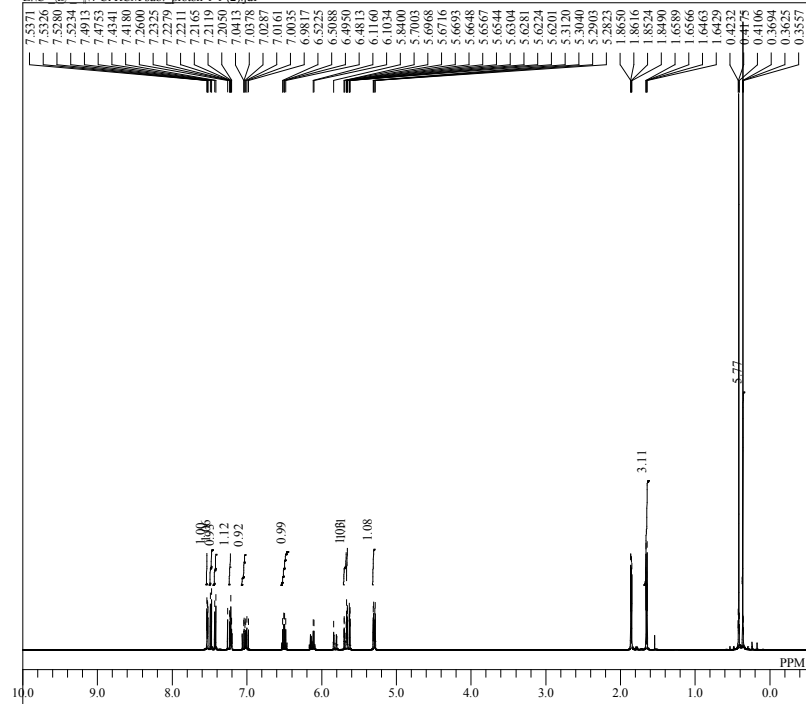


DFILE 3-Cl RCM sub_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-29 22:27:18
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 2048
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

3f

single_pulse

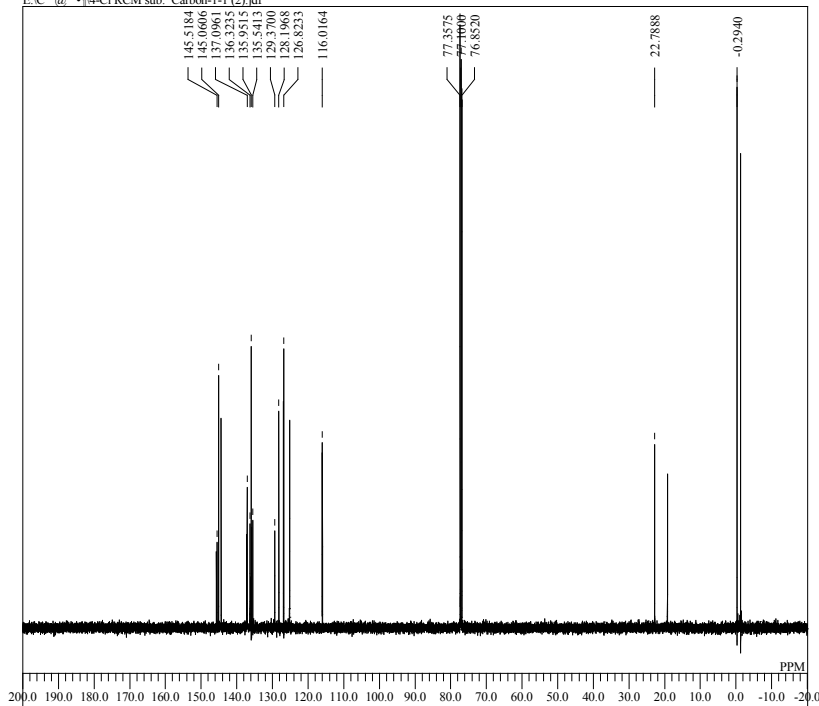
F1C' @ 1H-Cl RCM sub_proton-1-1 (2).jdf



DFILE 4-Cl RCM sub_proton-1-1 (2).jdf
 COMNT single_pulse
 DATIM 2018-11-20 12:05:22
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 16384
 FREQU 9384.38 Hz
 SCANS 16
 ACQTM 1.7459 sec
 PD 1.0000 sec
 PW1 6.50 usec
 IRNUC 1H
 CTEMP 20.6 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 34

3f

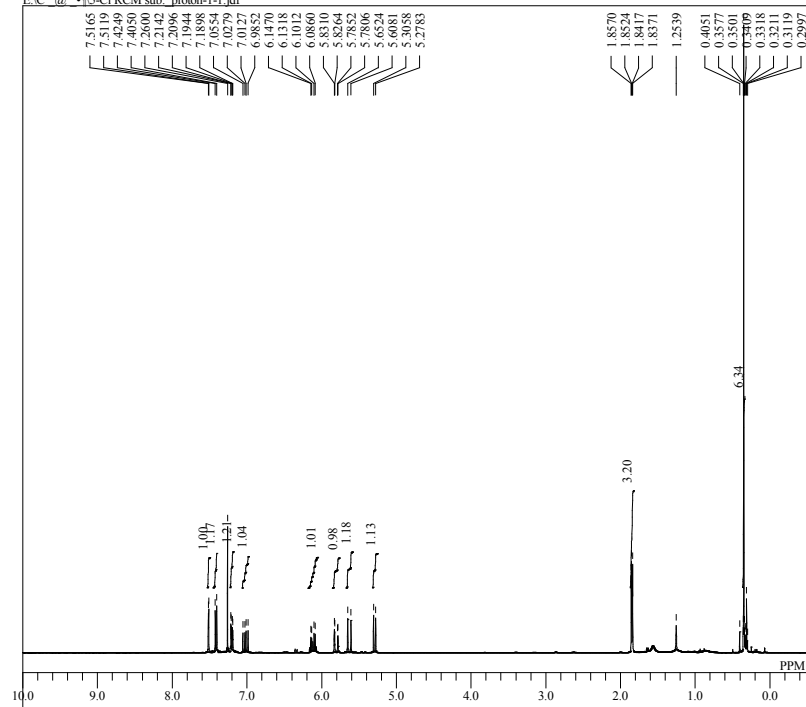
single pulse decoupled gated NOE

E: C¹³ @ 125 MHz 4-Cl RCM sub_Carbon-1-1 (2).jdf

DFILE 4-Cl RCM sub_Carbon-1-1 (2).jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-11-20 12:08:24
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 32767
 FREQU 39308.18 Hz
 SCANS 287
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.27 usec
 IRNUC 1H
 CTEMP 21.1 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

3g

single_pulse

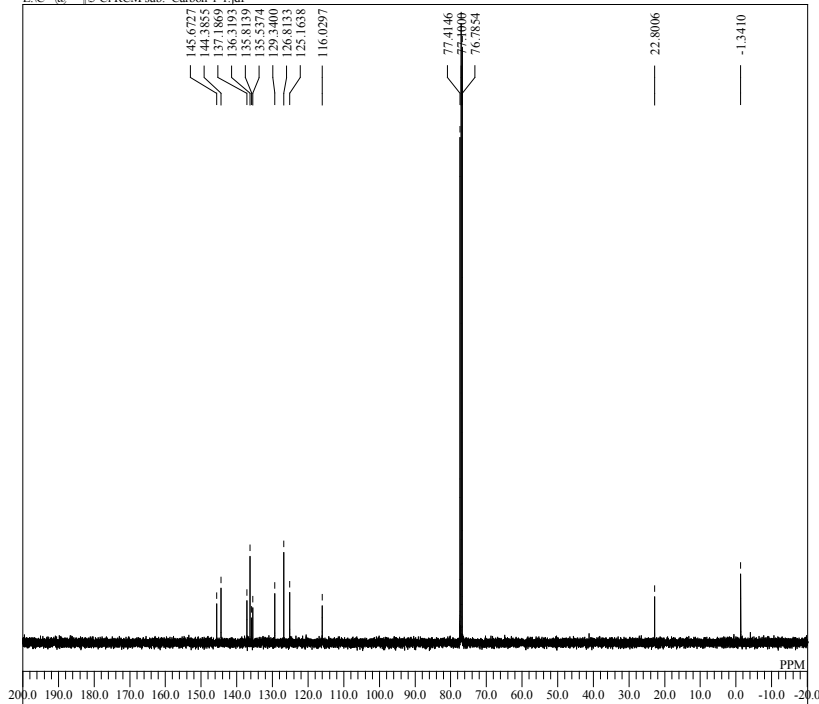
E: C¹³ @ 125 MHz 5-Cl RCM sub_proton-1-1.jdf

DFILE 5-Cl RCM sub_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-01-28 15:49:09
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 18.6 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 46

3g

single pulse decoupled gated NOE

E: C¹³ @ 100.53 MHz 5-Cl RCM sub_Carbon-1-1.jdf

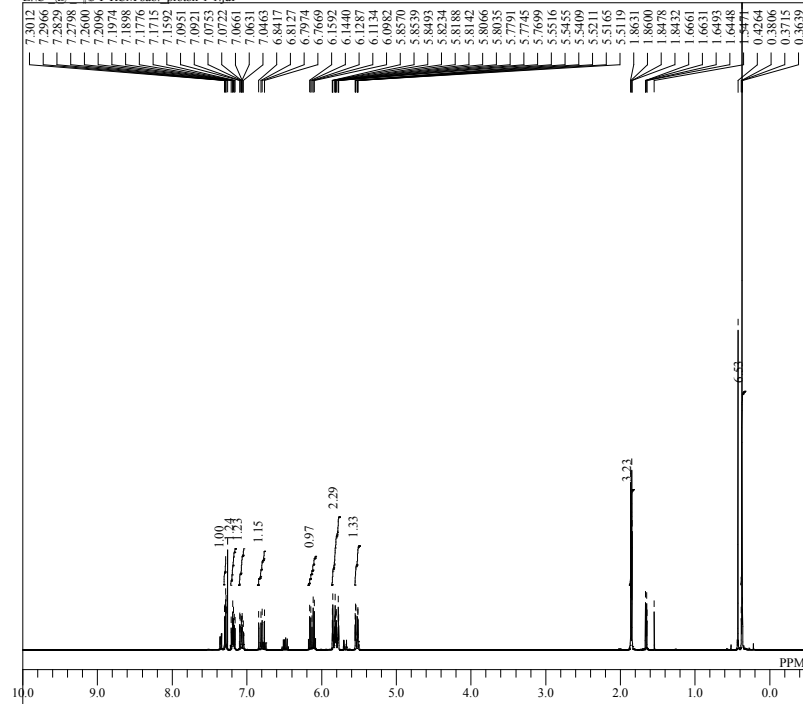


DFILE 5-Cl RCM sub_Carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2019-01-30 00:17:52
OBNUC 13C
EXMOD carbon.jpg
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 602
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

3h

single pulse

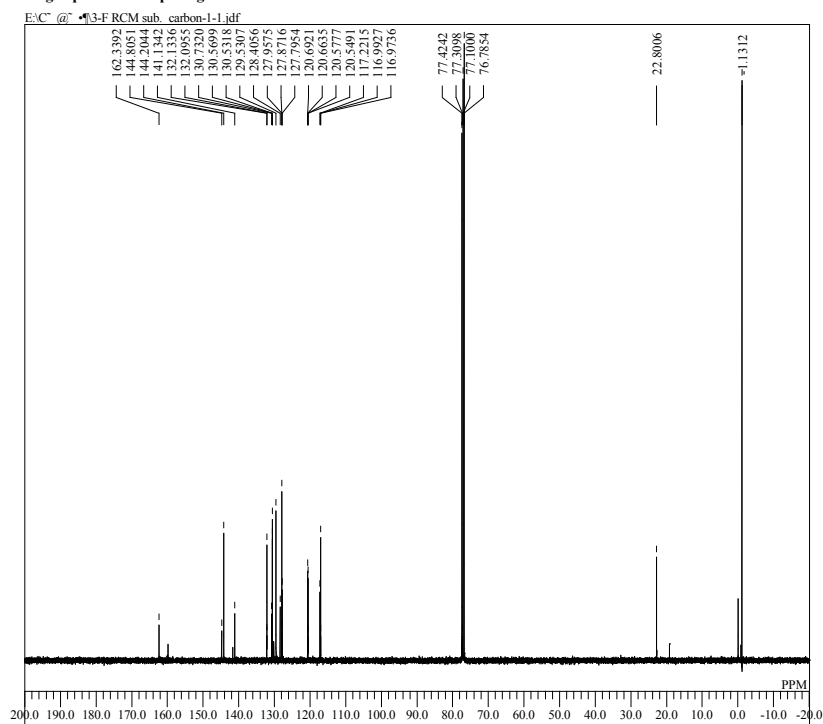
E: C¹³ @ 100.53 MHz 3-F RCM sub_proton-1-1.jdf



DFILE 3-F RCM sub_proton-1-1.jdf
COMNT single_pulse
DATIM 2019-01-31 21:00:29
OBNUC 1H
EXMOD proton.jpg
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 10000.00 Hz
SCANS 16
ACQTM 1.6384 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

3h

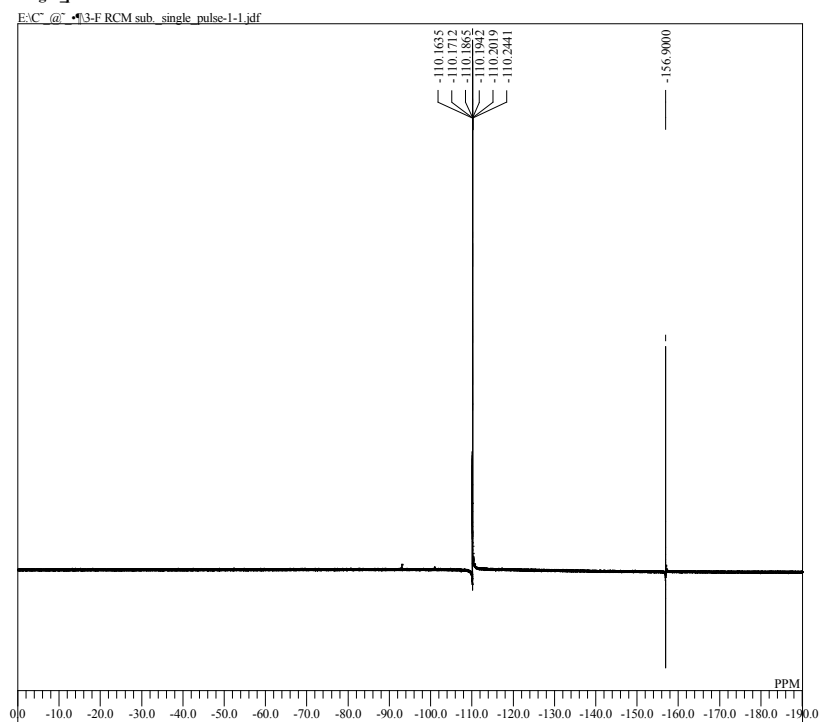
single pulse decoupled gated NOE



DFILE 3-F RCM sub_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-31 21:03:15
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 1892
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

3h

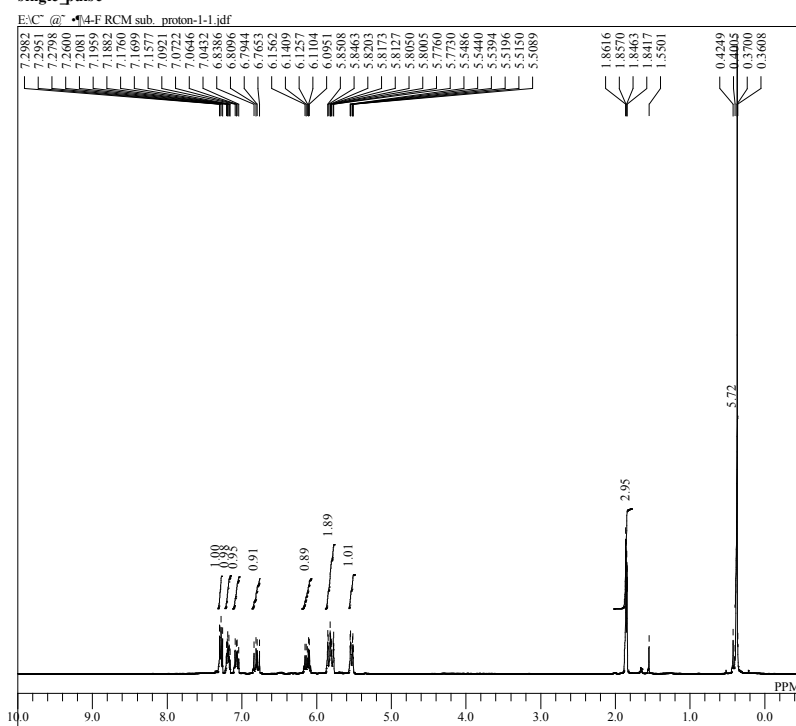
single_pulse



DFILE 3-F RCM sub_single_pulse-1-1.jdf
 COMNT single_pulse
 DATIM 2019-02-02 00:55:12
 OBNUC 13F
 EXMOD single_pulse.jxp
 OBFREQ 376.17 MHz
 OBSET 1.05 KHz
 OBFIN 3.93 Hz
 POINT 131071
 FREQU 189393.94 Hz
 SCANS 17
 ACQTM 0.6921 sec
 PD 5.0000 sec
 PW1 7.80 usec
 IRNUC 19F
 CTEMP 18.7 c
 SLVNT CDCL3
 EXREF -156.90 ppm
 BF 0.12 Hz
 RGAIN 42

3i

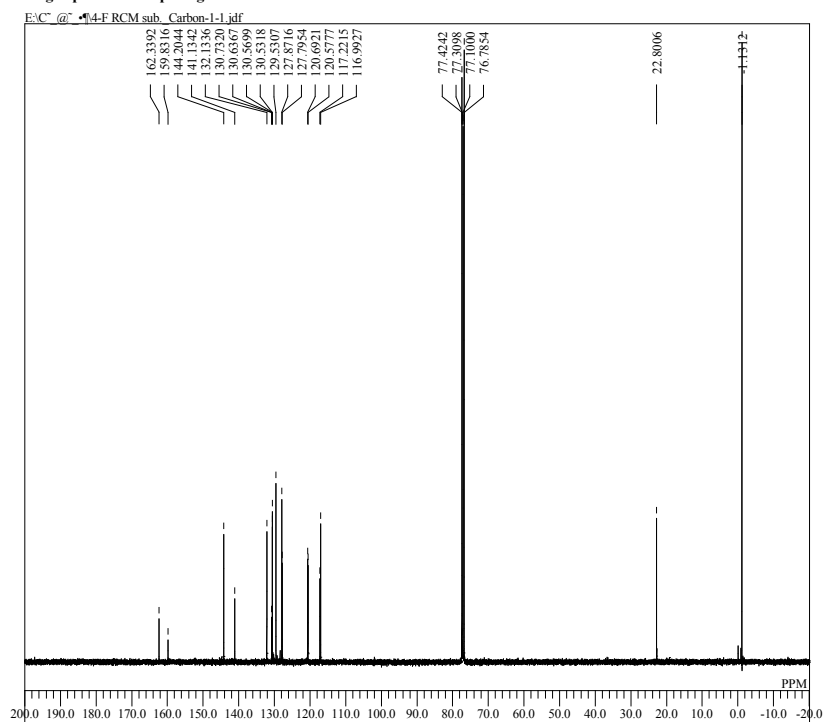
single pulse



DFILE 4-F RCM sub. proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-01-28 16:09:59
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 2.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 40

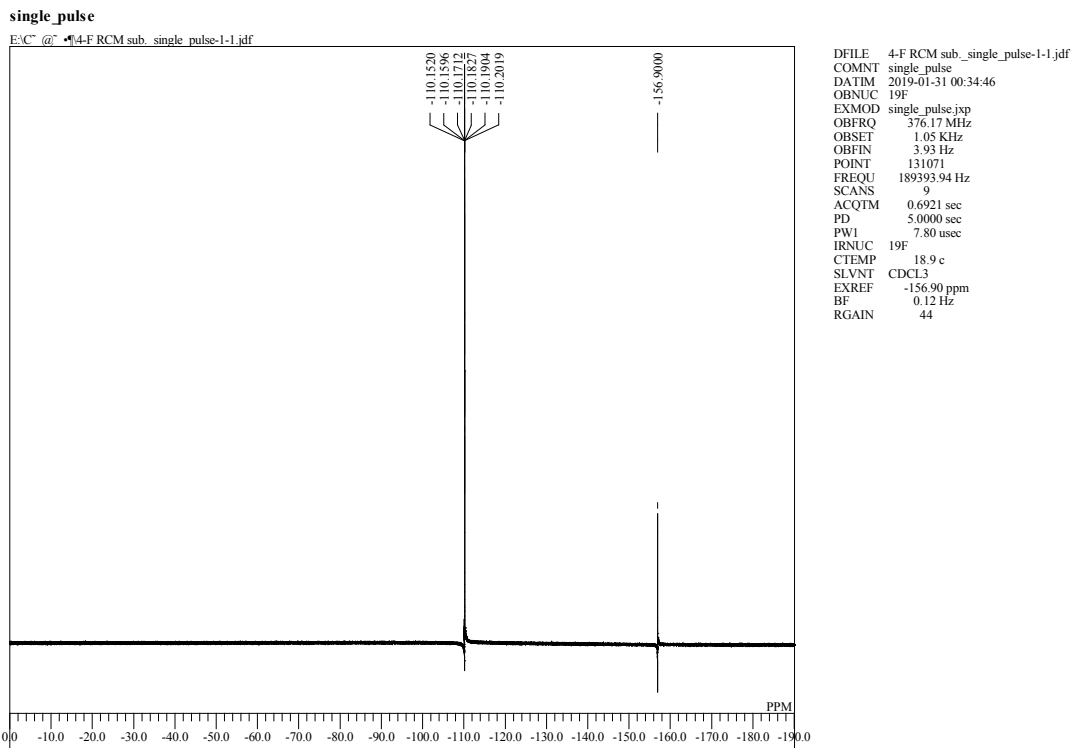
3i

single pulse decoupled gated NOE

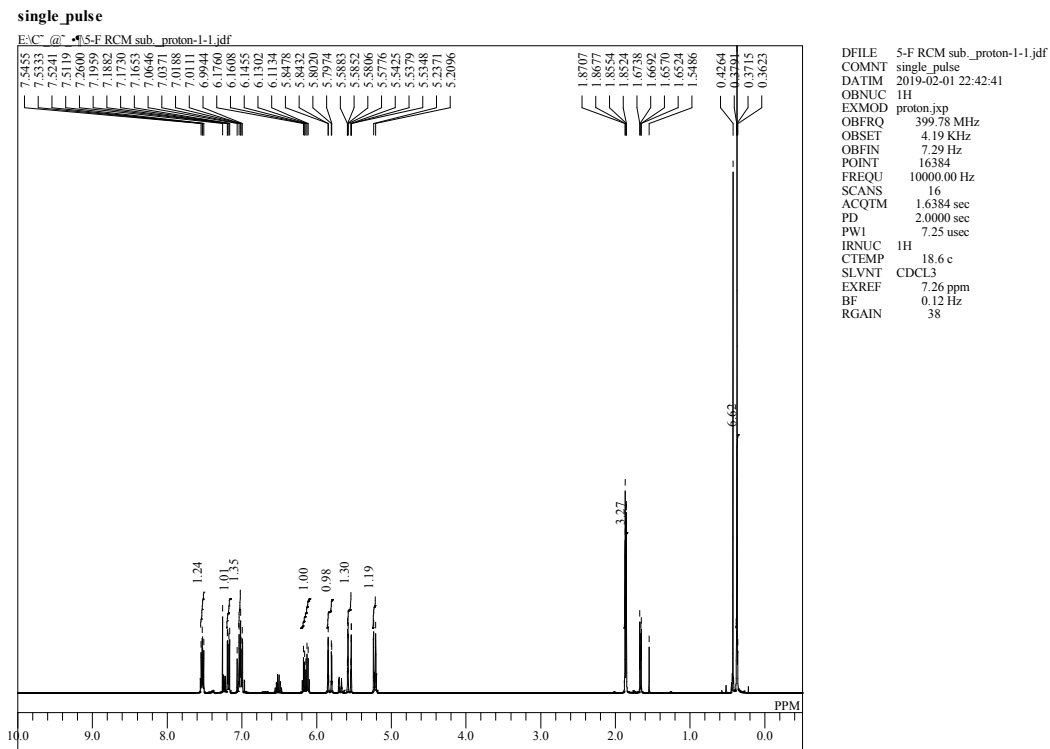


DFILE 4-F RCM sub. Carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-01-30 22:31:15
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 2307
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

3i

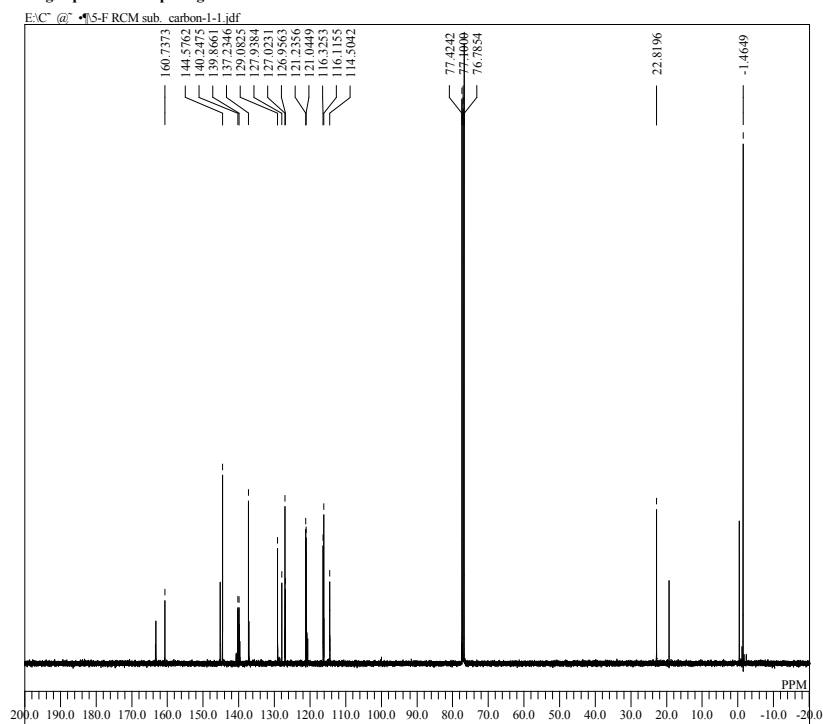


3j



3j

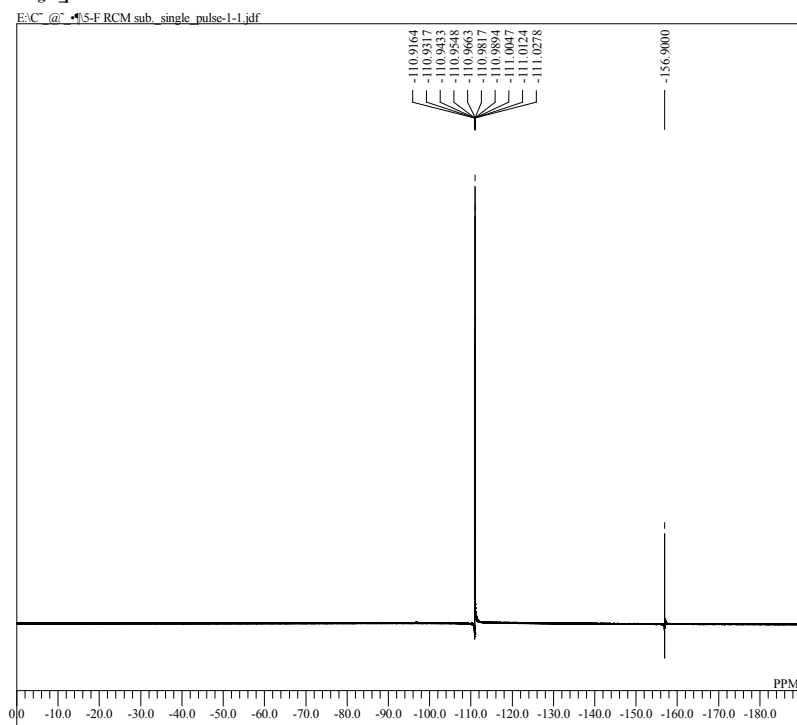
single pulse decoupled gated NOE



DFILE 5-F RCM sub_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-02-01 22:45:24
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 2181
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

3j

single_pulse

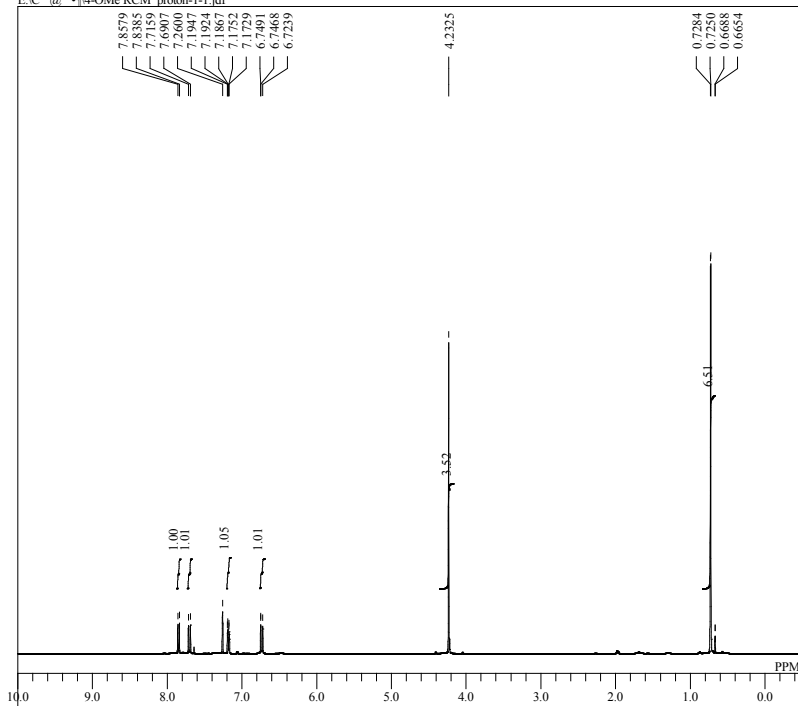


DFILE 5-F RCM sub_single_pulse-1-1.jdf
 COMNT single_pulse
 DATIM 2019-02-02 01:03:05
 OBNUC 19F
 EXMOD single_pulse.jxp
 OBFREQ 376.17 MHz
 OBSET 1.05 KHz
 OBFIN 3.93 Hz
 POINT 131071
 FREQU 189393.94 Hz
 SCANS 32
 ACQTM 0.6921 sec
 PD 5.0000 sec
 PW1 7.80 usec
 IRNUC 19F
 CTEMP 18.5 c
 SLVNT CDCL3
 EXREF -156.90 ppm
 BF 0.12 Hz
 RGAIN 44

4a

single pulse

E: C¹³ @ 4-Ome RCM proton-1-1.jdf

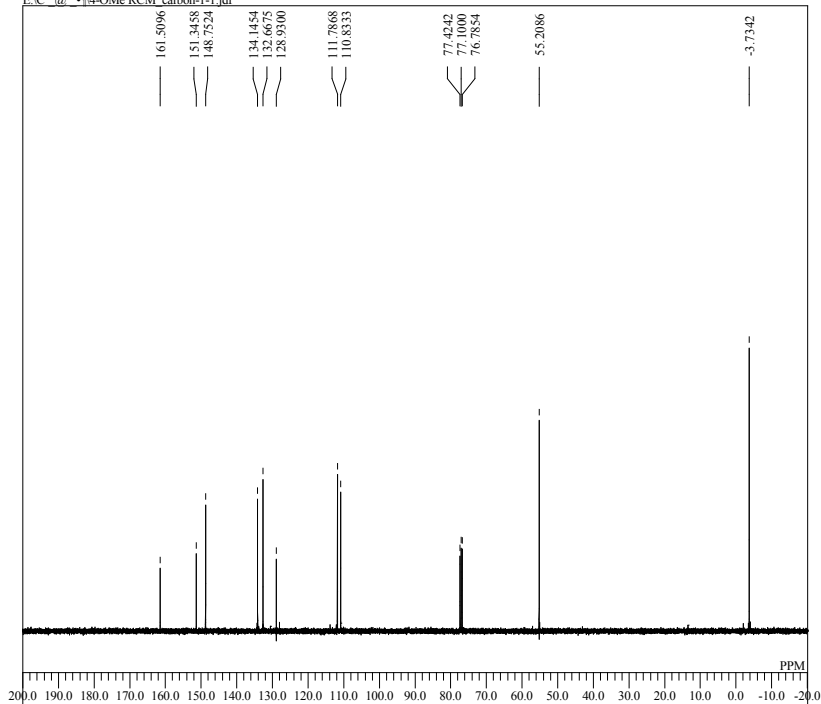


DFILE 4-Ome RCM_proton-1-1.jdf
COMNT single_pulse
DATIM 2018-12-08 20:43:26
OBNUC 1H
EXMOD proton.jsp
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 16
ACQTM 2.1837 sec
PD 1.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 28

4a

single pulse decoupled gated NOE

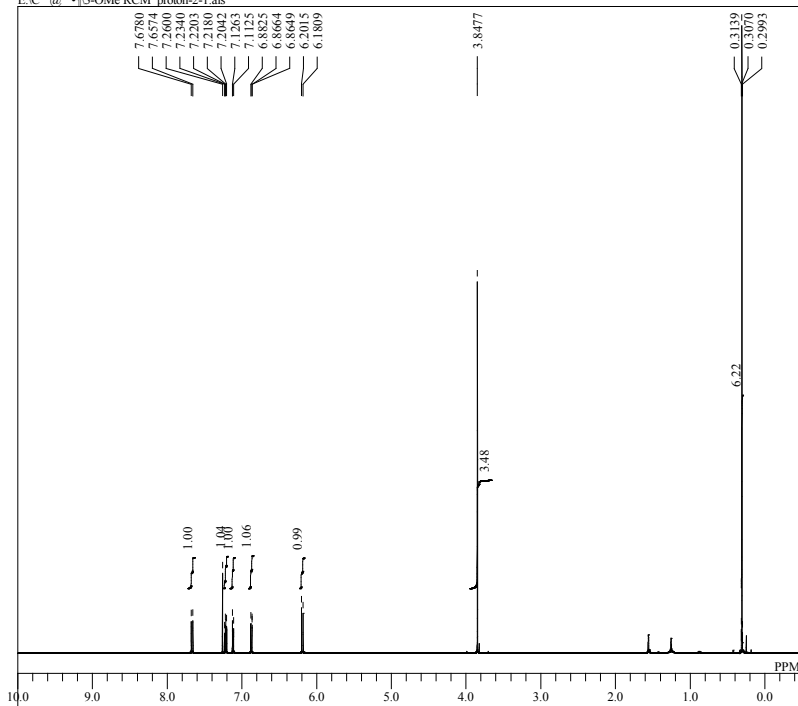
E: C¹³ @ 4-Ome RCM carbon-1-1.jdf



DFILE 4-Ome RCM_carbon-1-1.jdf
COMNT single_pulse decoupled gated NOE
DATIM 2018-12-08 20:46:12
OBNUC 13C
EXMOD carbon.jsp
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 52
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

4b

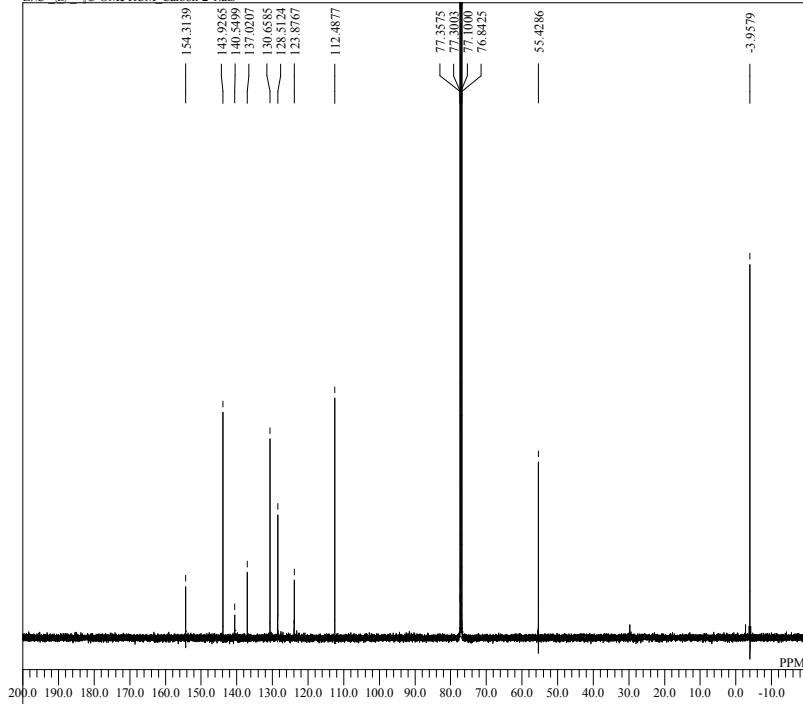
single pulse

F¹C¹³ @ 125 MHz 3-OMe RCM proton-2-1.als

DFILE 3-OMe RCM_proton-2-1.als
 COMNT single_pulse
 DATIM 2019-05-09 17:31:11
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 500.16 MHz
 OBSET 2.41 KHz
 OBFIN 6.01 Hz
 POINT 26214
 FREQU 10020.04 Hz
 SCANS 16
 ACQTM 2.6162 sec
 PD 2.0000 sec
 PW1 6.50 usec
 IRNUC 1H
 CTEMP 18.1 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 46

4b

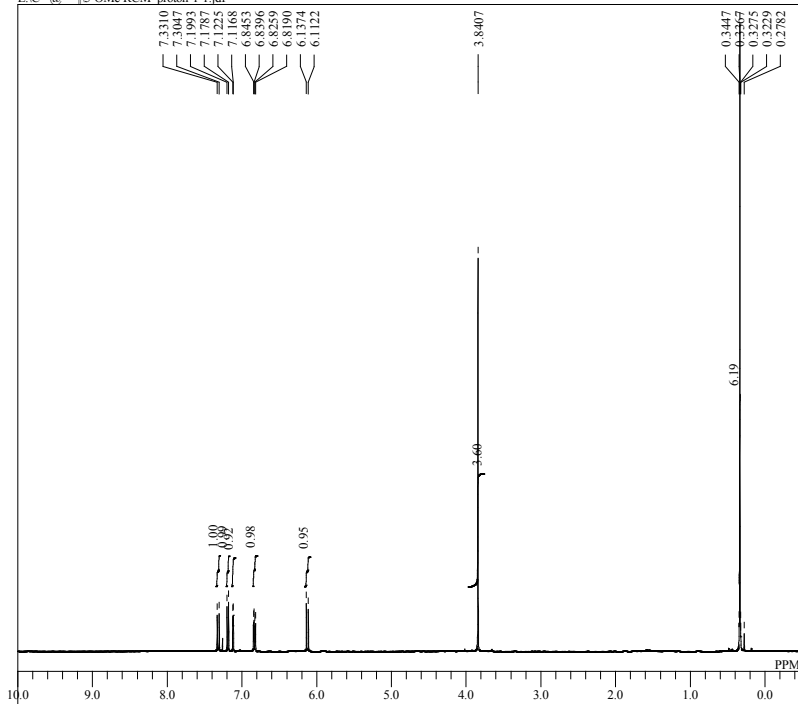
single pulse decoupled gated NOE

F¹C¹³ @ 125 MHz 3-OMe RCM Carbon-2-1.als

DFILE 3-OMe RCM_Carbon-2-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2019-05-09 17:34:40
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 26214
 FREQU 31446.54 Hz
 SCANS 3596
 ACQTM 0.8336 sec
 PD 1.0000 sec
 PW1 3.27 usec
 IRNUC 1H
 CTEMP 18.6 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

4c

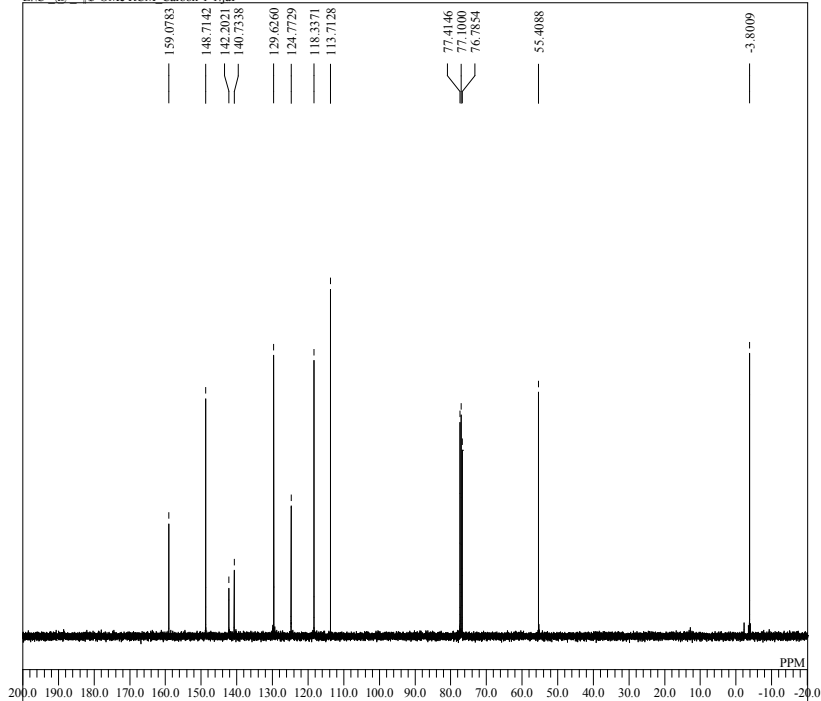
single pulse

F¹C¹³ @ 125 MHz 5-OMe RCM proton-1-1.jdf

DFILE 5-OMe RCM_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2018-12-08 20:55:01
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 32

4c

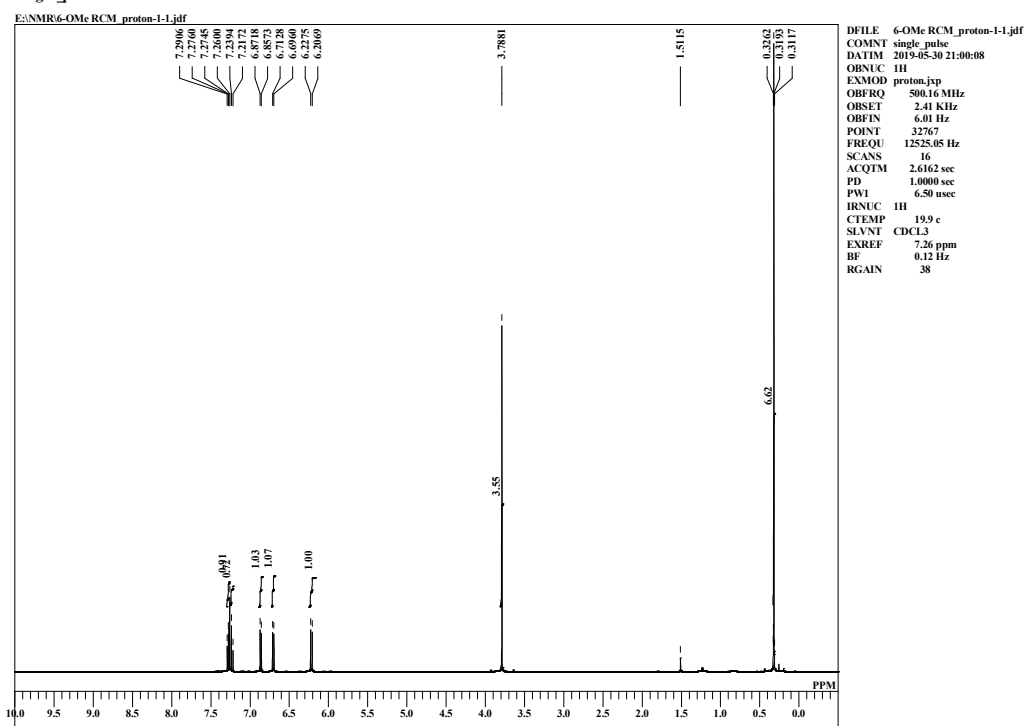
single pulse decoupled gated NOE

F¹C¹³ @ 125 MHz 5-OMe RCM Carbon-1-1.jdf

DFILE 5-OMe RCM_Carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-08 20:57:40
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 148
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

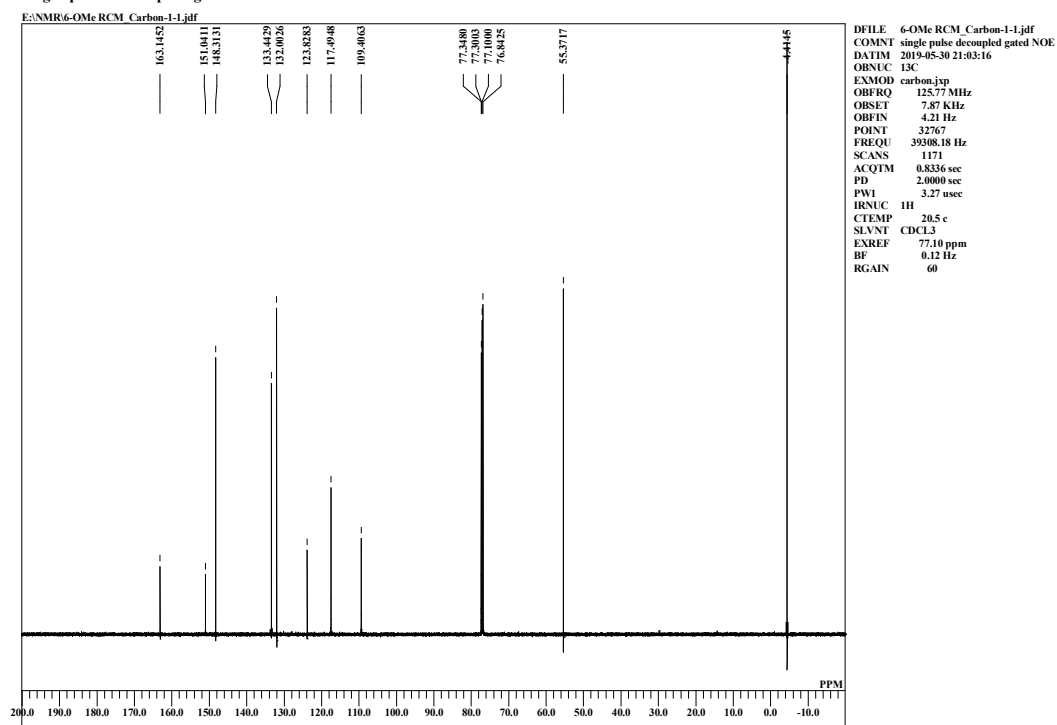
4d

single pulse



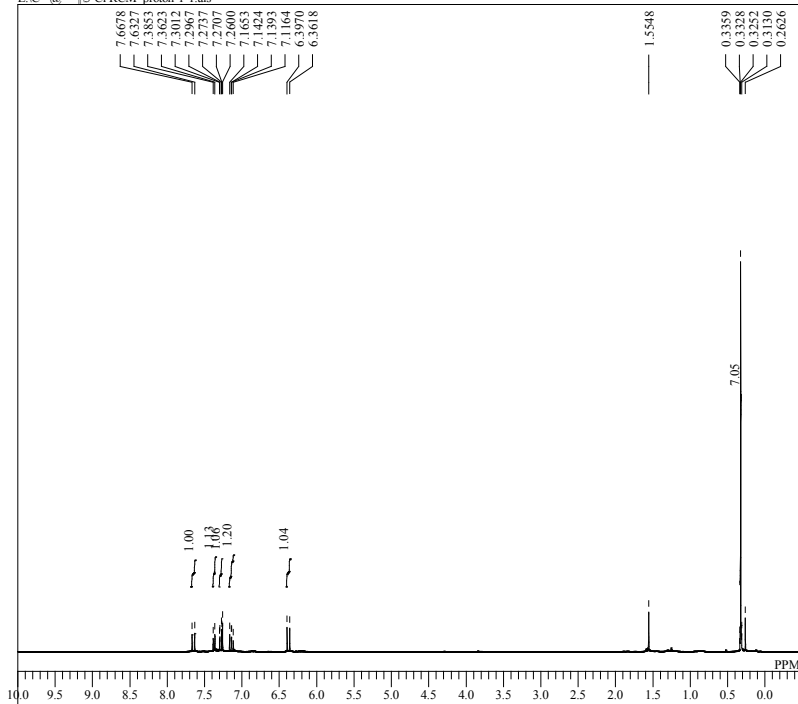
4d

single pulse decoupled gated NOE



4e

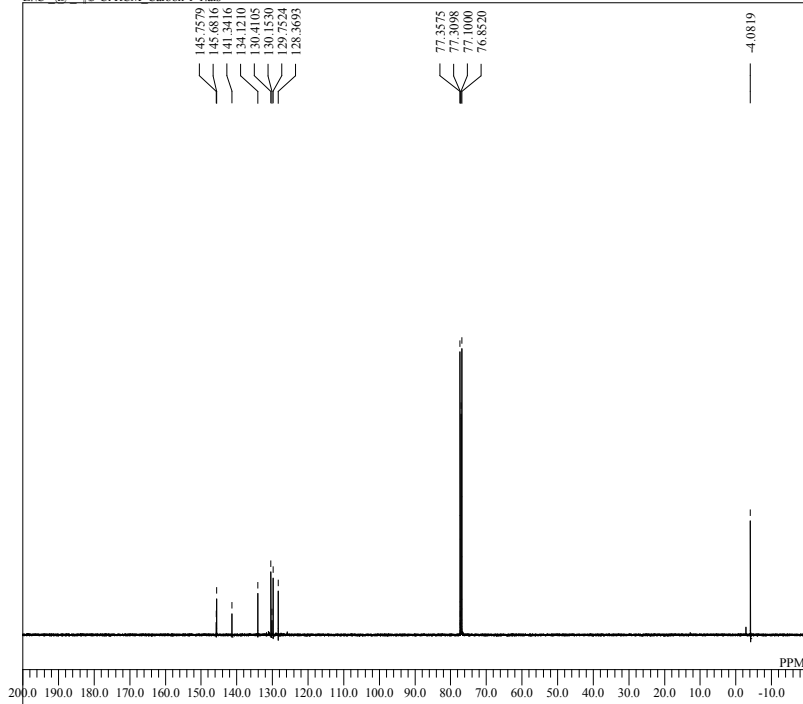
single pulse

F¹C¹ @ 300 MHz 3-Cl RCM proton-1-1.als

DFILE 3-Cl RCM_proton-1-1.als
 COMNT single_pulse
 DATIM 2019-05-16 22:57:05
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 300.53 MHz
 OBSET 1.15 KHz
 OBFIN 8.57 Hz
 POINT 13107
 FREQU 6016.85 Hz
 SCANS 16
 ACQTM 2.1784 sec
 PD 1.0000 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 19.9 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 38

4e

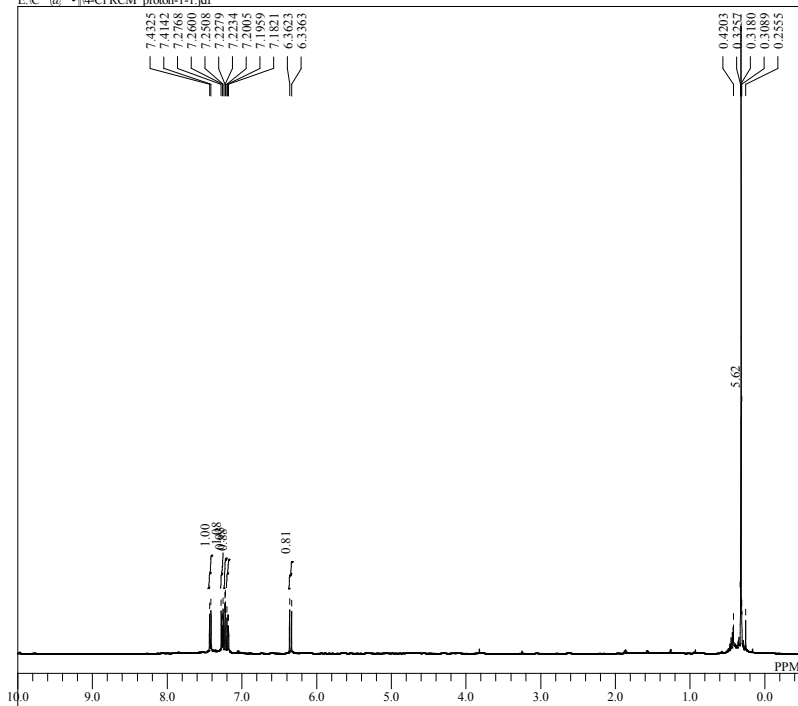
single pulse decoupled gated NOE

F¹C¹ @ 125 MHz 3-Cl RCM Carbon-1-1.als

DFILE 3-Cl RCM_Carbon-1-1.als
 COMNT single pulse decoupled gated NOE
 DATIM 2019-05-17 10:25:28
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 125.77 MHz
 OBSET 7.87 KHz
 OBFIN 4.21 Hz
 POINT 26214
 FREQU 31446.54 Hz
 SCANS 1882
 ACQTM 0.8336 sec
 PD 3.0000 sec
 PW1 3.27 usec
 IRNUC 1H
 CTEMP 20.5 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 56

4f

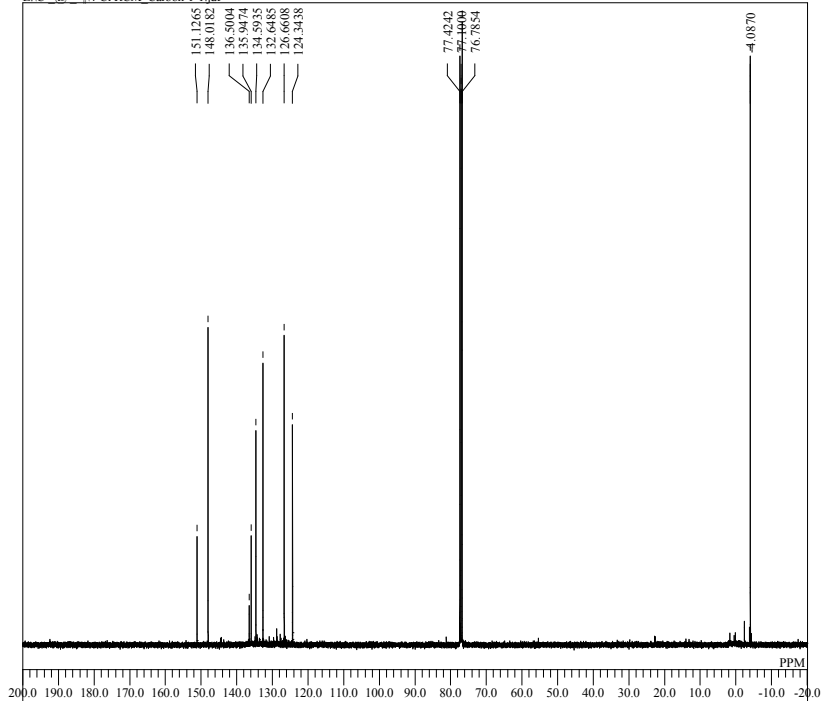
single pulse

E: C¹³ @ 4-Cl RCM_proton-1-1.jdf

DFILE 4-Cl RCM_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-02-05 07:05:42
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 18.9 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 38

4f

single pulse decoupled gated NOE

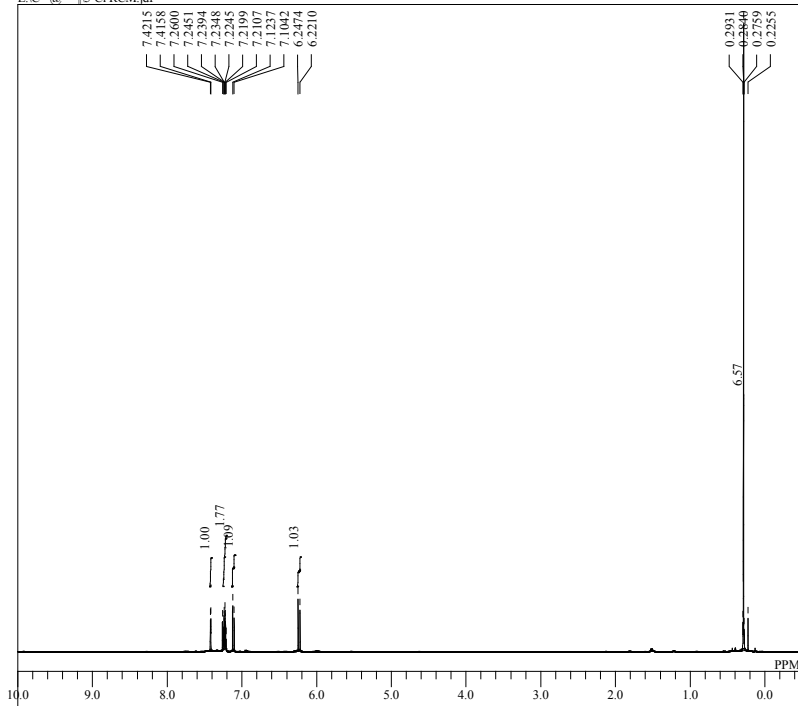
E: C¹³ @ 4-Cl RCM_Carbon-1-1.jdf

DFILE 4-Cl RCM_Carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-02-05 07:25:37
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 2444
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

4g

single pulse

E: C¹³ @ 125 MHz 5-Cl RCM.jdf

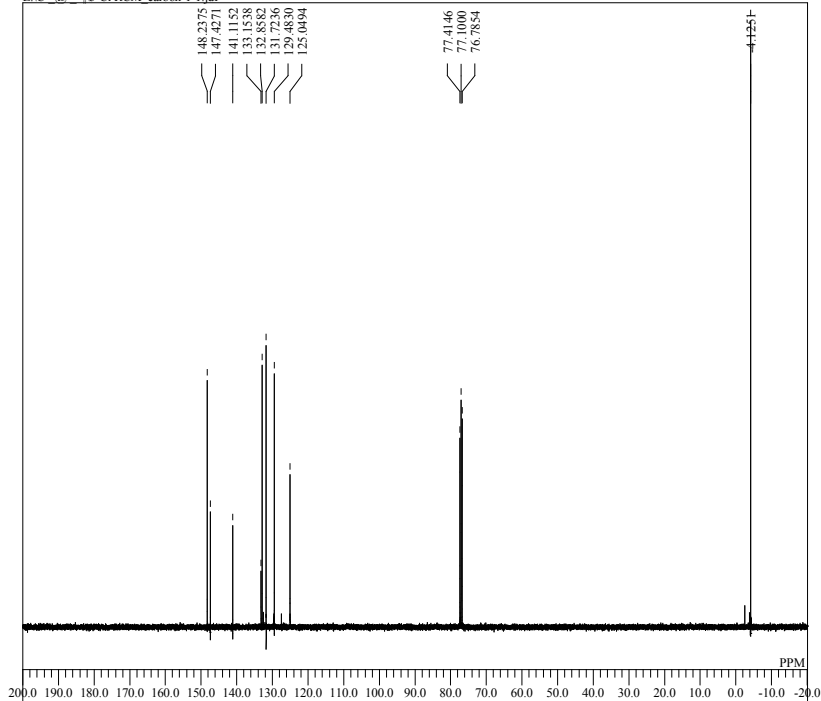


DFILE 5-Cl RCM.jdf
COMNT single_pulse
DATIM 2018-12-08 18:12:02
OBNUC 1H
EXMOD proton.jsp
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 16
ACQTM 2.1837 sec
PD 1.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 36

4g

single pulse decoupled gated NOE

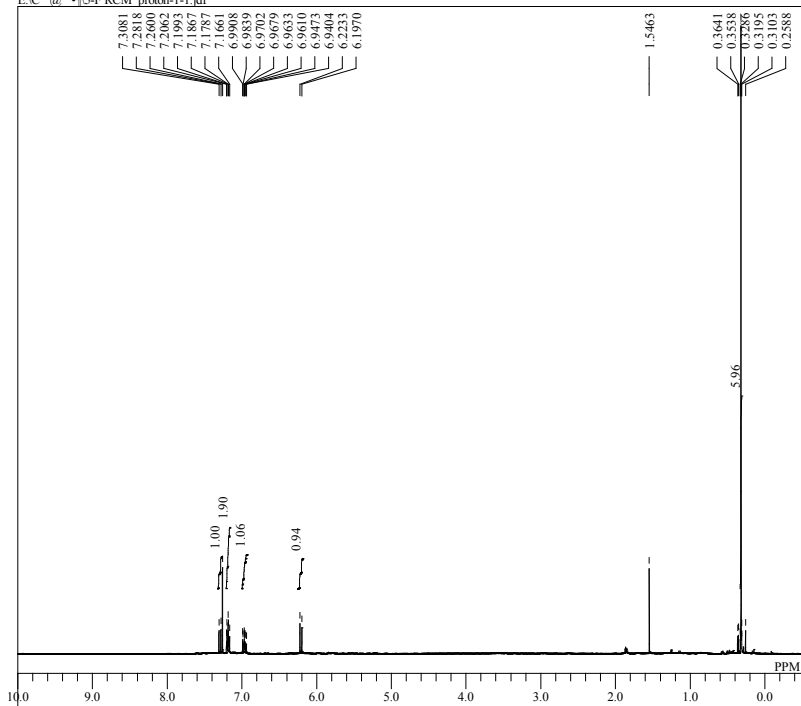
E: C¹³ @ 125 MHz 5-Cl RCM_carbon-1-1.jdf



DFILE 5-Cl RCM_carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2018-12-08 18:14:42
OBNUC 13C
EXMOD carbon.jsp
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 294
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

4j

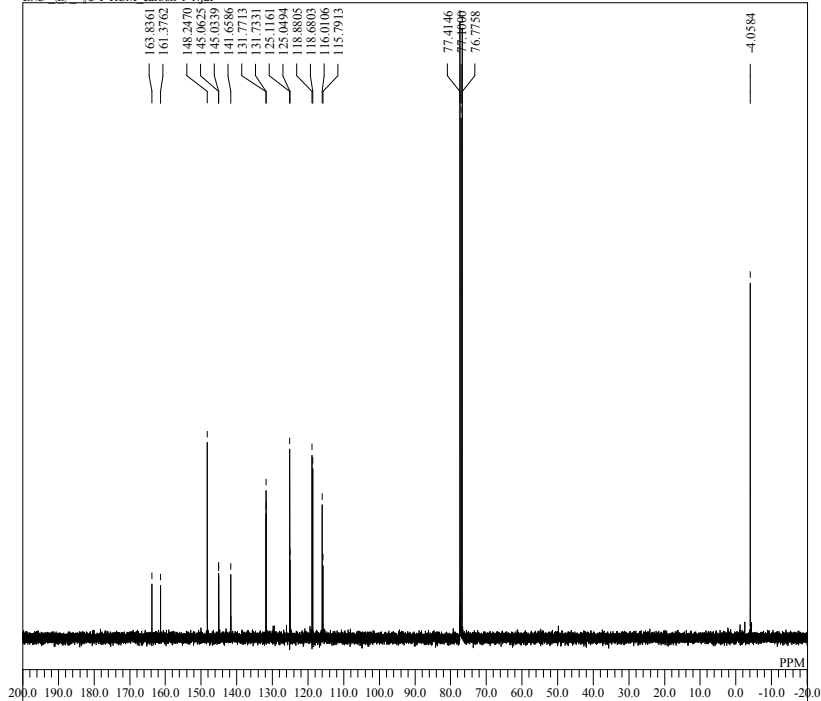
single pulse

F¹C¹³ @ 125 MHz 5-F RCM_proton-1-1.jdf

DFILE 5-F RCM_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2018-11-27 00:14:41
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 2.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 52

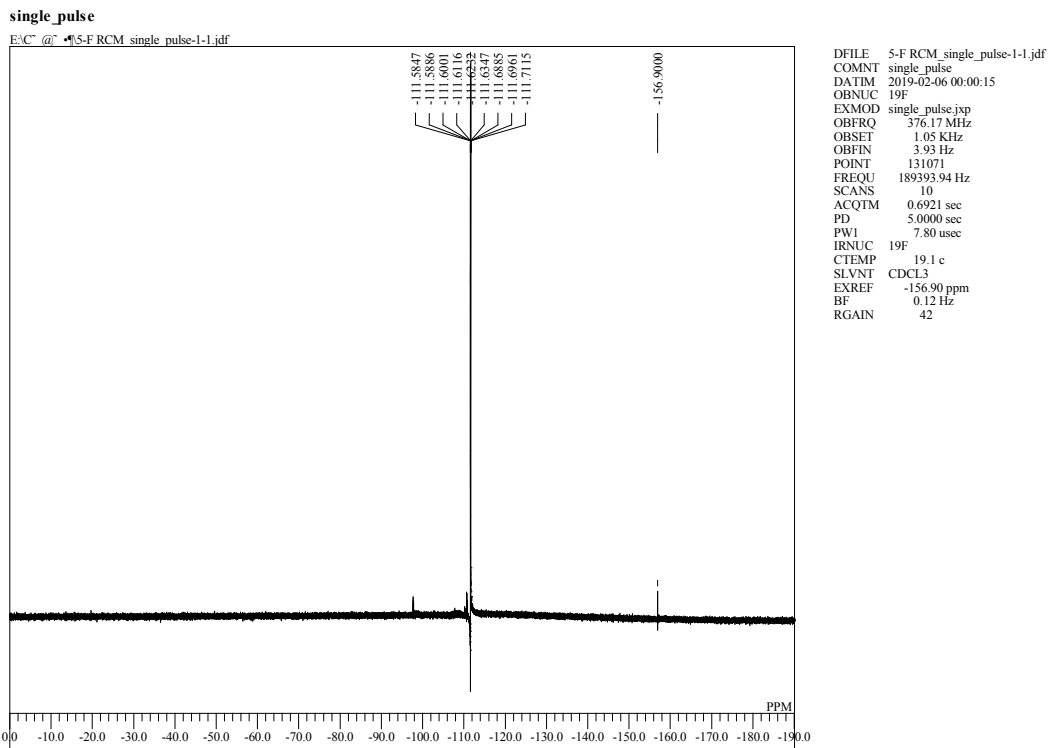
4j

single pulse decoupled gated NOE

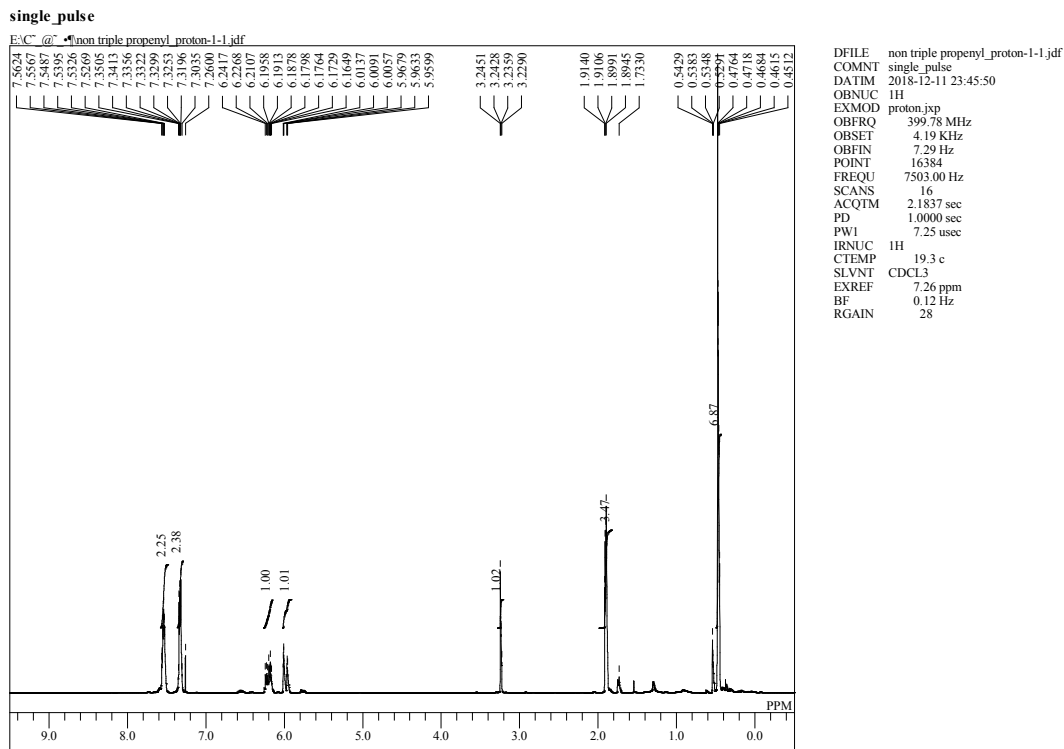
F¹C¹³ @ 125 MHz 5-F RCM_carbon-1-1.jdf

DFILE 5-F RCM_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-08 20:16:58
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 406
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

4j



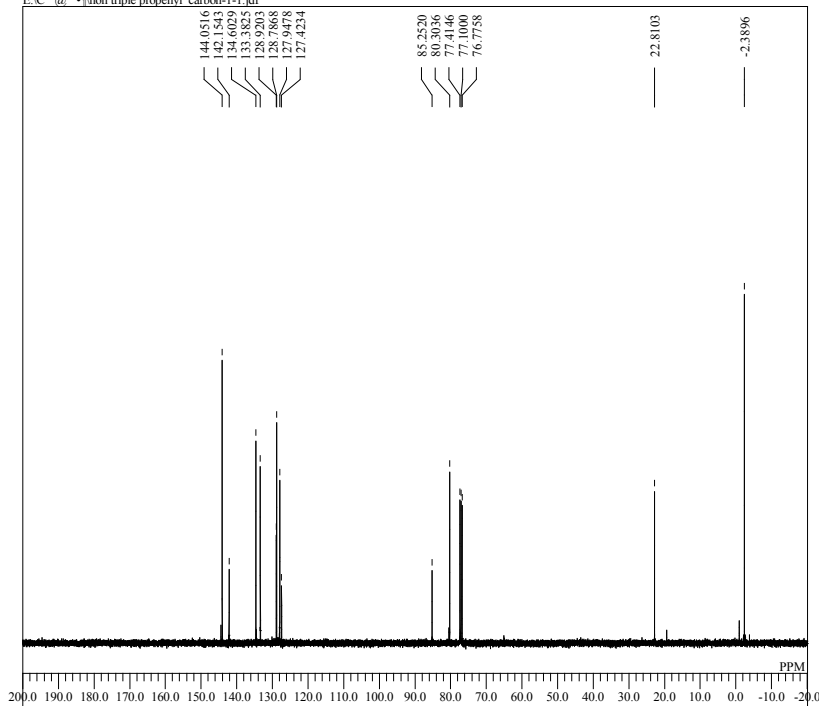
5a



5a

single pulse decoupled gated NOE

E: C¹³ @ 100.53 MHz non triple propenyl_carbon-1-1.jdf

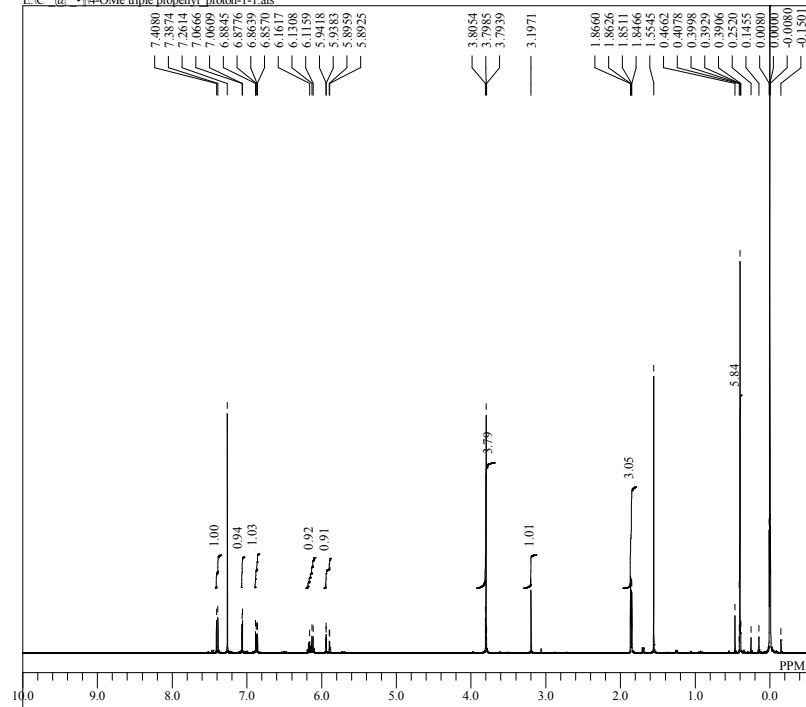


DFILE non triple propenyl_carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2018-12-11 23:48:30
OBNUC 13C
EXMOD carbon.jxp
OBFREQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 200
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.17 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.10 ppm
BF 0.12 Hz
RGAIN 60

5b

single_pulse

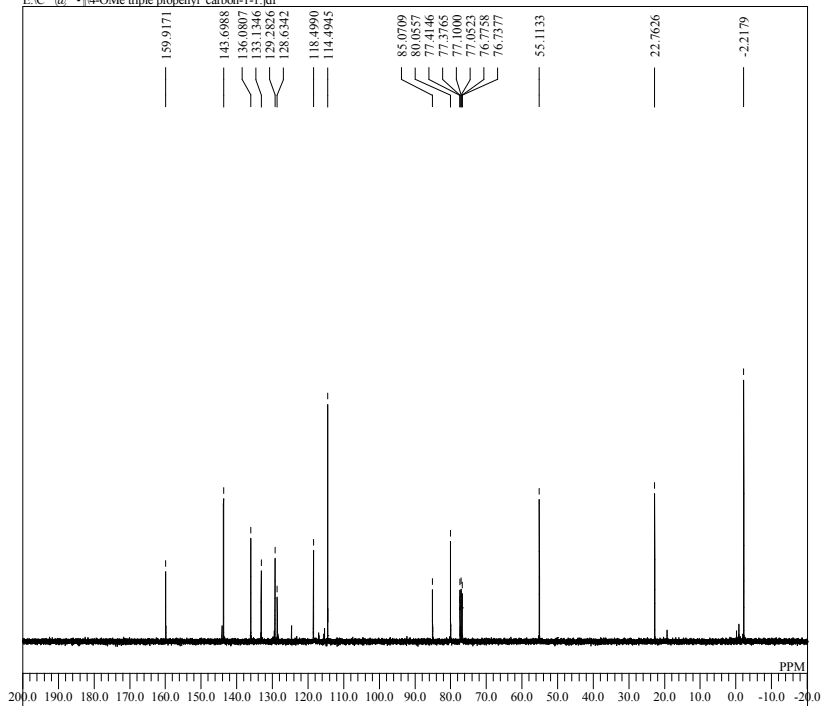
E: C¹³ @ 100.53 MHz 4-OMe triple propenyl_proton-1-1.als



DFILE 4-OMe triple propenyl_proton-1-1.als
COMNT single_pulse
DATIM 2019-01-07 14:36:43
OBNUC 1H
EXMOD proton.jxp
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 2.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48

5b

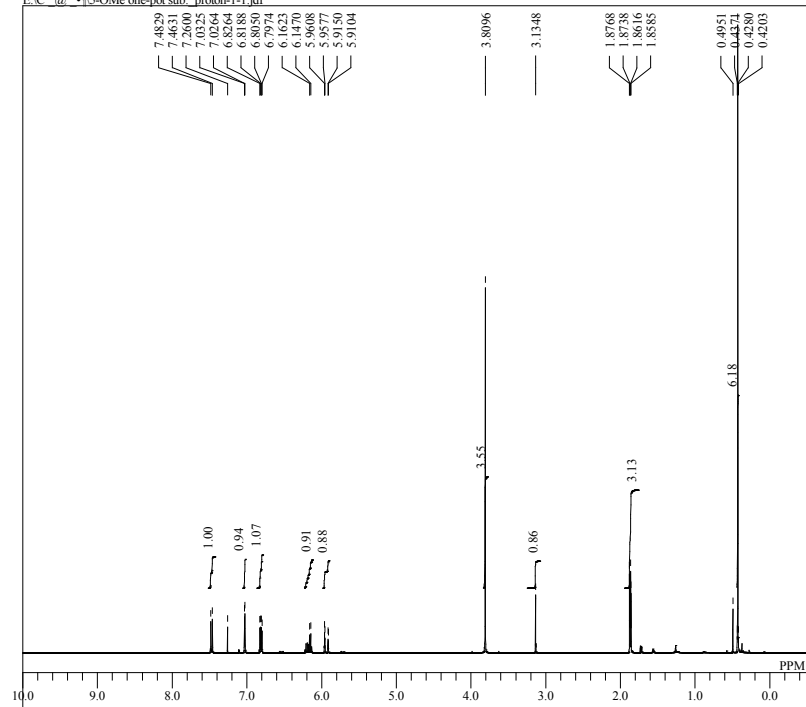
single pulse decoupled gated NOE

E: C¹³ @ 100 MHz 4-OMe triple propenyl carbon-1-1.jdf

DFILE 4-OMe triple propenyl carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-11 23:36:46
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 106
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

5c

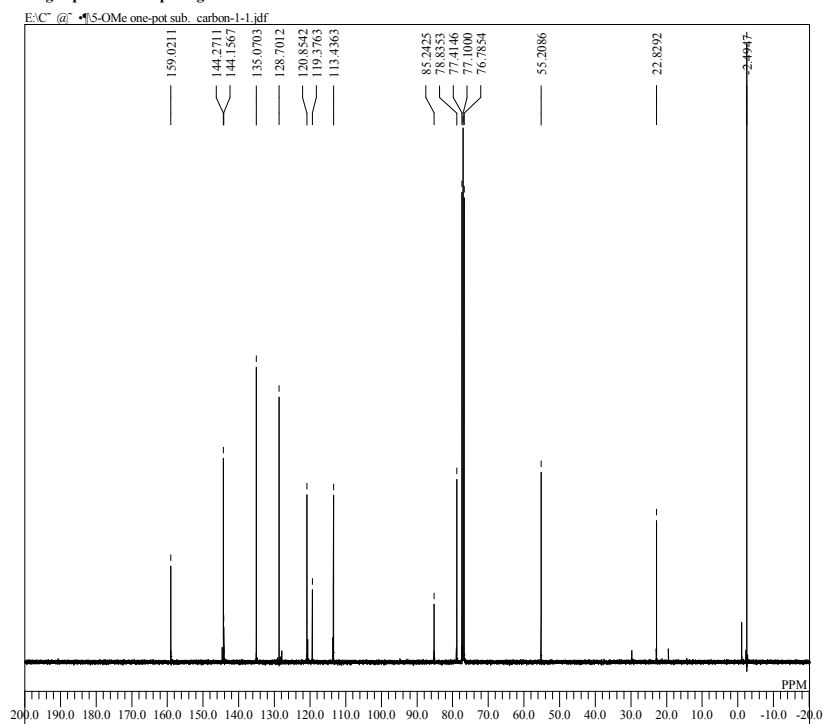
single_pulse

E: C¹³ @ 100 MHz 5-OMe one-pot sub_proton-1-1.jdf

DFILE 5-OMe one-pot sub_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2019-02-05 04:18:60
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 10000.00 Hz
 SCANS 16
 ACQTM 1.6384 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 38

5c

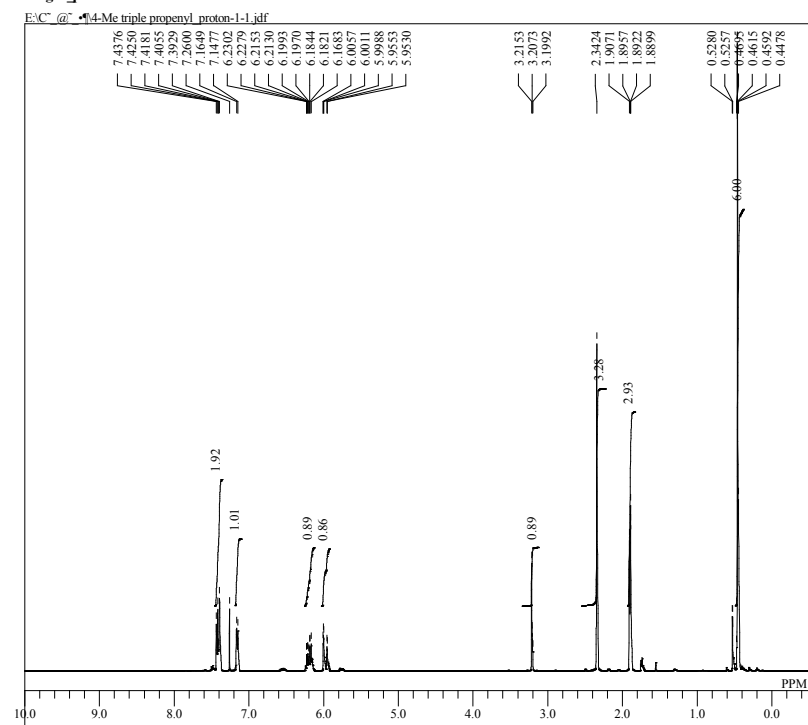
single pulse decoupled gated NOE



DFILE 5-Ome one-pot sub. carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-02-05 04:21:27
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 3000
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

5d

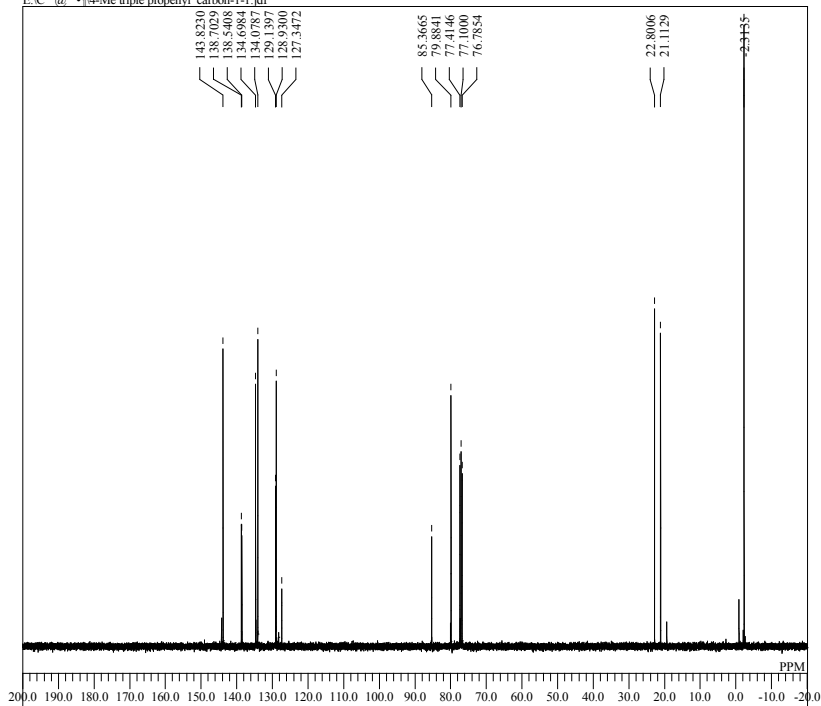
single_pulse



DFILE 4-Me triple propenyl_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2018-12-14 22:53:54
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 28

5d

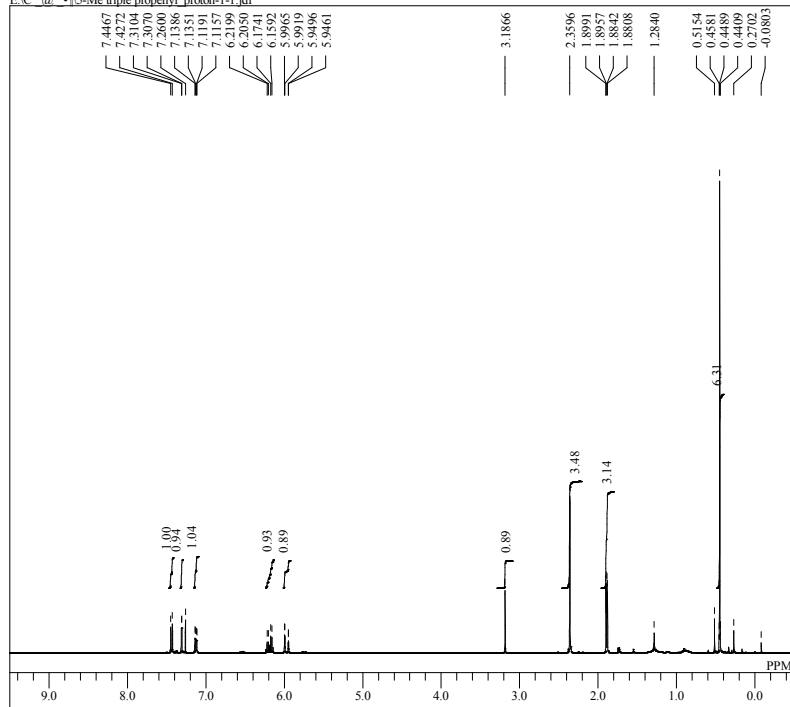
single pulse decoupled gated NOE

E: C¹³ @ 100 MHz 4-Me triple propenyl_carbon-1-1.jdf

DFILE 4-Me triple propenyl_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-14 22:56:34
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 253
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

5e

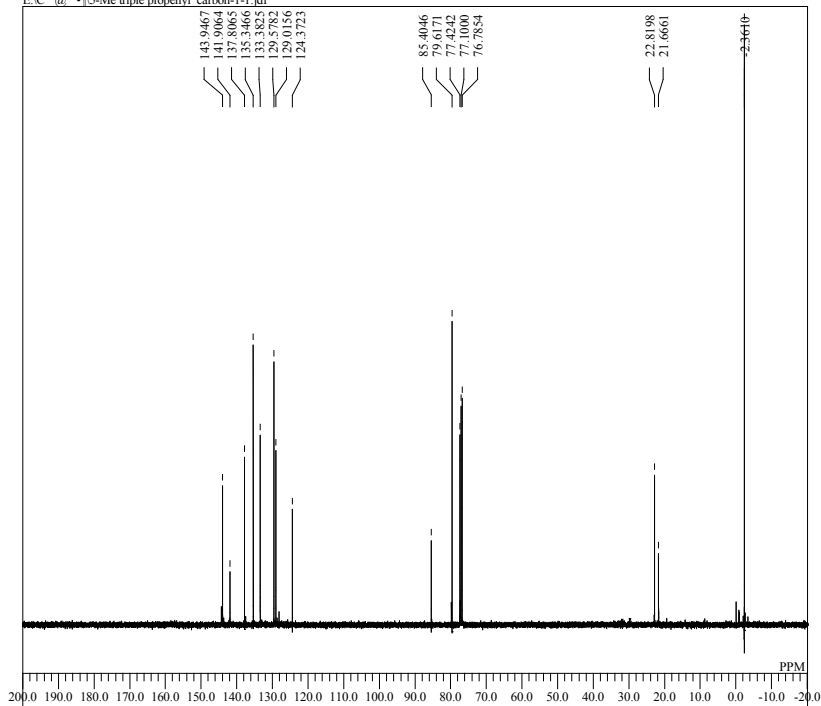
single_pulse

E: C¹³ @ 100 MHz 5-Me triple propenyl_proton-1-1.jdf

DFILE 5-Me triple propenyl_proton-1-1.jdf
 COMNT single_pulse
 DATIM 2018-12-11 23:14:55
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 19.3 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 28

5e

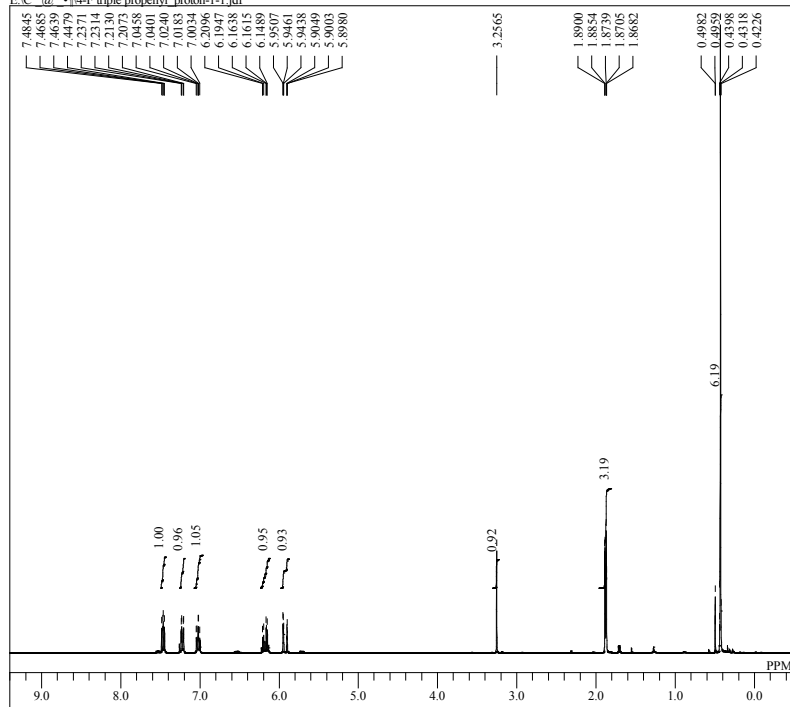
single pulse decoupled gated NOE

F¹C¹³ @ 125 MHz 5-Me triple propenyl carbon-1-1.jdf

DFILE 5-Me triple propenyl carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-11 23:17:35
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 244
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

5f

single pulse

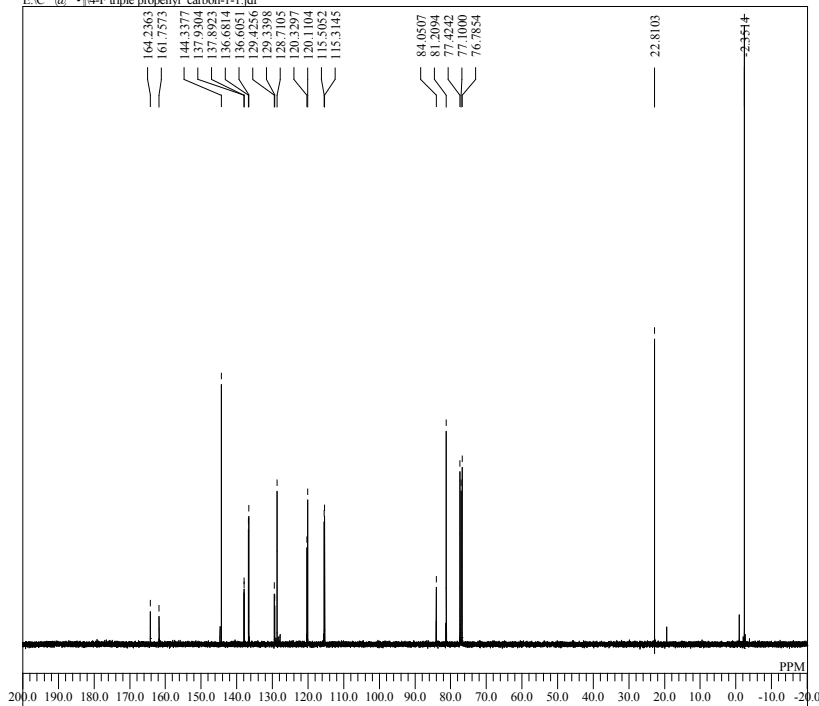
F¹C¹³ @ 125 MHz 4-F triple propenyl proton-1-1.jdf

DFILE 4-F triple propenyl proton-1-1.jdf
 COMNT single_pulse
 DATIM 2018-12-11 22:29:08
 OBNUC 1H
 EXMOD proton.jxp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16384
 FREQU 7503.00 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 1.0000 sec
 PW1 7.25 usec
 IRNUC 1H
 CTEMP 18.8 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 28

5f

single pulse decoupled gated NOE

E:\C\ @\ 4-F triple propenyl carbon-1-1.jdf

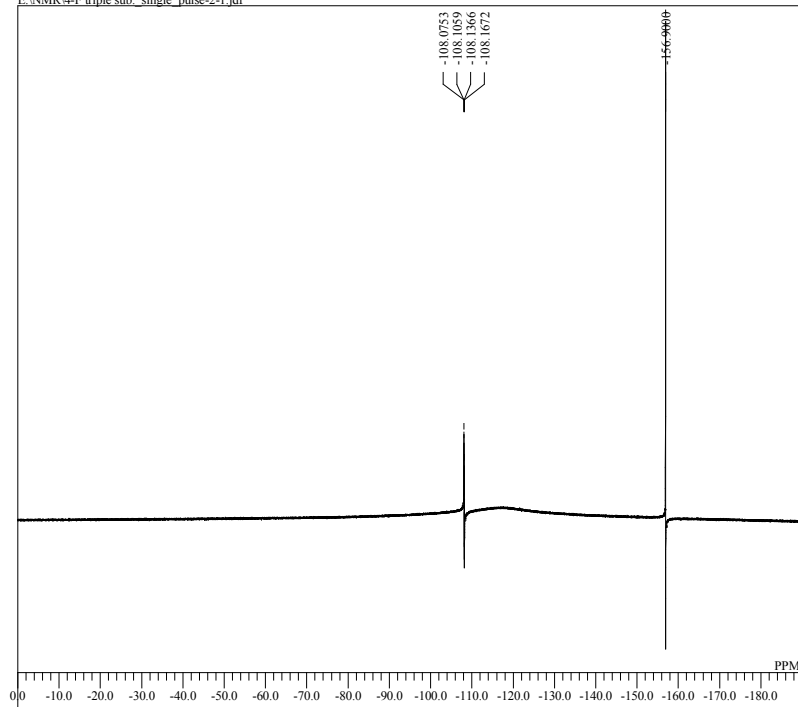


DFILE 4-F triple propenyl carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-11 22:31:51
 OBNUC 13C
 EXMOD carbon.jxp
 OBFREQ 100.53 MHz
 OBSET 5.33 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 202
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.1 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

5f

single pulse

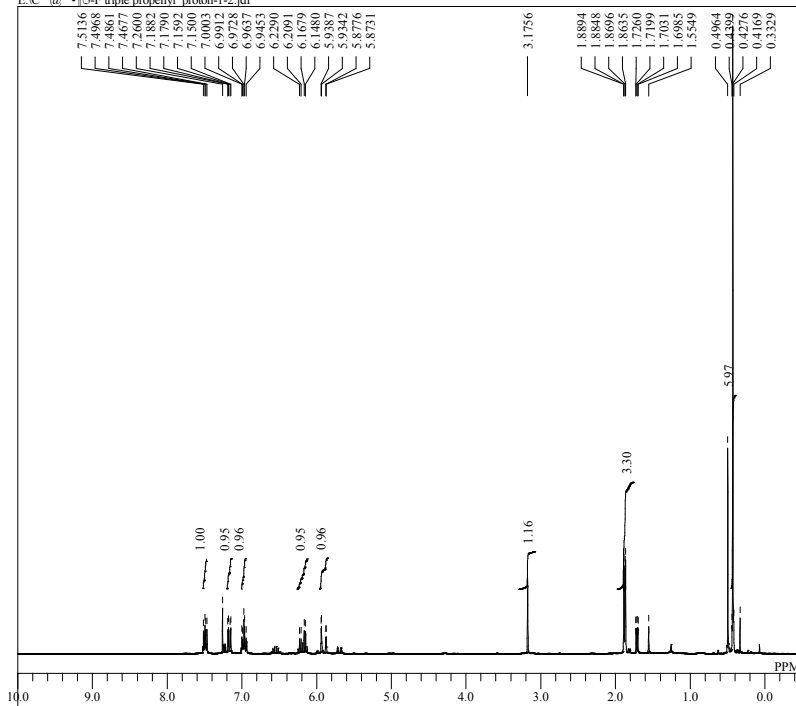
E:\NMR\4-F triple sub_single_pulse-2-1.jdf



DFILE 4-F triple sub_single_pulse-2-1.jdf
 COMNT single_pulse
 DATIM 2019-02-06 08:34:36
 OBNUC 19F
 EXMOD single_pulse.jxp
 OBFREQ 282.75 MHz
 OBSET 2.09 KHz
 OBFIN 0.53 Hz
 POINT 16384
 FREQU 71022.73 Hz
 SCANS 32
 ACQTM 0.2307 sec
 PD 5.0000 sec
 PW1 6.00 usec
 IRNUC 19F
 CTEMP 18.0 c
 SLVNT CDCL3
 EXREF -156.90 ppm
 BF 0.12 Hz
 RGAIN 38

5g

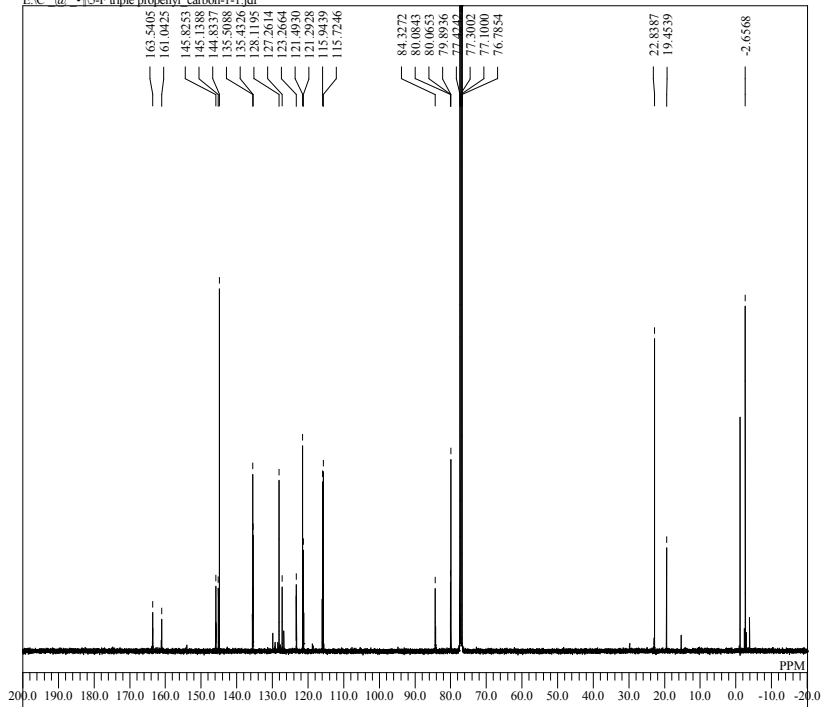
single pulse

F¹C¹³ @ 5-F triple propenyl_proton-1-2.jdf

DFILE 5-F triple propenyl_proton-1-2.jdf
 COMNT single_pulse
 DATIM 2018-12-13 10:24:39
 OBNUC 1H
 EXMOD proton.jsp
 OBFREQ 300.53 MHz
 OBSET 1.15 KHz
 OBFIN 8.57 Hz
 POINT 16384
 FREQU 7521.06 Hz
 SCANS 16
 ACQTM 2.1784 sec
 PD 1.0000 sec
 PW1 5.50 usec
 IRNUC 1H
 CTEMP 18.5 c
 SLVNT CDCL3
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 38

5g

single pulse decoupled gated NOE

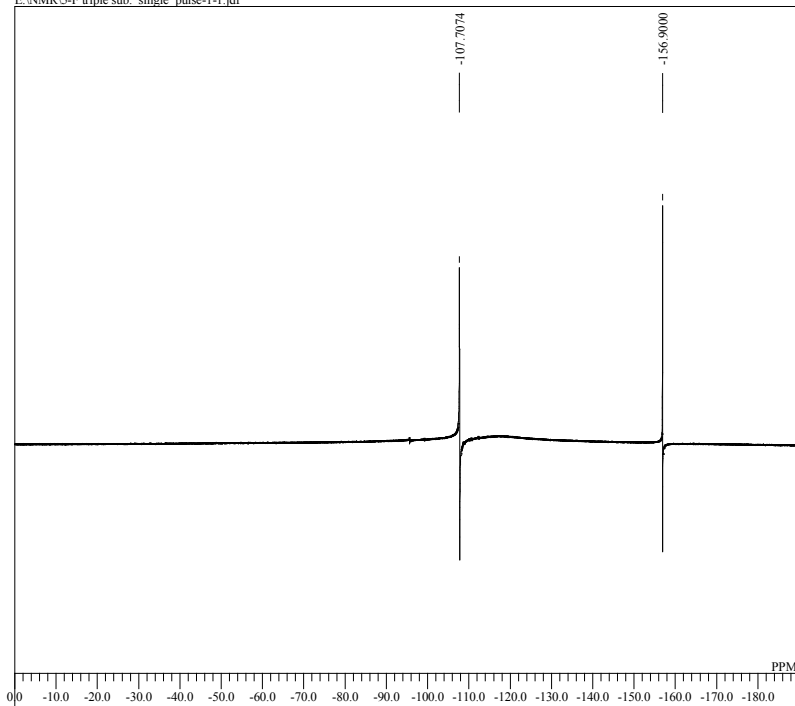
F¹C¹³ @ 5-F triple propenyl_carbon-1-1.jdf

DFILE 5-F triple propenyl_carbon-1-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2018-12-15 00:19:33
 OBNUC 13C
 EXMOD carbon.jsp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 11179
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC 1H
 CTEMP 19.2 c
 SLVNT CDCL3
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

5g

single_pulse

E:\NMR\5-F triple sub. single_pulse-1-1.jdf

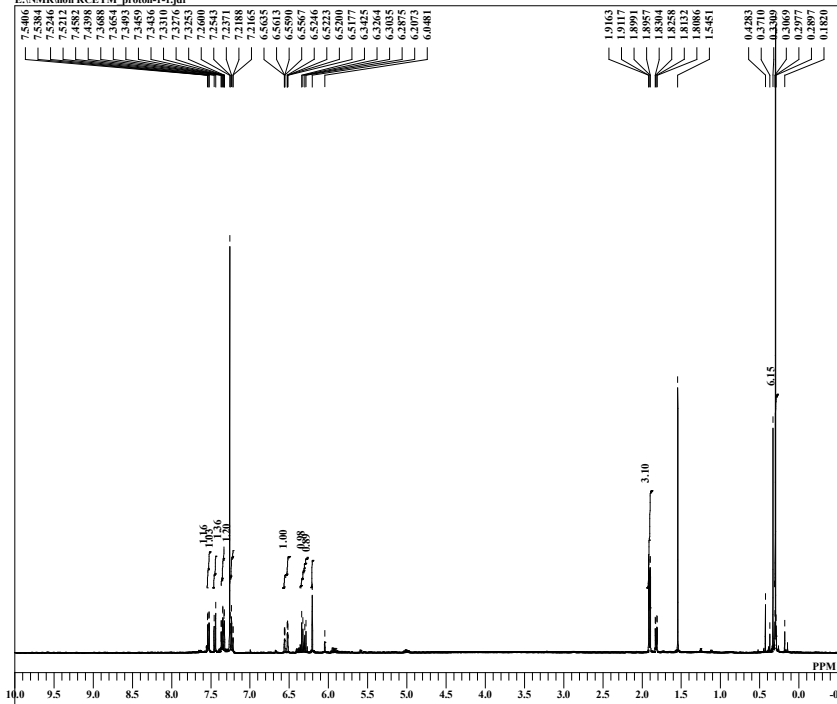


DFILE 5-F triple sub. single_pulse-1-1.jdf
COMINT single_pulse
DATIM 2019-02-06 08:40:01
OBNUC 19F
EXMOD single_pulse.jsp
OBFREQ 282.75 MHz
OBSET 2.09 KHz
OBFIN 0.53 Hz
POINT 16384
FREQU 71022.73 Hz
SCANS 32
ACQTM 0.2307 sec
PD 5.0000 sec
PW1 6.00 usec
IRNUC 19F
CTEMP 17.9 c
SLVNT CDCL3
EXREF -156.90 ppm
BF 0.12 Hz
RGAIN 38

6a

single_pulse

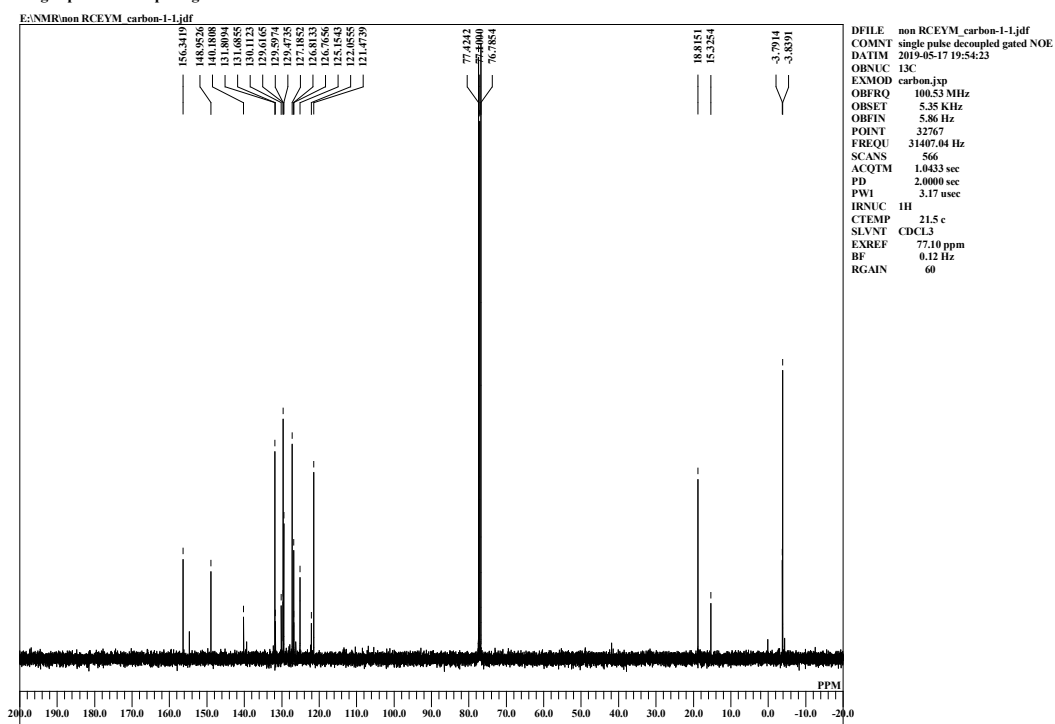
E:\NMR\non RCEYM proton-1-1.jdf



DFILE non RCEYM proton-1-1.jdf
COMINT single_pulse
DATIM 2019-05-17 19:41:54
OBNUC 1H
EXMOD proton.jsp
OBFREQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 16
ACQTM 2.1837 sec
PD 1.0000 sec
PW1 7.25 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 54

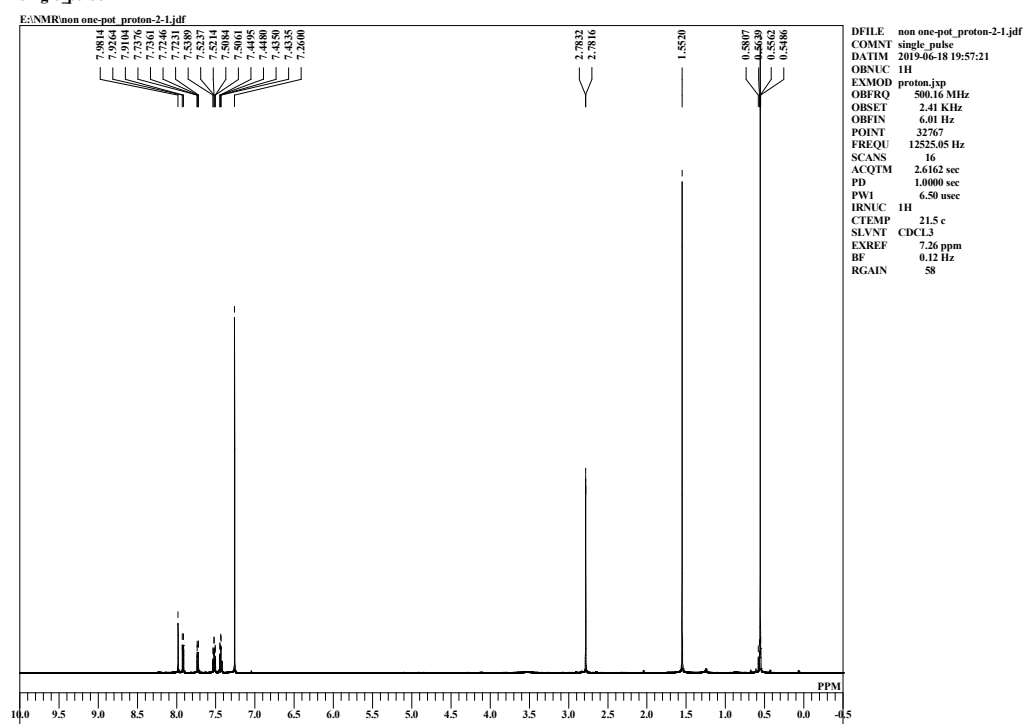
6a

single pulse decoupled gated NOE



7a

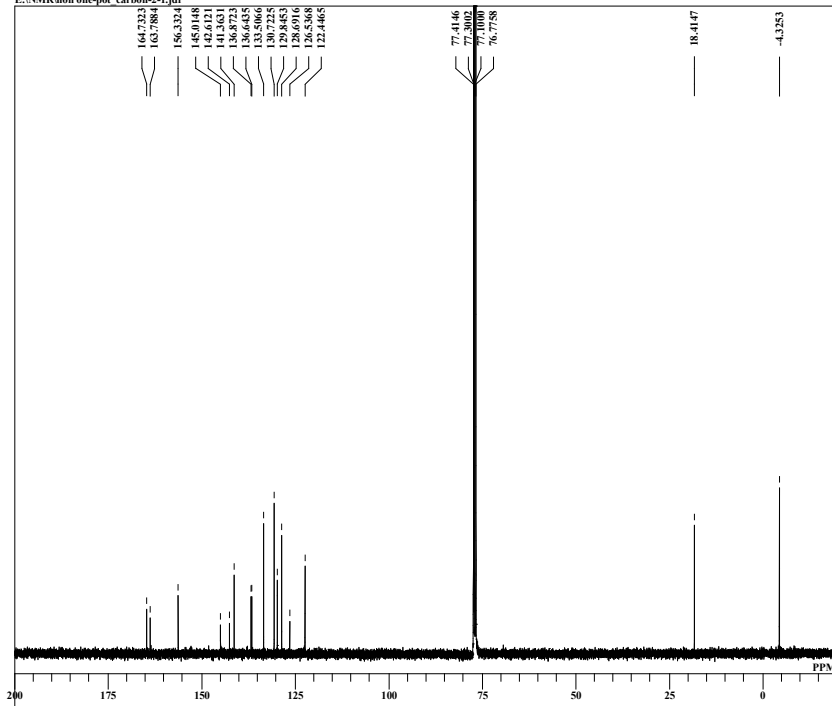
single pulse



7a

single pulse decoupled gated NOE

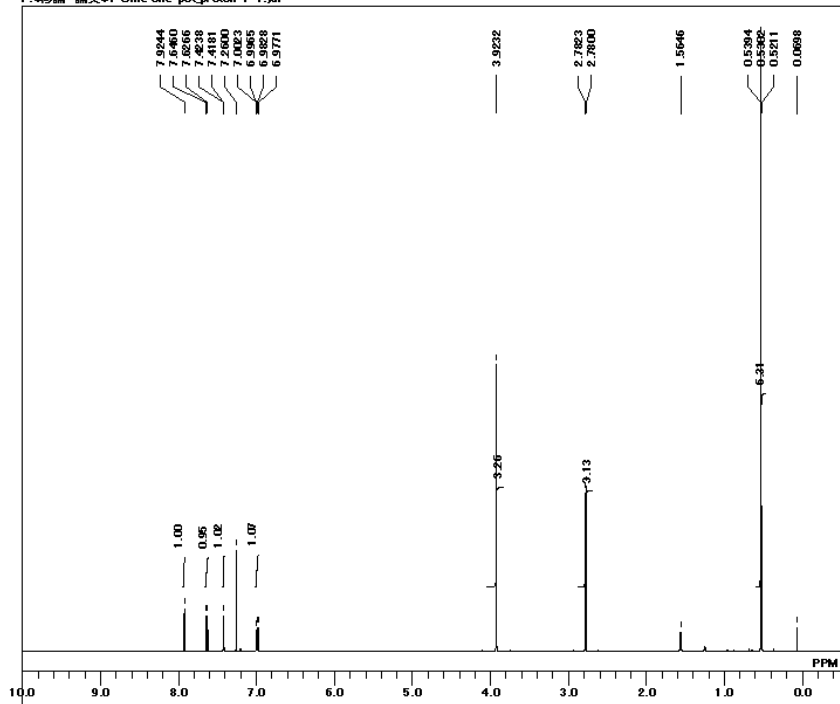
E:\NMR\non one-pot_carbon-2-1.jdf



DFILE non one-pot_carbon-2-1.jdf
 COMNT single pulse decoupled gated NOE
 DATIM 2019-06-30 11:15:39
 OBNUC ¹³C
 EXMOD carbon.jsp
 OBFREQ 100.53 MHz
 OBSET 5.35 KHz
 OBFIN 5.86 Hz
 POINT 32767
 FREQU 31407.04 Hz
 SCANS 26268
 ACQTM 1.0433 sec
 PD 2.0000 sec
 PW1 3.17 usec
 IRNUC ¹H
 CTEMP 22.0 c
 SLVNT CDCL₃
 EXREF 77.10 ppm
 BF 0.12 Hz
 RGAIN 60

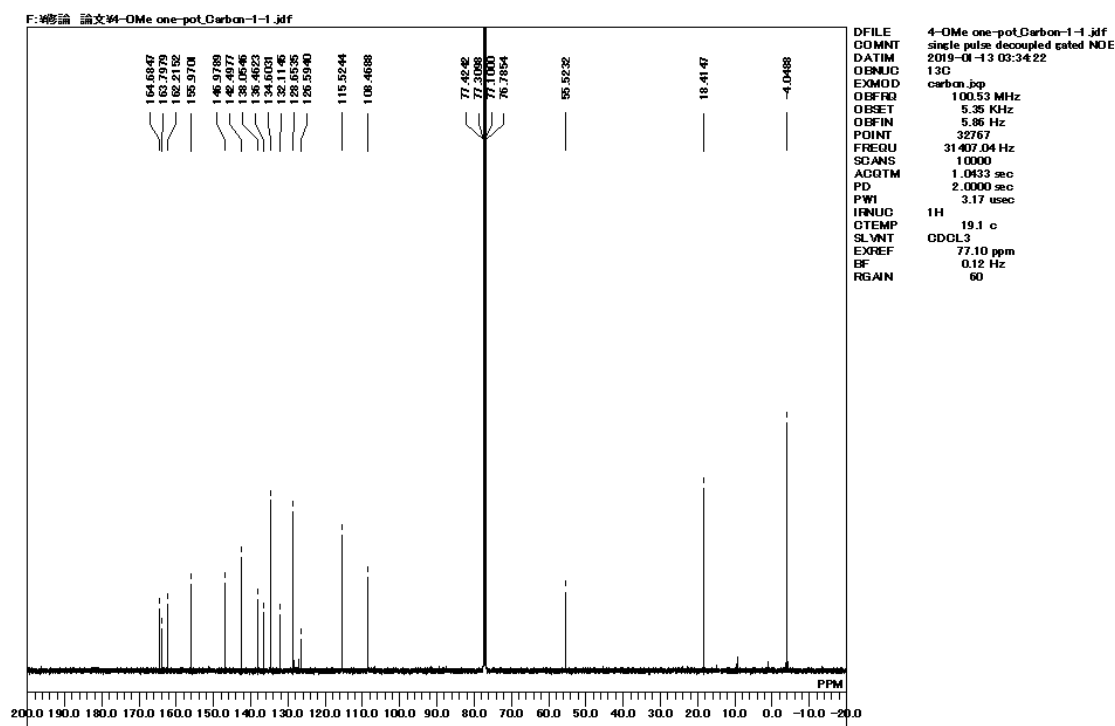
7b

F:\物論 論文\4-Ome one-pot_proton-1-1.jdf

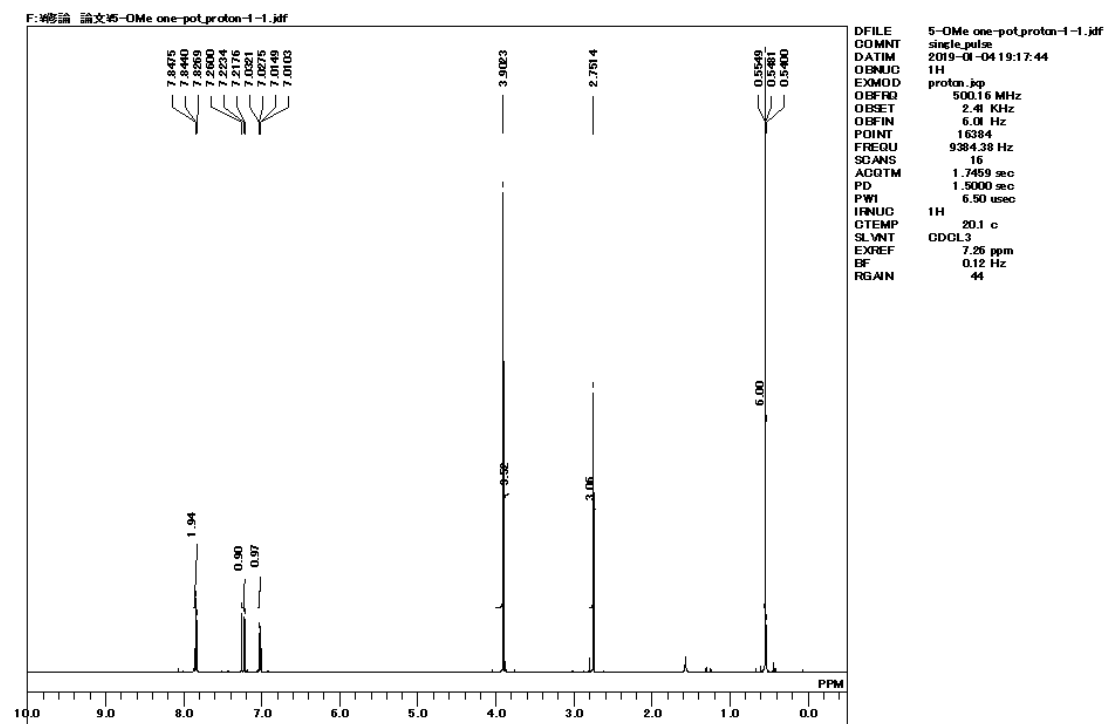


DFILE 4-Ome one-pot_proton-1-1.jdf
 COMNT single pulse
 DATIM 2019-06-13 03:31:27
 OBNUC ¹H
 EXMOD proton.jsp
 OBFREQ 399.78 MHz
 OBSET 4.19 KHz
 OBFIN 7.29 Hz
 POINT 16394
 FREQU 7503.00 Hz
 SCANS 16
 ACQTM 2.1837 sec
 PD 2.0000 sec
 PW1 7.29 usec
 IRNUC ¹H
 CTEMP 18.6 c
 SLVNT CDCL₃
 EXREF 7.26 ppm
 BF 0.12 Hz
 RGAIN 48

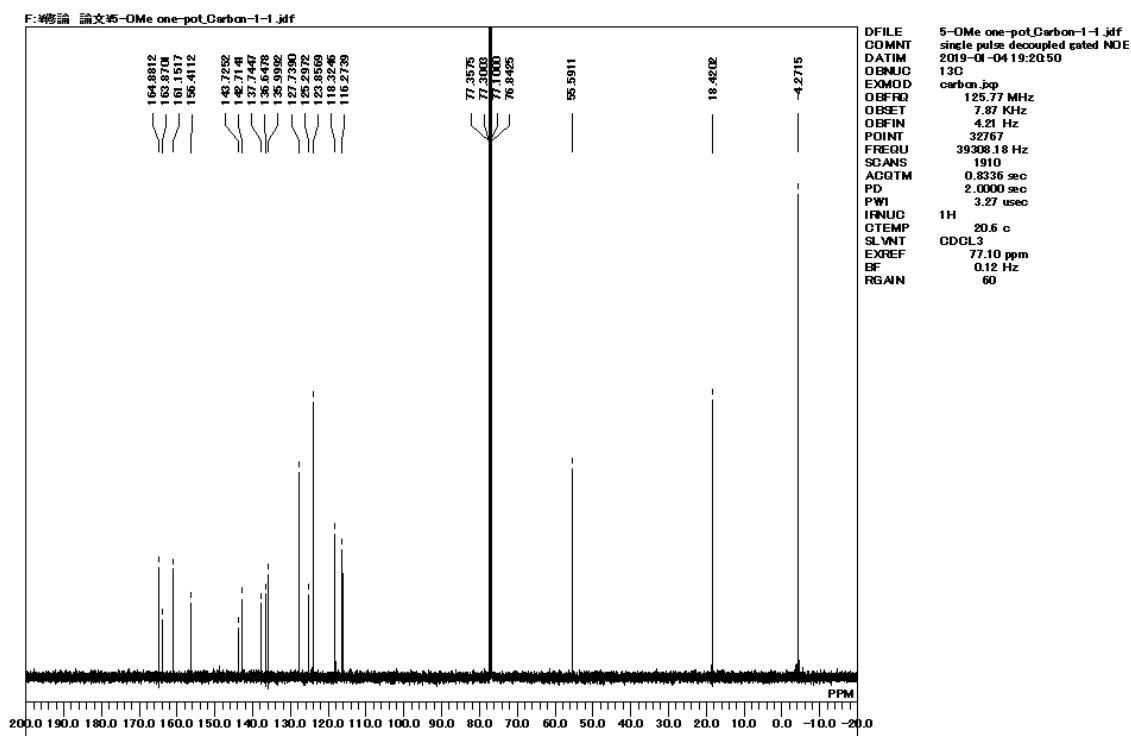
7b



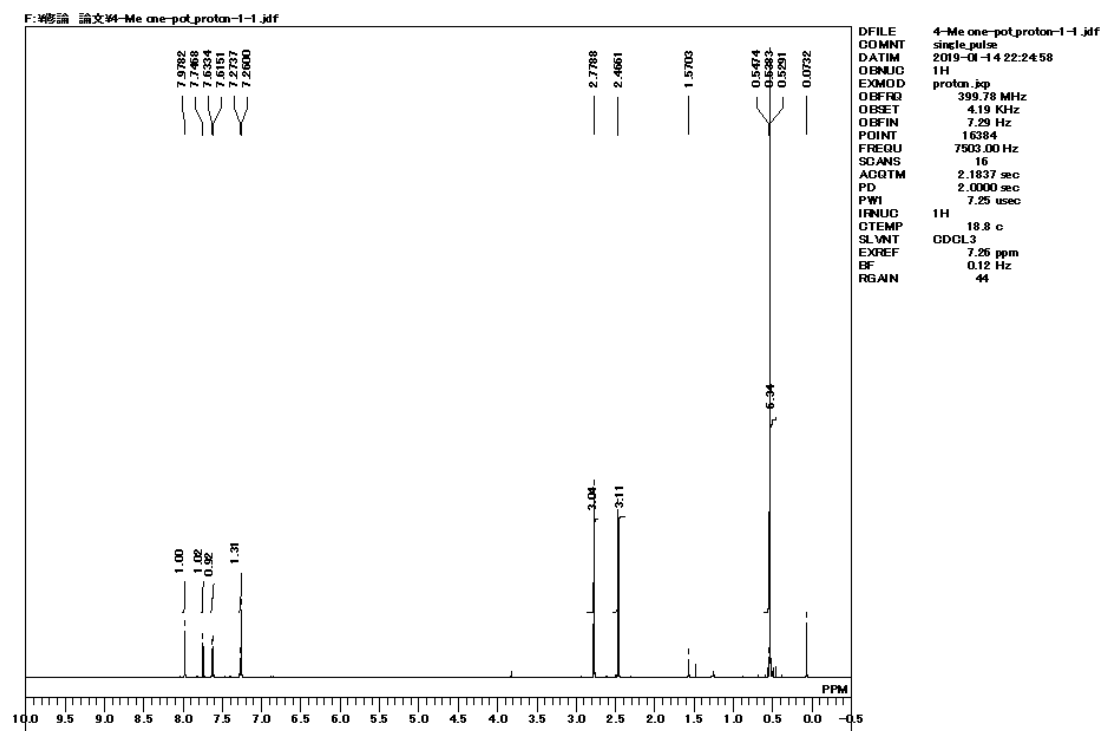
7c



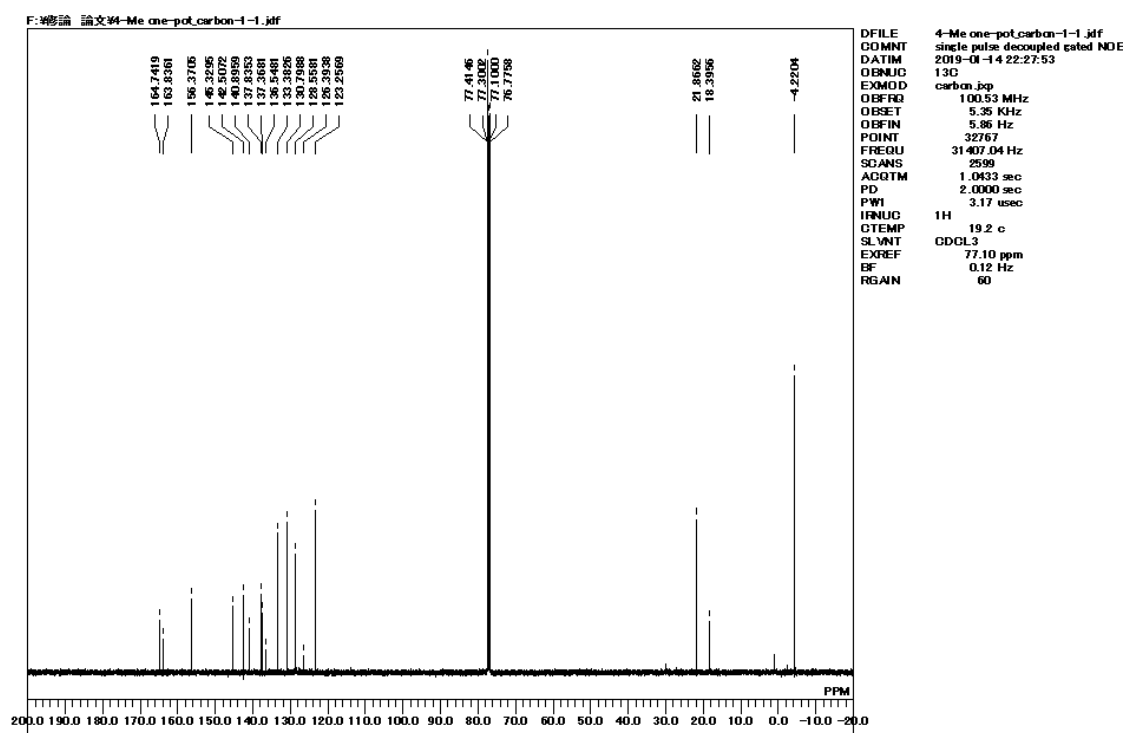
7c



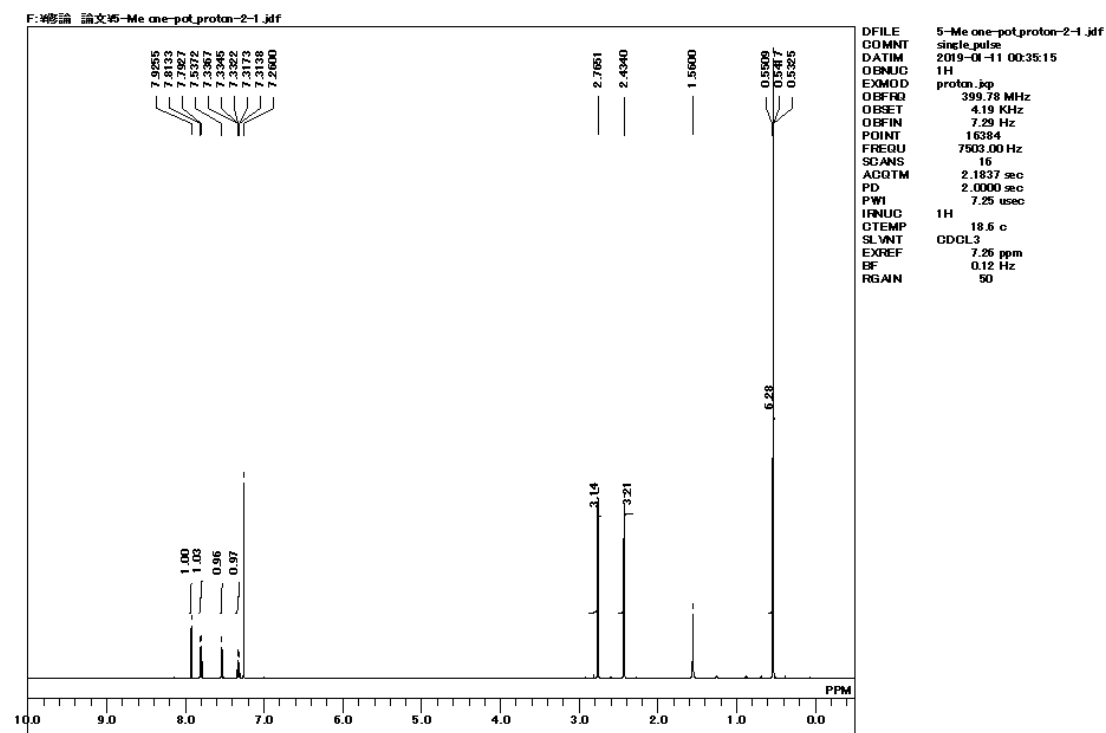
7d



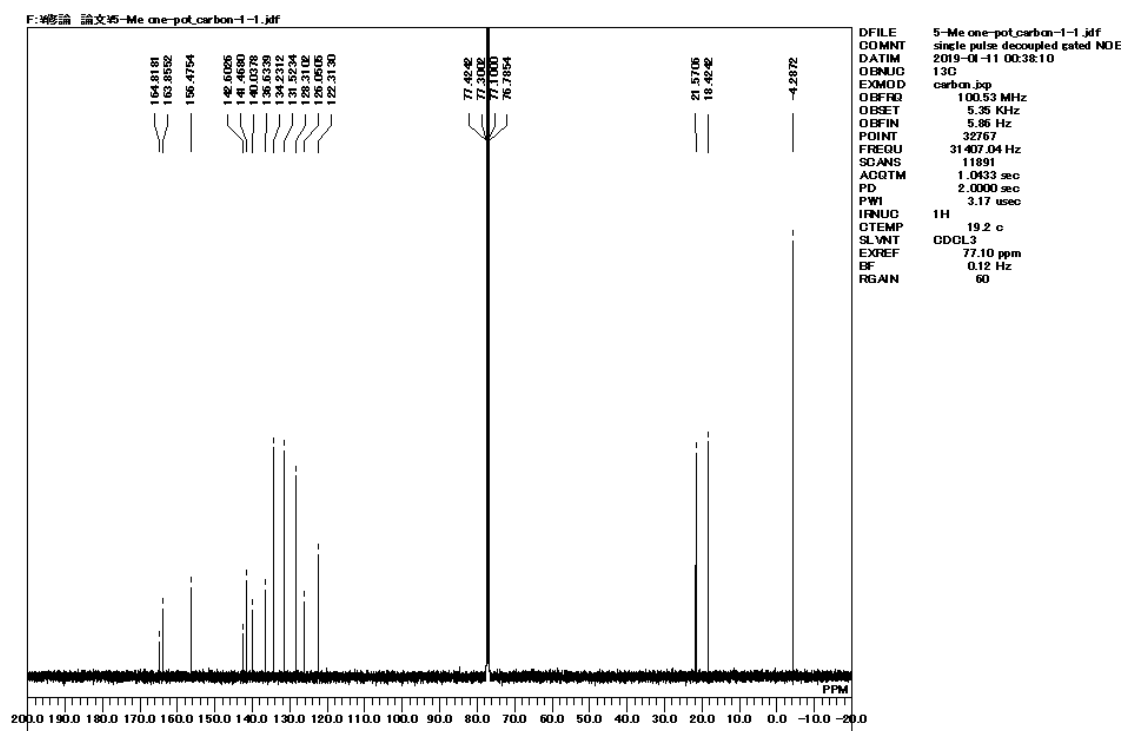
7d



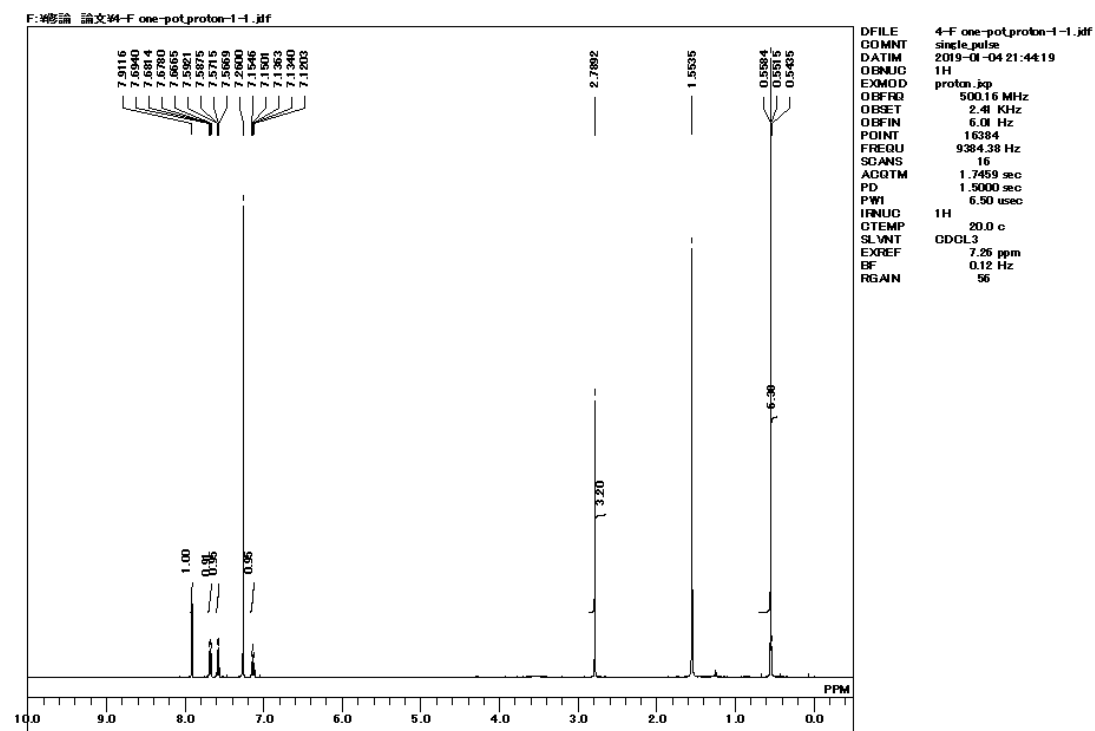
7e



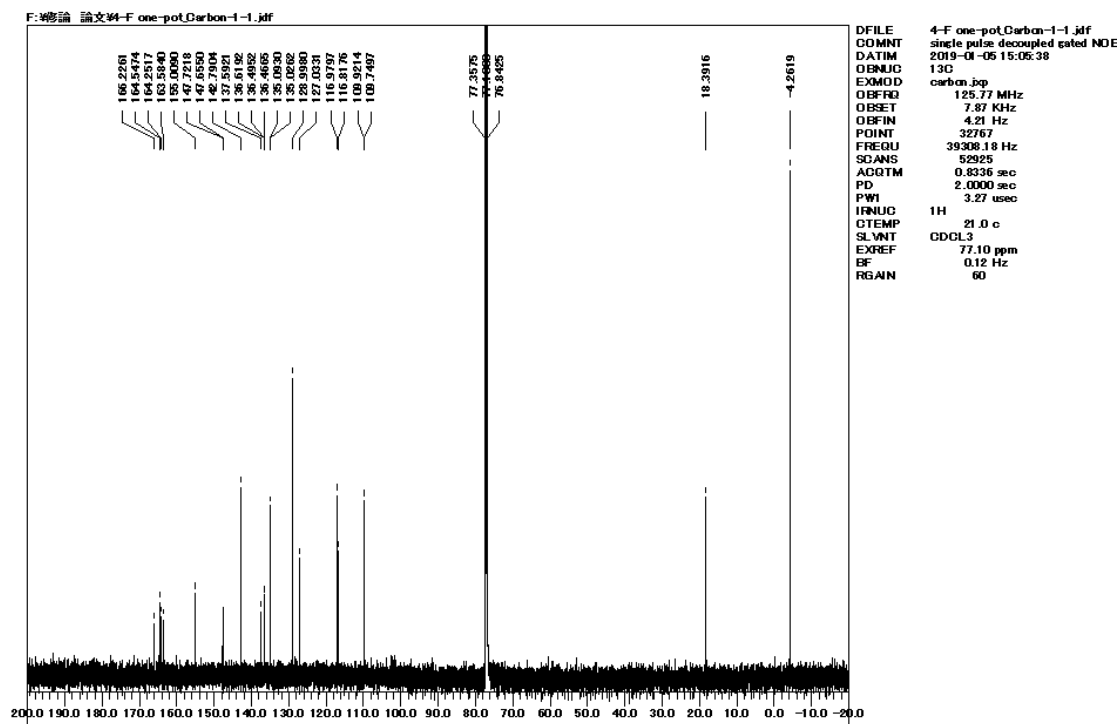
7e



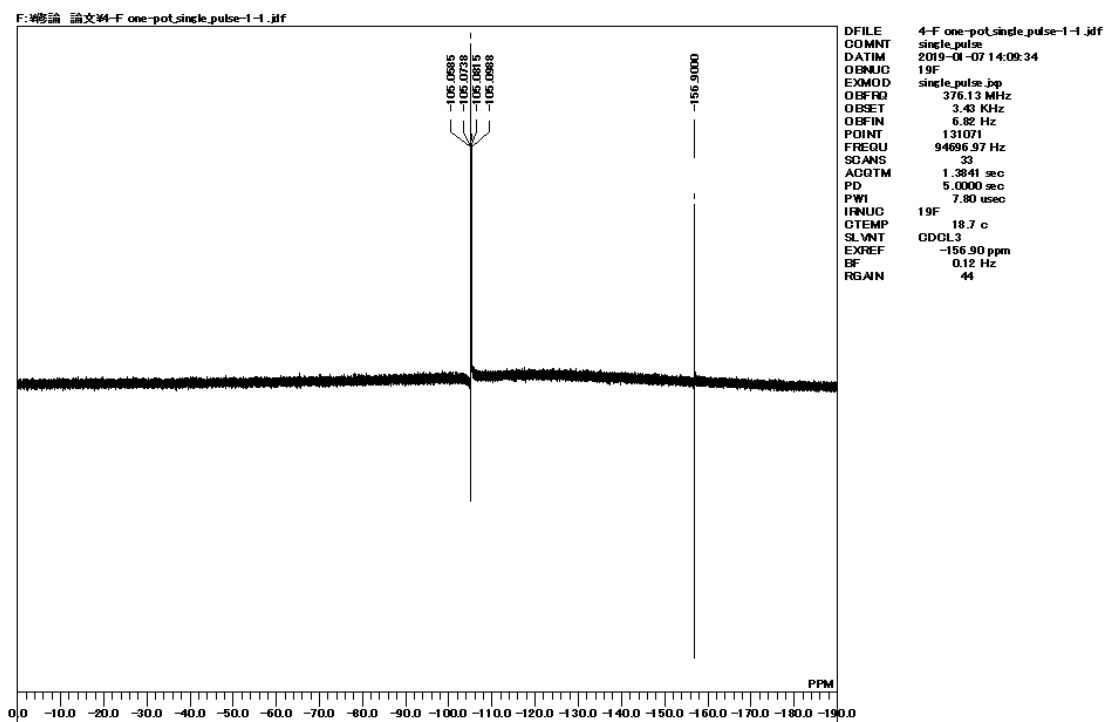
7f



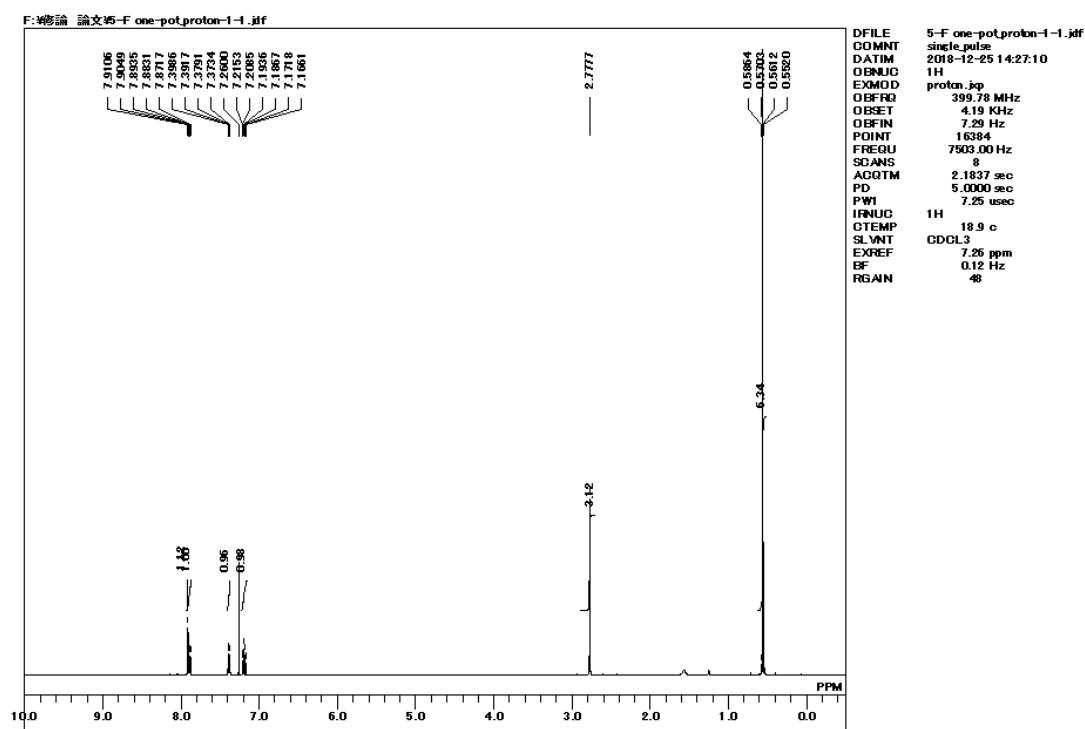
7f



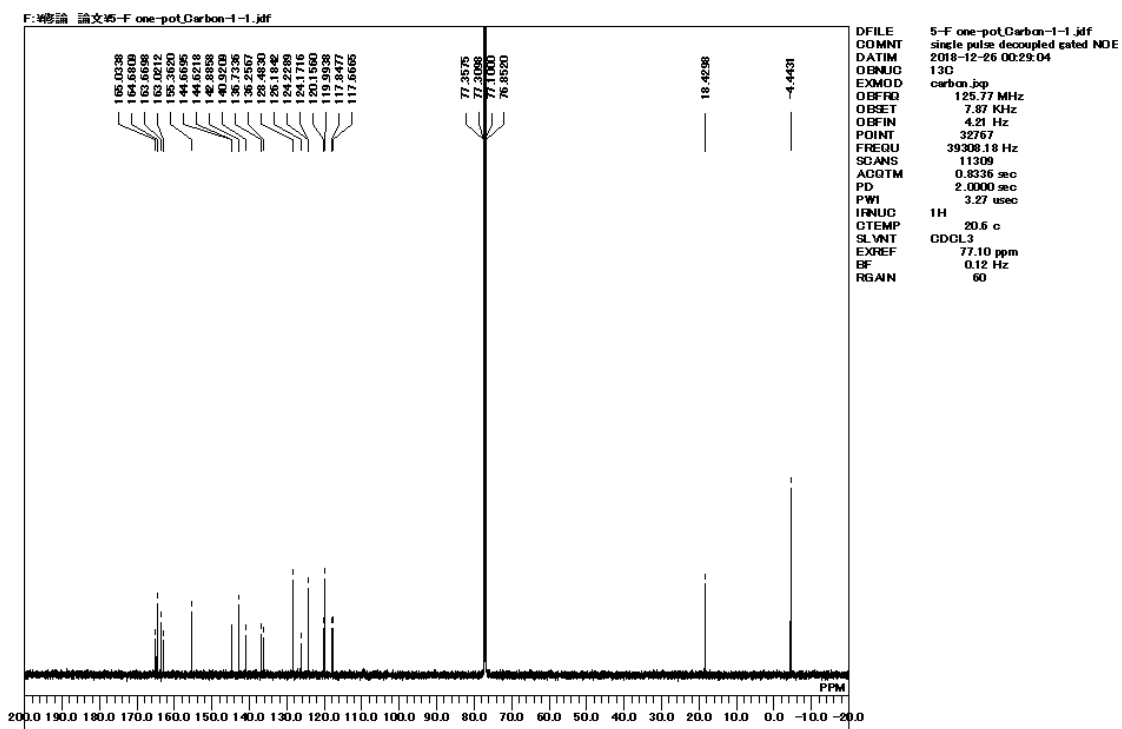
7f



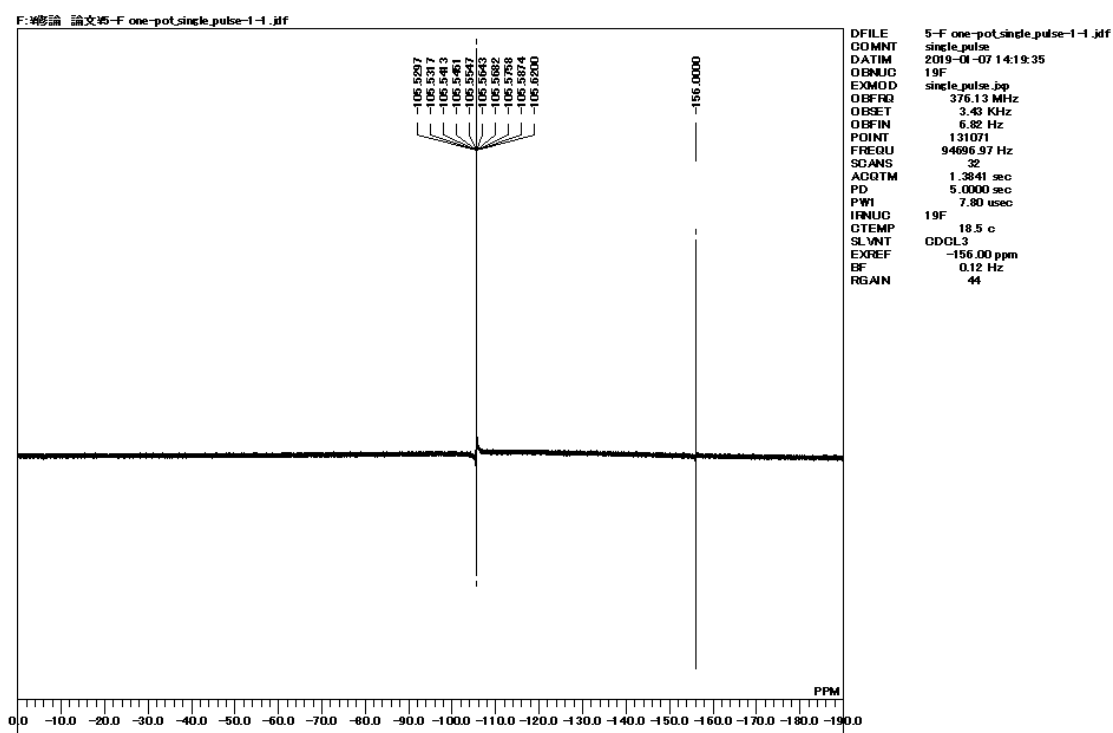
7g



7g



7g



Carbide complex

single_pulse

E:\carbide 2_proton-1-1.jdf

