Electronic Supplementary Information

Enyne Metathesis/Diels-Alder/Oxidation One-pot Reaction for 6-Membered Ring Silacycle Containing Multi-ring Core

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Contents

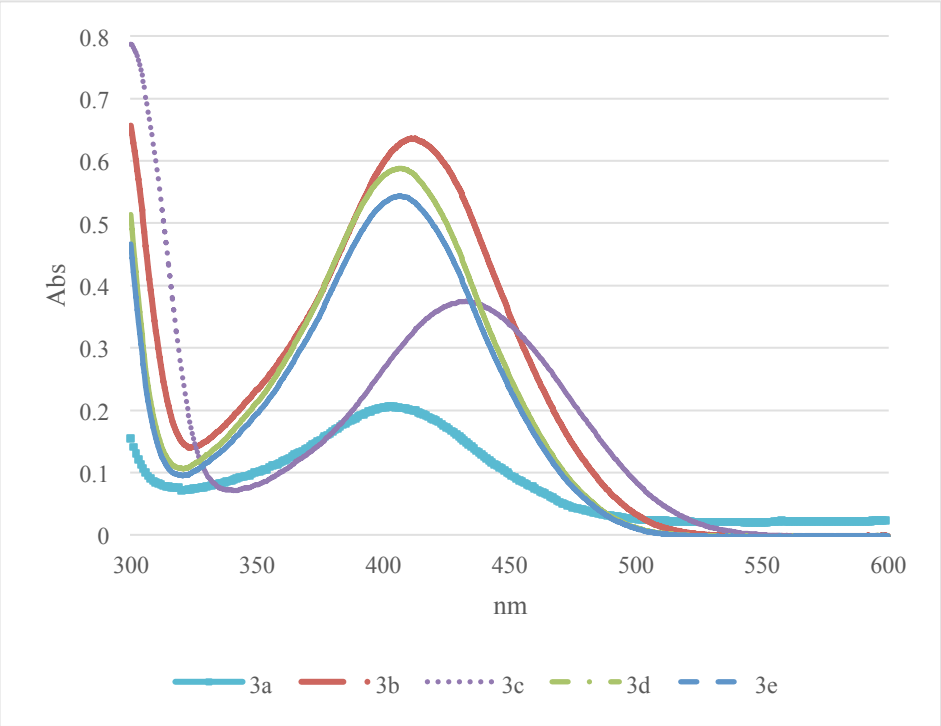
Synthesis and reaction

General

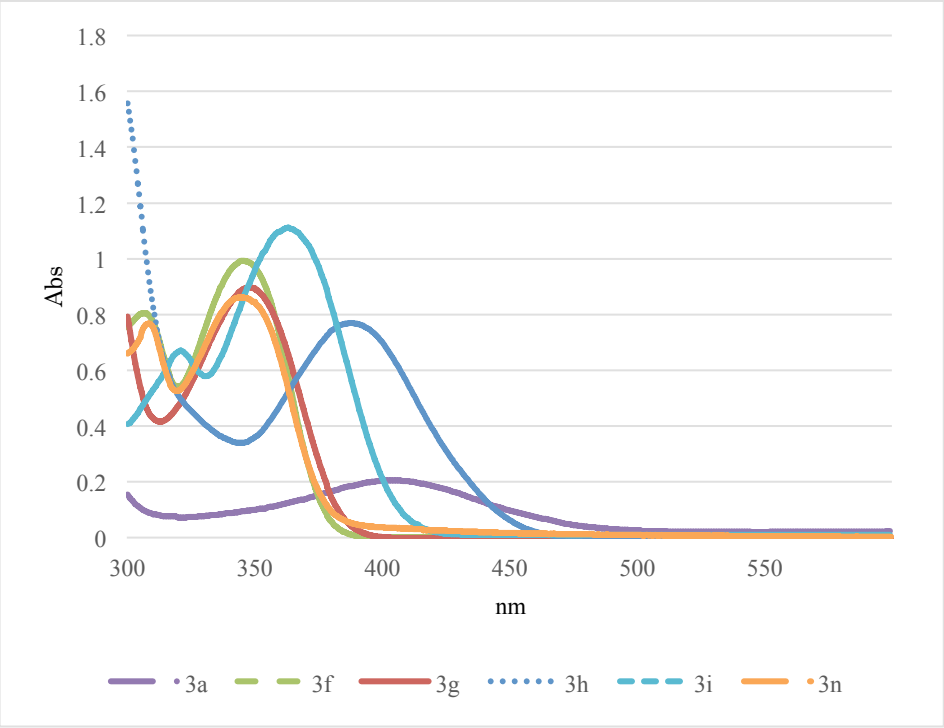
^{1}H NMR spectra were recorded in $CDCl_3$ at 25 °C unless otherwise noted, at 300, 400 or 500 MHz, with TMS as an internal standard. ^{13}C NMR spectra were recorded in $CDCl_3$ at 25 °C unless otherwise noted, at 300, 400 or 500MHz with TMS or $CDCl_3$ as an internal standard. ^{19}F NMR spectra were recorded in $CDCl_3$ at 25 °C unless otherwise noted, at 470 MHz, with C_6F_6 as an internal standard

Absorption and fluorescence data

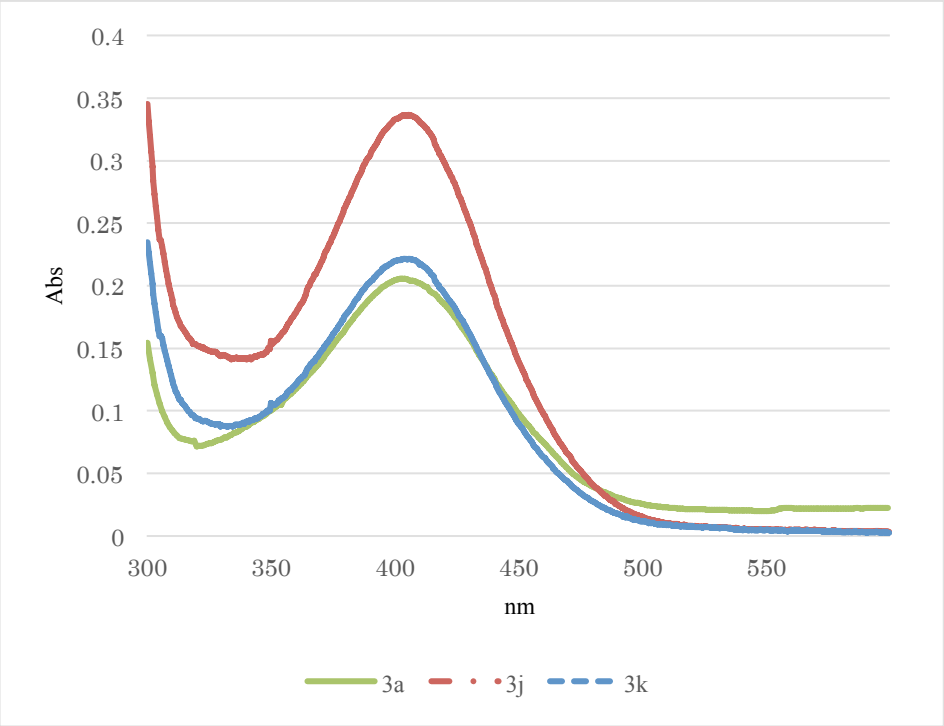
Figure S1. Absorption spectra of **3a–n** in $CHCl_3$ (0.1 mM).



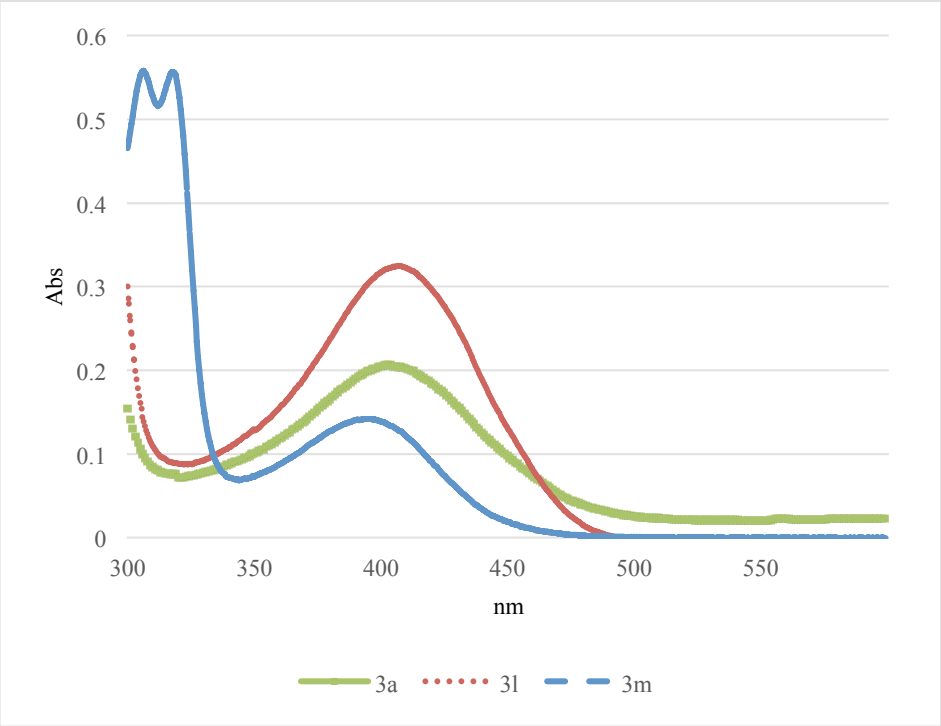
3a ε(404) = 2060, **3b** ε(411) = 6350, **3c** ε(433) = 3750, **3d** ε(407) = 5880, **3e** ε(406) = 5440



3a $\varepsilon(404) = 2060$, **3f** $\varepsilon(346) = 9930$, **3g** $\varepsilon(348) = 8990$, **3h** $\varepsilon(387) = 7690$, **3i** $\varepsilon(363) = 11100$, **3n** $\varepsilon(345) = 8620$

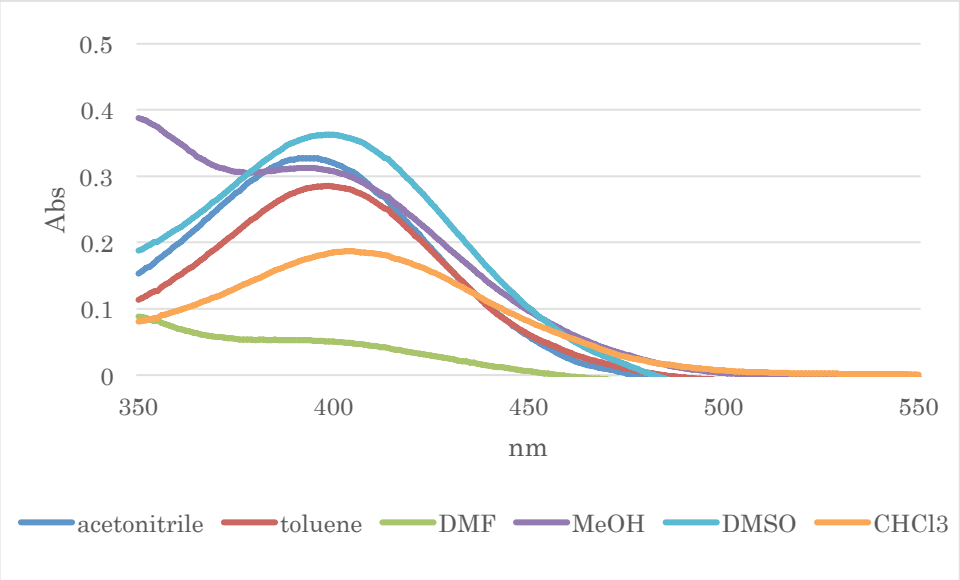


3a $\varepsilon(404) = 2060$, **3j** $\varepsilon(404) = 3360$, **3k** $\varepsilon(404) = 2220$



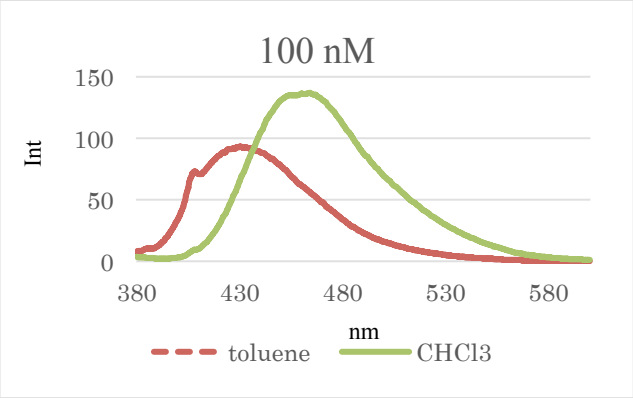
3a ε(404) = 2060, **3l** ε(407) = 3250, **3m** ε(394) = 1420

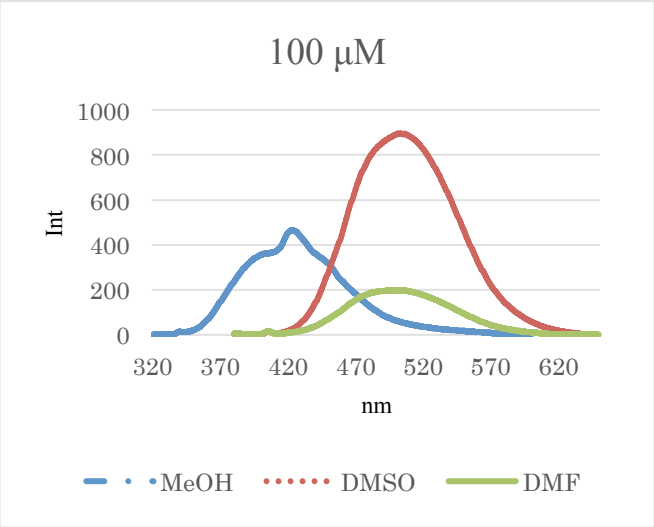
Figure S2. Absorption spectra of **3a** in various solvents (0.1 mM).

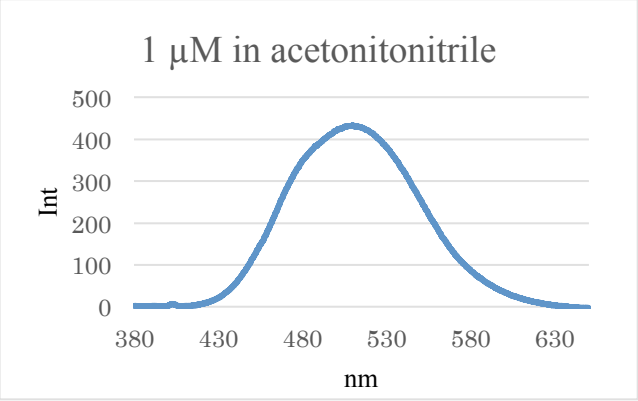


acetonitrile ε(394) = 3270, toluene ε(399) = 2850, DMF ε(392) = 528, MeOH ε(392) = 3120, DMSO ε(400)=3630, $CHCl_3$ ε(404)=1870

Figure S3. Fluorescence spectra of **3i** in various solvents.







toluene λ_{ex} = 362 nm, λ_{em} = 436 nm, Φ = 0.0418

$CHCl_3$ λ_{ex} = 363 nm, λ_{em} = 464 nm, Φ = 0.0727

MeOH λ_{ex} = 309 nm, λ_{em} = 423 nm, Φ = 0.000000134

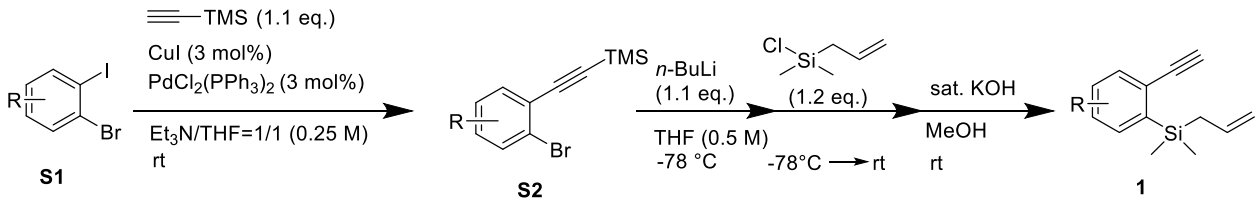
DMSO λ_{ex} = 361 nm, λ_{em} = 506 nm, Φ = 0.00000126

DMF λ_{ex} = 363 nm, λ_{em} = 502 nm, Φ = 0.000000266

acetonitrile λ_{ex} = 360.5 nm, λ_{em} = 510 nm, Φ = 0.0000310

Preparation of 1

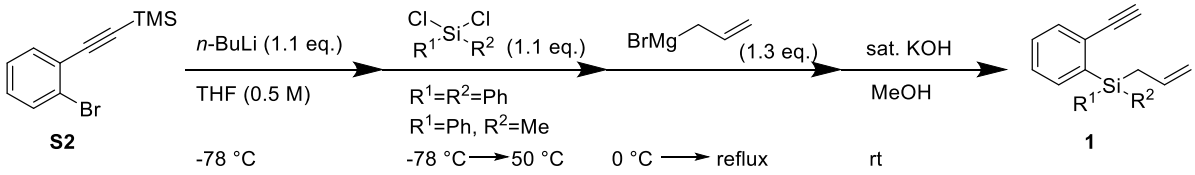
General procedure A



To a solution of **S1,** $PdCl_2(PPh_3)_2$ (3 mol %) and CuI (3 mol%) in Et_3N/THF = 1/1 (0.25 M) was added trimethylsilylacetylene (1.1 eq.) at room temperature. After the mixture was stirred for 2 h, the mixture was filtered through Celite cake and the solvent was evaporated under reduced pressure. The residue was subjected to column chromatography on silica gel to give **S2**.

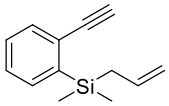
To a solution of compound **S2** in THF (0.5 M) was added dropwise *n*-BuLi (1.1 eq.) at -78 °C, After the mixture was stirred at -78 °C for 1 h, allylchlorodimethylsilane (1.2 eq.) was added dropwise to the mixture. The reaction mixture was stirred at -78 °C for 1 h, and warmed to room temperature. After then, saturated KOH in MeOH was added to the reaction mixture and the whole was stirred at room temperature for 10 min. The mixture was diluted with H_2O and organic compound was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, and the solvent was evaporated. The residue was subjected to column chromatography on silica gel to give **1**.

General procedure B



To a solution of **S2** in THF (0.5 M) was added dropwise *n*-BuLi (1.1 eq.) at -78 °C. After the mixture was stirred at -78 °C for 1 h, $R^1R^2SiCl_2$ (1.1 eq.) was added dropwise to the mixture. The reaction mixture was warmed to room temperature over 2 h, and heated over 4 h at 50 °C. After then, allylmagnesiumbromide in ethyl ether (1.3 eq.) was added dropwise to the mixtire at 0 °C, and the whole was refluxed for 18 h. Saturated KOH in MeOH was added to the reaction mixture. The mixture was diluted with H_2O, and the organic compound was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, and the solvent was evaporated. The residue was subjected to column chromatography on silica gel to give compound **1**.

1a: [1]

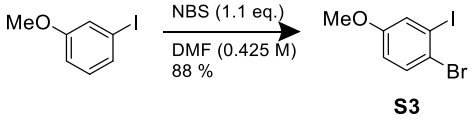


1a (93%, 936 mg, 4.67 mmol, pale yellow oil) was prepared from commercially available *o*-bromoiodobenzene (1.41 g, 5.00 mmol) by a general procedure **A**

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.54-7.51 (1H, m), 7.48-7.45 (1H, m), 7.33-7.30 (2H, m), 5.77 (1H, ddt, *J* = 17.5, 10.8, 8.3 Hz), 4.87 (1H, d, *J* = 17.5 Hz), 4.82 (1H, d, *J* = 10.8 Hz), 3.24 (1H, s), 1.96 (2H, d, *J* = 8.3 Hz), 0.37 (6H, s).

^{13}C-NMR ($CDCl_3$, 125 MHz) δ: 141.19, 134.79, 134.29, 133.36, 128.84, 127.90, 127.27, 113.33, 85.08, 80.35, 22.81, -3.28

Preparation of 1b:

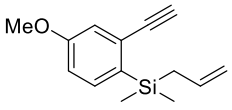


S3

S3 [2]

To a solution of commercially available 2-iodoanisole (4.68 g, 20.0 mmol) in DMF (47.1 mL, 0.425 M) was added in portions NBS (3.92 g, 22.0 mmol) at room temperature. The reaction mixture was heated to 80 °C and stirred for 4 h. The solution was cooled to room temperature, washed by water and the organic compound was extracted with AcOEt. After the mixture was dried over Na_2SO_4, the solvent was evaporated under reduced pressure. The residue was subjected to column chromatography on acid flash silica gel (*n*-hexane) to give **S3** (5.47 g, 17.5 mmol, 88%) as a yellow oil.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.47 (1H, d, *J* = 8.9 Hz), 7.38 (1H, d, *J* = 3.1 Hz), 6.77 (1H, dd, *J* = 8.9, 3.1 Hz), 3.77 (3H, s).



1b (49%, 1.95 g, 8.47 mmol, yellow oil) was prepared from **S3** (5.46 g, 17.4 mmol) by a general procedure **A**.

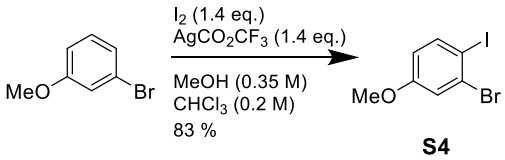
^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.37 (1H, d, *J* = 8.3 Hz), 7.07 (1H, d, *J* = 2.4 Hz), 6.88 (1H, dd, *J* = 8.3, 2.4 Hz), 5.77 (1H, ddt, *J* = 15.8, 9.9, 7.9 Hz), 4.86 (2H, d, *J* = 15.8 Hz), 4.81 (1H, d, *J* = 9.9 Hz), 3.79 (3H, s), 3.22 (1H, s), 1.93 (2H, d, *J* = 7.9 Hz), 0.35 (6H, s).

^{13}C-NMR ($CDCl_3$, 125 MHz) δ: 159.91, 135.79, 134.99, 132.23, 128.49, 118.50, 114.47, 113.17, 84.92, 80.06, 55.10, 23.04, -3.13

HRMS (EI) calcd for $C_{14}H_{18}OSi$: 230.1127, found 230.1151

Anal. calcd for $C_{14}H_{18}O_2Si$: C, 72.99; H, 7.88, found: C, 72.99; H, 7.96

Preparation of 1c:

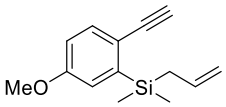


S4

S4 [3]

Reaction equipment was covered with aluminium foil. To a solution of iodine (3.55 g, 14.0 mmol) in $CHCl_3$ (28.6 mL, 0.35 M) was added dropwise commercially available 3-bromoanisole (1.26 mL, 10.0 mmol). To a stirring mixture was added silver trifluoroacetate (3.09 g, 14.0 mmol) in MeOH (50 mL, 0.2 M) at 0 °C. After warmed to room temperature, the mixture was stirred for four days. The reaction was stopped by sat. Na_2SO_3 aq, and the organic compound was extracted with $CHCl_3$ and the organic layer was dried over Na_2SO_4. After the solvent was evaporated by reduced pressure, the residue was subjected to column chromatography on acid flash silica gel (hexane/AcOEt = 50/1) to give **S4** (83%, 2.59 g, 8.29 mmol) as a yellow oil.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.69 (1H, d, *J* = 8.6 Hz), 7.19 (1H, d, *J* = 2.8 Hz), 6.60 (1H, dd, *J* = 8.6, 2.8 Hz), 3.78 (3H, s).



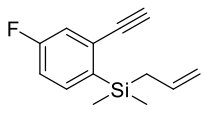
1c (quant, 691 mg, 3.00 mmol, yellow oil) was prepared from **S4** (849 mg, 3.00 mmol) by a general procedure **A**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.48 (1H, d, *J* = 8.4 Hz), 6.99 (1H, d, *J* = 2.9 Hz), 6.83 (1H, dd, *J* = 8.4, 2.9 Hz), 5.78 (1H, ddt, *J* = 16.9, 9.9, 8.1 Hz), 4.88 (1H, d, *J* = 16.9 Hz), 4.83 (1H, d, *J* = 9.9 Hz), 3.82 (4H, s), 3.16 (1H, s), 1.96 (2H, d, *J* = 8.1 Hz), 0.37 (6H, s).

^{13}C-NMR ($CDCl_3$, 125 MHz) δ:160.16, 144.33, 136.24, 135.95, 121.71, 120.41, 114.76, 114.57, 86.27, 80.09, 56.29, 23.94, -2.16

HRMS (EI) calcd for $C_{14}H_{18}OSi$: 230.1127, found 230.1120

1d:



1d (54%, 592 mg, 2.71 mmol, colorless oil) was prepared from commercially available 1-bromo-4-fluoro-2-iodobenzene (1.50 g, 5.00 mmol) by a general procedure **A**.

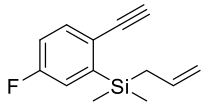
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 7.41 (1H, dd, *J* = 8.4, 6.6 Hz), 7.22 (1H, dd, *J* = 9.7, 2.4 Hz), 7.01 (1H, ddd, *J* = 8.8, 8.4, 2.4 Hz), 5.75 (1H, ddt, *J* = 17.4, 11.0, 8.1 Hz), 4.86 (1H, d, *J* = 17.4 Hz), 4.82 (1H, d, *J* = 11.0 Hz), 3.27 (1H, s), 1.94 (2H, d, *J* = 8.1 Hz), 0.36 (6H, s)

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 162.96 (d, *J* = 247.0 Hz), 136.90 (d, *J* = 3.8 Hz), 136.30 (d, *J* = 8.6 Hz), 134.54, 129.25, 120.24 (d, *J* = 21.0 Hz), 115.40 (d, *J* = 19.1 Hz), 113.52, 83.87 (d, *J* = 3.7 Hz), 81.26, 22.80, -3.22

^{19}F-NMR ($CDCl_3$, 470 MHz) δ: -115.69

HRMS (EI) calcd for $C_{13}H_{15}FSi$: 218.0927, found 218.0927

1e:



1e (43%, 282 mg, 1.29 mmol, yellow oil) was prepared from commercially available 2-bromo-4-fluoro-1-iodobenzene (903 mg, 3.00 mmol) by a general procedure **A**.

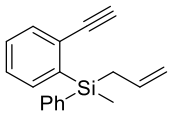
^{1}H-NMR ($CDCl_3$: 400 MHz) δ: 7.51 (1H, dd, *J* = 8.5, 5.3 Hz), 7.14 (1H, dd, *J* = 9.2, 2.7 Hz), 6.99 (1H, ddd, *J* = 8.5, 8.5, 2.7 Hz), 5.75 (1H, ddt, *J* = 16.9, 10.0, 8.2 Hz), 4.87 (1H, d, *J* = 16.9 Hz), 4.83 (1H, d, *J* = 10.0 Hz), 3.21 (1H, s), 1.96 (2H, d, *J* = 8.2 Hz), 0.37 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 162.21 (*J* = 201.3 Hz), 144.78, 135.48 (d, *J* = 5.7 Hz), 134.33, 123.13 (d, *J* = 2.9 Hz), 121.13 (d, *J* = 12.5 Hz), 115.99 (d, *J* = 9.0 Hz), 113.69, 84.16, 79.98, 22.48, -3.49

^{19}F-NMR ($CDCl_3$, 470 MHz) δ: -114.37

HRMS (EI) calcd for $C_{13}H_{15}FSi$: 218.0927, found 218.0927

1j:



1j (94%, 757 mg, 2.88 mmol, yellow oil) was prepared from **S2** (760 mg, 3.00 mmol) and $MePhSiCl_2$ (536 μL, 3.3 mmol) by a general procedure **B**.

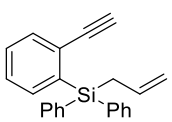
^{1}H-NMR ($CDCl_3$, 500 MHz) δ: 7.54-7.52 (3H, m), 7.39-7.26 (6H, m), 5.79 (1H, ddt, *J* = 17.0, 10.0, 7.7 Hz), 4.92 (1H, d, *J* = 17.0 Hz), 4.84 (1H, d, *J* = 10.0 Hz), 3.11 (1H, s), 2.36 (1H, dd, *J* = 13.7, 7.7 Hz), 2.22 (1H, dd, *J* = 13.7, 7.7 Hz), 0.66 (3H, s).

^{13}C-NMR ($CDCl_3$, 125 MHz) δ: 139.34, 136.30, 135.52, 134.64, 134.32, 133.52, 129.15, 127.92, 127.76, 127.65, 114.18, 84.92, 80.82, 21.64, -4.54

HRMS (EI) calcd for $C_{18}H_{18}Si$: 262.1178, found 262.1159

Anal. calcd for $C_{18}H_{18}Si$: H, 6.91; C, 82.38, found: H, 7.05 ; C, 82.52

1k:



1k (74%, 719 mg, 2.22 mmol, white solid) was prepared from **S2** (760 mg, 3.00 mmol) and Ph_2SiCl_2 (685 μL, 3.3 mmol) by a general procedure **B**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.56-7.53 (5H, m), 7.40-7.30 (9H, m), 5.91 (1H, ddt, *J* = 16.9, 10.3, 8.0 Hz), 4.99 (1H, ddt, *J* = 16.9, 1.8, 1.5 Hz), 4.88 (1H, ddt, *J* = 10.3, 1.8, 1.1 Hz), 2.94 (1H, s), 2.62 (2H, ddd, *J* = 8.0, 1.5, 1.1 Hz).

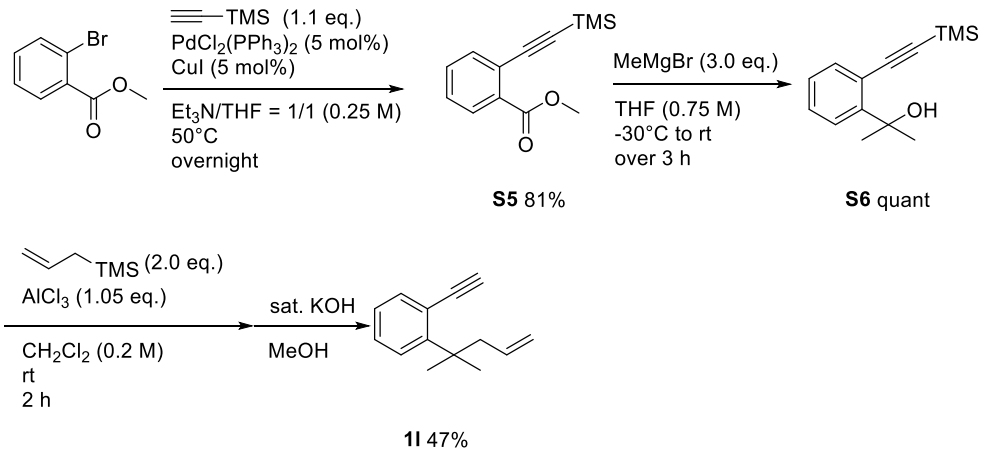
^{13}C-NMR ($CDCl_3$, 125 MHz) δ:137.87, 137.47, 136.14, 134.89, 134.72, 133.94, 129.80, 129.72, 128.74, 128.30, 128.00, 115.22, 85.08, 81.67, 21.55

HRMS (MALDI) calcd for $C_{23}H_{20}Si$: 347.12265 ([M+Na^+]), found 347.12271 ([M+Na^+])

Anal. calcd for $C_{23}H_{20}Si$: H, 6.21; C, 85.13, found: H, 6.49 ; C, 85.36

m.p. 67.0-68.5 °C (from $CHCl_3$, a colorless column)

1l:



To a solution of commercially available methyl *o*-bromoacetate (701 μL, 5.00 mmol), $PdCl_2(PPh_3)_2$ (176 mg, 0.250 mmol) and CuI (47.6 mg, 0.250 mmol) in Et_3N/THF=1/1 (0.25 M, 20 mL) was added trimethylsilylacetylene (830 μL, 6.0 mmol) at room temperature and the reaction mixture was heated to 50 °C. After the mixture was stirred overnight, 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 (*n*-hexane / AcOEt = 20 / 1) to give compound **S5** (81%, 943 mg, 4.06 mmol).

To a solution of compound **S5** (943 mg, 4.06 mmol) in THF (0.75 M, 5.4 mL) was added dropwise MeMgBr in THF (0.98 M; 12.4 mL, 12.2 mmol) at -30°C and the mixture was warmed to room temperature over 2 h and heated at 50 °C for 1 h. After saturated NH_4Cl aq was added to the reaction mixture, organic compound was extracted with AcOEt and the organic layer was washed with brine. The organic layer was dried over Na_2SO_4, and the solvent was evaporated. The residue was subjected to column chromatography on neutral flash silica gel (*n*-hexane / AcOEt = 10 / 1) to give compound **S6** (quant, 944 mg, 4.06 mmol).

To a solution of compound **S6** (930 mg, 4.00 mmol) in CH_2Cl_2 (0.2 M, 20 mL) was added $AlCl_3$ (560.0 mg, 4.20 mmol) and allyltrimethylsilane (1.40 mL, 8.80 mmol). After the mixture was stirred at room temperature for 1 h,

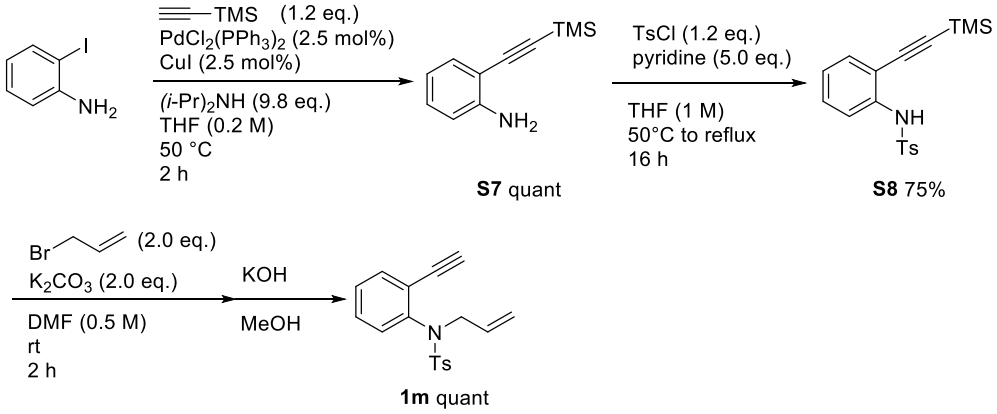
saturated KOH in MeOH was added to the reaction mixture and that was stirred at room temperature for 10 min. The mixture was diluted with water and organic compound was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, and the solvent was evaporated. The residue was subjected to column chromatography on neutral flash silica gel (*n*-hexane) to give compound **S7** (47%, 347 mg, 1.88 mmol).

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.53 (1H, dd, *J* = 7.3, 1.4 Hz), 7.30-7.28 (2H, m), 7.15 (1H, ddd, *J* = 8.2, 6.2, 2.4 Hz), 5.51 (1H, ddt, *J* = 16.8, 10.0, 7.6 Hz), 5.02-4.94 (1H, m), 4.92-4.88 (1H, m), 3.42 (1H, s), 2.82 (2H, d, *J* = 7.6 Hz), 1.47 (3H×2, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 150.36, 136.26, 135.87, 128.75, 126.68, 125.70, 120.50, 116.68, 85.18, 83.37, 45.33, 38.93, 20.05

HRMS (EI) calcd for $C_{14}H_{16}$: 184.1252, found 184.1255

1m: [4]



To a solution of commercially available *o*-bromoaniline (4.38 g, 20.0 mmol) $PdCl_2(PPh_3)_2$ (351 mg, 0.500 mmol), CuI (95.2 mg, 0.500 mmol) and $(i\text{-Pr})_2$NH (8 mL, 196 mmol) in THF (100 mL, 0.200 M) was added trimethylsilylacetylene (3.32 mL, 24.0 mmol) at room temperature and the reaction mixture was heated to 50 °C. After the mixture was stirred for 2 h, the mixture was filtered through Celite cake and the solvent was evaporated under reduced pressure. The residue was subjected to column chromatography on neutral silica gel (*n*-hexane / AcOEt = 10 / 1) to give compound **S7** (quant, 3.79 g, 20.0 mmol).

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.29 (1H, dd, *J* = 7.7, 1.5 Hz), 7.11 (1H, ddd, *J* = 8.8, 7.7, 1.5 Hz), 6.70-6.63 (2H, m), 4.22 (2H, s), 0.26 (9H, s).

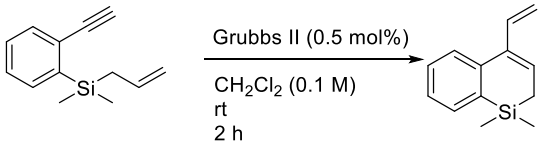
To a solution of pyridine (4.03 mL, 50.0 mmol) and **S7** (1.89 mg, 10.0 mmol) in THF (1 M, 10.0 mL) was added TsCl (2.29 g, 12.0 mmol) at room temperature and the whole was refluxed for 16 h. Then the reaction mixture was quenched by the addition of 1 M HCl aq and the organic compound was extracted wth AcOEt. The organic layer was dried over Na_2SO_4 and the solvent was evaporated. The residue was subjected to column chromatography on neutral flash silica gel (*n*-hexane/AcOEt = 10/1) to give compound **S8** (75%, 2.58 g, 7.52 mmol).

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.64-7.61 (3H, m), 7.31-7.24 (2H, m), 7.21-7.19 (3H, m), 7.00 (1H, ddd, *J* = 7.6, 7.6, 1.1 Hz), 2.37 (3H, s), 0.27 (9H, s).

To a solution of **S8** (172 mg, 0.500 mmol) and K_2CO_3 (138 mg, 1.00 mmol) in DMF (0.5 M, 1.00 mL) was added to allylbromide (84.6 μL, 1.00 mmol) at room temperature and stirred for 2 h. After then, sat.KOH in MeOH was added to the reaction mixture and that was stirred at room temperature for 10 min. The reaction mixture was quenched by sat.NH_4Claq and the organic compound was extracted with Et_2O and the organic layer was washed with brine. The organic layer was dried over Na_2SO_4 and the solvent was evaporated. The residue was subjected to column chromatography on neutral silica gel (*n*-hexane/AcOEt = 10/1) to give compound **1m** (quant, 156 mg, 0.500 mmol).

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.61 (2H, d, *J* = 8.3 Hz), 7.43 (1H, d, *J* = 7.6 Hz), 7.30-7.29 (2H, m), 7.25-7.22 (3H, m), 5.84 (2H, ddt, *J* = 18.2, 10.3, 6.5 Hz), 5.09 (1H, d, *J* = 18.2 Hz), 5.04 (1H, d, *J* = 10.3 Hz), 4.31 (2H, d, *J* = 6.5 Hz), 2.96 (1H, s), 2.41 (3H, s).

Preparation of 2a from 1a



1a **2a**

2a:

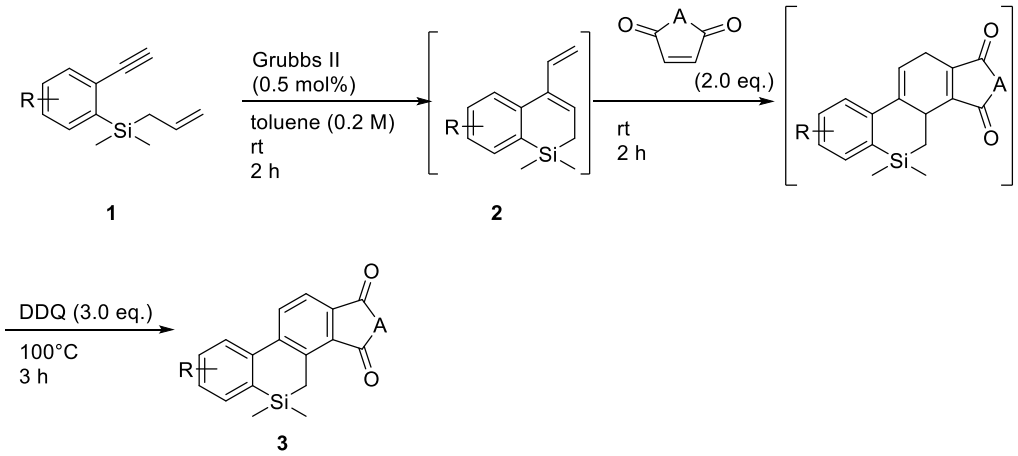
To a solution of **1a** (19.2 mg, 0.0960 mmol) in CH_2Cl_2 (1 mL) was added Grubbs II (0.9 mg, 1 mol%) and the mixture was stirred at room temperature for 1 h. The solvent was evaporated and the residue was subjected to column chromatography on acid flash silica gel (*n*-hexane) to give **2a** (90%, 17.2 mg, 0.0860 mmol) as a yellow oil.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.52-7.49 (1H, m), 7.38-7.33 (2H, m), 7.26-7.21 (1H, m), 6.58 (1H, dd, *J* = 17.0, 10.7 Hz), 6.29 (1H, t, *J* = 6.2 Hz), 5.43 (1H, dd, *J* = 17.0, 1.7 Hz), 5.12 (1H, dd, *J* = 10.7, 1.7 Hz), 2.96 (1H, s), 1.62 (2H, d, *J* = 6.2 Hz), 0.28 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 142.13, 139.14, 138.72, 134.95, 132.63, 129.46, 126.49, 126.15, 125.67, 114.44, 13.69, -3.59

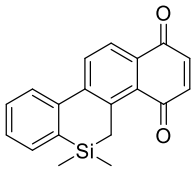
Anal. calcd for $C_{13}H_{16}Si$: C, 77.93 ; H, 8.05, found: C, 77.82 ; H, 8.30

One-pot preparation of 3 from 1 (Genaral Procedure C)



To a solution of **1** in toluene (0.2 M) was added Grubbs II (0.5 mol%) and the mixture was stirred at room temperature for 2 h. Then dienophile (2.0 eq.) was added to the mixture and the mixture was stirred at room temperature for 2 h. 2,3-dichloro-5,6-dicyano-*p*-benzoquinone (3.0 eq.) was added to the reaction mixture and the mixture was stirred at 100°C for 3 h. The solvent was evaporated and the residue was subjected to column chromatography on neutral flash silica gel to give **3**.

3a:



3a (78%, 23.8 mg, 0.0783 mmol, orange solid) was prepared from **1a** (20.0 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 8.03 (1H, d, *J* = 8.0 Hz), 7.87 (1H, d, *J* = 8.0 Hz), 7.66 (1H, d, *J* = 8.0 Hz), 7.58 (1H, dd, *J* = 7.6, 1.1 Hz), 7.53 (1H, ddd, *J* = 7.6, 7.6, 1.1 Hz), 7.40 (1H, ddd, *J* = 7.6, 7.6, 1.1 Hz), 6.92 (2H, m), 3.09 (2H, s), 0.23 (6H, s).

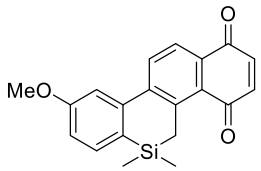
^{13}C-NMR ($CDCl_3$, 125 MHz) δ: 188.15, 185.16, 145.32, 143.16, 141.46, 141.16, 137.05, 136.37, 133.02, 132.38, 132.26, 130.42, 129.88, 128.04, 127.34, 124.83, 16.62, -4.67

HRMS (MALDI) calcd for $C_{19}H_{16}O_2Si$: 304.09141, found 304.09098

Anal. calcd for $C_{19}H_{16}O_2Si$: H, 5.30; C, 74.97, found: H, 5.41; C,74.87

m.p. 163.5-164.0 °C (recrystallized from $CHCl_3$; yellow needle)

3b:



3b (71%, 23.7 mg, 0.0709 mmol, orange solid) was prepared from **1b** (20.0 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 500 MHz) δ: 8.03 (1H, d, *J* = 8.0 Hz), 7.87 (1H, d, *J* = 8.0 Hz), 7.51 (1H, d, *J* = 8.0 Hz), 7.21 (1H, d, *J* = 2.3 Hz), 6.96 (1H, dd, *J* = 8.0, 2.3 Hz), 6.94 (1H, d, *J* = 10.0 Hz), 6.90 (1H, d, *J* = 10.0 Hz), 3.89 (3H, s), 3.06 (2H, s), 0.21 (6H, s).

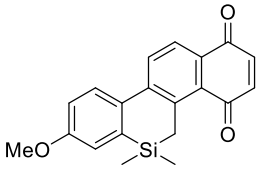
^{13}C-NMR ($CDCl_3$, 125 MHz) δ: 188.17, 185.22, 161.57, 145.31, 144.88, 141.66, 141.55, 136.40, 133.80, 132.97, 132.51, 130.00, 128.20, 124.83, 113.72, 113.40, 55.27, 17.03, -4.39

HRMS (MALDI) calcd for $C_{20}H_{18}O_3Si$: 334.10197, found 334.10086

Anal. calcd for $C_{20}H_{18}O_3Si$: H, 5.43; C, 71.83, found: H, 5.71; C, 71.72

m.p. 120.0-121.0 °C (recrystallized from $CHCl_3$; yellow needle)

3c:



3c (62%, 20.7 mg, 0.0623 mmol, red solid) was prepared from **1c** (23.0 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 8.01 (1H, d, *J* = 8.3 Hz), 7.82 (1H, d, *J* = 8.3 Hz), 7.63 (1H, d, *J* = 8.6 Hz), 7.09 (1H, d, *J* = 2.8 Hz), 7.04 (1H, dd, *J* = 8.6, 2.8 Hz), 6.93 (1H, d, *J* = 10.3 Hz), 6.89 (1H, d, *J* = 10.3 Hz), 3.89 (3H, s), 3.07 (2H, s), 0.23 (6H, s).

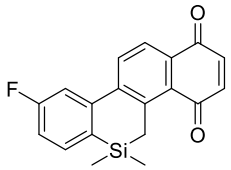
^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 188.32, 185.23, 159.30, 145.30, 141.43, 140.57, 138.99, 136.46, 135.68, 132.31, 131.85, 129.99, 129.10, 124.90, 117.61, 115.37, 55.31, 16.67, -4.62

HRMS (MALDI) calcd for $C_{20}H_{18}O_3Si$: 334.10197, found 334.10112

Anal. calcd for $C_{20}H_{18}O_3Si$: H, 5.43; C, 71.83, found: H, 5.67; C, 71.76

m.p. 174.0-175.0 °C (recrystallized from $CHCl_3$; red column)

3d:



3d (68%, 21.8 mg, 0.0676 mmol, yellow solid) was prepared from **1d** (21.8 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 8.03 (1H, d, *J* = 8.2 Hz), 7.82 (1H, d, *J* = 8.2 Hz), 7.65 (1H, dd, *J* = 8.7, 4.6 Hz), 7.25 (1H, dd, *J* = 7.8, 2.9 Hz), 7.19 (1H, ddd, *J* = 8.7, 8.5, 2.9 Hz), 6.94 (1H, d, *J* = 10.1 Hz), 6.91 (1H, d, *J* = 10.1 Hz), 3.08 (2H, s), 0.23 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 188.07, 185.10, 164.63 (*J* = 246.0 Hz), 145.59 (*J* = 6.7 Hz), 144.27, 141.55, 141.34, 136.45, 134.33, 133.01, 132.81, 132.63 (*J* = 3.8 Hz), 130.07, 124.97, 115.05 (*J* = 19.1 Hz), 114.49 (*J* = 21.0 Hz), 16.71, -4.57

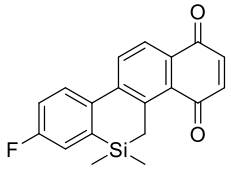
HRMS (MALDI) calcd for $C_{19}H_{15}O_2SiF$: 322.08199, found 322.08120

^{19}F-NMR ($CDCl_3$, 470 MHz) δ: -116.56

Anal. calcd for $C_{19}H_{15}O_2SiF$ + 0.03 $CHCl_3$: H, 4.64; C, 70.07, found: H, 4.86; C, 70.10

m.p. 167.0-170.0 °C (recrystallized from $CHCl_3$; yellow needle)

3e:



3d (55%, 17.8 mg, 0.0552 mmol, yellow solid) was prepared from **1d** (21.8 mg, 0.100 mmol) and benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 8.03 (1H, d, *J* = 8.3 Hz), 7.82 (1H, d, *J* = 8.3 Hz), 7.65 (1H, dd, *J* = 8.6, 4.8 Hz), 7.25 (1H, dd, *J* = 7.7, 2.6 Hz), 7.18 (1H, ddd, *J* = 8.6, 8.6, 2.6 Hz), 6.94 (1H, d, *J* = 10.3 Hz), 6.90 (1H, d, *J* = 10.3 Hz), 3.08 (2H, s), 0.23 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 188.16, 185.12, 162.47 (*J* = 250.7 Hz), 144.54, 141.49, 140.64, 140.38, 140.32 (*J* = 2.4 Hz), 139.08 (*J* = 2.9 Hz), 136.48, 132.81, 130.02, 129.70, 129.63, 124.99, 118.67 (*J* = 18.1 Hz), 117.25 (*J* = 21.0 Hz), 16.49, -4.78

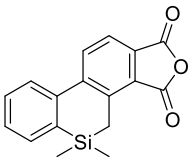
^{19}F-NMR ($CDCl_3$, 470 MHz) δ: -116.56

HRMS (MALDI) calcd for $C_{19}H_{15}O_2SiF$: 322.08199, found 322.08120

Anal. calcd for $C_{19}H_{15}O_2SiF$: H, 4.69; C, 70.78, found: H, 4.83; C, 70.51

m.p. 163.0-164.0 °C (recrystallized from $CHCl_3$; yellow needle)

3f:



3f (49%, 14.4 mg, 0.0489 mmol, white solid) was prepared from **1a** (20.0 mg, 0.100 mmol) and maleimic anhydride (19.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 500 MHz) δ: 8.03 (1H, d, *J* = 8.2 Hz), 7.85 (1H, d, *J* = 8.2 Hz), 7.70 (1H, dd, *J* = 7.7, 0.9 Hz), 7.61 (1H, dd, *J* = 7.4, 1.5 Hz), 7.56 (1H, ddd, *J* = 7.7, 7.4, 1.5 Hz), 7.45 (1H, ddd, *J* = 7.7, 7.4, 0.9 Hz), 2.87 (2H, s), 0.27 (6H, s).

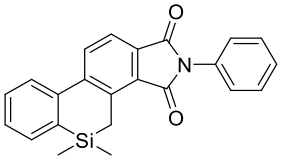
^{13}C-NMR ($CDCl_3$, 125 MHz) δ: 163.92, 162.61, 146.52, 141.56, 140.52, 136.30, 135.13, 132.93, 130.81, 129.91, 128.86, 128.52, 127.23, 122.90, 15.55, -4.43

HRMS (MALDI) calcd for $C_{17}H_{14}O_3Si$: 294.07067, found 294.07082

Anal. calcd for $C_{19}H_{16}O_2Si$: H, 4.79; C, 69.36, found: H, 5.09; C, 69.27

m.p. 148.0-151.5 °C (recrystallized from $CHCl_3$; colorless column)

3g:



3g (34%, 12.7 mg, 0.0343 mmol, white solid) was prepared from **1a** (20.0 mg, 0.10 mmol) and *N*-phenylmaleimid (34.6 mg, 0.20 mmol) by a general procedure **C**.

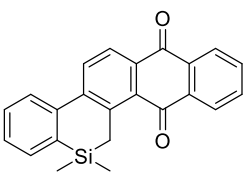
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 7.95 (1H, d, *J* = 7.8 Hz), 7.82 (1H, d, *J* = 7.8 Hz), 7.72 (1H, d, *J* = 7.8 Hz), 7.61 (1H, dd, *J* = 7.3, 1.4 Hz), 7.55-7.50 (3H, m), 7.48-7.41 (4H, m), 2.97 (2H, s), 0.28 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 168.69, 166.84, 145.15, 142.40, 138.58, 136.44, 133.50, 132.74, 131.84, 130.90, 130.59, 129.02, 128.24, 128.20, 127.96, 127.96, 127.96, 127.05, 126.75, 121.12, 14.92, -4.37

HRMS (MALDI) calcd for $C_{23}H_{20}NO_2Si$: 370.12578, found 370.12645

m.p. 173.0-174.0 °C (recrystallized from $CHCl_3$; white needle)

3h:



3h (41%, 14.4 mg, 0.0406 mmol, yellow solid) was prepared from **1a** (20.0 mg, 0.100 mmol) and

1,4-naphtoquinone (19.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 8.27-8.24 (3H, m), 7.92 (1H, d, *J* = 8.0 Hz), 7.80 (1H, ddd, *J* = 7.6, 7.3, 1.5 Hz), 7.76 (1H, ddd, *J* = 7.6, 7.3, 1.9 Hz), 7.69 (1H, dd, *J* = 7.6, 1.9 Hz), 7.60 (1H, dd, *J* = 7.1, 1.1 Hz), 7.54 (1H, ddd, *J* = 7.6, 7.6, 1.5 Hz), 7.41 (1H, ddd, *J* = 7.3, 7.3, 1.1 Hz), 3.15 (2H, s), 0.25 (6H, s).

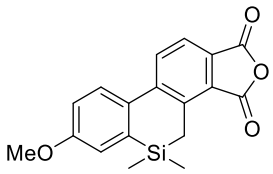
^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 186.64, 183.43, 145.47, 143.42, 137.18, 136.02, 134.07, 133.93, 133.34, 133.25, 132.82, 132.31, 132.29, 130.43, 128.05, 127.40, 127.30, 126.44, 125.45, 17.57, -4.64

HRMS (MALDI) calcd for $C_{23}H_{18}O_2Si$: 354.10706, found 354.10708

Anal. calcd for $C_{23}H_{28}O_2Si$: H, 5.12; C, 77.93, found: H, 5.13; C, 77.85

m.p. 156.0-157.0 °C (recrystallized from $CHCl_3$; yellow needle)

3i:



3i (44%, 14.4 mg, 0.0406 mmol, beige solid) was prepared from **1c** (20.0 mg, 0.100 mmol) and maleimic anhydride (19.6 mg, 0.200 mmol) by a general procedure **C**.

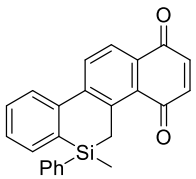
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 7.97 (1H, d, *J* = 8.0 Hz), 7.82 (1H, d, *J* = 8.0 Hz), 7.67 (1H, d, *J* = 8.7 Hz), 7.11 (1H, d, *J* = 2.7 Hz), 7.06 (1H, dd, *J* = 8.7, 2.7 Hz), 3.90 (3H, s), 2.85 (2H, s), 0.27 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 164.05, 162.69, 159.84, 146.43, 139.71, 138.22, 134.29, 134.00, 129.02, 128.55, 122.89, 118.29, 115.61,55.35, 15.46, -4.42

HRMS (MALDI) calcd for $C_{18}H_{16}O_4Si$: 324.08116, found 324.08124

m.p. 186.5-187.0 °C (recrystallized from $CHCl_3$; beige irregular)

3j:



3j (43%, 15.9 mg, 0.0433 mmol, yellow solid) was prepared from **1j** (20.0 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 8.02 (1H, d, *J* = 8.3 Hz), 7.89 (1H, d, *J* = 8.3 Hz), 7.72-7.70 (1H, m), 7.57-7.55 (2H, m), 7.44-7.25 (8H, m), 6.91 (1H, d, *J* = 10.3 Hz), 6.86 (1H, d, *J* = 10.0 Hz), 3.63 (1H, d, *J* = 15.1 Hz), 3.08 (1H, d, *J* = 15.1 Hz), 2.35 (1H, s), 0.53 (3H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 188.06, 185.16, 145.38, 143.69, 141.48, 140.42, 136.35, 135.24, 134.37, 133.38,

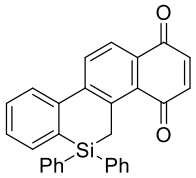
133.12, 132.48, 130.79, 129.97, 129.76, 129.01, 128.20, 128.15, 127.90, 127.54, 125.27, 125.04, 15.82, -5.84

HRMS (MALDI) calcd for $C_{24}H_{18}O_2Si$: 366.10706, found 366.10638

Anal. calcd for $C_{24}H_{18}O_2Si$: H, 4.95; C, 78.66, found: H, 5.23; C, 78.63

m.p. 104.5-105.0 °C (recrystallized from $CHCl_3$; yellow needle)

3k:



3k (74%, 31.6 mg, 0.0738 mmol, yellow solid) was prepared from **1k** (20.0 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 300 MHz) δ: 7.95 (1H, d, *J* = 8.3 Hz), 7.85 (1H, d, *J* = 8.3 Hz), 7.74 (1H, d, *J* = 7.9 Hz), 7.60-7.47 (6H, m), 7.39-7.24 (7H, m), 6.90 (1H, d, *J* = 10.3 Hz), 6.84 (1H, d, *J* = 10.3 Hz), 3.68 (2H, s).

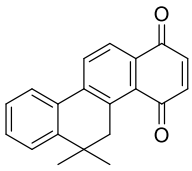
^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 188.06, 185.15, 145.38, 143.69, 141.47, 140.42, 136.35, 135.24, 134.37, 134.12, 133.38, 133.12, 132.48, 130.79, 129.97, 129.76, 129.01, 128.20, 128.15, 127.90, 127.54, 125.27, 125.04, 15.82, -5.84

HRMS (MALDI) calcd for $C_{29}H_{20}O_2Si$: 428.12271, found 428.12212

Anal. calcd for $C_{29}H_{20}O_2Si$: H, 4.70; C, 81.26, found: H, 5.00; C, 81.53

m.p 181.0-182.0 °C (recrystallized from $CHCl_3$; yellow needle)

3l:



3l (78%, 22.5 mg, 0.0780 mmol, yellow solid) was prepared from **1l** (18.4 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

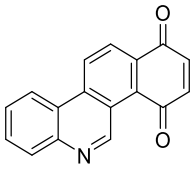
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 8.11 (2H, s), 7.76 (1H, d, *J* = 7.8 Hz), 7.48-7.36 (3H, m), 6.93 (2H, s), 3.47 (2H, s), 1.27 (3H×2, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 187.64, 184.97, 146.04, 141.42, 140.81, 139.46, 136.94, 132.04, 131.63, 130.02, 129.75, 128.44, 126.81, 125.77, 125.19, 124.11, 39.97, 33.51, 28.10

HRMS (MALDI) calcd for $C_{20}H_{16}O_2$: 288.11330, found 288.11448

m.p. 150.0-150.5 °C (recrystallized from $CHCl_3$; yellow needle)

3m:



3m (19%, 5.0 mg, 0.019 mmol, yellow solid) was prepared from **1m** (31.1 mg, 0.100 mmol) and 1,4-benzoquinone (21.6 mg, 0.200 mmol) by a general procedure **C**.

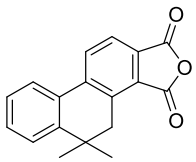
^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 10.81 (1H, s), 8.98 (1H, d, *J* = 8.7 Hz), 8.60 (1H, d, *J* = 7.5 Hz), 8.53 (1H, d, *J* = 8.7 Hz), 8.25 (1H, d, *J* = 7.5 Hz), 7.87 (1H, t, *J* = 7.6 Hz), 7.76 (1H, t, *J* = 7.6 Hz), 7.09 (1H, d, *J* = 10.1 Hz), 7.05 (1H, d, *J* = 10.1 Hz).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 187.39, 184.63, 151.40, 144.60, 140.57, 136.49, 136.45, 132.04, 130.63, 130.33, 128.43, 128.03, 127.96, 127.20, 123.12, 122.69, 122.61

HRMS (MALDI) calcd for $C_{17}H_{10}NO_2$: 260.07009 ([M+H$^+$]), found 260.07060 ([M+H$^+$])

m.p. >282.0 °C (recrystallized from $CHCl_3$; yellow needle)

3n



3n (10%, 5.6 mg, 0.020 mmol, yellow solid) was prepared from **1a** (31.1 mg, 0.200 mmol) and maleimic anhydride (39.2 mg, 0.400 mmol) by a general procedure **C**.

^{1}H-NMR ($CDCl_3$, 400 MHz) δ: 8.22 (1H, d, *J* = 8.0 Hz), 7.94 (1H, d, *J* = 8.0 Hz), 7.82 (1H, dd, *J* = 7.8, 1.4 Hz), 7.51 (1H, dd, *J* = 7.6, 1.4 Hz), 7.46 (1H, ddd, *J* = 7.8, 7.6, 1.4 Hz), 7.39 (1H, ddd, *J* = 7.8, 7.8, 1.4 Hz), 3.34 (2H, s), 1.32 (6H, s).

^{13}C-NMR ($CDCl_3$, 100 MHz) δ: 163.36, 162.71, 146.19, 142.97, 138.13, 130.65, 130.44, 129.62, 128.54, 127.18, 125.24, 124.80, 124.02, 37.63, 33.54, 27.97

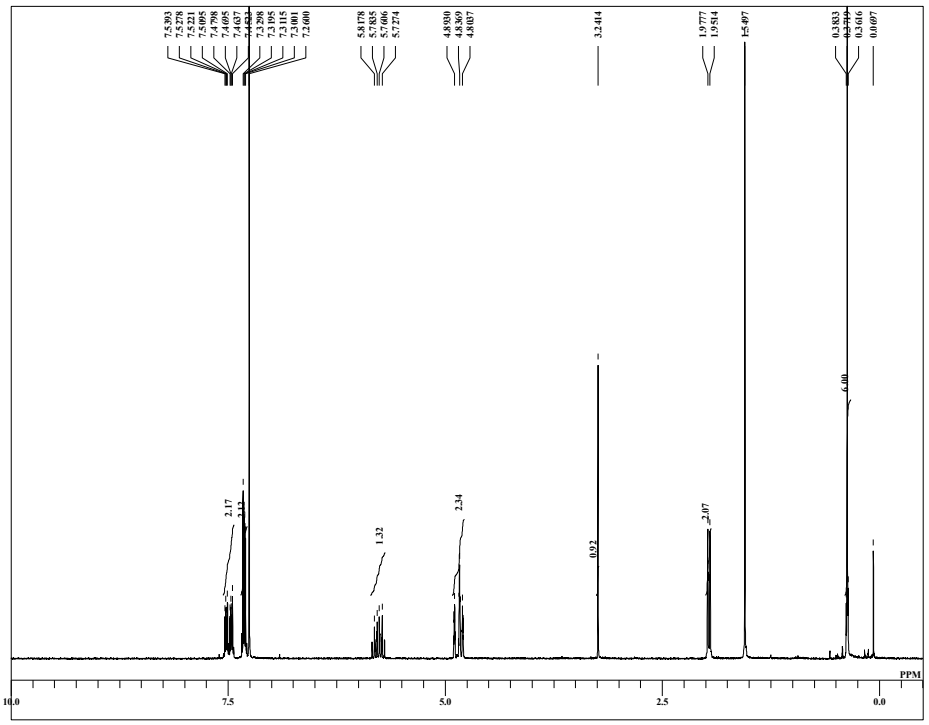
HRMS (MALDI) calcd for $C_{18}H_{15}O_3$: 279.10150, found 279.10157

m.p. 170.5-172.0 °C (recrystallized from $CHCl_3$; pale yellow needle)

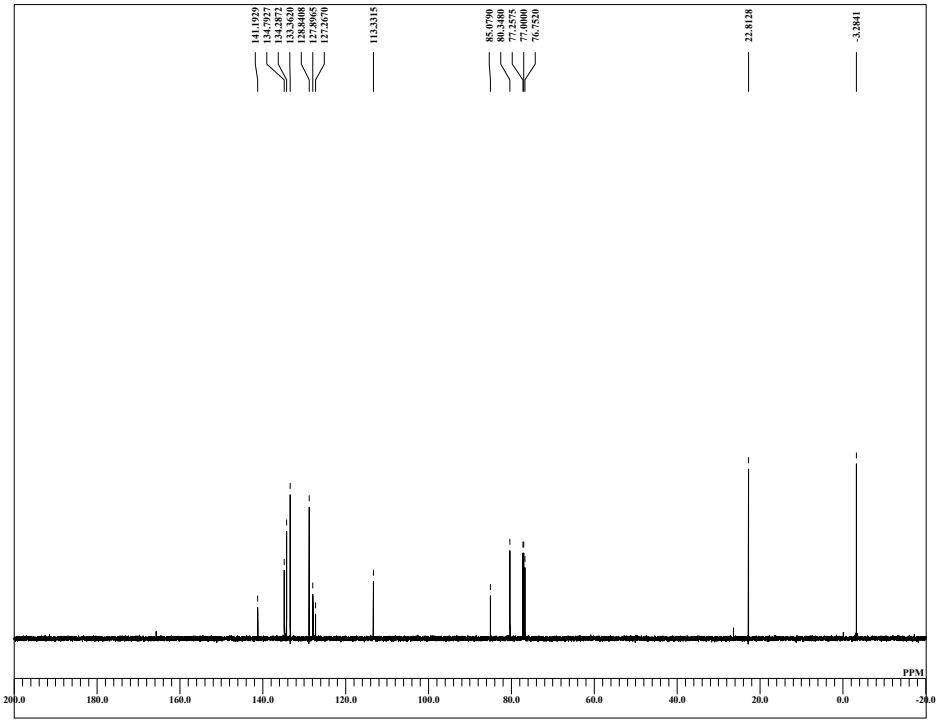
reference

1. T. Matsuda, S. Kadowaki, Y. Yamaguchi, M. Murakami, *Chem. Commun*. 2008, **24**, 2744-2746
2. S. Bhunia, K. Wang, R. Liu, *Angew. Chem. Int. Edit.* 2008, **47**, 5063-5066
3. M. Matveenko, G. Liang, E. M. W. Lauterwasser, E. Aubia, D. Treuner, *J. Am. Chem. Soc.*, 2012, **134**, 9291-9295
4. M. Mori, D. Tanaka, N. Saito, Y. Sato, *Organometallics* 2008, **27**, 6313-6320

1a



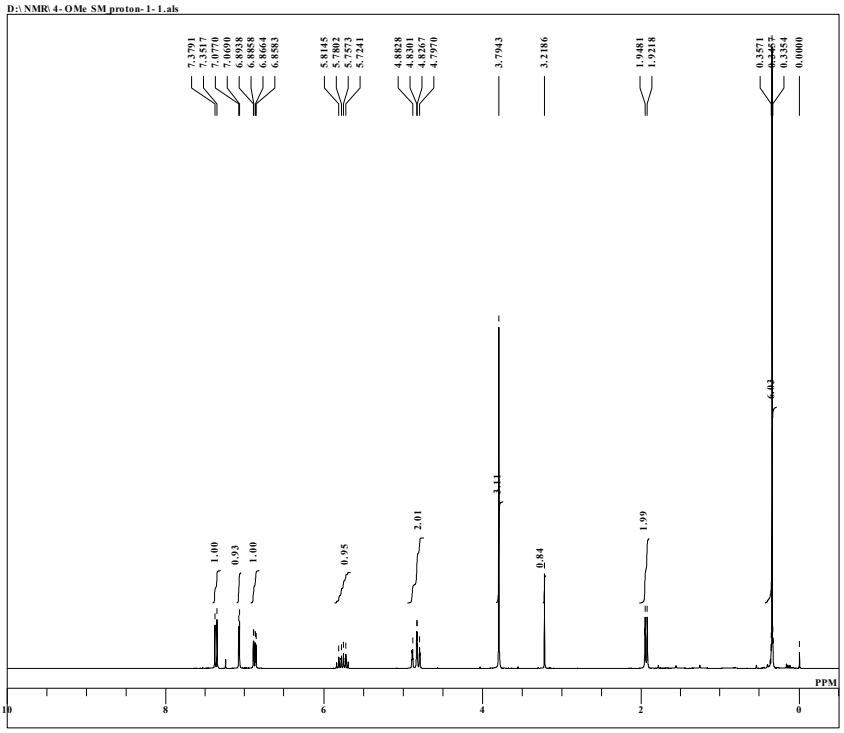
DFILE 1500033_proton-1-1.als
COMNT single_pulse
DATIM 19-11-2015 18:12:52
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 13107
FREQU 4508.57 Hz
SCANS 16
ACQTM 2.9072 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 44



DFILE 1500033_Carbon-1-1.jdf
COMNT single pulse decoupled ga
DATIM 19-11-2015 19:28:06
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 30
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

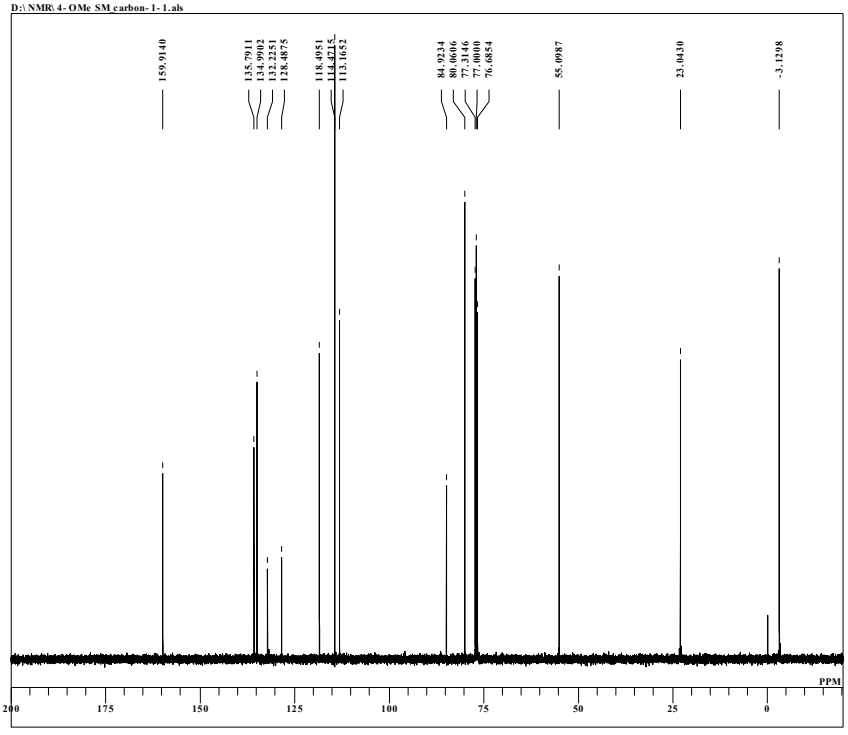
1b

single_pulse



DFILE	4- OMe SM_proton- 1- 1.als
COMNT	single_pulse
DATIM	01- 06- 2016 23:04:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	300.53 MHz
OBSET	1.15 KHz
OBFIN	8.57 Hz
POINT	13107
FREQU	4508.57 Hz
SCANS	8
ACQTM	2.9072 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	21.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30

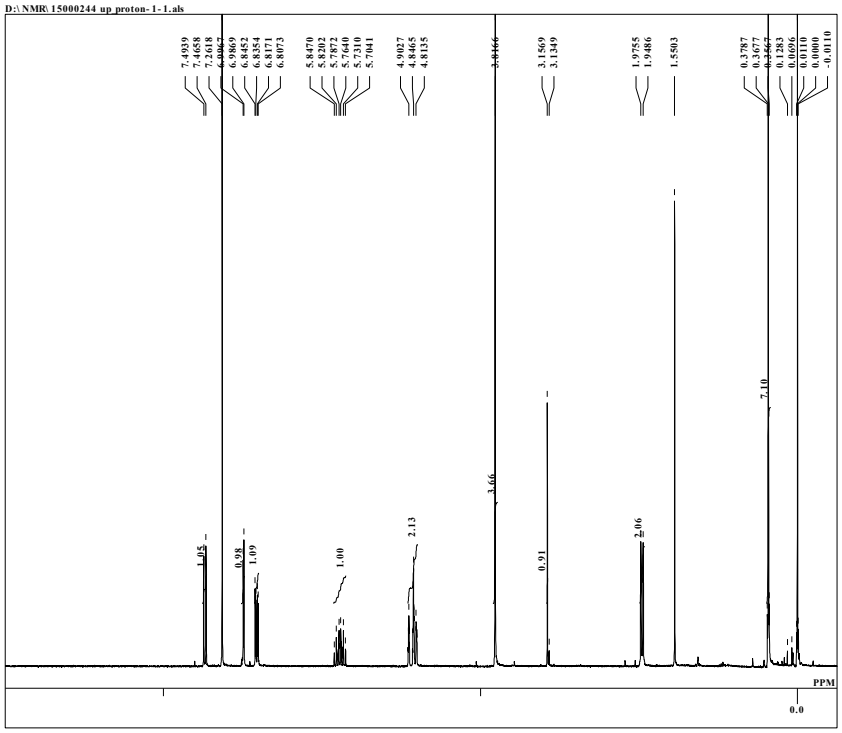
single pulse decoupled gated NOE



DFILE	4- OMe SM_carbon- 1- 1.als
COMNT	single pulse decoupled gated NOE
DATIM	2016- 06- 02 09:31:07
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	326
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.05 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

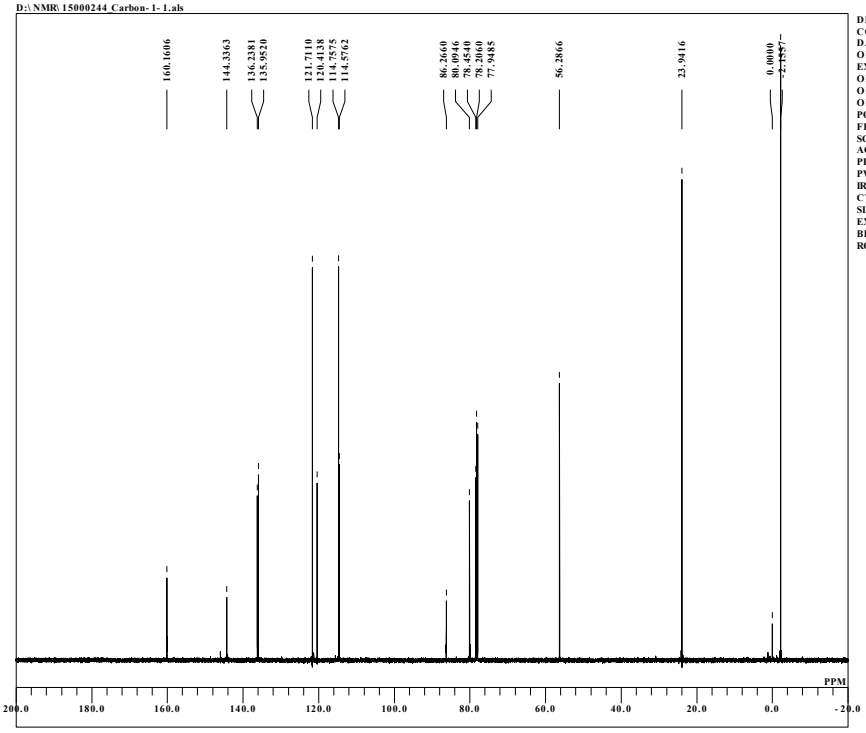
1c

single_pulse



DFILE 15000244 up_proton- 1- 1.als
COMNT single_pulse
DATIM 2016- 02- 19 12:44:21
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.45 KHz
OBFIN 9.10 Hz
POINT 13107
FREQU 4812.32 Hz
SCANS 16
ACQTM 2.7237 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

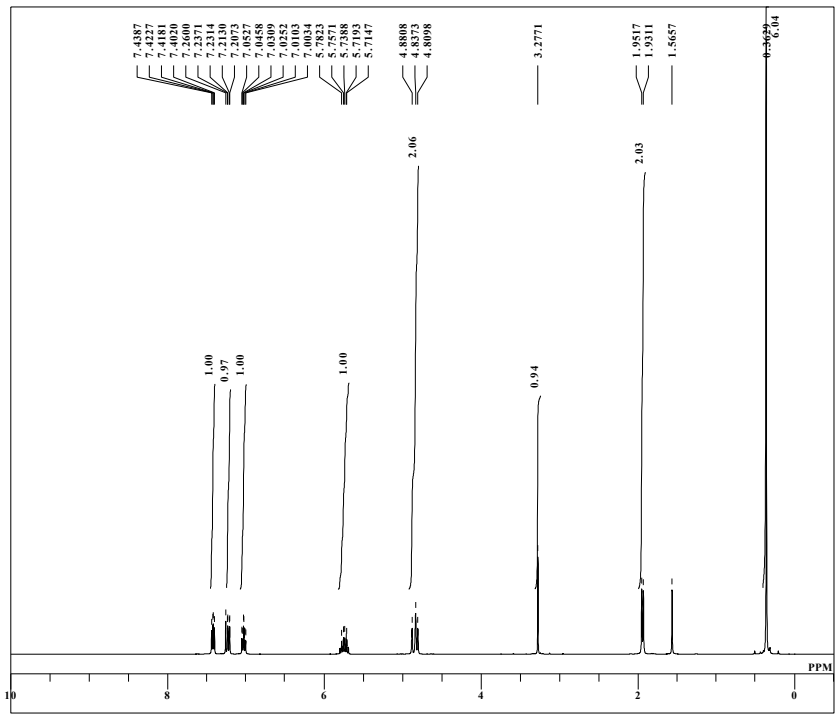
single pulse decoupled gated NOE



DFILE 15000244_Carbon- 1- 1.als
COMNT single pulse decoupled gated NOE
DATIM 2016- 02- 19 13:18:39
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 400
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60

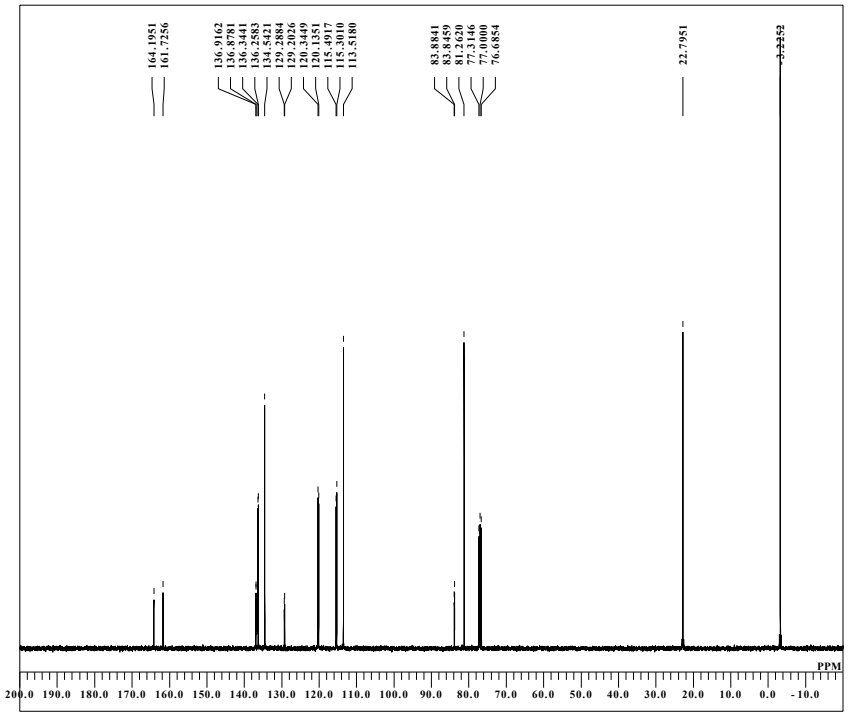
1d

single_pulse



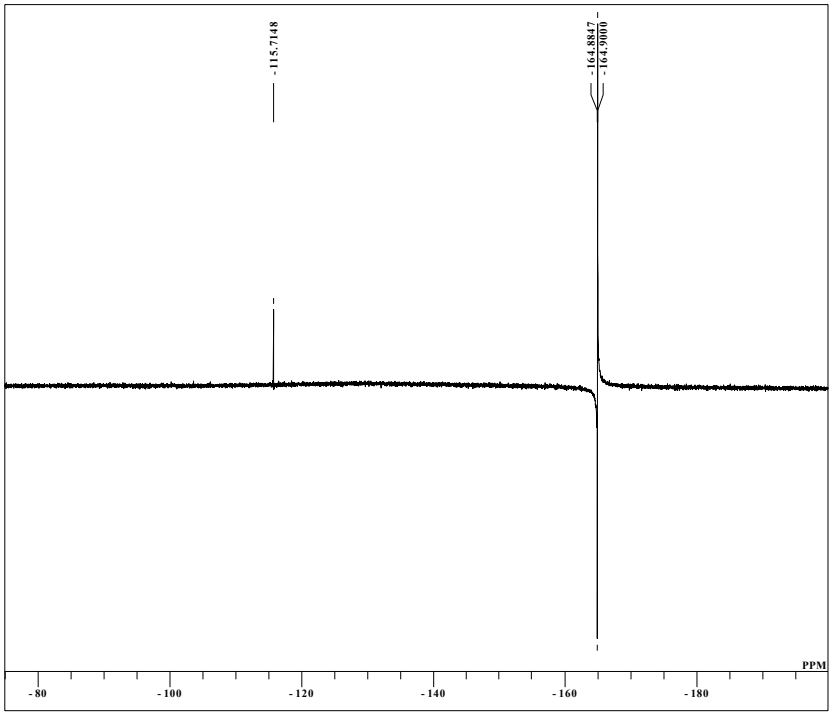
DFILE 4-F SM_proton-1-1.als
COMNT single_pulse
DATIM 2016-08-24 12:11:09
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.64 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 1.20 Hz
RGAIN 40

single pulse decoupled gated NOE



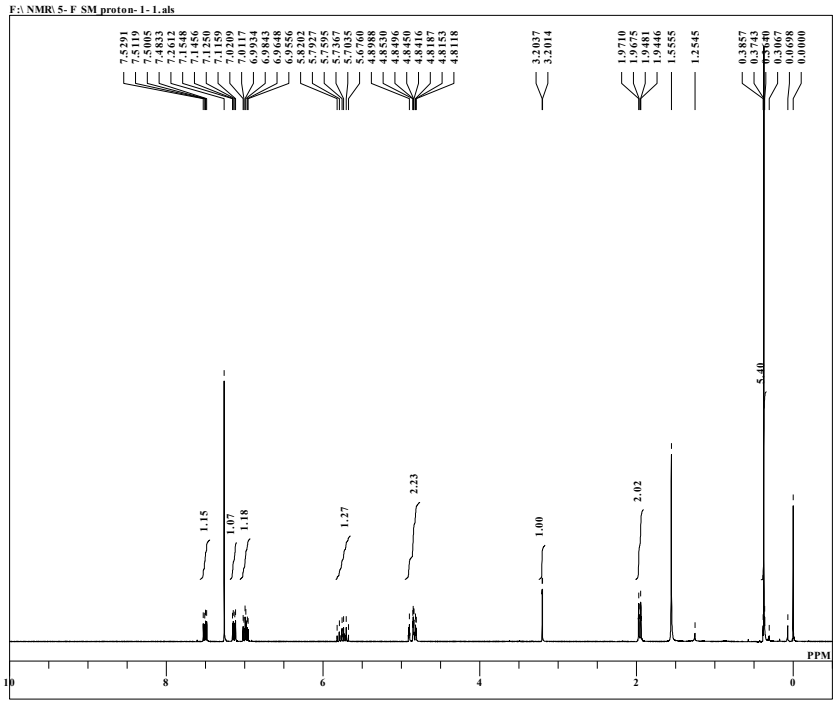
DFILE 4-F SM_carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-08-24 00:46:19
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 67
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

single_pulse

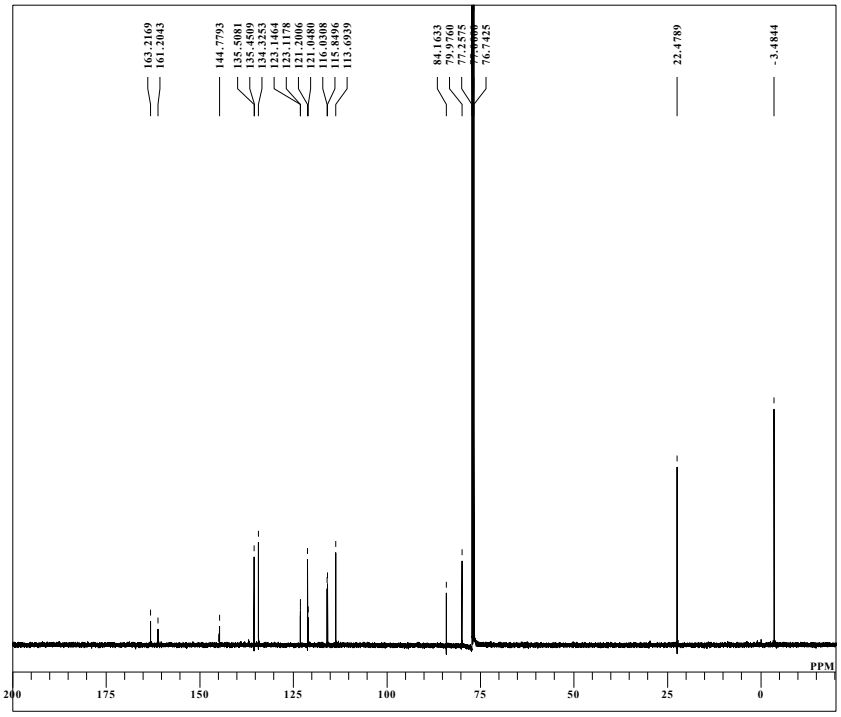


DFILE	4-F SM_single_pulse-2-1.als
COMNT	single_pulse
DATIM	04-07-2016 21:57:26
OBNUC	19F
EXMOD	single_pulse.jxp
OBFRQ	470.57 MHz
OBSET	3.39 KHz
OBFIN	8.79 Hz
POINT	13107
FREQU	94339.62 Hz
SCANS	8
ACQTM	0.1389 sec
PD	5.0000 sec
PW1	5.80 usec
IRNUC	19F
CTEMP	21.7 c
SLVNT	CDCl3
EXREF	-164.90 ppm
BF	0.12 Hz
RGAIN	42

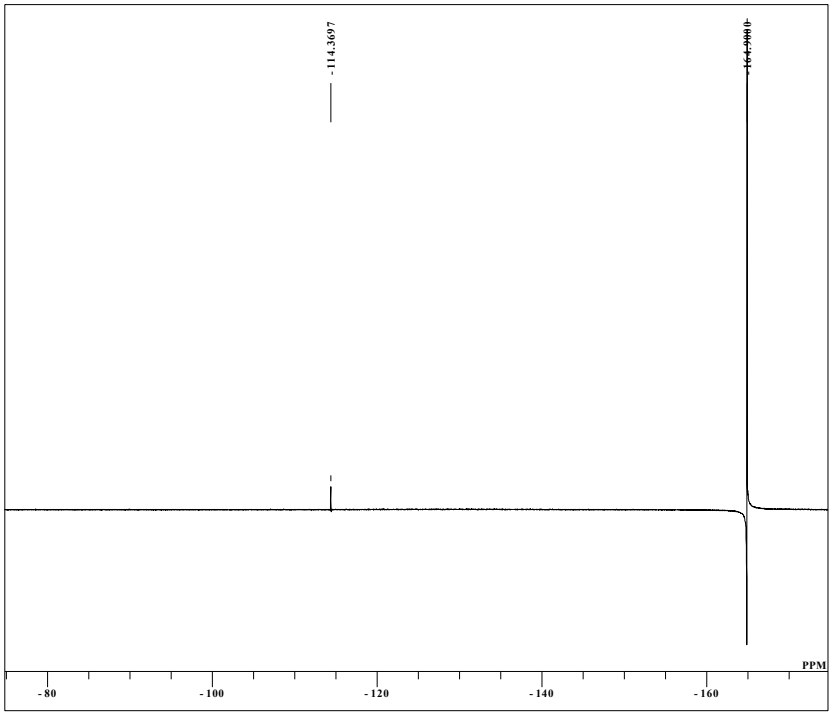
1e



single pulse decoupled gated NOE



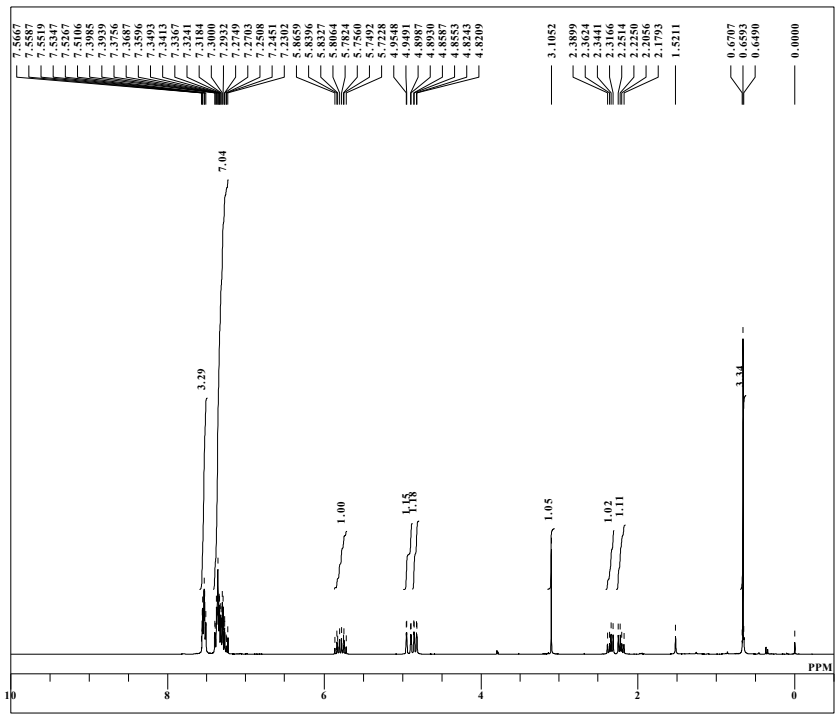
single_pulse



DFILE	5-F SM_single_pulse-1-1.jdf
COMNT	single_pulse
DATIM	2016-07-04 22:05:52
OBNUC	19F
EXMOD	single_pulse.jxp
OBFRQ	470.57 MHz
OBSET	3.39 KHz
OBFIN	8.79 Hz
POINT	16384
FREQU	117924.53 Hz
SCANS	8
ACQTM	0.1389 sec
PD	5.0000 sec
PW1	5.80 usec
IRNUC	19F
CTEMP	21.7 c
SLVNT	CDCl3
EXREF	-164.90 ppm
BF	0.12 Hz
RGAIN	42

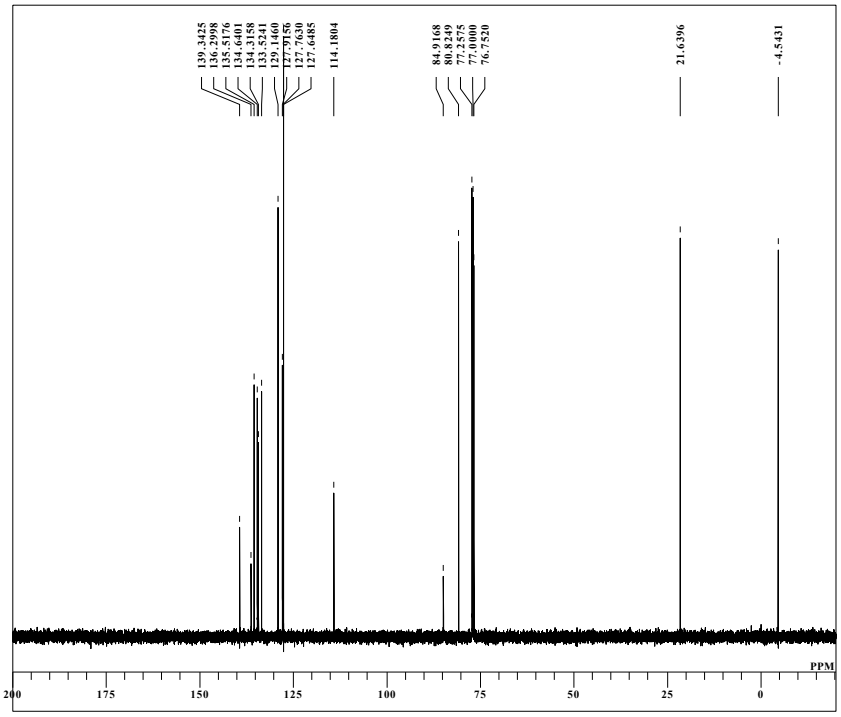
1j

single_pulse



DFILE MePh SM_proton-1-1.jdf
COMNT single_pulse
DATIM 2016-07-06 18:25:36
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 16384
FREQU 5635.71 Hz
SCANS 16
ACQTM 2.9072 sec
PD 5.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

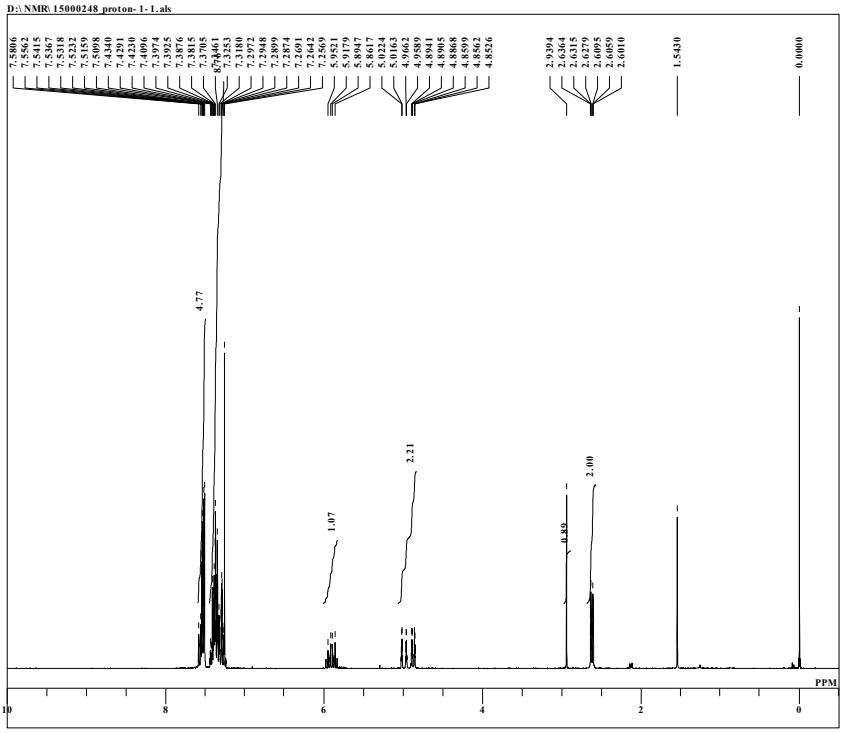
single pulse decoupled gated NOE



DFILE MePh SM_Carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2016-07-06 20:55:02
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 114
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

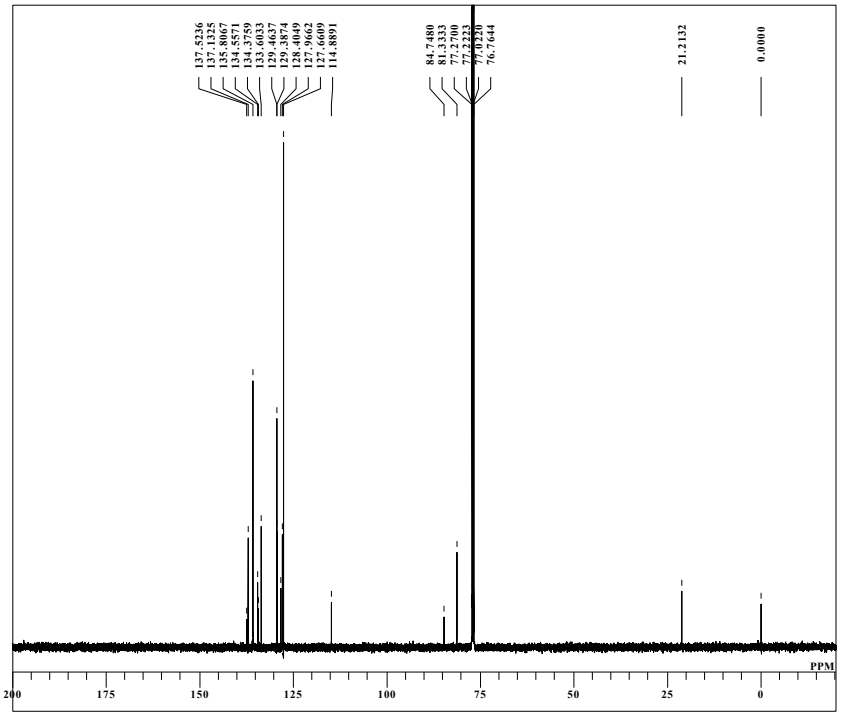
1k

single_pulse



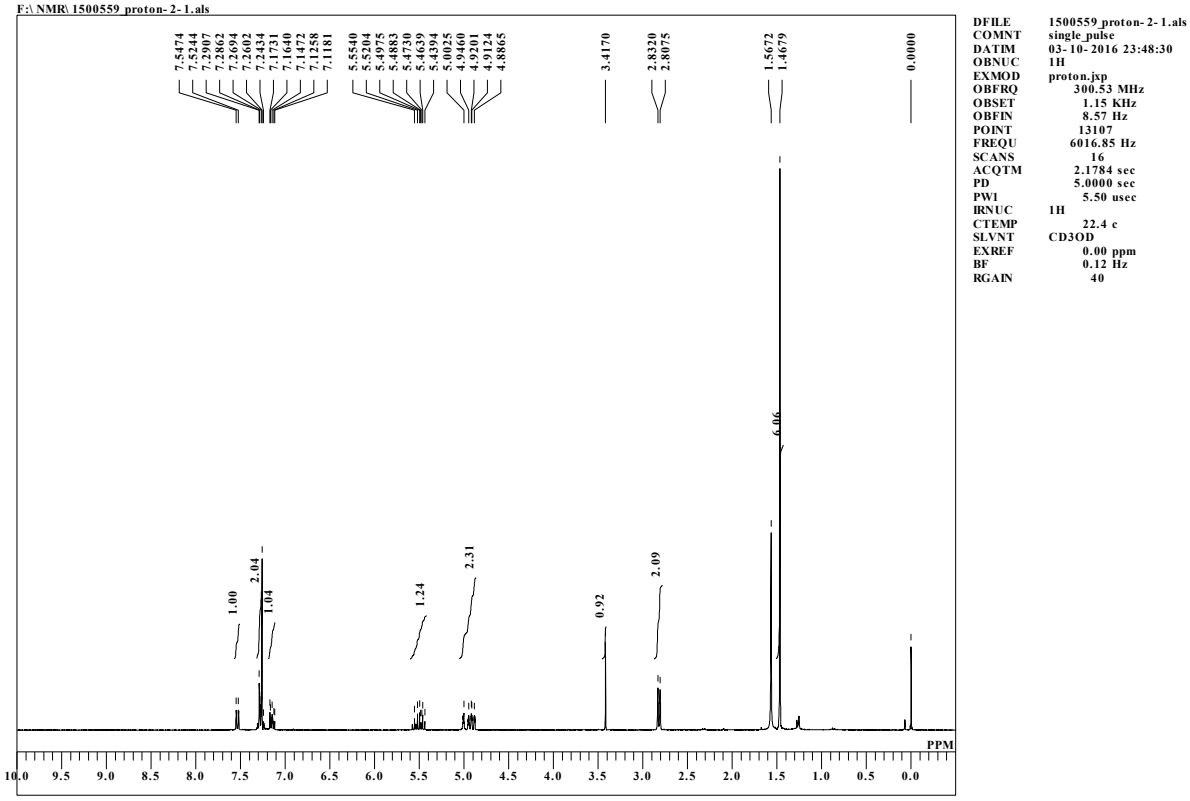
DFILE	15000248_proton- 1- 1.als
COMNT	single_pulse
DATIM	23- 02- 2016 00:47:05
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	300.53 MHz
OBSET	1.45 KHz
OBFIN	9.10 Hz
POINT	13107
FREQU	4812.32 Hz
SCANS	16
ACQTM	2.7237 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

single pulse decoupled gated NOE

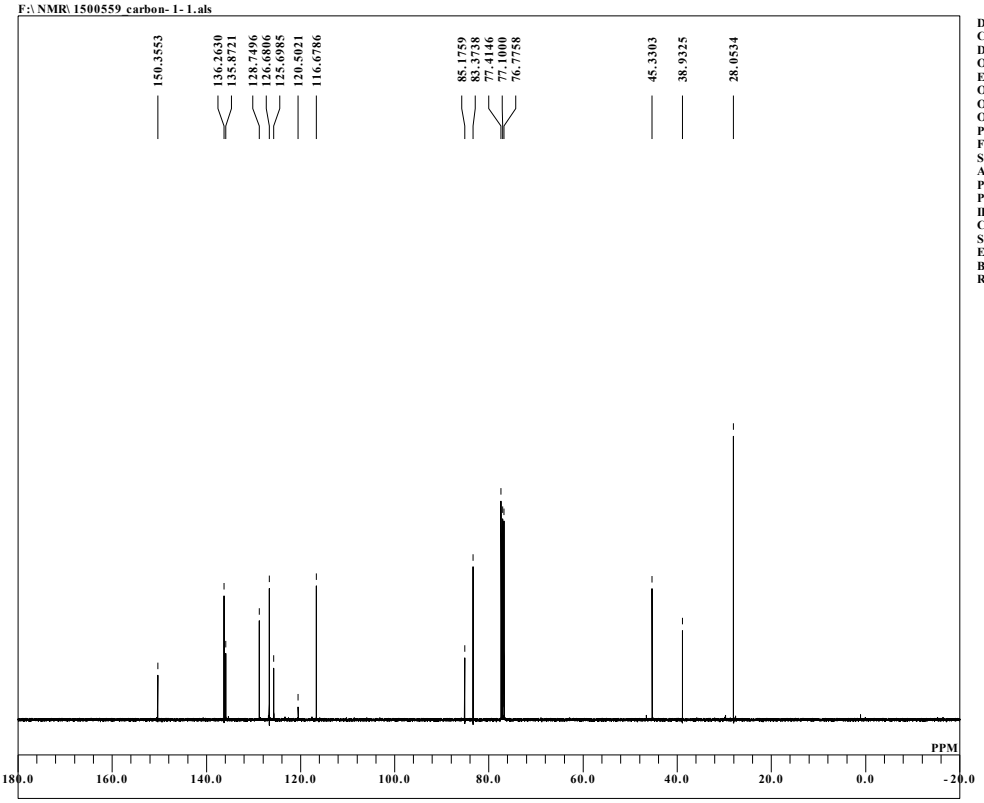


DFILE	PhPh SM Carbon- 2- 1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016- 07- 06 05:37:08
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	2337
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.20 usec
IRNUC	1H
CTEMP	22.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	60

1l



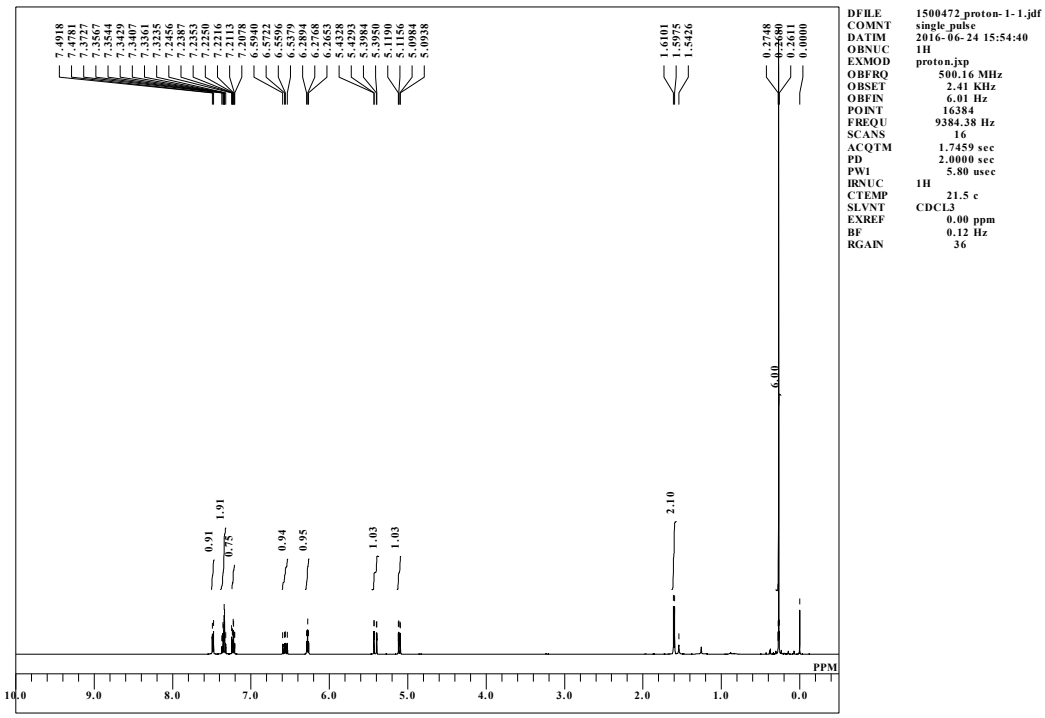
DFILE	1500559_proton-2-1.als
COMNT	single_pulse
DATIM	03-10-2016 23:48:30
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	300.53 MHz
OBSET	1.15 KHz
OBFIN	8.57 Hz
POINT	13107
FREQU	6016.85 Hz
SCANS	16
ACQTM	2.1784 sec
PD	5.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CD3OD
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



DFILE	1500559_carbon-1-1.als
COMNT	single pulse decoupled gated N
DATIM	2016-10-04 01:22:38
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	1134
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.05 usec
IRNUC	1H
CTEMP	20.4 c
SLVNT	CDCL3
EXREF	77.10 ppm
BF	0.12 Hz
RGAIN	60

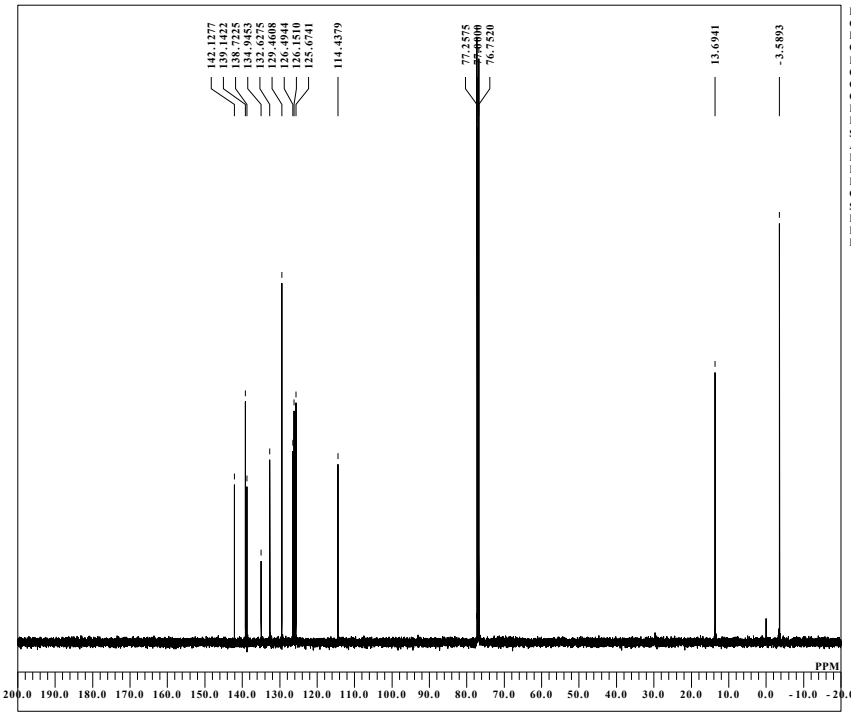
2a

single_pulse



DFILE 1500472_proton-1-1.jdf
COMNT single_pulse
DATIM 2016-06-24 15:54:40
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 16
ACQTM 1.7459 sec
PD 2.0000 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36

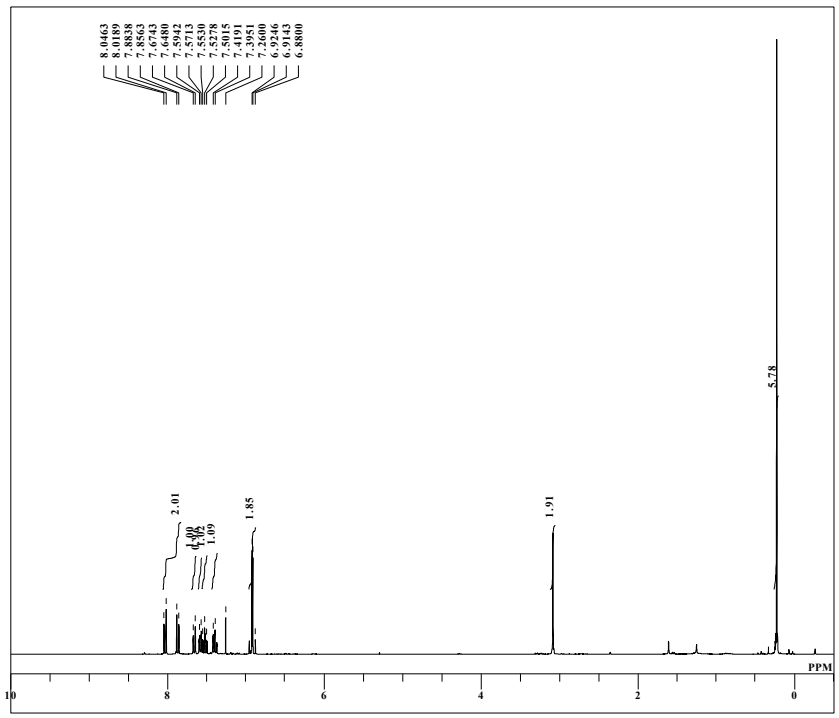
single pulse decoupled gated NOE



DFILE 1500472_Carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2016-06-24 15:58:08
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 512
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

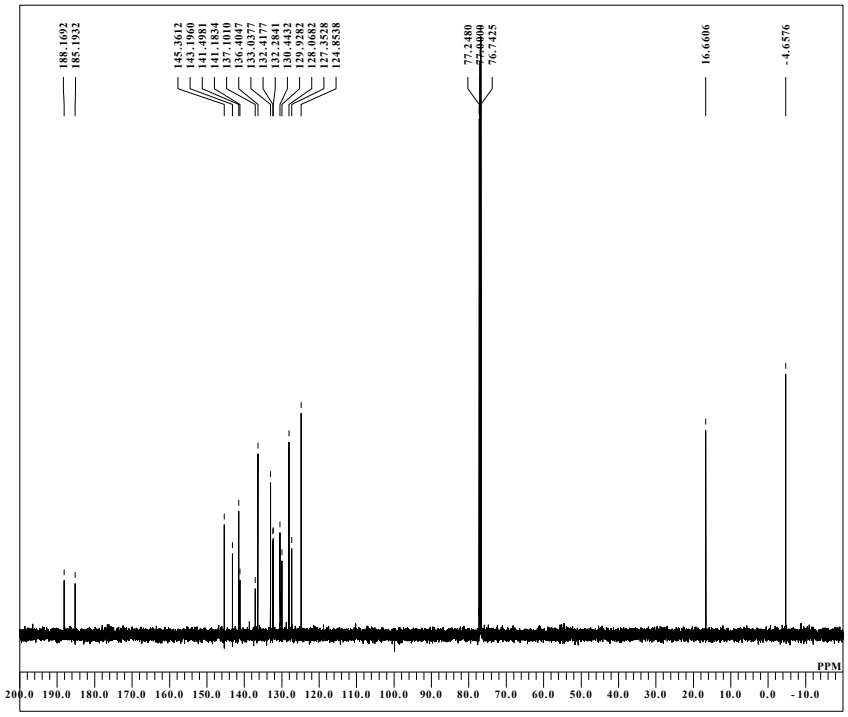
3a

single_pulse



DFILE 15000151_proton-1-1.als
COMNT single_pulse
DATIM 21-11-2015 20:07:29
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 13107
FREQU 4508.57 Hz
SCANS 16
ACQTM 2.9072 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

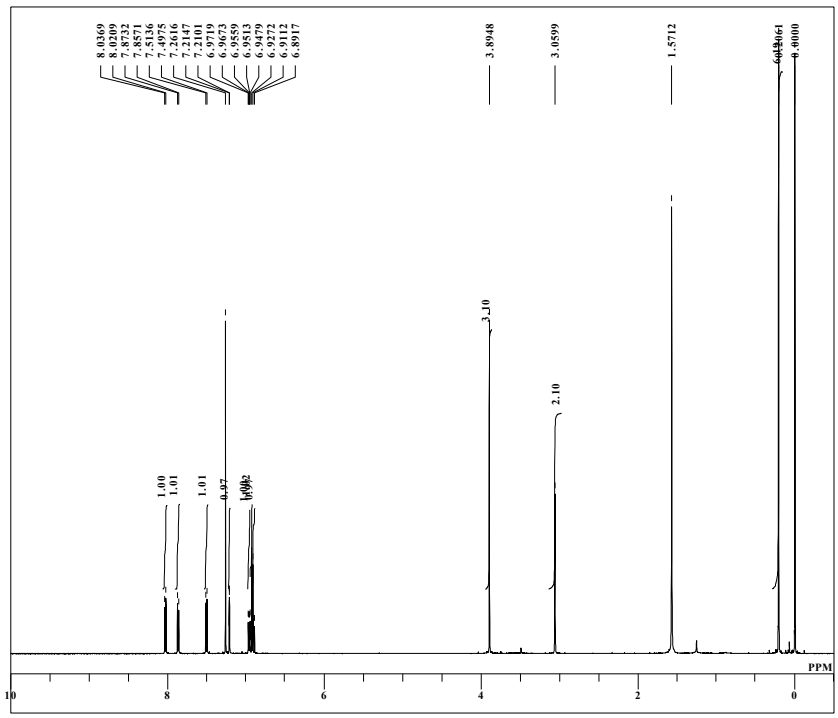
single pulse decoupled gated NOE



DFILE 15000151_Carbon-1-1.jdf
COMNT single pulse decoupled gated NOE
DATIM 2015-11-21 20:10:53
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32767
FREQU 39308.18 Hz
SCANS 177
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

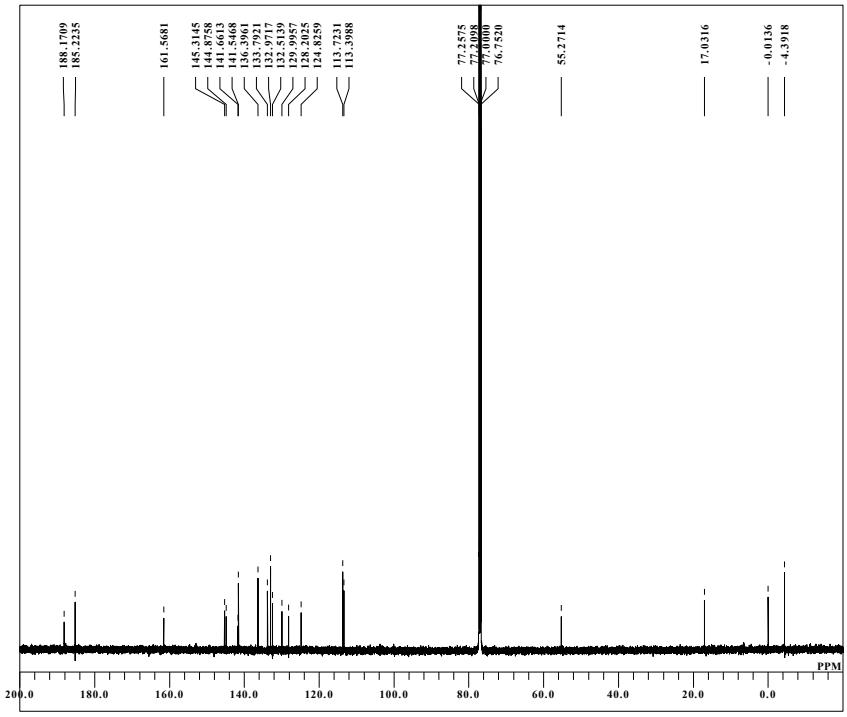
3b

single_pulse



DFILE 4-OMe_proton-1-2.als
COMNT single_pulse
DATIM 2016-07-06 10:07:02
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 16
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

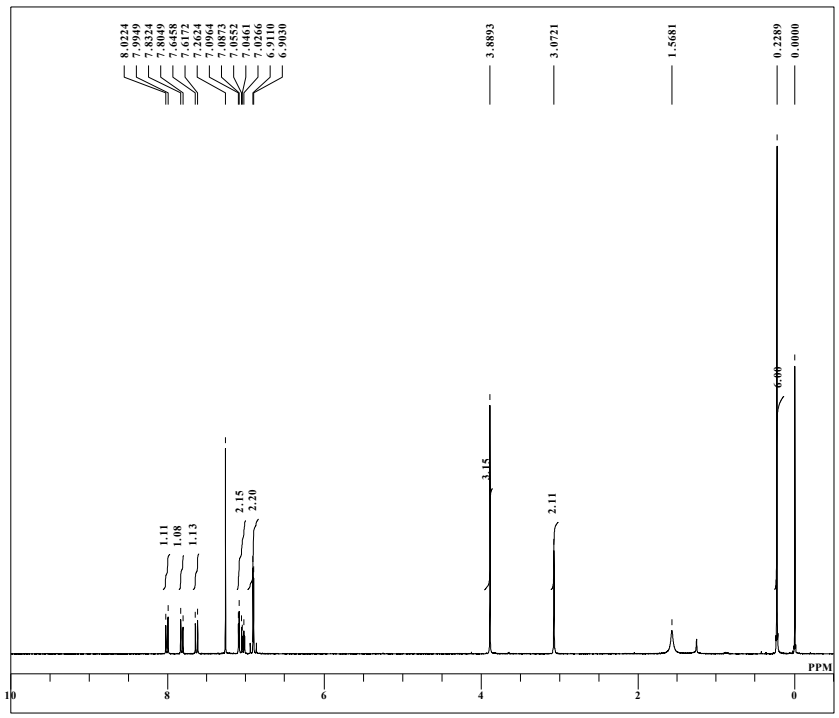
single pulse decoupled gated NOE



DFILE 4-OMe_Carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 07-07-2016 01:37:55
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 9774
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

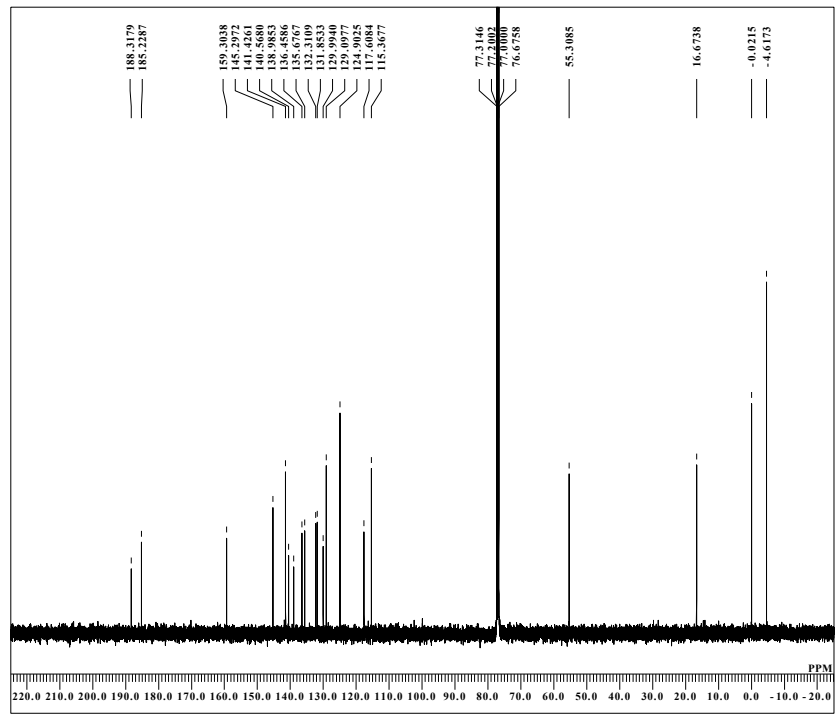
3c

single_pulse



DFILE 5-OMe_proton-1-1.als
COMNT single_pulse
DATIM 02-06-2016 00:04:47
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 13107
FREQU 4508.57 Hz
SCANS 8
ACQTM 2.9072 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

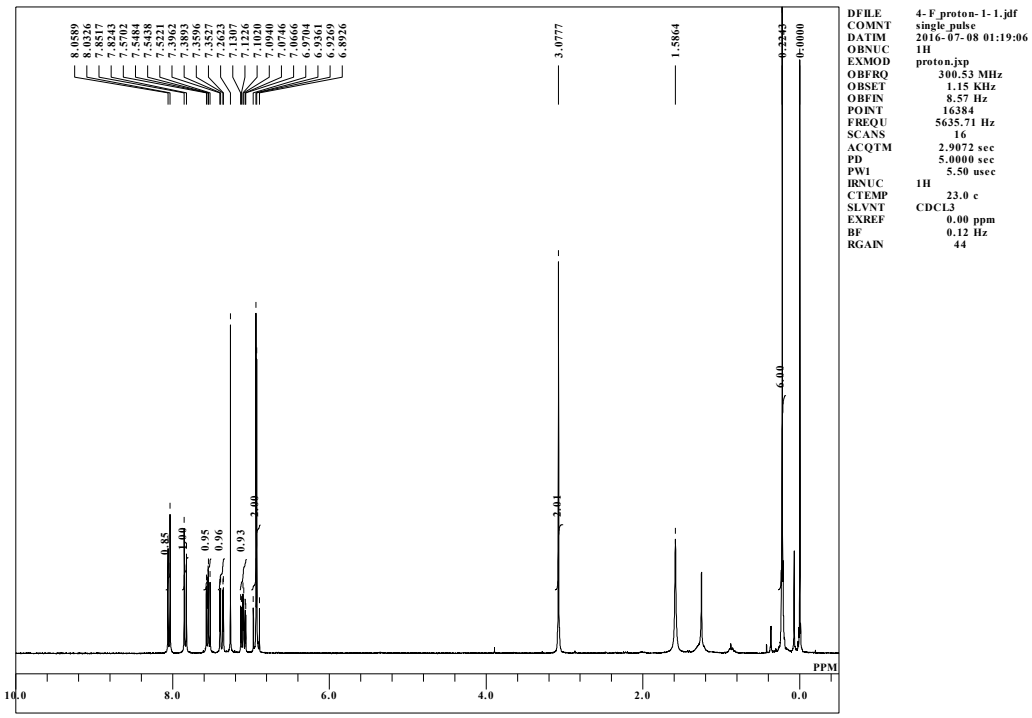
single pulse decoupled gated NOE



DFILE 5-OMe_carbon-2-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-06-03 01:10:15
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 8937
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

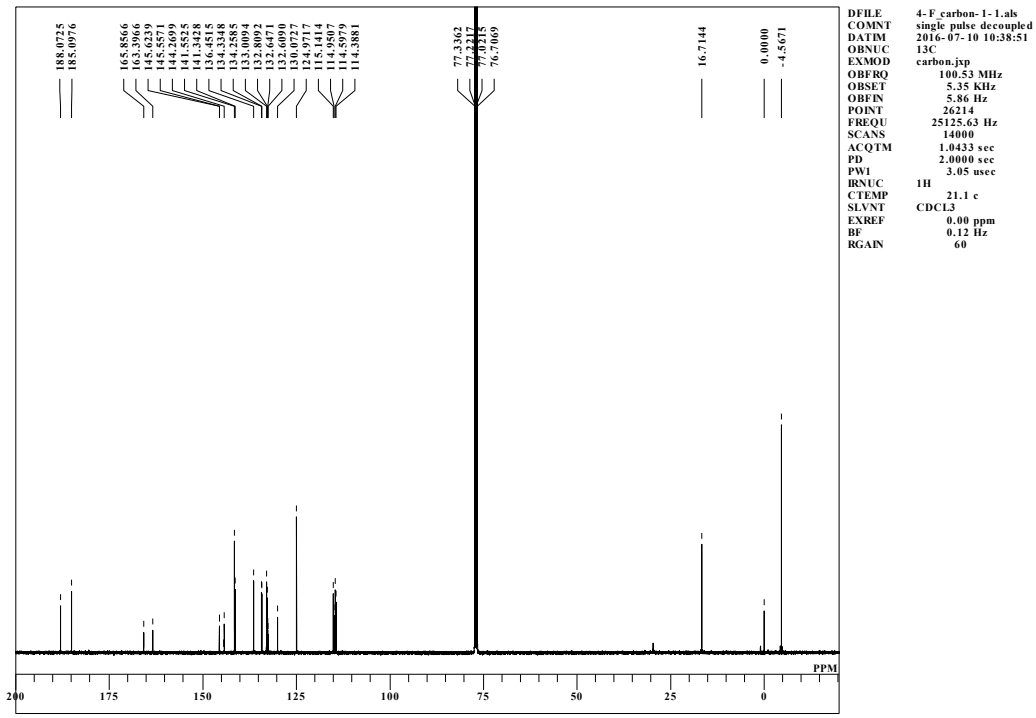
3d

single_pulse



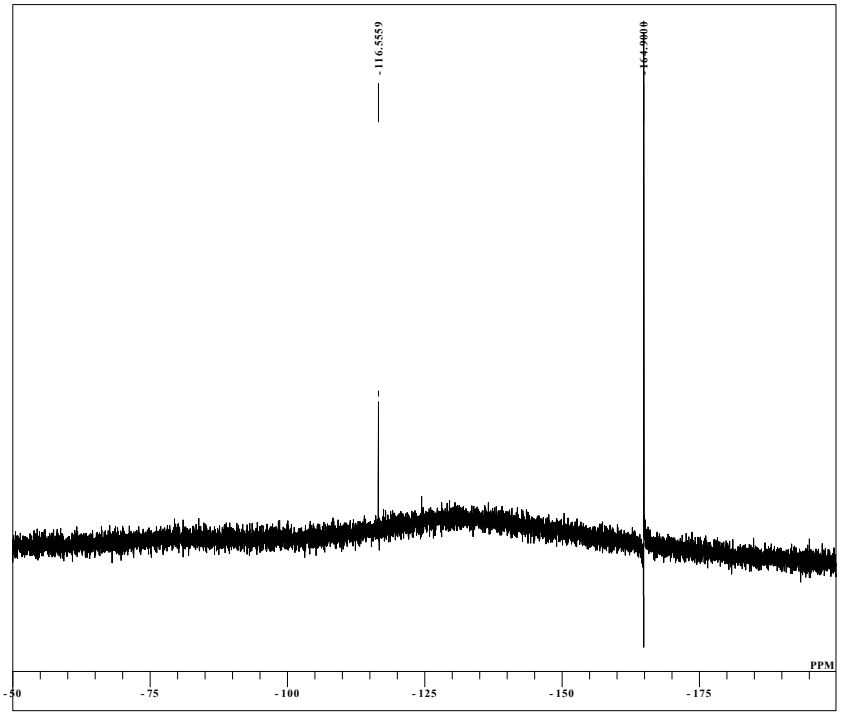
DFILE 4-F_proton-1-1.jdf
COMNT single_pulse
DATIM 2016-07-08 01:19:06
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 16384
FREQU 5635.71 Hz
SCANS 16
ACQTM 2.9072 sec
PD 5.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

single pulse decoupled gated NOE



DFILE 4-F_carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-07-10 10:38:51
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 14000
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60

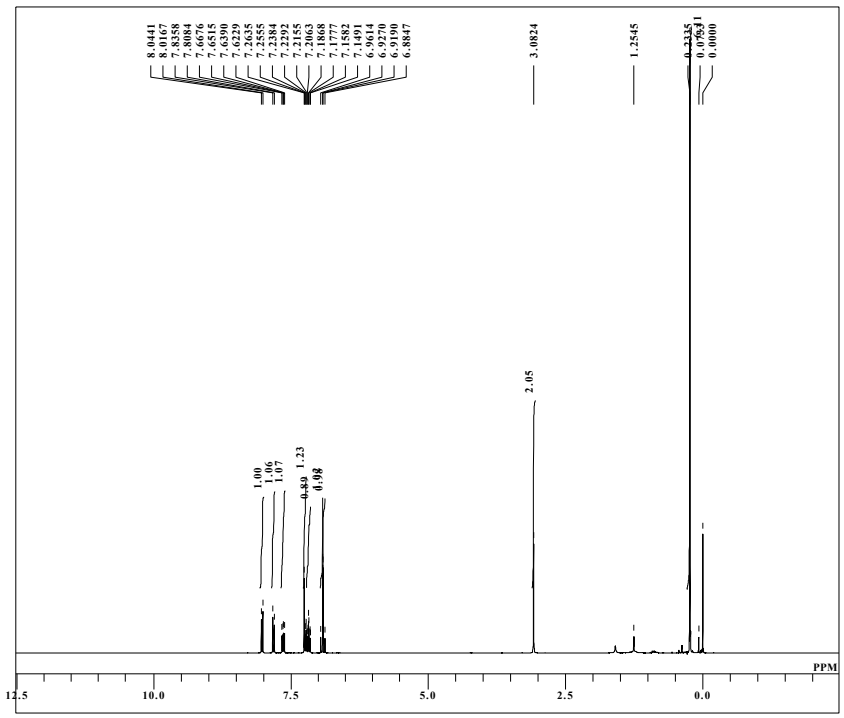
single_pulse



DFILE	4-F_single_pulse-1-1.als
COMNT	single_pulse
DATIM	02-07-2016 20:32:45
OBNUC	19F
EXMOD	single_pulse.jxp
OBFRQ	470.57 MHz
OBSET	3.39 KHz
OBFIN	8.79 Hz
POINT	13107
FREQU	94339.62 Hz
SCANS	16
ACQTM	0.1389 sec
PD	5.0000 sec
PW1	5.80 usec
IRNUC	19F
CTEMP	21.5 c
SLVNT	CDCl3
EXREF	-164.90 ppm
BF	0.12 Hz
RGAIN	42

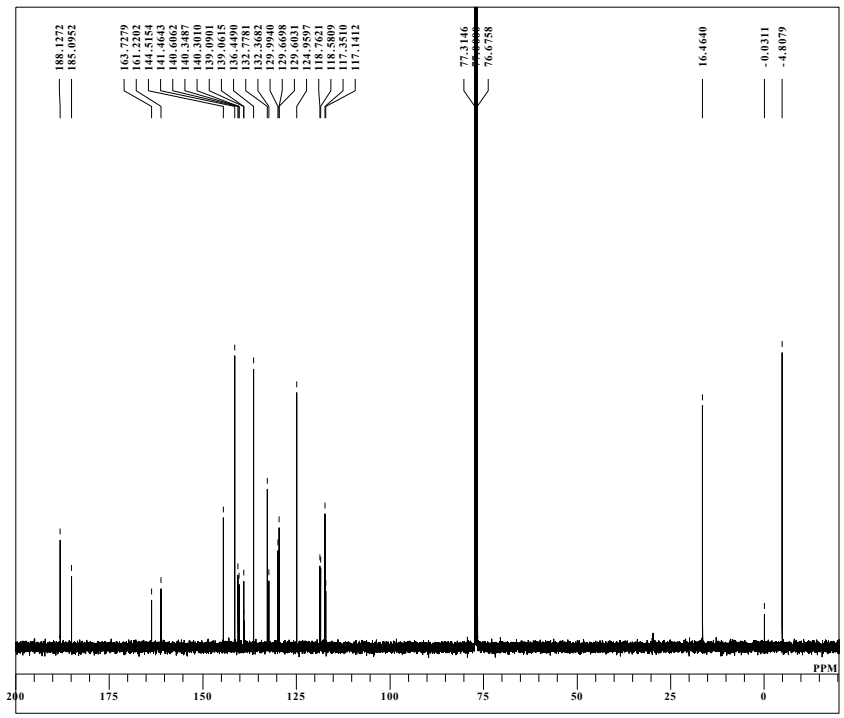
3e

single_pulse



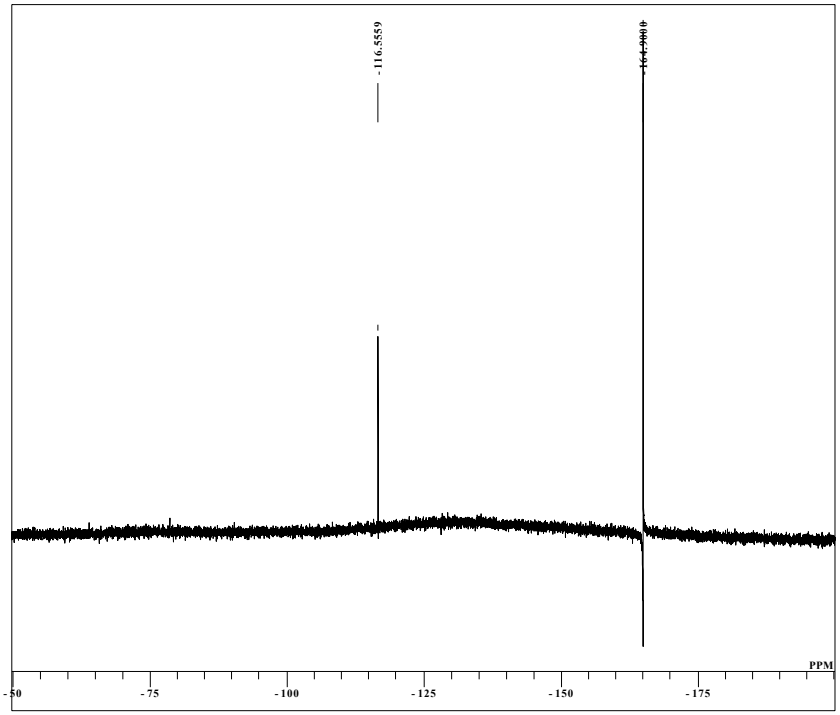
DFILE 5-F_proton-1-1.als
COMNT single_pulse
DATIM 01-06-2016 22:58:51
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 13107
FREQU 4508.57 Hz
SCANS 8
ACQTM 2.9072 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

single pulse decoupled gated NOE



DFILE 5-F_carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-06-03 00:19:49
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 800
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

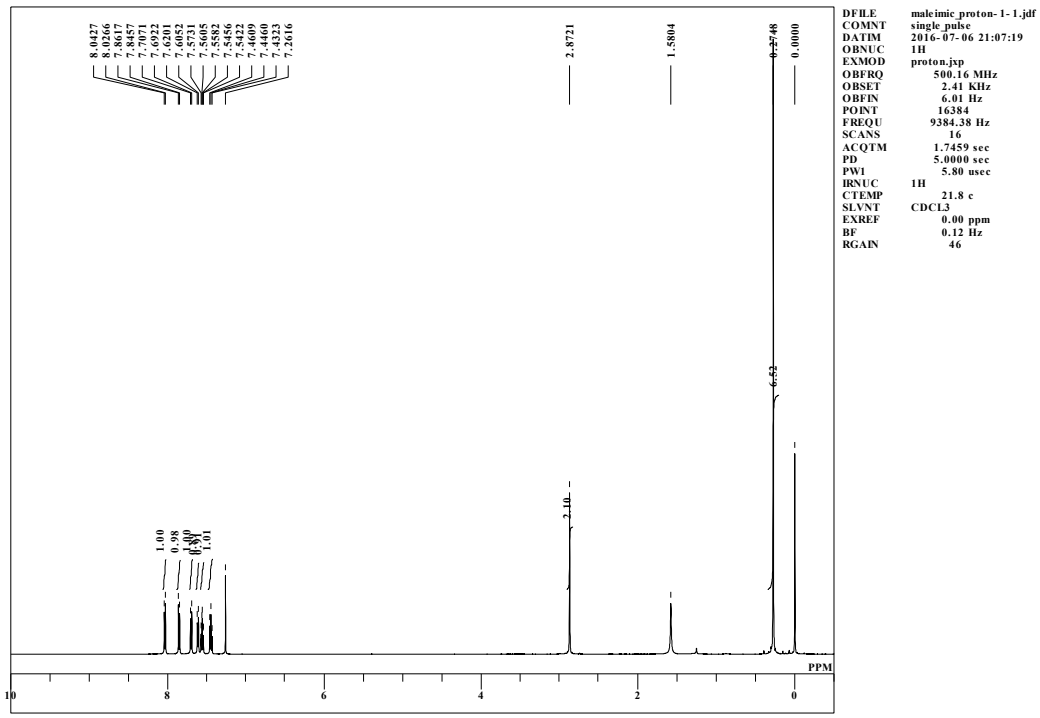
single_pulse



DFILE	5-F_single_pulse-2-1.als
COMNT	single_pulse
DATIM	02-07-2016 20:23:19
OBNUC	19F
EXMOD	single_pulse.jxp
OBFRQ	470.57 MHz
OBSET	3.39 KHz
OBFIN	8.79 Hz
POINT	13107
FREQU	94339.62 Hz
SCANS	16
ACQTM	0.1389 sec
PD	5.0000 sec
PW1	5.80 usec
IRNUC	19F
CTEMP	21.5 c
SLVNT	CDCl3
EXREF	-164.90 ppm
BF	0.12 Hz
RGAIN	42

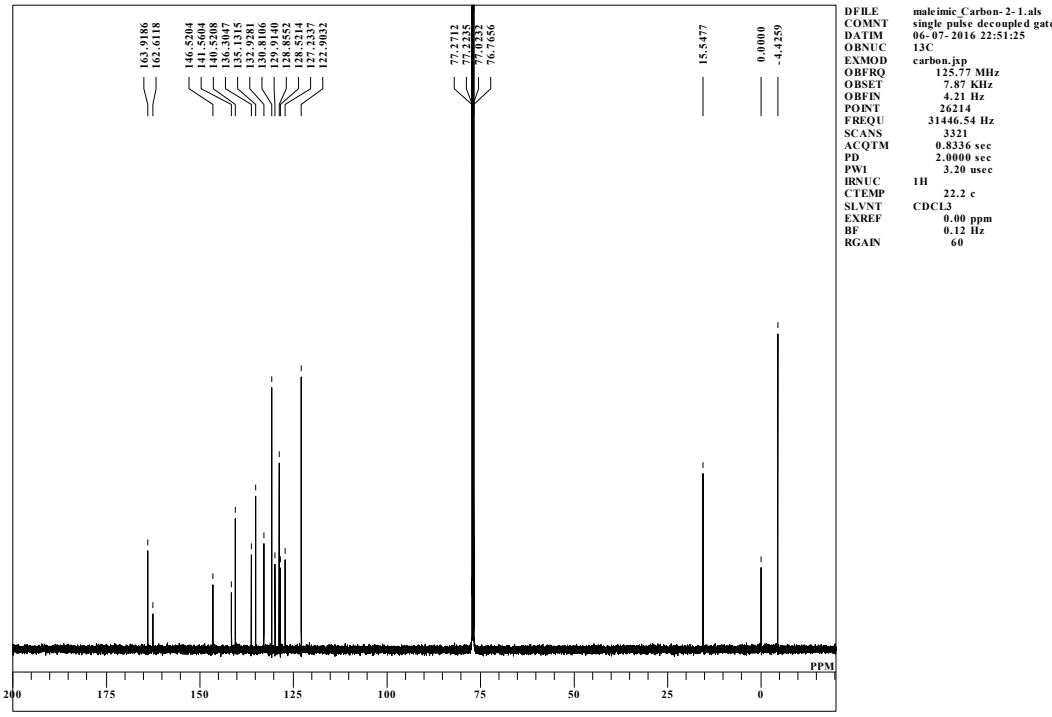
3f

single_pulse



DFILE maleimic_proton-1-1.jdf
COMNT single_pulse
DATIM 2016-07-06 21:07:19
OBNUC 1H
EXMOD proton.jxp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 16
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.80 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

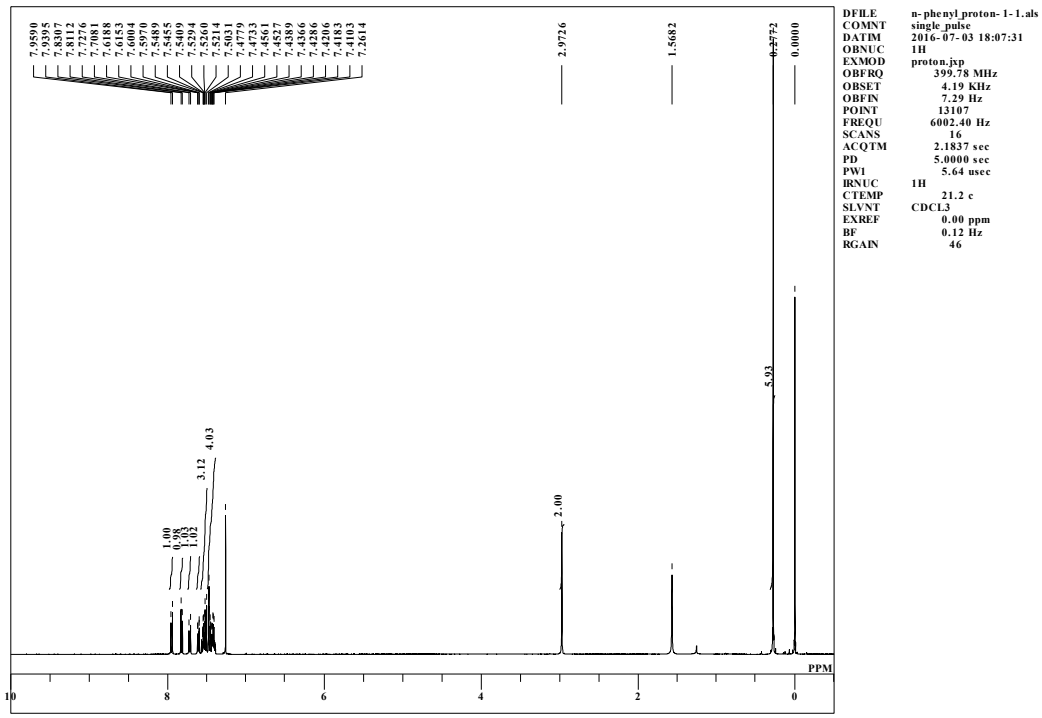
single pulse decoupled gated NOE



DFILE maleimic_Carbon-2-1.als
COMNT single pulse decoupled gated NOE
DATIM 06-07-2016 22:51:25
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.54 Hz
SCANS 3321
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.20 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60

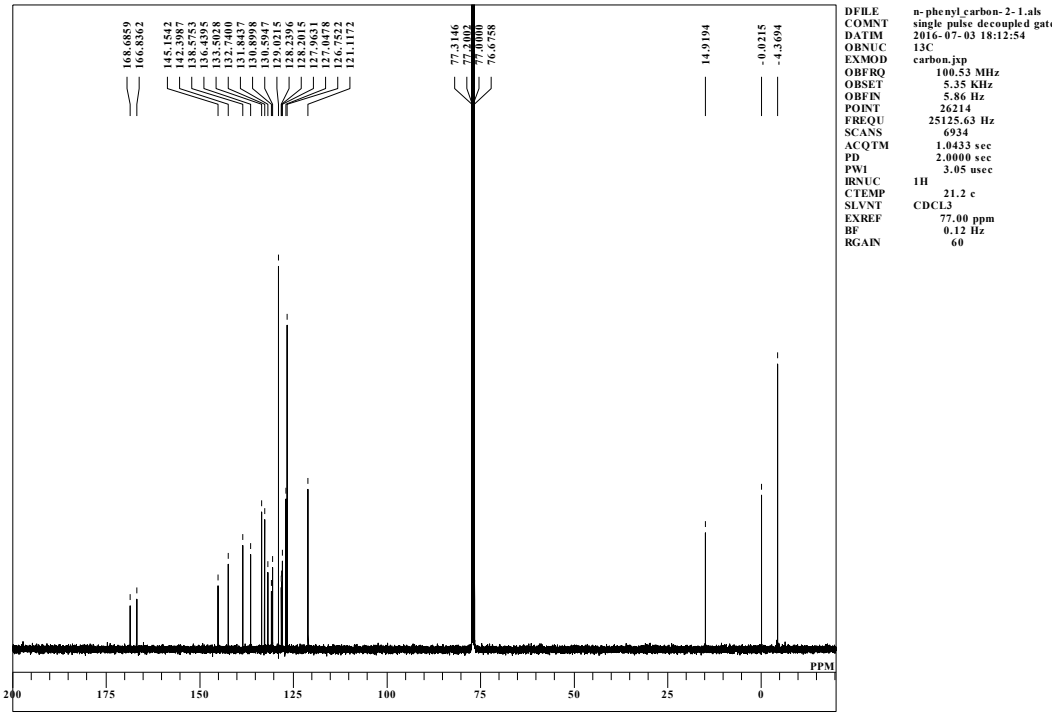
3g

single_pulse



DFILE n-phenyl_proton-1-1.als
COMNT single_pulse
DATIM 2016-07-03 18:07:31
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.64 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

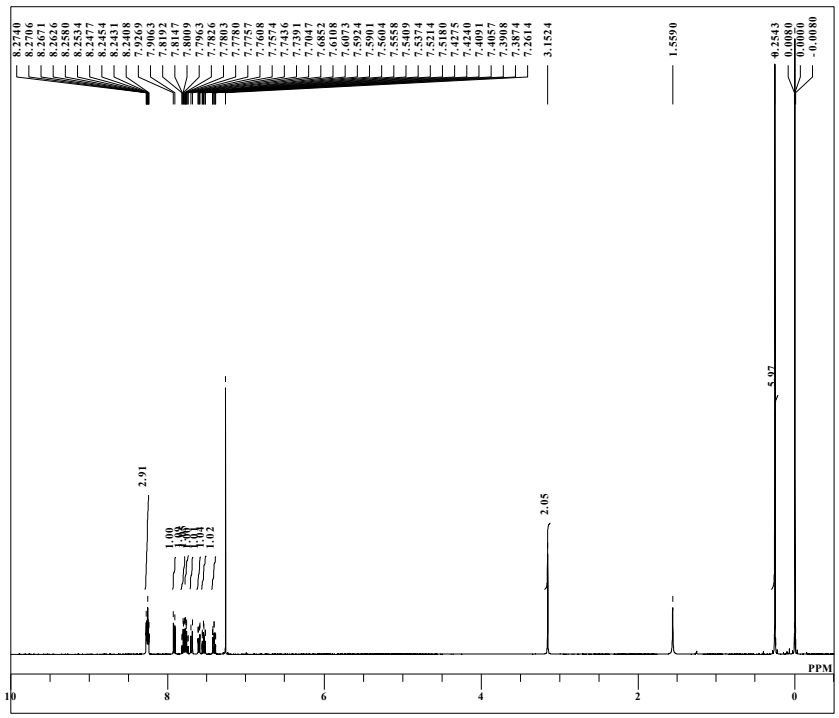
single pulse decoupled gated NOE



DFILE n-phenyl_carbon-2-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-07-03 18:12:54
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 6934
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

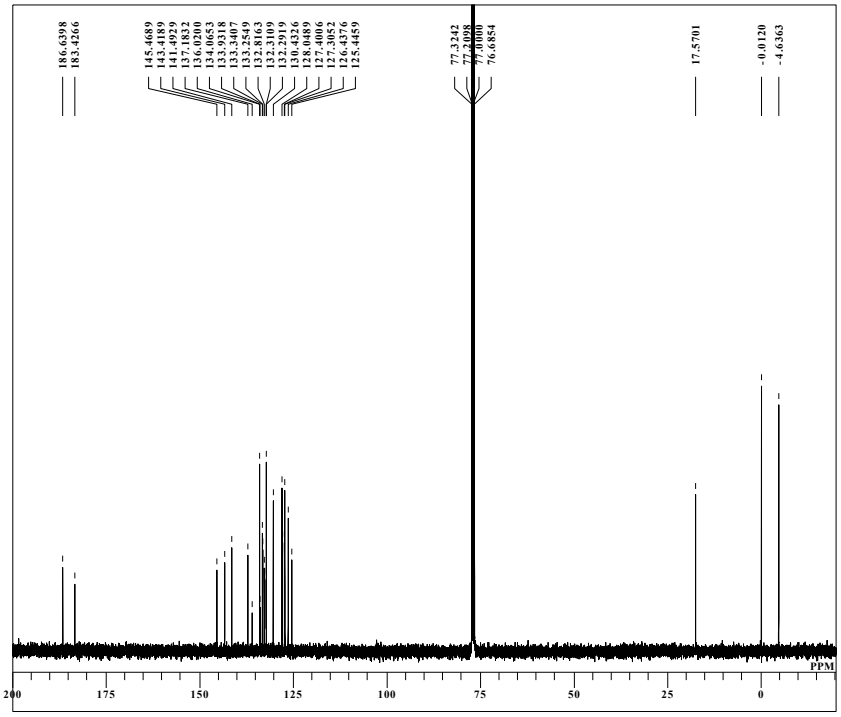
3h

single_pulse



DFILE naphto_proton-1-1.als
COMNT single_pulse
DATIM 2016-07-03 17:58:51
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.64 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

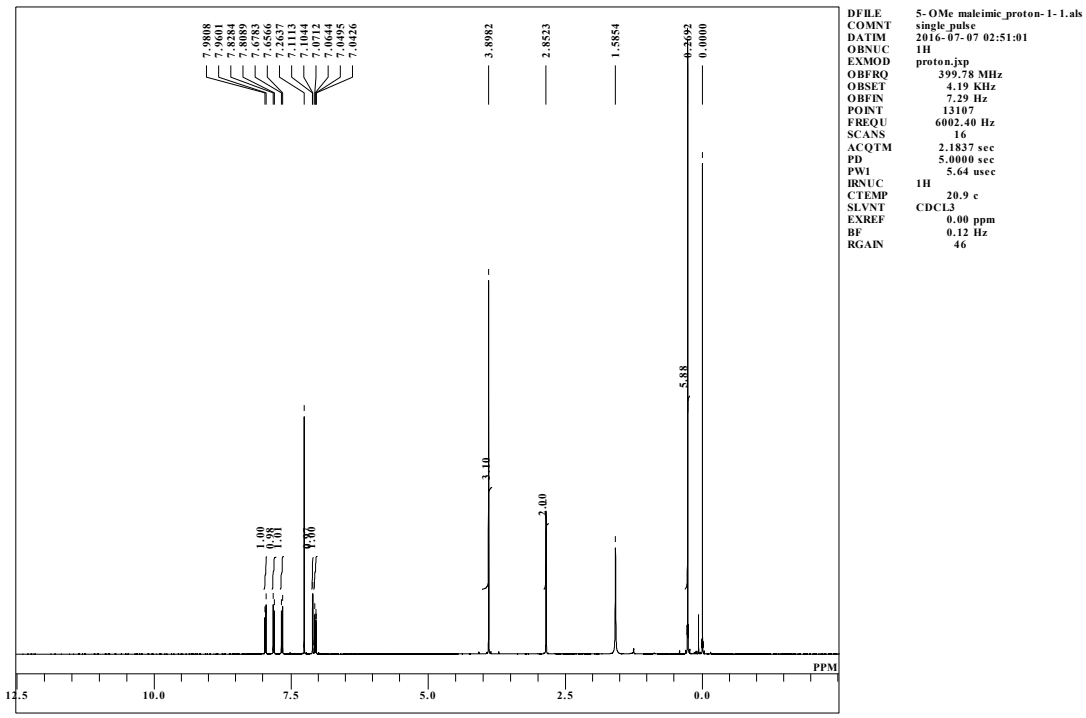
single pulse decoupled gated NOE



DFILE naphto_carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-07-03 10:18:50
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 8904
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

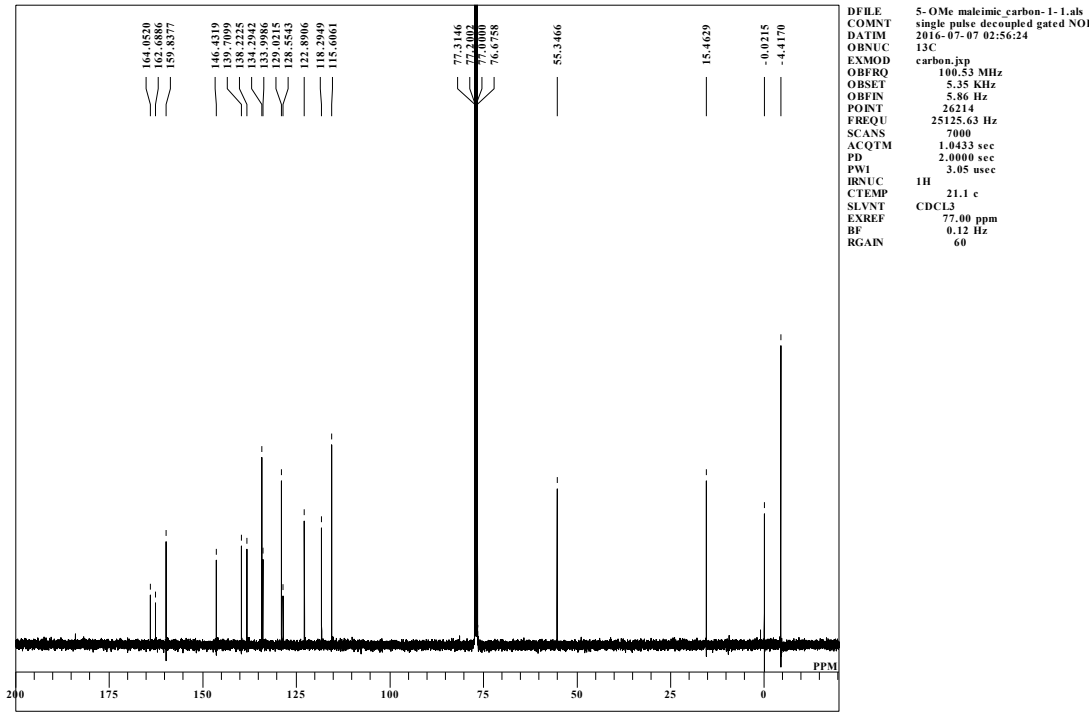
3i

single_pulse



DFILE	5-OMe maleimic_proton-1-1.als
COMNT	single_pulse
DATIM	2016-07-07 02:51:01
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	16
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.64 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	46

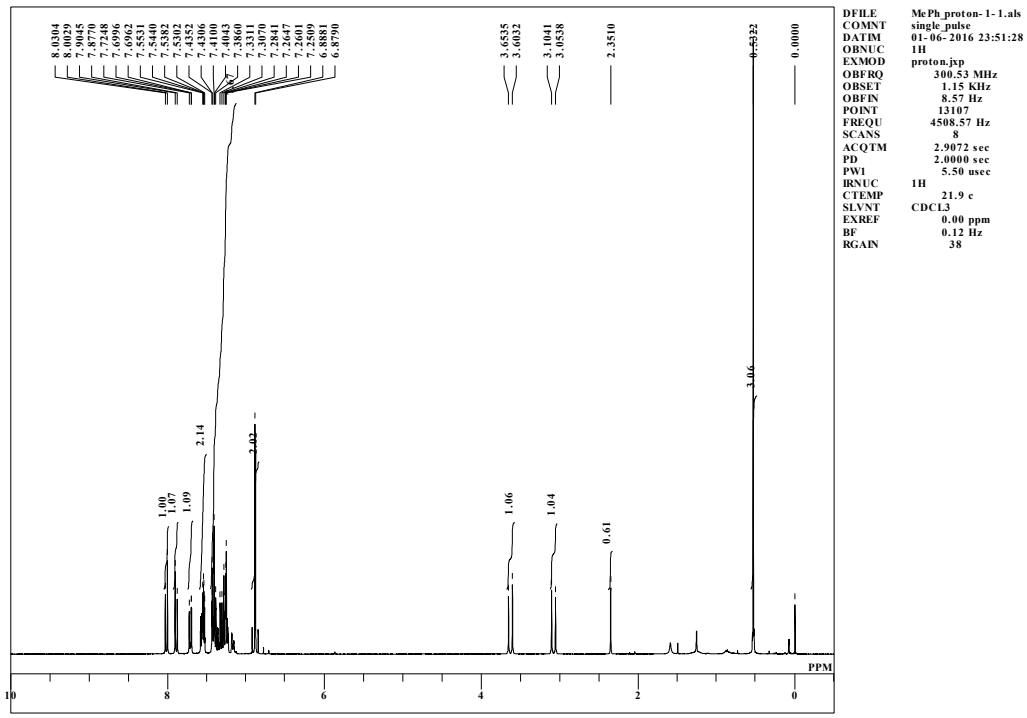
single pulse decoupled gated NOE



DFILE	5-OMe maleimic_carbon-1-1.als
COMNT	single pulse decoupled gated NOE
DATIM	2016-07-07 02:56:24
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	7000
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.05 usec
IRNUC	1H
CTEMP	21.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

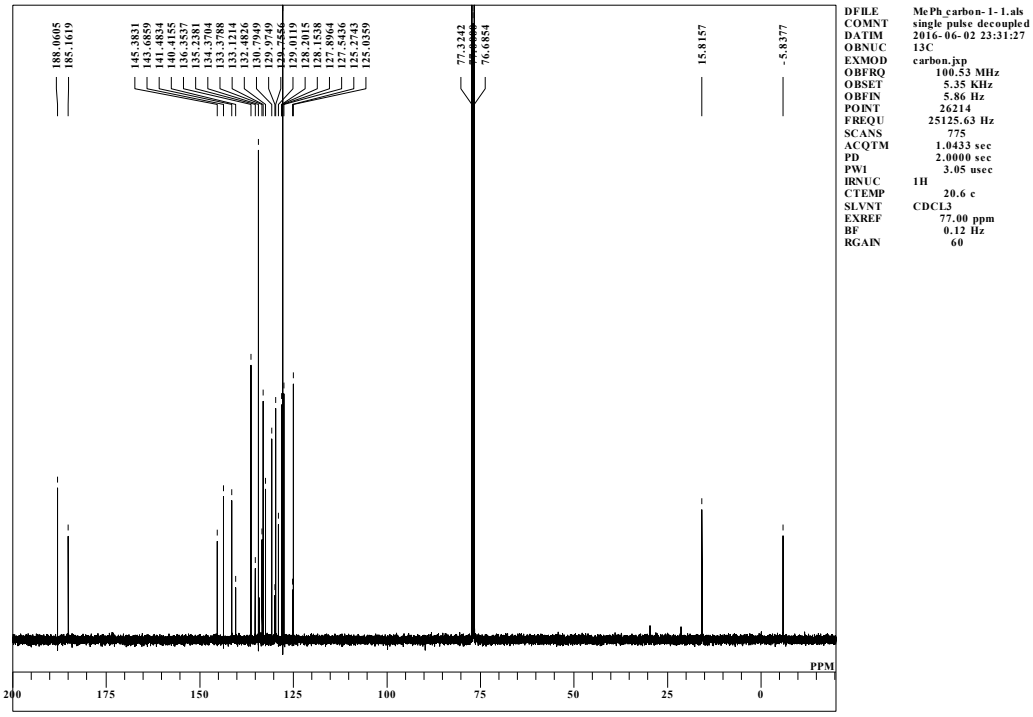
3j

single_pulse



DFILE MePh_proton-1-1.als
COMNT single_pulse
DATIM 01-06-2016 23:51:28
OBNUC 1H
EXMOD proton.jxp
OBFRQ 300.53 MHz
OBSET 1.15 KHz
OBFIN 8.57 Hz
POINT 13107
FREQU 4508.57 Hz
SCANS 8
ACQTM 2.9072 sec
PD 2.0000 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38

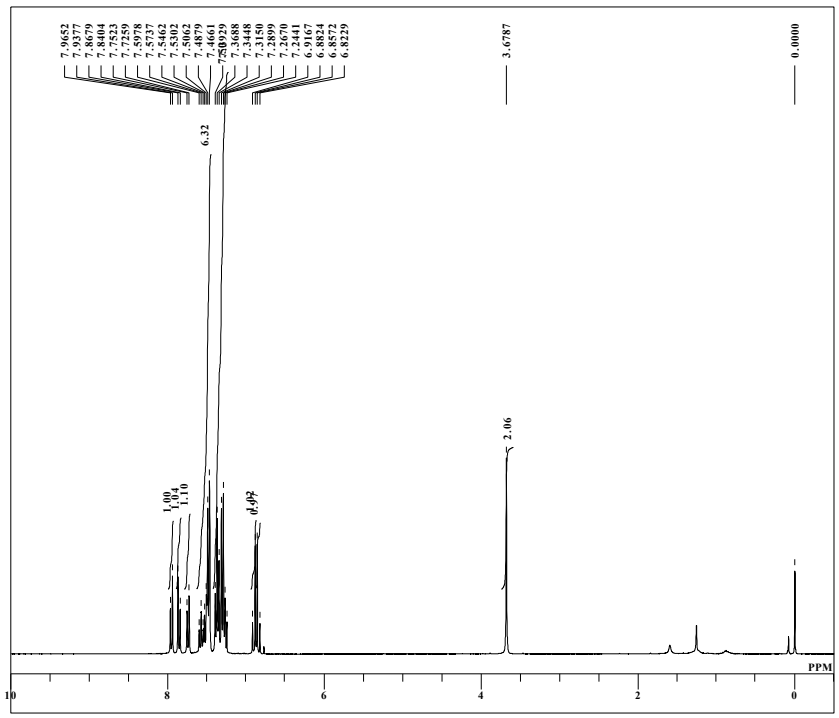
single pulse decoupled gated NOE



DFILE MePh_carbon-1-1.als
COMNT single pulse decoupled gated NOE
DATIM 2016-06-02 23:31:27
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 775
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

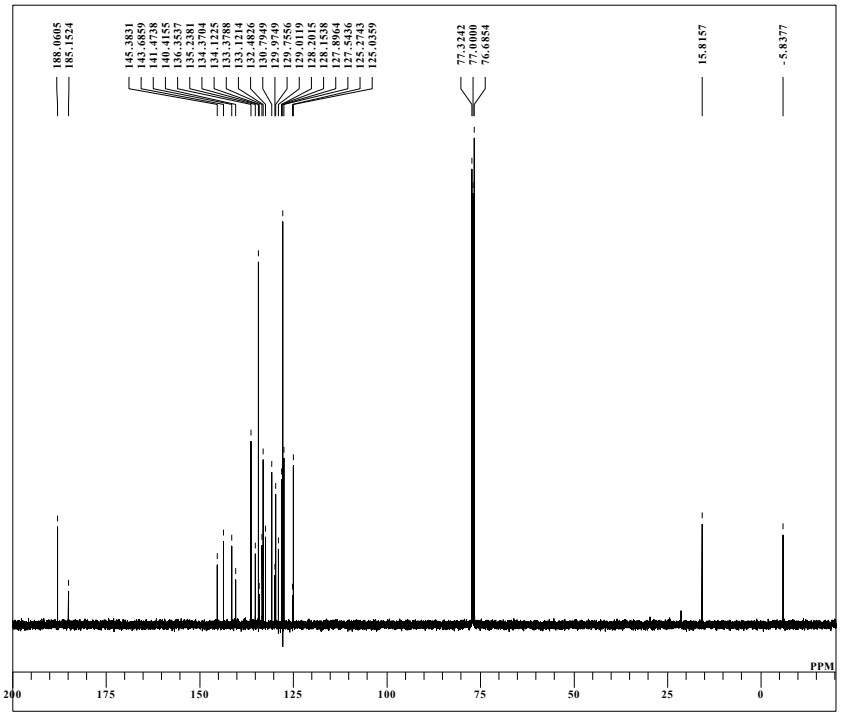
3k

single_pulse



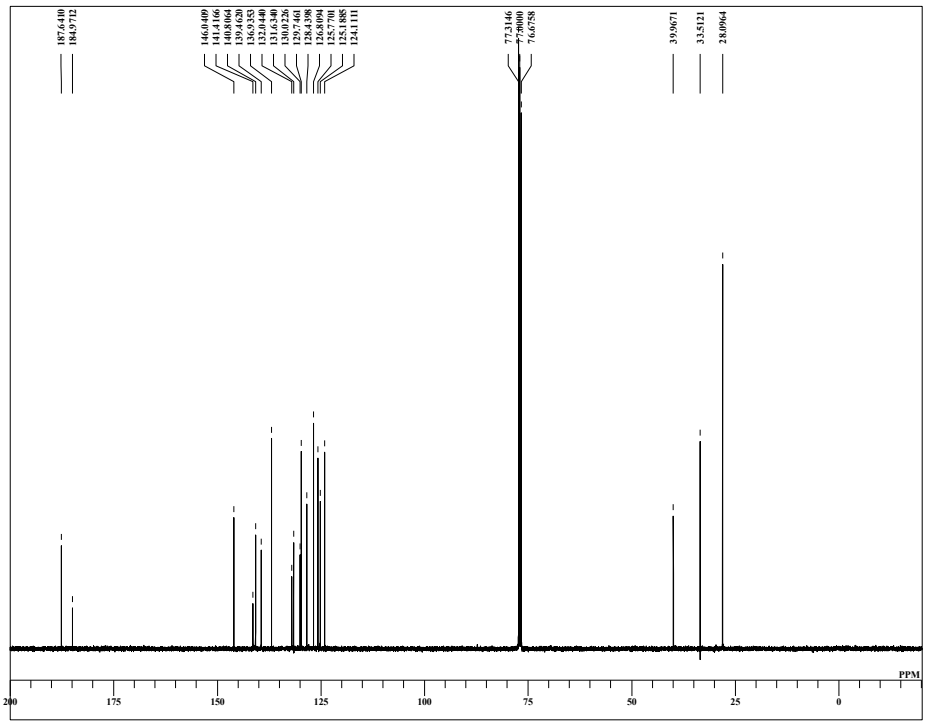
DFILE	PhPh_proton-1-1.als
COMNT	single_pulse
DATIM	01-06-2016 23:58:01
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	300.53 MHz
OBSET	1.15 KHz
OBFIN	8.57 Hz
POINT	13107
FREQU	4508.57 Hz
SCANS	8
ACQTM	2.9072 sec
PD	2.0000 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	21.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

single pulse decoupled gated NOE

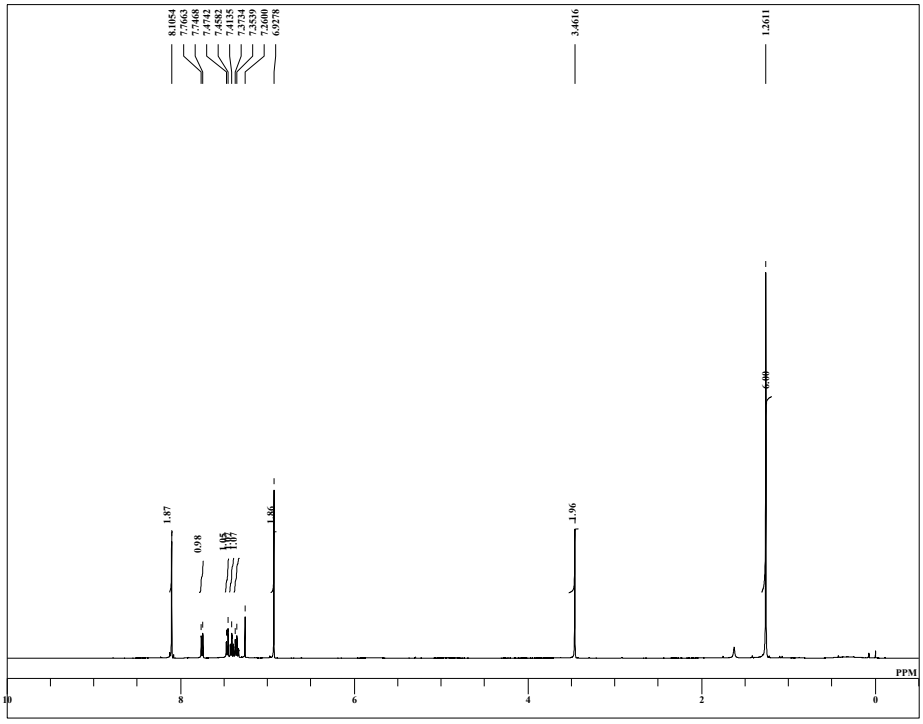


DFILE	PhPh_carbon-1-1.als
COMNT	single pulse decoupled gated NOE
DATIM	2016-06-02 10:26:56
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	433
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.05 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60

3l

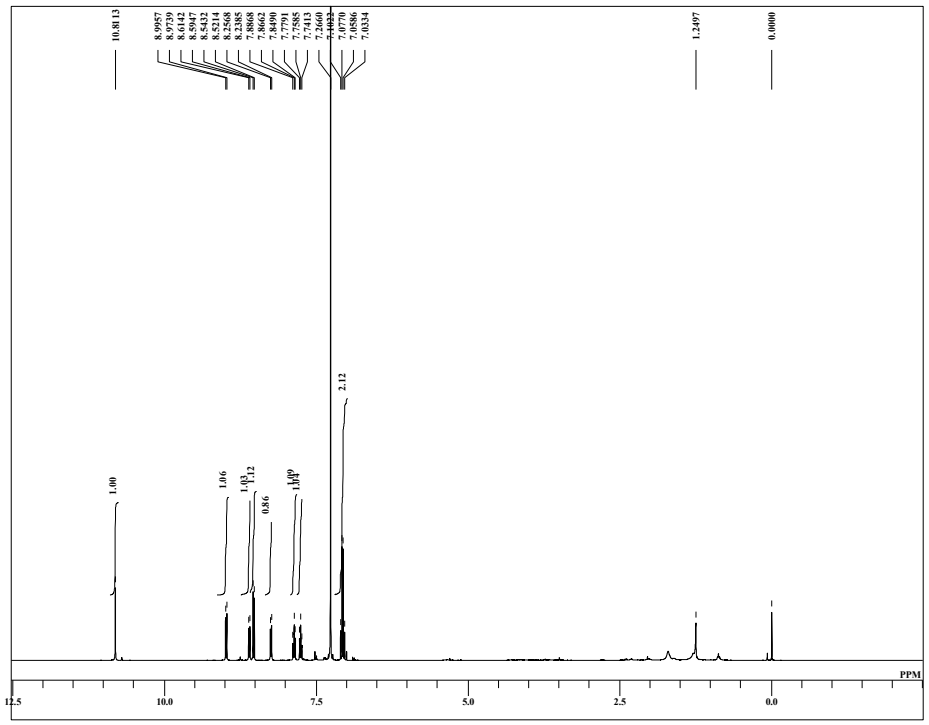


DFILE C_carbon-1-1.als
COMNT single pulse decoupled ga
DATIM 2016-11-13 12:59:56
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 3040
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

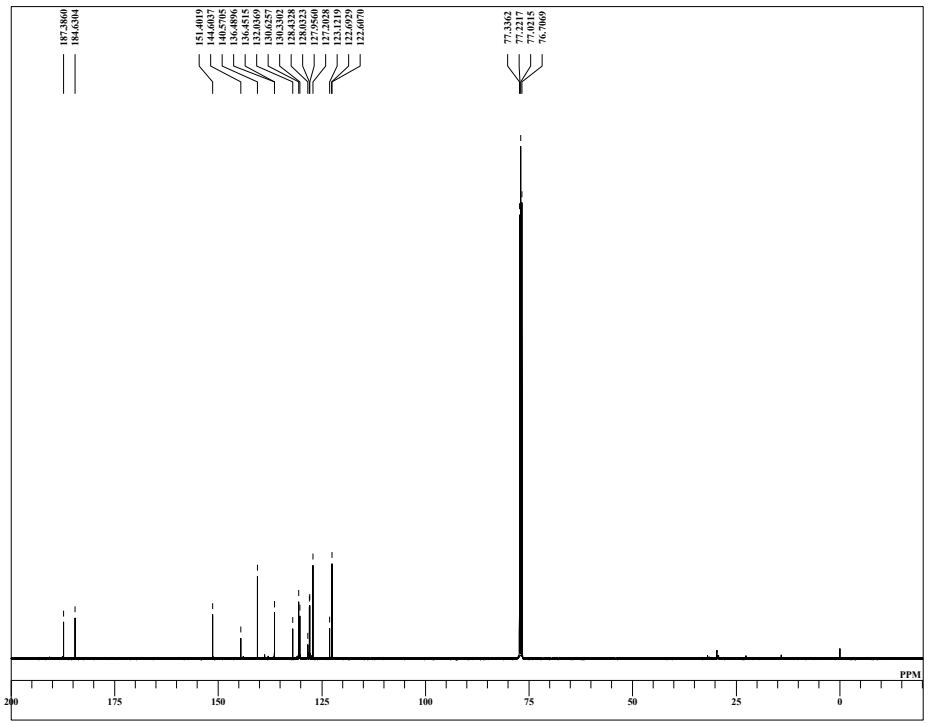


DFILE C_proton-2-1.jdf
COMNT single_pulse
DATIM 13-11-2016 12:54:39
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.64 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.12 Hz
RGAIN 38

3m

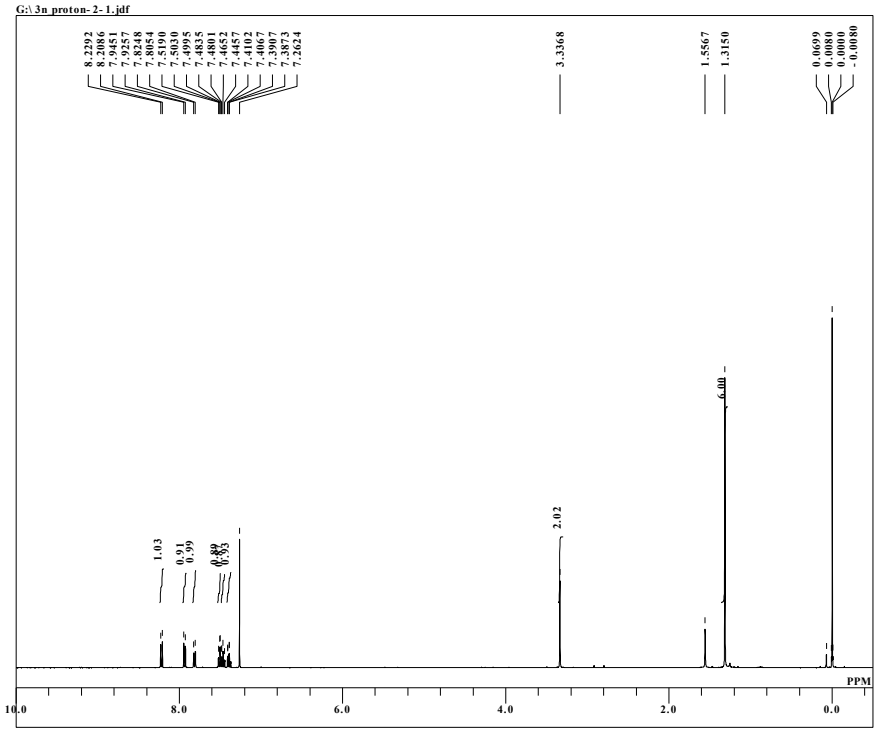


DFILE N_proton-3-1.als
COMNT single_pulse
DATIM 2016-11-26 00:04:04
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.64 usec
IRNUC 1H
CTEMP 19.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

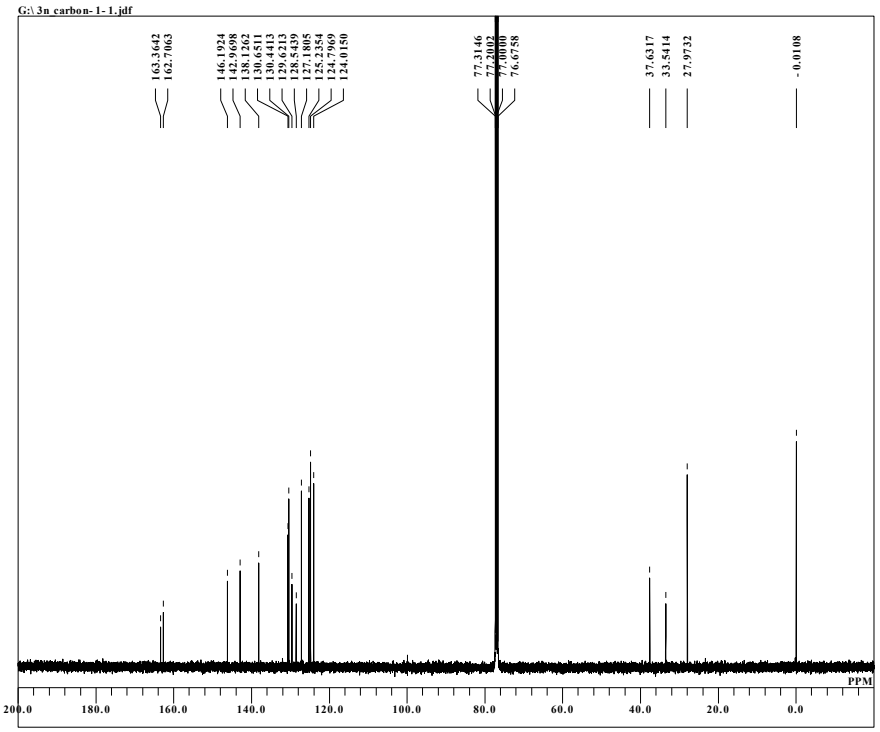


DFILE N_carbon-1-1.als
COMNT single pulse decoupled ga
DATIM 2016-11-26 00:09:19
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 11000
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60

3n



DFILE 3n_proton-2-1.jdf
COMNT single_pulse
DATIM 2017-04-01 23:25:56
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 16384
FREQU 7503.00 Hz
SCANS 16
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.64 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50



DFILE 3n_carbon-1-1.jdf
COMNT single pulse decoupled gated N
DATIM 2017-04-01 23:31:13
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 19043
ACQTM 1.0433 sec
PD 2.5000 sec
PW1 3.05 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60