Supporting Information

Photochemically Induced Radical Alkenylation of C(sp^3)–H Bonds

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Contents

General Information

All reactions sensitive to air or moisture were carried out under argon atmosphere and anhydrous conditions unless otherwise noted. Reagents were used as supplied unless otherwise stated. Analytical thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 pre-coated plates. Flash column chromatography was performed using 40-50 μm Silica Gel 60N (Kanto Chemical Co., Inc.). ^{1}H and ^{13}C NMR spectra were recorded on JEOL JNM-ECX-500 (500 MHz), JNM-ECA-500 (500 MHz), JNM-ECS-400 (400 MHz), or Bruker AVANCE III-400 (400 MHz) spectrometer. Chemical shifts are reported in δ (ppm) with reference to residual solvent signals [^{1}H NMR: $CHCl_3$ (7.26); ^{13}C NMR: $CDCl_3$ (77.0), DMSO-d_6 (39.5)]. Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. Infrared (IR) spectra were recorded on a JASCO FT/IR-4100 spectrometer. High resolution mass spectra (HRMS) were recorded on a JEOL JMS-T100LP (ESI) or BRUKER DALTONICS micrOTOF II (APCI) instrument. Melting points were measured on a Yanaco MP-J3 or CORNES MPA100 micro melting point apparatus. UV irradiation was carried out by using a Riko 100 W medium-pressure mercury lamp.

General Procedure for Synthesis of 3

Conditions A:

The solution of 5-methyl-2-pyrrolidone **1a** (27.3 mg, 0.275 mmol), benzophenone (50.2 mg, 0.275 mmol) and *trans*-1,2-bis(phenylsulfonyl)ethylene (102 mg, 0.330 mmol) in *t*-butyl acetate (5.5 mL) was irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 24 h. The reaction mixture was then neutralized with saturated aqueous $NaHCO_3$, and extracted with EtOAc (3 mL ×3). The combined organic layers were dried over Na_2SO_4, and concentrated. The residue was purified with flash column chromatography (silica gel, toluene-acetone 3:1) to provide (*E*)-5-methyl-5-(2-(phenylsulfonyl)vinyl)-2-pyrrolidone **3a** in 77% yield (56.2 mg).

Conditions B:

The solution of 5-methyl-2-pyrrolidone **1a** (97.1 mg, 0.980 mmol), benzophenone (35.7 mg, 0.196 mmol) and *trans*-1,2-bis(phenylsulfonyl)ethylene (60.5 mg, 0.196 mmol) in acetonitrile (3.9 mL) was irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 5 h. The reaction mixture was then neutralized with saturated aqueous $NaHCO_3$, and extracted with EtOAc (3 mL ×3). The combined organic layers were dried over Na_2SO_4, and concentrated. The residue was purified with flash column chromatography (silica gel, toluene-acetone 3:1) to provide (*E*)-5-methyl-5-(2-(phenylsulfonyl)vinyl)-2-pyrrolidone **3a** in 94% yield (49.0 mg).

Preparation of 3a on a gram scale

The solution of 5-methyl-2-pyrrolidone **1a** (3.22 g, 31.8 mmol), benzophenone (2.32 g, 6.36 mmol) and *trans*-1,2-bis(phenylsulfonyl)ethylene (2.00 g, 6.36 mmol) in acetonitrile (127 mL) was irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 5 h. The reaction mixture was then neutralized with saturated aqueous $NaHCO_3$, and extracted with EtOAc (40 mL ×3). The combined organic layers were dried over Na_2SO_4, and concentrated. The residue was purified with flash column chromatography (silica gel, toluene-acetone 3:1 to 2:1) to provide (*E*)-5-methyl-5-(2-(phenylsulfonyl)vinyl)-2-pyrrolidone **3a** in 92% yield (1.55 g).

Measurement of the kinetic isotope effect:

To a solution of benzophenone (38.8 mg, 0.213 mmol) and *trans*-1,2-bis(phenylsulfonyl)ethylene (65.6 mg, 0.213 mmol) in acetonitrile (8.6 mL) was added cyclohexane **1w** (179 mg, 2.13 mmol) and cyclohexane-d_{12} **1w-d_{12}** (205 mg, 2.13 mmol). The solution was irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 9 h. The reaction mixture was then neutralized with saturated aqueous $NaHCO_3$, and extracted with EtOAc (3 mL ×3). The combined organic layers were dried over Na_2SO_4, and concentrated. The residue was purified with flash column chromatography (silica gel, hexane-ether 5:1) to provide the 4:1 mixture of

(*E*)-(2-(phenylsulfonyl)vinyl)cyclohexane **3w** and (*E*)-(2-(phenylsulfonyl)vinyl)cyclohexane-d_{11} **3w**-$\boldsymbol{d_{11}}$.

General Procedure for Synthesis of 4

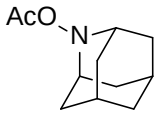
To a solution of **3a** (25.0 mg, 0.0942 mmol) in isopropanol (9.4 mL) was added acetone (547 mg, 9.42 mmol) at room temperature. The mixture was irradiated with a Riko 100 W medium-pressure mercury lamp at room temperature for 24 h. The reaction mixture was then neutralized with saturated aqueous $NaHCO_3$, and extracted with EtOAc (3 mL ×3). The combined organic layers were dried over Na_2SO_4, and concentrated. The residue was purified with flash column chromatography (silica gel, chloroform-methanol 15:1) to provide (*E*)-5-(3-hydroxy-3-methyl-1-butenyl)-5-methyl-2-pyrrolidone **4a** in 65% yield (11.2 mg).

General Procedure for Synthesis of 5

To a suspension of *t*-BuOK (65.1 mg, 0.58 mmol, 6 equiv) in THF (0.2 mL) was added ethyl isocyanoacetate (52.7 μL, 0.48 mmol, 5 equiv) and then the mixture was stirred at 0 °C for 15 min. A solution of vinyl sulfone **3h** (33.8 mg, 0.096 mmol, 1 equiv) in THF (0.2 mL) was added dropwise, and the resulting solution was stirred for 15 min at room temperature and for 60 min at 60 °C. The mixture was cooled to room temperature. The reaction was quenched with saturated aqueous NH_4Cl and extracted with EtOAc. The extracts were combined and washed with brine, dried over Na_2SO_4, and concentrated. The residue was purified with flash column chromatography (DNH-Silica Gel [DNH MB 100-40/75, Fuji Silysia Chemical Ltd.], hexane-AcOEt 5:1 to 2:1) to provide pyrrole **5h** in 69% yield (21.5 mg).

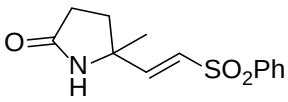
Analytical Data

***Compound* 1s**:



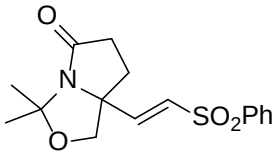
colorless oil; IR (film) 1762, 1446, 1363, 1229, 1206, 1102 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.47 (2H, m), 1.80-1.87 (4H, m), 1.95 (2H, m), 2.08 (3H, s), 2.18 (2H, m), 2.32 (2H, m), 3.43 (2H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.9, 25.7, 26.2, 30.2, 36.1, 36.5, 55.6, 169.5; HRMS (ESI) calcd for $C_{11}H_{17}NNaO_2$ $[M+Na]^+$ 218.1151, found 218.1140.

***Compound* 3a**:



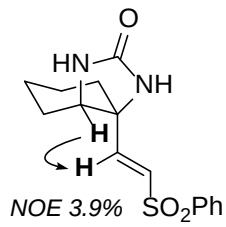
94% yield (49.0 mg); colorless solid; m.p. 159-160 °C; IR (film) 3339, 3235, 3060, 1696, 1307, 1146, 1086, 754, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.46 (3H, s), 2.05-2.19 (2H, m), 2.32-2.38 (2H, m), 5.58 (1H, br s), 6.43 (1H, d, *J* = 15.5 Hz), 6.98 (1H, d, *J* = 15.5 Hz), 7.57 (2H, t, *J* = 7.5 Hz), 7.66 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.6, 29.6, 34.1, 59.6, 127.5, 128.9, 129.3, 133.6, 139.8, 149.2, 177.4; HRMS (ESI) calcd for $C_{13}H_{15}NNaO_3S$ $[M+Na]^+$ 288.0665, found 288.0674.

***Compound* 3b**:



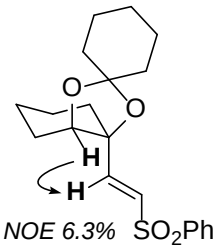
88% yield (58.5 mg); colorless oil; IR (film) 1698, 1447, 1367, 1308, 1147, 1086, 1049, 756, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, s), 1.61 (3H, s), 2.06-2.18 (2H, m), 2.50 (1H, ddd, *J* = 11.8, 7.5, 2.0 Hz), 2.74 (1H, ddd, *J* = 17.0, 11.8, 9.0 Hz), 3.78 (1H, d, *J* = 9.2 Hz), 4.10 (1H, d, *J* = 9.2 Hz), 6.57 (1H, d, *J* = 15.1 Hz), 7.04 (1H, d, *J* = 15.1 Hz), 7.56 (2H, t, *J* = 7.5 Hz), 7.65 (1H, t, *J* = 7.5 Hz), 7.89 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.3, 27.0, 30.8, 35.2, 71.5, 73.1, 93.4, 127.6, 129.5, 131.5, 133.8, 139.7, 145.5, 172.9; HRMS (ESI) calcd for $C_{16}H_{19}NNaO_4S$ $[M+Na]^+$ 344.0927, found 344.0926.

***Compound* 3c**:



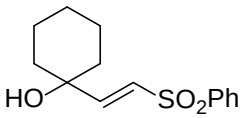
81% yield (41.2 mg); colorless solid; m.p. 209-210 °C; IR (film) 3362, 3223, 3059, 1698, 1446, 1308, 1146, 1086, 752, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.41-1.80 (8H, m), 3.53 (1H, m), 4.31 (1H, br s), 4.33 (1H, br s), 6.60 (1H, d, *J* = 15.5 Hz), 7.05 (1H, d, *J* = 15.5 Hz), 7.56 (2H, t, *J* = 7.5 Hz), 7.65 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, DMSO-d_6) δ 19.3, 19.8, 27.7, 31.5, 54.7, 59.2, 127.2, 129.3, 129.7, 133.8, 140.2, 149.6, 162.0; HRMS (ESI) calcd for $C_{15}H_{18}N_2NaO_3S$ $[M+Na]^+$ 329.0930, found 329.0942.

***Compound* 3d**:



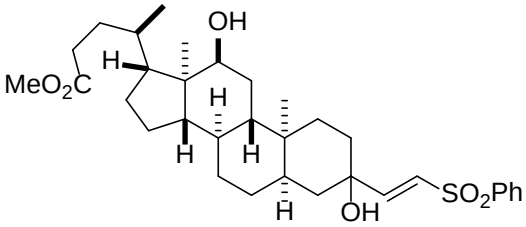
83% yield (48.8 mg); colorless solid; m.p. 118-119 °C; IR (film) 3060, 1629, 1447, 1366, 1308, 1146, 1117, 1085, 747, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.25-1.75 (17H, m), 2.15 (1H, m), 3.90 (1H, t, *J* = 3.0 Hz), 6.69 (1H, d, *J* = 15.0 Hz), 7.11 (1H, d, *J* = 15.0 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.63 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.0, 21.8, 23.6, 23.8, 24.9, 26.0, 34.0, 35.5, 37.8, 76.5, 79.4, 109.1, 127.6, 129.3, 129.6, 133.4, 140.2, 145.4; HRMS (ESI) calcd for $C_{20}H_{26}NaO_4S$ $[M+Na]^+$ 385.1444, found 385.1435.

***Compound* 3e**[1] [CAS 129083-25-6]:



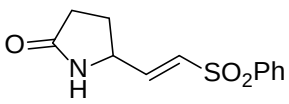
95% yield (45.8 mg); colorless solid; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (1H, m), 1.46-1.65 (10H, m), 6.60 (1H, d, *J* = 15.0 Hz), 7.04 (1H, d, *J* = 15.0 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 24.9, 36.7, 72.0, 127.6, 128.3, 129.2, 133.3, 140.4, 152.3.

***Compound* 3f**:



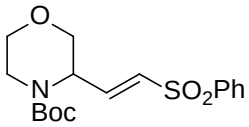
75% yield (dr = 1.7:1, 75.2 mg); colorless oil; IR (film) 3437, 3063, 1735, 1446, 1379, 1307, 1146, 1086, 754, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) major diastereomer; δ 0.69 (3H, s), 0.95-1.91 (31H, m), 2.08-2.27 (2H, m), 2.37 (1H, tt, *J* = 10.5, 5.0 Hz), 3.67 (3H, s), 3.99 (1H, t, *J* = 2.5 Hz), 6.64 (1H, d, *J* = 15.0 Hz), 7.36 (1H, d, *J* = 15.0 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); minor diastereomer; δ 0.69 (3H, s), 0.95-1.72 (28H, m), 1.77-1.95 (3H, m), 2.04 (1H, t, *J* = 13.8 Hz), 2.24 (1H, ddd, *J* = 10.5, 9.0, 7.0 Hz), 2.38 (1H, tt, *J* = 10.5, 5.0 Hz), 3.67 (3H, s), 3.99 (1H, t, *J* = 2.8 Hz), 6.59 (1H, d, *J* = 14.9 Hz), 6.99 (1H, d, *J* = 14.9 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) major diastereomer; δ 12.7, 17.3, 23.0, 23.5, 25.7, 26.7, 27.4, 28.6, 30.9, 31.1, 33.3, 33.6, 33.9, 34.3, 35.1, 35.8, 39.8, 40.4, 46.5, 47.4, 48.2, 51.5, 73.1, 73.3, 127.6, 129.2, 129.3, 133.4, 140.3, 149.2, 174.7; minor diastereomer; δ 12.7, 17.3, 23.4, 23.5, 25.9, 26.3, 27.4, 28.8, 30.7, 30.9, 31.0, 31.1, 32.8, 34.0, 35.0, 35.7, 36.7, 37.6, 46.5, 47.4, 48.2, 51.5, 72.9, 73.1, 127.6, 128.2, 129.3, 133.3, 140.4, 153.1, 174.7; HRMS (ESI) calcd for $C_{33}H_{48}NaO_6S$ $[M+Na]^+$ 595.3064, found 595.3063.

***Compound* 3g**:



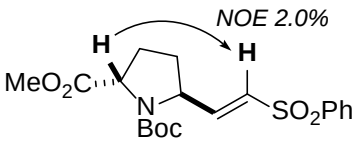
81% yield (50.9 mg); colorless oil; IR (film) 3343, 3239, 3057, 1695, 1633, 1308, 1146, 1086, 755, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.95 (1H, m), 2.30-2.52 (3H, m), 4.37 (1H, m), 5.67 (1H, br s), 6.50 (1H, dd, *J* = 15.4, 1.3 Hz), 6.95 (1H, dd, *J* = 15.4, 5.9 Hz), 7.57 (2H, t, *J* = 7.5 Hz), 7.66 (1H, t, *J* = 7.5 Hz), 7.89 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.8, 29.1, 53.8, 127.7, 129.4, 130.7, 133.7, 139.7, 145.0, 178.3; HRMS (ESI) calcd for $C_{12}H_{13}NNaO_3S$ $[M+Na]^+$ 274.0508, found 274.0505.

***Compound* 3h**:



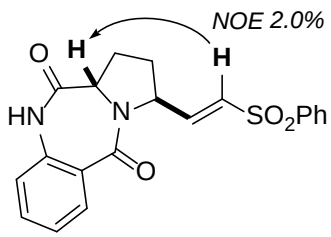
90% yield (60.1 mg); colorless oil; mixture of rotamers; IR (film) 3059, 1697, 1631, 1400, 1308, 1148, 1119, 1086, 755, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, at 50 °C) δ 1.39 (9H, s), 3.06 (1H, m), 3.46 (1H, td, *J* = 12.0, 3.0 Hz), 3.65-3.74 (2H, m), 3.84 (1H, dd, *J* = 12.0, 3.5 Hz), 3.93 (1H, d, *J* = 12.0 Hz), 4.59 (1H, m), 6.48 (1H, dd, *J* = 15.3, 1.2 Hz), 7.02 (1H, dd, *J* = 15.3, 5.6 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.89 (2H, d, *J* = 7.5 Hz); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$, at 50 °C) δ 27.9, 28.1, 28.2, 28.4, 40.0, 51.6, 66.5, 66.8, 68.3, 68.5, 68.7, 80.9, 127.6, 129.2, 129.4, 133.1, 133.3, 140.4, 141.7, 141.8, 142.0, 154.4; HRMS (ESI) calcd for $C_{17}H_{23}NNaO_5S$ $[M+Na]^+$ 376.1189, found 376.1183.

***Compound* 3i**:



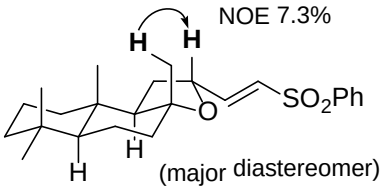
99% yield (59.1 mg); colorless solid; m.p. 180-181 °C; 1.5:1 mixture of rotamers; IR (film) 3057, 1746, 1698, 1447, 1389, 1308, 1147, 1087, 755, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.16 (27/5H, s), 1.35 (18/5H, s), 1.78-1.84 (1H, m), 1.94-2.01 (1H, m), 2.12-2.38 (2H, m), 3.72 (3H, s), 4.30 (2/5H, d, *J* = 8.0 Hz), 4.39 (3/5H, d, *J* = 8.5 Hz), 4.59 (3/5H, m), 4.72 (2/5H, m), 6.31 (3/5H, dd, *J* = 15.0, 1.3 Hz), 6.39 (2/5, dd, *J* = 15.0, 1.3 Hz), 6.83 (3/5, dd, *J* = 15.0, 5.9 Hz), 6.88 (2/5H, dd, *J* = 15.0, 5.7 Hz), 7.48-7.66 (3H, m), 7.85-7.91 (2H, m); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.2, 27.8, 28.0, 28.3, 28.4, 29.1, 52.1, 52.2, 57.4, 57.5, 59.2, 59.4, 80.6, 127.3, 127.6, 129.2, 129.3, 130.3, 130.7, 133.3, 133.5, 140.2, 145.1, 145.6, 153.1, 153.5, 172.5, 172.9; HRMS (ESI) calcd for $C_{19}H_{25}NNaO_6S$ $[M+Na]^+$ 418.1295, found 418.1293.

***Compound* 3j**:



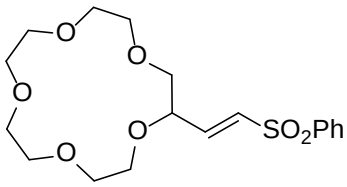
68% yield (12.4 mg); colorless oil; IR (film) 3489, 3223, 3162, 3092, 3064, 1692, 1625, 1480, 1445, 1412, 1146, 755, 701 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.95 (1H, m), 2.08 (1H, m), 2.31 (1H, m), 2.74 (1H, dd, *J* = 13.0, 6.3 Hz), 4.19 (1H, d, *J* = 8.0 Hz), 4.95 (1H, m), 6.46 (1H, dd, *J* = 15.5, 1.2 Hz), 6.97 (1H, dd, *J* = 15.5, 6.0 Hz), 6.99 (1H, d, *J* = 8.0 Hz), 7.26 (1H, t, *J* = 8.0 Hz), 7.49 (1H, t, *J* = 8.0 Hz), 7.56 (2H, t, *J* = 7.5 Hz), 7.64 (1H, t, *J* = 7.5 Hz), 7.80 (1H, d, *J* = 8.0 Hz), 7.91 (2H, d, *J* = 7.5 Hz), 8.28 (1H, br); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.4, 29.2, 56.9, 58.3, 121.0, 125.5, 127.2, 127.5, 129.3, 131.1, 131.2, 132.8, 133.5, 134.5, 140.1, 143.7, 165.0, 170.1; HRMS (ESI) calcd for $C_{20}H_{18}N_2NaO_4S$ $[M+Na]^+$ 405.0879, found 405.0868.

***Compound* 3k**:



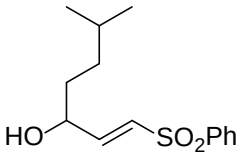
77% yield (dr = 3.8:1, 49.6 mg); colorless oil; IR (film) 3061, 1627, 1446, 1379, 1308, 1146, 1086, 753, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) major diastereomer; δ 0.81 (3H, s), 0.82 (3H, s), 0.87 (3H, s), 0.92-1.03 (2H, m), 1.12 (3H, s), 1.15-1.48 (7H, m), 1.56-1.67 (2H, m), 1.76 (1H, m), 1.92 (1H, dt, *J* = 11.9, 3.2 Hz), 2.07 (1H, ddd, *J* = 13.7, 11.8, 10.2 Hz), 4.70 (1H, m), 6.55 (1H, dd, *J* = 15.0, 1.9 Hz), 7.00 (1H, d, *J* = 15.0, 3.7 Hz), 7.51 (2H, t, *J* = 7.5 Hz), 7.59 (1H, t, *J* = 7.5 Hz), 7.87 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) major diastereomer; δ 15.1, 18.3, 20.5, 21.1, 21.7, 28.3, 33.0, 33.5, 36.1, 39.5, 39.7, 42.3, 56.9, 57.6, 73.5, 82.6, 127.7, 129.1, 129.2, 133.3, 140.5, 147.8; HRMS (ESI) calcd for $C_{24}H_{34}NaO_3S$ $[M+Na]^+$ 425.2121, found 425.2111.

***Compound* 3l**:



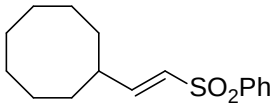
71% yield (43.4 mg); colorless oil; IR (film) 3086, 1631, 1447, 1308, 1145, 1126, 1086, 757, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.52-3.78 (18H, m), 4.40 (1H, m), 6.65 (1H, dd, *J* = 15.2, 1.7 Hz), 6.99 (1H, dd, *J* = 15.2, 4.0 Hz), 7.53 (2H, t, *J* = 7.5 Hz), 7.61 (1H, t, *J* = 7.5 Hz), 7.89 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 70.1, 70.4 (×2), 70.5, 70.6, 70.8 (×2), 71.1, 72.7, 78.1, 127.7, 129.2, 131.1, 133.3, 140.4, 144.3; HRMS (ESI) calcd for $C_{18}H_{26}NaO_7S$ $[M+Na]^+$ 409.1291, found 409.1280.

***Compound* 3m**:



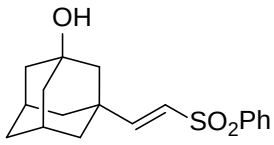
74% yield (29.1 mg); colorless solid; m.p. 83-84 °C; IR (film) 3497, 3061, 1628, 1447, 1306, 1146, 1085, 754, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.88 (3H, d, *J* = 6.8 Hz), 0.89 (3H, d, *J* = 6.8 Hz), 1.20-1.35 (2H, m), 1.49-1.70 (4H, m), 4.36 (1H, m), 6.60 (1H, dd, *J* = 15.5, 2.0 Hz), 7.00 (1H, dd, *J* = 15.5, 3.9 Hz), 7.55 (2H, t, *J* = 7.5 Hz), 7.63 (1H, t, *J* = 7.5 Hz), 7.90 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.3, 22.5, 27.9, 34.1, 34.3, 70.5, 127.6, 129.3, 129.6, 133.4, 140.3, 148.2; HRMS (ESI) calcd for $C_{14}H_{20}NaO_3S$ $[M+Na]^+$ 291.1025, found 291.1038.

***Compound* 3n**:



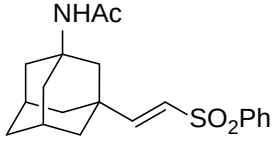
86% yield (39.9 mg); colorless oil; IR (film) 3059, 1619, 1307, 1146, 1087, 752, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.42-1.77 (14H, m), 2.43 (1H, m), 6.23 (1H, dd, *J* = 15.2, 1.3 Hz), 6.98 (1H, dd, *J* = 15.2, 7.2 Hz), 7.53 (2H, t, *J* = 7.5 Hz), 7.61 (1H, t, *J* = 7.5 Hz), 7.87 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.7, 25.7, 26.9, 30.2, 40.0, 127.4, 127.8, 129.1, 133.1, 140.8, 152.8; HRMS (ESI) calcd for $C_{16}H_{22}NaO_2S$ $[M+Na]^+$ 301.1233, found 301.1225.

***Compound* 3o**:



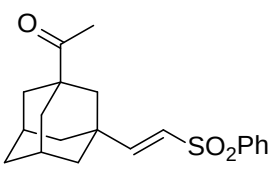
91% yield (52.1 mg); colorless oil; IR (film) 3485, 3386, 3058, 1618, 1447, 1302, 1146, 1086, 754, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.47-1.75 (13H, m), 2.27 (2H, s), 6.18 (1H, d, *J* = 15.2 Hz), 6.86 (1H, d, *J* = 15.2 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.86 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.0, 34.8, 39.36, 39.45, 44.1, 47.9, 68.1, 127.0, 127.4, 129.2, 133.2, 140.5, 153.9; HRMS (ESI) calcd for $C_{18}H_{22}NaO_3S$ $[M+Na]^+$ 341.1182, found 341.1186.

***Compound* 3p**:



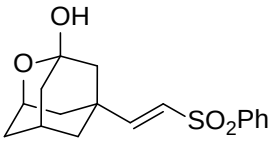
90% yield (40.7 mg); colorless oil; IR (film) 3374, 3308, 3062, 1656, 1540, 1446, 1303, 1146, 1086, 753, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.50-1.70 (6H, m), 1.81 (2H, d, *J* = 12.0 Hz), 1.90 (3H, s), 1.94 (2H, s), 2.02 (2H, d, *J* = 12.0 Hz), 2.20 (2H, s), 5.14 (1H, br s), 6.17 (1H, d, *J* = 15.2 Hz), 6.83 (1H, d, *J* = 15.2 Hz), 7.53 (2H, t, *J* = 7.5 Hz), 7.61 (1H, t, *J* = 7.5 Hz), 7.85 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.5, 28.8, 35.2, 37.8, 39.6, 40.5, 43.5, 51.9, 127.1, 127.5, 129.2, 133.2, 140.5, 154.0, 169.6; HRMS (ESI) calcd for $C_{20}H_{25}NNaO_3S$ $[M+Na]^+$ 382.1447, found 382.1437.

***Compound* 3q**:



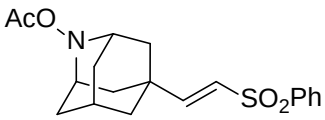
75% yield (37.0 mg); colorless oil; IR (film) 3056, 1697, 1618, 1446, 1305, 1146, 1086, 753, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.50-1.70 (10H, m), 1.84 (2H, d, *J* = 12.4 Hz), 2.10 (3H, s), 2.19 (2H, s), 6.20 (1H, d, *J* = 15.5 Hz), 6.86 (1H, d, *J* = 15.5 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.87 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.4, 27.7, 35.2, 36.3, 37.3, 39.8, 40.8, 46.6, 127.2, 127.5, 129.2, 133.2, 140.5, 154.4, 212.6; HRMS (ESI) calcd for $C_{20}H_{24}NaO_3S$ $[M+Na]^+$ 367.1338, found 367.1340.

***Compound* 3r**[2]:



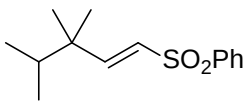
90% yield (34.9 mg); colorless oil; IR (film) 3447, 3381, 3057, 1620, 1446, 1303, 1146, 1086, 1000, 754, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.45-1.84 (9H, m), 1.92 (1H, m), 2.42, (1H, m), 2.58 (1H, br s), 4.37 (1H, m), 6.20 (1H, d, *J* = 15.0 Hz), 6.86 (1H, d, *J* = 15.0 Hz), 7.55 (2H, t, *J* = 7.5 Hz), 7.63 (1H, t, *J* = 7.5 Hz), 7.87 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.5, 33.4, 37.8, 38.0, 38.4, 40.7, 44.5, 70.8, 93.7, 127.5, 127.9, 129.3, 133.4, 140.3, 152.2; HRMS (ESI) calcd for $C_{17}H_{20}NaO_4S$ $[M+Na]^+$ 343.0975, found 343.0982.

***Compound* 3s**:



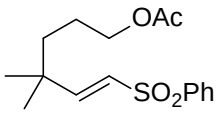
72% yield (22.0 mg); colorless oil; 2:1 mixture of rotamers; IR (film) 3057, 1754, 1618, 1446, 1304, 1215, 1146, 1086, 755, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34-1.42 (2H, m), 1.66-1.76 (4H, m), 2.05-2.20 (22/3H, m), 2.31 (2/3H, m), 3.48 (2/3H, s), 3.54 (4/3H, s), 6.18 (1/3H, d, *J* = 15.5 Hz), 6.19 (2/3H, d, *J* = 15.5 Hz), 6.82 (2/3H, d, *J* = 15.5 Hz), 6.83 (1/3H, d, *J* = 15.5 Hz), 7.51-7.58 (2H, m), 7.60-7.65 (1H, m), 7.83-7.90 (2H, m); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 19.77, 19.80, 22.6, 25.3, 25.8, 29.1, 31.5, 33.2, 34.5, 34.7, 34.9, 39.2, 39.65, 39.70, 54.6, 55.2, 127.4, 127.6, 127.8, 129.2, 129.3, 133.3, 140.4, 140.5, 153.0, 153.6, 169.21, 169.24; HRMS (ESI) calcd for $C_{19}H_{23}NNaO_4S$ $[M+Na]^+$ 384.1240, found 384.1227.

***Compound* 3t**:



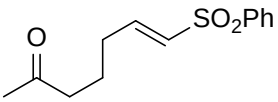
65% yield (26.7 mg); colorless oil; IR (film) 1619, 1466, 1446, 1381, 1319, 1305, 1146, 1087, 755, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.82 (6H, d, *J* = 6.9 Hz), 0.99 (6H, s), 1.62 (1H, septet, *J* = 6.9 Hz), 6.19 (1H, d, *J* = 15.5 Hz), 6.98 (1H, d, *J* = 15.5 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.61 (1H, t, *J* = 7.5 Hz), 7.87 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.6, 23.1, 36.8, 40.0, 127.4, 127.7, 129.2, 133.1, 140.9, 156.0; HRMS (ESI) calcd for $C_{14}H_{20}NaO_2S$ $[M+Na]^+$ 275.1076, found 275.1064.

***Compound* 3u**:



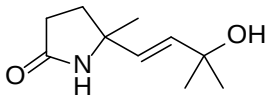
65% yield (24.2 mg); colorless oil; IR (film) 3058, 1736, 1619, 1446, 1367, 1307, 1240, 1146, 1087, 1040, 754, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.07 (6H, s), 1.25-1.55 (4H, m), 2.04 (3H, s), 4.00 (2H, t, *J* = 6.4 Hz), 6.21 (1H, d, *J* = 15.1 Hz), 6.93 (1H, d, *J* = 15.1 Hz), 7.53 (2H, t, *J* = 7.5 Hz), 7.63 (1H, t, *J* = 7.5 Hz), 7.87 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.9, 23.8, 25.9, 36.8, 38.1, 64.4, 127.5, 127.9, 129.3, 133.2, 140.7, 155.1, 171.0; HRMS (ESI) calcd for $C_{16}H_{22}NaO_4S$ $[M+Na]^+$ 333.1131, found 333.1142.

***Compound* 3v**:



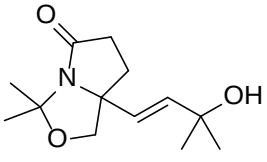
20% yield (8.6 mg); colorless oil; IR (film) 3059, 1713, 1626, 1446, 1366, 1307, 1146, 1086, 754, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.74 (2H, quintet, *J* = 7.2 Hz), 2.12 (3H, s), 2.25 (2H, tdd, *J* = 7.2, 6.8, 1.5 Hz), 2.45 (2H, t, *J* = 7.2 Hz), 6.33 (1H, dt, *J* = 14.9, 1.5 Hz), 6.95 (1H, dt, *J* = 14.9, 6.8 Hz), 7.54 (2H, t, *J* = 7.5 Hz), 7.62 (1H, t, *J* = 7.5 Hz), 7.88 (2H, d, *J* = 7.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 30.0, 30.6, 42.2, 127.6, 129.3, 131.1, 133.3, 140.5, 145.9, 207.6; HRMS (ESI) calcd for $C_{13}H_{16}NaO_3S$ $[M+Na]^+$ 275.0712, found 275.0710.

***Compound* 4a**:



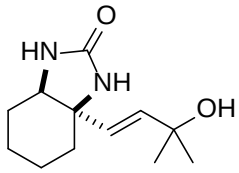
65% yield (11.2 mg); colorless oil; IR (film) 3252, 1690, 1376, 1154 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (6H, s), 1.38 (3H, s), 1.66 (1H, br), 1.94-2.08 (2H, m), 2.31-2.45 (2H, m), 5.53 (1H, br s), 5.71 (1H, d, *J* = 16.0 Hz), 5.72 (1H, d, *J* = 16.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.5, 30.0 (×3), 35.2, 59.3, 70.4, 131.5, 135.7, 177.5; HRMS (ESI) calcd for $C_{10}H_{17}NNaO_2$ $[M+Na]^+$ 206.1151, found 206.1144.

***Compound* 4b**:



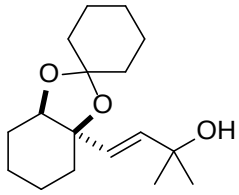
65% yield (9.1 mg); colorless oil; IR (film) 3416, 1683, 1379, 1255, 1049 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, s), 1.35 (3H, s), 1.52 (3H, s), 1.65 (3H, s), 1.95-2.08 (2H, m), 2.45 (1H, ddd, *J* = 11.8, 7.5, 1.4 Hz), 2.80 (1H, ddd, *J* = 16.5, 11.8, 8.5 Hz), 3.72 (1H, d, *J* = 8.7 Hz), 4.00 (1H, d, *J* = 8.7 Hz), 5.77 (1H, d, *J* = 15.6 Hz), 5.89 (1H, d, *J* = 15.6 Hz), O–H missing; ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.6, 27.0, 29.9, 30.0, 32.1, 35.6, 70.6, 71.8, 74.3, 92.5, 127.3, 138.1, 173.0; HRMS (ESI) calcd for $C_{13}H_{21}NNaO_3$ $[M+Na]^+$ 262.1414, found 262.1403.

***Compound* 4c**:



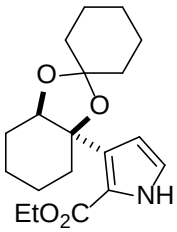
79% yield (8.2 mg); colorless solid; m.p. 130-131 °C; IR (film) 3251, 1697, 1374, 1153 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30-1.80 (15H, m), 3.51 (1H, m), 4.38 (2H, br s), 5.78 (1H, d, *J* = 16.0 Hz), 5.89 (1H, d, *J* = 16.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.5, 20.6, 27.0, 29.9, 30.0, 33.5, 57.8, 59.6, 70.6, 128.4, 137.9, 163.1; HRMS (ESI) calcd for $C_{12}H_{20}N_2NaO_2$ $[M+Na]^+$ 247.1417, found 247.1411.

***Compound* 4d**:



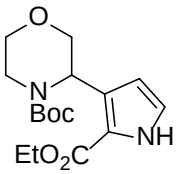
74% yield (9.0 mg); colorless oil; IR (film) 3434, 1448, 1364, 1278, 1116 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.20-1.81 (24H, m), 2.10 (1H, m), 3.86 (1H, t, *J* = 2.5 Hz), 5.76 (1H, d, *J* = 16.0 Hz), 5.98 (1H, d, *J* = 16.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.5, 22.6, 23.7, 24.0, 25.1, 26.2, 29.9 (×2), 34.4, 35.9, 38.1, 70.7, 77.8, 79.4, 108.0, 127.3, 137.9; HRMS (ESI) calcd for $C_{17}H_{28}NaO_3$ $[M+Na]^+$ 303.1931, found 303.1937.

***Compound* 5d**:



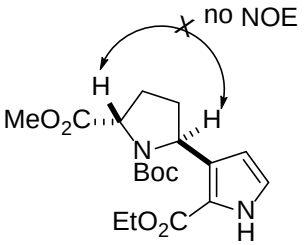
Following the general procedure except for the use of *t*-BuOK (5 equiv) and ethyl isocyanoacetate (5.5 equiv) and silica gel for purification. The reaction was conducted using vinyl sulfone **3d** (0.026 mmol, 9.5 mg). 63% yield (5.5 mg); colorless viscous oil; IR (ATR) 3355, 2934, 2861, 1709, 1465, 1447, 1411, 1366, 1308, 1267, 1200, 1165, 1139, 1119, 1091, 1071, 1023, 972, 784 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.30-1.44 (3H, m), 1.36 (3H, t, *J* = 7.1 Hz), 1.44-1.59 (5H, m), 1.59-1.77 (4H, m), 1.77-1.94 (4H, m), 2.07 (1H, m), 2.16 (1H, m), 4.32 (2H, q, *J* = 7.1 Hz), 4.64 (1H, d, *J* = 4.4 Hz), 6.44 (1H, t, *J* = 2.8 Hz), 6.79 (1H, t, *J* = 2.8 Hz), 9.05 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 17.7, 18.9, 24.0, 24.2, 25.3, 27.2, 34.7, 36.6, 37.8, 60.2, 78.2, 80.6, 108.0, 111.1, 117.4, 120.0, 138.5, 160.3; HRMS (APCI) calcd for $C_{19}H_{28}NO_4$ $[M+H]^+$ 334.2013, found 334.2000.

***Compound* 5h**:



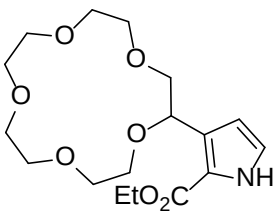
69% yield (21.5 mg); colorless oil; IR (ATR) 3309, 2978, 2930, 2857, 1693, 1555, 1476, 1454, 1415, 1367, 1352, 1326, 1280, 1254, 1232, 1171, 1122, 1081, 1041, 873, 757 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, t, *J* = 7.1 Hz), 1.37 (9H, s), 3.49 (1H, ddd, *J* = 4.1, 12.0, 12.4 Hz), 3.56 (1H, ddd, *J* = 2.8, 12.0, 12.4 Hz), 3.82 (1H, dd, *J* = 4.1, 11.8 Hz), 3.86 (1H, br d, *J* = 10.6 Hz), 3.95 (1H, br dd, *J* = 2.8, 10.6 Hz), 4.16 (1H, br d, *J* = 11.8 Hz), 4.22-4.38 (2H, m), 5.51 (1H, br d, *J* = 3.6 Hz), 6.25 (1H, t, *J* = 2.9 Hz), 6.82 (1H, t, *J* = 2.9 Hz), 9.15 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 28.2, 41.1, 48.9, 60.2, 66.7, 71.5, 79.9, 110.3, 118.3, 121.5, 132.3, 155.4, 160.6; HRMS (APCI) calcd for $C_{16}H_{25}N_2O_5$ $[M+H]^+$ 325.1758, found 325.1744.

***Compound* 5i**:



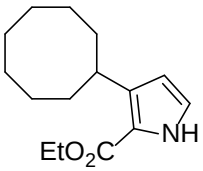
Following the general procedure except for the use of *t*-BuOK (5 equiv) and ethyl isocyanoacetate (5.5 equiv). The reaction was conducted using vinyl sulfone **3i** (0.025 mmol, 10.0 mg). 83% yield (7.6 mg); colorless oil; 1.3:1 mixture of rotamers; IR (ATR) 3323, 2978, 2958, 2931, 1746, 1692, 1478, 1391, 1367, 1317, 1255, 1200, 1163, 1124, 1077, 1062, 1049, 993, 908, 894, 780, 752 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.25 (36/9H, s), 1.35 (15/9H, t, *J* = 7.0 Hz), 1.37 (12/9H, t, *J* = 7.0 Hz), 1.41 (45/9H, s), 1.80 (1H, dd, *J* = 6.4, 12.2 Hz), 1.93 (1H, td, *J* = 7.2, 12.2 Hz), 2.27 (1H, m), 2.42 (1H, m), 3.75 (15/9H, s), 3.76 (12/9H, s), 4.22-4.42 (2H, m), 4.47 (5/9H, dd, *J* = 0.7, 9.1 Hz), 4.57 (4/9H, dd, *J* = 1.1, 9.1 Hz), 5.57 (4/9H, d, *J* = 8.0 Hz), 5.63 (5/9H, d, *J* = 8.0 Hz), 6.02 (1H, dt, *J* = 2.9, 5.6 Hz), 6.81 (1H, dd, *J* = 2.9, 6.7 Hz), 9.01 (4/9H, br s), 9.03 (5/9H. br s); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ14.4, 14.5, 27.6, 28.1, 28.28, 28.33, 32.6, 33.2, 52.0, 52.1, 55.5, 55.9, 59.4, 60.0, 60.2, 79.7, 80.0, 108.9, 109.1, 117.5, 117.6, 121.3, 121.4, 134.7, 135.8, 153.4, 154.4, 161.06, 161.12, 173.4, 173.7; HRMS (APCI) calcd for $C_{18}H_{27}N_2O_6$ $[M+H]^+$ 367.1864, found 367.1875.

***Compound* 5l**:



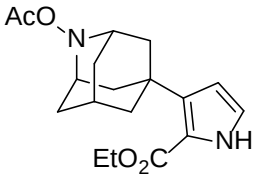
The reaction was conducted using vinyl sulfone **3l** (0.10 mmol, 39.0 mg). 31% yield (10.9 mg); brown oil; IR (ATR) 3308, 2925, 2873, 1700, 1419, 1320, 1263, 1127, 789 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.37 (3H, br t, *J* = 6.6 Hz), 3.52-3.89 (18H, m), 4.34 (2H, br q, *J* = 6.6 Hz), 5.34 (1H, br d, *J* = 7.0 Hz), 6.38 (1H, br s), 6.89 (1H, br s), 9.13 (1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 60.3, 68.2, 69.0-70.5 (broad peak was observed), 74.2, 74.5, 109.9, 119.3, 122.2, 148.4, 160.8; HRMS (APCI) calcd for $C_{17}H_{26}NO_7$ $[M-H]^-$ 356.1711, found 356.1715.

***Compound* 5n**:



Following the general procedure except for the use of *t*-BuOK (5 equiv) and ethyl isocyanoacetate (5.5 equiv). The reaction was conducted using vinyl sulfone **3n** (0.10 mmol, 27.8 mg). 72% yield (18.0 mg); pale yellow solid; mp 58-60 °C; IR (ATR) 3302, 2978, 2958, 2911, 2856, 2840, 1669, 1476, 1445, 1415, 1390, 1321, 1267, 1214, 1130, 1029, 902, 860, 779, 751 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.37 (3H, t, *J* = 7.1 Hz), 1.54-1.90 (14H, m), 3.55 (1H, m), 4.32 (2H, q, *J* = 7.1 Hz), 6.17 (1H, t, *J* = 2.8 Hz), 6.82 (1H, t, *J* = 2.8 Hz), 8.91(1H, br s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.5, 25.9, 26.7, 27.0, 34.1, 34.6, 59.9, 109.1, 117.3, 121.5, 141.0, 161.5; HRMS (APCI) calcd for $C_{15}H_{24}NO_2$ $[M+H]^+$ 250.1802, found 250.1792.

***Compound* 5s**:

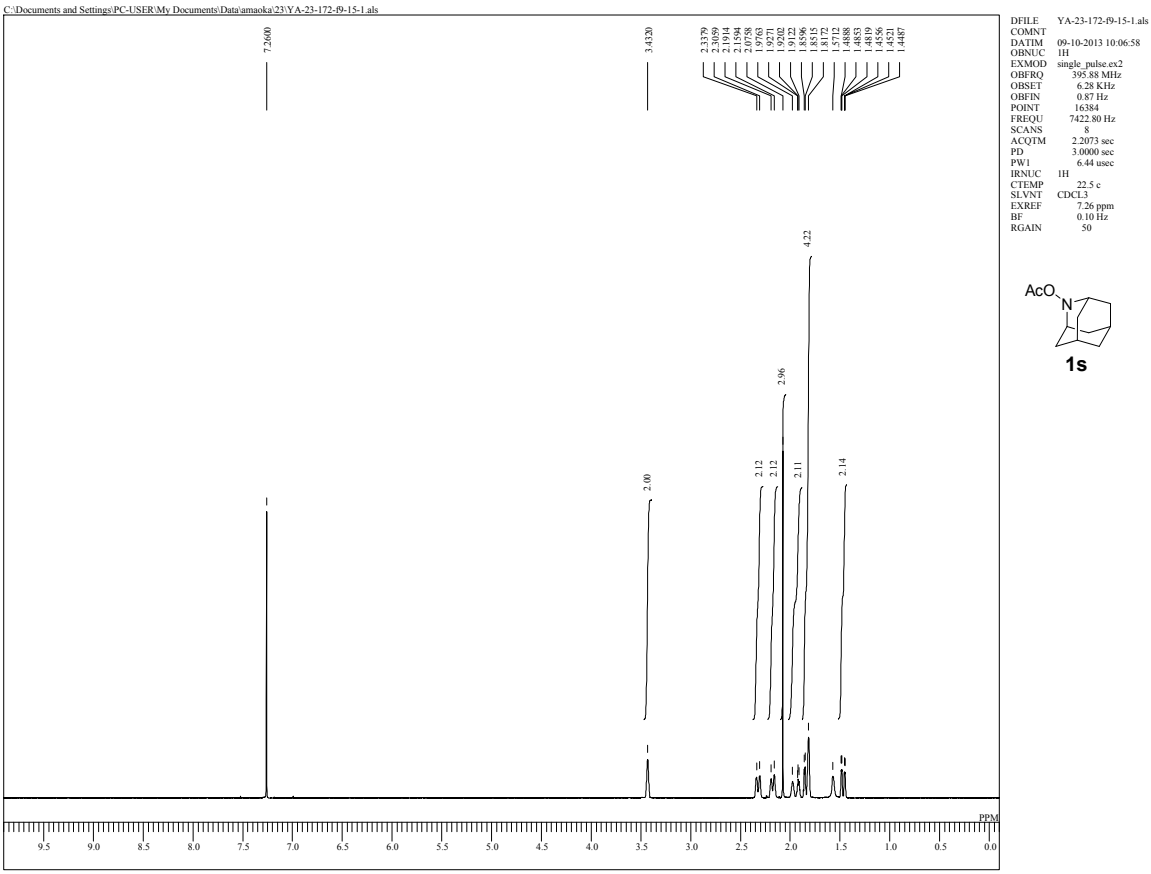


Following the general procedure except for the use of *t*-BuOK (5 equiv) and ethyl isocyanoacetate (5.5 equiv) and silica gel for purification. The reaction was conducted using vinyl sulfone **3s** (0.029 mmol, 10.5 mg). 31% yield (3.0 mg); colorless viscous oil; 10:7 mixture of rotamers; IR (ATR) 3333, 2931, 2856, 1741, 1704, 1407, 1365, 1256, 1217, 1142, 1083, 1031, 1000, 781 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.38 (3H, t, *J* = 7.2 Hz), 1.56 (14/17H, br d, *J* = 10.9 Hz), 1.90 (20/17H, br d, *J* = 10.9 Hz), 2.00-2.24 (6H, br m), 2.07 (30/17H, s), 2.10 (21/17H, s), 2.27-2.48 (3H, br m), 3.57 (14/17H, br s), 3.60 (20/17H, br s), 4.25-4.36 (2H, m), 6.15 (7/17H, t, *J* = 2.8 Hz), 6.20 (10/17H, t, *J* = 2.8 Hz), 6.83 (7/17H, t, *J* = 2.8 Hz), 6.83 (10/17H, t, *J* = 2.8 Hz), 9.15 (1H, br); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.57, 14.59, 19.9, 20.0, 26.4, 26.8, 29.5, 29.7, 32.3, 34.1, 35.3, 39.8, 40.0, 56.0, 56.7, 60.1, 60.2, 109.0, 109.3, 118.0, 120.46, 120.51, 140.7, 141.7, 160.1, 160.2, 169.6, 169.7; HRMS (APCI-TOF/MS) calcd for $C_{18}H_{25}N_2O_4$ $[M+H]^+$ 333.1809, found 333.1816.

References

(1) E. Dominguez and J. C. Carretero, *Tetrahedron*, 1990, **46**, 7197.

(2) For the preparation of **1r**, see: H. Stetter, P. Tacke and J. Gärtner, *Chem. Ber.*, 1964, **97**, 3480.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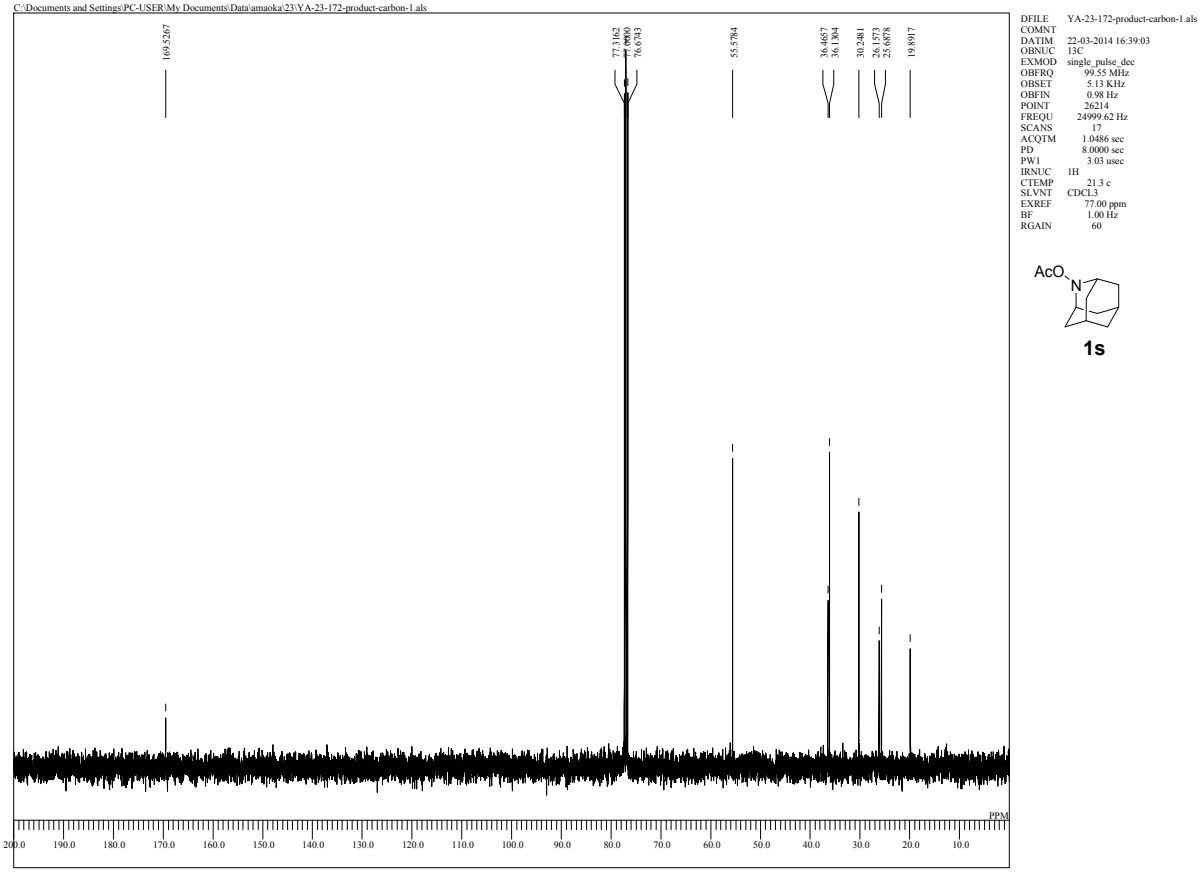
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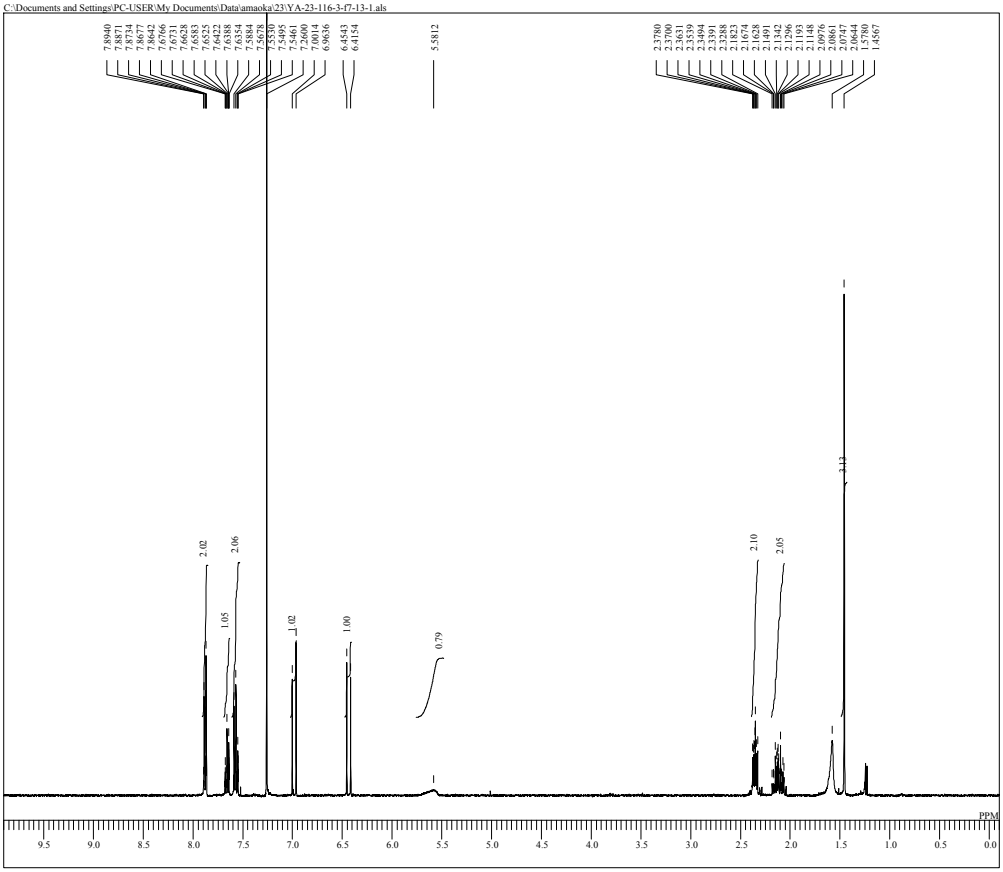
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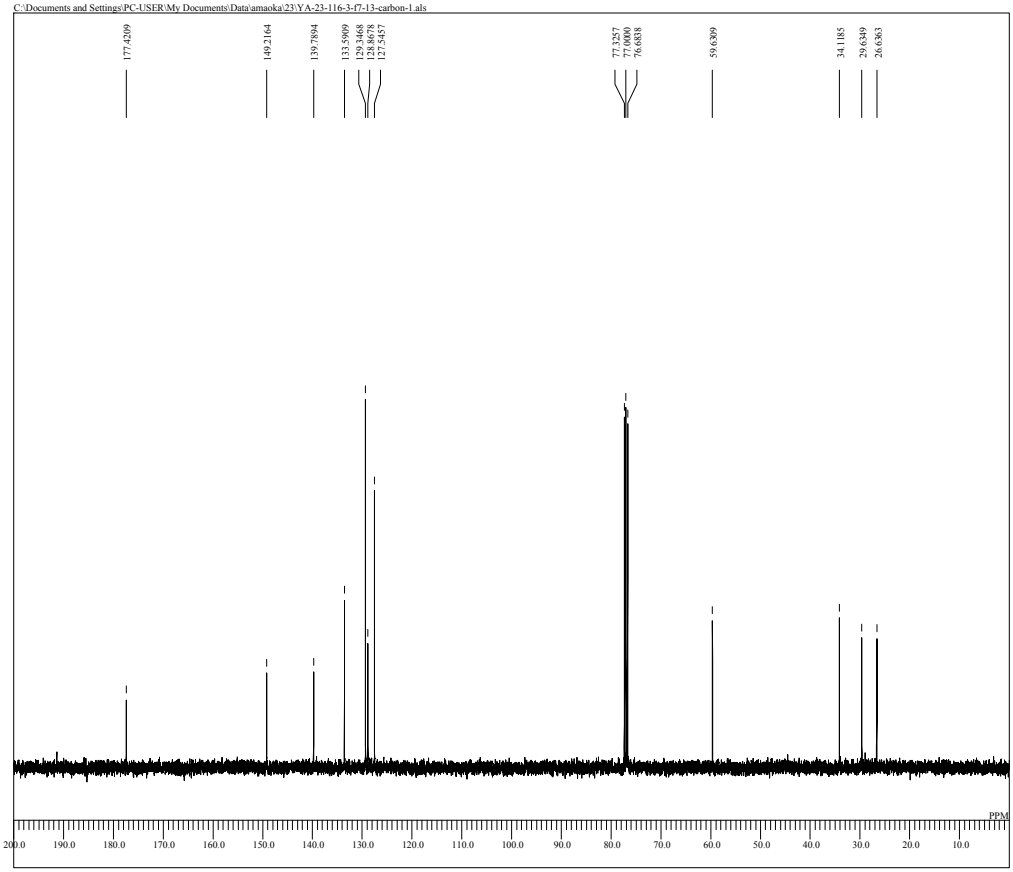
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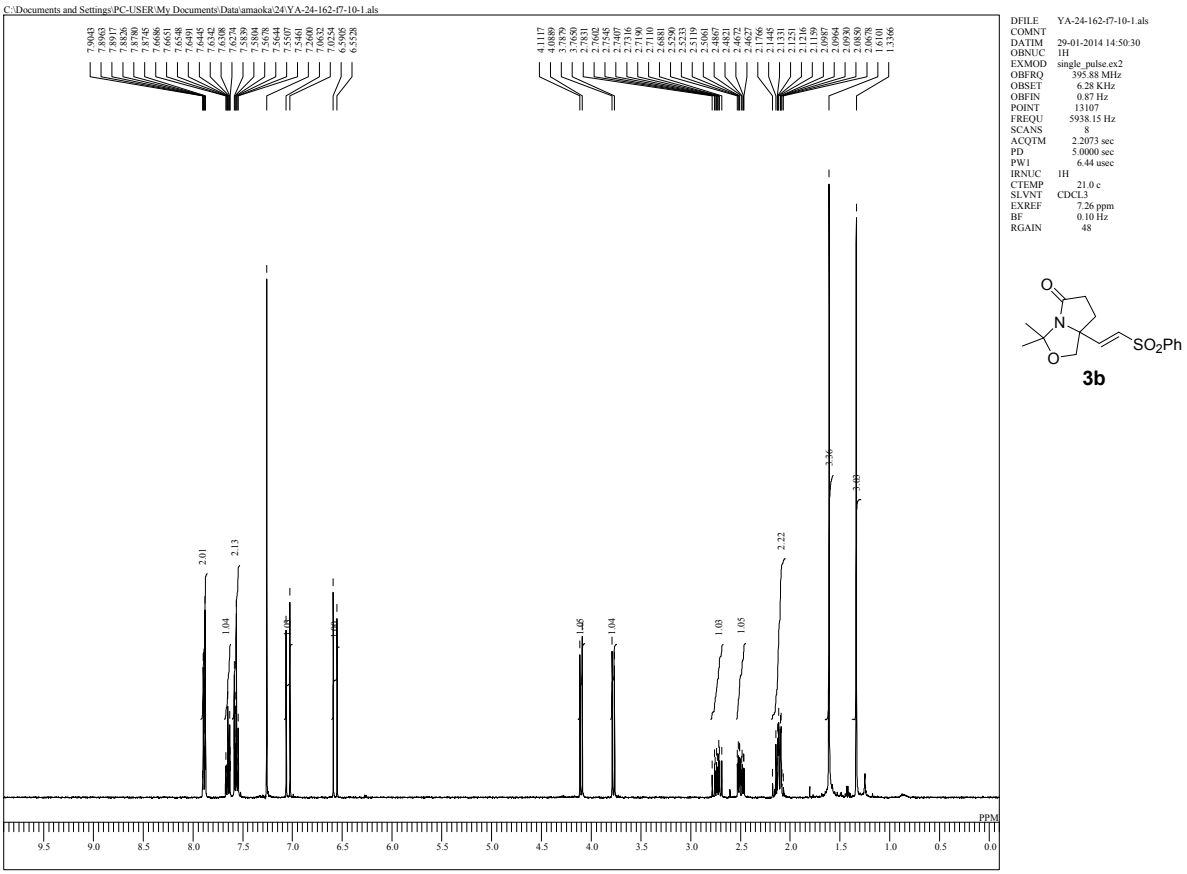
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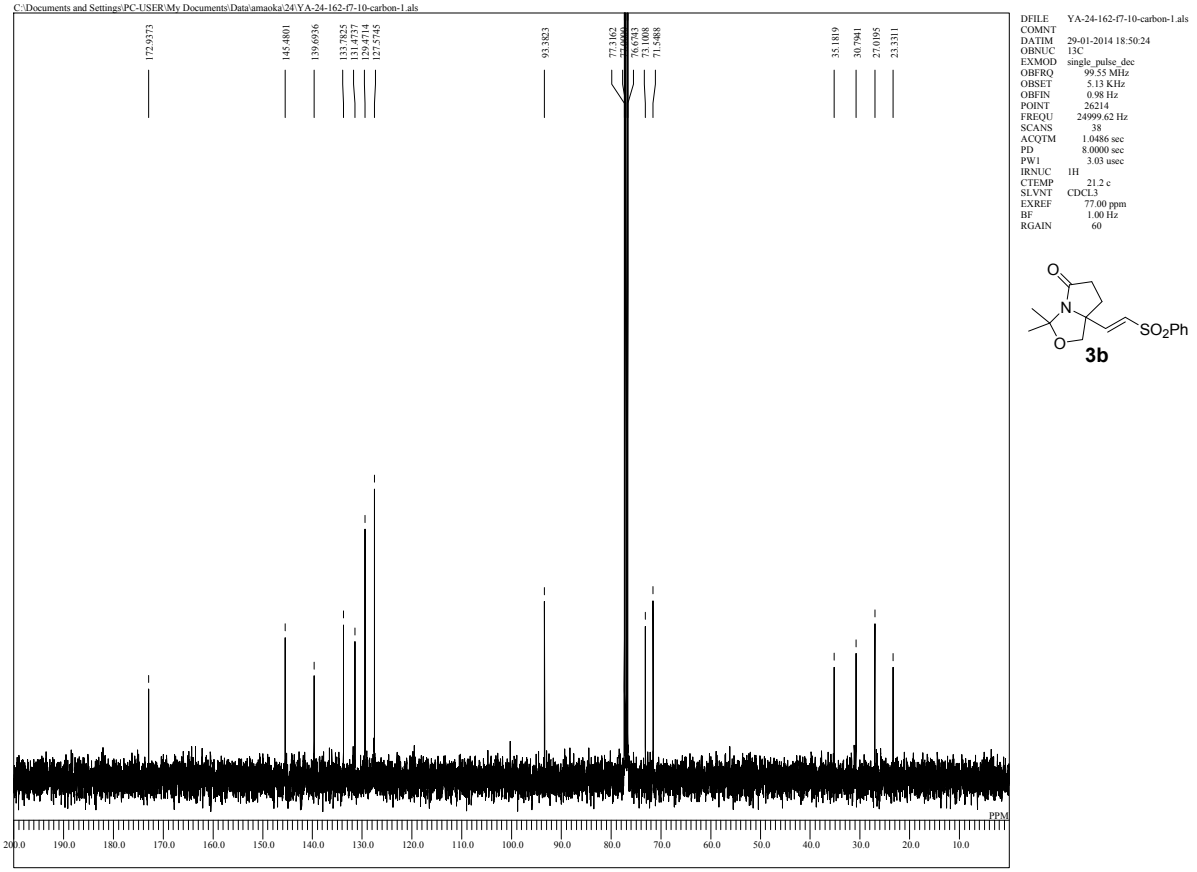
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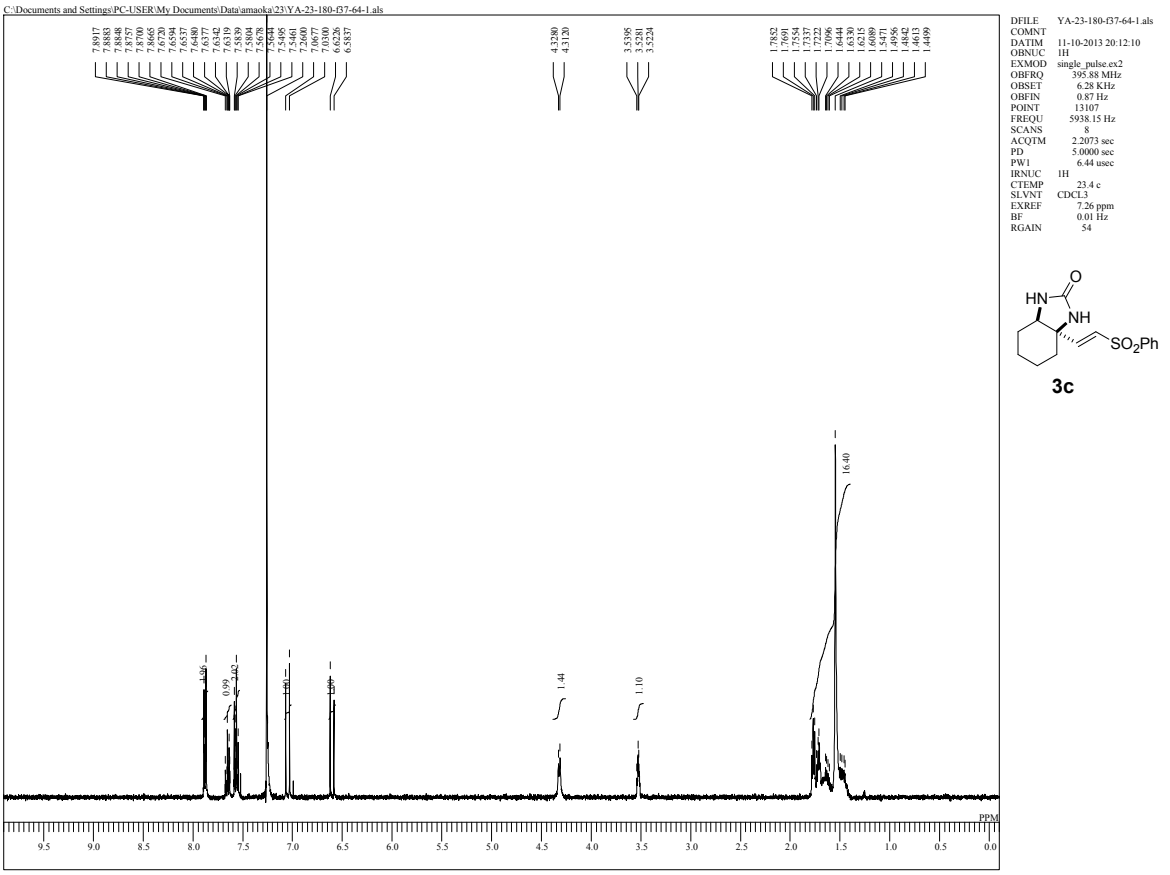
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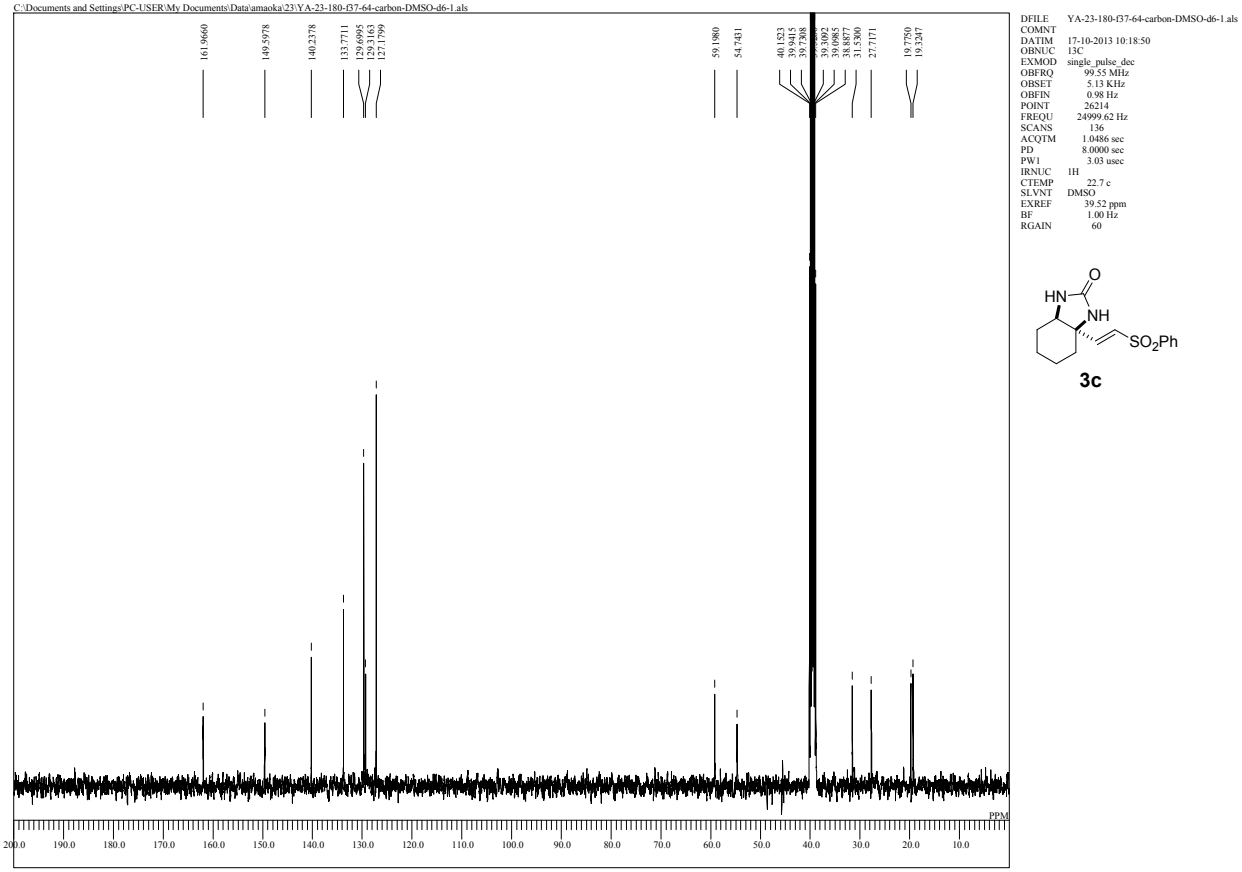
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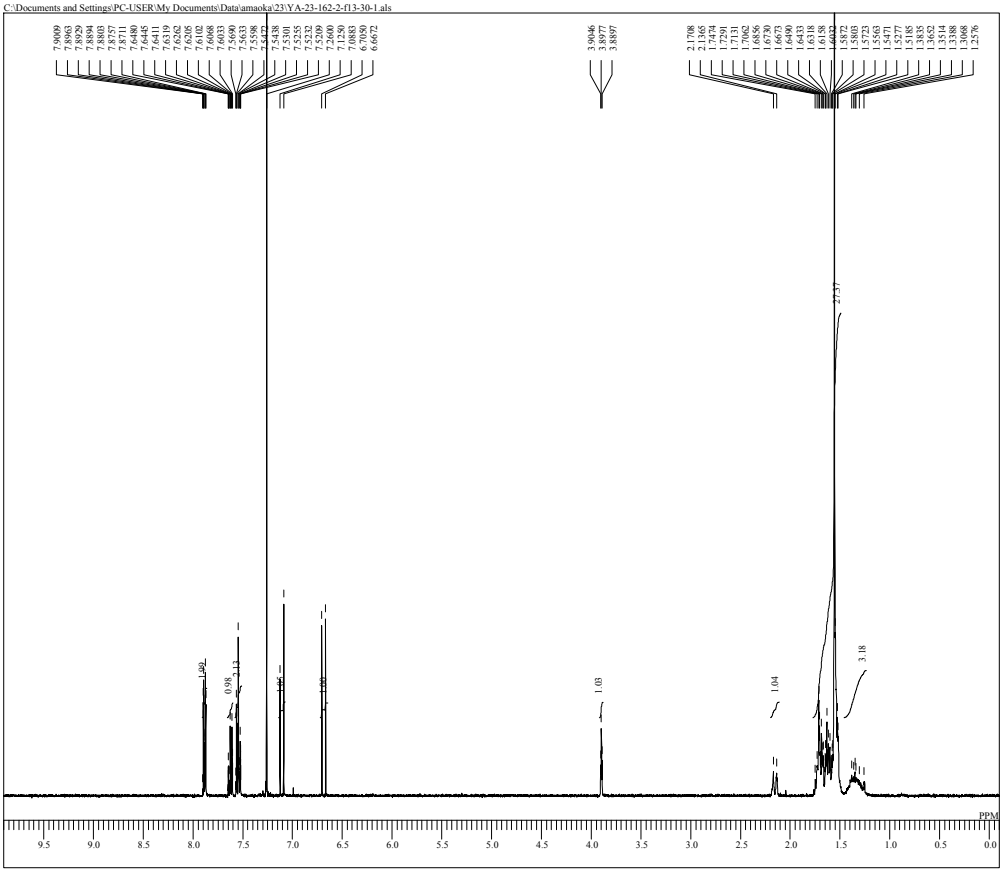
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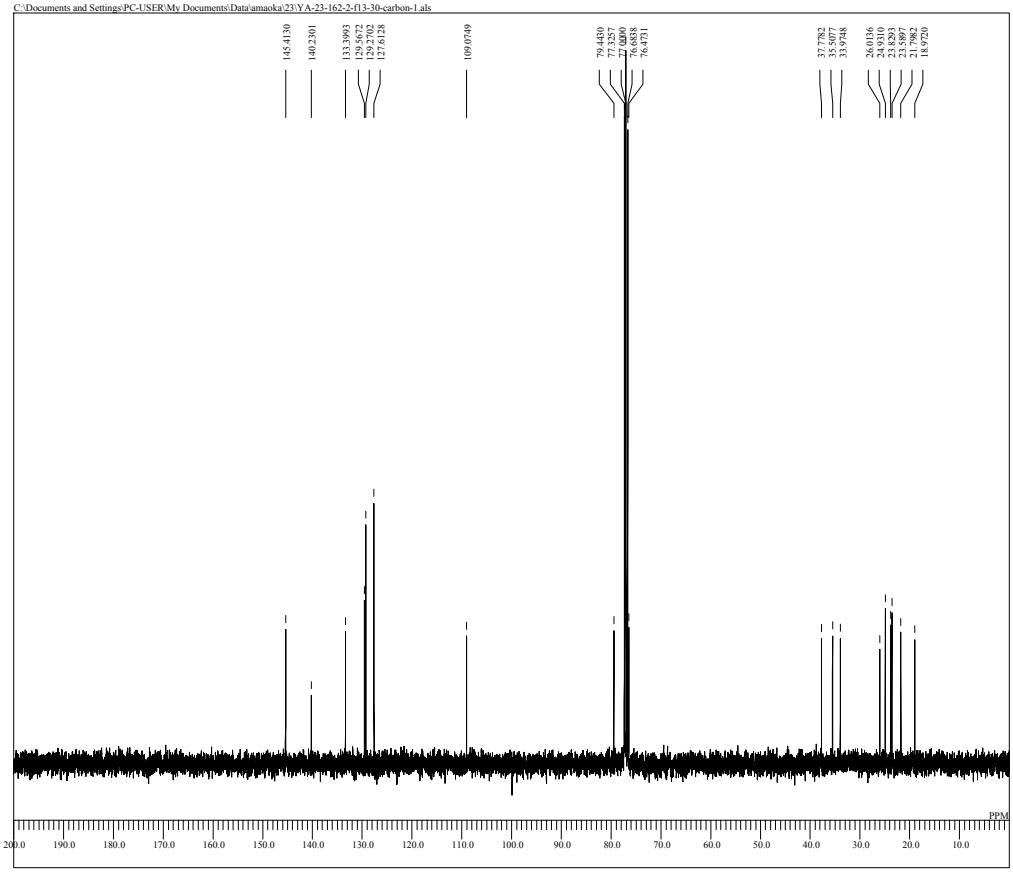
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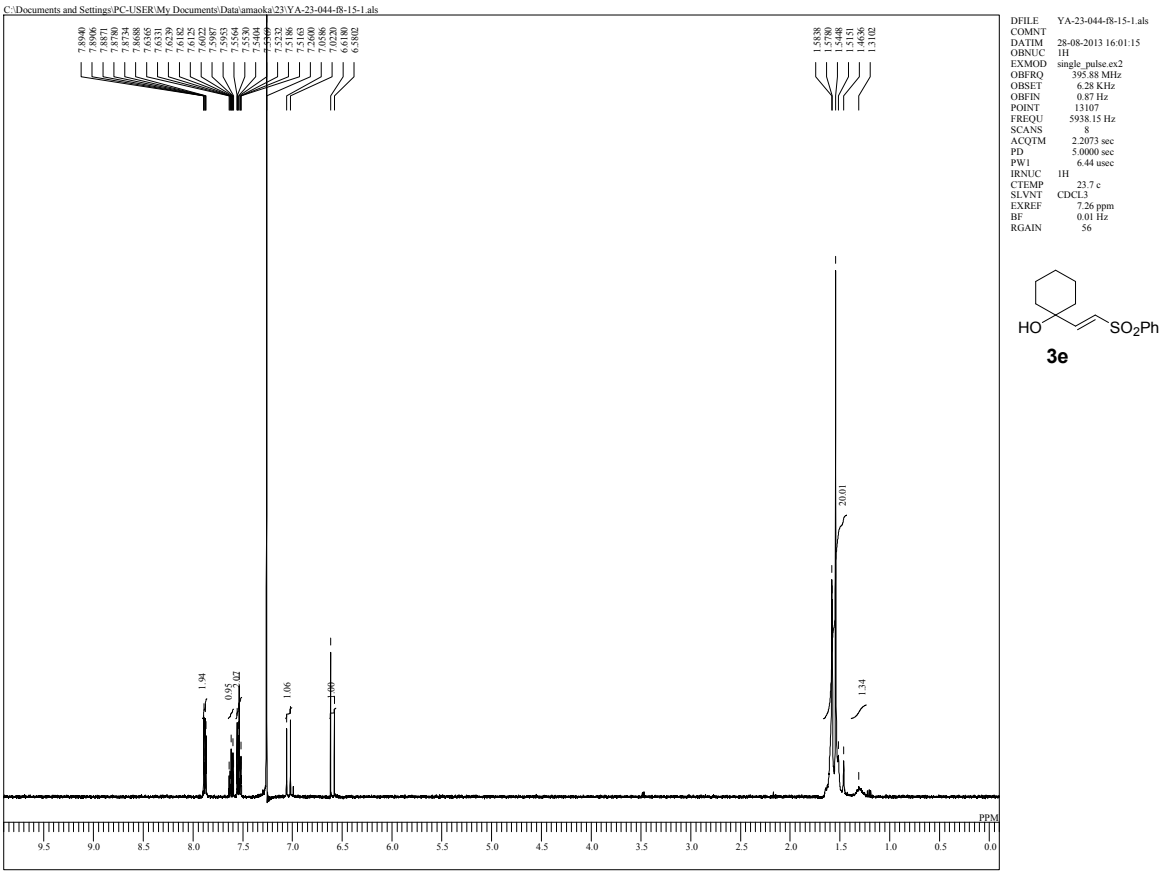

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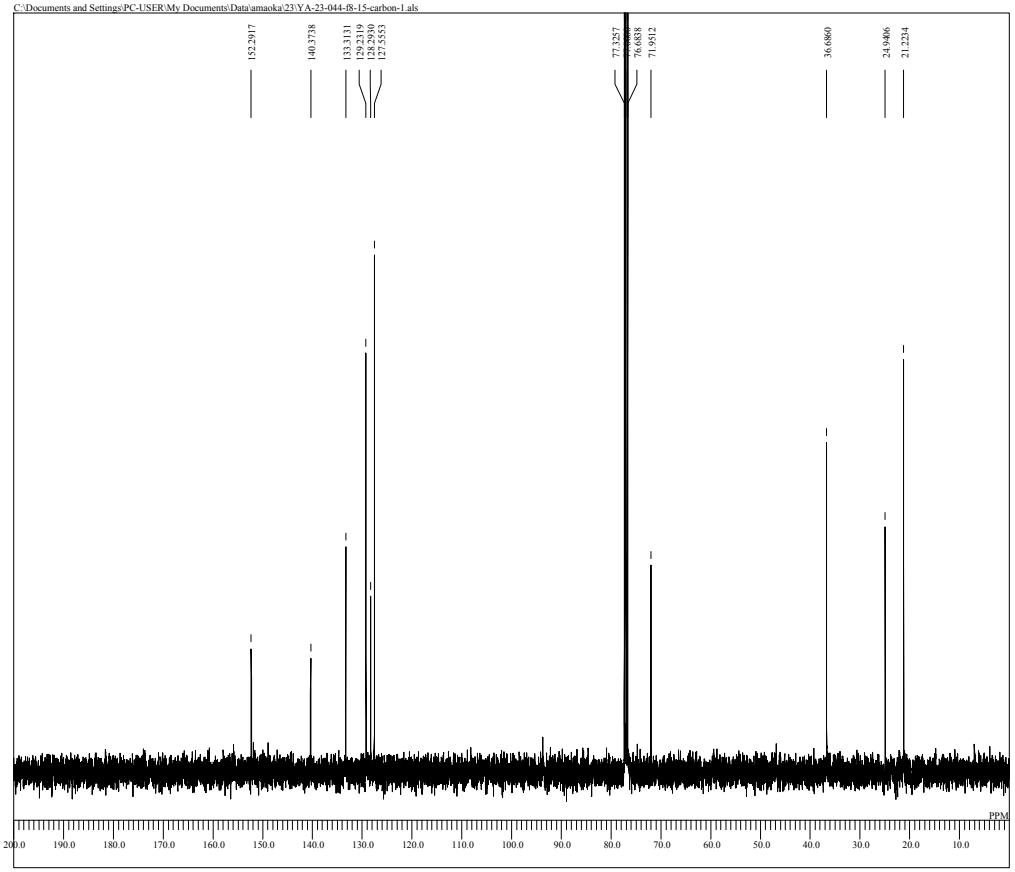

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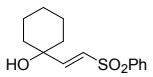
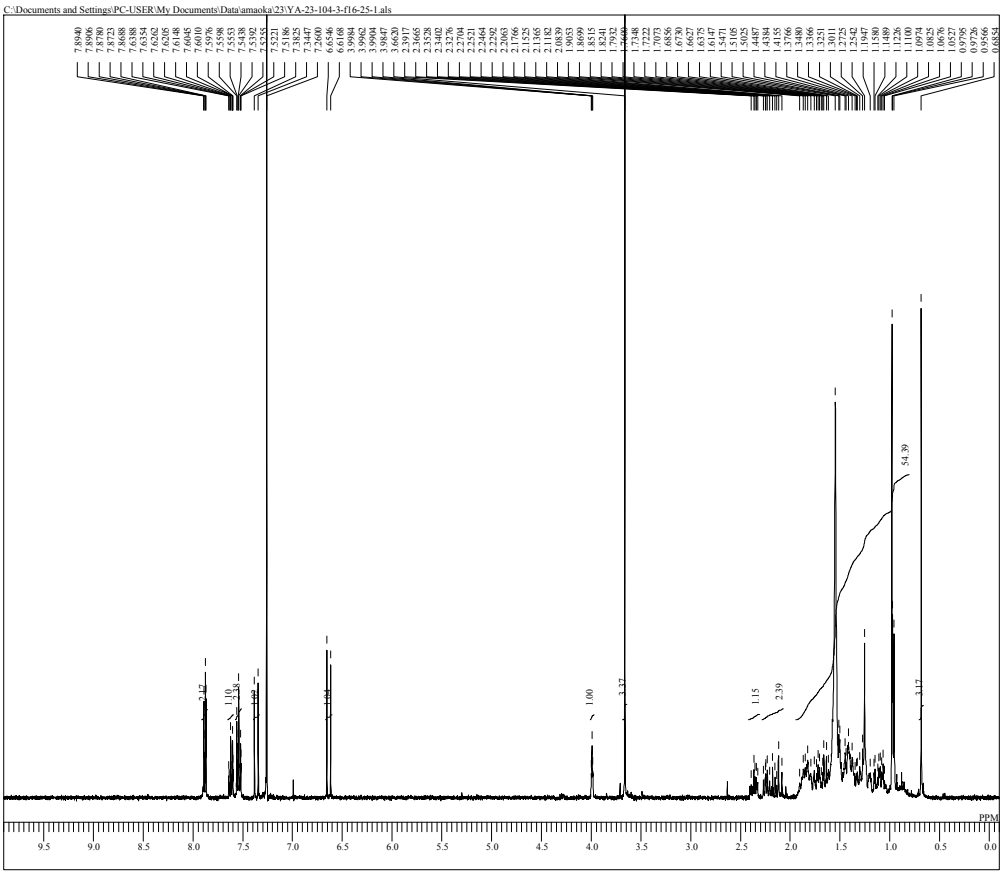
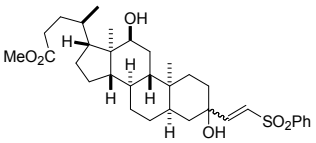
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 17
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 24.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60

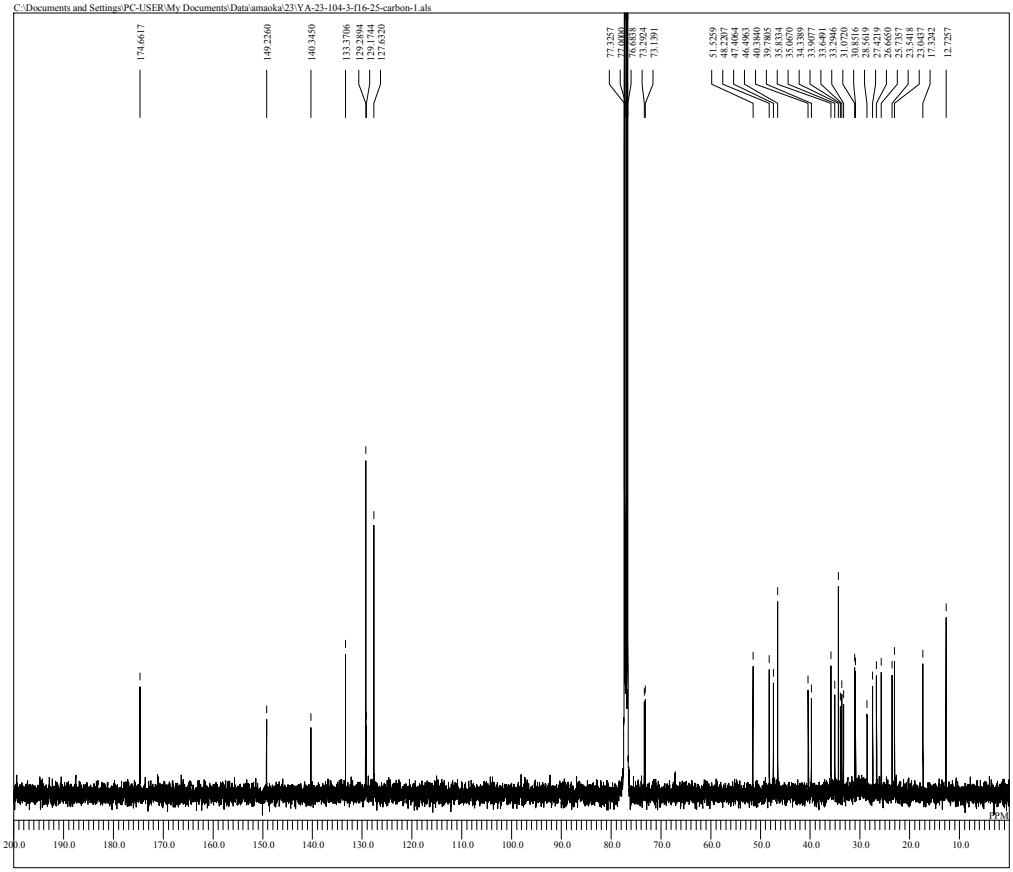


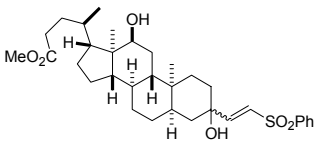
3e



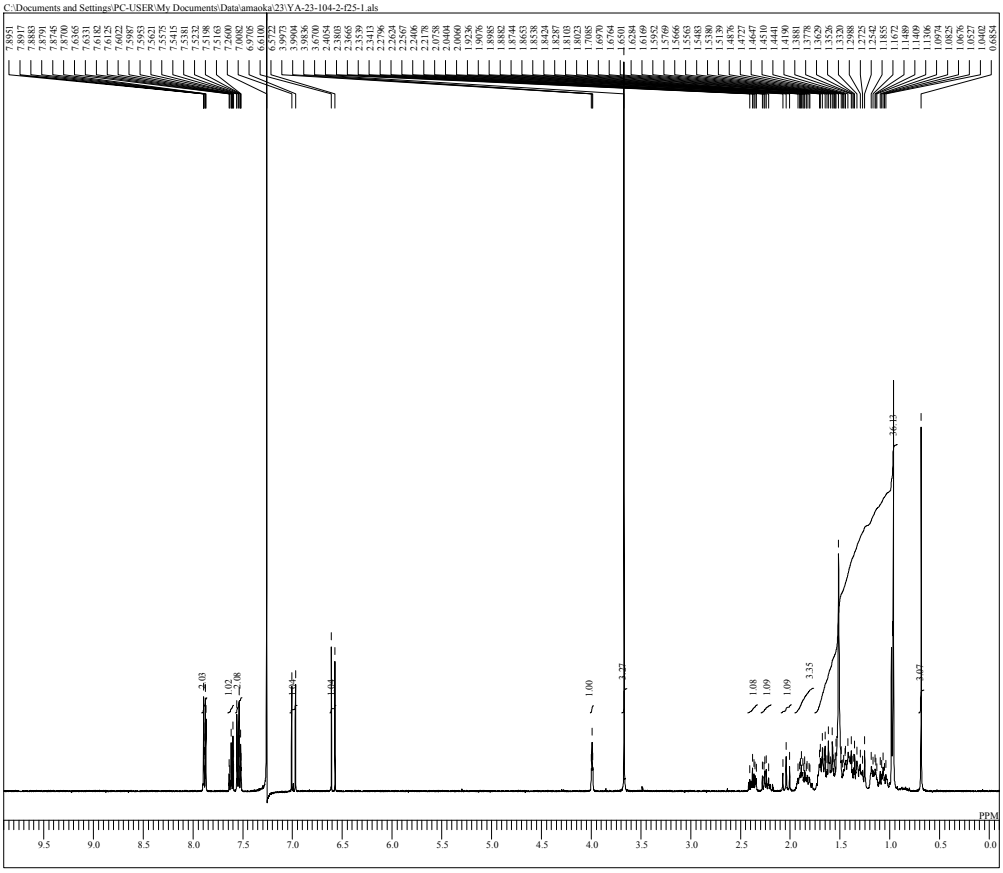


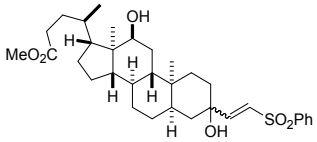
3f (major isomer)



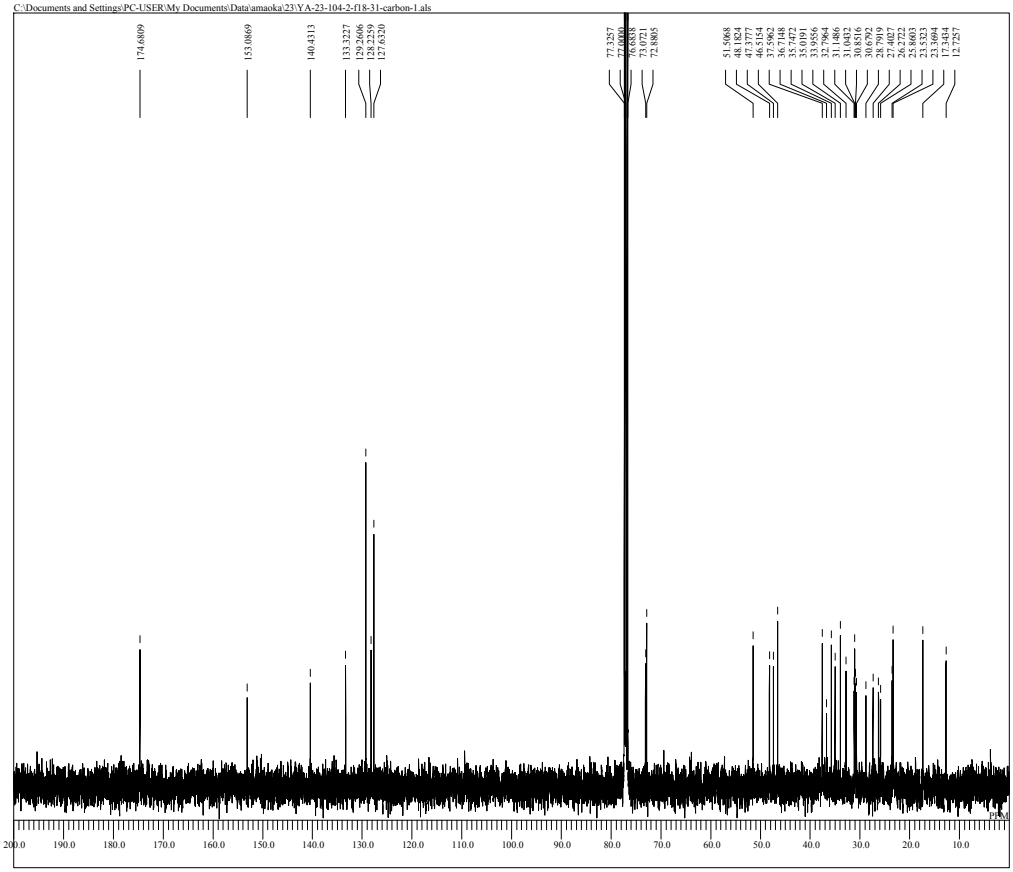


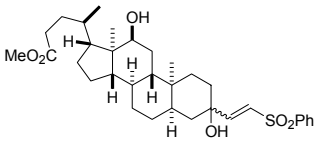
3f (major isomer)



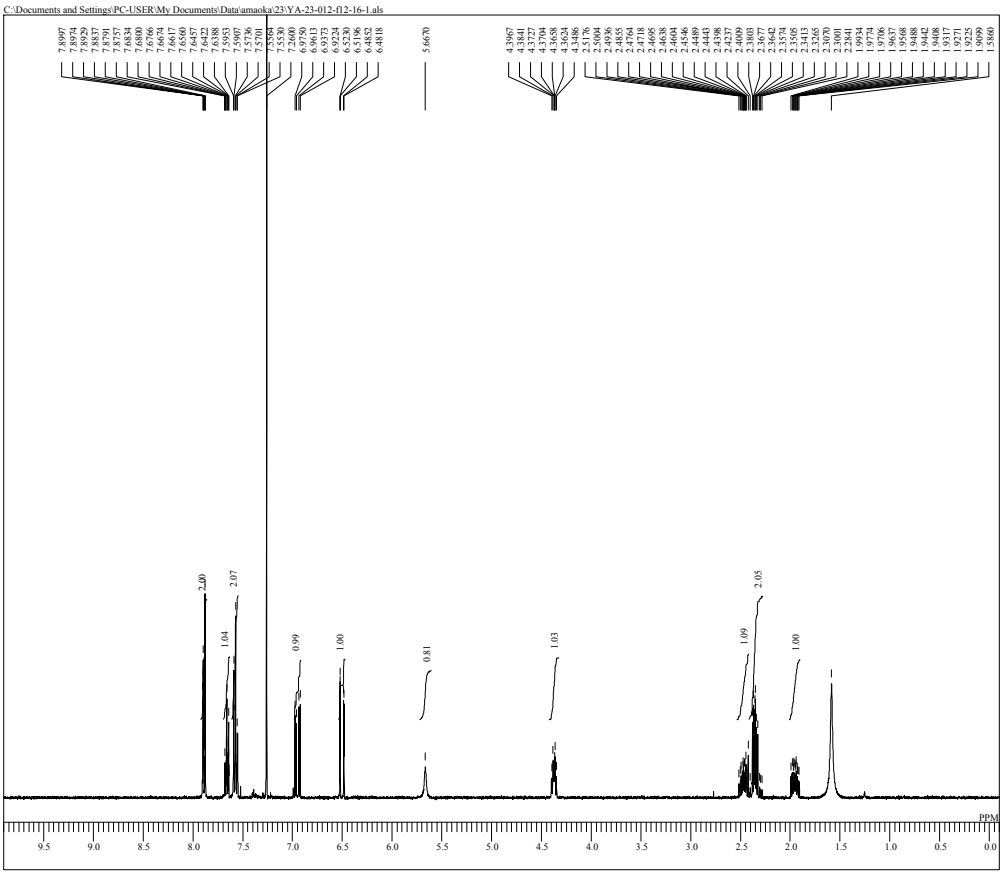


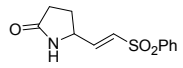
3f (minor isomer)



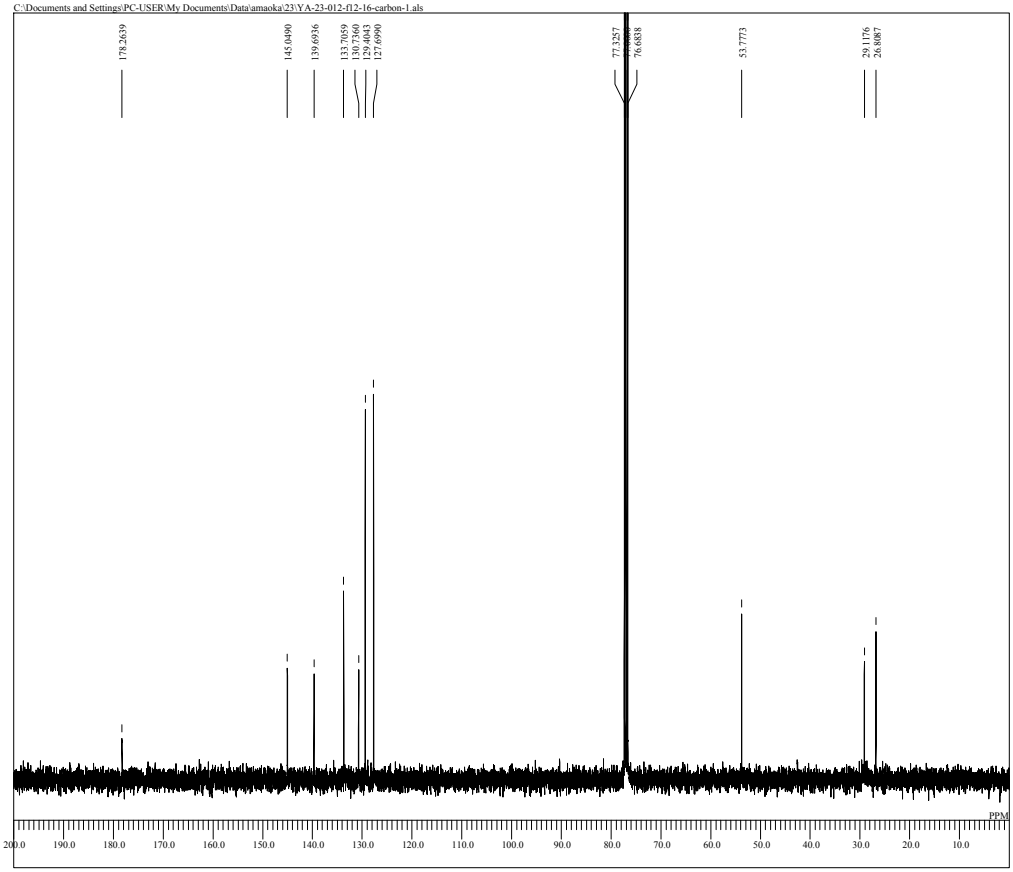


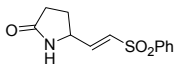
3f (minor isomer)


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-012-f12-16-1.als
DFILE YA-23-012-f12-16-1.als
COMNT
DATIM 29-07-2013 12:38:01
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 52
2.00
2.07
1.04
0.99
1.00
0.81
1.03
1.09
2.05
1.00
PPM

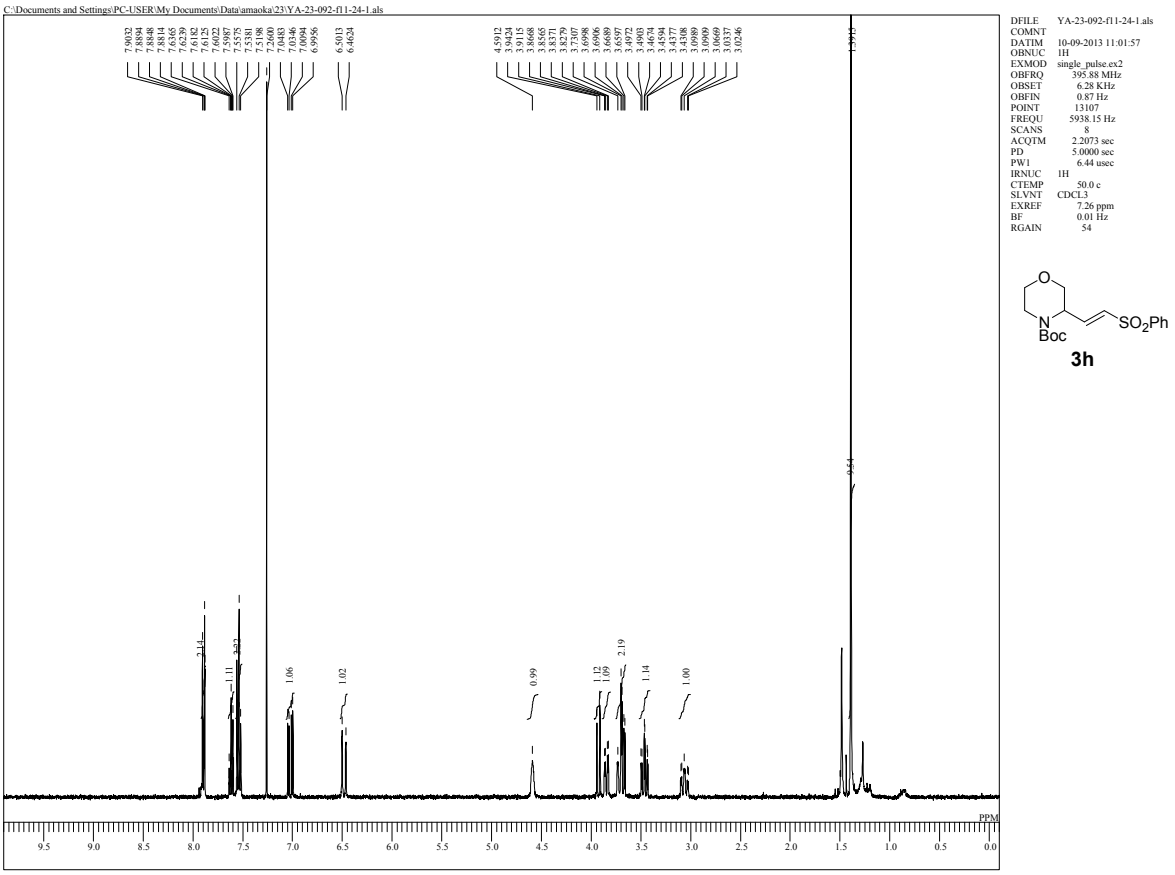

O
N
H
SO2Ph

3g


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-012-f12-16-carbon-1.als
DFILE YA-23-012-f12-16-carbon-1.als
COMNT
DATIM 29-07-2013 12:49:39
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 50
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 2.90 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
178.2639
145.0490
139.6936
133.7059
130.7360
129.0643
127.6990
77.3257
77.0000
76.6838
53.7773
29.1176
26.8087
PPM

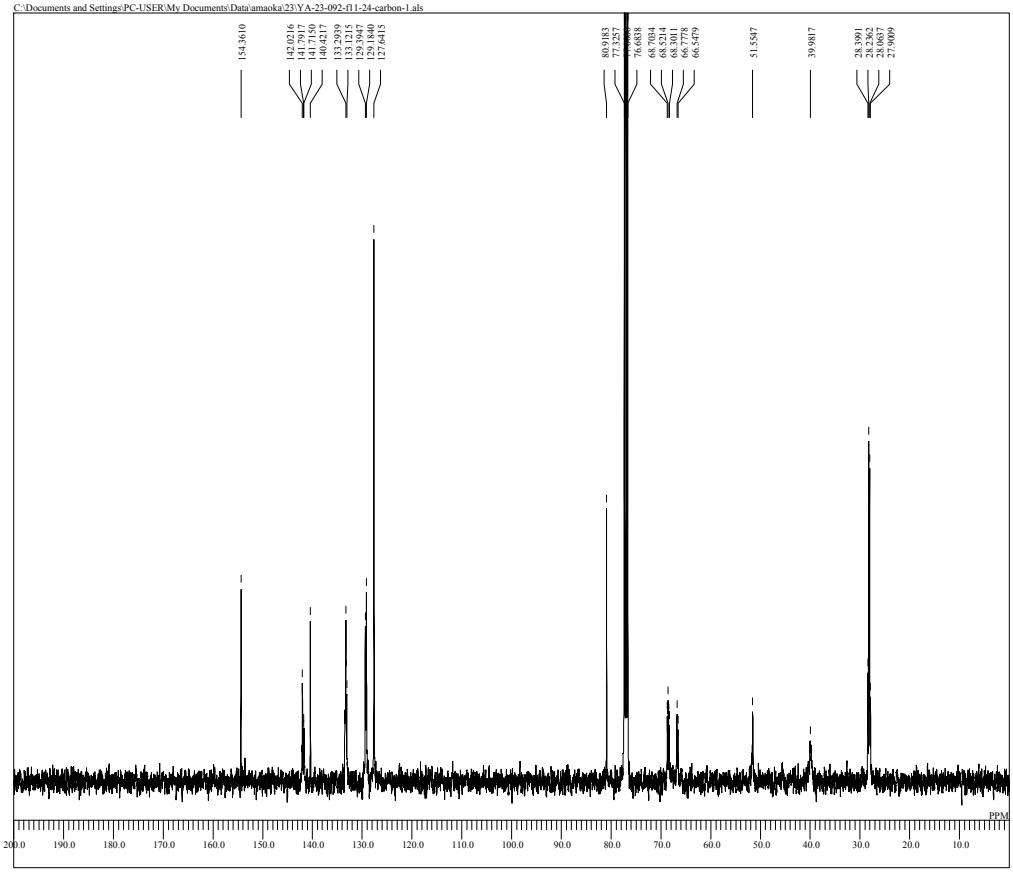

O
N
H
SO2Ph

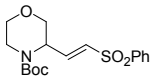
3g


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-092-f11-24-1.als
DFILE YA-23-092-f11-24-1.als
COMNT
DATIM 10-09-2013 11:01:57
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 50.0 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 54
7.9032
7.8894
7.8848
7.8614
7.6345
7.6239
7.6182
7.6125
7.6027
7.5987
7.5575
7.5381
7.5198
7.2600
7.0483
7.0346
7.0094
6.9936
6.5013
6.4624
4.5912
3.9424
3.9115
3.8668
3.8585
3.8371
3.8279
3.7307
3.6998
3.6896
3.6689
3.6597
3.4972
3.4903
3.4654
3.4594
3.4377
3.4308
3.0989
3.0669
3.0337
3.0246
1.3919
9.44
2.14
1.11
3.22
1.06
1.02
0.99
1.12
1.08
2.19
1.14
1.00
PPM
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

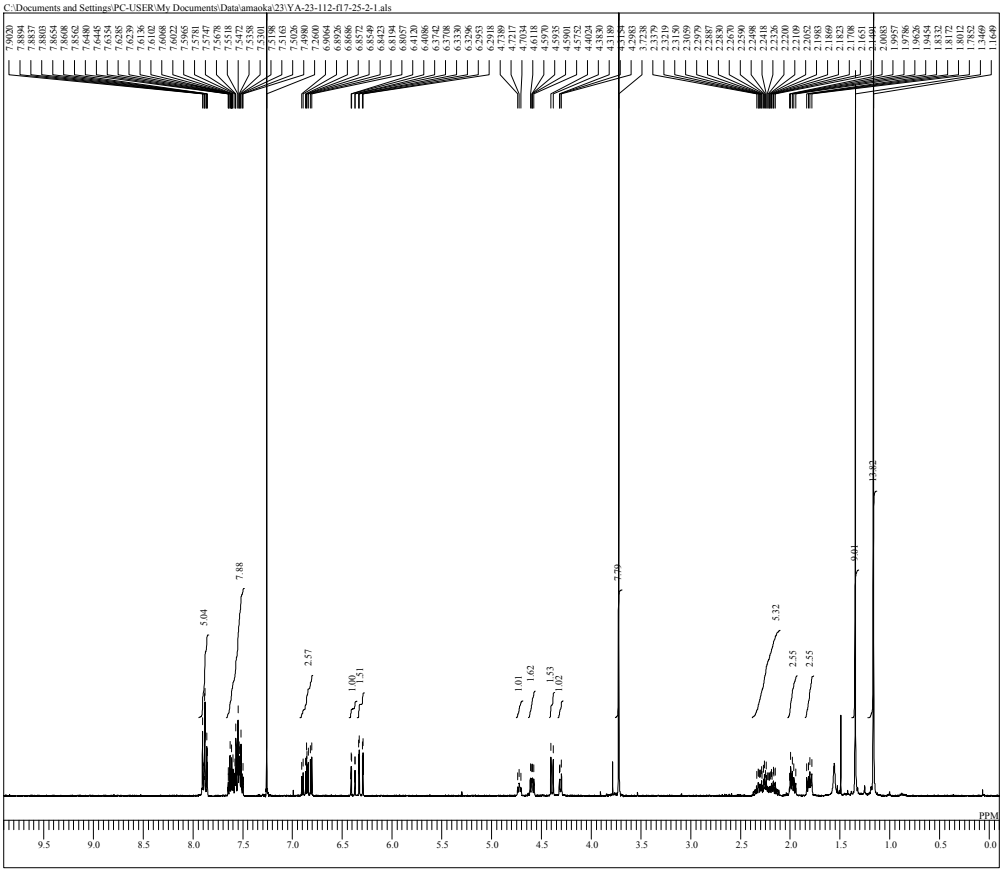
O
N
Boc
SO2Ph

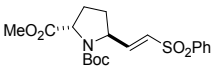
3h


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-092-f11-24-carbon-1.als
DFILE YA-23-092-f11-24-carbon-1.als
COMNT
DATIM 10-09-2013 14:27:09
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 102
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 50.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 3.00 Hz
RGAIN 60
154.3610
142.0216
141.7917
141.7159
140.1427
133.2939
133.1215
129.3947
129.1840
127.6415
80.9183
77.3257
76.6838
68.7084
68.5214
66.9011
66.7778
66.5479
51.5547
39.9817
28.3991
28.3562
28.0683
27.9009
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0

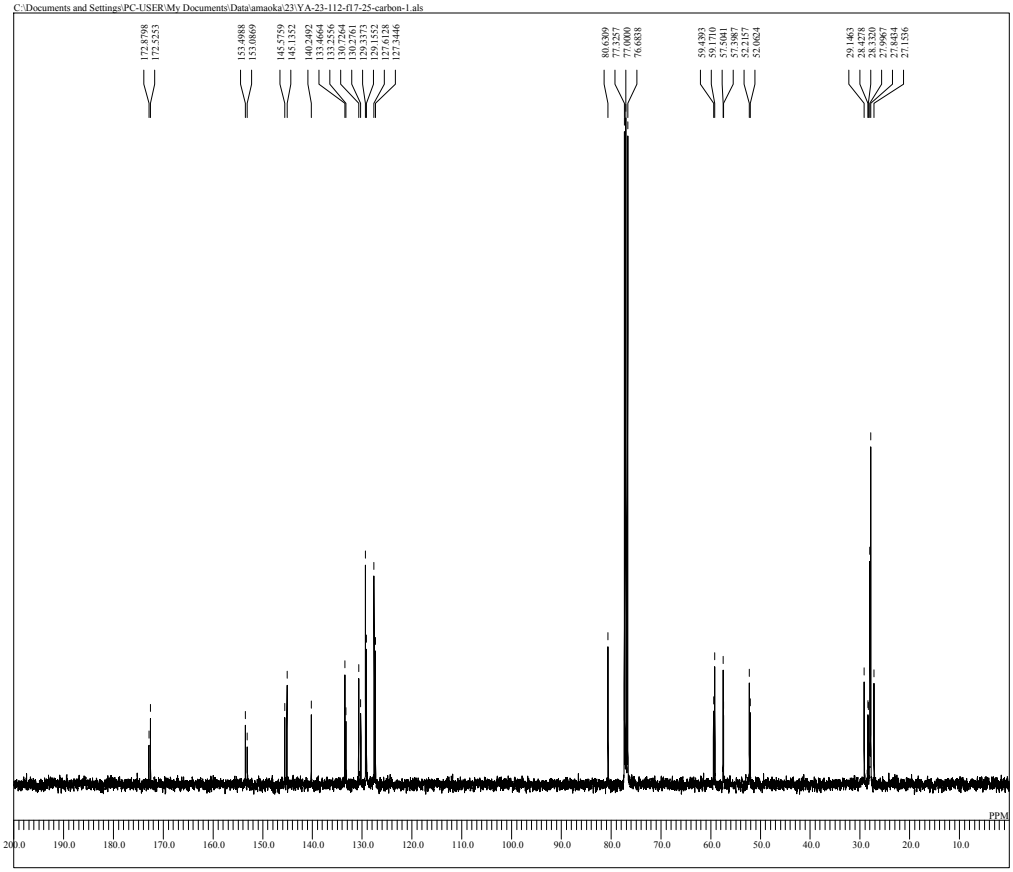

O
N
Boc
SO2Ph

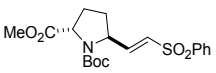
3h


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-112-f17-25-2-1.als
DFILE YA-23-112-f17-25-2-1.als
COMNT
DATIM 17-09-2013 18:26:06
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 50
5.04
7.88
2.57
1.00
1.51
1.01
1.62
1.53
1.02
3.79
5.32
2.55
2.55
9.01
14.82
PPM

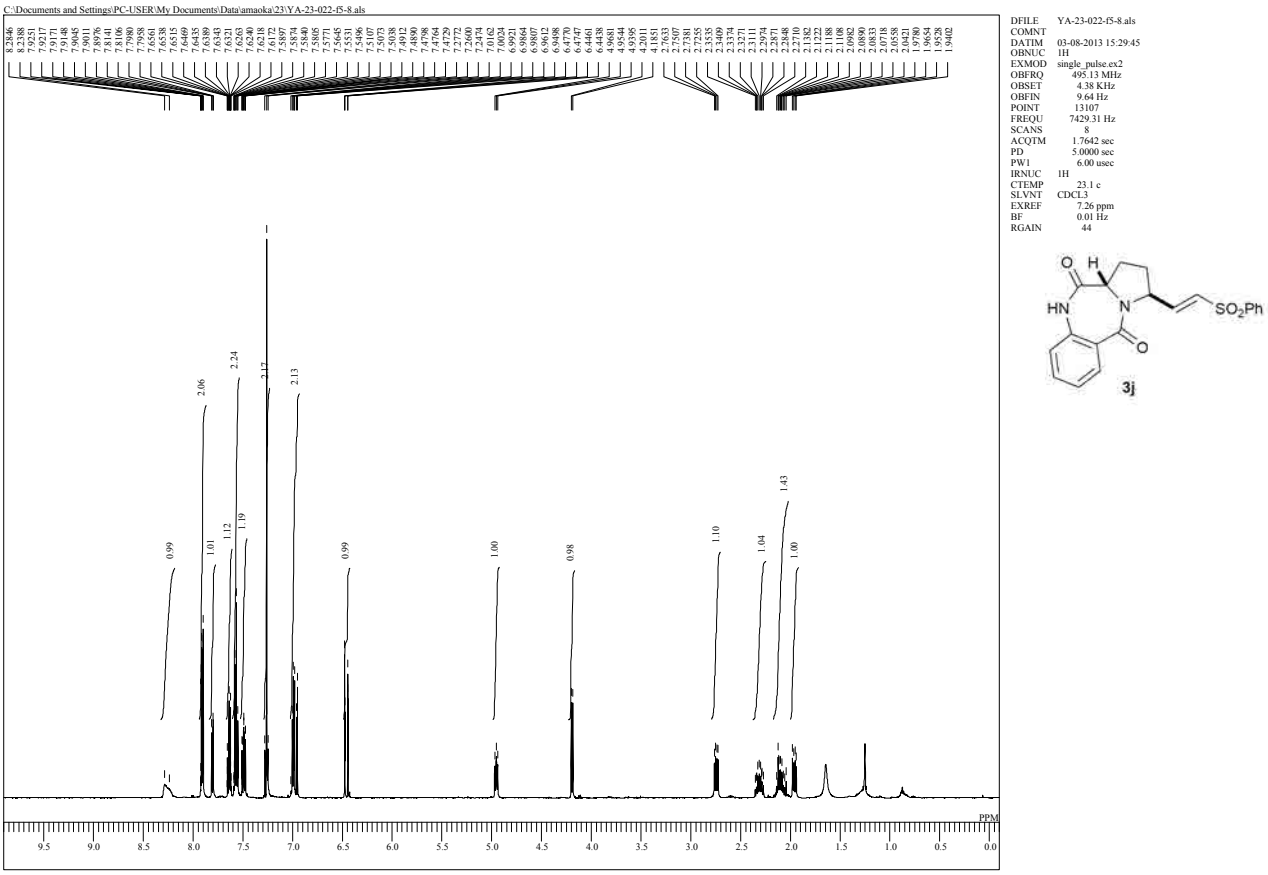

MeO2C
N
Boc
SO2Ph

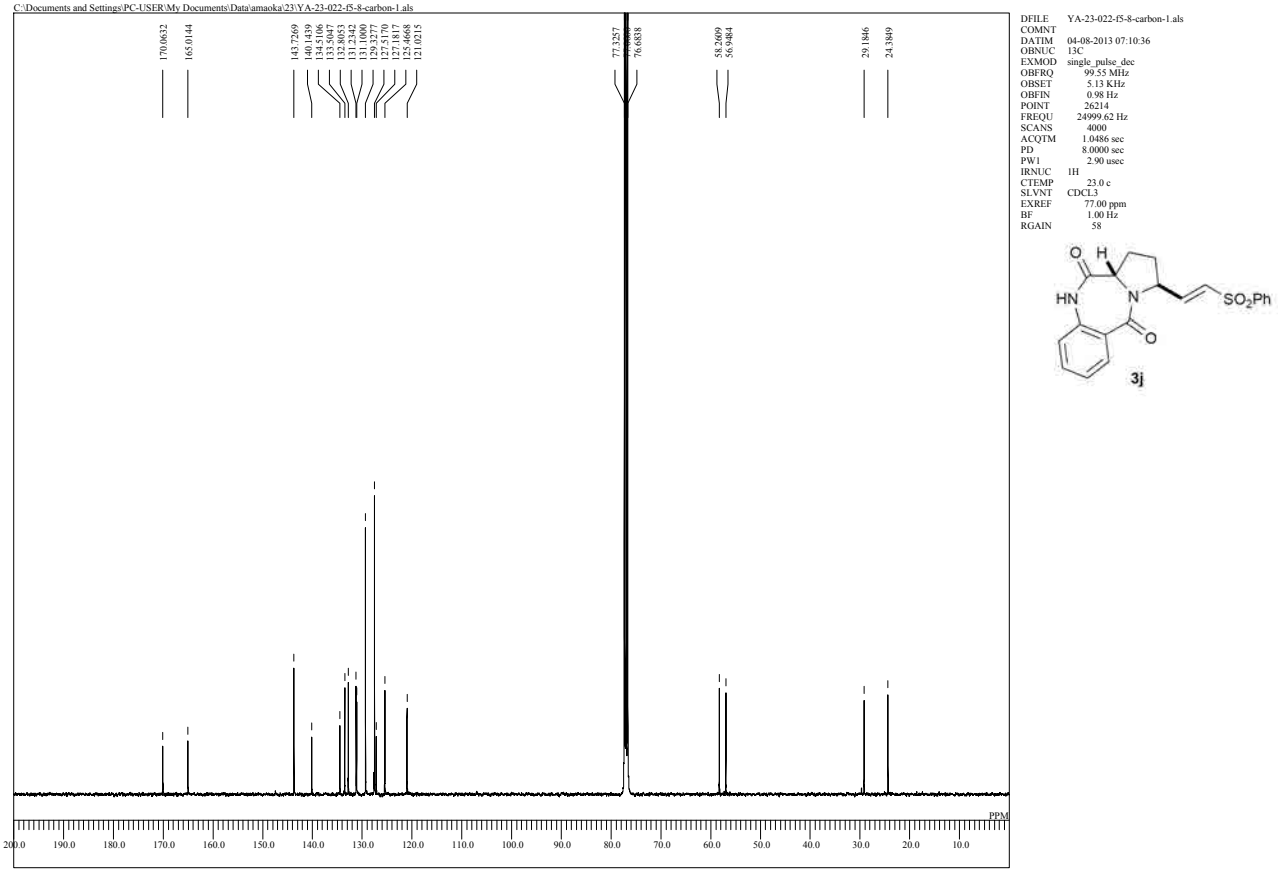
3i

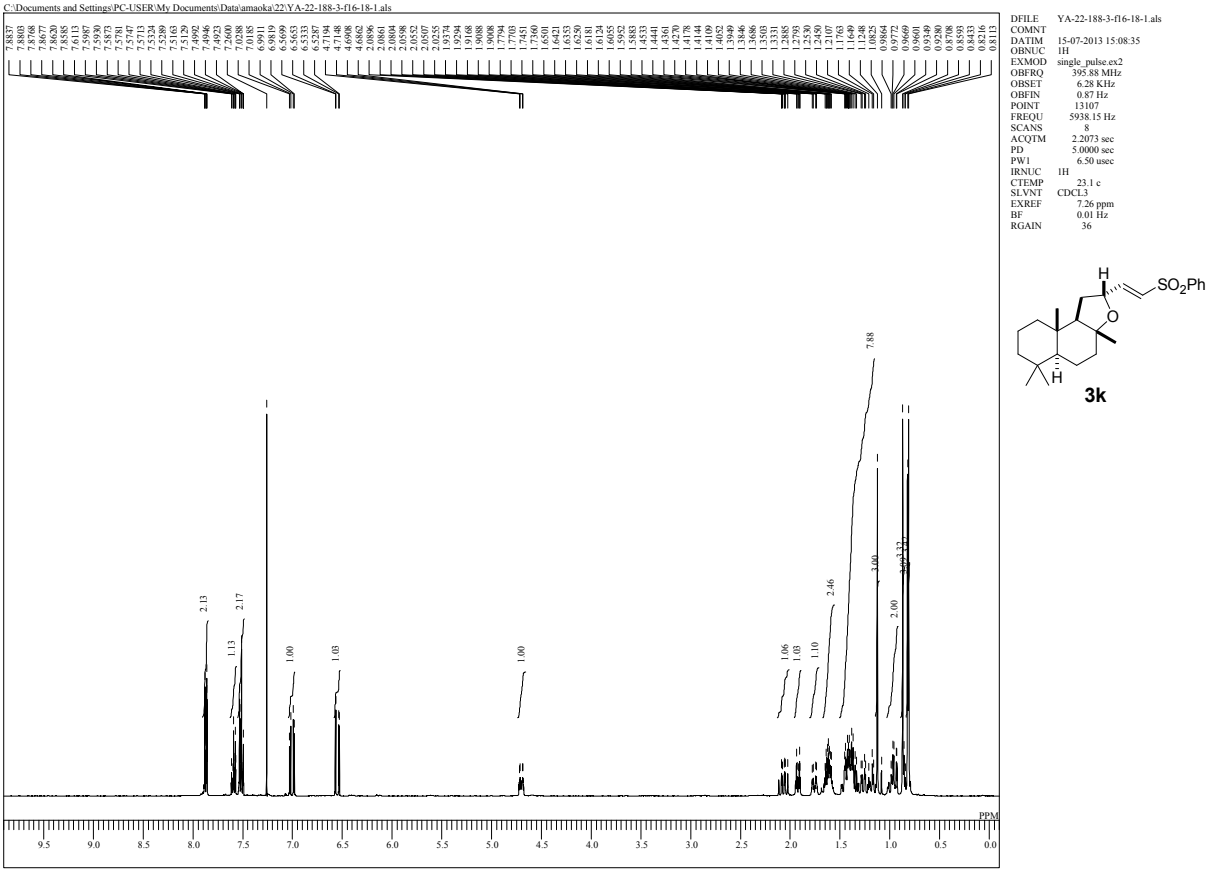

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-112-f17-25-carbon-1.als
DFILE YA-23-112-f17-25-carbon-1.als
COMNT
DATIM 17-09-2013 18:37:30
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 52
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 2.00 Hz
RGAIN 60
172.8798
172.5253
153.4988
153.0869
145.5759
145.1352
140.2492
133.4664
133.2556
130.7264
130.3761
129.3373
129.1552
127.6128
127.3446
80.6399
77.3257
77.0000
76.6838
59.4393
59.1710
57.5941
57.3987
53.1577
52.6624
29.1463
28.4278
28.3320
27.3967
27.8434
27.1536
PPM


MeO2C
N
Boc
SO2Ph

3i

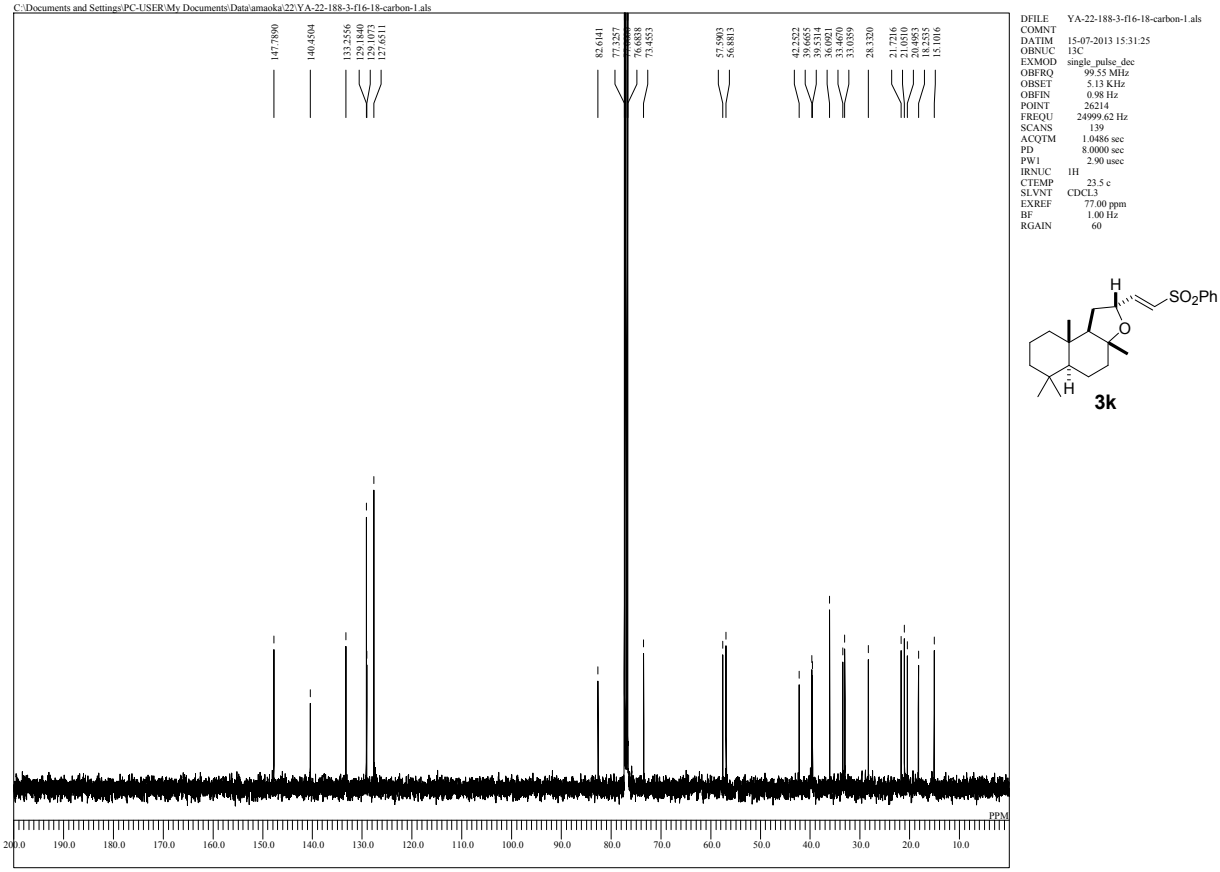

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-022-f5-8.als
DFILE YA-23-022-f5-8.als
COMNT
DATIM 03-08-2013 15:29:45
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 44
0.99
2.06
1.01
1.12
2.24
1.19
2.17
2.13
0.99
1.00
0.98
1.10
1.04
1.43
1.00
PPM
3j


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-022-f5-8-carbon-1.als
DFILE YA-23-022-f5-8-carbon-1.als
COMNT
DATIM 04-08-2013 07:10:36
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 4000
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 2.90 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 58
170.0632
165.0144
143.7269
140.1439
134.5106
133.5047
132.8053
131.2342
131.1060
129.3277
127.5170
125.1648
121.0215
77.3257
76.6838
58.3499
56.9484
29.1846
24.3849
PPM
3j


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\22\YA-22-188-3-f16-18-1.als
DFILE YA-22-188-3-f16-18-1.als
COMNT
DATIM 15-07-2013 15:08:35
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 36
2.13
2.17
1.13
1.00
1.03
1.00
1.06
1.03
1.10
2.46
3.00
2.00
7.88
PPM

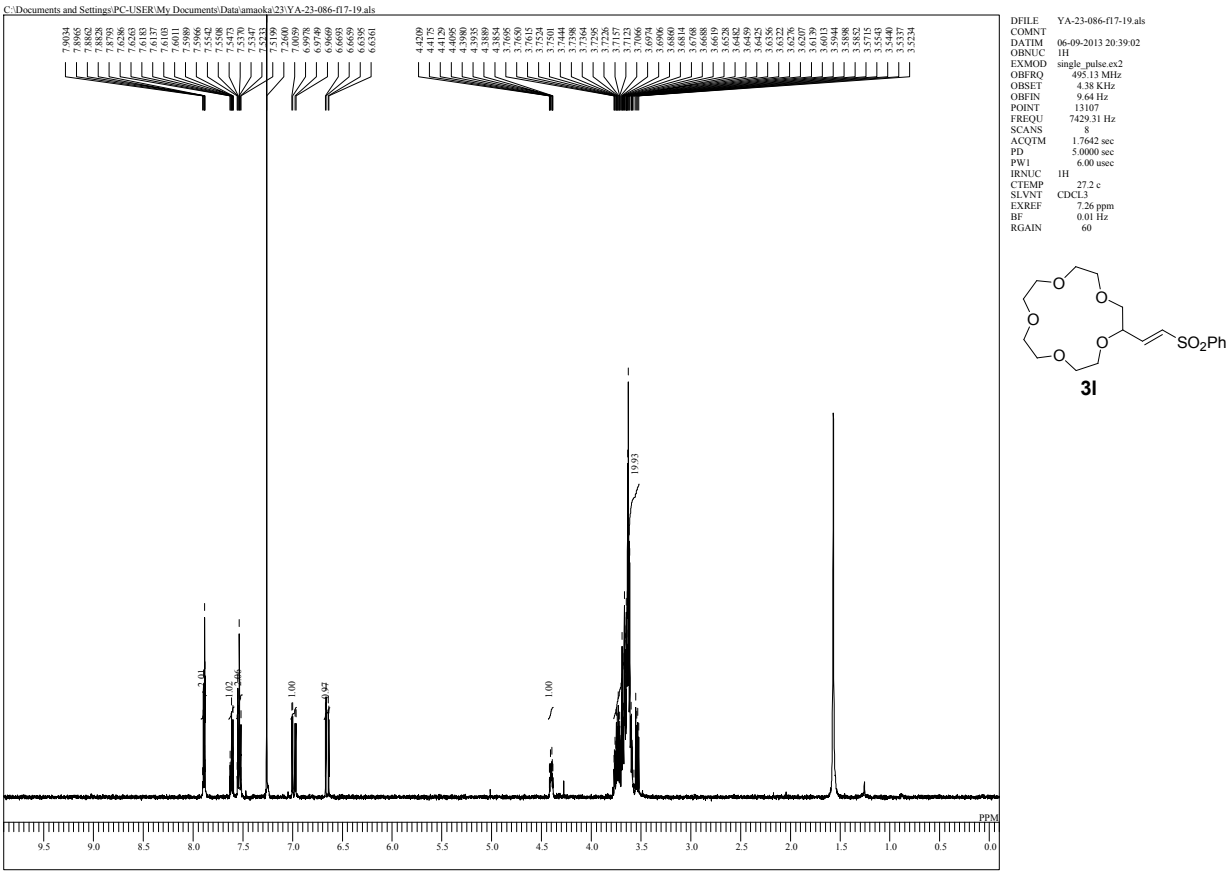
H
SO2Ph
O
H

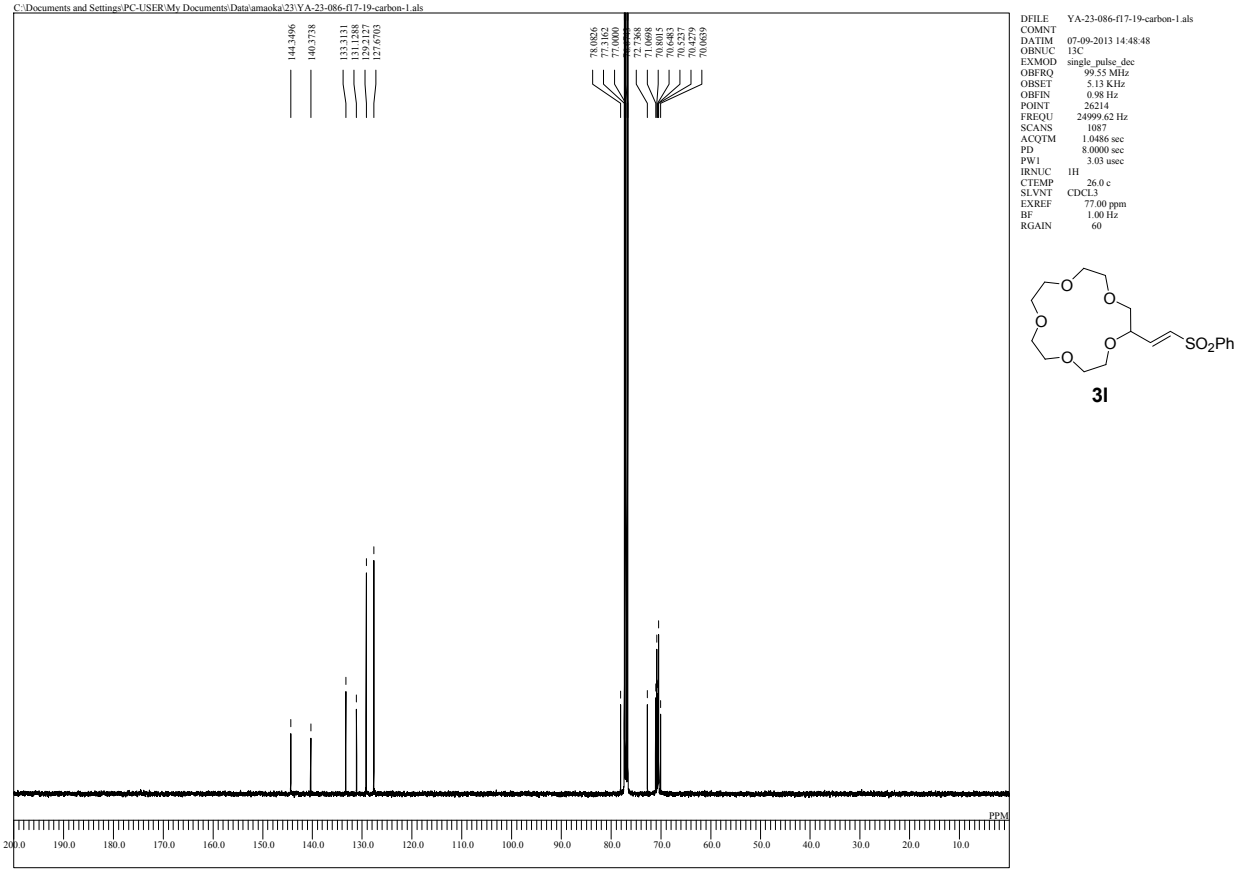
3k

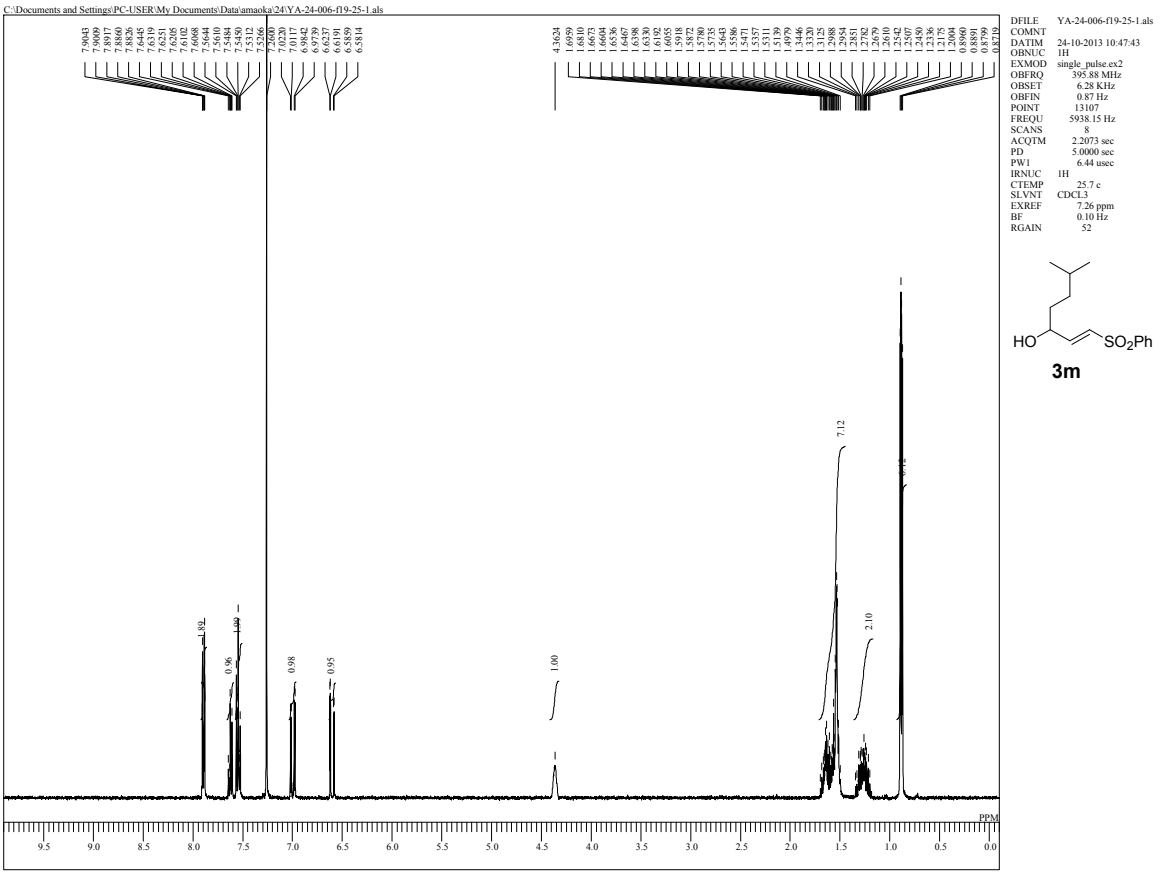

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\22\YA-22-188-3-f16-18-carbon-1.als
DFILE YA-22-188-3-f16-18-carbon-1.als
COMNT
DATIM 15-07-2013 15:31:25
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 139
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 2.90 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
147.7890
140.4504
133.2556
129.1840
129.1073
127.6511
82.6141
77.3257
76.6838
73.4553
57.5903
56.6813
42.2522
39.6665
39.5314
36.6980
33.4670
33.0359
28.3320
21.7216
21.0510
20.4953
18.5255
15.1016
PPM

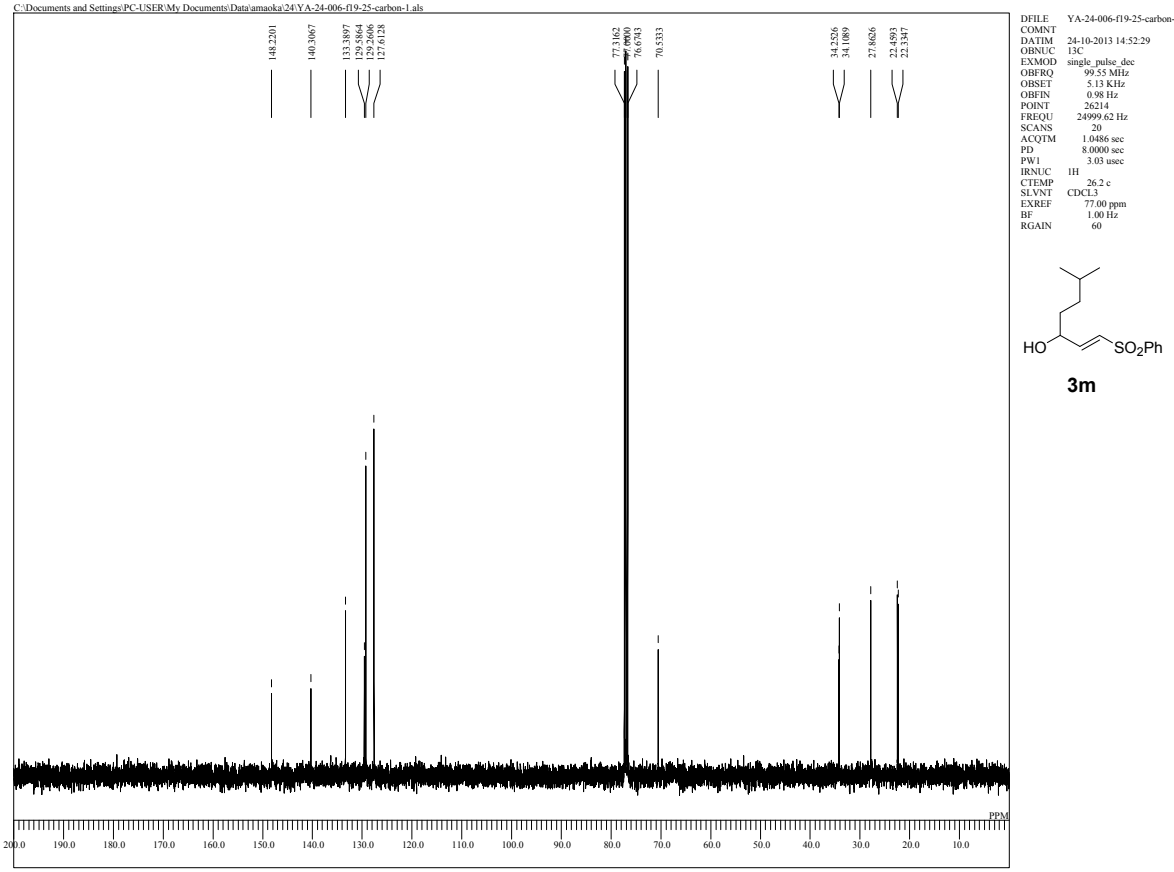
H
SO2Ph
O
H

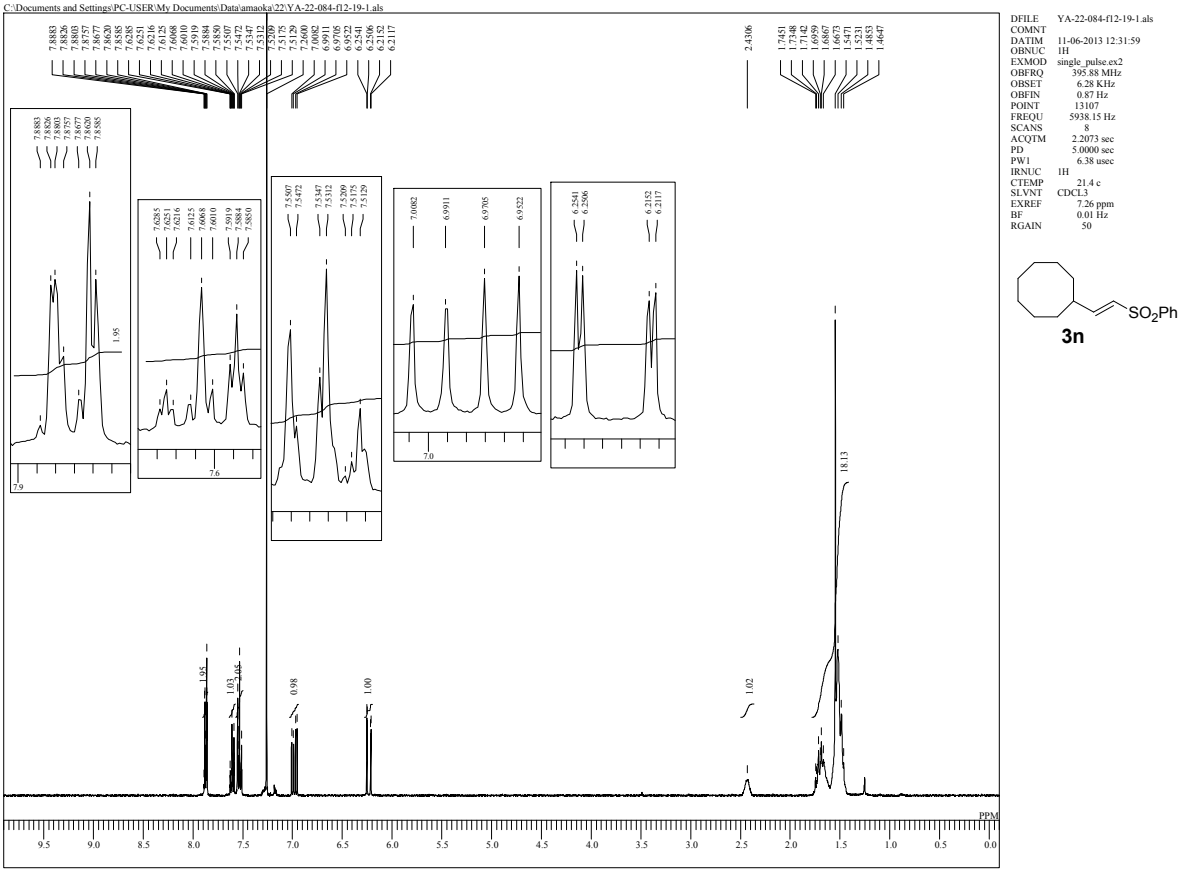
3k

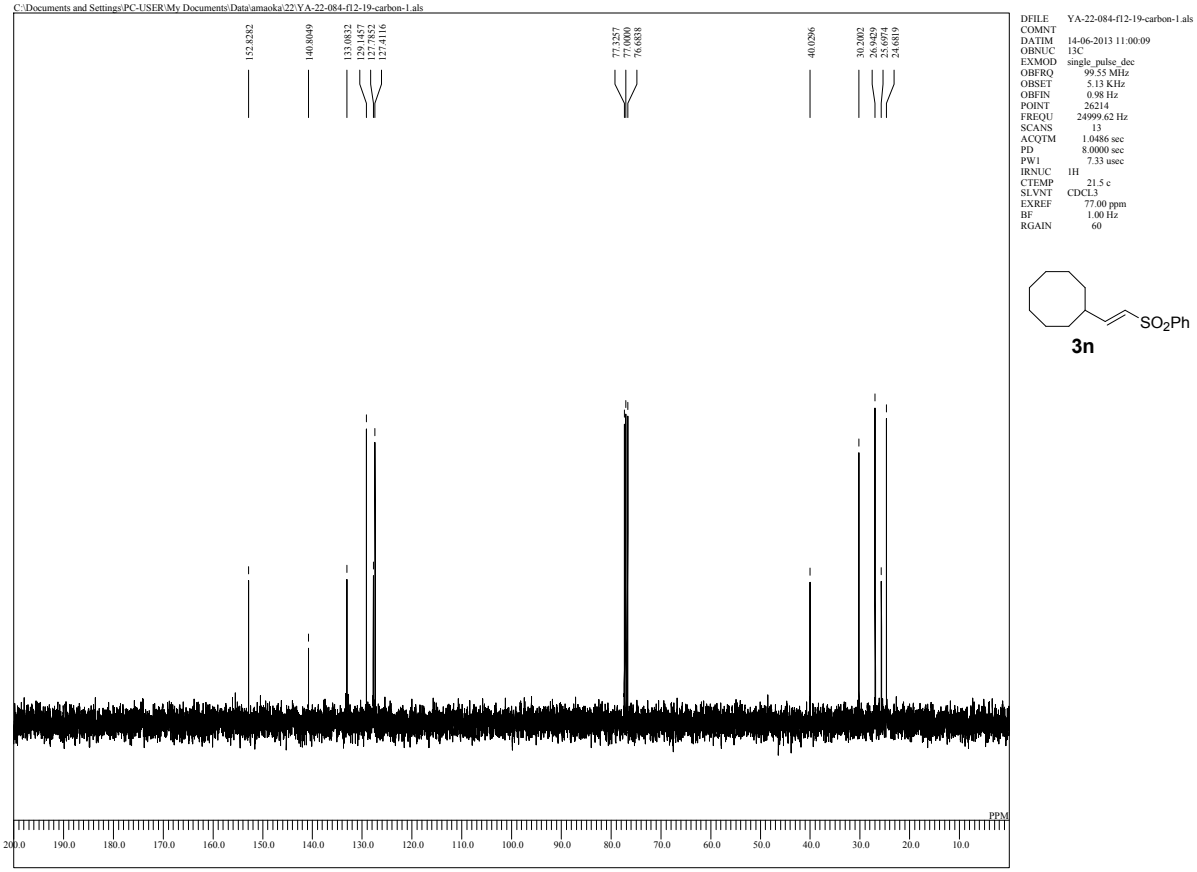

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-086-f17-19.als
DFILE YA-23-086-f17-19.als
COMNT
DATIM 06-09-2013 20:39:02
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
POINT 13107
FREQU 7429.31 Hz
SCANS 8
ACQTM 1.7642 sec
PD 5.0000 sec
PW1 6.00 usec
IRNUC 1H
CTEMP 27.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 60
SO2Ph
3l
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-086-f17-19-carbon-1.als
DFILE YA-23-086-f17-19-carbon-1.als
COMNT
DATIM 07-09-2013 14:48:48
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 1087
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 26.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
SO2Ph
3l
PPM

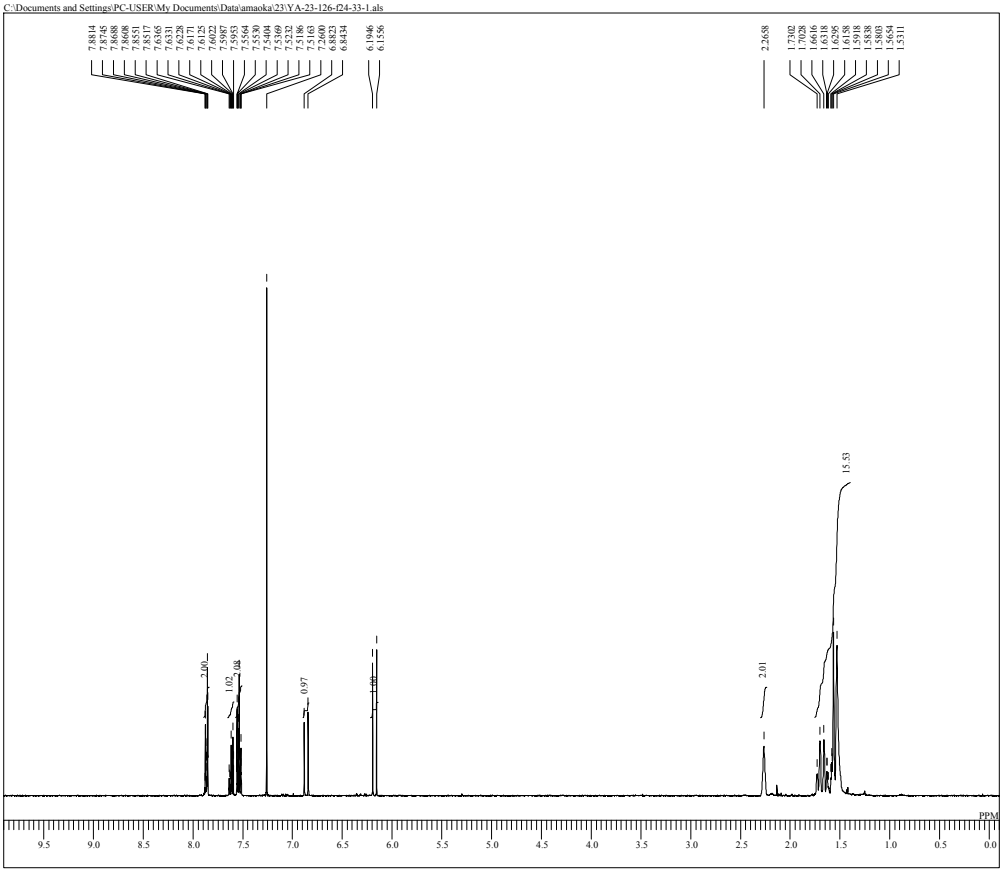

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-006-f19-25-1.als
DFILE YA-24-006-f19-25-1.als
COMNT
DATIM 24-10-2013 10:47:43
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 25.7 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 52
HO
SO2Ph
3m
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-006-f19-25-carbon-1.als
DFILE YA-24-006-f19-25-carbon-1.als
COMNT
DATIM 24-10-2013 14:52:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 20
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 26.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
HO
SO2Ph
3m
PPM

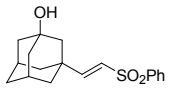

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\22\YA-22-084-f12-19-1.als
DFILE YA-22-084-f12-19-1.als
COMNT
DATIM 11-06-2013 12:31:59
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.38 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 50
SO2Ph
3n
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\22\YA-22-084-f12-19-carbon-1.als
DFILE YA-22-084-f12-19-carbon-1.als
COMNT
DATIM 14-06-2013 11:00:09
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 13
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 7.33 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
SO2Ph
3n
PPM

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-126-f24-33-1.als

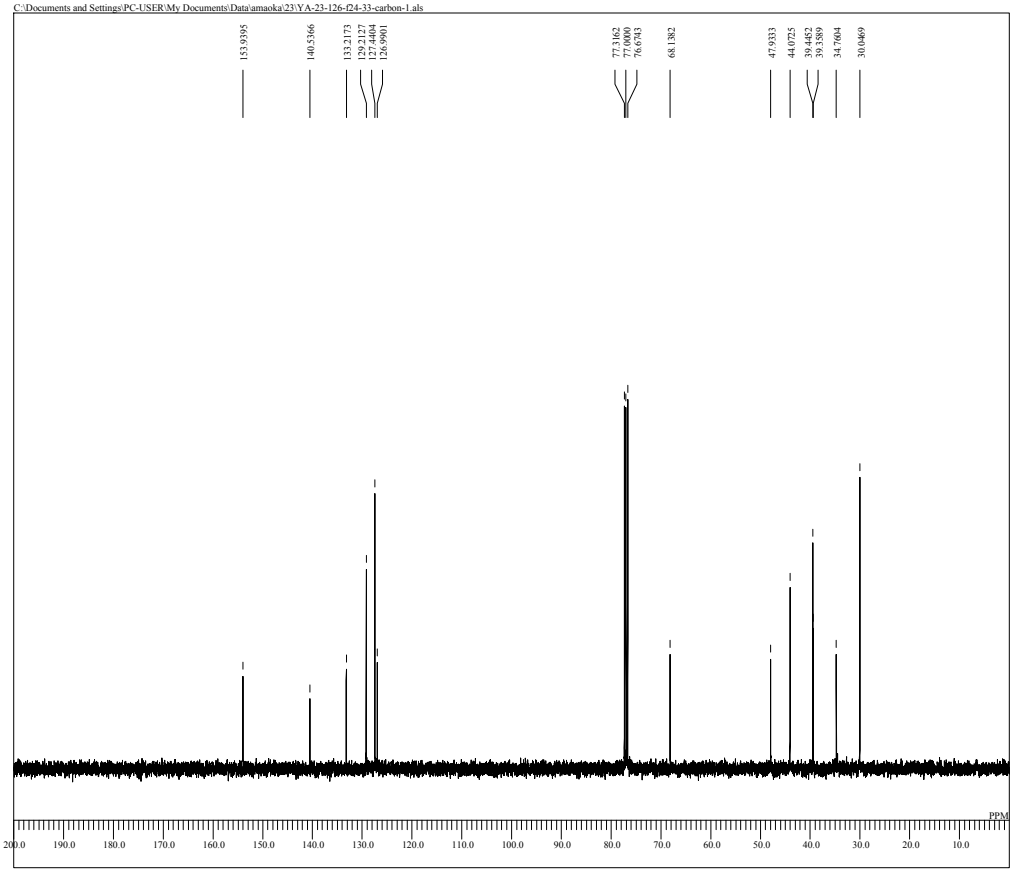


DFILE YA-23-126-f24-33-1.als
COMNT
DATIM 28-09-2013 10:14:35
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 46

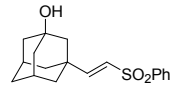


3o

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-126-f24-33-carbon-1.als



DFILE YA-23-126-f24-33-carbon-1.als
COMNT
DATIM 28-09-2013 10:25:07
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 13
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60



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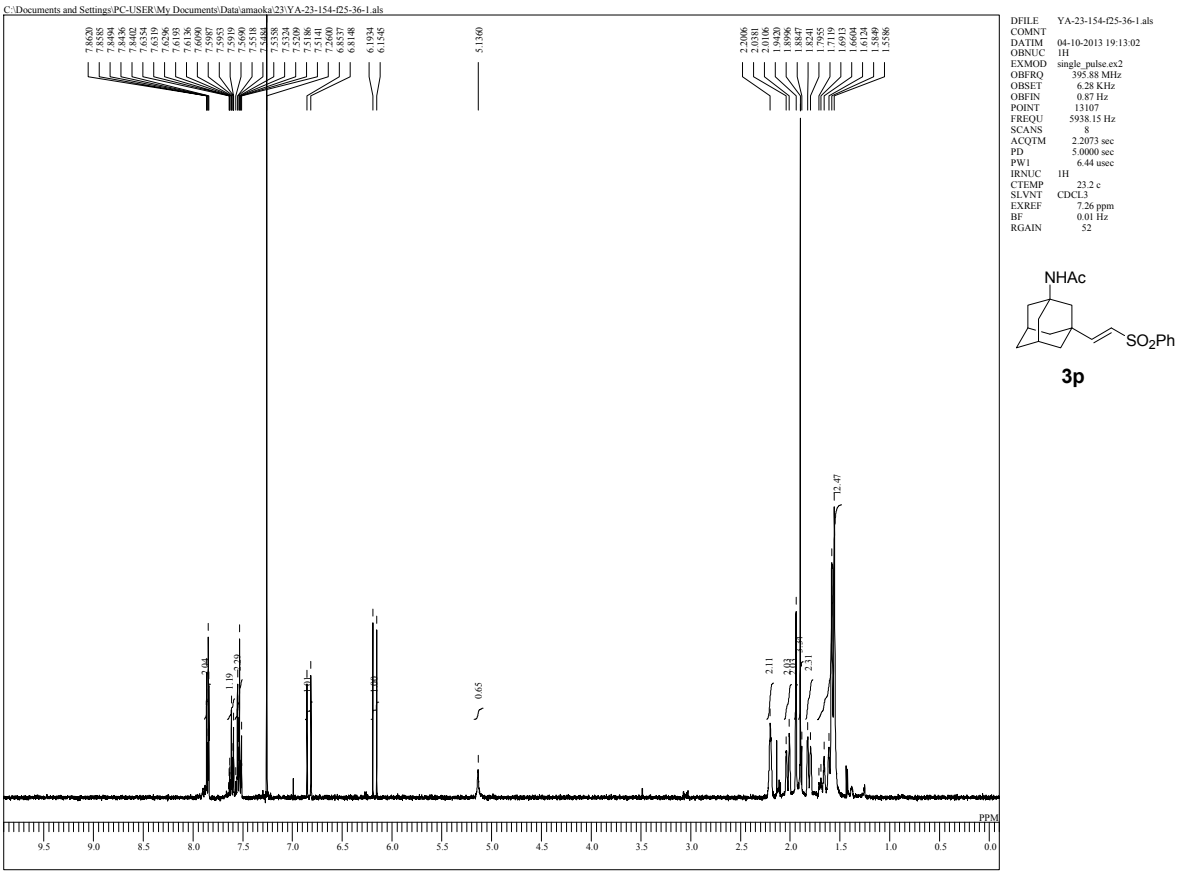
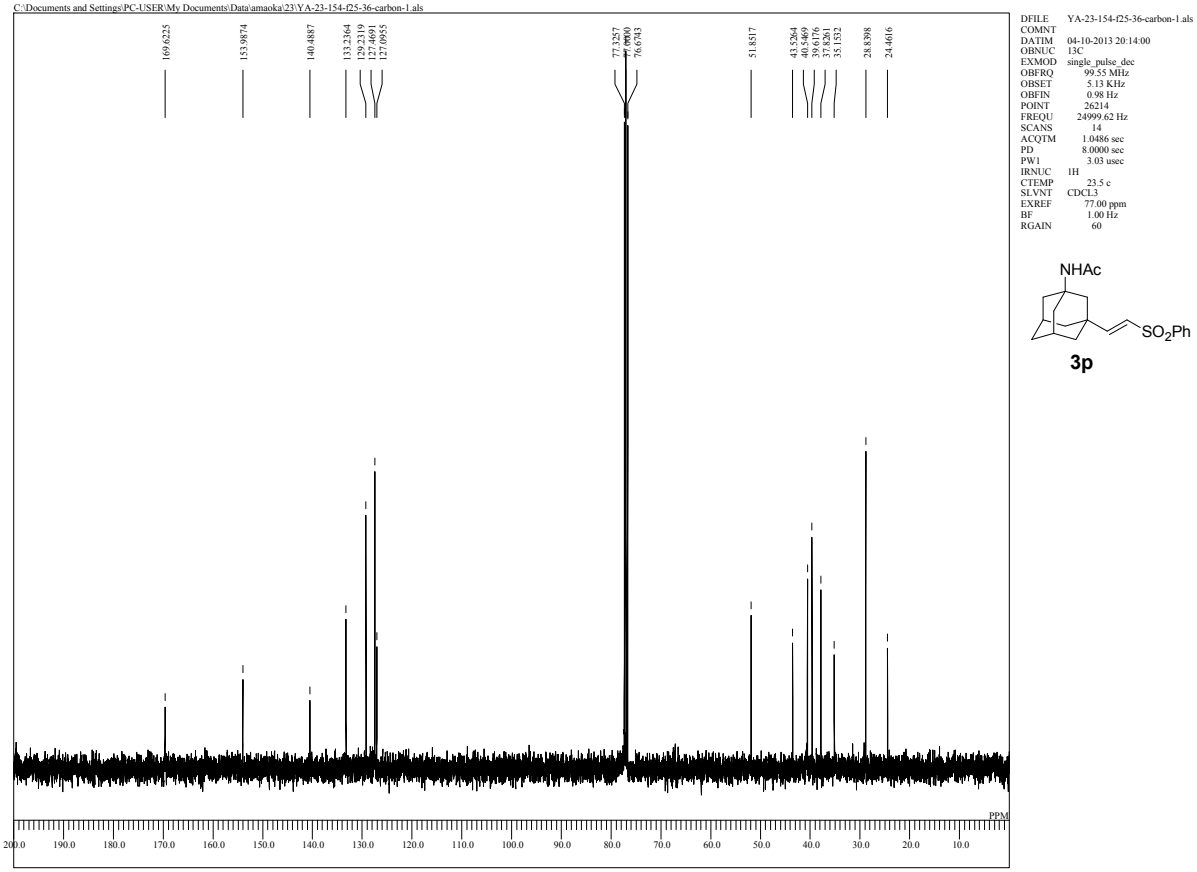
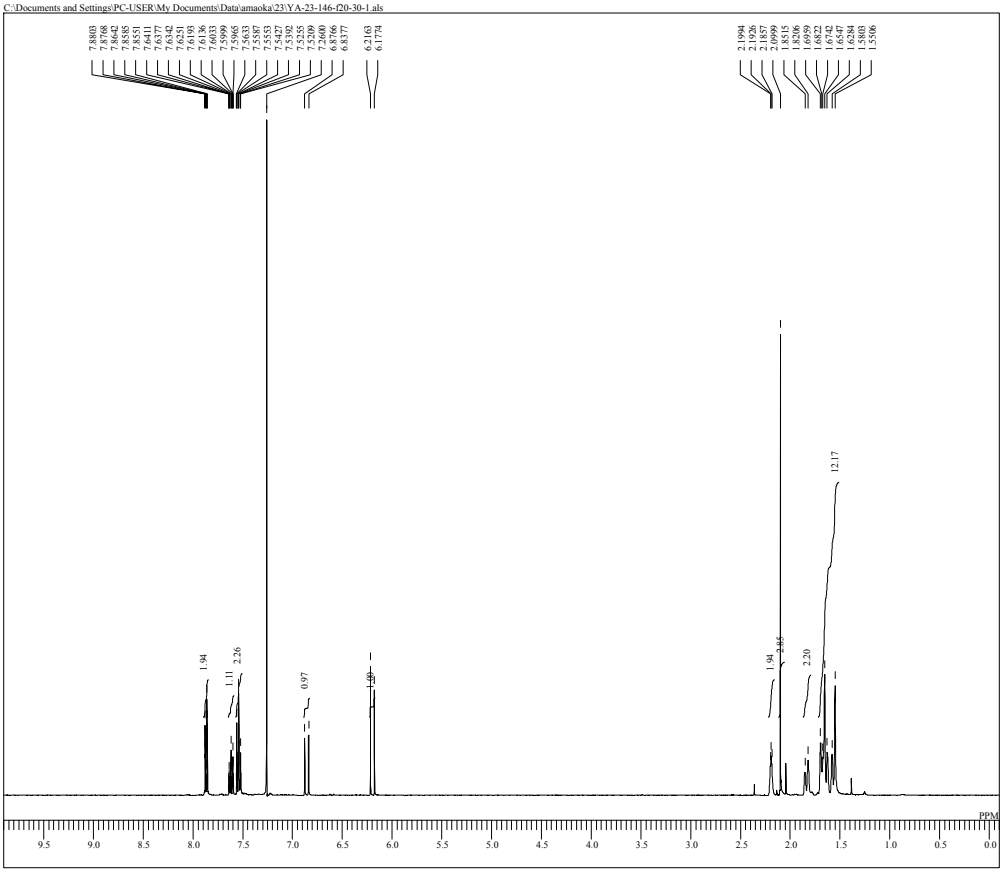
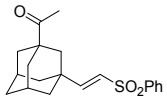

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-154-f25-36-1.als
DFILE YA-23-154-f25-36-1.als
COMNT
DATIM 04-10-2013 19:13:02
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 52
NHAc
SO2Ph
3p
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-154-f25-36-carbon-1.als
DFILE YA-23-154-f25-36-carbon-1.als
COMNT
DATIM 04-10-2013 20:14:00
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 14
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 23.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
NHAc
SO2Ph
3p
PPM

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-146-f20-30-1.als

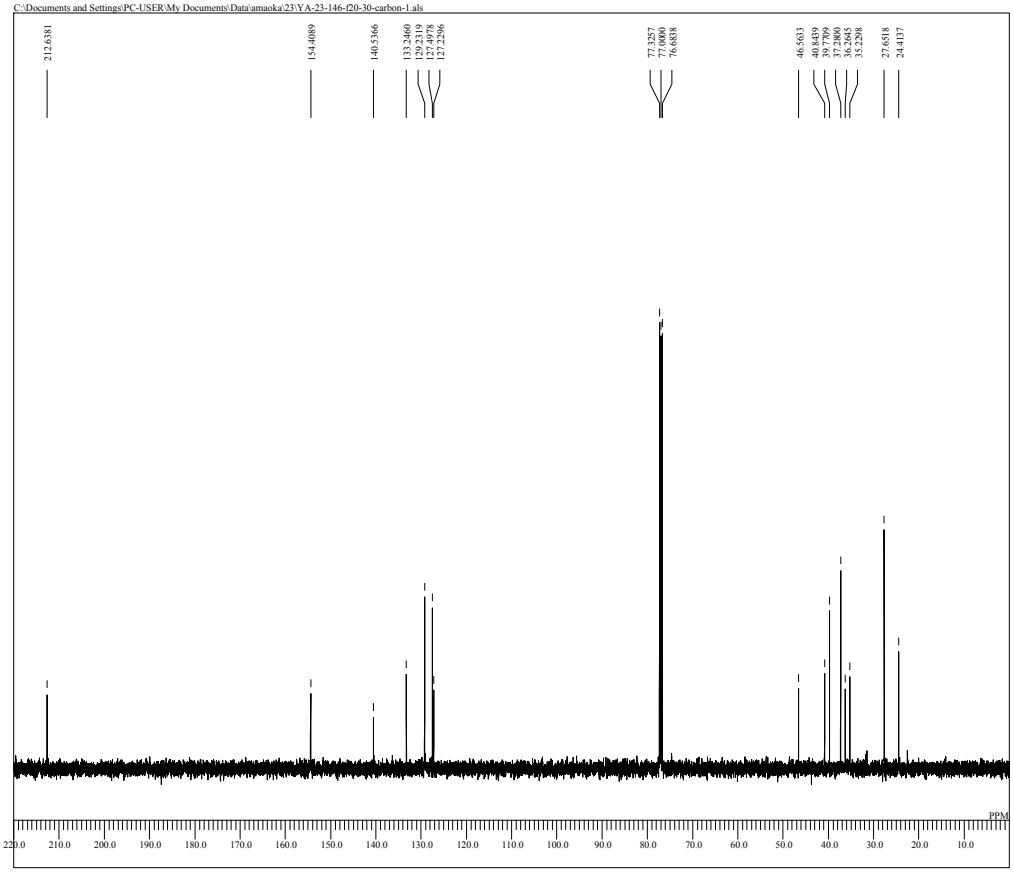


DFILE YA-23-146-f20-30-1.als
COMNT
DATIM 01-10-2013 18:24:23
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 48

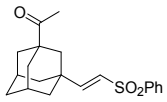


3q

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-146-f20-30-carbon-1.als



DFILE YA-23-146-f20-30-carbon-1.als
COMNT
DATIM 01-10-2013 19:42:21
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 15
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 23.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60



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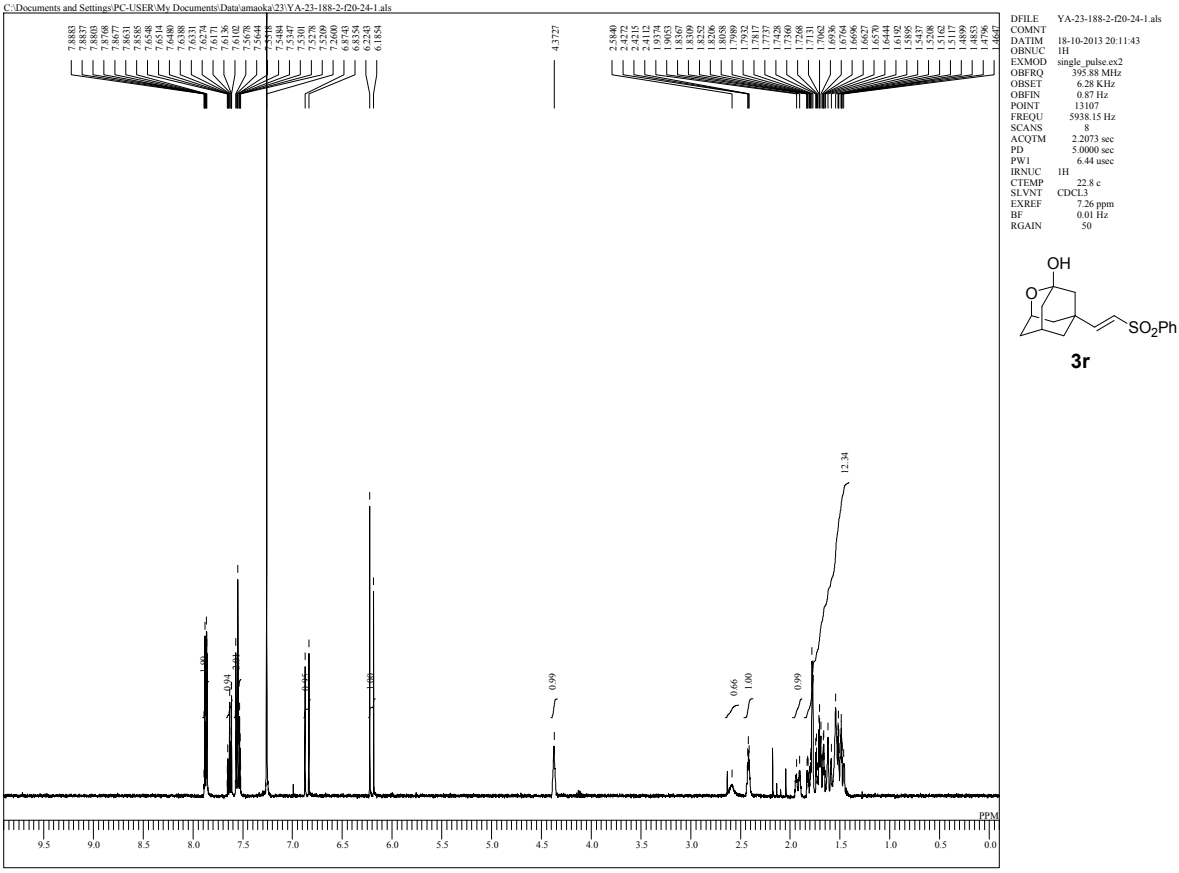
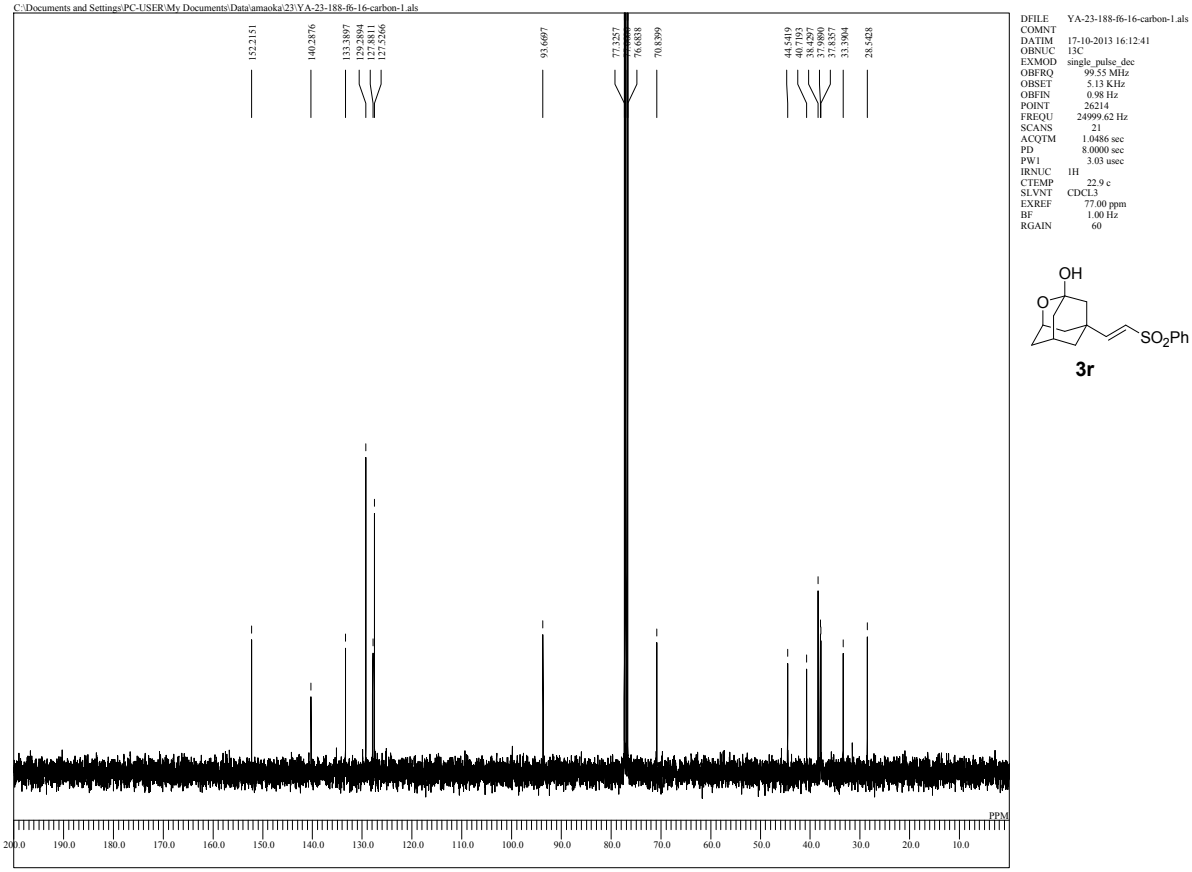
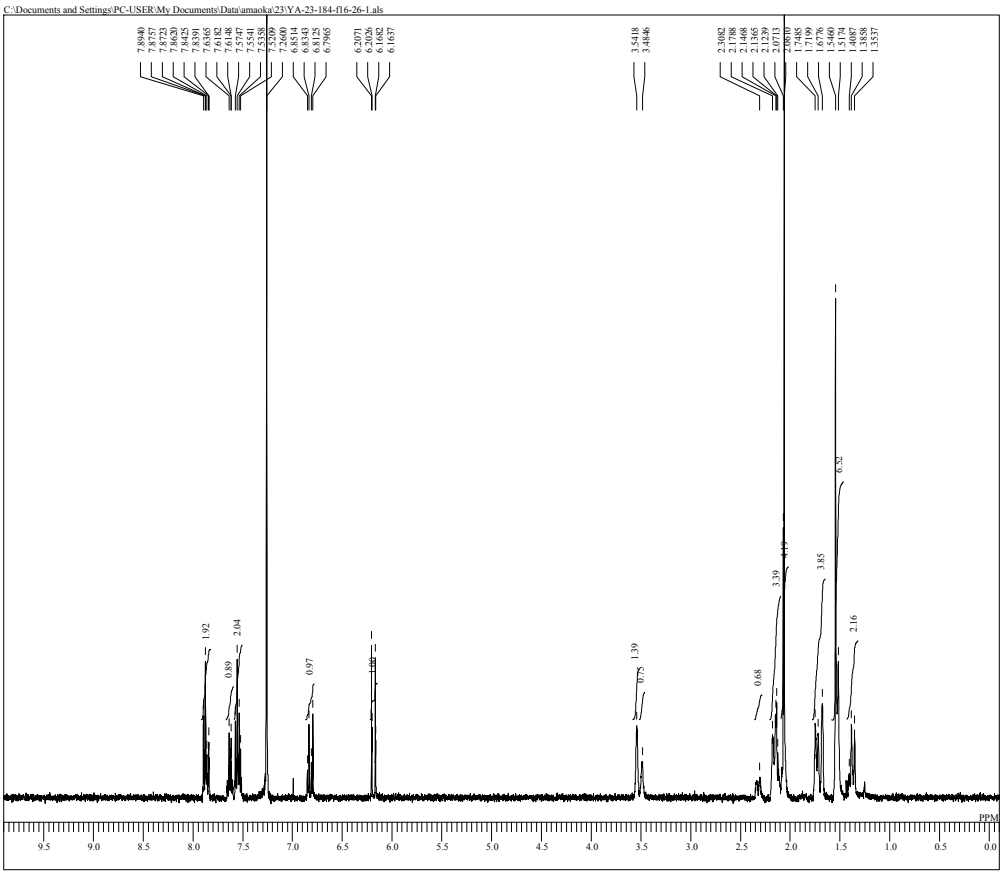
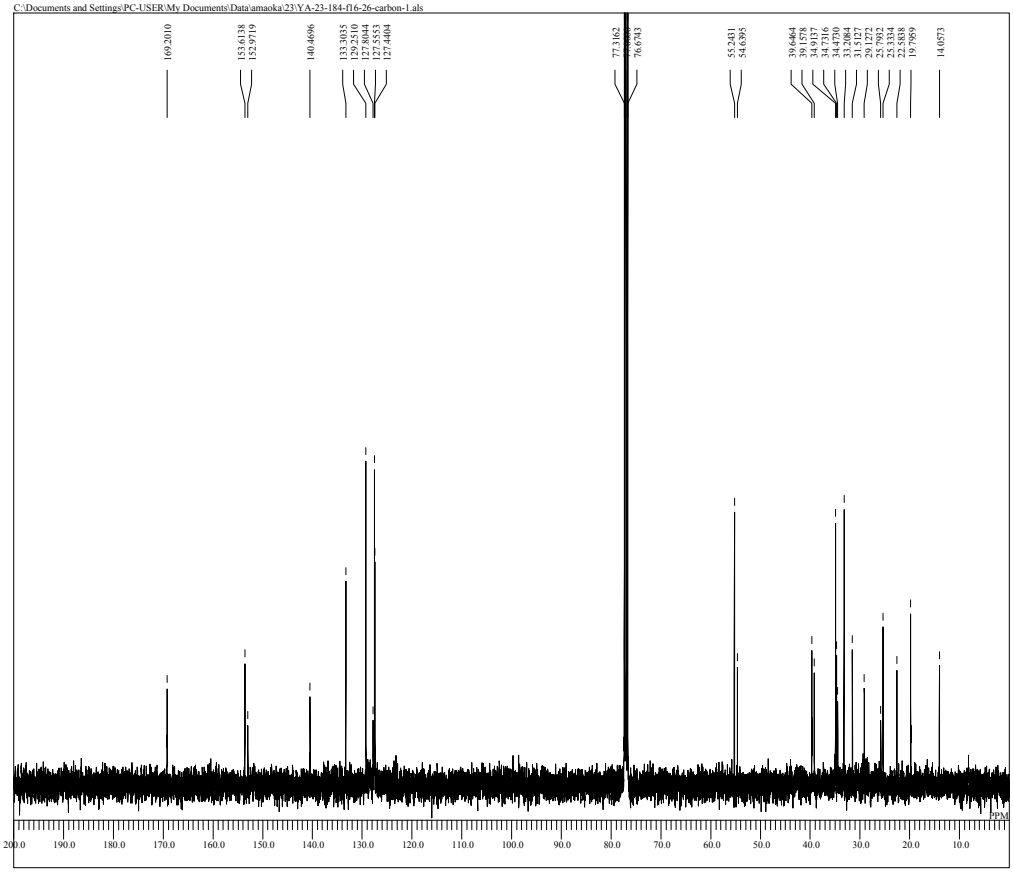
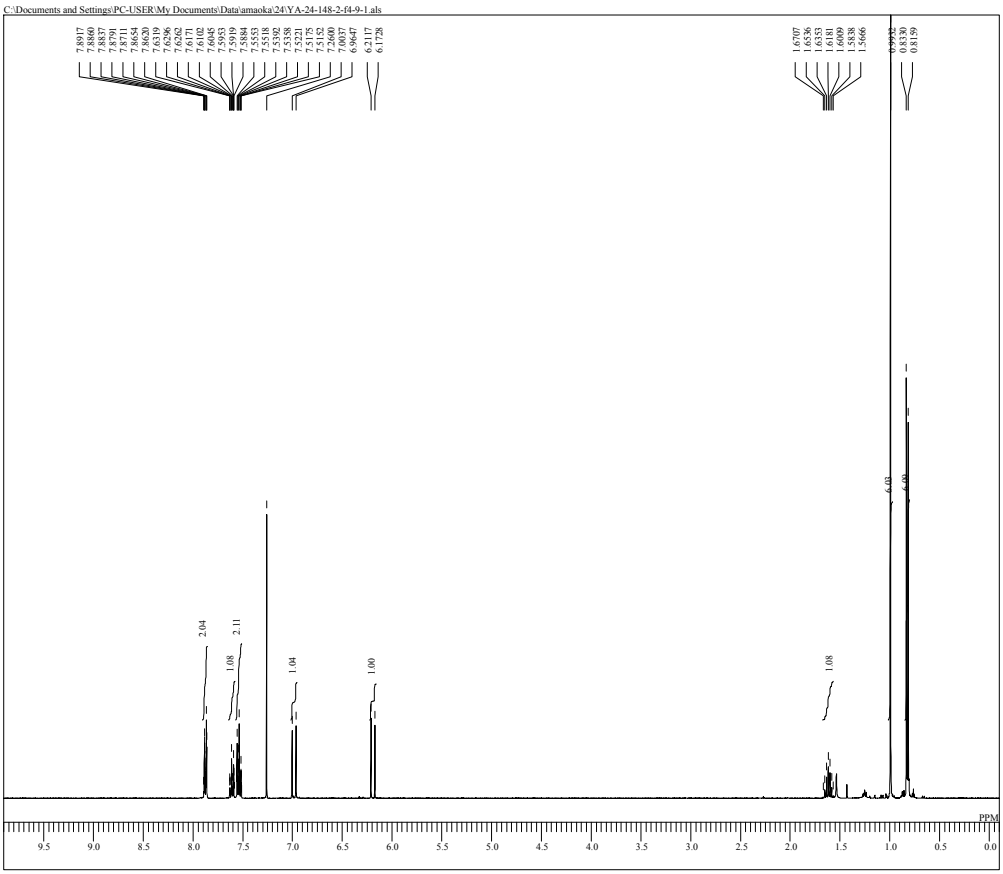
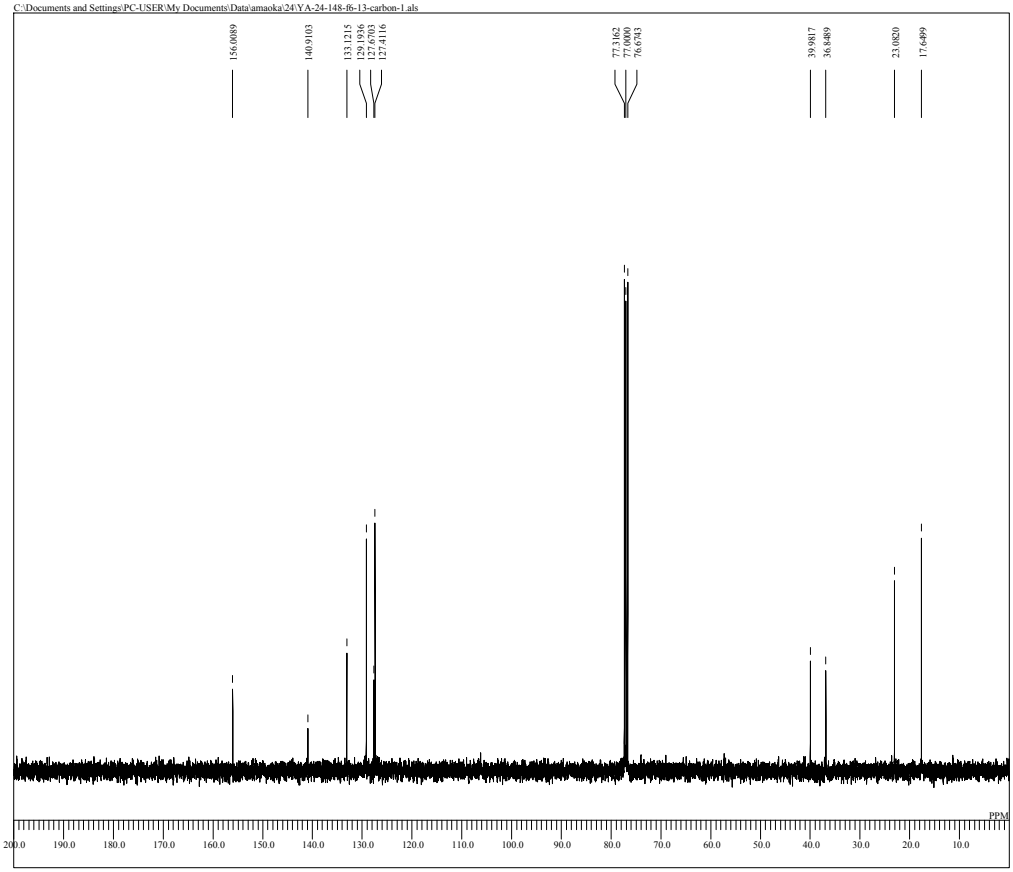

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-188-2-f20-24-1.als
DFILE YA-23-188-2-f20-24-1.als
COMNT
DATIM 18-10-2013 20:11:43
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 50
OH
O
SO2Ph
3r
PPM

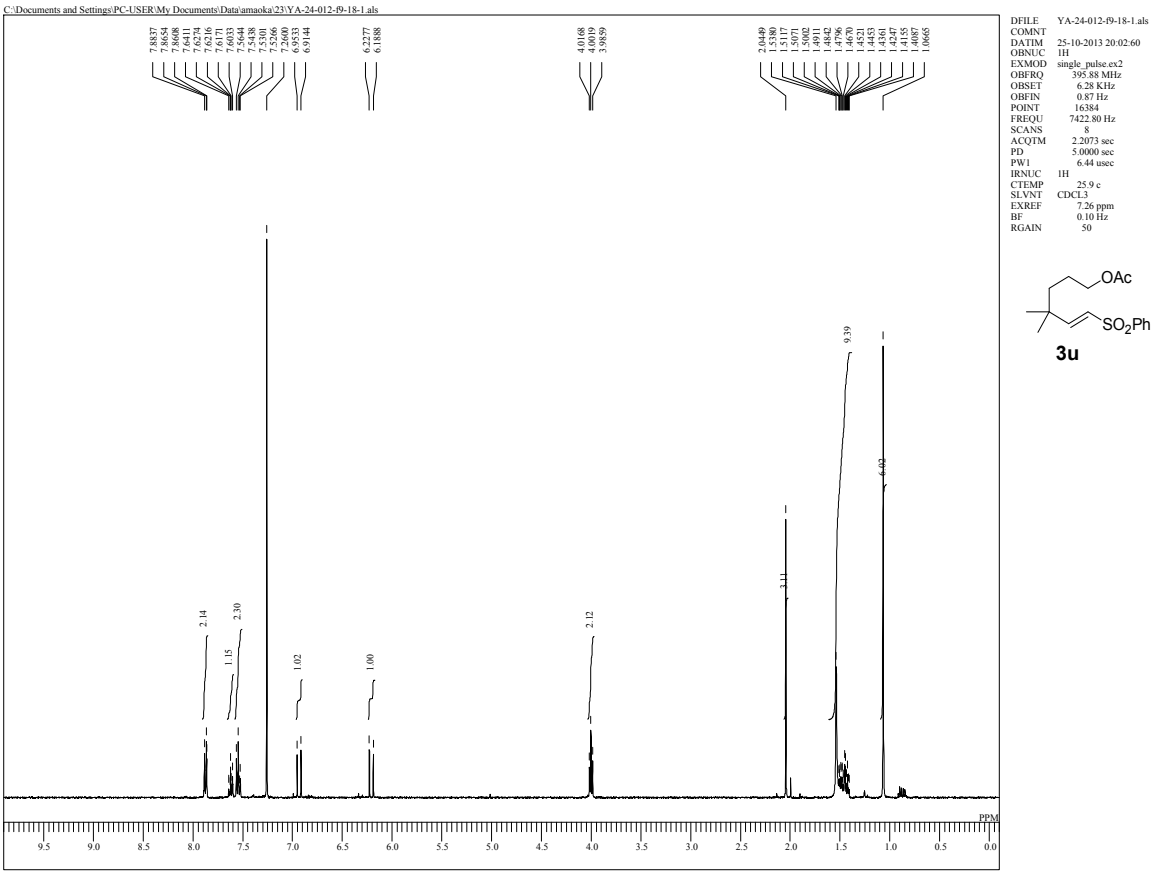

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-188-f6-16-carbon-1.als
DFILE YA-23-188-f6-16-carbon-1.als
COMNT
DATIM 17-10-2013 16:12:41
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 21
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
OH
O
SO2Ph
3r
PPM

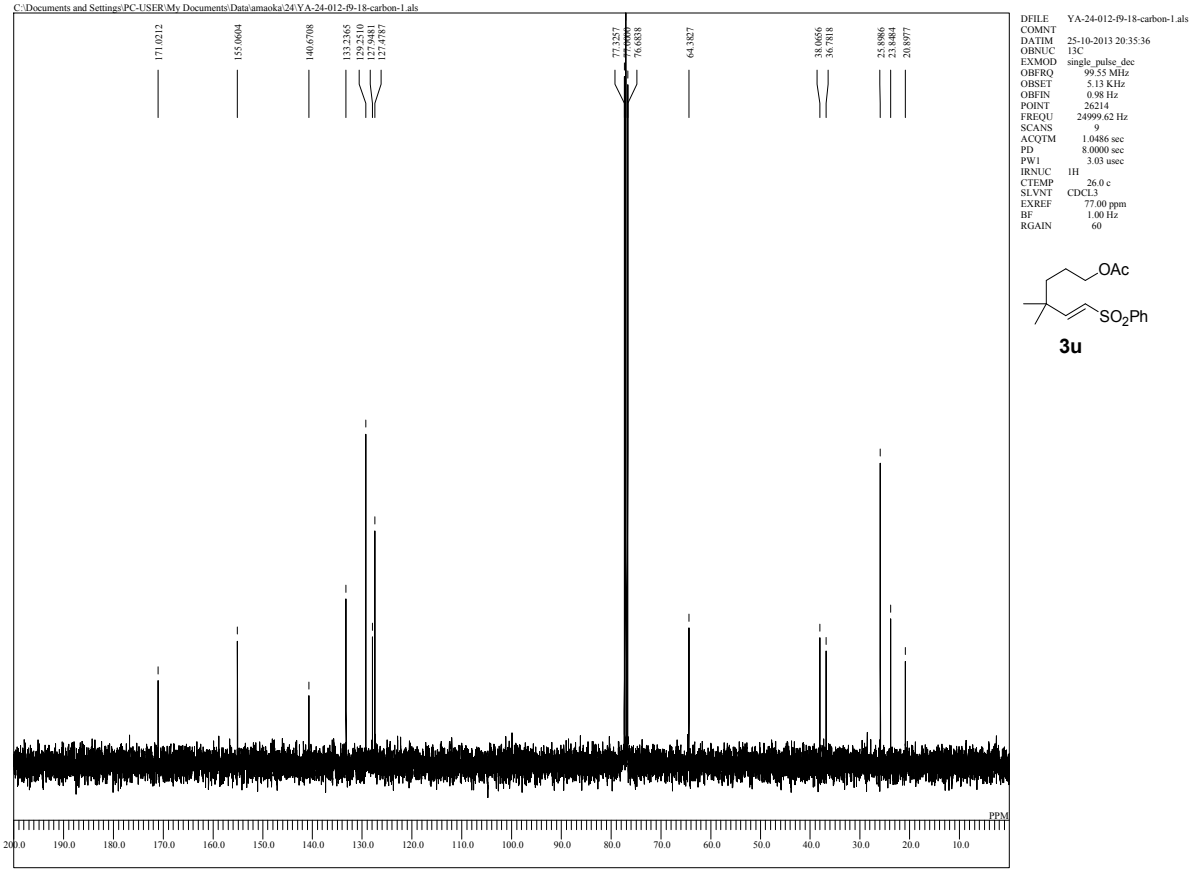

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-184-f16-26-1.als
DFILE YA-23-184-f16-26-1.als
COMNT
DATIM 12-10-2013 15:12:55
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.01 Hz
RGAIN 54
AcO
N
SO2Ph
3s
PPM

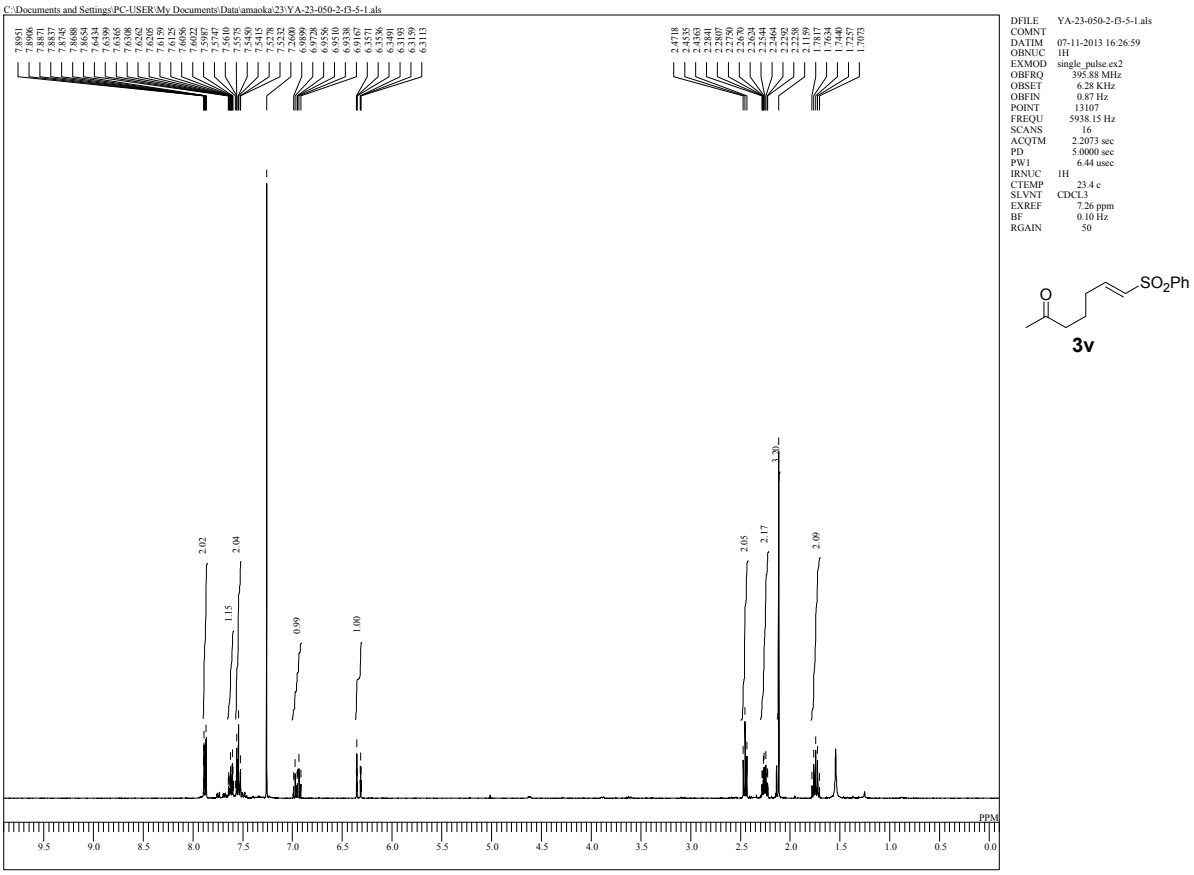

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-184-f16-26-carbon-1.als
DFILE YA-23-184-f16-26-carbon-1.als
COMNT
DATIM 15-10-2013 15:38:42
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 32768
FREQU 31250.00 Hz
SCANS 86
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
AcO
N
SO2Ph
3s
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-148-2-f4-9-1.als
DFILE YA-24-148-2-f4-9-1.als
COMNT
DATIM 22-01-2014 20:12:51
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 46
SO2Ph
3t
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-148-f6-13-carbon-1.als
DFILE YA-24-148-f6-13-carbon-1.als
COMNT
DATIM 22-01-2014 10:34:16
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 16
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
SO2Ph
3t
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-24-012-f9-18-1.als
DFILE YA-24-012-f9-18-1.als
COMNT
DATIM 25-10-2013 20:02:60
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 16384
FREQU 7422.80 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 25.9 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 50
OAc
SO2Ph
3u
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-012-f9-18-carbon-1.als
DFILE YA-24-012-f9-18-carbon-1.als
COMNT
DATIM 25-10-2013 20:35:36
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 9
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 26.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
OAc
SO2Ph
3u
PPM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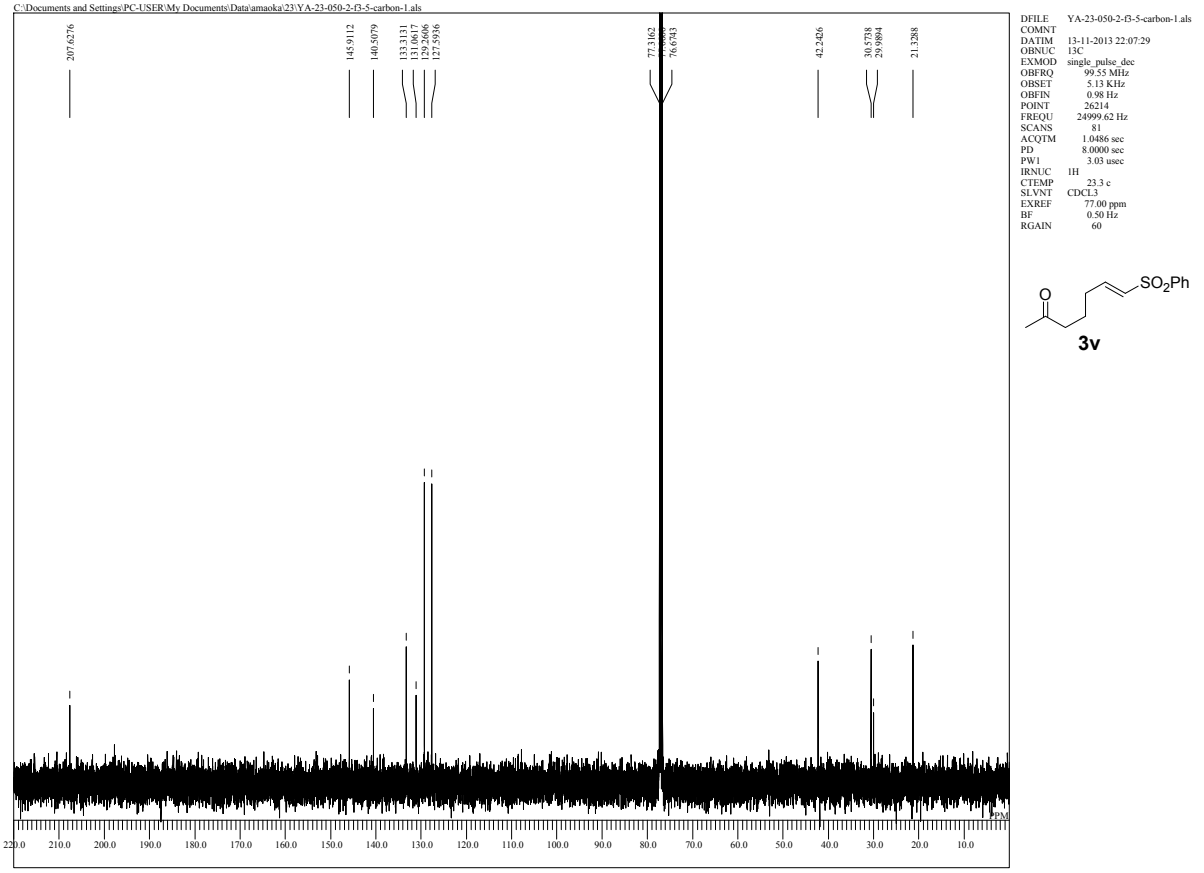
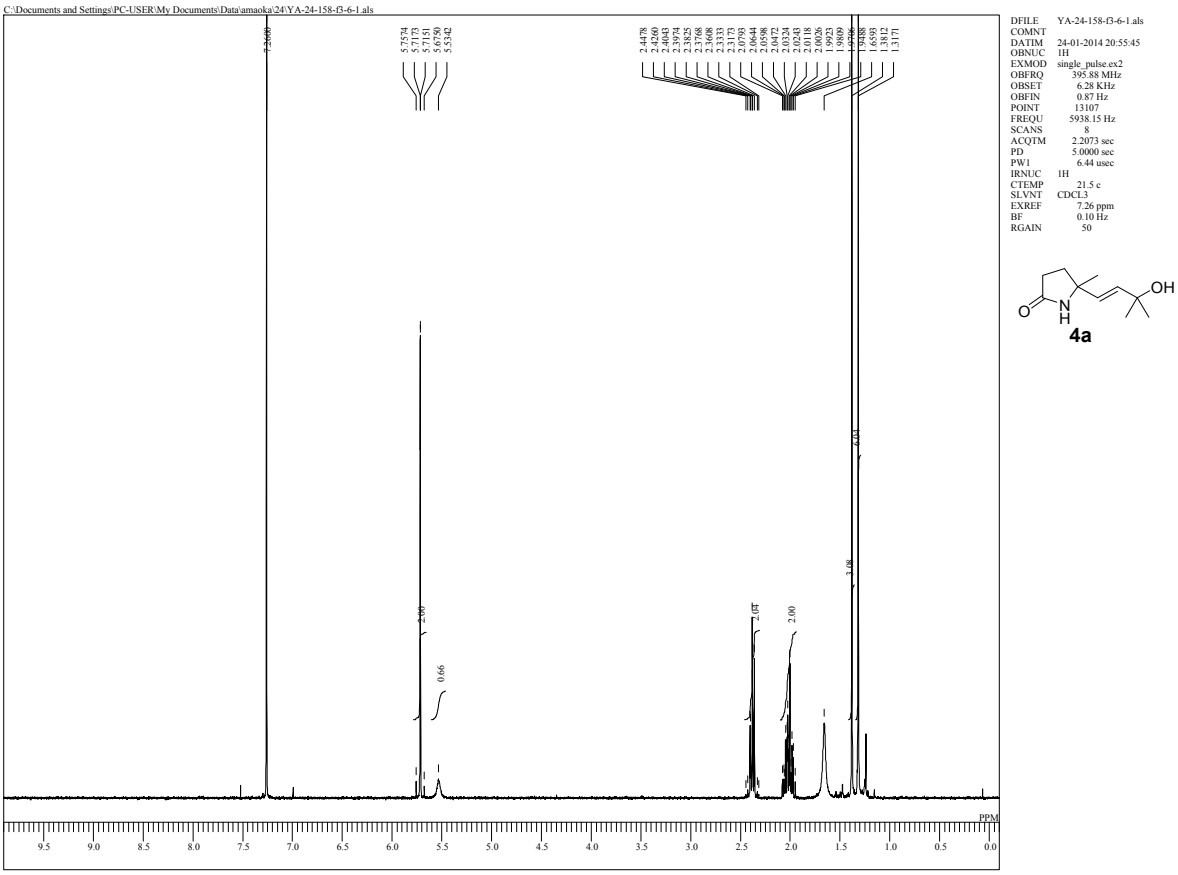
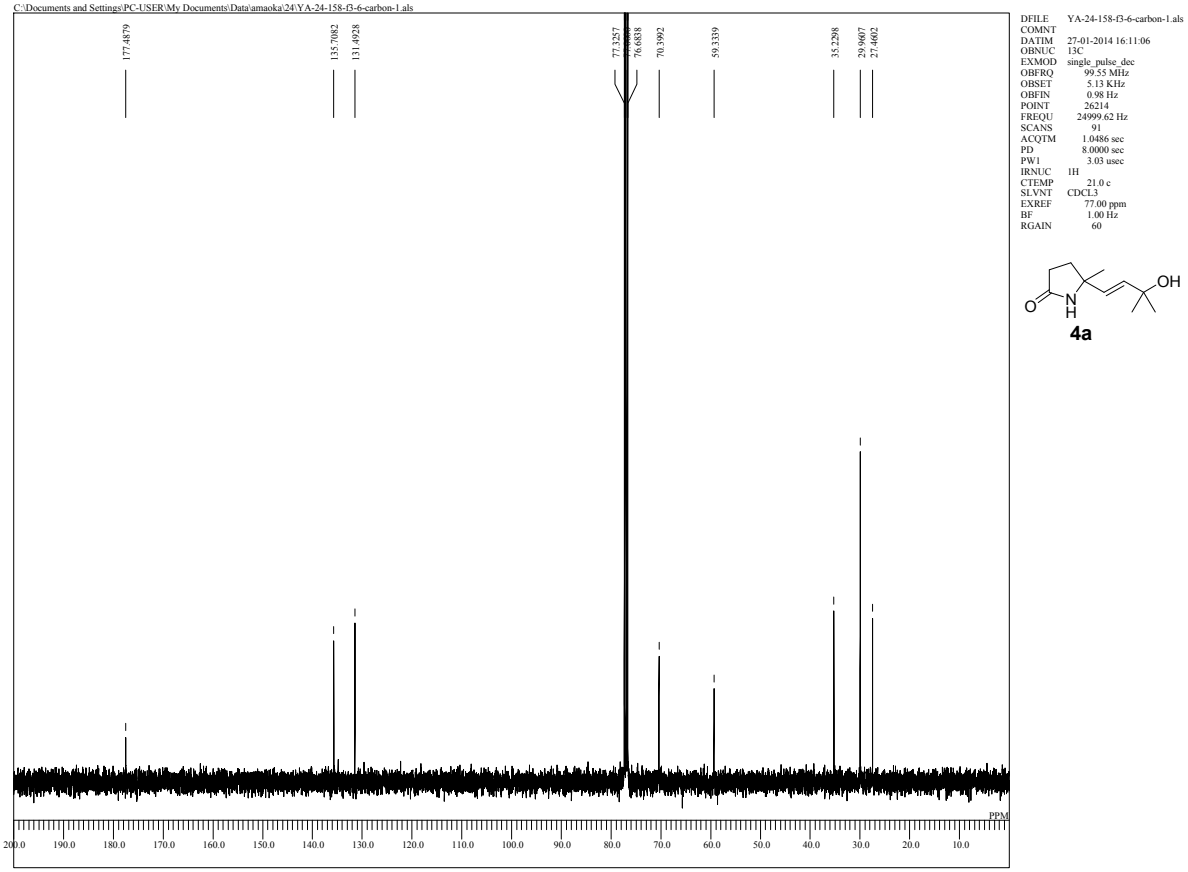
DFILE YA-23-050-2-f3-5-1.als
COMNT
DATIM 07-11-2013 16:26:59
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 16
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 50
SO2Ph
O
3v
PPM


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\23\YA-23-050-2-f3-5-carbon-1.als
DFILE YA-23-050-2-f3-5-carbon-1.als
COMNT
DATIM 13-11-2013 22:07:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 81
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.50 Hz
RGAIN 60
SO2Ph
O
3v
PPM



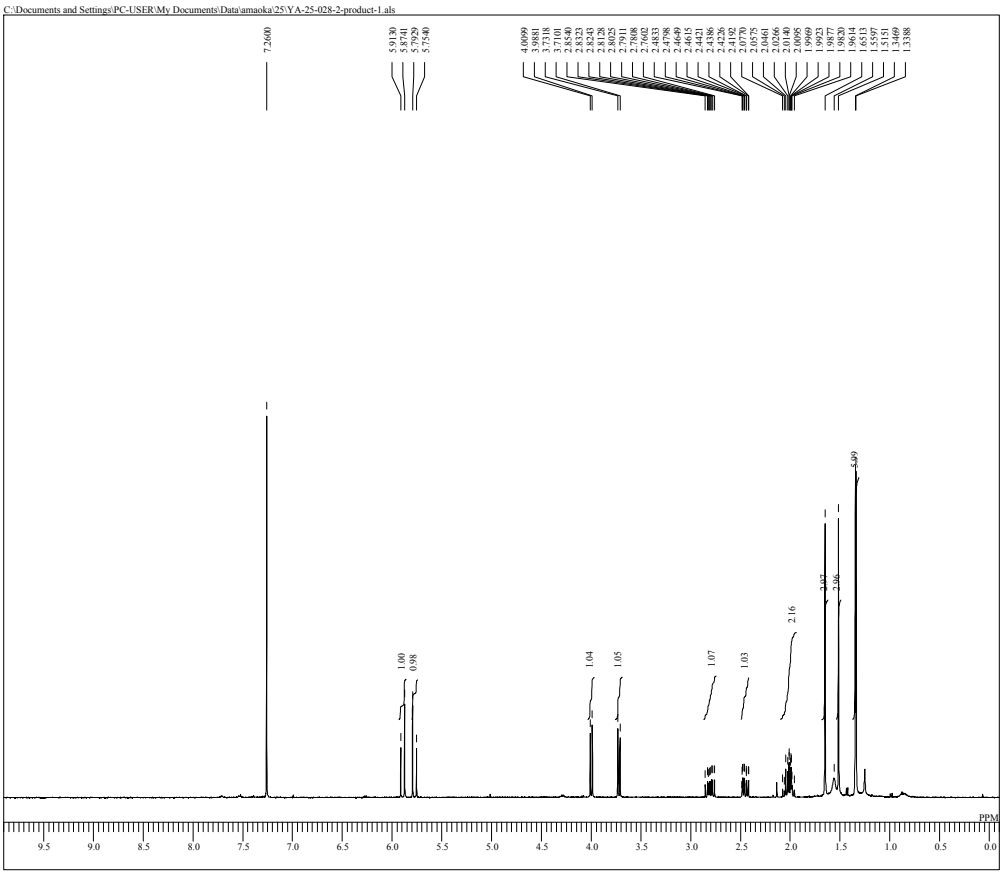
DFILE YA-24-158-f3-6-1.als
COMNT
DATIM 24-01-2014 20:55:45
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 50

4a

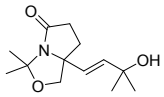


DFILE YA-24-158-f3-6-carbon-1.als
COMNT
DATIM 27-01-2014 16:11:06
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 91
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60

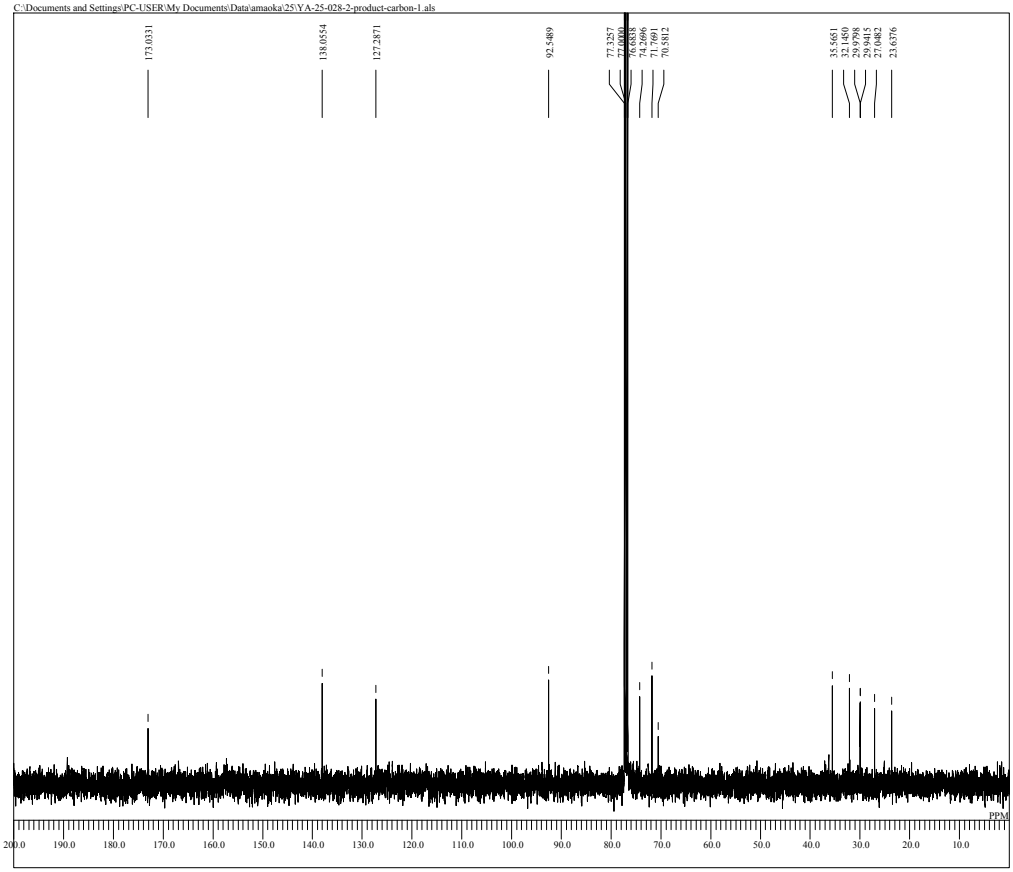
4a



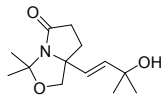
DFILE YA-25-028-2-product-1.als
COMNT
DATIM 20-02-2014 20:09:20
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 46



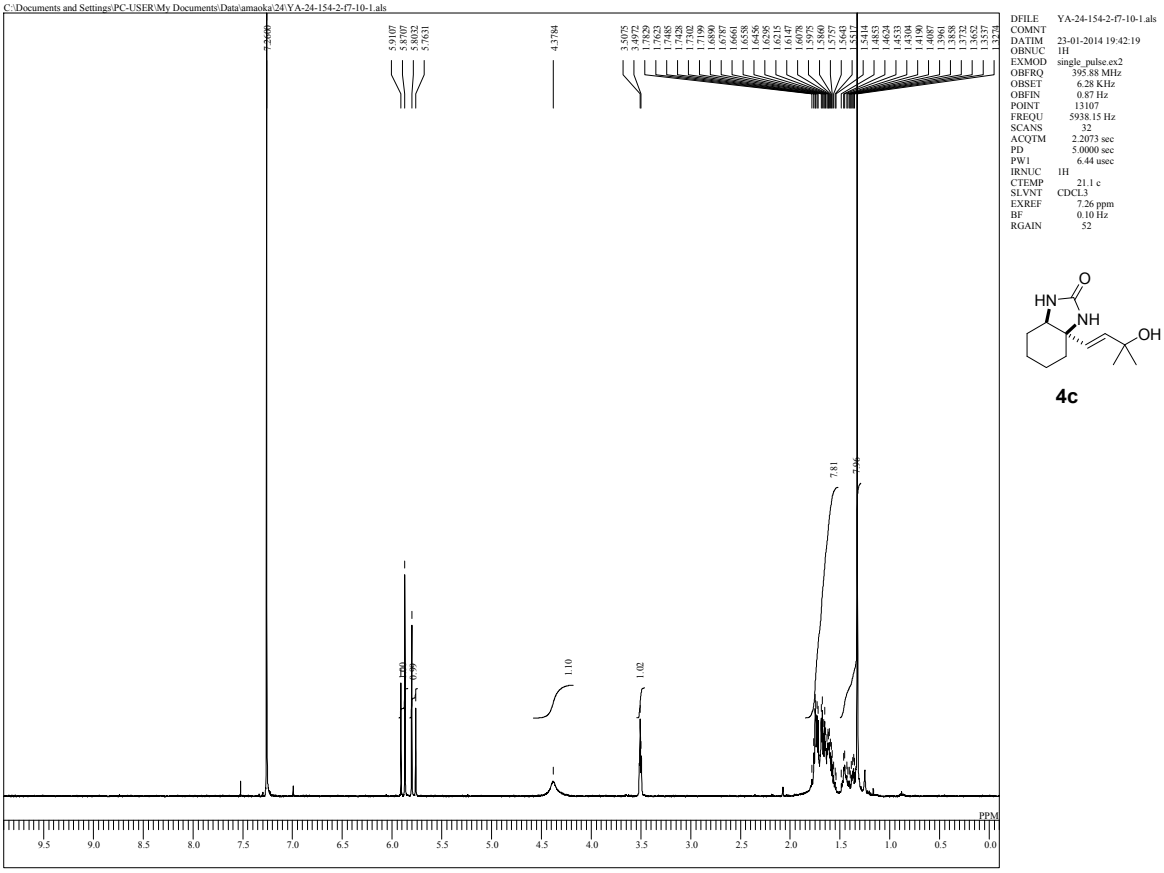
4b

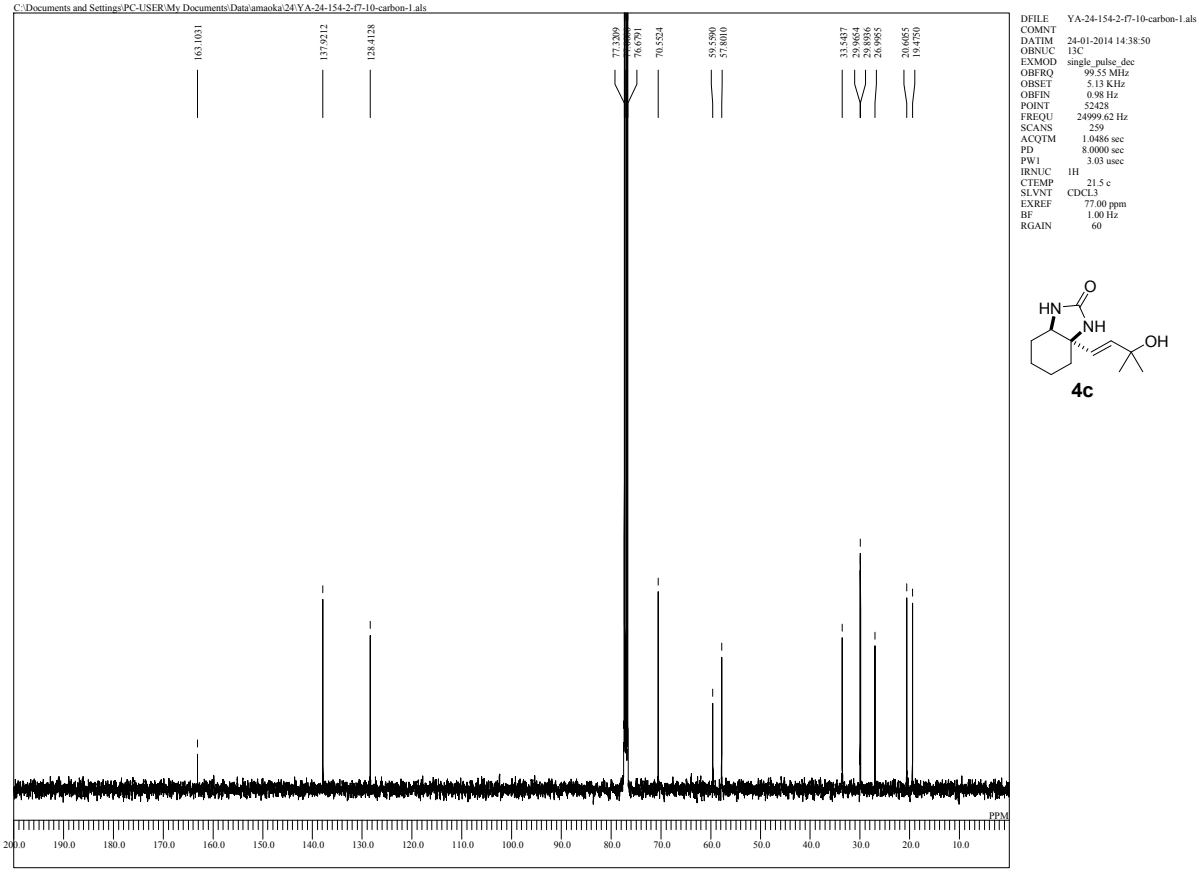


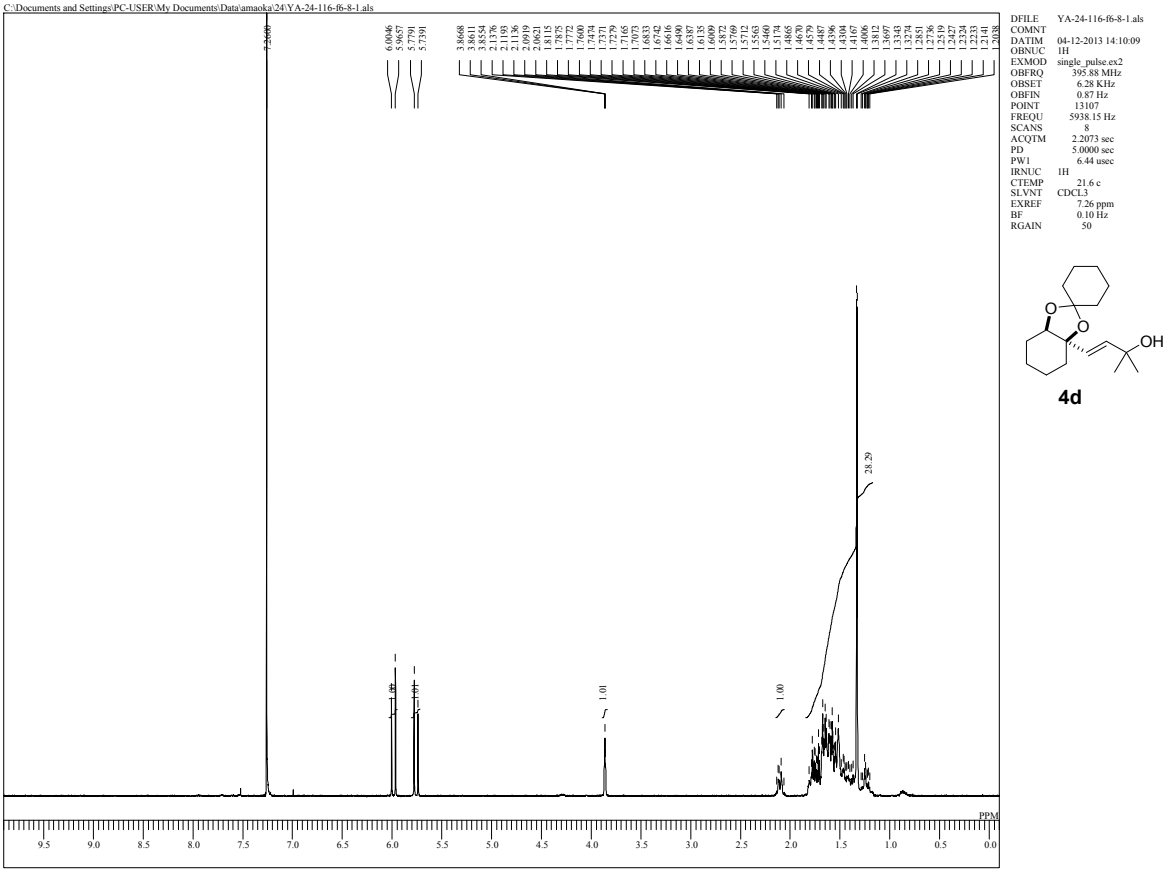
DFILE YA-25-028-2-product-carbon-1.als
COMNT
DATIM 21-02-2014 10:58:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 91
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60

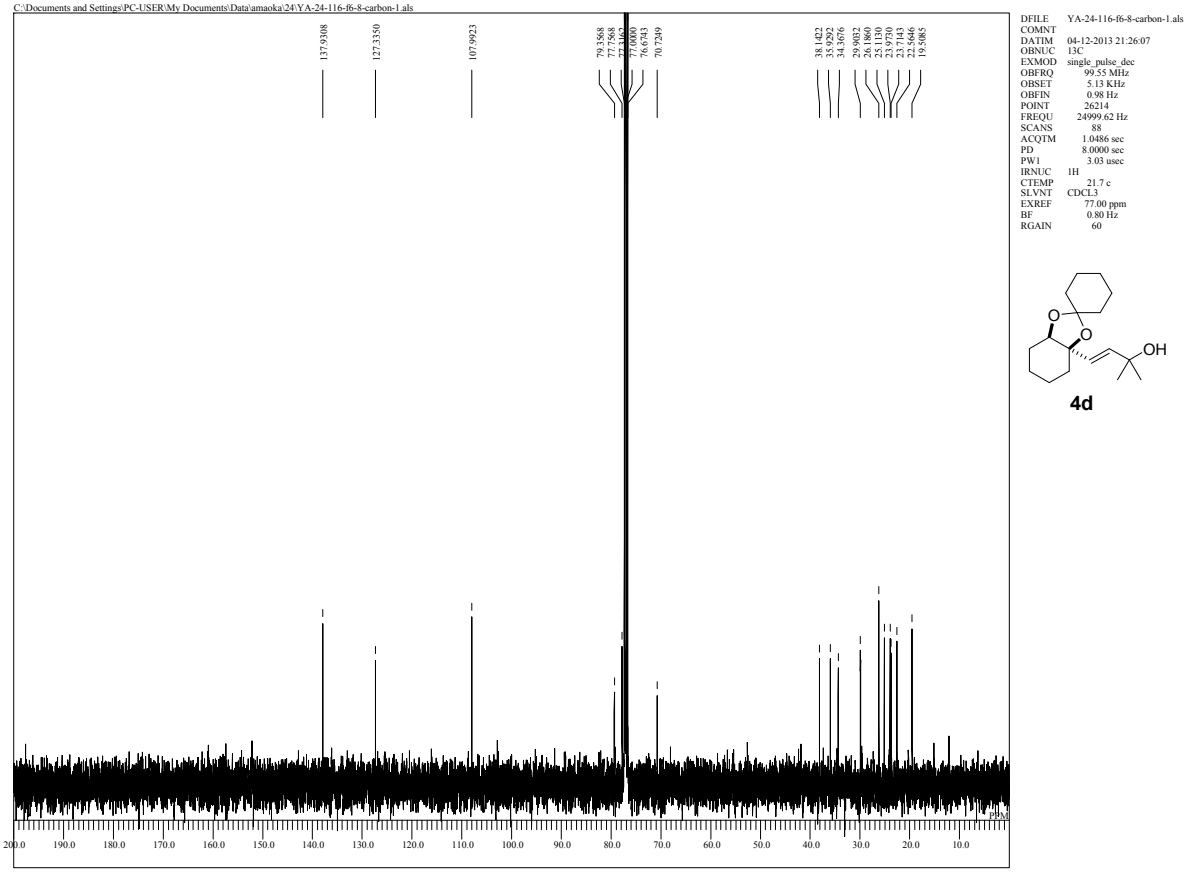


4b

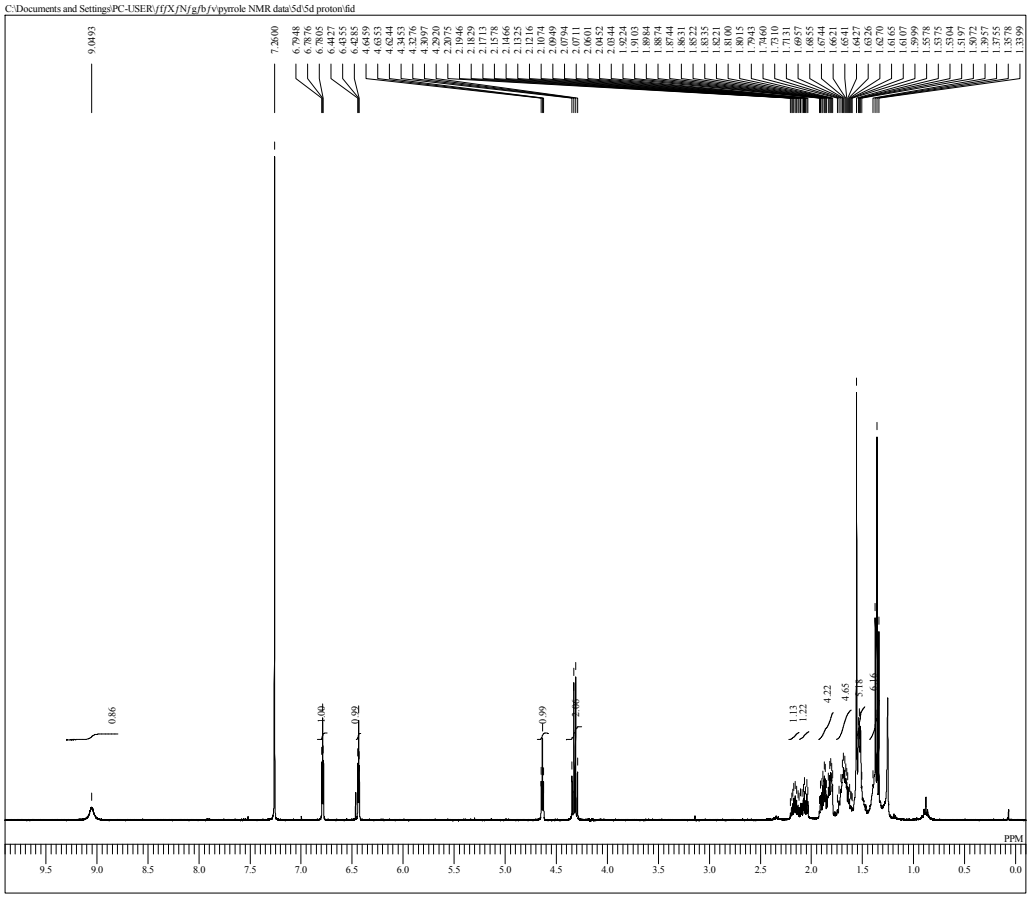

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-154-2-f7-10-1.als
7.2660
5.9107
5.8707
5.8032
5.7631
4.3784
3.5075
3.4972
1.7829
1.7623
1.7485
1.7338
1.7192
1.7119
1.6890
1.6787
1.6641
1.6558
1.6436
1.6295
1.6147
1.6078
1.5975
1.5860
1.5757
1.5643
1.5317
1.5414
1.4853
1.4624
1.4533
1.4304
1.4190
1.4067
1.3961
1.3858
1.3732
1.3572
1.3274
7.81
7.66
1.10
1.02
1.00
0.99
PPM
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
DFILE YA-24-154-2-f7-10-1.als
COMNT
DATIM 23-01-2014 19:42:19
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 32
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 52
HN
O
NH
OH
4c


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-154-2-f7-10-carbon-1.als
163.1031
137.9212
128.4128
77.3209
76.6791
70.5524
59.5590
57.8010
33.5437
29.9954
29.8936
26.9955
20.6055
19.4750
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
DFILE YA-24-154-2-f7-10-carbon-1.als
COMNT
DATIM 24-01-2014 14:38:50
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 52428
FREQU 24999.62 Hz
SCANS 259
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 60
HN
O
NH
OH
4c

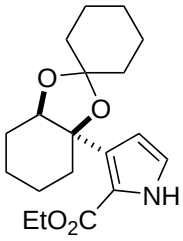

C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-116-f6-8-1.als
DFILE YA-24-116-f6-8-1.als
COMNT
DATIM 04-12-2013 14:10:09
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
POINT 13107
FREQU 5938.15 Hz
SCANS 8
ACQTM 2.2073 sec
PD 5.0000 sec
PW1 6.44 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 50
7.2660
6.0046
5.9957
5.7991
5.7391
1.00
1.01
1.01
1.00
28.29
PPM
4d


C:\Documents and Settings\PC-USER\My Documents\Data\amaoka\24\YA-24-116-f6-8-carbon-1.als
DFILE YA-24-116-f6-8-carbon-1.als
COMNT
DATIM 04-12-2013 21:26:07
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
POINT 26214
FREQU 24999.62 Hz
SCANS 88
ACQTM 1.0486 sec
PD 8.0000 sec
PW1 3.03 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.80 Hz
RGAIN 60
137.9308
127.3350
107.9923
79.3568
77.7568
77.0000
76.6743
70.7249
38.1422
35.5972
34.3476
29.9052
26.1860
25.1130
23.9730
23.1743
22.5646
19.5085
PPM
4d

MW04_11

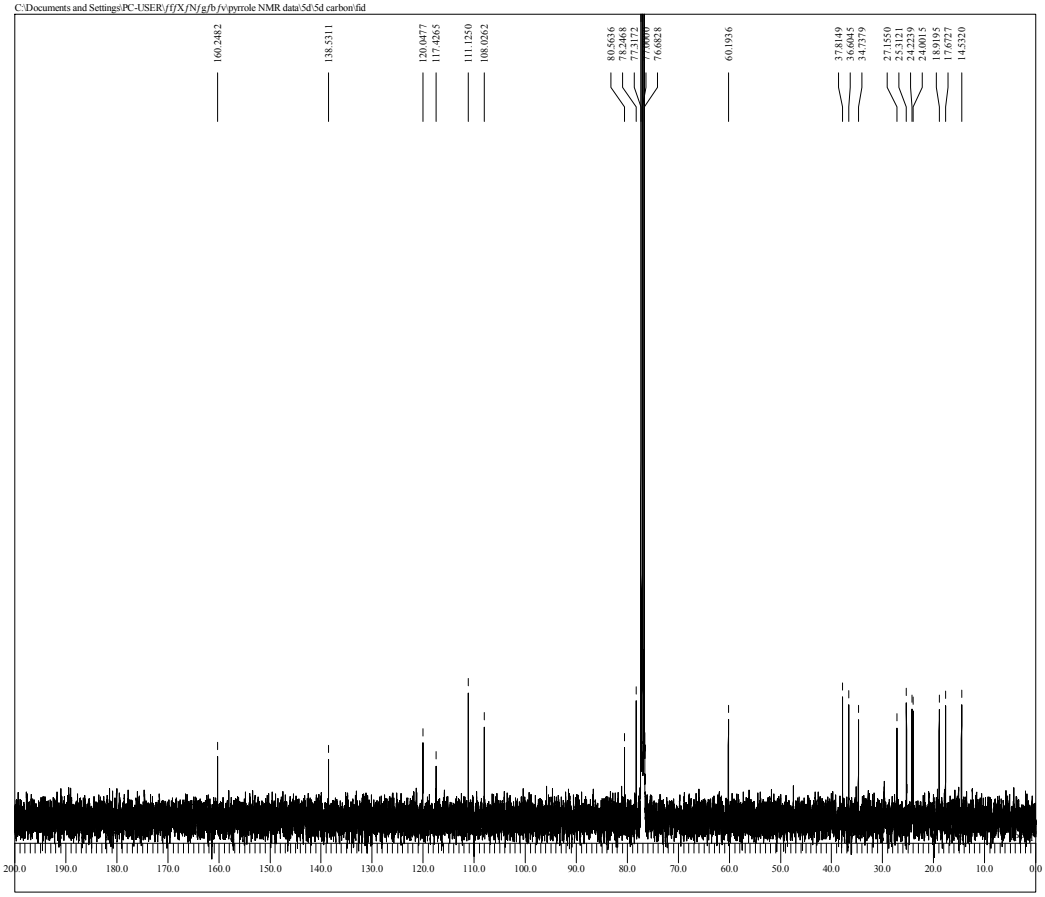


DFILE fid
COMNT MW04_11
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datase
OBNUC 1H
EXMOD zg30
OBFRQ 400.13 MHz
OBSET 2.47 KHz
OBFIN 0.97 Hz
POINT 131072
FREQU 8223.68 Hz
SCANS 16
ACQTM 3.9846 sec
PD 1.0000 sec
PW1 14.00 usec
IRNUC
CTEMP 24.4 c
SLVNT CDCl3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 203

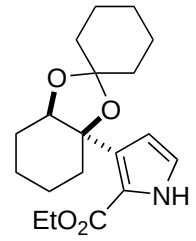


5d

MW04_11

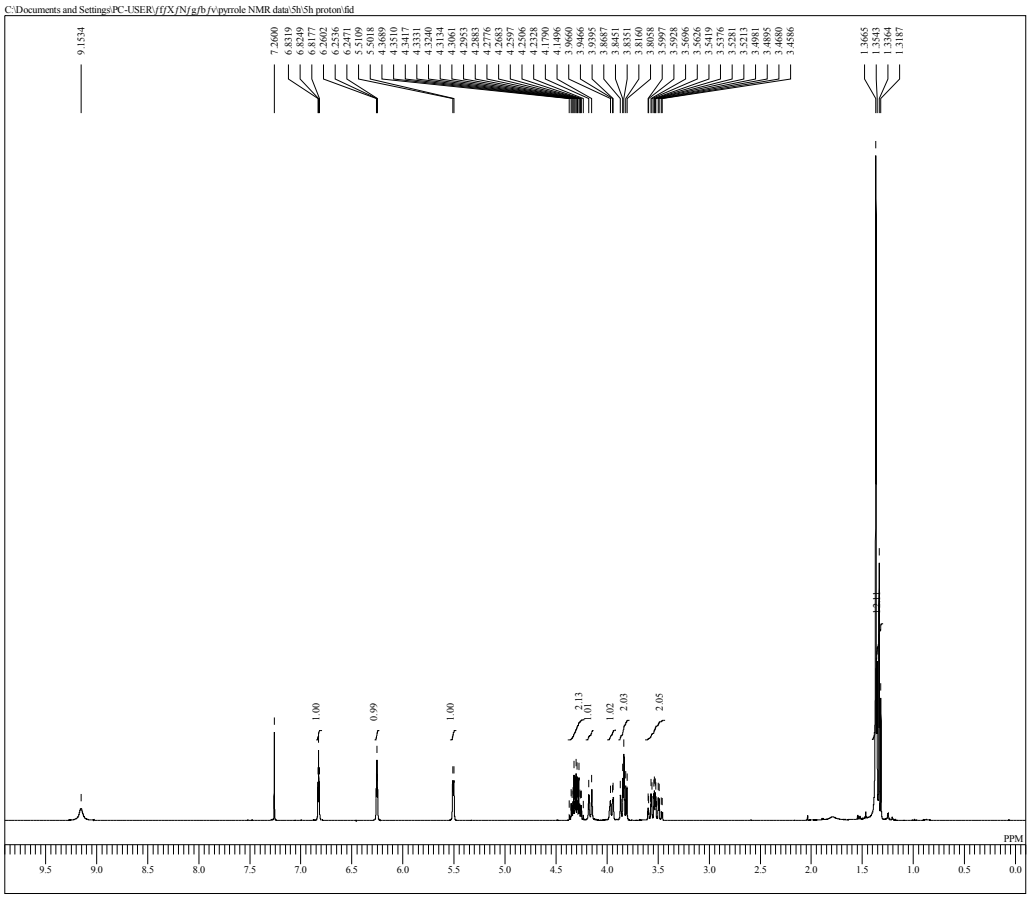


DFILE fid
COMNT MW04_11
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datase
OBNUC 13C
EXMOD zgpg30
OBFRQ 100.62 MHz
OBSET 3.83 KHz
OBFIN 6.40 Hz
POINT 131072
FREQU 24038.46 Hz
SCANS 483
ACQTM 1.3632 sec
PD 2.0000 sec
PW1 9.50 usec
IRNUC
CTEMP 26.8 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 203

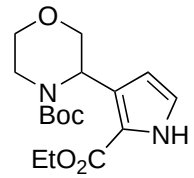


5d

KT10_18

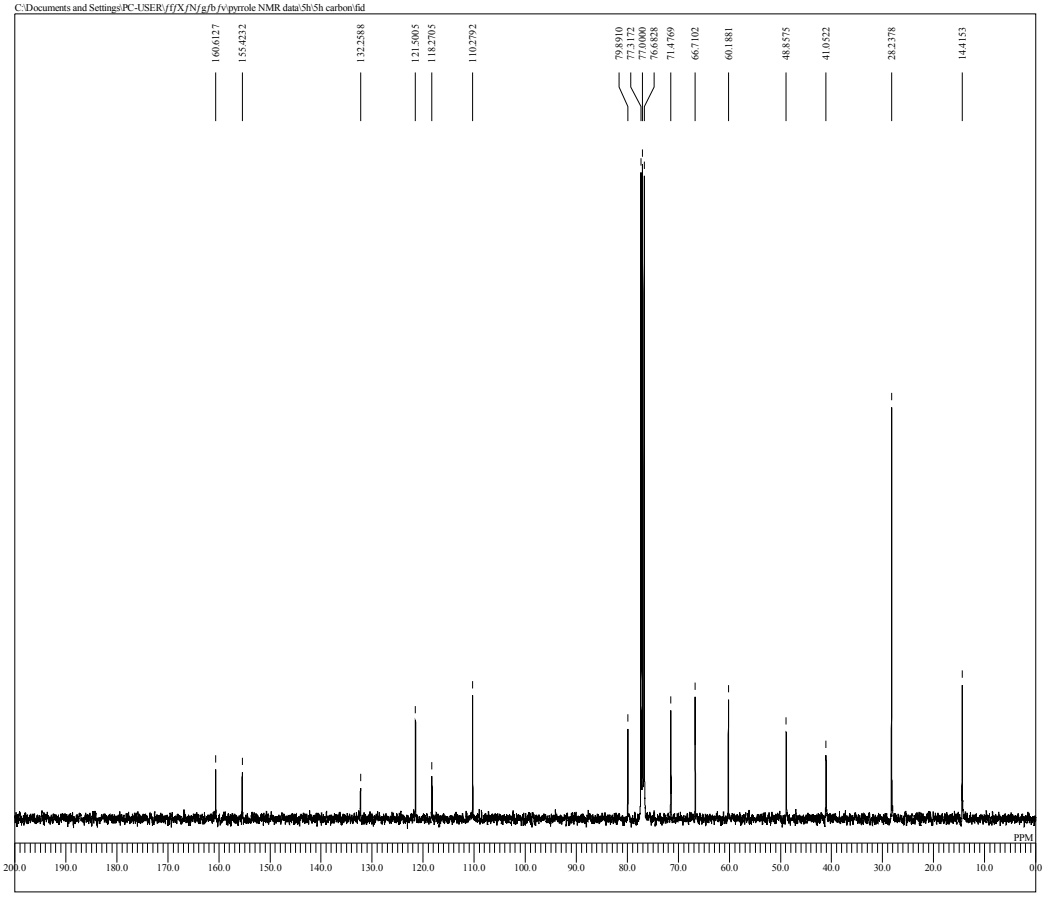


DFILE fid
COMNT KT10_18
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC 1H
EXMOD zg30
OBFRQ 400.13 MHz
OBSET 2.47 KHz
OBFIN 0.97 Hz
POINT 131072
FREQU 8223.68 Hz
SCANS 16
ACQTM 3.9846 sec
PD 1.0000 sec
PW1 14.00 usec
IRNUC
CTEMP 24.2 c
SLVNT CDCl3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 101

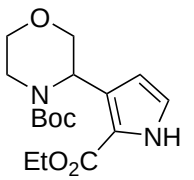


5h

KT10_18

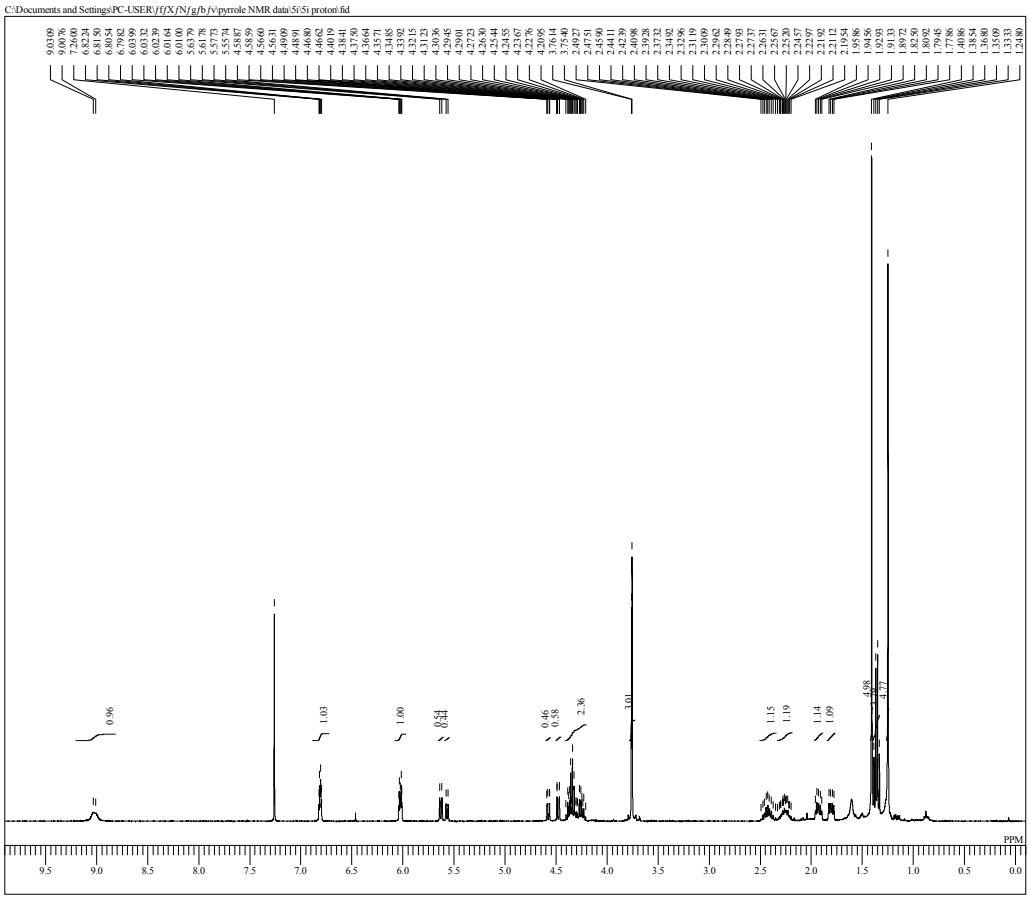


DFILE fid
COMNT KT10_18
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC 13C
EXMOD zgpg30
OBFRQ 100.62 MHz
OBSET 3.83 KHz
OBFIN 6.40 Hz
POINT 131072
FREQU 24038.46 Hz
SCANS 128
ACQTM 1.3632 sec
PD 2.0000 sec
PW1 9.50 usec
IRNUC
CTEMP 26.2 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 3.00 Hz
RGAIN 203

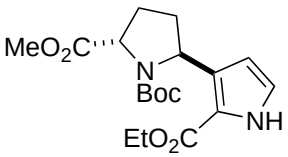


5h

KT10_22

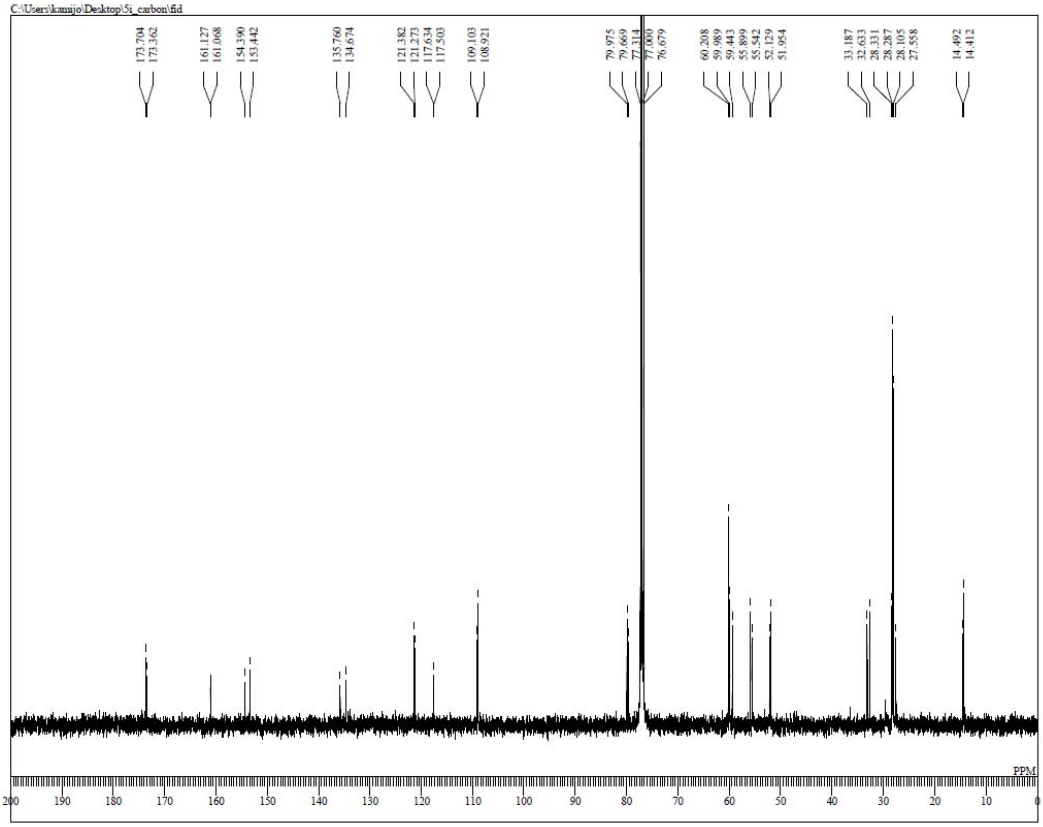


DFILE fid
COMNT KT10_22
DATIM 3.2/prog/mod/dataserver /opt/topspin3.2/prog/mod/dataserver AGLlo
OBNUC 1H
EXMOD zg30
OBFRQ 400.13 MHz
OBSET 2.47 KHz
OBFIN 0.97 Hz
POINT 131072
FREQU 8223.68 Hz
SCANS 16
ACQTM 3.9846 sec
PD 1.0000 sec
PW1 14.00 usec
IRNUC
CTEMP 24.0 c
SLVNT CDCl3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 203

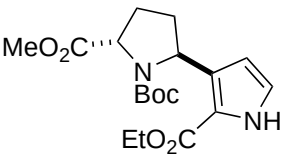


5i

KT10_22

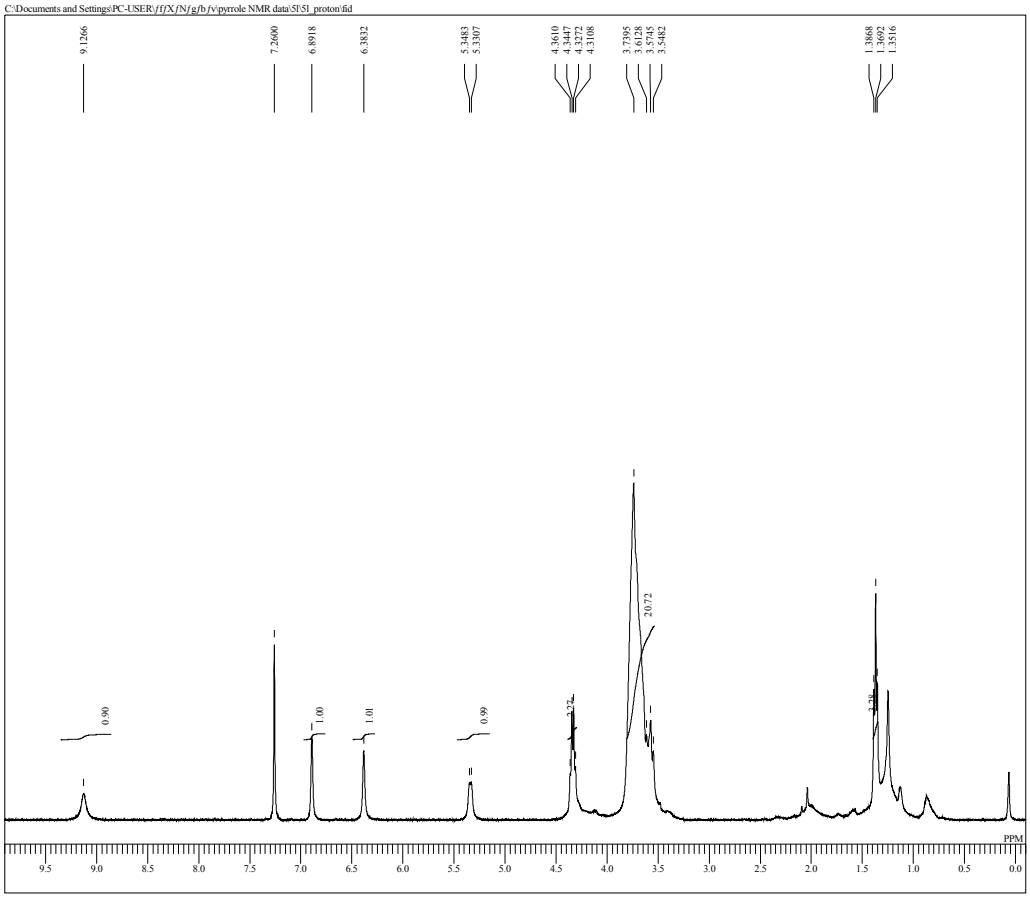


DFILE fid
COMNT KT10_22
DATIM 2014-06-03 12:29:52
OBNUC 13C
EXMOD zgpg30
OBFRQ 100.62 MHz
OBSET 3.83 KHz
OBFIN 6.40 Hz
POINT 32768
FREQU 24038.46 Hz
SCANS 1280
ACQTM 1.3632 sec
PD 2.0000 sec
PW1 9.50 usec
IRNUC
CTEMP 27.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 203

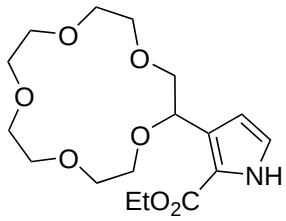


5i

MW 3-33 colum3 F46-58

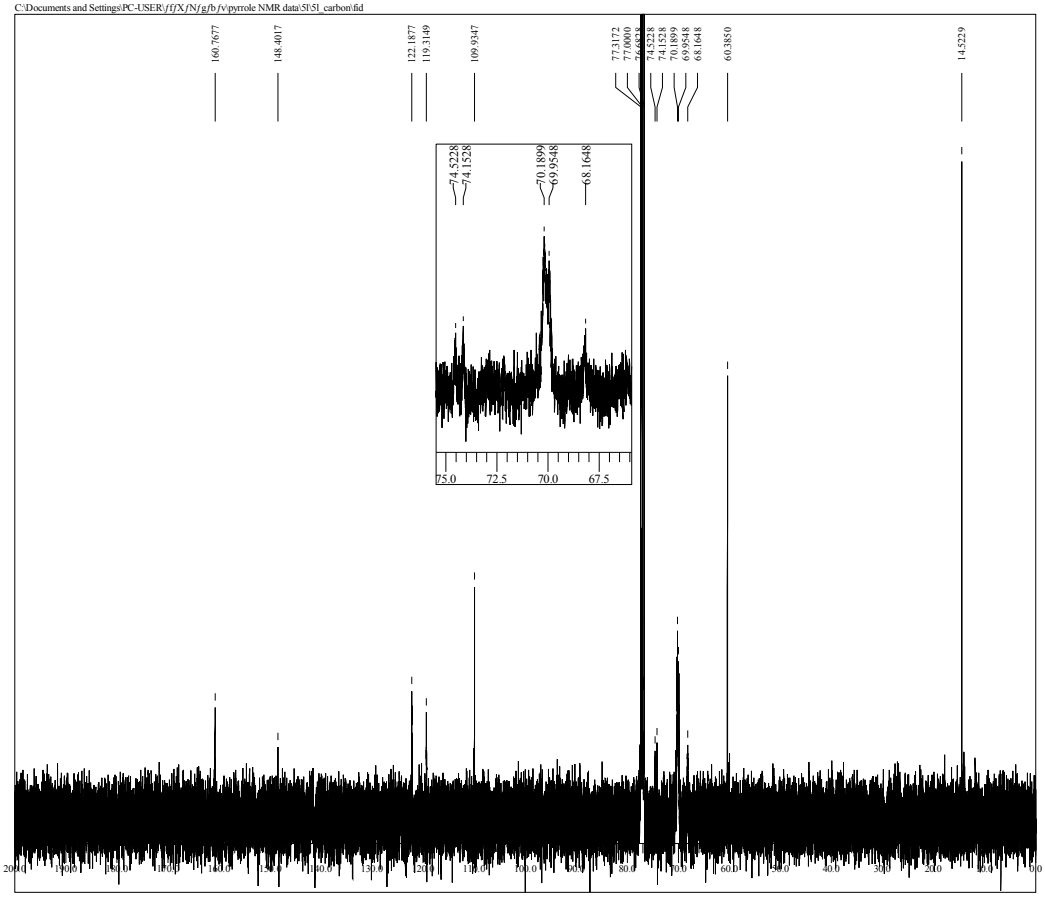


DFILE fid
COMNT MW 3-33 colum3 F46-58
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC 1H
EXMOD zg30
OBFRQ 400.13 MHz
OBSET 2.47 KHz
OBFIN 0.97 Hz
POINT 32768
FREQU 8223.68 Hz
SCANS 16
ACQTM 3.9846 sec
PD 1.0000 sec
PW1 14.00 usec
IRNUC
CTEMP 23.3 c
SLVNT CDCl3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 203

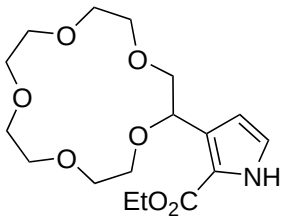


5l

MW 3-33 colum3 F46-58 13C

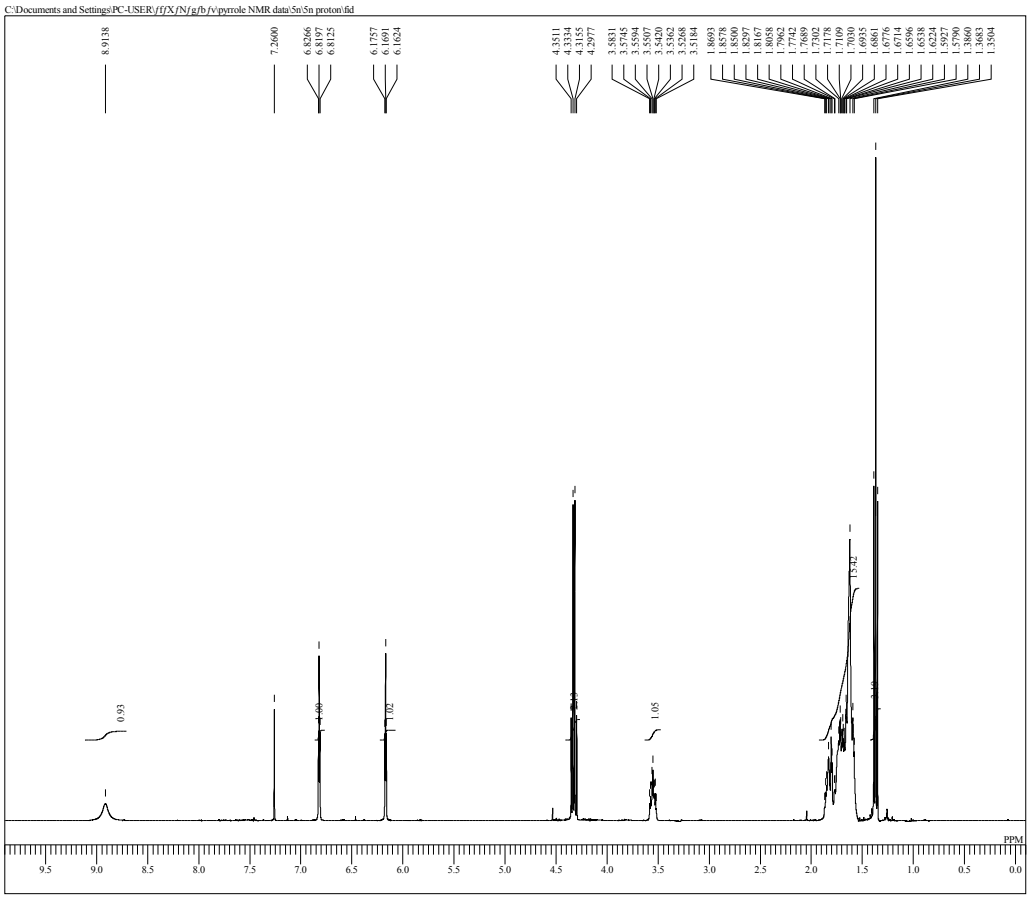


DFILE fid
COMNT MW 3-33 colum3 F46-58 13C
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC 13C
EXMOD zgpg30
OBFRQ 100.62 MHz
OBSET 2.82 KHz
OBFIN 9.80 Hz
POINT 131072
FREQU 24038.46 Hz
SCANS 5053
ACQTM 1.3632 sec
PD 2.0000 sec
PW1 9.50 usec
IRNUC
CTEMP 25.4 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.30 Hz
RGAIN 203

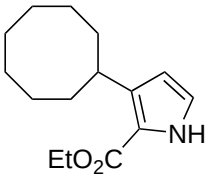


5l

KT10_16

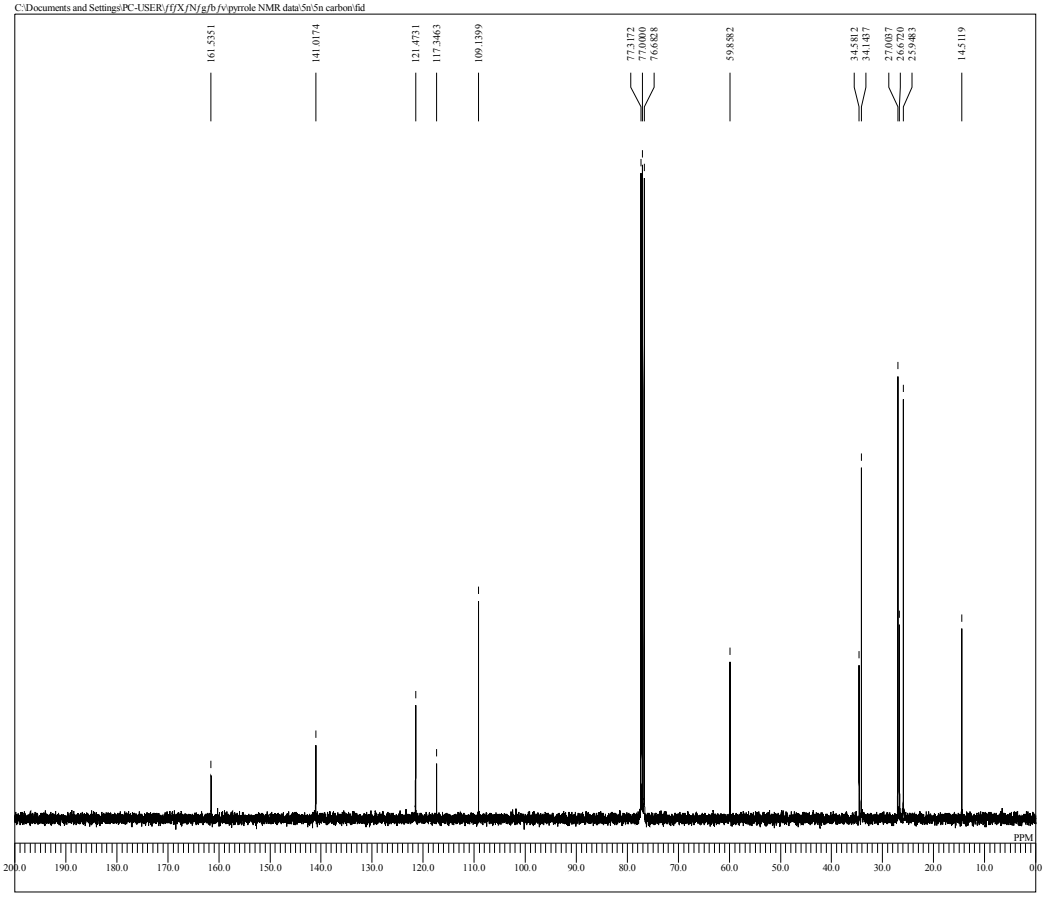


DFILE fid
COMNT KT10_16
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC 1H
EXMOD zg30
OBFRQ 400.13 MHz
OBSET 2.47 KHz
OBFIN 0.97 Hz
POINT 131072
FREQU 8223.68 Hz
SCANS 16
ACQTM 3.9846 sec
PD 1.0000 sec
PW1 14.00 usec
IRNUC
CTEMP 24.5 c
SLVNT CDCl3
EXREF 7.26 ppm
BF 0.10 Hz
RGAIN 101

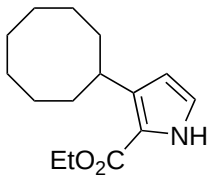


5n

KT10_16

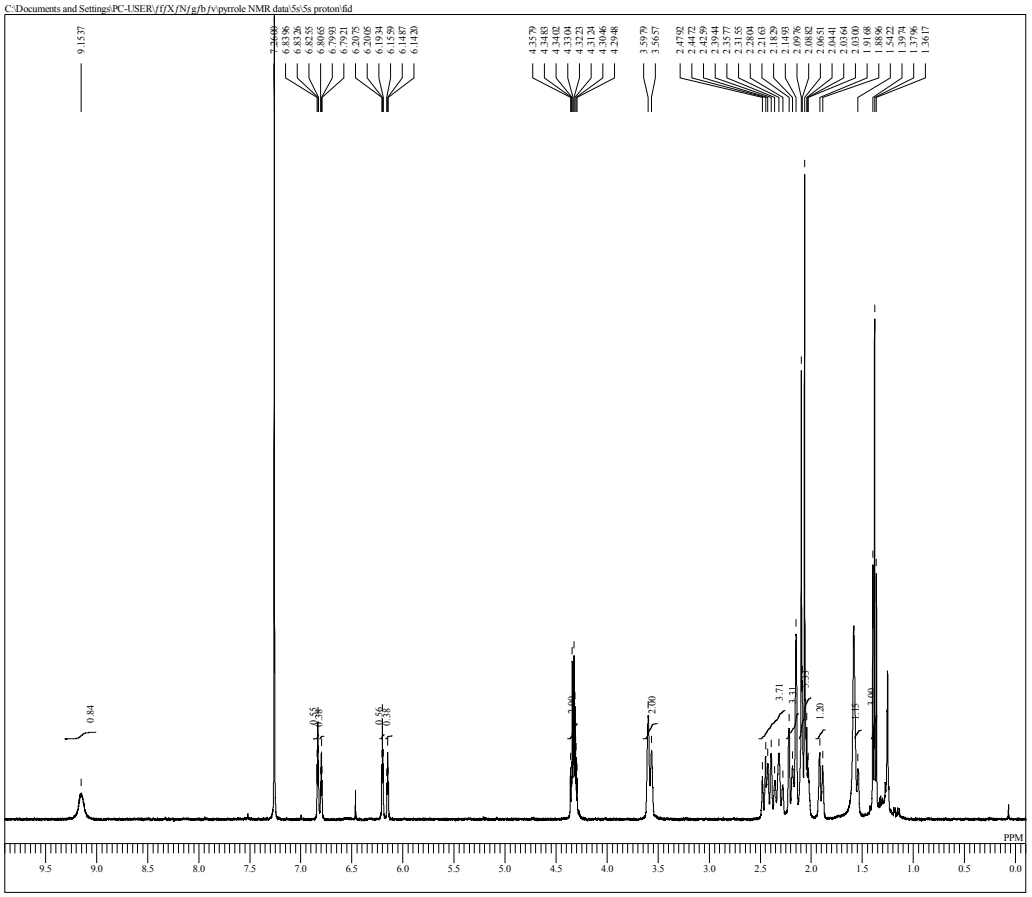


DFILE fid
COMNT KT10_16
DATIM PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC 13C
EXMOD zgpg30
OBFRQ 100.62 MHz
OBSET 3.83 KHz
OBFIN 6.40 Hz
POINT 131072
FREQU 24038.46 Hz
SCANS 170
ACQTM 1.3631 sec
PD 2.0000 sec
PW1 9.50 usec
IRNUC
CTEMP 26.7 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 203

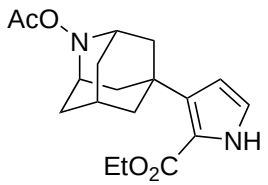


5n

KT10_29

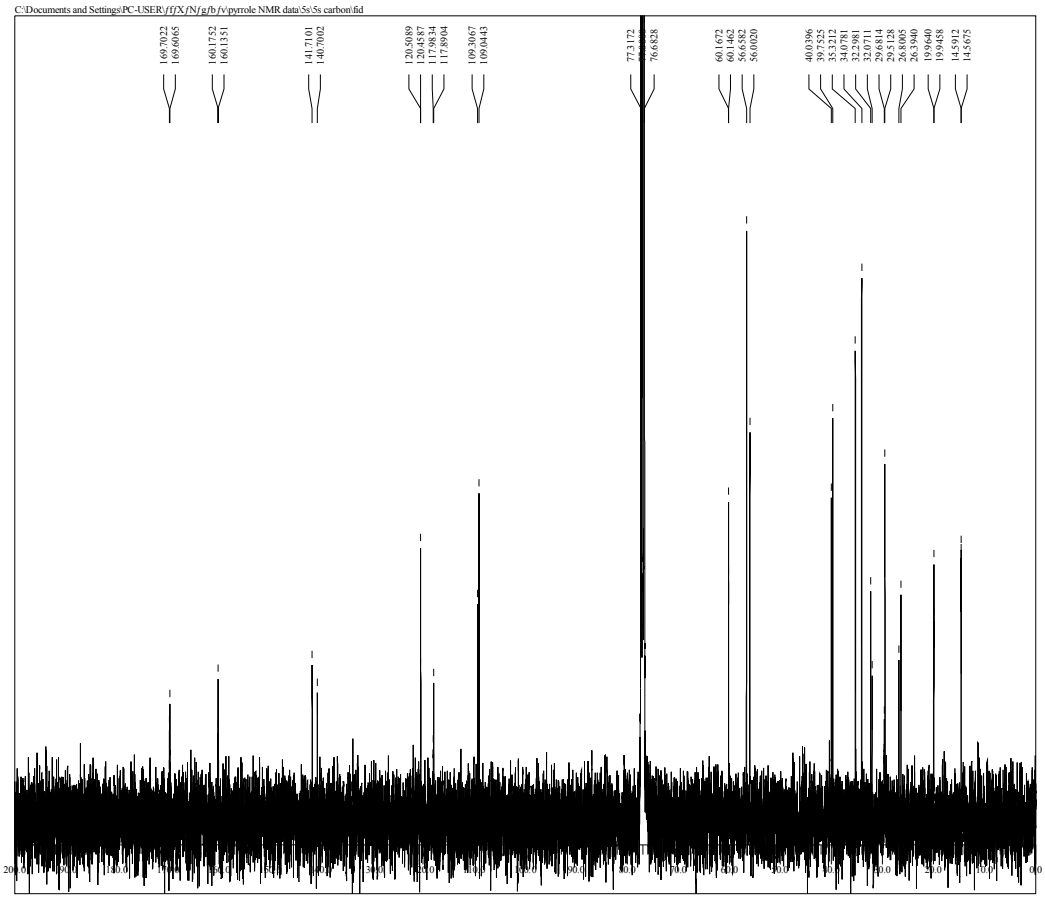



```
DFILE   fid
COMNT   KT10_29
DATIM   PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC   1H
EXMOD   zg30
OBFRQ     400.13 MHz
OBSET       2.47 KHz
OBFIN       0.97 Hz
POINT     131072
FREQU    8223.68 Hz
SCANS         16
ACQTM     3.9846 sec
PD        1.0000 sec
PW1         14.00 usec
IRNUC
CTEMP       24.0 c
SLVNT   CDCl3
EXREF       7.26 ppm
BF          0.10 Hz
RGAIN        203
```

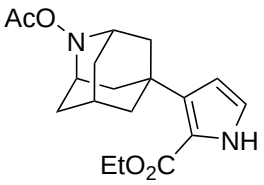


5s

KT10_29




```
DFILE   fid
COMNT   KT10_29
DATIM   PSPIN\prog\mod\dataserver.exe C:\Bruker\TOPSPIN\prog\mod\datas
OBNUC   13C
EXMOD   zgpg30
OBFRQ     100.62 MHz
OBSET       3.83 KHz
OBFIN       6.40 Hz
POINT     262144
FREQU   24038.46 Hz
SCANS       1338
ACQTM     1.3632 sec
PD        2.0000 sec
PW1         9.50 usec
IRNUC
CTEMP       26.5 c
SLVNT   CDCl3
EXREF       77.00 ppm
BF          1.00 Hz
RGAIN        203
```



5s