N-Heterocyclic Carbene-Promoted [3+2] Cycloaddition of Allenyl Sulfone and Arylidenemalononitriles

Satoru Kuwano,* Toshinobu Masuda, Koki Yamaguchi, and Takayoshi Arai

Supporting information

Contents

1. General S2

2. Preparation of arylidenemalononitriles S2

3. NHC-promoted [3+2] cyclization of allenyl sulfone and arylidenemalononitrile S5

4. NMR spectra S9

5. Single Crystal X-Ray Analysis of **4d** S23

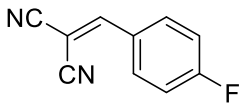
1. General

Dry solvents were purchased from commercial suppliers and used without further purification. Analytical thin-layer chromatography (TLC) was performed on glass plates coated with 0.25 mm 230-400 mesh silica gel containing a fluorescent indicator (Merck, #1.05715.0009). Silica gel column chromatography was performed on Kanto silica gel 60 (spherical, 100-210 μm). IR spectra were recorded on JASCO FT/IR-4100 using ATR. ^{1}H-NMR spectra were recorded on JEOL ECS-400 (400MHz) spectrometers. Chemical shifts of ^{1}H-NMR spectra were reported relative to tetramethyl silane (δ 0) or acetone–d_6 (δ 2.05). ^{13}C-NMR spectra were recorded on JEOL ECS-400 (100MHz) spectrometers. Chemical shifts of ^{13}C-NMR spectra were reported relative to $CDCl_3$ (δ 77.0) or acetone–d_6 (δ 29.84). Splitting patterns were reported as s, singlet; d, doublet; t, triplet; m, multiplet.

2. Preparation of arylidenemalononitriles

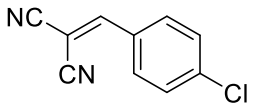
Arylidenemalononitriles **3b**–**n** were prepared through Knoevenagel condensation of the corresponding aldehydes with malononitrile and recrystallized from EtOH and hexane, according to the literature.[1]

2-(4-Fluorobenzylidene)malononitrile (3b)



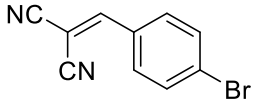
Pale pink solids; R_f=0.50 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.98-7.95 (m, 2H), 7.74 (s, 1H), 7.26-7.21 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 160.1 (d, $^{1}J_{CF}$ = 264.1 Hz), 158.3, 133.4 (d, $^{3}J_{CF}$ = 9.5 Hz), 127.3, 117.1 (d, $^{2}J_{CF}$ = 21.9 Hz), 113.5, 112.5, 82.3; HRMS calcd for $C_{10}H_4FN_2$ [M－H]$^+$: 171.0359, found: *m/z* 171.0358; IR (neat) 2231, 1595, 1575, 1506, 1416, 1382, 1306, 1242, 1163 cm^{-1}.

2-(4-Chlorobenzylidene)malononitrile (3c)



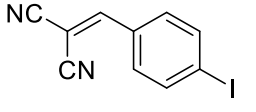
White solids; R_f=0.53 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.5 Hz, 1H), 7.73 (s, 1H), 7.52 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.3, 141.1, 131.8, 130.1, 129.2, 113.4, 112.3, 83.3; HRMS calcd for $C_{10}H_4ClN_2$ [M－H]$^+$: 187.0063, found: *m/z* 187.0065; IR (neat) 2227, 1587, 1276, 1103, 1088 cm^{-1}.

2-(4-Bromobenzylidene)malononitrile (3d)



White solids; R_f=0.53 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.9 Hz, 1H), 7.72 (s, 1H), 7.69 (d, *J* = 8.9 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.4, 133.0, 131.8, 129.9, 129.6, 113.4, 112.3, 83.4; HRMS calcd for $C_{10}H_4BrN_2$ [M－H]$^+$: 230.9558, found: *m/z* 230.9565; IR (neat) 2229, 1578, 1276, 1259 cm^{-1}.

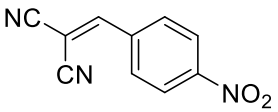
2-(4-Iodobenzylidene)malononitrile (3e)



White solids; R_f=0.53 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.5 Hz, 1H), 7.70 (s, 1H), 7.60 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.7, 139.0, 131.4, 130.0, 113.4, 112.3, 102.8, 83.4; HRMS calcd for $C_{10}H_4IN_2$ [M－H]$^+$: 278.9419, found: *m/z* 278.9430; IR (neat) 2225, 1576, 1397, 1275 cm^{-1}.

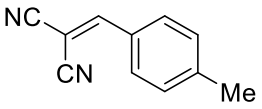
[1] J. S. Yadav, B.V.S. Reddy, A.K. Basak, B. Visali, A.V. Narsaiah, K. Nagaiah, *Eur. J. Org. Chem.* **2004**, *546*.

2-(4-Nitrobenzylidene)malononitrile (3f)



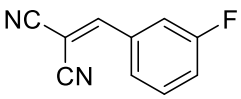
Pale brown solids; R_f=0.47 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.41-8.38 (m, 2H), 8.10-8.06 (m, 2H), 7.88 (s, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 156.9, 150.3, 135.8, 131.3, 124.6, 112.6, 111.6, 87.4; HRMS calcd for $C_{10}H_4N_3O_2$ [M－H]$^+$: 198.0304, found: *m/z* 198.0305; IR (neat) 2230, 1579, 1521, 1343, 1213 cm^{-1}.

2-(4-Methylbenzylidene)malononitrile (3g)



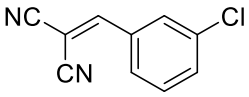
Gray solids; R_f=0.53 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.2 Hz, 1H), 7.72 (s, 1H), 7.84 (d, *J* = 8.2 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 159.8, 146.4, 130.9, 130.3, 128.4, 114.0, 112.8, 81.1, 22.0; HRMS calcd for $C_{11}H_7N_2$ [M－H]$^+$: 167.0609, found: *m/z* 167.0609; IR (neat) 2224, 1605, 1587, 1509, 1412, 1375, 1302, 1221, 1191 cm^{-1}.

2-(3-Fluorobenzylidene)malononitrile (3h)



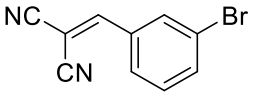
White solids; R_f=0.50 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.69-7.62 (m, 2H), 7.57-7.52 (m, 1H), 7.37-7.30 (m, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 162.7 (d, $^1J_{CF}$ = 253.7 Hz), 158.3, 132.5 (d, $^3J_{CF}$ = 8.6 Hz), 131.4 (d, $^3J_{CF}$ = 7.6 Hz), 126.7, 121.6 (d, $^2J_{CF}$ = 21.0 Hz), 116.8 (d, $^2J_{CF}$ = 22.8 Hz), 113.2, 112.0, 84.6; HRMS calcd for $C_{10}H_4FN_2$ [M－H]$^+$: 171.0359, found: *m/z* 171.0358; IR (neat) 2229, 1597, 1576, 1492, 1432, 1301, 1276, 1263, 1156 cm^{-1}.

2-(3-Chlorobenzylidene)malononitrile (3i)



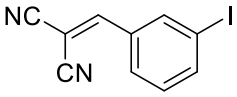
White solids; R_f=0.53 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.85-7.83 (m, 2H), 7.73 (s, 1H), 7.62-7.59 (m, 1H), 7.52-7.48 (m, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.2, 135.8, 134.3, 132.3, 130.9, 130.4, 128.3, 113.2, 112.0, 84.6; HRMS calcd for $C_{10}H_4ClN_2$ [M－H]$^+$: 187.0063, found: *m/z* 187.0066; IR (neat) 2230, 1591, 1559, 1478, 1417, 1366, 1293, 1266, 1215, 1098, 1080 cm^{-1}.

2-(3-Bromobenzylidene)malononitrile (3j)



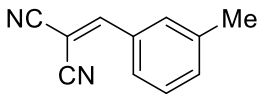
Pale gray solids; R_f=0.50 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.93-7.89 (m, 2H), 7.77-7.75 (m, 1H), 7.71 (s, 1H), 7.46-7.42 (m, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.1, 137.2, 133.4, 132.5, 131.1, 128.6, 123.6, 113.1, 112.0, 84.6; HRMS calcd for $C_{10}H_4BrN_2$ [M－H]$^+$: 230.9558, found: *m/z* 230.9564; IR (neat) 2229, 1557, 1476, 1415, 1293, 1267, 1212 cm^{-1}.

2-(3-Iodobenzylidene)malononitrile (3k)



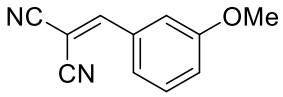
Pale yellow solids; R_f=0.56 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.14-8.13 (m, 1H), 7.97-7.94 (m, 2H), 7.68 (s, 1H), 7.31-7.27 (m, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.0, 143.1, 139.4, 132.6, 131.0, 129.0, 113.2, 112.0, 94.8, 84.3; HRMS calcd for $C_{10}H_4IN_2$ [M－H]$^+$: 278.9419, found: *m/z* 278.9430; IR (neat) 2226, 1591, 1411, 1214 cm^{-1}.

2-(3-Methylbenzylidene)malononitrile (3l)



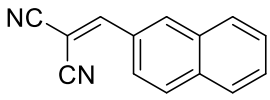
White solids; R_f=0.56 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.74-7.70 (m, 3H), 7.45-7.41 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 160.1, 139.6, 135.5, 131.2, 130.9, 129.5, 127.9, 113.8, 112.6, 82.4, 21.2; HRMS calcd for $C_{11}H_7N_2$ $[M-H]^+$: 167.0609, found: *m/z* 167.0609; IR (neat) 2226, 1574, 1303, 1267, 1251, 1180 cm^{-1}.

2-(3-Methoxybenzylidene)malononitrile (3m)



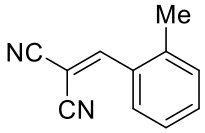
Pale yellow solids; R_f=0.50 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.50-7.41 (m, 3H), 7.19-7.16 (m, 1H), 3.87 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 160.0, 159.9, 132.0, 130.5, 123.9, 121.3, 114.0, 113.6, 112.6, 82.8, 55.5; HRMS calcd for $C_{11}H_7N_2O$ $[M-H]^+$: 183.0558, found: *m/z* 183.0560; IR (neat) 2229, 1595, 1496, 1466, 1270, 1248, 1163 cm^{-1}.

2-(Naphthalen-2-ylmethylene)malononitrile (3n)



Yellow solids; R_f=0.54 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.28 (s, 1H), 8.07 (dd, *J* = 1.6, 8.8 Hz, 1H), 7.98-7.91 (m, 3H), 7.89 (s, 1H), 7.71-7.59 (m, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 159.6, 135.7, 134.4, 132.4, 129.9, 129.56, 129.52, 128.4, 127.9, 127.6, 124.0, 113.9, 112.8, 82.0; HRMS calcd for $C_{14}H_7N_2$ $[M-H]^+$: 203.0609, found: *m/z* 203.0612; IR (neat) 2224, 1583, 1269, 1245 cm^{-1}.

2-(2-Methylbenzylidene)malononitrile (3o)



White solids; R_f=0.64 (hexane:EtOAc = 3:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.11-8.08 (m, 2H), 7.53-7.48 (m, 1H), 7.38-7.32 (m, 2H), 2.45 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 158.1, 139.7, 134.1, 131.4, 129.8, 128.2, 127.0, 113.8, 112.4, 83.9; HRMS calcd for $C_{11}H_7N_2$ $[M-H]^+$: 167.0609, found: *m/z* 167.0609; IR (neat) 2226, 1578, 1482, 1372, 1304, 1269, 1231 cm^{-1}.

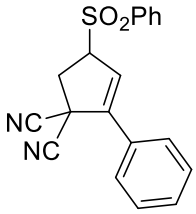
3. NHC-promoted [3+2] cyclization of allenyl sulfone and arylidenemalononitrile

a. General procedure for NHC-promoted [3+2] cyclization of allenyl sulfone and arylidenemalononitriles

A 10 mL flame-dried test tube with a magnetic stirring bar was charged with **1a** (1.2 mg, 0.003 mmol), Cs_2CO_3 (1.0 mg, 0.003 mmol), allenyl sulfone **2a** (18 mg, 0.1 mmol), and arylidenemalononitrile **3** (0.12 mmol). The test tube was filled with argon by the evacuation−refill process. After addition of THF (0.5 mL), the mixture was stirred at ambient temperature until TLC monitoring showed that **2a** was completely consumed. After evaporation of THF *in vacuo*, the residue was purified by silica-gel column chromatography to give product **4**.

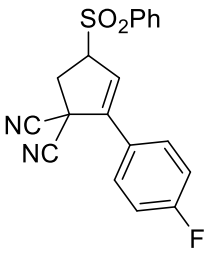
b. Analytical data for product 4

2-Phenyl-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4a)



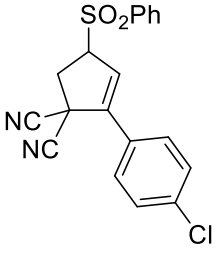
Accoding to the General Procedure, the title compound was obtained after 24 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 2:1) gave the product as pale green oil (31 mg, 92% yield). R_f=0.30 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.67-7.59 (m, 4H), 7.49-7.46 (m, 3H), 6.44 (d, *J* = 2.8 Hz, 1H), 4.67 (ddd, *J* = 2.8, 5.6, 8.4 Hz, 1H), 3.26 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.20 (dd, *J* = 8.4, 15.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 142.8, 135.9, 135.0, 130.7, 129.8, 129.2, 129.1, 128.9, 126.7, 125.9, 114.5, 113.4, 69.7, 40.0, 38.2; HRMS calcd for $C_{19}H_{18}N_3O_2S$ $[M+NH_4]^+$: 352.1120, found: *m/z* 352.1118; IR (neat) 2241, 1585, 1497, 1448, 1308, 1224, 1142, 1089 cm^{-1}.

2-(4-Fluorophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4b)



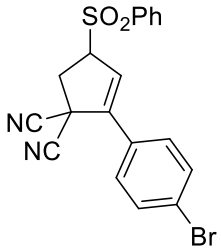
Accoding to the General Procedure, the title compound was obtained after 30 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 3:1) gave the product as pale green oil (30 mg, 84% yield). R_f=0.30 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.76 (tt, *J* = 1.2, 7.6 Hz, 1H), 7.67-7.59 (m, 4H), 7.19-7.15 (m, 2H), 6.38 (d, *J* = 2.8 Hz, 1H), 4.66 (ddd, *J* = 2.4, 5.6, 8.4 Hz, 1H), 3.26 (dd, *J* = 5.6, 15.2 Hz, 1H), 3.18 (dd, *J* = 8.8, 15.2 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 163.9 (d, $^1J_{CF}$ = 256.5 Hz), 141.9, 135.9, 135.0, 129.9, 128.93, 128.86 (d, $^3J_{CF}$ = 6.7 Hz), 125.9, 125.5, 116.6 (d, $^2J_{CF}$ = 22.9 Hz), 114.3, 113.3, 69.7, 40.2, 38.3; HRMS calcd for $C_{19}H_{17}FN_3O_2S$ $[M+NH_4]^+$: 370.1026, found: *m/z* 370.1028; IR (neat) 2283, 1604, 1512, 1447, 1321, 1239, 1150, 1085 cm^{-1}.

2-(4-Chlorophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4c)



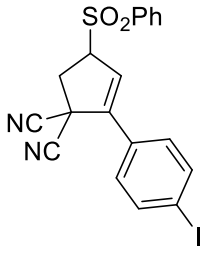
Accoding to the General Procedure, the title compound was obtained after 24 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 3:1) gave the product as pale green oil (34 mg, 92% yield). R_f=0.33 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.92 (m, 2H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.67-7.63 (m, 2H), 7.56-7.53 (m, 2H), 7.46-7.44 (m, 2H), 6.44 (d, *J* = 2.8 Hz, 1H), 4.66 (ddd, *J* = 2.8, 5.6, 8.4 Hz, 1H), 3.26 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.18 (dd, *J* = 8.4, 15.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 141.7, 136.9, 135.8, 135.0, 129.9, 129.6, 128.9, 128.0, 127.6, 126.6, 114.2, 113.2, 69.7, 40.0, 38.2; HRMS calcd for $C_{19}H_{17}ClN_3O_2S$ $[M+NH_4]^+$: 386.0730, found: *m/z* 386.0736; IR (neat) 2262, 1494, 1448, 1405, 1311, 1266, 1219, 1154, 1096 cm^{-1}.

2-(4-Bromophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4d)



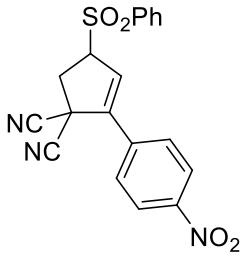
Accoding to the General Procedure, the title compound was obtained after 8 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 3:1) gave the product as pale green solids (38 mg, 92% yield). R_f=0.33 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.92 (m, 2H), 7.77 (tt, *J* = 7.6, 1.2 Hz, 1H), 7.67-7.55 (m, 4H), 7.49-7.45 (m, 2H), 6.44 (d, *J* = 2.8 Hz, 1H), 4.66 (ddd, *J* = 2.8, 5.6, 8.4 Hz, 1H), 3.26 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.18 (dd, *J* = 8.4, 15.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 141.8, 135.8, 135.0, 132.5, 129.9, 128.9, 128.1, 128.0, 126.7, 125.2, 114.2, 113.2, 69.7, 39.9, 38.2; HRMS calcd for $C_{19}H_{17}BrN_3O_2S$ $[M+NH_4]^+$: 430.0225, found: *m/z* 430.0226; IR (neat) 2233, 1492, 1447, 1322, 1276, 1266, 1152, 1085 cm^{-1}. Single crystals were obtained by recrystallization from acetone and hexane.

2-(4-Iodophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4e)



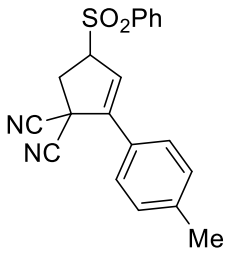
Accoding to the General Procedure, the title compound was obtained after 48 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 2:1) gave the product as gray solids (25 mg, 54% yield). R_f=0.33 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.0 Hz, 2H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.76 (t, *J* = 8.0 Hz, 1H), 7.65 (t, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.47 (d, *J* = 2.8 Hz, 1H), 4.64 (ddd, *J* = 2.4, 5.6, 8.4 Hz, 1H), 3.25 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.18 (dd, *J* = 8.4, 15.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 142.0, 138.5, 135.8, 135.1, 129.9, 128.9, 128.6, 128.1, 126.7, 114.2, 113.2, 97.3, 69.7, 39.9, 38.2; HRMS calcd for $C_{19}H_{17}IN_3O_2S$ $[M+NH_4]^+$: 478.0086, found: *m/z* 478.0081; IR (neat) 2256, 1583, 1488, 1447, 1397, 1309, 1155, 1085, 1005 cm^{-1}.

2-(4-Nitrophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4f)



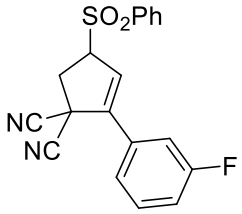
A 10 mL flame-dried test tube with a magnetic stirring bar was charged with **1a** (1.2 mg, 0.003 mmol), Cs_2CO_3 (1.0 mg, 0.003 mmol), allenyl sulfone **2a** (18 mg, 0.1 mmol), and **3f** (24 mg, 0.12 mmol). The test tube was filled with argon by the evacuation−refill process. After addition of THF (0.5 mL), the mixture was stirred at ambient for 12 h. To the suspension, additional **1a** (1.2 mg, 0.003 mmol) and Cs_2CO_3 (1.0 mg, 0.003 mmol) were added and the mixture was stirred for 5 h. After evaporation of THF *in vacuo*, the residue was purified by silica-gel column chromatography (hexane:EtOAc = 4:1 to 1:2) to give product **4f** as white solids (20 mg, 53% yield). R_f=0.24 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 8.36-8.33 (m, 2H), 7.97-7.94 (m, 2H), 7.82-7.77 (m, 3H), 7.70-7.66 (m, 2H), 6.64 (d, *J* = 2.4 Hz, 1H), 4.71 (ddd, *J* = 2.8, 5.6, 8.4 Hz, 1H), 3.32 (dd, *J* = 5.6, 15.2 Hz, 1H), 3.21 (dd, *J* = 8.8, 15.2 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 148.6, 140.9, 135.9, 135.2, 135.1, 130.4, 130.0, 128.8, 127.8, 124.5, 113.9, 112.9, 69.7, 40.1, 38.2; HRMS calcd for $C_{19}H_{17}N_4O_4S$ $[M+NH_4]^+$: 397.0971, found: *m/z* 397.0968; IR (neat) 2249, 1598, 1516, 1447, 1352, 1155, 1084 cm^{-1}.

4-(Phenylsulfonyl)-2-(*p*-tolyl)cyclopent-2-ene-1,1-dicarbonitrile (4g)



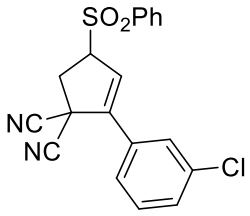
Accoding to the General Procedure, the title compound was obtained after 20 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 2:1) gave the product as pale yellow oil (31 mg, 88% yield). R_f=0.39 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.94-7.92 (m, 2H), 7.75 (tt, *J* = 1.6, 7.2 Hz, 1H), 7.66-7.62 (m, 2H), 7.51-7.49 (m, 2H), 7.27-7.25 (m, 2H), 6.39 (d, *J* = 2.4 Hz, 1H), 4.66 (ddd, *J* = 2.4, 5.6, 8.4 Hz, 1H), 3.24 (dd, *J* = 5.2, 15.6 Hz, 1H), 3.18 (dd, *J* = 8.8, 15.2 Hz, 1H), 2.40 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 142.8, 141.1, 135.8, 134.9, 129.9, 129.8, 128.9, 126.6, 126.3, 124.7, 114.6, 113.5, 69.7, 39.9, 38.2, 21.3; HRMS calcd for $C_{20}H_{20}N_3O_2S$ $[M+NH_4]^+$: 366.1276, found: *m/z* 366.1275; IR (neat) 2260, 1509, 1447, 1309, 1152, 1085 cm^{-1}.

2-(3-Fluorophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4h)



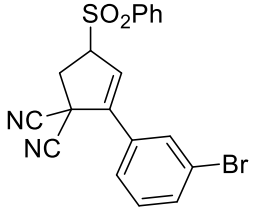
Accoding to the General Procedure, the title compound was obtained after 31 h. Silica gel column chromatography (hexane:EtOAc = 4:1) gave the product as pale green oil (30 mg, 85% yield). R_f=0.33 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.97-7.93 (m, 2H), 7.77 (tt, *J* = 1.2, 7.6 Hz, 1H), 7.68-7.62 (m, 2H), 7.48-7.39 (m, 2H), 7.32-7.16 (m, 2H), 6.45 (d, *J* = 2.8 Hz, 1H), 4.67 (ddd, *J* = 2.8, 5.6, 8.4 Hz, 1H), 3.27 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.19 (dd, *J* = 8.8, 15.2 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 162.8 (d, $^1J_{CF}$ = 252.8 Hz), 141.7, 135.8, 135.0, 131.2 (d, $^3J_{CF}$ = 7.6 Hz), 131.1 (d, $^3J_{CF}$ = 8.6 Hz), 129.9, 128.9, 127.6, 122.5, 117.8 (d, $^2J_{CF}$ = 22.0 Hz), 114.2, 113.9 (d, $^2J_{CF}$ = 23.8 Hz), 113.2, 69.6, 40.1, 38.2; HRMS calcd for $C_{19}H_{17}FN_3O_2S$ $[M+NH_4]^+$: 370.1026, found: *m/z* 370.1021; IR (neat) 2225, 1446, 1322, 1266, 1153, 1085 cm^{-1}.

2-(3-Chlorophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4i)



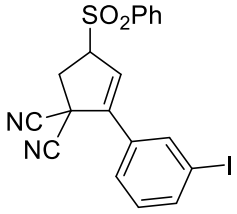
Accoding to the General Procedure, the title compound was obtained after 20 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 3:1) gave the product as pale green oil (31 mg, 84% yield). R_f=0.33 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.78 (tt, *J* = 1.2, 7.6 Hz, 1H), 7.70-7.64 (m, 2H), 7.55-7.40 (m, 4H), 6.45 (d, *J* = 2.8 Hz, 1H), 4.67 (ddd, *J* = 2.4, 5.2, 8.4 Hz, 1H), 3.28 (dd, *J* = 5.2, 15.2 Hz, 1H), 3.19 (dd, *J* = 8.8, 15.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 141.6, 135.9, 135.3, 135.1, 130.9, 130.8, 130.6, 129.9, 128.9, 127.7, 126.9, 124.7, 114.2, 113.2, 69.6, 40.0, 38.2; HRMS calcd for $C_{19}H_{17}ClN_3O_2S$ $[M+NH_4]^+$: 386.0730, found: *m/z* 386.0732; IR (neat) 2278, 1567, 1447, 1321, 1268, 1085 cm^{-1}.

2-(3-Bromophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4j)



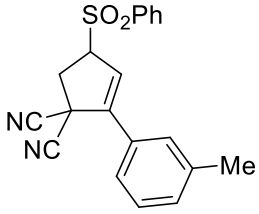
Accoding to the General Procedure, the title compound was obtained after 20 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 3:1) gave the product as pale green oil (36 mg, 87% yield). R_f=0.32 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.2 Hz, 2H), 7.77 (t, *J* = 7.2 Hz, 1H), 7.70-7.55 (m, 5H), 7.36 (t, *J* = 8.0 Hz, 1H), 6.45 (d, *J* = 2.4 Hz, 1H), 4.67 (ddd, *J* = 2.4, 5.6, 8.4 Hz, 1H), 3.27 (dd, *J* = 5.2, 15.2 Hz, 1H), 3.19 (dd, *J* = 8.8, 15.6 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 141.5, 135.9, 135.1, 133.7, 131.2, 130.8, 129.9, 129.8, 128.9, 127.7, 125.1, 123.3, 114.1, 113.1, 69.6, 40.0, 38.2; HRMS calcd for $C_{19}H_{17}BrN_3O_2S$ $[M+NH_4]^+$: 430.0225, found: *m/z* 430.0223; IR (neat) 2229, 1560, 1447, 1322, 1085 cm^{-1}.

2-(3-Iodophenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4k)



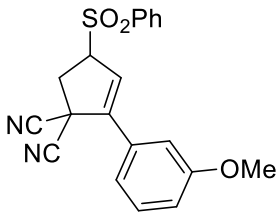
Accoding to the General Procedure, the title compound was obtained after 46 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 2:1) gave the product as pale yellow oil (42 mg, 91% yield). R_f=0.32 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.89-7.87 (m, 1H), 7.82-7.76 (m, 2H), 7.68-7.58 (m, 2H), 7.36 (t, *J* = 8.0 Hz, 1H), 6.44 (d, *J* = 2.4 Hz, 1H), 4.66 (ddd, *J* = 2.8, 5.2, 8.4 Hz, 1H), 3.27 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.18 (dd, *J* = 8.8, 15.2 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 141.4, 139.6, 135.9, 135.7, 135.1, 131.2, 130.8, 129.9, 128.9, 127.5, 125.6, 114.1, 113.1, 94.9, 69.6, 40.0, 38.3; HRMS calcd for $C_{19}H_{17}IN_3O_2S$ $[M+NH_4]^+$: 478.0086, found: *m/z* 478.0087; IR (neat) 2249, 1554, 1447, 1309, 1151, 1082 cm^{-1}.

4-(Phenylsulfonyl)-2-(*m*-tolyl)cyclopent-2-ene-1,1-dicarbonitrile (4l)



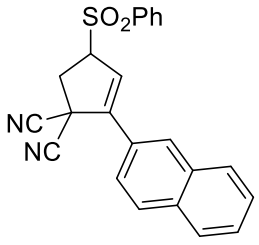
Accoding to the General Procedure, the title compound was obtained after 19 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 1:1) gave the product as pale green oil (31 mg, 88% yield). R_f=0.32 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.75 (tt, *J* = 1.2, 7.6 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 2H), 7.42-7.25 (m, 4H), 6.41 (d, *J* = 2.4 Hz, 1H), 4.66 (ddd, *J* = 2.4, 5.6, 8.4 Hz, 1H), 3.24 (dd, *J* = 5.6, 15.2 Hz, 1H), 3.18 (dd, *J* = 8.8, 15.6 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 143.0, 139.1, 135.9, 134.9, 131.5, 129.8, 129.10, 129.08, 128.9, 127.3, 125.6, 123.8, 114.5, 113.5, 69.7, 40.0, 38.3, 21.4; HRMS calcd for $C_{20}H_{20}N_3O_2S$ $[M+NH_4]^+$: 366.1276, found: *m/z* 366.1272; IR (neat) 2283, 1585, 1448, 1322, 1267, 1153, 1086 cm^{-1}.

2-(3-Methoxyphenyl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4m)



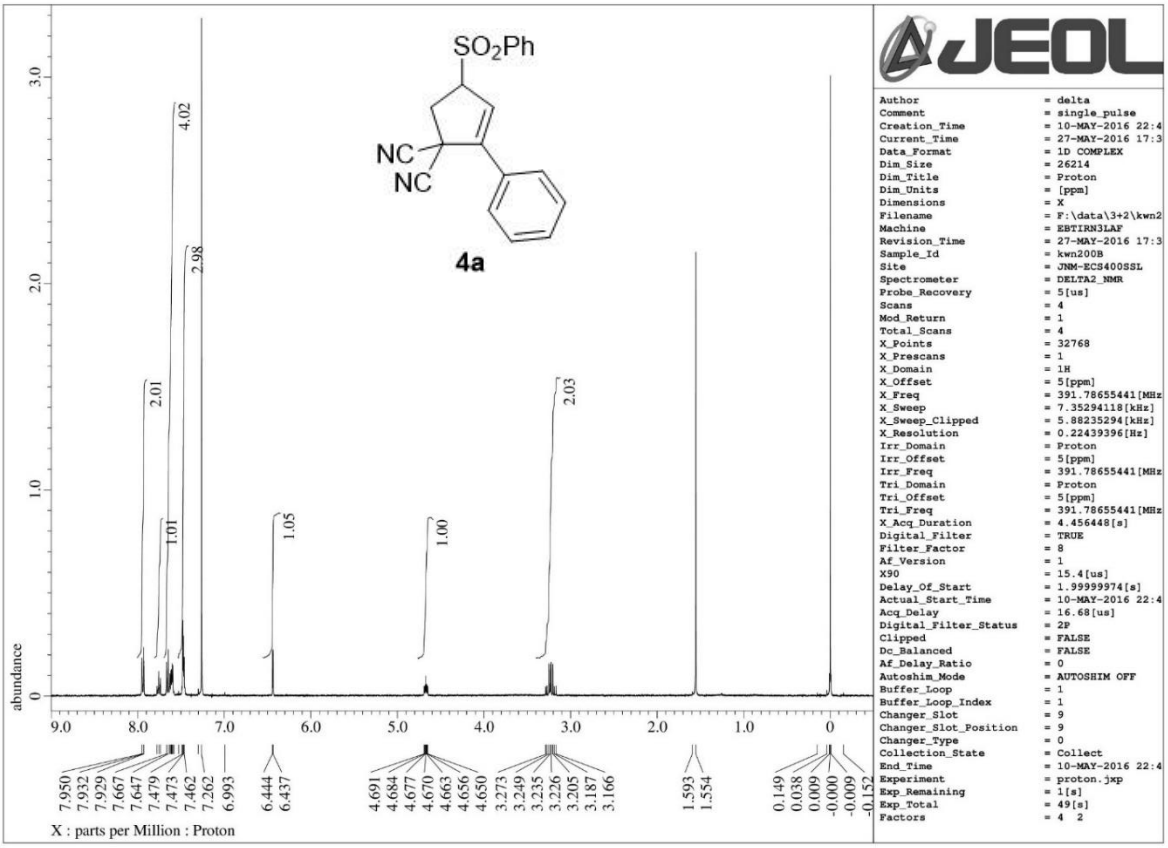
Accoding to the General Procedure, the title compound was obtained after 19 h. Silica gel column chromatography (hexane:EtOAc = 3:1) gave the product as pale yellow oil (35 mg, 95% yield). R_f=0.26 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, $CDCl_3$) δ 7.95-7.92 (m, 2H), 7.75 (tt, *J* = 1.2, 7.2 Hz, 1H), 7.64 (t, *J* = 7.2 Hz, 2H), 7.38 (t, *J* = 8.0 Hz, 1H), 7.20-7.19 (m, 1H), 7.09-7.08 (m, 1H), 7.02-6.99 (m, 1H), 6.42 (d, *J* = 2.8 Hz, 1H), 4.66 (ddd, *J* = 2.4, 5.6, 8.0 Hz, 1H), 3.85 (s, 3H), 3.25 (dd, *J* = 5.6, 15.6 Hz, 1H), 3.18 (dd, *J* = 8.4, 15.2 Hz, 1H); ^{13}C NMR (100MHz, $CDCl_3$) δ 159.9, 142.7, 135.8, 135.0, 130.4, 130.3, 129.8, 128.9, 126.3, 119.0, 116.2, 114.5, 113.4, 112.3, 69.6, 55.3, 40.1, 38.2; HRMS calcd for $C_{20}H_{20}N_3O_3S$ $[M+NH_4]^+$: 382.1225, found: *m/z* 382.1223; IR (neat) 2256, 1601, 1580, 1491, 1447, 1319, 1217, 1151, 1085, 1038 cm^{-1}.

2-(Naphthalen-2-yl)-4-(phenylsulfonyl)cyclopent-2-ene-1,1-dicarbonitrile (4n)

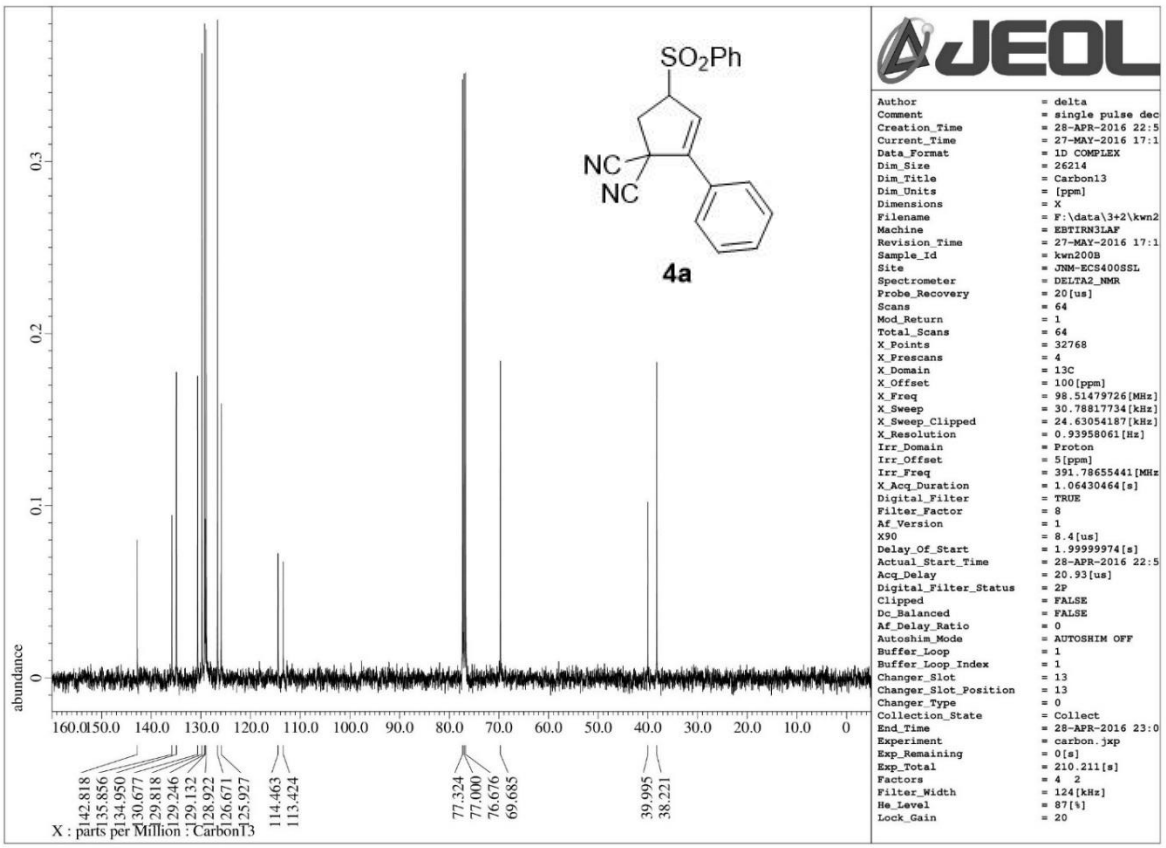


Accoding to the General Procedure, the title compound was obtained after 45 h. Silica gel column chromatography (hexane:EtOAc = 4:1 to 1:3) gave the product as pale yellow solids (28 mg, 73% yield). R_f=0.29 (hexane:EtOAc = 2:1); ^{1}H NMR (400MHz, acetone–d_6) δ 8.29 (d, *J* = 1.6 Hz, 1H), 8.06-7.90 (m, 6H), 7.83 (tt, *J* = 1.6, 7.6 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 2H), 7.65-7.60 (m, 2H), 6.91 (d, *J* = 2.8 Hz, 1H), 5.15 (ddd, *J* = 2.8, 4.4, 8.8 Hz, 1H), 3.57 (dd, *J* = 8.8, 15.6 Hz, 1H), 3.43 (dd, *J* = 4.4, 15.6 Hz, 1H); ^{13}C NMR (100MHz, acetone-d_6) δ 142.8, 138.1, 135.7, 134.9, 134.0, 130.8, 130.2, 130.1, 129.7, 129.3, 128.8, 128.4, 128.2, 127.4, 124.9, 116.3, 115.5, 71.0, 41.2, 39.1; HRMS calcd for $C_{23}H_{20}N_3O_2S$ $[M+NH_4]^+$: 402.1276, found: *m/z* 402.1273; IR (neat) 2229, 1315, 1146 cm^{-1}.

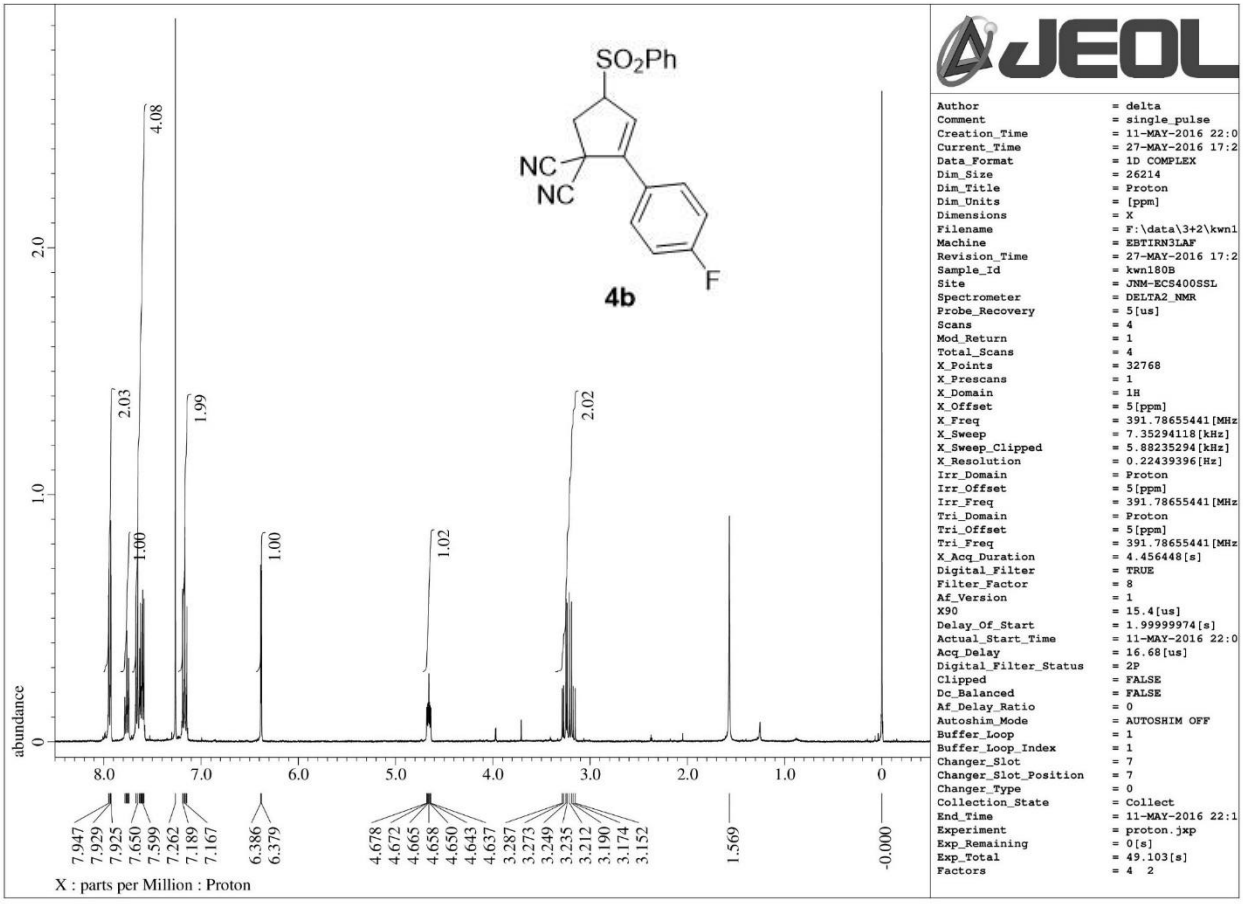
4. NMR spectra



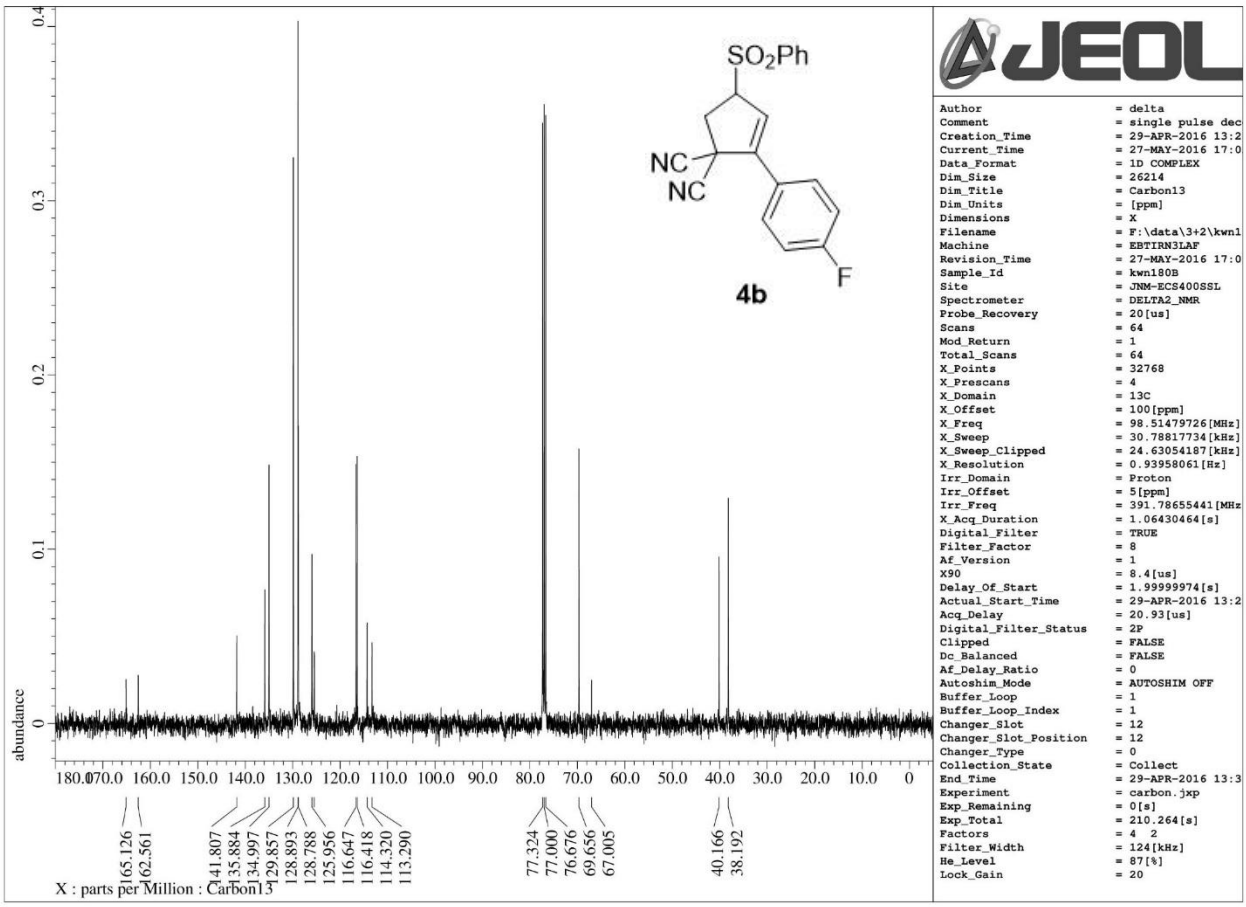
Author	= delta
Comment	= single_pulse
Creation_Time	= 10-MAY-2016 22:4
Current_Time	= 27-MAY-2016 17:3
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:3
Sample_Id	= kwn200B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 10-MAY-2016 22:4
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 9
Changer_Slot_Position	= 9
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 10-MAY-2016 22:4
Experiment	= proton.jxp
Exp_Remaining	= 1[s]
Exp_Total	= 49[s]
Factors	= 4 2



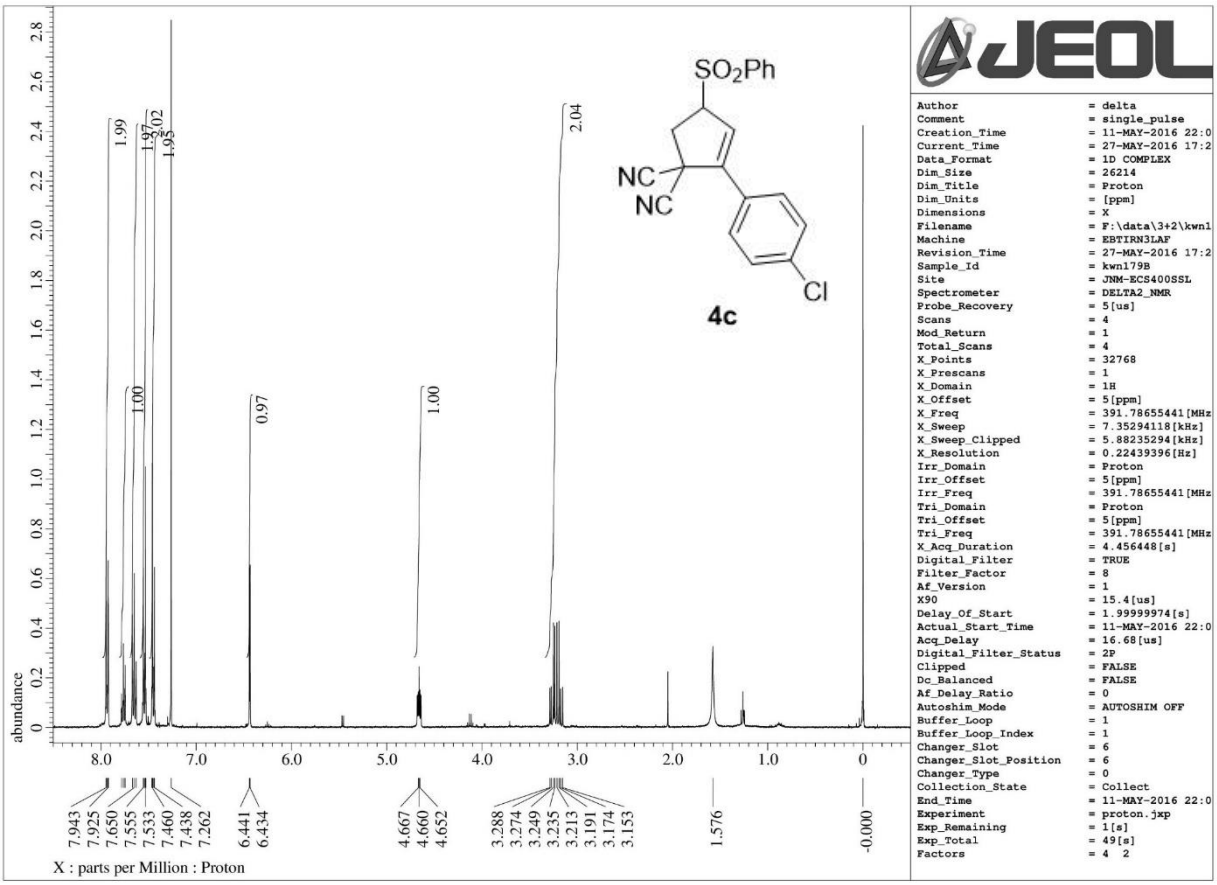
Author	= delta
Comment	= single pulse dec
Creation_Time	= 28-APR-2016 22:5
Current_Time	= 27-MAY-2016 17:1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:1
Sample_Id	= kwn200B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 64
Mod_Return	= 1
Total_Scans	= 64
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 28-APR-2016 22:5
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 13
Changer_Slot_Position	= 13
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 28-APR-2016 23:0
Experiment	= carbon.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 210.211[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 87[%]
Lock_Gain	= 20



Author	= delta
Comment	= single_pulse
Creation_Time	= 11-MAY-2016 22:0
Current_Time	= 27-MAY-2016 17:2
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:2
Sample_Id	= kwn180B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 11-MAY-2016 22:0
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 7
Changer_Slot_Position	= 7
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 11-MAY-2016 22:1
Experiment	= proton.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 49.103[s]
Factors	= 4 2

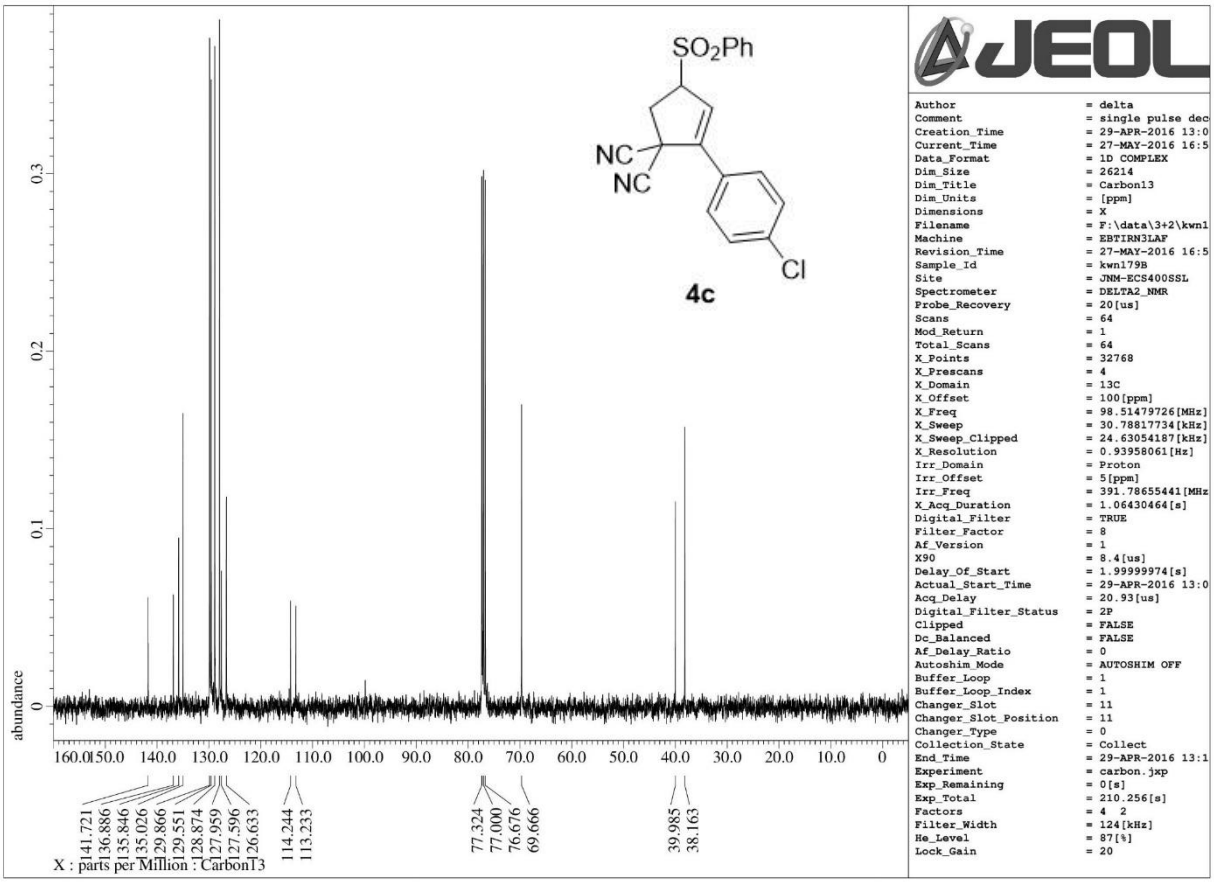


Author	= delta
Comment	= single pulse dec
Creation_Time	= 29-APR-2016 13:2
Current_Time	= 27-MAY-2016 17:0
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:0
Sample_Id	= kwn180B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 64
Mod_Return	= 1
Total_Scans	= 64
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 29-APR-2016 13:2
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 12
Changer_Slot_Position	= 12
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 29-APR-2016 13:3
Experiment	= carbon.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 210.264[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 87[%]
Lock_Gain	= 20



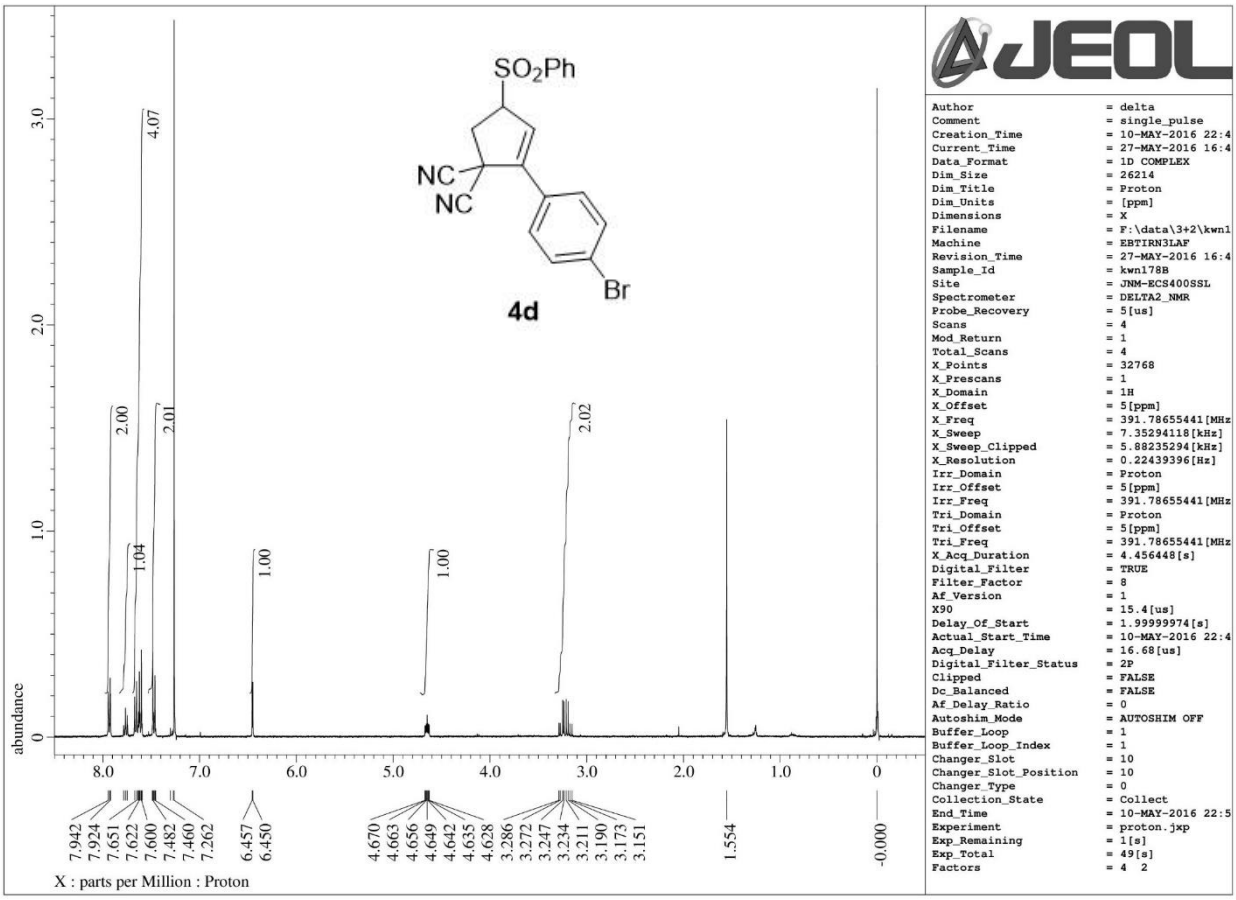
JEOL

Author	= delta
Comment	= single_pulse
Creation_Time	= 11-MAY-2016 22:0
Current_Time	= 27-MAY-2016 17:2
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:2
Sample_Id	= kwn179B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 11-MAY-2016 22:0
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 6
Changer_Slot_Position	= 6
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 11-MAY-2016 22:0
Experiment	= proton.jxp
Exp_Remaining	= 1[s]
Exp_Total	= 49[s]
Factors	= 4 2

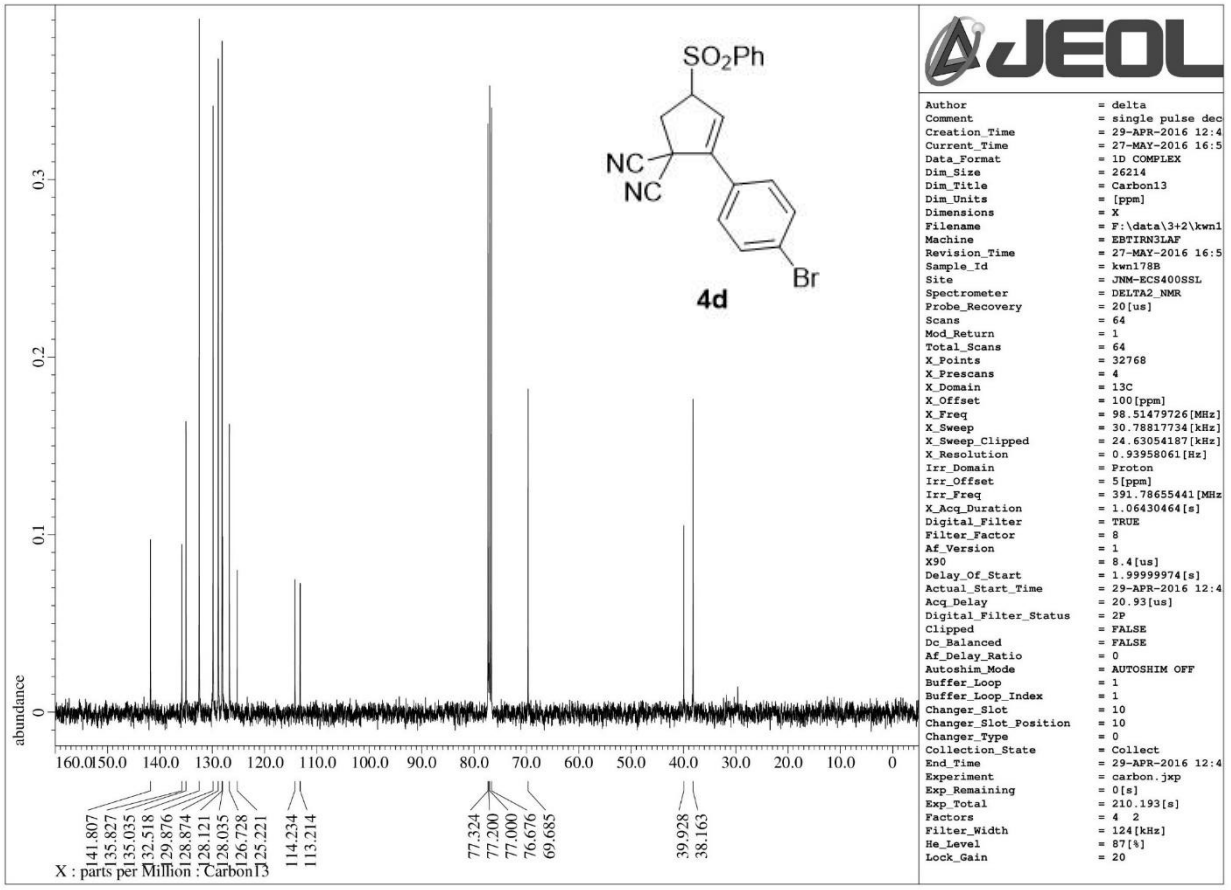


JEOL

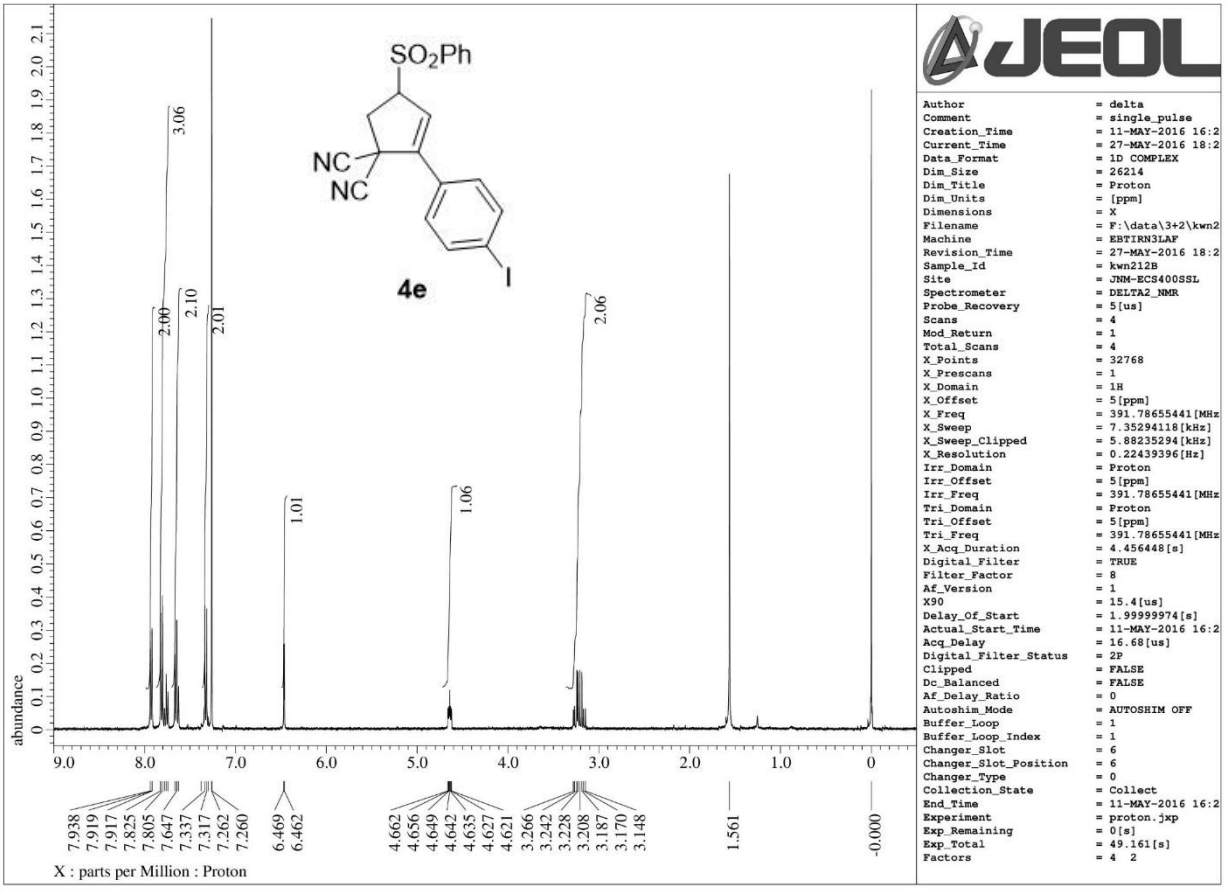
Author	= delta
Comment	= single pulse dec
Creation_Time	= 29-APR-2016 13:0
Current_Time	= 27-MAY-2016 16:5
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 16:5
Sample_Id	= kwn179B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 64
Mod_Return	= 1
Total_Scans	= 64
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 29-APR-2016 13:0
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 11
Changer_Slot_Position	= 11
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 29-APR-2016 13:1
Experiment	= carbon.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 210.256[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 87[%]
Lock_Gain	= 20



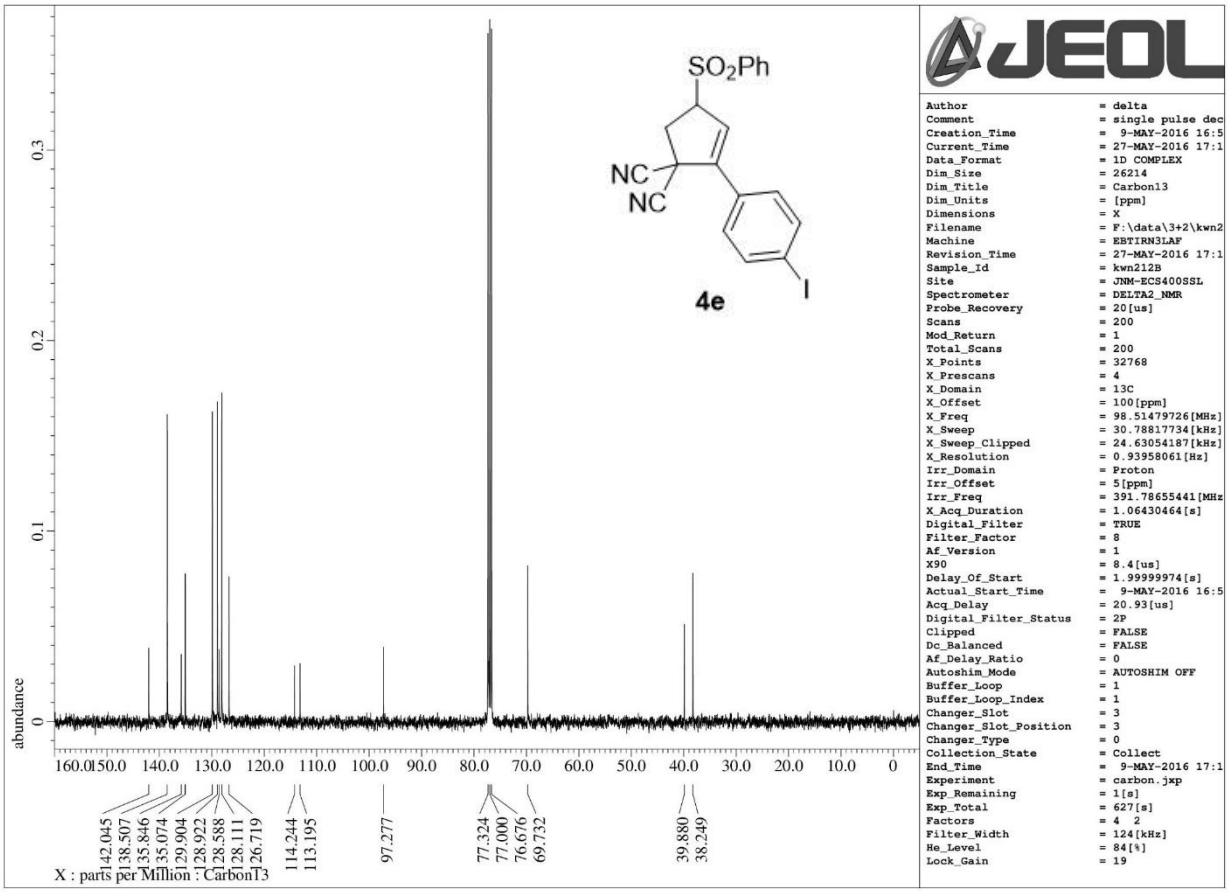
Author	= delta
Comment	= single_pulse
Creation_Time	= 10-MAY-2016 22:4
Current_Time	= 27-MAY-2016 16:4
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 16:4
Sample_Id	= kwn178B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 10-MAY-2016 22:4
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 10
Changer_Slot_Position	= 10
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 10-MAY-2016 22:5
Experiment	= proton.jxp
Exp_Remaining	= 1[s]
Exp_Total	= 49[s]
Factors	= 4 2



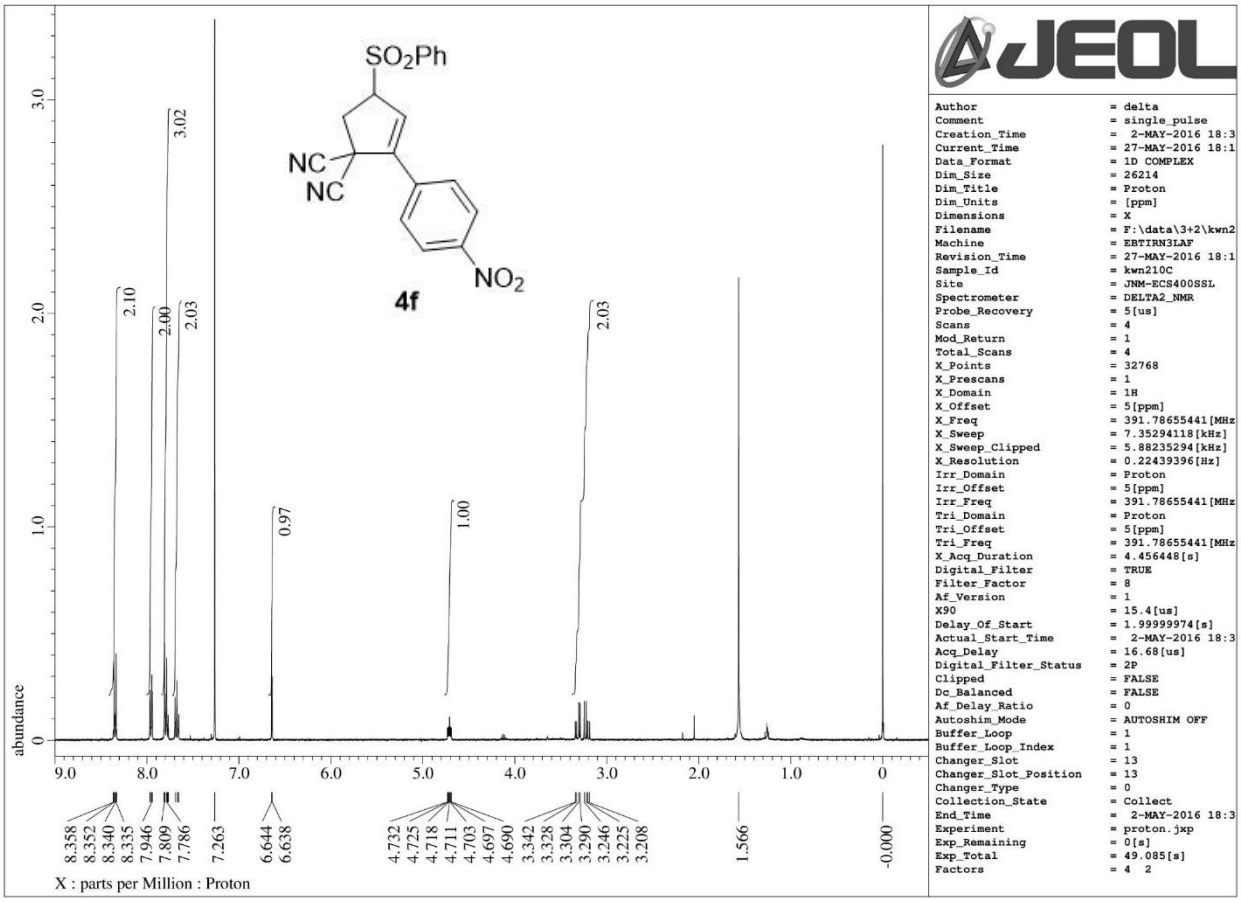
Author	= delta
Comment	= single pulse dec
Creation_Time	= 29-APR-2016 12:4
Current_Time	= 27-MAY-2016 16:5
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 16:5
Sample_Id	= kwn178B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 64
Mod_Return	= 1
Total_Scans	= 64
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 29-APR-2016 12:4
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 10
Changer_Slot_Position	= 10
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 29-APR-2016 12:4
Experiment	= carbon.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 210.193[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 87[%]
Lock_Gain	= 20



Author	= delta
Comment	= single_pulse
Creation_Time	= 11-MAY-2016 16:2
Current_Time	= 27-MAY-2016 18:2
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 18:2
Sample_Id	= kwn212B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 11-MAY-2016 16:2
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 6
Changer_Slot_Position	= 6
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 11-MAY-2016 16:2
Experiment	= proton.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 49.161[s]
Factors	= 4 2

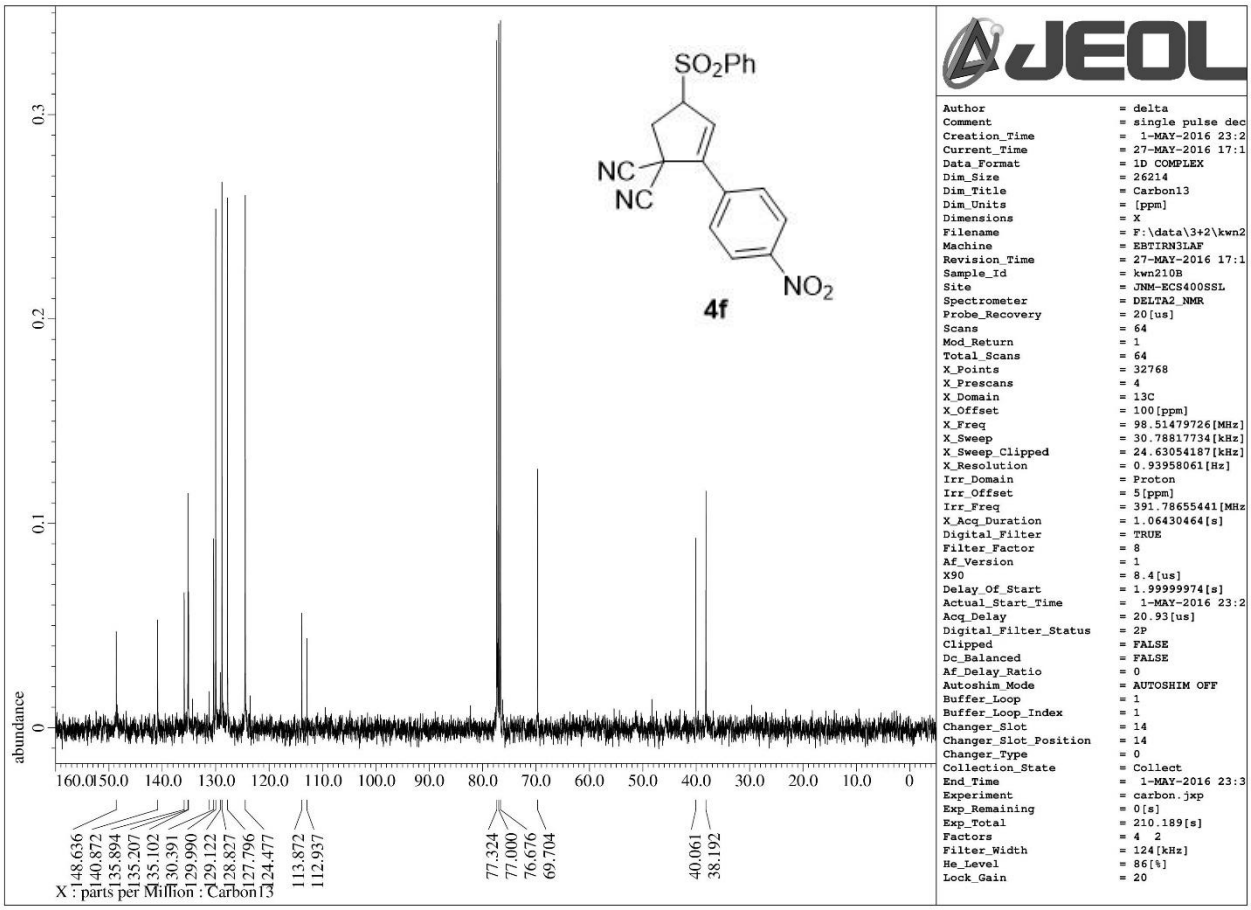


Author	= delta
Comment	= single pulse dec
Creation_Time	= 9-MAY-2016 16:5
Current_Time	= 27-MAY-2016 17:1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:1
Sample_Id	= kwn212B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 200
Mod_Return	= 1
Total_Scans	= 200
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 9-MAY-2016 16:5
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 3
Changer_Slot_Position	= 3
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 9-MAY-2016 17:1
Experiment	= carbon.jxp
Exp_Remaining	= 1[s]
Exp_Total	= 627[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 84[%]
Lock_Gain	= 19



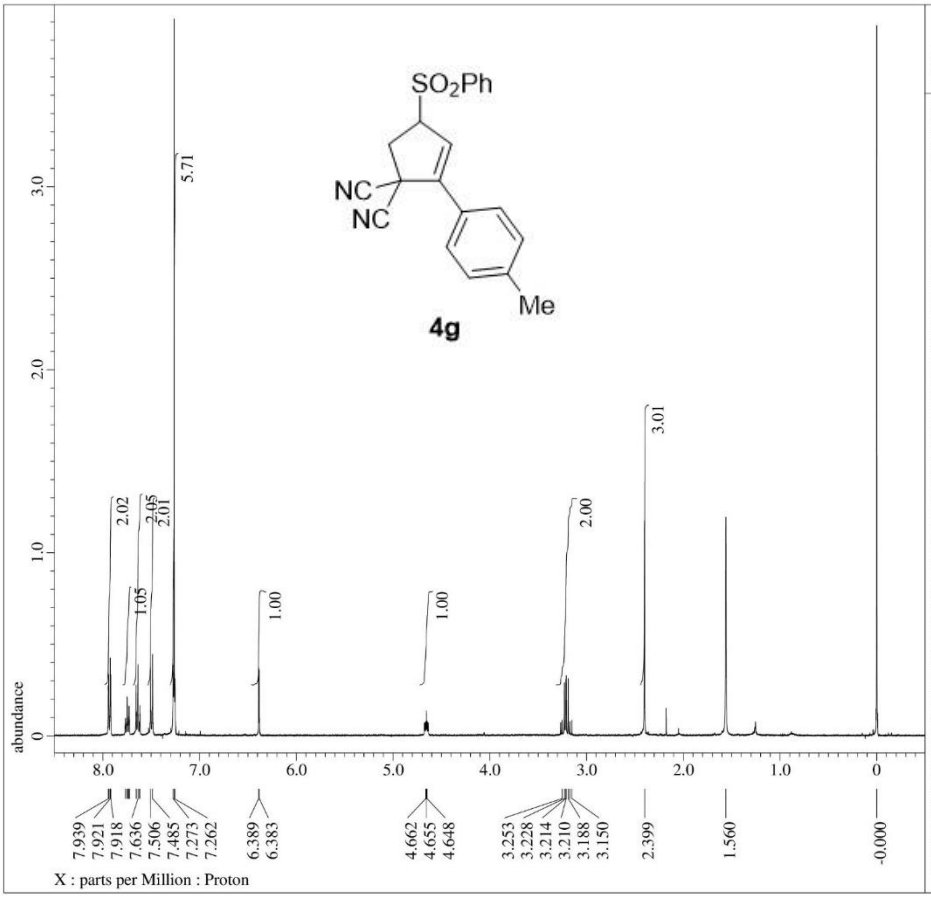
JEOL

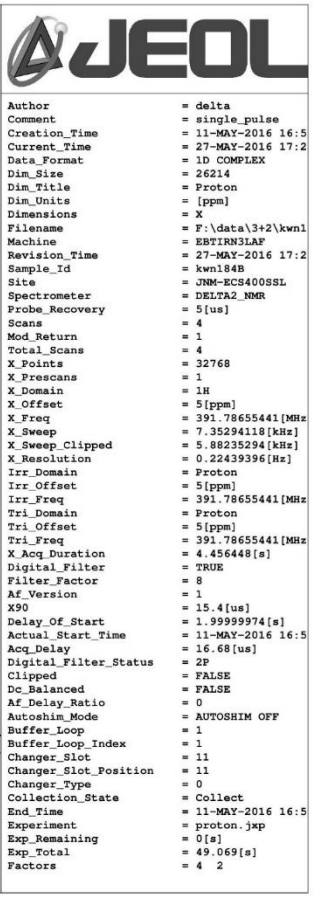
Author	= delta
Comment	= single_pulse
Creation_Time	= 2-MAY-2016 18:3
Current_Time	= 27-MAY-2016 18:1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 18:1
Sample_Id	= kwn210C
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 2-MAY-2016 18:3
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 13
Changer_Slot_Position	= 13
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 2-MAY-2016 18:3
Experiment	= proton.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 49.085[s]
Factors	= 4 2

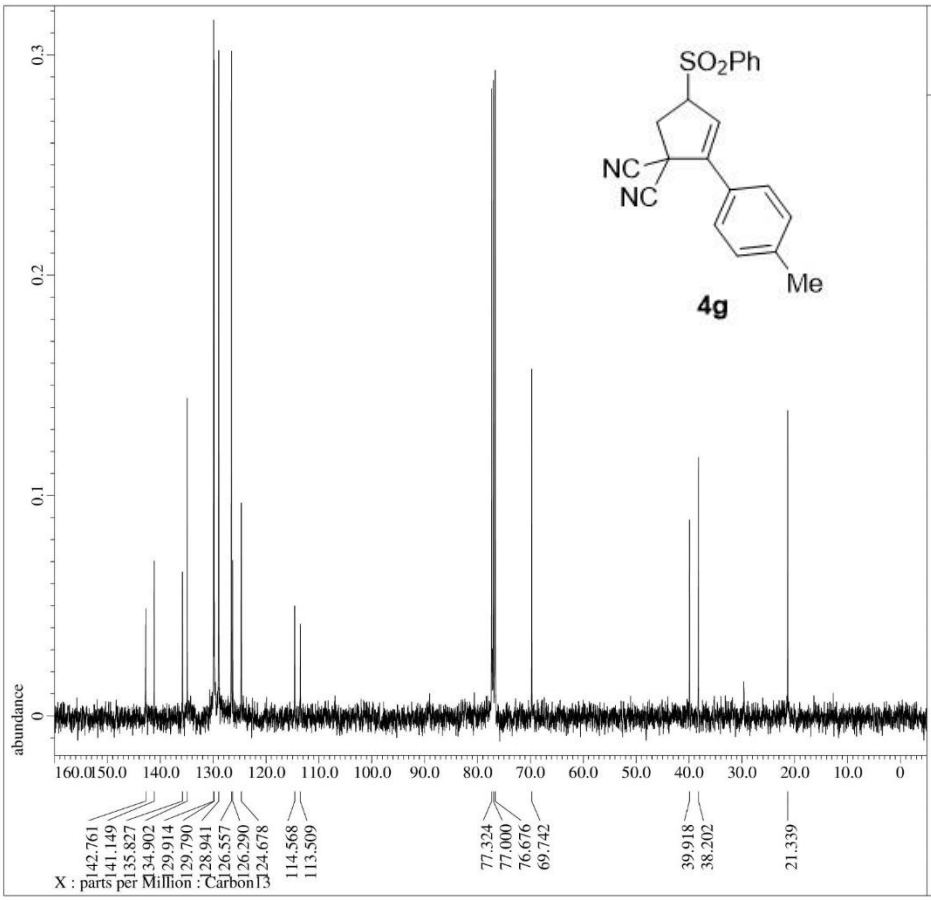


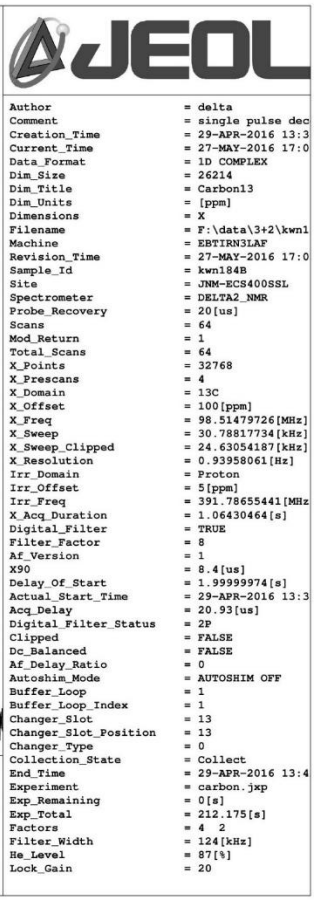
JEOL

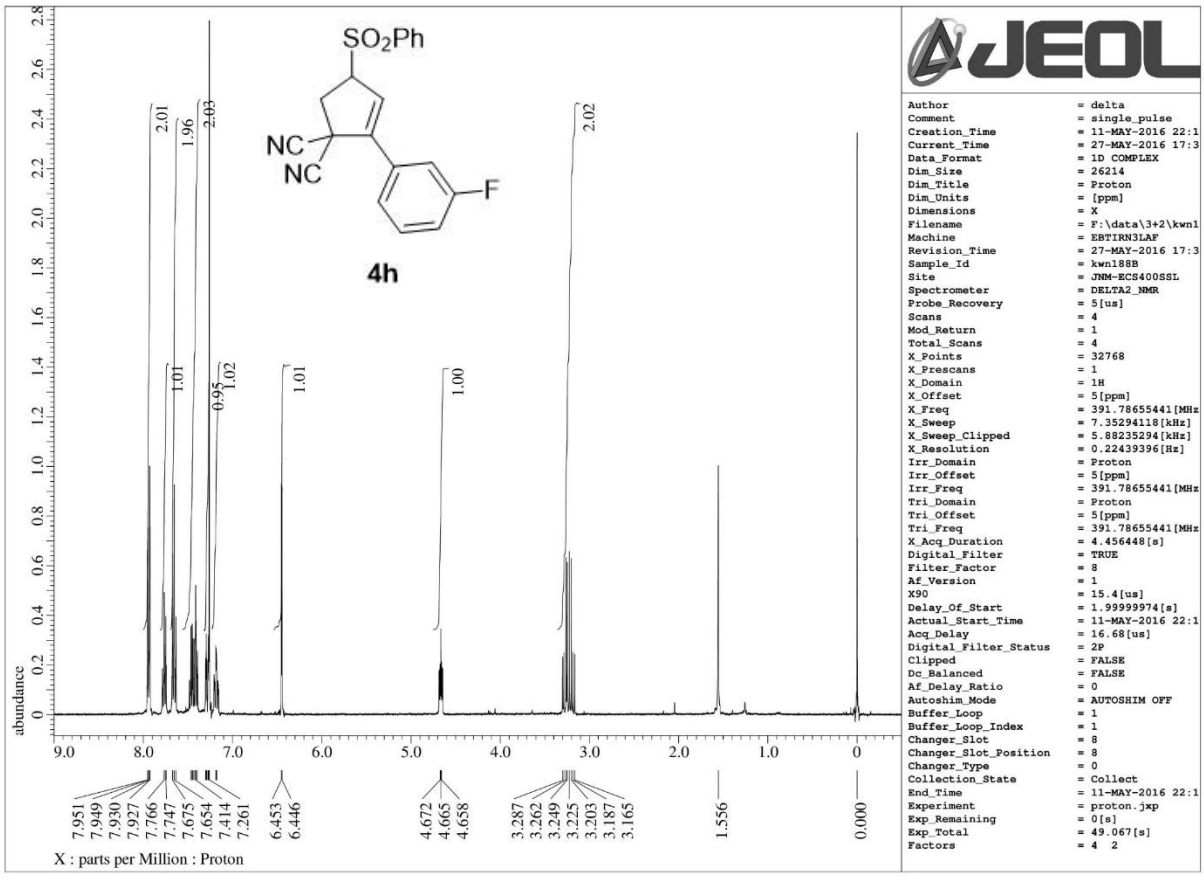
Author	= delta
Comment	= single pulse dec
Creation_Time	= 1-MAY-2016 23:2
Current_Time	= 27-MAY-2016 17:1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:1
Sample_Id	= kwn210B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 64
Mod_Return	= 1
Total_Scans	= 64
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 1-MAY-2016 23:2
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 14
Changer_Slot_Position	= 14
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 1-MAY-2016 23:3
Experiment	= carbon.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 210.189[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 86[%]
Lock_Gain	= 20

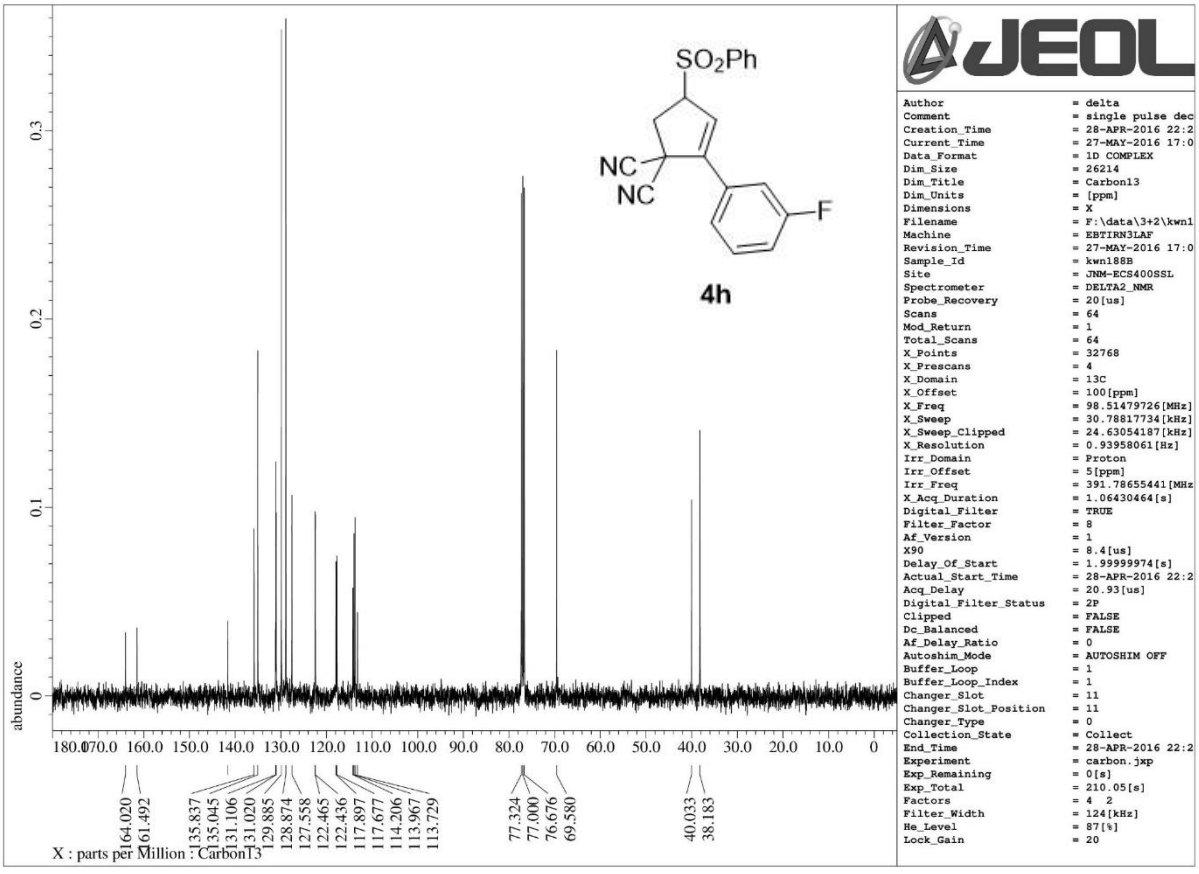

SO2Ph
NC
NC
Me
4g
5.71
3.01
2.02
2.05
2.01
2.00
1.05
1.00
1.00
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.939
7.921
7.918
7.636
7.506
7.485
7.273
7.262
6.389
6.383
4.662
4.655
4.648
3.253
3.228
3.214
3.210
3.188
3.150
2.399
1.560
-0.000
X : parts per Million : Proton

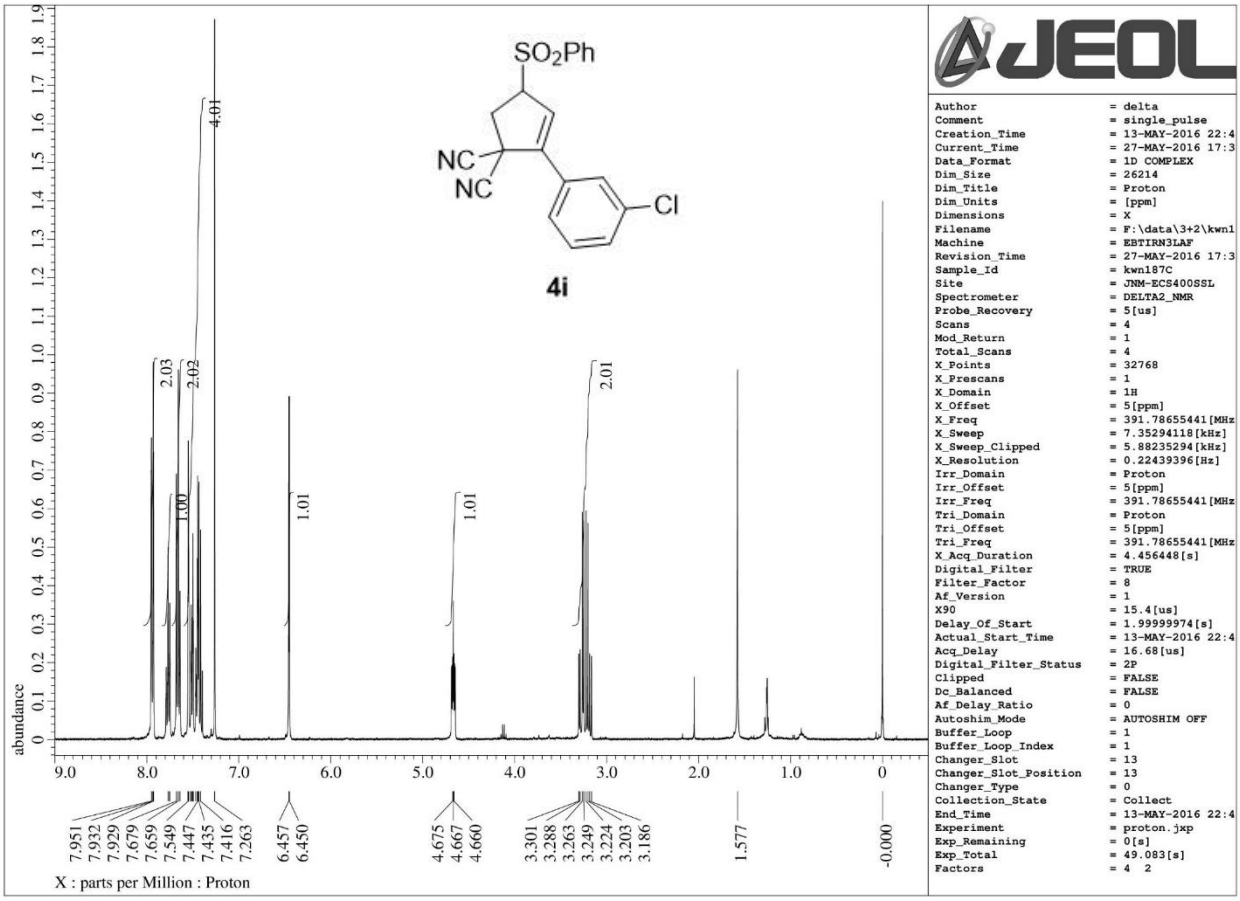

JEOL
Author = delta
Comment = single_pulse
Creation_Time = 11-MAY-2016 16:5
Current_Time = 27-MAY-2016 17:2
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn1
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:2
Sample_Id = kwn184B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 5[us]
Scans = 4
Mod_Return = 1
Total_Scans = 4
X_Points = 32768
X_Prescans = 1
X_Domain = 1H
X_Offset = 5[ppm]
X_Freq = 391.78655441[MHz
X_Sweep = 7.35294118[kHz]
X_Sweep_Clipped = 5.88235294[kHz]
X_Resolution = 0.22439396[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
Tri_Domain = Proton
Tri_Offset = 5[ppm]
Tri_Freq = 391.78655441[MHz
X_Acq_Duration = 4.456448[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 15.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 11-MAY-2016 16:5
Acq_Delay = 16.68[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 11
Changer_Slot_Position = 11
Changer_Type = 0
Collection_State = Collect
End_Time = 11-MAY-2016 16:5
Experiment = proton.jxp
Exp_Remaining = 0[s]
Exp_Total = 49.069[s]
Factors = 4 2


SO2Ph
NC
NC
Me
4g
abundance
0.3
0.2
0.1
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
0
142.761
141.149
135.827
134.902
129.914
129.790
128.941
126.557
126.290
124.678
114.568
113.509
77.324
77.000
76.676
69.742
39.918
38.202
21.339
X : parts per Million : Carbon13


JEOL
Author = delta
Comment = single_pulse dec
Creation_Time = 29-APR-2016 13:3
Current_Time = 27-MAY-2016 17:0
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn1
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:0
Sample_Id = kwn184B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 20[us]
Scans = 64
Mod_Return = 1
Total_Scans = 64
X_Points = 32768
X_Prescans = 4
X_Domain = 13C
X_Offset = 100[ppm]
X_Freq = 98.51479726[MHz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
X_Resolution = 0.93958061[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
X_Acq_Duration = 1.06430464[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 8.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 29-APR-2016 13:3
Acq_Delay = 20.93[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 13
Changer_Slot_Position = 13
Changer_Type = 0
Collection_State = Collect
End_Time = 29-APR-2016 13:4
Experiment = carbon.jxp
Exp_Remaining = 0[s]
Exp_Total = 212.175[s]
Factors = 4 2
Filter_Width = 124[kHz]
He_Level = 87[%]
Lock_Gain = 20

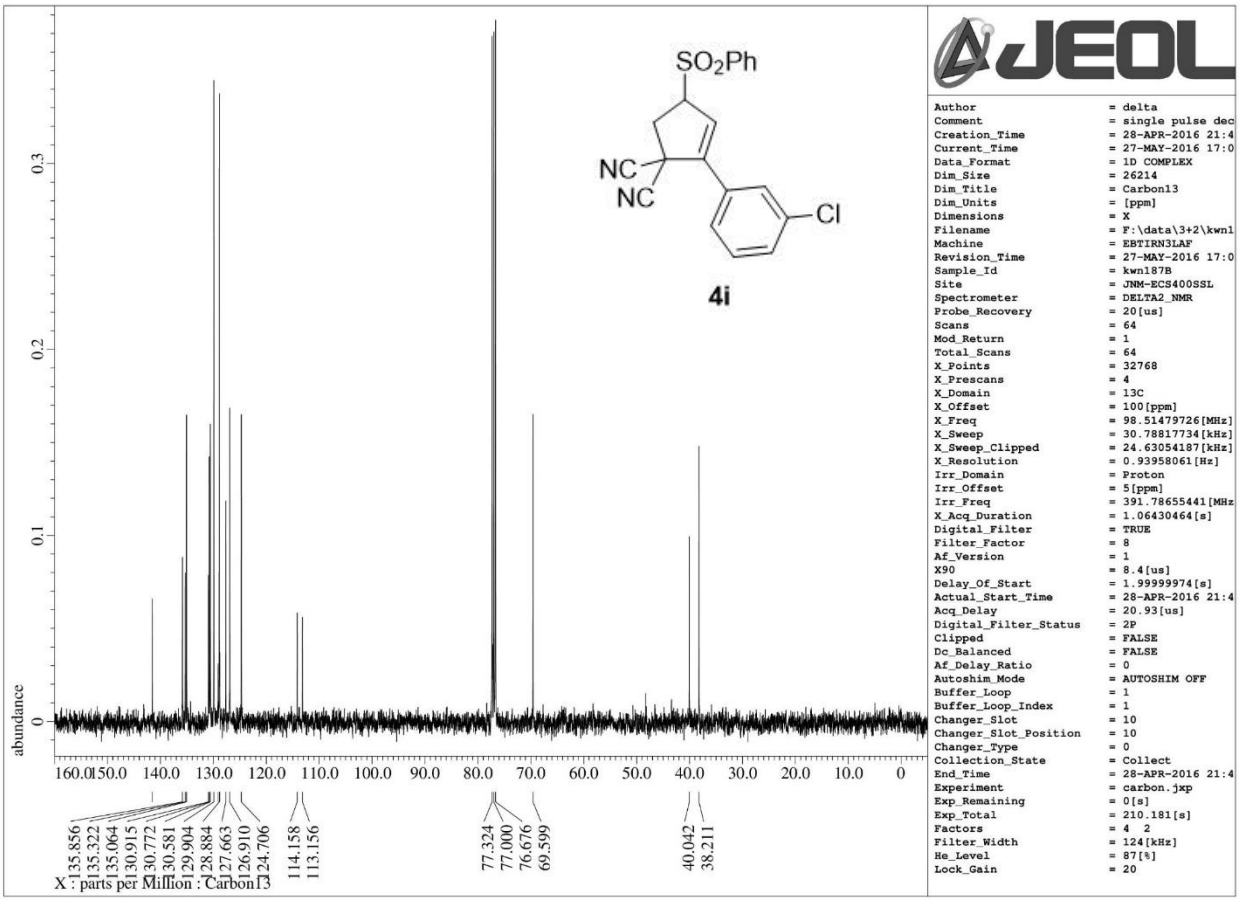

SO2Ph
NC
NC
F
4h
2.01
1.96
2.03
2.02
1.01
1.01
0.95
1.02
1.01
1.00
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.951
7.949
7.930
7.927
7.766
7.747
7.675
7.654
7.414
7.261
6.453
6.446
4.672
4.665
4.658
3.287
3.262
3.249
3.225
3.203
3.187
3.165
1.556
0.000
X : parts per Million : Proton
Author = delta
Comment = single_pulse
Creation_Time = 11-MAY-2016 22:1
Current_Time = 27-MAY-2016 17:3
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn1
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:3
Sample_Id = kwn188B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 5[us]
Scans = 4
Mod_Return = 1
Total_Scans = 4
X_Points = 32768
X_Prescans = 1
X_Domain = 1H
X_Offset = 5[ppm]
X_Freq = 391.78655441[MHz
X_Sweep = 7.35294118[kHz]
X_Sweep_Clipped = 5.88235294[kHz]
X_Resolution = 0.22439396[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
Tri_Domain = Proton
Tri_Offset = 5[ppm]
Tri_Freq = 391.78655441[MHz
X_Acq_Duration = 4.456448[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 15.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 11-MAY-2016 22:1
Acq_Delay = 16.68[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 8
Changer_Slot_Position = 8
Changer_Type = 0
Collection_State = Collect
End_Time = 11-MAY-2016 22:1
Experiment = proton.jxp
Exp_Remaining = 0[s]
Exp_Total = 49.067[s]
Factors = 4 2


SO2Ph
NC
NC
F
4h
abundance
0.3
0.2
0.1
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
0
164.020
161.492
135.837
135.045
131.106
131.020
129.885
128.874
127.558
122.465
122.436
117.897
117.677
114.206
113.967
113.729
77.324
77.000
76.676
69.580
40.033
38.183
X : parts per Million : Carbon13
Author = delta
Comment = single pulse dec
Creation_Time = 28-APR-2016 22:2
Current_Time = 27-MAY-2016 17:0
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn1
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:0
Sample_Id = kwn188B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 20[us]
Scans = 64
Mod_Return = 1
Total_Scans = 64
X_Points = 32768
X_Prescans = 4
X_Domain = 13C
X_Offset = 100[ppm]
X_Freq = 98.51479726[MHz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
X_Resolution = 0.93958061[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
X_Acq_Duration = 1.06430464[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 8.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 28-APR-2016 22:2
Acq_Delay = 20.93[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 11
Changer_Slot_Position = 11
Changer_Type = 0
Collection_State = Collect
End_Time = 28-APR-2016 22:2
Experiment = carbon.jxp
Exp_Remaining = 0[s]
Exp_Total = 210.05[s]
Factors = 4 2
Filter_Width = 124[kHz]
He_Level = 87[%]
Lock_Gain = 20



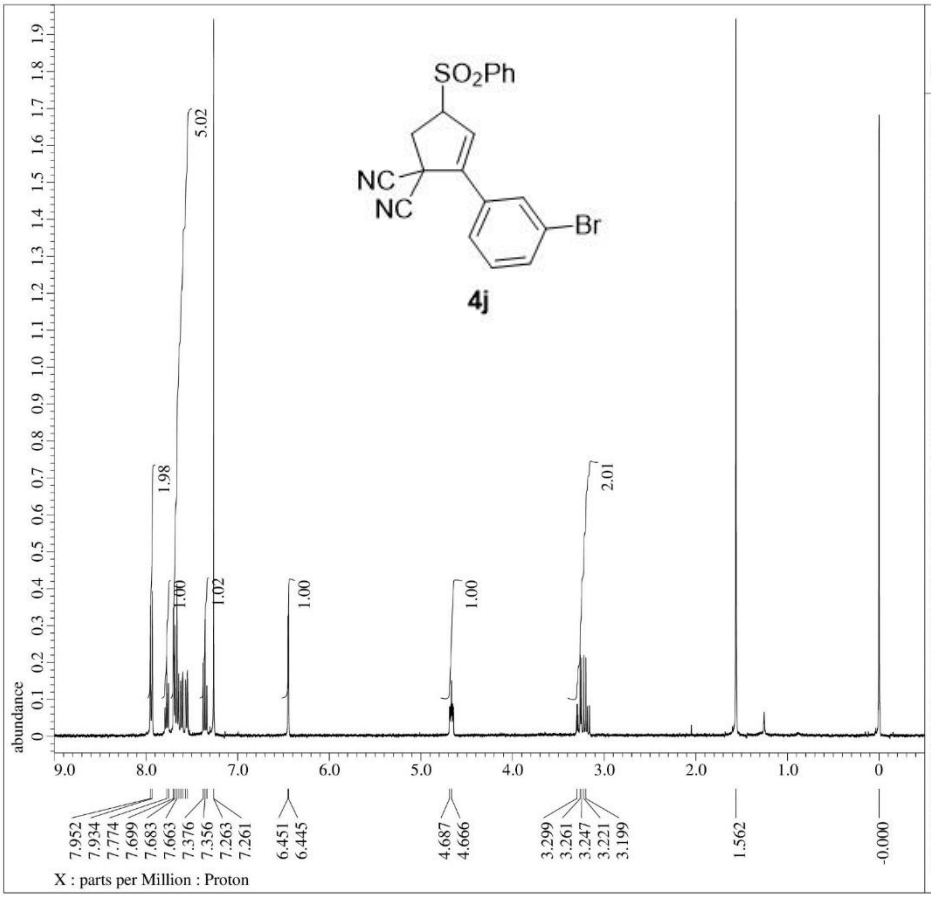
JEOL

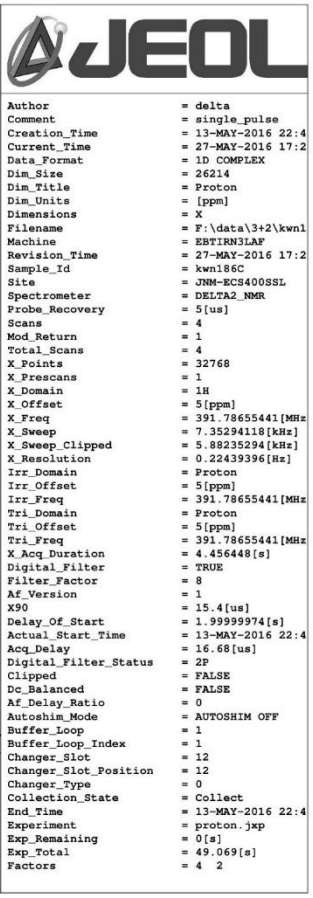
Author	= delta
Comment	= single_pulse
Creation_Time	= 13-MAY-2016 22:4
Current_Time	= 27-MAY-2016 17:3
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:3
Sample_Id	= kwn187C
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 13-MAY-2016 22:4
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 13
Changer_Slot_Position	= 13
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 13-MAY-2016 22:4
Experiment	= proton.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 49.083[s]
Factors	= 4 2

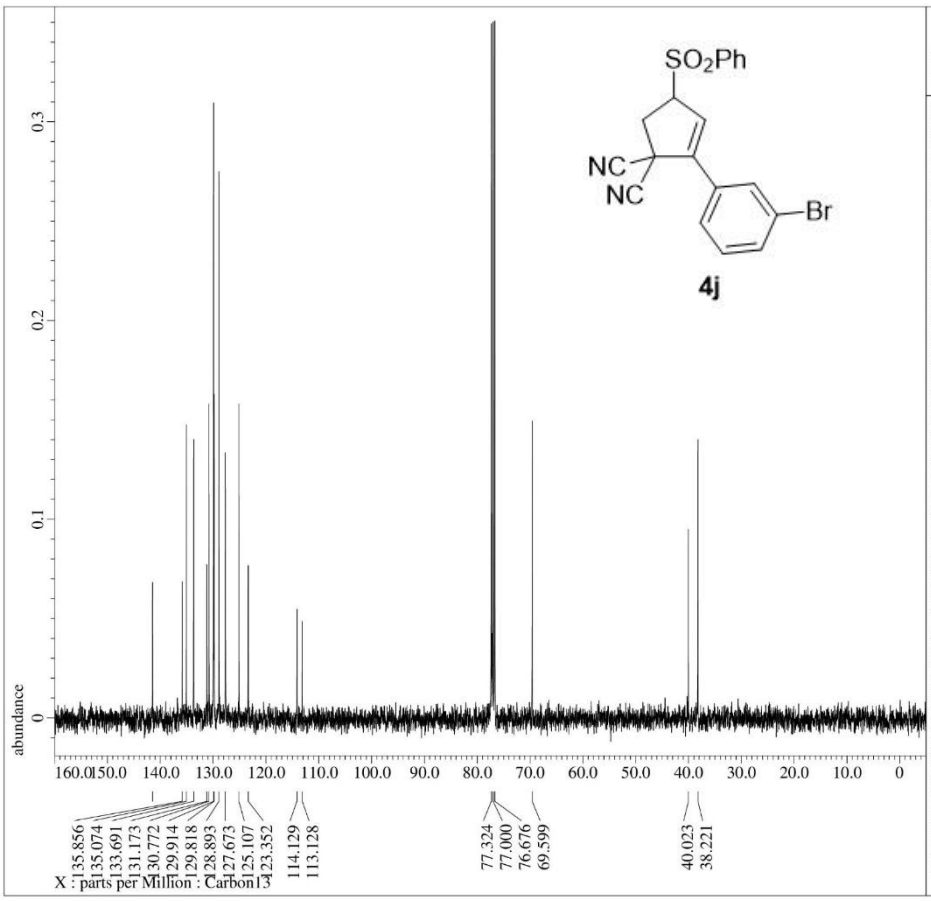


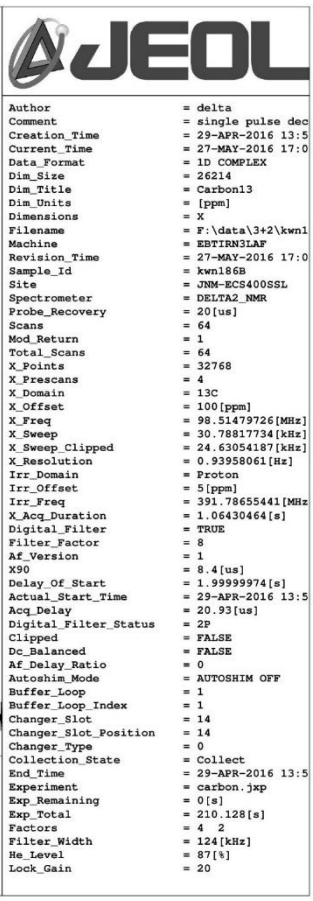
JEOL

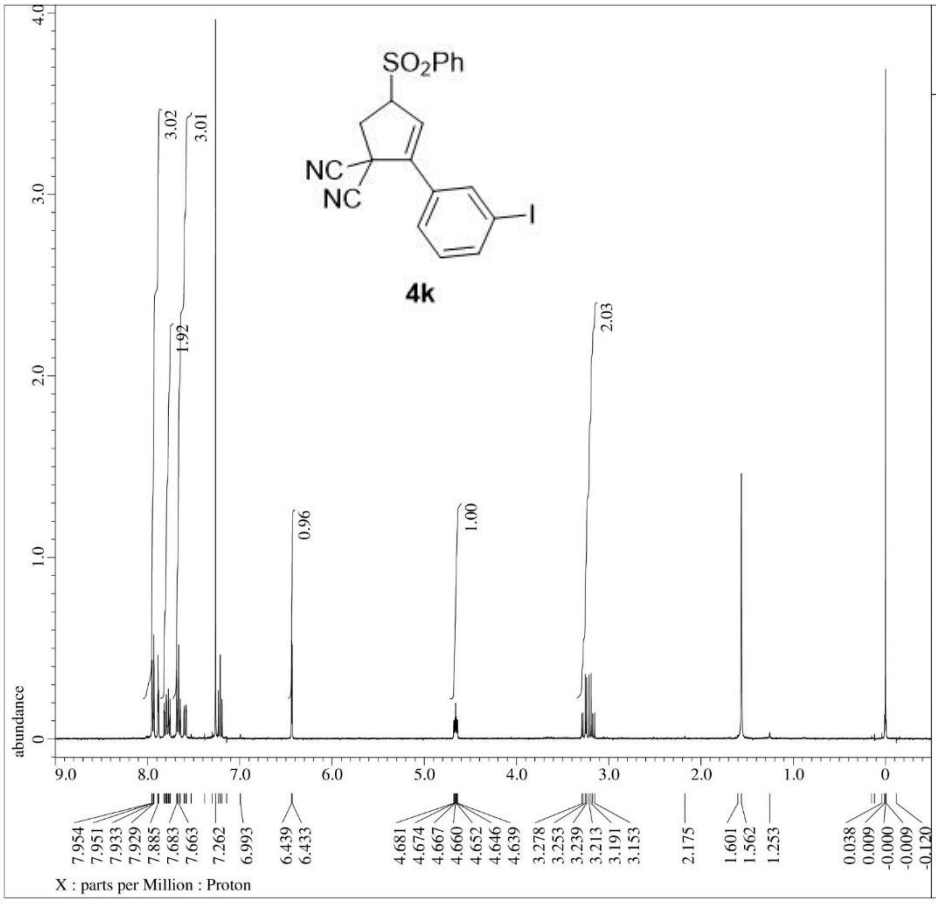
Author	= delta
Comment	= single pulse dec
Creation_Time	= 28-APR-2016 21:4
Current_Time	= 27-MAY-2016 17:0
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:0
Sample_Id	= kwn187B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 64
Mod_Return	= 1
Total_Scans	= 64
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 28-APR-2016 21:4
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 10
Changer_Slot_Position	= 10
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 28-APR-2016 21:4
Experiment	= carbon.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 210.181[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 87[%]
Lock_Gain	= 20


SO2Ph
NC
NC
Br
4j
5.02
1.98
1.00
1.02
1.00
1.00
2.01
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.952
7.934
7.774
7.699
7.683
7.663
7.376
7.356
7.263
7.261
6.451
6.445
4.687
4.666
3.299
3.261
3.247
3.221
3.199
1.562
-0.000
X : parts per Million : Proton

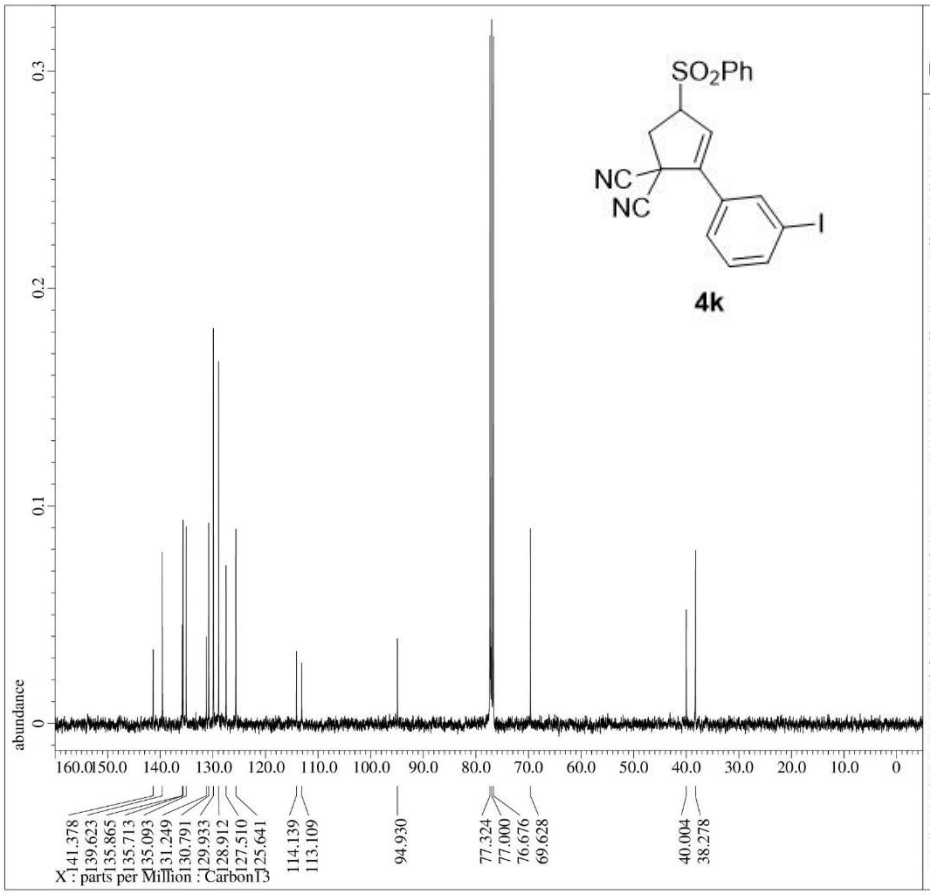

JEOL
Author = delta
Comment = single_pulse
Creation_Time = 13-MAY-2016 22:4
Current_Time = 27-MAY-2016 17:2
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn1
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:2
Sample_Id = kwn186C
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 5[us]
Scans = 4
Mod_Return = 1
Total_Scans = 4
X_Points = 32768
X_Prescans = 1
X_Domain = 1H
X_Offset = 5[ppm]
X_Freq = 391.78655441[MHz
X_Sweep = 7.35294118[kHz]
X_Sweep_Clipped = 5.88235294[kHz]
X_Resolution = 0.22439396[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
Tri_Domain = Proton
Tri_Offset = 5[ppm]
Tri_Freq = 391.78655441[MHz
X_Acq_Duration = 4.456448[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 15.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 13-MAY-2016 22:4
Acq_Delay = 16.68[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 12
Changer_Slot_Position = 12
Changer_Type = 0
Collection_State = Collect
End_Time = 13-MAY-2016 22:4
Experiment = proton.jxp
Exp_Remaining = 0[s]
Exp_Total = 49.069[s]
Factors = 4 2


SO2Ph
NC
NC
Br
4j
abundance
0.3
0.2
0.1
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
0
135.856
135.074
133.691
131.173
130.772
129.914
129.818
128.893
127.673
125.107
123.352
114.129
113.128
77.324
77.000
76.676
69.599
40.023
38.221
X : parts per Million : Carbon13

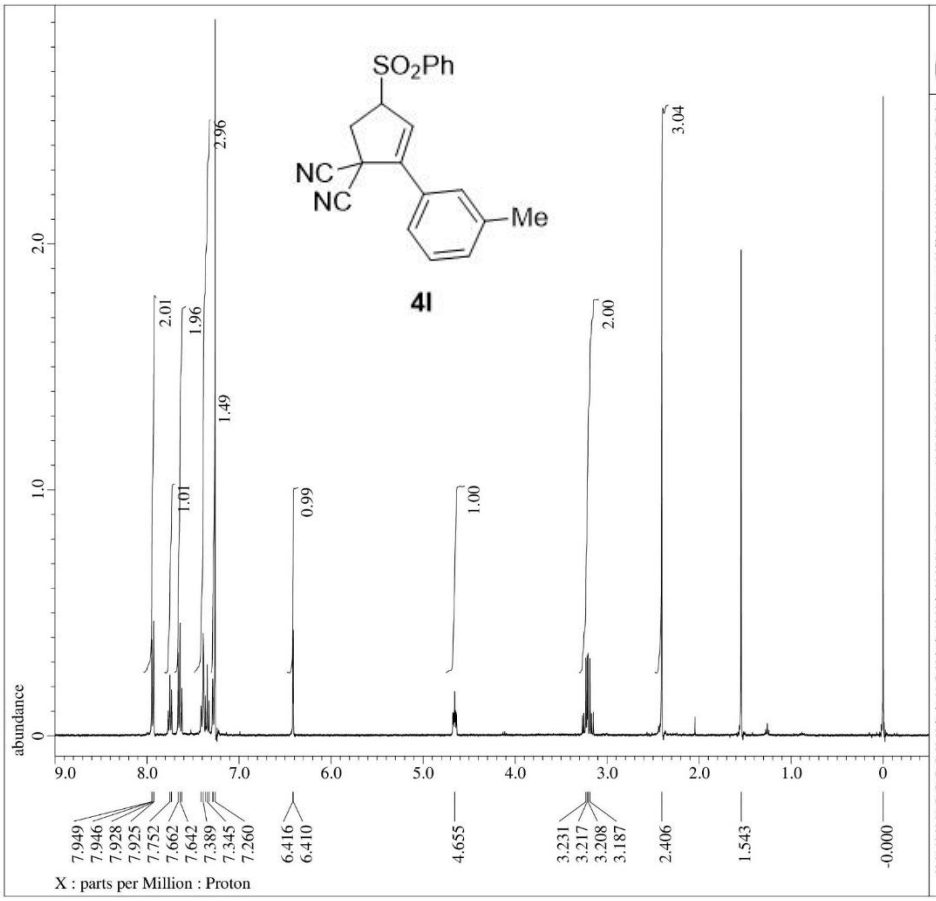

JEOL
Author = delta
Comment = single pulse dec
Creation_Time = 29-APR-2016 13:5
Current_Time = 27-MAY-2016 17:0
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn1
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:0
Sample_Id = kwn186B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 20[us]
Scans = 64
Mod_Return = 1
Total_Scans = 64
X_Points = 32768
X_Prescans = 4
X_Domain = 13C
X_Offset = 100[ppm]
X_Freq = 98.51479726[MHz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
X_Resolution = 0.93958061[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
X_Acq_Duration = 1.06430464[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 8.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 29-APR-2016 13:5
Acq_Delay = 20.93[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 14
Changer_Slot_Position = 14
Changer_Type = 0
Collection_State = Collect
End_Time = 29-APR-2016 13:5
Experiment = carbon.jxp
Exp_Remaining = 0[s]
Exp_Total = 210.128[s]
Factors = 4 2
Filter_Width = 124[kHz]
He_Level = 87[%]
Lock_Gain = 20

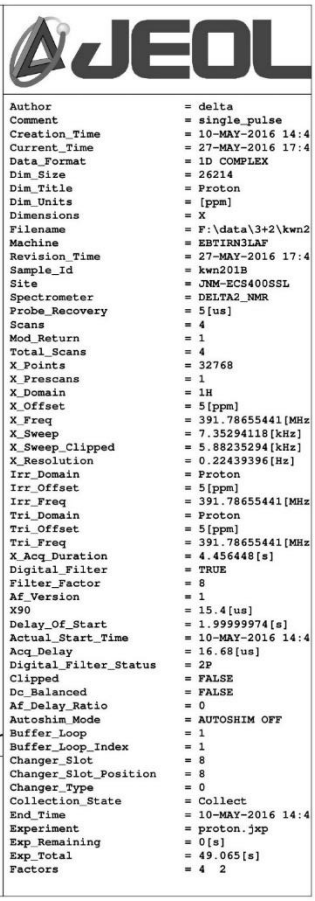


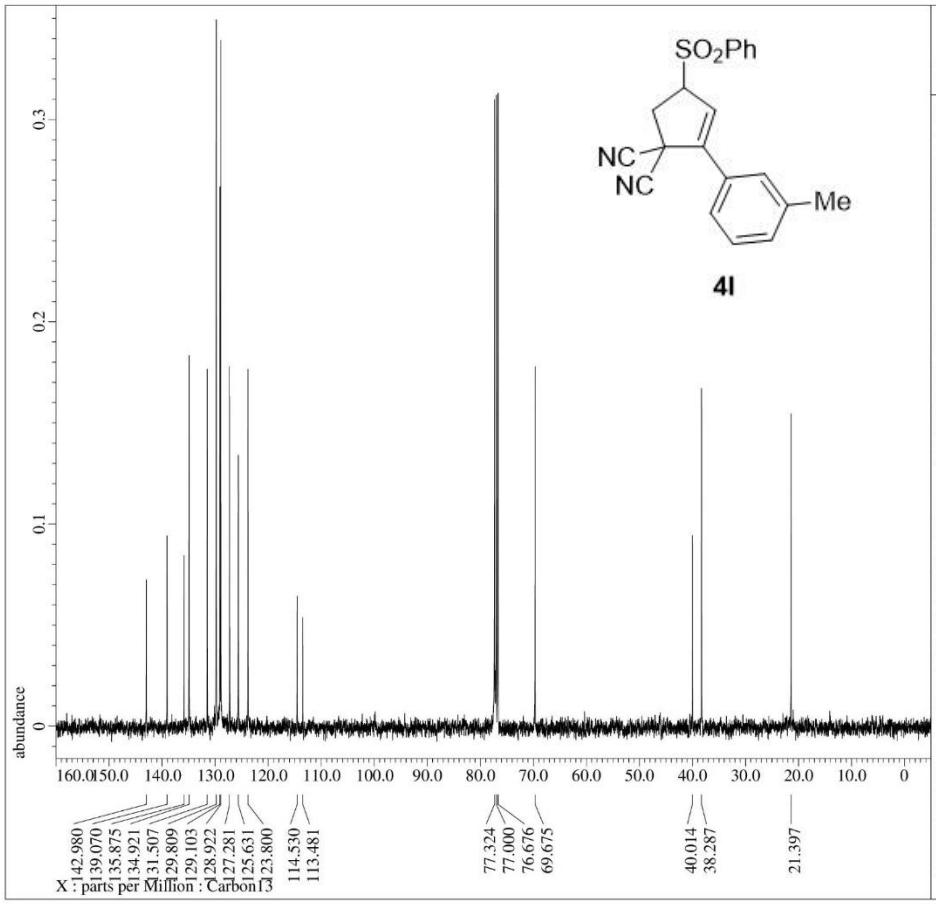
Author	= delta
Comment	= single_pulse
Creation_Time	= 11-MAY-2016 16:2
Current_Time	= 27-MAY-2016 18:2
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 18:2
Sample_Id	= kwn213B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 11-MAY-2016 16:2
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 7
Changer_Slot_Position	= 7
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 11-MAY-2016 16:2
Experiment	= proton.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 49.069[s]
Factors	= 4 2

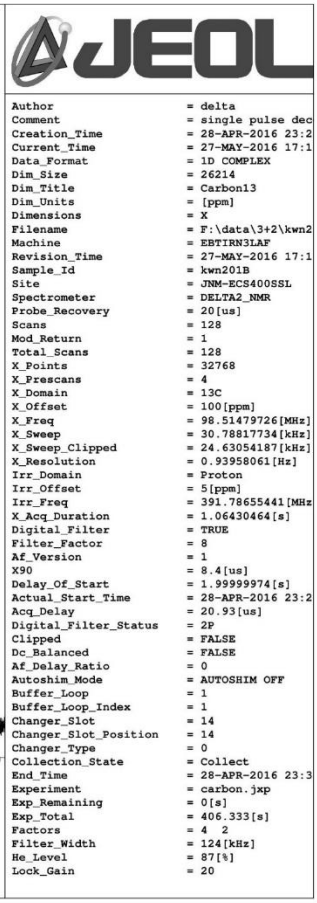


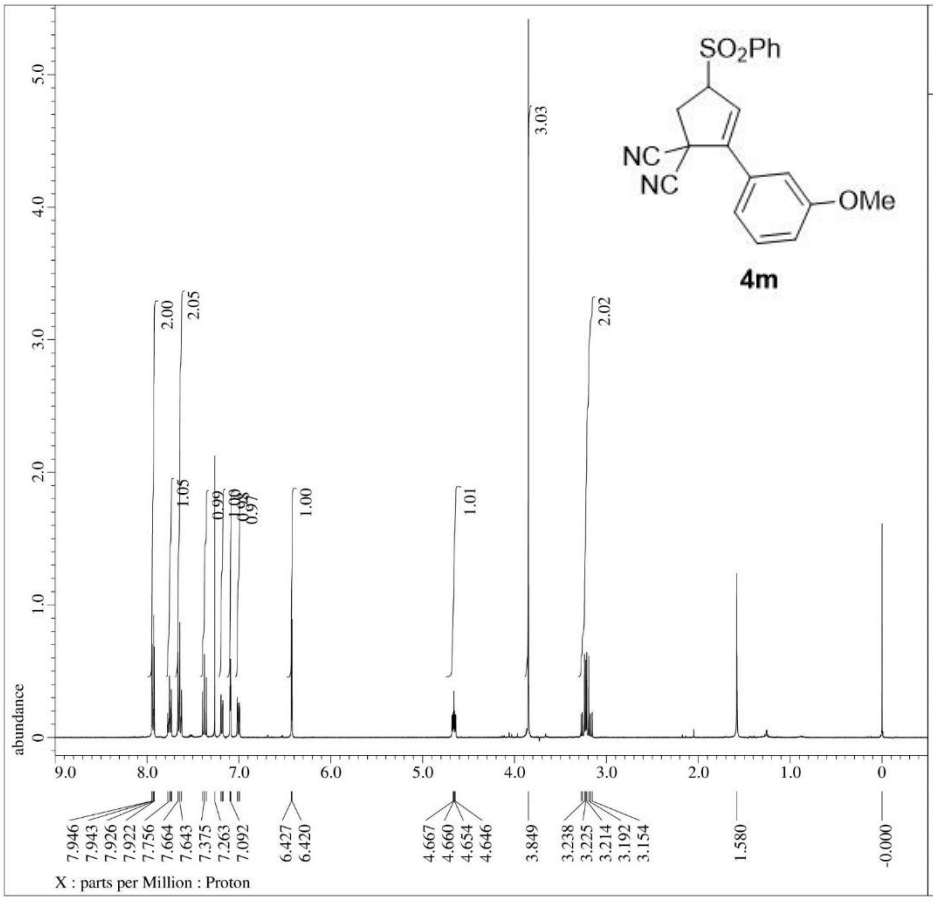
Author	= delta
Comment	= single pulse dec
Creation_Time	= 14-MAY-2016 09:2
Current_Time	= 27-MAY-2016 17:1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn2
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:1
Sample_Id	= kwn213B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 256
Mod_Return	= 1
Total_Scans	= 256
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 14-MAY-2016 09:2
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 12
Changer_Slot_Position	= 12
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 14-MAY-2016 09:3
Experiment	= carbon.jxp
Exp_Remaining	= 1[s]
Exp_Total	= 799[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 83[%]
Lock_Gain	= 20


SO2Ph
NC
NC
Me
4l
2.96
2.01
1.96
1.49
1.01
0.99
1.00
2.00
3.04
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.949
7.946
7.928
7.925
7.752
7.662
7.642
7.389
7.345
7.260
6.416
6.410
4.655
3.231
3.217
3.208
3.187
2.406
1.543
-0.000
X : parts per Million : Proton

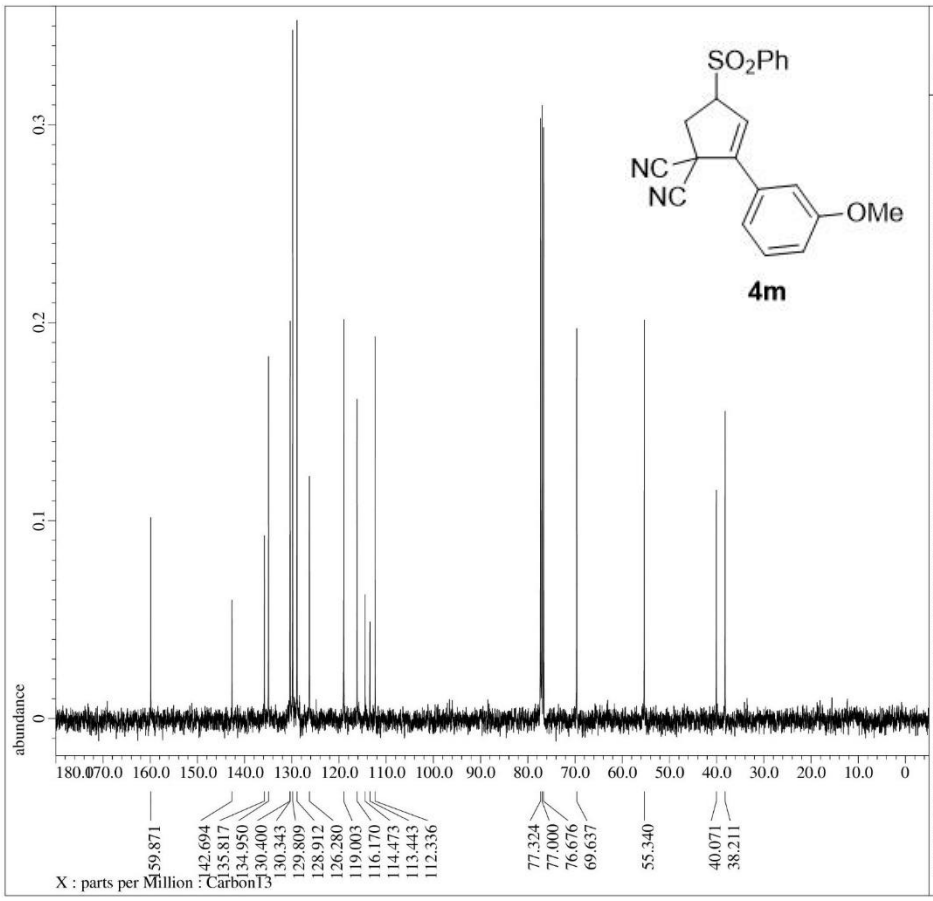

JEOL
Author = delta
Comment = single_pulse
Creation_Time = 10-MAY-2016 14:4
Current_Time = 27-MAY-2016 17:4
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn2
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:4
Sample_Id = kwn201B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 5[us]
Scans = 4
Mod_Return = 1
Total_Scans = 4
X_Points = 32768
X_Prescans = 1
X_Domain = 1H
X_Offset = 5[ppm]
X_Freq = 391.78655441[MHz
X_Sweep = 7.35294118[kHz]
X_Sweep_Clipped = 5.88235294[kHz]
X_Resolution = 0.22439396[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
Tri_Domain = Proton
Tri_Offset = 5[ppm]
Tri_Freq = 391.78655441[MHz
X_Acq_Duration = 4.456448[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 15.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 10-MAY-2016 14:4
Acq_Delay = 16.68[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 8
Changer_Slot_Position = 8
Changer_Type = 0
Collection_State = Collect
End_Time = 10-MAY-2016 14:4
Experiment = proton.jxp
Exp_Remaining = 0[s]
Exp_Total = 49.065[s]
Factors = 4 2


SO2Ph
NC
NC
Me
4l
abundance
0.3
0.2
0.1
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
0
142.980
139.070
135.875
134.921
131.507
129.809
129.103
128.922
127.281
125.631
123.800
114.530
113.481
77.324
77.000
76.676
69.675
40.014
38.287
21.397
X : parts per Million : Carbon13

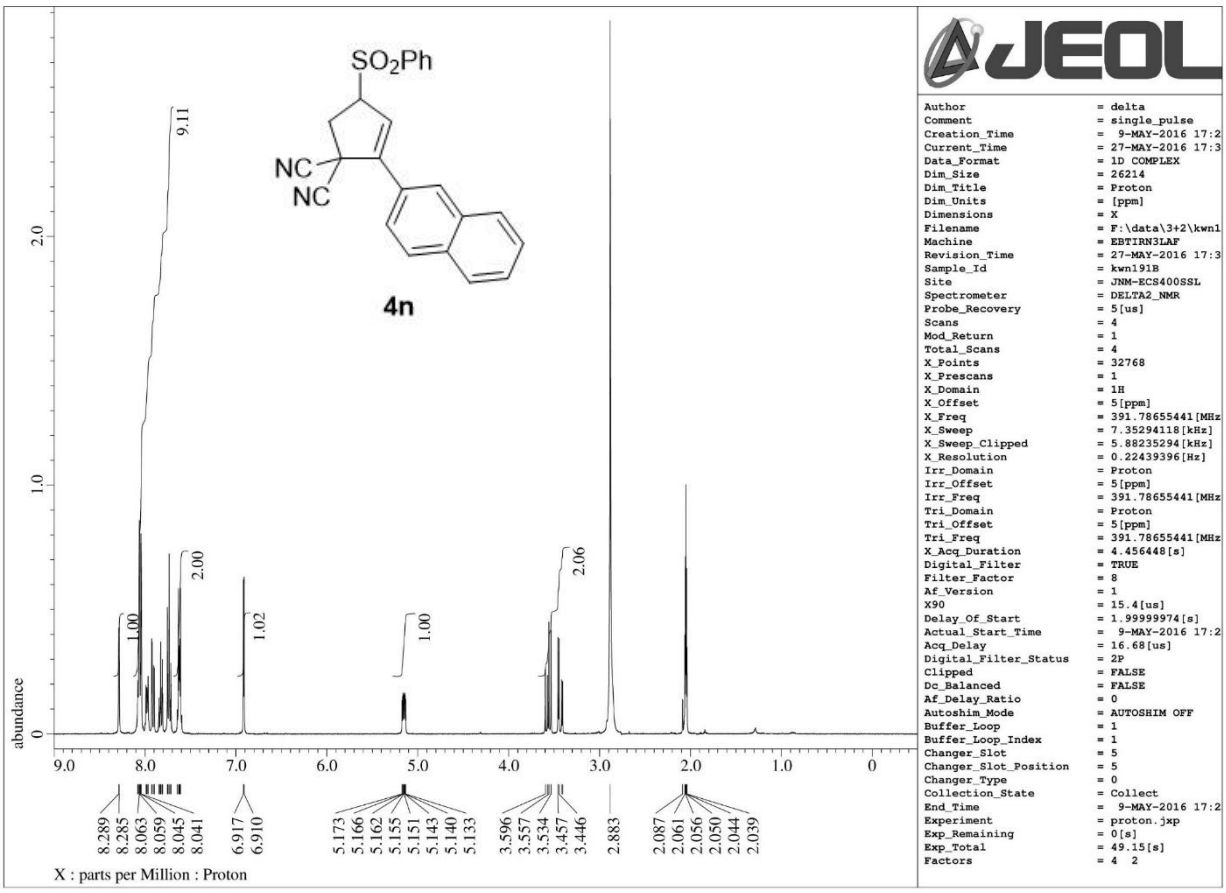

JEOL
Author = delta
Comment = single pulse dec
Creation_Time = 28-APR-2016 23:2
Current_Time = 27-MAY-2016 17:1
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn2
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:1
Sample_Id = kwn201B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 20[us]
Scans = 128
Mod_Return = 1
Total_Scans = 128
X_Points = 32768
X_Prescans = 4
X_Domain = 13C
X_Offset = 100[ppm]
X_Freq = 98.51479726[MHz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
X_Resolution = 0.93958061[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
X_Acq_Duration = 1.06430464[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 8.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 28-APR-2016 23:2
Acq_Delay = 20.93[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 14
Changer_Slot_Position = 14
Changer_Type = 0
Collection_State = Collect
End_Time = 28-APR-2016 23:3
Experiment = carbon.jxp
Exp_Remaining = 0[s]
Exp_Total = 406.333[s]
Factors = 4 2
Filter_Width = 124[kHz]
He_Level = 87[%]
Lock_Gain = 20



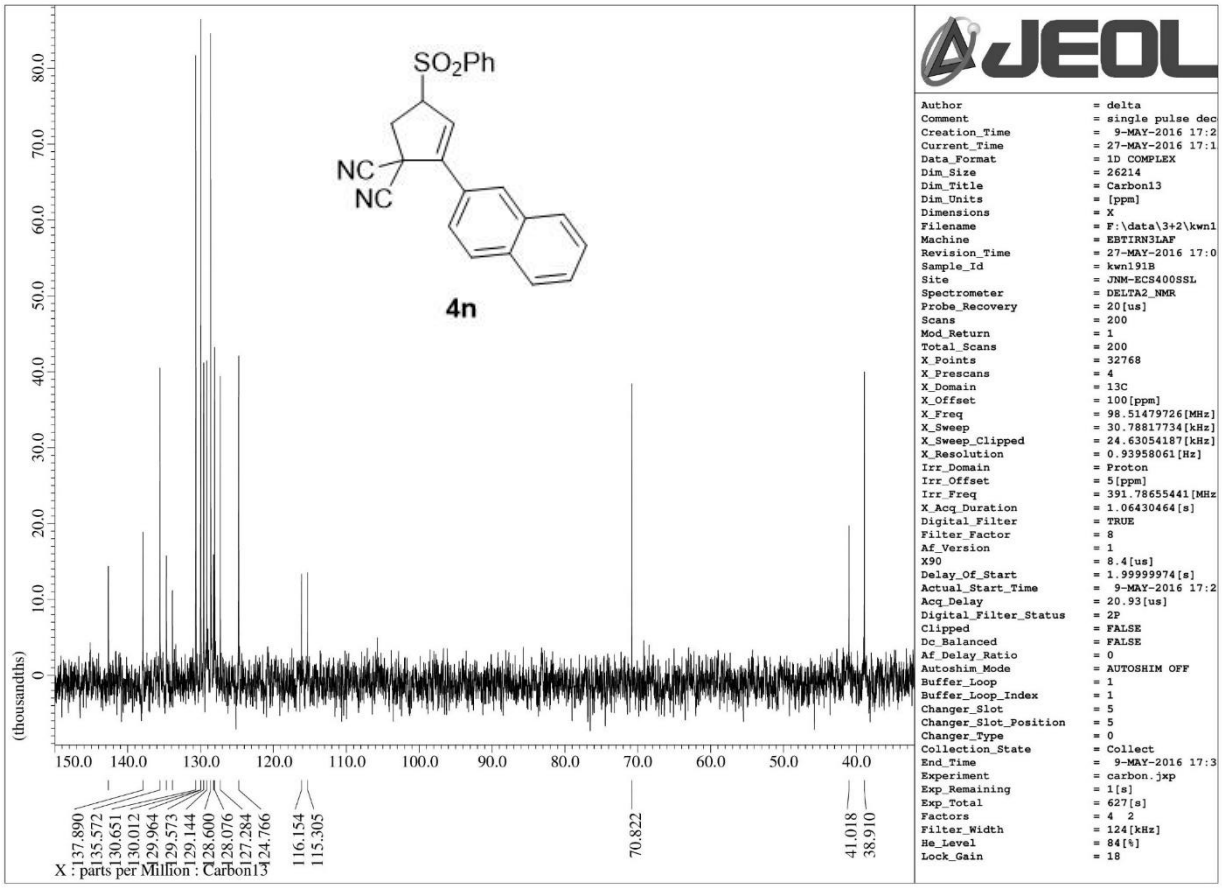
Author = delta
Comment = single_pulse
Creation_Time = 11-MAY-2016 16:4
Current_Time = 27-MAY-2016 18:1
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn2
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 18:1
Sample_Id = kwn202C
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 5[us]
Scans = 4
Mod_Return = 1
Total_Scans = 4
X_Points = 32768
X_Prescans = 1
X_Domain = 1H
X_Offset = 5[ppm]
X_Freq = 391.78655441[MHz
X_Sweep = 7.35294118[kHz]
X_Sweep_Clipped = 5.88235294[kHz]
X_Resolution = 0.22439396[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
Tri_Domain = Proton
Tri_Offset = 5[ppm]
Tri_Freq = 391.78655441[MHz
X_Acq_Duration = 4.456448[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 15.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 11-MAY-2016 16:4
Acq_Delay = 16.68[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 9
Changer_Slot_Position = 9
Changer_Type = 0
Collection_State = Collect
End_Time = 11-MAY-2016 16:4
Experiment = proton.jxp
Exp_Remaining = 0[s]
Exp_Total = 49.018[s]
Factors = 4 2



Author = delta
Comment = single pulse dec
Creation_Time = 29-APR-2016 12:2
Current_Time = 27-MAY-2016 17:1
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Filename = F:\data\3+2\kwn2
Machine = EBTIRN3LAF
Revision_Time = 27-MAY-2016 17:1
Sample_Id = kwn202B
Site = JNM-ECS400SSL
Spectrometer = DELTA2_NMR
Probe_Recovery = 20[us]
Scans = 64
Mod_Return = 1
Total_Scans = 64
X_Points = 32768
X_Prescans = 4
X_Domain = 13C
X_Offset = 100[ppm]
X_Freq = 98.51479726[MHz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
X_Resolution = 0.93958061[Hz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Irr_Freq = 391.78655441[MHz
X_Acq_Duration = 1.06430464[s]
Digital_Filter = TRUE
Filter_Factor = 8
Af_Version = 1
X90 = 8.4[us]
Delay_Of_Start = 1.99999974[s]
Actual_Start_Time = 29-APR-2016 12:2
Acq_Delay = 20.93[us]
Digital_Filter_Status = 2P
Clipped = FALSE
Dc_Balanced = FALSE
Af_Delay_Ratio = 0
Autoshim_Mode = AUTOSHIM OFF
Buffer_Loop = 1
Buffer_Loop_Index = 1
Changer_Slot = 9
Changer_Slot_Position = 9
Changer_Type = 0
Collection_State = Collect
End_Time = 29-APR-2016 12:3
Experiment = carbon.jxp
Exp_Remaining = 0[s]
Exp_Total = 210.13[s]
Factors = 4 2
Filter_Width = 124[kHz]
He_Level = 87[%]
Lock_Gain = 20



Author	= delta
Comment	= single_pulse
Creation_Time	= 9-MAY-2016 17:2
Current_Time	= 27-MAY-2016 17:3
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:3
Sample_Id	= kwn191B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 5[us]
Scans	= 4
Mod_Return	= 1
Total_Scans	= 4
X_Points	= 32768
X_Prescans	= 1
X_Domain	= 1H
X_Offset	= 5[ppm]
X_Freq	= 391.78655441[MHz
X_Sweep	= 7.35294118[kHz]
X_Sweep_Clipped	= 5.88235294[kHz]
X_Resolution	= 0.22439396[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
Tri_Domain	= Proton
Tri_Offset	= 5[ppm]
Tri_Freq	= 391.78655441[MHz
X_Acq_Duration	= 4.456448[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 15.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 9-MAY-2016 17:2
Acq_Delay	= 16.68[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 5
Changer_Slot_Position	= 5
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 9-MAY-2016 17:2
Experiment	= proton.jxp
Exp_Remaining	= 0[s]
Exp_Total	= 49.15[s]
Factors	= 4 2

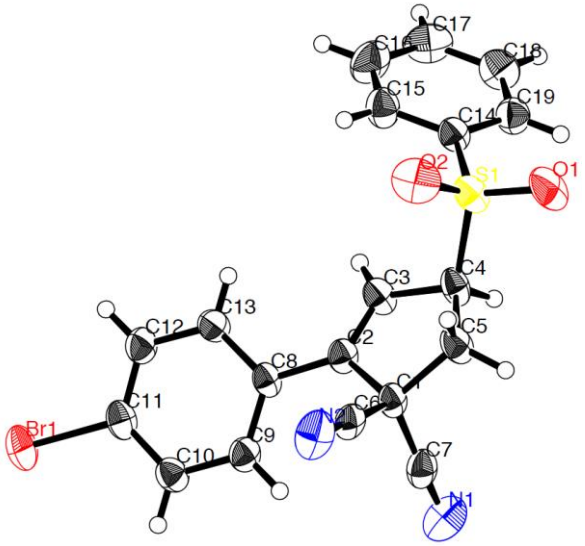


Author	= delta
Comment	= single pulse dec
Creation_Time	= 9-MAY-2016 17:2
Current_Time	= 27-MAY-2016 17:1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Filename	= F:\data\3+2\kwn1
Machine	= EBTIRN3LAF
Revision_Time	= 27-MAY-2016 17:0
Sample_Id	= kwn191B
Site	= JNM-ECS400SSL
Spectrometer	= DELTA2_NMR
Probe_Recovery	= 20[us]
Scans	= 200
Mod_Return	= 1
Total_Scans	= 200
X_Points	= 32768
X_Prescans	= 4
X_Domain	= 13C
X_Offset	= 100[ppm]
X_Freq	= 98.51479726[MHz]
X_Sweep	= 30.78817734[kHz]
X_Sweep_Clipped	= 24.63054187[kHz]
X_Resolution	= 0.93958061[Hz]
Irr_Domain	= Proton
Irr_Offset	= 5[ppm]
Irr_Freq	= 391.78655441[MHz
X_Acq_Duration	= 1.06430464[s]
Digital_Filter	= TRUE
Filter_Factor	= 8
Af_Version	= 1
X90	= 8.4[us]
Delay_Of_Start	= 1.99999974[s]
Actual_Start_Time	= 9-MAY-2016 17:2
Acq_Delay	= 20.93[us]
Digital_Filter_Status	= 2P
Clipped	= FALSE
Dc_Balanced	= FALSE
Af_Delay_Ratio	= 0
Autoshim_Mode	= AUTOSHIM OFF
Buffer_Loop	= 1
Buffer_Loop_Index	= 1
Changer_Slot	= 5
Changer_Slot_Position	= 5
Changer_Type	= 0
Collection_State	= Collect
End_Time	= 9-MAY-2016 17:3
Experiment	= carbon.jxp
Exp_Remaining	= 1[s]
Exp_Total	= 627[s]
Factors	= 4 2
Filter_Width	= 124[kHz]
He_Level	= 84[%]
Lock_Gain	= 18

5. Single Crystal X-Ray Analysis of 4d

A colorless prism crystal having approximate dimensions of 0.500 x 0.500 x 0.300 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-*Kα* radiation. The data were collected at a temperature of 23 + 1 °C to a maximum 2θ value of 54.9°. The structures were solved by direct method (SIR-92)[2], and hydrogen atoms were placed at the calculation. A full-matrix least-squares technique was using with anisotropic thermal parameters for non-hydrogen atoms and riding model for hydrogen atoms. All calculations were performed using the Crystal Structure[3,4] crystallographic software package.

4d; $C_{19}H_{13}BrN_2O_2S$, *M*=413.29, monoclinic, space group $P2_1/n$ (#14), *a*=10.2777(8), *b*=10.2776(10), *c*=16.4290(12) Å, *V*=1732.3(2) Å^3, *Dc*=1.585 gcm^{-3}, *Z*=4, *R*=0.0514 for 2543 observed reflections ($I > 2.00\sigma(I)$), *Rw*=0.0644.



Deposition number CCDC-1485660 for compound **4d**. Free copies of the data can be obtained via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK; Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

---

[2] SIR92: A. Altomare, G. Cascarano, C. Giacovazzo, and A. Guagliardi, *J. Appl. Cryst.*, 1993, **26**, 343.

[3] CrystalStructure 4.2.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2016). Tokyo 196-8666, Japan.

[4] CRYSTALS Issue 11: J. R. Carruthers, J. S. Rollett, P. W. Betteridge, D. Kinna, L. Pearce, A. Larsen, and E. Gabe, Chemical Crystallography Laboratory, Oxford, UK. (1999)