Supplementary Information

Catalytic [3 + 2] annulation of ketimines with alkynes via C–H activation by a cationic iridium(cod) complex

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1. General

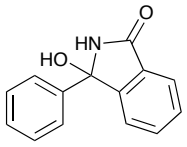
All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen. NMR spectra were recorded on a JEOL JNM ECA-600 spectrometer (600 MHz for ^{1}H, 150 MHz for ^{13}C). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) and DMSO-d_6 (δ 2.49) for ^{1}H NMR, and $CDCl_3$ (δ 77.00) for ^{13}C NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: m, multiplet: br, broad. High-resolution mass spectra were obtained with a Bruker micrOTOF spectrometer. Preparative thin-layer chromatography was performed with Silica Gel 60 PF_{254} (Merck). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque. Preparative recycling gel permeation chromatography was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as an eluent.

2. Materials

Toluene was purified by passing through a neutral alumina column under N_2. Iridium complexes, $[IrCl(cod)]_2$[1] and $[IrCl(coe)_2]_2$,[2] were prepared according to the reported procedures. $NaBAr^F_4$ was prepared according to the reported procedures.[3]

3. Preparation of 1

Compounds **1a** (CAS: 6637-53-2),[4] **1b** (CAS: 39127-18-9),[4] **1c** (CAS: 92553-10-1),[4] **1d**,[4] **1e** (CAS: 956-92-3),[4] **1f** (CAS: 53440-57-6),[5] **1g** (CAS: 10399-00-5),[6] and **1h** (CAS: 1002116-11-1),[6] were prepared according to the reported procedures. A typical procedure for the synthesis of **1a** is shown below.



1a

Compound 1a. To a solution of phthalimide (CAS: 85-41-6) (2.94 g, 20.0 mmol) in CH_2Cl_2 (50 mL) was slowly added PhMgBr (1.0 M in THF, 60 mL, 60 mmol) at 0 °C, and the mixture was stirred at room temperature for 5 h. Saturated NH_4Cl solution (50 mL) was added and the mixture was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The solid residue was recrystallized from hot CH_2Cl_2 and hexane to give **1a** (4.08 g, 18.1 mmol, 91% yield).

1 J. L. Herde, J. C. Lambert and C. V. Senoff, *Inorg. Synth.,* 1974, **15**, 18.
2 R. Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.,* 1985, **23**, 126.
3 M. Brookhart, B. Grant and A. F. Volpe, Jr., *Organometallics,* 1992, **11**, 3920.
4 T. Nishimura, A. Noishiki, Y. Ebe and T. Hayashi, *Angew. Chem., Int. Ed.,* 2013, **52**, 1777.
5 T. Nishimura, A. Noishiki, G. C. Tsui and T. Hayashi, *J. Am. Chem. Soc.*, 2012, **134**, 5056.
6 R. N. Ram and A. A. Khan, *Synth. Commun.,* 2001, **31**, 841.

4. Preparation of alkynes 2

Diphenylacetylene (**2m**), 4-octyne (**2s**), 1-phenyl-1-propyne (**2v**), and (*tert*-butyldimethylsilyl)acetylene (**2w**) were purchased from commercial suppliers and used as received. Alkynes **2n** (CAS: 2789-88-0),[7] **2o** (CAS: 2132-62-9), **2p** (CAS: 119757-51-6), **2q** (CAS: 2789-89-1), **2r** (CAS: 38399-13-2), **2t** (CAS: 40474-01-9),[8] and **2u** (CAS: 21375-88-2) were prepared according to the reported procedures.

5. A general procedure for Ir-catalyzed [3 + 2] annulation of 3-hydroxy-3-phenylisoindolin-1-one (1a) with diphenylacetylene (2m) (Table 1)

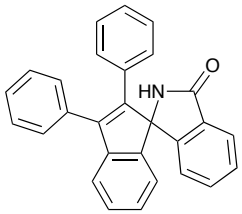
3-Hydroxy-3-phenylisoindolin-1-one (**1a**, 45.0 mg, 0.20 mmol), $NaBAr^F_4$ (18.4 mg calculated as the dihydrate, 0.020 mmol, 10 mol%), diphenylacetylene (**2m**, 53.5 mg, 0.30 mmol), and an iridium complex (0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.8 mL) was added and the Schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. The mixture was passed through a short column of alumina with EtOAc as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:1) to give **3am**.

6. A general procedure for Ir-catalyzed annulation of ketimines 1 with alkynes 2 (Scheme 2 and Scheme 3)

Ketimine **1** (0.20 mmol), $NaBAr^F_4$ (18.4 mg calculated as the dihydrate, 0.020 mmol, 10 mol%), alkyne **2** (for solid compounds, 0.30 mmol), and $[IrCl(cod)]_2$ (3.4 mg, 0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.8 mL) and alkyne **2** (for liquid compounds, 0.30 mmol) were added successively. The Schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. The mixture was passed through a short column of alumina with EtOAc as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel to give **3**.

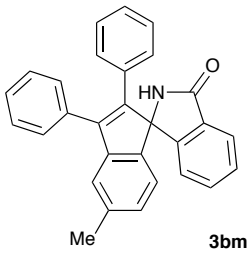
7 T. Yamakawa and N. Yoshikai, *Org. Lett.,* 2013, **15**, 196.
8 M. J. Mio, L. C. Kopel, J. B. Braun, T. L. Gadzikwa, K. L. Hull, R. D. Brisbois, C. J. Markworth and P. A. Grieco, *Org. Lett.,* 2002, **4**, 3199.

7. Characterization of the products



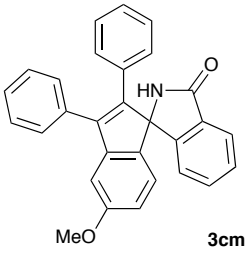
3am

Compound 3am. A solution of EtOAc/hexane (1:1) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 6.05 (br s, 1H), 6.70 (d, *J* = 7.5 Hz, 2H), 6.98 (t, *J* = 7.5 Hz, 2H), 7.03 (d, *J* = 7.5 Hz, 1H), 7.05 (t, *J* = 7.5 Hz, 1H), 7.10–7.13 (m, 1H), 7.18 (t, *J* = 7.5 Hz, 1H), 7.31–7.40 (m, 7H), 7.45–7.48 (m, 2H), 7.85–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 74.9, 121.3, 121.6, 122.6, 124.2, 127.1, 127.6, 128.01, 128.04, 128.6, 128.7, 128.8, 128.9, 129.3, 131.9, 132.7, 133.3, 134.0, 142.7, 143.3, 143.5, 144.8, 146.8, 171.0. HRMS (ESI) calcd for $C_{28}H_{19}NNaO$ $(M+Na)^+$ 408.1359, found 408.1358.



3bm

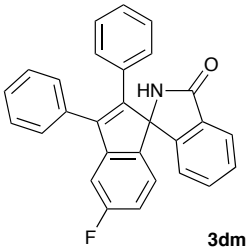
Compound 3bm. A solution of EtOAc/hexane (1:1) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 2.37 (s, 3H), 6.18 (br s, 1H), 6.72 (d, *J* = 7.5 Hz, 2H), 6.92 (d, *J* = 7.5 Hz, 1H), 6.96–7.01 (m, 3H), 7.04 (t, *J* = 7.5 Hz, 1H), 7.10–7.14 (m, 1H), 7.19 (s, 1H), 7.32–7.40 (m, 5H), 7.43–7.48 (m, 2H), 7.84–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 21.6, 74.7, 121.6, 122.1, 122.3, 124.2, 127.5, 127.7, 127.9, 128.0, 128.5, 128.6, 128.8, 129.3, 131.9, 132.6, 133.4, 134.1, 138.9, 141.8, 142.7, 143.5, 143.7, 147.1, 171.0. HRMS (ESI) calcd for $C_{29}H_{21}NNaO$ $(M+Na)^+$ 422.1515, found 422.1517.



3cm

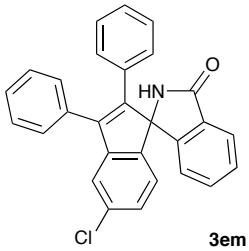
Compound 3cm. A solution of EtOAc/hexane (1:2) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 3.79 (s, 3H), 6.23 (br s, 1H), 6.68–6.73 (m, 3H), 6.92–6.95 (m, 2H), 6.97 (t, *J* = 7.5 Hz, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 1H), 7.31–7.39 (m, 5H), 7.43–

7.49 (m, 2H), 7.84–7.87 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 55.6, 74.4, 108.0, 111.7, 121.6, 132.3, 124.1, 127.6, 127.98, 128.00, 128.5, 128.6, 128.8, 129.2, 131.8, 132.7, 133.3, 133.9, 136.3, 142.3, 144.6, 145.1, 147.2, 160.7, 171.0. HRMS (ESI) calcd for $C_{29}H_{21}NNaO_2$ $(M+Na)^+$ 438.1465, found 438.1453.



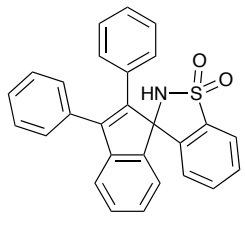
3dm

Compound 3dm. A solution of EtOAc/$CHCl_3$ (1:10) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 6.11 (br s, 1H), 6.70 (d, J = 7.5 Hz, 2H), 6.86 (t, J = 7.5 Hz, 1H), 6.95–7.01 (m, 3H), 7.04–7.10 (m, 2H), 7.10–7.14 (m, 1H), 7.33–7.40 (m, 5H), 7.46–7.51 (m, 2H), 7.85–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 74.3, 109.0 (d, $J_{F\text{-}C}$ = 24 Hz), 113.5 (d, $J_{F\text{-}C}$ = 24 Hz), 121.5, 123.7 (d, $J_{F\text{-}C}$ = 9 Hz), 124.3, 127.9, 128.1, 128.3, 128.7, 128.77, 128.83, 129.1, 131.8, 132.8, 132.9, 133.4, 139.9, 141.7, 145.2, 145.7 (d, $J_{F\text{-}C}$ = 9 Hz), 146.4, 163.7 (d, $J_{F\text{-}C}$ = 247 Hz), 170.9. HRMS (ESI) calcd for $C_{28}H_{18}FNNaO$ $(M+Na)^+$ 426.1265, found 426.1261.



3em

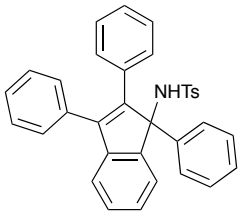
Compound 3em. A solution of EtOAc/hexane (1:1) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 6.45 (br s, 1H), 6.70 (d, J = 7.5 Hz, 2H), 6.89–6.94 (m, 1H), 6.97 (t, J = 7.5 Hz, 2H), 7.05 (t, J = 7.5 Hz, 1H), 7.09–7.15 (m, 2H), 7.31–7.40 (m, 6H), 7.44–7.50 (m, 2H), 7.83–7.87 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 74.6, 121.6, 121.8, 123.7, 124.4, 127.0, 128.0, 128.2, 128.4, 128.8, 128.9, 129.0, 129.3, 131.9, 133.0, 133.4, 135.1, 141.8, 143.1, 145.0, 145.4, 146.3, 171.1. HRMS (ESI) calcd for $C_{28}H_{18}ClNNaO$ $(M+Na)^+$ 442.0969, found 442.0965.



3fm

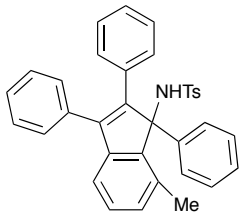
Compound 3fm. A solution of $CHCl_3$/EtOAc (12:1) was used as an eluent for preparative

TLC. ^{1}H NMR ($CDCl_3$) δ 4.85 (br s, 1H), 6.90 (d, *J* = 7.2 Hz, 2H), 7.07 (t, *J* = 7.2 Hz, 2H), 7.11–7.16 (m, 2H), 7.22 (t, *J* = 7.2 Hz, 1H), 7.32–7.43 (m, 7H), 7.45 (d, *J* = 7.2 Hz, 1H), 7.53 (t, *J* = 7.2 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.87 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 75.3, 121.6, 121.9, 123.4, 123.5, 127.8, 128.3, 128.40, 128.43, 128.9, 129.27, 129.31, 129.33, 129.9, 132.4, 133.8, 133.9, 135.4, 139.7, 142.3, 143.0, 143.4, 147.5. HRMS (ESI) calcd for $C_{27}H_{19}NNaO_2S$ $(M+Na)^+$ 444.1029, found 444.1024.



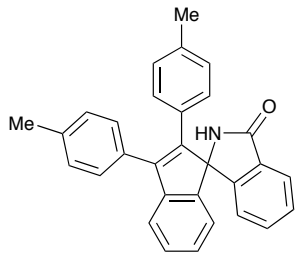
3gm

Compound 3gm. $CHCl_3$ was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 2.42 (s, 3H), 5.36 (s, 1H), 6.71 (d, *J* = 8.1 Hz, 2H), 7.01–7.07 (m, 8H), 7.11–7.15 (m, 3H), 7.16–7.21 (m, 2H), 7.27–7.37 (m, 6H), 7.60 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.5, 73.9, 120.5, 124.9, 126.1, 127.6, 127.74, 127.77, 127.83, 127.9, 128.2, 128.3, 128.5, 128.8, 129.2, 129.6, 132.8, 134.2, 137.6, 141.7, 141.8, 142.8, 143.3, 144.3, 144.6. HRMS (ESI) calcd for $C_{34}H_{27}NNaO_2S$ $(M+Na)^+$ 536.1655, found 536.1650.



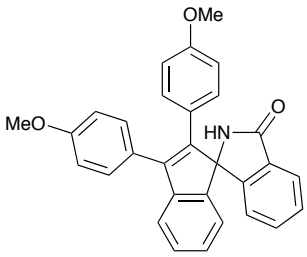
3hm

Compound 3hm. $CHCl_3$ was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 1.74 (s, 3H), 2.44 (s, 3H), 5.80 (br s, 1H), 6.71 (d, *J* = 7.5 Hz, 2H), 6.88–6.93 (m, 3H), 7.00–7.05 (m, 5H), 7.06–7.12 (m, 3H), 7.15–7.20 (m, 3H), 7.24–7.30 (m, 4H), 7.41 (t, *J* = 7.5 Hz, 1H), 8.40 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 19.7, 21.6, 74.4, 120.2, 126.3, 126.8, 127.1, 127.9, 128.0, 128.4, 128.49, 128.54, 128.7, 128.8, 128.9, 129.0, 129.5, 129.7, 132.4, 134.0, 134.1, 135.1, 138.7, 139.4, 140.4, 140.8, 142.4, 144.8, 145.4. HRMS (ESI) calcd for $C_{35}H_{29}NNaO_2S$ $(M+Na)^+$ 550.1811, found 550.1804.



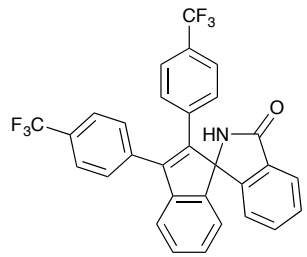
3an

Compound 3an. A solution of EtOAc/hexane (1:2) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 2.16 (s, 3H), 2.38 (s, 3H), 6.15 (br s, 1H), 6.61 (d, *J* = 7.5 Hz, 2H), 6.79 (d, *J* = 7.5 Hz, 2H), 7.01 (d, *J* = 7.5 Hz, 1H), 7.08–7.12 (m, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 7.5 Hz, 1H), 7.44–7.48 (m, 2H), 7.85–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 21.1, 21.3, 74.9, 121.2, 121.6, 122.4, 124.1, 126.8, 128.5, 128.6, 128.8, 129.2, 129.3, 130.4, 131.1, 131.9, 132.7, 137.2, 137.7, 142.1, 142.8, 143.8, 144.7, 147.1, 171.1. HRMS (ESI) calcd for $C_{30}H_{23}NNaO$ $(M+Na)^+$ 436.1672, found 436.1672.



3ao

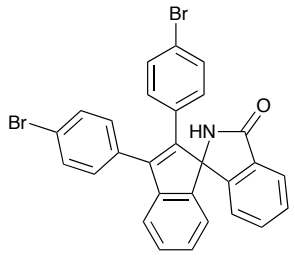
Compound 3ao. A solution of EtOAc/hexane (1:1) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 3.66 (s, 3H), 3.83 (s, 3H), 6.12 (br s, 1H), 6.53 (d, *J* = 8.9 Hz, 2H), 6.66 (d, *J* = 8.9 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 7.00 (d, *J* = 7.5 Hz, 1H), 7.07–7.11 (m, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.29–7.34 (m, 3H), 7.38 (d, *J* = 7.5 Hz, 1H), 7.43–7.48 (m, 2H), 7.85–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 55.0, 55.2, 74.8, 113.6, 114.1, 121.1, 121.6, 122.4, 124.2, 125.8, 126.4, 126.8, 128.6, 128.8, 130.0, 130.6, 131.9, 132.7, 141.3, 142.0, 143.8, 144.7, 147.2, 158.8, 159.2, 171.1. HRMS (ESI) calcd for $C_{30}H_{23}NNaO_3$ $(M+Na)^+$ 468.1570, found 468.1567.



3ap

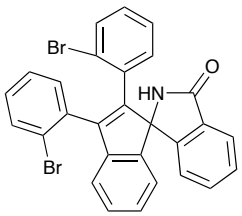
Compound 3ap. A solution of EtOAc/hexane (2:3) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 6.50 (br s, 1H), 6.82 (d, *J* = 8.1 Hz, 2H), 7.04 (d, *J* = 7.5 Hz, 1H), 7.09–

7.13 (m, 1H), 7.22 (d, J = 7.5 Hz, 1H), 7.26 (d, J = 8.1 Hz, 2H), 7.33–7.37 (m, 2H), 7.47–7.52 (m, 4H), 7.65 (d, J = 8.1 Hz, 2H), 7.85–7.89 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 74.9, 121.4, 121.5, 122.9, 123.7 (q, $J_{F\text{-}C}$ = 272 Hz), 123.9 (q, $J_{F\text{-}C}$ = 272 Hz), 124.5, 125.3 (q, $J_{F\text{-}C}$ = 4 Hz), 125.9 (q, $J_{F\text{-}C}$ = 4 Hz), 127.9, 129.0, 129.1, 129.3, 129.6, 129.9 (q, $J_{F\text{-}C}$ = 33 Hz), 130.4 (q, $J_{F\text{-}C}$ = 33 Hz), 131.8, 133.0, 136.6, 137.2, 142.4, 142.8, 143.3, 144.6, 146.0, 171.0. HRMS (ESI) calcd for $C_{30}H_{17}F_6NNaO$ $(M+Na)^+$ 544.1107, found 544.1093.



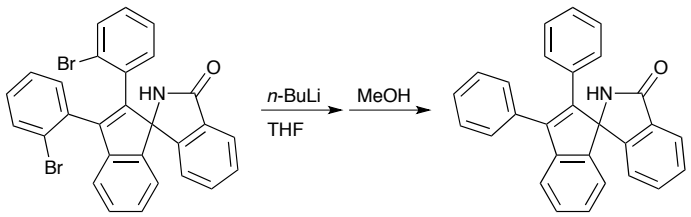
3aq

Compound 3aq. A solution of EtOAc/$CHCl_3$ (1:10) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 6.58–6.63 (m, 3H), 7.01 (d, J = 7.5 Hz, 1H), 7.05–7.09 (m, 1H), 7.13 (d, J = 8.8 Hz, 2H), 7.16–7.21 (m, 1H), 7.25 (d, J = 8.8 Hz, 2H), 7.31–7.35 (m, 2H), 7.45–7.48 (m, 2H), 7.52 (d, J = 8.8 Hz, 2H), 7.83–7.87 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 74.8, 121.2, 121.4, 122.1, 122.4, 122.7, 124.3, 127.4, 128.9, 129.1, 130.3, 130.8, 131.4, 131.8, 131.9, 132.0, 132.5, 132.8, 142.0, 142.7, 142.7, 144.5, 146.2, 171.1. HRMS (ESI) calcd for $C_{28}H_{17}Br_2NNaO$ $(M+Na)^+$ 563.9569, found 563.9559.



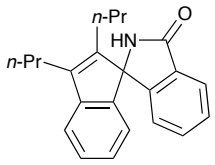
3ar

Compound 3ar. A solution of EtOAc/$CHCl_3$ (1:10) was used as an eluent for preparative TLC to give **3ar** as a mixture of four atropisomers. ^{1}H NMR (DMSO-d_6) δ 6.47 (dd, J = 7.5, 1.4 Hz), 6.54 (dd, J = 7.5, 1.4 Hz), 6.84 (dd, J = 7.5, 1.4 Hz), 6.87–6.93 (m), 6.95–7.05 (m), 7.08 (d, J = 7.5 Hz), 7.12 (d, J = 7.5 Hz), 7.17–7.34 (m), 7.36–7.41 (m), 7.41–7.60 (m), 7.63–7.73 (m), 7.76 (d, J = 7.5 Hz), 7.80 (d, J = 7.5 Hz), 8.77 (s), 8.78 (br s), 8.78 (br s), 9.05 (br s), 9.21 (br s). ^{1}H NMR ($CDCl_3$) δ 6.36 (br s), 6.44 (br s), 6.49 (d, J = 7.5 Hz), 6.57 (dd, J = 7.5, 1.4 Hz), 6.80–6.88 (m), 6.91–6.95 (m), 7.02 (d, J = 7.5 Hz), 7.06–7.24 (m), 7.27–7.47 (m), 7.48–7.60 (m), 7.63–7.66 (m), 7.69–7.72 (m), 7.75 (d, J = 7.5 Hz), 7.78 (d, J = 7.5 Hz). HRMS (ESI) calcd for $C_{28}H_{17}Br_2NNaO$ $(M+Na)^+$ 563.9569, found 563.9569.



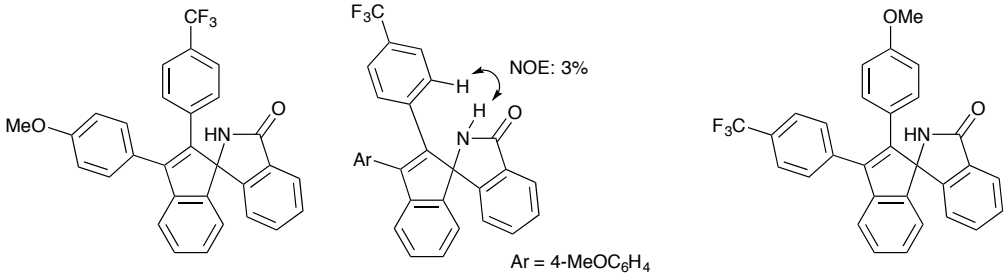
3ar **3am**

Transformation of 3ar into 3am. To a solution of **3ar** (54.3 mg, 0.10 mmol) in THF (1.0 mL) was added *n*-BuLi (1.60 M in *n*-hexane, 500 μL, 0.80 mmol) at –78 °C, and the mixture was stirred for 1 h. MeOH (160 μL, 4.0 mmol) was added, then the mixture was stirred at –78 °C for 20 min. Saturated NH_4Cl solution was added to the mixture and it was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/EtOAc (1:1) to give **3am** (32.5 mg, 0.084 mmol, 84% yield).



3as

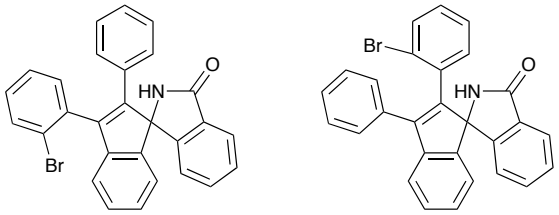
Compound 3as. A solution of EtOAc/hexane (1:1) was used as an eluent for preparative TLC. ^{1}H NMR ($CDCl_3$) δ 0.76 (t, *J* = 7.1 Hz, 3H), 1.03–1.11 (m, 1H), 1.04 (t, *J* = 7.1 Hz, 3H), 1.14–1.25 (m, 1H), 1.65–1.74 (m, 2H), 1.81 (ddd, *J* = 14.4, 10.3, 5.7 Hz, 1H), 2.07 (ddd, *J* = 14.4, 10.3, 5.7 Hz, 1H), 2.54 (t, *J* = 7.5 Hz, 2H), 5.83 (br s, 1H), 6.82 (d, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 7.5 Hz, 1H), 7.02–7.07 (m, 1H), 7.25–7.29 (m, 2H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.89 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.5, 14.6, 22.0, 23.1, 27.3, 28.0, 74.6, 119.4, 121.8, 122.2, 124.1, 125.9, 128.5, 128.8, 131.8, 132.4, 141.1, 143.2, 144.3, 144.7, 147.4, 171.2. HRMS (ESI) calcd for $C_{22}H_{23}NNaO$ $(M+Na)^+$ 340.1672, found 340.1668.



3at **3at'**

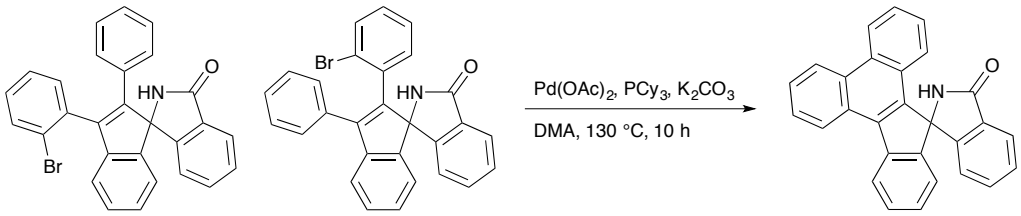
Compound 3at. A solution of EtOAc/hexane (1:1) was used as an eluent for preparative TLC. The stereostructure of **3at** was assigned by an NOE experiment. ^{1}H NMR ($CDCl_3$) δ 3.83 (s, 3H), 6.19 (br s, 1H), 6.83 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 7.02 (d, *J* = 7.5 Hz, 1H), 7.07–7.11 (m, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.9 Hz, 2H), 7.34

(t, J = 7.5 Hz, 1H), 7.41 (d, J = 7.5 Hz, 1H), 7.45–7.50 (m, 2H), 7.86–7.90 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 55.2, 74.7, 114.3, 121.6, 121.7, 122.6, 123.9 (q, $J_{F\text{-}C}$ = 271 Hz), 124.4, 125.1 (q, $J_{F\text{-}C}$ = 3 Hz), 125.5, 127.5, 128.9, 129.0, 129.3 (q, $J_{F\text{-}C}$ = 33 Hz), 130.5, 131.8, 132.9, 137.5, 140.5, 143.2, 143.9, 144.8, 146.6, 159.6, 170.9. HRMS (ESI) calcd for $C_{30}H_{20}F_3NNaO_2$ $(M+Na)^+$ 506.1338, found 506.1339. **Compound 3at'**. ^{1}H NMR ($CDCl_3$) δ 3.66 (s, 3H), 6.11 (br s, 1H), 6.53 (d, J = 8.9 Hz, 2H), 6.61 (d, J = 8.9 Hz, 2H), 7.04 (d, J = 7.0 Hz, 1H), 7.08–7.12 (m, 1H), 7.19 (t, J = 7.0 Hz, 1H), 7.30–7.35 (m, 2H), 7.46–7.50 (m, 2H), 7.51 (d, J = 7.8 Hz, 2H), 7.64 (d, J = 7.8 Hz, 2H), 7.87–7.91 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 55.0, 74.9, 113.8, 120.8, 121.6, 122.8, 124.0 (q, $J_{F\text{-}C}$ = 273 Hz), 124.3, 124.9, 125.7 (q, $J_{F\text{-}C}$ = 3 Hz), 127.2, 128.8, 129.0, 129.7, 129.9 (q, $J_{F\text{-}C}$ = 33 Hz), 129.9, 131.8, 132.8, 138.1, 140.3, 143.0, 144.6, 144.7, 146.6, 159.2, 171.0. HRMS (ESI) calcd for $C_{30}H_{20}F_3NNaO_2$ $(M+Na)^+$ 506.1338, found 506.1348.



3au

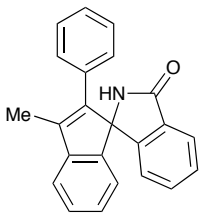
Compound 3au. A solution of EtOAc/$CHCl_3$ (1:10) was used as an eluent for preparative TLC. The title compound was obtained as a mixture of regio- and atropisomers. ^{1}H NMR ($CDCl_3$) δ 6.06 (br s), 6.15 (br s), 6.26 (br s), 6.42 (dd, J = 7.5, 1.4 Hz), 6.68 (d, J = 7.5 Hz), 6.85–6.89 (m), 6.95–7.06 (m), 7.09 (d, J = 7.5 Hz), 7.13–7.48 (m), 7.49–7.53 (m), 7.67 (d, J = 7.5 Hz), 7.69–7.72 (m), 7.78 (d, J = 7.5 Hz), 7.83–7.86 (m), 7.91–7.94 (m). HRMS (ESI) calcd for $C_{28}H_{18}BrNNaO$ $(M+Na)^+$ 486.0464, found 486.0450.



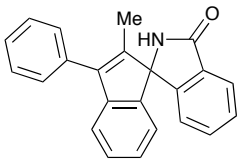
3au **5**

Transformation of 3au into 5. **3au** (46.4 mg, 0.10 mmol), K_2CO_3 (25.6 mg, 0.40 mmol), PCy_3 (2.9 mg, 0.010 mmol, 10 mol%), and $Pd(OAc)_2$ (0.0050 mmol, 5 mol%) were placed in a Schlenk tube under nitrogen. DMA (0.5 mL) was added and the Schlenk tube was capped with a glass stopper. The mixture was heated at 130 °C for 10 h with stirring. H_2O was added to the mixture and it was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/$CHCl_3$ (1:10) and GPC with $CHCl_3$ to give **5** (27.6 mg,

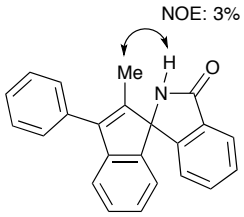
0.072 mmol, 72% yield). ^{1}H NMR ($CDCl_3$) δ 6.27 (br s, 1H), 6.74 (d, *J* = 8.1 Hz, 1H), 7.11 (d, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.31–7.37 (m, 3H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.55–7.60 (m, 1H), 7.78–7.84 (m, 2H), 8.11 (d, *J* = 7.5 Hz, 1H), 8.43 (d, *J* = 8.1 Hz, 1H), 8.72 (d, *J* = 8.1 Hz, 1H), 8.83 (d, *J* = 8.1 Hz, 1H), 8.94 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 72.0, 122.0, 123.1, 123.3, 123.5, 123.7, 123.8, 124.5, 124.8, 126.8, 127.28, 127.33, 127.4, 127.6, 127.7, 128.3, 128.8, 129.4, 130.9, 131.3, 132.2, 133.1, 136.0, 137.1, 140.8, 147.1, 148.8, 171.1. HRMS (ESI) calcd for $C_{28}H_{17}NNaO_3$ $(M+Na)^+$ 406.1202, found 406.1197.



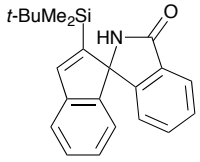
3av



3av'



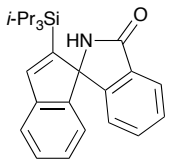
Compound 3av. A solution of EtOAc/$CHCl_3$ (1:10) was used as an eluent for preparative TLC to give a mixture of **3av** and **3av'**. The mixture was subjected to preparative HPLC (Inertsil SIL 100A 5um) with a solution of EtOAc/hexane (2/1) as an eluent to give each regioisomer. The stereostructure of **3av'** was assigned by an NOE experiment. ^{1}H NMR ($CDCl_3$) δ 2.23 (s, 3H), 5.92 (br s, 1H), 6.85–6.89 (m, 2H), 6.97 (d, *J* = 7.5 Hz, 1H), 7.00–7.03 (m, 1H), 7.15–7.19 (m, 4H), 7.36–7.40 (m, 2H), 7.42–7.45 (m, 2H), 7.81–7.85 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 12.1, 74.9, 119.9, 121.8, 122.2, 124.1, 127.0, 127.8, 128.3, 128.6, 129.1, 131.9, 132.6, 133.7, 138.6, 142.3, 144.5, 144.8, 147.0, 180.0. HRMS (ESI) calcd for $C_{23}H_{17}NNaO$ $(M+Na)^+$ 346.1202, found 346.1193. **Compound 3av'**. ^{1}H NMR ($CDCl_3$) δ 1.62 (s, 3H), 5.94 (br s, 1H), 6.93 (d, *J* = 6.8 Hz, 1H), 7.02 (d, *J* = 6.8 Hz, 1H), 7.11 (t, *J* = 6.8 Hz, 1H), 7.24–7.29 (m, 2H), 7.39–7.52 (m, 7H), 7.93 (d, *J* = 6.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 9.8, 74.5, 120.3, 121.4, 122.7, 124.1, 126.1, 128.0, 128.7, 128.8, 128.9, 131.8, 132.7, 133.9, 140.5, 141.4, 143.9, 144.3, 146.9, 171.2. HRMS (ESI) calcd for $C_{23}H_{17}NNaO$ $(M+Na)^+$ 346.1202, found 346.1197.



3aw

Compound 3aw. The crude product was subjected to preparative TLC with EtOAc/hexane (1:2) and GPC with $CHCl_3$. ^{1}H NMR ($CDCl_3$) δ –0.39 (s, 3H), –0.08 (s, 3H), 0.84 (s, 9H), 5.92 (br s, 1H), 6.85 (d, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 7.5 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 7.23 (s, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 7.5 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.90 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ –6.1, –4.7, 17.0, 26.8, 77.6, 121.5, 121.7,

122.1, 124.1, 126.9, 128.4, 128.8, 131.8, 132.4, 143.0, 145.0, 146.9, 147.6, 149.9, 170.9. HRMS (ESI) calcd for $C_{22}H_{25}NNaOSi$ $(M+Na)^+$ 370.1598, found 370.1591.

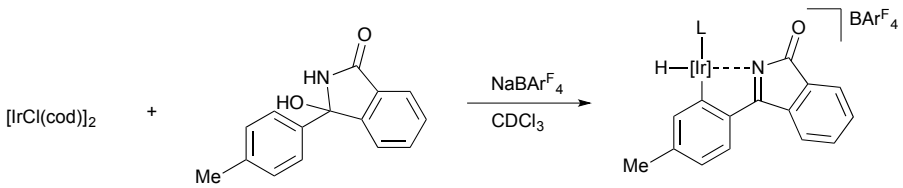


3ax

Compound 3ax. The crude product was subjected to preparative TLC with EtOAc/hexane (1:2) and GPC with $CHCl_3$. ^{1}H NMR ($CDCl_3$) δ 0.75 (d, *J* = 7.5 Hz, 9H), 0.98 (d, *J* = 7.5 Hz, 9H), 1.01–1.09 (m, 3H), 5.90 (br s, 1H), 6.88 (d, *J* = 7.5 Hz, 2H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.22 (s, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.89 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 11.4, 18.1, 18.9, 77.7, 121.4, 121.9, 122.1, 124.3, 126.9, 128.4, 128.8, 131.5, 132.4, 142.7, 145.9, 147.1, 148.1, 148.4, 170.9. HRMS (ESI) calcd for $C_{25}H_{31}NNaOSi$ $(M+Na)^+$ 412.2067, found 412.2066.

8. Reaction between $[IrCl(cod)]_2$ and 1b

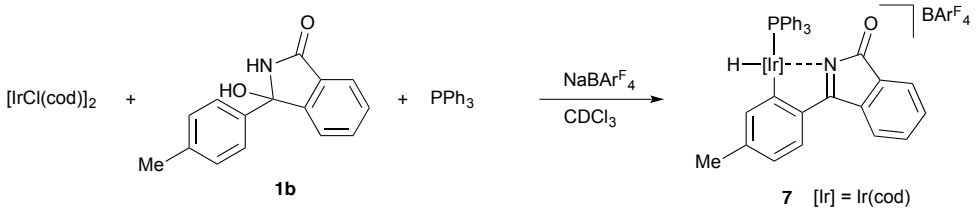
8-1. The reaction of 1b with $[IrCl(cod)]_2$



1b **6** [Ir] = Ir(cod)

Compound **1b** (4.8 mg, 0.020 mmol), $NaBAr^F_4$ (9.2 mg calculated as the dihydrate, 0.010 mmol), 1,4-dimethoxybenzene (1.4 mg, 0.010 mmol) as an internal standard, and $[IrCl(cod)]_2$ (3.4 mg, 0.010 mmol of Ir) were placed in an NMR tube under nitrogen. $CDCl_3$ (0.7 mL) was added, and the tube was capped with a rubber septum. ^{1}H NMR of the mixture showed a singlet peak of the hydride at –12.2 ppm, and the yield of the iridium-hydride complex was estimated to be 1.4 % after 3 h.

8-2. The reaction of 1b with $[IrCl(cod)]_2$ in the presence of PPh_3

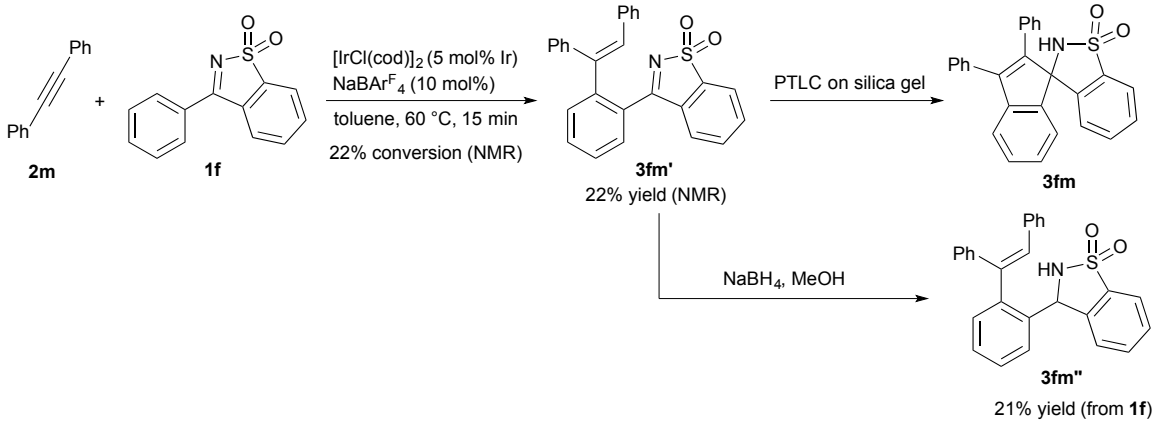


1b **7** [Ir] = Ir(cod)

Compound **1b** (4.8 mg, 0.020 mmol), $NaBAr^F_4$ (9.2 mg calculated as the dihydrate, 0.010

mmol), 1,4-dimethoxybenzene (1.4 mg, 0.010 mmol), and $[IrCl(cod)]_2$ (3.4 mg, 0.010 mmol of Ir) were placed in an NMR tube under nitrogen. $CDCl_3$ (0.7 mL) was added, and the tube was capped with a rubber septum. A singlet peak of the hydride was observed at –13.5 ppm by ^1H NMR. The yield of the iridium-hydride complex **7** was 57% after 20 h. The mixture was passed through a short column of alumina under N_2 with $CHCl_3$ as an eluent, and concentrated in vacuo to give **7**. ^1H NMR ($CDCl_3$) δ –13.47 (d, $J_{P–H}$ = 11.3 Hz, 1H), 1.71–1.81 (m, 1H), 1.81–1.91 (m, 1H), 2.26 (s, 3H), 2.27–2.34 (m, 1H), 2.50–2.57 (m, 1H), 2.59–2.65 (m, 1H), 2.75–2.83 (m, 1H), 2.93–3.00 (m, 1H), 3.28 (dd, *J* = 15.9, 9.1 Hz, 1H), 3.39–3.44 (m, 1H), 4.08 (s, 1H), 5.02 (d, *J* = 4.5 Hz, 1H), 6.08 (dd, *J* = 14.7, 8.0 Hz, 1H), 6.98 (d, *J* = 6.8 Hz, 1H), 7.09–7.13 (m, 6H), 7.21–7.26 (m, 7H), 7.30 (t, *J* = 6.8 Hz, 3H), 7.49–7.60 (m, 6H), 7.69–7.73 (m, 9H), 7.85 (d, *J* = 6.8 Hz, 1H), 7.99 (d, *J* = 6.8 Hz, 1H); ^{31}P NMR ($CDCl_3$) 11.8. HRMS (ESI) calcd for $C_{41}H_{38}IrNOP$ $(M–BAr^F_4)^+$ 784.2317, found 784.2301.

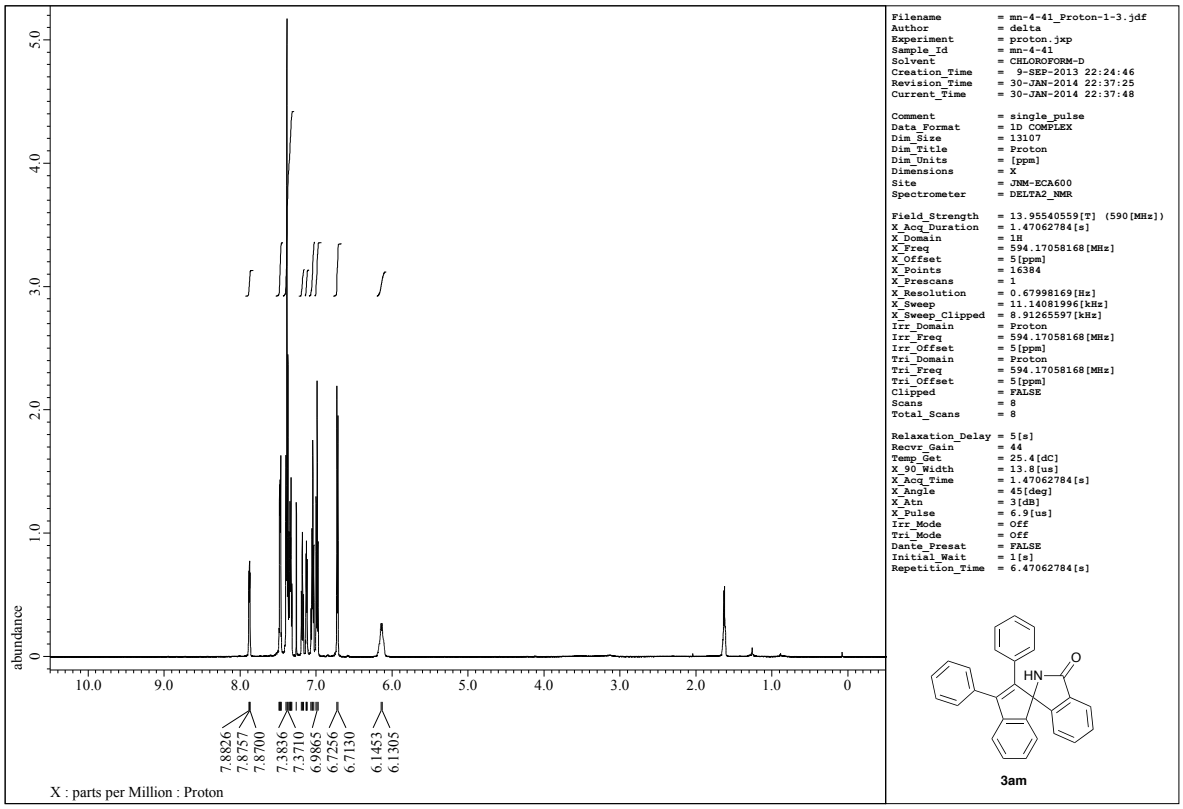
9. Observation of the intermediate

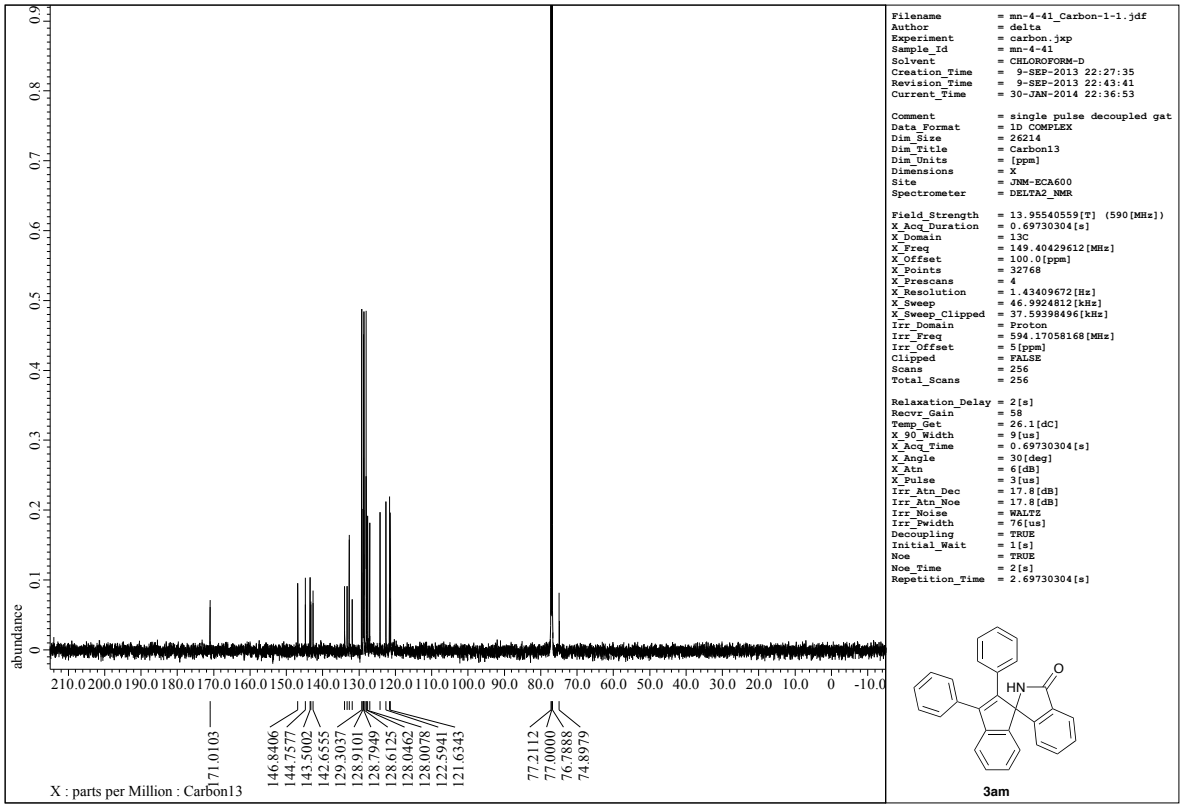


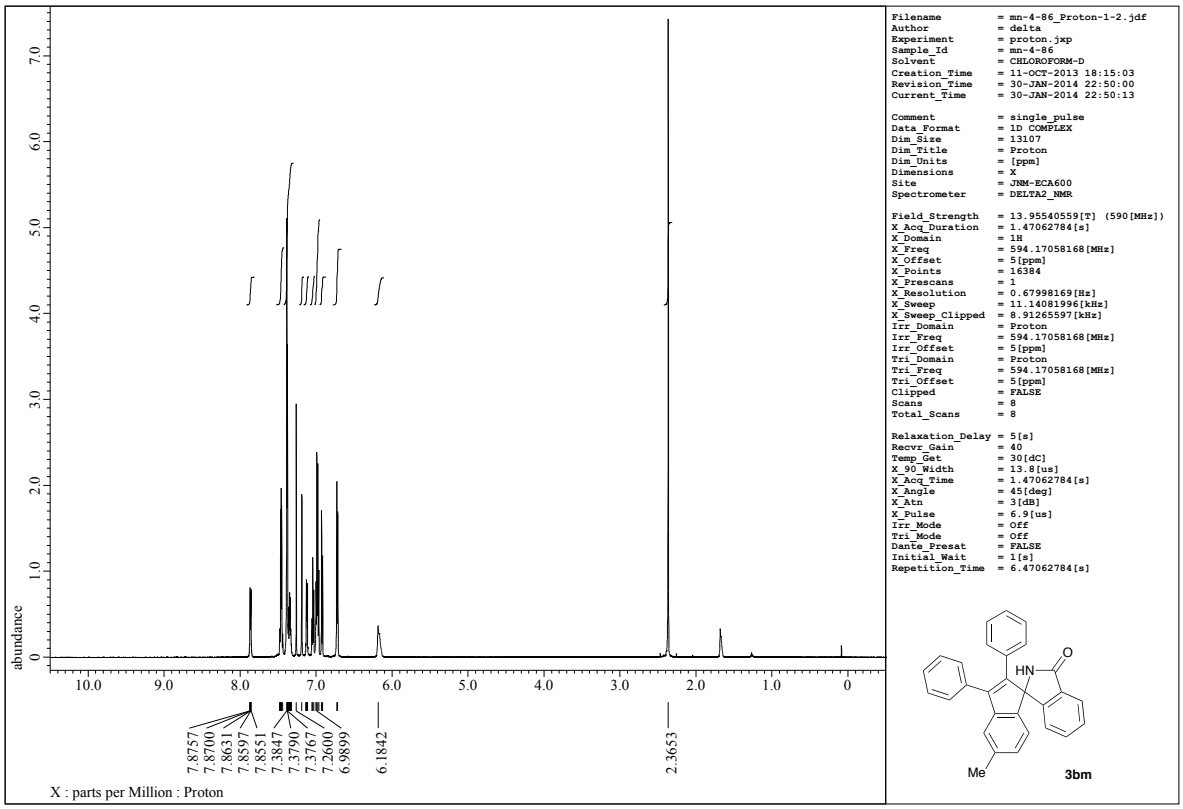
A mixture of ketimine **1f** (24.3 mg, 0.10 mmol), $NaBAr^F_4$ (9.2 mg calculated as the dihydrate, 0.010 mmol, 10 mol%), alkyne **2m** (26.7 mg, 0.15 mmol), and $[IrCl(cod)]_2$ (1.7 mg, 0.0050 mmol of Ir, 5 mol% of Ir) in toluene (0.4 mL) was heated at 60 °C for 15 min with stirring. The mixture was passed through a short column of alumina with EtOAc as an eluent, and the solvent was removed on a rotary evaporator. The ^1H NMR of the crude mixture showed the peaks of compound **3fm'** and unreacted **1f** and **2m**, where the formation of annulation product **3fm** was not observed. When the residue was subjected to preparative TLC on silica gel, **3fm'** was partially transformed into **3fm**. On the other hand, treatment of the crude mixture with $NaBH_4$ (5.7 mg, 0.15 mmol) in MeOH (0.4 mL) at room temperature overnight gave **3fm"**, which was isolated as a pure form (9.2 mg, 21% yield from **1f**; preparative TLC on silica gel with EtOAc/hexane (1:3)). **Compound 3fm"**. ^1H NMR ($CDCl_3$) δ 4.16 (br s, 1H), 5.96 (d, *J* = 4.1 Hz, 1H), 6.74 (br s, 1H), 6.81 (s, 1H), 7.04 (d, *J* = 7.5 Hz, 1H), 7.15–7.22 (m, 7H), 7.24–7.31 (m, 4H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.43–7.50 (m, 3H), 7.76 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 57.9, 121.1, 125.4, 127.5,

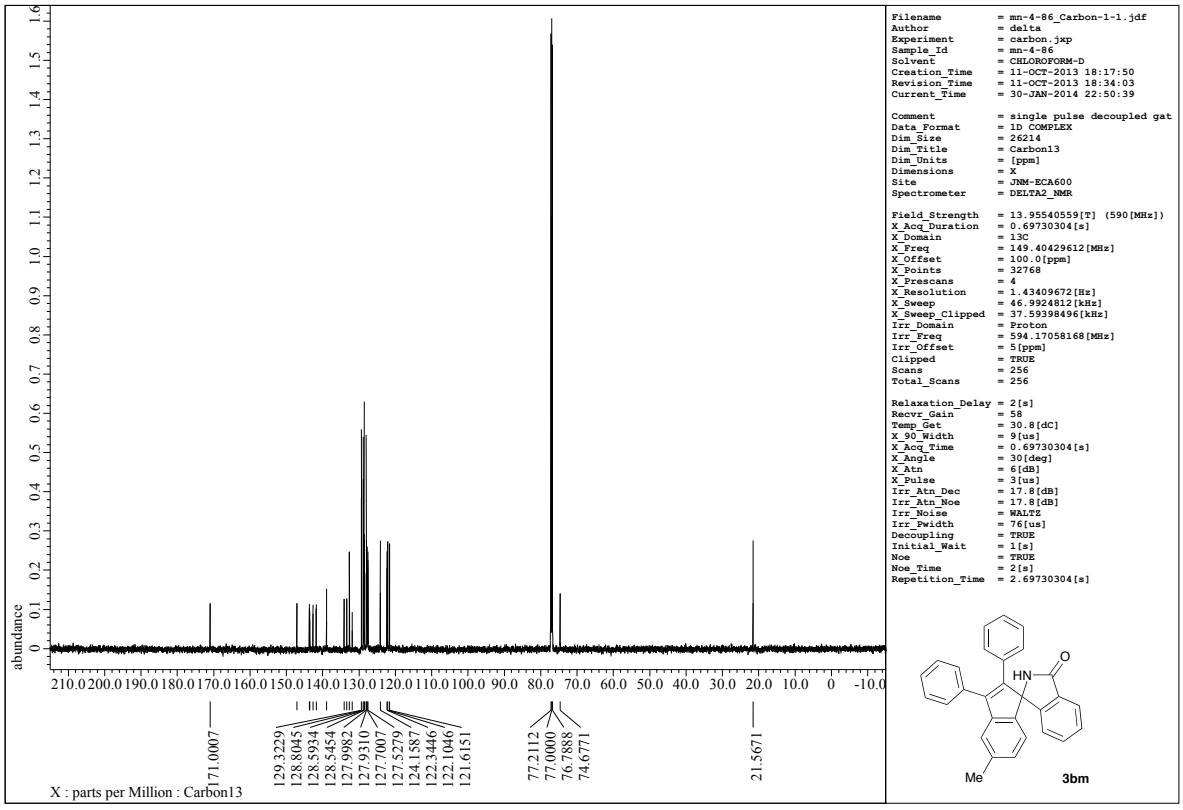
128.2, 128.3, 128.8, 128.9, 129.0, 129.1, 129.4, 129.5, 129.9, 130.9, 131.9, 133.3, 135.3, 136.5, 137.3, 140.2, 140.4, 141.1, 144.7. HRMS (ESI) calcd for $C_{27}H_{21}NNaO_2S$ $(M+Na)^+$ 446.1185, found 446.1173.

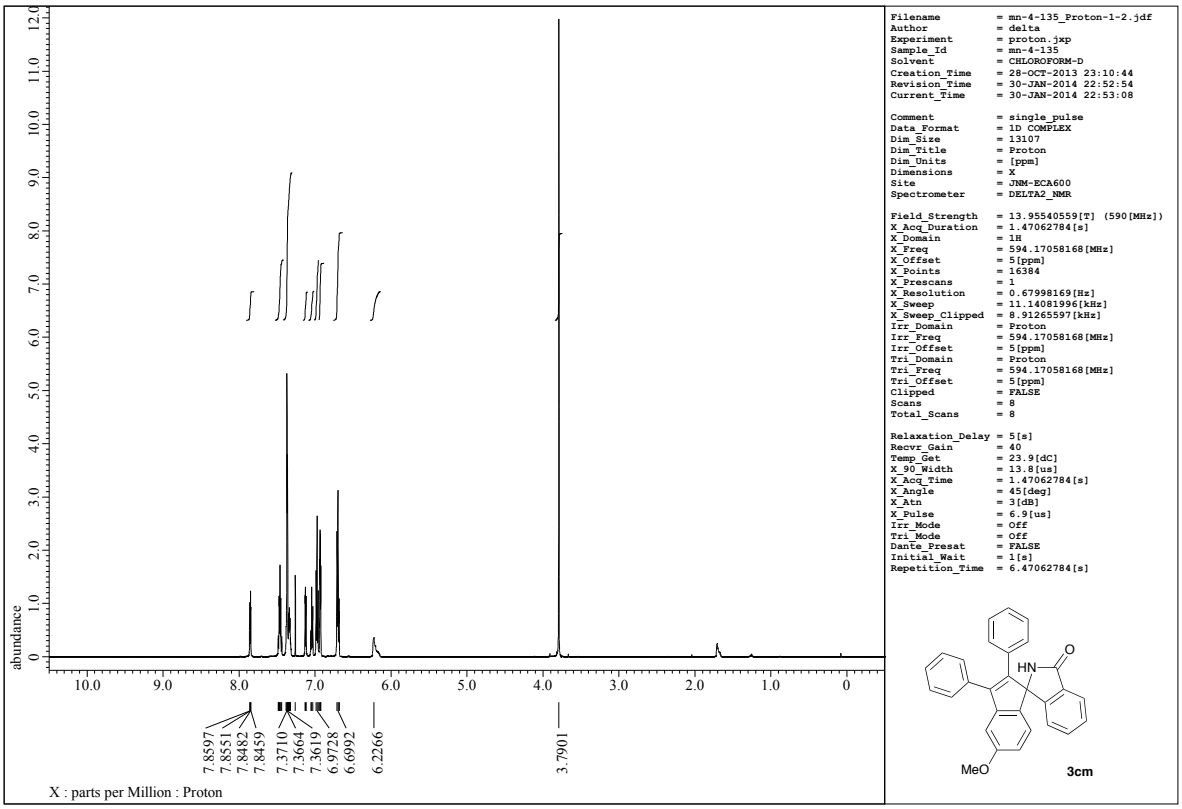
10. ^{1}H and ^{13}C NMR spectra

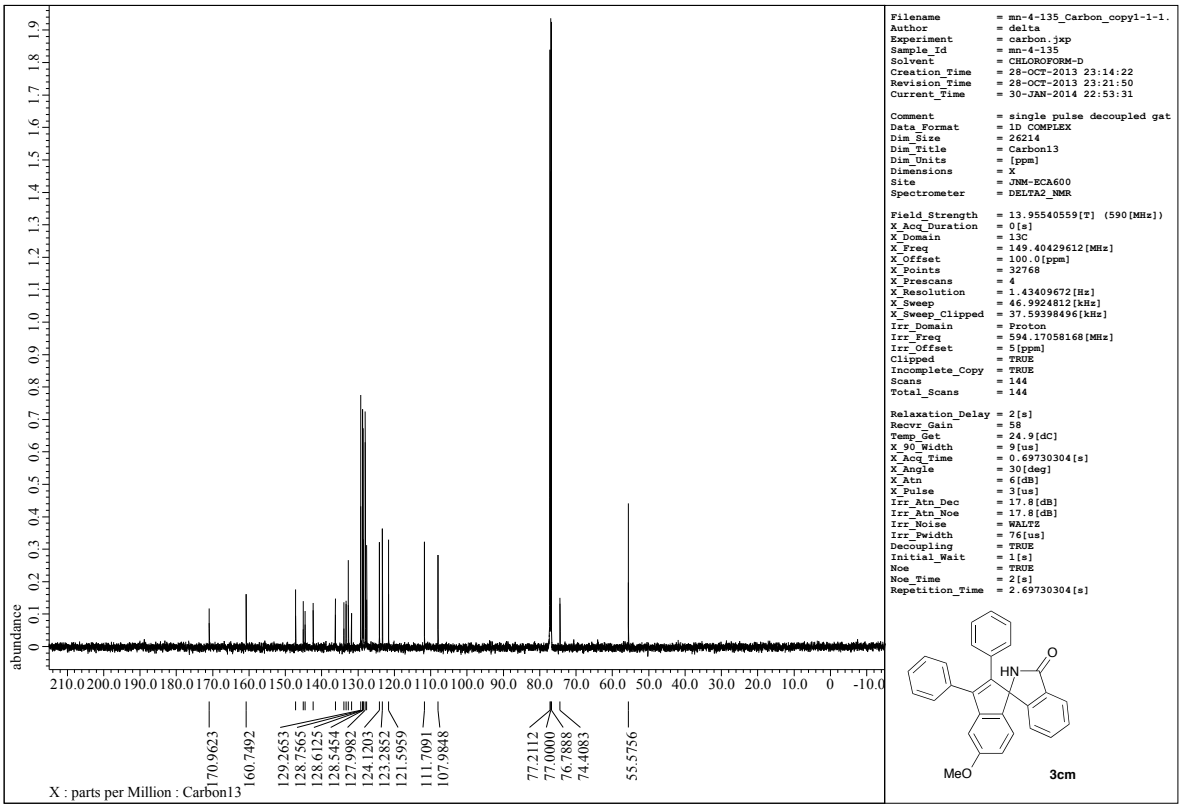


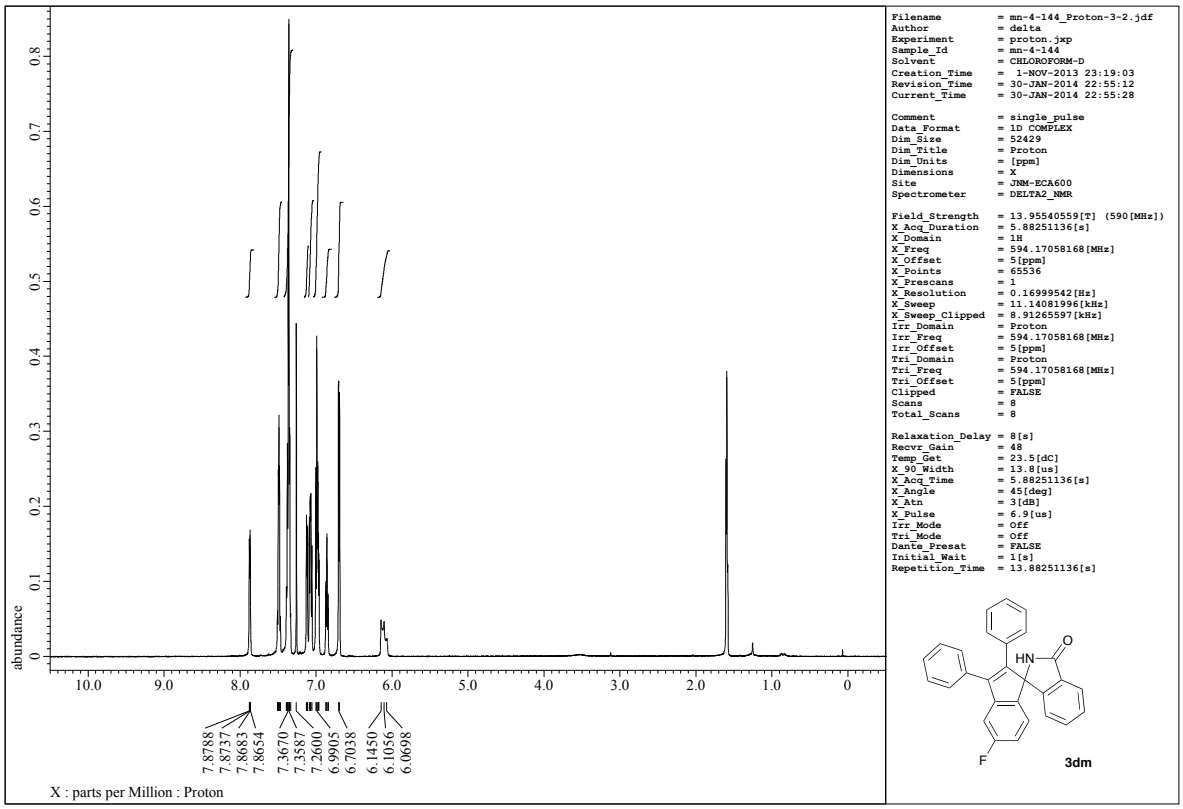


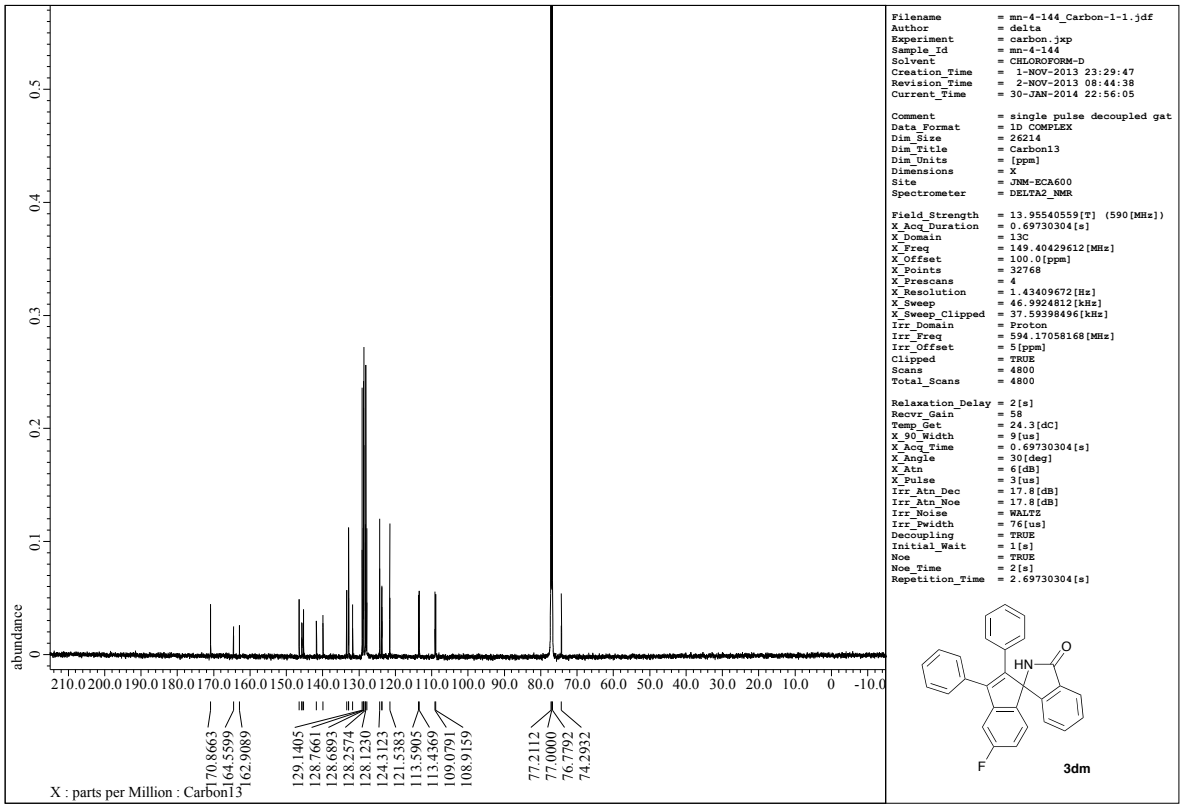

abundance
7.8757
7.8700
7.8631
7.8597
7.8551
7.3847
7.3790
7.3767
7.2600
6.9899
6.1842
2.3653
X : parts per Million : Proton
Filename = mn-4-86_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-86
Solvent = CHLOROFORM-D
Creation_Time = 11-OCT-2013 18:15:03
Revision_Time = 30-JAN-2014 22:50:00
Current_Time = 30-JAN-2014 22:50:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 30[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
HN
O
Me
3bm

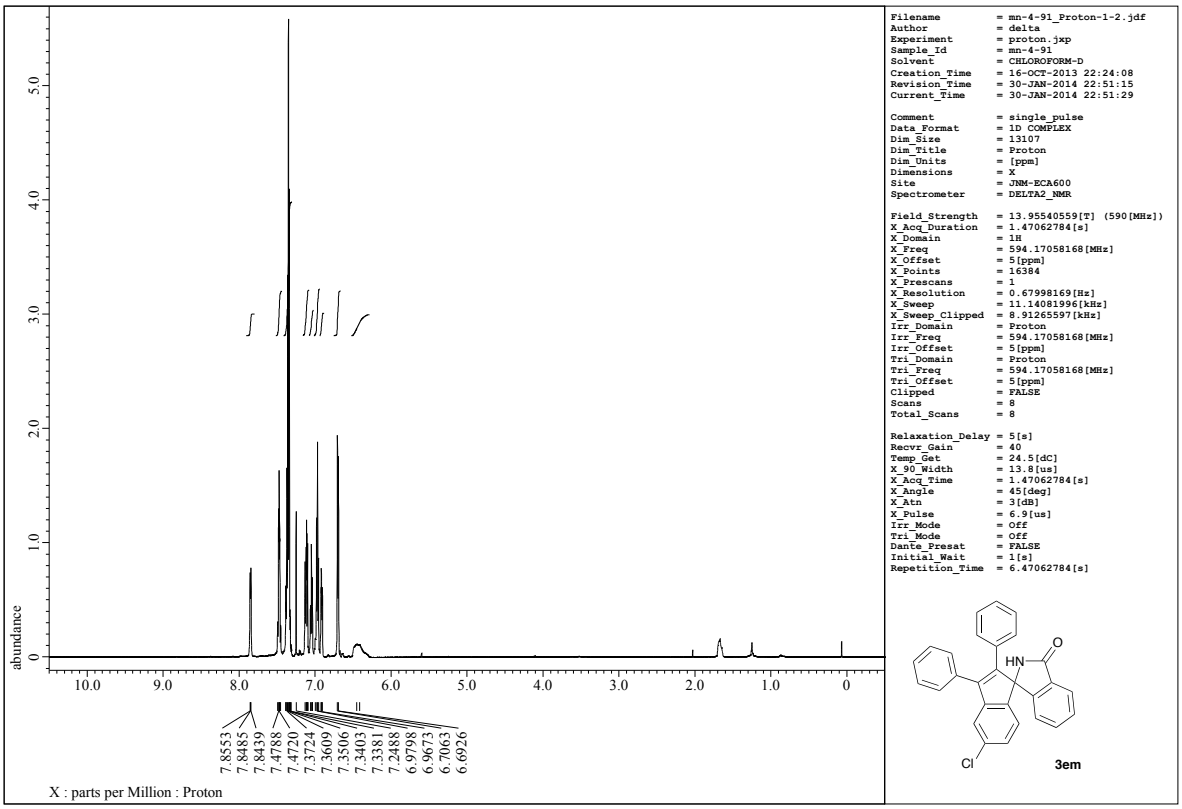

abundance
171.0007
129.3229
128.8045
128.5934
128.5454
127.9982
127.9310
127.7007
127.5279
124.1587
122.3446
122.1046
121.6151
77.2112
77.0000
76.7888
74.6771
21.5671
X : parts per Million : Carbon13
Filename = mn-4-86_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-86
Solvent = CHLOROFORM-D
Creation_Time = 11-OCT-2013 18:17:50
Revision_Time = 11-OCT-2013 18:34:03
Current_Time = 30-JAN-2014 22:50:39
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 30.8[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
HN
O
Me
3bm

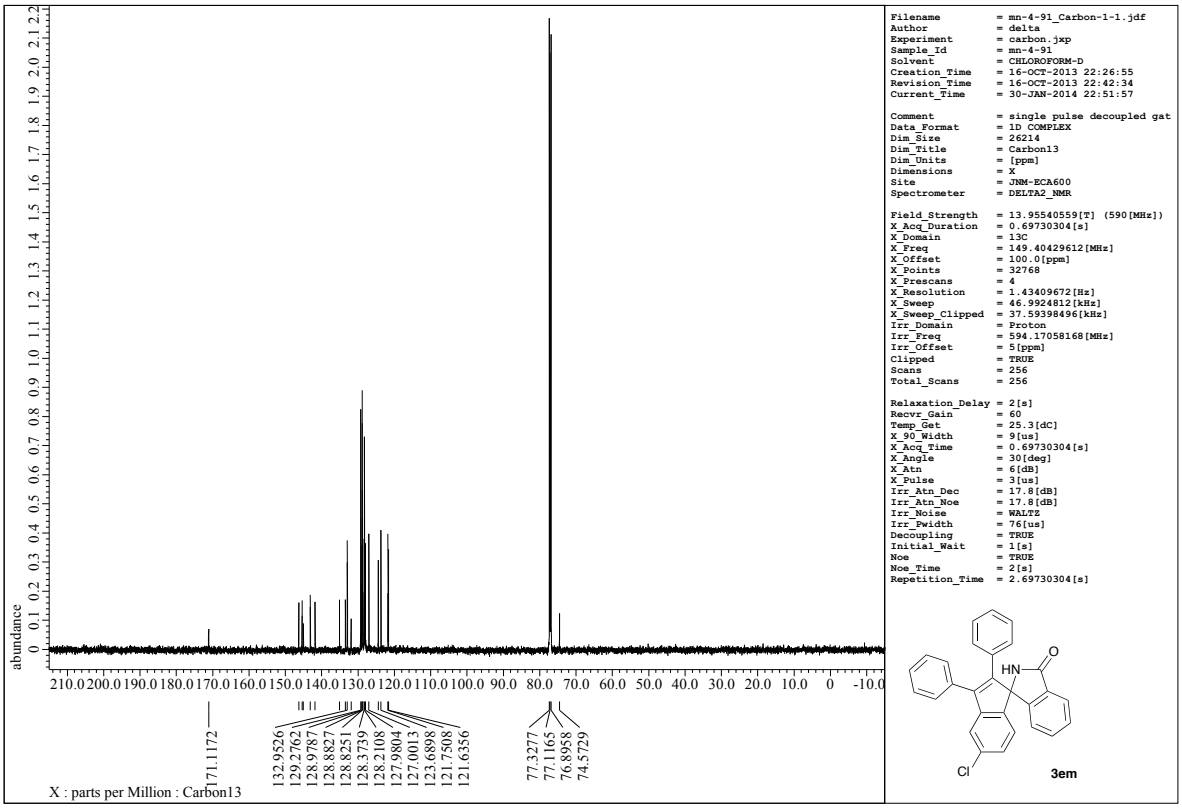

abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.8597
7.8551
7.8482
7.8459
7.3710
7.3664
7.3619
7.3728
6.6992
6.2266
3.7901
X : parts per Million : Proton
Filename = mn-4-135_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-135
Solvent = CHLOROFORM-D
Creation_Time = 28-OCT-2013 23:10:44
Revision_Time = 30-JAN-2014 22:52:54
Current_Time = 30-JAN-2014 22:53:08
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 23.9[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
HN
O
MeO
3cm

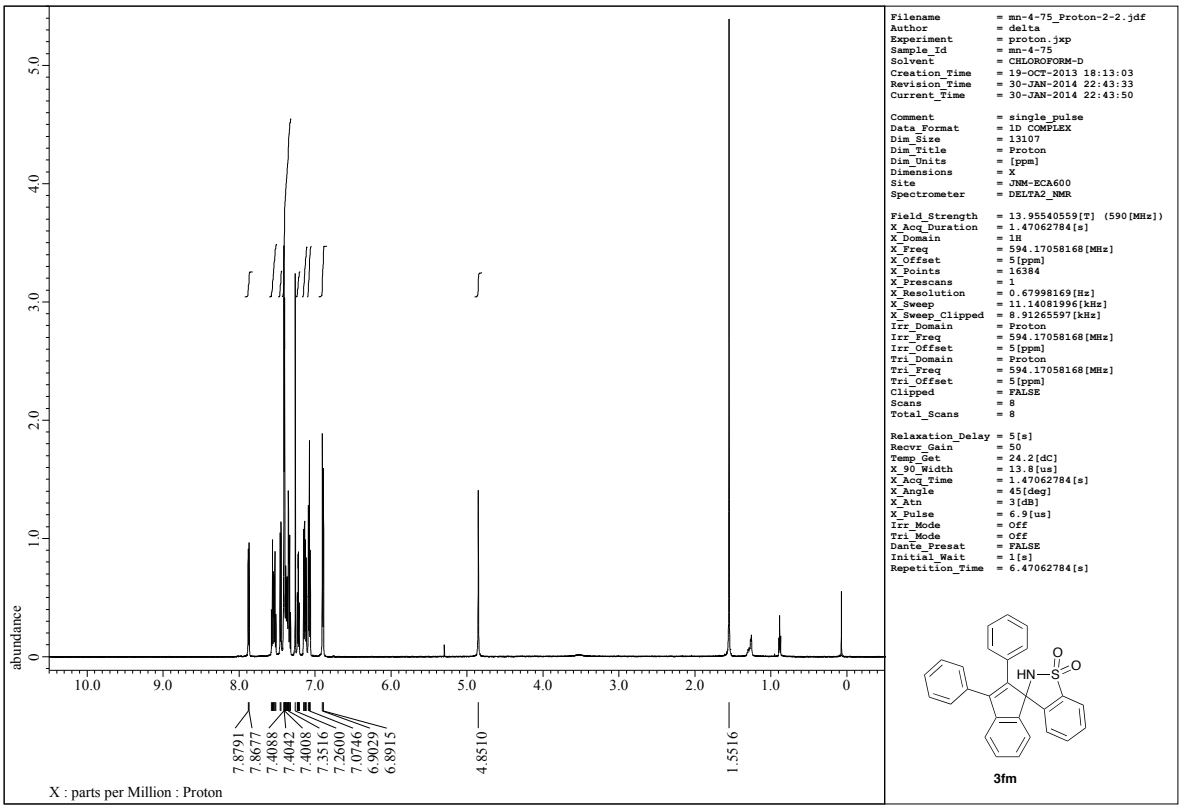

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
170.9623
160.7492
129.2653
128.7565
128.6125
128.5454
127.9982
124.1203
123.2852
121.5959
111.7091
107.9848
77.2112
77.0000
76.7888
74.4083
55.5756
X : parts per Million : Carbon13
Filename = mn-4-135_Carbon_copy1-1-1.
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-135
Solvent = CHLOROFORM-D
Creation_Time = 28-OCT-2013 23:14:22
Revision_Time = 28-OCT-2013 23:21:50
Current_Time = 30-JAN-2014 22:53:31
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Incomplete_Copy = TRUE
Scans = 144
Total_Scans = 144
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 24.9[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
HN
O
MeO
3cm

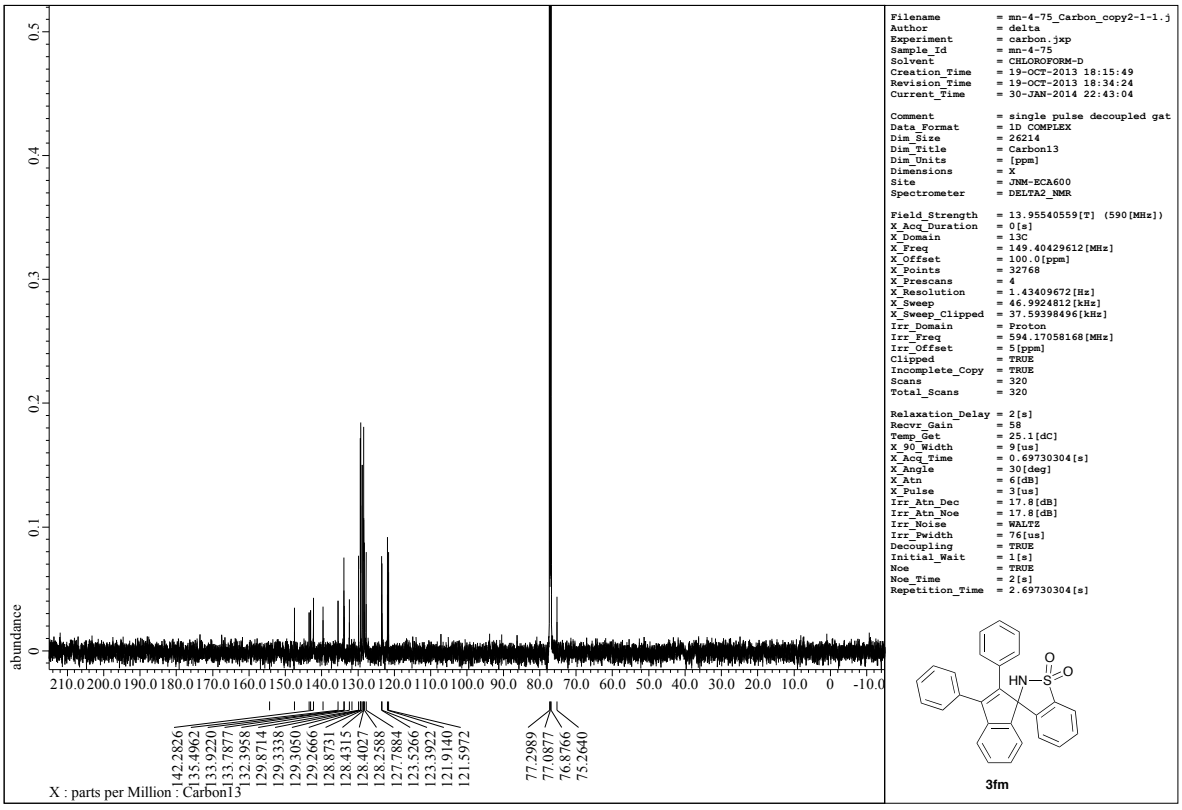

abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.8788
7.8737
7.8683
7.8654
7.3670
7.3587
7.2600
6.9905
6.7038
6.1450
6.1056
6.0698
X : parts per Million : Proton
Filename = mn-4-144_Proton-3-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-144
Solvent = CHLOROFORM-D
Creation_Time = 1-NOV-2013 23:19:03
Revision_Time = 30-JAN-2014 22:55:12
Current_Time = 30-JAN-2014 22:55:28
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 52429
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 5.88251136[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 65536
X_Prescans = 1
X_Resolution = 0.16999542[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 8[s]
Recvr_Gain = 48
Temp_Get = 23.5[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 5.88251136[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 13.88251136[s]
HN
O
F
3dm

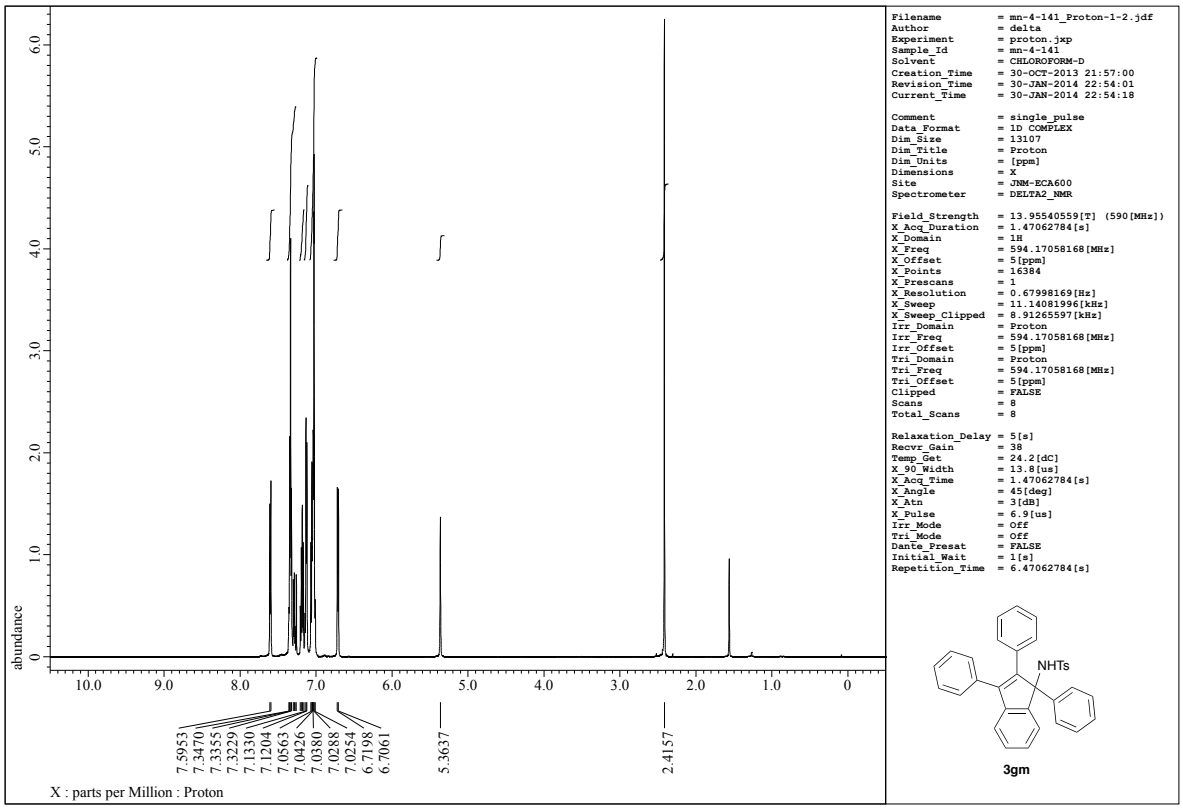

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
170.8663
164.5599
162.9089
129.1405
128.7661
128.6893
128.2574
128.1230
124.3123
121.5383
113.5905
113.4369
109.0791
108.9159
77.2112
77.0000
76.7792
74.2932
X : parts per Million : Carbon13
Filename = mn-4-144_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-144
Solvent = CHLOROFORM-D
Creation_Time = 1-NOV-2013 23:29:47
Revision_Time = 2-NOV-2013 08:44:38
Current_Time = 30-JAN-2014 22:56:05
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 4800
Total_Scans = 4800
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 24.3[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
HN
O
F
3dm

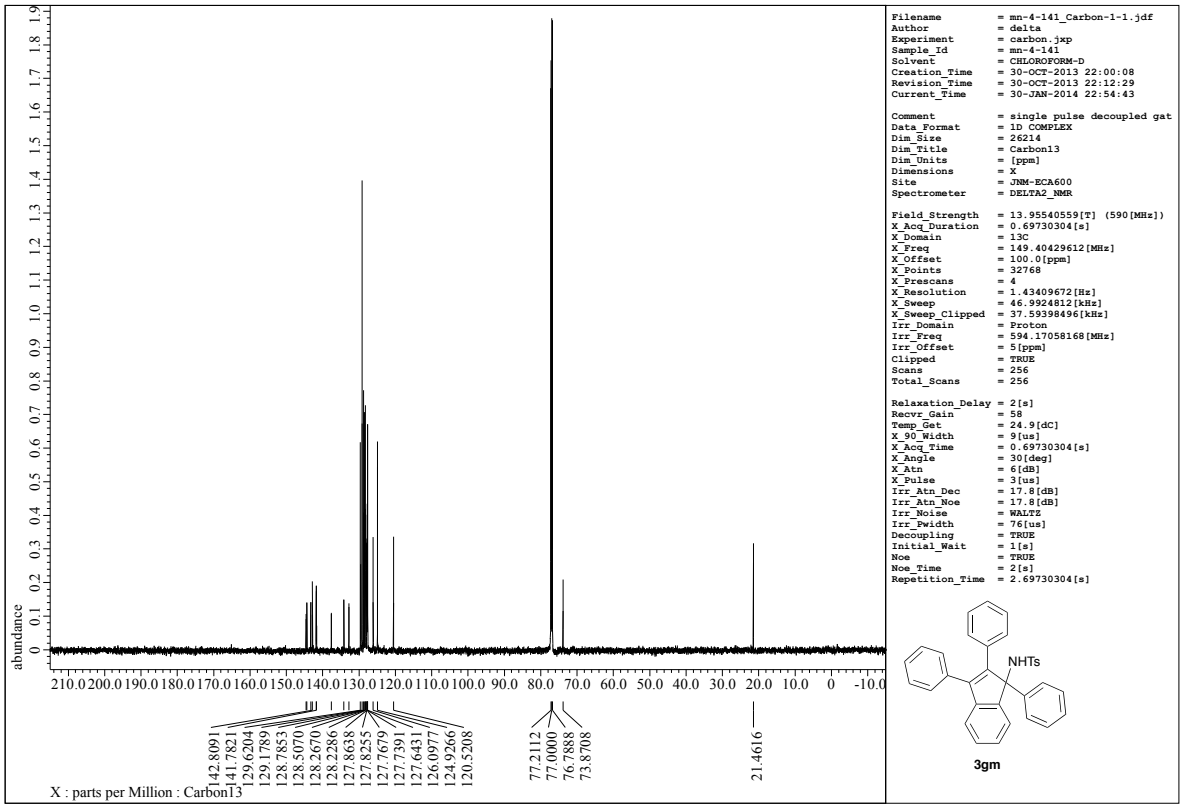

abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.8553
7.8485
7.8439
7.4788
7.4720
7.3724
7.3609
7.3506
7.3403
7.3381
7.2488
6.9798
6.9673
6.7063
6.6926
X : parts per Million : Proton
Filename = mn-4-91_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-91
Solvent = CHLOROFORM-D
Creation_Time = 16-OCT-2013 22:24:08
Revision_Time = 30-JAN-2014 22:51:15
Current_Time = 30-JAN-2014 22:51:29
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 24.5[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
HN
O
Cl
3em

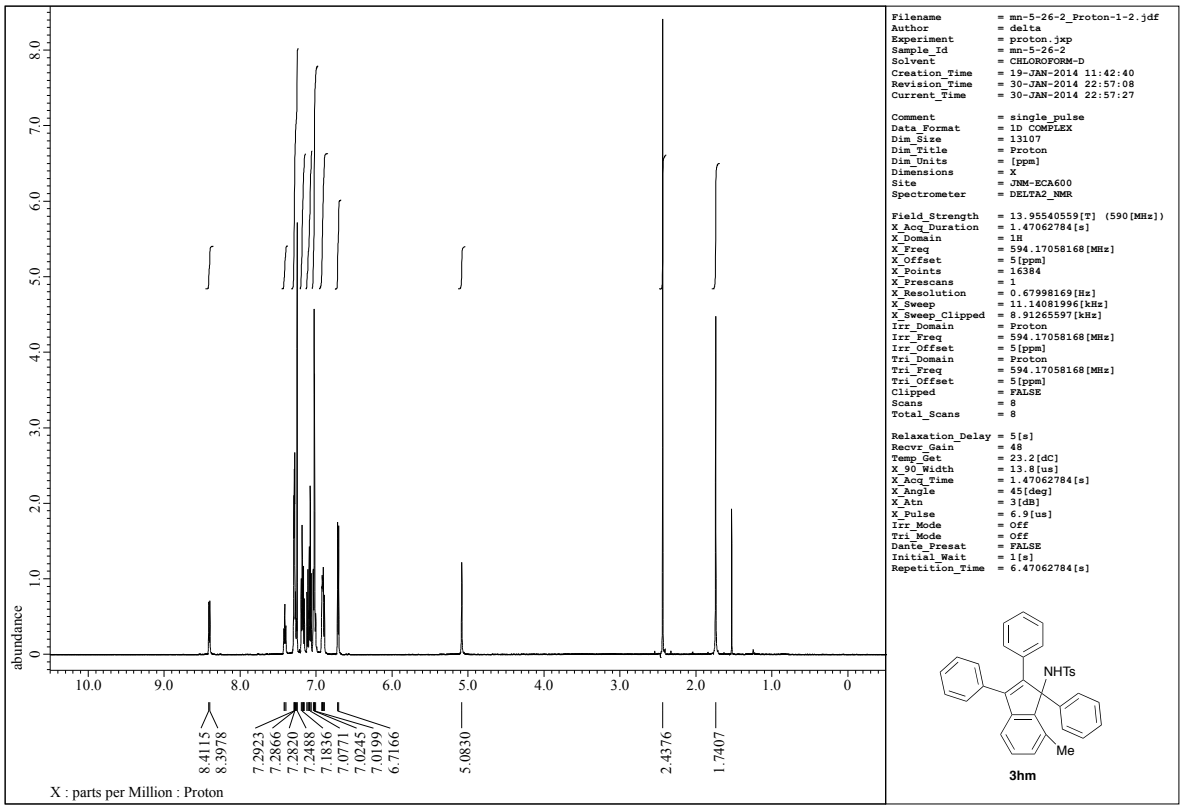

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
171.1172
132.9526
129.2762
128.9787
128.8827
128.8251
128.3739
128.2108
127.9804
127.0013
123.6898
121.7508
121.6356
77.3277
77.1165
76.8958
74.5729
X : parts per Million : Carbon13
Filename = mn-4-91_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-91
Solvent = CHLOROFORM-D
Creation_Time = 16-OCT-2013 22:26:55
Revision_Time = 16-OCT-2013 22:42:34
Current_Time = 30-JAN-2014 22:51:57
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 25.3[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
HN
O
Cl
3em

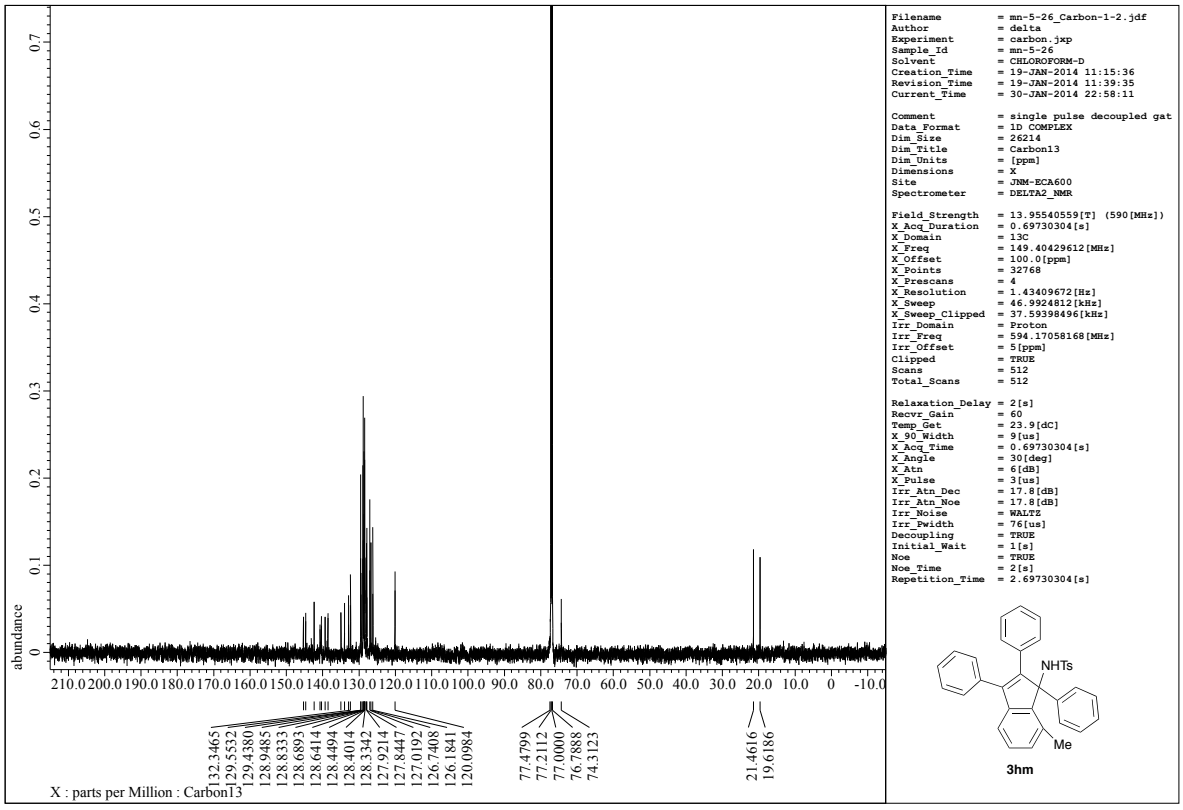

abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.8791
7.8677
7.4088
7.4042
7.4008
7.3516
7.2600
7.0746
6.9029
6.8915
4.8510
1.5516
X : parts per Million : Proton
Filename = mn-4-75_Proton-2-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-75
Solvent = CHLOROFORM-D
Creation_Time = 19-OCT-2013 18:13:03
Revision_Time = 30-JAN-2014 22:43:33
Current_Time = 30-JAN-2014 22:43:50
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 24.2[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
HN
S
O
O
3fm

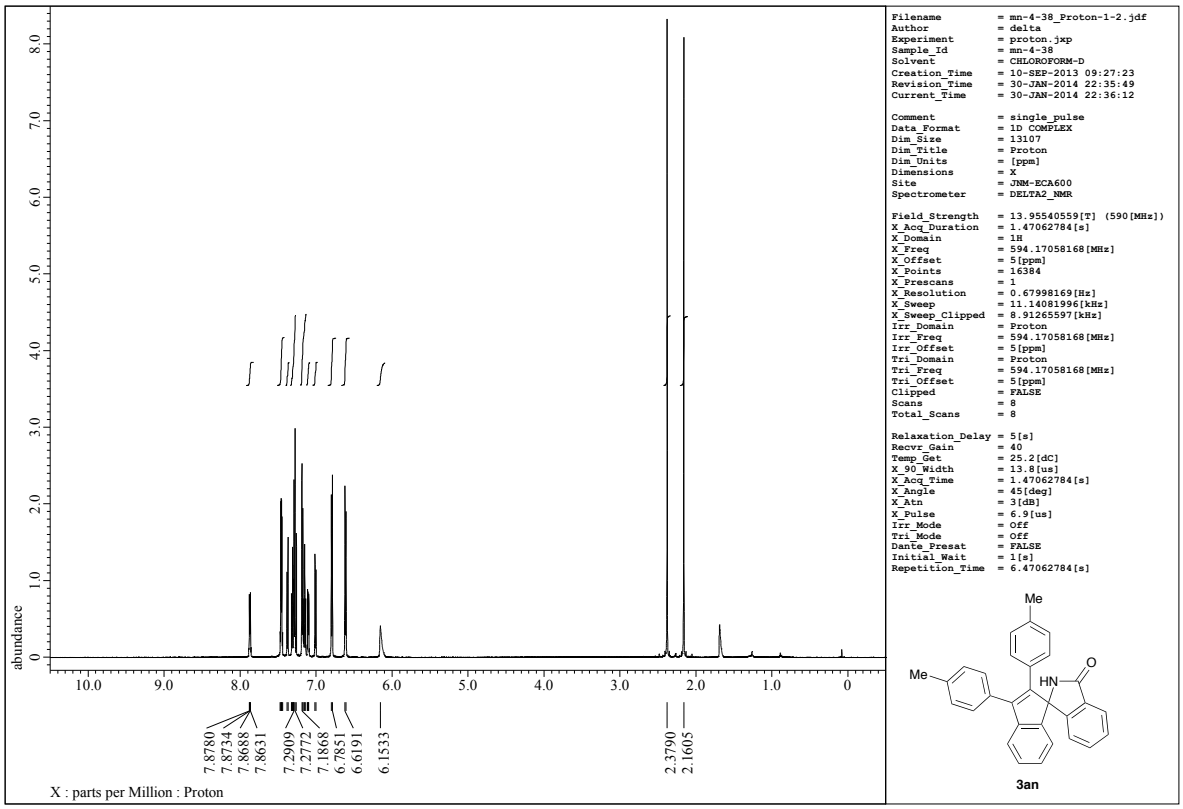

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
142.2826
135.4962
133.9220
133.7877
132.3958
129.8714
129.3338
129.3050
129.2666
128.8731
128.4315
128.4027
128.2588
127.7884
123.5266
123.3922
121.9140
121.5972
77.2989
77.0877
76.8766
75.2640
X : parts per Million : Carbon13
Filename = mn-4-75_Carbon_copy2-1-1.j
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-75
Solvent = CHLOROFORM-D
Creation_Time = 19-OCT-2013 18:15:49
Revision_Time = 19-OCT-2013 18:34:24
Current_Time = 30-JAN-2014 22:43:04
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Incomplete_Copy = TRUE
Scans = 320
Total_Scans = 320
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 25.1[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
HN
S
O
O
3fm

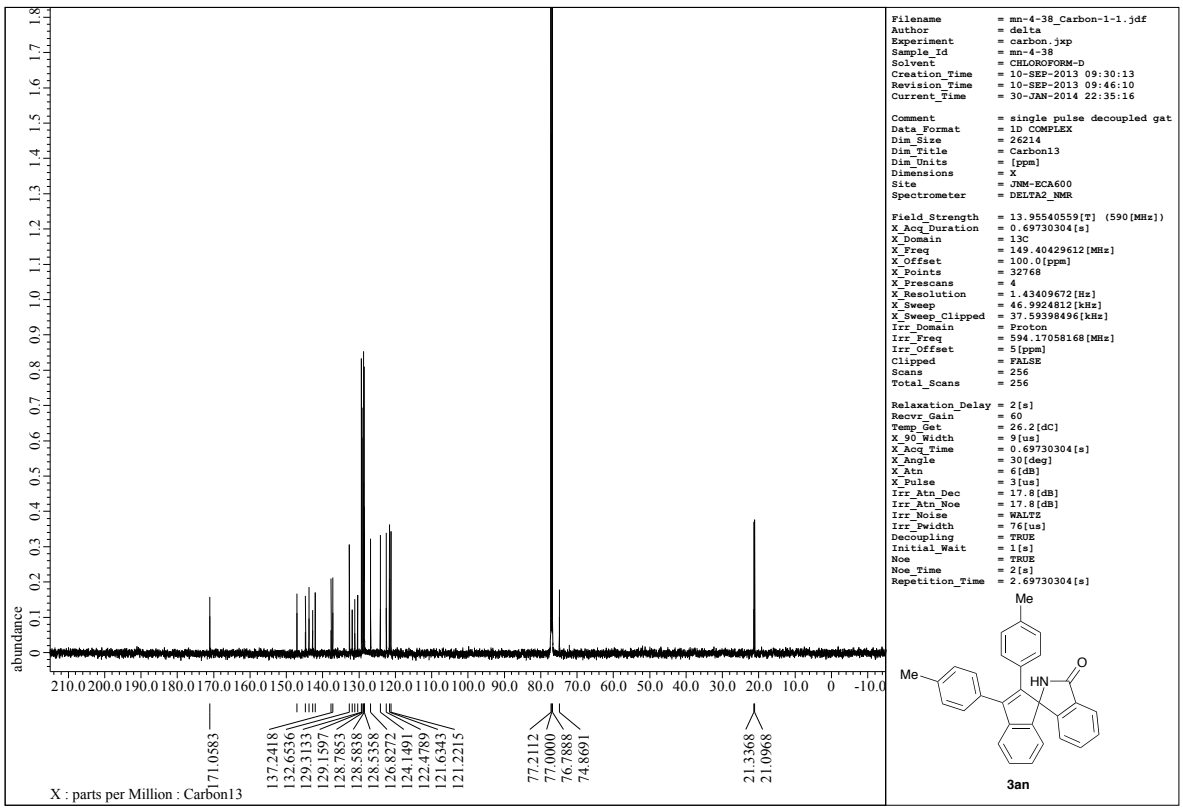

abundance
7.5953
7.3470
7.3355
7.3229
7.1330
7.1204
7.0563
7.0426
7.0380
7.0288
7.0254
6.7198
6.7061
5.3637
2.4157
X : parts per Million : Proton
Filename = mn-4-141_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-141
Solvent = CHLOROFORM-D
Creation_Time = 30-OCT-2013 21:57:00
Revision_Time = 30-JAN-2014 22:54:01
Current_Time = 30-JAN-2014 22:54:18
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 24.2[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
NHTs
3gm

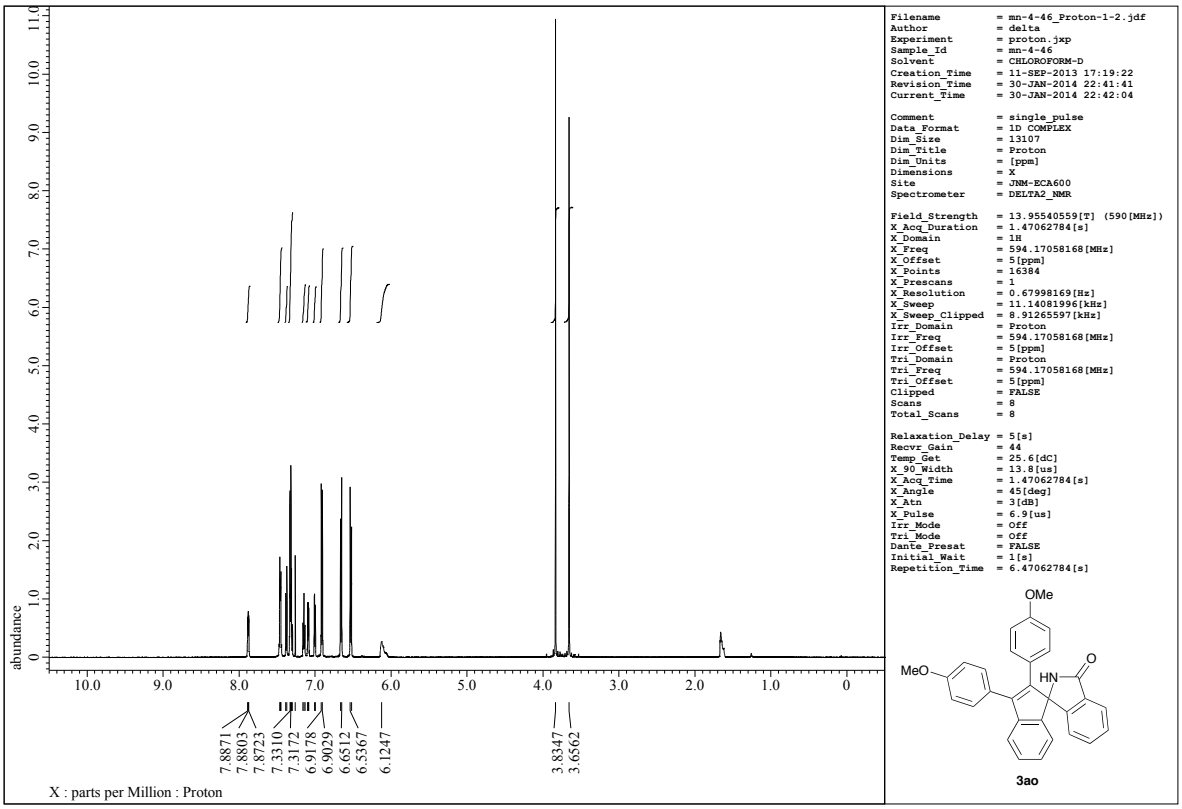

abundance
142.8091
141.7821
129.6204
129.1789
128.7853
128.5070
128.2670
128.2286
127.8638
127.8255
127.7679
127.7391
127.6431
126.0977
124.9266
120.5208
77.2112
77.0000
76.7888
73.8708
21.4616
X : parts per Million : Carbon13
Filename = mn-4-141_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-141
Solvent = CHLOROFORM-D
Creation_Time = 30-OCT-2013 22:00:08
Revision_Time = 30-OCT-2013 22:12:29
Current_Time = 30-JAN-2014 22:54:43
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 24.9[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
NHTs
3gm

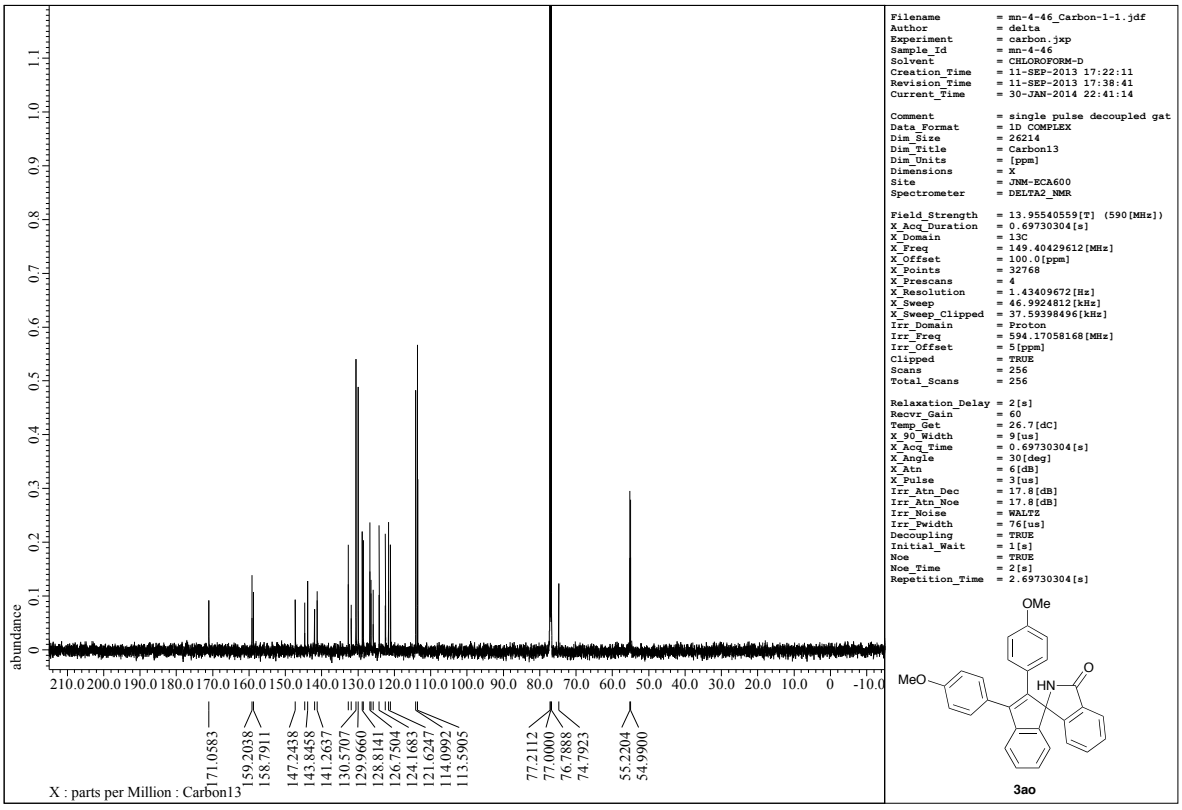

abundance
X : parts per Million : Proton
8.4115
8.3978
7.2923
7.2866
7.2820
7.2488
7.1836
7.0771
7.0245
7.0199
6.7166
5.0830
2.4376
1.7407
Filename = mn-5-26-2_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-5-26-2
Solvent = CHLOROFORM-D
Creation_Time = 19-JAN-2014 11:42:40
Revision_Time = 30-JAN-2014 22:57:08
Current_Time = 30-JAN-2014 22:57:27
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 23.2[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
NHTs
Me
3hm

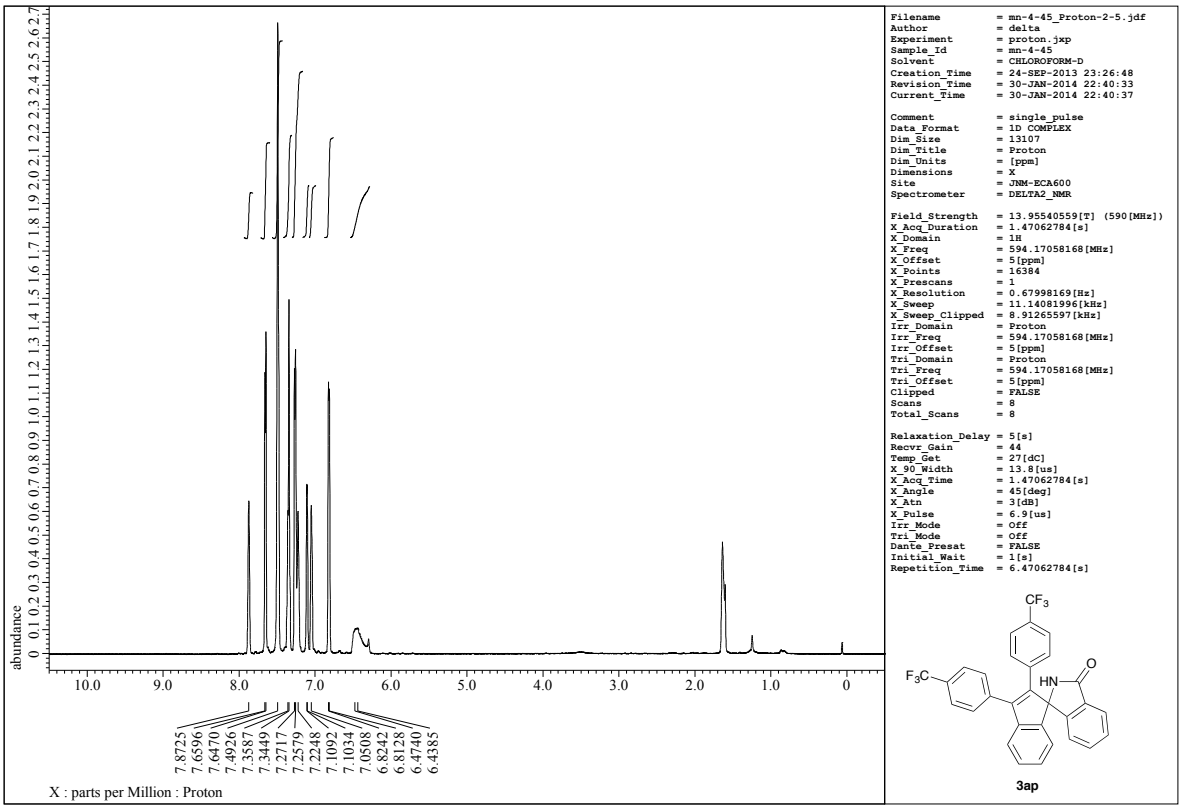

abundance
X : parts per Million : Carbon13
132.3465
129.5532
129.4380
128.9485
128.8333
128.6893
128.6414
128.4494
128.4014
128.3342
127.9214
127.8447
127.0192
126.7408
126.1841
120.0984
77.4799
77.2112
77.0000
76.7888
74.3123
21.4616
19.6186
Filename = mn-5-26_Carbon-1-2.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-5-26
Solvent = CHLOROFORM-D
Creation_Time = 19-JAN-2014 11:15:36
Revision_Time = 19-JAN-2014 11:39:35
Current_Time = 30-JAN-2014 22:58:11
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.9[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
NHTs
Me
3hm

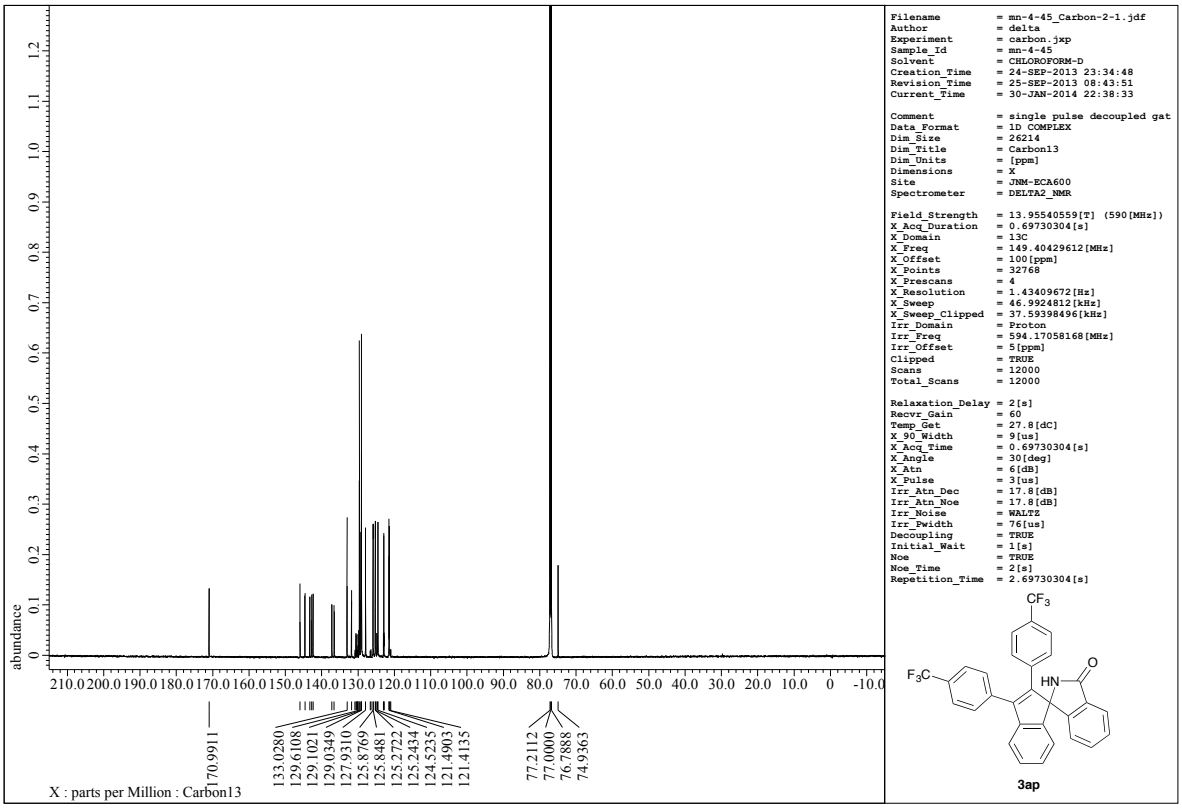

abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.8780
7.8734
7.8688
7.8631
7.2909
7.2772
7.1868
6.7851
6.6191
6.1533
2.3790
2.1605
X : parts per Million : Proton
Filename = mn-4-38_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-38
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2013 09:27:23
Revision_Time = 30-JAN-2014 22:35:49
Current_Time = 30-JAN-2014 22:36:12
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 40
Temp_Get = 25.2[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
Me
Me
HN
O
3an

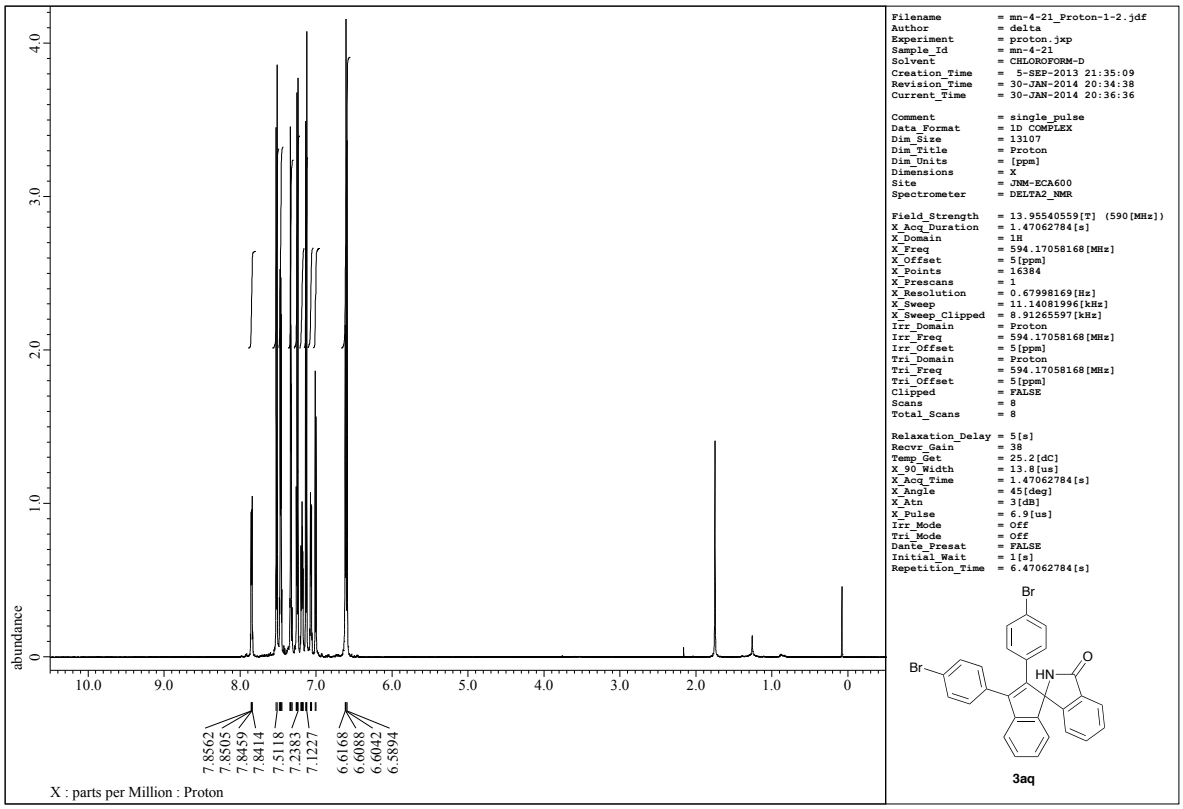

abundance
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
171.0583
137.2418
132.6536
129.3133
129.1597
128.7853
128.5838
128.5358
126.8272
124.1491
122.4789
121.6343
121.2215
77.2112
77.0000
76.7888
74.8691
21.3368
21.0968
X : parts per Million : Carbon13
Filename = mn-4-38_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-38
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2013 09:30:13
Revision_Time = 10-SEP-2013 09:46:10
Current_Time = 30-JAN-2014 22:35:16
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 26.2[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
Me
Me
HN
O
3an

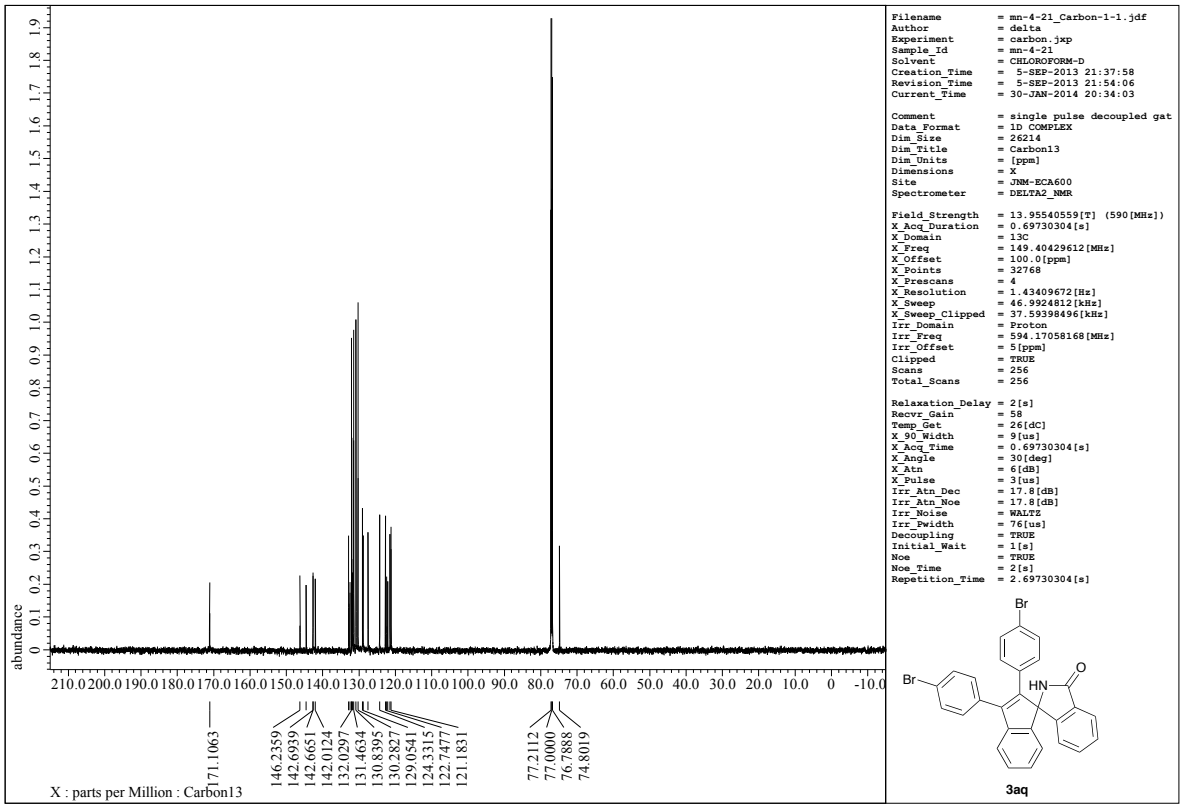

abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.8871
7.8803
7.8723
7.3310
7.3172
6.9178
6.9029
6.6512
6.5367
6.1247
3.8347
3.6562
X : parts per Million : Proton
Filename = mn-4-46_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-46
Solvent = CHLOROFORM-D
Creation_Time = 11-SEP-2013 17:19:22
Revision_Time = 30-JAN-2014 22:41:41
Current_Time = 30-JAN-2014 22:42:04
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 44
Temp_Get = 25.6[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
OMe
MeO
HN
O
3ao

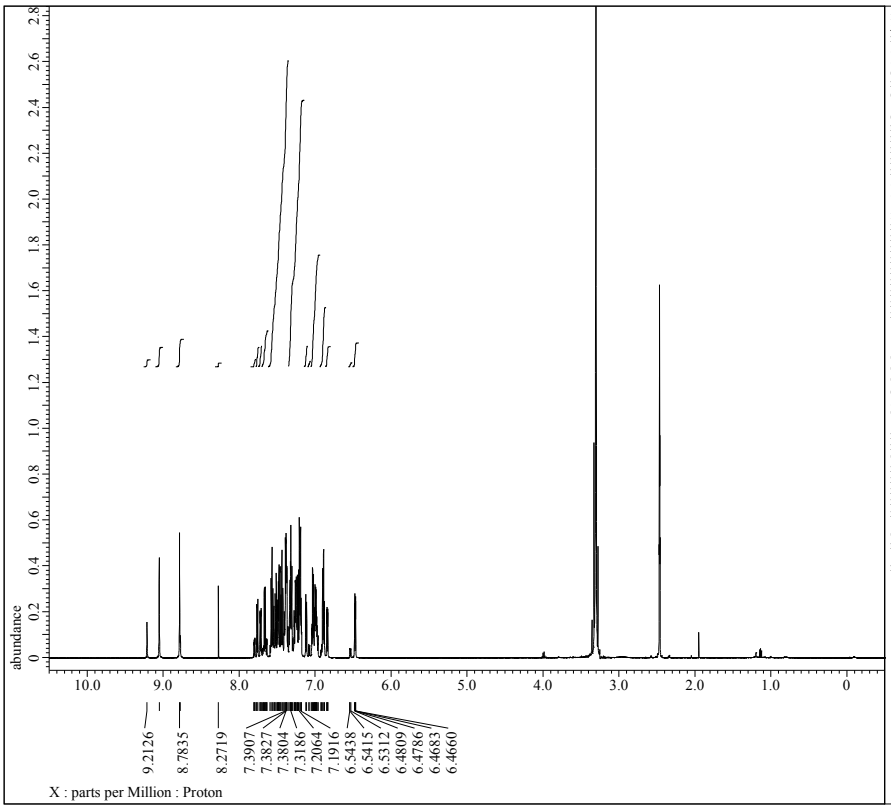

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
171.0583
159.2038
158.7911
147.2438
143.8458
141.2637
130.5707
129.9660
128.8141
126.7504
124.1683
121.6247
114.0992
113.5905
77.2112
77.0000
76.7888
74.7923
55.2204
54.9900
X : parts per Million : Carbon13
Filename = mn-4-46_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-46
Solvent = CHLOROFORM-D
Creation_Time = 11-SEP-2013 17:22:11
Revision_Time = 11-SEP-2013 17:38:41
Current_Time = 30-JAN-2014 22:41:14
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 26.7[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
OMe
MeO
HN
O
3ao


abundance
X : parts per Million : Proton
7.8725
7.6596
7.6470
7.4926
7.3587
7.3449
7.2717
7.2579
7.2248
7.1092
7.1034
7.0508
6.8242
6.8128
6.4740
6.4385
Filename = mn-4-45_Proton-2-5.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-45
Solvent = CHLOROFORM-D
Creation_Time = 24-SEP-2013 23:26:48
Revision_Time = 30-JAN-2014 22:40:33
Current_Time = 30-JAN-2014 22:40:37
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 44
Temp_Get = 27[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
CF3
F3C
HN
O
3ap

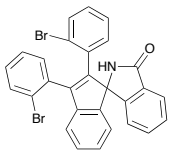

abundance
X : parts per Million : Carbon13
170.9911
133.0280
129.6108
129.1021
129.0349
127.9310
125.8769
125.8481
125.2722
125.2434
124.5235
121.4903
121.4135
77.2112
77.0000
76.7888
74.9363
Filename = mn-4-45_Carbon-2-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-45
Solvent = CHLOROFORM-D
Creation_Time = 24-SEP-2013 23:34:48
Revision_Time = 25-SEP-2013 08:43:51
Current_Time = 30-JAN-2014 22:38:33
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 12000
Total_Scans = 12000
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 27.8[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
CF3
F3C
HN
O
3ap


abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.8562
7.8505
7.8459
7.8414
7.5118
7.2383
7.1227
6.6168
6.6088
6.6042
6.5894
X : parts per Million : Proton
Filename = mn-4-21_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-21
Solvent = CHLOROFORM-D
Creation_Time = 5-SEP-2013 21:35:09
Revision_Time = 30-JAN-2014 20:34:38
Current_Time = 30-JAN-2014 20:36:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 38
Temp_Get = 25.2[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
Br
Br
HN
O
3aq

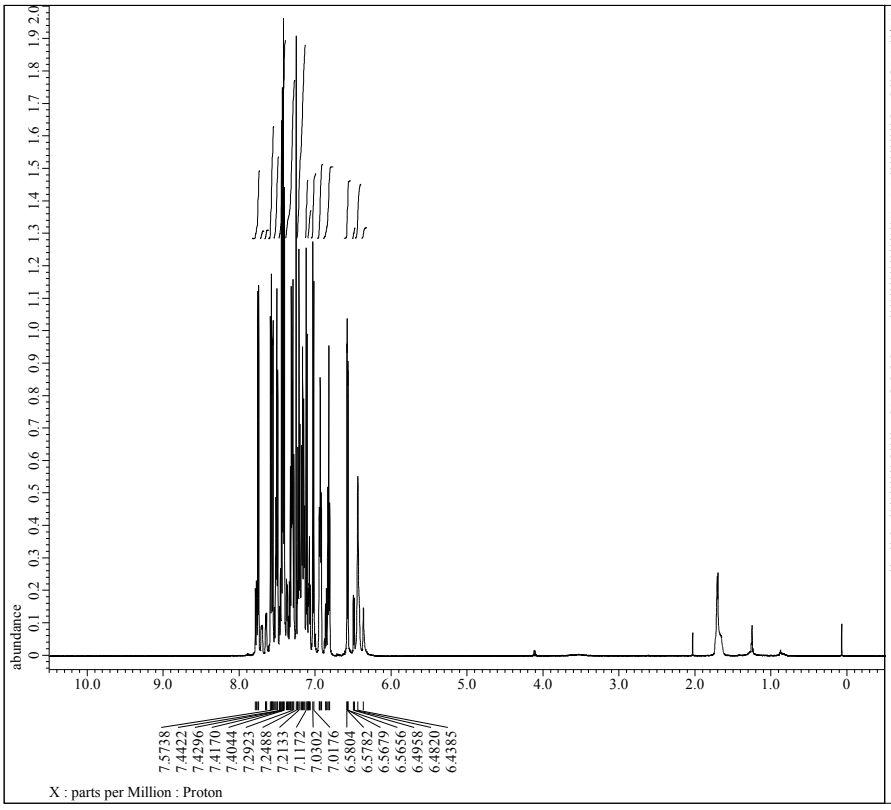

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
171.1063
146.2359
142.6939
142.6651
142.0124
132.0297
131.4634
130.8395
130.2827
129.0541
124.3315
122.7477
121.1831
77.2112
77.0000
76.7888
74.8019
X : parts per Million : Carbon13
Filename = mn-4-21_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-21
Solvent = CHLOROFORM-D
Creation_Time = 5-SEP-2013 21:37:58
Revision_Time = 5-SEP-2013 21:54:06
Current_Time = 30-JAN-2014 20:34:03
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 26[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
Br
Br
HN
O
3aq



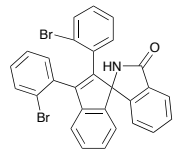

```
Filename          = mn-4-34_Proton-2-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-4-34-vt
Solvent           = DMSO-D6
Creation_Time     =  7-SEP-2013 17:29:27
Revision_Time     =  6-FEB-2014 14:15:14
Current_Time      =  6-FEB-2014 14:16:44

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 26.4[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



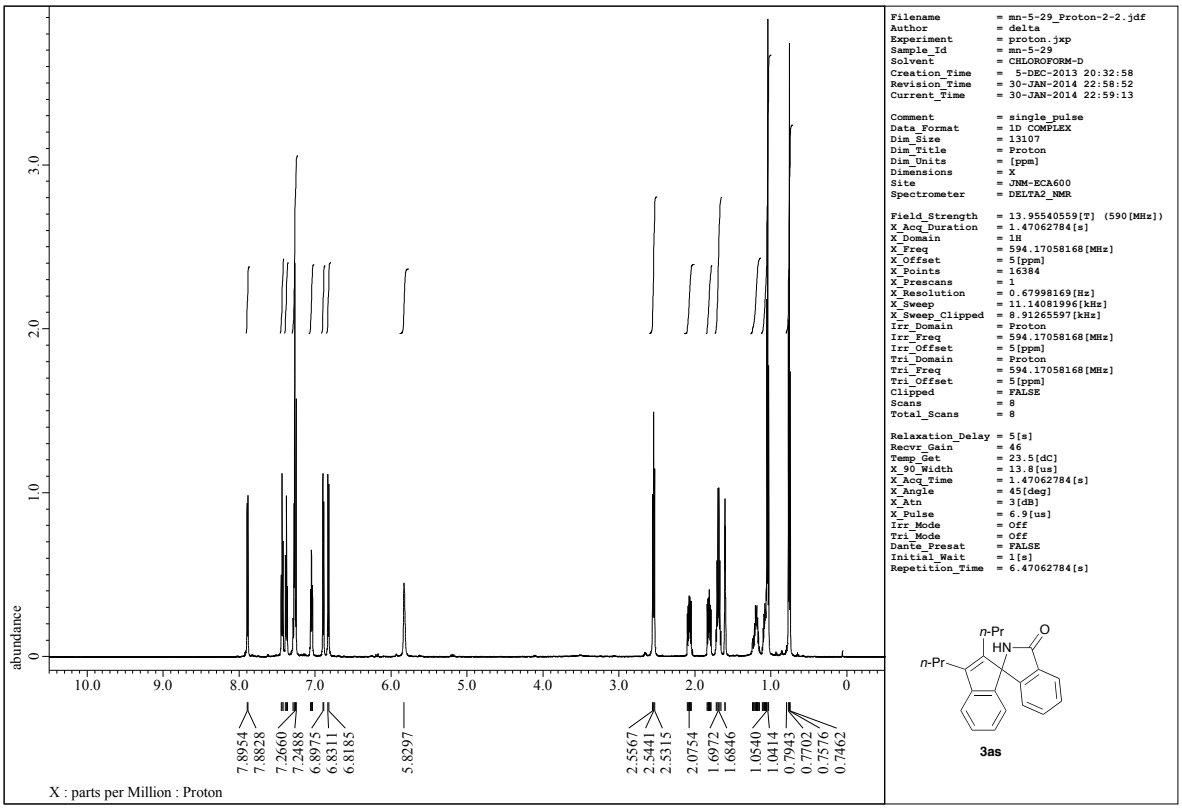
3ar (DMSO-d_6)

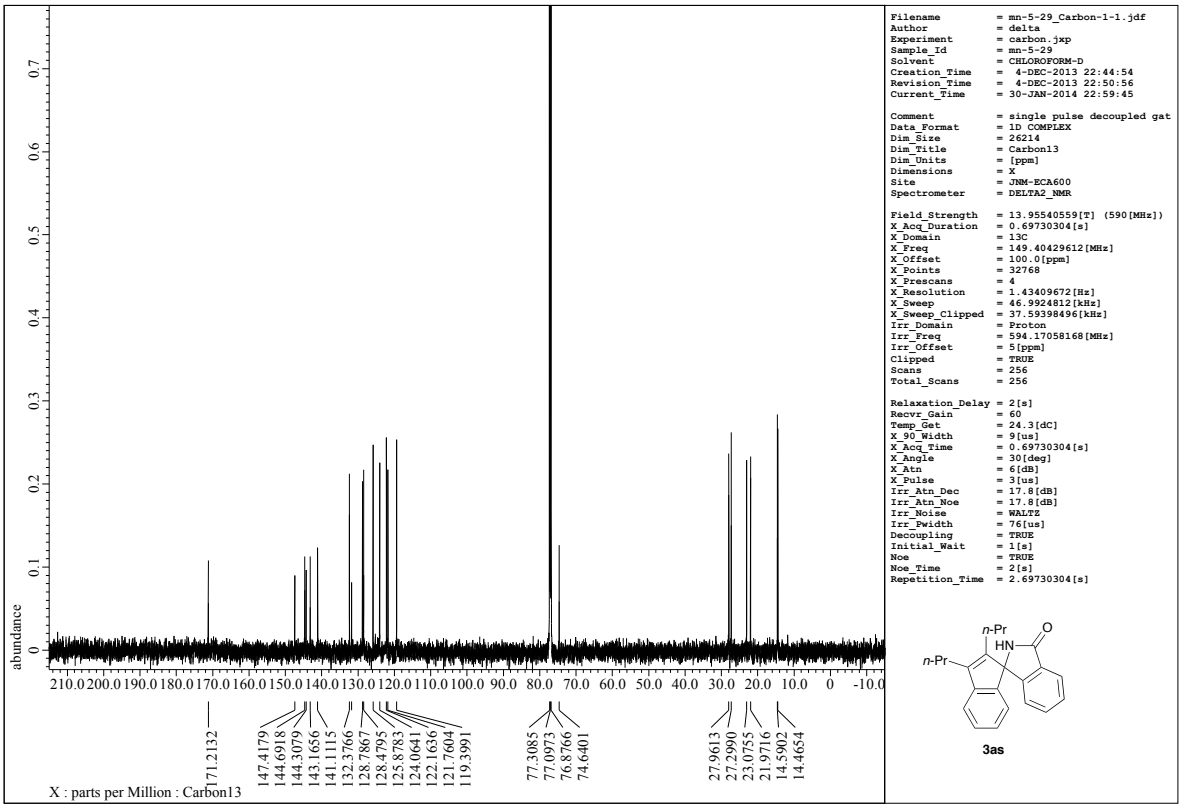


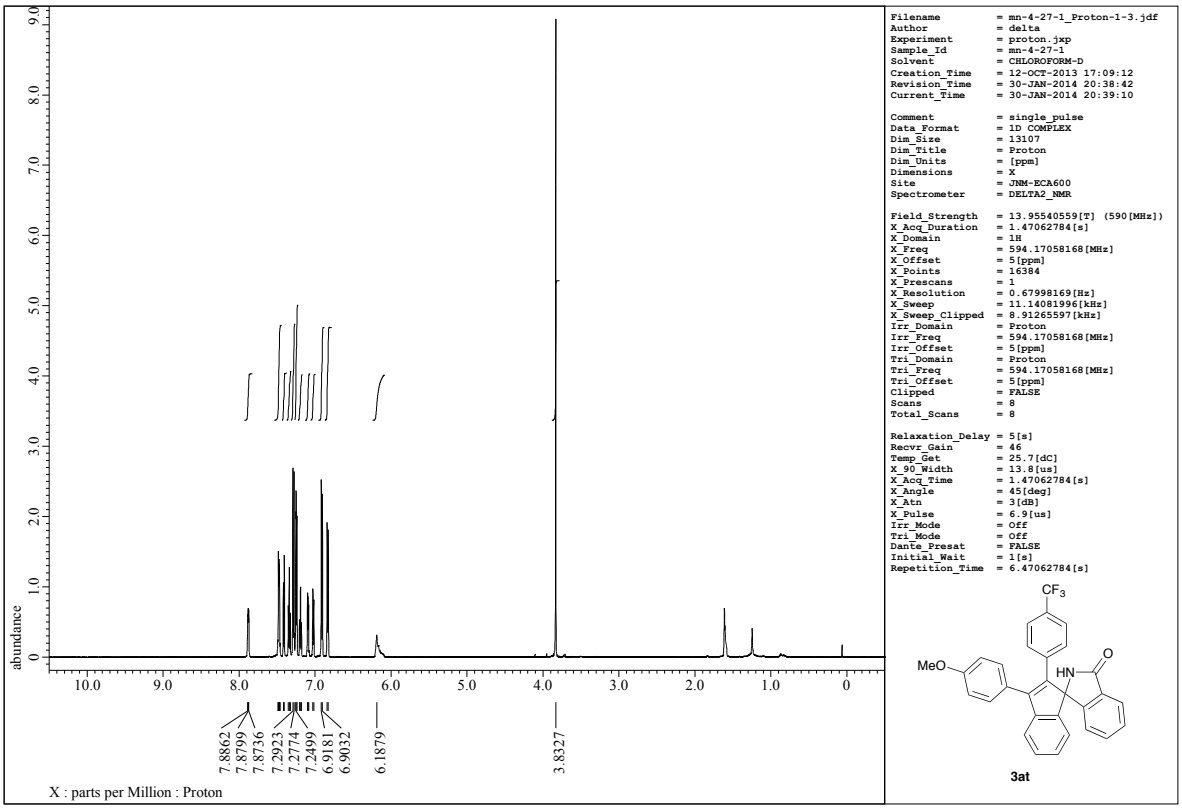

```
Filename          = mn-4-34_Proton-2-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-4-34
Solvent           = CHLOROFORM-D
Creation_Time     = 11-SEP-2013 10:21:45
Revision_Time     =  6-FEB-2014 14:18:05
Current_Time      =  6-FEB-2014 14:18:34

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 25.5[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



3ar ($CDCl_3$)

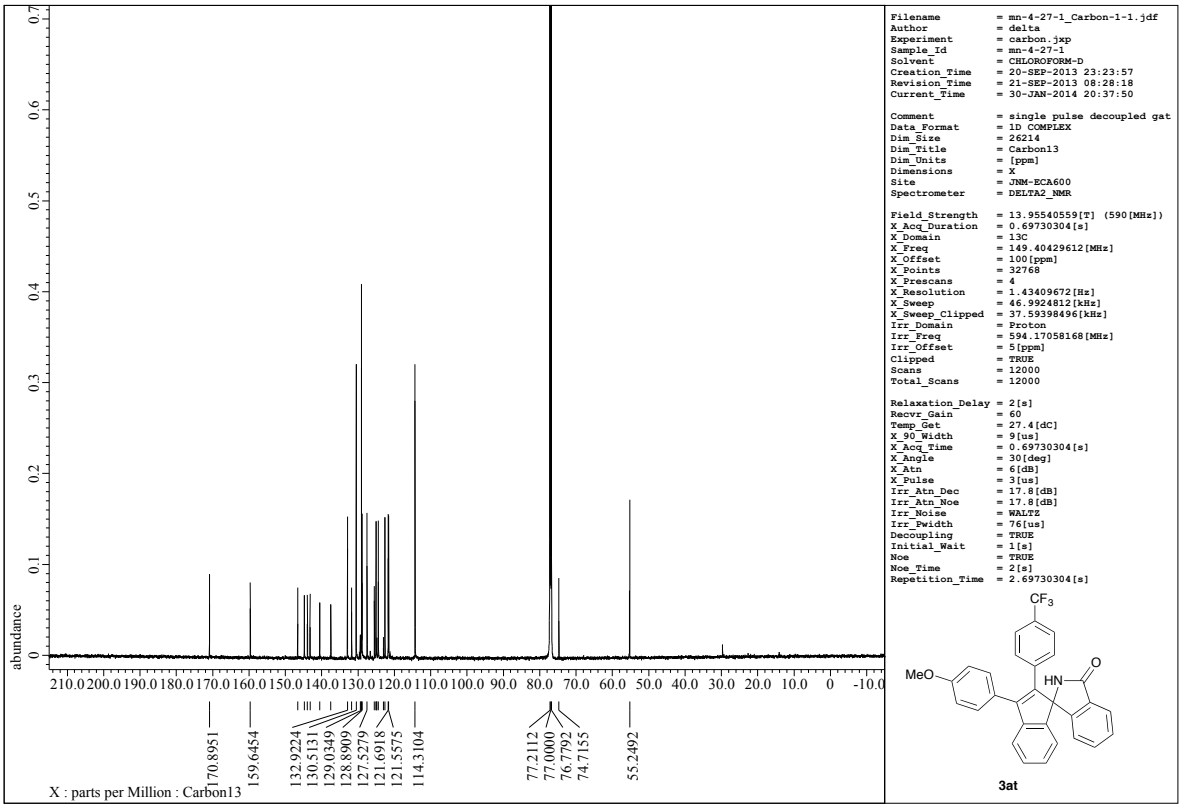

abundance
X : parts per Million : Proton
7.8954
7.8828
7.2660
7.2488
6.8975
6.8311
6.8185
5.8297
2.5567
2.5441
2.5315
2.0754
1.6972
1.6846
1.0540
1.0414
0.7943
0.7702
0.7576
0.7462
Filename = mn-5-29_Proton-2-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-5-29
Solvent = CHLOROFORM-D
Creation_Time = 5-DEC-2013 20:32:58
Revision_Time = 30-JAN-2014 22:58:52
Current_Time = 30-JAN-2014 22:59:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 23.5[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
n-Pr
HN
O
n-Pr
3as


abundance
X : parts per Million : Carbon13
171.2132
147.4179
144.6918
144.3079
143.1656
141.1115
132.3766
128.7867
128.4795
125.8783
124.0641
122.1636
121.7604
119.3991
77.3085
77.0973
76.8766
74.6401
27.9613
27.2990
23.0755
21.9716
14.5902
14.4654
Filename = mn-5-29_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-5-29
Solvent = CHLOROFORM-D
Creation_Time = 4-DEC-2013 22:44:54
Revision_Time = 4-DEC-2013 22:50:56
Current_Time = 30-JAN-2014 22:59:45
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.3[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
n-Pr
HN
O
n-Pr
3as



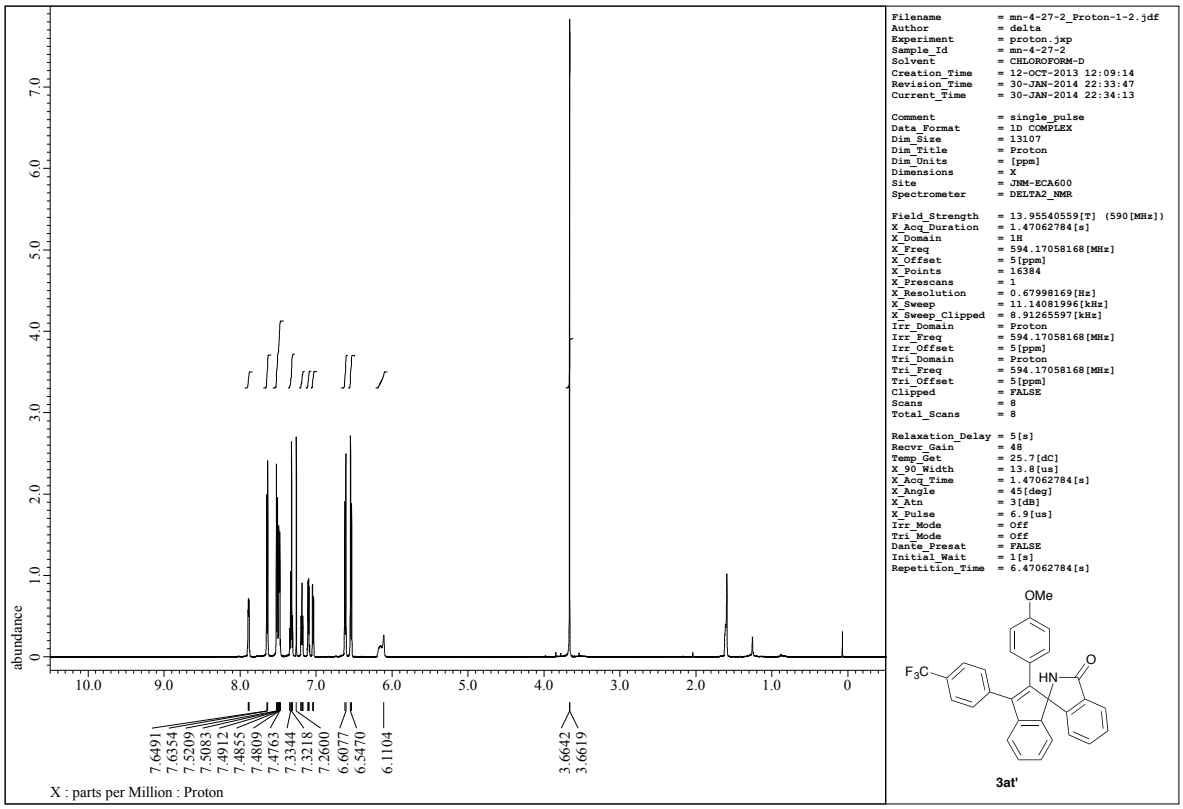

```
Filename          = mn-4-27-1_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-4-27-1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2013 17:09:12
Revision_Time     = 30-JAN-2014 20:38:42
Current_Time      = 30-JAN-2014 20:39:10

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 25.7[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```

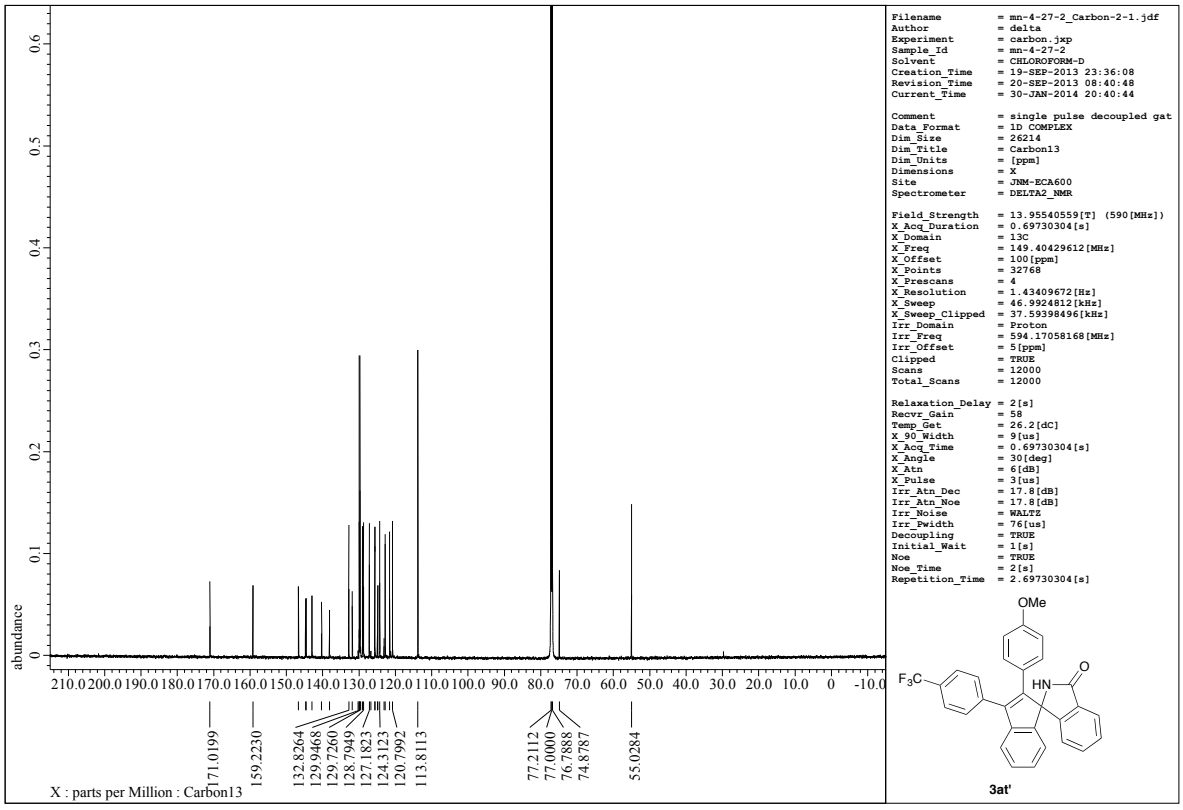

3at

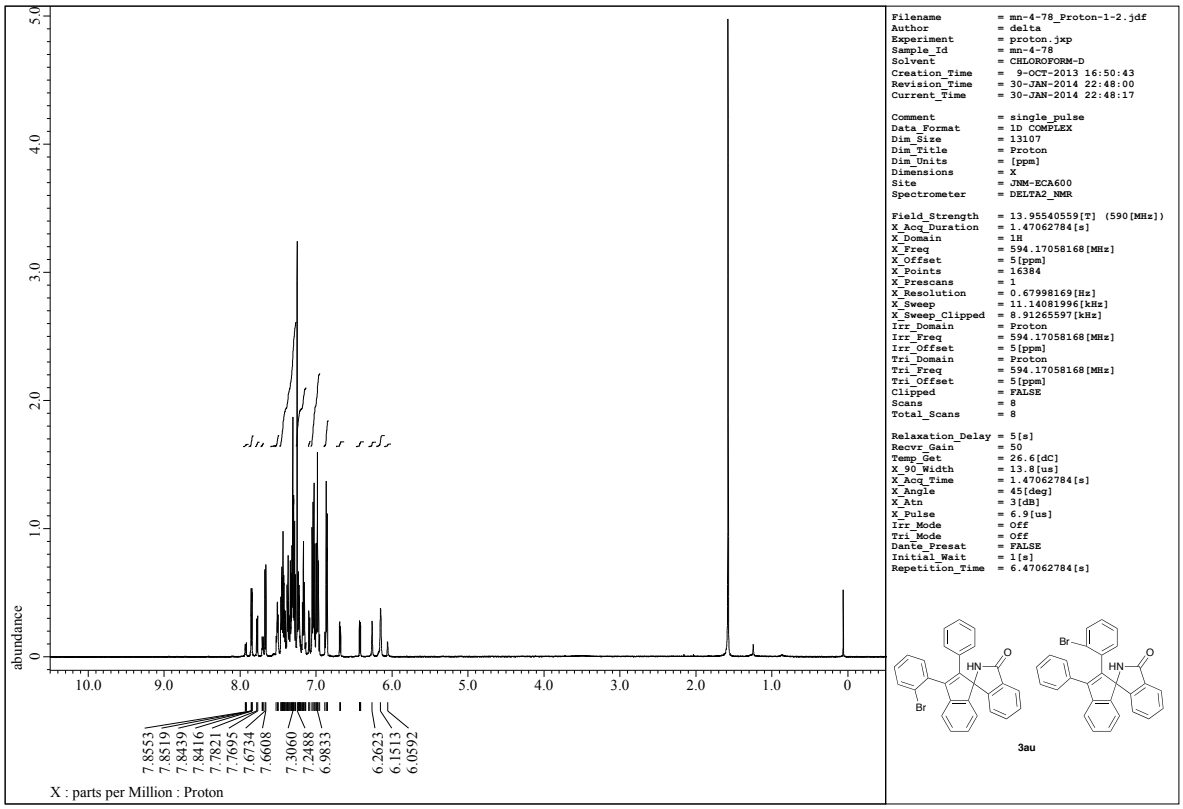


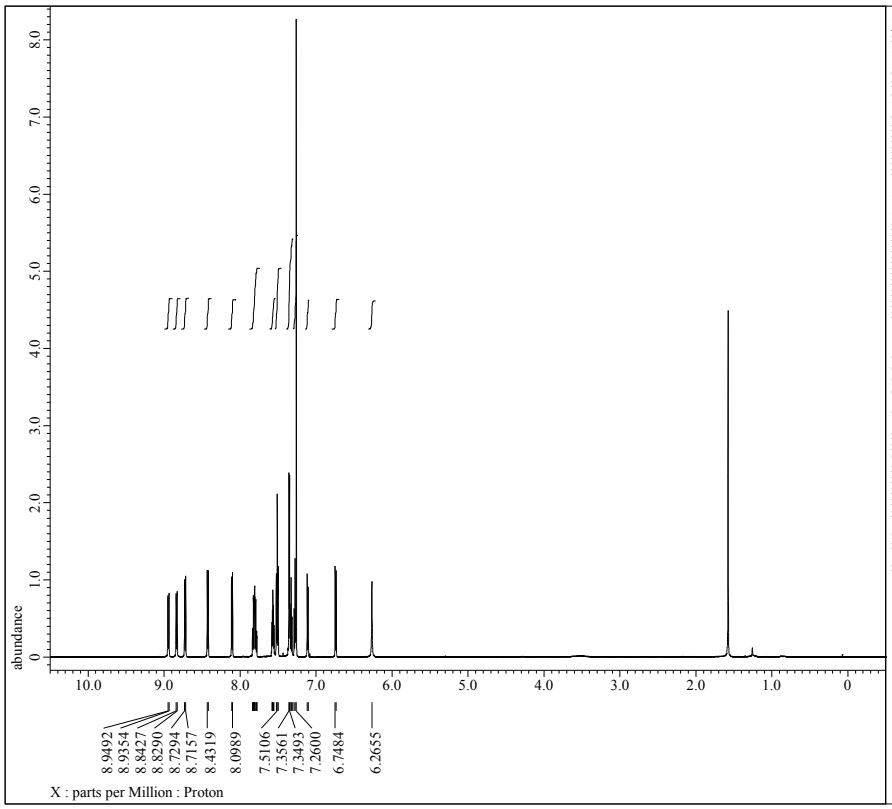

```
Filename          = mn-4-27-1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-4-27-1
Solvent           = CHLOROFORM-D
Creation_Time     = 20-SEP-2013 23:23:57
Revision_Time     = 21-SEP-2013 08:28:18
Current_Time      = 30-JAN-2014 20:37:50

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 12000
Total_Scans       = 12000

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 27.4[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```


3at

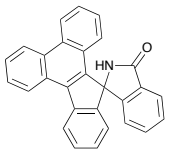

abundance
X : parts per Million : Proton
7.6491
7.6354
7.5209
7.5083
7.4912
7.4855
7.4809
7.4763
7.3344
7.3218
7.2600
6.6077
6.5470
6.1104
3.6642
3.6619
Filename = mn-4-27-2_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-27-2
Solvent = CHLOROFORM-D
Creation_Time = 12-OCT-2013 12:09:14
Revision_Time = 30-JAN-2014 22:33:47
Current_Time = 30-JAN-2014 22:34:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 25.7[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
OMe
F3C
HN
O
3at'


abundance
X : parts per Million : Carbon13
171.0199
159.2230
132.8264
129.9468
129.7260
128.7949
127.1823
124.3123
120.7992
113.8113
77.2112
77.0000
76.7888
74.8787
55.0284
Filename = mn-4-27-2_Carbon-2-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-27-2
Solvent = CHLOROFORM-D
Creation_Time = 19-SEP-2013 23:36:08
Revision_Time = 20-SEP-2013 08:40:48
Current_Time = 30-JAN-2014 20:40:44
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 12000
Total_Scans = 12000
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 26.2[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
OMe
F3C
HN
O
3at'

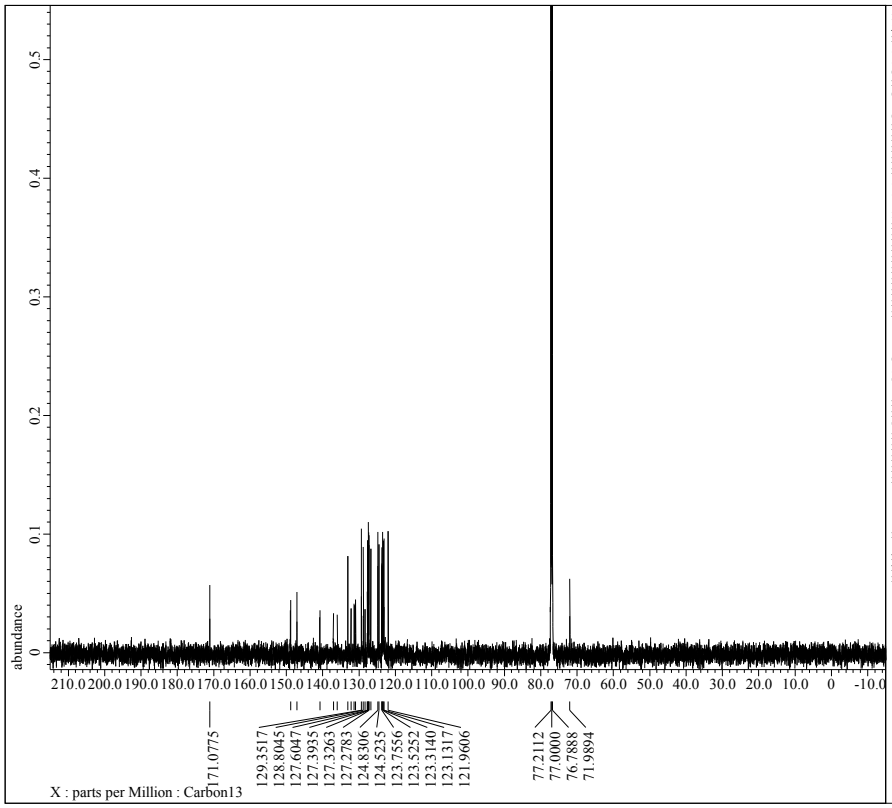

abundance
5.0
4.0
3.0
2.0
1.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.8553
7.8519
7.8439
7.8416
7.7821
7.7695
7.6734
7.6608
7.3060
7.2488
6.9833
6.2623
6.1513
6.0592
X : parts per Million : Proton
Filename = mn-4-78_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-78
Solvent = CHLOROFORM-D
Creation_Time = 9-OCT-2013 16:50:43
Revision_Time = 30-JAN-2014 22:48:00
Current_Time = 30-JAN-2014 22:48:17
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 26.6[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
HN
O
Br
HN
O
Br
3au



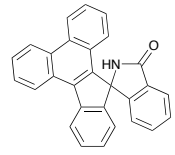

```
Filename          = mn-4-85_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-4-85
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2013 12:08:10
Revision_Time     = 30-JAN-2014 22:49:17
Current_Time      = 30-JAN-2014 22:49:32

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 24.7[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



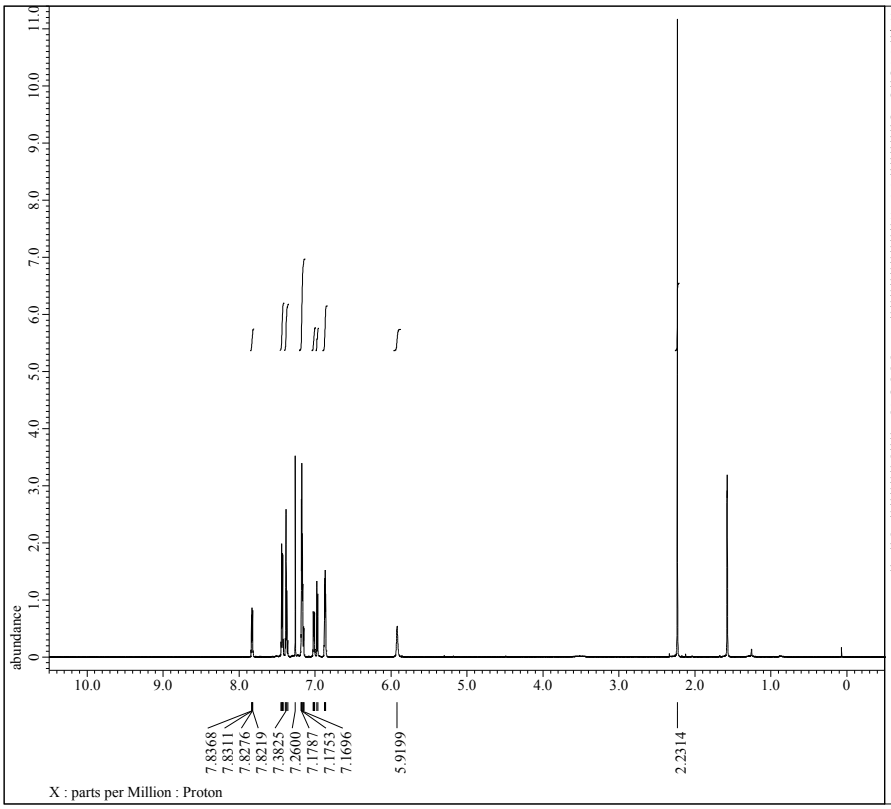
5



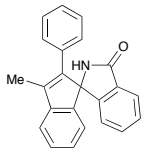

```
Filename          = mn-4-85_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-4-85
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2013 12:15:53
Revision_Time     = 17-OCT-2013 12:41:52
Current_Time      = 30-JAN-2014 22:48:56

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 480
Total_Scans       = 480

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25.6[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



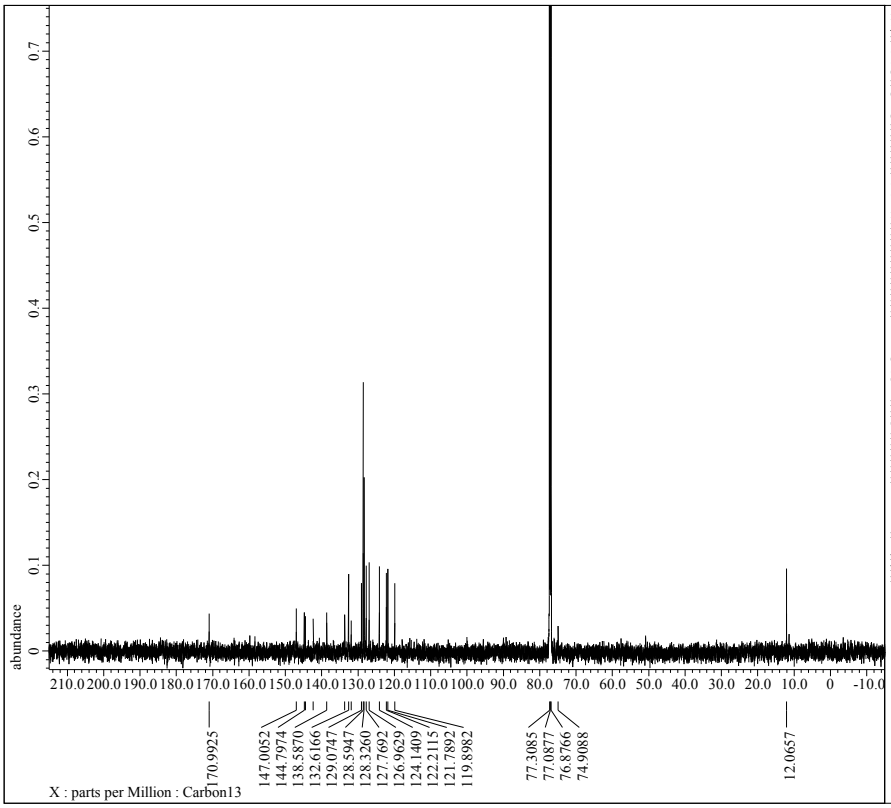
5



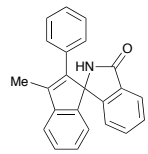

```
Filename          = mn-4-77-1_Proton-2-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-4-77-1
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2013 14:27:59
Revision_Time     = 30-JAN-2014 22:44:52
Current_Time      = 30-JAN-2014 22:45:08

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 52
Temp_Get          = 23.3[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



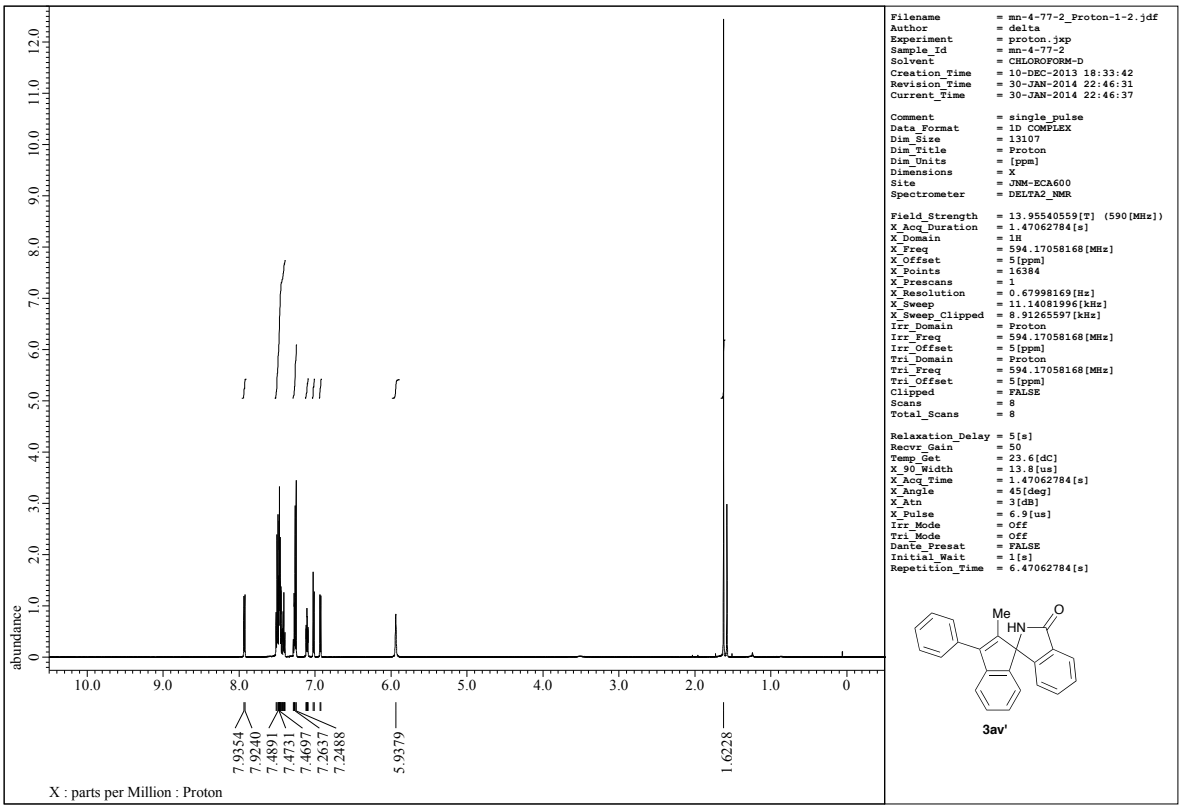
3av

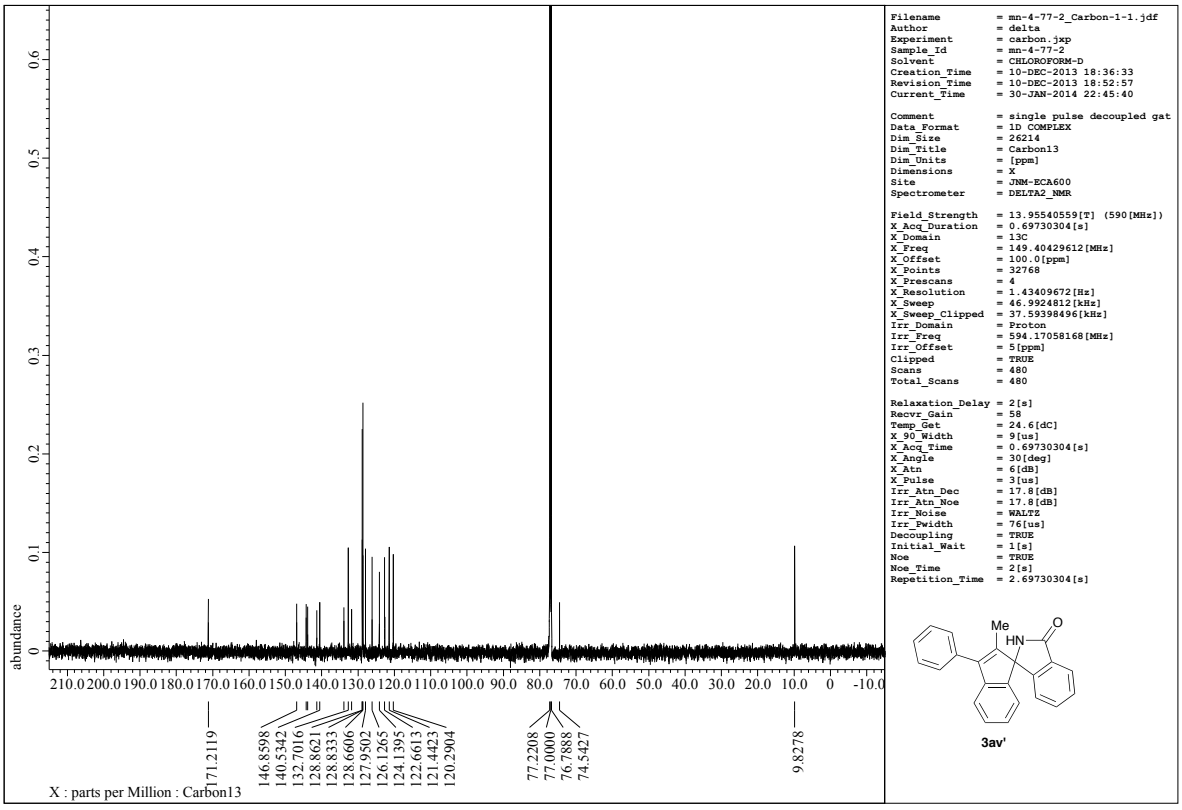


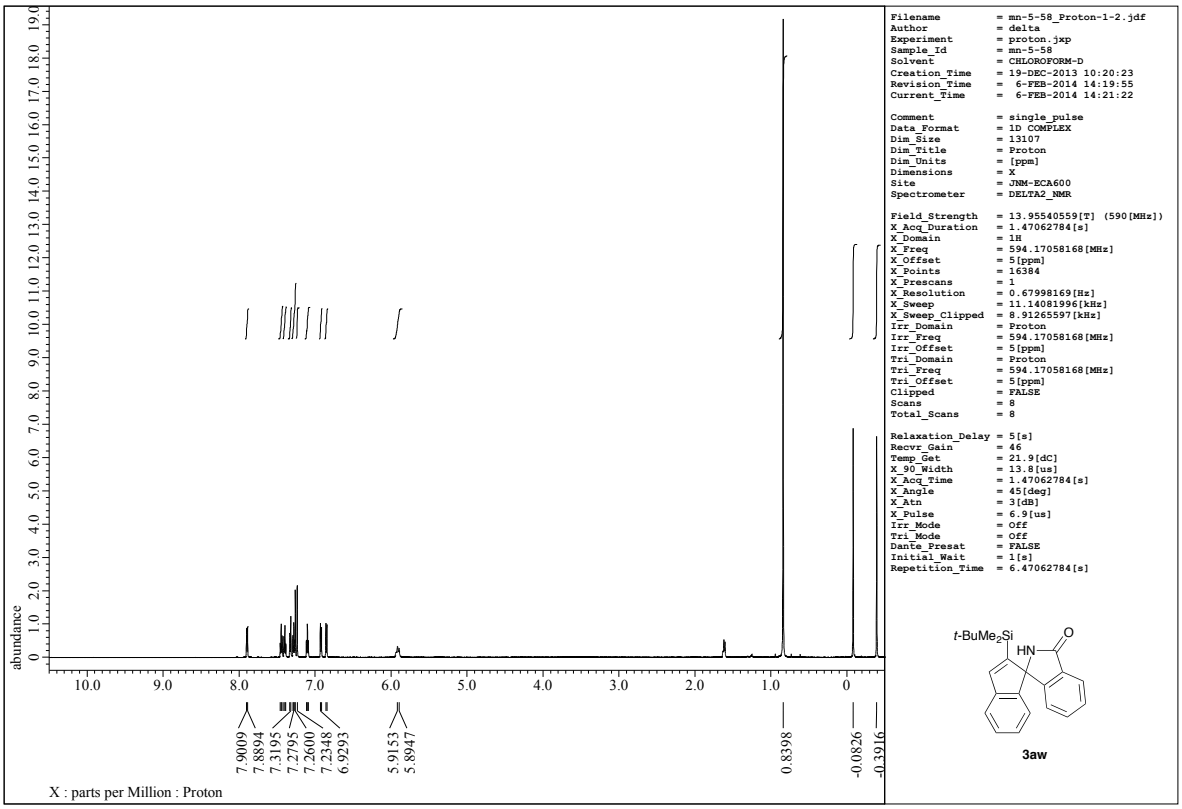

```
Filename          = mn-4-77-1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-4-77-1
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2013 10:28:10
Revision_Time     = 10-DEC-2013 10:37:21
Current_Time      = 30-JAN-2014 22:44:29

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 320
Total_Scans       = 320

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```

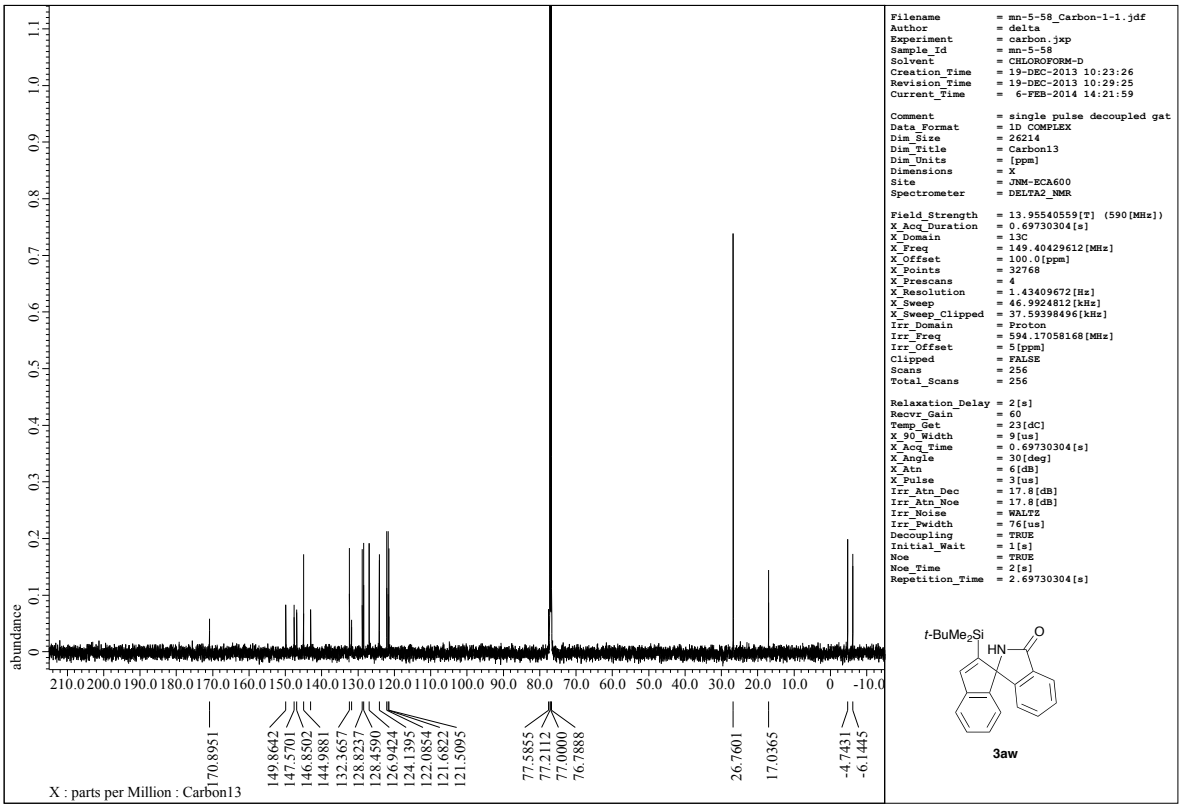


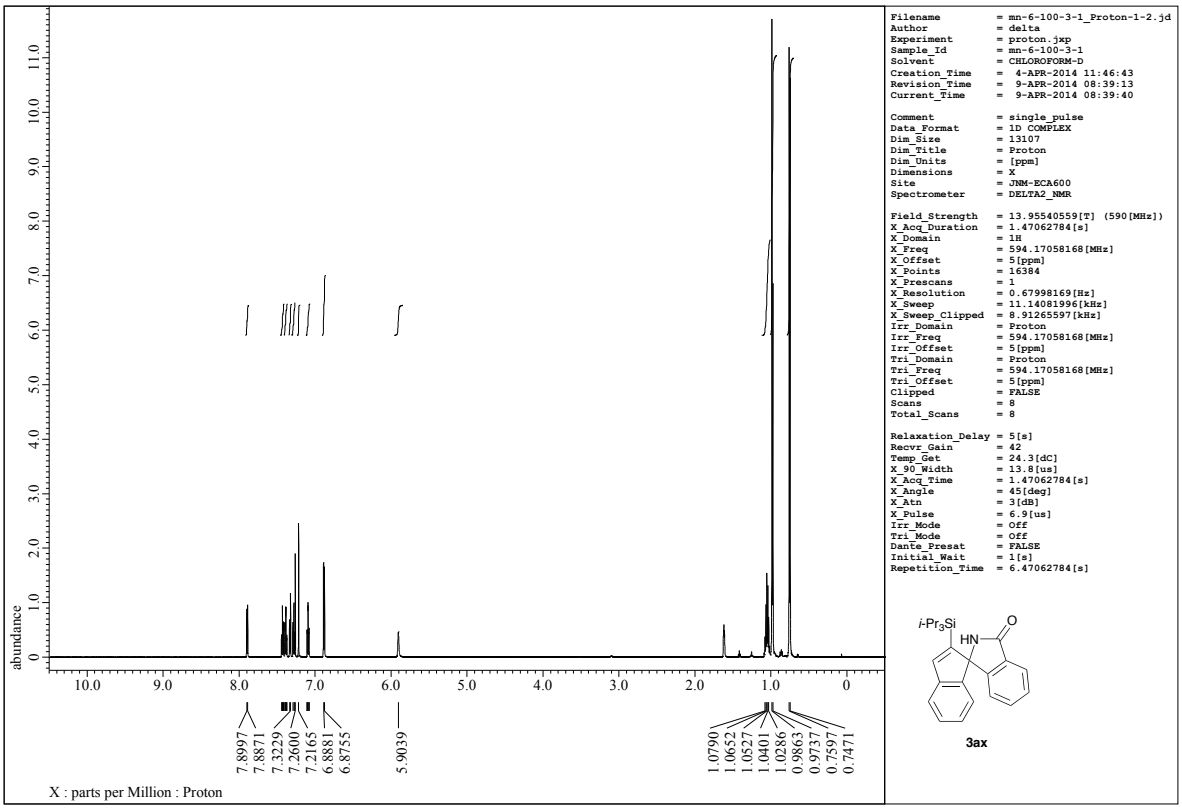
3av

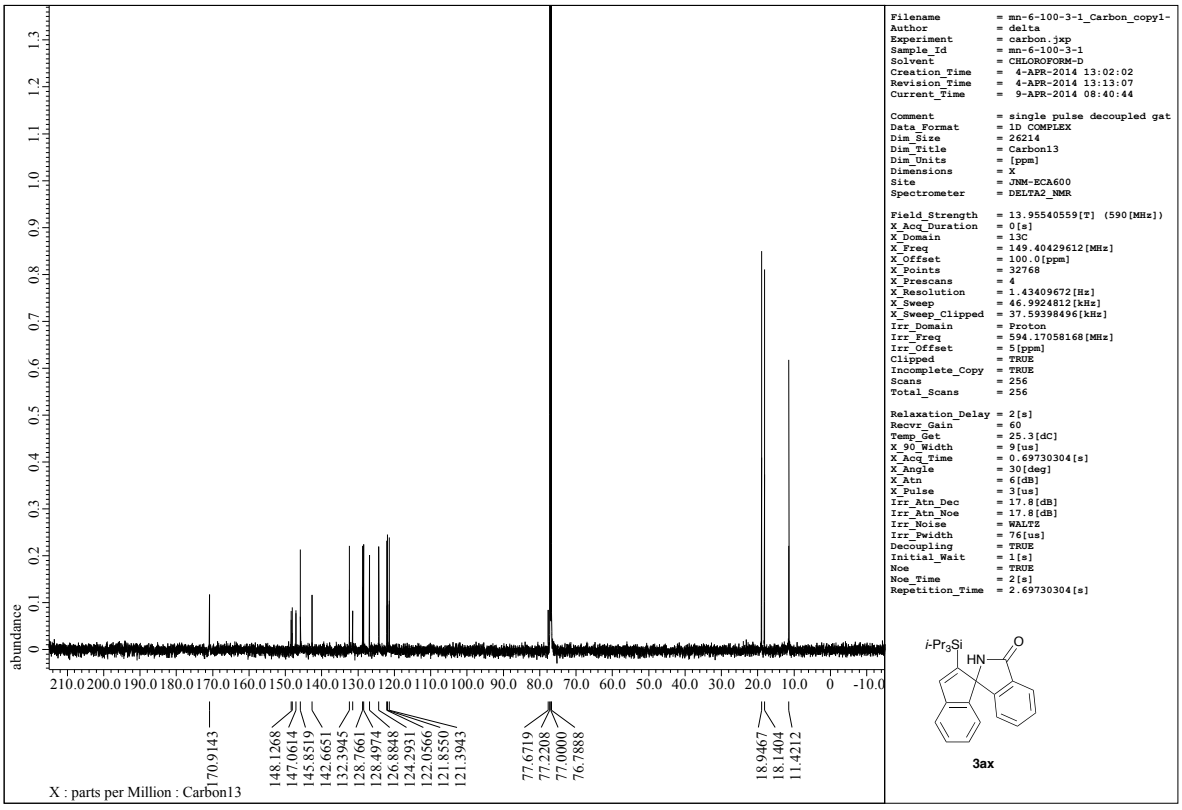

abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.9354
7.9240
7.4891
7.4731
7.4697
7.2637
7.2488
5.9379
1.6228
X : parts per Million : Proton
Filename = mn-4-77-2_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-4-77-2
Solvent = CHLOROFORM-D
Creation_Time = 10-DEC-2013 18:33:42
Revision_Time = 30-JAN-2014 22:46:31
Current_Time = 30-JAN-2014 22:46:37
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 23.6[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
Me
HN
O
3av'

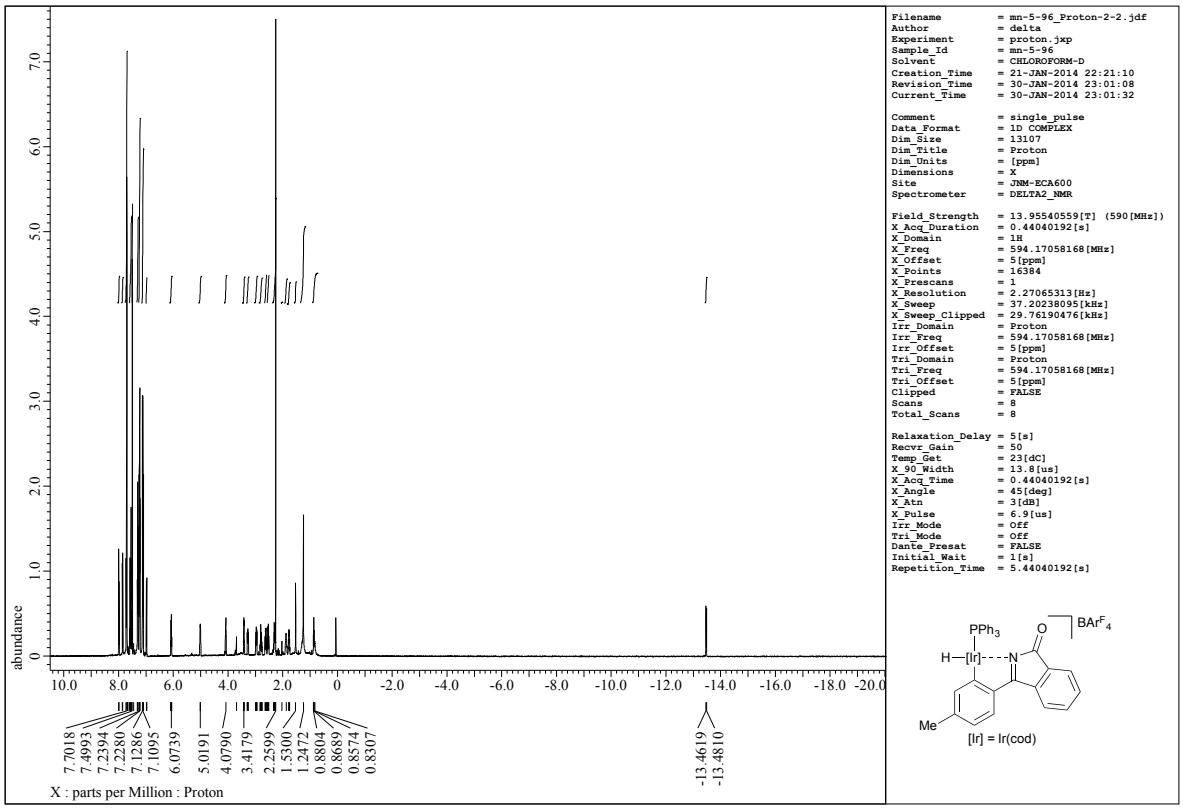

abundance
0.6
0.5
0.4
0.3
0.2
0.1
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
171.2119
146.8598
140.5342
132.7016
128.8621
128.8333
128.6606
127.9502
126.1265
124.1395
122.6613
121.4423
120.2904
77.2208
77.0000
76.7888
74.5427
9.8278
X : parts per Million : Carbon13
Filename = mn-4-77-2_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-4-77-2
Solvent = CHLOROFORM-D
Creation_Time = 10-DEC-2013 18:36:33
Revision_Time = 10-DEC-2013 18:52:57
Current_Time = 30-JAN-2014 22:45:40
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 480
Total_Scans = 480
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 24.6[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
Me
HN
O
3av'

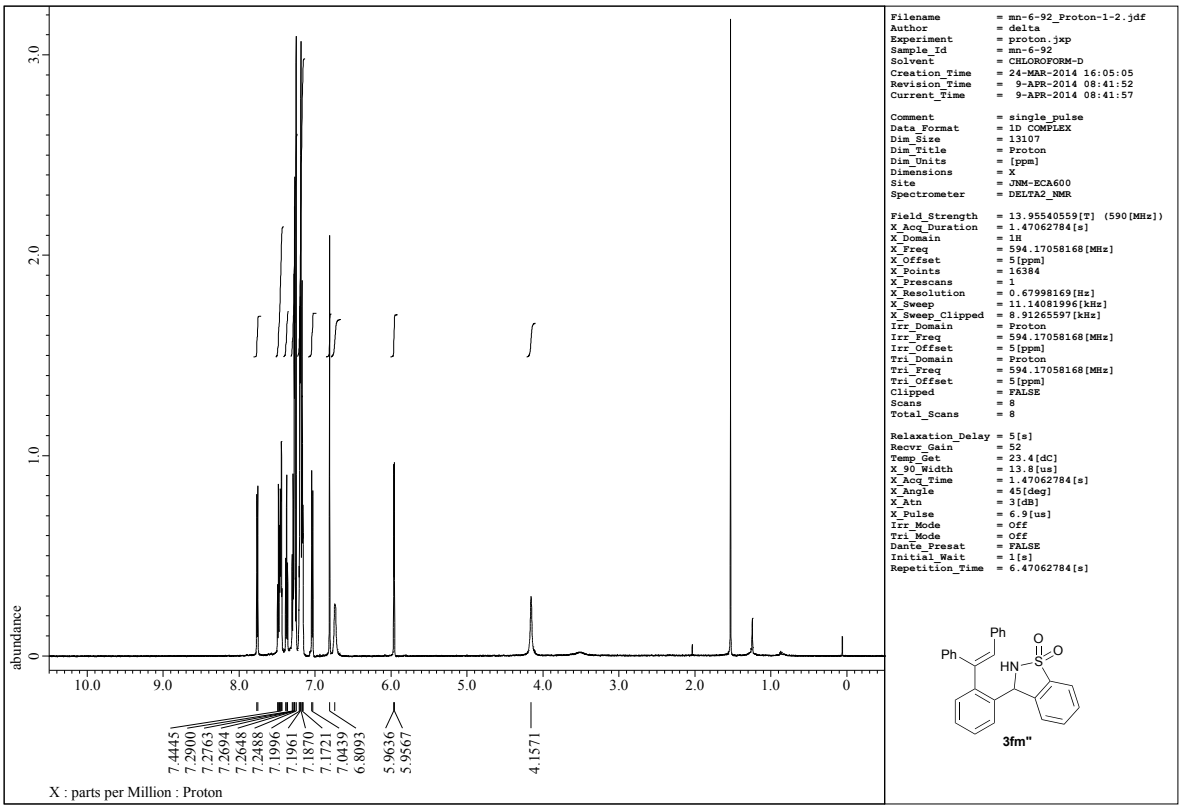

Filename = mn-5-58_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-5-58
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2013 10:20:23
Revision_Time = 6-FEB-2014 14:19:55
Current_Time = 6-FEB-2014 14:21:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 21.9[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
t-BuMe2Si
HN
O
3aw
abundance
7.9009
7.8894
7.3195
7.2795
7.2600
7.2348
6.9293
5.9153
5.8947
0.8398
-0.0826
-0.3916
X : parts per Million : Proton

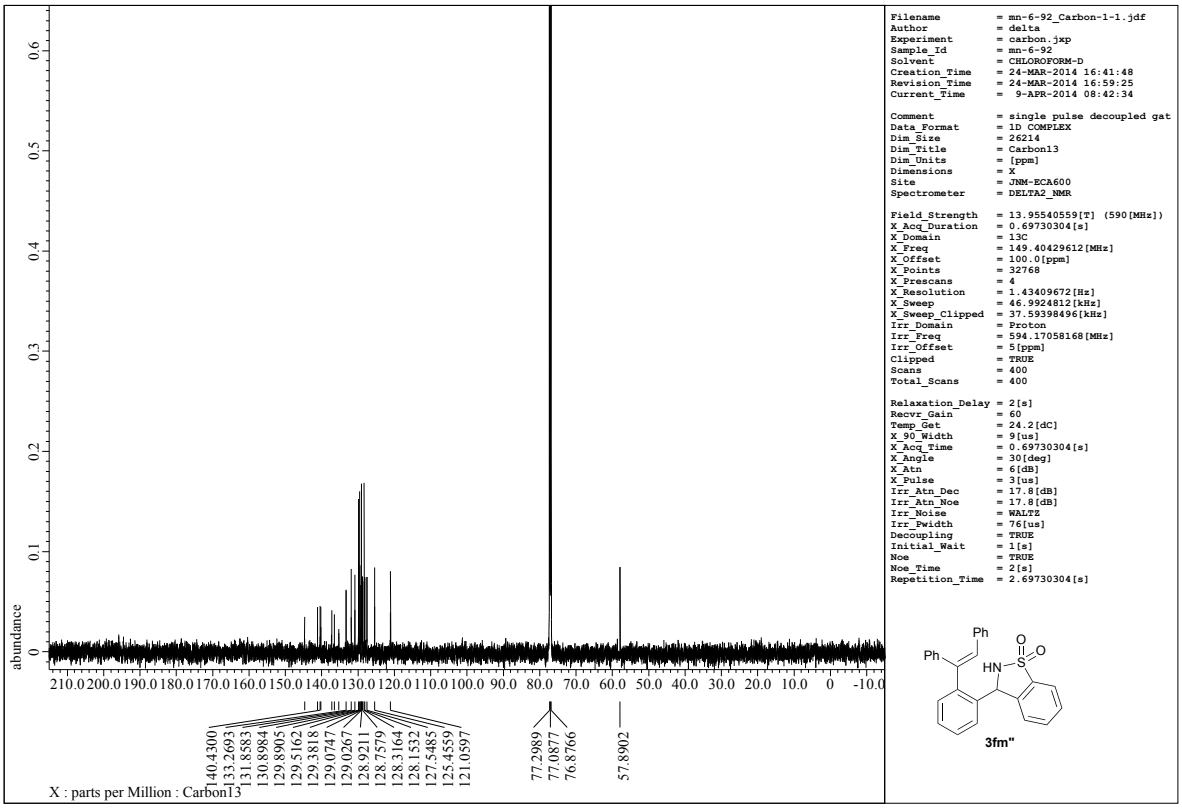

Filename = mn-5-58_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-5-58
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2013 10:23:26
Revision_Time = 19-DEC-2013 10:29:25
Current_Time = 6-FEB-2014 14:21:59
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
t-BuMe2Si
HN
O
3aw
abundance
170.8951
149.8642
147.5701
146.8502
144.9881
132.3657
128.8237
128.4590
126.9424
124.1395
122.0854
121.6822
121.5095
77.5855
77.2112
77.0000
76.7888
26.7601
17.0365
-4.7431
-6.1445
X : parts per Million : Carbon13


abundance
X : parts per Million : Proton
7.8997
7.8871
7.3229
7.2600
7.2165
6.8881
6.8755
5.9039
1.0790
1.0652
1.0527
1.0401
1.0286
0.9863
0.9737
0.7597
0.7471
Filename = mn-6-100-3-1_Proton-1-2.jd
Author = delta
Experiment = proton.jxp
Sample_Id = mn-6-100-3-1
Solvent = CHLOROFORM-D
Creation_Time = 4-APR-2014 11:46:43
Revision_Time = 9-APR-2014 08:39:13
Current_Time = 9-APR-2014 08:39:40
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 42
Temp_Get = 24.3[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
i-Pr3Si
HN
O
3ax


abundance
X : parts per Million : Carbon13
170.9143
148.1268
147.0614
145.8519
142.6651
132.3945
128.7661
128.4974
126.8848
124.2931
122.0566
121.8550
121.3943
77.6719
77.2208
77.0000
76.7888
18.9467
18.1404
11.4212
Filename = mn-6-100-3-1_Carbon_copy1-
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-6-100-3-1
Solvent = CHLOROFORM-D
Creation_Time = 4-APR-2014 13:02:02
Revision_Time = 4-APR-2014 13:13:07
Current_Time = 9-APR-2014 08:40:44
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Incomplete_Copy = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 25.3[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
i-Pr3Si
HN
O
3ax


abundance
10.0 8.0 6.0 4.0 2.0 0 -2.0 -4.0 -6.0 -8.0 -10.0 -12.0 -14.0 -16.0 -18.0 -20.0
7.7018
7.4993
7.2394
7.2280
7.1286
7.1095
6.0739
5.0191
4.0790
3.4179
2.2599
1.5300
1.2472
0.8804
0.8689
0.8574
0.8307
-13.4619
-13.4810
X : parts per Million : Proton
Filename = mn-5-96_Proton-2-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-5-96
Solvent = CHLOROFORM-D
Creation_Time = 21-JAN-2014 22:21:10
Revision_Time = 30-JAN-2014 23:01:08
Current_Time = 30-JAN-2014 23:01:32
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.44040192[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 2.27065313[Hz]
X_Sweep = 37.20238095[kHz]
X_Sweep_Clipped = 29.76190476[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 23[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 0.44040192[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 5.44040192[s]
PPh3
O
BArF4
H—[Ir]
N
Me
[Ir] = Ir(cod)


abundance
3.0
2.0
1.0
0
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.4445
7.2900
7.2763
7.2694
7.2648
7.2488
7.1996
7.1961
7.1870
7.1721
7.0439
6.8093
5.9636
5.9567
4.1571
X : parts per Million : Proton
Filename = mn-6-92_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = mn-6-92
Solvent = CHLOROFORM-D
Creation_Time = 24-MAR-2014 16:05:05
Revision_Time = 9-APR-2014 08:41:52
Current_Time = 9-APR-2014 08:41:57
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 1.47062784[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.67998169[Hz]
X_Sweep = 11.14081996[kHz]
X_Sweep_Clipped = 8.91265597[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 594.17058168[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 52
Temp_Get = 23.4[dC]
X_90_Width = 13.8[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.9[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
Ph
Ph
O
O
HN
S
3fm"


abundance
0.6
0.5
0.4
0.3
0.2
0.1
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
140.4300
133.2693
131.8583
130.8984
129.8905
129.5162
129.3818
129.0747
129.0267
128.9211
128.7579
128.3164
128.1532
127.5485
125.4559
121.0597
77.2989
77.0877
76.8766
57.8902
X : parts per Million : Carbon13
Filename = mn-6-92_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = mn-6-92
Solvent = CHLOROFORM-D
Creation_Time = 24-MAR-2014 16:41:48
Revision_Time = 24-MAR-2014 16:59:25
Current_Time = 9-APR-2014 08:42:34
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 400
Total_Scans = 400
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.2[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
Ph
Ph
O
O
HN
S
3fm"