Supporting Information

Iridium-Catalyzed Branch-Selective Hydroarylation of Vinyl Ethers via C–H Bond Activation

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Contents of Supporting Information:

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) 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: quint, quintet: sept, septet: m, multiplet: br, broad. High-resolution mass spectra (TOF-MS) were obtained with a Bruker micrOTOF spectrometer. Flash column chromatography was performed with Silica Gel 60 N (spherical, neutral) (Cica-Reagent). Preparative thin-layer chromatography was performed with Silica Gel 60 PF_{254} (Merck). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque.

2. Materials

Toluene, dioxane, and dichloromethane were purified by passing through a neutral alumina column under N_2. Iridium complexes $[IrCl(cod)]_2$,[1] $[IrCl(coe)_2]_2$,[2] and $[IrCl((S,S)\text{-Fc-tfb*})]_2$[3] were prepared according to the reported procedures. $NaBAr^F_4$ was prepared according to the reported procedures.[4]

3. Preparation of 1 and vinyl ethers 2

Compounds **1a** and **1h** were purchased from commercial suppliers and used as received. Compounds **1b** (CAS: 4467-06-5), **1c** (CAS: 5957-90-4), **1d** (CAS: 58861-53-3), **1e** (CAS: 4373-61-9), **1f** (CAS: 3319-99-1), **1g** (46181-30-0), and **1a-*d*$_5$** (CAS: 105664-48-0) were prepared according to the reported procedures.[5] Compounds **1i** (CAS: 15903-58-9),[6] **1j** (CAS: 71885-44-4),[7] **1k** (CAS: 3376-33-8),[8] and **1l** (CAS: 2743-00-2)[9] were prepared according to the reported procedures. Vinyl ethers **2a–c** and **2m–t** were purchased from commercial suppliers and used as received. Vinyl ethers **2d** (CAS: 935-04-6), **2e** (CAS: 108388-36-9), **2f** (CAS: 766-94-9), **2g** (CAS: 1005-62-5), **2h** (CAS: 4024-19-5), **2i** (CAS: 351-93-9), **2j** (CAS: 1074-56-2), **2k** (CAS: 1005-40-9), and **2l** (CAS: 934-21-4) were prepared according to the reported procedures.[10]

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4. General procedure for iridium-catalyzed hydroarylation of *n*-butyl vinyl ether (2a) with 2-phenylpyridine (1a) (Table 1)

$NaBAr^F_4$ (9.2 mg calculated as the dihydrate, 0.010 mmol, 10 mol%) and a transition metal catalyst (0.0050 mmol of the metal, 5 mol % of the metal) were placed in a Schlenk tube under N_2. Toluene (0.4 mL), 2-phenylpyridine (**1a**) (15.5 mg, 0.10 mmol), and *n*-butyl vinyl ether (**2a**) (15.0 mg, 0.15 mmol) were added to the tube successively, and the mixture was stirred at 80 ºC for 12 h. 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 yields of the products were determined by ^{1}H NMR using nitromethane as an internal standard.

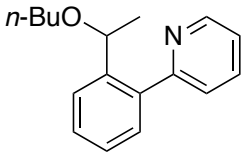
5. General procedure for iridium-catalyzed hydroarylation of vinyl ethers 2 with 2-phenylpyridines 1 (Tables 2 and 3)

$NaBAr^F_4$ (18.4 mg, 0.020 mmol, 10 mol%) and $[IrCl(cod)]_2$ (3.4 mg, 0.0050 mmol, 5 mol % of Ir) were placed in a Schlenk tube under N_2. Toluene (0.8 mL), phenylpyridine **1** (0.20 mmol), and vinyl ether **2** (0.30 mmol) were added to the tube successively, and the mixture was stirred at 60 ºC for 12 h. 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 yields of the products were determined by ^{1}H NMR using nitromethane as an internal standard. The residue was subjected to preparative TLC on silica gel to give **3**. For isolation of the product, a solution of EtOAc and $CHCl_3$ (1:30) was used as an eluent unless otherwise specified.

6. General procedure for iridium-catalyzed hydroarylation of vinyl ethers 2 with aromatic compounds 1 (Table 4)

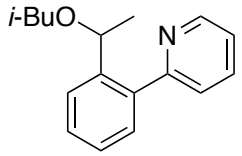
$NaBAr^F_4$ (18.4 mg, 0.02 mmol, 10 mol%) and $[IrCl(cod)]_2$ (3.4 mg, 0.005 mmol, 5 mol % of Ir) were placed in a Schlenk tube under nitrogen. Toluene (0.8 mL), an aromatic compound **1** (0.20 mmol), and vinyl ether **2** (0.60 mmol) were added to the tube successively, and the mixture was stirred at 80 ºC for 48 h. 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 yields of the products were determined by ^{1}H NMR using nitromethane as an internal standard. The residue was subjected to preparative TLC on silica gel to give **3**.

7. Characterization of the products



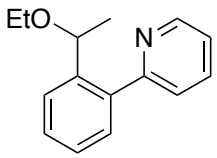
3aa

Compound 3aa (Table 2, entry 1: 51.1 mg, 99% yield). ^{1}H NMR ($CDCl_3$) δ 0.84 (t, *J* = 7.3 Hz, 3H), 1.22–1.35 (m, 2H), 1.40 (d, *J* = 6.4 Hz, 3H), 1.41–1.51 (m, 2H), 3.15 (dt, *J* = 7.8, 6.5 Hz, 1H), 3.22 (dt, *J* = 7.8, 6.8 Hz, 1H), 4.65 (q, *J* = 6.4 Hz, 1H), 7.26 (ddd, *J* = 7.8, 4.9, 1.2 Hz, 1H), 7.31–7.35 (m, 2H), 7.36 (dt, *J* = 6.4, 1.0 Hz, 1H), 7.42–7.47 (m, 1H), 7.64 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.75 (td, *J* = 7.8, 1.9 Hz, 1H), 8.68 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.3, 23.9, 32.0, 68.2, 73.5, 121.7, 124.1, 126.0, 126.9, 128.8, 129.3, 136.1, 139.8, 142.5, 149.1, 159.7. HRMS (ESI) calcd for $C_{17}H_{21}NNaO$ $(M+Na)^+$ 278.1515, found 278.1523.



3ab

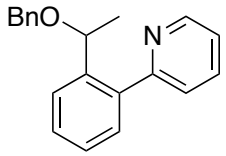
Compound 3ab (Table 2, entry 2: 49.9mg, 97% yield). ^{1}H NMR ($CDCl_3$) δ 0.82 (d, *J* = 6.6 Hz, 3H), 0.84 (d, *J* = 6.6 Hz, 3H), 1.40 (d, *J* = 6.3 Hz, 3H), 1.75 (sept, *J* = 6.6 Hz, 1H), 2.93 (dd, *J* = 9.2, 6.3 Hz, 1H), 2.98 (dd, *J* = 9.2, 7.0 Hz, 1H), 4.63 (q, *J* = 6.3 Hz, 1H), 7.26 (ddd, *J* = 7.8, 4.9, 1.2 Hz, 1H), 7.31–7.35 (m, 2H), 7.36 (dt, *J* = 6.6, 1.0 Hz, 1H), 7.42–7.46 (m, 1H), 7.64 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.75 (td, *J* = 7.8, 1.8 Hz, 1H), 8.68 (ddd, *J* = 4.9, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 19.4, 19.5, 23.9, 28.6, 73.6, 75.3, 121.7, 124.1, 126.1, 126.8, 128.7, 129.2, 136.1, 139.7, 142.5, 149.1, 159.7. HRMS (ESI) calcd for $C_{17}H_{21}NNaO$ $(M+Na)^+$ 278.1515, found 278.1513.



3ac

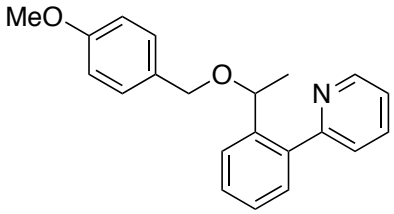
Compound 3ac (Table 2, entry 3: 41.0 mg, 90% yield). A solution of EtOAc and hexane (1:5) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 1.09 (t, *J* = 7.0 Hz, 3H), 1.41 (d, *J* = 6.5 Hz, 3H), 3.22 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.28 (dq, *J* = 9.3, 7.0 Hz, 1H), 4.68 (q, *J* = 6.5 Hz, 1H), 7.26 (ddd, *J* = 7.8, 4.9, 1.2 Hz, 1H), 7.31–7.35 (m, 2H), 7.36 (dt, *J* = 6.6, 1.0 Hz, 1H), 7.42–7.47 (m, 1H), 7.65 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.75 (td, *J* = 7.8, 1.8 Hz, 1H), 8.68 (ddd, *J* = 4.9, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 15.3, 23.8, 63.6, 73.4, 121.7, 124.1, 126.0,

126.9, 128.8, 129.3, 136.1, 139.8, 142.4, 149.1, 159.7. HRMS (ESI) calcd for $C_{15}H_{17}NNaO$ $(M+Na)^+$ 250.1202, found 250.1208.



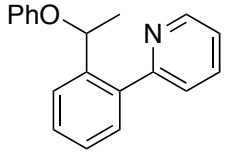
3ad

Compound 3ad (Table 2, entry 4: 52.8 mg, 91% yield). A solution of EtOAc and $CHCl_3$ (1/30) was used as an eluent for preparative TLC on silica gel twice. ^1H NMR ($CDCl_3$) δ 1.48 (d, *J* = 6.5 Hz, 3H), 4.18 (d, *J* = 11.8 Hz, 1H), 4.37 (d, *J* = 11.8 Hz, 1H), 4.79 (q, *J* = 6.5 Hz, 1H), 7.20–7.28 (m, 6H), 7.33 (dt, *J* = 6.4, 1.0 Hz, 1H), 7.36–7.39 (m, 2H), 7.47–7.51 (m, 1H), 7.71 (td, *J* = 7.7, 1.8 Hz, 1H), 7.75 (d, *J* = 7.7 Hz, 1H), 8.66 (ddd, *J* = 4.8, 1.8, 1.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 24.0, 70.2, 73.0, 121.7, 124.1, 126.3, 127.1, 127.2, 127.7, 128.2, 128.9, 129.4, 136.1, 138.7, 139.9, 142.0, 149.1, 159.4. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1362.



3ae

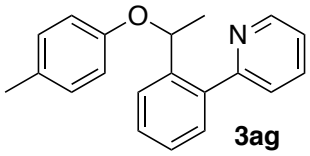
Compound 3ae (Table 2, entry 5: 61.5 mg, 96% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^1H NMR ($CDCl_3$) δ 1.46 (d, *J* = 6.3 Hz, 3H), 3.77 (s, 3H), 4.11 (d, *J* = 11.2 Hz, 1H), 4.29 (d, *J* = 11.2 Hz, 1H), 4.76 (q, *J* = 6.3 Hz, 1H), 6.79 (d, *J* = 8.7 Hz, 2H), 7.13 (d, *J* = 8.7 Hz, 2H), 7.25 (ddd, *J* = 7.8, 4.9, 1.1 Hz, 1H), 7.33 (dt, *J* = 6.4, 1.0 Hz, 1H), 7.35–7.38 (m, 2H), 7.46–7.51 (m, 1H), 7.71 (td, *J* = 7.8, 1.8 Hz, 1H), 7.74 (dd, *J* = 7.8, 1.1 Hz, 1H), 8.67 (ddd, *J* = 4.9, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 24.0, 55.2, 69.8, 72.7, 113.6, 121.7, 124.1, 126.3, 127.1, 128.9, 129.3, 129.4, 130.8, 136.1, 139.9, 142.1, 149.1, 159.0, 159.5. HRMS (ESI) calcd for $C_{21}H_{21}NNaO_2$ $(M+Na)^+$ 342.1465, found 342.1461.



3af

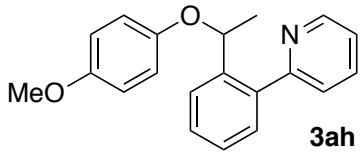
Compound 3af (Table 2, entry 6: 50.5 mg, 91% yield). A solution of EtOAc and hexane (1:3) was used as an eluent for preparative TLC on silica gel. ^1H NMR ($CDCl_3$) δ 1.58 (d, *J* = 6.3

Hz, 3H), 5.75 (q, *J* = 6.3 Hz, 1H), 6.80–6.84 (m, 3H), 7.12–7.16 (m, 2H), 7.29 (ddd, *J* = 7.7, 4.8, 1.1 Hz, 1H), 7.32–7.39 (m, 3H), 7.45 (dt, *J* = 6.6, 0.9Hz, 1H), 7.66 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.80 (td, *J* = 7.7, 1.9 Hz, 1H), 8.72 (ddd, *J* = 4.8, 1.9, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 23.8, 72.1, 115.8, 120.3, 121.9, 124.0, 125.8, 127.2, 129.0, 129.2, 129.5, 136.6, 138.6, 141.6, 149.1, 157.9, 159.6. HRMS (ESI) calcd for $C_{19}H_{17}NNaO$ $(M+Na)^+$ 298.1202, found 298.1198.



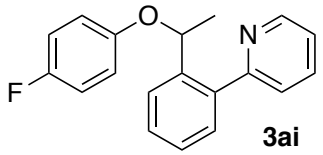
3ag

Compound 3ag (Table 2, entry 7: 56.2 mg, 98% yield). ^{1}H NMR ($CDCl_3$) δ 1.56 (d, *J* = 6.4 Hz, 3H), 2.20 (s, 3H), 5.68 (q, *J* = 6.4 Hz, 1H), 6.70 (d, *J* = 8.2 Hz, 2H), 6.93 (d, *J* = 8.2 Hz, 2H), 7.29 (ddd, *J* = 7.7, 4.9, 1.1 Hz, 1H), 7.31 (td, *J* = 7.7, 1.5 Hz, 1H), 7.34 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.37 (td, *J* = 7.7, 1.5 Hz, 1H), 7.44 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.65 (dd, *J* = 7.8, 1.1 Hz, 1H), 7.79 (td, *J* = 7.8, 1.8 Hz, 1H), 8.72 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 20.4, 23.8, 72.2, 115.6, 121.9, 124.0, 125.8, 127.1, 129.0, 129.4, 129.5, 129.6, 136.5, 138.6, 141.7, 149.1, 155.7, 159.6. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1361.



3ah

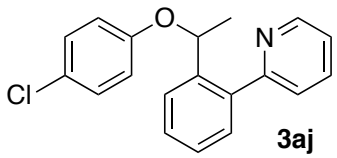
Compound 3ah (Table 2, entry 8: 56.9 mg, 93% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 1.56 (d, *J* = 6.4 Hz, 3H), 3.69 (s, 3H), 5.64 (q, *J* = 6.4 Hz, 1H), 6.70 (d, *J* = 9.1 Hz, 2H), 6.75 (d, *J* = 9.1 Hz, 2H), 7.29 (ddd, *J* = 7.7, 4.8, 1.0 Hz ,1H), 7.30–7.35 (m, 2H), 7.39 (t, *J* = 7.4 Hz, 1H), 7.42 (d, *J* = 6.5 Hz, 1H), 7.66 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.78 (td, *J* = 7.7, 1.8 Hz, 1H), 8.72 (ddd, *J* = 4.8, 1.8, 0.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 23.8, 55.6, 72.7, 114.4, 116.7, 121.9, 124.0, 125.9, 127.2, 129.0 129.5, 136.5, 138.7, 141.7, 149.1, 151.9, 153.5, 159.6. HRMS (ESI) calcd for $C_{20}H_{19}NNaO_2$ $(M+Na)^+$ 328.1308, found 328.1310.



3ai

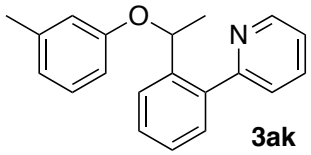
Compound 3ai (Table 2, entry 9: 57.8 mg, 90% yield, **3ai**/**4ai** = 94/6). ^{1}H NMR ($CDCl_3$) ^{1}H NMR ($CDCl_3$) δ 1.56 (d, *J* = 6.3 Hz, 3H), 5.71 (q, *J* = 6.3 Hz, 1H), 6.75–6.79 (m, 2H), 6.80–6.85 (m, 2H), 7.29 (ddd, *J* = 7.7, 4.9, 1.1 Hz ,1H), 7.31–7.36 (m, 2H), 7.39 (td, *J* = 7.2, 2.0 Hz, 1H), 7.43 (dt, *J* = 6.6, 1.0 Hz, 1H), 7.63 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.80 (td, *J* = 7.7, 1.9 Hz, 1H), 8.72 (ddd, *J* =

4.9, 1.9, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 23.8, 72.7, 115.5 (d, J_{F-C} = 23 Hz), 116.7 (d, J_{F-C} = 29 Hz), 122.0, 124.0, 125.7, 127.3, 129.0, 129.6, 136.7, 138.7, 141.3, 149.1, 153.9, 156.9 (d, J_{F-C} = 238 Hz), 159.5. HRMS (ESI) calcd for $C_{19}H_{16}FNNaO$ $(M+Na)^+$ 316.1108, found 316.1105.



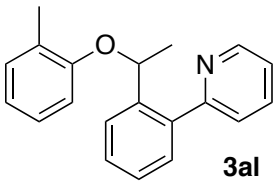
3aj

Compound 3aj (Table 2, entry 10: 57.8 mg, 93% yield). ^{1}H NMR ($CDCl_3$) δ 1.56 (d, *J* = 6.3 Hz, 3H), 5.74 (q, *J* = 6.3 Hz, 1H), 6.77 (d, *J* = 9.0 Hz, 2H), 7.08 (d, *J* = 9.0 Hz, 2H), 7.30 (ddd, *J* = 7.7, 4.8, 1.1 Hz , 1H), 7.31–7.36 (m, 2H), 7.38 (td, *J* = 6.5, 2.0 Hz, 1H), 7.44 (dt, *J* = 6.5, 0.9 Hz, 1H), 7.60 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.81 (td, *J* = 7.7, 1.8 Hz, 1H), 8.71 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 23.7, 72.5, 117.1, 122.0, 124.0, 125.1, 125.7, 127.4, 129.1, 129.6, 136.7, 138.6, 141.2, 149.1, 156.5, 159.5. HRMS (ESI) calcd for $C_{19}H_{16}ClNNaO$ $(M+Na)^+$ 332.0813, found 332.0814.



3ak

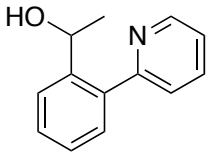
Compound 3ak (Table 2, entry 11: 54.6 mg, 91% yield, **3ak**/**4ak** = 98/2). ^{1}H NMR ($CDCl_3$) δ 1.59 (d, *J* = 6.4 Hz, 3H), 2.21 (s, 3H), 5.69 (q, *J* = 6.4 Hz, 1H), 6.59 (dd, *J* = 7.8, 2.1 Hz, 1H), 6.60–6.64 (m, 1H), 6.61 (s, 1H), 7.00 (t, *J* = 7.8 Hz, 1H), 7.28–7.33 (m, 2H), 7.35 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.38 (td, *J* = 7.8, 1.2 Hz, 1H), 7.45 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.60 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.80 (td, *J* = 7.7, 1.8 Hz, 1H), 8.73 (ddd, *J* = 4.8, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.4, 23.9, 72.0, 112.5, 116.6, 121.1, 121.9, 124.0, 125.9, 127.2, 128.9, 129.0, 129.4, 136.5, 138.7, 139.1, 141.7, 149.1, 157.8, 159.6. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1364.



3al

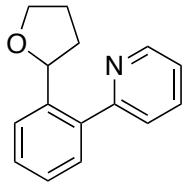
Compound 3al (Table 2, entry 12: 56.6 mg, 98% yield). ^{1}H NMR ($CDCl_3$) δ 1.59 (d, *J* = 6.8 Hz, 3H), 2.28 (s, 3H), 5.73 (q, *J* = 6.8 Hz, 1H), 6.61 (d, *J* = 7.5 Hz, 1H), 6.74 (t, *J* = 7.5 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 7.5 Hz, 1H), 7.29 (dd, *J* = 6.8, 4.8 Hz, 1H), 7.32 (dd, *J* = 8.1, 6.8 Hz, 1H), 7.36 (d, *J* = 6.8 Hz, 1H), 7.38 (dd, *J* = 8.1, 6.8 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.65 (d, *J* = 8.1 Hz, 1H), 7.79 (ddd, *J* = 8.1, 6.8, 2.0 Hz, 1H), 8.72 (dd, *J* = 4.8, 2.0 Hz, 1H); ^{13}C NMR

($CDCl_3$) δ 16.5, 23.9, 72.1, 113.0, 119.9, 121.9, 124.0, 125.7, 126.5, 127.0, 127.1, 129.0, 129.4, 130.4, 136.5, 138.6, 141.9, 149.1, 155.9, 159.6. HRMS (ESI) calcd for $C_{20}H_{19}NNaO$ $(M+Na)^+$ 312.1359, found 312.1358.



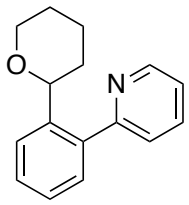
3am

Compound 3am (CAS: 864286-94-2, Table 2, entry 13: 34.2 mg, 88% yield). After the reaction was carried out at 80 ºC for 48 h, the mixture was passed through a short alumina column with EtOAc. The solvent was removed on a rotary evaporator, and the residue was treated with K_2CO_3 (41.5 mg, 0.30 mmol) in methanol (1.6 mL) at room temperature for 2 h. The solvent was removed and the residue was passed through a short silica gel column with EtOAc. The solvent was removed in vacuo and the residue was subjected to preparative TLC on silica gel eluted with a solution of EtOAc and hexane (1:2) to give **3am**. ^{1}H NMR ($CDCl_3$) δ 1.49 (d, J = 6.7 Hz, 3H), 4.74 (q, J = 6.7 Hz, 1H), 6.38 (s, 1H), 7.30 (ddd, J = 7.8, 4.8, 1.1 Hz, 1H), 7.35–7.38 (m, 1H), 7.42–7.45 (m, 2H), 7.56 (d, J = 7.8 Hz, 1H), 7.60 (dd, J = 7.8, 1.1 Hz, 1H), 7.83 (td, J = 7.8, 1.8 Hz, 1H), 8.62 (ddd, J = 4.8, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 20.4, 66.7, 122.1, 124.2, 126.4, 127.5, 129.1, 130.6, 137.5, 139.5, 143.4, 147.8, 159.8. HRMS (ESI) calcd for $C_{13}H_{13}NNaO$ $(M+Na)^+$ 222.0889, found 222.0885.



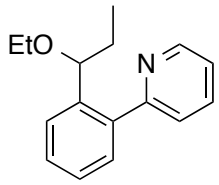
3an

Compound 3an (Table 2, entry 14: 43.0 mg, 95% yield). A solution of EtOAc and hexane (1:3) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 1.71–1.78 (m, 1H), 1.84–1.92 (m, 1H), 1.94–2.01 (m, 1H), 2.15–2.22 (m, 1H), 3.83 (dt, J = 14.4, 7.1 Hz, 1H), 4.09 (dt, J = 14.4, 7.3 Hz, 1H), 5.08 (t, J = 7.3 Hz, 1H), 7.25 (ddd, J = 7.7, 4.9, 1.0 Hz, 1H), 7.31–7.36 (m, 2H), 7.40–7.44 (m, 2H), 7.64 (dd, J = 7.7, 1.0 Hz, 1H), 7.74 (td, J = 7.7, 1.7Hz, 1H), 8.67 (ddd, J = 4.9, 1.7, 0.8 Hz, 1H);^{13}C NMR ($CDCl_3$) δ 26.3, 34.9, 68.7, 77.9, 121.7, 124.2, 125.8, 126.9, 128.6, 129.4, 136.2, 139.1, 141.7, 149.0, 159.7. HRMS (ESI) calcd for $C_{15}H_{15}NNaO$ $(M+Na)^+$ 248.1046, found 248.1050.



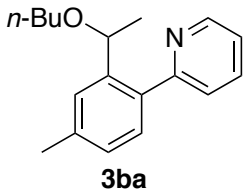
3ao

Compound 3ao (Table 2, entry 15: 32.5 mg, 68% yield). A solution of EtOAc and hexane (1:3) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 1.42–1.52 (m, 2H), 1.59–1.72 (m, 2H), 1.82–1.88 (m, 2H), 3.43 (ddd, *J* = 12.4, 11.5, 2.2 Hz, 1H), 4.01–4.07 (m, 1H), 4.51 (dd, *J* = 11.1, 1.7 Hz, 1H), 7.26 (ddd, *J* = 7.7, 4.9, 1.1 Hz, 1H), 7.32–7.37 (m, 2H), 7.43 (td, *J* = 7.5, 1.7 Hz, 1H), 7.45 (dt, *J* = 6.6, 1.0 Hz, 1H), 7.66 (d, *J* = 7.7 Hz, 1H), 7.75 (td, *J* = 7.7, 1.9 Hz, 1H), 8.69 (ddd, *J* = 4.9, 1.7, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 24.0, 25.9, 33.4, 68.8, 76.9, 121.7, 124.3, 126.5, 127.2, 128.6, 129.5, 136.1, 139.1, 141.0, 149.0, 159.7. HRMS (ESI) calcd for $C_{16}H_{17}NNaO$ $(M+Na)^+$ 262.1202, found 262.1210.



3ap

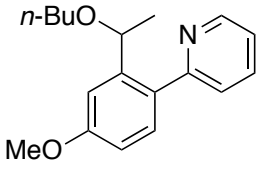
Compound 3ap (Table 2, entry 16: 9.4 mg, 19% yield). A mixture of (*E*) and (*Z*)-isomers (1:2) of **2p** was used. ^{1}H NMR ($CDCl_3$) δ 0.82 (t, *J* = 7.5 Hz, 3H), 1.11 (t, *J* = 7.5 Hz, 3H), 1.68 (quint, *J* = 7.5 Hz, 2H), 3.23 (dq, *J* = 9.5, 7.5 Hz, 1H), 3.37 (dq, *J* = 9.5, 7.5 Hz, 1H), 4.45 (t, *J* = 7.5 Hz, 1H), 7.24–7.29 (m, 1H), 7.30–7.37 (m, 3H), 7.42–7.46 (m, 1H), 7.61 (d, *J* = 8.1 Hz, 1H), 7.75 (td, *J* = 7.6, 1.9 Hz, 1H), 8.68 (d, *J* = 4.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 10.5, 15.3, 31.1, 63.9, 78.8, 121.7, 124.2, 126.3, 126.8, 128.6, 129.2, 136.1, 140.2, 141.4, 149.0, 159.8. HRMS (ESI) calcd for $C_{16}H_{19}NNaO$ $(M+Na)^+$ 264.1359, found 264.1363.



3ba

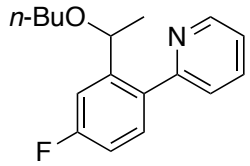
Compound 3ba (Table 3, entry 1: 52.2 mg, 97% yield). A solution of EtOAc and $CHCl_3$ (1:10) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.84 (t, *J* = 7.4 Hz, 3H), 1.23–1.34 (m, 2H), 1.40 (d, *J* = 6.5 Hz, 3H), 1.38–1.51 (m, 2H), 2.42 (s, 3H), 3.15 (dt, *J* = 7.8, 6.5 Hz, 1H), 3.23 (dt, *J* = 7.8, 6.8 Hz, 1H), 4.66 (q, *J* = 6.5 Hz, 1H), 7.14 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.23 (d, *J* = 7.8 Hz, 1H), 7.24 (ddd, *J* = 7.8, 4.9, 1.1 Hz, 1H), 7.34 (dd, *J* = 7.8, 1.1 Hz, 1H),

7.44 (s, 1H), 7.73 (td, J = 7.8, 1.7 Hz, 1H), 8.67 (ddd, J = 4.9, 1.7, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.8, 19.3, 21.4, 23.8, 32.0, 68.1, 73.4, 121.5, 124.2, 126.5, 127.7, 129.3, 136.0, 137.0, 138.5, 142.2, 149.1, 159.7. HRMS (ESI) calcd for $C_{18}H_{23}NNaO$ $(M+Na)^+$ 292.1672, found 292.1673.



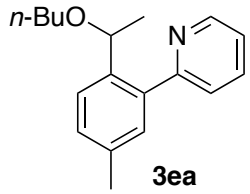
3ca

Compound 3ca (Table 3, entry 2: 53.0 mg, 93% yield). A solution of EtOAc and hexane (1:3) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.85 (t, J = 7.4 Hz, 3H), 1.24–1.36 (m, 2H), 1.40 (d, J = 6.5 Hz, 3H), 1.40–1.52 (m, 2H), 3.17 (dt, J = 7.8, 6.5 Hz, 1H), 3.26 (dt, J = 7.8, 6.8 Hz, 1H), 3.87 (s, 3H), 4.71 (q, J = 6.5 Hz, 1H), 6.87 (dd, J = 8.5, 2.7 Hz, 1H), 7.19 (d, J = 2.7 Hz, 1H), 7.23 (ddd, J = 7.5, 4.9, 1.2 Hz, 1H), 7.28 (d, J = 8.5, 1H), 7.33 (dd, J = 7.8, 1.2 Hz, 1H), 7.72 (td, J = 7.8, 1.9 Hz, 1H), 8.66 (ddd, J = 4.8, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.4, 23.9, 32.0, 55.3, 68.3, 73.6, 110.6, 112.9, 121.4, 124.1, 130.8, 132.5, 136.1, 144.3, 149.1, 159.4, 160.1. HRMS (ESI) calcd for $C_{18}H_{23}NNaO_2$ $(M+Na)^+$ 308.1621, found 308.1625.



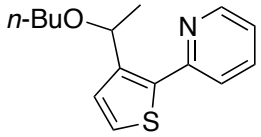
3da

Compound 3da (Table 3, entry 3: 50.5 mg, 92% yield). A solution of EtOAc and $CHCl_3$ (1:30) was used as an eluent for preparative TLC on silica gel twice. ^{1}H NMR ($CDCl_3$) δ 0.85 (t, J = 7.4 Hz, 3H), 1.24–1.36 (m, 2H), 1.37 (d, J = 6.4 Hz, 3H), 1.41–1.52 (m, 2H), 3.17 (dt, J = 7.7, 6.6 Hz, 1H), 3.23 (dt, J = 7.7, 6.8 Hz, 1H), 4.65 (qd, J = 6.4, 1.8 Hz, 1H), 7.01 (td, J = 8.5, 2.9 Hz, 1H), 7.27 (ddd, J = 7.7, 4.9, 1.2 Hz, 1H), 7.30 (dd, J = 8.5, 5.8, 1H), 7.32–7.35 (m, 2H), 7.75 (td, J = 7.7, 1.9 Hz, 1H), 8.67 (ddd, J = 4.9, 1.9, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.3, 23.7, 32.0, 68.4, 73.4, 112.7 (d, $J_{F\text{-}C}$ = 22 Hz), 113.9 (d, $J_{F\text{-}C}$ = 22 Hz), 121.9, 124.1, 131.2 (d, $J_{F\text{-}C}$ = 7 Hz), 135.7, 136.3, 145.7, 149.2, 158.8, 163.3 (d, $J_{F\text{-}C}$ = 247 Hz). HRMS (ESI) calcd for $C_{17}H_{20}FNNaO$ $(M+Na)^+$ 296.1421, found 296.1418.



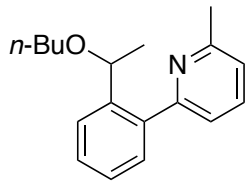
3ea

Compound 3ea (Table 3, entry 4: 47.9 mg, 89% yield). ^{1}H NMR ($CDCl_3$) δ 0.83 (t, *J* = 7.4 Hz, 3H), 1.22–1.34 (m, 2H), 1.38–1.50 (m, 2H), 1.39 (d, *J* = 6.4 Hz, 3H), 2.38 (s, 3H), 3.13 (dt, *J* = 7.7, 6.5 Hz, 1H), 3.21 (dt, *J* = 7.7, 6.8 Hz, 1H), 4.59 (q, *J* = 6.4 Hz, 1H), 7.14–7.16 (m, 1H), 7.24–7.27 (m, 2H), 7.35 (dd, *J* = 6.4, 1.0, 1H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.73 (td, *J* = 7.7, 1.8 Hz, 1H), 8.68 (ddd, *J* = 4.9, 1.8, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.3, 21.0, 23.9, 32.0, 68.1, 73.3, 121.7, 124.1, 126.0, 129.6, 129.9, 136.0, 136.5, 139.3, 139.7, 149.2, 159.7. HRMS (ESI) calcd for $C_{18}H_{23}NNaO$ $(M+Na)^+$ 292.1672, found 292.1664.



3fa

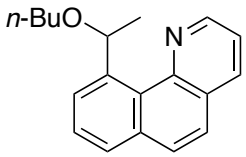
Compound 3fa (Table 3, entry 5: 37.3 mg, 72% yield). A solution of EtOAc/Hexane (1:6) and EtOAc/$CHCl_3$ (1:20) was used as eluents for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.85 (t, *J* = 7.4 Hz, 3H), 1.24–1.37 (m, 2H), 1.42–1.53 (m, 2H), 1.53 (d, *J* = 6.5 Hz, 3H), 3.24 (dt, *J* = 7.7, 6.5 Hz, 1H), 3.27 (dt, *J* = 7.7, 6.8 Hz, 1H), 5.08 (q, *J* = 6.5 Hz, 1H), 7.17 (ddd, *J* = 7.8, 4.9, 1.0 Hz, 1H), 7.21 (d, *J* = 5.3 Hz, 1H), 7.32 (d, *J* = 5.3, 1H), 7.50 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.70 (td, *J* = 7.8, 1.7 Hz, 1H), 8.64 (ddd, *J* = 4.9, 1.7, 0.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.4, 22.7, 32.0, 68.3, 71.8, 121.6, 122.6, 126.0, 127.8, 136.4, 138.7, 143.7, 149.6, 153.1. HRMS (ESI) calcd for $C_{15}H_{19}NNaOS$ $(M+Na)^+$ 284.1080, found 284.1077.



3ga

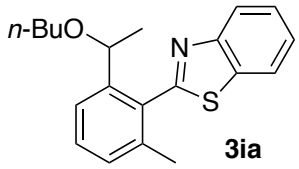
Compound 3ga (Table 3, entry 6: 51.7 mg, 96% yield). ^{1}H NMR ($CDCl_3$) δ 0.84 (t, *J* = 7.4 Hz, 3H), 1.23–1.36 (m, 2H), 1.42 (d, *J* = 6.5 Hz, 3H), 1.40–1.51 (m, 2H), 2.60 (s, 3H), 3.16 (dt, *J* = 7.8, 6.5 Hz, 1H), 3.23 (dt, *J* = 7.8, 6.8 Hz, 1H), 4.63 (q, *J* = 6.5 Hz, 1H), 7.12 (d, *J* = 7.7 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 1H), 7.29–7.33 (m, 2H), 7.40–7.44 (m, 1H), 7.626 (d, *J* = 7.7 Hz, 1H), 7.633 (t, *J* = 7.7 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.3, 24.0, 24.6, 32.0, 68.2, 73.6, 121.0, 121.2,

126.0, 126.9, 128.6, 129.2, 136.4, 140.0, 142.5, 157.7, 159.0. HRMS (ESI) calcd for $C_{18}H_{23}NNaO$ $(M+Na)^+$ 292.1672, found 292.1677.



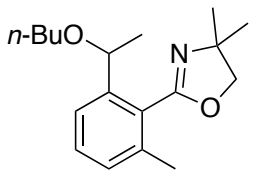
3ha

Compound 3ha (Table 3, entry 7: 63.0 mg, 90% yield). A solution of EtOAc and hexane (1:6) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.91 (t, *J* = 7.4 Hz, 3H), 1.32–1.47 (m, 2H), 1.57–1.67 (m, 2H), 1.67 (d, *J* = 6.1 Hz, 3H), 3.38 (t, *J* = 6.6 Hz, 2H), 6.88 (q, *J* = 6.1 Hz, 1H), 7.45 (dd, *J* = 8.0, 4.2 Hz, 1H), 7.65 (d, *J* = 8.7 Hz, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.82–7.85 (m, 2H), 8.15–8.19 (m, 2H), 9.01 (dd, *J* = 4.2, 1.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 14.0, 19.5, 24.3, 32.4, 68.8, 76.1, 120.7, 125.2, 125.3, 127.5, 127.6, 127.9, 128.8, 129.0, 135.0, 135.5, 146.0, 147.1, 148.1. HRMS (ESI) calcd for $C_{19}H_{21}NNaO$ $(M+Na)^+$ 302.1515, found 302.1506.



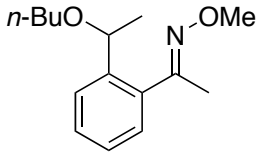
3ia

Compound 3ia (Table 4, entry 1: 51.9 mg, 80% yield). A solution of EtOAc/Hexane (1:7) and EtOAc/$CHCl_3$ (1:20) was used as eluents for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.85 (t, *J* = 8.4 Hz, 3H), 1.25–1.37 (m, 2H), 1.37 (d, *J* = 6.4 Hz, 3H), 1.42–1.53 (m, 2H), 2.19 (s, 3H), 3.19 (dt, *J* = 7.7, 6.6 Hz, 1H), 3.28 (dt, *J* = 7.7, 6.8 Hz, 1H), 4.27 (q, *J* = 6.4 Hz, 1H), 7.21 (d, *J* = 7.7 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.45–7.49 (m, 2H), 7.55 (t, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 8.13 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.3, 20.0, 24.4, 32.0, 68.4, 74.2, 121.5, 123.2, 123.5, 125.3, 126.1, 128.8, 130.2, 132.1, 136.3, 136.9, 144.2, 153.4, 166.4. HRMS (ESI) calcd for $C_{20}H_{23}NNaOS$ $(M+Na)^+$ 348.1393, found 348.1389.



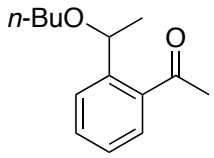
3ja

Compound 3ja (Table 4, entry 2: 54.5 mg, 94% yield). A mixture of EtOAc and hexane (1:4) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.86 (t, *J* = 7.4 Hz, 3H), 1.26–1.39 (m, 2H), 1.417 (d, *J* = 6.5 Hz, 3H), 1.422 (s, 6H), 1.44–1.56 (m, 2H), 2.33 (s, 3H), 3.22 (dt, *J* = 7.9, 6.6 Hz, 1H), 3.26 (dt, *J* = 7.9, 6.9 Hz, 1H), 4.10 (s, 2H), 4.51 (q, *J* = 6.5 Hz, 1H), 7.10 (d, *J* = 7.3 Hz, 1H), 7.31 (t, *J* = 7.3 Hz, 1H), 7.34 (d, *J* = 7.3 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.29, 19.32, 24.3, 28.37, 28.45, 32.0, 68.1, 68.5, 74.7, 78.9, 122.8, 127.7, 128.6, 129.8, 136.4, 143.6, 161.5. HRMS (ESI) calcd for $C_{18}H_{27}NNaO_2$ $(M+Na)^+$ 312.1934, found 312.1933.



3ka

Compound 3ka (Table 4, entry 3: 30.3 mg, 61% yield). $CHCl_3$ was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 0.87 (t, *J* = 7.3 Hz, 3H), 1.27-1.40 (m, 2H), 1.44 (d, *J* = 6.5 Hz, 3H), 1.46–1.56 (m, 2H), 2.18 (s, 3H), 3.22 (dt, *J* = 7.7, 6.5 Hz, 1H), 3.24 (dt, *J* = 7.7, 6.3 Hz, 1H), 3.96 (s, 3H), 4.67 (q, *J* = 6.5 Hz, 1H), 7.19 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.27 (td, *J* = 7.8, 1.0 Hz, 1H), 7.37 (td, *J* = 7.8, 1.0 Hz, 1H), 7.56 (dd, *J* = 7.8, 1.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 16.9, 19.4, 24.2, 32.0, 61.8, 68.4, 74.0, 126.2, 127.0, 127.8, 128.9, 136.3, 142.6, 155.9. HRMS (ESI) calcd for $C_{15}H_{23}NNaO_2$ $(M+Na)^+$ 272.1621, found 272.1616.



3la

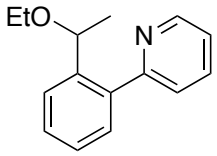
Compound 3la (Table 4, entry 4: 37.1 mg, 84% yield). After the reaction was carried out at 80 ºC for 48 h, the mixture was passed through a short alumina column with EtOAc. The solvent was removed on a rotary evaporator. The residue was treated with HCl aq. (1.0 mL, 1.0 M) in THF (0.8 mL) at room temperature for 2 h. Brine was added to the reaction mixture, and the mixture was extracted with Et_2O. The solvent was removed in vacuo and the residue was subjected to preparative TLC on silica gel eluted with a mixture of EtOAc and hexane (1:7) to give **3la**. ^{1}H NMR ($CDCl_3$) δ 0.88 (t, *J* = 7.4 Hz, 3H), 1.28–1.41 (m, 2H), 1.43 (d, *J* = 6.4 Hz, 3H),

1.48–1.58 (m, 2H), 2.59 (s, 3H), 3.23 (dt, J = 7.6, 6.8 Hz, 1H), 3.28 (dt, J = 7.6, 6.6 Hz, 1H), 5.01 (q, J = 6.4 Hz, 1H), 7.32 (t, J = 7.5 Hz, 1H), 7.51 (t, J = 7.5 Hz, 1H), 7.65 (d, J = 7.5 Hz, 1H), 7.70 (d, J = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 19.4, 23.9, 29.9, 32.1, 68.7, 74.0, 126.6, 126.7, 128.6, 131.9, 137.1, 145.4, 201.9. HRMS (ESI) calcd for $C_{14}H_{20}NaO_2$ $(M+Na)^+$ 243.1356, found 243.1356.

8. General procedure for iridium-catalyzed asymmetric hydroarylation of vinyl ethers 2 with 2-phenyl pyridine (1a) (Scheme 1)

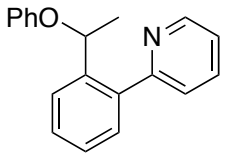
$NaBAr^F_4$ (18.4 mg, 0.020 mmol, 10 mol%) and $[IrCl((S,S)\text{-Fc-tfb*})]_2$ (8.2 mg, 0.010 mmol of Ir, 5 mol % of Ir) were placed in a Schlenk tube under N_2. Chlorobenzene (0.8 mL), 2-phenyl pyridine (**1a**) (31.0 mg, 0.20 mmol), and vinyl ether **2** (0.30 mmol) were added to the tube successively, and the mixture was stirred at 80 ºC for 20 h. 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**.

9. Data for Scheme 1



3ac

Compound 3ac (Scheme 1: 37.5 mg, 82% yield). The ee was measured by HPLC (Chiralpak OD-H column, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 8.8 min (major), t_2 = 10.4 min (minor); $[\alpha]^{20}_D$ –34 (c 1.07, $CHCl_3$) for 77% ee.



3af

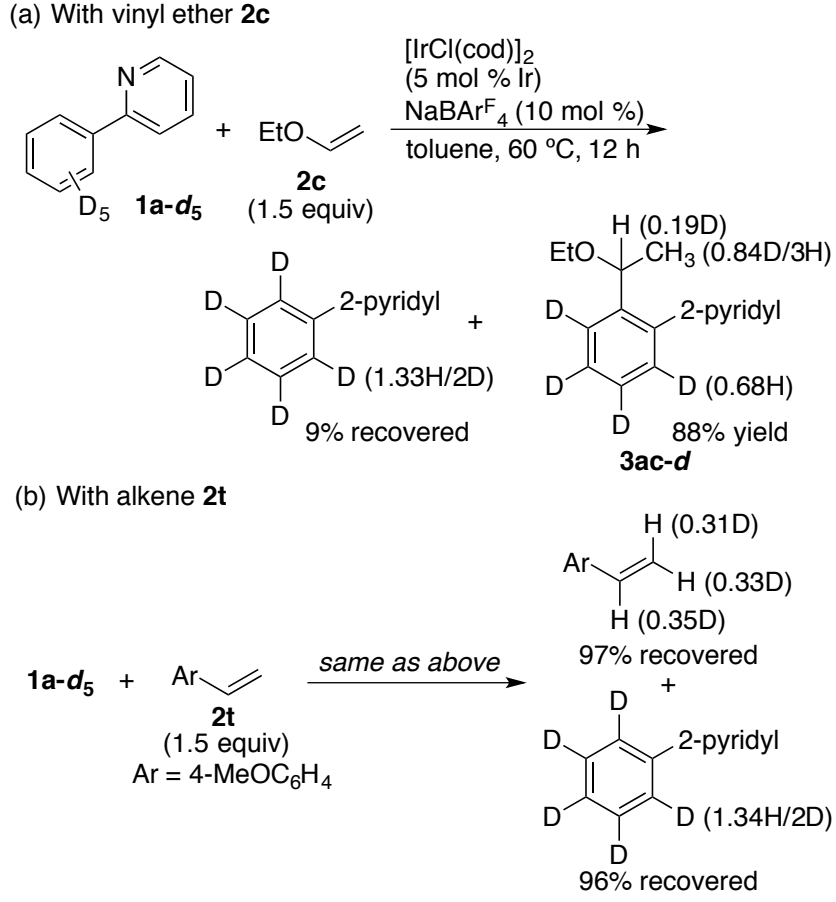
Compound 3af (Scheme 1: 51.5 mg, 92% yield). The ee was measured by HPLC (Chiralpak OD-H column, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 9.6 min (major), t_2 = 10.4 min (minor); $[\alpha]^{20}_D$ +159 (c 1.08, $CHCl_3$) for 76% ee.

10. Deuterium-labeling experiments (Scheme S1)

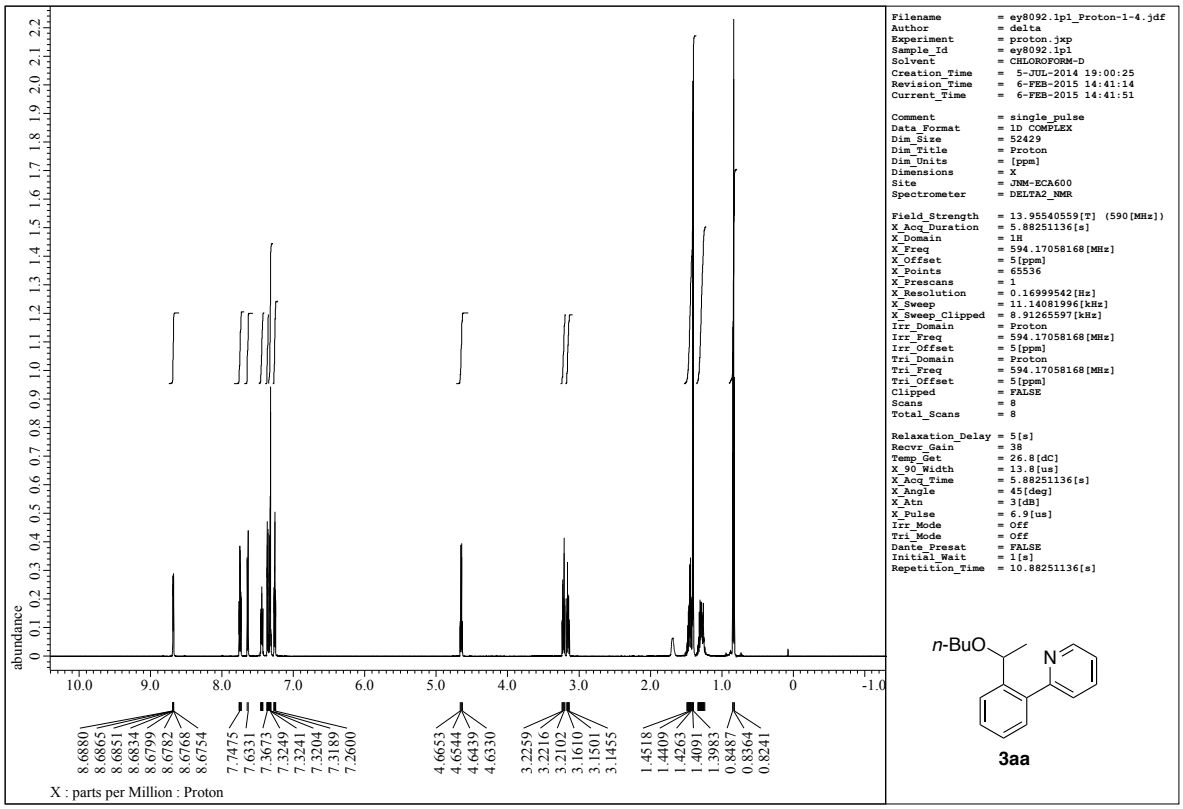
$NaBAr^F_4$ (9.2 mg, 0.010 mmol, 10 mol%) in a Schlenk tube was dried under vacuum at 80 ºC for 1 h. After the tube was cooled to room temperature, $[IrCl(cod)]_2$ (1.7 mg, 0.005 mmol of Ir, 5 mol % of Ir) was added and the tube was purged with nitrogen. Toluene (0.4 mL), **1a-d_5** (16.0

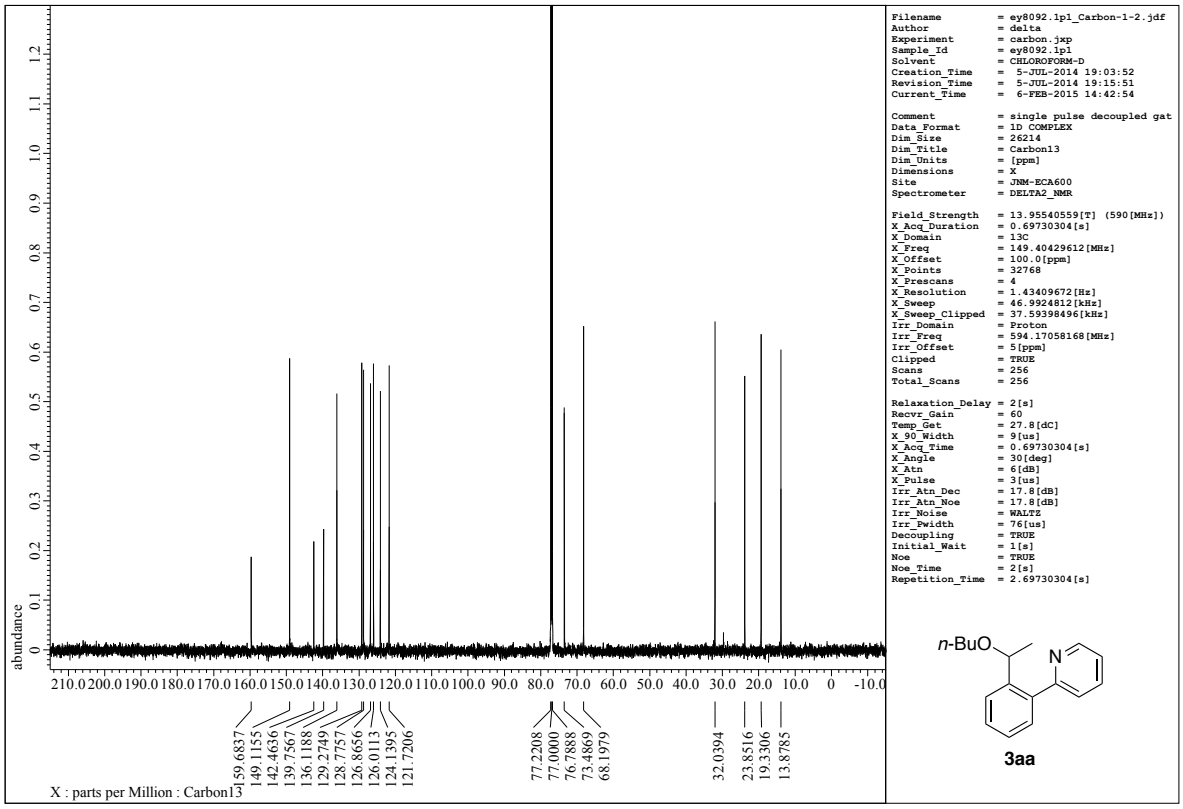
mg, 0.10 mmol), and vinyl ether **2c** or alkene **2t** (0.15 mmol) were added to the tube successively, and the mixture was stirred at 60 ºC for 12 h. The mixture was passed through a short column of alumina with EtOAc as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel (for (a)). The yields of the products were determined by ^{1}H NMR using nitromethane as an internal standard (for (b)). The results are shown in Scheme S1.

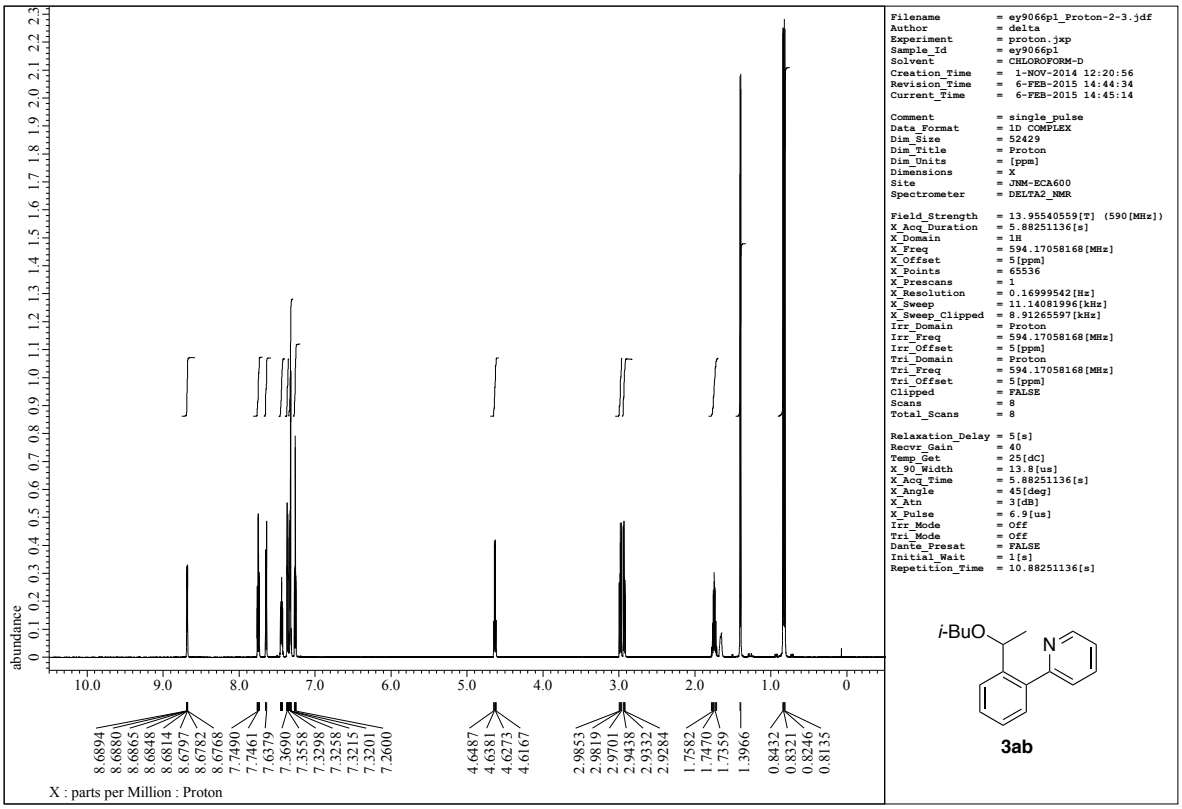
Scheme S1. Deuterium-Labeling Experiments



11. ^{1}H and ^{13}C NMR spectra



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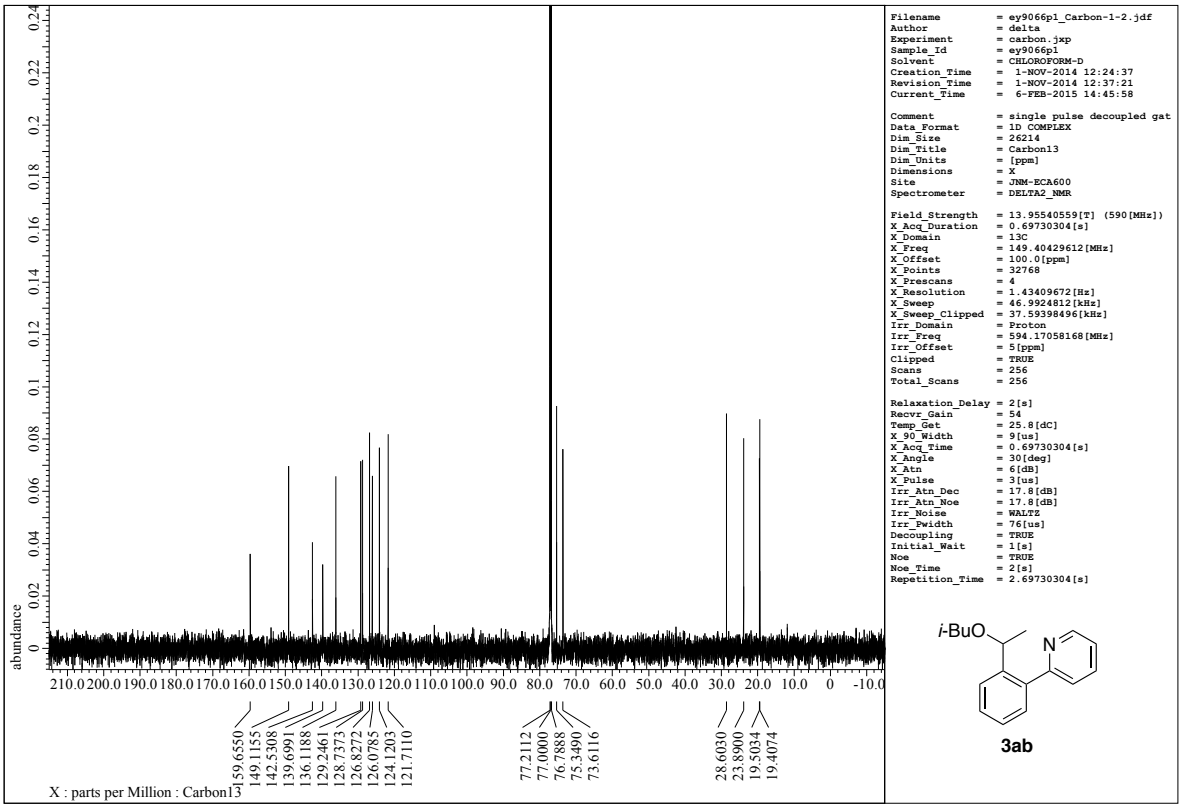
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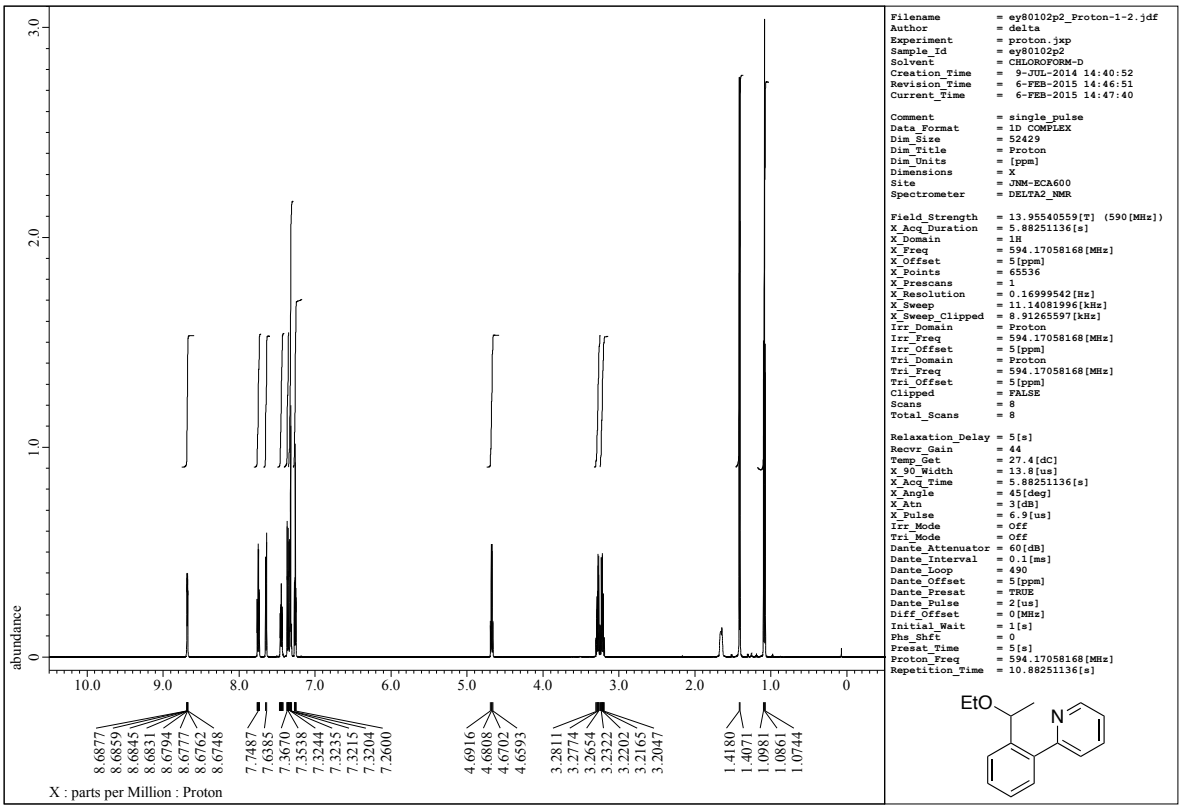
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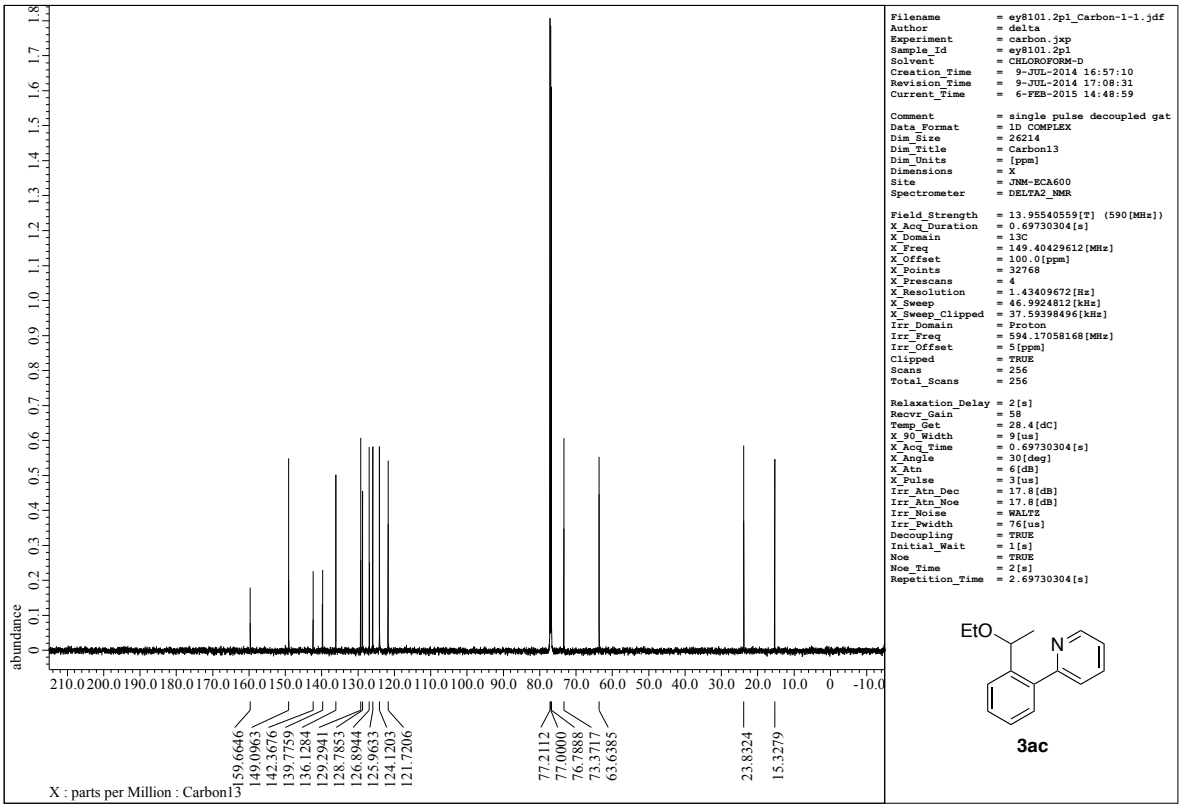
3ac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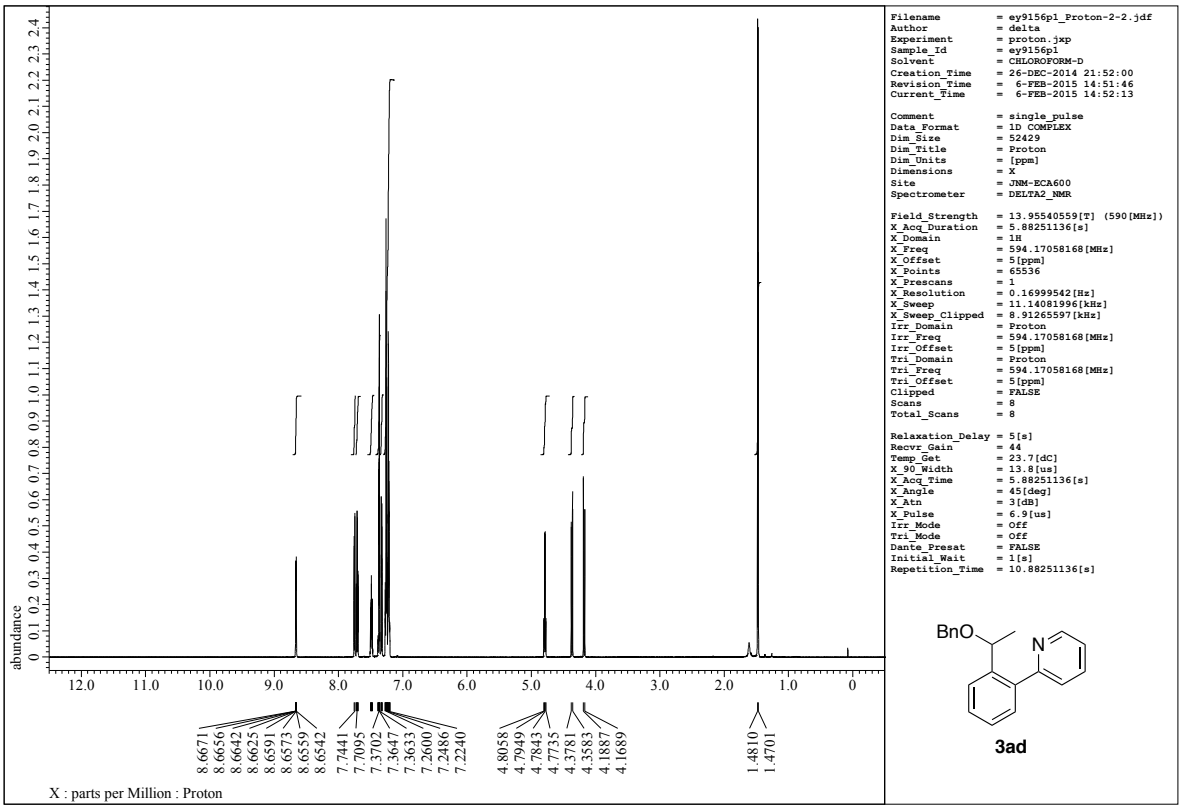
3ac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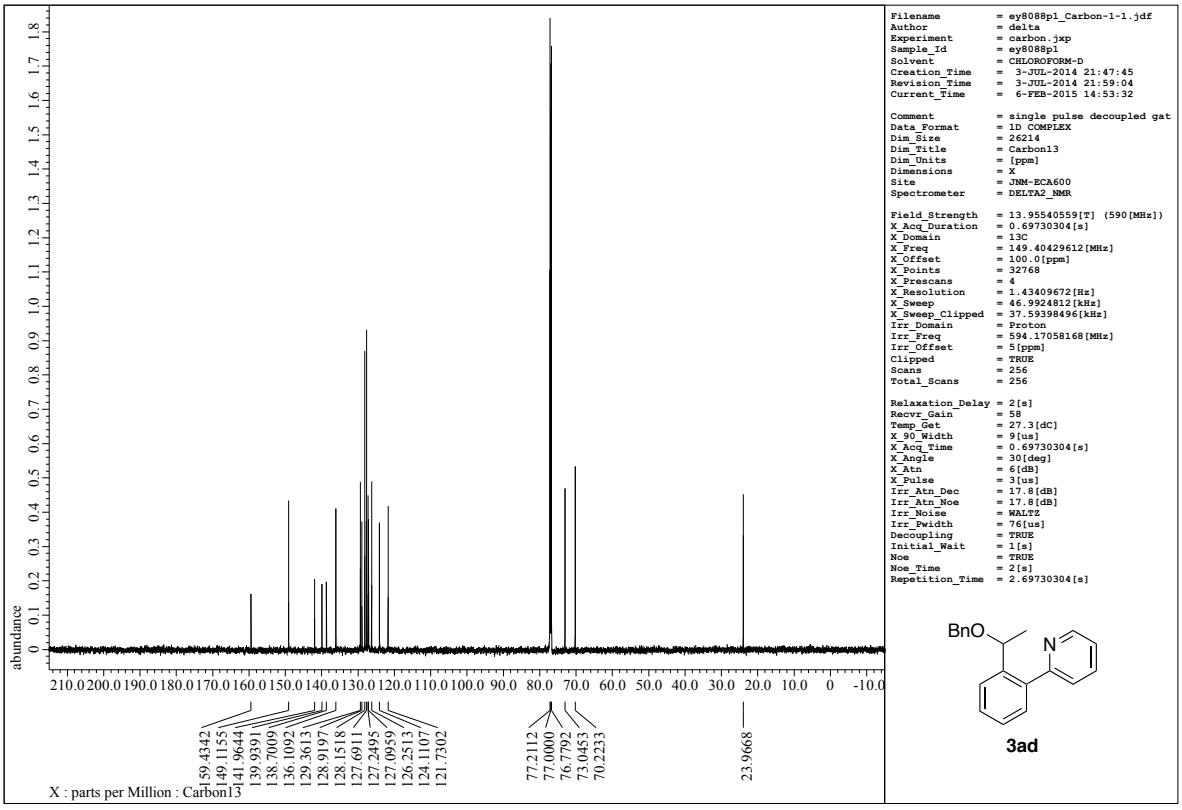
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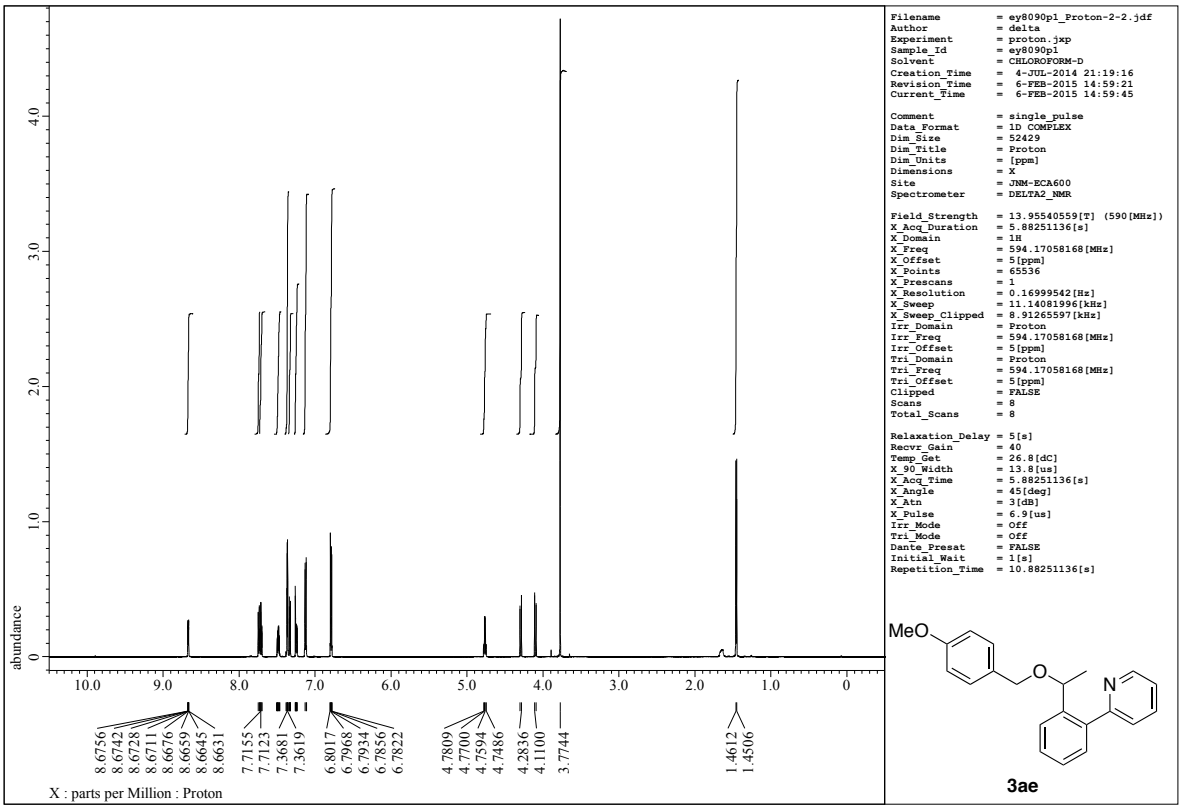
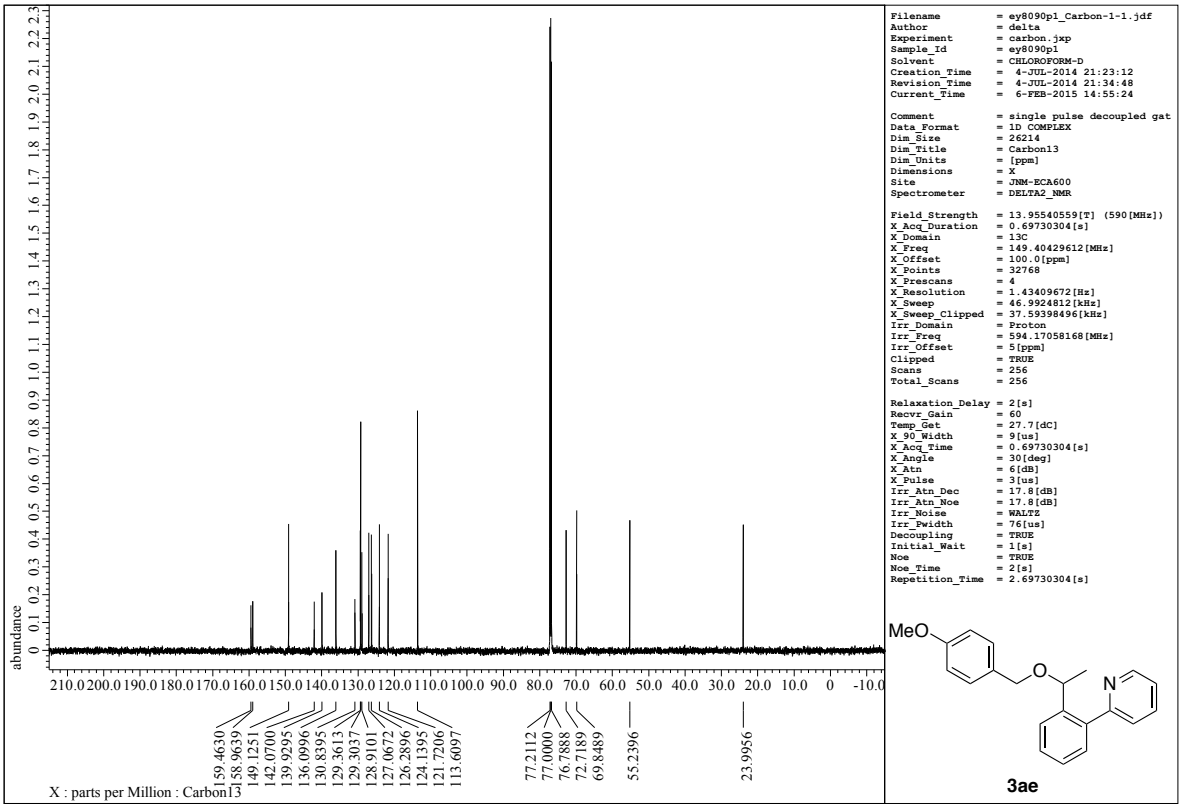
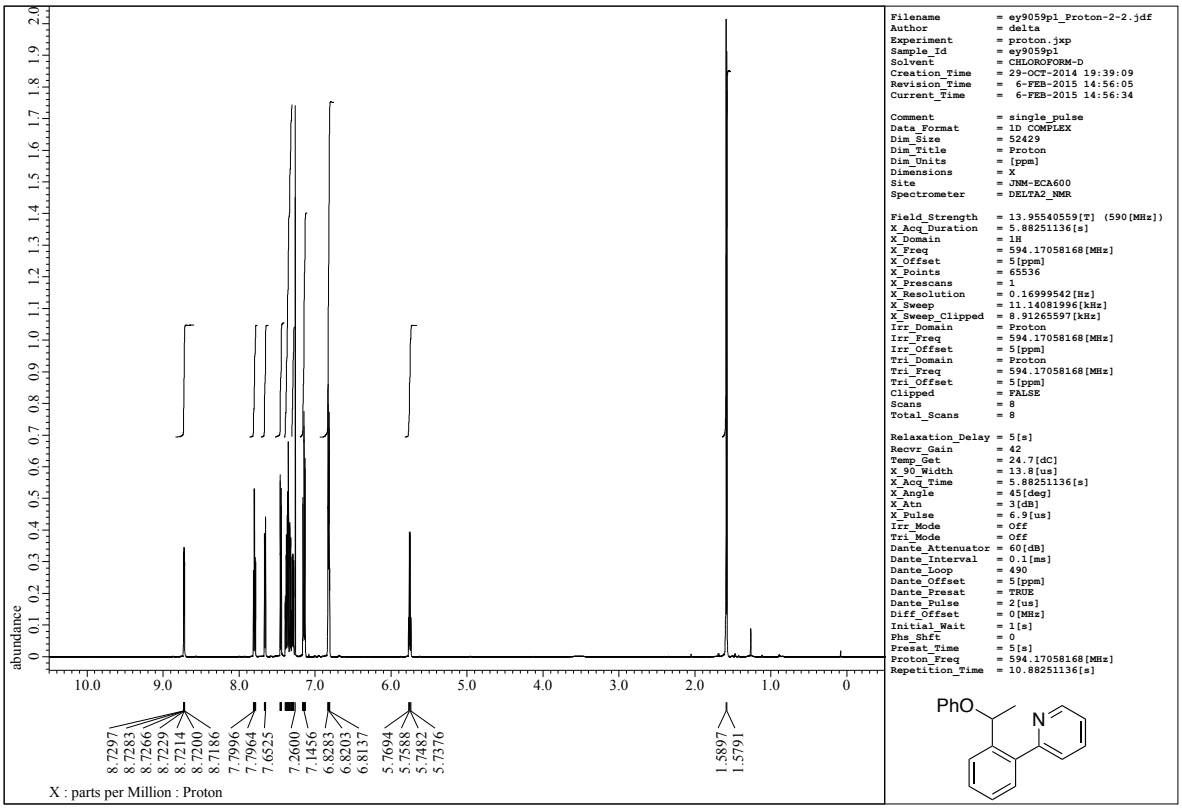
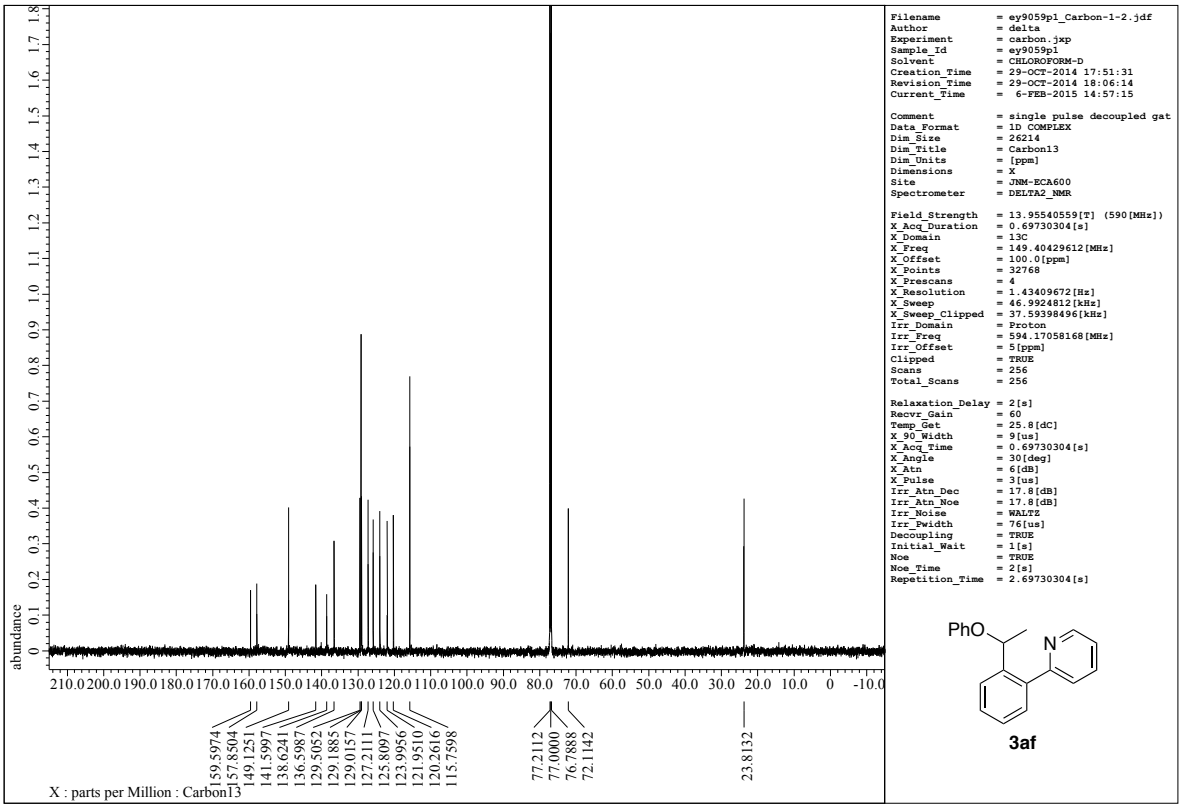
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 27.3[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
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]
BnO
N
3ad


abundance
X : parts per Million : Proton
8.6756
8.6742
8.6728
8.6711
8.6676
8.6659
8.6645
8.6631
7.7155
7.7123
7.3681
7.3619
6.8017
6.7968
6.7934
6.7856
6.7822
4.7809
4.7700
4.7594
4.7486
4.2836
4.1100
3.7744
1.4612
1.4506
Filename = ey8090p1_Proton-2-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey8090p1
Solvent = CHLOROFORM-D
Creation_Time = 4-JUL-2014 21:19:16
Revision_Time = 6-FEB-2015 14:59:21
Current_Time = 6-FEB-2015 14:59:45
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 = 5[s]
Recvr_Gain = 40
Temp_Get = 26.8[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 = 10.88251136[s]
MeO
O
N
3ae

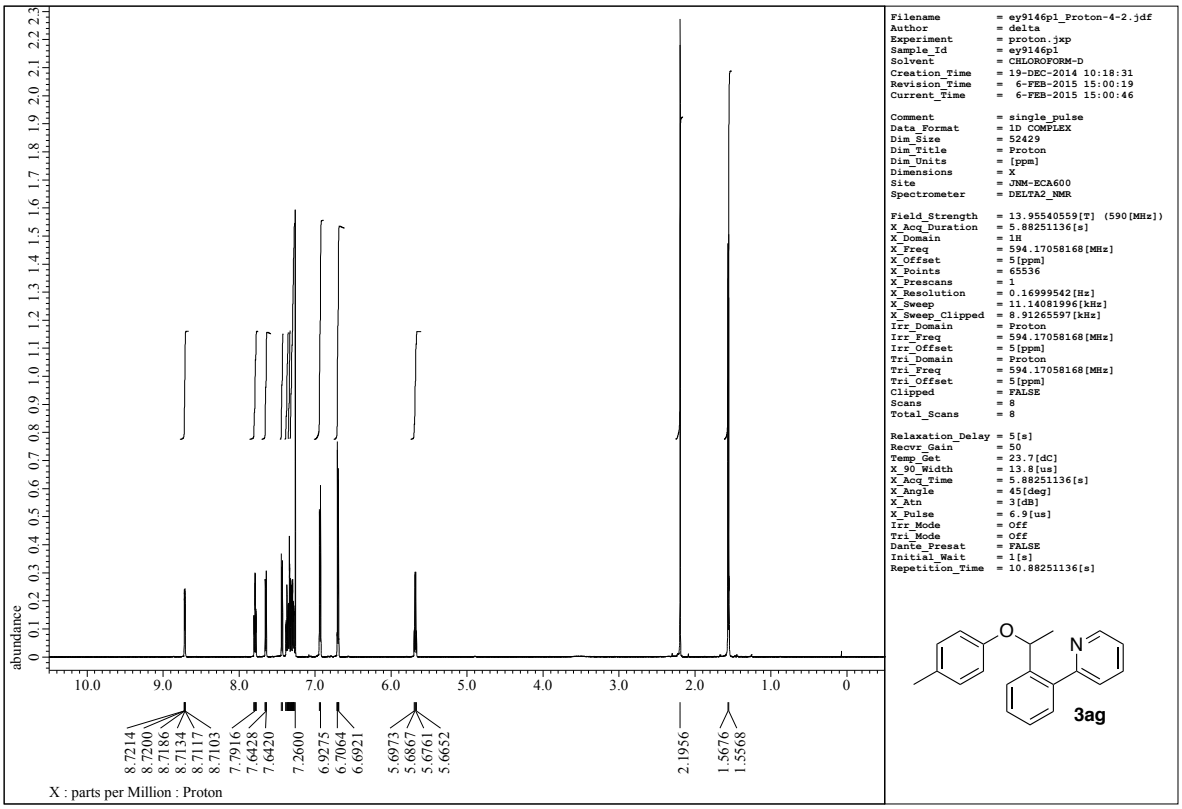

abundance
X : parts per Million : Carbon13
159.4630
158.9639
149.1251
142.0700
139.9295
136.0996
130.8395
129.3613
129.3037
128.9101
127.0672
126.2896
124.1395
121.7206
113.6097
77.2112
77.0000
76.7888
72.7189
69.8489
55.2396
23.9956
Filename = ey8090p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey8090p1
Solvent = CHLOROFORM-D
Creation_Time = 4-JUL-2014 21:23:12
Revision_Time = 4-JUL-2014 21:34:48
Current_Time = 6-FEB-2015 14:55:24
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 = 27.7[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]
MeO
O
N
3ae

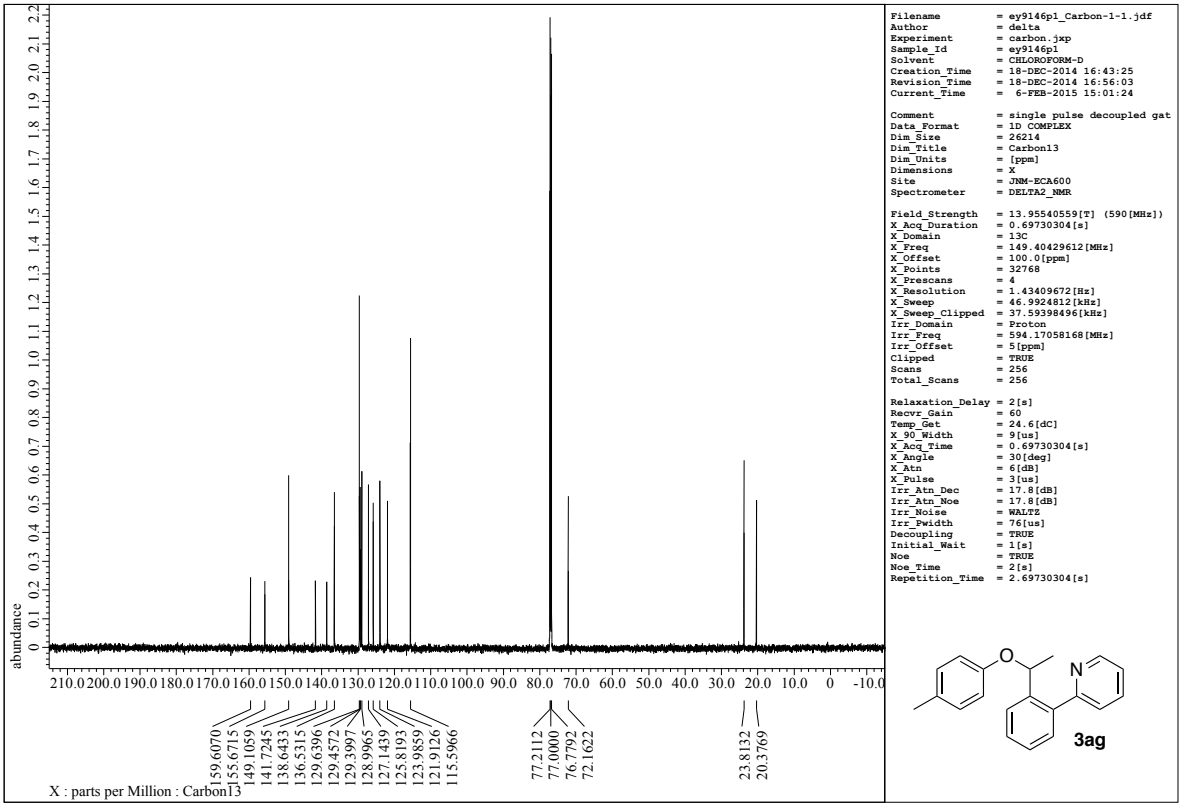

abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.7297 8.7283 8.7266 8.7229 8.7214 8.7200 8.7186 7.7996 7.7964 7.6525 7.2600 7.1456 6.8283 6.8203 6.8137 5.7694 5.7588 5.7482 5.7376 1.5897 1.5791
X : parts per Million : Proton
Filename = ey9059p1_Proton-2-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9059p1
Solvent = CHLOROFORM-D
Creation_Time = 29-OCT-2014 19:39:09
Revision_Time = 6-FEB-2015 14:56:05
Current_Time = 6-FEB-2015 14:56:34
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 = 5[s]
Recvr_Gain = 42
Temp_Get = 24.7[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_Attenuator = 60[dB]
Dante_Interval = 0.1[ms]
Dante_Loop = 490
Dante_Offset = 5[ppm]
Dante_Presat = TRUE
Dante_Pulse = 2[us]
Diff_Offset = 0[MHz]
Initial_Wait = 1[s]
Phs_Shft = 0
Presat_Time = 5[s]
Proton_Freq = 594.17058168[MHz]
Repetition_Time = 10.88251136[s]
PhO
N

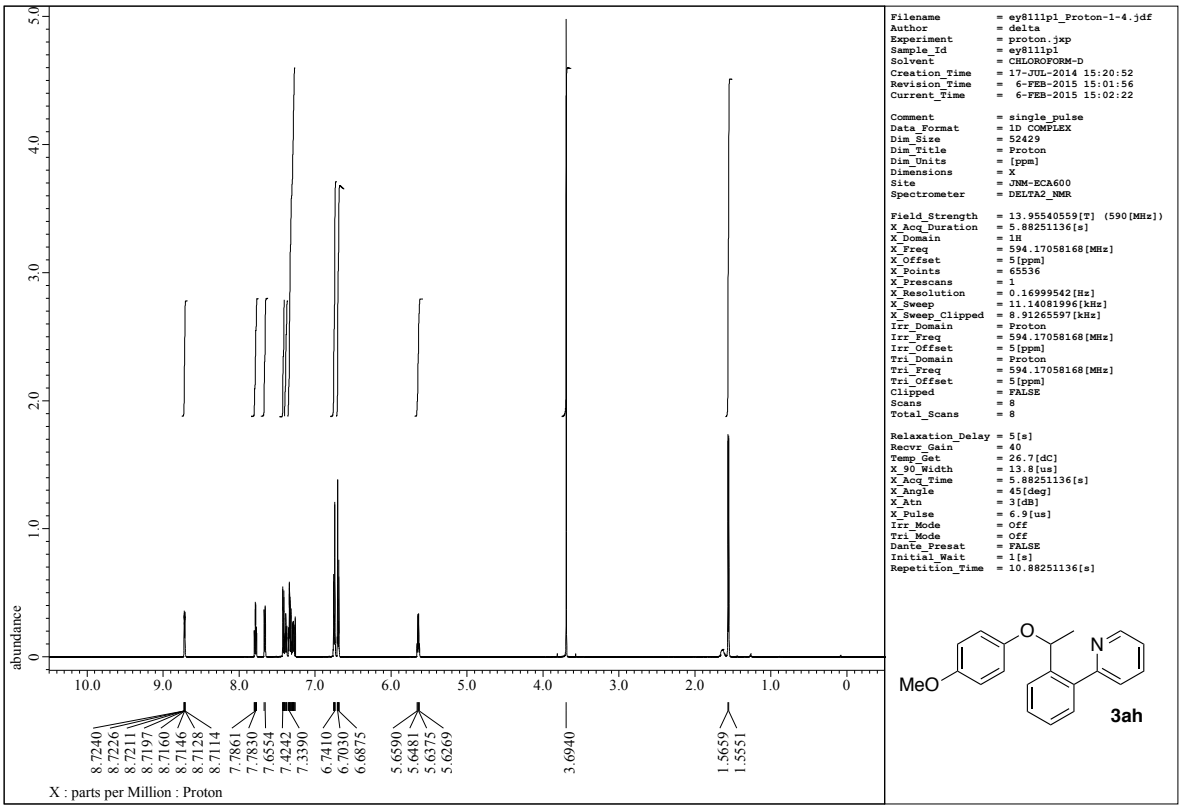
3af

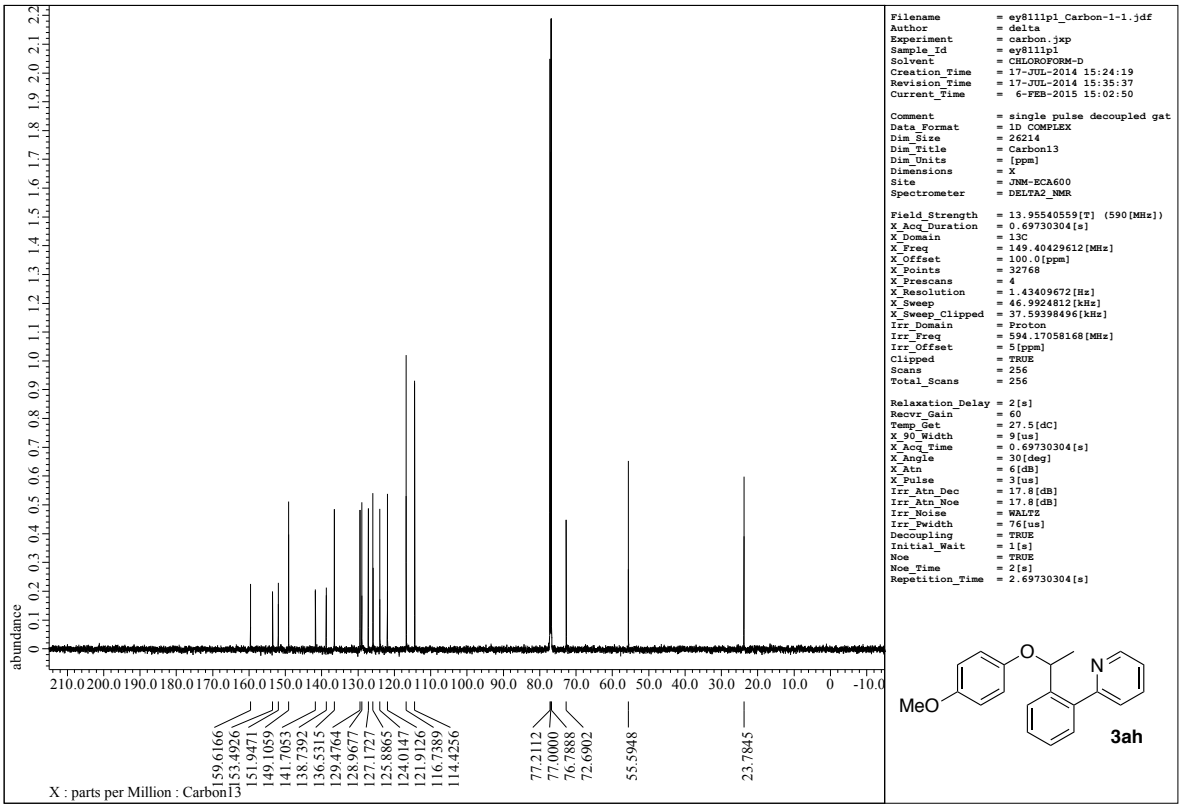

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
159.5974 157.8504 149.1251 141.5997 138.6241 136.5987 129.5052 129.1885 129.0157 127.2111 125.8097 123.9956 121.9510 120.2616 115.7598 77.2112 77.0000 76.7888 72.1142 23.8132
X : parts per Million : Carbon13
Filename = ey9059p1_Carbon-1-2.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9059p1
Solvent = CHLOROFORM-D
Creation_Time = 29-OCT-2014 17:51:31
Revision_Time = 29-OCT-2014 18:06:14
Current_Time = 6-FEB-2015 14:57:15
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 = 25.8[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]
PhO
N

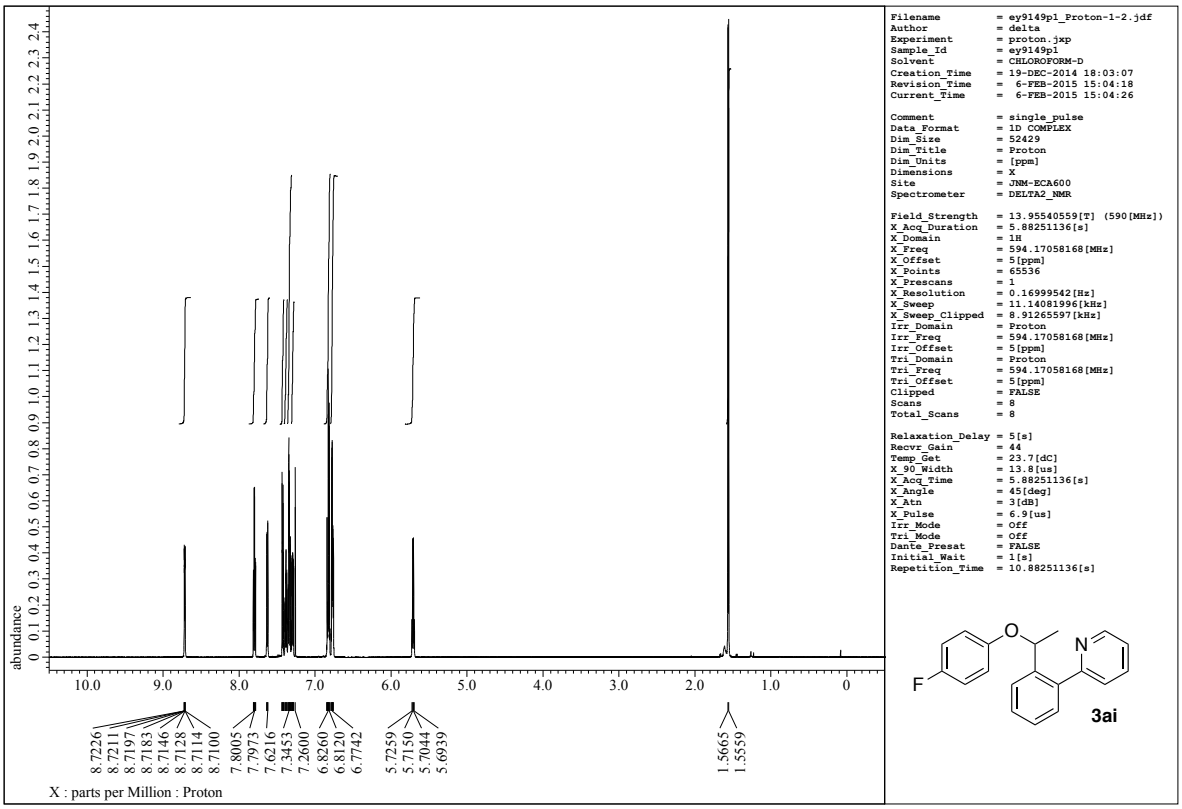
3af

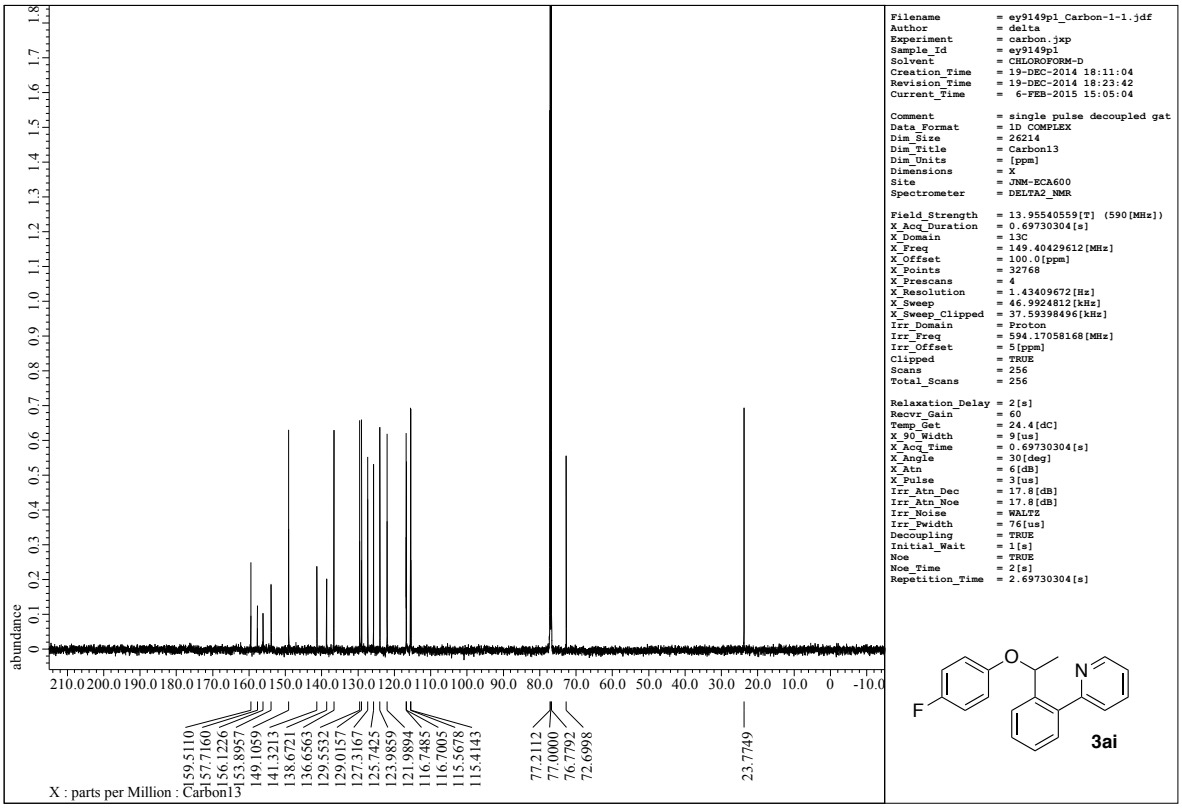

abundance
8.7214
8.7200
8.7186
8.7134
8.7117
8.7103
7.7916
7.6428
7.6420
7.2600
6.9275
6.7064
6.6921
5.6973
5.6867
5.6761
5.6652
2.1956
1.5676
1.5568
X : parts per Million : Proton
Filename = ey9146p1_Proton-4-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9146p1
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2014 10:18:31
Revision_Time = 6-FEB-2015 15:00:19
Current_Time = 6-FEB-2015 15:00:46
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 = 5[s]
Recvr_Gain = 50
Temp_Get = 23.7[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 = 10.88251136[s]
O
N
3ag

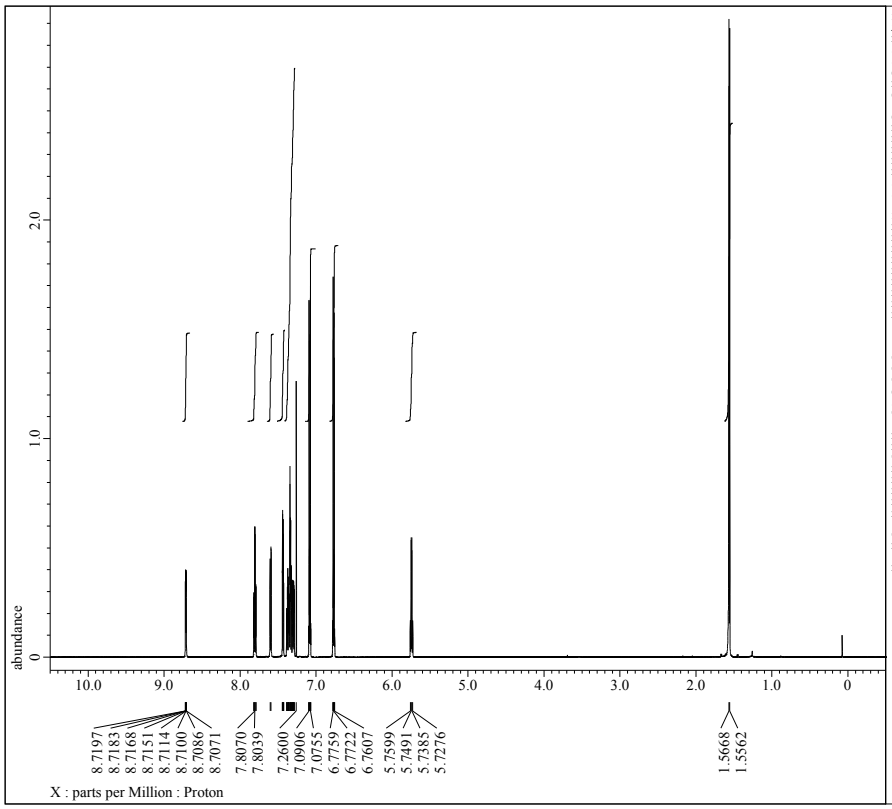

abundance
159.6070
155.6715
149.1059
141.7245
138.6433
136.5315
129.6396
129.4572
129.3997
128.9965
127.1439
125.8193
123.9859
121.9126
115.5966
77.2112
77.0000
76.7792
72.1622
23.8132
20.3769
X : parts per Million : Carbon13
Filename = ey9146p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9146p1
Solvent = CHLOROFORM-D
Creation_Time = 18-DEC-2014 16:43:25
Revision_Time = 18-DEC-2014 16:56:03
Current_Time = 6-FEB-2015 15:01:24
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 = 24.6[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]
O
N
3ag


abundance
8.7240
8.7226
8.7211
8.7197
8.7160
8.7146
8.7128
8.7114
7.7861
7.7830
7.6554
7.4242
7.3390
6.7410
6.7030
6.6875
5.6590
5.6481
5.6375
5.6269
3.6940
1.5659
1.5551
X : parts per Million : Proton
Filename = ey8111p1_Proton-1-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey8111p1
Solvent = CHLOROFORM-D
Creation_Time = 17-JUL-2014 15:20:52
Revision_Time = 6-FEB-2015 15:01:56
Current_Time = 6-FEB-2015 15:02:22
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 = 5[s]
Recvr_Gain = 40
Temp_Get = 26.7[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 = 10.88251136[s]
MeO
O
N
3ah

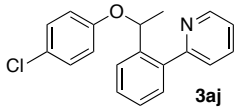

abundance
159.6166
153.4926
151.9471
149.1059
141.7053
138.7392
136.5315
129.4764
128.9677
127.1727
125.8865
124.0147
121.9126
116.7389
114.4256
77.2112
77.0000
76.7888
72.6902
55.5948
23.7845
X : parts per Million : Carbon13
Filename = ey8111p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey8111p1
Solvent = CHLOROFORM-D
Creation_Time = 17-JUL-2014 15:24:19
Revision_Time = 17-JUL-2014 15:35:37
Current_Time = 6-FEB-2015 15:02:50
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 = 100.0[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 = 27.5[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]
MeO
O
N
3ah


abundance
8.7226
8.7211
8.7197
8.7183
8.7146
8.7128
8.7114
8.7100
7.8005
7.7973
7.6216
7.3453
7.2600
6.8260
6.8120
6.7742
5.7259
5.7150
5.7044
5.6939
1.5665
1.5559
X : parts per Million : Proton
Filename = ey9149p1_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9149p1
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2014 18:03:07
Revision_Time = 6-FEB-2015 15:04:18
Current_Time = 6-FEB-2015 15:04:26
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 = 5[s]
Recvr_Gain = 44
Temp_Get = 23.7[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 = 10.88251136[s]
O
N
F
3ai

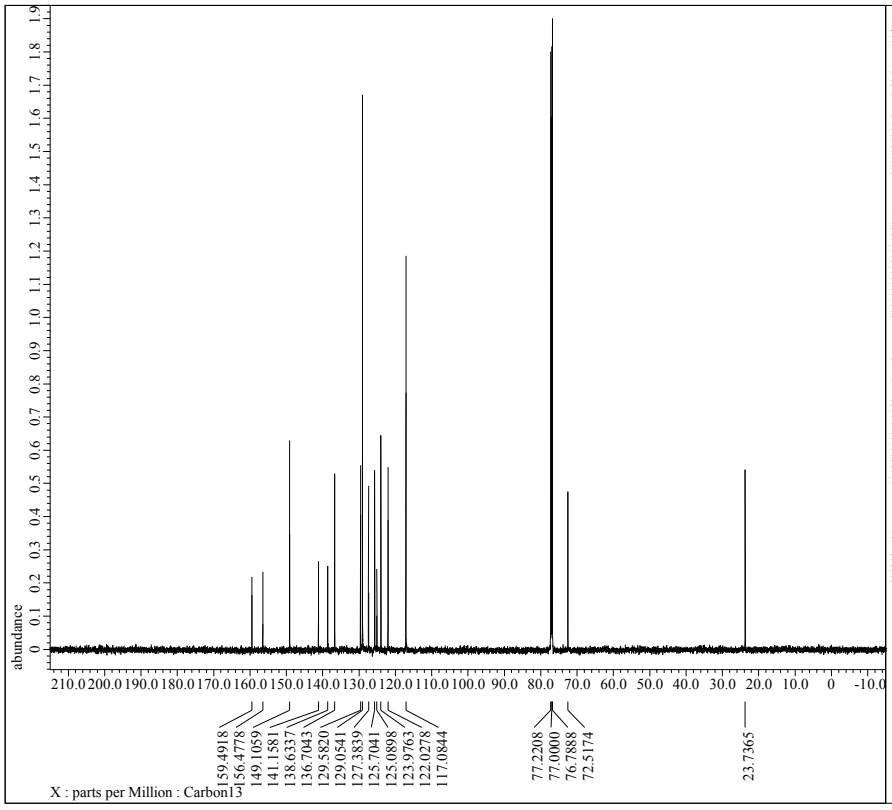

abundance
159.5110
157.7160
156.1226
153.8957
149.1059
141.3213
138.6721
136.6563
129.5532
129.0157
127.3167
125.7425
123.9859
121.9894
116.7485
116.7005
115.5678
115.4143
77.2112
77.0000
76.7792
72.6998
23.7749
X : parts per Million : Carbon13
Filename = ey9149p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9149p1
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2014 18:11:04
Revision_Time = 19-DEC-2014 18:23:42
Current_Time = 6-FEB-2015 15:05:04
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 = 0.69730304[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 = 24.4[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]
O
N
F
3ai



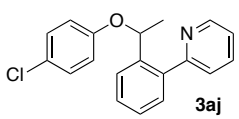

```
Filename          = ey9157p1_Proton-3-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9157p1
Solvent           = CHLOROFORM-D
Creation_Time     = 26-DEC-2014 21:02:10
Revision_Time     =  6-FEB-2015 15:05:33
Current_Time      =  6-FEB-2015 15:05:55

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  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 23.8[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   = 10.88251136[s]
```



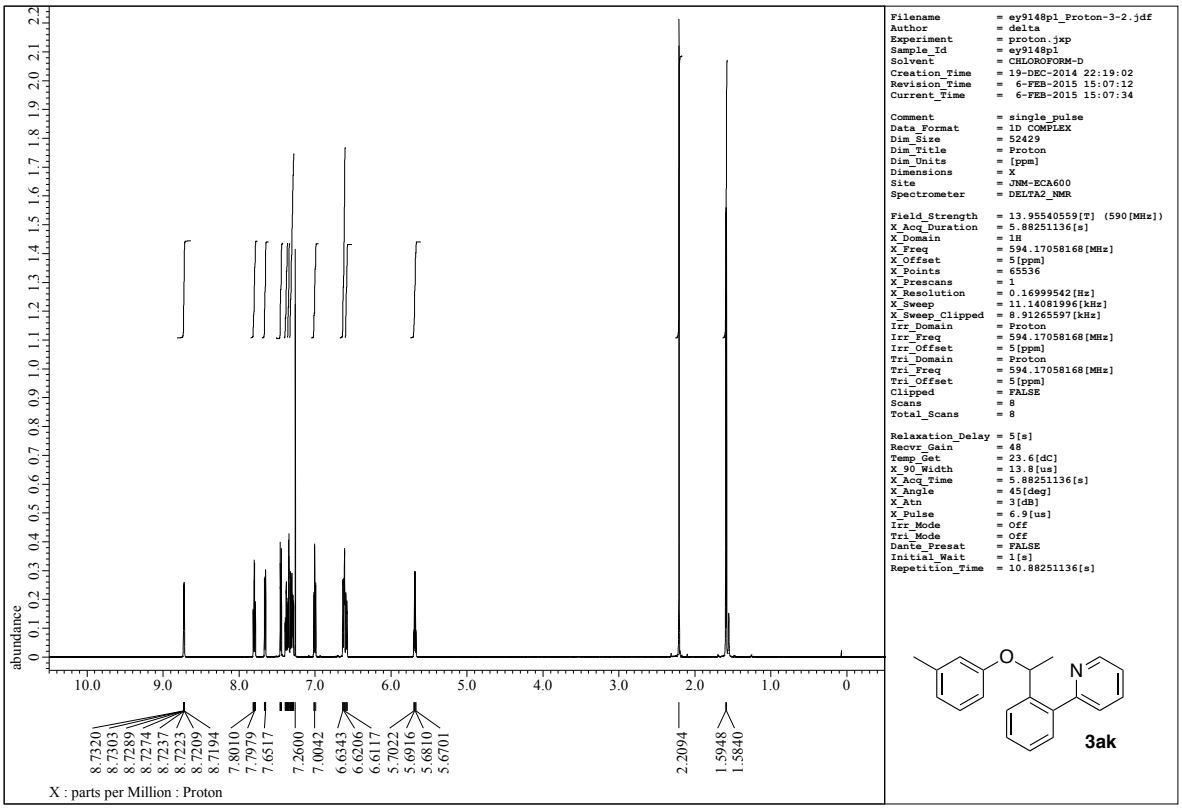
3aj

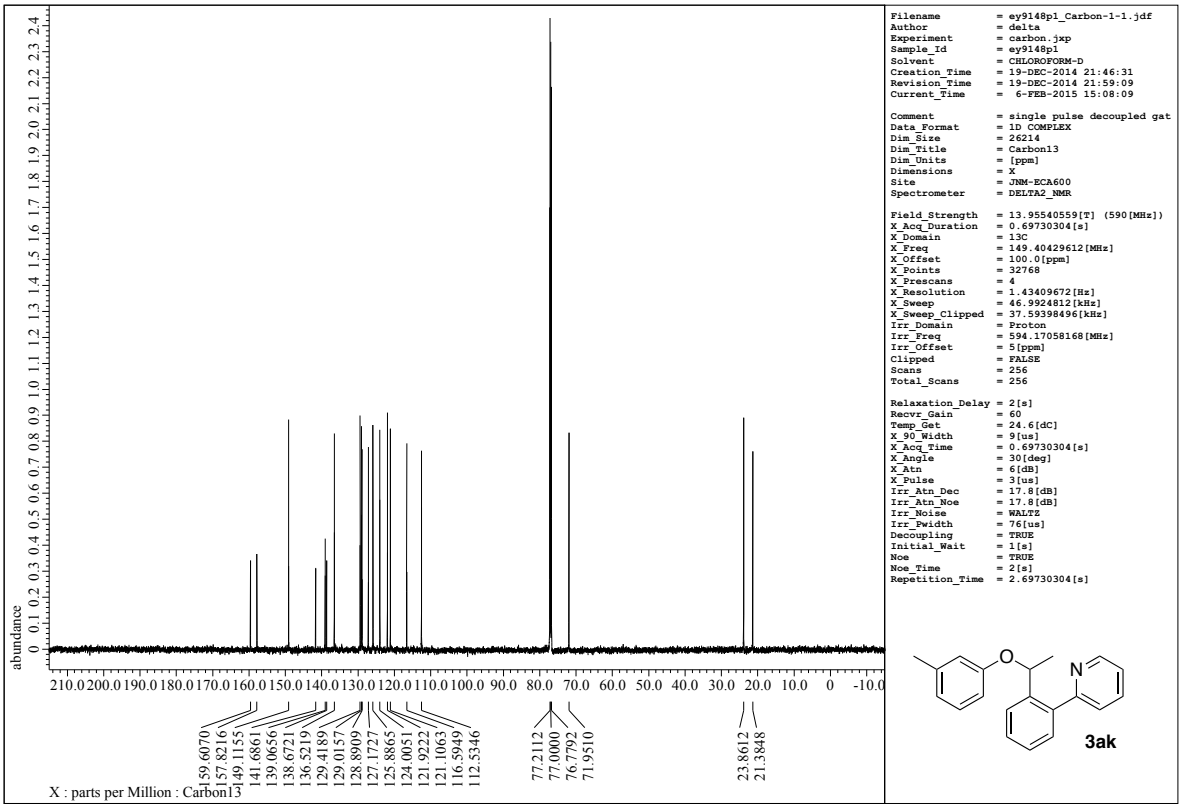


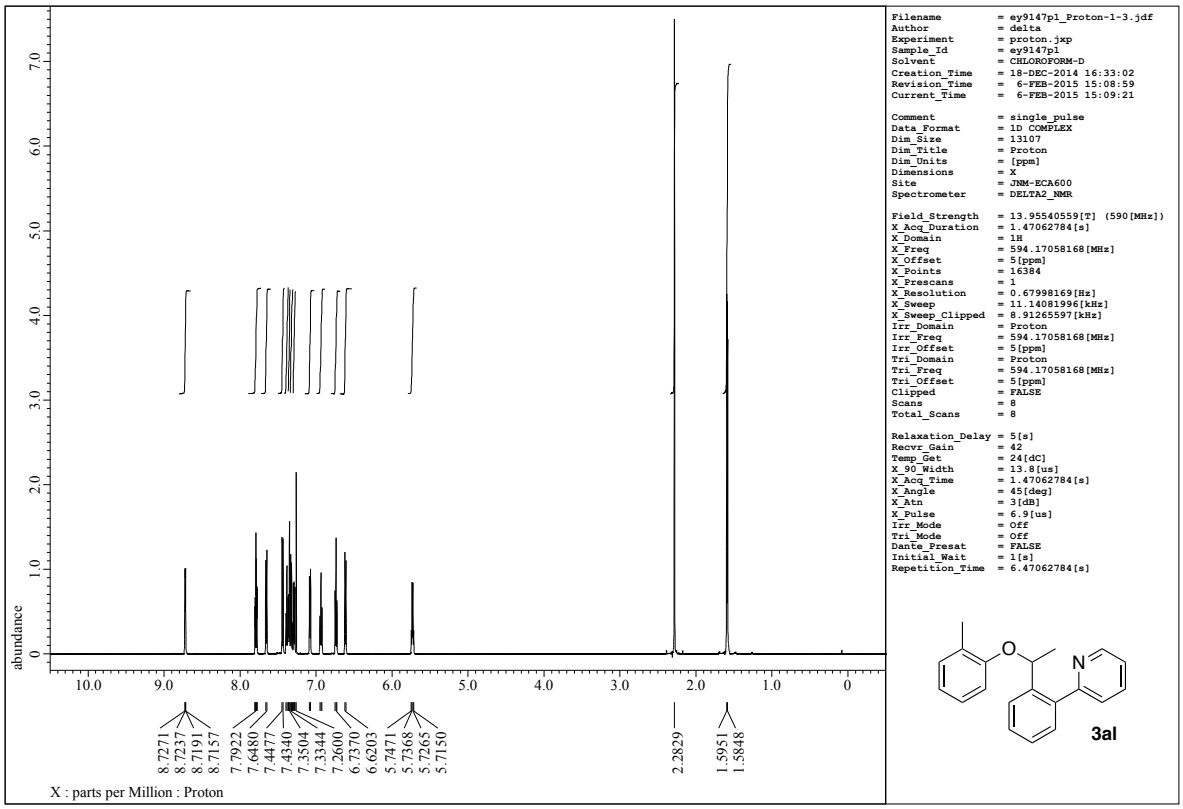

```
Filename          = ey8119p1_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey8119p1
Solvent           = CHLOROFORM-D
Creation_Time     = 18-JUL-2014 20:13:00
Revision_Time     = 18-JUL-2014 20:25:58
Current_Time      =  6-FEB-2015 15:06:44

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          = 100.0[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        = 58
Temp_Get          = 27.5[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]
```

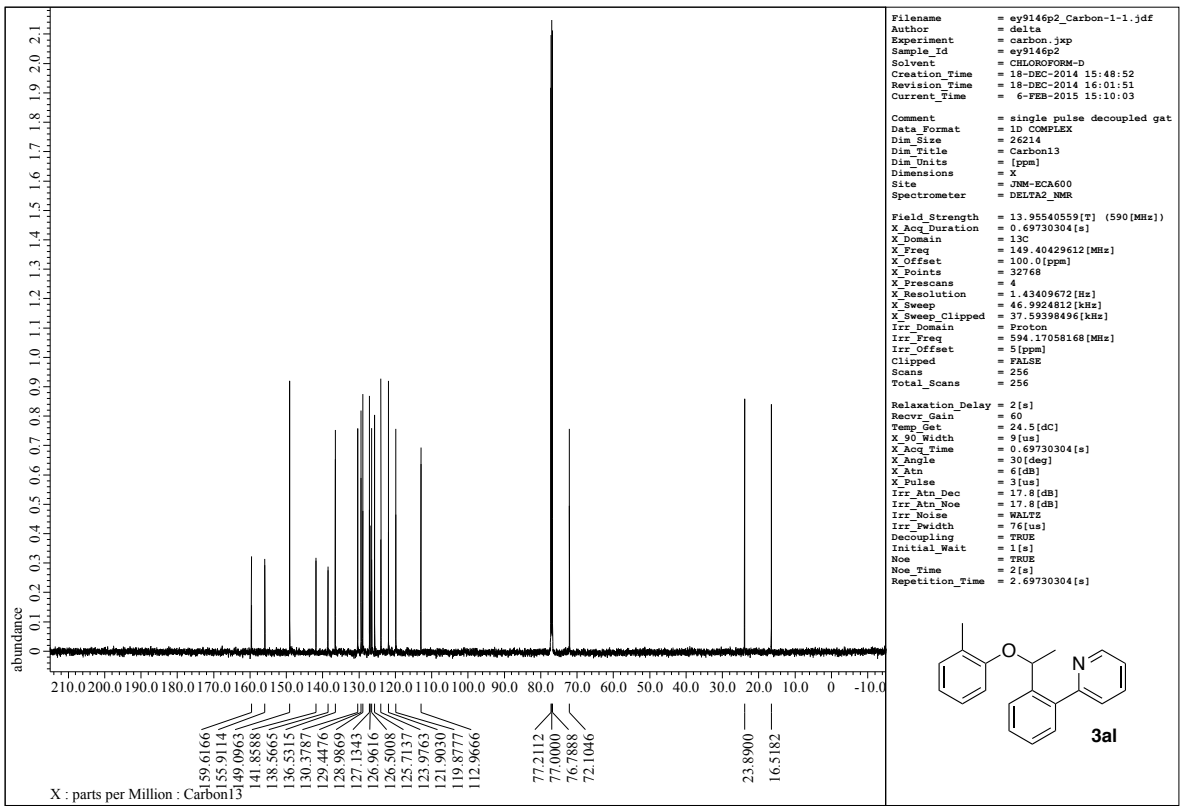


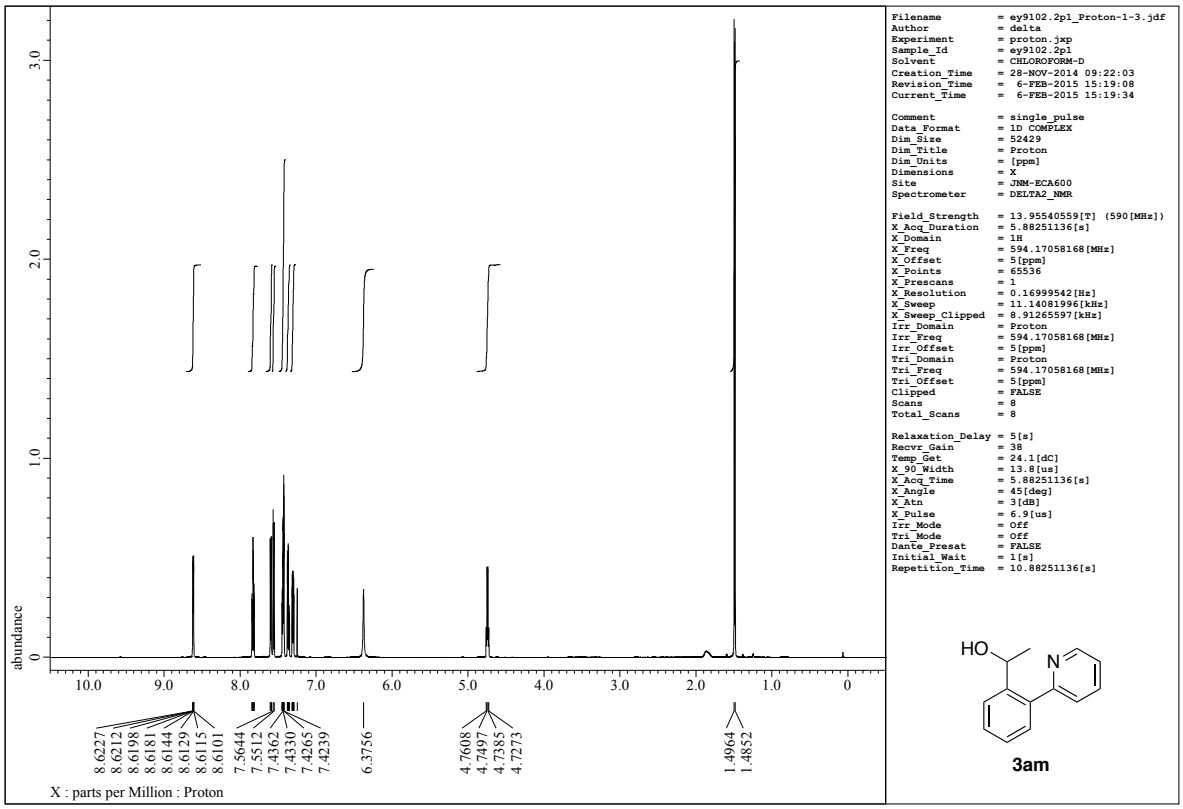
3aj

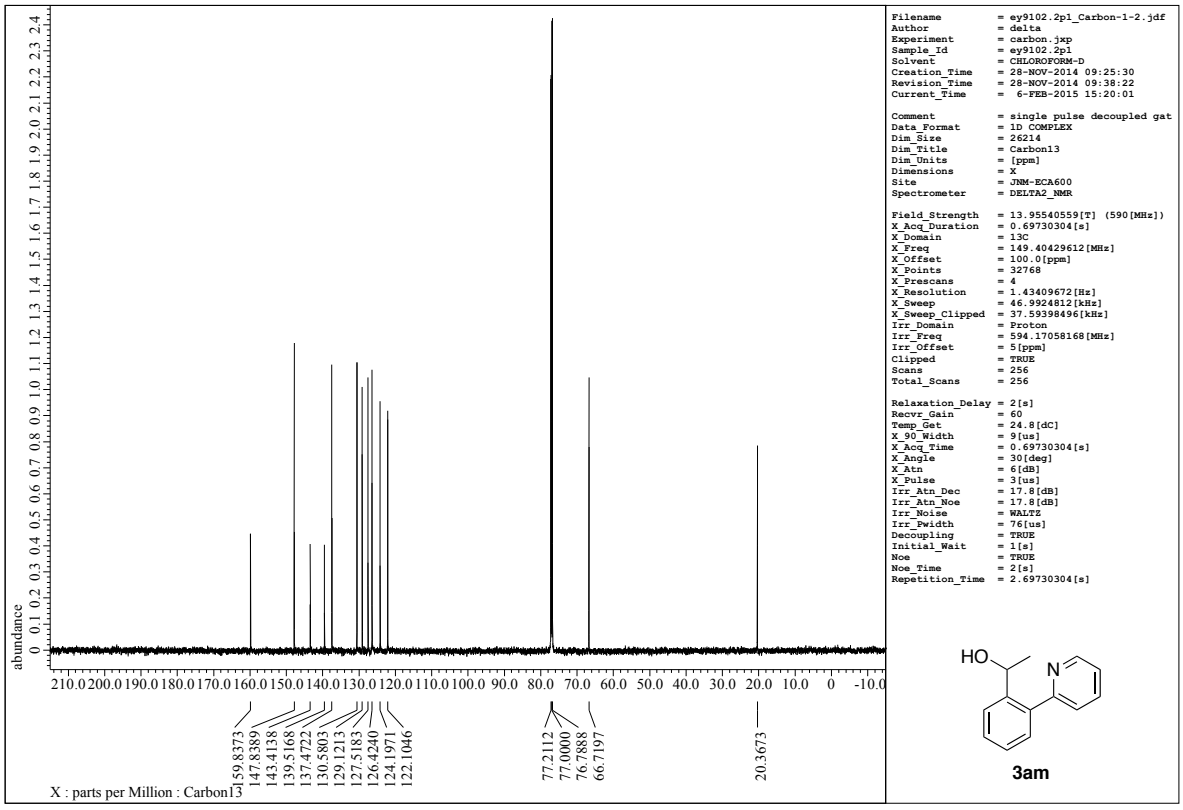

abundance
8.7320
8.7303
8.7289
8.7274
8.7237
8.7223
8.7209
8.7194
7.8010
7.7979
7.6517
7.2600
7.0042
6.6343
6.6206
6.6117
5.7022
5.6916
5.6810
5.6701
2.2094
1.5948
1.5840
X : parts per Million : Proton
Filename = ey9148p1_Proton-3-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9148p1
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2014 22:19:02
Revision_Time = 6-FEB-2015 15:07:12
Current_Time = 6-FEB-2015 15:07:34
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 = 5[s]
Recvr_Gain = 48
Temp_Get = 23.6[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 = 10.88251136[s]
O
N
3ak

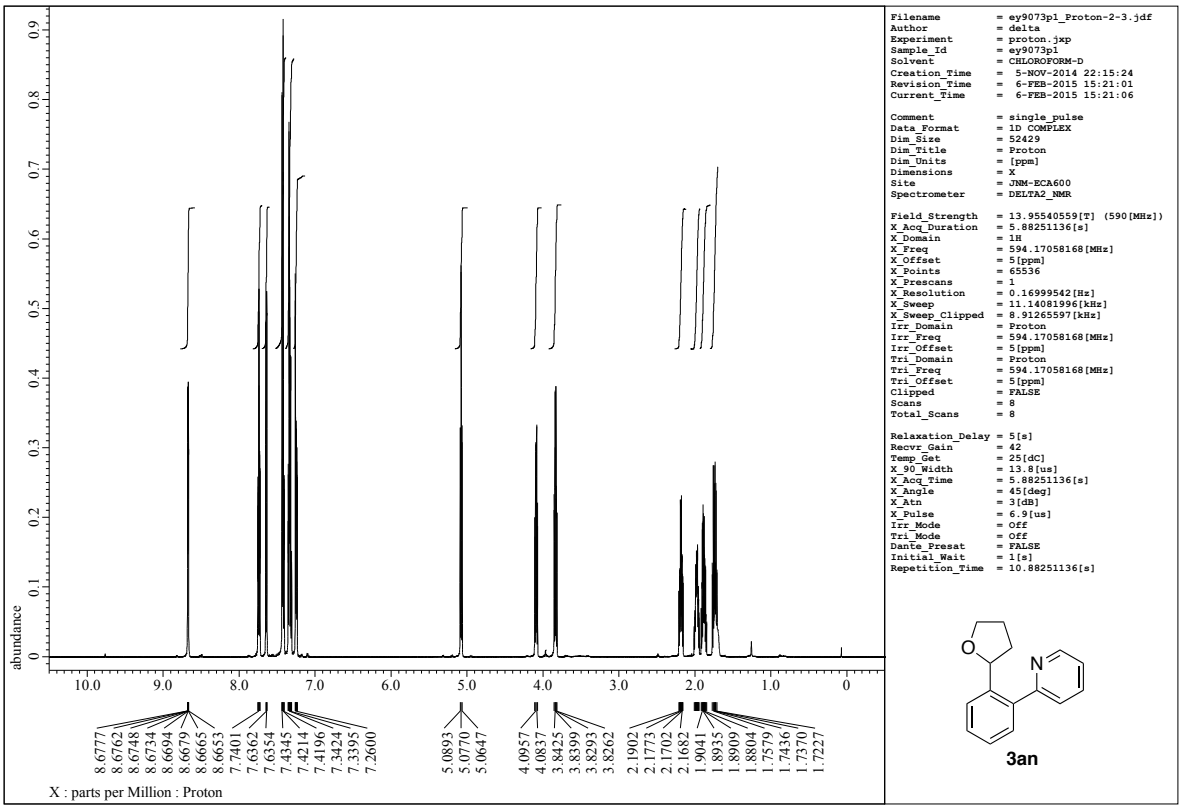

abundance
159.6070
157.8216
149.1155
141.6861
139.0656
138.6721
136.5219
129.4189
129.0157
128.8909
127.1727
125.8865
124.0051
121.9222
121.1063
116.5949
112.5346
77.2112
77.0000
76.7792
71.9510
23.8612
21.3848
X : parts per Million : Carbon13
Filename = ey9148p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9148p1
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2014 21:46:31
Revision_Time = 19-DEC-2014 21:59:09
Current_Time = 6-FEB-2015 15:08:09
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 = 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]
O
N
3ak

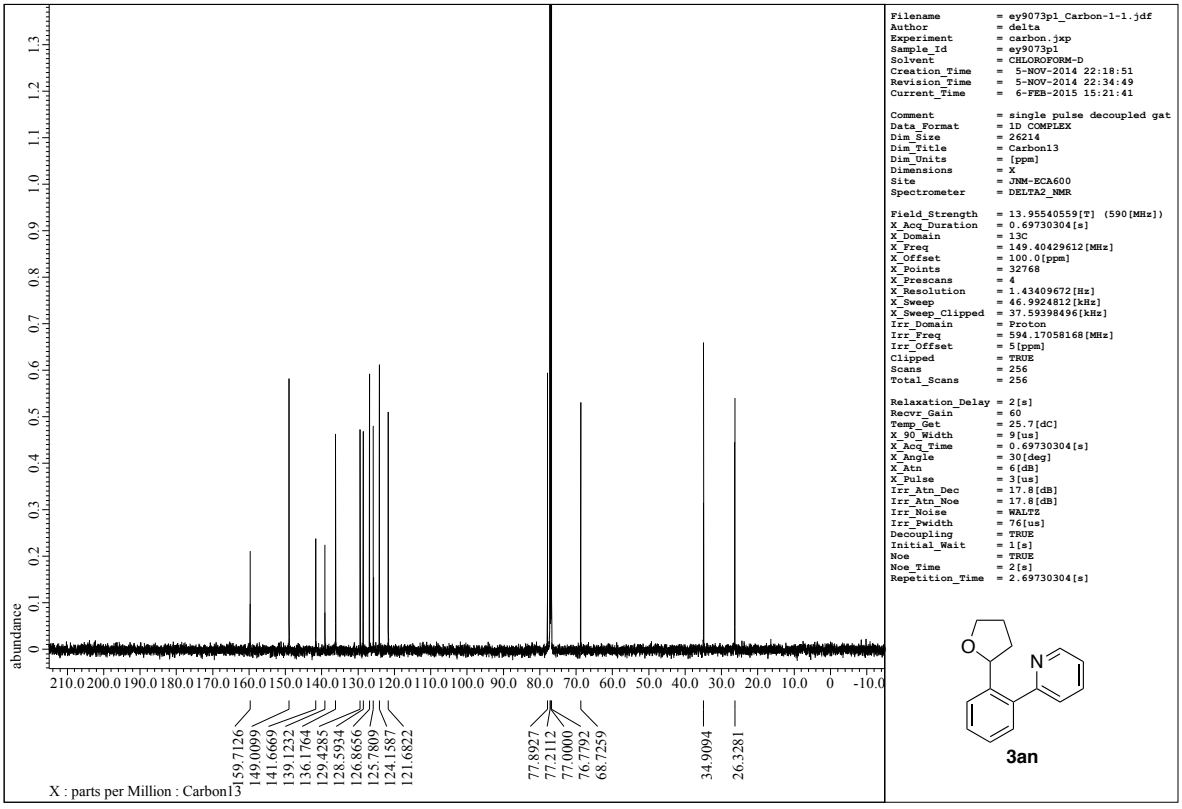

abundance
8.7271
8.7237
8.7191
8.7157
7.7922
7.6480
7.4477
7.4340
7.3504
7.3344
7.2600
7.7370
6.6203
5.7471
5.7368
5.7265
5.7150
2.2829
1.5951
1.5848
X : parts per Million : Proton
Filename = ey9147p1_Proton-1-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9147p1
Solvent = CHLOROFORM-D
Creation_Time = 18-DEC-2014 16:33:02
Revision_Time = 6-FEB-2015 15:08:59
Current_Time = 6-FEB-2015 15:09:21
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[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]
O
N
3al

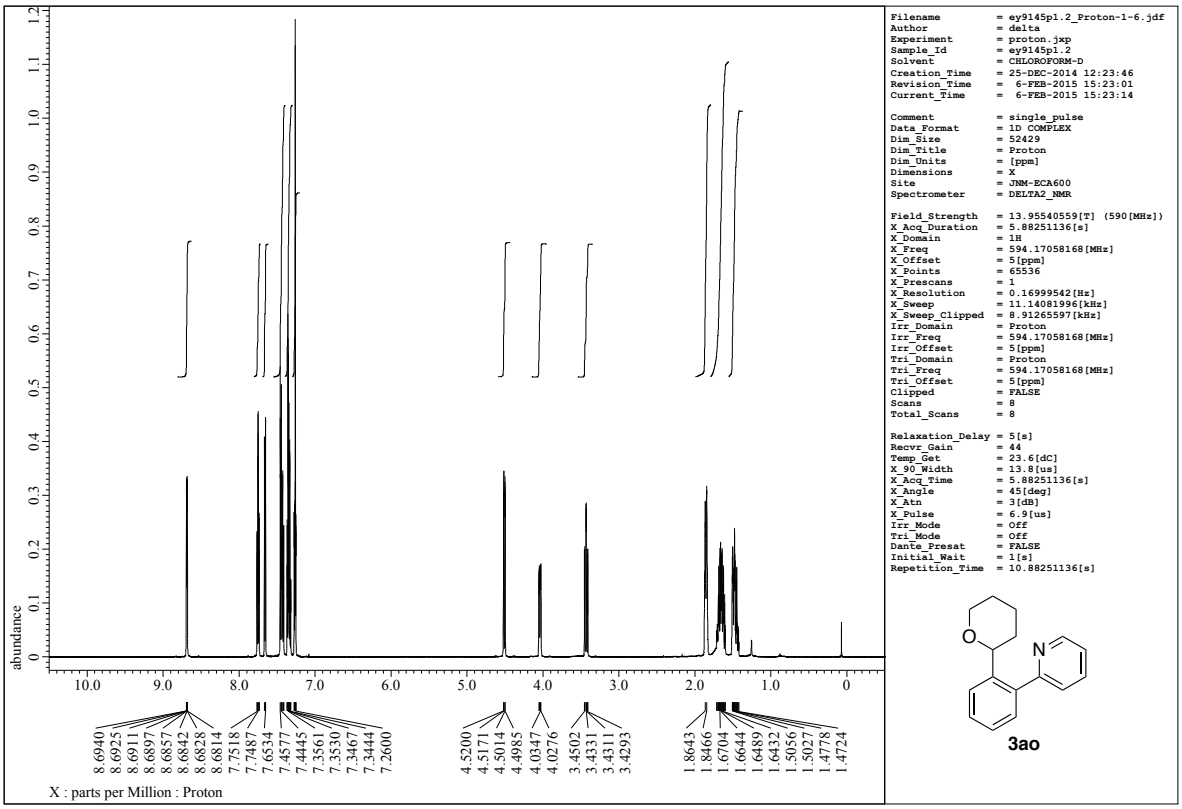

abundance
159.6166
155.9114
149.0963
141.8588
138.5665
136.5315
130.3787
129.4476
128.9869
127.1343
126.9616
126.5008
125.7137
123.9763
121.9030
119.8777
112.9666
77.2112
77.0000
76.7888
72.1046
23.8900
16.5182
X : parts per Million : Carbon13
Filename = ey9146p2_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9146p2
Solvent = CHLOROFORM-D
Creation_Time = 18-DEC-2014 15:48:52
Revision_Time = 18-DEC-2014 16:01:51
Current_Time = 6-FEB-2015 15:10:03
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 = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.5[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]
O
N
3al

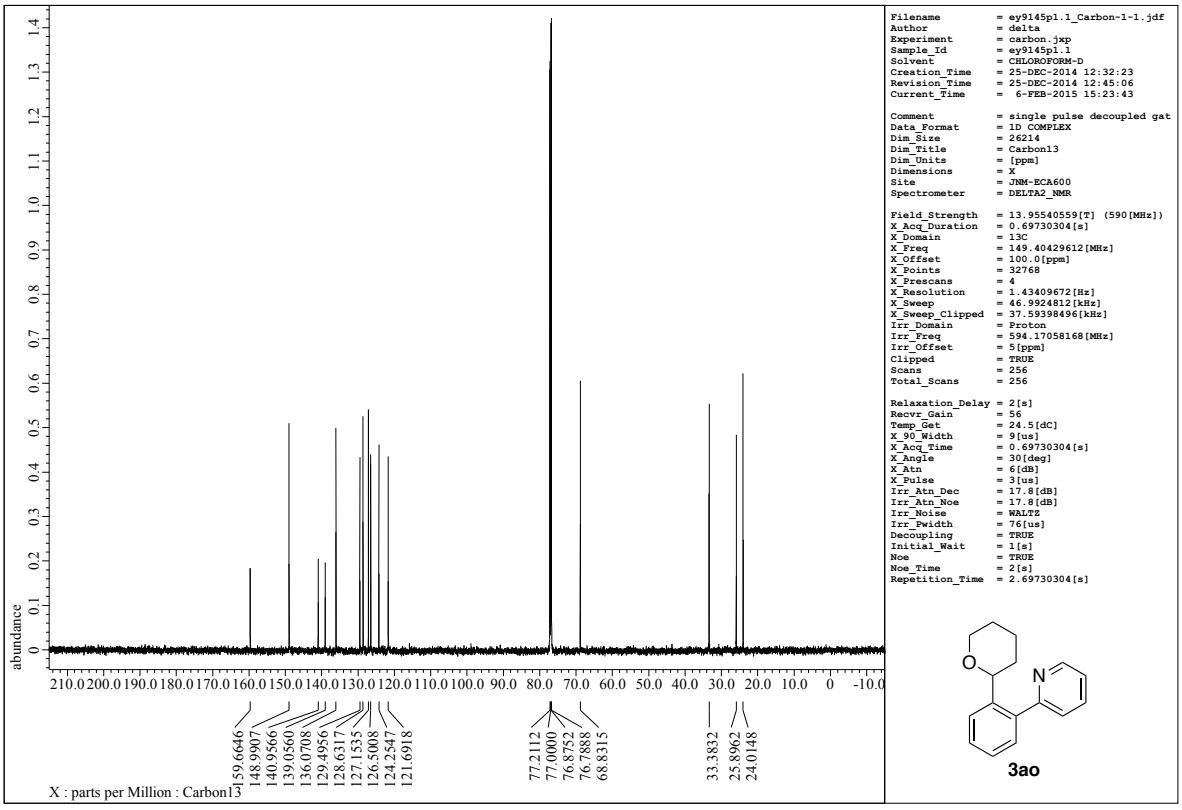

abundance
8.6227
8.6212
8.6198
8.6181
8.6144
8.6129
8.6115
8.6101
7.5644
7.5512
7.4362
7.4330
7.4265
7.4239
6.3756
4.7608
4.7497
4.7385
4.7273
1.4964
1.4852
X : parts per Million : Proton
Filename = ey9102.2p1_Proton-1-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9102.2p1
Solvent = CHLOROFORM-D
Creation_Time = 28-NOV-2014 09:22:03
Revision_Time = 6-FEB-2015 15:19:08
Current_Time = 6-FEB-2015 15:19:34
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 = 5[s]
Recvr_Gain = 38
Temp_Get = 24.1[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 = 10.88251136[s]
HO
N
3am

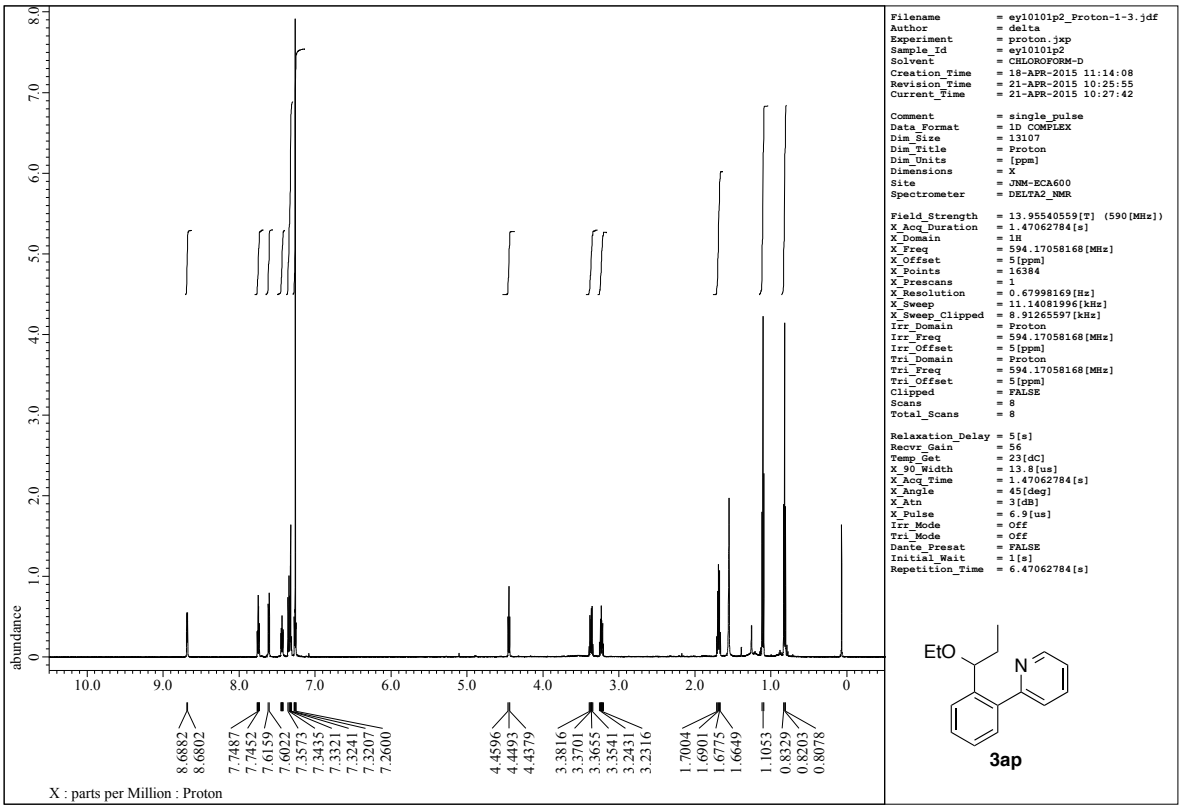

abundance
159.8373
147.8389
143.4138
139.5168
137.4722
130.5803
129.1213
127.5183
126.4240
124.1971
122.1046
77.2112
77.0000
76.7888
66.7197
20.3673
X : parts per Million : Carbon13
Filename = ey9102.2p1_Carbon-1-2.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9102.2p1
Solvent = CHLOROFORM-D
Creation_Time = 28-NOV-2014 09:25:30
Revision_Time = 28-NOV-2014 09:38:22
Current_Time = 6-FEB-2015 15:20:01
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 = 24.8[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]
HO
N
3am

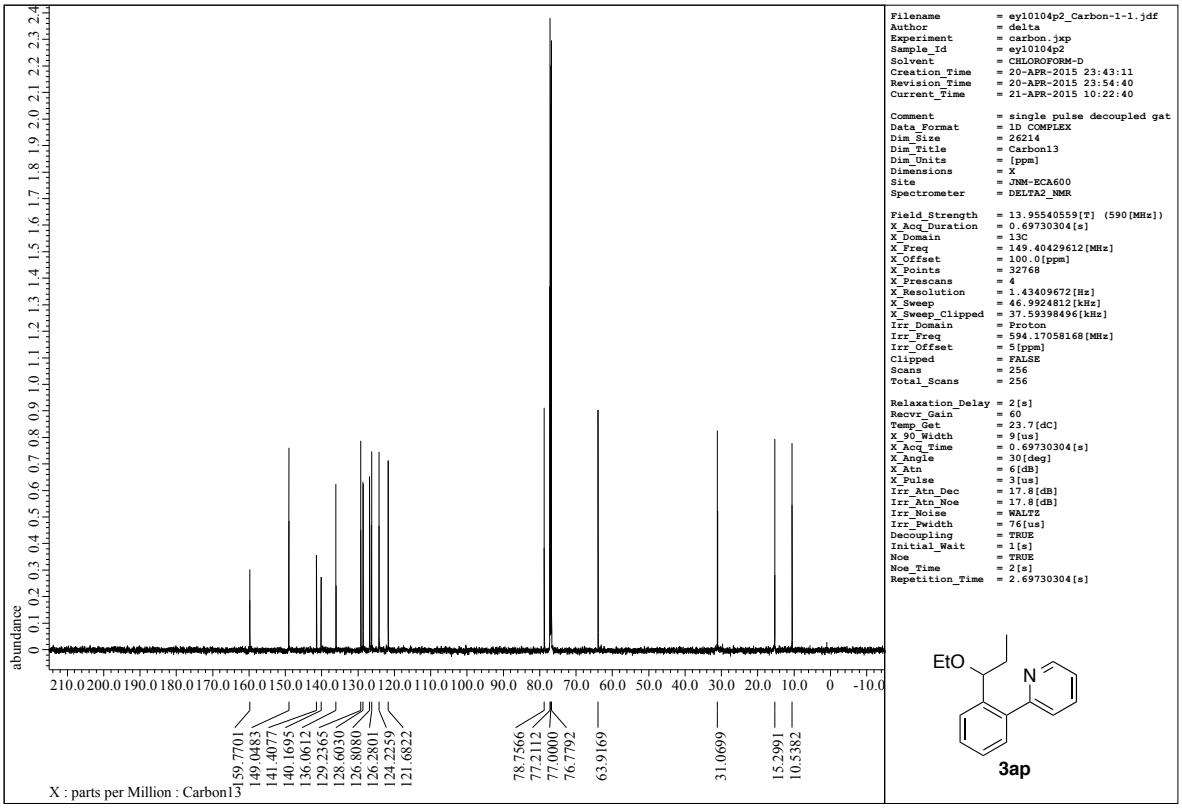

abundance
X : parts per Million : Proton
8.6777
8.6762
8.6748
8.6734
8.6694
8.6679
8.6665
8.6653
7.7401
7.6362
7.6354
7.4345
7.4214
7.4196
7.3424
7.3395
7.2600
5.0893
5.0770
5.0647
4.0957
4.0837
3.8425
3.8399
3.8293
3.8262
2.1902
2.1773
2.1702
2.1682
1.9041
1.8935
1.8909
1.8804
1.7579
1.7436
1.7370
1.7227
Filename = ey9073pl_Proton-2-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9073pl
Solvent = CHLOROFORM-D
Creation_Time = 5-NOV-2014 22:15:24
Revision_Time = 6-FEB-2015 15:21:01
Current_Time = 6-FEB-2015 15:21:06
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 = 5[s]
Recvr_Gain = 42
Temp_Get = 25[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 = 10.88251136[s]
O
N
3an

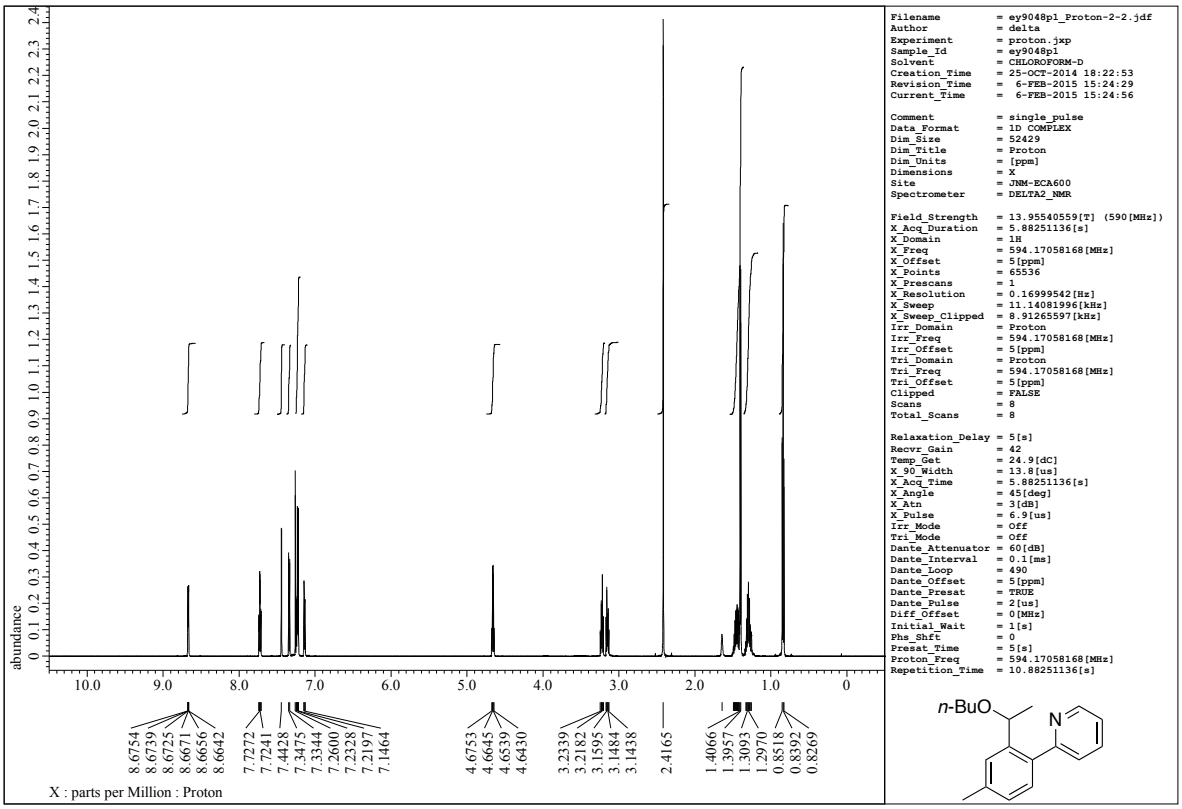

abundance
X : parts per Million : Carbon13
159.7126
149.0099
141.6669
139.1232
136.1764
129.4285
128.5934
126.8656
125.7809
124.1587
121.6822
77.8927
77.2112
77.0000
76.7792
68.7259
34.9094
26.3281
Filename = ey9073pl_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9073pl
Solvent = CHLOROFORM-D
Creation_Time = 5-NOV-2014 22:18:51
Revision_Time = 5-NOV-2014 22:34:49
Current_Time = 6-FEB-2015 15:21:41
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 = 25.7[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]
O
N
3an


abundance
X : parts per Million : Proton
8.6940
8.6925
8.6911
8.6897
8.6857
8.6842
8.6828
8.6814
7.7518
7.7487
7.6534
7.4577
7.4445
7.3561
7.3530
7.3467
7.3444
7.2600
4.5200
4.5171
4.5014
4.4985
4.0347
4.0276
3.4502
3.4331
3.4311
3.4293
1.8643
1.8466
1.6704
1.6644
1.6489
1.6432
1.5056
1.5027
1.4778
1.4724
Filename = ey9145p1.2_Proton-1-6.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9145p1.2
Solvent = CHLOROFORM-D
Creation_Time = 25-DEC-2014 12:23:46
Revision_Time = 6-FEB-2015 15:23:01
Current_Time = 6-FEB-2015 15:23:14
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 = 5[s]
Recvr_Gain = 44
Temp_Get = 23.6[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 = 10.88251136[s]
O
N
3ao

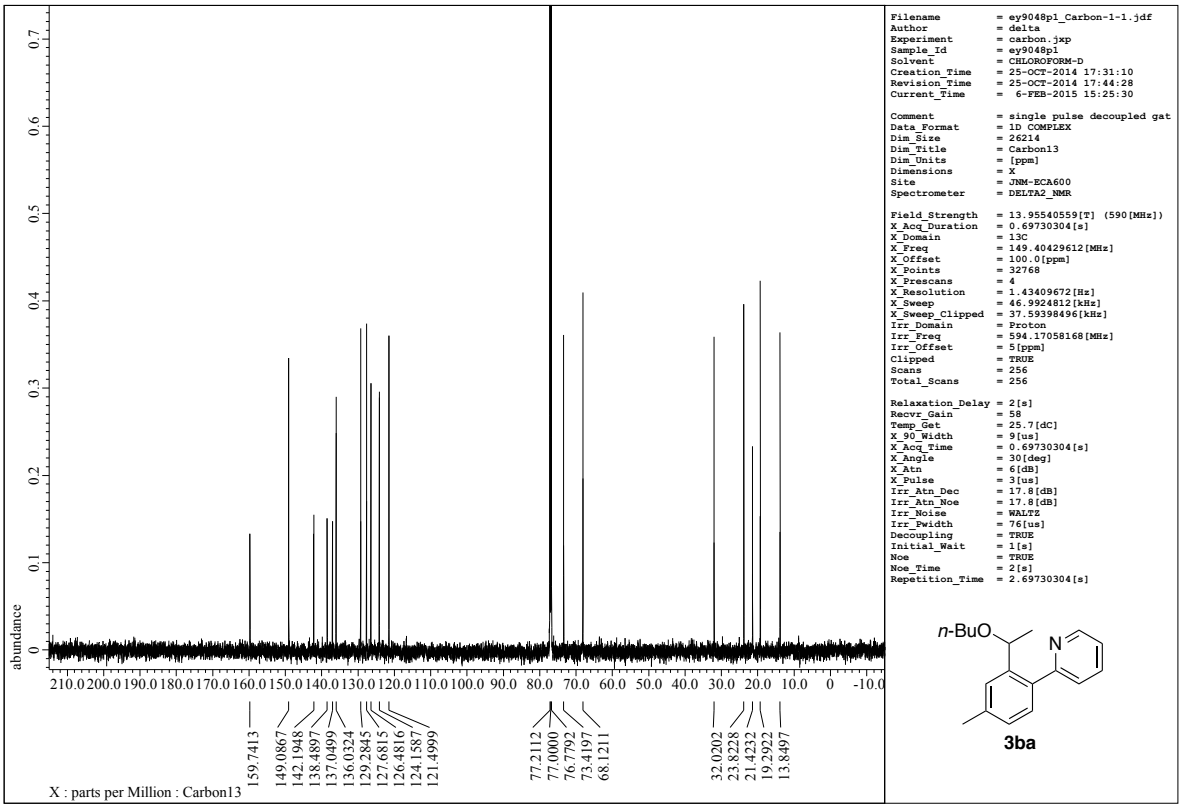

abundance
X : parts per Million : Carbon13
159.6646
148.9907
140.9566
139.0560
136.0708
129.4956
128.6317
127.1535
126.5008
124.2547
121.6918
77.2112
77.0000
76.8752
76.7888
68.8315
33.3832
25.8962
24.0148
Filename = ey9145p1.1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9145p1.1
Solvent = CHLOROFORM-D
Creation_Time = 25-DEC-2014 12:32:23
Revision_Time = 25-DEC-2014 12:45:06
Current_Time = 6-FEB-2015 15:23:43
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 = 56
Temp_Get = 24.5[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]
O
N
3ao


abundance
8.6882
8.6802
7.7487
7.7452
7.6159
7.6022
7.3573
7.3435
7.3321
7.3241
7.3207
7.2600
4.4596
4.4493
4.4379
3.3816
3.3701
3.3655
3.3541
3.2431
3.2316
1.7004
1.6901
1.6775
1.6649
1.1053
0.8329
0.8203
0.8078
X : parts per Million : Proton
Filename = ey10101p2_Proton-1-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey10101p2
Solvent = CHLOROFORM-D
Creation_Time = 18-APR-2015 11:14:08
Revision_Time = 21-APR-2015 10:25:55
Current_Time = 21-APR-2015 10:27:42
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 = 56
Temp_Get = 23[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]
EtO
N
3ap

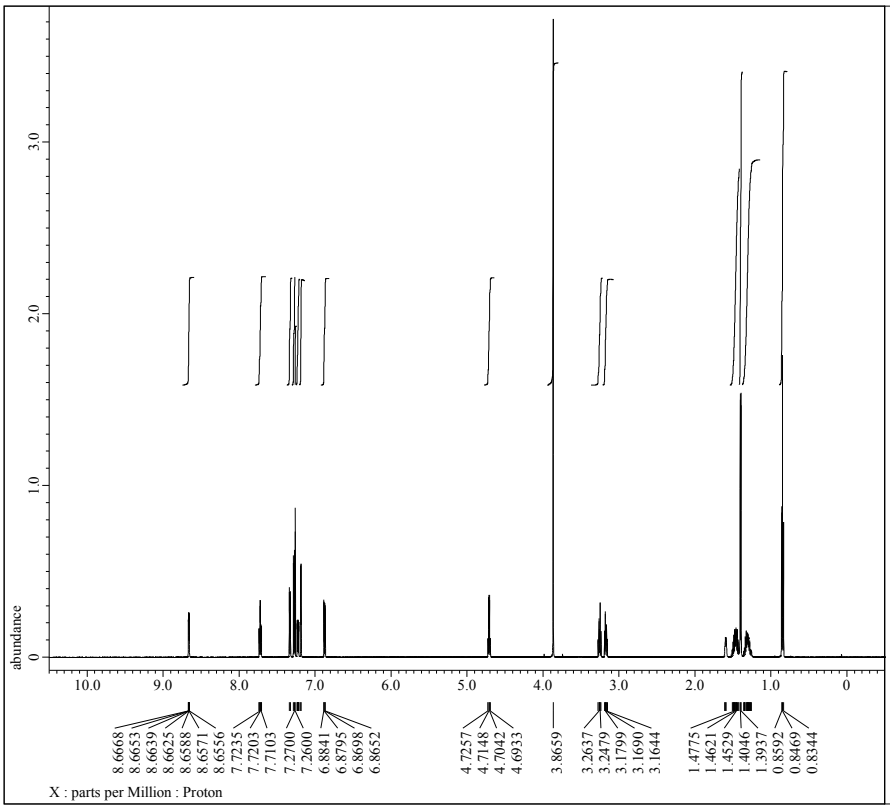

abundance
159.7701
149.0483
141.4077
140.1695
136.0612
129.2365
128.6030
126.8080
126.2801
124.2259
121.6822
78.7566
77.2112
77.0000
76.7792
63.9169
31.0699
15.2991
10.5382
X : parts per Million : Carbon13
Filename = ey10104p2_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey10104p2
Solvent = CHLOROFORM-D
Creation_Time = 20-APR-2015 23:43:11
Revision_Time = 20-APR-2015 23:54:40
Current_Time = 21-APR-2015 10:22:40
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 = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.7[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]
EtO
N
3ap


abundance
X : parts per Million : Proton
8.6754
8.6739
8.6725
8.6671
8.6656
8.6642
7.7272
7.7241
7.4428
7.3475
7.3344
7.2600
7.2328
7.2197
7.1464
4.6753
4.6645
4.6539
4.6430
3.2339
3.2182
3.1595
3.1484
3.1438
2.4165
1.4066
1.3957
1.3093
1.2970
0.8518
0.8392
0.8269
Filename = ey9048p1_Proton-2-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9048p1
Solvent = CHLOROFORM-D
Creation_Time = 25-OCT-2014 18:22:53
Revision_Time = 6-FEB-2015 15:24:29
Current_Time = 6-FEB-2015 15:24:56
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 = 5[s]
Recvr_Gain = 42
Temp_Get = 24.9[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_Attenuator = 60[dB]
Dante_Interval = 0.1[ms]
Dante_Loop = 490
Dante_Offset = 5[ppm]
Dante_Presat = TRUE
Dante_Pulse = 2[us]
Diff_Offset = 0[MHz]
Initial_Wait = 1[s]
Phs_Shft = 0
Presat_Time = 5[s]
Proton_Freq = 594.17058168[MHz]
Repetition_Time = 10.88251136[s]
n-BuO
N

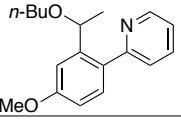
3ba


abundance
X : parts per Million : Carbon13
159.7413
149.0867
142.1948
138.4897
137.0499
136.0324
129.2845
127.6815
126.4816
124.1587
121.4999
77.2112
77.0000
76.7792
73.4197
68.1211
32.0202
23.8228
21.4232
19.2922
13.8497
Filename = ey9048p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9048p1
Solvent = CHLOROFORM-D
Creation_Time = 25-OCT-2014 17:31:10
Revision_Time = 25-OCT-2014 17:44:28
Current_Time = 6-FEB-2015 15:25:30
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 = 25.7[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]
n-BuO
N

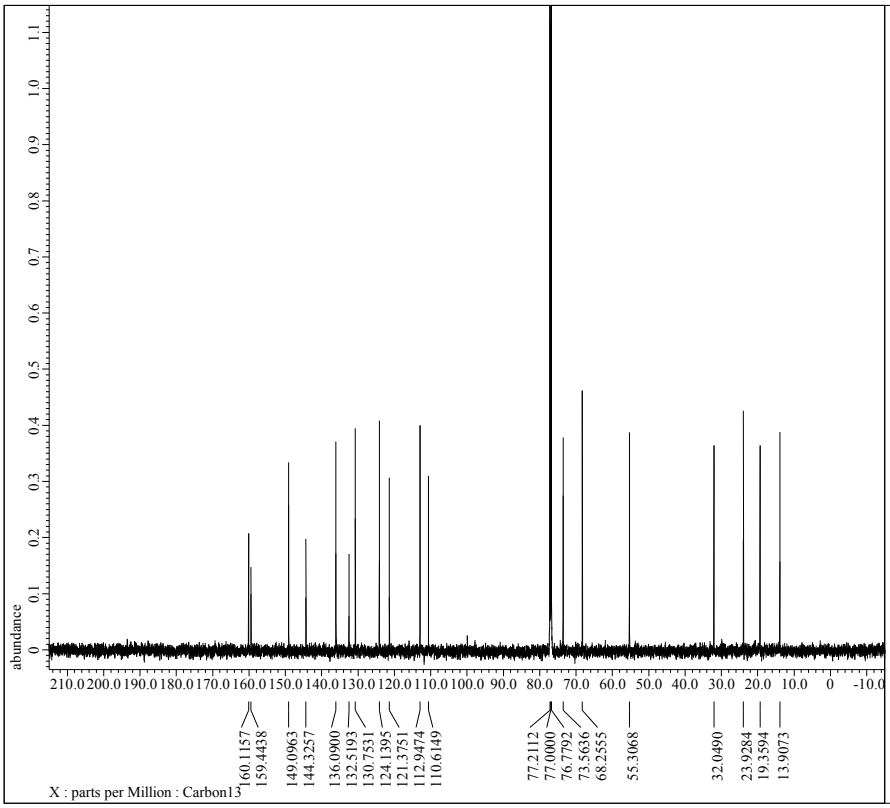
3ba



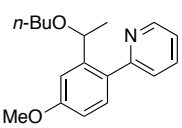

```
Filename          = ey9084p1_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9084p1
Solvent           = CHLOROFORM-D
Creation_Time     = 10-NOV-2014 14:42:30
Revision_Time     =  6-FEB-2015 15:26:23
Current_Time      =  6-FEB-2015 15:26:44

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  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 24.5[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_Attenuator  = 60[dB]
Dante_Interval    = 0.1[ms]
Dante_Loop        = 490
Dante_Offset      = 5[ppm]
Dante_Presat      = TRUE
Dante_Pulse       = 2[us]
Diff_Offset       = 0[MHz]
Initial_Wait      = 1[s]
Phs_Shft          = 0
Presat_Time       = 5[s]
Proton_Freq       = 594.17058168[MHz]
Repetition_Time   = 10.88251136[s]
```



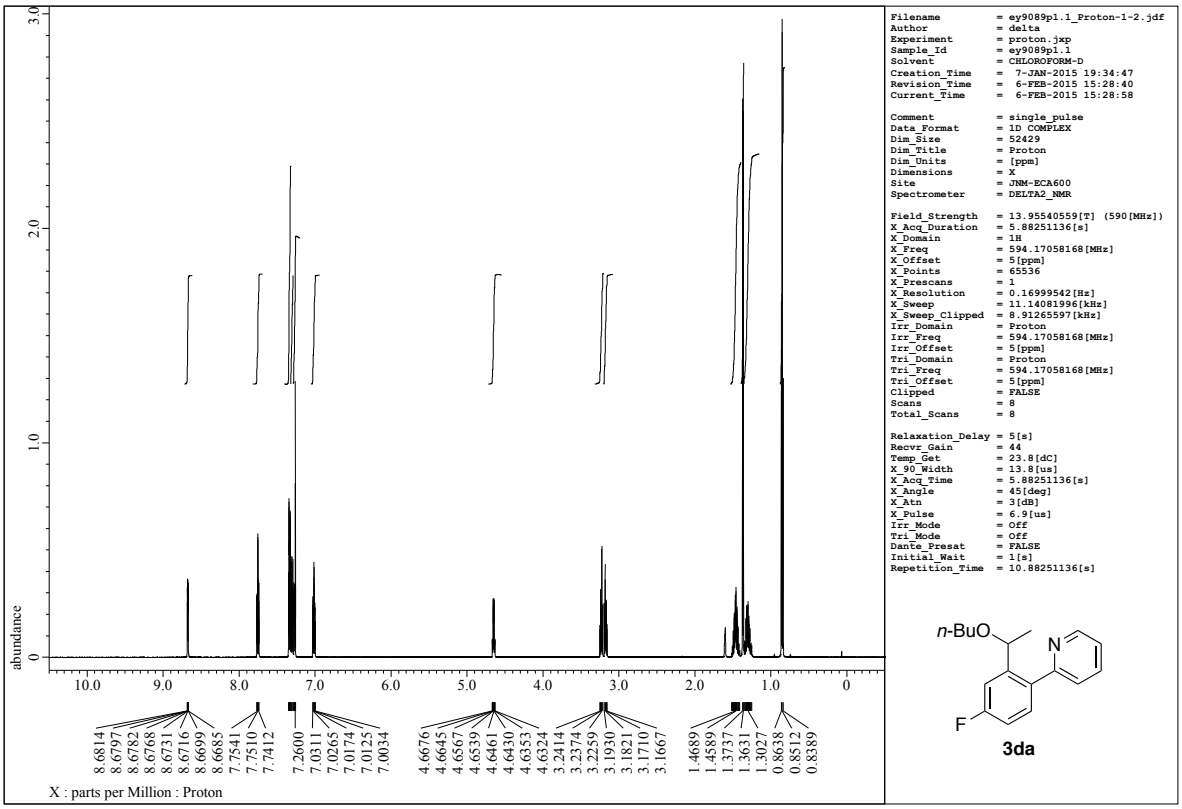
3ca

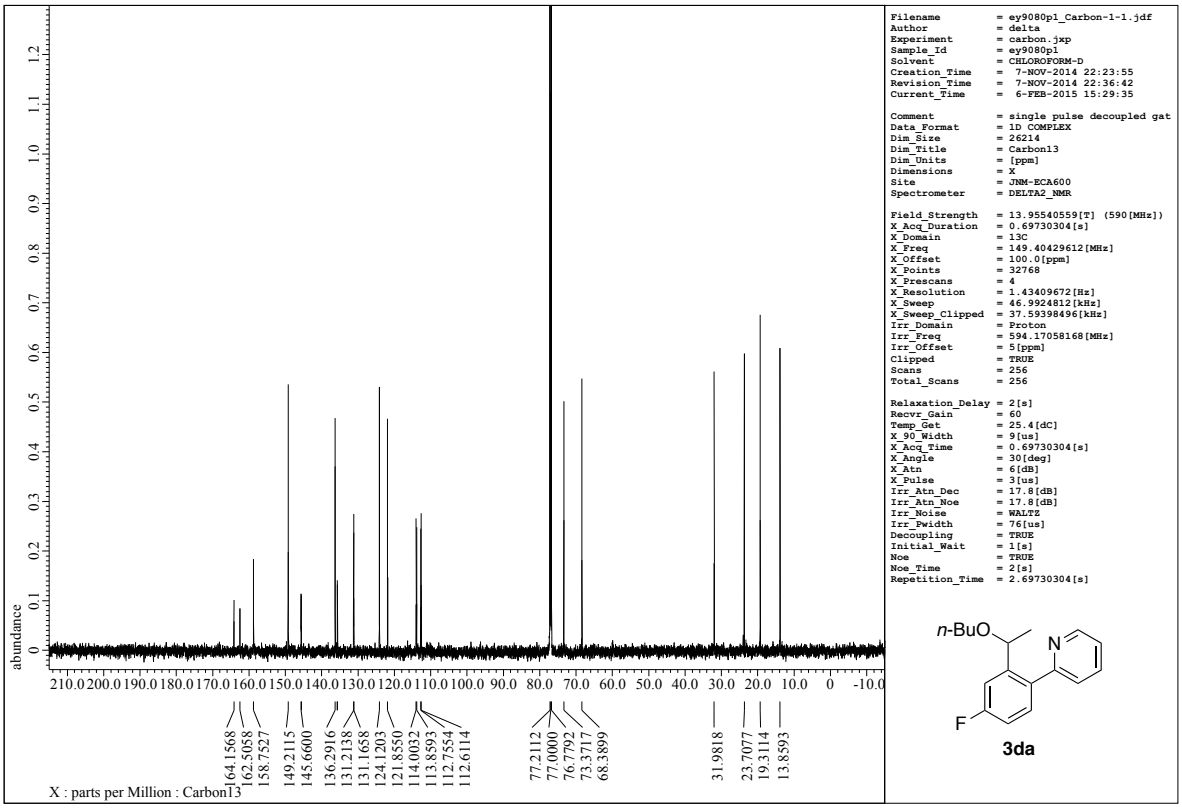


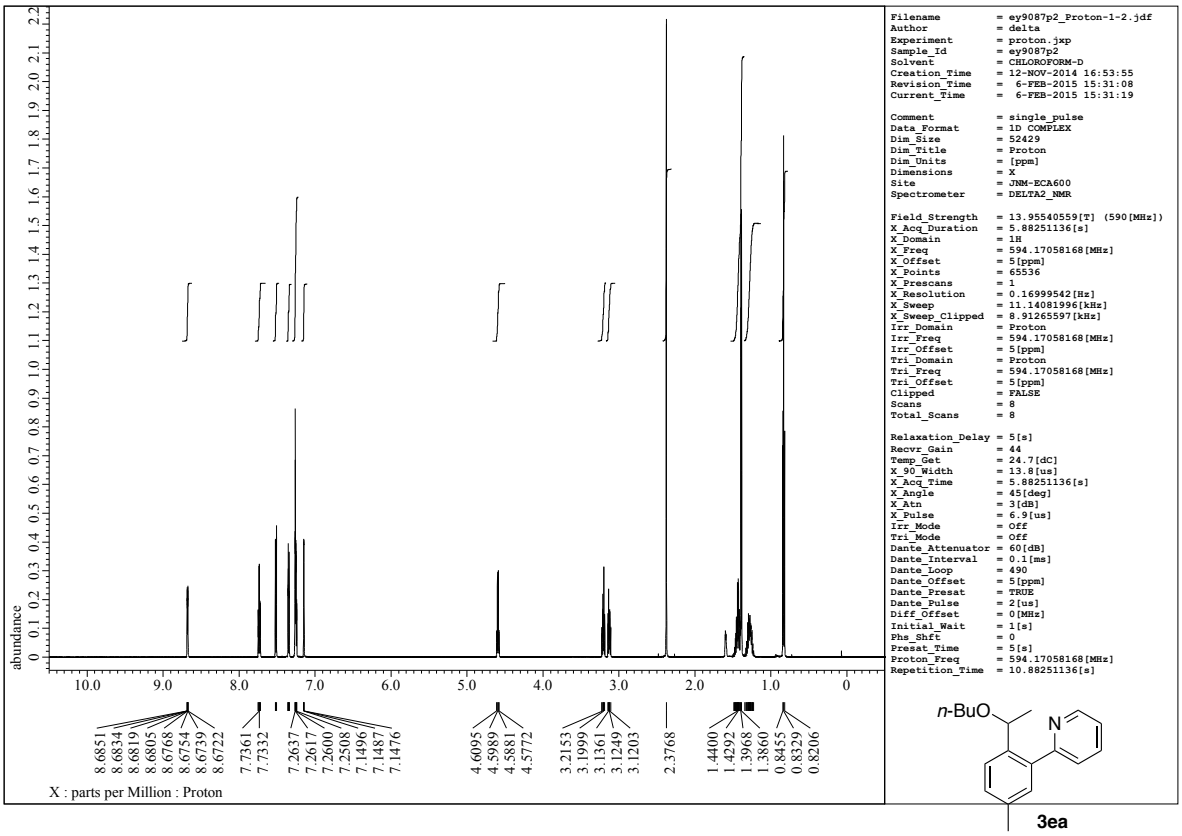

```
Filename          = ey9079p1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey9079p1
Solvent           = CHLOROFORM-D
Creation_Time     =  7-NOV-2014 21:30:34
Revision_Time     =  7-NOV-2014 21:43:08
Current_Time      =  6-FEB-2015 15:27:21

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          = 25.5[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]
```

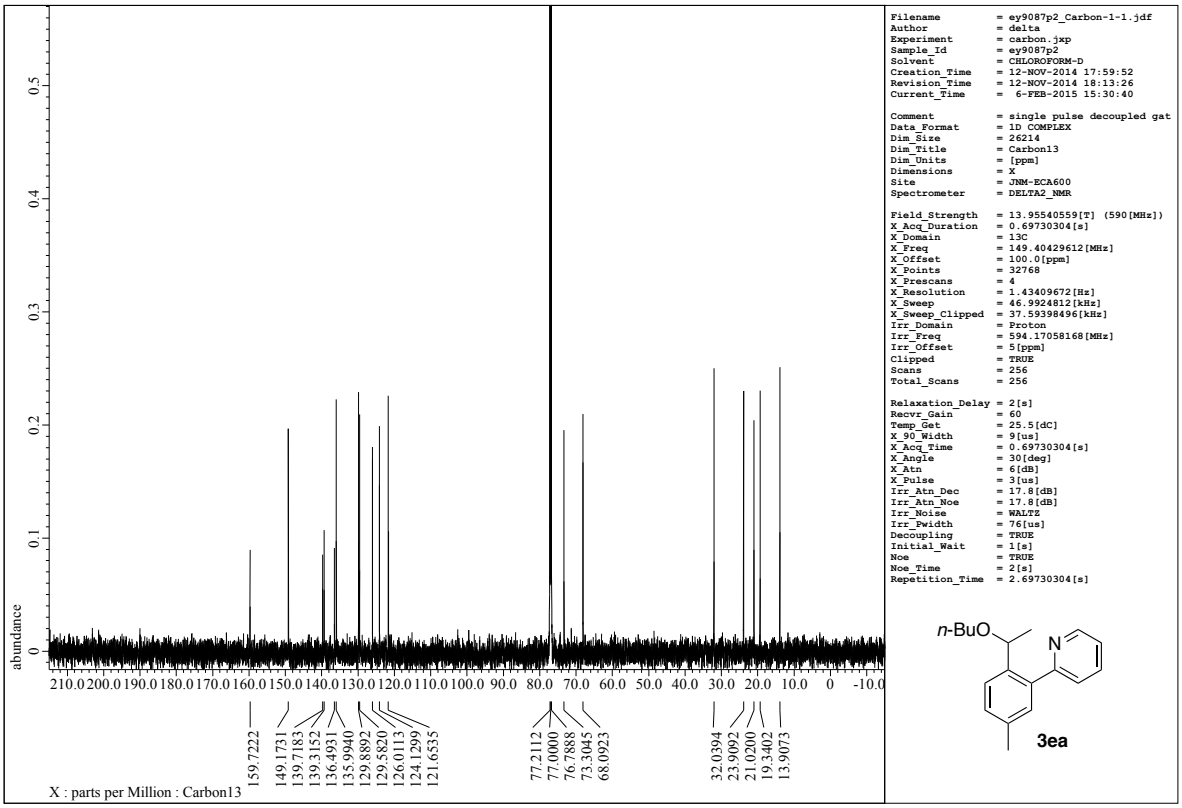


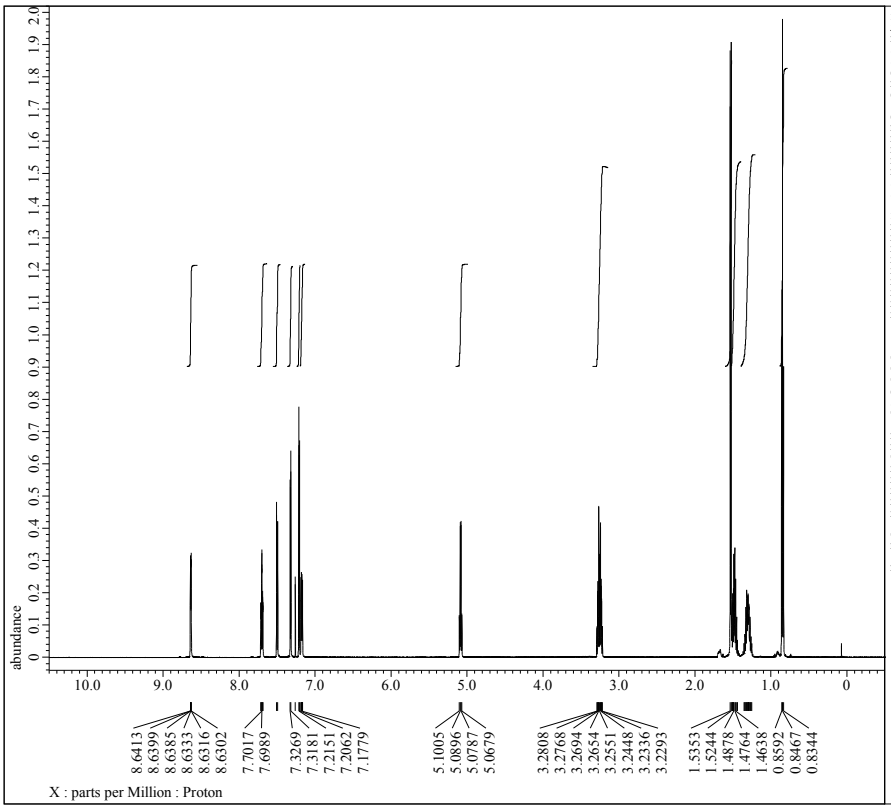
3ca


abundance
X : parts per Million : Proton
8.6814
8.6797
8.6782
8.6768
8.6731
8.6716
8.6699
8.6685
7.7541
7.7510
7.7412
7.2600
7.0311
7.0265
7.0174
7.0125
7.0034
4.6676
4.6645
4.6567
4.6539
4.6461
4.6430
4.6353
4.6324
3.2414
3.2374
3.2259
3.1930
3.1821
3.1710
3.1667
1.4689
1.4589
1.3737
1.3631
1.3027
0.8638
0.8512
0.8389
Filename = ey9089p1.1_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9089p1.1
Solvent = CHLOROFORM-D
Creation_Time = 7-JAN-2015 19:34:47
Revision_Time = 6-FEB-2015 15:28:40
Current_Time = 6-FEB-2015 15:28:58
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 = 5[s]
Recvr_Gain = 44
Temp_Get = 23.8[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 = 10.88251136[s]
n-BuO
N
F
3da

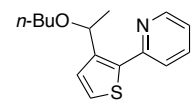

abundance
X : parts per Million : Carbon13
164.1568
162.5058
158.7527
149.2115
145.6600
136.2916
131.2138
131.1658
124.1203
121.8550
114.0032
113.8593
112.7554
112.6114
77.2112
77.0000
76.7792
73.3717
68.3899
31.9818
23.7077
19.3114
13.8593
Filename = ey9080p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9080p1
Solvent = CHLOROFORM-D
Creation_Time = 7-NOV-2014 22:23:55
Revision_Time = 7-NOV-2014 22:36:42
Current_Time = 6-FEB-2015 15:29:35
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 = 25.4[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]
n-BuO
N
F
3da


abundance
X : parts per Million : Proton
8.6851
8.6834
8.6819
8.6805
8.6768
8.6754
8.6739
8.6722
7.7361
7.7332
7.2637
7.2617
7.2600
7.2508
7.1496
7.1487
7.1476
4.6095
4.5989
4.5881
4.5772
3.2153
3.1999
3.1361
3.1249
3.1203
2.3768
1.4400
1.4292
1.3968
1.3860
0.8455
0.8329
0.8206
Filename = ey9087p2_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9087p2
Solvent = CHLOROFORM-D
Creation_Time = 12-NOV-2014 16:53:55
Revision_Time = 6-FEB-2015 15:31:08
Current_Time = 6-FEB-2015 15:31:19
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 = 5[s]
Recvr_Gain = 44
Temp_Get = 24.7[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_Attenuator = 60[dB]
Dante_Interval = 0.1[ms]
Dante_Loop = 490
Dante_Offset = 5[ppm]
Dante_Presat = TRUE
Dante_Pulse = 2[us]
Diff_Offset = 0[MHz]
Initial_Wait = 1[s]
Phs_Shft = 0
Presat_Time = 5[s]
Proton_Freq = 594.17058168[MHz]
Repetition_Time = 10.88251136[s]
n-BuO
N
3ea

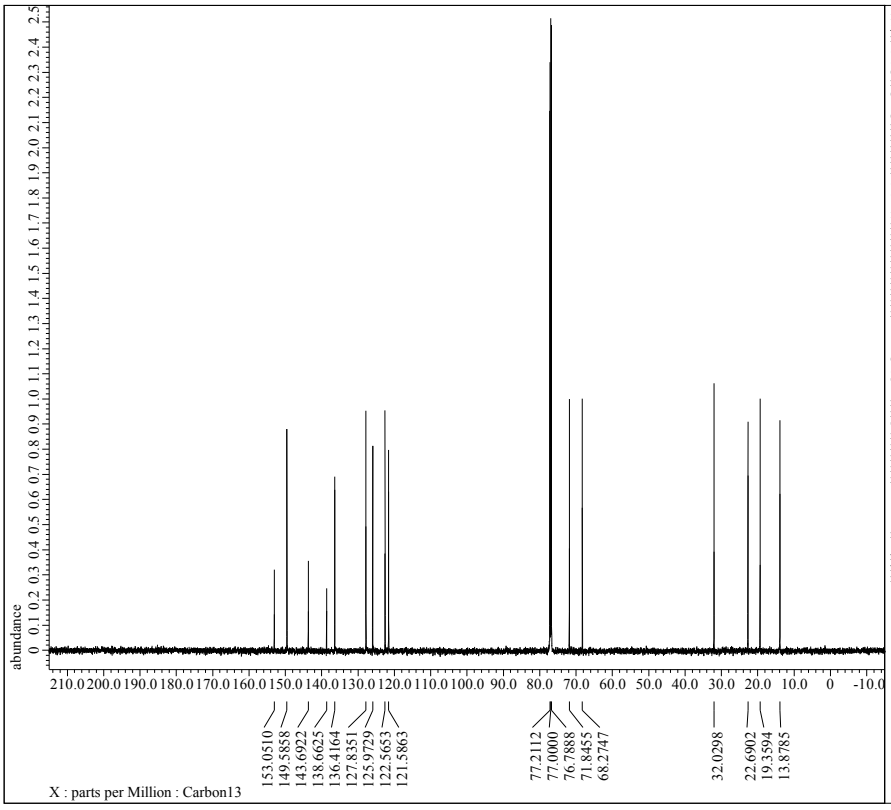

abundance
X : parts per Million : Carbon13
159.7222
149.1731
139.7183
139.3152
136.4931
135.9940
129.8892
129.5820
126.0113
124.1299
121.6535
77.2112
77.0000
76.7888
73.3045
68.0923
32.0394
23.9092
21.0200
19.3402
13.9073
Filename = ey9087p2_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9087p2
Solvent = CHLOROFORM-D
Creation_Time = 12-NOV-2014 17:59:52
Revision_Time = 12-NOV-2014 18:13:26
Current_Time = 6-FEB-2015 15:30:40
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 = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 25.5[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-BuO
N
3ea



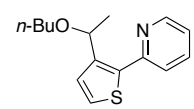

```
Filename          = ey9118p1.2_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9118p1.2
Solvent           = CHLOROFORM-D
Creation_Time     =  8-DEC-2014 20:22:49
Revision_Time     =  6-FEB-2015 15:32:07
Current_Time      =  6-FEB-2015 15:32:15

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 = 5[s]
Recvr_Gain        = 36
Temp_Get          = 23.7[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   = 10.88251136[s]
```



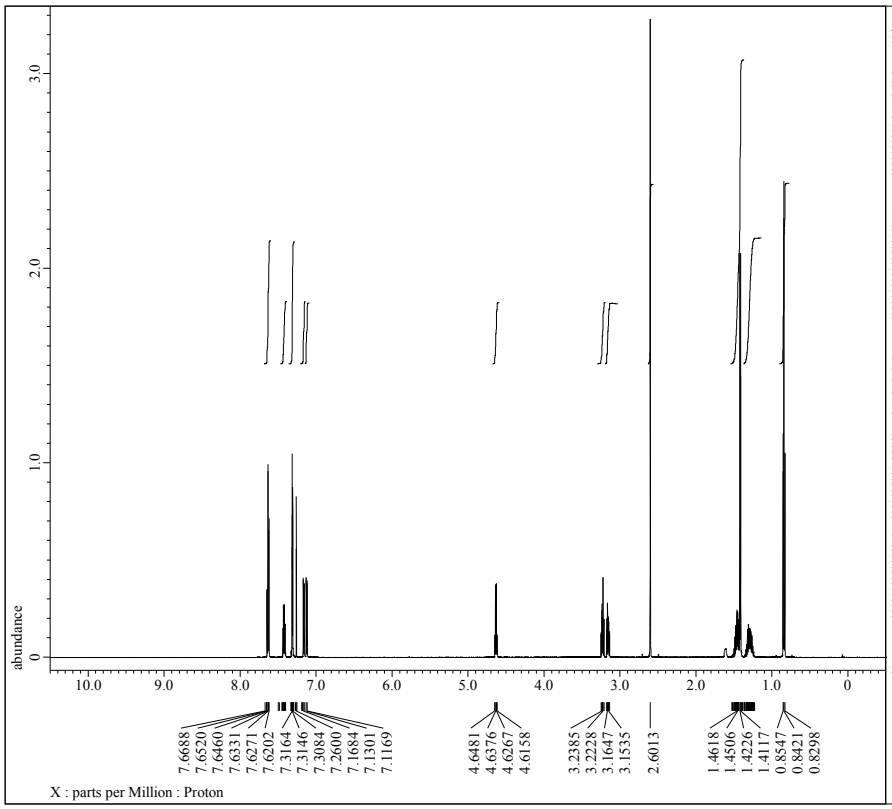
3fa



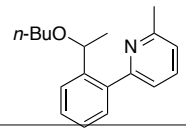

```
Filename          = ey9118p1.2_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey9118p1.2
Solvent           = CHLOROFORM-D
Creation_Time     =  8-DEC-2014 20:26:15
Revision_Time     =  8-DEC-2014 20:39:05
Current_Time      =  6-FEB-2015 15:32:47

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          = 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]
```



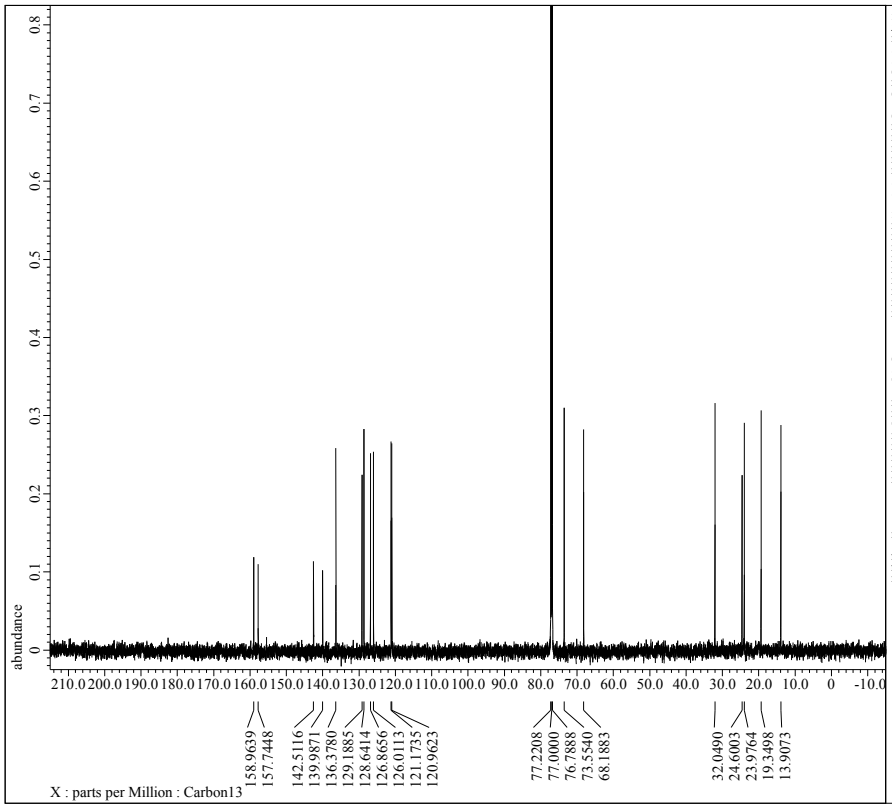
3fa



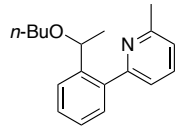

```
Filename          = ey9088p1_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9088p1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2014 17:01:35
Revision_Time     =  6-FEB-2015 15:35:02
Current_Time      =  6-FEB-2015 15:35:25

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  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 24.6[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_Attenuator  = 60[dB]
Dante_Interval    = 0.1[ms]
Dante_Loop        = 490
Dante_Offset      = 5[ppm]
Dante_Presat      = TRUE
Dante_Pulse       = 2[us]
Diff_Offset       = 0[MHz]
Initial_Wait      = 1[s]
Phs_Shft          = 0
Presat_Time       = 5[s]
Proton_Freq       = 594.17058168[MHz]
Repetition_Time   = 10.88251136[s]
```



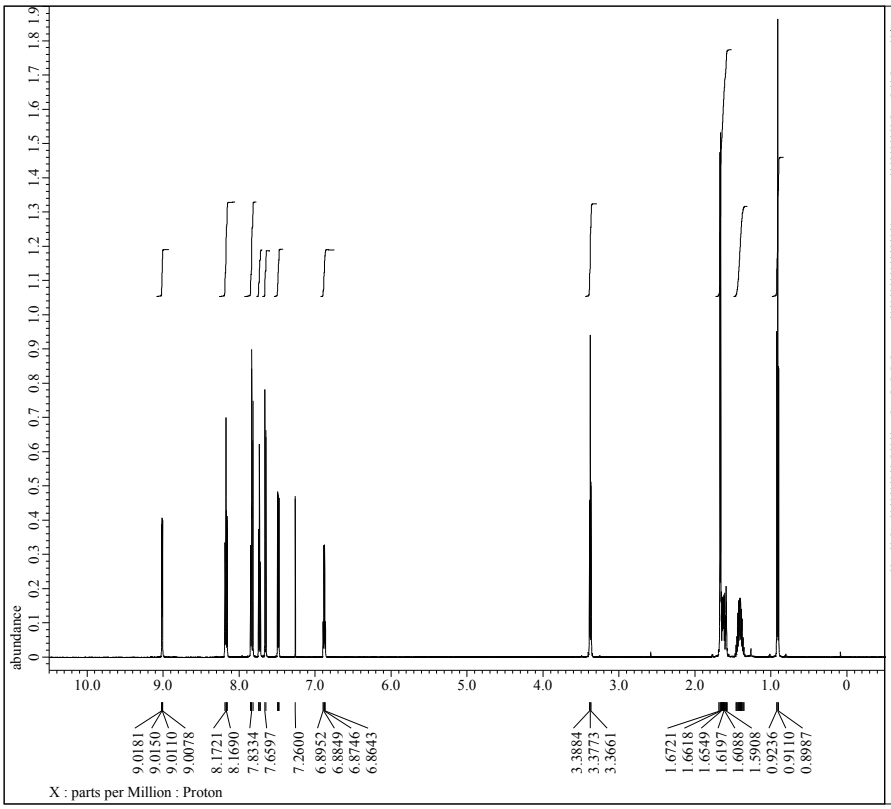
3ga



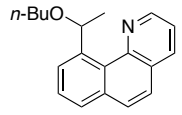

```
Filename          = ey9088p1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey9088p1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-NOV-2014 18:17:42
Revision_Time     = 12-NOV-2014 18:30:22
Current_Time      =  6-FEB-2015 15:35:56

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             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
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]
```



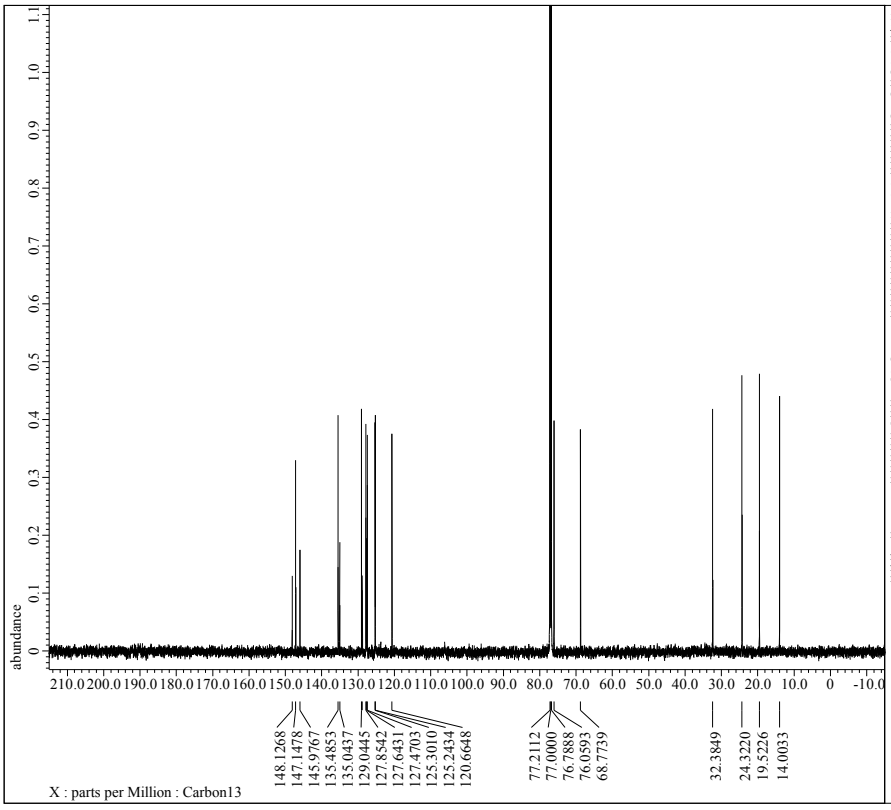
3ga



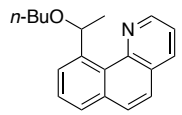

```
Filename          = ey9049p1_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9049p1
Solvent           = CHLOROFORM-D
Creation_Time     = 25-OCT-2014 17:49:00
Revision_Time     =  6-FEB-2015 15:36:49
Current_Time      =  6-FEB-2015 15:37:13

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  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 25[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   = 10.88251136[s]
```



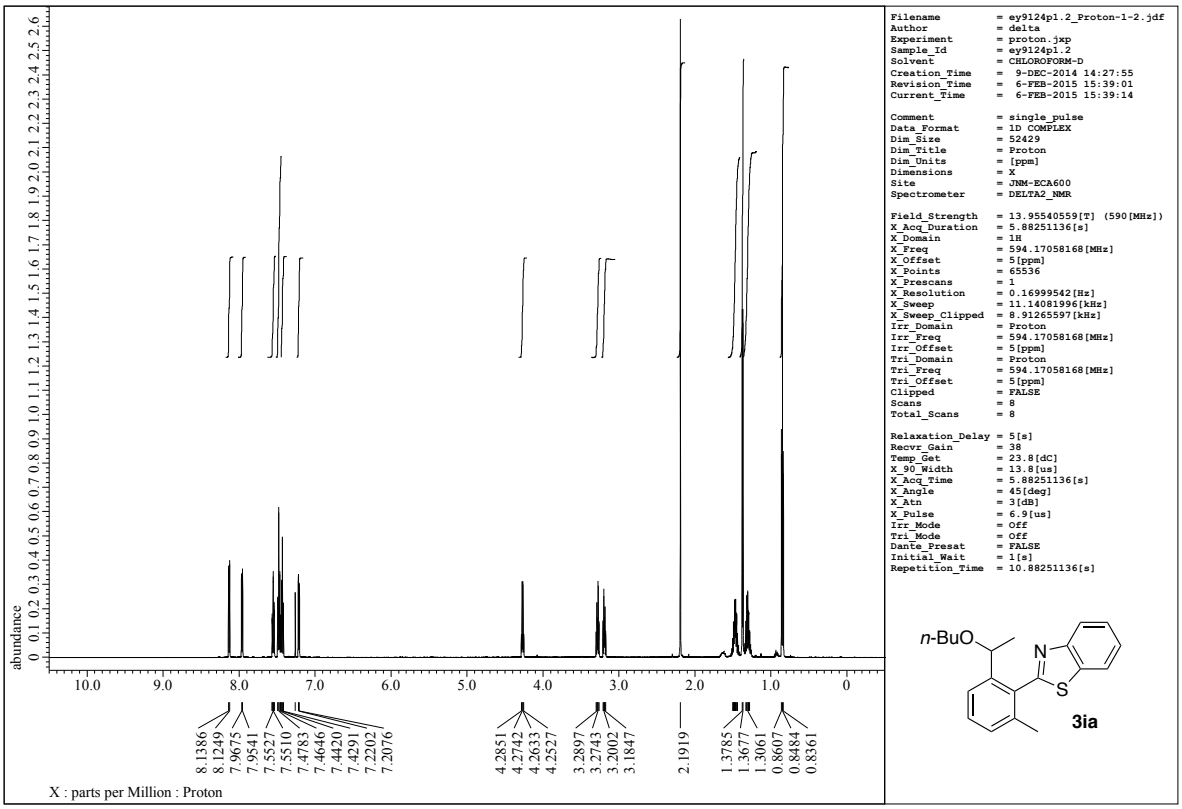
3ha

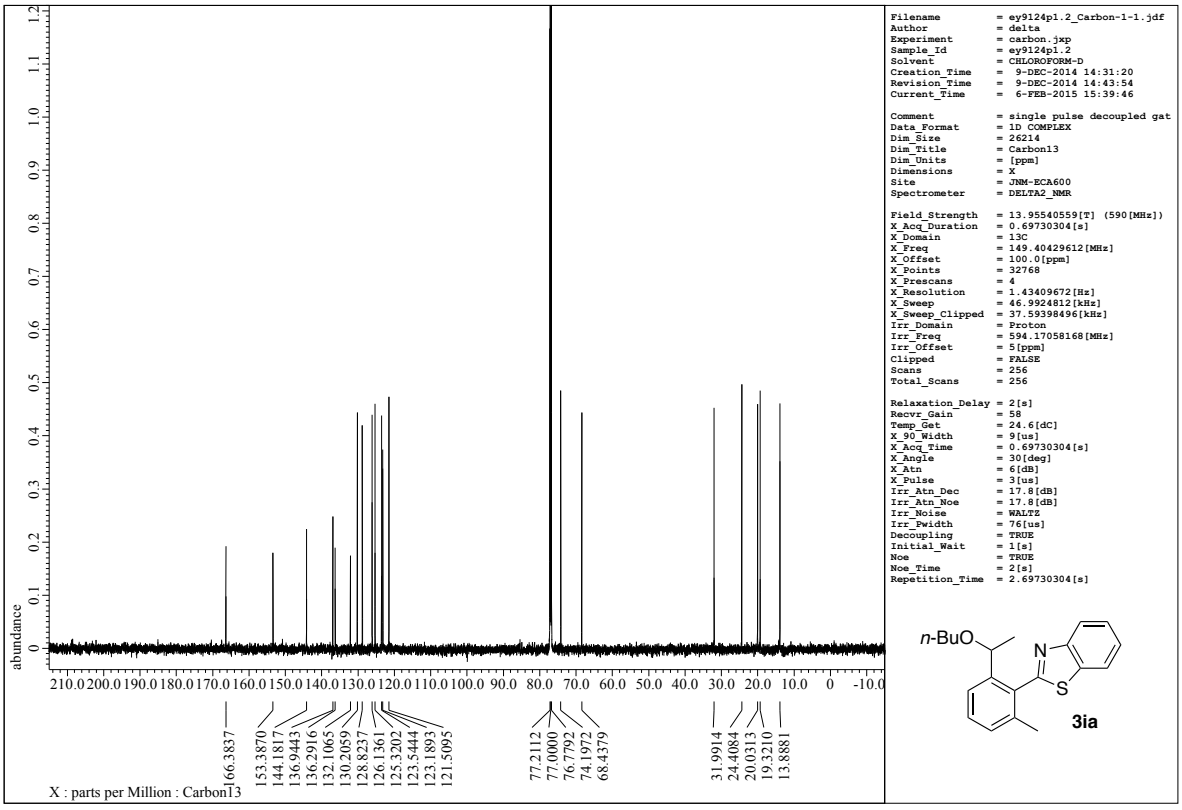


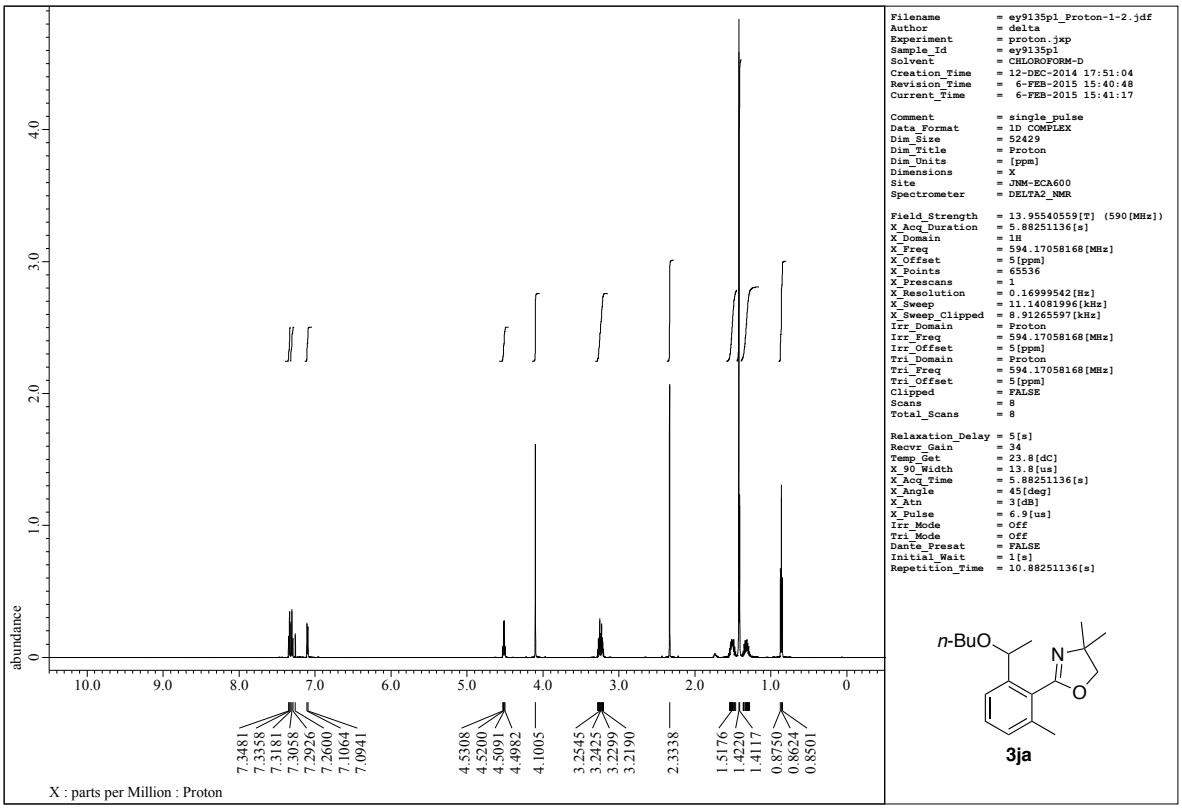

```
Filename          = ey9049p1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey9049p1
Solvent           = CHLOROFORM-D
Creation_Time     = 25-OCT-2014 17:52:45
Revision_Time     = 25-OCT-2014 18:05:19
Current_Time      =  6-FEB-2015 15:37:47

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          = 25.7[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]
```

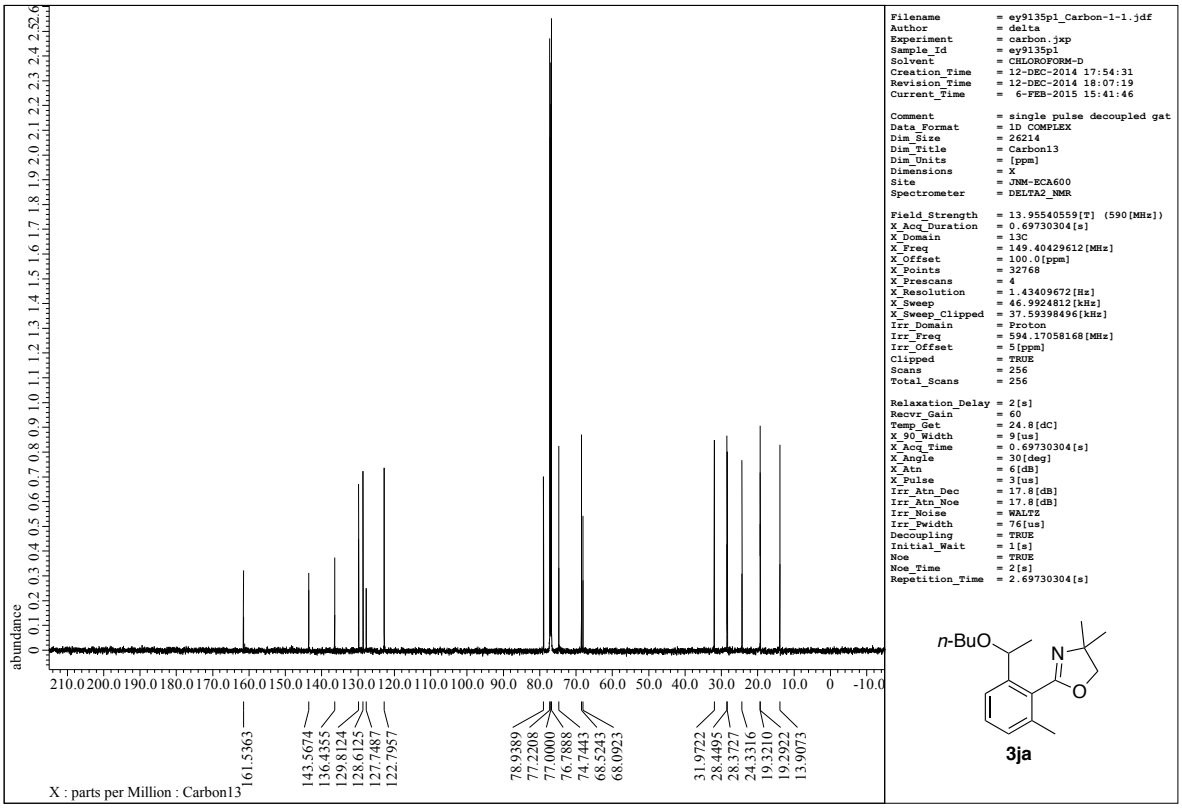


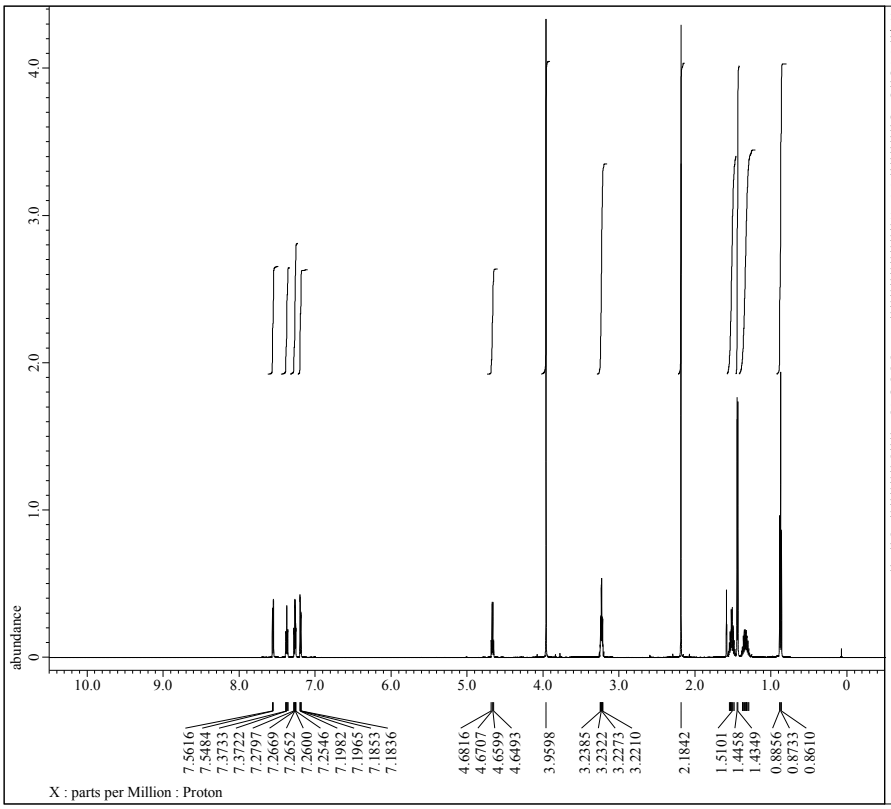
3ha


abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.1386
8.1249
7.9675
7.9541
7.5527
7.5510
7.4783
7.4646
7.4420
7.4291
7.2202
7.2076
4.2851
4.2742
4.2633
4.2527
3.2897
3.2743
3.2002
3.1847
2.1919
1.3785
1.3677
1.3061
0.8607
0.8484
0.8361
X : parts per Million : Proton
Filename = ey9124p1.2_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9124p1.2
Solvent = CHLOROFORM-D
Creation_Time = 9-DEC-2014 14:27:55
Revision_Time = 6-FEB-2015 15:39:01
Current_Time = 6-FEB-2015 15:39:14
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 = 5[s]
Recvr_Gain = 38
Temp_Get = 23.8[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 = 10.88251136[s]
n-BuO
N
S
3ia

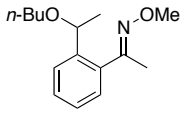

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
166.3837
153.3870
144.1817
136.9443
136.2916
132.1065
130.2059
128.8237
126.1361
125.3202
123.5444
123.1893
121.5095
77.2112
77.0000
76.7792
74.1972
68.4379
31.9914
24.4084
20.0313
19.3210
13.8881
X : parts per Million : Carbon13
Filename = ey9124p1.2_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9124p1.2
Solvent = CHLOROFORM-D
Creation_Time = 9-DEC-2014 14:31:20
Revision_Time = 9-DEC-2014 14:43:54
Current_Time = 6-FEB-2015 15:39:46
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 = 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]
n-BuO
N
S
3ia


abundance
X : parts per Million : Proton
7.3481
7.3358
7.3181
7.3058
7.2926
7.2600
7.1064
7.0941
4.5308
4.5200
4.5091
4.4982
4.1005
3.2545
3.2425
3.2299
3.2190
2.3338
1.5176
1.4220
1.4117
0.8750
0.8624
0.8501
Filename = ey9135p1_Proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = ey9135p1
Solvent = CHLOROFORM-D
Creation_Time = 12-DEC-2014 17:51:04
Revision_Time = 6-FEB-2015 15:40:48
Current_Time = 6-FEB-2015 15:41:17
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 = 5[s]
Recvr_Gain = 34
Temp_Get = 23.8[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 = 10.88251136[s]
n-BuO
N
O
3ja

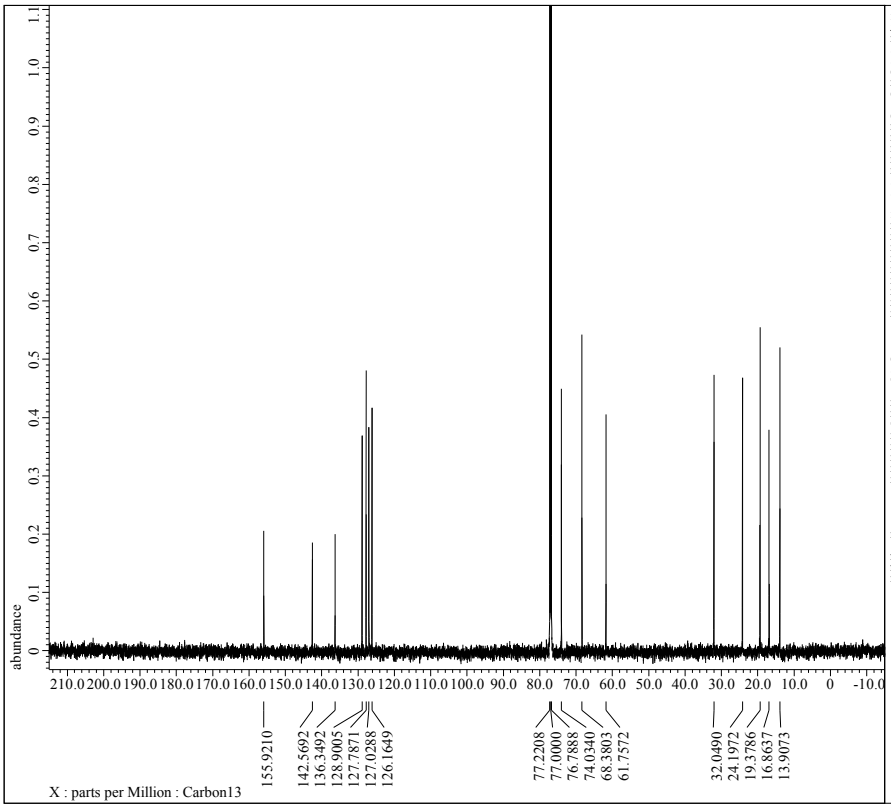

abundance
X : parts per Million : Carbon13
161.5363
143.5674
136.4355
129.8124
128.6125
127.7487
122.7957
78.9389
77.2208
77.0000
76.7888
74.7443
68.5243
68.0923
31.9722
28.4495
28.3727
24.3316
19.3210
19.2922
13.9073
Filename = ey9135p1_Carbon-1-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = ey9135p1
Solvent = CHLOROFORM-D
Creation_Time = 12-DEC-2014 17:54:31
Revision_Time = 12-DEC-2014 18:07:19
Current_Time = 6-FEB-2015 15:41:46
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 = 24.8[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]
n-BuO
N
O
3ja



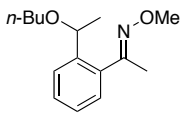

```
Filename          = ey9104p1_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9104p1
Solvent           = CHLOROFORM-D
Creation_Time     = 29-NOV-2014 11:52:44
Revision_Time     =  6-FEB-2015 15:45:05
Current_Time      =  6-FEB-2015 15:45:35

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  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 24.7[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   = 10.88251136[s]
```



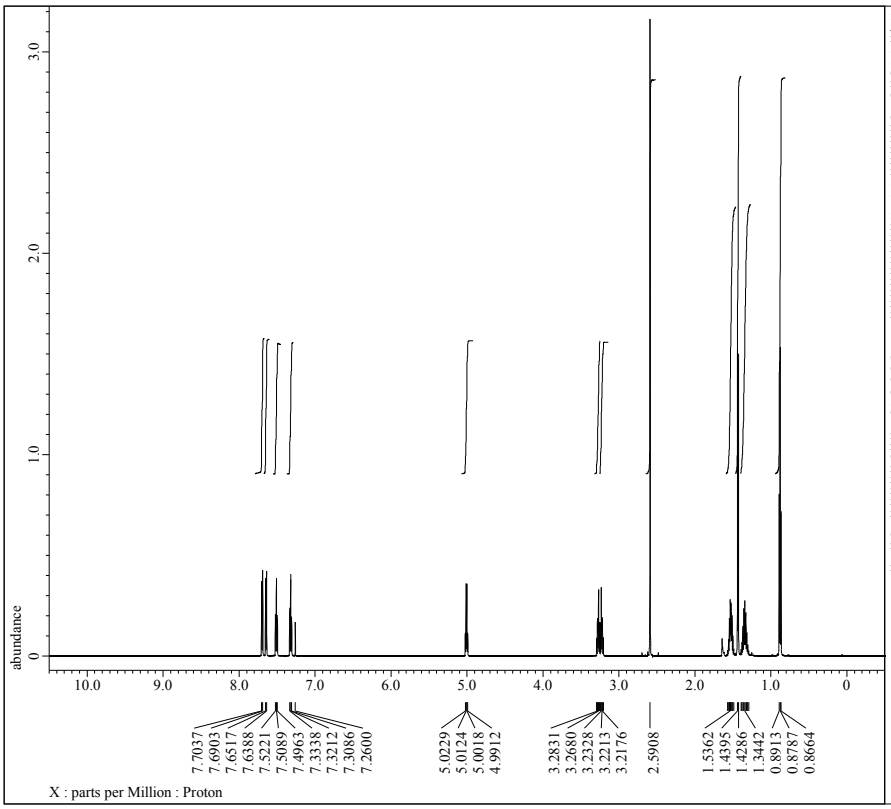
3ka



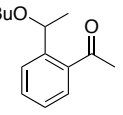

```
Filename          = ey9104p1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey9104p1
Solvent           = CHLOROFORM-D
Creation_Time     = 29-NOV-2014 11:56:06
Revision_Time     = 29-NOV-2014 12:12:08
Current_Time      =  6-FEB-2015 15:46:03

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        = 60
Temp_Get          = 25.5[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]
```



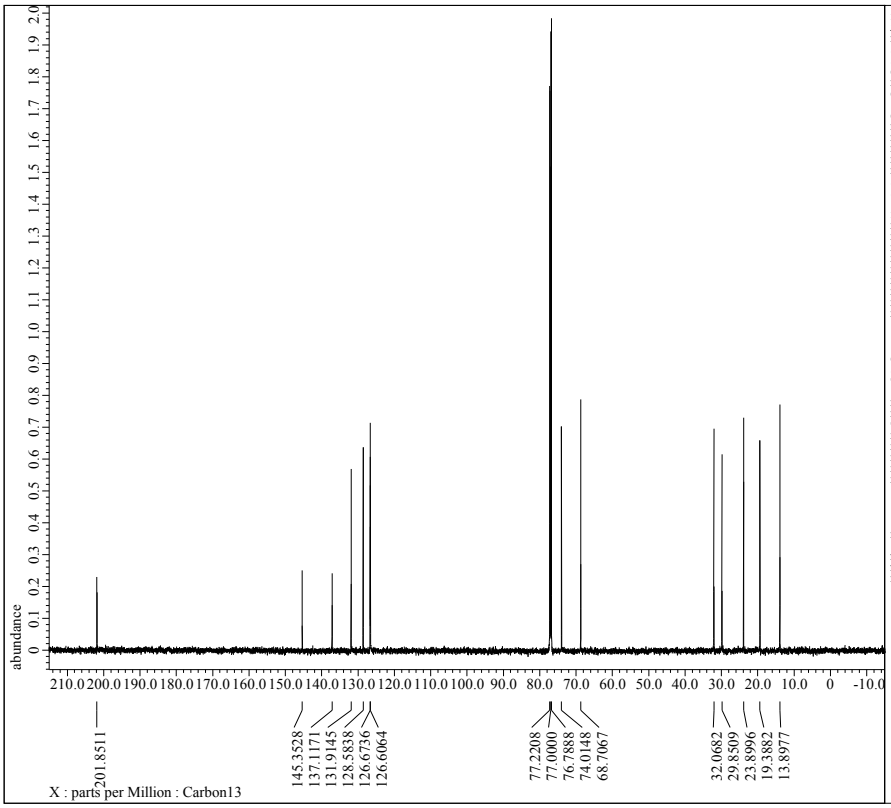
3ka



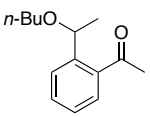

```
Filename          = ey9127.2p1_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey9127.2p1
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2014 22:35:16
Revision_Time     =  6-FEB-2015 15:46:41
Current_Time      =  6-FEB-2015 15:47:03

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  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 23.8[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   = 10.88251136[s]
```



3la

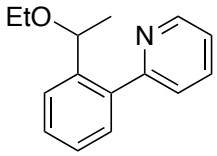



```
Filename          = ey9127.2p1_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey9127.2p1
Solvent           = CHLOROFORM-D
Creation_Time     = 10-DEC-2014 22:38:42
Revision_Time     = 10-DEC-2014 22:51:20
Current_Time      =  6-FEB-2015 15:47:31

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    = 0.69730304[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          = 24.7[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]
```

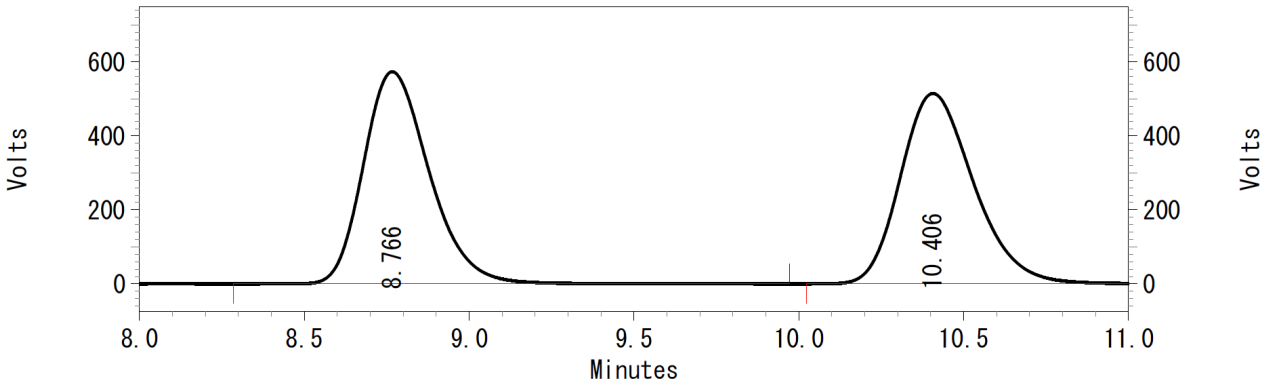


3la

12. Chiral HPLC charts

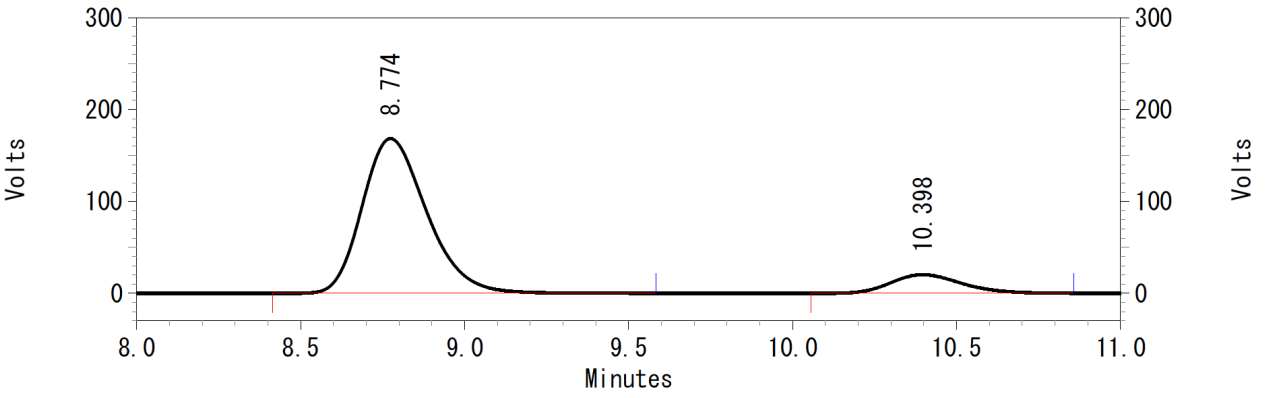


3ac



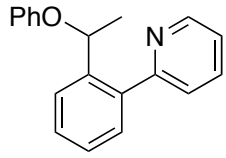
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	8.766	7978314	50.002	573827
2	10.406	7977736	49.998	514746
Totals		15956050	100.000	1088573

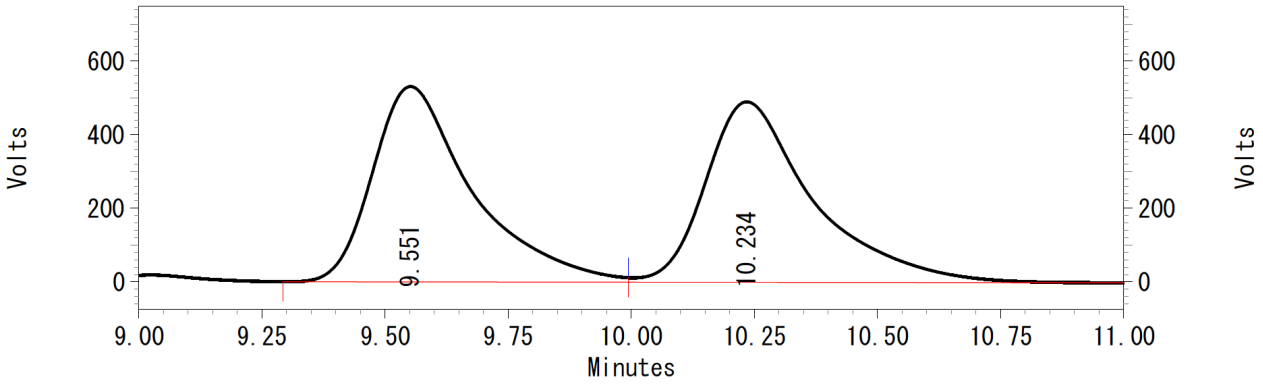


UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	8.774	2305665	88.275	168376
2	10.398	306241	11.725	20308
Totals		2611906	100.000	188684

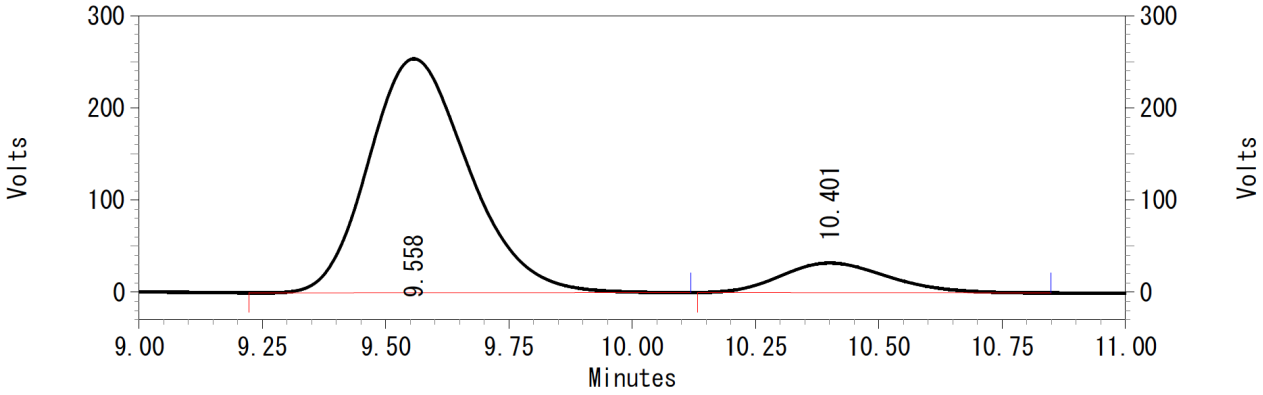


3af



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	9.551	7469909	50.045	531016
2	10.234	7456509	49.955	490572
Totals		14926418	100.000	1021588



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	9.558	3582243	88.005	253900
2	10.401	488274	11.995	32205
Totals		4070517	100.000	286105