Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Iridium-catalyzed enantioselective addition of an *N*-methyl C–H bond to α-trifluoromethylstyrenes via C–H activation

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Contents of Supplementary Information:

1. General method

All manipulations of oxygen- and moisture-sensitive materials were carried out using standard Schlenk techniques under a nitrogen atmosphere. NMR spectra were recorded on a JEOL JNM ECZ-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C) or Bruker DMX NMR spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, 376 MHz for ^{19}F). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) for ^{1}H NMR, $CDCl_3$ (δ 77.00) for ^{13}C NMR, and CF_3CO_2H as an external standard (δ –76.0) for ^{19}F NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: sext, sextet: m, multiplet: br, broad. High-resolution mass spectra were obtained with JEOL AccuTOF LC-plus 4G spectrometer. Optical rotations were measured on JASCO P-2200 polarimeter. Flash column chromatography was performed with Silica Gel 70 PF_{254} (Wako). Preparative thin-layer chromatography was performed with Wakogel® B-5F (Wako).

2. Materials

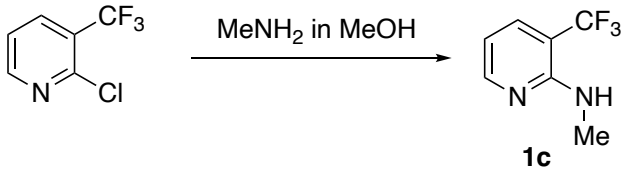
Dehydrated 1,2-dichloroethane, toluene, DMF, and 1,4-dioxane were purchased and used after deoxygenated by bubbling N_2. Iridium complexes, $[IrCl(cod)]_2$[1] and $[IrCl(coe)_2]_2$,[2] were prepared according to the reported procedures. $NaBAr^F{}_4$ was prepared according to the reported procedures.[3] Ligands, (*R*)-binap, (*R*)-tol-binap, (*R*)-DM-binap, (*S*)-segphos, (*S*,*S*)-QuinoxP*, (*S*,*S*)-chiraphos, were purchased from commercial suppliers and used as received. The corresponding racemic products were prepared by using racemic binap as a ligand. Other chemicals were purchased from commercial suppliers and used as received.

3. Preparation of methylamine derivatives 1 and alkenes 2

Compound **1g** (CAS: 4597-87-9) was purchased from commercial suppliers and used as received. Compounds **1a** (CAS: 103976-61-0),[4] **1b** (CAS: 468718-67-4),[5] **1e** (CAS: 902837-10-9),[6] and **1f** (CAS: 156267-13-9)[5] were prepared according to the reported procedures.

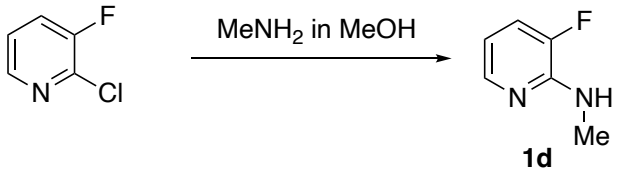
Alkenes **2x** was purchased from commercial suppliers and used as received. **2a** (CAS: 384-64-5),[7] **2b** (CAS: 69843-09-0),[7] **2c** (CAS: 185140-55-0),[7] **2d** (CAS: 69843-08-9),[7] **2e** (CAS: 1601477-35-3),[7] **2f** (CAS: 655-29-8),[7] **2g** (CAS: 69843-10-3),[7] **2h** (CAS: 155855-37-1),[7] **2i** (CAS: 946614-13-7),[7] **2j** (CAS: 2369617-16-1),[7] **2k** (CAS: 1261137-92-1),[7] **2l** (CAS: 1989676-91-6),[7] **2m** (CAS: 437552-55-1),[7] **2n** (CAS: 185140-57-2),[7] **2o** (CAS: 2358848-30-1),[8] **2p** (CAS: 136476-29-4),[7] **2q** (CAS: 1422521-62-7),[7] **2r** (CAS: 1541177-26-7),[7] **2s** (CAS: 2071669-70-8),[7] **2t** (CAS: 437552-58-4),[9] **2u** (CAS: 112298-41-6),[9] **2v** (CAS: 1808992-30-4),[10] **2w** (CAS: 1352954-05-2),[11] **2y** (CAS: 17498-71-4),[12] and **2z** (CAS: 1865-29-8)[13] were prepared according to the reported procedure.

Procedures for the preparation of compounds **1c** (CAS: 1036584-14-1), **1d** (CAS: 220714-69-2), **1h** (CAS: 4214-80-6),[14] **1i** (CAS: 1882650-38-5), **2n** (CAS: 185140-57-2), **2n-*d*$_2$**, and **2r-*d*$_2$** were shown below.

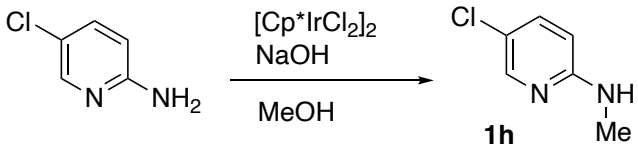


1c

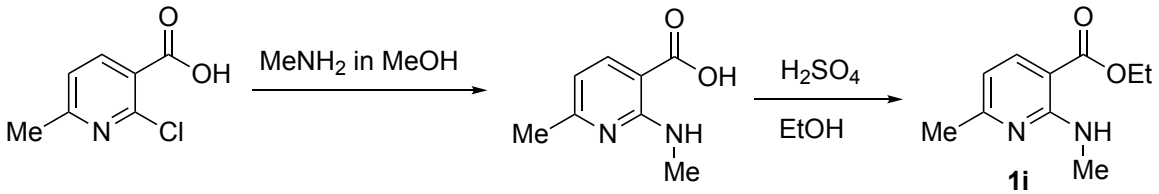
Compound 1c: To a 2-chloro-3-trifluoromethylpyridine (3.63 g, 20 mmol) in a pressure bottle with a Teflon valve was added $MeNH_2$ (9.8 M in MeOH, 20 mL, 200 mmol). The mixture was heated at 80 °C for 3 days with stirring. The mixture was concentrated on a rotary evaporator, and the residue was subjected to flash column chromatography on silica gel (hexane: EtOAc = 10:1) to give **1c** as a colorless oil (2.46 g, 70% yield). ^{1}H NMR ($CDCl_3$) δ 8.29 (d, J = 4.8 Hz, 1H), 7.63 (d, J = 6.8 Hz, 1H), 6.60 (dd, J = 6.8, 4.8 Hz, 1H), 4.96–4.83 (br, 1H), 3.04 (d, J = 5.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 155.2, 151.6, 134.8 (d, $J_{F\text{-}C}$ = 5 Hz), 124.5 (q, $J_{F\text{-}C}$ = 271 Hz), 111.0, 108.6 (q, $J_{F\text{-}C}$ = 32 Hz), 28.5; ^{19}F NMR ($CDCl_3$) δ –64.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_7H_8N_2F_3$ 177.0640; Found 177.0628.



Compound 1d: 2-Chloro-3-fluoropyridine (575 mg, 5.0 mmol) in a pressure bottle with a Teflon valve was added $MeNH_2$ (9.8 M in MeOH, 5.1 mL, 50 mmol). The mixture was heated at 80 °C for 3 days with stirring. The mixture was concentrated on a rotary evaporator, and the residue was subjected to flash column chromatography on silica gel (hexane: EtOAc = 2:1) to give **1d** as a colorless oil (318 mg, 50% yield). ^{1}H NMR ($CDCl_3$) δ 7.91 (d, J = 4.8 Hz, 1H), 7.11 (dd, J = 11.4, 7.8 Hz, 1H), 6.54–6.46 (m, 1H), 4.74–4.54 (br, 1H), 3.05 (d, J = 4.4 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 149.3 (d, $J_{F\text{-}C}$ = 11 Hz), 147.3 (d, $J_{F\text{-}C}$ = 252 Hz), 142.7 (d, $J_{F\text{-}C}$ = 7 Hz), 119.6 (d, $J_{F\text{-}C}$ = 15 Hz), 111.4, 27.8; ^{19}F NMR ($CDCl_3$) δ –141.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_6H_8N_2F_1$ 127.0672; Found 127.0665.

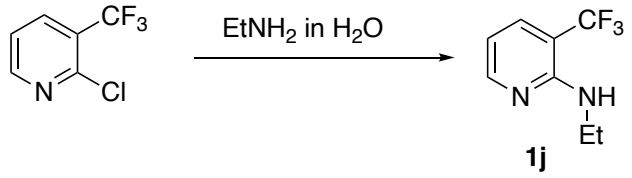


Compound 1h (CAS: 4214-80-6): A mixture of 2-amino-5-chloropyridine (257 mg, 2.0 mmol), methanol (1 mL), $[Cp^*IrCl_2]_2$ (1.6 mg, 0.0020 mmol), and NaOH (80 mg, 2.0 mmol) in a pressure bottle with a Teflon valve was heated at 150 °C for 12 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to flash column chromatography on silica gel (hexane: EtOAc = 3:1) to give **1h** as a colorless solid (223 mg, 78% yield). ^{1}H NMR ($CDCl_3$) δ 8.03 (d, J = 2.4 Hz, 1H), 7.38 (dd, J = 8.8, 2.4 Hz, 1H), 6.33 (d, J = 8.8 Hz, 1H), 4.63–4.49 (br, 1H), 2.90 (d, J = 4.8 Hz, 3H).



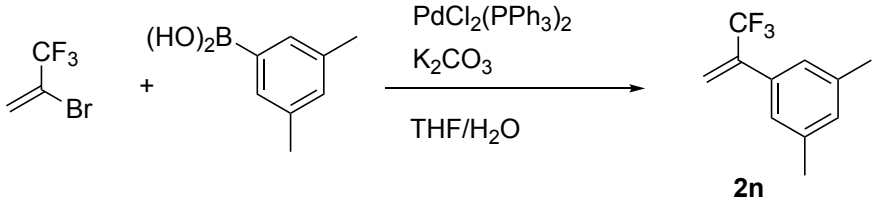
Compound 1i: To 2-chloro-6-methylnicotinic acid (858 mg, 5.0 mmol) in a pressure bottle with a Teflon valve was added $MeNH_2$ (9.8 M in MeOH, 5.1 mL, 50 mmol). The mixture was heated at 80 °C for 2 days with stirring, and the mixture was concentrated on a rotary evaporator. To the residue in EtOH (20 ml) was added conc. H_2SO_4 (1.4 mL) at room temperature. The mixture was stirred under reflux for 3 days. The mixture was poured into cold Na_2CO_3aq and extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a

rotary evaporator. The residue was subjected to flash column chromatography on silica gel (hexane: EtOAc = 4:1) to give **1i** as a colorless solid (490 mg, 51% yield in 2 steps). ^{1}H NMR ($CDCl_3$) δ 7.98 (d, J = 8.4 Hz, 1H), 7.94–7.66 (br, 1H), 6.36 (d, J = 8.4 Hz, 1H), 4.29 (q, J = 7.2 Hz, 2H), 3.05 (d, J = 5.2 Hz, 3H), 2.42 (s, 3H), 1.36 (t, J = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.7, 163.4, 159.0, 139.9, 110.2, 103.1, 60.3, 27.6, 25.1, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{10}H_{15}N_2O_2$ 195.1134; Found 195.1132.



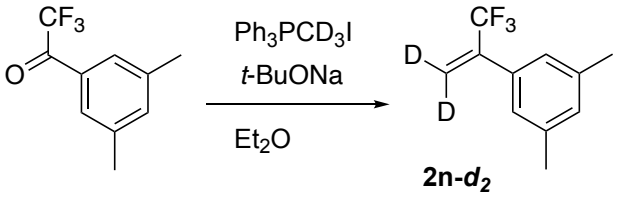
1j

Compound 1j: To a 2-chloro-3-trifluoromethylpyridine (908 mg, 5.0 mmol) in a pressure bottle with a Teflon valve was added $EtNH_2$ (70% in H_2O, 3.2 mL, 50 mmol). The mixture was heated at 80 °C for 2 days with stirring. The mixture was concentrated on a rotary evaporator, and the residue was subjected to column chromatography on silica gel eluted with CH_2Cl_2 to give **1j** as a colorless oil (706 mg, 74% yield). ^{1}H NMR ($CDCl_3$) δ 8.26 (dd, J = 5.1, 1.2 Hz, 1H), 7.63 (dd, J = 6.8, 1.2 Hz, 1H), 6.59 (dd, J = 6.8, 5.1 Hz, 1H), 4.81 (br s, 1H), 3.52 (qd, J = 7.3, 5.1 Hz, 2H), 1.26 (t, J = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 154.6, 151.7, 134.9 (d, J_{F-C} = 5 Hz), 124.5 (q, J_{F-C} = 272 Hz), 111.0, 108.4 (q, J_{F-C} = 31 Hz), 36.3, 14.7; ^{19}F NMR ($CDCl_3$) δ –64.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_8H_{10}F_3N_2$ 191.0796; Found 191.0785.



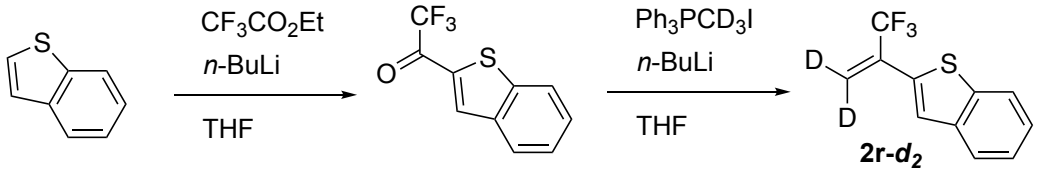
2n

Compound 2n (CAS: 185140-57-2): (3,5-Dimethylphenyl)boronic acid (1.13g, 7.5 mmol) and $PdCl_2(PPh_3)_2$ (158 mg, 3 mol%) were placed in a Schlenk tube under N_2. 2-Bromo-3,3,3-trifluoroprop-1-ene (2.62 g, 15 mmol) in THF (15 mL) and K_2CO_3 (4.14 g, 30 mmol) in H_2O (10 mL) were added to the tube successively, and the mixture was stirred at 60 °C for 20 h. The reaction mixture was cooled to rt and filtered through a pad of celite with Et_2O. The organic layer was separated and washed with brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with hexane to give **2n** as a colorless oil (725 mg, 48% yield). ^{1}H NMR ($CDCl_3$) δ 7.08 (s, 2H), 7.04 (s, 1H), 5.92 (d, J = 1.2 Hz, 1H), 5.74 (d, J = 1.2 Hz, 1H), 2.35 (s, 6H).



2n-d_2

Compound 2n-d_2: To a solution of Ph_3PCD_3I (CAS: 1560-56-1, 449 mg, 1.1 mmol)[15] in Et_2O (5 mL) was added sodium *tert*-butoxide (106 mg, 1.1 mmol) at 0 °C, and the mixture was stirred at room temperature for 30 min. After cooling the mixture to –78 °C, 1-(3,5-dimethylphenyl)-2,2,2-trifluoroethan-1-one (202 mg, 1.0 mmol)[16] was added to the mixture. The reaction mixture was allowed to warm to room temperature and stirred for 20 h. The mixture was filtered and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with hexane to give **2n-d_2** as a colorless oil (97 mg, 48% yield). Deuterium contents of **2n-d_2** was determined by ^{1}H NMR (1.89D/2H). ^{1}H NMR ($CDCl_3$) δ 7.08 (s, 2H), 7.04 (s, 1H), 5.69–5.58 (m, 0.11H), 2.35 (s, 6H).



2r-d_2

Compound 2r-d_2: To a solution of benzothiophene (1.34 g, 10 mmol) in THF (20 mL) was added dropwise *n*-BuLi (1.6 M in THF, 7.5 ml, 12 mmol) at –78 °C and the mixture was stirred at the same temperature for 90 min. Then, ethyl trifluoroacetate (2.13 g, 15 mmol) was added to the mixture, and the resulting mixture was allowed to warm to room temperature and stirred for 2 h. Saturated NH_4Cl solution was added to the mixture at 0 ºC, and the resulting mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel (hexane: EtOAc = 40:1) to give 2-benzothienyl trifluoromethyl ketone (1.51 g, 66% yield). To a solution of Ph_3PCD_3I (CAS: 1560-56-1, 470 mg, 1.15 mmol)[15] in THF (5 mL) was added *n*-BuLi (0.73 ml, 1.6 M in THF, 1.2 mmol) at –78 °C, and the mixture was stirred at the same temperature for 90 min. Then, 2-benzothienyl trifluoromethyl ketone (265 mg, 1.15 mmol) was added to the mixture, and the resulting mixture was allowed to warm to room temperature and stirred for 2 h. Saturated NH_4Cl solution was added to the mixture at 0 ºC, and the resulting mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with hexane to give **2r-d_2** as a colorless oil (1.11 g, 73% yield). Deuterium contents of **2r-d_2** was determined by ^{1}H NMR (1.96D / 2H). ^{1}H NMR ($CDCl_3$) δ 7.82–7.74 (m, 2H), 7.28 (d, J = 0.8 Hz, 1H), 7.40–7.34 (m, 2H), 5.95–5.90 (m, 0.04H).

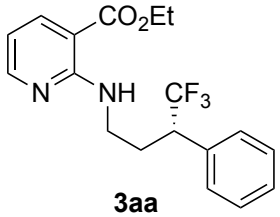
4. General procedure for Table 1

3-Ethoxycarbonyl-2-(methylamino)pyridine (**1a**: 18.0 mg, 0.10 mmol), $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% of Ir), a ligand (0.0060 mmol, 6 mol%), and $NaBAr^F_4$ (9.2 mg, 0.010 mmol, 10 mol%) were placed in a Schlenk tube under N_2. Toluene (0.4 mL) and α-trifluoromethylstyrene (**2a**, 16.1 mg, 0.30 mmol) were added to the mixture successively, and the mixture was stirred at 80 °C for 48 h. The mixture was concentrated on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel (hexane/$CHCl_3$ = 2:3) to give **3aa**.

5. General procedure for Schemes 2 and 3

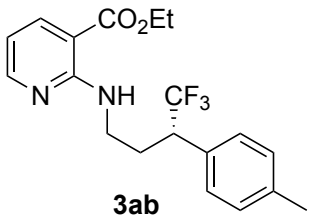
Methylamine derivatives **1** (0.10 mmol), $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% of Ir), (*R*)-tol-binap (0.0060 mmol, 6 mol%), and $NaBAr^F_4$ (9.2 mg, 0.010 mmol, 10 mol%) were placed in a Schlenk tube under N_2. Toluene (0.4 mL) and alkene **2** (0.30 mmol) were added to the mixture successively, and the mixture was stirred at 80 °C for 48 h. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel to give **3**. The absolute configuration of **3aa** was determined to be (*S*)-(+) by correlation with compound **5** (see, S-25). The absolute configurations of other compounds were assigned by analogy with (*S*)-**3aa**.

6. Characterization of the products



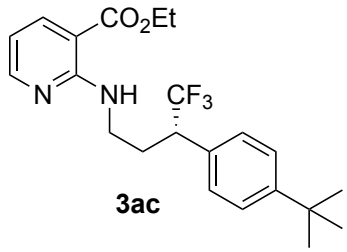
3aa

Compound 3aa (Table 1, entry 2; colorless oil, 33.0 mg, 94% yield, 88% ee; entry 11, 731 mg, 99% yield, 88% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 15.6 min (major), t_2 = 17.8 min (minor)); $[\alpha]^{20}_D$ +67.5 (*c* 1.08, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 6.0, 1.8 Hz, 1H), 8.10 (dd, *J* = 11.6, 1.8 Hz, 1H), 8.20–7.90 (br, 1H), 7.39–7.30 (m, 5H), 6.52 (dd, *J* = 11.6, 6.0 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.56–3.45 (m, 1H), 3.45–3.28 (m, 2H), 2.49–2.39 (m, 1H), 2.24–2.14 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 139.9, 134.2, 129.1, 128.7, 128.2, 126.9 (q, *J* = 280 Hz), 111.1, 106.2, 60.8, 47.8 (q, *J* = 27 Hz), 38.0, 28.6, 14.3; ^{19}F NMR ($CDCl_3$) δ –69.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{20}F_3N_2O_2$ 353.1477; Found 353.1478.



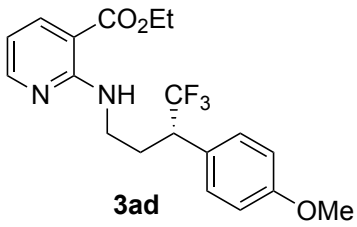
3ab

Compound 3ab (Scheme 2; colorless oil, 35.6 mg, 97% yield, 91% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IC, hexane/EtOH = 500:1, flow 0.5 mL/min, 254 nm, t_1 = 19.9 min (minor), t_2 = 21.4 min (major)); $[\alpha]^{20}_D$ +79.6 (*c* 0.98, $CHCl_3$) for 91% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.6, 1.8 Hz, 1H), 8.10 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.98–7.92 (br, 1H), 7.22 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 6.51 (dd, *J* = 7.8, 4.6 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.56–3.45 (m, 1H), 3.44–3.29 (m, 2H), 2.46–2.37 (m, 1H), 2.35 (s, 3H), 2.22–2.11 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 139.9, 138.0, 131.1, 129.4, 128.9, 127.0 (q, *J* = 279 Hz), 111.1, 106.2, 60.8, 47.8 (q, *J* = 27 Hz), 38.0, 28.6, 21.1, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{22}F_3N_2O_2$ 367.1633; Found 367.1640.



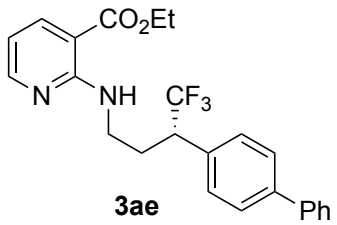
3ac

Compound 3ac (Scheme 2; colorless oil, 37.5 mg, 92% yield, 87% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 10.4 min (minor), t_2 = 11.1 min (major)); $[\alpha]^{20}_D$ +74.8 (*c* 0.99, $CHCl_3$) for 87% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.4, 2.0 Hz, 1H), 8.20–7.93 (br, 1H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.51 (dd, *J* = 7.4, 4.8 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.57–3.46 (m, 1H), 3.44–3.29 (m, 2H), 2.48–2.38 (m, 1H), 2.23–2.12 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H), 1.32 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 151.0, 139.9, 131.1, 128.7, 127.0 (q, *J* = 285 Hz), 125.6, 111.0, 106.2, 60.7, 47.3 (q, *J* = 27 Hz), 38.1, 34.5, 31.3, 28.6, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{28}F_3N_2O_2$ 409.2103; Found 409.2099.



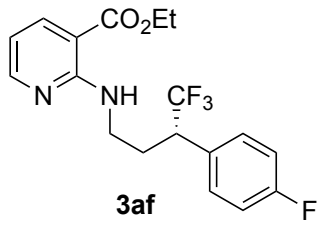
3ad

Compound 3ad (Scheme 2; colorless oil, 34.5 mg, 90% yield, 81% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 14.3 min (major), t_2 = 16.5 min (minor)); $[\alpha]^{20}_D$ +77.7 (*c* 0.87, $CHCl_3$) for 81% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.8, 1.6 Hz, 1H), 8.11 (dd, *J* = 7.4, 1.6 Hz, 1H), 8.03–7.90 (br, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 6.89 (d, *J* = 8.0 Hz, 2H), 6.53 (dd, *J* = 7.4, 4.8 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.81 (s, 3H), 3.56–3.46 (m, 1H), 3.41–3.27 (m, 2H), 2.46–2.35 (m, 1H), 2.20–2.08 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 159.4, 158.4, 153.3, 139.9, 130.1, 127.2 (q, *J* = 237 Hz), 126.0, 114.1, 111.1, 106.2, 60.8, 55.2, 47.0 (q, *J* = 27 Hz), 38.0, 28.5, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{22}F_3N_2O_3$ 383.1583; Found 383.1579.



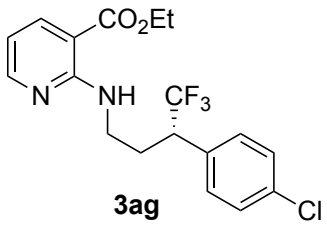
3ae

Compound 3ae (Scheme 2; pale yellow oil, 37.0 mg, 86% yield, 87% ee). A solution of hexane/EtOAc (4:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 18.7 min (minor), t_2 = 23.2 min (major)); $[\alpha]^{20}_D$ +84.7 (*c* 1.02, $CHCl_3$) for 87% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.24 (dd, *J* = 4.6, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.8, 2.0 Hz, 1H), 8.03–7.94 (br, 1H), 7.61–7.55 (m, 4H), 7.47–7.33 (m, 5H), 6.53 (dd, *J* = 7.8, 4.6 Hz, 1H), 4.30 (q, *J* = 7.2 Hz, 2H), 3.62–3.51 (m, 1H), 3.50–3.36 (m, 2H), 2.51–2.42 (m, 1H), 2.30–2.19 (m, 1H), 1.36 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 141.1, 140.5, 139.9, 133.2, 129.5, 128.8, 127.4, 127.1, 126.9 (q, *J* = 280 Hz), 111.1, 106.2, 60.8, 47.6 (q, *J* = 27 Hz), 38.1, 28.6, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{24}F_3N_2O_2$ 429.1790; Found 429.1801.



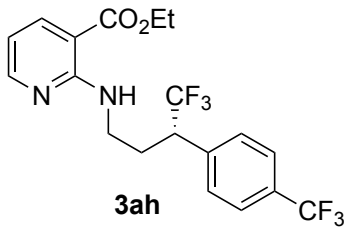
3af

Compound 3af (Scheme 2; colorless solid, 35.5 mg, 95% yield, 87% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 14.1 min (minor), t_2 = 16.9 min (major)); $[\alpha]^{20}_D$ +52.7 (*c* 0.98, $CHCl_3$) for 87% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 5.2, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.97–7.88 (br, 1H), 7.29 (dd, *J* = 8.4, 5.2 Hz, 2H), 7.04 (t, *J* = 8.4 Hz, 2H), 6.52 (dd, *J* = 7.6, 5.2 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.56–3.46 (m, 1H), 3.46–3.29 (m, 2H), 2.46–2.37 (m, 1H), 2.21–2.11 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 162.6 (d, *J* = 247 Hz), 158.4, 153.4, 139.9, 130.7 (d, *J* = 7.7 Hz), 130.0, 126.7 (q, *J* = 280 Hz), 115.7 (d, *J* = 22 Hz), 111.2, 106.2, 60.8, 47.2 (q, *J* = 27 Hz), 37.9, 28.6, 14.2; ^{19}F NMR ($CDCl_3$) δ –70.0, –114.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{19}F_4N_2O_2$ 371.1383; Found 371.1378.



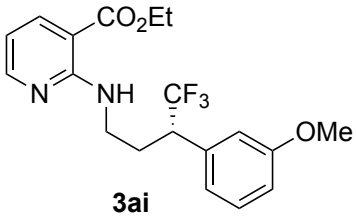
3ag

Compound 3ag (Scheme 2; colorless oil, 37.1 mg, 96% yield, 86% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 14.0 min (minor), t_2 = 16.2 min (major)); $[\alpha]^{20}_D$ +79.4 (*c* 1.07, $CHCl_3$) for 86% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 4.6, 2.2 Hz, 1H), 8.10 (dd, *J* = 7.8, 2.2 Hz, 1H), 7.96–7.85 (br, 1H), 7.32 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 6.52 (dd, *J* = 7.8, 4.6 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.57–3.47 (m, 1H), 3.46–3.30 (m, 2H), 2.46–2.36 (m, 1H), 2.23–2.12 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.4, 153.4, 139.9, 134.2, 132.7, 130.4, 128.9, 126.6 (q, *J* = 278 Hz), 111.2, 106.2, 60.8, 47.4 (q, *J* = 26 Hz), 37.9, 28.5, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{19}{}^{35}ClF_3N_2O_2$ 387.1087; Found 387.1081.



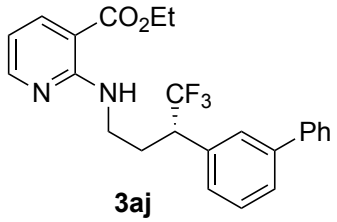
3ah

Compound 3ah (Scheme 2; colorless oil, 37.6 mg, 89% yield, 78% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 13.5 min (minor), t_2 = 16.7 min (major)); $[\alpha]^{20}_D$ +50.4 (*c* 1.05, $CHCl_3$) for 78% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.21 (dd, *J* = 4.6, 2.0 Hz, 1H), 8.09 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.99–7.90 (br, 1H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 6.53 (dd, *J* = 7.6, 4.6 Hz, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 3.58–3.45 (m, 2H), 3.43–3.32 (m, 1H), 2.49–2.40 (m, 1H), 2.30–2.19 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.3, 153.3, 140.0, 138.4, 130.5 (q, *J* = 33 Hz), 129.5, 126.6 (q, *J* = 265 Hz), 125.6, 123.9 (q, *J* = 256 Hz), 111.3, 106.3,. 60.8, 47.9 (q, *J* = 28 Hz), 37.9, 28.5, 14.2; ^{19}F NMR ($CDCl_3$) δ –62.7, –69.5. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{19}F_6N_2O_2$ 421.1351; Found 421.1345.



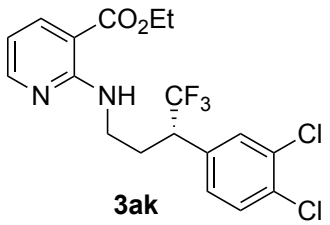
3ai

Compound 3ai (Scheme 2; colorless oil, 36.8 mg, 96% yield, 86% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 20.0 min (minor), t_2 = 21.7 min (major)); $[\alpha]^{20}_D$ +82.4 (*c* 0.37, $CHCl_3$) for 86% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 4.6, 2.2 Hz, 1H), 8.09 (dd, *J* = 7.6, 2.2 Hz, 1H), 8.01–7.91 (br, 1H), 7.26 (dd, *J* = 9.4, 6.0 Hz, 1H), 6.91 (d, *J* = 9.4 Hz, 1H), 6.86 (s, 1H), 6.85 (d, *J* = 6.0 Hz, 1H), 6.51 (dd, *J* = 7.6, 4.6 Hz, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 3.80 (s, 3H), 3.56–3.47 (m, 1H), 3.43–3.28 (m, 2H), 2.45–2.36 (m, 1H), 2.21–2.11 (m, 1H), 1.37 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 159.7, 158.5, 153.4, 139.9, 135.7, 129.7, 127.0 (q, *J* = 281 Hz), 121.4, 114.8, 113.6, 111.1, 106.2, 60.8, 55.2, 47.9 (q, *J* = 27 Hz), 38.0, 28.6, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{22}F_3N_2O_3$ 383.1583; Found 383.1589.



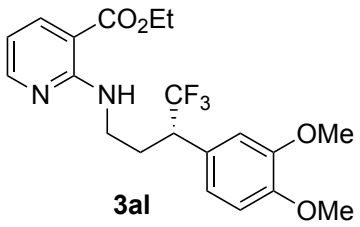
3aj

Compound 3aj (Scheme 2; colorless oil, 35.6 mg, 83% yield, 88% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 1.0 mL/min, 254 nm, t_1 = 9.1 min (major), t_2 = 17.9 min (minor)); $[\alpha]^{20}_D$ +79.4 (*c* 1.09, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 4.6, 1.8 Hz, 1H), 8.10 (dd, *J* = 7.8, 1.8 Hz, 1H), 8.06–7.98 (br, 1H), 7.61 (d, *J* = 7.8 Hz, 2H), 7.57 (d, *J* = 7.8 Hz, 2H), 7.48–7.42 (m, 3H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 1H), 6.51 (dd, *J* = 7.8, 4.6 Hz, 1H), 4.32 (q, *J* = 7.0 Hz, 2H), 3.64–3.54 (m, 1H), 3.54–3.46 (m, 1H), 3.45–3.34 (m, 1H), 2.53–2.42 (m, 1H), 2.34–2.22 (m, 1H), 1.38 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 141.7, 140.7, 139.9, 134.7, 129.1, 128.7, 128.1, 127.9, 127.4, 127.3, 127.1, 127.0 (q, *J* = 280 Hz), 111.1, 106.2, 60.8, 47.9 (q, *J* = 27 Hz), 38.0, 28.5, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{24}F_3N_2O_2$ 429.1790; Found 429.1787.



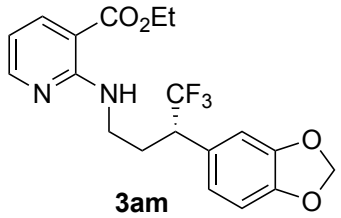
3ak

Compound 3ak (Scheme 2; pale yellow oil, 38.5 mg, 89% yield, 82% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-Hx2, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 36.0 min (minor), t_2 = 45.1 min (major)); $[\alpha]^{20}_D$ +52.2 (*c* 0.98, $CHCl_3$) for 82% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.09 (dd, *J* = 7.8, 2.0 Hz, 1H), 7.96–7.87 (br, 1H), 7.41 (s, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 6.53 (dd, *J* = 7.8, 4.8 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.57–3.48 (m, 1H), 3.45–3.32 (m, 2H), 2.43–2.33 (m, 1H), 2.25–2.14 (m, 1H), 1.38 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.4, 153.3, 139.9, 134.5, 132.8, 132.5, 131.1, 130.5, 128.5, 126.4 (q, *J* = 279 Hz), 111.4, 106.2, 60.8, 47.3 (q, *J* = 27 Hz), 37.8, 28.4, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}F_3N_2O_2Cl_2$ 421.0697; Found 421.0697.



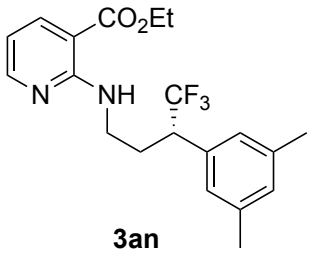
3al

Compound 3al (Scheme 2; colorless solid, 37.0 mg, 90% yield, 87% ee). A solution of hexane/EtOAc (4:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 17.9 min (major), t_2 = 22.9 min (minor)); $[\alpha]^{20}_D$ +80.9 (*c* 1.24, $CHCl_3$) for 87% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.10 (dd, *J* = 8.0, 2.0 Hz, 1H), 8.02–7.91 (br, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.82 (s, 1H), 6.52 (dd, *J* = 8.0, 4.8 Hz, 1H), 4.31 (q, *J* = 7.0 Hz, 2H), 3.88 (s, 3H), 3.87 (s, 3H), 3.61–3.50 (m, 1H), 3.41–3.27 (m, 2H), 2.45–2.34 (m, 1H), 2.20–2.09 (m, 1H), 1.38 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.4, 153.4, 149.0, 148.9, 139.9, 127.0 (q, *J* = 280 Hz), 126.5, 121.4, 111.9, 111.14, 111.05, 106.2, 60.7, 55.8, 47.4 (q, *J* = 27 Hz), 37.9, 28.5, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{24}F_3N_2O_4$ 413.1688; Found 413.1697.



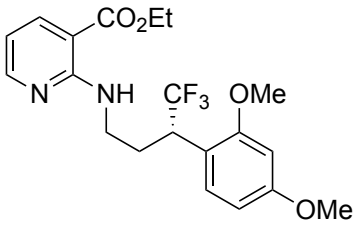
3am

Compound 3am (Scheme 2; pale yellow oil, 38.8 mg, 98% yield, 88% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 21.5 min (minor), t_2 = 24.2 min (major)); $[\alpha]^{20}_D$ +82.3 (*c* 1.40, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 5.0, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.99–7.90 (m, 1H), 6.82 (s, 1H), 6.77 (s, 2H), 6.52 (dd, *J* = 7.8, 5.0 Hz, 1H), 5.96 (s, 2H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.57–3.47 (m, 1H), 3.40-3.27 (m, 2H), 2.43–2.33 (m, 1H), 2.16–2.04 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 148.0, 147.5, 139.9, 127.7, 126.8 (q, *J* = 279 Hz), 123.0, 111.1, 108.9, 108.3, 106.2, 101.2, 60.8, 47.5 (q, *J* = 27 Hz), 37.9, 28.6, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{20}F_3N_2O_4$ 397.1375; Found 397.1370.



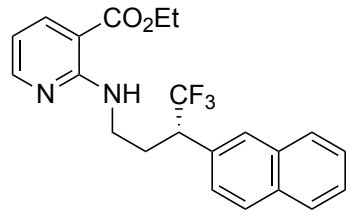
3an

Compound 3an (Scheme 2; colorless oil, 35.0 mg, 92% yield, 88% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 9.8 min (minor), t_2 = 11.3 min (major)); $[\alpha]^{20}_D$ +66.5 (*c* 1.02, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.99–7.92 (br, 1H), 6.95 (s, 1H), 6.92 (s, 2H), 6.51 (dd, *J* = 7.6, 4.8 Hz, 1H), 4.32 (q, *J* = 7.1 Hz, 2H), 3.53–3.43 (m, 1H), 3.42–3.26 (m, 2H), 2.47–2.34 (m, 1H), 2.31 (s, 6H), 2.22–2.12 (m, 1H),1.38 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 139.9, 138.1, 134.0, 129.9, 127.0 (q, *J* = 279 Hz), 126.9, 111.0, 106.2, 60.7, 47.8 (q, *J* = 27 Hz), 38.1, 28.6, 21.3, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{24}F_3N_2O_2$ 381.1790; Found 381.1794.



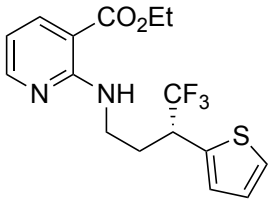
3ao

Compound 3ao (Scheme 2; colorless solid, 30.6 mg, 74% yield, 82% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 23.4 min (minor), t_2 = 32.4 min (major)); $[\alpha]^{20}_D$ +62.8 (*c* 0.95, $CHCl_3$) for 82% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.09 (dd, *J* = 7.6, 2.0 Hz, 1H), 8.02–7.95 (br, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 6.51–6.46 (m, 2H), 6.45 (d, *J* = 2.8 Hz, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 4.13–4.01 (m, 1H), 3.80 (s, 3H), 3.79 (s, 3H), 3.63–3.51 (m, 1H), 3.30–3.19 (m, 1H), 2.41–2.31 (m, 1H), 2.09–1.99 (m, 1H), 1.37 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.3, 160.3, 158.9, 158.4, 153.3, 139.9, 128.7, 127.4 (q, *J* = 280 Hz), 115.1, 110.8, 106.1, 104.6, 98.4, 60.7, 55.5, 55.3, 37.8, 37.7 (q, *J* = 19 Hz), 28.7, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{24}F_3N_2O_4$ 413.1688; Found 413.1688.



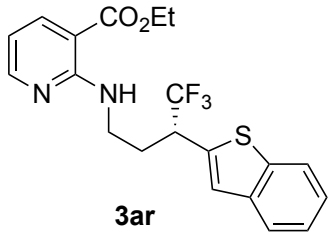
3ap

Compound 3ap (Scheme 2; pale yellow oil, 37.5 mg, 93% yield, 87% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 19.7 min (major), t_2 = 24.7 min (minor)); $[\alpha]^{20}_D$ +90.2 (*c* 0.82, $CHCl_3$) for 87% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 4.4, 2.0 Hz, 1H), 8.09 (dd, *J* = 8.0, 2.0 Hz, 1H), 8.01–7.95 (br, 1H), 7.90–7.77 (m, 4H), 7.56–7.42 (m, 3H), 6.51 (dd, *J* = 8.0, 4.4 Hz, 1H), 4.28 (q, *J* = 7.2 Hz, 2H), 3.67–3.50 (m, 2H), 3.45–3.35 (m, 1H), 2.58–2.46 (m, 1H), 2.41–2.29 (m, 1H),1.37 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.4, 158.4, 153.4, 139.8, 133.2, 133.1, 131.6, 128.8, 128.44, 128.38, 127.9, 127.6, 127.0 (q, *J* = 280 Hz), 126.24, 126.17, 111.1, 106.2, 60.7,148.1 (q, *J* = 27 Hz), 38.1, 28.6, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{22}F_3N_2O_2$ 403.1633; Found 403.1638.



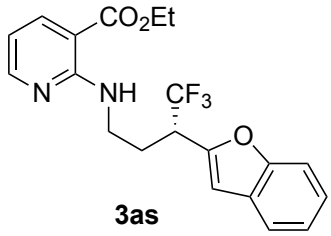
3aq

Compound 3aq (Scheme 2; colorless solid, 33.9 mg, 95% yield, 98% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 12.3 min (major), t_2 = 13.8 min (minor)); $[\alpha]^{20}_D$ +92.5 (*c* 0.91, $CHCl_3$) for 98% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 8.25 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.11 (dd, *J* = 7.8, 2.0 Hz, 1H), 8.04–7.95 (br, 1H), 7.30 (d, *J* = 5.4 Hz, 1H), 7.07 (d, *J* = 3.2 Hz, 1H), 7.01 (dd, *J* = 5.4, 3.2 Hz, 1H), 6.53 (dd, *J* = 7.8, 4.8 Hz, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 3.81–3.69 (m, 1H), 3.64–3.55 (m, 1H), 3.44–3.35 (m, 1H), 2.52–2.43 (m, 1H), 2.16–2.05 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 139.9, 136.1, 127.8, 127.0 (q, *J* = 279 Hz), 126.9, 125.7, 111.2, 106.3, 60.8, 43.2 (q, *J* = 28 Hz), 37.9, 30.0, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{18}F_3N_2O_2S$ 359.1041; Found 359.1047.



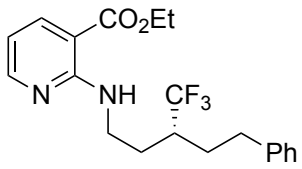
3ar

Compound 3ar (Scheme 2; pale yellow oil, 23.9 mg, 59% yield, 96% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 15.2 min (major), t_2 = 18.5 min (minor)); $[\alpha]^{20}_D$ +123.1 (*c* 1.04, $CHCl_3$) for 96% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 8.24 (dd, *J* = 4.6, 1.8 Hz, 1H), 8.10 (dd, *J* = 7.8, 1.8 Hz, 1H), 8.06–7.98 (br, 1H), 7.81 (dd, *J* = 6.8, 2.0 Hz, 1H), 7.75 (dd, *J* = 6.8, 2.4 Hz, 1H), 7.38–7.31 (m, 2H), 7.30 (s, 1H), 6.53 (dd, *J* = 7.8, 4.6 Hz, 1H), 4.29 (q, *J* = 7.2 Hz, 2H), 3.88–3.77 (m, 1H), 3.70–3.61 (m, 1H), 3.52–3.42 (m, 1H), 2.56–2.47 (m, 1H), 2.25–2.15 (m, 1H), 1.37 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 139.9, 139.8, 139.2, 137.0, 126.1 (q, *J* = 280 Hz), 124.9, 124.5, 124.4, 123.6, 122.2, 111.3, 106.3, 60.8, 44.2 (q, *J* = 29 Hz), 37.9, 29.7, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{20}F_3N_2O_2S$ 409.1198; Found 409.1191.



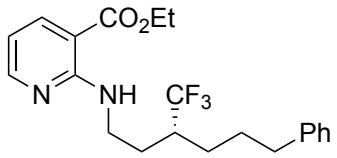
3as

Compound 3as (Scheme 2; pale yellow oil, 24.6 mg, 63% yield, 90% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 11.9 min (major), t_2 = 14.6 min (minor)); $[\alpha]^{20}_D$ +108.1 (*c* 0.98, $CHCl_3$) for 90% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.08 (dd, *J* = 7.8, 2.0 Hz, 1H), 8.06–7.97 (br, 1H), 7.54 (d, *J* = 6.8 Hz, 1H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.29 (td, *J* = 8.4, 1.5 Hz, 1H), 7.22 (td, *J* = 6.8, 1.5 Hz, 1H), 6.74 (s, 1H), 6.52 (dd, *J* = 7.8, 4.8 Hz, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 3.79–3.61 (m, 2H), 3.55–3.45 (m, 1H), 2.48–2.32 (m, 2H), 1.36 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.4, 158.5, 155.0, 153.4, 150.8, 139.9, 128.0, 125.7 (q, *J* = 278 Hz), 124.4, 122.9, 121.0, 111.3, 111.2, 106.6, 106.3, 60.7, 42.5 (q, *J* = 29 Hz), 38.0, 27.1, 14.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{20}F_3N_2O_3$ 393.1426; Found 393.1433.



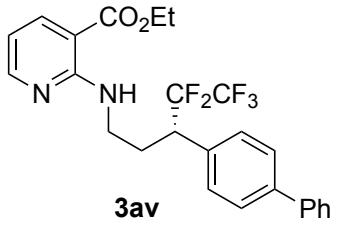
3at

Compound 3at (Scheme 2; colorless oil, 36.2 mg, 95% yield, 65% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, t_1 = 11.7 min (major), t_2 = 14.0 min (minor)); $[\alpha]^{20}_D$ –5.8 (*c* 0.79, $CHCl_3$) for 65% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 8.29 (dd, *J* = 4.4, 1.2 Hz, 1H), 8.14 (dd, *J* = 7.0, 1.2 Hz, 1H), 8.10–7.96 (br, 1H), 7.26 (t, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.56 (dd, *J* = 7.0, 4.4 Hz, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 3.74–3.64 (m, 1H), 3.64–3.52 (m, 1H), 2.79–2.65 (m, 2H), 2.30–2.16 (m, 1H), 2.04–1.86 (m, 3H), 1.86–1.76 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.4, 153.4, 141.2, 139.9, 128.5 (q, *J* = 270 Hz), 128.4, 128.3, 126.0, 111.1, 106.2, 60.8, 39.8 (q, *J* = 25 Hz), 38.3, 32.7, 29.7, 27.7, 14.3; ^{19}F NMR ($CDCl_3$) δ –69.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{24}F_3N_2O_2$ 381.1790; Found 381.1785.



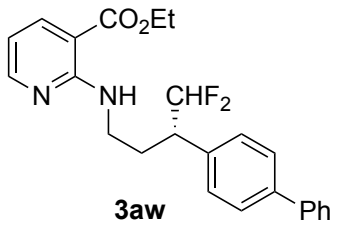
3au

Compound 3au (Scheme 2; colorless oil, 26.0 mg, 66% yield, 65% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 1.0 mL/min, 254 nm, t_1 = 8.3 min (minor), t_2 = 10.3 min (major)); $[\alpha]^{20}_D$ –7.9 (*c* 1.31, $CHCl_3$) for 65% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 8.26 (dd, *J* = 4.6, 2.0 Hz, 1H), 8.13 (dd, *J* = 7.6, 2.0 Hz, 1H), 8.09–7.99 (br, 1H), 7.27 (t, *J* = 7.2 Hz, 2H), 7.22–7.13 (m, 3H), 6.54 (dd, *J* = 7.6, 4.6 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.72–3.52 (m, 2H), 2.61 (t, *J* = 7.2 Hz, 2H), 2.30–2.14 (m, 1H), 2.01–1.91 (m, 1H), 1.84–1.65 (m, 4H), 1.62–1.50 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.37, 153.35, 141.8, 140.0, 128.5 (q, *J* = 279 Hz), 128.3, 125.8, 111.1, 106.2, 60.8, 40.4 (q, *J* = 25 Hz), 38.5, 35.8, 28.4, 27.9, 27.7, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{21}H_{26}F_3N_2O_2$ 395.1946; Found 395.1946.



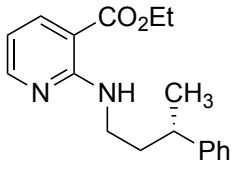
3av

Compound 3av (Scheme 2; colorless oil, 44.5 mg, 93% yield, 92% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IC, hexane/EtOH = 500:1, flow 0.8 mL/min, 254 nm, t_1 = 16.2 min (minor), t_2 = 21.9 min (major)); $[\alpha]^{20}_D$ +97.4 (*c* 1.11, $CHCl_3$) for 92% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.24 (dd, *J* = 4.8, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.6, 2.0 Hz, 1H), 8.00–7.91 (br, 1H), 7.60 (d, *J* = 6.8 Hz, 2H), 7.58 (d, *J* = 8.8 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.40–7.33 (m, 3H), 6.52 (dd, *J* = 7.6, 4.8 Hz, 1H), 4.30 (q, *J* = 7.2 Hz, 2H), 3.56–3.40 (m, 2H), 3.40–3.29 (m, 1H), 2.59–2.49 (m, 1H), 2.28–2.18 (m, 1H), 1.36 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 141.0, 140.4, 139.9, 132.7 (d, *J* = 6 Hz), 129.7, 128.8, 127.4, 127.3, 127.0, 120.7–113.1 (m, CF_3–CF_2–), 111.1, 106.2, 60.8, 45.3 (t, *J* = 21 Hz), 38.1, 27.7, 14.2; ^{19}F NMR ($CDCl_3$) δ –81.2, –115.5 (d, *J* = 273 Hz), –120.5 (d, *J* = 273 Hz). HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{25}H_{24}F_5N_2O_2$ 479.1758; Found 479.1764.



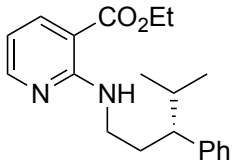
3aw

Compound 3aw (Scheme 2; colorless oil, 31.2 mg, 76% yield, 63% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 35.0 min (minor), t_2 = 40.5 min (major)); $[\alpha]^{20}_D$ +62.4 (*c* 1.00, $CHCl_3$) for 63% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.24 (dd, *J* = 4.4, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.8, 2.0 Hz, 1H), 8.04–7.95 (br, 1H), 7.59 (d, *J* = 7.6 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.38–7.32 (m, 3H), 6.51 (dd, *J* = 7.8, 4.4 Hz, 1H), 5.92 (td, *J* = 56.4, 3.6 Hz, 1H), 4.30 (q, *J* = 7.2 Hz, 2H), 3.63–3.54 (m, 1H), 3.49–3.40 (m, 1H), 3.28–3.16 (m, 1H), 2.41–2.31 (m, 1H), 2.20–2.09 (m, 1H), 1.36 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 140.7, 140.6, 139.9, 135.3, 129.3, 128.7, 127.4, 127.3, 127.1, 117.8 (t, *J* = 244 Hz), 111.0, 106.2, 60.7, 47.4 (t, *J* = 20 Hz), 38.3, 28.2, 14.2; ^{19}F NMR ($CDCl_3$) δ –118.8 (d, *J* = 278 Hz), –121.9 (d, *J* = 278 Hz). HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{25}F_2N_2O_2$ 411.1884; Found 411.1873.



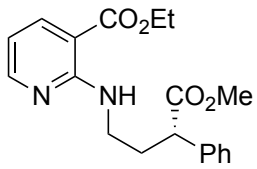
3ax

Compound 3ax (Scheme 2; pale yellow oil, 25.1 mg, 79% yield, 49% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 12.8 min (minor), t_2 = 13.9 min (major)); $[\alpha]^{20}_D$ –25.2 (*c* 0.86, $CHCl_3$) for 49% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.25 (dd, *J* = 4.4, 2.0 Hz, 1H), 8.12 (dd, *J* = 7.4, 2.0 Hz, 1H), 8.06–7.93 (br, 1H), 7.32–7.15 (m, 5H), 6.50 (dd, *J* = 7.4, 4.4 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.52–3.34 (m, 2H), 2.88 (sext, *J* = 7.2 Hz, 1H), 2.04–1.88 (m, 2H), 1.38 (t, *J* = 7.2 Hz, 3H), 1.30 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 146.8, 139.9, 128.4, 127.0, 126.0, 110.6, 105.9, 60.7, 39.3, 37.8, 37.6, 22.5, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{23}N_2O_2$ 299.1760; Found 299.1755.



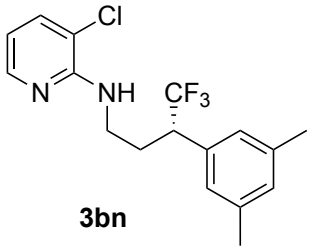
3ay

Compound 3ay (Scheme 2; pale yellow oil, 23.8 mg, 72% yield, 38% ee). A solution of hexane/$CHCl_3$ (2:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 13.6 min (major), t_2 = 18.5 min (minor)); $[\alpha]^{20}_D$ –17.4 (*c* 1.08, $CHCl_3$) for 38% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 8.22 (dd, *J* = 5.2, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.6, 2.0 Hz, 1H), 8.02–7.82 (br, 1H), 7.27 (t, *J* = 7.6 Hz, 2H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.6 Hz, 2H), 6.48 (dd, *J* = 7.6, 5.2 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 3.39–3.20 (m, 2H), 2.47–2.37 (m, 1H), 2.23–2.13 (m, 1H), 1.96–1.78 (m, 2H), 1.38 (t, *J* = 7.2 Hz, 3H), 0.95 (d, *J* = 7.2 Hz, 3H), 0.73 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 167.5, 158.5, 153.4, 143.6, 139.9, 128.5, 128.0, 125.9, 110.5, 105.8, 60.6, 50.6, 39.6, 33.5, 32.6, 20.9, 20.5, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{27}N_2O_2$ 327.2073; Found 327.2075.



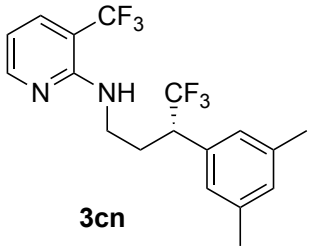
3az

Compound 3az (Scheme 2; colorless solid, 24.7 mg, 72% yield, 36% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 9.9 min (major), t_2 = 14.6 min (minor)); $[\alpha]^{20}_D$ +27.9 (*c* 0.83, $CHCl_3$) for 36% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.24 (d, *J* = 3.6 Hz, 1H), 8.12 (d, *J* = 6.8 Hz, 1H), 8.08–7.95 (br, 1H), 7.36–7.29 (m, 4H), 7.29–7.22 (m, 1H), 6.52 (dd, *J* = 6.8, 3.6 Hz, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 3.79–3.66 (m, 1H), 3.64 (s, 3H), 3.60–3.49 (m, 1H), 3.49–3.38 (m, 1H), 2.53–2.41 (m, 1H), 2.18–2.07 (m, 1H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 174.1, 167.5, 158.4, 153.3, 140.0, 138.6, 128.7, 127.9, 127.3, 110.9, 106.2, 60.7, 52.1, 49.1, 38.8, 33.0, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{23}N_2O_4$ 343.1658; Found 343.1655.



3bn

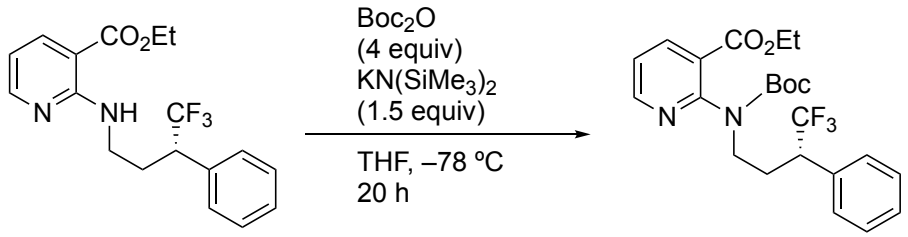
Compound 3bn (Scheme 3; pale yellow oil, 30.4 mg, 99% yield, 88% ee). A solution of hexane/EtOAc (10:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 21.2 min (major), t_2 = 26.9 min (minor)); $[\alpha]^{20}_D$ +67.9 (*c* 1.05, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 7.99 (dd, *J* = 5.2 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 6.97 (s, 1H), 6.92 (s, 2H), 6.52 (dd, *J* = 7.8, 5.2 Hz, 1H), 4.90–4.82 (br, 1H), 3.45–3.38 (m, 2H), 3.36–3.25 (m, 1H), 2.48–2.35 (m, 1H), 2.31 (s, 6H), 2.22–2.10 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 153.6, 145.9, 138.3, 135.9, 134.0, 130.0, 126.9 (q, *J* = 280 Hz), 126.8, 115.3, 112.9, 47.9 (q, *J* = 27 Hz), 38.9, 28.5, 21.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{17}H_{19}{}^{35}ClF_3N_2$ 343.1189; Found 343.1187.



3cn

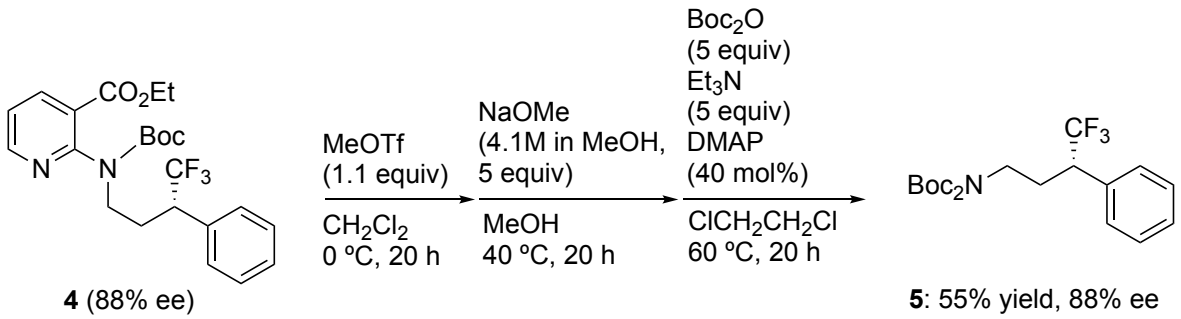
Compound 3cn (Scheme 3; colorless oil, 31.5 mg, 84% yield, 81% ee). A solution of hexane/EtOAc (20:1) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB x 2, hexane/2-propanol = 500:1, flow 0.5 mL/min, 254 nm, t_1 = 20.3 min (major), t_2 = 20.9 min (minor)); $[\alpha]^{20}_D$ +71.7 (*c* 0.96, $CHCl_3$) for 81% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.23 (dd, *J* = 5.0 Hz, 1H), 7.63 (d, *J* = 7.0 Hz, 1H), 6.98 (s, 1H), 6.91 (s, 2H), 6.62 (dd, *J* = 7.0, 5.0 Hz, 1H), 4.83–4.75 (br, 1H), 3.53–3.43 (m, 1H), 3.41–3.22 (m, 2H), 2.48–2.37 (m, 1H), 2.32 (s, 6H), 2.18–2.07 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 154.3, 151.6, 138.3, 135.0 (q, *J* = 5 Hz), 133.9, 130.1, 126.9 (q, *J* = 279 Hz), 124.4 (q, *J* = 272 Hz), 126.8, 111.5, 108.6 (q, *J* = 31 Hz), 47.7 (q, *J* = 27 Hz), 38.7, 28.3, 21.3; ^{19}F NMR ($CDCl_3$) δ –63.9, –69.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{19}F_6N_2$ 377.1452; Found 377.1450.

7. Removal of a pyridyl group (Scheme 4)



3aa (88% ee) **4**: 99% yield, 88% ee

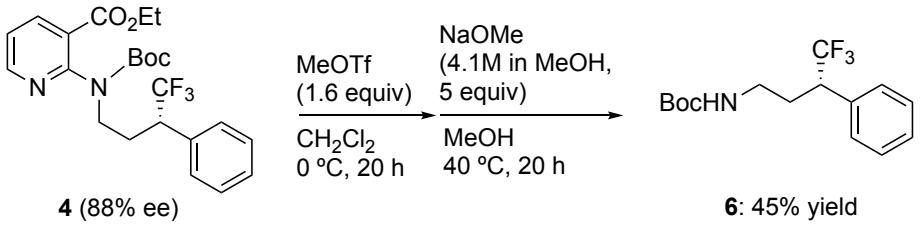
Transformation of 3aa into 4: To a solution of **1aa** (88% ee, 705 mg, 2.0 mmol) in THF (8.0 mL) was added potassium bis(trimethylsilyl)amide solution (8.0 mL, 0.5 M in toluene, 4.0 mmol) at –78 °C under N_2. After stirring the mixture at –78 °C for 1 h, di-*tert*-butyl dicarbonate (352 mg, 8.0 mmol) was added to the mixture, and the reaction mixture was stirred at the same temperature for 20 h. H_2O was added to the mixture at –78 °C and the resulting mixture was allowed to warm to room temperature. The resulting solution was extracted with EtOAc and the organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with hexane to give **4** as a colorless oil (725 mg, 99% yield). The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, t_1 = 10.8 min (major), t_2 = 11.7 min (minor)); $[\alpha]^{20}_D$ +5.3 (*c* 1.08, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 8.54–8.33 (m, 1H), 8.22–8.04 (m, 1H), 7.36–7.08 (m, 6H), 4.35 (q, *J* = 7.4 Hz, 2H), 3.99–3.38 (m, 3H), 2.55–2.05 (m, 2H), 1.52–1.26 (m, 9H), 1.38 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 166.0, 150.6, 150.0, 139.5, 138.6, 134.1, 129.1, 128.5, 128.1, 126.9 (q, *J* = 279 Hz), 124.5, 120.7, 120.3, 81.5, 61.5, 47.9–46.7 (m), 46.1–44.9 (m), 28.1, 14.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{28}F_3N_2O_4$ 453.2001; Found 453.2012.



4 (88% ee) **5**: 55% yield, 88% ee

Transformation of 4 into 5: To a solution of **4** (88% ee, 87.5 mg, 0.19 mmol) in CH_2Cl_2 (0.8 mL) was added methyl trifluoromethanesulfonate (23 μL, 0.21 mmol) at 0 °C, and the mixture was stirred at the same temperature for 20 h. The solvent was removed on a rotary evaporator, and the residue was dissolved in methanol (0.8 mL). Sodium methoxide (4.1 M in methanol, 225 μL, 0.97 mmol) was added to the mixture, and the resulting mixture was stirred at 40 °C for 20 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent, and the solvent

was removed on a rotary evaporator. After dissolving the residue in dichloroethane (0.8 mL), di-*tert*-butyl dicarbonate (211 mg, 0.97 mmol), triethylamine (135 μL, 0.97 mmol), and 4-dimethylaminopyridine (9.4 mg, 0.080 mmol) were added to the mixture, and the reaction mixture was stirred at 60 °C for 20 h. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel (hexane/EtOAc = 10:1) to give **5** as a colorless oil (42.7 mg, 55% yield in 3 steps). The ee was measured by HPLC (Chiralpak AD-H x 3, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 26.7 min (major), t_2 = 27.7 min (minor)); $[\alpha]^{20}_D$ +15.4 (*c* 0.60, $CHCl_3$) for 88% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 7.39–7.29 (m, 5H), 3.55–3.38 (m, 2H), 3.34–3.22 (m, 1H), 2.33–2.15 (m, 2H), 1.47 (s, 18H); ^{13}C NMR ($CDCl_3$) δ 152.2, 134.0, 129.3, 128.9, 128.7, 128.3, 126.6 (q, *J* = 279 Hz), 82.5, 48.1 (q, *J* = 27 Hz), 44.3, 28.0, 27.9; ^{19}F NMR ($CDCl_3$) δ –69.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{29}F_3N_1O_4$ 404.2049; Found 404.2046. The absolute configuration of **5** was determined to be *S*-(+) by comparison of its specific rotation with the value reported previously.[17] The previously reported structure of **5** has been wrongly assigned to be **6**.[17] The reported NMR data and specific rotation value are in good agreement with the data obtained for compound **5** in our hands. We also prepared compound **6** and characterized as shown below.

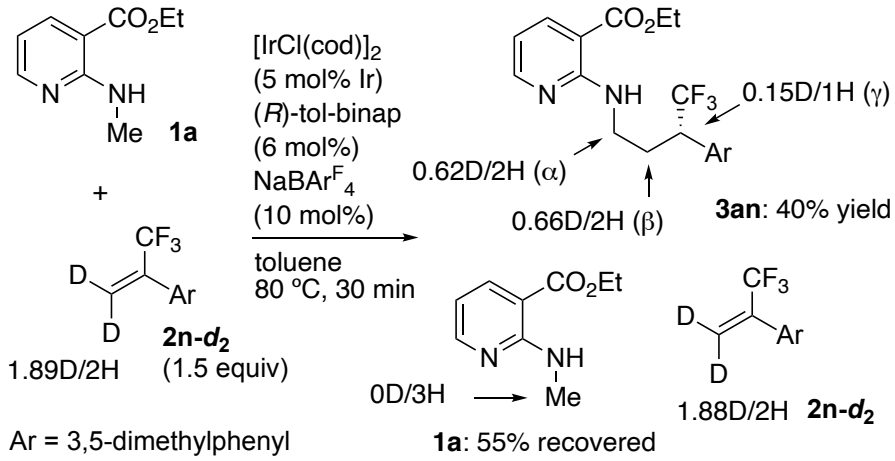


4 (88% ee) **6**: 45% yield

Transformation of 4 into 6: To a solution of **4** (88% ee, 49.8 mg, 0.11 mmol) in CH_2Cl_2 (0.4 mL) was added methyl trifluoromethanesulfonate (19 μL, 0.18 mmol) at 0 °C, and the mixture was stirred at the same temperature for 20 h. The solvent was removed on a rotary evaporator, and the residue was dissolved in methanol (0.4 mL). Sodium methoxide (4.1 M in methanol, 134 μL, 0.55 mmol) was added to the mixture, and the resulting mixture was stirred at 40 °C for 20 h. The mixture was passed through a short column of alumina with CH_2Cl_2 as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel (hexane/EtOAc = 5:1) to give **6** as a colorless oil (15.3 mg, 45% yield in 2 steps). $[\alpha]^{20}_D$ +49.0 (*c* 0.77, $CHCl_3$). ^{1}H NMR ($CDCl_3$) δ 7.39–7.33 (m, 3H), 7.31–7.27 (m, 2H), 4.50–4.40 (br, 1H), 3.38–3.25 (m, 1H), 3.05–2.92 (m, 2H), 2.28–2.18 (m, 1H), 2.05–1.99 (m, 1H), 1.42 (s, 9H); ^{13}C NMR ($CDCl_3$) δ 155.7, 134.0, 128.9, 128.8, 128.4, 126.8 (q, *J* = 279 Hz), 79.4, 47.7 (q, *J* = 26 Hz), 38.0, 29.3, 28.3; ^{19}F NMR ($CDCl_3$) δ –69.7. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{15}H_{21}F_3N_1O_2$ 304.1524; Found 304.1532.

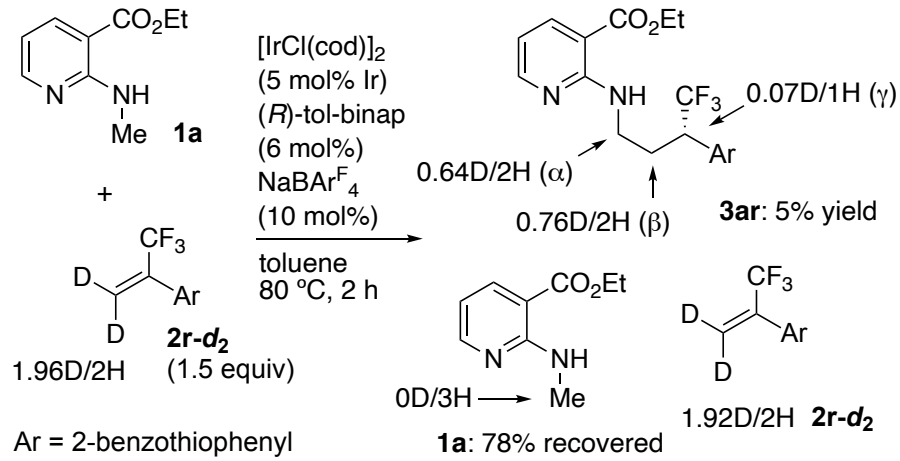
8. Deuterium-labeling experiments

In order to gain some mechanistic insights into the present hydroalkylation reaction, deuterium-labeling experiments were performed (Schemes S1 and S2). The reaction of **1a** with deuterated styrene **2n-*d*$_2$** (1.89D/2H) at 80 °C for 30 min gave **3an** in 40% yield, where deuterium incorporation was observed at the α- and γ-positions of **3an** (Scheme S1). In contrast, H/D exchange at the methyl group of recovered **1a** was not observed. The similar trend was observed for the addition to **2r-*d*$_2$**, which has lower reactivity than **2n**. Treatment of **1a** with **2r-*d*$_2$** in the presence of the Ir catalyst at 80 °C for 2 hours gave 5% yield of the addition product **3ar**, involving migration of deuterium from β- to α- and γ-positions (Scheme S2). In addition, no racemization of **3an** (88% ee) was observed in the presence of an Ir/(*S*)-binap catalyst.



Scheme S1 The reaction of **1a** with **2n-*d*$_2$**.

A mixture of **1a** (18.0 mg, 0.10 mmol), **2n-*d*$_2$** (30.3 mg, 0.15 mmol, 1.5 equiv), $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% of Ir), (*R*)-tol-binap (4.1 mg, 0.0060 mmol, 6 mol%), and $NaBAr^F{}_4$ (9.2 mg, 0.010 mmol, 10 mol%) were placed in a Schlenk tube under N_2. Toluene (0.4 mL) was added to the mixture, and the resulting mixture was stirred at 80 °C for 30 min. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel. The yields and deuterium contents of **1a**, **2n-*d*$_2$**, and **3an** were determined by ^{1}H NMR.

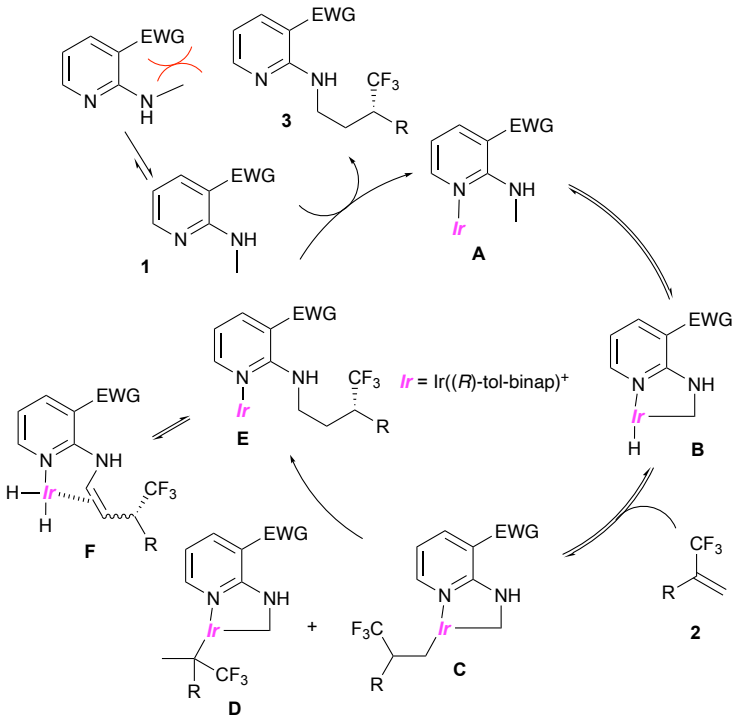


Scheme S2 The reaction of **1a** with **2r-*d*$_2$**.

A mixture of **1a** (18.0 mg, 0.10 mmol), **2r-*d*$_2$** (32.9 mg, 0.15 mmol, 1.5 equiv), $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% of Ir), (*R*)-tol-binap (4.1 mg, 0.0060 mmol, 6 mol%), and $NaBAr^F{}_4$ (9.2 mg, 0.010 mmol, 10 mol%) were placed in a Schlenk tube under N_2. Toluene (0.4 mL) was added to the mixture, and the mixture was stirred at 80 °C for 2 h. The solvent was removed on a rotary evaporator, and the residue was subjected to preparative TLC on silica gel. The yields and deuterium contents of **1a**, **2r-*d*$_2$**, and **3ar** were determined by ^{1}H NMR.

9. A plausible catalytic cycle

Based on the deuterium-labeling experiments, a proposed catalytic cycle of the present hydroalkylation reaction of the 1,1'-disubstituted alkenes is shown in Scheme S3. Linear-selective insertion of **2** to hydridoiridium species **B**, which is formed from species **A**, occurs to give **C**, and successive irreversible reductive elimination gives **E**. Ligand exchange of **E** with **1** gives the product **3** and regenerates **A**. The H/D exchange observed at the α- and γ-positions may occur by way of species **D**, which did not lead to the addition products. In addition, species **F**, which is formed by C–H activation of **E** and successive β-elimination, may lead to the H/D exchange at the α-position.[18]

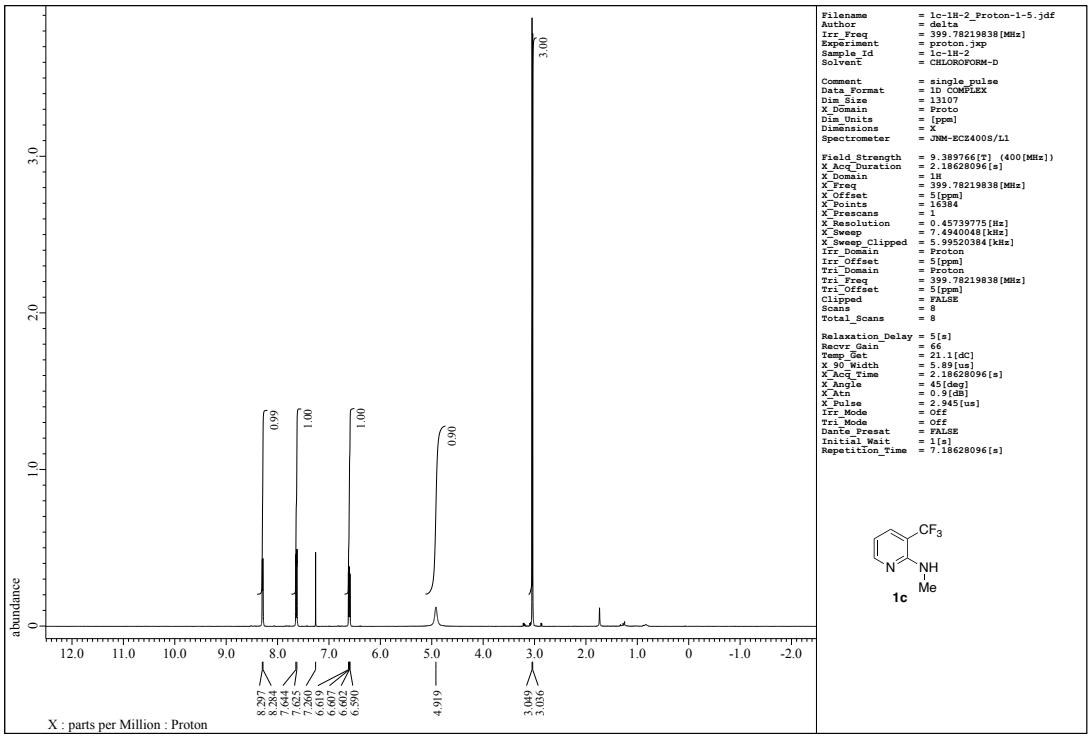


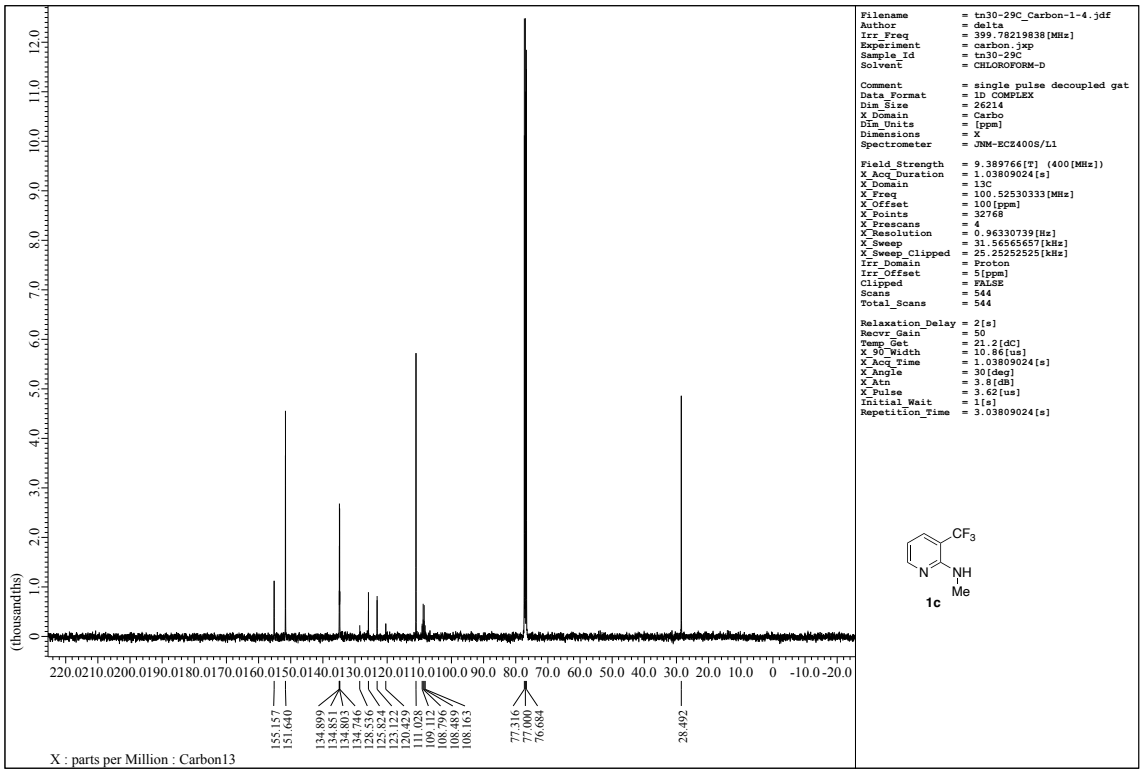
Scheme S3 A plausible catalytic cycle.

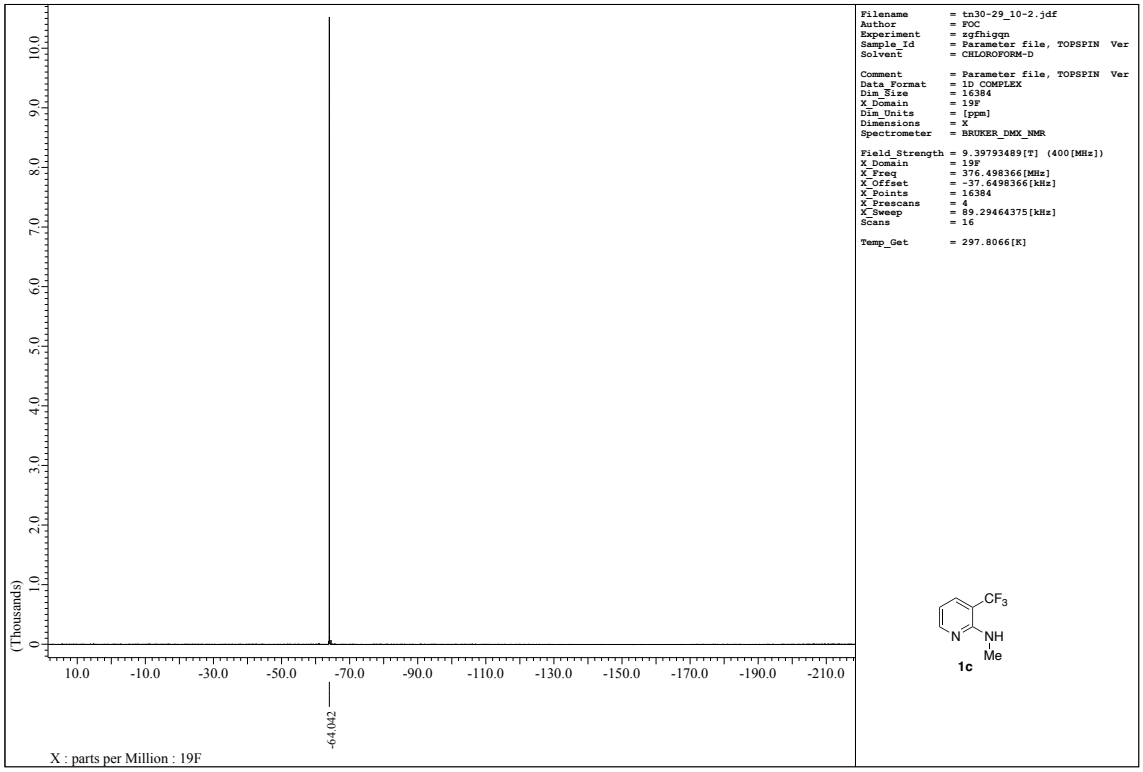
10. References

1. J. L. Herde, J. C. Lambert and C. V. Senoff, *Inorg. Synth.,* 1974, **15**, 18.
2. R, Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.,* 1985, **23**, 126.
3. M. Brookhart, B. Grant and A. F. Volpe, Jr., *Organometallics,* 1992, 11, 3920.
4. M. Woźniak, D. J. Buurman and H. C. Van Der Plas, *J. Heterocyclic Chem.,* 1985, **22**, 765.
5. T. Mita, K. Michigam and, Y. Sato, *Chem. Asian. J.,* 2013, **8**, 2970.
6. C. C. D. Wybon, C. Mensch, K. Hollanders, C. Gadais, W. A. Herrebout, S. Ballet and B. U. W. Maes, *ACS Catal.,* 2018, **8**, 203.
7. Y. Chen, N. Ni, D. Cheng and X. Xu, *Tetrahedron Lett*., 2020, **61**, 152425.
8. R. J. Wiles, J. P. Phelan and G. A. Molander, *Chem. Commun.,* 2019, **55**, 7599.
9. S. B. Lang, R. J. Wiles, C. B. Kelly and G. A. Molander, *Angew. Chem., Int. Ed*., 2017, **56**, 15073.
10. T. Ichitsuka, T. Fujita and J. Ichikawa, *ACS Catal.,* 2015, **5**, 5947.
11. M. Bergeron, T. Johnson and J.-F. Paquin, *Angew. Chem., Int. Ed*., 2011, **50**, 11112.
12. S. Zhang, Z. Shen and H. Jian, *J. Org. Chem.,* 2020, **85**, 6143.
13. J. Wu, P. S. Grant, X. Li, A. Nobel and V. K. Aggarwal, *Angew. Chem., Int. Ed*., 2019, **58**, 5697.
14. F. Li, J. Wie, H. Shan, C. Sun and L. Chen, *RSC Adv*., 2012, **2**, 8645.
15. S. Feng, K. Wang, Y. Ping and J. Wang, *J. Am. Chem. Soc.,* 2020, **142**, 21032.
16. E. Halbfinder, K. Gorochesky, S. A. Lévesque, A. R. Beaudoin, L. Sheihet, S. Margel and B. Fischer, *Org. Biomol. Chem.,* 2003, **1**, 2821.
17. P. Poutrel, M. V. Ivanova, X. Pannecoucke, P. Jubault and T. Poisson, *Chem*. *Eur*. *J*., 2019, **25**, 15262.
18. M. Nagai, M. Nagamoto, T. Nishimura and H. Yorimitsu, *Chem. Lett.,* 2017, **46**, 1176.

11. NMR spectra and chiral HPLC charts



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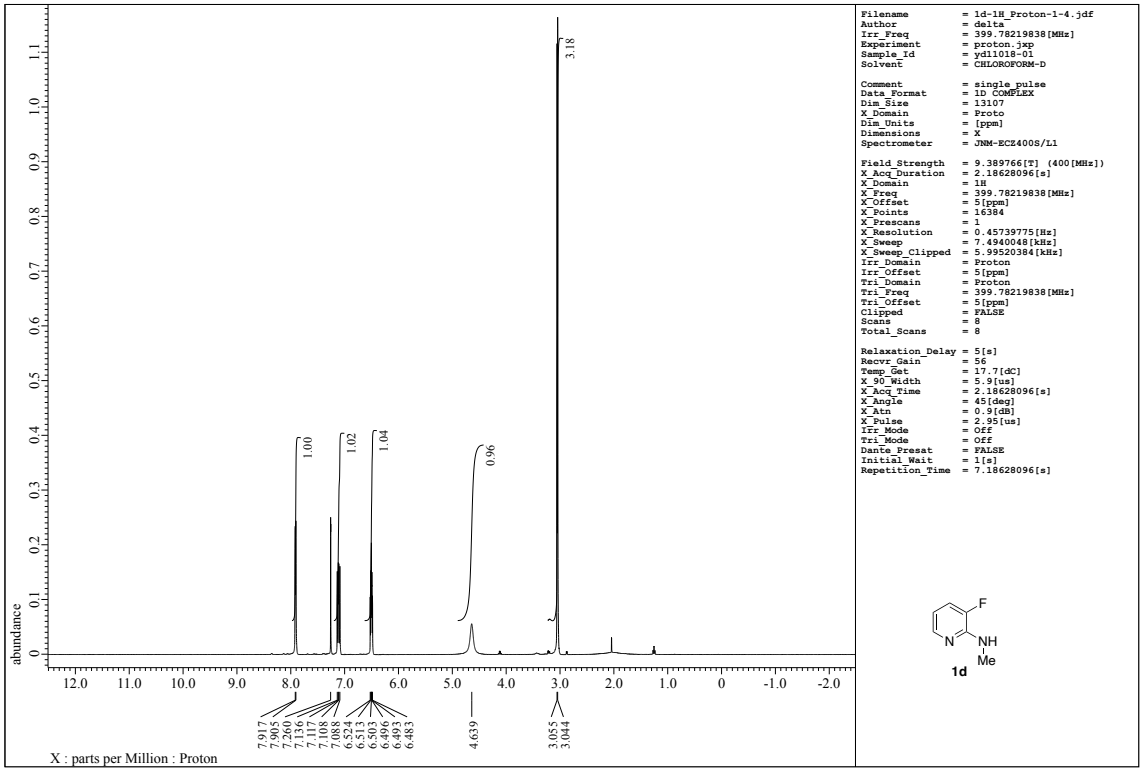
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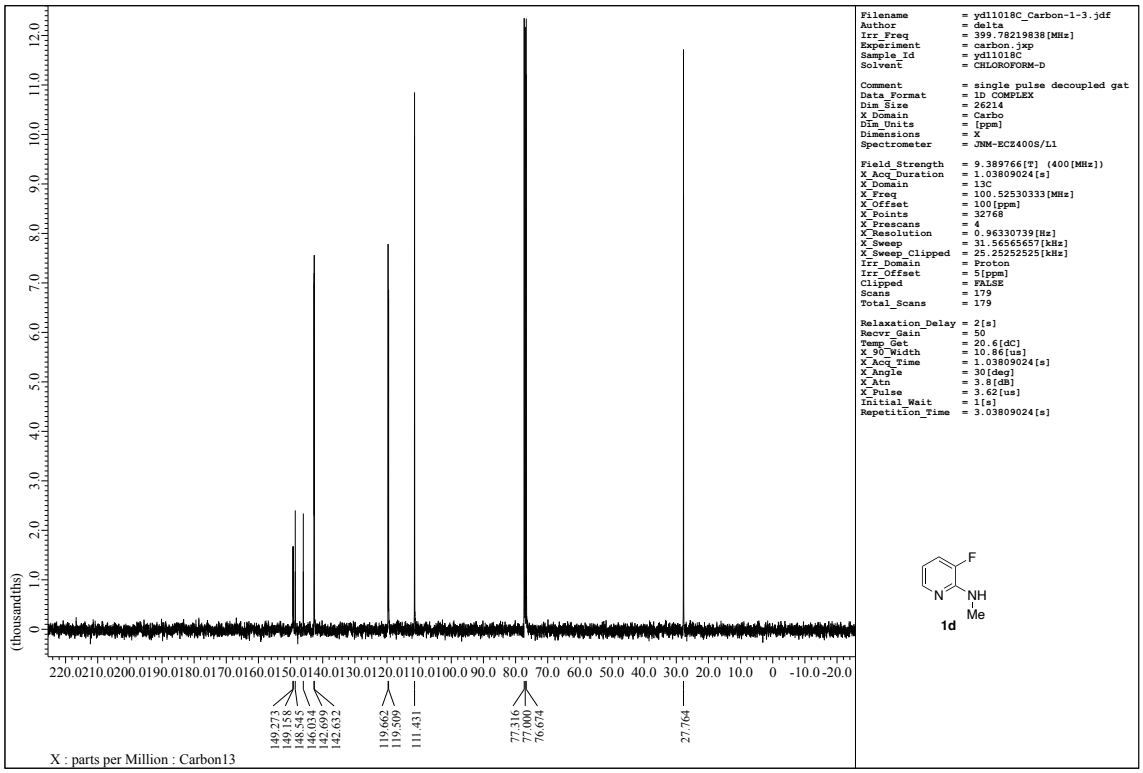
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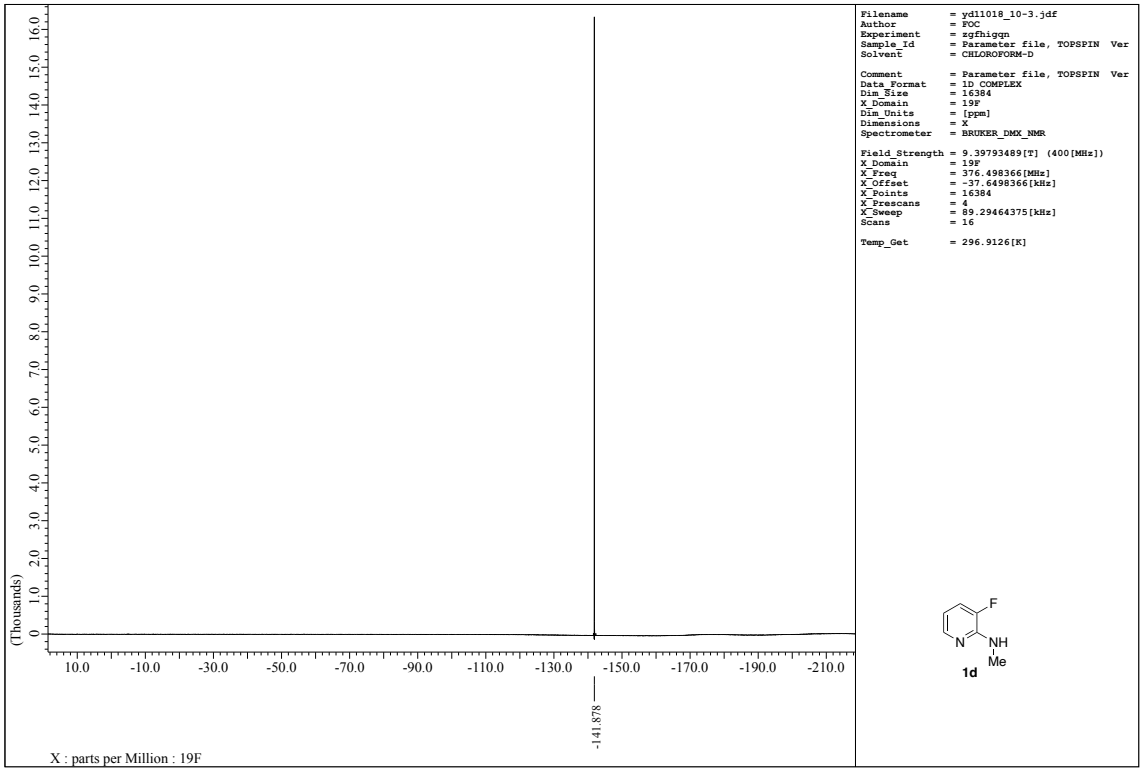
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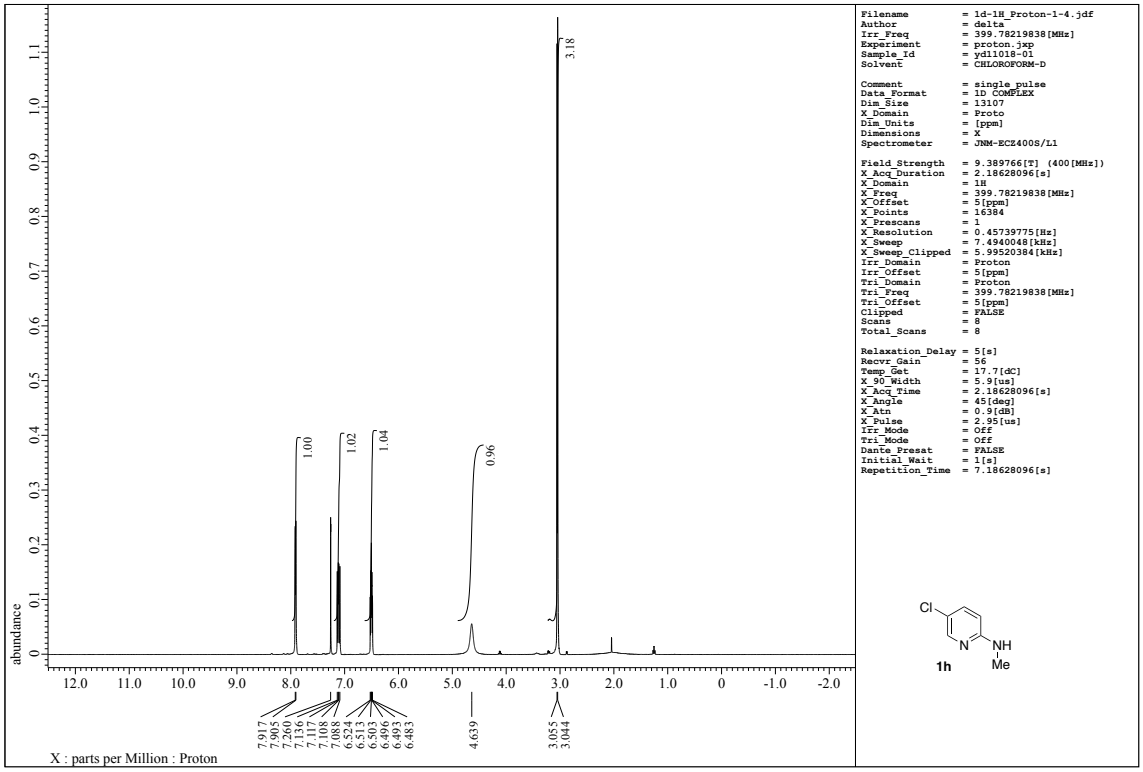
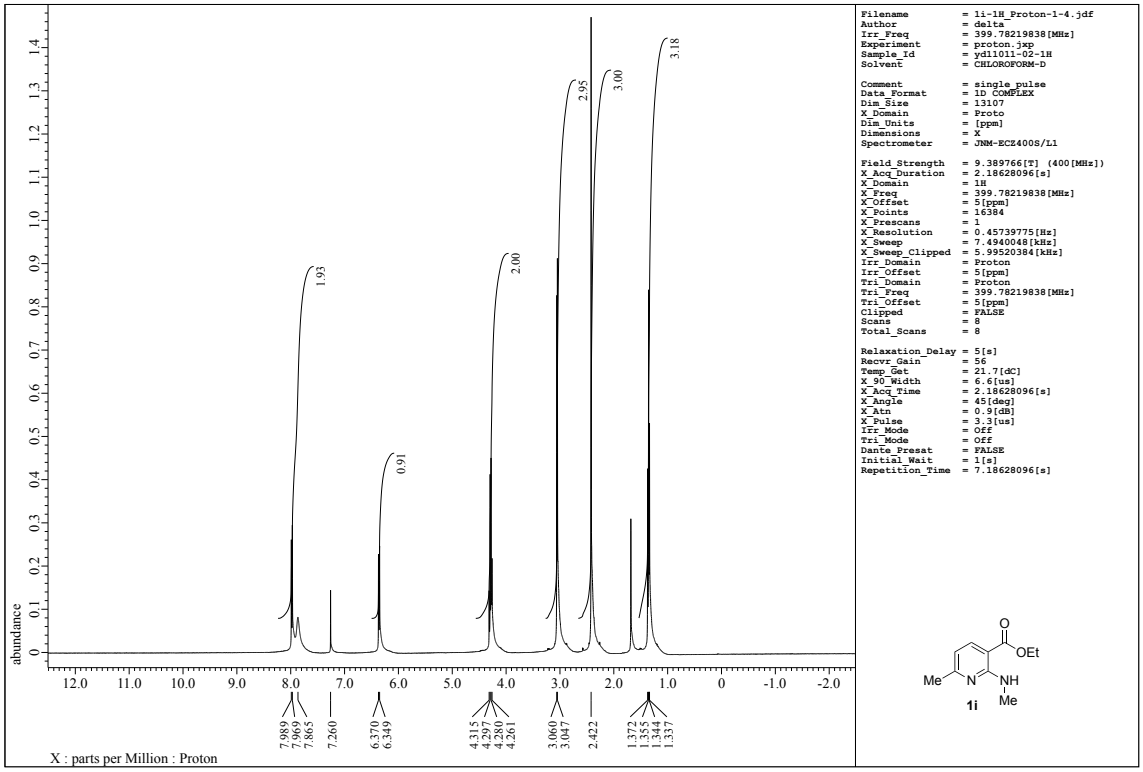
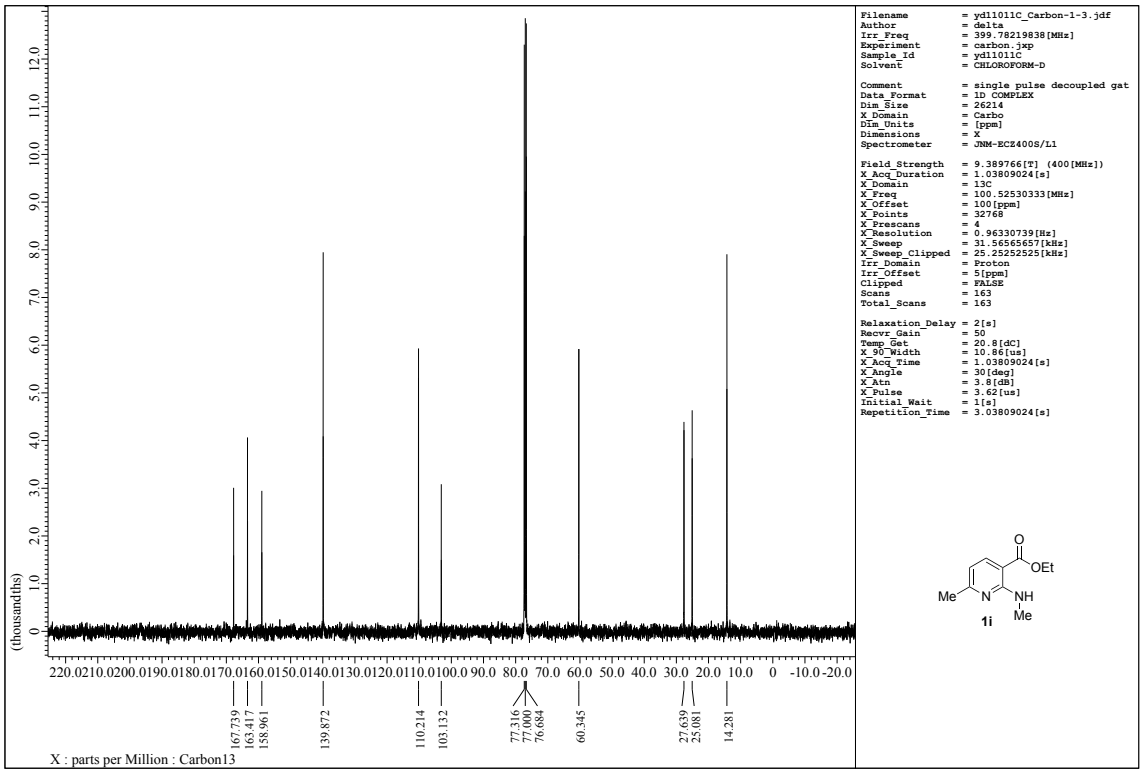
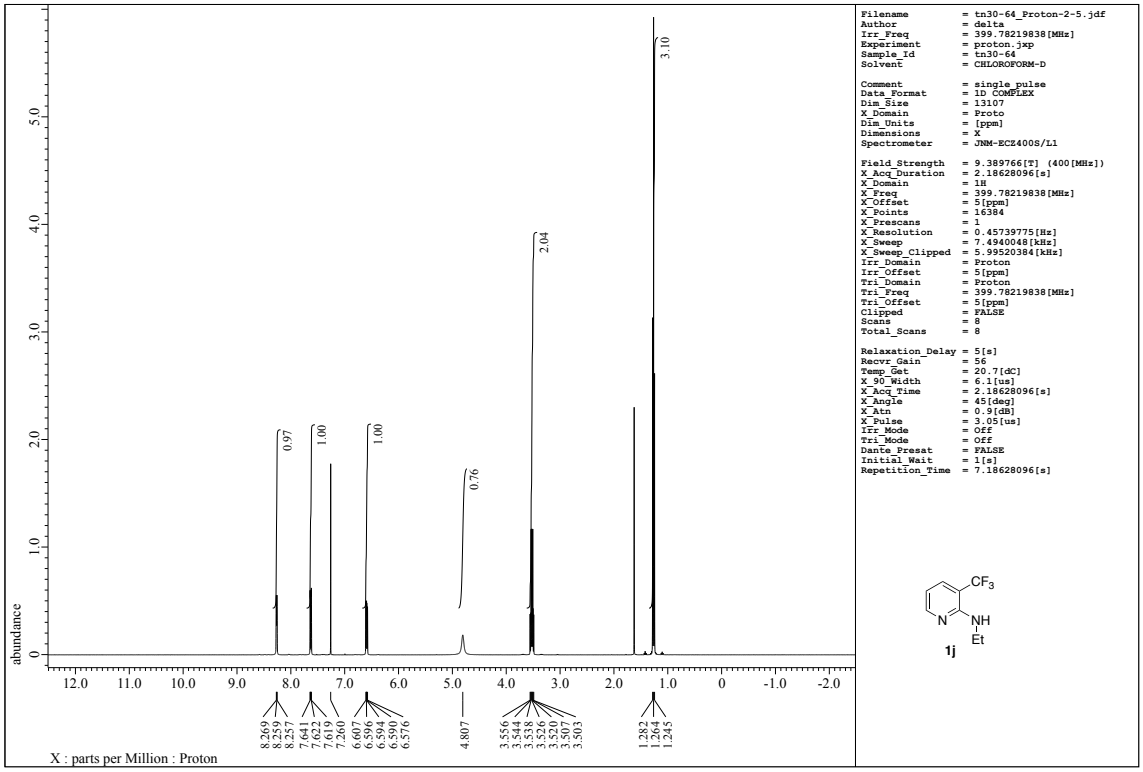
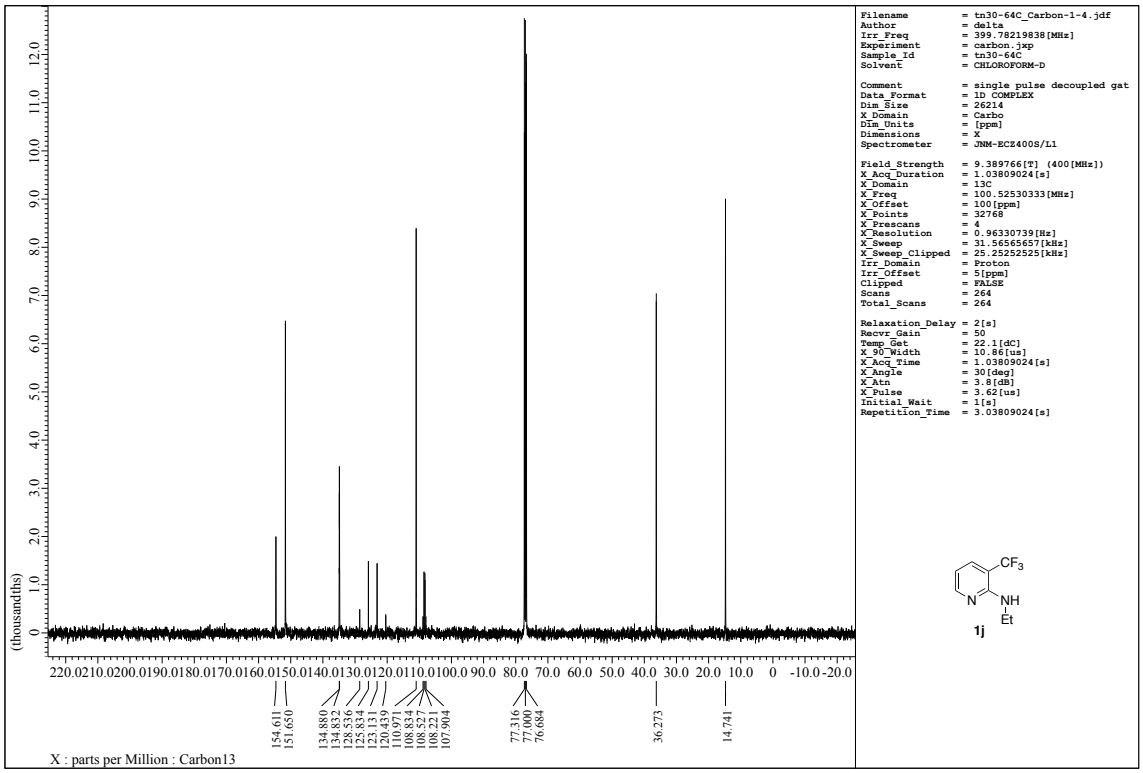
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 19F
X_Freq = 376.498366[MHz]
X_Offset = -37.6498366[kHz]
X_Points = 16384
X_Prescans = 4
X_Sweep = 89.29464375[kHz]
Scans = 16
Temp_Get = 296.9126[K]
F
N
NH
Me
1d

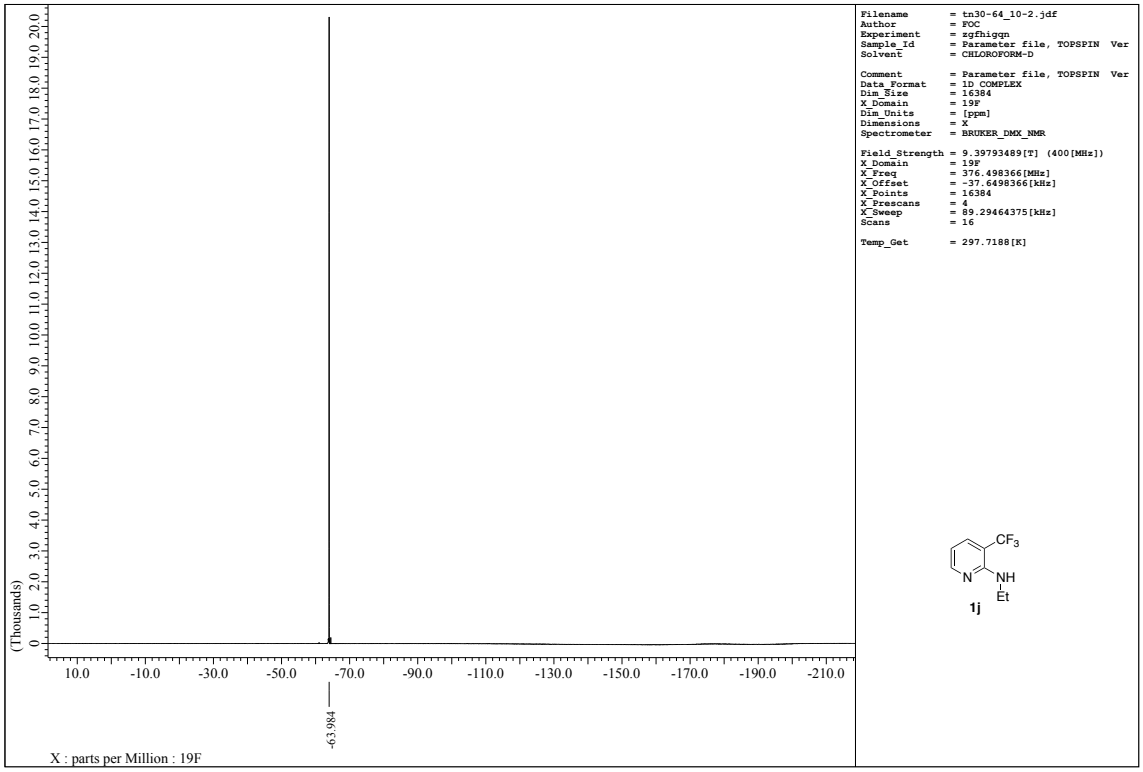

abundance
3.18
1.00
1.02
1.04
0.96
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.917
7.905
7.896
7.130
7.117
7.108
7.088
6.524
6.513
6.504
6.493
6.483
4.639
3.055
3.044
X : parts per Million : Proton
Filename = 1d-1H_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1018-01
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 17.7[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
Cl
N
NH
Me
1h

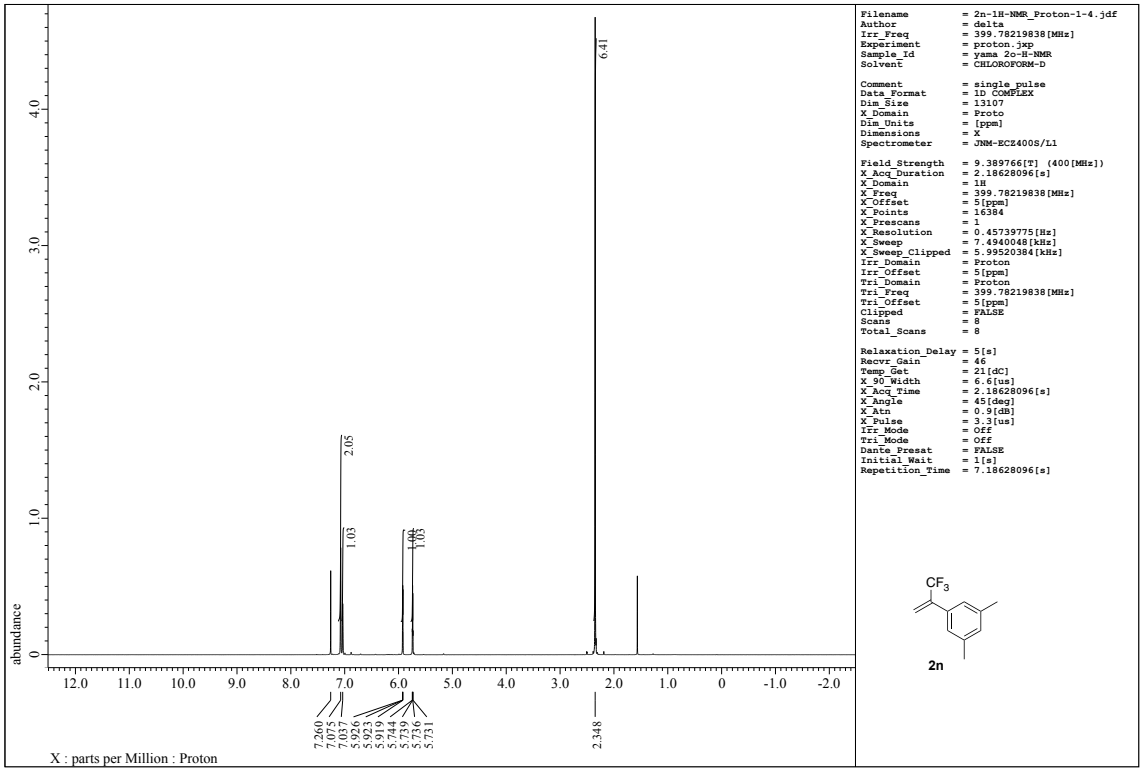

abundance
X : parts per Million : Proton
1.93
0.91
2.00
2.95
3.00
3.18
7.989
7.969
7.865
7.260
6.370
6.349
4.315
4.297
4.280
4.261
3.060
3.047
2.422
1.372
1.355
1.344
1.337
Filename = 1i-1H_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1011-02-1H
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.7[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
O
OEt
Me
N
NH
Me
1i

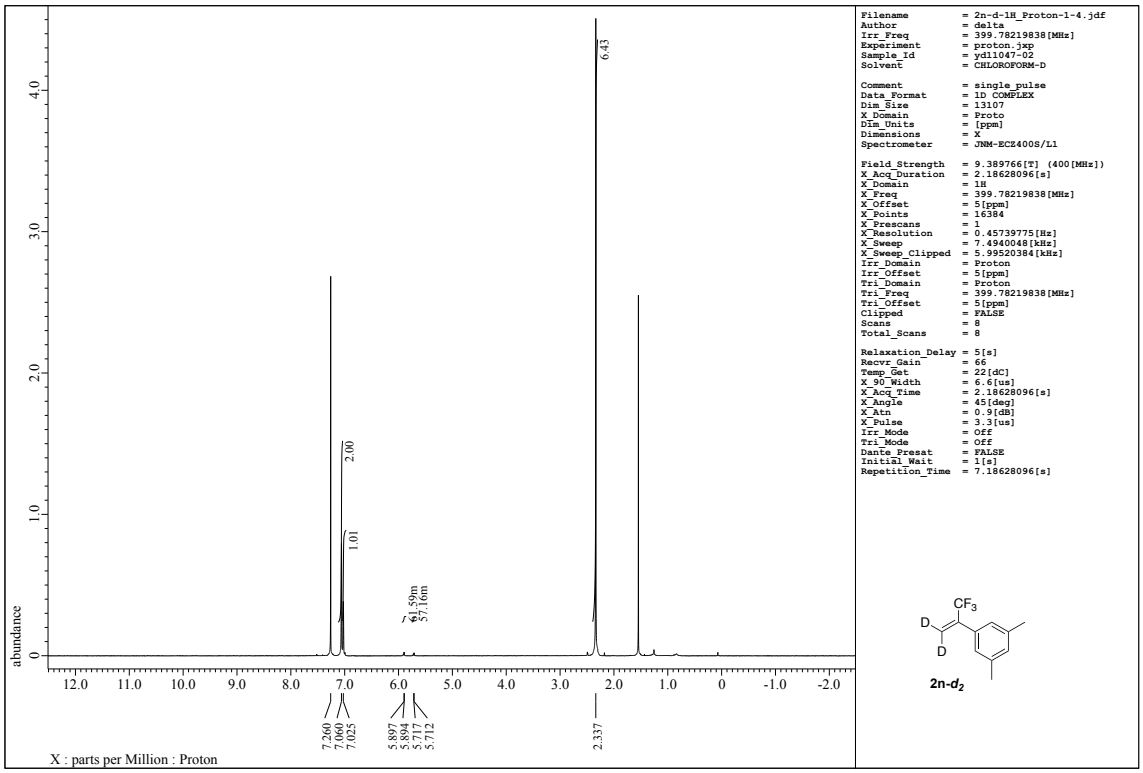

(thousandths)
X : parts per Million : Carbon13
167.739
163.417
158.961
139.872
110.214
103.132
77.316
77.000
76.684
60.345
27.639
25.081
14.281
Filename = ydl1011C_Carbon-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = ydl1011C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 163
Total_Scans = 163
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.8[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
O
OEt
Me
N
NH
Me
1i

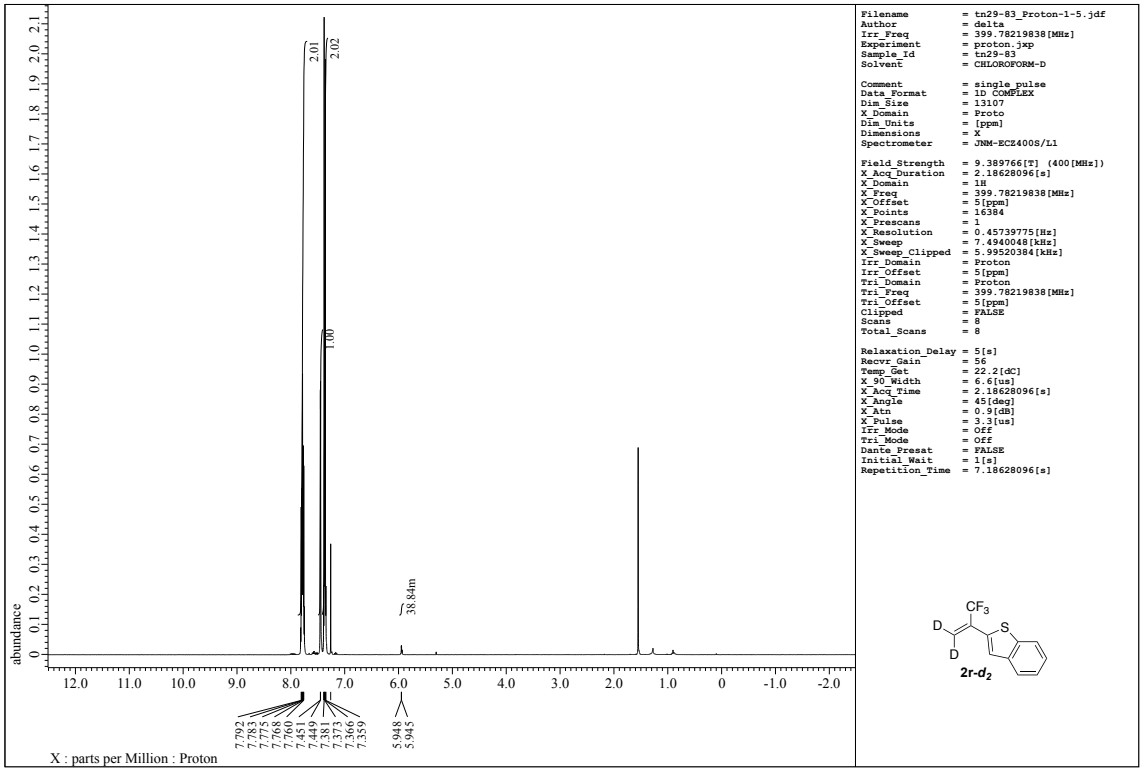

Filename = tn30-64_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-64
Solvent = CHLOROFORM-D
Comment = single pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 20.7[dC]
X_90_Width = 6.1[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
0.97
1.00
1.00
0.76
2.04
3.10
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
8.269 8.259 8.257 7.641 7.622 7.610 7.260 6.607 6.596 6.594 6.590 6.576 4.807 3.556 3.544 3.538 3.526 3.520 3.507 3.503 1.282 1.264 1.245
X : parts per Million : Proton
CF3
N
NH
Et
1j

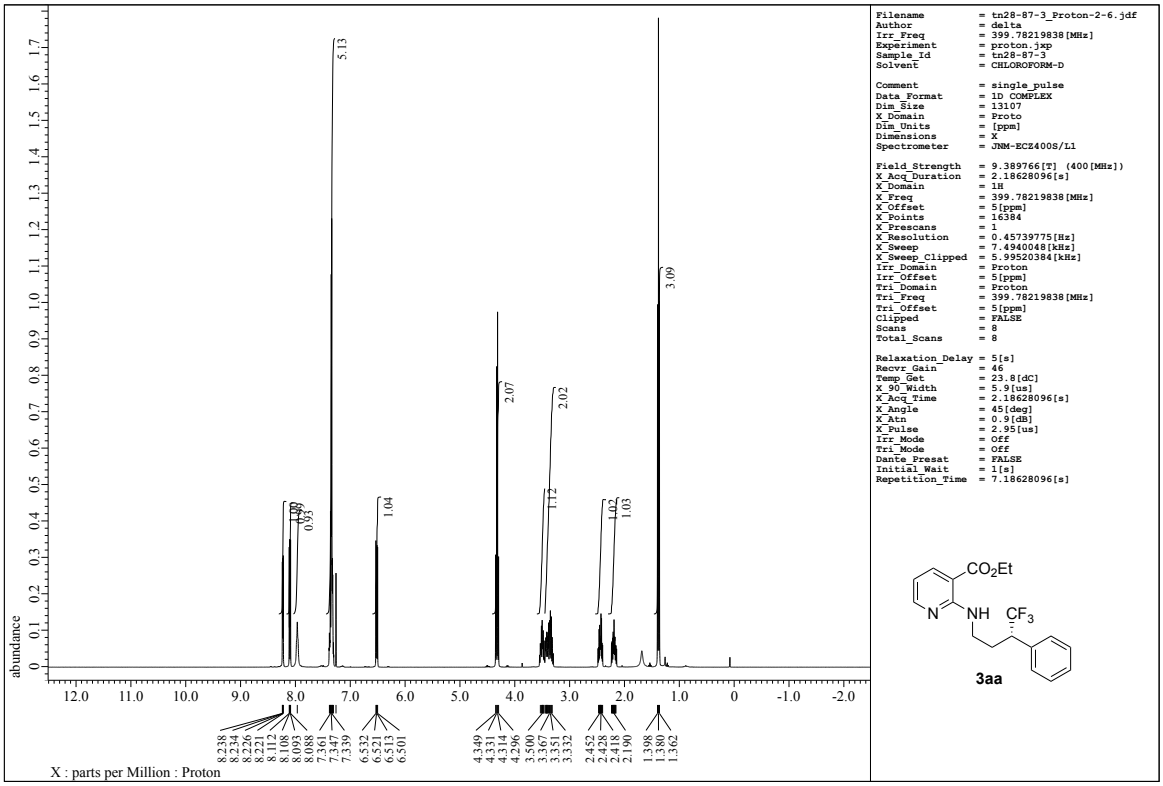

Filename = tn30-64C_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn30-64C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 264
Total_Scans = 264
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.1[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
154.611 151.650 134.880 134.832 128.536 125.834 123.131 120.429 110.971 108.834 108.527 108.221 107.904 77.316 77.000 76.684 36.273 14.741
X : parts per Million : Carbon13
CF3
N
NH
Et
1j

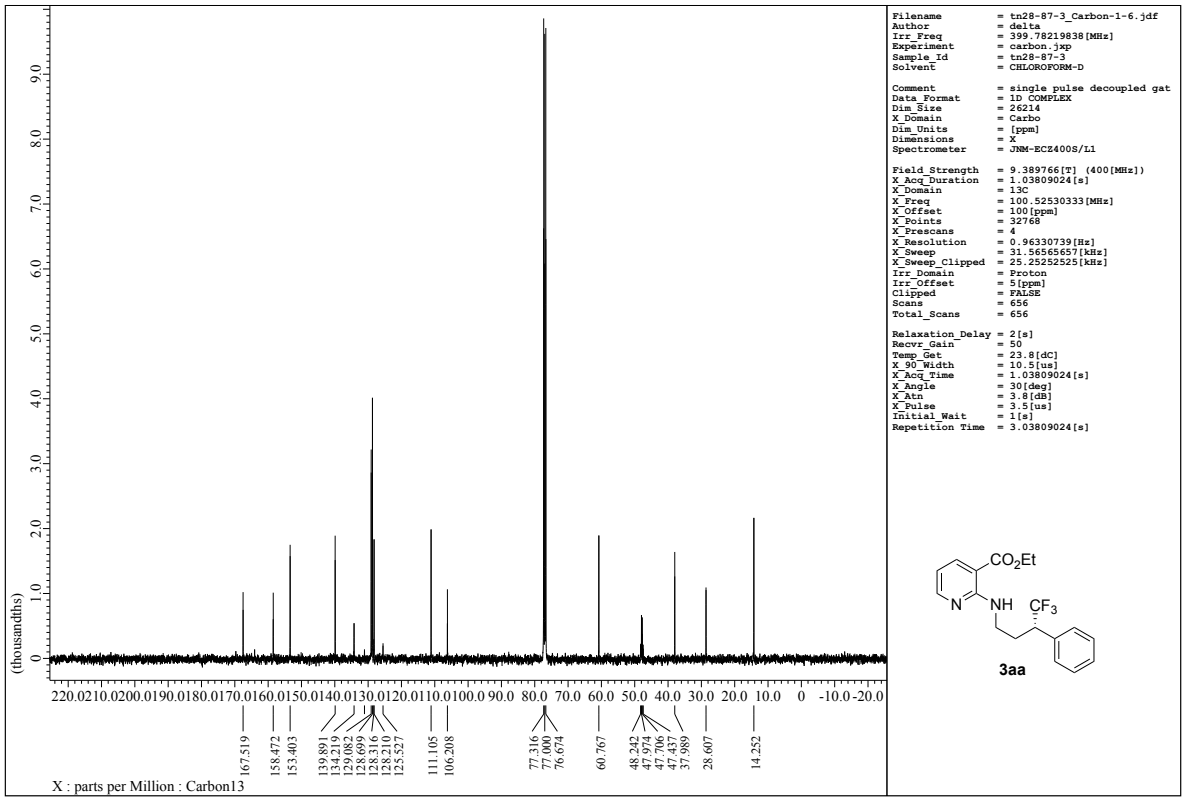

(Thousands)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0
10.0 -10.0 -30.0 -50.0 -70.0 -90.0 -110.0 -130.0 -150.0 -170.0 -190.0 -210.0
-63.984
X : parts per Million : 19F
Filename = tn30-64_10-2.jdf
Author = FOC
Experiment = zgfhigqn
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 16384
X_Domain = 19F
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 19F
X_Freq = 376.498366[MHz]
X_Offset = -37.6498366[kHz]
X_Points = 16384
X_Prescans = 4
X_Sweep = 89.29464375[kHz]
Scans = 16
Temp_Get = 297.7188[K]
CF3
N
NH
Et
1j

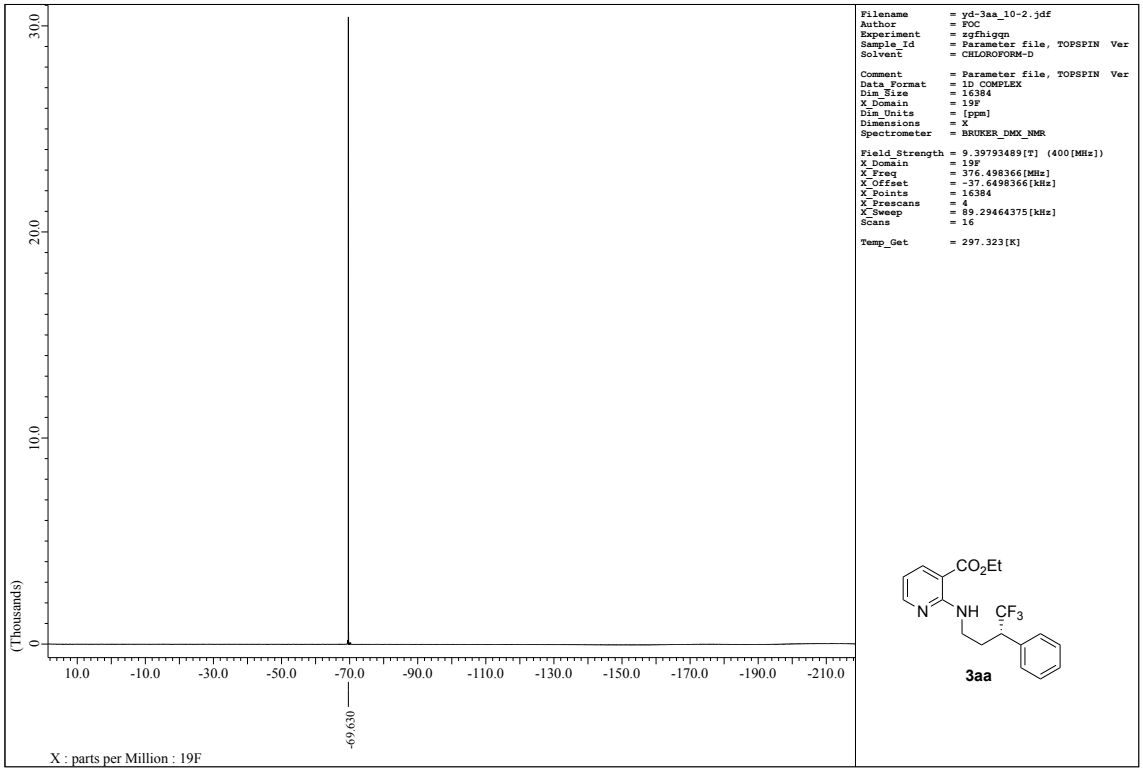

abundance
6.41
2.05
1.03
1.00
1.03
7.260
7.075
7.038
5.926
5.923
5.919
5.744
5.739
5.736
5.731
2.348
X : parts per Million : Proton
Filename = 2n-1H-NMR_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yama 2o-H-NMR
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 21[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CF3
2n

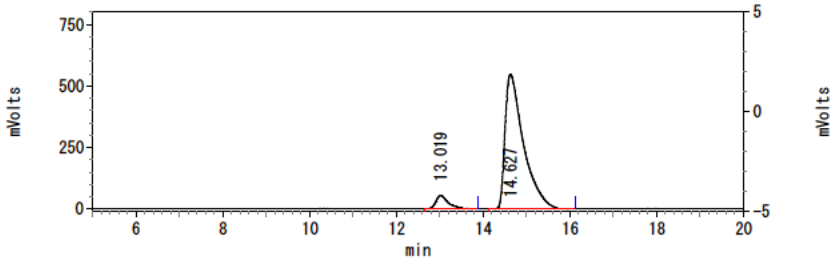

abundance
6.43
2.00
1.01
1.59
1.16
7.260
7.060
7.025
5.897
5.894
5.717
5.712
2.337
X : parts per Million : Proton
Filename = 2n-d-1H_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd11047-02
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 66
Temp_Get = 22[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CF3
D
D
2n-d2


abundance
2.01
2.02
1.00
38.84m
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
7.792
7.783
7.775
7.768
7.760
7.445
7.440
7.381
7.373
7.366
7.359
5.948
5.945
X : parts per Million : Proton
Filename = tn29-83_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn29-83
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.2[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CF3
D
S
D
2r-d2

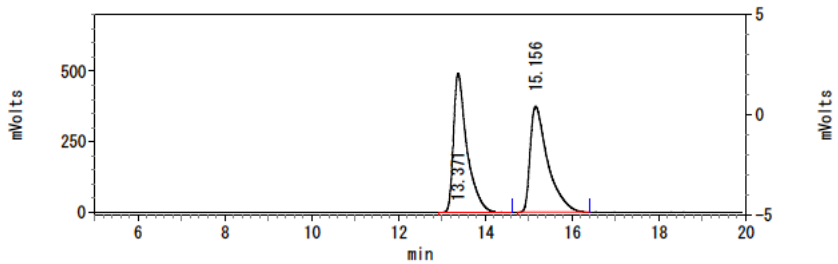

Filename = tn28-87-3_Proton-2-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-87-3
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[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 = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
5.13
3.09
2.07
2.02
1.12
1.04
1.02
1.03
0.96
0.93
CO2Et
N
NH
CF3
3aa
X : parts per Million : Proton


Filename = tn28-87-3_Carbon-1-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-87-3
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 656
Total_Scans = 656
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.8[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
167.519
158.472
153.403
139.891
134.219
129.082
128.699
128.316
128.210
125.527
111.105
106.208
77.316
77.000
76.674
60.767
48.242
47.974
47.706
47.437
37.989
28.607
14.252
CO2Et
N
NH
CF3
3aa
X : parts per Million : Carbon13

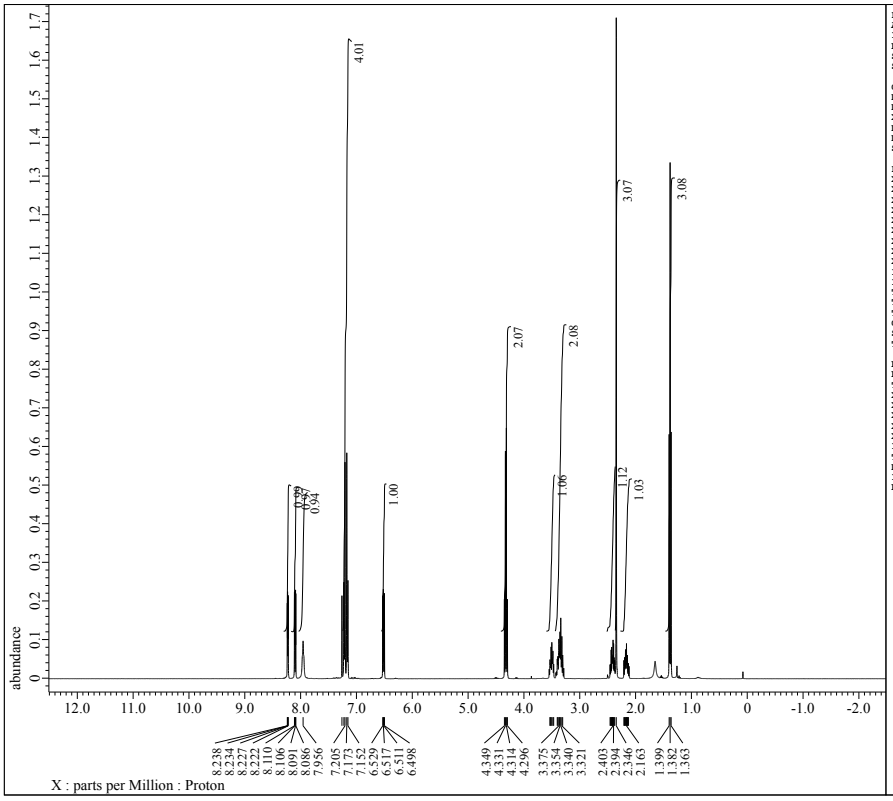




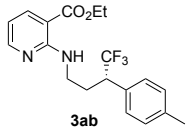
Pk #	Retention Time	Area	Area Percent
1	13.019	1067638	6.205
2	14.627	16137954	93.795



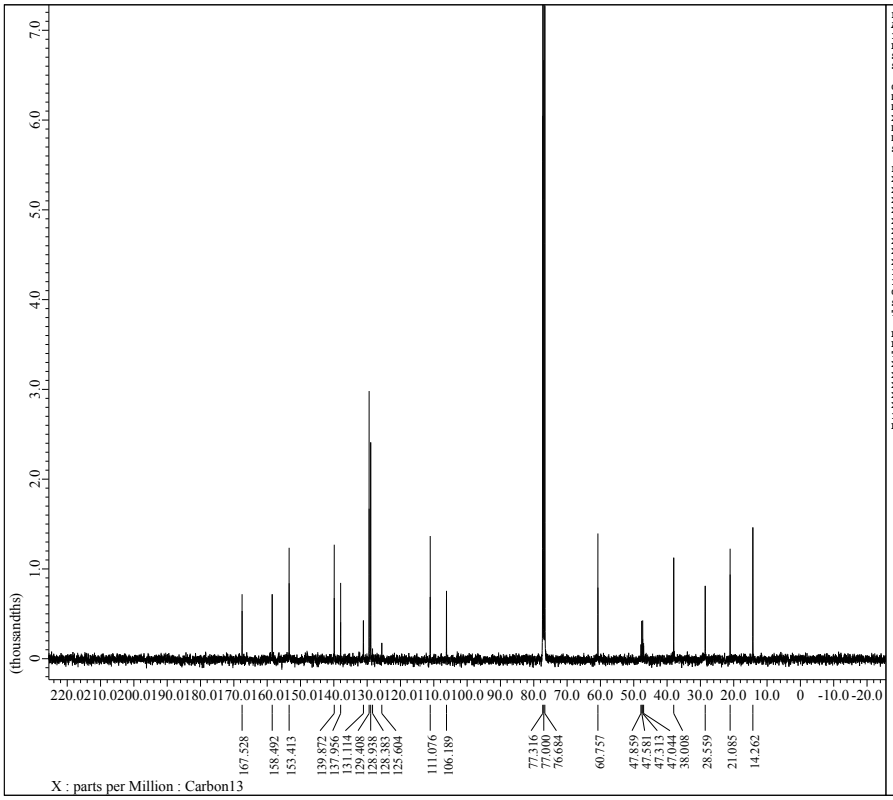
Pk #	Retention Time	Area	Area Percent
1	13.371	10553360	50.018
2	15.156	10545603	49.982



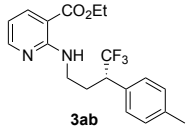

```
Filename          = tn28-86-2_Proton-2-6.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn28-86-2
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 23.6[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



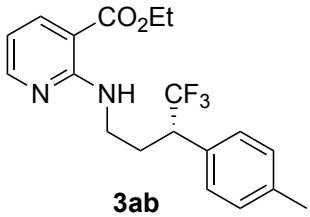
3ab



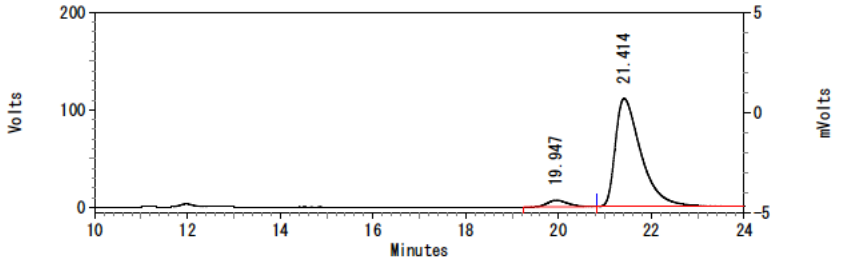

```
Filename          = tn28-86-2_Carbon-2-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn28-86-2
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 960
Total_Scans       = 960

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 23.5[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



3ab

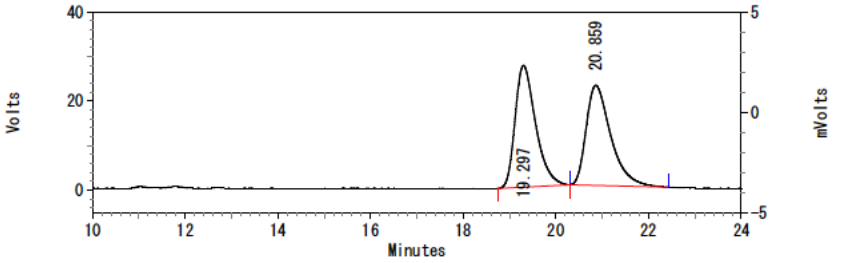


3ab



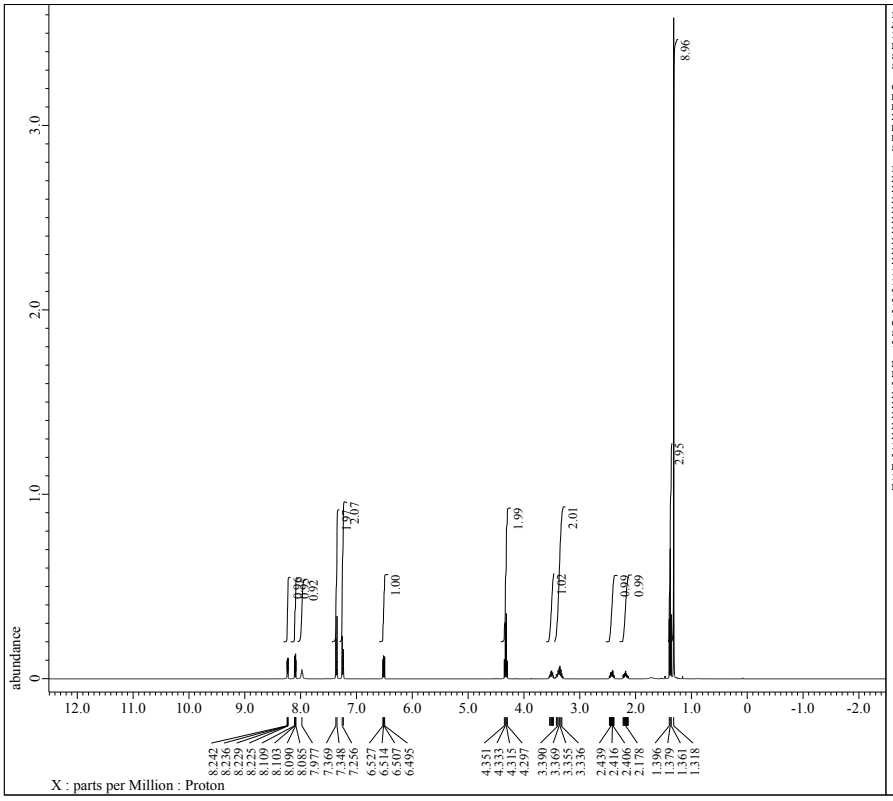
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	19.947	205664	4.560	6745
2	21.414	4304095	95.440	110810

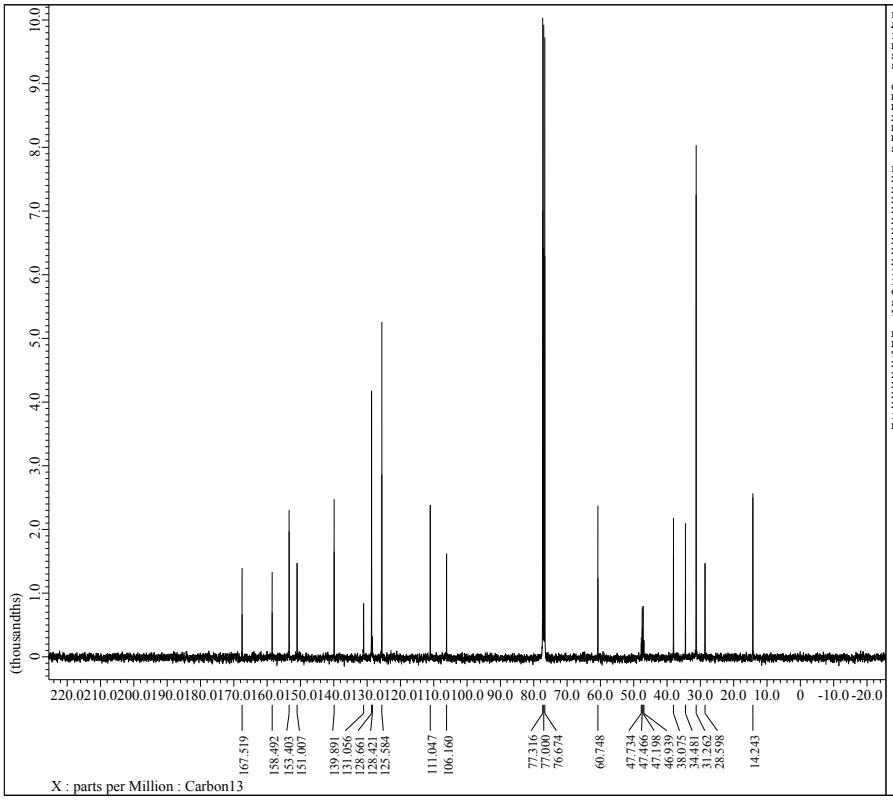


UV Results

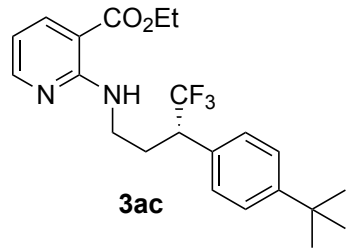
Pk #	Retention Time	Area	Area Percent	Height
1	19.297	841737	50.319	27339
2	20.859	831055	49.681	22448



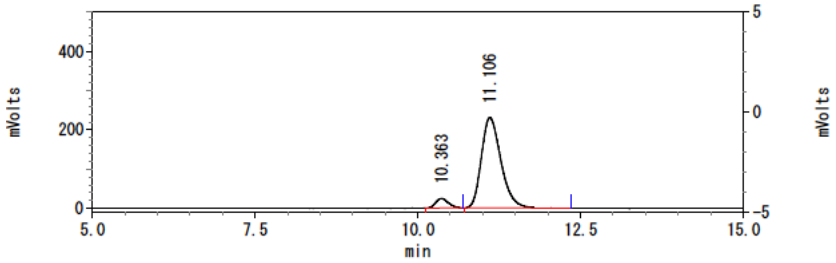

```
Filename          = tn29-5_Proton-2-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn29-5
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[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        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



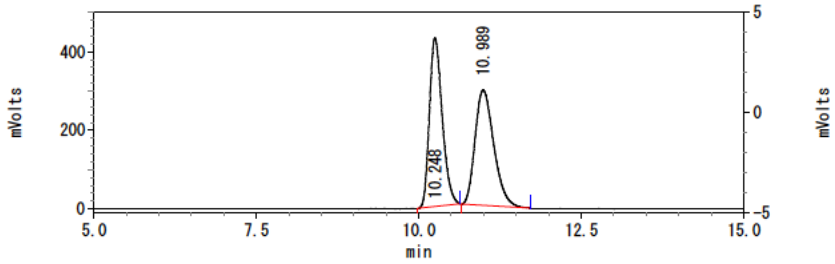

```
Filename          = tn29-5_Carbon-1-3.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn29-5
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 683
Total_Scans       = 683

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.9[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



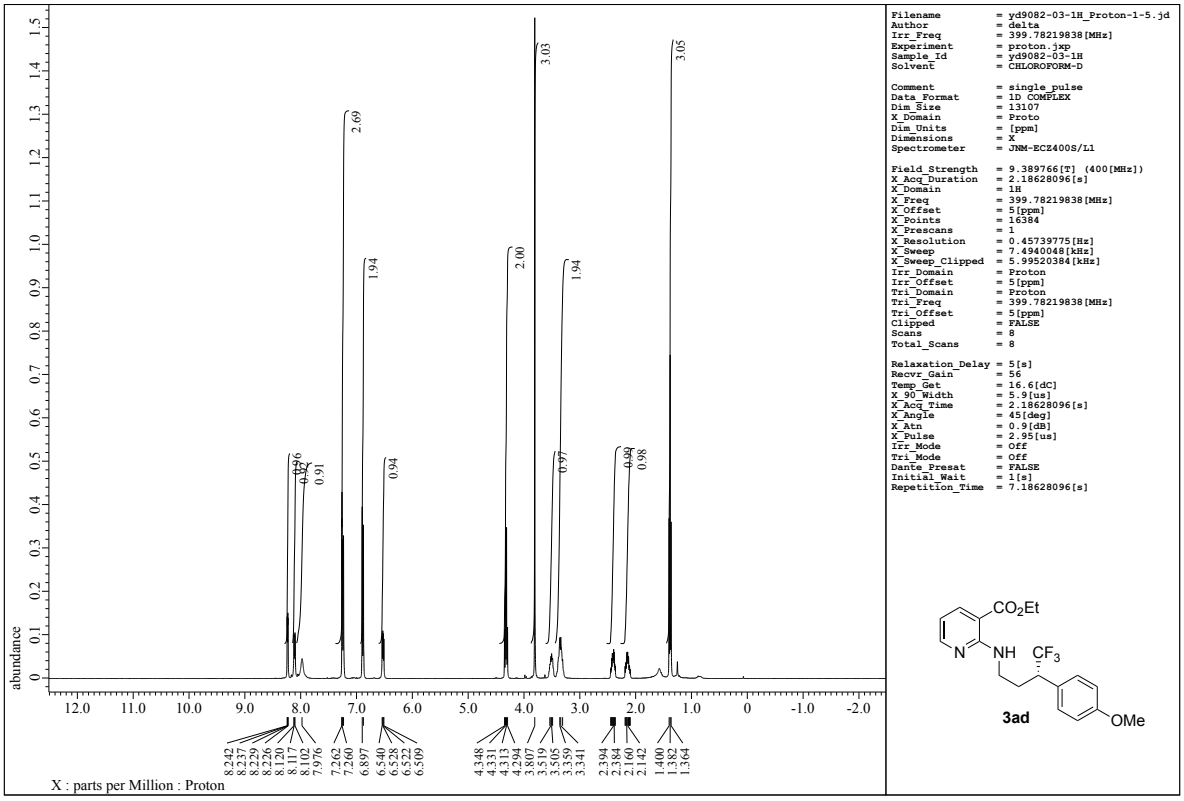
3ac

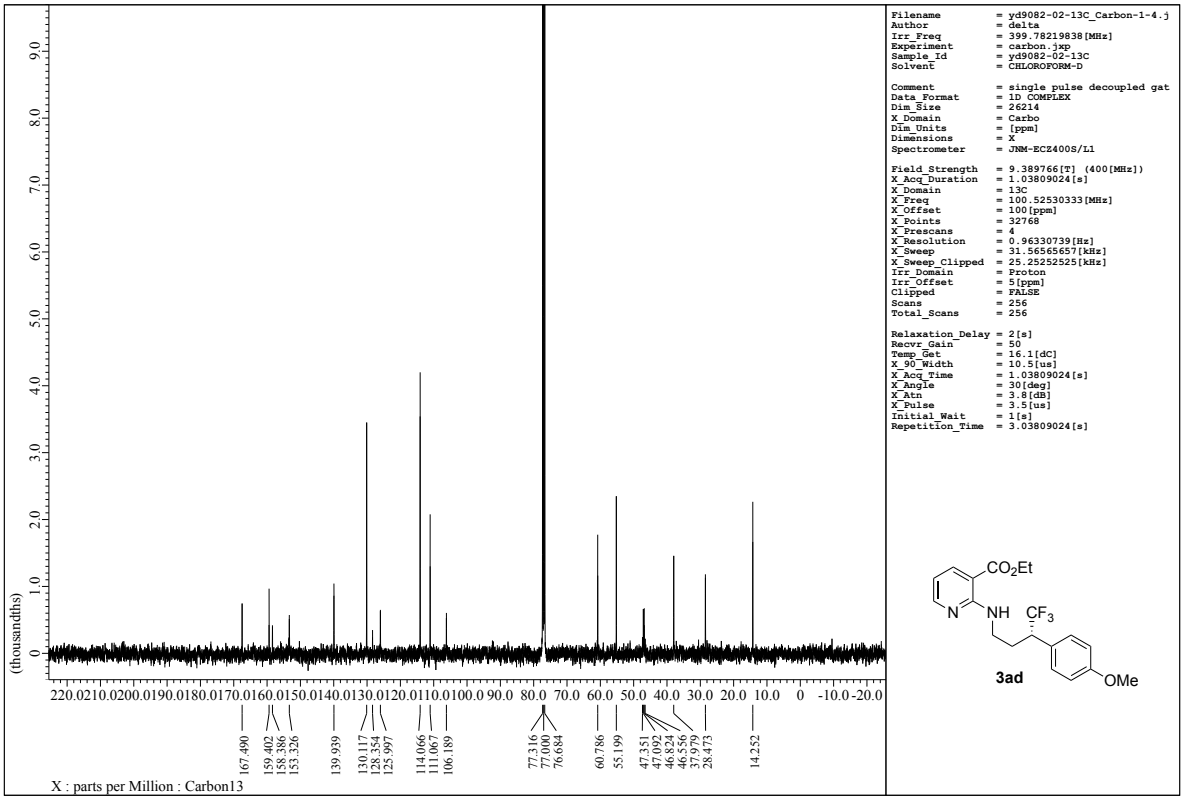


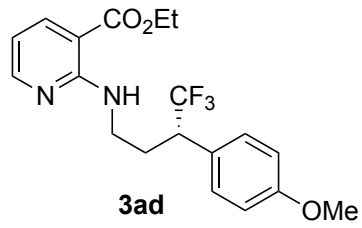
Pk #	Retention Time	Area	Area Percent
1	10.363	327395	6.373
2	11.106	4809571	93.627



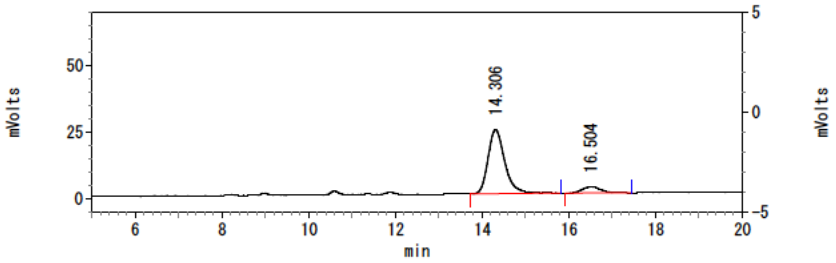
Pk #	Retention Time	Area	Area Percent
1	10.248	5860620	49.868
2	10.989	5891544	50.132


abundance
X : parts per Million : Proton
Filename = yd9082-03-1H_Proton-1-5.jd
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd9082-03-1H
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 16.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CF3
3ad
OMe

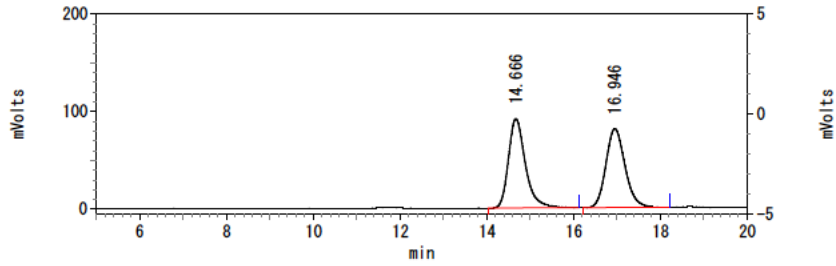

(thousandths)
X : parts per Million : Carbon13
Filename = yd9082-02-13C_Carbon-1-4.j
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = yd9082-02-13C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 16.1[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CF3
3ad
OMe



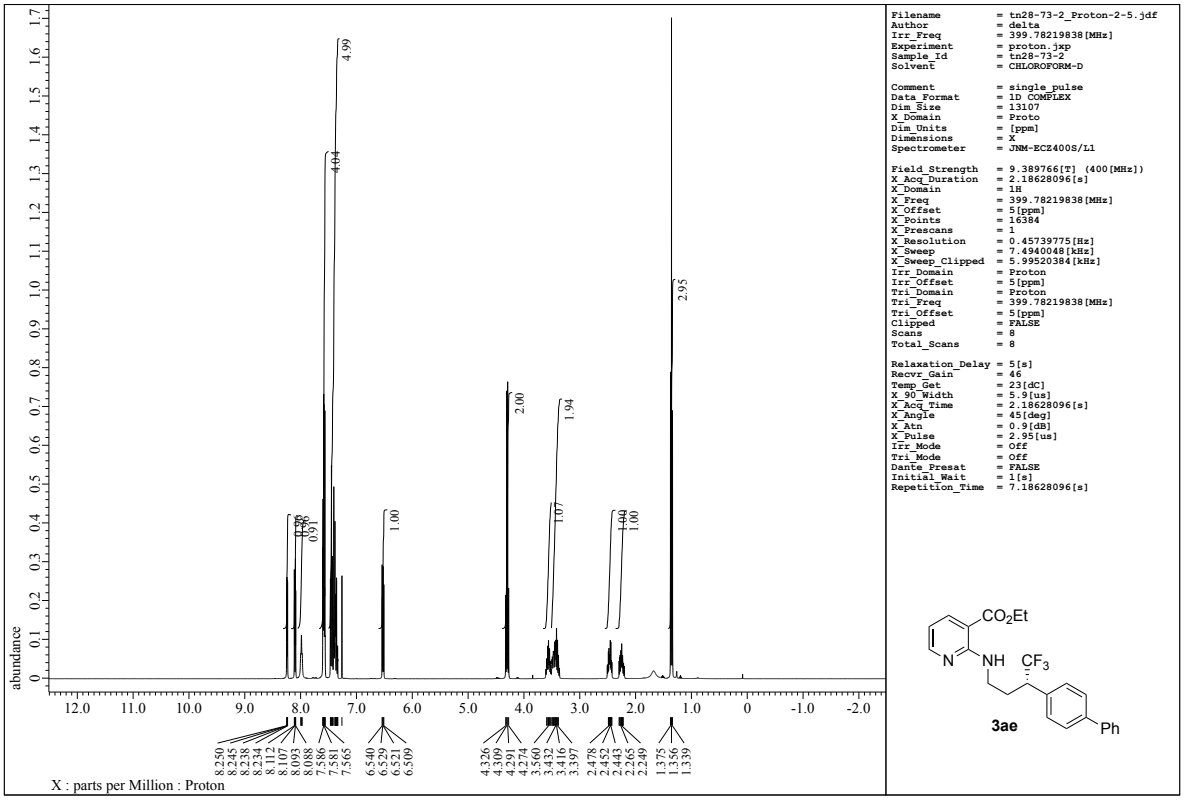
3ad

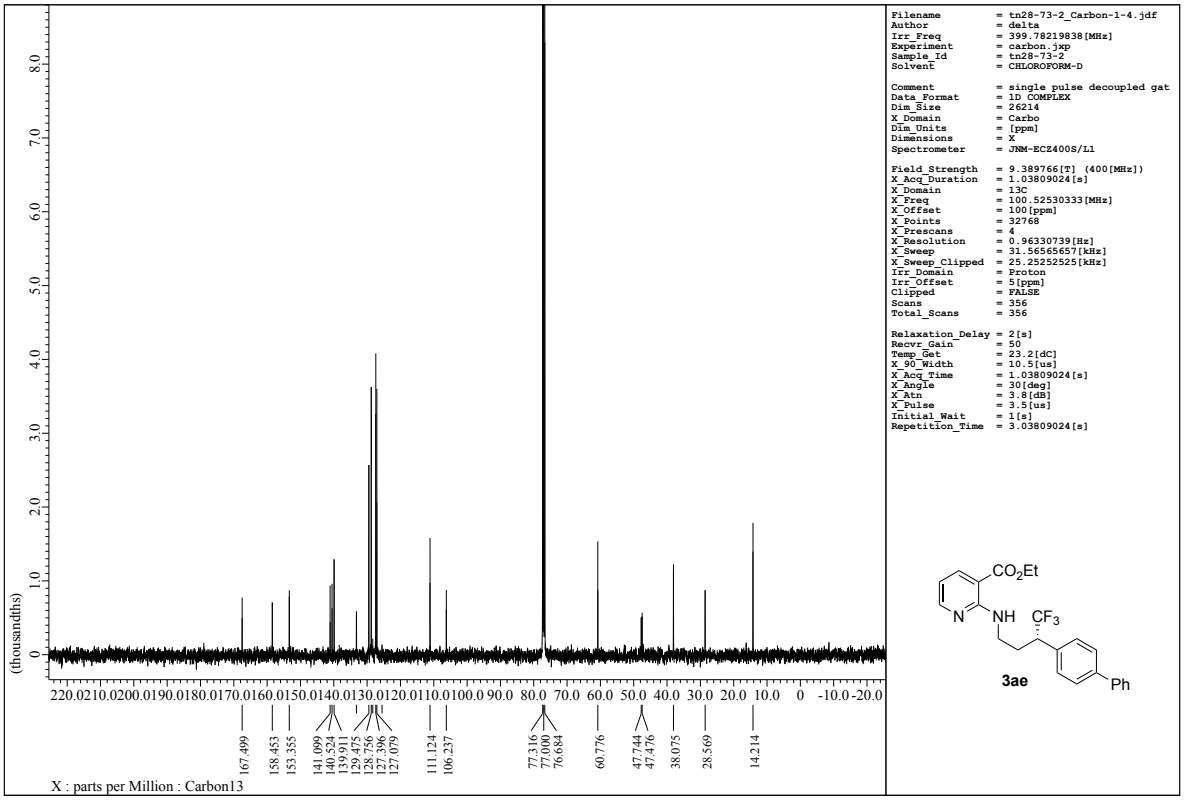


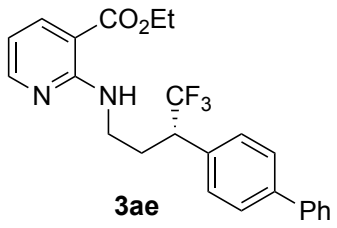
Pk #	Retention Time	Area	Area Percent
1	14.306	647973	90.350
2	16.504	69208	9.650



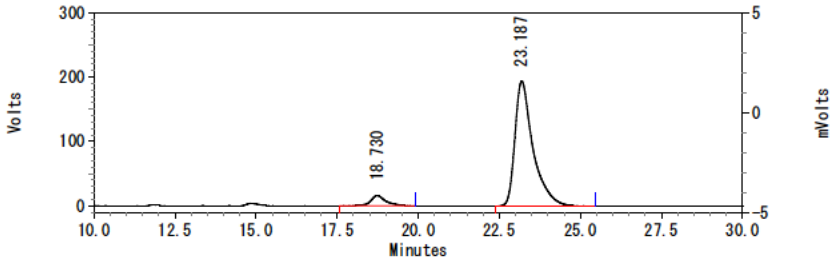
Pk #	Retention Time	Area	Area Percent
1	14.666	2487062	50.155
2	16.946	2471708	49.845


Filename = tn28-73-2_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-73-2
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 23[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
CO2Et
NH
CF3
Ph
3ae


Filename = tn28-73-2_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-73-2
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 356
Total_Scans = 356
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.2[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
CO2Et
NH
CF3
Ph
3ae

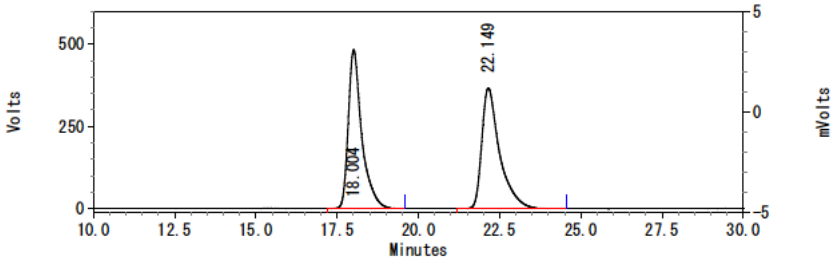


3ae



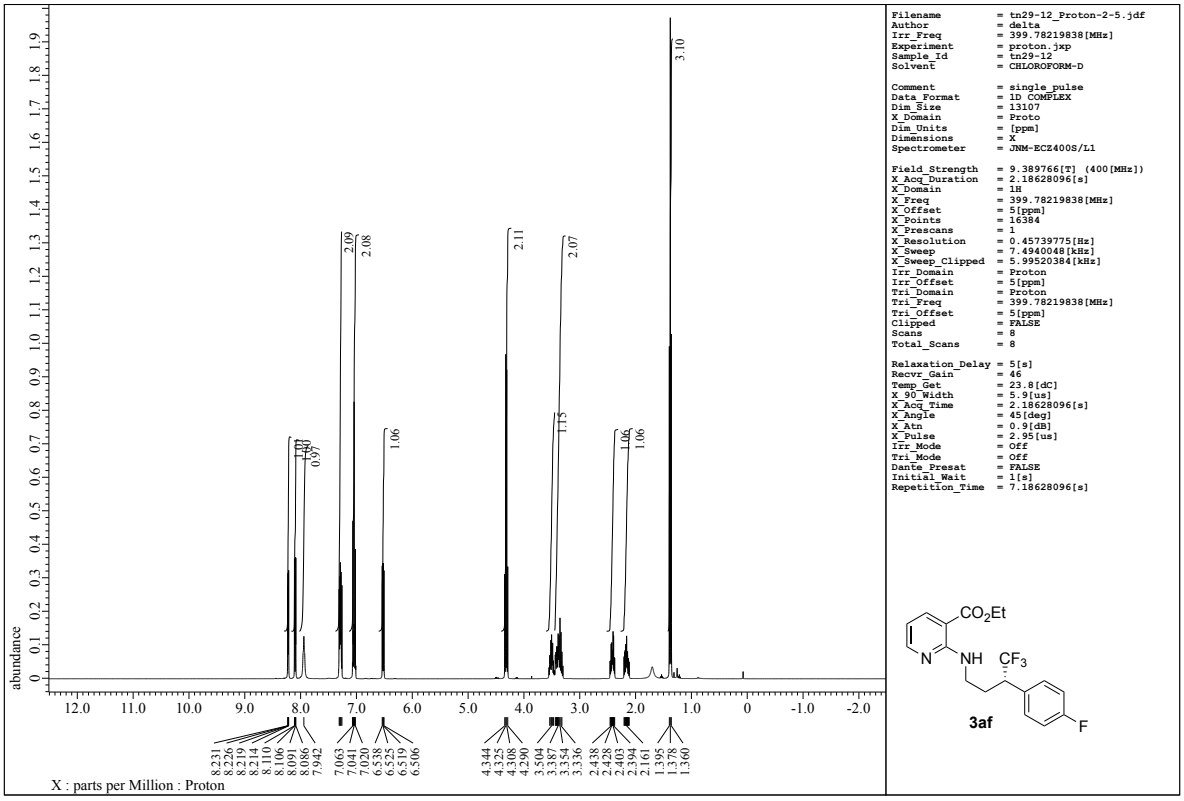
UV Results

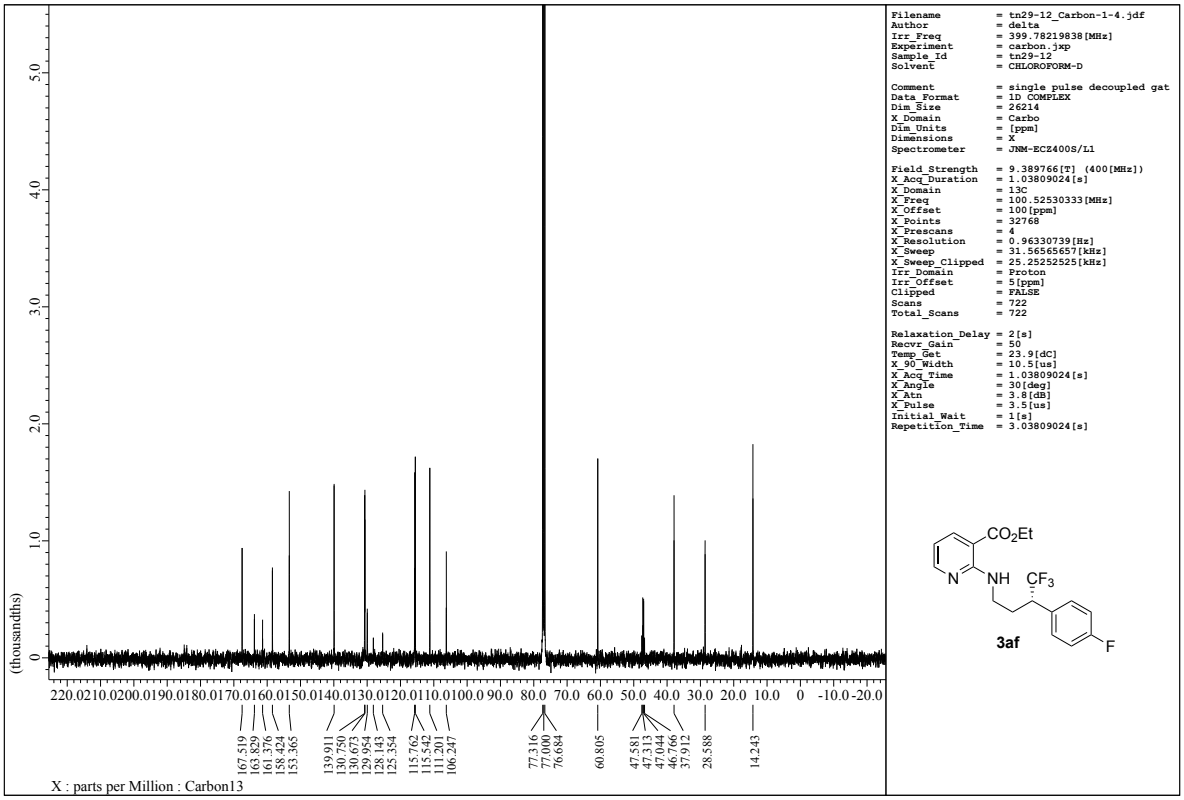
Pk #	Retention Time	Area	Area Percent	Height
1	18.730	516372	6.470	15803
2	23.187	7464398	93.530	193672

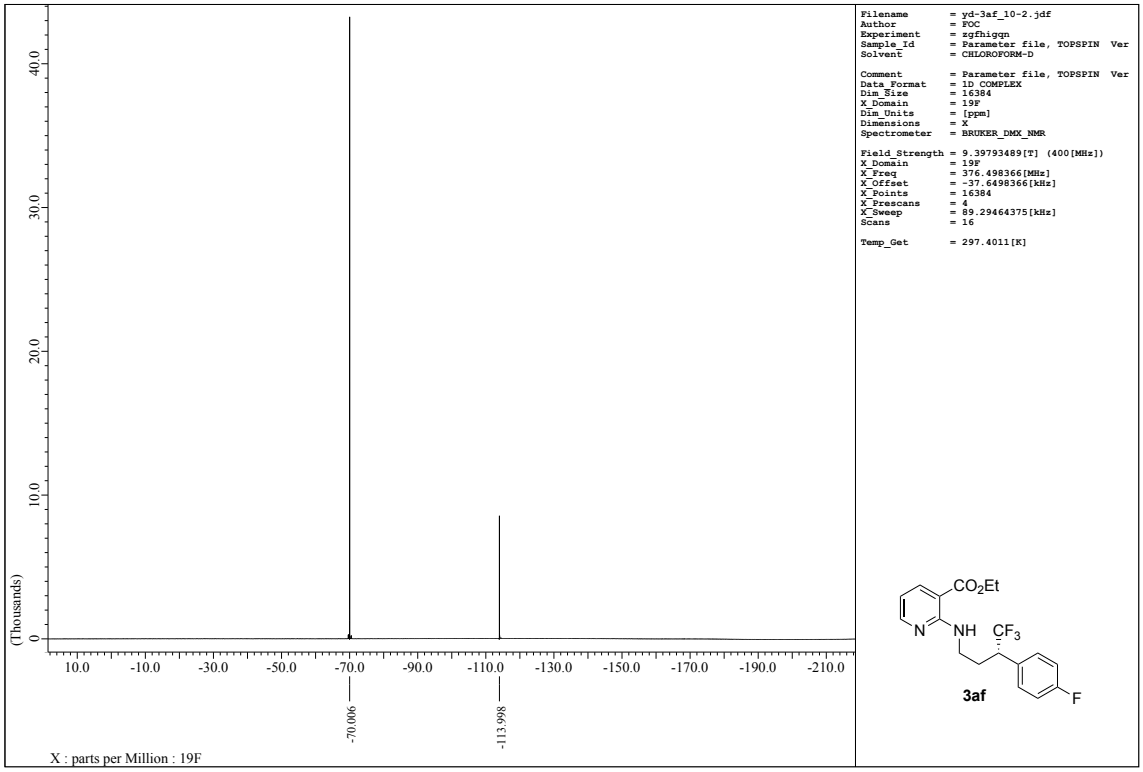


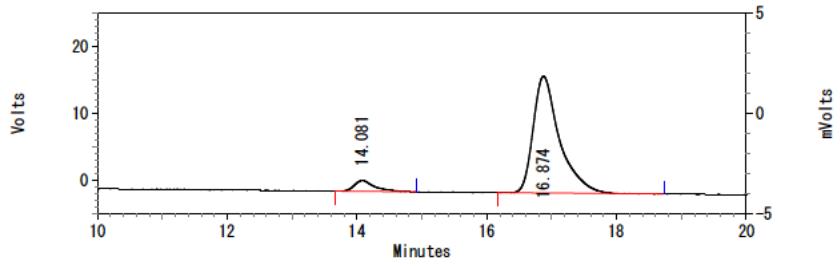
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	18.004	13906205	49.916	482570
2	22.149	13952852	50.084	366932


Filename = tn29-12_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn29-12
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[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 = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
CO2Et
N
NH
CF3
F
3af

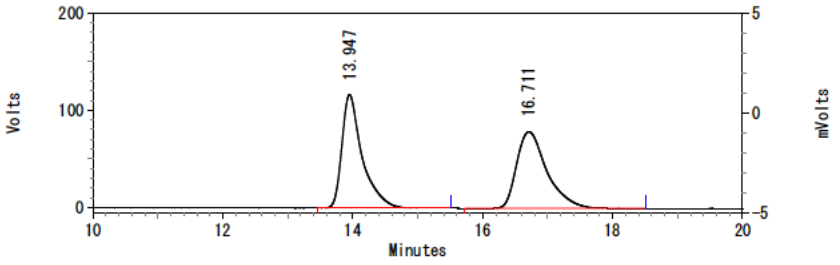

Filename = tn29-12_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn29-12
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 722
Total_Scans = 722
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.9[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
CO2Et
N
NH
CF3
F
3af





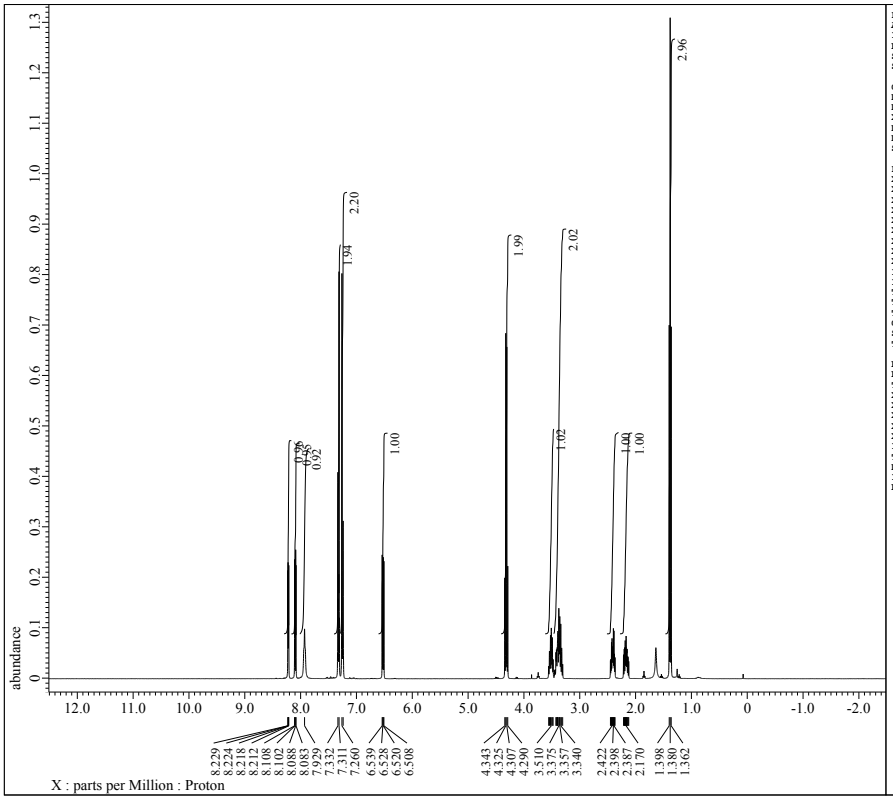
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	14.081	35143	6.666	1608
2	16.874	492034	93.334	17453

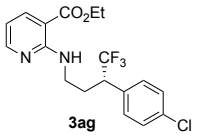


UV Results

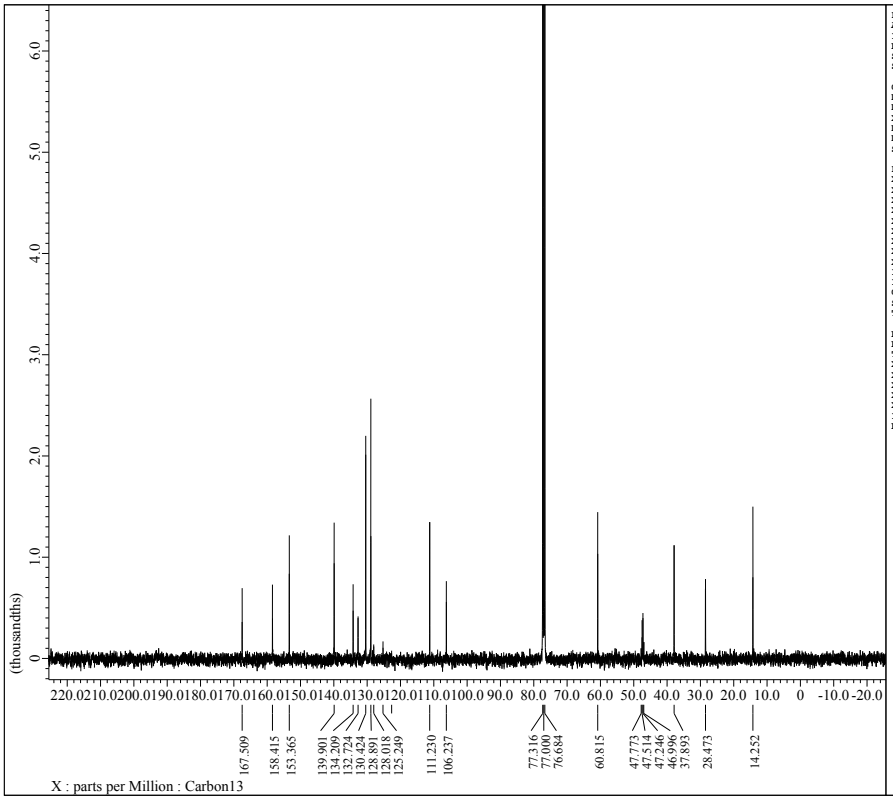
Pk #	Retention Time	Area	Area Percent	Height
1	13.947	2533214	49.956	116560
2	16.711	2537654	50.044	78610



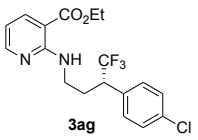

```
Filename          = tn28-86-1_Proton-1-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn28-86-1
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 23.5[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



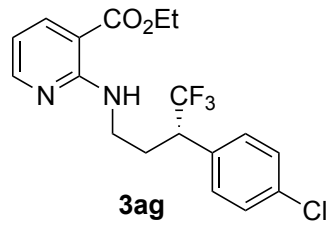
3ag



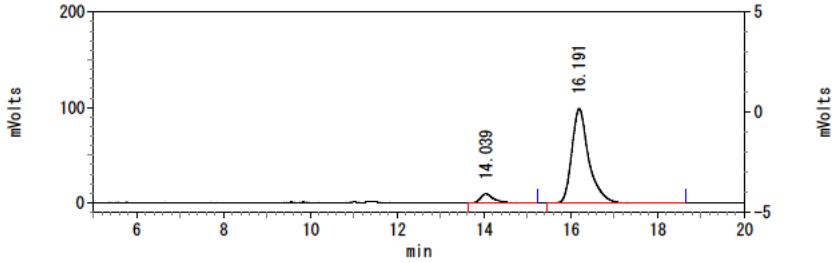

```
Filename          = tn28-86-1_Carbon-1-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn28-86-1
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 720
Total_Scans       = 720

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



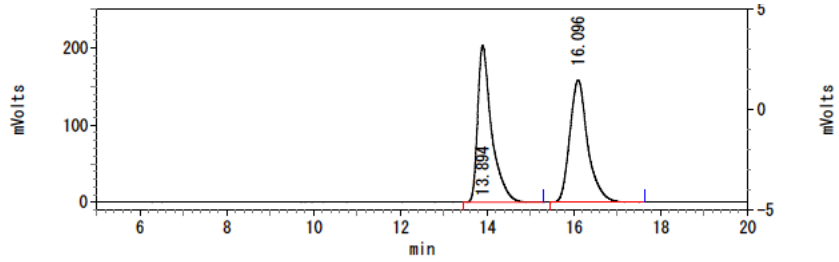
3ag



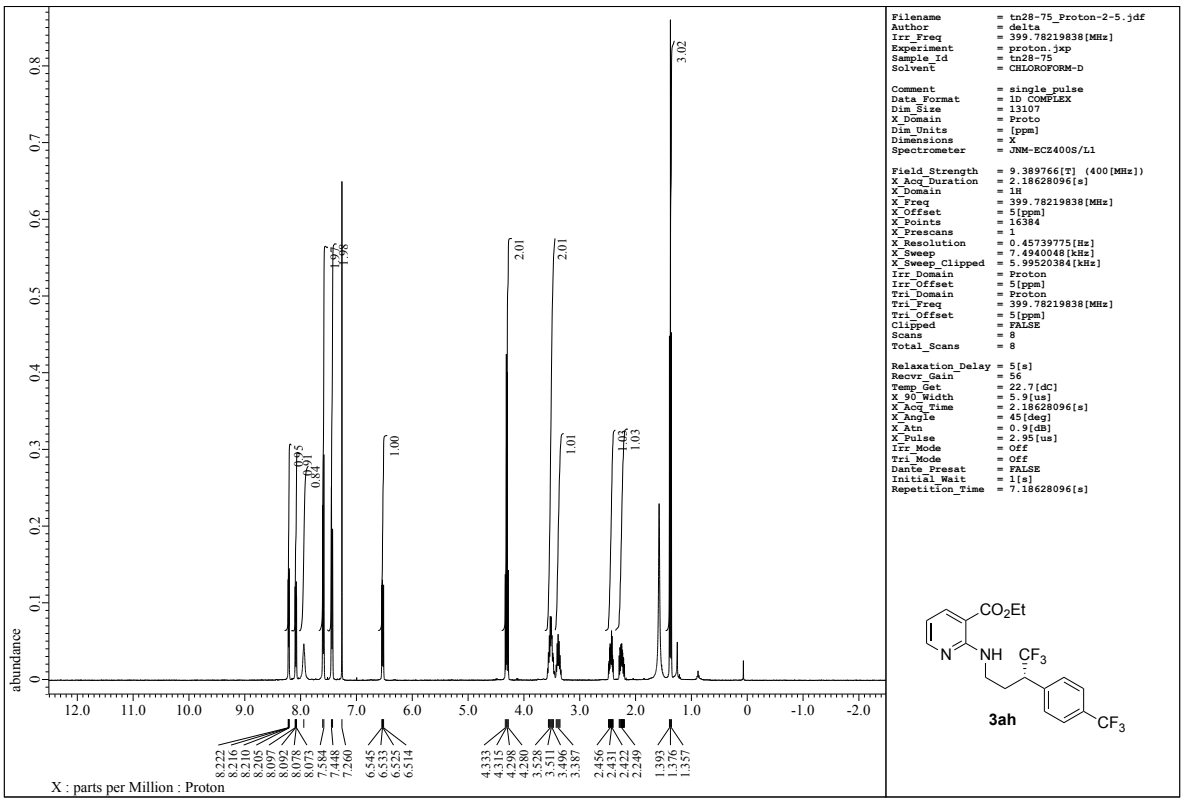
3ag

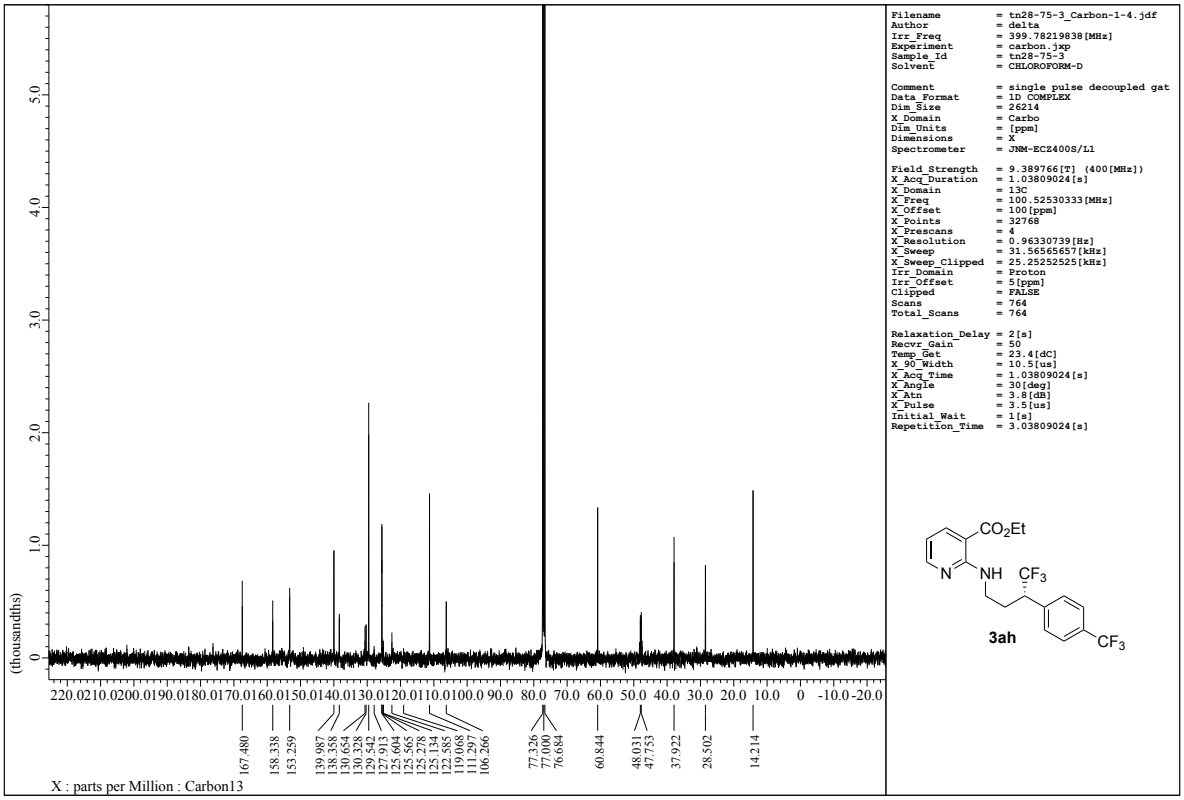


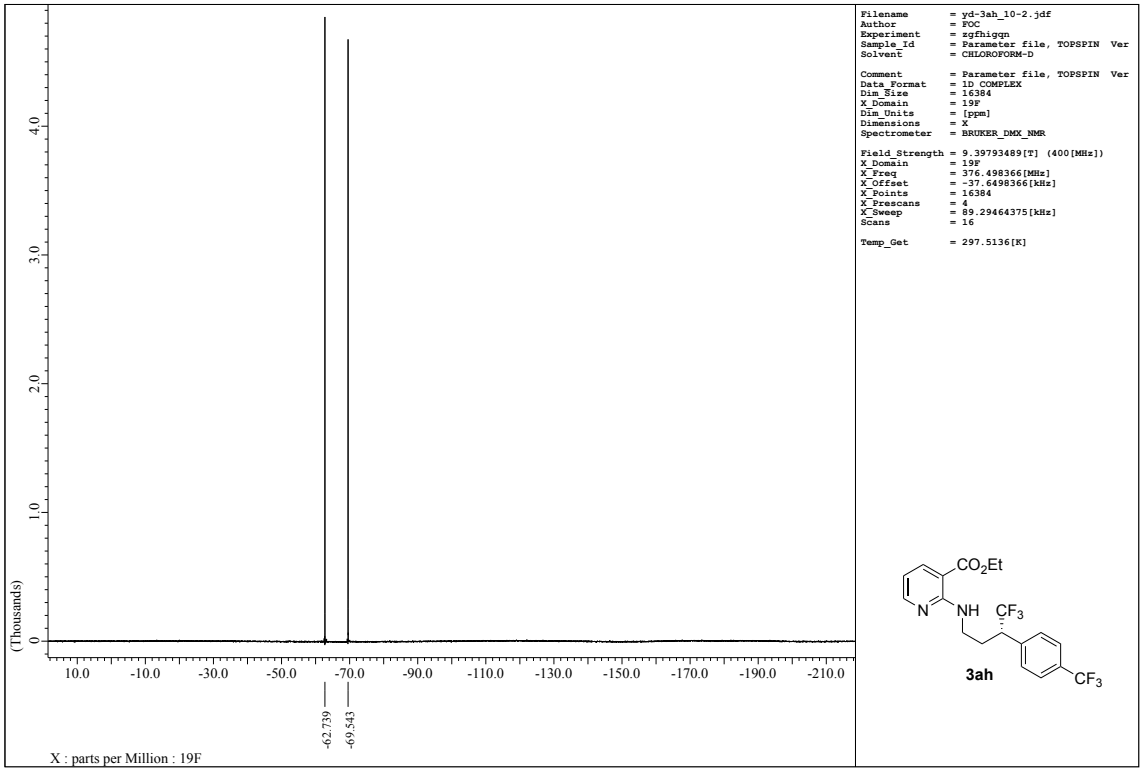
Pk #	Retention Time	Area	Area Percent
1	14.039	205081	7.113
2	16.191	2678057	92.887

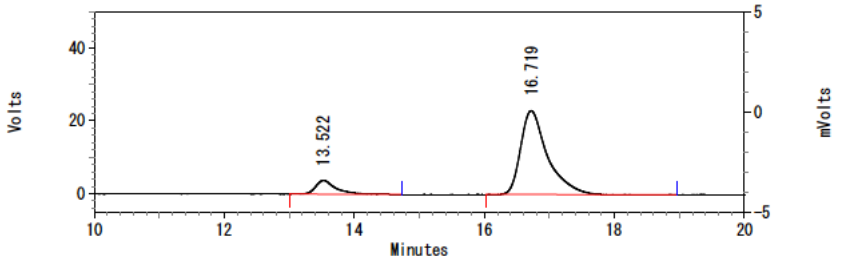


Pk #	Retention Time	Area	Area Percent
1	13.894	4525200	49.885
2	16.096	4546123	50.115


abundance
X : parts per Million : Proton
Filename = tn28-75_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-75
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.7[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
NH
CF3
3ah

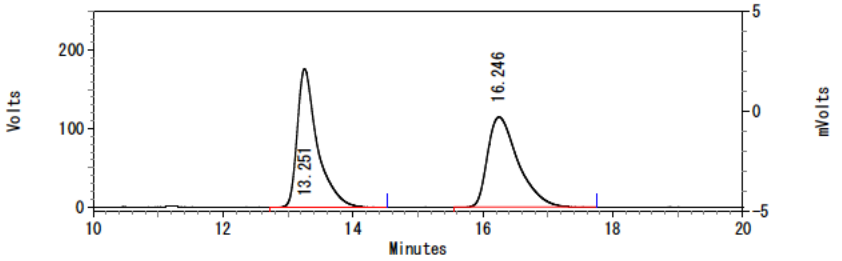

(thousandths)
X : parts per Million : Carbon13
Filename = tn28-75-3_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-75-3
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 764
Total_Scans = 764
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.4[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
NH
CF3
3ah





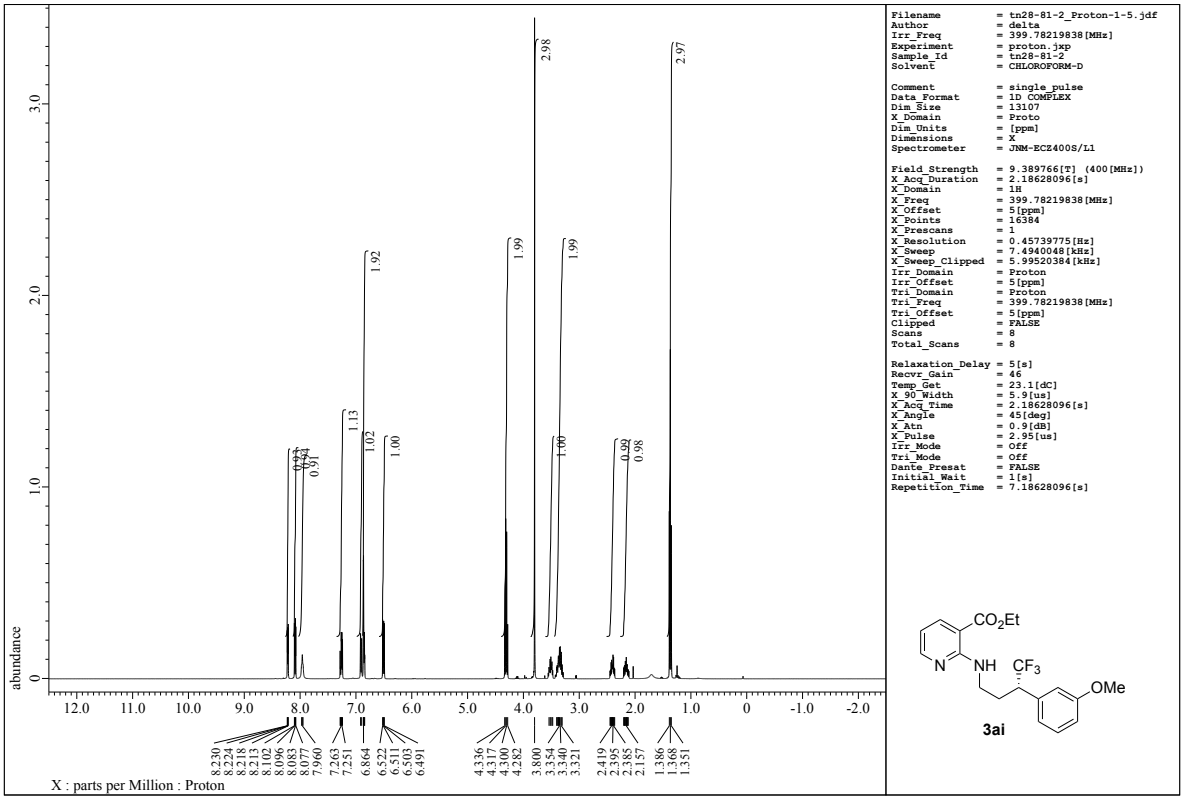
UV Results

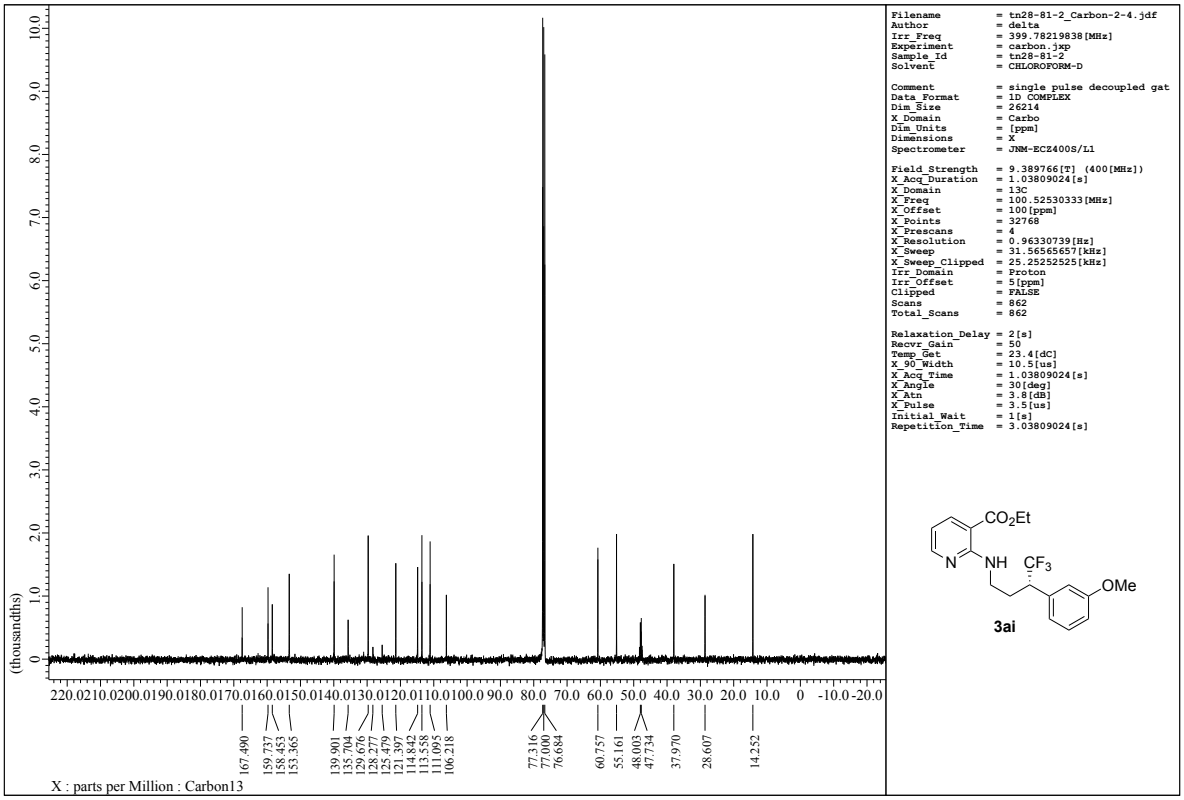
Pk #	Retention Time	Area	Area Percent	Height
1	13.522	81146	10.933	3769
2	16.719	661086	89.067	23005

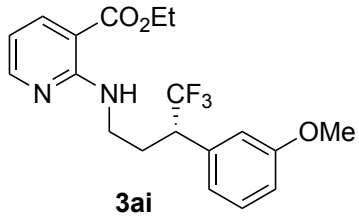


UV Results

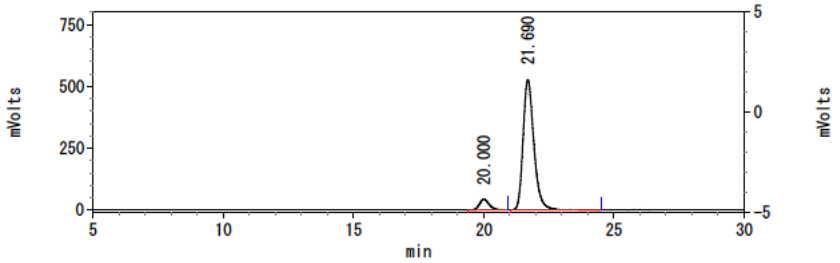
Pk #	Retention Time	Area	Area Percent	Height
1	13.251	3762136	50.175	176133
2	16.246	3735921	49.825	114577


abundance
X : parts per Million : Proton
Filename = tn28-81-2_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-81-2
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 23.1[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CF3
OMe
3ai

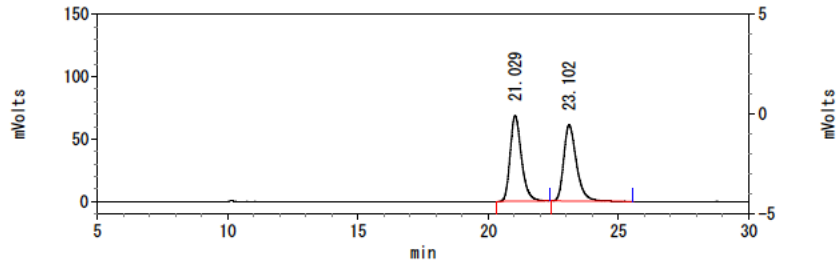

(thousandths)
X : parts per Million : Carbon13
Filename = tn28-81-2_Carbon-2-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-81-2
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 862
Total_Scans = 862
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.4[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CF3
OMe
3ai



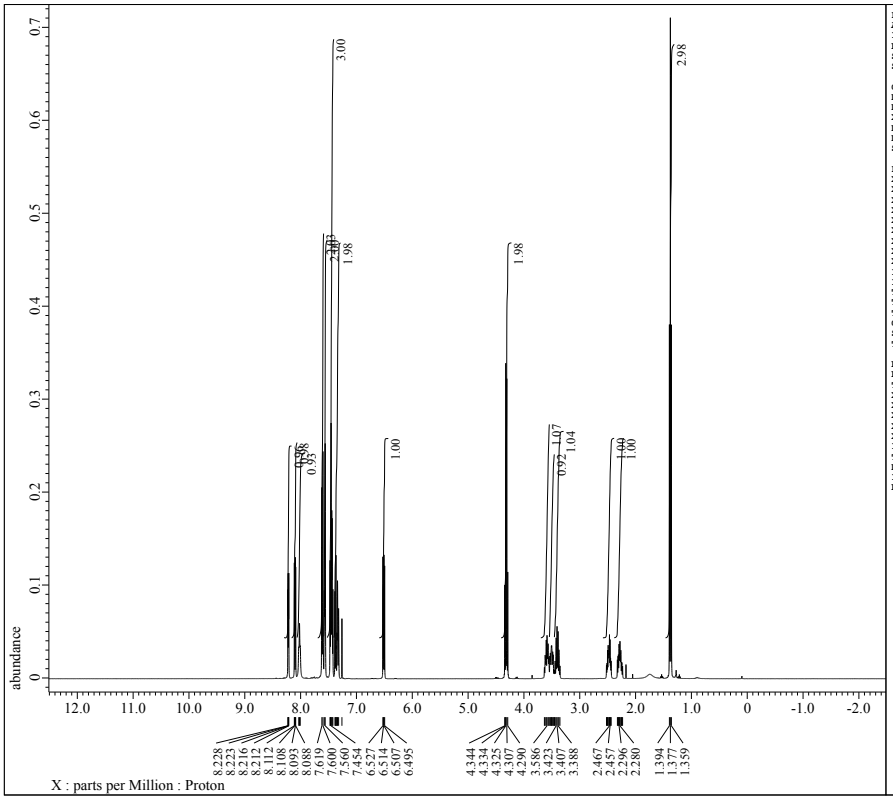
3ai



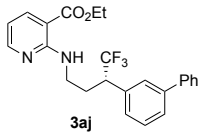
Pk #	Retention Time	Area	Area Percent
1	20.000	1111825	6.830
2	21.690	15167840	93.170



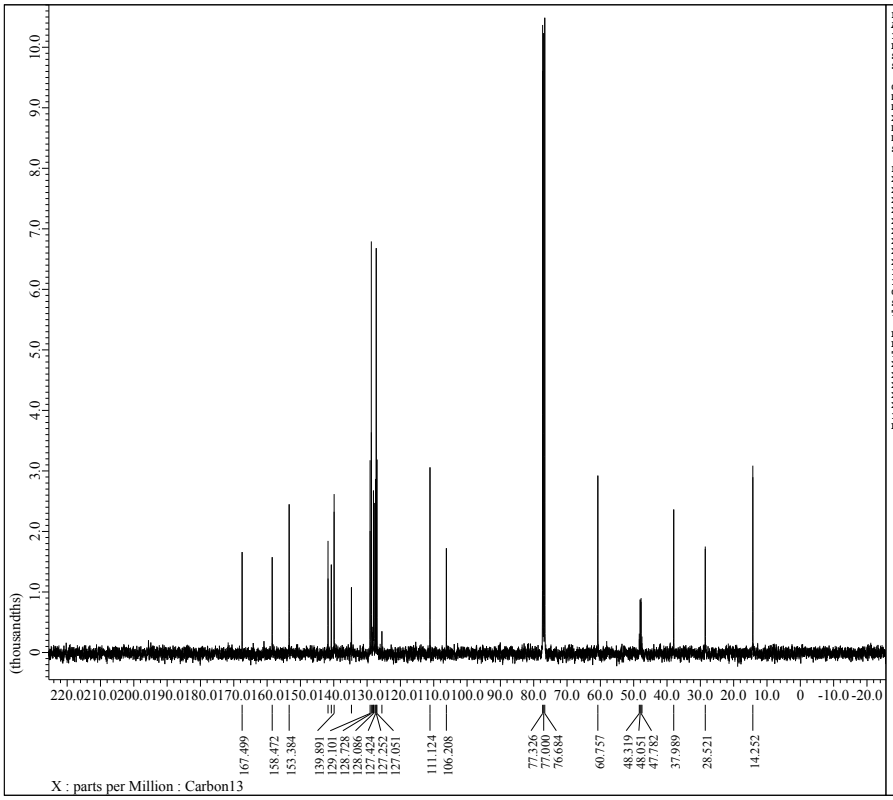
Pk #	Retention Time	Area	Area Percent
1	21.029	2070126	50.000
2	23.102	2070145	50.000



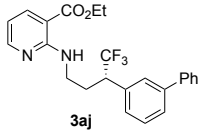

```
Filename          = tn28-68-2_Proton-2-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn28-68-2
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 23.3[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



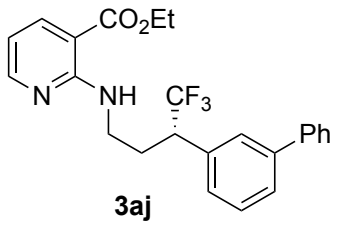
3aj



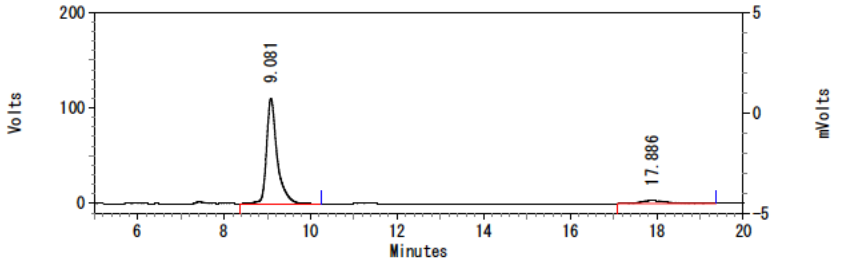

```
Filename          = tn28-68-2_Carbon-1-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn28-68-2
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 23.4[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



3aj

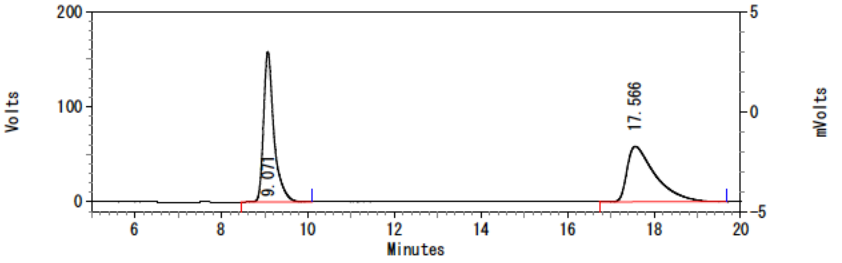


3aj



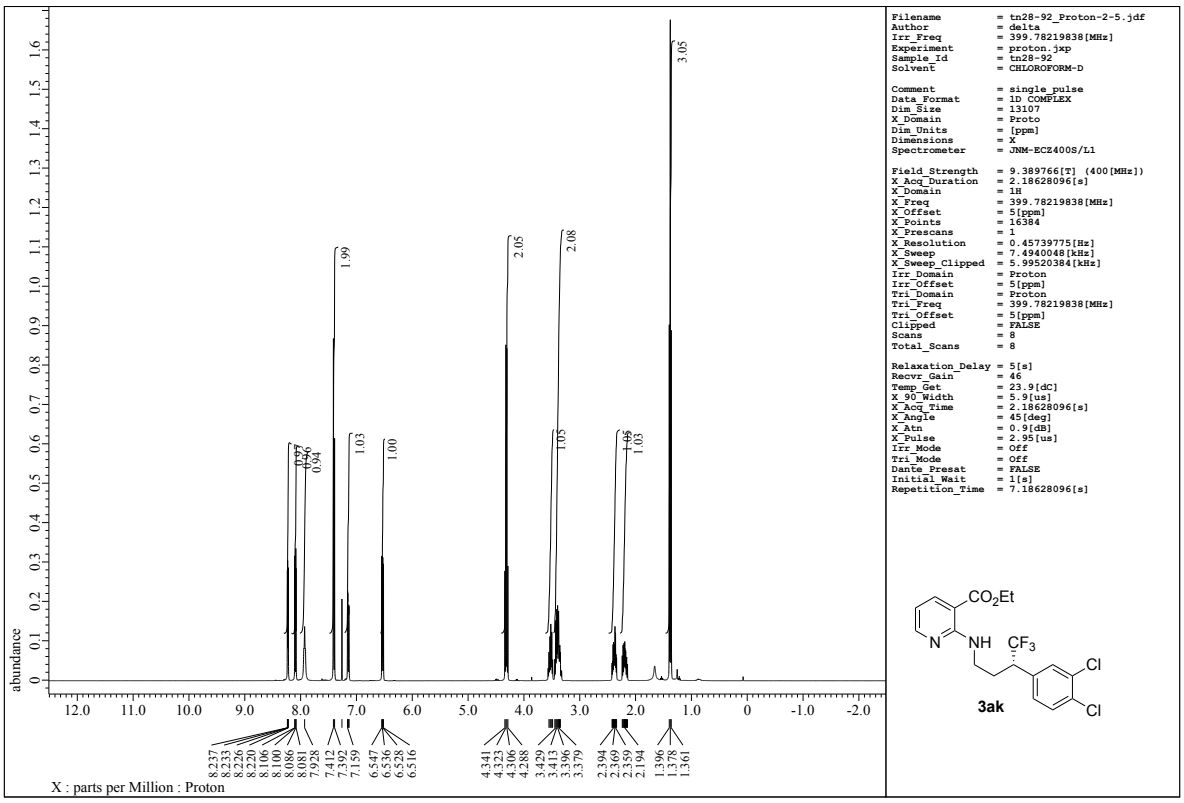
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	9.081	1858309	93.765	109845
2	17.886	123574	6.235	2999

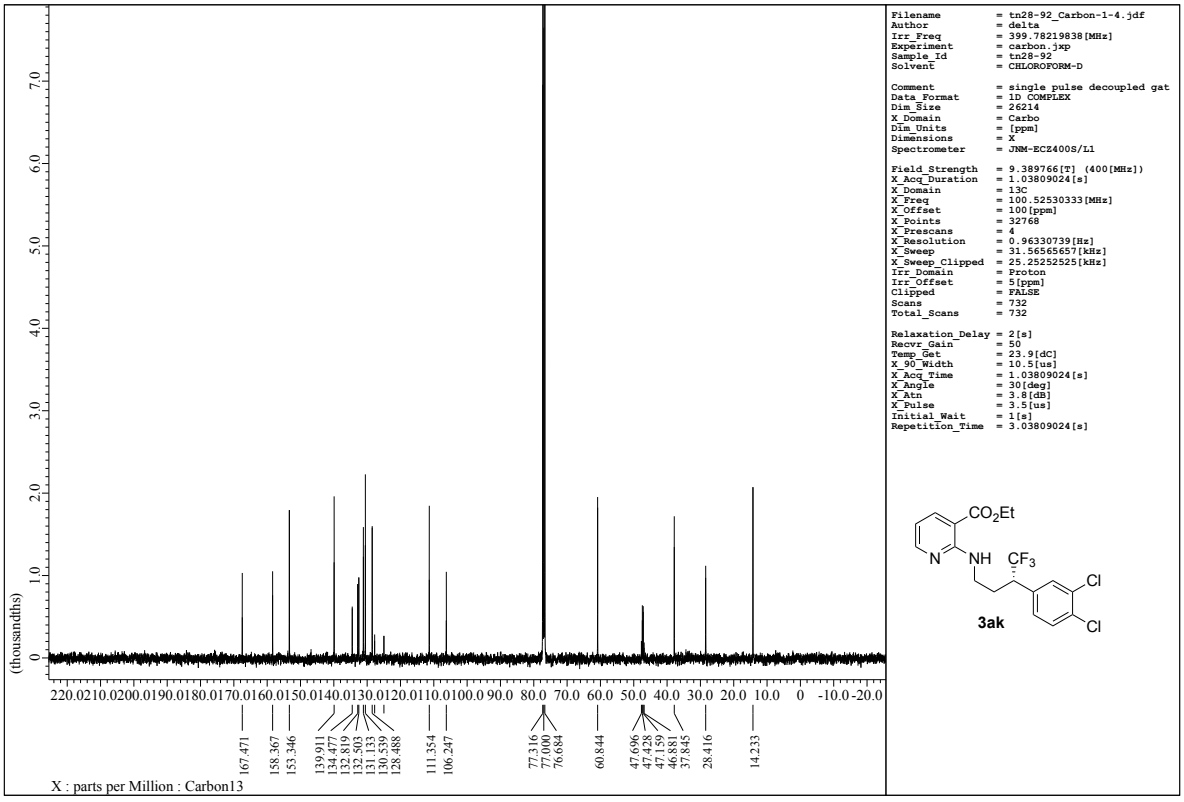


UV Results

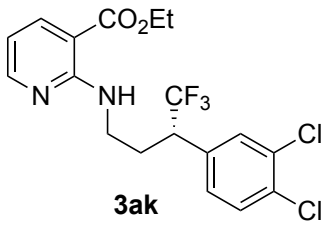
Pk #	Retention Time	Area	Area Percent	Height
1	9.071	2632745	50.049	157776
2	17.566	2627640	49.951	58208



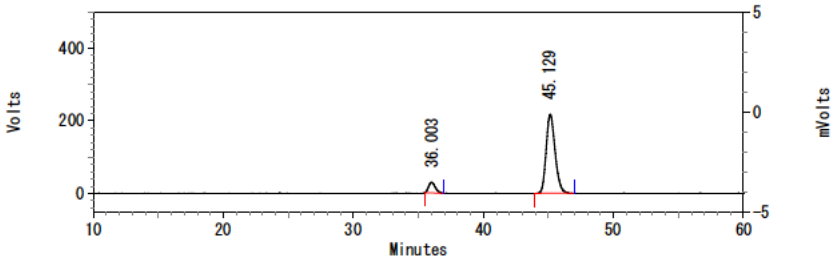

```
Filename          = tn28-92_Proton-2-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn28-92
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 23.9[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```




```
Filename          = tn28-92_Carbon-1-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn28-92
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 732
Total_Scans       = 732

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 23.9[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```

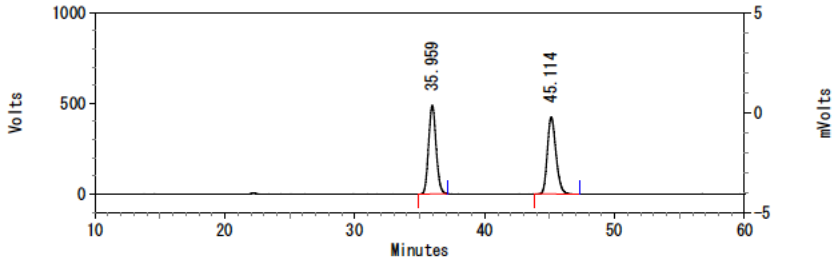


3ak



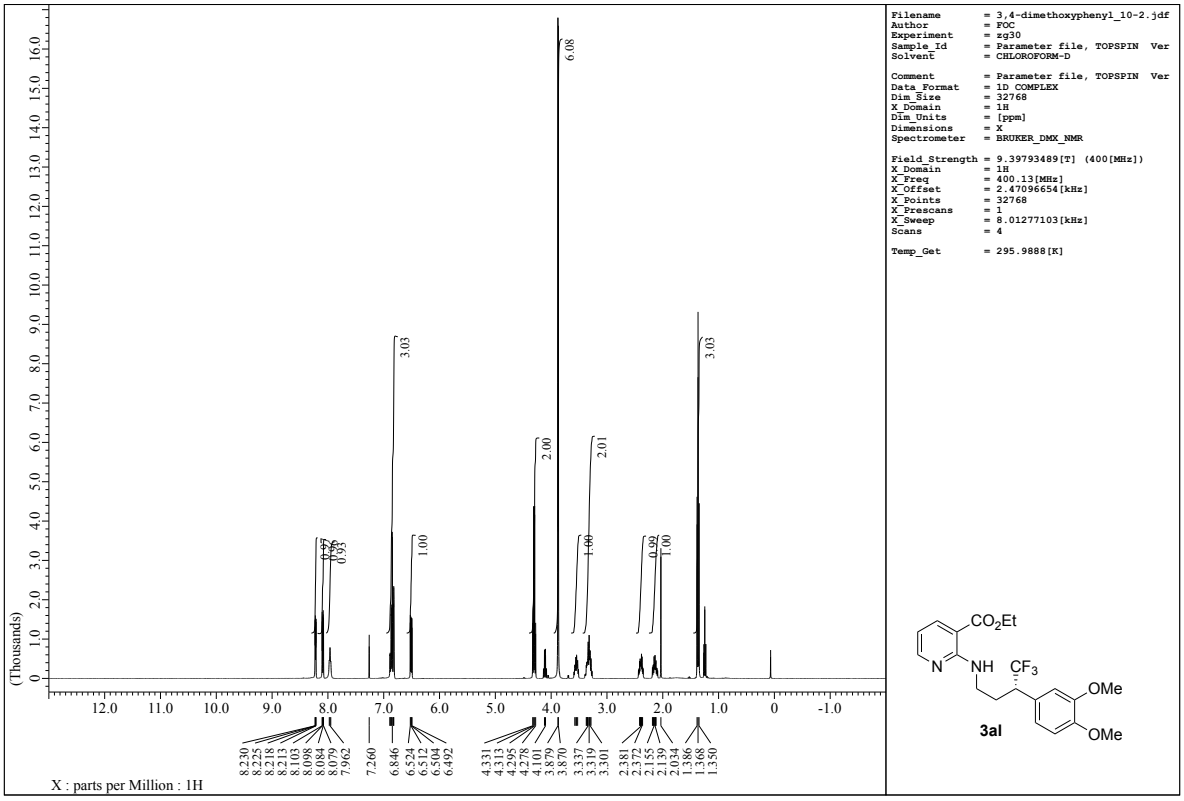
UV-970 Results

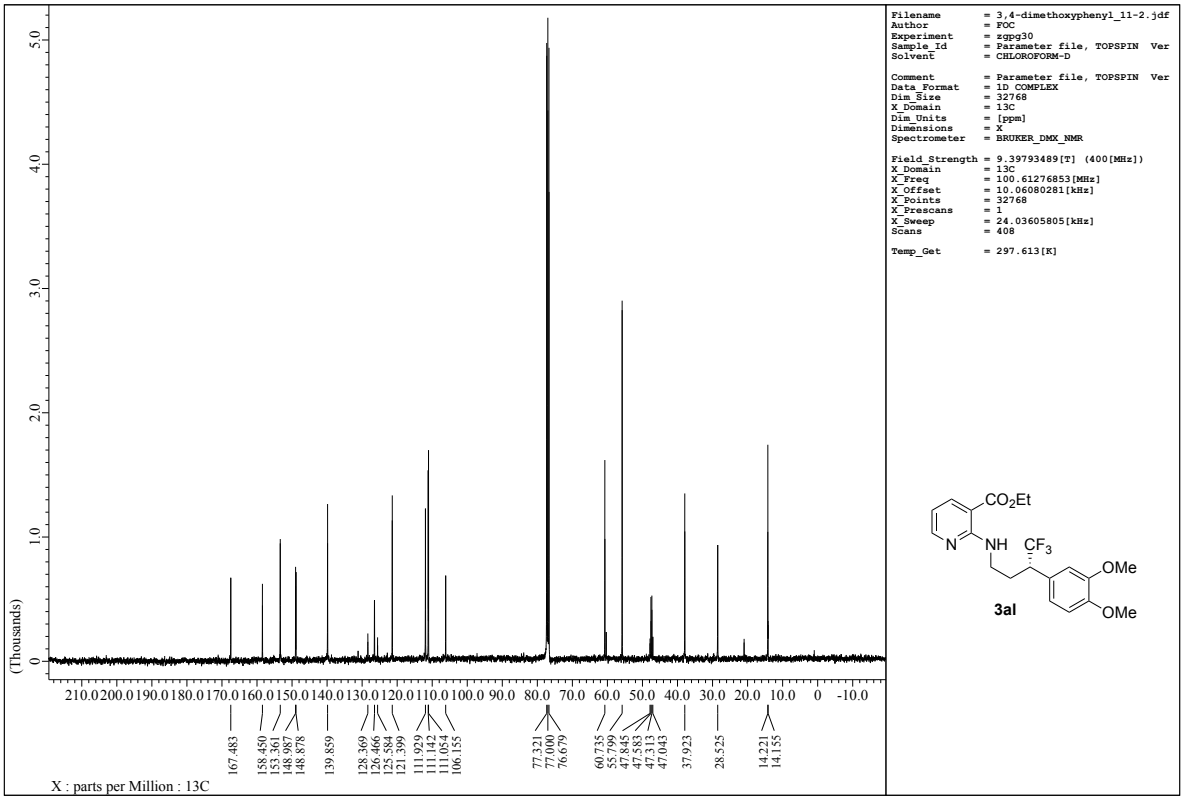
Pk #	Retention Time	Area	Area Percent	Height
1	36.003	1041508	9.135	29273
2	45.129	10360026	90.865	217143

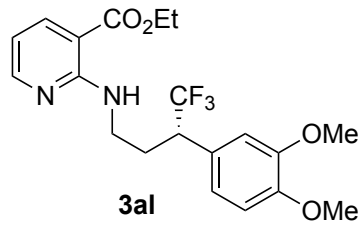


UV-970 Results

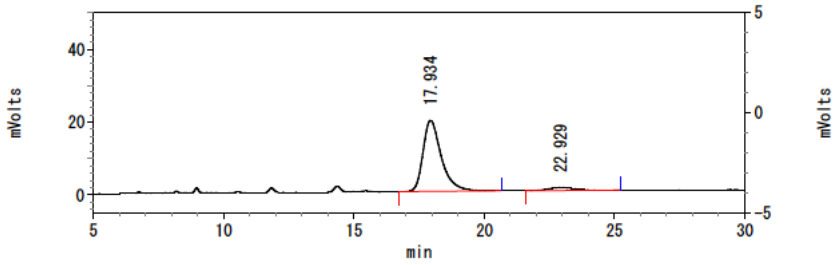
Pk #	Retention Time	Area	Area Percent	Height
1	35.959	19636609	50.179	486850
2	45.114	19496433	49.821	422811


Filename = 3,4-dimethoxyphenyl_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 295.9888[K]
(Thousands)
X : parts per Million : 1H
CO2Et
NH
CF3
OMe
OMe
3al

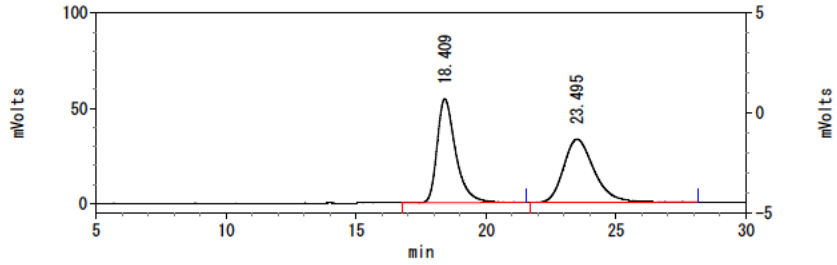

Filename = 3,4-dimethoxyphenyl_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 408
Temp_Get = 297.613[K]
(Thousands)
X : parts per Million : 13C
CO2Et
NH
CF3
OMe
OMe
3al



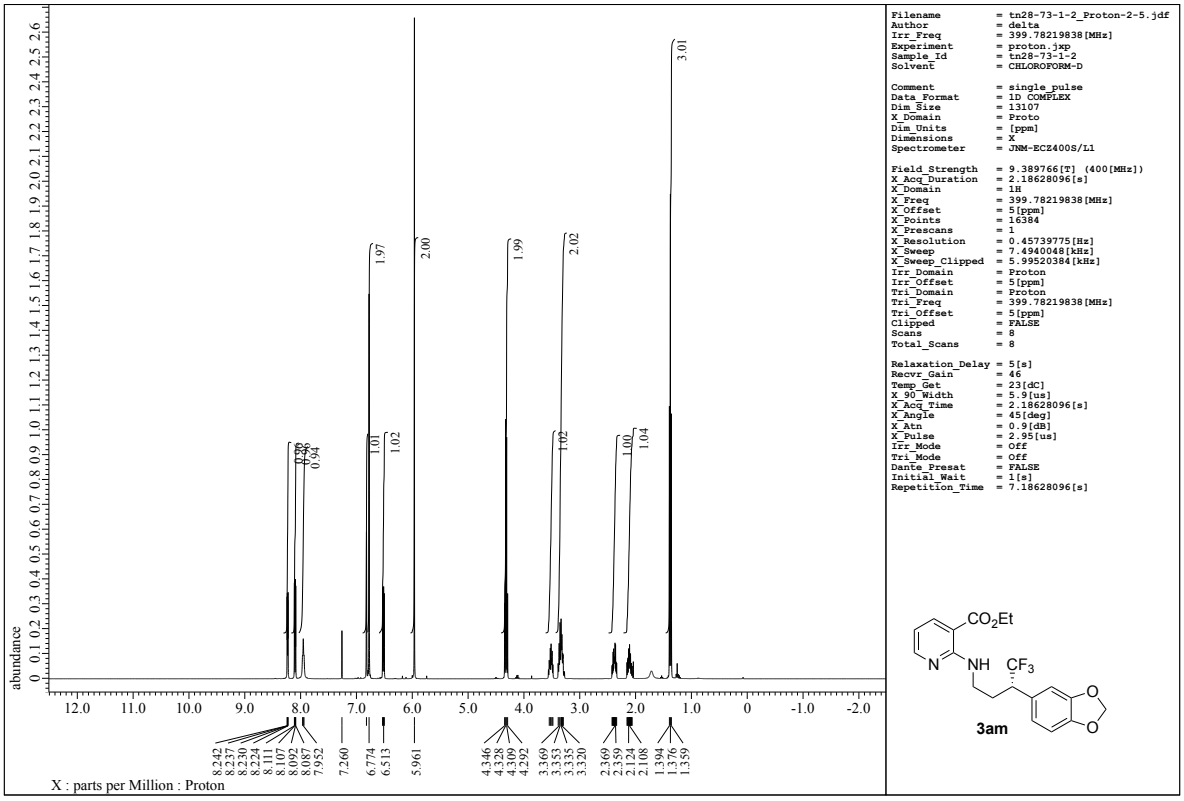
3al

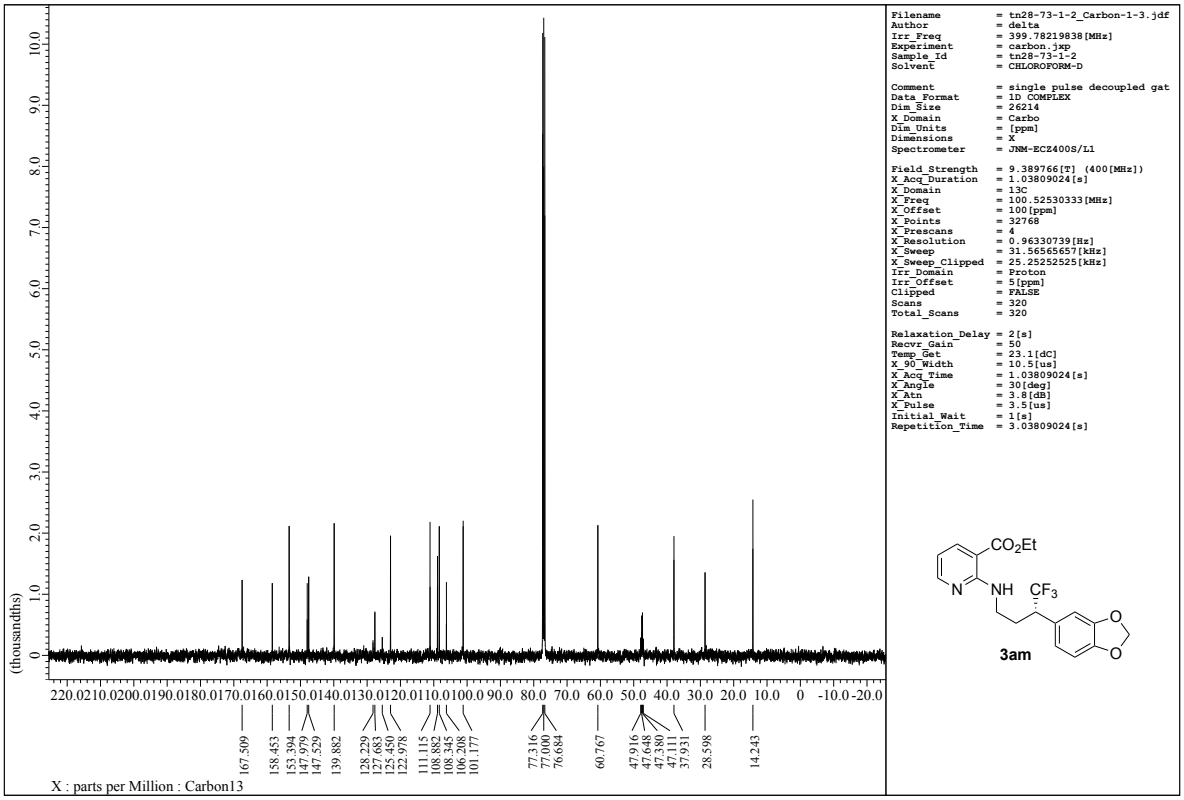


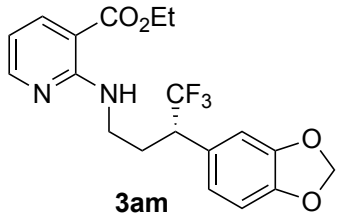
Pk #	Retention Time	Area	Area Percent
1	17.934	960489	93.572
2	22.929	65987	6.428



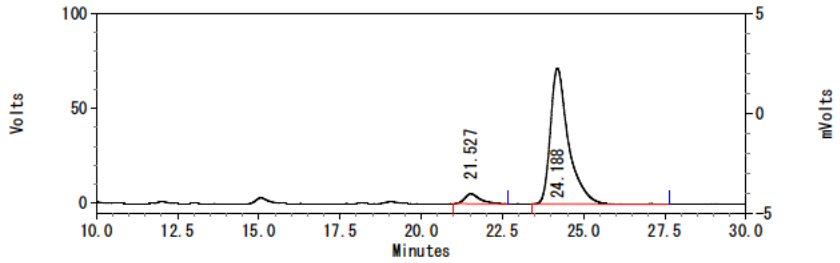
Pk #	Retention Time	Area	Area Percent
1	18.409	2705054	49.930
2	23.495	2712650	50.070


abundance
X : parts per Million : Proton
Filename = tn28-73-1-2_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-73-1-2
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 23[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CF3
O
O
3am


(thousandths)
X : parts per Million : Carbon13
Filename = tn28-73-1-2_Carbon-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-73-1-2
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 320
Total_Scans = 320
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.1[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CF3
O
O
3am

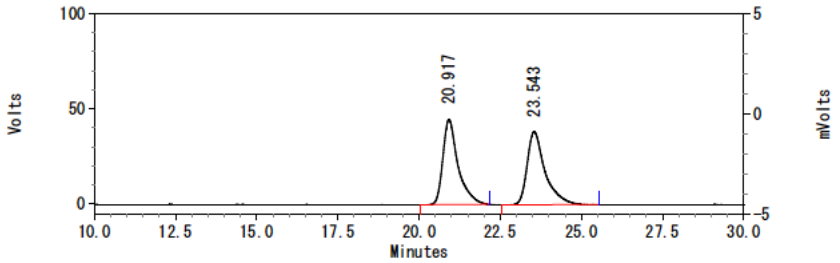


3am



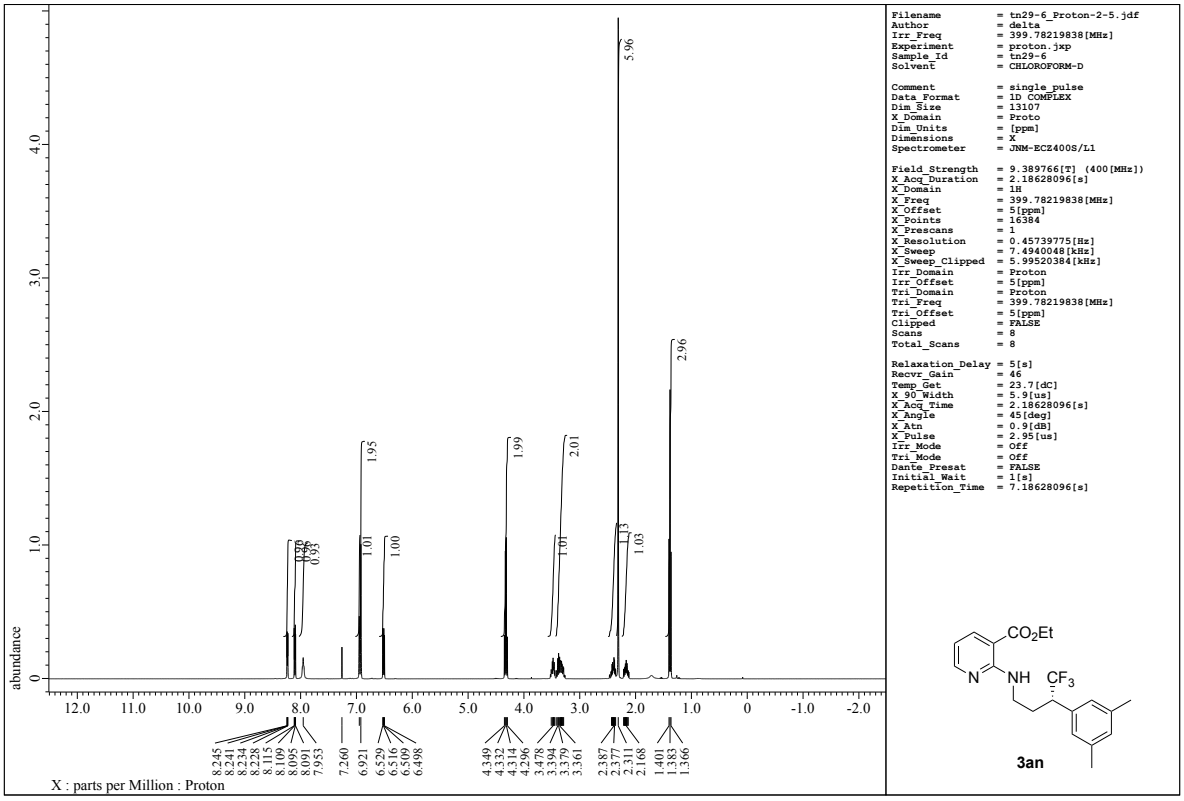
UV Results

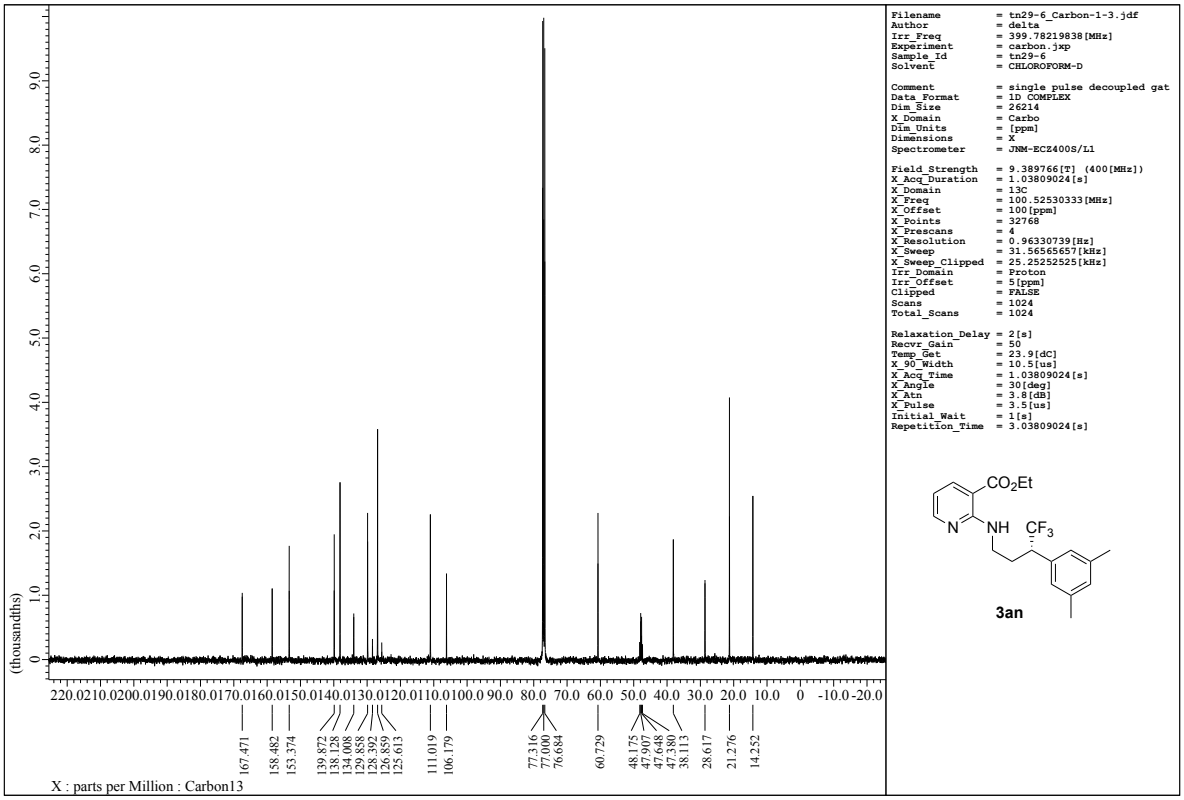
Pk #	Retention Time	Area	Area Percent	Height
1	21.527	168714	5.790	5134
2	24.188	2745303	94.210	71401

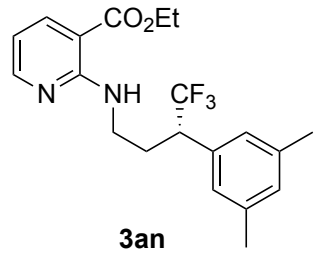


UV Results

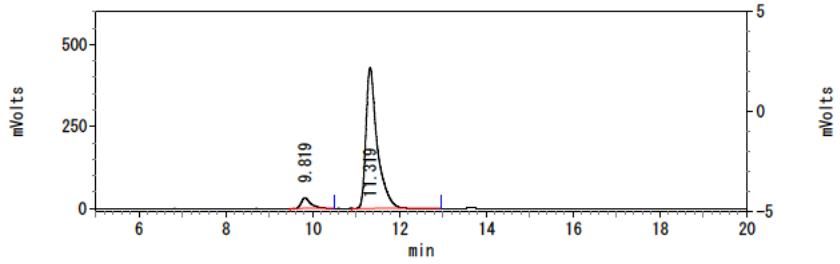
Pk #	Retention Time	Area	Area Percent	Height
1	20.917	1453333	49.763	44674
2	23.543	1467191	50.237	38274


Filename = tn29-6_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn29-6
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 23.7[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
CO2Et
N
NH
CF3
3an

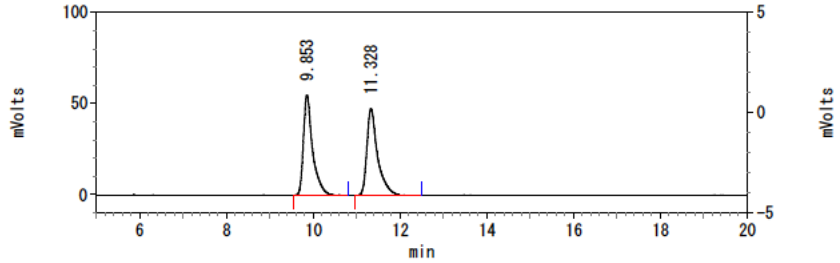

Filename = tn29-6_Carbon-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn29-6
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.9[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
CO2Et
N
NH
CF3
3an



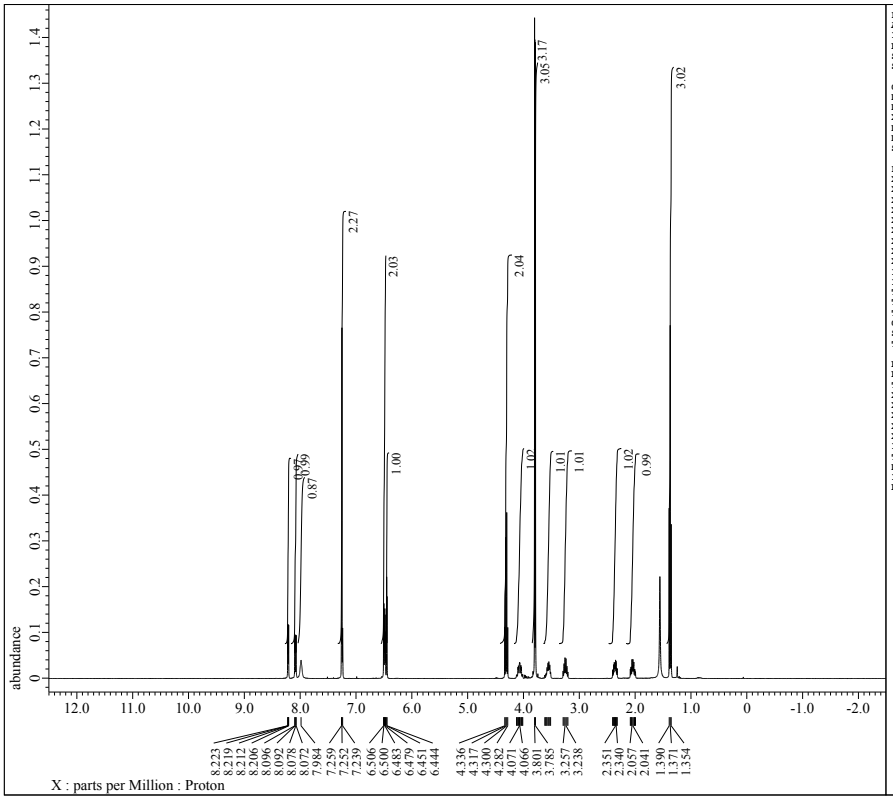
3an



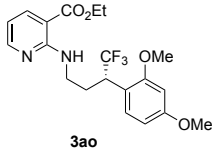
Pk #	Retention Time	Area	Area Percent
1	9.819	482077	5.859
2	11.319	7745573	94.141



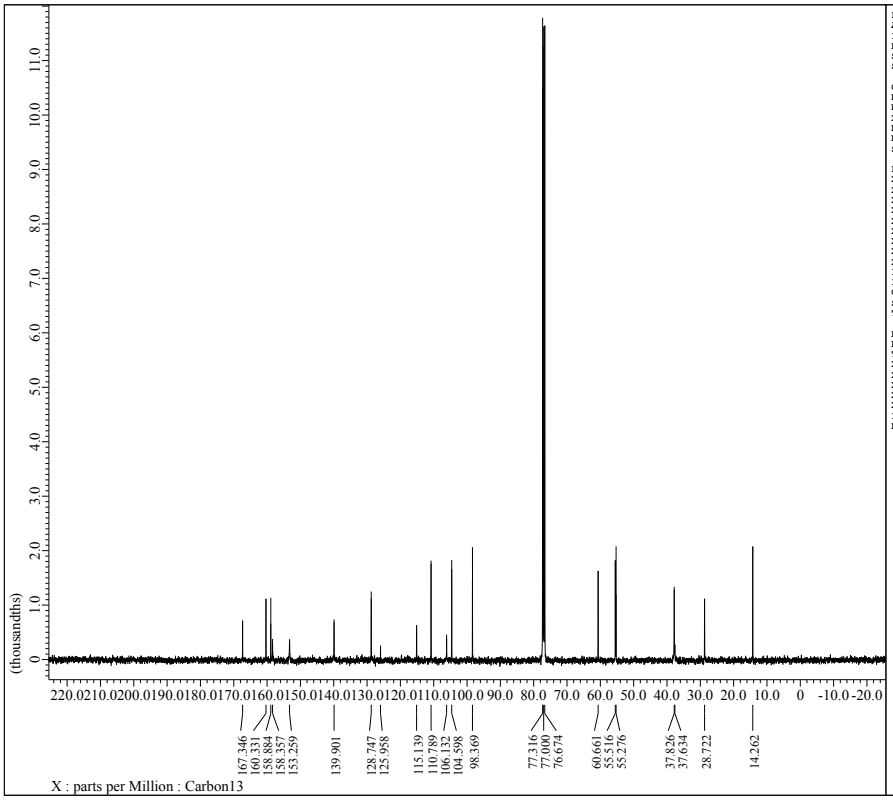
Pk #	Retention Time	Area	Area Percent
1	9.853	804745	49.767
2	11.328	812295	50.233



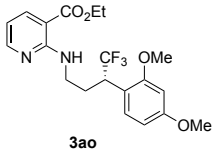

```
Filename          = yd10060-01_Proton-1-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = yd10060-01
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 23.3[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



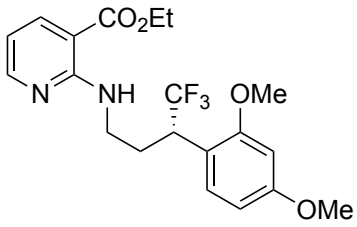
3ao



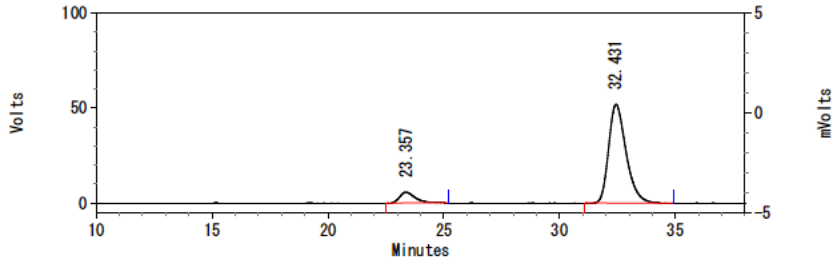

```
Filename          = yd10060-03-13C_Carbon-1-4.
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = yd10060-03-13C
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 16.8[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



3ao

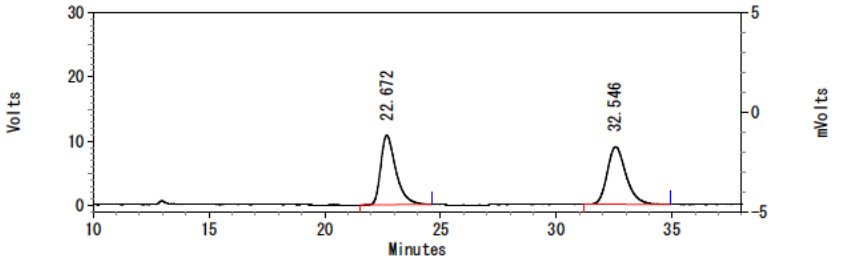


3ao



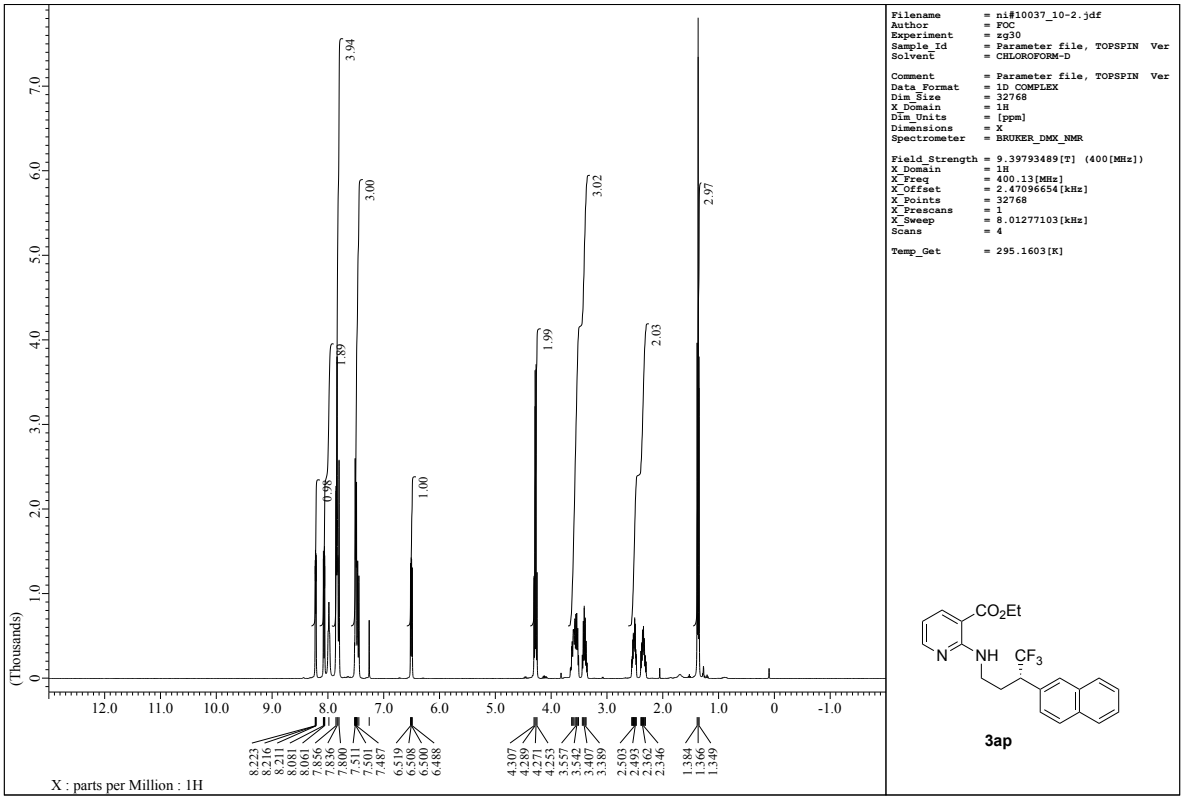
UV Results

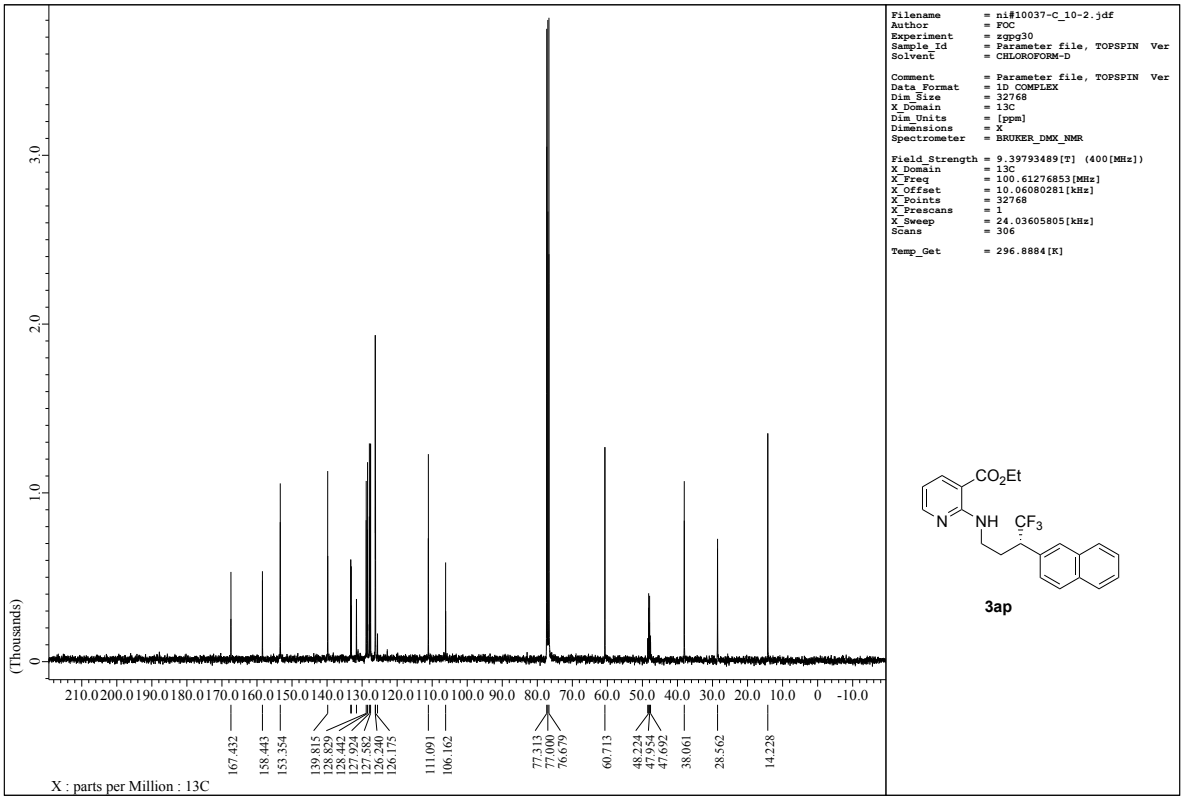
Pk #	Retention Time	Area	Area Percent	Height
1	23.357	262671	8.833	5675
2	32.431	2711225	91.167	51829

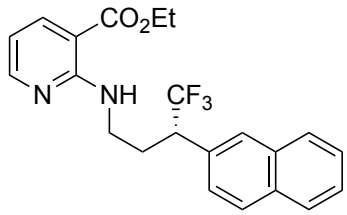


UV Results

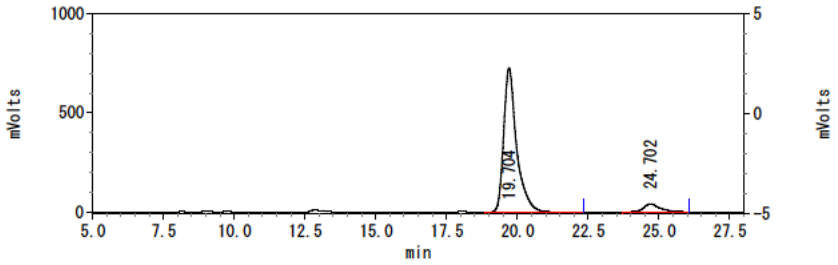
Pk #	Retention Time	Area	Area Percent	Height
1	22.672	490154	49.395	10809
2	32.546	502163	50.605	8948


Filename = ni#10037_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 295.1603[K]
3.94
3.00
3.02
2.97
1.99
2.03
1.89
0.98
1.00
(Thousands)
X : parts per Million : 1H
CO2Et
NH
CF3
3ap

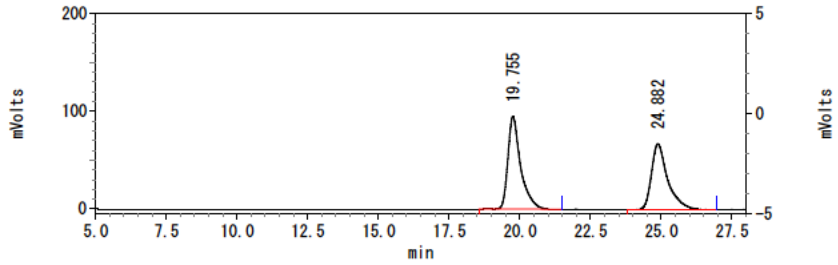

Filename = ni#10037-C_10-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 306
Temp_Get = 296.8884[K]
167.432
158.443
153.354
139.815
128.829
128.442
127.924
127.582
126.240
126.175
111.091
106.162
77.313
77.000
76.679
60.713
48.224
47.954
47.692
38.061
28.562
14.228
(Thousands)
X : parts per Million : 13C
CO2Et
NH
CF3
3ap



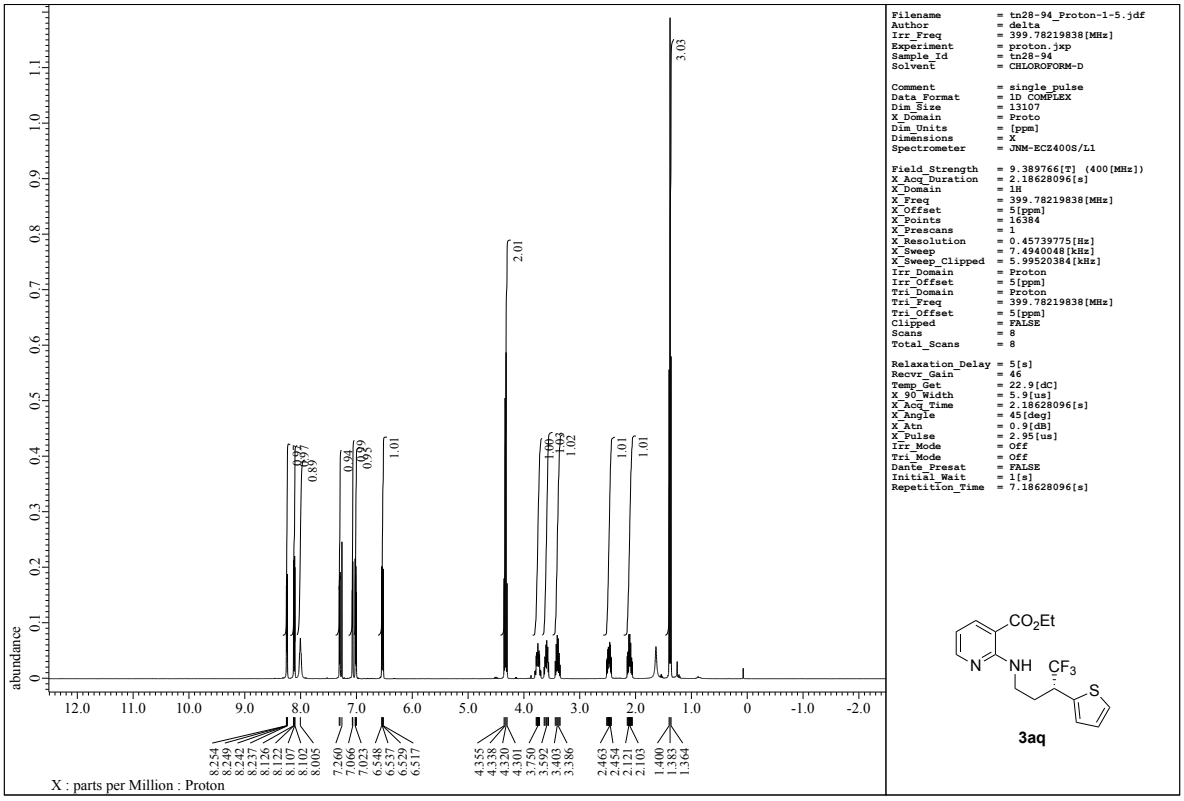
3ap

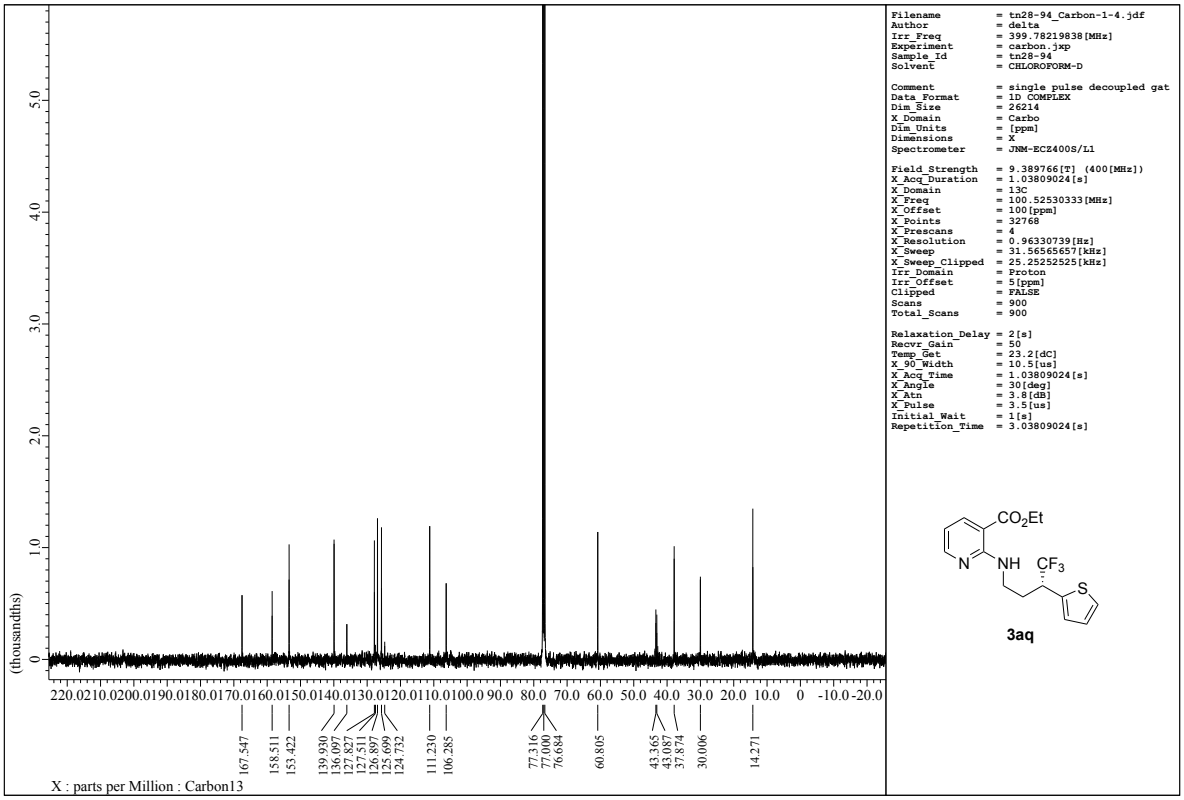


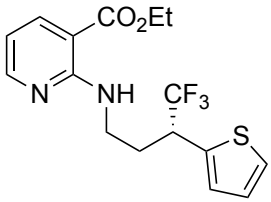
Pk #	Retention Time	Area	Area Percent
1	19.704	22821743	93.721
2	24.702	1528957	6.279



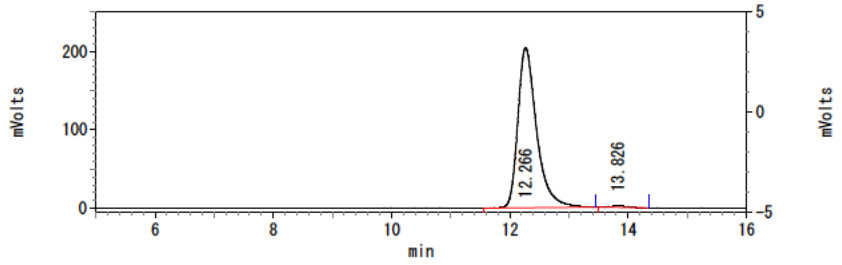
Pk #	Retention Time	Area	Area Percent
1	19.755	2993780	52.855
2	24.882	2670351	47.145


Filename = tn28-94_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-94
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 22.9[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3aq

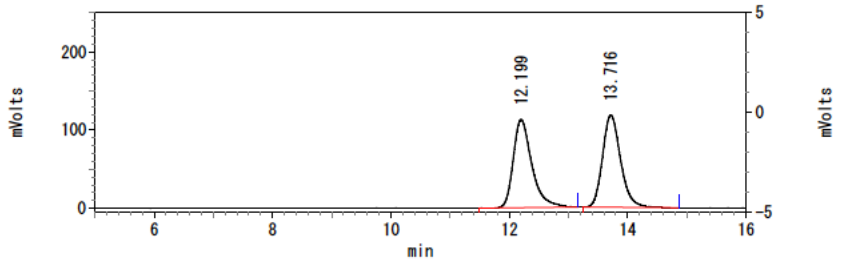

Filename = tn28-94_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-94
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 900
Total_Scans = 900
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.2[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
3aq



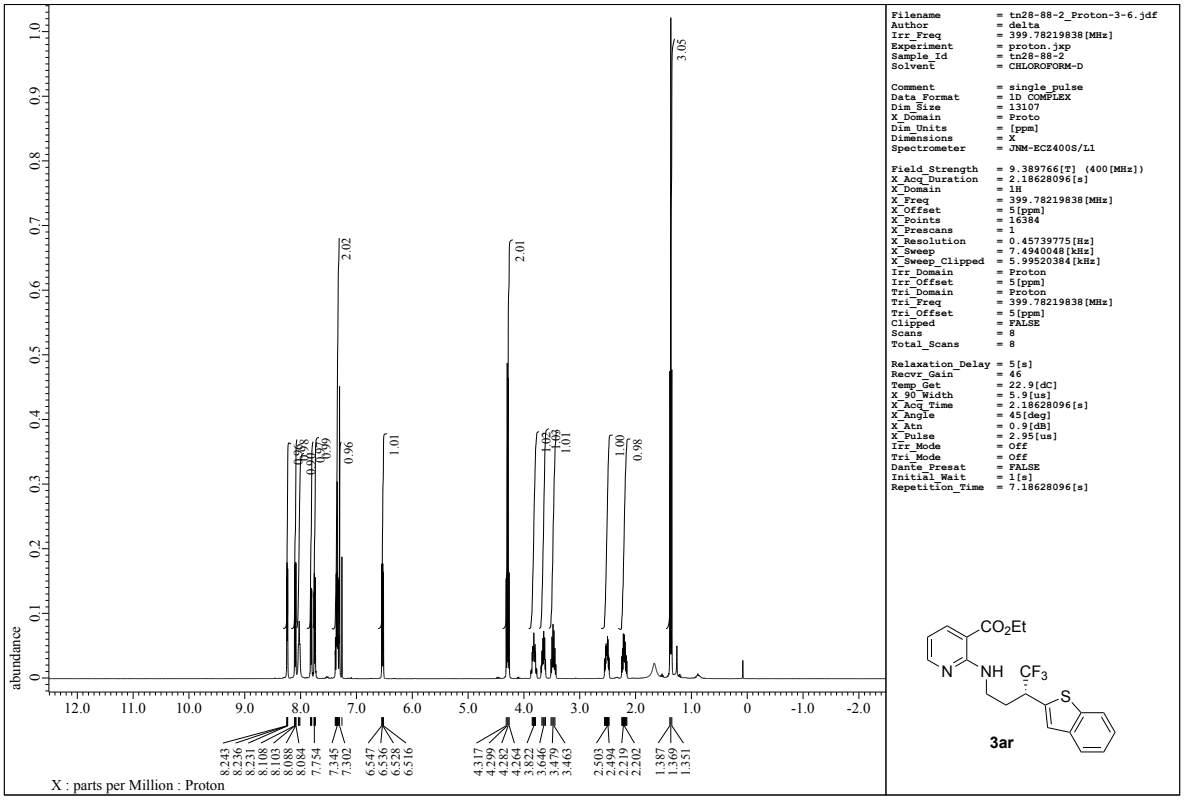
3aq

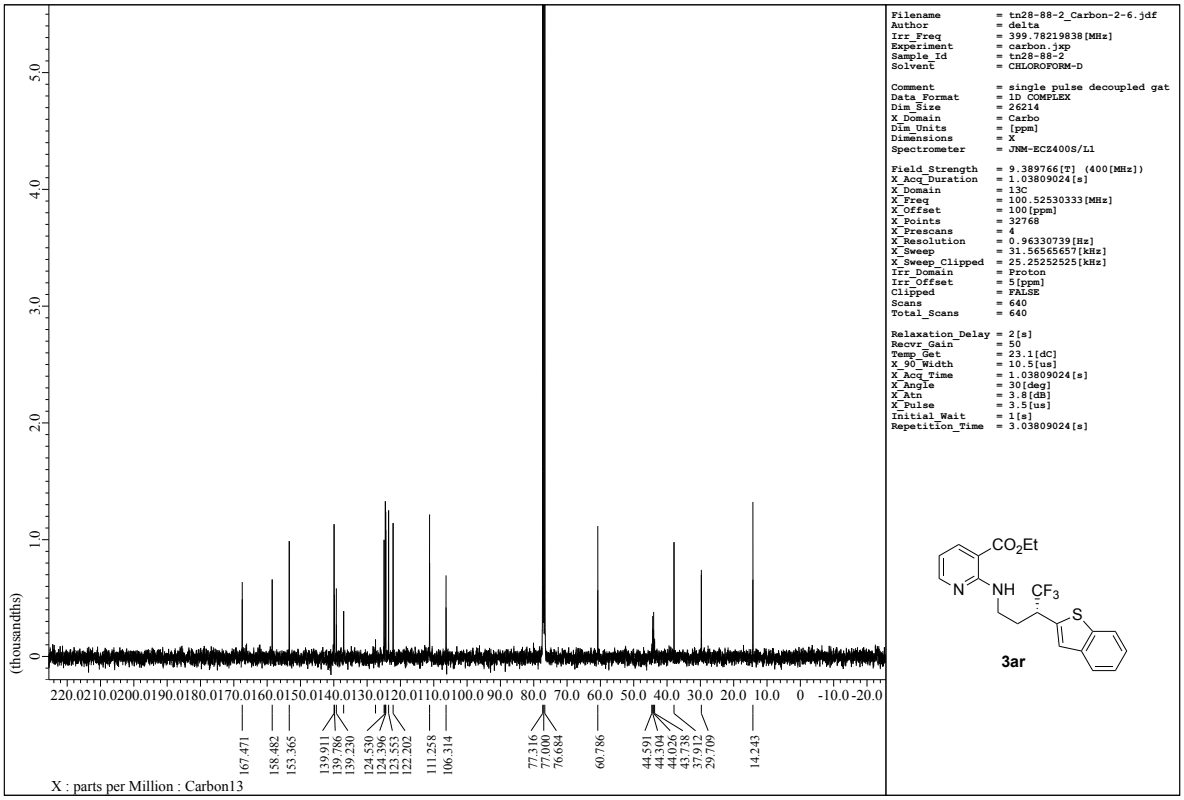


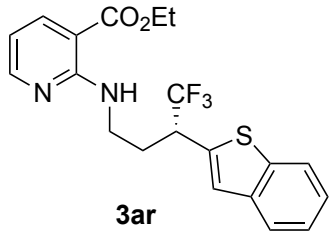
Pk #	Retention Time	Area	Area Percent
1	12.266	4499761	99.111
2	13.826	40356	0.889



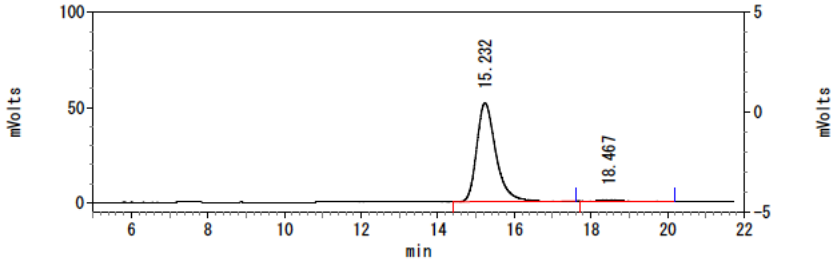
Pk #	Retention Time	Area	Area Percent
1	12.199	2456835	49.526
2	13.716	2503814	50.474


abundance
X : parts per Million : Proton
Filename = tn28-88-2_Proton-3-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-88-2
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 22.9[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CF3
S
3ar

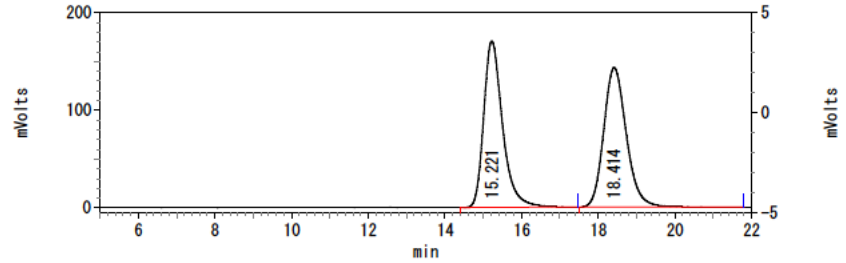

(thousandths)
X : parts per Million : Carbon13
Filename = tn28-88-2_Carbon-2-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-88-2
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 640
Total_Scans = 640
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.1[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CF3
S
3ar



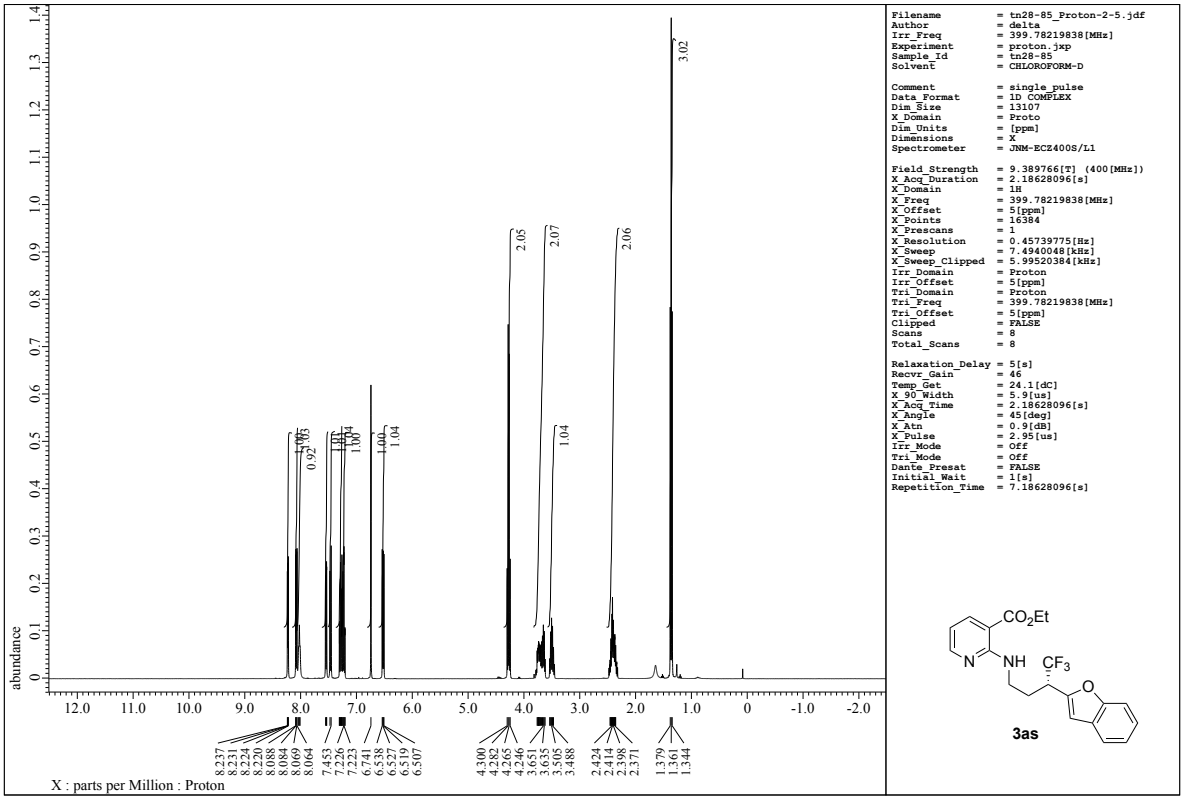
3ar

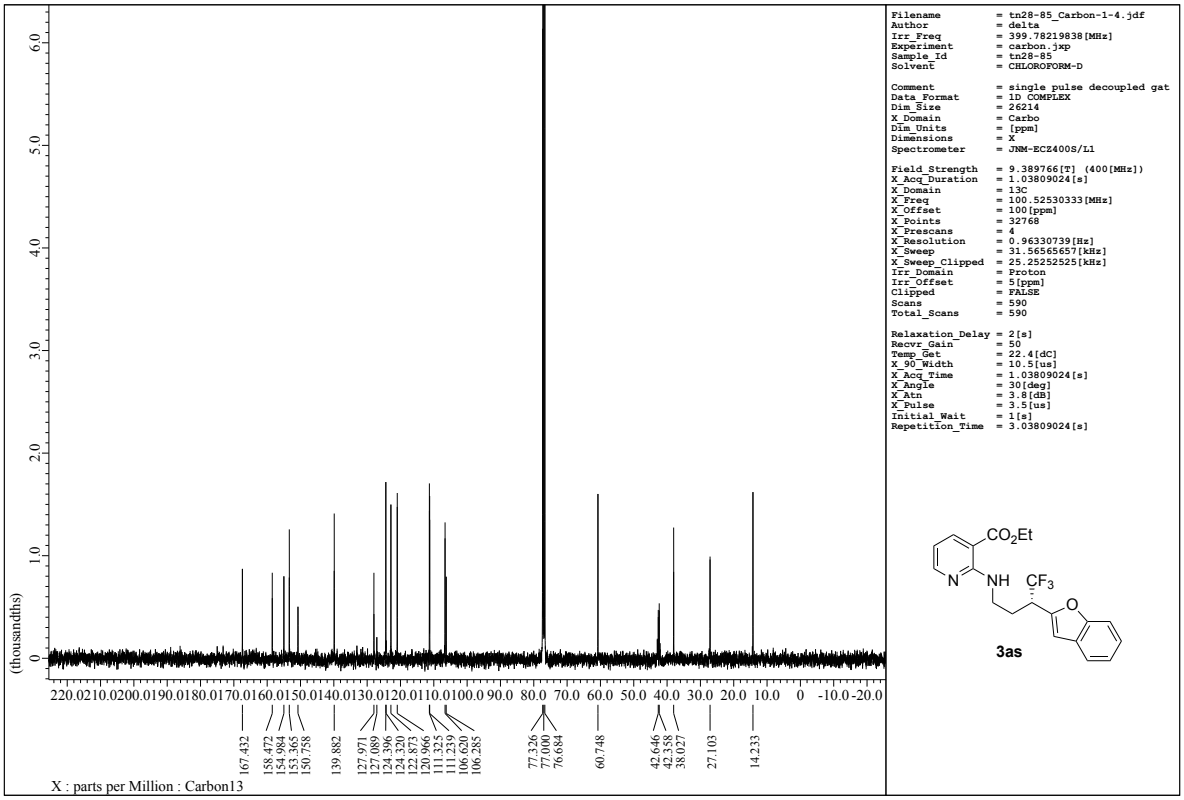


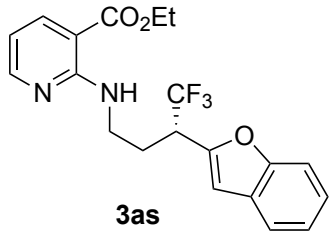
Pk #	Retention Time	Area	Area Percent
1	15.232	1812344	98.018
2	18.467	36649	1.982



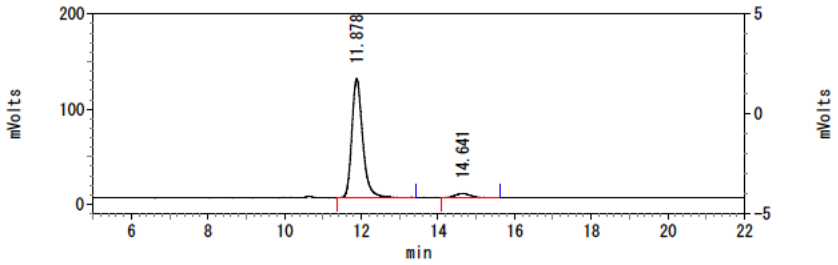
Pk #	Retention Time	Area	Area Percent
1	15.221	5841280	49.849
2	18.414	5876660	50.151


abundance
X : parts per Million : Proton
Filename = tn28-85_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-85
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 24.1[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CF3
O
3as

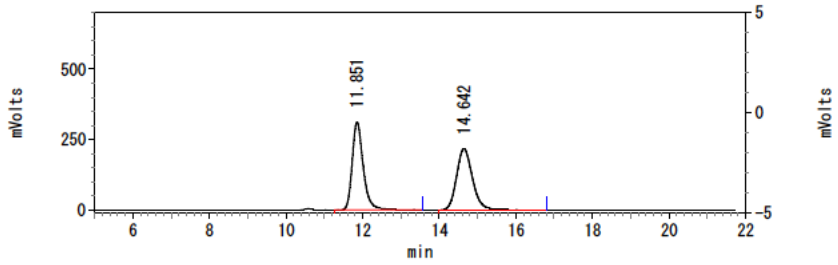

(thousandths)
X : parts per Million : Carbon13
Filename = tn28-85_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-85
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 590
Total_Scans = 590
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.4[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CF3
O
3as



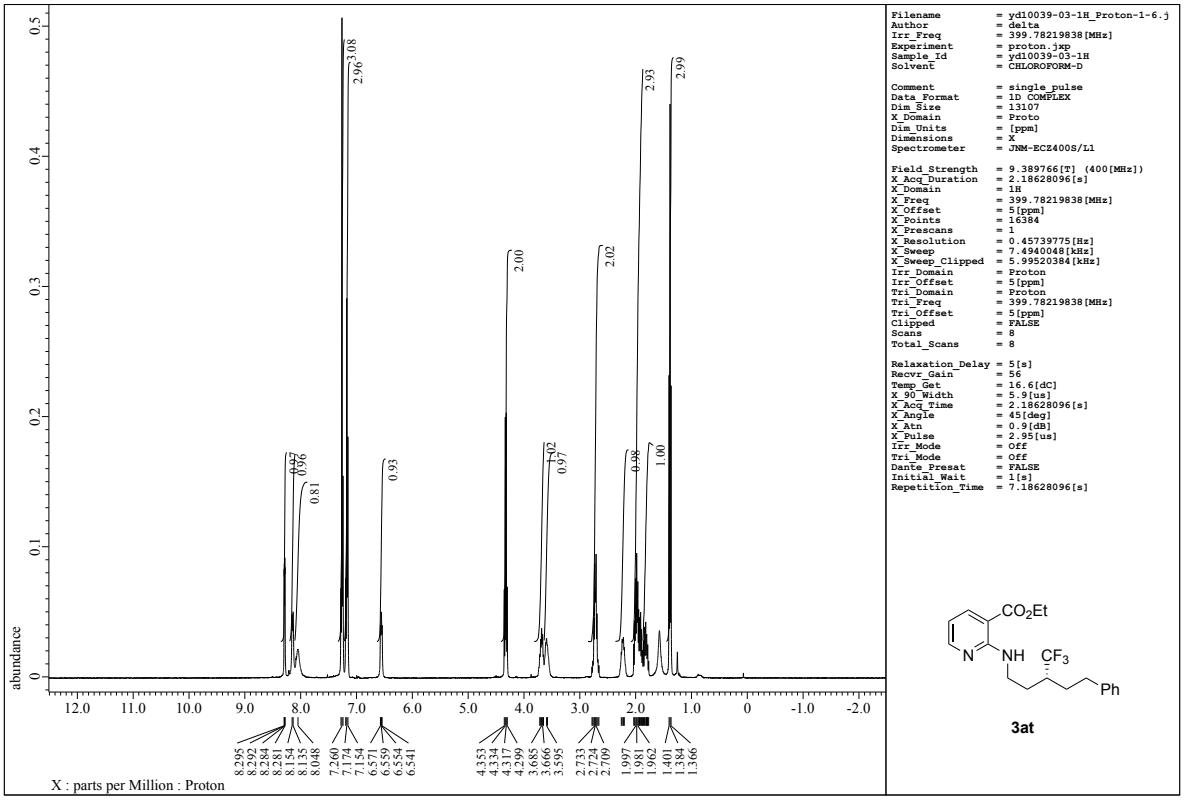
3as

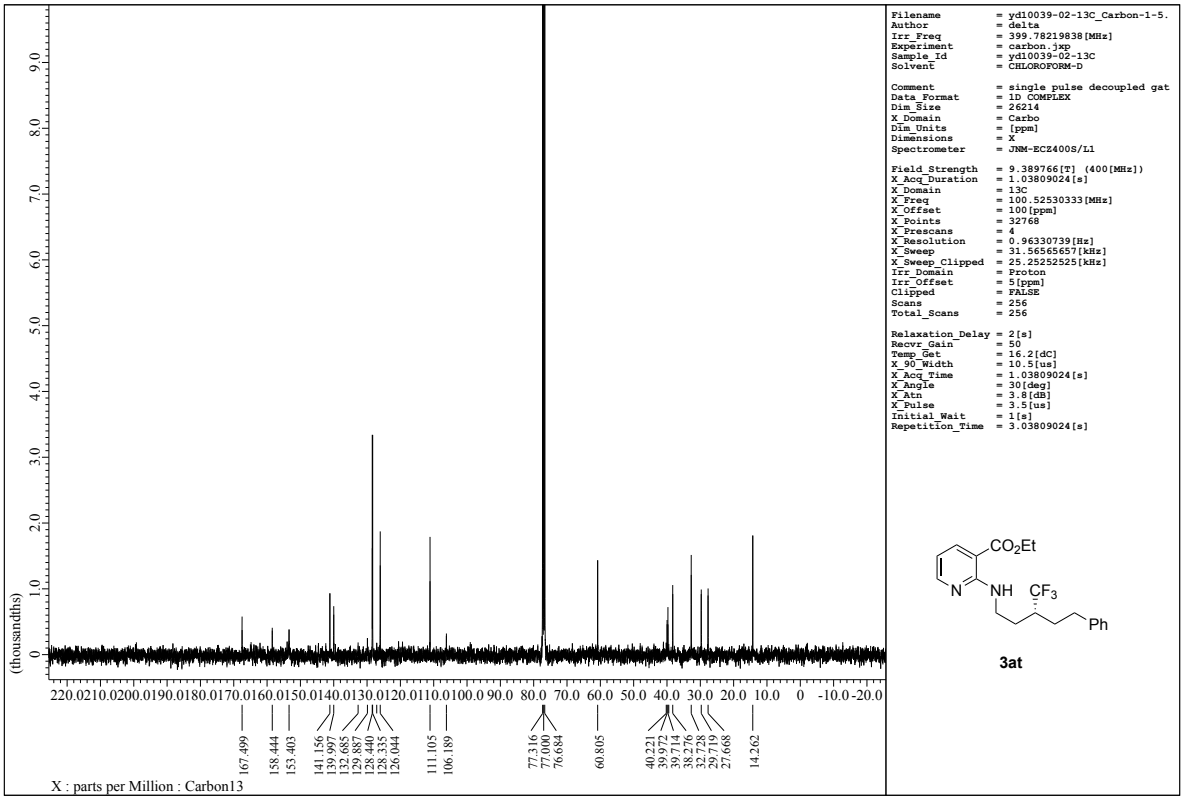


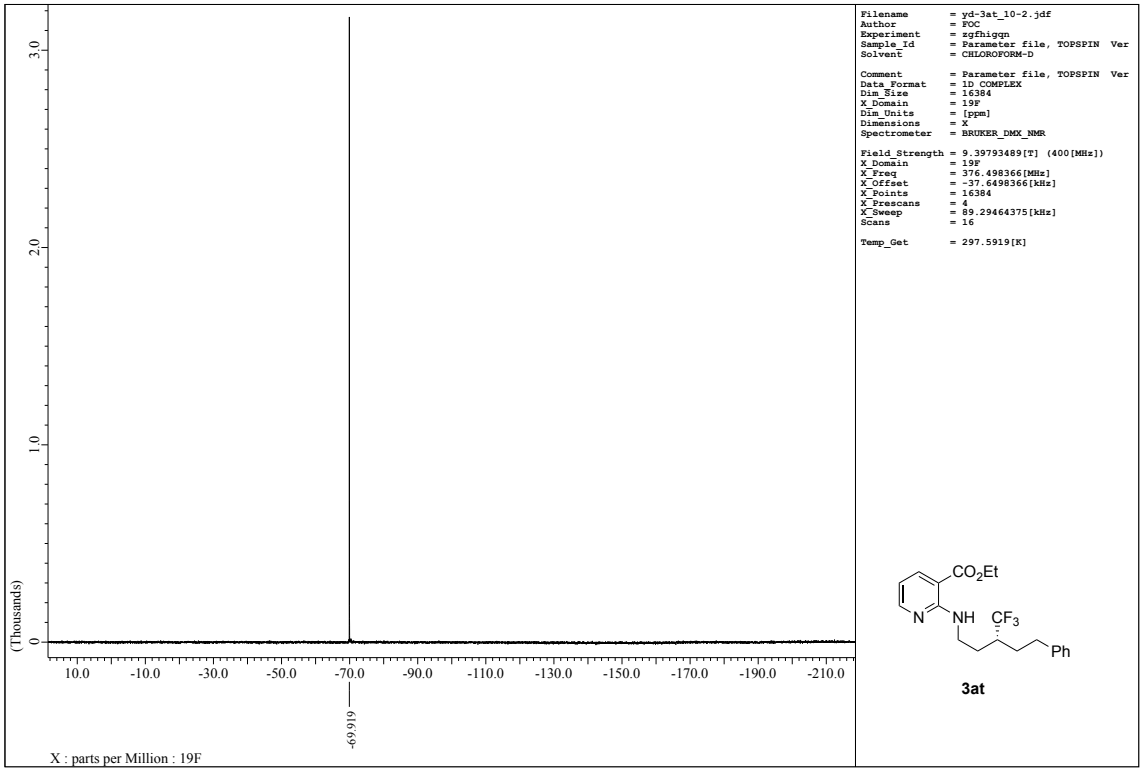
Pk #	Retention Time	Area	Area Percent
1	11.878	2476374	95.134
2	14.641	126666	4.866

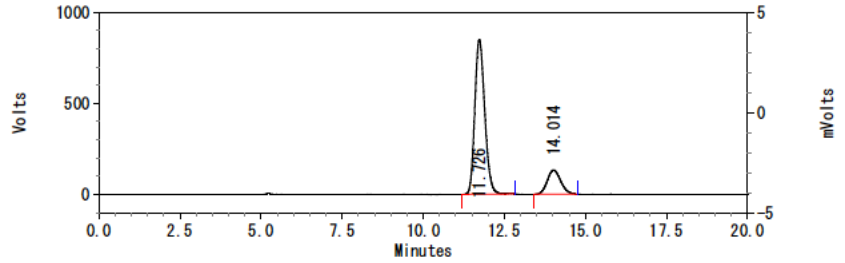


Pk #	Retention Time	Area	Area Percent
1	11.851	6238768	50.083
2	14.642	6218121	49.917


abundance
X : parts per Million : Proton
3.08
2.96
2.93
2.99
2.00
2.02
0.81
0.96
0.97
0.93
1.02
0.98
1.00
Filename = yd10039-03-1H_Proton-1-6.j
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd10039-03-1H
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 16.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CF3
Ph
3at

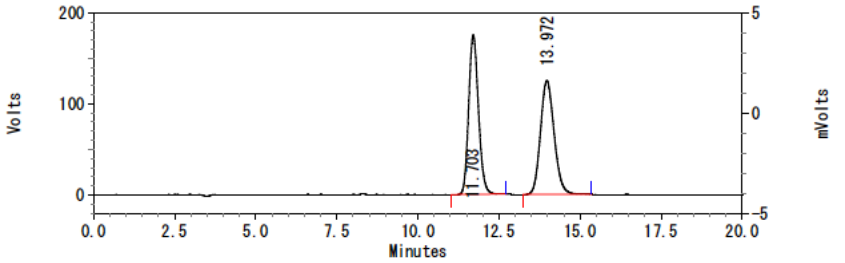

(thousandths)
X : parts per Million : Carbon13
167.499
158.444
153.403
141.156
139.997
132.685
129.587
128.440
128.335
126.044
111.105
106.189
77.316
77.000
76.684
60.805
40.221
39.972
39.714
38.276
32.728
29.719
27.668
14.262
Filename = yd10039-02-13C_Carbon-1-5.
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = yd10039-02-13C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 16.2[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CF3
Ph
3at





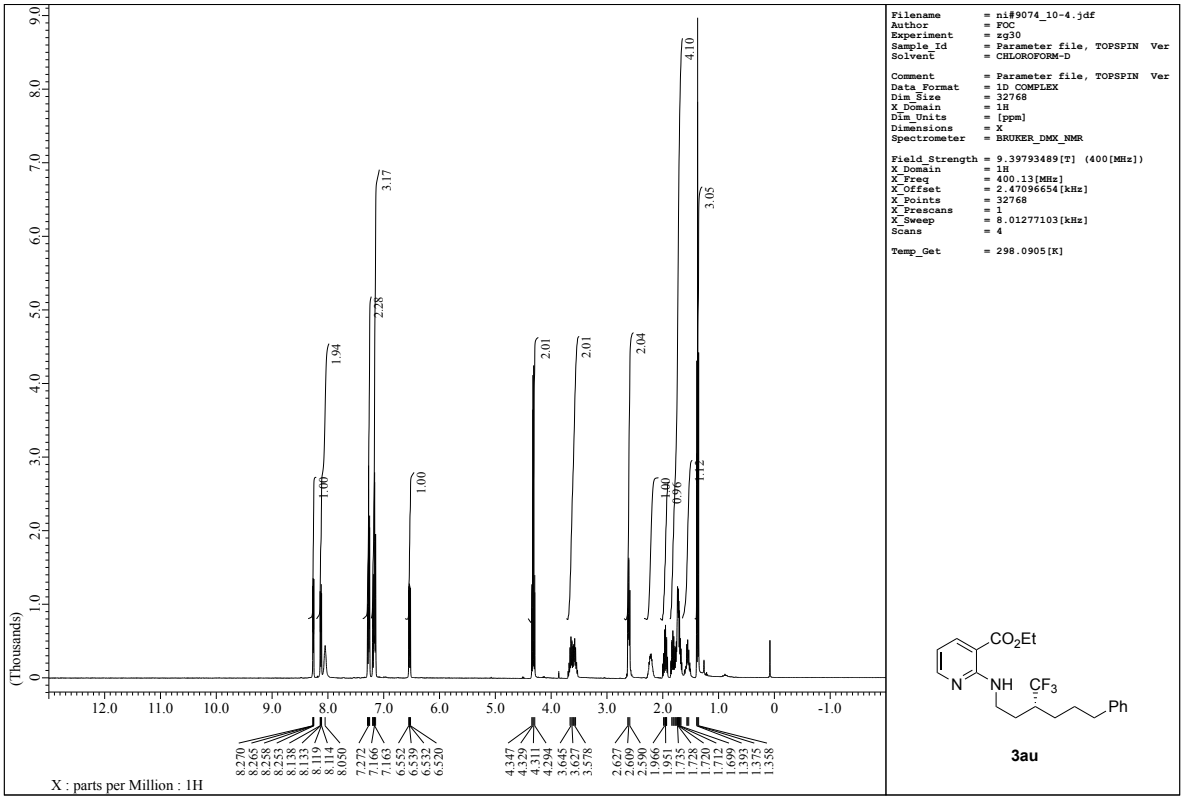
UV Results

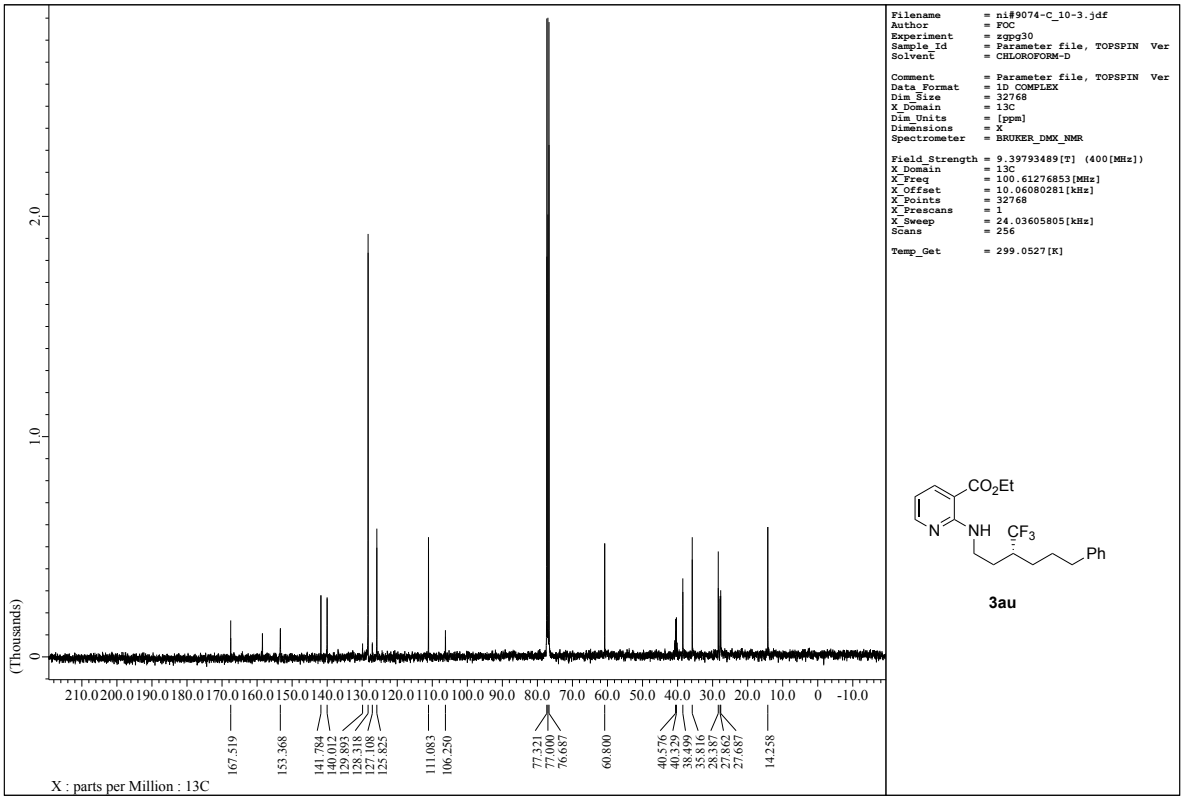
Pk #	Retention Time	Area	Area Percent	Height
1	11.726	18049406	82.590	849590
2	14.014	3804702	17.410	132503

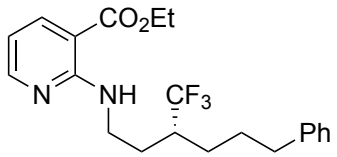


UV Results

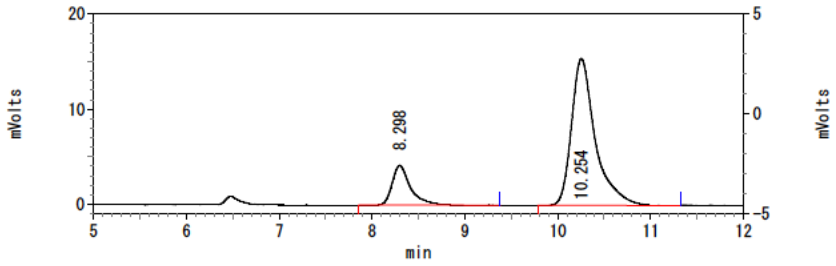
Pk #	Retention Time	Area	Area Percent	Height
1	11.703	3650661	49.923	174722
2	13.972	3661927	50.077	124887


Filename = ni#9074_10-4.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.0905[K]
(Thousands)
X : parts per Million : 1H
CO2Et
NH
CF3
Ph
3au

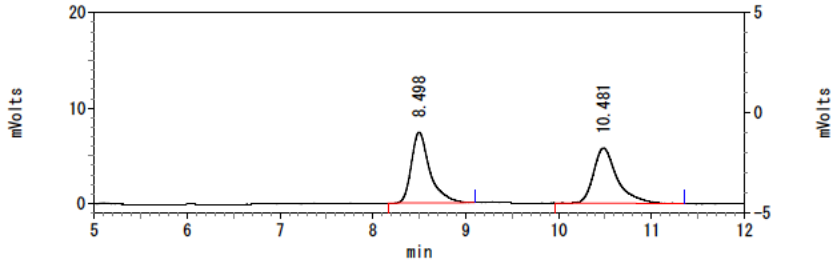

Filename = ni#9074-C_10-3.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 256
Temp_Get = 299.0527[K]
(Thousands)
X : parts per Million : 13C
CO2Et
NH
CF3
Ph
3au



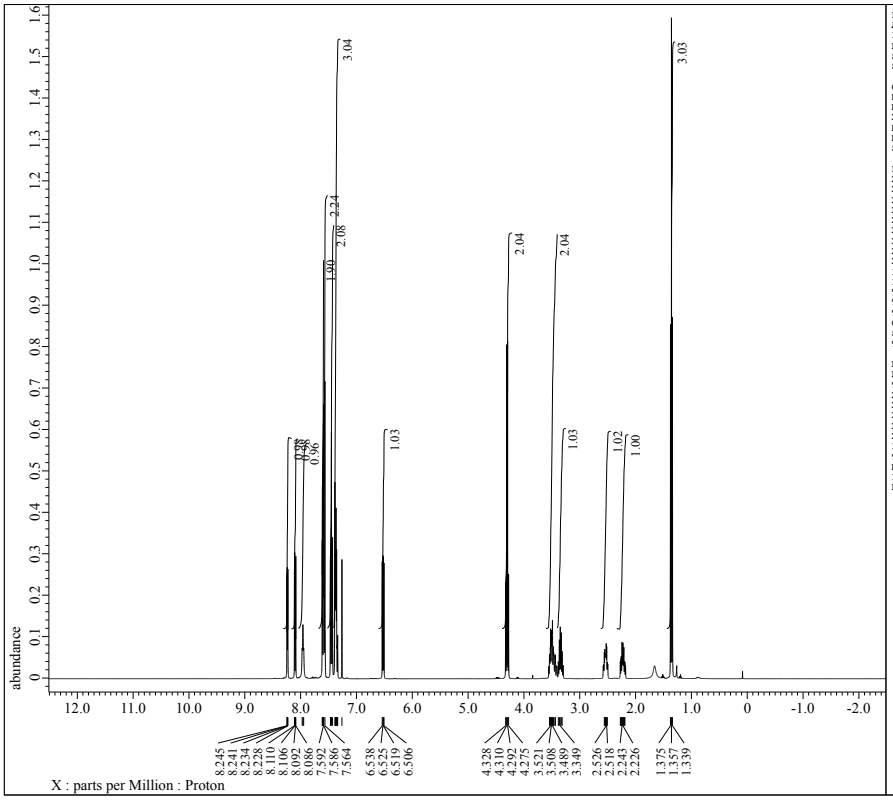
3au



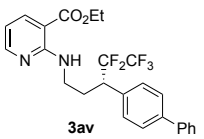
Pk #	Retention Time	Area	Area Percent
1	8.298	57720	17.329
2	10.254	275355	82.671



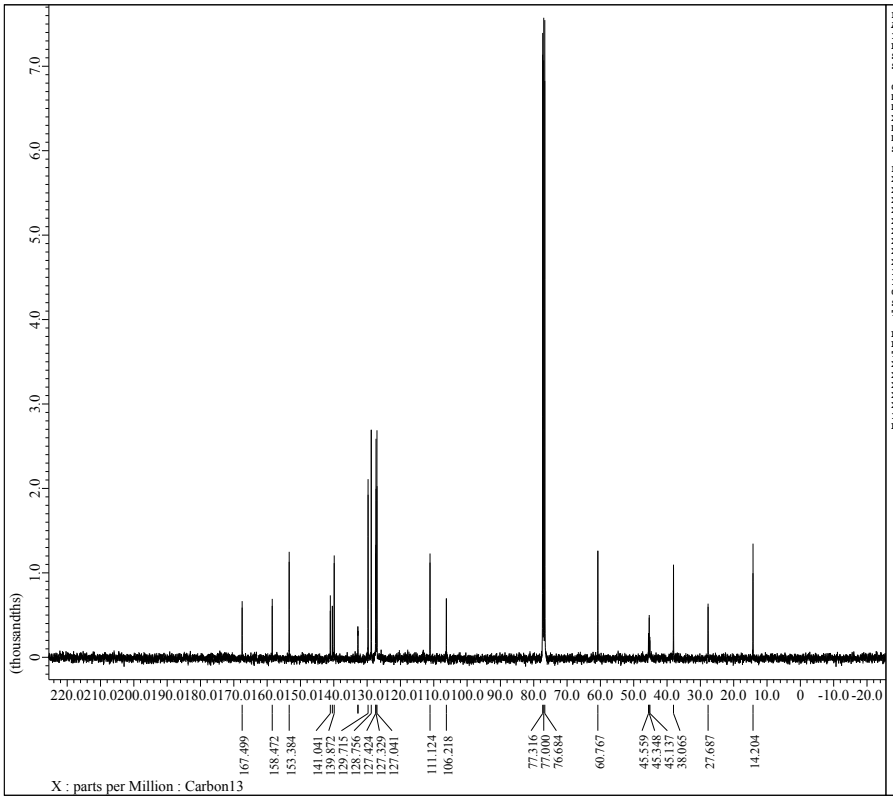
Pk #	Retention Time	Area	Area Percent
1	8.498	102847	49.978
2	10.481	102939	50.022



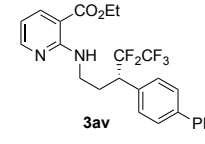

```
Filename          = tn28-93_Proton-3-7.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn28-93
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 22.8[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



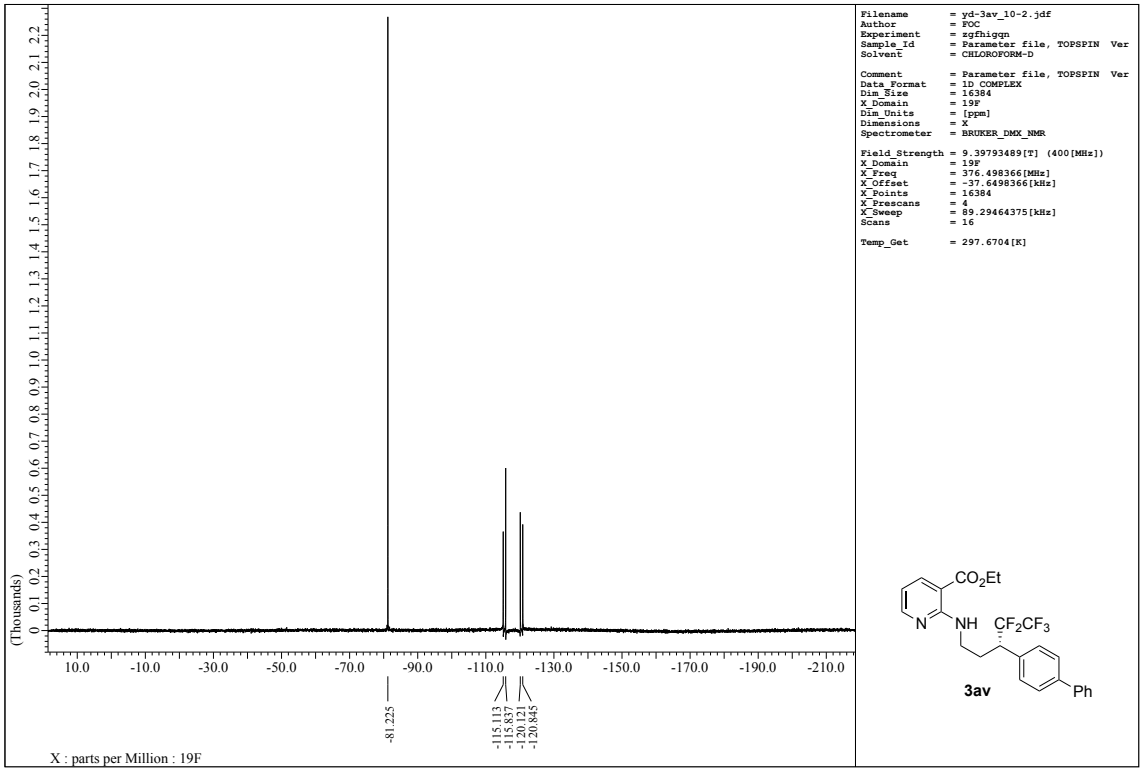
3av

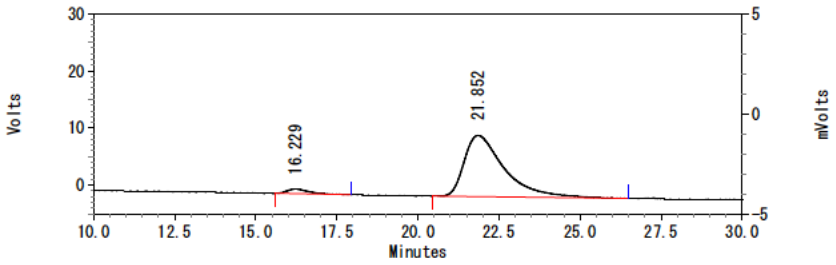



```
Filename          = tn28-93_Carbon-1-3.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn28-93
Solvent           = NONE

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 993
Total_Scans       = 993

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.1[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



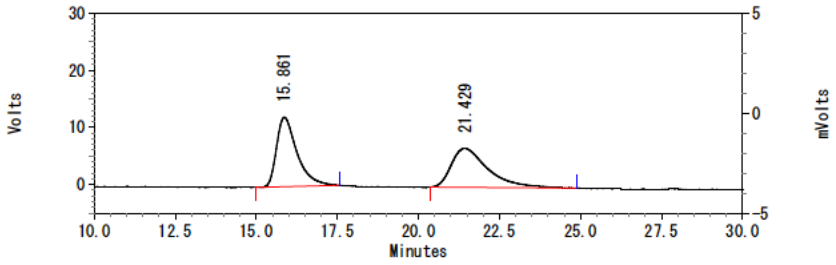
3av





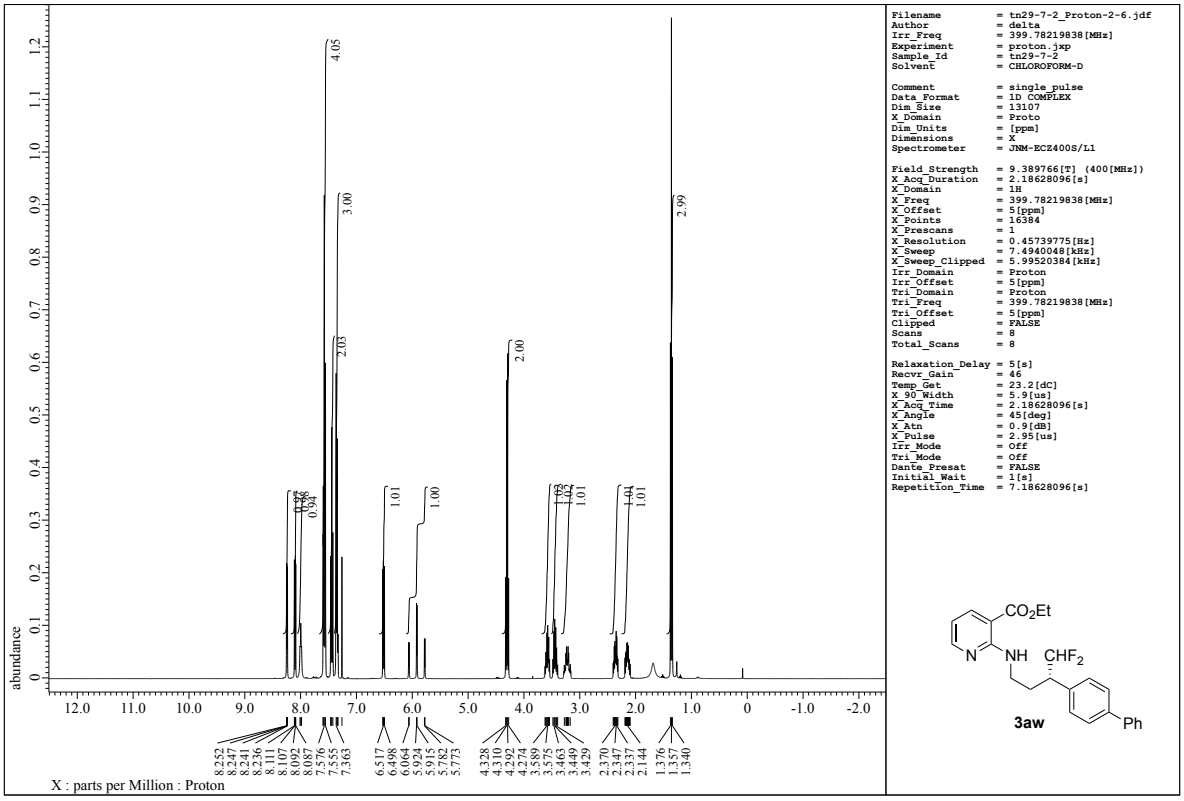
UV Results

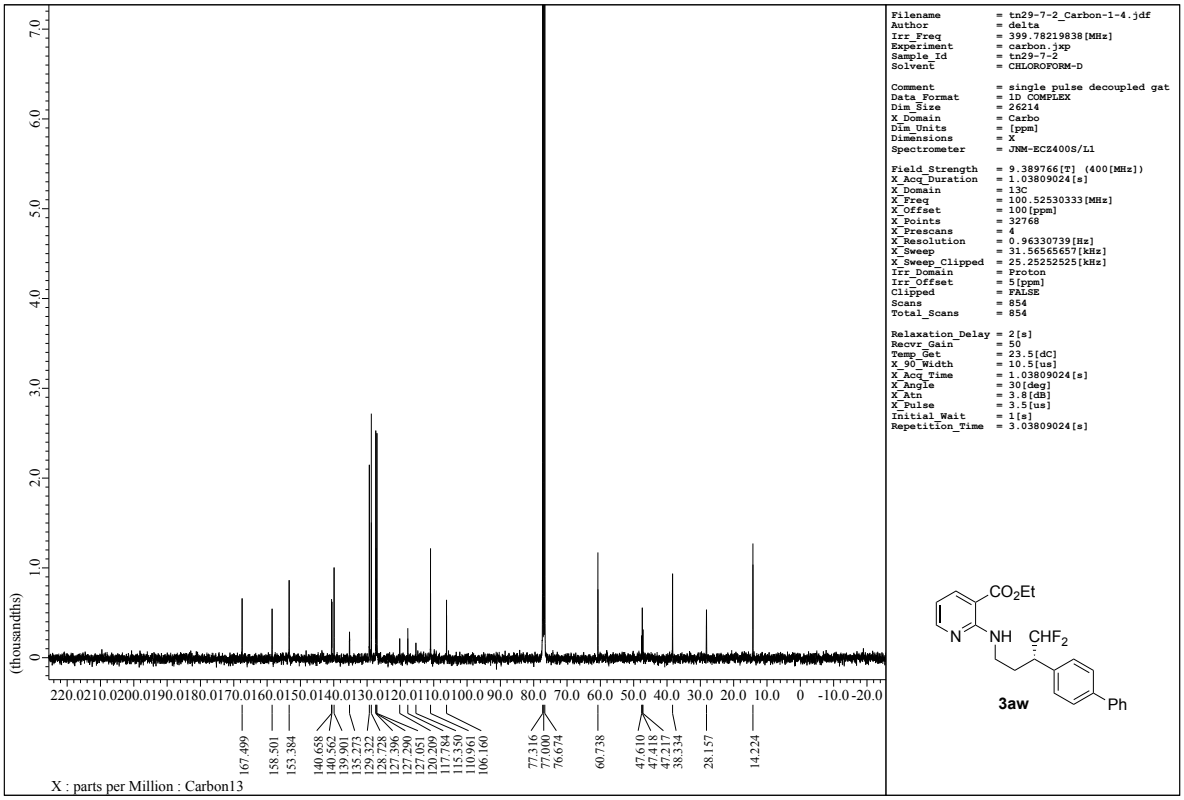
Pk #	Retention Time	Area	Area Percent	Height
1	16.229	37234	3.895	787
2	21.852	918656	96.105	10692

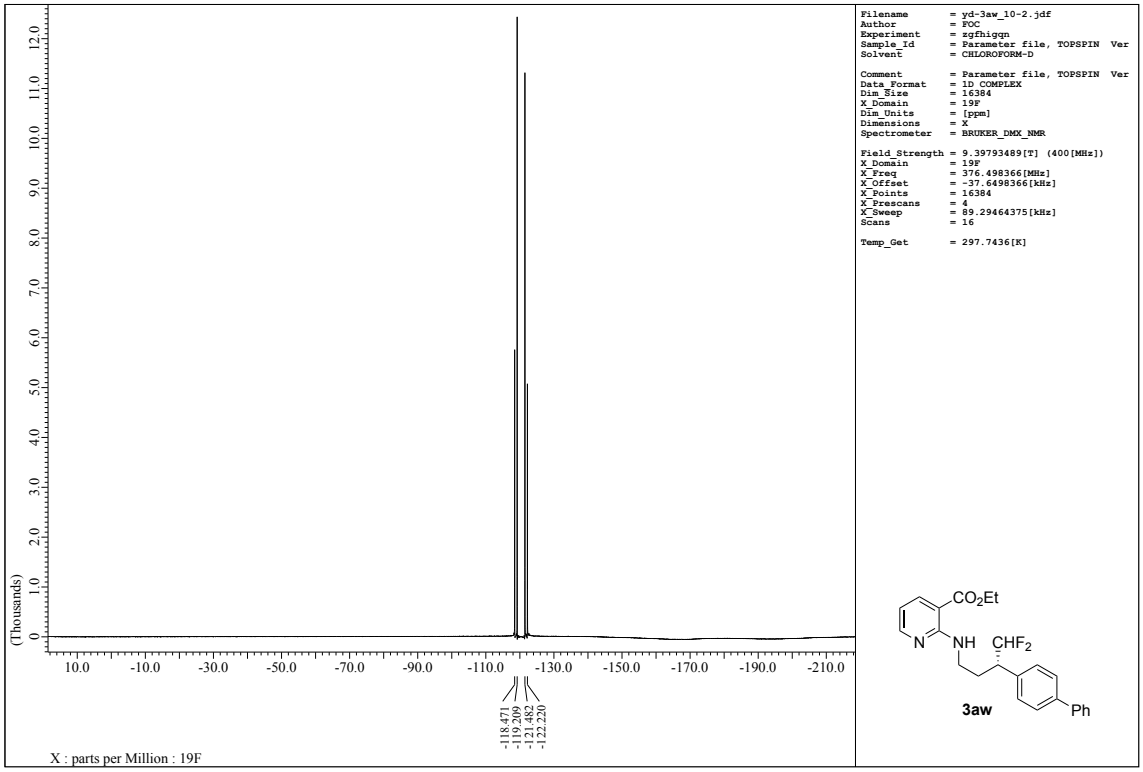


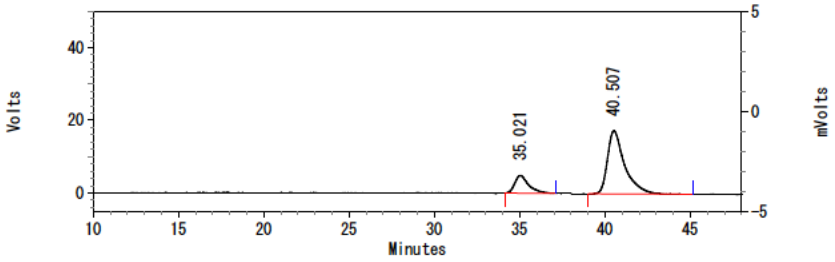
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	15.861	532829	49.765	12130
2	21.429	537869	50.235	6821


abundance
X : parts per Million : Proton
4.05
3.00
2.99
2.03
2.00
1.01
1.00
1.01
1.01
Filename = tn29-7-2_Proton-2-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn29-7-2
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 23.2[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
CHF2
3aw
Ph

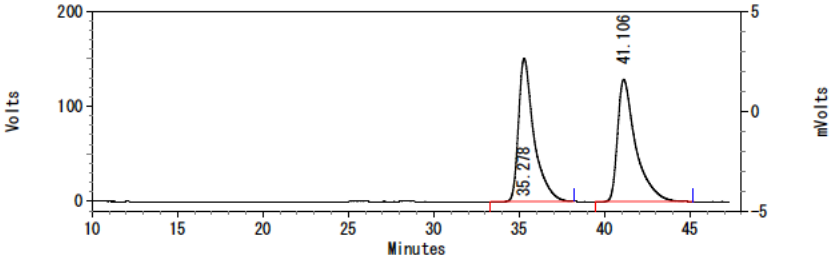

(thousandths)
X : parts per Million : Carbon13
167.499
158.501
153.384
140.658
140.562
139.901
135.273
129.322
128.728
127.396
127.290
127.051
120.209
117.784
115.330
110.961
106.160
77.316
77.000
76.674
60.738
47.610
47.418
47.217
38.334
28.157
14.224
Filename = tn29-7-2_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn29-7-2
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 854
Total_Scans = 854
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 23.5[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
CHF2
3aw
Ph





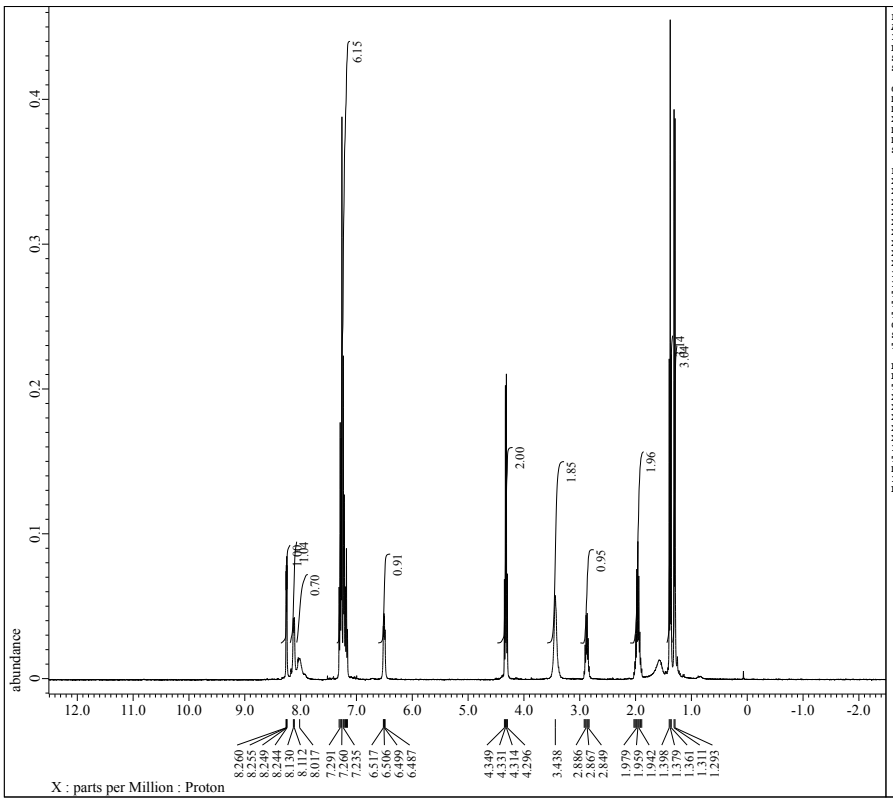
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	35.021	276339	18.549	4858
2	40.507	1213434	81.451	17350

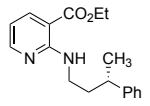


UV Results

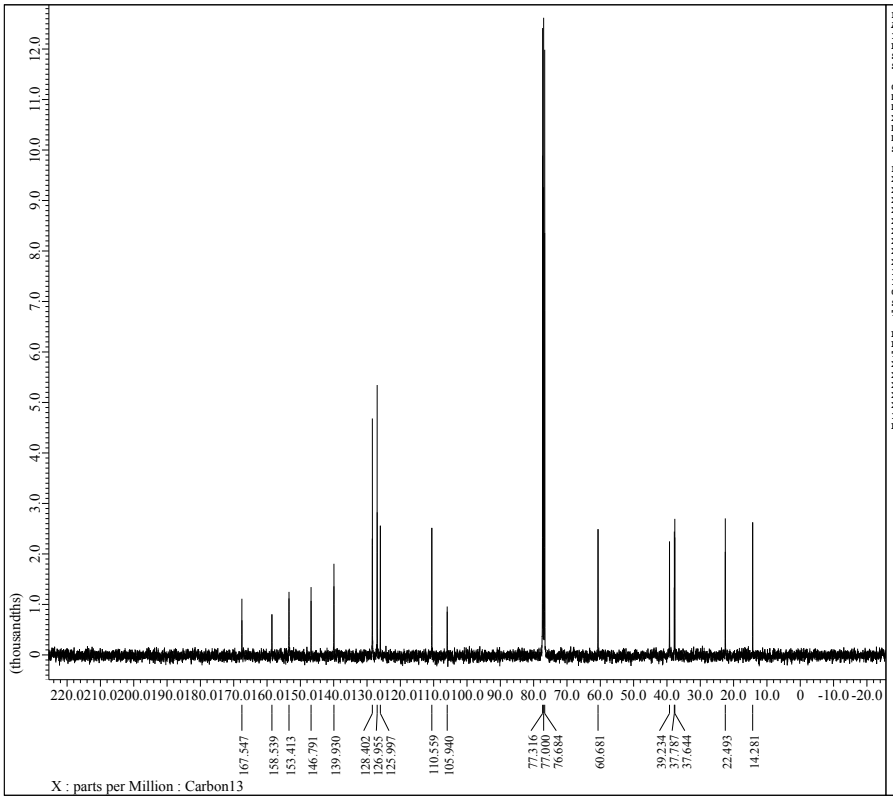
Pk #	Retention Time	Area	Area Percent	Height
1	35.278	9435889	49.906	150500
2	41.106	9471257	50.094	128396



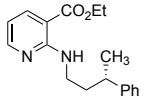

```
Filename          = yd10075-03-1H_Proton-1-4.j
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = yd10075-03-1H
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 16.6[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



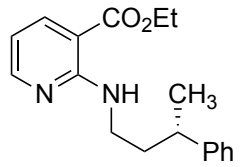
3ax



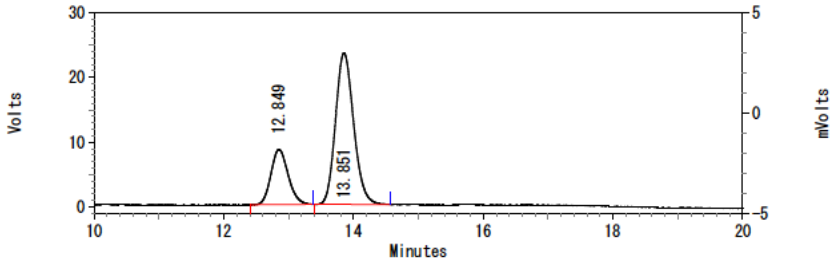

```
Filename          = yd10075-02-13C_Carbon-1-3.
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = yd10075-02-13C
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 16.5[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



3ax

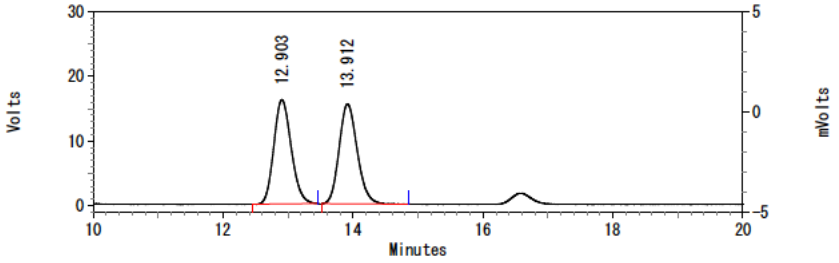


3ax



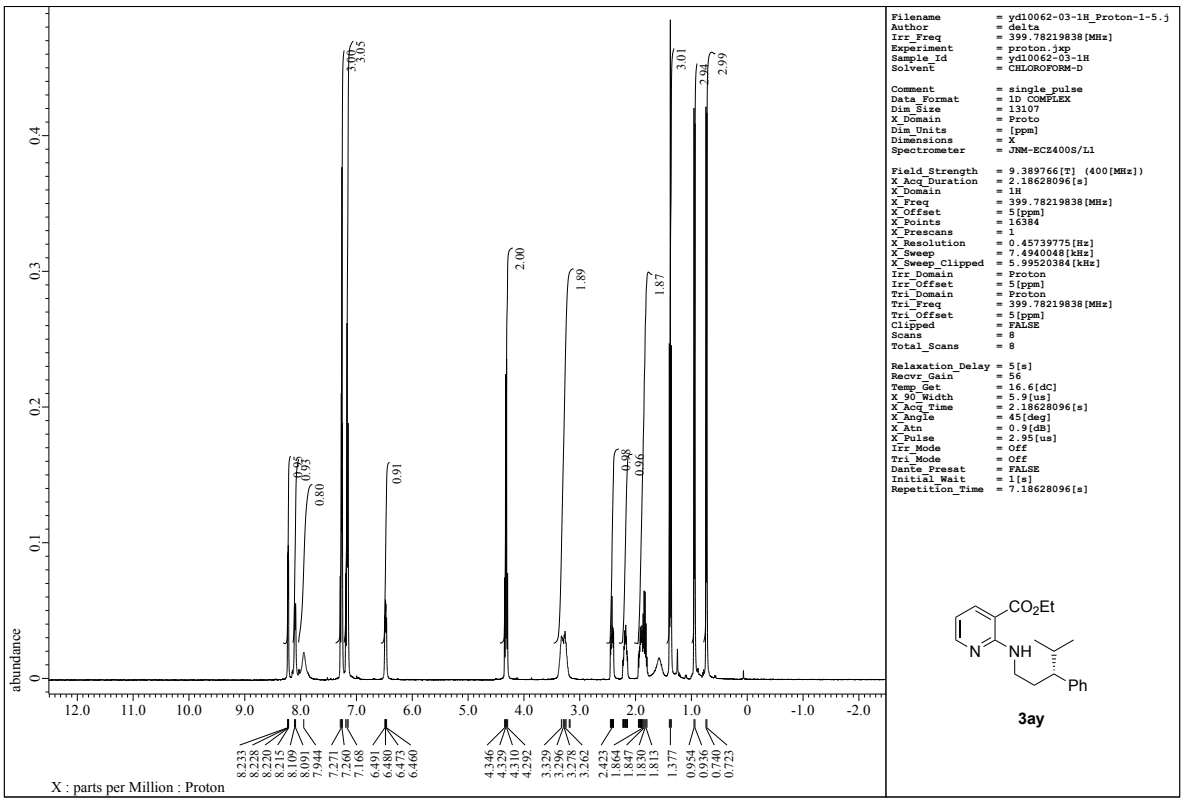
UV Results

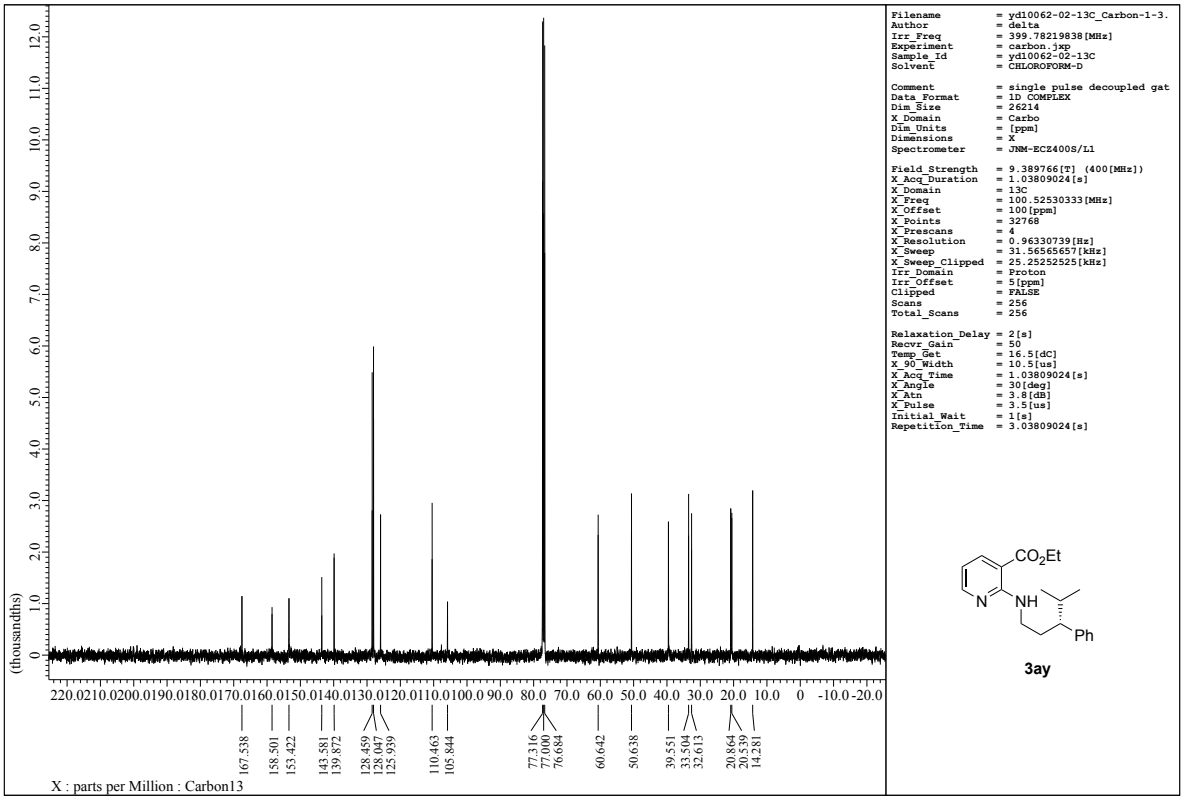
Pk #	Retention Time	Area	Area Percent	Height
1	12.849	156966	25.633	8523
2	13.851	455386	74.367	23400

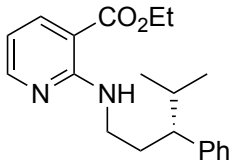


UV Results

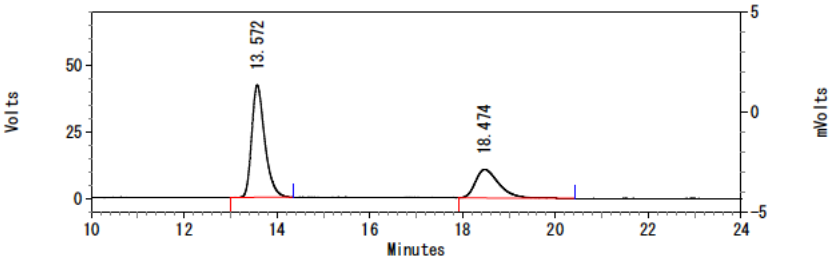
Pk #	Retention Time	Area	Area Percent	Height
1	12.903	300239	50.040	16118
2	13.912	299765	49.960	15460


abundance
X : parts per Million : Proton
Filename = yd10062-03-1H_Proton-1-5.j
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd10062-03-1H
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 16.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CO2Et
N
NH
Ph
3ay


(thousandths)
X : parts per Million : Carbon13
Filename = yd10062-02-13C_Carbon-1-3.
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = yd10062-02-13C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 16.5[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CO2Et
N
NH
Ph
3ay

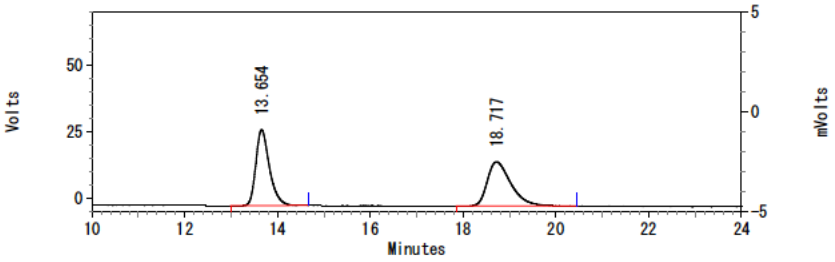


3ay



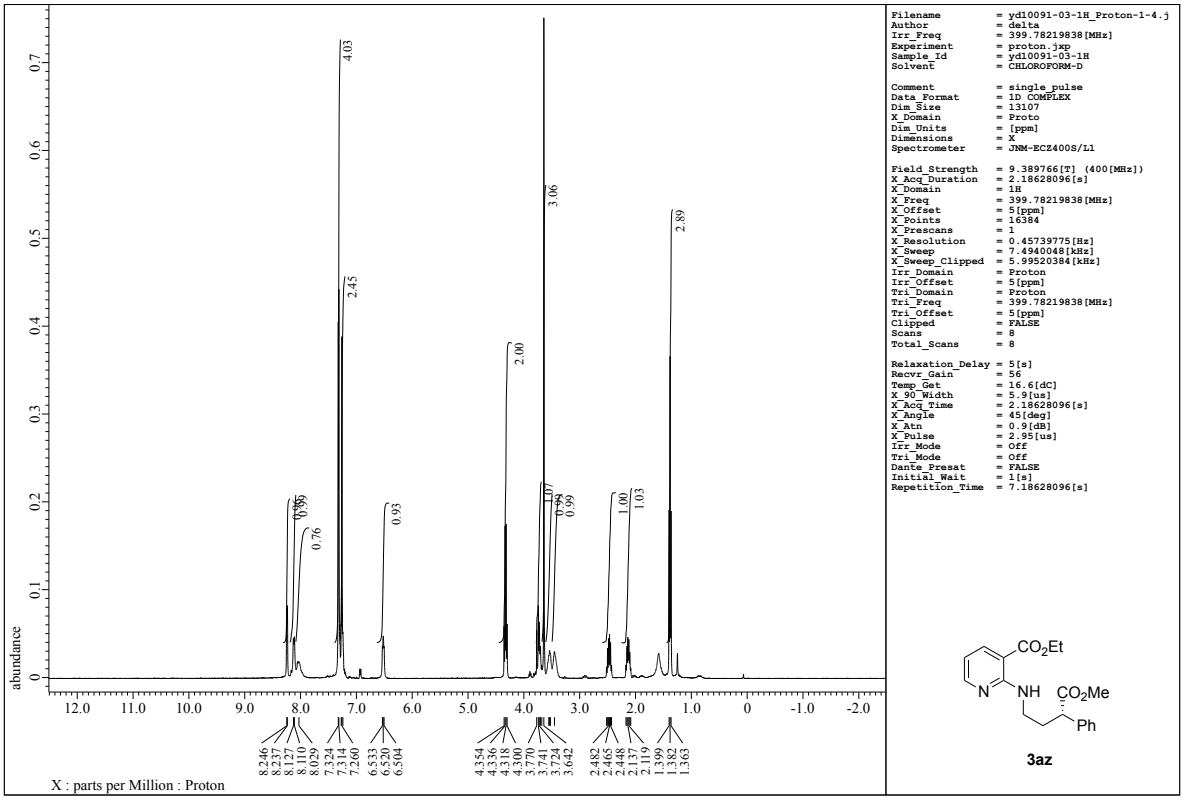
UV Results

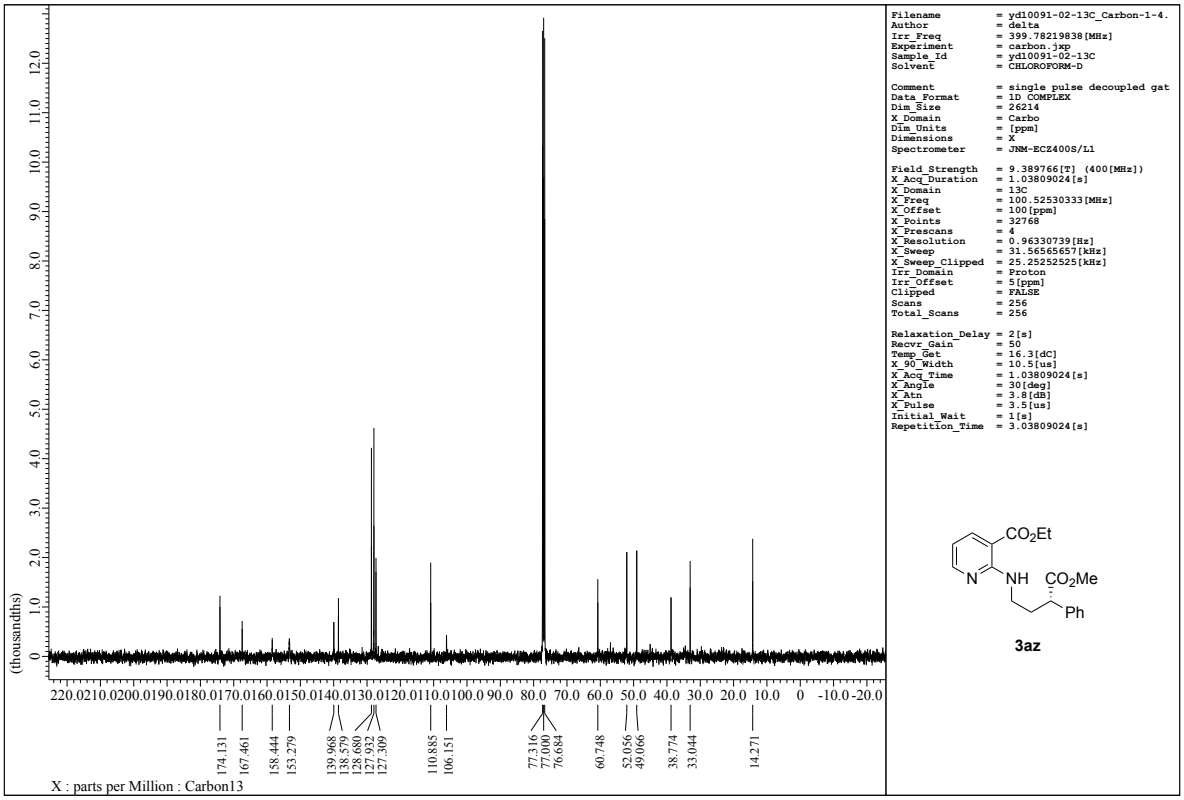
Pk #	Retention Time	Area	Area Percent	Height
1	13.572	823358	68.803	42070
2	18.474	373325	31.197	10669

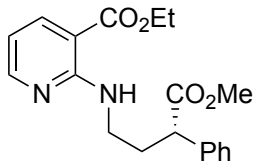


UV Results

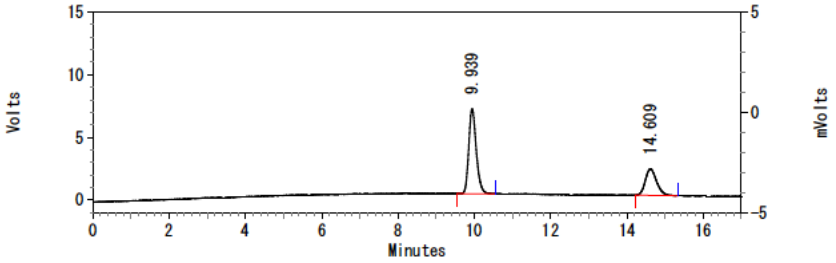
Pk #	Retention Time	Area	Area Percent	Height
1	13.654	579664	49.898	28495
2	18.717	582041	50.102	16531


Filename = yd10091-03-1H_Proton-1-4.j
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd10091-03-1H
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 16.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
4.03
3.06
2.89
2.45
2.00
1.07
1.03
1.00
0.99
0.93
0.76
CO2Et
N
NH
CO2Me
Ph
3az


Filename = yd10091-02-13C_Carbon-1-4.
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = yd10091-02-13C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 16.3[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
174.131
167.461
158.444
153.279
139.968
138.579
138.089
127.932
127.309
110.885
106.151
77.316
77.000
76.684
60.748
52.056
49.066
38.774
33.044
14.271
CO2Et
N
NH
CO2Me
Ph
3az

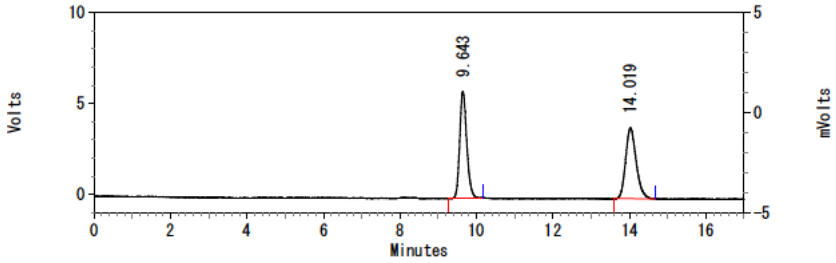


3az



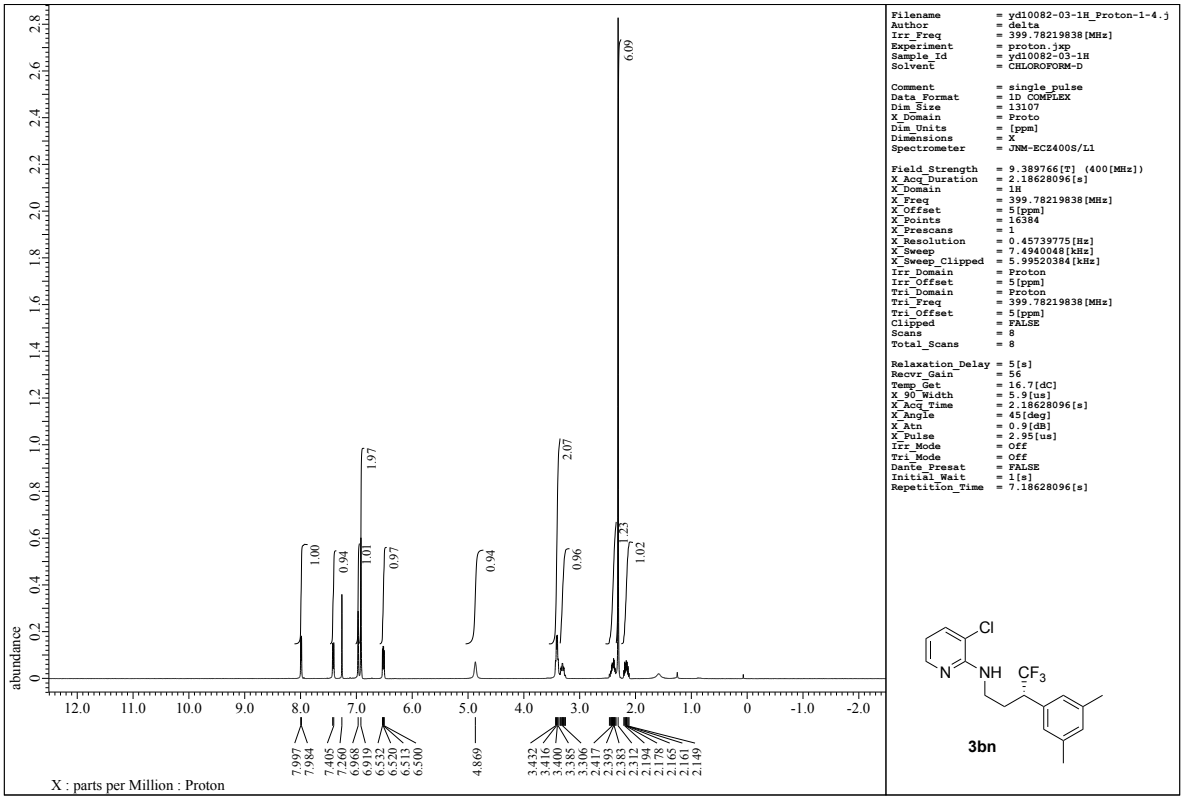
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	9.939	92255	68.087	6750
2	14.609	43241	31.913	2114

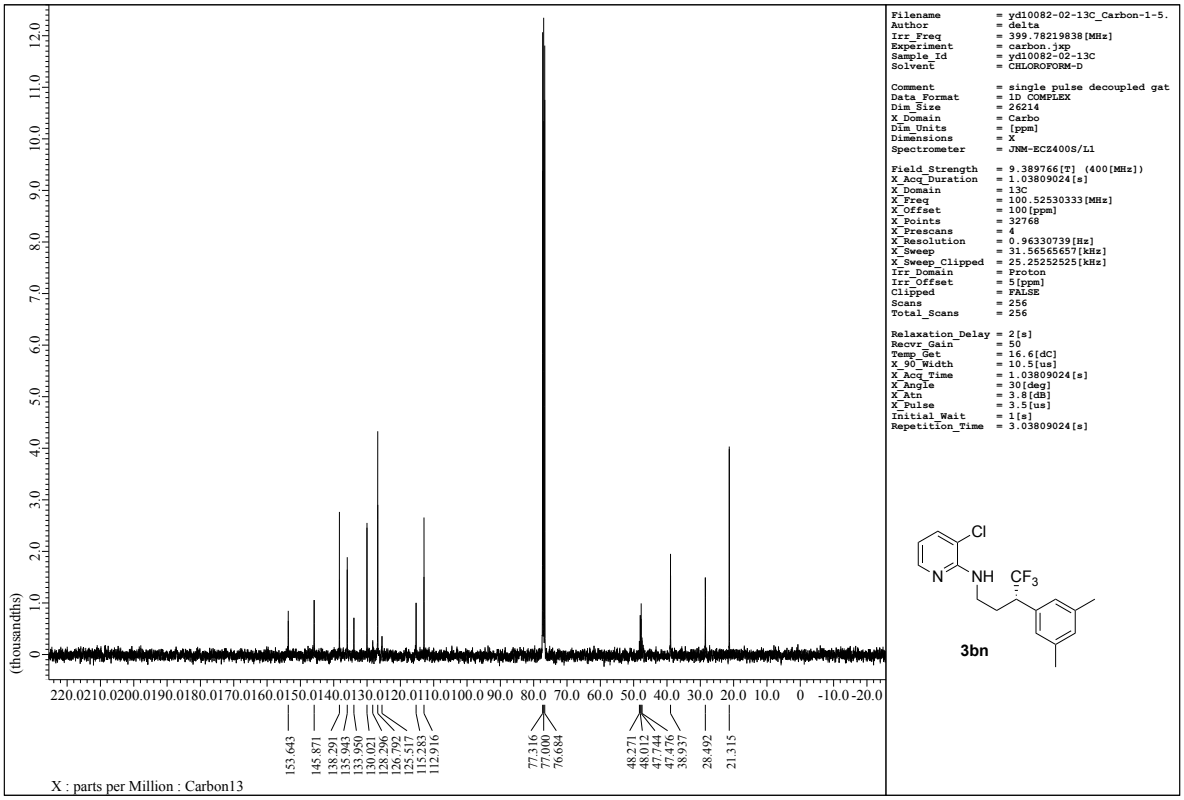


UV Results

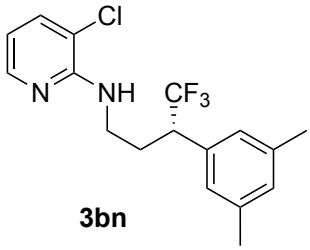
Pk #	Retention Time	Area	Area Percent	Height
1	9.643	74819	50.093	5854
2	14.019	74542	49.907	3880



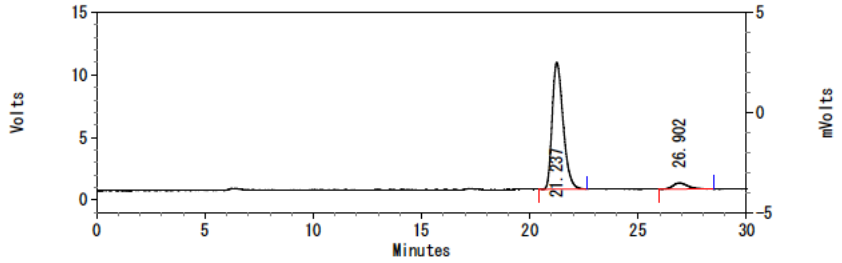

```
Filename          = yd10082-03-1H_Proton-1-4.j
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = yd10082-03-1H
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 16.7[dC]
X_90_Width        = 5.9[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 2.95[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```




```
Filename          = yd10082-02-13C_Carbon-1-5.
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = yd10082-02-13C
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 16.6[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```

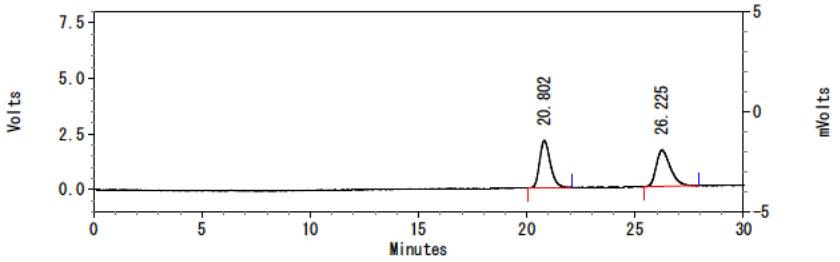


3bn



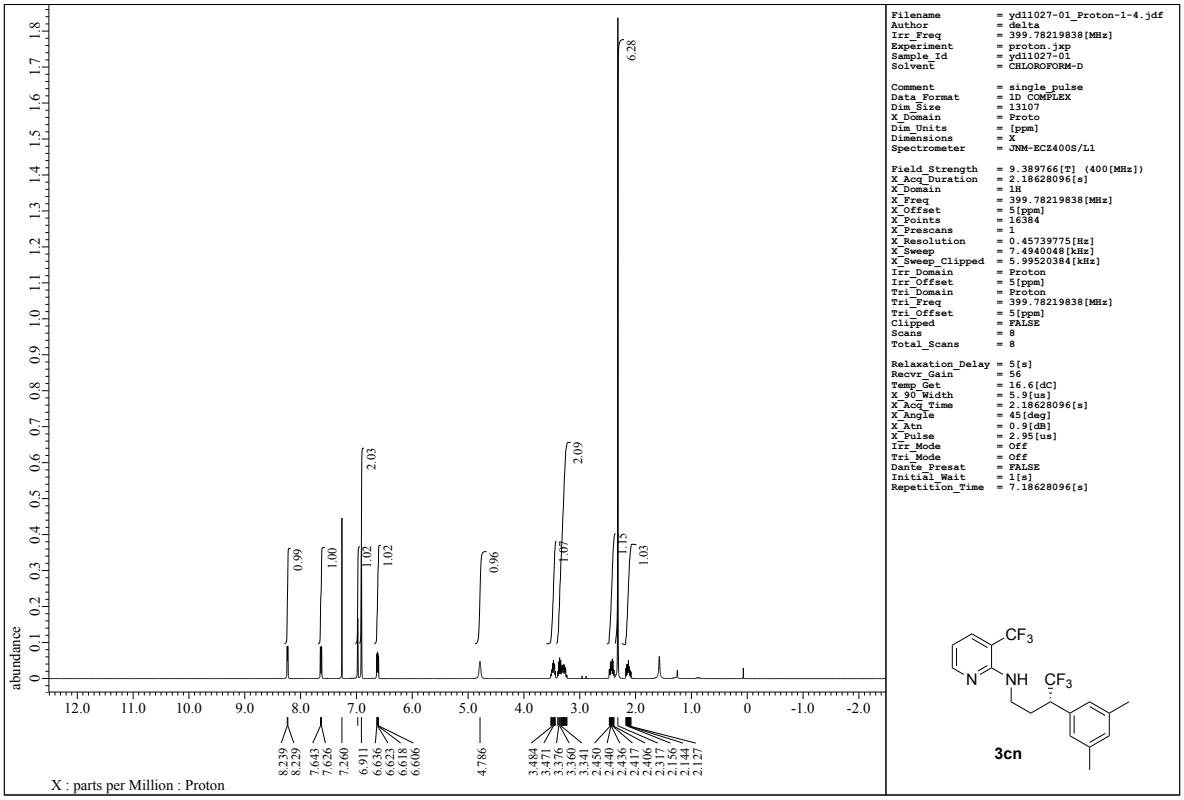
UV Results

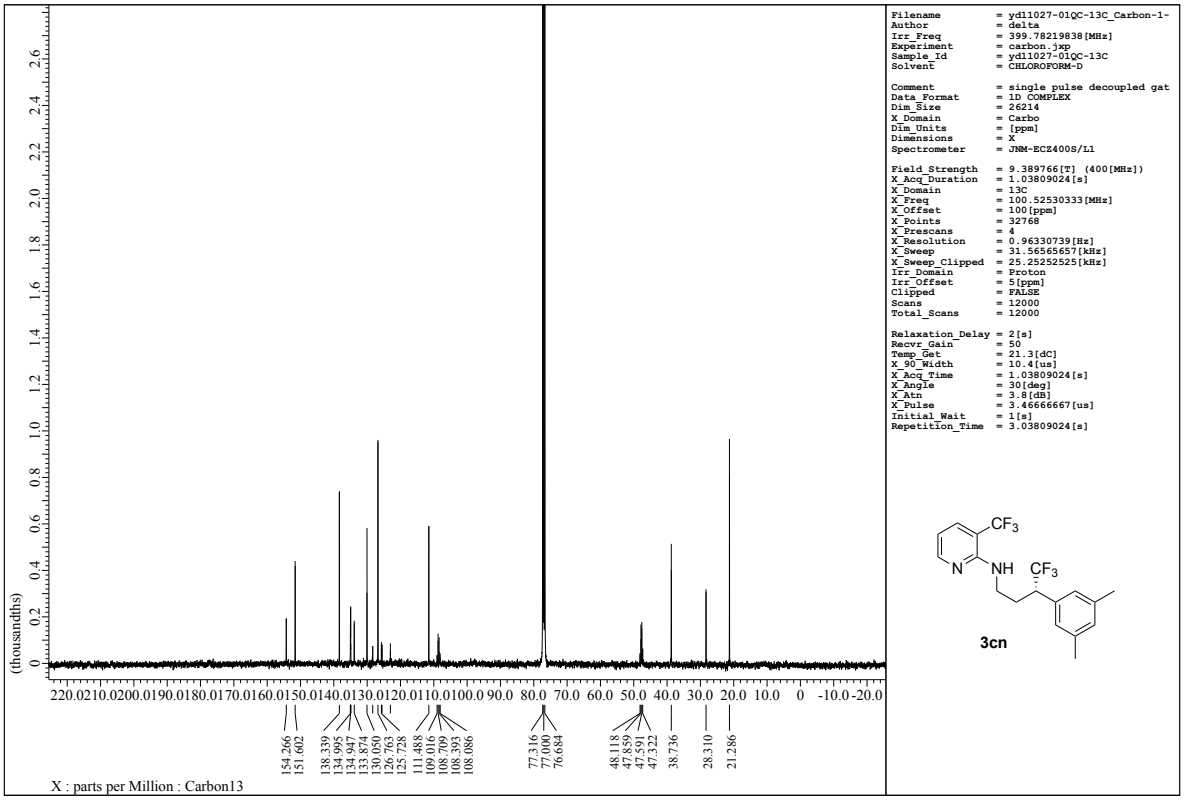
Pk #	Retention Time	Area	Area Percent	Height
1	21.237	356146	94.033	10123
2	26.902	22600	5.967	484

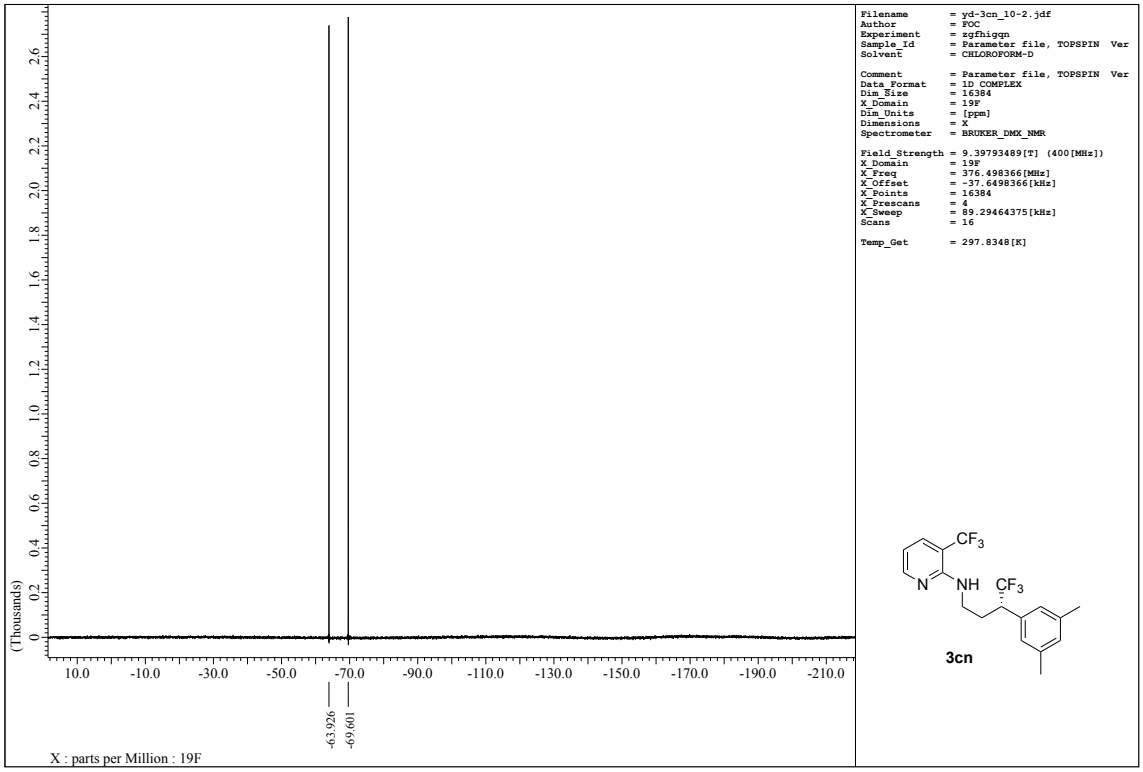


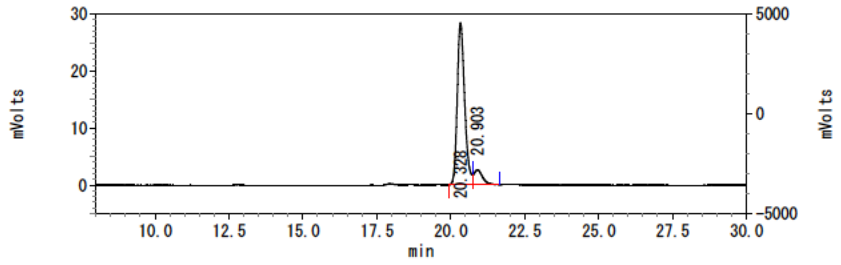
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	20.802	73046	49.805	2123
2	26.225	73618	50.195	1629

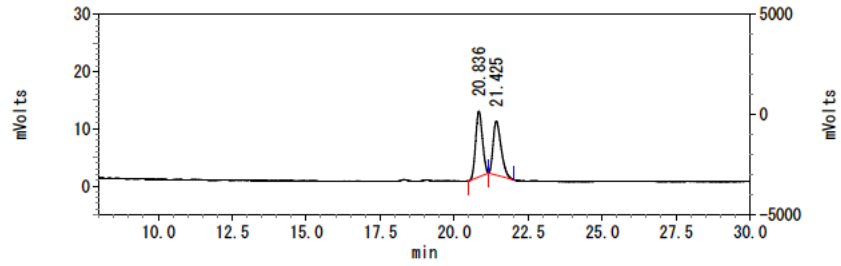

abundance
X : parts per Million : Proton
6.28
2.03
2.09
0.99
1.00
1.02
1.02
0.96
1.07
1.15
1.03
8.239
8.229
7.643
7.626
7.260
6.911
6.636
6.623
6.618
6.606
4.786
3.484
3.471
3.376
3.360
3.341
2.450
2.440
2.436
2.417
2.406
2.317
2.156
2.144
2.127
Filename = yd11027-01_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd11027-01
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 16.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CF3
N
NH
CF3
3cn


(thousandths)
X : parts per Million : Carbon13
154.266
151.602
138.339
134.995
134.947
133.874
130.050
126.763
125.728
111.488
109.016
108.709
108.393
108.086
77.316
77.000
76.684
48.118
47.859
47.591
47.322
38.736
28.310
21.286
Filename = yd11027-01QC-13C_Carbon-1-
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = yd11027-01QC-13C
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 12000
Total_Scans = 12000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.3[dC]
X_90_Width = 10.4[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.46666667[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CF3
N
NH
CF3
3cn

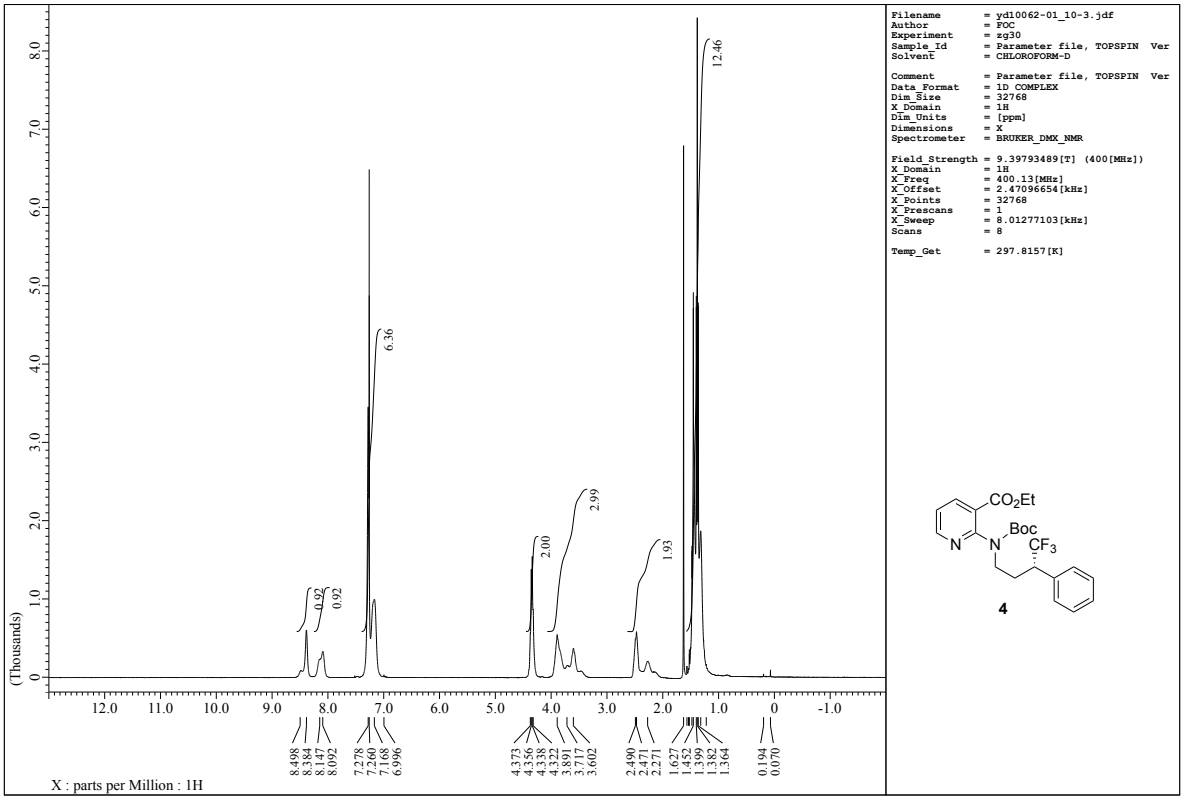


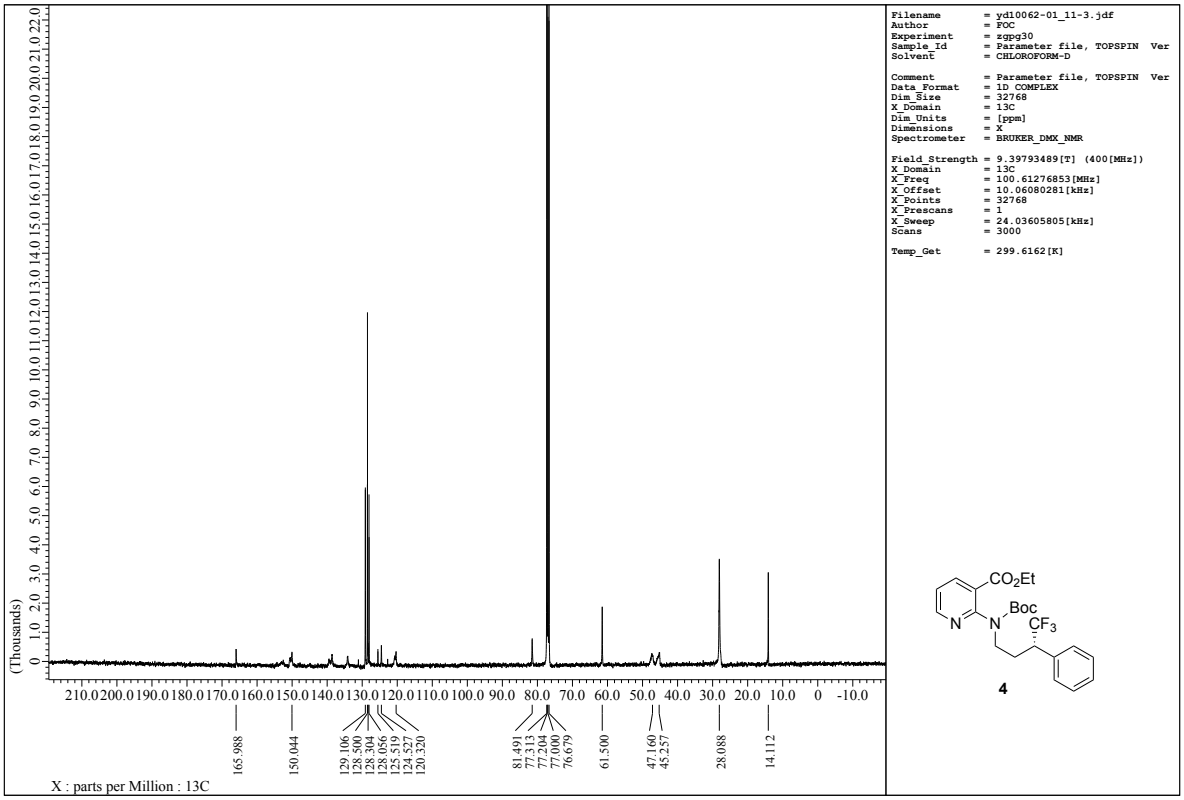


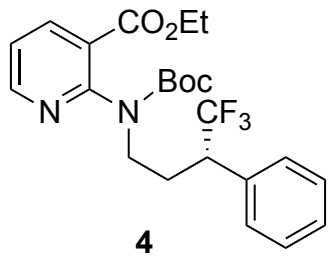
Pk #	Retention Time	Area	Area Percent
1	20.328	495474	90.284
2	20.903	53321	9.716



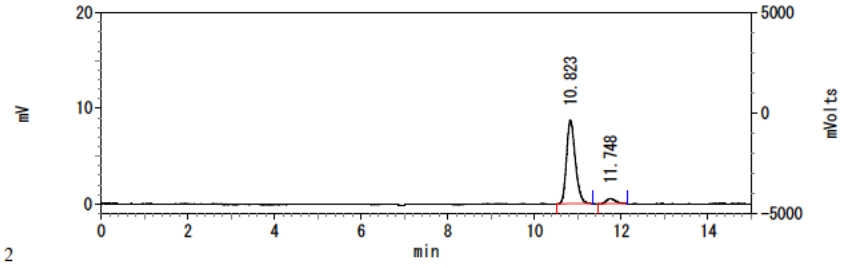
Pk #	Retention Time	Area	Area Percent
1	20.836	181653	50.672
2	21.425	176838	49.328


Filename = yd10062-01_10-3.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 8
Temp_Get = 297.8157[K]
(Thousands)
X : parts per Million : 1H
CO2Et
Boc
CF3
4

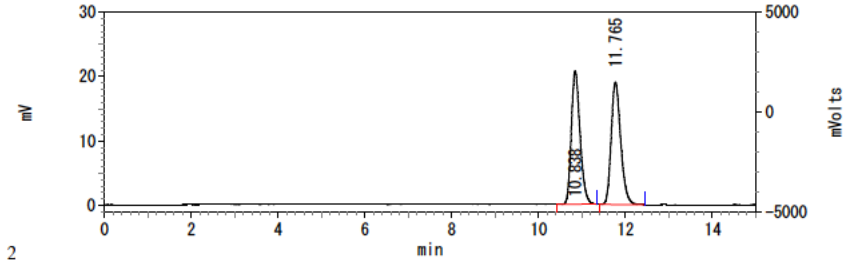

Filename = yd10062-01_11-3.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 3000
Temp_Get = 299.6162[K]
(Thousands)
X : parts per Million : 13C
CO2Et
Boc
CF3
4



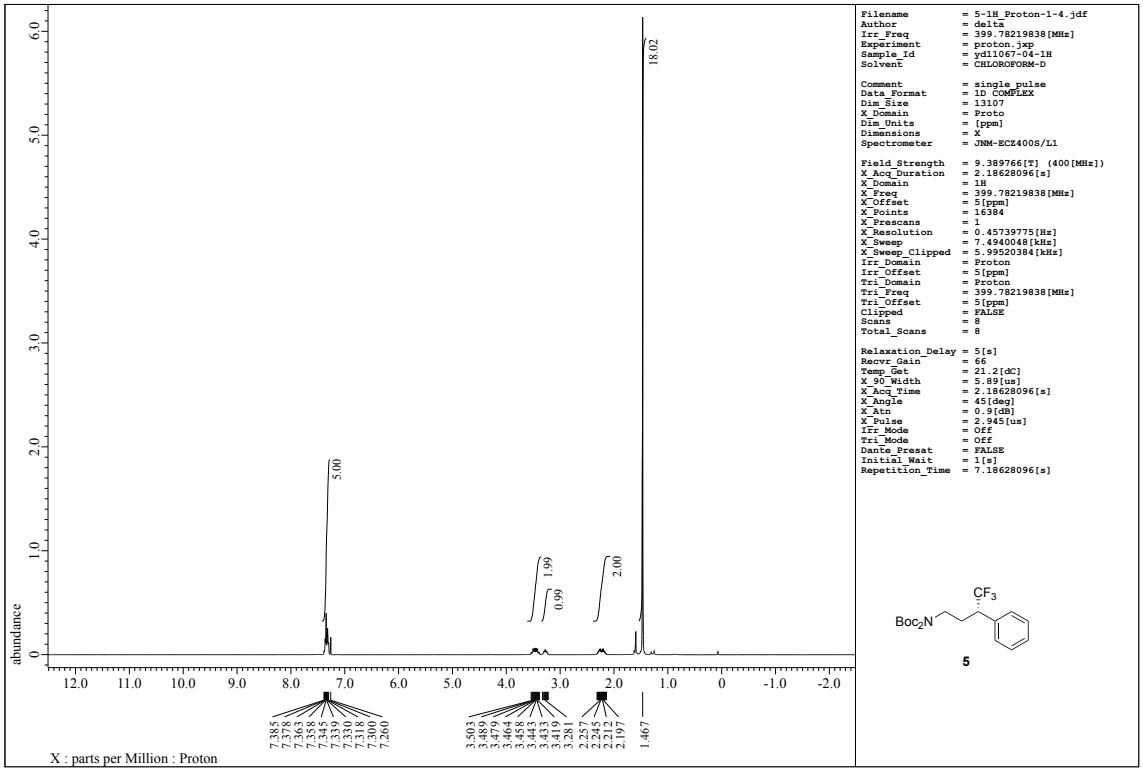
4

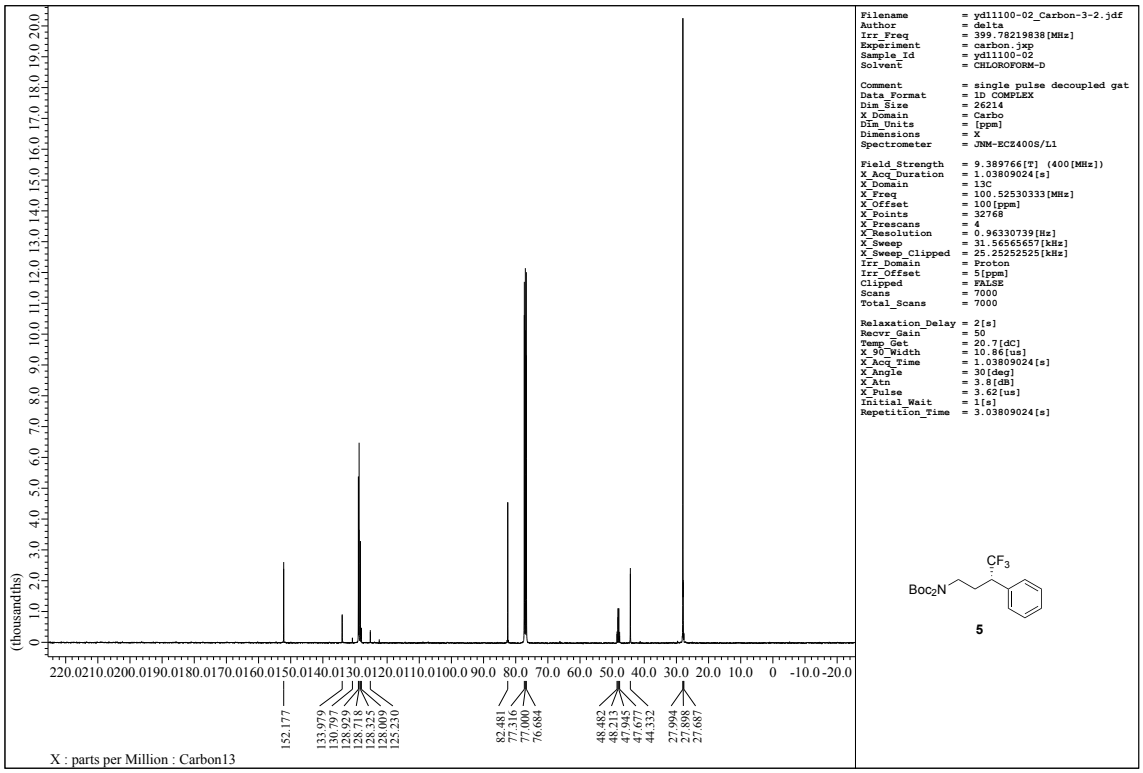


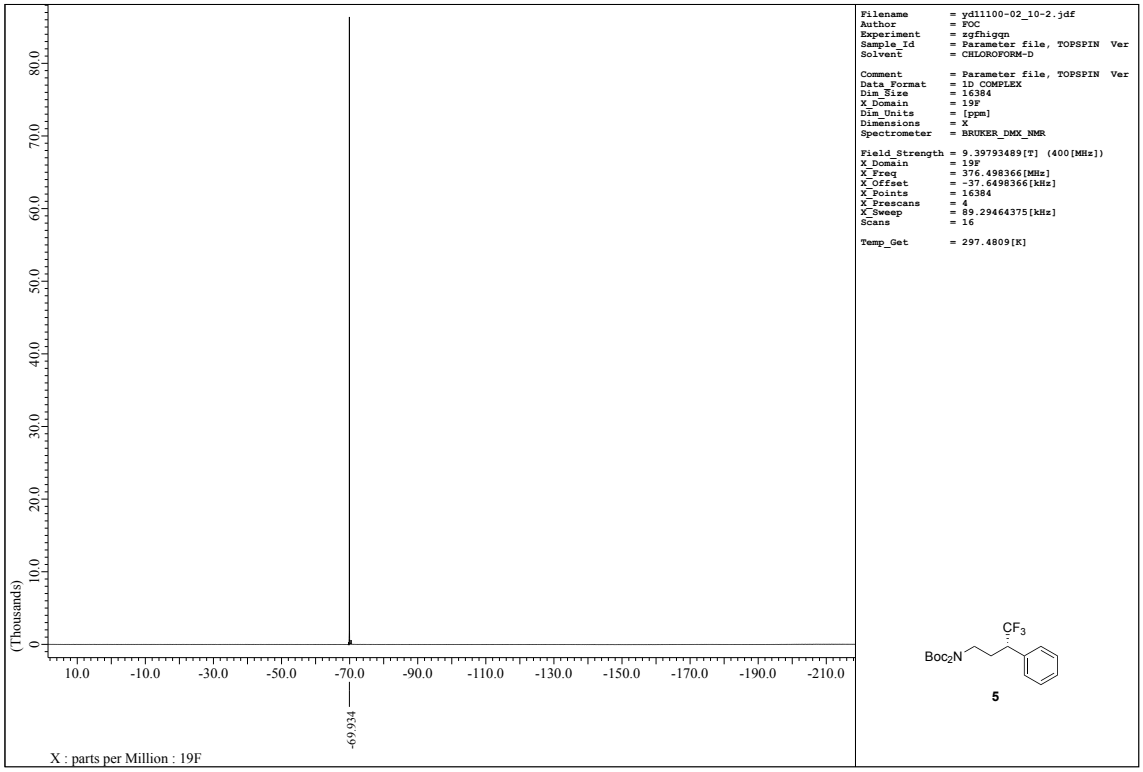
Pk #	Retention Time	Area	Area Percent
1	10.823	122411	94.023
2	11.748	7782	5.977

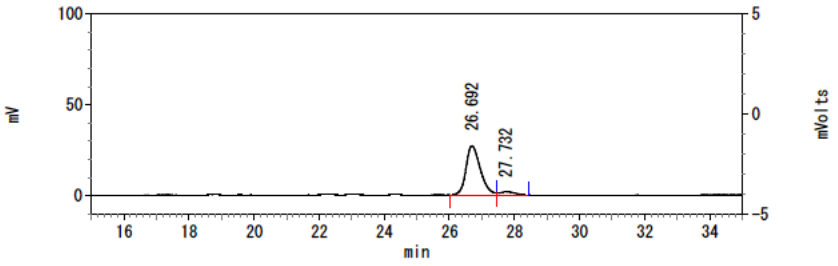


Pk #	Retention Time	Area	Area Percent
1	10.838	288554	49.937
2	11.765	289287	50.063

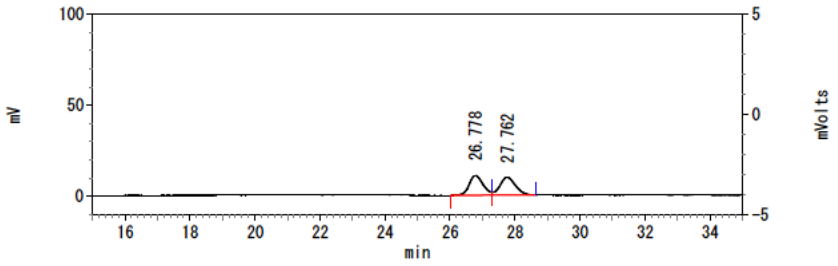

Filename = 5-1H_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1067-04-1H
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 66
Temp_Get = 21.2[dC]
X_90_Width = 5.89[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.945[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
18.02
5.00
1.99
0.99
2.00
7.385 7.381 7.365 7.358 7.345 7.339 7.330 7.318 7.300 7.260
3.503 3.489 3.479 3.464 3.458 3.443 3.433 3.419 3.281 2.257 2.245 2.213 2.197 1.467
X : parts per Million : Proton
CF3
Boc2N
5


Filename = ydl1100-02_Carbon-3-2.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = ydl1100-02
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 7000
Total_Scans = 7000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.7[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
152.177
133.979 130.797 128.929 128.718 128.235 128.009 125.230
82.481 77.316 77.000 76.684
48.482 48.213 47.945 47.677 44.332
27.994 27.898 27.687
X : parts per Million : Carbon13
CF3
Boc2N
5

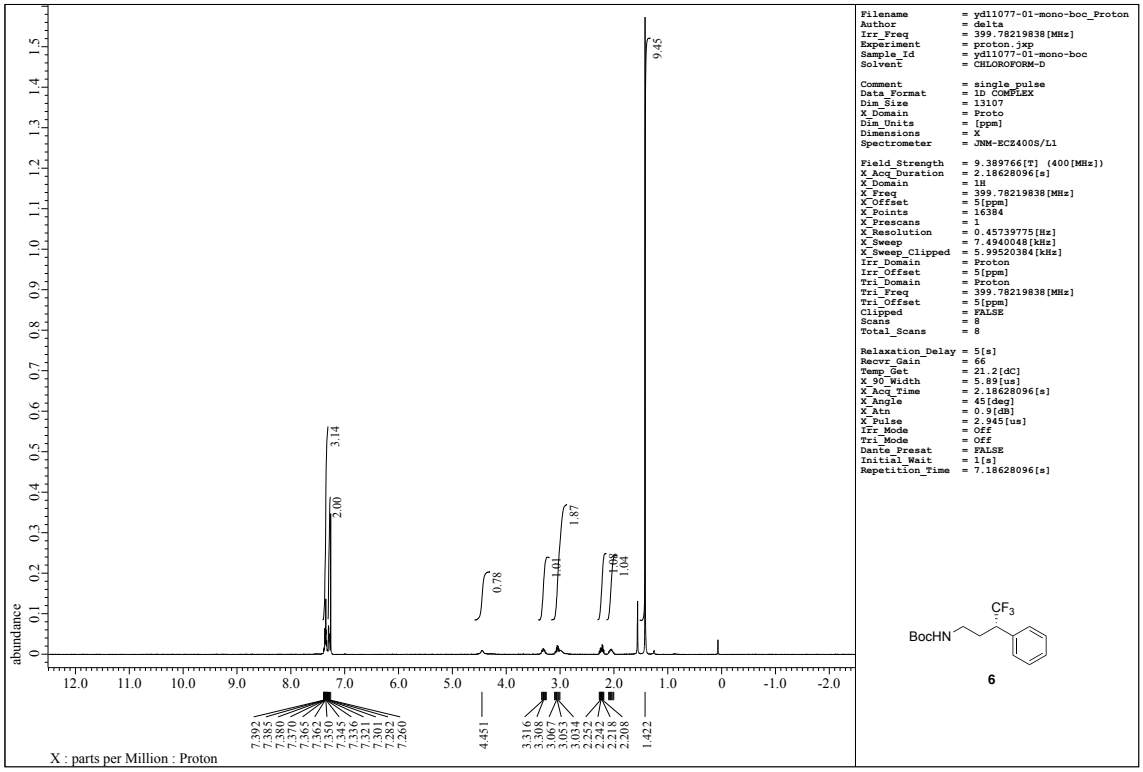


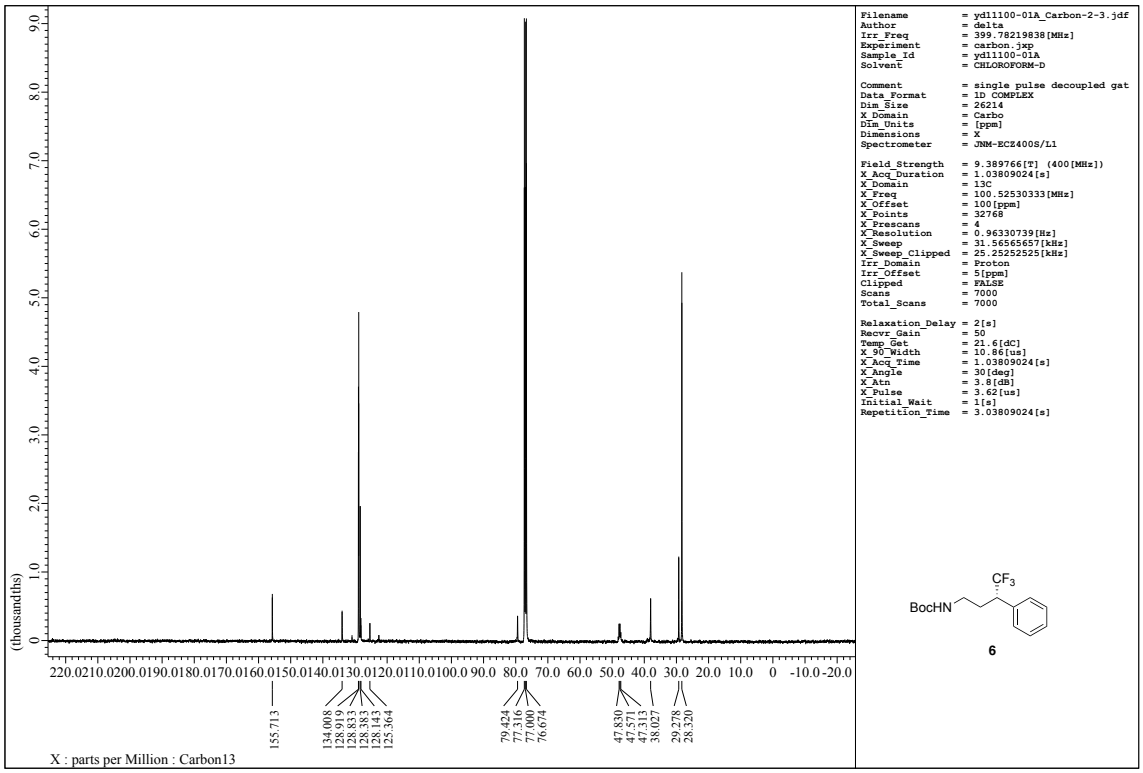


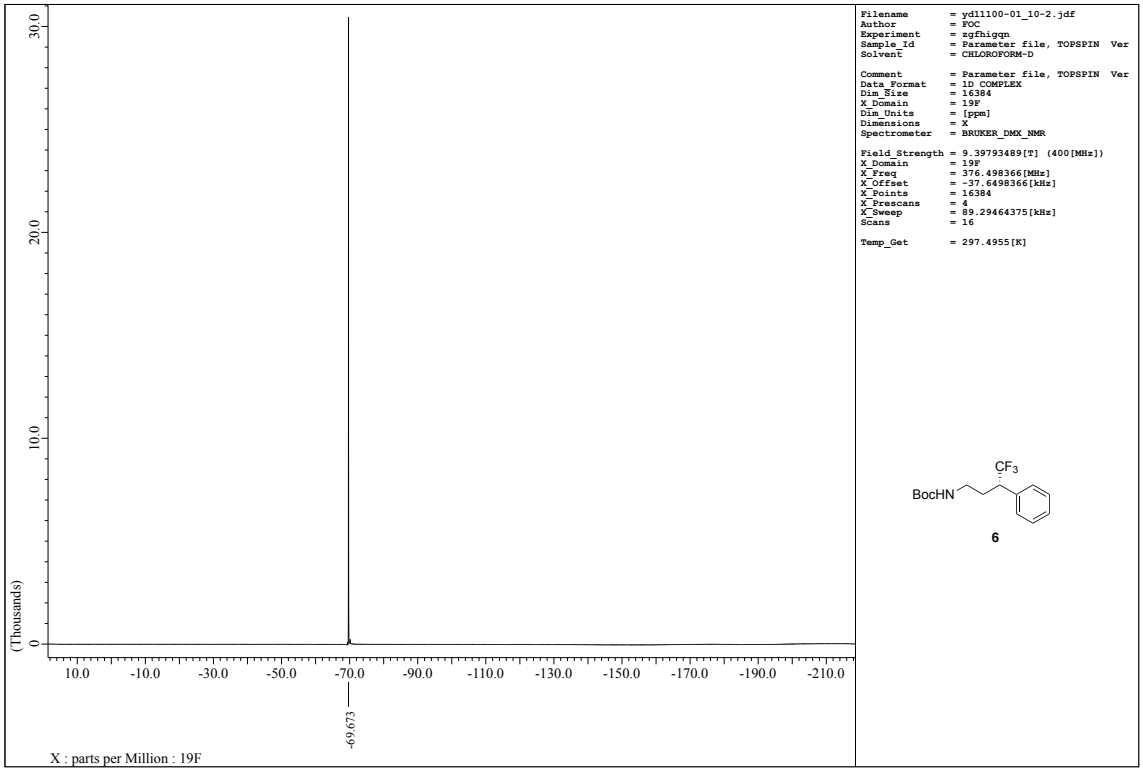
Pk #	Retention Time	Area	Area Percent
1	26.692	826587	93.893
2	27.732	53766	6.107

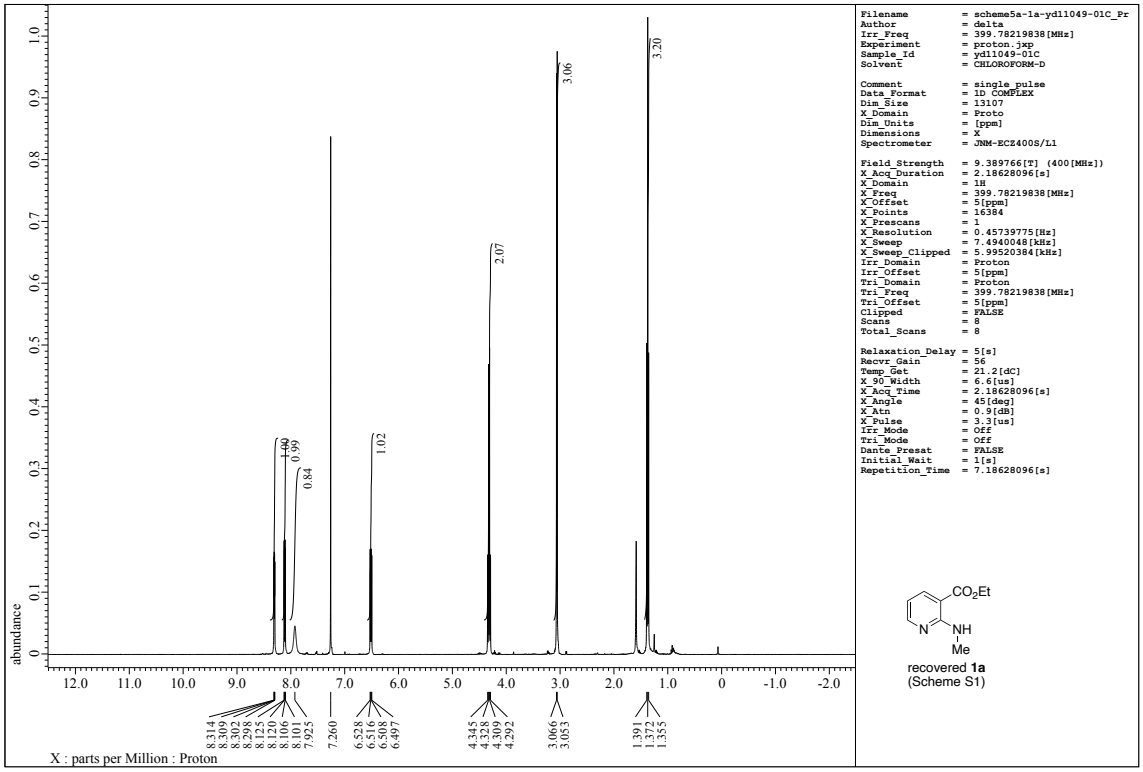


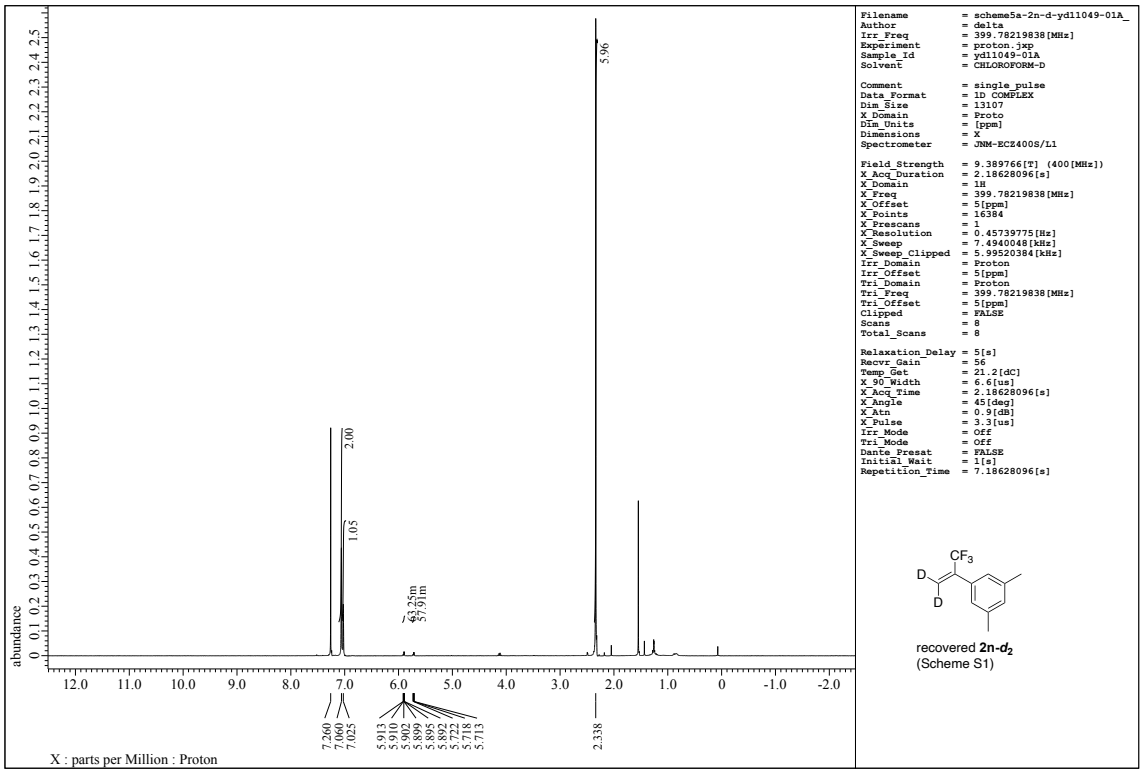
Pk #	Retention Time	Area	Area Percent
1	26.778	321124	49.984
2	27.762	321335	50.016

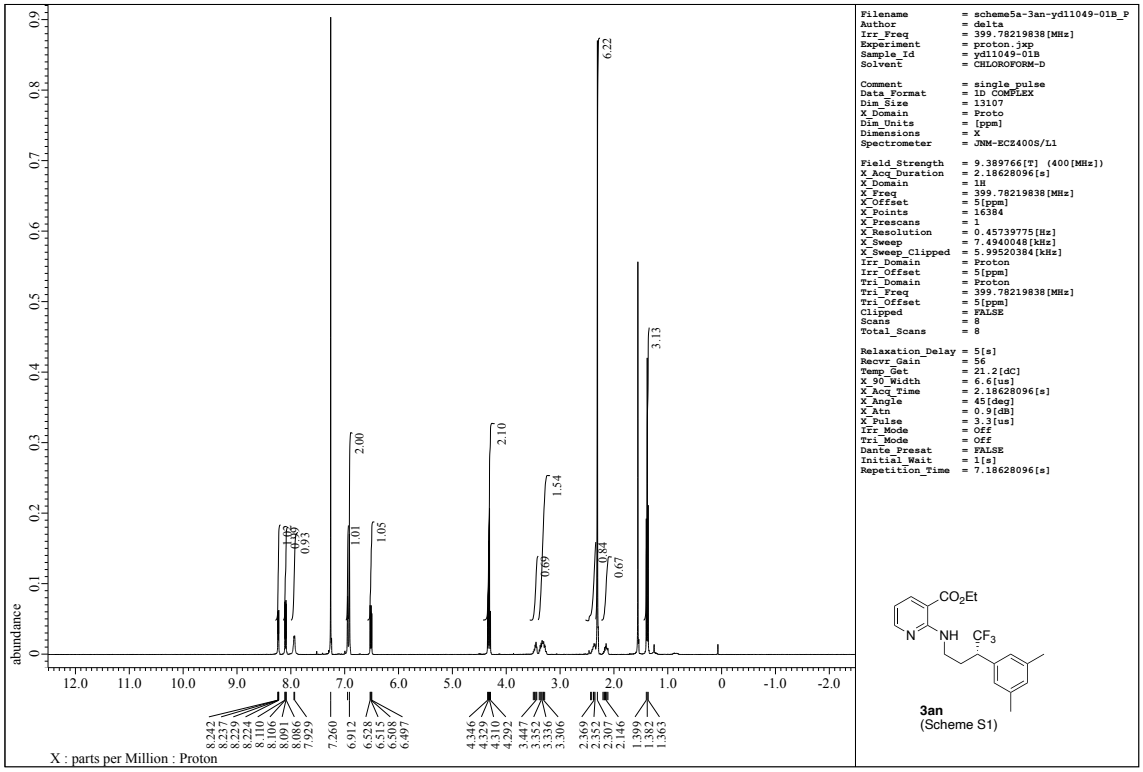

abundance
X : parts per Million : Proton
9.45
3.14
2.00
1.87
1.01
1.04
0.78
7.392
7.385
7.380
7.370
7.365
7.362
7.345
7.336
7.321
7.301
7.282
7.260
4.451
3.316
3.308
3.067
3.053
3.034
2.252
2.242
2.218
2.208
1.422
Filename = yd11077-01-mono-boc_Proton
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd11077-01-mono-boc
Solvent = CHLOROFORM-D
Comment = single pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 66
Temp_Get = 21.2[dC]
X_90_Width = 5.89[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.945[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
CF3
BocHN
6

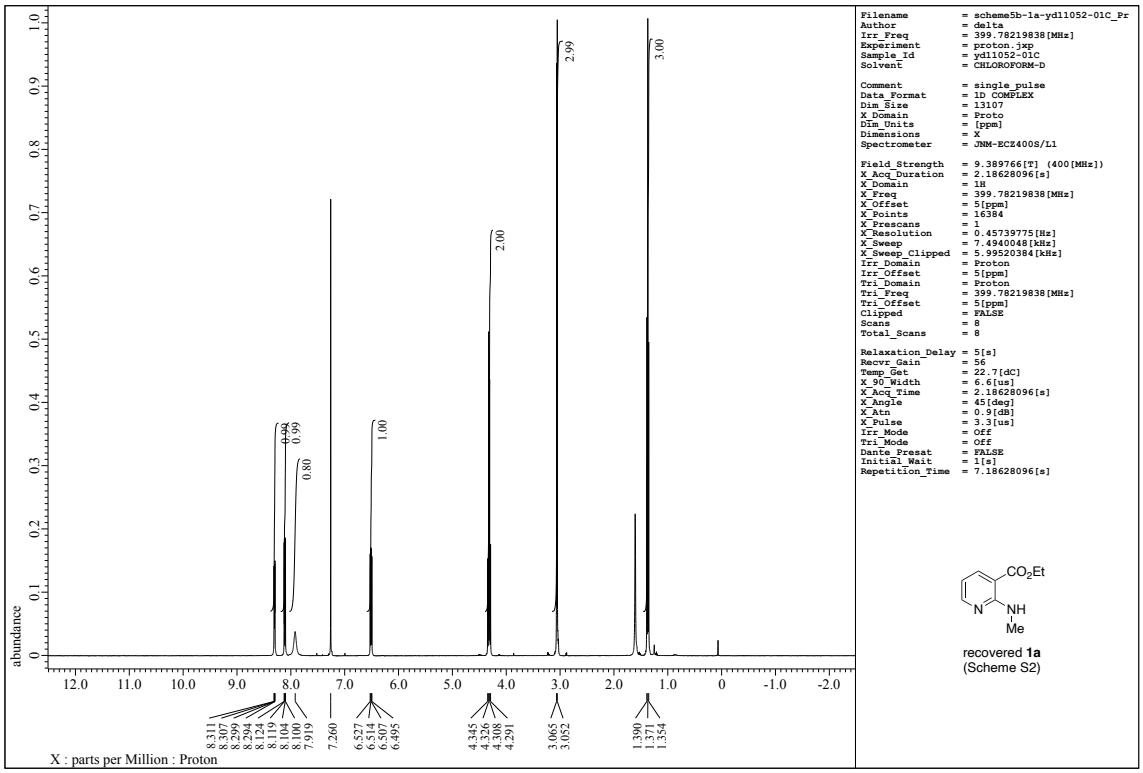

(thousandths)
X : parts per Million : Carbon13
155.713
134.008
128.919
128.833
128.383
128.143
125.364
79.424
77.316
77.000
76.674
47.830
47.571
47.313
38.027
29.278
28.320
Filename = yd11100-01A_Carbon-2-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = yd11100-01A
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 7000
Total_Scans = 7000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.6[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CF3
BocHN
6

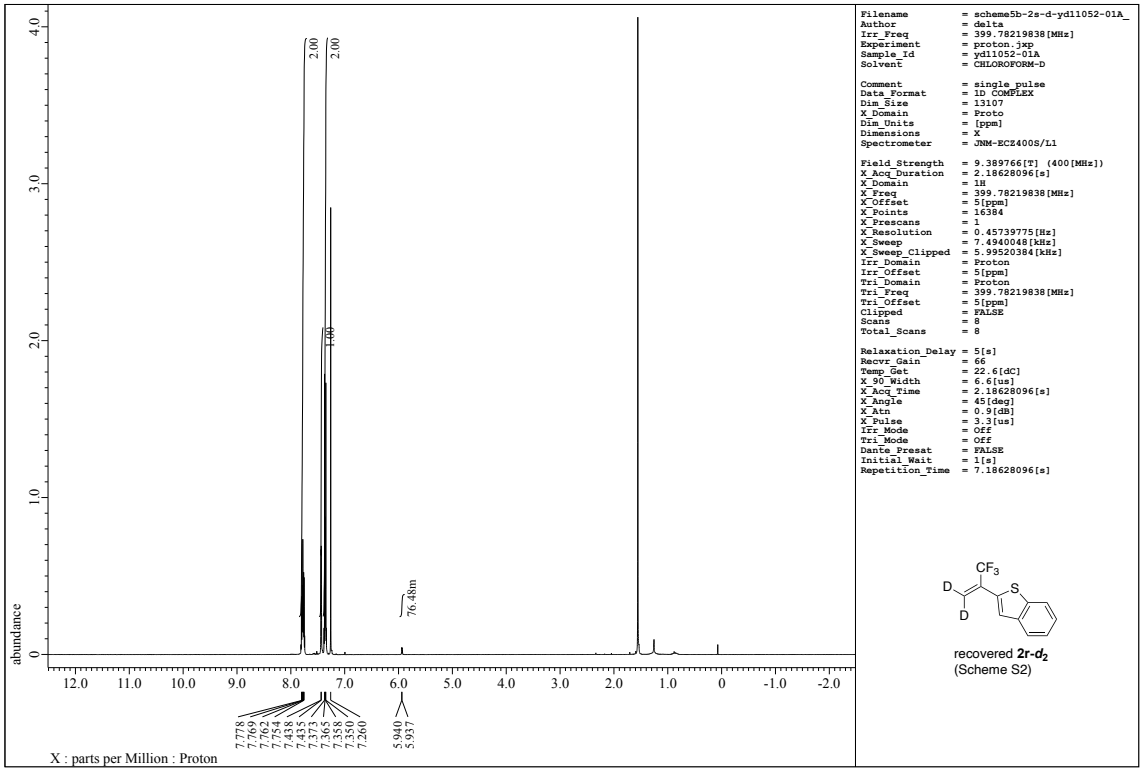

30.0
20.0
10.0
0
(Thousands)
10.0
-10.0
-30.0
-50.0
-70.0
-90.0
-110.0
-130.0
-150.0
-170.0
-190.0
-210.0
-69.673
X : parts per Million : 19F
Filename = yd11100-01_10-2.jdf
Author = FOC
Experiment = zgfhigqn
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 16384
X_Domain = 19F
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 19F
X_Freq = 376.498366[MHz]
X_Offset = -37.6498366[kHz]
X_Points = 16384
X_Prescans = 4
X_Sweep = 89.29464375[kHz]
Scans = 16
Temp_Get = 297.4955[K]
CF3
BocHN
6

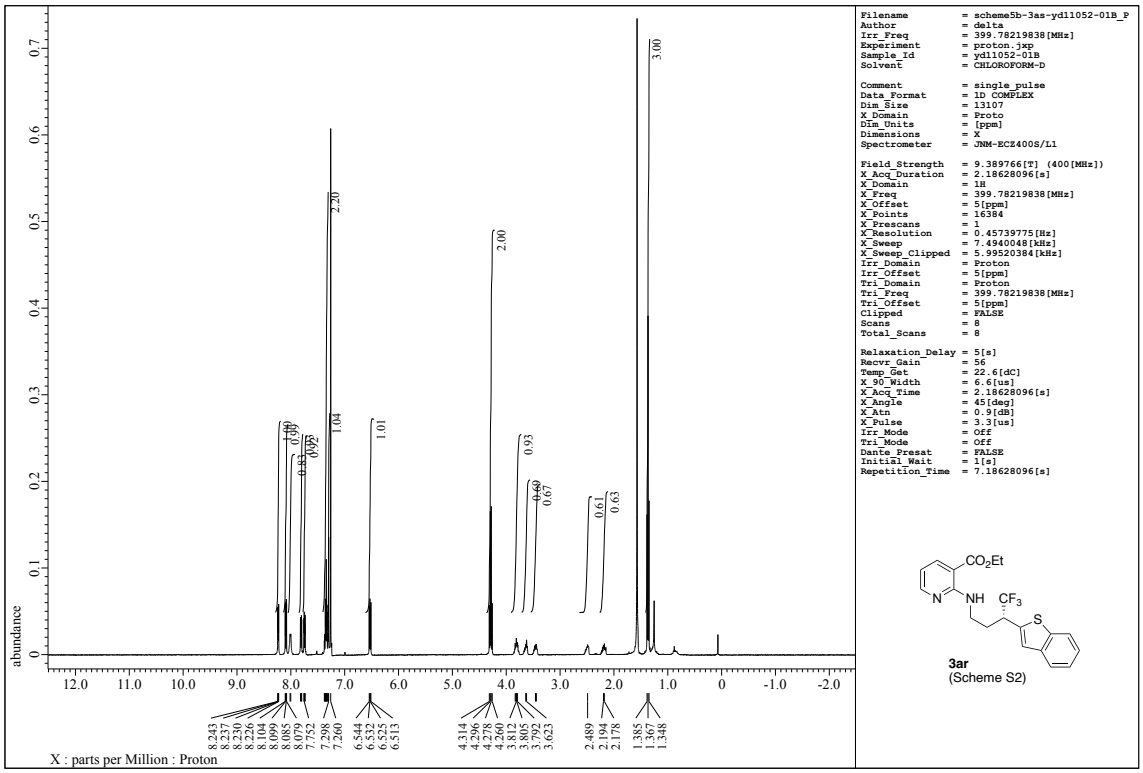

Filename = scheme5a-1a-ydl1049-01C_Pr
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1049-01C
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.2[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3.20
3.06
2.07
1.02
1.00
0.99
0.84
CO2Et
NH
Me
recovered 1a
(Scheme S1)


Filename = scheme5a-2n-d-ydl1049-01A_
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1049-01A
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.2[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
5.96
2.00
1.05
CF3
D
D
recovered 2n-d2
(Scheme S1)


Filename = scheme5a-3an-yd11049-01B_P
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd11049-01B
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.2[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
CO2Et
N
NH
CF3
3an
(Scheme S1)


Filename = scheme5b-1a-yd11052-01C_Pr
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = yd11052-01C
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.7[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
CO2Et
N
NH
Me
recovered 1a
(Scheme S2)


Filename = scheme5b-2s-d-ydl1052-01A_
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1052-01A
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 66
Temp_Get = 22.6[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
2.00
2.00
1.00
76.48mn
7.778
7.769
7.762
7.754
7.435
7.433
7.373
7.365
7.358
7.350
7.260
5.940
5.937
CF3
D
S
D
recovered 2r-d2
(Scheme S2)


Filename = scheme5b-3as-ydl1052-01B_P
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = ydl1052-01B
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.6[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3.00
2.20
2.00
1.04
1.01
0.93
0.60
0.67
0.61
0.63
CO2Et
N
NH
CF3
S
3ar
(Scheme S2)