Electronic Supplementary Material (ESI) for ChemComm.


**Supplementary Information**

# Asymmetric addition of *N*-methyl C($sp^3$)–H bond to cyclic alkenes enabled by an iridium/phosphine-olefin catalyst

Kana Sakamoto and Takahiro Nishimura*

Department of Chemistry, Graduate School of Science, Osaka Metropolitan University, Sumiyoshi, Osaka 558-8585, Japan. E-mail: tnishi@omu.ac.jp

**Contents of Supplementary Information:**

## 1. General information

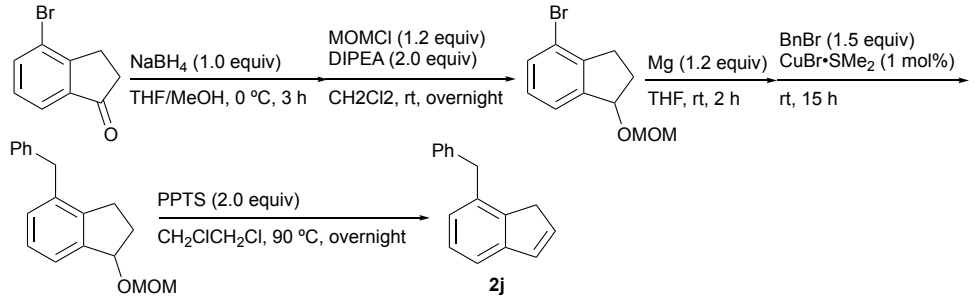
All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under pre-dried nitrogen. NMR spectra were recorded on either a JEOL JNM ECZ-400 spectrometer (400 MHz for 1 H, 100 MHz for $^{13}C$) or a Bruker Avance III HD 400 spectrometer (400 MHz for 1 H, 100 MHz for $^{13}C$). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) for $^1H$ NMR, and $CDCl_3$ (δ 77.00) for $^{13}C$ NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: quint, quintet: sext, sextet: sept, septet; m, multiplet: br, broad. Optical rotations were measured on a JASCO P-2200 polarimeter. High-resolution mass spectra were obtained with JEOL AccuTOF LC-plus 4G spectrometer. Flash column chromatography was performed with Silica Gel 60 N (Wako). Preparative thin-layer chromatography was performed with Wakogel® B-5F (Wako).

## 2. Materials

Dehydrated solvents were purchased and used after being deoxygenated by bubbling $N_2$. $[IrCl(coe)_2]_2$[1] and $NaBAr^F_4$ [$Ar^F$ = 3,5-$(CF_3)_2C_6H_3$][2] were prepared according to the reported procedures.

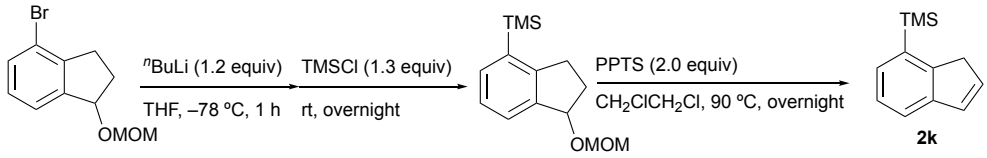
## 3. Preparation of substrates 1 and 2

Compounds **1a** (CAS: 1036584-14-1),[3] **1b** (CAS: 468718-67-4),[3] **1c** (CAS: 103976-61-0),[3] **1d** (CAS: 1251349-49-1),[3] **1e** (CAS: 156267-13-9),[3] **1g** (937602-15-8),[3] **1a-*d*$_3$**,[4] **2b** (CAS: 78383-19-4),[5] **2c** (CAS: 2576641-39-7),[5] **2d** (CAS: 2576641-40-0),[5] **2e** (CAS: 2576641-41-1),[5] **2f**,[5] **2g** (CAS: 2576641-44-4),[5] **2h**,[5] **2i**,[5] **2m** (CAS: 4453-90-1),[6] **2n** (CAS: 75715-21-8),[7] **2q** (CAS: 19345-99-4),[8] and **2r** (CAS: 912329-24-9)[5] were prepared according to the reported procedures. Compounds **2j**, **2k,** and **2o** (CAS: 62791-44-0) were prepared as shown below. Other chemicals were purchased from commercial suppliers and used as received.



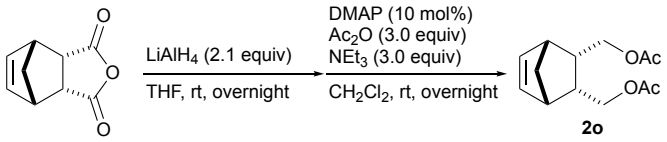

To a solution of 4-bromoindanone (2.1 g, 10 mmol) in THF (20 mL) and MeOH (20 mL) was added $NaBH_4$ (378 mg, 10 mmol, 1.0 equiv) at 0 ºC and the mixture was stirred at the same temperature for 3 h. The reaction was quenched with $NH_4Cl$aq, and the resulting mixture was extracted with EtOAc (x3). The combined organic layer was dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was passed through a short column of silica gel with EtOAc as an eluent, and the filtrate was concentrated on a rotary evaporator. The residue was subjected to the next reaction without further purifications. The residue and *N*,*N*-

diisopropylethylamine (DIPEA, 5.1 mL, 30 mmol, 2.0 equiv) were dissolved in $CH_2Cl_2$ (15 mL). Chloromethyl methyl ether (MOMCl, 1.8 mL, 24 mmol, 1.2 equiv) was added dropwise to the solution at 0 ºC under $N_2$, and the mixture was stirred at room temperature for 24 h. The reaction was quenched with $H_2O$, and the aqueous layer was extracted with $CH_2Cl_2$ (x3). The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane/EtOAc (20:1) to give 4-bromo-2,3-dihydro-1-(methoxymethoxy)-1*H*-indene (2.52 g, 80% yield). To a suspension of Mg turnings (280 mg, 11.5 mmol, 1.2 equiv) in a small amount of THF was added 4-bromo-2,3-dihydro-1-(methoxymethoxy)-1*H*-indene (2.47 g, 9.6 mmol) in THF (10 mL) dropwise under $N_2$, and the resulting solution was stirred at room temperature for 2 h. BnBr (1.70 mL, 14.4 mmol, 1.5 equiv) and CuBr•$SMe_2$ (19.7 mg, 0.096 mmol, 1 mol%) were added to the mixture at 0 ºC, and the mixture was stirred at room temperature for 15 h. The reaction was quenched with $NH_4$Claq, and the aqueous layer was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane/EtOAc (20:1) to give 4-benzyl-2,3-dihydro-1-(methoxymethoxy)-1*H*-indene (1.24 g, 4.6 mmol, 48% yield). A mixture of 4-benzyl-2,3-dihydro-1-(methoxymethoxy)-1*H*-indene (1.07 g, 4.0 mmol) and PPTS (2.01 g, 8.0 mmol, 2.0 equiv) in 1,2-dichloroethane (20 mL) was heated to 90 ºC and stirred overnight. The reaction was quenched with $H_2O$, and the aqueous layer was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane to give 7-benzylindene (**2j**) (685 mg, 3.3 mmol, 83% yield).



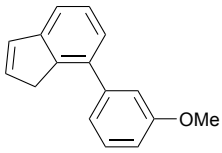

To a solution of 4-bromo-2,3-dihydro-1-(methoxymethoxy)-1*H*-indene (1.29 g, 5 mmol) in THF was added dropwise BuLi (15 w/w% hexane solution, 3.8 mL, 6 mmol, 1.2 equiv) at –78 ºC under $N_2$, and the resulting mixture was stirred at the same temperature for 1 h. Chlorotrimethylsilane (TMSCl, 0.79 mL, 7.0 mmol, 1.3 equiv) was added dropwise to the mixture at –78 ºC, and the mixture was stirred overnight. The reaction was quenched with $H_2O$, and the aqueous layer was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane/EtOAc (20:1) to give 2,3-dihydro-1-(methyoxymethoxy)-4-trimethylsilyl-1*H*-indene (554 mg, 2.2 mmol, 55% yield). The mixture of 2,3-dihydro-1-(methoxymethoxy)-4-trimethylsilyl-1*H*-indene (554 mg, 2.2 mmol) and pyridinium *p*-toluene sulfonate (PPTS, 1.10 g, 4.4 mmol, 2.0 equiv) in 1,2-dichloroethane (10 mL) was heated to 90 ºC and stirred overnight. The reaction was quenched with $H_2O$, and the aqueous layer was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated on a

rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane to give 7-benzylindene (**2j**, 160 mg, 0.85 mmol, 39% yield).



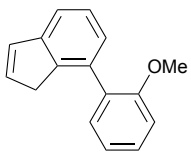

Carbic anhydride was prepared according to the reported procedure.[9] To a solution of $LiAlH_4$ (797 mg, 21 mmol, 2.1 equiv) in THF (20 mL) was added carbic anhydride (1.64 g, 10 mmol) in portions at 0 ºC, and the resulting mixture was stirred at room temperature overnight. The reaction was quenched by adding $H_2O$ (0.8 mL) slowly, 15% NaOH (0.8 mL), and then, $H_2O$ (2.4 mL) at 0 ºC. The mixture was stirred at room temperature for 2 h. The resulting precipitate was removed through a pad of celite eluted with $Et_2O$. The filtrate was concentrated on a rotary evaporator and the residue was used for the next reaction without further purification. To a solution of the residue, 4-dimthylaminopyridine (DMAP, 12.2 mg, 0.10 mmol, 10 mol%), and $NEt_3$ (4.2 mL, 30 mmol, 3.0 equiv) in $CH_2Cl_2$ (50 mL) was added $Ac_2O$ (2.8 mL, 30 mmol, 3.0 equiv) dropwise at 0 ºC. The resulting mixture was stirred overnight at room temperature. The reaction was quenched with $H_2O$, and the aqueous layer was extracted with EtOAc (x3). The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane/EtOAc to give **2o** (2.19 g, 9.2 mmol, 92% yield).

## 4. Characterization of substrates




**2f**

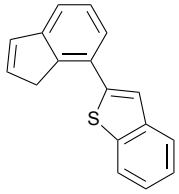
$^1$H NMR ($CDCl_3$) δ 7.43 (dd, *J* = 7.4, 1.0 Hz, 1H), 7.43–7.32 (m, 2H), 7.25 (d, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.11 (t, *J* = 2.0 Hz, 1H), 6.95 (td, *J* = 5.6, 2.0 Hz, 1H), 6.93 (dd, *J* = 8.0, 2.8 Hz, 1H), 6.59 (td, *J* = 5.6, 2.0 Hz, 1H), 3.86 (s, 3H), 3.50 (t, *J* = 2.0 Hz, 2H); $^{13}$C NMR ($CDCl_3$) δ 159.6, 145.4, 142.6, 141.2, 137.7, 134.4, 132.0, 129.4, 126.9, 125.1, 120.9, 120.2, 114.1, 112.5, 55.2, 39.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{15}O_1$ 223.1123; Found 223.1117.




**2h**

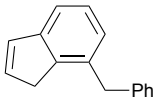
$^1$H NMR ($CDCl_3$) δ 7.45 (d, *J* = 7.2 Hz, 1H), 7.46–7.29 (m, 3H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.08 (d, *J* = 8.8 Hz, 1H), 7.03 (d, J = 8.8 Hz, 1H), 6.96 (d, *J* = 5.6 Hz, 1H), 6.58 (d, *J* = 5.6 Hz, 1H), 3.80

(s, 3H), 3.33 (s, 2H); $^{13}$C NMR ($CDCl_3$) δ 156.4, 144.6, 142.8, 134.6, 134.4, 131.9, 131.0, 129.9, 128.7, 126.3, 126.2, 120.4, 120.1, 110.8, 55.3, 38.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{15}O_1$ 223.1123; Found 223.1123.



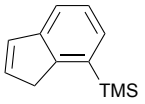

**2i**

$^1$H NMR ($CDCl_3$) δ 7.72 (s, 1H), 7.62 (d, $J$ = 8.0 Hz, 2H), 7.52 (dd, $J$ = 7.6, 2.0 Hz, 1H), 7.49–7.40 (m, 3H), 7.33 (t, $J$ = 7.4 Hz, 1H), 6.92 (dt, $J$ = 5.6, 2.0 Hz, 1H), 6.60 (dt, $J$ =5.6, 2.0 Hz, 1H), 3.48 (s, 2H); $^{13}$C NMR ($CDCl_3$) δ 145.9, 143.4, 140.6, 140.3, 139.3, 134.4, 131.8,130.1, 127.1, 124.7, 124.4, 124.2, 123.5, 122.0, 121.4, 121.1, 40.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{17}H_{13}S_1$ 249.0738; Found 249.0732.



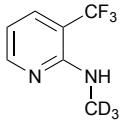

**2j**

$^1$H NMR ($CDCl_3$) δ 7.33–7.21 (m, 4H), 7.24–7.15 (m, 3H), 7.01 (d, $J$ = 7.6 Hz, 1H), 6.89 (dt, $J$ = 5.6, 2.0 Hz, 1H), 6.53 (dt, $J$ = 5.6, 2.0 Hz, 1H), 4.08 (s, 2H), 3.26 (t, $J$ = 2.0 Hz, 2H); $^{13}$C NMR ($CDCl_3$) δ 144.9, 142.3, 140.2, 135.9, 133.8, 132.2, 128.8, 128.4, 126.8, 126.0, 125.7, 119.3, 39.5, 37.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{15}$ 207.1174; Found 207.1181.




**2k**

$^1$H NMR ($CDCl_3$) δ 7.42 (dd, $J$ = 7.2, 1.2 Hz, 1H), 7.35 (dd, $J$ = 7.2, 1.2 Hz, 1H), 7.27 (t, $J$ = 7.2 Hz, 1H), 6.90 (dt, $J$ = 5.4, 2.0 Hz, 1H), 6.58 (dt, $J$ = 5.4, 2.0 Hz, 1H), 3.44 (t, $J$ = 2.0 Hz, 2H), 0.35 (s, 9H); $^{13}$C NMR ($CDCl_3$) δ 144.9, 143.8, 134.0, 132.0, 130.1, 125.7, 121.9, 40.2, 18.4, –0.76. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{17}Si_1$ 189.1099; Found 189.1094.




**1a-*d*$_3$**

$^1$H NMR ($CDCl_3$) δ 7.64 (d, $J$ = 8.0 Hz, 1H), 6.60 (dd, $J$ = 8.0, 4.8 Hz, 1H), 8.29 (d, $J$ = 4.8 Hz, 1H), 4.90 (bs, 1H).; $^{13}$C NMR ($CDCl_3$) δ 155.2, 151.6, 134.8 (q, $J_{F–C}$ = 5 Hz), 124.5 (q, $J_{F–C}$ = 270

Hz), 111.0, 108.6 (q, $J_{F-C}$ = 31 Hz), 27.8 (sept, $J_{D-C}$ = 21 Hz). HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_7{}^1H_5{}^2H_3F_3N_2$ 180.0824; Found 180.0828.

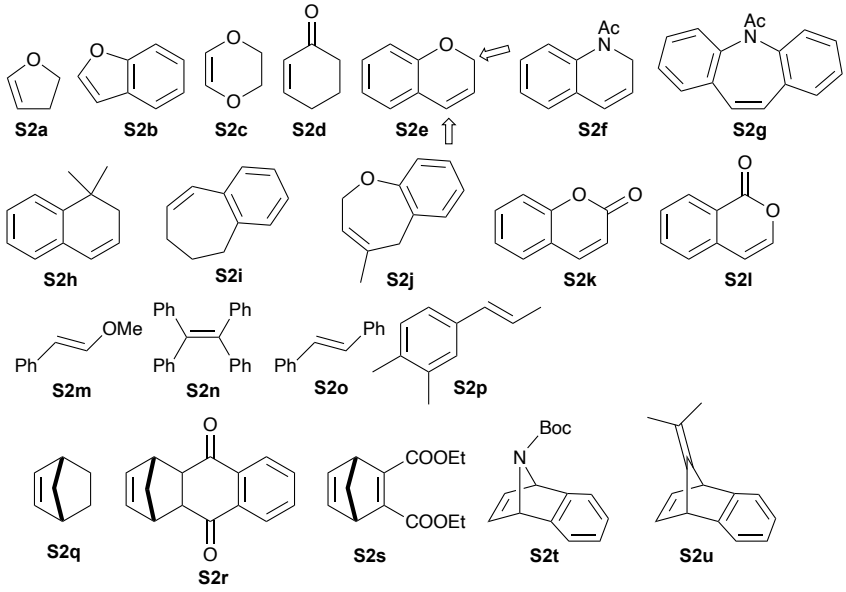
## 5. General procedure for Table 1 and Scheme 2

A mixture of $[IrCl(coe)_2]_2$ (2.2 mg, 0.0025 mmol, 5 mol% Ir), ligand (6 mol%), and $NaBAr^F{}_4$ (9.2 mg, 0.010 mmol, 10 mol%) in toluene (0.2 mL) in a Schlenk tube was stirred at room temperature for 10 min under $N_2$, and then, 2-(*N*-methylamino)pyridine **1** (0.10 mmol) and indene (**2a**, 34.8 mg, 0.30 mmol) were added to the mixture. The mixture was stirred at 100 ºC for 18 h. After the reaction mixture was concentrated on a rotary evaporator, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (10:1) to give the addition product including a small amount of impurities. The mixture was subjected again to preparative TLC on silica gel eluted with hexane/$CH_2Cl_2$ (2:1) to give the product **3aa**.

## 6. General procedure for Scheme 3

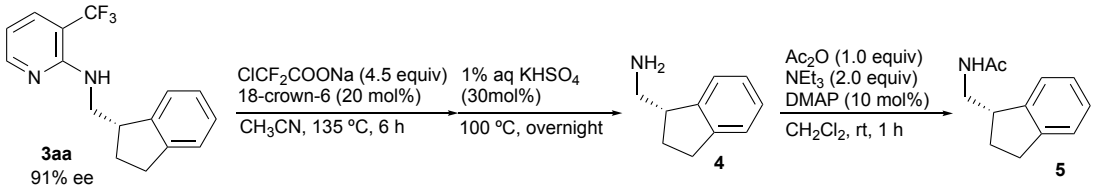
A mixture of $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% Ir), **L1** (8.3 mg, 0.012 mmol, 12 mol%), and $NaBAr^F{}_4$ (18.4 mg, 0.020 mmol, 20 mol%) in toluene (0.2 mL) in a Schlenk tube was stirred at room temperature for 10 min under $N_2$, and then, 2-(*N*-methylamino)pyridine (**1a**, 17.8 mg, 0.10 mmol) and cyclic alkenes **2** (0.12–0.30 mmol) were added to the mixture. The mixture was stirred at 100 ºC for 18 h. After the reaction mixture was concentrated on a rotary evaporator, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (10:1) to give the addition product including a small amount of impurities. The mixture was subjected again to preparative TLC on silica gel eluted with hexane/$CH_2Cl_2$ (2:1) to give the product **3**.

## 7. Unsuccessful substrates for Scheme 3



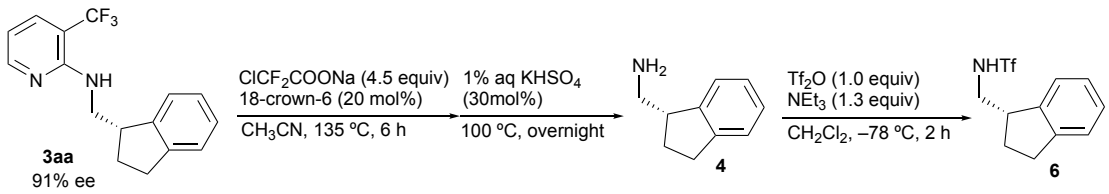

The above alkenes gave no addition product except for **S2e** and **S2q**. **S2e** underwent the addition to give two regioisomers. **S2q** gave the corresponding addition product, but we failed to determine the ee by chiral HPLC analysis.

## 8. Procedure for Scheme 4




A mixture of **3aa** (87.7 mg, 0.30 mmol, 91% ee), $ClCF_2COONa$[10] (68.6 mg, 0.45 mmol, 1.5 equiv), and 18-crown-6 (15.8 mg, 0.060 mmol, 20 mol%) in $CHCN_3$ (1.2 mL, 0.25 M) was stirred at 120 ºC for 3 h. Then, $ClCF_2COONa$ (1.5 equiv) was added to the mixture every 3 h twice during the mixture was heated at 120 °C. After cooling to room temperature, 1% $KHSO_4$ aq (0.7 mL, 30 mol%) was added to the mixture and the resulting mixture was stirred at 100 ºC overnight. The reaction mixture was basified by the addition of 1N NaOH and the aqueous layer was extracted with EtOAc (x3). The combined organic layer was dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue containing amine **4** was used for the following reactions without further purification.

To the mixture of amine **4**, $NEt_3$ (83 µL, 0.60 mmol, 2.0 equiv), and DMAP (3.6 mg, 0.0030 mmol, 10 mol%) in $CH_2Cl_2$ (1.1 mL, 0.27 M) was added dropwise $Ac_2O$ (28 µL, 0.30 mmol, 1.0 equiv) at 0 ºC and the mixture was stirred at rt for 1 h. The reaction was quenched by adding $H_2O$, and the resulting mixture was extracted with $CH_2Cl_2$ (x3). The combined organic layer was dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel eluted with hexane/Acetone (2:1) to give the acetyl amine **5** (colorless solid, 23.0 mg, 40% yield, 91% ee).




**3aa** (42.9 mg, 0.15 mmol) was transformed into **4** according to the same procedure as above. To the mixture of amine **4** and $NEt_3$ (26 μL, 0.18 mmol, 1.3 equiv) in $CH_2Cl_2$ was added dropwise $Tf_2O$ (25 μL, 0.15 mmol, 1.0 equiv) at –78 ºC and the mixture was stirred at –78 ºC for 2 h. The reaction was quenched with $H_2O$, and the resulting mixture was extracted with $CH_2Cl_2$ (x3). The combined organic layer was dried over $Na_2SO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (10:1) to give the trifluoromethyl sulfonyl amine **6** (colorless solid, 8.3 mg, 20% yield, 91% ee). The absolute configuration of trifluromethylsulfonylamine **6** was determined to be *S*-(+) by the comparison of its specific rotation with that of the previously reported one.[11]

## 9. Procedure for Scheme 5a

A mixture of $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% Ir), **L1** (8.3 mg, 0.012 mmol, 12 mol%), and $NaBAr^F_4$ (18.4 mg, 0.020 mmol, 20 mol%) in toluene (0.2 mL) in a Schlenk tube was stirred at room temperature for 10 min under $N_2$, and then, **1a-*d*$_3$** (17.9 mg, 0.10 mmol) and indene **2a** (34.8 mg, 0.30 mmol) were added to the mixture. The reaction mixture was stirred at 100 ºC for 18 h. After the reaction mixture was concentrated on a rotary evaporator, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (10:1) to give **3aa-*d*** (25.8 mg, 87% yield). The deuterium content of **3aa-*d*** was determined by $^1$H NMR. $^1$H NMR ($CDCl_3$) δ 8.28 (d, *J* = 5.0 Hz, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.38–7.10 (m, 4H), 6.62 (dd, *J* = 7.6, 5.0 Hz, 1H), 5.00 (bs, 1H), 3.90–3.76 (m, 0.88H), 3.75–3.60 (m, 0.92H), 3.60–3.48 (m, 0.87H), 3.10–2.80 (m, 1.84H), 2.41–2.25 (m, 1H), 1.95–1.83 (m, 0.78H).

## 10. Procedure for Scheme 5b

A mixture of $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% Ir), **L1** (8.3 mg, 0.012 mmol, 12 mol%), and $NaBAr^F_4$ (18.4 mg, 0.020 mmol, 20 mol%) in toluene (0.2 mL) in a Schlenk tube was stirred at room temperature for 10 min under $N_2$, and then, 2-(*N*-methylamino)pyridine (**1a**, 17.8 mg, 0.10 mmol) and 6-phenylindene (**2r**, 57.6 mg, 0.30 mmol) were added to the mixture. The mixture was stirred at 100 ºC for 18 h. After the reaction mixture was concentrated on a rotary evaporator, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (10:1) to give the addition product including a small amount of impurities. The mixture was subjected again to preparative TLC on silica gel eluted with hexane/$CH_2Cl_2$ (2:1) to give a mixture of **3ar** and **3ar'**. $^1$H NMR ($CDCl_3$) δ 8.29 (d, *J* = 4.8 Hz, 1H, overlapped), 7.66 (d, *J* = 7.2 Hz, 1H, overlapped), 7.63–7.54 (m, 2H, overlapped), 7.53 (s, 0.41H, minor), 7.49 (s, 0.59H, major), 7.48–7.39 (m, 3H, overlapped), 7.39–7.29 (m, 2H, overlapped), 6.63 (dd, *J* = 7.2, 4.8 Hz, 1H, overlapped), 5.06 (bs, 1H, overlapped), 3.96–3.84 (m, 0.41H, minor), 3.78–3.86 (m, 0.59H, major), 3.78–3.64 (m, 1H, overlapped), 3.68–3.54 (m, 1H, overlapped), 3.12–2.86 (m, 2H, overlapped), 2.46–2.32 (m, 1H,

overlapped), 2.02–1.88 (m, 1H, overlapped). HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{22}H_{19}F_3N_2Na_1$ 391.1400; Found 391.1398.

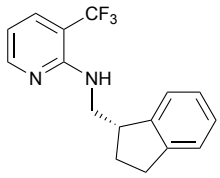
**11. Procedure for Scheme 5c and 5d**

A mixture of $[IrCl(coe)_2]_2$ (2.2 mg, 0.0025 mmol, 5 mol% Ir), **L1** (4.5 mg, 0.0060 mmol, 6 mol%), and $NaBAr^F_4$ (9.2 mg, 0.010 mmol, 10 mol%) in toluene (0.2 mL) in a Schlenk tube was stirred at room temperature for 10 min under $N_2$, and then, indene **2** (0.10 mmol) were added to the mixture. The reaction mixture was stirred at 100 ºC for 30 min. After the reaction mixture was concentrated on a rotary evaporator, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (10:1) to give indene **2** and **2'**. The ratio of **2** to **2'** was determined by $^1$H NMR. Scheme 5c: $^1$H NMR ($CDCl_3$) δ 7.72 (s, 0.67H, **2r**), 7.65–7.59 (m, 2.33H, **2r**+**2r'**), 7.56–7.49 (m, 1H, overlapped), 7.49–7.40 (m, 3H, overlapped), 7.36–7.28 (m, 1H, overlapped), 6.96–6.89 (m, 1H, overlapped), 6.64–6.57 (m, 1H, overlapped), 3.47 (s, 1.33H, **2r**), 3.45 (s, 0.67H, **2r'**). Scheme 5d: $^1$H NMR ($CDCl_3$) δ 7.50–7.19 (m, 7H, overlapped), 7.10–7.04 (m, 0.37H, **2c'**), 6.98–6.89 (m, 0.63H, **2c**), 6.62–6.55 (m, 1H, overlapped), 3.52–3.48 (m, 2H, overlapped), 2.42 (s, 3H, overlapped).

## 12. References

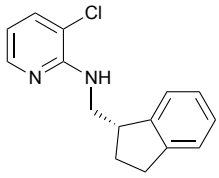

1. R, Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.*, 1985, **23**, 126.
2. M. Brookhart, B. Grant and A. F. Volpe, *Organometallics*, 1992, **11**, 3920.
3. D. Yamauchi, I. Nakamura and T. Nishimura, *Chem. Commun.*, 2021, **57**, 11787.
4. K. Tanaka, H. Hattori, R. Yabe and T. Nishimura, *Chem. Commun.*, 2022, **58**, 5371.
5. M. Umeda, H. Noguchi and T. Nishimura, *Org. Lett*., 2020, **22**, 9597.
6. B.-Y. Wang, D. A. Turner, T. Zujovic, C. M. Hadad and J. D. Badjic, *Chem. Eur. J*., 2011, **17**, 8870.
7. (a) H. Gybäck, J. Malmstöm and T. E. Gitte, *Patent* WO2012/87229 A1, 2012; (b) K. Mori, Y. Ichikawa, M. Kobayashi, S. Shibata, M. Yamanaka and T. Akiyama, *J. Am. Chem. Soc.,* 2013, **135**, 3964.
8. L. Zhao, R. I. Kaiser, W. Lu, M. Ahmed, A. D. Oleinikov, V. N. Azyazov, A. M. Mebel, A. H. Howlader and S. F. WnukePhys. *Chem. Chem. Phys.*, 2020, **22**, 15381.
9. D. Birney, T. K. Lim, J. H. P. Koh, B. R. Pool and J. M. White, *J. Am. Chem. Soc.,* 2002, **124**, 5091.
10. M. Ando, T. Wada and N. Sato, *Org. Lett*., 2006, **8**, 3805.
11. Z. Zhang, J. Wang, J. Li, F. Yang, G. Liu, W. Tang, W. He, J.-J., Fu, Y.-H. Shen, A. Li and W.-D. Zhang, *J. Am. Chem. Soc.*, 2017, **139**, 5558.

## 13. Characterization of the products



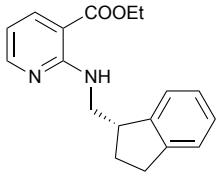
**3aa**

**Compound 3aa** (Scheme 2: colorless solid, 25.8 mg, 88% yield, 91% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 16.6 min (minor), $t_2$ = 18.4 min (major)): $[\alpha]^{25}_D$ –36 (*c* 0.95, $CHCl_3$) for 91% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.27 (d, *J* = 5.0 Hz, 1H), 7.64 (d, *J* = 6.8 Hz, 1H), 7.31–7.23 (m, 2H), 7.23–7.14 (m, 2H), 6.61 (dd, *J* = 6.8, 5.2 Hz, 1H), 4.99 (bs, 1H), 3.85–3.73 (m, 1H), 3.66 (ddd, *J* = 12.6, 7.2, 4.8 Hz, 1H), 3.59–3.48 (m, 1H), 3.00 (ddd, *J* = 14.8, 8.0, 6.8 Hz, 1H), 2.89 (ddd, *J* = 14.8, 8.4, 6.4 Hz, 1H), 2.49–2.26 (m, 1H), 1.96–1.84 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 144.5, 144.3, 134.9 (q, $J_{F-C}$ = 5 Hz), 126.9, 126.2, 124.7, 124.4 (q, $J_{F-C}$ = 270 Hz), 124.1, 111.2, 108.5 (q, $J_{F-C}$ = 32 Hz), 45.3, 44.3, 31.2, 29.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{16}F_3N_2$ 293.1266; Found 293.1258.



**3ba**

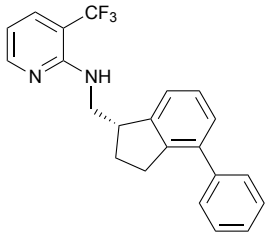
**Compound 3ba** (Scheme 2: colorless oil, 12.1 mg, 47% yield, 88% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 27.2 min (major), $t_2$ = 34.4 min (minor)): $[\alpha]^{25}_D$ –21 (*c* 0.51, $CHCl_3$) for 88% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.04 (dd, *J* = 5.2, 2.0 Hz, 1H), 7.44 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.36–7.20 (m, 2H), 7.26–7.10 (m, 2H), 6.52 (dd, *J* = 7.6, 5.2 Hz, 1H), 5.10 (bs, 1H), 3.82–3.69 (m, 1H), 3.64 (ddd, *J* = 12.8, 6.8, 5.6 Hz, 1H), 3.59–3.47 (m, 1H), 3.08–2.94 (m, 1H), 2.89 (ddd, *J* = 15.4, 8.8, 6.4 Hz, 1H), 2.40–2.26 (m, 1H), 1.98–1.86 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.1, 146.0, 144.7, 144.4, 135.9, 126.9, 126.2, 124.7, 124.1, 115.4, 112.7, 45.3, 44.5, 31.2, 29.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{15}H_{16}Cl_1N_2$ 259.1002; Found 259.0990.



**3ca**

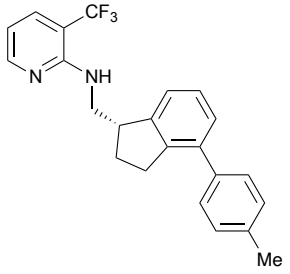
**Compound 3ca** (Scheme 2: colorless oil, 7.0 mg, 24% yield, 87% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 22.1 min (minor), $t_2$ = 23.9 min (major)): $[\alpha]^{25}_D$ –37 (*c* 0.35, $CHCl_3$) for 87% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.28

(dd, $J$ = 5.2, 2.0 Hz, 1H), 8.16 (bs, 1H), 8.13 (dd, $J$ = 7.6, 2.0 Hz, 1H), 7.38–7.30 (m, 1H), 7.24 (d, $J$ = 4.0 Hz, 1H), 7.22–7.13 (m, 2H), 6.52 (dd, $J$ = 7.6, 5.2 Hz, 1H), 4.32 (q, $J$ = 7.2 Hz, 2H), 3.95–3.83 (m, 1H), 3.60 (ddd, $J$ = 12.6, 7.6, 5.2 Hz, 1H), 3.59–3.47 (m, 1H), 3.01 (ddd, $J$ = 16.2, 8.8, 6.0 Hz, 1H), 2.95–2.81 (m, 1H), 2.42–2.28 (m, 1H), 1.98–1.85 (m, 1H), 1.37 (t, $J$ = 7.2 Hz, 3H); $^{13}$C NMR ($CDCl_3$) δ 167.5, 158.8, 153.5, 144.9, 144.3, 140.0, 126.8, 126.2, 124.6, 124.2, 110.8, 106.1, 60.7, 45.2, 44.7, 31.2, 30.3, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{21}N_2O_2$ 297.1603; Found 297.1590.




**3ab**

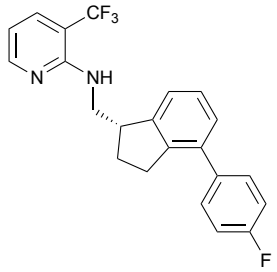
**Compound 3ab** (Scheme 3: colorless oil, 23.9 mg, 65% yield, 88% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 17.8 min (major), $t_2$ = 20.5 min (minor)): $[\alpha]^{25}_D$ –77 (*c* 1.13, $CHCl_3$) for 88% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.30 (d, $J$ = 5.0 Hz, 1H), 7.66 (d, $J$ = 7.2 Hz, 1H), 7.48–7.40 (m, 4H), 7.32–7.38 (m, 1H), 7.31–7.24 (m, 3H), 6.63 (dd, $J$ = 7.2, 5.0 Hz, 1H), 5.05 (bs, 1H), 3.92–3.81 (m, 1H), 3.71 (ddd, $J$ = 12.6, 7.2, 5.2 Hz, 1H), 3.66–3.54 (m, 1H), 3.07 (ddd, $J$ = 16.0, 8.4, 6.4 Hz, 1H), 2.93 (ddd, $J$ = 16.0, 8.8, 6.0 Hz, 1H), 2.38–2.23 (m, 1H), 1.96–1.82 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 145.3, 142.0, 141.2, 138.6, 134.9 (q, $J_{F–C}$ = 5 Hz), 128.5, 128.2, 127.5, 126.9, 124.5 (q, $J_{F–C}$ = 270 Hz), 123.1, 111.3, 108.5 (q, $J_{F–C}$ = 32 Hz), 45.3, 44.6, 31.1, 30.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{20}F_3N_2$ 369.1579; Found 369.1592.




**3ac**

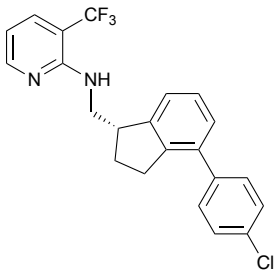
**Compound 3ac** (Scheme 3: colorless oil, 23.4 mg, 61% yield, 87% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 16.0 min (major), $t_2$ = 19.8 min (minor)): $[\alpha]^{25}_D$ –57 (*c* 1.16, $CHCl_3$) for 87% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.28 (d, $J$ = 4.4 Hz, 1H), 7.65 (dd, $J$ = 7.6, 1.2 Hz, 1H), 7.34 (d, $J$ = 7.6 Hz, 2H), 7.30–7.19 (m, 5H), 6.62 (dd, $J$ = 7.6, 4.4 Hz, 1H), 5.04 (bs, 1H), 3.92–3.78 (m, 1H), 3.70 (ddd, $J$ = 12.6, 7.2, 5.2 Hz, 1H), 3.64–3.51 (m, 1H), 3.06 (ddd, $J$ = 16.0, 8.0, 6.6 Hz, 1H), 2.92 (ddd, $J$ = 16.0, 8.8, 6.0 Hz, 1H), 2.40 (s, 3H), 2.36–2.22 (m, 1H), 1.94–1.82 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 145.3, 141.9, 138.6, 138.2, 136.6, 134.9 (q, $J_{F–C}$ = 5 Hz), 128.4, 127.5, 126.9, 125.8, 124.5 (q, $J_{F–C}$ = 269 Hz), 122.9,

111.2, 108.5 (q, $J_{F-C}$ = 31 Hz), 45.3, 44.6, 31.1, 30.2, 21.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}F_3N_2$ 383.1735; Found 383.1724.



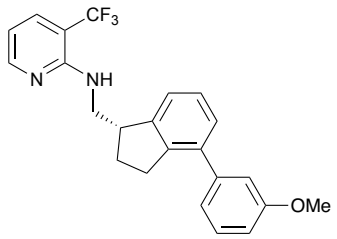

**3ad**

**Compound 3ad** (Scheme 3: colorless oil, 23.1 mg, 60% yield, 90% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 15.8 min (major), $t_2$ = 24.3 min (minor)): $[\alpha]^{25}_D$ –75 (*c* 1.13, $CHCl_3$) for 90% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.28 (d, *J* = 5.0 Hz, 1H), 7.65 (d, *J* = 7.8, 1H), 7.39 (d, *J* = 8.8 Hz, 1H), 7.38 (d, *J* = 8.8 Hz, 1H), 7.27 (d, *J* = 2.4 Hz, 1H), 7.26 (d, *J* = 5.9 Hz, 1H), 7.20 (dd, *J* = 5.9, 2.4 Hz, 1H), 7.11 (d, *J* = 8.8 Hz, 1H), 7.09 (d, *J* = 8.8 Hz, 1H), 6.62 (dd, *J* = 7.8, 5.0 Hz, 1H), 5.02 (bs, 1H), 3.91–3.80 (m, 1H), 3.70 (ddd, *J* = 12.6, 7.4, 5.0 Hz, 1H), 3.63–3.53 (m, 1H), 3.01 (ddd, *J* = 16.0, 8.0, 6.8 Hz, 1H), 2.89 (ddd, *J* = 16.0, 8.8, 6.0 Hz, 1H), 2.37–2.22 (m, 1H), 1.95–1.81 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 162.0 (d, $J_{F-C}$ = 244 Hz), 154.7, 151.7, 145.4, 141.9, 137.6, 137.1 (d, $J_{F-C}$ = 3 Hz), 135.0 (q, $J_{F-C}$ = 5 Hz), 130.0 (d, $J_{F-C}$ = 8 Hz), 127.4, 127.0, 124.5 (q, $J_{F-C}$ = 271 Hz), 123.2, 115.1 (d, $J_{F-C}$ = 21 Hz), 111.3, 108.5 (q, $J_{F-C}$ = 31 Hz), 45.3, 44.6, 31.1, 30.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{19}F_4N_2$ 387.1484; Found 387.1496.



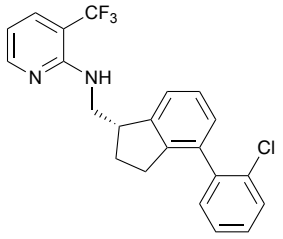

**3ae**

**Compound 3ae** (Scheme 3: colorless oil, 16.0 mg, 40% yield, 88% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 14.7 min (major), $t_2$ = 18.5 min (minor)): $[\alpha]^{25}_D$ –69 (*c* 0.80, $CHCl_3$) for 88% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.29 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.66 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.43–7.34 (m, 4H), 7.32–7.24 (m, 2H), 7.21 (dd, *J* = 6.8, 2.0 Hz, 1H), 6.63 (dd, *J* = 7.8, 5.0 Hz, 1H), 5.02 (bs, 1H), 3.91–3.80 (m, 1H), 3.70 (ddd, *J* = 12.8, 7.2, 5.6 Hz, 1H), 3.64–3.53 (m, 1H), 3.03 (ddd, *J* = 16.0, 8.4, 6.8 Hz, 1H), 2.89 (ddd, *J* = 16.0, 8.4, 5.8 Hz, 1H), 2.38–2.23 (m, 1H), 1.95–1.83 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.2, 145.5, 141.9, 139.6, 137.4, 135.0 (q, $J_{F-C}$ = 5 Hz), 133.0, 129.8, 128.4, 127.3, 127.0, 124.5 (q, $J_{F-C}$ = 270 Hz), 123.4, 111.3, 108.5 (q, $J_{F-C}$ = 31 Hz), 45.3, 44.6, 31.1, 30.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{19}Cl_1F_3N_2$ 403.1189; Found 403.1170.



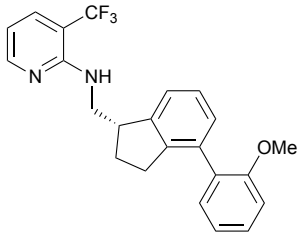

**3af**

**Compound 3af** (Scheme 3: colorless oil, 24.2 mg, 61% yield, 91% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 45.0 min (major), $t_2$ = 56.6 min (minor)): $[\alpha]^{25}_D$ –32 (*c* 1.68, $CHCl_3$) for 91% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.30 (d, *J* = 4.8 Hz, 1H), 7.66 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.35 (t, *J* = 8.0 Hz, 1H), 7.32–7.24 (m, 3H), 7.04 (ddd, *J* = 8.0, 1.4, 1.0 Hz, 1H), 6.99 (dd, *J* = 2.6, 1.4 Hz, 1H), 6.90 (ddd, *J* = 8.0, 2.6, 1.0 Hz, 1H), 6.63 (dd, *J* = 7.6, 4.8 Hz, 1H), 5.05 (bs, 1H), 3.92–3.81 (m, 1H), 3.85 (s, 3H), .3.71 (ddd, *J* = 12.6, 7.2, 5.2 Hz, 1H), 3.64–3.54 (m, 1H), 3.08 (ddd, *J* = 16.0, 8.2, 6.6 Hz, 1H), 2.94 (ddd, *J* = 16.0, 8.4, 5.6 Hz, 1H), 2.38–2.22 (m, 1H), 1.96–1.81 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 159.4, 154.7, 151.7, 145.3, 142.6, 142.0, 138.5, 134.9 (q, $J_{F-C}$ = 5 Hz), 129.2, 127.4, 126.9, 124.5 (q, $J_{F-C}$ = 270 Hz), 123.2, 121.0, 114.2, 112.5, 111.3, 108.5 (q, $J_{F-C}$ = 31 Hz), 55.2, 45.4, 44.6, 31.2, 30.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}F_3N_2O_1$ 399.1684; Found 399.1677.



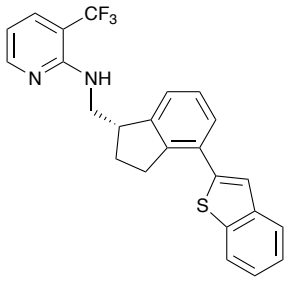

**3ag**

**Compound 3ag** (Scheme 3: colorless oil, 21.3 mg, 53% yield, 87% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 14.5 min (major), $t_2$ = 17.5 min (minor)): $[\alpha]^{25}_D$ –74 (*c* 0.99, $CHCl_3$) for 87% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.28 (d, *J* = 4.0 Hz, 1H), 7.64 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.49–7.42 (m, 1H), 7.35–7.21 (m, 5H), 7.10 (d, *J* = 7.2 Hz, 1H), 6.61 (dd, *J* = 8.0, 5.2 Hz, 1H), 5.02 (bs, 1H), 3.90–3.79 (m, 1H), 3.69 (ddd, *J* = 12.4, 7.0, 5.4 Hz, 1H), 3.65–3.55 (m, 1H), 3.08–2.49 (m, 2H), 2.40–2.18 (m, 1H), 1.96–1.78 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 153.7, 151.7, 144.7, 143.2, 140.0, 136.4, 134.9 (q, $J_{F-C}$ = 5 Hz), 133.0, 130.9, 129.5, 128.6, 127.9, 126.5, 126.3, 124.5 (q, $J_{F-C}$ = 270 Hz), 123.1, 111.3, 108.5 (q, $J_{F-C}$ = 31 Hz), 45.4, 44.7, 30.4, 29.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{19}{}^{35}Cl_1F_3N_2$ 403.1189; Found 403.1182.



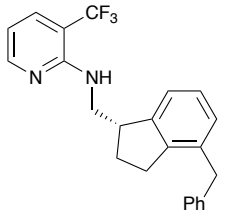

**3ah**

**Compound 3ah** (Scheme 3: colorless oil, 33.6 mg, 84% yield, 90% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 19.1 min (major), $t_2$ = 22.0 min (minor)): $[\alpha]^{25}_D$ –32 (*c* 1.68, $CHCl_3$) for 90% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.29 (d, *J* = 5.0 Hz, 1H), 7.66 (dd, *J* = 7.0, 1.2 Hz, 1H), 7.34 (td, *J* = 8.0, 1.6 Hz, 1H), 7.27 (t, *J* = 7.6 Hz, 1H), 7.26 (d, *J* = 7.2 Hz, 1H), 7.22 (d, *J* = 7.8, 1.8 Hz, 1H), 7.18 (dd, *J* = 7.4, 1.4 Hz, 1H), 7.02 (td, *J* = 7.6, 1.2 Hz, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 6.62 (dd, *J* = 7.0, 5.0 Hz, 1H), 5.06 (bs, 1H), 3.90–3.80 (m, 1H), 3.79 (s, 3H), 3.71 (ddd, *J* = 12.8, 7.2, 5.4 Hz, 1H), 3.64–3.54 (m, 1H), 2.85 (ddd, *J* = 16.0, 8.2, 6.6 Hz, 1H), 2.73 (ddd, *J* = 16.0, 8.0, 6.0 Hz, 1H), 2.35–2.21 (m, 1H), 1.91–1.78 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 156.4, 154.7, 151.7, 144.4, 143.6, 135.6, 134.9 (q, $J_{F-C}$ = 5 Hz), 131.0, 130.0, 128.6, 128.4, 126.2, 124.5 (q, $J_{F-C}$ = 270 Hz), 123.0, 120.4, 111.2. 110.7, 108.5 (q, $J_{F-C}$ = 32 Hz), 55.3, 45.4, 44.6, 30.6, 29.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}F_3N_2O_1$ 399.1684; Found 399.1698.



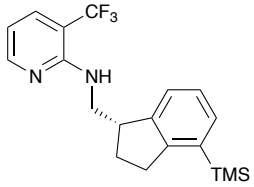

**3ai**

**Compound 3ai** (Scheme 3: colorless solid, 14.0 mg, 33% yield, 86% ee). The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, $t_1$ = 14.6 min (minor), $t_2$ = 15.4 min (major)): $[\alpha]^{25}_D$ –32 (*c* 1.68, $CHCl_3$) for 86% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.30 (d, *J* = 4.8 Hz, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 1H), 7.67 (d, *J* = 6.8 Hz, 1H), 7.52 (dd, *J* = 6.4, 2.4 Hz, 1H), 7.42 (s, 1H), 7.41–7.23 (m, 4H), 6.64 (dd, *J* 6.8, 4.8 Hz, 1H), 5.04 (bs, 1H), 3.89–3.79 (m, 1H), 3.72 (ddd, *J* = 12.8, 7.4, 5.6 Hz, 1H), 3.67–3.56 (m, 1H), 3.29 (ddd, *J* = 15.6, 8.4, 6.8 Hz, 1H), 3.17 (ddd, *J* = 15.6, 8.8, 5.6 Hz, 1H), 2.43–2.31 (m, 1H), 2.03–1.91 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 156.6, 151.7, 146.0, 143.4, 141.9, 140.4, 139.6, 135.0 (q, $J_{F-C}$ = 5 Hz), 131.3, 127.4, 127.1, 124.5 (q, $J_{F-C}$ = 270 Hz), 124.4, 124.2, 124.0, 123.5, 122.0, 121.9, 111.4, 108.5 (q, $J_{F-C}$ = 31 Hz), 45.4, 44.5, 32.0, 29.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{20}F_3S_1$ 425.1299; Found 425.1288.



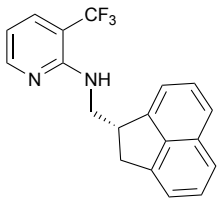

**3aj**

**Compound 3aj** (Scheme 3: colorless oil, 18.7 mg, 49% yield, 87% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 28.1 min (major), $t_2$ = 36.6 min (minor)): $[\alpha]^{25}_D$ –42 (*c* 0.78, $CHCl_3$) for 87% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.28 (dd, *J* = 5.2, 1.2 Hz, 1H), 7.65 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.33–7.23 (m, 2H), 7.25–7.11 (m, 5H), 7.01 (dd, *J* = 6.8, 2.0 Hz, 1H), 6.62 (dd, *J* = 7.6, 5.2 Hz, 1H), 4.98 (bs, 1H), 3.97 (s, 2H), 3.84–3.73 (m, 1H), 3.65 (ddd, *J* = 12.8, 6.8, 5.2 Hz, 1H), 3.60–3.49 (m, 1H), 2.89 (ddd, *J* = 15.6, 8.8, 6.8 Hz, 1H), 2.78 (ddd, *J* = 15.6, 8.8, 6.0 Hz, 1H), 2.38–2.22 (m, 1H), 1.95–1.81 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 144.7, 143.2, 140.3, 137.0, 134.9 (q, $J_{F-C}$ = 5 Hz), 128.8, 128.4, 127.8, 126.7, 125.9, 124.4 (q, $J_{F-C}$ = 270 Hz), 122.1, 111.2, 108.5 (q, $J_{F-C}$ = 31 Hz), 45.4, 44.5, 39.6, 29.8, 29.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{22}F_3N_2$ 383.1735; Found 383.1736.




**3ak**

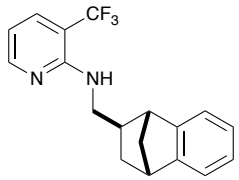
**Compound 3ak** (Scheme 3: colorless solid, 19.1 mg, 52% yield, 86% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane only, flow 0.5 mL/min, 254 nm, $t_1$ = 18.9 min (major), $t_2$ = 22.7 min (minor)): $[\alpha]^{25}_D$ –48 (*c* 0.97, $CHCl_3$) for 86% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.28 (d, *J* = 4.8 Hz, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.29 (d, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 6.61 (dd, *J* = 7.6, 4.8 Hz, 1H), 4.99 (bs, 1H), 3.83–3.74 (m, 1H), 3.65 (ddd, *J* = 12.6, 7.2, 5.6 Hz, 1H), 3.55–3.45 (m, 1H), 3.12–3.00 (m, 1H), 3.01–2.90 (m, 1H), 2.30–2.25 (m, 1H), 2.00–1.82 (m, 1H), 0.31 (s, 9H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 149.6, 143.6, 135.8, 135.0 (q, $J_{F-C}$ = 5 Hz), 132.6, 125.7, 125.0, 124.5 (q, $J_{F-C}$ = 270 Hz), 111.2, 108.4 (q, $J_{F-C}$ = 31 Hz), 45.5, 43.8, 32.3, 29.7, –0.48. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{24}F_3N_2Si$ 365.1653; Found 365.1661.




**3al**

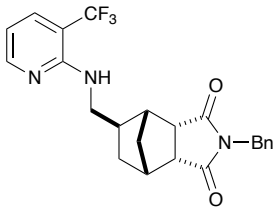
**Compound 3al** (Scheme 3: colorless solid, 28.0 mg, 85% yield, 88% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, $t_1$ = 10.2 min

(minor), $t_2$ = 11.3 min (major)): $[\alpha]^{25}_D$ –42 (*c* 0.74, $CHCl_3$) for 88% ee (*S*). $^1$H NMR ($CDCl_3$) δ 8.30 (d, *J* = 4.8 Hz, 1H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.52–7.42 (m, 2H), 7.36 (d, *J* = 7.2 Hz, 1H), 7.29 (d, *J* = 6.8 Hz, 1H), 6.64 (dd, *J* = 8.0, 4.8 Hz, 1H), 5.15 (bs, 1H), 4.10 (qd, *J* = 7.2, 2.8 Hz, 1H), 3.90 (ddd, *J* = 12.8, 7.2, 5.6 Hz, 1H), 3.77 (ddd, *J* = 13.2, 7.2, 5.6 Hz, 1H), 3.63 (dd, *J* = 17.6, 8.0 Hz, 1H), 3.18 (dd, *J* = 17.6, 3.2 Hz, 1H); $^{13}$C NMR ($CDCl_3$) δ 154.5, 151.7, 146.5, 143.9, 138.8, 135.0 (q, $J_{F-C}$ = 5 Hz), 131.6, 128.0, 127.8, 124.5 (q, $J_{F-C}$ = 270 Hz), 123.3, 122.5, 119.5, 119.4, 111.4, 108.9 (q, $J_{F-C}$ = 31 Hz), 46.5, 42.8, 35.7. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{16}F_3N_2$ 329.1263; Found 329.1266.




**3am**

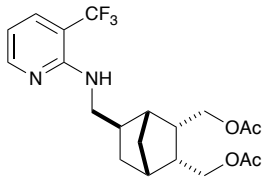
**Compound 3am** (Scheme 3: colorless oil, 23.8 mg, 75% yield, 42% ee). The ee was measured by HPLC (Chiralcel OJ-H-OJ-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 40.4 min (major), $t_2$ = 42.6 min (minor)): $[\alpha]^{25}_D$ +28 (*c* 1.12, $CHCl_3$) for 42% ee. $^1$H NMR ($CDCl_3$) δ 8.26 (d, *J* = 4.8 Hz, 1H), 7.65 (d, *J* = 7.4, 1.0 Hz, 1H), 7.16 (d, *J* = 5.2 Hz, 1H), 7.14 (d, *J* = 5.2 Hz, 1H), 7.07 (d, *J* = 5.2 Hz, 1H), 7.05 (d, *J* = 5.2 Hz, 1H), 6.61 (dd, *J* = 7.4, 4.8 Hz, 1H), 4.97 (bs, 1H), 3.71–3.54 (m, 2H), 3.36 (s, 1H), 3.21 (s, 1H), 1.92–1.74 (m, 3H), 1.58–1.50 (m, 2H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 148.2, 148.2, 135.0 (q, $J_{F-C}$ = 5 Hz), 125.6, 125.6, 124.5 (q, $J_{F-C}$ = 270 Hz), 120.7, 120.4, 111.2, 108.5 (q, $J_{F-C}$ = 31 Hz), 46.5, 46.4, 46.0, 43.8, 40.8, 33.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}F_3N_2$ 319.1422; Found 319.1409.




**3an**

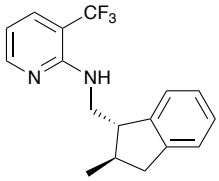
**Compound 3an** (Scheme 3: colorless oil, 31.8 mg, 74% yield, 89% ee). Hexane was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, $t_1$ = 28.4 min (minor), $t_2$ = 31.2 min (major)): $[\alpha]^{25}_D$ +37 (*c* 1.54, $CHCl_3$) for 89% ee. $^1$H NMR ($CDCl_3$) δ 8.20 (d, *J* = 4.8 Hz, 1H), 7.62 (d, *J* = 7.4 Hz, 1H), 7.45–7.36 (m, 2H), 7.29–7.20 (m, 3H), 6.60 (dd, *J* = 7.4, 4.8 Hz, 1H), 4.61 (s, 2H), 4.53 (bs, 1H), 3.31 (ddd, *J* = 12.8, 6.8, 5.2 Hz, 1H), 3.16 (ddd, *J* = 12.8, 8.0, 5.2 Hz, 1H), 3.13–3.01 (m, 2H), 2.74 (d, *J* = 4.2 Hz, 1H), 2.60 (d, *J* = 4.2 Hz, 1H), 1.82–1.69 (m, 1H), 1.57–1.39 (m, 2H), 1.27–1.04 (m, 2H); $^{13}$C NMR ($CDCl_3$) δ 177.6, 177.5, 154.4, 151.5, 136.1, 134.9 (q, $J_{F-C}$ = 5 Hz), 129.0, 128.4, 128.1, 124.3 (q, $J_{F-C}$ = 271 Hz), 111.4, 108.5 (q, $J_{F-C}$ = 31 Hz), 48.3, 47.9, 45.8, 42.4, 42.1, 39.6,

39.4, 36.6, 30.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{23}F_3N_3O_2$ 430.1742; Found 430.1736.



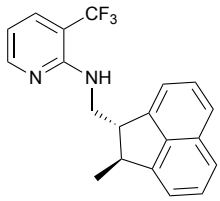

**3ao**

**Compound 3ao** (Scheme 3: colorless oil, 34.9 mg, 84% yield, 81% ee). Hexane was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, $t_1$ = 13.1 min (minor), $t_2$ = 14.0 min (major)): $[\alpha]^{25}_D$ –5 (*c* 1.75, $CHCl_3$) for 81% ee. $^1$H NMR ($CDCl_3$) δ 8.24 (d, *J* = 5.2 Hz, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 6.59 (dd, *J* = 7.6, 5.2 Hz, 1H), 4.90 (bs, 1H), 4.18 (dd, *J* =10.6, 5.8 Hz, 1H), 4.10 (t, *J* = 10.6 Hz, 1H), 4.06 (s, 1H), 4.04 (s, 1H), 3.39–3.20 (m, 2H), 2.38–2.28 (m, 3H), 2.25–2.20 (m, 1H), 2.12–2.05 (m, 1H), 2.03 (s, 3H), 1.98 (s, 3H), 1.73 (ddd, *J* = 13.2, 8.8, 2.4 Hz, 1H), 1.58 (d, *J* = 10.4 Hz, 1H), 1.34 (d, *J* = 10.4 Hz, 1H), 1.03 (dt, *J* = 13.2, 4.4 Hz, 1H); $^{13}$C NMR ($CDCl_3$) δ 171.0, 170.9, 154.5, 151.7, 134.8 (q, $J_{F-C}$ = 5 Hz), 124.5 (q, $J_{F-C}$ = 273 Hz), 111.1, 108.2 (q, $J_{F-C}$ = 32 Hz), 62.5, 61.4, 45.9, 41.9, 39.7, 39.6, 38.3, 36.3, 33.3, 28.7, 21.0, 20.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{26}F_3N_2O_4$ 415.1845; Found 415.1835.



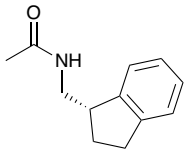

**3ap**

**Compound 3ap** (Scheme 5: colorless oil, 21.2 mg, 70% yield, 81% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 12.4 min (minor), $t_2$ = 13.2 min (major)): $[\alpha]^{25}_D$ –42 (*c* 0.13, $CHCl_3$) for 81% ee. $^1$H NMR ($CDCl_3$) δ 8.28 (d, *J* = 4.4 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.30–7.12 (m, 4H), 6.61 (dd, *J* = 7.8, 4.4 Hz, 1H), 4.95 (bs, 1H), 3.84–3.70 (m, 2H), 3.15 (dd, *J* = 15.6, 8.0 Hz, 1H), 3.08 (q, *J* = 6.0 Hz, 1H), 2.54 (dd, *J* = 15.6, 6.2 Hz, 1H), 2.34 (sept, *J* = 6.8 Hz, 1H), 1.78 (d, *J* = 6.8 Hz, 3H); $^{13}$C NMR ($CDCl_3$) δ 154.7, 151.7, 143.9, 143.4, 134.9 (q, $J_{F-C}$ = 5 Hz), 127.0, 126.3, 124.8, 124.4 (q, $J_{F-C}$ = 270 Hz), 124.1, 111.2, 108.5 (q, $J_{F-C}$ = 34 Hz), 52.1, 44.2, 39.9, 38.2, 20.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{17}H_{18}F_3N_2$ 307.1422; Found 307.1414.



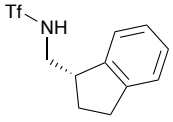
**3aq**

**Compound 3aq** (Scheme 5: colorless oil, 21.2 mg, 62% yield, 84% ee). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, $t_1$ = 21.9 min (minor), $t_2$ = 27.1 min (major)): $[\alpha]^{25}_D$ –49 (*c* 0.84, $CHCl_3$) for 84% ee. $^1$H NMR ($CDCl_3$) δ 8.30 (d, *J* = 4.8 Hz, 1H), 7.72–7.61 (m, 3H), 7.54–7.45 (m, 2H), 7.36 (d, *J* = 6.8 Hz, 1H), 7.27 (d, *J* = 6.8 Hz, 1H), 6.64 (dd, *J* = 7.2, 4.8 Hz, 1H), 5.15 (bs, 1H), 3.91 (t, *J* = 6.4 Hz, 2H), 3.63 (td, *J* = 6.4, 3.2 Hz, 1H), 3.51 (qd, *J* = 7.2, 3.2 Hz, 1H), 1.48 (d, *J* = 7.4 Hz, 3H); $^{13}$C NMR ($CDCl_3$) δ 154.6, 151.7, 149.0, 145.1, 137.7, 135.0 (q, $J_{F-C}$ = 5 Hz), 131.5, 128.1, 127.9, 124.4 (q, $J_{F-C}$ = 270 Hz), 123.4, 122.8, 119.4, 118.7, 111.4, 108.5 (q, $J_{F-C}$ = 31 Hz), 52.2, 45.7, 43.7, 21.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{18}F_3N_2$ 343.1422; Found 343.1409.



**5**

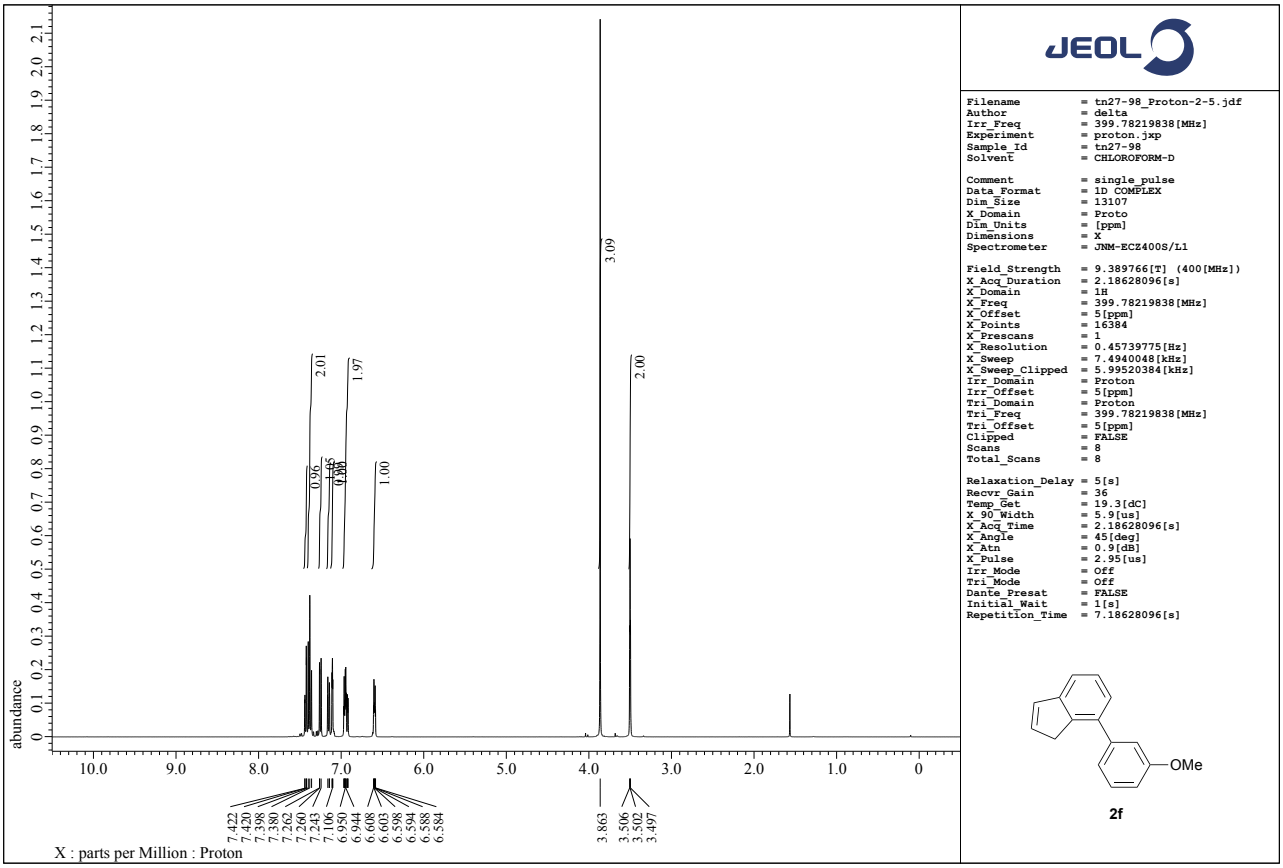
**Compound 5** (Scheme 4: colorless solid, 23.0 mg, 40% yield, 91% ee). The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$/EtOH = 8:2:1, flow 0.5 mL/min, 254 nm, $t_1$ = 9.6 min (major), $t_2$ = 10.0 min (minor)): $[\alpha]^{25}_D$ +14 (*c* 0.67, $CHCl_3$) for 91% ee (*S*). $^1$H NMR ($CDCl_3$) δ 7.28–7.15 (m, 4H), 5.52 (bs, 1H), 3.70–3.57 (m, 1H), 3.46–3.31 (m, 2H), 3.01–2.80 (m, 2H), 2.32–2.20 (m, 1H), 1.97 (s, 3H), 1.87–1.76 (m, 1H); $^{13}$C NMR ($CDCl_3$) δ 170.3, 144.5, 143.9, 127.0, 126.3, 124.8, 123.7, 44.6, 42.8, 31.2, 29.5, 23.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{16}NO$ 190.1240; Found 190.1232.

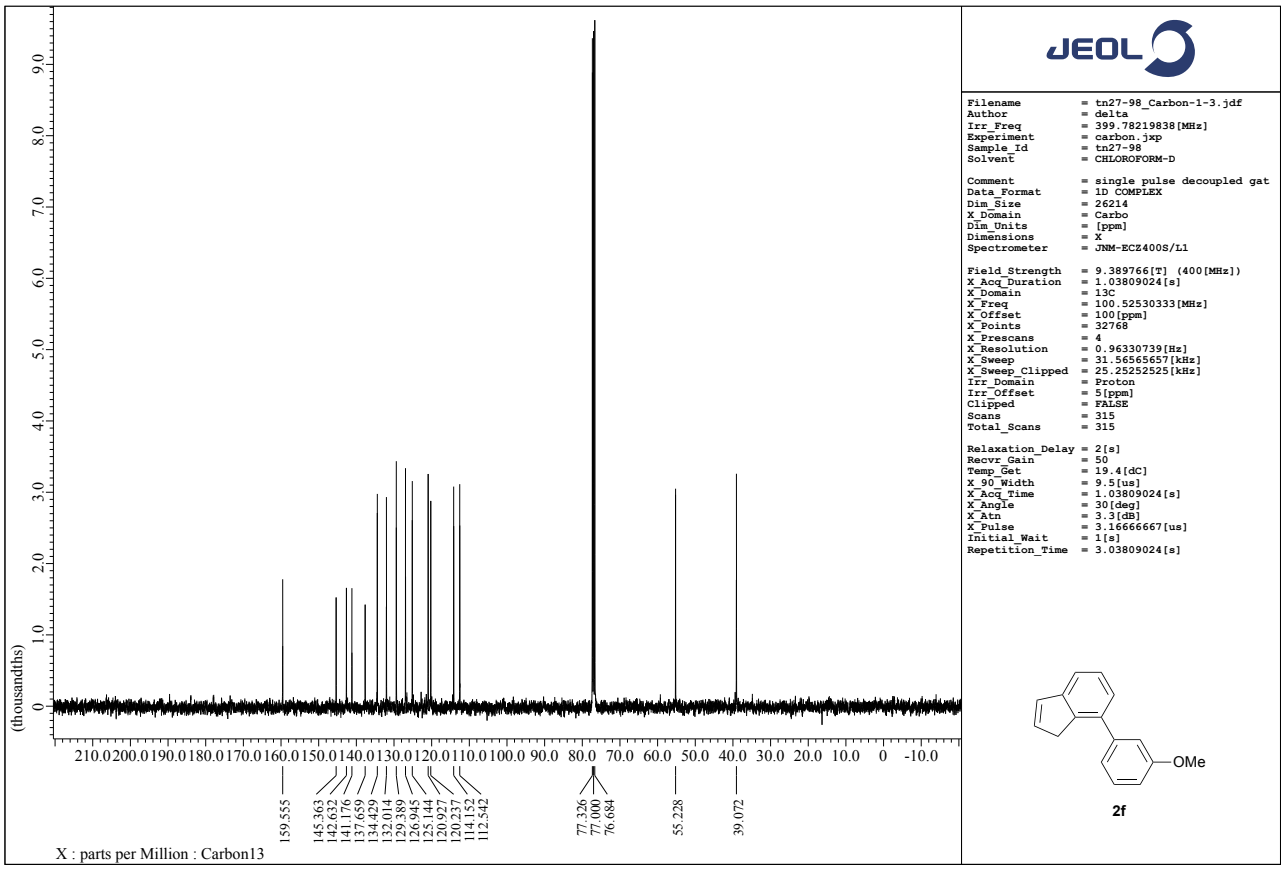


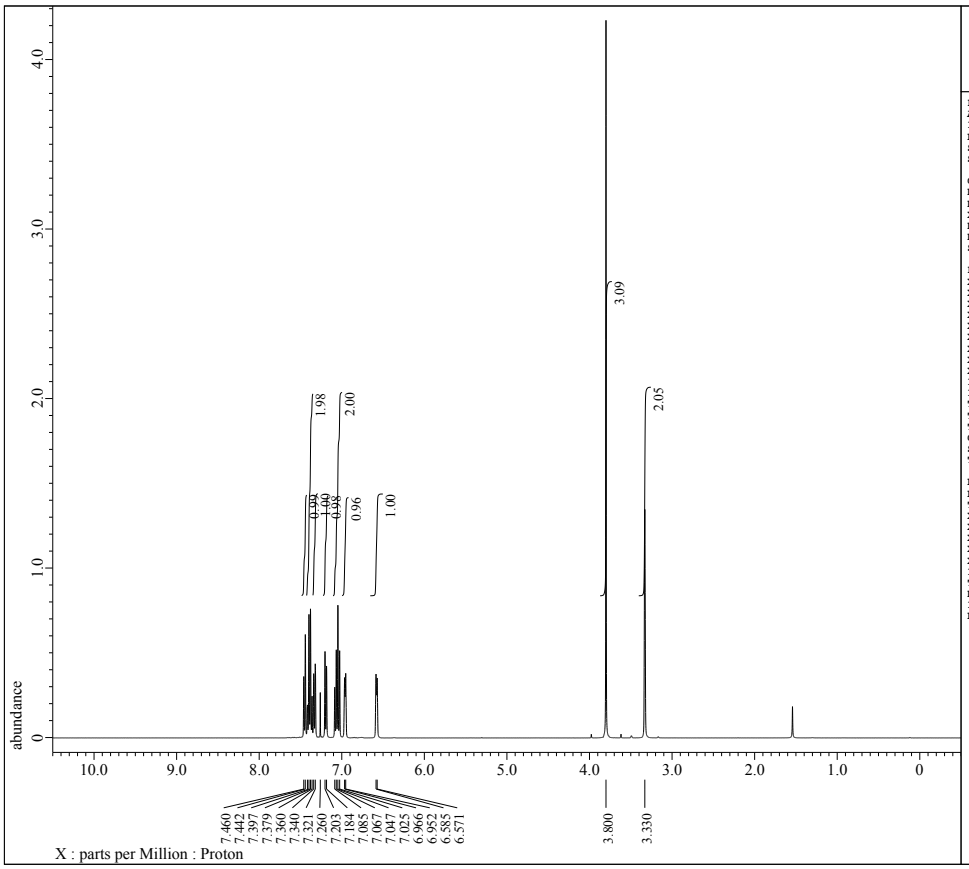
**6**

**Compound 6** (Scheme 4: colorless solid, 8.3 mg, 20% yield, 91% ee, CAS: 2088274-15-9 for (*R*)-**6**). The ee was measured by HPLC (Chiralcel OJ-H, hexane/2-propanol = 18:1, flow 0.5 mL/min, 254 nm, $t_1$ = 35.2 min (minor), $t_2$ = 44.5 min (major)): $[\alpha]^{25}_D$ +5 (*c* 0.26, $CHCl_3$) for 91% ee (*S*). $^1$H NMR ($CDCl_3$) δ 7.28–7.12 (m, 4H), 6.25 (s, 1H), 3.72–3.62 (m, 1H), 3.60–3.49 (m, 1H), 3.53–3.39 (m, 1H), 3.09–2.80 (m, 2H), 2.39–2.23 (m, 1H), 1.90–1.78 (m, 1H).

## 14. $^{1}H$, $^{13}C$ NMR spectra and chiral HPLC charts



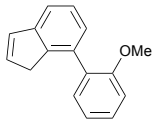



Filename = tn28-8-3_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn28-8-3
Solvent = CHLOROFORM-D

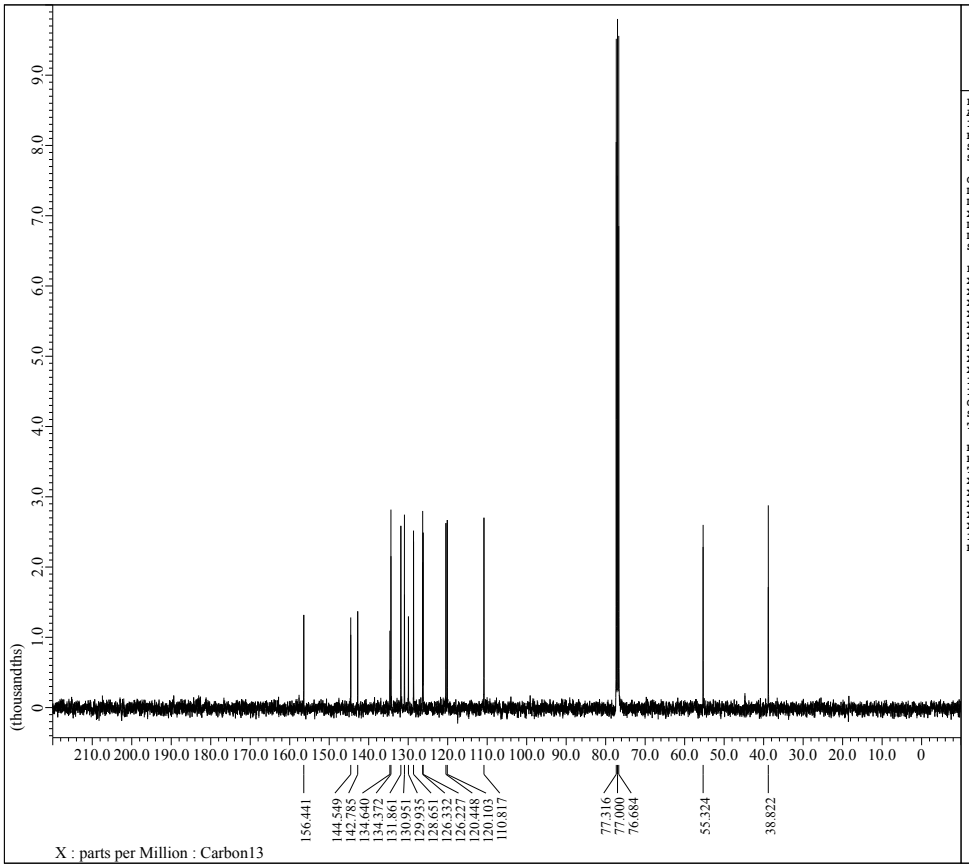
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 18.3[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]




**2h**

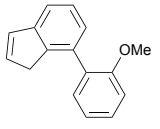



Filename = tn28-8-3_Carbon-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn28-8-3
Solvent = CHLOROFORM-D

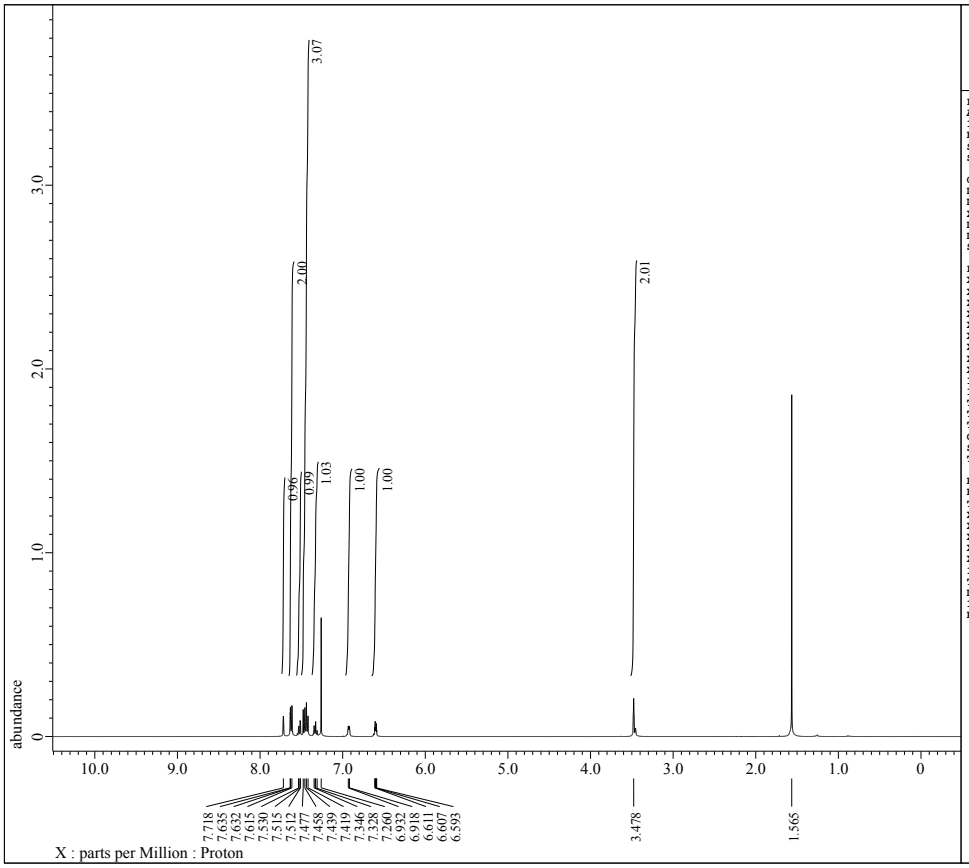
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 256
Total_Scans = 256

Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 18.3[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]

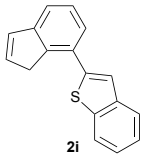



**2h**

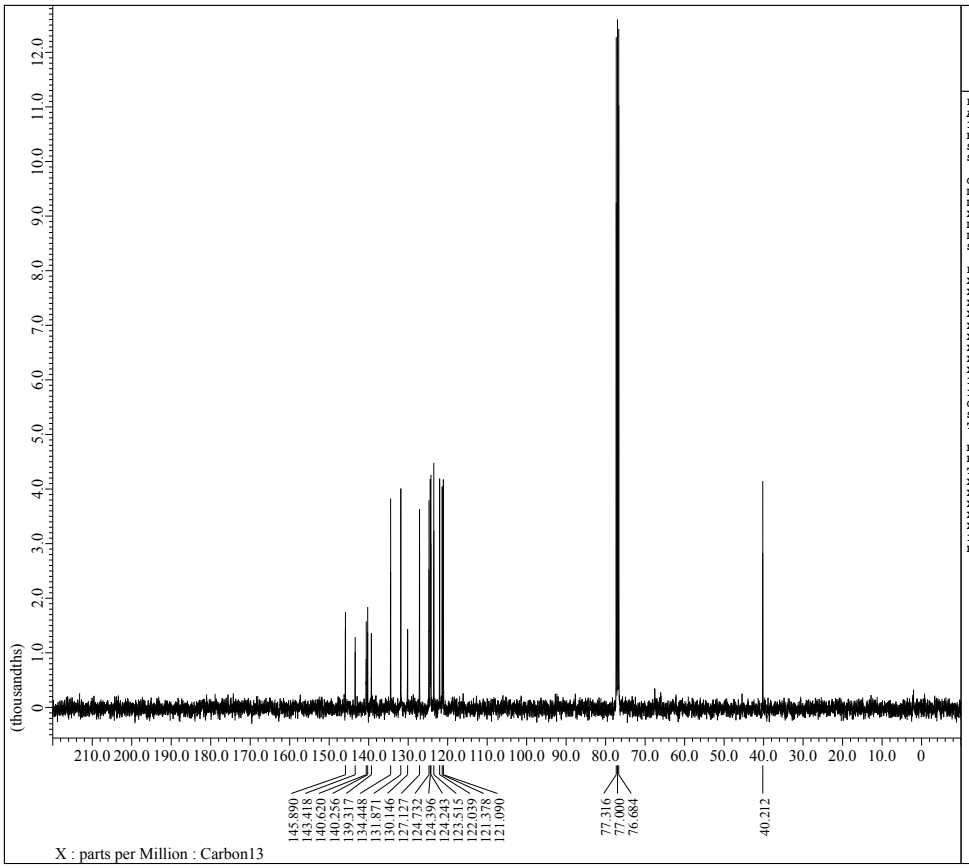



JEOL

```
Filename          = sk#15-023-1_Proton-2-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = sk#15-023-1
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        = 56
Temp_Get          = 22.2[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]
```

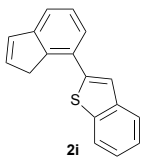



2i

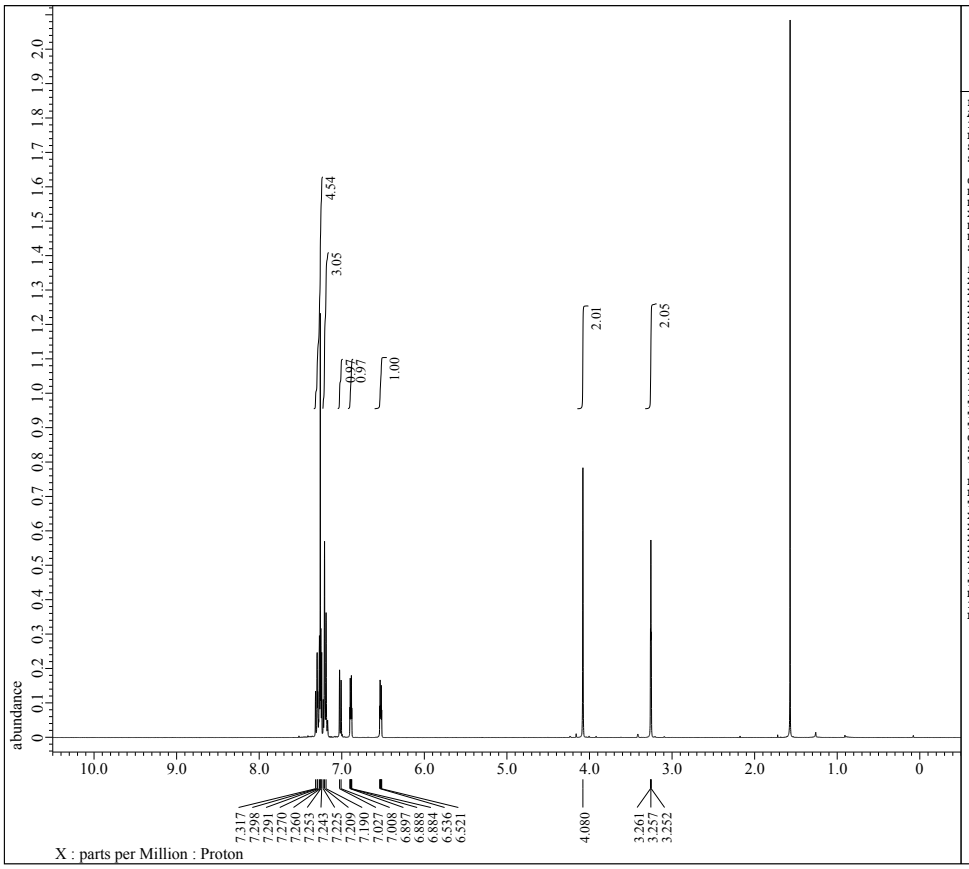



JEOL

```
Filename          = sk#thiophenyl_Carbon-1-3.j
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = sk#thiophenyl
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             = 121
Total_Scans       = 121

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 20.9[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]
```

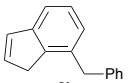



2i

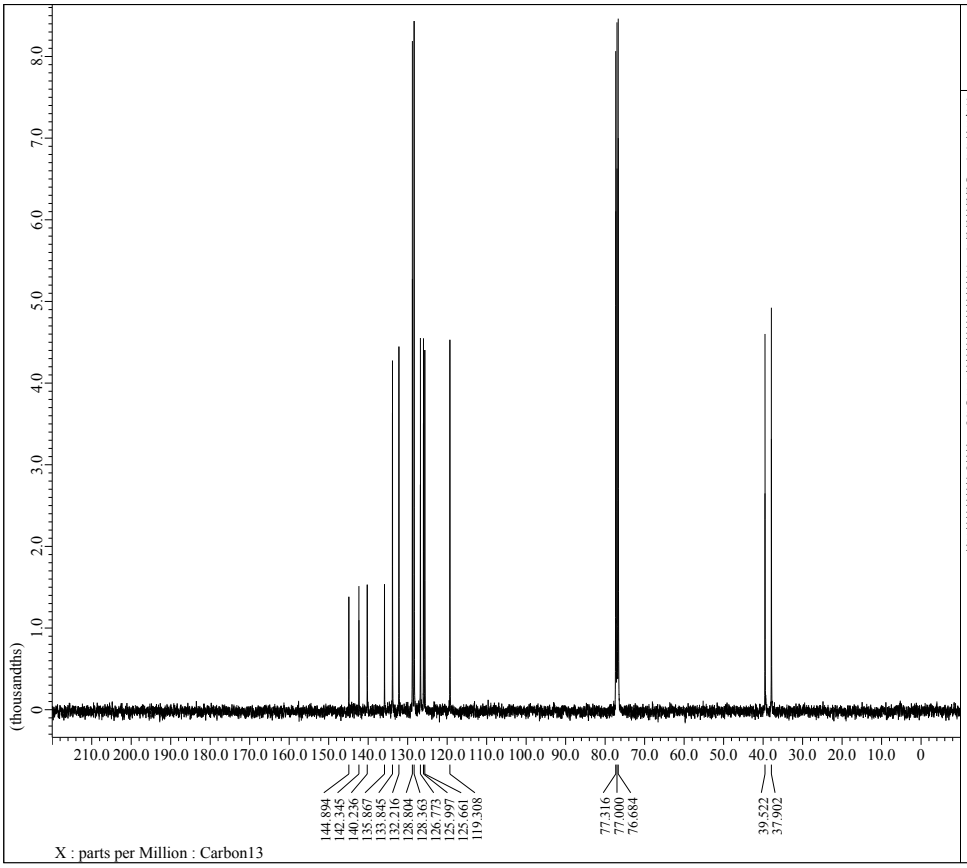



JEOL

```
Filename          = tn30-47_Proton-2-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn30-47
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          = 21.3[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]
```

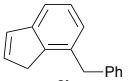



**2j**

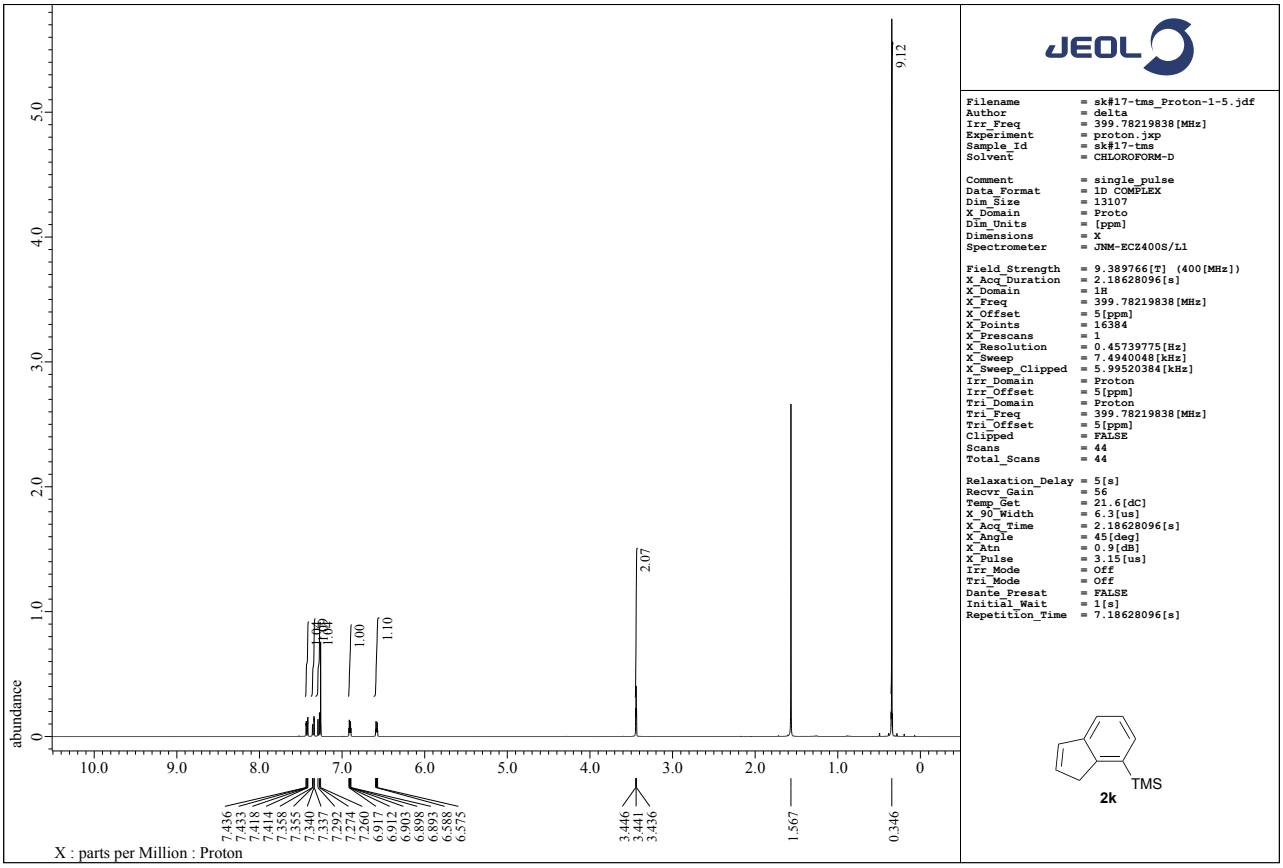


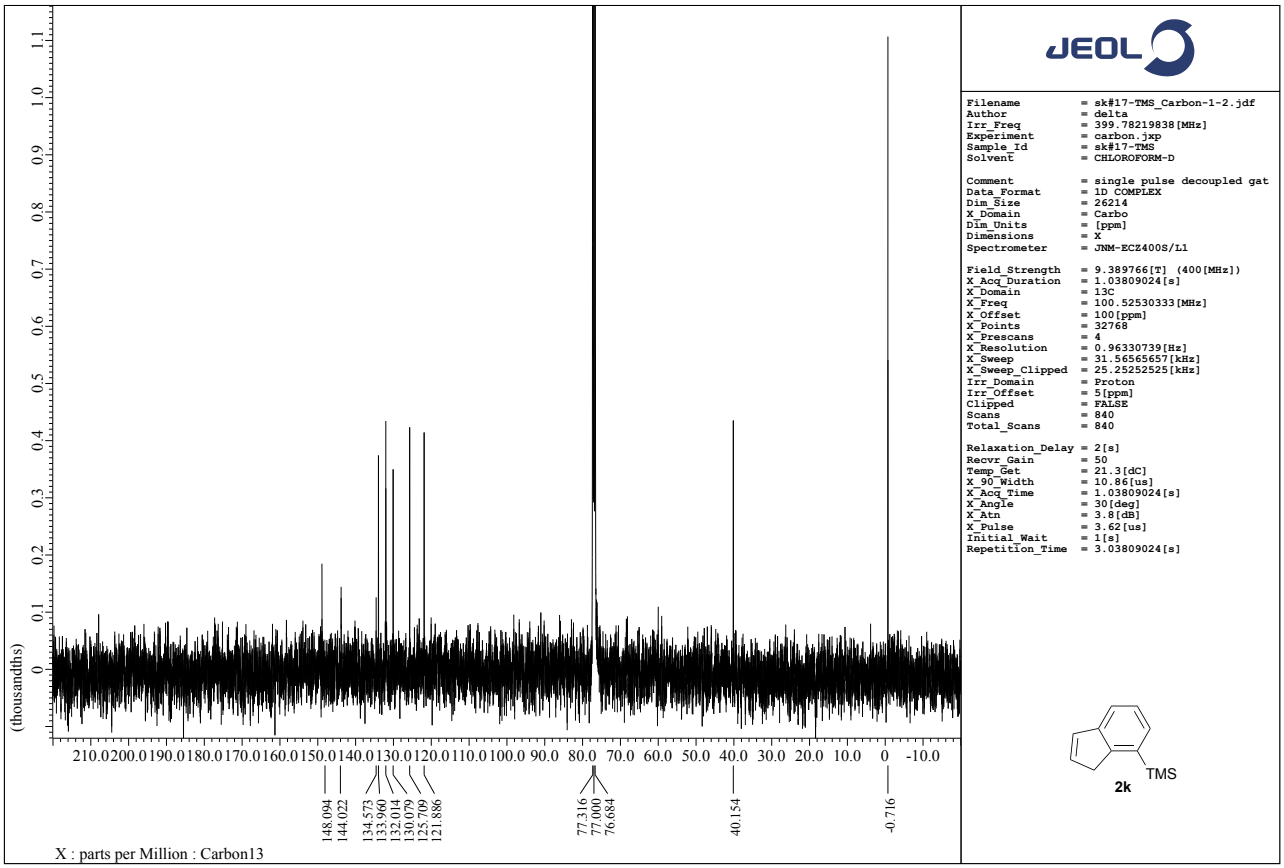

JEOL

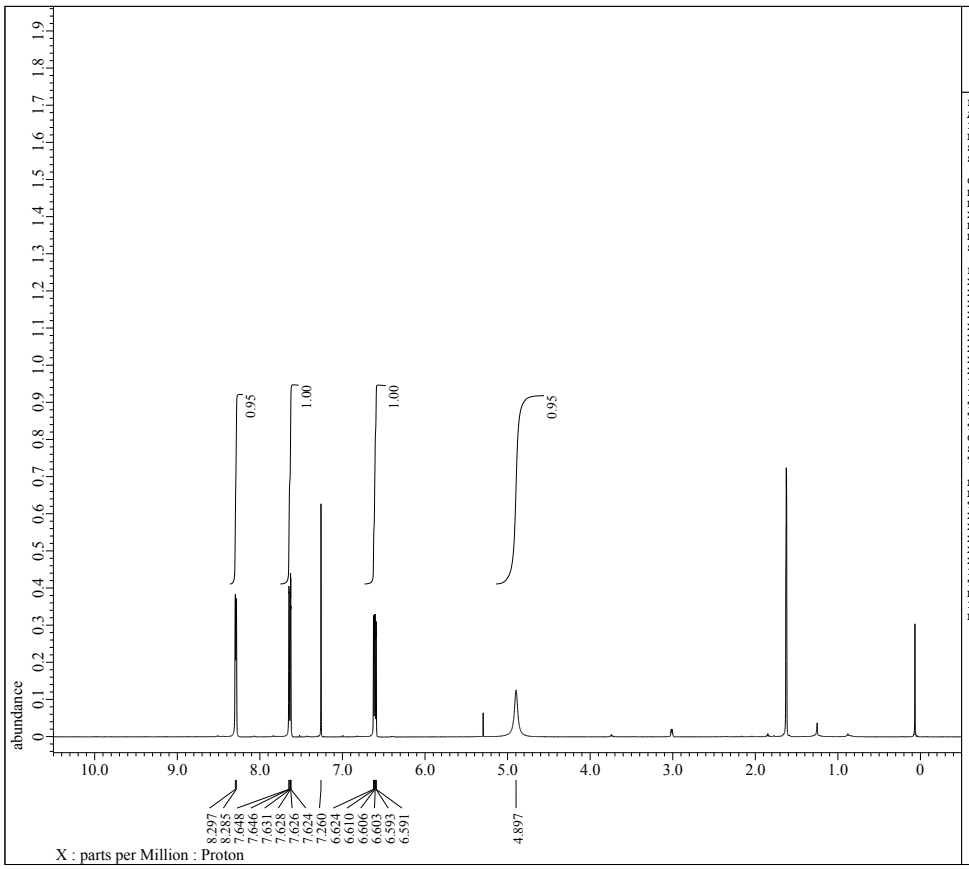
```
Filename          = tn30-47_Carbon-1-3.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn30-47
Solvent           = CHLOROFORM-D

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             = 534
Total_Scans       = 534

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.2[dC]
X_90_Width        = 10.55[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.51666667[us]
Initial_Wait      = 1[s]
RepetitIon_Time   = 3.03809024[s]
```




**2j**

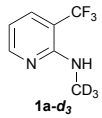

abundance
X : parts per Million : Proton
9.12
2.07
1.00
1.00
1.10
7.436
7.433
7.418
7.414
7.358
7.356
7.340
7.337
7.292
7.274
7.260
6.917
6.912
6.903
6.898
6.893
6.588
6.575
3.446
3.441
3.436
1.567
0.346
Filename = sk#17-tms_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = sk#17-tms
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 = 44
Total_Scans = 44
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.6[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
TMS
2k


(thousandths)
X : parts per Million : Carbon13
148.094
144.022
134.573
133.960
132.014
130.079
125.709
121.886
77.316
77.000
76.684
40.154
-0.716
Filename = sk#17-TMS_Carbon-1-2.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = sk#17-TMS
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 = 840
Total_Scans = 840
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.3[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]
TMS
2k

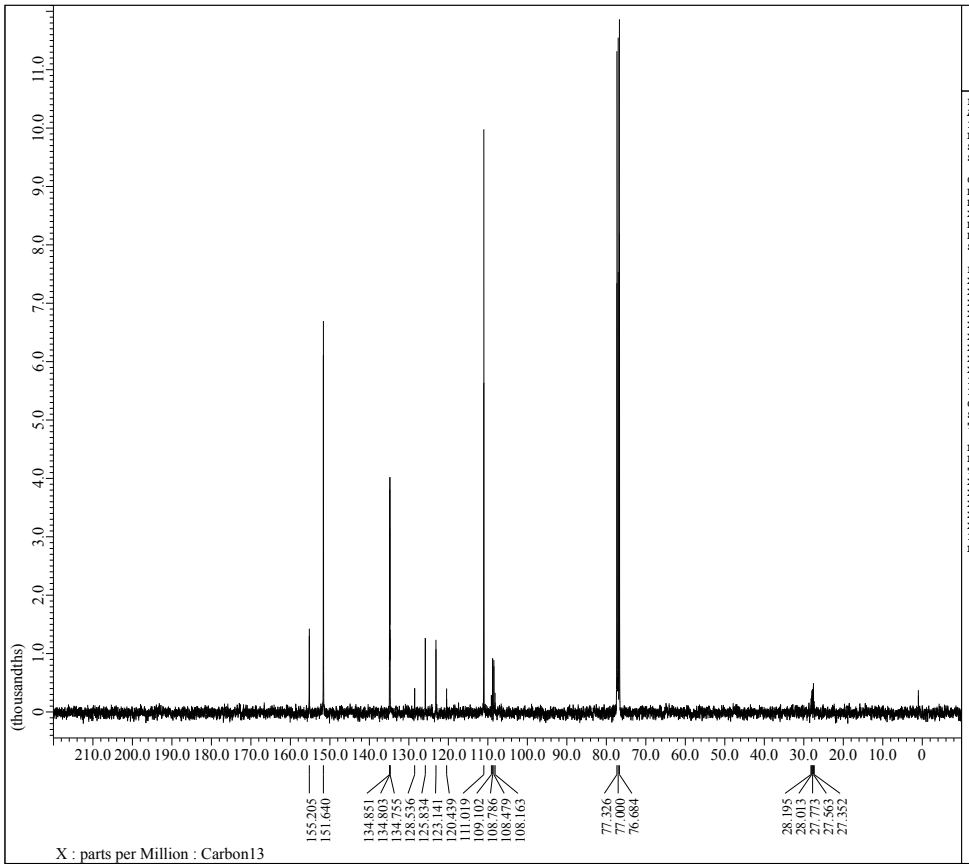



JEOL

```
Filename          = sk#17-081-H_Proton-1-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = sk#17-081-H
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[dC]
X_90_Width        = 6.3[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.15[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```

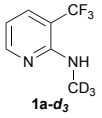



**1a-$d_3$**

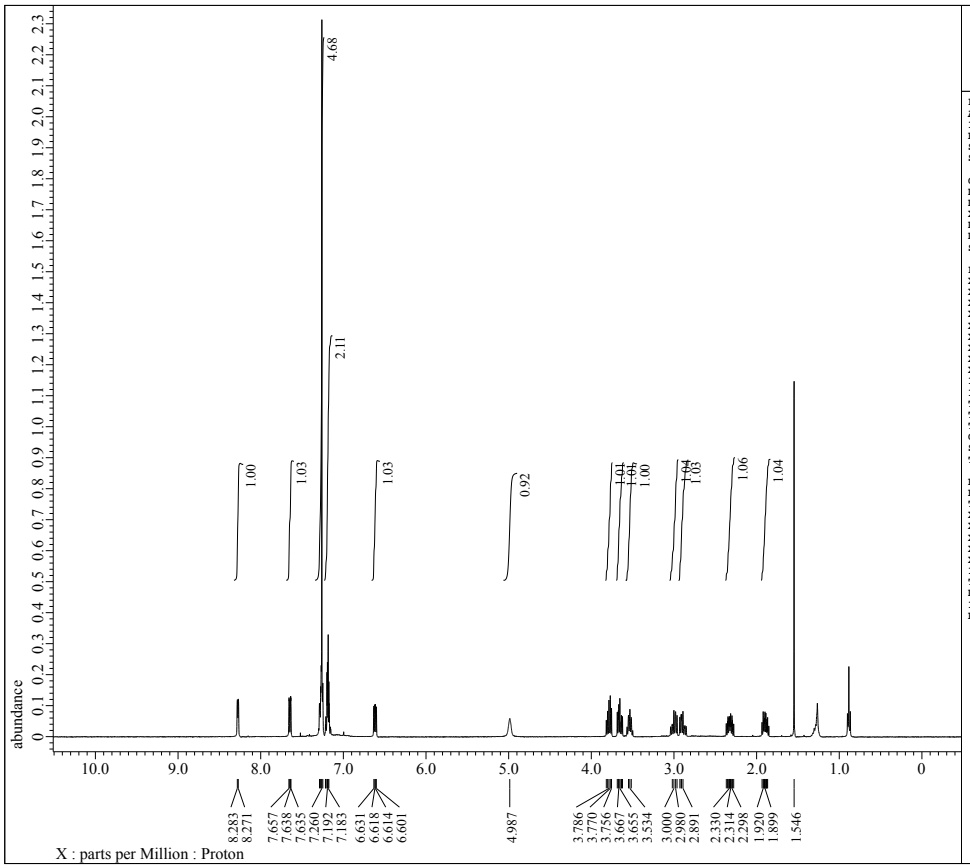



JEOL

```
Filename          = sk#17-081-C_Carbon_copy3-1
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = sk#17-081-C
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    = 0[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             = 282
Total_Scans       = 282

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 23[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]
```

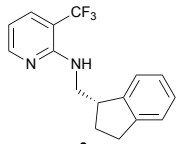



**1a-$d_3$**

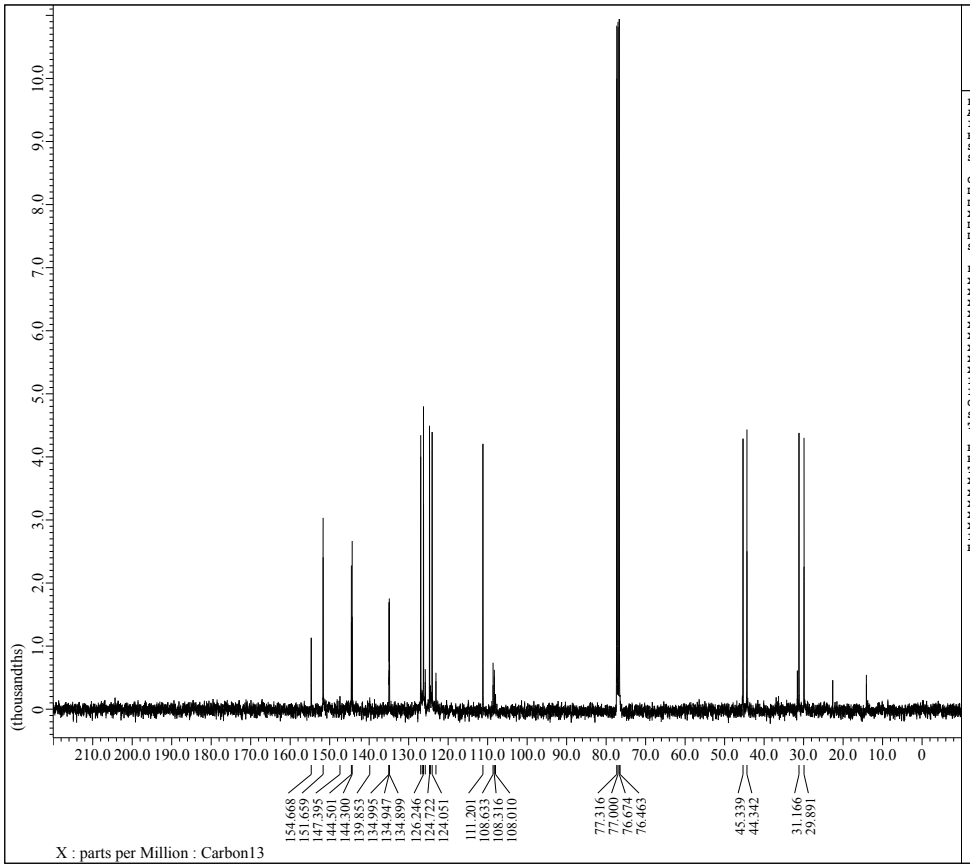



JEOL

```
Filename          = 3aa-proton-1.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = sk#16-sm
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[dC]
X_90_Width        = 6.3[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.15[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```

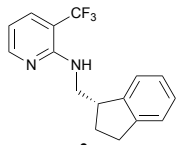



**3aa**

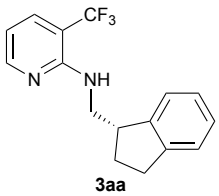



JEOL

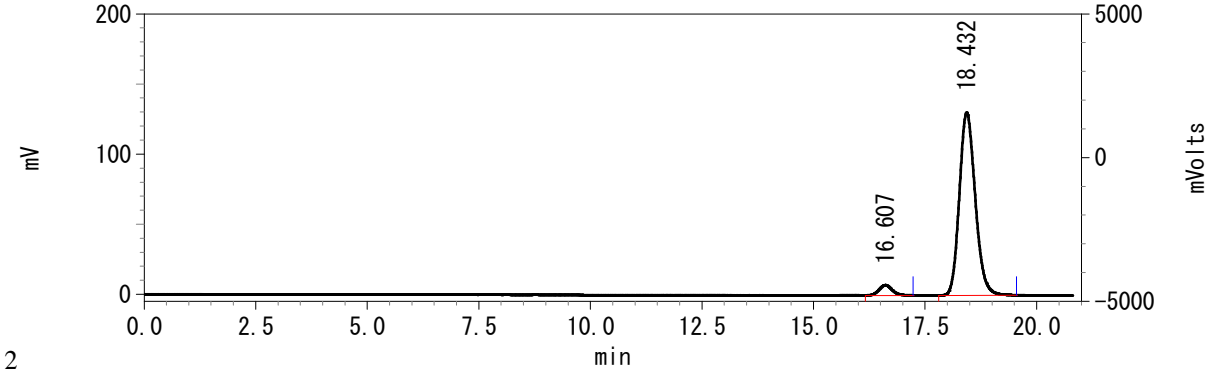
```
Filename          = 3aa-carbon-1.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = sk#16-0xx
Solvent           = CHLOROFORM-D

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             = 238
Total_Scans       = 238

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]
```




**3aa**

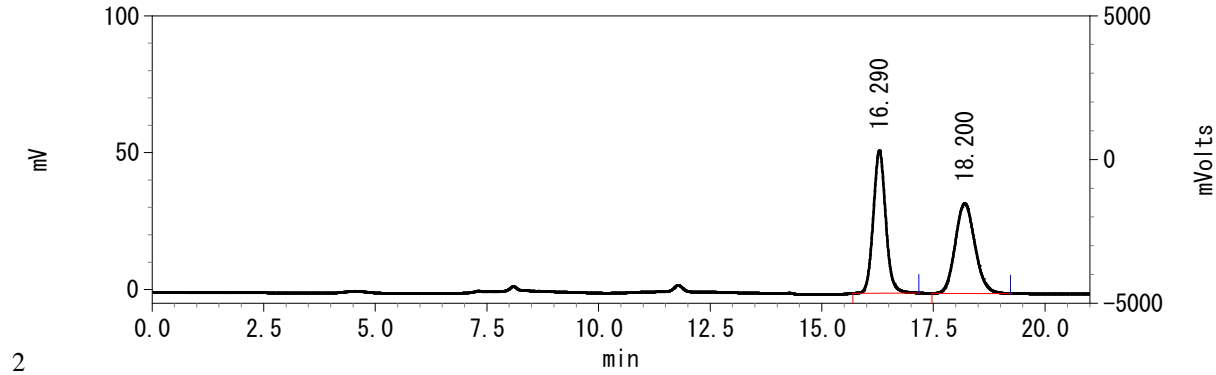



**3aa**



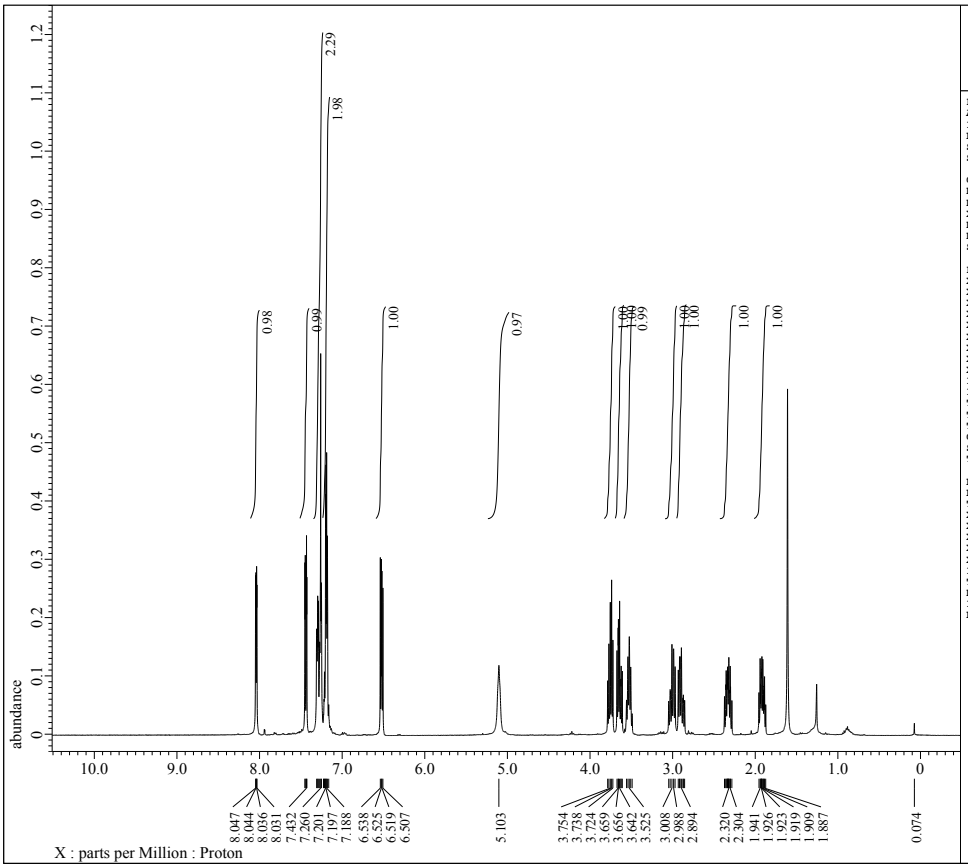

2

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 16.607 | 149004 | 4.411 |
| 2 | 18.432 | 3229208 | 95.589 |



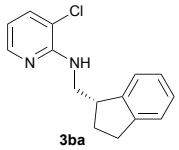

2

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 16.290 | 1007032 | 49.980 |
| 2 | 18.200 | 1007831 | 50.020 |

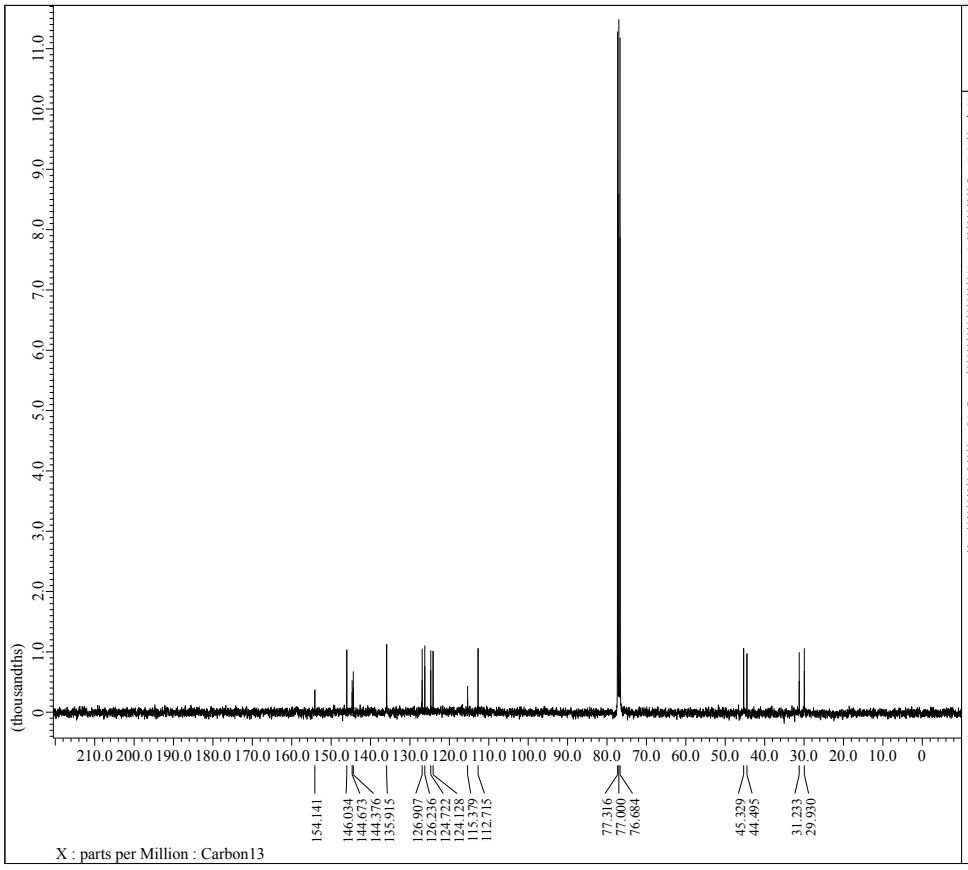



JEOL

```
Filename          = sk#15-050-1_Proton-1-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = sk#15-050-1
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        = 56
Temp_Get          = 21.2[dC]
X_90_Width        = 6.3[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.15[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```

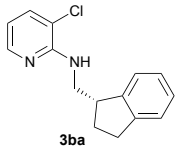



3ba

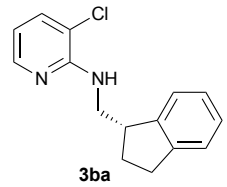



JEOL

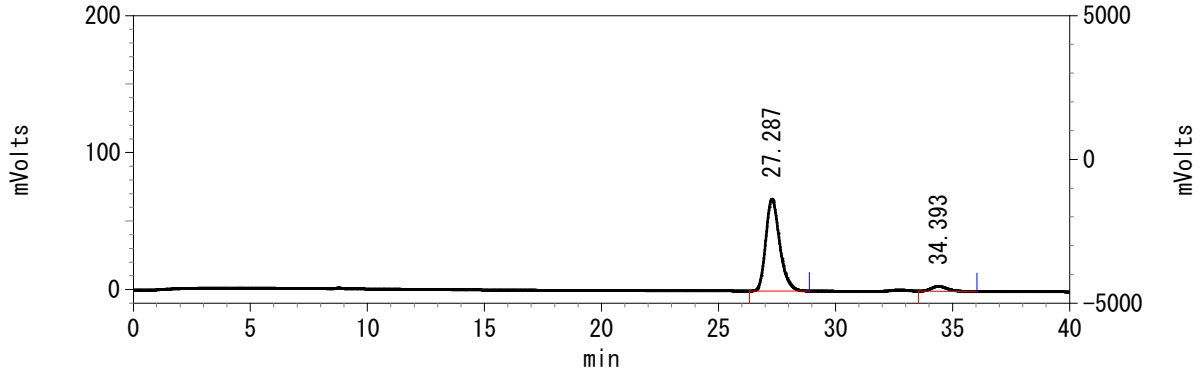
```
Filename          = sk#15-050-1_Carbon-1-5.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = sk#15-050-1
Solvent           = CHLOROFORM-D

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             = 477
Total_Scans       = 477

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.3[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]
```



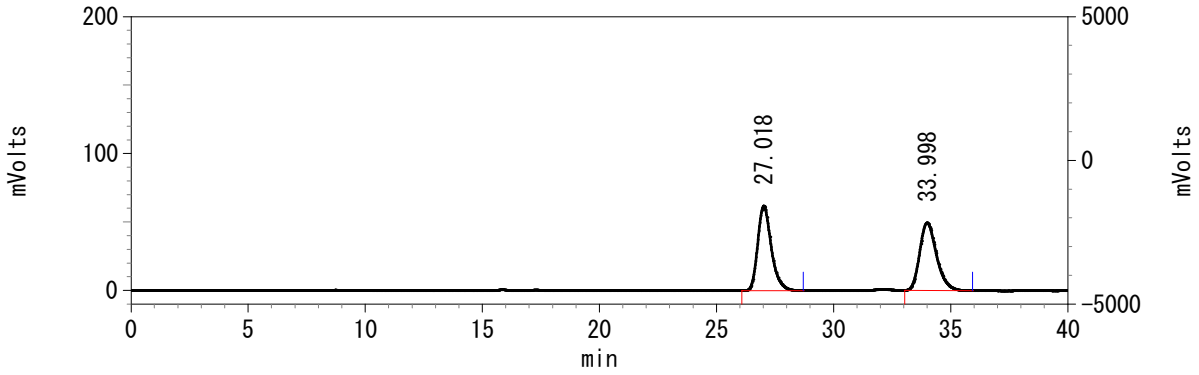

3ba



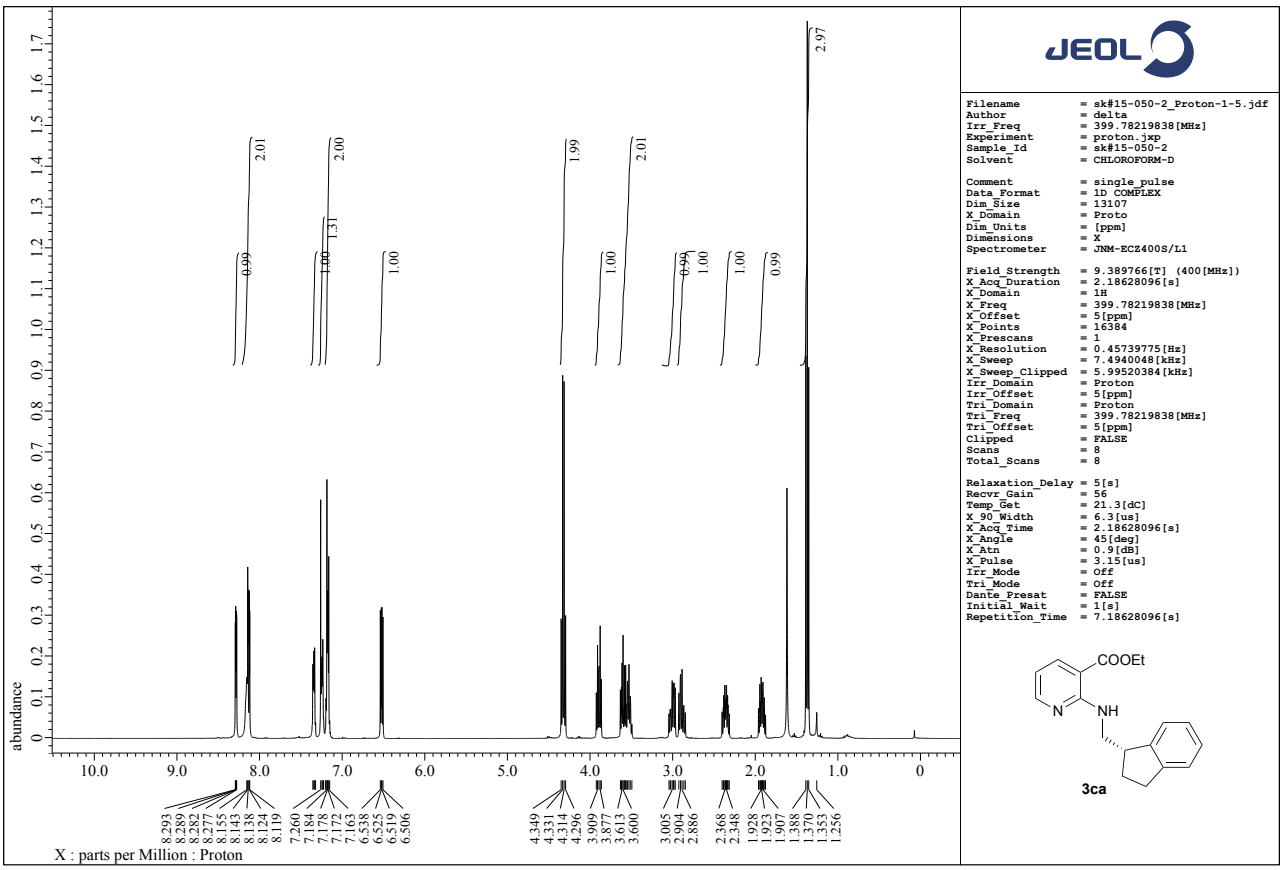

3ba

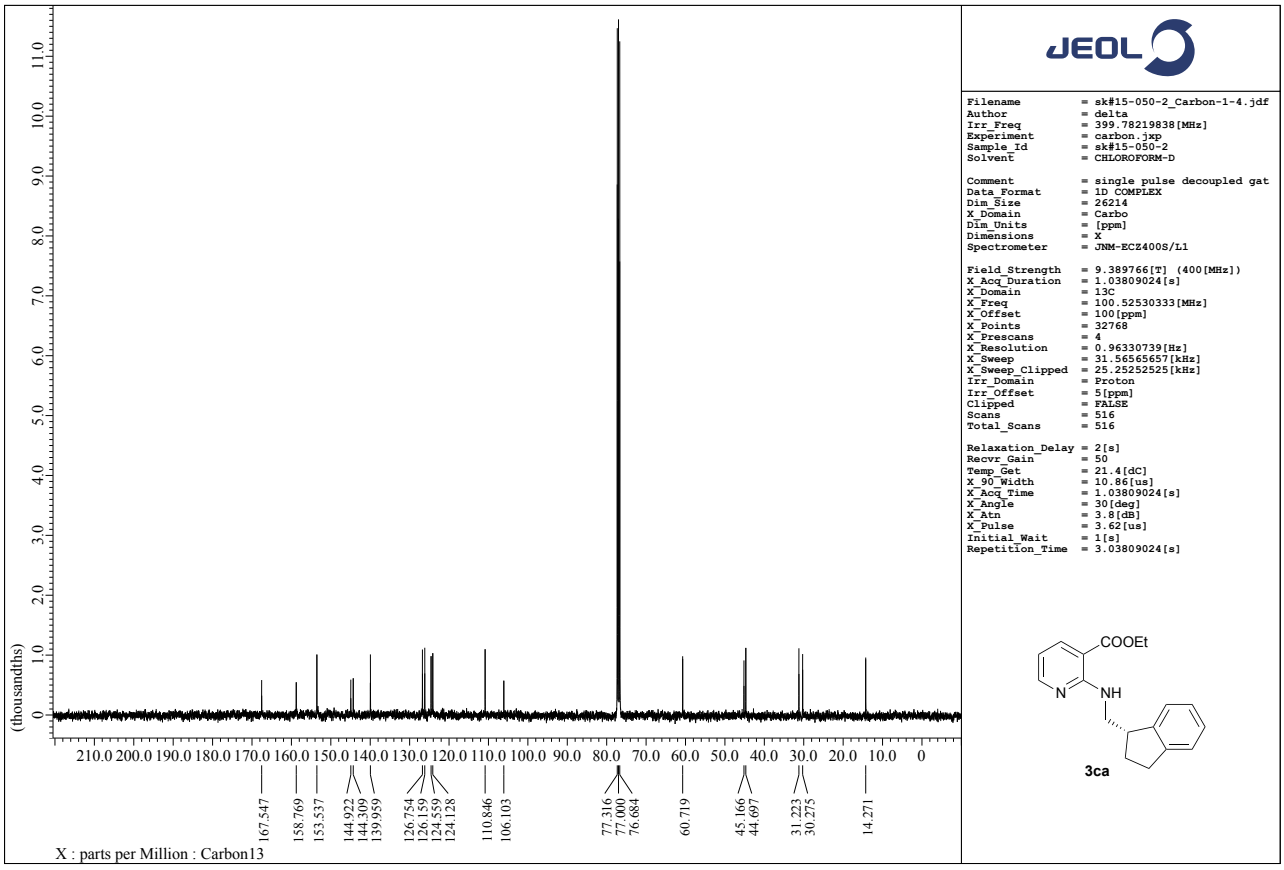


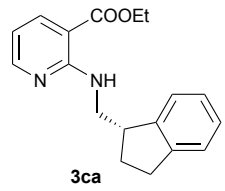

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 27.287 | 2777261 | 93.997 |
| 2 | 34.393 | 177365 | 6.003 |



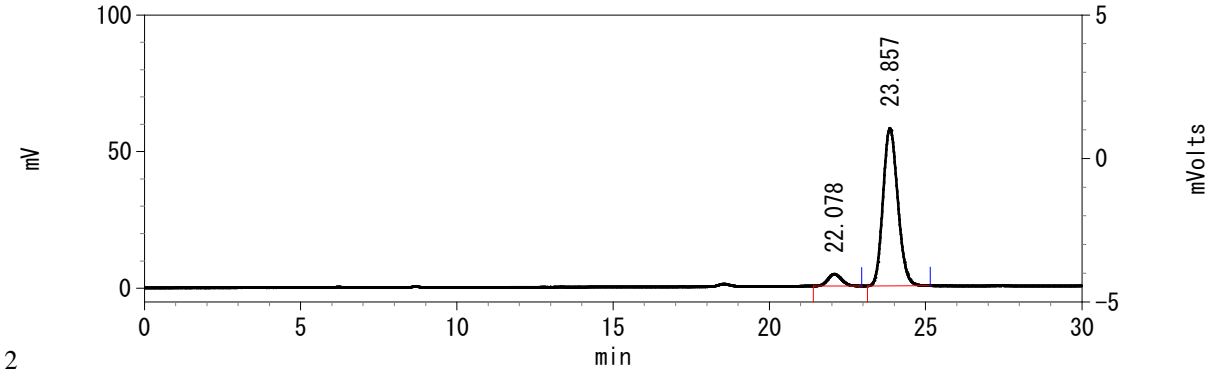

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 27.018 | 2502256 | 50.289 |
| 2 | 33.998 | 2473479 | 49.711 |


abundance
X : parts per Million : Proton
Filename = sk#15-050-2_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = sk#15-050-2
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 = 56
Temp_Get = 21.3[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
COOEt
N
NH
3ca


(thousandths)
X : parts per Million : Carbon13
Filename = sk#15-050-2_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = sk#15-050-2
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 = 516
Total_Scans = 516
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.4[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]
COOEt
N
NH
3ca

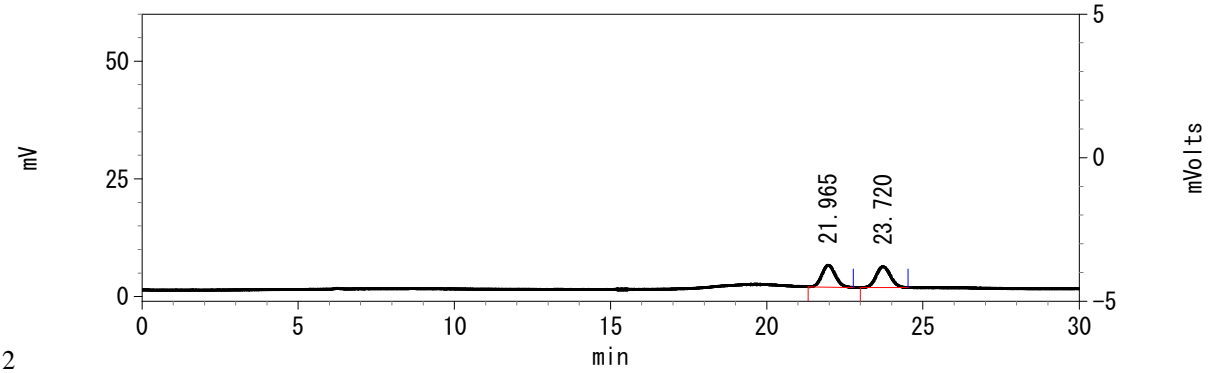



3ca



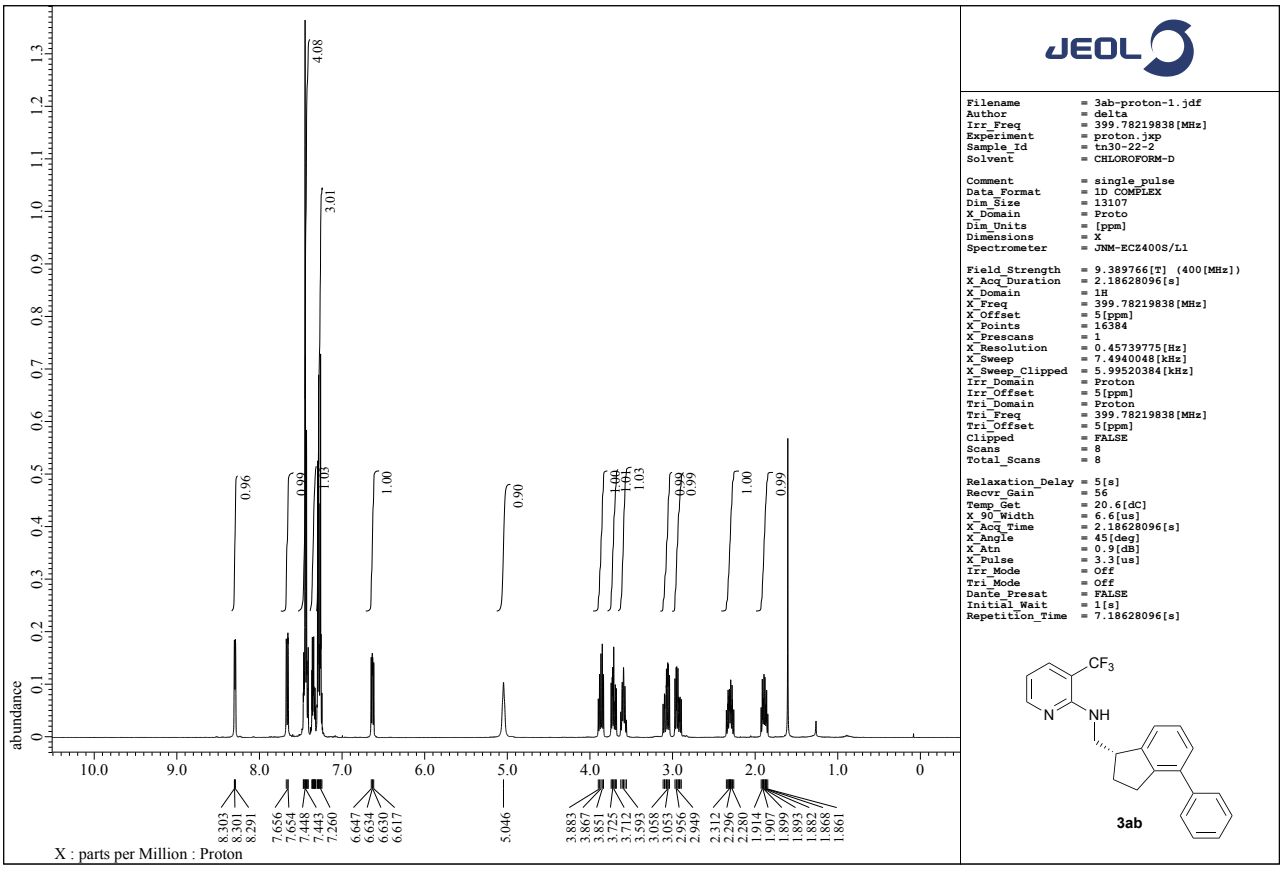

2

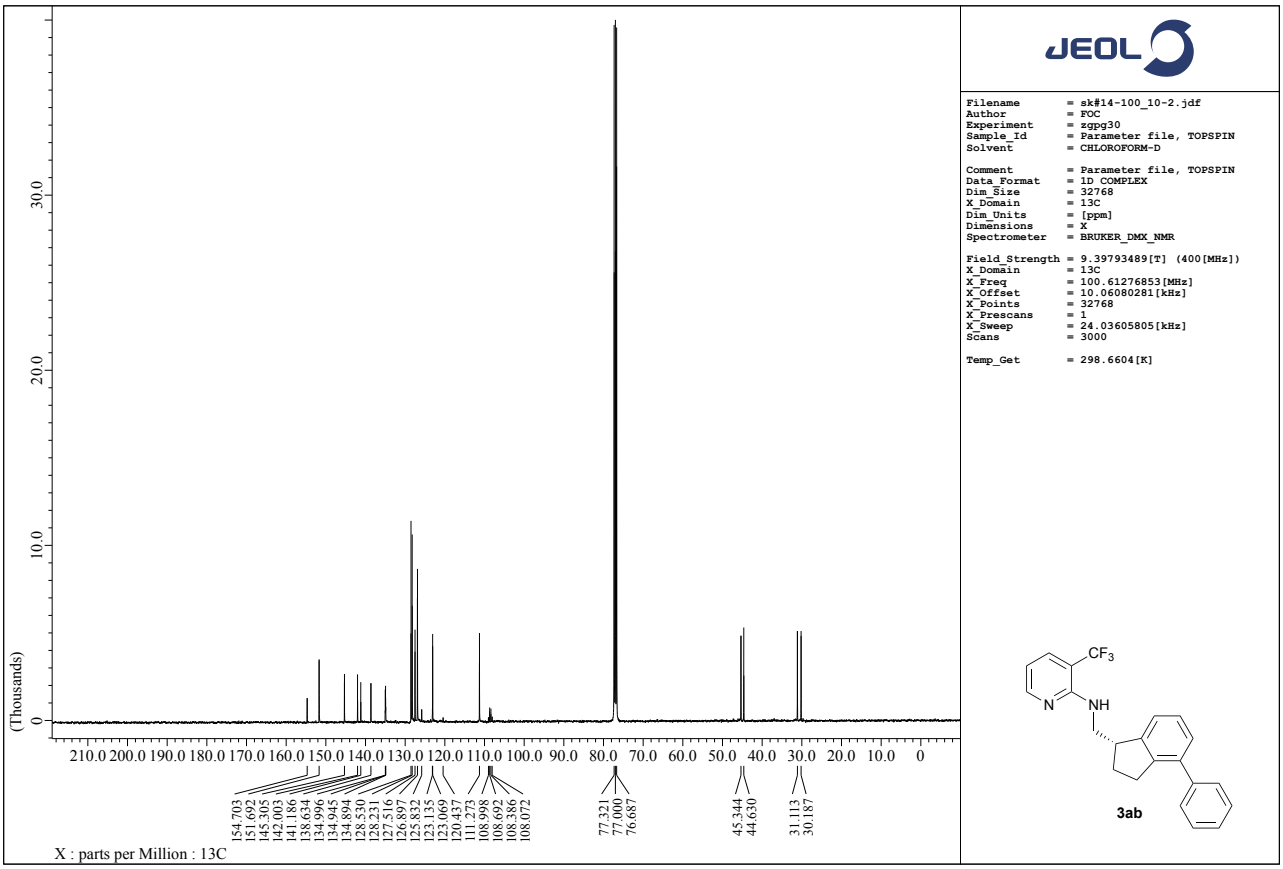
| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 22.078 | 134201 | 6.586 |
| 2 | 23.857 | 1903380 | 93.414 |

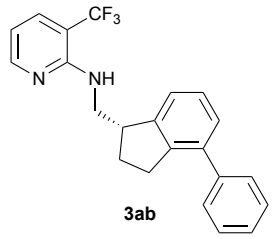



2

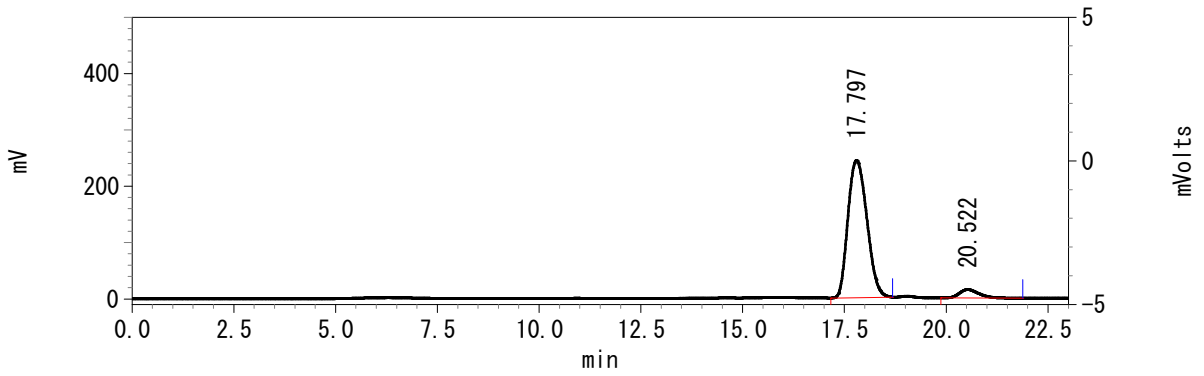
| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 21.965 | 138424 | 49.132 |
| 2 | 23.720 | 143313 | 50.868 |


abundance
X : parts per Million : Proton
Filename = 3ab-proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-22-2
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 = 56
Temp_Get = 20.6[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]
CF3
NH
N
3ab

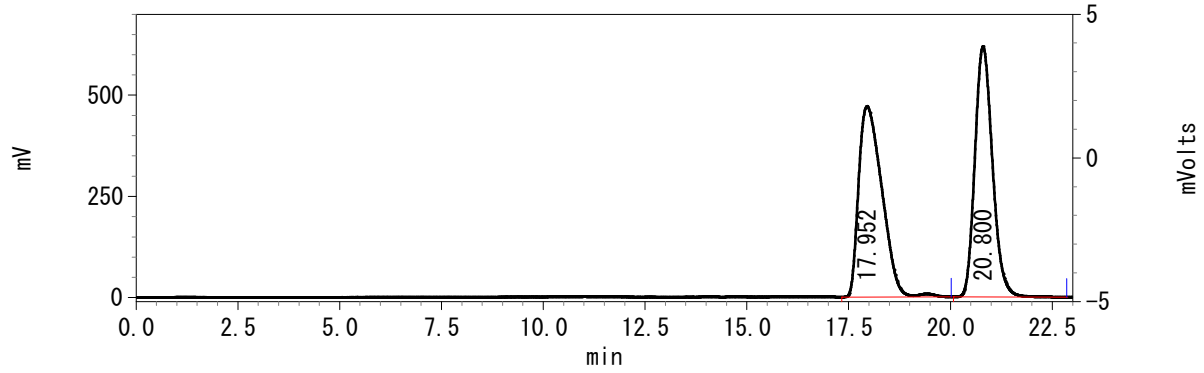

(Thousands)
X : parts per Million : 13C
Filename = sk#14-100_10-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = 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 = 298.6604[K]
CF3
NH
N
3ab



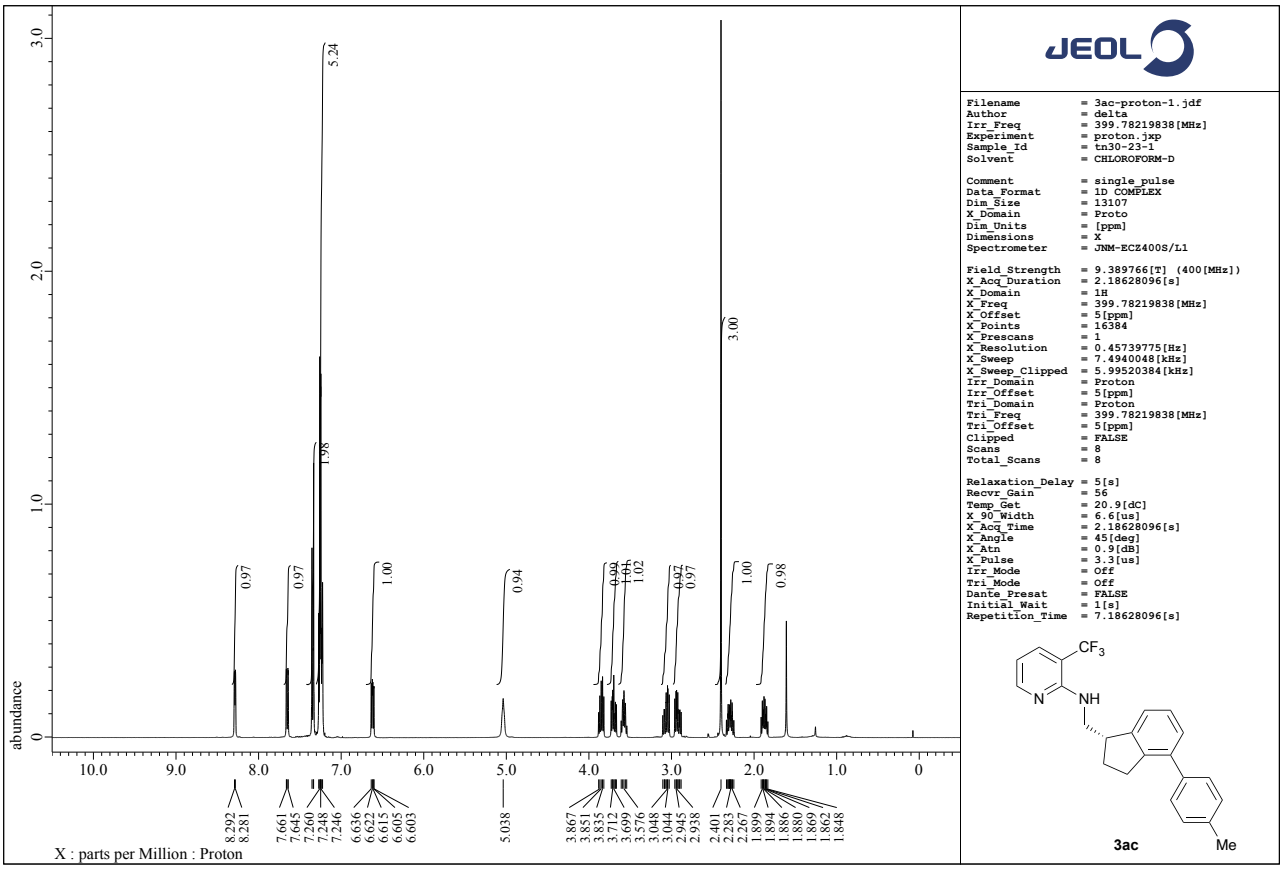

**3ab**

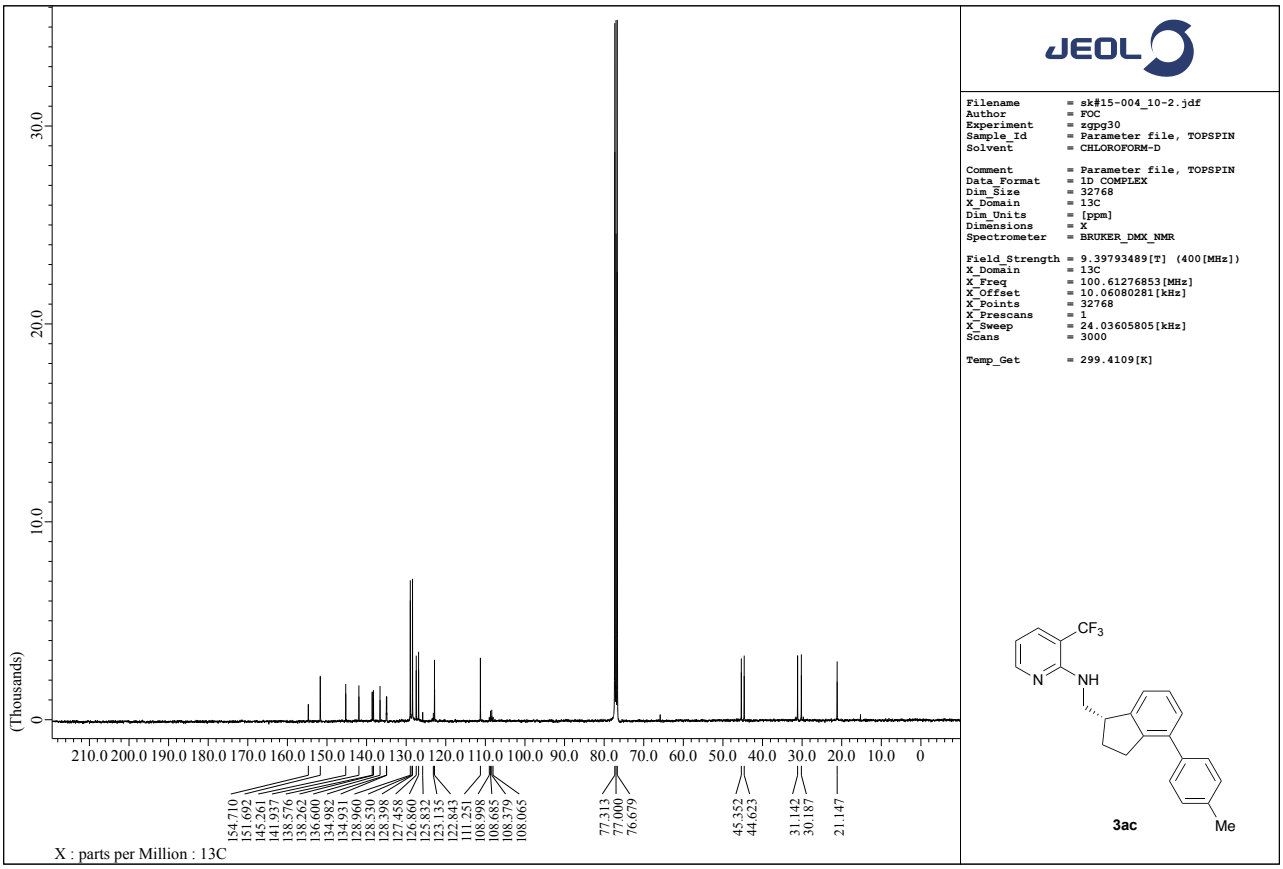


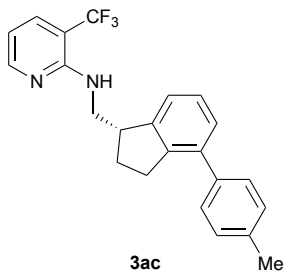

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 17.797 | 7894415 | 94.004 |
| 2 | 20.522 | 503580 | 5.996 |



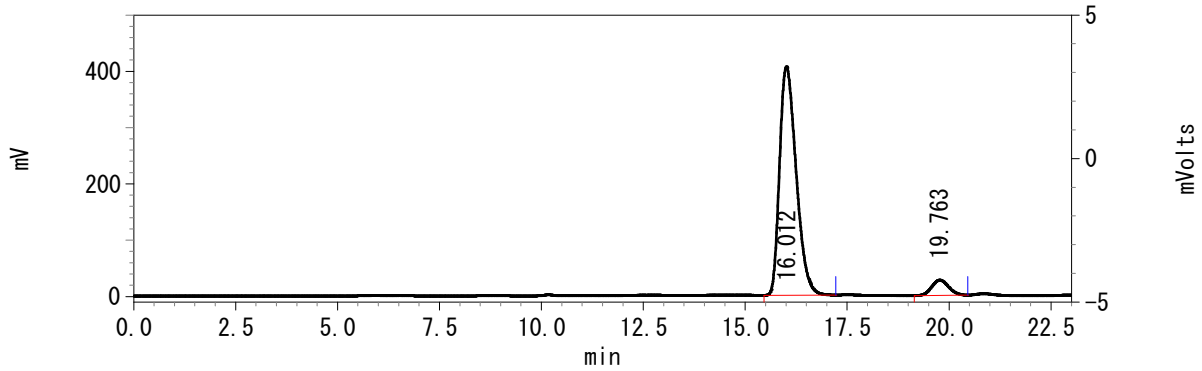

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 17.952 | 18776782 | 50.244 |
| 2 | 20.800 | 18594506 | 49.756 |


abundance
X : parts per Million : Proton
Filename = 3ac-proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-23-1
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 = 56
Temp_Get = 20.9[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
N
NH
Me
3ac

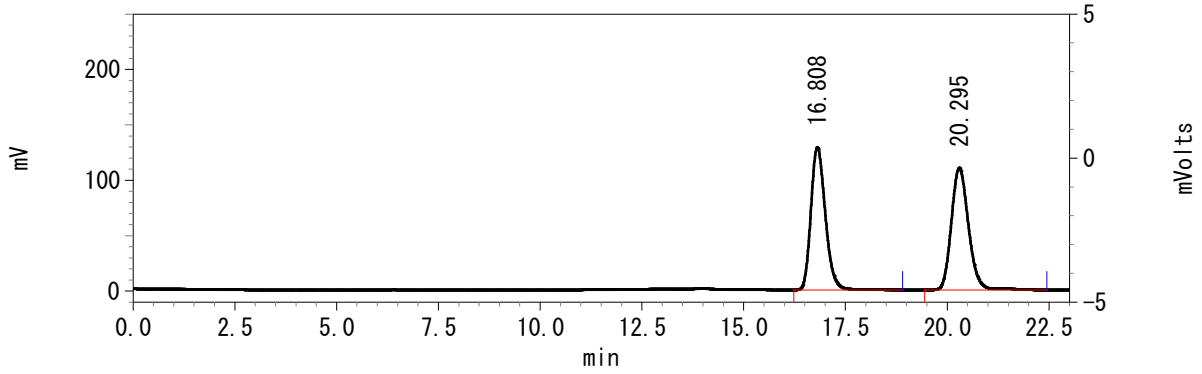

(Thousands)
X : parts per Million : 13C
Filename = sk#15-004_10-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = 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 = 299.4109[K]
CF3
N
NH
Me
3ac



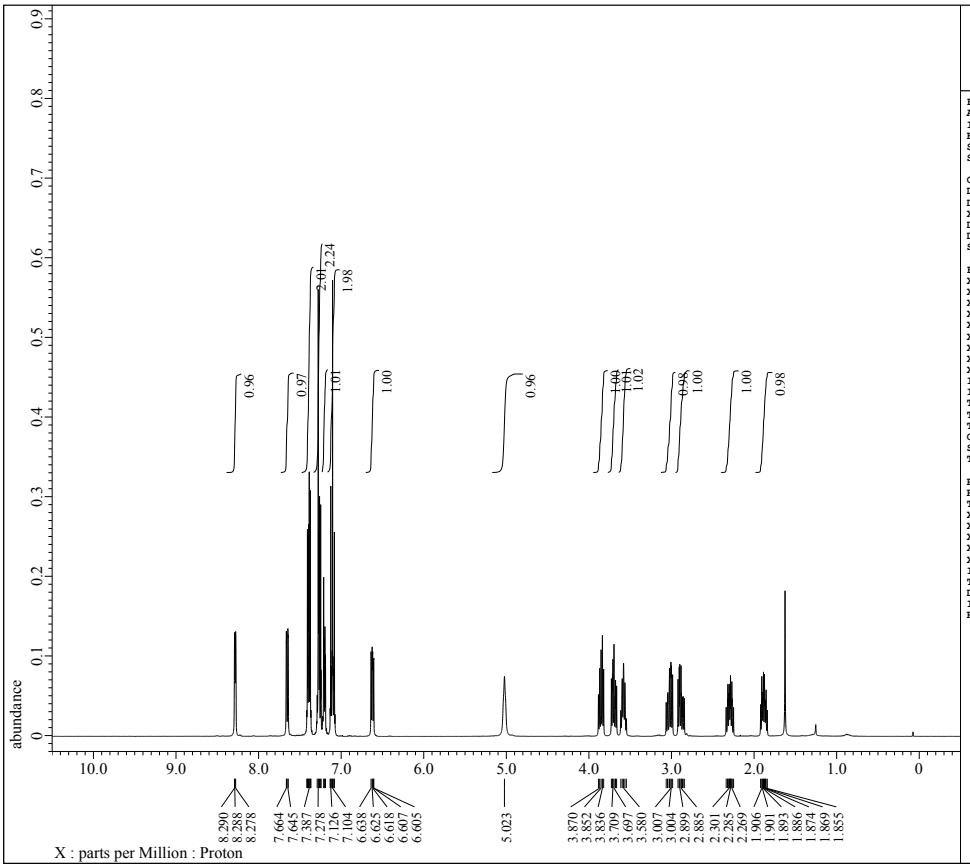

3ac

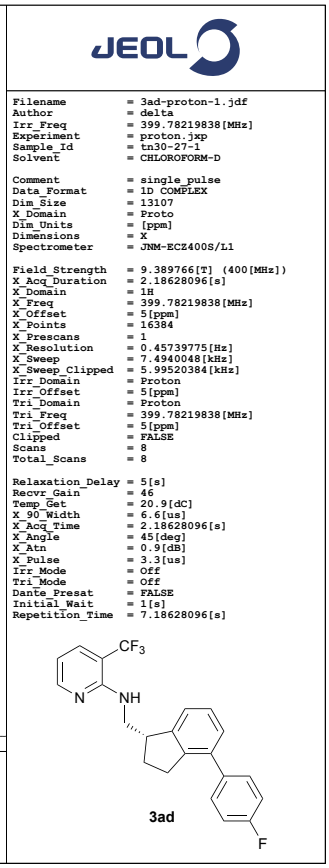


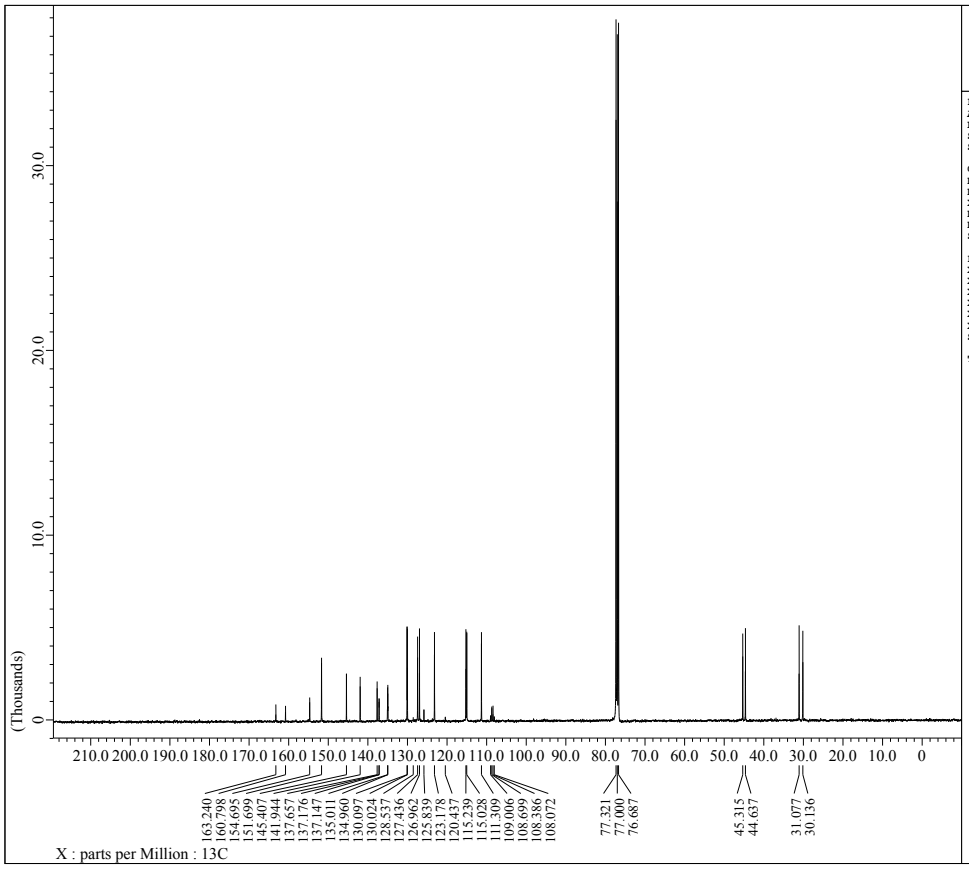

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 16.012 | 11488774 | 93.616 |
| 2 | 19.763 | 783515 | 6.384 |

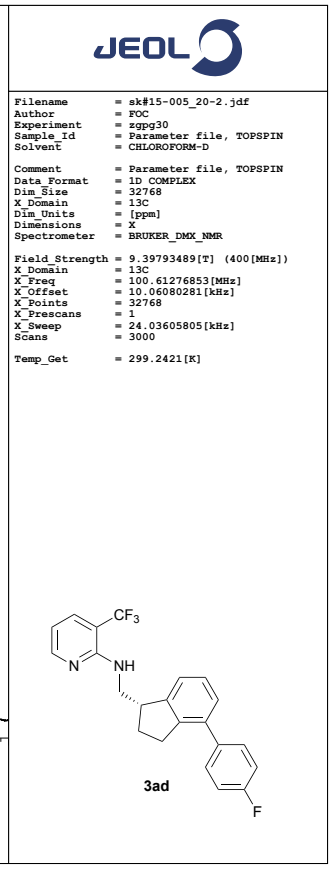


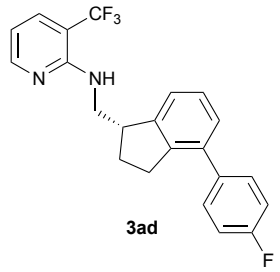

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 16.808 | 3177575 | 49.625 |
| 2 | 20.295 | 3225662 | 50.375 |


abundance
X : parts per Million : Proton
0.96
0.97
2.01
2.24
1.98
1.01
1.00
0.96
1.00
1.01
1.02
0.98
1.00
1.00
0.98
8.290
8.288
8.278
7.664
7.645
7.387
7.278
7.126
7.104
6.638
6.625
6.618
6.607
6.605
5.023
3.870
3.852
3.836
3.709
3.697
3.580
3.007
3.004
2.899
2.885
2.301
2.285
2.269
1.906
1.901
1.893
1.886
1.874
1.869
1.855

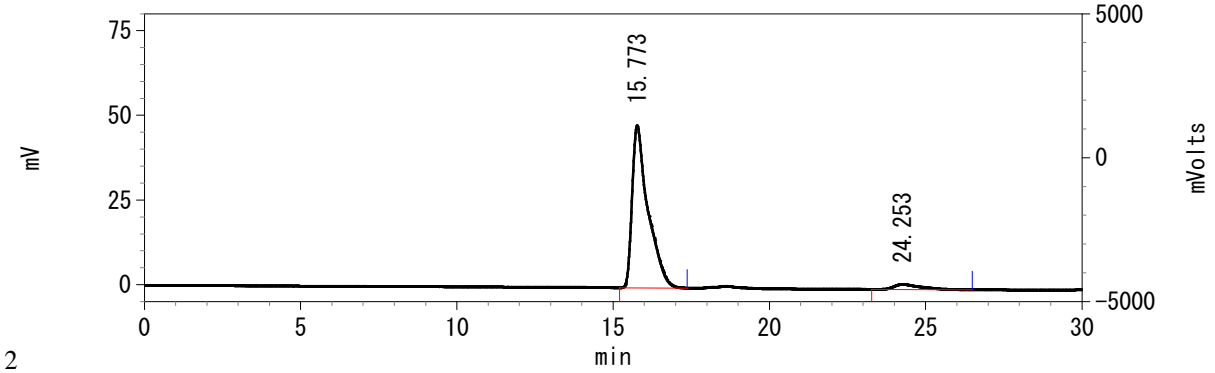

Filename = 3ad-proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-27-1
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 20.9[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
N
NH
3ad
F


(Thousands)
X : parts per Million : 13C
163.240
160.798
154.695
151.699
145.407
141.944
137.657
137.176
137.147
135.011
134.960
130.097
130.024
128.537
127.436
126.962
125.839
123.178
120.437
115.239
115.028
111.309
109.006
108.699
108.386
108.072
77.321
77.000
76.687
45.315
44.637
31.077
30.136


Filename = sk#15-005_20-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
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 = 299.2421[K]
CF3
N
NH
3ad
F

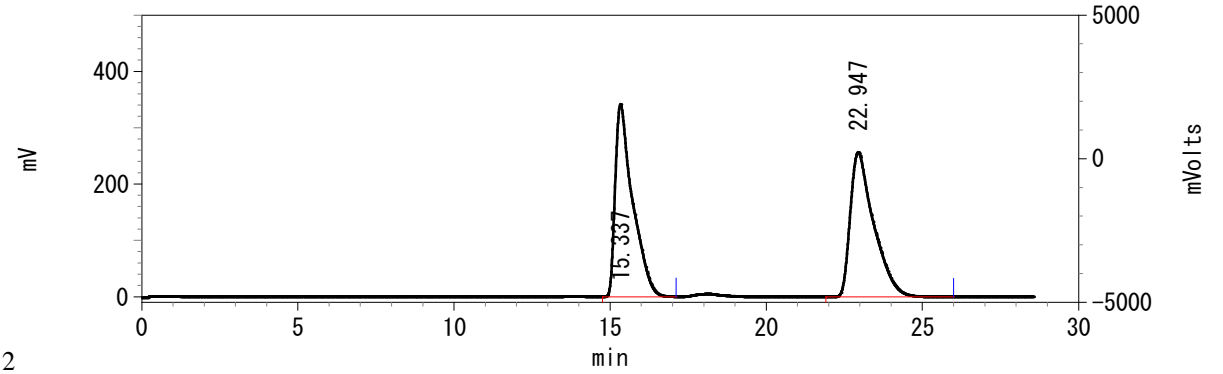



3ad



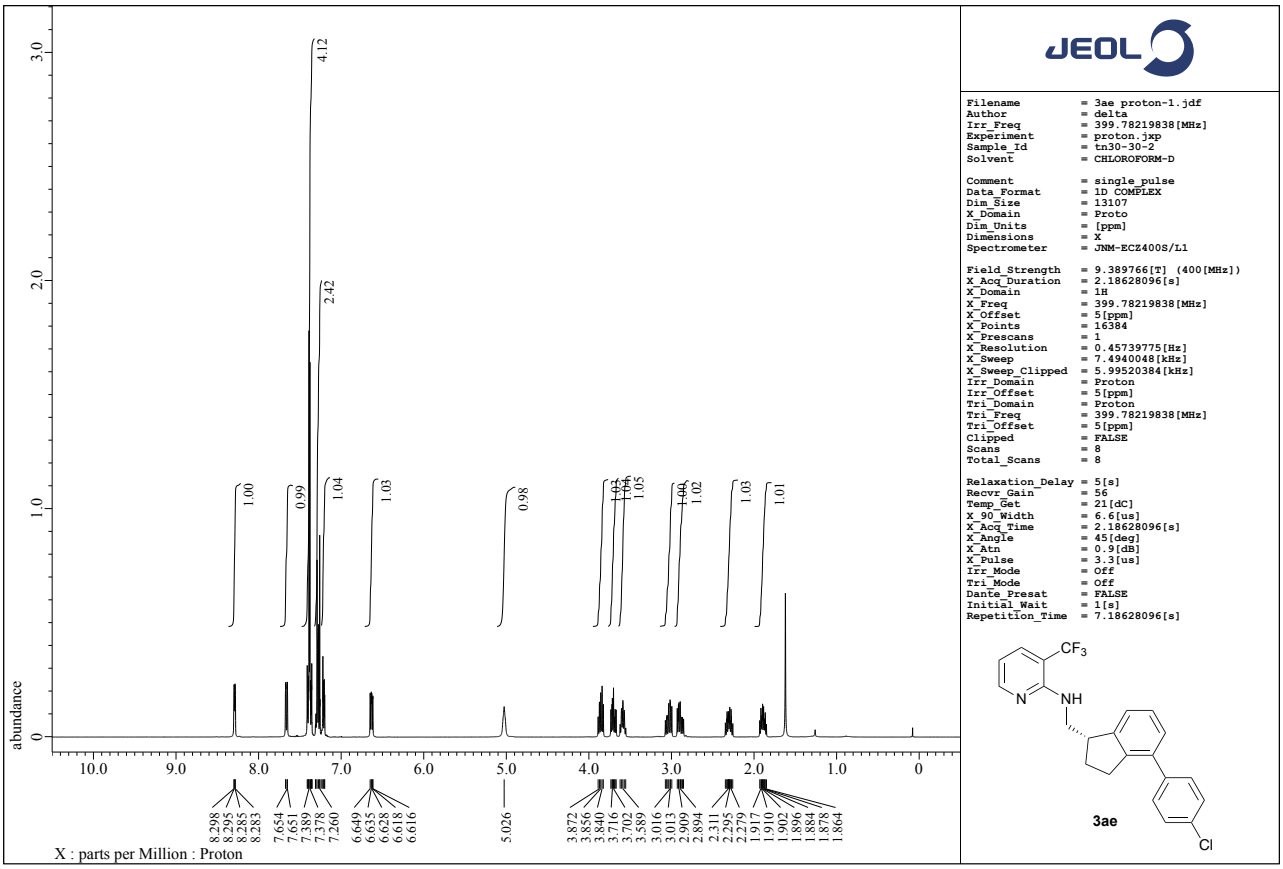

2

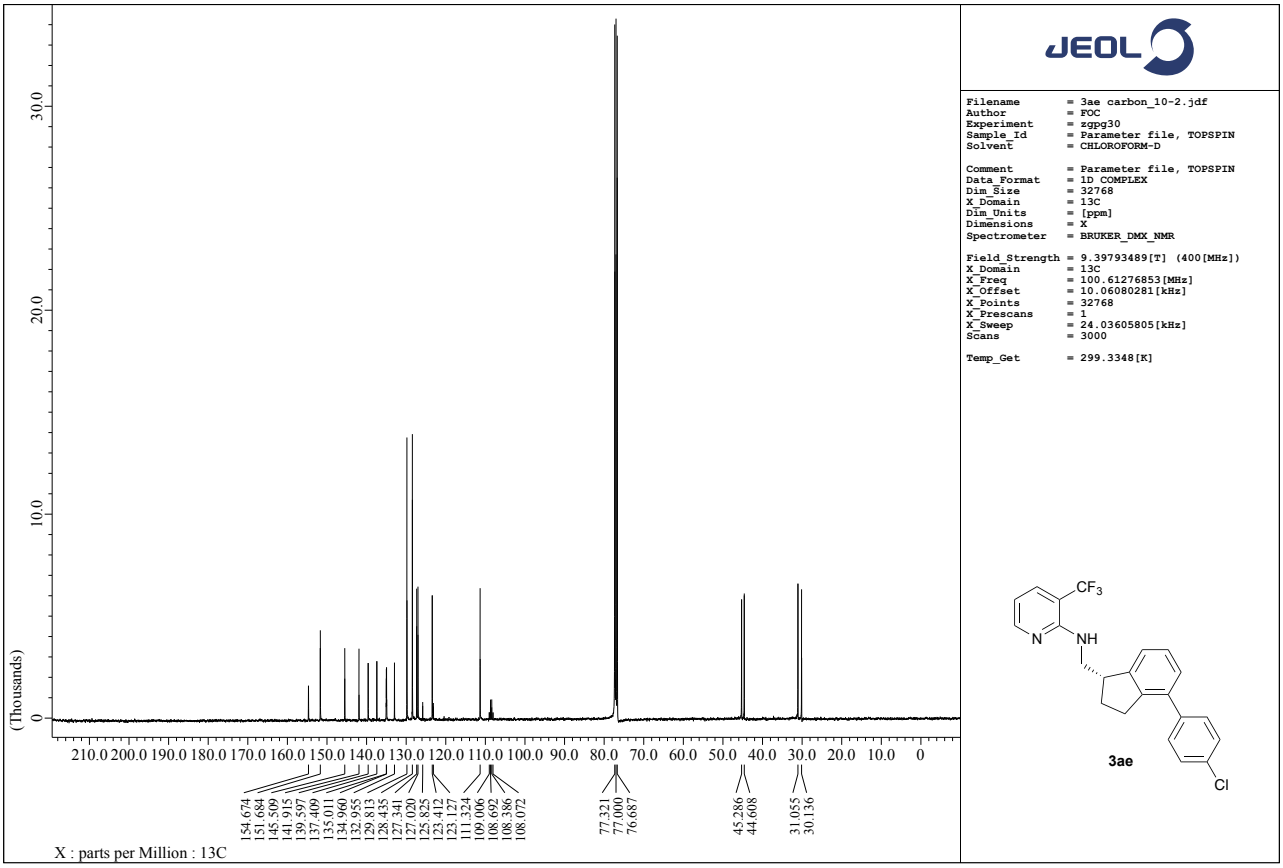
| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 15.773 | 1692724 | 94.885 |
| 2 | 24.253 | 91259 | 5.115 |

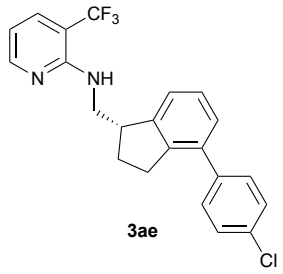



2

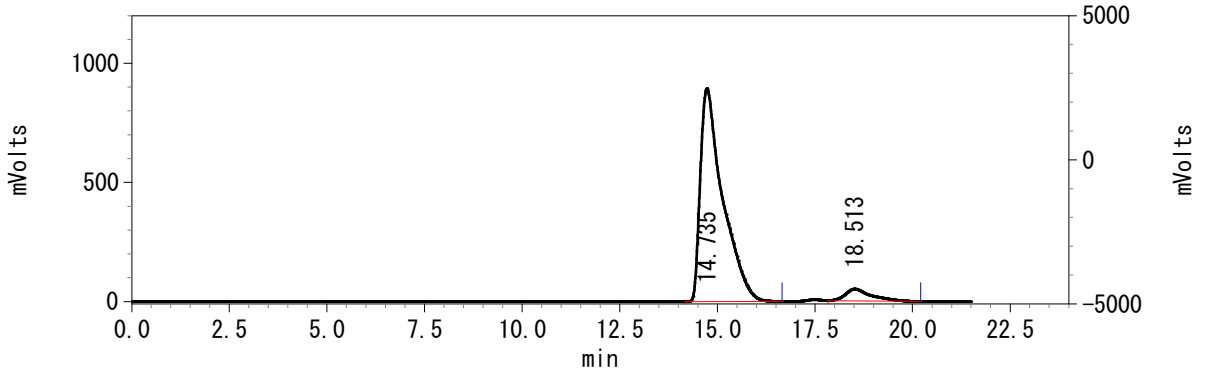
| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 15.337 | 13242652 | 49.729 |
| 2 | 22.947 | 13386789 | 50.271 |


abundance
X : parts per Million : Proton
Filename = 3ae proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-30-2
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 = 56
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
NH
N
Cl
3ae

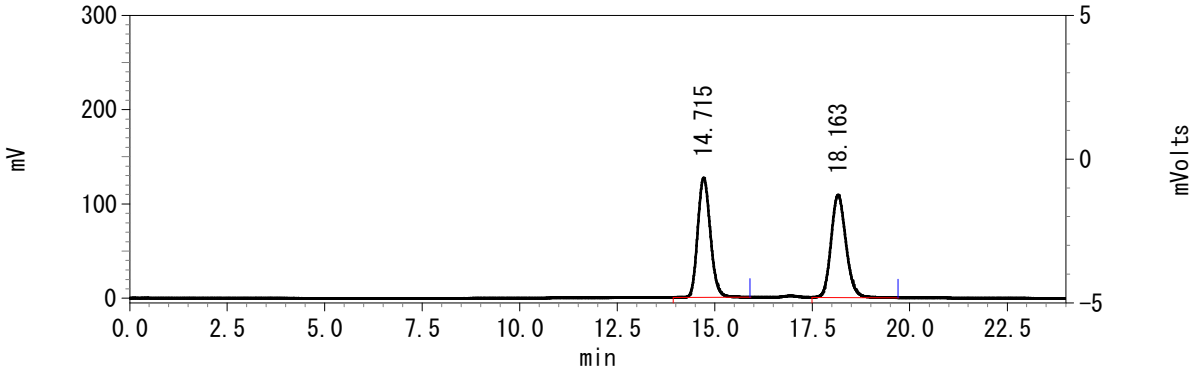

(Thousands)
X : parts per Million : 13C
Filename = 3ae carbon_10-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = 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 = 299.3348[K]
CF3
NH
N
Cl
3ae



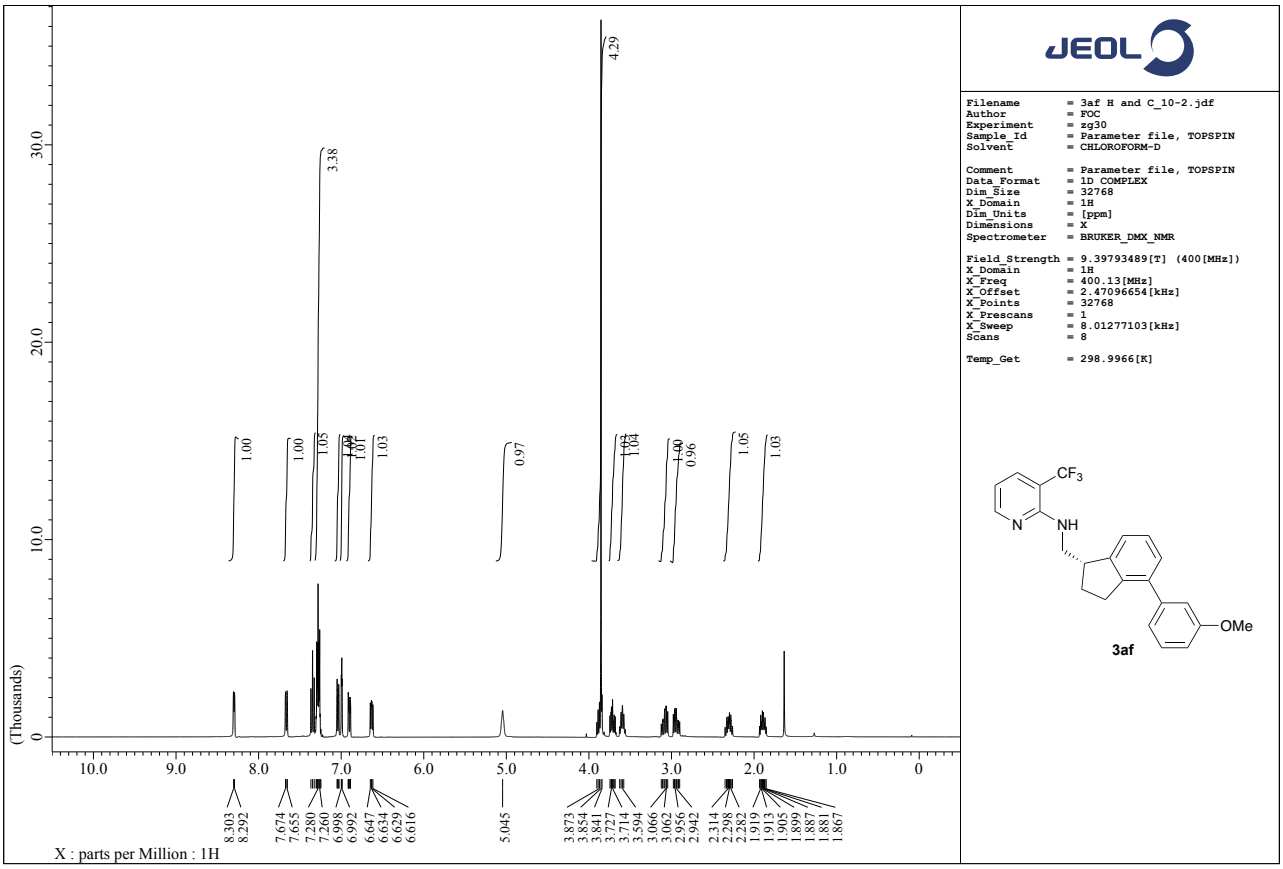

3ae



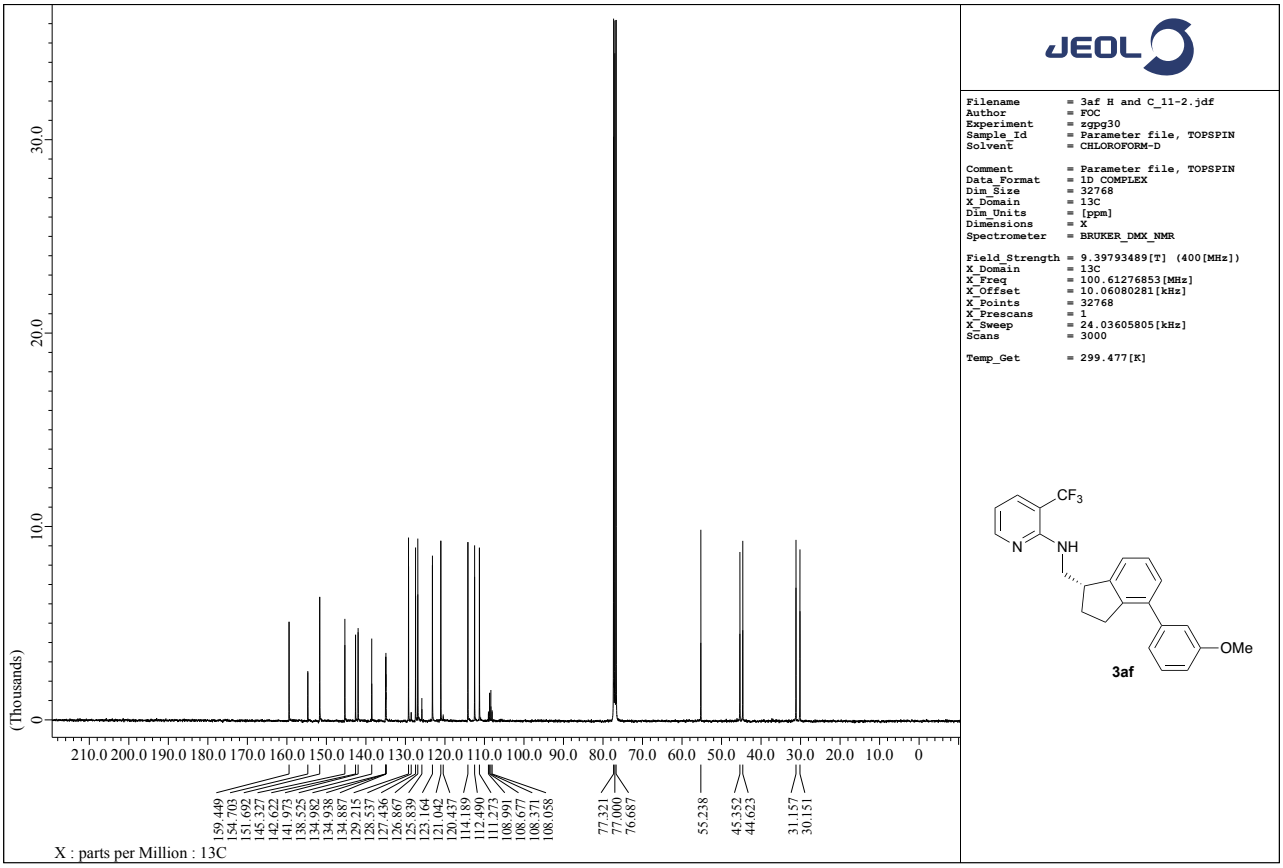

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 14.735 | 35611257 | 93.982 |
| 2 | 18.513 | 2280218 | 6.018 |



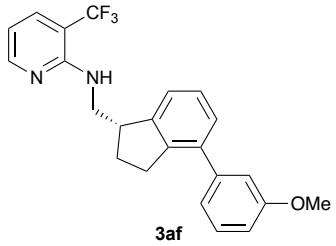

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 14.715 | 2847446 | 49.740 |
| 2 | 18.163 | 2877259 | 50.260 |


(Thousands)
30.0
20.0
10.0
0
4.29
3.38
1.00
1.00
1.05
1.00
1.07
1.03
0.97
1.03
1.04
1.00
0.96
1.05
1.03
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.303 8.292 7.674 7.655 7.280 7.260 6.998 6.992 6.647 6.634 6.629 6.616 5.045 3.873 3.854 3.841 3.727 3.714 3.594 3.066 3.062 2.956 2.942 2.314 2.298 2.282 1.919 1.913 1.905 1.899 1.887 1.881 1.867
X : parts per Million : 1H

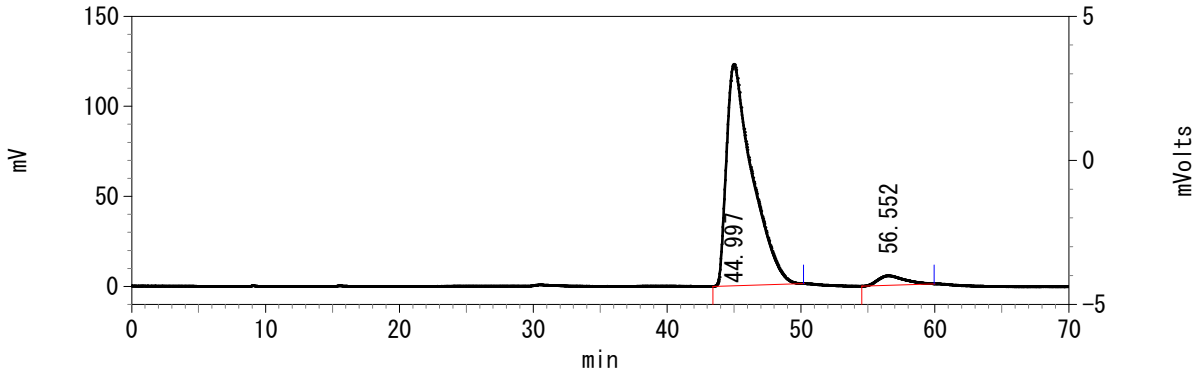

Filename = 3af H and C_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 298.9966[K]
CF3
N
NH
OMe
3af


(Thousands)
30.0
20.0
10.0
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
159.449 154.703 151.692 145.327 142.622 141.973 138.525 134.982 134.938 134.887 129.215 128.537 127.436 126.867 125.839 123.164 121.042 120.437 114.189 112.490 111.273 108.991 108.677 108.371 108.058
77.321 77.000 76.687
55.238
45.352 44.623
31.157 30.151
X : parts per Million : 13C

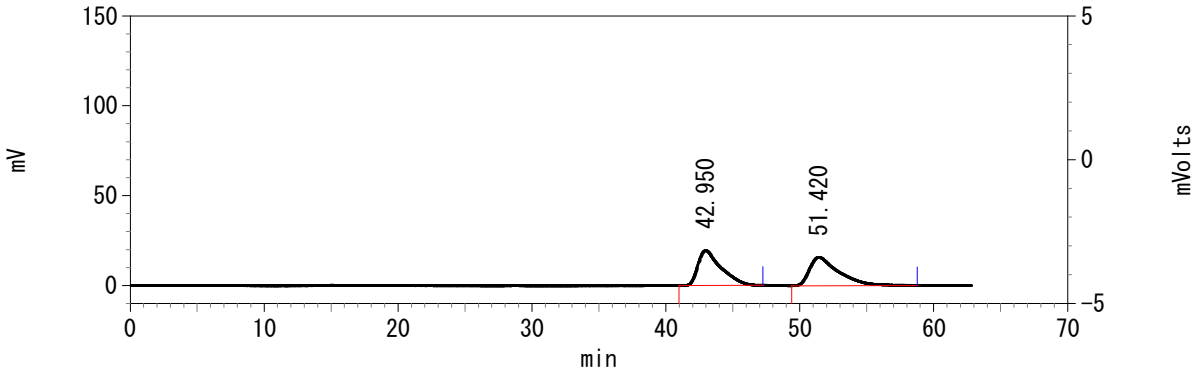

Filename = 3af H and C_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 299.477[K]
CF3
N
NH
OMe
3af



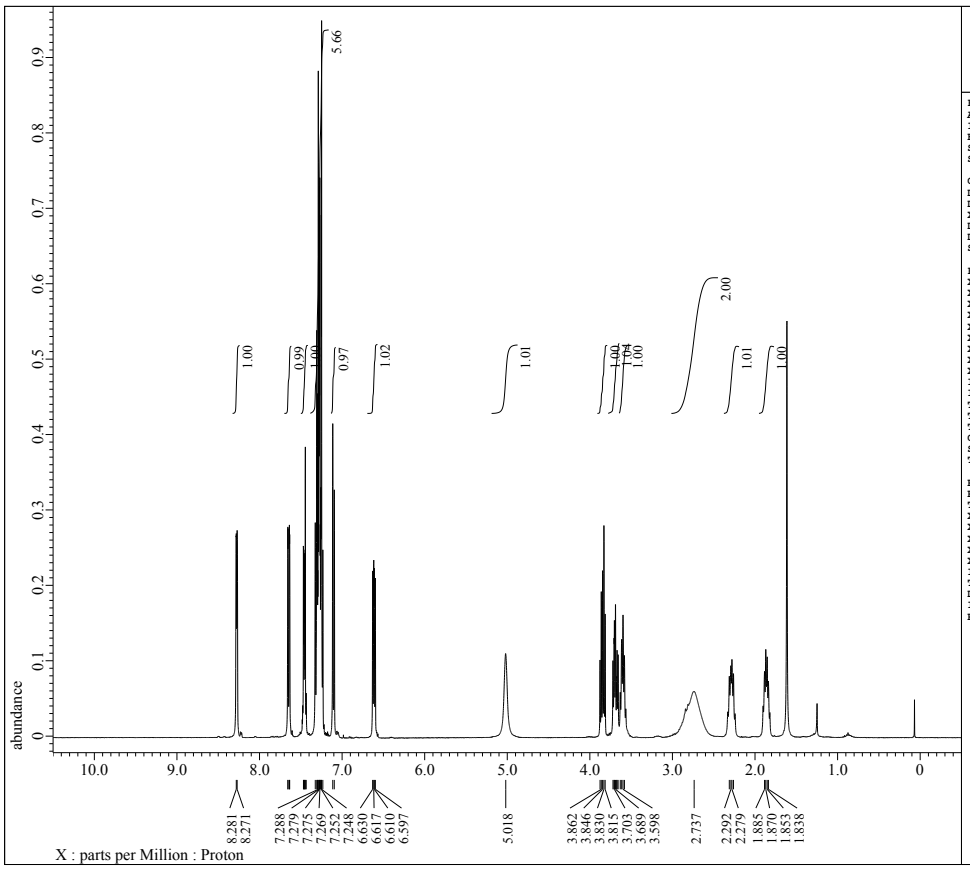

3af




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 44.997 | 16334425 | 95.691 |
| 2 | 56.552 | 735559 | 4.309 |




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 42.950 | 2338092 | 49.506 |
| 2 | 51.420 | 2384785 | 50.494 |

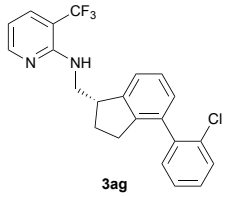



Filename = tn30-32-1_Proton-1-9.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-32-1
Solvent = CHLOROFORM-D

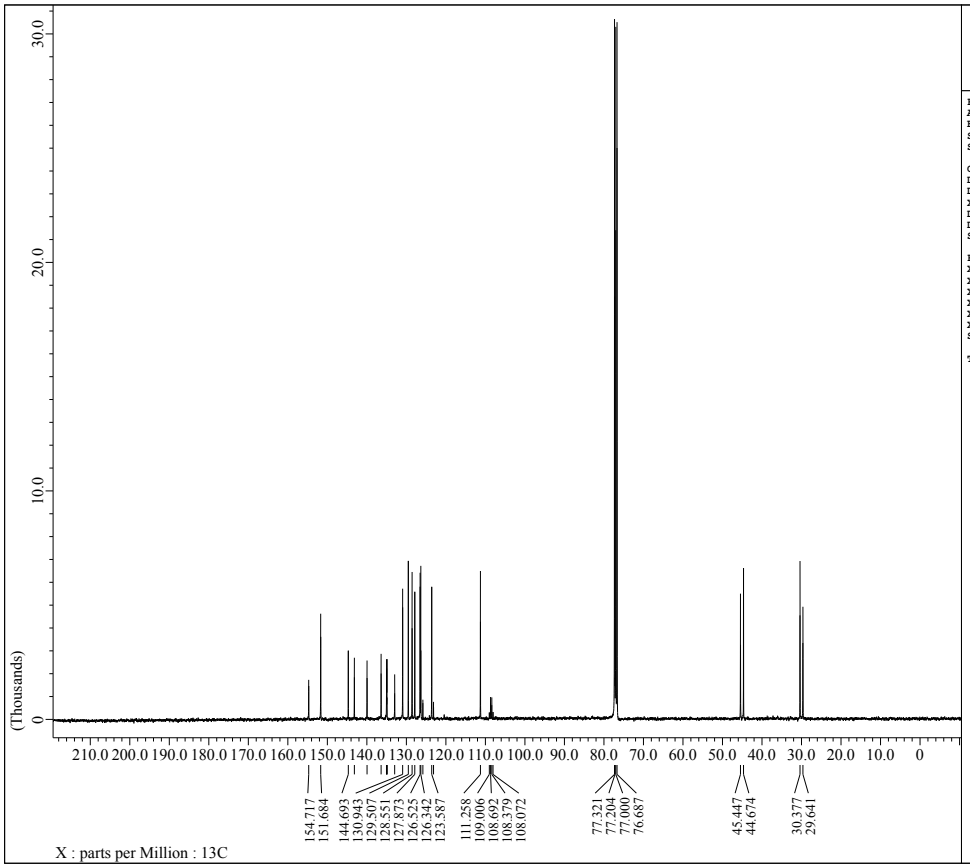
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 56
Temp_Get = 20.9[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]




**3ag**

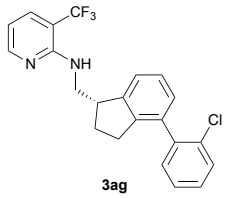



Filename = 3ag carbon_10-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D

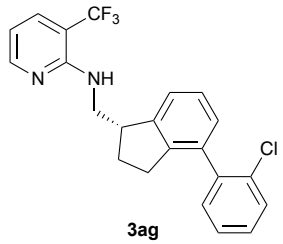
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR

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

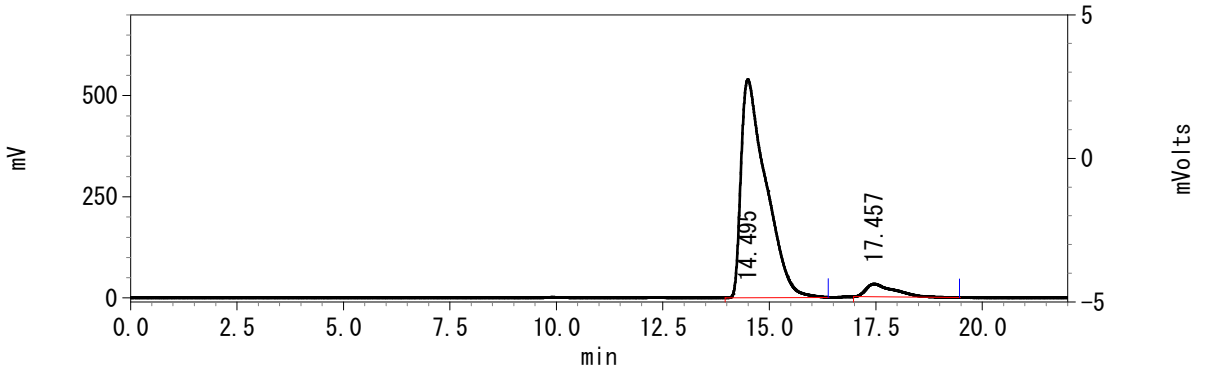
Temp_Get = 299.6772[K]



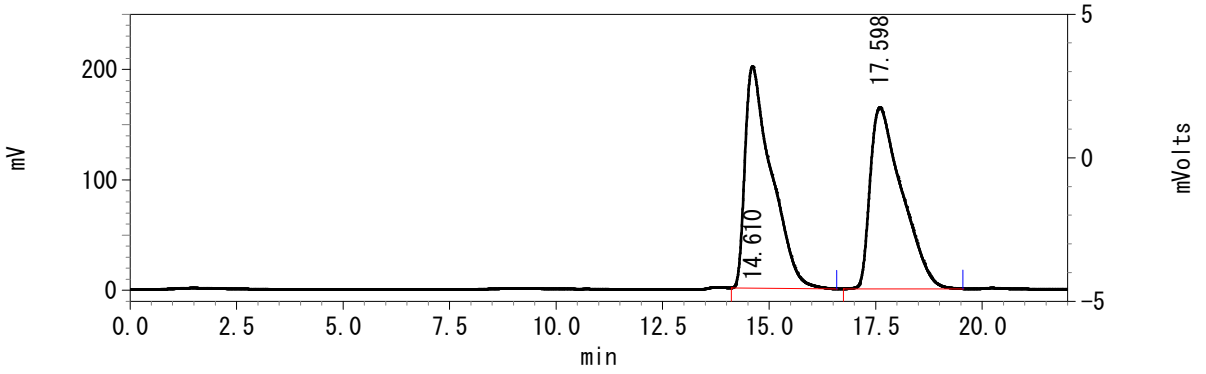

**3ag**



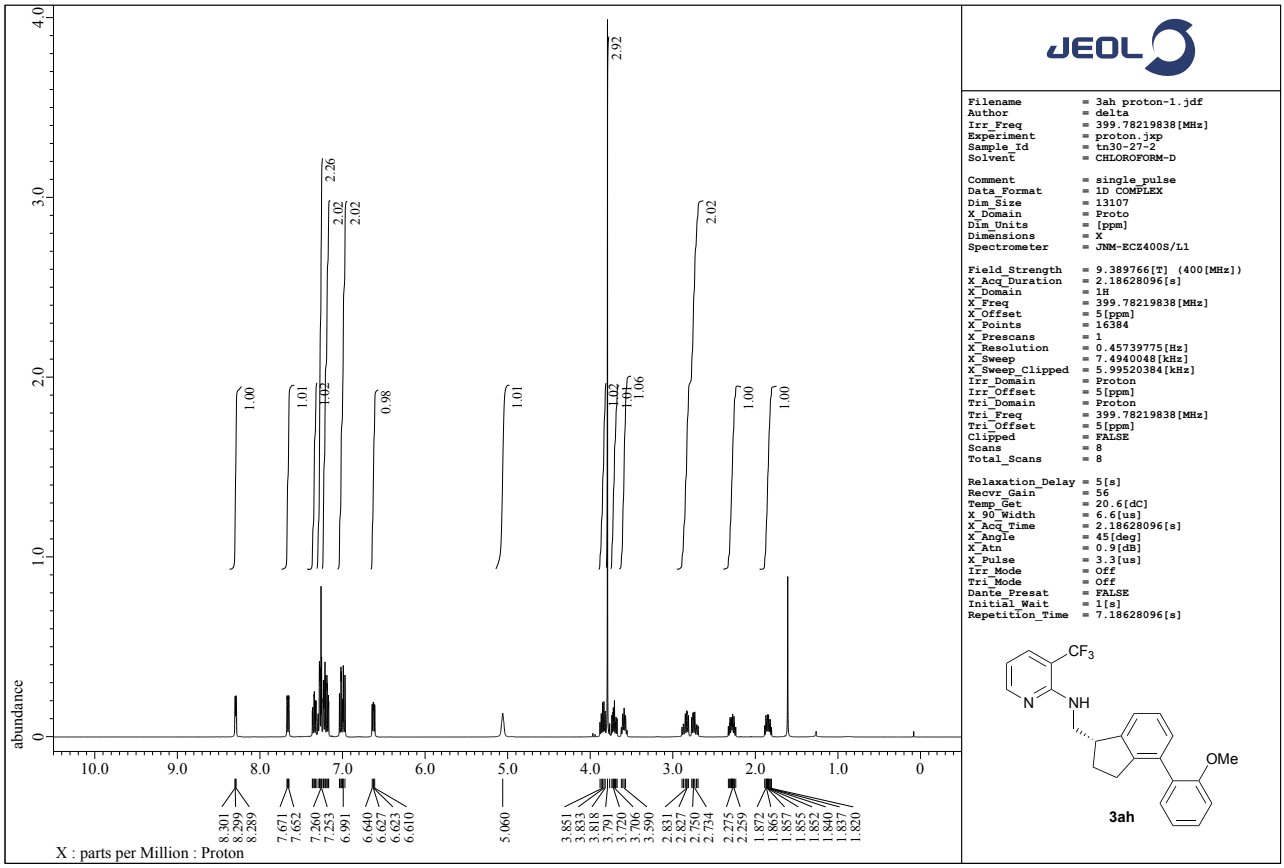

3ag

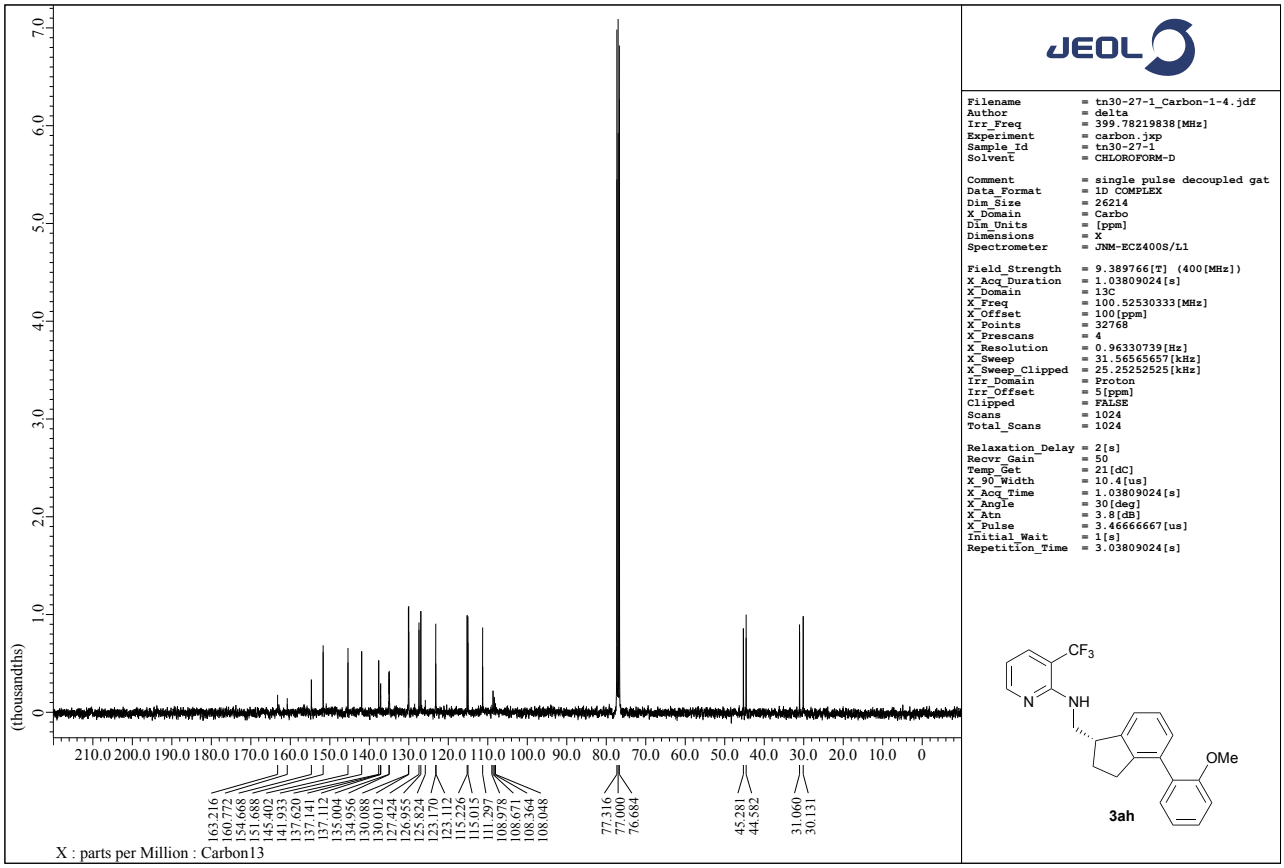


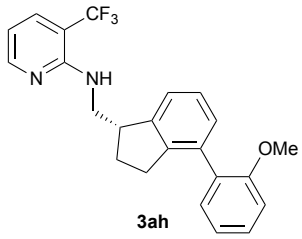

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 14.495 | 21878952 | 93.664 |
| 2 | 17.457 | 1479946 | 6.336 |



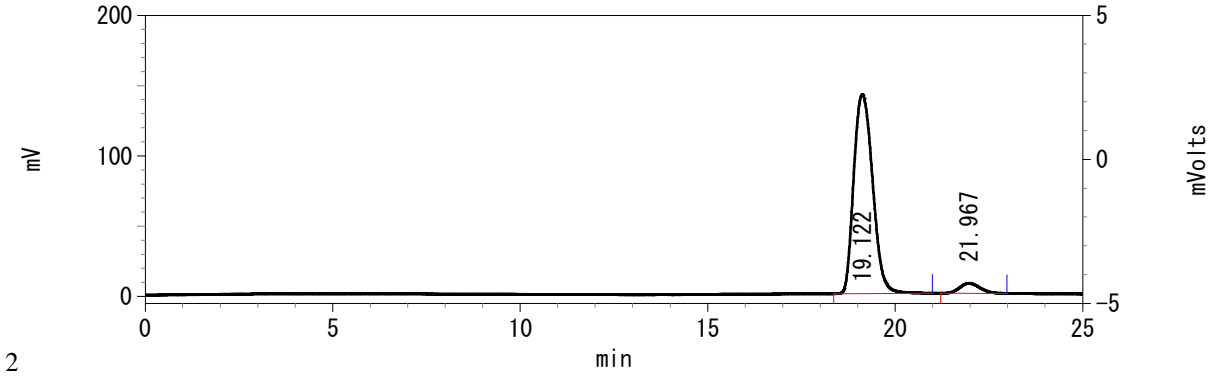

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 14.610 | 8688604 | 50.405 |
| 2 | 17.598 | 8548932 | 49.595 |


abundance
X : parts per Million : Proton
Filename = 3ah proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-27-2
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 = 56
Temp_Get = 20.6[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]
CF3
N
NH
OMe
3ah

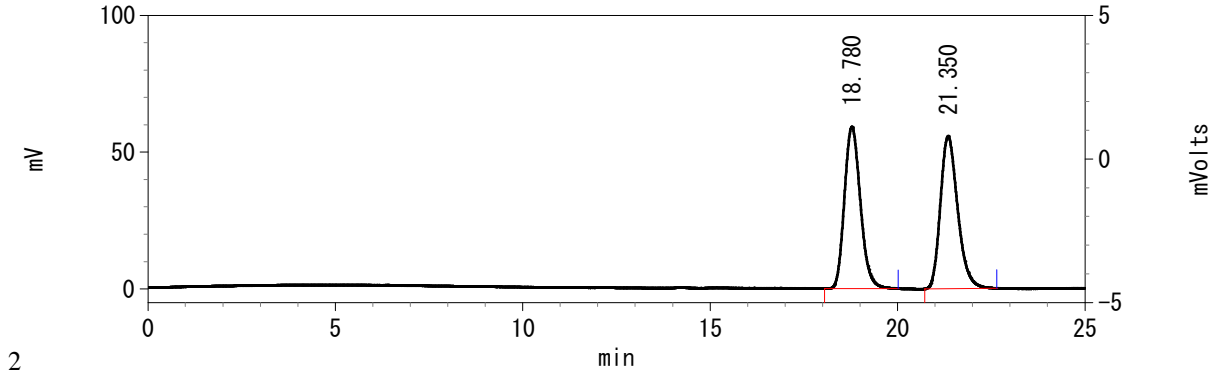

(thousandths)
X : parts per Million : Carbon13
Filename = tn30-27-1_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn30-27-1
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [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 = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21[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
OMe
3ah



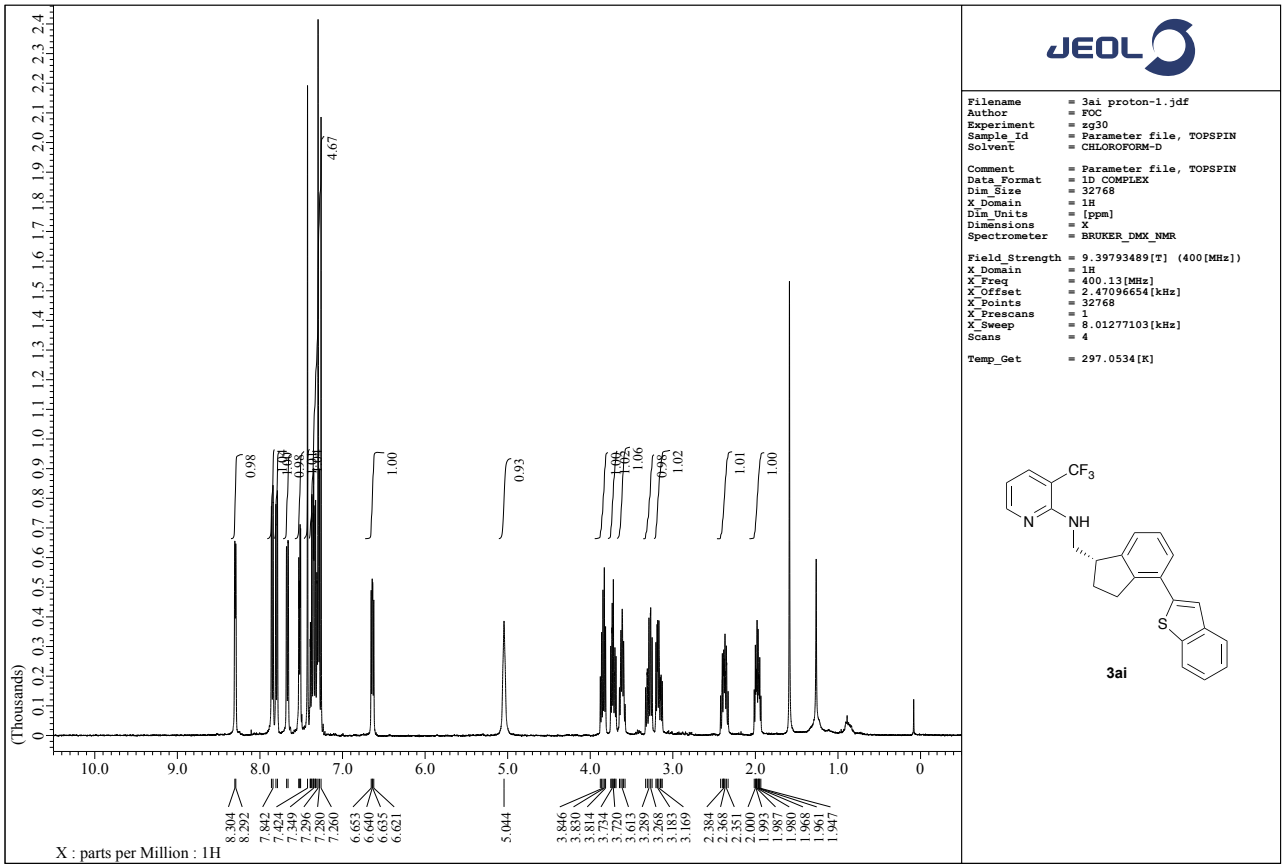

3ah

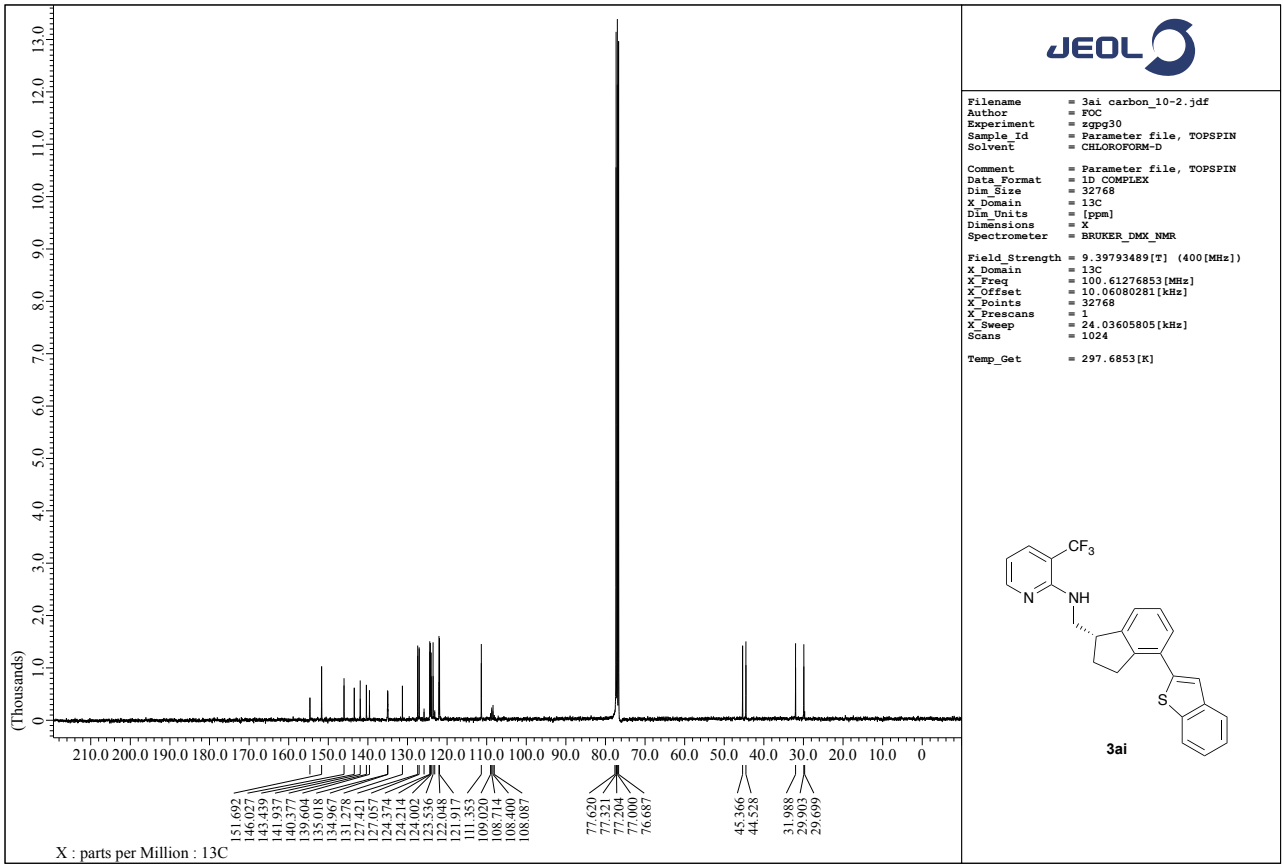


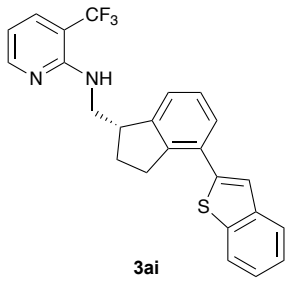

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 19.122 | 4972653 | 94.881 |
| 2 | 21.967 | 268258 | 5.119 |



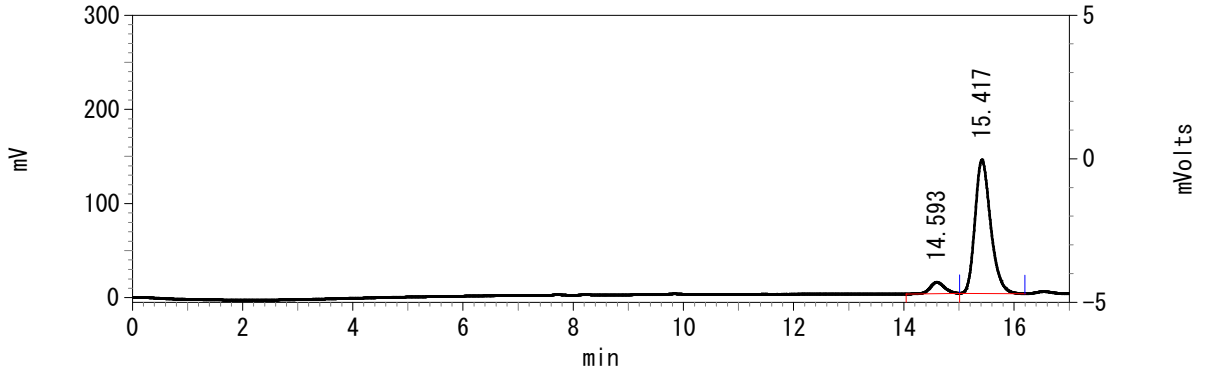

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 18.780 | 1756323 | 49.507 |
| 2 | 21.350 | 1791293 | 50.493 |


Filename = 3ai proton-1.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
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 = 297.0534[K]
(Thousands)
X : parts per Million : 1H
3ai

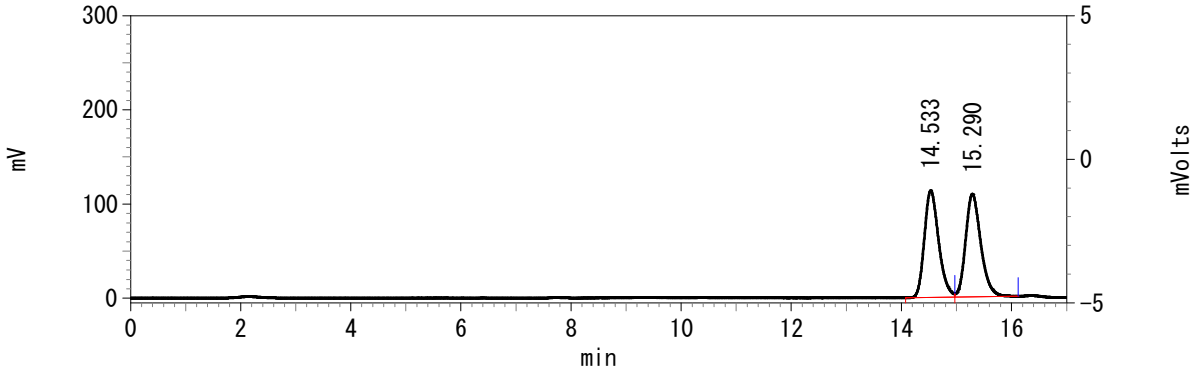

Filename = 3ai carbon_10-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 1024
Temp_Get = 297.6853[K]
(Thousands)
X : parts per Million : 13C
3ai



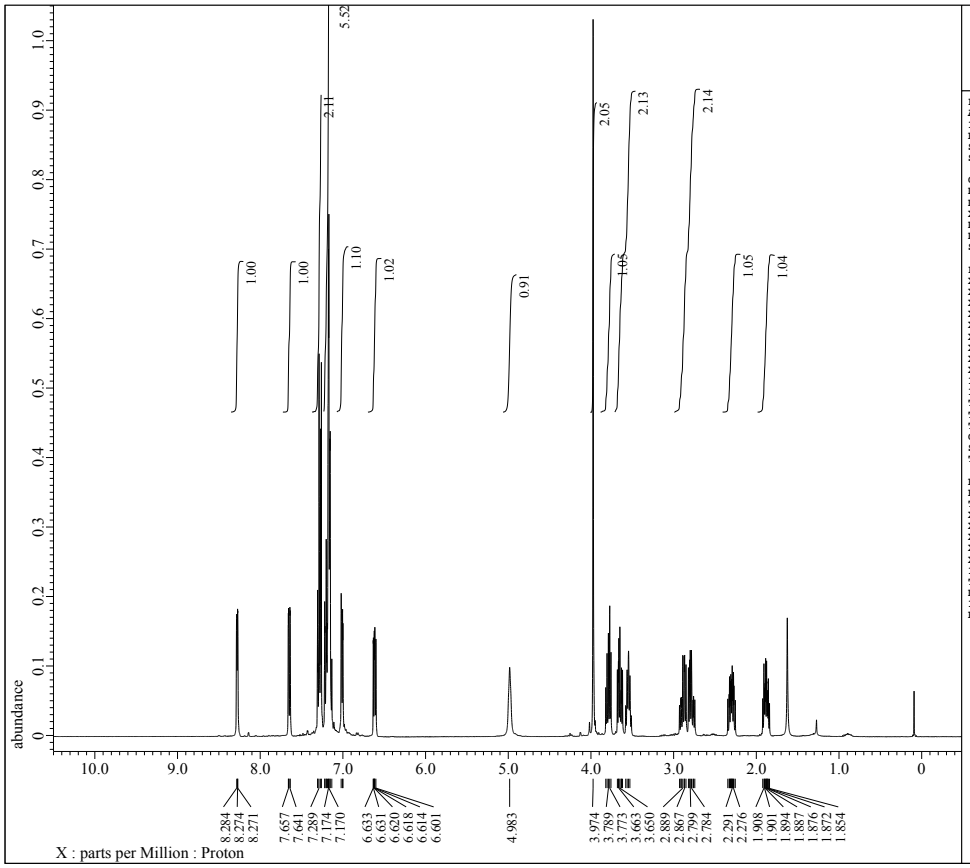

3ai

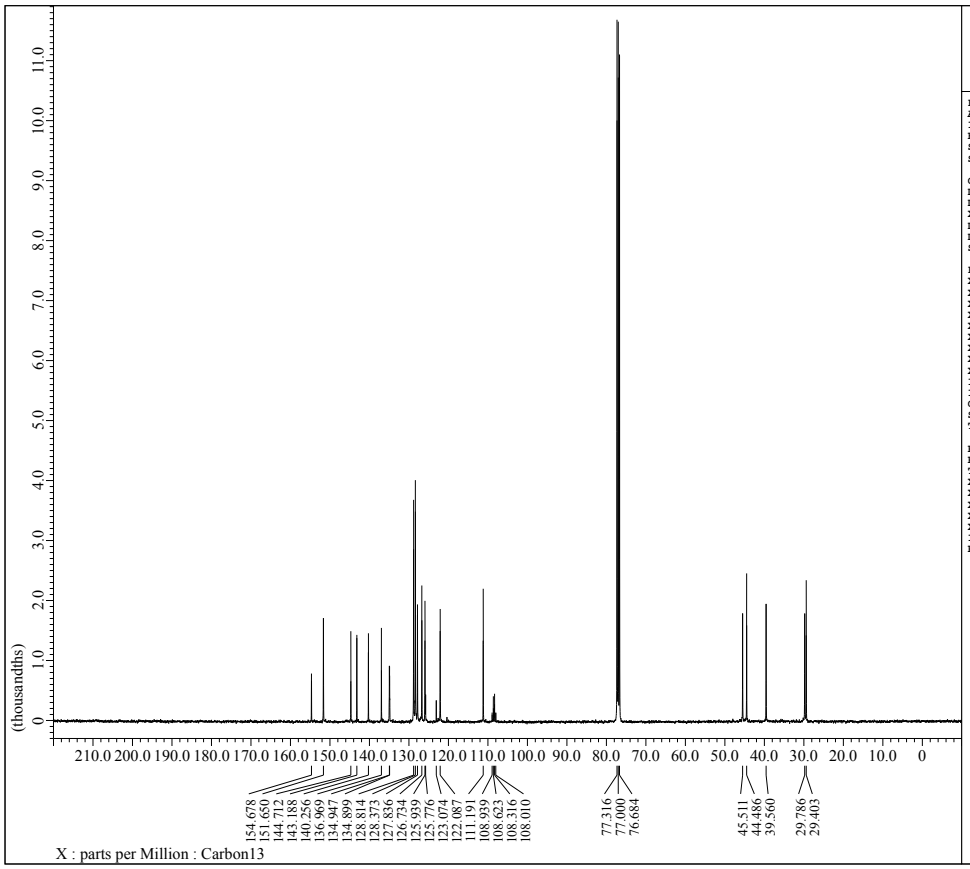


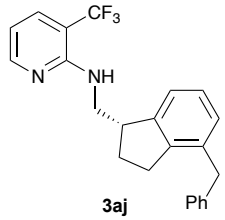

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 14.593 | 218379 | 7.244 |
| 2 | 15.417 | 2796317 | 92.756 |



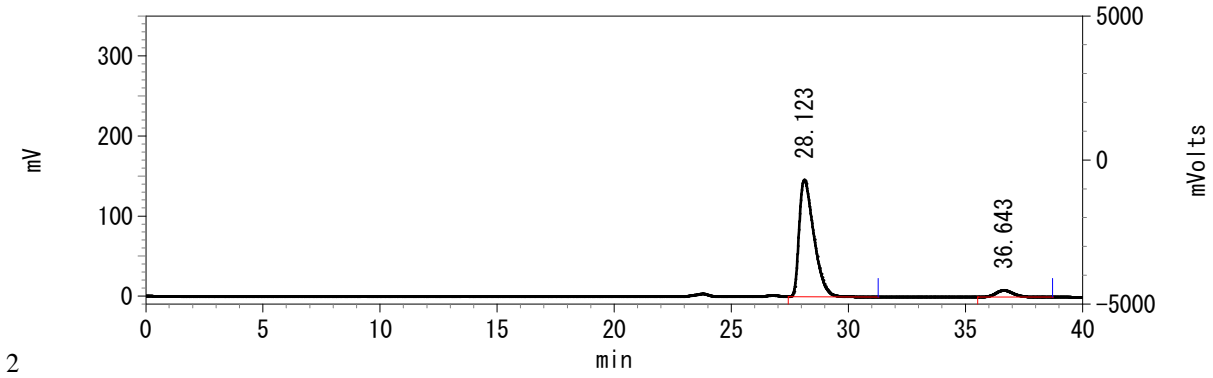

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 14.533 | 2044353 | 49.412 |
| 2 | 15.290 | 2093022 | 50.588 |


abundance
X : parts per Million : Proton
Filename = tn30-50_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-50
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 = 20.8[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
NH
N
3aj
Ph

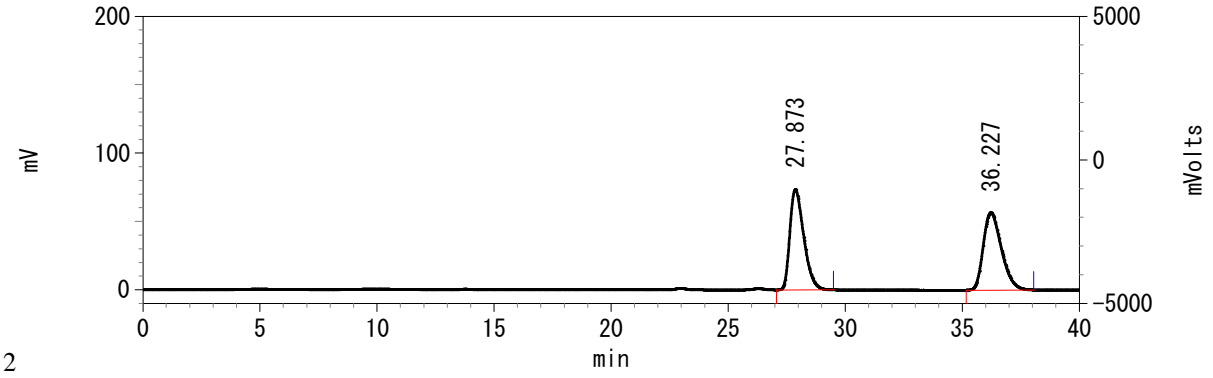

(thousandths)
X : parts per Million : Carbon13
Filename = tn30-50_Carbon-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn30-50
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
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 = TRUE
Scans = 7000
Total_Scans = 7000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.8[dC]
X_90_Width = 10.55[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.51666667[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
CF3
NH
N
3aj
Ph



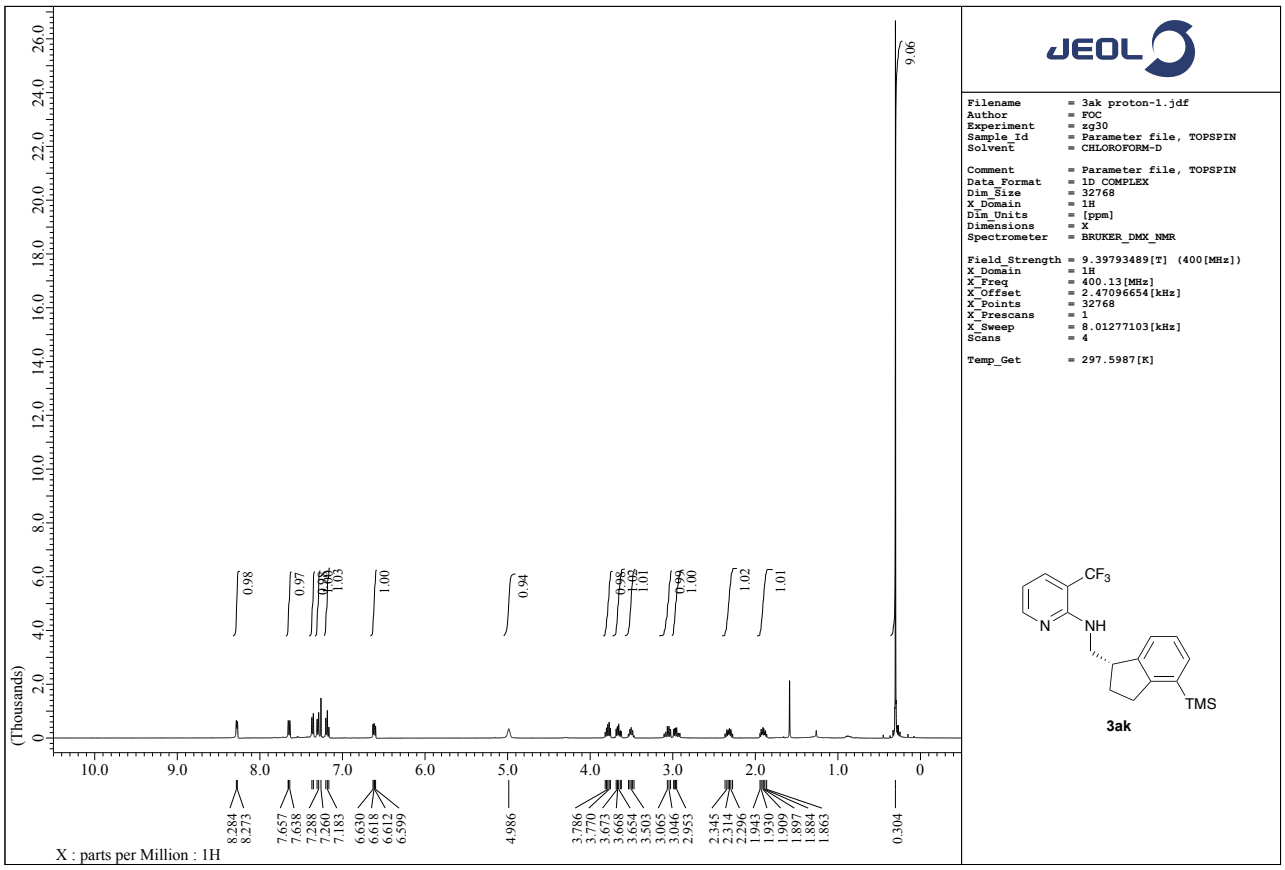

**3aj**

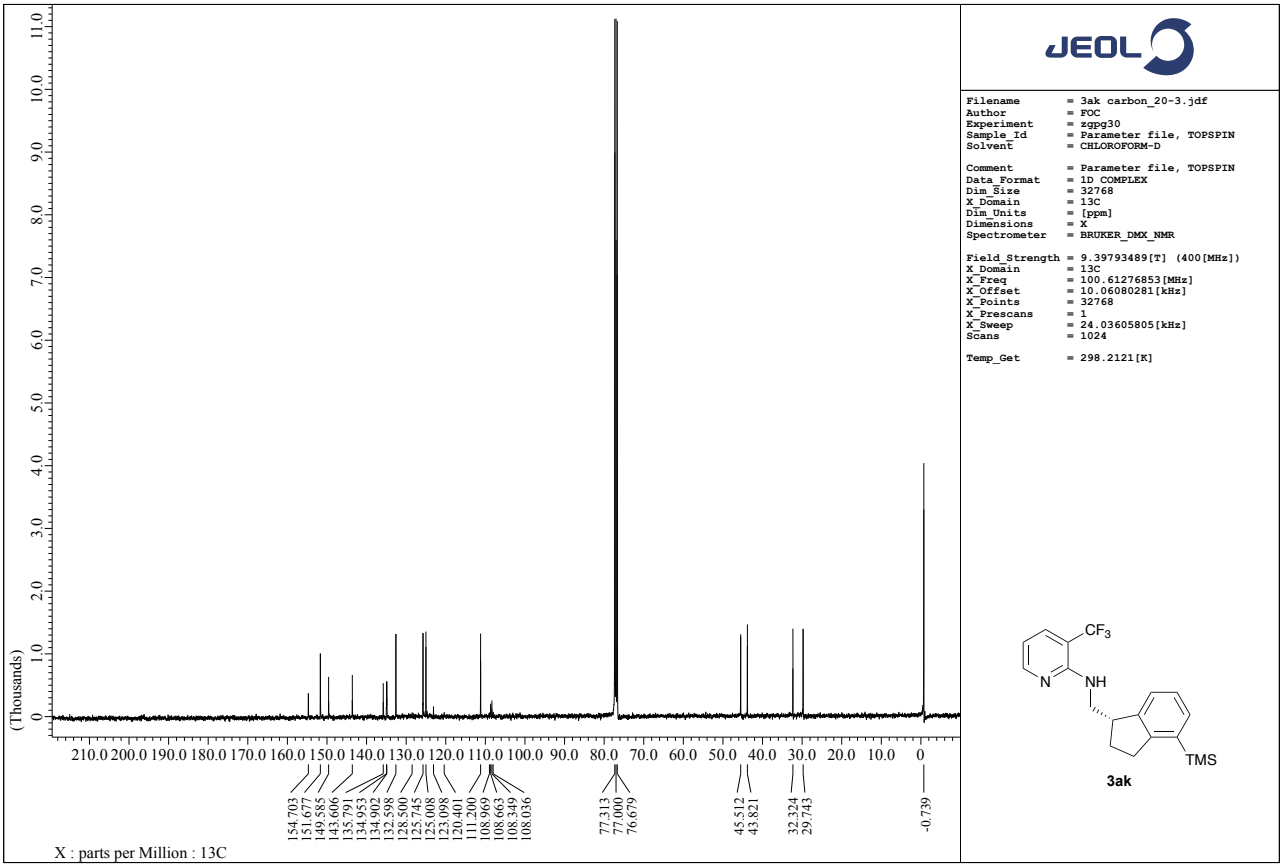


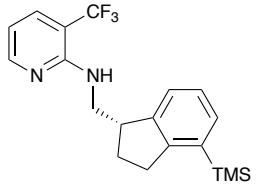

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 28.123 | 6370610 | 93.541 |
| 2 | 36.643 | 439901 | 6.459 |



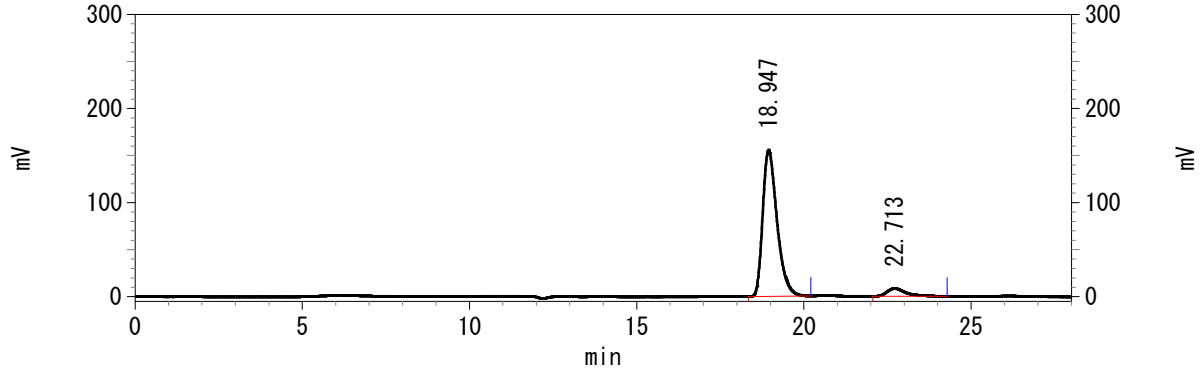

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 27.873 | 3032972 | 49.835 |
| 2 | 36.227 | 3053022 | 50.165 |


JEOL
Filename = 3ak proton-1.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
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 = 297.5987[K]
CF3
N
NH
TMS
3ak
(Thousands)
X : parts per Million : 1H

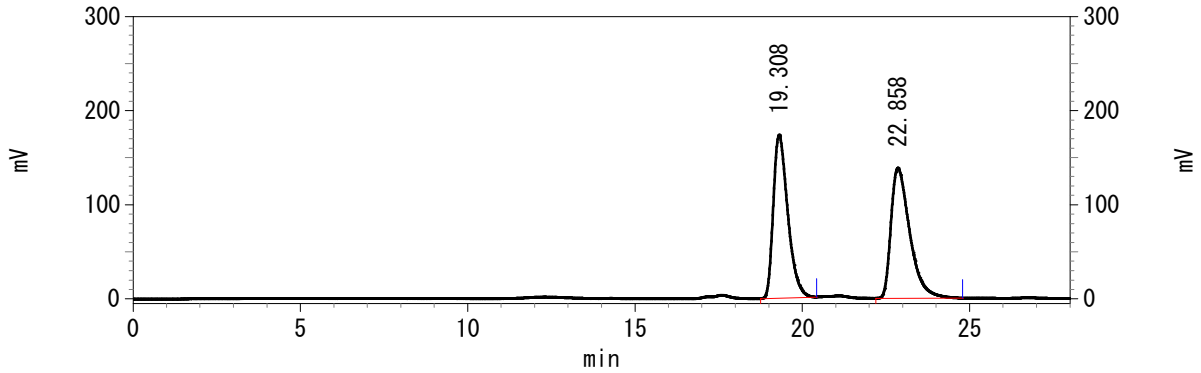

JEOL
Filename = 3ak carbon_20-3.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 1024
Temp_Get = 298.2121[K]
CF3
N
NH
TMS
3ak
(Thousands)
X : parts per Million : 13C



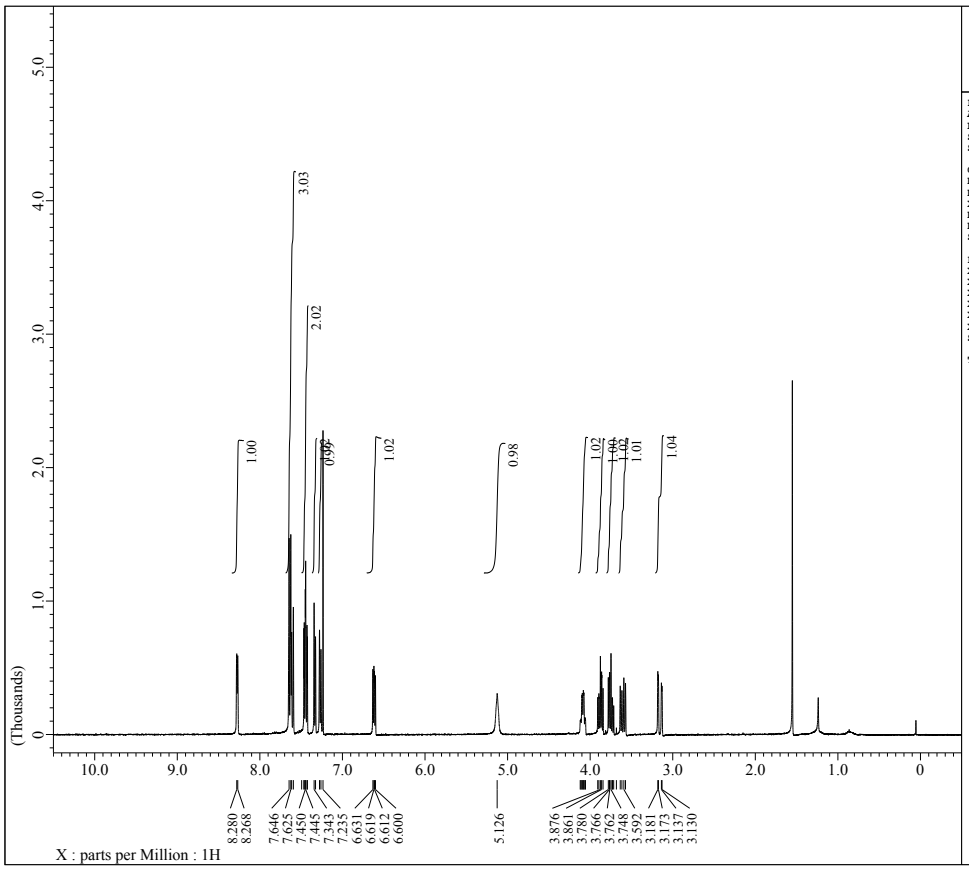

**3ak**




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 18.947 | 4615164 | 93.153 |
| 2 | 22.713 | 339212 | 6.847 |




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 19.308 | 5215635 | 49.147 |
| 2 | 22.858 | 5396636 | 50.853 |

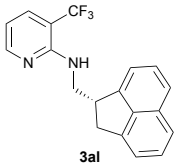



Filename = 3al proton-1.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D

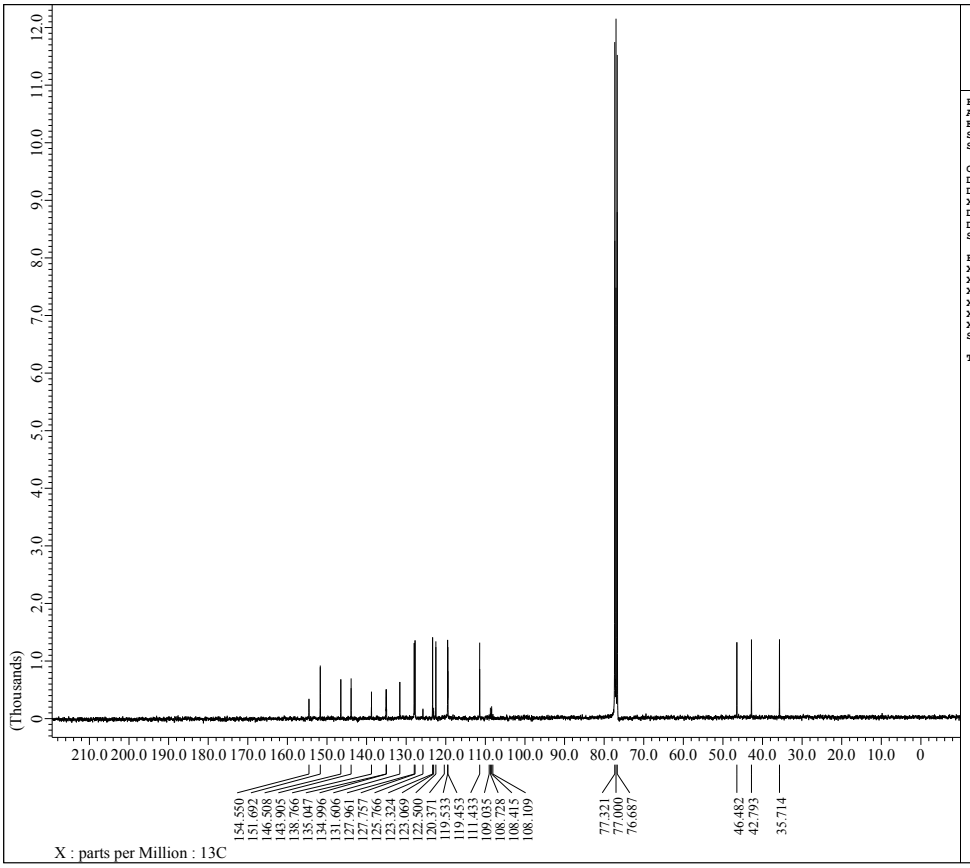
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR

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 = 298.1123[K]




3al

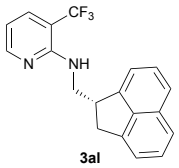



Filename = 3al carbon-1.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D

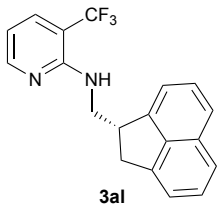
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR

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 = 1024

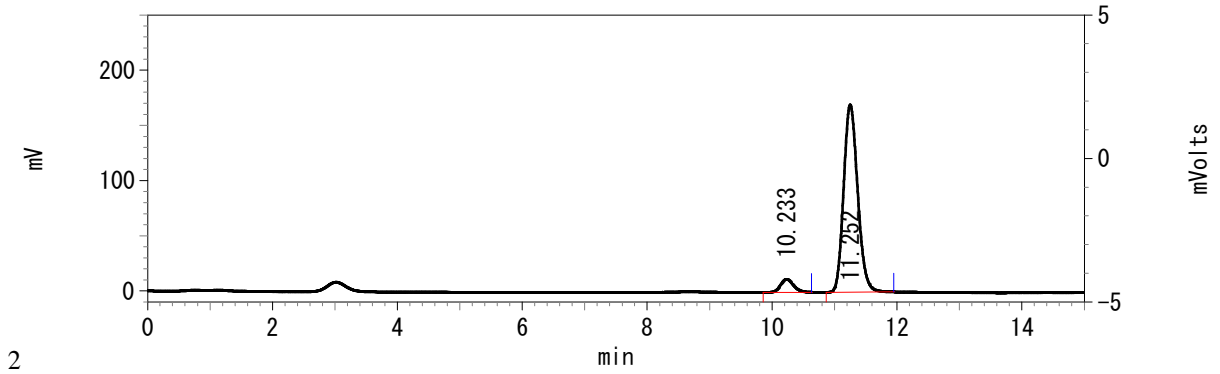
Temp_Get = 298.6896[K]



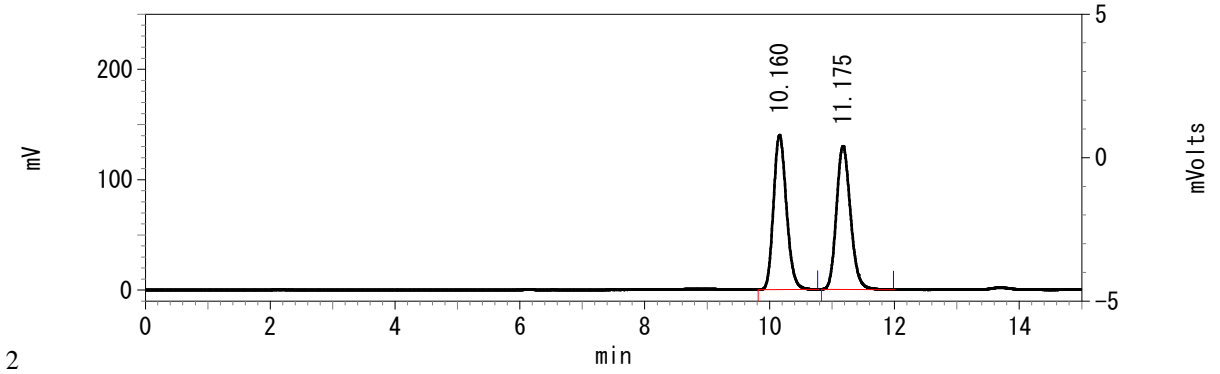

3al



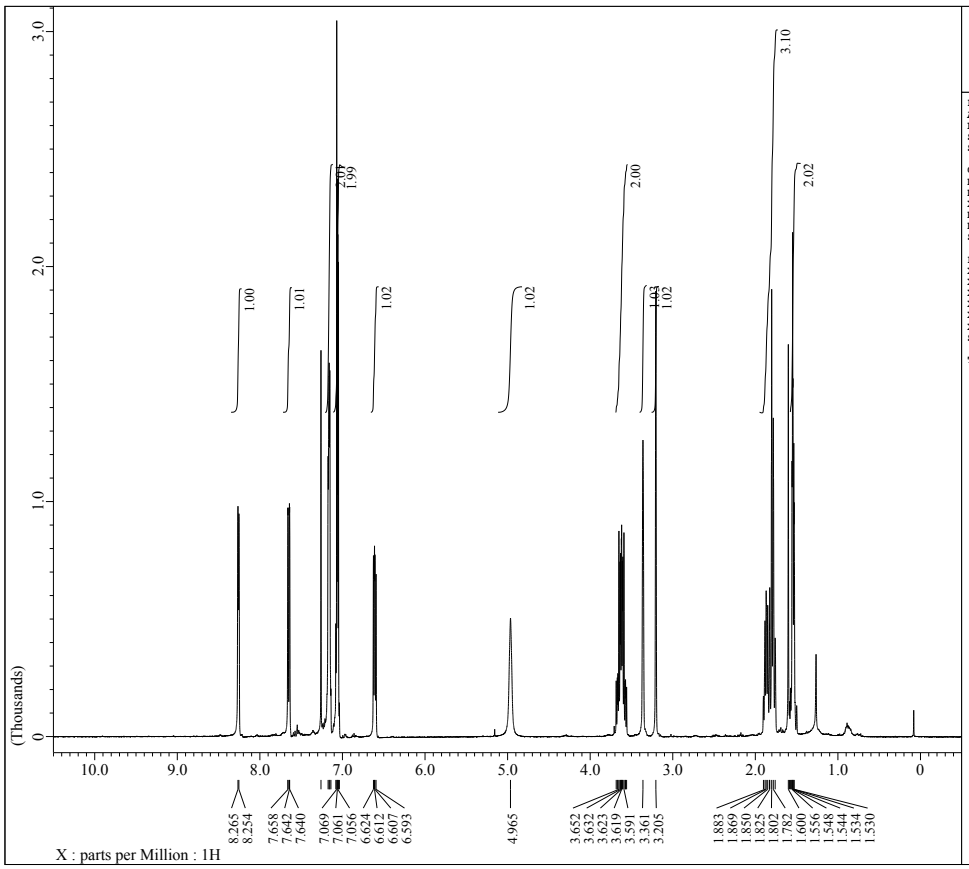

3al

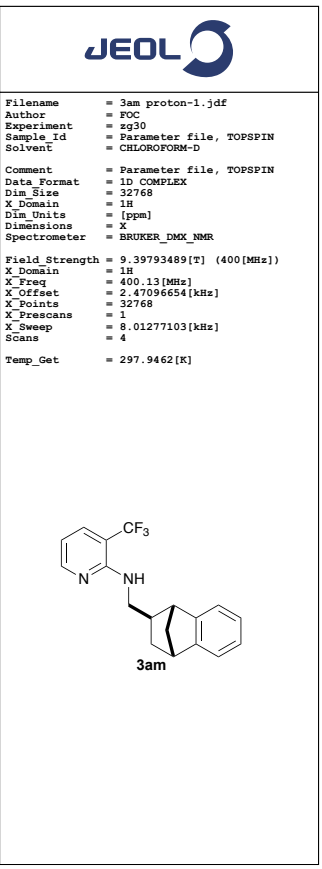


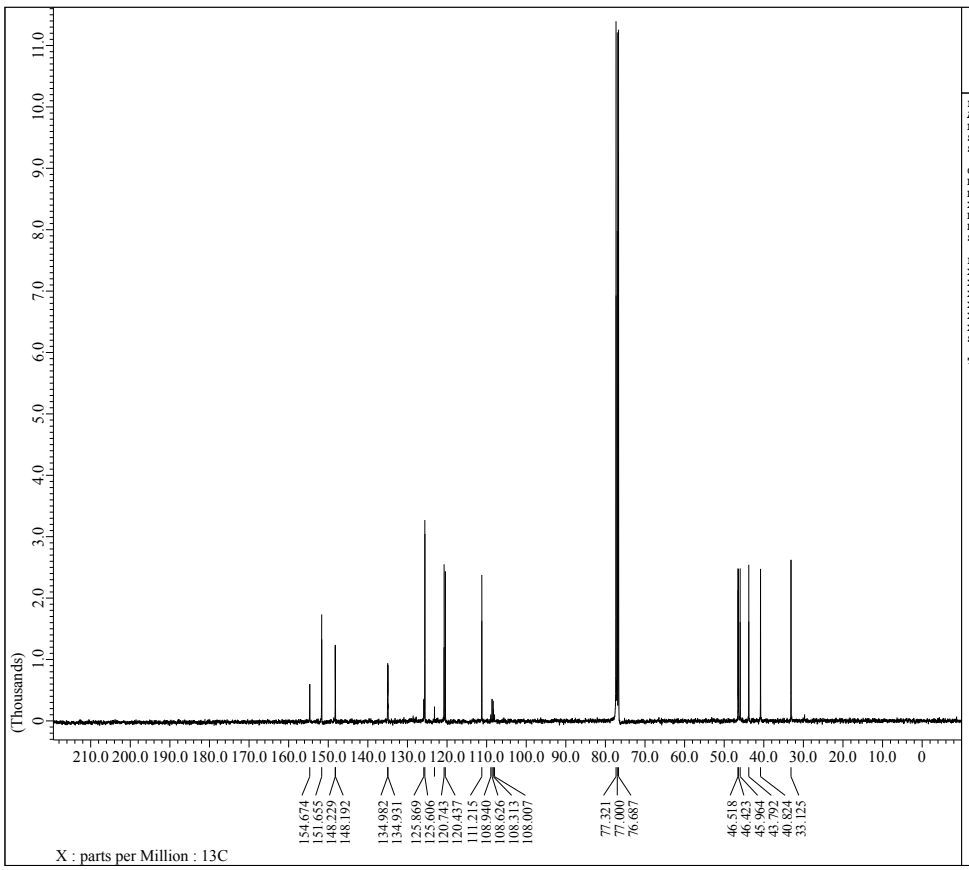

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 10.233 | 169518 | 5.999 |
| 2 | 11.252 | 2656101 | 94.001 |

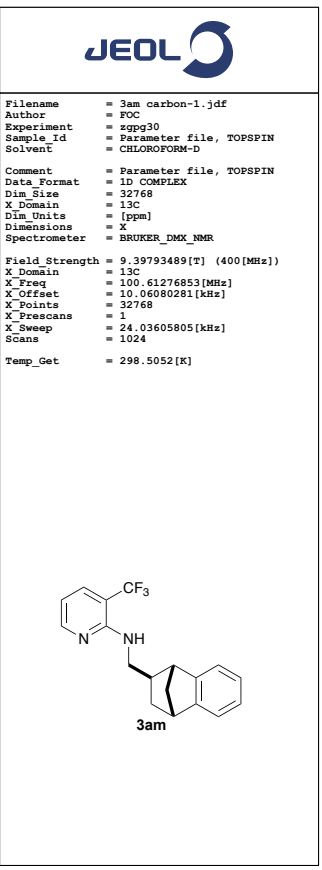


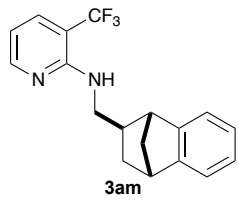

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 10.160 | 2016923 | 49.848 |
| 2 | 11.175 | 2029219 | 50.152 |


(Thousands)
X : parts per Million : 1H
8.265
8.254
7.658
7.642
7.640
7.069
7.061
7.056
6.624
6.612
6.607
6.593
4.965
3.652
3.632
3.613
3.610
3.591
3.361
3.205
1.883
1.869
1.850
1.825
1.802
1.782
1.600
1.556
1.548
1.544
1.534
1.530
1.00
1.01
1.02
1.02
2.07
1.99
2.00
1.03
1.02
3.10
2.02

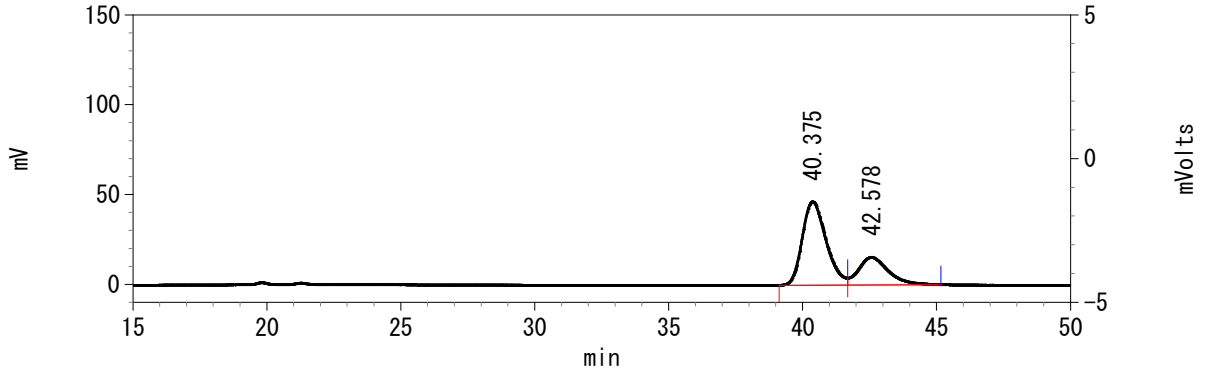

Filename = 3am proton-1.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 297.9462[K]
CF3
N
NH
3am


(Thousands)
X : parts per Million : 13C
154.674
151.655
148.229
148.192
134.982
134.931
125.869
125.606
120.743
120.437
111.215
108.940
108.626
108.313
108.007
77.321
77.000
76.687
46.518
46.423
45.964
43.792
40.824
33.125

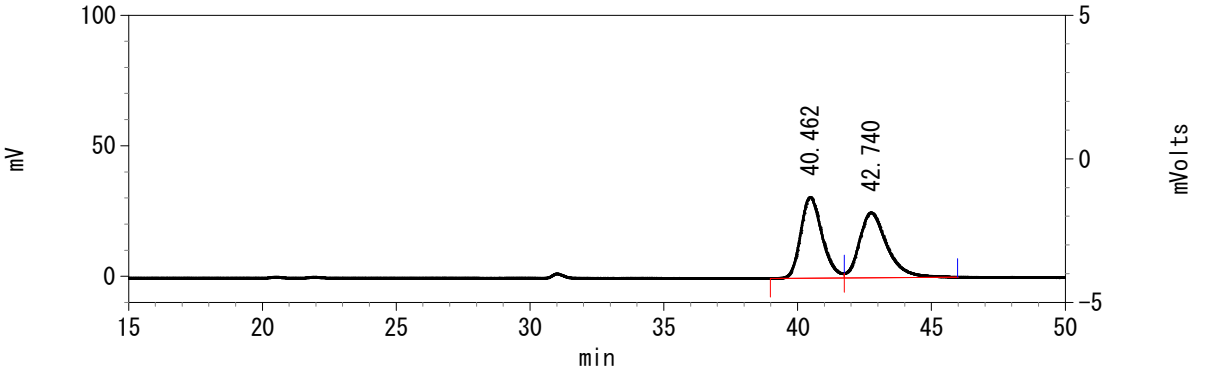

Filename = 3am carbon-1.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 1024
Temp_Get = 298.5052[K]
CF3
N
NH
3am



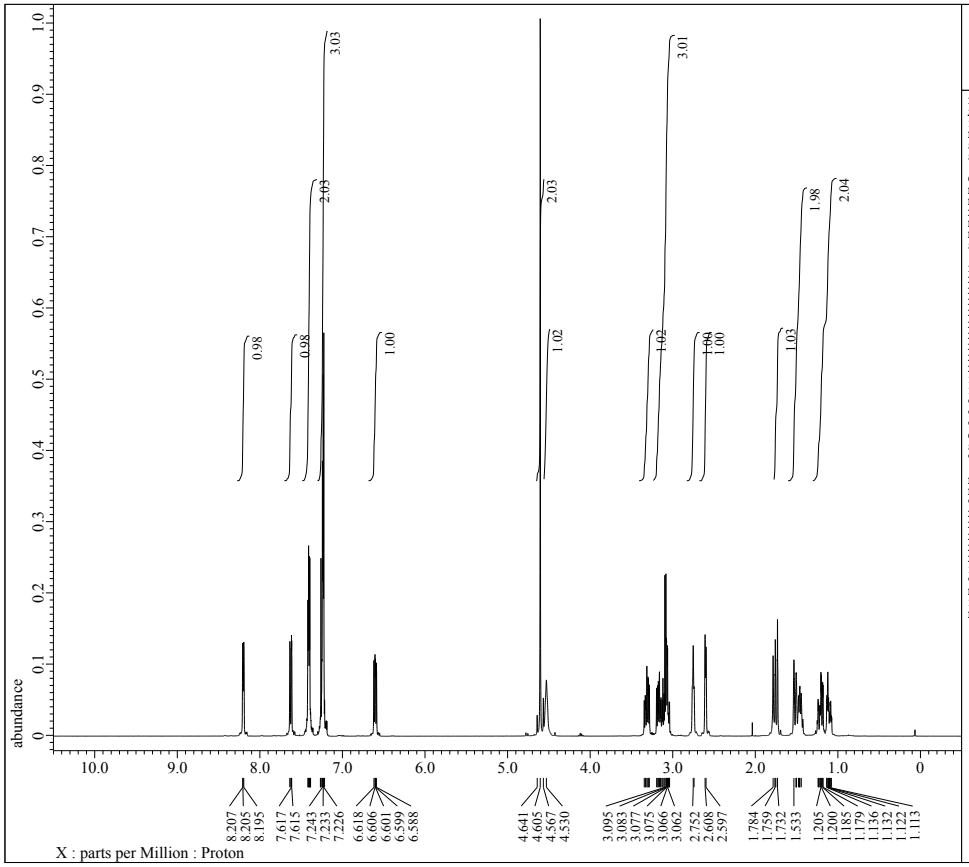

3am




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 40.375 | 2883637 | 70.889 |
| 2 | 42.578 | 1184176 | 29.111 |




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 40.462 | 1721311 | 49.191 |
| 2 | 42.740 | 1777953 | 50.809 |

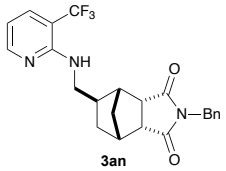



Filename = 3an proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-6-2
Solvent = CHLOROFORM-D

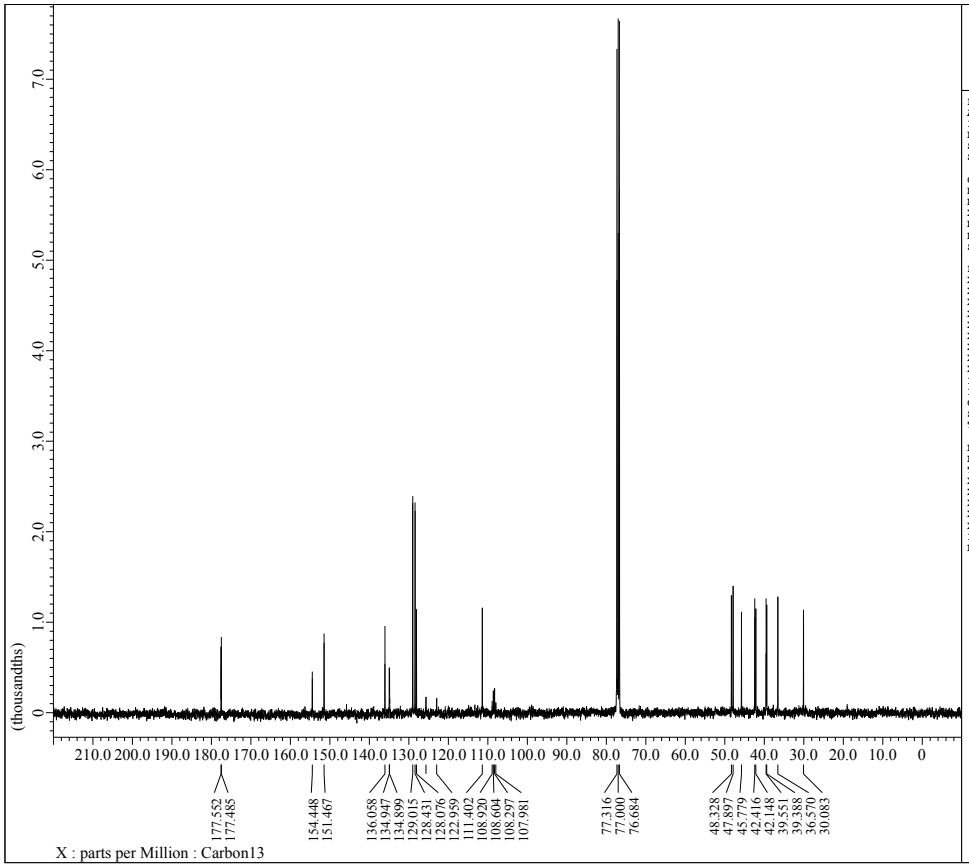
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 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]




3an

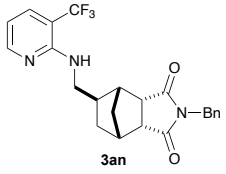



Filename = 3an carbon-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn30-6-2
Solvent = CHLOROFORM-D

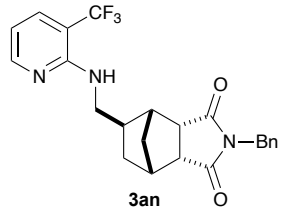
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 906
Total_Scans = 906

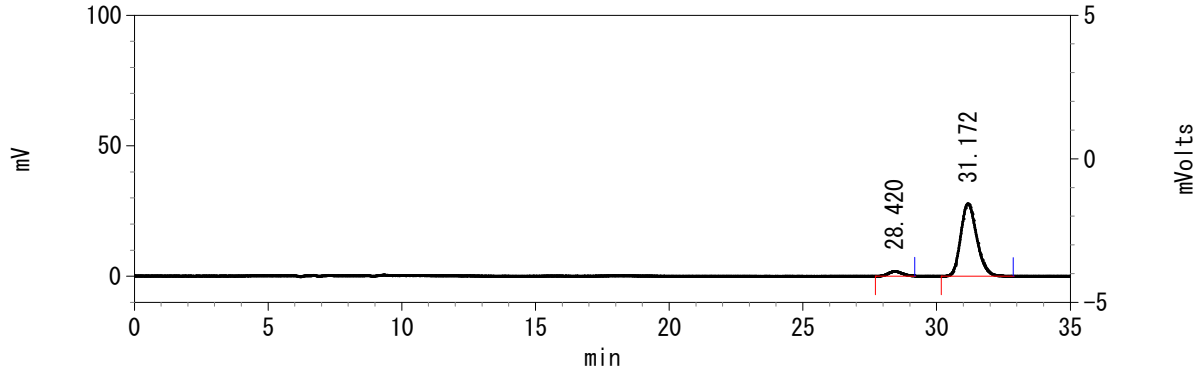
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.3[dC]
X_90_Width = 10.4[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.46666667[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]



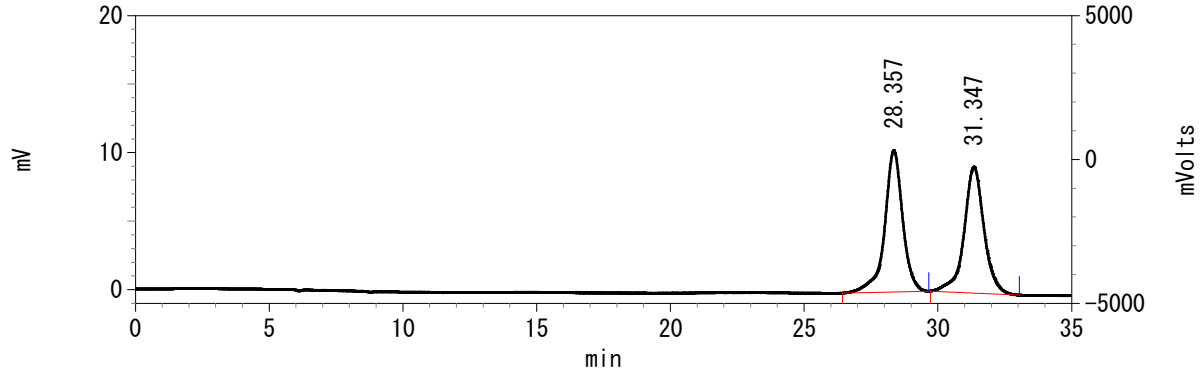

3an



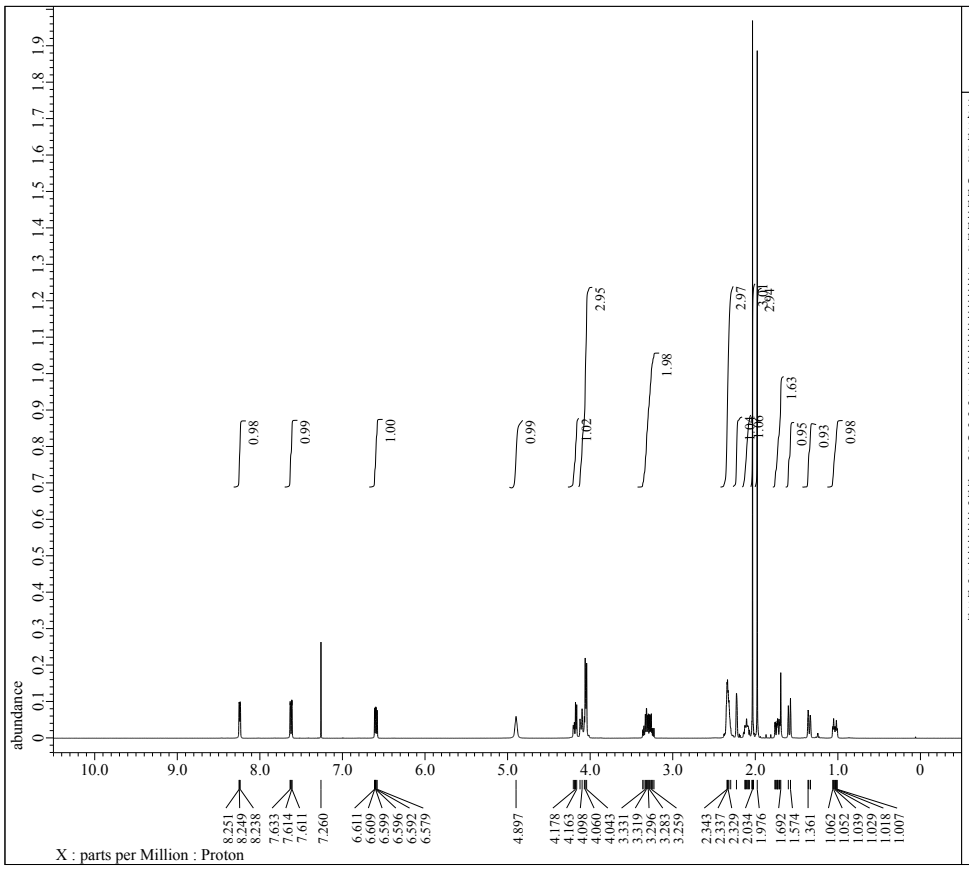

3an

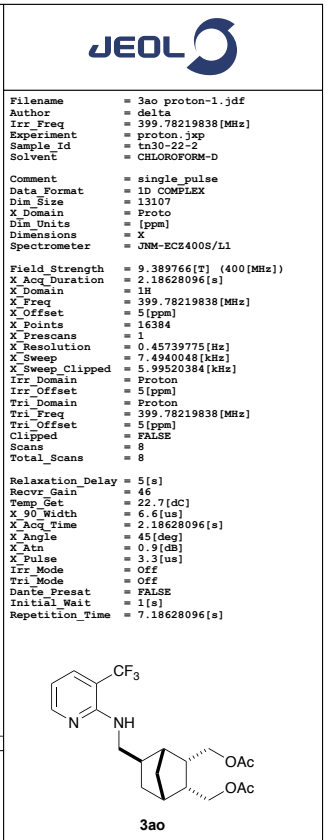


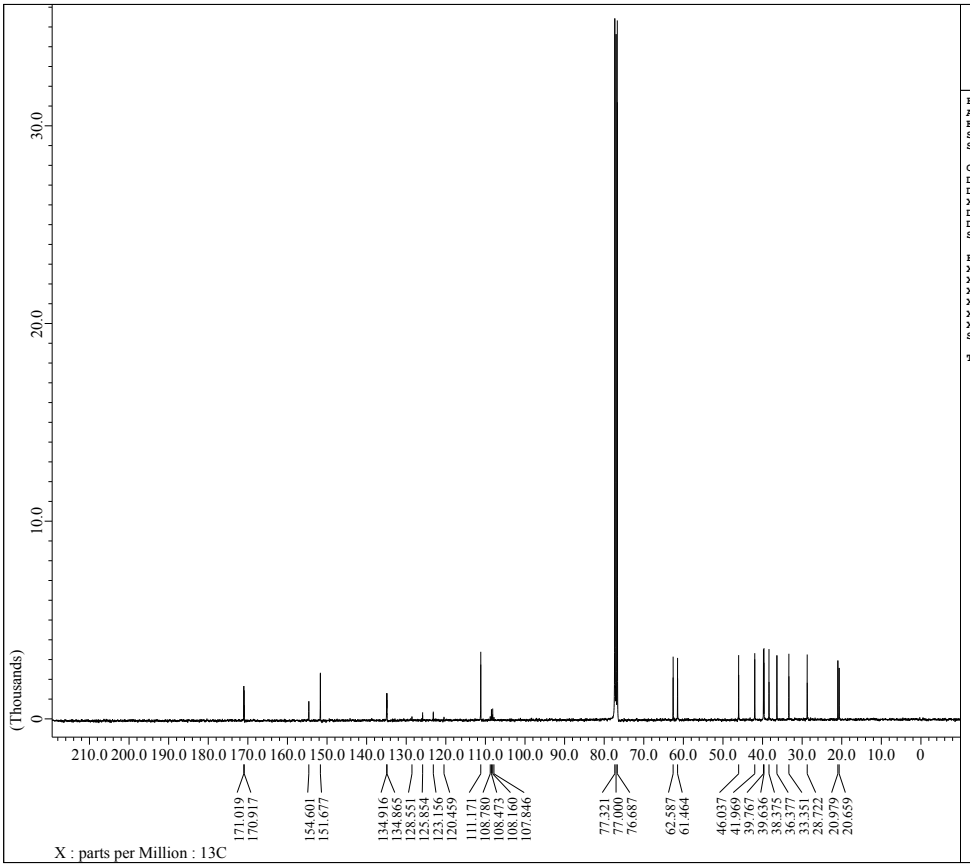

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 28.420 | 65417 | 5.343 |
| 2 | 31.172 | 1158845 | 94.657 |

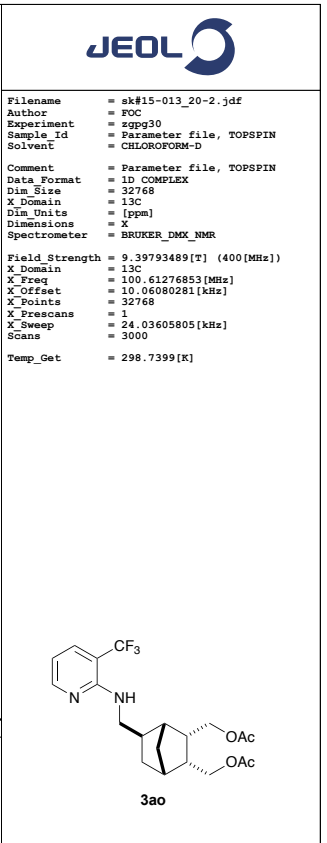


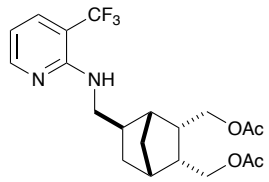

| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 28.357 | 485318 | 50.410 |
| 2 | 31.347 | 477421 | 49.590 |


abundance
0.98
0.99
1.00
0.99
1.02
2.95
1.98
2.97
2.94
1.63
0.95
0.93
0.98
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.251
8.249
8.238
7.633
7.614
7.611
7.260
6.611
6.609
6.599
6.596
6.592
6.579
4.897
4.178
4.163
4.098
4.060
4.043
3.331
3.319
3.296
3.283
3.259
2.343
2.337
2.329
2.034
1.976
1.692
1.574
1.361
1.062
1.052
1.039
1.029
1.018
1.007
X : parts per Million : Proton

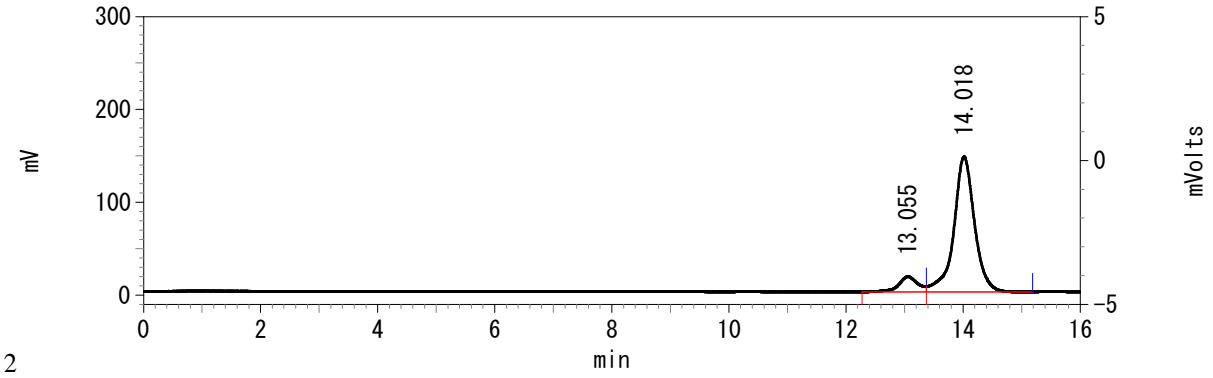

JEOL
Filename = 3ao proton-1.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-22-2
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 22.7[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]
CF3
N
NH
OAc
OAc
3ao


(Thousands)
30.0
20.0
10.0
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
171.019
170.917
154.601
151.677
134.916
134.865
128.551
125.854
123.156
120.459
111.171
108.780
108.473
108.160
107.846
77.321
77.000
76.687
62.587
61.464
46.037
41.969
39.767
39.636
38.375
36.377
33.351
28.722
20.979
20.659
X : parts per Million : 13C

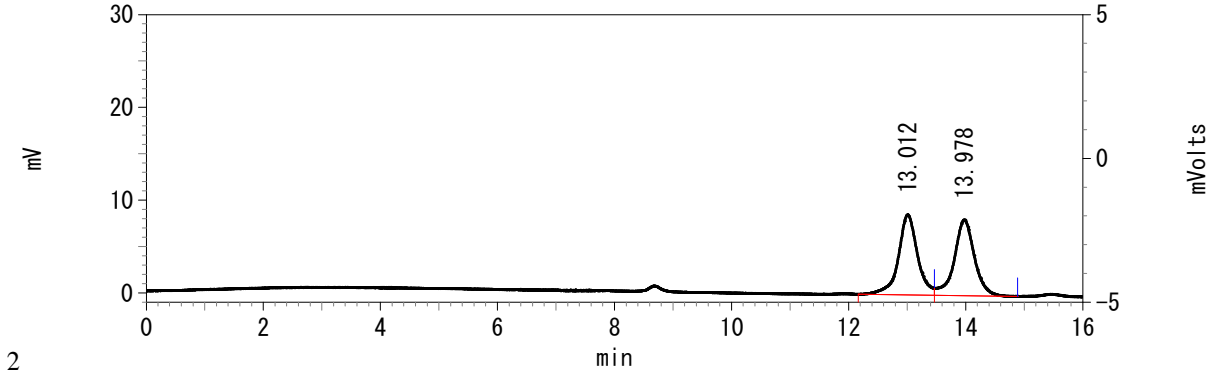

JEOL
Filename = sk#15-013_20-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 298.7399[K]
CF3
N
NH
OAc
OAc
3ao



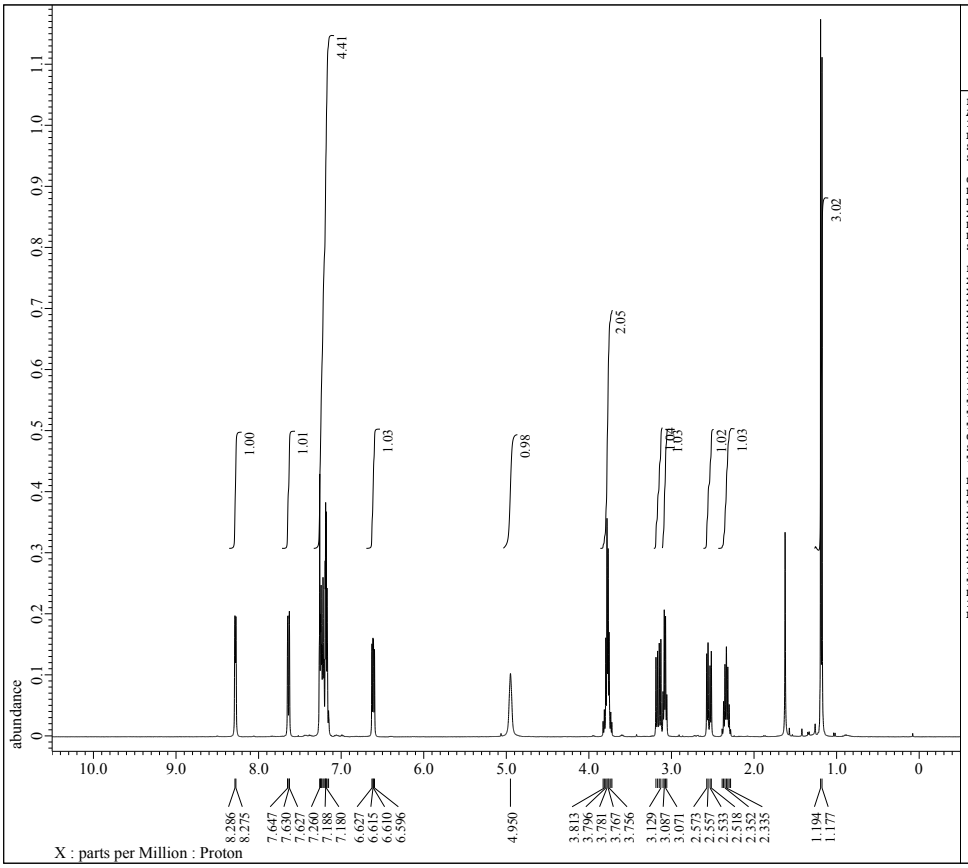

**3ao**




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 13.055 | 375946 | 9.532 |
| 2 | 14.018 | 3568092 | 90.468 |




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 13.012 | 196266 | 50.023 |
| 2 | 13.978 | 196084 | 49.977 |

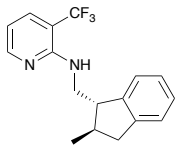



Filename = tn30-59-2_Proton-2-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn30-59-2
Solvent = CHLOROFORM-D

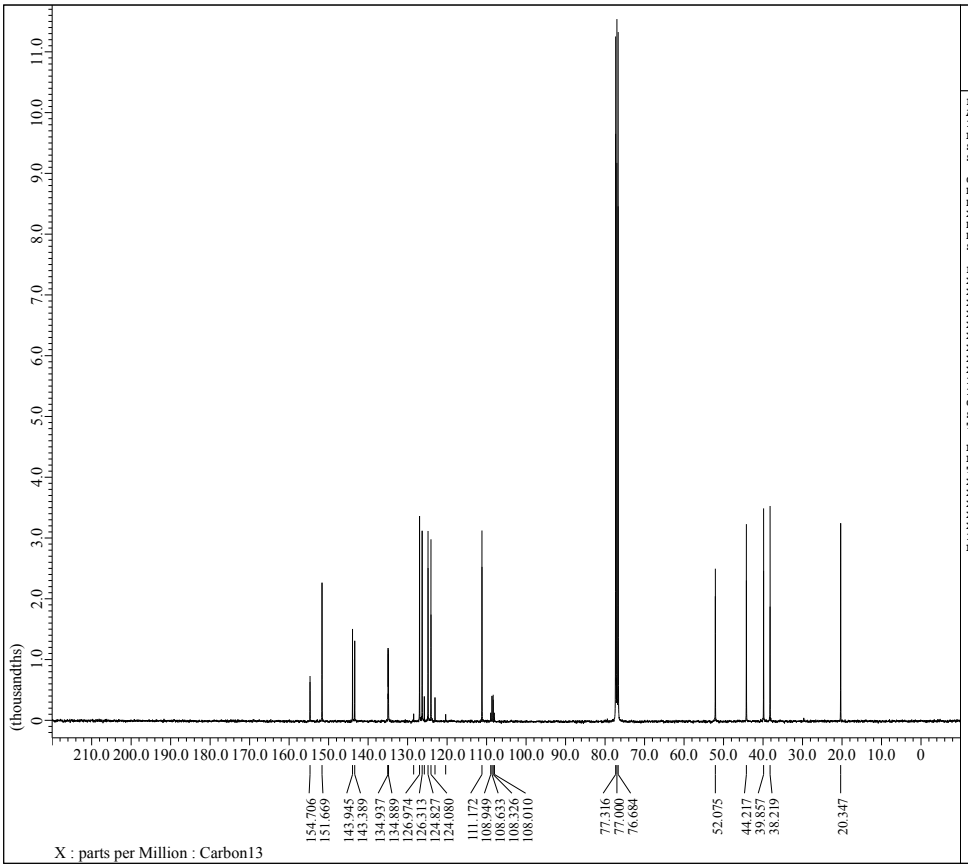
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 20.4[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]




**3ap**

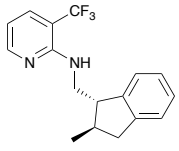



Filename = tn30-59-2_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn30-59-2
Solvent = CHLOROFORM-D

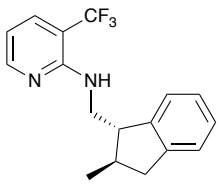
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 8000
Total_Scans = 8000

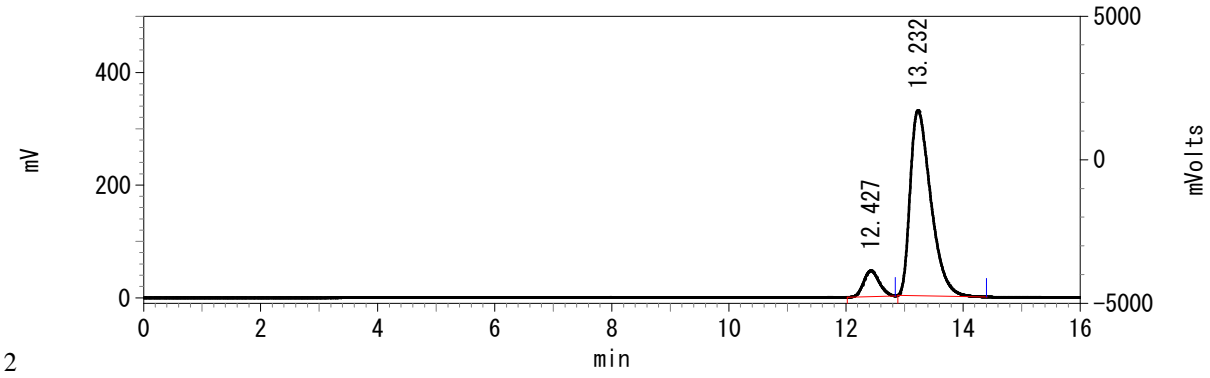
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.7[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]



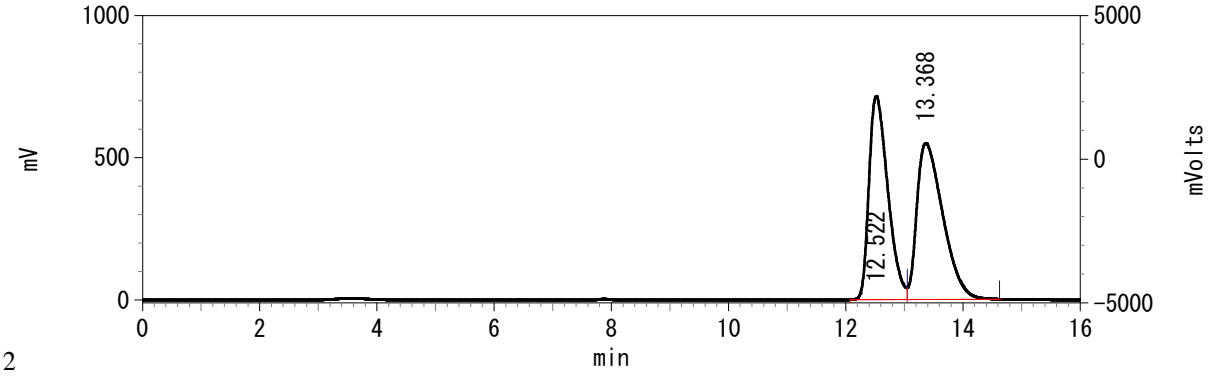

**3ap**



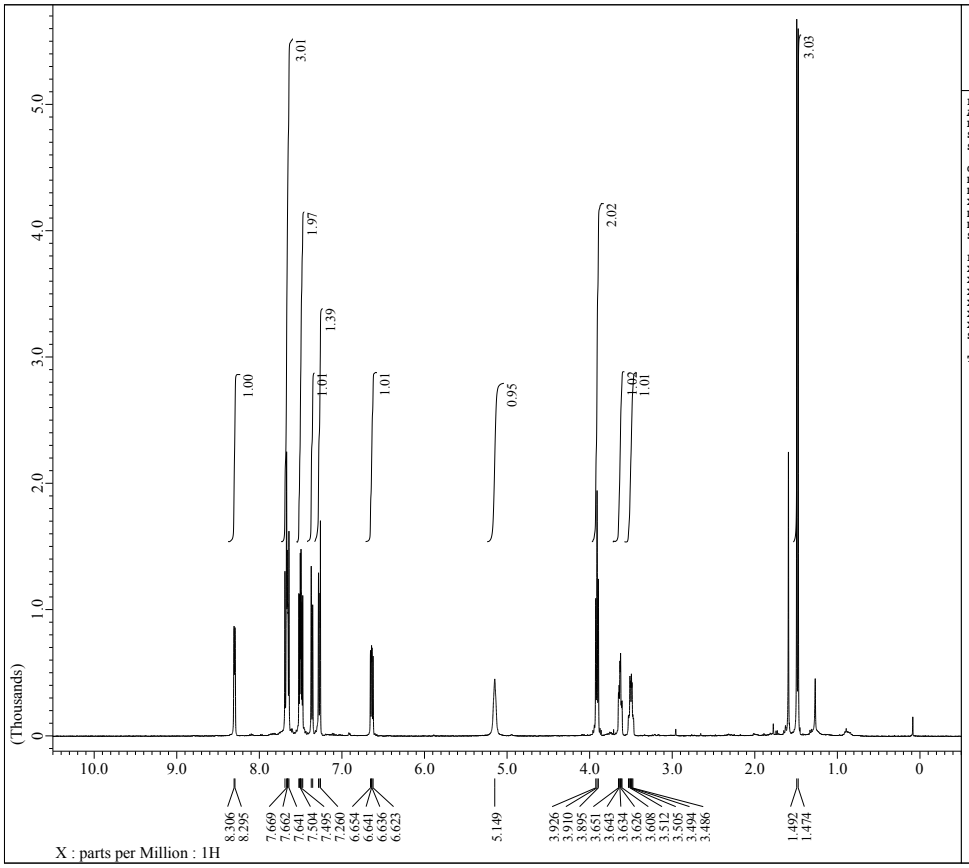

**3ap**




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 12.427 | 854320 | 9.579 |
| 2 | 13.232 | 8064246 | 90.421 |

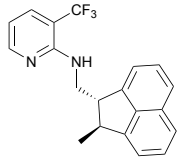



| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 12.522 | 16026614 | 48.571 |
| 2 | 13.368 | 16969863 | 51.429 |

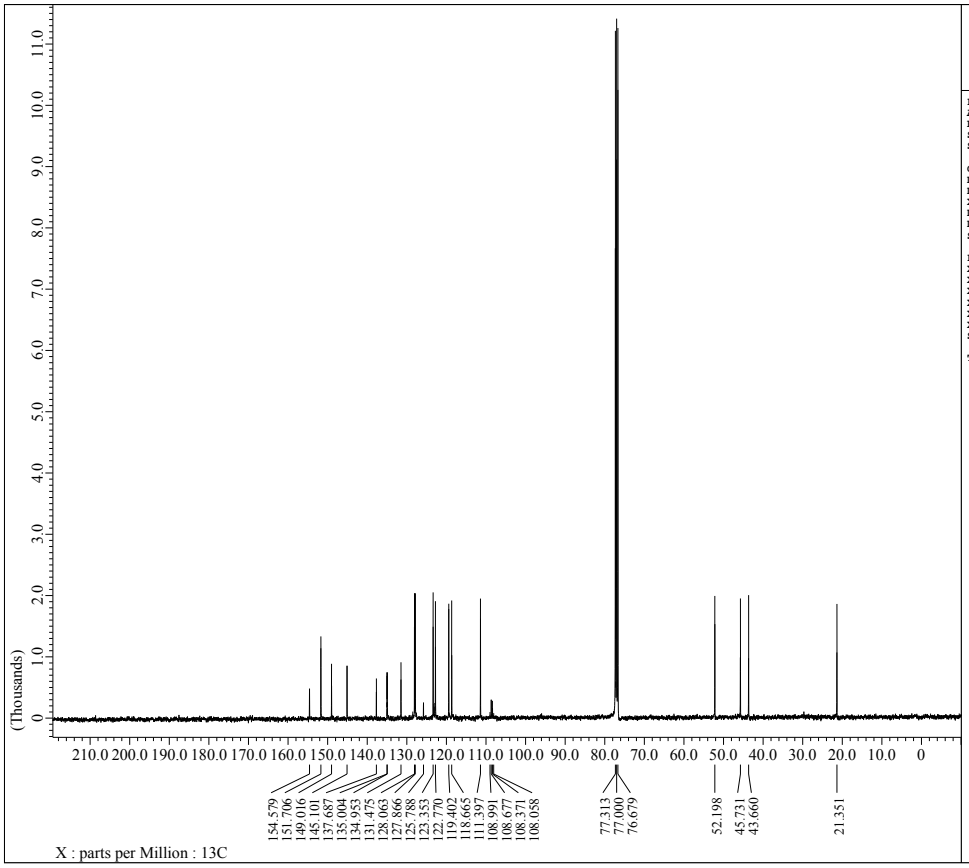



JEOL

```
Filename        = 3aq proton-1.jdf
Author          = FOC
Experiment      = zg30
Sample_Id       = Parameter file, TOPSPIN
Solvent         = CHLOROFORM-D

Comment         = Parameter file, TOPSPIN
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        = 297.9536[K]
```

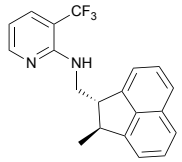



**3aq**

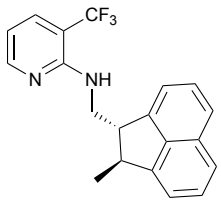



JEOL

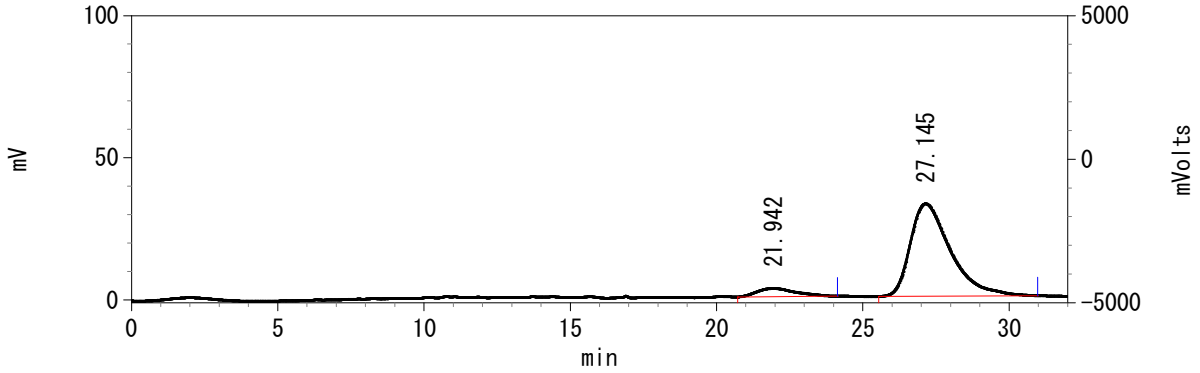
```
Filename        = 3aq carbon-1.jdf
Author          = FOC
Experiment      = zgpg30
Sample_Id       = Parameter file, TOPSPIN
Solvent         = CHLOROFORM-D

Comment         = Parameter file, TOPSPIN
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           = 1024

Temp_Get        = 298.5378[K]
```



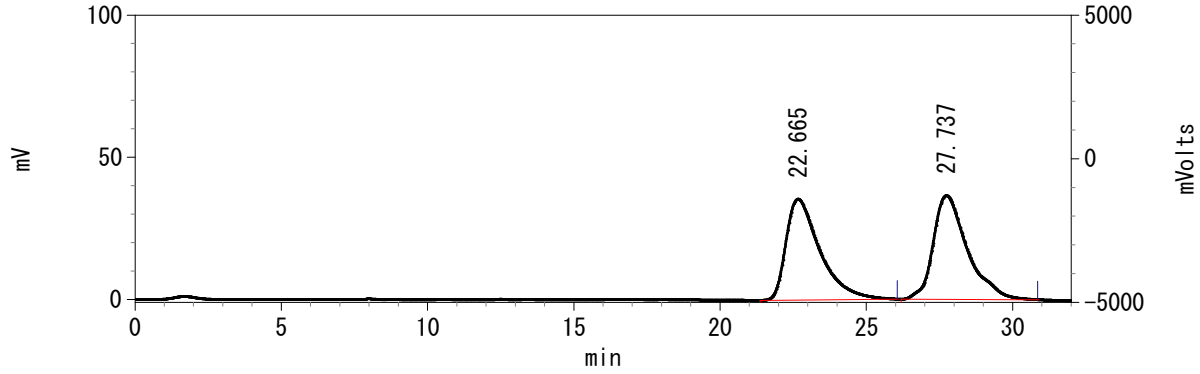

**3aq**



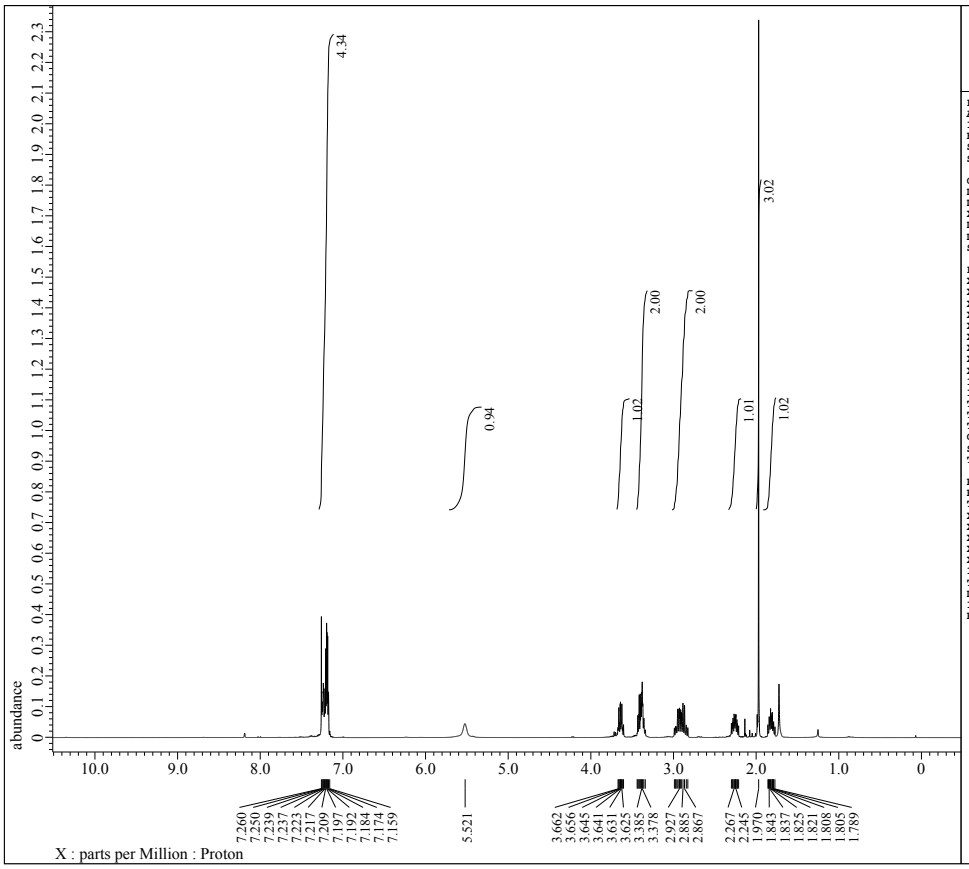

**3aq**




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 21.942 | 262644 | 7.821 |
| 2 | 27.145 | 3095656 | 92.179 |




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 22.665 | 2852721 | 49.076 |
| 2 | 27.737 | 2960116 | 50.924 |

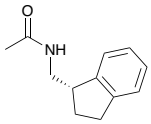



Filename = sk#17-003-2_Proton-1-2.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = sk#17-003-2
Solvent = CHLOROFORM-D

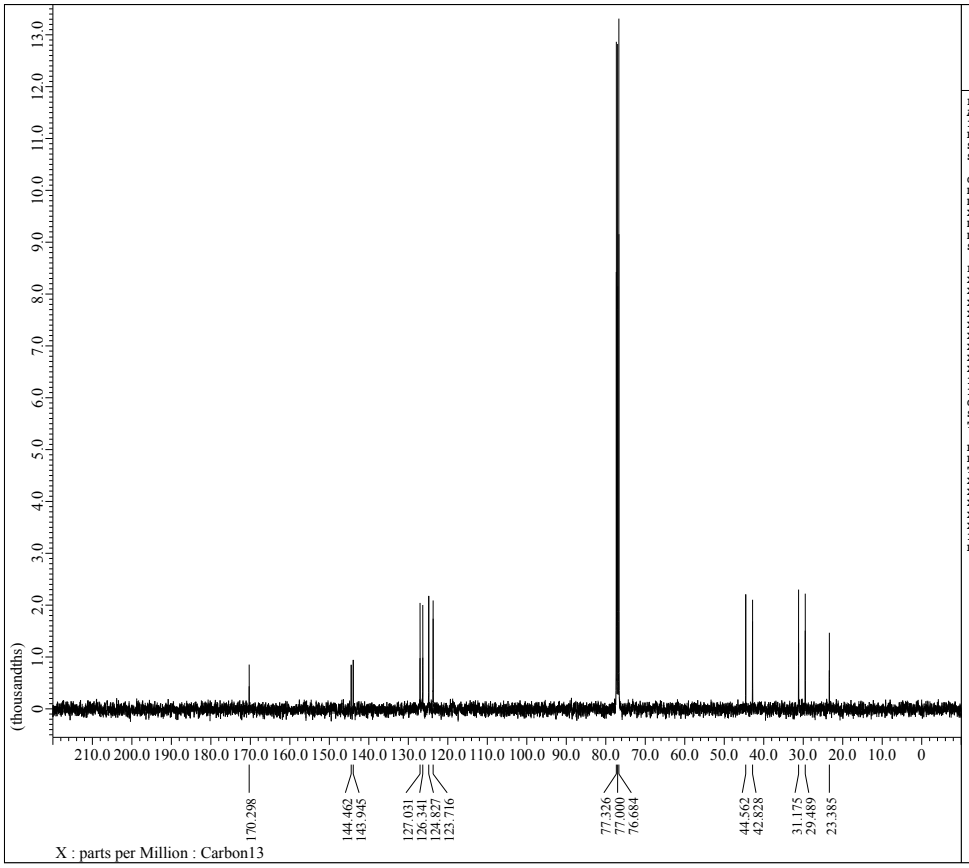
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 20.9[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]




**5**

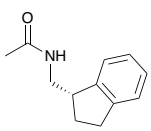



Filename = sk#17-003-2_Carbon-1-2.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = sk#17-003-2
Solvent = CHLOROFORM-D

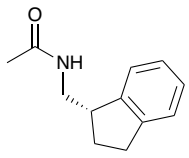
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 175
Total_Scans = 175

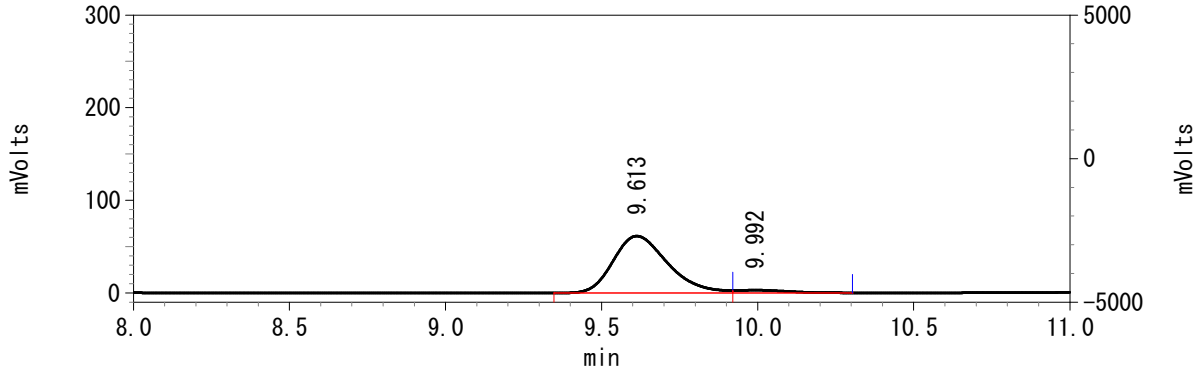
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.8[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]



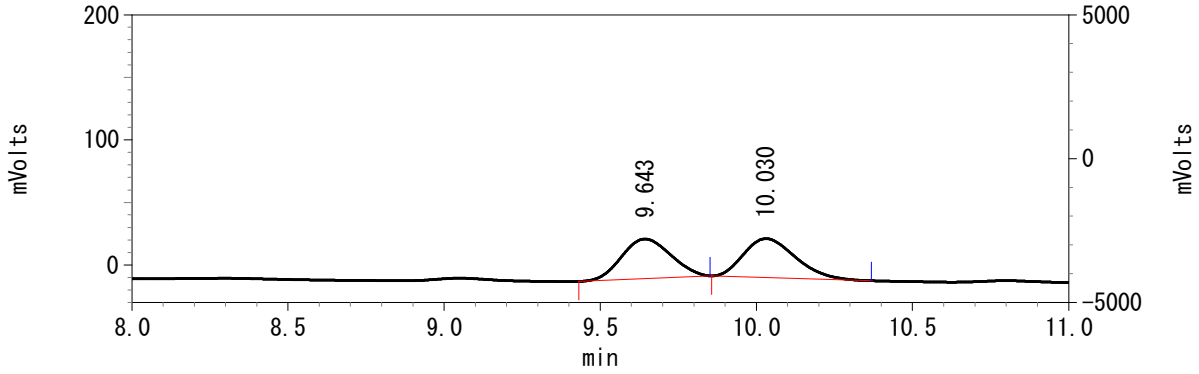

**5**



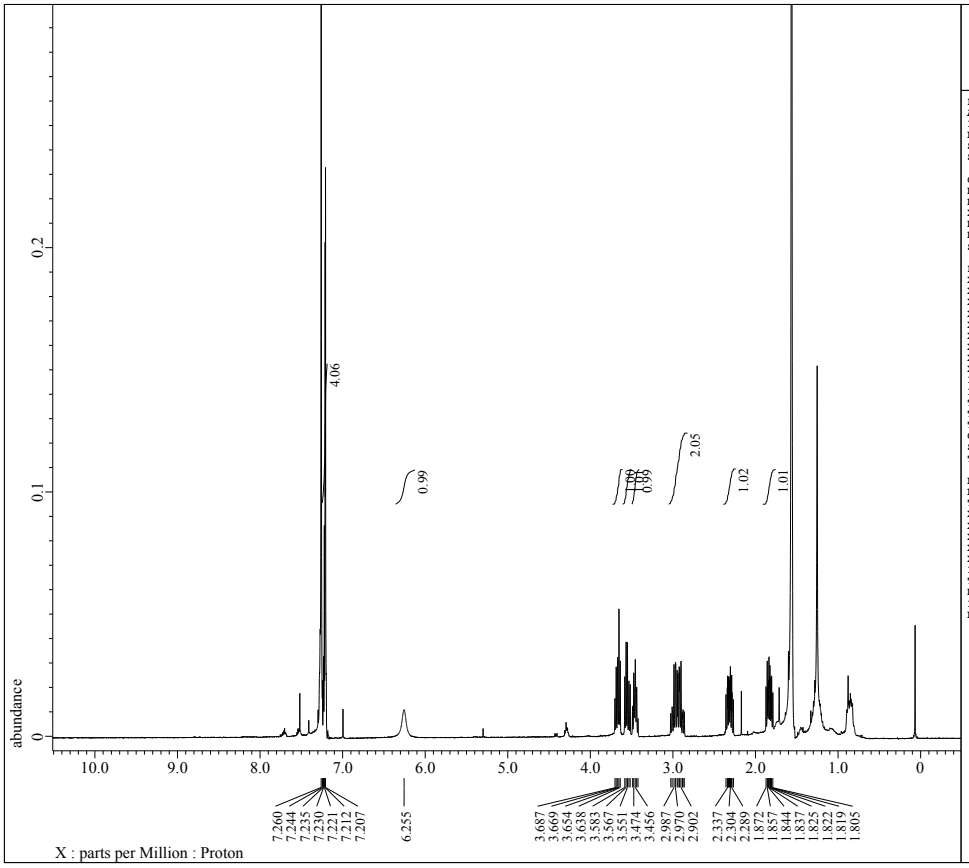

**5**




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 9.613 | 748447 | 95.698 |
| 2 | 9.992 | 33644 | 4.302 |

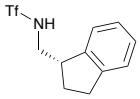



| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 9.643 | 345879 | 49.013 |
| 2 | 10.030 | 359816 | 50.987 |

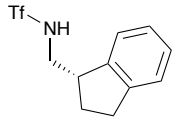



JEOL

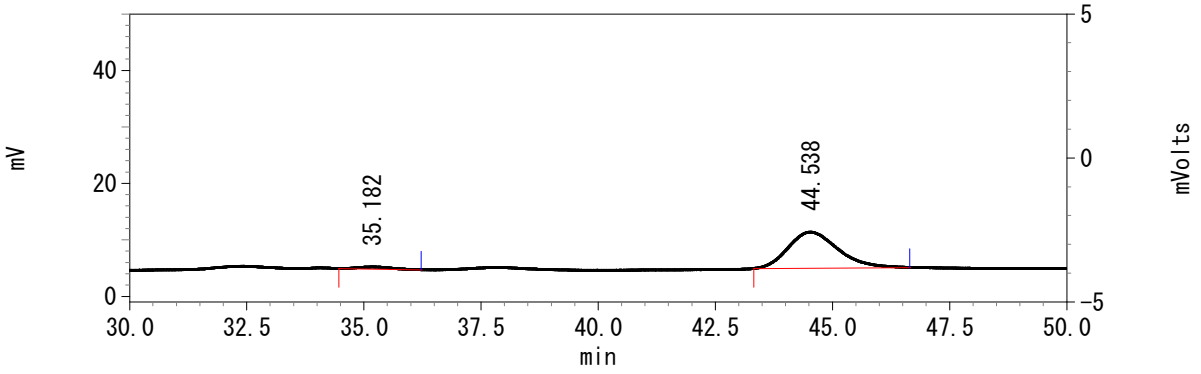
```
Filename          = 6 proton-1.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = sk#17-tf-gpc-2
Solvent           = CHLOROFORM-D

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             = 144
Total_Scans       = 144

Relaxation_Delay  = 5[s]
Recvr_Gain        = 66
Temp_Get          = 22.6[dC]
X_90_Width        = 6.3[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.15[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



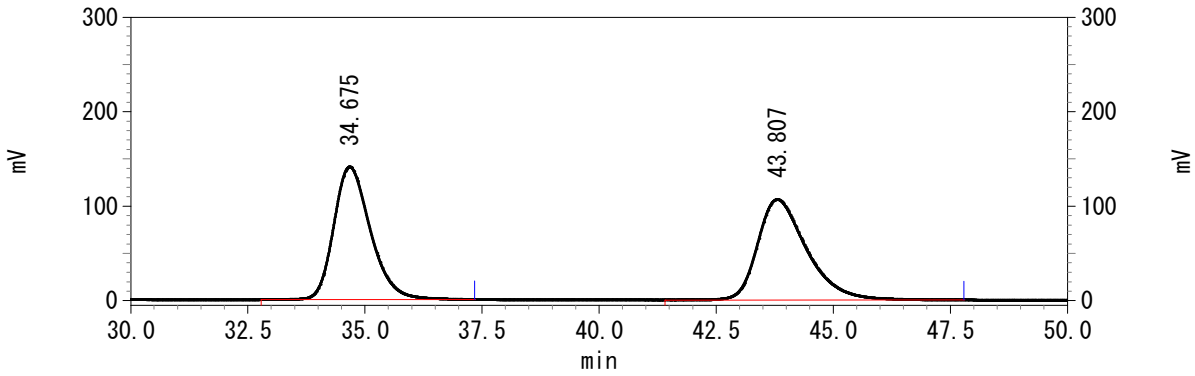

**6**



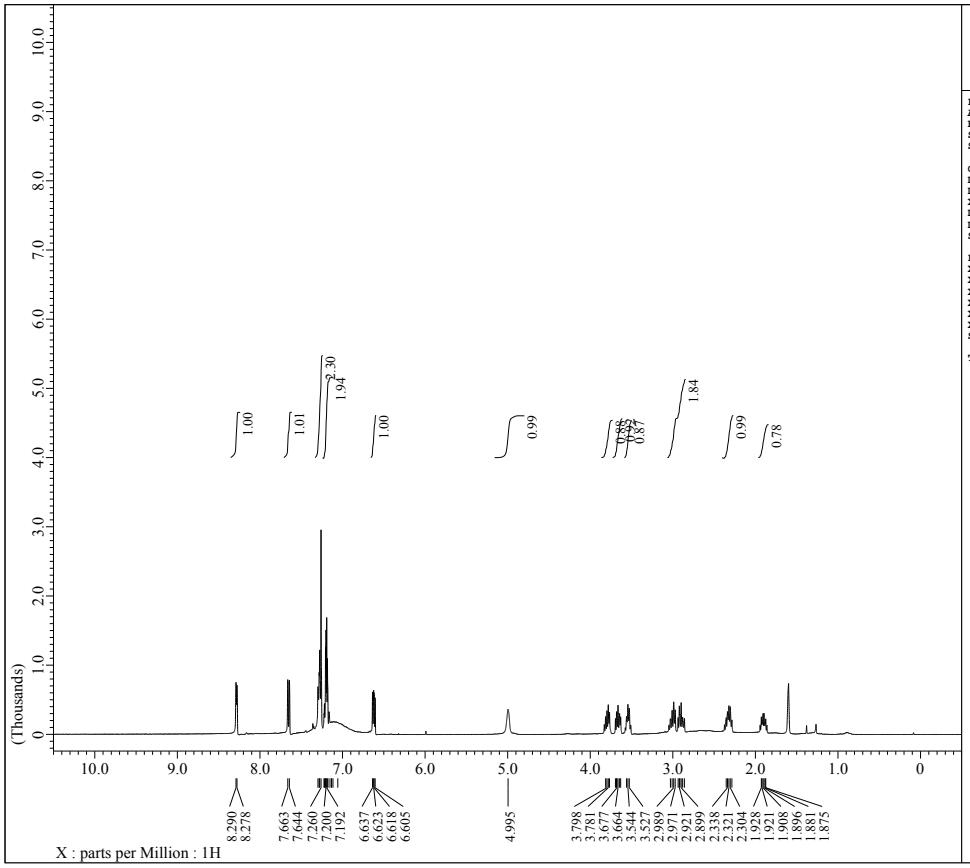

6




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 35.182 | 19746 | 4.017 |
| 2 | 44.538 | 471830 | 95.983 |




| Pk # | Retention Time | Area | Area Percent |
|---|---|---|---|
| 1 | 34.675 | 7666070 | 49.712 |
| 2 | 43.807 | 7754866 | 50.288 |

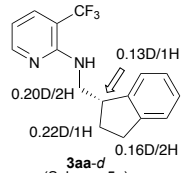



Filename = Scheme 5a_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D

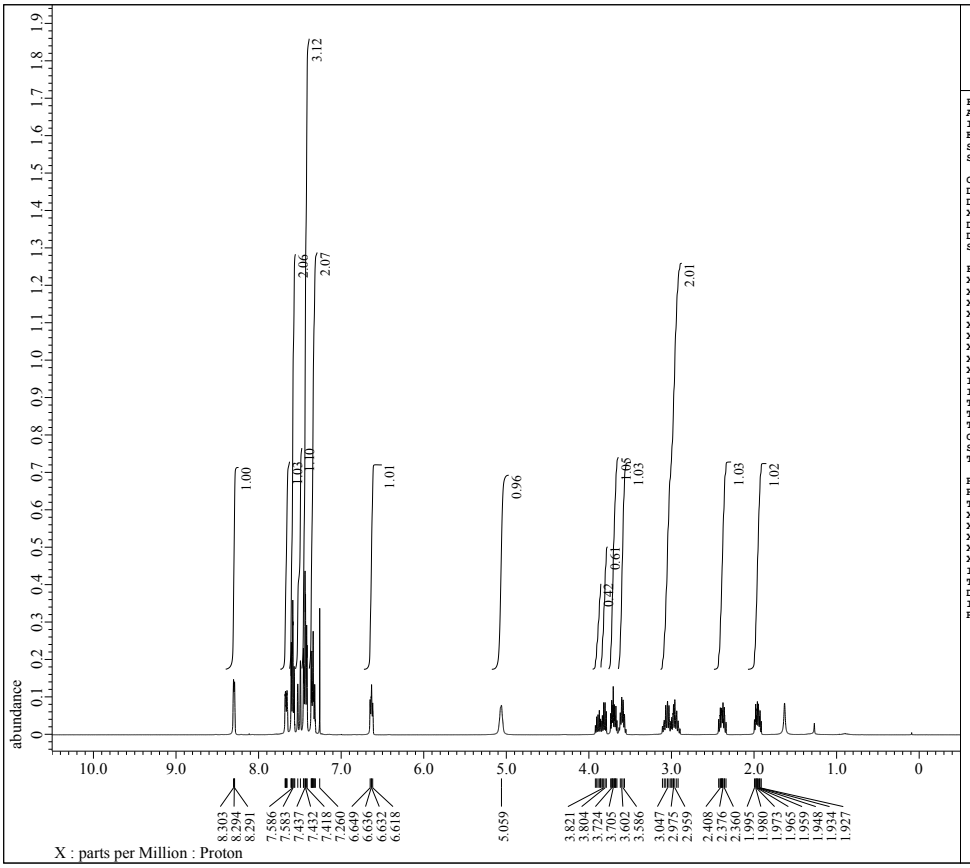
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR

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 = 296.8291[K]




**3aa**-*d*
(Scheme 5a)

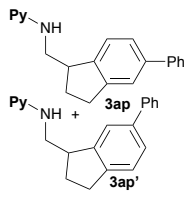



Filename = sk#15-035_Proton-1-1のコピー-
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = sk#15-035
Solvent = CHLOROFORM-D

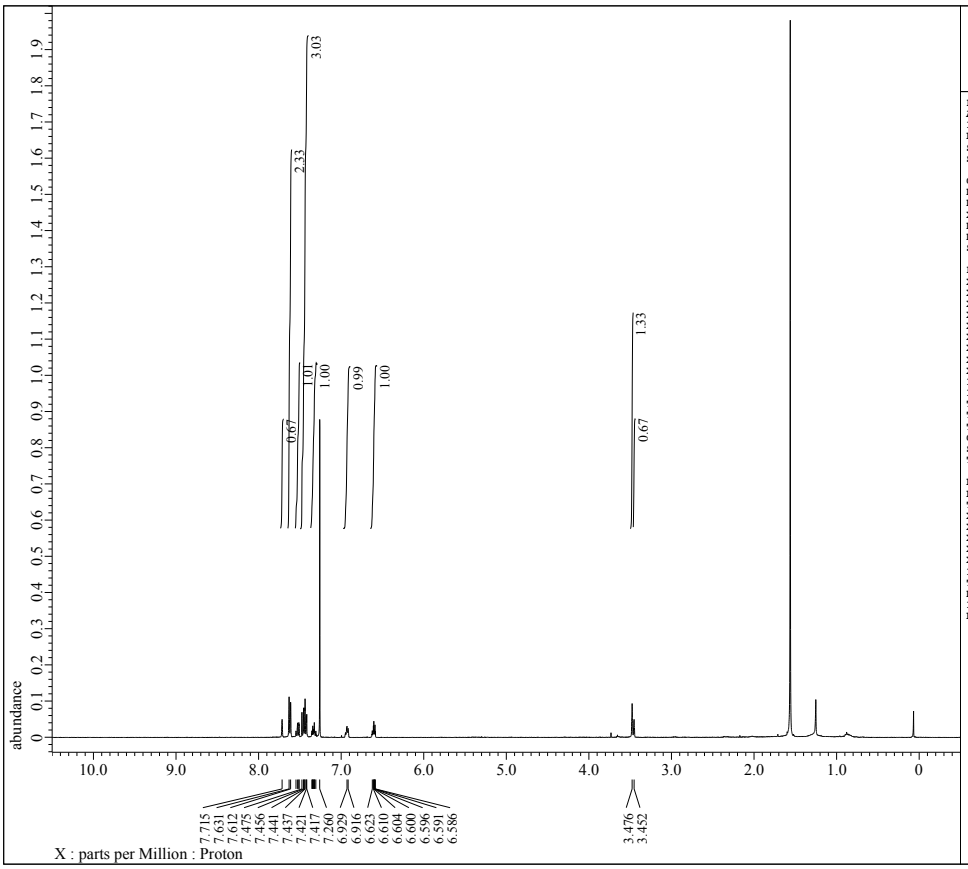
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

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 = 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]

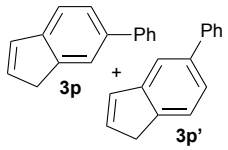



(Scheme 5b)

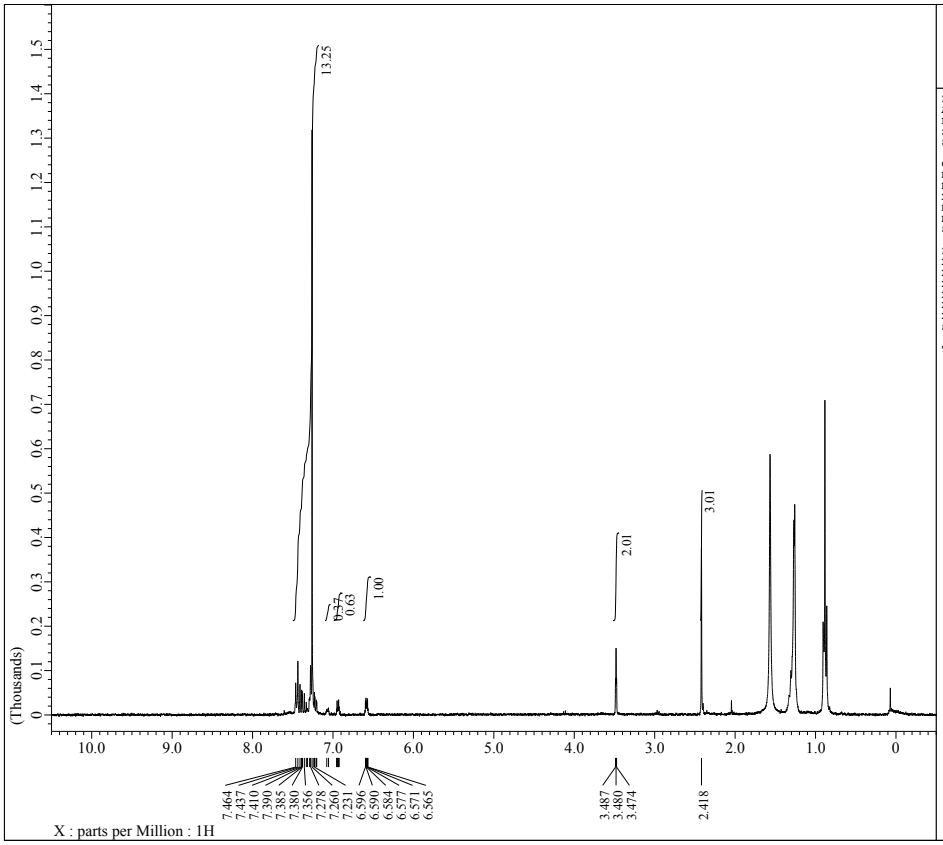




```
Filename          = sk#scheme5c_Proton-1-6.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = sk#scheme5c
Solvent           = CHLOROFORM-D

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          = 21.9[dC]
X_90_Width        = 6.3[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.15[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```

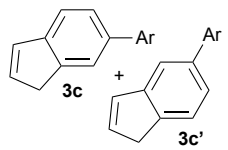




(Scheme 5c)





```
Filename          = Scheme 5d_10-4.jdf
Author            = FOC
Experiment        = zg30
Sample_Id         = Parameter file, TOPSPIN
Solvent           = CHLOROFORM-D

Comment           = Parameter file, TOPSPIN
Data_Format       = 1D COMPLEX
Dim_Size          = 32768
X_Domain          = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = BRUKER_DMX_NMR

Field_Strength    = 7.0492145[T] (300[MHz])
X_Domain          = 1H
X_Freq            = 300.13[MHz]
X_Offset          = 1.85342561[kHz]
X_Points          = 32768
X_Prescans        = 2
X_Sweep           = 6.1880806[kHz]
Scans             = 4

Temp_Get          = 294.46[K]
```


Ar = 4-methylphenyl
(Scheme 5d)