Supplementary Information

Ir-catalyzed cyclization of α,ω-dienes with an *N*-methyl group via two C–H activation steps

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Contents of Supplementary Information:

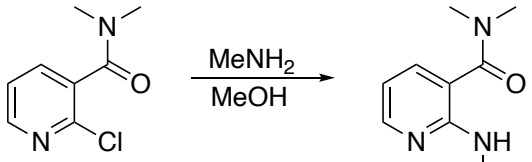
1. General

All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried 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 ^{1}H NMR, and $CDCl_3$ (δ 77.00) for ^{13}C NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: quint, quintet: 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). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque. Preparative thin-layer chromatography was performed with Wakogel® B-5F (Wako). Preparative recycling gel permeation chromatography (GPC) was performed using YMC-GPC T2000 (40 mm I.D.) or JAIGEL-1H and -2H using chloroform as eluent.

2. Materials

Dehydrated solvents were purchased and used after deoxygenated by bubbling N_2. $[IrCl(coe)_2]_2$,[1] $[IrCl(cod)]_2$,[2] and $NaBAr^F{}_4$ [Ar^F = 3,5-$(CF_3)_2C_6H_3$][3] were prepared according to the reported procedures. Compounds **1a** (CAS: 1251349-49-1),[4] **1c** (CAS: 97510-78-6),[5] **1d** (CAS: 103976-61-0),[6] **1e** (CAS: 468718-67-4),[7] **1f** (CAS: 46000-11-7),[8] **1h** (CAS: 4214-80-6),[9] **1i** (CAS: 1250153-33-3),[10] and **2r** (594858-64-7)[11] were prepared according to the reported procedures. Compounds **1b**, **2b**–**h**, **2j**–**2q**, and **2s**–**2u** were prepared as shown below. Other chemicals were purchased from commercial suppliers and used as received.

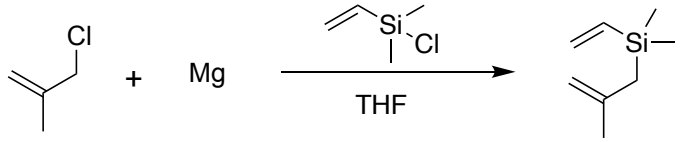
3. Preparation of substrates



s1b **1b**

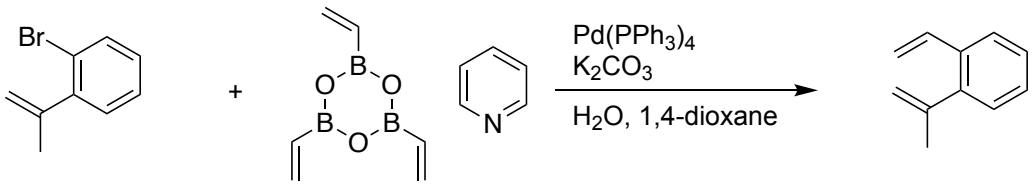
A mixture of **s1b** (CAS: 52943-21-2, 816 mg, 4.4 mmol) and $MeNH_2$ (9.8 M in MeOH, 10.2 mL, 100 mmol) in a pressure tube was stirred at 60 °C for 5 days. The mixture was concentrated on a rotary evaporator, and the residue was dissolved in CH_2Cl_2. To the solution was added silica gel (ca. 3 g) and the solvent was removed on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with EtOAc as an eluent to give **1b** (257 mg, 32% yield,

colorless solid). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.29 (dd, *J* = 7.4, 1.8 Hz, 1H), 6.51 (dd, *J* = 7.4, 5.0 Hz, 1H), 5.86 (br s, 1H), 3.04 (s, 6H), 2.95 (d, *J* = 4.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 157.1, 149.4, 135.9, 113.5, 110.6, 37.6 (br), 28.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_9H_{14}N_3O$ 180.1137; Found 180.1139.



2b

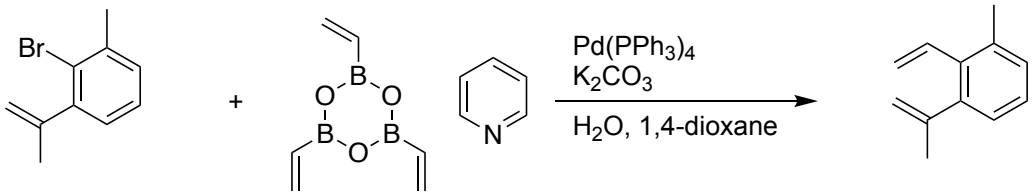
To a solution of (2-methylallyl)magnesium chloride, which was prepared from β-methallyl chloride (906 mg, 10 mmol) and Mg turnings (729 mg, 30 mmol) in THF (20 mL), was added dropwise chlorodimethylvinylsilane (965 mg, 8.0 mmol) in THF (10 mL) at 0 °C. The mixture was allowed to warm to room temperature and stirred overnight. H_2O was added to the mixture, and the resulting mixture was extracted with pentane three times. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator to give **2b** (338 mg) as a colorless oil, which was used in the next reaction without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.16 (dd, *J* = 20.2, 14.8 Hz, 1H), 5.96 (dd, *J* = 14.8, 3.6 Hz, 1H), 5.69 (dd, *J* = 20.2, 3.6 Hz, 1H), 4.62–4.57 (m, 1H), 4.52–4.46 (m, 1H), 1.71 (s, 3H), 1.60 (s, 2H), 0.10 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 138.9, 131.7, 108.4, 27.4, 25.2, –3.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_8H_{17}Si$ 141.1100; Found 141.1101.



s2c **2c**

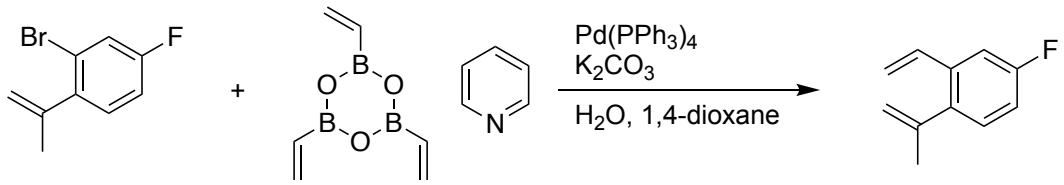
A solution of **s2c** [12] (CAS: 7073–70–3, 394 mg, 2.0 mmol) and tetrakis(triphenylphosphine)palladium (23.1 mg, 0.020 mmol, 1 mol %) in 1,4-dioxane (20 mL) was stirred at room temperature for 20 min. Potassium carbonate (276 mg, 2.0 mmol), water (6 mL), and 2,4,6-trivinylcycloboroxine pyridine complex (361 mg, 1.5 mmol) were added to the mixture. After the mixture was stirred at 100 °C overnight, H_2O was added the mixture, and the resulting mixture was extracted with pentane three times. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with pentane as an eluent to give **2c**[13] (CAS: 31382-76-0, colorless oil, 248 mg, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.52 (m, 1H), 7.28–7.20 (m, 1H), 7.19–

7.14 (m, 2H), 6.92 (dd, *J*= 17.6, 11.0 Hz, 1H), 5.69 (dd, *J*= 17.6, 1.2 Hz, 1H), 5.28–5.20 (m, 2H), 4.90–4.87 (m, 1H), 2.05 (dd, *J* = 1.5, 1.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.8, 142.9, 135.6, 134.8, 128.0, 127.5, 127.0, 125.4, 115.9, 114.2, 24.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{11}H_{13}$ 145.1017; Found 145.1013.



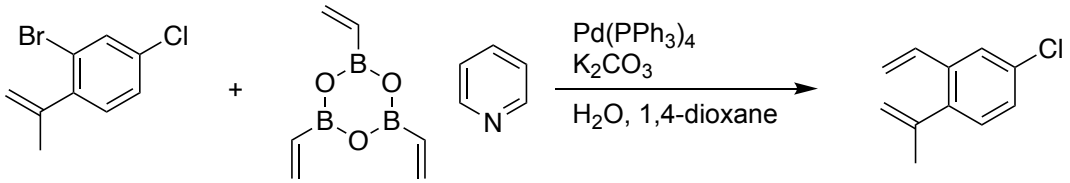
s2d **2d**

Compound **2d** was prepared by the reaction of **s2d**[14] (CAS:1838205-49-4, 605 mg, 3.0 mmol) with 2,4,6-trivinylcycloboroxine pyridine complex (361 mg, 1.5 mmol) in the presence of tetrakis(triphenylphosphine)palladium (23.1 mg, 0.020 mmol) in 1,4-dioxane (20 mL) and H_2O (6.0 mL) following the procedure for **2c** (colorless oil, 264 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16–7.10 (m, 2H), 7.08–7.01 (m, 1H), 6.77 (dd, *J* = 17.7, 11.6 Hz, 1H), 5.45 (dd, *J* = 5.5, 2.0 Hz, 1H), 5.42 (dd, *J* = 11.6, 2.0 Hz, 1H), 5.16–5.11 (m, 1H), 4.95–4.91 (m, 1H), 2.36 (s, 3H), 2.02 (dd, *J* = 1.4, 1.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 143.5, 135.8, 135.1, 135.0, 129.1, 126.7, 126.3, 119.1, 115.2, 24.2, 21.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{15}$ 159.1174; Found 159.1170.



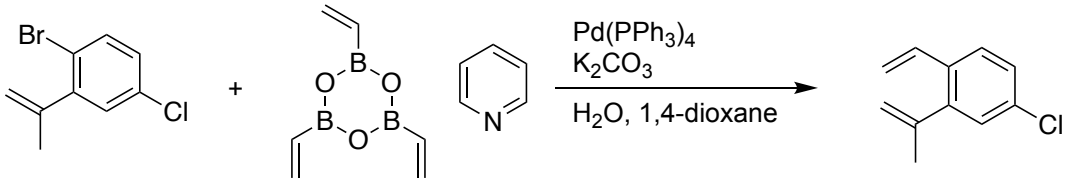
s2e **2e**

Compound **2e** was prepared by the reaction of **s2e**[15] (CAS: 51788-87-5) with 2,4,6-trivinylcycloboroxine pyridine complex following the procedure for **2c** (colorless oil, 279 mg, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (dd, *J* = 10.9, 2.7 Hz, 1H), 7.11 (dd. *J* = 8.4, 5.9 Hz, 1H) 6.92 (td, *J* = 8.4, 2.7 Hz, 1H), 6.86 (ddd, *J* = 17.5, 10.9, 1.7 Hz, 1H), 5.67 (dd, *J* = 17.5, 0.8 Hz, 1H), 5.30–5.22 (m, 2H), 4.87–4.84 (m, 1H), 2.04–2.00 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.9 (d, $J_{F–C}$ = 245 Hz), 138.9 (d, $J_{F–C}$ = 3 Hz), 136.9 (d, $J_{F–C}$ = 7 Hz), 134.8 (d, $J_{F–C}$ = 2 Hz), 129.6 (d, $J_{F–C}$ = 8 Hz), 116.5, 115.3, 114.3 (d, $J_{F–C}$ = 21 Hz), 111.7 (d, $J_{F–C}$ = 22 Hz), 24.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{11}H_{12}F$ 163.0923; Found 163.0917.



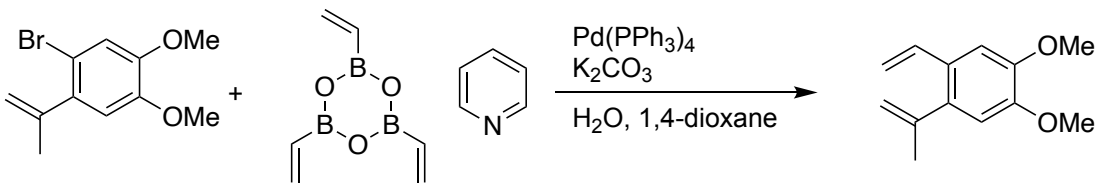
s2f **2f**

Compound **2f** was prepared by the reaction of **s2f**[14] (463 mg, 2.0 mmol, CAS: 1788090-78-7) with 2,4,6-trivinylcycloboroxine pyridine complex (361 mg, 1.5 mmol) in the presence of tetrakis(triphenylphosphine)palladium (23.1 mg, 0.020 mmol) in 1,4-dioxane (20 mL) and H_2O (6.0 mL) following the procedure for **2c** (colorless oil, 227 mg, 64% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, *J* = 2.2 Hz, 1H), 7.19 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.08 (d, *J* = 8.2 Hz, 1H), 6.84 (dd, *J* = 17.5, 11.0 Hz, 1H), 5.69 (dd, *J* = 17.5, 1.0 Hz, 1H), 5.31–5.23 (m, 2H), 4.90–4.84 (m, 1H), 2.04–2.00 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 141.2, 136.6, 134.6, 132.8, 129.4, 127.4, 125.4, 116.6, 115.5, 24.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{11}H_{12}{}^{35}Cl$ 179.0628; Found 179.0623.



s2g **2g**

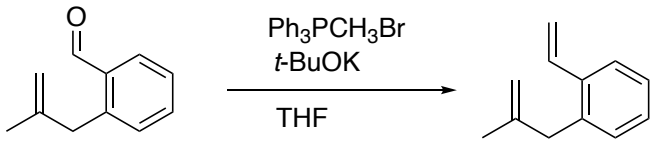
Compound **2g** was prepared by the reaction of **s2g**[16] (CAS: 1838205-47-2) with 2,4,6-trivinylcyclo pyridine complex following the procedure for **2c** (colorless oil, 349 mg, 98% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (dd, *J* = 8.4 Hz, 1H), 7.21 (dd, *J* = 8.4, 2.2 Hz, 1H), 7.14 (d, *J* = 2.2 Hz, 1H), 6.84 (dd, *J* = 17.6, 11.0 Hz, 1H), 5.66 (dd, *J* = 17.6, 1.0 Hz, 1H), 5.28–5.22 (m, 2H), 5.01–4.88 (m, 1H), 2.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 143.7, 134.5, 133.4, 133.0, 128.0, 127.1, 126.8, 116.7, 114.9, 24.5. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{11}H_{12}{}^{35}Cl$ 179.0628; Found 179.0624.



s2h **2h**

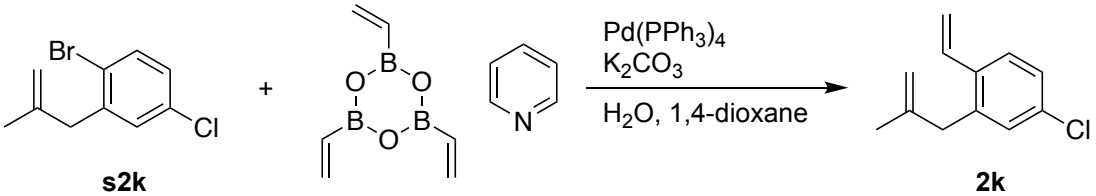
Compound **2h** was prepared by the reaction of **s2h**[17] (CAS: 171512-96-2) with 2,4,6-trivinylcycloboroxine pyridine complex following the procedure for **2c** (colorless oil, 416 mg, 99% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (s, 1H), 6.87 (dd, *J* = 17.5, 11.0 Hz, 1H), 6.66 (s, 1H), 5.56 (dd, *J* = 17.5, 1.1 Hz, 1H), 5.25–5.21 (m, 1H), 5.14 (dd, *J* = 11.0, 1.1 Hz, 1H), 4.89–4.85 (m,

1H), 3.92 (s, 3H), 3.88 (s, 3H), 2.04 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 147.9, 144.4, 135.9, 135.2, 127.2, 116.1, 112.1, 110.9, 107.9, 55.9, 24.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{17}O_2$ 205.1229; Found 205.1222.



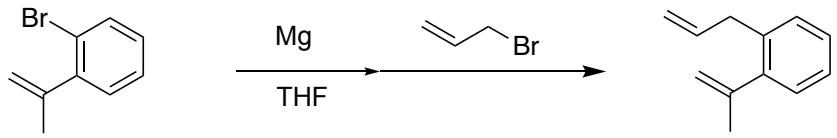
s2j **2j**

To a suspension of methyltriphenylphosphonium bromide (429 mg, 1.2 mmol) in THF (10 mL) at 0 °C was added *t*-BuOK (123 mg, 1.1 mmol). The resulting yellow solution was stirred for 30 min, and then, **s2j**[18] (CAS: 660820-44-0, 160 mg, 1.0 mmol) was added to the mixture. The mixture was stirred at room temperature overnight. H_2O was added to the mixture, and the resulting mixture was extracted with CH_2Cl_2 three times. The organic layer was dried over Na_2SO_4, filtered, and concentrated on rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane as an eluent to give **2j** (colorless oil, 137 mg, 83% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55–7.49 (m, 1H), 7.25–7.20 (m, 2H), 7.17–7.13 (m, 1H), 6.95 (dd, *J* = 17.4, 11.0 Hz, 1H), 5.64 (dd, *J* = 17.4, 1.4 Hz, 1H), 5.27 (dd, *J* = 11.0, 1.4 Hz, 1H), 4.86–4.80 (m, 1H), 4.55–4.50 (m, 1H), 3.38 (s, 2H), 1.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.7, 41.4, 111.9, 115.2, 125.6, 126.6, 127.7, 130.3, 134.7, 136.8, 137.2, 144.5. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{15}$ 159.1174; Found 159.1174.



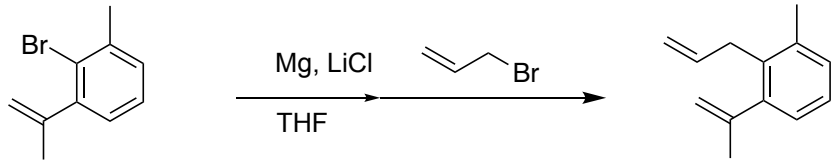
s2k **2k**

Compound **2k** was prepared by the reaction of **s2k**[19] (CAS: 1788090-78-7) with 2,4,6-trivinylcycloboroxine pyridine complex following the procedure for **2c** (colorless oil, 374 mg, 97% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.2 Hz, 1H), 7.18 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.13 (d, *J* = 2.2 Hz, 1H), 6.86 (dd, *J* = 17.4, 11.0 Hz, 1H), 5.61 (dd, *J* = 17.4, 1.2 Hz, 1H), 5.28 (dd, *J* = 11.0, 1.2 Hz, 1H), 4.89–4.82 (m, 1H), 4.54–4.50 (m, 1H), 3.33 (s, 2H), 1.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 138.6, 135.7, 133.7, 133.2, 130.0, 127.0, 126.7, 115.9, 112.5, 41.2, 22.7. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{14}{}^{35}Cl$ 193.0784; Found 193.0780.



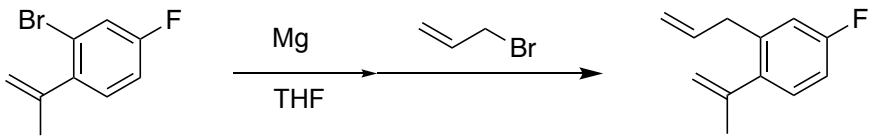
s2c **2l**

To a solution of (2-(prop-1-en-2-yl)phenyl)magnesium bromide in THF (14 mL), which was prepared from **s2c**[12] (CAS: 7073-70-3, 1.38 g, 7.0 mmol), a chip of I_2, and Mg turnings (204 mg, 8.4 mmol), was added dropwise allyl bromide (1.40 mL, 16.8 mmol) at 0 °C. The mixture was allowed to warm to room temperature and stirred overnight. Saturated NH_4Cl aq. and 2 M HCl were added to the mixture, and the resulting mixture was extracted with Et_2O three times. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with pentane as an eluent to give **2l**[20] (CAS: 21919-45-9, colorless oil, 743 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.13 (m, 4H), 5.98 (ddt, *J* = 16.7, 10.2, 6.5 Hz, 1H), 5.24–5.19 (m, 1H), 5.10–5.01 (m, 2H), 4.90–4.85 (m, 1H), 3.44 (d, *J* = 6.5 Hz, 2H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.4, 143.7, 138.0, 136.4, 129.5, 128.1, 126.9, 125.9, 115.6, 114.9, 37.2, 25.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{15}$ 159.1174; Found 159.1167.



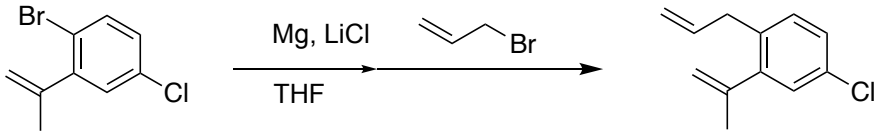
s2d **2m**

To a solution of (2-methyl-6-(prop-1-en-2-yl)phenyl)magnesium bromide in THF (7 mL), which was prepared from **s2d**[16] (CAS: 1838205-49-4, 781 mg, 3.7 mmol), LiCl (235 mg, 5.5 mmol), and Mg turnings (134 mg, 5.5 mmol), was added dropwise allyl bromide (0.94 mL, 11 mmol) at room temperature, and the mixture was stirred overnight. Saturated NH_4Cl aq. was added to the mixture, and the resulting mixture was extracted with Et_2O three times. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane as an eluent to give **2m** (colorless oil, 289 mg), which was further purified by GPC to remove a small amount of impurities. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13–7.06 (m, 2H), 6.99 (dd, *J* = 7.0, 1.8 Hz, 1H), 5.98 (ddt, *J* = 17.0, 10.4, 5.5 Hz, 1H), 5.18–5.14 (m, 1H), 5.98 (dq, *J* = 10.4, 1.6 Hz, 1H), 4.89–4.82 (m, 2H), 3.44 (dt, *J* = 5.5, 1.9 Hz, 2H), 2.32 (s, 3H), 2.03 (t, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 144.5, 137.4, 137.0, 134.2, 128.9, 125.9, 115.0, 114.4, 34.3, 25,6, 19.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{17}$ 173.1330; Found 173.1322.



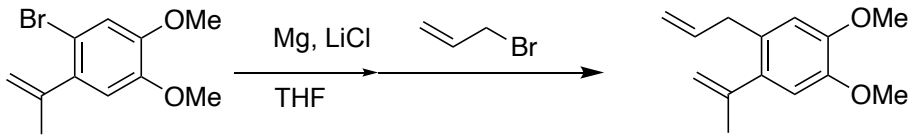
s2e **2n**

Compound **2n** was prepared from **s2e**[15] (430.1 mg, 2.0 mmol) following the procedure for **2l** (colorless oil, 193 mg, 55% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.13 (m, 4H), 5.98 (ddt, J = 16.7, 10.2, 6.5 Hz, 1H), 5.24–5.19 (m, 1H), 5.10–5.01 (m, 1H), 4.90–4.85 (m, 1H), 3.44 (d, J = 6.5 Hz, 2H), 2.06 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.7 (d, $J_{F–C}$ = 245 Hz) 144.5, 139.5 (d, $J_{F–C}$ = 3 Hz), 138.8 (d, $J_{F–C}$ = 7 Hz), 137.1, 129.5 (d, $J_{F–C}$ = 8 Hz), 116.3, 115.9 (d, $J_{F–C}$ = 21 Hz), 115.5, 112.7 (d, $J_{F–C}$ = 21 Hz), 37.2, 25.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{14}F$ 177.1080; Found 177.1077.



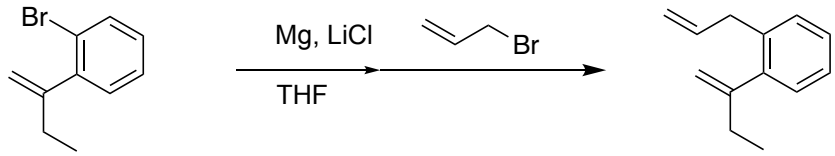
s2o **2o**

Compound **2o** was prepared from **s2o**[17] (CAS: 1838205-47-2, 875 mg, 3.8 mmol) following the procedure for **2m** (colorless oil, 861 mg, which include a amall amount of impurities). The obtained **2o** was used in the next reaction without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (dd, J = 8.4, 2,2 Hz, 1H), 7.130 (d, J = 8.4 Hz, 1H), 7.126 (d, J = 2.2 Hz, 1H), 5.99–5.85 (m, 1H), 5.24–5.19 (m, 1H), 5.10–5.01 (m, 2H), 4.89–4.85 (m, 1H), 3.37 (d, J = 6.4 Hz, 2H), 2.07–1.99 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.3, 144.1, 137.5, 134.9, 131.4, 130.9, 128.0, 126.9, 116.0, 115.7, 36.5, 24.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{12}H_{14}{}^{35}Cl$ 193.0784; Found 193.0775.



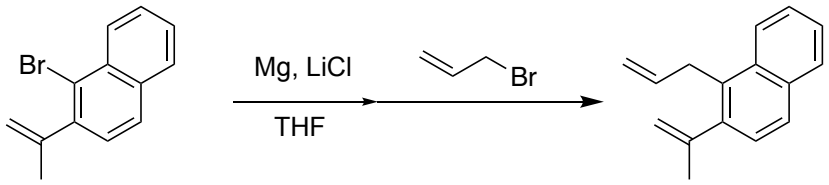
s2p **2p**

Compound **2p** was prepared from **s2p**[17] (CAS: 171512-96-2, 1.03 g, 4.0 mmol) following the procedure for **2m** (colorless oil, 531 mg; the obtained **2p** was further purified by GPC to remove a small amount of impurities). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.70 (s, 1H), 6.66 (s, 1H), 5.96 (ddt, J = 16.6, 10.2, 6.4 Hz, 1H), 5.20–5.14 (m, 1H), 5.08–4.97 (m, 2H), 4.87–4.83 (m, 1H), 3.86 (s, 6H), 3.36 (dt, J = 6.4, 1.6 Hz, 2H), 2.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 146.8, 145.0, 138.2, 135.8, 128.3, 115.3, 114.9, 112.4, 111.2, 55.78, 55.75, 36.8, 25.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{14}H_{19}O_2$ 219.1385; Found 219.1381.



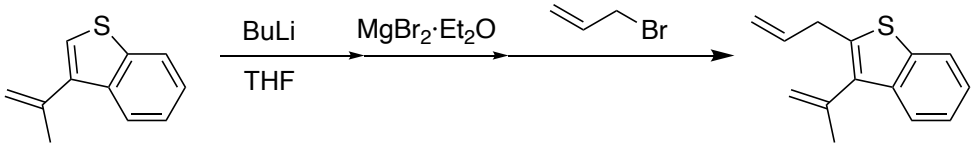
s2q **2q**

Compound **2q** was prepared from **s2q**[17] (CAS: 326879-16-7, 1.43 g, 6.8 mmol) following the procedure for **2m** (colorless oil, 823 mg; the obtained **2q** was further purified by GPC to remove a small amount of impurities). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24–7.12 (m, 3H), 7.11–7.06 (m, 1H), 5.94 (ddt, *J* = 16.8, 10.4, 6.6 Hz, 1H), 5.18–5.16 (m, 1H), 5.09–4.96 (m, 2H), 4.87–4.85 (m, 1H), 3.38 (dt, *J* = 6.4, 1.6 Hz, 2H), 2.36–2.29 (m, 2H), 1.04 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.1, 143.3, 138.1, 136.7, 129.4, 128.5, 126.8, 125.7, 115.5, 112.7, 37.1, 31.1, 12.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{17}$ 173.1330; Found 173.1324.



s2s **2s**

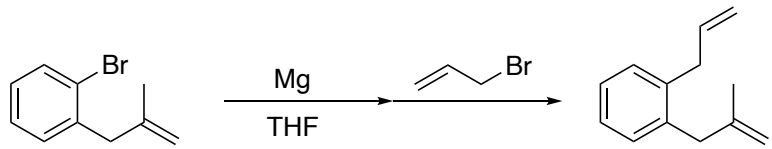
Compound **2s** was prepared from **s2s**[15] (CAS: 1673538-53-8, 1.48 g, 6.0 mmol), following the procedure for **2m** (colorless oil, 1.10 g; the obtained **2s** was further purified by GPC to remove a small amount of impurities). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.4 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 1H), 7.71 (d, *J* = 8.8 Hz, 1H), 7.53–7.42 (m, 2H), 7.29 (d, *J* = 8.8 Hz, 1H), 6.15–6.04 (m, 1H), 5.29–5.26 (m, 1H), 5.05 (dq, *J* = 10.0, 1.9 Hz, 1H), 4.96–4.88 (m, 2H), 3.91–3.87 (m, 2H), 2.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.0, 141.5, 137.9, 132.8, 132.4, 131.1, 128.4, 126.6, 125.9, 125.1, 125.0, 115.7, 115.0, 33.6, 25.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{17}$ 209.1330; Found 209.1322.



s2t **2t**

To a solution of **s2t**[21] (CAS: 69994-86-1, 977 mg, 5.6 mmol) in THF (10 mL) at –78 °C was added BuLi (1.6 M in hexane, 5.3 mL, 8.4 mmol), and the mixture was stirred at the same temperature for 2 h. To the mixture was added $MgBr_2 \cdot Et_2O$ (2.2 g, 8.4 mmol) in THF (5 mL), and the mixture was allowed to warm to room temperature and stirred for 1 h. To the solution was added allyl bromide (0.95 mL, 11.2 mmol) and the mixture was stirred overnight. Saturated NH_4Cl aq. and 2 M HCl were added to the mixture, and the resulting mixture was extracted with Et_2O three times.

The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with pentane as an eluent to give **2t** (colorless oil, 899 mg), which was further purified by GPC to remove a small amount of impurities. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 7.2 Hz, 1H), 7.64 (d, J = 8.0 Hz, 1H), 7.35 (td, J = 7.5, 1.2 Hz, 1H), 7.30 (td, J = 7.5, 1.5 Hz, 1H), 6.04 (ddt, J = 17.0, 10.2, 6.6 Hz, 1H), 5.40–5.50 (m, 1H), 5.23–5.11 (m, 2H), 5.06–5.01 (m, 1H), 3.66 (dt, J = 6.6, 1.4 Hz, 2H), 2.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6, 139.5, 138.7, 137.5, 136.4, 135.9, 123.9, 123.7, 122.4, 122.1, 117.4, 116.4, 33.1, 23.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{14}H_{15}S$ 215.0895; Found 215.0902.



s2u **2u**

Compound **2u** was prepared from **s2u**[18] (CAS: 514821-14-8, 422 mg, 2.0 mmol) following the procedure for **2l** (colorless oil, 196 mg, which includes a amall amount of impurities). The obtained **2u** was used in the next reaction without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.13 (m, 4H), 5.98 (ddt, J = 16.8, 10.0, 6.5 Hz, 1H), 5.07 (ddd, J = 10.0, 3.4, 1.4 Hz, 1H), 5.02 (ddd, J = 16.8, 3.4, 1.8 Hz, 1H) 4.84 (d, J = 0.80 Hz, 1H), 4.55 (d, J = 0.80 Hz, 1H), 3.39 (dt, J = 6.5, 1.4 Hz, 1H), 3.35 (s, 3H), 1.75 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 138.4, 137.6, 137.2, 130.2, 129.5, 126.5, 126.3, 115.6, 111.8, 41.2, 37.1, 22.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{17}$ 173.1330; Found 173.1322.

4. Procedure for Table 1

$NaBAr^F_4$ (18.4 mg, 0.020 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir), ligand (0.012 mmol), and a solvent (0.40 mL) were added to the tube, and the mixture was stirred at room temperature for 10 min. Then, **1a** (20.5 mg, 0.10 mmol) and 2-methyl-1,5-hexadiene (**2a**, 14.4 mg, 0.15 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the yields of the products were determined by ^{1}H NMR using benzyl phenyl ether as an internal standard.

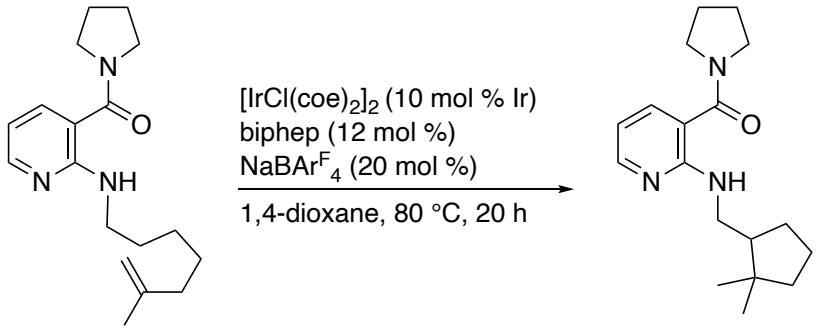
5. Procedure for Scheme 2

$NaBAr^F_4$ (36.8 mg, 0.040 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (8.9 mg, 0.010 mmol, 10 mol% of Ir), biphep (12.5 mg, 0.024 mmol), and 1,4-dioxane (0.80 mL) were added to the tube, and the mixture was stirred at room temperature for 10 min. Then, **1** (0.20 mmol) and **2a** (28.9 mg, 0.30 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel to give **3**. In the reactions of compounds **1d**, racemic binap was used as a ligand instead of biphep because of the low reactivity. The procedure for **1f** was carried out in a half scale reaction.

6. Procedure for Table 2

$NaBAr^F_4$ (36.8 mg, 0.040 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (8.9 mg, 0.010 mmol, 10 mol% of Ir), biphep (12.5 mg, 0.024 mmol), and 1,4-dioxane (0.80 mL) were added to the tube, and the mixture was stirred at room temperature for 10 min. Then, **1** (0.20 mmol) and **2** (0.30 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel to give **3**. In the reactions of compounds **2h** (Table 2, entry 9) and **2r** (entry 19), racemic binap was used as a ligand instead of biphep. In entries 14 and 16–19 of Table 2, the procedures were carried out in a half scale reaction (0.10 mmol of **1**).

7. Reaction of 4aa (eqn (1))

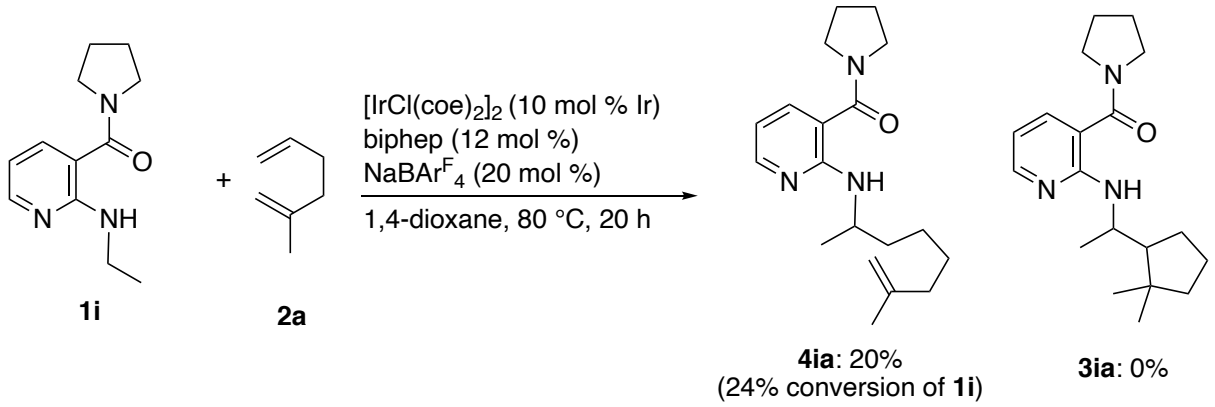


4aa **3aa**: quant.

$NaBAr^F_4$ (18.4 mg, 0.020 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir), biphep (6.2 mg, 0.012 mmol), and a solution of **4aa** in 1,4-dioxane (0.25 M, 0.40 mL, 0.10 mmol) were added to the tube, and the mixture was stirred

at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and dried under vacuum. The quantitative formation of **3aa** was observed by ^{1}H NMR.

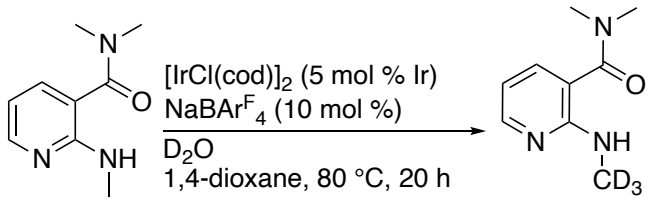
8. Reaction of 1h (eqn (2))



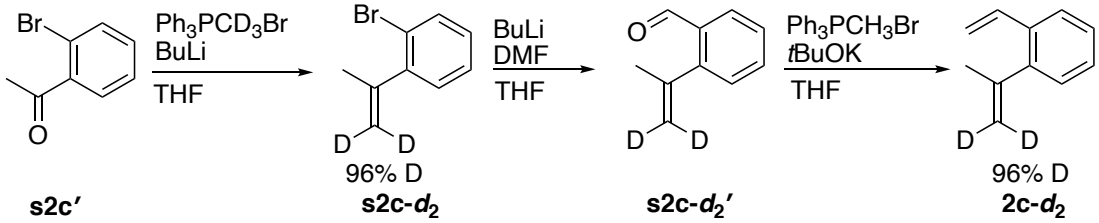
$NaBAr^F_4$ (18.4 mg, 0.020 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir) and biphep (6.3 mg, 0.012 mmol), and 1,4-dioxane (0.40 mL) were added to the tube, and the mixture was stirred at room temperature for 10 min. Then, **1i** (21.7 mg, 0.10 mmol) and **2a** (20 μL, 0.15 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with a solution of EtOAc and hexane (1:1) to give **4ia** (6.0 mg, 20% yield) as well as unreacted **1i** (16.4 mg, 76% recovered). **Compound 4ia**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (dd, J = 5.2, 2.0 Hz, 1H), 7.41 (dd, J = 7.2, 2.0 Hz, 1H), 6.45 (dd, J = 7.2, 5.2 Hz, 1H), 6.42–6.27 (m, 1H), 4.65 (s, 1H), 4.63 (s, 1H), 4.21–4.07 (m, 1H), 3.88–3.30 (m, 4H), 2.06–1.73 (m, 6H), 1.67 (s, 3H), 1.63–1.28 (m, 6H), 1.19 (d, J = 7.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 156.5, 149.9, 146.1, 136.1, 113.3, 109.9, 109.6, 49.8 (br), 46.1 (br), 37.7, 36.9, 27.6, 26.3 (br), 25.7, 24.3 (br), 22.4, 20.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{30}N_3O$ 316.2389; Found 316.2374.

9. Deuterium-labeling experiments

9-1. Preparation of deuterated substrates



$NaBAr^F_4$ (46.1 mg, 0.0500 mmol, 10 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(cod)]_2$ (8.3 mg, 0.0250 mmol, 10 mol% of Ir), a solution of **1b** in 1,4-dioxane (0.25 M, 0.40 mL, 0.10 mmol), and D_2O (1.8 mL, 100 mmol) were added to the tube, and the mixture was stirred at 80 °C for 20 h. The mixture was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on alumina with ethyl acetate as an eluent to give **1b-*d*$_3$** (colorless solid, 89.1 mg, 98% yield). The deuterium content of the product was determined by ^{1}H NMR: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, J = 5.0 Hz, 1H), 7.31 (dd, J = 7.2, 1.4 Hz, 1H), 6.53 (dd, J = 7.2, 5.0 Hz, 1H), 5.88 (br s, 1H), 3.05 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 157.3, 149.6, 135.8, 113.5, 110.6, 37.3 (br), 27.5 (sept, $J_{D–C}$ = 20.9). HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_9H_{11}{}^2H_3N_3O$ 183.1325; Found 183.1320.



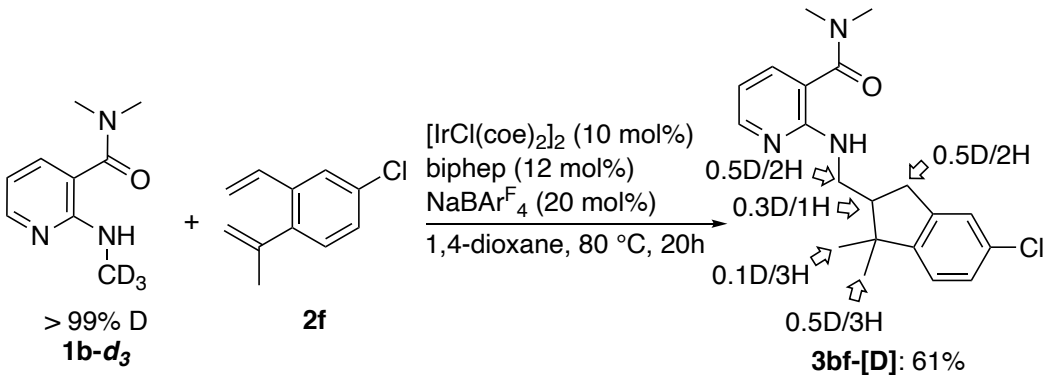
To a solution of methyl-d_3-triphenylphosphonium bromide[22] (CAS: 57472-87-4, 96% D, 468 mg, 1.3 mmol) in THF (2 mL) at –78 °C was added dropwise BuLi (1.6 M in hexane, 0.70 mL, 1.1 mmol), and the mixture was stirred at the same temperature for 30 min. To the mixture was added **s2c′** (CAS: 2142-69-0, 0.13 mL, 1.0 mmol), and the mixture was allowed to warm to room temperature and stirred overnight. Pentane was added to the mixture, and the resulting mixture was filtered and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with pentane as an eluent to give **s2c-*d*$_2$** (colorless oil, 109 mg, 55% yield).

To a solution of **s2c-*d*$_2$** (109 mg, 0.55 mmol) in THF (1.0 mL) at –78 °C was added dropwise BuLi (1.6 M in hexane, 5.3 mL, 8.4 mmol), and the mixture was stirred at the same temperature for 3 h. To the mixture was added DMF (0.25 mL, 0.32 mmol) in THF (0.70 mL), and the mixture was

allowed to warm to room temperature and stirred for 1 h. Saturated NH_4Cl aq. was added to the mixture, and the resulting mixture was extracted with CH_2Cl_2 three times. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was used in the next reaction without further purification.

To a solution of methyltriphenylphosphonium bromide (255 mg, 0.7 mmol) in THF (1.0 mL) at 0 °C was added potassium *tert*-butoxide (73.8 mg, 0.66 mmol), and the mixture was allowed to warm to room temperature and stirred for 30 min. The resulting solution was cooled to 0 °C and **s2c-*d*$_2$′** in THF (1 mL) was added to the mixture. The resulting mixture was allowed to warm to room temperature and stirred overnight. The reaction was quenched with H_2O, and the resulting mixture was extracted with CH_2Cl_2 three times. The organic layer was dried over anhydrous sodium sulfate, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with pentane as an eluent to give **2c-*d*$_2$** (colorless, oil, 26.8 mg, 33% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59–7.52 (m, 1H), 7.29–7.20 (m, 2H), 7.19–7.13 (m, 1H), 6.92 (dd, *J* = 17.6, 11.0 Hz, 1H), 5.69 (dd, *J* = 17.6, 1.2 Hz, 1H), 5.23 (dd, *J* = 11.0, 1.2 Hz, 1H), 4.91–4.84 (m, 0.04H), 2.05 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ x144.6, 142.9, 135.6, 134.8, 128.0, 127.5, 127.0, 125.3, 114.3, 115.9 (m), 24.7. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{11}H_{11}{}^2H_2$ 147.1143; Found 147.1136.

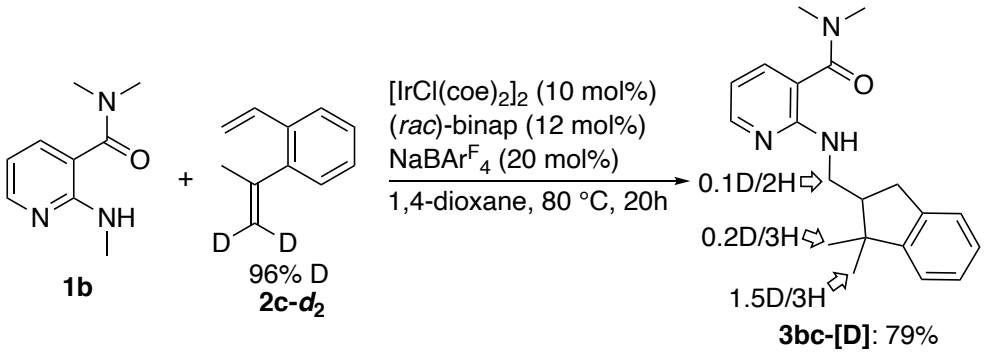
9-2. Reaction of 1b-*d*$_3$ with 2f



$NaBAr^F{}_4$ (18.4 mg, 0.020 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir), biphep (6.2 mg, 0.012 mmol), and 1,4-dioxane (0.40 mL) were added, and the mixture was stirred at room temperature for 10 min. Then, **1b-*d*$_3$** (17.9 mg, 0.10 mmol) and **2f** (26.8 mg, 0.15 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with a solution of EtOAc and hexane (1:2) to give **3bf-[D]** (23.1 mg, 61% yield). The deuterium contents of the products were determined by ^{1}H

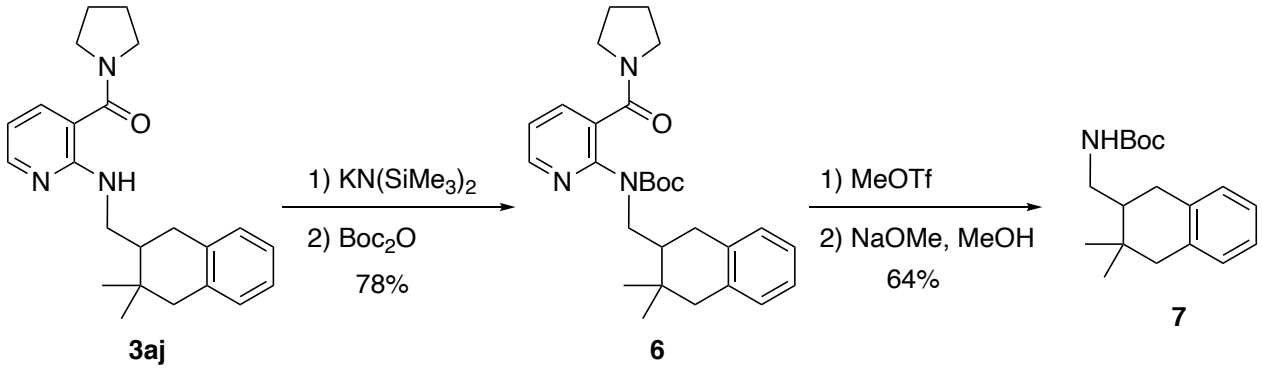
NMR: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8. 17 (dd, *J* = 5.0, 1.5 Hz, 1H), 7.34 (dd, *J* = 7.1, 1.5 Hz, 1H), 7.20–7.10 (m, 2H), 7.08–7.00 (m, 1H), 6.54 (dd, *J* = 7.1, 5.0 Hz, 1H), 6.05 (br s, 1H), 3.75–3.61 (m, 0.75H), 3.54–3.39 (m, 0.75H), 3.07 (s, 6H), 3.18–2.93 (m, 0.87H), 2.78–2.63 (m, 0.65H), 2.47–2.30 (m, 0.70H), 1.37 (s, 2.93H), 1.10 (s, 2.48H). ^{2}H NMR (61 MHz, $CHCl_3$) δ 3.68 (br s), 3.47 (br s), 3.05 (br s), 2.71 (br s), 2.38 (br s), 1.37 (br s), 1.10 (br s).

9-3. The reaction of 1b with 2c-*d*$_2$



$NaBAr^F_4$ (18.4 mg, 0.020 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir), (*rac*)-binap (7.5 mg, 0.012 mmol), and 1,4-dioxane (0.40 mL, 0.25 M) were added, and the mixture was stirred at room temperature for 10 min. Then, **1b** (17.9 mg, 0.10 mmol) and **2c-*d*$_2$** (22.0 mg, 0.15 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with a solution of EtOAc and hexane (1:2) to give **3bc-[D]** (25.4 mg, 79% yield). The deuterium contents of the products were determined by ^{1}H NMR($CDCl_3$): ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.34 (dd, *J* = 7.4, 1.8 Hz, 1H), 7.24–7.08 (m, 4H), 6.54 (dd, *J* = 7.4, 5.0 Hz, 1H), 6.04 (br s, 1H), 3.71 (dt, *J* = 12.7, 5.4 Hz, 0.96H), 3.55–3.41 (m, 0.97H), 3.19–2.98 (m, 1H), 3.07 (s, 6H), 2.75 (dd, *J* = 15.1, 10.1 Hz, 1H), 2.48–2.31 (m, 1H), 1.40 (s, 2.48H), 1.17 (m, 1.53H). ^{2}H NMR (61 MHz, $CHCl_3$) δ 3.70 (br s), 3.50 (br s), 1.40 (br s), 1.11 (br s).

10. Transformation of the pyridyl group



To a solution of compound **3aj** (89.9 mg, 0.25 mmol) in THF (5.0 mL) was added potassium bis(trimethylsilyl)amide solution (0.50 M in toluene, 1.0 mL, 0.50 mmol) at room temperature under N_2. After the mixture was stirred at room temperature for 30 min, di-*tert*-butyl dicarbonate (Boc_2O, 216 mg, 1.0 mmol) was added to the tube and the mixture was stirred at 40 °C for 30 min and at room temperature for 20 h. H_2O was added to the mixture and the resulting mixture was extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under vacuum. The residue was subjected to flash column chromatography on silica gel with EtOAc/hexane (1:1) to give **6** (89.3 mg, 0.19 mmol, 78% yield, brown oil). **Compound 6**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (dd, *J* = 4.8, 1.9 Hz, 1H), 7.60 (dd, *J* = 7.5, 1.9 Hz, 1H), 7.10 (dd, *J* = 7.5, 4.8 Hz, 1H), 6.95–7.06 (m, 4H), 4.01–4.28 (m, 1H), 3.78–3.94 (m, 1H), 3.54 (t, *J* = 6.7 Hz, 2H), 3.34–3.52 (m, 2H), 3.17 (dd, *J* = 17.5, 5.4 Hz, 1H), 2.60–2.78 (m, 1H), 2.59 (d, *J* = 16.5 Hz, 1H), 2.48 (d, *J* = 16.5 Hz, 1H), 2.03–2.17 (m, 1H), 1.82–1.97 (m, 4H), 1.43 (s, 9H), 1.03 (s, 3H), 0.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 154.9, 152.3, 148.3, 135.9, 135.6, 129.6, 129.1, 128.8, 125.2, 125.0, 119.9, 80.7, 49.1, 48.8, 45.7, 44.0, 31.6, 29.9, 28.4, 28.2, 26.2, 24.6, 22.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{28}H_{38}N_3O_3$ 464.2913; Found 464.2909.

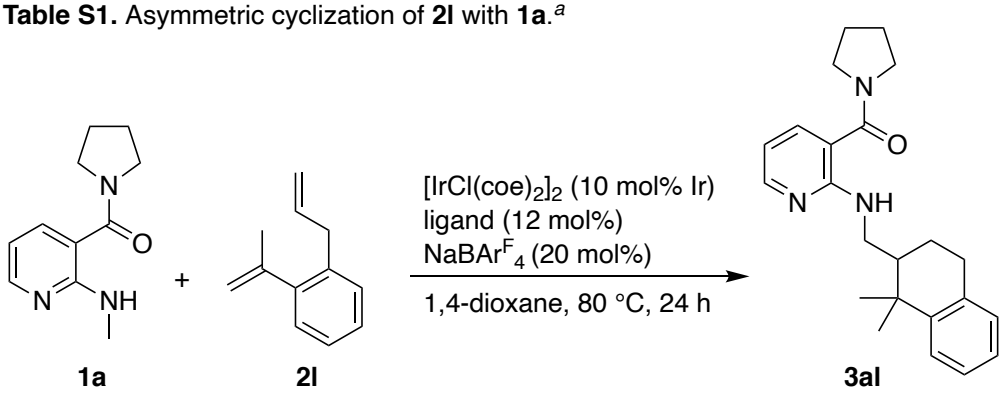
To a solution of compound **6** (89.3 mg, 0.19 mmol) in CH_2Cl_2 (0.7 mL) was added methyl trifluoromethanesulfonate (21.1 μL, 0.29 mmol) at 0 °C, and the mixture was stirred at the same temperature for 20 h. The solvent was removed under vacuum, and the residue was dissolved in methanol (0.7 mL). Sodium methoxide solution (28% in MeOH, 0.2 mL) was added to the mixture at room temperature, and the mixture was stirred at 60 °C for 23 h. The solvent was removed under vacuum and the residue was dissolved in CH_2Cl_2. The solution was washed with brine, dried over Na_2SO_4, filtered, and concentrated under vacuum. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:15) to give **7** (35.7 mg, 0.12 mmol, 64% yield, colorless oil). **Compound 7**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06–7.14 (m, 3H), 7.00–7.05 (m, 1H), 4.57 (br s, 1H), 3.40–3.58 (m, 1H), 2.93 (dd, *J* = 17.2, 5.4 Hz, 1H), 2.83–2.93 (m, 2H), 2.65 (d, *J* = 16.5 Hz, 1H), 2.55 (dd, *J* = 17.2, 10.2 Hz, 1H), 2.48 (d, *J* = 16.5 Hz, 1H), 1.68–1.80 (m, 1H), 1.46 (s, 9H), 1.10 (s,

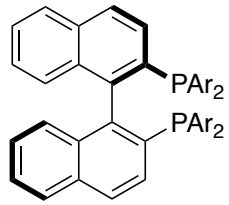
3H), 0.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 135.9, 134.9, 129.1, 128.8, 125.7, 125.5, 79.2, 44.8, 43.2, 41.6, 31.7, 30.6, 28.8, 28.4, 21.2. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{29}NO_2$ 290.2120; Found 290.2127.

11. Ligand Screening

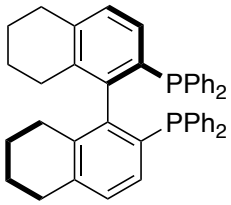
$NaBAr^F_4$ (18.4 mg, 0.0200 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir), ligand (0.012 mmol), and 1,4-dioxane (2.0 mL for Table S1, 0.4 mL for Table S2) were added, and the mixture was stirred at room temperature for 10 min. Then, **1a** (20.5 mg, 0.10 mmol) and **2l** (23.7 mg, 0.15 mmol for Table S1) or **2a** (14.4 mg, 0.15 mmol for Table S2) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with a solution of EtOAc and hexane (1:2) to give **3**. The ee of **3** was determined by HPLC analysis with a chiral stationary column: Chiralcel OD-H.

Table S1. Asymmetric cyclization of **2l** with **1a**.[a]

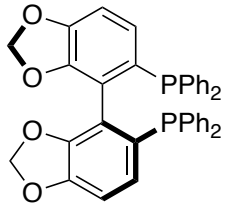




(*R*)-binap (Ar = Ph)
(*R*)-xyl-binap (Ar = 3,5-$Me_2C_6H_3$)
(*R*)-dtbm-binap
(Ar = 3,5-(*t*-Bu)$_2$-4-$MeOC_6H_2$)



(*R*)-H_8-binap

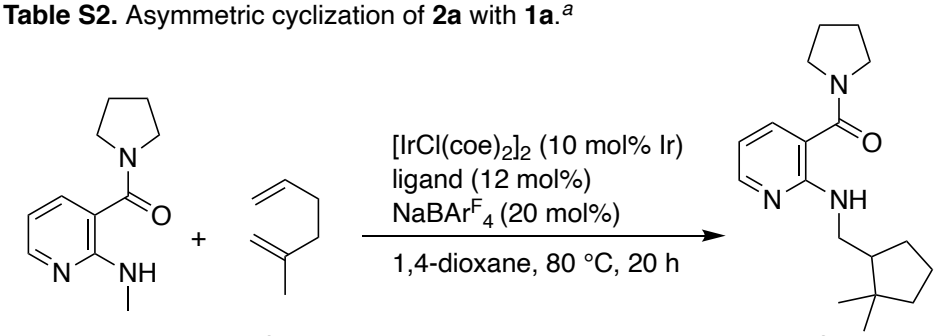


(*S*)-segphos

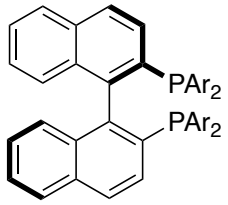
entry	ligand	yield (%)[b]	ee (%)[c]
1	(*R*)-binap	79	75
2	(*R*)-xyl-binap	81	84
3	(*R*)-dtbm-binap	49[d]	25
4	(*R*)-H_8-binap	73	74
5	(*S*)-segphos	43[d]	42

[a]Reaction conditons: **1a** (0.10 mmol), **2l** (0.15 mmol), $[IrCl(coe)_2]_2$ (0.0050 mmol, 10 mol% of Ir), ligand (0.012 mmol), and $NaBAr^F_4$ (0.020 mmol) in 1,4-dioxane (2.0 mL) at 80 °C for 24 h. [b]Isolated yield. [c]Determined by HPLC analysis with a chiral stationary phese column: Chiralcel OD-H. [d]Determined by ^1H NMR.

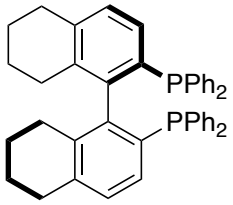
Table S2. Asymmetric cyclization of **2a** with **1a**.[a]



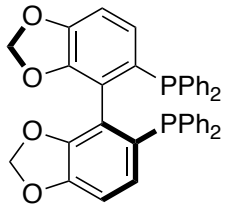
1a **2a** **3aa**



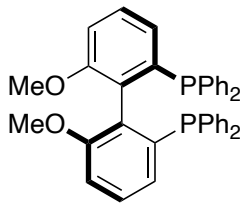
(*R*)-binap (Ar = Ph)
(*R*)-tol-binap (Ar = *p*-tol)
(*R*)-xyl-binap (Ar = 3,5-$Me_2C_6H_3$)
(*R*)-dtbm-binap
(Ar = 3,5-(*t*-Bu)$_2$-4-$MeOC_6H_2$)



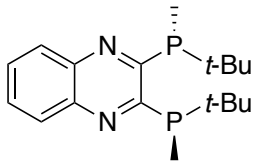
(*R*)-H_8-binap



(*S*)-segphos



(*R*)-MeO-biphep



(*R*,*R*)-QuinoxP*

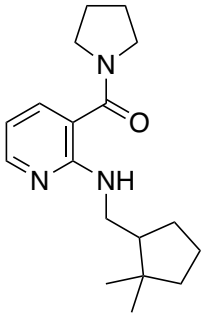
entry	ligand	yield (%)[b]	ee (%)[c]
1	(*R*)-binap	56	31
2	(*R*)-tol-binap	46	29
3	(*R*)-xyl-binap	74	36
4	(*R*)-dtbm-binap	84	1
5	(*R*)-H_8-binap	60	56
6	(*S*)-segphos	82	0
7	(*R*)-MeO-biphep	40	0
8	(*R*,*R*)-QuinoxP*	85	9

[a]Reaction conditons: **1a** (0.10 mmol), **2a** (0.15 mmol), $[IrCl(coe)_2]_2$ (0.0050 mmol, 10 mol % of Ir), ligand (0.012 mmol), and $NaBAr^F_4$ (0.020 mmol) in 1,4-dioxane (0.4 mL) at 80 °C for 20 h. [b]Determined by ^1H NMR. [c]Determined by HPLC analysis with a chiral stationary phese column: Chiralcel OD-H.

12. Procedure for Scheme 4

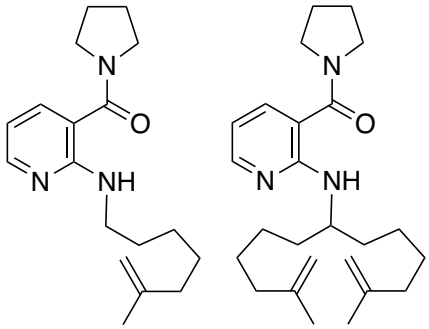
$NaBAr^F_4$ (18.4 mg, 0.0200 mmol, 20 mol%) in a Schlenk tube with a Teflon valve was dried under vacuum at 120 °C for 1 h. After the tube was cooled to room temperature under N_2, $[IrCl(coe)_2]_2$ (4.5 mg, 0.0050 mmol, 10 mol% of Ir), (*R*)-xyl-binap (8.8 mg, 0.012 mmol), and 1,4-dioxane (2.0 mL) were added to the tube, and the mixture was stirred at room temperature for 10 min. Then, **1a** (20.5 mg, 0.10 mmol) and **2** (0.15 mmol) were added to the tube successively, and the mixture was stirred at 80 °C for 20 h. The mixture was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with a solution of EtOAc and hexane to give **3**. The ee of **3** was determined by HPLC analysis with chiral stationary columns.

13. Characterization of the products



3aa

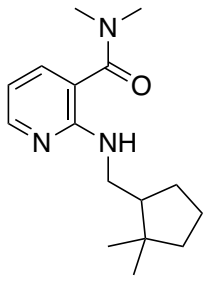
Compound 3aa (Scheme 2; colorless oil, 50.8 mg, 85% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.41 (dd, *J* = 7.4, 1.8 Hz, 1H), 6.46 (dd, *J* = 7.4, 5.0 Hz, 1H), 6.53–6.43 (m, 1H), 3.48 (dt, *J* = 12.5, 4.8 Hz, 1H), 3.75–3.33 (m, 4H), 3.17 (ddd, *J* = 12.5, 9.4, 4.8 Hz, 1H), 2.01–1.77 (m, 5H), 1.75–1.66 (m, 1H), 1.64–1.50 (m, 2H), 1.48–1.36 (m, 3H), 1.05 (s, 3H), 0.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 157.0, 149.7, 135.9, 113.5, 110.0, 49.8 (br), 49.0, 46.4 (br), 42.7, 42.3, 40.4, 29.7, 28.5, 26.4 (br), 24.3 (br), 22.0, 21.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{28}N_3O$ 302.2232; Found 302.2236.



4aa **5aa**

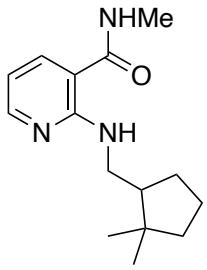
Compounds **4aa** and **5aa** were obtained in the absence of biphep using $[IrCl(cod)]_2$ as a catalyst (Table 1, entry 5). **Compound 4aa**: A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.41 (dd, *J* = 7.4, 1.8 Hz, 1H), 6.57–6.45 (m, 1H), 6.47 (dd, *J* = 7.4, 5.0 Hz, 1H), 4.66 (s, 1H), 4.64 (s, 1H), 3.78–3.38 (m, 4H), 3.39 (q, *J* = 6.3 Hz, 2H), 2.00 (t, *J* = 7.4 Hz, 2H), 1.98–1.81 (m, 4H), 1.68 (s, 3H), 1.63 (quint, *J* = 7.4 Hz, 2H), 1.50–1.33 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 156.9, 149.7, 146.0, 136.0, 113.6, 110.1, 109.6, 49.8 (br), 46.2 (br), 41.3, 37.7, 29.3, 27.4, 26.8, 26.4 (br), 24.4 (br), 22.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{18}H_{28}N_3O$ 302.2232; Found 302.2234. **Compound 5aa**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 4.9, 1.7 Hz, 1H), 7.40 (dd, *J* = 7.4, 1.7 Hz, 1H), 6.43 (dd, *J* = 7.4, 4.9 Hz, 1H), 6.27–6.43 (m, 1H), 4.63 (s, 2H), 4.61 (s, 2H), 4.11–4.22 (m, 1H), 3.33–3.77 (m, 4H), 1.96 (t, *J* = 7.3 Hz, 4H), 1.98–1.83 (m, 4H), 1.67 (s, 6H),

1.62–1.24 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 156.8, 149.6, 146.1, 136.2, 113.1, 109.7, 109.5, 49.9, 49.8 (br), 46.3 (br), 37.7, 34.7, 27.7, 26.3 (br), 25.4, 24.4 (br), 22.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{25}H_{40}N_3O$ 398.3171; Found 398.3165.



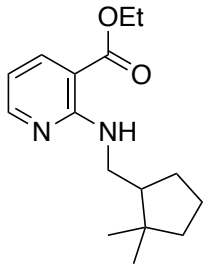
3ba

Compound 3ba (Scheme 2; colorless oil, 44.6 mg, 82% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.29 (dd, *J* = 7.3, 1.8 Hz, 1H), 6.49 (dd, *J* = 7.3, 5.0 Hz, 1H), 5.83 (br s, 1H), 3.47 (dt, *J* = 12.5, 4.9 Hz, 1H), 3.17 (ddd, *J* = 12.5, 9.4, 4.9 Hz, 1H), 3.03 (s, 6H), 1.99–1.89 (m, 1H), 1.74–1.63 (m, 1H), 1.64–1.50 (m, 2H), 1.48–1.36 (m, 3H), 1.05 (s, 3H), 0.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 156.7, 149.4, 136.1, 113.1, 110.4, 49.0, 42.9, 42.3, 40.4, 37.6 (br), 29.7, 28.5, 22.0, 21.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{16}H_{26}N_3O$ 276.2076; Found 276.2076.



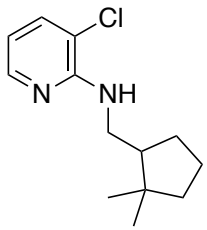
3ca

Compound 3ca (Scheme 2; colorless solid, 35.9 mg, 69% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 4.9, 1.7 Hz, 1H), 8.06 (br s, 1H), 7.51 (dd, *J* = 7.6, 1.7 Hz, 1H), 6.40 (dd, *J* = 7.6, 4.9 Hz, 1H), 6.28 (br s, 1H), 3.51 (dt, *J* = 12.7, 4.8 Hz, 1H), 3.19 (ddd, *J* = 12.7, 9.5, 4.8 Hz, 1H), 2.92 (d, *J* = 4.8 Hz, 3H), 2.05–1.93 (m, 1H), 1.79–1.68 (m, 1H), 1.68–1.50 (m, 2H), 1.49–1.38 (m, 3H), 1.06 (s, 3H), 0.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 158.0, 151.7, 134.9, 109.9, 109.8, 49.0, 42.5, 42.3, 40.3, 29.8, 28.6, 26.6, 22.0, 21.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{15}H_{24}N_3O$ 262.1919; Found 262.1917.



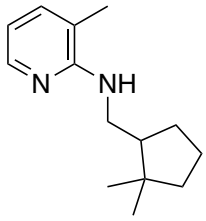
3da

Compound 3da (Scheme 2; colorless oil, 40.8 mg, 78% yield). A solution of EtOAc and hexane (1:15) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 4.4, 2.0 Hz, 1H), 8.10 (dd, *J* = 7.8, 2.0 Hz, 1H), 7.95 (br s, 1H), 6.48 (dd, *J* = 7.8, 4.4 Hz, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 3.58 (dt, *J* = 12.8, 5.2 Hz, 1H), 3.27 (ddd, *J* = 12.8, 8.7, 4.1 Hz, 1H), 2.07–1.96 (m, 1H), 1.83–1.42 (m, 6H), 1.36 (t, *J* = 7.2 Hz, 3H), 1.09 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 158.7, 153.5, 139.9, 110.4, 105.9, 60.7, 49.0, 42.5, 42.3, 40.4, 29.8, 28.6, 22.0, 21.4, 14.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{15}H_{25}N_2O$ 277.1916; Found 277.1913.



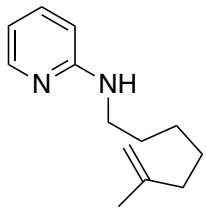
3ea

Compound 3ea (Scheme 2; colorless oil, 40.0 mg, 84% yield). A solution of EtOAc and hexane (1:10) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.41 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.48 (dd, *J* = 8.0, 4.8 Hz, 1H), 4.87 (br s, 1H), 3.53 (dt, *J* = 12.8, 5.3 Hz, 1H), 3.27 (ddd, *J* = 12.8, 8.4, 4.2 Hz, 1H), 2.12–1.92 (m, 1H), 1.80–1.40 (m, 6H), 1.09 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.2, 146.0, 135.7, 115.3, 112.3, 49.1, 43.1, 42.3, 40.5, 29.6, 28.6, 22.0, 21.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{20}N_2$ 239.1315; Found 239.1309.



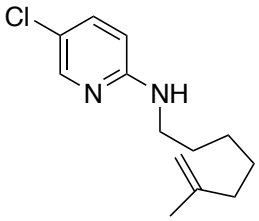
3fa

Compound 3fa (Scheme 2; colorless oil, 7.5 mg obtained in the reaction of 0.10 mmol of **1f**, 34% yield). A solution of EtOAc and hexane (1:5) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (dd, *J* = 5.1, 1.4 Hz, 1H), 7.19 (d, *J* = 7.2 Hz, 1H), 6.49 (dd, *J* = 7.2, 5.1 Hz, 1H), 3.97 (br s, 1H), 3.61–3.49 (m, 1H), 3.33–3.16 (m, 1H), 2.06 (s, 3H), 2.01–1.89 (m, 1H), 1.79–1.41 (m, 6H), 1.10 (s, 3H), 0.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 145.5, 136.5, 116.3, 112.2, 49.4, 43.2, 42.4, 40.5, 29.7, 28.8, 22.1, 21.4, 16.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{14}H_{23}N_2$ 219.1861; Found 219.1853.



4ga

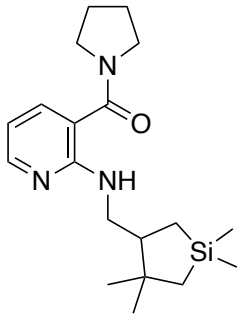
Compound 4ga (Scheme 2; colorless oil, 30.3 mg, 74% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 8.06 (dd, *J* = 5.3, 1.2 Hz, 1H), 7.40 (ddd, *J* = 8.4, 6.6, 1.2 Hz, 1H), 6.54 (dd, *J* = 6.6, 5.3 Hz, 1H), 6.36 (d, *J* = 8.4 Hz, 1H), 4.69 (s, 1H), 4.65 (s, 1H), 4.62–4.52 (m, 1H), 3.23 (q, *J* = 6.4 Hz, 2H), 2.01 (t, *J* = 7.3 Hz, 2H), 1.70 (s, 3H), 1.63 (quint, *J* = 7.3 Hz, 2H), 1.52–1.35 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 148.0, 145.8, 137.4, 112.5, 109.8, 106.3, 42.2, 37.6, 29.4, 27.2, 26.6, 22.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{21}N_2$ 205.1705; Found 205.1704.



4ha

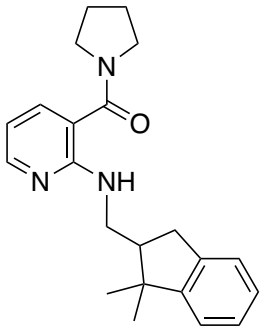
Compound 4ha (Scheme 2; colorless oil, 18.0 mg obtained in the reaction of 0.10 mmol of **1h**, 75% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR ($CDCl_3$) δ 8.01 (d, *J* = 2.8 Hz, 1H), 7.35 (dd, *J* = 9.2, 2.8 Hz, 1H), 6.31 (d,

J = 9.2 Hz, 1H), 4.70–4.64 (m, 2H), 4.52 (br s, 1H), 3.26–3.18 (m, 2H), 2.02 (t, J = 7.6 Hz, 2H), 1.70 (s, 3H), 1.63 (quint, J = 7.6 Hz, 2H), 1.52–1.34 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 146.5, 145.8, 137.1, 119.3, 109.9, 107.1, 42.4, 37.6, 29.3, 27.2, 26.6, 22.3. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{13}H_{20}{}^{35}ClN_2$ 239.1315; Found 239.1306.



3ab

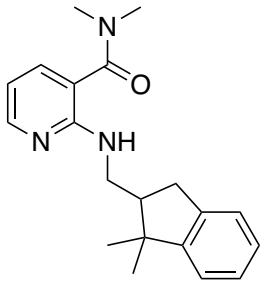
Compound 3ab (Table 2, entry 1; colorless oil, 45.1 mg, 65% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, J = 5.0, 1.8 Hz, 1H), 7.41 (dd, J = 7.4, 1.8 Hz, 1H), 6.56 (br s, 1H), 6.46 (dd, J = 7.4, 5.0 Hz, 1H), 3.69 (ddd, J = 12.3, 5.8, 4.0 Hz, 1H), 3.70–3.34 (m, 4H), 3.05 (ddd, J = 12.3, 10.3, 4.0 Hz, 1H), 2.02–1.79 (m, 4H), 1.69–1.60 (m, 1H), 1.27 (s, 3H), 1.03 (dd, J = 14.6, 6.9 Hz, 1H), 0.83 (s, 3H), 0.68 (d, J = 14.6 Hz, 1H), 0.52 (dd, J = 14.6 Hz, 1H), 0.48 (dd, J = 14.6, 11.7 Hz, 1H), 0.13 (s, 3H), 0.07 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 157.1, 149.8, 136.0, 113.5, 109.9, 49.8 (br), 49.0, 46.2 (br), 44.4, 40.9, 32.1, 31.4, 26.5 (br), 24.3 (br), 23.2, 16.9, –0.3, –0.8. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{19}H_{32}N_3OSi$ 346.2315; Found 346.2312.



3ac

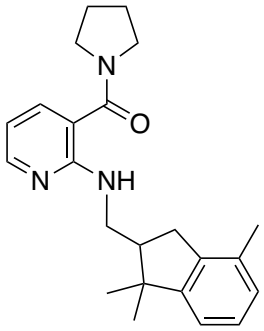
Compound 3ac (Table 2, entry 2; colorless oil, 69.6 mg, 73% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (dd, J = 5.0, 1.8 Hz, 1H), 7.47 (dd, J = 7.4, 1.8 Hz, 1H), 7.22–7.10 (m, 4H), 6.84– 6.70 (m, 1H), 6.52 (dd, J = 7.4, 5.0 Hz, 1H), 3.73 (dt, J = 12.7, 5.5 Hz, 1H), 3.70–3.44 (m, 5H), 3.11 (dd, J = 15.6, 7.6 Hz, 1H), 3.11 (dd, J = 15.6, 7.6 Hz, 1H), 2.46–2.35 (m, 1H), 2.05–1.83 (m, 4H), 1.41 (s, 3H), 1.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 156.9, 152.9, 149.4, 140.9,

136.2, 126.4, 126.2., 124.4, 121.8, 113.6, 110.3, 50.1, 50.0 (br), 46.3 (br), 45.1, 42.1, 35.4, 27.5, 26.5 (br), 24.3 (br), 23.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{28}N_3O$ 350.2232; Found 350.2233.



3bc

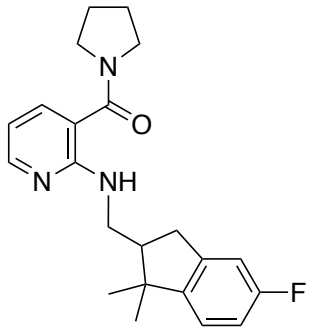
Compound 3bc (Table 2, entry 4; colorless oil, 49.0 mg, 76% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 4.8, 2.0 Hz, 1H), 7.33 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.21–7.06 (m, 4H), 6.54 (dd, *J* = 7.6, 4.8 Hz, 1H), 6.11–5.90 (m, 1H), 3.71 (dt *J* = 13.2, 5.4 Hz, 1H), 3.48 (ddd, *J* = 13.2, 8.7, 4.1 Hz, 1H), 3.13–3.04 (m, 7H), 2.75 (dd *J* = 15.8, 10.2 Hz, 1H), 2.45–2.34 (m, 1H), 1.40 (s, 3H), 1.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 156.9, 152.9, 149.7, 140.9, 136.1, 126.5, 126.3, 124.4, 121.8, 113.0, 110.7, 50.2, 45.1, 42.0, 40.2–33.0 (br, 2C), 35.4, 27.6, 23.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{20}H_{26}N_3O$ 324.2076; Found 324.2062.



3ad

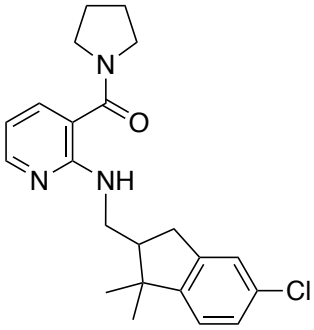
Compound 3ad (Table 2, entry 4; colorless oil, 72.4 mg, 89% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.47 (dd, *J* = 7.4, 1.8 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 7.5 Hz, 1H), 6.96 (d, *J* = 7.5 Hz, 1H), 6.78–6.68 (m, 1H), 6.52 (dd, *J* = 7.4, 5.0 Hz, 1H), 3.72 (dt, *J* = 12.8, 5.2 Hz, 1H), 3.70–3.40 (m, 5H), 3.08 (dd, *J* = 15.6, 7.7 Hz, 1H), 2.61 (dd, *J* = 15.6, 10.0 Hz, 1H), 2.44–2.36 (m, 1H), 2.25 (s, 3H), 2.02–1.83 (m, 4H), 1.40 (s, 3H), 1.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 156.9, 152.7, 149.7, 139.6, 136.0, 133.6, 127.2, 126.7, 119.1, 113.5,

110.3, 50.0 (br), 49.7, 46.3 (br), 45.3, 42.1, 34.0, 27.7, 26.4 (br), 24.2 (br), 23.7, 19.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{30}N_3O$ 364.2389; Found 364.2386.



3ae

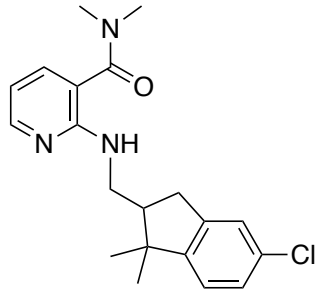
Compound 3ae (Table 2, entry 5; colorless oil, 56.1 mg, 77% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (dd, J = 5.0, 1.8 Hz, 1H), 7.46 (dd, J = 7.4, 1.8 Hz, 1H), 7.06–7.01 (m, 1H), 6.86–6.80 (m, 2H), 6.68–6.78 (m, 1H), 6.51 (dd, J = 7.4, 5.0 Hz, 1H), 3.69 (dt, J = 12.8, 5.2 Hz, 1H), 3.47 (ddd, J = 12.9, 9.2, 5.2 Hz, 1H), 3.69–3.41 (m, 4H), 3.07 (dd, J = 15.8, 7.6 Hz, 1H), 2.72 (dd, J = 15.8, 10.1 Hz, 1H), 2.46–2.36 (m, 1H), 2.02–1.83 (m, 4H), 1.37 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 161.8 (d, $J_{F–C}$ = 242 Hz), 157.0, 149.7, 148.4 (d, $J_{F–C}$ = 2 Hz), 143.0 (d, $J_{F–C}$ = 8 Hz), 136.1, 122.7 (d, $J_{F–C}$ = 9 Hz), 113.4, 113.2, (d, $J_{F–C}$ = 22 Hz), 111.3 (d, $J_{F–C}$ = 22 Hz), 110.4, 50.5, 49.9 (br), 46.3 (br), 44.5, 41.8, 35.3 (d, $J_{F–C}$ = 2 Hz), 27.7, 26.5 (br), 24.3 (br), 23.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{22}H_{27}FN_3O$ 368.2138; Found 368.2135.



3af

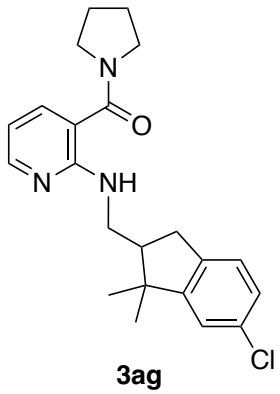
Compound 3af (Table 2, entry 6; colorless oil, 62.8 mg, 82% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, J = 5.0, 1.8 Hz, 1H), 7.46 (dd, J = 7.4, 1.8 Hz, 1H), 7.15–7.10 (m, 2H), 7.06–7.00 (m, 1H), 6.77–6.68 (m, 1H), 6.51 (dd, J = 7.4, 5.0 Hz, 1H), 3.69 (ddd, J = 12.9, 5.2 Hz, 1H), 3.47 (ddd, J = 12.9, 9.2, 5.2 Hz, 1H), 3.70–3.43 (m, 4H), 3.06 (dd, J = 15.8, 7.6 Hz, 1H), 2.71 (dd, J = 15.8, 10.0 Hz, 1H), 2.45–2.35 (m, 1H), 2.01–1.83 (m, 4H), 1.37 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 157.0, 151.4, 149.7, 142.9, 136.1, 131.7, 126.6, 124.6, 122.9, 113.5, 110.4,

50.2, 49.9 (br), 46.3 (br), 44.8, 41.8, 35.2, 27.5, 26.4 (br), 24.2 (br), 23.4. HRMS (DART) m/z: [M + H]$^+$ Calcd for $C_{22}H_{27}{}^{35}ClN_3O$ 384.1843; Found 384.1843.



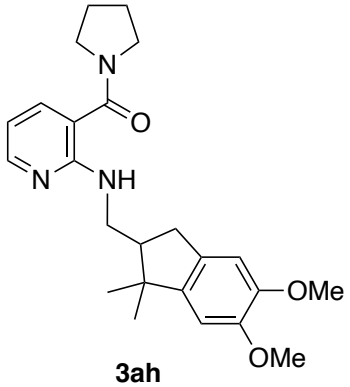
3bf

Compound 3bf (Table 2, entry 7; colorless oil, 60.2 mg, 84% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (dd, *J* = 5.0, 1.7 Hz, 1H), 7.34 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.18–7.10 (m, 2H), 7.09–7.00 (m, 1H), 6.55 (dd, *J* = 7.4, 5.0 Hz, 1H), 6.14–6.02 (m, 1H), 3.69 (td, *J* = 9.1, 4.2 Hz, 1H), 3.53–3.44 (m, 1H), 3.07 (s, 6H), 3.07–3.01 (m, 1H), 2.71 (dd, *J* = 15.8, 9.9 Hz, 1H), 2.45–2.35 (m, 1H), 1.37 (s, 3H), 1.10 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 156.7, 151.4, 149.3, 142.9, 136.3, 131.8, 126.6, 124.6, 123.0, 113.0, 110.7, 50.2, 44.8, 42.0, 38.1 (br), 35.2, 27.5, 23.5. HRMS (DART) m/z: [M + H]$^+$ Calcd for $C_{20}H_{25}{}^{35}ClN_3O$ 358.1686; Found 358.1674.



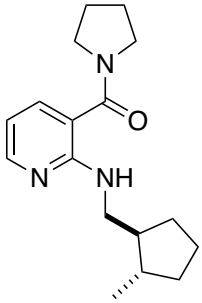
3ag

Compound 3ag (Table 2, entry 8; colorless oil, 63.1 mg, 83% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.45 (dd, *J* = 7.4, 1.8 Hz, 1H), 7.07 (s, 3H), 6.79–6.70 (m, 1H), 6.50 (dd, *J* = 7.4, 5.0 Hz, 1H), 3.69 (dt, *J* = 12.8, 5.2 Hz, 1H), 3.46 (ddd, *J* = 12.8, 9.2, 5.2 Hz, 1H), 3.69–3.40 (m, 4H), 3.05 (dd, *J* = 15.7, 7.6 Hz, 1H), 2.68 (dd, *J* = 15.7, 10.1 Hz, 1H), 2.45–2.35 (m, 1H), 2.01–1.81 (m, 4H), 1.37 (s, 3H), 1.10 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 157.0, 155.0, 149.7, 139.4, 136.0, 132.0, 126.3, 125.5, 122.2, 113.4, 110.4, 50.4, 49.9 (br), 46.4 (br), 45.4, 41.7, 34.9, 27.3, 26.4 (br), 24.3 (br), 23.3. HRMS (DART) m/z: [M + H]$^+$ Calcd for $C_{22}H_{27}{}^{35}ClN_3O$ 384.1843; Found 384.1842.



3ah

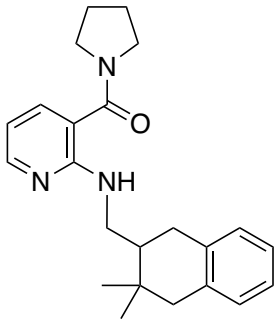
Compound 3ah (Table 2, entry 9; colorless oil, 71.2 mg, 87% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.45 (dd, *J* = 7.5, 1.8 Hz, 1H), 6.71 (s, 1H), 6.70 (br s, 1H), 6.64 (s, 1H), 6.50 (dd, *J* = 7.5, 5.0 Hz, 1H), 3.85 (s, 3H), 3.81 (s, 3H), 3.71–3.45 (m, 6H), 3.03 (dd, *J* = 15.2, 7.6 Hz, 1H), 2.67 (dd, *J* = 15.2, 9.9 Hz, 1H), 2.45–2.35 (m, 1H), 2.00–1.70 (m, 4H), 1.36 (s, 3H), 1.08 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 156.9, 149.6, 148.1, 147.8, 144.6, 136.0, 132.2, 113.5, 110.3, 107.7, 105.3, 56.0, 55.9, 50.5, 49.9 (br), 46.3 (br), 45.1, 42.0, 35.2, 27.7, 26.4 (br), 24.2 (br), 23.5. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{32}{}^{35}ClN_3O$ 410.2443; Found 410.2441.



3ai

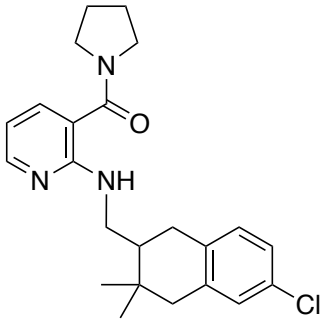
Compound 3ai (Table 2, entry 10; colorless oil, 27.6 mg isolated as a mixture of two diastereomers, 48% yield, *trans*:*cis* = 78:22). The relative stereochemistry of the major diastereomer was tentatively assigned to be *trans* by NOE experiments (S-87, 88). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ***trans*-3ai**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 5.0, 1.7 Hz, 1H, overlapped with peaks of *cis*-**3ai**), 7.44 (dd, *J* = 7.4, 1.7 Hz, 1H, overlapped with peaks of *cis*-**3ai**), 6.75–6.63 (m, 1H, overlapped with peaks of *cis*-**3ai**), 6.48 (dd, *J* = 7.4, 5.0 Hz, 1H, overlapped with peaks of *cis*-**3ai**), 3.72–3.40 (m, 4H, overlapped with peaks of *cis*-**3ai**), 3.52 (td, *J* = 8.8, 4.1 Hz, 1H), 3.35–3.28 (m, 1H), 2.03–1.80 (m, 7H, overlapped with peaks of *cis*-**3ai**), 1.70–1.50 (m, 3H, overlapped with peaks of *cis*-**3ai**), 1.40–

1.28 (m, 1H), 1.20–1.12 (m, 1H), 1.02 (d, J = 6.2 Hz, 3H); ***cis*-3ai**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, J = 5.0, 1.7 Hz, 1H, overlapped with peaks of *trans*-**3ai**), 7.44 (dd, J = 7.4, 1.7 Hz, 1H, overlapped with peaks of *trans*-**3ai**), 6.75–6.63 (m, 1H, overlapped with peaks of *trans*-**3ai**), 6.48 (dd, J = 7.4, 5.0 Hz, 1H, overlapped with peaks of *trans*-**3ai**), 3.72–3.40 (m, 4H, overlapped with peaks of *trans*-**3ai**), 3.44–3.36 (m, 1H), 3.28–3.18 (m, 1H), 2.20–2.06 (m, 1H), 2.03–1.80 (m, 6H, overlapped with peaks of *trans*-**3ai**), 1.70–1.50 (m, 3H, overlapped with peaks of *trans*-**3ai**), 1.45–1.32 (m, 1H), 1.25–1.16 (m, 1H), 0.89 (d, J = 6.8 Hz, 3H); ^{13}C NMR (*trans*-**3ai**, 100 MHz, $CDCl_3$) δ 168.5, 156.9, 149.4, 136.1, 113.6, 110.1, 49.9 (br), 47.0, 46.3 (br), 42.7, 38.5, 34.9, 30.9, 26.4 (br), 24.3 (br), 23.7, 19.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{17}H_{26}N_3O$ 288.2076; Found 288.2078.



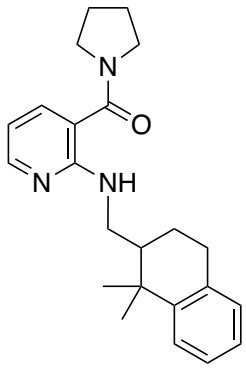
3aj

Compound 3aj (Table 2, entry 11; colorless oil, 54.5 mg, 76% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, J = 5.0, 1.7 Hz, 1H), 7.45 (dd, J = 7.4, 1.7 Hz, 1H), 7.12–7.04 (m, 3H), 7.04–6.99 (m, 1H), 6.74–6.65 (m, 1H), 6.49 (dd, J = 7.4, 5.0 Hz, 1H), 3.85–3.75 (m, 1H), 3.71–3.41 (m, 4H), 3.23 (ddd, J = 12.8, 9.5, 5.5 Hz, 1H), 3.03 (dd, J = 17.2, 5.5 Hz, 1H), 2.69–2.60 (m, 2H), 2.52 (d, J = 16.5 Hz, 1H), 2.01–1.84 (m, 5H), 1.49 (s, 3H), 0.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 157.0, 149.6, 136.1, 135.9, 135.2, 129.0, 128.9, 125.5, 125.4, 113.5, 110.1, 50.0 (br), 46.3 (br), 44.9, 42.8, 42.2, 31.9, 31.2, 28.9, 26.5 (br), 24.3 (br), 21.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{30}N_3O$ 364.2389; Found 364.2385.



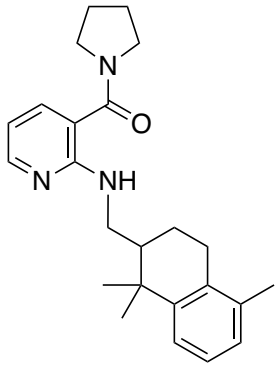
3ak

Compound 3ak (Table 2, entry 12; colorless oil, 52.6 mg, 67% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 5.0, 1.9 Hz, 1H), 7.44 (dd, *J* = 7.4, 1.9 Hz, 1H), 7.05–6.97 (m, 3H), 6.78–6.67 (m, 1H), 6.49 (dd, *J* = 7.4, 5.0 Hz, 1H), 3.81–3.71 (m, 1H), 3.70–3.38 (m, 4H), 3.21 (ddd, *J* = 12.9, 9.6, 5.6 Hz, 1H), 2.98 (dd, *J* = 17.0, 5.6 Hz, 1H), 2.61 (d, *J* = 16.8 Hz, 1H), 2.58 (dd, *J* = 17.3, 10.0 Hz, 1H), 2.47 (d, *J* = 16.8 Hz, 1H), 2.03–1.82 (m, 5H), 1.13 (s, 3H), 0.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 157.1, 149.8, 137.8, 136.1, 133.7, 130.8, 130.2, 128.7, 125.5, 113.3, 110.2, 50.0 (br), 46.4 (br), 44.6, 42.6, 42.0, 31.7, 30.6, 28.8, 26.4 (br), 24.3 (br), 21.4. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}{}^{35}ClN_3O$ 398.1999; Found 398.1992.



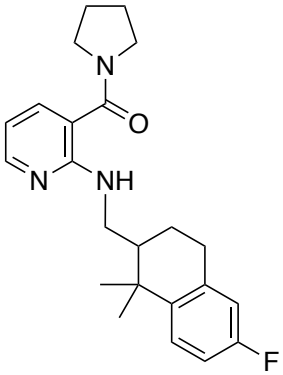
3al

Compound 3al (Table 2, entry 13; colorless oil, 53.9 mg, 74% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, *J* = 5.0, 1.7 Hz, 1H), 7.47 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.15 (t, *J* = 7.4 Hz, 1H), 7.00–7.10 (m, 2H), 6.89–6.71 (m, 1H), 6.50 (dd, *J* = 7.4, 5.0 Hz, 1H), 3.86–3.74 (m, 1H), 3.70–3.40 (m, 4H), 3.27 (ddd, *J* = 12.9, 10.1, 5.5 Hz, 1H), 2.89–2.74 (m, 2H), 2.12–1.78 (m, 6H), 1.76–1.64 (m, 1H), 1.44 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 157.2, 149.6, 146.0, 136.2, 135.7, 128.9, 126.8, 125.9, 125.2, 113.4, 110.1, 50.1 (br), 46.3 (br), 44.9, 42.1, 36.8, 30.3, 29.2, 26.4, 26.3 (br), 24.2 (br), 22.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{30}N_3O$ 364.2389; Found 364.2381.



3am

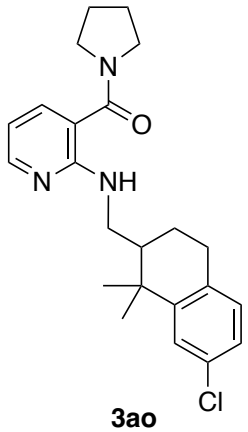
Compound 3am (Table 2, entry 14; colorless solid, 28.7 mg obtained in the reaction of 0.10 mmol of **2m**, 76% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, J = 4.9, 1.5 Hz, 1H), 7.45 (dd, J = 7.3, 1.8 Hz, 1H), 7.25 (d, J = 7.5 Hz, 1H), 7.08 (t, J = 7.5 Hz, 1H), 6.96 (d, J = 7.5 Hz, 1H), 6.76–6.67 (m, 1H), 6.49 (dd, J = 7.4, 4.9 Hz, 1H), 3.76 (ddd, J = 12.8, 5.2, 3.6 Hz, 1H), 3.70–3.47 (m, 4H), 3.24 (ddd, J = 12.8, 10.0, 5.2 Hz, 1H), 2.78–2.68 (m, 1H), 2.63–2.52 (m, 1H), 2.20 (s, 3H), 2.17–1.68 (m, 7H), 1.42 (s, 3H), 1.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.4, 150.0, 145.9, 136.0, 136.0, 134.4, 126.8, 125.4, 124.6, 113.3, 110.1, 50.0 (br), 46.3 (br), 44.2, 41.9, 36.9, 30.8, 26.6 (br), 26.5, 26.2, 24.4 (br), 22.5, 19.9. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{32}N_3O$ 378.2545; Found 378.2531.



3an

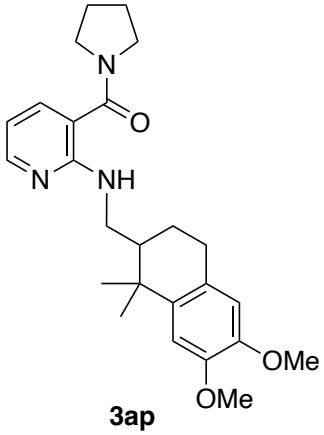
Compound 3an (Table 2, entry 15; colorless oil, 55.7 mg, 74% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, J = 4.9, 1.8 Hz, 1H), 7.45 (dd, J = 7.4, 1.8 Hz, 1H), 7.28 (dd, J = 8.6, 5.8 Hz, 1H), 6.82 (td, J = 8.6, 2.8 Hz, 1H), 6.79–6.72 (m, 1H), 6.70 (dd, J = 9.6, 2.8 Hz, 1H), 6.49 (dd, J = 7.4, 4.9 Hz, 1H), 3.75 (ddd, J = 13.0, 5.0, 3.4 Hz, 1H), 3.71–3.35 (m, 4H), 3.24 (ddd, J = 13.0, 10.0, 5.6 Hz, 1H), 2.85–2.70 (m, 2H), 2.11–1.82 (m, 5H), 1.81 (tt, J = 10.1, 3.0 Hz, 1H), 1.74–1.65 (m, 1H), 1.40 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 160.4 ($J_{F–C}$ = 243 Hz), 157.3, 149.8, 141.6 ($J_{F–C}$ = 3 Hz), 137.9 ($J_{F–C}$ = 7 Hz), 136.1, 128.4 ($J_{F–C}$ = 8 Hz), 114.5 ($J_{F–C}$ = 20 Hz),

113.3, 112.9 (J_{F-C} = 21 Hz), 110.2, 50.0 (br), 46.5 (br), 44.7, 41.8, 36.4, 30.4, 29.2, 26.5, 26.3 (br), 24.4 (br), 22.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}FN_3O$ 382.2295; Found 382.2297.



3ao

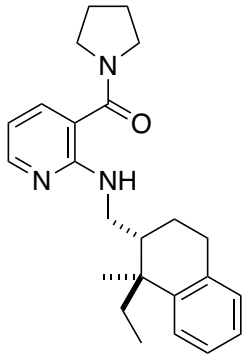
Compound 3ao (Table 2, entry 16; colorless solid, 25.0 mg obtained in the reaction of 0.10 mmol of **2o**, 63% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.45 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.30 (d, *J* = 2.4 Hz, 1H), 7.02 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.79–6.71 (m, 1H), 6.44 (dd, *J* = 7.6, 5.0 Hz, 1H), 3.75 (ddd, *J* = 12.8, 4.9, 3.5 Hz, 1H), 3.70–3.40 (m, 4H), 3.22 (ddd, *J* = 12.8, 10.0, 5.8 Hz, 1H), 2.83–2.66 (m, 2H), 2.09–2.00 (m, 1H), 2.00–1.76 (m, 5H), 1.72–1.61 (m, 1H), 1.41 (s, 3H), 1.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.3, 149.9, 148.0, 136.1, 134.2, 131.2, 130.2, 126.8, 125.4, 113.2, 110.2, 50.0 (br), 46.4 (br), 44.4, 41.8, 37.0, 30.2, 28.5, 26.7 (br), 26.2, 24.3 (br), 22.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{23}H_{29}{}^{35}ClN_3O$ 398.1999; Found 398.1995.



3ap

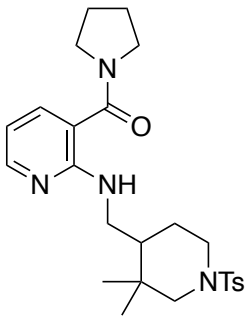
Compound 3ap (Table 2, entry 17; colorless solid, 29.5 mg obtained in the reaction of 0.10 mmol of **2p**, 70% yield). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 5.0, 2.0 Hz, 1H), 7.44 (dd, *J* = 7.6,

2.0 Hz, 1H), 6.82 (s, 1H), 7.74–6.67 (m, 1H), 6.50 (s, 1H), 6.48 (dd, *J* = 7.4, 5.0 Hz, 1H), 3.85 (s, 3H), 3.82 (s, 3H), 3.75 (ddd, *J* = 12.8, 4.8, 3.2 Hz, 1H), 3.70–3.45 (m, 4H), 3.23 (ddd, *J* = 12.8, 10.0, 5.6 Hz, 1H), 2.72 (dd, *J* = 7.2, 5.6 Hz, 1H), 2.18–1.60 (m, 8H), 1.41 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.3, 149.9, 147.1,146.7, 137.9, 136.0, 128.0, 113.3, 111.2, 110.1, 110.0, 56.0, 55.7, 49.9 (br), 46.3 (br), 44.8, 42.0, 36.5, 30.3, 28.8, 26.4 (br), 26.3, 24.3 (br), 23.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{25}H_{34}N_3O_3$ 424.2600; Found 424.2597.



3aq

Compound 3aq (Table 2, entry 18; colorless oil, 35.8 mg obtained in the reaction of 0.10 mmol of **2q**, 95% yield, *trans*:*cis* = 97:3). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. The relative stereochemistry of the major diastereomer was tentatively assigned to be *trans* by NOE experiments (S-97). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.44 (dd, *J* = 7.4, 1.6 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 1H), 7.16–7.10 (m, 1H), 7.09–7.00 (m, 2H), 6.77–6.68 (m, 1H), 6.48 (dd, *J* = 7.4, 4.8 Hz, 1H), 3.76 (ddd, *J* = 12.9, 5.4, 3.2 Hz, 1H), 3.70–3.45 (m, 4H), 3.18 (ddd, *J* = 14.7, 8.8, 4.2 Hz, 1H), 2.87–2.70 (m, 2H), 2.10–1.62 (m, 9H), 1.23 (s, 3H), 0.67 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.3, 149.9, 144.2, 137.1, 136.0, 128.8, 126.7, 125.8, 125.0, 113.3, 110.1, 49.9 (br), 46.3 (br), 41.8, 40.4, 38.8, 32.7, 29.4, 26.5 (br), 26.2, 24.3 (br), 22.5, 8.7. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{32}N_3O$ 378.2545; Found 378.2532.

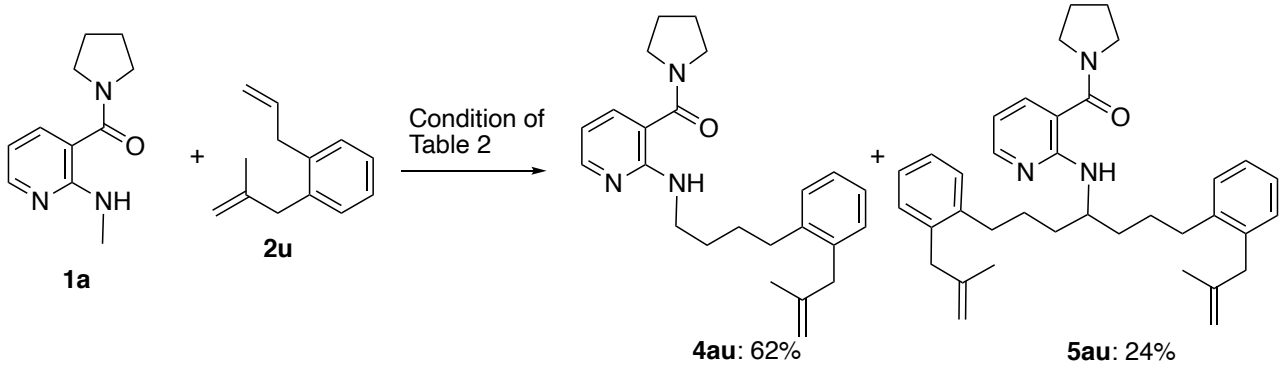


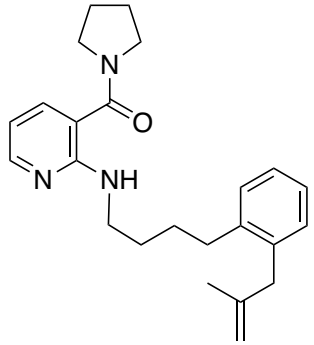
3ar

Compound 3ar (Table 2, entry 19; colorless oil, 27.8 mg obtained in the reaction of 0.10 mmol of **2r**, 59% yield). After the reaction, the inseparable mixture of **3ar** and **4ar** was obtained by preparative TLC, and the mixture was again treated with the catalytic conditions. A solution of EtOAc and hexane (1:1) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 5.0 Hz, 1H), 7.59 (d, *J* = 7.6 Hz, 2H), 7.41 (d, *J* = 5.0 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 2H), 6.59 (t, *J* = 5.2 Hz, 1H), 6.46 (ddd, *J* = 7.3, 5.0, 0.8 Hz, 1H), 3.73 (d, *J* = 11.6 Hz, 1H), 3.79–3.31 (m, 4H), 3.63 (dt, *J* = 13.6, 4.3 Hz, 1H), 3.24 (d, *J* = 11.6 Hz, 1H), 3.12–2.98 (m, 1H), 2.41 (s, 3H), 2.15 (td, *J* = 11.6, 2.5 Hz, 1H), 2.09–1.66 (m, 6H), 1.57 (qd, *J* = 12.6, 4.2 Hz, 1H), 1.34–1.20 (m, 1H), 1.02 (s, 3H), 1.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 157.2, 149.9, 143.3, 136.0, 133.0, 129.5, 127.6, 113.1, 110.3, 58.8, 49.9 (br), 46.8, 46.3 (br), 44.3, 41.4, 33.3, 26.6, 26.5 (br), 25.8, 24.2 (br), 21.4, 19.1. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{25}H_{35}N_4O_3S$ 471.2430; Found 471.2411.

14. Reaction of 1,7-diene 2u

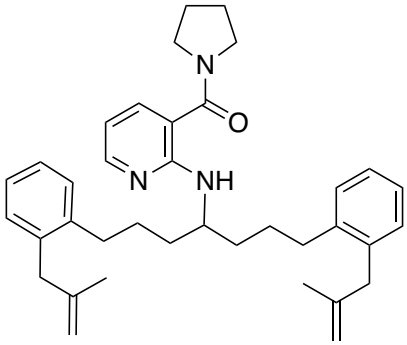
The reaction of **2u** was carried out under the reaction conditions in Table 2. The formation of the corresponding cyclized products was not observed.





4au

Compound **4au** (colorless oil, 48.5 mg, 62% yield). A solution of EtOAc and hexane (2:1) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, *J* = 5.0, 1.6 Hz, 1H), 7.43 (d, *J* = 7.2, 1.6 Hz, 1H), 7.19–6.99 (m, 4H), 6.65–6.45 (m, 1H), 6.49 (dd, *J* = 7.2, 5.0 Hz, 1H), 4.79 (s, 1H), 4.51 (s, 1H), 3.74–3.36 (m, 6H), 3.32 (s, 2H), 2.63 (t, *J* = 7.4 Hz, 2H), 2.09–1.78 (m, 4H),1.78–1.59 (m, 7H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 156.8, 149.6, 144.8, 140.8, 137.2, 136.0, 130.0, 129.1, 126.2, 125.7, 113.5, 111.7, 110.2, 49.9 (br), 46.3 (br), 41.2, 41.1, 32.4, 29.5, 28.3, 26.4 (br), 24.3 (br), 22.7. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{24}H_{32}N_3O$ 378.2545; Found 378.2540.



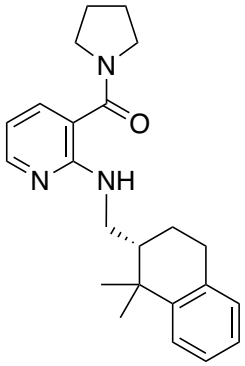
5au

Compound **5au** (Table 2, entry 20; colorless oil, 27.8 mg, 24% yield). A solution of EtOAc and hexane (2:1) was used as an eluent for preparative TLC on silica gel. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, *J* = 4.8, 1.6 Hz, 1H), 7.42 (dd, *J* = 7.4, 1.6 Hz, 1H), 7.16–7.00 (m, 8H), 6.49–6.39 (m, 2H), 4.77 (s, 2H), 4.51 (s, 2H), 4.31 (br s, 1H), 3.76–3.30 (m, 4H), 3.28 (s, 4H), 2.67–2.48 (m, 4H), 2.12–1.72 (m, 4H), 1.73–1.47 (m, 8H), 1.68 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.1, 149.9, 144.9, 141.1, 137.2, 136.2, 130.1, 129.1, 126.2, 125.7, 112.9, 111.7, 109.8, 49.5, 46.3 (br), 41.1, 35.2, 32.7, 26.5 (br), 24.4 (br), 22.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{37}H_{48}N_3O$ 550.3797; Found 550.3780.

15. Data for Scheme 4

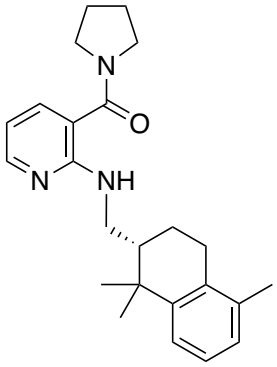
The absolute configuration of **3at** obtained by use of (*R*)-xyl-binap was determined to be (*R*)-(+) by X-ray crystallographic analysis of (*S*)-**3at** (page S-41). For others, they were assigned by analogy with **3at**.

Specific rotation values of the enantio-enriched compounds and chiral HPLC analytical conditions are Shown below.



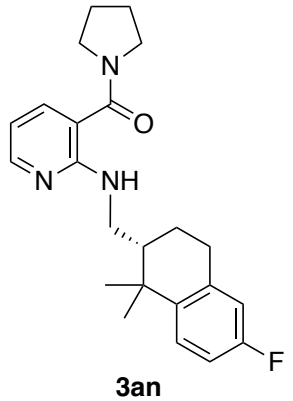
3al

Compound 3al (Scheme 3; colorless solid, 29.3 mg, 84% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 24.4min (major), t_2 = 28.7 min (minor)): $[\alpha]_D^{20}$ –7.5 (*c* 0.94, $CHCl_3$) for 84% ee (*R*).



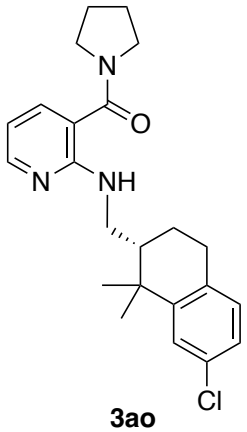
3am

Compound 3am (Scheme 3; colorless oil, 28.7 mg, 76% yield, 75% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 24.2 min (major), t_2 = 28.3 min (minor)): $[\alpha]_D^{20}$ –11.1 (*c* 1.52, $CHCl_3$) for 75% ee (*R*).



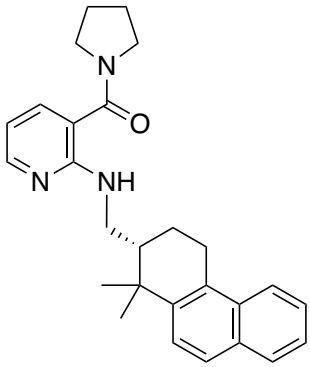
3an

Compound 3an (Scheme 3; colorless oil, 28.7 mg, 75% yield, 83% ee). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 27.7min (major), t_2 = 37.4 min (minor)): $[\alpha]_D^{20}$ –6.3 (*c* 1.35, $CHCl_3$) for 83% ee (*R*).



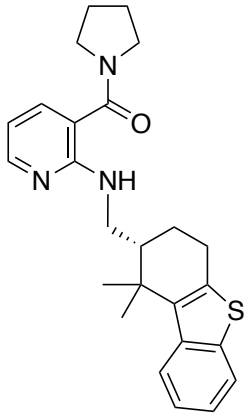
3ao

Compound 3ao (Scheme 3; colorless solid, 27.1 mg, 78% yield, 71% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 28.0 min (major), t_2 = 35.7 min (minor)): $[\alpha]_D^{20}$ –7.8 (*c* 0.29, $CHCl_3$) for 71% ee (*R*).



3as

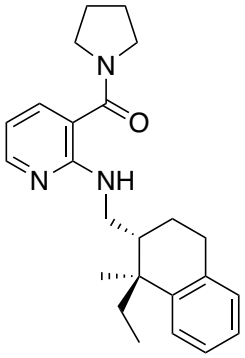
Compound 3as (Scheme 3; colorless oil, 33.4 mg, 81% yield, 83% ee). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 40.0 min (major), t_2 = 54.9 min (minor)): $[\alpha]_D^{20}$ –6.9 (*c* 1.93, $CHCl_3$) for 83% ee (*R*). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.77 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.68 (d, *J* = 8.8 Hz, 1H), 7.37–7.58 (m, 4H), 6.85–6.77 (m, 1H), 6.51 (dd, *J* = 7.4, 5.0 Hz, 1H), 3.82 (ddd, *J* = 12.8, 5.0, 3.2 Hz, 1H), 3.78–3.41 (m, 4H), 3.40–3.21 (m, 2H), 3.14–3.00 (m, 1H), 2.30–2.20 (m, 1H), 2.10–1.77 (m, 6H), 1.49 (s, 3H), 1.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.4, 149.9, 142.7, 136.1, 132.0, 131.5, 130.5, 128.0, 126.1, 125.8, 125.3, 124.9, 123.4, 113.3, 110.2, 50.0 (br), 46.3 (br), 44.3, 42.0, 37.1, 30.2, 26.5 (br), 25.8, 25.5, 24.3, 22.6. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{27}H_{32}N_3O$ 414.2545; Found 414.2536.



3at

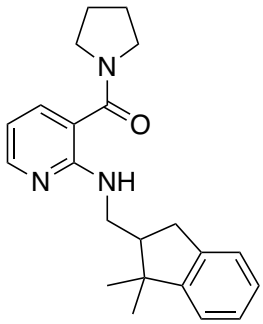
Compound 3at (Scheme 3; colorless solid, 31.5 mg, 75% yield, 72% ee). A solution of EtOAc and hexane (1:2) was used as an eluent for preparative TLC on silica gel. The ee was measured by HPLC (Chiralpak IA, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 21.1 min (minor, *S*), t_2 = 25.4 min (major, *R*)): $[\alpha]_D^{20}$ +36.3 (*c* 0.93, $CHCl_3$) for 72% ee (*R*). The enantiomerically pure (*S*)-**3at** for X-ray crystallographic analysis was obtained by preparative chiral HPLC using a Chiralpak IA column (2.0 cm I.D. × 25 cm) with hexane/EtOAc (4:1). $[\alpha]_D^{20}$ –49.7

(*c* 0.22, $CHCl_3$) for >99% ee (*R*). ^1H NMR (400 MHz, $CDCl_3$) δ 8.17 (dd, *J* = 5.0, 1.8 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.47 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.32–7.27 (m, 1H), 7.25–7.18 (m, 1H), 6.80–6.75 (m, 1H), 6.51 (dd, *J* = 7.3, 4.9 Hz, 1H), 3.86 (ddd, *J* = 12.8, 4.8, 2.8 Hz, 1H), 3.74–3.49 (m, 4H), 3.34 (ddd, *J* = 12.8, 10.0, 5.6 Hz, 1H), 2.93–2.79 (m, 1H), 2.17–2.09 (m, 1H), 2.03–1.69 (m, 7H), 1.67 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 157.4, 150.0, 139.3, 138.5, 137.3, 136.8, 136.1, 123.5, 123.2, 122.8, 122.6, 113.3, 110.3, 50.0 (br), 46.8, 46.4 (br), 41.6, 37.1, 27.2, 26.5 (br), 26.2, 24.4 (br), 23.7, 22.0. HRMS (DART) m/z: $[M + H]^+$ Calcd for $C_{25}H_{30}N_3OS$ 420.2110; Found 420.2097.



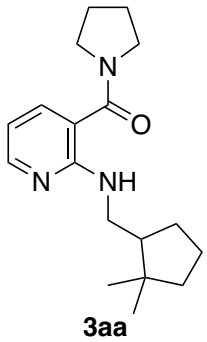
3aq

Compound 3aq (Scheme 3; colorless, 27.3 mg, 73% yield, *trans*:*cis* = 97:3, 89% ee for *trans*-isomer). The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t =38.7 min (minor), t = 42.5 min (major)): $[\alpha]_D^{20}$ +7.7 (*c* 1.44, $CHCl_3$) for 89% ee (1*S*,2*R*).



3ac

Compound 3ac (Scheme 3; colorless oil, 27.9 mg, 80% yield, 66% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 28.1 min (major), t_2 = 34.9 min (minor)): $[\alpha]_D^{20}$ –19.7 (*c* 0.86, $CHCl_3$) for 66% ee.



3aa

Compound 3aa (Scheme 3, colorless oil, 19.7 mg, 65% yield, 36% ee). The ee was measured by HPLC (Chiralcel OD-H, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 19.7 min (major), t_2 = 23.6 min (minor)): $[\alpha]_D^{20}$ –7.0 (*c* 0.94, $CHCl_3$) for 36% ee.

16. X-ray data

Compound 3ca: A colorless crystal of **3ca** suitable for X-ray crystallographic analysis was obtained by recrystallization from hexane/dichloromethane. The ORTEP drawing of **3ca** is shown in Figure S1. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 2132242). X-ray data were collected on a Rigaku/MSC Mercury CCD using a graphite monochromator with Mo-Kα radiation (λ = 0.71070 Å) at 150 K. The structure was solved by direct method (SIR97) and refined with full-matrix least-square technique (SHELXL-2014). The data for **3ca** are summarized in Table S3.

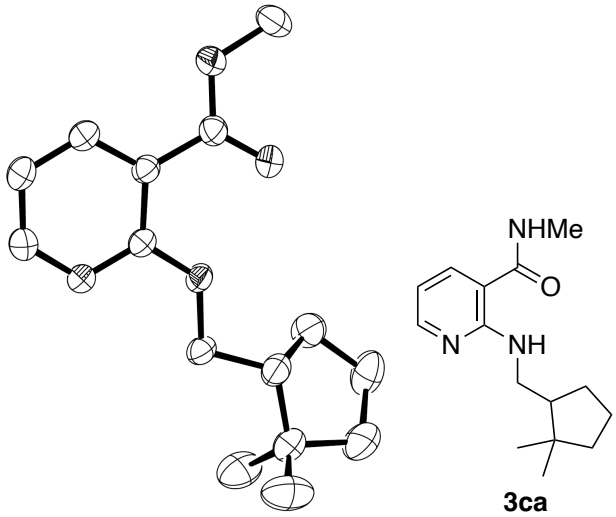


Figure S1. ORTEP illustration of (*S*)-**3ca** with thermal ellipsoids drawn at 50% probability level.

Table S3. Crystal data and structure refinement for **3ca**.

Empirical formula	$C_{15}H_{23}N_3O$
Formula weight	261.36
Temperature	150(2) K
Crystal system	Orthorhombic
Space group	P b c a (#61)
Unit cell dimensions	a = 15.847(3) Å
	b = 15.797(3) Å
	c = 23.662(5) Å
Volume	5924(2) $Å^3$
Z	16
Density (calculated)	1.172 Mg/m^3
F(000)	2272
Reflections collected	40477

Independent reflections	6125 [R(int) = 0.0527]
Completeness to θ = 25.242°	99.8 %
Refinement method	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	1.005
Final R indices [I>2sigma(I)]	R_1 = 0.0850, wR_2 = 0.2246
R indices (all data)	R_1 = 0.0966, wR_2 = 0.2361
Largest diff. peak and hole	0.774 and –0.380 e.Å^{-3}

Compound 3at: A colorless crystal of enantiopure **3at** suitable for X-ray crystallographic analysis was obtained by recrystallization from methanol/dichloromethane. The ORTEP drawing of (*S*)-**3at** is shown in Figure S2. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 2132243). X-ray data were collected on a Rigaku AFC11 with Saturn 724+ CCD using a graphite monochromator with Mo-Kα radiation (λ = 0.71075 Å) at 100 K. The structure was solved by direct method (SIR97) and refined with full-matrix least-square technique (SHELXL-2018/3). The data for (*S*)-**3at** are summarized in Table S4.

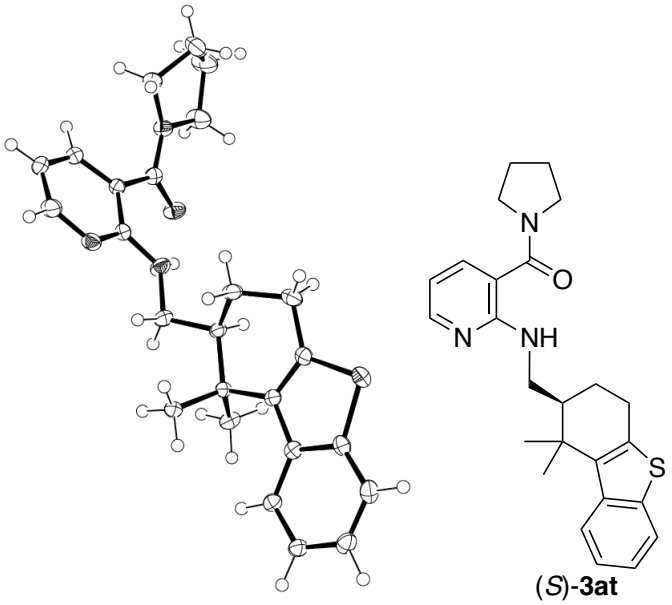


Figure S2. ORTEP illustration of (*S*)-**3at** with thermal ellipsoids drawn at 50% probability level.

Table S4. Crystal data and structure refinement for **3at**.

Empirical formula	$C_{25}H_{29}N_3OS$
Formula weight	419.57
Temperature	100(2) K
Crystal system	Orthorhombic
Space group	$P2_12_12_1$ (#19)
Unit cell dimensions	a = 7.764(3) Å
	b = 15.357(5) Å
	c = 18.436(6) Å
Volume	2198.1(13) $Å^3$
Z	4
Density (calculated)	1.268 Mg/m^3
Absorption coefficient	0.169 mm^{-1}
F(000)	896
Reflections collected	22594
Independent reflections	4988 [R(int) = 0.0754]
No. of parameters	387
Completeness to θ = 25.242°	99.4 %
Refinement method	Full-matrix least-squares on F^2
Goodness-of-fit on F^2	0.994
Final R indices [I>2sigma(I)]	R_1 = 0.0367, wR_2 = 0.0774
R indices (all data)	R_1 = 0.0420, wR_2 = 0.0802
Absolute structure parameter	–0.09(5)
Largest diff. peak and hole	0.215 and –0.181 e.$Å^{-3}$

17. References

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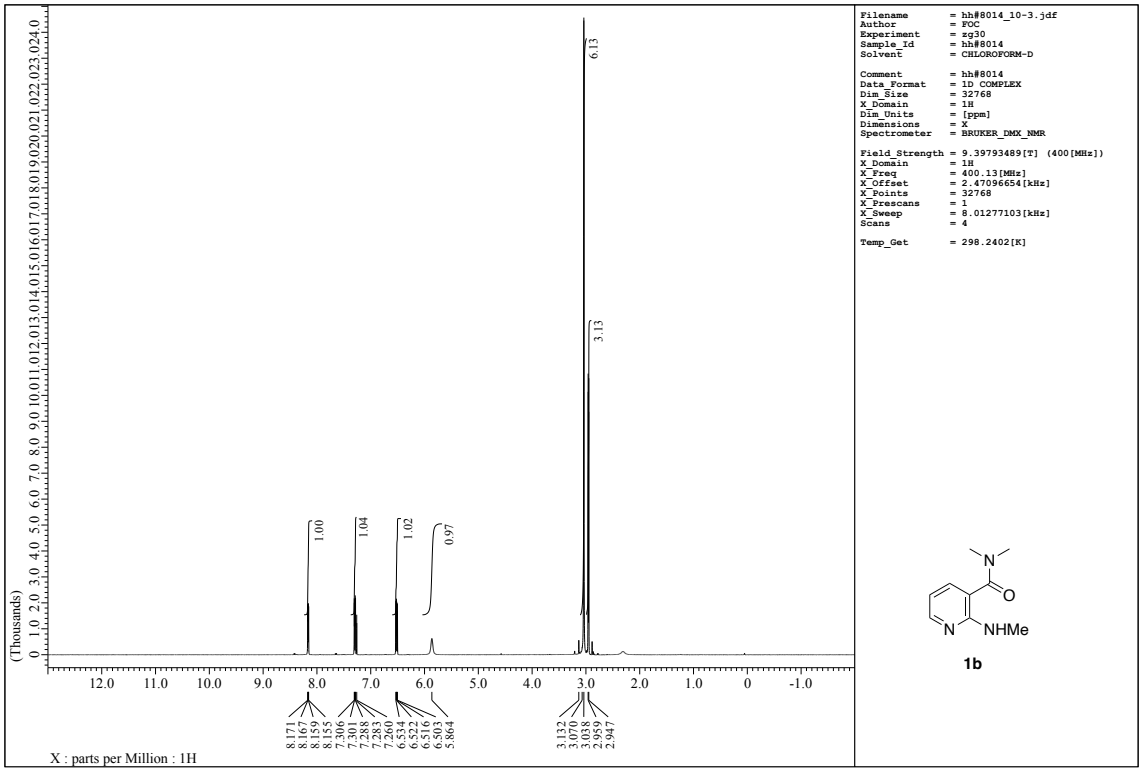
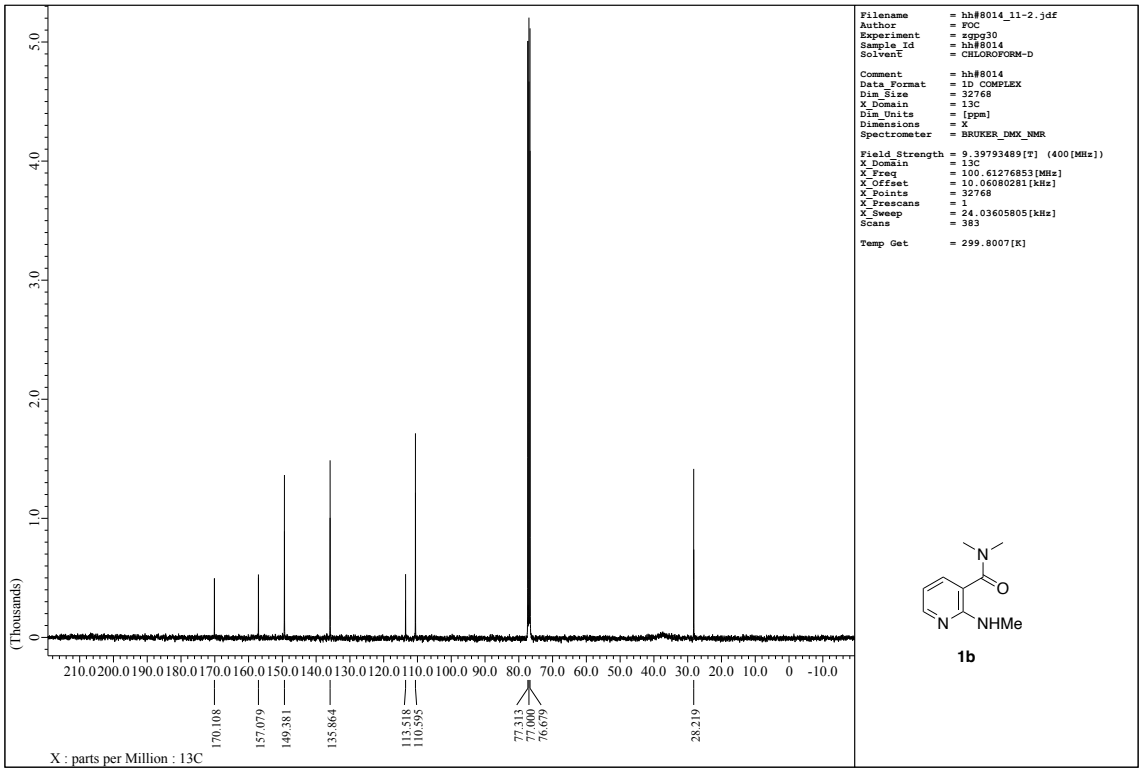
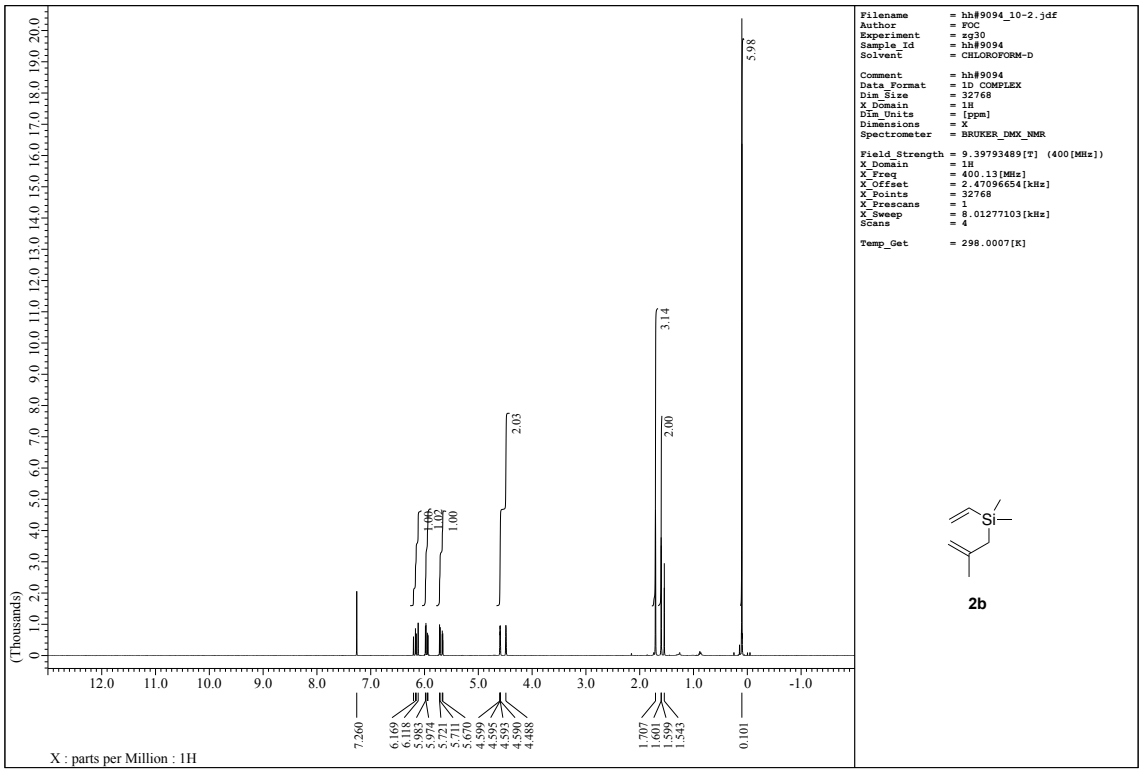
18. ^{1}H and ^{13}C NMR spectra



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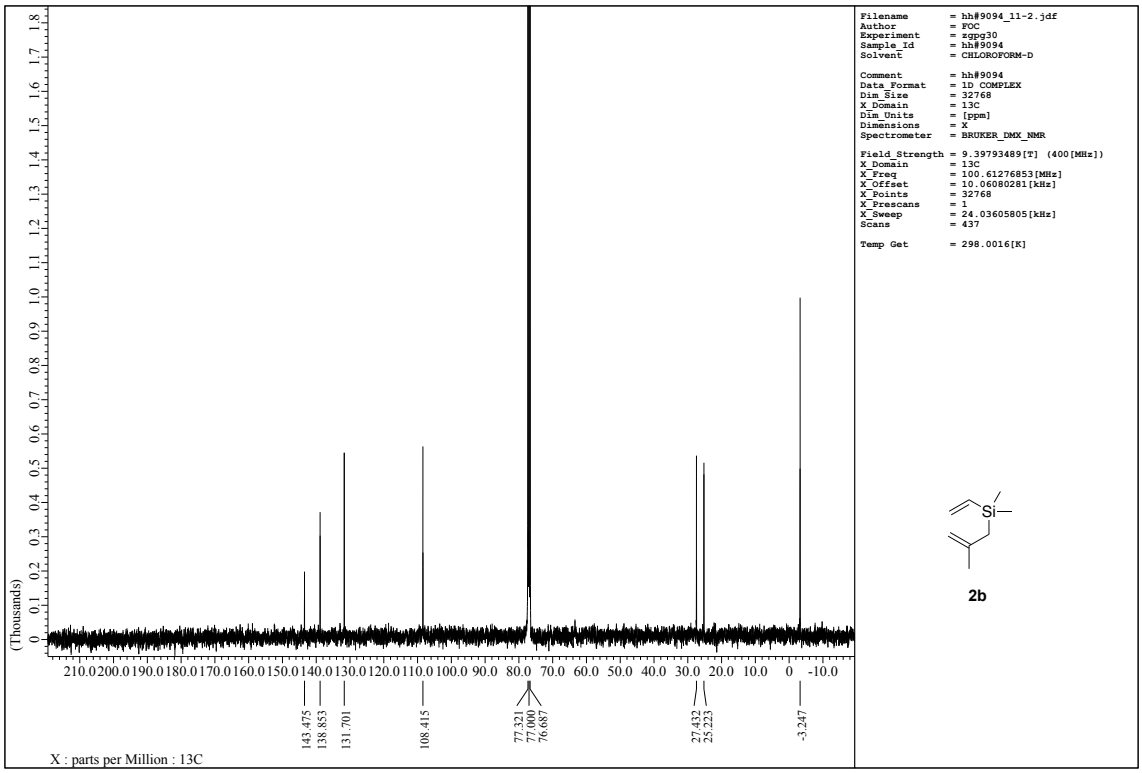
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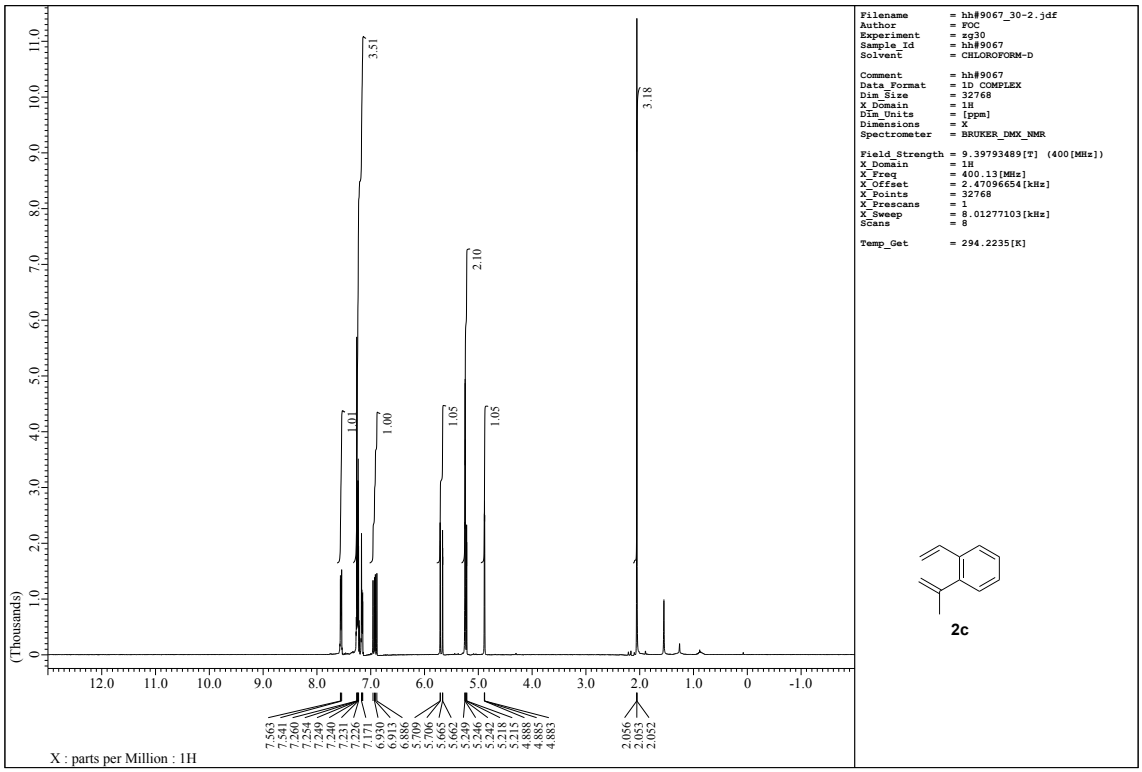
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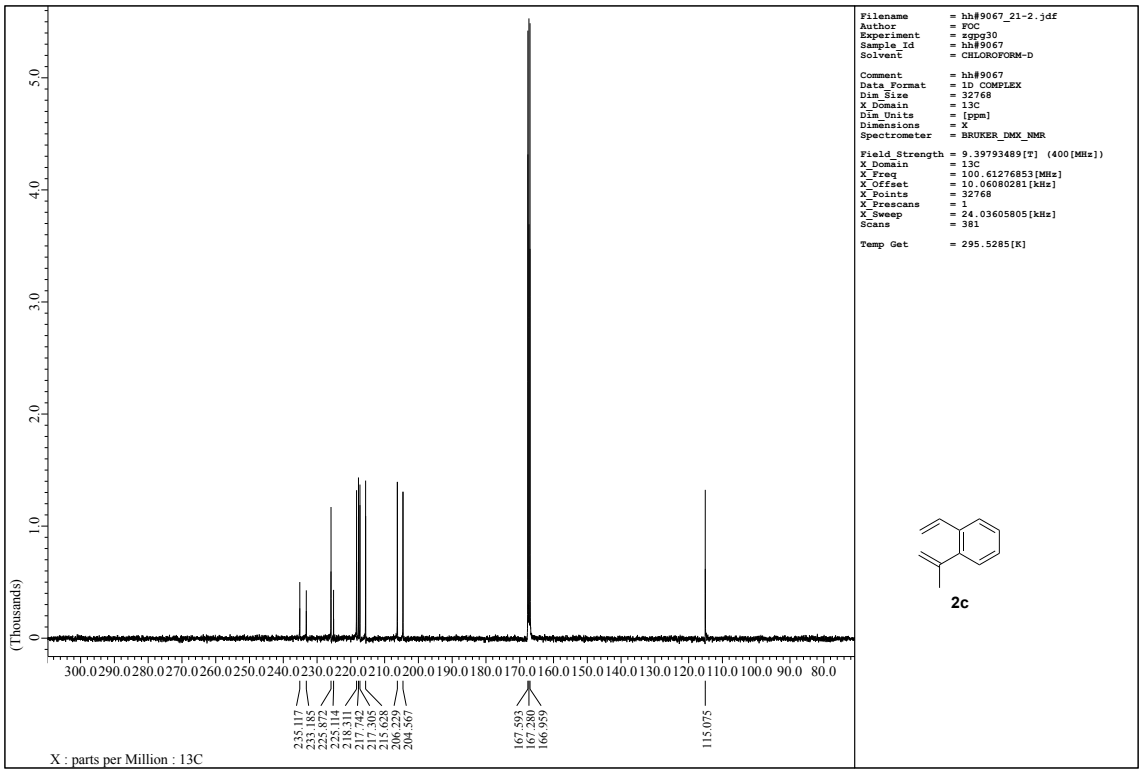
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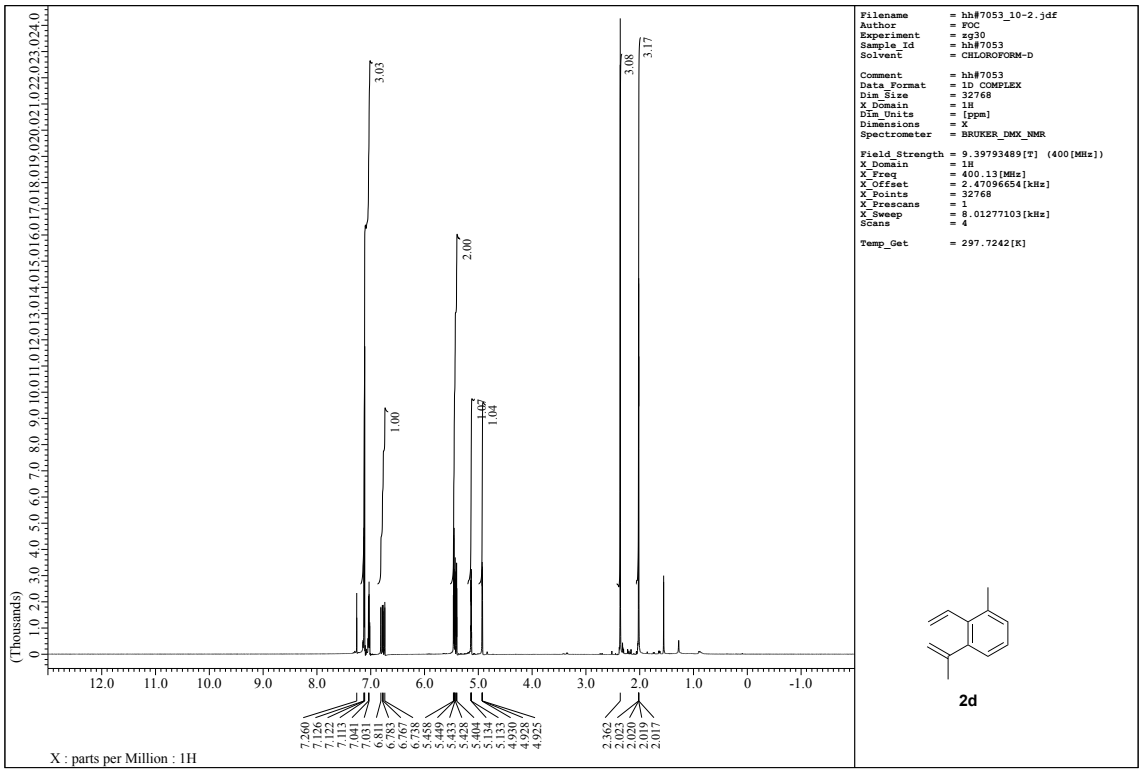
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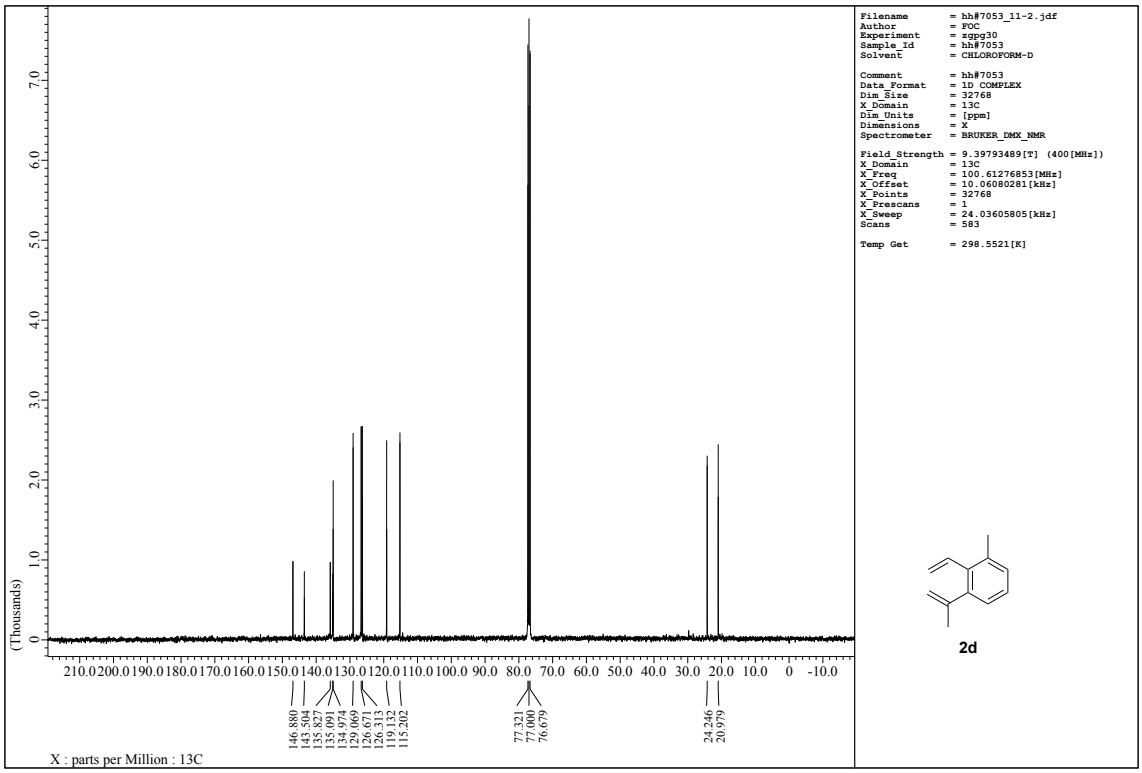
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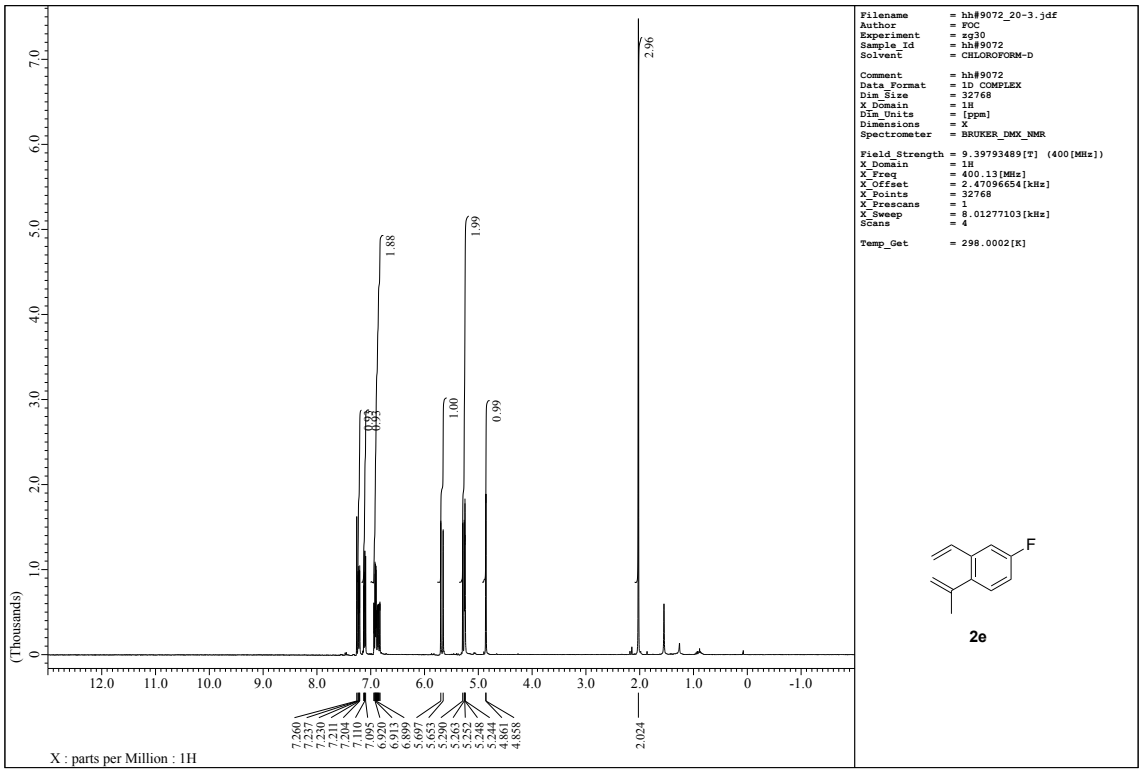
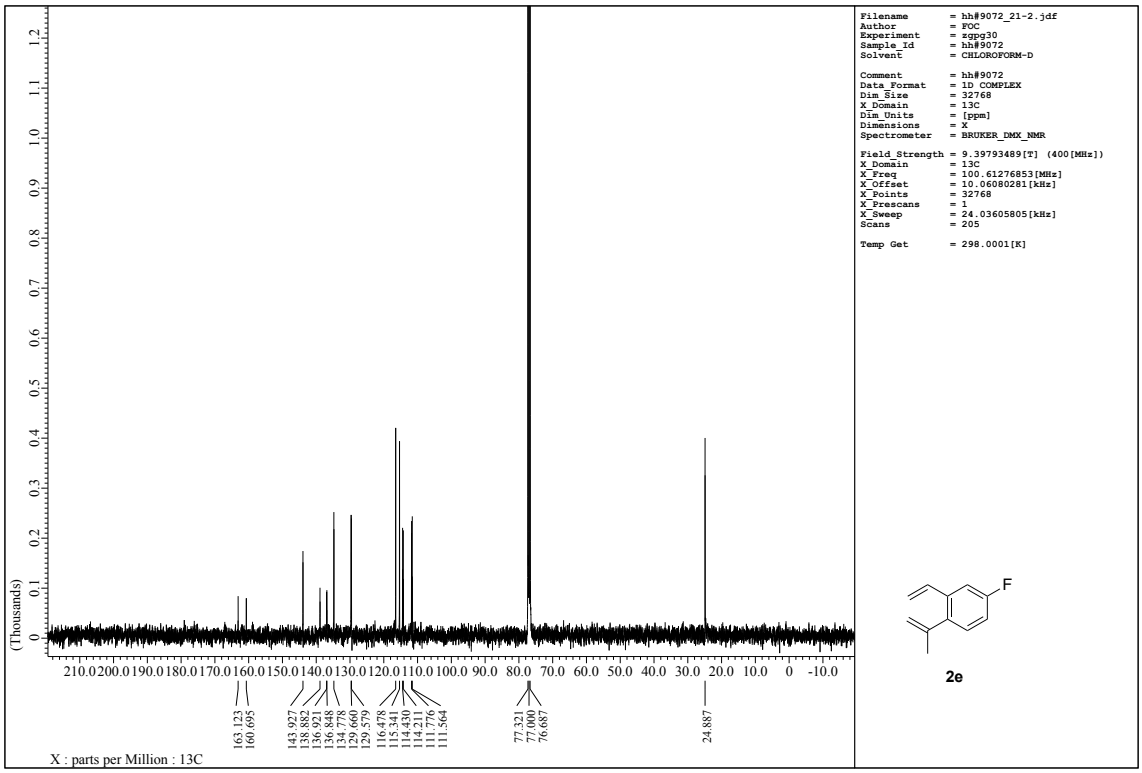
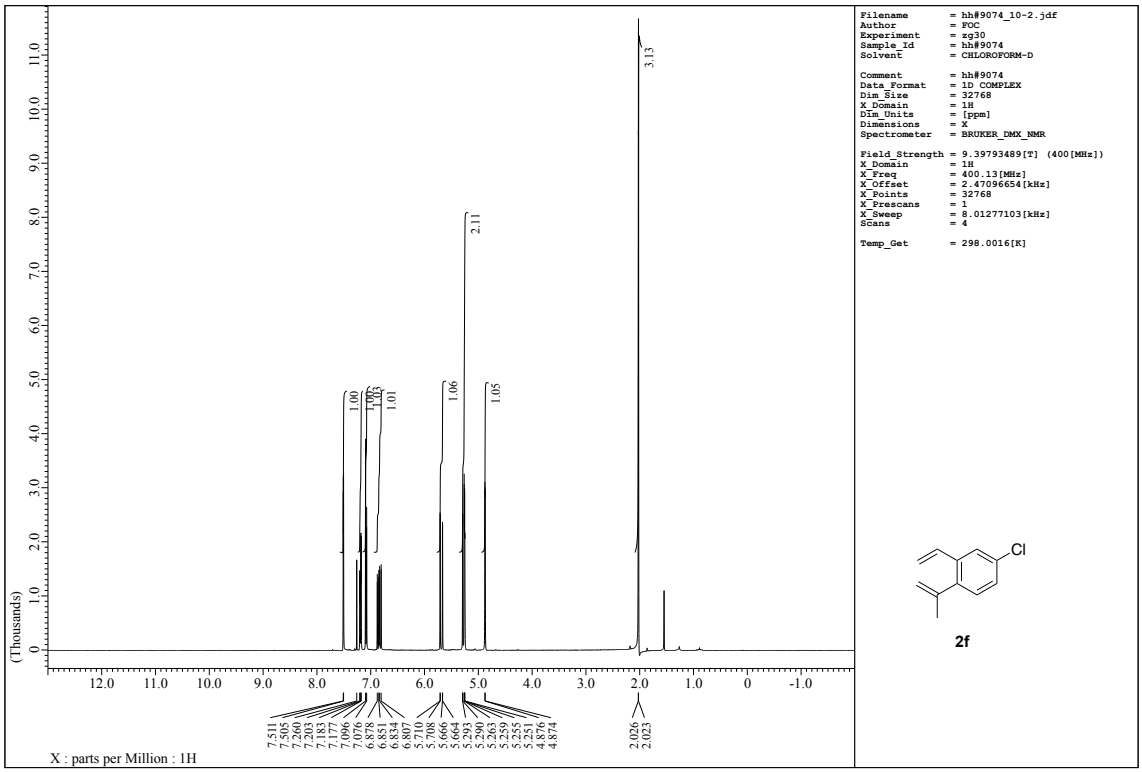
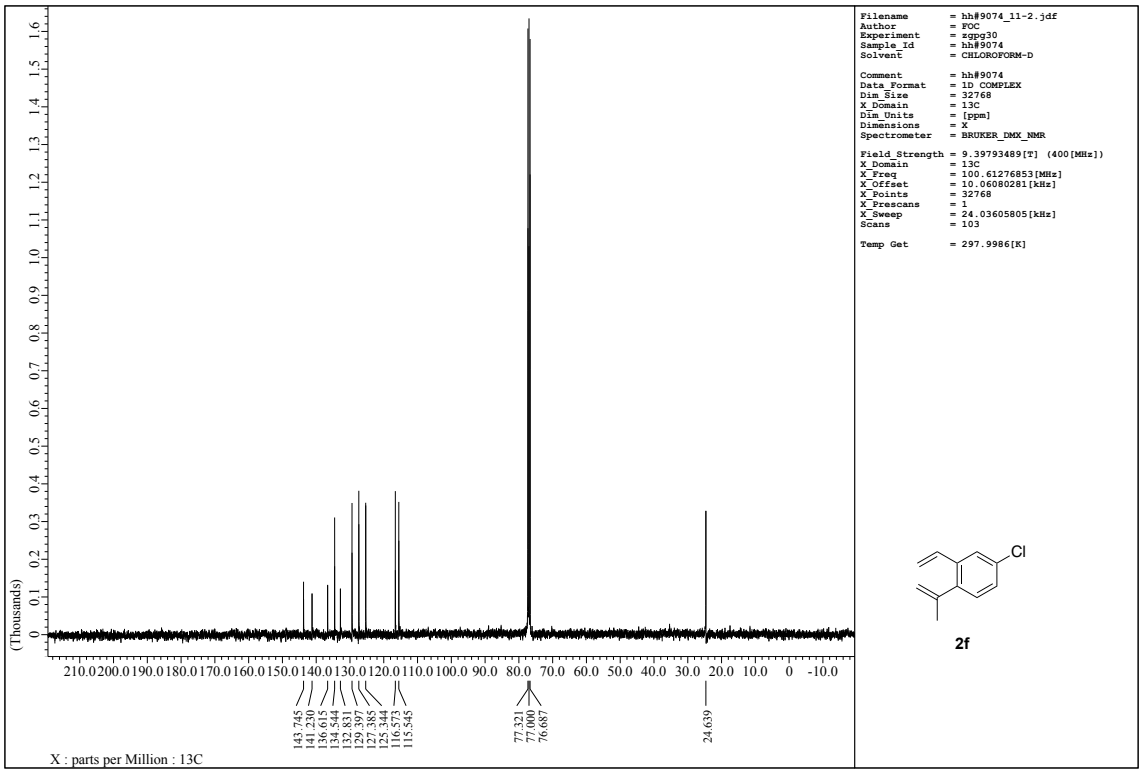
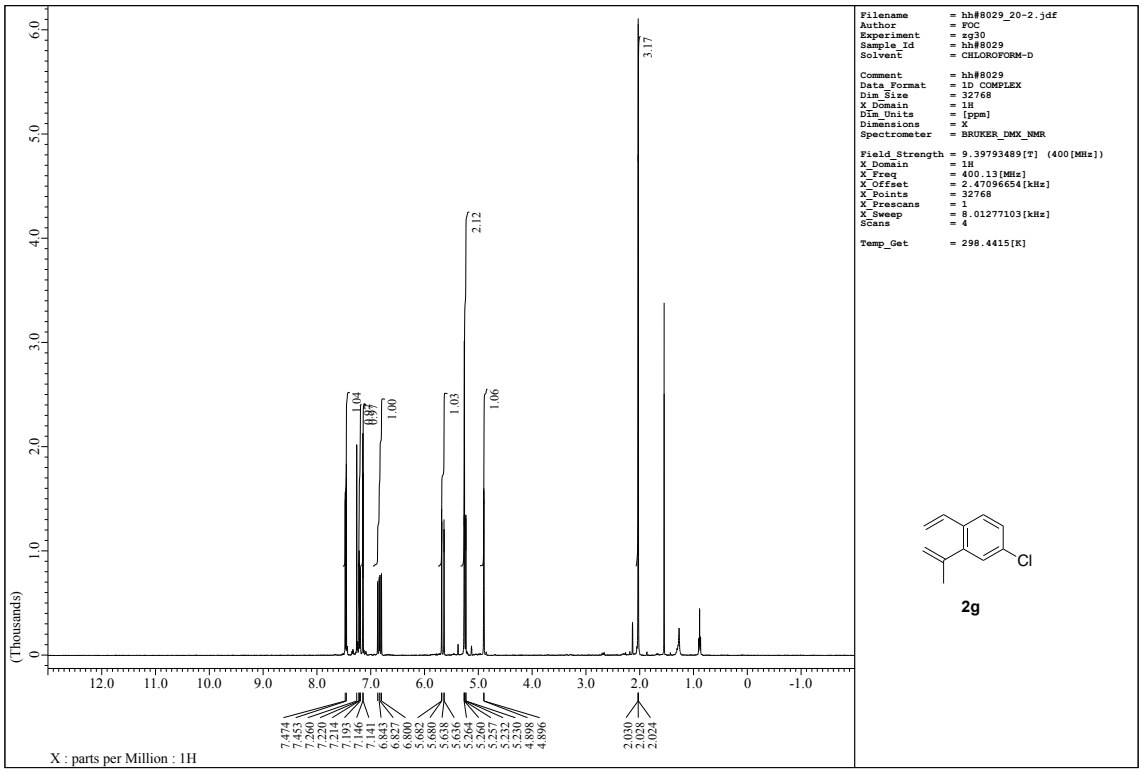
X : parts per Million : 13C
2d

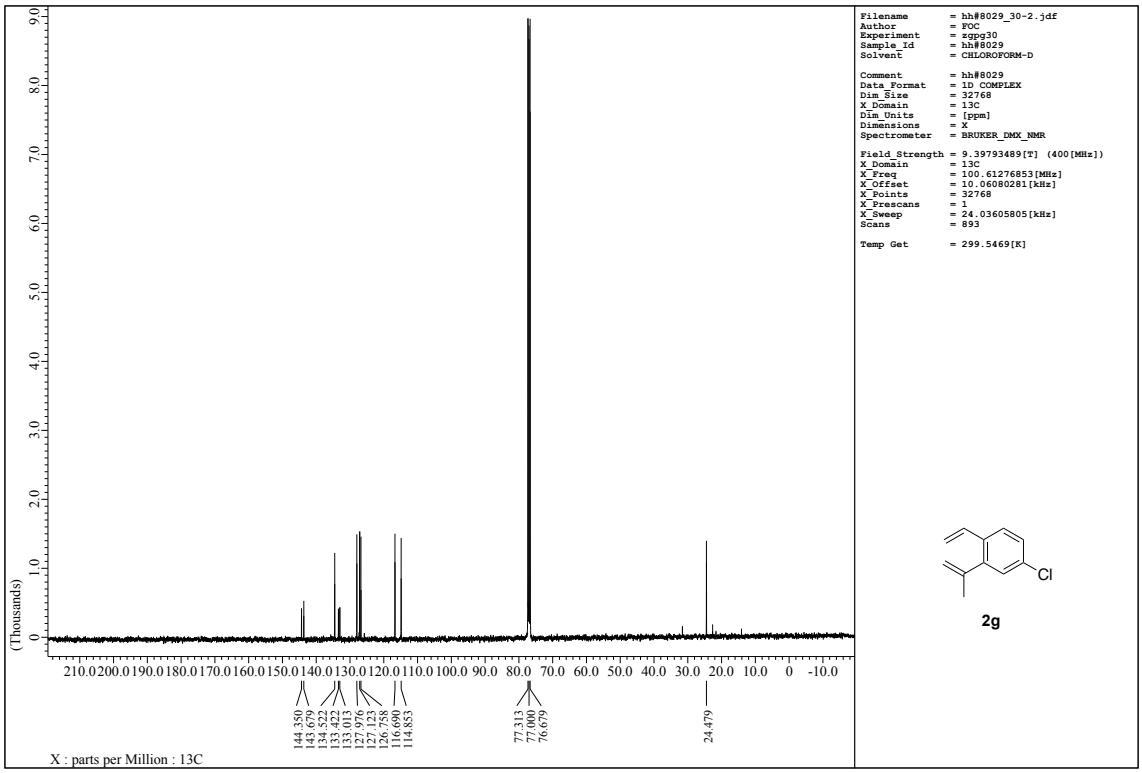

Filename = hh#9072_20-3.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9072
Solvent = CHLOROFORM-D
Comment = hh#9072
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.0002[K]
(Thousands)
X : parts per Million : 1H
F
2e

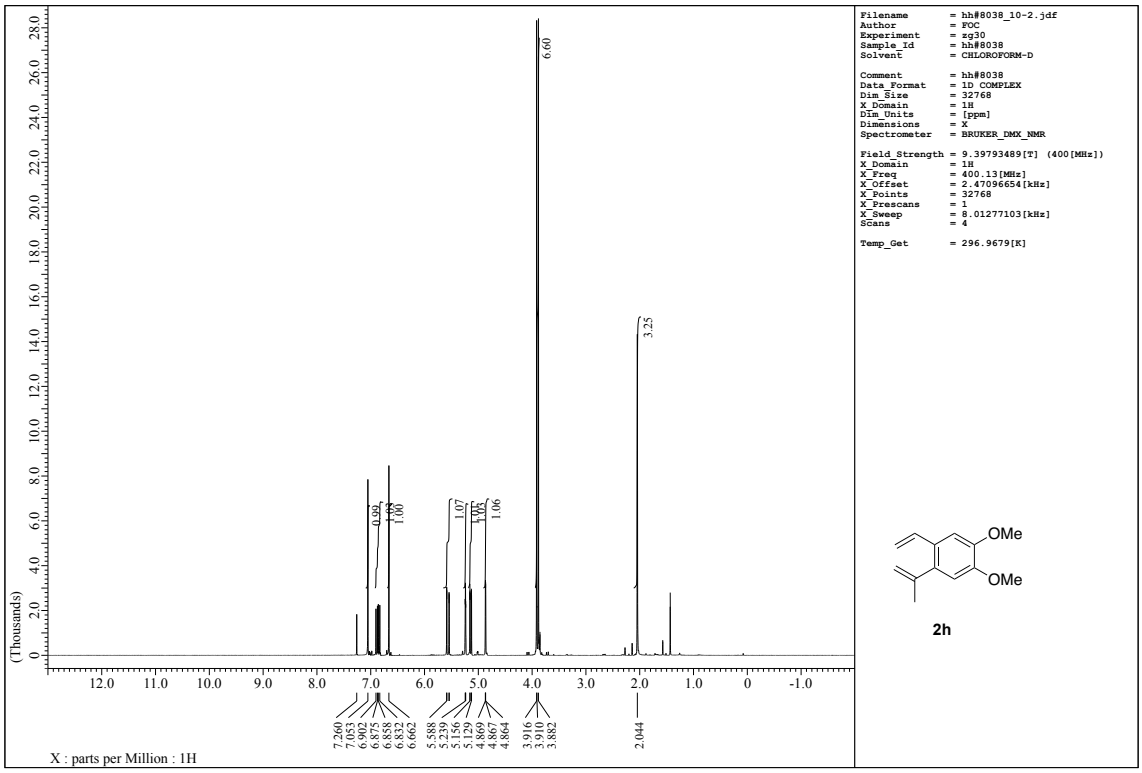

Filename = hh#9072_21-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9072
Solvent = CHLOROFORM-D
Comment = hh#9072
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 205
Temp Get = 298.0001[K]
(Thousands)
X : parts per Million : 13C
F
2e

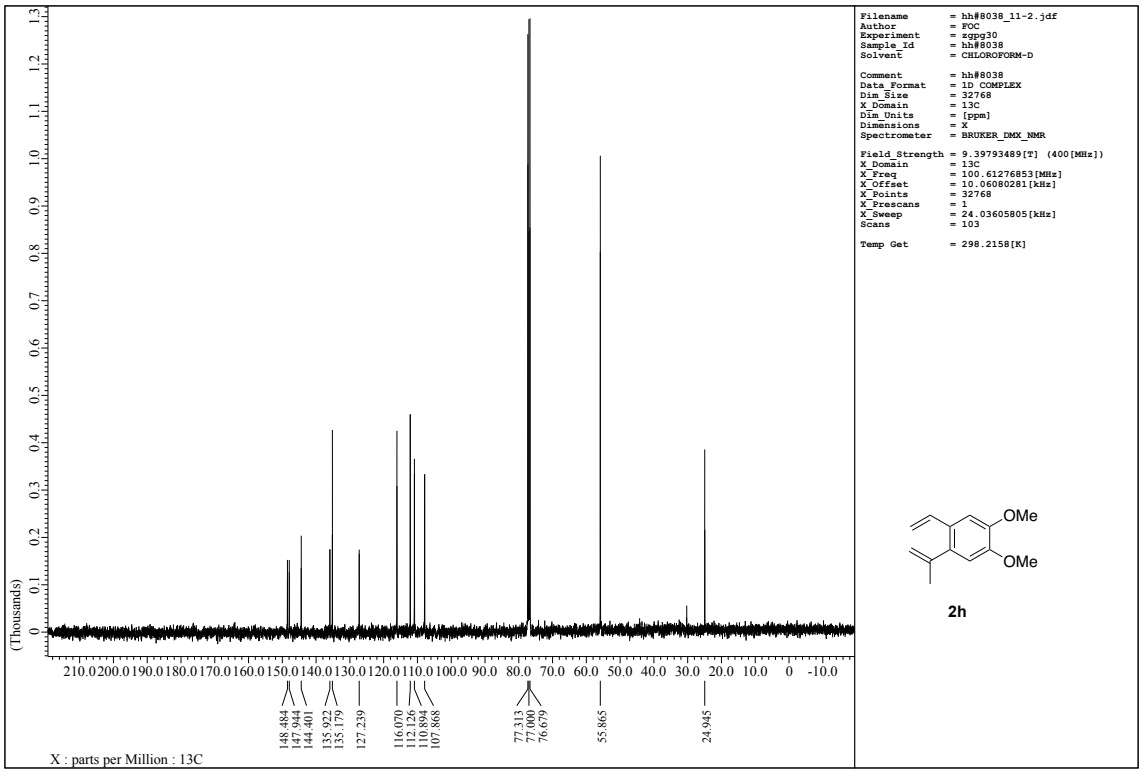

Filename = hh#9074_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9074
Solvent = CHLOROFORM-D
Comment = hh#9074
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.0016[K]
(Thousands)
3.13
2.11
1.06
1.05
1.00
1.03
1.01
7.511
7.505
7.260
7.203
7.183
7.177
7.096
7.078
6.878
6.851
6.834
6.807
5.710
5.708
5.666
5.664
5.294
5.290
5.263
5.259
5.255
5.251
4.876
4.874
2.026
2.023
X : parts per Million : 1H
Cl
2f

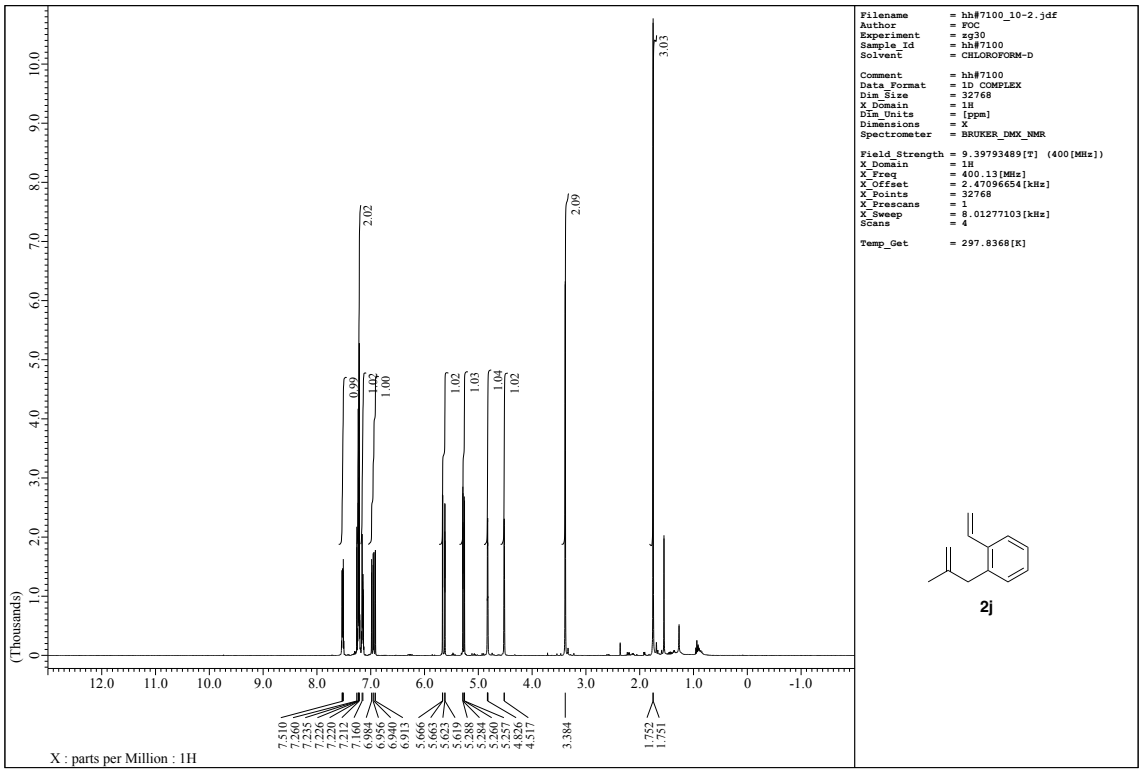

Filename = hh#9074_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9074
Solvent = CHLOROFORM-D
Comment = hh#9074
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 103
Temp Get = 297.9986[K]
(Thousands)
143.745
141.230
136.615
134.544
132.831
129.397
127.385
125.344
116.573
115.545
77.321
77.000
76.687
24.639
X : parts per Million : 13C
Cl
2f

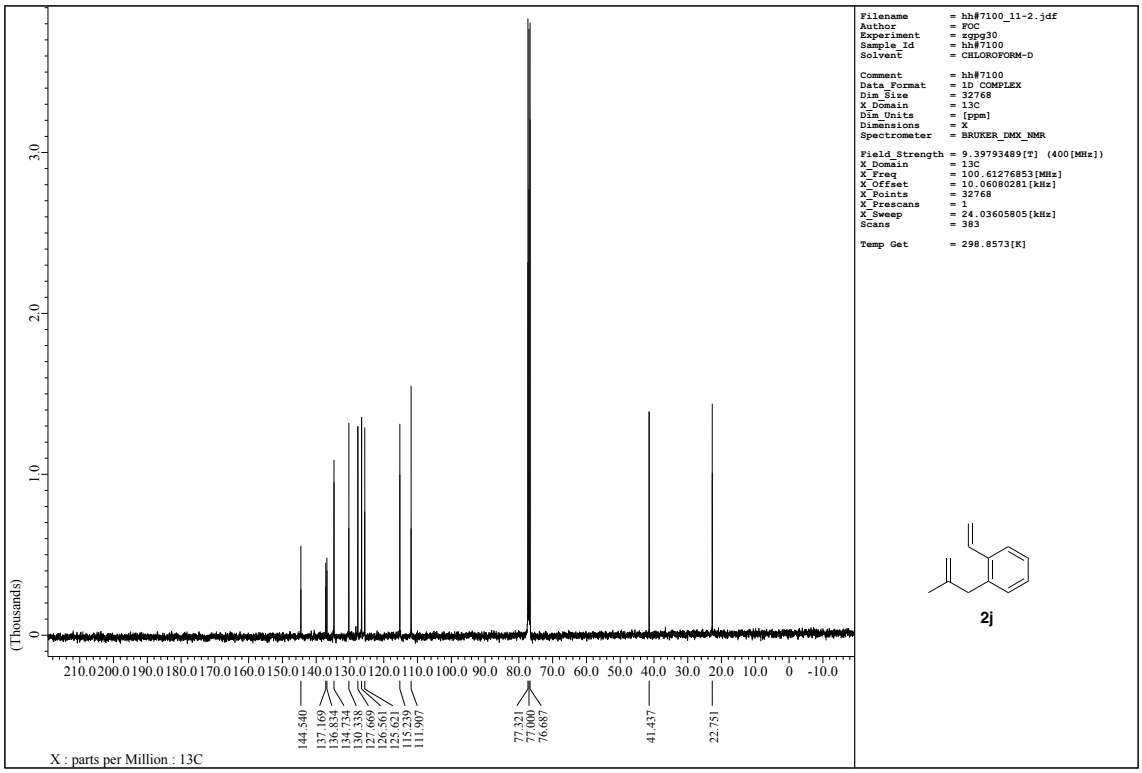

Filename = hh#8029_20-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8029
Solvent = CHLOROFORM-D
Comment = hh#8029
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.4415[K]
(Thousands)
3.17
2.12
1.04
0.97
1.00
1.03
1.06
7.474
7.453
7.260
7.220
7.214
7.193
7.146
7.141
6.843
6.827
6.800
5.682
5.680
5.638
5.636
5.264
5.260
5.257
5.232
5.230
4.898
4.896
2.030
2.028
2.024
X : parts per Million : 1H
Cl
2g

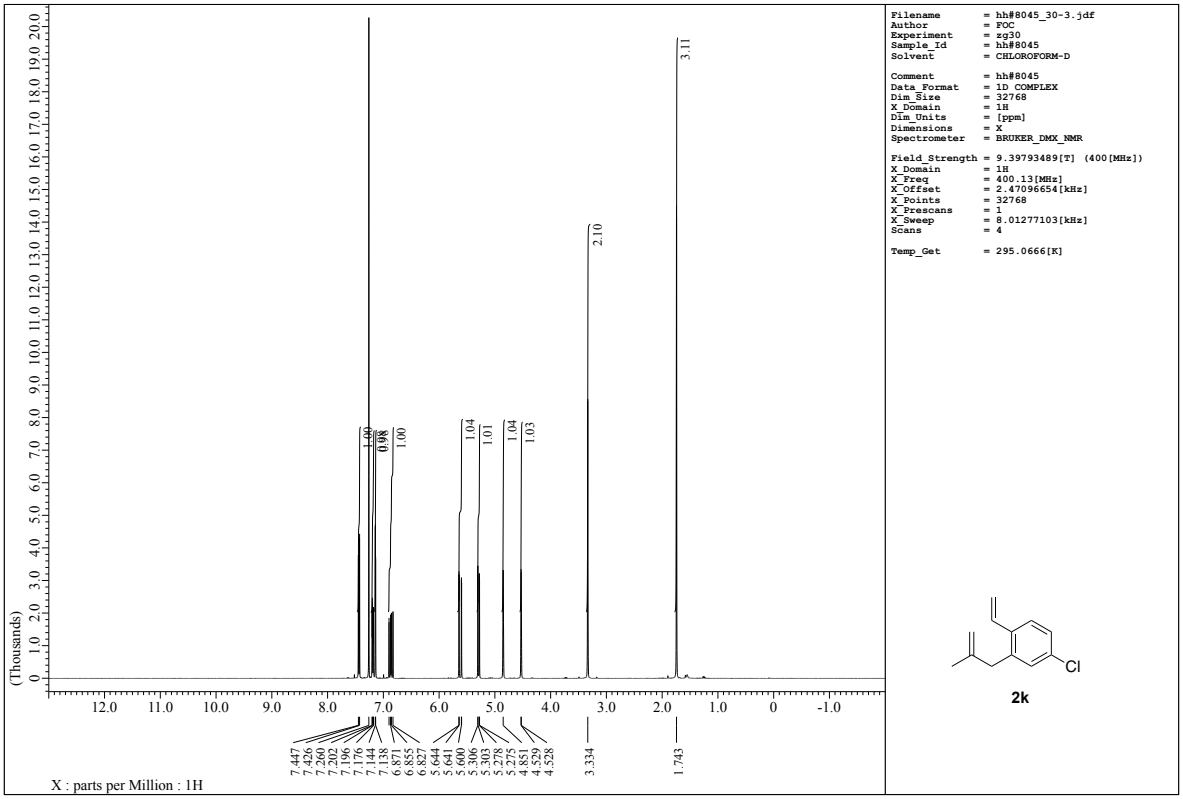

Filename = hh#8029_30-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8029
Solvent = CHLOROFORM-D
Comment = hh#8029
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 893
Temp_Get = 299.5469[K]
(Thousands)
144.350
143.679
134.522
133.422
133.013
127.976
127.153
126.758
116.690
114.853
77.313
77.000
76.679
24.479
X : parts per Million : 13C
Cl
2g

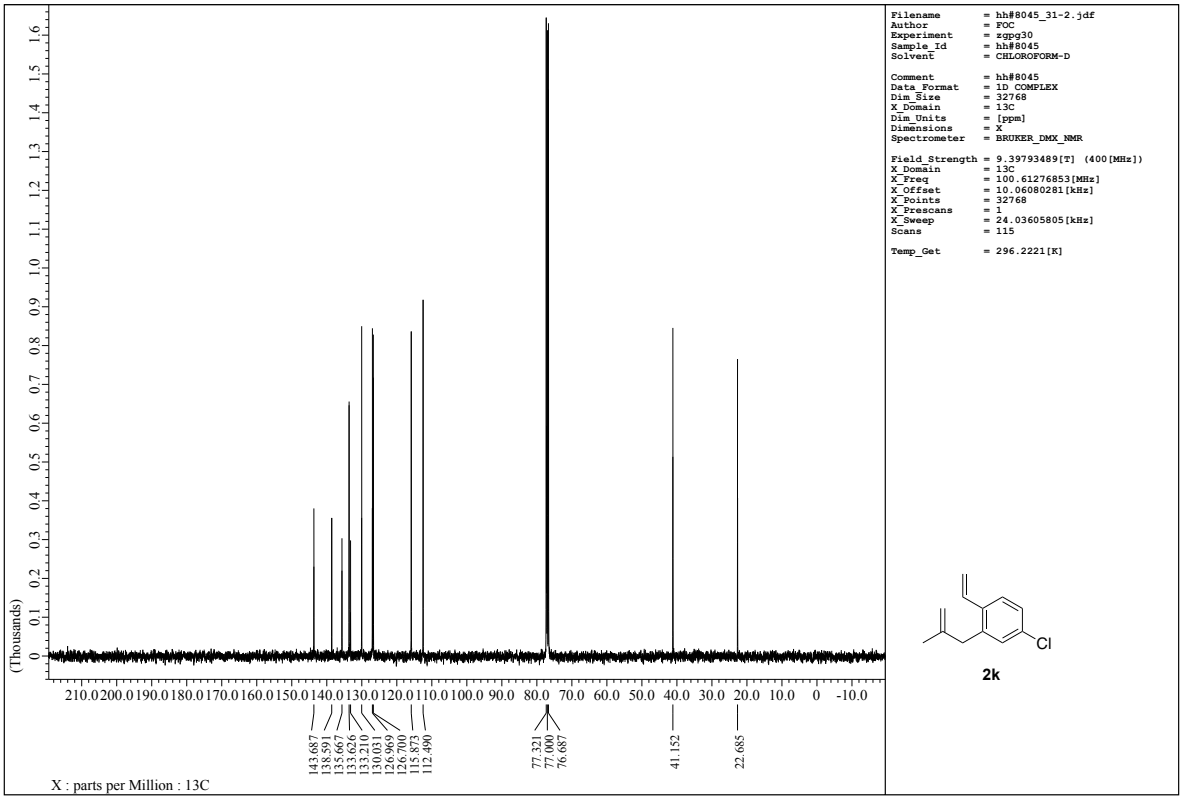

Filename = hh#8038_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8038
Solvent = CHLOROFORM-D
Comment = hh#8038
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 296.9679[K]
6.60
3.25
0.99
1.03
1.00
1.07
1.05
1.06
(Thousands)
7.260
7.053
6.902
6.875
6.858
6.832
6.662
5.588
5.239
5.156
5.139
4.869
4.867
4.864
3.916
3.910
3.882
2.044
X : parts per Million : 1H
OMe
OMe
2h

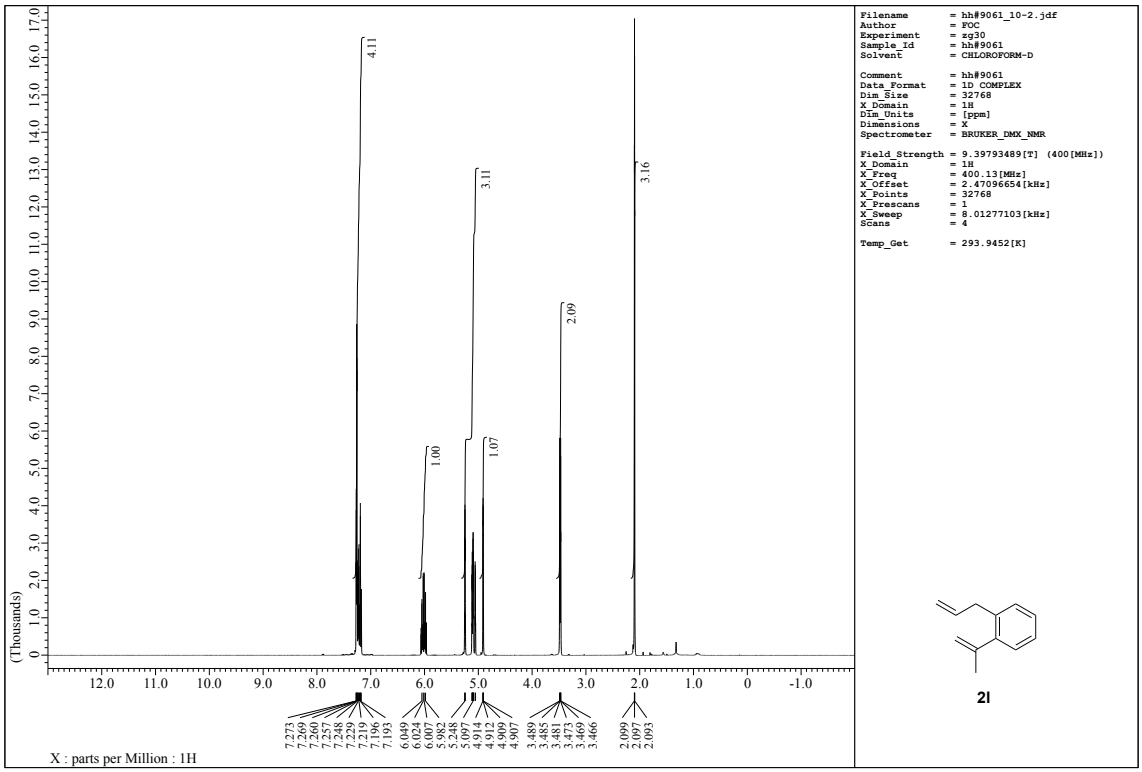

Filename = hh#8038_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8038
Solvent = CHLOROFORM-D
Comment = hh#8038
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 103
Temp Get = 298.2158[K]
(Thousands)
148.484
147.944
144.401
135.922
135.179
127.239
116.070
112.126
110.894
107.868
77.313
77.000
76.679
55.865
24.945
X : parts per Million : 13C
OMe
OMe
2h

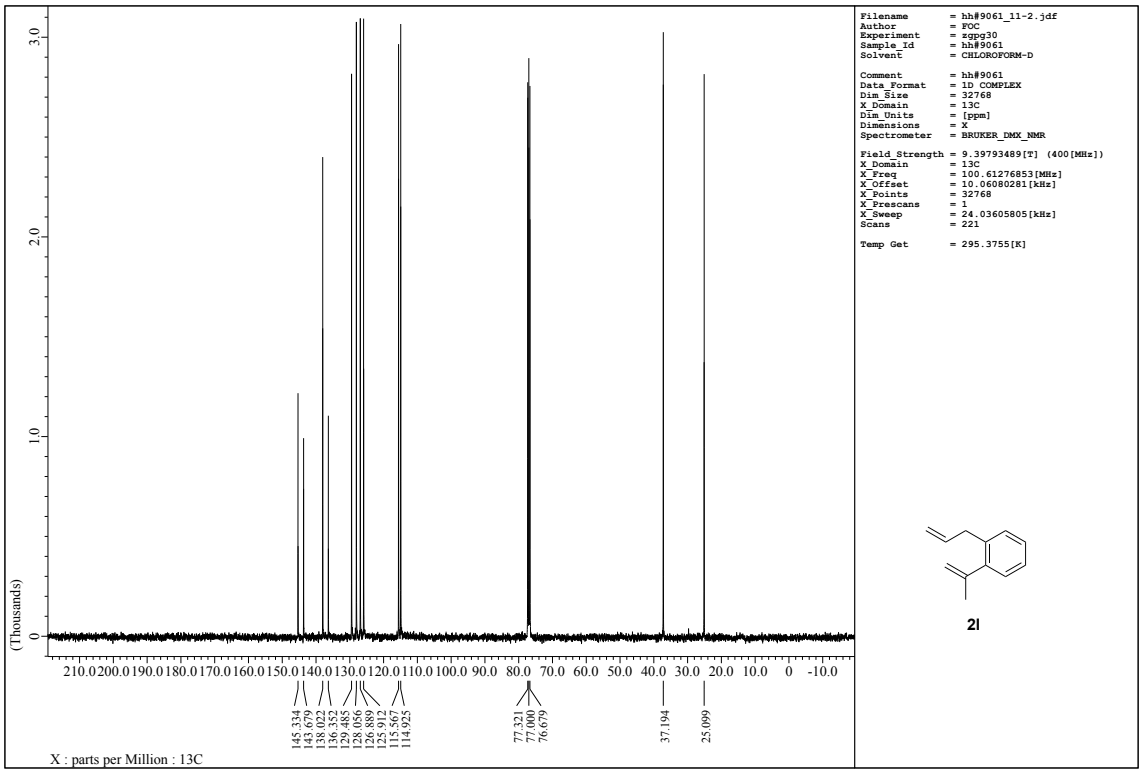

Filename = hh#7100_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#7100
Solvent = CHLOROFORM-D
Comment = hh#7100
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.8368[K]
2j
X : parts per Million : 1H

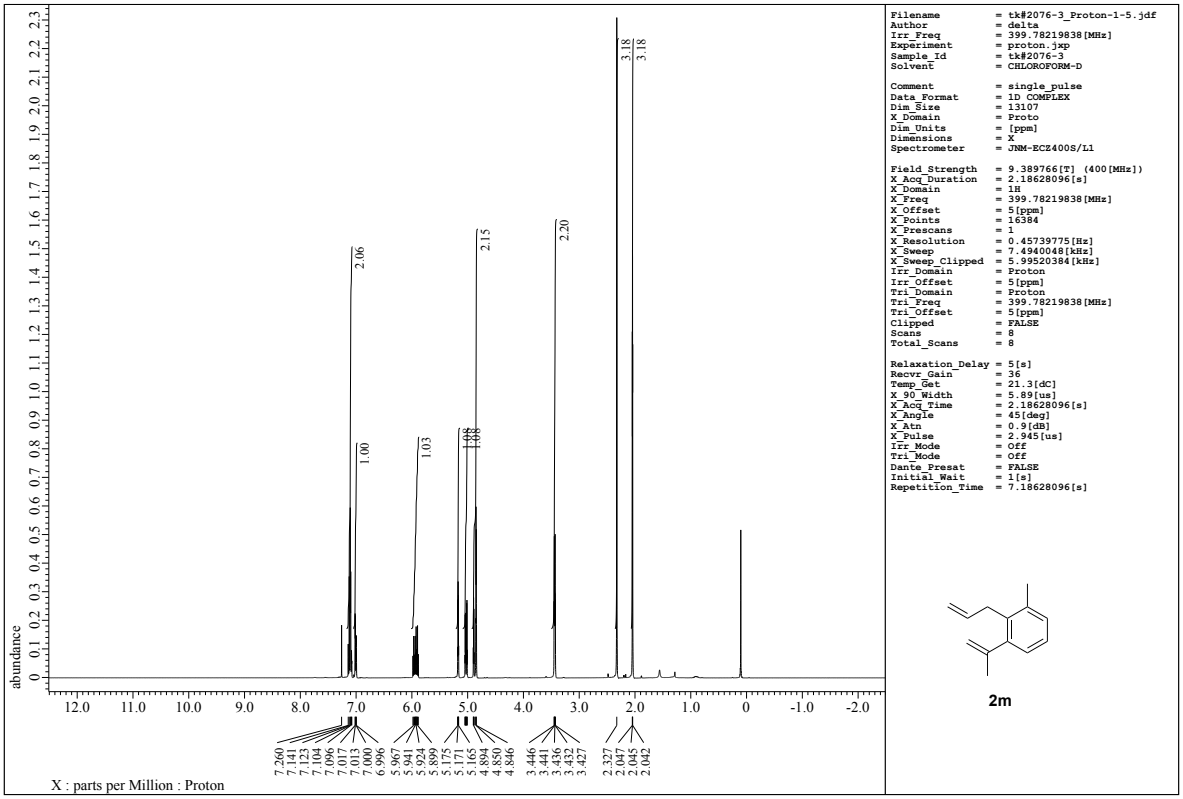

Filename = hh#7100_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#7100
Solvent = CHLOROFORM-D
Comment = hh#7100
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 383
Temp Get = 298.8573[K]
2j
X : parts per Million : 13C


Filename = hh#8045_30-3.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8045
Solvent = CHLOROFORM-D
Comment = hh#8045
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 295.0666[K]
Cl
2k
X : parts per Million : 1H

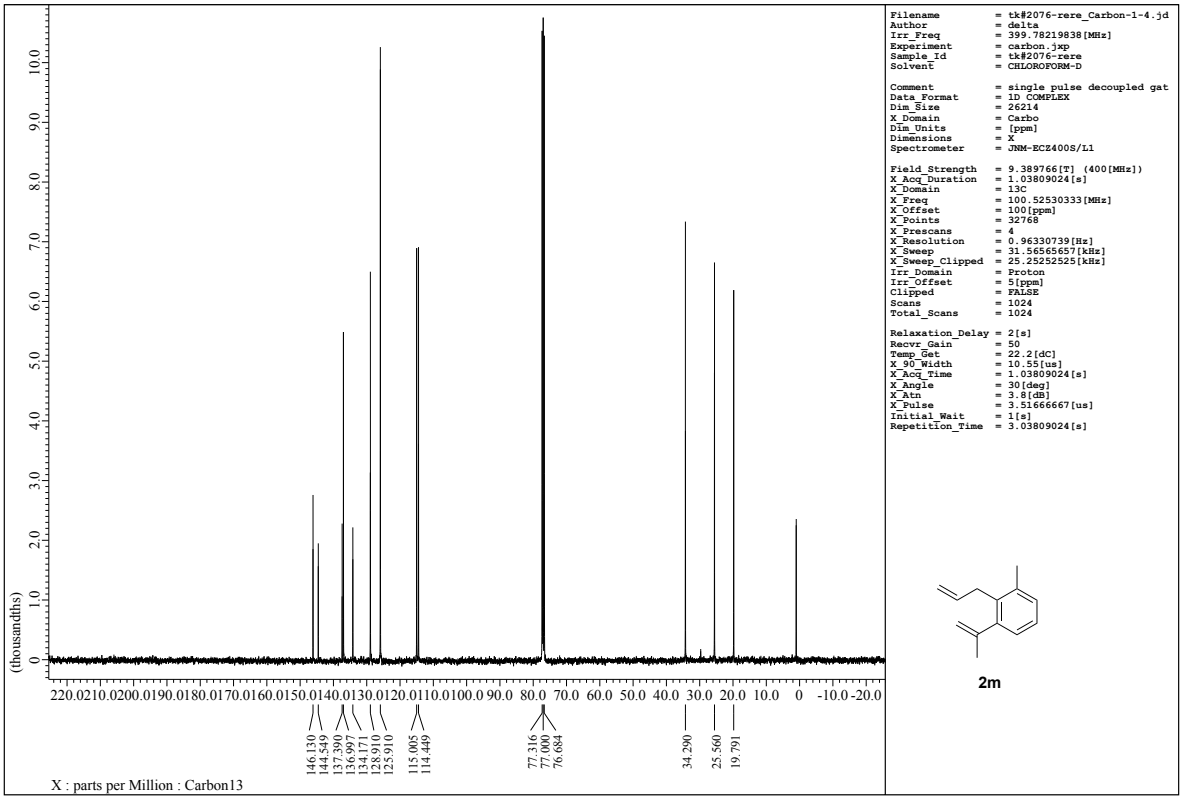

Filename = hh#8045_31-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8045
Solvent = CHLOROFORM-D
Comment = hh#8045
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 115
Temp_Get = 296.2221[K]
Cl
2k
X : parts per Million : 13C


Filename = hh#9061_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9061
Solvent = CHLOROFORM-D
Comment = hh#9061
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 = 293.9452[K]
4.11
3.11
3.16
2.09
1.00
1.07
(Thousands)
X : parts per Million : 1H
2l

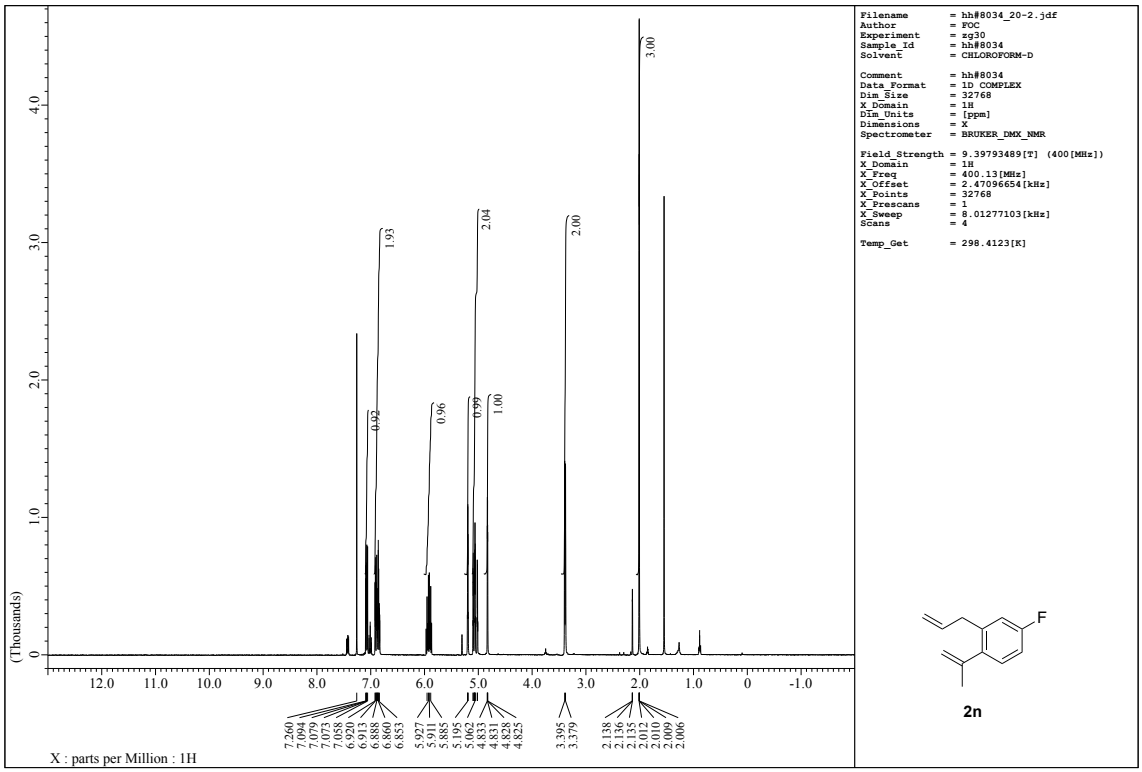

Filename = hh#9061_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9061
Solvent = CHLOROFORM-D
Comment = hh#9061
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 221
Temp Get = 295.3755[K]
(Thousands)
X : parts per Million : 13C
2l

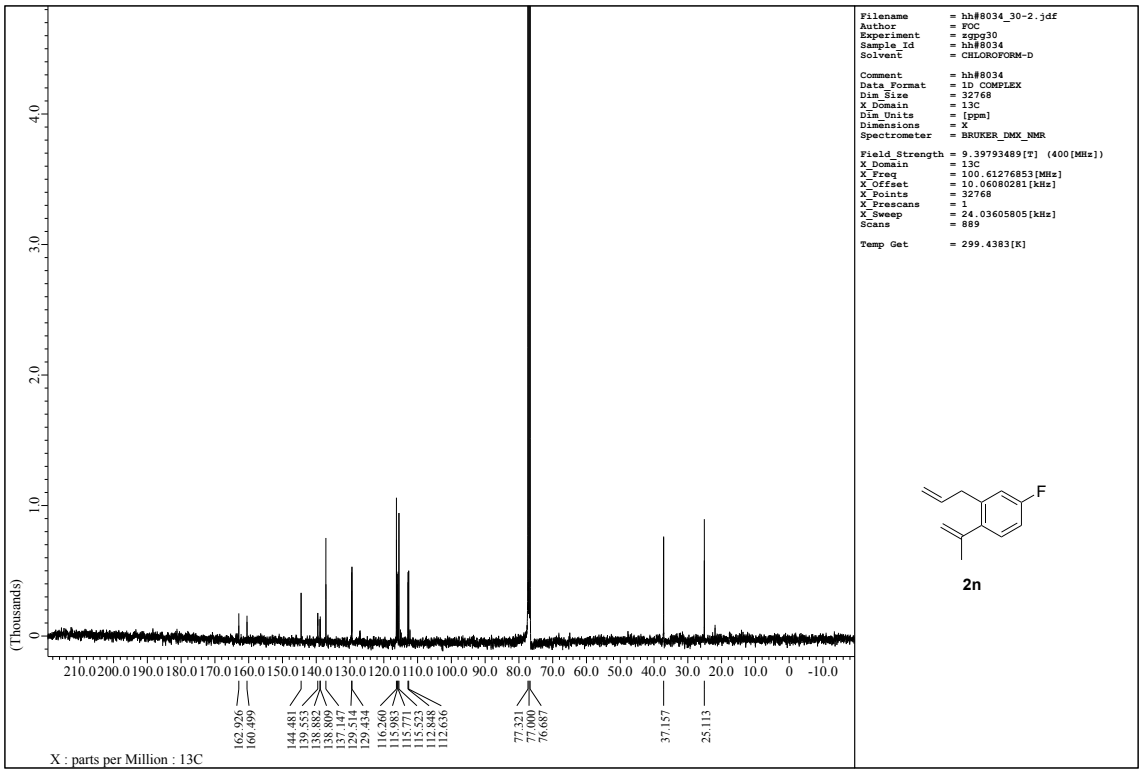

abundance
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
2.06
1.00
1.03
1.08
1.08
2.15
2.20
3.18
3.18
7.260
7.141
7.123
7.104
7.096
7.017
7.013
7.000
6.996
5.967
5.941
5.924
5.899
5.175
5.171
5.165
5.161
4.894
4.890
4.846
3.446
3.441
3.436
3.432
3.427
2.327
2.047
2.045
2.042
X : parts per Million : Proton
Filename = tk#2076-3_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#2076-3
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 = 36
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]

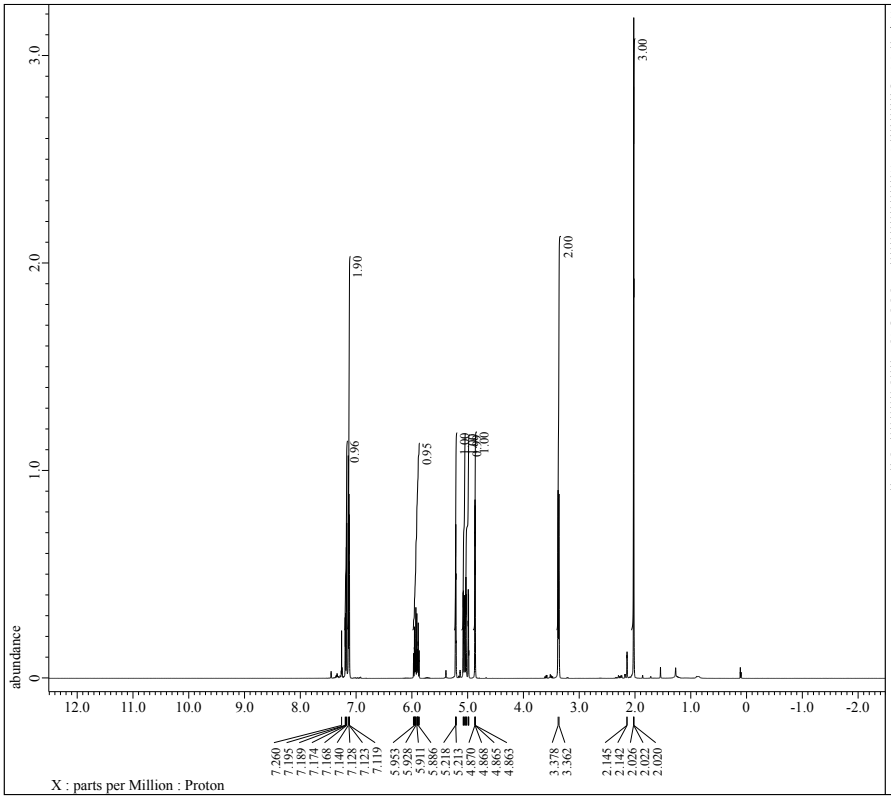
2m


(thousandths)
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
146.130
144.549
137.390
136.997
134.171
128.910
125.910
115.005
114.449
77.316
77.000
76.684
34.290
25.560
19.791
X : parts per Million : Carbon13
Filename = tk#2076-rere_Carbon-1-4.jd
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#2076-rere
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 = 22.2[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]

2m


Filename = hh#8034_20-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8034
Solvent = CHLOROFORM-D
Comment = hh#8034
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 = 298.4123[K]
(Thousands)
X : parts per Million : 1H
F
2n


Filename = hh#8034_30-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8034
Solvent = CHLOROFORM-D
Comment = hh#8034
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 = 889
Temp Get = 299.4383[K]
(Thousands)
X : parts per Million : 13C
F
2n



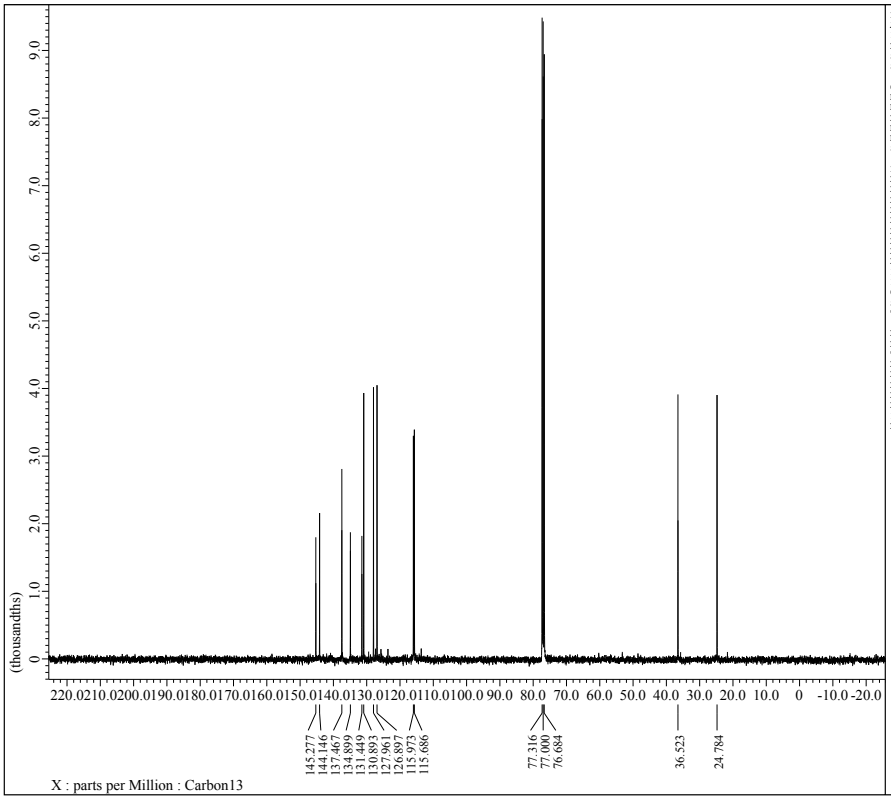
Filename = tk#2038-1_Proton-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#2038-1
Solvent = CHLOROFORM-D

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 = 18.7[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]

2o



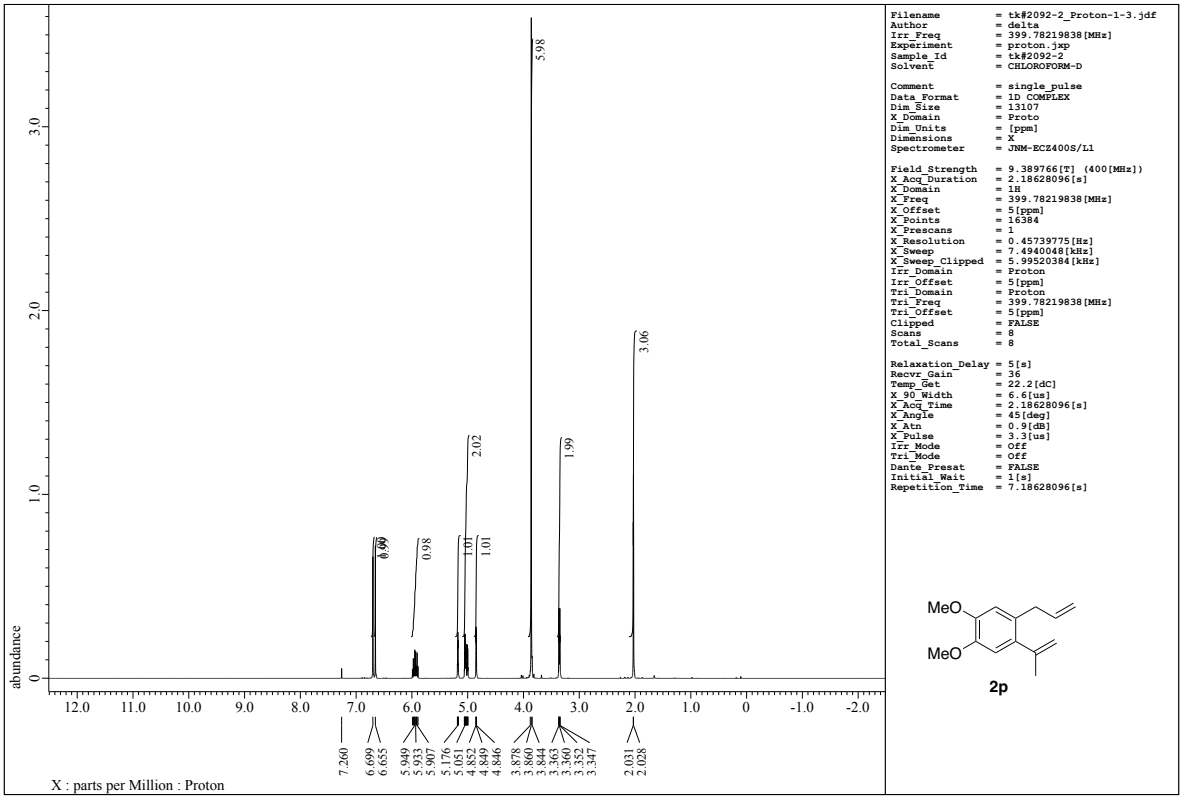
Filename = tk#2038-1_Carbon-1-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#2038-1
Solvent = CHLOROFORM-D

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

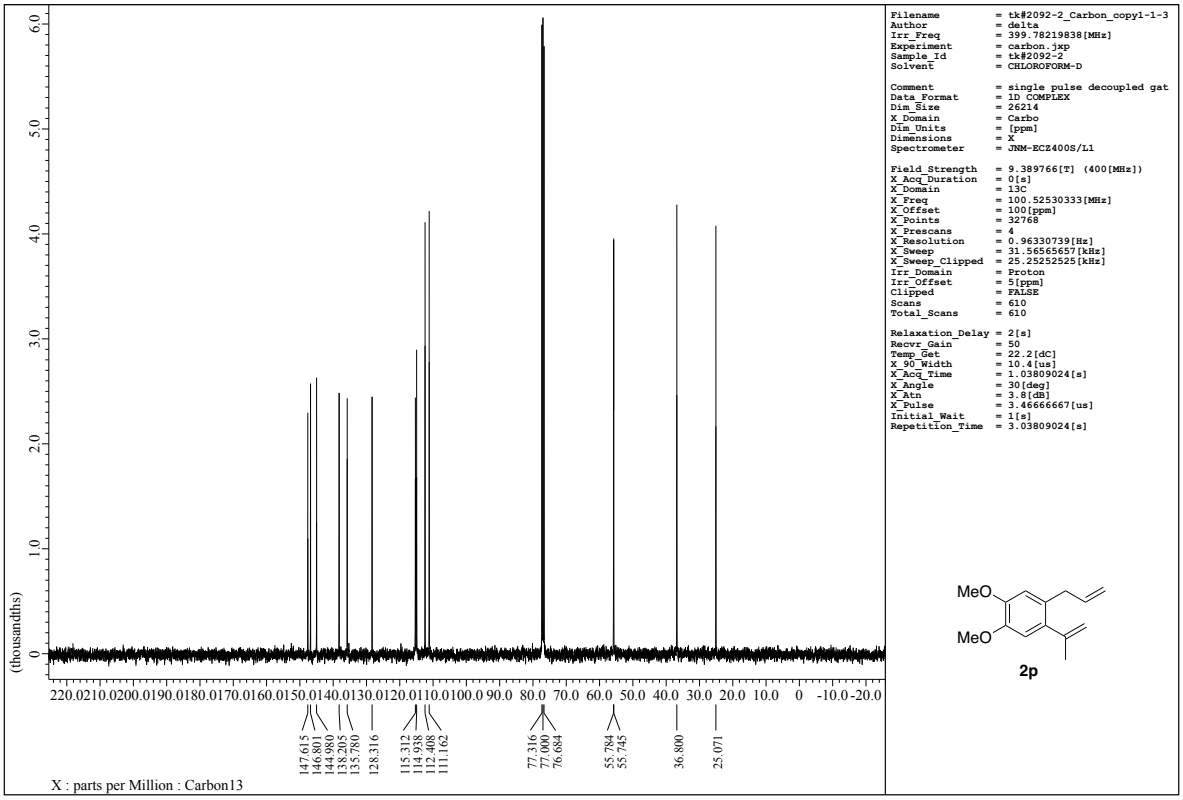
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped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

Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 18.7[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]

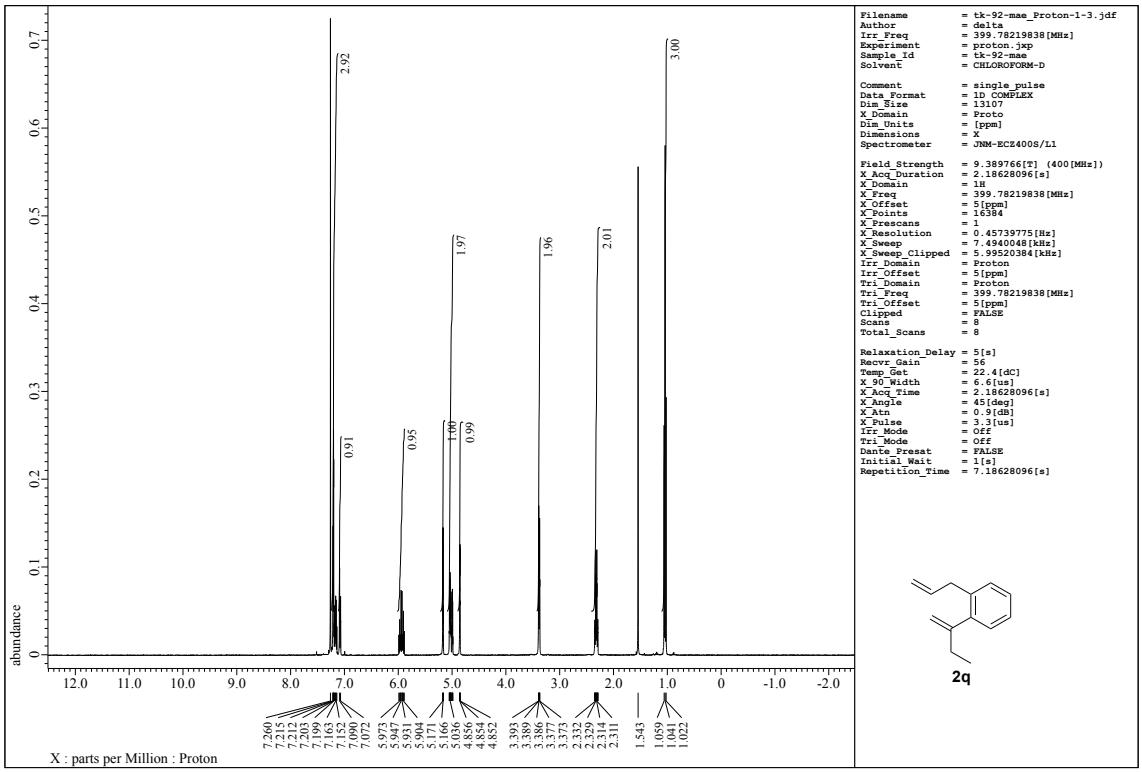
2o



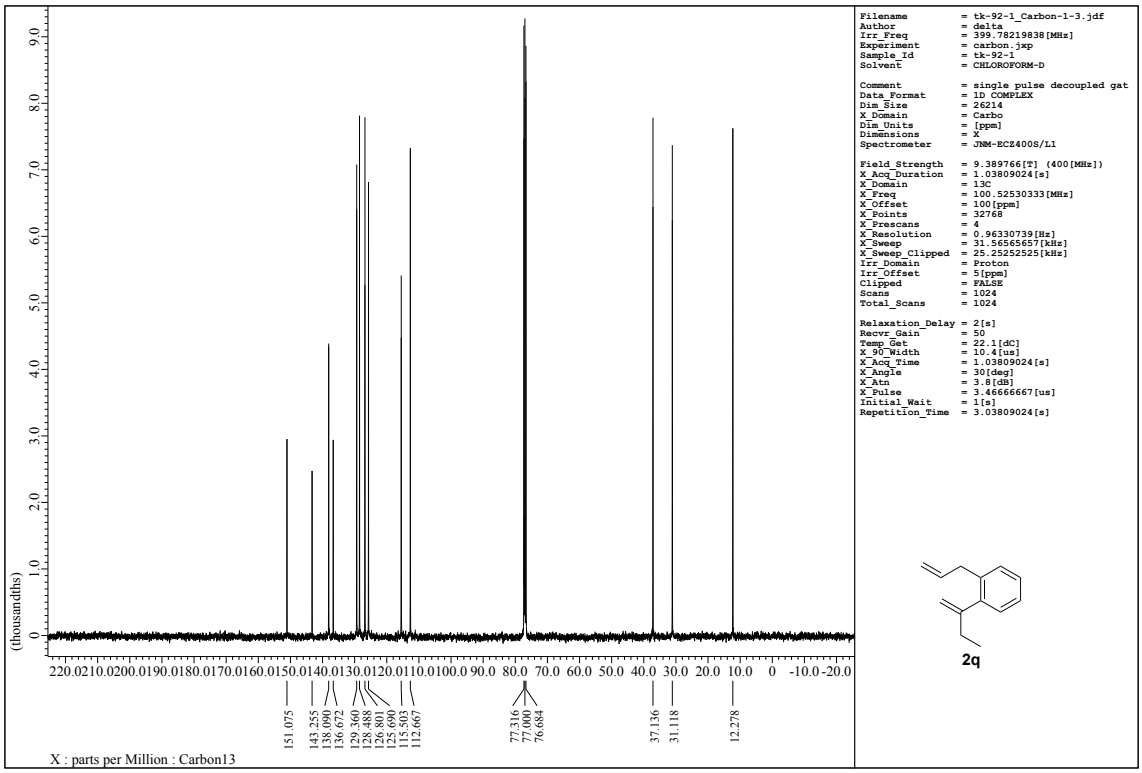

```
Filename          = tk#2092-2_Proton-1-3.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tk#2092-2
Solvent           = CHLOROFORM-D

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

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 22.2[dC]
X_90_Width        = 6.6[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.3[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



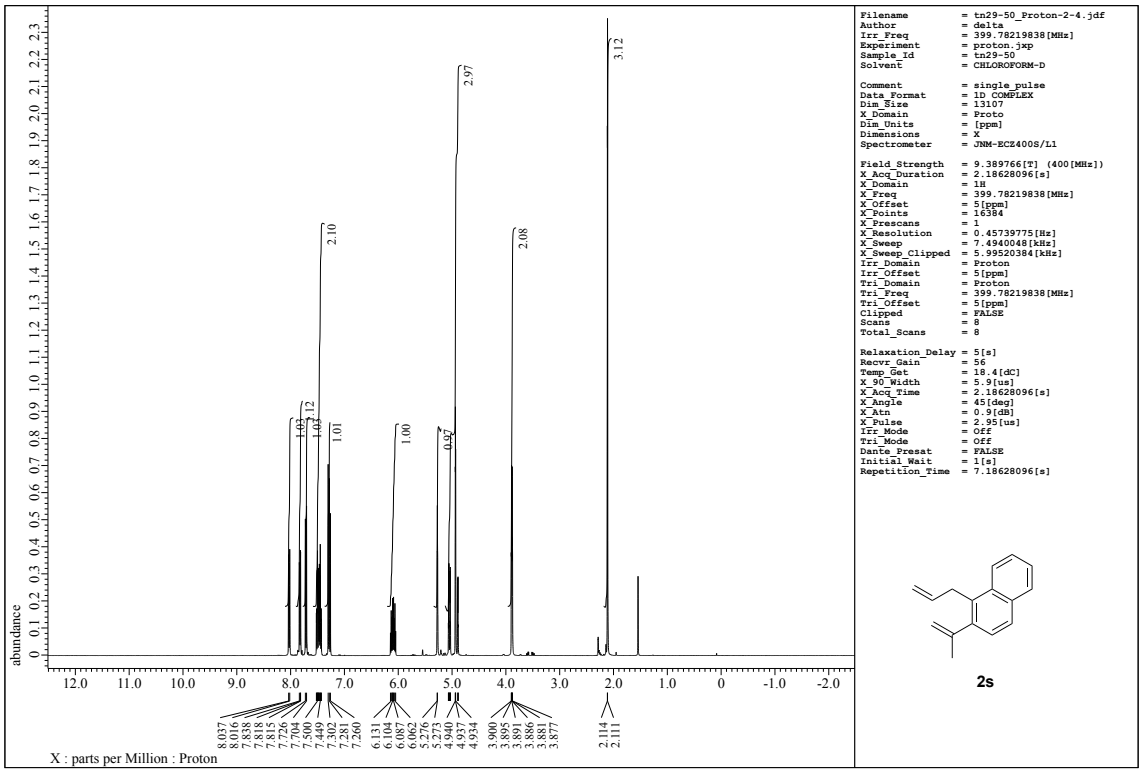

```
Filename          = tk#2092-2_Carbon_copy1-1-3
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tk#2092-2
Solvent           = CHLOROFORM-D

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    = 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             = 610
Total_Scans       = 610

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.2[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]
```


abundance
2.92
0.91
0.95
1.00
1.97
0.99
1.96
2.01
3.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.260 7.215 7.212 7.203 7.199 7.165 7.152 7.090 7.072
5.973 5.947 5.931 5.904 5.171 5.166 5.036 4.856 4.854 4.852
3.393 3.389 3.386 3.377 3.373
2.332 2.329 2.314 2.311
1.543
1.059 1.041 1.022
X : parts per Million : Proton
Filename = tk-92-mae_Proton-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk-92-mae
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 = 22.4[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]

2q

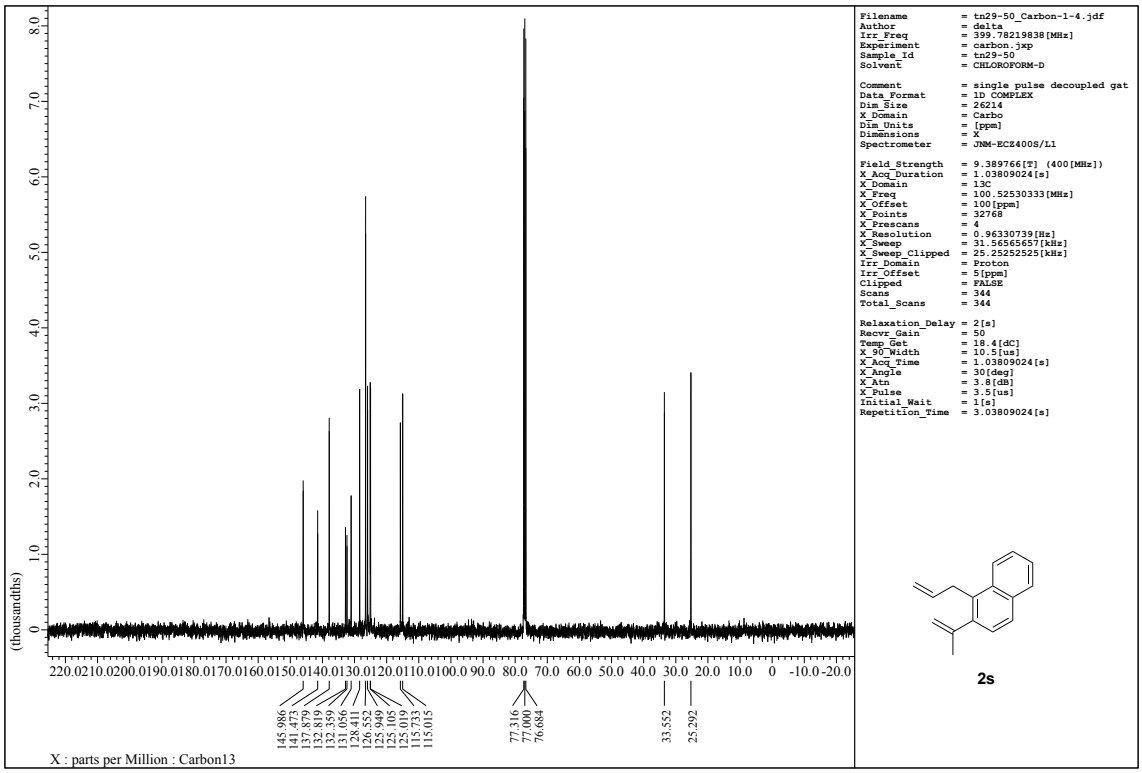

(thousandths)
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
151.075
143.255
138.090
136.672
129.360
128.488
126.801
125.690
115.503
112.667
77.316
77.000
76.684
37.136
31.118
12.278
X : parts per Million : Carbon13
Filename = tk-92-1_Carbon-1-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk-92-1
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 = 22.1[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]

2q



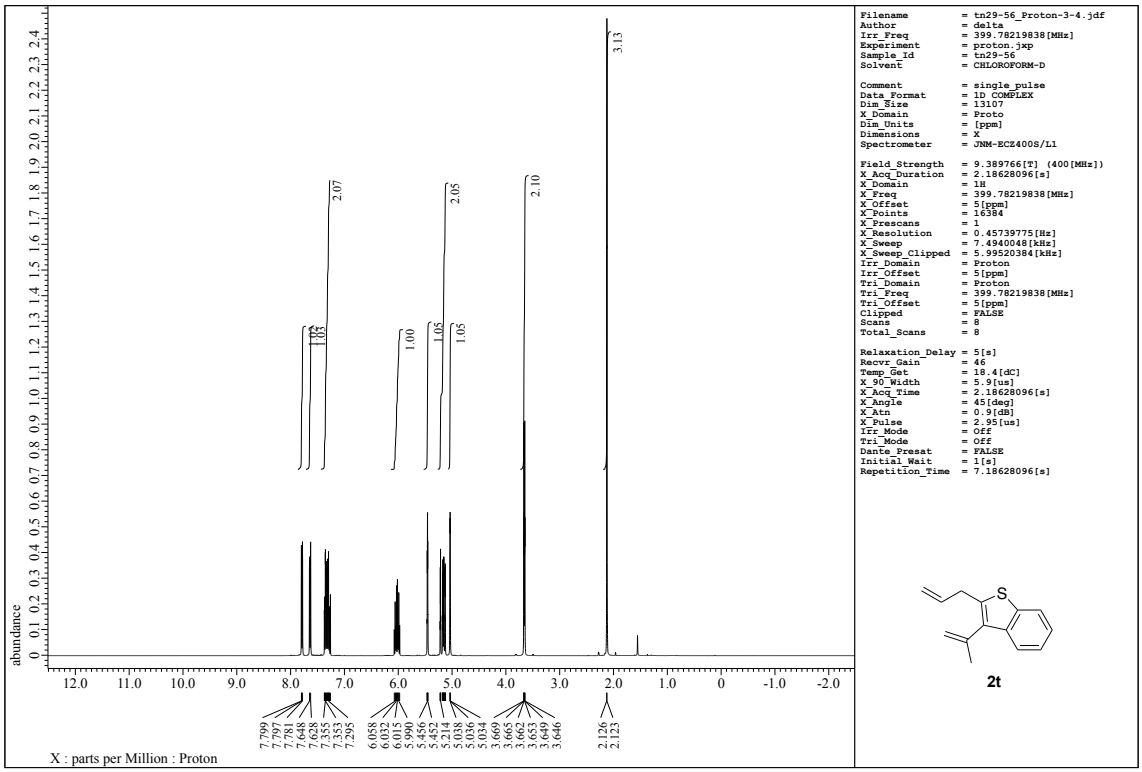

```
Filename          = tn29-50_Proton-2-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Sample_Id         = tn29-50
Solvent           = CHLOROFORM-D

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          = 18.4[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]
```

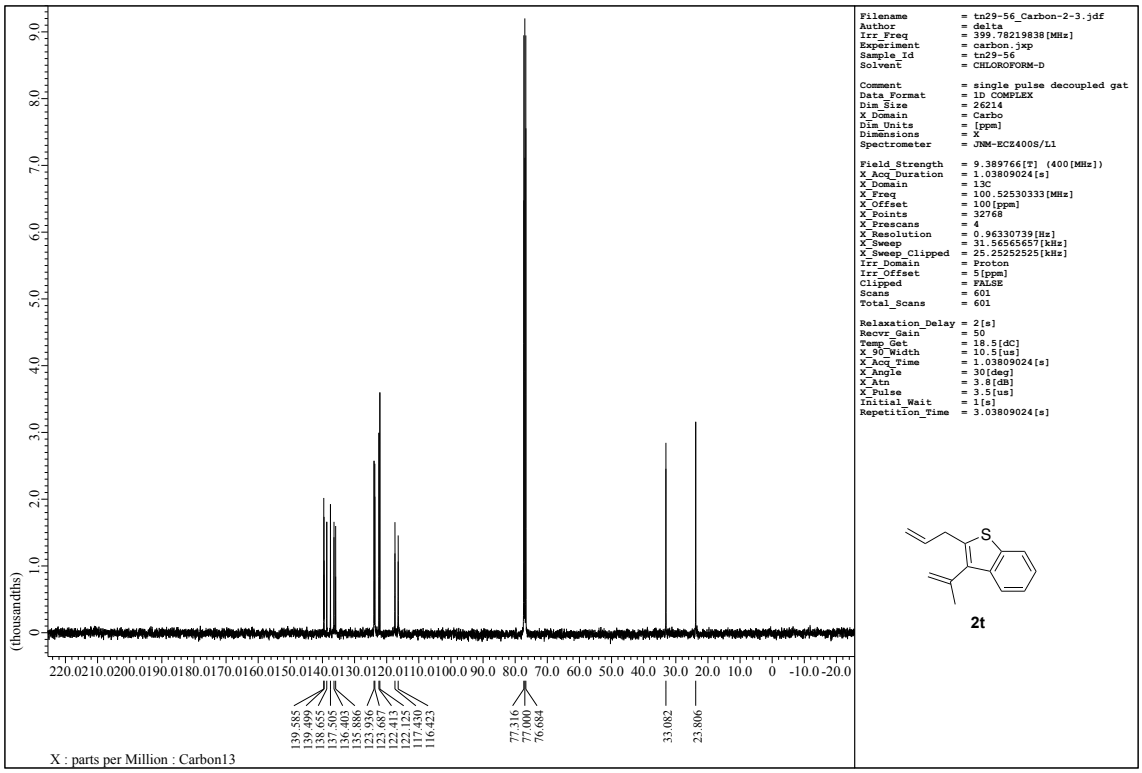

2s

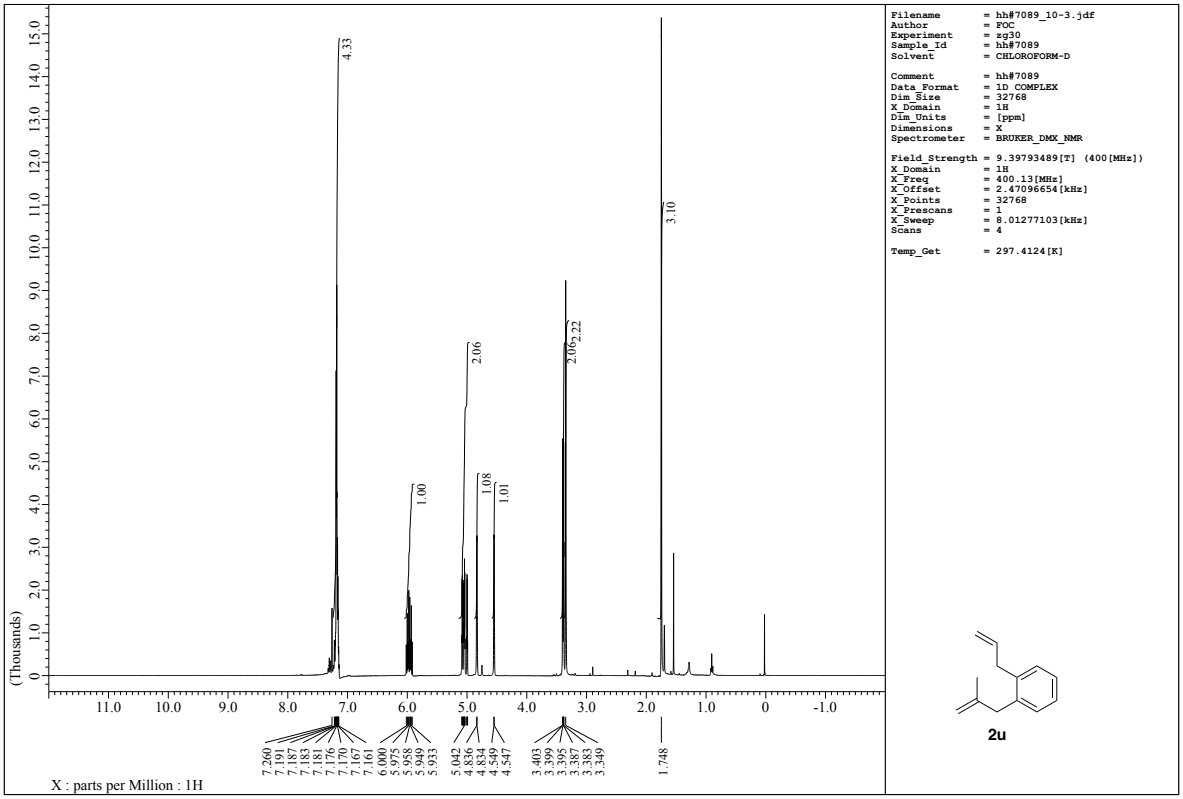


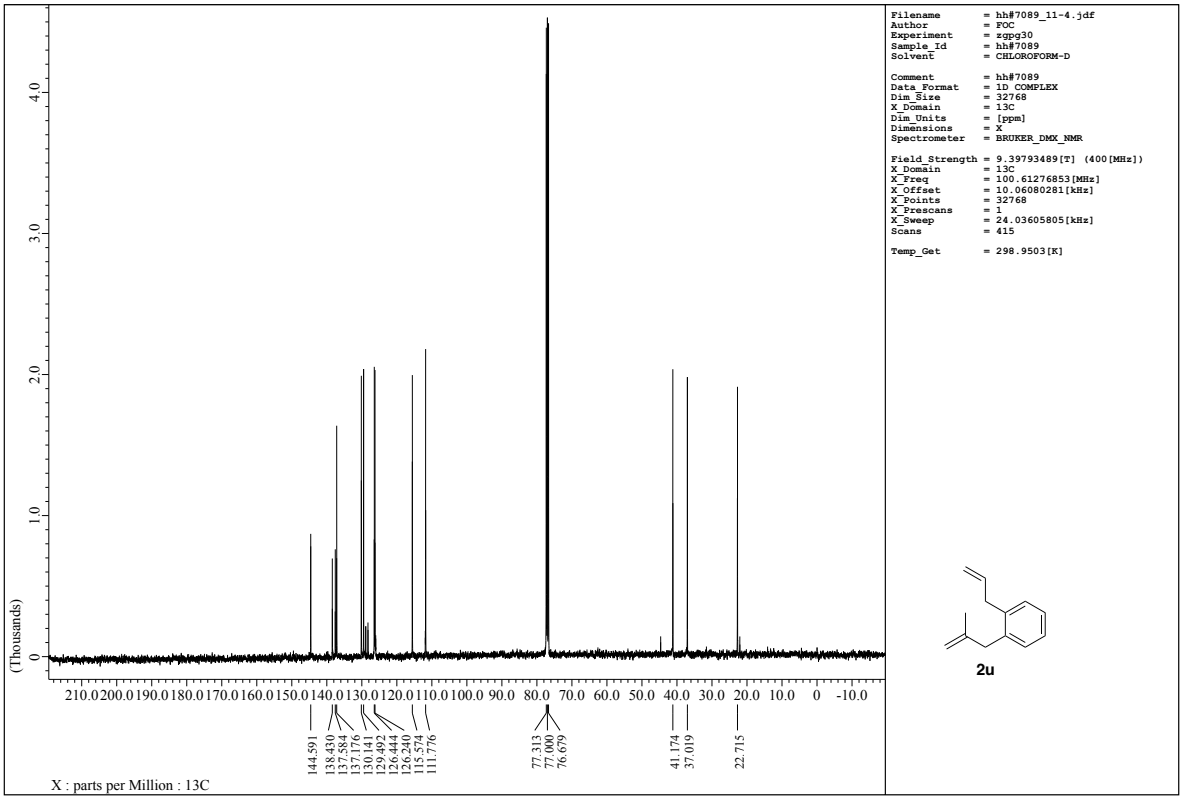

```
Filename          = tn29-50_Carbon-1-4.jdf
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Sample_Id         = tn29-50
Solvent           = CHLOROFORM-D

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           = FALSE
Scans             = 344
Total_Scans       = 344

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.4[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```

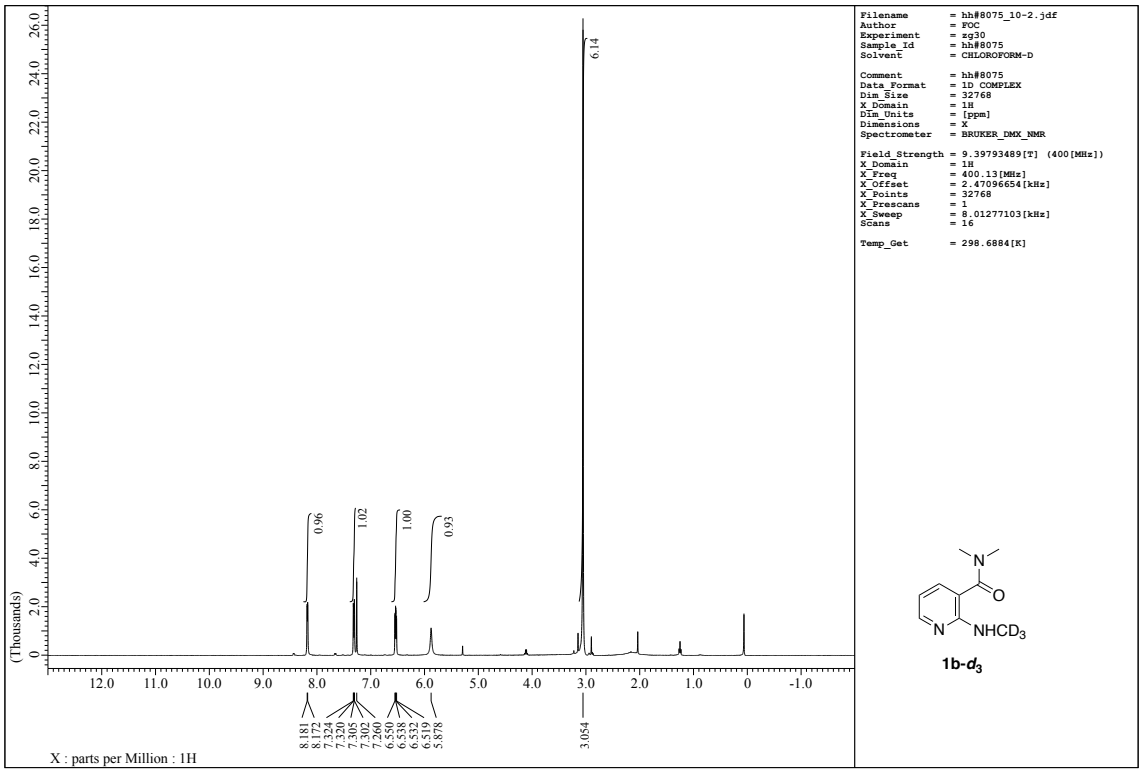

2s

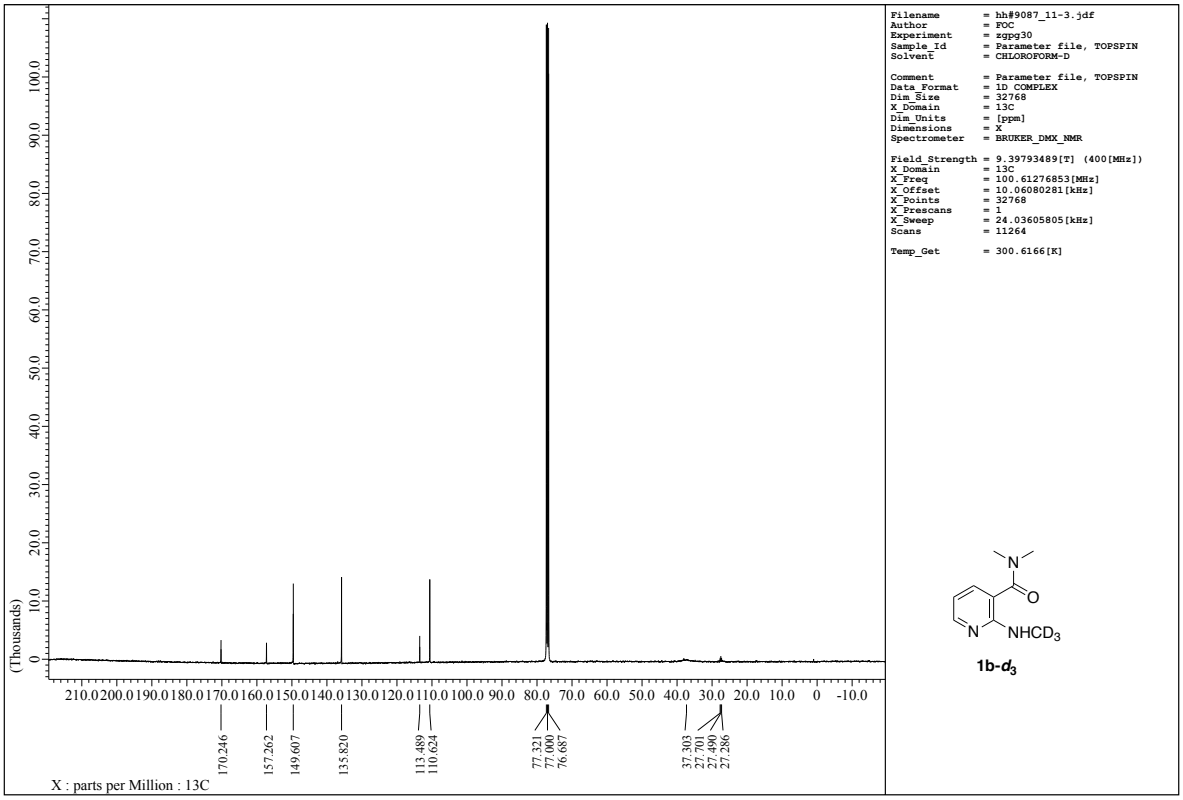

Filename = tn29-56_Proton-3-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn29-56
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 18.4[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
3.13
2.07
2.05
2.10
1.03
1.03
1.00
1.05
1.05
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.799 7.797 7.781 7.658 7.648 7.355 7.353 7.295
6.058 6.037 6.015 5.990 5.456 5.452 5.214 5.038 5.036 5.034 3.669 3.665 3.662 3.653 3.649 3.646
2.126 2.123
X : parts per Million : Proton
S
2t

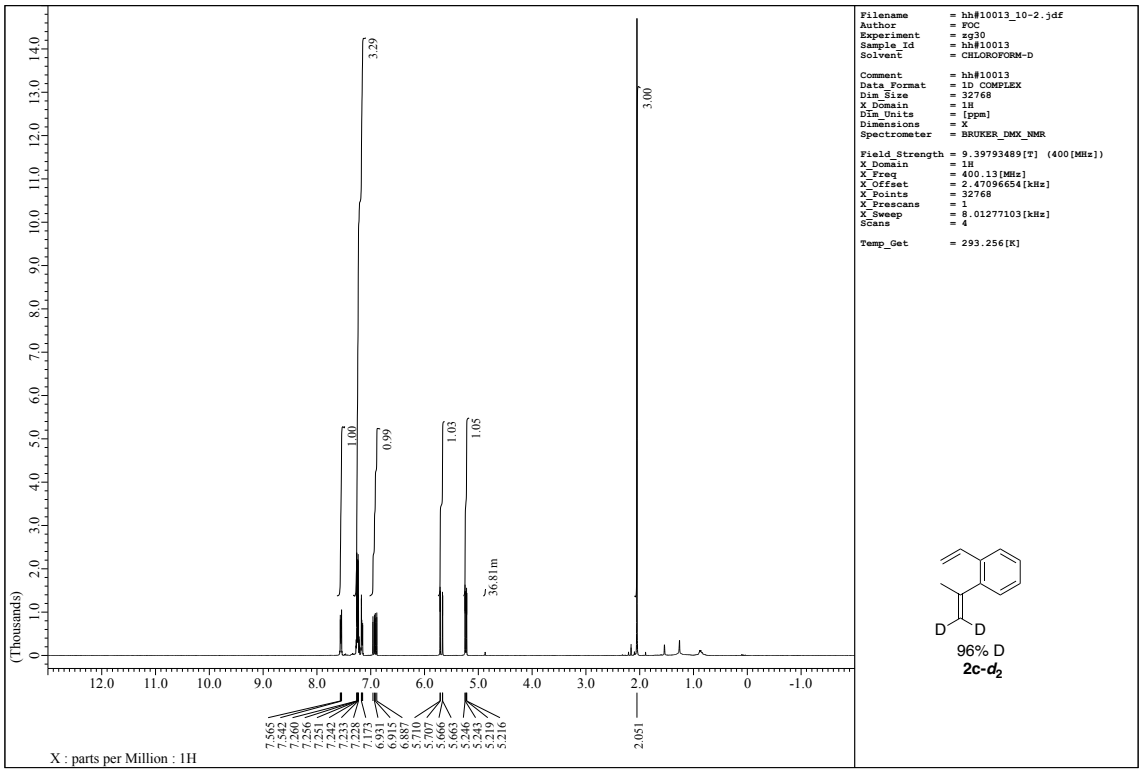

Filename = tn29-56_Carbon-2-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn29-56
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
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 = 601
Total_Scans = 601
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 18.5[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
139.585 139.499 138.655 137.505 136.403 135.986 123.936 123.687 122.413 122.125 117.430 116.423
77.316 77.000 76.684
33.082
23.806
X : parts per Million : Carbon13
S
2t

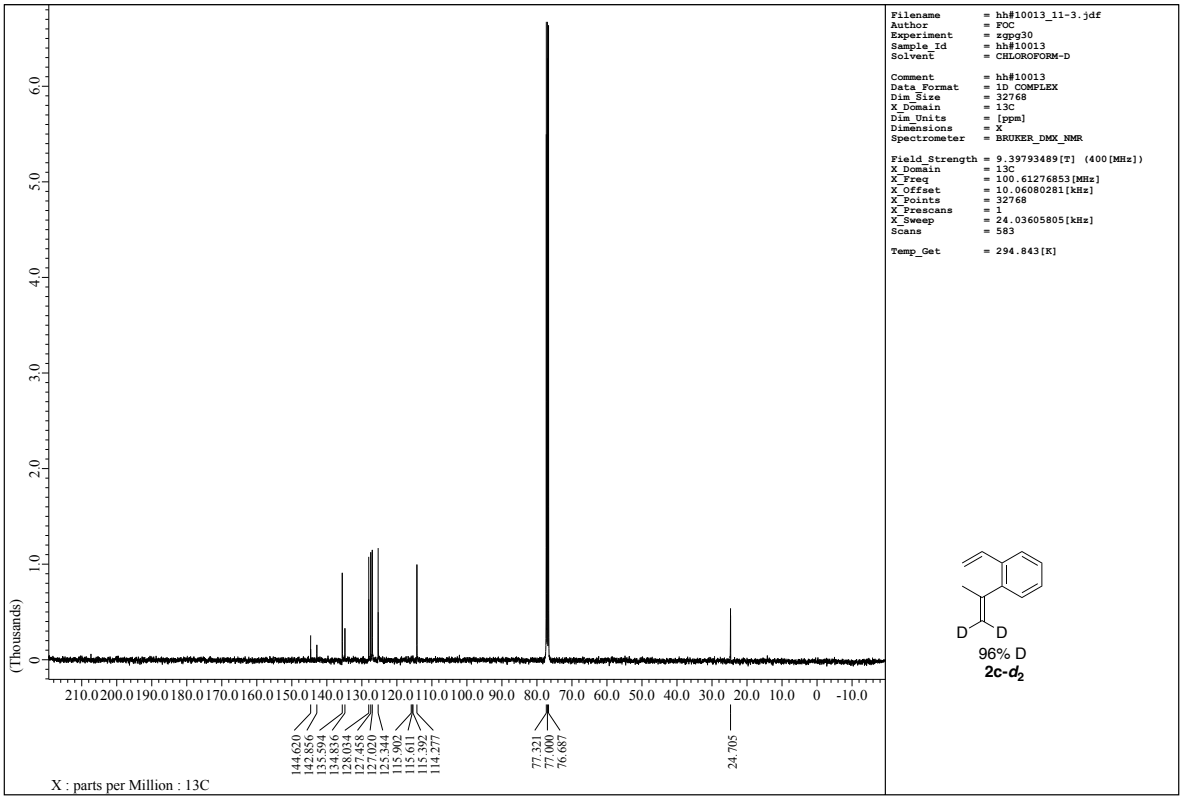

Filename = hh#7089_10-3.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#7089
Solvent = CHLOROFORM-D
Comment = hh#7089
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.4124[K]
4.33
3.10
2.06
2.06
2.22
1.00
1.08
1.01
(Thousands)
7.260
7.191
7.187
7.183
7.181
7.176
7.170
7.167
7.161
6.000
5.975
5.958
5.949
5.933
5.042
4.836
4.834
4.549
4.547
3.403
3.399
3.395
3.387
3.383
3.349
1.748
X : parts per Million : 1H
2u

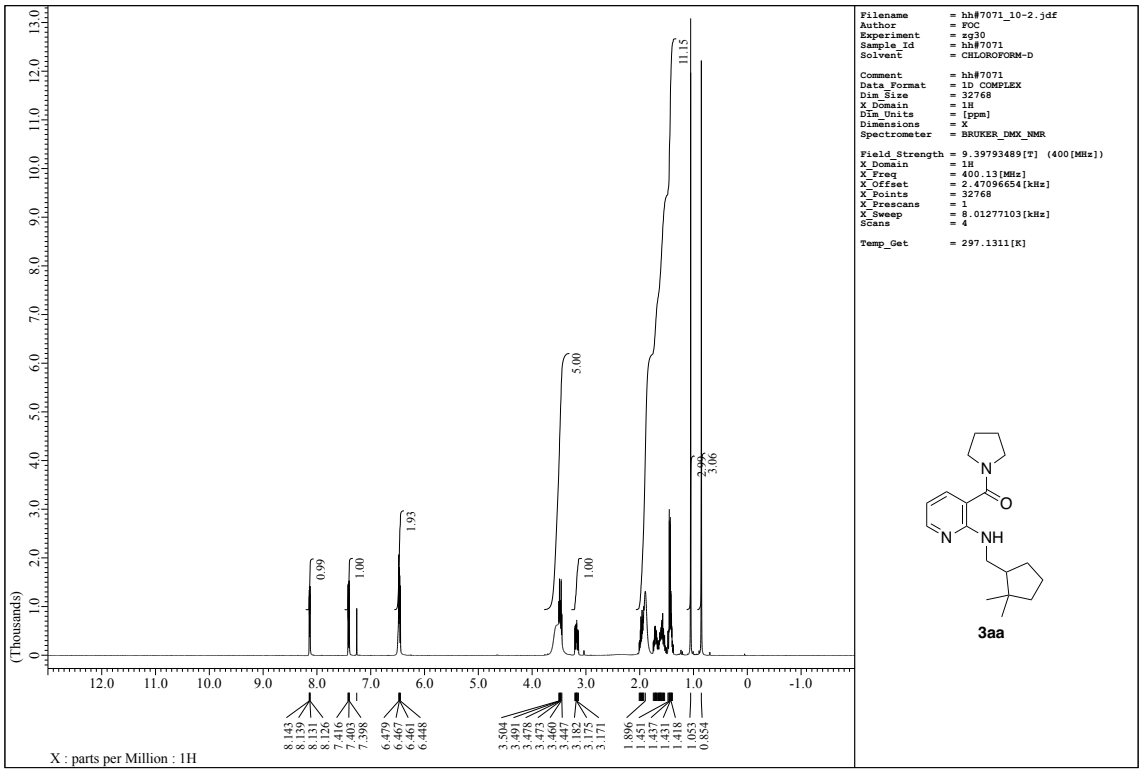

Filename = hh#7089_11-4.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#7089
Solvent = CHLOROFORM-D
Comment = hh#7089
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 415
Temp_Get = 298.9503[K]
(Thousands)
144.591
138.430
137.584
137.176
130.141
129.492
126.444
126.240
115.574
111.776
77.313
77.000
76.679
41.174
37.019
22.715
X : parts per Million : 13C
2u

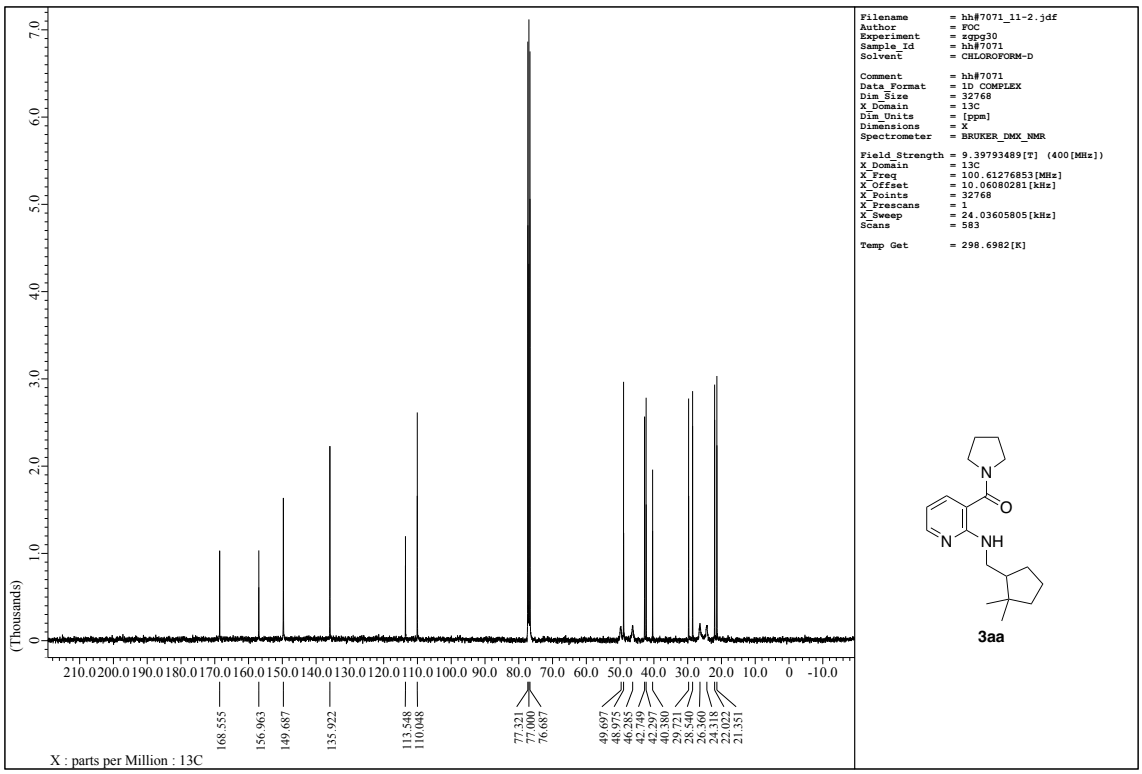

Filename = hh#8075_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8075
Solvent = CHLOROFORM-D
Comment = hh#8075
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 16
Temp_Get = 298.6884[K]
6.14
0.96
1.02
1.00
0.93
8.181
8.172
7.324
7.320
7.305
7.302
7.260
6.550
6.538
6.532
6.519
5.878
3.054
(Thousands)
X : parts per Million : 1H
N
O
N
NHCD3
1b-d3

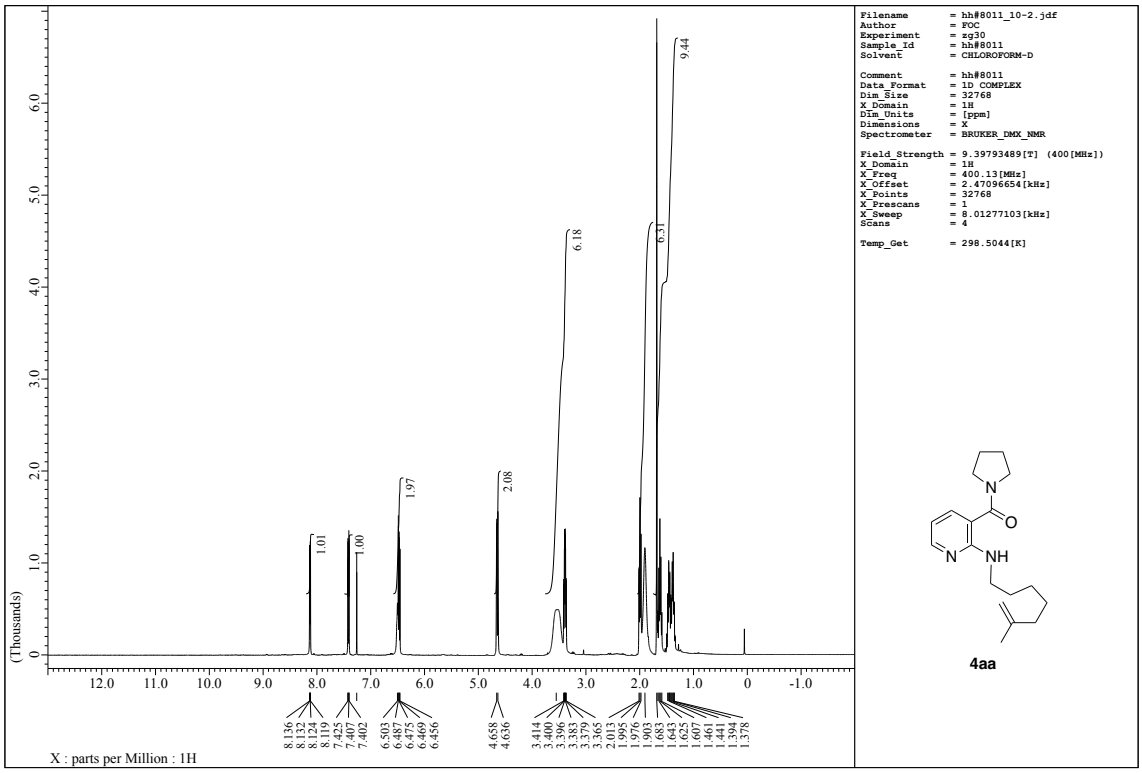

Filename = hh#9087_11-3.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 11264
Temp_Get = 300.6166[K]
170.246
157.262
149.607
135.820
113.489
110.624
77.321
77.000
76.687
37.303
27.701
27.490
27.286
(Thousands)
X : parts per Million : 13C
N
O
N
NHCD3
1b-d3

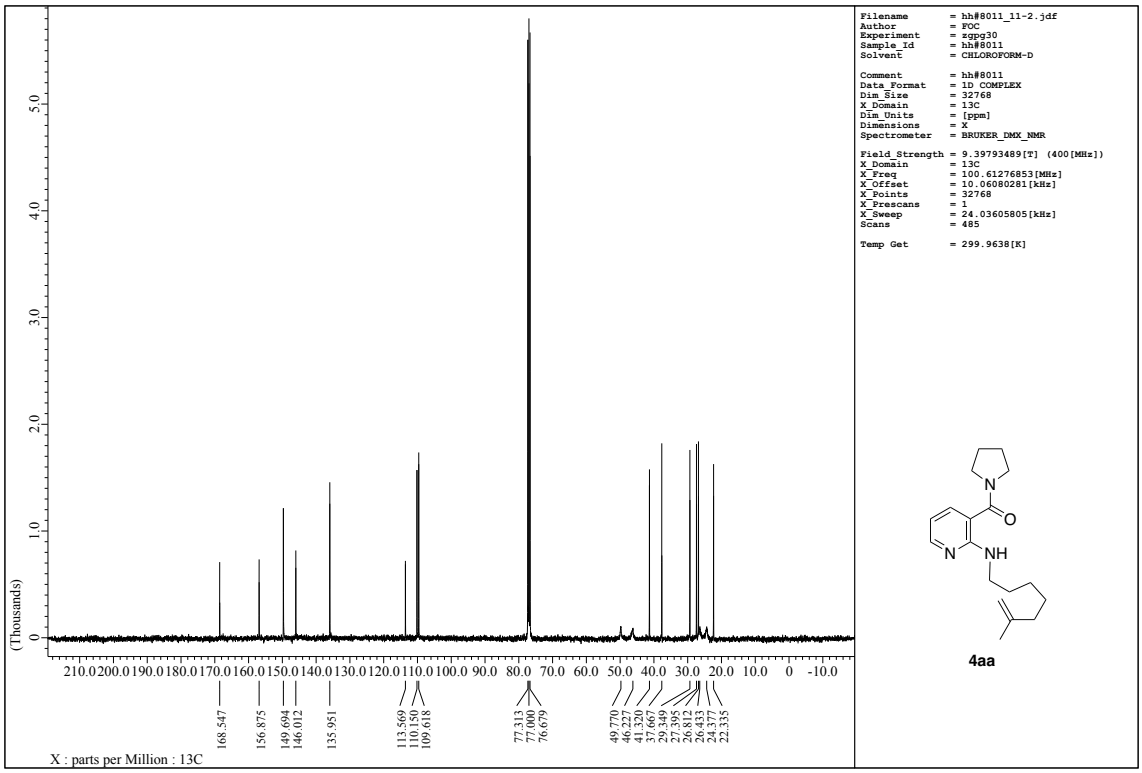

Filename = hh#10013_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#10013
Solvent = CHLOROFORM-D
Comment = hh#10013
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 293.256[K]
3.29
3.00
1.00
0.99
1.03
1.05
36.81m
(Thousands)
7.565
7.542
7.260
7.256
7.251
7.242
7.238
7.173
6.931
6.915
6.887
5.710
5.707
5.665
5.246
5.243
5.219
5.216
2.051
X : parts per Million : 1H
D
D
96% D
2c-d_2

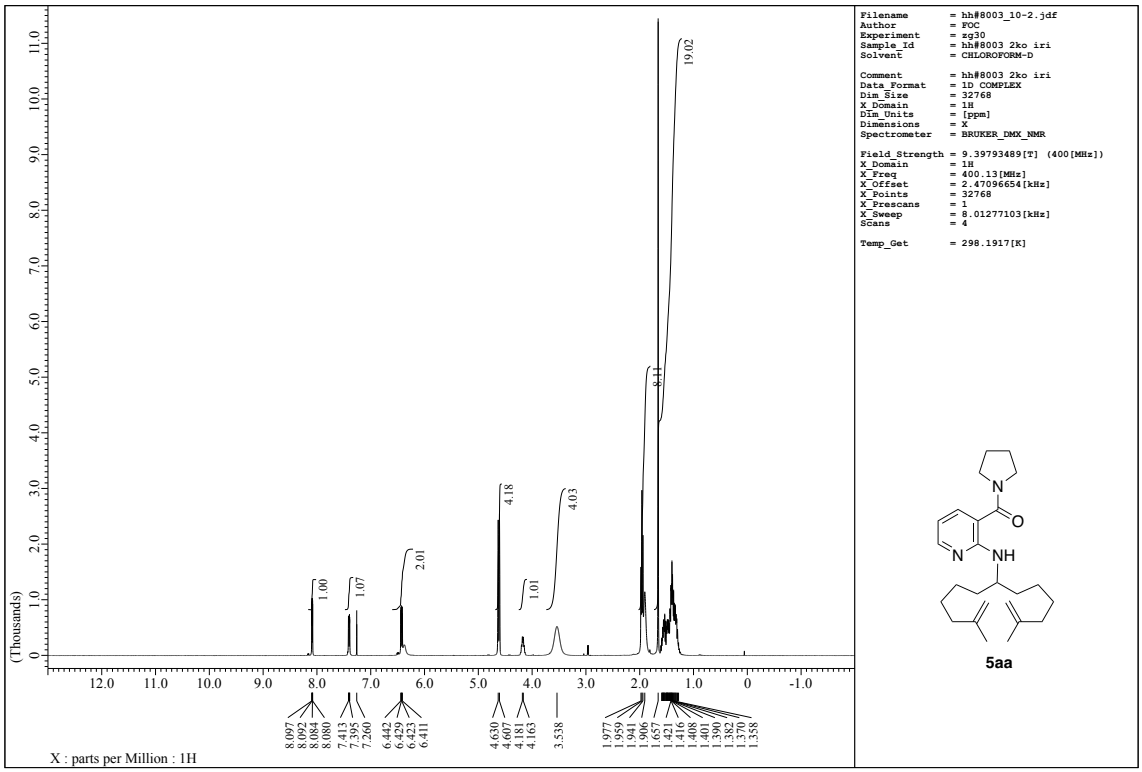

Filename = hh#10013_11-3.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#10013
Solvent = CHLOROFORM-D
Comment = hh#10013
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 583
Temp_Get = 294.843[K]
(Thousands)
144.620
142.856
135.594
134.836
128.034
127.458
127.010
125.344
115.902
115.611
115.392
114.277
77.321
77.000
76.687
24.705
X : parts per Million : 13C
D
D
96% D
2c-d_2

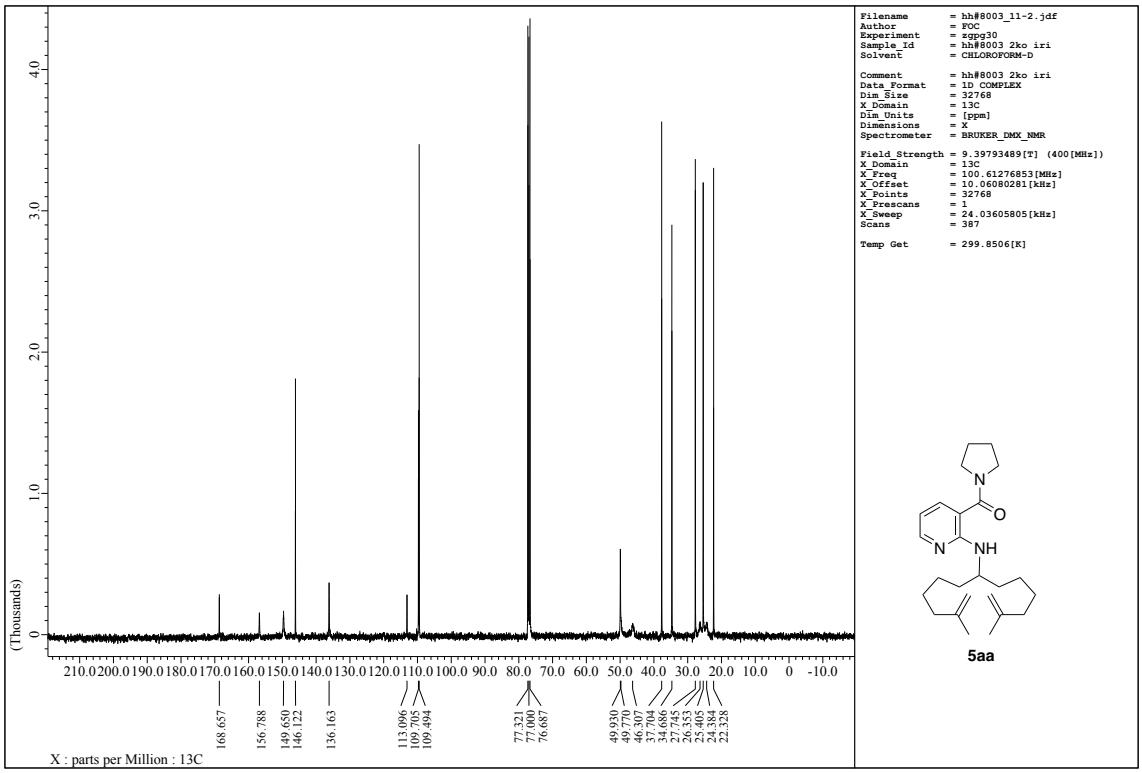

Filename = hh#7071_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#7071
Solvent = CHLOROFORM-D
Comment = hh#7071
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.1311[K]
(Thousands)
X : parts per Million : 1H
3aa

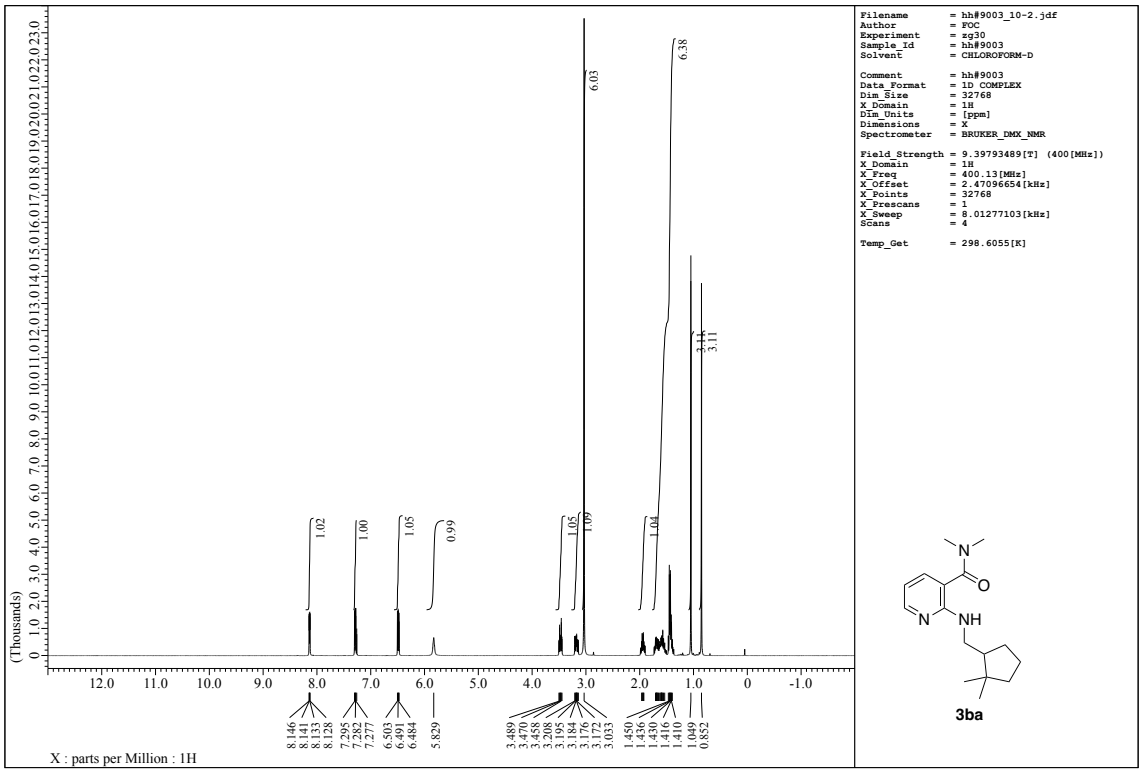

Filename = hh#7071_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#7071
Solvent = CHLOROFORM-D
Comment = hh#7071
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 583
Temp Get = 298.6982[K]
(Thousands)
X : parts per Million : 13C
3aa

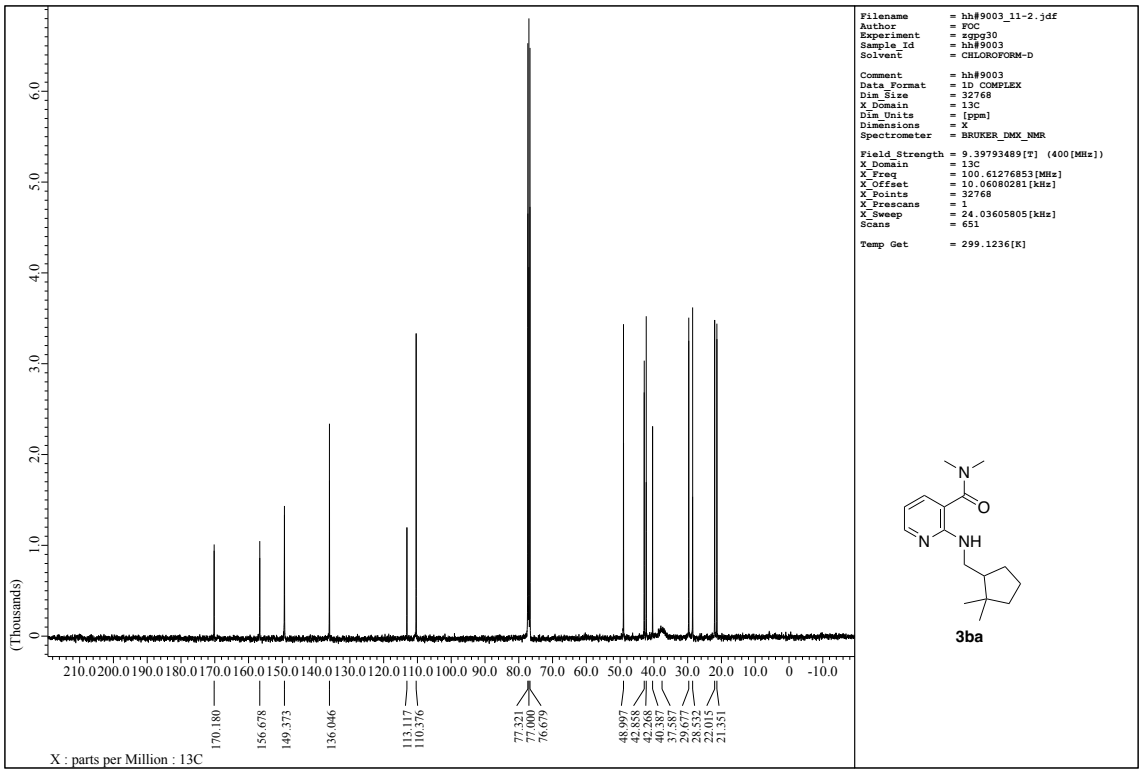

Filename = hh#8011_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8011
Solvent = CHLOROFORM-D
Comment = hh#8011
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.5044[K]
(Thousands)
X : parts per Million : 1H
4aa

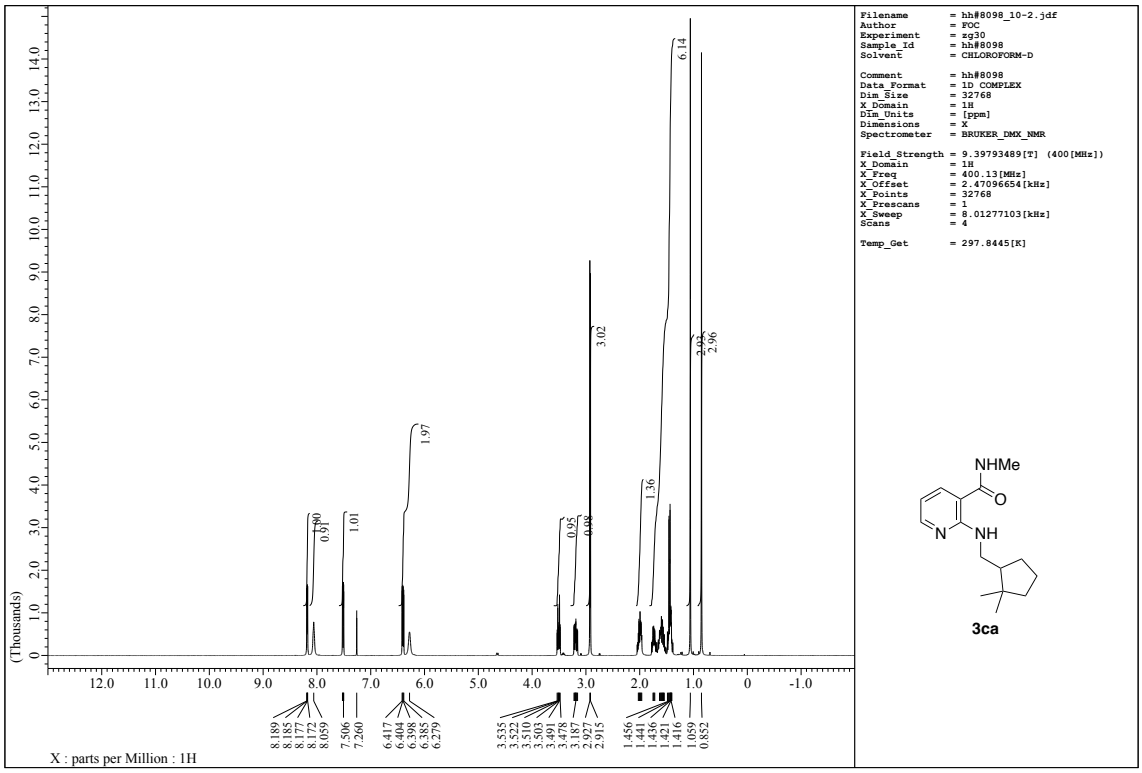

Filename = hh#8011_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8011
Solvent = CHLOROFORM-D
Comment = hh#8011
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 485
Temp Get = 299.9638[K]
(Thousands)
X : parts per Million : 13C
4aa

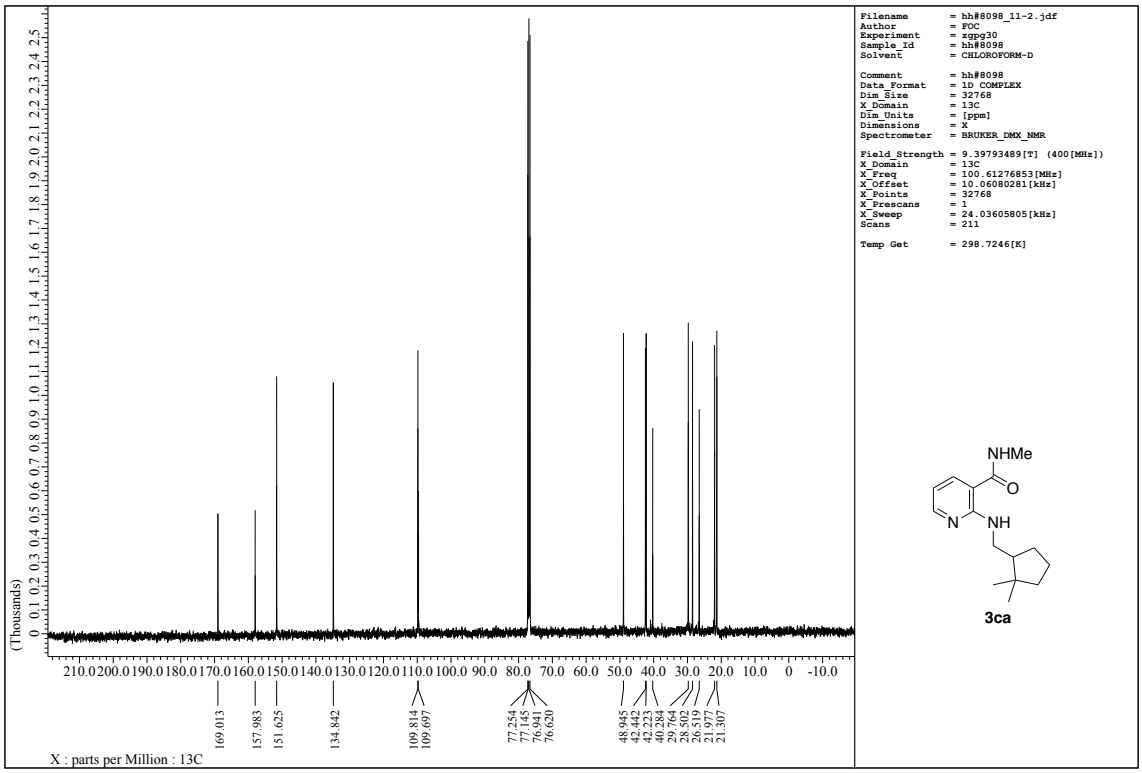

Filename = hh#8003_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8003 2ko iri
Solvent = CHLOROFORM-D
Comment = hh#8003 2ko iri
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 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 = 298.1917[K]
(Thousands)
1.00
1.07
2.01
4.18
1.01
4.03
8.11
19.02
8.097
8.092
8.084
8.080
7.413
7.395
7.260
6.442
6.429
6.423
6.411
4.630
4.607
4.181
4.163
3.538
1.977
1.959
1.941
1.906
1.657
1.421
1.416
1.406
1.401
1.390
1.382
1.370
1.358
X : parts per Million : 1H
5aa

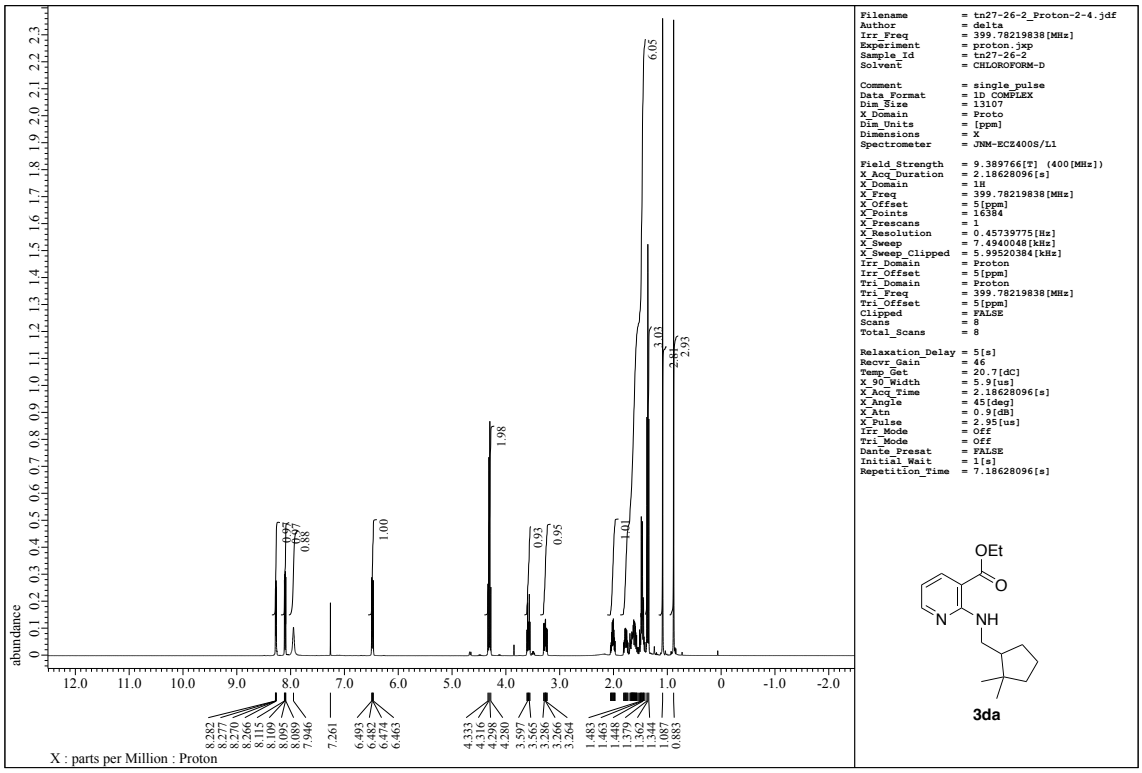

Filename = hh#8003_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8003 2ko iri
Solvent = CHLOROFORM-D
Comment = hh#8003 2ko iri
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 387
Temp Get = 299.8506[K]
(Thousands)
168.657
156.788
149.650
146.122
136.163
113.096
109.705
109.494
77.321
77.000
76.687
49.930
49.770
46.307
37.704
34.686
27.745
26.353
25.405
24.384
22.328
X : parts per Million : 13C
5aa


Filename = hh#9003_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9003
Solvent = CHLOROFORM-D
Comment = hh#9003
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 = 298.6055[K]
(Thousands)
6.38
6.03
3.14
3.11
1.02
1.00
1.05
0.99
1.05
1.09
1.04
X : parts per Million : 1H
3ba


Filename = hh#9003_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9003
Solvent = CHLOROFORM-D
Comment = hh#9003
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 = 651
Temp_Get = 299.1236[K]
(Thousands)
170.180
156.678
149.373
136.046
113.117
110.376
77.321
77.000
76.679
48.997
42.858
42.268
40.387
37.587
29.677
28.532
22.015
21.351
X : parts per Million : 13C
3ba


Filename = hh#8098_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8098
Solvent = CHLOROFORM-D
Comment = hh#8098
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 = 297.8445[K]
NHMe
O
N
NH
3ca
X : parts per Million : 1H


Filename = hh#8098_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8098
Solvent = CHLOROFORM-D
Comment = hh#8098
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 = 211
Temp Get = 298.7246[K]
NHMe
O
N
NH
3ca
X : parts per Million : 13C

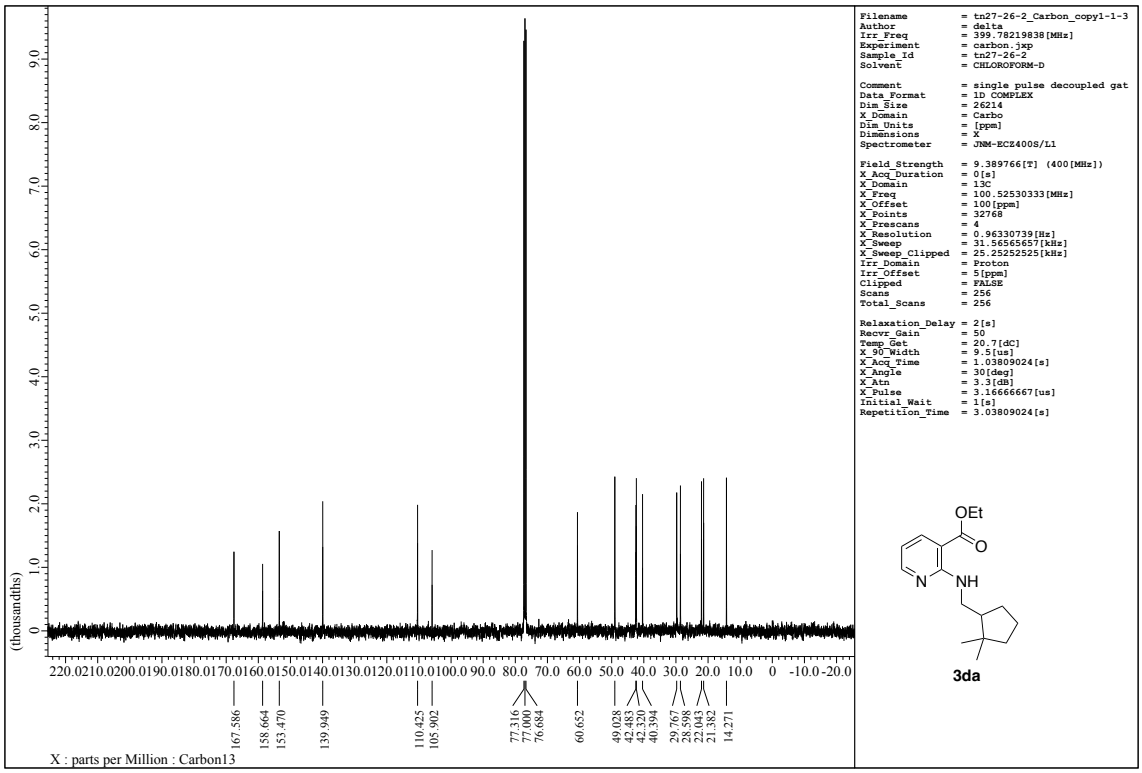


Filename = tn27-26-2_Proton-2-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn27-26-2
Solvent = CHLOROFORM-D

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 = 20.7[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.91[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]

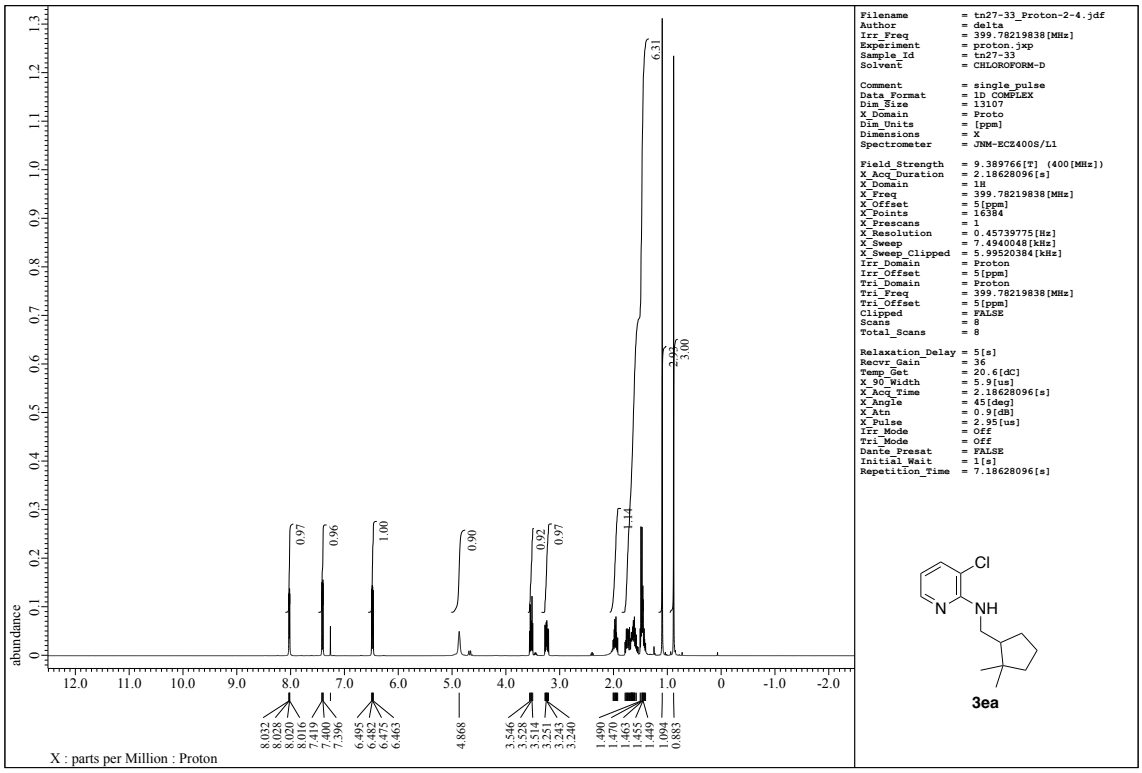


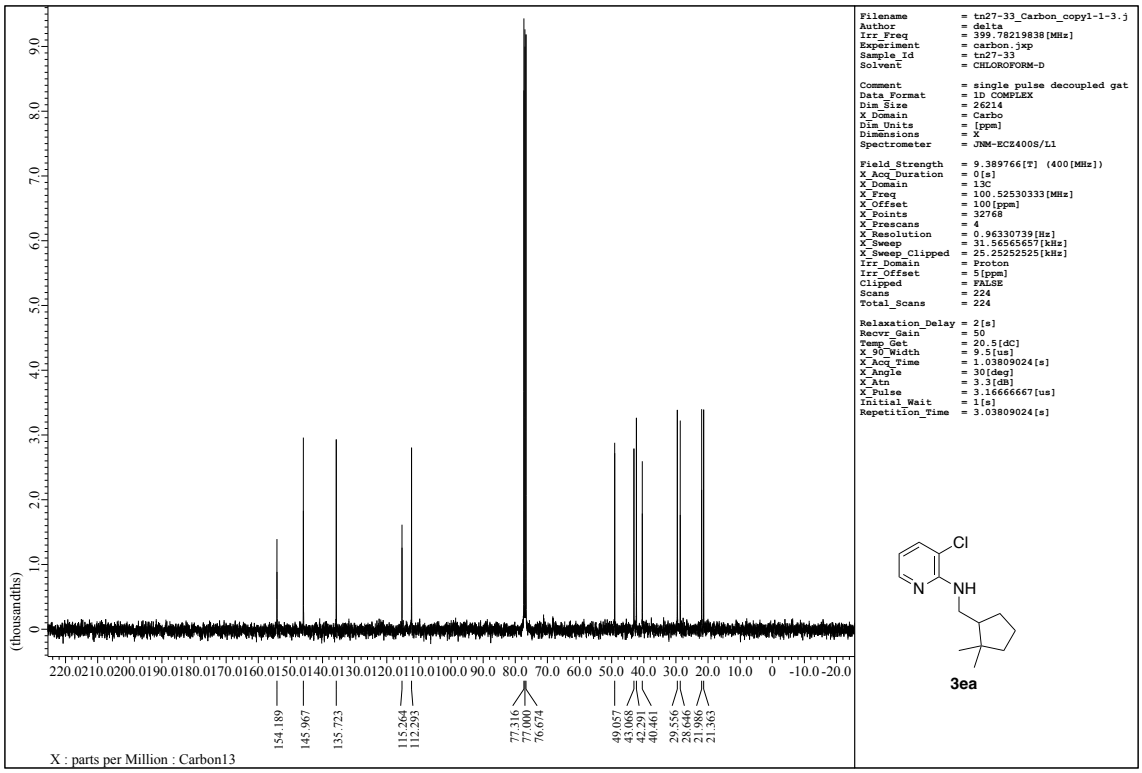
Filename = tn27-26-2_Carbon_copy1-1-3
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn27-26-2
Solvent = CHLOROFORM-D

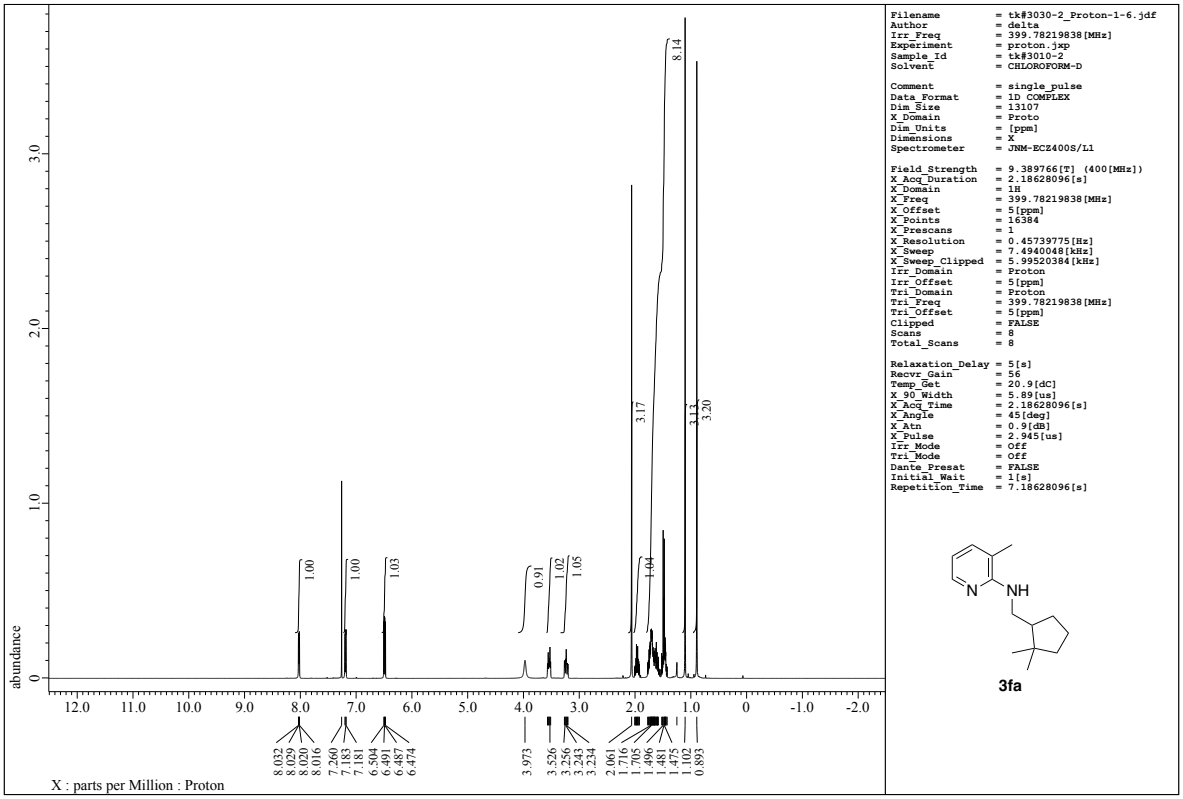
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1

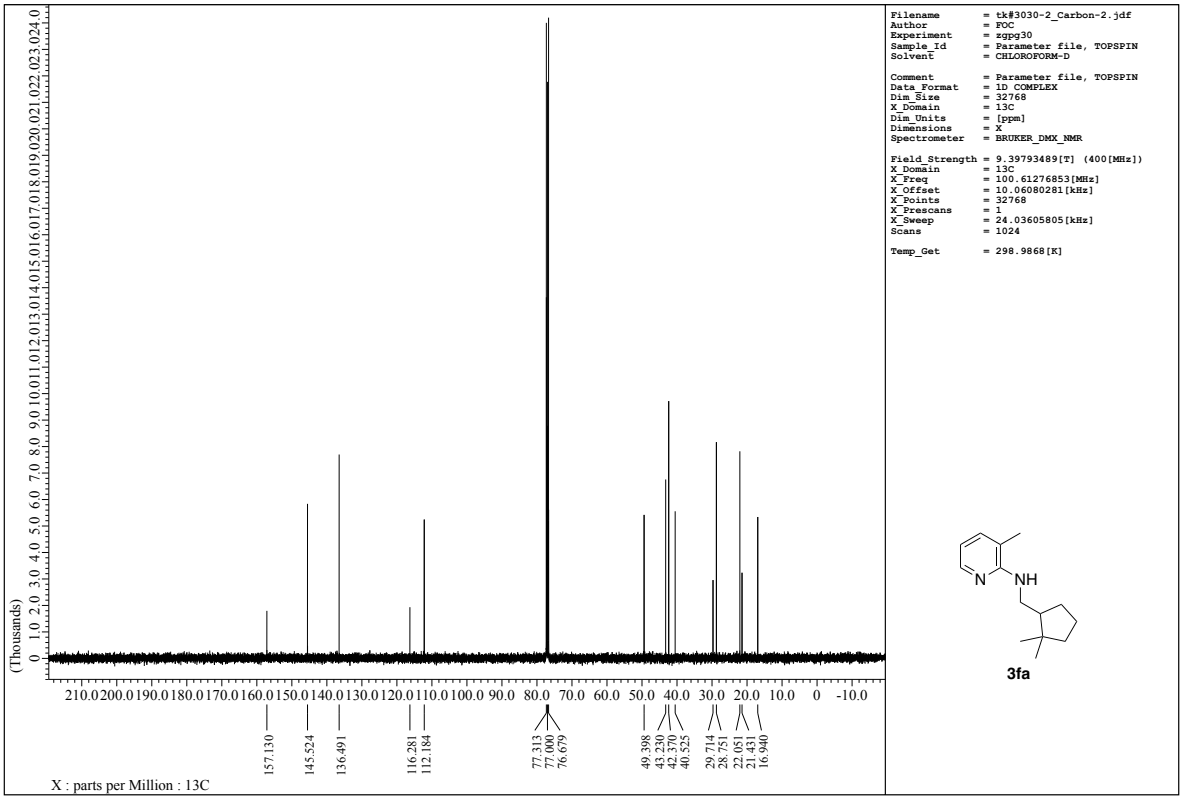
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 0[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256

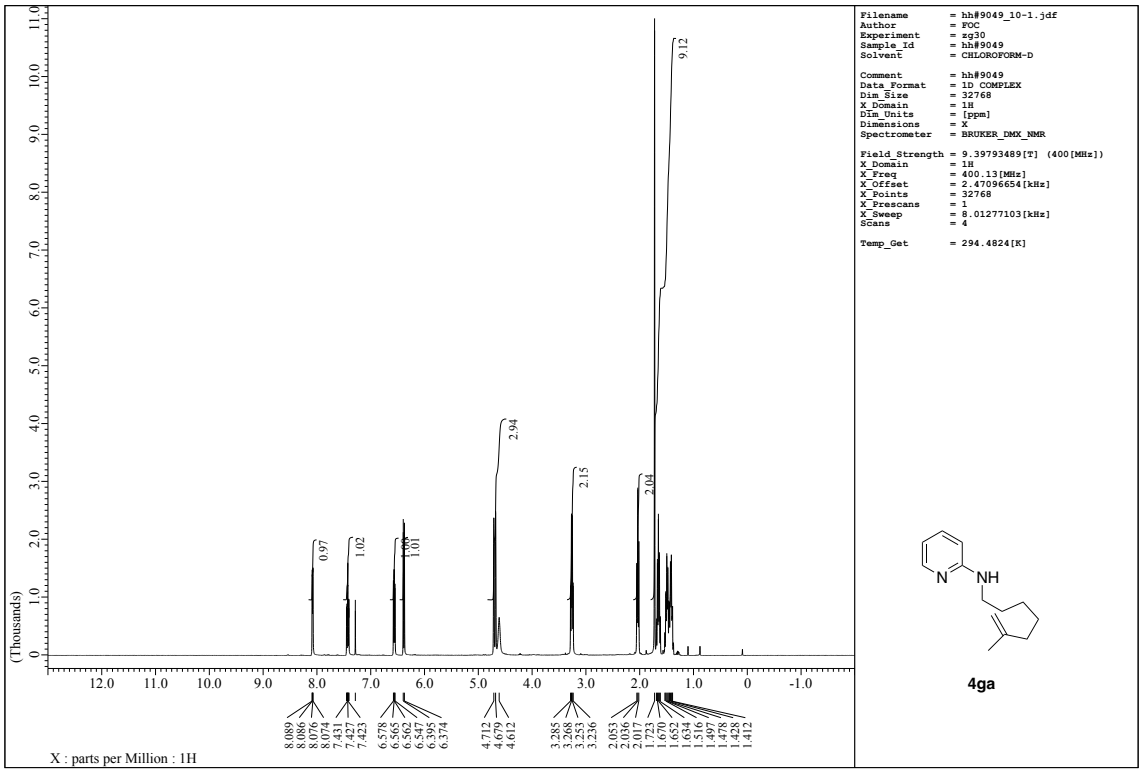
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.7[dC]
X_90_Width = 9.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.3[dB]
X_Pulse = 3.16666667[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]


Filename = tn27-33_Proton-2-4.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tn27-33
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 20.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
0.97
0.96
1.00
0.90
0.92
0.97
1.14
6.31
2.03
3.00
8.032
8.028
8.020
8.016
7.419
7.400
7.396
6.495
6.492
6.475
6.463
4.868
3.546
3.528
3.514
3.251
3.243
3.240
1.490
1.470
1.463
1.455
1.449
1.094
0.883
Cl
N
NH
3ea

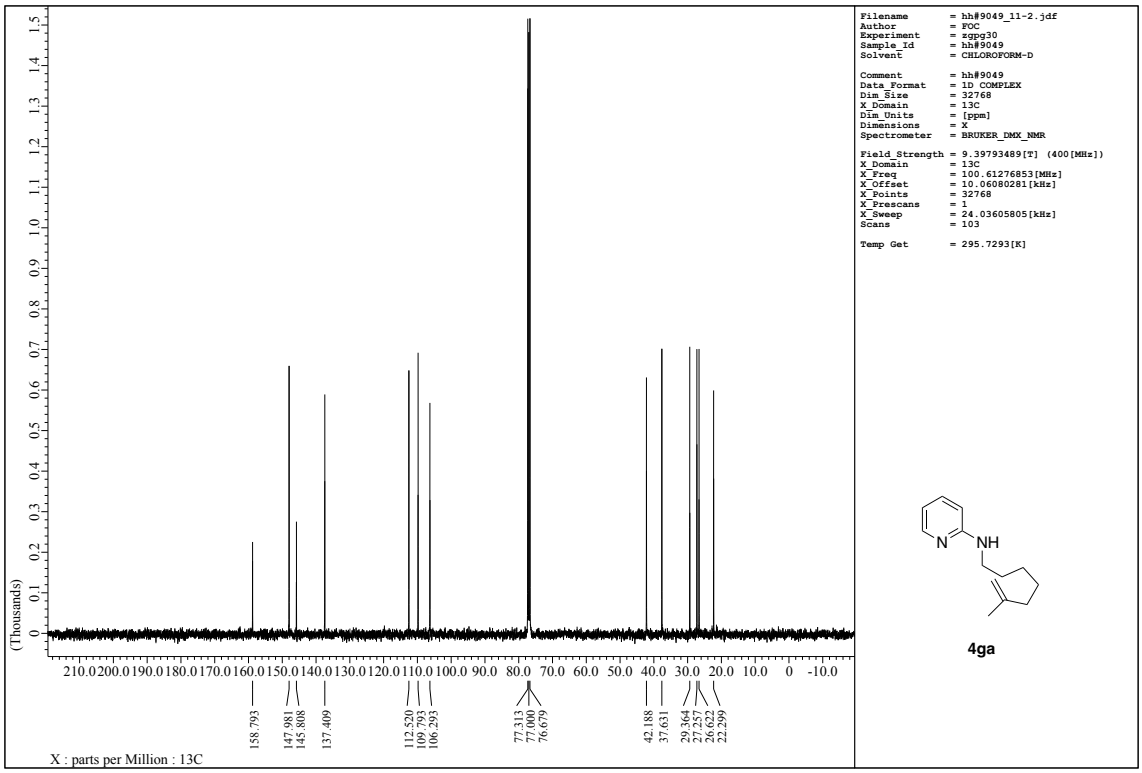

Filename = tn27-33_Carbon_copy1-1-3.j
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tn27-33
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
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 = 224
Total_Scans = 224
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 20.5[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]
(thousandths)
X : parts per Million : Carbon13
154.189
145.967
135.723
115.264
112.293
77.316
77.000
76.674
49.057
43.068
42.291
40.461
29.556
28.646
21.986
21.363
Cl
N
NH
3ea


Filename = tk#3030-2_Proton-1-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#3010-2
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 = 20.9[dC]
X_90_Width = 5.89[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.945[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3fa

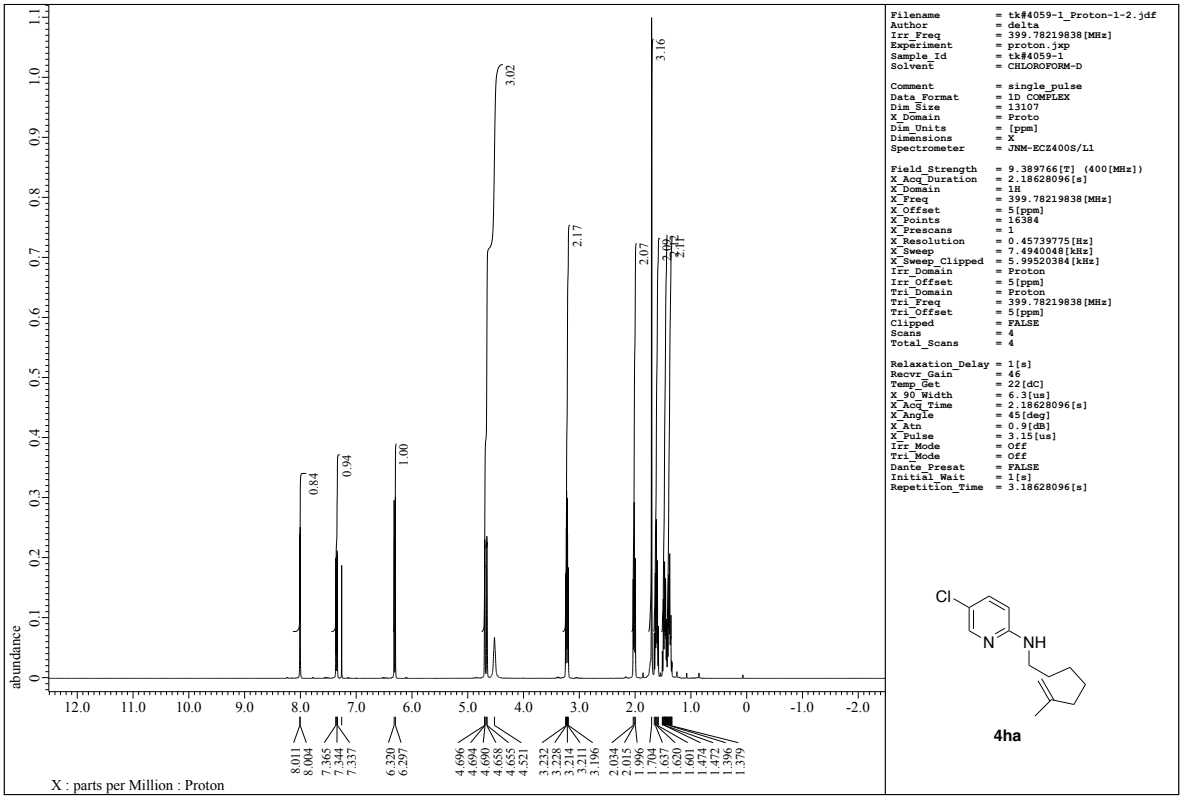

Filename = tk#3030-2_Carbon-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 298.9868[K]
(Thousands)
X : parts per Million : 13C
3fa

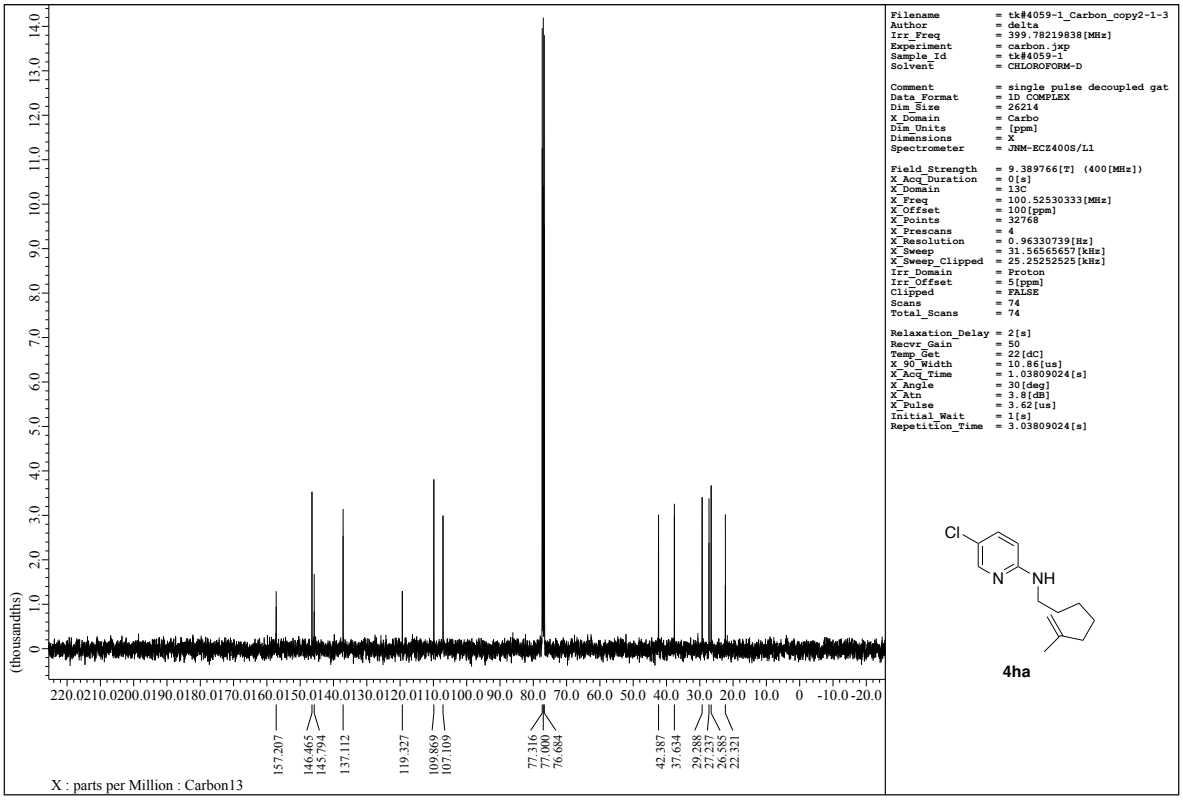

Filename = hh#9049_10-1.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9049
Solvent = CHLOROFORM-D
Comment = hh#9049
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 294.4824[K]
9.12
2.94
2.15
2.04
0.97
1.02
1.08
1.01
(Thousands)
X : parts per Million : 1H
8.089
8.086
8.076
8.074
7.431
7.427
7.423
6.578
6.565
6.562
6.547
6.395
6.374
4.712
4.679
4.612
3.285
3.268
3.253
3.236
2.053
2.036
2.017
1.723
1.670
1.652
1.634
1.516
1.497
1.478
1.433
1.412
N
NH

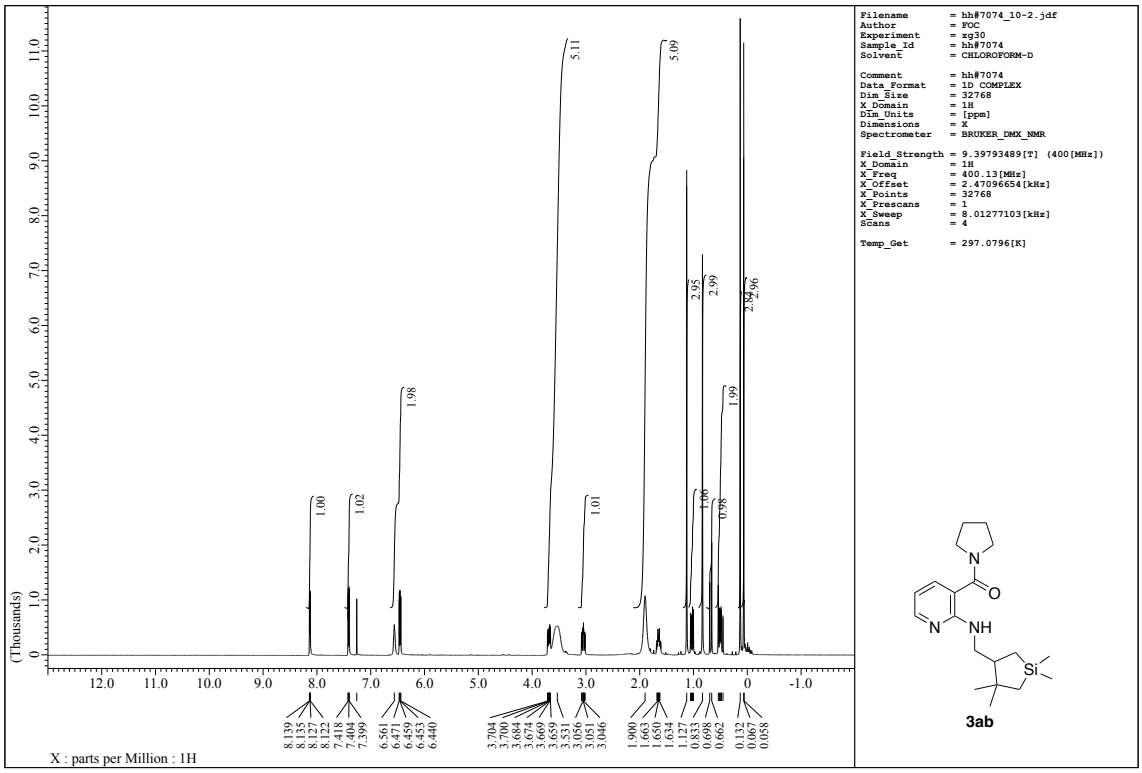
4ga

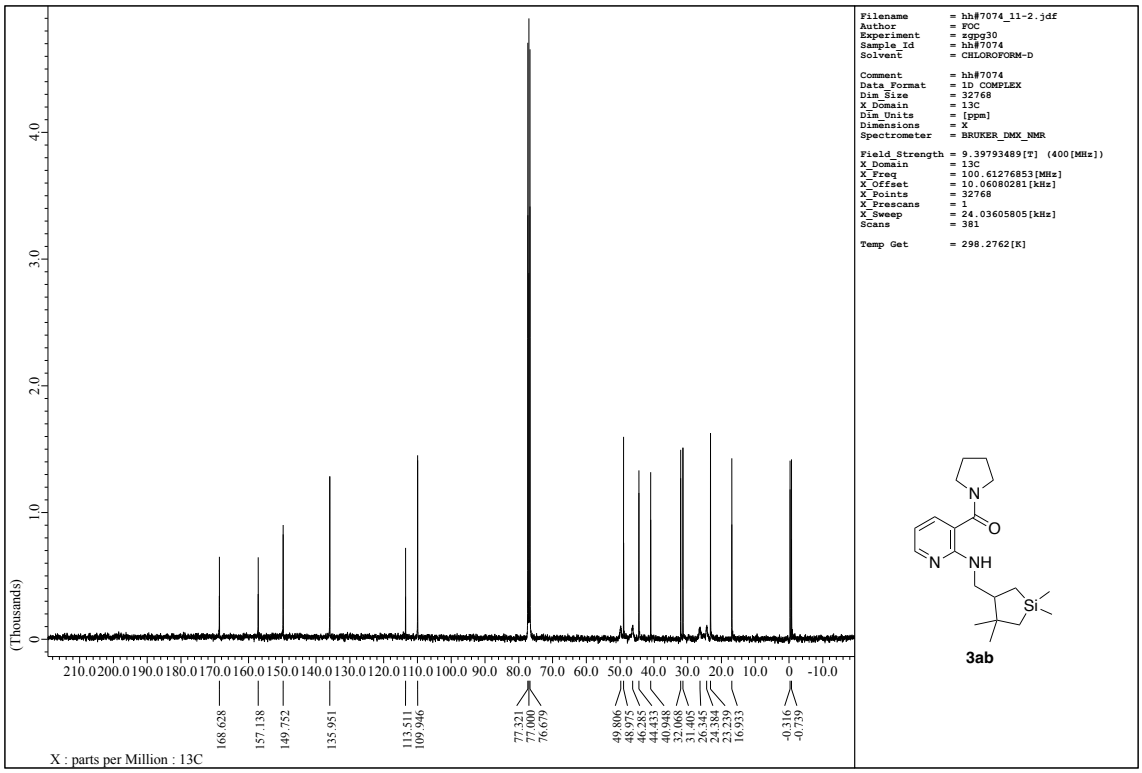

Filename = hh#9049_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9049
Solvent = CHLOROFORM-D
Comment = hh#9049
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 103
Temp Get = 295.7293[K]
(Thousands)
X : parts per Million : 13C
158.793
147.981
145.808
137.409
112.520
109.793
106.293
77.313
77.000
76.679
42.188
37.631
29.364
27.257
26.622
22.299
N
NH

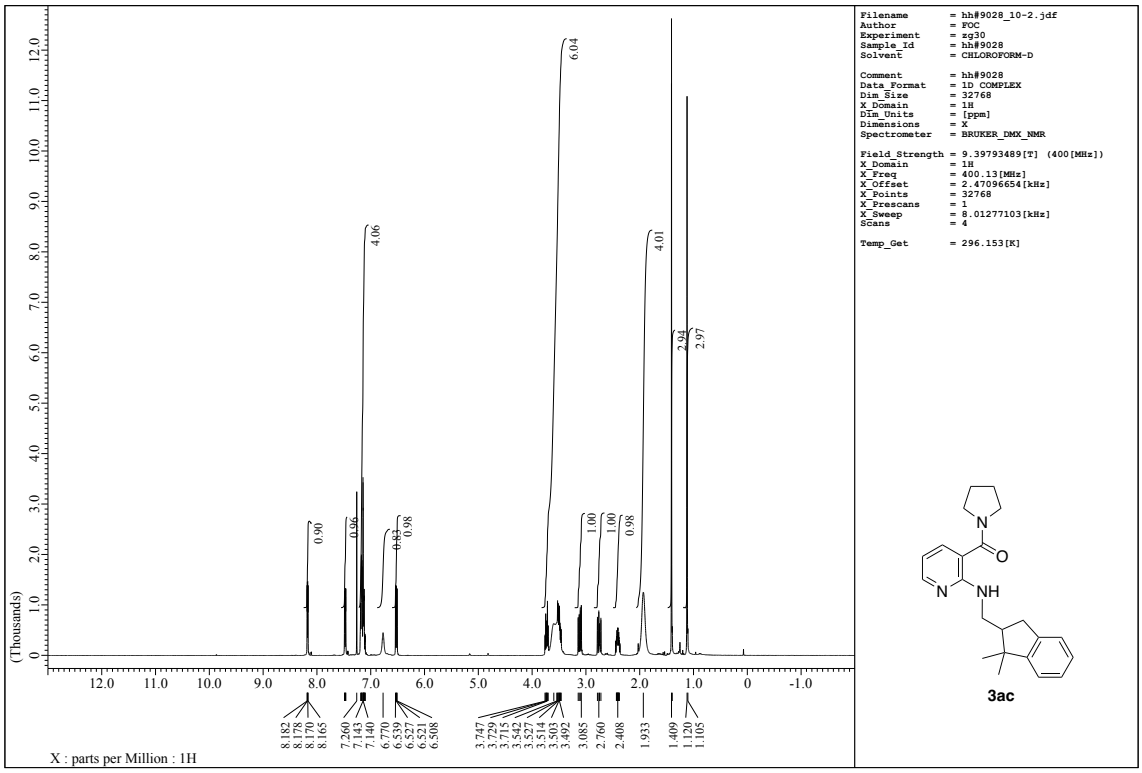
4ga

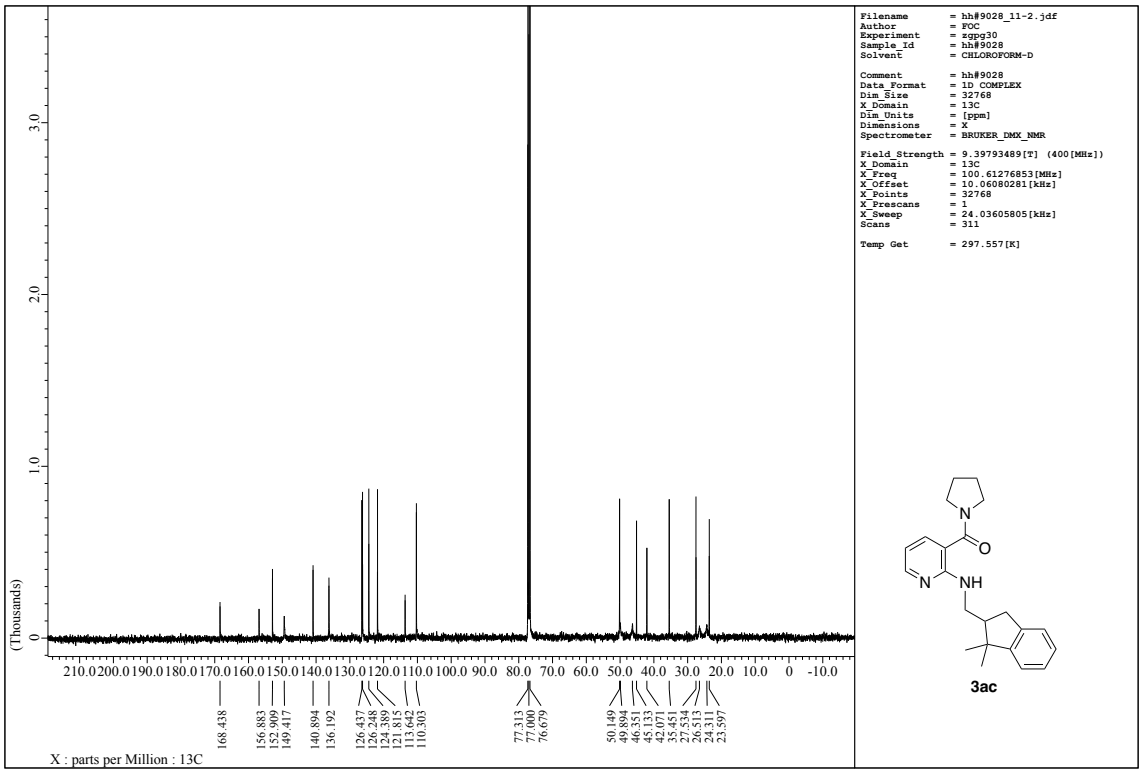

abundance
X : parts per Million : Proton
0.84
0.94
1.00
3.02
2.17
2.07
3.16
Filename = tk#4059-1_Proton-1-2.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#4059-1
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 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 = 4
Total_Scans = 4
Relaxation_Delay = 1[s]
Recvr_Gain = 46
Temp_Get = 22[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 = 3.18628096[s]
Cl
N
NH
4ha

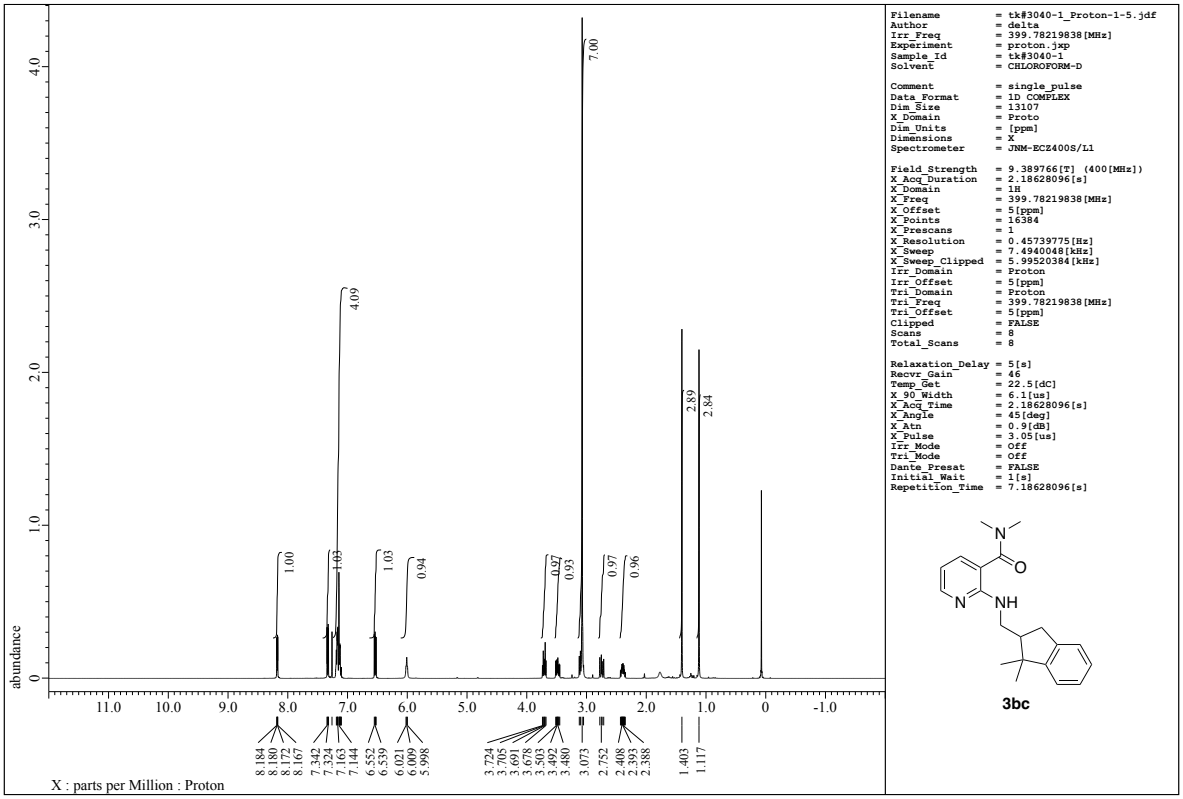

(thousandths)
X : parts per Million : Carbon13
157.207
146.465
145.794
137.112
119.327
109.869
107.109
77.316
77.000
76.684
42.387
37.634
29.288
27.237
25.585
22.321
Filename = tk#4059-1_Carbon_copy2-1-3
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#4059-1
Solvent = 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 = 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 = 74
Total_Scans = 74
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22[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]
Cl
N
NH
4ha

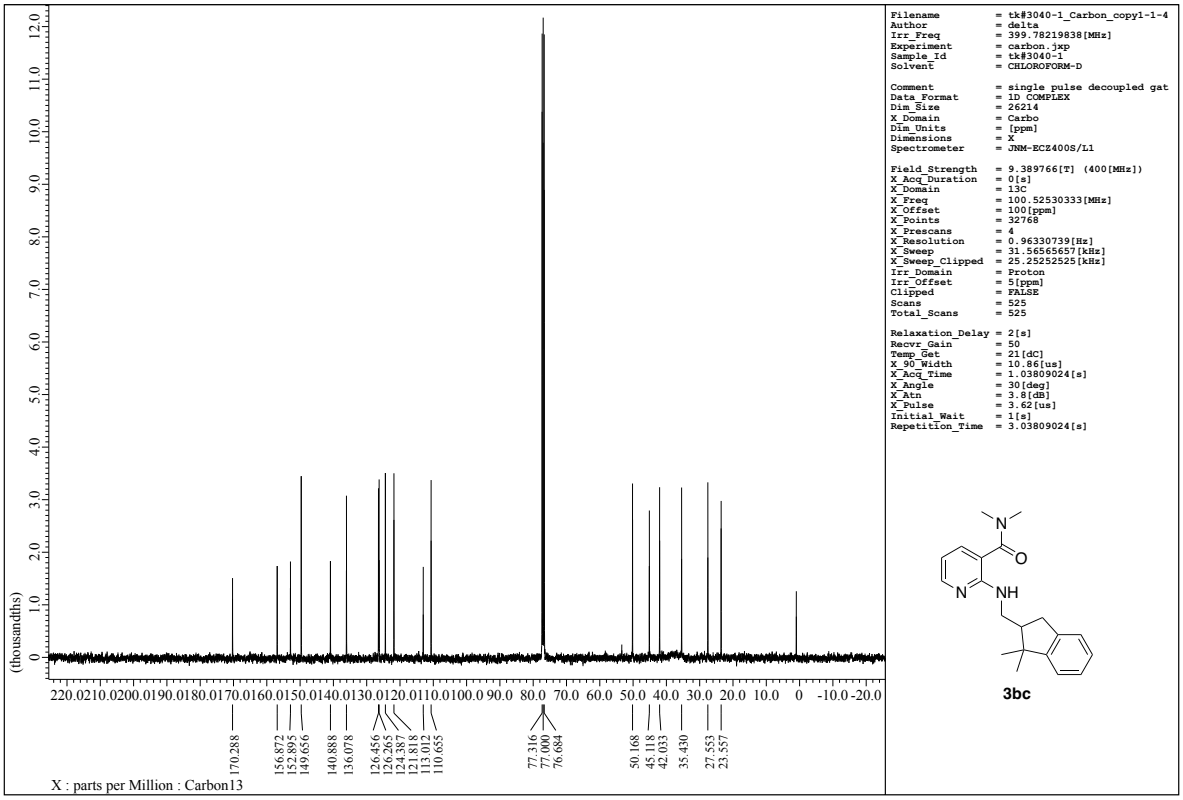

Filename = hh#7074_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#7074
Solvent = CHLOROFORM-D
Comment = hh#7074
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.0796[K]
(Thousands)
X : parts per Million : 1H
3ab

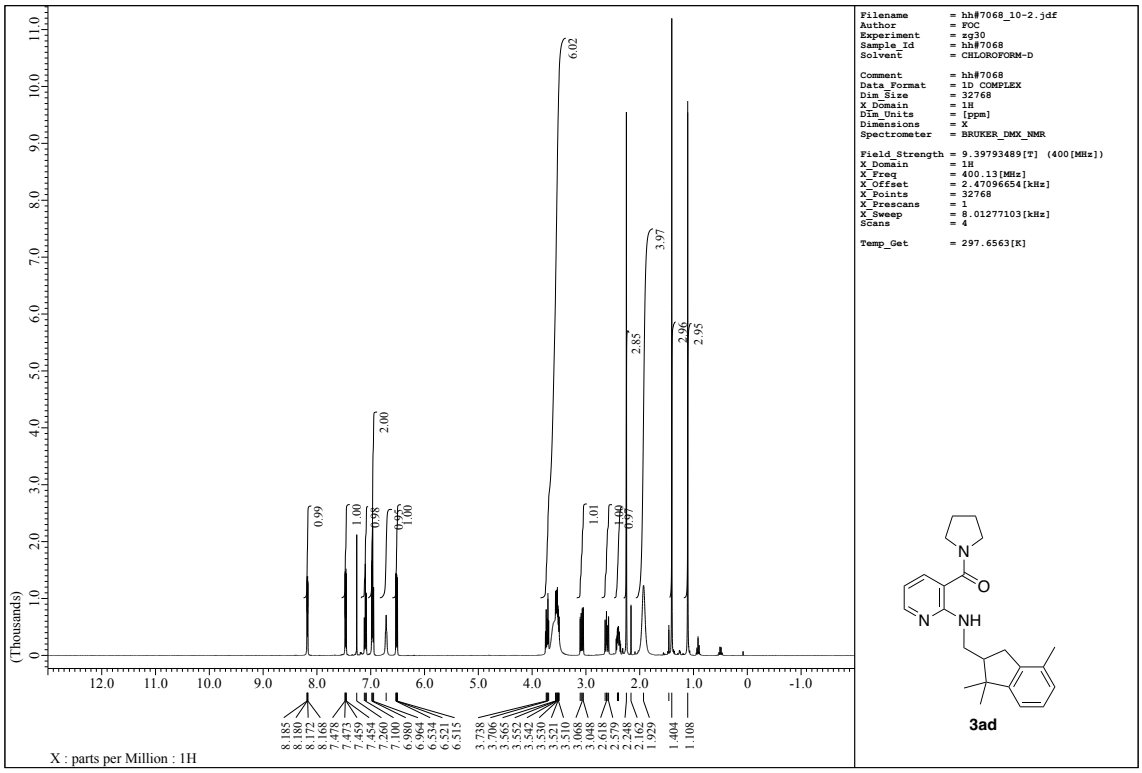

Filename = hh#7074_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#7074
Solvent = CHLOROFORM-D
Comment = hh#7074
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 381
Temp Get = 298.2762[K]
(Thousands)
X : parts per Million : 13C
3ab

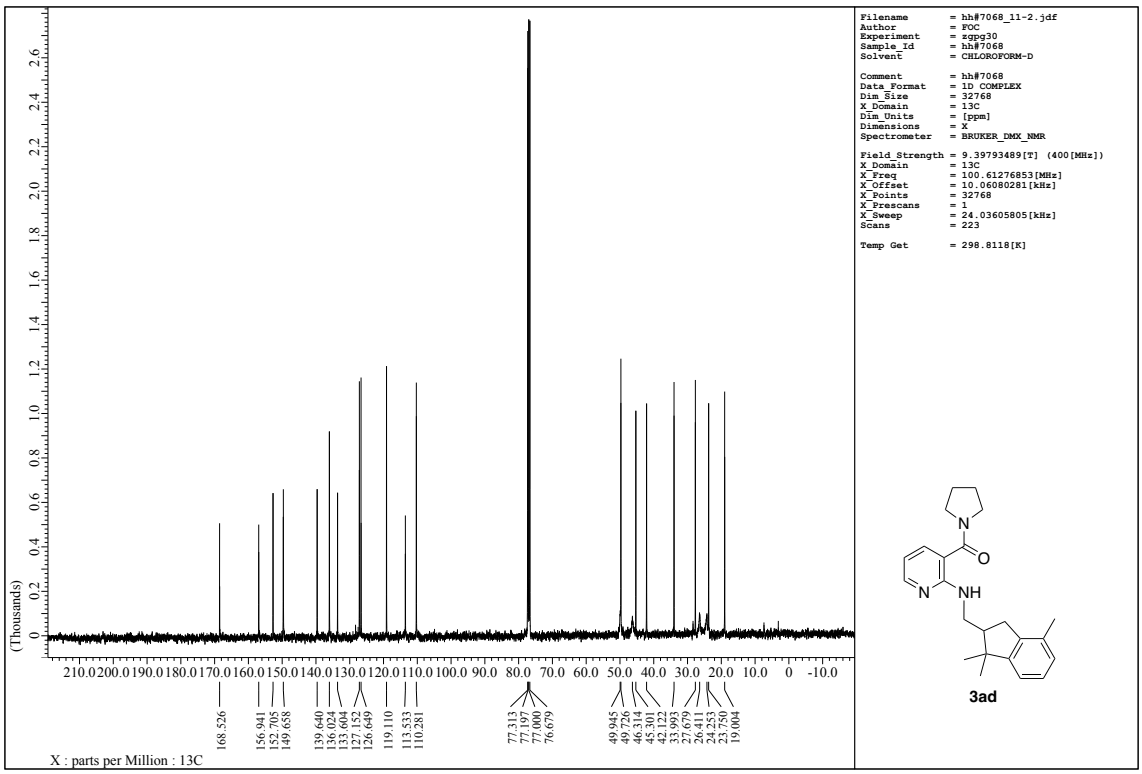

Filename = hh#9028_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9028
Solvent = CHLOROFORM-D
Comment = hh#9028
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 296.153[K]
(Thousands)
X : parts per Million : 1H
3ac

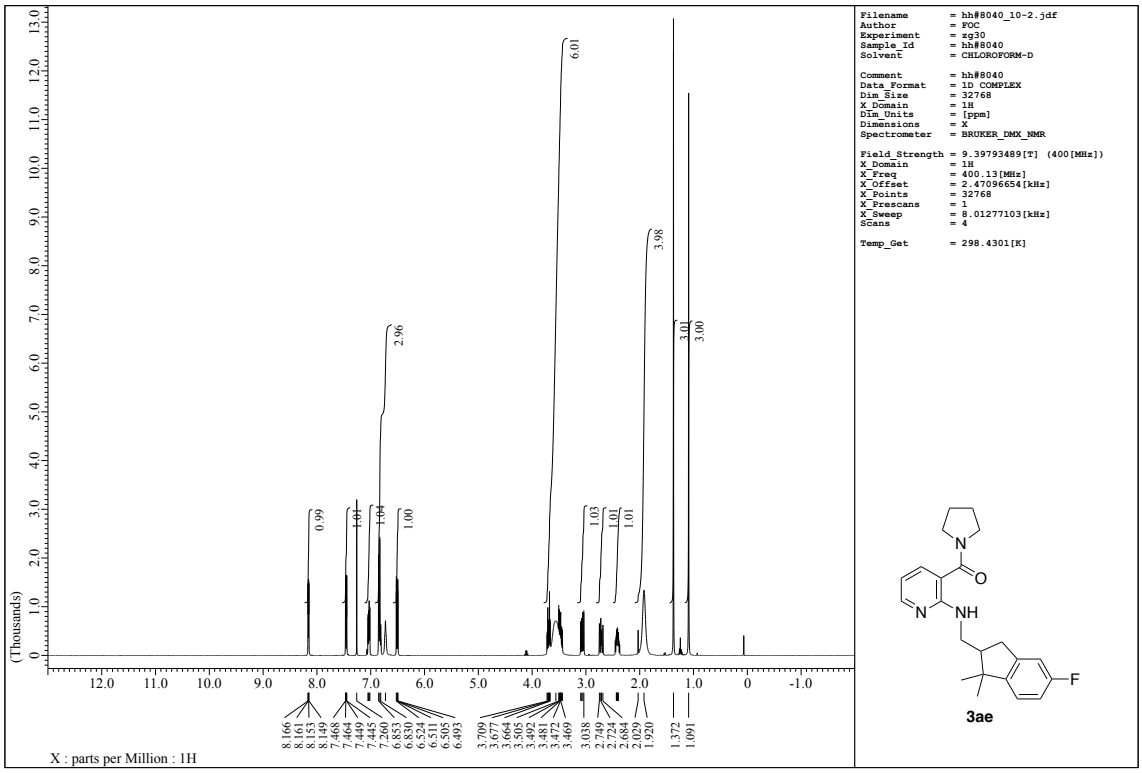

Filename = hh#9028_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9028
Solvent = CHLOROFORM-D
Comment = hh#9028
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 311
Temp Get = 297.557[K]
(Thousands)
X : parts per Million : 13C
3ac

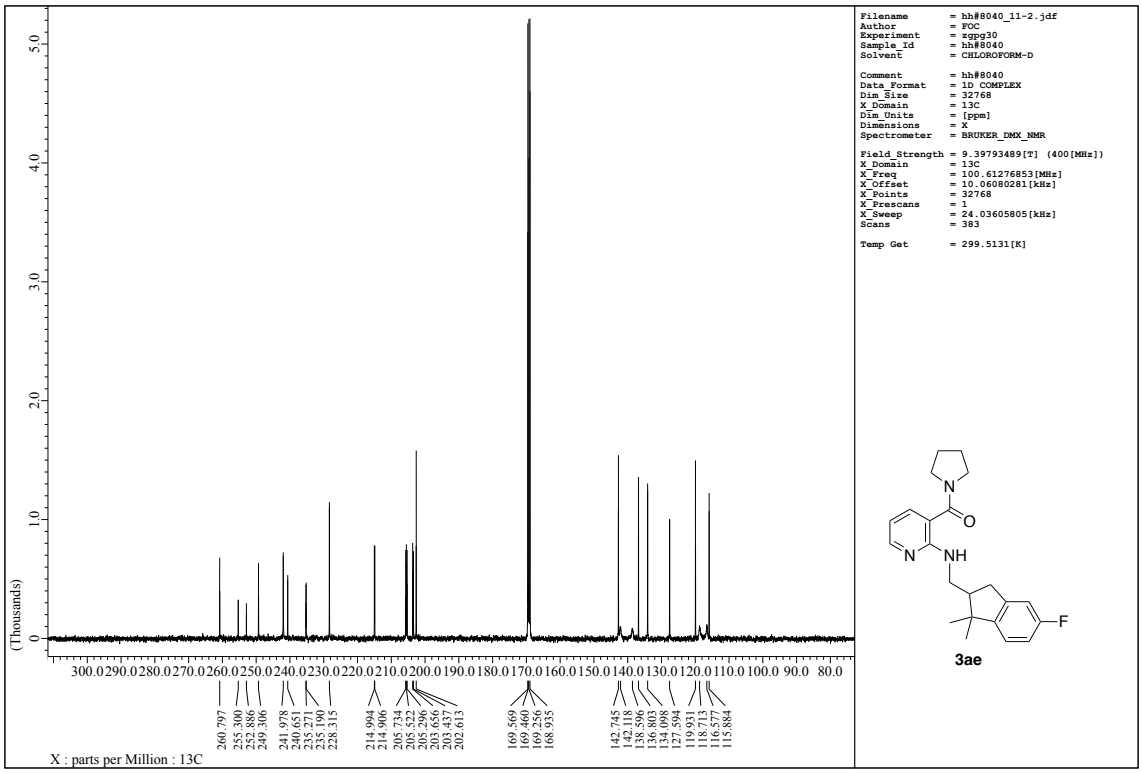

Filename = tk#3040-1_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#3040-1
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 22.5[dC]
X_90_Width = 6.1[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3bc

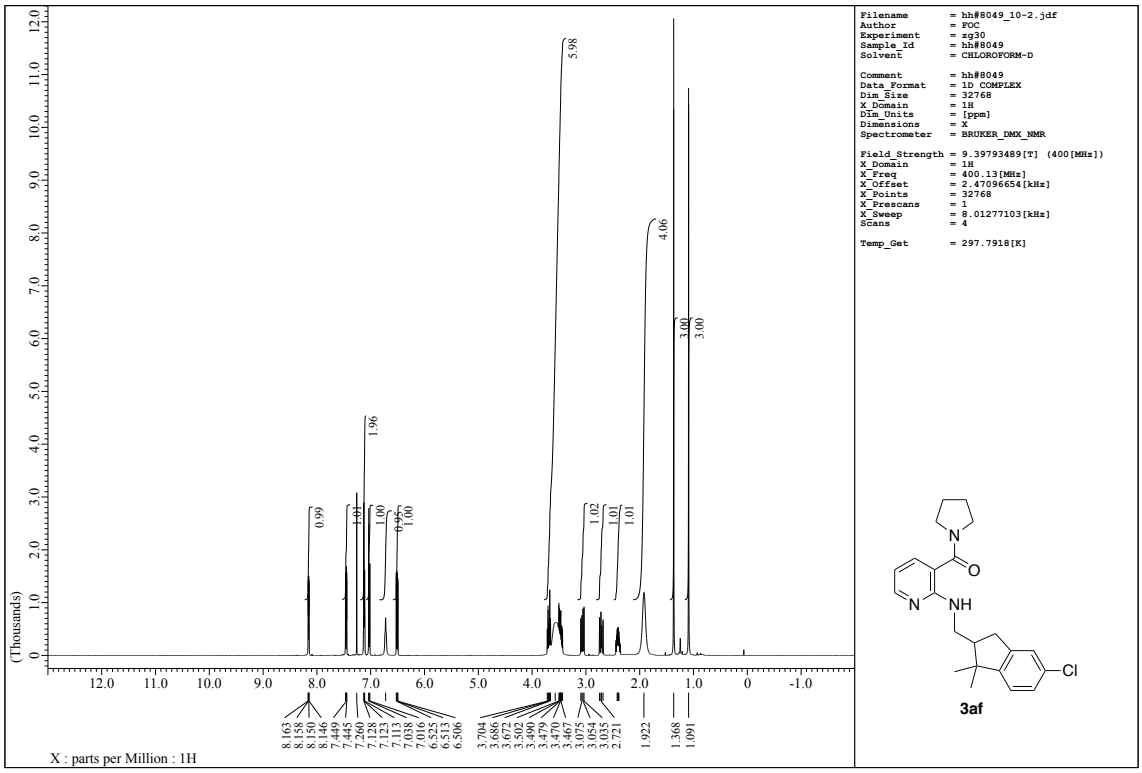

Filename = tk#3040-1_Carbon_copy1-1-4
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#3040-1
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 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 = 525
Total_Scans = 525
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21[dC]
X_90_Width = 10.86[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.62[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
3bc

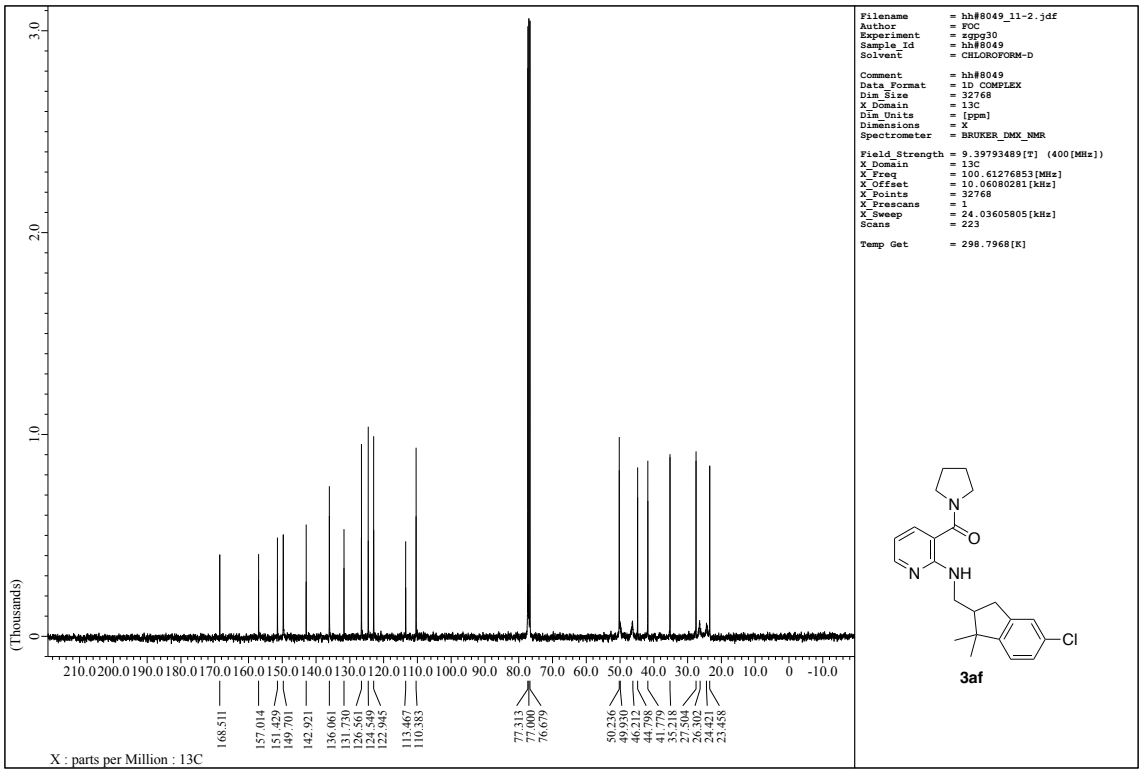

Filename = hh#7068_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#7068
Solvent = CHLOROFORM-D
Comment = hh#7068
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.6563[K]
(Thousands)
X : parts per Million : 1H
3ad

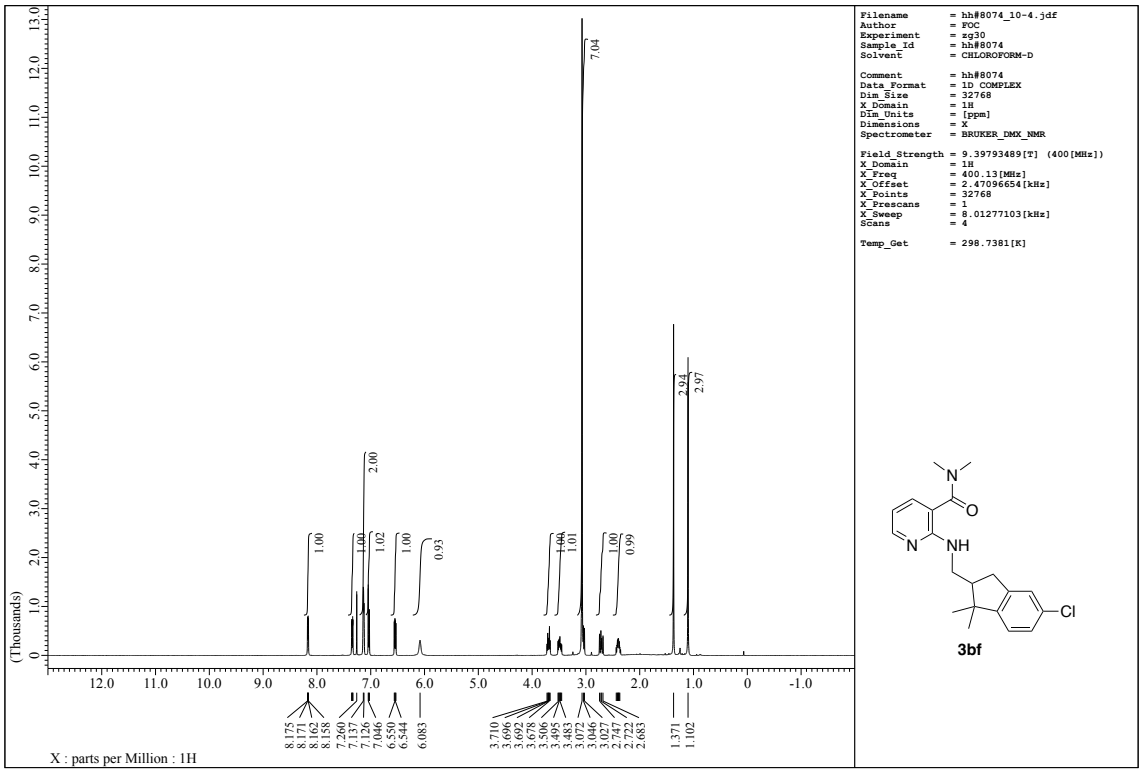

Filename = hh#7068_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#7068
Solvent = CHLOROFORM-D
Comment = hh#7068
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 223
Temp Get = 298.8118[K]
(Thousands)
X : parts per Million : 13C
3ad

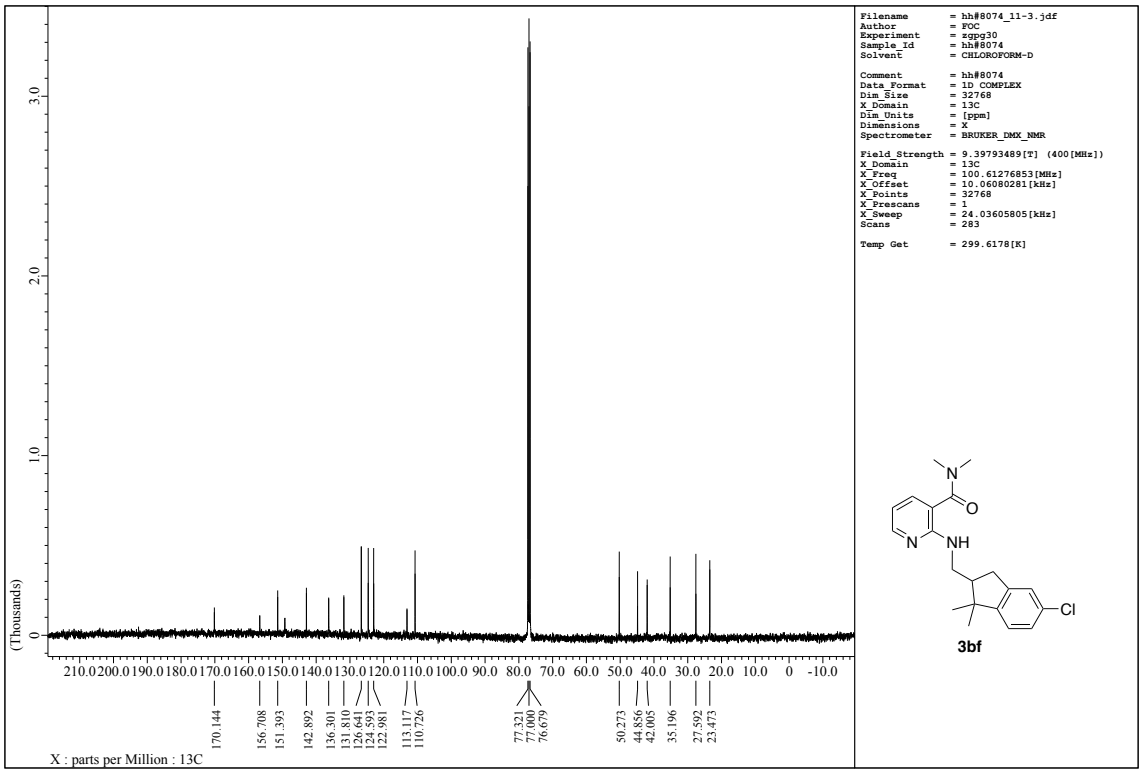

Filename = hh#8040_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8040
Solvent = CHLOROFORM-D
Comment = hh#8040
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.4301[K]
(Thousands)
X : parts per Million : 1H
3ae

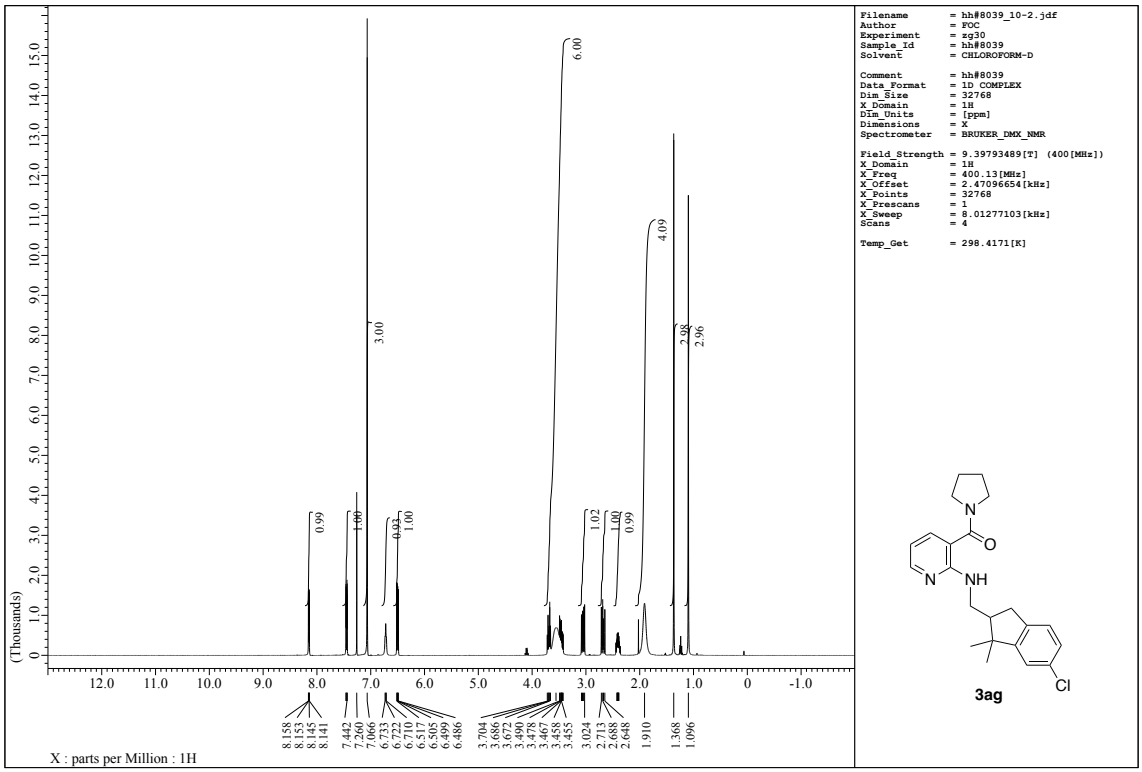

Filename = hh#8040_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8040
Solvent = CHLOROFORM-D
Comment = hh#8040
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 383
Temp Get = 299.5131[K]
(Thousands)
X : parts per Million : 13C
3ae

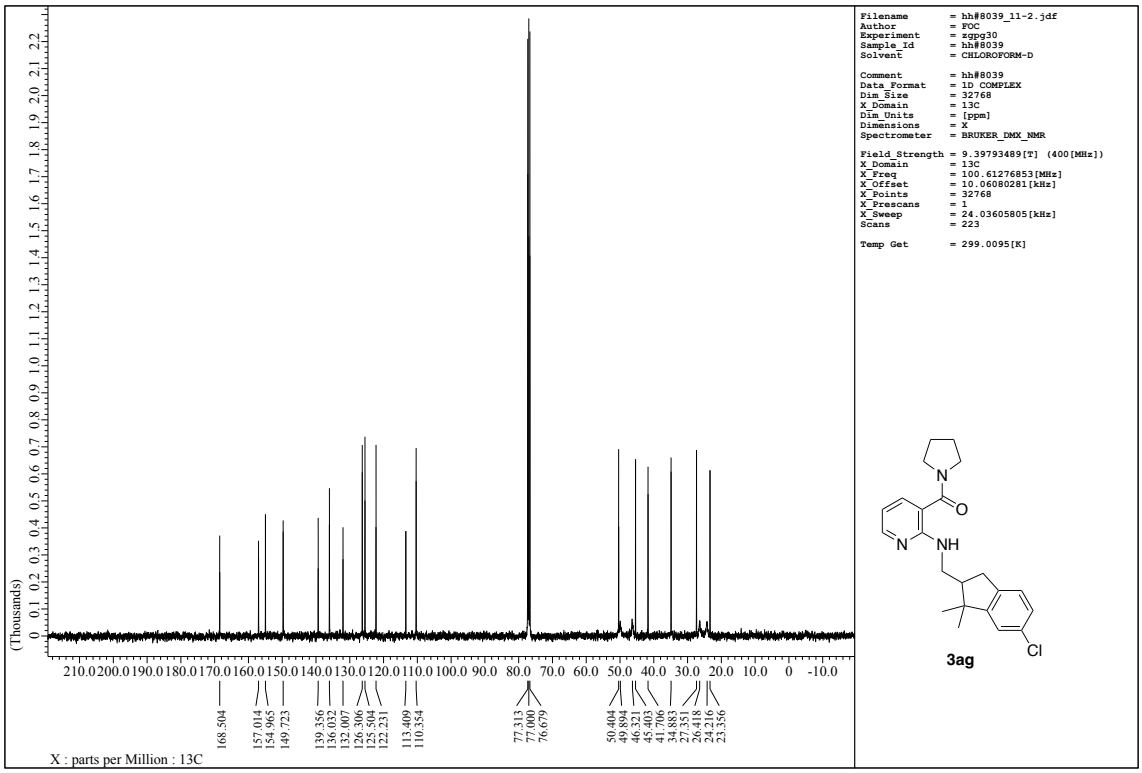

Filename = hh#8049_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8049
Solvent = CHLOROFORM-D
Comment = hh#8049
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.7918[K]
(Thousands)
X : parts per Million : 1H
3af

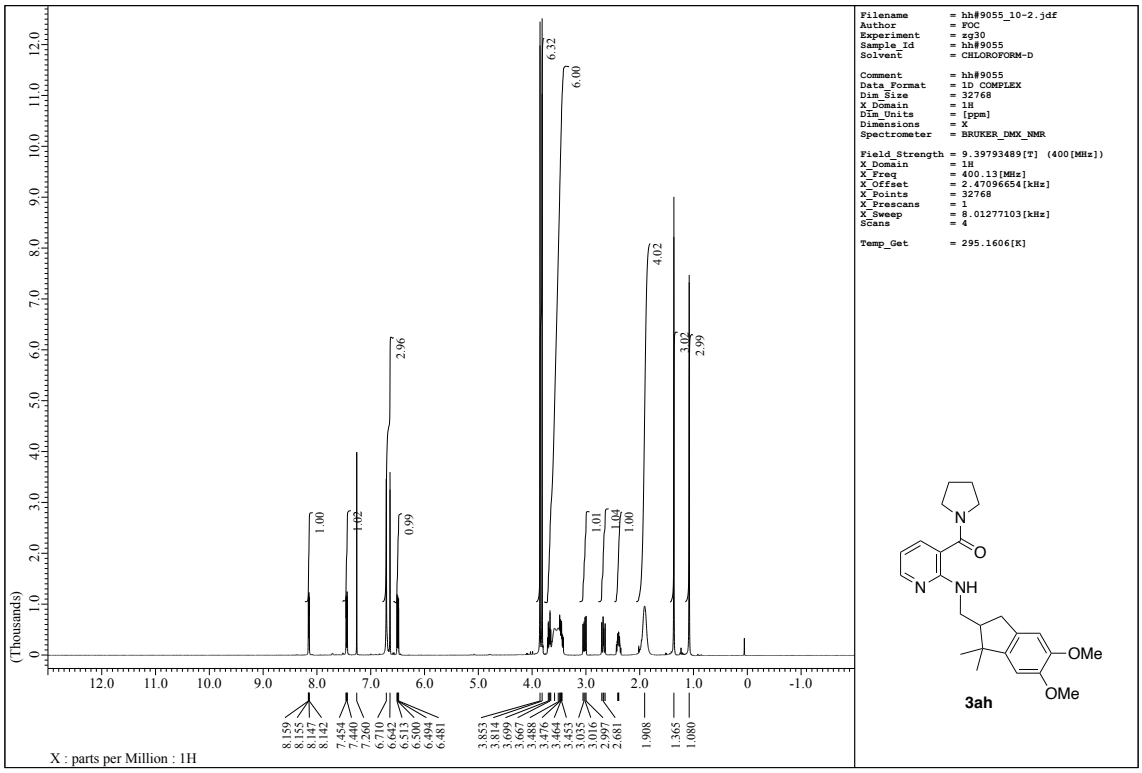

Filename = hh#8049_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8049
Solvent = CHLOROFORM-D
Comment = hh#8049
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 223
Temp_Get = 298.7968[K]
(Thousands)
X : parts per Million : 13C
3af

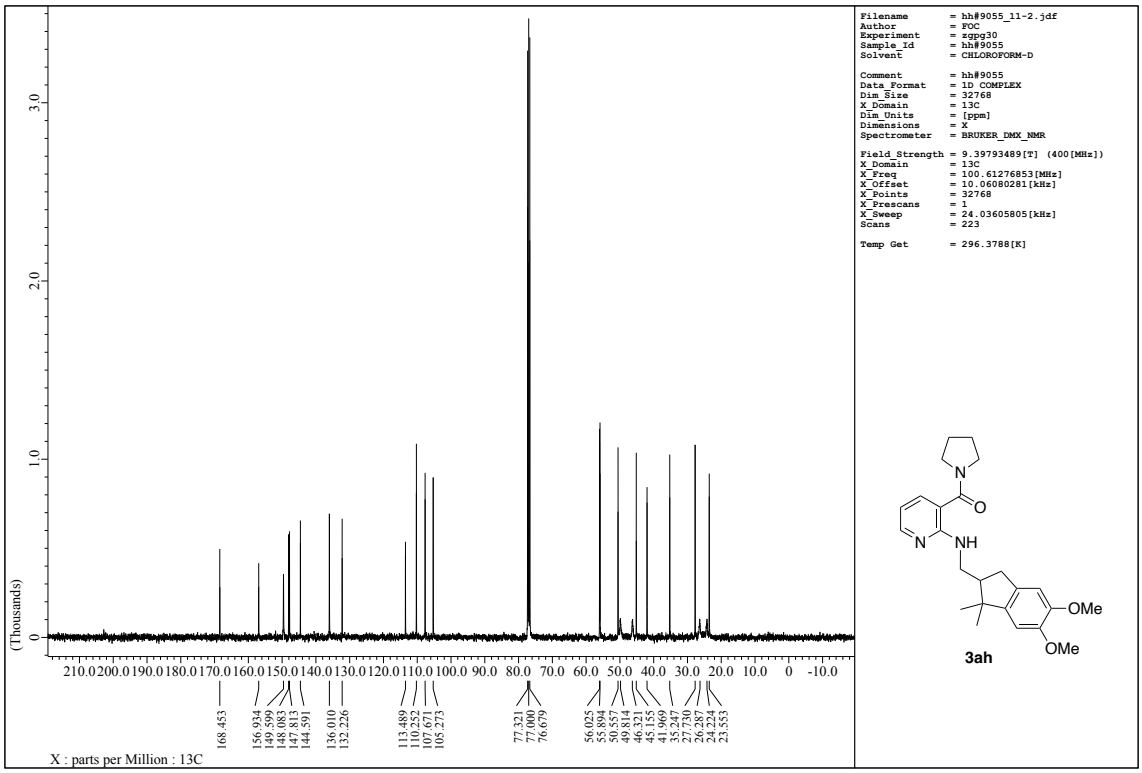

Filename = hh#8074_10-4.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8074
Solvent = CHLOROFORM-D
Comment = hh#8074
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.7381[K]
X : parts per Million : 1H
3bf

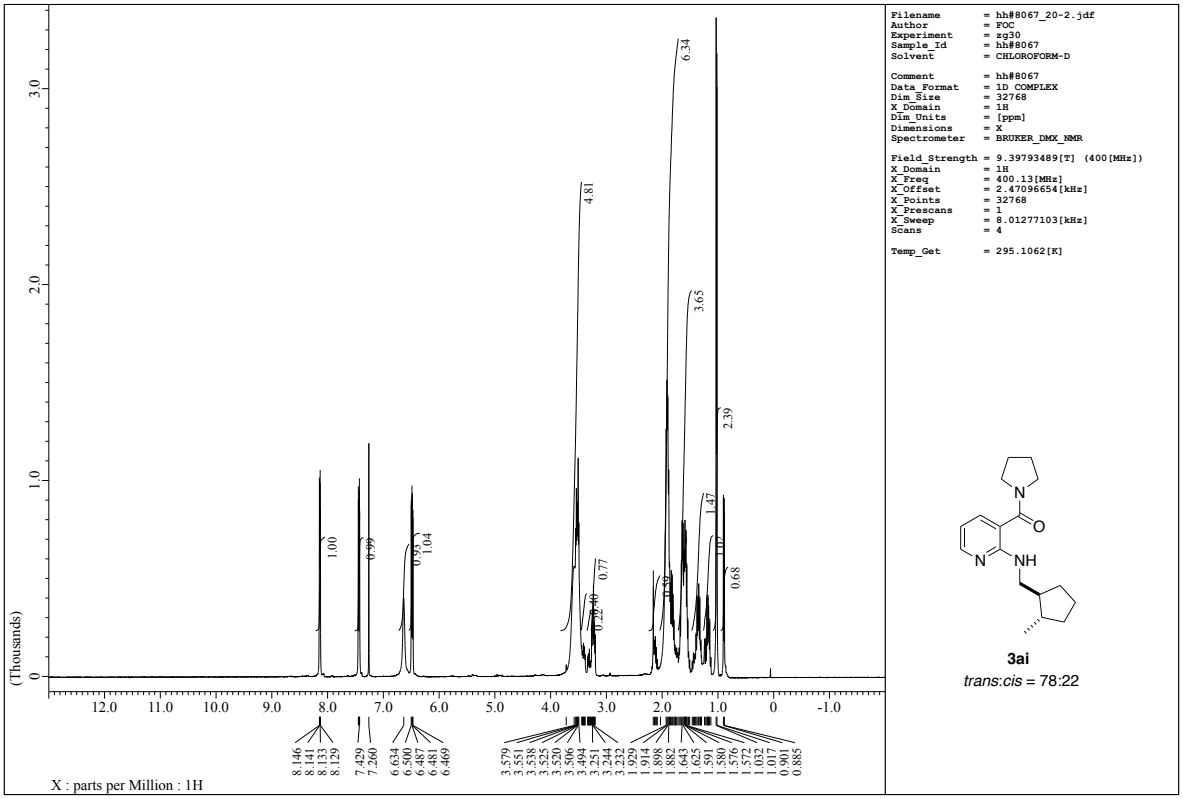

Filename = hh#8074_11-3.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8074
Solvent = CHLOROFORM-D
Comment = hh#8074
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 283
Temp Get = 299.6178[K]
X : parts per Million : 13C
3bf

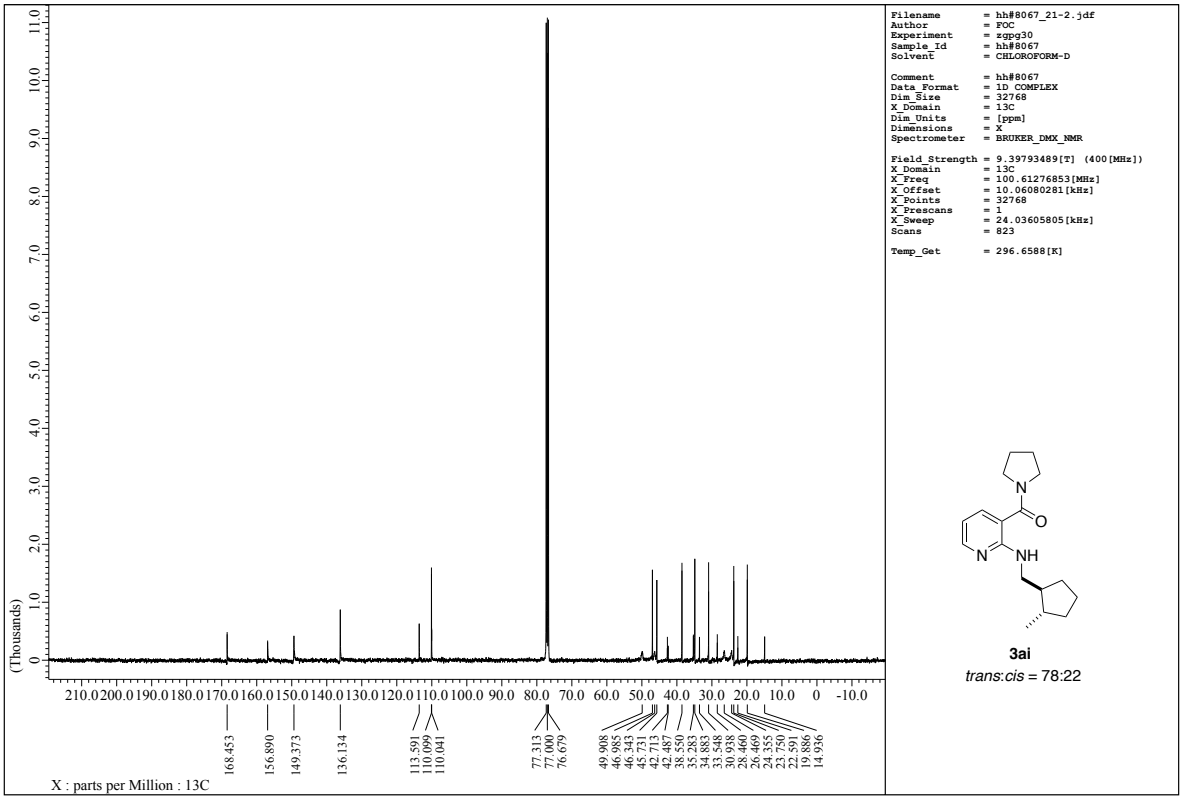

Filename = hh#8039_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8039
Solvent = CHLOROFORM-D
Comment = hh#8039
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 298.4171[K]
(Thousands)
X : parts per Million : 1H
3ag

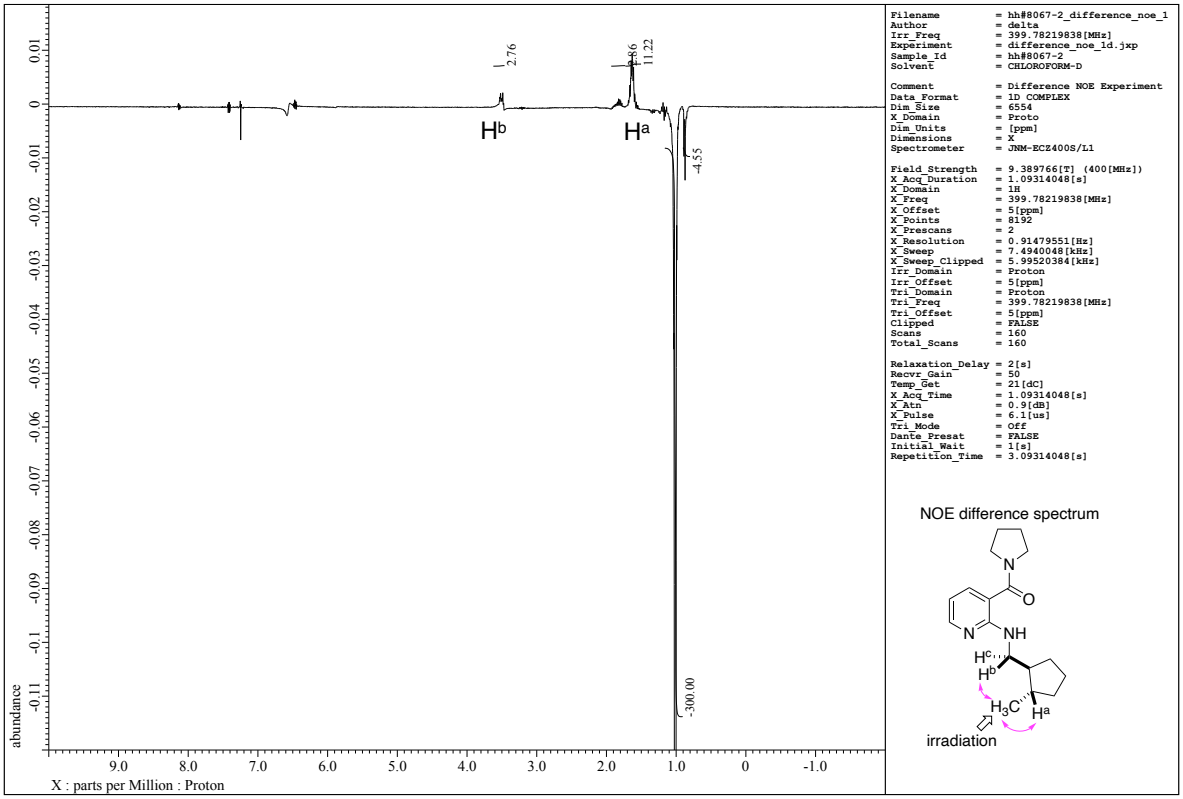

Filename = hh#8039_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8039
Solvent = CHLOROFORM-D
Comment = hh#8039
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 223
Temp Get = 299.0095[K]
(Thousands)
X : parts per Million : 13C
3ag

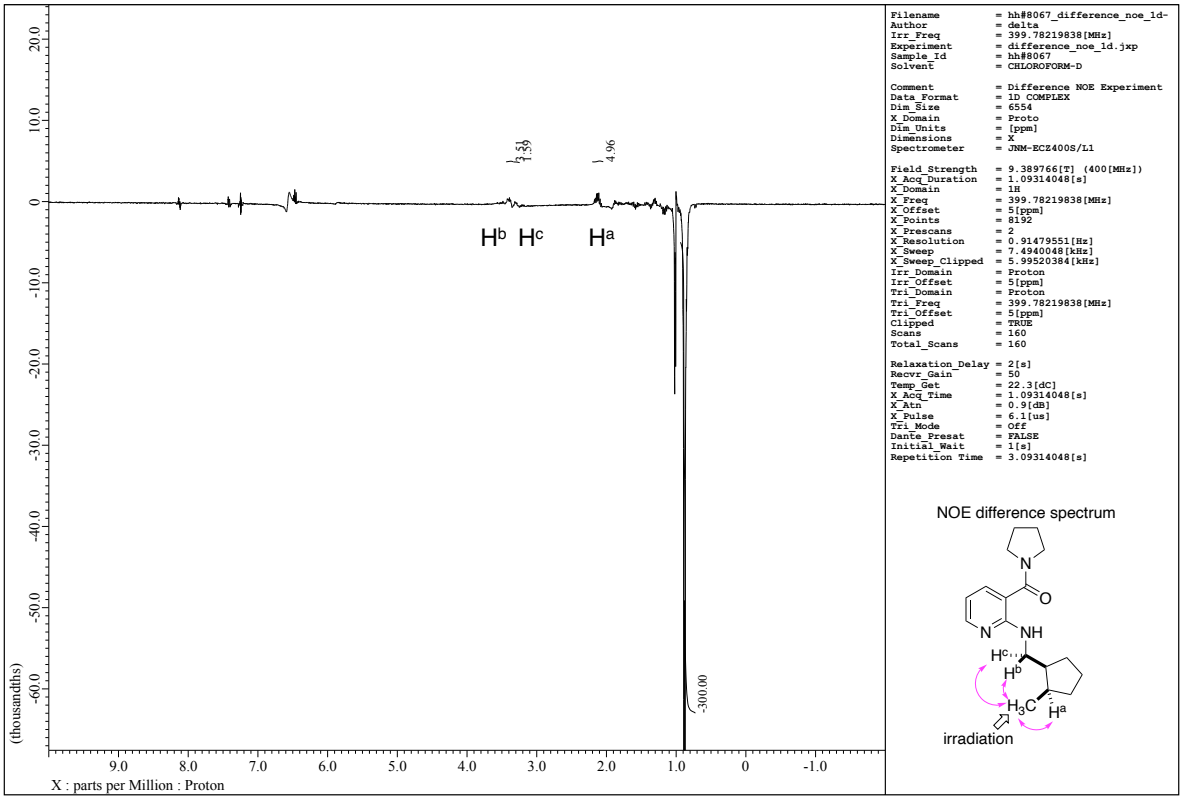

Filename = hh#9055_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9055
Solvent = CHLOROFORM-D
Comment = hh#9055
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 295.1606[K]
(Thousands)
X : parts per Million : 1H
3ah
OMe
OMe

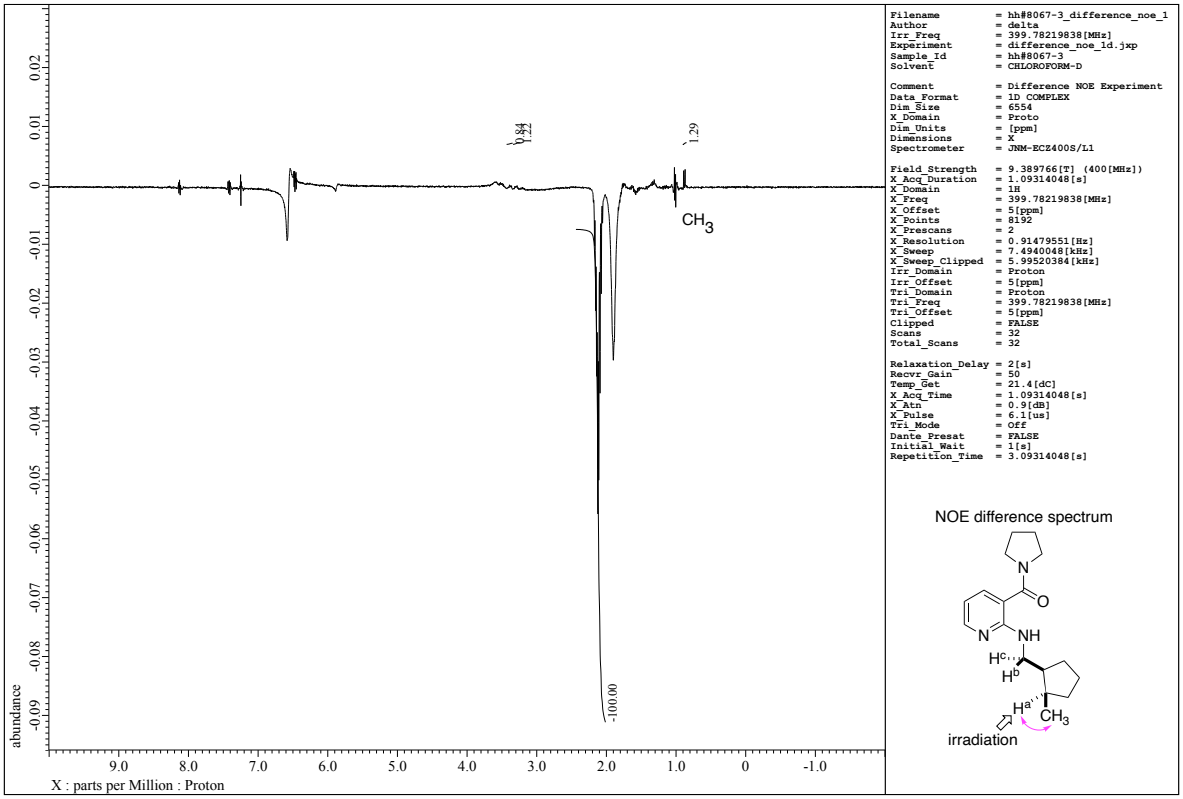

Filename = hh#9055_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9055
Solvent = CHLOROFORM-D
Comment = hh#9055
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 223
Temp Get = 296.3788[K]
(Thousands)
X : parts per Million : 13C
3ah
OMe
OMe

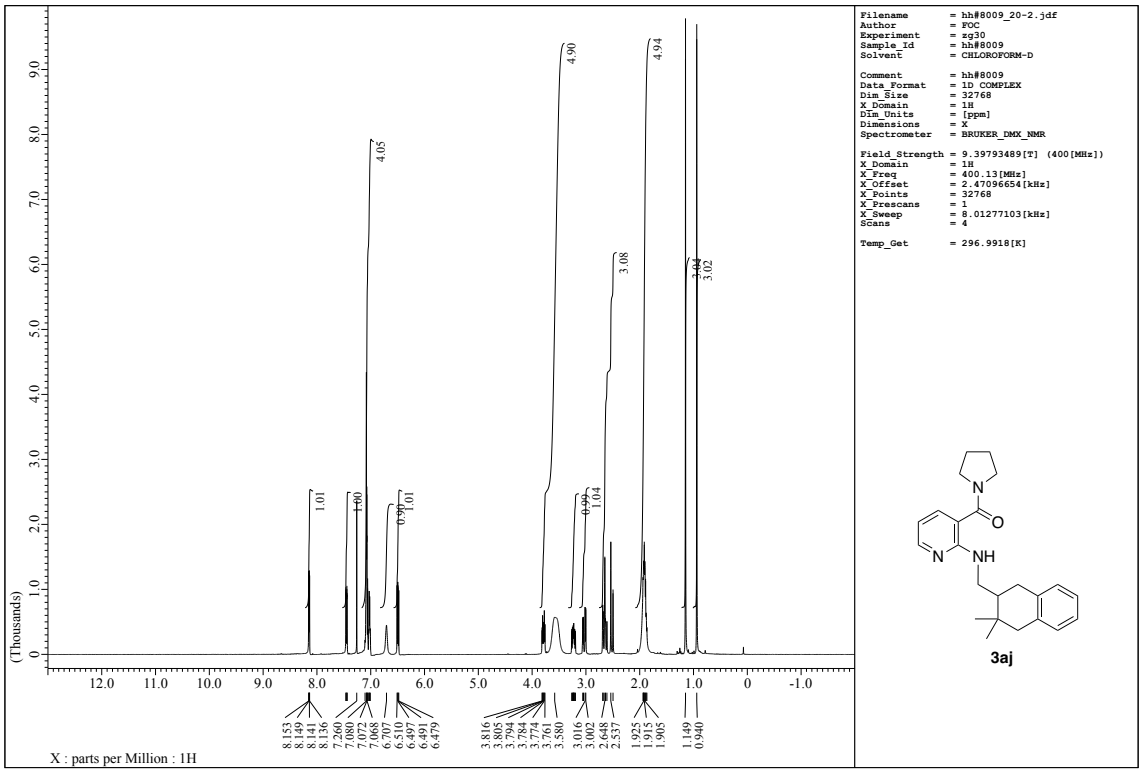

Filename = hh#8067_20-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8067
Solvent = CHLOROFORM-D
Comment = hh#8067
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 295.1062[K]
(Thousands)
X : parts per Million : 1H
3ai
trans:cis = 78:22

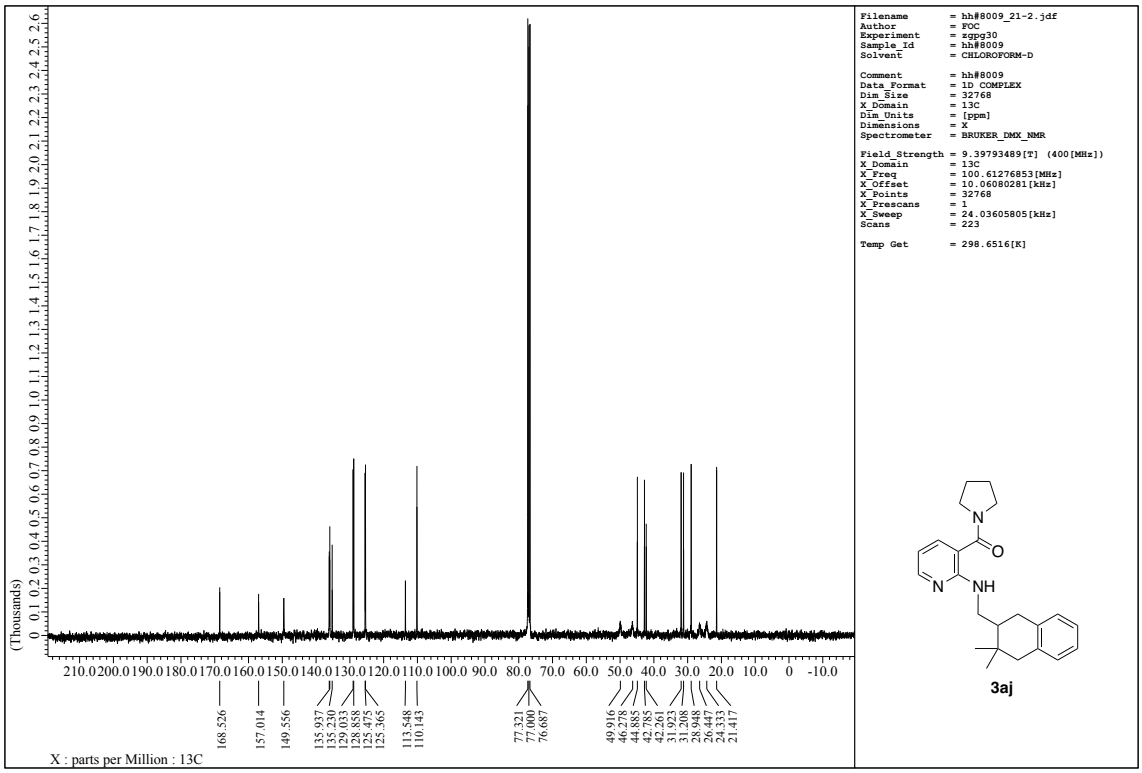

Filename = hh#8067_21-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8067
Solvent = CHLOROFORM-D
Comment = hh#8067
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 823
Temp_Get = 296.6588[K]
(Thousands)
X : parts per Million : 13C
3ai
trans:cis = 78:22

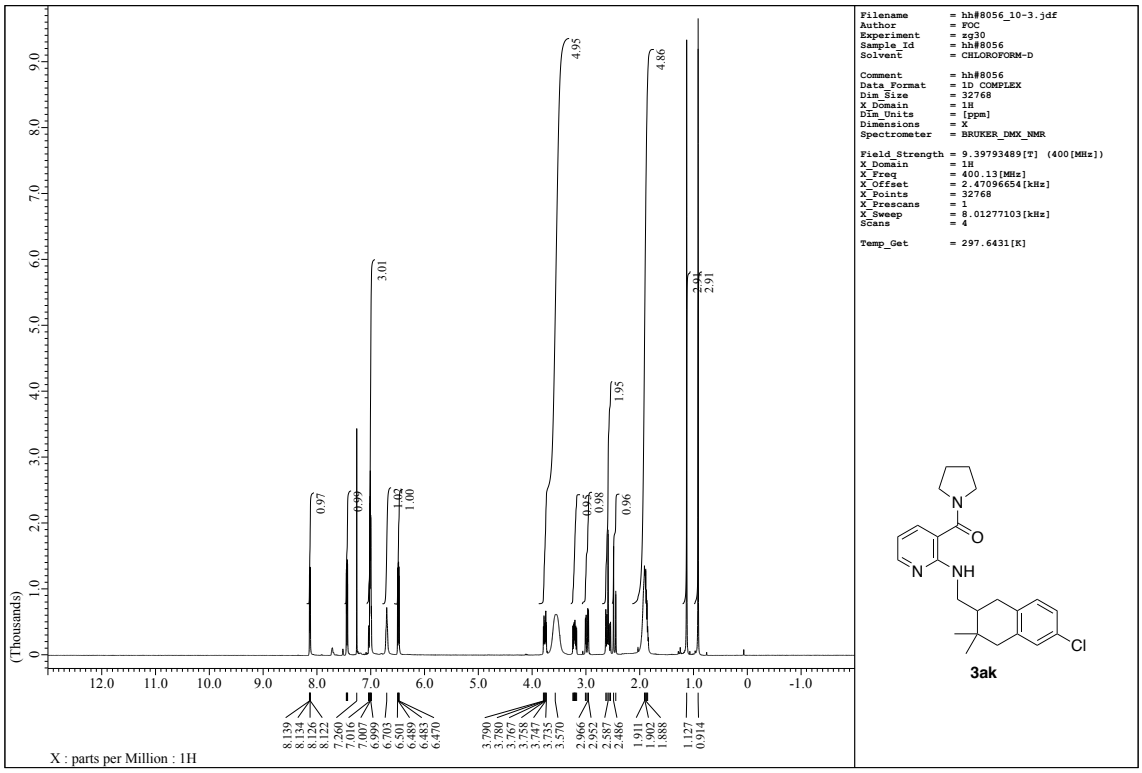

2.76
11.22
Hb
Ha
-4.55
-300.00
abundance
X : parts per Million : Proton
Filename = hh#8067-2_difference_noe_1
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = difference_noe_1d.jxp
Sample_Id = hh#8067-2
Solvent = CHLOROFORM-D
Comment = Difference NOE Experiment
Data_Format = 1D COMPLEX
Dim_Size = 6554
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.09314048[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 8192
X_Prescans = 2
X_Resolution = 0.91479551[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 = 160
Total_Scans = 160
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21[dC]
X_Acq_Time = 1.09314048[s]
X_Atn = 0.9[dB]
X_Pulse = 6.1[us]
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 3.09314048[s]
NOE difference spectrum
irradiation

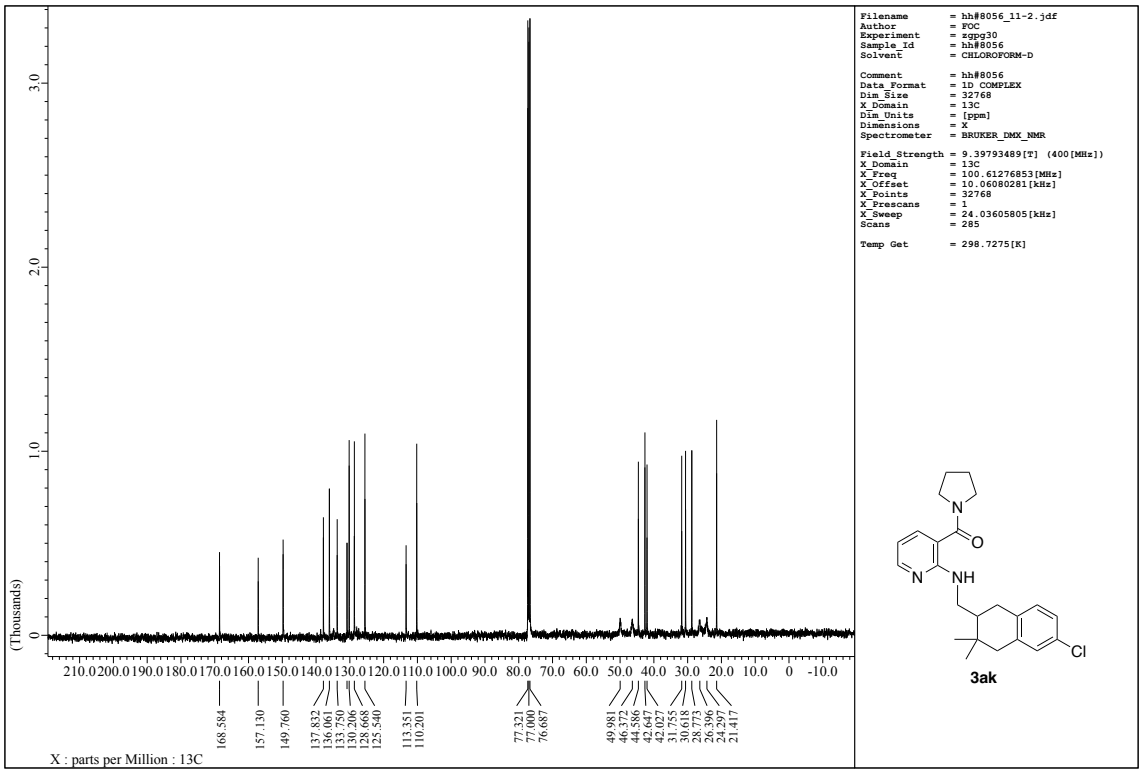

3.51
1.59
4.96
Hb Hc
Ha
-300.00
(thousandths)
X : parts per Million : Proton
Filename = hh#8067_difference_noe_1d-
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = difference_noe_1d.jxp
Sample_Id = hh#8067
Solvent = CHLOROFORM-D
Comment = Difference NOE Experiment
Data_Format = 1D COMPLEX
Dim_Size = 6554
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.09314048[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 8192
X_Prescans = 2
X_Resolution = 0.91479551[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 = TRUE
Scans = 160
Total_Scans = 160
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.3[dC]
X_Acq_Time = 1.09314048[s]
X_Atn = 0.9[dB]
X_Pulse = 6.1[us]
Tri_Mode = Off
DanTe_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 3.09314048[s]
NOE difference spectrum
irradiation

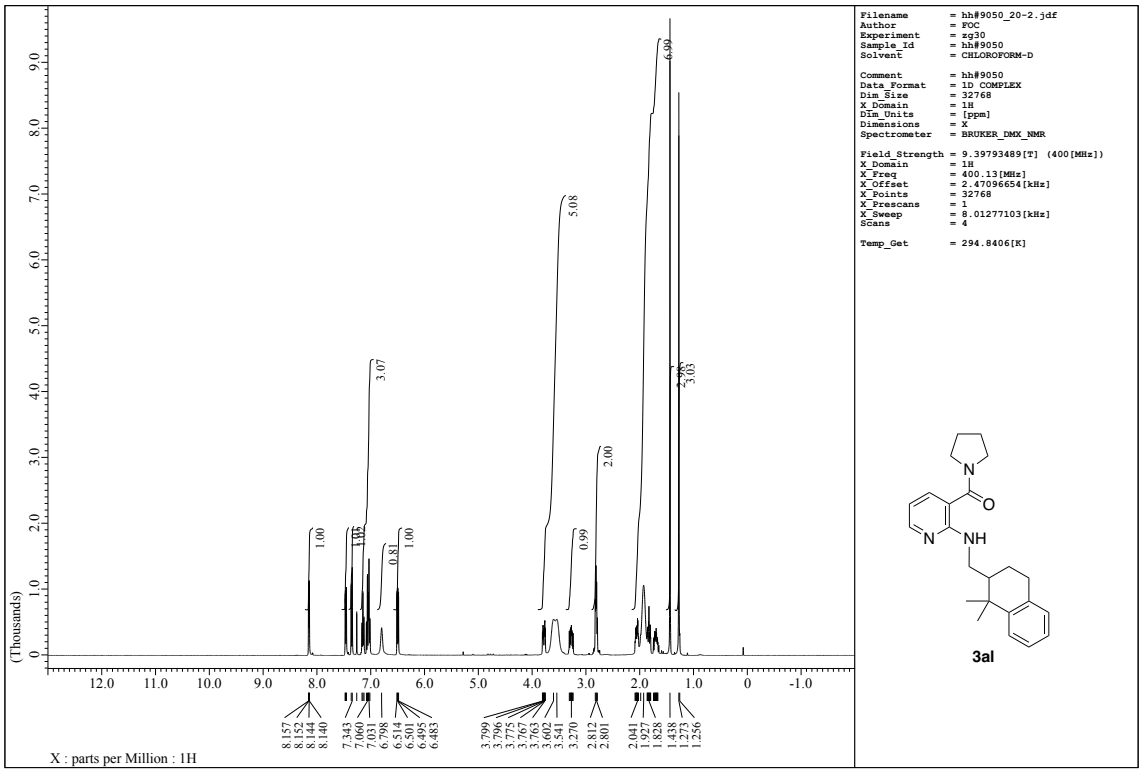

abundance
0.02
0.01
0
-0.01
-0.02
-0.03
-0.04
-0.05
-0.06
-0.07
-0.08
-0.09
1.22
1.29
CH3
-100.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
X : parts per Million : Proton
Filename = hh#8067-3_difference_noe_1
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = difference_noe_1d.jxp
Sample_Id = hh#8067-3
Solvent = CHLOROFORM-D
Comment = Difference NOE Experiment
Data_Format = 1D COMPLEX
Dim_Size = 6554
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.09314048[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 8192
X_Prescans = 2
X_Resolution = 0.91479551[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 = 32
Total_Scans = 32
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.4[dC]
X_Acq_Time = 1.09314048[s]
X_Atn = 0.9[dB]
X_Pulse = 6.1[us]
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 3.09314048[s]
NOE difference spectrum
N
O
N
NH
Hc
Hb
Ha
CH3
irradiation

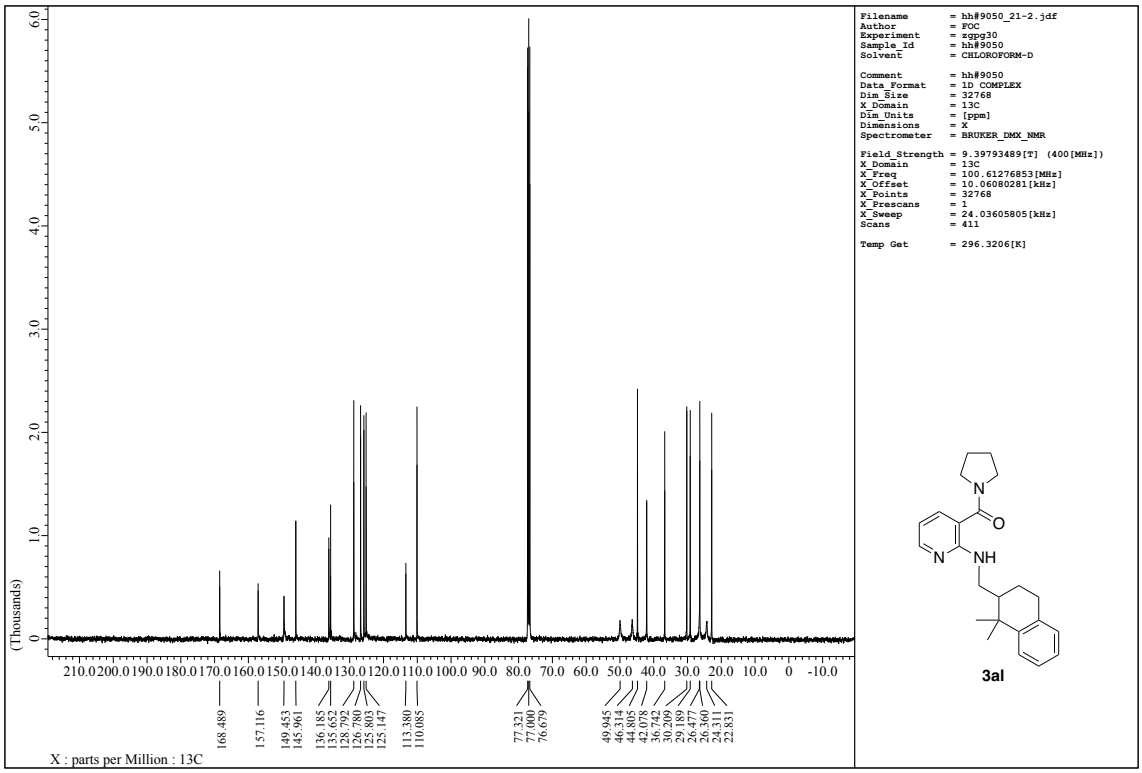

Filename = hh#8009_20-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8009
Solvent = CHLOROFORM-D
Comment = hh#8009
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 296.9918[K]
(Thousands)
X : parts per Million : 1H
3aj

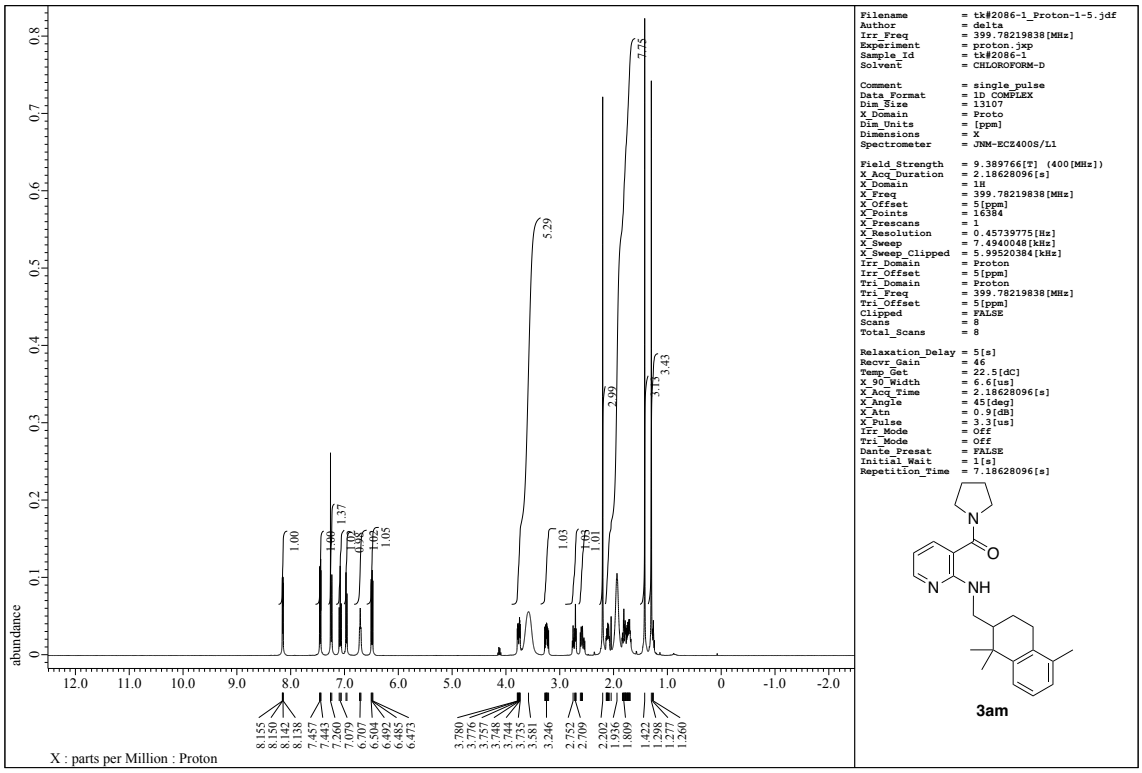

Filename = hh#8009_21-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8009
Solvent = CHLOROFORM-D
Comment = hh#8009
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 223
Temp Get = 298.6516[K]
(Thousands)
X : parts per Million : 13C
3aj

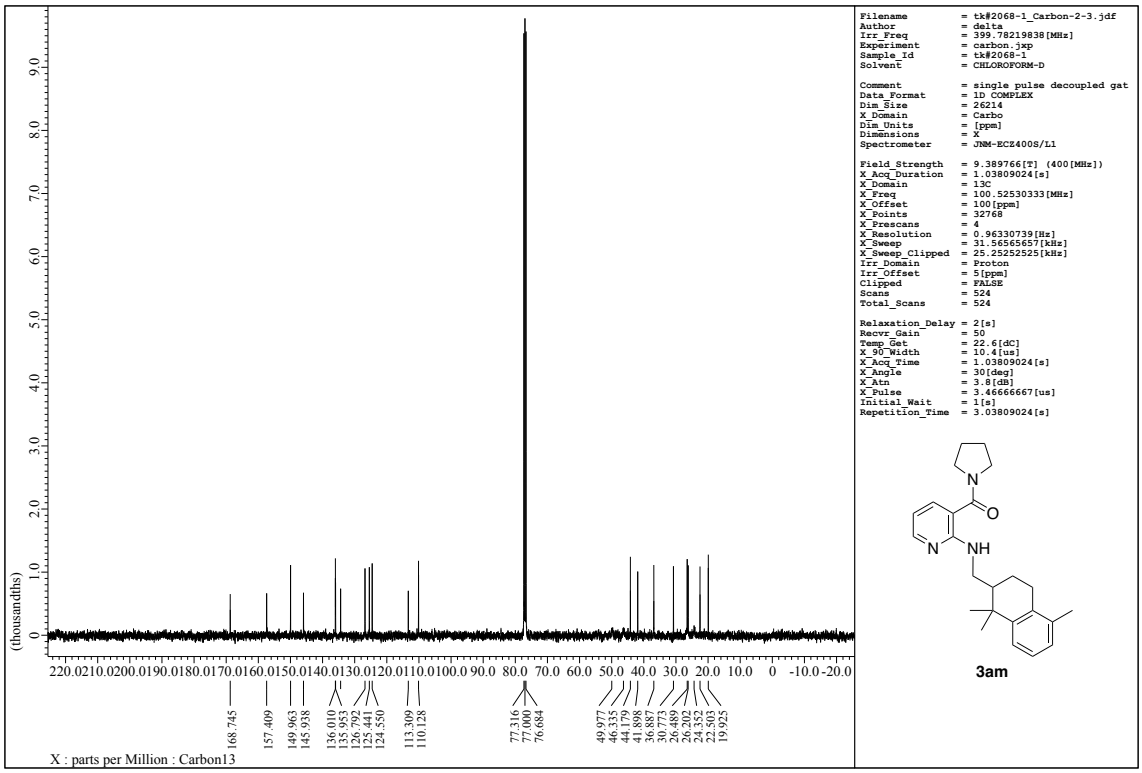

Filename = hh#8056_10-3.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8056
Solvent = CHLOROFORM-D
Comment = hh#8056
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.6431[K]
(Thousands)
X : parts per Million : 1H
3ak

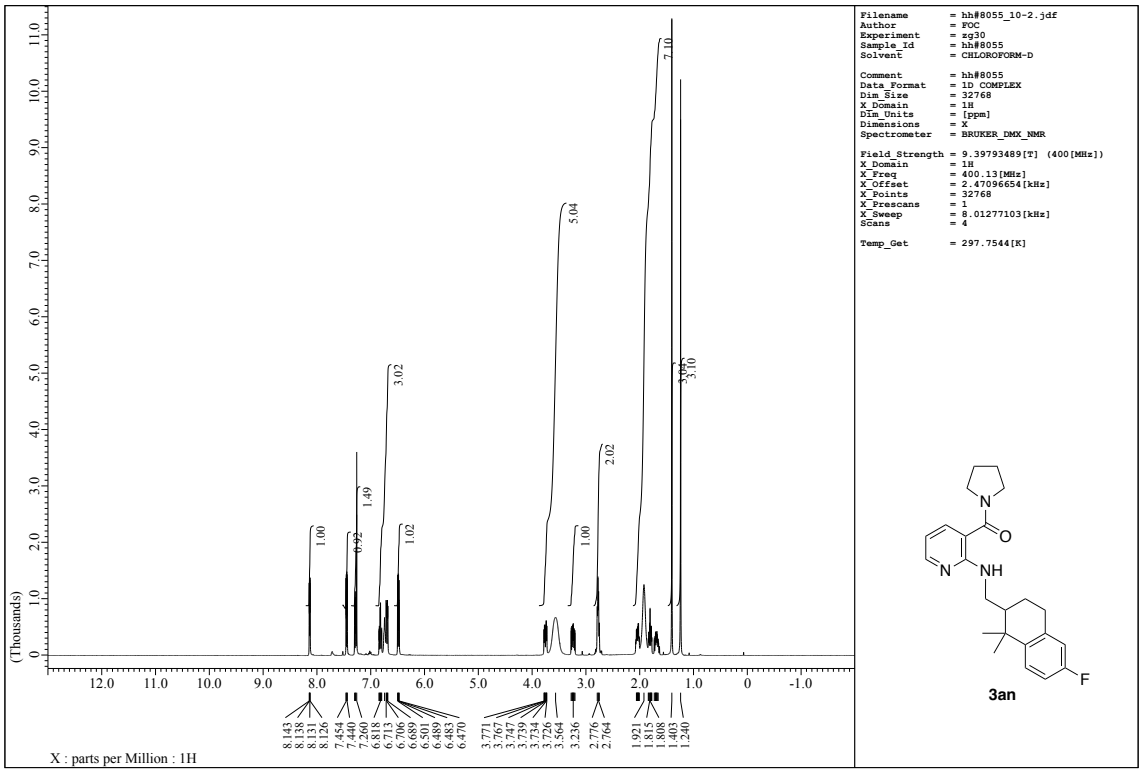

Filename = hh#8056_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8056
Solvent = CHLOROFORM-D
Comment = hh#8056
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 285
Temp Get = 298.7275[K]
(Thousands)
X : parts per Million : 13C
3ak

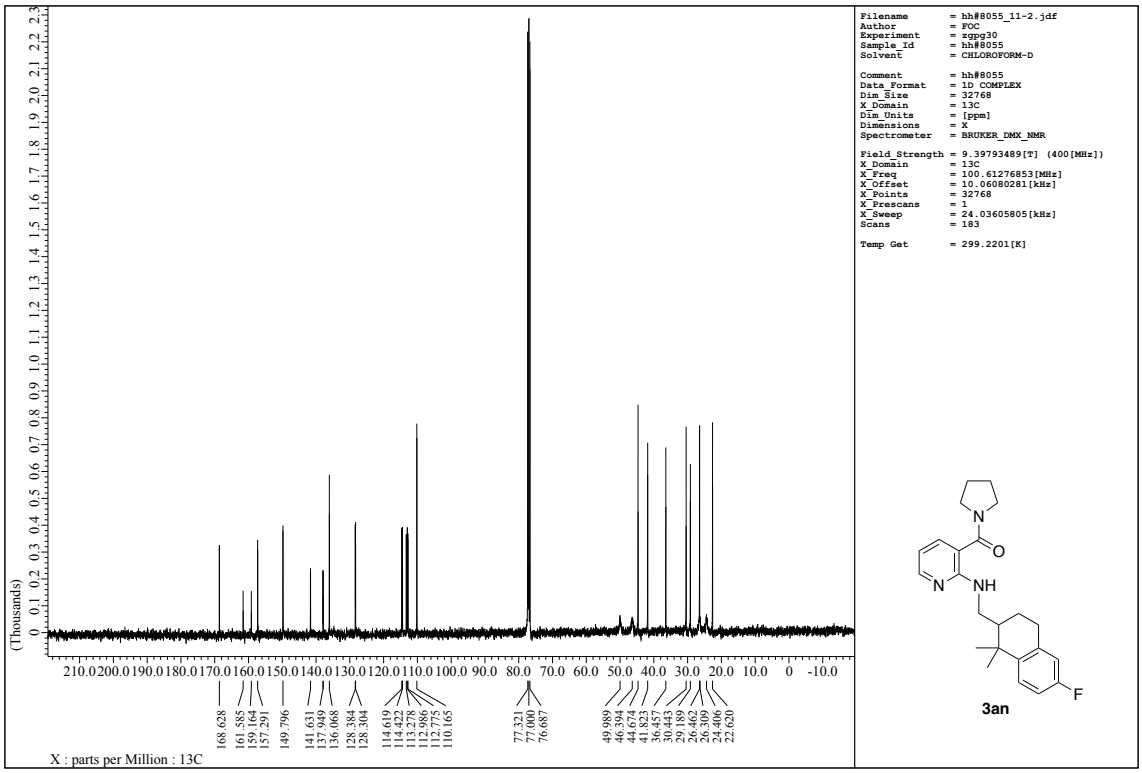

Filename = hh#9050_20-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9050
Solvent = CHLOROFORM-D
Comment = hh#9050
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 294.8406[K]
(Thousands)
X : parts per Million : 1H
3al

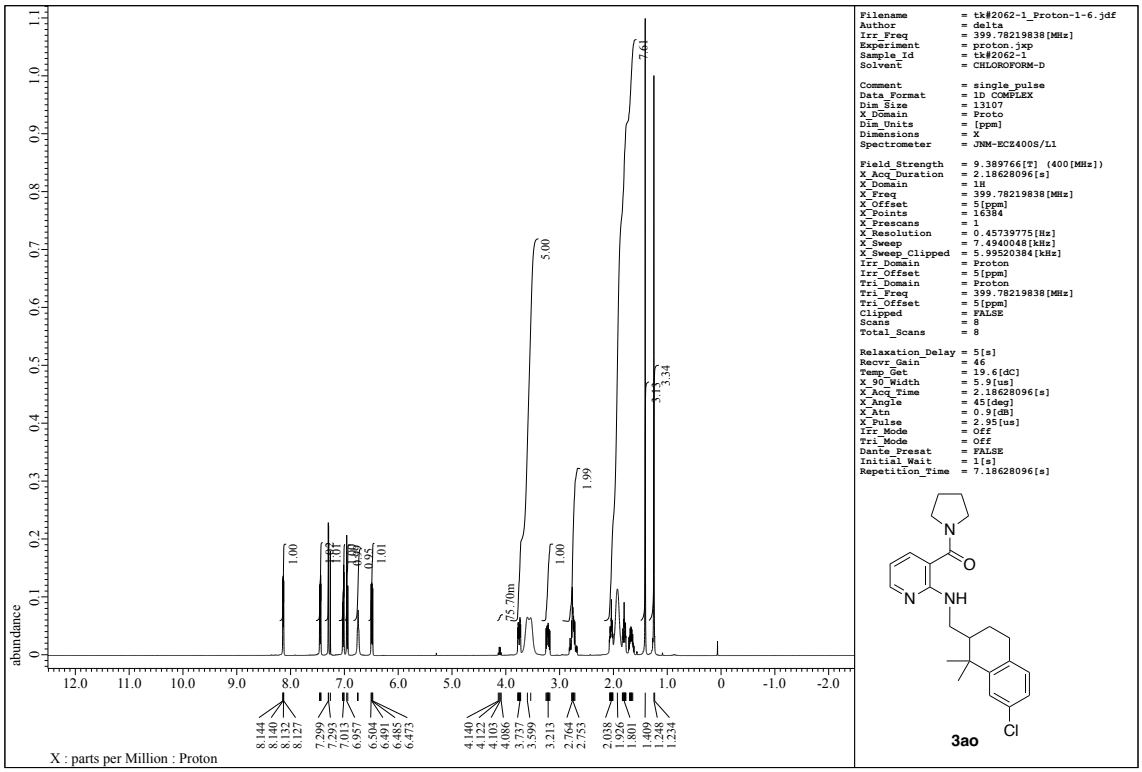

Filename = hh#9050_21-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9050
Solvent = CHLOROFORM-D
Comment = hh#9050
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 411
Temp Get = 296.3206[K]
(Thousands)
X : parts per Million : 13C
3al

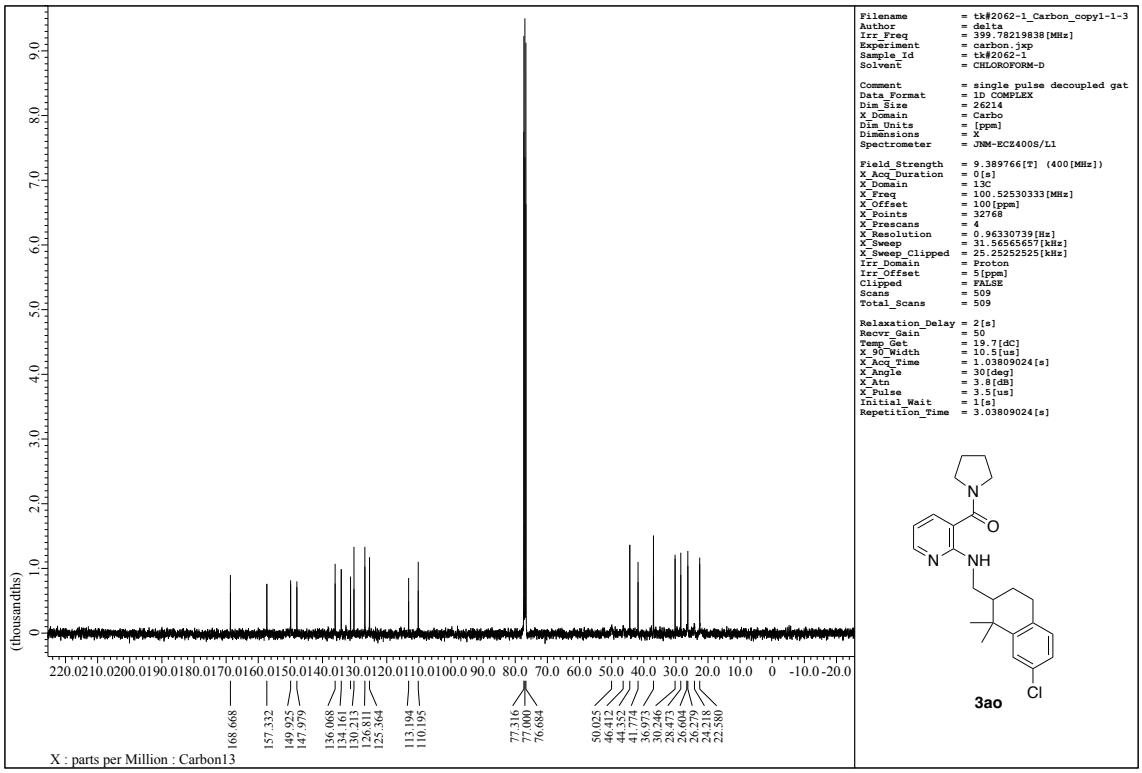

Filename = tk#2086-1_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experimnt = proton.jxp
Sample_Id = tk#2086-1
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 22.5[dC]
X_90_Width = 6.6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 3.3[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3am

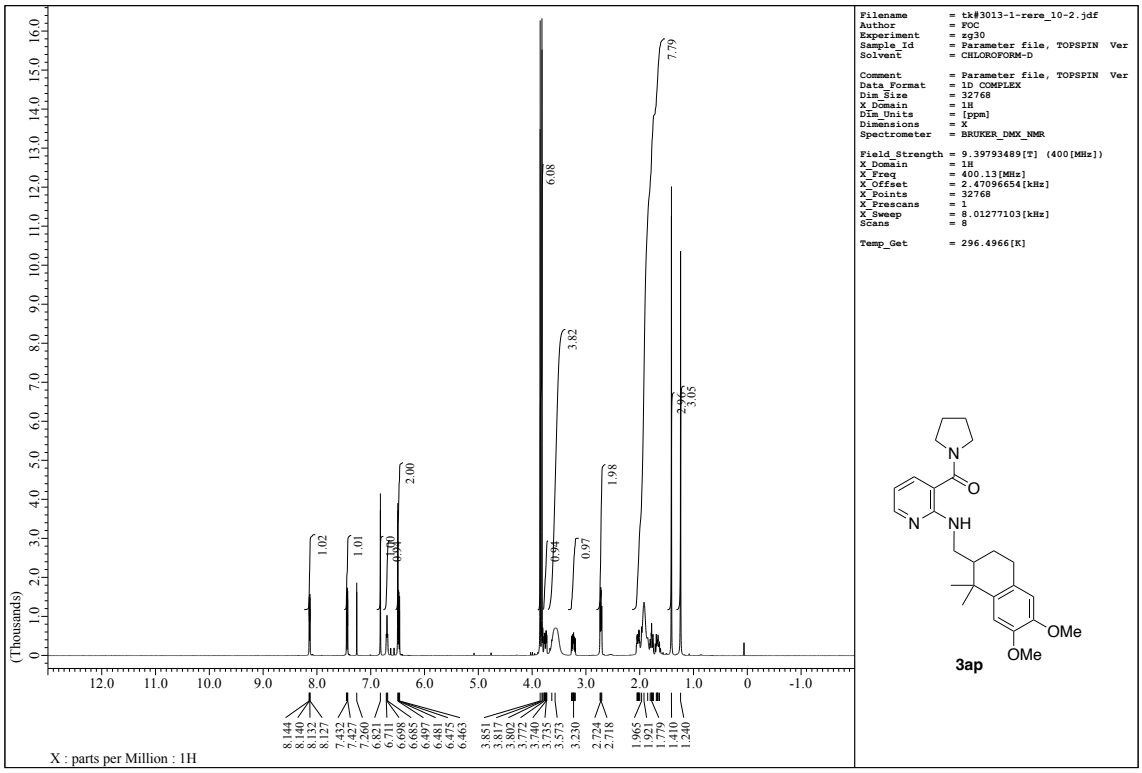

Filename = tk#2068-1_Carbon-2-3.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#2068-1
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
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 = 524
Total_Scans = 524
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.6[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]
(thousandths)
X : parts per Million : Carbon13
3am

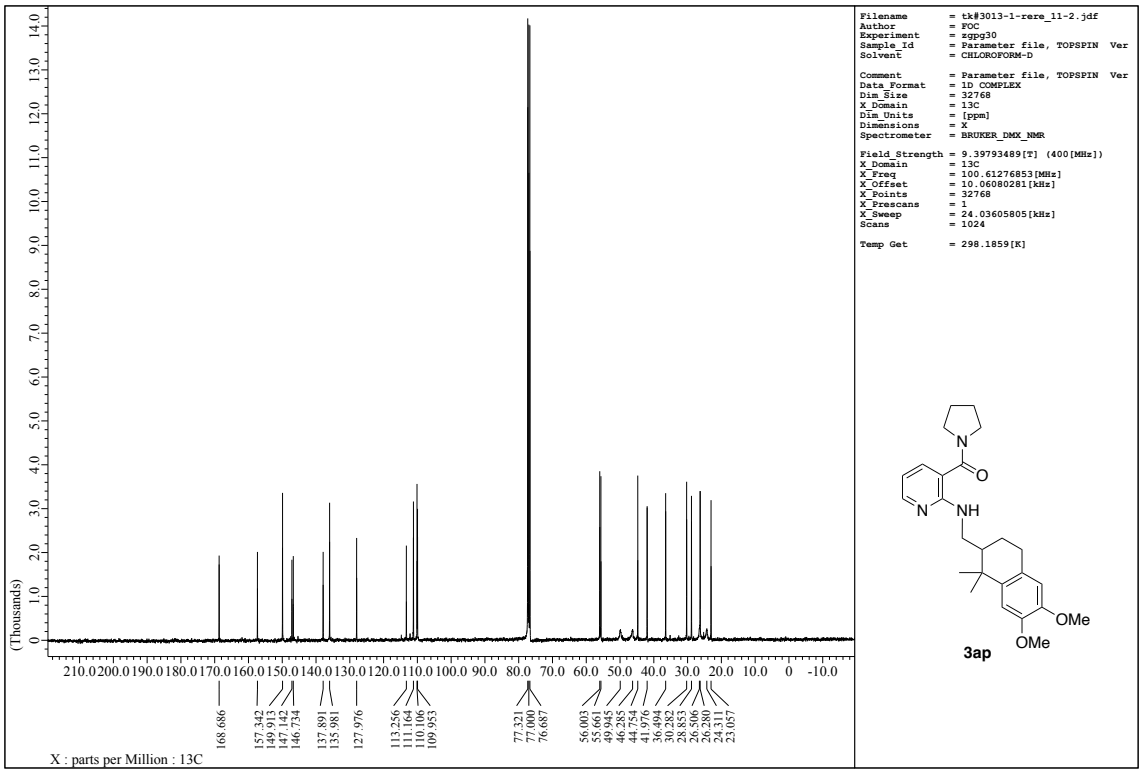

Filename = hh#8055_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#8055
Solvent = CHLOROFORM-D
Comment = hh#8055
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.7544[K]
(Thousands)
X : parts per Million : 1H
3an

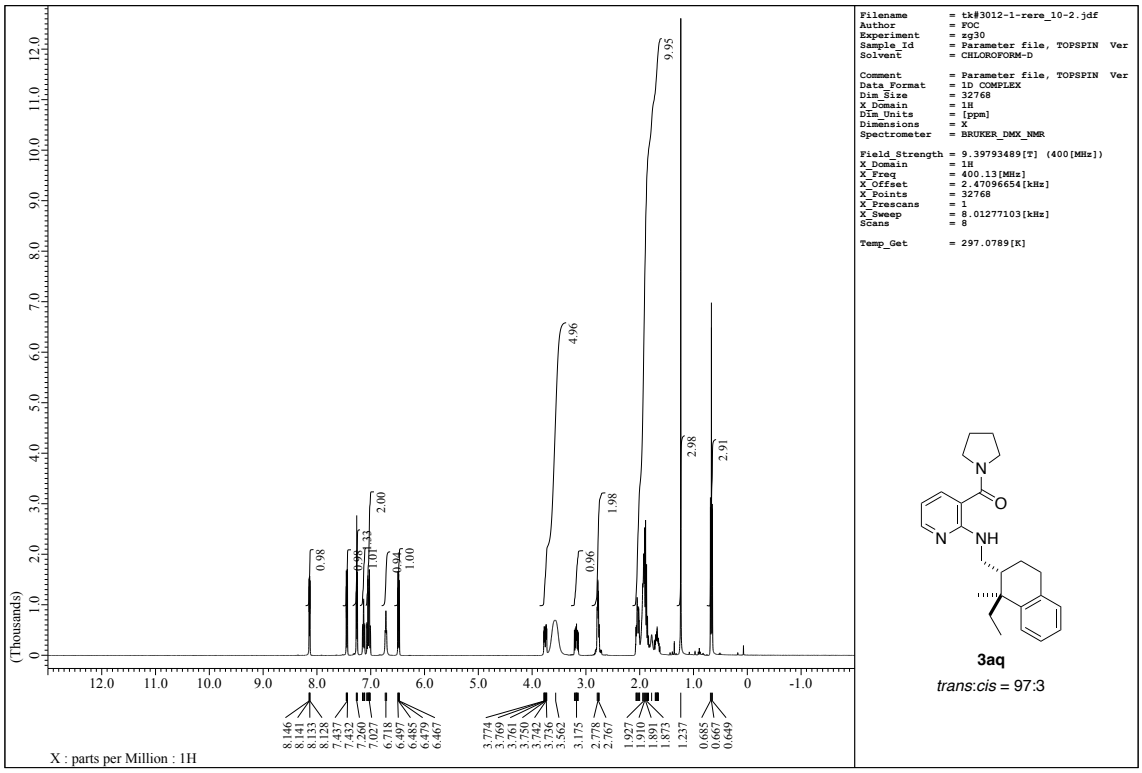

Filename = hh#8055_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#8055
Solvent = CHLOROFORM-D
Comment = hh#8055
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 183
Temp Get = 299.2201[K]
(Thousands)
X : parts per Million : 13C
3an

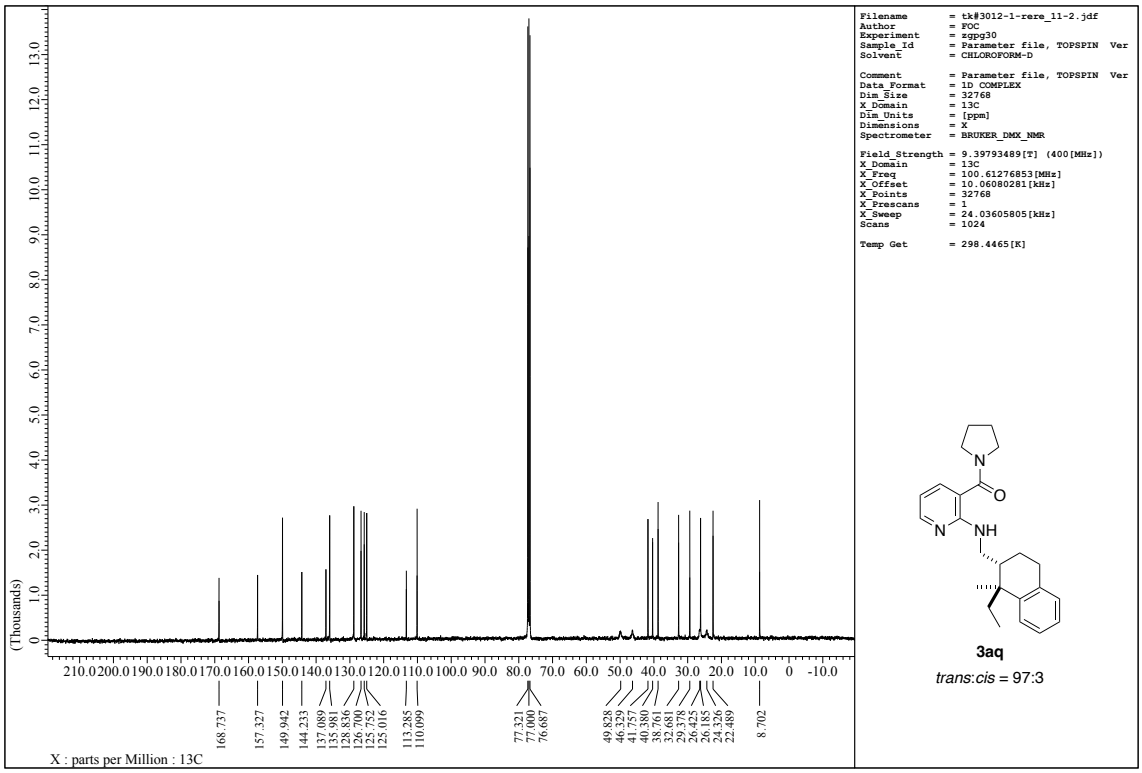

Filename = tk#2062-1_Proton-1-6.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experimnt = proton.jxp
Sample_Id = tk#2062-1
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 19.6[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.91[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3ao

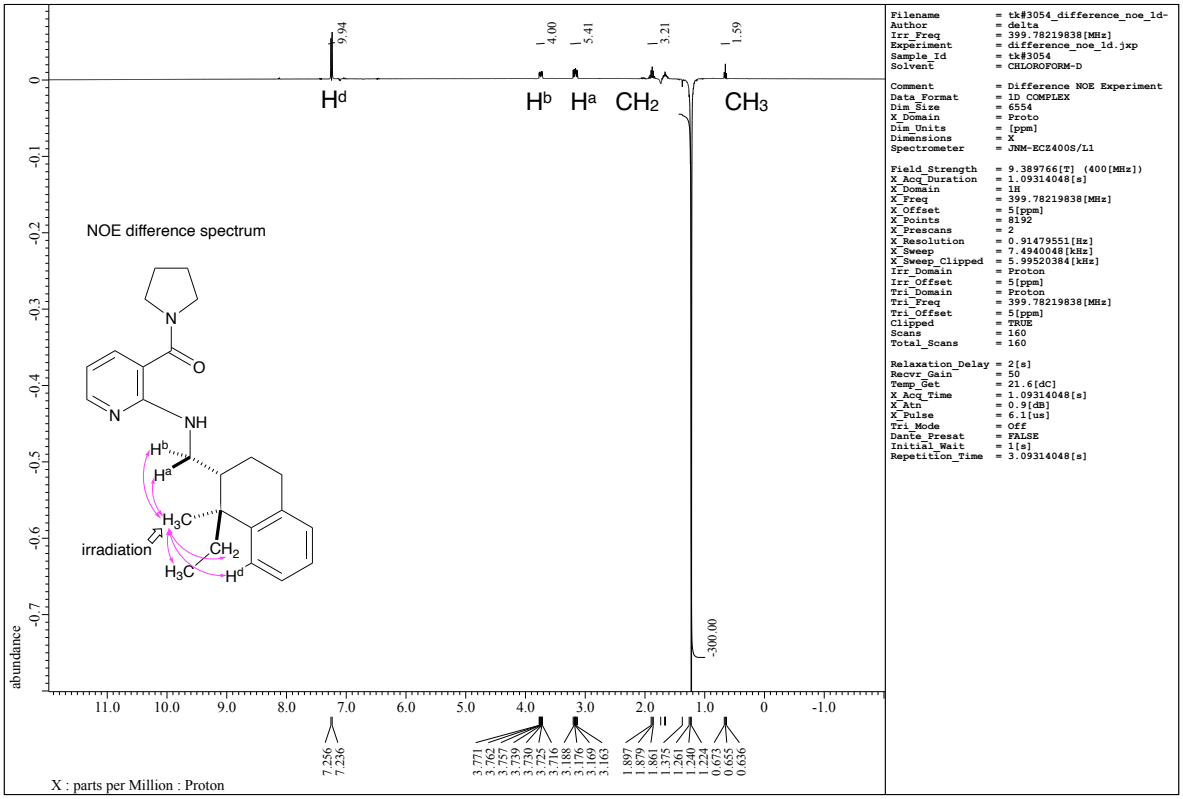

Filename = tk#2062-1_Carbon_copy1-1-3
Author = delta
Irr_Freq = 399.78219838[MHz]
Experimnt = carbon.jxp
Sample_Id = tk#2062-1
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 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 = 509
Total_Scans = 509
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.7[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
3ao

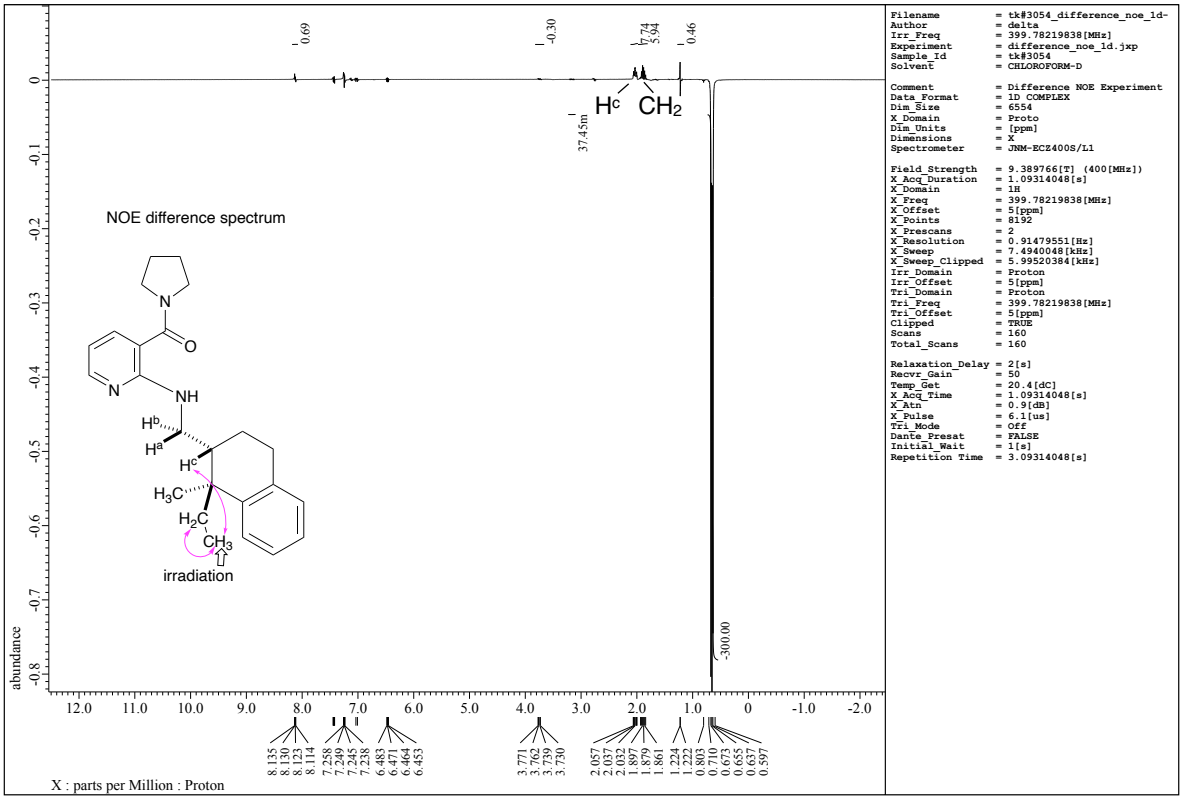

Filename = tk#3013-1-rere_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 8
Temp_Get = 296.4966[K]
(Thousands)
X : parts per Million : 1H
OMe
OMe
3ap

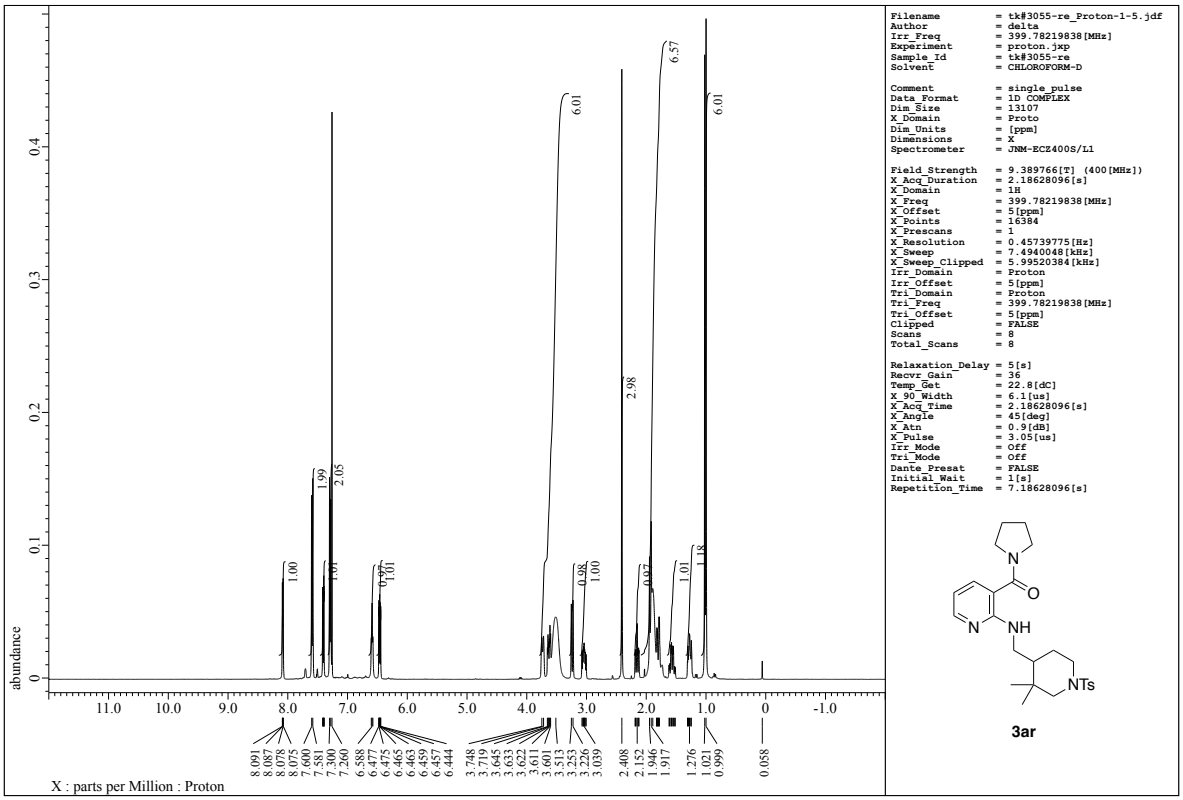

Filename = tk#3013-1-rere_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 1024
Temp Get = 298.1859[K]
(Thousands)
X : parts per Million : 13C
OMe
OMe
3ap

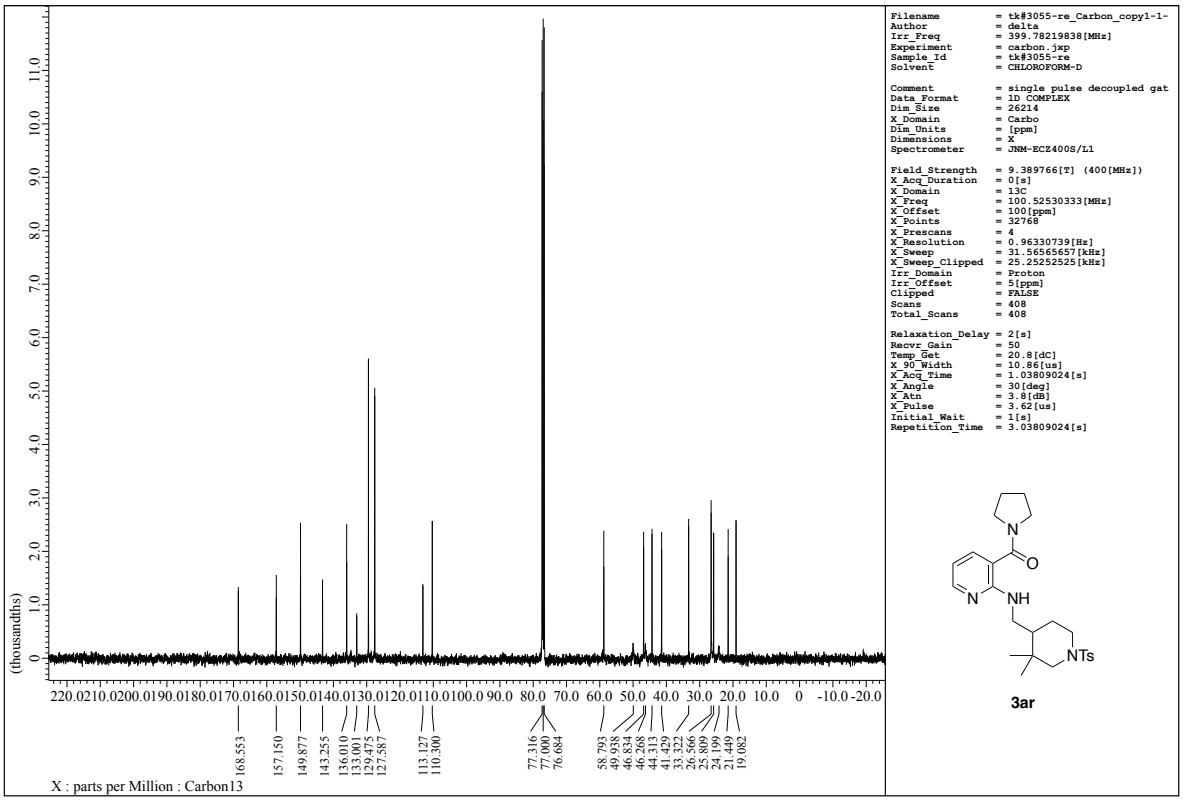

Filename = tk#3012-1-rere_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 8
Temp_Get = 297.0789[K]
(Thousands)
X : parts per Million : 1H
3aq
trans:cis = 97:3

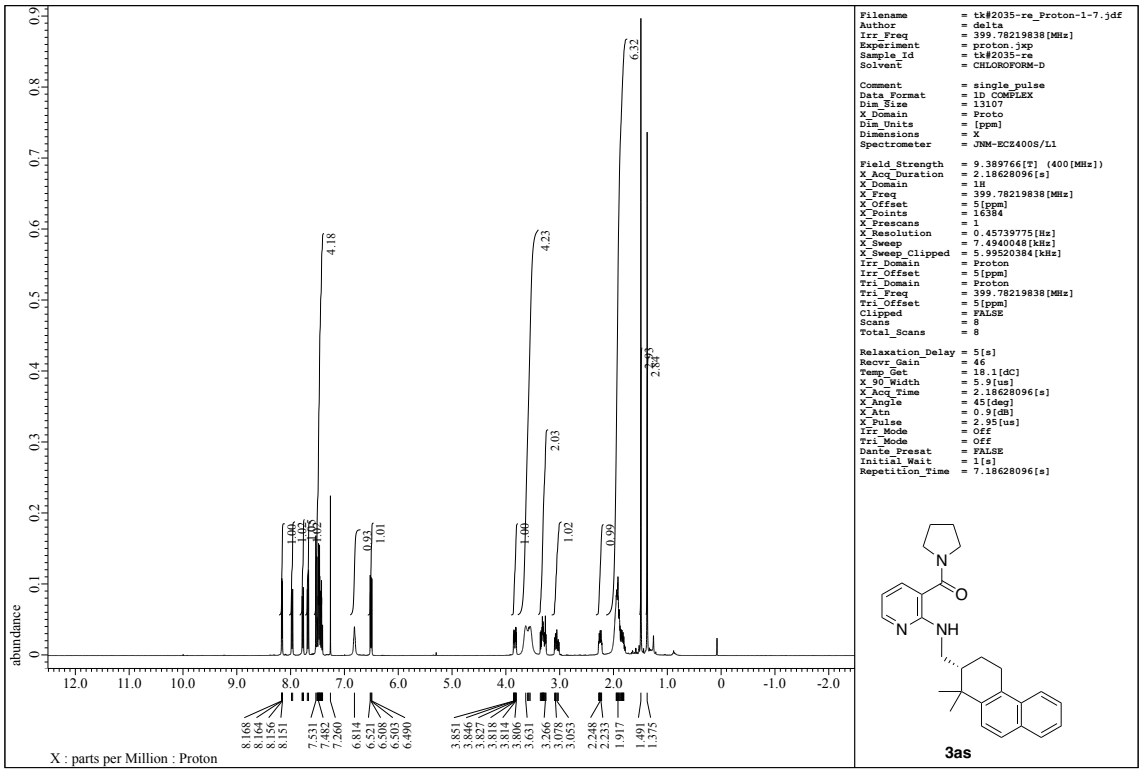

Filename = tk#3012-1-rere_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 1024
Temp Get = 298.4465[K]
(Thousands)
X : parts per Million : 13C
3aq
trans:cis = 97:3

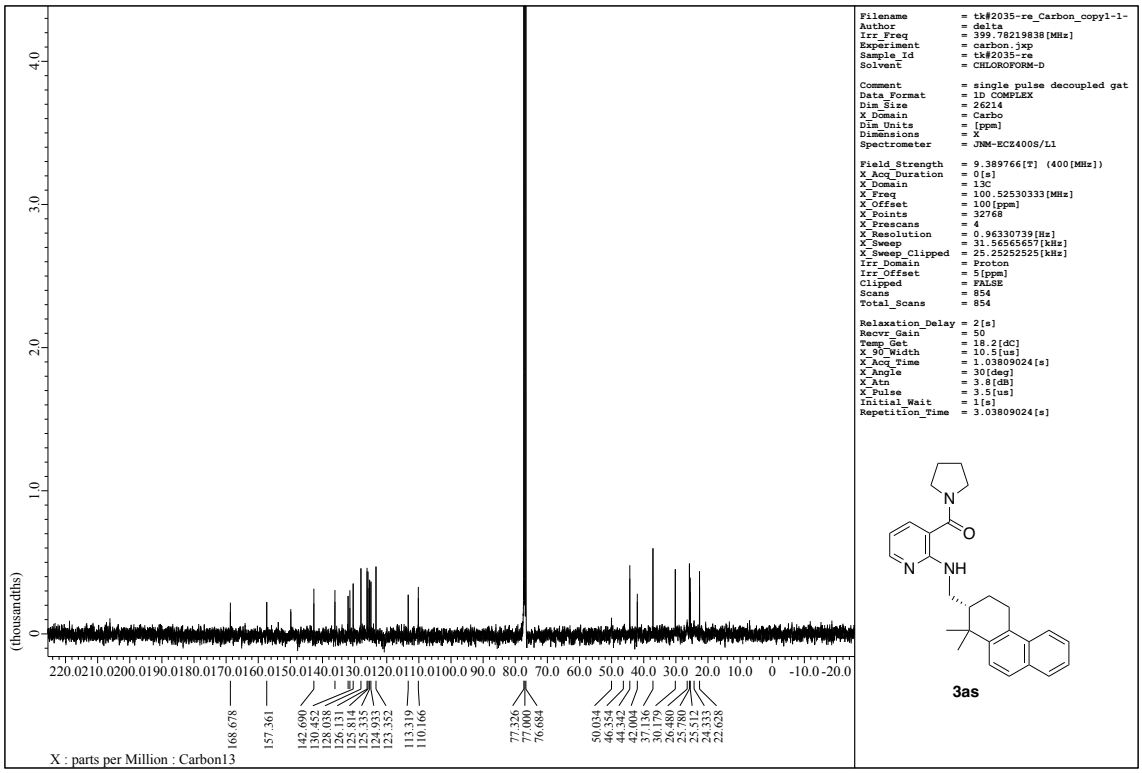

NOE difference spectrum
irradiation
X : parts per Million : Proton
abundance

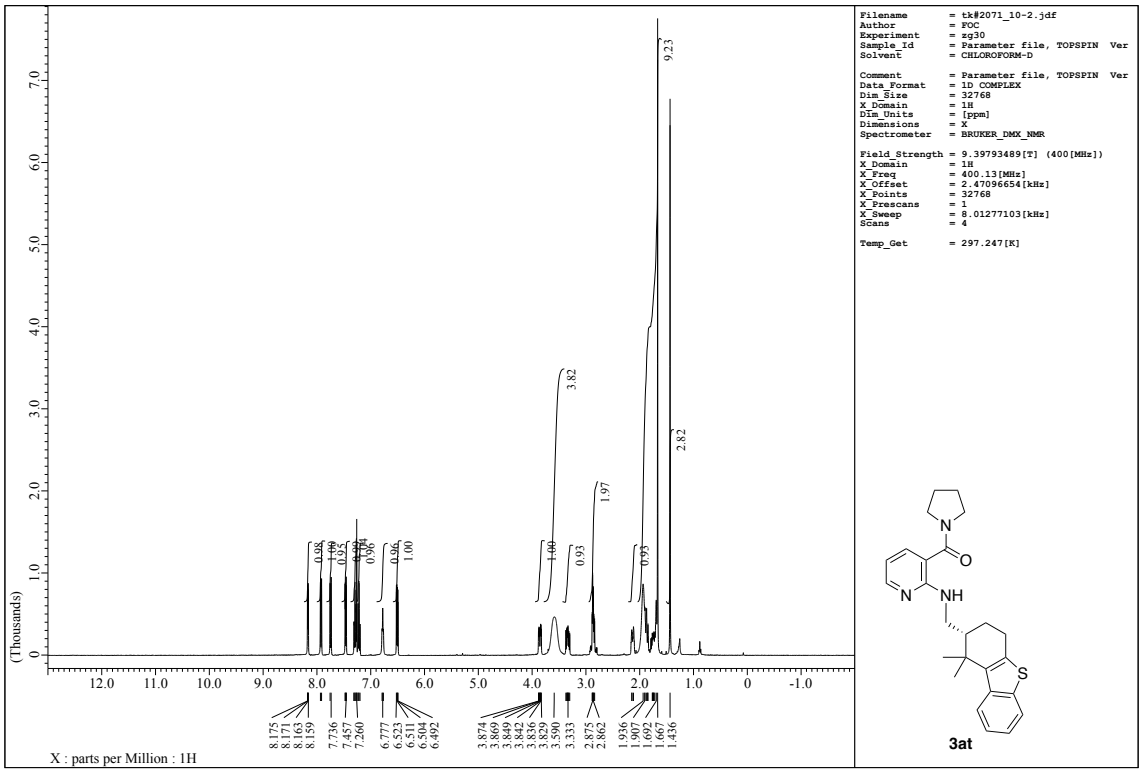

NOE difference spectrum
irradiation
X : parts per Million : Proton
abundance

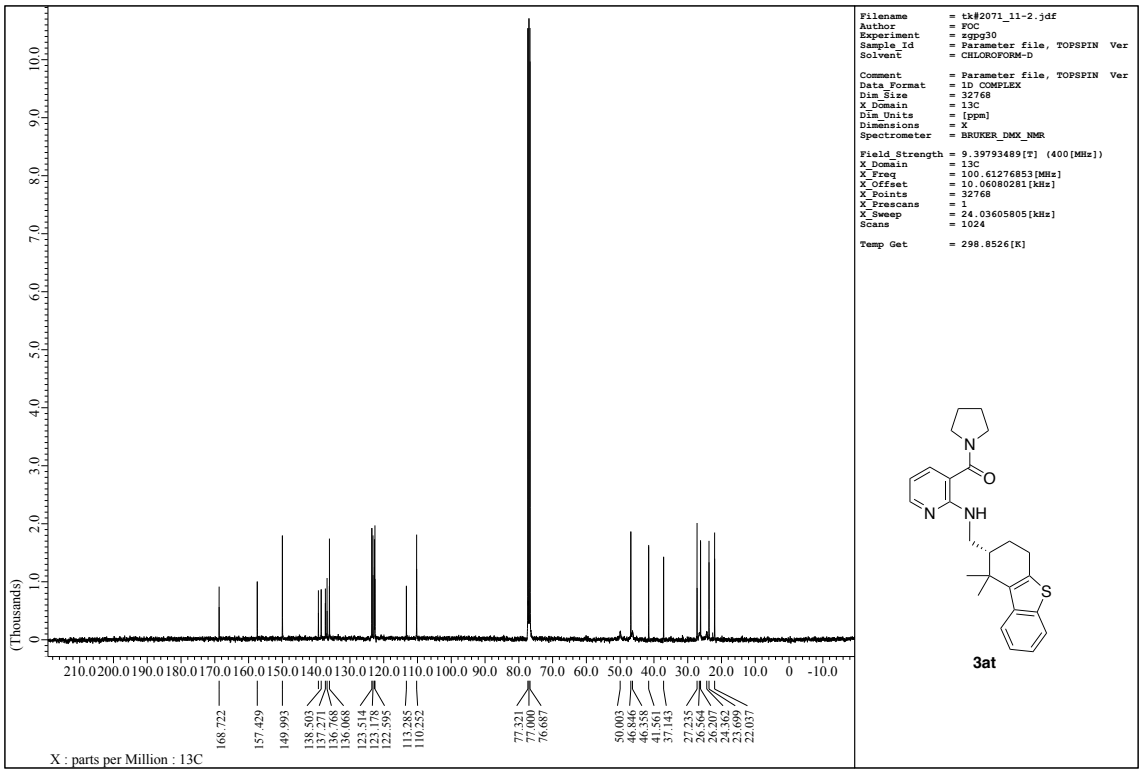

abundance
X : parts per Million : Proton
Filename = tk#3055-re_Proton-1-5.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#3055-re
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45739775[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 22.8[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]
3ar

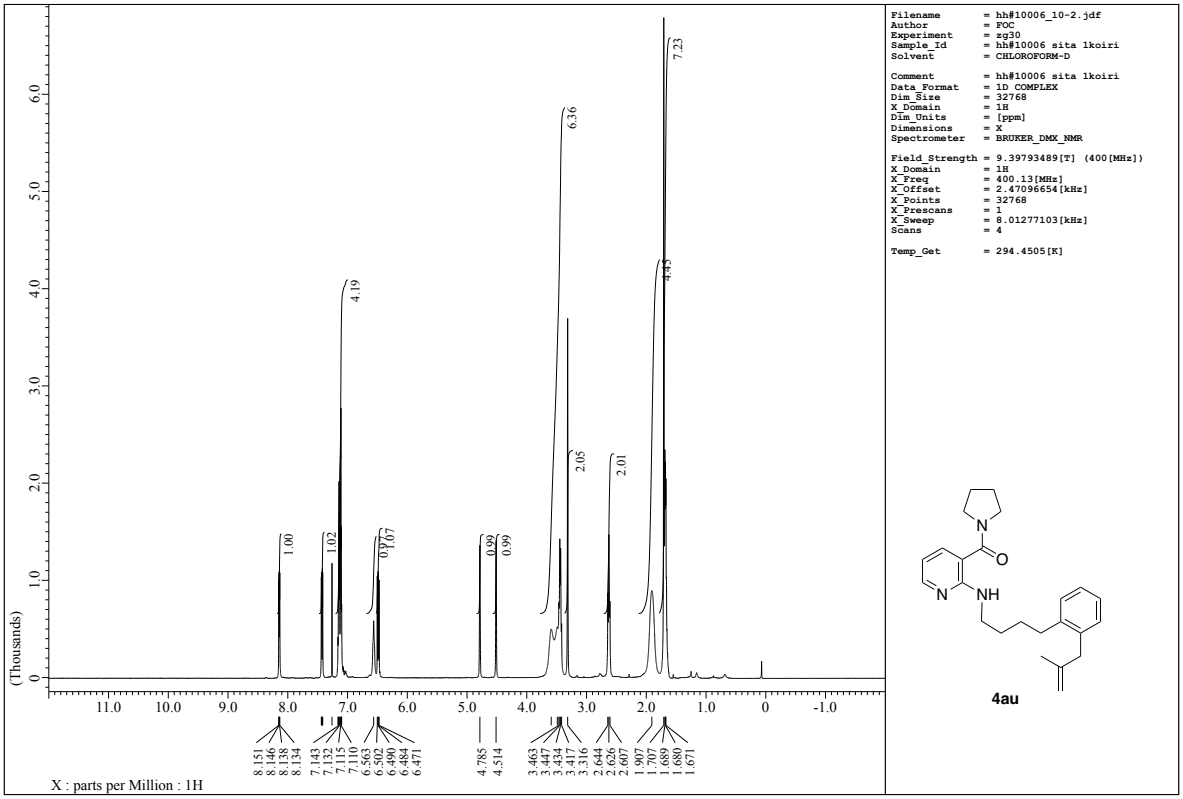

(thousandths)
X : parts per Million : Carbon13
Filename = tk#3055-re_Carbon_copy1-1-
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#3055-re
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [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 = 408
Total_Scans = 408
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]
3ar

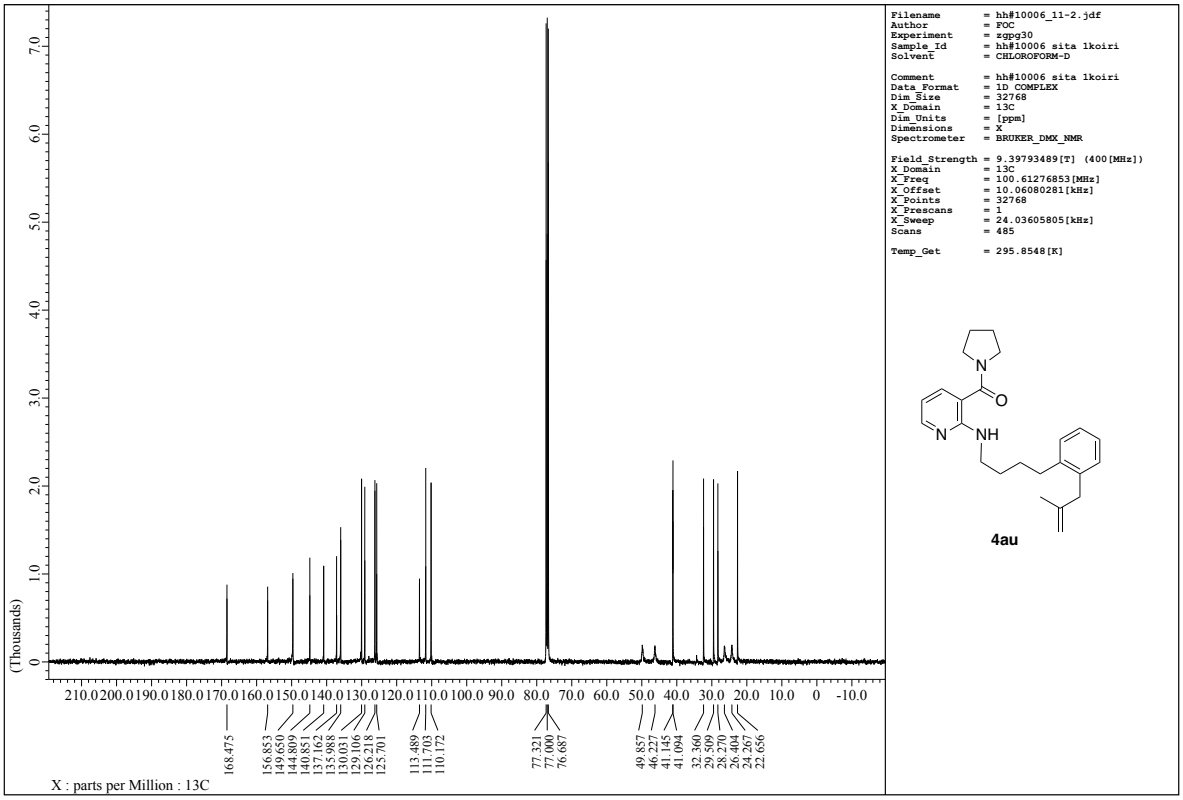

Filename = tk#2035-re_Proton-1-7.jdf
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#2035-re
Solvent = CHLOROFORM-D
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proto
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
Field_Strength = 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 = 18.1[dC]
X_90_Width = 5.9[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.9[dB]
X_Pulse = 2.95[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18628096[s]
abundance
X : parts per Million : Proton
3as

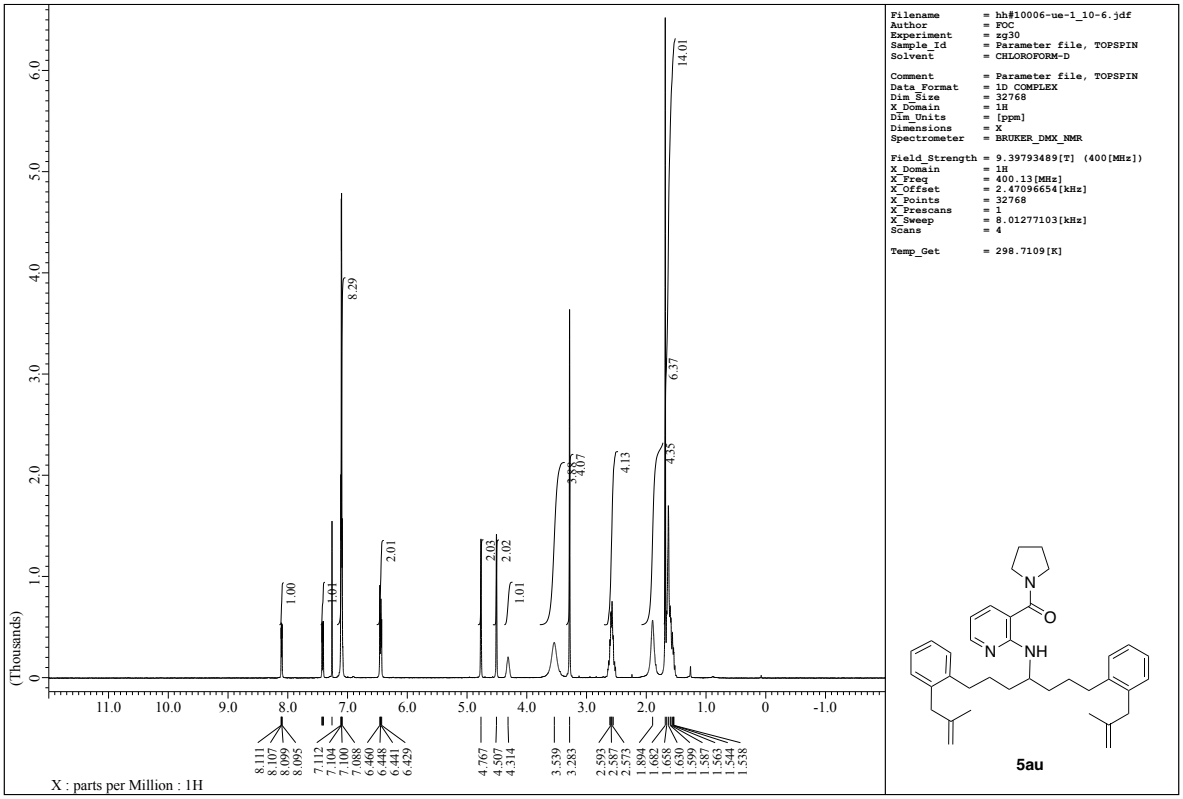

Filename = tk#2035-re_Carbon_copy1-1-
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#2035-re
Solvent = CHLOROFORM-D
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbo
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ400S/L1
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 = 854
Total_Scans = 854
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 18.2[dC]
X_90_Width = 10.5[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.5[us]
Initial_Wait = 1[s]
Repetition_Time = 3.03809024[s]
(thousandths)
X : parts per Million : Carbon13
3as

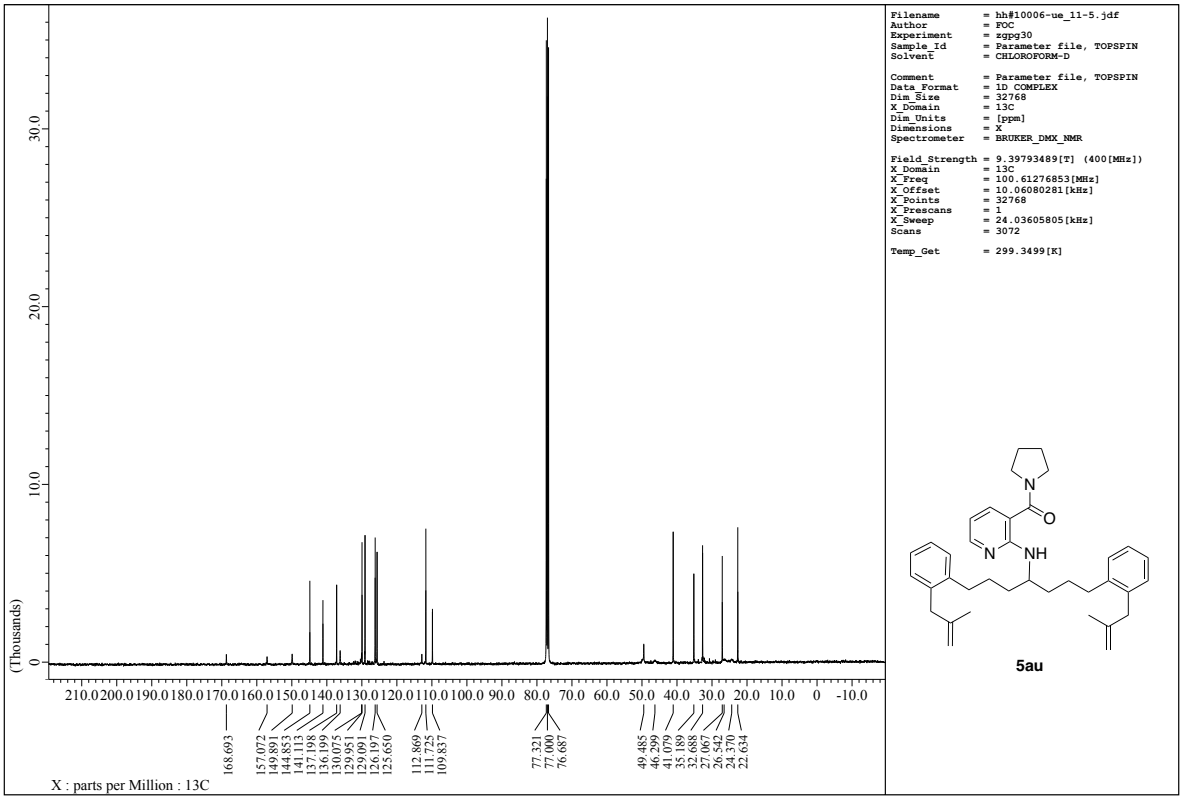

Filename = tk#2071_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 297.247[K]
(Thousands)
X : parts per Million : 1H
3at

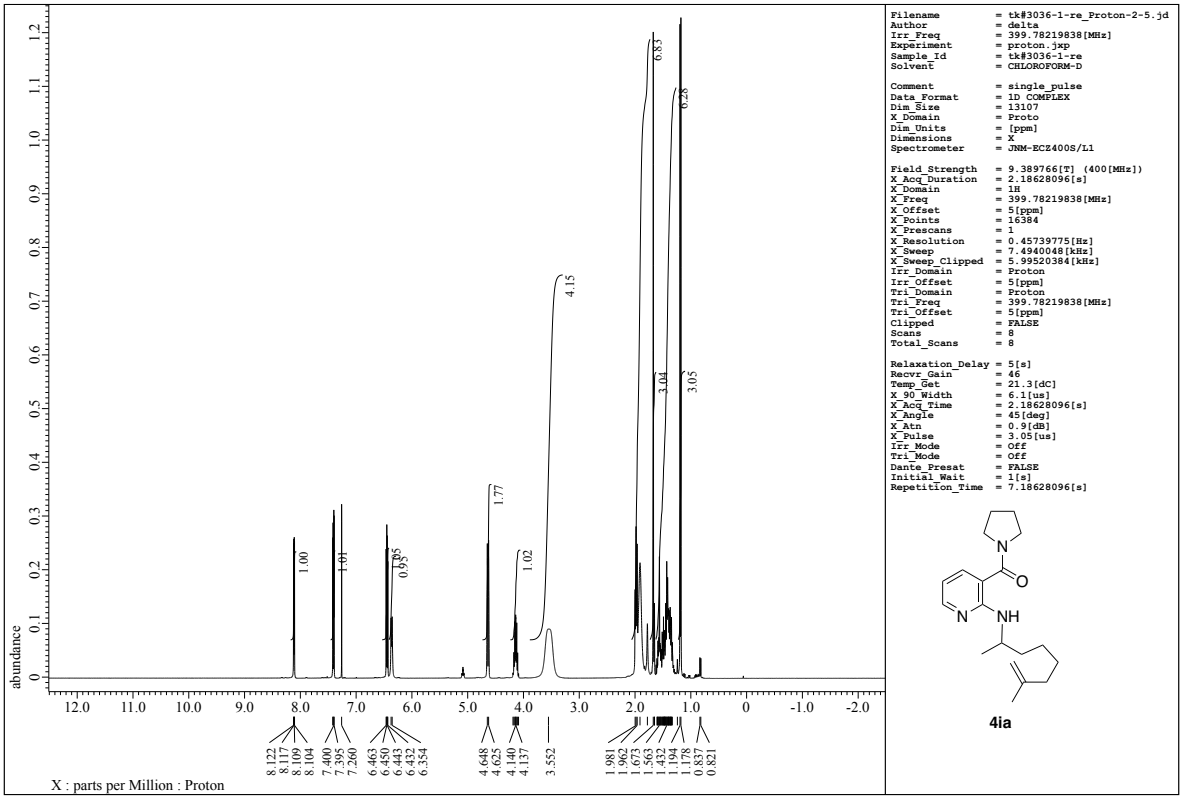

Filename = tk#2071_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 1024
Temp Get = 298.8526[K]
(Thousands)
X : parts per Million : 13C
3at

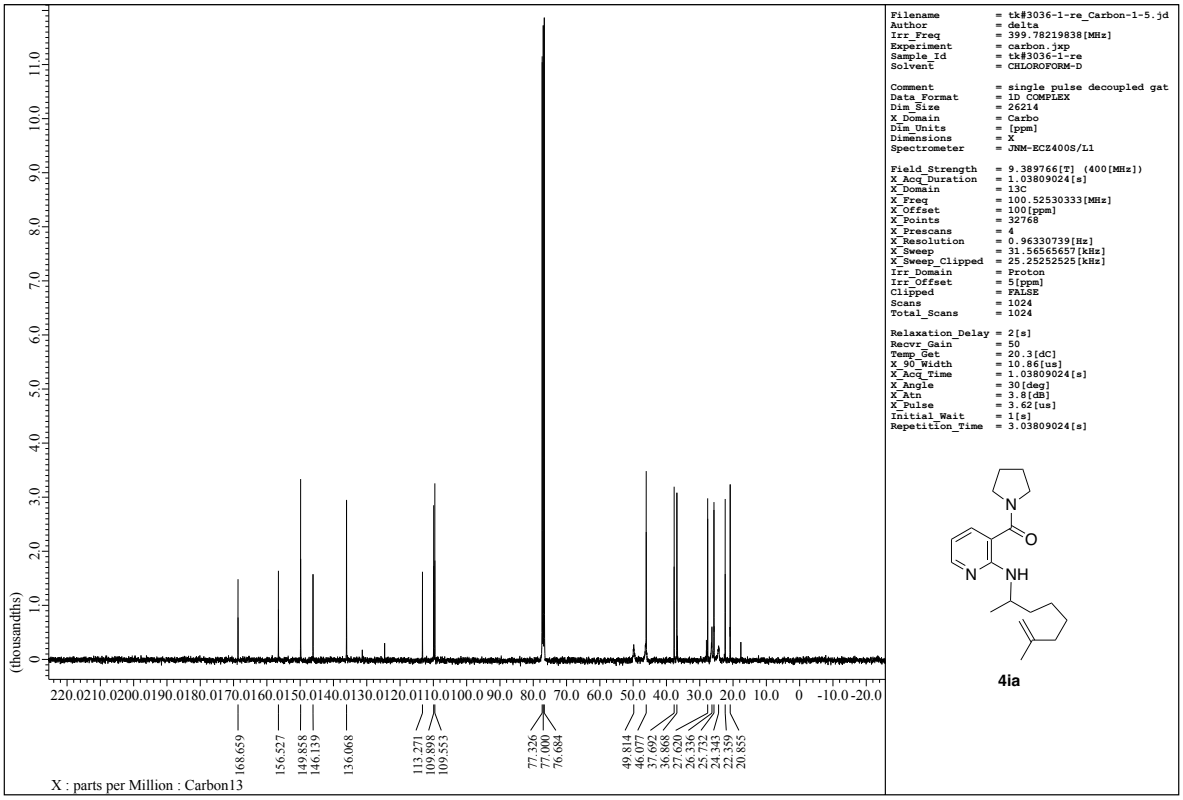

Filename = hh#10006_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#10006 sita lkoiri
Solvent = CHLOROFORM-D
Comment = hh#10006 sita lkoiri
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
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 = 294.4505[K]
(Thousands)
X : parts per Million : 1H
4au

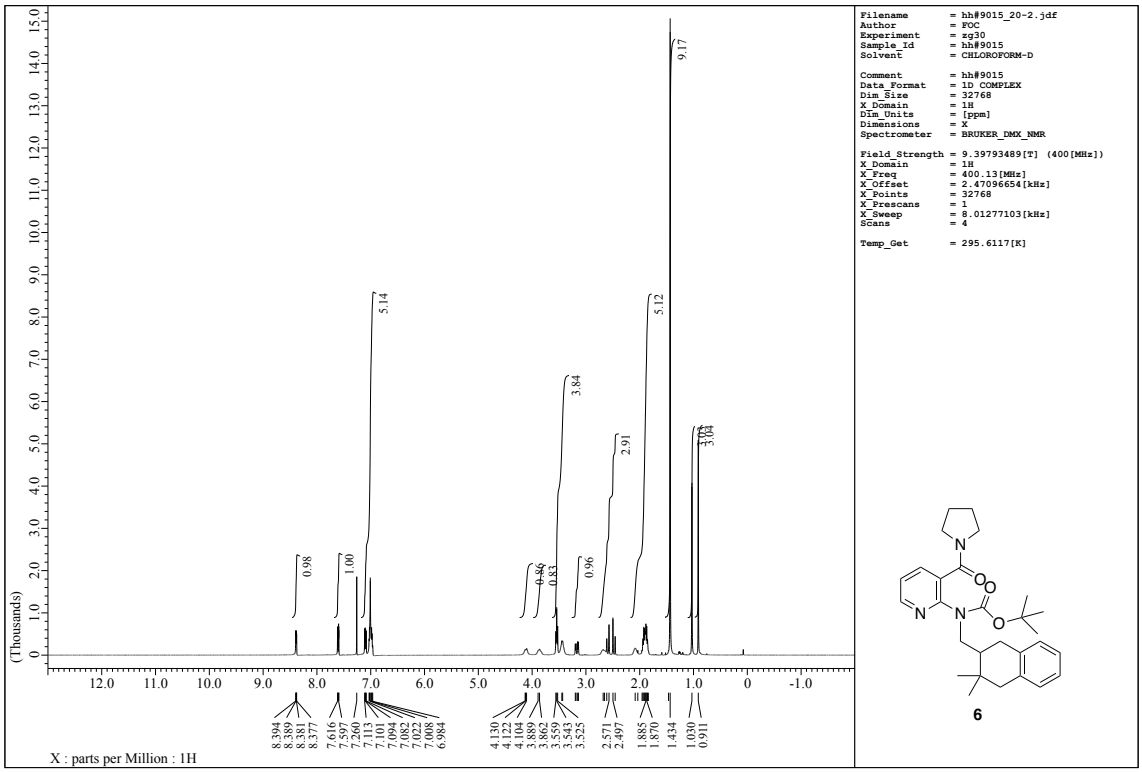

Filename = hh#10006_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#10006 sita lkoiri
Solvent = CHLOROFORM-D
Comment = hh#10006 sita lkoiri
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
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 = 485
Temp_Get = 295.8548[K]
(Thousands)
X : parts per Million : 13C
4au

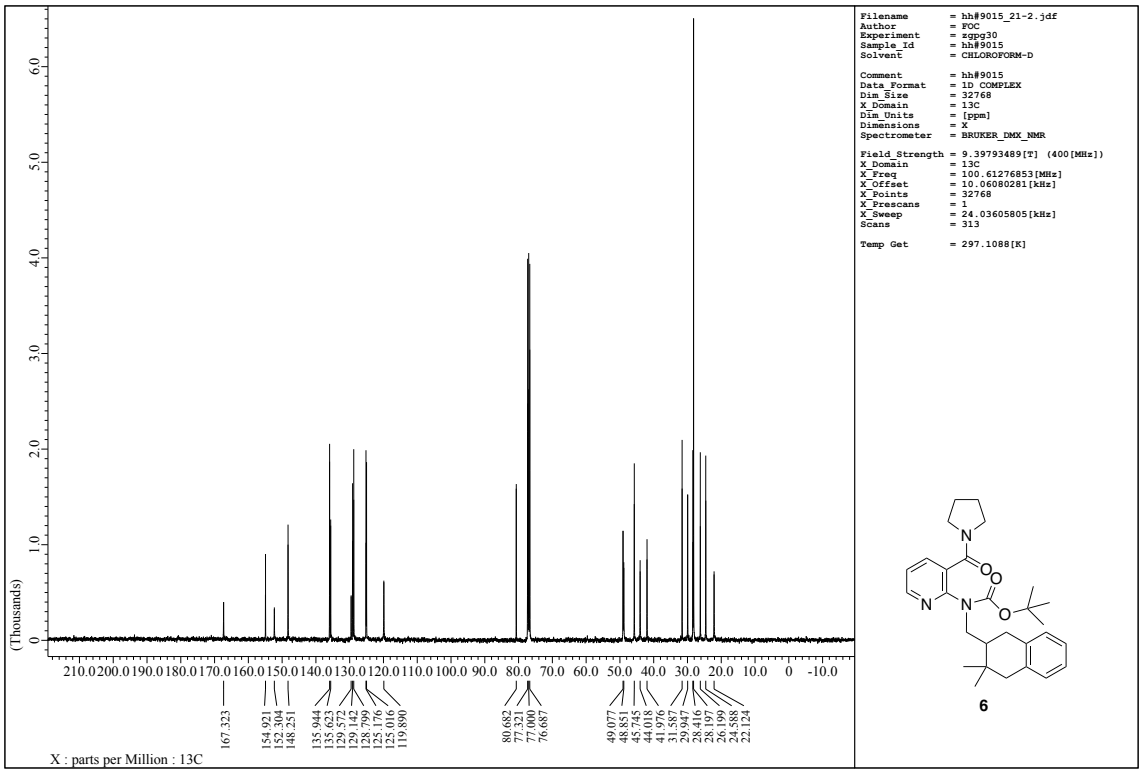

Filename = hh#10006-ue-1_10-6.jdf
Author = FOC
Experiment = zg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 298.7109[K]
(Thousands)
X : parts per Million : 1H
5au

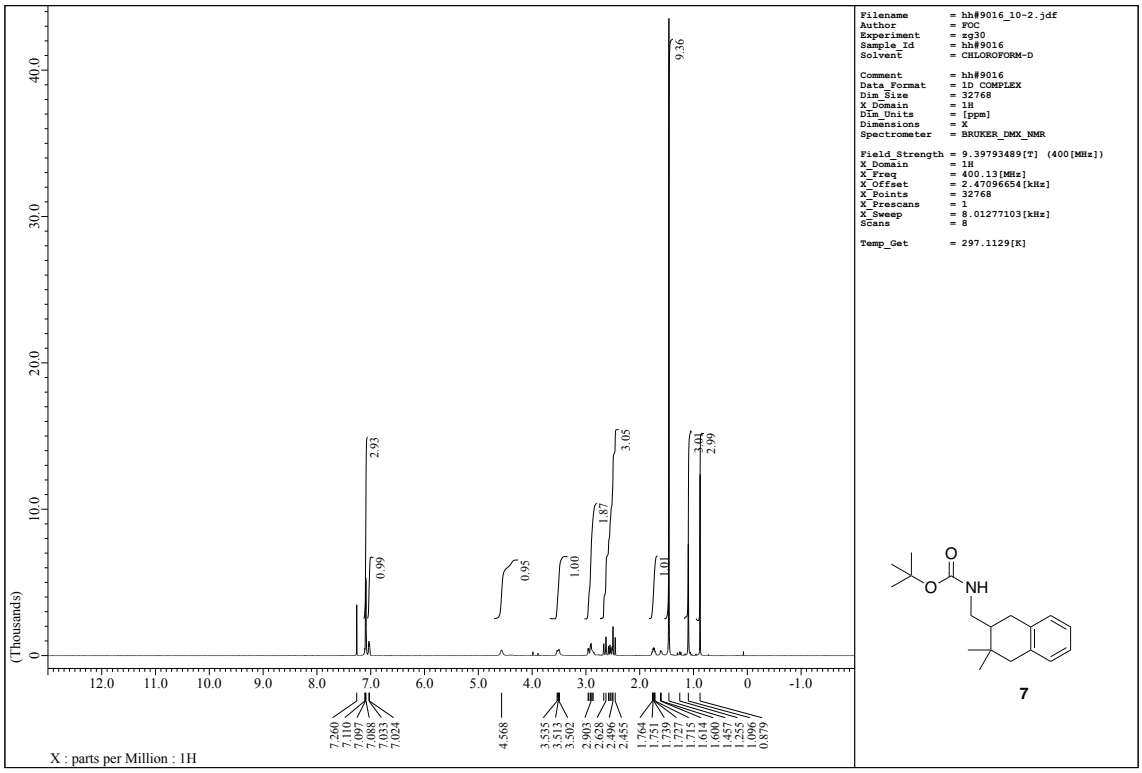

Filename = hh#10006-ue_11-5.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = Parameter file, TOPSPIN
Solvent = CHLOROFORM-D
Comment = Parameter file, TOPSPIN
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (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 = 3072
Temp_Get = 299.3499[K]
(Thousands)
X : parts per Million : 13C
5au

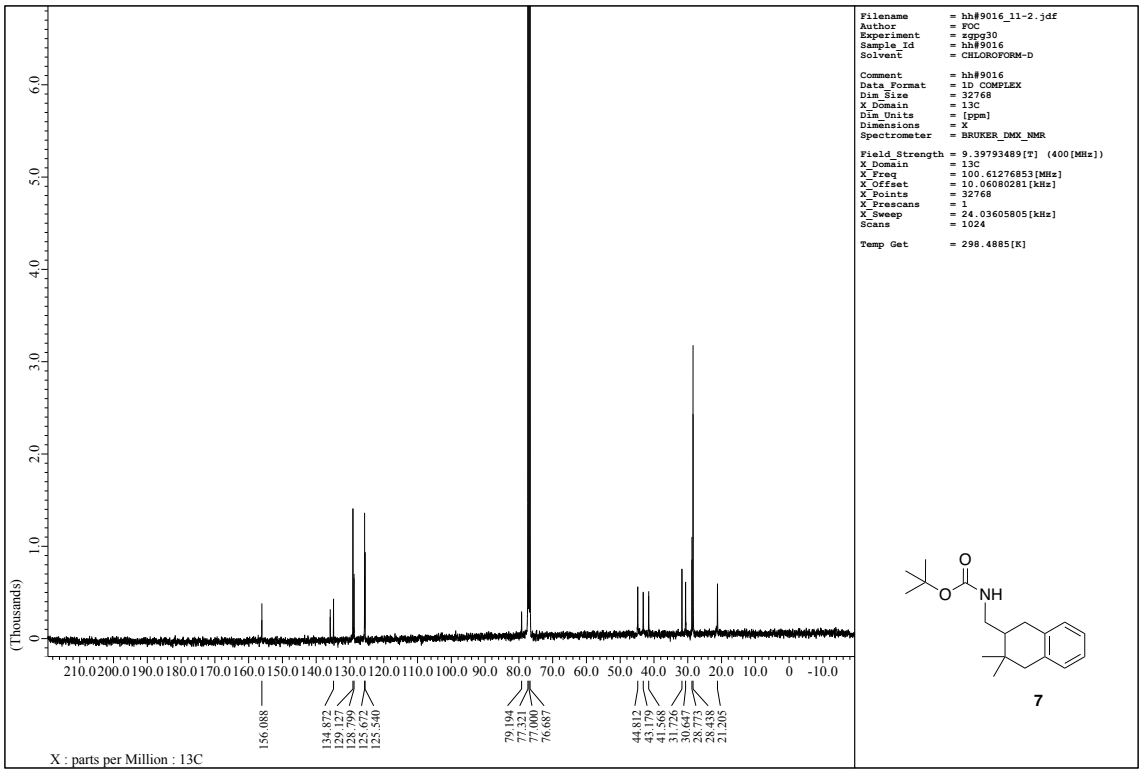

abundance
X : parts per Million : Proton
1.00
1.01
0.95
0.95
1.77
1.02
4.15
6.83
6.28
3.04
3.05
8.122
8.117
8.109
8.104
7.400
7.395
7.260
6.463
6.450
6.443
6.432
6.354
4.648
4.625
4.140
4.137
3.552
1.981
1.962
1.675
1.563
1.432
1.194
1.178
0.837
0.821
Filename = tk#3036-1-re_Proton-2-5.jd
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = proton.jxp
Sample_Id = tk#3036-1-re
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 = 21.3[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]
4ia

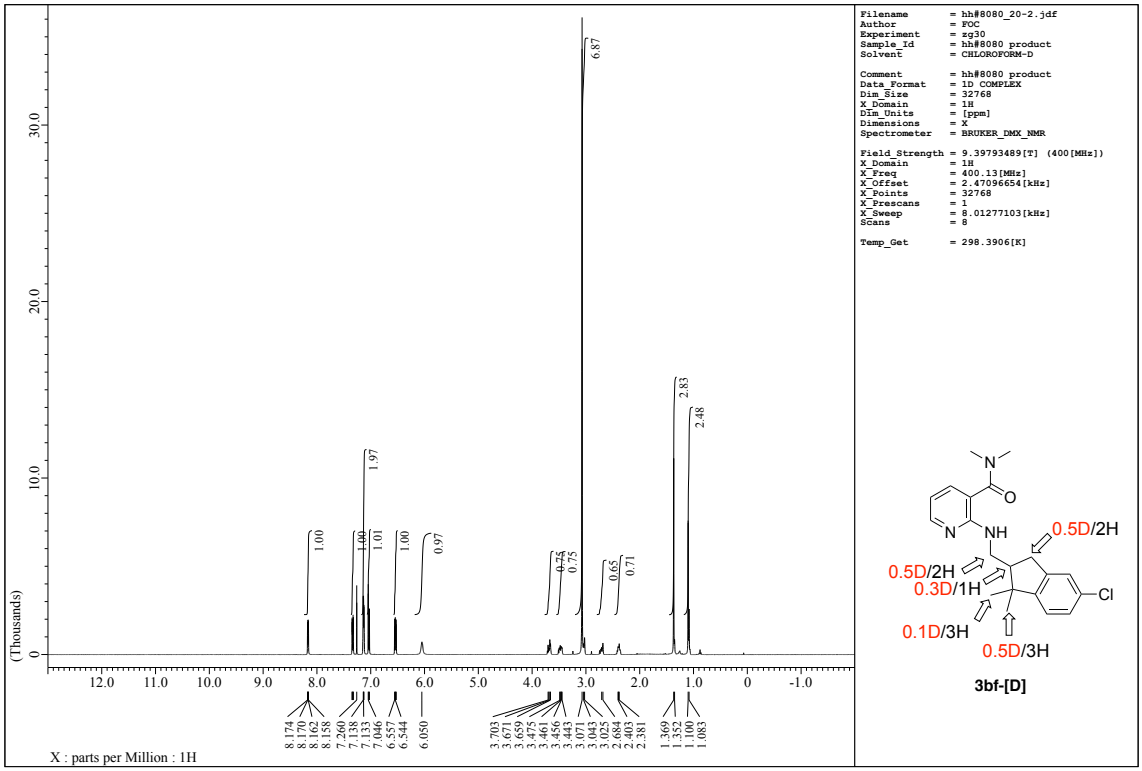

(thousandths)
X : parts per Million : Carbon13
168.659
156.527
149.858
146.139
136.068
113.271
109.898
109.553
77.326
77.000
76.684
49.814
46.077
37.692
36.668
27.620
26.336
25.732
24.343
22.359
20.855
Filename = tk#3036-1-re_Carbon-1-5.jd
Author = delta
Irr_Freq = 399.78219838[MHz]
Experiment = carbon.jxp
Sample_Id = tk#3036-1-re
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 = 20.3[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]
4ia

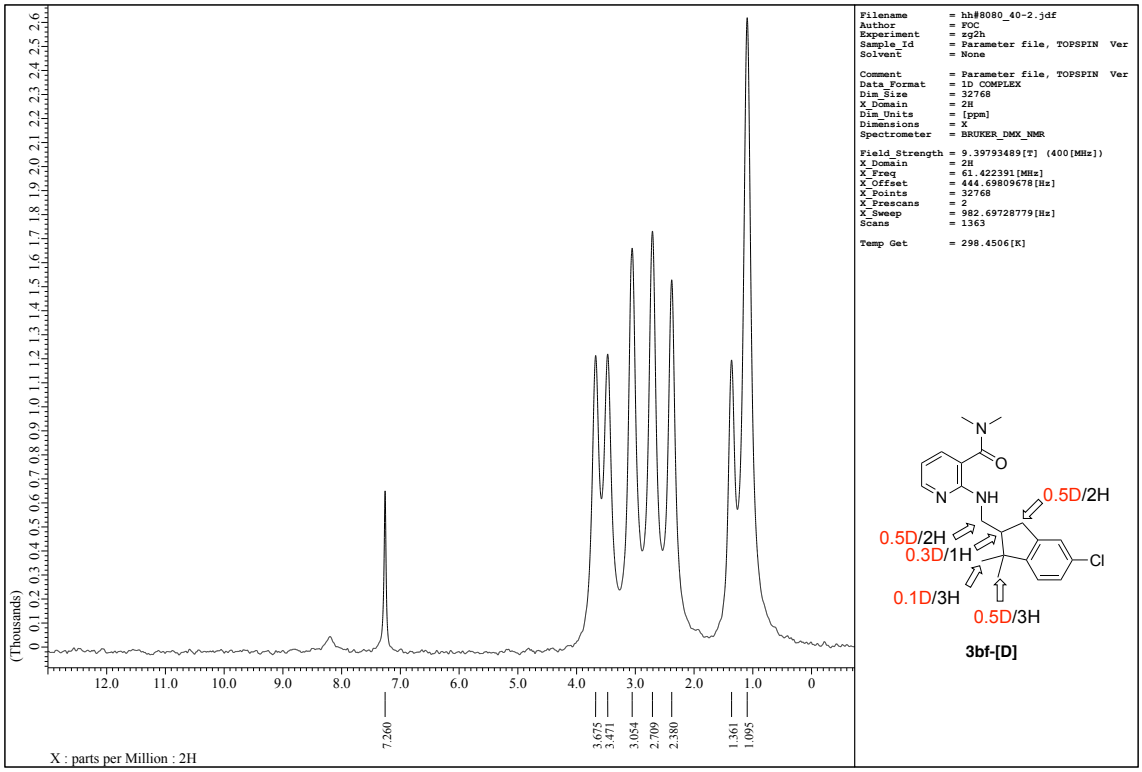

Filename = hh#9015_20-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9015
Solvent = CHLOROFORM-D
Comment = hh#9015
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 4
Temp_Get = 295.6117[K]
(Thousands)
X : parts per Million : 1H
6

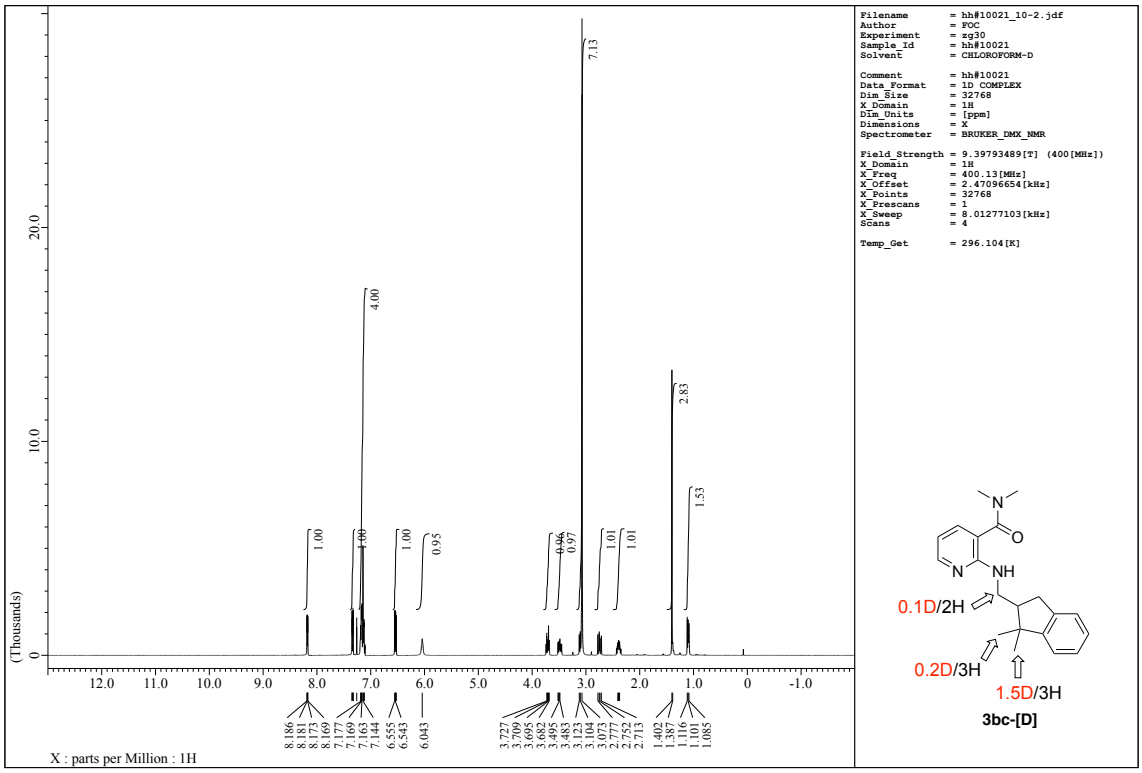

Filename = hh#9015_21-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9015
Solvent = CHLOROFORM-D
Comment = hh#9015
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 13C
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 13C
X_Freq = 100.61276853[MHz]
X_Offset = 10.06080281[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 24.03605805[kHz]
Scans = 313
Temp Get = 297.1088[K]
(Thousands)
X : parts per Million : 13C
6

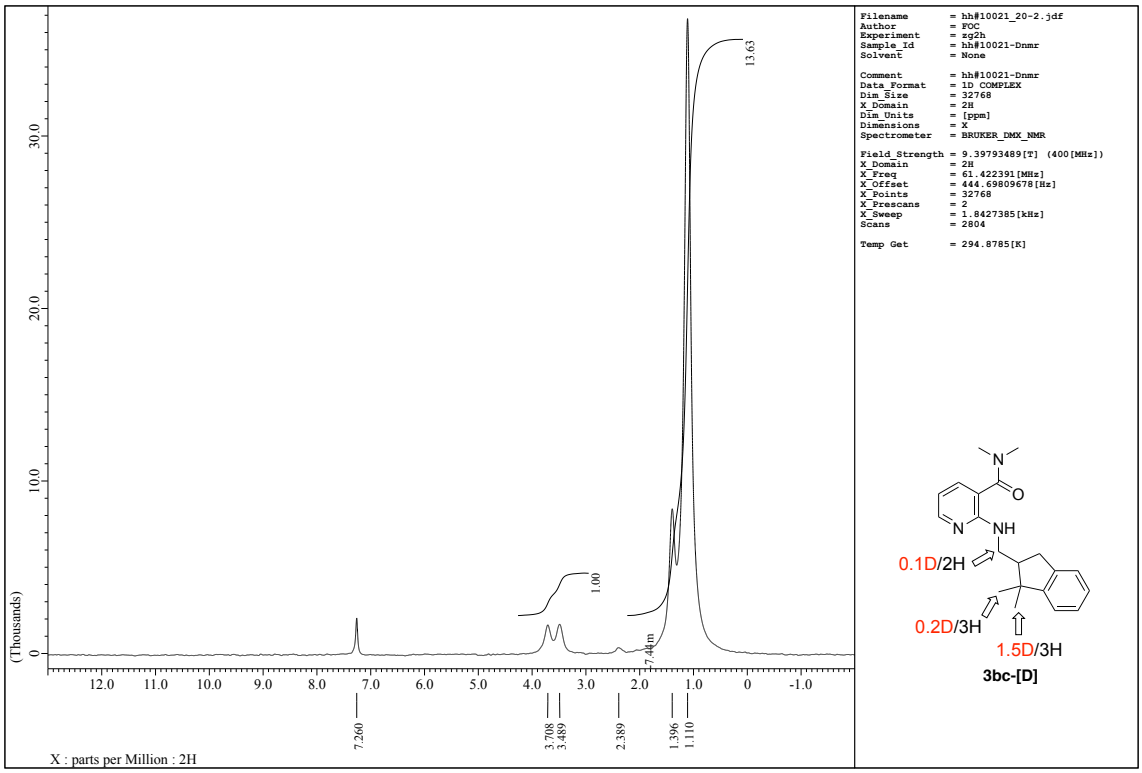

Filename = hh#9016_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#9016
Solvent = CHLOROFORM-D
Comment = hh#9016
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 1H
X_Freq = 400.13[MHz]
X_Offset = 2.47096654[kHz]
X_Points = 32768
X_Prescans = 1
X_Sweep = 8.01277103[kHz]
Scans = 8
Temp_Get = 297.1129[K]
(Thousands)
X : parts per Million : 1H
9.36
2.93
0.99
0.95
1.00
1.87
3.05
1.01
3.01
2.99
7.260
7.110
7.097
7.088
7.053
7.024
4.568
3.535
3.513
3.502
2.903
2.628
2.496
2.455
1.764
1.751
1.739
1.727
1.715
1.614
1.600
1.457
1.255
1.096
0.879
7


Filename = hh#9016_11-2.jdf
Author = FOC
Experiment = zgpg30
Sample_Id = hh#9016
Solvent = CHLOROFORM-D
Comment = hh#9016
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 = 298.4885[K]
(Thousands)
X : parts per Million : 13C
156.088
134.872
129.127
128.799
125.672
125.540
79.194
77.321
77.000
76.687
44.812
43.179
41.568
31.726
30.647
28.773
28.438
21.205
7

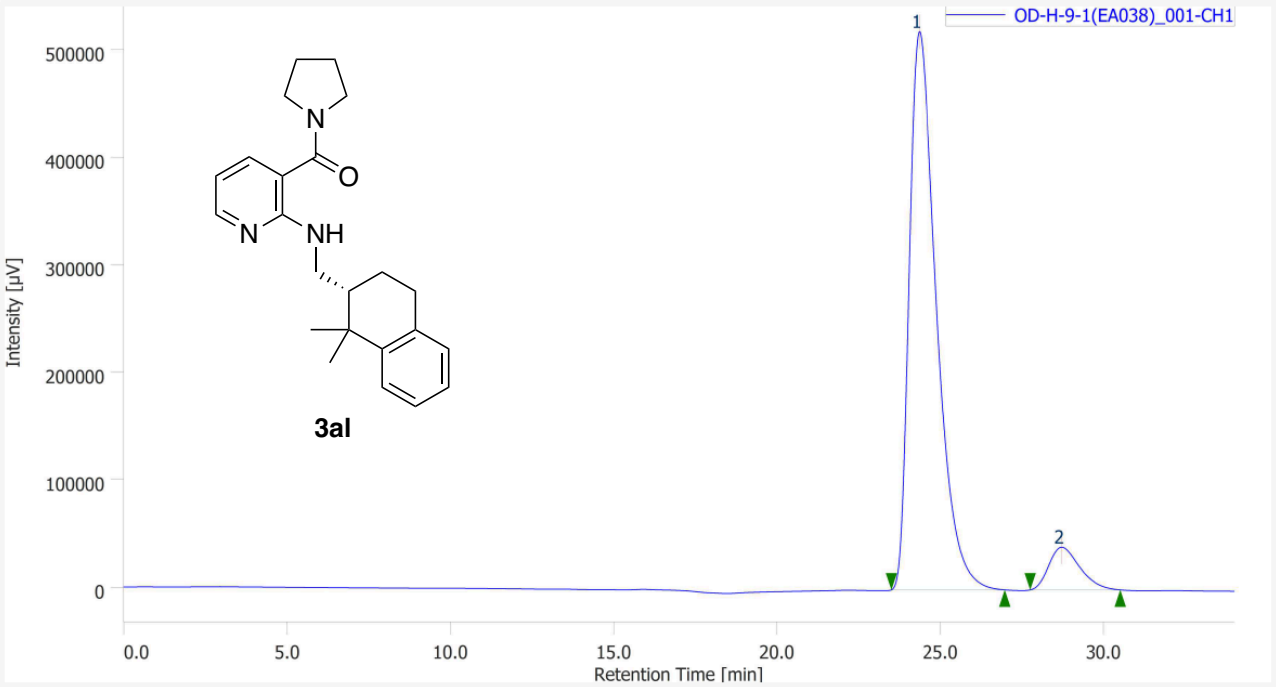

Filename = hh#8080_20-2.jdf
Author = FOC
Experiment = sg30
Sample_Id = hh#8080 product
Solvent = CHLOROFORM-D
Comment = hh#8080 product
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 1H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 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 = 298.3906[K]
(Thousands)
X : parts per Million : 1H
0.5D/2H
0.5D/2H
0.3D/1H
0.1D/3H
0.5D/3H
Cl
NH
3bf-[D]


Filename = hh#8080_40-2.jdf
Author = FOC
Experiment = sg2h
Sample_Id = Parameter file, TOPSPIN Ver
Solvent = None
Comment = Parameter file, TOPSPIN Ver
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 2H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 2H
X_Freq = 61.422391[MHz]
X_Offset = 444.69809678[Hz]
X_Points = 32768
X_Prescans = 2
X_Sweep = 982.69728779[Hz]
Scans = 1363
Temp Get = 298.4506[K]
(Thousands)
X : parts per Million : 2H
0.5D/2H
0.5D/2H
0.3D/1H
0.1D/3H
0.5D/3H
Cl
NH
3bf-[D]


Filename = hh#10021_10-2.jdf
Author = FOC
Experiment = zg30
Sample_Id = hh#10021
Solvent = CHLOROFORM-D
Comment = hh#10021
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 = 296.104[K]
(Thousands)
X : parts per Million : 1H
0.1D/2H
0.2D/3H
1.5D/3H
3bc-[D]

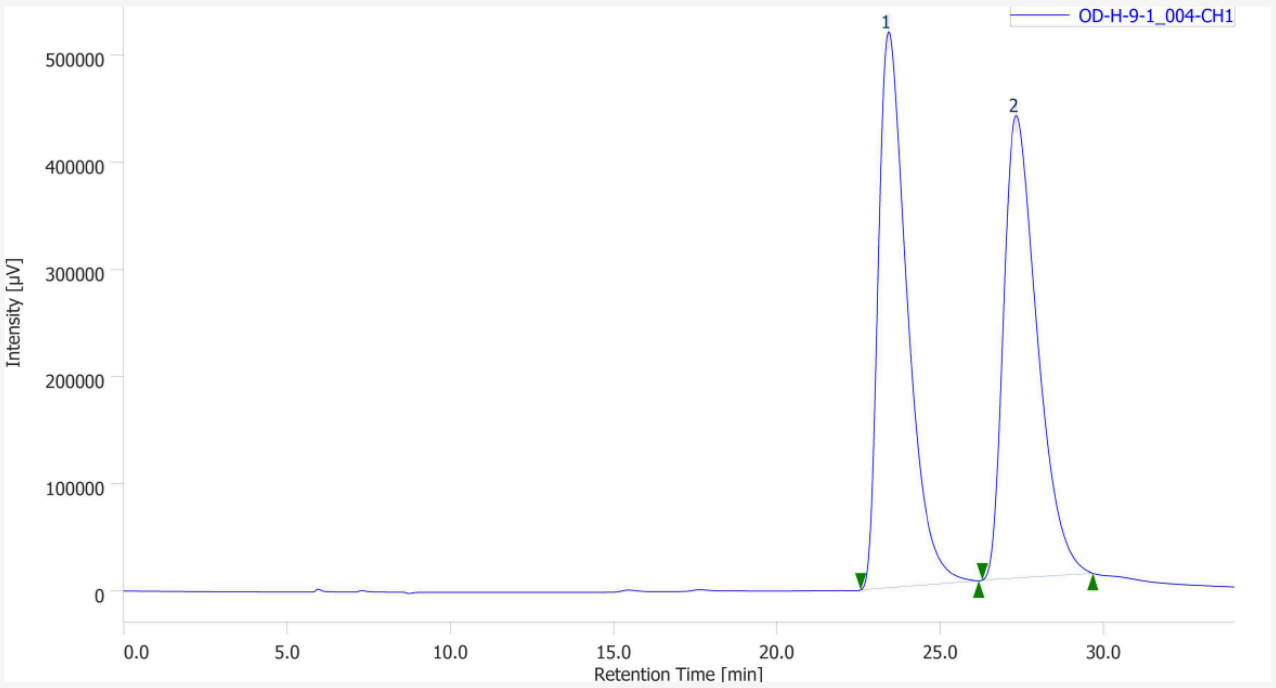

Filename = hh#10021_20-2.jdf
Author = FOC
Experiment = zg2h
Sample_Id = hh#10021-Dnmr
Solvent = None
Comment = hh#10021-Dnmr
Data_Format = 1D COMPLEX
Dim_Size = 32768
X_Domain = 2H
Dim_Units = [ppm]
Dimensions = X
Spectrometer = BRUKER_DMX_NMR
Field_Strength = 9.39793489[T] (400[MHz])
X_Domain = 2H
X_Freq = 61.422391[MHz]
X_Offset = 444.69809678[Hz]
X_Points = 32768
X_Prescans = 2
X_Sweep = 1.8427385[kHz]
Scans = 2804
Temp Get = 294.8785[K]
(Thousands)
X : parts per Million : 2H
0.1D/2H
0.2D/3H
1.5D/3H
3bc-[D]

19. Chiral HPLC charts



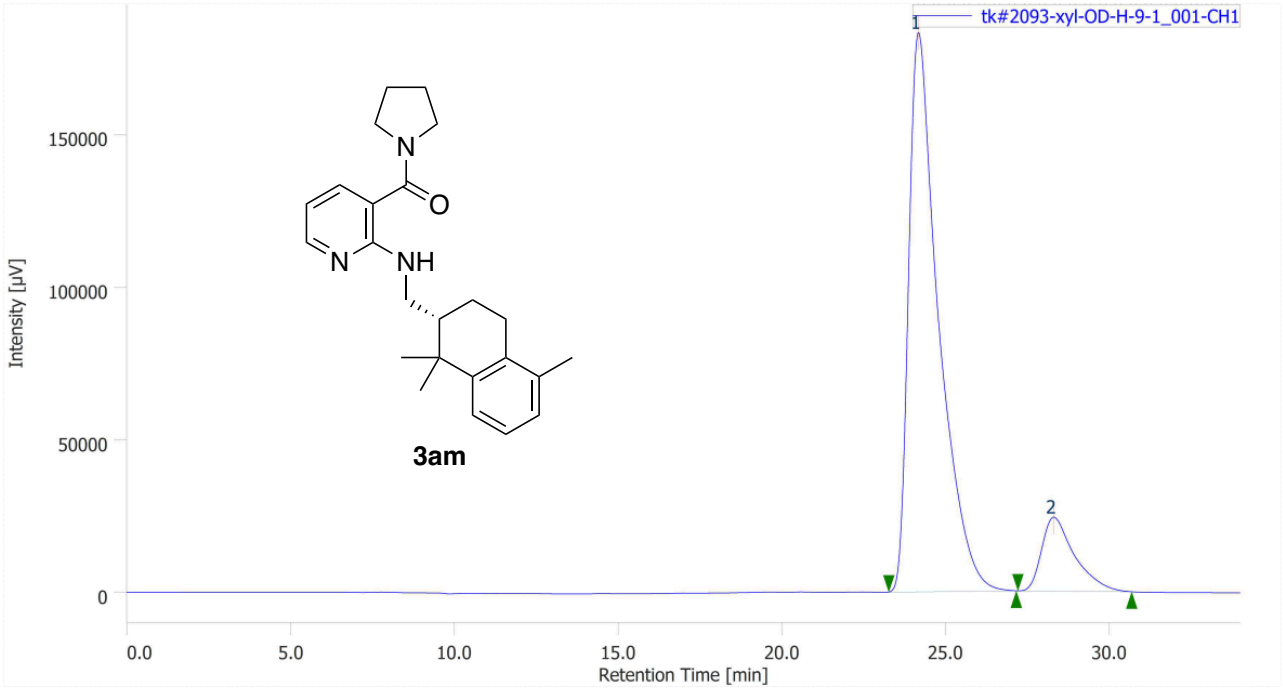
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
24.358	30014044	92.010	519158
28.717	2606315	7.990	39662



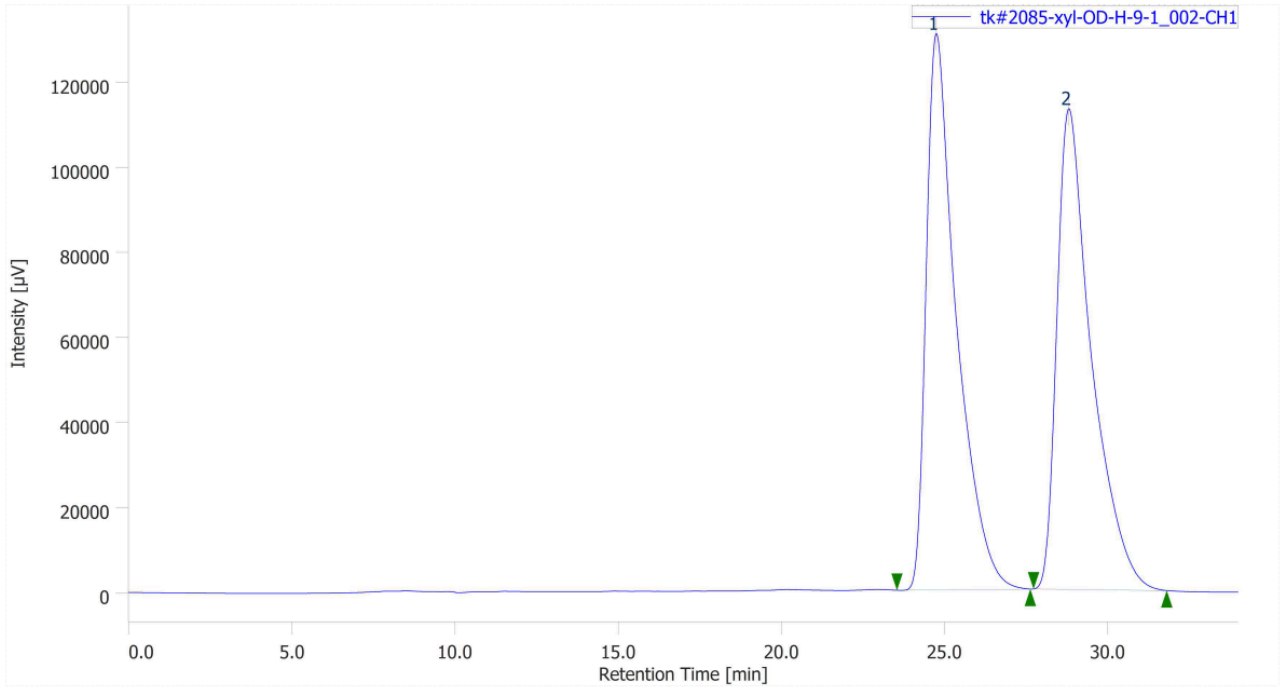
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
23.417	31918817	50.763	518593
27.325	30959428	49.237	431331



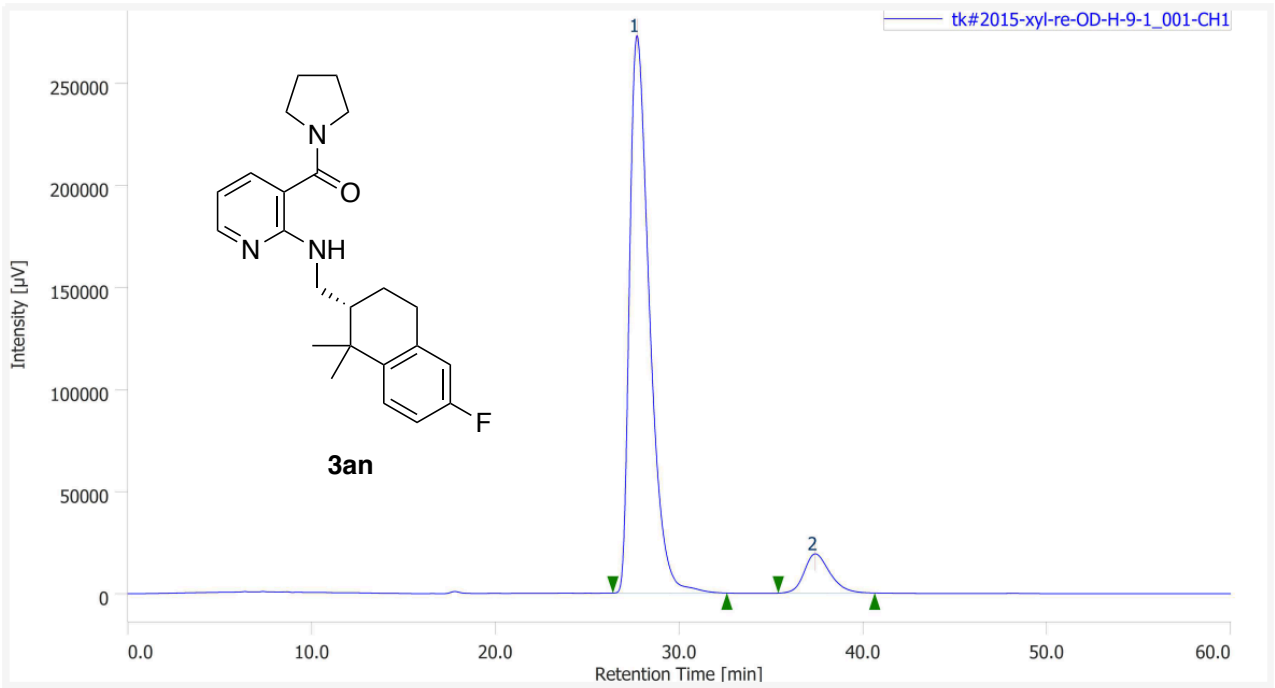
Peak Information

#	Peak Name	CH	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	Unknown	1	24.175	11599663	87.301	183142
2	Unknown	1	28.300	1687330	12.699	24248



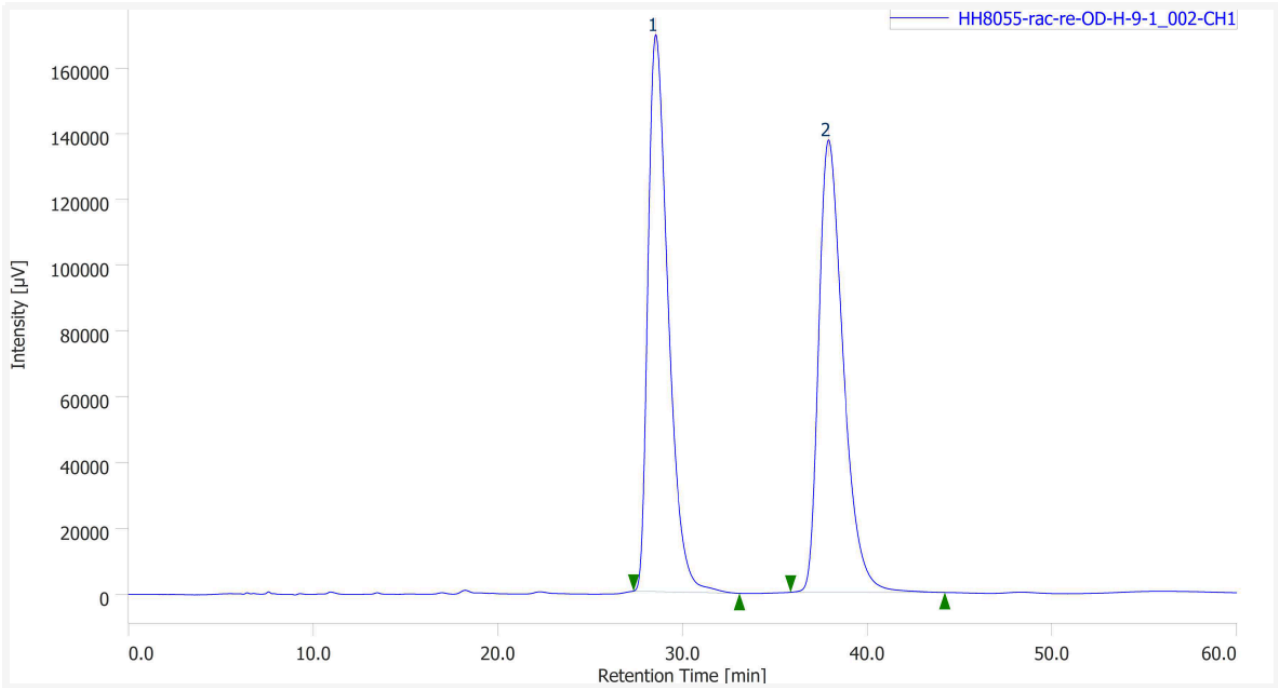
Peak Information

#	Peak Name	CH	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	Unknown	1	24.750	8425255	50.308	130884
2	Unknown	1	28.808	8322130	49.692	113090



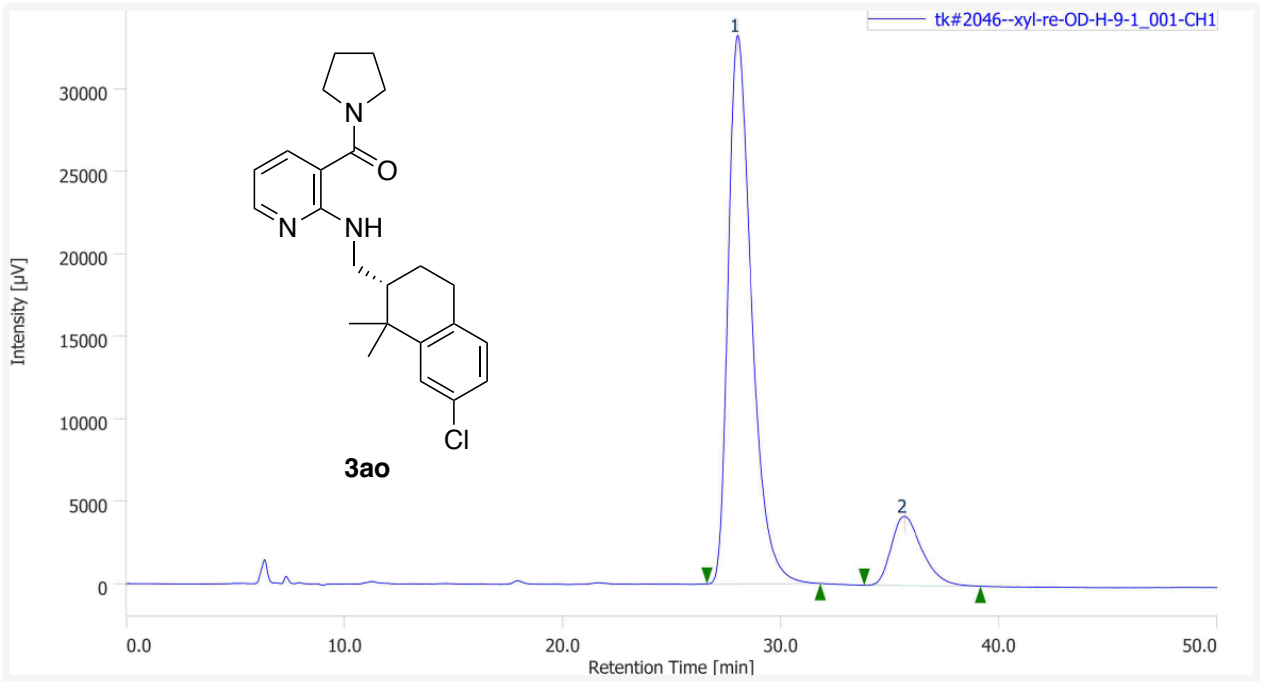
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
27.700	20418685	91.630	273061
37.392	1865159	8.370	19241



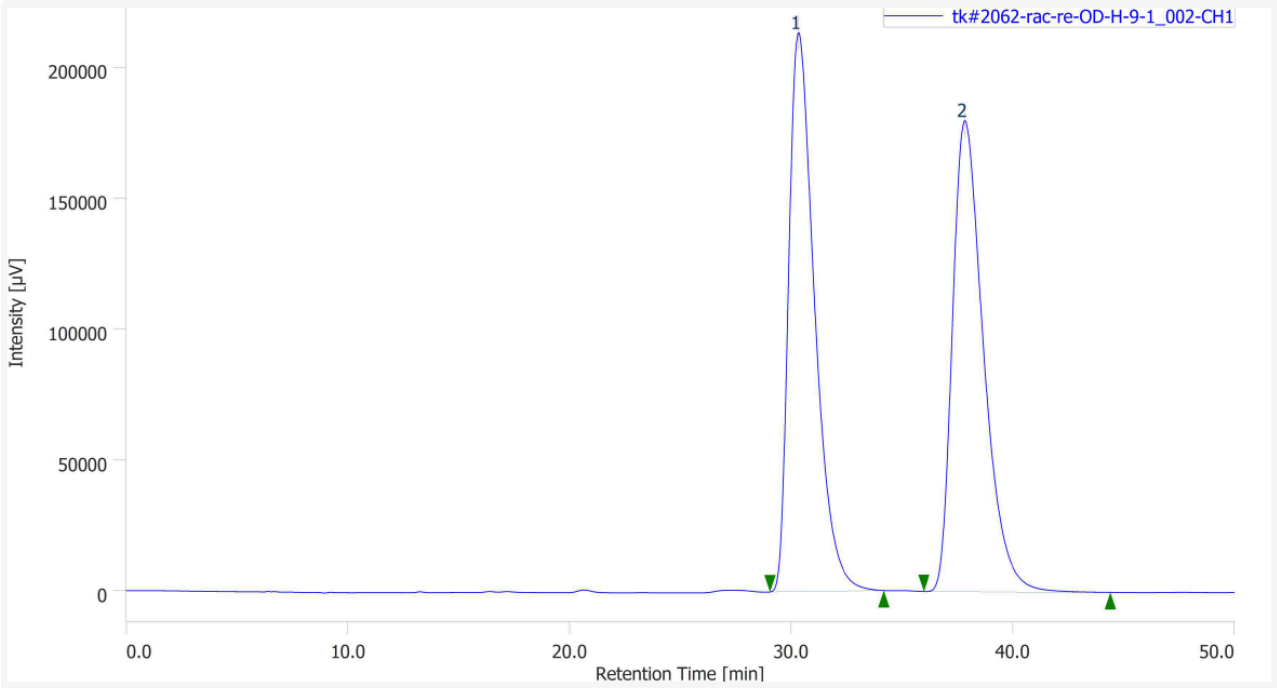
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
28.542	12852352	50.285	169267
37.892	12706485	49.715	137506



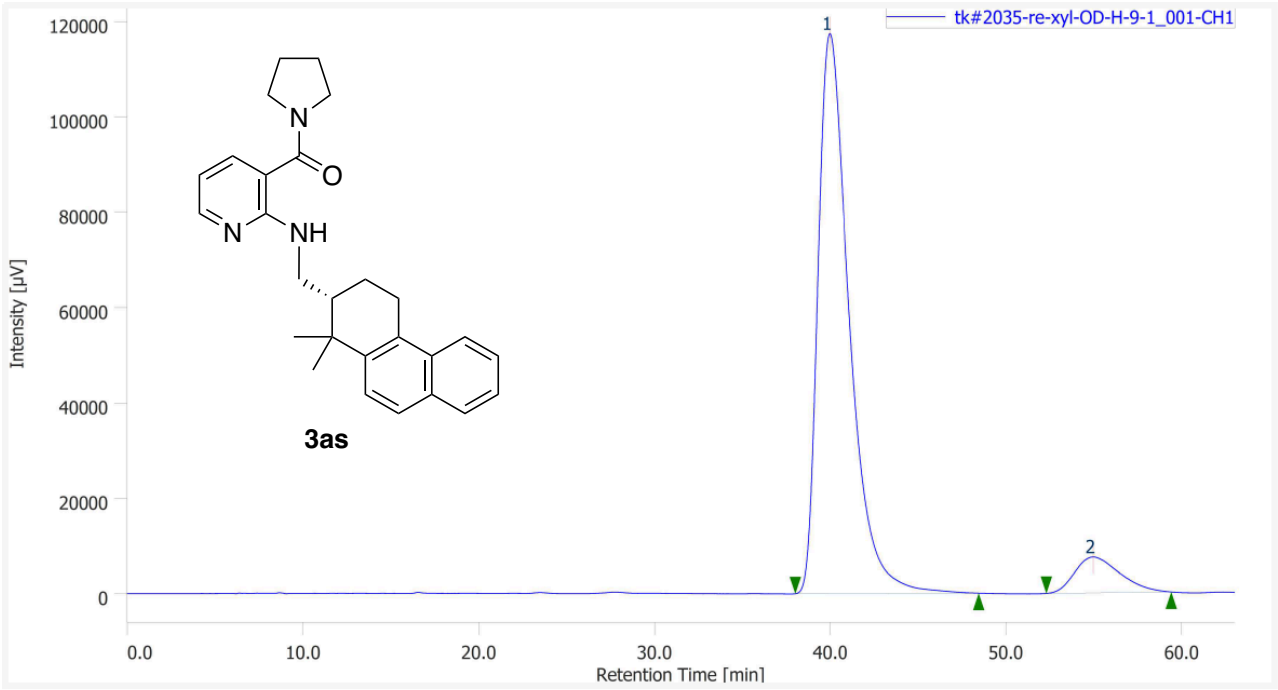
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
28.017	2491098	85.747	33247
35.675	414074	14.253	4202



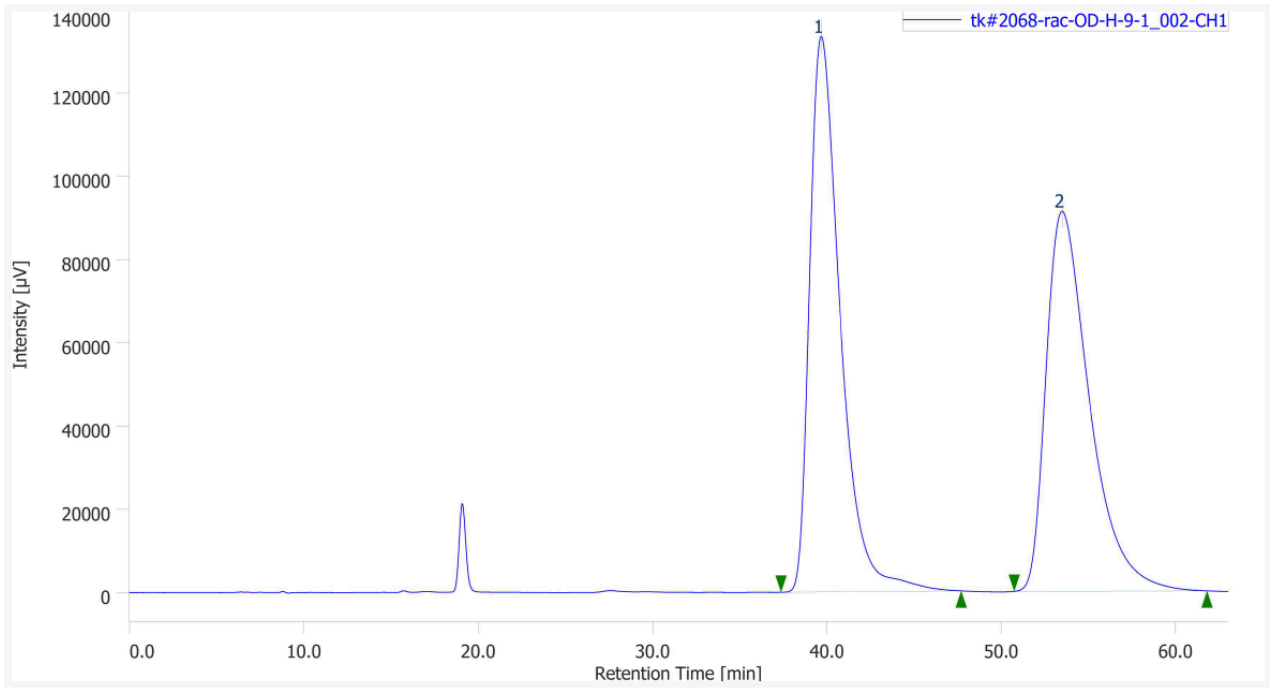
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
30.333	17238611	49.664	213495
37.833	17472103	50.336	179907



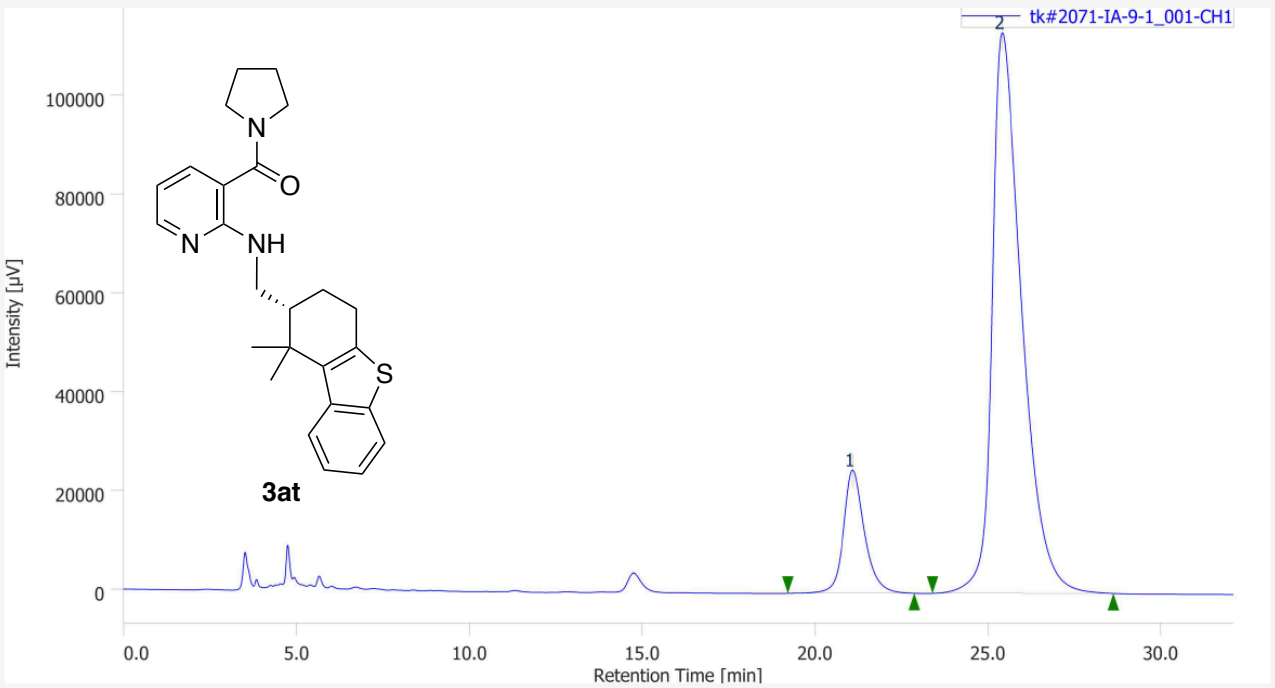
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
39.967	14524484	91.512	117474
54.942	1347242	8.488	7549



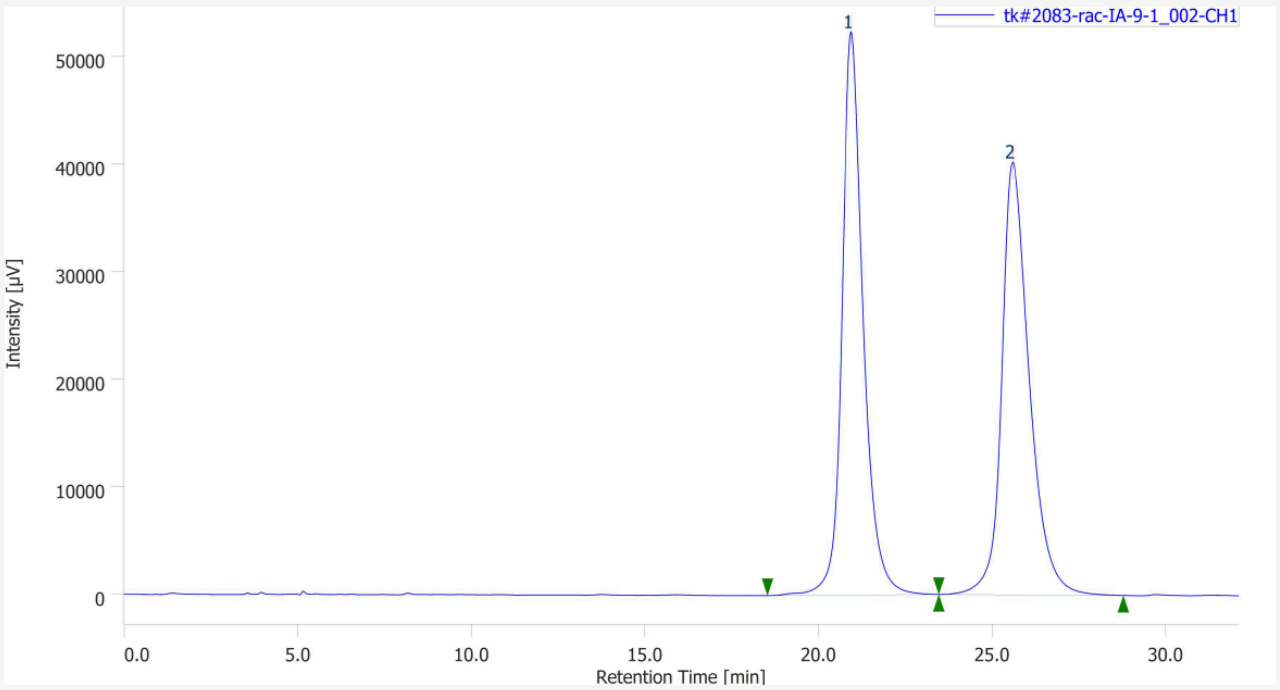
Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
39.667	16332401	50.865	133425
53.483	15776884	49.135	91257



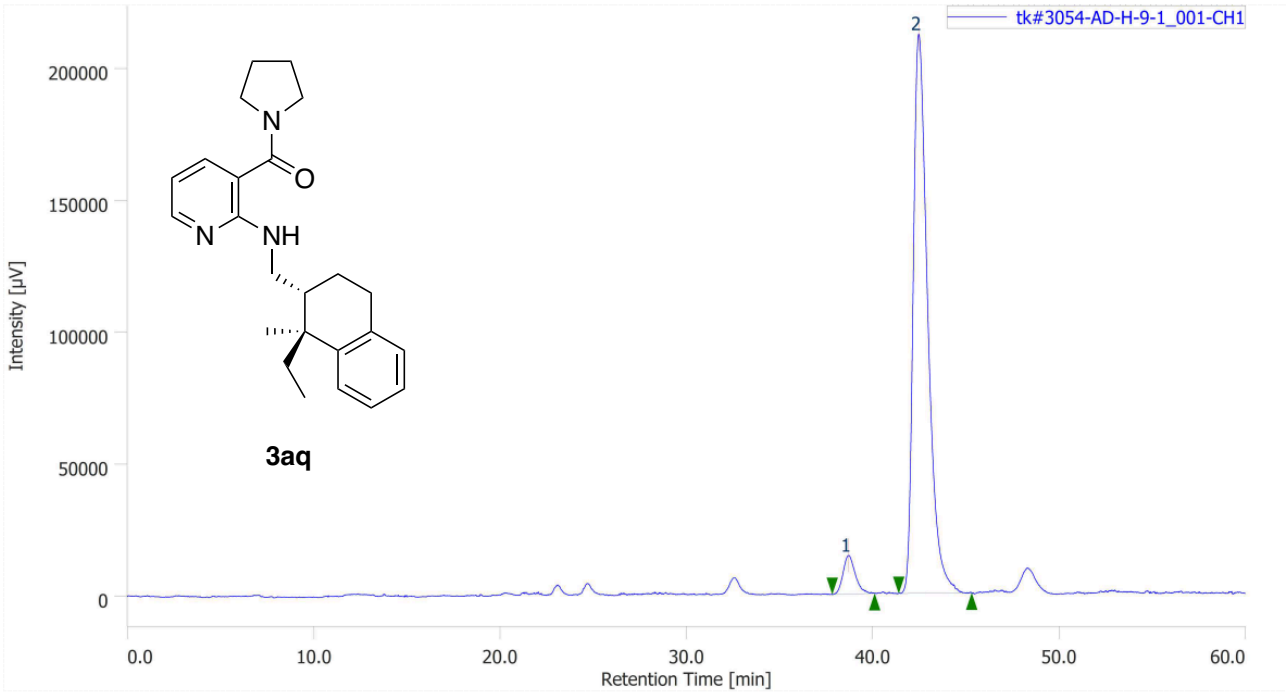
Peak Information

#	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	21.092	1058187	13.491	24923
2	25.417	6785486	86.509	113497



Peak Information

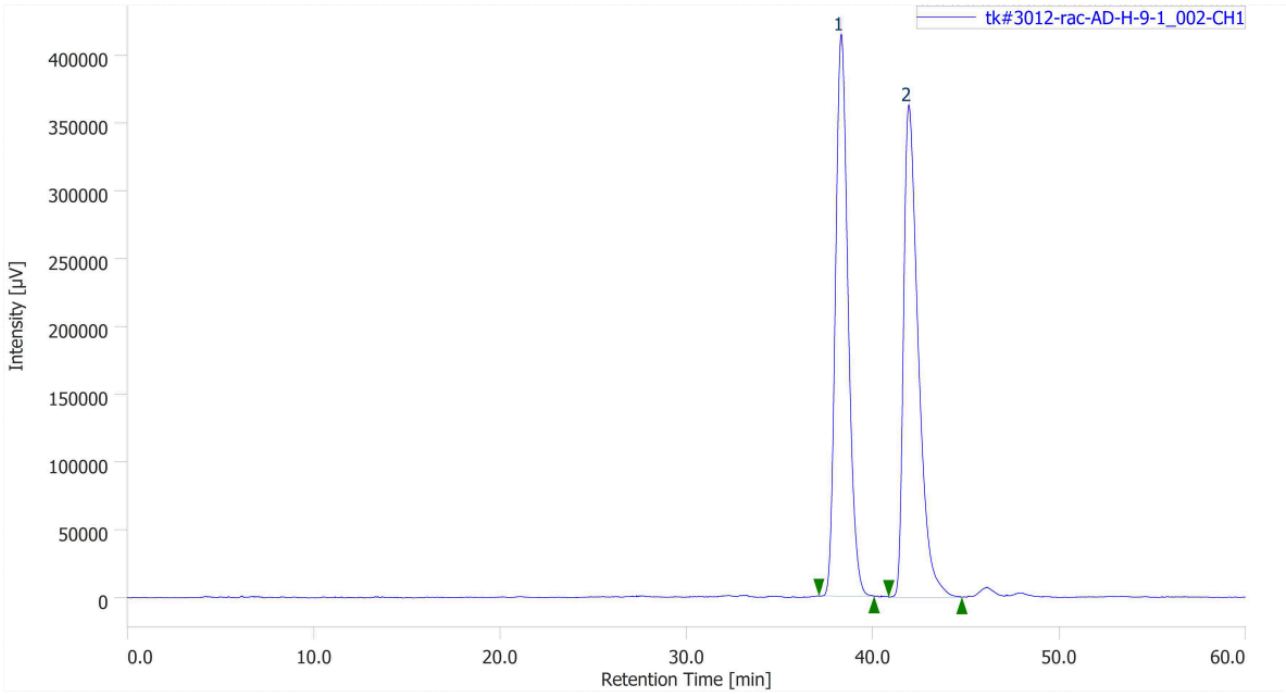
#	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	20.942	2235486	50.081	52333
2	25.592	2228231	49.919	40208



3aq

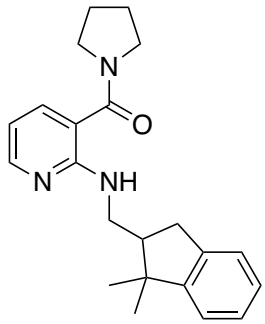
Peak Information

#	Peak Name	CH	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	Unknown	1	38.717	660589	5.456	14636
2	Unknown	1	42.467	11447060	94.544	212006

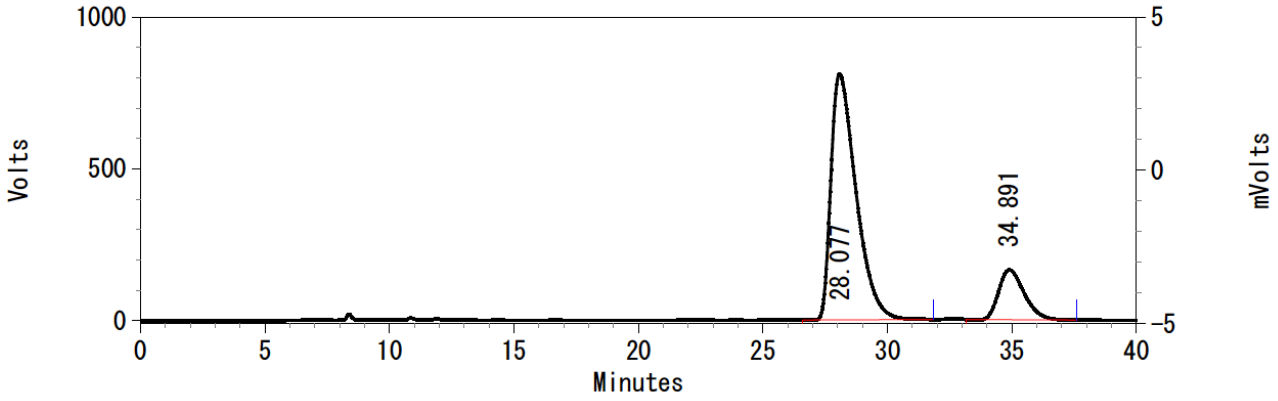


Peak Information

#	Peak Name	CH	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	Unknown	1	38.317	19502323	49.466	414479
2	Unknown	1	41.942	19923680	50.534	363055

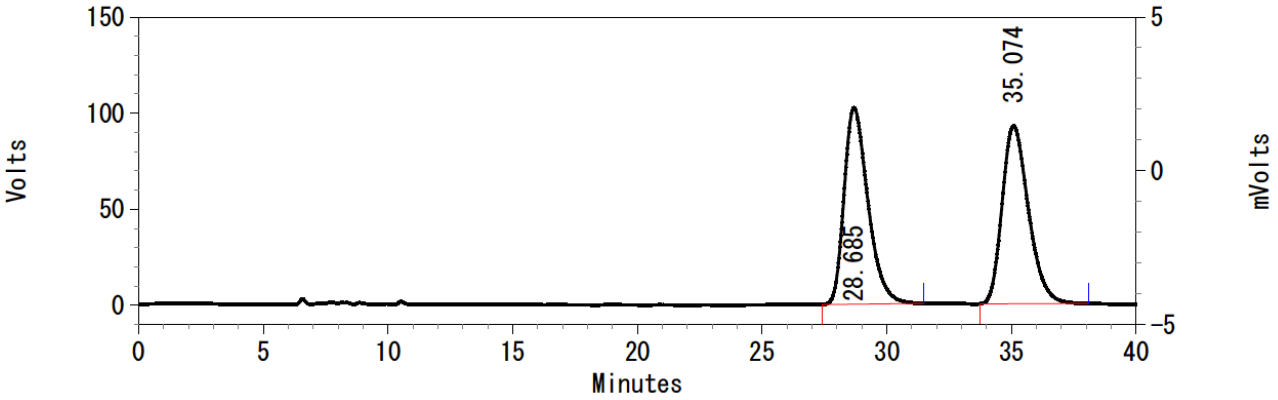


3ac



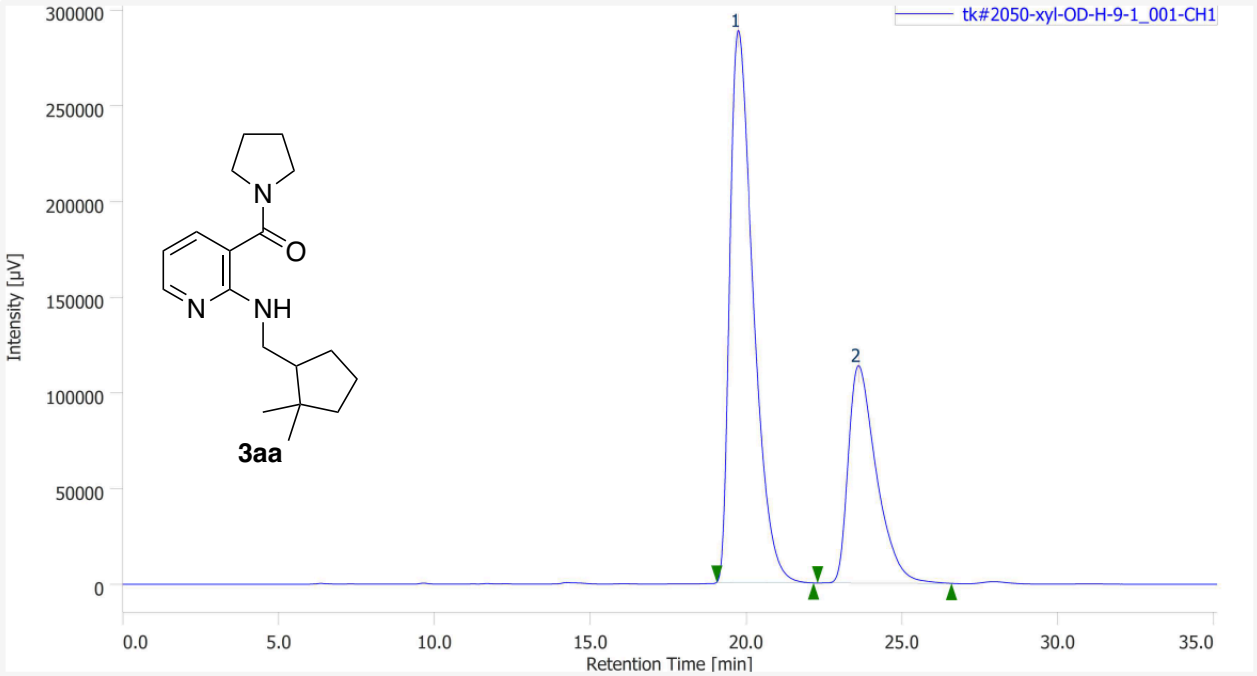
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	28.077	56852337	82.989	810575
2	34.891	11653279	17.011	164352
Totals		68505616	100.000	974927



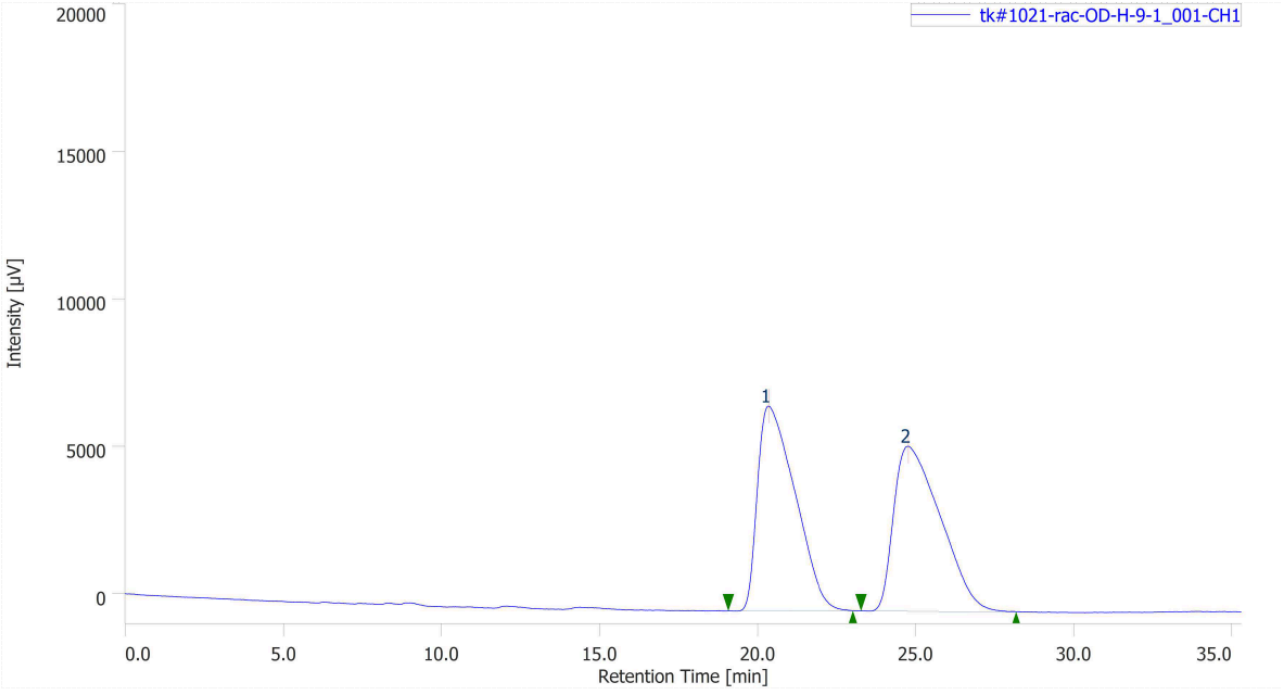
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	28.685	6814748	50.082	102214
2	35.074	6792341	49.918	92459
Totals		13607089	100.000	194673



Peak Information

Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
19.742	15081841	67.978	288388
23.600	7104417	32.022	113633



Peak Information

#	Peak Name	CH	Retention Time [min]	Area [μV·sec]	Area Percent	Height [μV]
1	Unknown	1	20.342	572931	50.027	6939
2	Unknown	1	24.767	572304	49.973	5585