Supporting Information for

Iridium-Catalyzed C3-Selective Asymmetric Allylation of 7-Azaindoles with Secondary Allylic Alcohols

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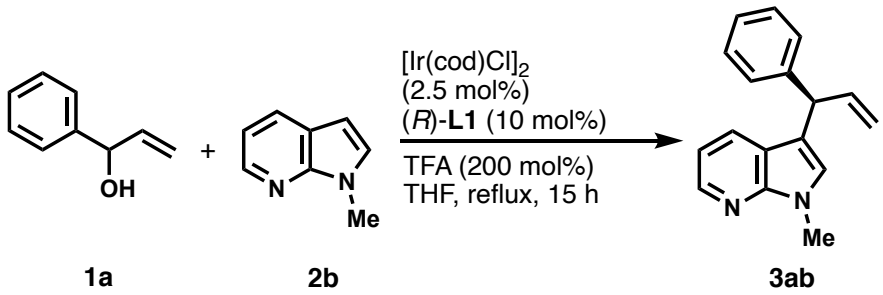
Contents of the Supporting Information

1. General Methods and Materials

All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried argon. ^{1}H, ^{13}C, and ^{19}F NMR spectra were measured on JEOL ECX 500II spectrometers (500 MHz for ^{1}H, 126 MHz for ^{13}C, 471 MHz for ^{19}F). Chemical shifts are reported in δ (ppm) referenced to the tetramethylsilane (δ 0.00) for ^{1}H NMR and the residual peaks of $CDCl_3$ (δ 77.00) for ^{13}C NMR. The following abbreviations are used; s: singlet, d: doublet, t: triplet, sext: sextet, m: multiplet, br: broad. High-resolution mass spectra were obtained with a JEOL JMS-700 Mstation. IR spectra were measured on a JASCO FT/IR-4100 spectrometer. Chiral High Performance Liquid Chromatography (HPLC) was performed on a Shimadzu system under the conditions given for each measurement. The products were purified by column chromatography on 63-210 mesh silica gel (Kanto Kagaku; Silica Gel 60N). Optical rotations were measured on a JASCO P-2200. Single-crystal X-ray diffraction was collected on a Rigaku RAXIS-RAPID imaging plate diffractometer with a graphite monochromatized Mo Kα (λ = 0.71069 Å) radiation. All solvents were dried and distilled before use by the usual procedures. $[Ir(cod)Cl]_2$, (*R*)-**L1** [CAS RN: 1265884-98-7], and (*R*)-**L2** [CAS RN: 2093047-92-6] were prepared as described in the literature.[1-3] 7-Azaindoles **2a** [CAS RN: 271-63-6], **2l** [CAS RN: 866546-07-8], and **2m** [CAS RN: 183208-35-7] were purchased and used as received. 7-Azaindoles **2b** [CAS RN: 27257-15-4], **2c** [CAS RN: 53277-42-2], **2d** [CAS RN: 183208-23-3], **2e** [CAS RN: 1260874-86-9], **2f** [CAS RN: 183208-22-2], **2g** [CAS RN: 74420-05-6], **2h** [CAS RN: 934568-25-9], **2i** [CAS RN: 1135437-92-1], **2j** [CAS RN: 113975-38-5], **2k** [CAS RN: 418795-13-8], and allylic alcohols **1a** [CAS RN: 4393-06-0], **1b** [CAS RN: 51410-44-7], **1c** [CAS RN: 58824-48-9], **1d** [CAS RN: 39627-62-8], **1e** [CAS RN: 1555908-83-2], **1f** [CAS RN: 58824-56-9], **1g** [CAS RN: 149946-79-2], **1h** [CAS RN: 196519-53-6], **1i** [CAS RN: 123232-63-3], **1j** [CAS RN: 76635-88-6], **1k** [CAS RN: 116914-87-5], **1l** [CAS RN: 393148-57-7] were prepared according to the reported procedures.[4-6]

2. General Procedure for the Asymmetric Allylation of 7-Azaindoles 2 with Allylic Alcohols 1 (Schemes 2 and 3)

Representative Procedure for the Reaction of Allylic Alcohol 1a with 7-Azaindole 2b

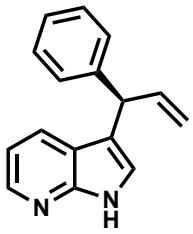


1a **2b** **3ab**

A mixture of $[Ir(cod)Cl]_2$ (8.9 mg, 0.132 mmol) and (*R*)-**L1** (26.3 mg, 0.0518 mmol) in THF (2.0 mL) was stirred at room temperature for 15 min. To the mixture was added **2b** (99.3 mg, 0.751 mmol), **1a** (67.6 mg, 0.504 mmol), and TFA (114 mg, 1.00 mmol), and the mixture was stirred under reflux for 15 h. After the solvent was removed on a rotary evaporator, the residue was subjected to column chromatography (silica gel, toluene/EtOAc = 98/2) to give compounds **3ab** (102.1 mg, 0.4111 mmol, 82% yield, 99% ee).

3. Characterization of 3

(*R*)-3-(1-Phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3aa)

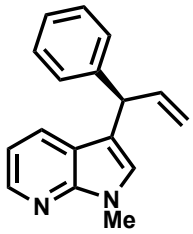


3aa

Compound **3aa** was prepared according to general procedure using **1a** (68.7 mg, 0.512 mmol) and **2a** (89.3 mg, 0.756 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (80/20) to afford **3aa** (28.1 mg, 0.1199 mmol, 23% yield, >99.5% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OJ-H column, 1.0 mL/min, hexane/2-propanol = 95/5, 230 nm, t_1 = 16.8 min (*minor*), t_2 = 23.7 min (*major*)); $[\alpha]^{24}_D$ –20 (*c* 0.50, $CHCl_3$) for >99.5% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 11.17 (s, 1H), 8.28 (dd, *J* = 4.6, 1.1 Hz, 1H), 7.67 (dd, *J* = 8.1, 1.5 Hz, 1H), 7.35–7.26 (m, 4H), 7.26–7.19 (m, 1H), 7.07 (s, 1H), 6.97 (dd, *J* = 8.0, 4.6 Hz, 1H), 6.34 (ddd, *J* = 17.2, 10.3, 7.0 Hz, 1H), 5.25–5.18 (m, 1H), 5.12–5.05 (m, 1H), 4.93 (d, *J* = 6.9 Hz, 1H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 149.4, 142.8, 142.3, 140.1, 128.4, 128.35, 128.33, 126.4, 123.3, 119.6, 116.5, 115.7, 115.2, 47.1; IR (neat, cm^{-1}) 3153, 3084, 3026, 2927, 2887, 2786, 1637,

1602, 1581, 1493, 1451, 1419, 1335, 1294, 1218, 1120, 995, 918, 807, 764, 702, 675, 651, 574, 528, 499; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{16}H_{15}N_2$ 235.1230; found 235.1233.

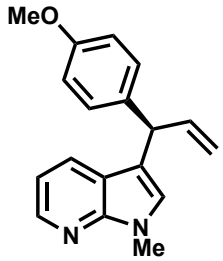
(*R*)-1-Methyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ab)



3ab

Compound **3ab** was prepared according to general procedure using **1a** (67.6 mg, 0.504 mmol) and **2b** (99.3 mg, 0.751 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (99/1) to afford **3ab** (102.1 mg, 0.4111 mmol, 82% yield, 99% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OJ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 20.1 min (*minor*), t_2 = 25.4 min (*major*)); $[\alpha]^{25}_D$ –6 (*c* 1.05, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29 (dd, *J* = 5.2, 1.4 Hz, 1H), 7.62 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.32–7.20 (m, 5H), 6.93 (dd, *J* = 8.0, 4.9 Hz, 1H), 6.85 (s, 1H), 6.31 (ddd, *J* = 17.2, 9.7, 7.2 Hz, 1H), 5.24–5.18 (m, 1H), 5.07 (dt, *J* = 16.6, 1.4 Hz, 1H), 4.91 (d, *J* = 7.5 Hz, 1H), 3.83 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.3, 142.8, 142.7, 140.1, 128.4, 128.3, 128.0, 127.1, 126.4, 119.5, 115.7, 115.3, 114.9, 47.0, 31.0; IR (neat, cm^{-1}) 3382, 3058, 3029, 2975, 2937, 2862, 1706, 1635, 1598, 1575, 1537, 1491, 1459, 1409, 1347, 1298, 1142, 997, 919, 796, 771, 702; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{17}N_2$ 249.1386; found 249.1382.

(*R*)-3-(1-(4-Methoxyphenyl)allyl)-1-methyl-1*H*-pyrrolo[2,3-*b*]pyridine (3bb)

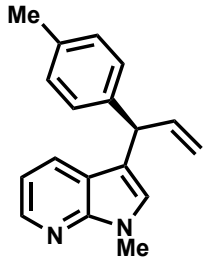


3bb

Compound **3bb** was prepared according to general procedure using **1b** (83.2 mg, 0.507 mmol) and **2b** (92.5 mg, 0.700 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3bb** (120.5 mg, 0.4329 mmol, 85% yield, 96% ee) as a yellow oil. The ee was measured by HPLC (Chiralcel OJ-H

column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 11.3 min (*major*), t_2 = 16.1 min (*minor*)); $[\alpha]^{25}_D$ –4 (*c* 1.01, $CHCl_3$) for 96% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 8.29 (d, *J* = 4.6 Hz, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.17 (d, *J* = 8.6 Hz, 2H), 6.93 (dd, *J* = 8.0, 5.0 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 2H), 6.84 (s, 1H), 6.29 (ddd, *J* = 17.2, 10.3, 7.2 Hz, 1H), 5.18 (d, *J* = 10.3 Hz, 1H), 5.05 (d, *J* = 17.2 Hz, 1H), 4.86 (d, *J* = 6.9 Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 158.2, 148.3, 142.8, 140.4, 134.9, 129.2, 128.0, 127.0, 119.5, 115.6, 115.4, 114.9, 113.8, 55.2, 46.2, 31.0; IR (neat, cm^{-1}) 3057, 3034, 3004, 2951, 2934, 2910, 2835, 1635, 1608, 1599, 1537, 1459, 1409, 1347, 1299, 1247, 1177, 1144, 1036, 919, 826, 807, 772, 643, 545; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{19}N_2O$ 279.1492; found 279.1489.

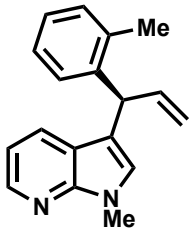
(*R*)-1-Methyl-3-(1-(*p*-tolyl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3cb)



3cb

Compound **3cb** was prepared according to general procedure using **1c** (74.0 mg, 0.499 mmol) and **2b** (99.5 mg, 0.753 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3cb** (111.2 mg, 0.4239 mmol, 85% yield, 98% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 8.5 min (*major*), t_2 = 10.0 min (*minor*)); $[\alpha]^{28}_D$ +5 (*c* 1.09, $CHCl_3$) for 98% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 8.29 (dd, *J* = 4.6, 1.2 Hz, 1H), 7.63 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.19–7.08 (m, 4H), 6.93 (dd, *J* = 8.0, 4.9 Hz, 1H), 6.84 (s, 1H), 6.30 (ddd, *J* = 17.2, 10.3, 7.2 Hz, 1H), 5.21–5.15 (m, 1H), 5.10–5.03 (m, 1H), 4.87 (d, *J* = 6.9 Hz, 1H), 3.82 (s, 3H), 2.32 (s, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 148.3, 142.8, 140.3, 139.8, 136.0, 129.1, 128.1, 128.0, 127.0, 119.6, 115.51, 115.46, 114.9, 46.7, 31.0, 21.0; IR (neat, cm^{-1}) 3051, 3007, 2971, 2922, 2854, 1635, 1597, 1571, 1537, 1511, 1490, 1459, 1409, 1347, 1298, 1143, 996, 918, 811, 771, 732, 640, 546, 523; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{19}N_2$ 263.1543; found 263.1544.

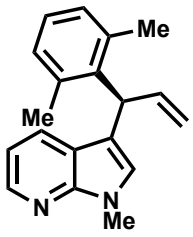
(*R*)-1-Methyl-3-(1-(*o*-tolyl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3db)



3db

Compound **3db** was prepared according to general procedure using **1d** (76.4 mg, 0.516 mmol) and **2b** (98.7 mg, 0.747 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (99/1) to afford **3db** (129.8 mg, 0.4947 mmol, 96% yield, >99.5% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 7.0 min (*major*), t_2 = 8.9 min (*minor*)); $[\alpha]^{27}_D$ +23 (*c* 0.54, $CHCl_3$) for >99.5% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29 (dd, *J* = 5.2, 1.4 Hz, 1H), 7.61 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.22–7.09 (m, 4H), 6.94 (dd, *J* = 8.0, 4.6 Hz, 1H), 6.75 (s, 1H), 6.27 (ddd, *J* = 17.2, 10.3, 6.7 Hz, 1H), 5.21 (dt, *J* = 9.8, 1.4 Hz, 1H), 5.09 (d, *J* = 5.8 Hz, 1H), 4.97 (dt, *J* = 17.2, 1.7 Hz, 1H), 3.82 (s, 3H), 2.33 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.3, 142.8, 140.7, 139.5, 136.0, 130.4, 128.0, 127.8, 127.5, 126.4, 126.0, 119.8, 115.8, 114.9, 114.8, 42.8, 31.0, 19.5; IR (neat, cm^{-1}) 3059, 3014, 2979, 2943, 1635, 1598, 1571, 1537, 1488, 1459, 1409, 1347, 1298, 1141, 998, 919, 797, 771, 749, 733, 545; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{19}N_2$ 263.1543; found 263.1542.

(*R*)-3-(1-(2,6-Dimethylphenyl)allyl)-1-methyl-1*H*-pyrrolo[2,3-*b*]pyridine (3eb)

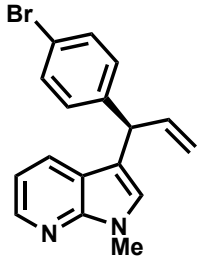


3eb

Compound **3eb** was prepared according to general procedure using **1e** (79.9 mg, 0.493 mmol) and **2b** (106.6 mg, 0.8066 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3eb** (66.6 mg, 0.2410 mmol, 49% yield, >99.5% ee) as a white solid (mp 125.1-126.1 °C). The ee was measured by HPLC (Chiralcel OJ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 5.7 min (*major*), t_2 = 6.5 min (*minor*)); $[\alpha]^{25}_D$ –76 (*c* 1.01, $CHCl_3$) for >99.5% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.30–7.24 (m, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 7.4 Hz, 2H), 6.87–6.82 (m, 2H), 6.48 (ddd, *J* = 16.9, 9.9, 6.7 Hz, 1H), 5.35

(d, J = 6.9 Hz, 1H), 5.24 (d, J = 10.3 Hz, 1H), 5.18–5.10 (m, 1H), 3.84 (s, 3H), 2.23 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.2, 142.7, 138.6, 137.9, 137.0, 129.2, 127.9, 126.5, 126.2, 119.7, 116.1, 114.83, 114.76, 42.6, 31.1, 21.3; IR (KBr, cm^{-1}) 3441, 3055, 3014, 2935, 1536, 1489, 1460, 1407, 1343, 1295, 1215, 1142, 1017, 925, 810, 794, 776; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{19}H_{21}N_2$ 277.1699; found 277.1703.

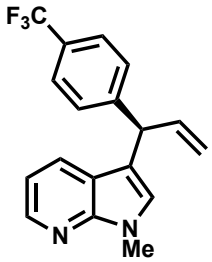
(*R*)-3-(1-(4-Bromophenyl)allyl)-1-methyl-1*H*-pyrrolo[2,3-*b*]pyridine (3fb)



3fb

Compound **3fb** was prepared according to general procedure using **1f** (107.3 mg, 0.504 mmol) and **2b** (98.7 mg, 0.747 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3fb** (103.1 mg, 0.3151 mmol, 63% yield, 99% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 9.6 min (*major*), t_2 = 10.9 min (*minor*)); $[\alpha]^{28}_D$ –10 (*c* 0.50, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.30 (dd, J = 5.2, 1.4 Hz, 1H), 7.58 (dd, J = 8.0, 1.7 Hz, 1H), 7.44–7.39 (m, 2H), 7.15–7.10 (m, 2H), 6.94 (dd, J = 8.0, 4.6 Hz, 1H), 6.85 (s, 1H), 6.26 (ddd, J = 17.2, 10.3, 7.0 Hz, 1H), 5.22 (d, J = 10.3 Hz, 1H), 5.06 (dt, J = 16.6, 1.4 Hz, 1H), 4.86 (d, J = 6.9 Hz, 1H), 3.84 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.3, 143.0, 141.8, 139.5, 131.5, 130.1, 127.8, 127.1, 120.3, 119.3, 116.2, 115.0, 114.7, 46.4, 31.1; IR (neat, cm^{-1}) 3080, 3057, 3007, 2979, 2935, 2858, 1636, 1597, 1572, 1537, 1487, 1459, 1408, 1347, 1298, 1143, 1072, 1011, 921, 802, 771, 546; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}BrN_2$ 327.0491; found 327.0495.

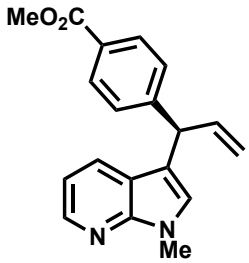
(*R*)-1-Methyl-3-(1-(4-(trifluoromethyl)phenyl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3gb)



3gb

Compound **3gb** was prepared according to general procedure using **1g** (109.2 mg, 0.540 mmol) and **2b** (100.8 mg, 0.7627 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3gb** (68.2 mg, 0.2156 mmol, 40% yield, 99% ee) as a yellow oil. The ee was measured by HPLC (Chiralcel OJ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 9.4 min (*minor*), t_2 = 10.5 min (*major*)); $[\alpha]^{25}_D$ –15 (*c* 0.97, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.59 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.56 (d, *J* = 8.1 Hz, 1H), 7.38 (d, *J* = 8.1 Hz, 2H), 6.95 (dd, *J* = 8.0, 4.6 Hz, 1H), 6.88 (s, 1H), 6.29 (ddd, *J* = 17.2, 9.8, 7.2 Hz, 1H), 5.28–5.23 (m, 1H), 5.13–5.05 (m, 1H), 4.97 (d, *J* = 7.5 Hz, 1H), 3.85 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.3, 146.9, 143.1, 139.2, 128.8 (q, $J_{C\text{-}F}$ = 32.2 Hz), 128.7, 127.8, 127.2, 125.4 (q, $J_{C\text{-}F}$ = 3.6 Hz), 124.2 (q, $J_{C\text{-}F}$ = 270.7 Hz), 119.3, 116.5, 115.1, 114.4, 46.8, 31.1; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.2; IR (neat, cm^{-1}) 3059, 3010, 2979, 2937, 2862, 1721, 1636, 1617, 1598, 1537, 1491, 1460, 1411, 1326, 1299, 1164, 1123, 1067, 1017, 924, 832, 805, 772, 602; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{16}F_3N_2$ 317.1260; found 317.1261.

Methyl (*R*)-4-(1-(1-methyl-1*H*-pyrrolo[2,3-b]pyridin-3-yl)allyl)benzoate (3hb)

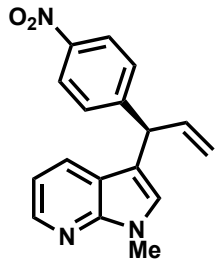


3hb

Compound **3hb** was prepared according to general procedure using **1h** (95.9 mg, 0.499 mmol) and **2b** (104.0 mg, 0.7869 mmol). The crude reaction mixture was purified by column chromatography using heaxne/EtOAc (3/1) to afford **3hb** (82.6 mg, 0.2696 mmol, 54% yield, 99% ee) as a yellow oil. The ee was measured by HPLC (Chiralcel OJ-3 column, 1.0 mL/min, hexane/2-propanol = 96/4, 230 nm, t_1 = 24.9 min (*minor*), t_2 = 28.2 min (*major*)); $[\alpha]^{25}_D$ –16 (*c* 0.99, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.30 (dd, *J* = 4.6, 1.7 Hz, 1H), 8.01–7.96 (m, 2H), 7.57 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.36–7.32 (m, 2H), 6.94 (dd, *J* = 8.1, 4.9 Hz, 1H), 6.87 (s, 1H), 6.30 (ddd, *J* = 17.2, 9.7, 7.2 Hz, 1H), 5.27–5.22 (m, 1H), 5.08 (dt, *J* = 17.2, 1.3 Hz, 1H), 4.96 (d, *J* = 6.9 Hz, 1H), 3.90 (s, 3H), 3.85 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.0, 148.3, 148.1, 143.0, 139.2, 129.8, 128.5, 128.4, 127.8, 127.2, 119.3, 116.4, 115.1, 114.5, 52.0, 47.0, 31.1; IR (neat, cm^{-1}) 3423, 3057,

3006, 2951, 2857, 1721, 1608, 1572, 1536, 1491, 1460, 1436, 1410, 1347, 1280, 1179, 1112, 1019, 922, 805, 767, 727, 710, 602, 579, 545; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{19}H_{19}N_2O_2$ 307.1441; found 307.1443.

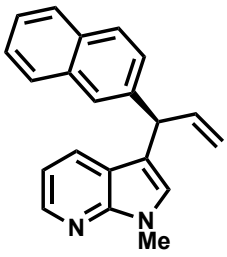
(*R*)-1-Methyl-3-(1-(4-nitrophenyl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ib)



3ib

Compound **3ib** was prepared according to general procedure using **1i** (90.1 mg, 0.503 mmol) and **2b** (99.4 mg, 0.752 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (3/1) to afford **3ib** (38.6 mg, 0.132 mmol, 26% yield, >99.5% ee) as a yellow oil. The ee was measured by HPLC (Chiralpak AD-3 column, 1.0 mL/min, hexane/2-propanol = 95/5, 230 nm, t_1 = 20.4 min (*major*), t_2 = 21.5 min (*minor*)); $[\alpha]^{24}_D$ –24 (*c* 0.17, $CHCl_3$) for >99.5% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 4.6, 1.7 Hz, 1H), 8.19–8.13 (m, 2H), 7.58–7.53 (m, 1H), 7.42 (d, *J* = 8.6 Hz, 2H), 6.96 (dd, *J* = 8.1, 4.6 Hz, 1H), 6.92 (s, 1H), 6.30 (ddd, *J* = 17.2, 10.3, 6.9 Hz, 1H), 5.29 (d, *J* = 9.8 Hz, 1H), 5.10 (d, *J* = 17.2 Hz, 1H), 5.03 (d, *J* = 6.9 Hz, 1H), 3.87 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 150.4, 148.3, 146.7, 143.3, 138.6, 129.2, 127.6, 127.2, 119.1, 117.1, 115.3, 113.7, 46.8, 31.1; IR (neat, cm^{-1}) 3105, 3077, 3058, 2926, 2855, 1720, 1657, 1637, 1598, 1519, 1491, 1460, 1409, 1345, 1299, 1142, 1123, 1109, 925, 853, 841, 805, 772, 753, 723, 705, 545; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}N_3O_2$ 294.1237; found 294.1237.

(*R*)-1-Methyl-3-(1-(naphthalen-2-yl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3jb)

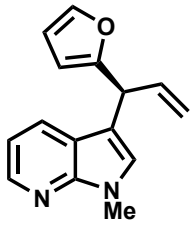


3jb

Compound **3jb** was prepared according to general procedure using **1j** (93.7 mg,

0.509 mmol) and **2b** (98.0 mg, 0.741 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (99/1) to afford **3jb** (143.1 mg, 0.4796 mmol, 94% yield, 99% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 11.2 min (*major*), t_2 = 14.6 min (*minor*)); $[\alpha]^{27}_D$ +3 (*c* 0.59, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.84-7.75 (m, 3H), 7.71 (s, 1H), 7.63 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.48-7.41 (m, 2H), 7.39 (dd, *J* = 8.0, 1.7 Hz, 1H), 6.91 (dd, *J* = 7.5, 4.9 Hz, 1H), 6.88 (s, 1H), 6.40 (ddd, *J* = 17.2, 9.8, 7.2 Hz, 1H), 5.29-5.23 (m, 1H), 5.16-5.05 (m, 2H), 3.84 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.3, 142.9, 140.3, 139.9, 133.5, 132.3, 128.0, 127.8, 127.6, 127.3, 127.1, 126.5, 126.0, 125.5, 119.6, 116.1, 115.2, 115.0, 47.1, 31.1,; IR (neat, cm^{-1}) 3055, 3007, 2971, 2938, 1632, 1598, 1571, 1537, 1490, 1459, 1409, 1347, 1298, 1143, 996, 919, 858, 755, 478; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{21}H_{19}N_2$ 299.1543; found 299.1546.

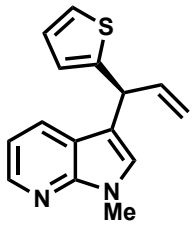
(*S*)-3-(1-(Furan-2-yl)allyl)-1-methyl-1*H*-pyrrolo[2,3-*b*]pyridine (3kb)



3kb

Compound **3kb** was prepared according to general procedure using **1k** (65.0 mg, 0.524 mmol) and **2b** (104.0 mg, 0.7869 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3kb** (83.0 mg, 0.348 mmol, 66% yield, 83% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 8.9 min (*minor*), t_2 = 9.8 min (*major*)); $[\alpha]^{21}_D$ +4 (*c* 1.00, $CHCl_3$) for 83% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 4.6, 1.1 Hz, 1H), 7.74 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.37 (s, 1H), 7.03–6.96 (m, 2H), 6.32 (dd, *J* = 3.5, 2.0 Hz, 1H), 6.26 (ddd, *J* = 17.2, 9.7, 7.0 Hz, 1H), 6.09 (d, *J* = 2.9 Hz, 1H), 5.21 (d, *J* = 9.7 Hz, 1H), 5.17–5.10 (m, 1H), 4.97 (d, *J* = 6.9 Hz, 1H), 3.85 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 155.8, 148.1, 142.9, 141.6, 137.4, 127.8, 126.9, 119.3, 116.1, 115.1, 112.5, 110.2, 106.2, 40.8, 31.1; IR (neat, cm^{-1}) 3116, 3058, 3009, 2975, 2941, 2862, 1723, 1639, 1598, 1538, 1460, 1409, 1348, 1299, 1146, 1010, 921, 799, 770, 599, 546; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{15}H_{15}N_2O$ 239.1179; found 239.1180.

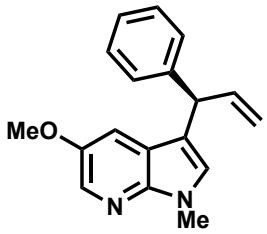
(*S*)-1-Methyl-3-(1-(thiophen-2-yl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3lb)



3lb

Compound **3lb** was prepared according to general procedure using **1l** (71.3 mg, 0.509 mmol) and **2b** (101.2 mg, 0.7657 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3lb** (107.5 mg, 0.4226 mmol, 83% yield, 90% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OJ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 26.6 min (*major*), t_2 = 33.1 min (*minor*)); $[\alpha]^{28}_D$ –17 (*c* 0.53, $CHCl_3$) for 90% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 4.6, 1.1 Hz, 1H), 7.72 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.18 (dd, *J* = 5.2, 1.1 Hz, 1H), 7.01–6.93 (m, 3H), 6.87 (d, *J* = 3.5 Hz, 1H), 6.33 (ddd, *J* = 16.6, 10.0, 7.2 Hz, 1H), 5.24–5.13 (m, 3H), 3.85 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.2, 146.9, 143.0, 139.6, 127.9, 126.9, 124.7, 126.6, 124.7, 124.0, 119.2, 115.7, 115.1, 115.0, 42.2, 31.1; IR (neat, cm^{-1}) 3059, 3007, 2975, 2933, 2858, 1636, 1598, 1571, 1537, 1490, 1459, 1436, 1408, 1347, 1299, 1227, 1141, 1037, 992, 922, 797, 771, 699, 545; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{15}H_{15}N_2S$ 255.0951; found 255.0951.

(*R*)-5-Methoxy-1-methyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ad)

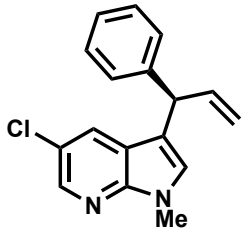


3ad

Compound **3ad** was prepared according to general procedure using **1a** (67.3 mg, 0.502 mmol) and **2d** (123.5 mg, 0.7614 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3ad** (113.4 mg, 0.4074 mmol, 81% yield, 83% ee) as a brown oil. The ee was measured by HPLC (Chiralpak AD-3 column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 15.7 min (*minor*), t_2 = 16.5 min (*major*)); $[\alpha]^{26}_D$ +11 (*c* 0.53, $CHCl_3$) for 83% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (d, *J* = 2.9 Hz, 1H), 7.34-7.20 (m, 5H), 7.09 (d, *J* = 2.5 Hz, 1H), 6.82 (s, 1H), 6.30 (ddd, *J* = 17.2, 9.7, 7.2 Hz), 5.21 (d, *J* = 10.3 Hz, 1H), 5.08 (d, *J* = 17.2 Hz, 1H), 4.86 (d, *J* = 7.5

Hz, 1H), 3.80 (s, 3H), 3.76 (s, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 150.6, 144.0, 142.7, 140.0, 133.2, 128.4, 128.3, 128.0, 126.5, 119.2, 115.7, 114.5, 111.0, 56.4, 47.1, 31.2; IR (neat, cm^{-1}) 3080, 3060, 3026, 3002, 2937, 2834, 1636, 1601, 1535, 1492, 1452, 1407, 1359, 1291, 1261, 1216, 1174, 1140, 1036, 917, 702; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{19}N_2O$ 279.1492; found 279.1494.

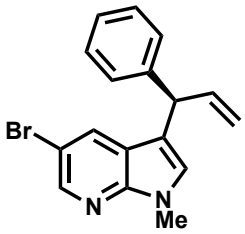
(*R*)-5-chloro-1-methyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ae)



3ae

Compound **3ae** was prepared according to general procedure using **1a** (67.1 mg, 0.500 mmol) and **2e** (125.4 mg, 0.7527 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3ae** (123.7 mg, 0.4375 mmol, 87% yield, 94% ee) as a yellow oil. The ee was measured by HPLC (Chiralcel OZ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 254 nm, t_1 = 4.9 min (*major*), t_2 = 5.6 min (*minor*)); $[\alpha]^{23}_D$ –3 (*c* 0.97, $CHCl_3$) for 94% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 8.22 (d, *J* = 2.3 Hz, 1H), 7.59 (d, *J* = 2.3 Hz, 1H), 7.35–7.29 (m, 2H), 7.27–7.22 (m, 3H), 6,87 (s, 1H), 6.28 (ddd, *J* = 17.5, 10.2, 7.2 Hz, 1H), 5.22 (d, *J* = 9.8 Hz, 1H), 5.07 (dt, *J* = 17.2, 1.4 Hz, 1H), 4.85 (d, *J* = 6.9 Hz, 1H), 3.80 (s, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 146.6, 142.3, 141.4, 139.7, 128.8, 128.5, 128.2, 127.1, 126.7, 123.0, 120.1, 116.1, 115.0, 46.9, 31.2; IR (neat, cm^{-1}) 3081, 3060, 3027, 2976, 2936, 2872, 1636, 1599, 1559, 1532, 1481, 1407, 1348, 1279, 1254, 1222, 1143, 1092, 921, 885, 763, 750, 723, 701, 607, 578; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}ClN_2$ 283.0997; found 283.0997.

(*R*)-5-Bromo-1-methyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3af)

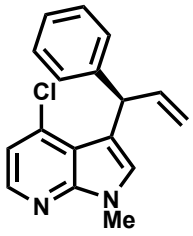


3af

Compound **3af** was prepared according to general procedure using **1a** (68.1 mg, 0.508 mmol) and **2f** (158.4 mg, 0.7505 mmol). The crude reaction mixture was purified

by column chromatography using toluene/EtOAc (99/1) to afford **3af** (125.6 mg, 0.3838 mmol, 76% yield, 97% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OZ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 5.3 min (*major*), t_2 = 6.0 min (*minor*)); $[\alpha]^{25}_D$ +18 (*c* 0.50, $CHCl_3$) for 97% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.30 (d, *J* = 1.7 Hz, 1H), 7.74 (d, *J* = 2.0 Hz, 1H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.27–7.22 (m, 3H), 6.84 (s, 1H), 6.28 (ddd, *J* = 17.2, 10.3, 7.0 Hz, 1H), 5.22 (d, *J* = 9.8 Hz, 1H), 5.06 (d, *J* = 17.2 Hz, 1H), 4.85 (d, *J* = 6.9 Hz, 1H), 3.80 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 146.6, 143.3, 142.3, 139.7, 130.0, 128.6, 128.5, 128.2, 126.7, 121.0, 116.1, 115.0, 111.0, 46.8, 31.2; IR (neat, cm^{-1}) 3076, 3060, 3027, 3004, 2979, 2938, 2872, 1636, 1598, 1557, 1531, 1481, 1451, 1408, 1349, 1278, 1255, 1222, 1142, 1079, 995, 920, 882, 815, 762, 701; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}BrN_2$ 327.0491; found 327.0488.

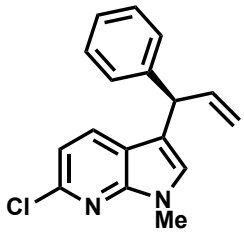
(*R*)-4-Chloro-1-methyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ag)



3ag

Compound **3ag** was prepared according to general procedure using **1a** (68.5 mg, 0.511 mmol) and **2g** (121.2 mg, 0.7275 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (9/1) to afford **3ag** (71.4 mg, 0.253 mmol, 49% yield, 97% ee) as a white solid (mp 69.0-69.3 °C). The ee was measured by HPLC (Chiralpak AD-3 column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 7.7 min (*minor*), t_2 = 9.1 min (*major*)); $[\alpha]^{25}_D$ –6 (*c* 0.99, $CHCl_3$) for 97% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.15 (d, *J* = 5.2 Hz, 1H), 7.33–7.27 (m, 2H), 7.26–7.19 (m, 3H), 6.98 (d, *J* = 5.2 Hz, 1H), 6.87 (s, 1H), 6.33 (ddd, *J* = 17.2, 10.3, 6.4 Hz, 1H), 5.46 (d, *J* = 6.3 Hz, 1H), 5.21 (d, *J* = 10.3 Hz, 1H), 4.88 (d, *J* = 17.2 Hz, 1H), 3.83 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 149.1, 143.0, 140.9, 136.2, 128.7, 128.2, 126.3, 117.1, 116.4, 115.9, 115.6, 46.0, 31.5; IR (KBr, cm^{-1}) 3444, 3085, 3029, 3005, 2941, 2896, 1637, 1590, 1551, 1523, 1487, 1453, 1408, 1348, 1312, 1295, 1150, 1005, 991, 914, 878, 819, 765, 704, 662, 631, 604, 594, 551; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}ClN_2$ 283.0997; found 283.1001.

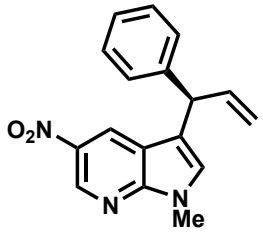
(*R*)-6-Chloro-1-methyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ah)



3ah

Compound **3ah** was prepared according to general procedure using **1a** (67.0 mg, 0.499 mmol) and **2h** (126.1 mg, 0.7569 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3ah** (116.3 mg, 0.4113 mmol, 82% yield, 80% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 12.3 min (*major*), t_2 = 13.0 min (*minor*)); $[\alpha]^{26}_D$ –3 (*c* 1.06, $CHCl_3$) for 80% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.34–7.28 (m, 2H), 7.27–7.21 (m, 3H), 6.93 (d, *J* = 8.1 Hz, 1H), 6.82 (s, 1H), 6.28 (ddd, *J* = 17.2, 9.8, 7.2 Hz, 1H), 5.21 (dt, *J* = 10.3, 1.3 Hz, 1H), 5.06 (dt, *J* = 16.6, 1.4 Hz, 1H), 4.86 (d, *J* = 6.9 Hz, 1H), 3.80 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 147.3, 144.5, 142.4, 139.8, 130.3, 128.5, 128.2, 127.3, 126.6, 118.0, 115.94, 115.87, 115.0, 47.0, 31.2; IR (neat, cm^{-1}) 3081, 3059, 3026, 2978, 2939, 2872, 1636, 1599, 1563, 1533, 1490, 1438, 1410, 1347, 1306, 1119, 998, 918, 876, 812, 759, 732, 701, 640, 589; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}ClN_2$ 283.0997; found 283.0997.

(*R*)-1-Methyl-5-nitro-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-b]pyridine (3ai)

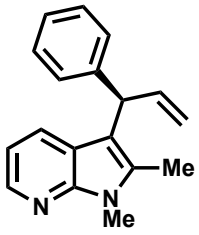


3ai

Compound **3ai** was prepared according to general procedure using **1a** (70.7 mg, 0.527 mmol) and **2i** (132.8 mg, 0.7496 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (95/5) to afford **3ai** (19.7 mg, 0.0672 mmol, 13% yield, 80% ee) as a yellow oil. The ee was measured by HPLC (Chiralpak AS-3 column, 1.0 mL/min, hexane/2-propanol = 95/5, 230 nm, t_1 = 8.9 min (*major*), t_2 = 10.0 min (*minor*)); $[\alpha]^{25}_D$ +31 (*c* 0.60, $CHCl_3$) for 80% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.18 (d, *J* = 2.3 Hz, 1H), 8.47 (d, *J* = 2.3 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 3H), 7.00 (s, 1H), 6.31 (ddd, *J* = 16.6, 10.0, 7.0 Hz, 1H), 5.27 (d, *J* = 10.9 Hz, 1H), 5.10 (d, *J* = 16.6 Hz, 1H), 4.94 (d, *J* = 6.9 Hz, 1H), 3.89 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$)

δ 149.9, 141.7, 139.6, 139.2, 138.5, 130.6, 128.7, 128.2, 127.0, 124.3, 118.5, 118.4, 116.6, 46.7, 31.6; IR (neat, cm^{-1}) 3079, 3023, 2978, 2938, 2855, 1855, 1712, 1637, 1560, 1575, 1538, 1511, 1492, 1447, 1414, 1332, 1298, 1214, 1151, 1105, 992, 920, 822, 778, 749, 703, 609; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}N_3O_2$ 294.1237; found 294.1237.

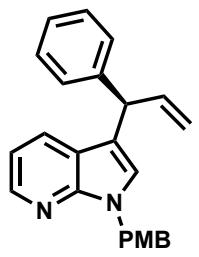
(*R*)-1,2-Dimethyl-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-b]pyridine (3aj)



3aj

Compound **3aj** was prepared according to general procedure using **1a** (61.0 mg, 0.455 mmol) and **2j** (107.2 mg, 0.7333 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (8/2) to afford **3aj** (90.7 mg, 0.346 mmol, 76% yield, 96% ee) as a yellow oil. The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 9.0 min (*major*), t_2 = 11.0 min (*minor*)); $[\alpha]^{25}_D$ –3 (*c* 0.91, $CHCl_3$) for 96% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 8.19 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.54 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.27 (d, *J* = 4.6 Hz, 4H), 7.19 (sextet, *J* = 4.2 Hz, 1H), 6.87 (dd, *J* = 7.5, 4.6 Hz, 1H), 6.42 (ddd, *J* = 17.2, 10.3, 6.9 Hz, 1H), 5.21 (dt, *J* = 10.3, 1.6 Hz, 1H), 5.04 (dt, *J* = 17.2, 1.7 Hz, 1H), 4.98 (d, *J* = 6.9 Hz, 1H), 3.79 (s, 3H), 2.38 (s, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 148.0, 142.9, 141.3, 139.8, 134.1, 128.2, 128.1, 127.0, 126.1, 119.6, 115.7, 115.0, 110.3, 45.8, 28.0, 10.7; IR (neat, cm^{-1}) 3079, 3058, 3026, 2974, 2935, 2859, 1718, 1659, 1635, 1597, 1573, 1548, 1490, 1459, 1416, 1337, 1307, 1173, 1127, 995, 917, 794, 771, 722, 701, 569; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{19}N_2$ 263.1543; found 263.1543.

(*R*)-1-(4-Methoxybenzyl)-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3ak)

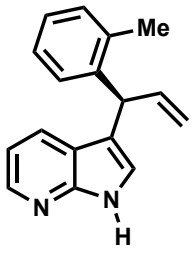


3ak

Compound **3ak** was prepared according to general procedure using **1a** (67.8 mg,

0.505 mmol) and **2k** (178.8 mg, 0.7503 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (98/2) to afford **3ak** (129.2 mg, 0.3645 mmol, 72% yield, 98% ee) as a brown oil. The ee was measured by HPLC (Chiralcel OZ-H column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 20.3 min (*major*), t_2 = 22.5 min (*minor*)); $[\alpha]^{26}_D$ –14 (*c* 0.55, $CHCl_3$) for 97% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 8.30 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.59 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.31-7.26 (m, 2H), 7.25-7.12 (m, 5H), 6.94 (dd, *J* = 7.5, 4.9 Hz, 1H), 6.90 (s, 1H), 6.83 (d, *J* = 9.2 Hz, 2H), 6.33-6.23 (m, 1H), 5.41 (d, *J* = 14.9 Hz, 1H), 5.35 (d, *J* = 15.5 Hz, 1H), 5.20-5.15 (m, 1H), 5.08-5.00 (m, 1H), 4.89 (d, *J* = 6.9 Hz, 1H), 3.77 (s, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 159.0, 148.2, 143.0, 142.7, 140.1, 130.0, 128.7, 128.4, 128.3, 128.1, 126.4, 125.8, 119.5, 115.9, 115.7, 115.2, 114.0, 55.2, 47.2, 47.1; IR (neat, cm^{-1}) 3059, 3027, 3000, 2956, 2932, 2836, 1725, 1636, 1612, 1597, 1513, 1487, 1451, 1358, 1303, 1249, 1175, 1035, 919, 819, 756, 702; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{24}H_{23}N_2O$ 355.1805; found 355.1805.

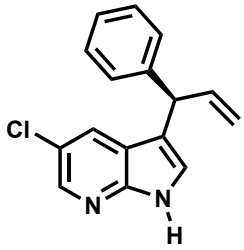
(*R*)-3-(1-(*o*-Tolyl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3da)



3da

Compound **3da** was prepared according to general procedure using **1d** (72.4 mg, 0.489 mmol) and **2a** (87.7 mg, 0.742 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (2/1) to afford **3da** (58.0 mg, 0.234 mmol, 48% yield, >99.5% ee) as a pale yellow solid (mp 127.1-127.7 °C). The ee was measured by HPLC (Chiralcel OJ-H column, 1.0 mL/min, hexane/2-propanol = 95/5, 230 nm, t_1 = 14.3 min (*minor*), t_2 = 15.9 min (*major*)); $[\alpha]^{25}_D$ +21 (*c* 1.05, $CHCl_3$) for >99.5% ee. ^1H NMR (500 MHz, $CDCl_3$) δ 10.74 (s, 1H), 8.27 (dd, *J* = 4.6, 1.5 Hz, 1H), 7.69-7.62 (m, 1H), 7.22-7.10 (m, 4H), 7.01-6.94 (m, 2H), 6.30 (ddd, *J* = 16.6, 10.0, 6.6 Hz, 1H), 5.23 (d, *J* = 10.3 Hz, 1H), 5.11 (d, *J* = 6.3 Hz, 1H), 4.98 (d, *J* = 17.2 Hz, 1H), 2.34 (s, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 149.3, 142.5, 140.6, 139.4, 136.1, 130.4, 128.13, 128.10, 126.4, 126.0, 123.5, 119.7, 116.1, 116.0, 115.3, 42.9, 19.5; IR (KBr, cm^{-1}) 3083, 2891, 1636, 1606, 1581, 1488, 1458, 1418, 1335, 1294, 1256, 1120, 997, 918, 897, 795, 767, 753, 726, 647, 505; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{17}N_2$ 249.1386; found 249.1387.

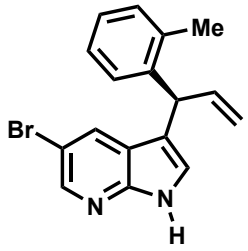
(*R*)-5-Chloro-3-(1-phenylallyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3al)



3al

Compound **3al** was prepared according to general procedure using **1a** (67.4 mg, 0.502 mmol) and **2l** (114.0 mg, 0.7471 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) followed by hexane/acetone (7/3) to afford **3al** (43.9 mg, 0.163 mmol, 33% yield, 99% ee) as a yellow oil. The ee was measured by HPLC (Chiralcel OJ-3 column, 1.0 mL/min, hexane/2-propanol = 95/5, 230 nm, t_1 = 10.9 min (*minor*), t_2 = 13.4 min (*major*)); $[\alpha]^{26}_D$ +23 (*c* 0.41, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.65 (s, 1H), 8.22 (d, *J* = 2.3 Hz, 1H), 7.64 (d, *J* = 2.3 Hz, 1H), 7.35-7.29 (m, 2H), 7.29-7.22 (m, 3H), 7.07 (d, *J* = 1.2 Hz, 1H), 6.31 (ddd, *J* = 17.2, 10.3, 7.0 Hz, 1H), 5.27-5.21 (m, 1H), 5.12-5.04 (m, 1H), 4.88 (d, *J* = 7.5 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 147.5, 142.2, 141.2, 139.6, 128.6, 128.3, 127.6, 126.7, 124.9, 123.3, 120.3, 116.6, 116.2, 46.9; IR (neat, cm^{-1}) 3154, 3029, 2915, 2874, 1719, 1636, 1601, 1573, 1477, 1452, 1406, 1289, 1251, 1107, 995, 914, 884, 767, 753, 702, 670, 661, 631; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{16}H_{14}ClN_2$ 269.0840; found 269.0841.

(*R*)-5-Bromo-3-(1-(*o*-tolyl)allyl)-1*H*-pyrrolo[2,3-*b*]pyridine (3dm)

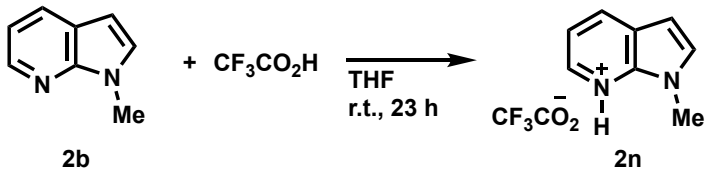


3dm

Compound **3dm** was prepared according to general procedure using **1d** (74.3 mg, 0.501 mmol) and **2m** (147.8 mg, 0.7501 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (7/3) followed by toluene/EtOAc (9/1) to afford **3dm** (76.9 mg, 0.235 mmol, 47% yield, 99% ee) as a pale yellow solid (m.p. 131.9 °C-132.3 °C). The ee was measured by HPLC (Chiralcel OZ-H column, 1.0 mL/min, hexane/2-propanol = 98/2, 230 nm, t_1 = 6.0 min (*major*), t_2 = 7.6 min (*minor*)); $[\alpha]^{24}_D$ +53 (*c* 1.03, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.64 (s, 1H), 8.31 (d, *J* = 1.7 Hz, 1H), 7.79 (d, *J* = 1.7 Hz, 1H), 7.24-7.10 (m, 4H), 6.94 (s, 1H), 6.27 (ddd, *J* = 16.6, 10.6, 6.4 Hz, 1H), 5.25 (d, *J* = 9.8 Hz, 1H), 5.05 (d, *J* = 6.3 Hz, 1H), 4.96 (d, *J* = 17.2 Hz,

1H), 2.32 (s, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 147.6, 143.1, 140.1, 139.0, 136.1, 130.6, 130.3, 127.9, 126.7, 126.1, 125.2, 121.4, 116.3, 115.9, 111.1, 42.6, 19.5; IR (KBr, cm^{-1}) 3147, 2847, 1636, 1603, 1568, 1481, 1461, 1404, 1287, 1251, 1209, 1107, 1083, 997, 921, 906, 879, 749, 724, 694, 634, 612, 549, 452; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{16}BrN_2$ 327.0491; found 327.0495.

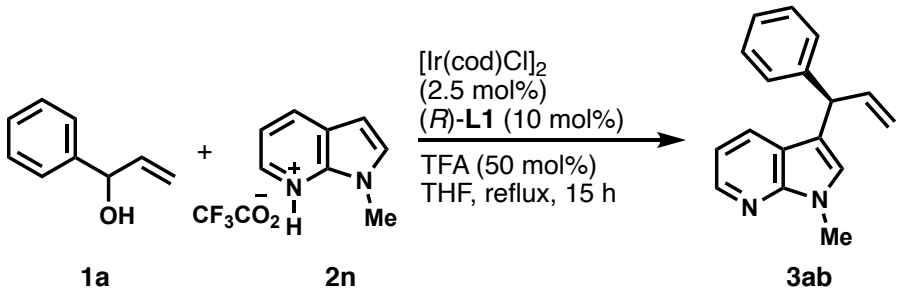
4. Procedure for the Protonation of 7-Azaindoles 2b with Trifluoroacetic Acid



To a solution of **2b** (194.9 mg, 1.475 mmol) in THF (3 mL) was added trifluoroacetic acid (227.4 mg, 1.995 mmol) at r.t. After the mixture was stirred at the same temperature for 23 h, it was concentrated on a rotary evaporator to give **2n** (329.4 mg, 1.338 mmol, 91% yield) as an orange-pink solid (m.p. 55.1 °C-55.3 °C).

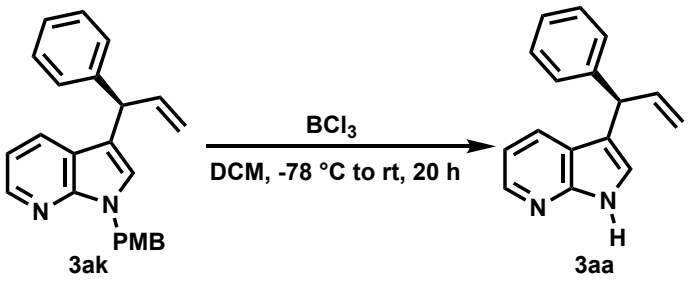
Compound **2n**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.56 (dd, J = 5.8, 1.2 Hz, 1H), 8.34 (dd, J = 8.1, 1.1 Hz, 1H), 7.38 (dd, J = 8.0, 5.8 Hz, 1H), 7.33 (d, J = 3.4 Hz, 1H), 6.72 (d, J = 3.5 Hz, 1H), 4.09 (s, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 161.6 (q, $J_{C\text{-}F}$ = 37.4 Hz), 140.7, 135.8, 135.6, 132.3, 125.2, 116.0 (q, $J_{C\text{-}F}$ = 287.7 Hz), 115.3, 102.2, 33.6; ^{19}F NMR (471 MHz, $CDCl_3$) δ -75.6; IR (KBr, cm^{-1}) 3104, 2954, 2822, 2378, 1789, 1685, 1646, 1624, 1527, 1418, 1341, 1271, 1207, 1195, 1134, 884, 834, 804, 734, 723, 709, 526.
After recrystalization of **2n** from hexane/$CHCl_3$, a crystal structure was obtained (Figure S1, Tables S1-S3).

5. Procedure for the Asymmetric Allylation of 7-Azaindole 2n with Allylic Alcohol 1a (Scheme 6)



A mixture of $[Ir(cod)Cl]_2$ (8.9 mg, 0.132 mmol) and (*R*)-**L1** (26.3 mg, 0.0518 mmol) in THF (2.0 mL) was stirred at room temperature for 15 min. To the mixture was added **2n** (185.0 mg, 0.7515 mmol), **1a** (68.9 mg, 0.513 mmol), and TFA (29.2 mg, 0.256 mmol), and the mixture was stirred under reflux for 15 h. After the solvent was removed on a rotary evaporator, the residue was subjected to column chromatography (silica gel, toluene/EtOAc = 98/2) to give compounds **3ab** (102.6 mg, 0.4132 mmol, 81% yield, 99% ee).

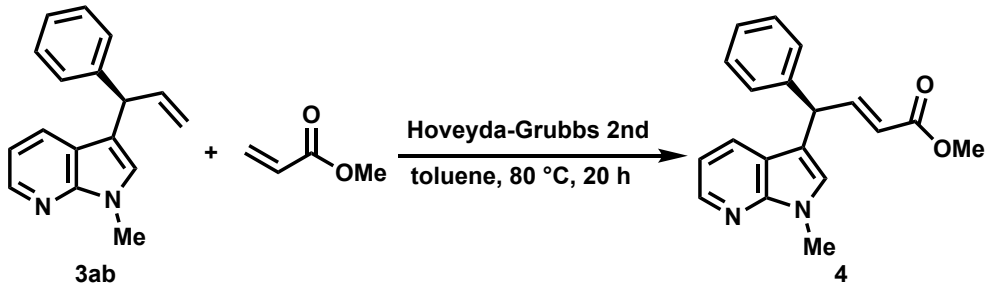
6. Procedure for the Deprotection of 7-Azaindoles 3ak (Scheme 6)



To a solution of **3ak** (129.7 mg, 0.3659 mmol, 98% ee) in DCM (2.0 mL) was added BCl_3 (1.0 M solution in THF, 2.2 mL, 2.2 mmol) at -78 °C. After the mixture was stirred at the same temperature for 30 min, it was warmed to -20 °C, and then stirred for 1 h before warming to room temperature. After the mixture was stirred for 20 h, MeOH was added. The solution was concentrated on a rotary evaporator. The residue was subjected to column chromatography (silica gel, $CHCl_3$/MeOH = 9/1 followed by hexane/acetone = 2/1) to give compound **3aa** (70.0 mg, 0.299 mmol, 82% yield, 99% ee) as a brown oil.

7. Procedure for the Metathesis Reaction of 7-Azaindoles 3ab with Methyl Acrylate

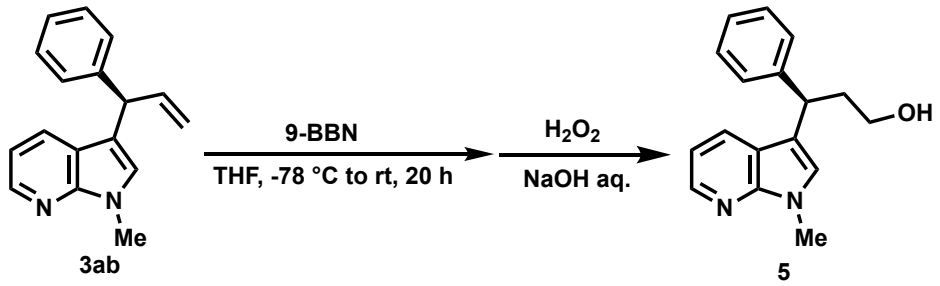
(Scheme 6)



To a solution of Hoveyda-Grubbs 2nd-generation catalyst (19.1 mg, 0.0307 mmol) in toluene (1.0 mL) was added the solution of **3ab** (73.1 mg, 0.294 mmol, 99% ee) and methyl acrylate (76.3mg, 0.886 mmol) in toluene (3.0 mL) at room temperature. After the mixture was stirred at 80 °C for 20 h, it was concentrated on a rotary evaporator. The residue was subjected to column chromatography (silica gel, hexane/EtOAc = 7/3) to give compound **4** (64.1 mg, 0.209 mmol, 71% yield, 99% ee) as a brown oil.

Compound **4:** The ee was measured by HPLC (Chiralcel OD-H column, 1.0 mL/min, hexane/2-propanol = 95/5, 230 nm, t_1 = 11.9 min (*minor*), t_2 = 16.5 min (*major*)); $[\alpha]^{26}_{D}$ –4 (*c* 0.99, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.58 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.43 (dd, *J* = 15.5, 7.2 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.30–7.22 (m, 3H), 6.96 (dd, *J* = 7.5, 4.9 Hz, 1H), 6.87 (s, 1H), 5.81 (dd, *J* = 15.5, 1.7 Hz, 1H), 5.06 (d, *J* = 7.5 Hz, 1H), 3.84 (s, 3H), 3.73 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.0, 149.6, 148.2, 143.1, 140.8, 128.7, 128.4, 127.7, 127.3, 127.1, 121.9, 119.2, 115.3, 113.5, 51.6, 45.5, 31.1; IR (neat, cm^{-1}) 3059, 3027, 2949, 1721, 1653, 1599, 1538, 1492, 1459, 1436, 1409, 1349, 1298, 1272, 1230, 1194, 1169, 1143, 1038, 985, 801, 756, 731, 702; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{19}H_{19}N_2O_2$ 307.1441; found 307.1442.

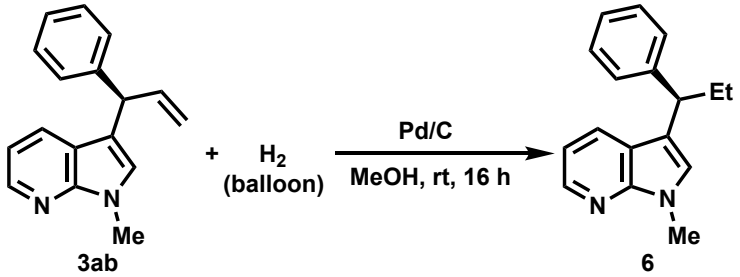
8. Procedure for the Hydroboration of 7-Azaindoles 3ab Followed by Oxidation (Scheme 6)



To a solution of **3ab** (74.8 mg, 0.301 mmol, 99% ee) in THF (1.0 mL) was added 9-BBN (1.25 mL, 0.5 M solution in THF, 0.625 mmol) at -78 °C. After the mixture was warmed to room temperature, it was stirred for 20 h. EtOH (0.49 mL), 3 M NaOH aq. (0.25 mL), and H_2O_2 (30% in water, 0.25 mL, 2.2 mmol) were added to the mixture at 0 °C. The mixture was extracted with 1M NaOH and Et_2O, and then sat.NH_4Cl and Et_2O. The combined organic extracts were dried over $MgSO_4$, and concentrated on a rotary evaporator. The residue was subjected to column chromatography (silica gel, hexane/EtOAc = 1/2) to give compound **5** (70.3 mg, 0.264 mmol, 88% yield, 99% ee) as a brown oil.

Compound **5:** The ee was measured by HPLC (Chiralpak AD-3 column, 1.0 mL/min, hexane/2-propanol = 90/10, 230 nm, t_1 = 35.7 min (*major*), t_2 = 38.8 min (*minor*)); $[\alpha]^{24}_D$ –21 (*c* 0.49, $CHCl_3$) for 99% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (dd, *J* = 4.6, 1.4 Hz, 1H), 7.67 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.33–7.23 (m, 4H), 7.21–7.15 (m, 1H), 7.01 (s, 1H), 6.93–6.88 (m, 1H), 4.36 (t, *J* = 7.7 Hz, 1H), 3.83 (s, 3H), 3.72–3.58 (m, 2H), 2.50–2.38 (m, 1H), 2.31–2.20 (m, 1H), 1.92 (br, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.1, 144.3, 142.8, 128.5, 127.8, 127.7, 126.3, 125.8, 119.7, 116.7, 114.9, 60.9, 39.1, 38.4, 31.1; IR (neat, cm^{-1}) 3338, 3060, 3026, 2932, 2880, 1600, 1575, 1538, 1492, 1461, 1410, 1348, 1295, 1143, 1038, 1011, 798, 759, 705, 664, 646, 585, 548; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{19}N_2O$ 267.1492; found 267.1491.

9. Procedure for the Hydrogenation of 7-Azaindole 3ab (Scheme 6)



To Pd/C (15.0 mg, 10%, 0.014 mmol) was added the solution of **3ab** (86.9 g, 0.35

mmol, 99% ee) in MeOH (2.0 mL) at rt. After the mixture was stirred under a hydrogen atmosphere at rt for 16 h, Pd/C was filtered off by celite, and the filtrate was concentrated on a rotary evaporator. The residue was subjected to column chromatography (silica gel, toluene/EtOAc = 98/2) to give compound **6** (77.8 mg, 0.311 mmol, 89% yield, 96% ee) as a colorless oil.

Compound **6:** The ee was measured by HPLC (OJ-3 column, 1.0 mL/min, hexane/2-propanol = 99/1, 230 nm, t_1 = 13.3 min (*minor*), t_2 = 20.2 min (*major*)); $[\alpha]^{24}_{D}$ – 36 (*c* 0.55, $CHCl_3$) for 96% ee. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.65 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.30–7.23 (m, 4H), 7.21–7.14 (m, 1H), 7.00 (s, 1H), 6.91 (dd, *J* = 8.1, 4.6 Hz, 1H), 4.00 (t, *J* = 7.4 Hz, 1H), 3.84 (s, 3H), 2.26–2.14 (m, 1H), 2.08–1.96 (m, 1H), 0.94 (t, *J* = 7.2, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 148.1, 144.9, 142.7, 128.3, 127.9, 127.6, 126.1, 125.7, 119.8, 117.3, 114.8, 44.8, 31.0, 28.8, 12.7; IR (neat, cm^{-1}) 3058, 3026, 2962, 2929, 2871, 1599, 1571, 1537, 1491, 1459, 1409, 1348, 1297, 1146, 798, 771, 704, 581, 545; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{17}H_{19}N_2$ 251.1543; found 251.1545.

10. The data for X-ray crystal structure of compound [CF_3COO][$HC_8N_2H_9$][CF_3COOH]

Colorless crystals of [CF_3COO][$HC_8N_2H_9$][CF_3COOH] suitable for X-ray crystallographic analysis were obtained by recrystallization from hexane/$CHCl_3$. The ORTEP drawing of [CF_3COO][$HC_8N_2H_9$][CF_3COOH] is shown in Figure S1. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 2080098). The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

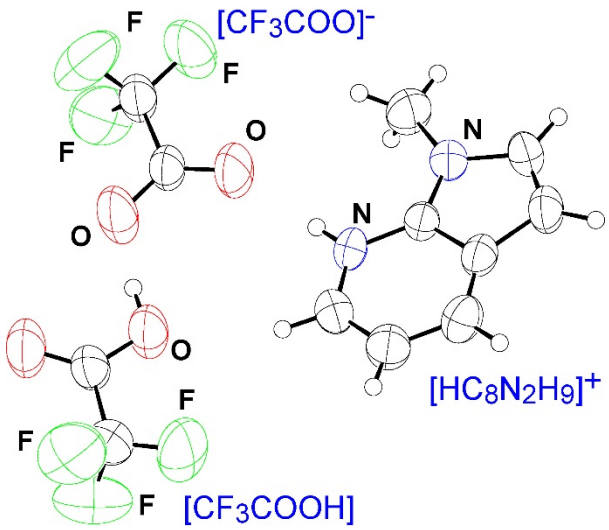


Figure S1. ORTEP illustration of [CF_3COO][$HC_8N_2H_9$][CF_3COOH] with thermal ellipsoids drawn at the 50% probability level.

Table S1. Crystal data of [CF_3COO][$HC_8N_2H_9$][CF_3COOH].

Empirical Formula	$C_{12}H_{10}F_6N_2O_4$
Formula Weight	360.21
Crystal Color, Habit	colorless, prism
Crystal Dimensions	0.5 × 0.5 × 0.5 mm
Crystal System	tricrinic
Lattice Type	Primitive
Lattice Parameters	a = 7.449(5) Å
	b = 10.164(8) Å
	c = 11.085(6) Å
	α = 115.47(2) °

	$\beta = 98.08(2)^\circ$
	$\gamma = 92.02(3)^\circ$
	$V = 745.9(8)$ Å^3
Space Group	P−1 (#2)
Z value	2
D_{calc}	1.604 g/cm^3
F_{000}	364.00
μ(MoKα)	1.667 cm^{-1}

Table S2. Intensity Measurements.

Diffractometer	R-AXIS RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50 kV, 90 mA
Temperature	23.0 °C
Detector Aperture	460.0 x 256.0 mm
Data Images	44 exposures
ω oscillation Range (χ = 45.0, ϕ = 0.0)	130.0 – 190.0 °
Exposure Rate	30.0 sec./°
ω oscillation Range (χ = 45.0, ϕ = 180.0)	0.0 – 160.0 °
Exposure Rate	30.0 sec. /°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9 °
No. of Reflections Measured	Total: 7411 Unique: 3387 (R_{int} = 0.0308)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.936 – 0.967)

Table S3. Structure Solution and Refinement.

Structure Solution	Direct Methods (SHELXT Version 2018/2)
Refinement	Full-matrix least-squares on F^2 (SHELXL Version 2018/3)

Function Minimized	Σ w $(F_0^2 - F_c^2)^2$
Least Squares Weights	$w = 1/[\sigma^2(F_0^2) + (0.1017 \cdot P)^2 + 0.0000 \cdot P]$
	where $P = (Max(F_0^2,0)+2Fc^2)/3$
2θmax cutoff	54.9 °
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3387
No. Variables	261
Reflection/Parameter Ratio	12.98
Residuals: R1 (I>2.00σ(I))	0.0632
Residuals: R (All reflections)	0.1315
Residuals: wR2 (All reflections)	0.2014
Goodness of Fit Indicator	1.037
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.24 $e^-/Å^3$
Minimum peak in Final Diff. Map	–0.20 $e^-/Å^3$

11. The data for X-ray crystal structure of compound $C_{17}H_{15}BrN_2$

Yellow crystals of $C_{17}H_{15}BrN_2$ suitable for X-ray crystallographic analysis were obtained by recrystallization from hexane/EtOAc. The ORTEP drawing of $C_{17}H_{15}BrN_2$ is shown in Figure S2. The unit cell of $C_{17}H_{15}BrN_2$ consists of two type crystallographically distinct molecules which form dimer by *hydrogen bonding* of the hydrogen of N-H and the nitrogen of 7-azaindol. Nineteen hydrogen atoms, H2A, H3A, H4A, H5A, H6A, H8A, H8B, H8C, H10A, H10B, H18, H21, H22, H25A, H25B, H25C, H26, H27A, and H27B, were placed at calculated positions and refined using a riding model. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 2080099). The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

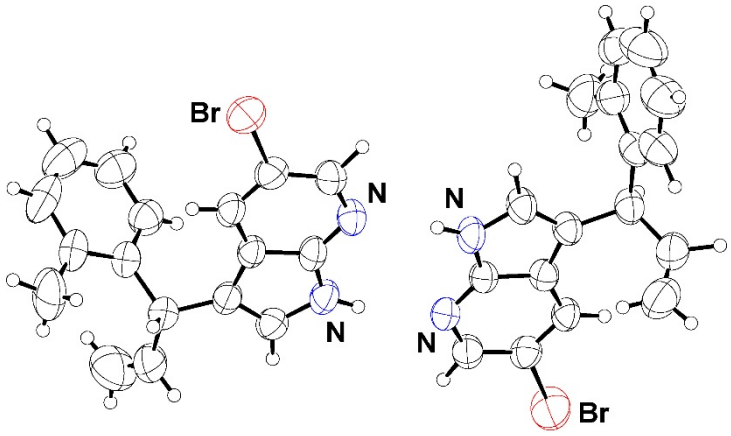


Figure S2. ORTEP illustration of $C_{17}H_{15}BrN_2$ (two crystallographically independent molecules) with thermal ellipsoids drawn at the 50% probability level.

Table S4. Crystal data of $C_{17}H_{15}BrN_2$.

Empirical Formula	$C_{17}H_{15}BrN_2$
Formula Weight	327.22
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.8 × 0.8 × 0.5 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 8.928(5) Å
	b = 17.983(10) Å
	c = 9.771(6) Å
	β = 93.34(2) °
	V = 1566.1(16) Å^3
Space Group	$P2_1$ (#4)
Z value	4
D_{calc}	1.388 g/cm^3
F_{000}	664.00
μ(MoKα)	26.241 cm^{-1}

Table S5. Intensity Measurements.

Diffractometer	R-AXIS RAPID
Radiation	MoKα (λ = 0.71075 Å)

	graphite monochromated
Voltage, Current	50kV, 90mA
Temperature	23.0 °C
Detector Aperture	460.0 x 256.0 mm
Data Images	44 exposures
ω oscillation Range (χ = 45.0, ϕ = 0.0)	130.0 – 190.0 °
Exposure Rate	30.0 sec./°
ω oscillation Range (χ = 45.0, ϕ = 180.0)	0.0 – 160.0 °
Exposure Rate	30.0 sec. /°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9 °
No. of Reflections Measured	Total: 15326
	Unique: 7100 (R_{int} = 0.0722)
Corrections	Lorentz-polarization
	Absorption
	(trans. factors: 0.188 – 0.269)
	Secondary Extinction
	(coefficient: 1.19700 e-002)

Table S6. Structure Solution and Refinement.

Structure Solution	Direct Methods (SHELXT Version 2018/2)
Refinement	Full-matrix least-squares on F^2 (SHELXL Version 2018/3)
Function Minimized	Σ w $(F_0^2 - F_c^2)^2$
Least Squares Weights	$w = 1/[\sigma^2(F_0^2) + (0.00611 \cdot P)^2 + 0.0000 \cdot P]$
where P = $(Max(F_0^2,0)+2Fc^2)/3$	
2θmax cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	7098
No. Variables	406
Reflection/Parameter Ratio	17.48
Residuals: R1 (I>2.00σ(I))	0.0513
Residuals: R (All reflections)	0.0920
Residuals: wR2 (All reflections)	0.1400

Goodness of Fit Indicator	0.931
Flack parameter	–0.004(11) (Parsons' quotients = 1347)
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.41 $e^{-}/Å^3$
Minimum peak in Final Diff. Map	–0.37 $e^{-}/Å^3$

12. References

[1] R. H. Crabtree, J. M. Quirk, H. Felkin, T. Fillebeen-Khan, *Synth. React. Inorg. Met.-Org. Chem.* 1982, **12**, 407–413.

[2] C. Defieber, M. A. Ariger, P. Moriel, E. M. Carreira, *Angew. Chem., Int. Ed*. 2007, **46**, 3139-3143.

[3] L. C. Burrows, L. T. Jesikiewicz, G. Lu, S. J. Geib, P. Liu, K. M. Brummond, *J. Am. Chem. Soc.* 2017, **139**, 15022−15032.

[4] J. Y. Hamilton, D. Sarlah, E. M. Carreira, *Org. Synth*. 2015, **92**, 1-12.

[5] P. Kannaboina, K. Anilkumar, S. Aravind, R. A. Vishwakarma, P. Das, *Org. Lett.* **2013**, *15*, 5718–5721.

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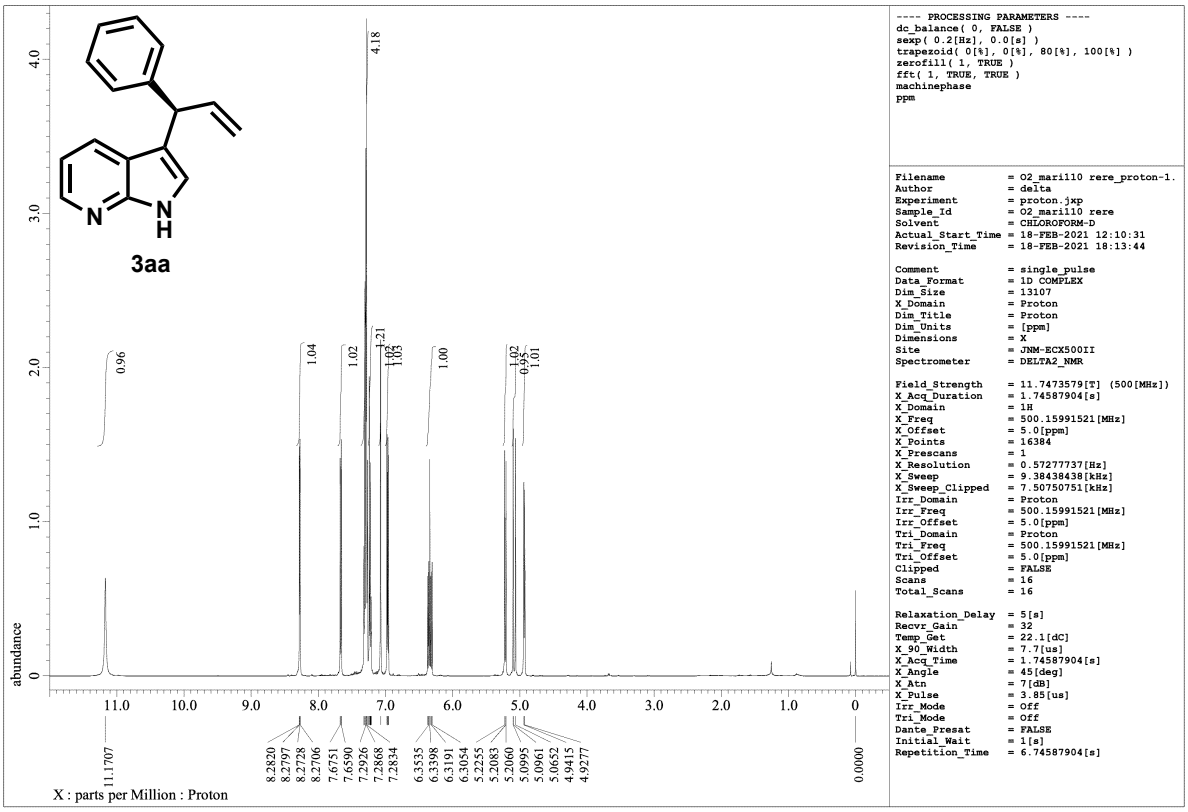
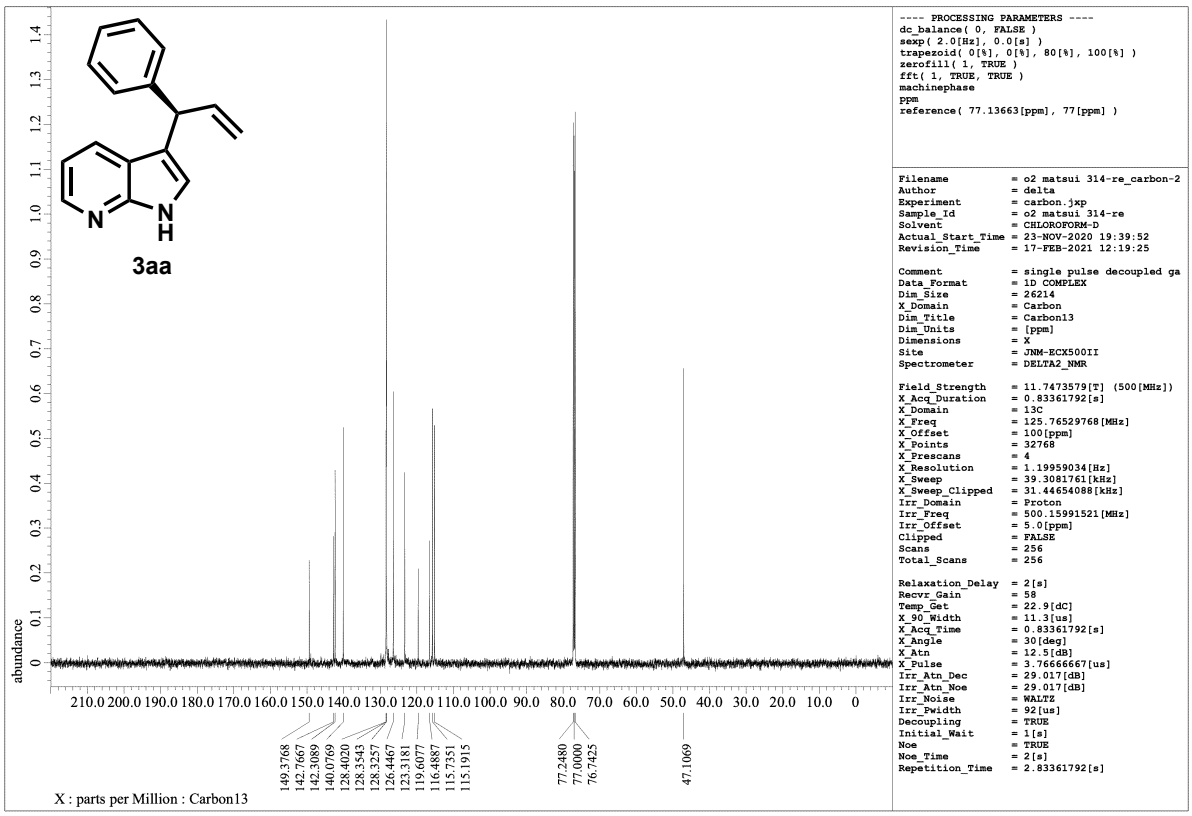
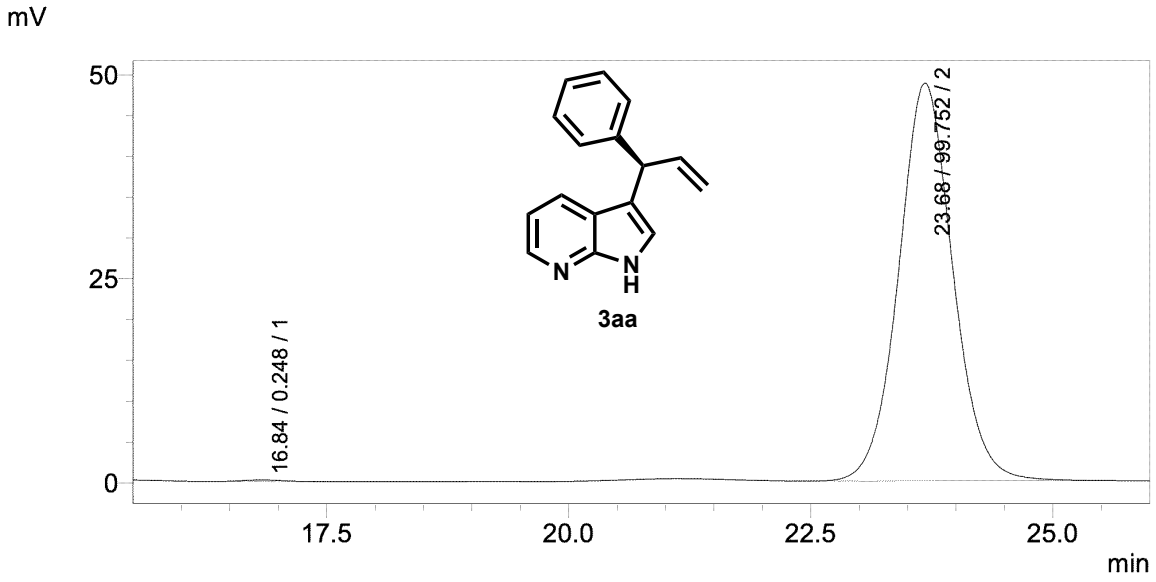
13. ^{1}H, ^{13}C, and ^{19}F NMR Spectra, and Chiral HPLC Charts







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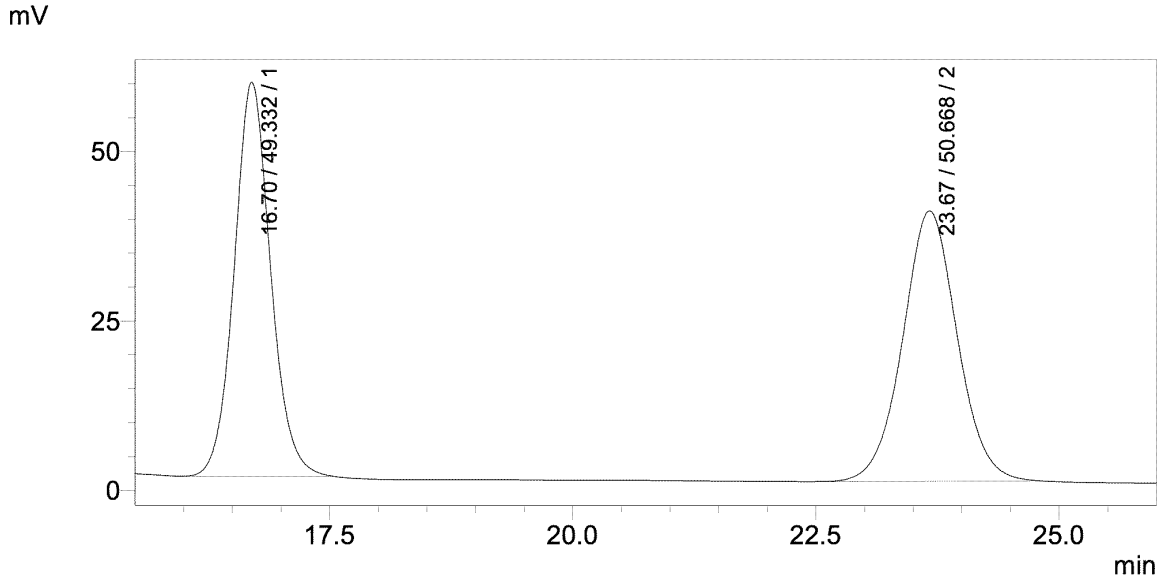

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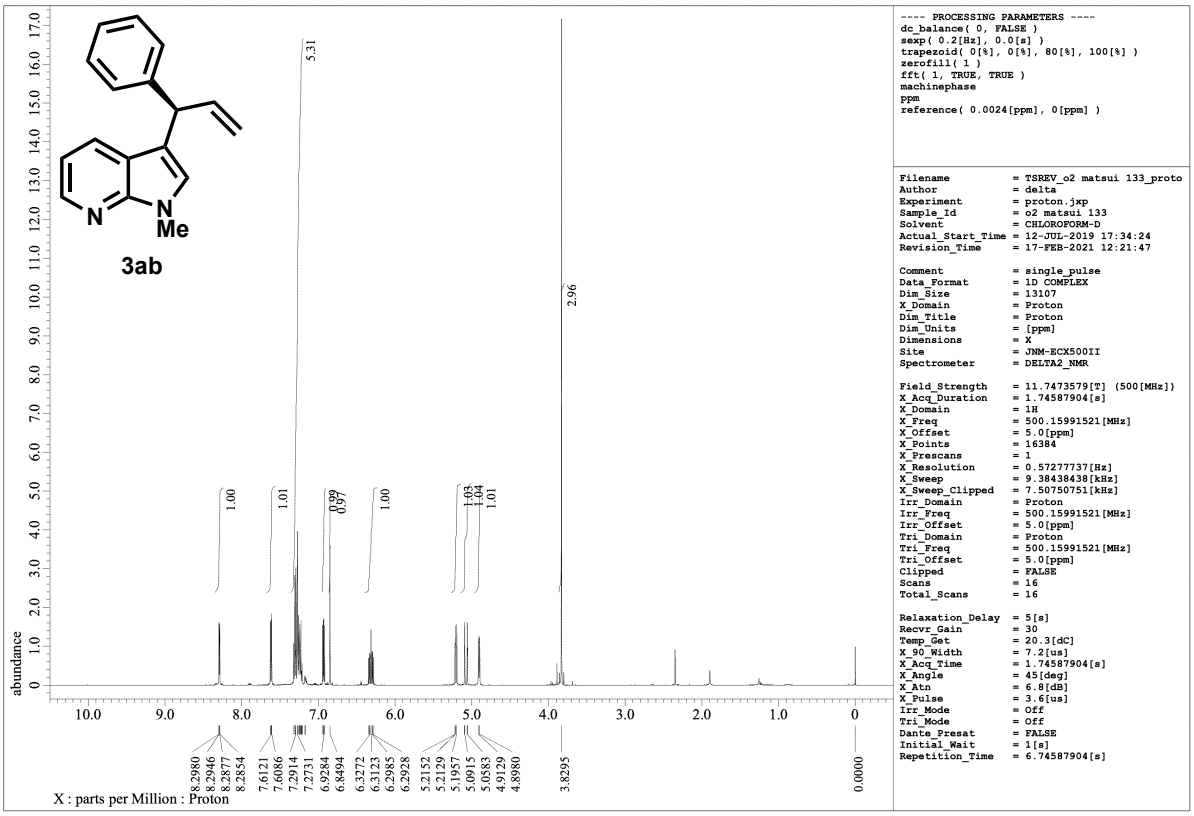
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N
Me
5.31
2.96
1.00
1.01
0.99
0.97
1.00
1.03
1.04
1.01
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.2980 8.2946 8.2877 8.2854 7.6121 7.6086 7.2914 7.2731 6.9284 6.8494 6.3272 6.3123 6.2985 6.2928 5.2152 5.2129 5.1957 5.0915 5.0583 4.9129 4.8980 3.8295 0.0000
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machinephase
ppm
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Recvr_Gain = 30
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X_Acq_Time = 1.74587904[s]
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X_Atn = 6.8[dB]
X_Pulse = 3.6[us]
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Tri_Mode = Off
Dante_Presat = FALSE
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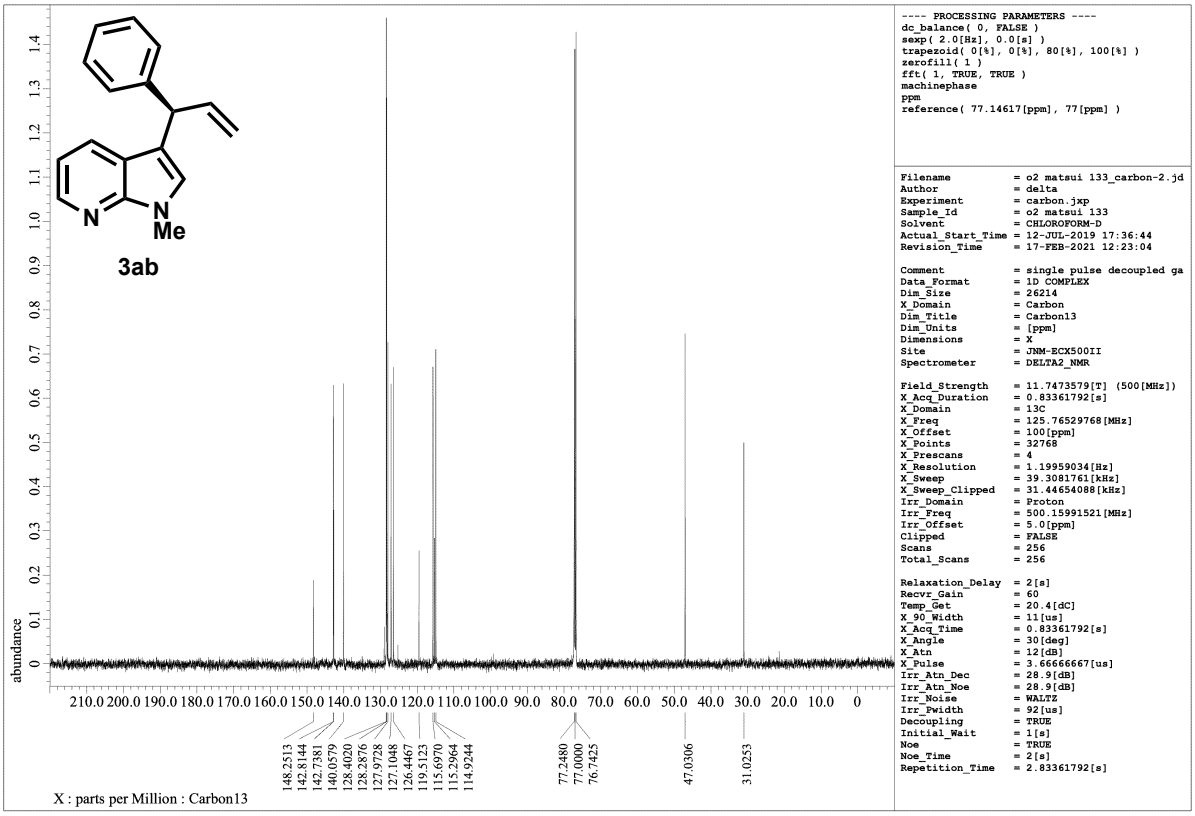
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N
N
Me
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
148.2513 142.8144 142.7381 140.0579 128.4020 128.2876 127.9728 127.1048 126.4467 119.5123 115.6970 115.2964 114.9244 77.2480 77.0000 76.7425 47.0306 31.0253
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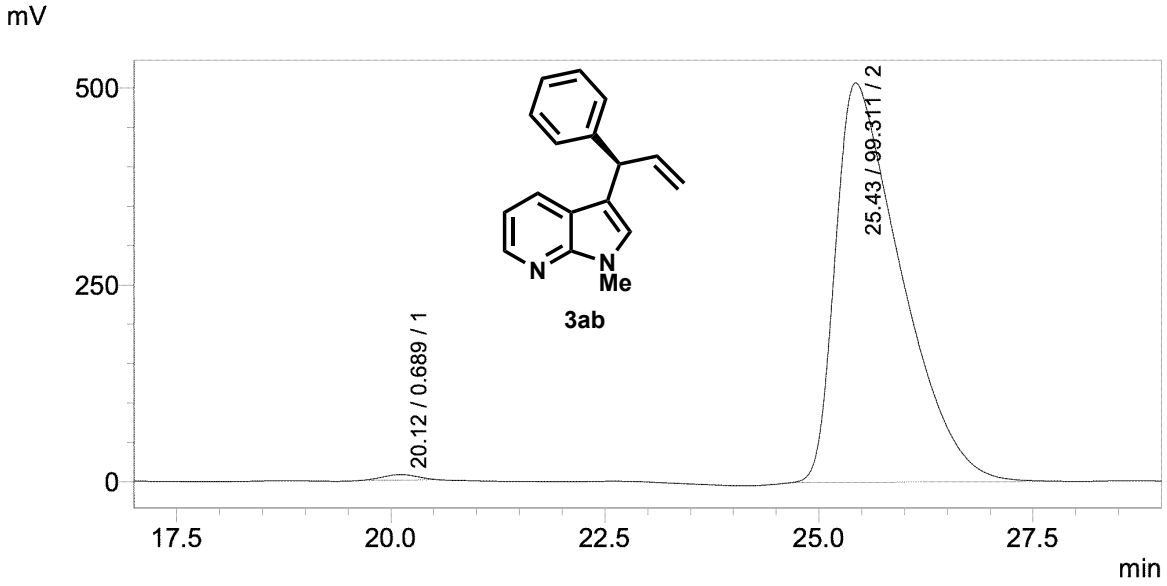

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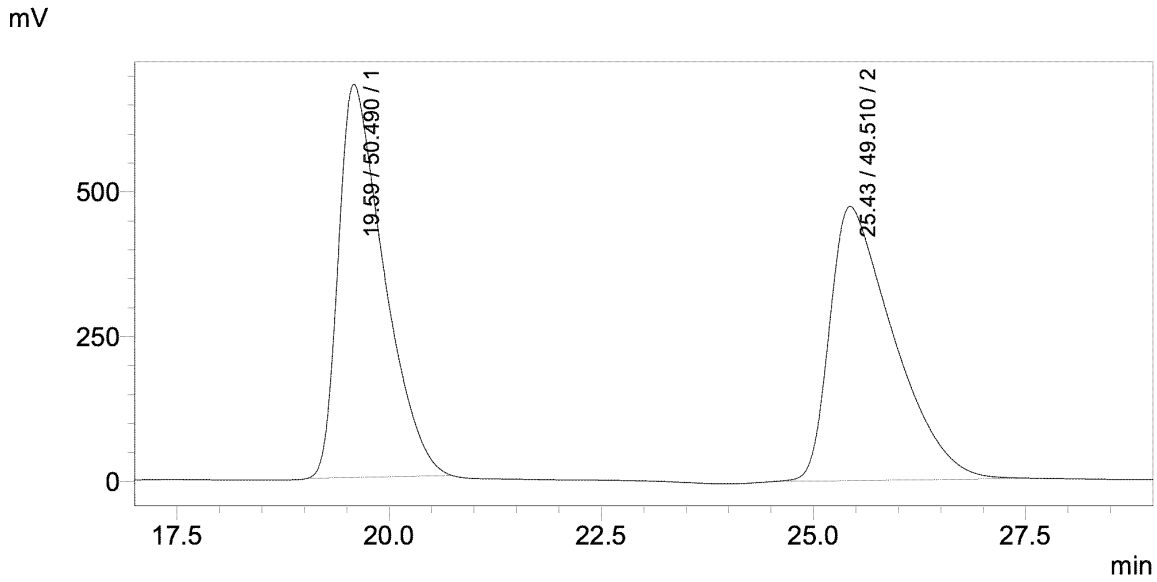

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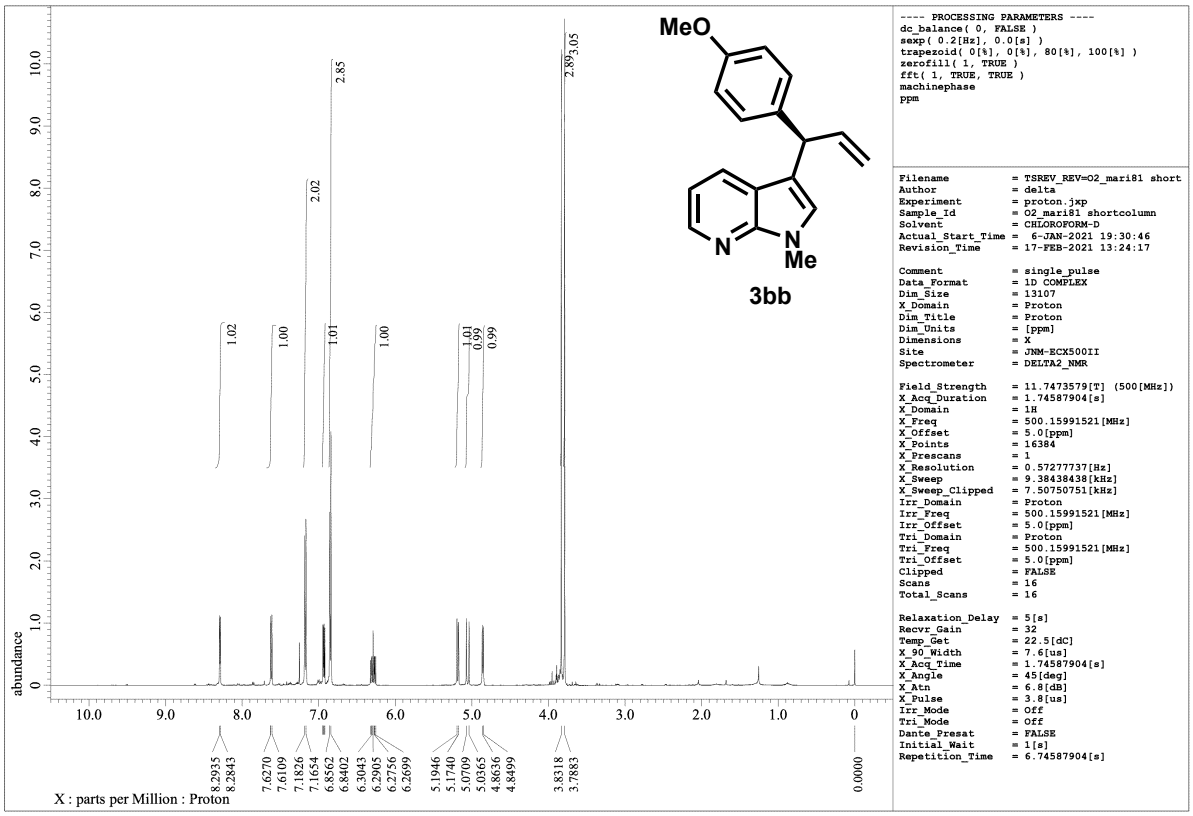


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ppm
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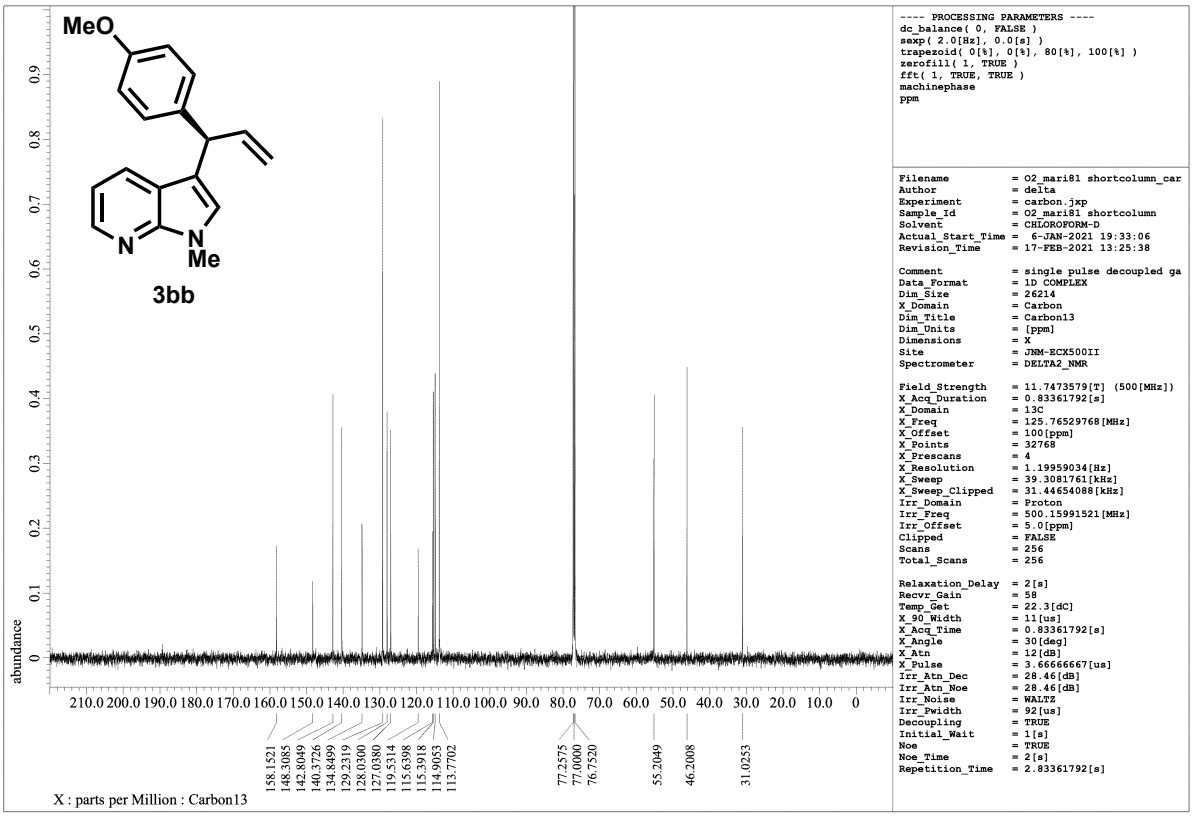


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machinephase
ppm
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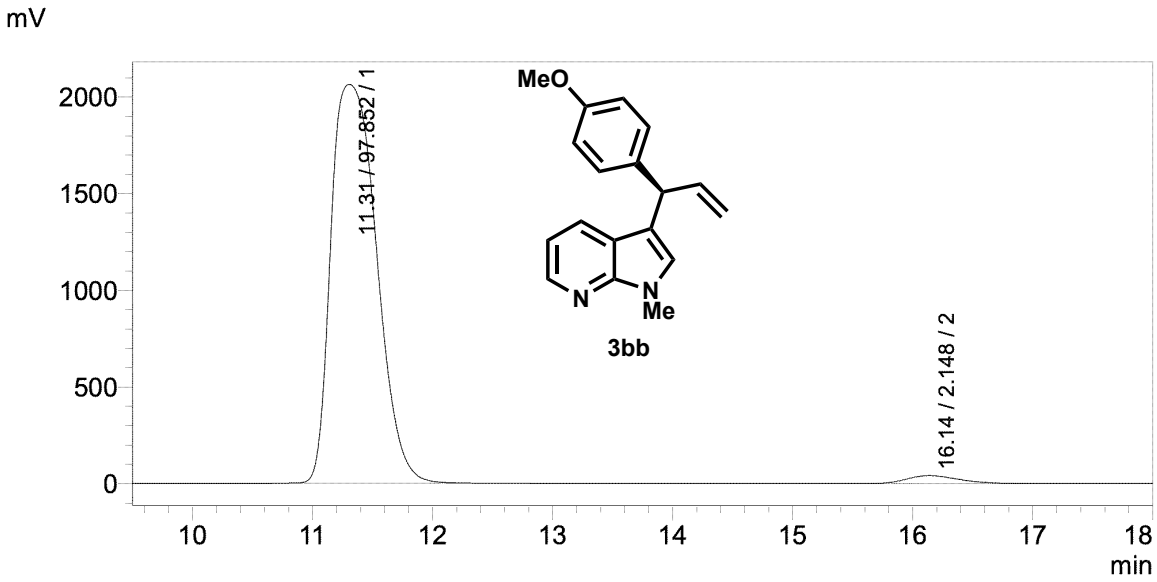
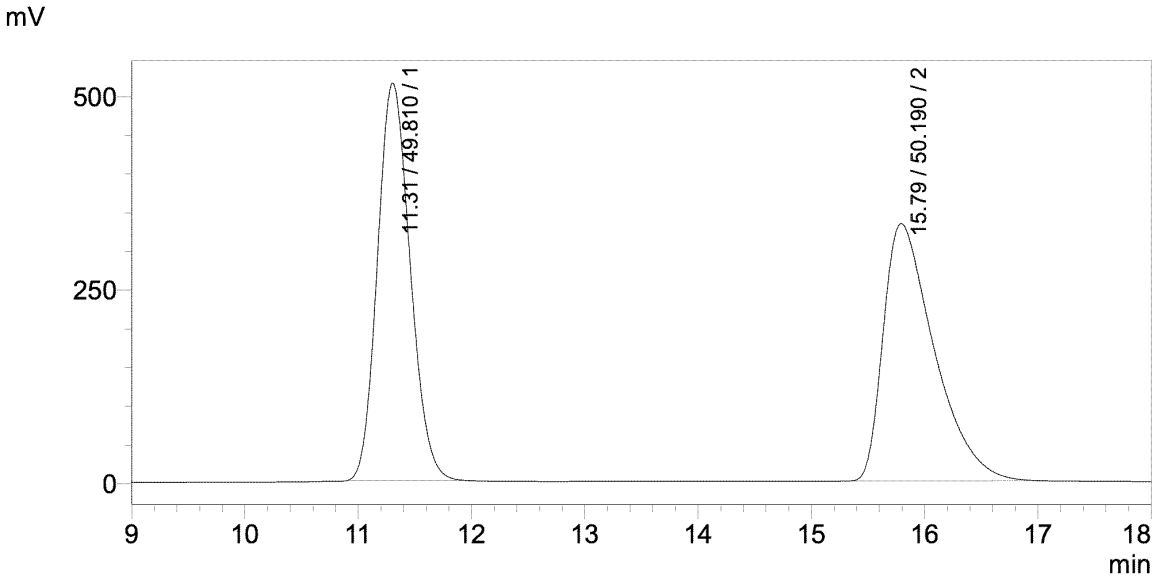
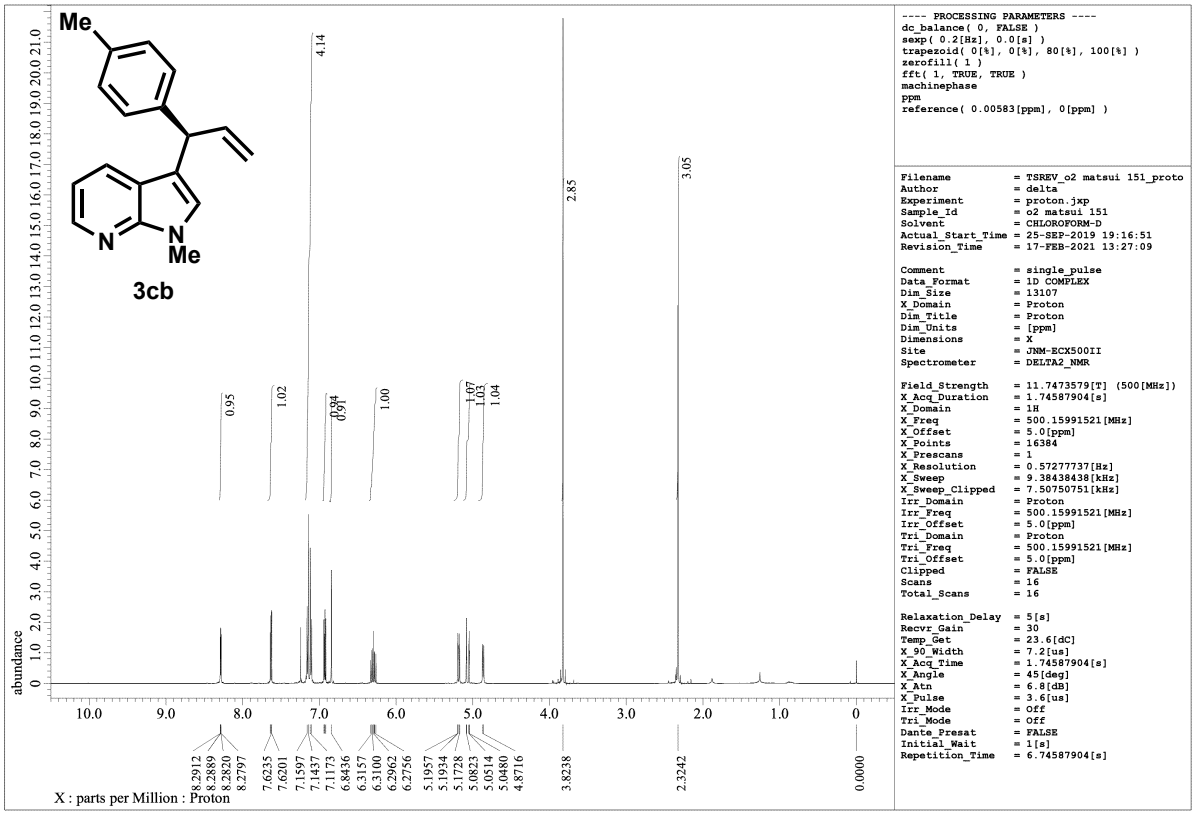
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```



Peak#	Ret. Time	Area%
1	11.31	97.852
2	16.14	2.148
Total		100.000



Peak#	Ret. Time	Area%
1	11.31	49.810
2	15.79	50.190
Total		100.000



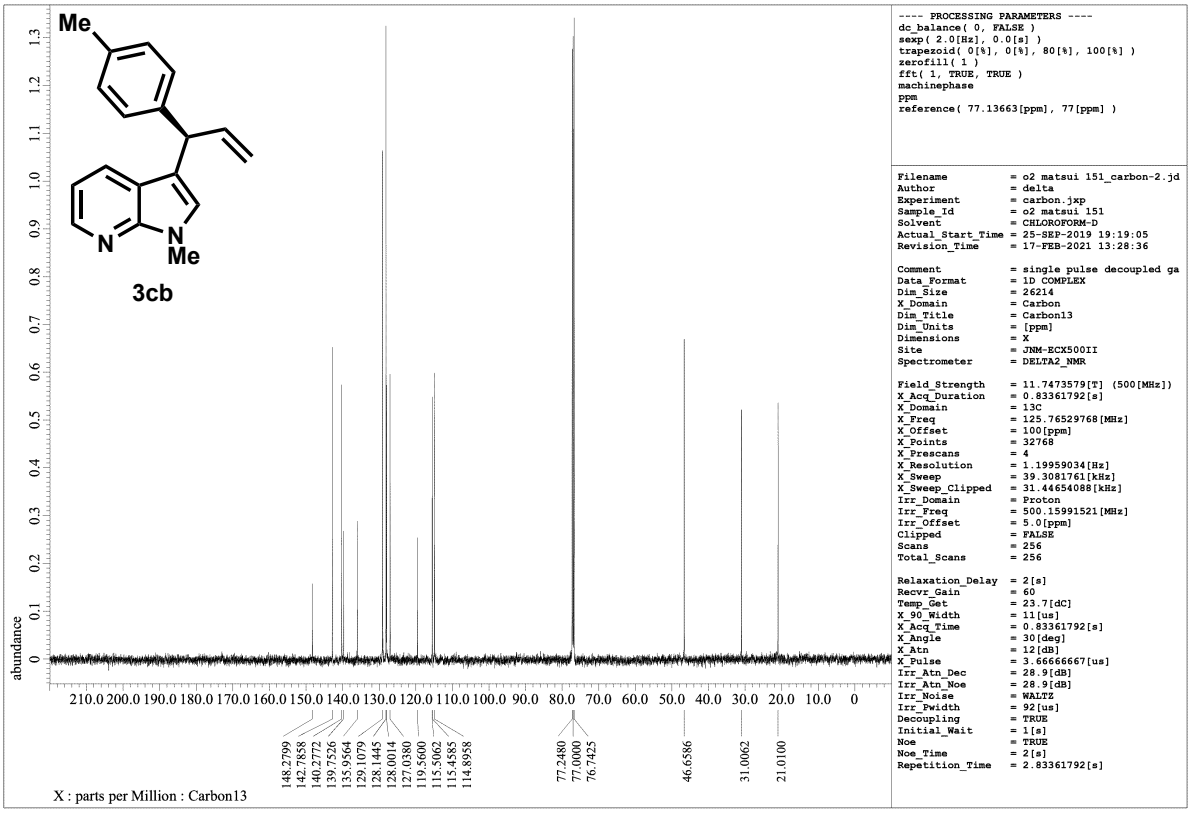

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 0.00583[ppm], 0[ppm] )
```

```
Filename          = TSREV_o2 matsui 151_proto
Author            = delta
Experiment        = proton.jxp
Sample_Id         = o2 matsui 151
Solvent           = CHLOROFORM-D
Actual_Start_Time = 25-SEP-2019 19:16:51
Revision_Time     = 17-FEB-2021 13:27:09

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proton
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECX500II
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 5.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38438438[kHz]
X_Sweep_Clipped   = 7.50750751[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Tri_Domain        = Proton
Tri_Freq          = 500.15991521[MHz]
Tri_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.6[dC]
X_90_Width        = 7.2[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 6.8[dB]
X_Pulse           = 3.6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



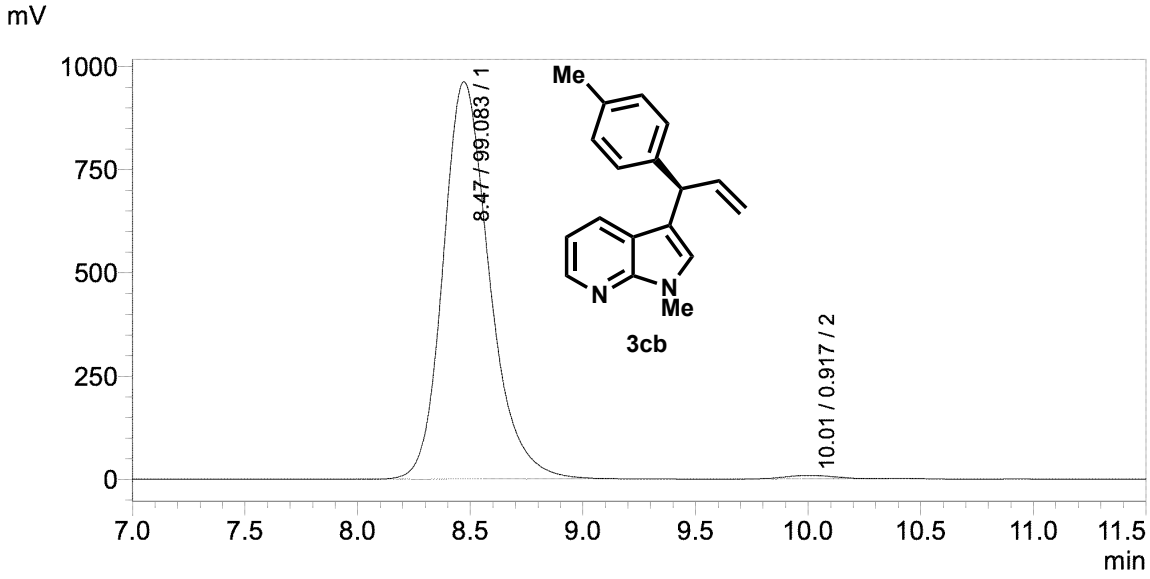

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.13663[ppm], 77[ppm] )
```

```
Filename          = o2 matsui 151_carbon-2.jd
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = o2 matsui 151
Solvent           = CHLOROFORM-D
Actual_Start_Time = 25-SEP-2019 19:19:05
Revision_Time     = 17-FEB-2021 13:28:36

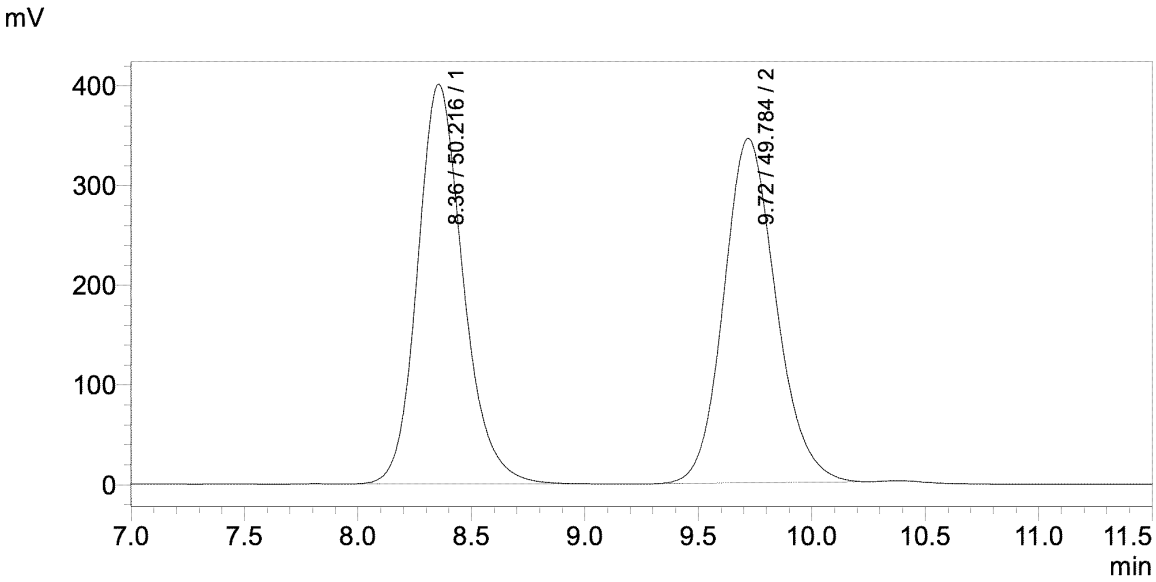
Comment           = single pulse decoupled ga
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbon
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECX500II
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

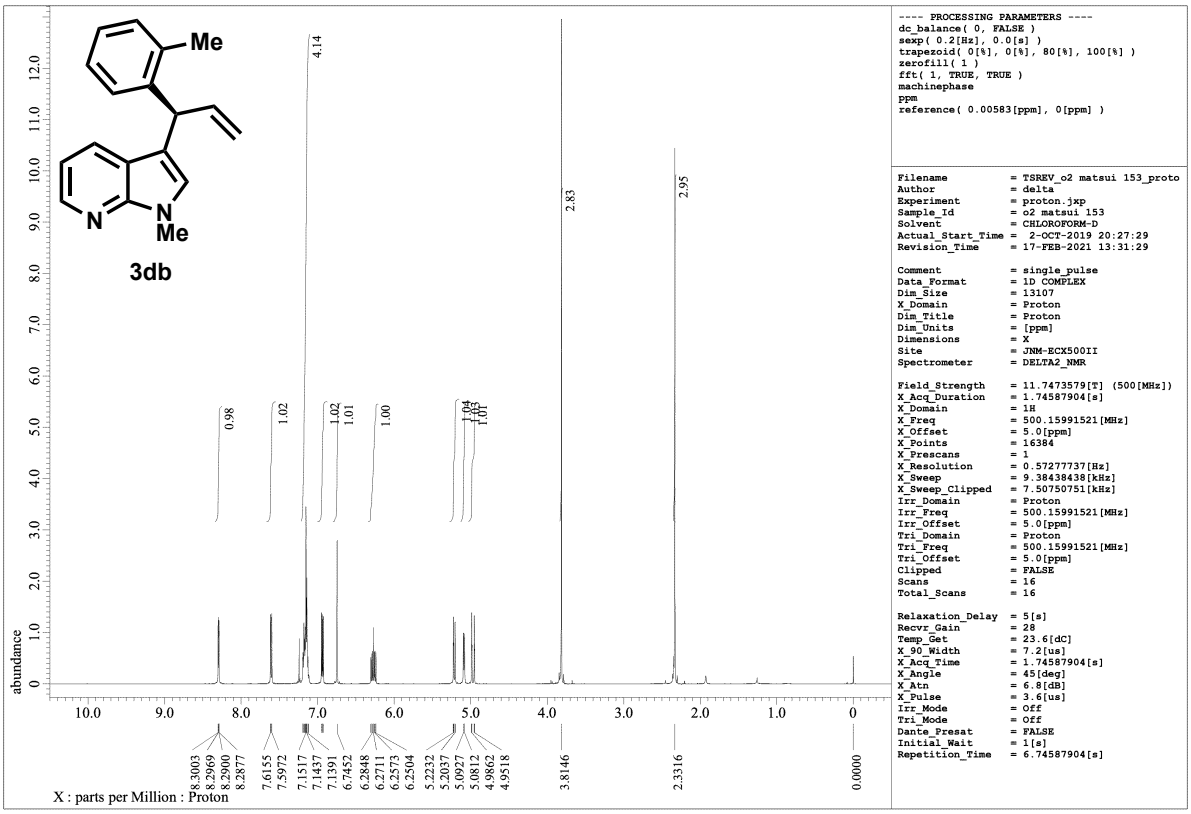
Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.7[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 12[dB]
X_Pulse           = 3.66666667[us]
Irr_Atn_Dec       = 28.9[dB]
Irr_Atn_Noe       = 28.9[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



Peak#	Ret. Time	Area%
1	8.47	99.083
2	10.01	0.917
Total		100.000



Peak#	Ret. Time	Area%
1	8.36	50.216
2	9.72	49.784
Total		100.000



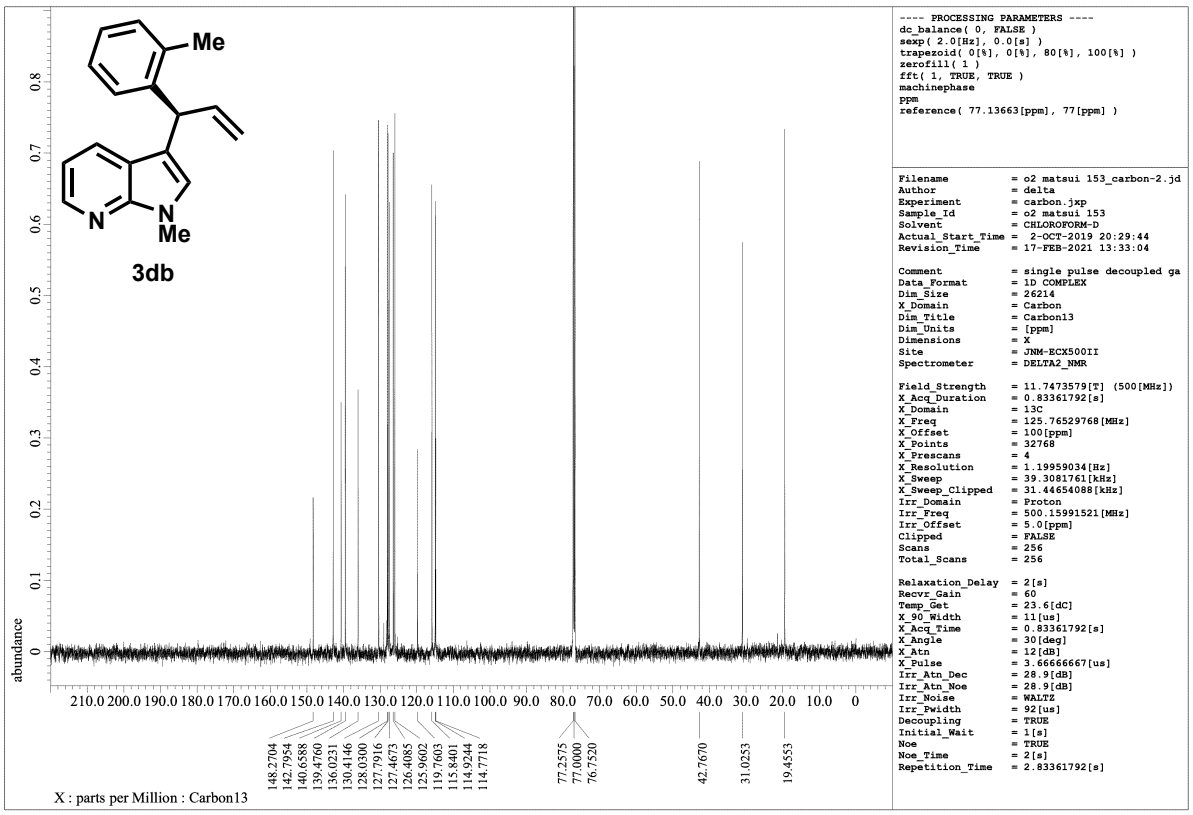

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 0.00583[ppm], 0[ppm] )
```

```
Filename            = TSREV_o2 matsui 153_proto
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 matsui 153
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  2-OCT-2019 20:27:29
Revision_Time       = 17-FEB-2021 13:31:29

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 28
Temp_Get            = 23.6[dC]
X_90_Width          = 7.2[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.6[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



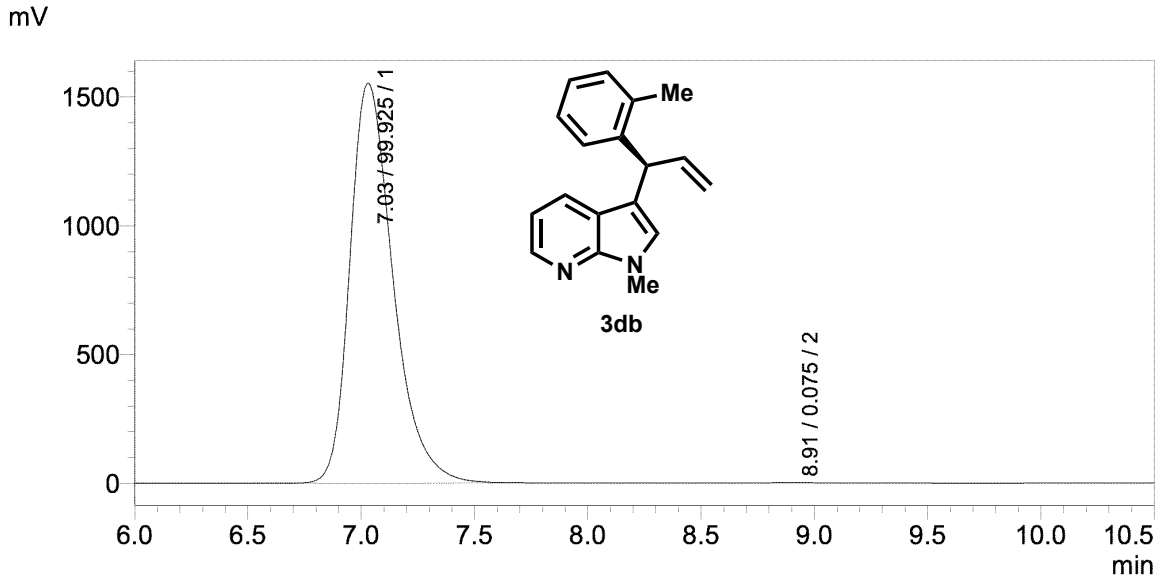

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.13663[ppm], 77[ppm] )
```

```
Filename            = o2 matsui 153_carbon-2.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 matsui 153
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  2-OCT-2019 20:29:44
Revision_Time       = 17-FEB-2021 13:33:04

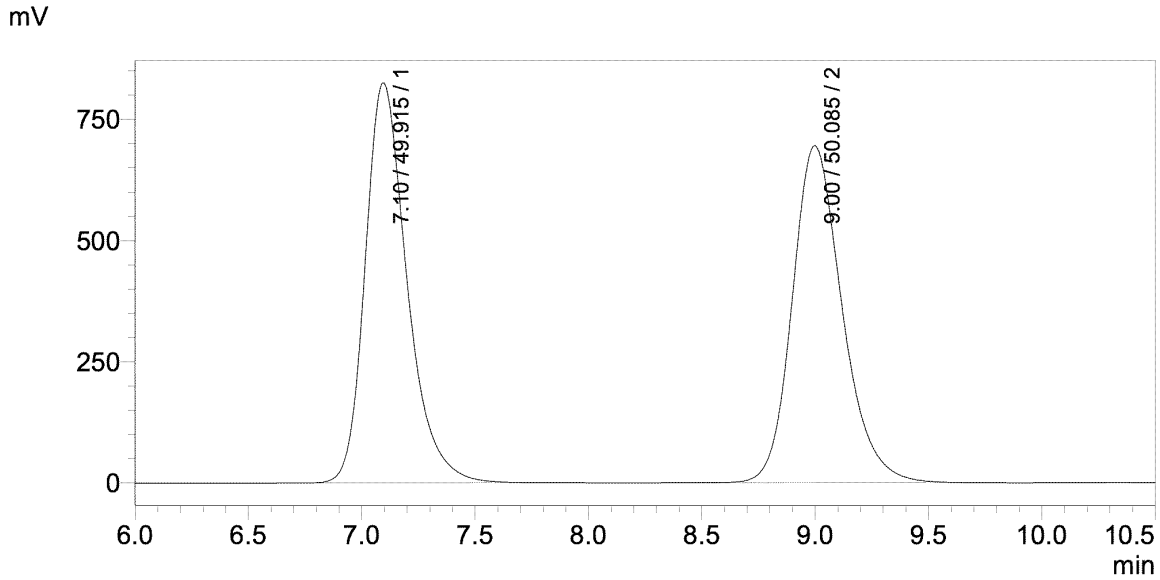
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

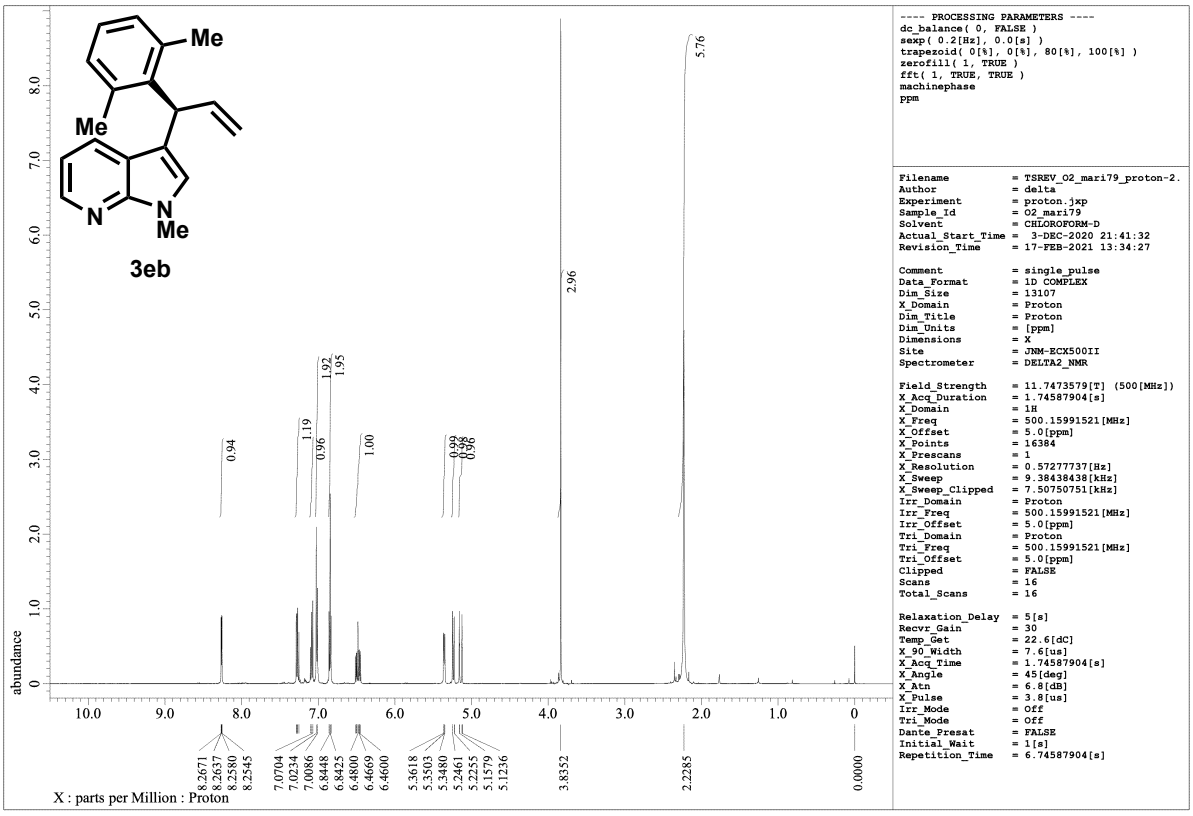
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 23.6[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.9[dB]
Irr_Atn_Noe         = 28.9[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

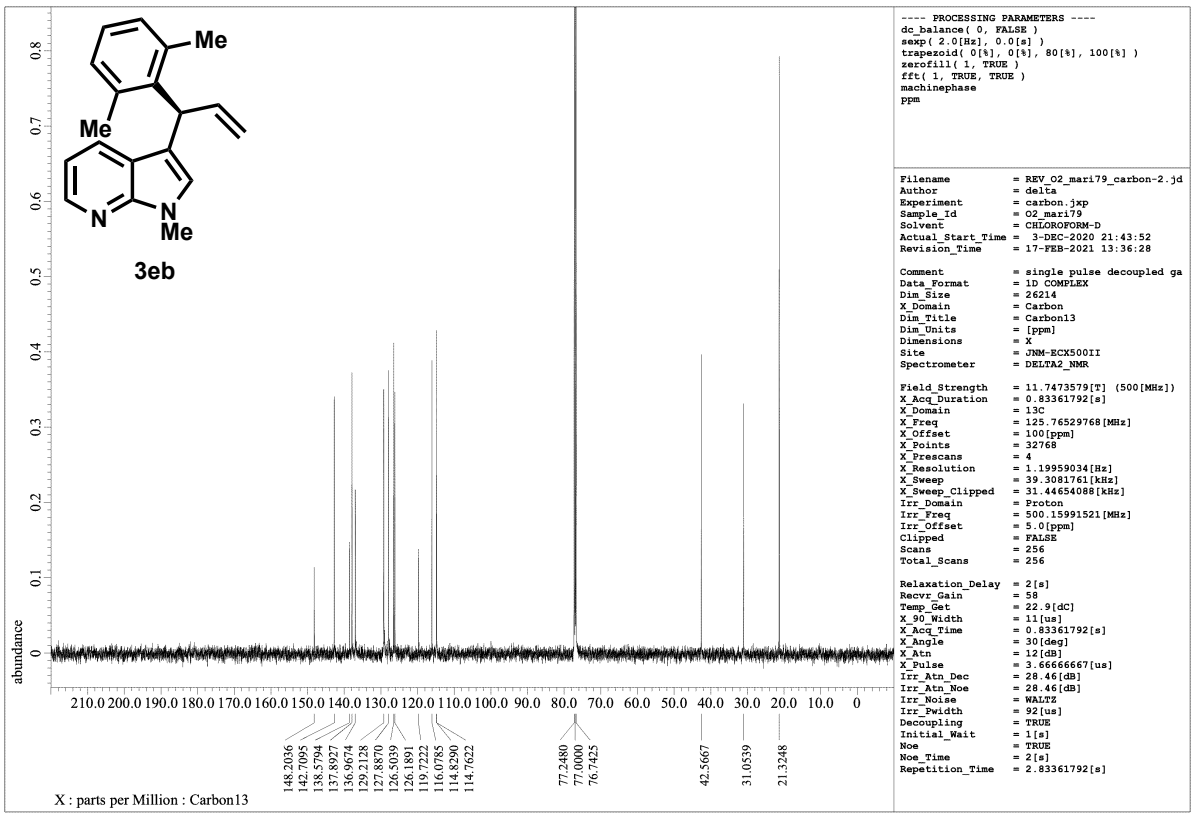


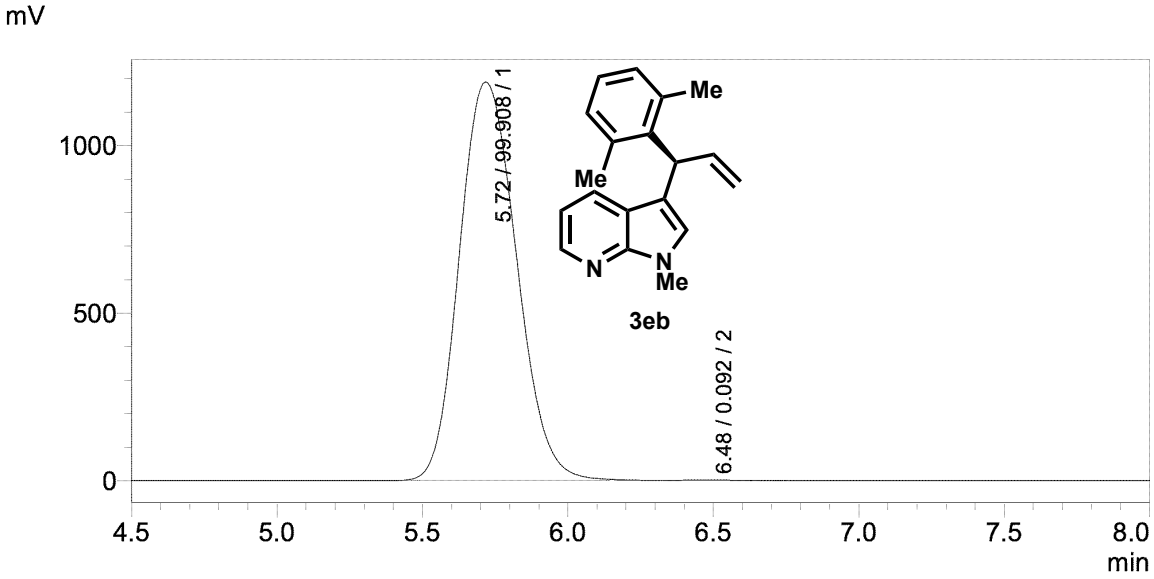
Peak#	Ret. Time	Area%
1	7.03	99.925
2	8.91	0.075
Total		100.000



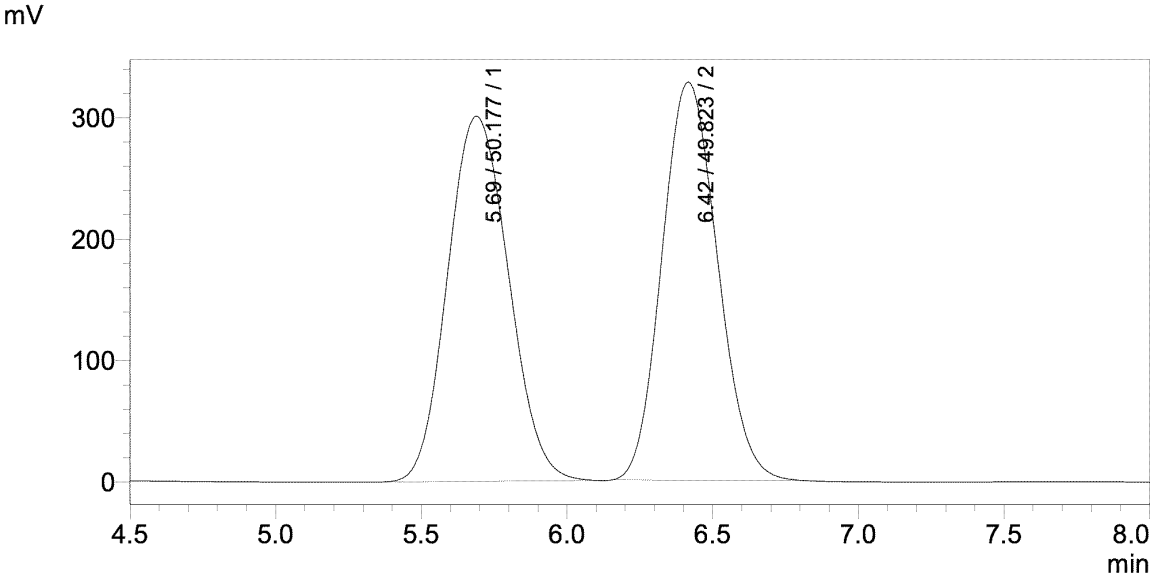
Peak#	Ret. Time	Area%
1	7.10	49.915
2	9.00	50.085
Total		100.000


Me
Me
N
N
Me
3eb
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
0.94
1.19
1.92
1.95
0.96
1.00
0.99
0.98
0.96
2.96
5.76
8.2671 8.2637 8.2580 8.2545
7.0704 7.0234 7.0086 6.8448 6.8425 6.4800 6.4669 6.4600
5.3618 5.3503 5.3480 5.2461 5.2255 5.1579 5.1236
3.8352
2.2285
0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = TSREV_O2_mari79_proton-2.
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari79
Solvent = CHLOROFORM-D
Actual_Start_Time = 3-DEC-2020 21:41:32
Revision_Time = 17-FEB-2021 13:34:27
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 30
Temp_Get = 22.6[dC]
X_90_Width = 7.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

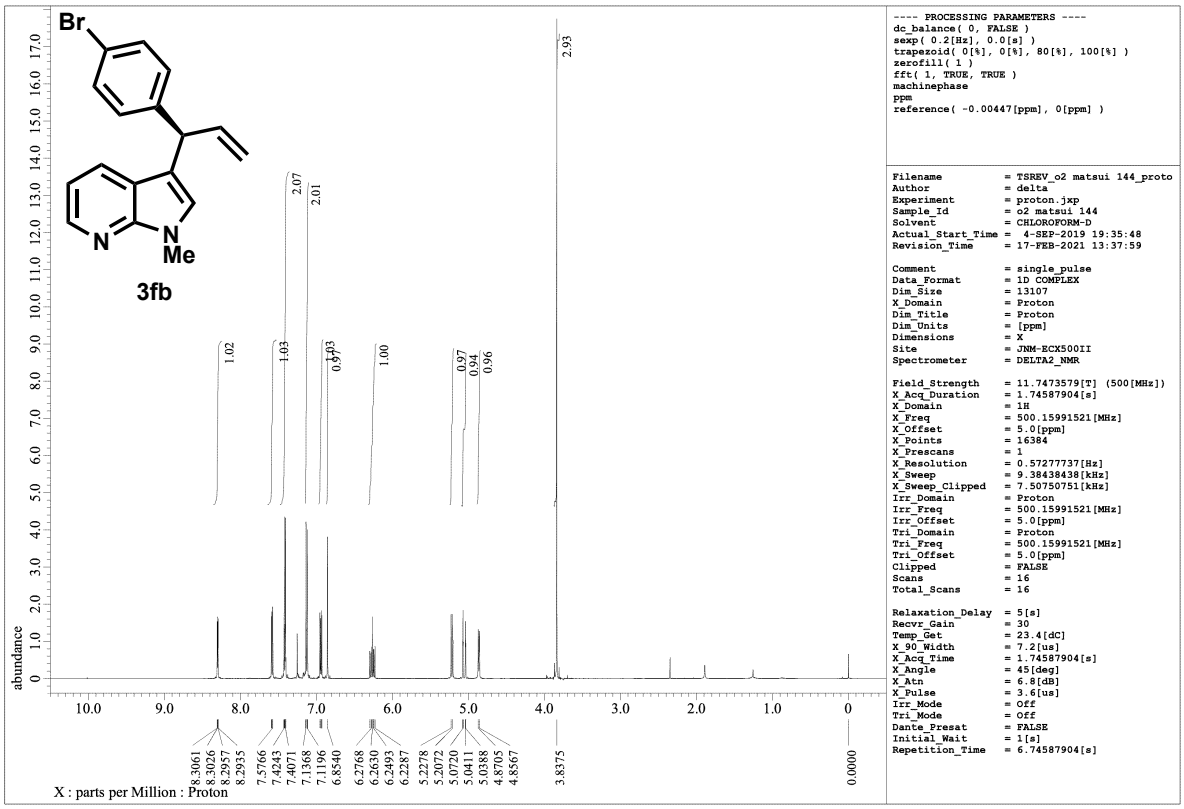

Me
Me
N
N
Me
3eb
abundance
0.8 0.7 0.6 0.5 0.4 0.3 0.2 0.1 0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
148.2036
142.7095
138.5794
137.8927
136.9674
129.2128
127.8870
126.5039
126.1891
119.7222
116.0785
114.8290
114.7622
77.2480
77.0000
76.7425
42.5667
31.0539
21.3248
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = REV_O2_mari79_carbon-2.jd
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari79
Solvent = CHLOROFORM-D
Actual_Start_Time = 3-DEC-2020 21:43:52
Revision_Time = 17-FEB-2021 13:36:28
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.4465408[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 22.9[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]

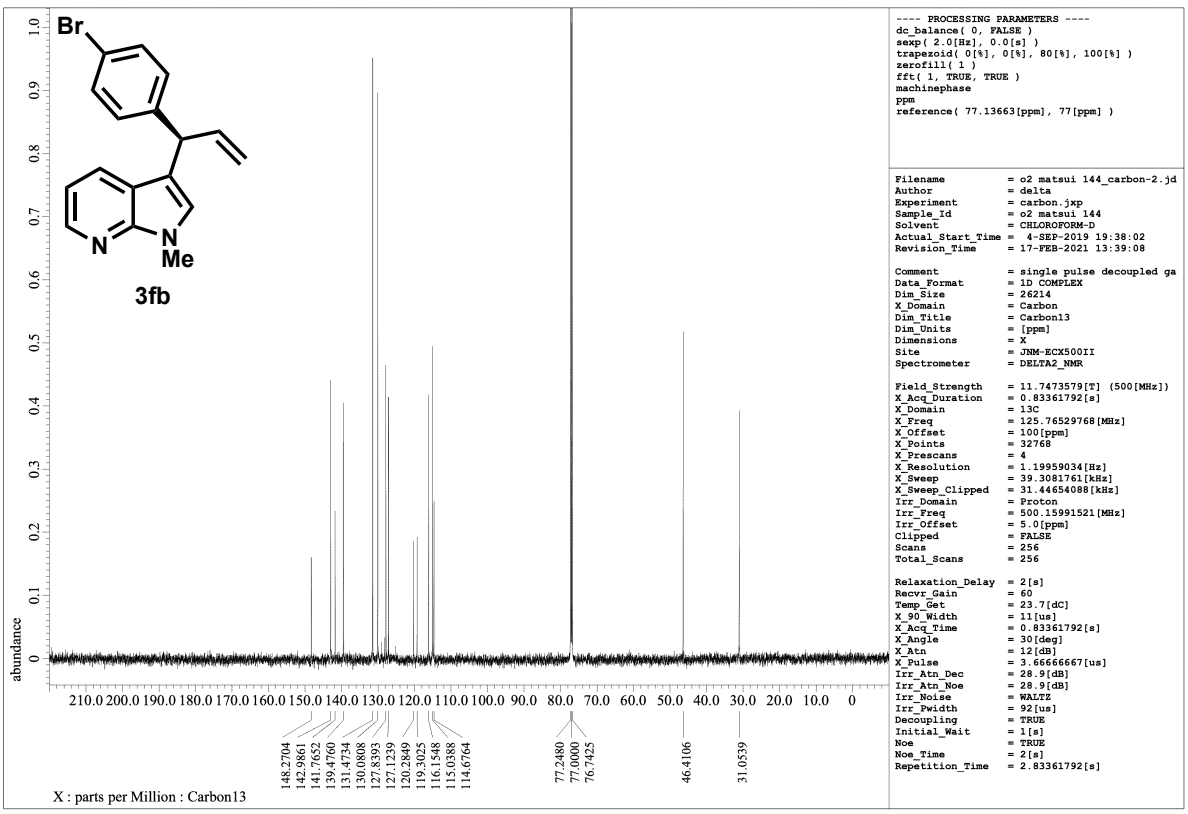


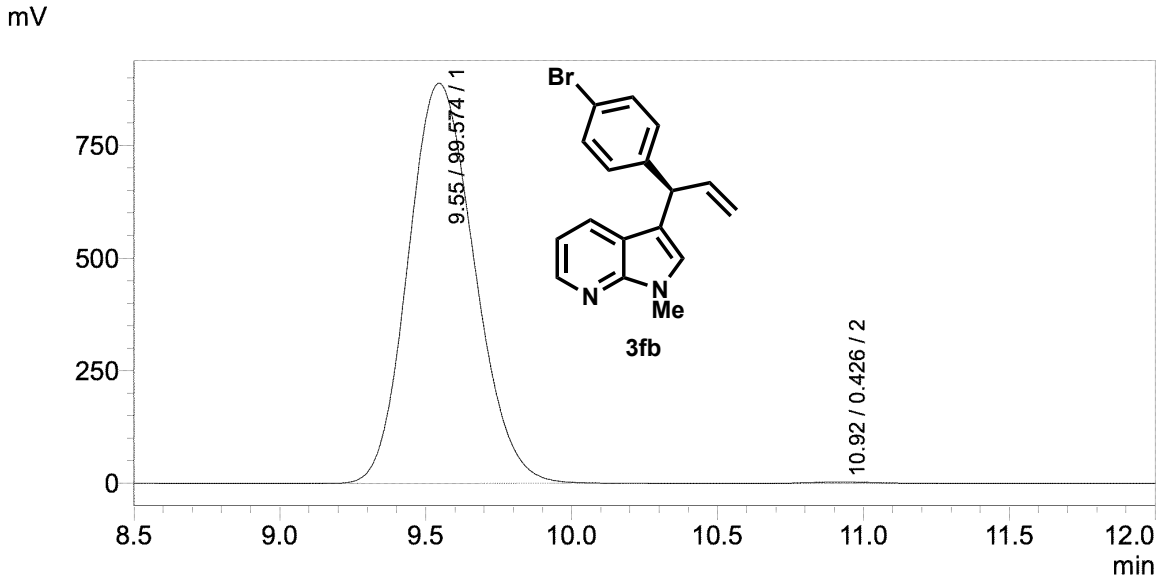
Peak#	Ret. Time	Area%
1	5.72	99.908
2	6.48	0.092
Total		100.000



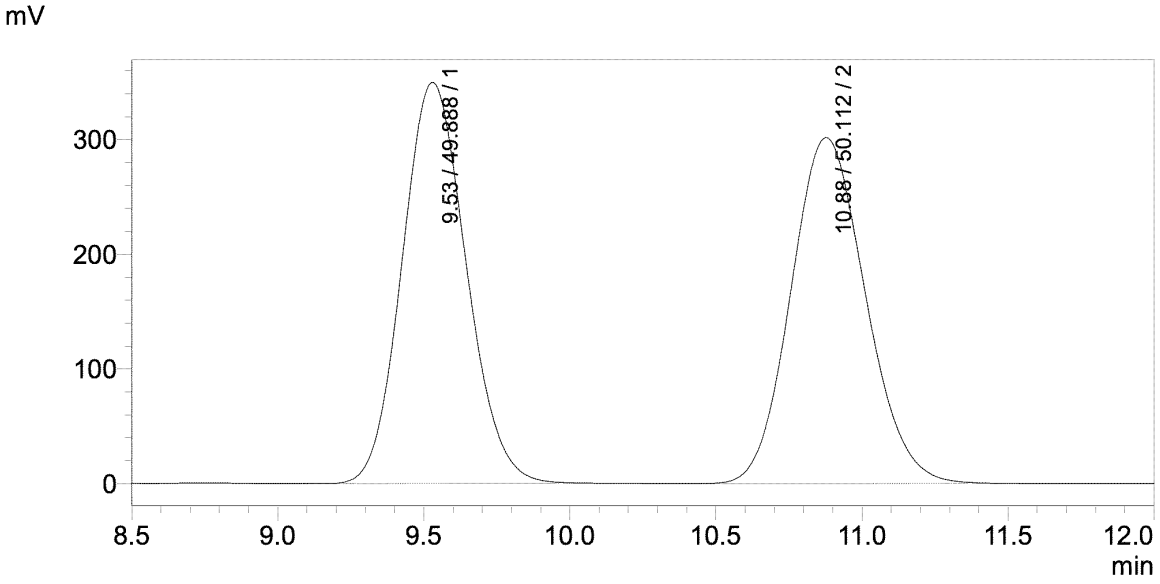
Peak#	Ret. Time	Area%
1	5.69	50.177
2	6.42	49.823
Total		100.000


Br
N
N
Me
3fb
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(-0.00447[ppm], 0[ppm])
Filename = TSREV_o2 matsui 144_proto
Author = delta
Experiment = proton.jxp
Sample_Id = o2 matsui 144
Solvent = CHLOROFORM-D
Actual_Start_Time = 4-SEP-2019 19:35:48
Revision_Time = 17-FEB-2021 13:37:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 30
Temp_Get = 23.4[dC]
X_90_Width = 7.2[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

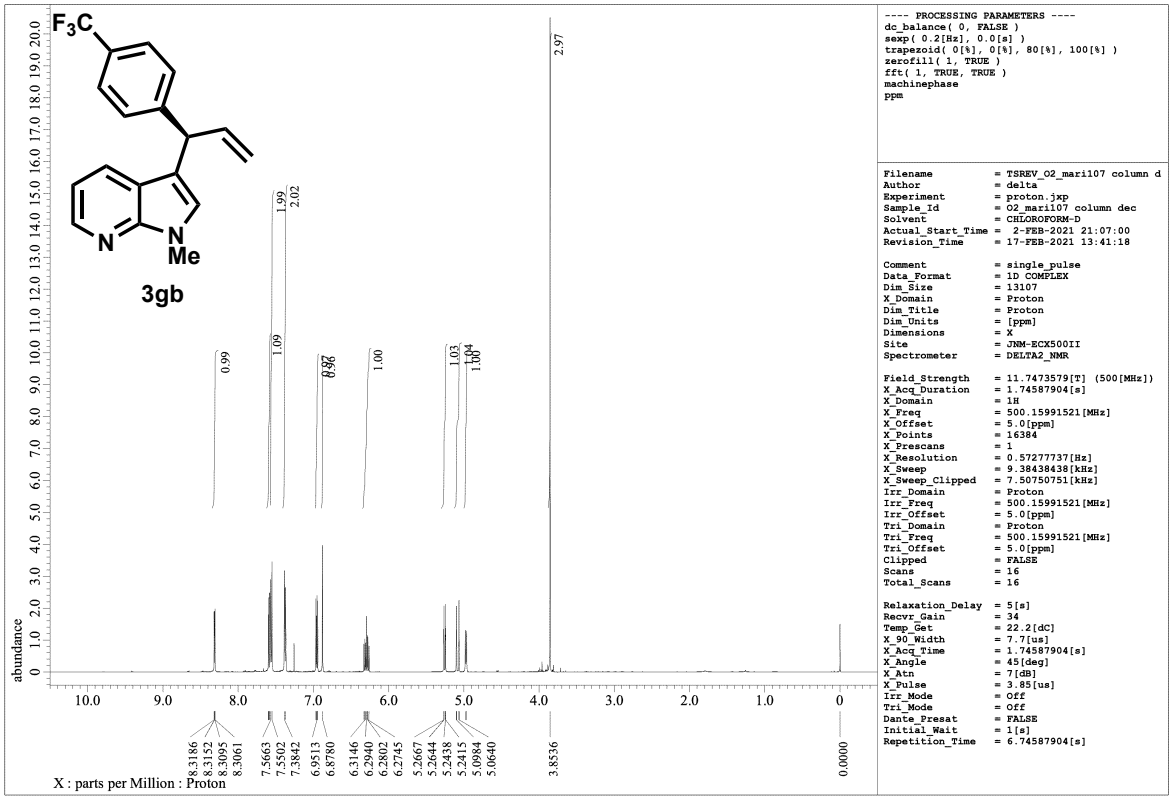

Br
N
N
Me
3fb
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(77.13663[ppm], 77[ppm])
Filename = o2 matsui 144_carbon-2.jd
Author = delta
Experiment = carbon.jxp
Sample_Id = o2 matsui 144
Solvent = CHLOROFORM-D
Actual_Start_Time = 4-SEP-2019 19:38:02
Revision_Time = 17-FEB-2021 13:39:08
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.7[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.9[dB]
Irr_Atn_Noe = 28.9[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	9.55	99.574
2	10.92	0.426
Total		100.000



Peak#	Ret. Time	Area%
1	9.53	49.888
2	10.88	50.112
Total		100.000



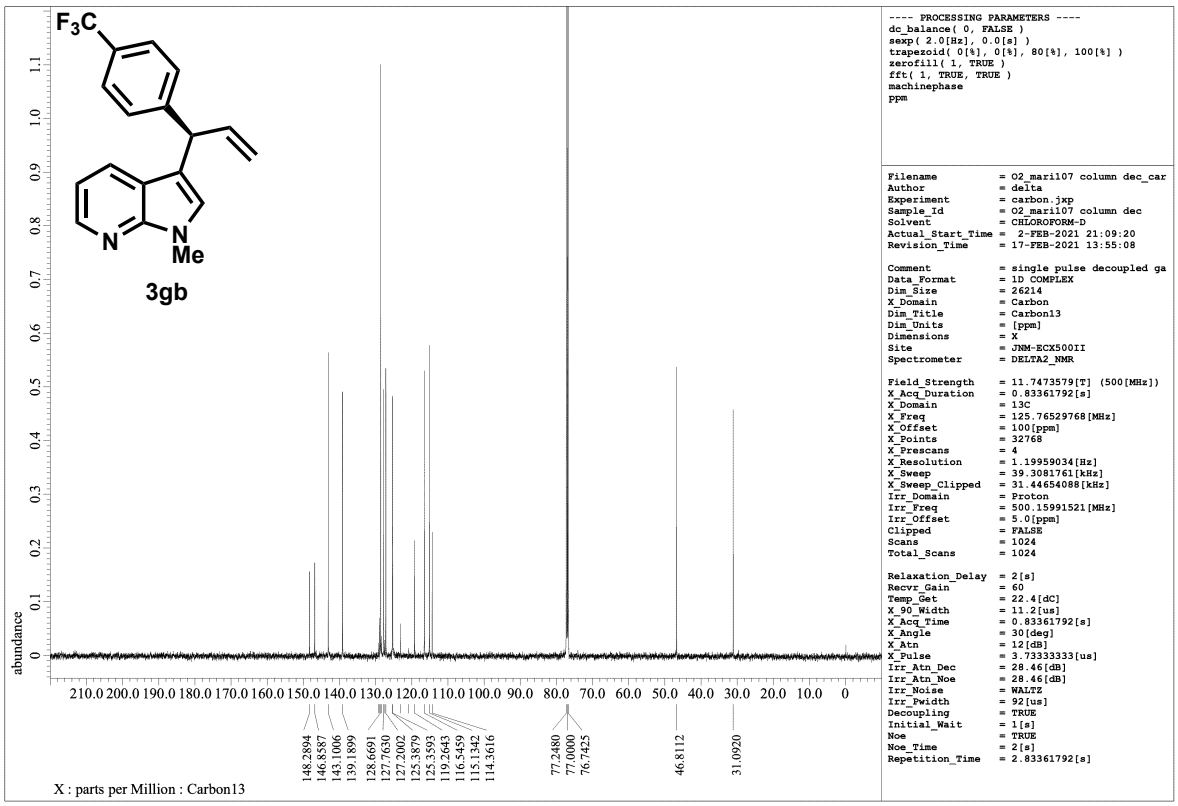

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = TSREV_O2_mari107 column d
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari107 column dec
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  2-FEB-2021 21:07:00
Revision_Time       = 17-FEB-2021 13:41:18

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 22.2[dC]
X_90_Width          = 7.7[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 7[dB]
X_Pulse             = 3.85[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



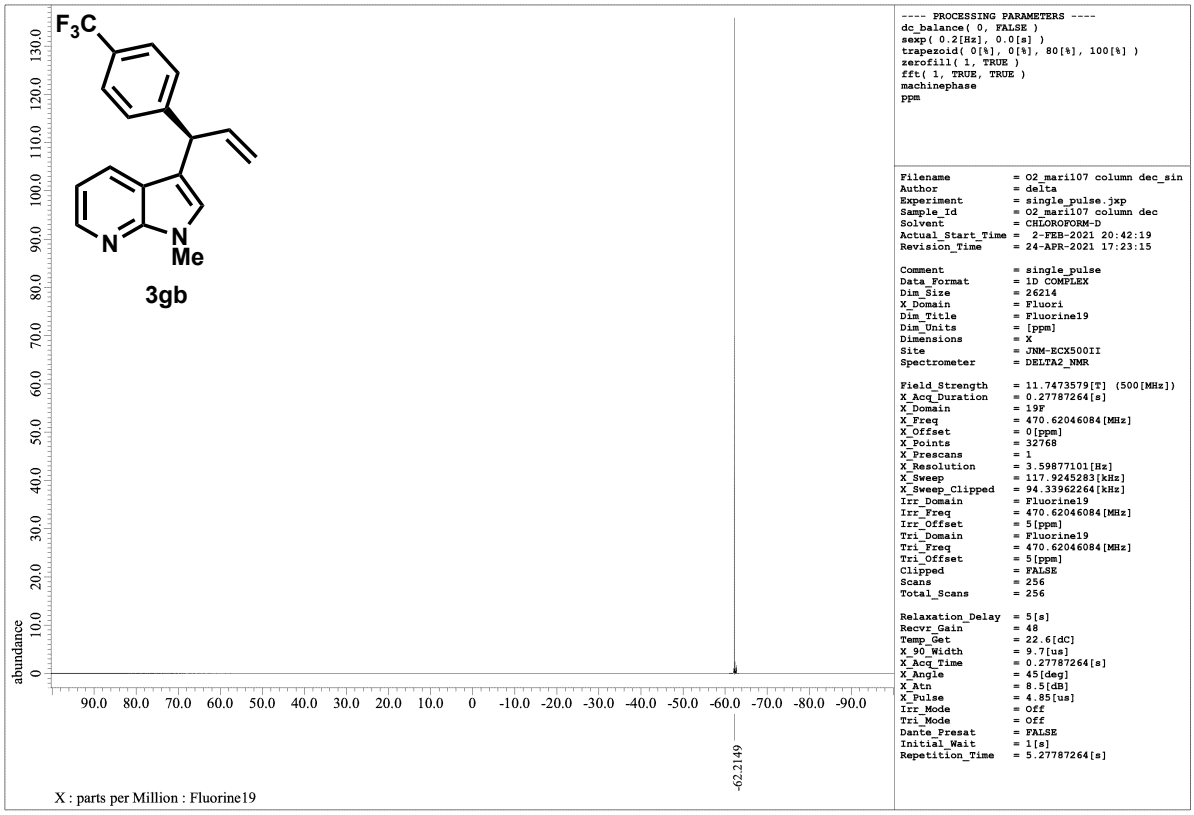

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

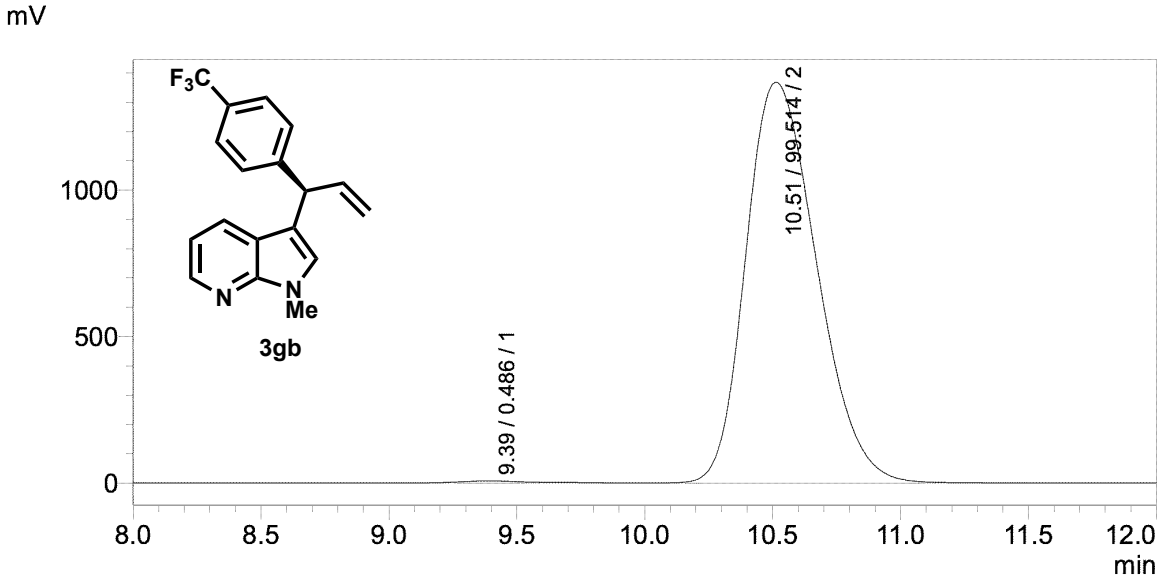
Filename            = O2_mari107 column dec_car
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari107 column dec
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  2-FEB-2021 21:09:20
Revision_Time       = 17-FEB-2021 13:55:08

Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

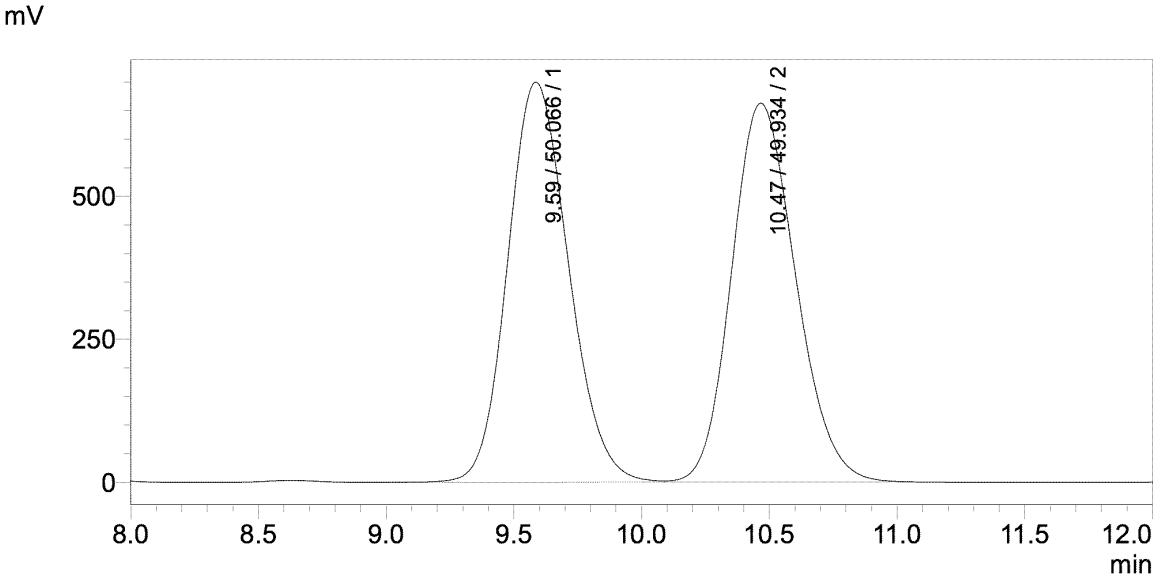
Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offfset           = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.4[dC]
X_90_Width          = 11.2[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.73333333[us]
Irr_Atn_Dec         = 28.46[dB]
Irr_Atn_Noe         = 28.46[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

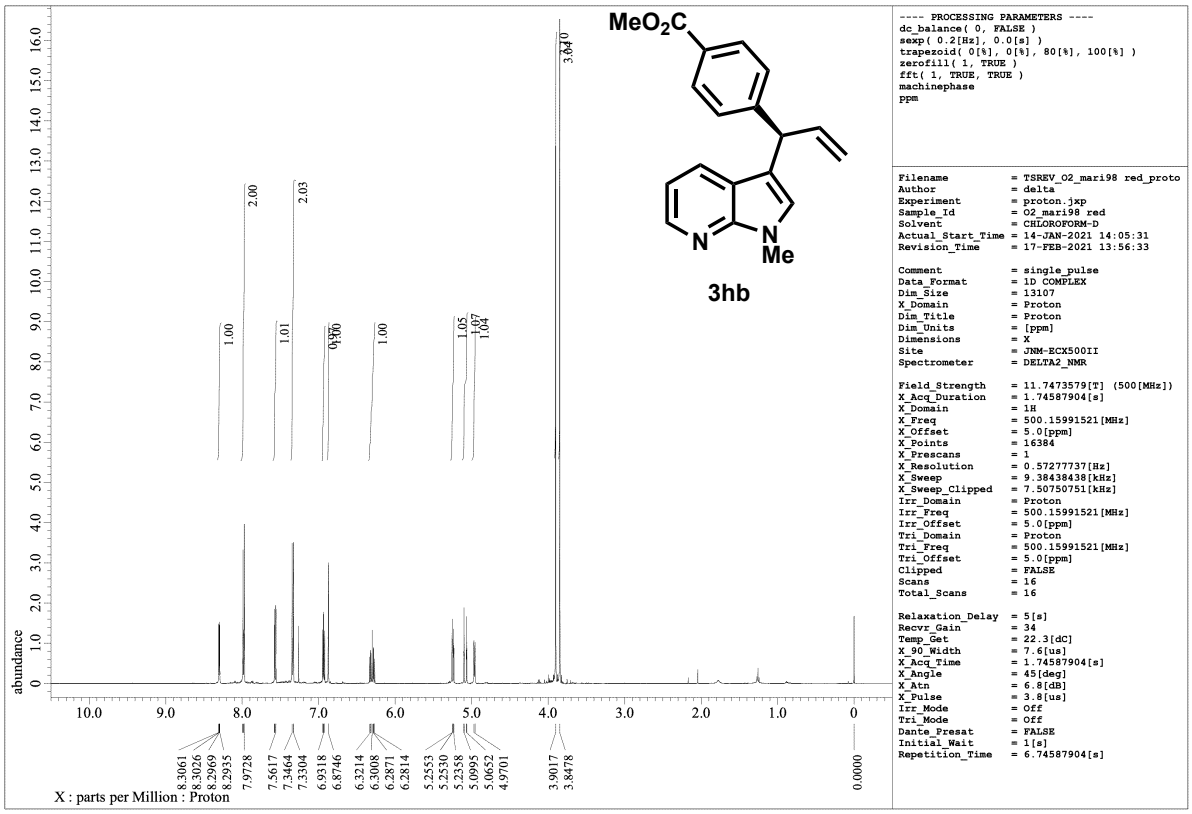

F3C
N
N
Me
3gb
abundance
90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0 -30.0 -40.0 -50.0 -60.0 -70.0 -80.0 -90.0
-62.2149
X : parts per Million : Fluorine19
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari107 column dec_sin
Author = delta
Experiment = single_pulse.jxp
Sample_Id = O2_mari107 column dec
Solvent = CHLOROFORM-D
Actual_Start_Time = 2-FEB-2021 20:42:19
Revision_Time = 24-APR-2021 17:23:15
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Fluori
Dim_Title = Fluorine19
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.27787264[s]
X_Domain = 19F
X_Freq = 470.62046084[MHz]
X_Offset = 0[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 3.59877101[Hz]
X_Sweep = 117.9245283[kHz]
X_Sweep_Clipped = 94.33962264[kHz]
Irr_Domain = Fluorine19
Irr_Freq = 470.62046084[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Fluorine19
Tri_Freq = 470.62046084[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 22.6[dC]
X_90_Width = 9.7[us]
X_Acq_Time = 0.27787264[s]
X_Angle = 45[deg]
X_Atn = 8.5[dB]
X_Pulse = 4.85[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 5.27787264[s]



Peak#	Ret. Time	Area%
1	9.39	0.486
2	10.51	99.514
Total		100.000



Peak#	Ret. Time	Area%
1	9.59	50.066
2	10.47	49.934
Total		100.000



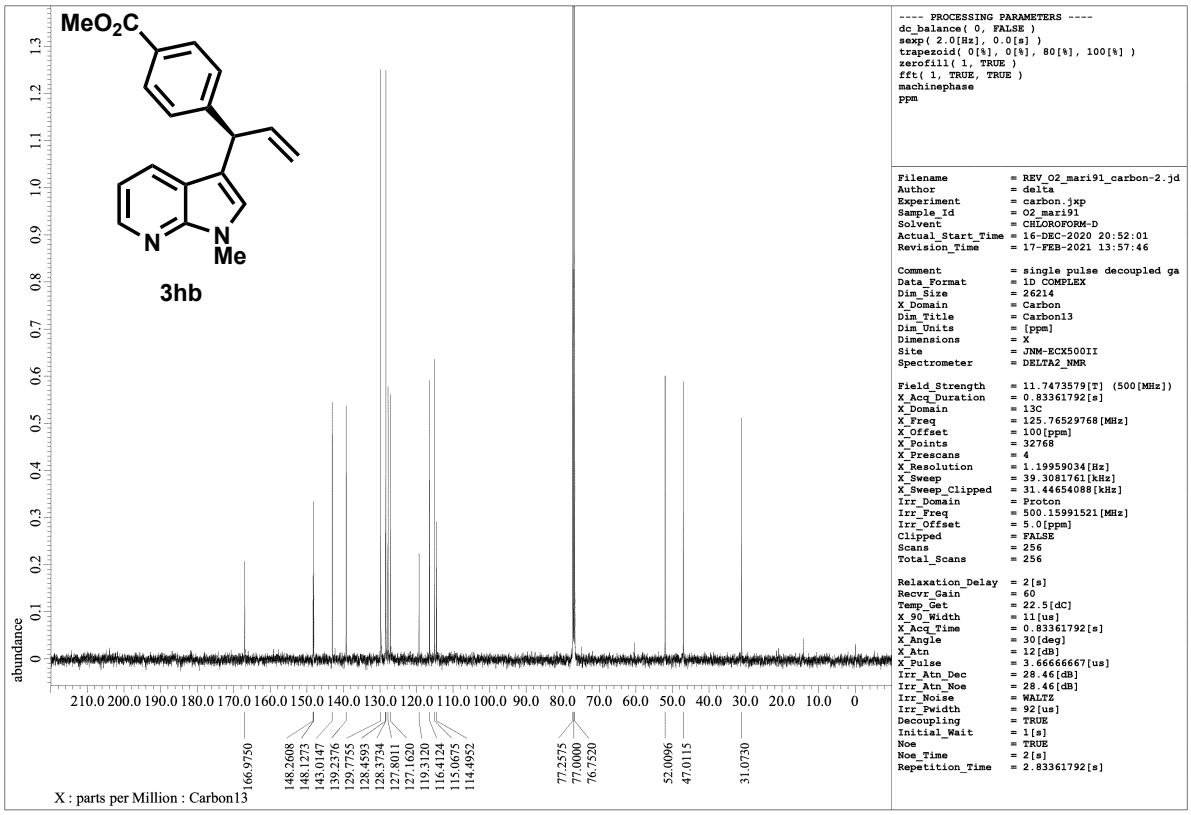

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = TSREV_O2_mari98 red_proto
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari98 red
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 14-JAN-2021 14:05:31
Revision_Time       = 17-FEB-2021 13:56:33

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 22.3[dC]
X_90_Width          = 7.6[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.8[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



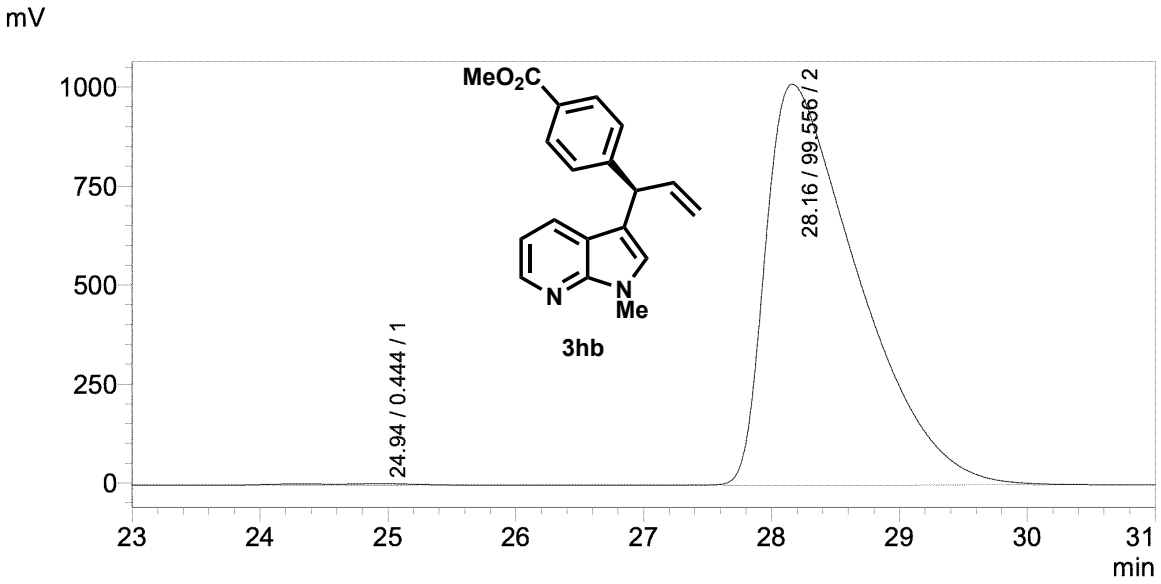

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = REV_O2_mari91_carbon-2.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari91
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-DEC-2020 20:52:01
Revision_Time       = 17-FEB-2021 13:57:46

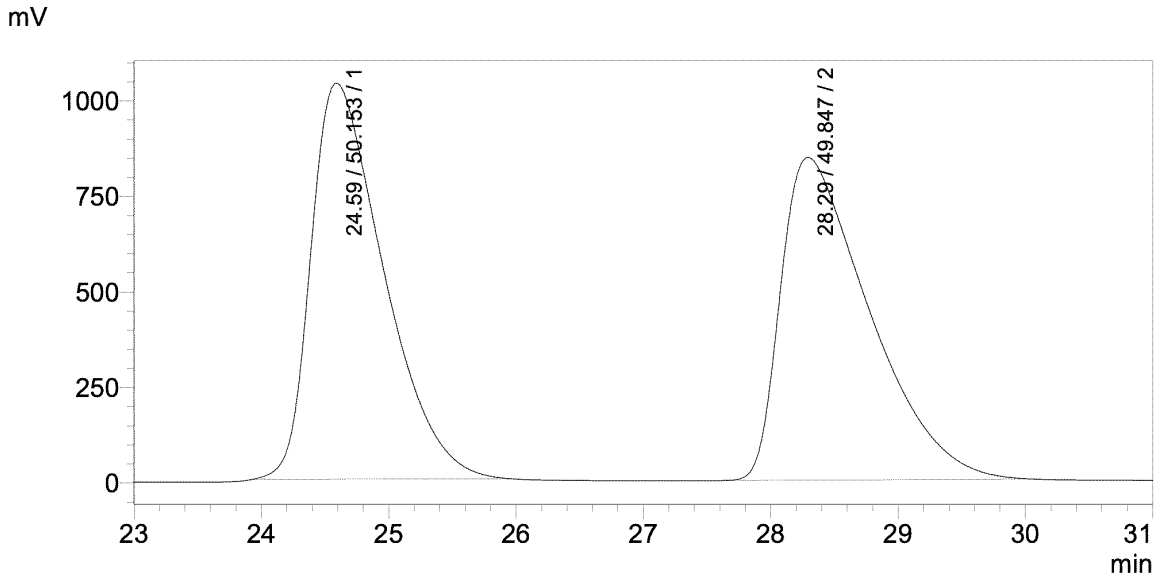
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

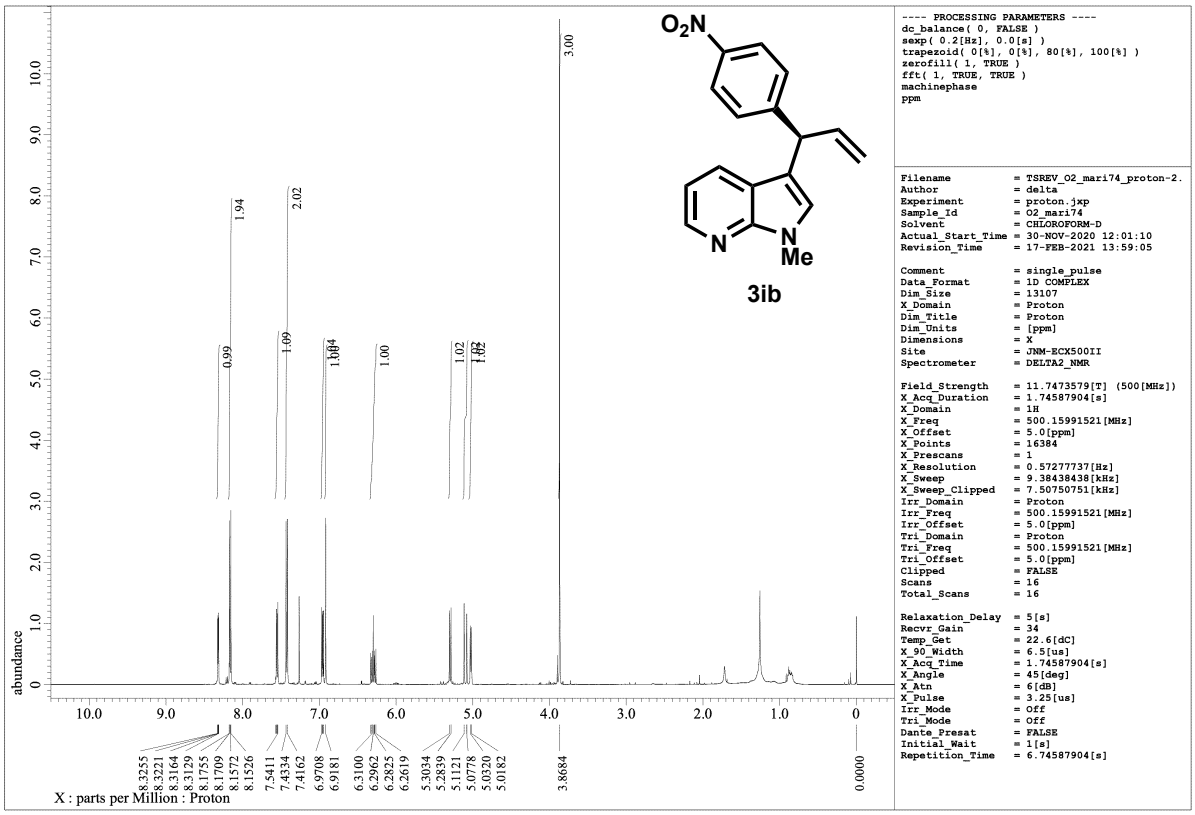
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.5[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.46[dB]
Irr_Atn_Noe         = 28.46[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

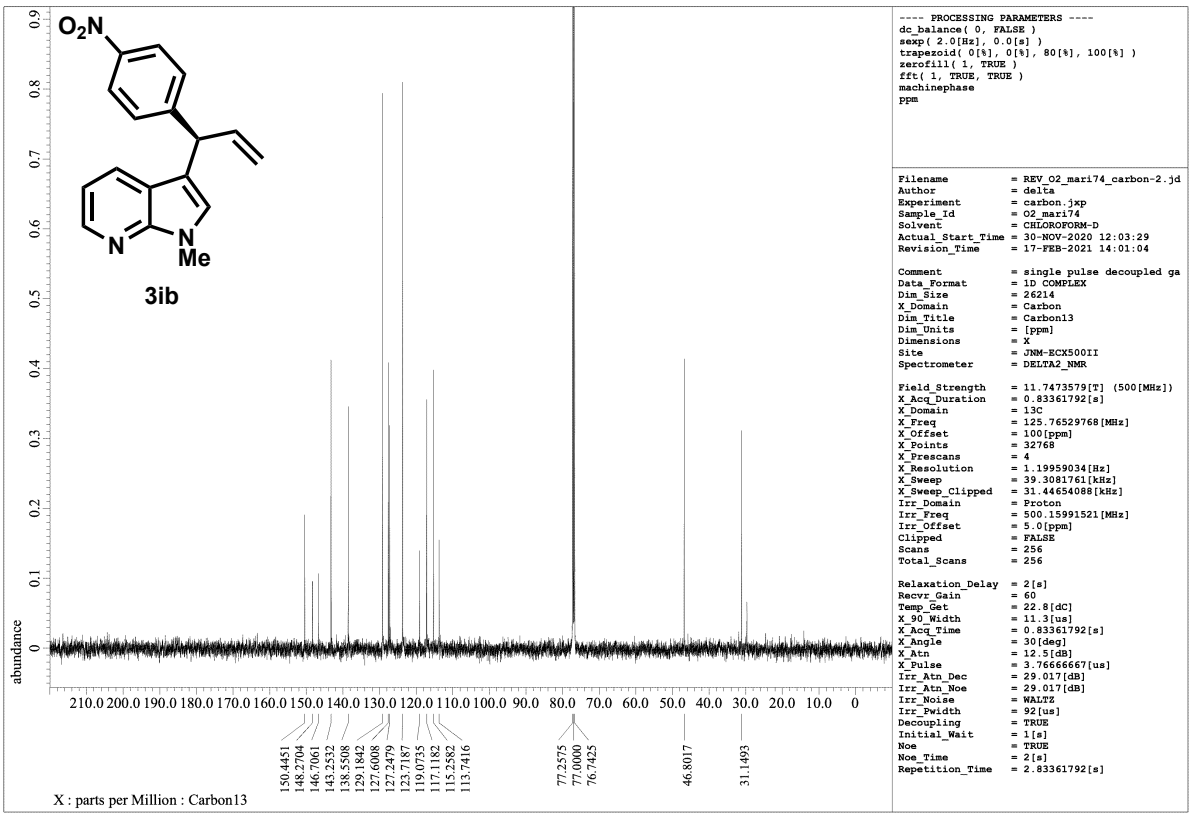


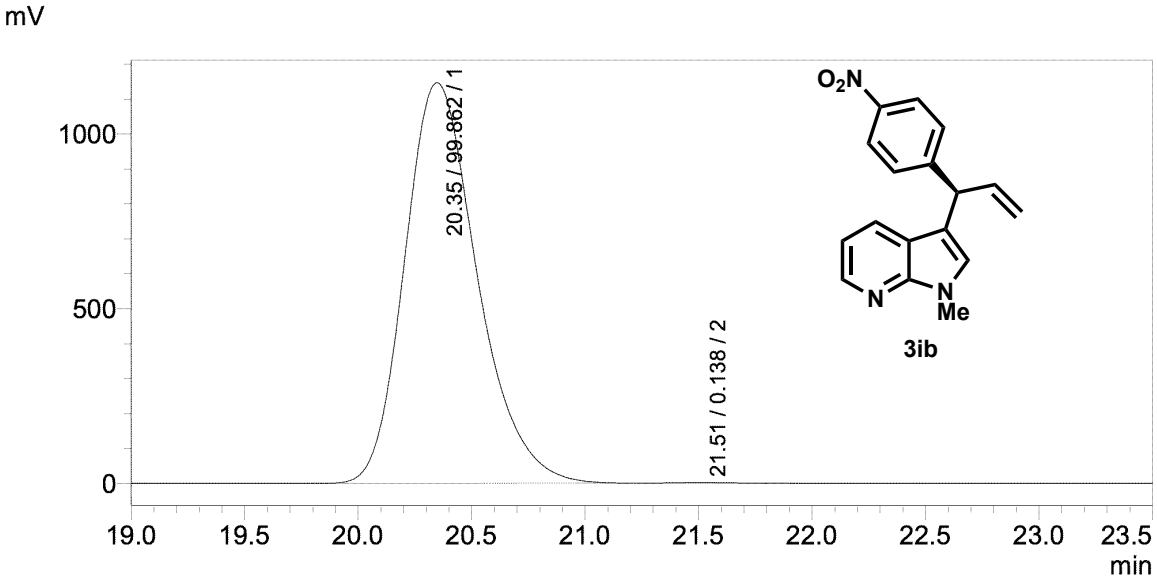
Peak#	Ret. Time	Area%
1	24.94	0.444
2	28.16	99.556
Total		100.000



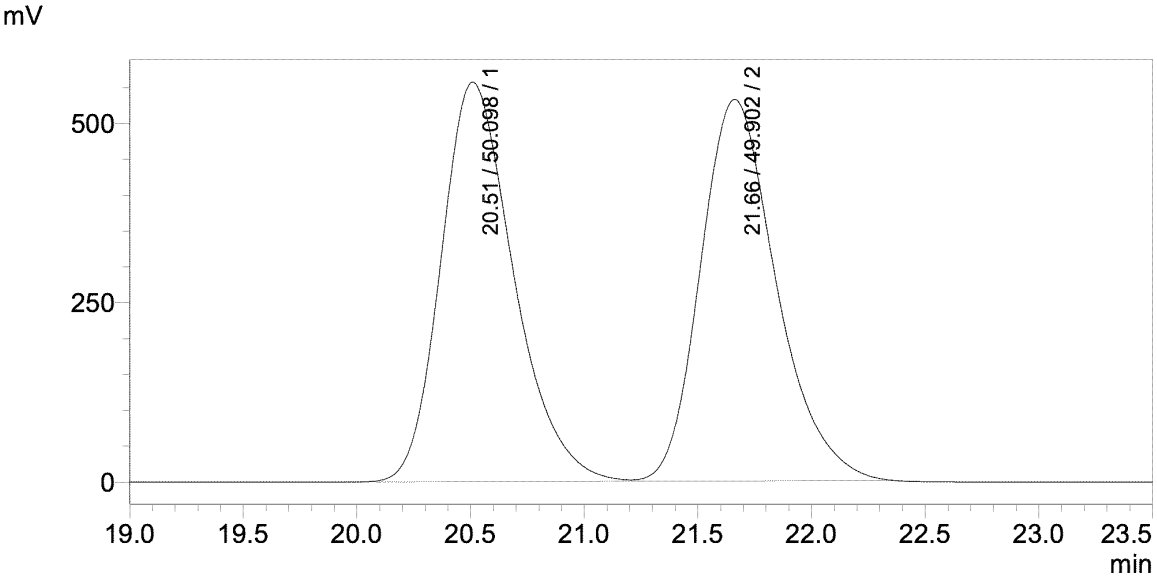
Peak#	Ret. Time	Area%
1	24.59	50.153
2	28.29	49.847
Total		100.000


O2N
N
N
Me
3ib
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = TSREV_O2_mari74_proton-2.
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari74
Solvent = CHLOROFORM-D
Actual_Start_Time = 30-NOV-2020 12:01:10
Revision_Time = 17-FEB-2021 13:59:05
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 22.6[dC]
X_90_Width = 6.5[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6[dB]
X_Pulse = 3.25[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

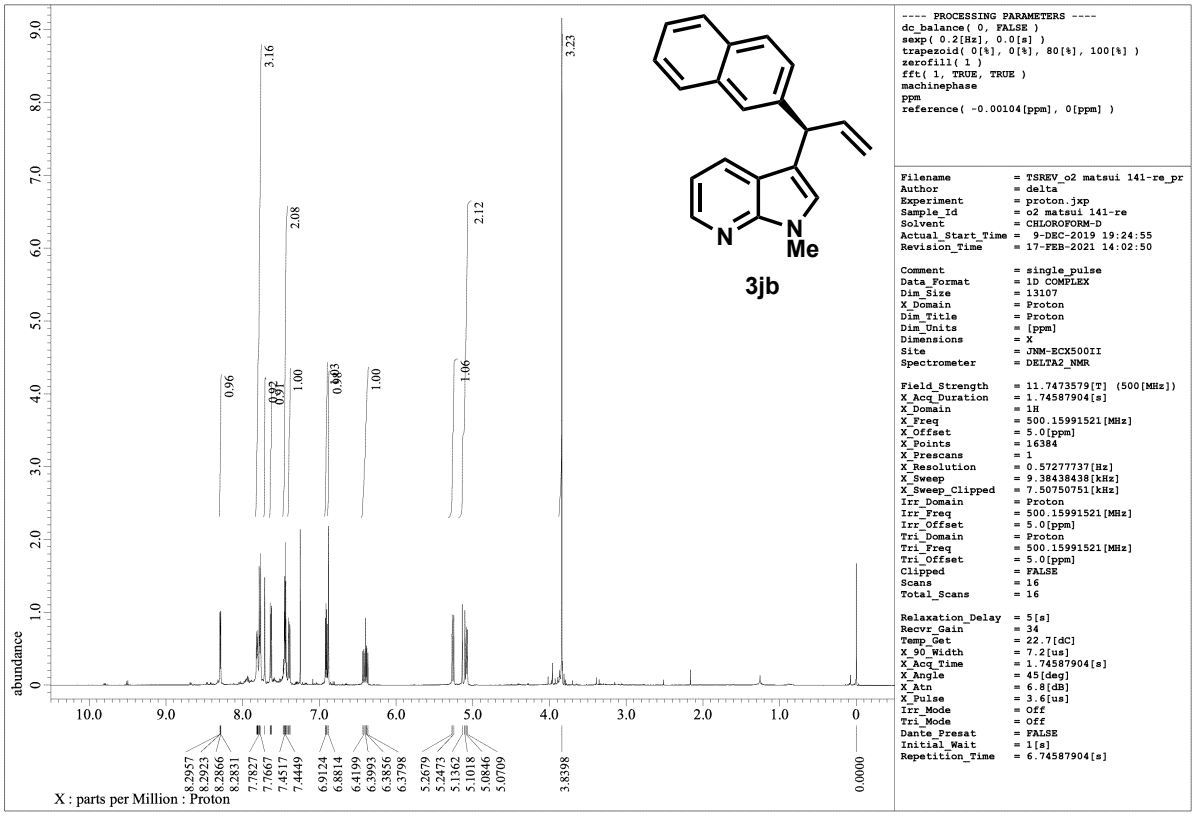

O2N
N
N
Me
3ib
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = REV_O2_mari74_carbon-2.jd
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari74
Solvent = CHLOROFORM-D
Actual_Start_Time = 30-NOV-2020 12:03:29
Revision_Time = 17-FEB-2021 14:01:04
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.8[dC]
X_90_Width = 11.3[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12.5[dB]
X_Pulse = 3.76666667[us]
Irr_Atn_Dec = 29.017[dB]
Irr_Atn_Noe = 29.017[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	20.35	99.862
2	21.51	0.138
Total		100.000



Peak#	Ret. Time	Area%
1	20.51	50.098
2	21.66	49.902
Total		100.000



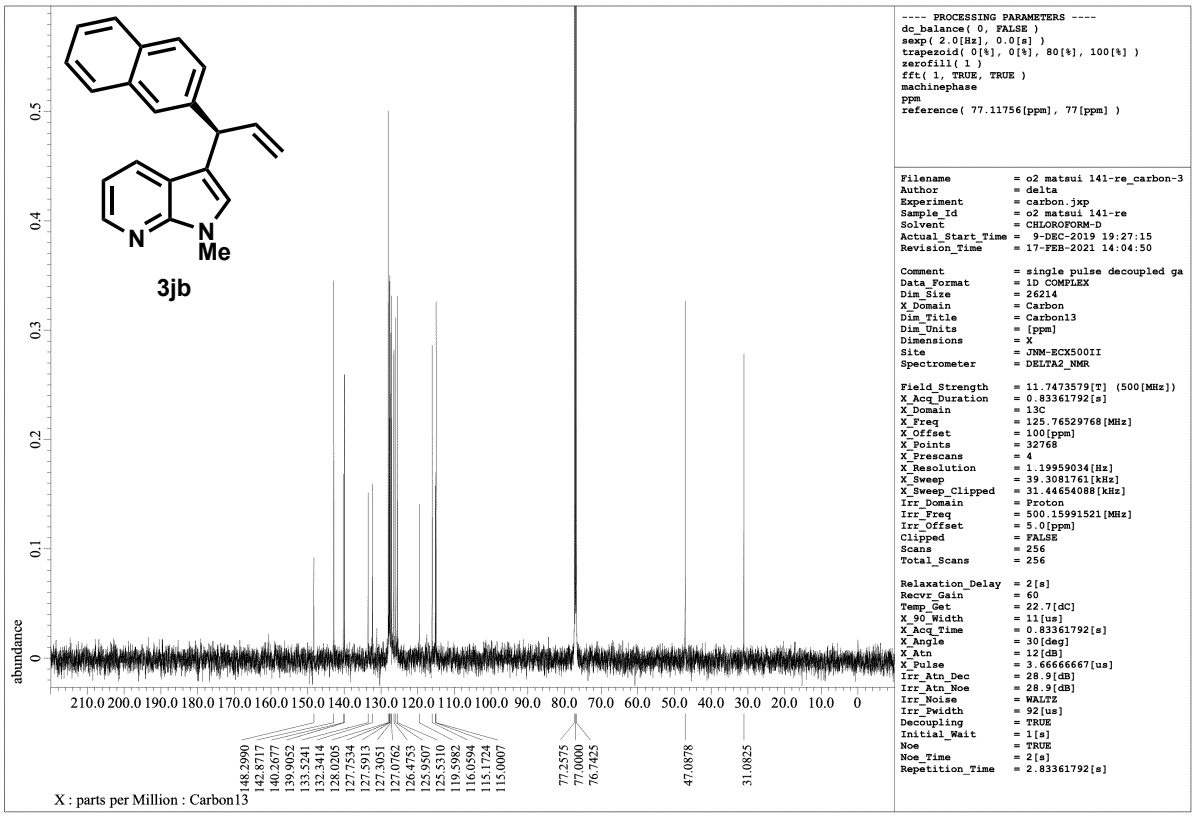

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.00104[ppm], 0[ppm] )

Filename            = TSREV_o2 matsui 141-re_pr
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 matsui 141-re
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  9-DEC-2019 19:24:55
Revision_Time       = 17-FEB-2021 14:02:50

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 22.7[dC]
X_90_Width          = 7.2[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.6[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



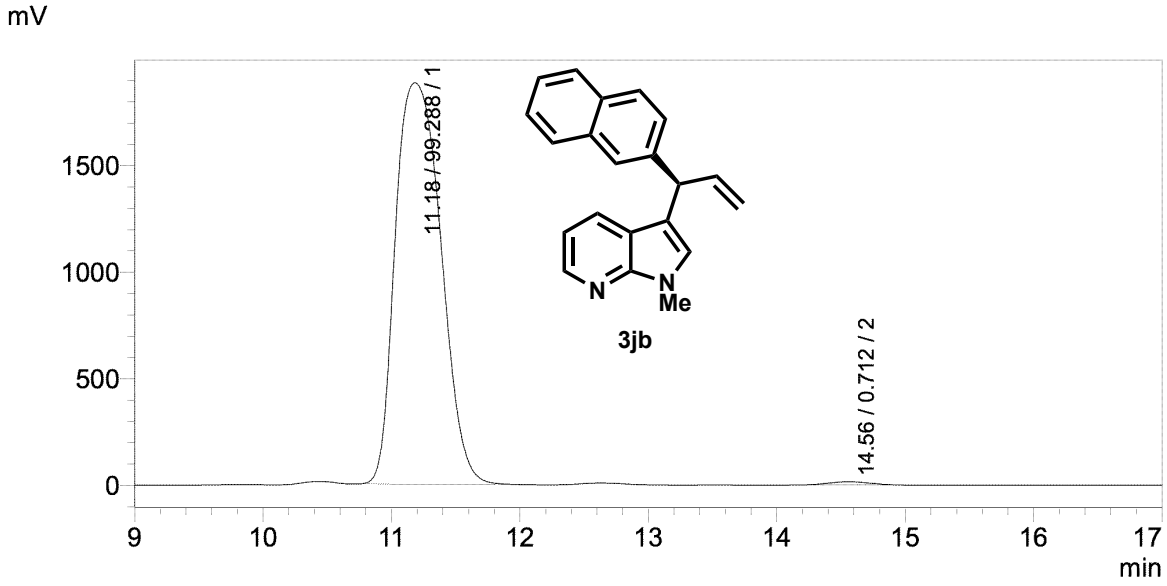

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.11756[ppm], 77[ppm] )

Filename            = o2 matsui 141-re_carbon-3
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 matsui 141-re
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  9-DEC-2019 19:27:15
Revision_Time       = 17-FEB-2021 14:04:50

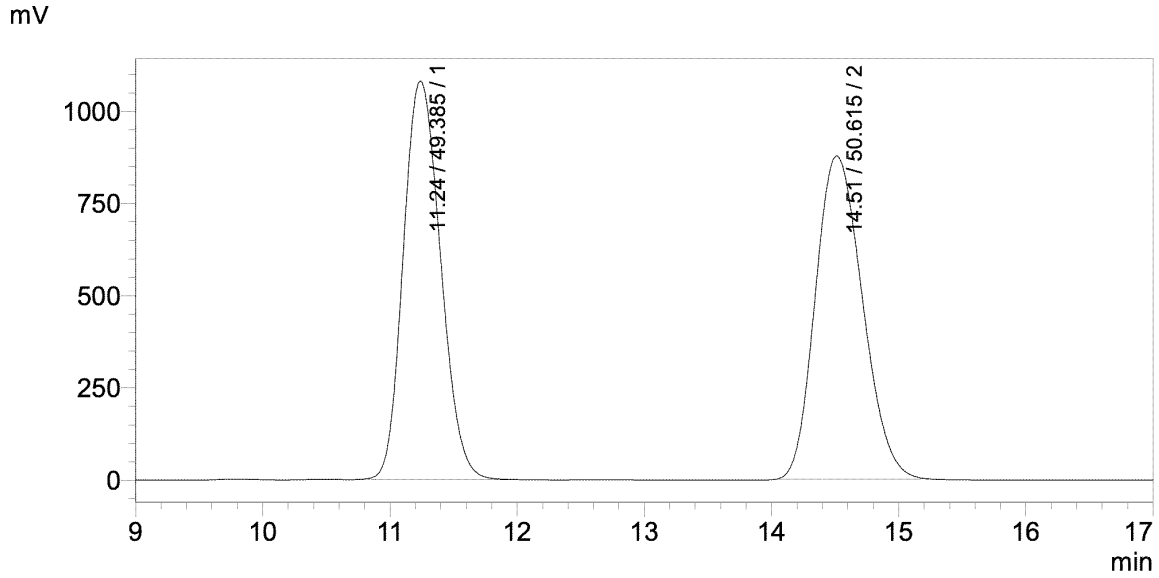
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

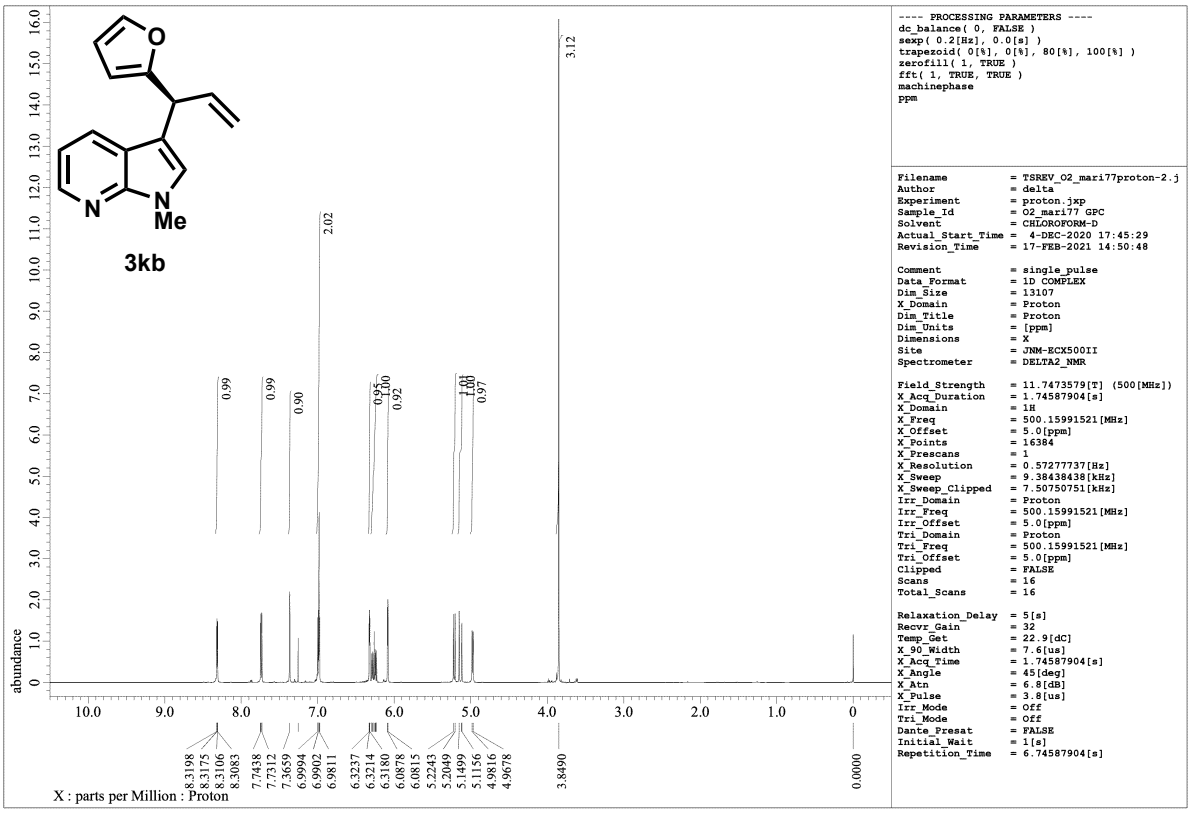
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.7[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.9[dB]
Irr_Atn_Noe         = 28.9[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```



Peak#	Ret. Time	Area%
1	11.18	99.288
2	14.56	0.712
Total		100.000



Peak#	Ret. Time	Area%
1	11.24	49.385
2	14.51	50.615
Total		100.000



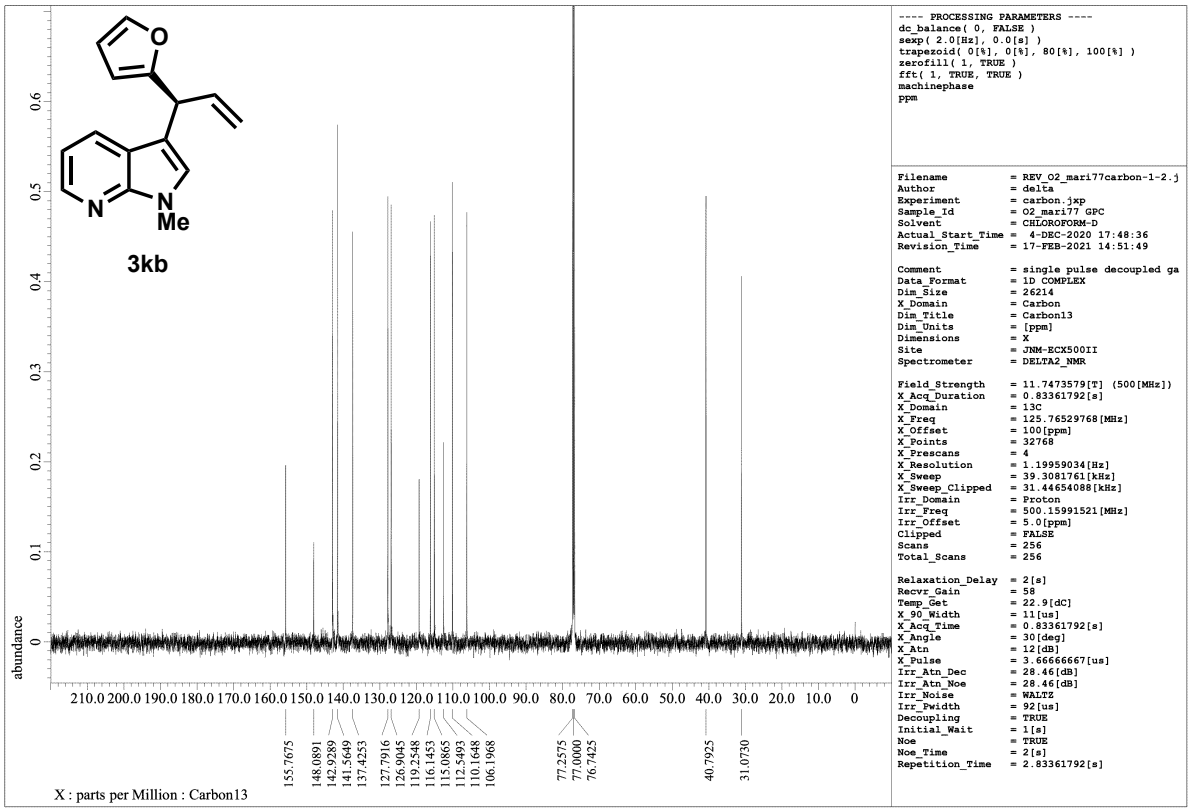

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = TSREV_O2_mari77proton-2.j
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari77 GPC
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-DEC-2020 17:45:29
Revision_Time       = 17-FEB-2021 14:50:48

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 32
Temp_Get            = 22.9[dC]
X_90_Width          = 7.6[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.8[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



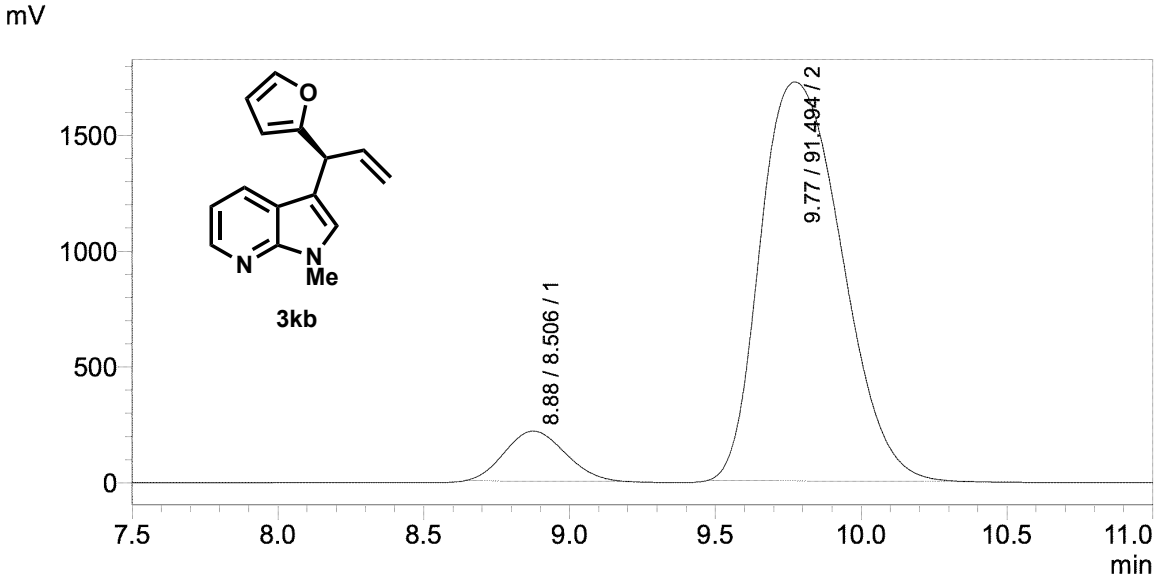

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = REV_O2_mari77carbon-1-2.j
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari77 GPC
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-DEC-2020 17:48:36
Revision_Time       = 17-FEB-2021 14:51:49

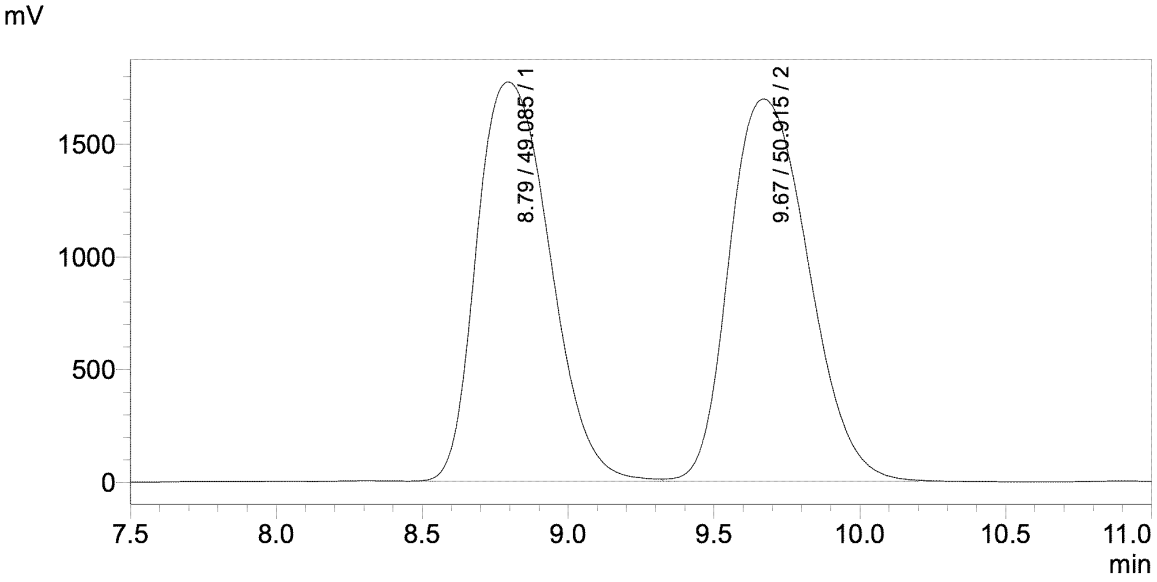
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.4465408[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

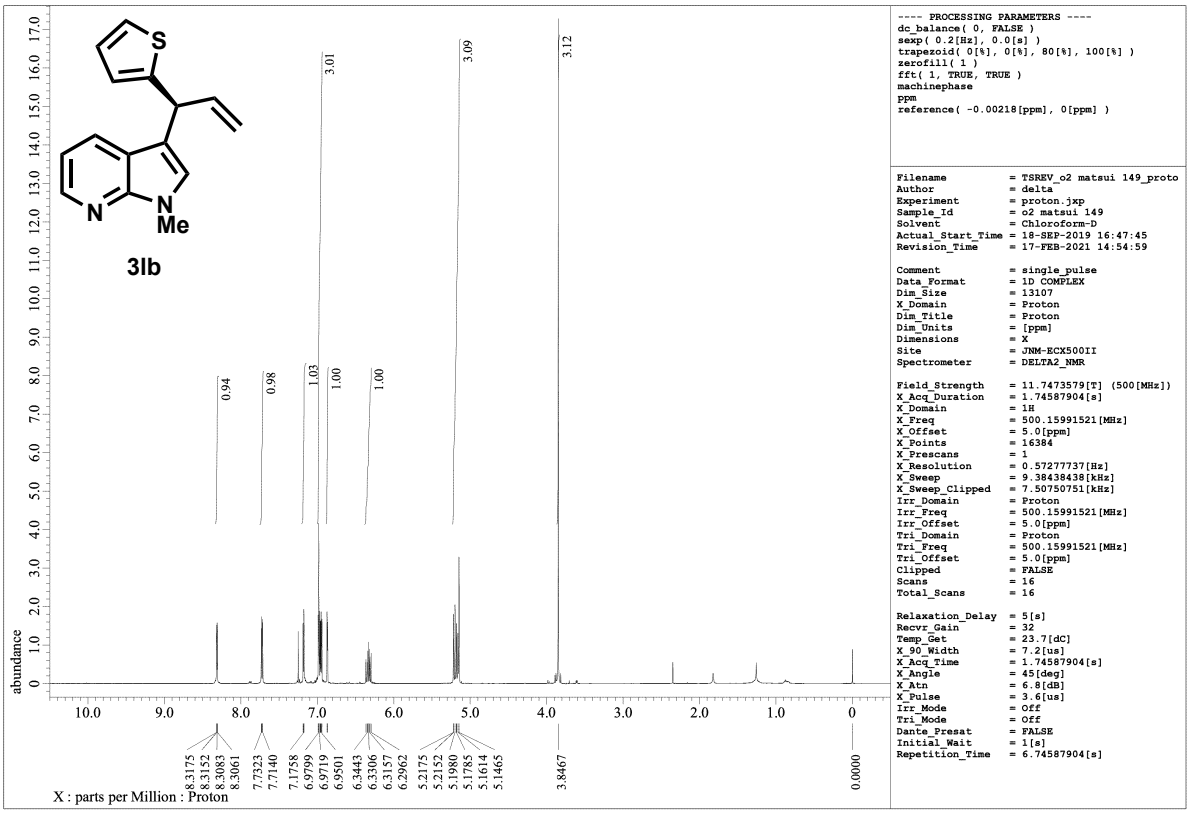
Relaxation_Delay    = 2[s]
Recvr_Gain          = 58
Temp_Get            = 22.9[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.46[dB]
Irr_Atn_Noe         = 28.46[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

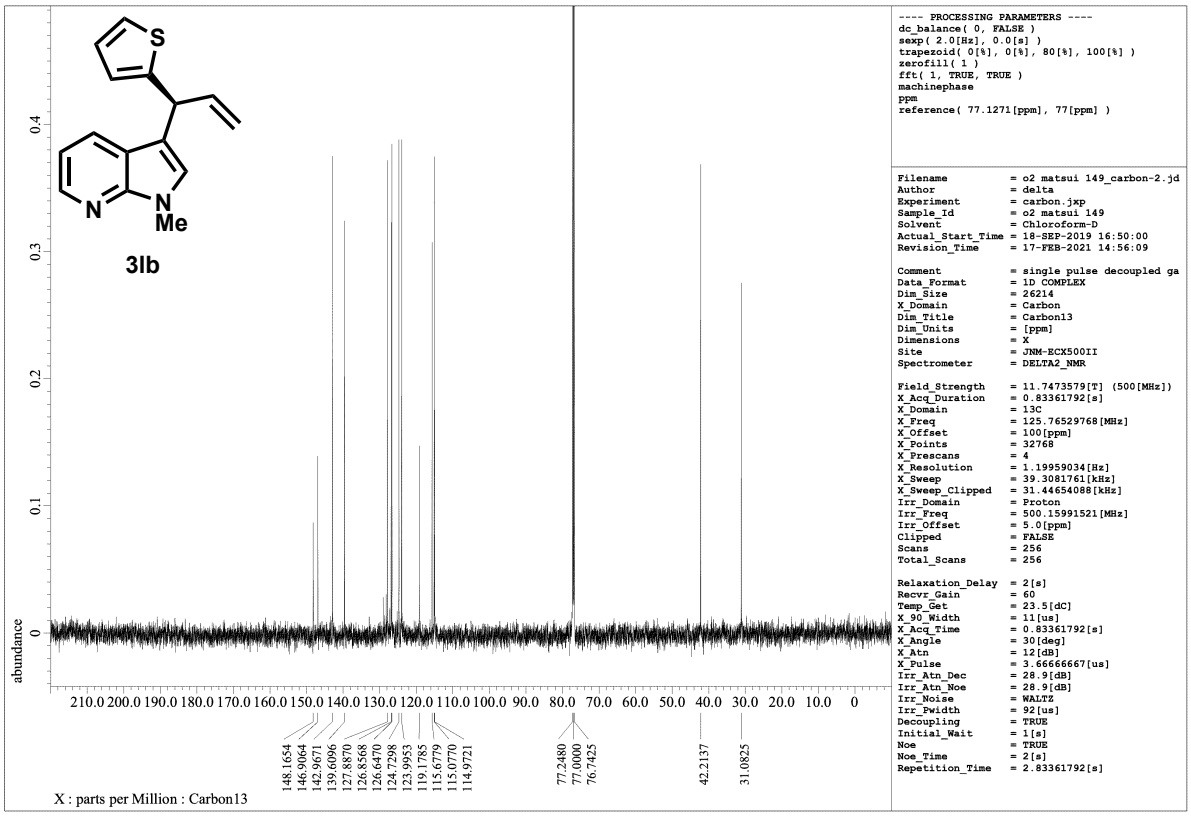


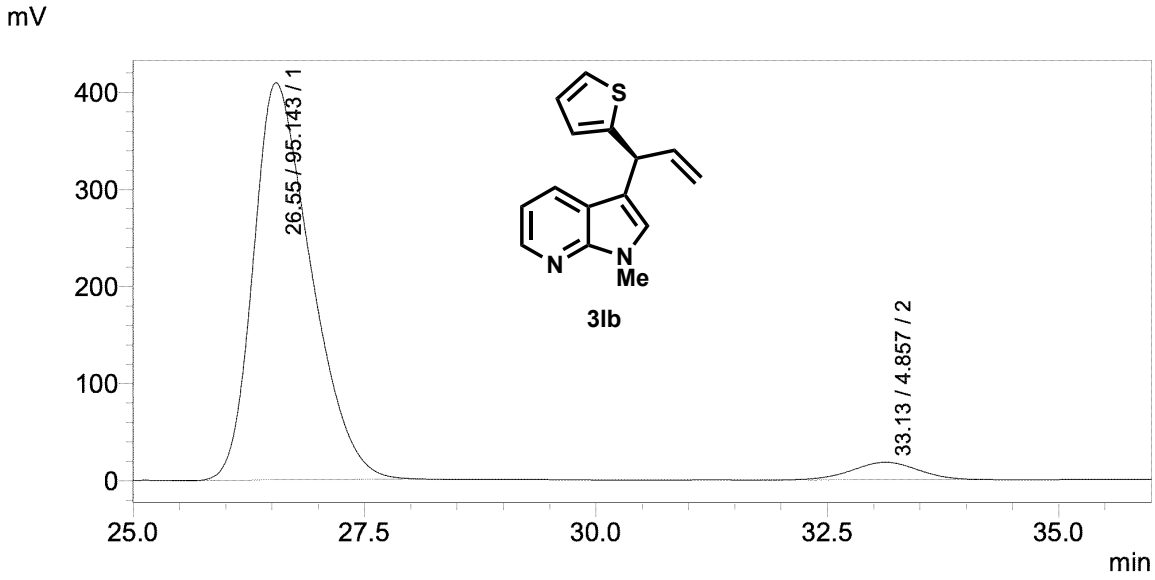
Peak#	Ret. Time	Area%
1	8.88	8.506
2	9.77	91.494
Total		100.000



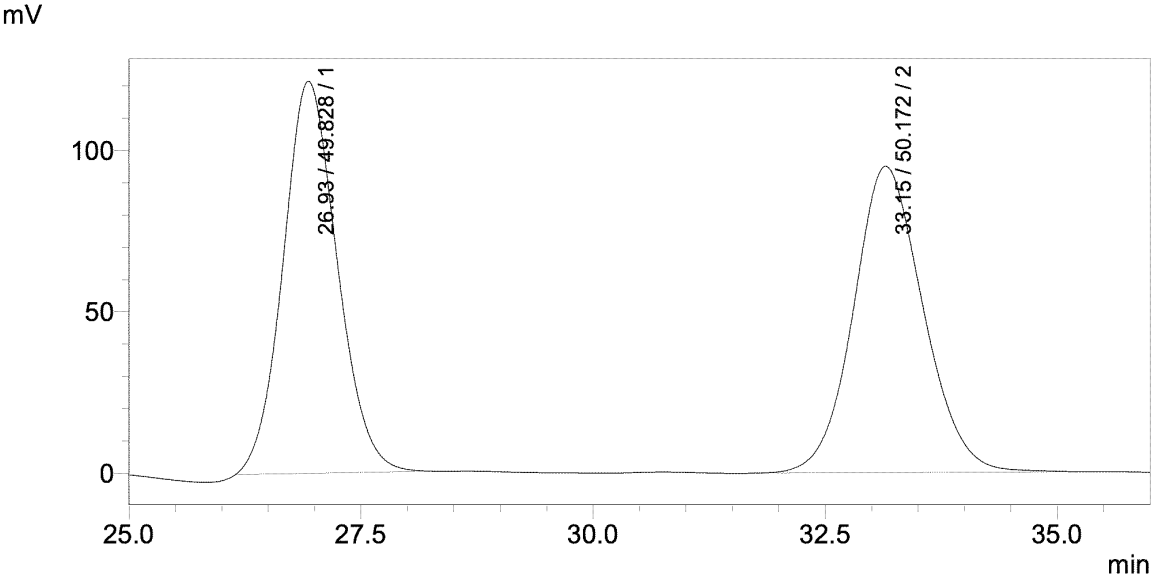
Peak#	Ret. Time	Area%
1	8.79	49.085
2	9.67	50.915
Total		100.000


3Ib
abundance
0.94
0.98
1.03
1.00
1.00
3.01
3.09
3.12
8.3175
8.3152
8.3083
8.3061
7.7323
7.7140
7.1758
6.9799
6.9719
6.9501
6.3443
6.3306
6.3157
6.2962
5.2175
5.2152
5.1980
5.1785
5.1614
5.1465
3.8467
0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(-0.00218[ppm], 0[ppm])
Filename = TSREV_o2 matsui 149_proto
Author = delta
Experiment = proton.jxp
Sample_Id = o2 matsui 149
Solvent = Chloroform-D
Actual_Start_Time = 18-SEP-2019 16:47:45
Revision_Time = 17-FEB-2021 14:54:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 32
Temp_Get = 23.7[dC]
X_90_Width = 7.2[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.6[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

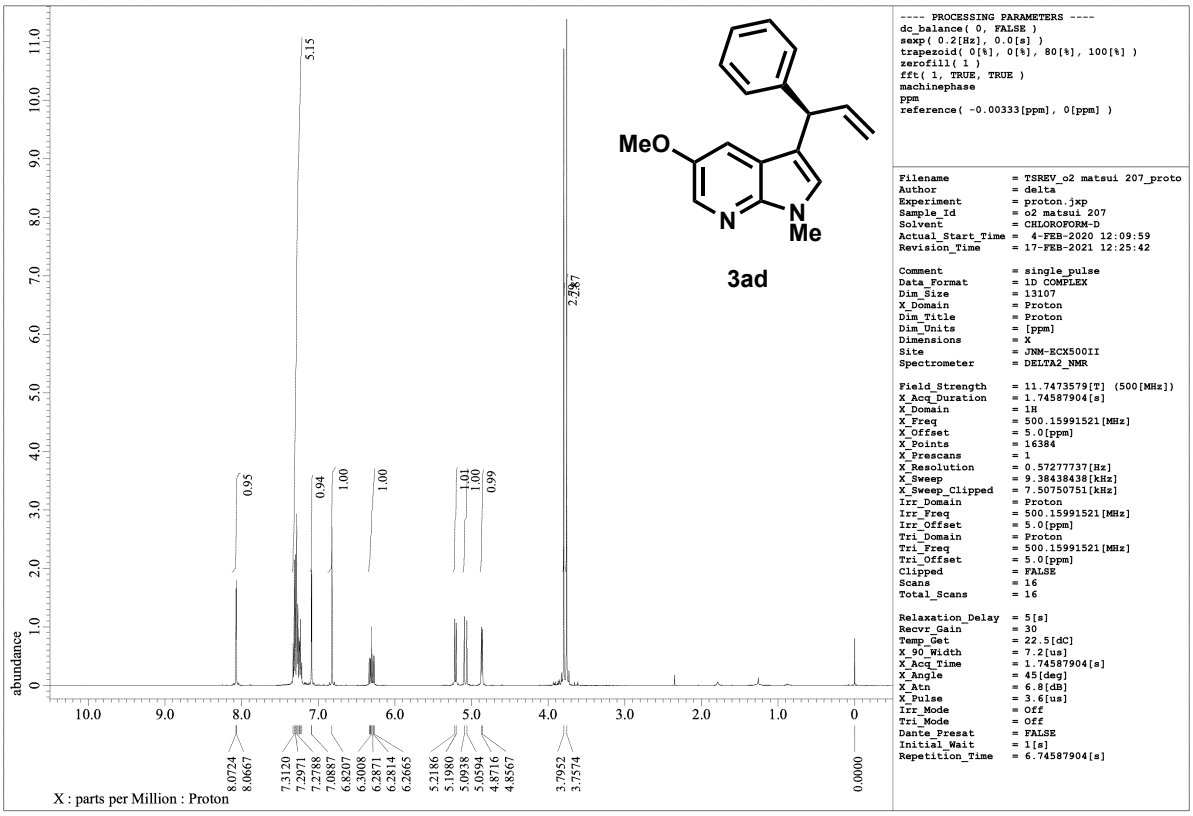

3Ib
abundance
148.1654
146.9064
142.9671
139.6096
127.8870
126.8568
126.6470
124.7298
123.9953
119.1785
115.6779
115.0770
114.9721
77.2480
77.0000
76.7425
42.2137
31.0825
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(77.1271[ppm], 77[ppm])
Filename = o2 matsui 149_carbon-2.jd
Author = delta
Experiment = carbon.jxp
Sample_Id = o2 matsui 149
Solvent = Chloroform-D
Actual_Start_Time = 18-SEP-2019 16:50:00
Revision_Time = 17-FEB-2021 14:56:09
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.5[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.9[dB]
Irr_Atn_Noe = 28.9[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	26.55	95.143
2	33.13	4.857
Total		100.000



Peak#	Ret. Time	Area%
1	26.93	49.828
2	33.15	50.172
Total		100.000



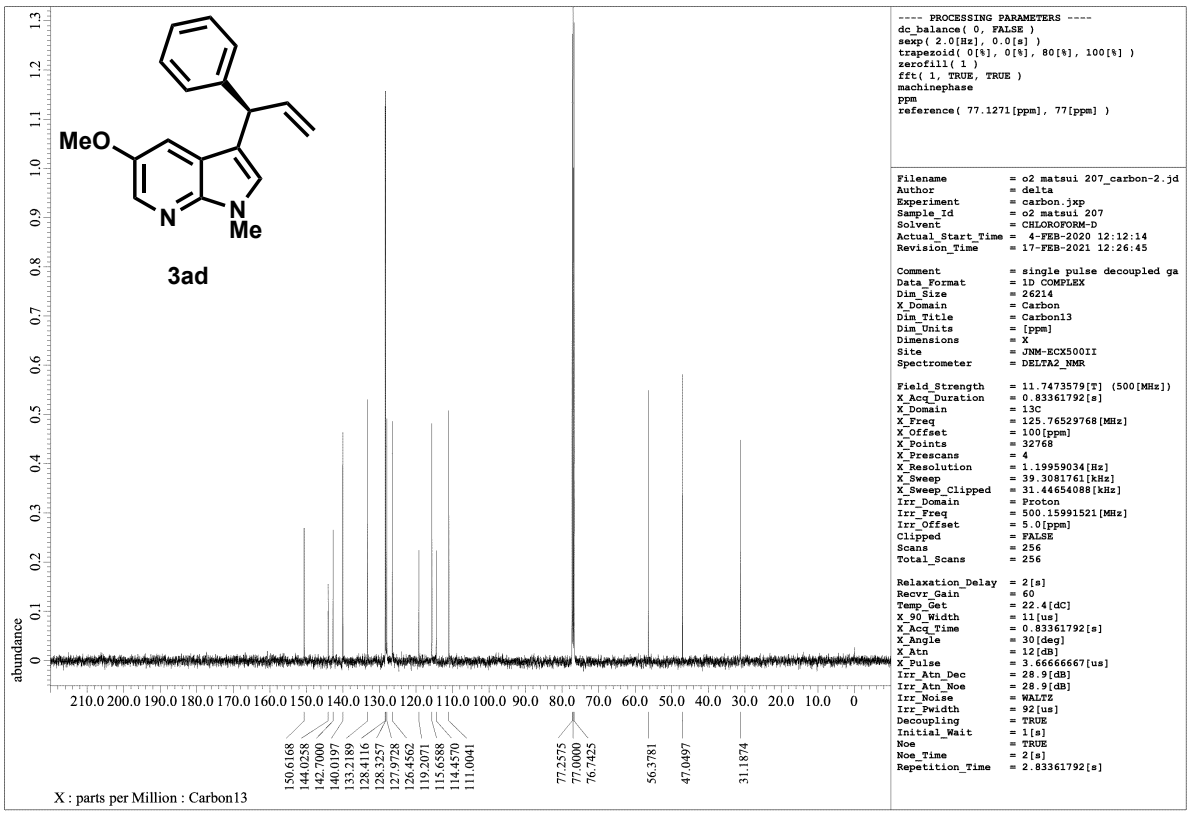

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.00333[ppm], 0[ppm] )

Filename            = TSREV_o2 matsui 207_proto
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 matsui 207
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-FEB-2020 12:09:59
Revision_Time       = 17-FEB-2021 12:25:42

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 22.5[dC]
X_90_Width          = 7.2[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.6[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



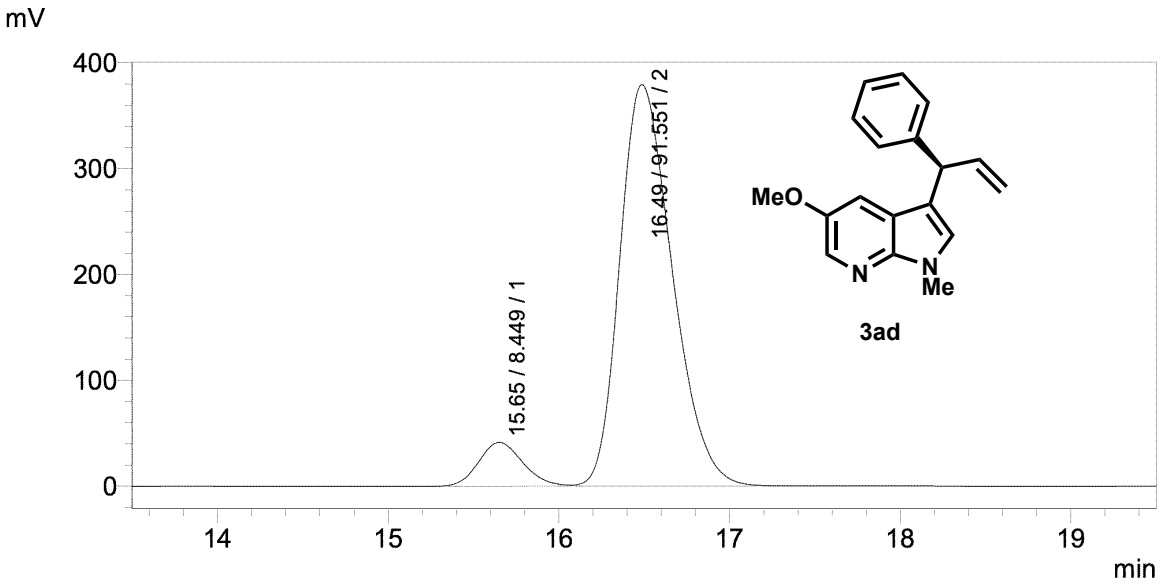

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.1271[ppm], 77[ppm] )

Filename            = o2 matsui 207_carbon-2.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 matsui 207
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-FEB-2020 12:12:14
Revision_Time       = 17-FEB-2021 12:26:45

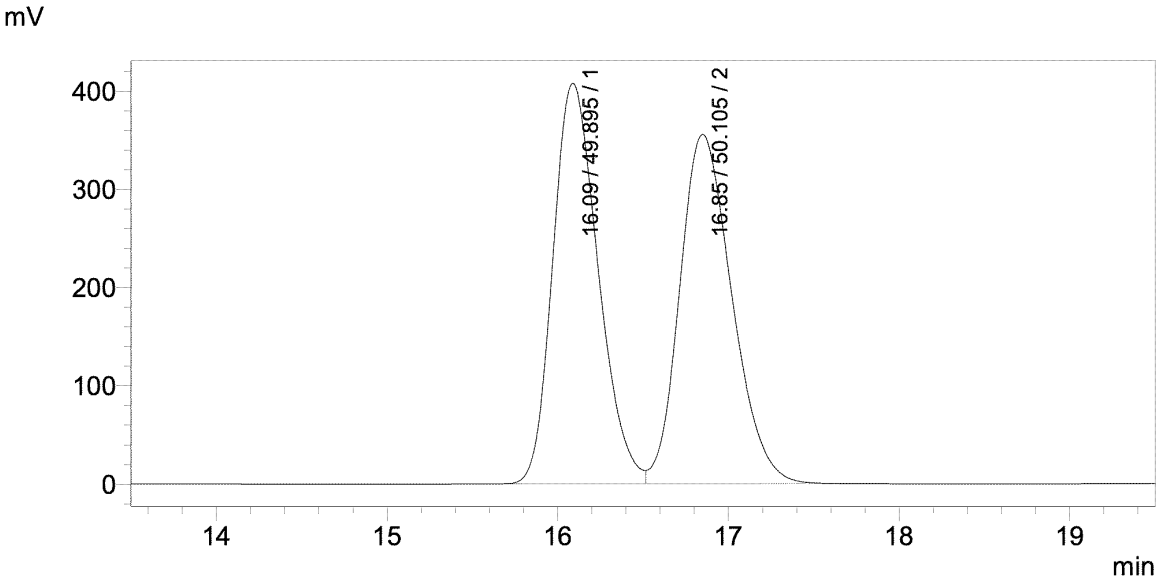
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

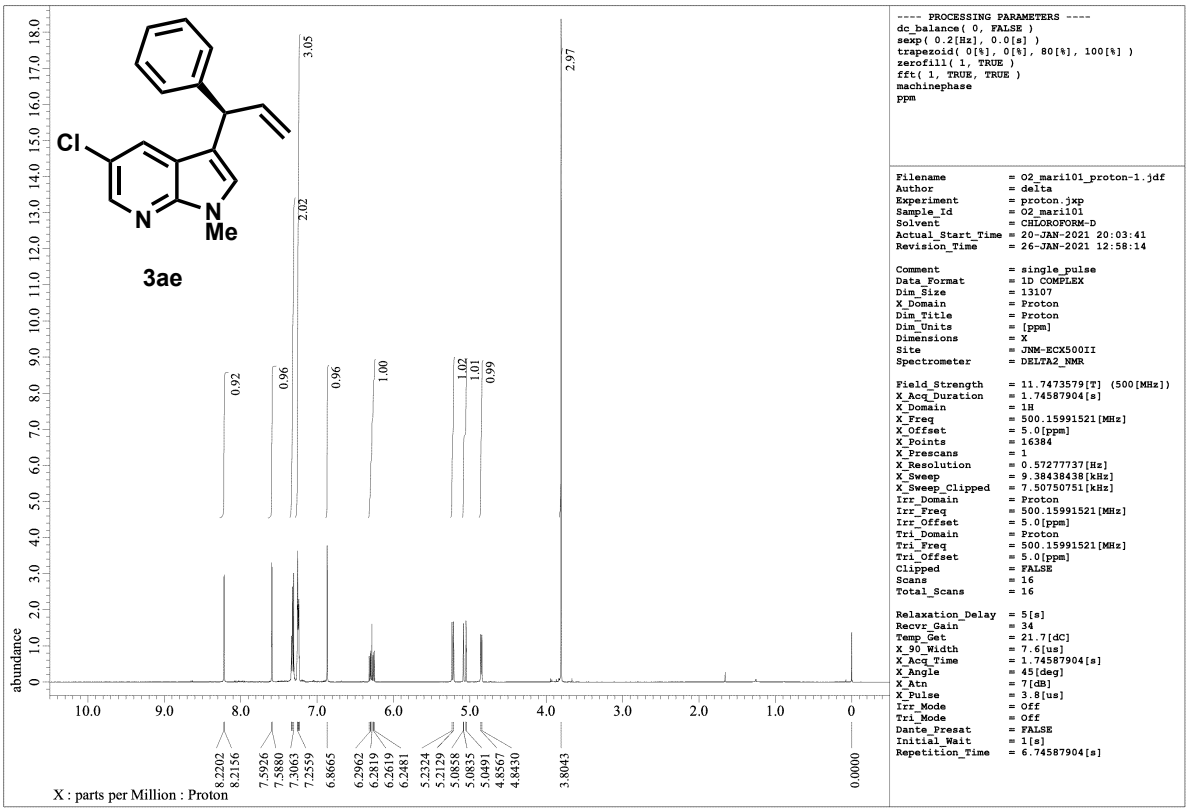
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.4[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.9[dB]
Irr_Atn_Noe         = 28.9[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

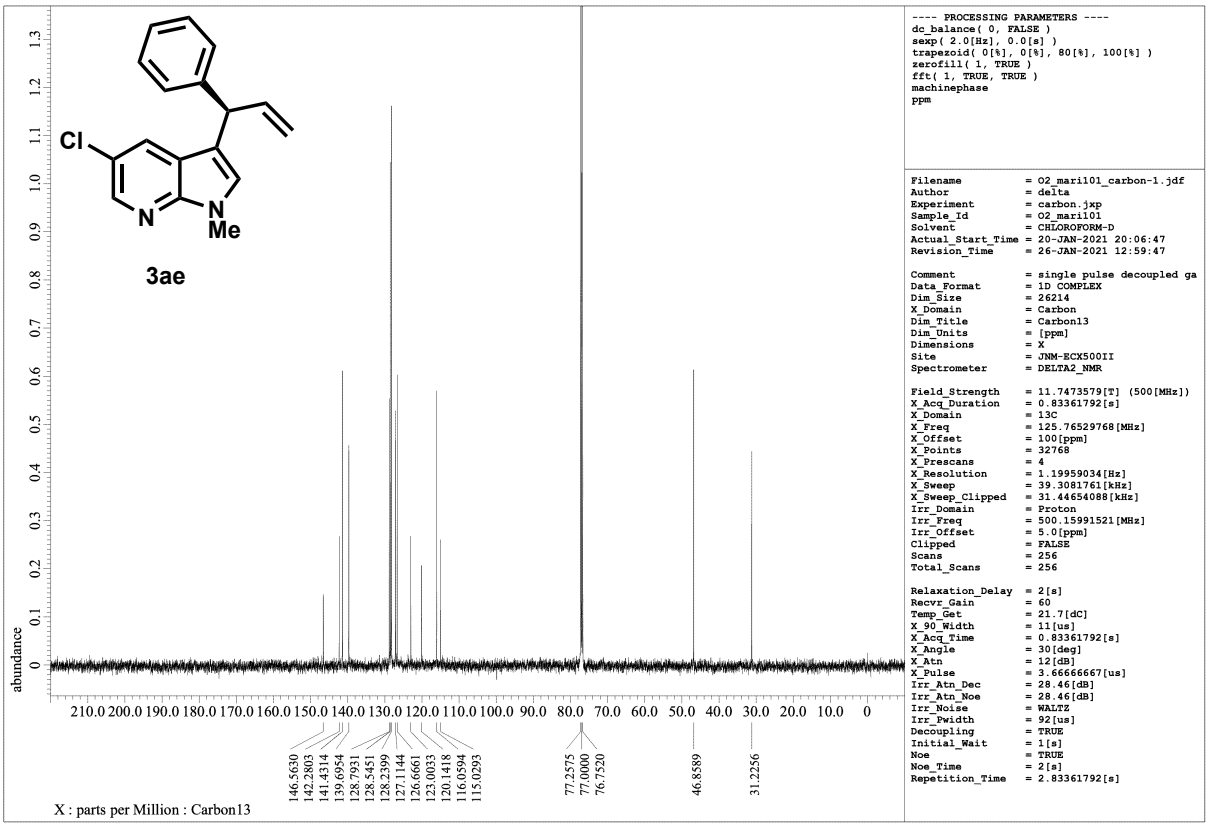


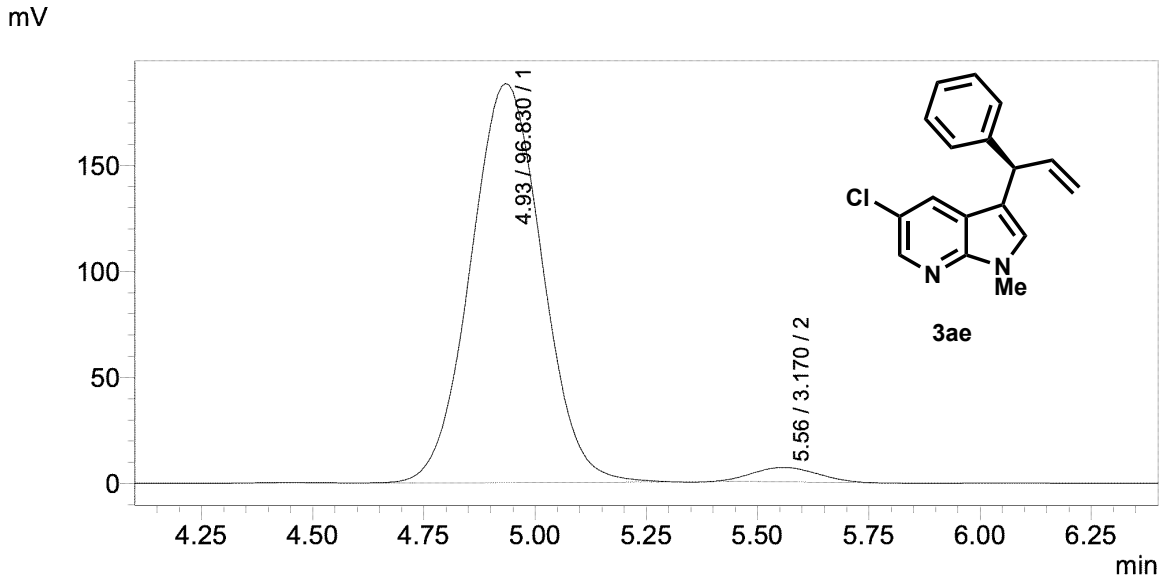
Peak#	Ret. Time	Area%
1	15.65	8.449
2	16.49	91.551
Total		100.000



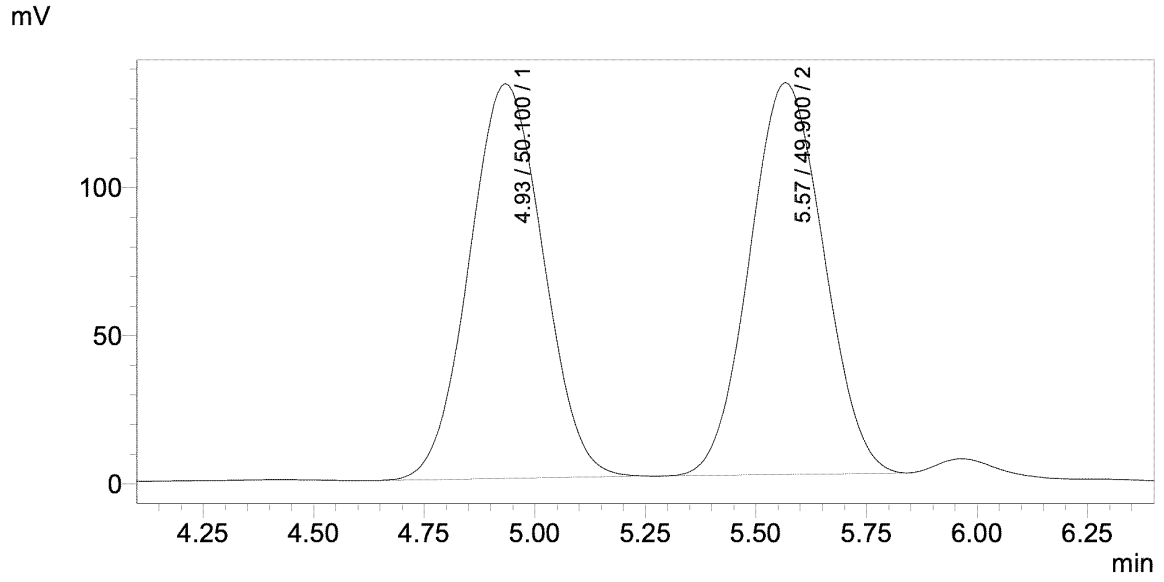
Peak#	Ret. Time	Area%
1	16.09	49.895
2	16.85	50.105
Total		100.000


3ae
Cl
N
N
Me
3.05
2.97
2.02
0.92
0.96
0.96
1.00
1.02
1.01
0.99
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.2202 8.2156 7.5926 7.5880 7.3063 7.2559 6.8665 6.2962 6.2819 6.2619 6.2481 5.2324 5.2129 5.0858 5.0835 5.0491 4.8567 4.8430 3.8043 0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari101_proton-1.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari101
Solvent = CHLOROFORM-D
Actual_Start_Time = 20-JAN-2021 20:03:41
Revision_Time = 26-JAN-2021 12:58:14
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 21.7[dC]
X_90_Width = 7.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 7[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

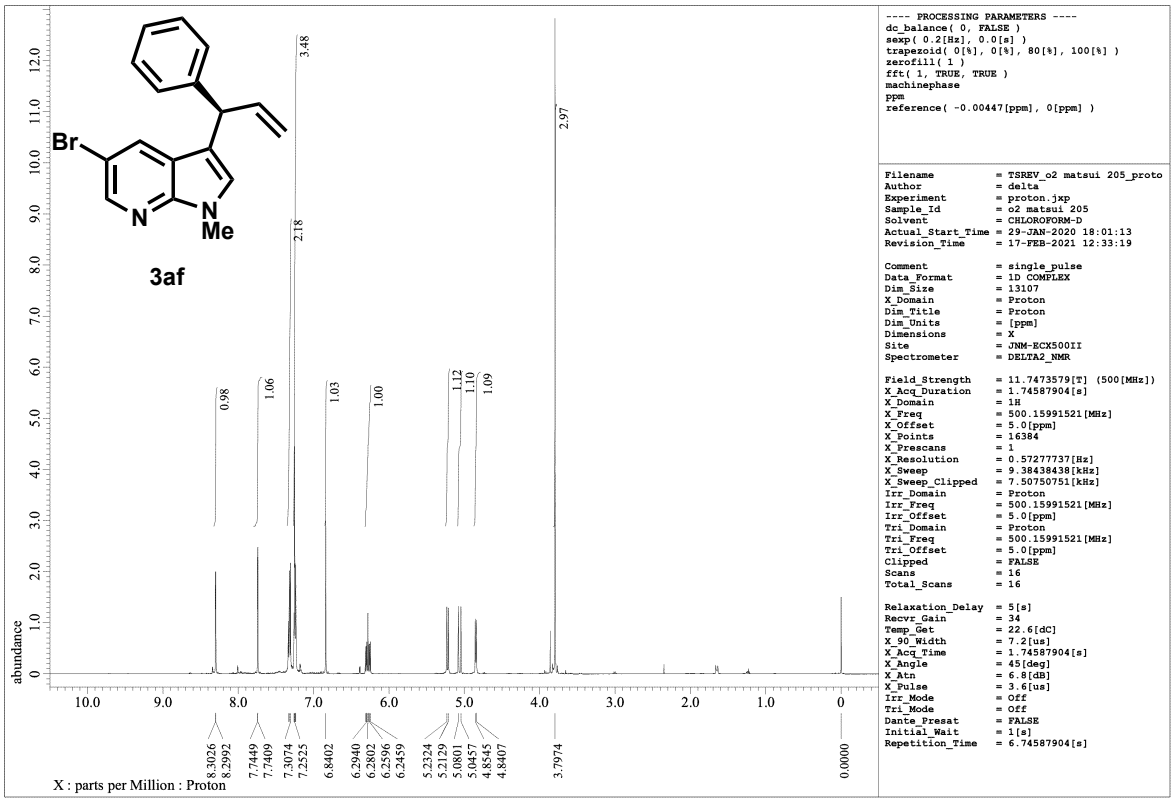

3ae
Cl
N
N
Me
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
146.5630 142.2803 141.4314 139.6954 128.7931 128.5451 128.2399 127.1144 126.6661 123.0033 120.1418 116.0594 115.0293 77.2575 77.0000 76.7520 46.8589 31.2256
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari101_carbon-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari101
Solvent = CHLOROFORM-D
Actual_Start_Time = 20-JAN-2021 20:06:47
Revision_Time = 26-JAN-2021 12:59:47
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21.7[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	4.93	96.830
2	5.56	3.170
Total		100.000



Peak#	Ret. Time	Area%
1	4.93	50.100
2	5.57	49.900
Total		100.000



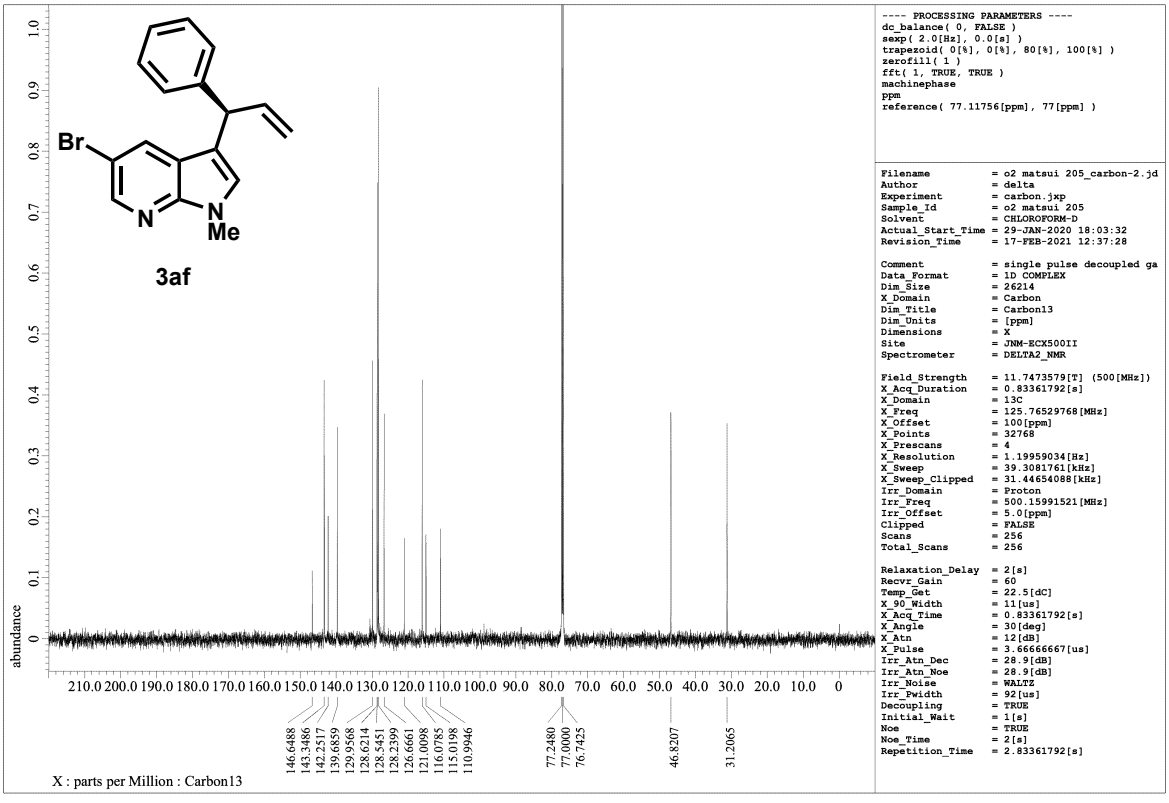

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.00447[ppm], 0[ppm] )
```

```
Filename            = TSREV_o2 matsui 205_proto
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 matsui 205
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 29-JAN-2020 18:01:13
Revision_Time       = 17-FEB-2021 12:33:19

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 22.6[dC]
X_90_Width          = 7.2[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.6[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



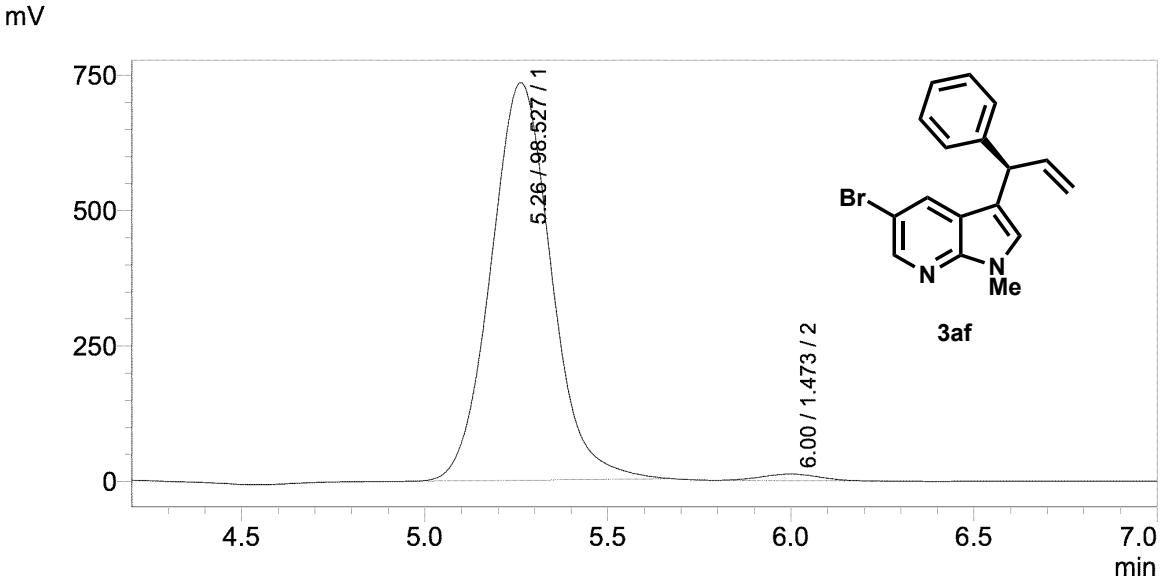

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.11756[ppm], 77[ppm] )
```

```
Filename            = o2 matsui 205_carbon-2.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 matsui 205
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 29-JAN-2020 18:03:32
Revision_Time       = 17-FEB-2021 12:37:28

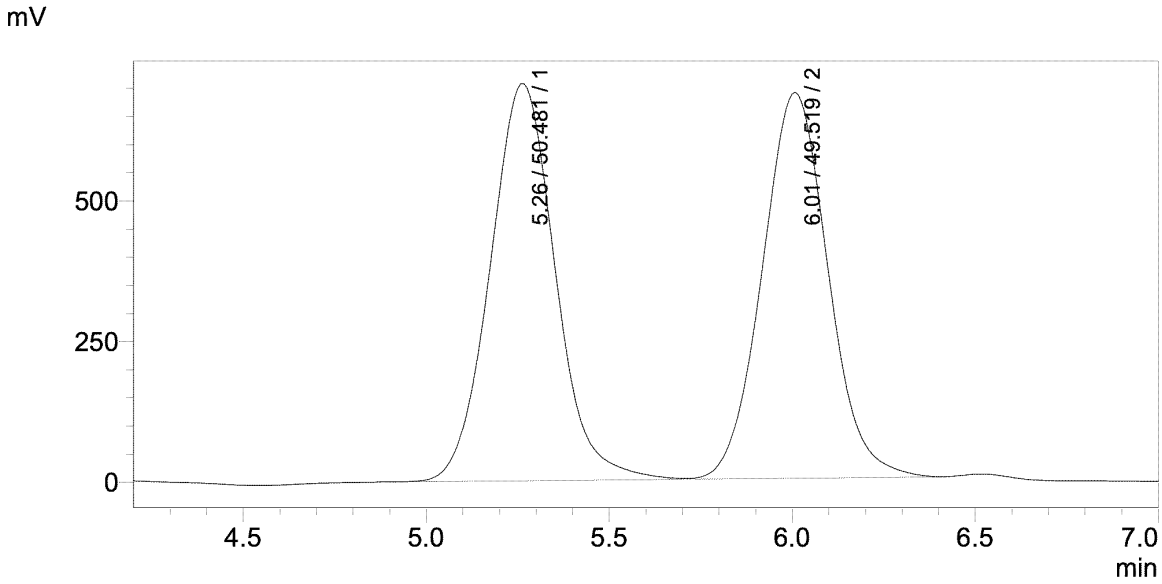
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

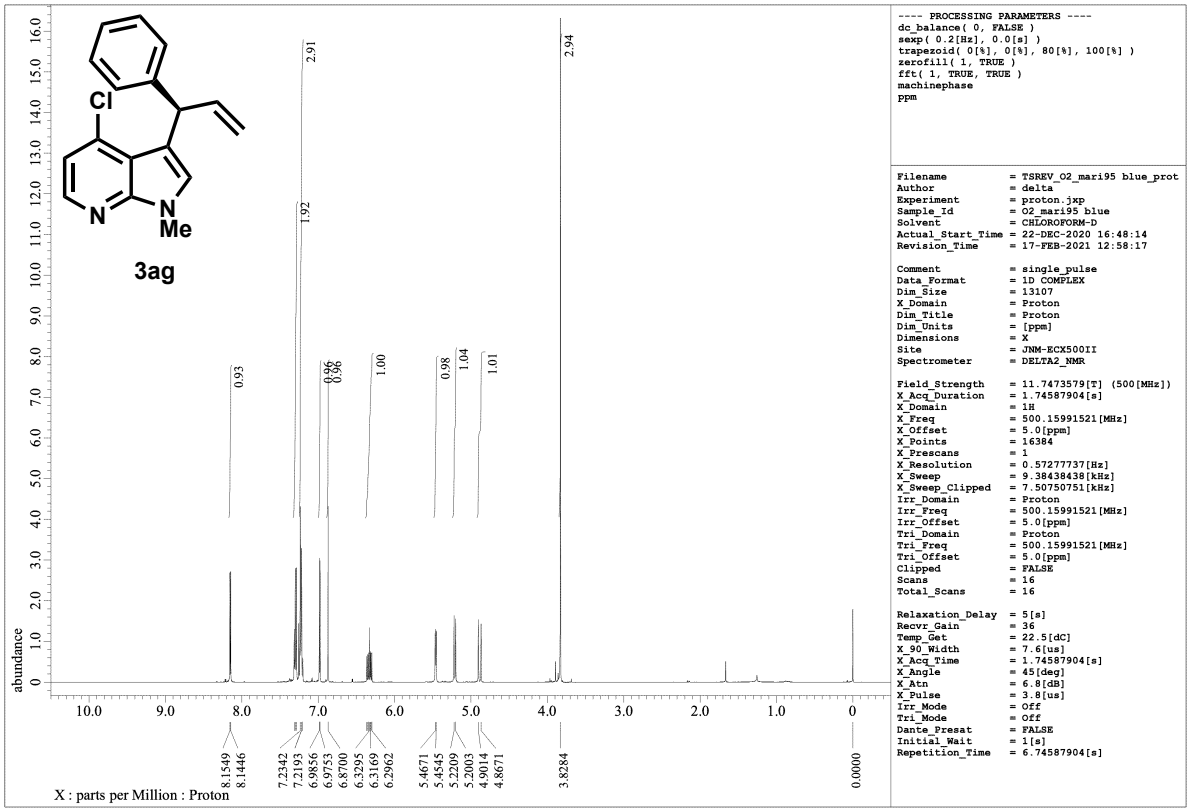
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.5[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.9[dB]
Irr_Atn_Noe         = 28.9[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

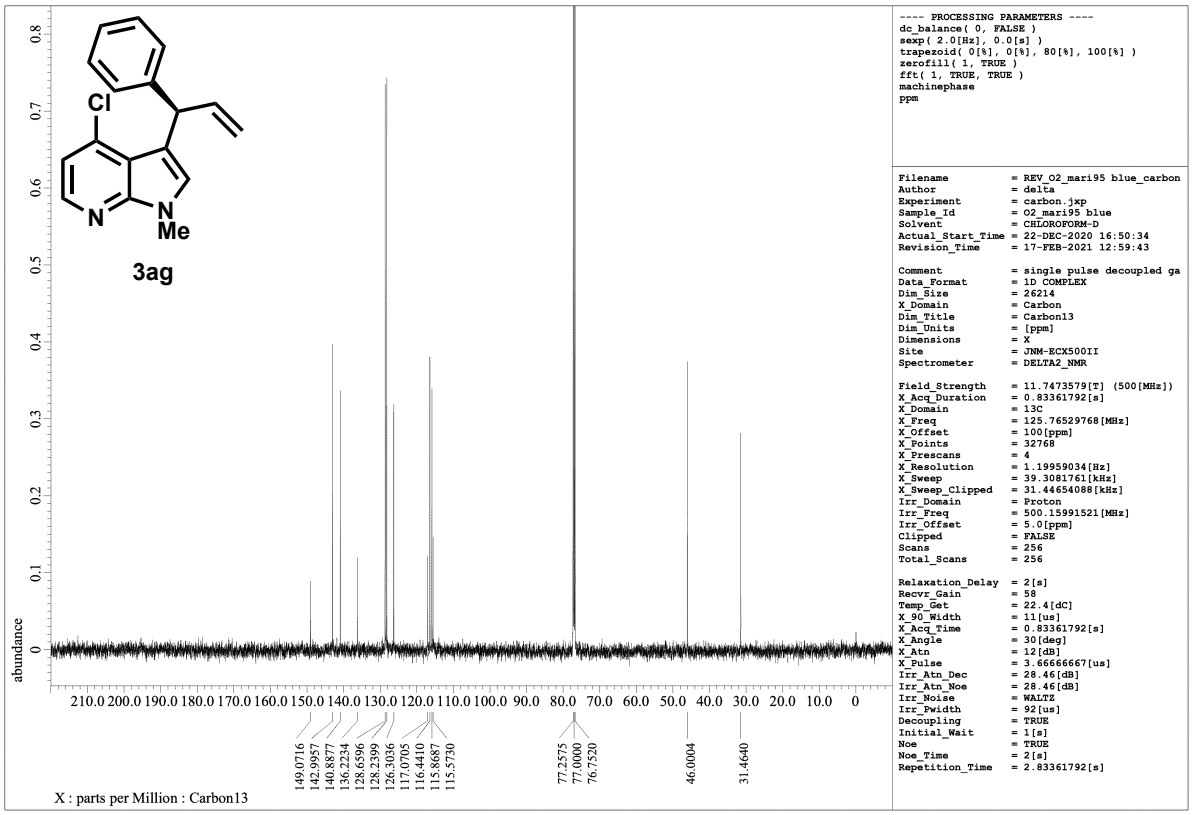


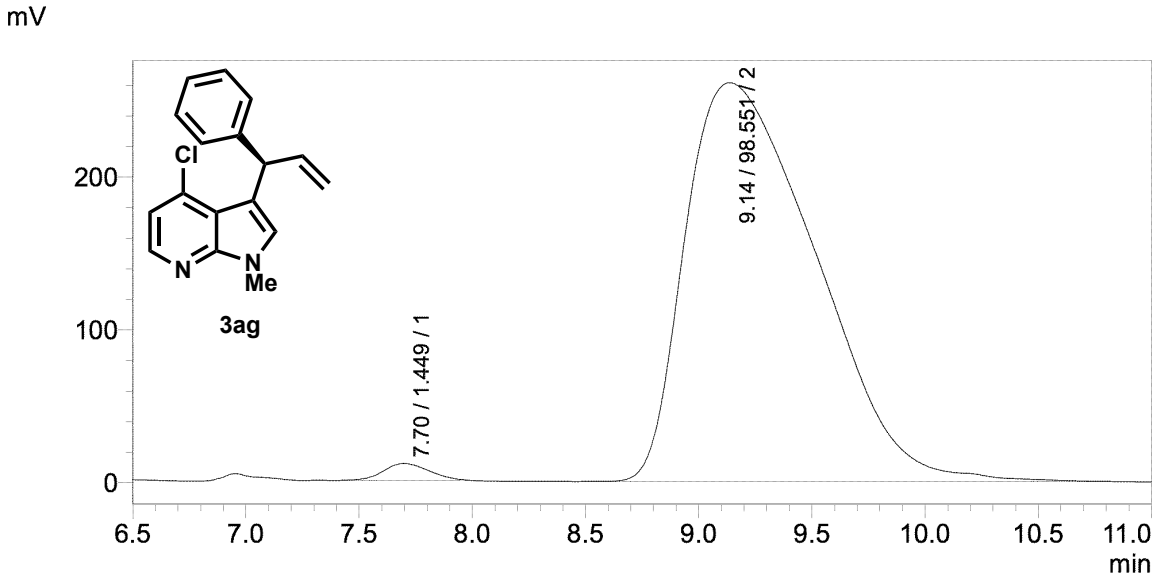
Peak#	Ret. Time	Area%
1	5.26	98.527
2	6.00	1.473
Total		100.000



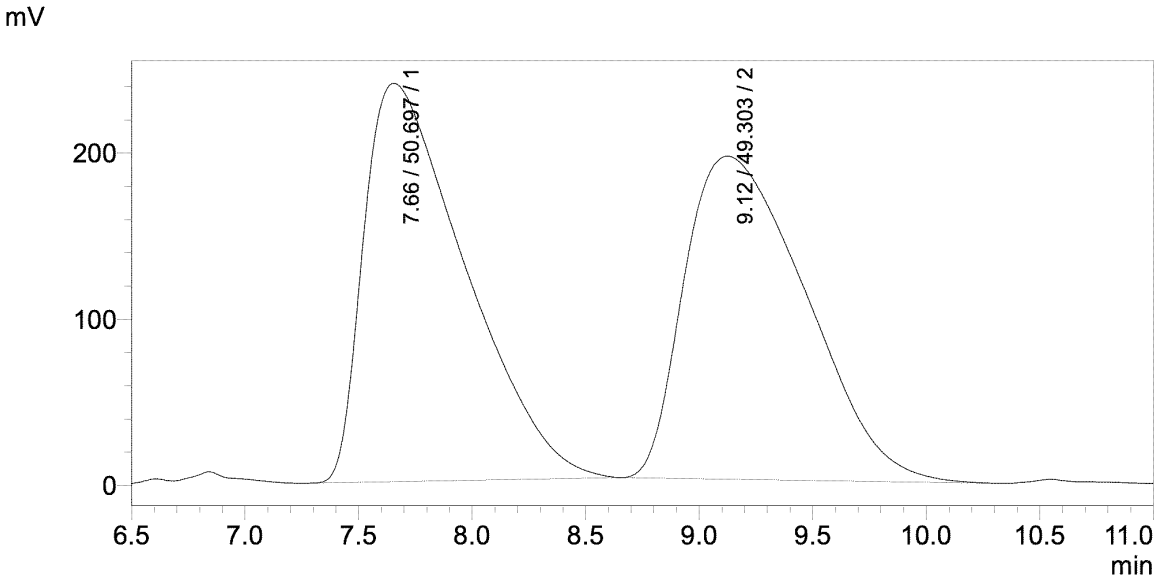
Peak#	Ret. Time	Area%
1	5.26	50.481
2	6.01	49.519
Total		100.000


Cl
N
N
Me
3ag
2.91
1.92
2.94
0.93
0.96
0.96
1.00
0.98
1.04
1.01
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.1549 8.1446 7.2342 7.2193 6.9856 6.9753 6.8700 6.3295 6.3169 6.2962 5.4671 5.4545 5.2209 5.2003 4.9014 4.8671 3.8284 0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = TSREV_O2_mari95 blue_prot
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari95 blue
Solvent = CHLOROFORM-D
Actual_Start_Time = 22-DEC-2020 16:48:14
Revision_Time = 17-FEB-2021 12:58:17
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 22.5[dC]
X_90_Width = 7.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

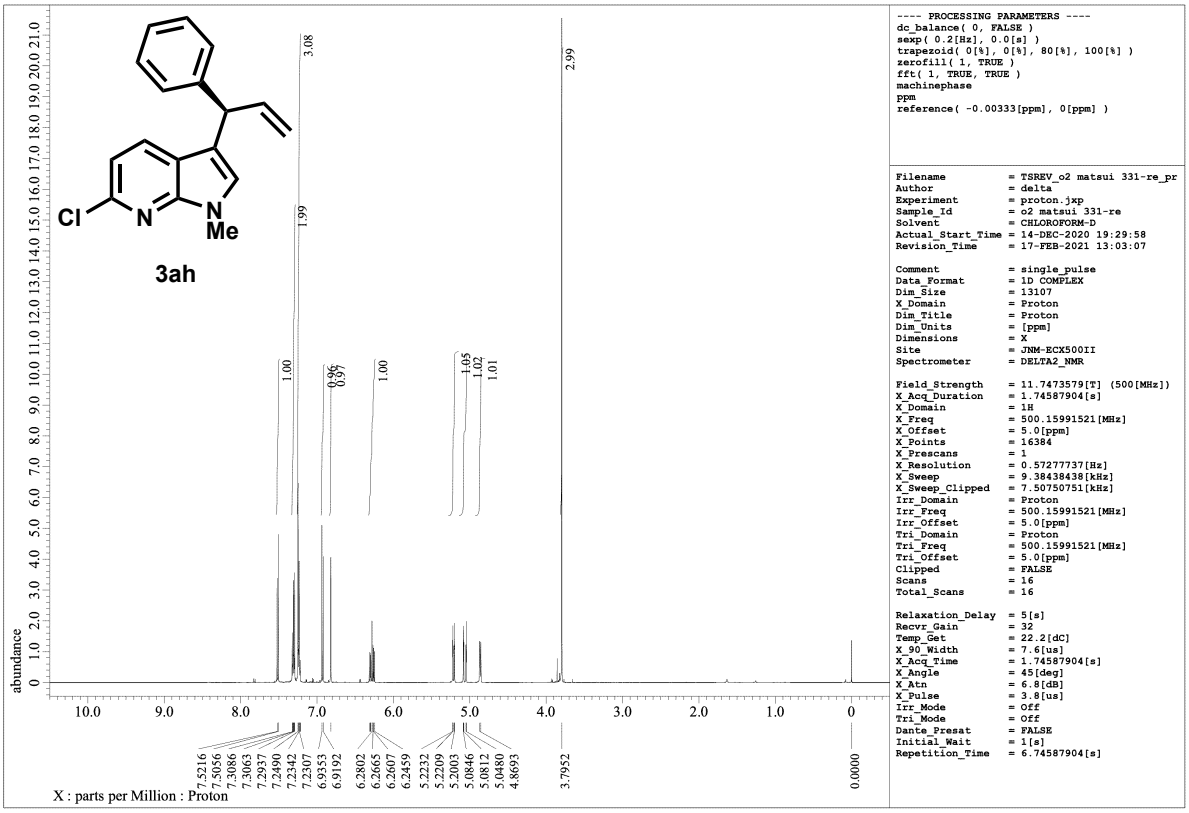

Cl
N
N
Me
3ag
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
149.0716 142.9957 140.8877 136.2234 128.6596 128.2399 126.3036 117.0705 116.4410 115.8687 115.5730 77.2575 77.0000 76.7520 46.0004 31.4640
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = REV_O2_mari95 blue_carbon
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari95 blue
Solvent = CHLOROFORM-D
Actual_Start_Time = 22-DEC-2020 16:50:34
Revision_Time = 17-FEB-2021 12:59:43
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 22.4[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]

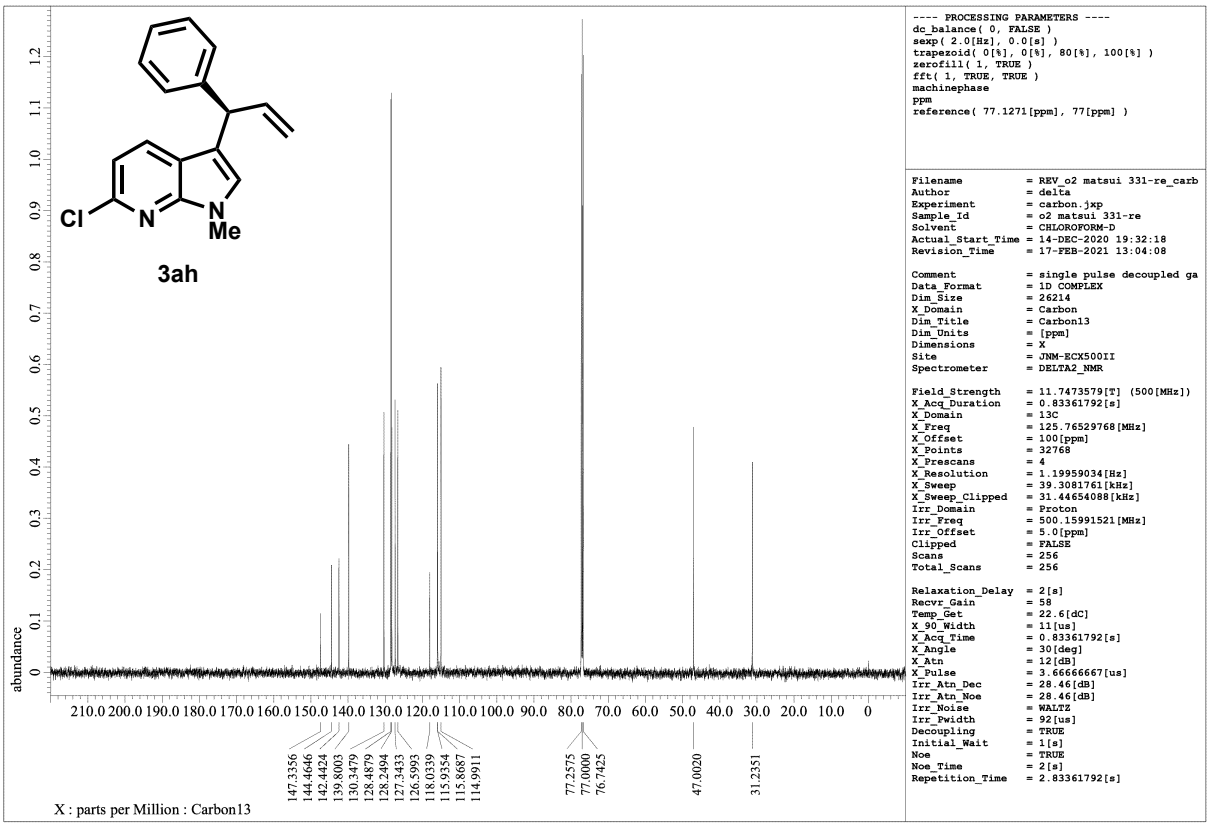


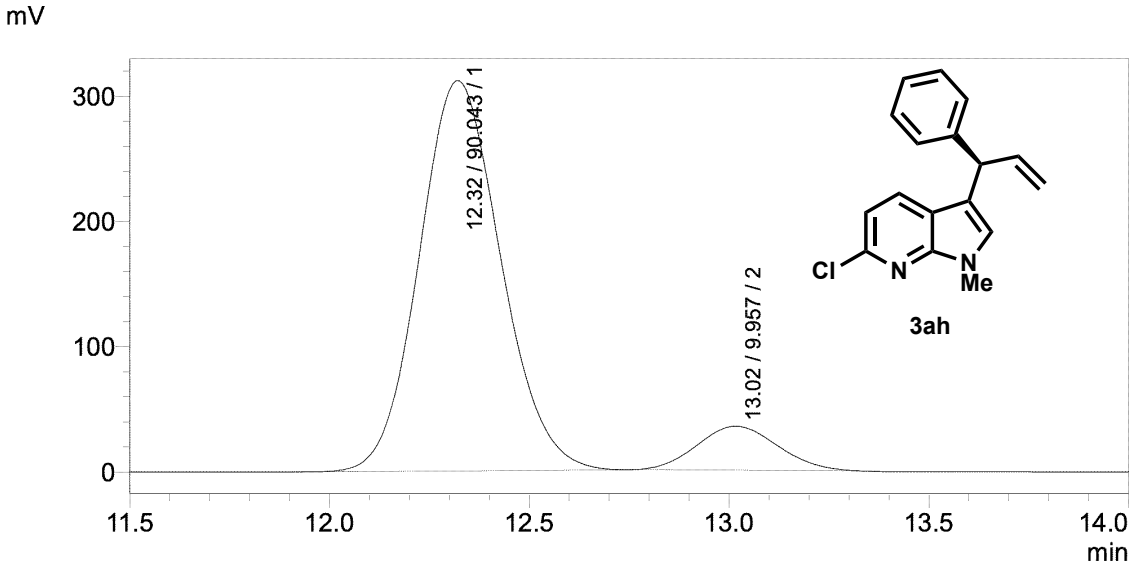
Peak#	Ret. Time	Area%
1	7.70	1.449
2	9.14	98.551
Total		100.000



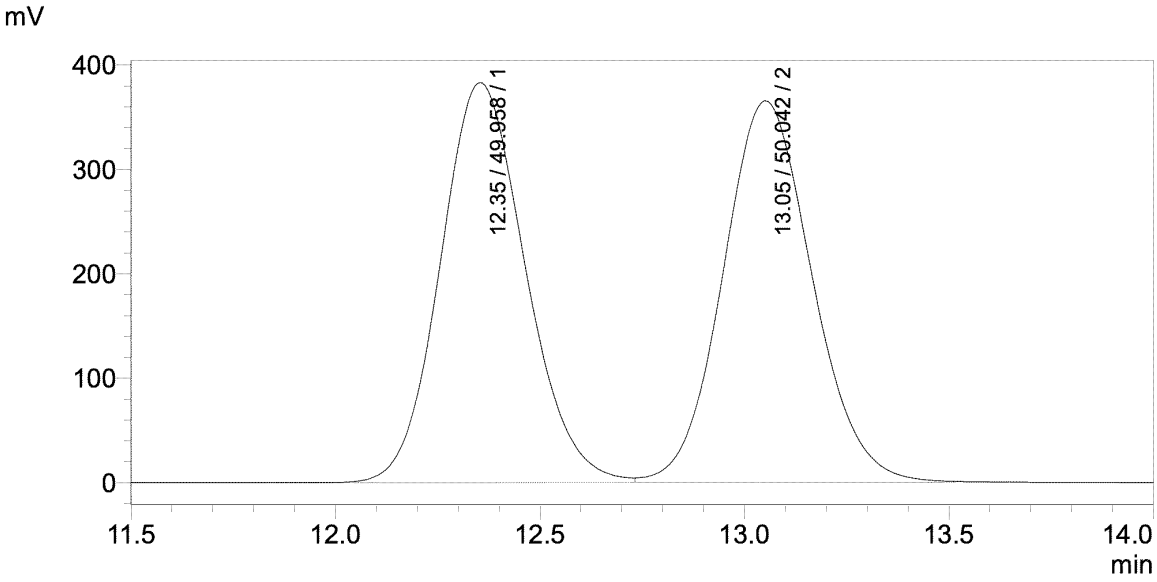
Peak#	Ret. Time	Area%
1	7.66	50.697
2	9.12	49.303
Total		100.000


3ah
Cl
N
N
Me
3.08
2.99
1.99
1.00
0.96
0.99
1.00
1.05
1.02
1.01
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.5216 7.5056 7.3086 7.3063 7.2937 7.2490 7.2342 7.2307 6.9353 6.9192 6.2802 6.2665 6.2607 6.2459 5.2232 5.2209 5.2003 5.0846 5.0812 5.0480 4.8693 3.7952 0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(-0.00333[ppm], 0[ppm])
Filename = TSREV_o2 matsui 331-re_pr
Author = delta
Experiment = proton.jxp
Sample_Id = o2 matsui 331-re
Solvent = CHLOROFORM-D
Actual_Start_Time = 14-DEC-2020 19:29:58
Revision_Time = 17-FEB-2021 13:03:07
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 32
Temp_Get = 22.2[dC]
X_90_Width = 7.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

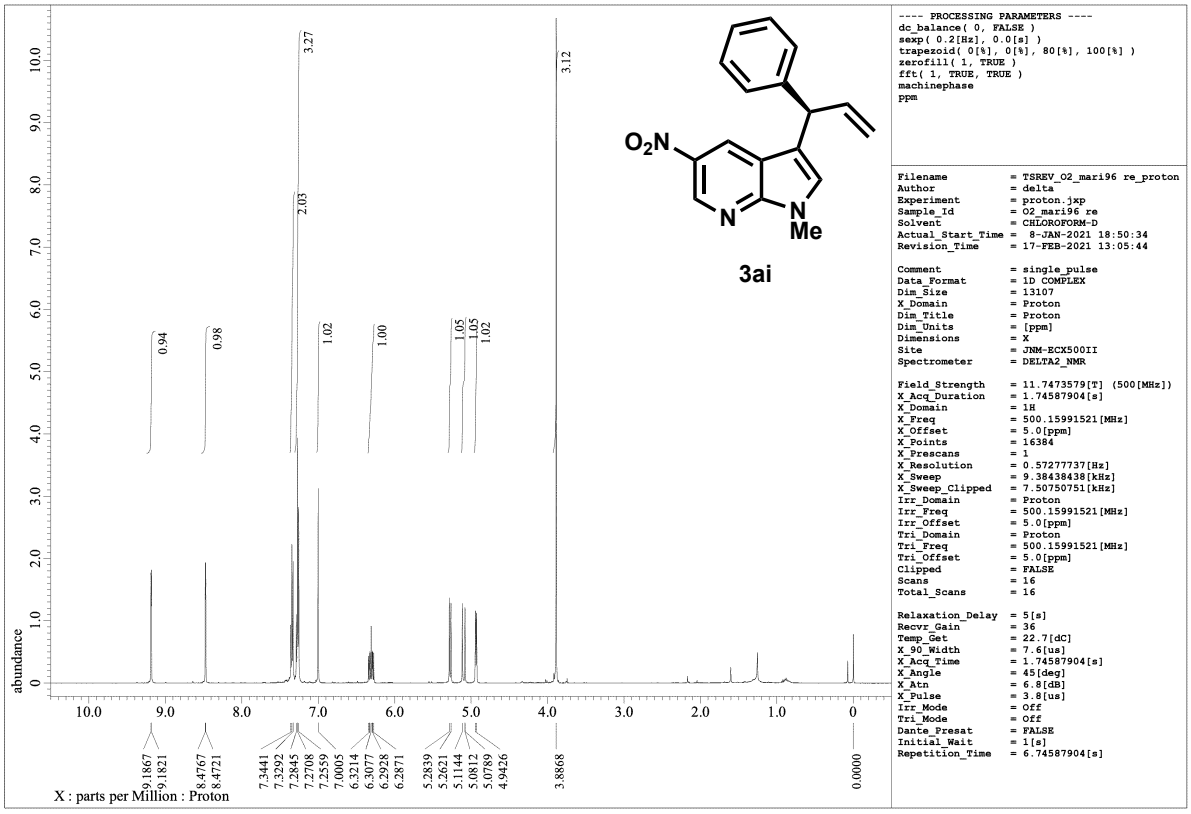

3ah
Cl
N
N
Me
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
147.3356 144.4646 142.4424 139.8003 130.3479 128.4879 128.2494 127.3433 126.5993 118.0339 115.9354 115.8687 114.9911 77.2575 77.0000 76.7425 47.0020 31.2351
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(77.1271[ppm], 77[ppm])
Filename = REV_o2 matsui 331-re_carb
Author = delta
Experiment = carbon.jxp
Sample_Id = o2 matsui 331-re
Solvent = CHLOROFORM-D
Actual_Start_Time = 14-DEC-2020 19:32:18
Revision_Time = 17-FEB-2021 13:04:08
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 22.6[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	12.32	90.043
2	13.02	9.957
Total		100.000



Peak#	Ret. Time	Area%
1	12.35	49.958
2	13.05	50.042
Total		100.000



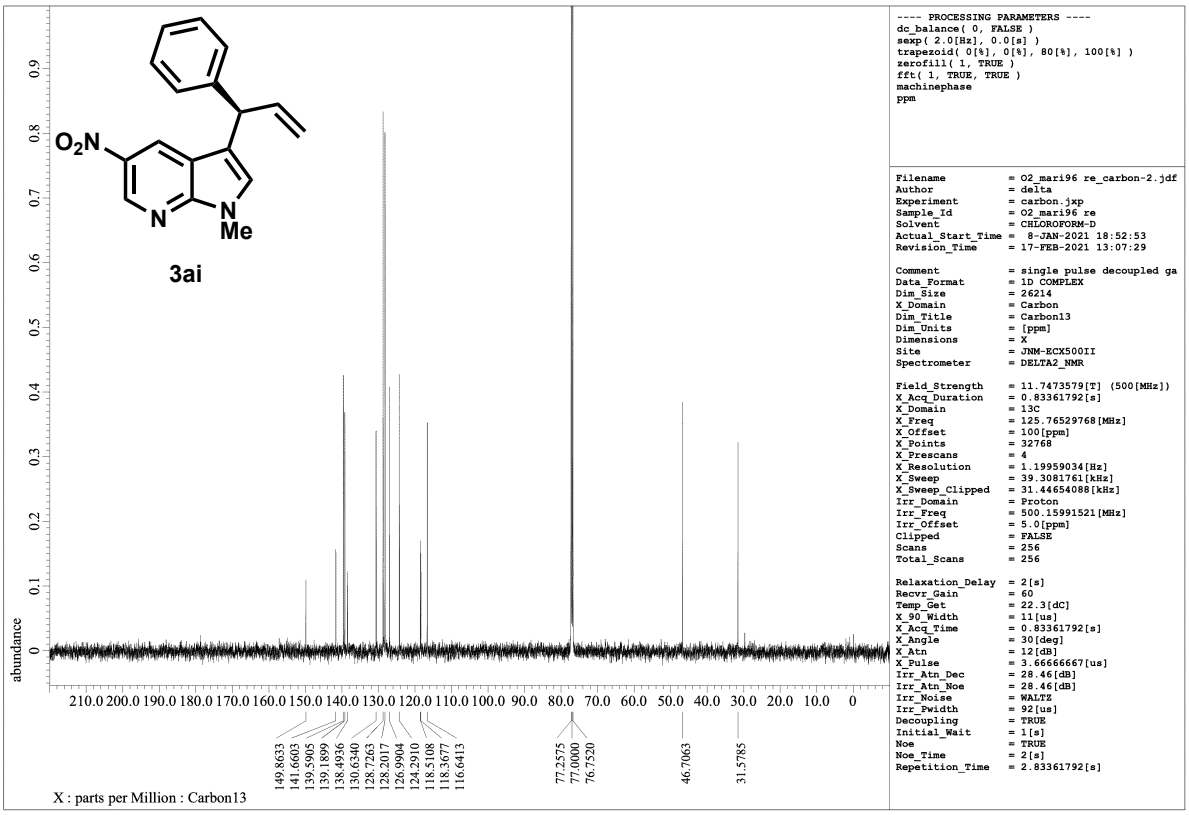

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = TSREV_O2_mari96 re_proton
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari96 re
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  8-JAN-2021 18:50:34
Revision_Time       = 17-FEB-2021 13:05:44

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 36
Temp_Get            = 22.7[dC]
X_90_Width          = 7.6[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.8[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



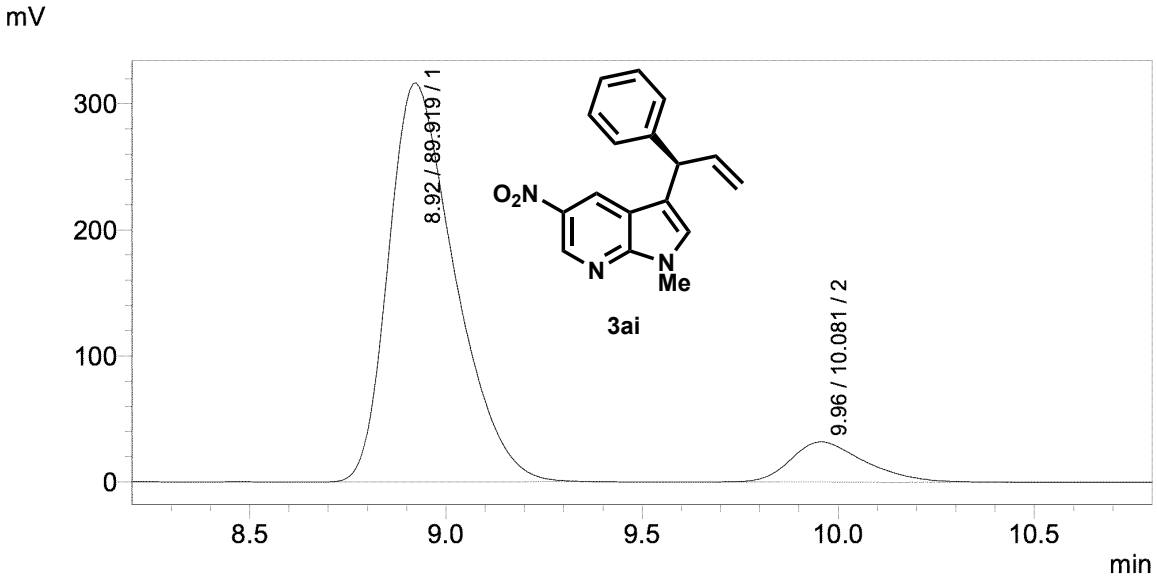

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = O2_mari96 re_carbon-2.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari96 re
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  8-JAN-2021 18:52:53
Revision_Time       = 17-FEB-2021 13:07:29

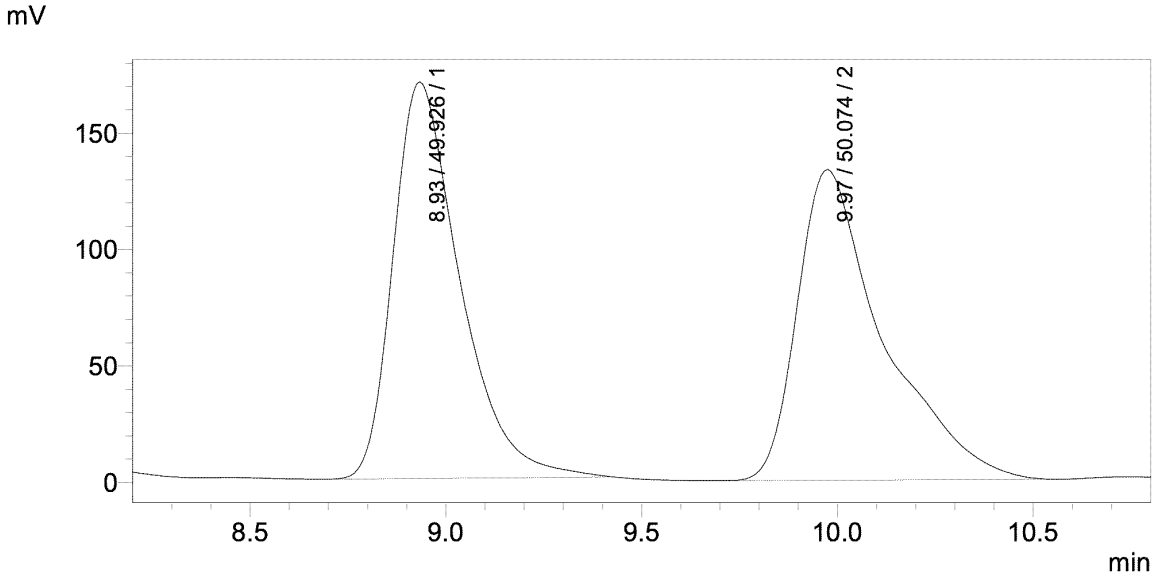
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

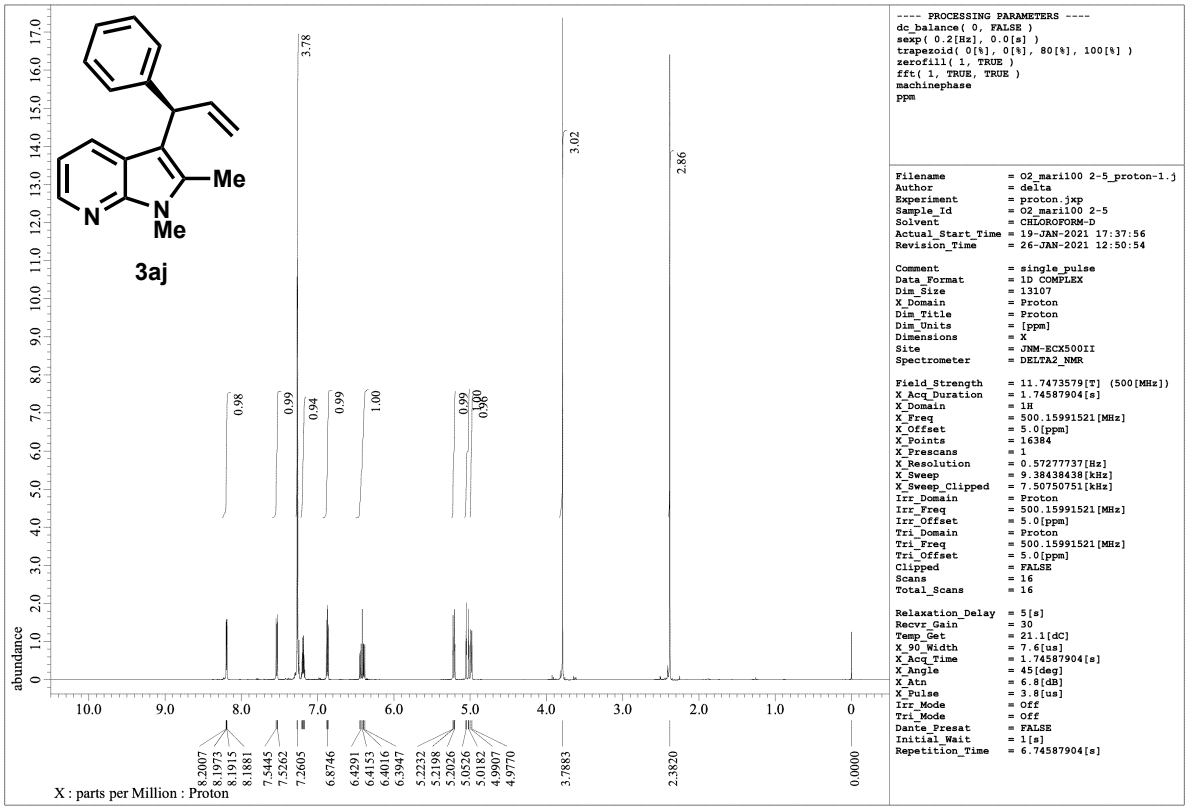
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.3[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.46[dB]
Irr_Atn_Noe         = 28.46[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

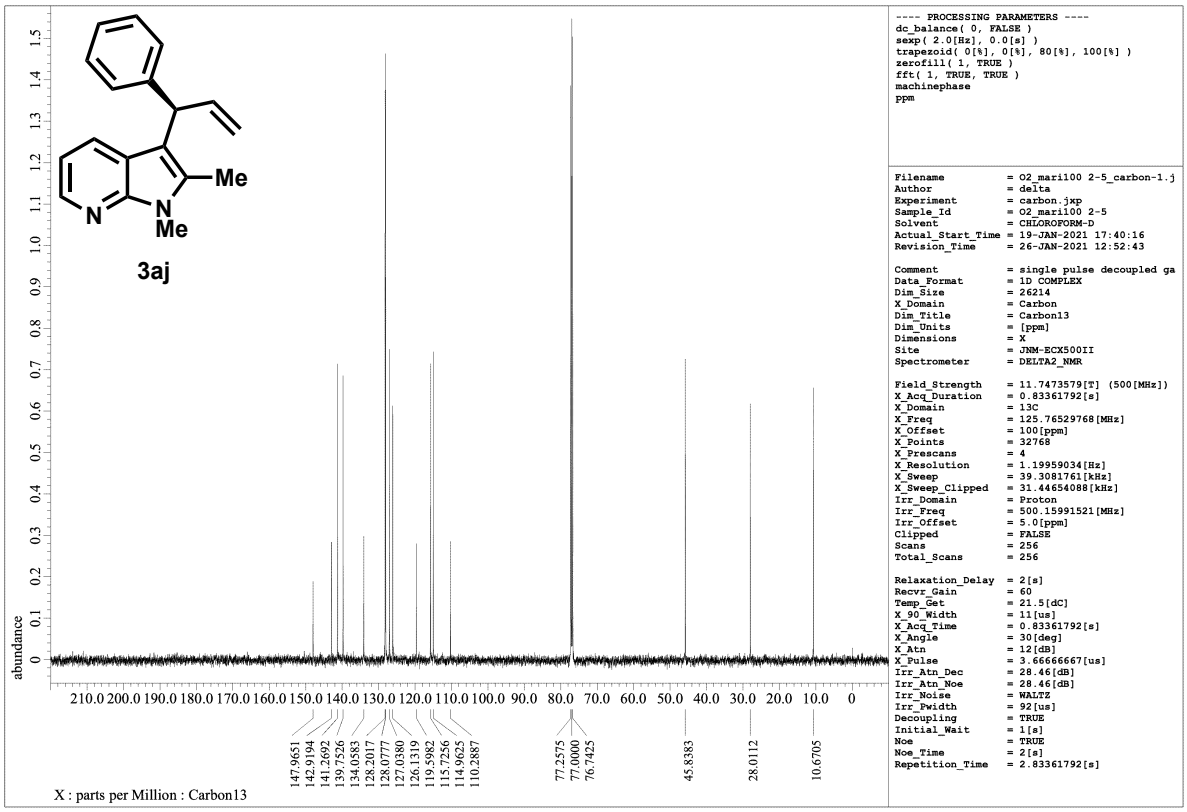


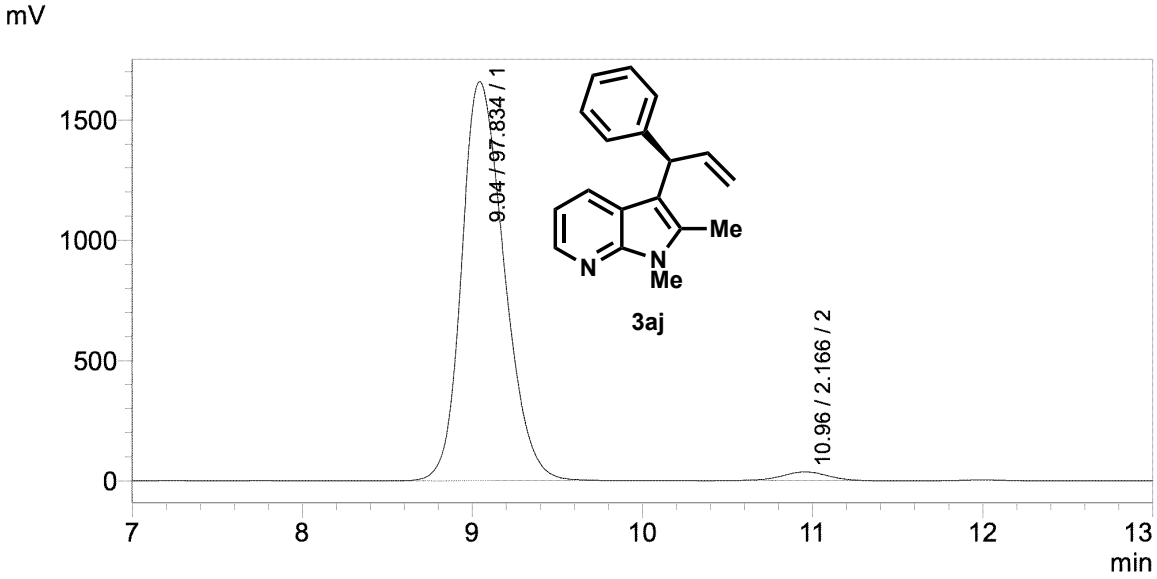
Peak#	Ret. Time	Area%
1	8.92	89.919
2	9.96	10.081
Total		100.000



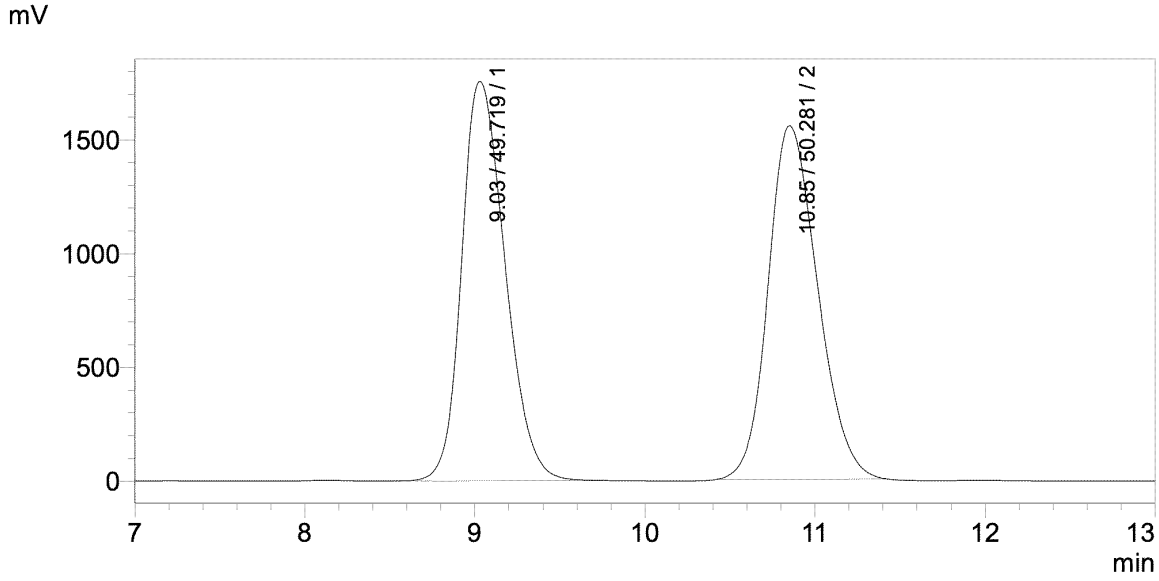
Peak#	Ret. Time	Area%
1	8.93	49.926
2	9.97	50.074
Total		100.000


3aj
Me
N
Me
3.78
3.02
2.86
0.98
0.99
0.94
0.99
1.00
0.99
1.00
0.98
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.2007 8.1973 8.1915 8.1881 7.5445 7.5262 7.2605 6.8746 6.4291 6.4153 6.4016 6.3947 5.2232 5.2198 5.2026 5.0536 5.0182 4.9907 4.9770 3.7883 2.3820 0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari100 2-5_proton-1.j
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari100 2-5
Solvent = CHLOROFORM-D
Actual_Start_Time = 19-JAN-2021 17:37:56
Revision_Time = 26-JAN-2021 12:50:54
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 30
Temp_Get = 21.1[dC]
X_90_Width = 7.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

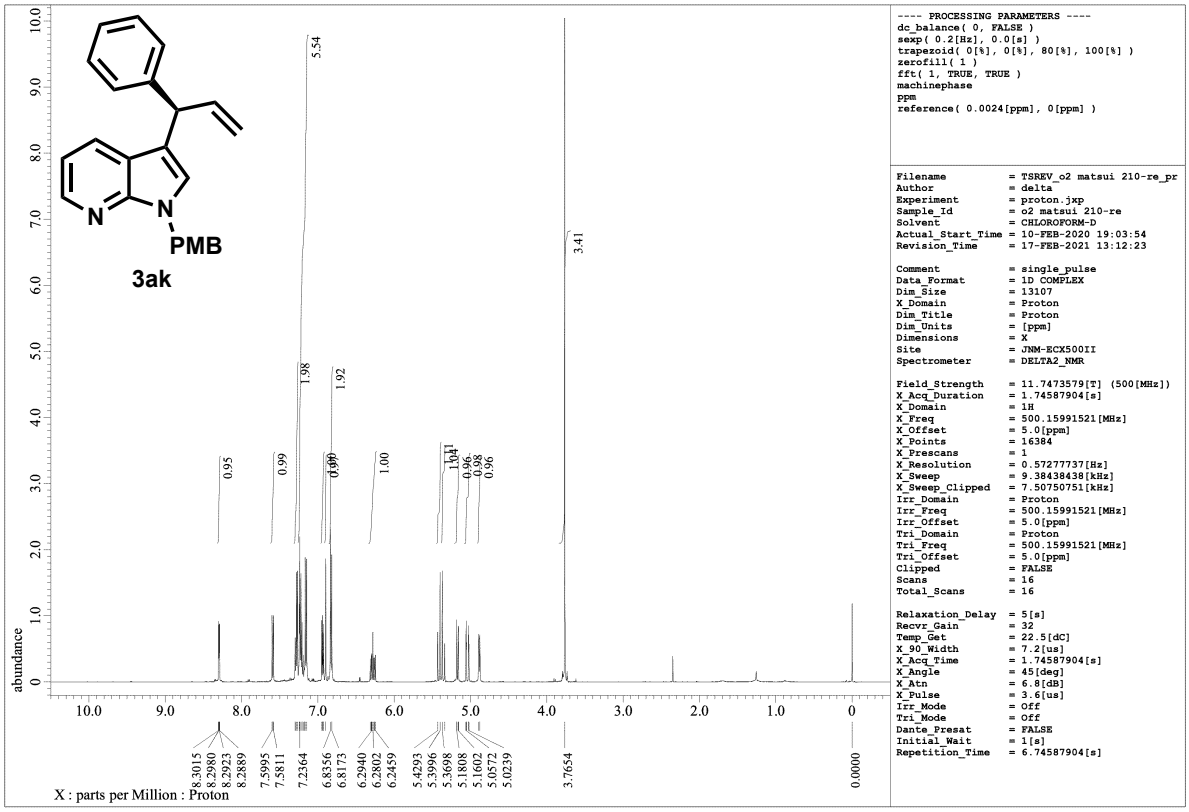

3aj
Me
N
Me
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
147.9651 142.9194 141.2692 139.7526 134.0583 128.2017 128.0777 127.0380 126.1319 119.5982 115.7256 114.9625 110.2887 77.2575 77.0000 76.7425 45.8383 28.0112 10.6705
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari100 2-5_carbon-1.j
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari100 2-5
Solvent = CHLOROFORM-D
Actual_Start_Time = 19-JAN-2021 17:40:16
Revision_Time = 26-JAN-2021 12:52:43
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 21.5[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	9.04	97.834
2	10.96	2.166
Total		100.000



Peak#	Ret. Time	Area%
1	9.03	49.719
2	10.85	50.281
Total		100.000



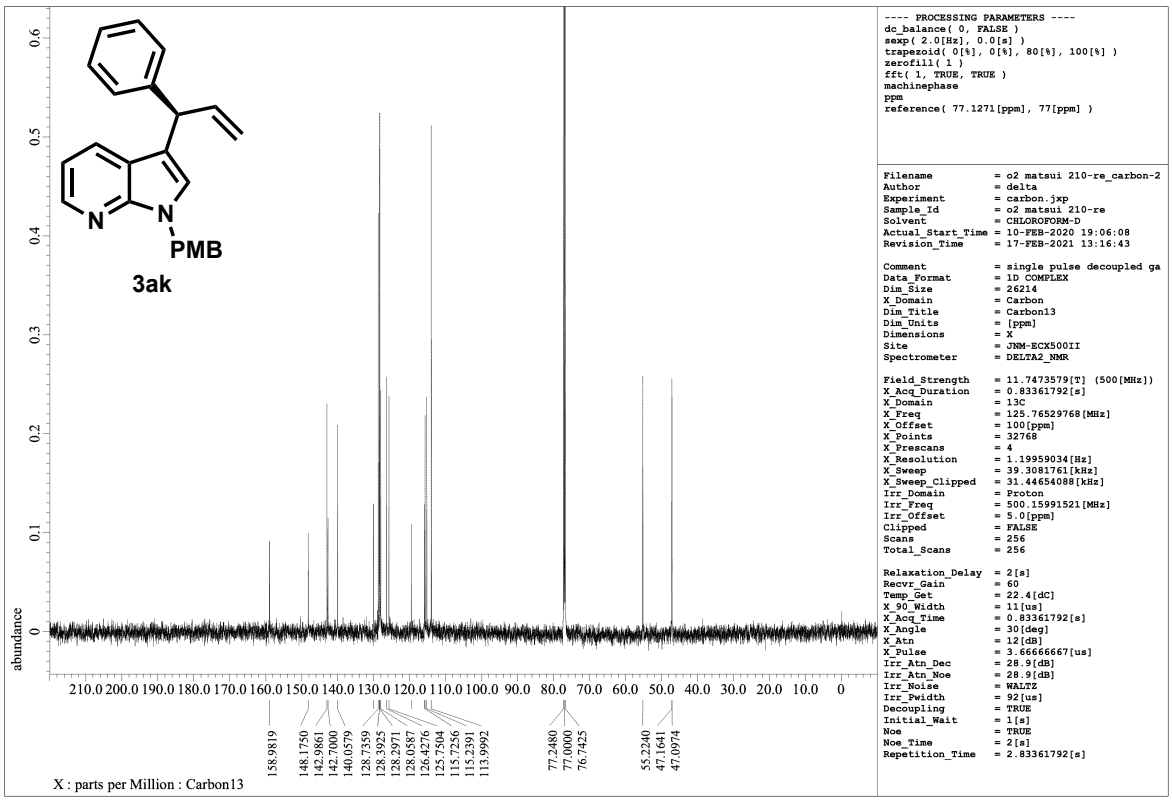

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 0.0024[ppm], 0[ppm] )
```

```
Filename           = TSREV_o2 matsui 210-re_pr
Author             = delta
Experiment         = proton.jxp
Sample_Id          = o2 matsui 210-re
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 10-FEB-2020 19:03:54
Revision_Time      = 17-FEB-2021 13:12:23

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
X_Domain           = Proton
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX500II
Spectrometer       = DELTA2_NMR

Field_Strength     = 11.7473579[T] (500[MHz])
X_Acq_Duration     = 1.74587904[s]
X_Domain           = 1H
X_Freq             = 500.15991521[MHz]
X_Offset           = 5.0[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.57277737[Hz]
X_Sweep            = 9.38438438[kHz]
X_Sweep_Clipped    = 7.50750751[kHz]
Irr_Domain         = Proton
Irr_Freq           = 500.15991521[MHz]
Irr_Offset         = 5.0[ppm]
Tri_Domain         = Proton
Tri_Freq           = 500.15991521[MHz]
Tri_Offset         = 5.0[ppm]
Clipped            = FALSE
Scans              = 16
Total_Scans        = 16

Relaxation_Delay   = 5[s]
Recvr_Gain         = 32
Temp_Get           = 22.5[dC]
X_90_Width         = 7.2[us]
X_Acq_Time         = 1.74587904[s]
X_Angle            = 45[deg]
X_Atn              = 6.8[dB]
X_Pulse            = 3.6[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 6.74587904[s]
```



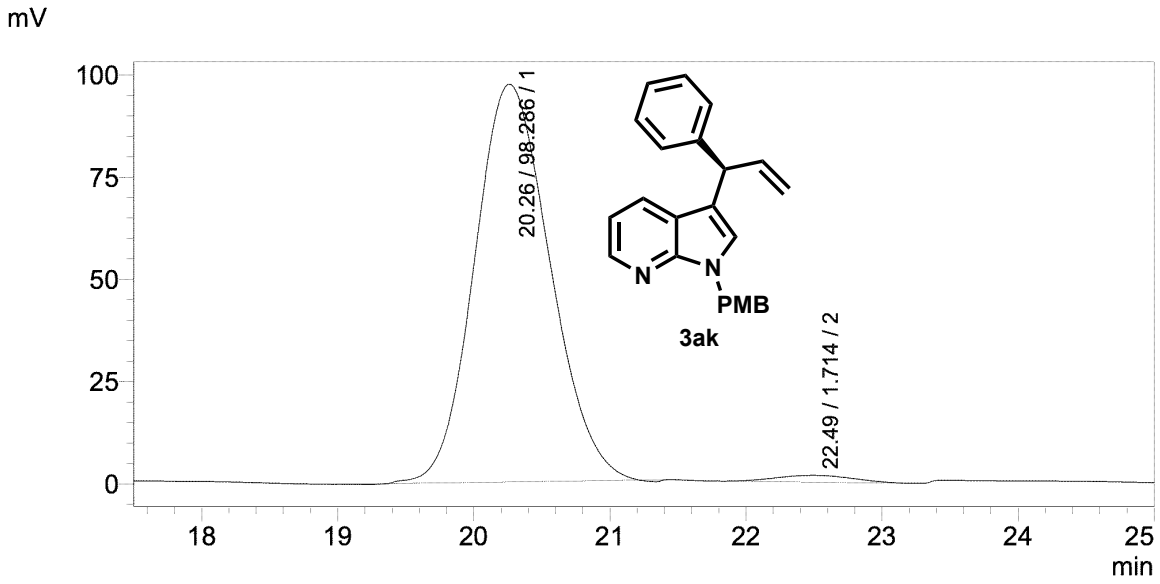

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.1271[ppm], 77[ppm] )
```

```
Filename           = o2 matsui 210-re_carbon-2
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = o2 matsui 210-re
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 10-FEB-2020 19:06:08
Revision_Time      = 17-FEB-2021 13:16:43

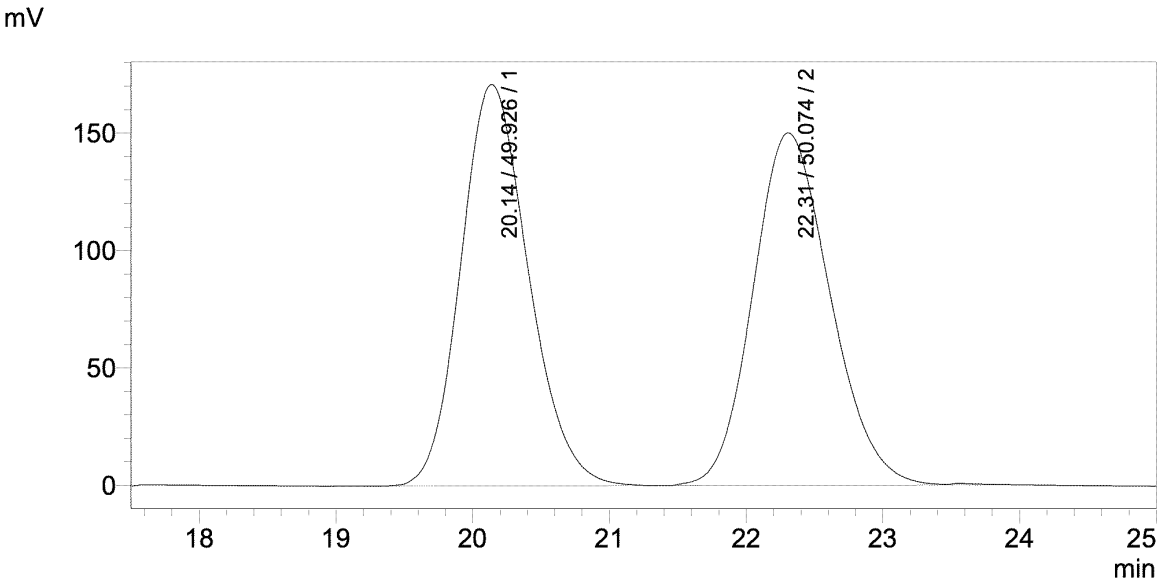
Comment            = single pulse decoupled ga
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = Carbon
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX500II
Spectrometer       = DELTA2_NMR

Field_Strength     = 11.7473579[T] (500[MHz])
X_Acq_Duration     = 0.83361792[s]
X_Domain           = 13C
X_Freq             = 125.76529768[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 1.19959034[Hz]
X_Sweep            = 39.3081761[kHz]
X_Sweep_Clipped    = 31.44654088[kHz]
Irr_Domain         = Proton
Irr_Freq           = 500.15991521[MHz]
Irr_Offset         = 5.0[ppm]
Clipped            = FALSE
Scans              = 256
Total_Scans        = 256

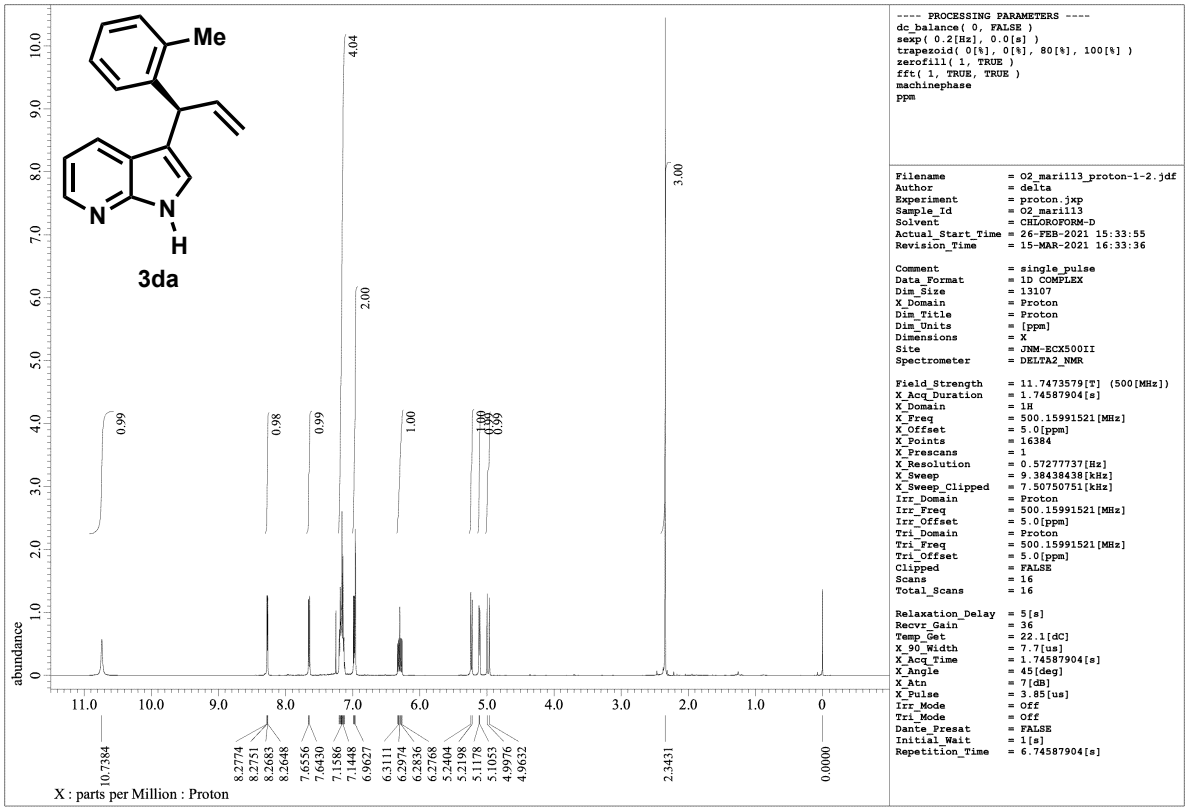
Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.4[dC]
X_90_Width         = 11[us]
X_Acq_Time         = 0.83361792[s]
X_Angle            = 30[deg]
X_Atn              = 12[dB]
X_Pulse            = 3.66666667[us]
Irr_Atn_Dec        = 28.9[dB]
Irr_Atn_Noe        = 28.9[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 92[us]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 2.83361792[s]
```

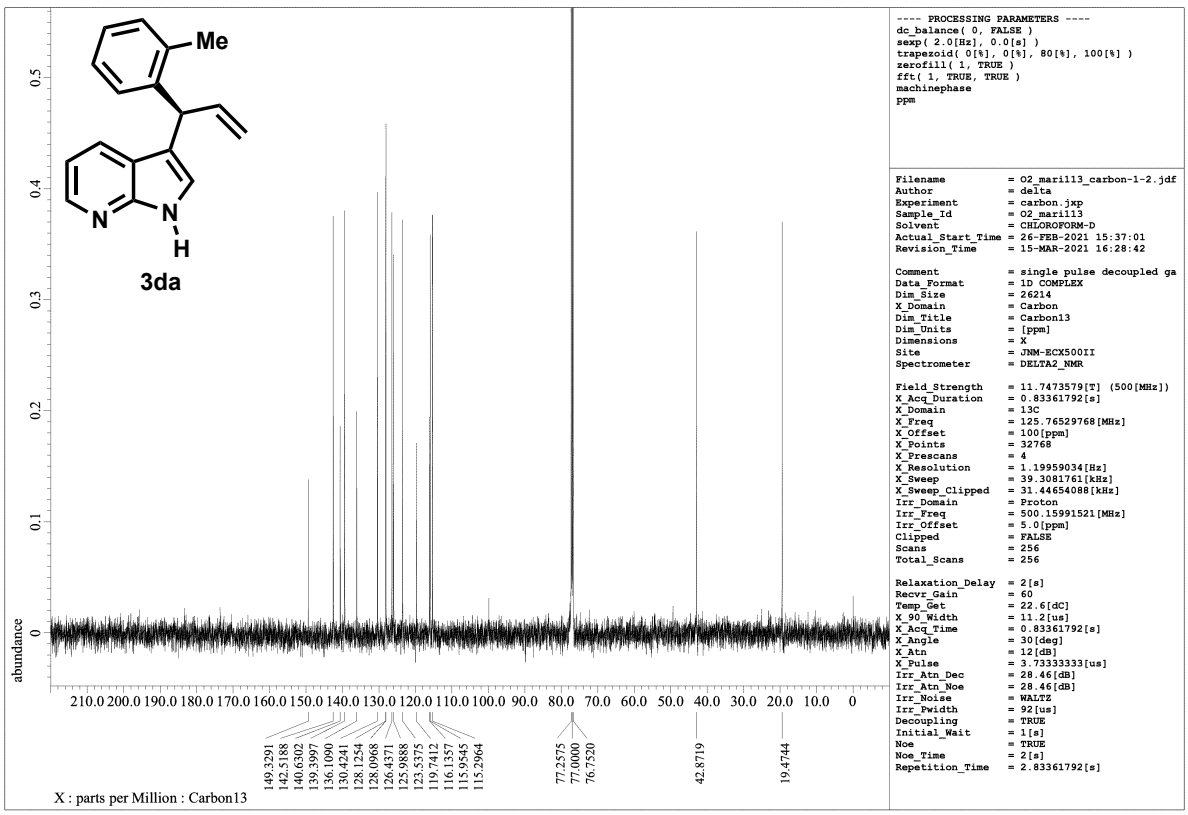


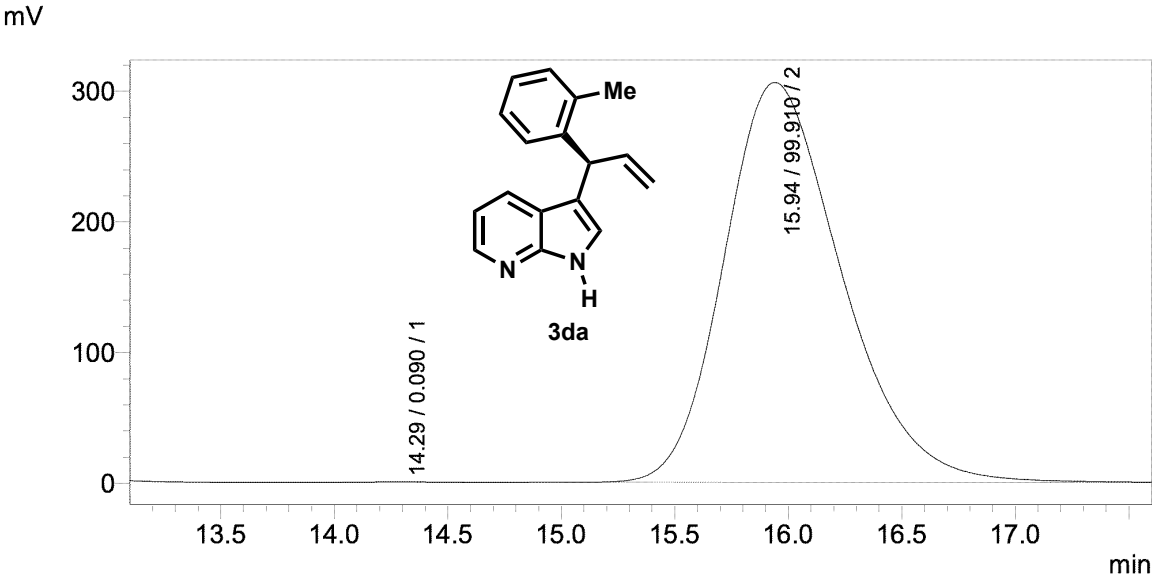
Peak#	Ret. Time	Area%
1	20.26	98.286
2	22.49	1.714
Total		100.000



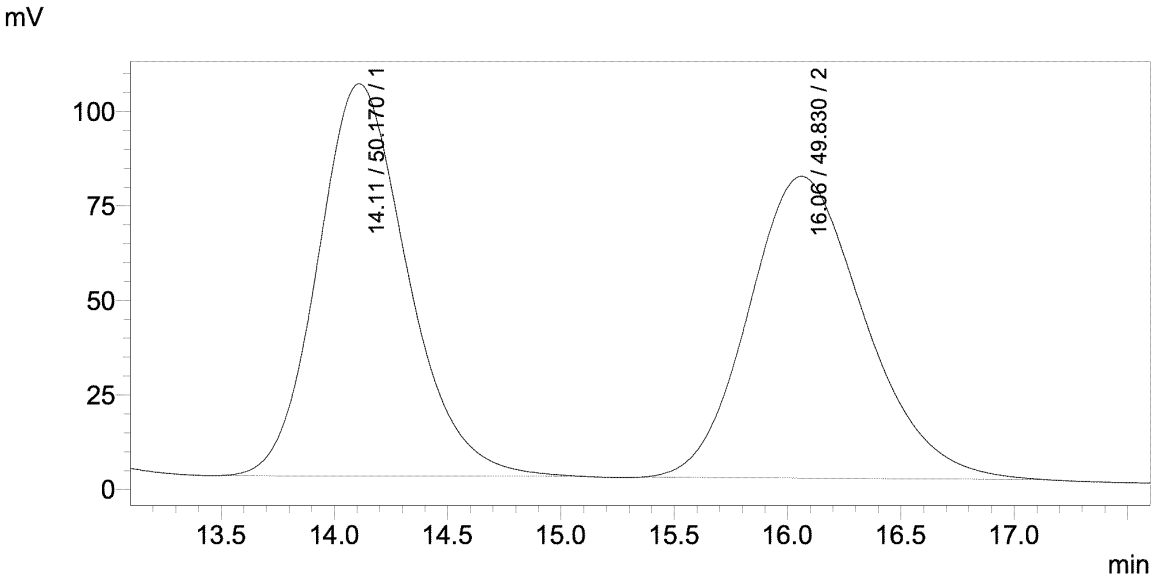
Peak#	Ret. Time	Area%
1	20.14	49.926
2	22.31	50.074
Total		100.000


Me
N
N
H
3da
4.04
3.00
2.00
0.99
0.98
0.99
1.00
1.00
1.00
0.99
0.99
abundance
11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
10.7384
8.2774 8.2751 8.2683 8.2648
7.6556 7.6430
7.1586 7.1448
6.9627
6.3111 6.2974 6.2836 6.2768
5.2404 5.2198 5.1178 5.1053 4.9976 4.9632
2.3431
0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari113_proton-1-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari113
Solvent = CHLOROFORM-D
Actual_Start_Time = 26-FEB-2021 15:33:55
Revision_Time = 15-MAR-2021 16:33:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 22.1[dC]
X_90_Width = 7.7[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 7[dB]
X_Pulse = 3.85[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

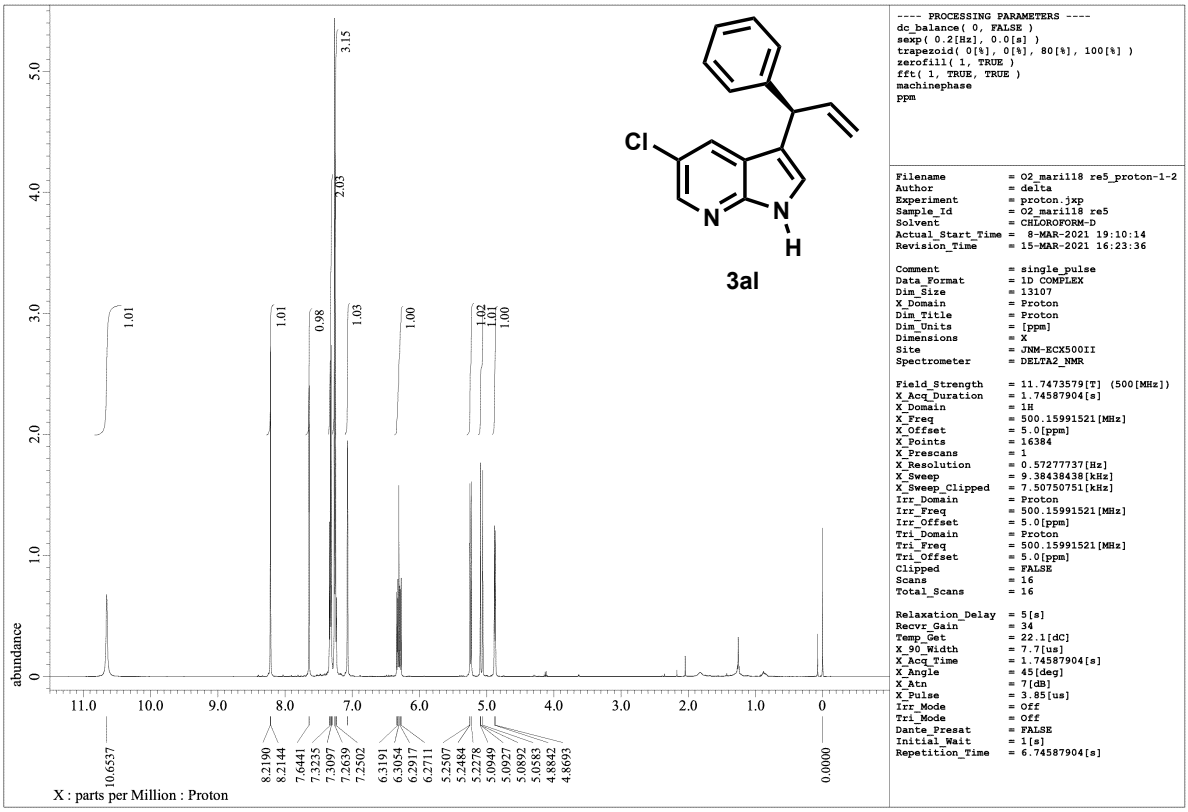

Me
N
N
H
3da
abundance
0.5 0.4 0.3 0.2 0.1 0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
149.3291
142.5188
140.6302
139.3997
136.1090
130.4241
128.1254
128.0968
126.4371
125.9888
123.5375
119.7412
116.1357
115.9545
115.2964
77.2575
77.0000
76.7520
42.8719
19.4744
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari113_carbon-1-2.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari113
Solvent = CHLOROFORM-D
Actual_Start_Time = 26-FEB-2021 15:37:01
Revision_Time = 15-MAR-2021 16:28:42
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.6[dC]
X_90_Width = 11.2[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.73333333[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	14.29	0.090
2	15.94	99.910
Total		100.000



Peak#	Ret. Time	Area%
1	14.11	50.170
2	16.06	49.830
Total		100.000



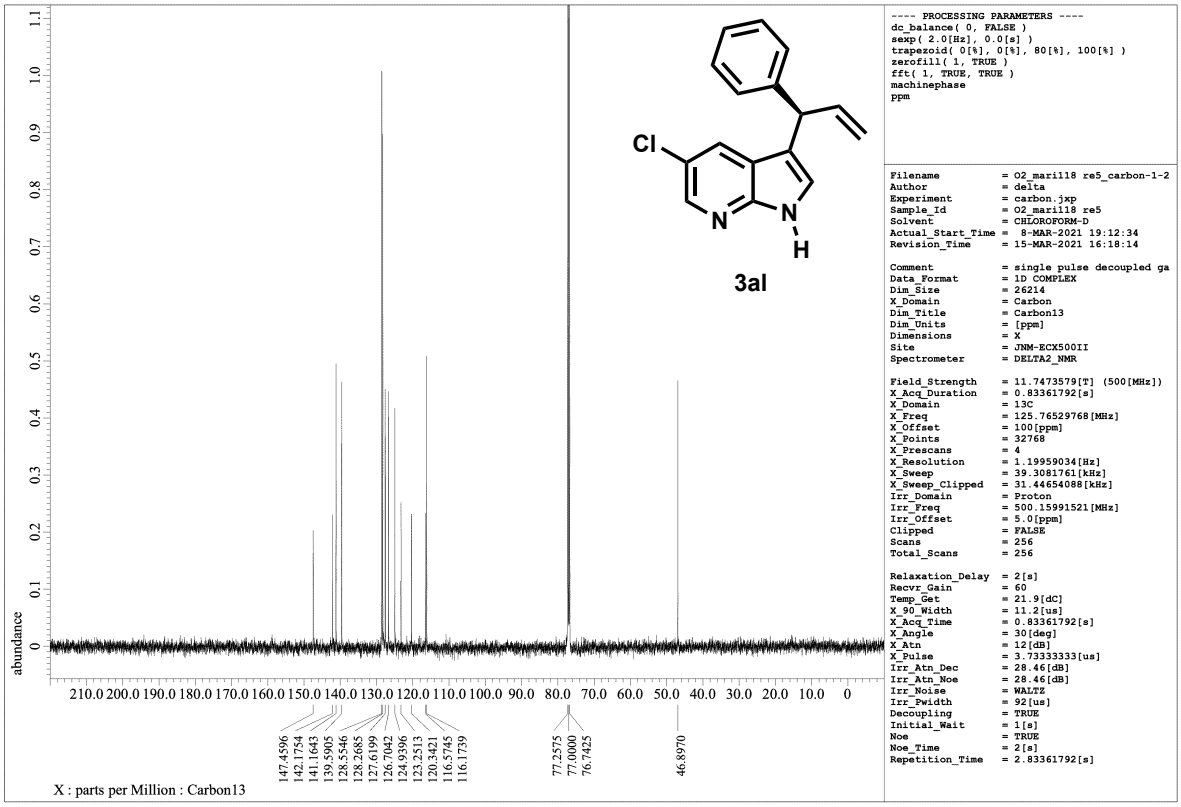

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = O2_mari118 re5_proton-1-2
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari118 re5
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  8-MAR-2021 19:10:14
Revision_Time       = 15-MAR-2021 16:23:36

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 22.1[dC]
X_90_Width          = 7.7[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 7[dB]
X_Pulse             = 3.85[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



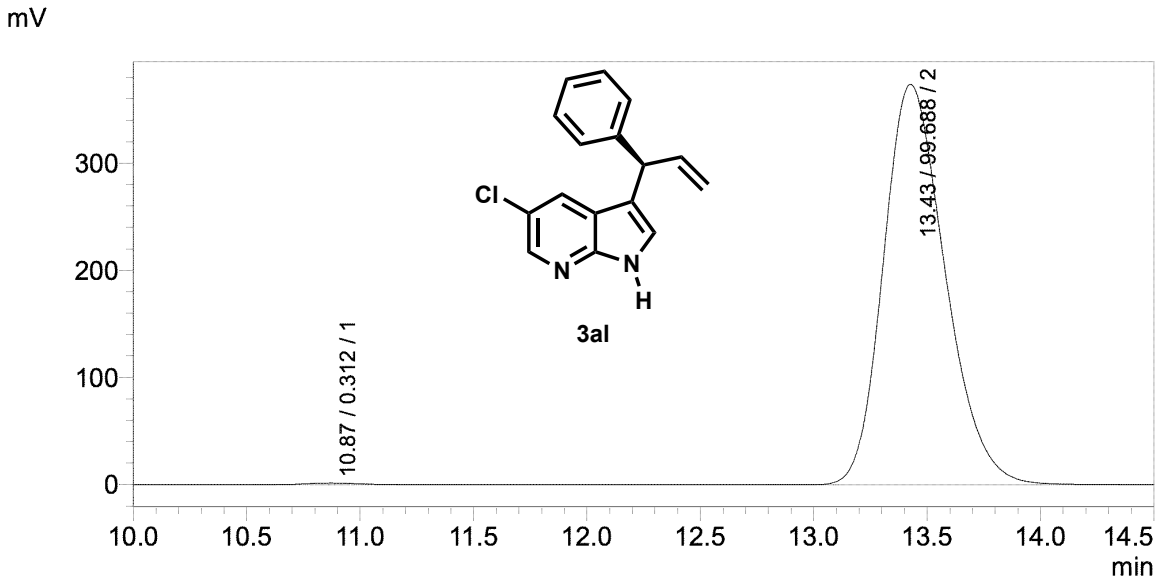

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = O2_mari118 re5_carbon-1-2
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari118 re5
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  8-MAR-2021 19:12:34
Revision_Time       = 15-MAR-2021 16:18:14

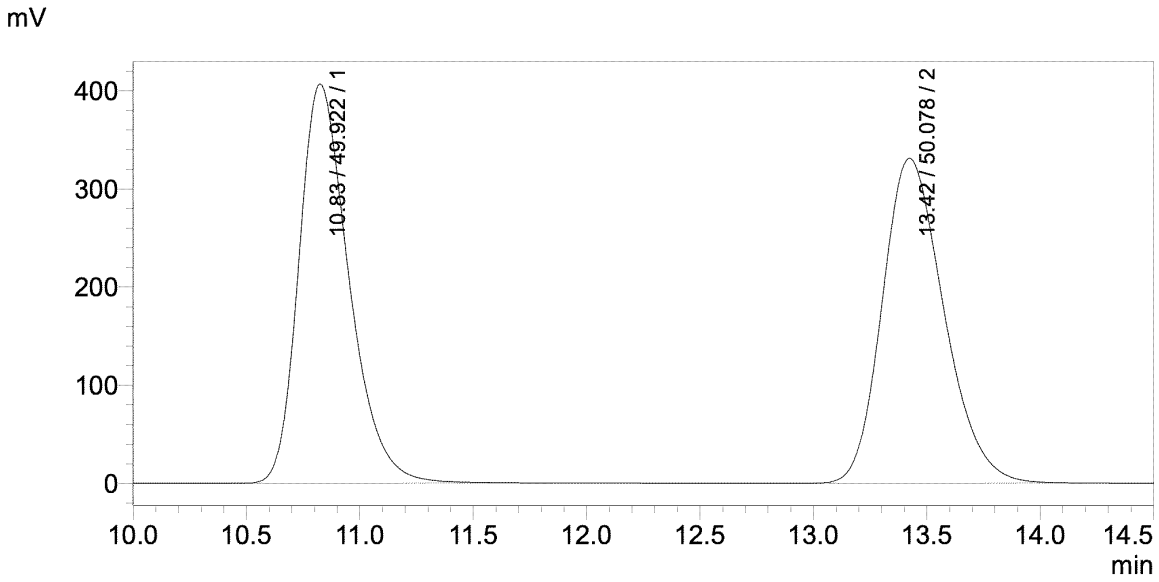
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

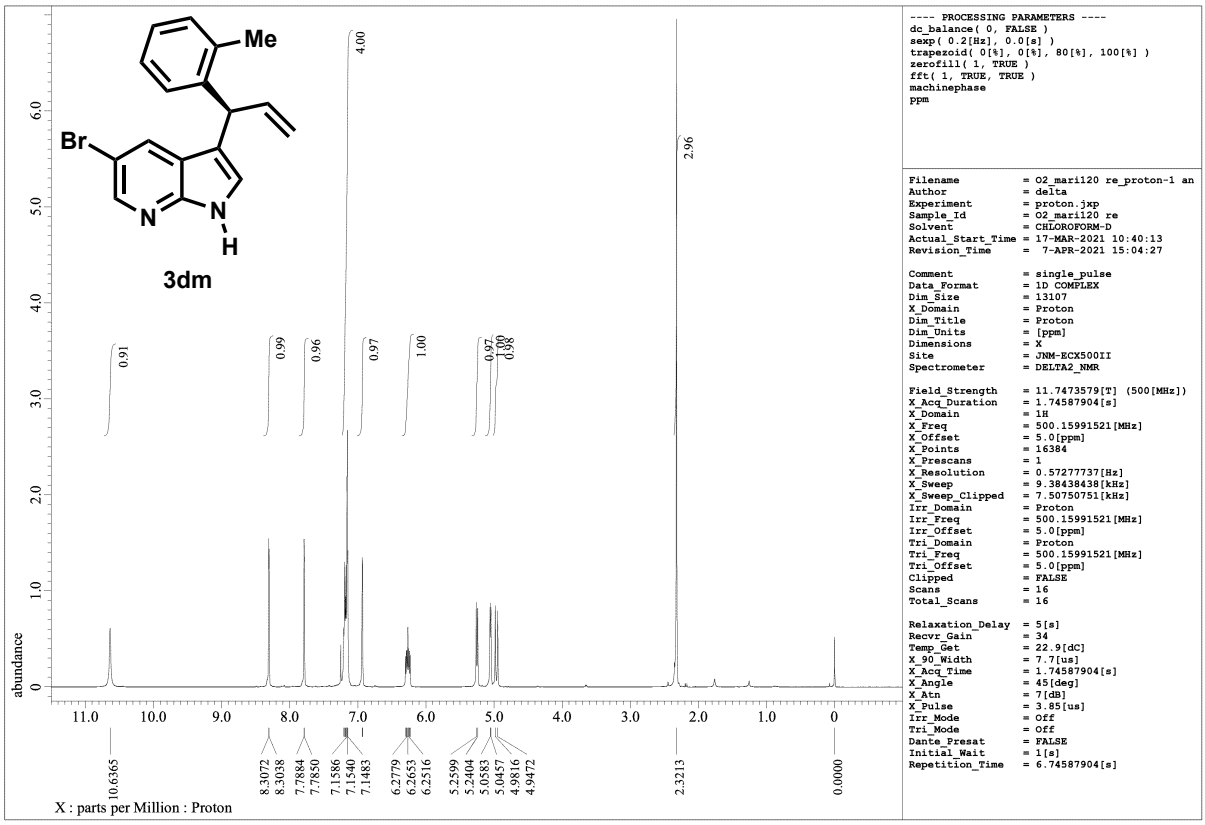
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 21.9[dC]
X_90_Width          = 11.2[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.73333333[us]
Irr_Atn_Dec         = 28.46[dB]
Irr_Atn_Noe         = 28.46[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```



Peak#	Ret. Time	Area%
1	10.87	0.312
2	13.43	99.688
Total		100.000

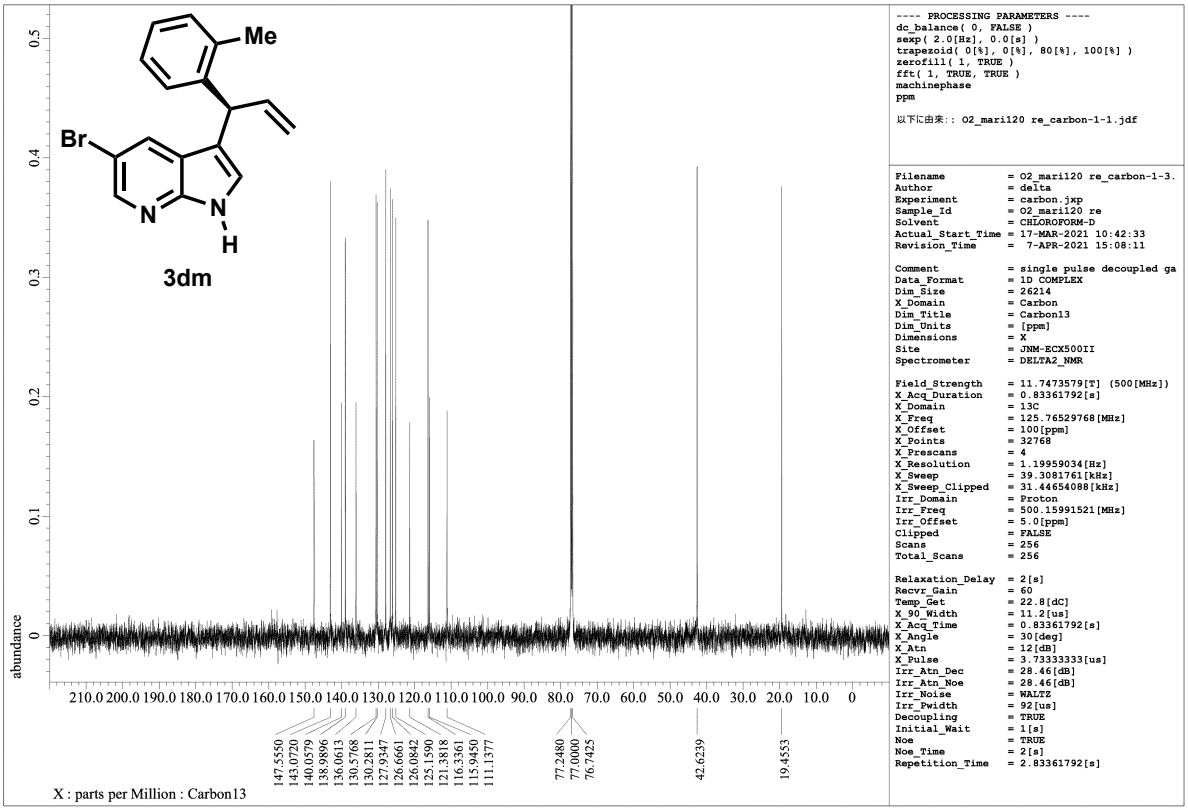


Peak#	Ret. Time	Area%
1	10.83	49.922
2	13.42	50.078
Total		100.000




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

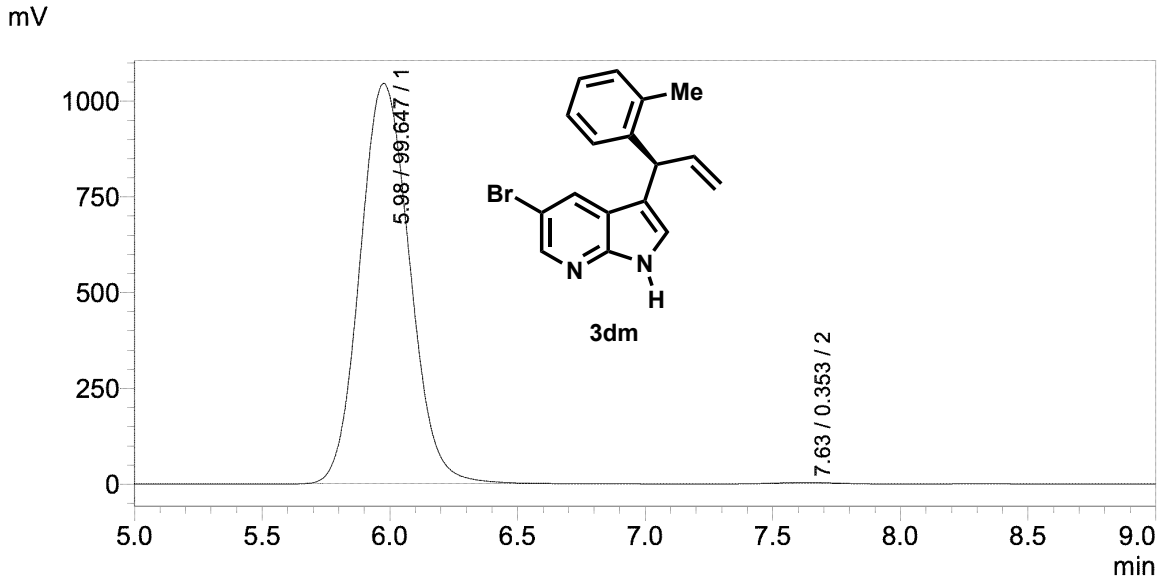
Filename	= O2_mari120 re_proton-1 an
Author	= delta
Experiment	= proton.jxp
Sample_Id	= O2_mari120 re
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 17-MAR-2021 10:40:13
Revision_Time	= 7-APR-2021 15:04:27
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECX500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 1.74587904[s]
X_Domain	= 1H
X_Freq	= 500.15991521[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.57277737[Hz]
X_Sweep	= 9.38438438[kHz]
X_Sweep_Clipped	= 7.50750751[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 500.15991521[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 5[s]
Recvr_Gain	= 34
Temp_Get	= 22.9[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 1.74587904[s]
X_Angle	= 45[deg]
X_Atn	= 7[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.74587904[s]



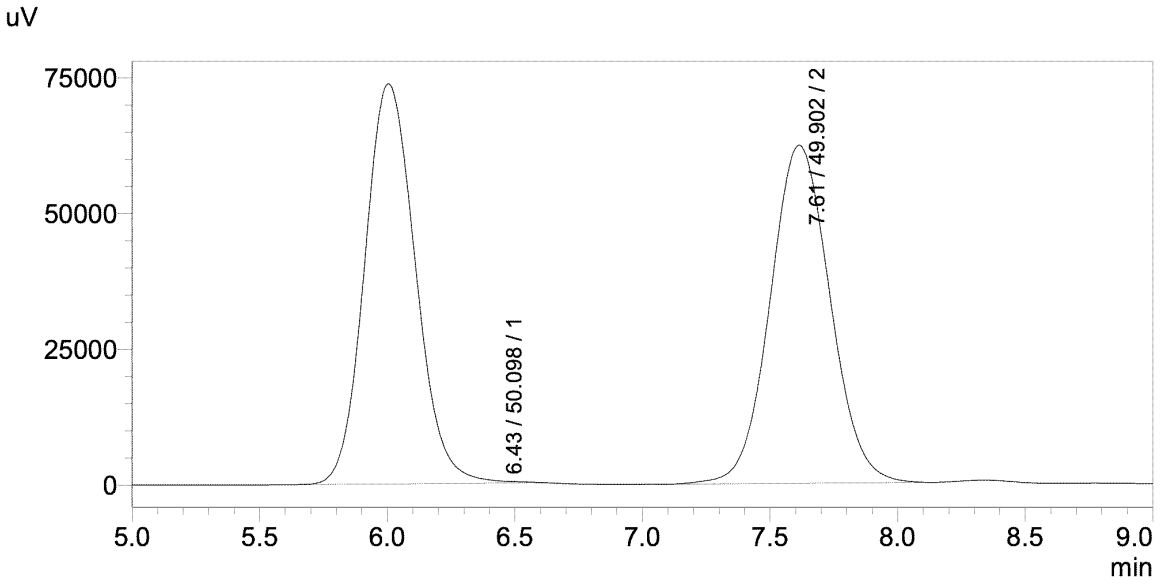

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来: : O2_mari120 re_carbon-1-1.jdf
```

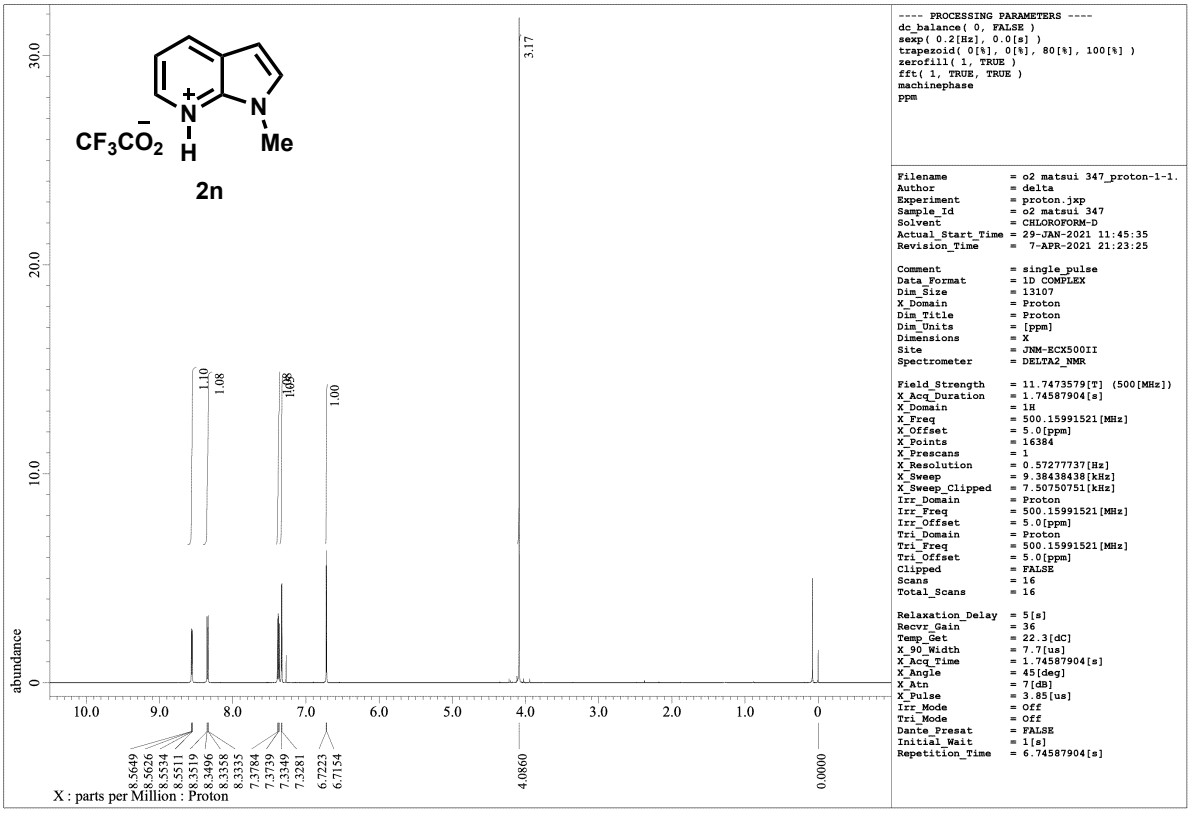
Filename	= O2_mari120 re_carbon-1-3.
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= O2_mari120 re
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 17-MAR-2021 10:42:33
Revision_Time	= 7-APR-2021 15:08:11
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECX500II
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.7473579[T] (500[MHz])
X_Acq_Duration	= 0.83361792[s]
X_Domain	= 13C
X_Freq	= 125.76529768[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.19959034[Hz]
X_Sweep	= 39.3081761[kHz]
X_Sweep_Clipped	= 31.44654088[kHz]
Irr_Domain	= Proton
Irr_Freq	= 500.15991521[MHz]
Irr_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 22.8[dC]
X_90_Width	= 11.2[us]
X_Acq_Time	= 0.83361792[s]
X_Angle	= 30[deg]
X_Atn	= 12[dB]
X_Pulse	= 3.73333333[us]
Irr_Atn_Dec	= 28.46[dB]
Irr_Atn_Noe	= 28.46[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 92[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.83361792[s]



Peak#	Ret. Time	Area%
1	5.98	99.647
2	7.63	0.353
Total		100.000



Peak#	Ret. Time	Area%
1	6.43	50.098
2	7.61	49.902
Total		100.000



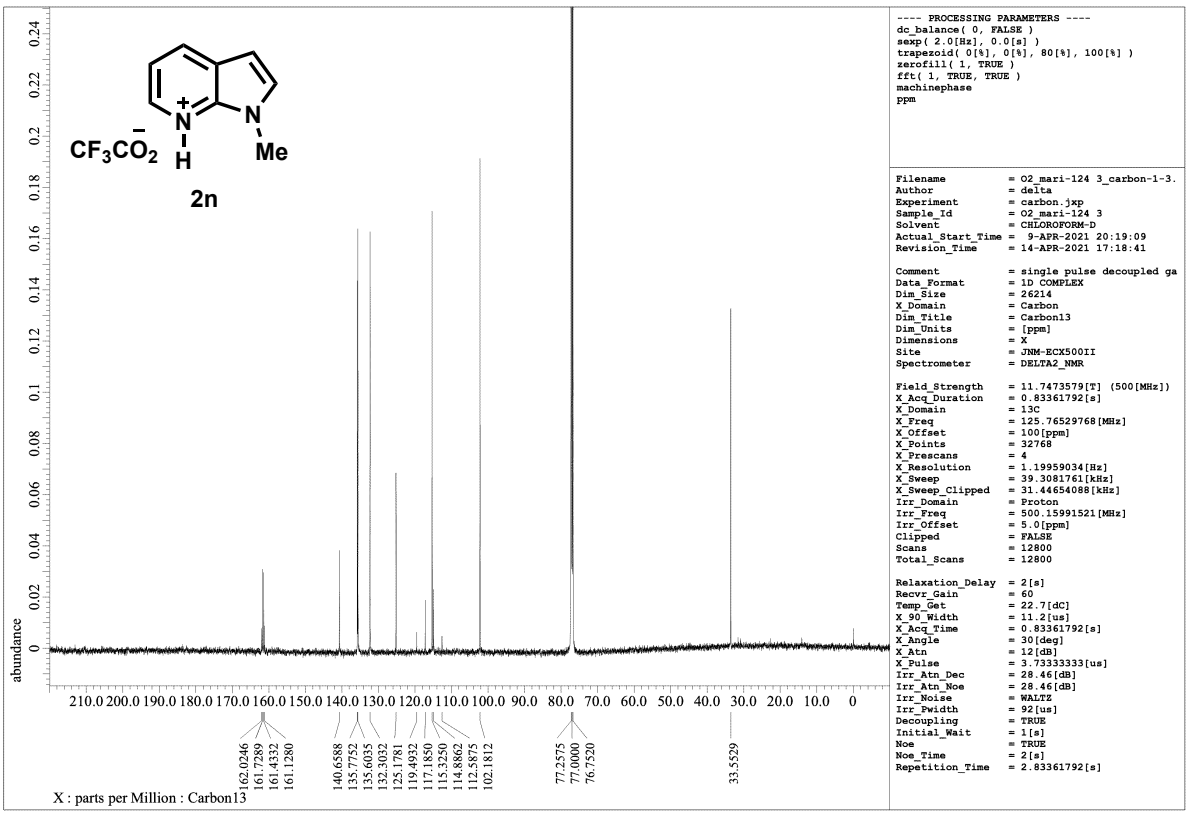

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename           = o2 matsui 347_proton-1-1.
Author             = delta
Experiment         = proton.jxp
Sample_Id          = o2 matsui 347
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 29-JAN-2021 11:45:35
Revision_Time      =  7-APR-2021 21:23:25

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
X_Domain           = Proton
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX500II
Spectrometer       = DELTA2_NMR

Field_Strength     = 11.7473579[T] (500[MHz])
X_Acq_Duration     = 1.74587904[s]
X_Domain           = 1H
X_Freq             = 500.15991521[MHz]
X_Offset           = 5.0[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.57277737[Hz]
X_Sweep            = 9.38438438[kHz]
X_Sweep_Clipped    = 7.50750751[kHz]
Irr_Domain         = Proton
Irr_Freq           = 500.15991521[MHz]
Irr_Offset         = 5.0[ppm]
Tri_Domain         = Proton
Tri_Freq           = 500.15991521[MHz]
Tri_Offset         = 5.0[ppm]
Clipped            = FALSE
Scans              = 16
Total_Scans        = 16

Relaxation_Delay   = 5[s]
Recvr_Gain         = 36
Temp_Get           = 22.3[dC]
X_90_Width         = 7.7[us]
X_Acq_Time         = 1.74587904[s]
X_Angle            = 45[deg]
X_Atn              = 7[dB]
X_Pulse            = 3.85[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 6.74587904[s]
```



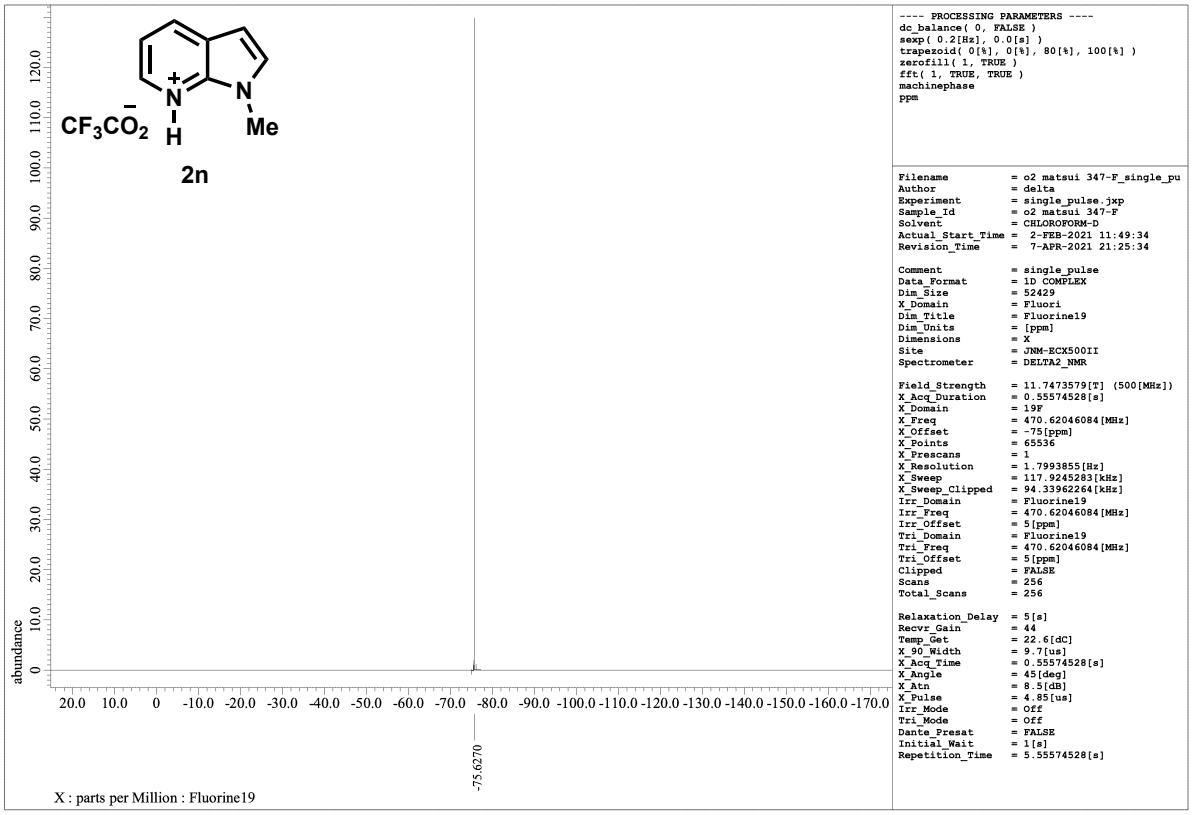

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

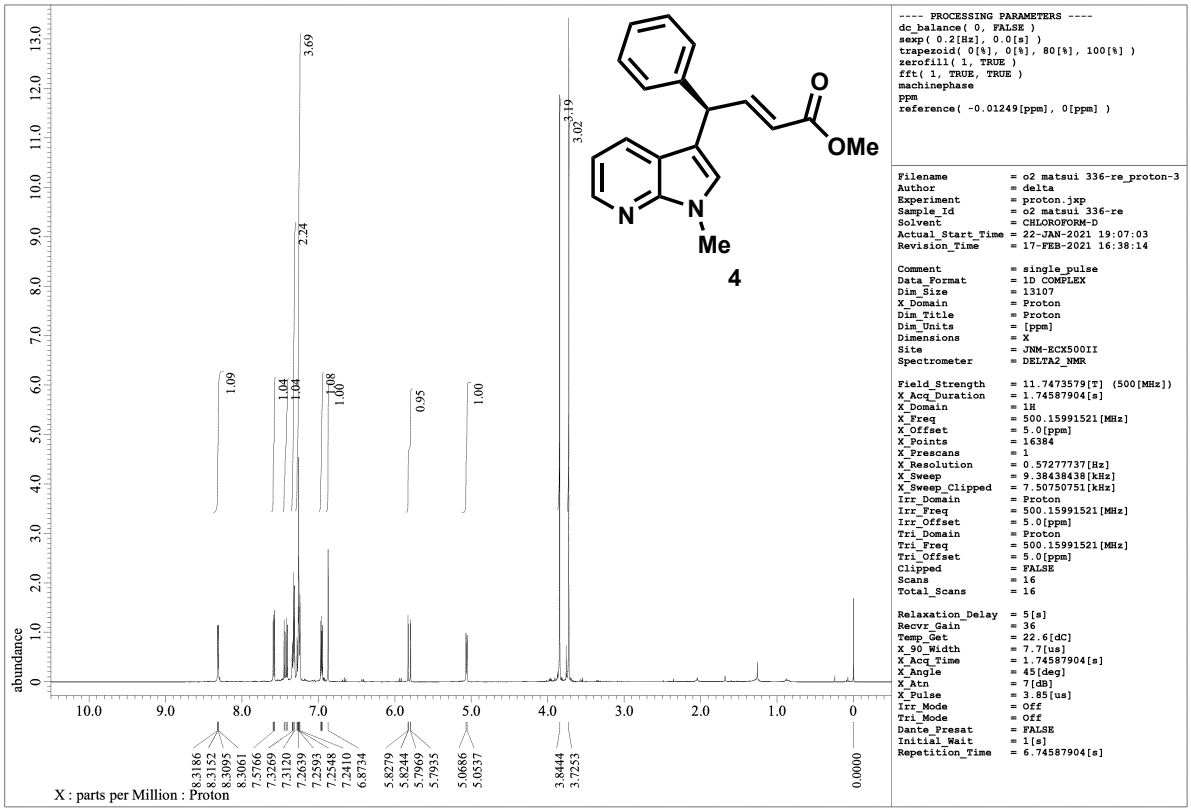
Filename           = O2_mari-124 3_carbon-1-3.
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = O2_mari-124 3
Solvent            = CHLOROFORM-D
Actual_Start_Time  =  9-APR-2021 20:19:09
Revision_Time      = 14-APR-2021 17:18:41

Comment            = single pulse decoupled ga
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = Carbon
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX500II
Spectrometer       = DELTA2_NMR

Field_Strength     = 11.7473579[T] (500[MHz])
X_Acq_Duration     = 0.83361792[s]
X_Domain           = 13C
X_Freq             = 125.76529768[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 1.19959034[Hz]
X_Sweep            = 39.3081761[kHz]
X_Sweep_Clipped    = 31.44654088[kHz]
Irr_Domain         = Proton
Irr_Freq           = 500.15991521[MHz]
Irr_Offset         = 5.0[ppm]
Clipped            = FALSE
Scans              = 12800
Total_Scans        = 12800

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.7[dC]
X_90_Width         = 11.2[us]
X_Acq_Time         = 0.83361792[s]
X_Angle            = 30[deg]
X_Atn              = 12[dB]
X_Pulse            = 3.73333333[us]
Irr_Atn_Dec        = 28.46[dB]
Irr_Atn_Noe        = 28.46[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 92[us]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 2.83361792[s]
```


2n
abundance
X : parts per Million : Fluorine19
-75.6270
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = o2 matsui 347-F_single_pu
Author = delta
Experiment = single_pulse.jxp
Sample_Id = o2 matsui 347-F
Solvent = CHLOROFORM-D
Actual_Start_Time = 2-FEB-2021 11:49:34
Revision_Time = 7-APR-2021 21:25:34
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 52429
X_Domain = Fluori
Dim_Title = Fluorine19
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.55574528[s]
X_Domain = 19F
X_Freq = 470.62046084[MHz]
X_Offset = -75[ppm]
X_Points = 65536
X_Prescans = 1
X_Resolution = 1.7993855[Hz]
X_Sweep = 117.9245283[kHz]
X_Sweep_Clipped = 94.33962264[kHz]
Irr_Domain = Fluorine19
Irr_Freq = 470.62046084[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Fluorine19
Tri_Freq = 470.62046084[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 5[s]
Recvr_Gain = 44
Temp_Get = 22.6[dC]
X_90_Width = 9.7[us]
X_Acq_Time = 0.55574528[s]
X_Angle = 45[deg]
X_Atn = 8.5[dB]
X_Pulse = 4.85[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 5.55574528[s]



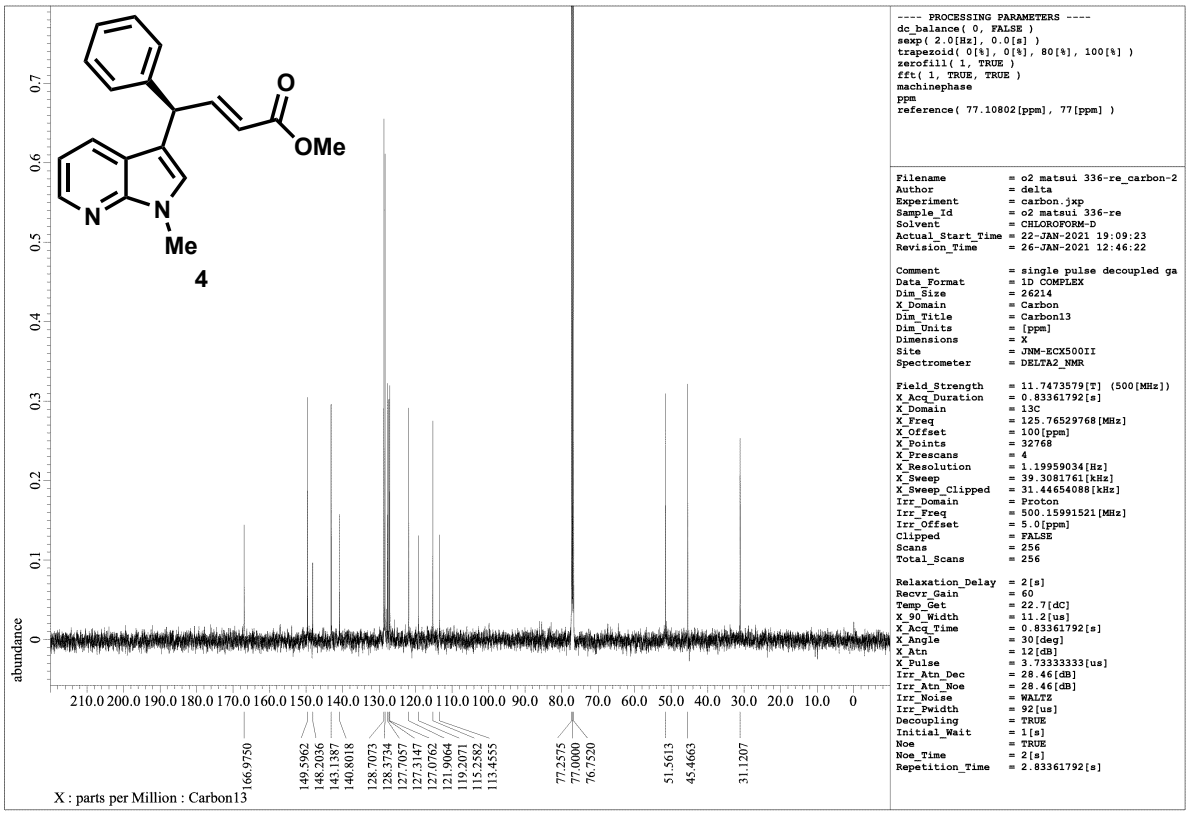

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.01249[ppm], 0[ppm] )
```

```
Filename           = o2 matsui 336-re_proton-3
Author             = delta
Experiment         = proton.jxp
Sample_Id          = o2 matsui 336-re
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 22-JAN-2021 19:07:03
Revision_Time      = 17-FEB-2021 16:38:14

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
X_Domain           = Proton
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX500II
Spectrometer       = DELTA2_NMR

Field_Strength     = 11.7473579[T] (500[MHz])
X_Acq_Duration     = 1.74587904[s]
X_Domain           = 1H
X_Freq             = 500.15991521[MHz]
X_Offset           = 5.0[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.57277737[Hz]
X_Sweep            = 9.38438438[kHz]
X_Sweep_Clipped    = 7.50750751[kHz]
Irr_Domain         = Proton
Irr_Freq           = 500.15991521[MHz]
Irr_Offset         = 5.0[ppm]
Tri_Domain         = Proton
Tri_Freq           = 500.15991521[MHz]
Tri_Offset         = 5.0[ppm]
Clipped            = FALSE
Scans              = 16
Total_Scans        = 16

Relaxation_Delay   = 5[s]
Recvr_Gain         = 36
Temp_Get           = 22.6[dC]
X_90_Width         = 7.7[us]
X_Acq_Time         = 1.74587904[s]
X_Angle            = 45[deg]
X_Atn              = 7[dB]
X_Pulse            = 3.85[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 6.74587904[s]
```



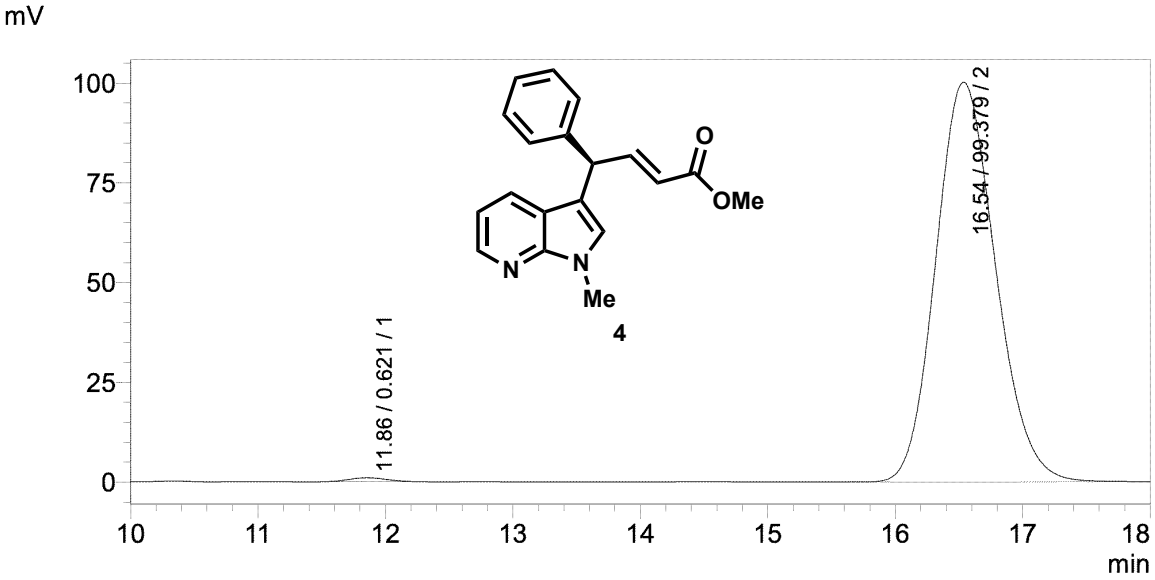

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.10802[ppm], 77[ppm] )
```

```
Filename           = o2 matsui 336-re_carbon-2
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = o2 matsui 336-re
Solvent            = CHLOROFORM-D
Actual_Start_Time  = 22-JAN-2021 19:09:23
Revision_Time      = 26-JAN-2021 12:46:22

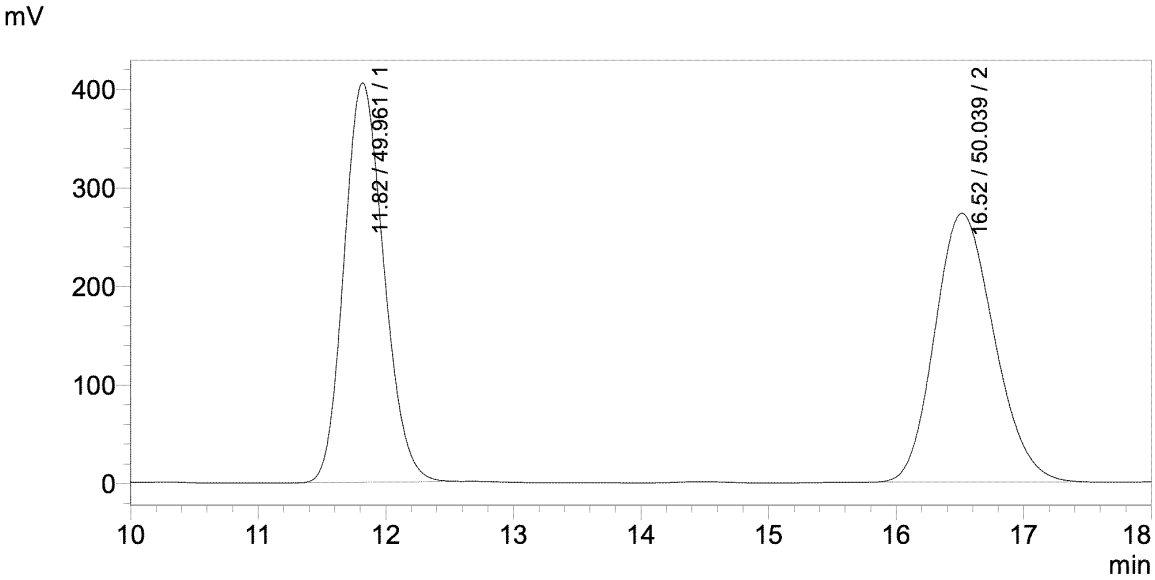
Comment            = single pulse decoupled ga
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = Carbon
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX500II
Spectrometer       = DELTA2_NMR

Field_Strength     = 11.7473579[T] (500[MHz])
X_Acq_Duration     = 0.83361792[s]
X_Domain           = 13C
X_Freq             = 125.76529768[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 1.19959034[Hz]
X_Sweep            = 39.3081761[kHz]
X_Sweep_Clipped    = 31.44654088[kHz]
Irr_Domain         = Proton
Irr_Freq           = 500.15991521[MHz]
Irr_Offset         = 5.0[ppm]
Clipped            = FALSE
Scans              = 256
Total_Scans        = 256

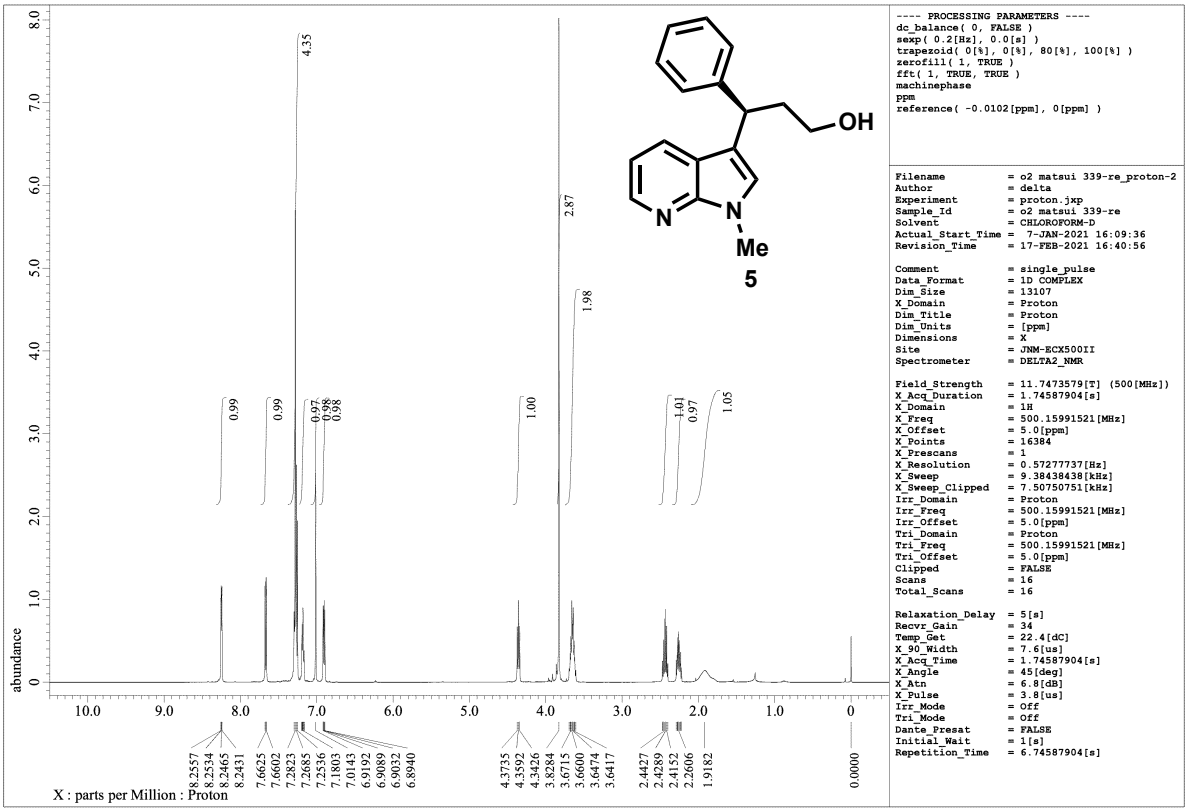
Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.7[dC]
X_90_Width         = 11.2[us]
X_Acq_Time         = 0.83361792[s]
X_Angle            = 30[deg]
X_Atn              = 12[dB]
X_Pulse            = 3.73333333[us]
Irr_Atn_Dec        = 28.46[dB]
Irr_Atn_Noe        = 28.46[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 92[us]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 2.83361792[s]
```



Peak#	Ret. Time	Area%
1	11.86	0.621
2	16.54	99.379
Total		100.000



Peak#	Ret. Time	Area%
1	11.82	49.961
2	16.52	50.039
Total		100.000



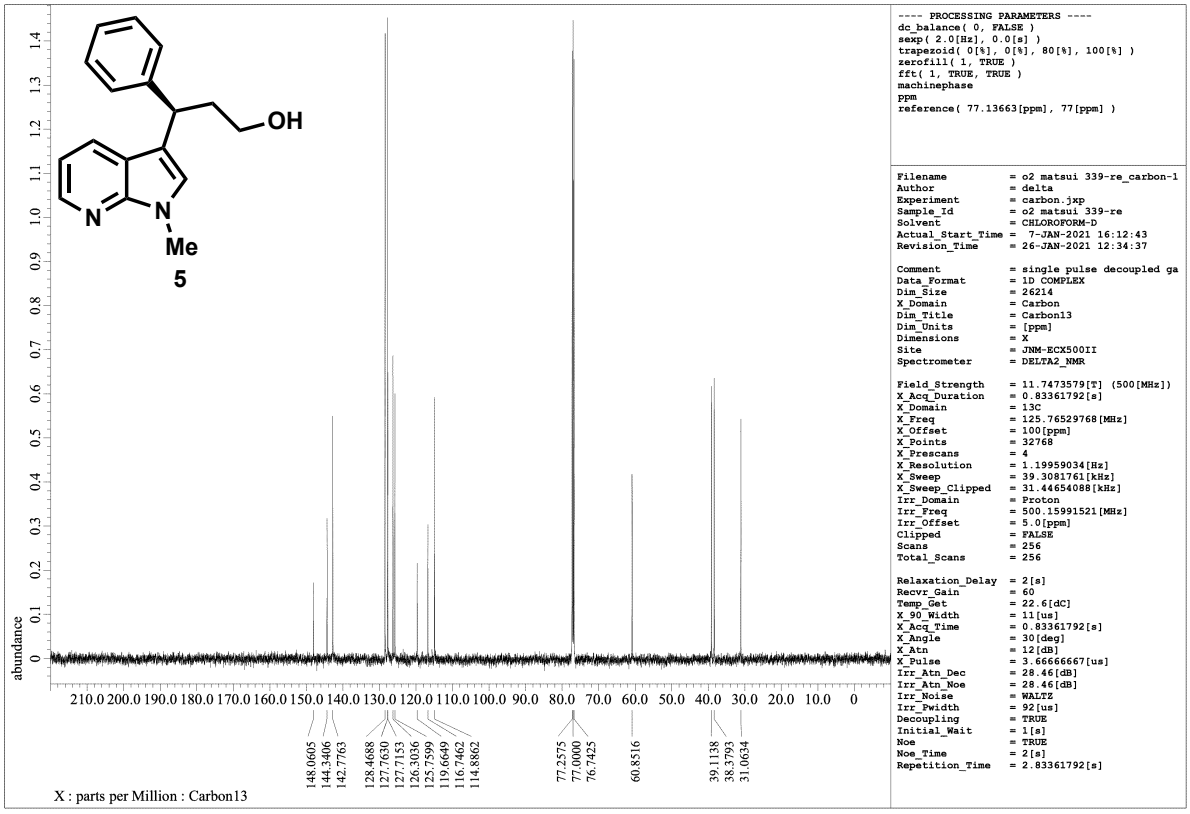

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.0102[ppm], 0[ppm] )
```

```
Filename            = o2 matsui 339-re_proton-2
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 matsui 339-re
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-JAN-2021 16:09:36
Revision_Time       = 17-FEB-2021 16:40:56

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
X_Domain            = Proton
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 1.74587904[s]
X_Domain            = 1H
X_Freq              = 500.15991521[MHz]
X_Offset            = 5.0[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.57277737[Hz]
X_Sweep             = 9.38438438[kHz]
X_Sweep_Clipped     = 7.50750751[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Tri_Domain          = Proton
Tri_Freq            = 500.15991521[MHz]
Tri_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 16
Total_Scans         = 16

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 22.4[dC]
X_90_Width          = 7.6[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.8[dB]
X_Pulse             = 3.8[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```



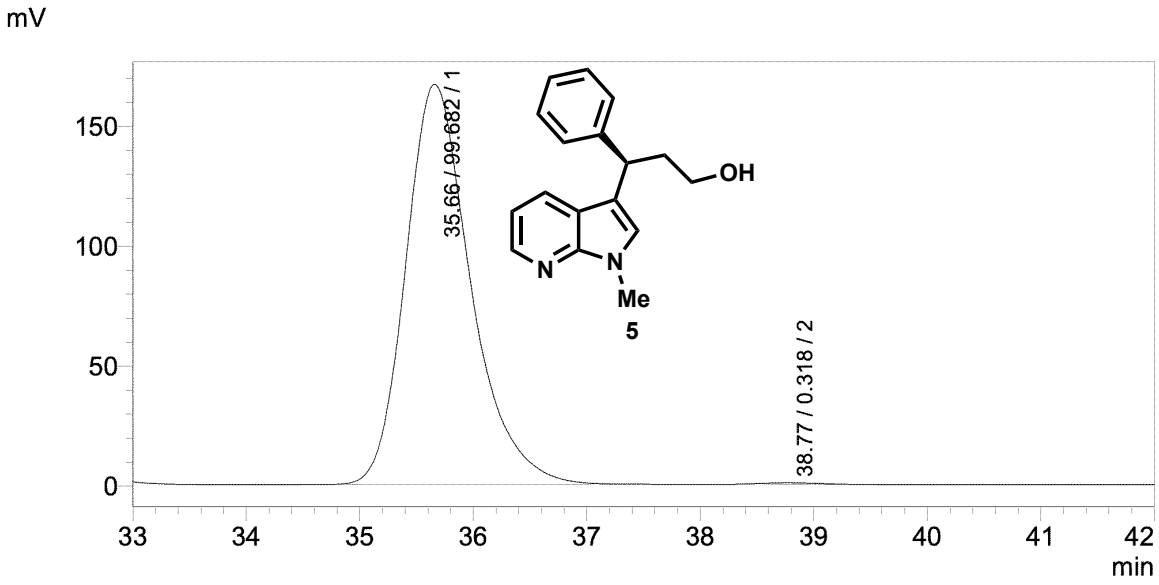

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.13663[ppm], 77[ppm] )
```

```
Filename            = o2 matsui 339-re_carbon-1
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 matsui 339-re
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-JAN-2021 16:12:43
Revision_Time       = 26-JAN-2021 12:34:37

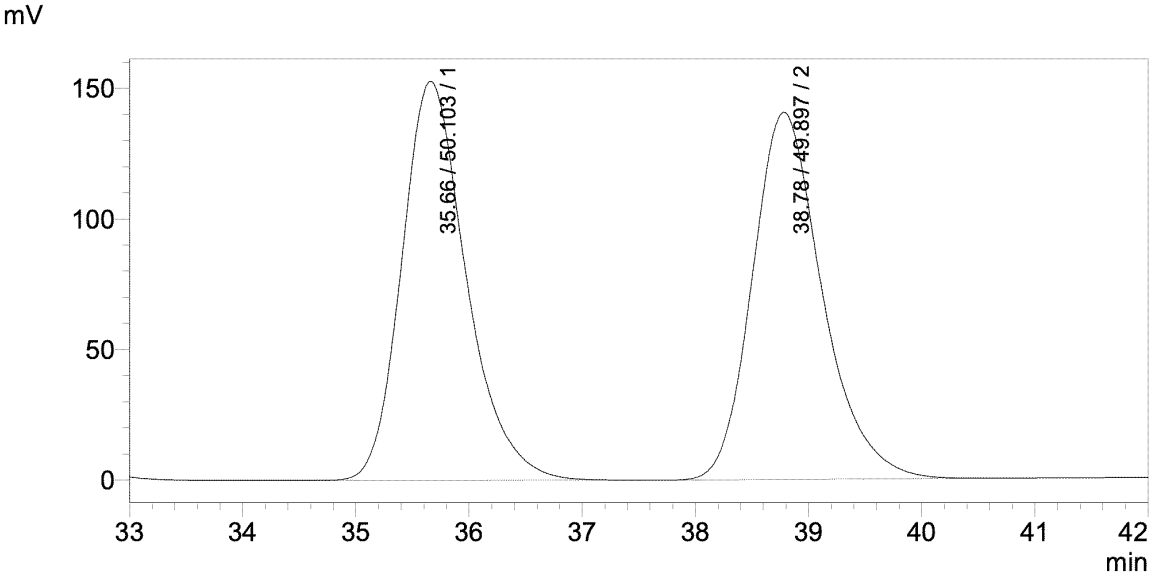
Comment             = single pulse decoupled ga
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
X_Domain            = Carbon
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX500II
Spectrometer        = DELTA2_NMR

Field_Strength      = 11.7473579[T] (500[MHz])
X_Acq_Duration      = 0.83361792[s]
X_Domain            = 13C
X_Freq              = 125.76529768[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 1.19959034[Hz]
X_Sweep             = 39.3081761[kHz]
X_Sweep_Clipped     = 31.44654088[kHz]
Irr_Domain          = Proton
Irr_Freq            = 500.15991521[MHz]
Irr_Offset          = 5.0[ppm]
Clipped             = FALSE
Scans               = 256
Total_Scans         = 256

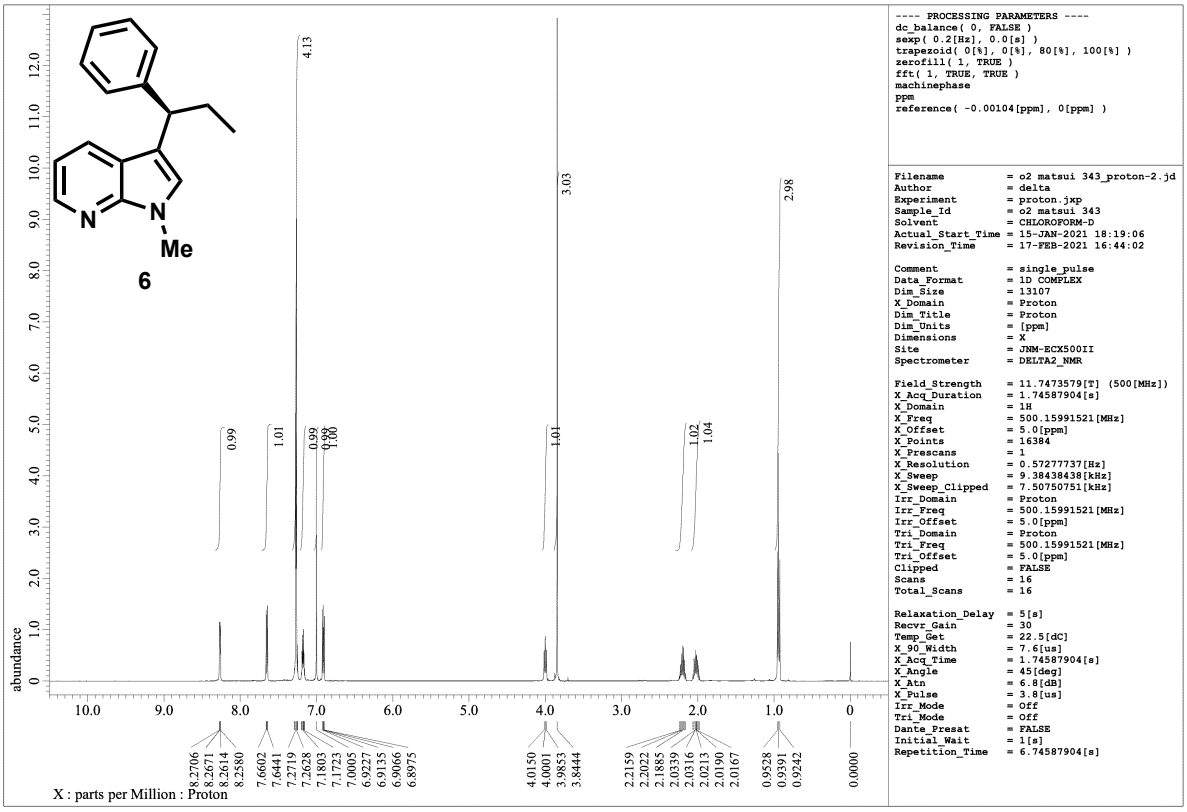
Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.6[dC]
X_90_Width          = 11[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12[dB]
X_Pulse             = 3.66666667[us]
Irr_Atn_Dec         = 28.46[dB]
Irr_Atn_Noe         = 28.46[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 92[us]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 2.83361792[s]
```

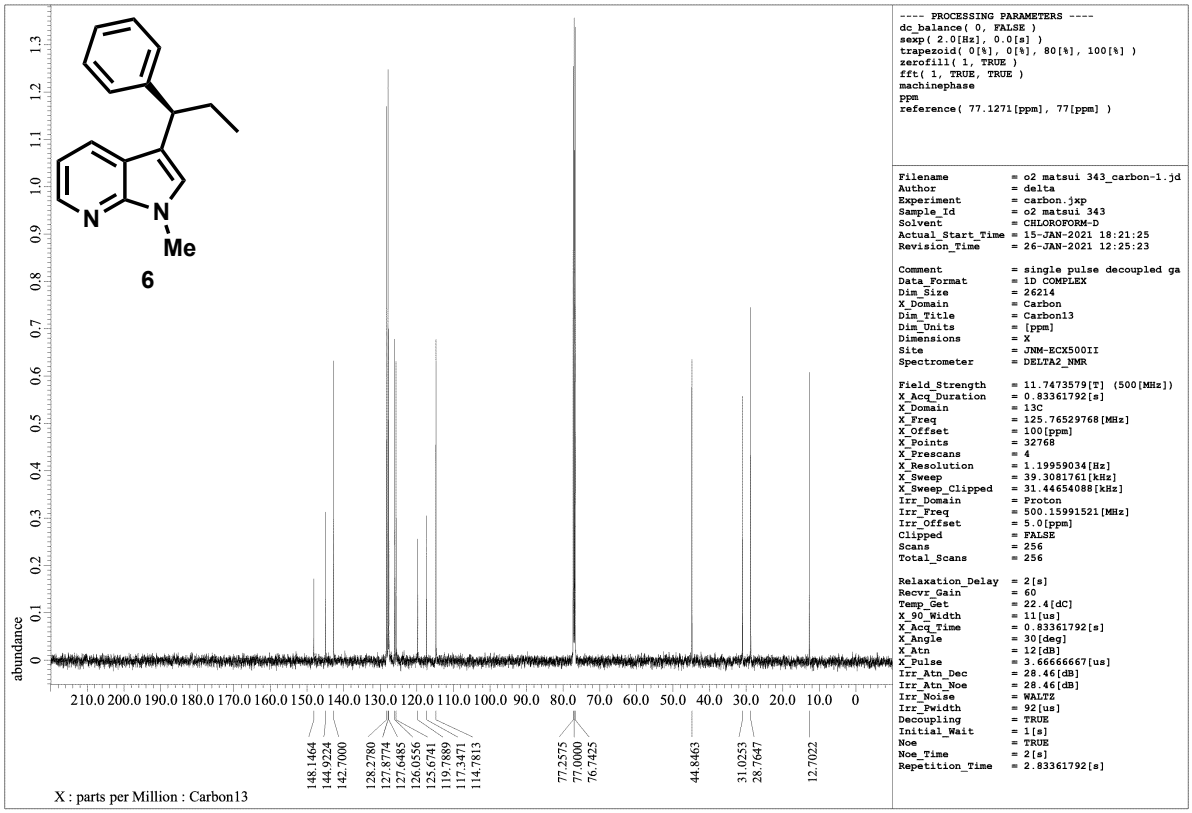


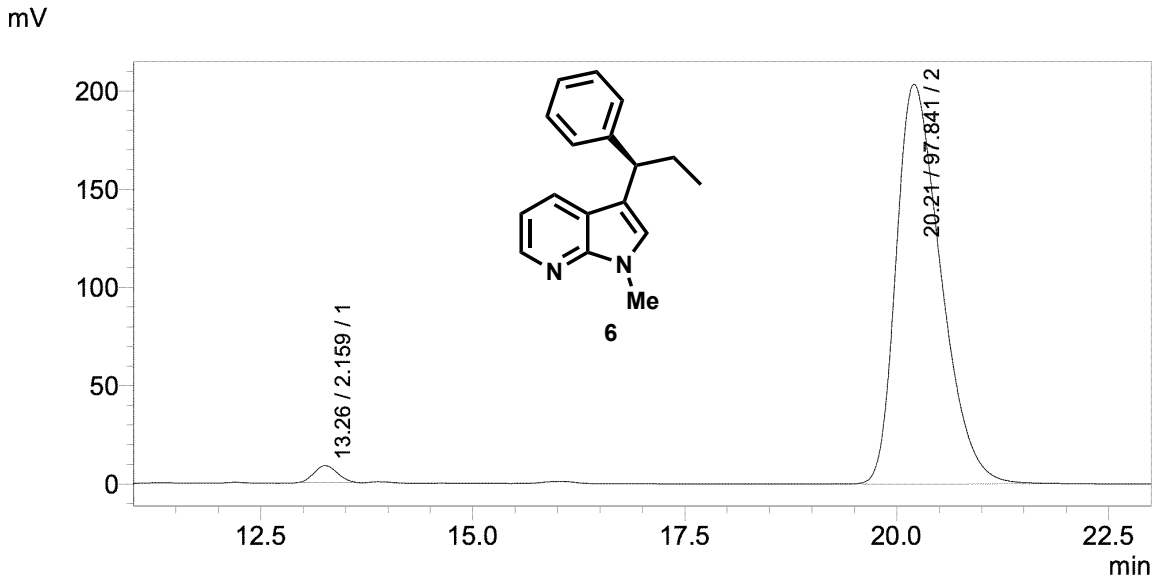
Peak#	Ret. Time	Area%
1	35.66	99.682
2	38.77	0.318
Total		100.000



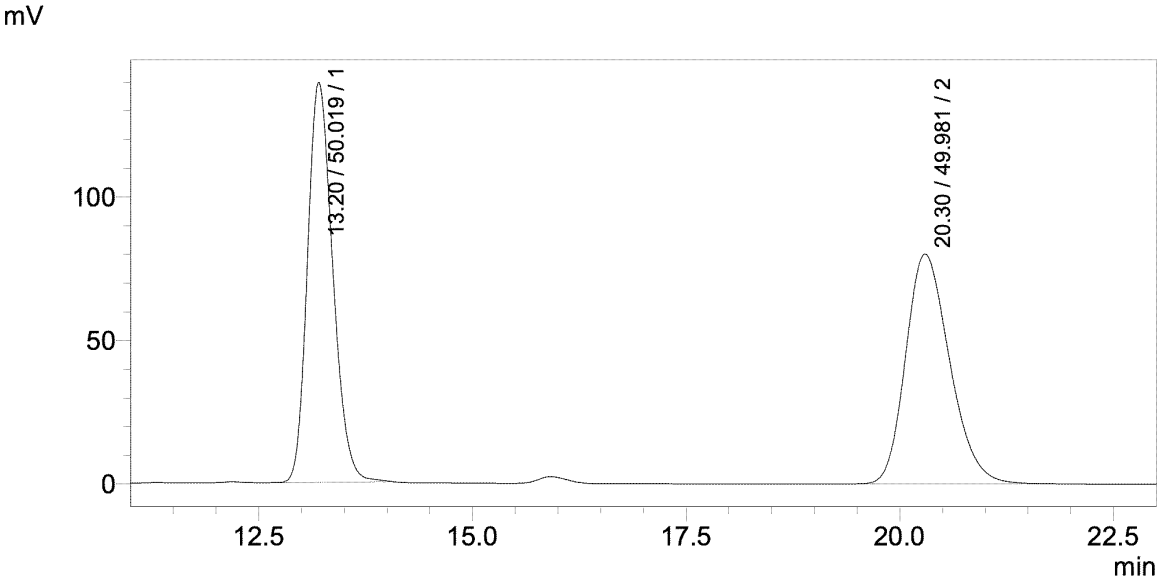
Peak#	Ret. Time	Area%
1	35.66	50.103
2	38.78	49.897
Total		100.000


Me
6
4.13
3.03
2.98
0.99
1.01
0.99
0.99
1.00
1.01
1.02
1.04
abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
8.2706 8.2671 8.2614 8.2580 7.6602 7.6441 7.2719 7.2628 7.1803 7.1723 7.0005 6.9227 6.9135 6.9066 6.8975
4.0150 4.0001 3.9853 3.8444
2.2159 2.2022 2.1885 2.0339 2.0316 2.0213 2.0190 2.0167
0.9528 0.9391 0.9242
0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(-0.00104[ppm], 0[ppm])
Filename = o2 matsui 343_proton-2.jd
Author = delta
Experiment = proton.jxp
Sample_Id = o2 matsui 343
Solvent = CHLOROFORM-D
Actual_Start_Time = 15-JAN-2021 18:19:06
Revision_Time = 17-FEB-2021 16:44:02
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = Proton
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 1.74587904[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.57277737[Hz]
X_Sweep = 9.38438438[kHz]
X_Sweep_Clipped = 7.50750751[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
Tri_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5[s]
Recvr_Gain = 30
Temp_Get = 22.5[dC]
X_90_Width = 7.6[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.8[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]


Me
6
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
148.1464 144.9224 142.7000 128.2780 127.8774 127.6485 126.0556 125.6741 119.7889 117.3471 114.7813
77.2575 77.0000 76.7425
44.8463
31.0253 28.7647
12.7022
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(77.1271[ppm], 77[ppm])
Filename = o2 matsui 343_carbon-1.jd
Author = delta
Experiment = carbon.jxp
Sample_Id = o2 matsui 343
Solvent = CHLOROFORM-D
Actual_Start_Time = 15-JAN-2021 18:21:25
Revision_Time = 26-JAN-2021 12:25:23
Comment = single pulse decoupled ga
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECX500II
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.4[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 28.46[dB]
Irr_Atn_Noe = 28.46[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 92[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.83361792[s]



Peak#	Ret. Time	Area%
1	13.26	2.159
2	20.21	97.841
Total		100.000



Peak#	Ret. Time	Area%
1	13.20	50.019
2	20.30	49.981
Total		100.000