Supporting Information for

Synthesis of Azafluoranthenes by Iridium-Catalyzed [2+2+2] Cycloaddition and Evaluation of their Fluorescent Properties

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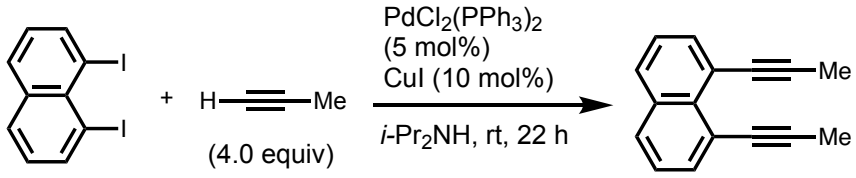
Contents of the Supporting Information

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, m: multiplet. High-resolution mass spectra were obtained with a JEOL JMS-700 Mstation. IR spectra were measured on a JASCO FT/IR-4100 spectrometer. Absorption spectra were obtained with a JASCO V-650. Fluorescence spectra were measured on a Shimadzu RF-6000. The products were purified by column chromatography on 63-210 mesh silica gel (Kanto Kagaku; Silica Gel 60N). Cells were incubated using a CO_2 incubator (MCO-18AIC, SANYO) at 37 ˚C in 5% CO_2. Cells were observed using a C2 confocal laser scanning microscope (Nikon). Cell viability was recorded on a xMark^{TM} microplate absorbance spectrophotometer (BIO RAD). SK-BR-3 was purchased from American Type Culture Collection (ATCC). All solvents were dried and distilled before use by the usual procedures. $[IrCl(cod)_2]$,[1] **1a** [22360-77-6], **1b** [17694-87-0], **2m** [14271-73-9],[2] **2n** [1530-89-8],[3] **2u** [1530-88-7],[3] **2v** [1530-87-6],[4] **2w** [2050-54-6],[3] and **2x** [18773-77-8][3] were prepared acoording to the literature. *rac*-BINAP, nitriles **2a** [100-47-0], **2b** [104-85-8], **2c** [874-90-8], **2d** [4421-09-4], **2e** [1443-80-7], **2f** [455-18-5], **2g** [623-00-7], **2h** [619-72-7], **2i** [1129-35-7], **2j** [100-70-9], **2k** [617-90-3], **2l** [1003-31-2], **2o** [623-49-4], **2p** [75-05-8], **2q** [628-73-9], **2r** [78-82-0], **2s** [5500-21-0] and **2t** [105-56-6] were purchased and used as received.

2. Preparation of Diynes 1a and 1b

Procedure for Preparation of Diyne 1a

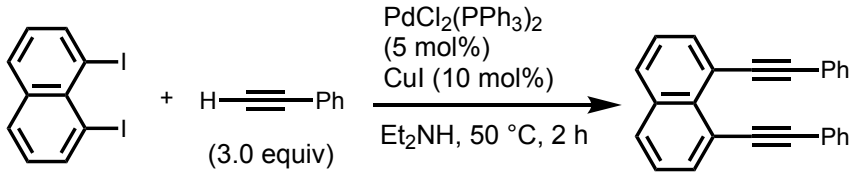


1a

To a solution of CuI (38.3 mg, 0.201 mmol) and $PdCl_2(PPh_3)_2$ (69.8 mg, 0.0994 mmol) in *i*-Pr_2NH (16 mL) were added 1,8-diiodonaphthelene (757.0 mg, 1.992 mmol) and 1-propyne (8.0 mL, 8.0 mmol, 1.0 M solution in THF) at room temperature. After the mixture was stirred for 22 h, the white solid was filtered off with Celite. sat. NH_4Cl aq. was added, and the mixture was extracted with Et_2O. The combined organic extracts were dried

($MgSO_4$), filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography (Hexane/CH_2Cl_2 = 95/5) to give **1a** (348.7 mg, 1.707 mmol, 86% yield) as a brown solid (m.p. 112.8 °C-112.9 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 8.6, 1.2 Hz, 2H), 7.66 (dd, *J* = 7.5, 1.2 Hz, 2H), 7.35 (t, *J* = 7.7 Hz, 2H), 2.20 (s, 6H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 134.2, 134.1, 131.7, 128.8, 125.3, 121.3, 92.9, 79.8, 5.3; IR (KBr, cm^{-1}) 3234, 3047, 2950, 2909, 2841, 2230, 1568, 1503, 1426, 1372, 1350, 1174, 830, 771, 726, 613; HRMS (FAB$^+$) *m/z* [M+H]$^+$ calcd for $C_{16}H_{12}$ 204.0934; found 204.0935.

Procedure for Preparation of Diyne 1b

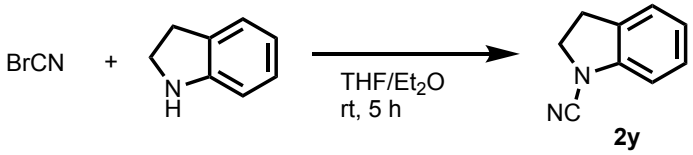


1b

To a solution of CuI (38.2 mg, 0.201 mmol) and $PdCl_2(PPh_3)_2$ (70.1 mg, 0.0999 mmol) in Et_2NH (12.0 mL) were added 1,8-diiodonaphthelene (762.5 mg, 2.007 mmol) and phenylacetylene (609.7 mg, 5.969 mmol) at room temperature. After the mixture was stirred at 50 °C for 2 h, sat. NH_4Cl aq. was added, and the mixture was extracted with EtOAc. The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography (Hexane/CH_2Cl_2 = 95/5) to give **1b** [CAS RN: 17694-87-0][5] (584.6 mg, 1.780 mmol, 89% yield) as a brown solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 7.5, 1.2 Hz, 2H), 7.82 (dd, *J* = 8.0, 1.1 Hz, 2H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.35 (dd, *J* = 8.0, 1.5 Hz, 4H), 7.21-7.16 (m, 2H), 7.11 (t, *J* = 7.5 Hz, 4H).

3. Preparation of Nitrile 2y

Procedure for Preparation of Nitrile 2y



2y

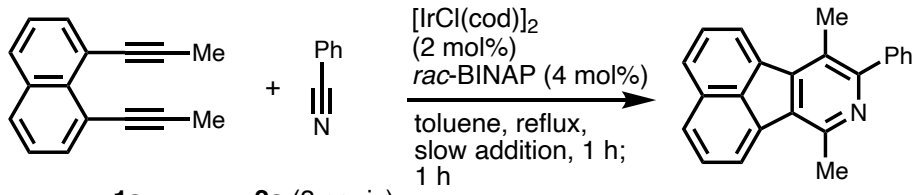
To a solution of BrCN (1894.8 mg, 17.889 mmol) in Et_2O (10 mL) and THF (10 mL) were added indoline (4461.1 mg, 37.438 mmol) at 0 °C. After the mixture was stirred at room temperature for 5 h, the white solid was filtered off with Celite, and the filtrate was

extracted with Et_2O and H_2O. The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography (Hexane/EtOAc = 95/5) to give **2y** [CAS RN: 90036-14-9][6] (1119.0 mg, 7.7613 mmol, 43% yield) as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25-7.20 (m, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 6.99 (t, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 4.05 (t, *J* = 8.6 Hz, 2H), 3.21 (t, *J* = 8.6 Hz, 2H).

4. General Procedure for the [2+2+2] Cycloaddition of Diyne 1a with Nitriles 2 (Scheme 2)

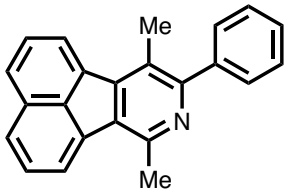
Representative Procedure for the Reaction of Diyne 1a with Nitrile 2a



To the mixture of $[Ir(cod)Cl]_2$ (6.8 mg, 0.010 mmol), *rac*-BINAP (12.5 mg, 0.0201 mmol), and toluene (1.0 mL) was added **2a** (159.0 mg, 1.542 mmol). A solution of **1a** (102.1 mg, 0.4998 mmol) in toluene (1.0 mL) was added over 1 h with a syringe pump under reflux. The vessel was washed with toluene (1.0 mL), and the reaction mixture was stirred for an additional 1 h. After the solvent was removed on a rotary evaporator, the residue was subjected to column chromatography (silica gel, hexane/EtOAc = 9/1) to give **3aa** (126.6 mg, 0.4118mmol, 82% yield).

5. Characterization of 3aa-3at and 4

7,10-Dimethyl-9-phenylacenaphtho[1,2-*c*]pyridine (3aa)

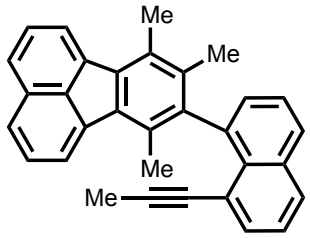


3aa

Compound **3aa** was prepared according to general procedure using **1a** (102.1 mg, 0.4998 mmol) and **2a** (159.0 mg, 1.542 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (9/1) to afford **3aa** (126.6 mg, 0.4118 mmol,

82% yield) as a yellow solid (m.p. 146.8-147.1 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (d, *J* = 7.5 Hz, 1H), 8.01 (d, *J* = 6.9 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.1 Hz, 1H), 7.68 (t, *J* = 8.0 Hz, 2H), 7.63-7.58 (m, 2H), 7.52-7.45 (m, 2H), 7.44-7.39 (m, 1H), 2.98 (s, 3H), 2.69 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 157.9, 150.0, 145.5, 140.9, 135.9, 135.6, 132.6, 130.4, 129.9, 129.5, 128.4, 128.1, 127.9, 127.7, 126.7, 125.2, 124.3, 123.1, 23.4, 17.0; IR (KBr, cm^{-1}) 3053, 2958, 2923, 2853, 1740, 1579, 1560, 1442, 1420, 1404, 1383, 1183, 1020, 822, 771, 752, 700; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{23}H_{18}N$ 308.1434; found 308.1431.

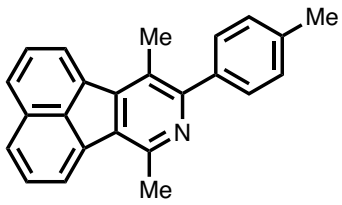
7,8,10-Trimethyl-9-(8-(prop-1-yn-1-yl)naphthalen-1-yl)fluoranthene (4)



4

Compound **4:** brown solid (m.p. 150.8-151.6 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.11 (d, *J* = 6.9 Hz, 1H), 8.00 (d, *J* = 7.5 Hz, 1H), 7.91-7.85 (m, 2H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.66 (t, *J* = 7.7 Hz, 1H), 7.62 (t, *J* = 7.7 Hz, 1H), 7.58-7.50 (m, 2H), 7.38 (t, *J* = 7.7 Hz, 1H), 7.27 (dd, *J* = 6.9, 1.1 Hz, 1H), 2.79 (s, 3H), 2.33 (s, 3H), 2.01 (s, 3H), 1.11 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 142.7, 139.9, 138.4, 138.3, 136.9, 135.8, 134.6, 134.4, 133.4, 133.1, 131.5, 131.4, 130.1, 129.9, 129.1, 128.9, 128.3, 127.8, 127.7, 125.9, 125.8, 125.6, 125.1, 122.9, 122.6, 121.5, 91.7, 78.7, 18.2, 17.9, 16.6, 4.2; IR (KBr, cm^{-1}) 3047, 2986, 2909, 2859, 1572, 1424, 1364, 1346, 831, 823, 770; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{32}H_{24}$ 408.1873; found 408.1876.

7,10-Dimethyl-9-(*p*-tolyl)acenaphtho[1,2-*c*]pyridine (3ab)

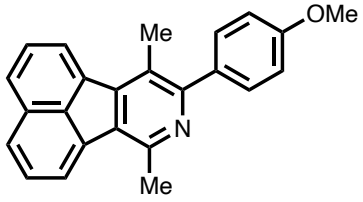


3ab

Compound **3ab** was prepared according to general procedure using **1a** (98.8 mg, 0.4837 mmol) and **2b** (224.4 mg, 1.915 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (8/2) to afford **3ab** (137.7 mg, 0.4284 mmol,

89% yield) as a yellow solid (m.p. 136.2-137.8 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (d, *J* = 6.9 Hz, 1H), 7.99 (d, *J* = 6.9 Hz, 1H), 7.93 (d, *J* = 8.6 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.66 (t, *J* = 7.7 Hz, 2H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.97 (s, 3H), 2.70 (s, 3H), 2.43 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 158.0, 150.0, 145.4, 138.0, 137.4, 136.0, 135.7, 132.6, 130.2, 129.9, 129.5, 128.8, 128.3, 128.1, 127.8, 126.6, 125.1, 124.3, 123.0, 23.4, 21.3, 17.1; IR (KBr, cm^{-1}) 3051, 3035, 2955, 2915, 2860, 1579, 1559, 1444, 1424, 1397, 1228, 1185, 1137, 1032, 1020, 822, 814, 791, 770, 744, 525; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{24}H_{20}N$ 322.1590; found 322.1591.

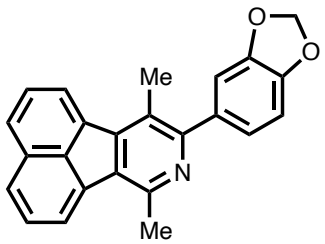
9-(4-Methoxyphenyl)-7,10-dimethylacenaphtho[1,2-*c*]pyridine (3ac)



3ac

Compound **3ac** was prepared according to general procedure using **1a** (105.3 mg, 0.5155 mmol) and **2c** (210.2 mg, 1.579 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (7/3) to afford **3ac** (141.6 mg, 0.4197 mmol, 81% yield) as a yellow solid (m.p. 138.2-139.2 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (d, *J* = 6.9 Hz, 1H), 8.06 (d, *J* = 7.5 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.1 Hz, 1H), 7.72 (t, *J* = 7.5 Hz, 2H), 7.60-7.54 (m, 2H), 7.06-7.00 (m, 2H), 3.88 (s, 3H), 3.00 (s, 3H), 2.75 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 159.4, 157.7, 150.0, 145.6, 136.1, 135.8, 133.4, 132.7, 130.9, 130.1, 130.0, 128.4, 128.2, 127.9, 126.6, 125.3, 124.3, 123.1, 113.6, 55.4, 23.4, 17.2; IR (KBr, cm^{-1}) 3053, 3003, 2955, 2933, 2911, 2835, 1607, 1577, 1513, 1461, 1421, 1400, 1293, 1251, 1179, 1023, 959, 842, 822, 797, 771, 589, 538; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{24}H_{20}NO$ 338.1539; found 338.1539.

9-(Benzo[*d*][1,3]dioxol-5-yl)-7,10-dimethylacenaphtho[1,2-*c*] (3ad)

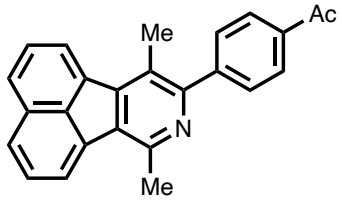


3ad

Compound **3ad** was prepared according to general procedure using **1a** (99.9 mg,

0.4891 mmol) and **2d** (221.7 mg, 1.507 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3ad** (157.7 mg, 0.4488 mmol, 92% yield) as a yellow solid (m.p. 186.5-187.8 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 (d, *J* = 6.9 Hz, 1H), 8.01 (d, *J* = 6.9 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.68 (t, *J* = 7.5 Hz, 2H), 7.14-7.10 (m, 1H), 7.07 (dd, *J* = 7.5, 1.4 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 6.02 (s, 2H), 2.97 (s, 3H), 2.71 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 157.4, 149.9, 147.5, 147.3, 145.6, 135.9, 135.6, 134.9, 132.6, 130.3, 129.9, 128.4, 128.2, 127.9, 126.7, 125.2, 124.3, 123.4, 123.1, 110.3, 108.0, 101.1, 23.4, 17.1; IR (KBr, cm^{-1}) 3049, 2991, 2952, 2892, 2786, 1580, 1563, 1502, 1464, 1441, 1412, 1379, 1335, 1240, 1189, 1173, 1122, 1097, 1064, 1037, 936, 905, 866, 822, 773, 565; HRMS (FAB) *m/z* [M+H]$^+$ calcd for $C_{24}H_{18}NO_2$ 352.1332; found 352.1331.

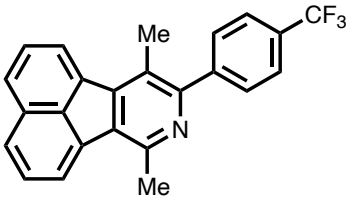
1-(4-(7,10-Dimethylacenaphtho[1,2-*c*]pyridin-9-yl)phenyl)ethan-1-one (3ae)



3ae

Compound **3ae** was prepared according to general procedure using **1a** (102.1 mg, 0.4998 mmol) and **2e** (218.2 mg, 1.503 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (9/1) to afford **3ae** (148.0 mg, 0.4235 mmol, 85% yield) as a yellow solid (m.p. 250.3-253.2 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14-8.11 (m, 1H), 8.11-8.06 (m, 2H), 8.04 (d, *J* = 6.9 Hz, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 8.6 Hz, 1H), 7.74-7.68 (m, 4H), 2.99 (s, 3H), 2.70 (s, 3H), 2.67 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 198.0, 156.5, 150.3, 145.64, 145.60, 136.2, 135.7, 135.3, 132.5, 130.9, 129.9, 128.6, 128.2, 128.0, 127.0, 125.4, 124.4, 123.4, 26.7, 23.3, 17.0; IR (KBr, cm^{-1}) 3050, 2992, 2950, 2914, 2866, 2857, 1677, 1603, 1577, 1415, 1396, 1356, 1265, 956, 849, 826, 776, 737, 693, 627, 602, 574, 590; HRMS (FAB) *m/z* [M+H]$^+$ calcd for $C_{25}H_{20}NO$ 350.1539; found 350.1539.

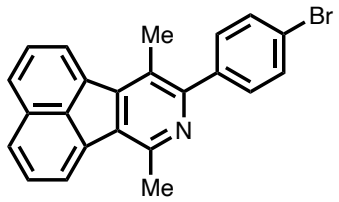
7,10-Dimethyl-9-(4-(trifluoromethyl)phenyl)acenaphtho[1,2-*c*]pyridine (3af)



3af

Compound **3af** was prepared according to general procedure using **1a** (101.8 mg, 0.4984 mmol) and **2f** (257.0 mg, 1.502 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3af** (165.7 mg, 0.4414 mmol, 89% yield) as a yellow solid (m.p. 216.2-218.1 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05 (d, J = 6.9 Hz, 1H), 7.98 (d, J = 6.9 Hz, 1H), 7.93 (d, J = 8.0 Hz, 1H), 7.86 (d, J = 8.3 Hz, 1H), 7.75 (d, J = 8.1 Hz, 2H), 7.71 (d, J = 8.0 Hz, 2H), 7.66 (t, J = 7.8 Hz, 2H), 2.94 (s, 3H), 2.65 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 156.1, 150.3, 145.6, 144.4, 135.5, 135.2, 132.5, 130.9, 129.96, 129.91 (q, J = 21.6 Hz), 128.6, 128.2, 127.9, 126.9, 125.3, 125.2-125.0 (m), 124.29 (q, J = 271.9 Hz), 124.26, 123.4., 23.3, 16.9; ^{19}F NMR (471 MHz, $CDCl_3$) δ -62.4; IR (KBr, cm^{-1}) 3054, 2991, 2958, 2925, 2863, 1614, 1577, 1560, 1447, 1423, 1399, 1324, 1166, 1107, 1065, 1016, 850, 823, 774, 758; HRMS (FAB) *m/z* [M+H]$^{+}$ calcd for $C_{24}H_{17}F_3N$ 376.1308; found .376.1307

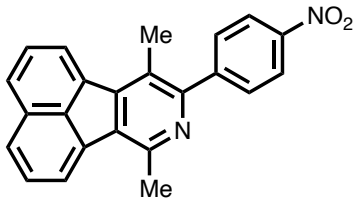
9-(4-Bromophenyl)-7,10-dimethylacenaphtho[1,2-*c*]pyridine (3ag)



3ag

Compound **3ag** was prepared according to general procedure using **1a** (91.8 mg, 0.4494 mmol) and **2g** (274.2 mg, 1.506 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3ag** (131.3 mg, 0.3399 mmol, 76% yield) as a yellow solid (m.p. 248.8-251.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (d, J = 6.9 Hz, 1H), 8.08 (d, J = 6.9 Hz, 1H), 8.00 (d, J = 8.0 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.73 (t, J = 7.5 Hz, 2H), 7.63 (d, J = 8.6 Hz, 2H), 7.50 (d, J = 8.0 Hz, 2H), 3.00 (s, 3H), 2.73 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 156.6, 150.3, 145.7, 139.8, 135.8, 135.5, 132.6, 131.3, 130.7, 129.9, 128.6, 128.3, 128.0, 126.9, 125.4, 124.3, 123.4, 122.1, 23.4, 17.0; IR (KBr, cm^{-1}) 3078, 3058, 2993, 2955, 2914, 2854, 1574, 1556, 1487, 1458, 1442, 1422, 1413, 1386, 1364, 1350, 1072, 1011, 831, 815, 793, 771, 746; HRMS (FAB) *m/z* [M+H]$^{+}$ calcd for $C_{23}H_{17}BrN$ 386.0539; found 386.0539.

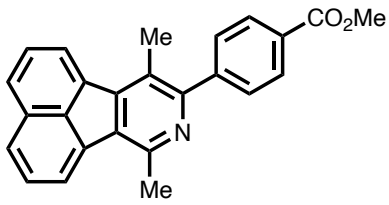
7,10-Dimethyl-9-(4-nitrophenyl)acenaphtho[1,2-*c*]pyridine (3ah)



3ah

Compound **3ah** was prepared according to general procedure using **1a** (94.9 mg, 0.4646 mmol) and **2h** (220.2 mg, 1.487 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3ah** (97.7 mg, 0.2772 mmol, 60% yield) as a brown solid (m.p. 280.0-281.1 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40-8.34 (m, 2H), 8.21 (d, *J* = 6.9 Hz, 1H), 8.14 (d, *J* = 6.9 Hz, 1H), 8.05 (d, *J* = 8.6 Hz, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.85-7.80 (m, 2H), 7.77 (t, *J* = 7.7 Hz, 2H), 3.04 (s, 3H), 2.77 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 155.2, 150.7, 147.5, 147.4, 145.9, 135.5, 135.2, 132.6, 131.4, 130.7, 130.0, 128.9, 128.4, 128.1, 127.3, 125.7, 124.5, 123.8, 123.4, 23.4, 17.0; IR (KBr, cm^{-1}) 3067, 3001, 2965, 2918, 2851, 2837, 1593, 1519, 1445, 1423, 1396, 1342, 1104, 861, 849, 823, 774, 748, 702, 576, 451; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{23}H_{17}N_2O_2$ 353.1285; found 353.1285.

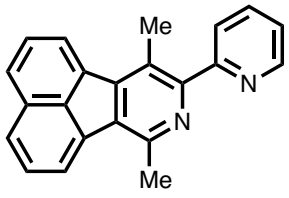
Methyl 4-(7,10-dimethylacenaphtho[1,2-*c*]pyridin-9-yl)benzoate (3ai)



3ai

Compound **3ai** was prepared according to general procedure using **1a** (101.9 mg, 0.4988 mmol) and **2i** (240.1 mg, 1.490 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (8/2) to afford **3ai** (159.0 mg, 0.4351 mmol, 87% yield) as a yellow solid (m.p. 236.6-236.9 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.1 Hz, 2H), 8.11 (d, *J* = 7.4 Hz, 1H), 8.04 (d, *J* = 6.9 Hz, 1H), 7.97 (d, *J* = 8.6 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.73-7.67 (m, 4H), 3.97 (s, 3H), 2.99 (s, 3H), 2.69 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.0, 156.6, 150.3, 145.6, 145.4, 135.7, 135.3, 132.5, 130.8, 129.9, 129.7, 129.5, 129.3, 128.6, 128.2, 127.9, 126.9, 125.4, 124.4, 123.4, 52.1, 23.3, 17.0; IR (KBr, cm^{-1}) 3051, 2993, 2948, 2928, 1726, 1712, 1607, 1575, 1435, 1424, 1397, 1276, 1113, 1101, 1017, 862, 824, 776, 751, 712; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{25}H_{20}NO_2$ 366.1489; found 366.1489.

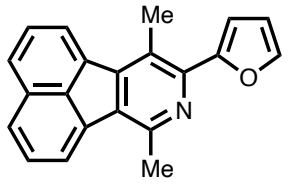
7,10-Dimethyl-9-(pyridin-2-yl)acenaphtho[1,2-*c*]pyridine (3aj)



3aj

Compound **3aj** was prepared according to general procedure using **1a** (99.9 mg, 0.4891 mmol) and **2j** (148.4 mg, 1.425 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (6/4) to afford **3aj** (129.8 mg, 0.4209 mmol, 86% yield) as a yellow solid (m.p. 156.0-157.3 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.77-8.73 (m, 1H), 8.16 (d, *J* = 6.9 Hz, 1H), 8.05 (d, *J* = 6.9 Hz, 1H), 7.96 (d, *J* = 8.1 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.80-7.87 (m, 2H), 7.70 (t, *J* = 7.8 Hz, 2H), 7.32 (ddd, *J* = 6.9, 5.2, 2.0 Hz, 1H), 3.01 (s, 3H), 2.82 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 159.2, 155.6, 150.0, 148.6, 145.9, 136.5, 135.9, 135.4, 132.6, 131.2, 129.9, 128.4, 128.1, 128.0, 126.9, 125.6, 125.4, 124.9, 123.4, 122.4, 23.3, 16.5; IR (KBr, cm^{-1}) 3044, 3002, 2959, 2911, 2852, 1587, 1558, 1460, 1441, 1423, 1405, 1381, 1364, 1032, 820, 783, 767, 745, 698, 687, 608; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{22}H_{17}N_2$ 309.1386; found 309.1391.

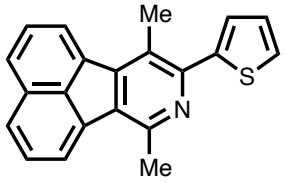
9-(Furan-2-yl)-7,10-dimethylacenaphtho[1,2-*c*]pyridine (3ak)



3ak

Compound **3ak** was prepared according to general procedure using **1a** (97.7 mg, 0.478 mmol) and **2k** (138.42 mg, 1.487 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3ak** (116.2 mg, 0.3908 mmol, 82% yield) as a yellow solid (m.p. 78.5-79.8 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (d, *J* = 6.9 Hz, 1H), 7.98 (d, *J* = 6.9 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.72-7.60 (m, 3H), 6.93 (d, *J* = 3.5 Hz, 1H), 6.61-6.57 (m, 1H), 2.96 (s, 3H), 2.88 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 153.8, 150.3, 146.9, 145.8, 142.9, 135.8, 135.3, 132.5, 130.3, 129.8, 128.5, 128.2, 127.9, 126.8, 125.5, 124.3, 123.2, 111.5, 111.3, 23.5, 16.4; IR (KBr, cm^{-1}) 3114, 3097, 3051, 3011, 2963, 2916, 2855, 1576, 1493, 1448, 1417, 1384, 1365, 1175, 1136, 1033, 1014, 914, 822, 768, 736, 596; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{21}H_{16}NO$ 298.1226; found 298.1226.

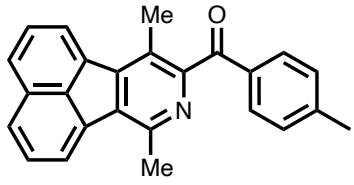
7,10-Dimethyl-9-(thiophen-2-yl)acenaphtho[1,2-*c*]pyridine (3al)



3al

Compound **3al** was prepared according to general procedure using **1a** (99.3 mg, 0.486 mmol) and **2l** (205.0 mg, 1.896 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3al** (136.1 mg, 0.4342 mmol, 89% yield) as a yellow solid (m.p. 91.3-92.5 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (d, *J* = 6.9 Hz, 1H), 7.94 (d, *J* = 7.5 Hz, 1H), 7.92 (d, *J* = 8.6 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.68-7.60 (m, 2H), 7.44 (d, *J* = 5.2 Hz, 1H), 7.41 (d, *J* = 4.0 Hz, 1H), 7.18-7.13 (m, 1H), 2.92 (s, 3H), 2.85 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 150.5, 150.1, 145.7, 144.6, 135.8, 135.3, 132.6, 130.1, 129.8, 128.4, 128.1, 127.8, 127.5, 127.2, 127.0, 126.7, 125.3, 124.0, 123.1, 23.3, 17.1; IR (KBr, cm^{-1}) 3078, 3043, 2987, 2964, 2914, 2859, 1575, 1560, 1436, 1422, 1411, 1363, 1215, 1054, 945, 820, 768, 689, 679; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{21}H_{16}NS$ 314.0998; found 314.0995.

(7,10-Dimethylacenaphtho[1,2-*c*]pyridin-9-yl)(*p*-tolyl)methanone (3am)

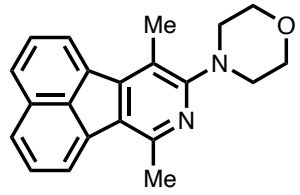


3am

Compound **3am** was prepared according to general procedure using **1a** (102.2 mg, 0.5003 mmol) and **2m** (146.0 mg, 1.006 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3am** (169.8 mg, 0.4859 mmol, 97% yield) as a light brown solid (m.p. 243.7-244.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.16 (d, *J* = 6.9 Hz, 1H), 8.11 (d, *J* = 6.9 Hz, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.95 (d, *J* = 8.1 Hz, 1H), 7.87 (d, *J* = 8.6 Hz, 2H), 7.77-7.70 (m, 2H), 7.29-7.24 (m, 2H), 2.98 (s, 3H), 2.72 (s, 3H), 2.42 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 196.0, 154.9, 150.1, 145.5, 144.6, 135.5, 135.2, 134.3, 132.6, 132.3, 130.9, 130.3, 130.0, 129.3, 129.0, 128.4, 128.2, 127.6, 125.8, 125.2, 124.1, 23.3, 21.9, 15.7; IR (KBr, cm^{-1}) 3054, 3018, 2984, 2952, 2919, 2857, 1662, 1604, 1573, 1561, 1444, 1422, 1374, 1319, 1302, 1238, 1174, 984, 930, 852, 821, 766, 741; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{25}H_{20}NO$ 350.1539; found

350.1540.

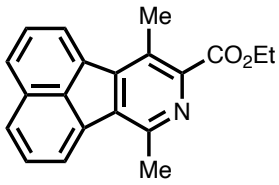
4-(7,10-Dimethylacenaphtho[1,2-*c*]pyridin-9-yl)morpholine (3an)



3an

Compound **3an** was prepared according to general procedure using **1a** (102.6 mg, 0.5023 mmol) and **2n** (114.4 mg, 1.020 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (8/2) to afford **3an** (146.7 mg, 0.4636 mmol, 92% yield) as a light yellow solid (m.p. 216.2-217.1 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04 (d, *J* = 6.9 Hz, 1H), 7.91 (d, *J* = 8.6 Hz, 1H), 7.89 (d, *J* = 6.9 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.67 (t, *J* = 7.5 Hz, 1H), 7.64 (t, *J* = 7.7 Hz, 1H), 3.92 (t, *J* = 4.6 Hz, 4H), 3.26 (t, *J* = 4.6 Hz, 4H), 2.85 (s, 3H), 2.66 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 160.6, 148.3, 146.9, 136.2, 136.1, 133.1, 130.0, 128.2, 127.9, 127.7, 126.9, 125.4, 124.5, 121.7, 117.9, 67.2, 51.0, 23.1, 15.2; IR (KBr, cm^{-1}) 2969, 2954, 2891, 2847, 2822, 2812, 1593, 1572, 1453, 1413, 1394, 1361, 1270, 1262, 1251, 1146, 1131, 1111, 1069, 1045, 1033, 984, 864, 827, 780; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{21}H_{21}N_2O$ 317.1648; found 317.1651.

Ethyl 7,10-dimethylacenaphtho[1,2-*c*]pyridine-9-carboxylate (3ao)

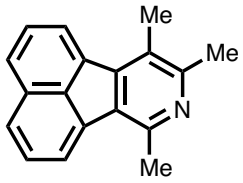


3ao

Compound **3ao** was prepared according to general procedure using **1a** (102.8 mg, 0.5033 mmol) and **2o** (147.2 mg, 1.486 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (8/2) to afford **3ao** (141.8 mg, 0.4674 mmol, 93% yield) as a yellow solid (m.p. 132.0-132.3 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.12 (d, *J* = 6.9 Hz, 1H), 8.03 (d, *J* = 7.5 Hz, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.74-7.64 (m, 2H), 4.54 (q, *J* = 7.3 Hz, 2H), 2.96 (s, 3H), 2.85 (s, 3H), 1.50 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.4, 150.3, 147.7, 145.6, 135.1, 134.6, 133.1, 132.4, 129.8, 128.9, 128.2, 128.0, 127.7, 126.8, 125.8, 124.2, 61.7, 23.4, 15.8, 14.4; IR (KBr, cm^{-1}) 3049, 2980, 2926, 2902, 2865, 1721, 1568, 1442, 1422, 1383, 1314, 1227, 1184, 1140, 1074, 1059, 1020, 822, 770; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}NO_2$ 304.1332;

found 304.1334.

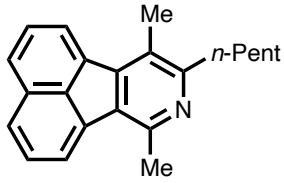
7,9,10-Trimethylacenaphtho[1,2-*c*]pyridine (3ap)



3ap

Compound **3ap** was prepared according to general procedure using **1a** (102.2 mg, 0.5003 mmol) and **2p** (219.6 mg, 5.349 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (7/3) to afford **3ap** (96.6 mg, 0.394 mmol, 79% yield) as a light yellow solid (m.p. 150.2-150.9 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.5 Hz, 1H), 7.93-7.86 (m, 2H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.68-7.59 (m, 2H), 2.86 (s, 3H), 2.63 (s, 3H), 2.60 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 155.6, 149.2, 144.5, 136.0, 135.8, 132.6, 129.9, 129.6, 128.0, 127.7, 126.2, 124.8, 122.5, 23.1, 22.7, 15.3; IR (KBr, cm^{-1}) 3067, 3042, 2986, 2943, 2916, 2862, 1585, 1567, 1439, 1411, 1388, 1186, 1063, 1014, 994, 963, 824, 775, 749; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{18}H_{16}N$ 246.1277; found 246.1278.

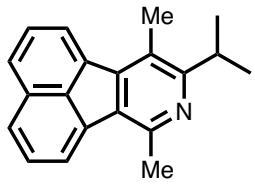
7,10-Dimethyl-9-pentylacenaphtho[1,2-*c*]pyridine (3aq)



3aq

Compound **3aq** was prepared according to general procedure using **1a** (102.5 mg, 0.5018 mmol) and **2q** (254.1 mg, 2.615 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (9/1) to afford **3aq** (121.5 mg, 0.4031 mmol, 80% yield) as a yellow solid (m.p. 111.9-112.1 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.5 Hz, 1H), 7.90 (d, *J* = 7.5 Hz, 1H), 7.88 (d, *J* = 8.6 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.68-7.57 (m, 2H), 2.97-2.90 (m, 2H), 2.88 (s, 3H), 2.66 (s, 3H), 1.80-1.68 (m, 2H), 1.51-1.33 (m, 4H), 0.93 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 159.7, 149.5, 144.8, 136.1, 135.9, 132.5, 129.9, 129.5, 128.05, 128.01 127.7, 126.2, 124.8, 124.2, 122.5, 36.0, 32.1, 29.7, 23.2, 22.7, 15.2, 14.1; IR (KBr, cm^{-1}) 2952, 2932, 2886, 2855, 1593, 1571, 1464, 1428, 1414, 1374, 1343, 1186, 824, 775, 731; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{22}H_{24}N$ 302.1903; found 302.1908.

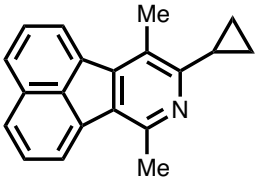
9-Isopropyl-7,10-dimethylacenaphtho[1,2-*c*]pyridine (3ar)



3ar

Compound **3ar** was prepared according to general procedure using **1a** (102.0 mg, 0.4993 mmol) and **2r** (115.3 mg, 1.668 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (95/5) to afford **3ar** (93.4 mg, 0.3417 mmol, 68% yield) as a light yellow solid (m.p. 170.2-171.6 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (d, *J* = 6.9 Hz, 1H), 7.93 (d, *J* = 6.9 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.69-7.59 (m, 2H), 3.50 (septet, *J* = 6.8 Hz, 1H), 2.91 (s, 3H), 2.72 (s, 3H), 1.37 (d, *J* = 6.9 Hz, 6H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 163.5, 149.5, 144.7, 136.5, 136.1, 132.6, 129.9, 129.1, 128.0, 127.9, 127.7, 126.1, 124.8, 123.5, 122.5, 31.1, 23.5, 22.1, 14.8; IR (KBr, cm^{-1}) 3049, 2979, 2964, 2928, 2867, 1580, 1562, 1441, 1414, 1381, 1356, 1330, 1187, 1141, 1050, 1030, 822, 773, 759, 622; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{20}H_{20}N$ 274.1590; found 274.1591.

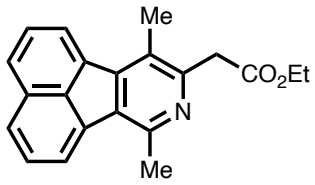
9-Cyclopropyl-7,10-dimethylacenaphtho[1,2-*c*]pyridine (3as)



3as

Compound **3as** was prepared according to general procedure using **1a** (101.9 mg, 0.4988 mmol) and **2s** (100.6 mg, 1.500 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (95/5) to afford **3as** (114.0 mg, 0.4201 mmol, 84% yield) as a yellow solid (m.p. 175.9-176.3 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.5 Hz, 1H), 7.92-7.86 (m, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.68-7.59 (m, 2H), 2.83 (s, 3H), 2.79 (s, 3H), 2.31-2.23 (m, 1H), 1.18-1.13 (m, 2H), 1.03-0.97 (m, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 158.8, 149.4, 144.2, 136.3, 136.1, 132.7, 129.9, 128.9, 128.0, 127.9, 127.7, 126.0, 124.8, 124.7, 122.3, 23.3, 14.7, 14.1, 8.5; IR (KBr, cm^{-1}) 3087, 3050, 3005, 2939, 2914, 2862, 1584, 1564, 1467, 1437, 1420, 1395, 1379, 1369, 1207, 1188, 1066, 1057, 1020, 972, 908, 859, 824, 776, 752; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{20}H_{18}N$ 272.1434; found 272.1435.

Ethyl 2-(7,10-dimethylacenaphtho[1,2-*c*]pyridin-9-yl)acetate (3at)

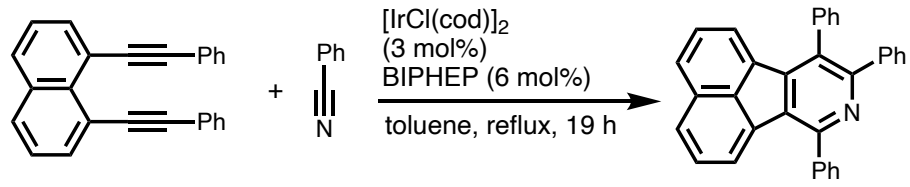


3at

Compound **3at** was prepared according to general procedure using **1a** (102.9 mg, 0.5037 mmol) and **2t** (175.5 mg, 1.552 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3at** (143.8 mg, 0.4531 mmol, 90% yield) as a red solid (m.p. 140.9-141.8 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (d, *J* = 6.9 Hz, 1H), 7.95 (d, *J* = 6.9 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 1H), 7.70-7.62 (m, 2H), 4.22 (q, *J* = 7.1 Hz, 2H), 4.04 (s, 2H), 2.90 (s, 3H), 2.66 (s, 3H), 1.28 (t, *J* = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 171.0, 151.7, 149.8, 145.1, 135.7, 135.5, 132.5, 130.8, 129.9, 128.3, 128.1, 127.8, 126.6, 125.6, 125.1, 123.0, 60.9, 42.2, 23.1, 15.5, 14.2; IR (KBr, cm^{-1}) 2984, 2974, 2952, 2927, 2901, 2858, 1735, 1589, 1576, 1450, 1432, 1415, 1366, 1312, 1263, 1151, 1038, 950, 827, 804, 781, 687, 581; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{21}H_{20}NO_2$ 318.1489; found 318.1488.

6. General Procedure for the [2+2+2] Cycloaddition of Diyne 1b with Nitriles 2 (Scheme 3)

Representative Procedure for the Reaction of Diyne 1b with Nitrile 2a

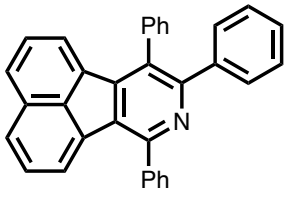


1b **2a** (3 equiv) **3ba**

To the solution of $[IrCl(cod)]_2$ (10.8 mg, 0.0161 mmol), BIPHEP (15.5 mg, 0.0297 mmol), and toluene (3.0 mL) was added **2a** (156.0 mg, 1.513 mmol) and **1b** (165.3 mg, 0.5033 mmol). The mixture was stirred under reflux for 19 h. After the solvent was removed on a rotary evaporator, the residue was subjected to column chromatography (silica gel, toluene) to give **3ba** (157.8 mg, 0.3657 mmol, 73% yield).

7. Characterization of 3ba-3by

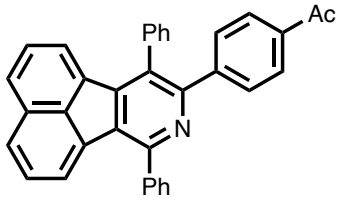
7,9,10-Triphenylacenaphtho[1,2-*c*]pyridine (3ba)



3ba

Compound **3ba** was prepared according to general procedure using **1b** (165.3 mg, 0.5033 mmol) and **2a** (156.0 mg, 1.513 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3ba** (157.8 mg, 0.3657 mmol, 73% yield) as a pale yellow solid (m.p. 206.2-206.8 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, J = 6.9 Hz, 2H), 7.87 (d, J = 8.1 Hz, 1H), 7.82 (d, J = 8.0 Hz, 1H), 7.65-7.51 (m, 4H), 7.51-7.34 (m, 9H), 7.23-7.17 (m, 3H), 6.90 (d, J = 6.9 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 156.0, 153.8, 146.3, 140.5, 140.3, 138.0, 134.8, 134.4, 133.0, 130.6, 130.2, 130.1, 129.9, 129.2, 128.83, 128.75, 128.5, 127.9, 127.8, 127.7, 127.5, 127.4, 127.2, 125.1, 123.5; IR (KBr, cm^{-1}) 3055, 3027, 2960, 2925, 2854, 1550, 1494, 1426, 1402, 1071, 1021, 985, 825, 775, 755, 702, 546; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{33}H_{22}N$ 432.1747; found 432.1736.

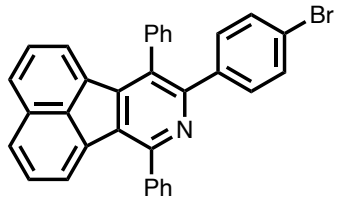
1-(4-(7,10-Diphenylacenaphtho[1,2-*c*]pyridin-9-yl)phenyl)ethan-1-one (3be)



3be

Compound **3be** was prepared according to general procedure using **1b** (167.8 mg, 0.5109 mmol) and **2e** (218.6 mg, 1.506 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3be** (239.0 mg, 0.5047 mmol, 99% yield) as a yellow solid (m.p. 274.3-274.6 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96-7.92 (m, 2H), 7.89 (d, *J* = 8.1 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.81-7.77 (m, 2H), 7.66-7.53 (m, 6H), 7.51-7.45 (m, 4H), 7.45-7.36 (m, 3H), 6.92 (d, *J* = 6.9 Hz, 1H), 2.55 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 198.0, 154.6, 154.0, 146.4, 145.1, 140.3, 137.5, 135.7, 134.5, 134.1, 133.0, 130.8, 130.6, 130.3, 130.1, 129.9, 129.2, 129.01, 128.97, 128.92, 128.6, 128.1, 127.9, 127.8, 127.6, 127.5, 125.3, 123.8, 26.6; IR (KBr, cm^{-1}) 3057, 3033, 1685, 1604, 1562, 1550, 1424, 1394, 1354, 1265, 1017, 985, 956, 849, 829, 778, 768, 721, 700; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{35}H_{24}NO$ 474.1852; found 474.1851.

9-(4-Bromophenyl)-7,10-diphenylacenaphtho[1,2-*c*]pyridine (3bg)

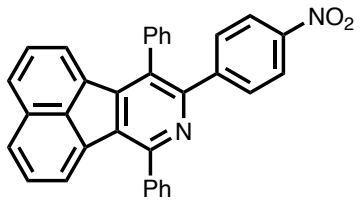


3bg

Compound **3bg** was prepared according to general procedure using **1b** (164.2 mg, 0.5000 mmol) and **2g** (261.4 mg, 1.436 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3bg** (203.5 mg, 0.3987 mmol, 80% yield) as a dark yellow solid (m.p. 249.9-252.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.1 Hz, 2H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.66-7.52 (m, 4H), 7.52-7.44 (m, 4H), 7.44-7.29 (m, 7H), 6.89 (d, *J* = 7.5 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 154.6, 153.9, 146.5, 140.3, 139.2, 137.7, 134.6, 134.2, 133.0, 131.8, 130.7, 130.5, 130.3, 130.1, 129.9, 129.2, 129.1, 128.9, 128.6, 128.1, 127.9, 127.7, 127.4, 125.2, 123.7, 121.9; IR (KBr, cm^{-1}) 3055, 3033, 1580, 1561, 1548, 1485, 1423, 1387, 1349, 1076, 1010, 984, 841,

829, 817, 774, 738, 701, 544; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{33}H_{21}BrN$ 510.0852; found 510.0852.

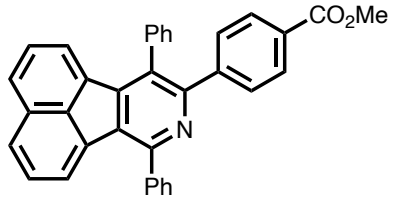
9-(4-Nitrophenyl)-7,10-diphenylacenaphtho[1,2-*c*]pyridine (3bh)



3bh

Compound **3bh** was prepared according to general procedure using **1b** (164.1 mg, 0.4997 mmol) and **2h** (227.0 mg, 1.533 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3bh** (205.4 mg, 0.4310 mmol, 86% yield) as a yellow solid (m.p. 136.3-136.4 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09-8.03 (m, 2H), 7.97-7.89 (m, 3H), 7.87 (d, *J* = 8.6 Hz, 1H), 7.70-7.55 (m, 6H), 7.54-7.48 (m, 4H), 7.45-7.38 (m, 3H), 6.95 (d, *J* = 6.9 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 154.2, 153.2, 146.9, 146.8, 146.6, 140.0, 137.1, 134.3, 133.9, 133.0, 131.04, 131.01, 130.9, 130.0, 129.9, 129.2, 129.1, 128.7, 128.4, 128.0, 127.83, 127.81, 125.4, 124.1, 122.8; IR (KBr, cm^{-1}) 3057, 3029, 1598, 1552, 1517, 1422, 1345, 862, 843, 828, 776, 701; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{33}H_{21}N_2O_2$ 477.1598; found 477.1600.

Methyl 4-(7,10-diphenylacenaphtho[1,2-*c*]pyridin-9-yl)benzoate (3bi)

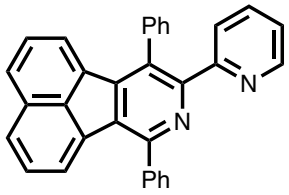


3bi

Compound **3bi** was prepared according to general procedure using **1b** (164.1 mg, 0.4997 mmol) and **2i** (242.3 mg, 1.503 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3bi** (224.8 mg, 0.4592 mmol, 92% yield) as a light yellow solid (m.p. 203.9-204.2 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96-7.92 (m, 2H), 7.88 (d, *J* = 8.6 Hz, 3H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.65-7.58 (m, 3H), 7.58-7.51 (m, 3H), 7.51-7.43 (m, 4H), 7.43-7.35 (m, 3H), 6.92 (d, *J* = 6.9 Hz, 1H), 3.87 (s, 3H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 167.1, 154.7, 154.0, 146.4, 144.9, 140.3, 137.5, 134.6, 134.1, 133.0, 130.8, 130.5, 130.15, 130.09, 129.9, 129.2, 129.0, 128.95, 128.91, 128.85, 128.75,

128.6, 128.1, 127.9, 127.8, 127.5, 125.3, 123.8, 52.0; IR (KBr, cm^{-1}) 3057, 3027, 2991, 2945, 2852, 1717, 1608, 1562, 1547, 1427, 1395, 1277, 1106, 1019, 863, 828, 779, 767, 701; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{35}H_{24}NO_2$ 490.1802; found 490.1800.

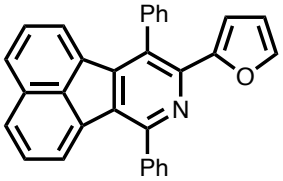
7-Methyl-10-phenyl-9-(pyridin-2-yl)acenaphtho[1,2-*c*]pyridine (3bj)



3bj

Compound **3bj** was prepared according to general procedure using **1b** (163.3 mg, 0.4972 mmol) and **2j** (157.3 mg, 1.511 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (8/2) to afford **3bj** (198.7 mg, 0.4594 mmol, 92% yield) as a yellow solid (m.p. 213.9-214.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.48 (d, *J* = 4.6 Hz, 1H), 7.95-7.90 (m, 2H), 7.88 (d, *J* = 8.1 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.61-7.35 (m, 13H), 7.12-7.06 (m, 1H), 6.97 (d, *J* = 7.4 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 158.3, 155.0, 153.8, 148.8, 146.3, 140.3, 137.6, 135.5, 134.6, 134.3, 133.0, 131.1, 130.9, 130.0, 129.8, 129.2, 128.9, 128.7, 128.6, 128.4, 127.9, 127.7, 127.6, 127.4, 125.3, 125.0, 123.8, 122.0; IR (KBr, cm^{-1}) 3052, 3025, 1585, 1549, 1476, 1424, 1402, 1169, 1146, 1019, 994, 826, 775, 767, 737, 703, 549; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{32}H_{21}N_2$ 433.1699; found 433.1699.

9-(Furan-2-yl)-7,10-diphenylacenaphtho[1,2-*c*]pyridine (3bk)

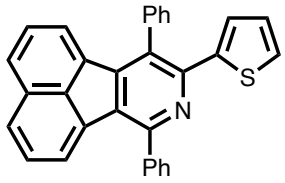


3bk

Compound **3bk** was prepared according to general procedure using **1b** (166.1 mg, 0.5058 mmol) and **2k** (143.1 mg, 1.537 mmol). The crude reaction mixture was purified by column chromatography using toluene to afford **3bk** (178.6 mg, 0.4237 mmol, 84% yield) as a light brown solid (m.p. 249.5-251.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95-7.89 (m, 2H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.6 Hz, 1H), 7.70-7.42 (m, 10H), 7.37 (t, *J* = 7.7 Hz, 1H), 6.66 (d, *J* = 7.5 Hz, 1H), 6.30-6.24 (m, 1H), 5.81 (d, *J* = 3.4 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 154.0, 152.6, 146.5, 145.5, 143.1, 140.4, 138.2, 134.6, 134.3,

133.0, 129.8, 129.6, 129.4, 129.3, 129.2, 128.9, 128.80, 128.76, 128.6, 128.4, 128.0, 127.7, 127.1, 125.3, 123.6, 112.6, 111.3; IR (KBr, cm^{-1}) 3129, 3104, 3078, 3054, 3030, 2960, 2925, 2858, 1763, 1719, 1600, 1566, 1484, 1425, 1389, 1344, 1226, 1189, 1175, 1018, 987, 827, 765, 725, 699, 549; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{31}H_{20}NO$ 422.1539; found 422.1540.

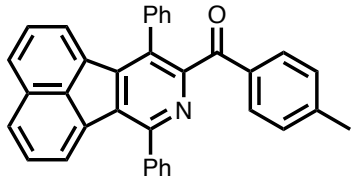
7,10-Diphenyl-9-(thiophen-2-yl)acenaphtho[1,2-*c*]pyridine (3bl)



3bl

Compound **3bl** was prepared according to general procedure using **1b** (162.8 mg, 0.4957 mmol) and **2l** (157.6 mg, 1.444 mmol). The crude reaction mixture was purified by column chromatography using hexane/DCM/EtOAc (97/2/1) to afford **3bl** (213.0 mg, 0.4868 mmol, 98% yield) as a yellow solid (m.p. 192.6-193.5 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02-7.95 (m, 2H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.69 (d, *J* = 6.9 Hz, 1H), 7.67-7.49 (m, 8H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.34 (t, *J* = 7.7 Hz, 1H), 7.26-7.21 (m, 1H), 6.81 (t, *J* = 4.3 Hz, 1H), 6.63 (d, *J* = 6.9 Hz, 1H), 6.57 (d, *J* = 3.5 Hz, 1H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 153.4, 148.6, 146.9, 145.5, 140.2, 138.1, 134.7, 134.3, 133.0, 129.83, 129.80, 129.6, 129.3, 129.2, 128.9, 128.7, 128.6, 128.45, 128.39, 127.9, 127.8, 127.7, 127.5, 127.1, 125.2, 123.5; IR (KBr, cm^{-1}) 3054, 2961, 2924, 1563, 1552, 1431, 1409, 1372, 1092, 1072, 1059, 1042, 1025, 826, 775, 768, 699, 549; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{31}H_{20}NS$ 438.1311; found 438.1309.

(7,10-Diphenylacenaphtho[1,2-*c*]pyridin-9-yl)(*p*-tolyl)methanone (3bm)

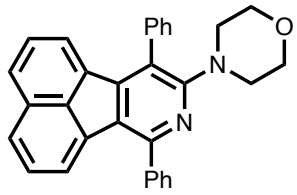


3bm

Compound **3bm** was prepared according to general procedure using **1b** (165.9 mg, 0.5052 mmol) and **2m** (218.8 mg, 1.507 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (75/25) followed by GPC to afford **3bm** (175.4 mg, 0.3703 mmol, 73% yield) as a light brown solid (m.p. 201.5-202.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91-7.84 (m, 4H), 7.79 (d, *J* = 8.6 Hz, 2H), 7.67 (d, *J* = 6.9 Hz, 1H),

7.60-7.37 (m, 10H), 7.18 (d, J = 8.0 Hz, 2H), 7.02 (d, J = 6.9 Hz, 1H), 2.36 (s, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 194.7, 154.4, 153.1, 146.1, 144.0, 139.7, 135.7, 134.4, 134.1, 134.0, 132.9, 131.7, 130.61, 130.57, 129.9, 129.5, 129.22, 129.18, 129.0, 128.9, 128.7, 128.6, 128.3, 128.0,. 127.91, 127.88, 125.5, 124.3, 21.7; IR (KBr, cm^{-1}) 3051, 3029, 1672, 1604, 1548, 1425, 1403, 1329, 1248, 1225, 1173, 1021, 954, 828; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{35}H_{24}NO$ 474.1852; found 474.1851.

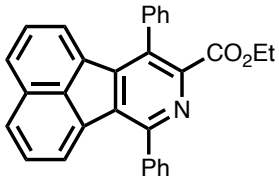
4-(7,10-Diphenylacenaphtho[1,2-*c*]pyridin-9-yl)morpholine (3bn)



3bn

Compound **3bn** was prepared according to general procedure using **1b** (165.8 mg, 0.5049 mmol) and **2n** (170.3 mg, 1.519 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (9/1) to afford **3bn** (216.5 mg, 0.4914 mmol, 97% yield) as a yellow solid (m.p. 195.6-196.0 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 (d, J = 6.9 Hz, 2H), 7.80 (d, J = 8.0 Hz, 1H), 7.70 (d, J = 8.0 Hz, 1H), 7.64-7.48 (m, 9H), 7.40 (t, J = 7.5 Hz, 1H), 7.33 (t, J = 7.8 Hz, 1H), 6.93 (d, J = 6.9 Hz, 1H), 3.55 (t, J = 4.6 Hz, 4H), 3.19 (t, J = 4.6 Hz, 4H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 158.6, 151.5, 147.5, 140.7, 137.5, 135.1, 134.9, 133.6, 130.0, 129.2, 129.1, 128.6, 128.4, 128.2, 128.0, 127.8, 127.4, 125.7, 125.4, 124.3, 122.8, 121.5, 66.9, 49.8; IR (KBr, cm^{-1}) 3049, 2953, 2907, 2887, 2852, 1584, 1569, 1409, 1389, 1362, 1270, 1240, 1116, 958, 826, 776, 703; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{31}H_{25}N_2O$ 441.1961; found 441.1960.

Ethyl 7,10-diphenylacenaphtho[1,2-*c*]pyridine-9-carboxylate (3bo)

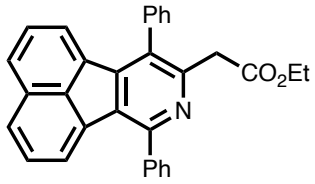


3bo

Compound **3bo** was prepared according to general procedure using **1b** (164.2 mg, 0.500 mmol) and **2o** (150.3 mg, 1.517 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc/CH_2Cl_2 (8/1/1) to afford **3bo** (195.9 mg, 0.4582 mmol, 92% yield) as a light yellow solid (m.p. 211.8-212.6 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93-7.83 (m, 4H), 7.65-7.51 (m, 9H), 7.48 (t, J = 7.8 Hz, 1H), 7.41 (t, J = 7.7 Hz,

1H), 6.96 (d, J = 6.9 Hz, 1H), 4.15 (q, J = 7.3 Hz, 2H), 1.03 (t, J = 7.2 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 166.9, 153.8, 148.1, 146.2, 139.6, 136.6, 133.9, 133.7, 132.8, 132.5, 131.0, 129.8, 129.3, 129.2, 129.1, 128.7, 128.6, 128.3, 128.1, 128.0, 127.9, 125.7, 124.6, 61.3, 13.7; IR (KBr, cm^{-1}) 3055, 3026, 2995, 2975, 2954, 2932, 1719, 1555, 1424, 1414, 1375, 1328, 1246, 1221, 1198, 1176, 1108, 1092, 1020, 824, 774, 723, 711, 702; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{30}H_{22}NO_2$ 428.1645; found 428.1644.

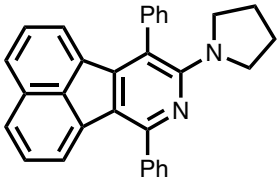
Ethyl 2-(7,10-diphenylacenaphtho[1,2-*c*]pyridin-9-yl)acetate (3bt)



3bt

Compound **3bt** was prepared according to general procedure using **1b** (163.5 mg, 0.4978 mmol) and **2t** (286.5 mg, 2.778 mmol). The crude reaction mixture was purified by column chromatography using toluene/EtOAc (8/2) to afford **3bt** (187.0 mg, 0.4235 mmol, 85% yield) as a white solid (m.p. 129.9-130.6 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90-7.82 (m, 3H), 7.80 (d, J = 8.0 Hz, 1H), 7.62-7.51 (m, 7H), 7.51-7.47 (m, 2H), 7.44 (t, J = 7.5 Hz, 1H), 7.36 (t, J = 7.7 Hz, 1H), 6,73 (d, J = 6.9 Hz, 1H), 4.09 (q, J = 7.3 Hz, 2H), 3.87 (s, 2H), 1.19 (t, J = 7.2 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 171.0, 153.6, 151.4, 146.0, 140.3, 137.3, 134.5, 134.4, 132.8, 131.7, 130.2, 129.8, 129.3, 129.2, 129.1, 128.73, 128.70, 128.6, 128.3, 127.9, 127.7, 127.2, 124.9, 123.5, 60.7, 41.9, 14.2; IR (KBr, cm^{-1}) 3058, 2982, 2940, 2925, 1736, 1553, 1423, 1367, 1318, 1184, 1165, 1025, 828, 777, 703, 664, 528; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{31}H_{24}NO_2$ 442.1802; found 442.1803.

7,10-Diphenyl-9-(pyrrolidin-1-yl)acenaphtho[1,2-*c*]pyridine (3bu)

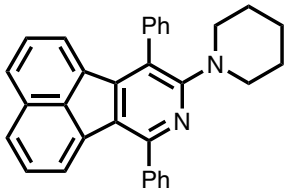


3bu

Compound **3bu** was prepared according to general procedure using **1b** (81.0 mg, 0.2466 mmol) and **2u** (75.0 mg, 0.780 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (98/2) to afford **3bu** (99.3 mg, 0.234 mmol, 95% yield) as a orange solid (m.p. 176.1-176.5 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, J = 7.5 Hz, 2H), 7.72 (d, J = 8.0 Hz, 1H), 7.60 (d, J = 8.1 Hz, 1H), 7.58-7.46 (m, 8H), 7.42

(d, *J* = 6.9 Hz, 1H), 7.34 (t, *J* = 7.7 Hz, 1H), 7.24 (t, *J* = 7.7 Hz, 1H), 6.48 (d, *J* = 7.5 Hz, 1H), 3.33-3.25 (m, 4H), 1.78-1.70 (m, 4H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 155.2, 151.6, 147.9, 141.3, 139.1, 135.8, 135.7, 133.6, 130.9, 130.0, 129.1, 128.6, 128.4, 128.2, 127.8, 127.7, 127.2, 124.3, 123.8, 120.6, 120.2, 117.8, 50.1, 25.6; IR (KBr, cm^{-1}) 3050, 3032, 2958, 2868, 1583, 1569, 1550, 1469, 1437, 1343, 1258, 1091, 1019, 768, 703; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{31}H_{25}N_2$ 425.2012; found 452.2019.

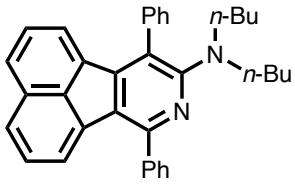
7,10-Diphenyl-9-(piperidin-1-yl)acenaphtho[1,2-*c*]pyridine (3bv)



3bv

Compound **3bv** was prepared according to general procedure using **1b** (164.2 mg, 0.5000 mmol) and **2v** (162.9 mg, 1.479 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (99/1) to afford **3bv** (213.7 mg, 0.4873 mmol, 97% yield) as a yellow solid (m.p. 184.0-184.3 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94-7.88 (m, 2H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.63-7.46 (m, 9H), 7.38 (t, *J* = 7.8 Hz, 1H), 7.31 (t, *J* = 7.7 Hz, 1H), 6.91 (d, *J* = 6.9 Hz, 1H), 3.24-3.10 (m, 4H), 1.52-1.42 (m, 2H), 1.42-1.32 (m, 4H); $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 160.0, 151.5, 147.2, 140.9, 138.0, 135.4, 135.1, 133.6, 130.1, 130.0, 129.2, 129.0, 128.5, 128.3, 128.0, 127.7, 127.6, 127.3, 125.4, 124.7, 124.0, 123.2, 121.2, 50.6, 25.8, 24.6; IR (KBr, cm^{-1}) 3048, 2975, 2931, 2912, 2814, 1585, 1572, 1556, 1410, 1370, 1239, 1126, 1090, 1030, 1018, 824, 812, 787, 776, 766, 707, 700, 666; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{32}H_{27}N_2$ 439.2169; found 439.2174.

***N*,*N*-Dibutyl-7,10-diphenylacenaphtho[1,2-*c*]pyridin-9-amine (3bw)**

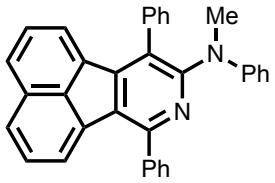


3bw

Compound **3bw** was prepared according to general procedure using **1b** (163.5 mg, 0.4978 mmol) and **2w** (229.1 mg, 1.485 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (99/1) to afford **3bw** (230.7 mg, 0.4780 mmol, 96% yield) as a brown-yellow solid (m.p. 142.6-142.9 °C). ^{1}H NMR (500 MHz,

$CDCl_3$) δ 7.94-7.89 (m, 2H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.66 (d, *J* = 8.1 Hz, 1H), 7.62-7.48 (m, 1H), 7.41-7.36 (m, 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 6.65 (d, *J* = 6.9 Hz, 1H), 3.16 (t, *J* = 7.5 Hz, 4H), 1.41-1.31 (m, 4H), 1.13 (sextet, *J* = 7.5 Hz, 4H), 0.81 (t, *J* = 7.5 Hz, 6H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 158.5, 151.2, 147.8, 141.1, 138.6, 135.6, 135.3, 133.5, 130.2, 130.0, 129.2, 129.1, 128.4, 128.2, 127.8, 127.7, 127.6, 127.3, 125.1, 124.0, 123.2, 122.5, 120.8, 51.4, 30.5, 20.3, 14.0; IR (KBr, cm^{-1}) 3057, 3033, 2953, 2927, 2867, 2828, 1583, 1569, 1554, 1464, 1449, 1413, 1362, 1344, 1299, 1223, 1125, 1091, 823, 770, 732, 715, 699, 666; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{35}H_{35}N_2$ 483.2795; found 483.2802.

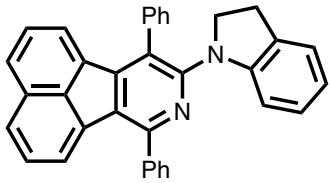
<u>*N*-Methyl-*N*,7,10-triphenylacenaphtho[1,2-*c*]pyridin-9-amine (3bx)</u>



3bx

Compound **3bx** was prepared according to general procedure using **1b** (163.3 mg, 0.4972 mmol) and **2x** (191.6 mg, 1.450 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (20/1) to afford **3bx** (216.8 mg, 0.4707 mmol, 95% yield) as a yellow solid (m.p. 207.5-208.1 °C). ^1H NMR (500 MHz, $CDCl_3$) δ 7.97-7.89 (m, 2H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.65-7.49 (m, 4H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.34-7.26 (m, 4H), 7.26-7.19 (m, 2H), 7.08-6.99 (m, 2H), 6.87 (d, *J* = 6.9 Hz, 1H), 6.78 (t, *J* = 7.5 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 2H), 3.29 (s, 3H); $^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$) δ 157.4, 152.6, 149.5, 148.2, 140.4, 136.6, 134.8, 134.6, 133.4, 130.0, 129.8, 129.2, 128.7, 128.51, 128.47, 128.43, 128.3, 127.8, 127.5, 127.3, 127.2, 126.7, 126.2, 124.5, 122.2, 120.8, 120.4, 40.5; IR (KBr, cm^{-1}) 3079, 3059, 3025, 2998, 2961, 2924, 2896, 1583, 1571, 1554, 1491, 1424, 1397, 1379, 1352, 1114, 1020, 778, 763, 695; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{34}H_{25}N_2$ 461.2012; found 461.2020.

<u>9-(Indolin-1-yl)-7,10-diphenylacenaphtho[1,2-*c*]pyridine (3by)</u>

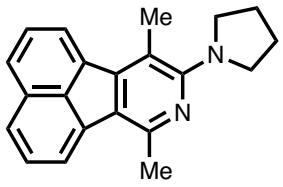


3by

Compound **3by** was prepared according to general procedure using **1b** (165.9 mg, 0.5052 mmol) and **2y** (213.8 mg, 1.483 mmol). The crude reaction mixture was purified by

column chromatography using hexane/EtOAc (9/1) to afford **3by** (229.3 mg, 0.4852 mmol, 96% yield) as a orange solid (m.p. 236.5-237.6 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95-7.89 (m, 2H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.63-7.48 (m, 10H), 7.43 (t, *J* = 8.8 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 1H), 7.09 (dd, *J* = 7.7 Hz, 1H), 7.05 (d, *J* = 7.5 Hz, 1H), 6.90 (d, *J* = 7.4 Hz, 1H), 6.80-6.70 (m, 1H), 3.43 (t, *J* = 8.6 Hz, 2H), 3.92 (t, *J* = 8.6 Hz, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 153.7, 152.1, 148.3, 147.4, 140.2, 137.2, 135.0, 134.7, 133.3, 130.7, 130.3, 130.0, 129.2, 129.0, 128.7, 128.5, 128.4, 128.1, 127.8, 127.5, 126.6, 126.1, 126.0, 124.6, 124.3, 123.6, 122.0, 119.7, 113.6, 52.6, 29.1; IR (KBr, cm^{-1}) 3054, 3033, 2952, 2892, 2877, 2844, 1560, 1582, 1564, 1582, 1564, 1478, 1460, 1407, 1390, 1363, 1247, 1021, 823, 770, 752, 701, 543; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{35}H_{25}N_2$ 473.2012; found 473.2019.

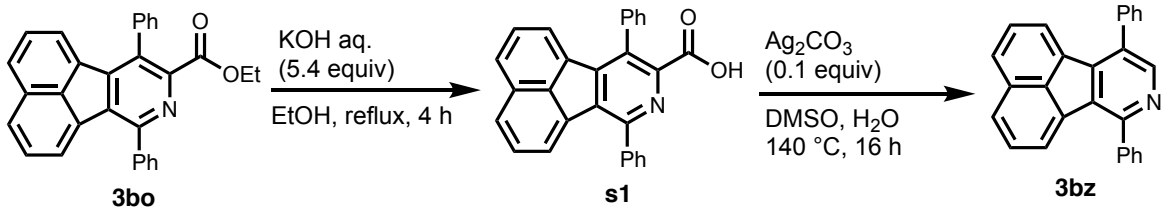
7,10-Dimethyl-9-(pyrrolidin-1-yl)acenaphtho[1,2-*c*]pyridine (3au)



3au

Compound **3au** was prepared according to general procedure using **1a** (102.9 mg, 0.5037 mmol) and **2u** (145.6 mg, 1.515 mmol). The crude reaction mixture was purified by column chromatography using hexane/EtOAc (98/2) to afford **3au** (133.7 mg, 0.4451 mmol, 88% yield) as a yellow solid (m.p. 143.7-144.1 °C). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 6.9 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.80 (d, *J* = 6.9 Hz, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.67-7.58 (m, 2H), 3.64-3.59 (m, 4H), 2.80 (s, 3H), 2.66 (s, 3H), 1.98-1.92 (m, 4H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 159.6, 147.6, 146.9, 136.72, 136.67, 133.4, 130.1, 128.2, 127.53, 127.51, 124.3, 124.1, 123.8, 120.4, 114.2, 50.6, 25.7, 23.1, 16.5; IR (KBr, cm^{-1}) 3054, 2964, 2924, 2867, 1596, 1565, 1460, 1419, 1343, 1252, 1186, 823, 771; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{21}H_{21}N_2$ 301.1699; found 301.1704.

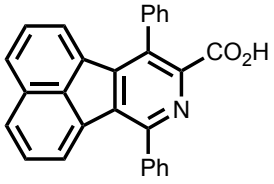
8. Procedure for the synthesis of azafluoranthene 3bz (Scheme 4)[7]



To a solution of KOH aq. (5.04 mL, 5.393 mmol, 1.07 M) and EtOH (5.04 mL) was added azafluoranthene **3bo** (427.5 mg, 0.9999 mmol) at room temperature. After the mixture was stirred under reflux for 4 h, the solution was concentrated on a rotary evaporator. HCl aq. was added, and the resulting solid was filtered and dried in vacuo to give **s1** (380.1 mg, 0.9516 mmol, 95% yield) as a light yellow solid (m.p. 206.9-207.9 °C).

A mixture of **s1** (199.7 mg, 0.4999 mmol) and Ag_2CO_3 (14 mg, 0.051 mmol) in H_2O (452 μL) and DMSO (2.5 mL) was stirred at 140 °C for 16 h. sat. $NaHCO_3$ aq. was added, and the mixture was extracted with Et_2O. The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography (Hexane/EtOAc = 4/1) to give **3bz** (166.0 mg, 0.4670 mmol, 93% yield) as a light yellow solid (m.p. 153.3-154.0 °C).

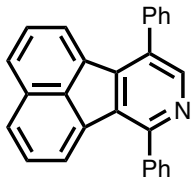
7,10-Diphenylacenaphtho[1,2-*c*]pyridine-9-carboxylic acid (s1)



s1

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 7.5, 1.1 Hz, 2H), 7.85-7.80 (m, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.38-7.32 (m, 4H), 7.22-7.16 (m, 2H), 7.11 (t, *J* = 7.5 Hz, 4H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 163.2, 152.1, 148.8, 140.6, 138.5, 136.6, 135.0, 134.0, 133.4, 133.0, 132.7, 129.8, 129.7, 129.6, 129.1, 129.0, 128.84, 128.81, 128.2, 128.1, 128.0, 126.9, 125.3; IR (KBr, cm^{-1}) 3411, 3058, 3027, 1763, 1560, 1469, 1423, 1378, 1359, 1336, 1308, 1144, 1019, 997, 827, 773, 741, 701, 552; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{28}H_{18}NO_2$ 400.1332; found 400.1333.

7,10-Diphenylacenaphtho[1,2-*c*]pyridine (3bz)



3bz

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (s, 1H), 7.91 (d, J = 8.0 Hz, 1H), 7.88-7.82 (m, 3H), 7.72-7.67 (m, 2H), 7.64-7.50 (m, 8H), 7.50-7.42 (m, 2H); ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 154.0, 148.8, 144.2, 140.3, 137.5, 134.6, 134.2, 132.6, 132.2, 131.1, 129.9, 129.2, 129.03, 128.96, 128.87, 128.81, 128.6, 128.4, 127.9, 127.7, 127.4, 125.0, 123.7; IR (KBr, cm^{-1}) 3046, 3025, 1578, 1558, 1461, 1431, 1311, 1176, 1070, 1019, 977, 911, 824, 772, 703, 524; HRMS (FAB) *m/z* $[M+H]^+$ calcd for $C_{27}H_{18}N$ 356.1434; found 356.1433.

9. UV/Vis and Fluorescence Spectra

Absorption spectra were obtained with a JASCO V-650. Fluorescence spectra were measured on a Shimadzu RF-6000. 9,10-Diphenylanthracene ($\Phi_F = 0.97$)[8] and fluorescein ($\Phi_F = 0.80$)[9] were used as standards for the determination of quantum yields.

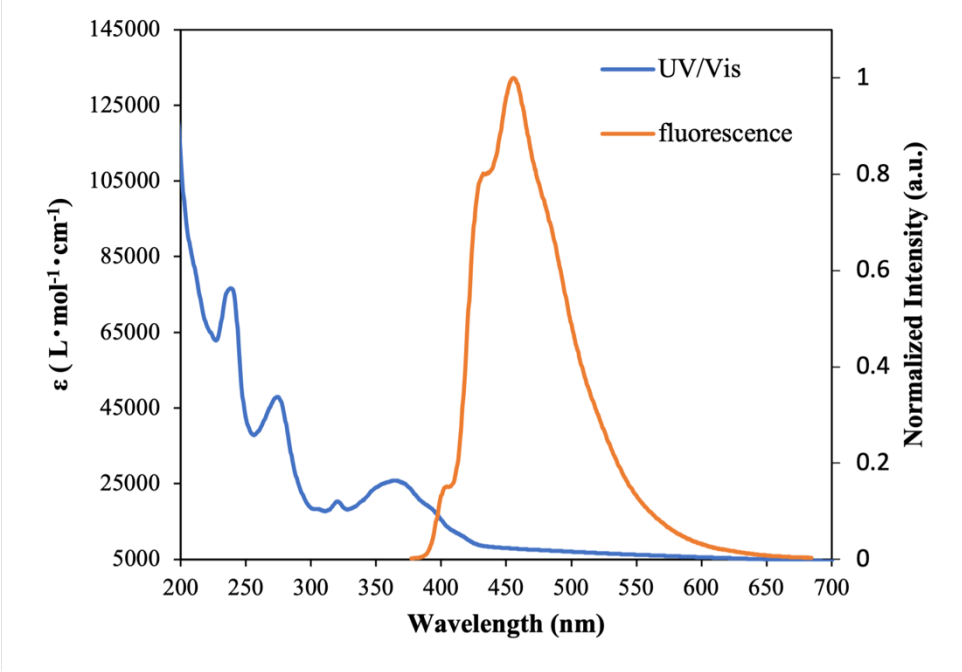


Figure S1. UV-Vis (blue) and fluorescence (orange, excitation at 365 nm) spectra of **3aa** in cyclohexane at 25 °C.

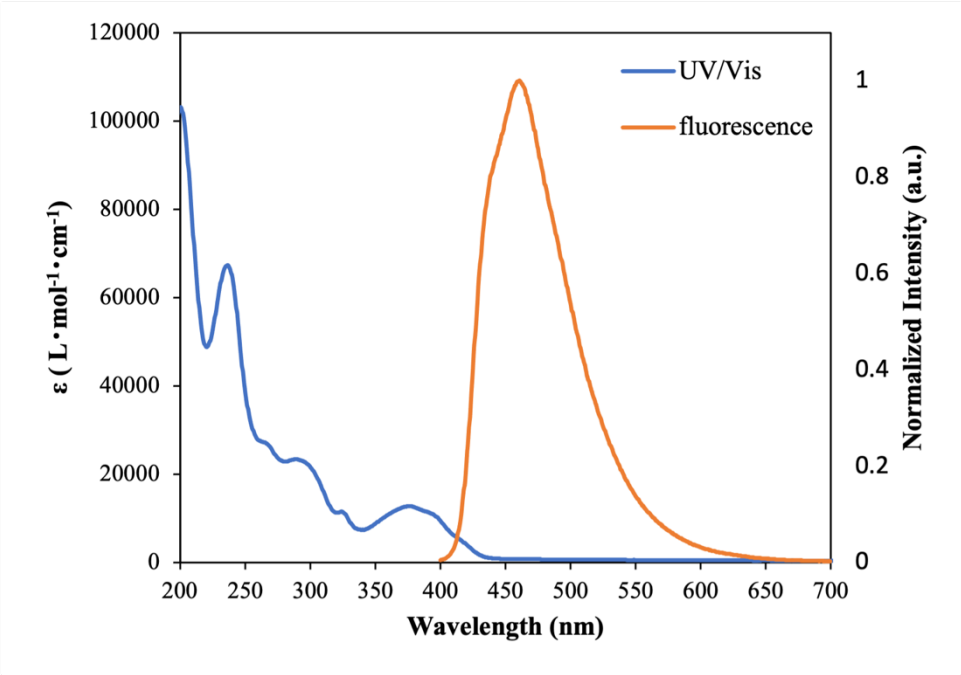


Figure S2. UV-Vis (blue) and fluorescence (orange, excitation at 376 nm) spectra of **3ba** in cyclohexane at 25 °C.

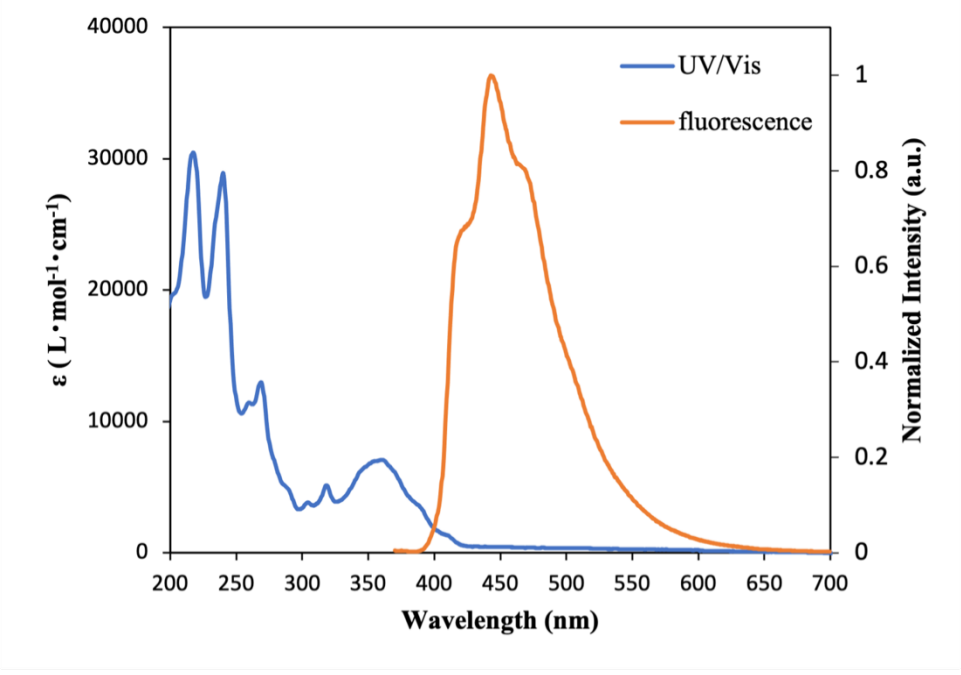


Figure S3. UV-Vis (blue) and fluorescence (orange, excitation at 360 nm) spectra of **3ap** in cyclohexane at 25 °C.

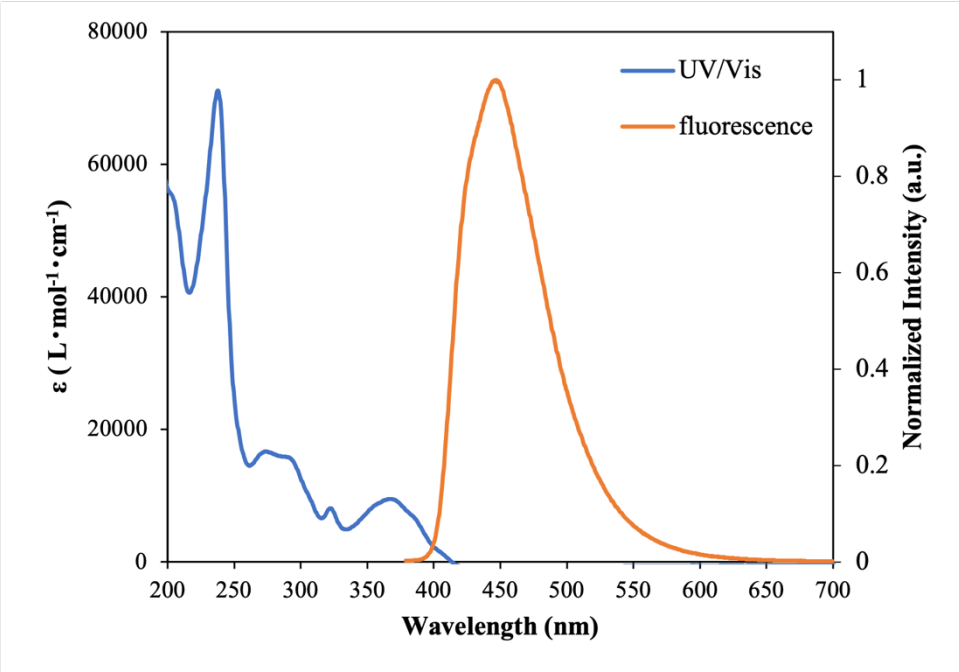


Figure S4. UV-Vis (blue) and fluorescence (orange, excitation at 367 nm) spectra of **3bz** in cyclohexane at 25 °C.

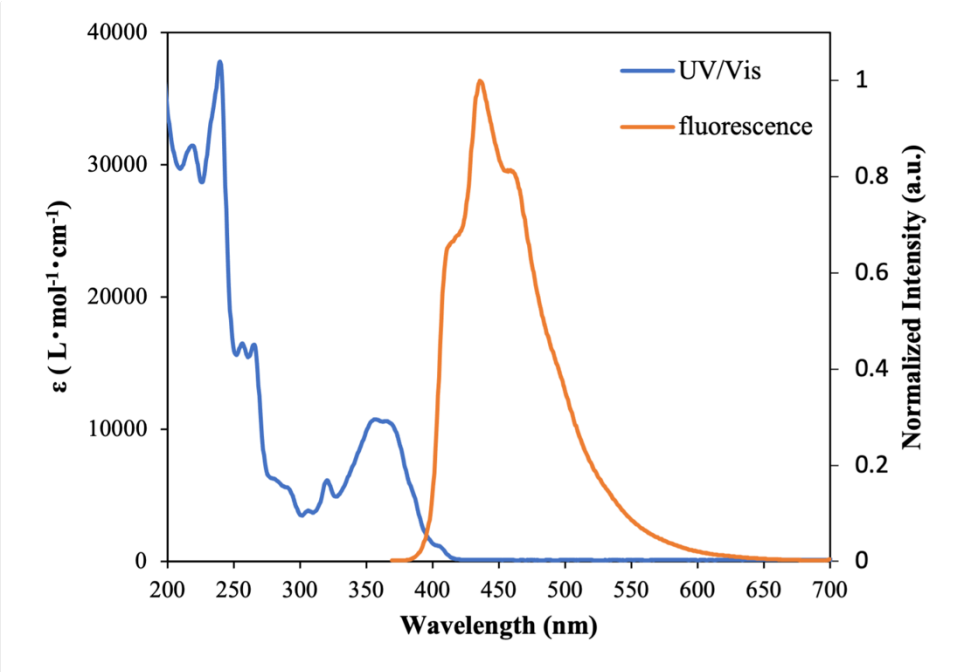


Figure S5. UV-Vis (blue) and fluorescence (orange, excitation at 357 nm) spectra of **3ao** in cyclohexane at 25 °C.

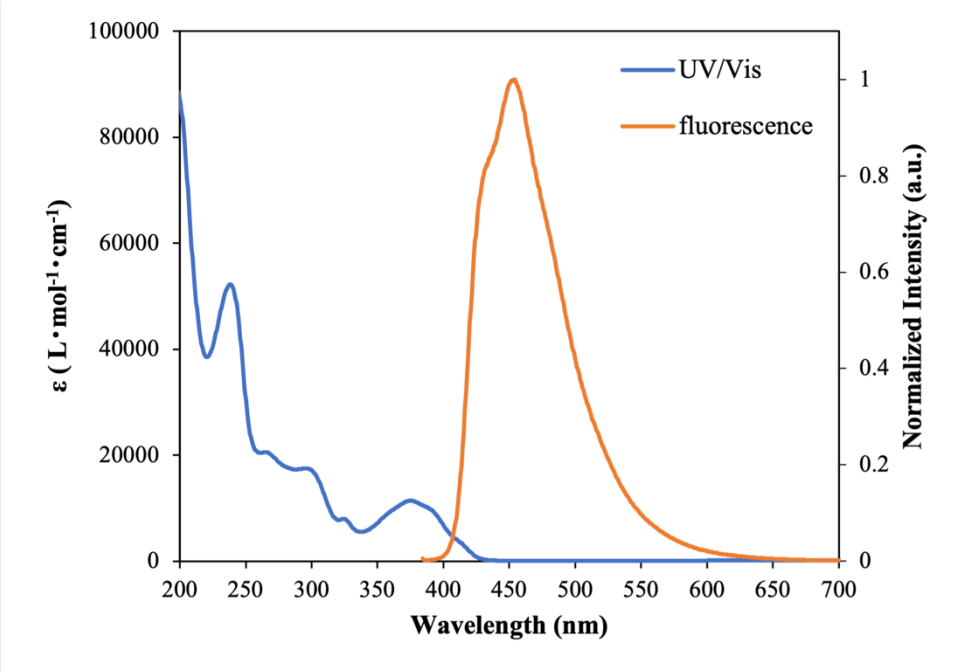


Figure S6. UV-Vis (blue) and fluorescence (orange, excitation at 375 nm) spectra of **3bj** in cyclohexane at 25 °C.

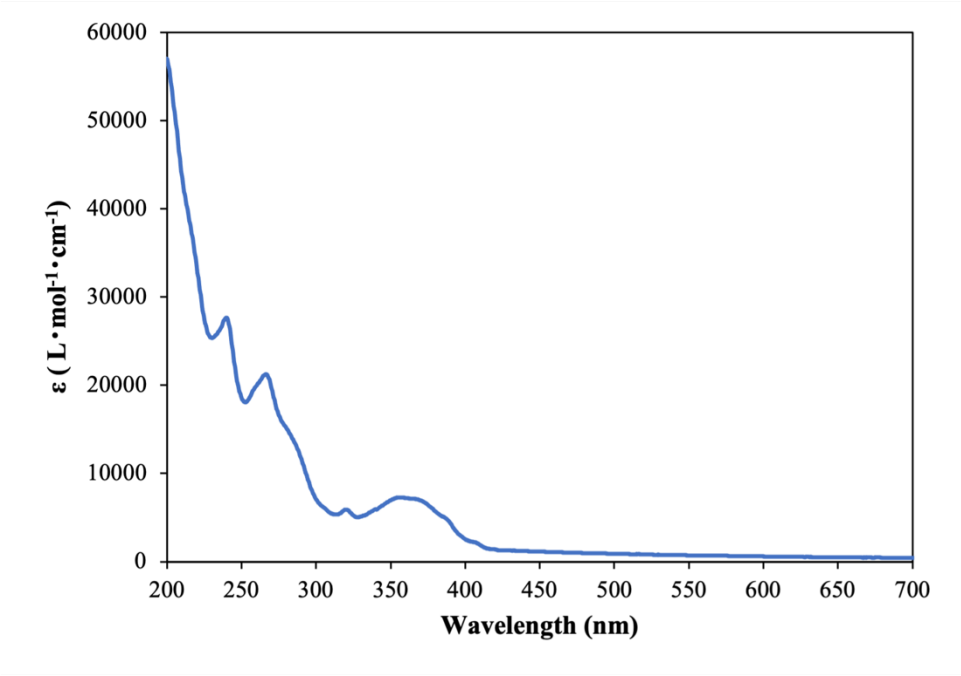


Figure S7. UV-Vis (blue) spectra of **3am** in cyclohexane at 25 °C.

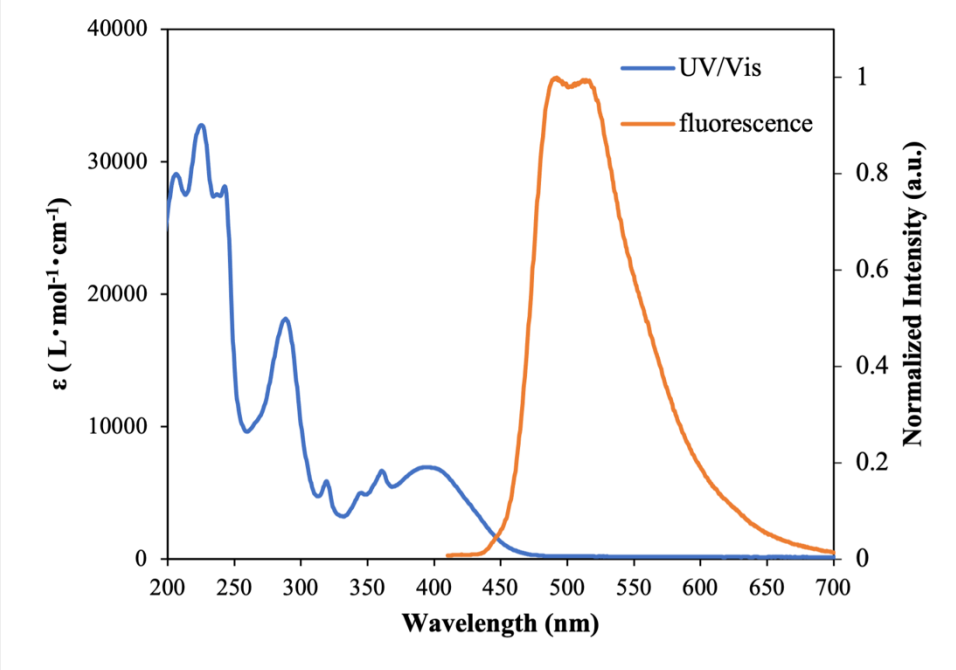


Figure S8. UV-Vis (blue) and fluorescence (orange, excitation at 395 nm) spectra of **3an** in cyclohexane at 25 °C.

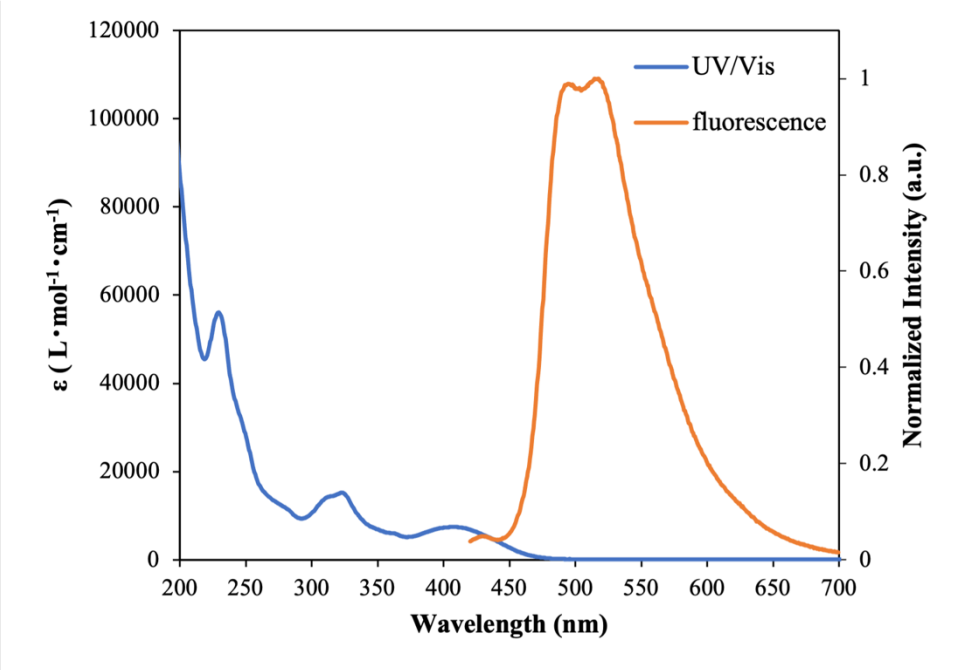


Figure S9. UV-Vis (blue) and fluorescence (orange, excitation at 407 nm) spectra of **3bn** in cyclohexane at 25 °C.

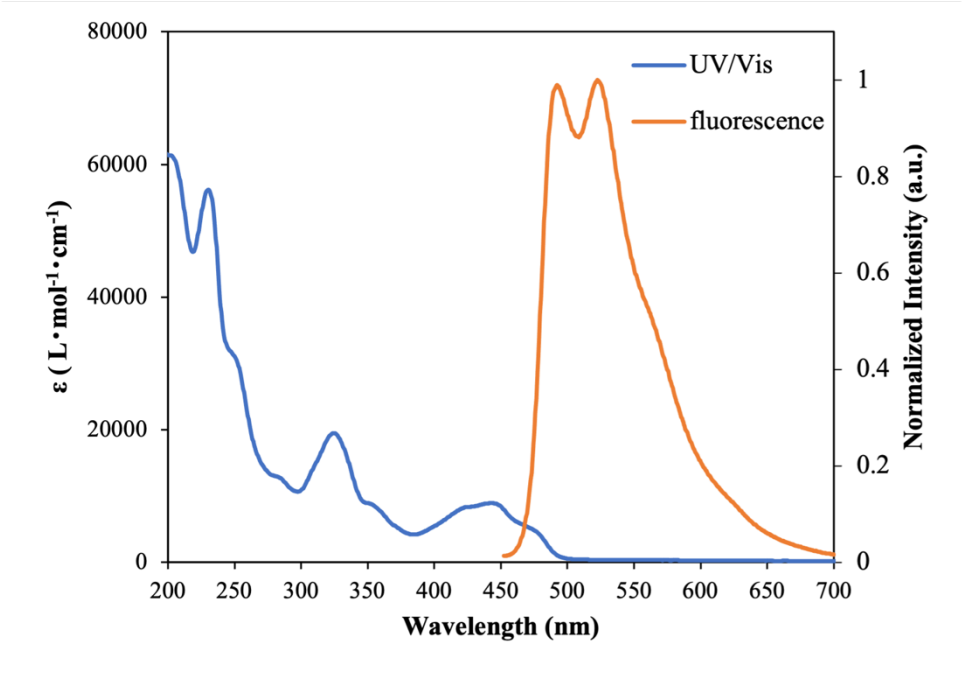


Figure S10. UV-Vis (blue) and fluorescence (orange, excitation at 443 nm) spectra of **3bu** in cyclohexane at 25 °C.

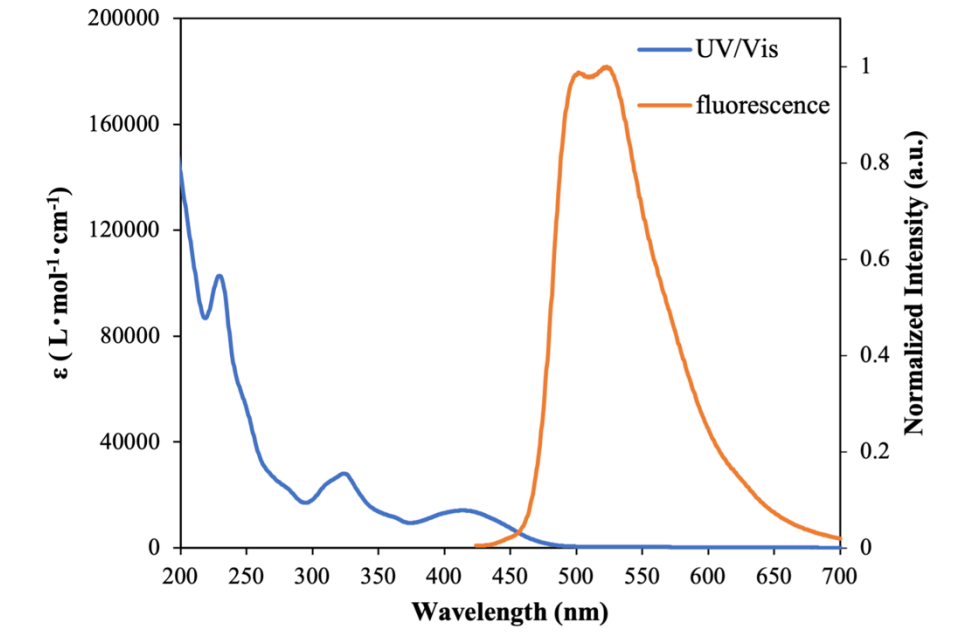


Figure S11. UV-Vis (blue) and fluorescence (orange, excitation at 414 nm) spectra of **3bv** in cyclohexane at 25 °C.

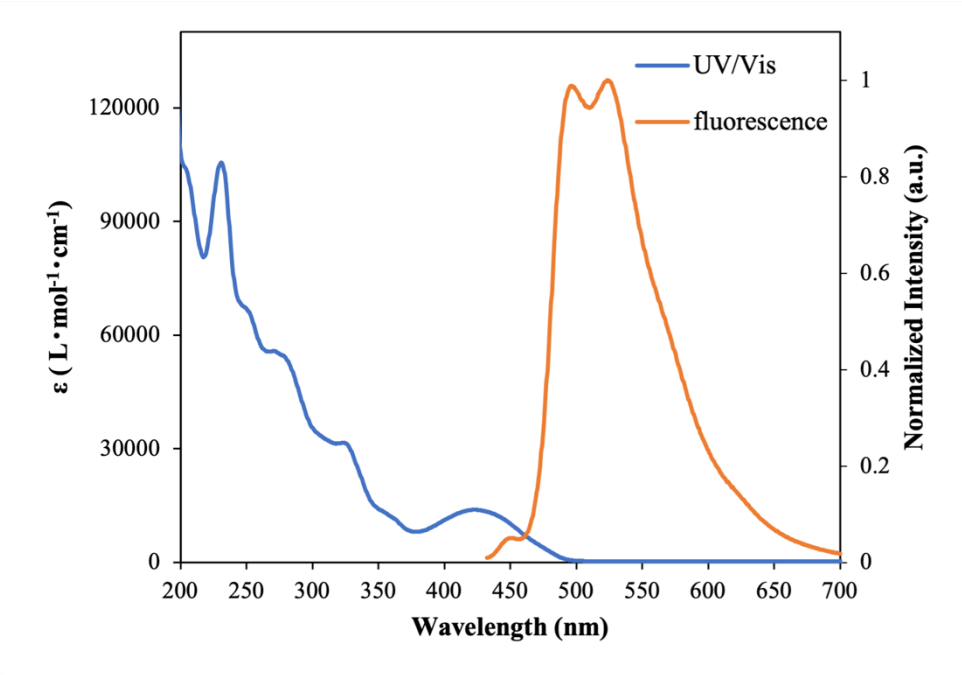


Figure S12. UV-Vis (blue) and fluorescence (orange, excitation at 423 nm) spectra of **3bw** in cyclohexane at 25 °C.

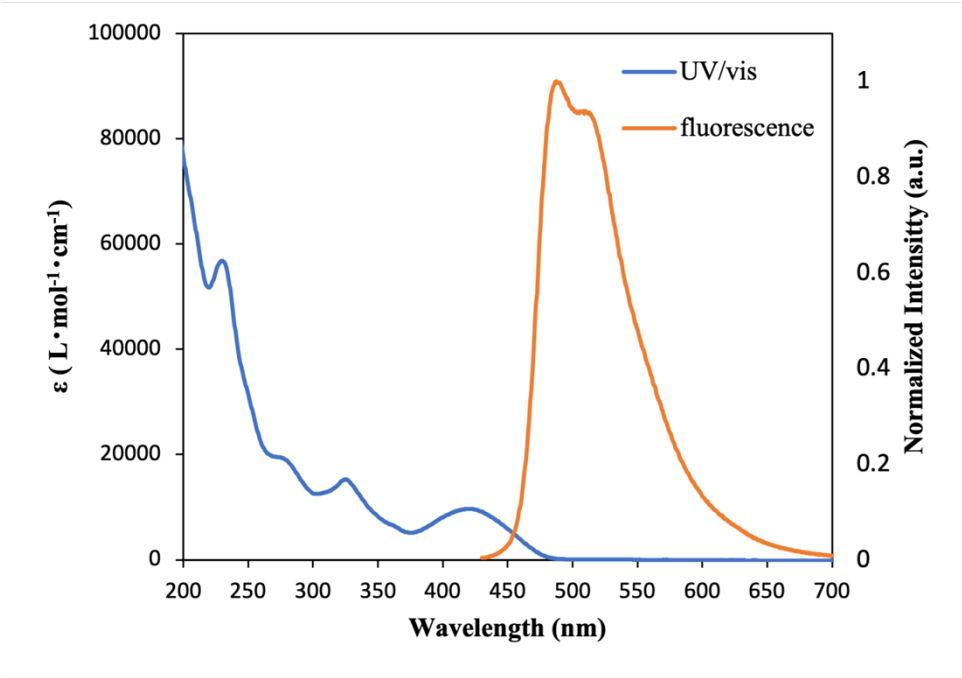


Figure S13. UV-Vis (blue) and fluorescence (orange, excitation at 420 nm) spectra of **3bx** in cyclohexane at 25 °C.

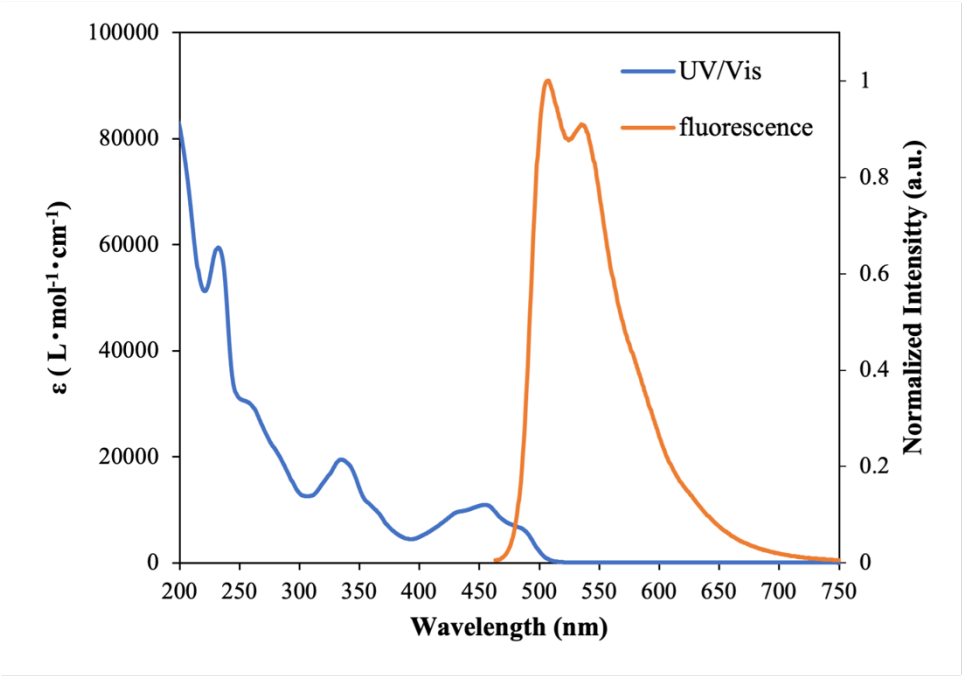


Figure S14. UV-Vis (blue) and fluorescence (orange, excitation at 455 nm) spectra of **3by** in cyclohexane at 25 °C.

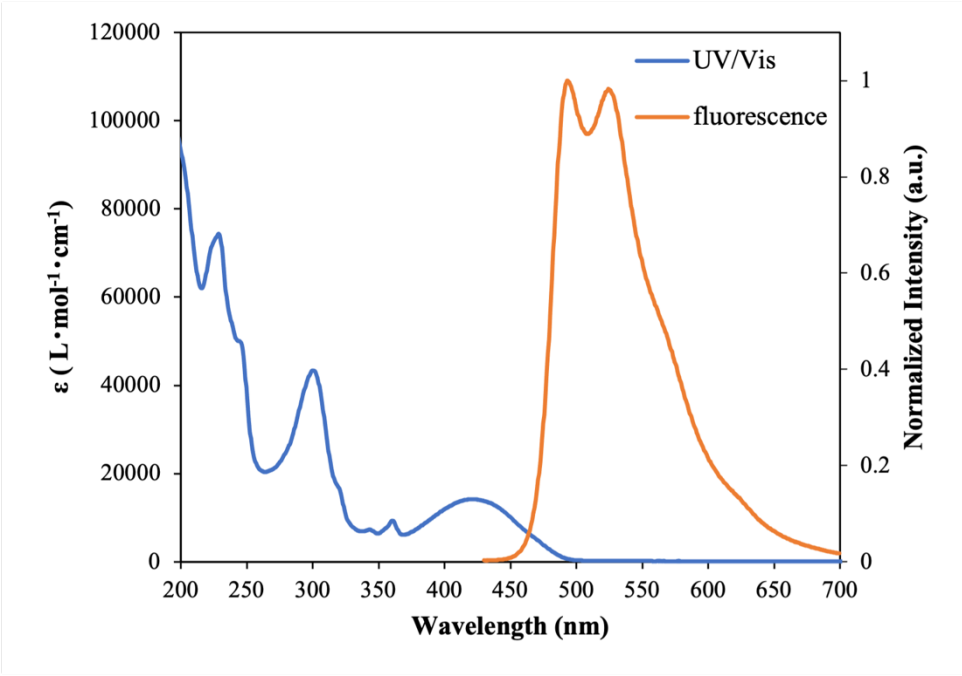


Figure S15. UV-Vis (blue) and fluorescence (orange, excitation at 420 nm) spectra of **3au** in cyclohexane at 25 °C.

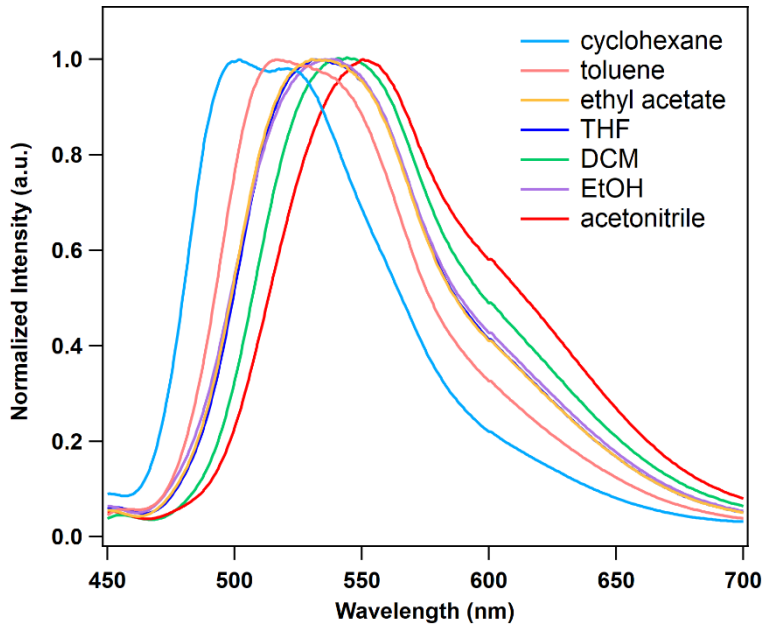


Figure S16. Normalized fluorescence spectra of **3bv** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) obtained by 440 nm excitation at 25 °C.

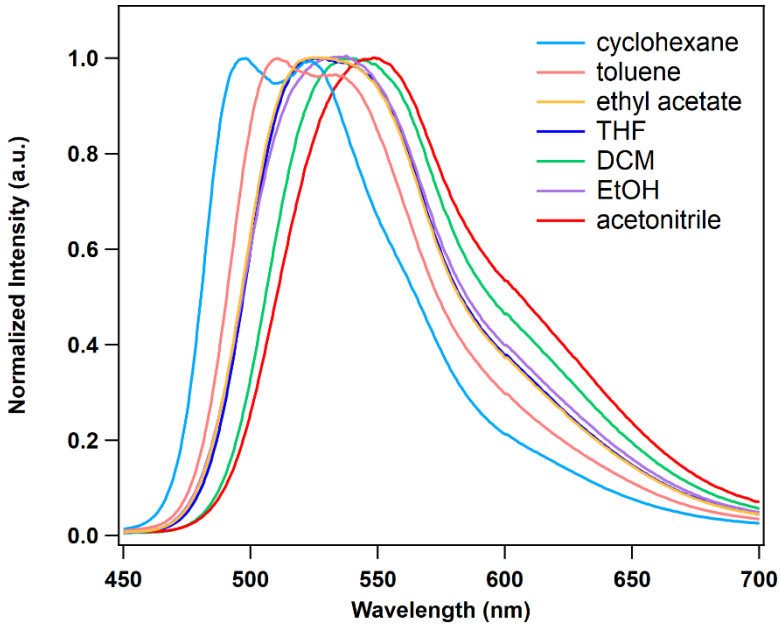


Figure S17. Normalized fluorescence spectra of **3bw** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) obtained by 440 nm excitation at 25 °C.

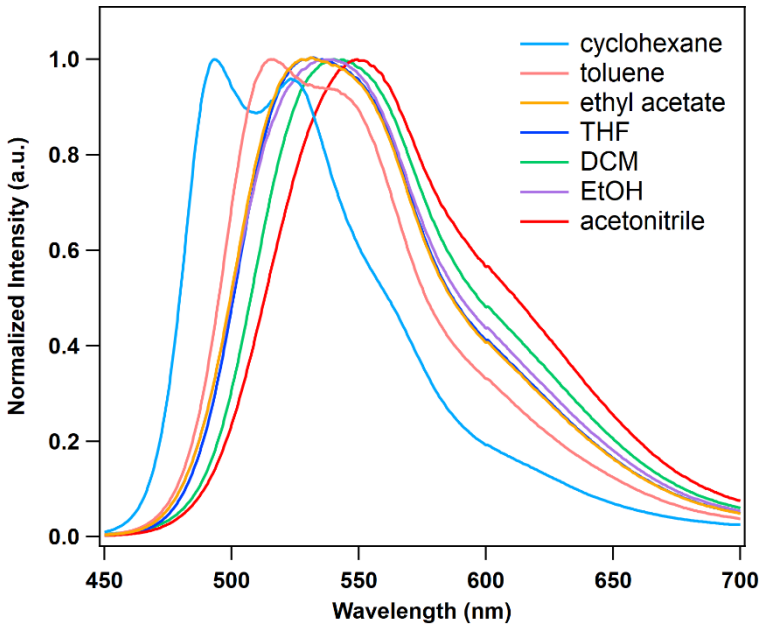


Figure S18. Normalized fluorescence spectra of **3au** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) obtained by 440 nm excitation at 25 °C.

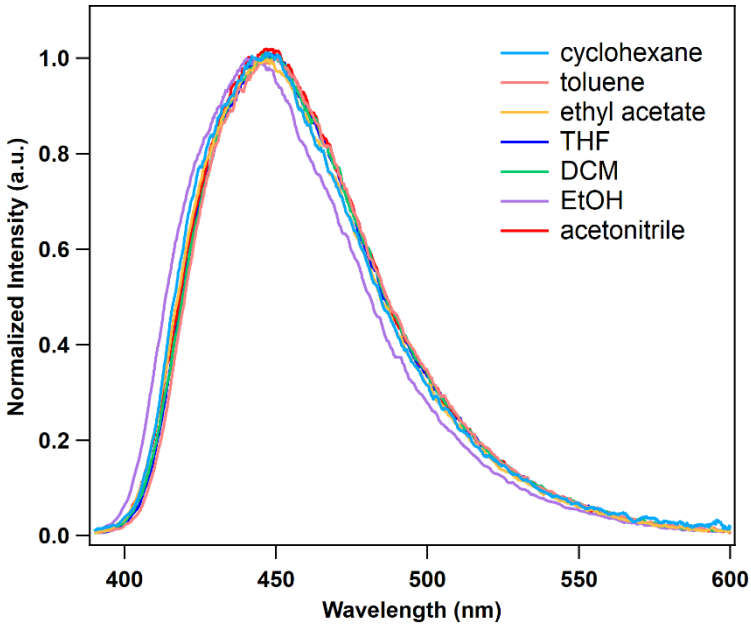


Figure S19. Normalized fluorescence spectra of **3bz** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) obtained by 370 nm excitation at 25 °C.

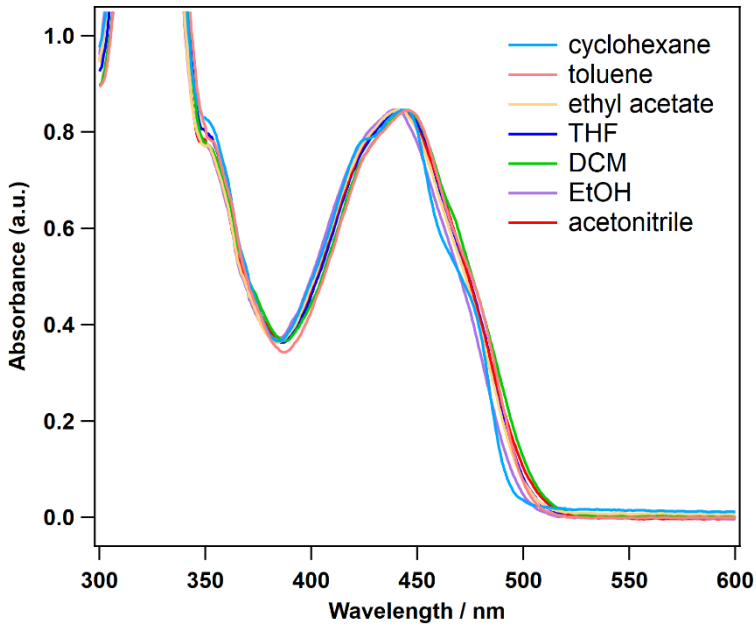


Figure S20. UV-Vis spectra of **3bu** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) at 25 °C.

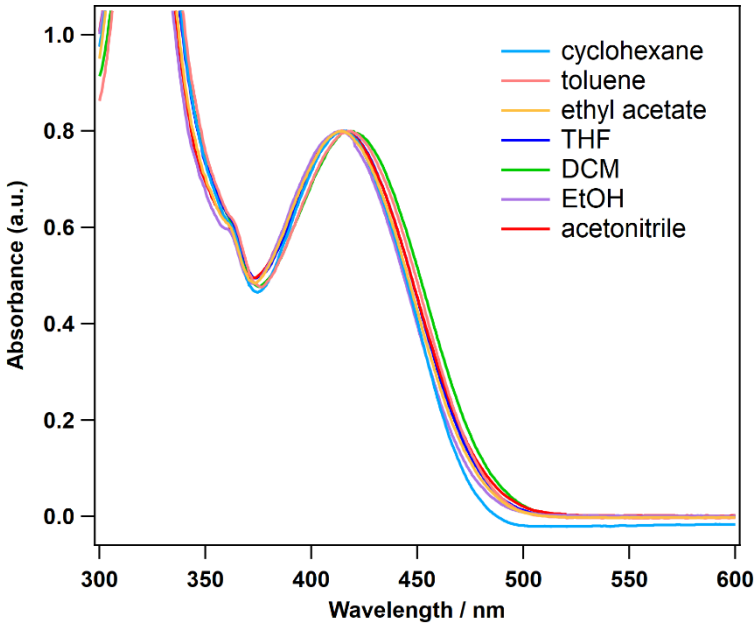


Figure S21. UV-Vis spectra of **3bv** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) at 25 °C.

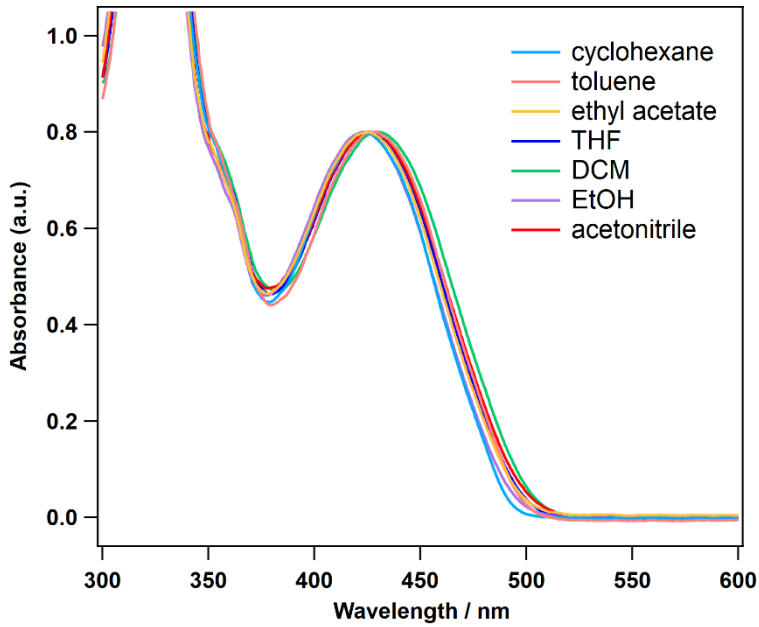


Figure S22. UV-Vis spectra of **3bw** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) at 25 °C.

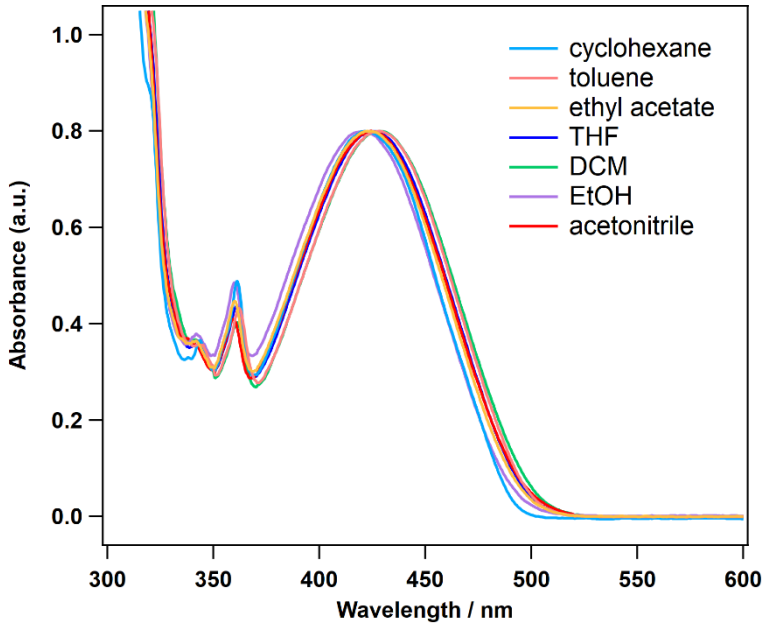


Figure S23. UV-Vis spectra of **3au** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) at 25 °C.

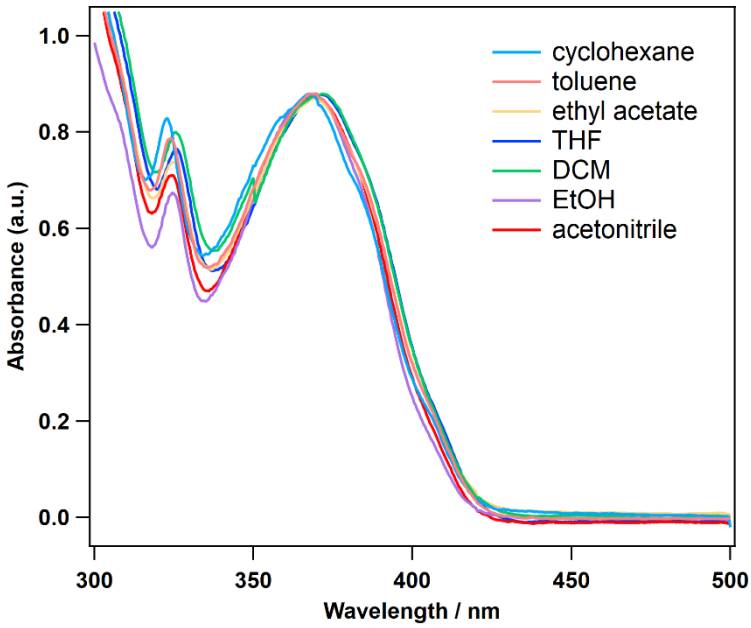
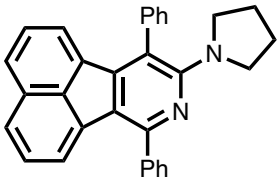


Figure S24. UV-Vis spectra of **3bz** in cyclohexane (sky blue line), toluene (pink line), ethyl acetate (yellow line), THF (blue line), DCM (green line), EtOH (purple line), and acetonitrile (red line) at 25 °C.

10. Fluorescence Quantum Yields of 3bu in a Variety of Solvents

Table S1.[a]



3bu

entry	solvent	Φ_F[c] (nm)[b]
1	cyclohexane	0.67 (440)
2	toluene	0.76 (440)
3	ethyl acetate	0.59 (440)
4	THF	0.60 (440)
5	DCM	0.75 (440)
6	EtOH	0.62 (440)
7	acetonitrile	0.54 (440)

[a]Measured at 25 °C. [b]Excitation wavelengh. [c]Standards for the determination of quantum yields: Fluorescein.

11. Theoretical Calculation

Quantum chemical calculations were performed with the Gaussian 09 program package.[10] Geometry optimization and vibrational frequency analysis in the ground state were performed at the B3LYP/6-311G(d,p) level with the polarizable continuum model (PCM) in cyclohexane and acetonitrile. The vertical transition energies, contributions of excitation configurations, and oscillator strengths of the transitions from the ground to the excited singlet states were estimated by time-dependent DFT (TD-DFT).

Table S2. Cartesian coordinates [Å] of the optimized geometries of **3bu**, **3bv**, **3bw**, **3bz**, and **3au** in the ground state calculated at the PCM/B3LYP/6-311G(d,p) level in cyclohexane and acetonitrile. Onsager cavity radius, a_0, was calculated at the PCM/B3LYP/6-311G(d,p) level in cyclohexane.

3bu ; a_0 = 6.14 [Å]

atom	cyclohexane			acetonitrile		
	X	Y	Z	X	Y	Z
C	-0.093705	2.280115	-0.063487	-0.090064	2.275195	-0.064202
C	0.738919	3.383310	-0.040576	0.743118	3.378645	-0.039267

C	-1.492921	2.497168	-0.023132	-1.489752	2.493612	-0.028704
C	-2.216584	1.275974	-0.016854	-2.215334	1.272916	-0.029650
C	-3.593577	1.346133	0.078575	-3.593786	1.344904	0.050917
H	-4.208324	0.456854	0.109879	-4.211553	0.457342	0.069506
C	-4.220086	2.619695	0.142964	-4.219639	2.619537	0.112209
C	-3.508307	3.801491	0.121718	-3.505862	3.801029	0.100807
C	-2.088438	3.767023	0.044948	-2.084827	3.764498	0.035536
C	-1.204629	4.879056	0.052260	-1.200272	4.876671	0.047572
C	0.161361	4.678293	0.011423	0.166225	4.674516	0.011772
H	0.823246	5.537179	0.025340	0.828633	5.532703	0.027380
H	-1.605976	5.886047	0.098934	-1.601132	5.883687	0.092189
H	-5.301834	2.655388	0.213625	-5.301900	2.656408	0.170989
H	-4.024541	4.754036	0.173297	-4.021005	4.754114	0.149065
H	1.815423	3.287946	-0.058049	1.819612	3.283507	-0.056855
C	-1.204873	0.204372	-0.068181	-1.204464	0.200794	-0.077211
C	0.093783	0.809056	-0.087751	0.096404	0.803558	-0.092173
N	-0.158424	-1.934130	0.055168	-0.161243	-1.940692	0.037156
C	1.251410	0.041252	-0.054963	1.253043	0.035239	-0.059986
C	-1.269174	-1.185826	-0.005253	-1.270708	-1.188448	-0.013907
C	1.064861	-1.380754	0.011940	1.066165	-1.389289	-0.006004
C	2.589444	0.702687	0.024331	2.589780	0.698937	0.031650
C	3.194215	1.260174	-1.109076	3.204495	1.256769	-1.096274
C	3.245815	0.812882	1.255824	3.231059	0.812884	1.270949
C	4.427494	1.901019	-1.016505	4.434093	1.903229	-0.990258
H	2.692121	1.188474	-2.067692	2.716330	1.179729	-2.061577
C	4.478364	1.456826	1.351208	4.460214	1.462141	1.379537
H	2.783660	0.393316	2.142466	2.762826	0.391610	2.153727
C	5.074055	2.001132	0.214993	5.066126	2.007863	0.248900
H	4.882783	2.323402	-1.905489	4.897972	2.325657	-1.874569
H	4.971410	1.534884	2.313881	4.942710	1.541993	2.347252
H	6.033430	2.500799	0.288167	6.022526	2.511413	0.332143
C	-2.544872	-1.959354	-0.002066	-2.550596	-1.956649	0.004139
C	-2.775760	-2.926570	0.983478	-2.795480	-2.887690	1.021538
C	-3.499919	-1.783553	-1.009773	-3.497114	-1.809083	-1.016668
C	-3.946461	-3.679039	0.978239	-3.970149	-3.635457	1.031272

H	-2.028077	-3.081795	1.751818	-2.059516	-3.018922	1.805899
C	-4.665295	-2.547595	-1.024480	-4.666714	-2.567778	-1.015361
H	-3.320292	-1.058037	-1.794880	-3.310011	-1.109332	-1.823220
C	-4.895574	-3.492312	-0.026373	-4.909548	-3.478482	0.011816
H	-4.116727	-4.415726	1.755657	-4.151157	-4.343498	1.832301
H	-5.389798	-2.408071	-1.819252	-5.384955	-2.449787	-1.818975
H	-5.804209	-4.083901	-0.034899	-5.821050	-4.065376	0.015547
C	3.447540	-2.102158	-0.509673	3.459244	-2.097802	-0.506447
C	1.807070	-3.711836	0.288793	1.813269	-3.724110	0.249572
C	3.925560	-3.533022	-0.799284	3.948939	-3.522848	-0.801073
H	3.405403	-1.493330	-1.413749	3.435015	-1.478219	-1.403105
H	4.118325	-1.602556	0.195143	4.109156	-1.606688	0.222958
C	3.186521	-4.370021	0.251391	3.197594	-4.372554	0.230041
H	1.280187	-3.853627	1.233855	1.274701	-3.873575	1.186834
H	1.155450	-4.098885	-0.503333	1.179718	-4.114868	-0.555507
H	5.012282	-3.619997	-0.744325	5.034676	-3.603493	-0.727078
H	3.613783	-3.837506	-1.803095	3.655844	-3.819153	-1.812759
H	3.678758	-4.278692	1.224793	3.671879	-4.285515	1.212443
H	3.130929	-5.430415	-0.001133	3.151843	-5.430813	-0.032068
N	2.102018	-2.284370	0.072132	2.099253	-2.292937	0.040164

3bv ; a_0 = 6.44 [Å]

atom	cyclohexane			acetonitrile		
	X	Y	Z	X	Y	Z
C	0.323160	-0.903463	-0.033063	0.315753	-0.901464	-0.037421
C	1.401623	0.039307	-0.082910	1.399736	0.036213	-0.079003
C	-0.999024	-0.472366	-0.011670	-1.004137	-0.463964	-0.012875
C	1.090662	1.395048	-0.022561	1.095778	1.392890	-0.018445
C	2.669427	-0.710662	-0.170615	2.664719	-0.719427	-0.160052
C	0.916075	-2.261890	0.011860	0.901102	-2.263170	-0.001980
C	-1.200732	0.943015	-0.061372	-1.200123	0.952468	-0.055964
C	-2.132499	-1.433348	0.133016	-2.139831	-1.423048	0.135013
C	2.104201	2.484322	0.068000	2.117665	2.475854	0.066912
N	-0.188427	1.810464	-0.047926	-0.181954	1.815822	-0.040921

C	2.317133	-2.082081	-0.095792	2.304251	-2.089673	-0.097973
C	4.004645	-0.394561	-0.331102	4.003803	-0.409043	-0.302532
C	0.433330	-3.550610	0.139101	0.410270	-3.551201	0.104795
N	-2.494728	1.488568	-0.050179	-2.492236	1.500908	-0.043205
C	-2.475197	-2.314229	-0.901323	-2.509760	-2.279700	-0.910178
C	-2.867548	-1.481224	1.323731	-2.842777	-1.497572	1.343948
C	3.106663	2.465108	1.045005	3.098264	2.470625	1.066517
C	2.023551	3.585908	-0.792926	2.069084	3.555427	-0.824776
C	3.244807	-3.135687	-0.127421	3.226805	-3.148300	-0.131362
C	4.961102	-1.443324	-0.386774	4.955660	-1.462668	-0.357075
H	4.341372	0.630047	-0.414406	4.348542	0.613958	-0.371411
C	1.351982	-4.632291	0.133255	1.323313	-4.638173	0.094743
H	-0.622492	-3.757727	0.242293	-0.647551	-3.754905	0.193147
C	-2.612260	2.901566	0.334473	-2.611908	2.909370	0.358841
C	-3.397589	1.143810	-1.162621	-3.386538	1.172363	-1.170077
C	-3.521984	-3.221144	-0.748750	-3.554154	-3.188816	-0.751016
H	-1.916797	-2.285730	-1.830707	-1.976438	-2.231681	-1.853307
C	-3.909727	-2.392384	1.479844	-3.883054	-2.410668	1.506305
H	-2.615596	-0.802287	2.129690	-2.568002	-0.840406	2.160764
C	4.015520	3.515868	1.149625	4.015335	3.515395	1.166002
H	3.163384	1.632918	1.736966	3.133610	1.654373	1.778728
C	2.940149	4.628794	-0.697952	2.993745	4.592631	-0.734138
H	1.233768	3.615597	-1.533489	1.302065	3.573717	-1.589702
C	4.611645	-2.774332	-0.280037	4.597610	-2.793302	-0.267212
C	2.713886	-4.447547	-0.004409	2.688062	-4.459272	-0.027162
H	6.005381	-1.179345	-0.512839	6.002550	-1.203194	-0.468401
H	0.962093	-5.639017	0.234692	0.926958	-5.643654	0.179687
C	-4.048201	3.212790	0.765942	-4.051966	3.219443	0.777327
H	-2.319321	3.559237	-0.499472	-2.310111	3.578115	-0.463025
H	-1.912481	3.097049	1.145212	-1.924296	3.092006	1.183379
C	-4.857069	1.408210	-0.788978	-4.848796	1.437752	-0.809503
H	-3.128752	1.747013	-2.047590	-3.106097	1.785763	-2.043625
H	-3.263453	0.099338	-1.430193	-3.251235	0.131207	-1.449401
C	-4.241451	-3.264614	0.444228	-4.242952	-3.258484	0.459223
H	-3.774025	-3.893272	-1.561537	-3.827548	-3.842381	-1.571752

H	-4.462787	-2.421014	2.412165	-4.411711	-2.460002	2.451689
C	3.939810	4.597241	0.273759	3.969927	4.576078	0.262271
H	4.778310	3.492095	1.920024	4.761576	3.502268	1.952438
H	2.871480	5.469653	-1.379268	2.950640	5.415512	-1.438882
H	5.373880	-3.545173	-0.316415	5.355640	-3.567995	-0.303247
H	3.381392	-5.302737	-0.015556	3.351127	-5.317545	-0.041281
C	-5.054831	2.858553	-0.334438	-5.046265	2.883140	-0.339540
H	-4.118138	4.273897	1.026160	-4.120441	4.277299	1.049979
H	-4.276849	2.641188	1.672605	-4.293374	2.637474	1.674078
H	-5.143129	0.722190	0.015391	-5.148916	0.744044	-0.016591
H	-5.492655	1.181976	-1.651158	-5.474075	1.224960	-1.682221
H	-5.054763	-3.971175	0.565446	-5.053786	-3.966881	0.585248
H	4.649636	5.413062	0.352547	4.686243	5.386383	0.336972
H	-6.080028	3.017142	0.013843	-6.074526	3.040996	-0.000744
H	-4.904896	3.529165	-1.190419	-4.883235	3.563234	-1.185314

3bw ; a_0 = 6.43 [Å]

atom	cyclohexane			acetonitrile		
	X	Y	Z	X	Y	Z
C	-1.388276	-2.428648	0.203478	-1.382204	2.429084	0.193809
C	-0.951934	-3.702917	0.512626	-0.942177	-3.706235	0.487738
C	-2.779799	-2.220091	0.039625	-2.775521	-2.220799	0.041500
C	-3.081398	-0.861801	-0.234888	-3.080816	-0.861254	-0.224498
C	-4.401530	-0.528051	-0.468130	-4.404123	-0.526706	-0.440656
H	-4.700093	0.485254	-0.701346	-4.707563	0.487638	-0.662730
C	-5.393355	-1.542722	-0.398594	-5.395119	-1.542506	-0.366110
C	-5.093301	-2.856686	-0.100766	-5.090905	-2.858835	-0.080401
C	-3.742931	-3.237555	0.131376	-3.737380	-3.239908	0.135775
C	-3.259903	-4.536663	0.443415	-3.250897	-4.541613	0.433930
C	-1.907622	-4.745208	0.633080	-1.896091	-4.750688	0.608535
H	-1.555044	-5.741104	0.877406	-1.540377	-5.748220	0.840418
H	-3.956434	-5.363498	0.534298	-3.945973	-5.369300	0.525142
H	-6.425249	-1.265076	-0.583630	-6.429027	-1.264066	-0.537171
H	-5.881564	-3.599920	-0.048137	-5.877766	-3.603002	-0.024289

H	0.094563	-3.927394	0.664354	0.105823	-3.932624	0.625620
C	-1.789236	-0.149694	-0.226756	-1.788816	-0.148741	-0.225114
C	-0.747154	-1.110474	-0.016811	-0.743982	-1.108759	-0.021840
N	-0.134451	1.551404	-0.410201	-0.136766	1.556166	-0.409479
C	0.588938	-0.726137	-0.023873	0.591262	-0.722013	-0.027826
C	-1.427715	1.189651	-0.348426	-1.429940	1.190468	-0.349090
C	0.846963	0.655798	-0.290393	0.847874	0.661433	-0.290187
C	1.674533	-1.691505	0.325778	1.676666	-1.687174	0.325348
C	2.282751	-1.626642	1.585392	2.259625	-1.637064	1.597844
C	2.080136	-2.695869	-0.561736	2.102554	-2.677583	-0.568249
C	3.264345	-2.545287	1.950629	3.239189	-2.556036	1.969337
H	1.977432	-0.854507	2.281906	1.936887	-0.877852	2.301086
C	3.067580	-3.610852	-0.201076	3.087015	-3.593521	-0.200680
H	1.614346	-2.762994	-1.538989	1.657663	-2.733921	-1.555745
C	3.661445	-3.539735	1.058111	3.657294	-3.536691	1.070543
H	3.718790	-2.483703	2.933242	3.674644	-2.505854	2.961077
H	3.369225	-4.380826	-0.902526	3.404633	-4.353011	-0.906250
H	4.426634	-4.253600	1.341406	4.420096	-4.251148	1.358403
C	-2.401972	2.315724	-0.420680	-2.409475	2.312580	-0.427449
C	-2.273586	3.288758	-1.419718	-2.309707	3.259773	-1.454958
C	-3.415578	2.460655	0.534117	-3.401084	2.477732	0.547534
C	-3.153575	4.365409	-1.478437	-3.194391	4.333265	-1.518792
H	-1.477400	3.191179	-2.147517	-1.535510	3.145873	-2.204215
C	-4.287605	3.546021	0.483994	-4.277941	3.559557	0.491603
H	-3.509902	1.730011	1.328995	-3.476380	1.765972	1.361439
C	-4.163981	4.497732	-0.526565	-4.181300	4.486942	-0.544940
H	-3.048427	5.104507	-2.264974	-3.111876	5.051642	-2.326741
H	-5.059426	3.650717	1.238479	-5.032997	3.679635	1.260357
H	-4.845307	5.340164	-0.568091	-4.866363	5.325901	-0.590927
N	2.160961	1.142603	-0.373124	2.161211	1.147882	-0.371137
C	3.053798	0.545971	-1.384987	3.052564	0.554491	-1.389416
C	4.473900	0.278733	-0.880026	4.471874	0.279077	-0.887600
H	3.091278	1.215215	-2.258961	3.091881	1.232230	-2.255889
H	2.614471	-0.385888	-1.737017	2.608791	-0.371940	-1.749602
C	5.399565	-0.243649	-1.984739	5.396037	-0.230628	-1.999719

H	4.896603	1.199271	-0.460793	4.896584	1.193828	-0.458221
H	4.425919	-0.444187	-0.060439	4.423420	-0.454991	-0.077544
C	6.820431	-0.529773	-1.490272	6.816851	-0.524883	-1.509666
H	4.972618	-1.159262	-2.411235	4.967383	-1.139433	-2.438858
H	5.438575	0.485329	-2.803519	5.435411	0.510121	-2.807499
H	7.457149	-0.900921	-2.298113	7.452194	-0.885303	-2.323415
H	7.287909	0.374072	-1.087048	7.284695	0.373335	-1.094464
H	6.816184	-1.282412	-0.696050	6.812920	-1.288915	-0.726141
C	2.342752	2.595258	-0.219164	2.349768	2.598982	-0.209121
C	2.150184	3.080322	1.219259	2.161767	3.078086	1.232168
H	1.669826	3.140924	-0.890640	1.682059	3.152635	-0.879470
H	3.366391	2.816471	-0.529740	3.373752	2.817237	-0.518955
C	2.362651	4.591666	1.360638	2.370321	4.589662	1.378579
H	1.141023	2.824649	1.554455	1.156304	2.816513	1.575177
H	2.850342	2.542985	1.869780	2.868139	2.542506	1.877244
C	2.189001	5.089268	2.798607	2.207364	5.080045	2.820337
H	3.366068	4.855465	1.003301	3.369872	4.856805	1.014165
H	1.656920	5.117268	0.706764	1.658330	5.116880	0.732446
H	2.342811	6.169692	2.868912	2.358051	6.160689	2.893476
H	1.183466	4.871259	3.171287	1.205982	4.856594	3.201506
H	2.903614	4.606764	3.472939	2.930236	4.596986	3.485297

3bz ; a_0 = 5.89 [Å]

atom	cyclohexane			acetonitrile		
	X	Y	Z	X	Y	Z
C	-1.177694	1.309944	0.075431	-1.174430	1.312931	0.066487
C	-2.405285	1.931982	0.194445	-2.400200	1.942520	0.171908
C	-0.018735	2.118301	-0.002946	-0.010115	2.115214	-0.002858
C	1.162419	1.339552	-0.086718	1.168179	1.330655	-0.074434
C	2.364168	2.005587	-0.229193	2.376078	1.990227	-0.198922
H	3.302562	1.475698	-0.320517	3.312063	1.458353	-0.275012
C	2.368062	3.425836	-0.256129	2.388134	3.410838	-0.224392
C	1.213845	4.174491	-0.140496	1.235708	4.165845	-0.124446
C	-0.042028	3.521569	-0.009484	-0.025548	3.519112	-0.009350

C	-1.319893	4.131892	0.109534	-1.301492	4.137227	0.095462
C	-2.454368	3.350233	0.215680	-2.441821	3.361353	0.190130
H	-3.421211	3.831436	0.311182	-3.404875	3.846841	0.273819
H	-1.401999	5.213561	0.117236	-1.377949	5.216869	0.101283
H	3.319305	3.934278	-0.366947	3.341418	3.913259	-0.320469
H	1.261534	5.258010	-0.154964	1.289493	5.246670	-0.137707
H	-3.325552	1.368137	0.268673	-3.323536	1.387052	0.236560
C	0.724127	-0.073656	-0.036277	0.721604	-0.079598	-0.028068
C	-0.704819	-0.087591	0.014178	-0.708206	-0.086970	0.014909
N	0.747032	-2.461236	-0.093624	0.736323	-2.468117	-0.068581
C	-1.385459	-1.305612	-0.009872	-1.393460	-1.303192	-0.002443
C	1.414850	-1.291692	-0.045743	1.408010	-1.299276	-0.033864
C	-0.582088	-2.455979	-0.079989	-0.594809	-2.457166	-0.055859
C	-2.867030	-1.450197	-0.000074	-2.875767	-1.443214	0.002280
C	-3.643233	-0.979138	-1.067738	-3.646508	-0.972089	-1.069741
C	-3.507562	-2.110187	1.056201	-3.521225	-2.099134	1.058759
C	-5.024216	-1.156276	-1.073492	-5.028590	-1.145168	-1.079532
H	-3.158351	-0.481905	-1.900296	-3.160194	-0.478243	-1.900585
C	-4.890310	-2.283961	1.051775	-4.905117	-2.268602	1.050154
H	-2.918420	-2.478992	1.888458	-2.938385	-2.468102	1.892517
C	-5.652534	-1.806934	-0.012114	-5.662547	-1.791547	-0.018076
H	-5.609219	-0.791701	-1.910521	-5.608570	-0.780357	-1.916892
H	-5.370598	-2.792208	1.880383	-5.388674	-2.771856	1.876884
H	-6.727839	-1.944568	-0.017305	-6.736068	-1.924753	-0.026087
C	2.898527	-1.417160	0.002076	2.892868	-1.424974	0.002260
C	3.552965	-2.267683	-0.897990	3.546713	-2.231553	-0.938565
C	3.654967	-0.760133	0.979820	3.650895	-0.807024	1.004527
C	4.933700	-2.430708	-0.842569	4.928996	-2.393862	-0.894536
H	2.966361	-2.802262	-1.635006	2.964534	-2.728560	-1.702265
C	5.035720	-0.934595	1.044324	5.032984	-0.980224	1.056511
H	3.158210	-0.127227	1.705956	3.157710	-0.203973	1.754894
C	5.680229	-1.763619	0.128179	5.676779	-1.768255	0.103359
H	5.427351	-3.084000	-1.553401	5.421277	-3.010229	-1.635129
H	5.605899	-0.429034	1.815774	5.603589	-0.504843	1.843284
H	6.755194	-1.896622	0.176074	6.750030	-1.898973	0.141287

H	-1.062420	-3.429563	-0.134329	-1.078796	-3.426778	-0.096266

3au ; a_0 = 5.30 [Å]

atom	cyclohexane			acetonitrile		
	X	Y	Z	X	Y	Z
C	-1.613276	-1.165599	-0.084366	-1.610110	-1.166035	-0.083687
C	-1.995730	-2.493940	-0.078275	-1.989123	-2.496042	-0.077012
C	-2.627384	-0.181197	0.028680	-2.627031	-0.183766	0.029186
C	-2.082778	1.129824	0.054501	-2.086340	1.129452	0.054593
C	-2.959595	2.191597	0.168351	-2.966516	2.189561	0.167680
H	-2.616321	3.217989	0.198736	-2.627433	3.217285	0.197721
C	-4.354264	1.928582	0.253877	-4.360903	1.922157	0.252896
C	-4.872756	0.649758	0.234722	-4.876132	0.641207	0.234438
C	-3.997087	-0.467140	0.123156	-3.996644	-0.473498	0.123622
C	-4.355765	-1.842403	0.105627	-4.352184	-1.850205	0.106589
C	-3.376600	-2.812175	0.012523	-3.369742	-2.817571	0.013751
H	-3.666085	-3.857210	0.011023	-3.655840	-3.863350	0.012569
H	-5.400019	-2.128835	0.173039	-5.395502	-2.139337	0.174005
H	-5.031584	2.771132	0.341895	-5.040324	2.762906	0.340157
H	-5.943471	0.492483	0.307673	-5.946213	0.480612	0.307198
H	-1.277526	-3.301541	-0.130723	-1.270144	-3.302688	-0.129277
C	-0.626505	0.965858	-0.049590	-0.629581	0.969296	-0.048819
C	-0.328529	-0.428685	-0.139213	-0.326773	-0.425667	-0.138216
N	1.692136	1.455353	-0.199231	1.688455	1.464611	-0.199153
C	0.978609	-0.869908	-0.293956	0.980788	-0.865015	-0.293271
C	0.420685	1.876414	-0.135915	0.414681	1.882687	-0.136911
C	1.982659	0.146034	-0.227389	1.985168	0.153640	-0.223667
C	3.889973	-1.292384	0.556536	3.896307	-1.303086	0.530538
C	4.324718	0.912852	-0.359591	4.328424	0.926091	-0.332756
C	5.257834	-0.781022	1.033519	5.262938	-0.801000	1.017983
H	3.227767	-1.546959	1.388111	3.236853	-1.584207	1.354739
H	4.015986	-2.190221	-0.056595	4.024135	-2.178772	-0.111856
C	5.652925	0.217636	-0.061956	5.656237	0.222207	-0.054714
H	4.276114	1.362112	-1.353734	4.278420	1.396375	-1.317200

H	4.124577	1.708128	0.366979	4.136358	1.705522	0.413081
H	5.975937	-1.592205	1.168120	5.980732	-1.615249	1.131195
H	5.153784	-0.260110	1.990109	5.159521	-0.302912	1.986617
H	6.016278	-0.313050	-0.947734	6.015974	-0.288075	-0.953636
H	6.426116	0.920843	0.252725	6.429872	0.918060	0.274054
N	3.333145	-0.171915	-0.237547	3.333228	-0.157859	-0.227658
C	1.302500	-2.300335	-0.648706	1.303367	-2.295778	-0.649035
H	2.207098	-2.344882	-1.255658	2.204286	-2.343186	-1.260723
H	1.455439	-2.943590	0.224479	1.460543	-2.937939	0.223530
H	0.493652	-2.733256	-1.238965	0.491400	-2.729489	-1.233979
C	0.203686	3.367473	-0.148216	0.190948	3.372875	-0.152623
H	-0.470658	3.664734	-0.957391	-0.486059	3.665919	-0.960928
H	-0.243671	3.710410	0.790634	-0.257890	3.716764	0.784948
H	1.159703	3.872522	-0.281189	1.142856	3.885437	-0.287931

Table S3. Vertical transition energies, contributions of excitation configurations, and oscillator strengths, *f*, of the transitions from the ground state to the first singlet excited state for **3bu**, **3bv**, **3bw**, **3bz**, and **3au** calculated at the PCM/TD-B3LYP/6-311G(d,p) level in cyclohexane and acetonitrile.

compound	solvent	transition energy / eV	contribution of excitation configuration	*f*
3bu	cyclohexane	2.739	LUMO ← HOMO (0.69)	0.229
3bv	cyclohexane	2.862	LUMO ← HOMO (0.69)	0.248
3bw	cyclohexane	2.832	LUMO ← HOMO (0.69)	0.237
3bz	cyclohexane	3.230	LUMO ← HOMO (0.68) LUMO ← HOMO−1 (0.16)	0.184
3au	cyclohexane	2.761	LUMO ← HOMO (0.70)	0.239
3bu	acetonitrile	2.729	LUMO ← HOMO (0.69)	0.229
3bv	acetonitrile	2.881	LUMO ← HOMO (0.69)	0.246
3bw	acetonitrile	2.844	LUMO ← HOMO (0.69)	0.235
3bz	acetonitrile	3.266	LUMO ← HOMO (0.68) LUMO ← HOMO−1 (0.16)	0.185
3au	acetonitrile	2.753	LUMO ← HOMO (0.70)	0.242

12. Procedure for Cellular Experiments

Cell culture.

A human breast adenocarcinoma cell line, SK-BR-3, was cultured in Dulbecco's Modified Eagle Medium (D-MEM) supplemented with 10 % fetal bovine serum (FBS), 1 % penicillin-streptomycin. The cells were maintained at 37 ˚C in 5% CO_2 / 95% air and kept in a logarithmic growth phase by routine passages every 2−3 days. Prior to the use of cells, the densities of cells were determined using a hemocytometer.

Cytotoxicity evaluation.

SK-BR-3 cells (5.0×10^3 cells per well) were seeded onto 96-well plates in D-MEM, and maintained at 37 ˚C in a 5% CO_2 / 95% air incubator for 24 h. After incubation, the cells were incubated in the medium containing 10 μM azafluoranthenes and 1% DMSO for 24 h. Next, 10 μL of Cell Counting Kit-8 solution (Dojindo) was added, and the plate was further incubated at 37 ˚C. After incubation for 210 min, absorbance at 450 nm was recorded on a microplate absorbance spectrophotometer to estimate the cytotoxic effects of azafluoranthenes.

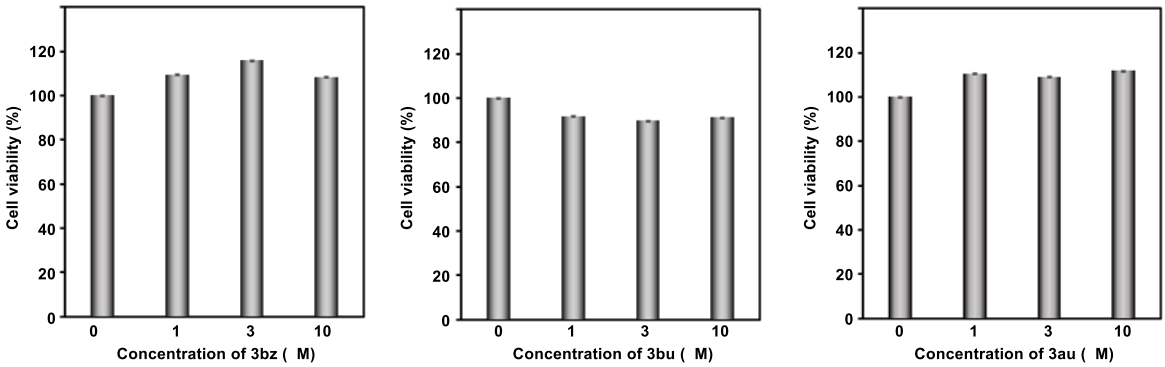


Figure S25. Cytotoxicity of **3bz**, **3bu** and **3au** against SK-BR-3 cells. SK-BR-3 cells were cultured in the presence or absence of azafluoranthenes at the designated concentrations for 24 h at 37 °C. Results are shown as the mean ± S.E. (n = 5).

Cellular imaging and cellular localization of azafluoranthenes.

SK-BR-3 cells (5×10^3 cells) were plated in 35 mm glass-bottom dishes and cultured for 24 h. Azafluoranthenes were then added to the medium containing 1% DMSO at a final concentration of 10 μM and incubated for 1 h. After the cells were washed, imaging by microscopy was conducted. Fluorescent images of azafluoranthenes in cells were acquired using excitation at 405 or 488 nm and a 526–575 nm band-pass filter for

emission.

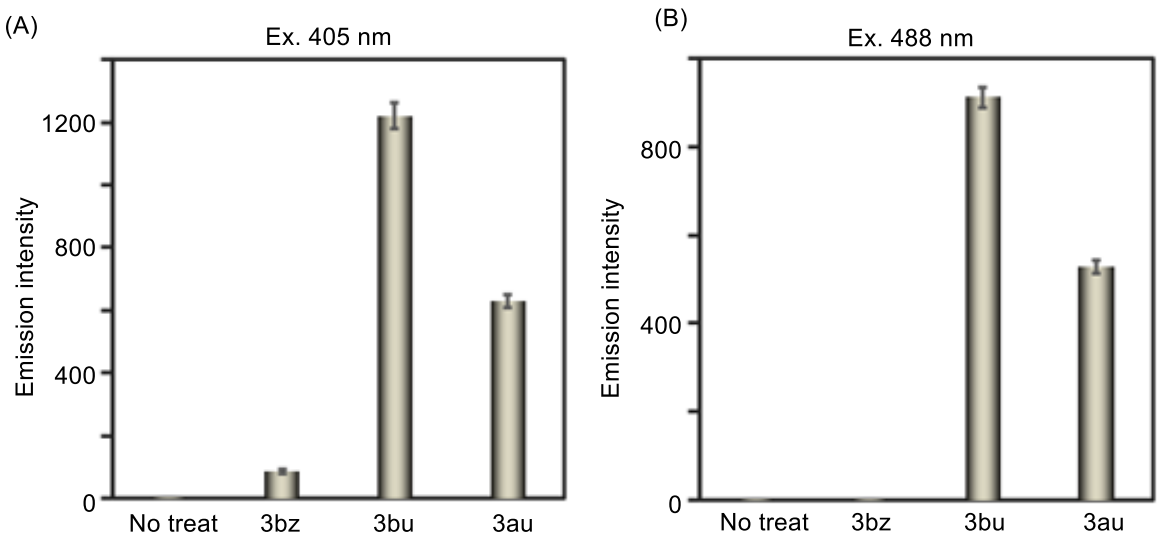


Figure S26. Emission intensity obtained from each cell. The fluorescence intensity was obtained from Figure 4A (A: Excitation at 405 nm) and 4B (B: Excitation at 488 nm).

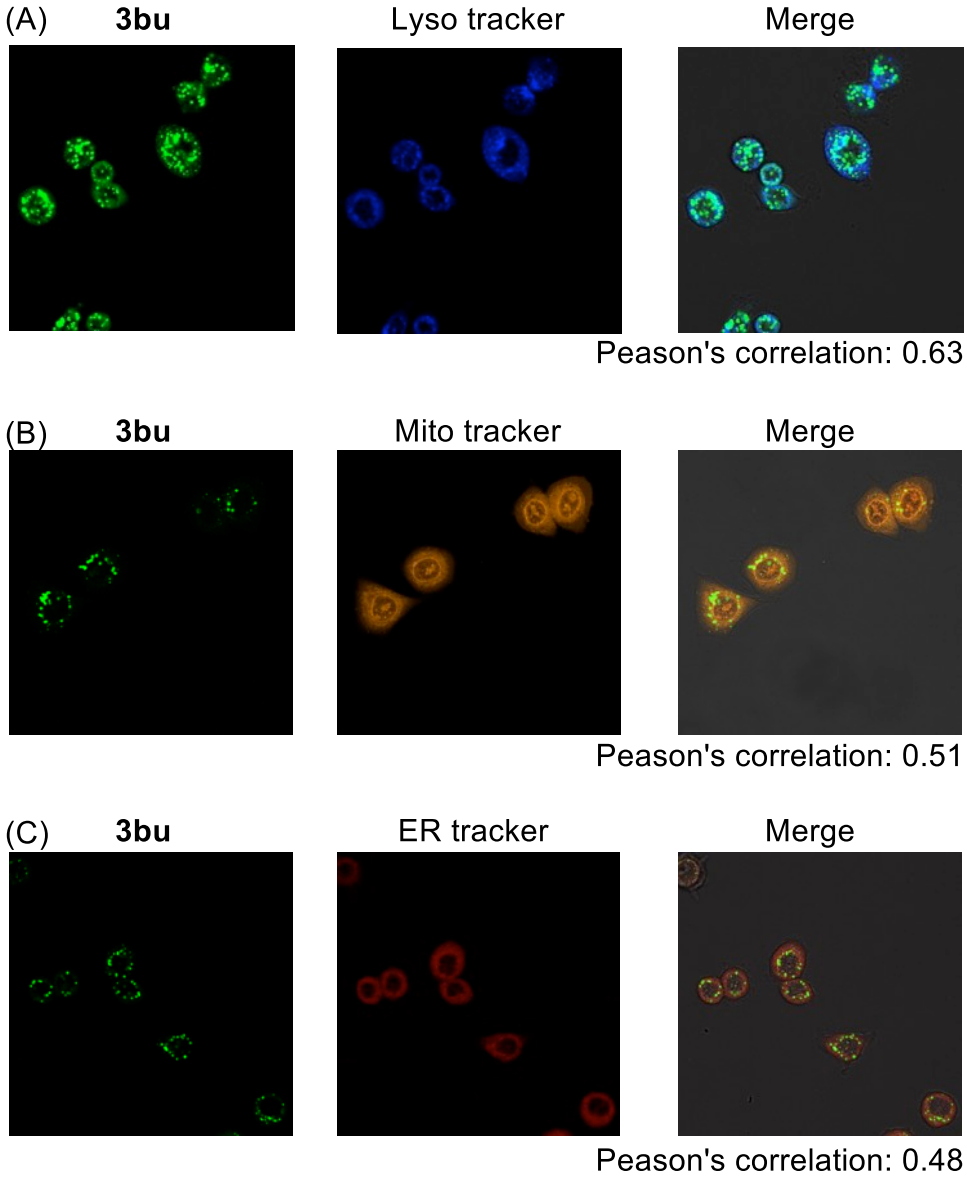


Figure S27. Localization of **3bu** in SK-BR-3 cells. The cells were incubated with **3bu** and then lysosome (A), mitochondria (B) and ER (C) were stained by organelle markers, Lyso tracker, Mito tracker and ER tracker, respectively. Right, merged pictures.

13. References

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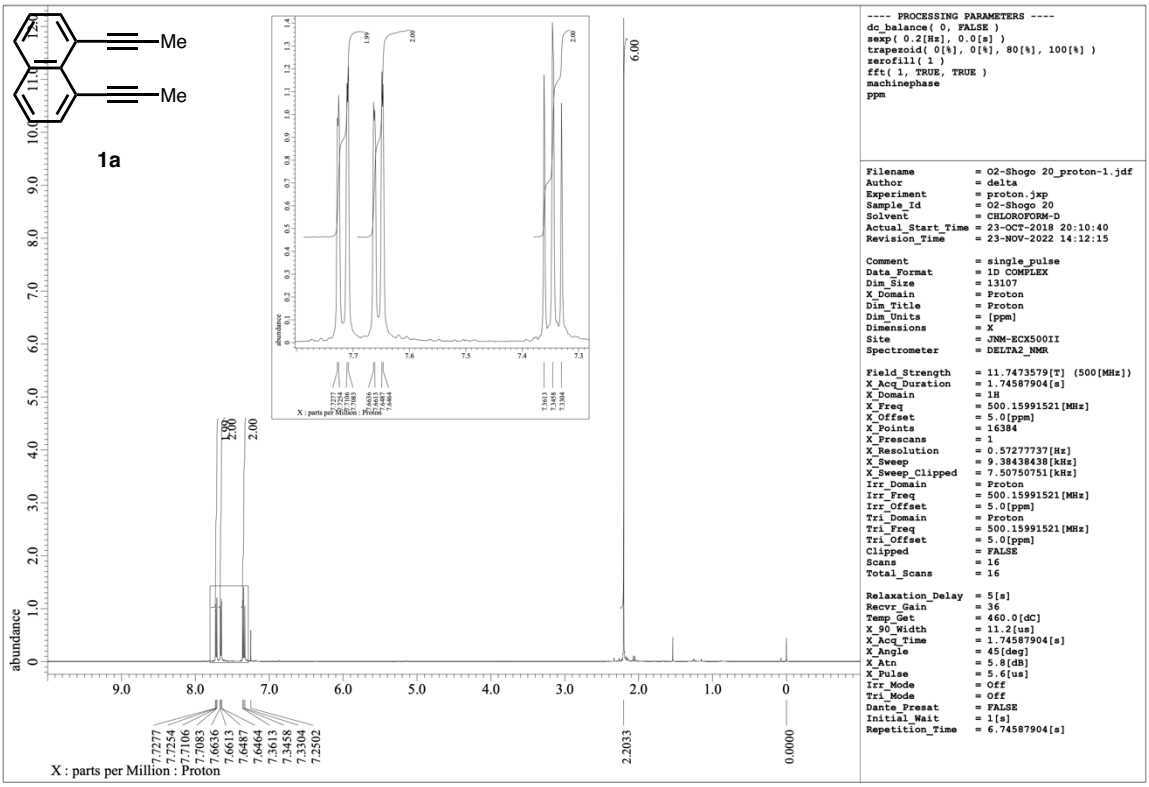
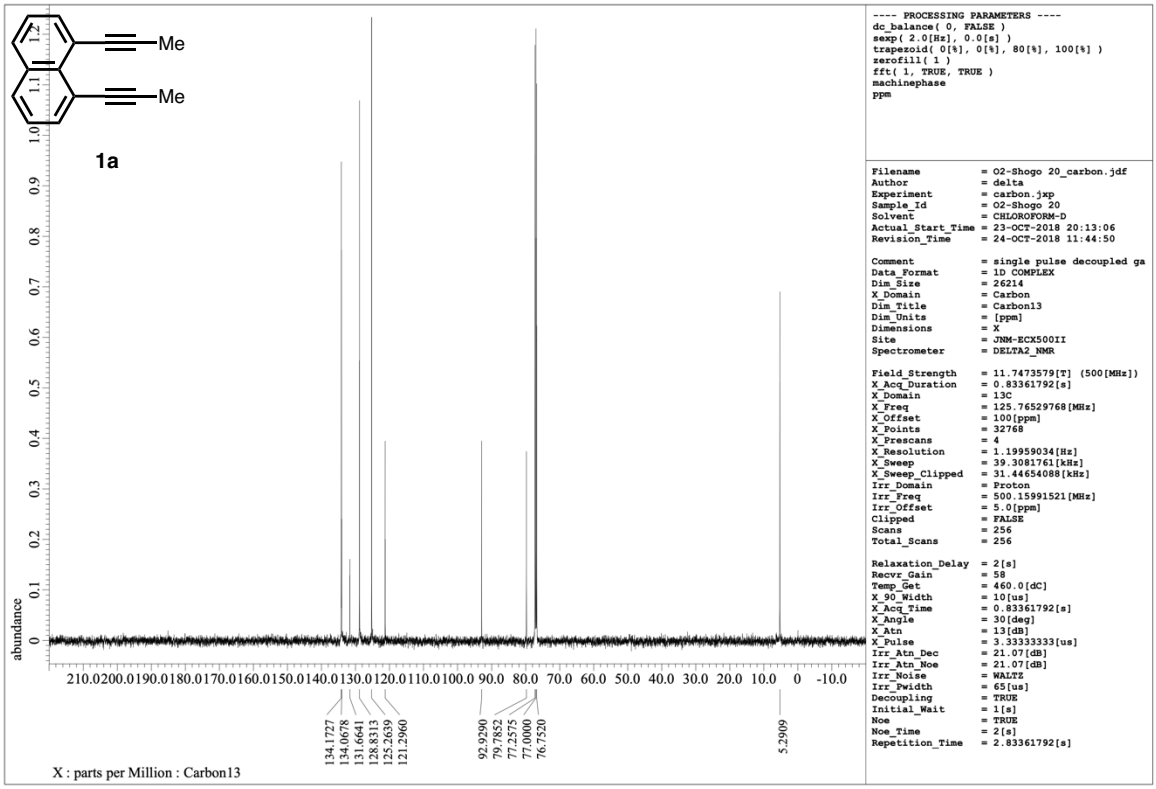
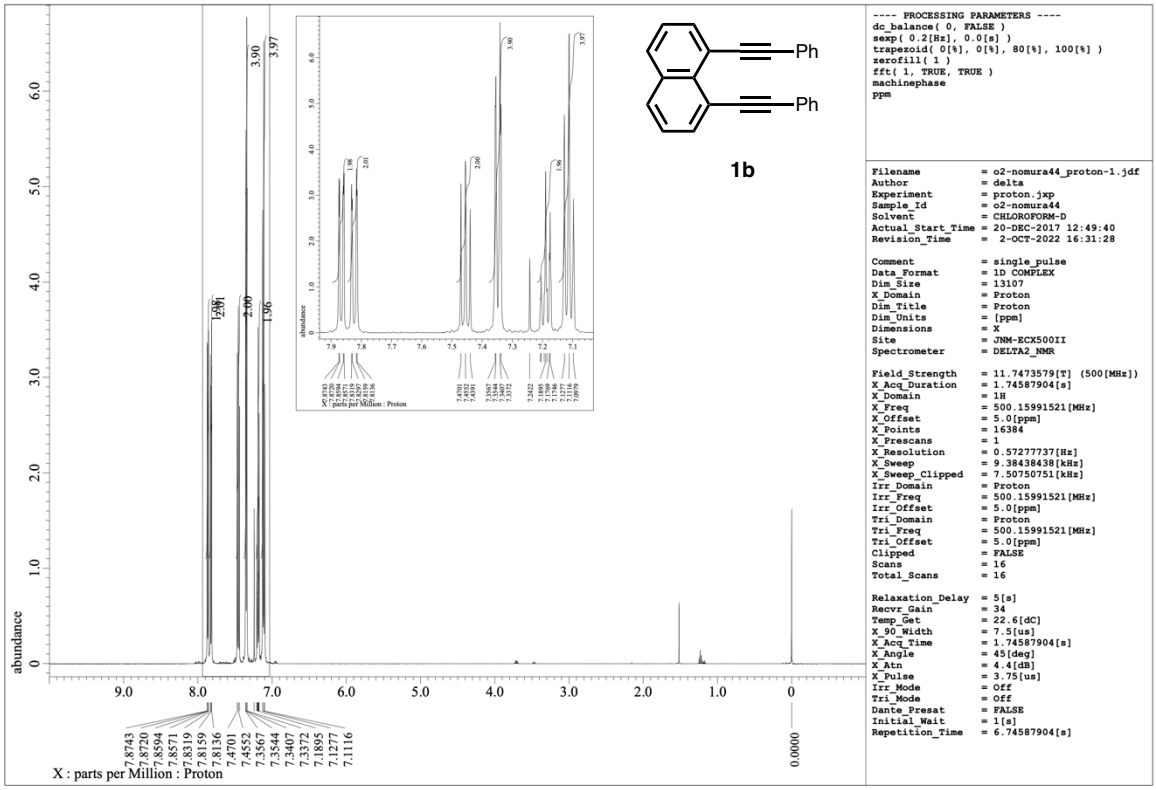
14. ^{1}H, ^{13}C, and ^{19}F NMR Charts





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machinephase
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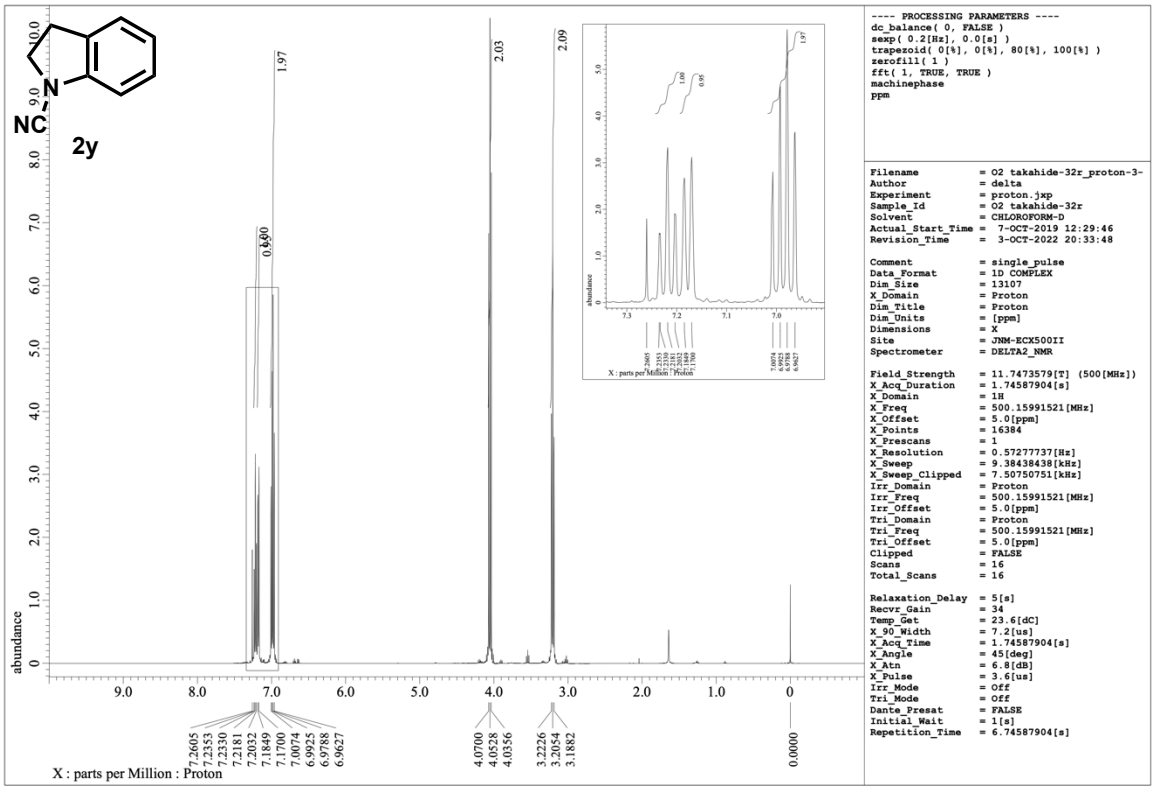


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machinephase
ppm

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Experiment          = proton.jxp
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Revision_Time       =  3-OCT-2022 20:33:48

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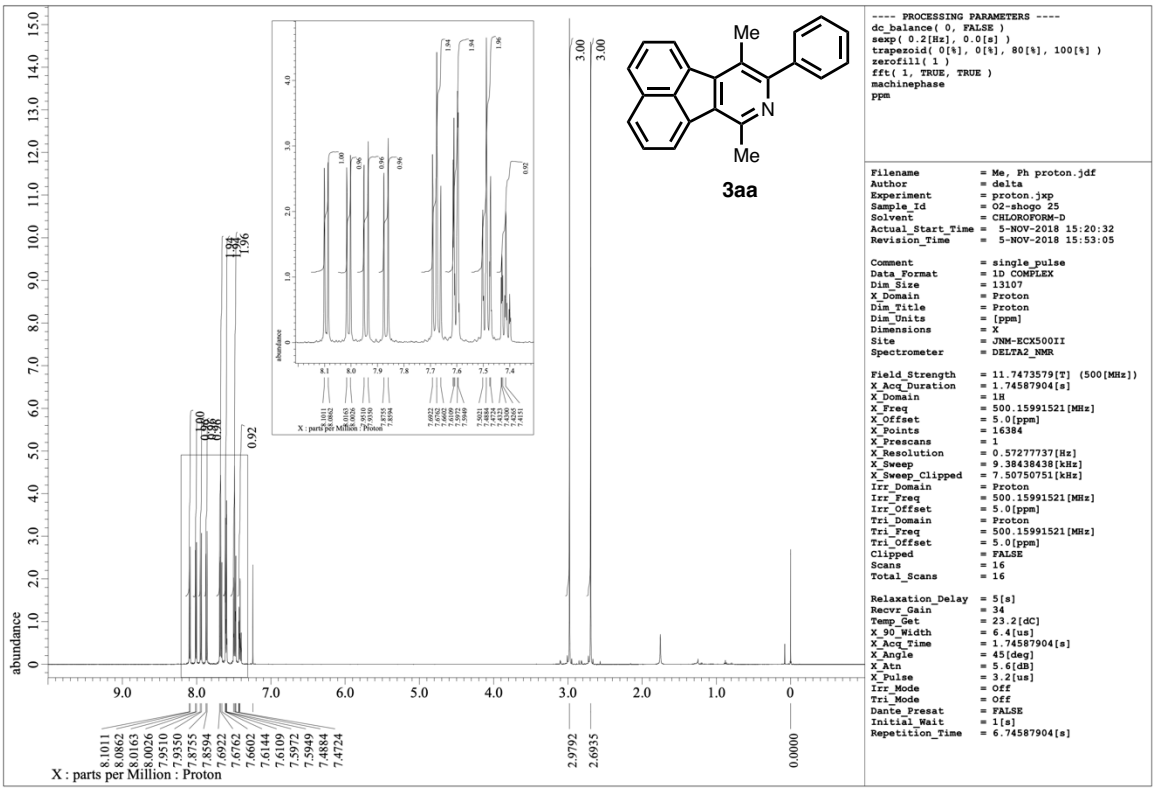
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3aa
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X : parts per Million : Proton
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Recvr_Gain = 34
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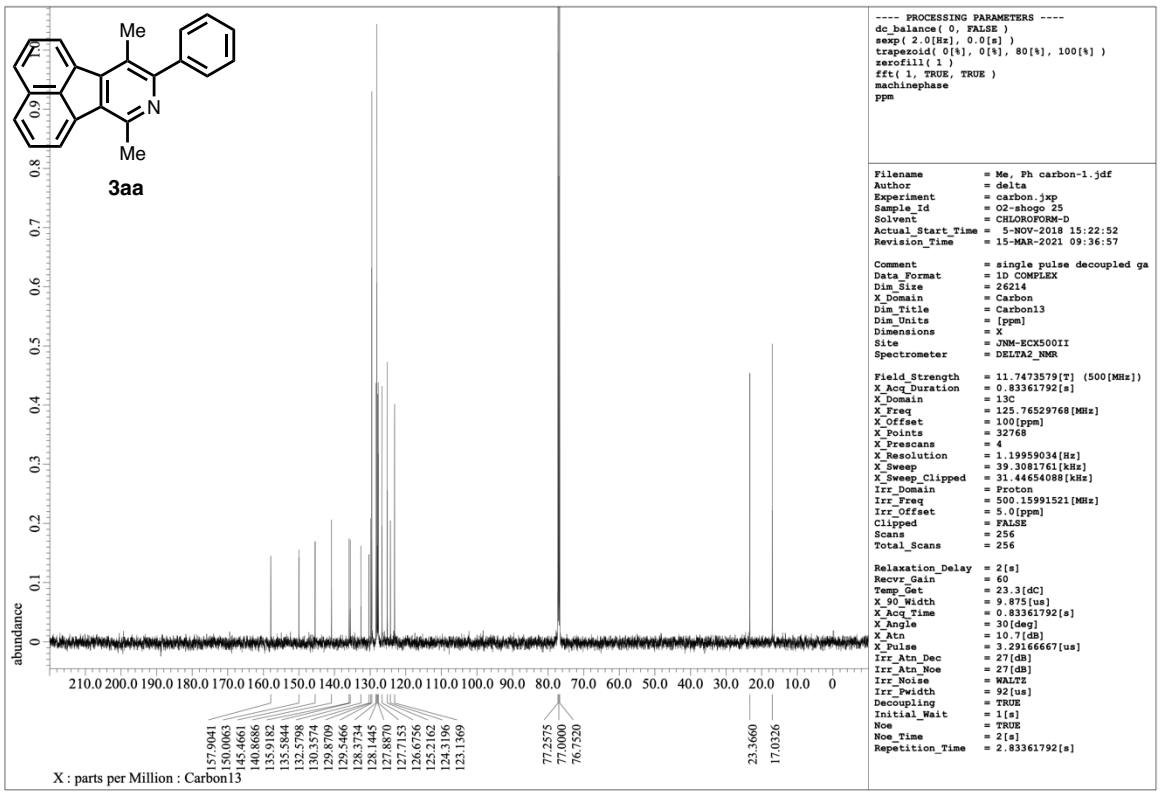
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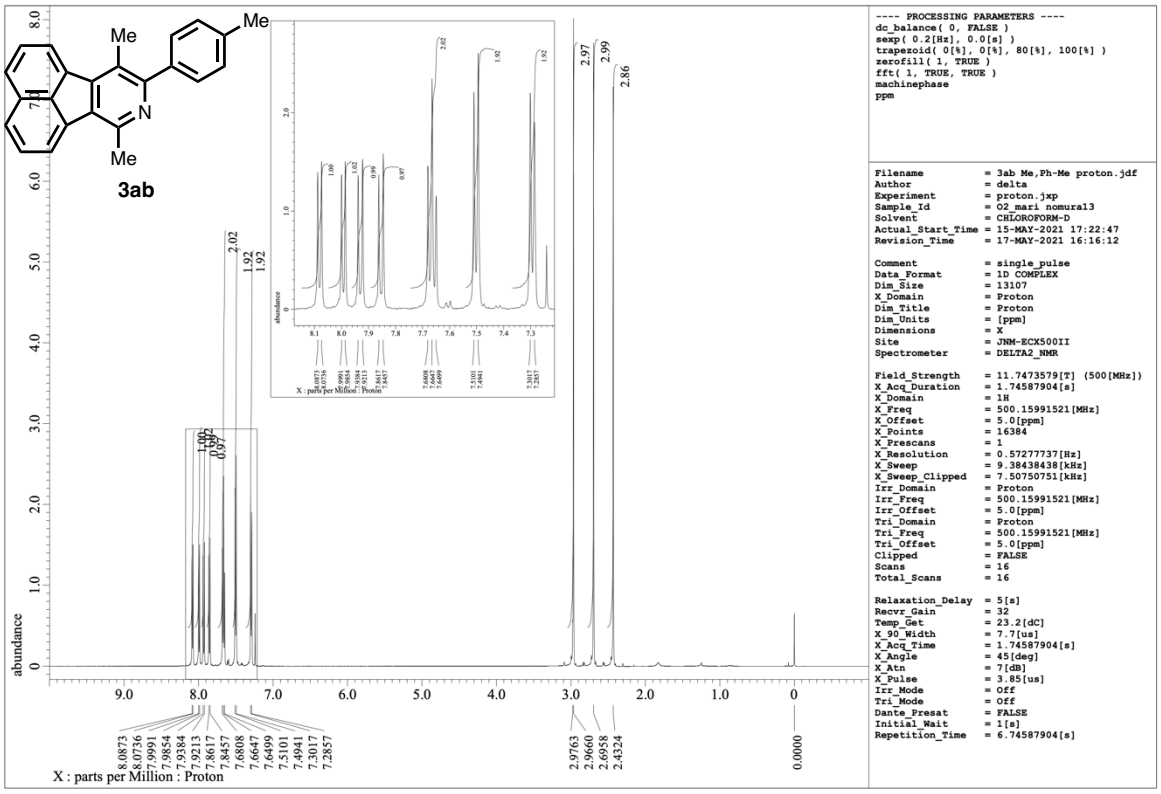
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N
Me
3ab
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X : parts per Million : Proton
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Clipped = FALSE
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Relaxation_Delay = 5[s]
Recvr_Gain = 32
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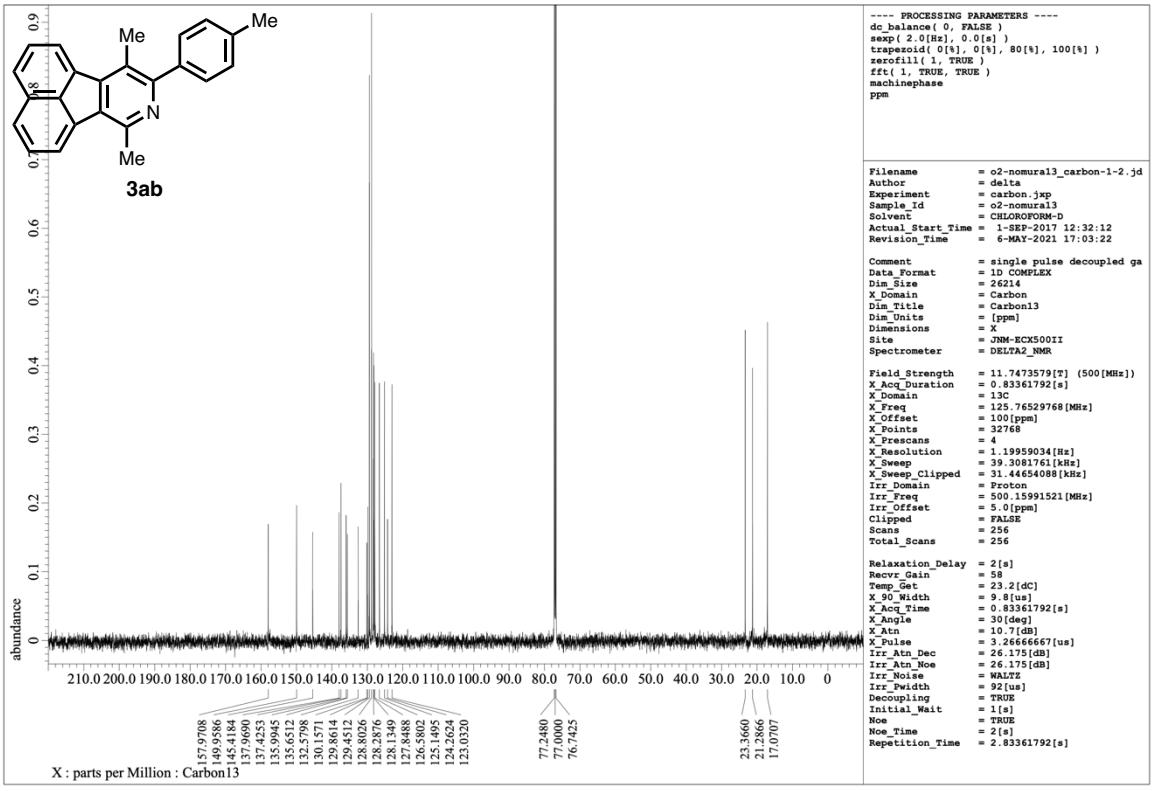
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Me
N
Me
3ab
abundance
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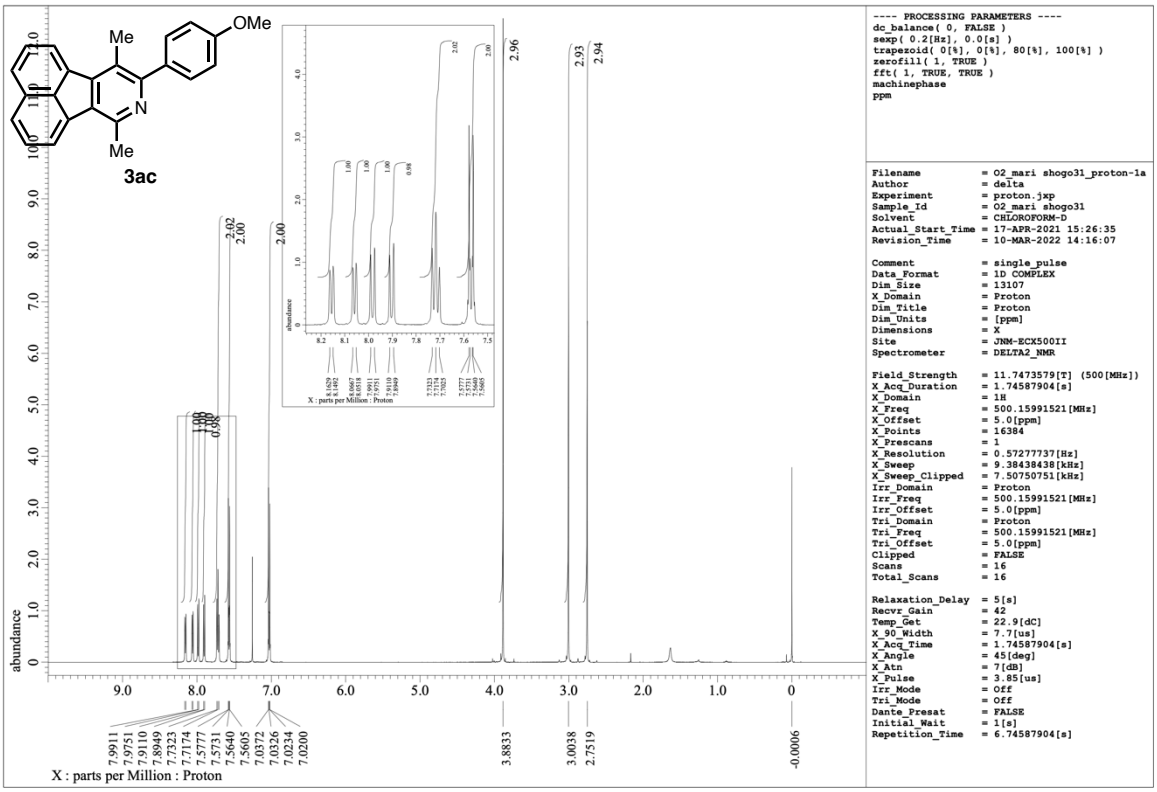
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OMe
N
Me
3ac
X : parts per Million : Proton
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Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Tri_Domain = Proton
Tri_Freq = 500.15991521[MHz]
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X_90_Width = 7.7[us]
X_Acq_Time = 1.74587904[s]
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X_Atn = 7[dB]
X_Pulse = 3.85[us]
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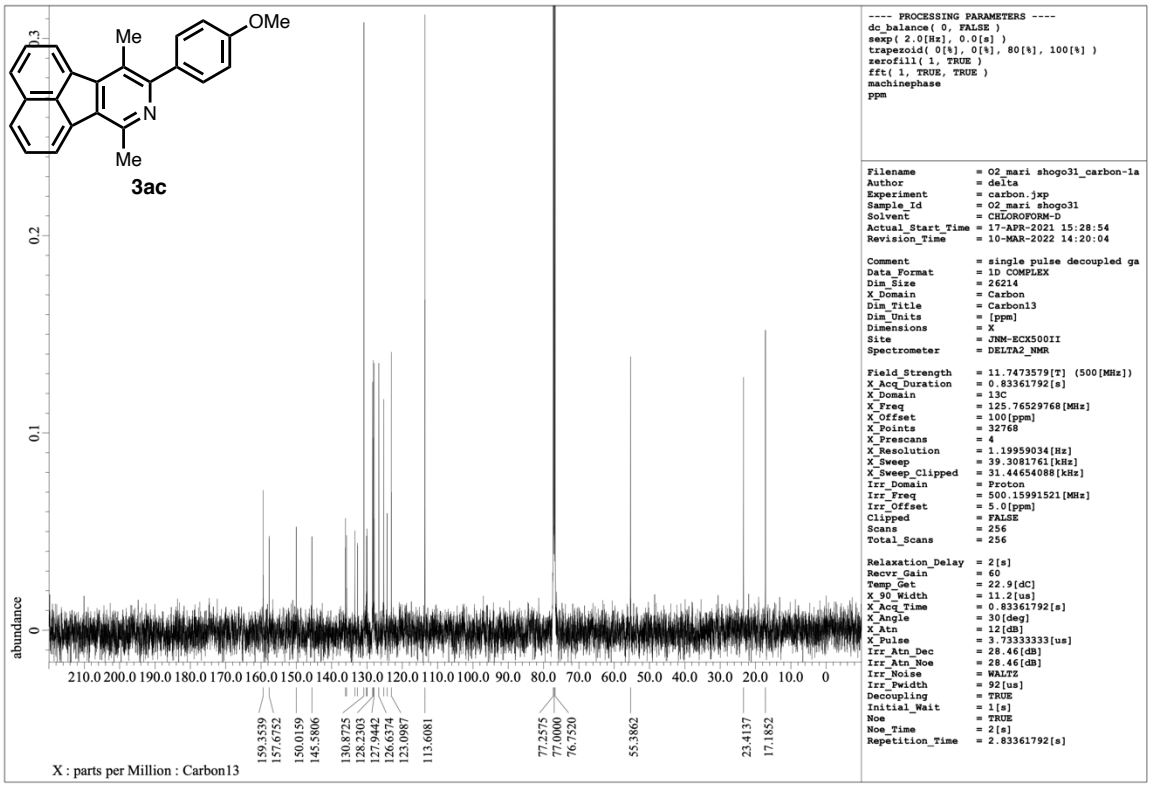
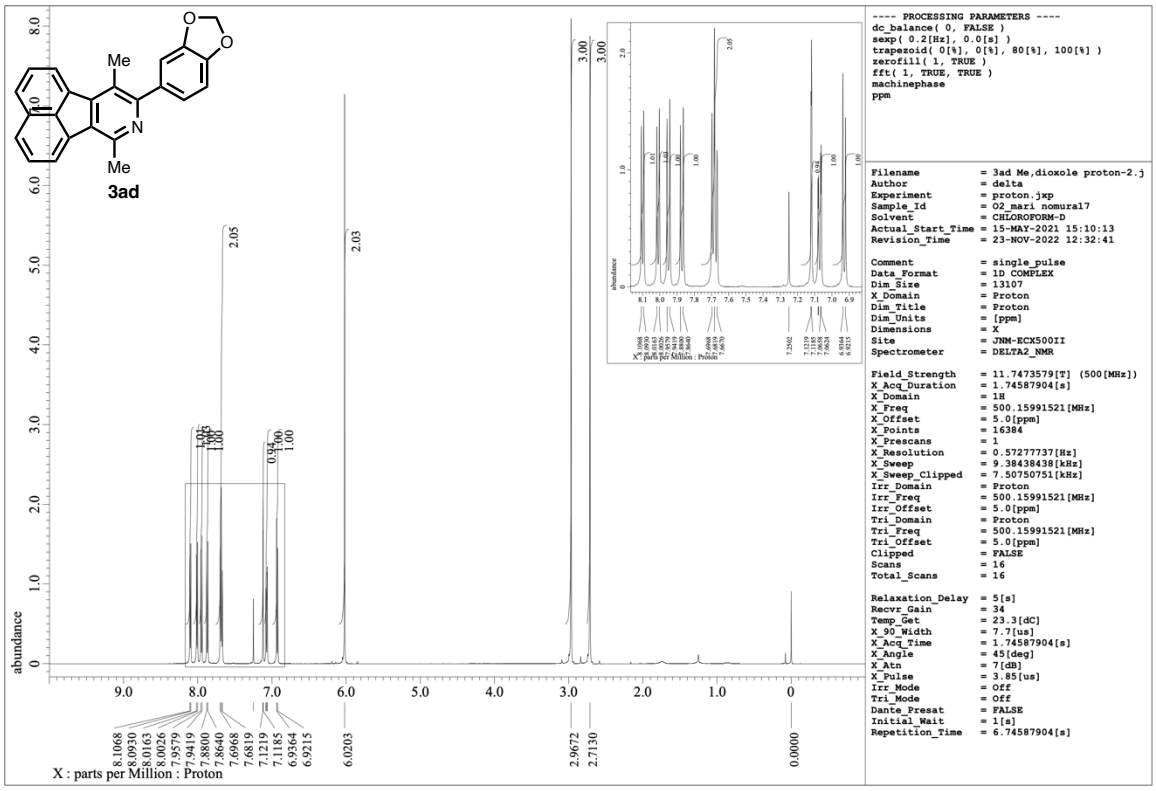
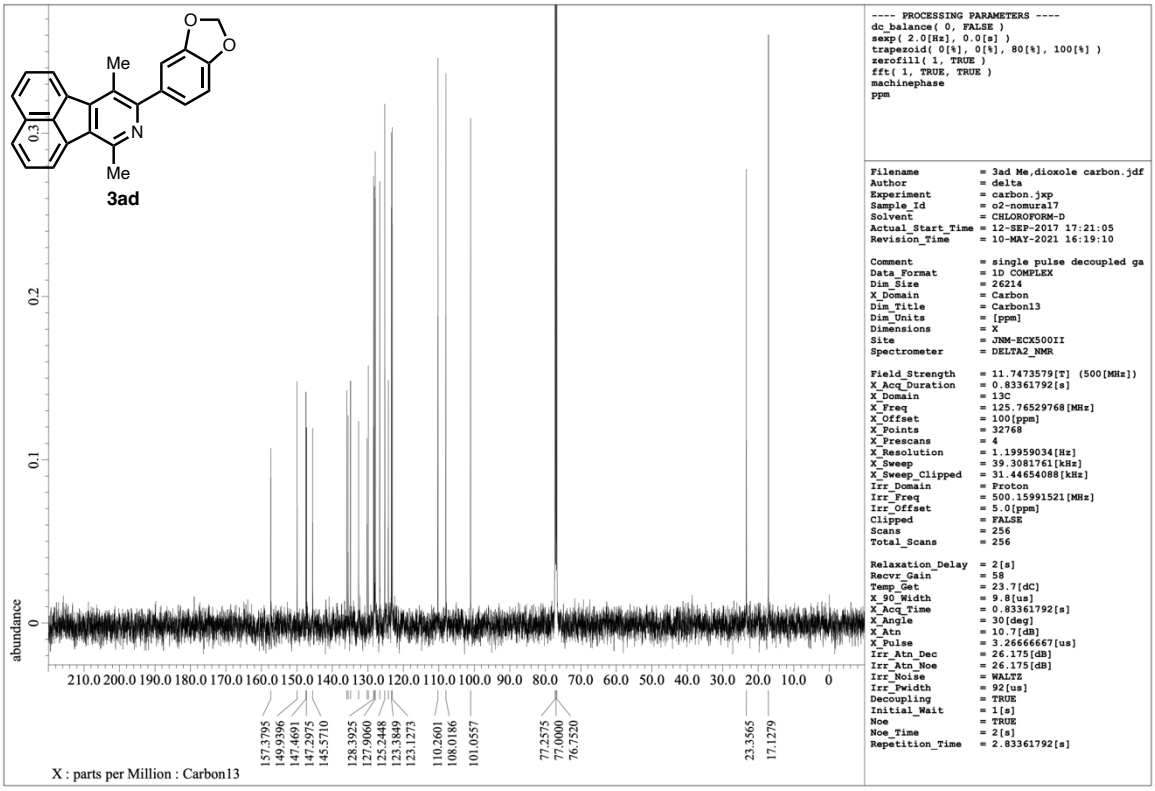
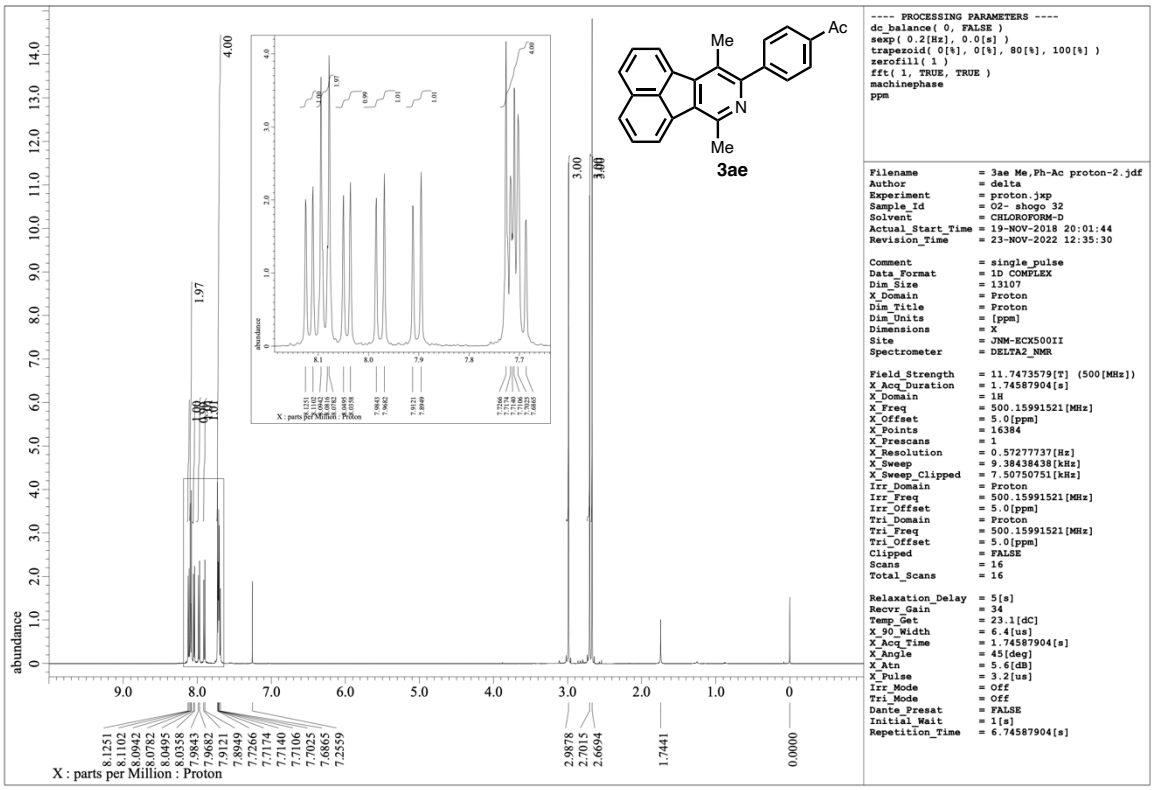
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

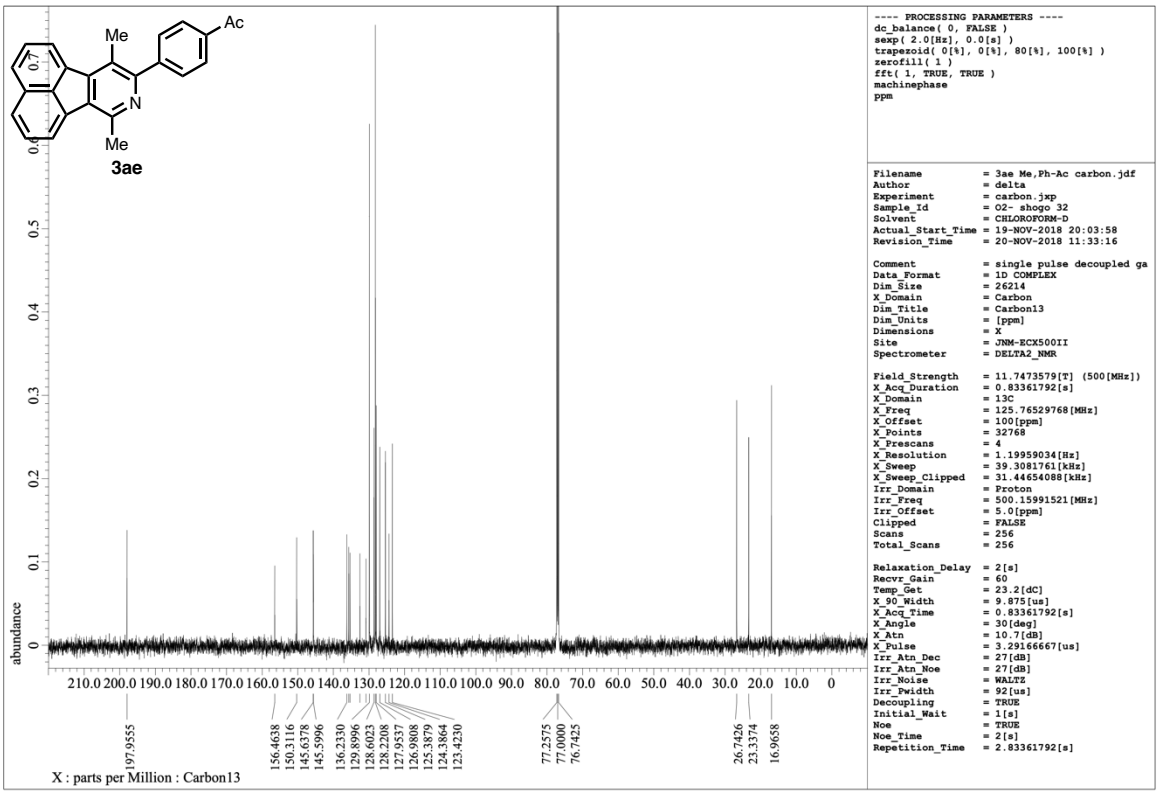

Me
OMe
N
Me
3ac
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari shogo31_carbon-1a
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari shogo31
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-APR-2021 15:28:54
Revision_Time = 10-MAR-2022 14:20:04
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 = 22.9[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]


Me
N
Me
3ad
2.05
2.03
3.00
3.00
0.94
1.00
1.00
abundance
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.1068
8.0930
8.0163
8.0026
7.9579
7.9419
7.8800
7.8640
7.6968
7.6819
7.1219
7.1185
6.9364
6.9215
6.0203
2.9672
2.7130
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 = 3ad Me,dioxole proton-2.j
Author = delta
Experiment = proton.jxp
Sample_Id = 02_mari nomural7
Solvent = CHLOROFORM-D
Actual_Start_Time = 15-MAY-2021 15:10:13
Revision_Time = 23-NOV-2022 12:32:41
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 = 23.3[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]

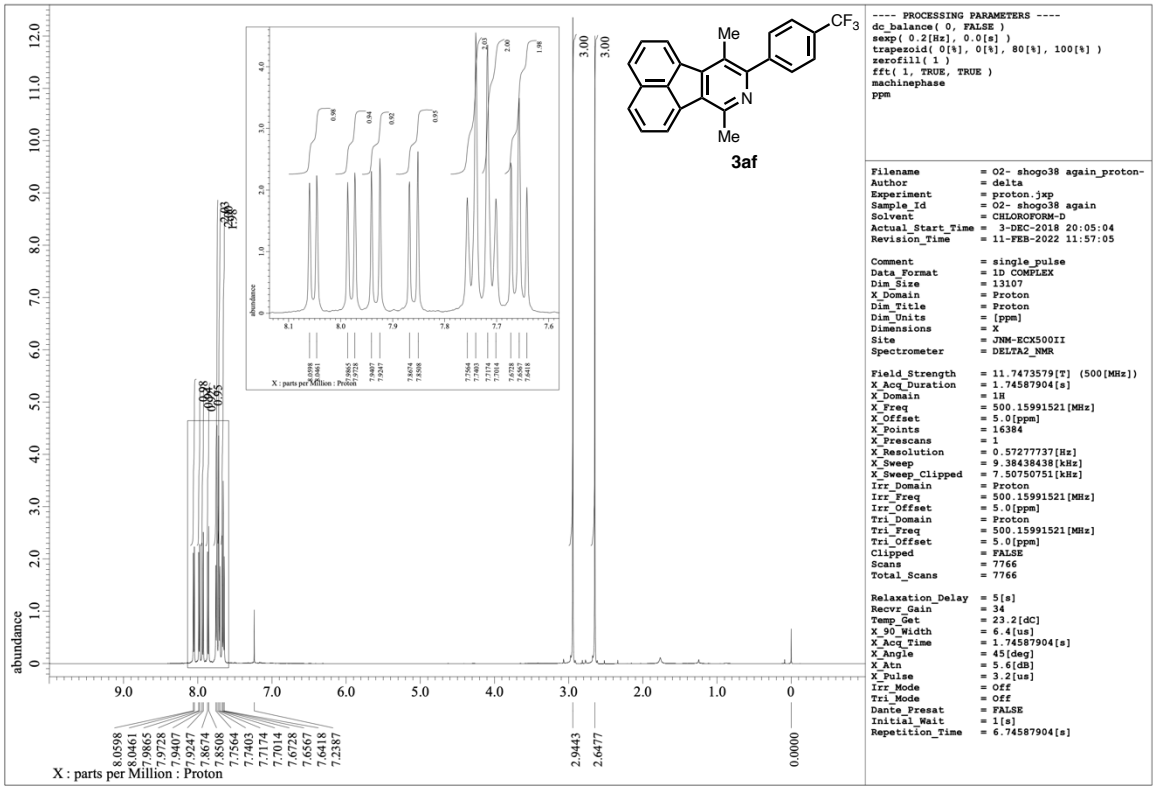

Me
N
Me
3ad
abundance
0.3
0.2
0.1
0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
157.3795
149.9396
147.4691
147.2975
145.5710
128.3925
127.9060
125.2448
123.3849
123.1273
110.2601
108.0186
101.0557
77.2575
77.0000
76.7520
23.3565
17.1279
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 = 3ad Me,dioxole carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-nomural7
Solvent = CHLOROFORM-D
Actual_Start_Time = 12-SEP-2017 17:21:05
Revision_Time = 10-MAY-2021 16:19:10
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 = 23.7[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]



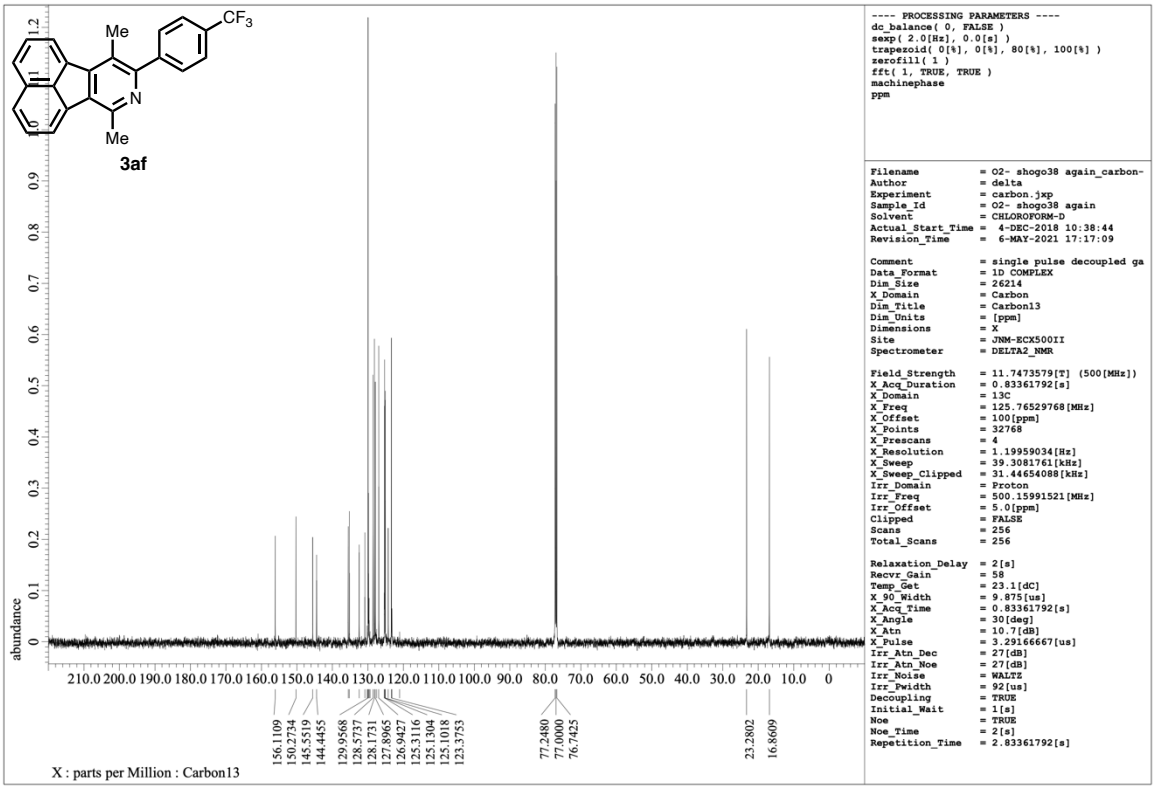

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = 3ae Me,Ph-Ac proton-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = O2- shogo 32
Solvent           = CHLOROFORM-D
Actual_Start_Time = 19-NOV-2018 20:01:44
Revision_Time     = 23-NOV-2022 12:35:30

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          = 23.1[dC]
X_90_Width        = 6.4[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 5.6[dB]
X_Pulse           = 3.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



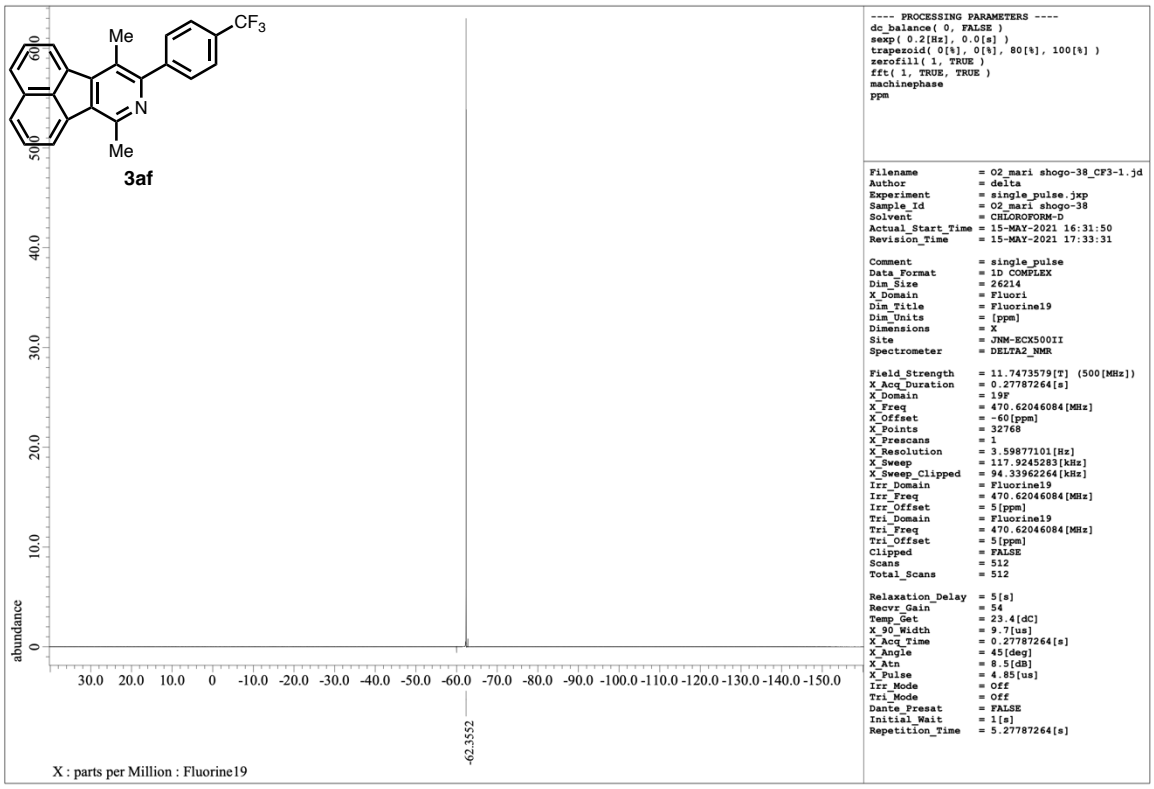

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = 3ae Me,Ph-Ac carbon.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = O2- shogo 32
Solvent           = CHLOROFORM-D
Actual_Start_Time = 19-NOV-2018 20:03:58
Revision_Time     = 20-NOV-2018 11:33:16

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          = 23.2[dC]
X_90_Width        = 9.875[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 10.7[dB]
X_Pulse           = 3.29166667[us]
Irr_Atn_Dec       = 27[dB]
Irr_Atn_Noe       = 27[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]
```

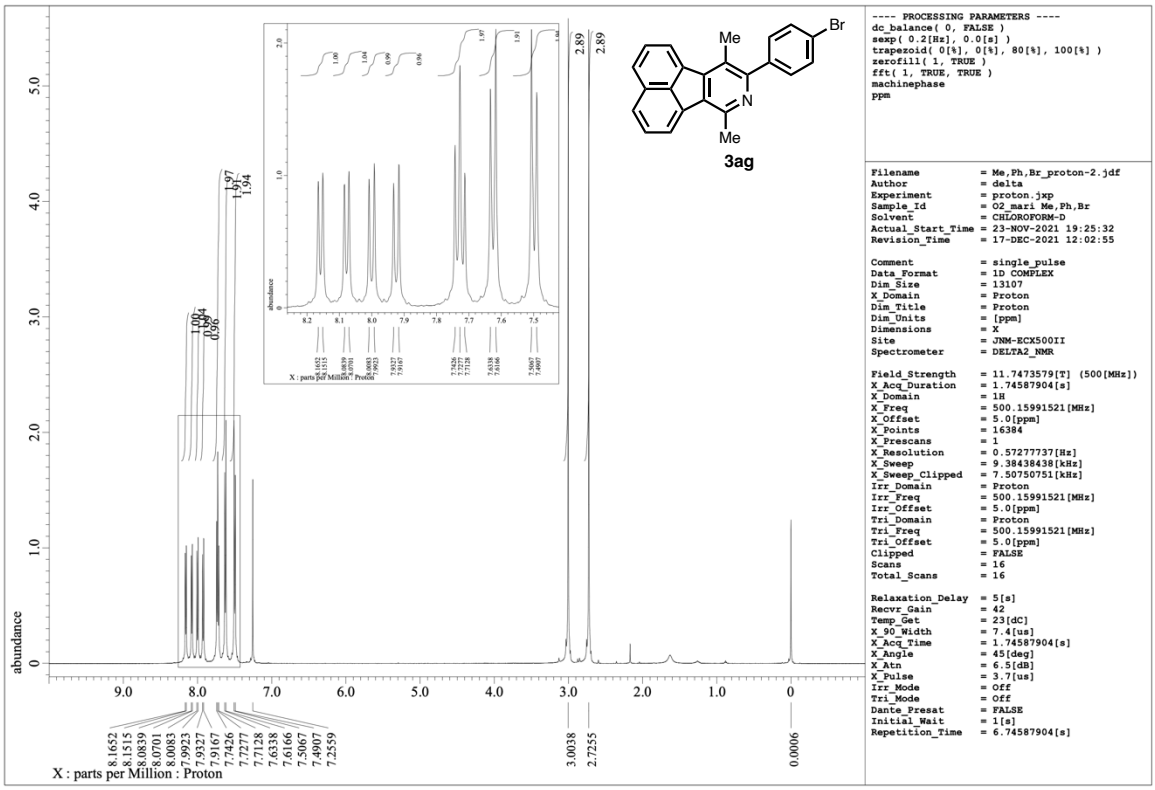



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = O2- shogo38 again_proton-
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2- shogo38 again
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  3-DEC-2018 20:05:04
Revision_Time       = 11-FEB-2022 11:57:05

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               = 7766
Total_Scans         = 7766

Relaxation_Delay    = 5[s]
Recvr_Gain          = 34
Temp_Get            = 23.2[dC]
X_90_Width          = 6.4[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 5.6[dB]
X_Pulse             = 3.2[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

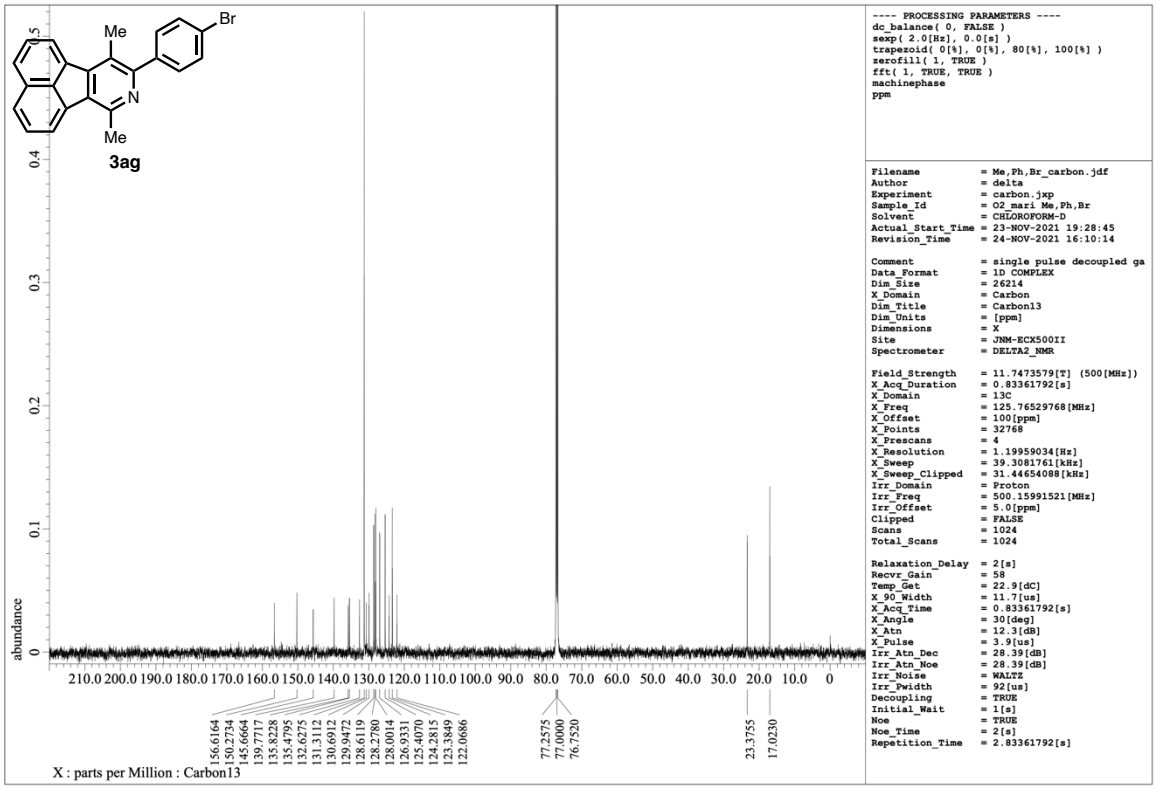



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = O2- shogo38 again_carbon-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2- shogo38 again
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-DEC-2018 10:38:44
Revision_Time       =  6-MAY-2021 17:17:09

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          = 58
Temp_Get            = 23.1[dC]
X_90_Width          = 9.875[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.29166667[us]
Irr_Atn_Dec         = 27[dB]
Irr_Atn_Noe         = 27[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]
```

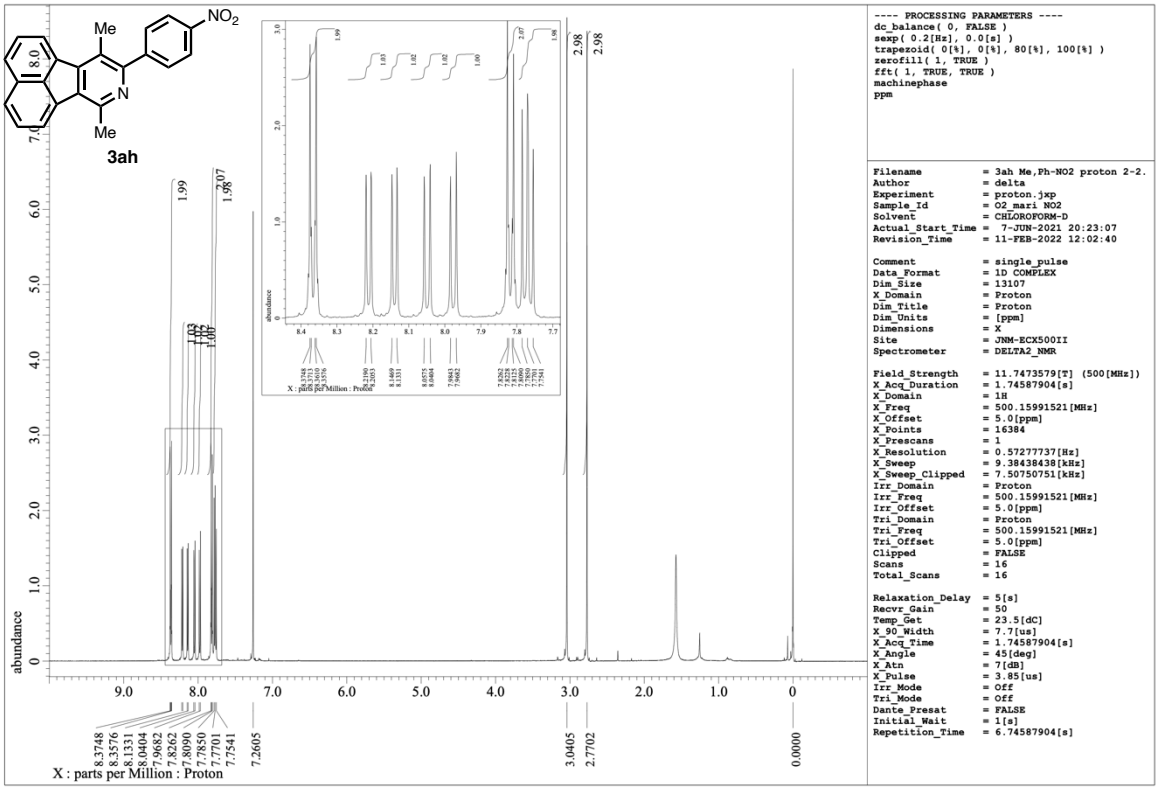

Me
CF3
N
Me
3af
abundance
60.0
50.0
40.0
30.0
20.0
10.0
0
30.0 20.0 10.0 0 -10.0 -20.0 -30.0 -40.0 -50.0 -60.0 -70.0 -80.0 -90.0 -100.0 -110.0 -120.0 -130.0 -140.0 -150.0
-62.3552
X : parts per Million : Fluorine19
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 02_mari shogo-38_CF3-1.jd
Author = delta
Experiment = single_pulse.jxp
Sample_Id = 02_mari shogo-38
Solvent = CHLOROFORM-D
Actual_Start_Time = 15-MAY-2021 16:31:50
Revision_Time = 15-MAY-2021 17:33:31
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 = -60[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 = 512
Total_Scans = 512
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 23.4[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]

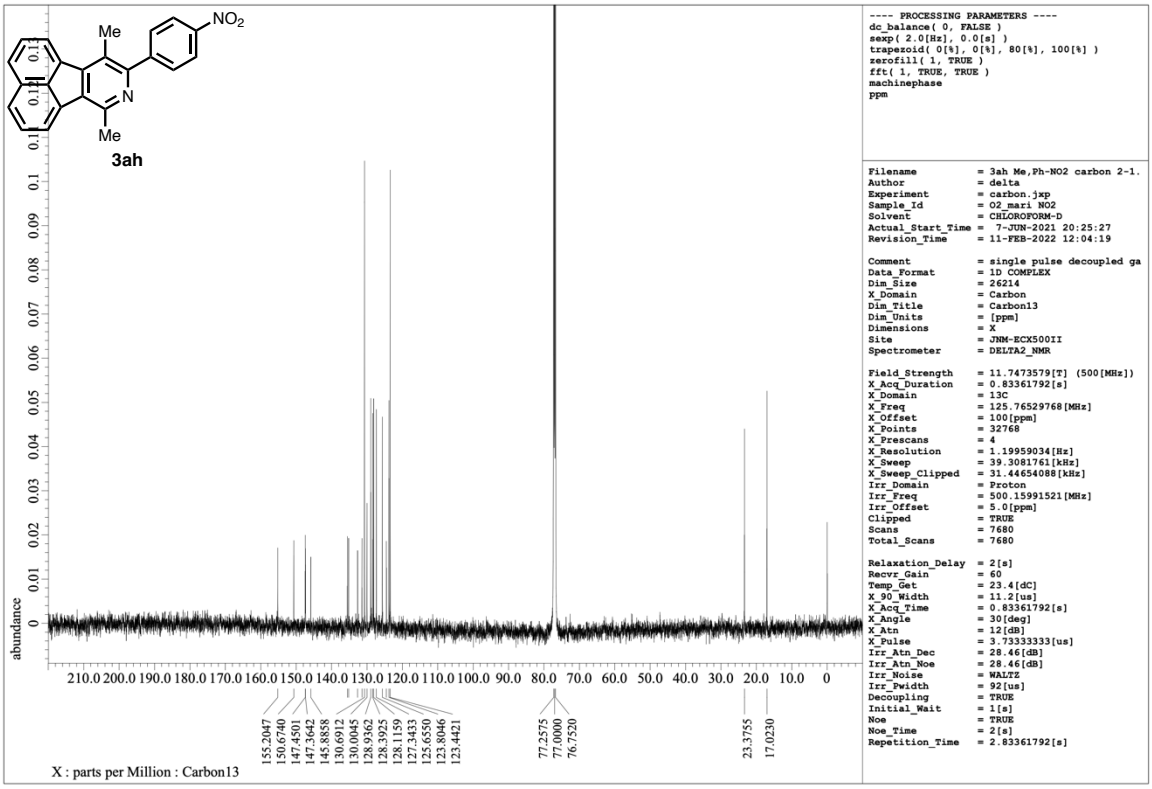


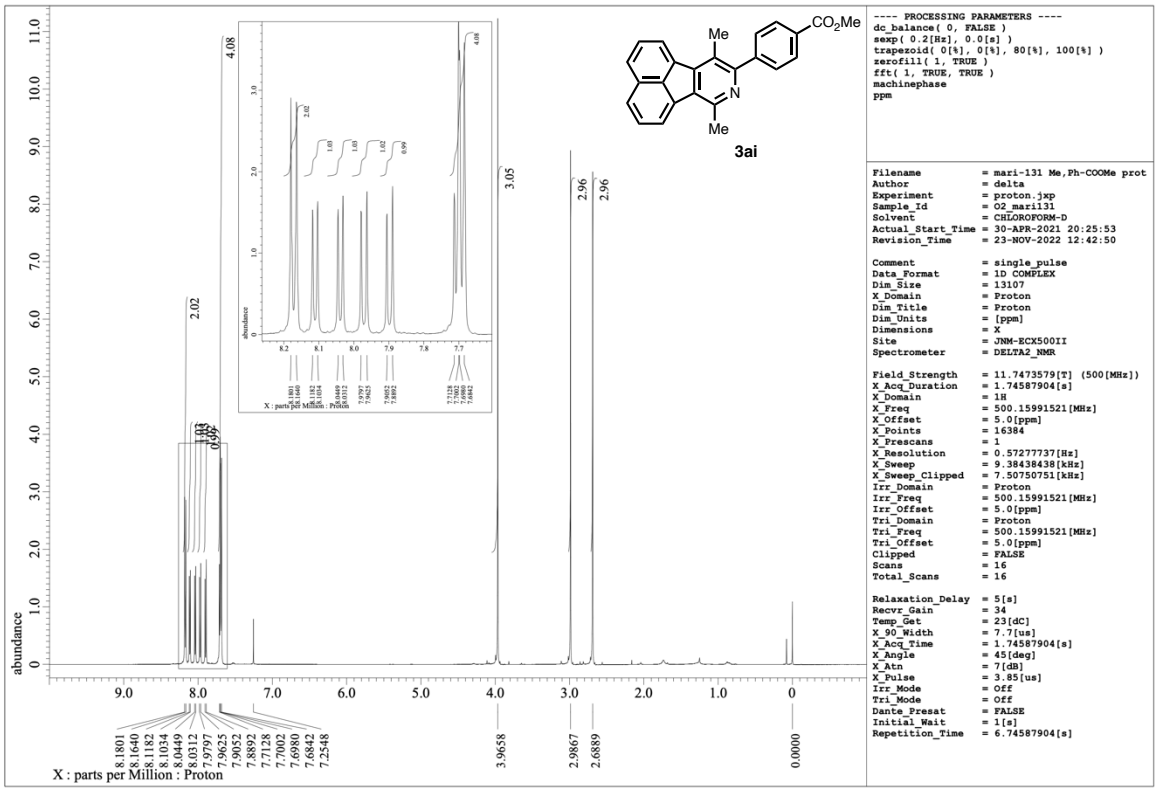

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = Me,Ph,Br_proton-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = O2_mari Me,Ph,Br
Solvent           = CHLOROFORM-D
Actual_Start_Time = 23-NOV-2021 19:25:32
Revision_Time     = 17-DEC-2021 12:02:55

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        = 42
Temp_Get          = 23[dC]
X_90_Width        = 7.4[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 3.7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



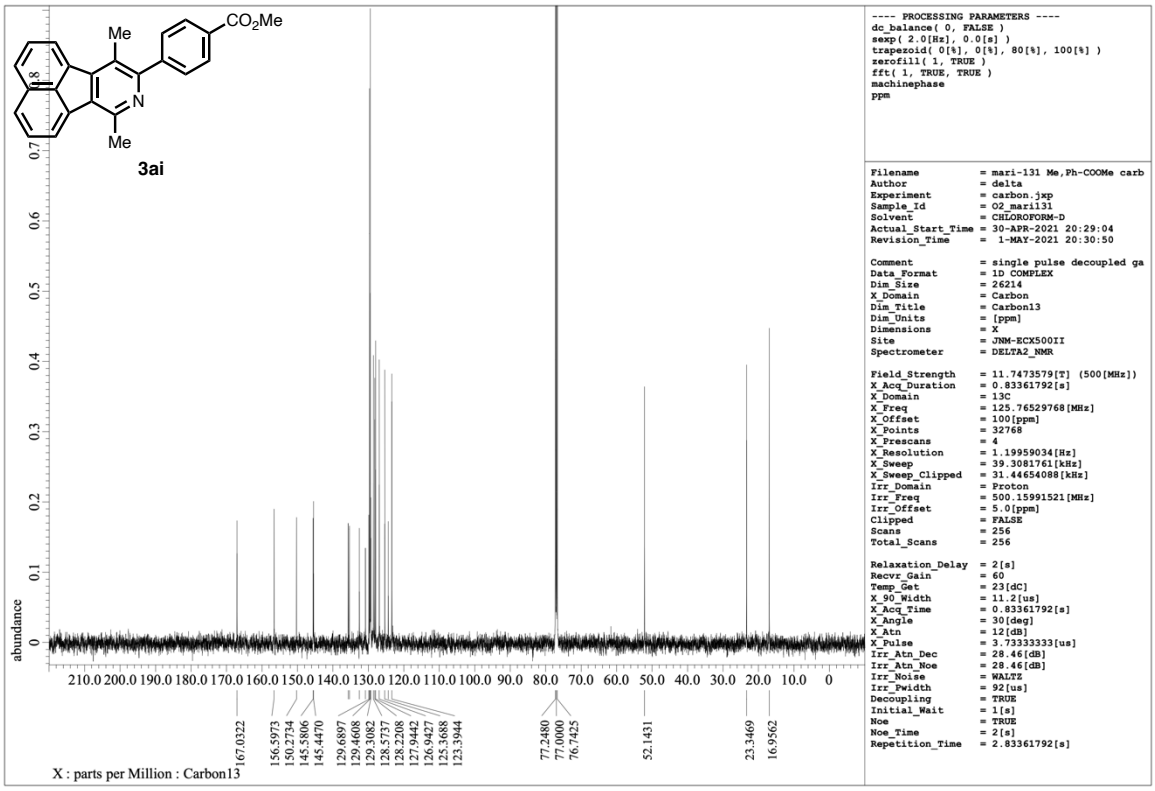

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = Me,Ph,Br_carbon.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = O2_mari Me,Ph,Br
Solvent           = CHLOROFORM-D
Actual_Start_Time = 23-NOV-2021 19:28:45
Revision_Time     = 24-NOV-2021 16:10:14

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             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 22.9[dC]
X_90_Width        = 11.7[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 12.3[dB]
X_Pulse           = 3.9[us]
Irr_Atn_Dec       = 28.39[dB]
Irr_Atn_Noe       = 28.39[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]
```


Me
NO2
N
Me
3ah
abundance
X : parts per Million : Proton
8.3748
8.3576
8.1331
8.0404
7.9682
7.8262
7.8090
7.7850
7.7701
7.7541
7.2605
3.0405
2.7702
0.0000
---- 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 = 3ah Me,Ph-NO2 proton 2-2.
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari NO2
Solvent = CHLOROFORM-D
Actual_Start_Time = 7-JUN-2021 20:23:07
Revision_Time = 11-FEB-2022 12:02:40
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 = 50
Temp_Get = 23.5[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]

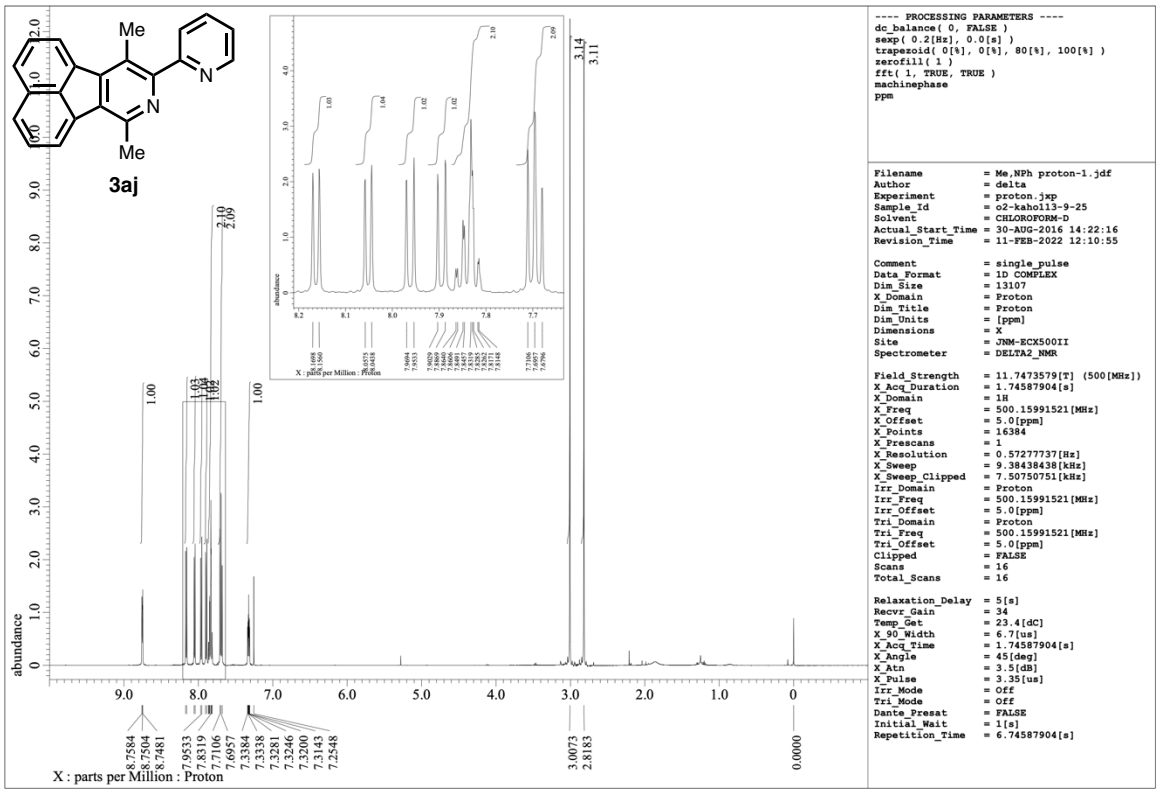

Me
NO2
N
Me
3ah
abundance
X : parts per Million : Carbon13
155.2047
150.6740
147.4501
147.3642
145.8858
130.6912
130.0045
128.9362
128.3925
128.1159
127.3433
125.6550
123.8046
123.4421
77.2575
77.0000
76.7520
23.3755
17.0230
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 3ah Me,Ph-NO2 carbon 2-1.
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari NO2
Solvent = CHLOROFORM-D
Actual_Start_Time = 7-JUN-2021 20:25:27
Revision_Time = 11-FEB-2022 12:04:19
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 = TRUE
Scans = 7680
Total_Scans = 7680
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.4[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]

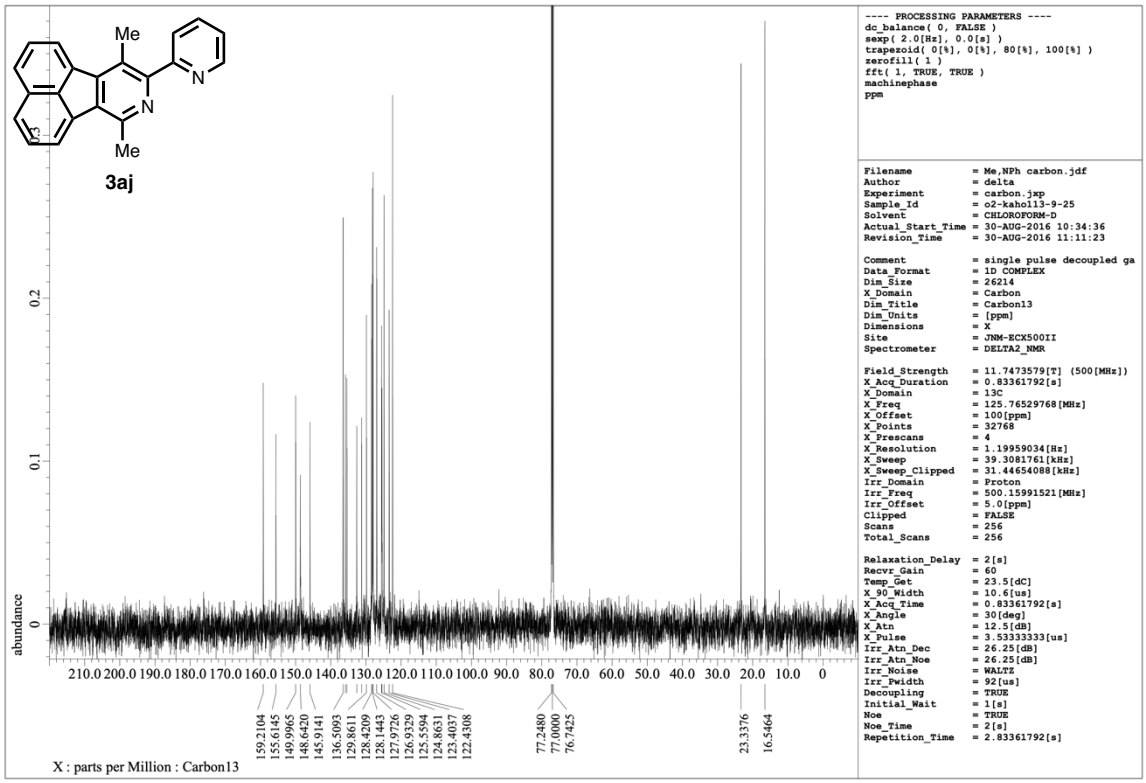


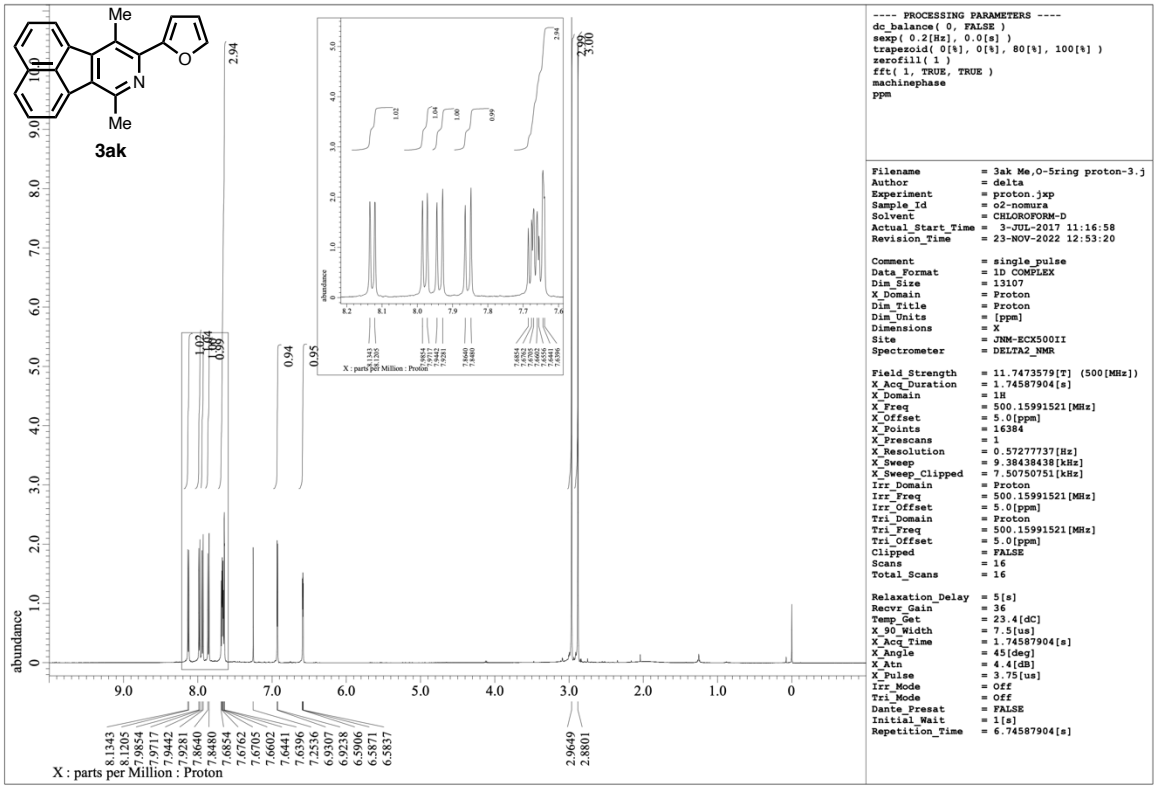

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = mari-131 Me,Ph-COOMe prot
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari131
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 30-APR-2021 20:25:53
Revision_Time       = 23-NOV-2022 12:42:50

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            = 23[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]
```

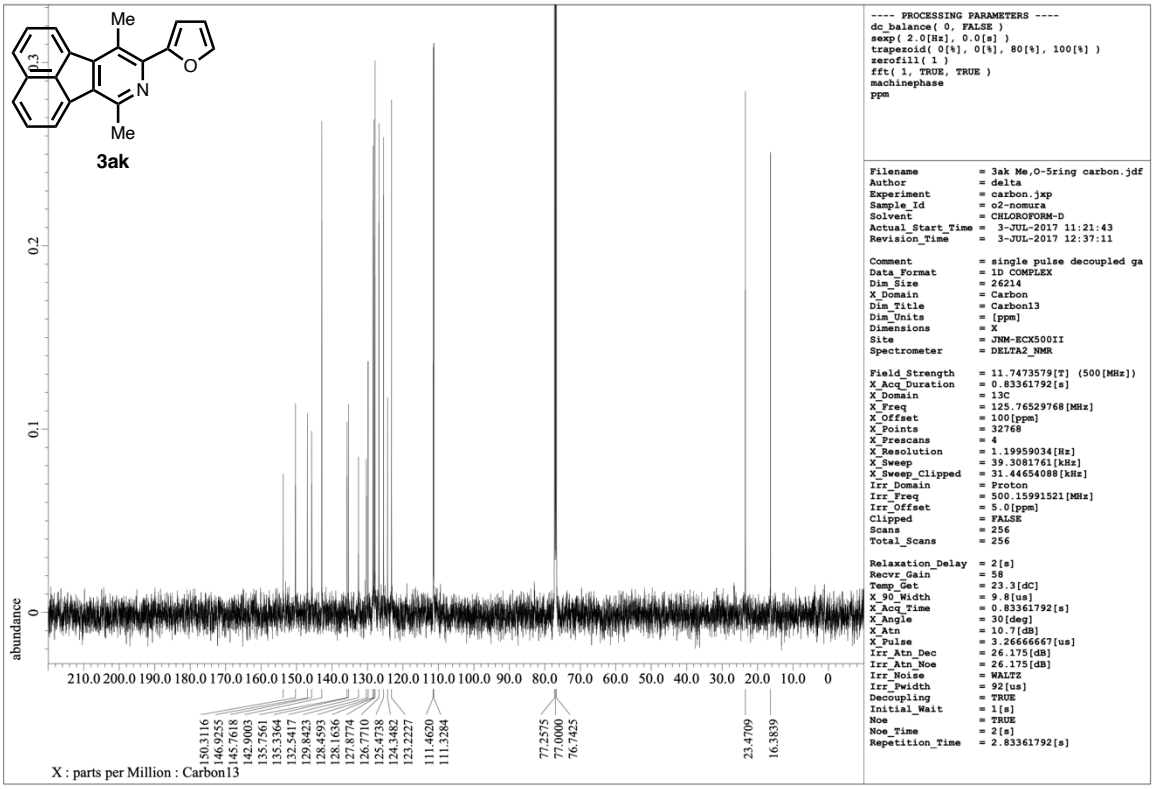


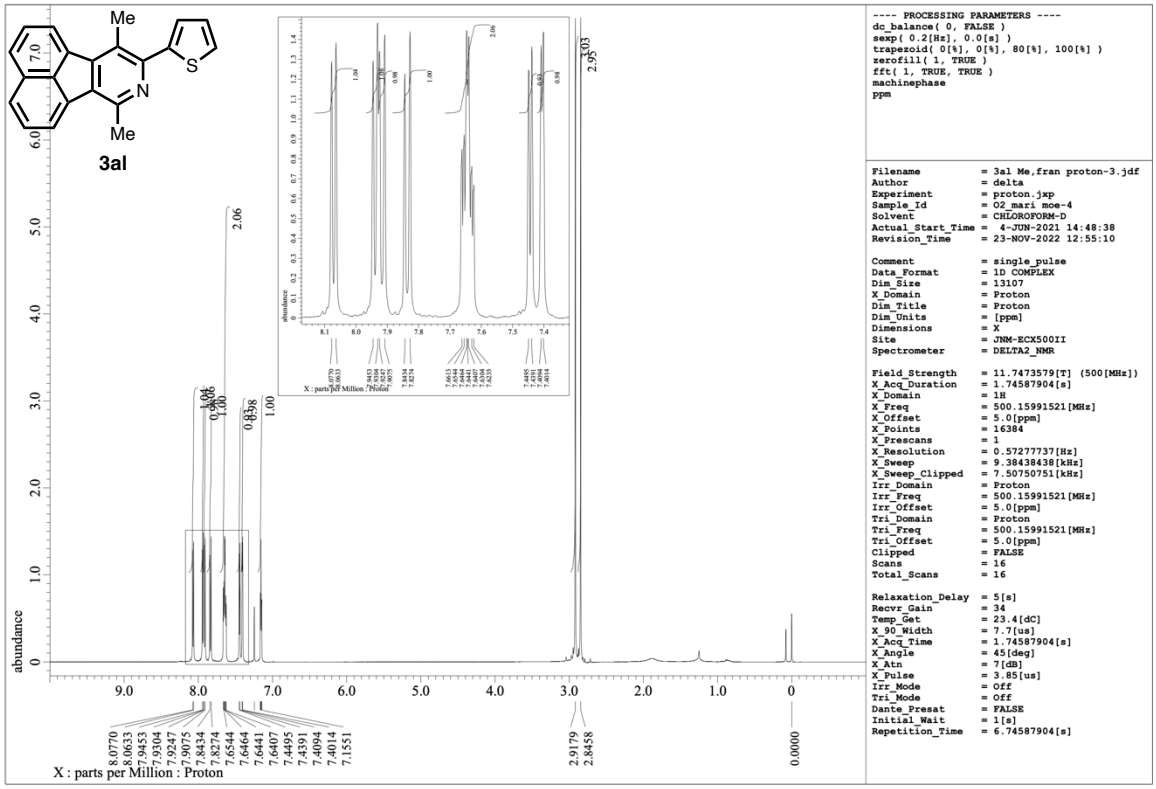

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = mari-131 Me,Ph-COOMe carb
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari131
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 30-APR-2021 20:29:04
Revision_Time       =  1-MAY-2021 20:30:50

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            = 23[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]
```

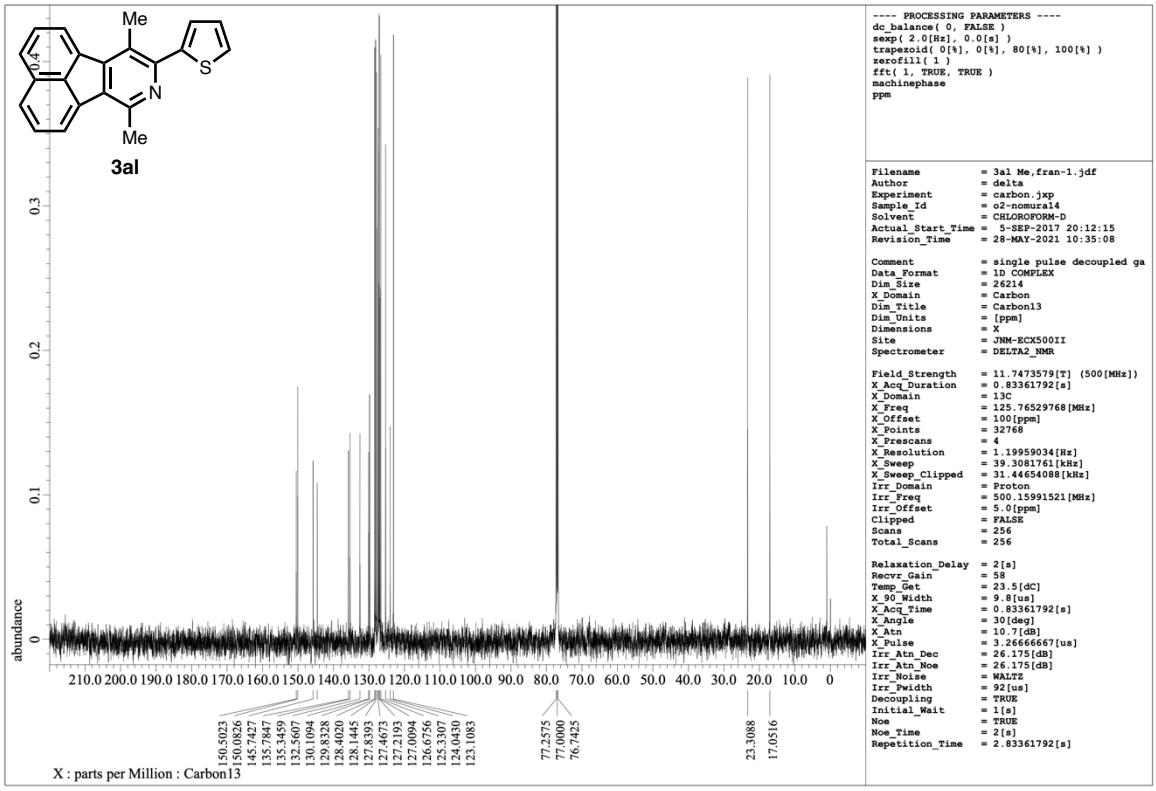

Me
N
N
Me
3aj
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Me,NPh proton-1.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kaho113-9-25
Solvent = CHLOROFORM-D
Actual_Start_Time = 30-AUG-2016 14:22:16
Revision_Time = 11-FEB-2022 12:10:55
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 = 23.4[dC]
X_90_Width = 6.7[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.5[dB]
X_Pulse = 3.35[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

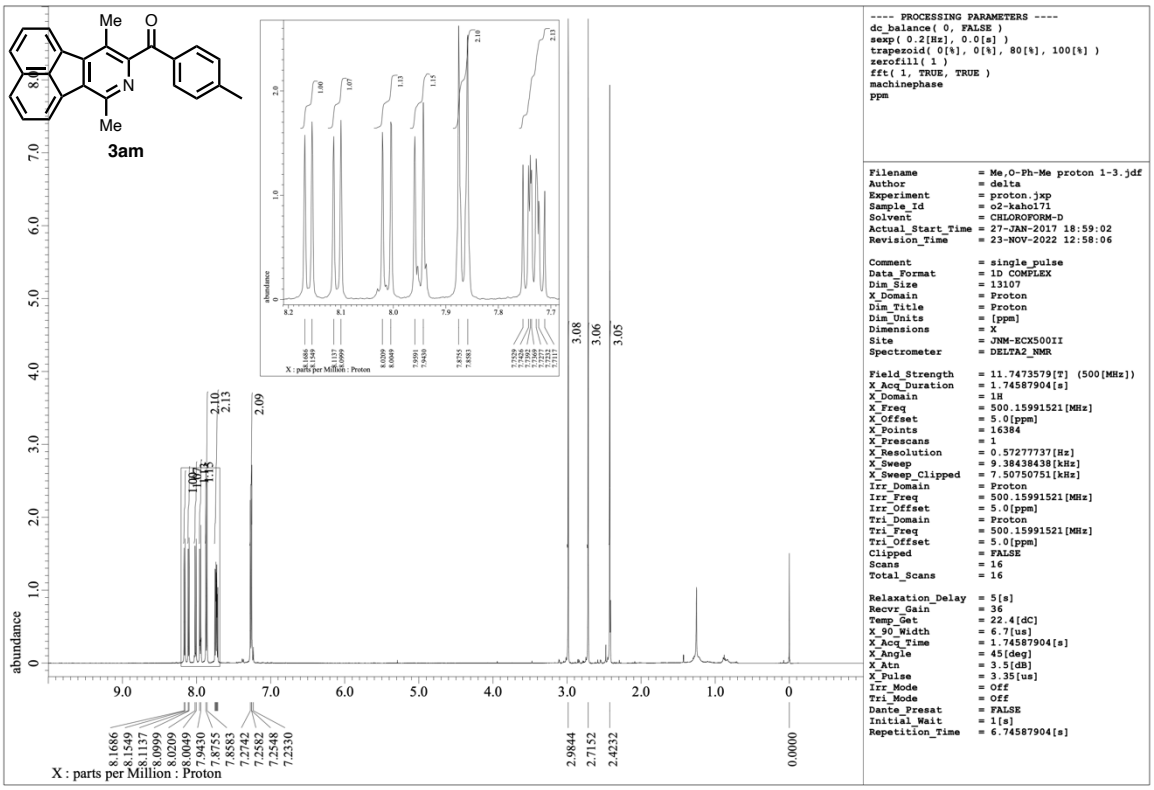

Me
N
N
Me
3aj
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Me,NPh carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-kaho113-9-25
Solvent = CHLOROFORM-D
Actual_Start_Time = 30-AUG-2016 10:34:36
Revision_Time = 30-AUG-2016 11:11:23
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 = 23.5[dC]
X_90_Width = 10.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12.5[dB]
X_Pulse = 3.53333333[us]
Irr_Atn_Dec = 26.25[dB]
Irr_Atn_Noe = 26.25[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]

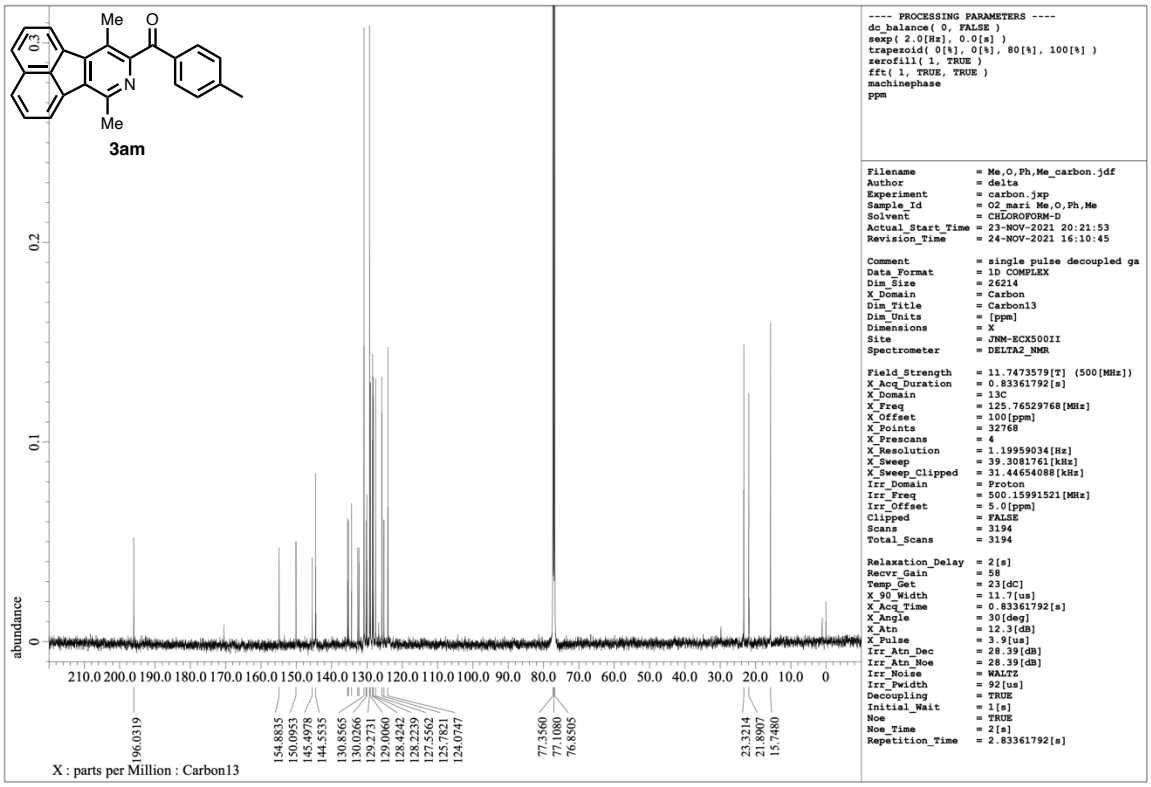

Me
N
O
Me
3ak
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 3ak Me,O-5ring proton-3.j
Author = delta
Experiment = proton.jxp
Sample_Id = o2-nomura
Solvent = CHLOROFORM-D
Actual_Start_Time = 3-JUL-2017 11:16:58
Revision_Time = 23-NOV-2022 12:53:20
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 = 23.4[dC]
X_90_Width = 7.5[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 3.75[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

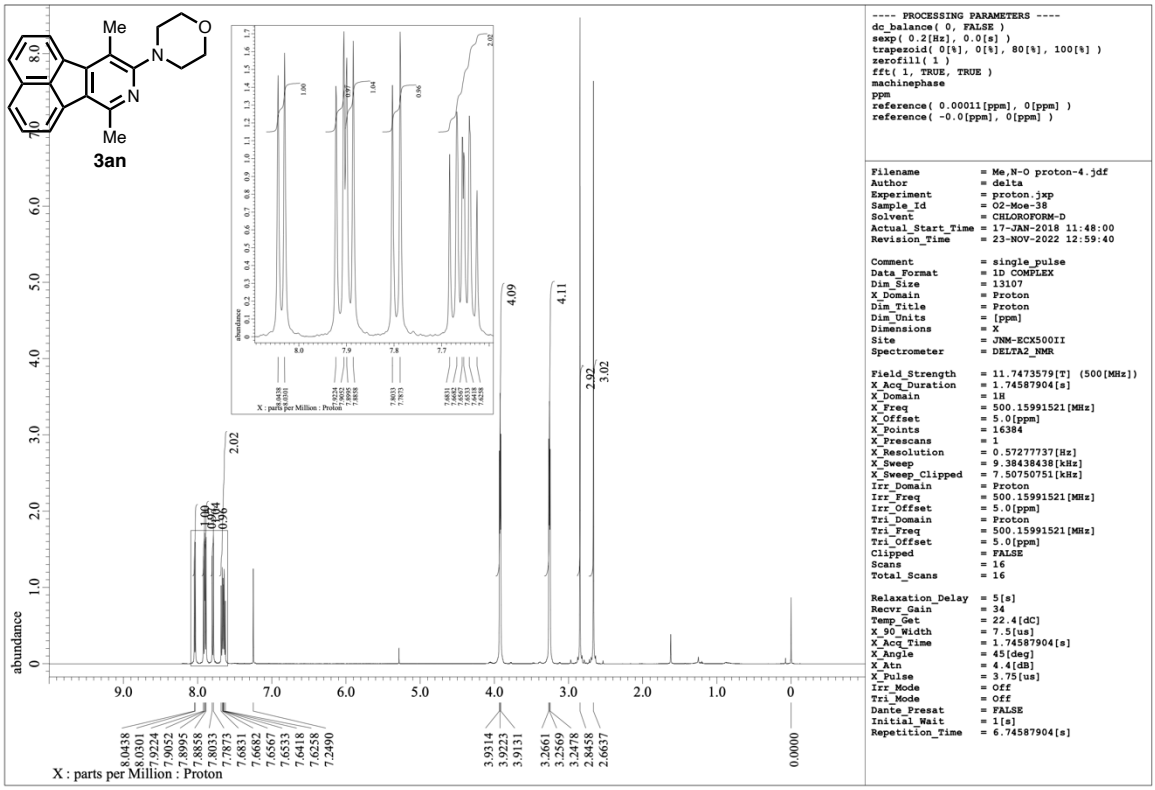

Me
N
O
Me
3ak
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 3ak Me,O-5ring carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-nomura
Solvent = CHLOROFORM-D
Actual_Start_Time = 3-JUL-2017 11:21:43
Revision_Time = 3-JUL-2017 12:37:11
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 = 39.30817761[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 = 23.3[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]

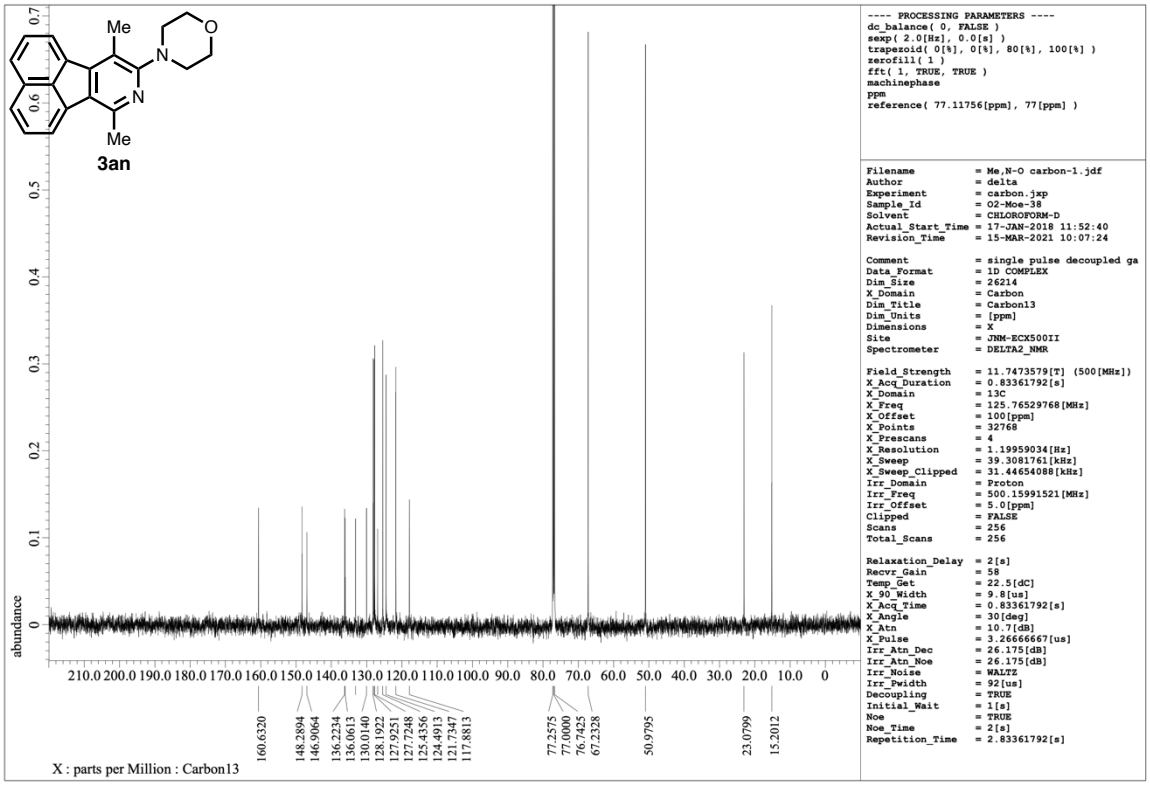

Me
S
N
Me
3al
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 3al Me,fran proton-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = 02_mari moe-4
Solvent = CHLOROFORM-D
Actual_Start_Time = 4-JUN-2021 14:48:38
Revision_Time = 23-NOV-2022 12:55:10
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 = 23.4[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]

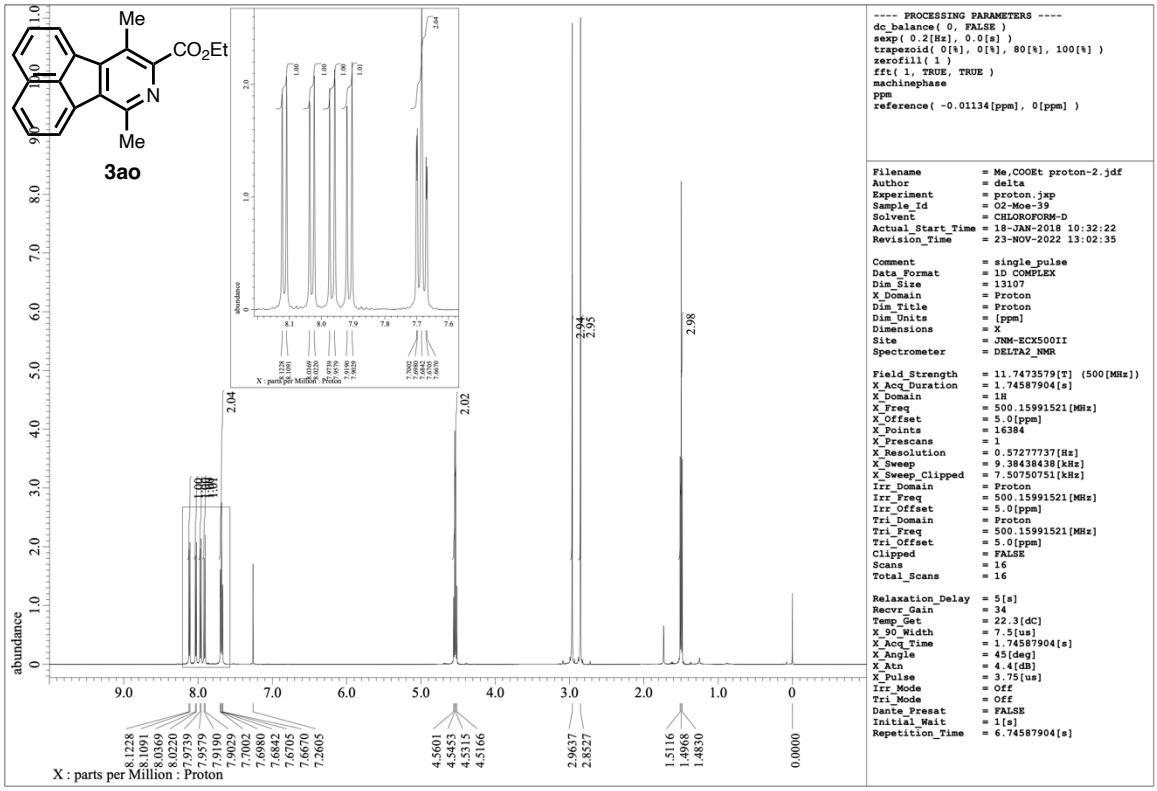

Me
S
N
Me
3al
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 3al Me,fran-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-nomura14
Solvent = CHLOROFORM-D
Actual_Start_Time = 5-SEP-2017 20:12:15
Revision_Time = 28-MAY-2021 10:35:08
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 = 23.5[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]


Me
O
N
Me
3am
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
Filename = Me,O-Ph-Me proton 1-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kaho171
Solvent = CHLOROFORM-D
Actual_Start_Time = 27-JAN-2017 18:59:02
Revision_Time = 23-NOV-2022 12:58:06
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 = 22.4[dC]
X_90_Width = 6.7[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.5[dB]
X_Pulse = 3.35[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]


Me
O
N
Me
3am
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 = Me,O,Ph,Me_carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari Me,O,Ph,Me
Solvent = CHLOROFORM-D
Actual_Start_Time = 23-NOV-2021 20:21:53
Revision_Time = 24-NOV-2021 16:10:45
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 = 3194
Total_Scans = 3194
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 23[dC]
X_90_Width = 11.7[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12.3[dB]
X_Pulse = 3.9[us]
Irr_Atn_Dec = 28.39[dB]
Irr_Atn_Noe = 28.39[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]

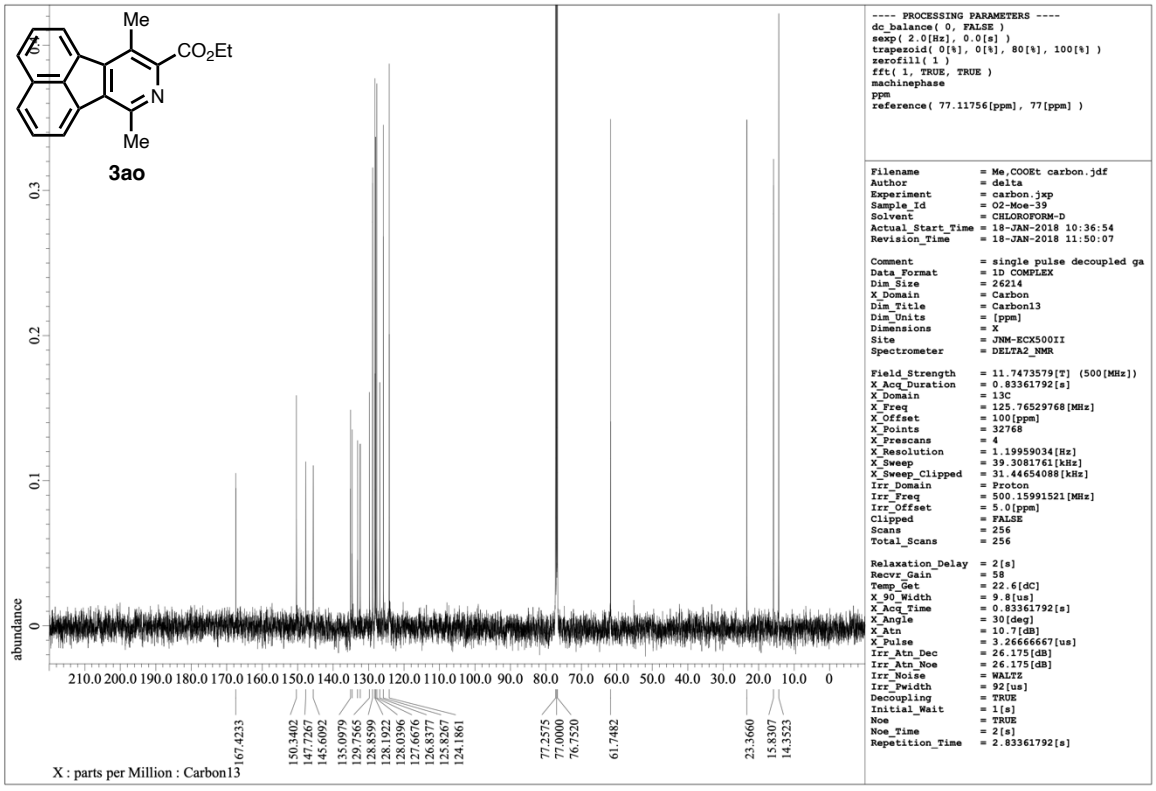

Me
O
N
N
Me
3an
abundance
X : parts per Million : Proton
8.0438
8.0301
7.9224
7.9052
7.8995
7.8858
7.8033
7.7873
7.6811
7.6682
7.6567
7.6533
7.6418
7.6258
7.2490
3.9314
3.9223
3.9131
3.2661
3.2569
3.2478
2.8458
2.6637
0.0000
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
reference(0.00011[ppm], 0[ppm])
reference(-0.0[ppm], 0[ppm])
Filename = Me,N-O proton-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = O2-Moe-38
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-JAN-2018 11:48:00
Revision_Time = 23-NOV-2022 12:59:40
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 = 7.5[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 3.75[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]


Me
O
N
N
Me
3an
abundance
X : parts per Million : Carbon13
160.6320
148.2894
146.9064
136.2234
136.0613
130.0140
128.1922
127.9251
127.7248
125.4356
124.4913
121.7347
117.8813
77.2575
77.0000
76.7425
67.2328
50.9795
23.0799
15.2012
---- 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 = Me,N-O carbon-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2-Moe-38
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-JAN-2018 11:52:40
Revision_Time = 15-MAR-2021 10:07:24
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 = 22.5[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]



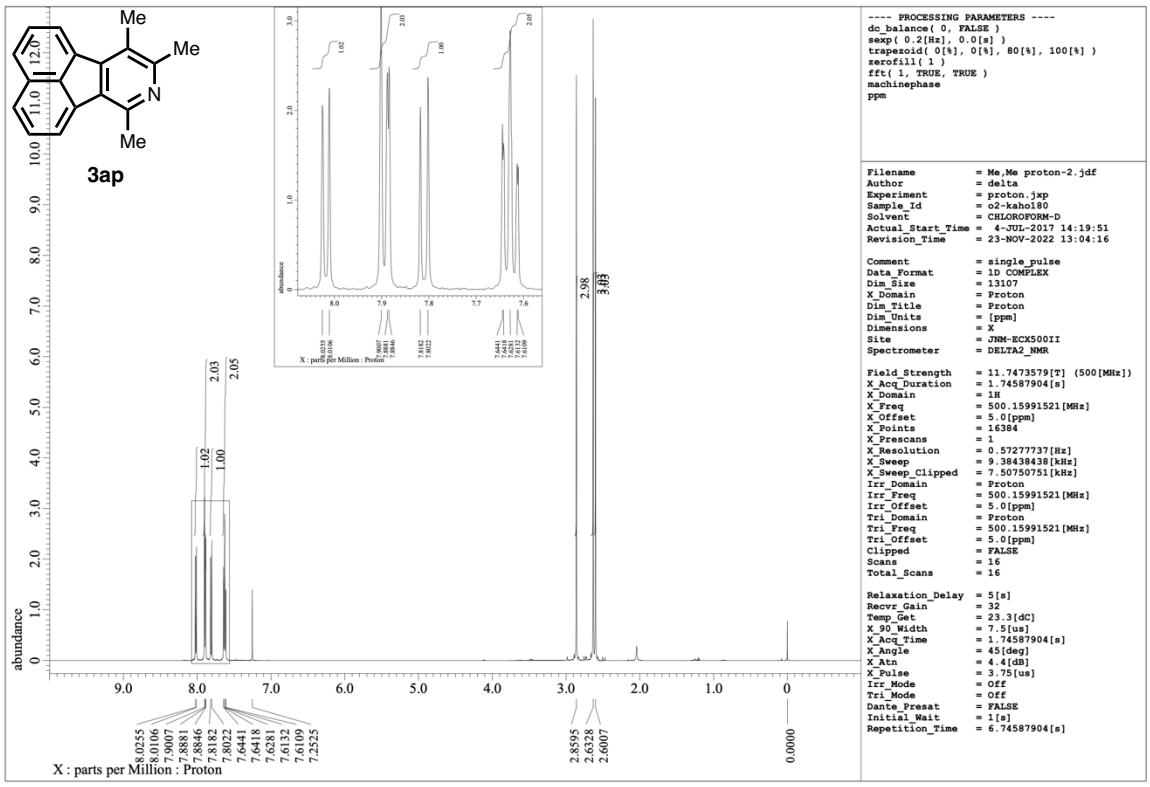

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( -0.01134[ppm], 0[ppm] )
```

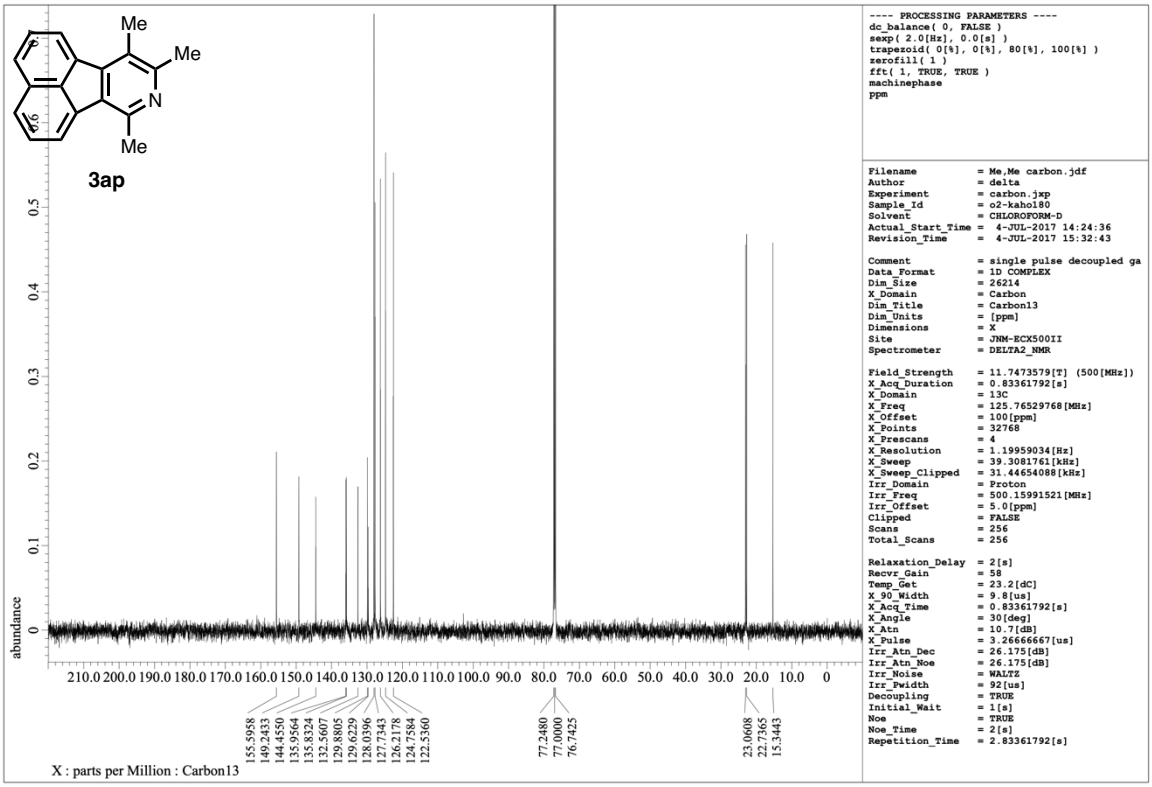
```
Filename            = Me,COOEt proton-2.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2-Moe-39
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 18-JAN-2018 10:32:22
Revision_Time       = 23-NOV-2022 13:02:35

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          = 7.5[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 4.4[dB]
X_Pulse             = 3.75[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

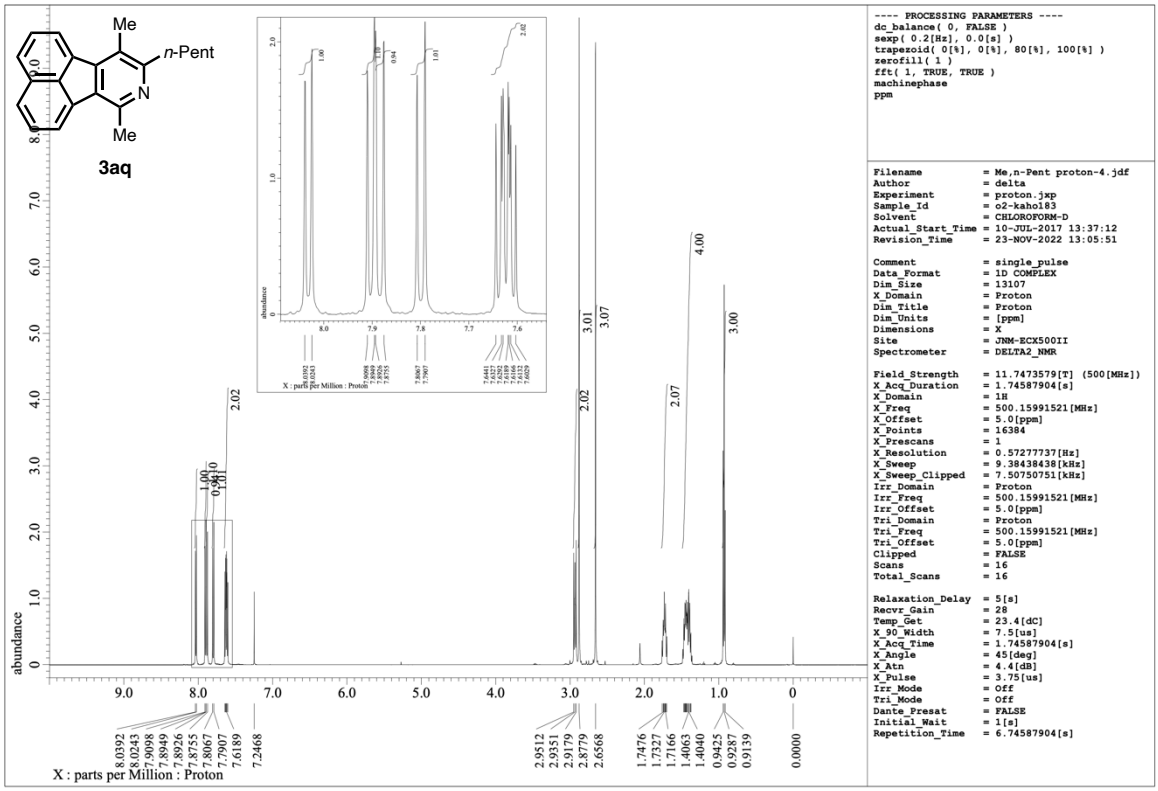


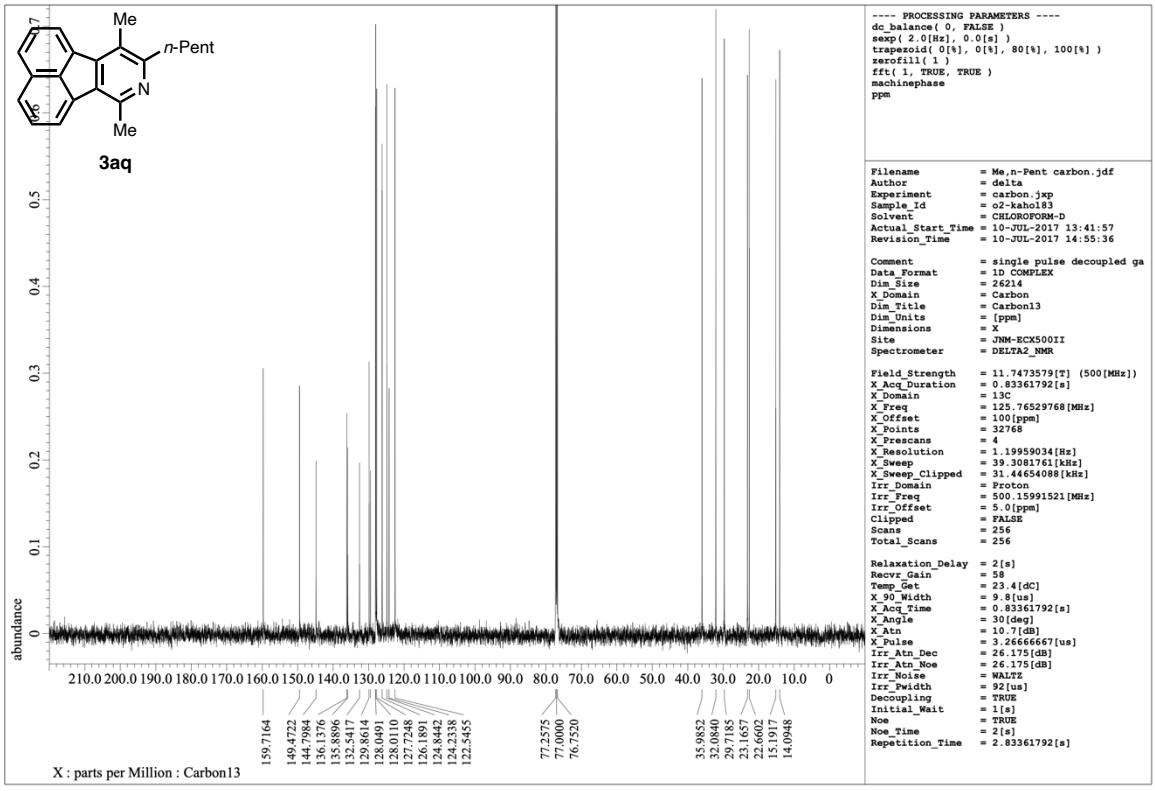

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.11756[ppm], 77[ppm] )
```

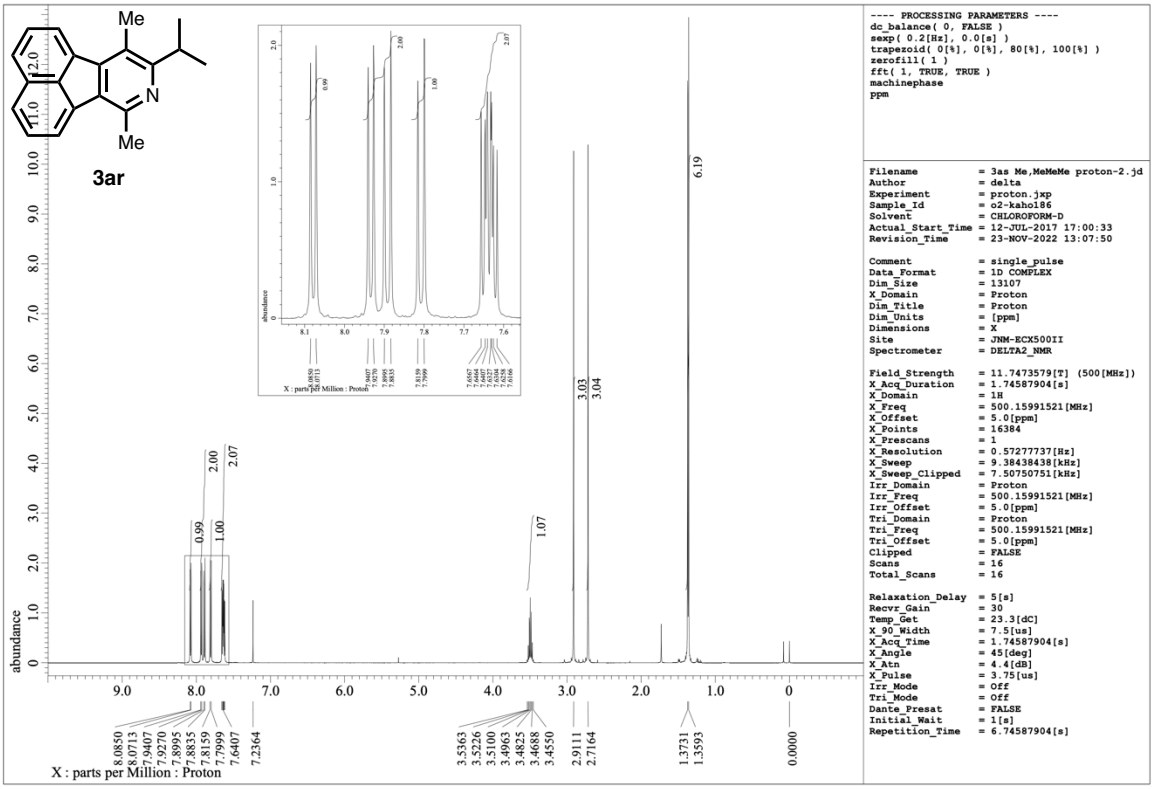
```
Filename            = Me,COOEt carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2-Moe-39
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 18-JAN-2018 10:36:54
Revision_Time       = 18-JAN-2018 11:50:07

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          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```


Me
Me
N
Me
3ap
abundance
X : parts per Million : Proton
2.03
2.05
1.02
1.00
2.98
3.03
8.0255
8.0106
7.9007
7.8881
7.8846
7.8182
7.8022
7.6441
7.6418
7.6281
7.6132
7.6109
7.2525
2.8595
2.6328
2.6007
0.0000
---- 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
Filename = Me,Me proton-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kaho180
Solvent = CHLOROFORM-D
Actual_Start_Time = 4-JUL-2017 14:19:51
Revision_Time = 23-NOV-2022 13:04:16
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 = 23.3[dC]
X_90_Width = 7.5[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 3.75[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

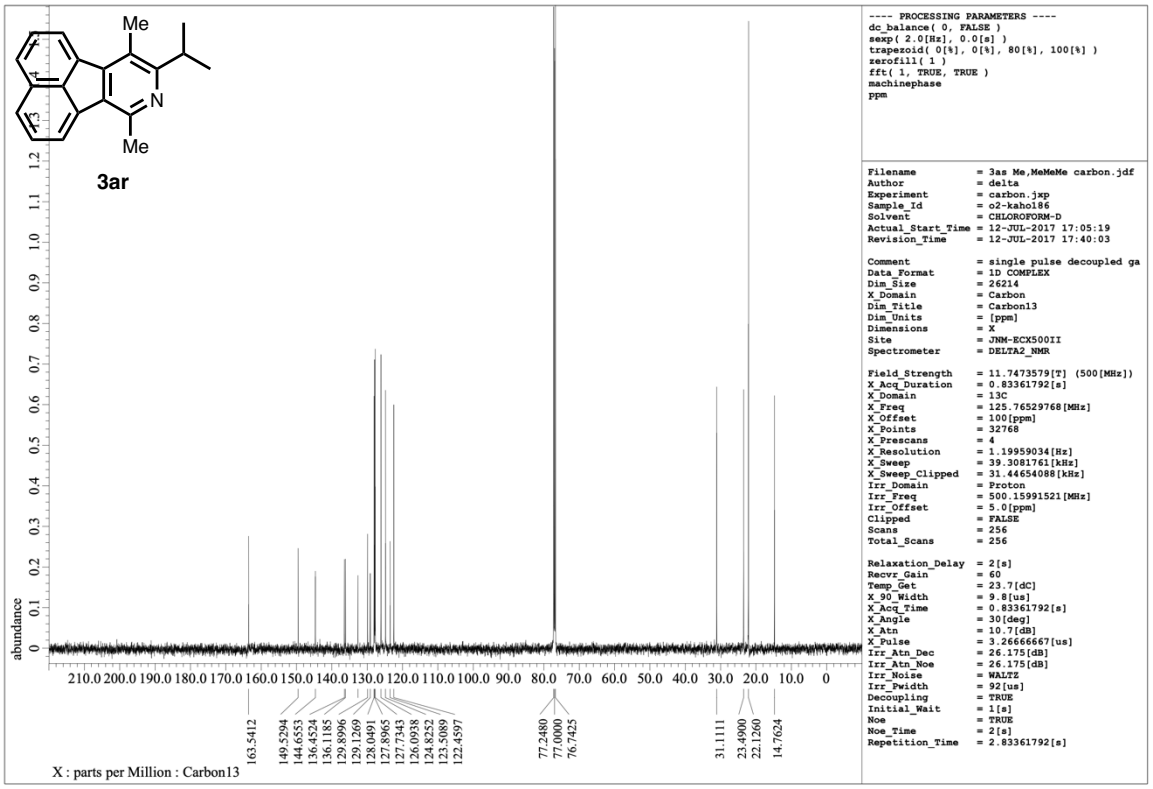

Me
Me
N
Me
3ap
abundance
X : parts per Million : Carbon13
155.5958
149.2433
144.4550
135.9564
135.8324
132.5607
129.8805
129.6229
128.0396
127.7343
126.2178
124.7584
122.5360
77.2480
77.0000
76.7425
23.0608
22.7365
15.3443
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Me,Me carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-kaho180
Solvent = CHLOROFORM-D
Actual_Start_Time = 4-JUL-2017 14:24:36
Revision_Time = 4-JUL-2017 15:32:43
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 = 23.2[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]


Me
n-Pent
N
Me
3aq
abundance
X : parts per Million : Proton
8.0392
8.0243
7.9098
7.8949
7.8926
7.8755
7.8067
7.7907
7.6189
7.2468
2.9512
2.9351
2.9179
2.8779
2.6568
1.7476
1.7327
1.7166
1.4063
1.4040
0.9425
0.9287
0.9139
0.0000
---- 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
Filename = Me,n-Pent proton-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kahol83
Solvent = CHLOROFORM-D
Actual_Start_Time = 10-JUL-2017 13:37:12
Revision_Time = 23-NOV-2022 13:05:51
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 = 23.4[dC]
X_90_Width = 7.5[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 3.75[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

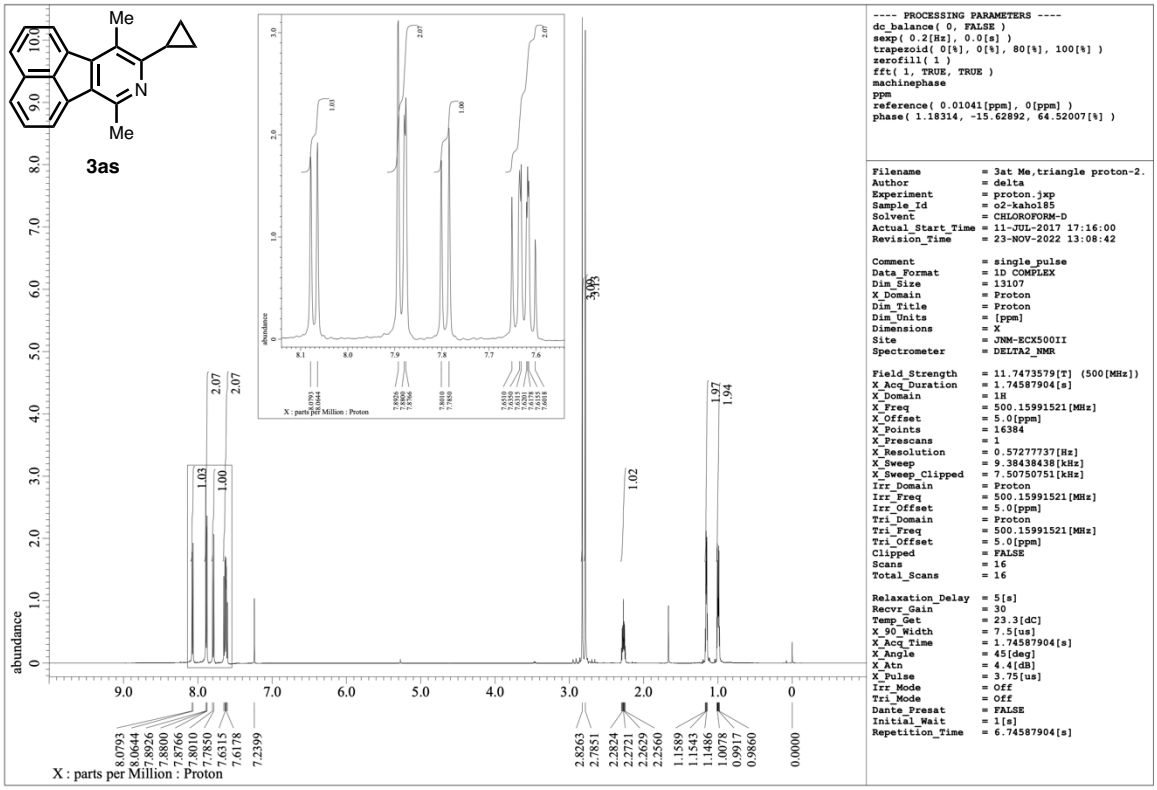

Me
n-Pent
N
Me
3aq
abundance
X : parts per Million : Carbon13
159.7164
149.4722
144.7984
136.1376
135.8896
132.5417
129.8614
128.0491
128.0110
127.7248
126.1891
124.8442
124.2338
122.5455
77.2575
77.0000
76.7520
35.9852
32.0840
29.7185
23.1657
22.6602
15.1917
14.0948
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Me,n-Pent carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-kahol83
Solvent = CHLOROFORM-D
Actual_Start_Time = 10-JUL-2017 13:41:57
Revision_Time = 10-JUL-2017 14:55:36
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 = 58
Temp_Get = 23.4[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]




```
---- 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

Filename            = 3as Me,MeMeMe proton-2.jd
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2-kaho186
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 12-JUL-2017 17:00:33
Revision_Time       = 23-NOV-2022 13:07:50

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          = 30
Temp_Get            = 23.3[dC]
X_90_Width          = 7.5[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 4.4[dB]
X_Pulse             = 3.75[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

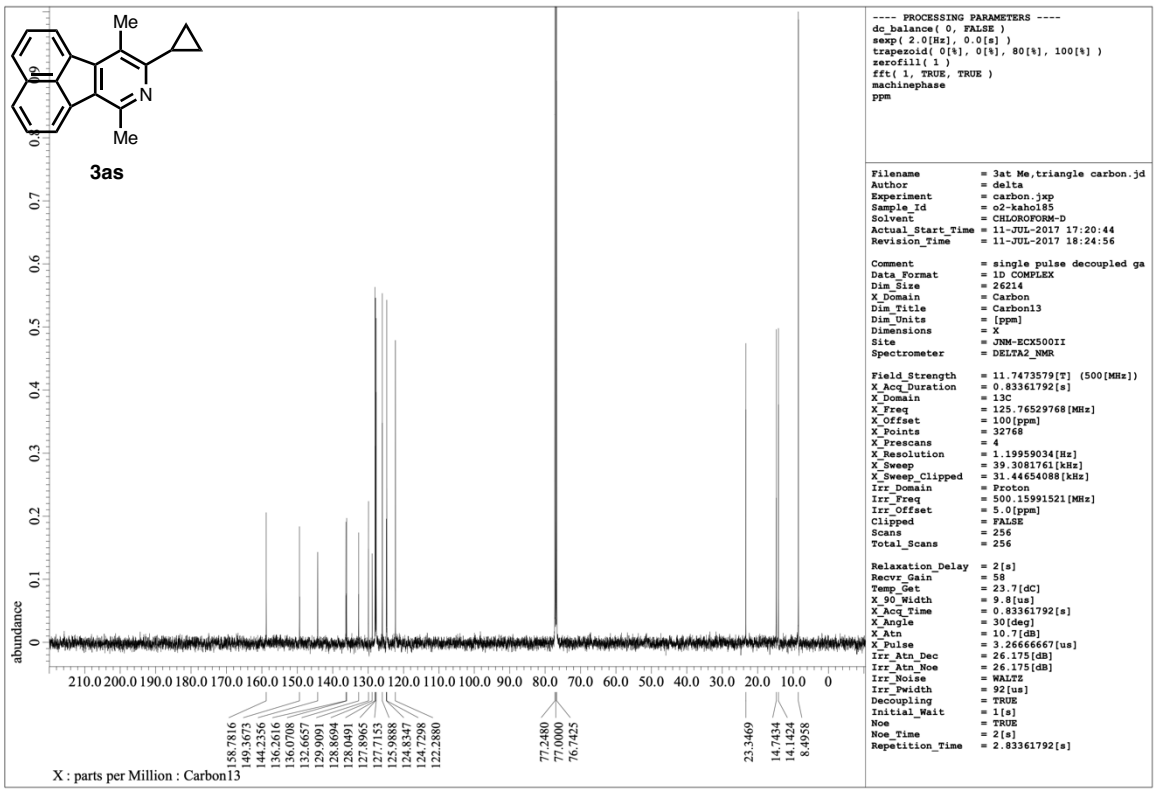



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3as Me,MeMeMe carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2-kaho186
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 12-JUL-2017 17:05:19
Revision_Time       = 12-JUL-2017 17:40:03

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          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```



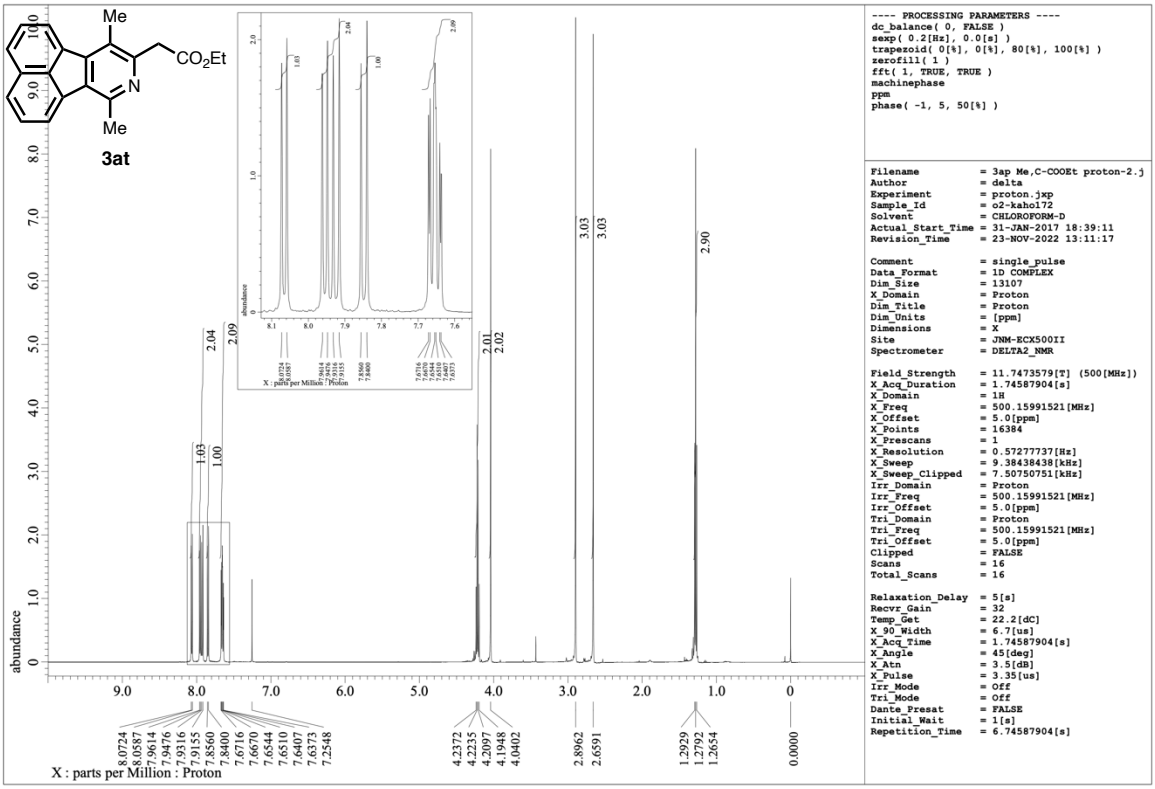

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 0.01041[ppm], 0[ppm] )
phase( 1.18314, -15.62892, 64.52007[%] )
```

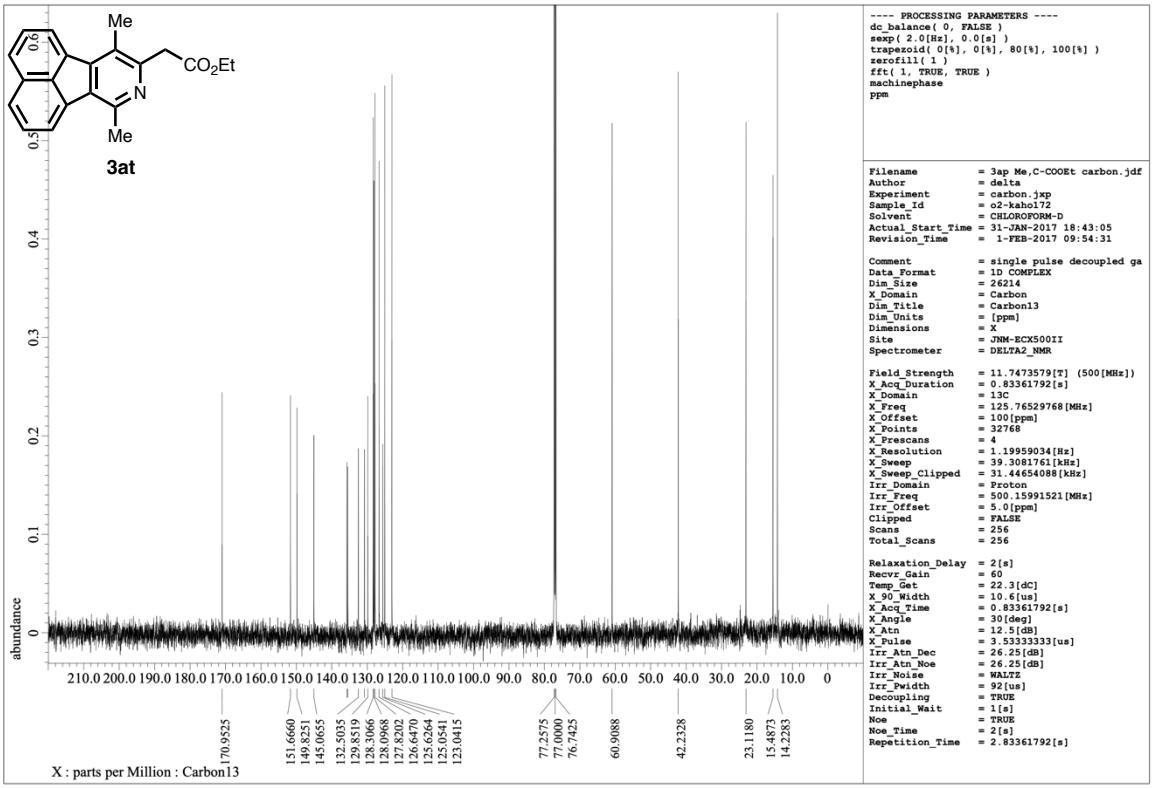
```
Filename            = 3at Me,triangle proton-2.
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2-kaho185
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 11-JUL-2017 17:16:00
Revision_Time       = 23-NOV-2022 13:08:42

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            = 23.3[dC]
X_90_Width          = 7.5[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 4.4[dB]
X_Pulse             = 3.75[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

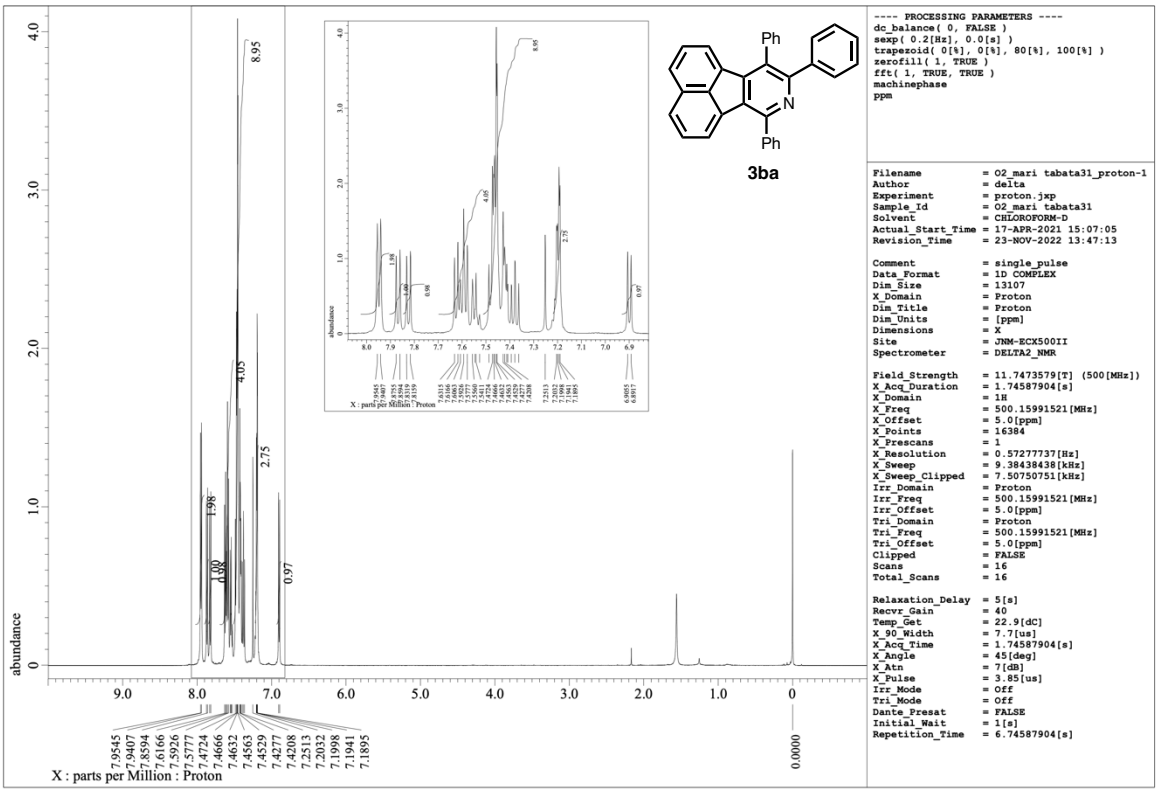



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

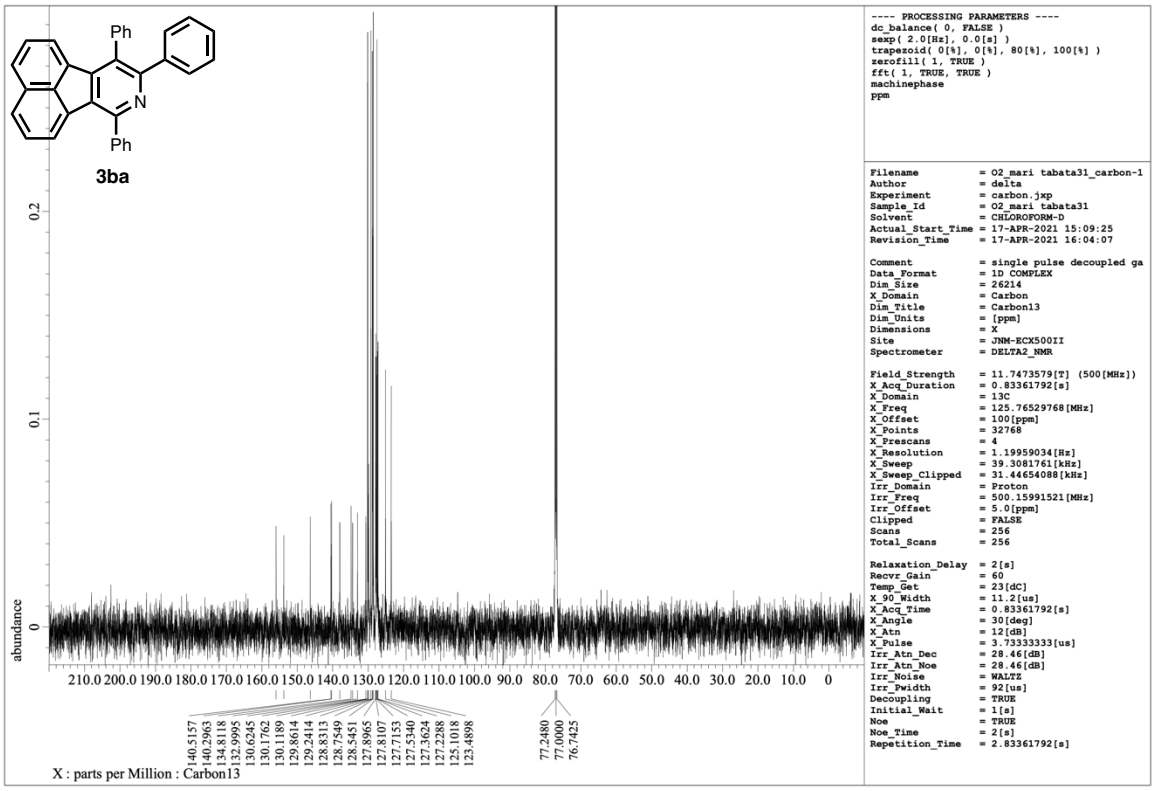
```
Filename            = 3at Me,triangle carbon.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2-kaho185
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 11-JUL-2017 17:20:44
Revision_Time       = 11-JUL-2017 18:24:56

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            = 23.7[dC]
X_90_Width          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```


Me
CO2Et
N
Me
3at
abundance
X : parts per Million : Proton
8.0724
8.0587
7.9614
7.9476
7.9316
7.9155
7.8560
7.8400
7.6716
7.6670
7.6544
7.6510
7.6407
7.6373
7.2548
4.2372
4.2235
4.2097
4.1948
4.0402
2.8962
2.6591
1.2929
1.2792
1.2654
0.0000
---- 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
phase(-1, 5, 50[%])
Filename = 3ap Me,C-COOEt proton-2.j
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kaho172
Solvent = CHLOROFORM-D
Actual_Start_Time = 31-JAN-2017 18:39:11
Revision_Time = 23-NOV-2022 13:11:17
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 = 32
Temp_Get = 22.2[dC]
X_90_Width = 6.7[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.5[dB]
X_Pulse = 3.35[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

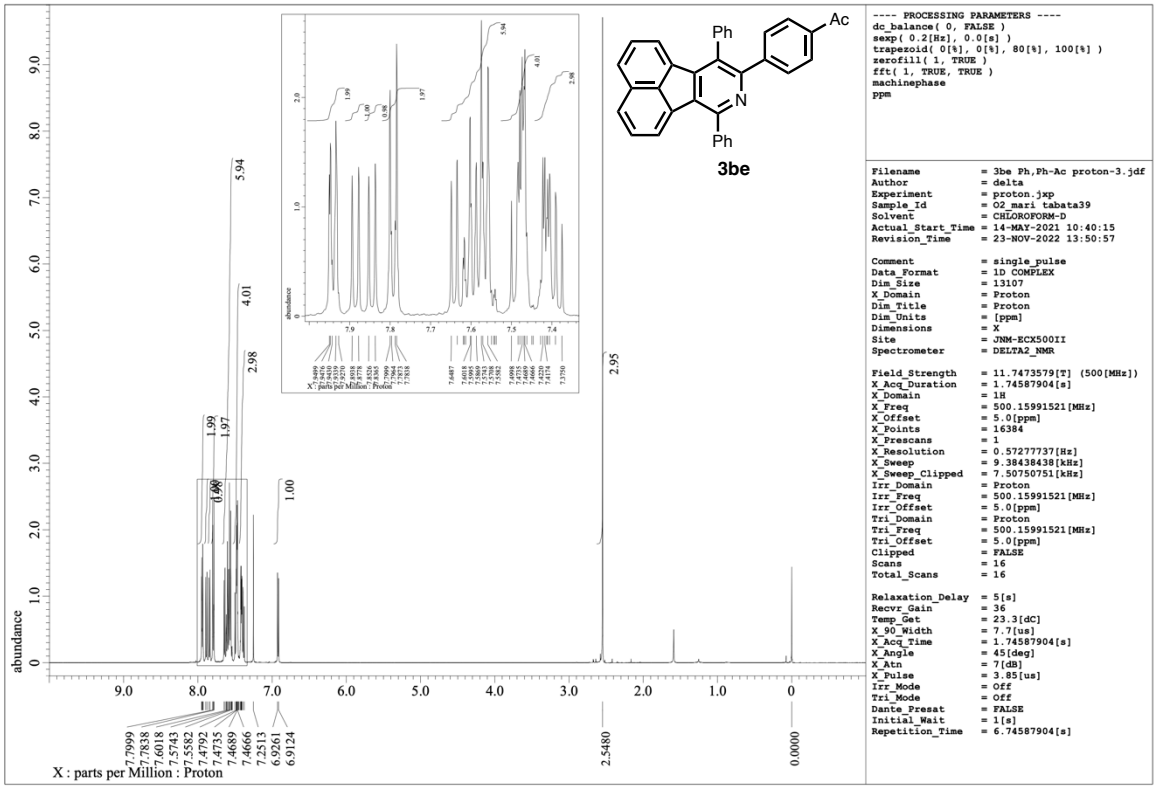

Me
CO2Et
N
Me
3at
abundance
X : parts per Million : Carbon13
170.9525
151.6660
149.8251
145.0655
132.5035
129.8519
128.3066
128.0968
127.8202
126.6470
125.6264
125.0541
123.0415
77.2575
77.0000
76.7425
60.9088
42.2328
23.1180
15.4873
14.2283
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = 3ap Me,C-COOEt carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-kaho172
Solvent = CHLOROFORM-D
Actual_Start_Time = 31-JAN-2017 18:43:05
Revision_Time = 1-FEB-2017 09:54:31
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 = 10.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12.5[dB]
X_Pulse = 3.53333333[us]
Irr_Atn_Dec = 26.25[dB]
Irr_Atn_Noe = 26.25[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]



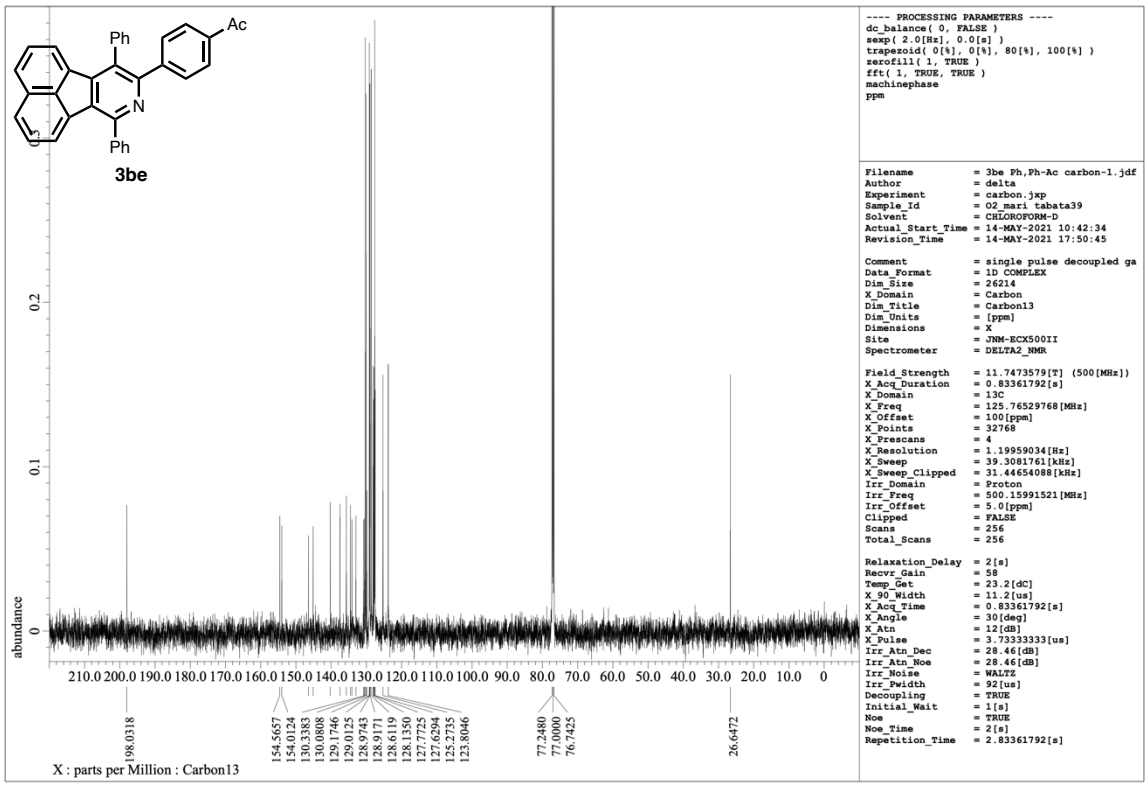

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = O2_mari tabata31_proton-1
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari tabata31
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 17-APR-2021 15:07:05
Revision_Time       = 23-NOV-2022 13:47:13

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          = 40
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]
```



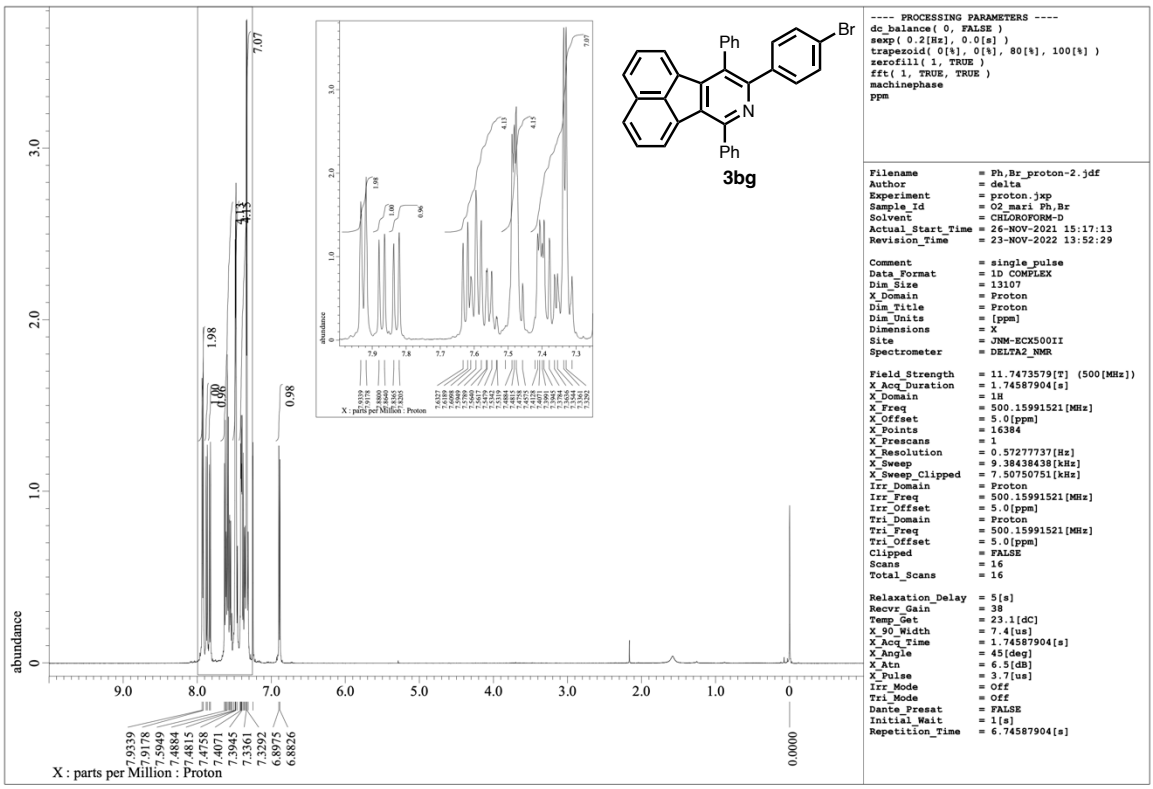

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = O2_mari tabata31_carbon-1
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari tabata31
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 17-APR-2021 15:09:25
Revision_Time       = 17-APR-2021 16:04:07

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[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]
```



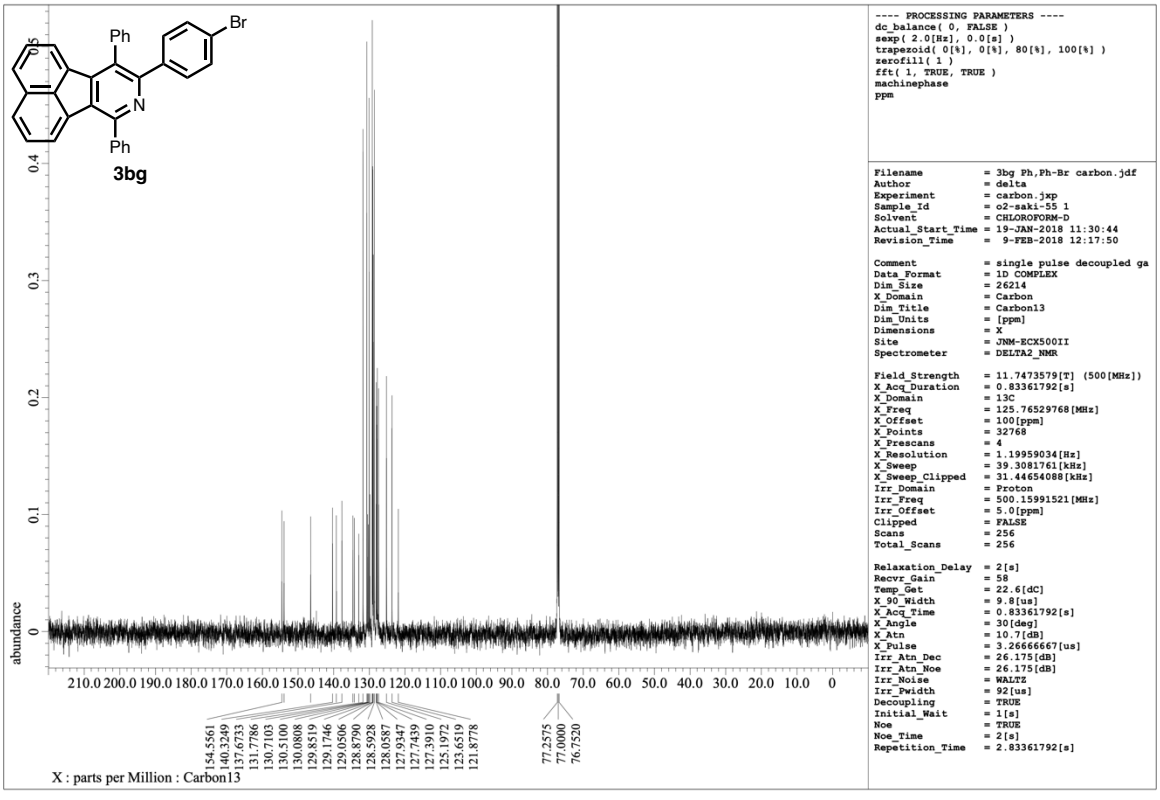

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3be Ph,Ph-Ac proton-3.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari tabata39
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 14-MAY-2021 10:40:15
Revision_Time       = 23-NOV-2022 13:50:57

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            = 23.3[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]
```



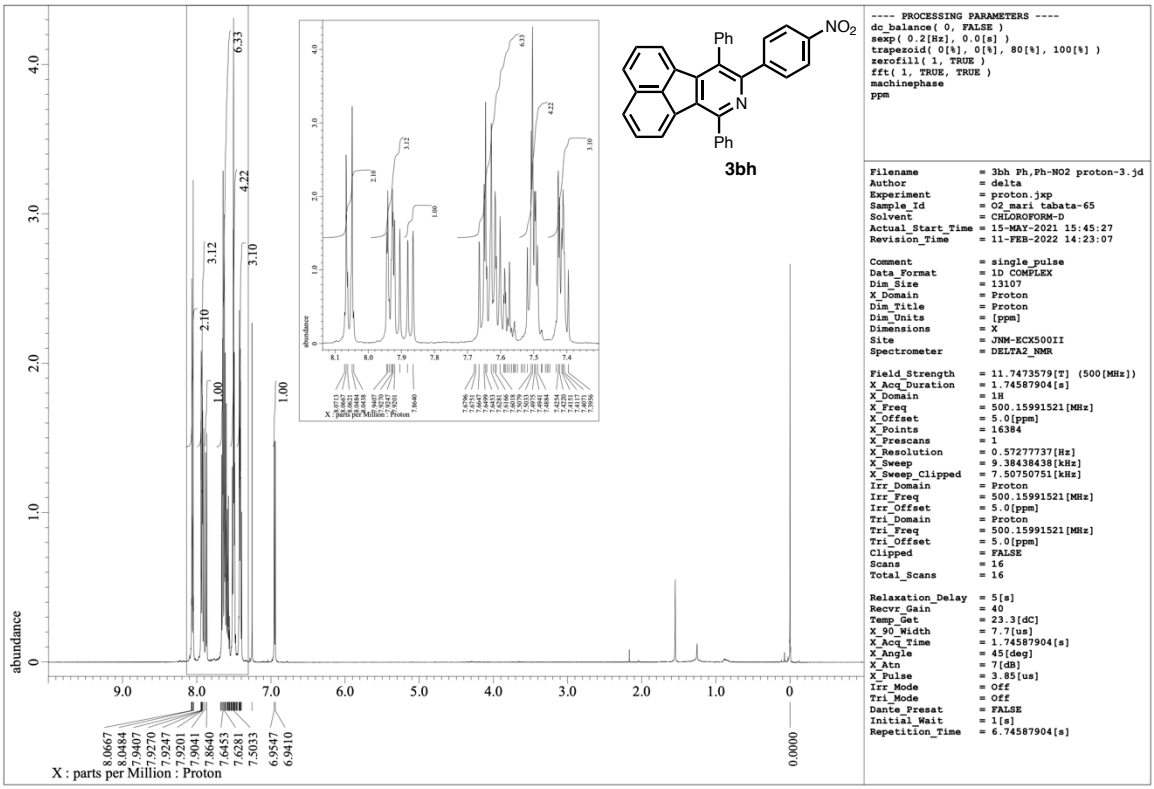

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3be Ph,Ph-Ac carbon-1.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari tabata39
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 14-MAY-2021 10:42:34
Revision_Time       = 14-MAY-2021 17:50:45

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          = 58
Temp_Get            = 23.2[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]
```



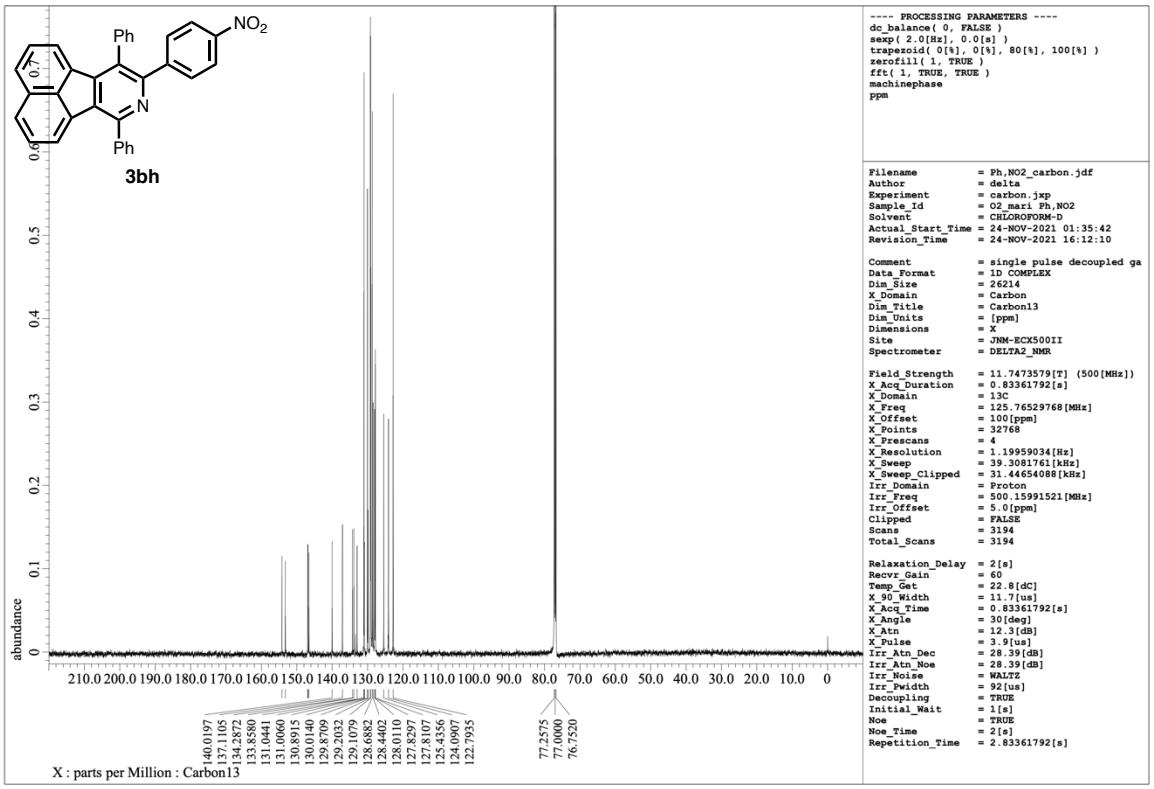

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = Ph,Br_proton-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = O2_mari Ph,Br
Solvent           = CHLOROFORM-D
Actual_Start_Time = 26-NOV-2021 15:17:13
Revision_Time     = 23-NOV-2022 13:52:29

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        = 38
Temp_Get          = 23.1[dC]
X_90_Width        = 7.4[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 3.7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



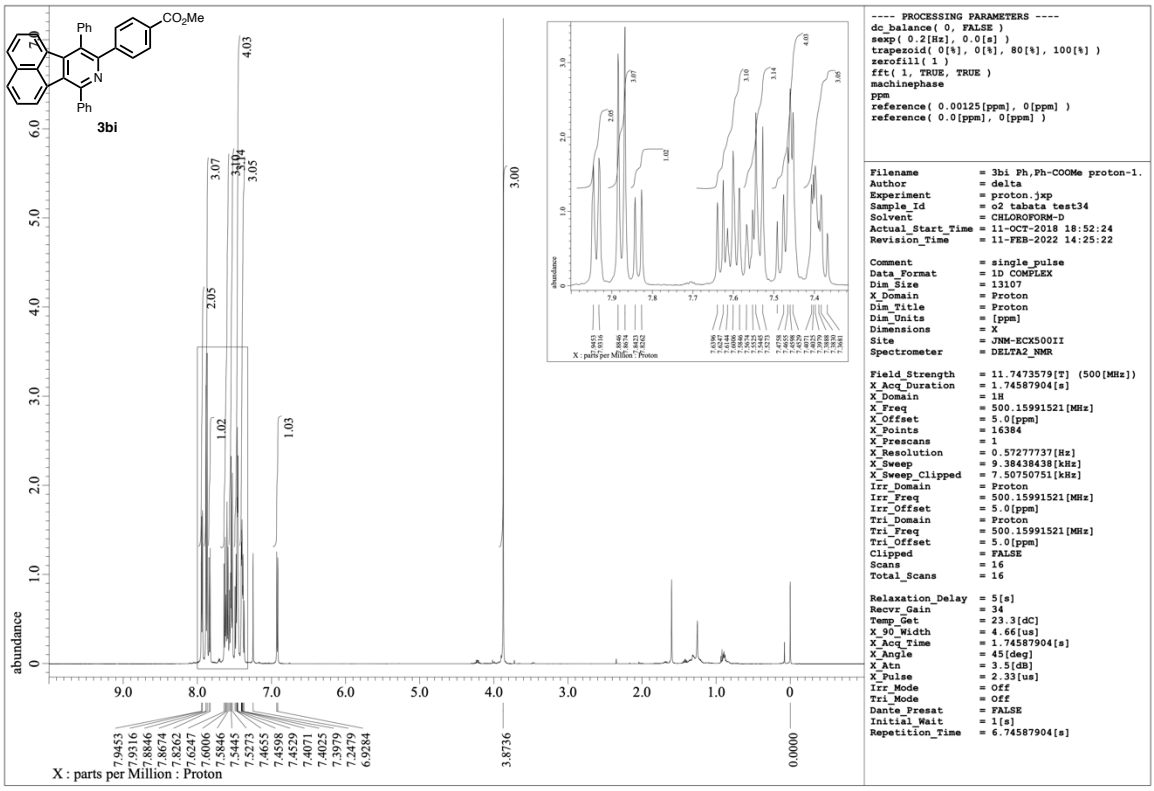

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename          = 3bg Ph,Ph-Br carbon.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = o2-saki-55 1
Solvent           = CHLOROFORM-D
Actual_Start_Time = 19-JAN-2018 11:30:44
Revision_Time     =  9-FEB-2018 12:17:50

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        = 58
Temp_Get          = 22.6[dC]
X_90_Width        = 9.8[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 10.7[dB]
X_Pulse           = 3.26666667[us]
Irr_Atn_Dec       = 26.175[dB]
Irr_Atn_Noe       = 26.175[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]
```




```
---- 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            = 3bh Ph,Ph-NO2 proton-3.jd
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari tabata-65
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-MAY-2021 15:45:27
Revision_Time       = 11-FEB-2022 14:23:07

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          = 40
Temp_Get            = 23.3[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]
```

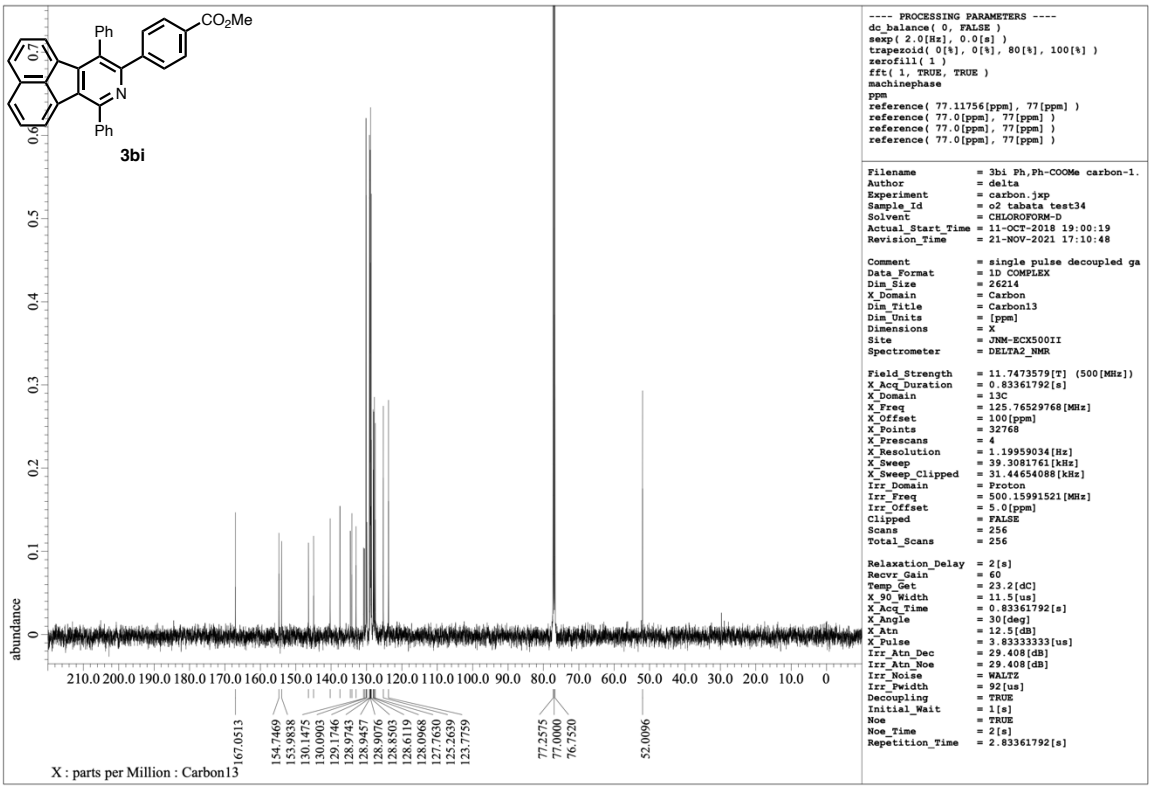



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = Ph,NO2_carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari Ph,NO2
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 24-NOV-2021 01:35:42
Revision_Time       = 24-NOV-2021 16:12:10

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               = 3194
Total_Scans         = 3194

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.8[dC]
X_90_Width          = 11.7[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12.3[dB]
X_Pulse             = 3.9[us]
Irr_Atn_Dec         = 28.39[dB]
Irr_Atn_Noe         = 28.39[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]
```



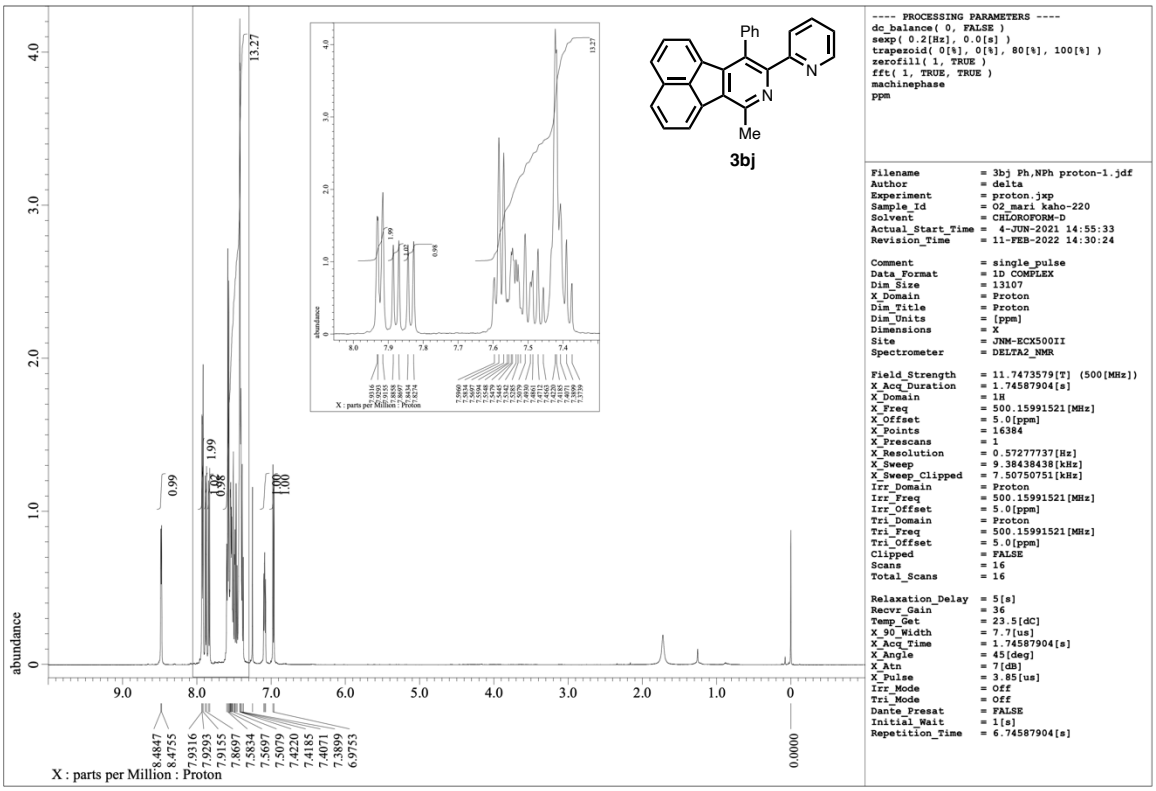

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 0.00125[ppm], 0[ppm] )
reference( 0.0[ppm], 0[ppm] )
```

```
Filename            = 3bi Ph,Ph-COOMe proton-1.
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 tabata test34
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 11-OCT-2018 18:52:24
Revision_Time       = 11-FEB-2022 14:25:22

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            = 23.3[dC]
X_90_Width          = 4.66[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 3.5[dB]
X_Pulse             = 2.33[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

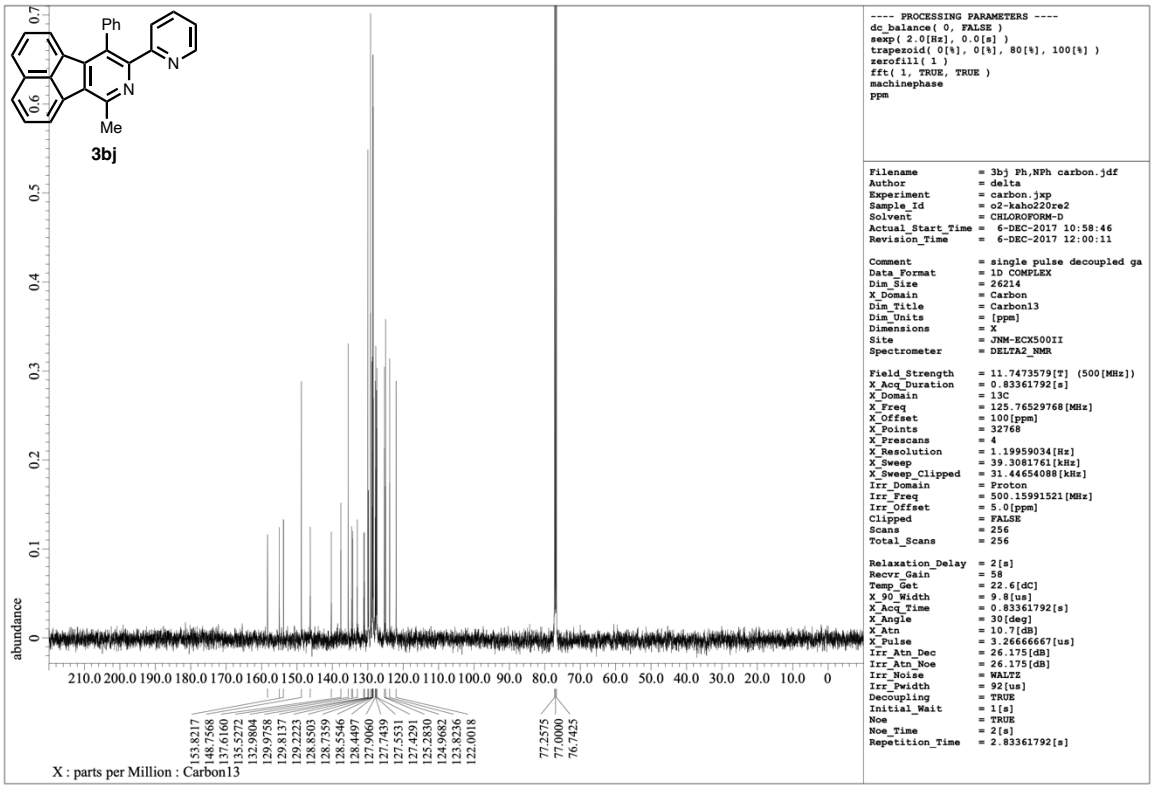



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
reference( 77.11756[ppm], 77[ppm] )
reference( 77.0[ppm], 77[ppm] )
reference( 77.0[ppm], 77[ppm] )
reference( 77.0[ppm], 77[ppm] )
```

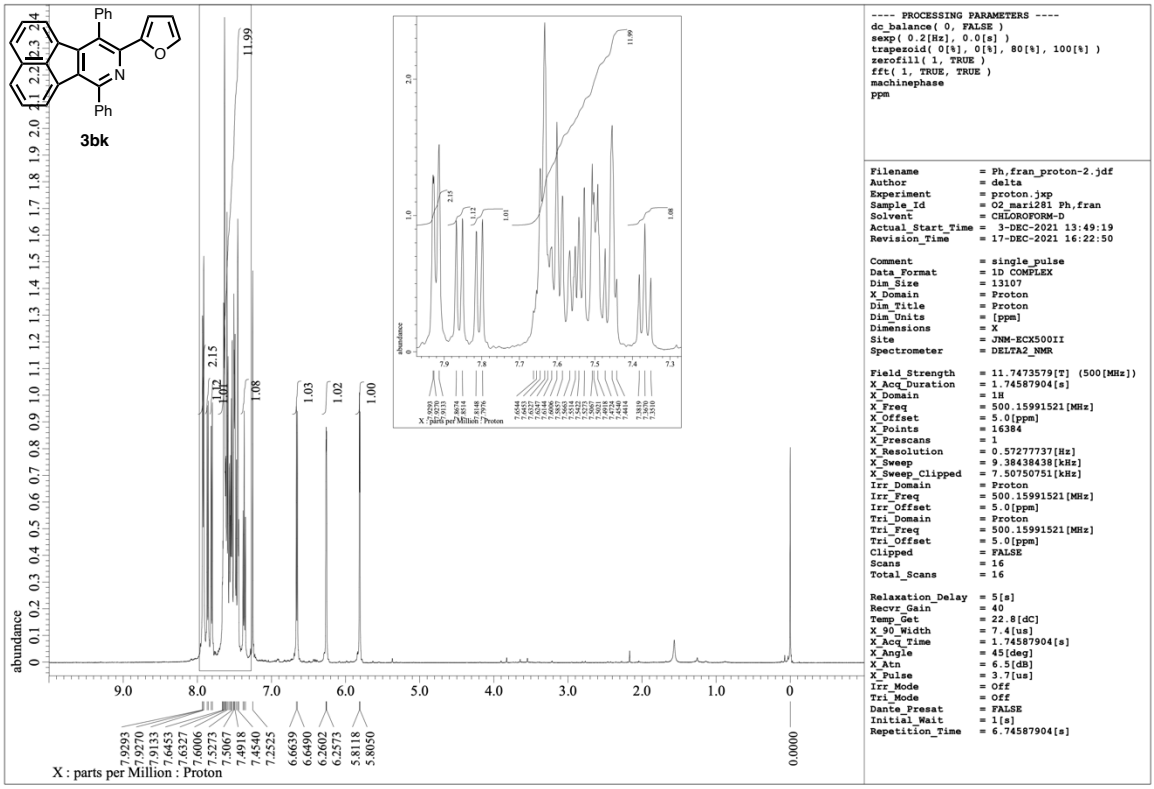
```
Filename            = 3bi Ph,Ph-COOMe carbon-1.
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 tabata test34
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 11-OCT-2018 19:00:19
Revision_Time       = 21-NOV-2021 17:10:48

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            = 23.2[dC]
X_90_Width          = 11.5[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12.5[dB]
X_Pulse             = 3.83333333[us]
Irr_Atn_Dec         = 29.408[dB]
Irr_Atn_Noe         = 29.408[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]
```

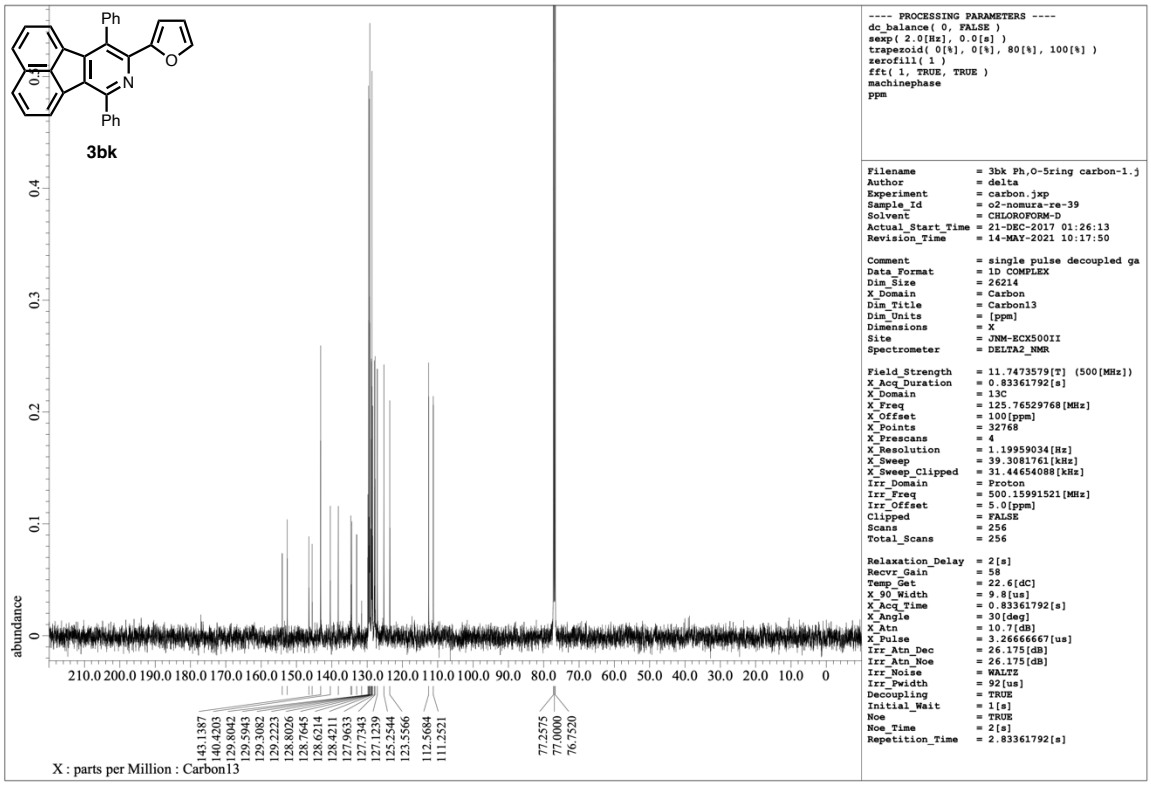


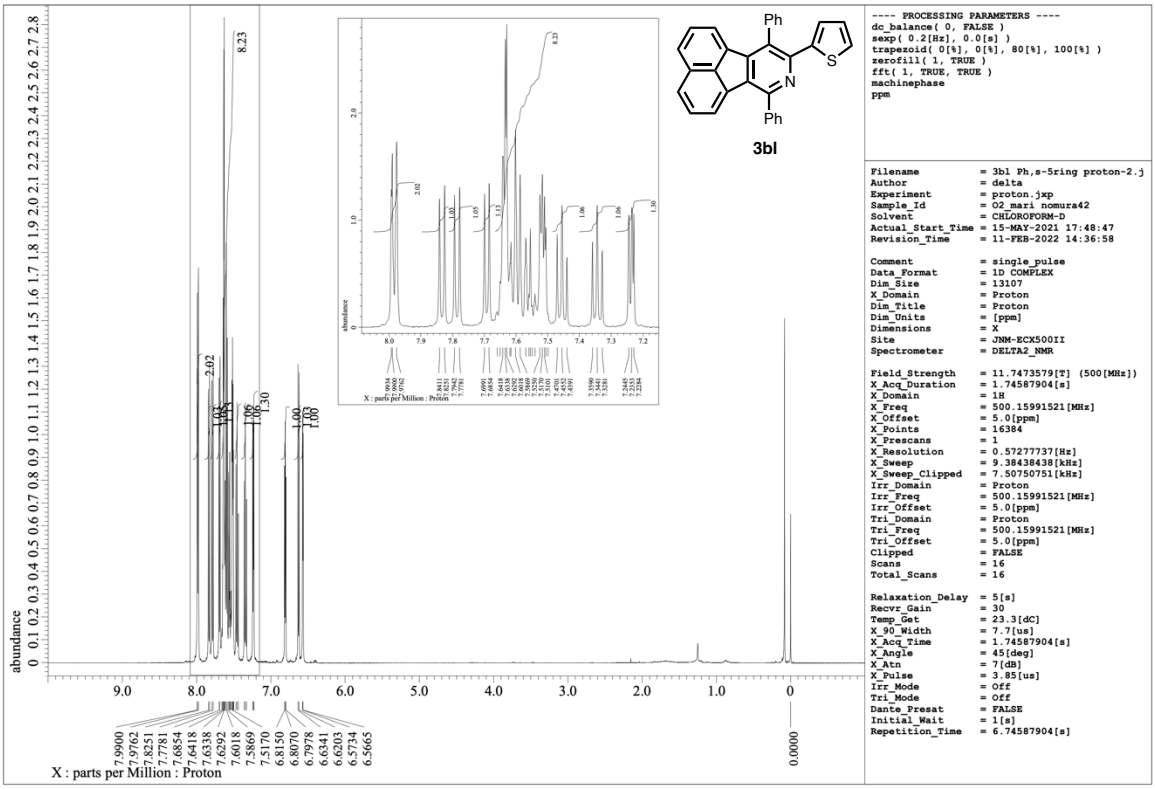

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bj Ph,NPh proton-1.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 02_mari kaho-220
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  4-JUN-2021 14:55:33
Revision_Time       = 11-FEB-2022 14:30:24

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            = 23.5[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 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bj Ph,NPh carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2-kaho220re2
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  6-DEC-2017 10:58:46
Revision_Time       =  6-DEC-2017 12:00:11

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          = 58
Temp_Get            = 22.6[dC]
X_90_Width          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```

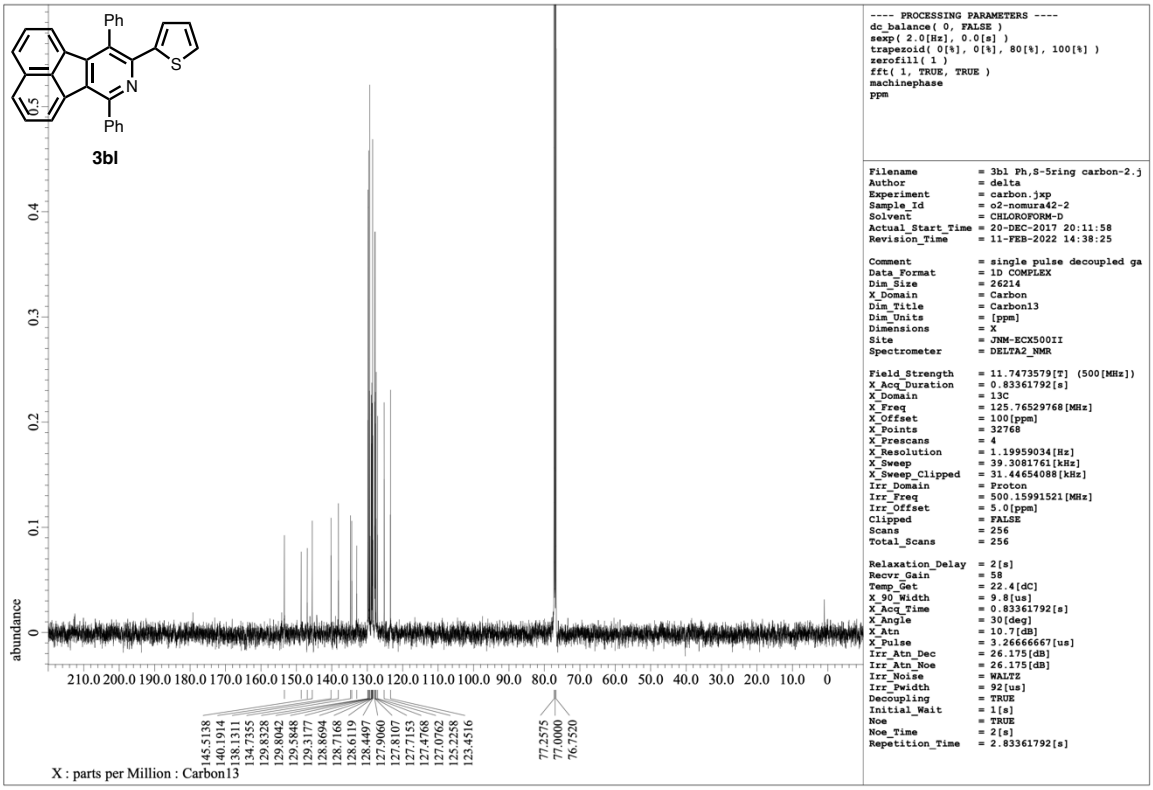

Ph
O
N
Ph
3bk
11.99
2.15
1.12
1.02
1.08
1.03
1.02
1.00
abundance
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.9293 7.9270 7.9133 7.6453 7.6327 7.6006 7.5273 7.5067 7.4918 7.4540 7.2525 6.6639 6.6490 6.2602 6.2573 5.8118 5.8050 0.0000
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Ph,fran_proton-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = 02_mari281 Ph,fran
Solvent = CHLOROFORM-D
Actual_Start_Time = 3-DEC-2021 13:49:19
Revision_Time = 17-DEC-2021 16:22:50
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 = 40
Temp_Get = 22.8[dC]
X_90_Width = 7.4[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 6.5[dB]
X_Pulse = 3.7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]


Ph
O
N
Ph
3bk
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
143.1387 140.4203 129.8042 129.5943 129.3062 129.2223 128.8026 128.7645 128.6214 128.4211 127.9633 127.7343 127.1239 125.2544 123.5566 112.5684 111.2521 77.2575 77.0000 76.7520
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
Filename = 3bk Ph,O-5ring carbon-1.j
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-nomura-re-39
Solvent = CHLOROFORM-D
Actual_Start_Time = 21-DEC-2017 01:26:13
Revision_Time = 14-MAY-2021 10:17:50
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 = 58
Temp_Get = 22.6[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]



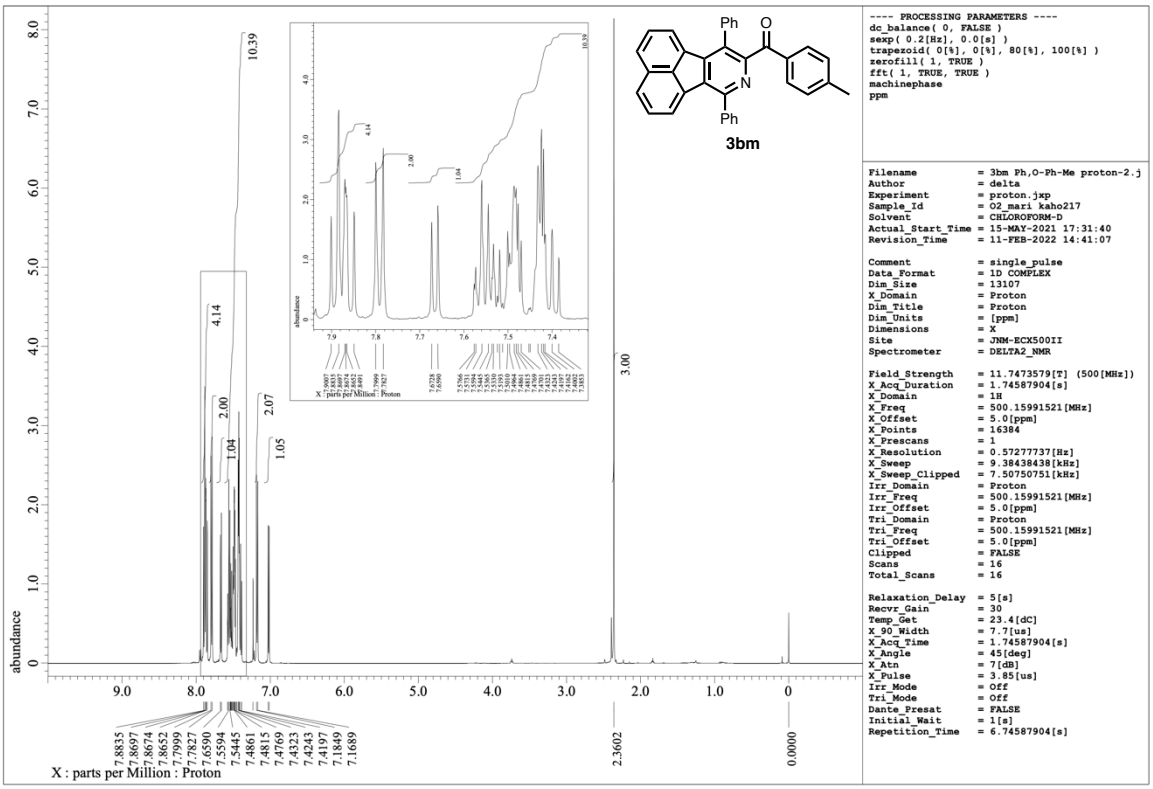

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename            = 3bl Ph,s-5ring proton-2.j
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari nomura42
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-MAY-2021 17:48:47
Revision_Time       = 11-FEB-2022 14:36:58

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            = 23.3[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]
```

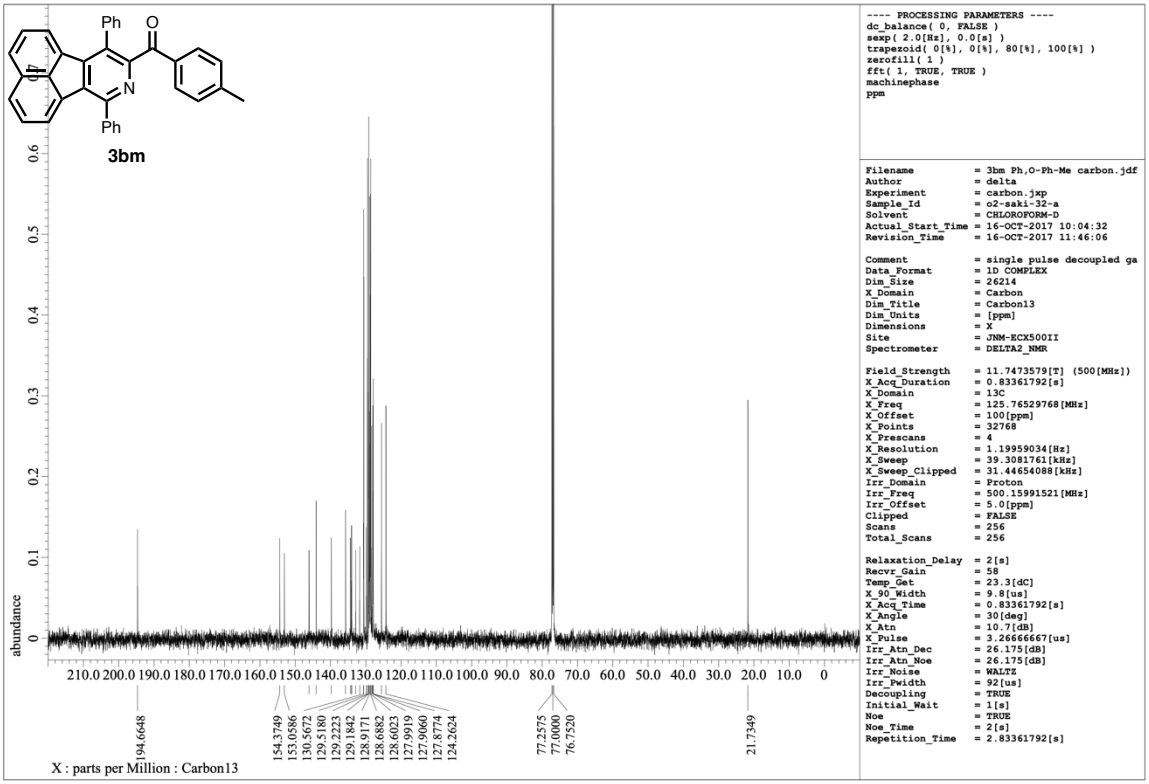



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

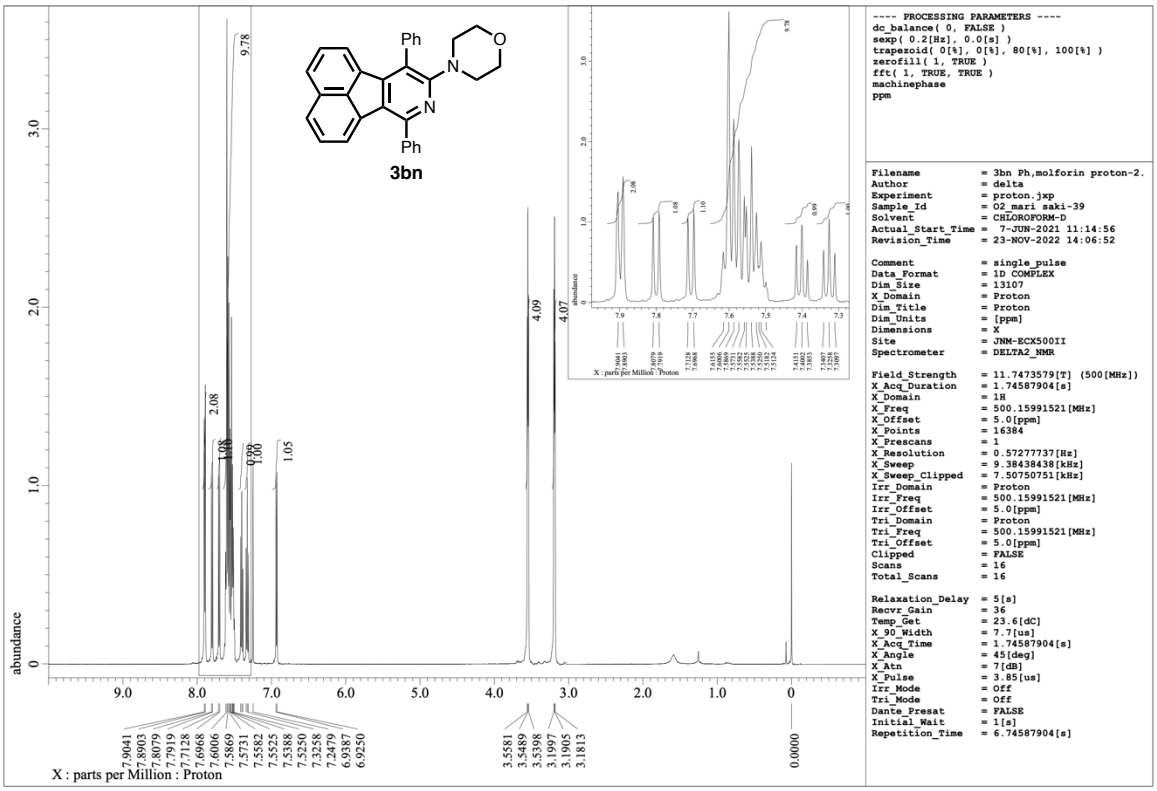
```
Filename            = 3bl Ph,S-5ring carbon-2.j
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2-nomura42-2
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 20-DEC-2017 20:11:58
Revision_Time       = 11-FEB-2022 14:38:25

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          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```



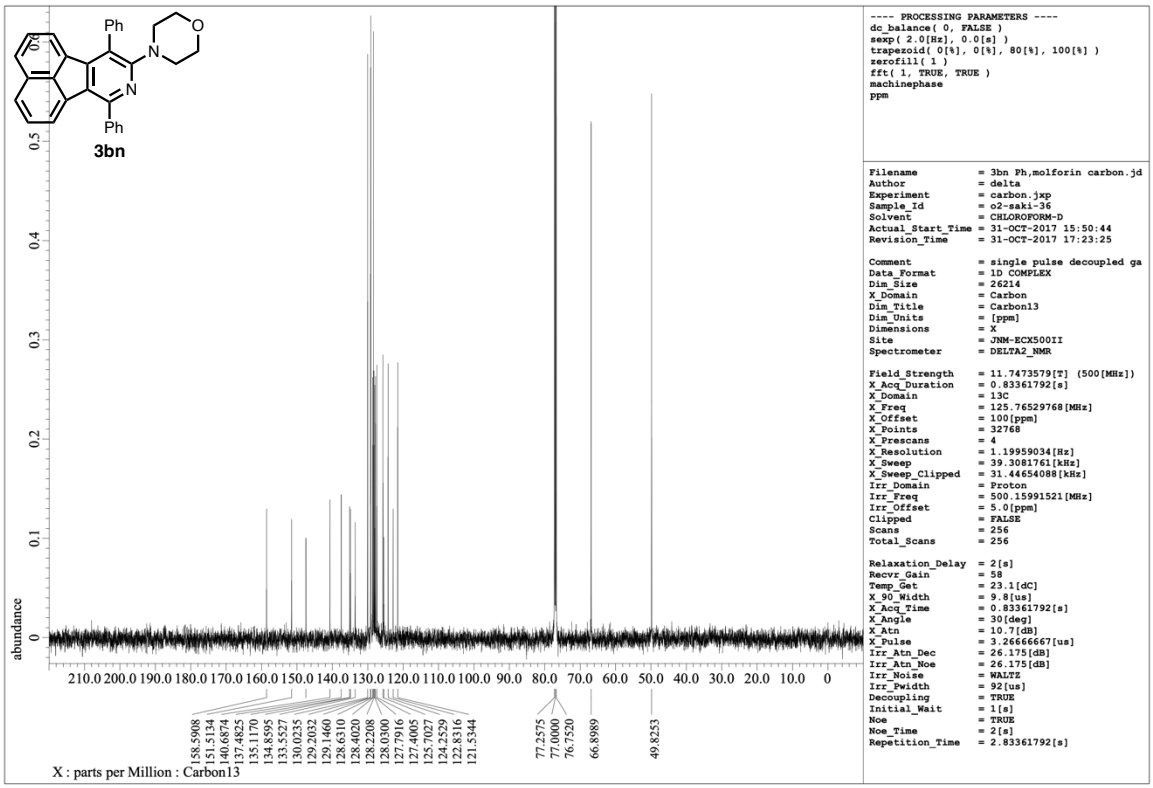

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bm Ph,O-Ph-Me proton-2.j
Author              = delta
Experiment          = proton.jxp
Sample_Id           = 02_mari kaho217
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 15-MAY-2021 17:31:40
Revision_Time       = 11-FEB-2022 14:41:07

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          = 30
Temp_Get            = 23.4[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]
```



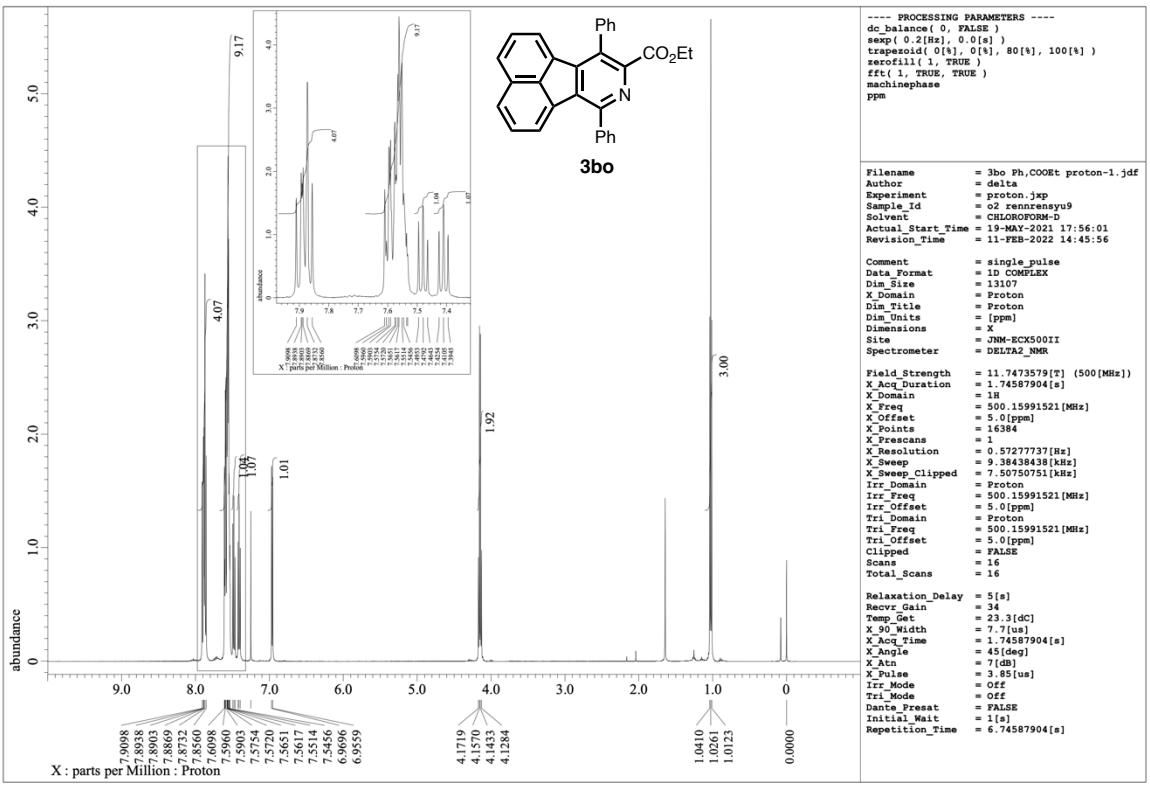

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bm Ph,O-Ph-Me carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2-saki-32-a
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 16-OCT-2017 10:04:32
Revision_Time       = 16-OCT-2017 11:46:06

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            = 23.3[dC]
X_90_Width          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```



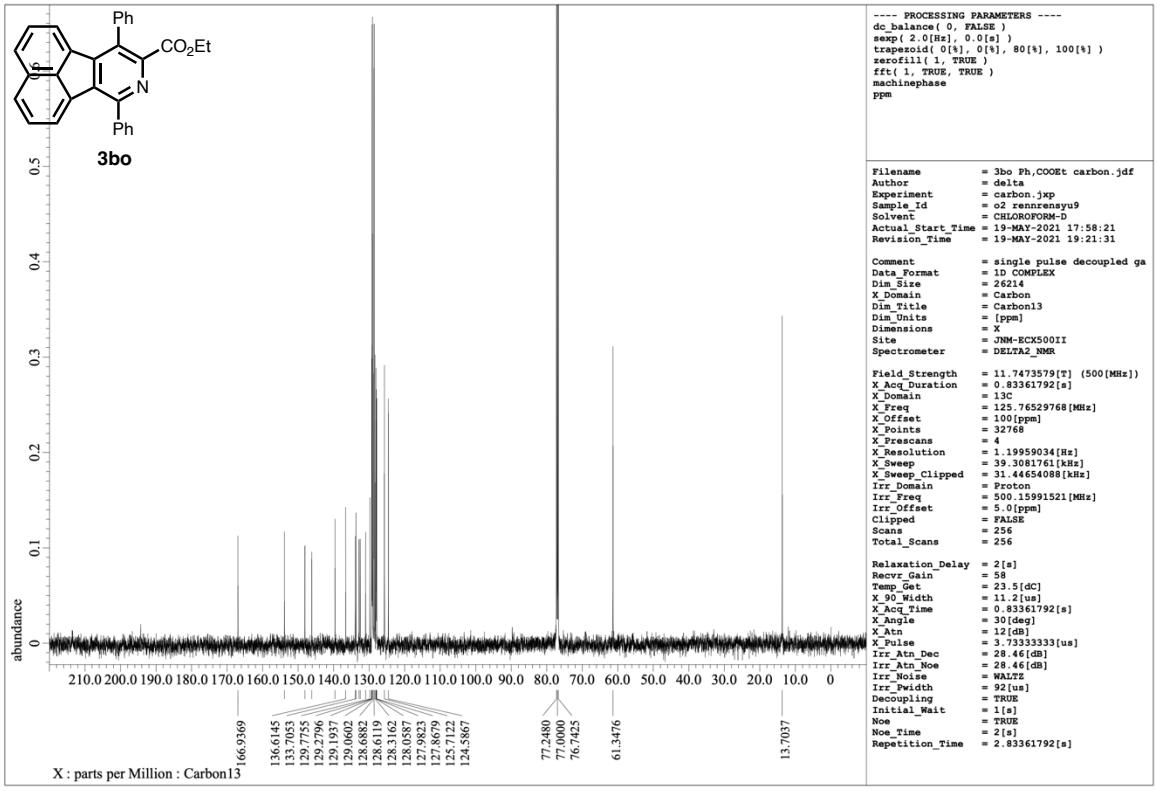

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bn Ph,molforin proton-2.
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari saki-39
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-JUN-2021 11:14:56
Revision_Time       = 23-NOV-2022 14:06:52

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            = 23.6[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]
```



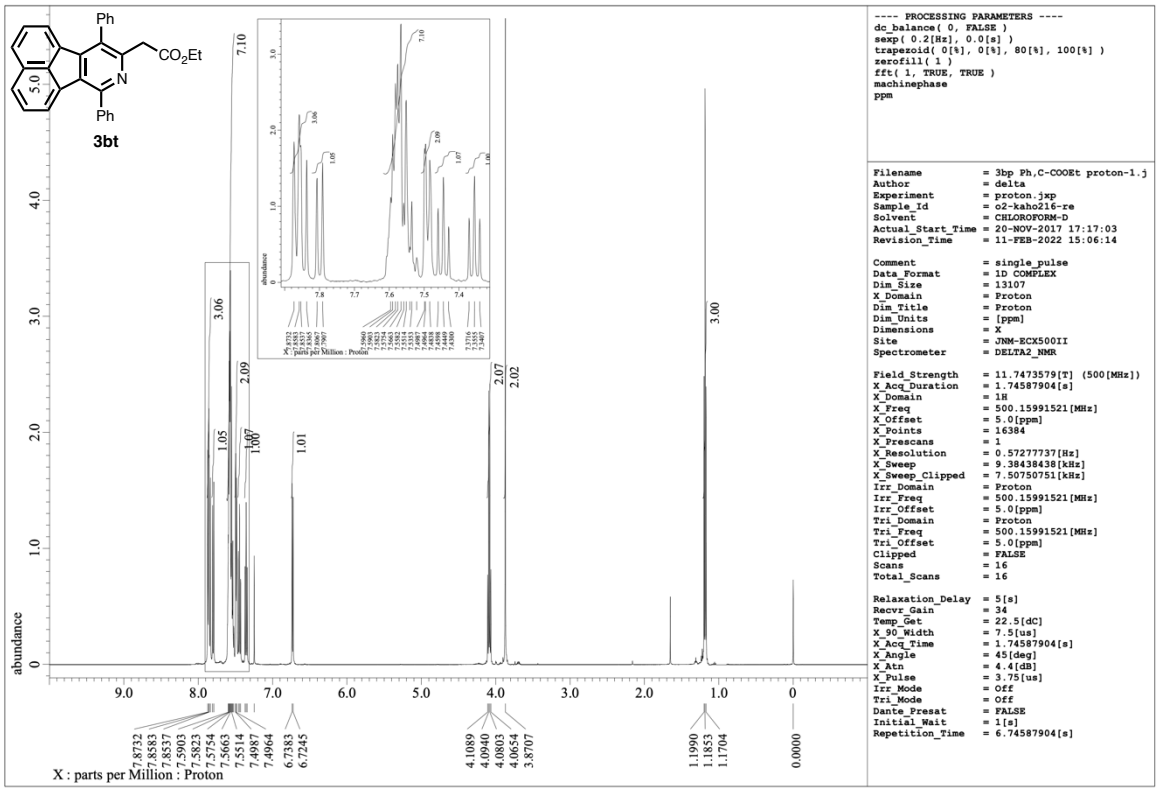

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bn Ph,molforin carbon.jd
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2-saki-36
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 31-OCT-2017 15:50:44
Revision_Time       = 31-OCT-2017 17:23:25

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            = 23.1[dC]
X_90_Width          = 9.8[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 10.7[dB]
X_Pulse             = 3.26666667[us]
Irr_Atn_Dec         = 26.175[dB]
Irr_Atn_Noe         = 26.175[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]
```

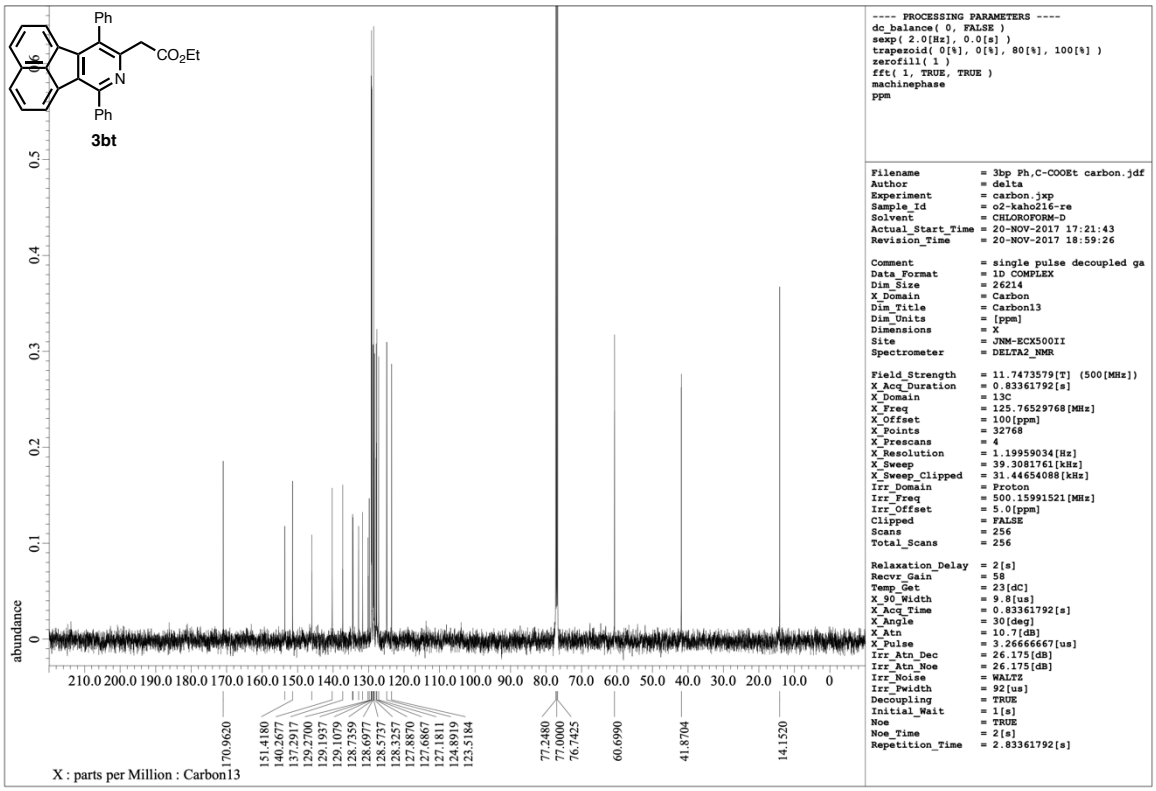


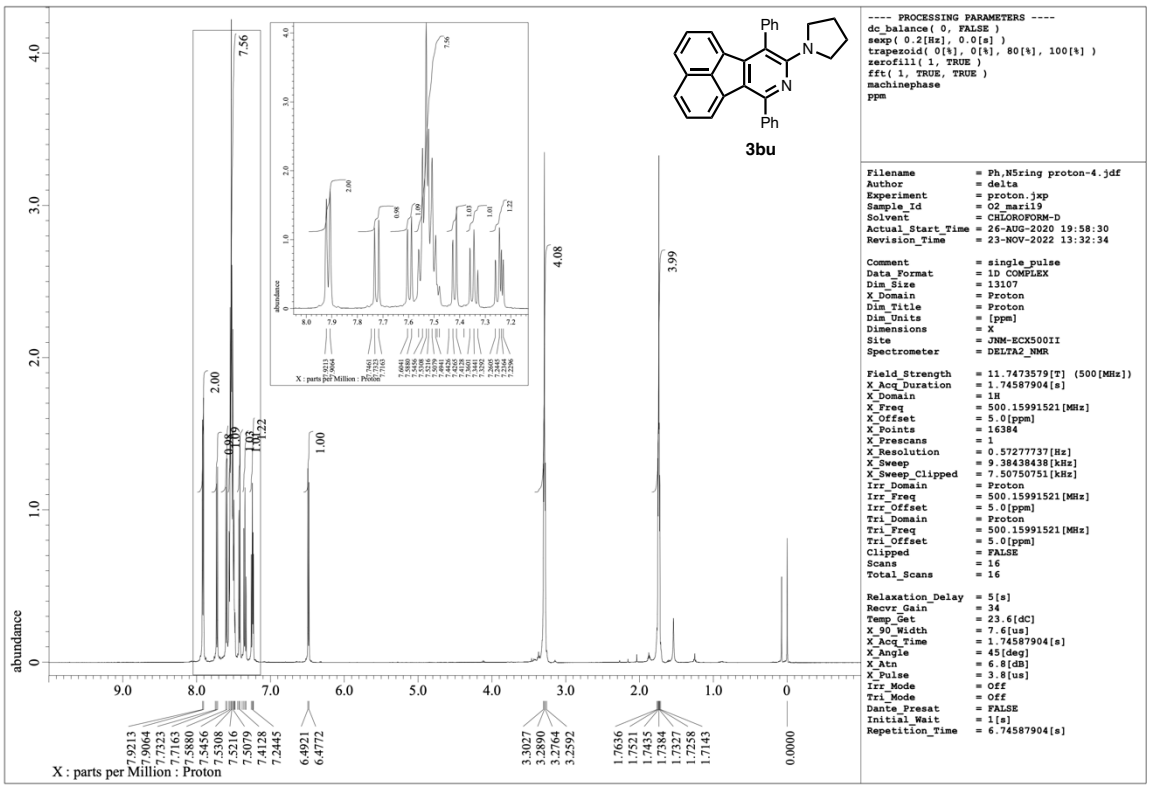

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bo Ph,COOEt proton-1.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2 rennrensyu9
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 19-MAY-2021 17:56:01
Revision_Time       = 11-FEB-2022 14:45:56

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            = 23.3[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]
```

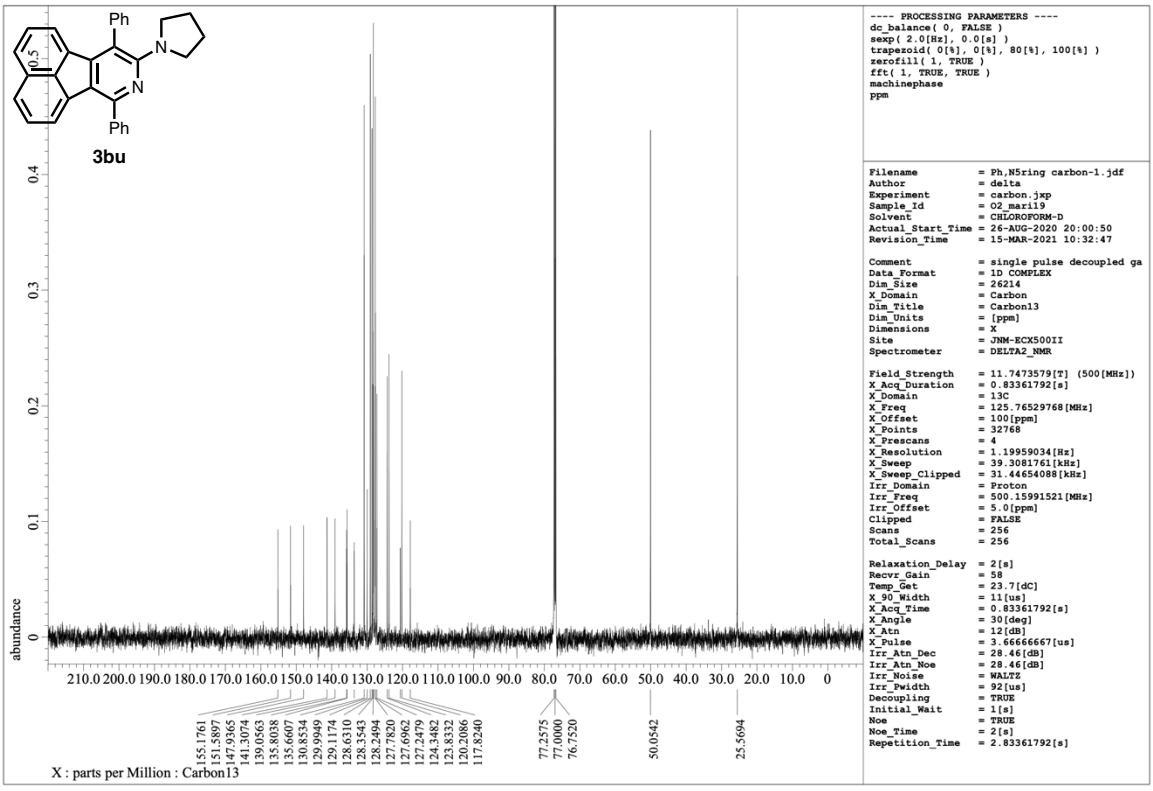


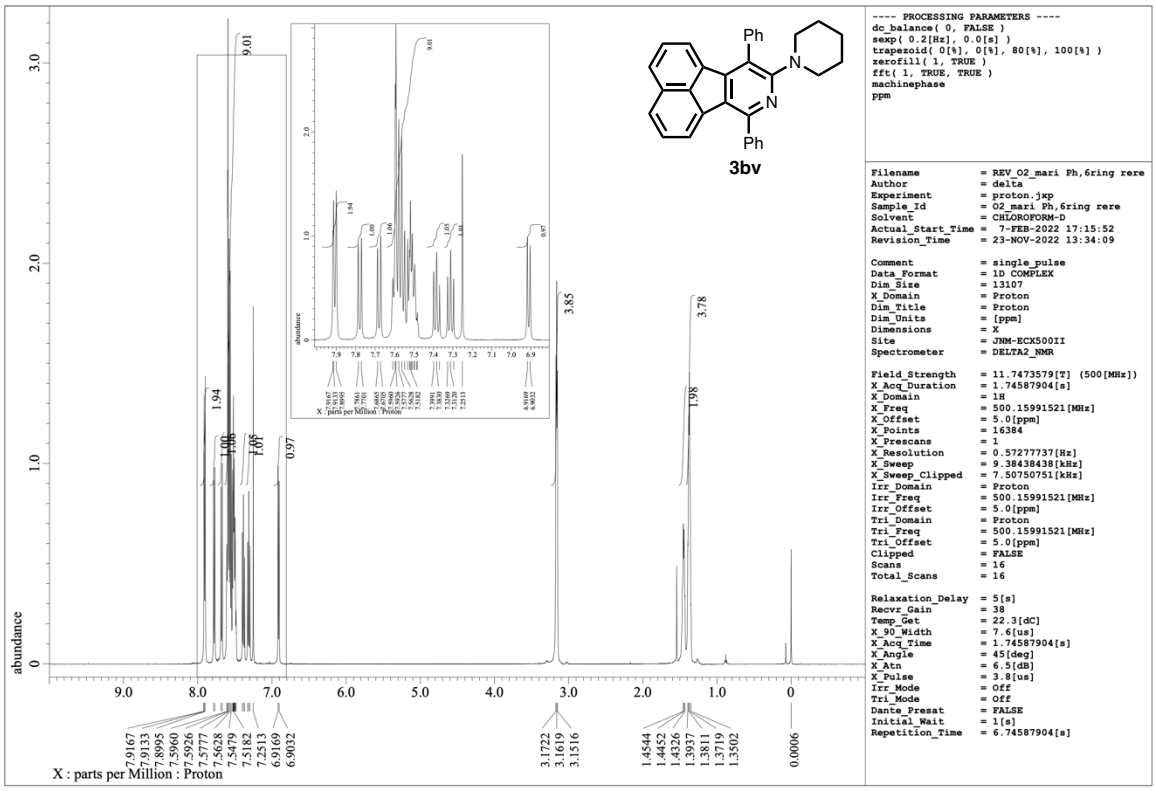

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 3bo Ph,COOEt carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2 rennrensyu9
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 19-MAY-2021 17:58:21
Revision_Time       = 19-MAY-2021 19:21:31

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            = 23.5[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]
```


Ph
CO2Et
N
Ph
3bt
5.0
7.10
3.06
2.09
1.05
1.07
1.00
1.01
2.07
2.02
3.00
abundance
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.8732 7.8583 7.8537 7.5903 7.5823 7.5754 7.5663 7.5514 7.4987 7.4964 6.7383 6.7245
4.1089 4.0940 4.0803 4.0654 3.8707
1.1990 1.1853 1.1704
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
Filename = 3bp Ph,C-COOEt proton-1.j
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kaho216-re
Solvent = CHLOROFORM-D
Actual_Start_Time = 20-NOV-2017 17:17:03
Revision_Time = 11-FEB-2022 15:06:14
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 = 22.5[dC]
X_90_Width = 7.5[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 4.4[dB]
X_Pulse = 3.75[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]

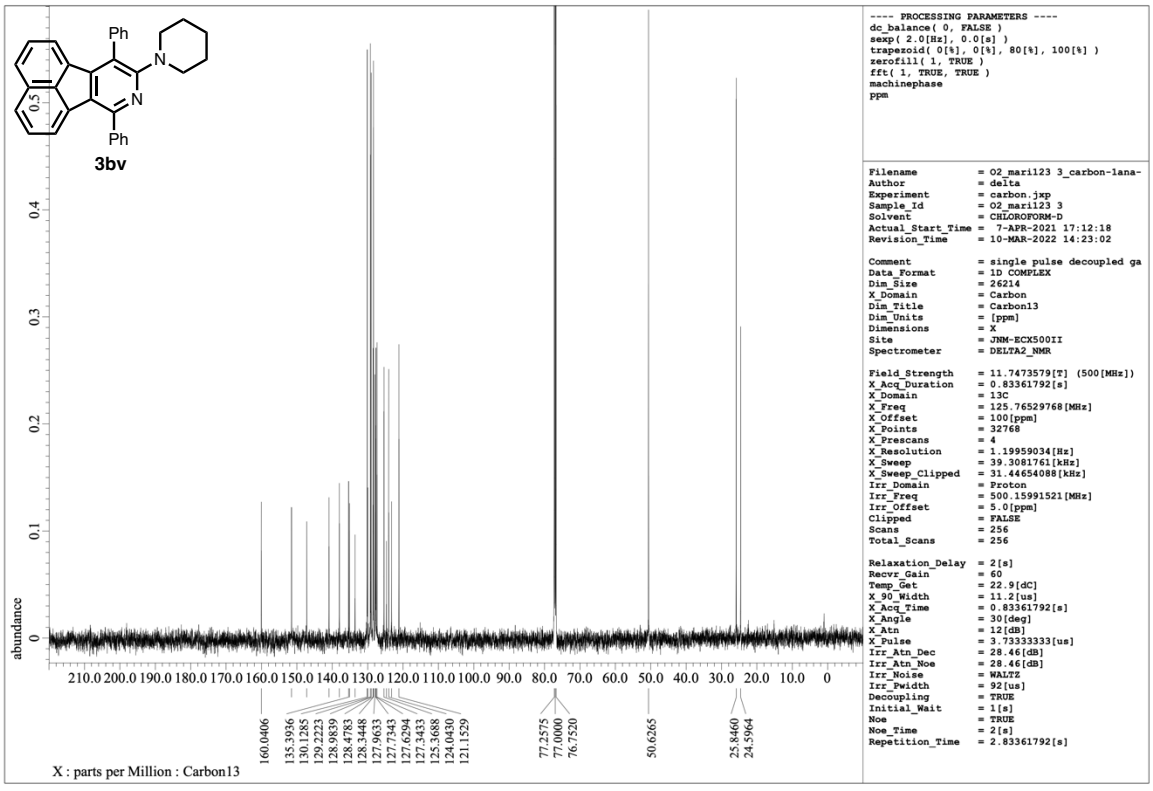

Ph
CO2Et
N
Ph
3bt
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
170.9620
151.4180 140.2677 137.2917 129.2700 129.1937 129.1079 128.7359 128.6977 128.5737 128.3257 127.8870 127.6867 127.1811 124.8919 123.5184
77.2480 77.0000 76.7425
60.6990
41.8704
14.1520
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
Filename = 3bp Ph,C-COOEt carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-kaho216-re
Solvent = CHLOROFORM-D
Actual_Start_Time = 20-NOV-2017 17:21:43
Revision_Time = 20-NOV-2017 18:59:26
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 = 23[dC]
X_90_Width = 9.8[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 10.7[dB]
X_Pulse = 3.26666667[us]
Irr_Atn_Dec = 26.175[dB]
Irr_Atn_Noe = 26.175[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]


Ph
N
N
Ph
3bu
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 = Ph,N5ring proton-4.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari19
Solvent = CHLOROFORM-D
Actual_Start_Time = 26-AUG-2020 19:58:30
Revision_Time = 23-NOV-2022 13:32:34
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 = 23.6[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]

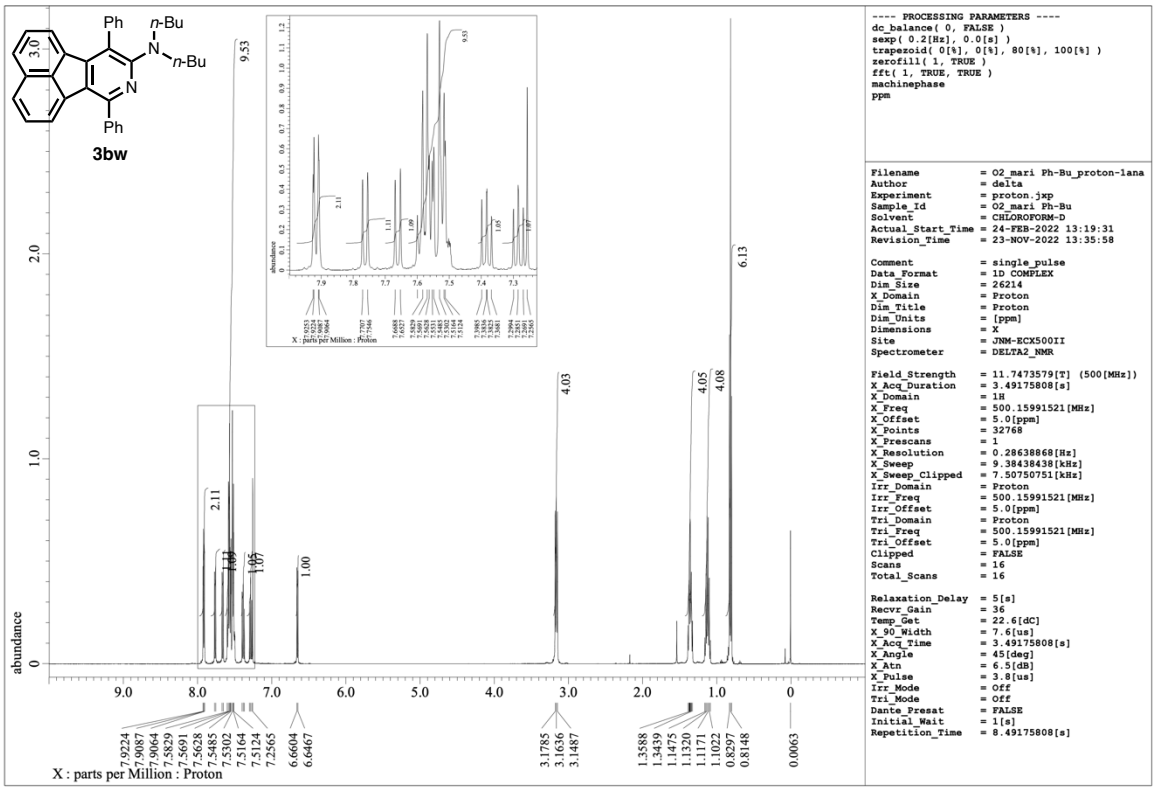

Ph
N
N
Ph
3bu
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Ph,N5ring carbon-1.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari19
Solvent = CHLOROFORM-D
Actual_Start_Time = 26-AUG-2020 20:00:50
Revision_Time = 15-MAR-2021 10:32:47
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 = 58
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 = 28.46[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]

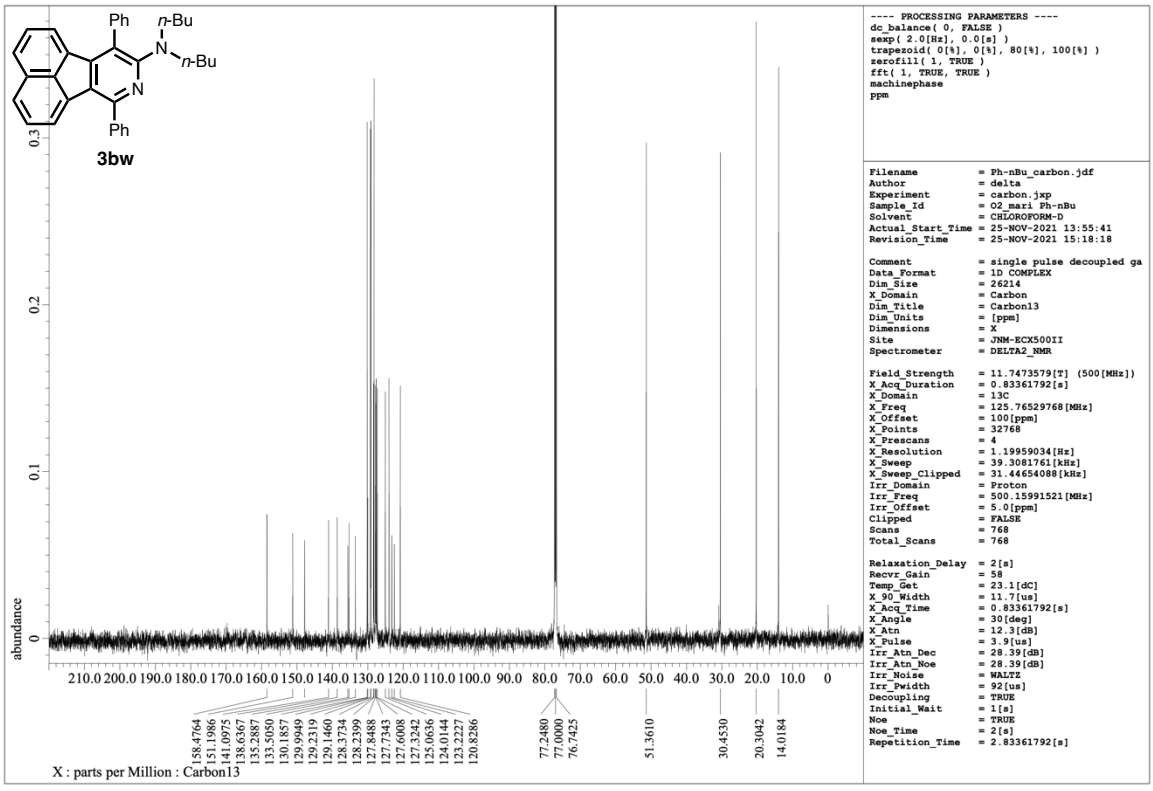


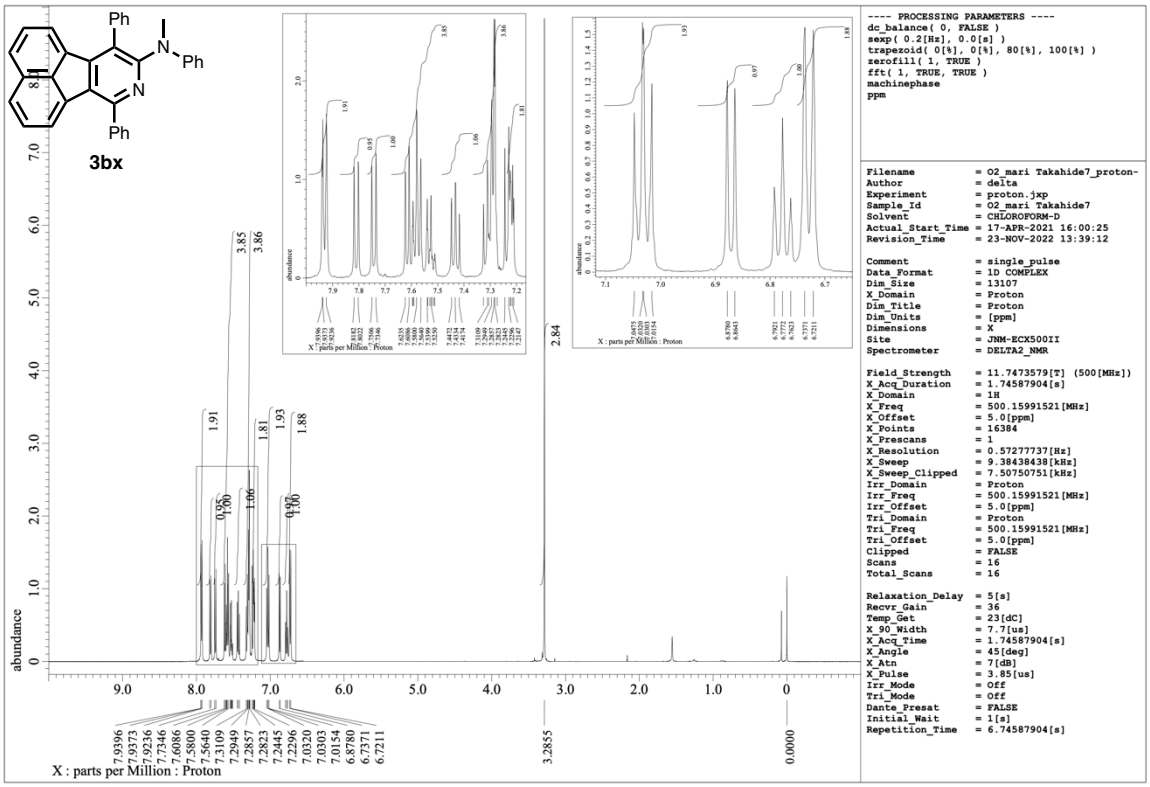

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = REV_O2_mari Ph,6ring rere
Author              = delta
Experiment          = proton.jxp
Sample_Id           = O2_mari Ph,6ring rere
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-FEB-2022 17:15:52
Revision_Time       = 23-NOV-2022 13:34:09

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          = 38
Temp_Get            = 22.3[dC]
X_90_Width          = 7.6[us]
X_Acq_Time          = 1.74587904[s]
X_Angle             = 45[deg]
X_Atn               = 6.5[dB]
X_Pulse             = 3.8[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 6.74587904[s]
```

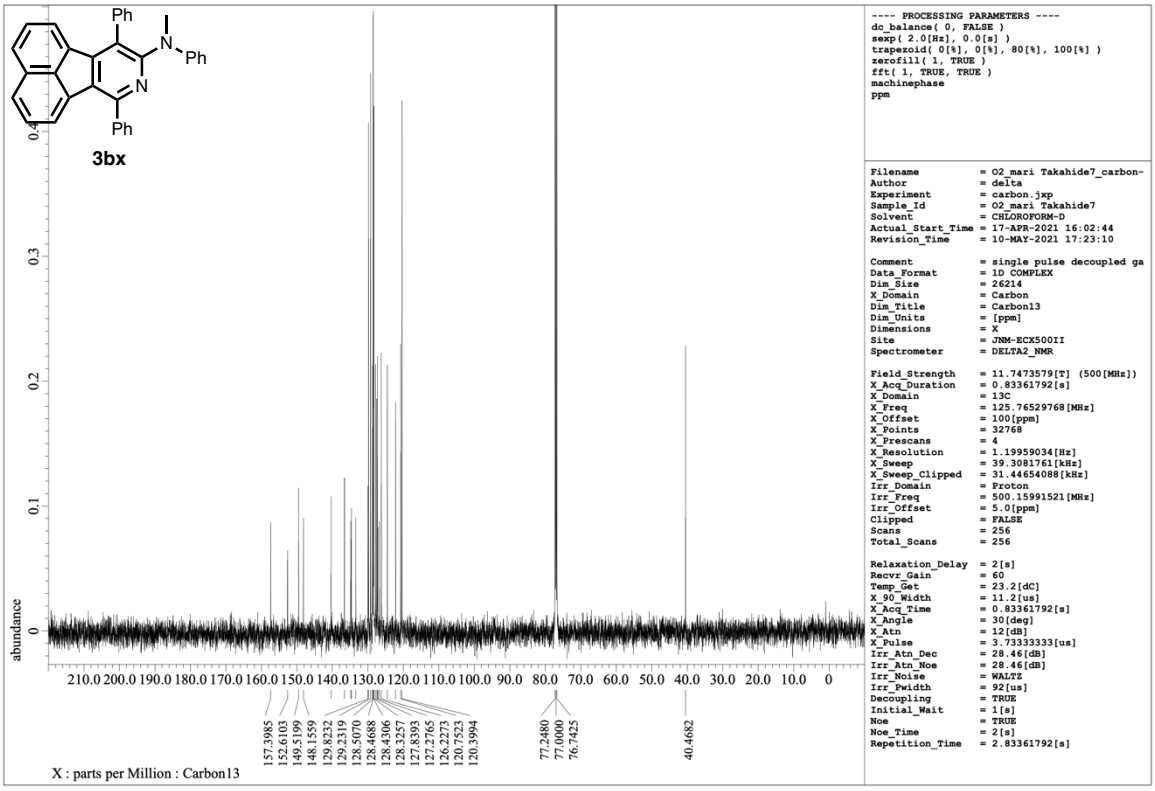


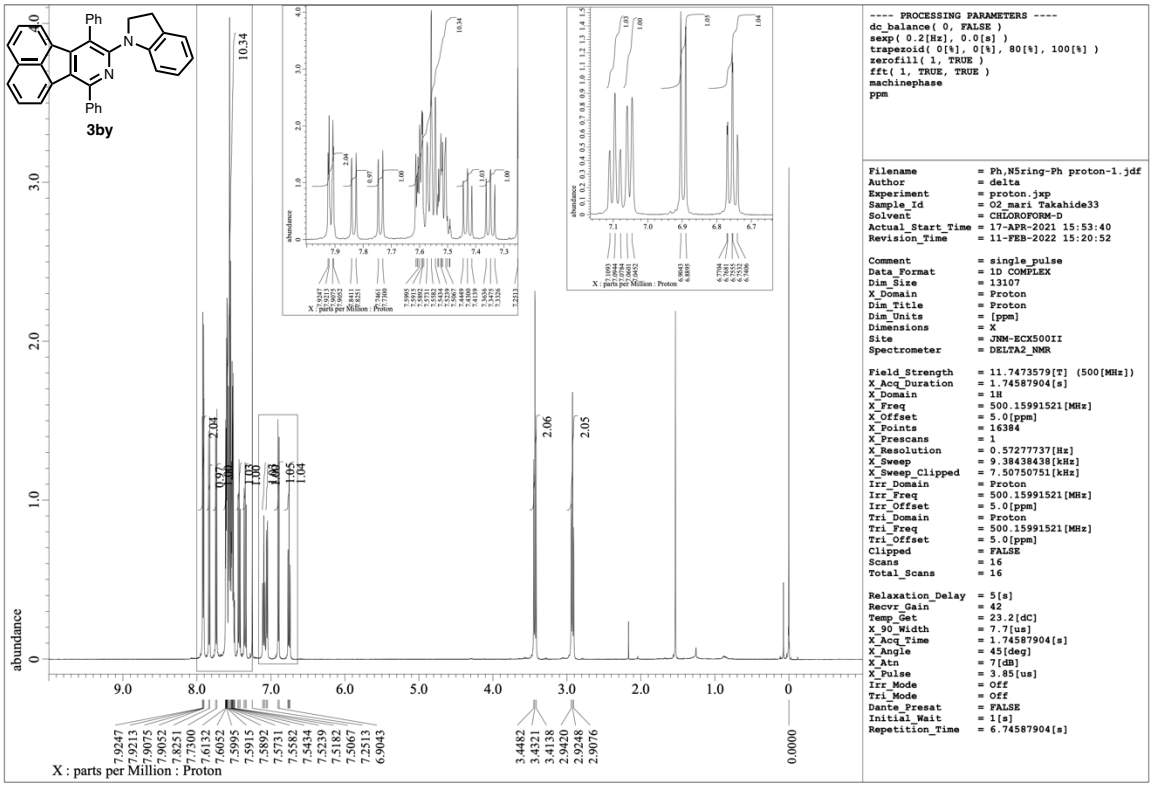

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = O2_mari123 3_carbon-lana-
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari123 3
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  7-APR-2021 17:12:18
Revision_Time       = 10-MAR-2022 14:23:02

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            = 22.9[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]
```

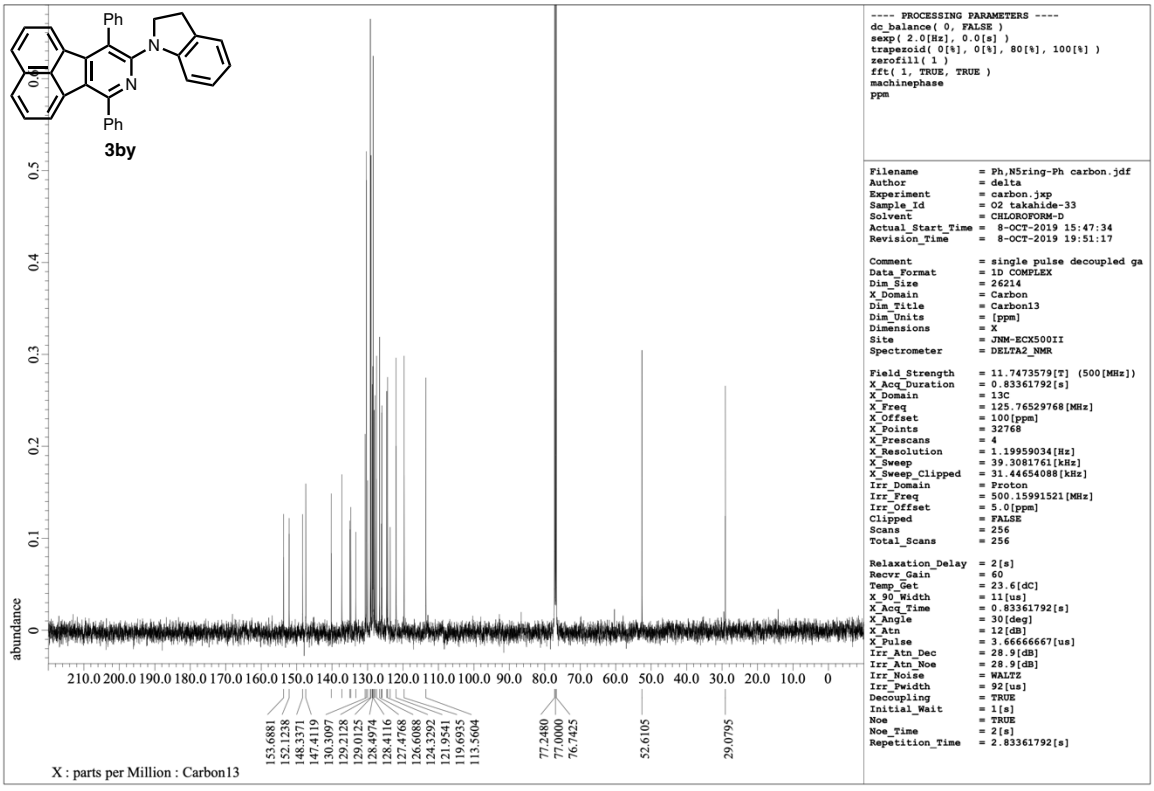

Ph
n-Bu
N
n-Bu
N
Ph
3bw
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari Ph-Bu_proton-1ana
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari Ph-Bu
Solvent = CHLOROFORM-D
Actual_Start_Time = 24-FEB-2022 13:19:31
Revision_Time = 23-NOV-2022 13:35:58
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
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 = 3.49175808[s]
X_Domain = 1H
X_Freq = 500.15991521[MHz]
X_Offset = 5.0[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.28638868[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 = 7.6[us]
X_Acq_Time = 3.49175808[s]
X_Angle = 45[deg]
X_Atn = 6.5[dB]
X_Pulse = 3.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 8.49175808[s]

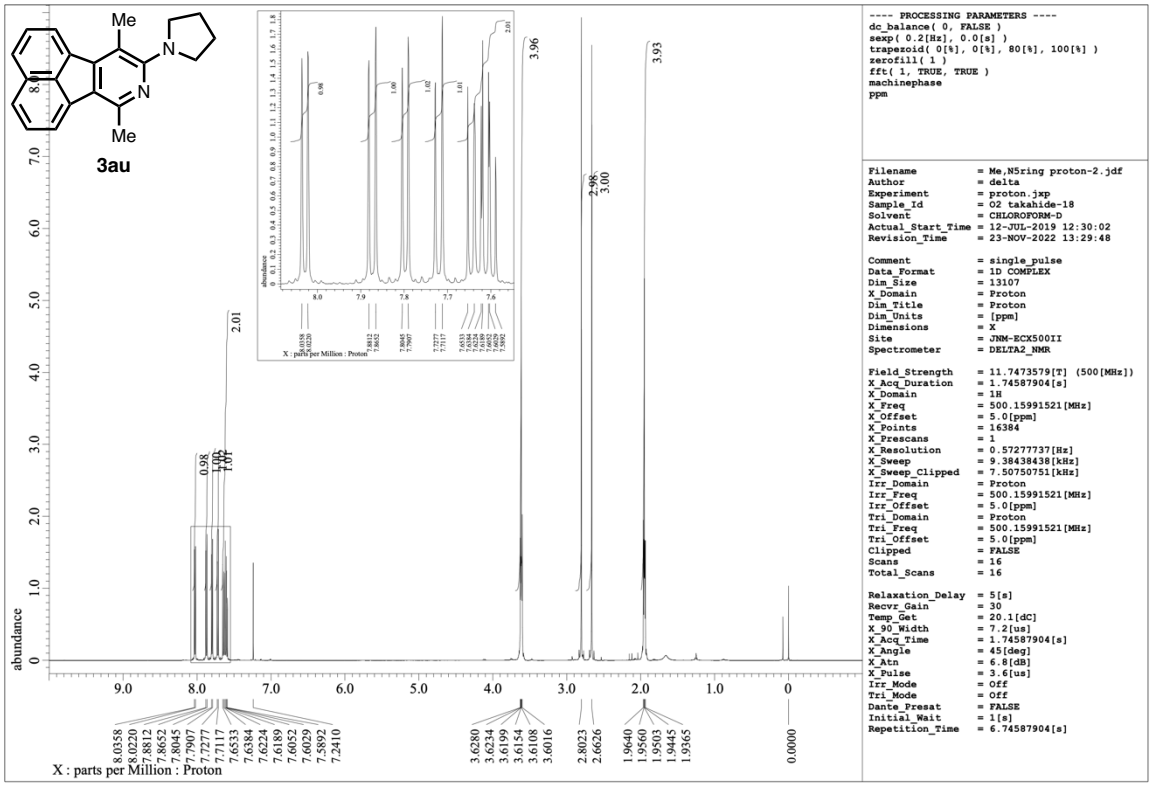

Ph
n-Bu
N
n-Bu
N
Ph
3bw
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Ph-nBu_carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari Ph-nBu
Solvent = CHLOROFORM-D
Actual_Start_Time = 25-NOV-2021 13:55:41
Revision_Time = 25-NOV-2021 15:18:18
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 = 768
Total_Scans = 768
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 23.1[dC]
X_90_Width = 11.7[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12.3[dB]
X_Pulse = 3.9[us]
Irr_Atn_Dec = 28.39[dB]
Irr_Atn_Noe = 28.39[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]

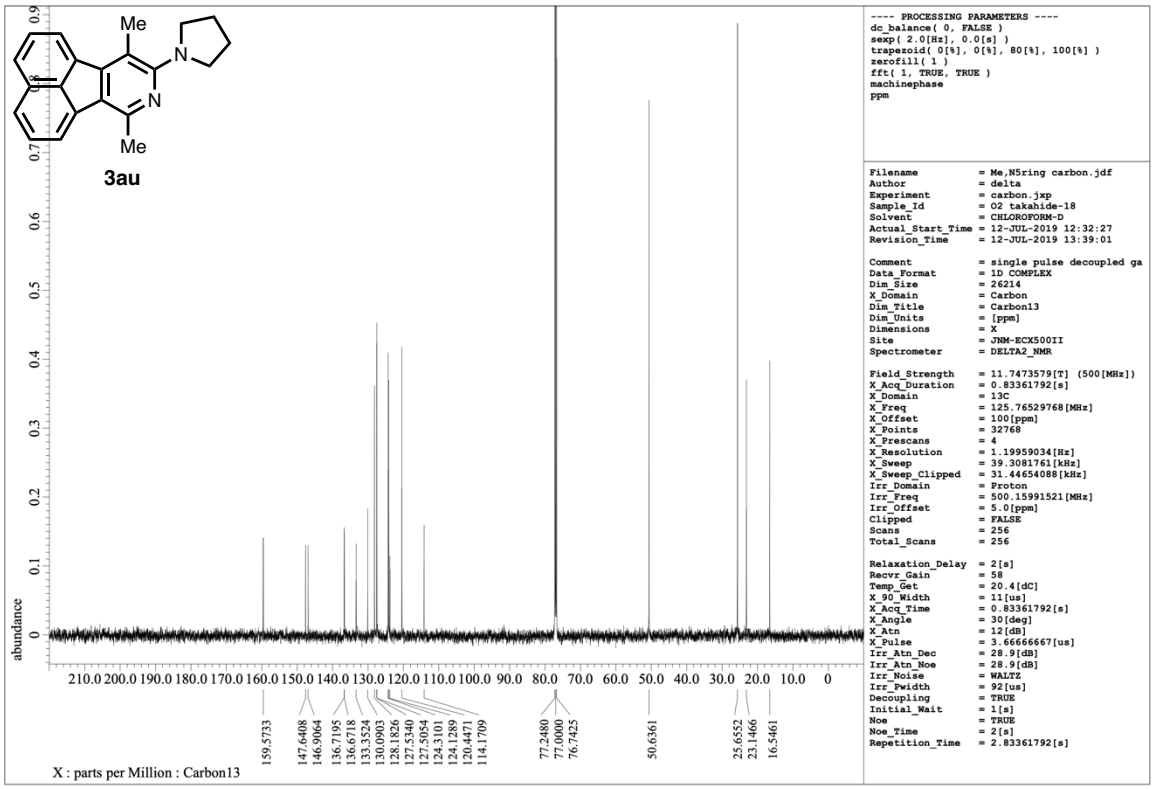

Ph
N
Ph
N
Ph
3bx
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari Takahide7_proton-
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari Takahide7
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-APR-2021 16:00:25
Revision_Time = 23-NOV-2022 13:39:12
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 = 23[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]

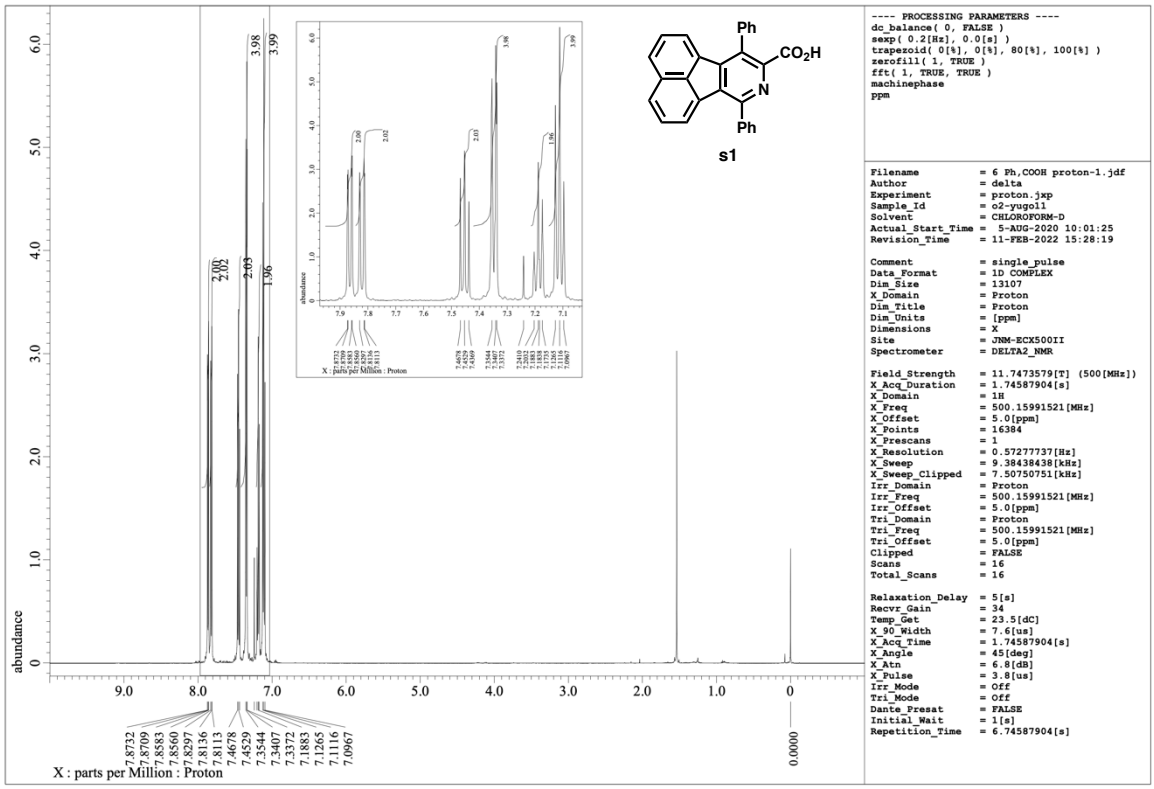

Ph
N
Ph
N
Ph
3bx
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1, TRUE)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = O2_mari Takahide7_carbon-
Author = delta
Experiment = carbon.jxp
Sample_Id = O2_mari Takahide7
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-APR-2021 16:02:44
Revision_Time = 10-MAY-2021 17:23:10
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 = 23.2[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]


Ph
N
N
Ph
3by
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 = Ph,N5ring-Ph proton-1.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = O2_mari Takahide33
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-APR-2021 15:53:40
Revision_Time = 11-FEB-2022 15:20:52
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 = 42
Temp_Get = 23.2[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]

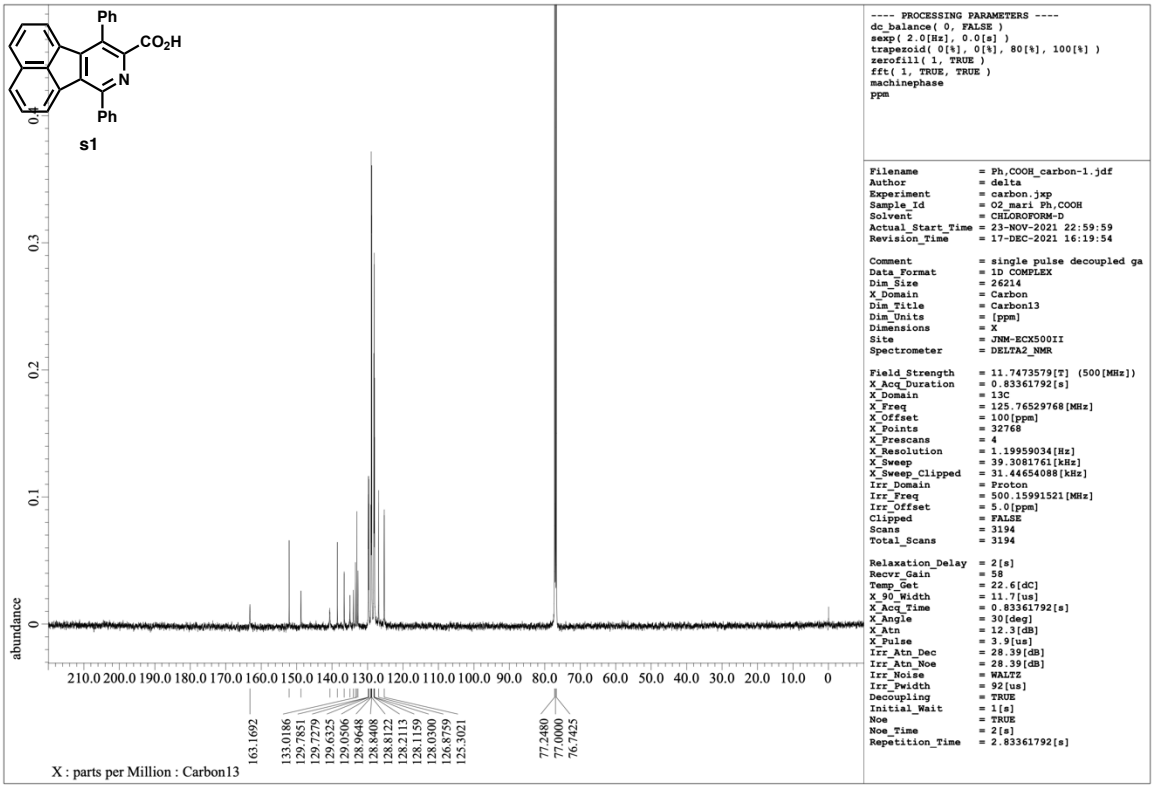

Ph
N
N
Ph
3by
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Ph,N5ring-Ph carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2 takahide-33
Solvent = CHLOROFORM-D
Actual_Start_Time = 8-OCT-2019 15:47:34
Revision_Time = 8-OCT-2019 19:51:17
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]


Me
N
Me
3au
X : parts per Million : Proton
abundance
---- 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
Filename = Me,N5ring proton-2.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = O2 takahide-18
Solvent = CHLOROFORM-D
Actual_Start_Time = 12-JUL-2019 12:30:02
Revision_Time = 23-NOV-2022 13:29:48
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 = 30
Temp_Get = 20.1[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]

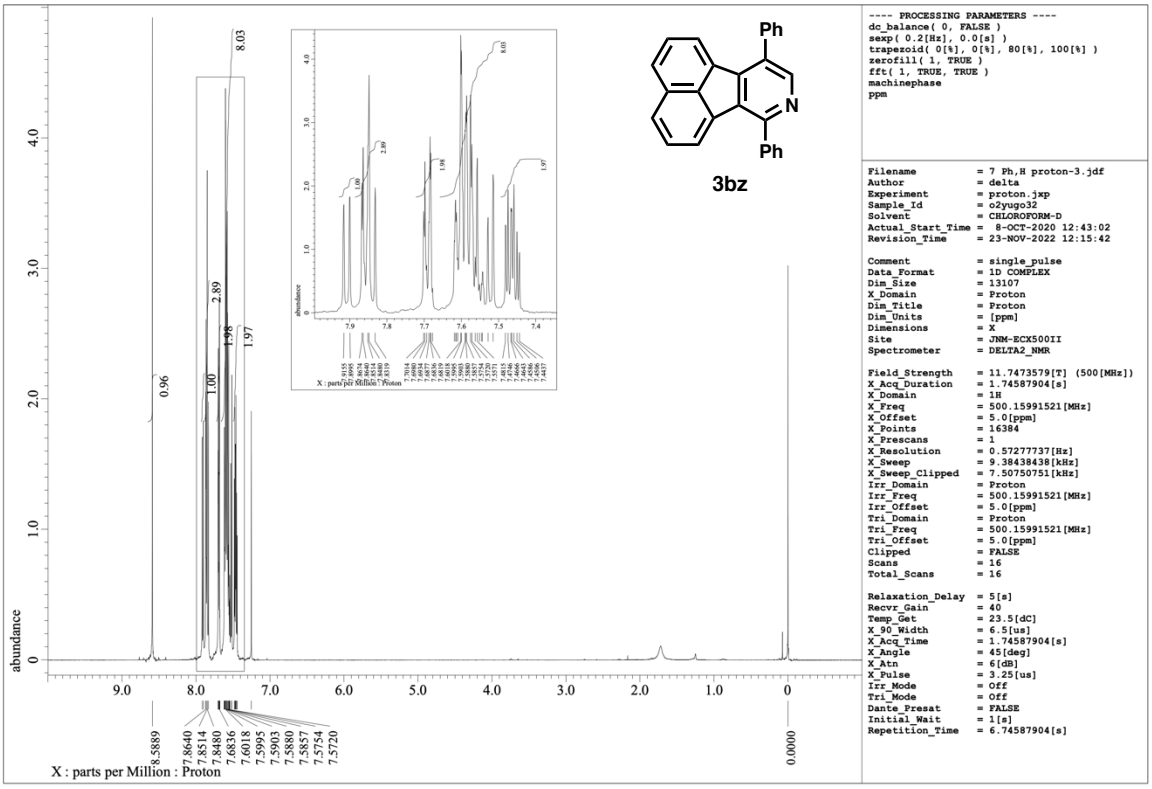

Me
N
Me
3au
X : parts per Million : Carbon13
abundance
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = Me,N5ring carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = O2 takahide-18
Solvent = CHLOROFORM-D
Actual_Start_Time = 12-JUL-2019 12:32:27
Revision_Time = 12-JUL-2019 13:39:01
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 = 20.4[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]



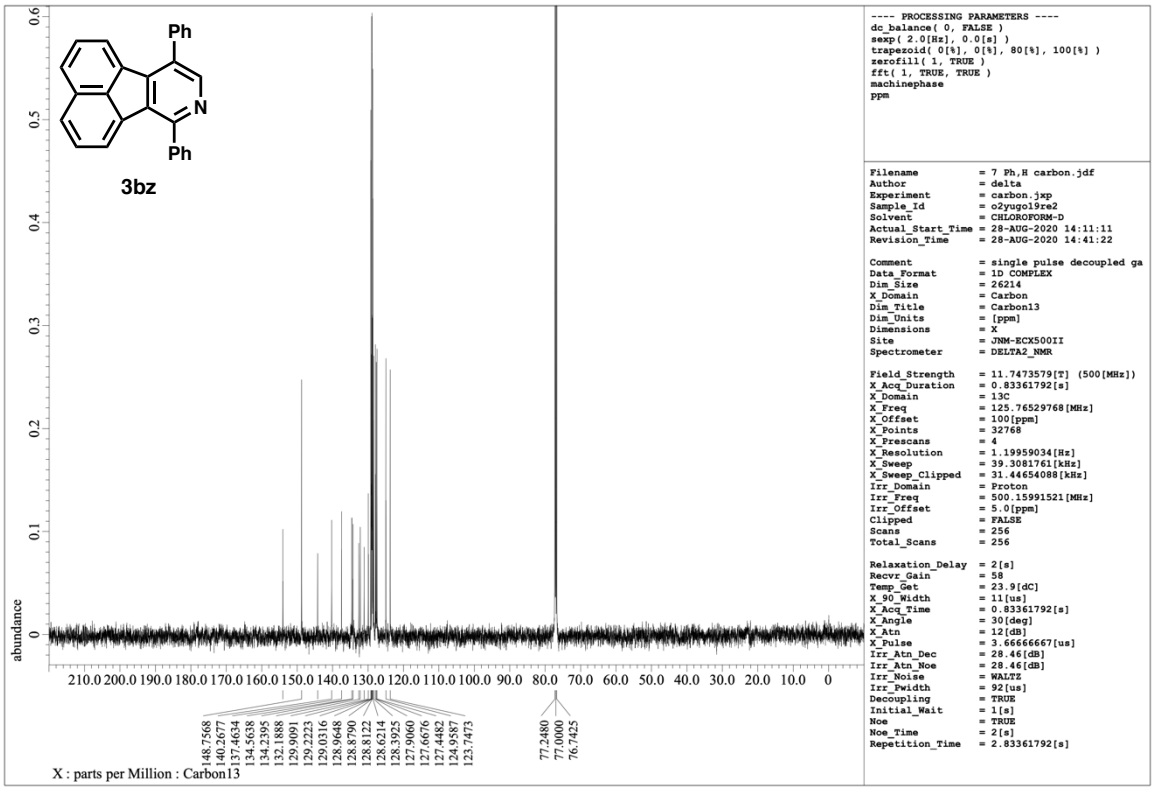

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 6 Ph,COOH proton-1.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2-yugo11
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  5-AUG-2020 10:01:25
Revision_Time       = 11-FEB-2022 15:28:19

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            = 23.5[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]
```



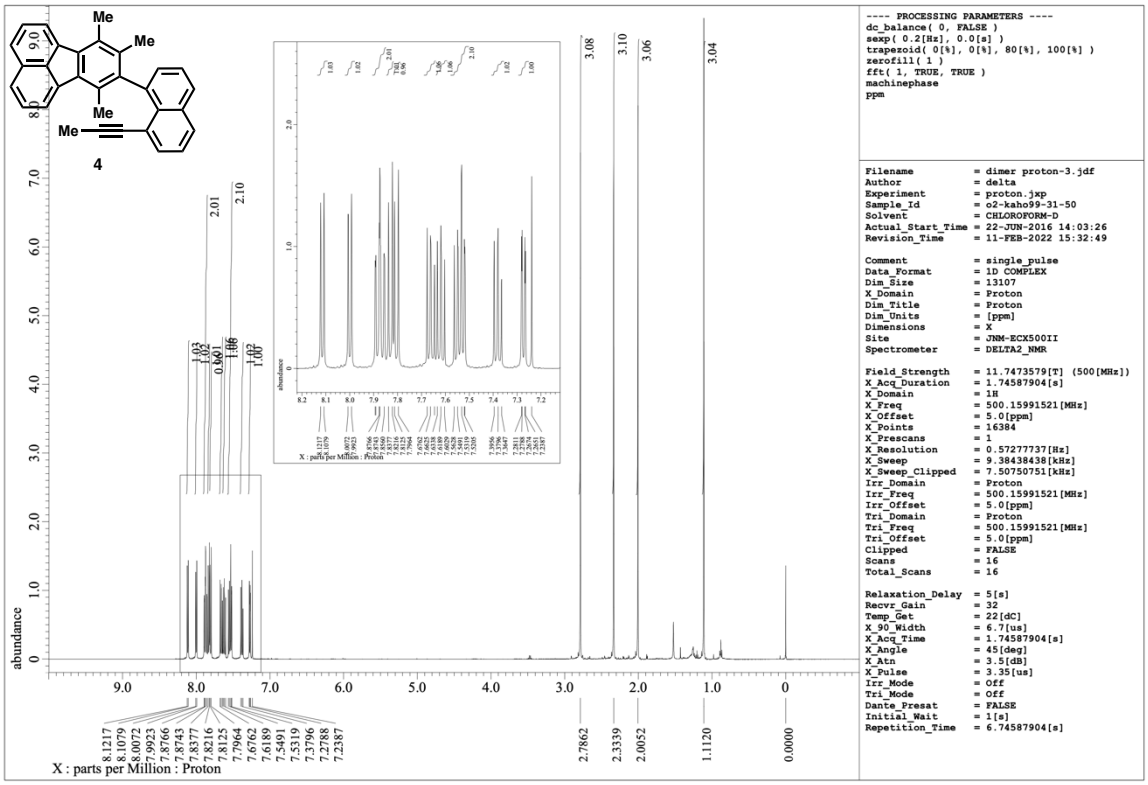

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = Ph,COOH_carbon-1.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = O2_mari Ph,COOH
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 23-NOV-2021 22:59:59
Revision_Time       = 17-DEC-2021 16:19:54

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               = 3194
Total_Scans         = 3194

Relaxation_Delay    = 2[s]
Recvr_Gain          = 58
Temp_Get            = 22.6[dC]
X_90_Width          = 11.7[us]
X_Acq_Time          = 0.83361792[s]
X_Angle             = 30[deg]
X_Atn               = 12.3[dB]
X_Pulse             = 3.9[us]
Irr_Atn_Dec         = 28.39[dB]
Irr_Atn_Noe         = 28.39[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]
```

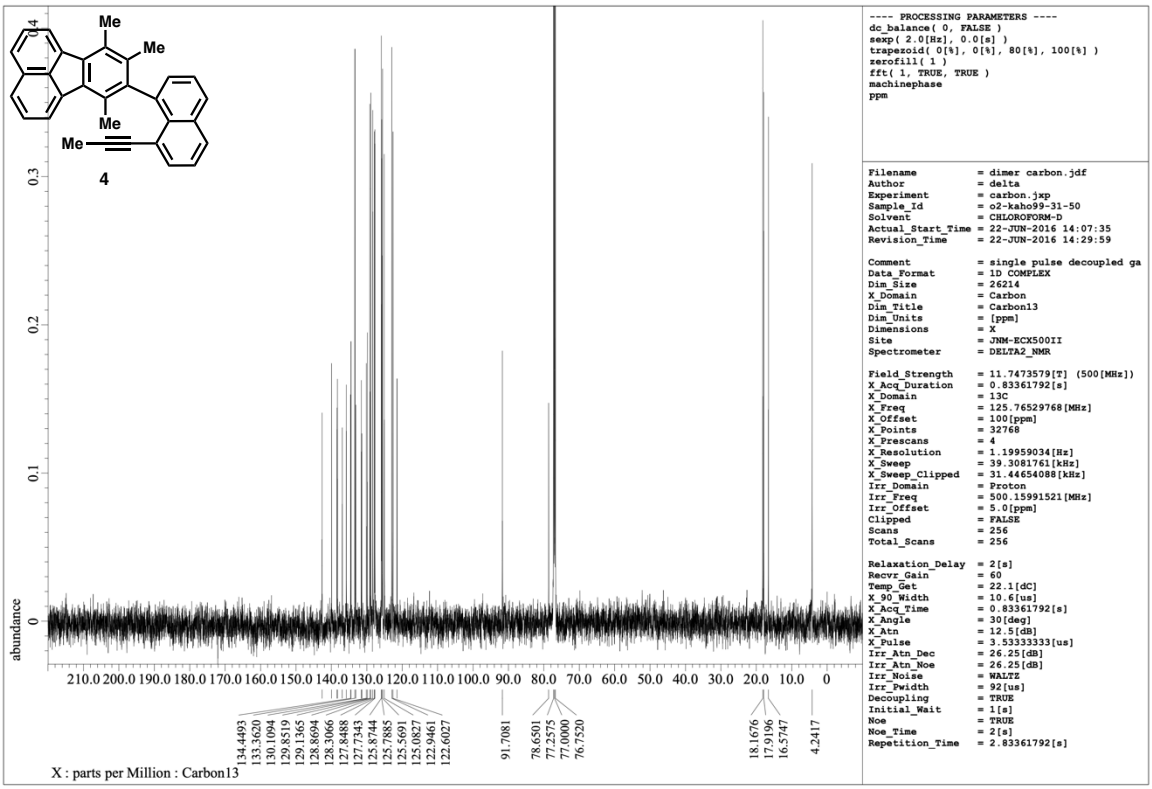



```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1, TRUE )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = 7 Ph,H proton-3.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = o2yugo32
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  8-OCT-2020 12:43:02
Revision_Time       = 23-NOV-2022 12:15:42

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          = 40
Temp_Get            = 23.5[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]
```




```
---- 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            = 7 Ph,H carbon.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = o2yugo19re2
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 28-AUG-2020 14:11:11
Revision_Time       = 28-AUG-2020 14:41:22

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          = 58
Temp_Get            = 23.9[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]
```


Me
Me
Me
Me
4
abundance
X : parts per Million : Proton
8.1217
8.1079
8.0072
7.9923
7.8766
7.8743
7.8377
7.8216
7.8125
7.7964
7.6762
7.6189
7.5491
7.5319
7.3796
7.2788
7.2387
2.7862
2.3339
2.0052
1.1120
0.0000
3.08
3.10
3.06
3.04
2.01
2.10
---- 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
Filename = dimer proton-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = o2-kaho99-31-50
Solvent = CHLOROFORM-D
Actual_Start_Time = 22-JUN-2016 14:03:26
Revision_Time = 11-FEB-2022 15:32:49
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[dC]
X_90_Width = 6.7[us]
X_Acq_Time = 1.74587904[s]
X_Angle = 45[deg]
X_Atn = 3.5[dB]
X_Pulse = 3.35[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.74587904[s]


Me
Me
Me
Me
4
abundance
X : parts per Million : Carbon13
134.4493
133.3620
130.1094
129.8519
129.1365
128.8694
128.3066
127.8488
127.7343
125.8744
125.7885
125.5691
125.0827
122.9461
122.6027
91.7081
78.6501
77.2575
77.0000
76.7520
18.1676
17.9196
16.5747
4.2417
---- 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
Filename = dimer carbon.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = o2-kaho99-31-50
Solvent = CHLOROFORM-D
Actual_Start_Time = 22-JUN-2016 14:07:35
Revision_Time = 22-JUN-2016 14:29:59
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 = 22.1[dC]
X_90_Width = 10.6[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 12.5[dB]
X_Pulse = 3.53333333[us]
Irr_Atn_Dec = 26.25[dB]
Irr_Atn_Noe = 26.25[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]