Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information

for

Nickel-Catalyzed Borylation of Arenes and Indoles via C-H Bond Cleavage

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Contents

1. General Information

^{1}H and ^{13}C NMR spectra were recorded on a JEOL ECS-400 spectrometer (JEOL, Tokyo, Japan) or VARIAN UNITY INOVA-600 spectrometer in $CDCl_3$ with tetramethylsilane as an internal reference standard. Some NMR analysis were conducted using C_6D_6 solvent and a residual C_6H_6 peak was set as an internal reference standard (δ = 7.15 ppm). NMR data have been reported as follows: chemical shift (δ) in ppm, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), coupling constant (*J*) in Hz, and integration. NOE experiments were conducted with Delta noe_1d_dpfgse pulse sequence and detail experimental parameter was listed in the spectroscopic data section for each compound. Infrared spectra (IR) were obtained on a JASCO FT/IR-4000 spectrometer, and the absorptions have been reported in reciprocal centimeters with the following relative intensities: s (strong), m (medium), or w (weak). Mass spectra were obtained on a Shimadzu GCMS-QP 5000 or GCMS-QP 2010 instrument with an ionization voltage of 70 eV. High resolution mass spectra (HRMS) were obtained on a JEOL JMS-DX303(EI) and a Bruker micrOTOF II (APCI). Analytical gas chromatography (GC) was carried out on Shimadzu GC-2014, equipped with a flame ionization detector. Melting points were determined using a Stanford Research Systems OptiMelt. Column chromatography was performed over SiO_2 (Silycycle Silica Flash F60 (230-400 mesh).

2. Materials

$Ni(cod)_2$ (Strem), $Ni(OAc)_2{\bullet}4H_2O$ (Wako), NaO^tBu (TCI), HBpin (TCI), B_2pin_2 (TCI), PCy_3 (Aldrich), IMes•HCl (TCI), IPr•HCl (TCI), IMe•HCl (TCI), $I^iPr{\bullet}HBF_4$ (TCI), I(1-Ad)•HCl (TCI), BICy•HCl (Aldrich) were obtained from commercial suppliers and used as received. $I^tBu{\bullet}HCl$,[1] ICy•HCl,[2] SICy•HCl[3] and I(2-Ad)•HCl[4] were synthesized according to the literature procedure. Methylcyclohexane (TCI), *N*-methylpyrrole (TCI), anisole (Nacalai), and *o*-xylene (TCI) were purified by distillation over CaH_2 prior use. 1,3-Bis(trifluoromethyl)benzene (Wako), *N*-methylindole (TCI), anhydrous benzene (Wako), benzene-d_6 (Wako), Hg (Wako) were obtained from commercial suppliers and used as received. 1-Butyl-1*H*-indole, 1-benzyl-1*H*-indole, 1-(4-methoxybenzyl)-1*H*-indole, 1,3-dimethyl-1*H*-indole, 1-(methoxymethyl)-1*H*-indole, 5-methoxy-1-methyl-1*H*-indole, 5-fluoro-1-methyl-1*H*-indole, 1-methyl-7-azaindole, were synthesized by the reaction of the corresponding indole with alkyl halide according to literature procedures.[5]

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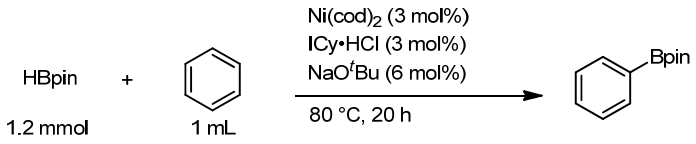
[1] Serpell, C. J.; Cookson, J.; Thompson, A. L.; Brown, C. M.; Beer, P. D. *Dalton Trans.* **2013**, *42*, 1385.
[2] Mistryukov, E. A. *Mendeleev Commun.* **2006**, *16*, 258.
[3] Gürbüz, N.; Özdemir, I.; Demir, S.; Çetinkaya, B. *J. Mol. Cat. A Chem.* **2004**, *209*, 23.
[4] Tobisu, M.; Morioka, T.; Ohtsuki, A.; Chatani, N. *submitted.*
[5] Huo, H.; Fu, C.; Harms, K.; Meggers, E. *J. Am. Chem. Soc.* **2014**, *136*, 2990.

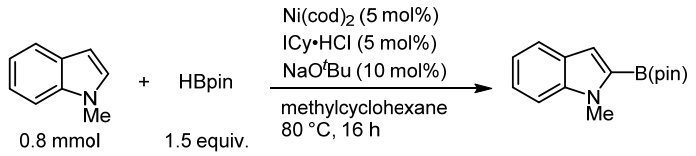
3. General Procedures for Nickel-Catalyzed Borylation of Arenes and Indoles

Method A: Procedure for the Nickel-Catalyzed Borylation of Benzene with Pinacolborane (Entry 8, Table 1)



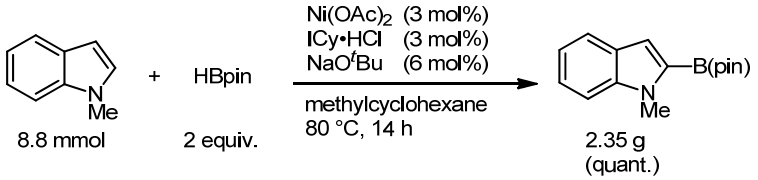
In a glovebox, $Ni(cod)_2$ (9.9 mg, 0.035 mmol), ICy•HCl (9.7 mg, 0.036 mmol), NaO^tBu (6.4 mg, 0.07 mmol) and HBpin (159 mg, 1.2 mmol) were added to a 10 mL-sample vial with a Teflon-sealed screwcap. Benzene (1.0 mL) was then added, and the cap was applied to seal the vial. The vial was stirred at 80 °C over a hot plate for 20 h and the resulting mixture was filtered through silica gel (eluting with 10 mL of hexane/AcOEt = 5/1). The filtrate was analyzed by gas chromatography (GC) using dodecane as an internal standard (75% GC yield). The filtrate was concentrated in vacuo and purified by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1) to give 4,4,5,5-tetramethyl-2-phenyl-1,3,2-dioxaborolane as a white solid (179 mg, 71% based on HBpin).

Method B: Procedure for the Nickel-Catalyzed Borylation of *N*-Methylindole with Pinacolborane (Entry 1, Table 3)



In a glovebox, $Ni(cod)_2$ (12.4 mg, 0.045 mmol), ICy•HCl (11.7 mg, 0.044 mmol), NaO^tBu (7.1 mg, 0.074 mmol), 1-methylindole (109 mg, 0.82 mmol) and HBpin (160 mg, 1.25 mmol) were added to a 10 mL-sample vial with a Teflon-sealed screwcap. Methylcyclohexane (1.0 mL) was then added, and the cap was applied. The vial was stirred at 80 °C over a hot plate for 20 h. The resulting mixture was filtered through silica gel (eluting with 10 mL of hexane/AcOEt = 5/1). The filtrate was concentrated in vacuo and purified by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1) to give 1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole as a white solid (165 mg, 78% based on 1-methylindole).

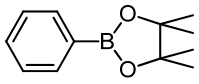
A Procedure for the Gram Scale Synthesis of 2-Borylated Indole without a Glovebox (Scheme 2)



To a two-necked flask equipped with a rubber septum and a three-way stopcock connected to a vacuum line and a N_2 line, $Ni(OAc)_2•4H_2O$ (76 mg, 0.31 mmol) was added. The flask was then heated by a heat gun under vacuum until $Ni(OAc)_2•4H_2O$ (initially light blue) completely changed color to a greenish yellow (ca. 1.5 min.). After the flask was cooled and refilled with N_2, ICy•HCl (84.4 mg, 0.31 mmol) and NaO^{t}Bu (50.8 mg, 0.53 mmol) were added under a N_2 flow. Following the addition of these solid reagents, the vessel was again placed under vacuum and its volume subsequently purged with N_2 three times. Following this, *N*-methylindole (1.16 g, 8.8 mmol), HBpin (2.44 g, 19.1 mmol) and methylcyclohexane (25 mL) were added via syringe through the rubber septum. The reaction mixture was heated at 80 °C for 14 h in an oil bath, during which time the loss of the initial reagent *N*-methylindole was monitored by GC. The reaction mixture was then poured onto silica gel and eluted with 100 mL of a hexane/AcOEt (10/1) solution. The filtrate was concentrated in vacuo and analytically pure 1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole was obtained quantitatively as a white solid (2.35 g, quant.).

4. Spectroscopic Data for the Products

4,4,5,5-Tetramethyl-2-phenyl-1,3,2-dioxaborolane (entry 8, Table 1) [CAS: 24388-23-6].



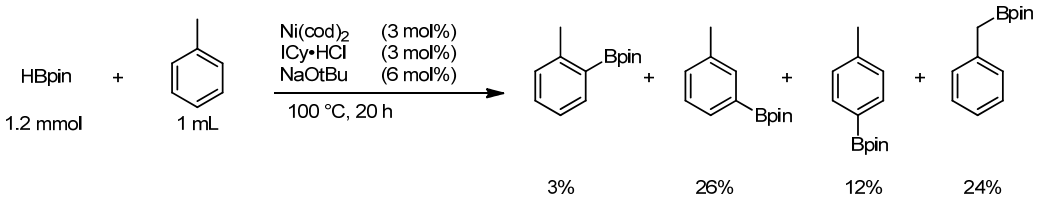
Rf 0.60 (hexane/EtOAc = 5/1). White solid (179mg, 71%).

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.34 (s, 12H), 7.37 (dt, *J* = 0.8, 7.2 Hz, 2H), 7.46 (tt, *J* = 1.6 Hz, 6.8 Hz, 1H). 7.81 (dd, *J* = 1.2 Hz, 7.2 Hz, 2H).

^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 24.8, 83.7, 127.6, 131.2, 134.7.

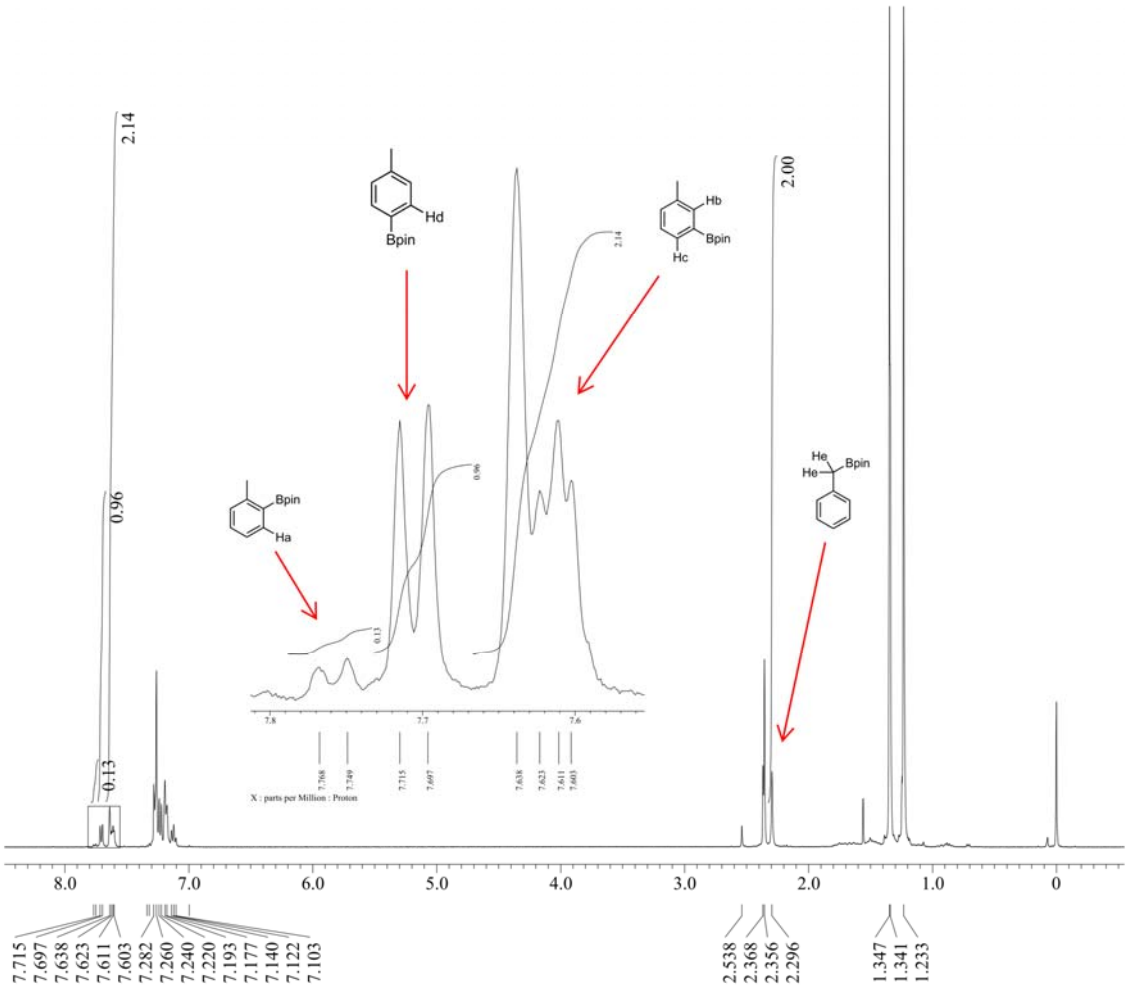
HRMS (EI): Calcd for $C_{12}H_{17}BO_2$ 204.1322, Found 204.1321.

Borylation of toluene (Entry 1, Table 2).

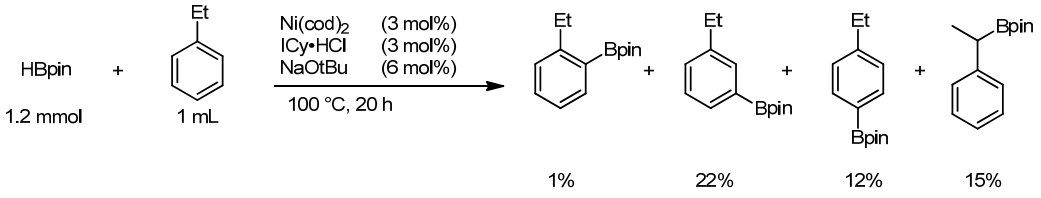


Method A was followed except that the reaction was conducted with HBpin (149 mg, 1.16 mmol) in toluene (1.0 mL) at 100 °C. After purification by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1), a mixture of four isomers was obtained (163 mg, 65%, >99% purity by GC). GC/MS analysis revealed the formation of four isomers of the borylated products, all of which had an m/z of 218 (M^+). The identity and ratio of each of the four isomers was determined by comparing the ^{1}H NMR spectrum of the product mixture with those reported in the literature.[6] The resonances specific to each isomer are as follows: 7.75 ppm (d, J = 7.6 Hz, 1H, ortho isomer, H_a); 7.60-7.64 ppm (m, 2H, meta isomer, H_b and H_c), 7.71 ppm (d, J = 6.0 Hz, 1H, para isomer, H_d); 2.29 ppm (s, 2H, benzylic isomer, H_e). (See below.)

[6] Yan, G.; Jiang, Y.; Kuang, C.; Wang, S.; Liu, H.; Zhang, Y,; Wang, J. *Chem. Commun.* **2010**, *46*, 3170. ;Pintaric, C.; Olivero, S.; Gimbert, Y.; Chavant, P. Y.; Duñach, D. *J. Am. Chem. Soc.* **2010**, *132*, 11825.

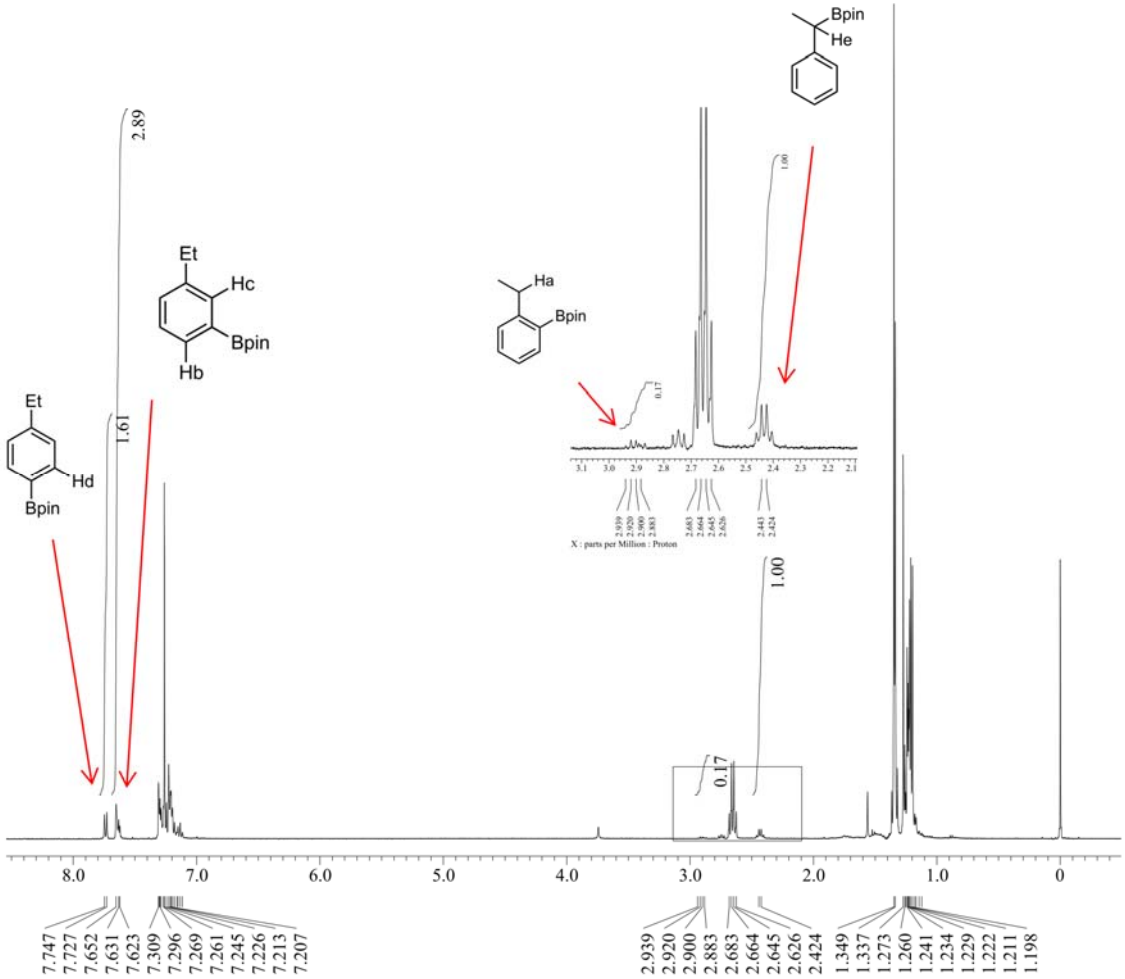


Borylation of ethylbenzene (Entry 2, Table 2).



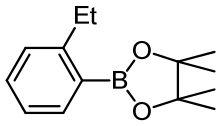
Method A was followed except that the reaction was conducted with HBpin (160 mg, 1.25 mmol) in ethylbenzene (1.0 mL) at 100 °C. After purification by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1), a mixture of four isomers was obtained (145 mg, 50%, >99% purity by GC). GC/MS analysis revealed the formation of four isomers of the borylated products, all of which had an m/z of 232 (M^+). The identities and proportions of the meta, para and benzylic isomers were determined by comparing the ^{1}H NMR spectrum of the

product mixture with those reported in the literature.[7] Because the ortho isomer was a new compound, an authentic sample was synthesized by the reaction of 2-ethylphenylboronic acid with pinacol (see below). The identity and ratio of the ortho isomer was determined by comparing the ^{1}H NMR spectrum of the product mixture with that of the authentic sample. The resonances specific to each isomer are as follows: 2.91 ppm (q, J = 8.0 Hz, 2H ortho isomer, H_a); 7.62-7.65 ppm (m, 2H, meta isomer, H_b and H_c), 7.74 ppm (d, J = 8.0 Hz, 2H, para isomer, H_d); 2.43 ppm (q, J = 8.0 Hz 1H, benzylic isomer, H_e).



[7] Boebel, T. A.; Hartwig, J. F. *Organometallics* **2008**, *27*, 6013.; Pintaric, C.; Olivero, S.; Gimbert, Y.; Chavant, P. Y.; Duñach, D. *J. Am. Chem. Soc.* **2010**, *132*, 11825.

2-(2-Ethylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane



A combination of *o*-ethylphenylboronic acid (300 mg, 2.0 mmol), pinacol (260 mg, 2.2 mmol) and Et_2O (8 mL) were added to a round bottom flask, after which $CaCl_2$ (ca. 300 mg) was added and the reaction mixture was stirred overnight. The resulting suspension was filtered and the filtrate was concentrated in vacuo. The residue was purified by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1) to give the title compound as a colorless oil (24 mg, 5%).

Rf 0.60 (hexane/EtOAc = 5/1). Colorless oil.

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.19 (t, *J* = 8.0 Hz, 3H), 1.34 (s, 12H), 2.91 (q, *J* = 8.0 Hz, 2H), 7.15-7.20 (m, 2H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.76 (d, *J* = 7.2 Hz, 1H).

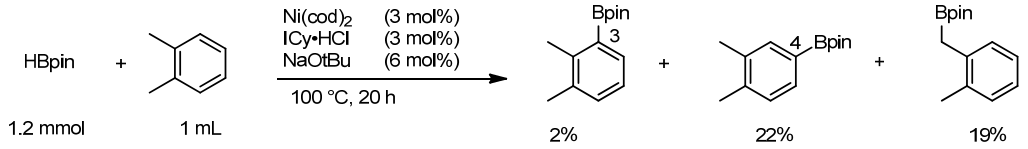
^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 17.3, 24.9, 28.9, 83.4, 124.9, 128.5, 131.0, 136.1, 151.5.

IR (ATR): 2977 w, 2931 w, 2872 w, 1600 w, 1570 w, 1489 w, 1442 w, 1381 m, 1345 s, 1310 m, 1272 w, 1214 w, 1145 s, 1126 w, 1077 m, 1031 w, 962 w, 862 m, 830 w, 792 w, 755 m, 661 m.

MS m/z (% relative intensity): 232 (M+, 11), 176 (11), 175 (100), 174 (33), 133 (59), 132 (87), 131 (45), 117 (36), 116 (11), 115 (27), 105 (32), 101 (63), 91 (19), 85 (23), 83 (28), 77 (11), 59 (15), 57 (17), 55 (13).

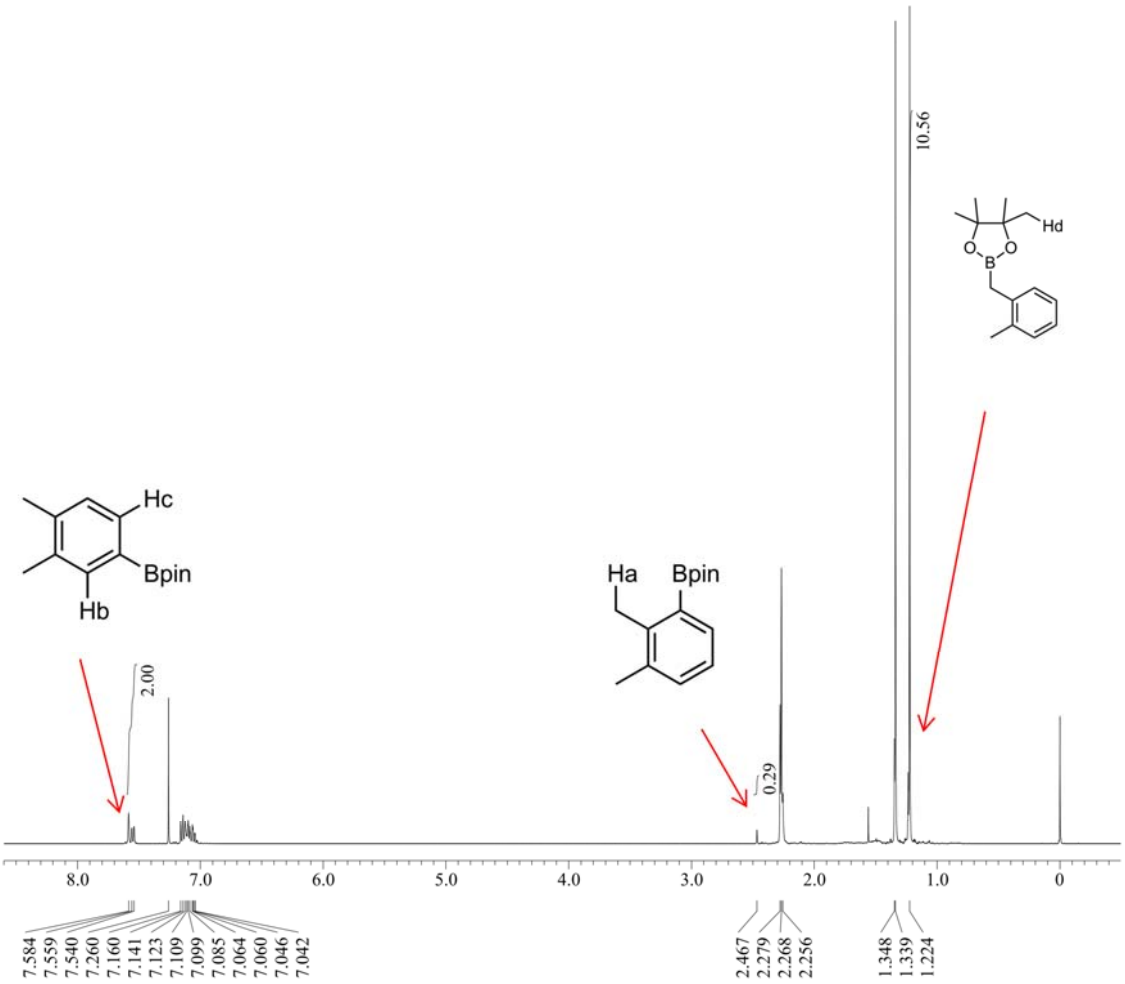
HRMS (EI): Calcd for $C_{14}H_{21}BO_2$ 232.1635, Found 232.1630.

Borylation of *o*-xylene (Entry 3, Table 2).

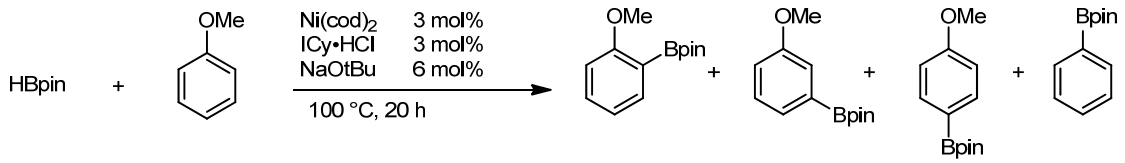


Method A was followed except that the reaction was conducted with HBpin (150 mg, 1.17 mmol) in *o*-xylene (1.0 mL) at 100 °C. After purification by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1), a mixture of three isomers was obtained (117 mg, 43%, >99% purity by GC). GC/MS analysis revealed the formation of three isomers of the borylated products, all of which had an m/z of 232 (M^+). The identity and ratio of each of the four isomers was determined by comparing the ^{1}H NMR spectrum of the product mixture with

those reported in the literature.[8] The resonances specific to each isomer are as follows: 2.46 ppm (s, 3H, 3-isomer, H_a); 7.54-7.58 ppm (m, 2H, 4-isomer, H_b and H_c), 1.22 ppm (s, 12H, benzylic isomer, H_d). (See below.)



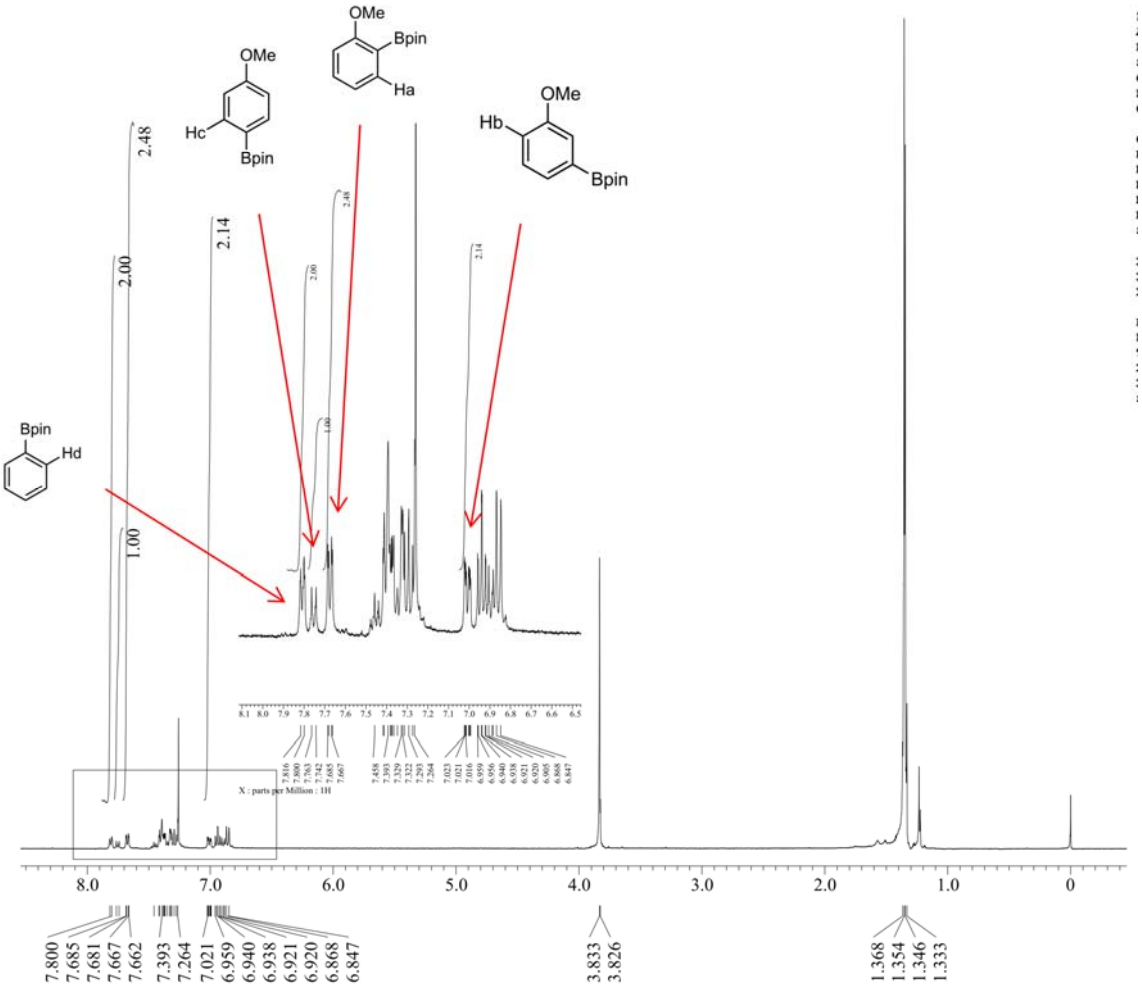
Borylation of anisole (Entry 4, Table 2).



Method A was followed except that the reaction was conducted with HBpin (160 mg, 1.25 mmol) in anisole (1.0 mL) at 100 °C. After purification by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1), a mixture of three isomers of the borylated

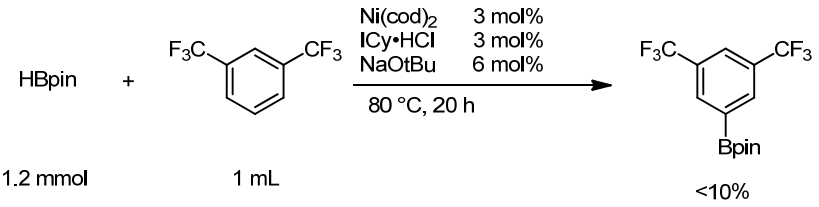
[8] Yan, G.; Jiang, Y.; Kuang, C.; Wang, S.; Liu, H.; Zhang, Y,; Wang, J. *Chem. Commun.* **2010**, *46*, 3170. ; Endo, K.; Ohkubo, T.; Shibata, T. *Org. Lett.* **2011**, *13*, 3368.

products and PhBpin were obtained (108 mg; PhBpin 7% and borylated anisole 33%; >99% purity by GC). GC/MS analysis revealed the formation of three isomers of the borylated products, all of which had an m/z of 218 (M^+) and PhBpin had an m/z of 204 (M^+). The identity and ratio of each of the borylated products was determined by comparing the ^{1}H NMR spectrum of the product mixture with those reported in the literature.[9] The resonances specific to each isomer are as follows: 7.67 ppm (dd, J =1.6 Hz, 7.2 Hz, 1H ortho isomer, H_a); 7.01 ppm (ddd, J =0.8 Hz, 2.8 Hz, 8.0 Hz, 1H, meta isomer, H_b), 7.75 ppm (d, J = 8.2 Hz, 2H, para isomer, H_c); 7.81 ppm (dd, J = 1.6 Hz, 6.4 Hz, 2H, PhBpin, H_d). (See below.)



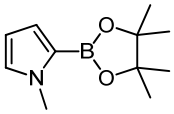
[9] Ishiyama, T.; Takagi, J.; Ishida, K.; Miyaura, N.; Anastasi, N. R.; Hartwig, J. F. *J. Am. Chem. Soc.* **2002**, *124*, 390.

Borylation of 1,3-bis(trifluoromethyl)benzene.



Method A was followed except that the reaction was conducted with HBpin (155 mg, 1.21 mmol) in 1,3-bis(trifluoromethyl)benzene (1.0 mL) at 80 °C. After purification by flash column chromatography over silica gel (eluting with hexane/AcOEt = 20/1), the product was obtained as a mixture with unidentifiable byproducts (44 mg, <10%, 84% purity by GC). HRMS analysis of this sample unambiguously determined the formation of the borylated product (Calcd for $C_{14}H_{15}BF_6O_2$ 340.1069, Found 340.1062). The identity of the borylated product was determined by comparing the ^{1}H NMR spectrum of the product mixture with that reported in the literature.[10] The resonances specific to the compound are as follows: 7.94 ppm (s, 1H, H_a), 8.24 pp (s, 2H, H_b).

1-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-pyrrole (Entry 5, Table 2) [CAS: 850567-47-4].



Method A was followed except that the reaction was conducted with HBpin (160 mg, 1.25 mmol) in *N*-methylpyrrole (1.0 mL) at 80 °C.

Rf 0.49 (hexane/EtOAc = 5/1). White solid (177 mg, 68%).

^{1}H and ^{13}C NMR data in $CDCl_3$ were in agreement with those reported for 2-borylated pyrrole.[11] However, the title compound gradually decomposed in a $CDCl_3$ solution to form *N*-metylpyrrole.

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.30 (s, 12H), 3.66 (s, *N*-methylpyrrole), 3.83 (s, 3H), 6.15-6.16 (m, 1H + *N*-methylpyrrole), 6.61 (s, *N*-methylpyrrole), 6.80-6.82 (m, 2H).

^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 24.5 (*N*-methylpyrrole), 24.7, 36.5, 83.0, 108.1 (*N*-methylpyrrole), 108.3, 121.6 (*N*-methylpyrrole), 121.8, 128.1.

Pure spectra were obtained using C_6D_6 as the solvent.

^{1}H NMR (C_6D_6, 399.78 MHz): δ 1.09 (s, 12H), 3.51 (s, 3H), 6.29-6.30 (m, 1H), 6.51-6.52 (m, 1H), 7.32-7.34 (m, 1H).

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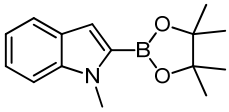
[10] Wilckens, K.; Lentz, D.; Czekelius, C. *Organometallics* **2011**, *30*, 1287.

[11] Grosso, A. D.; Singleton, P. J.; Muryn, C. A.; Ingleson, M. J. *Angew. Chem., Int. Ed.* **2011**, *50*, 2102.

^{13}C NMR (C_6D_6, 100.53 MHz): δ 24.8, 36.2, 82.9, 109.1, 123.3. One carbon peak is overlapped with the peak of residual solvent.

HRMS (EI): Calcd for $C_{11}H_{18}BNO_2$ 207.1431, Found 207.1431.

1-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 1, Table 3) [CAS: 596819-10-2].



Rf 0.54 (hexane/EtOAc = 5/1). White solid (165 mg, 78%).

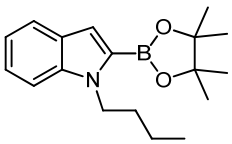
^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.36 (s, 12H), 3.97 (s, 3H), 7.06 (t, *J* = 7.2 Hz, 1H), 7.13 (s, 1H), 7.23-7.27 (m, 1H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 24.9, 32.3, 83.8, 109.8, 114.3, 119.4, 121.7, 123.3, 127.9, 140.2.

HRMS (EI): Calcd for $C_{15}H_{20}BNO_2$ 257.1587, Found 257.1584.

All spectroscopic data were in agreement with the reported 2-borylated product.[12]

1-Butyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 2, Table 3).



Method B was followed using 1-butyl-1*H*-indole (146 mg, 0.84 mmol) instead of 1-methylindole.

Rf 0.69 (hexane/EtOAc = 5/1). White solid (171 mg, 68%). Mp = 47-48 °C.

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 0.92 (t, *J* = 7.2 Hz, 3H), 1.29-1.34 (m, 2H), 1.36 (s, 12H), 1.74(t, *J* = 7.2 Hz, 2H), 4.41 (t, *J* = 7.2 Hz, 2H), 7.06 (t, *J* = 7.6 Hz, 1H), 7.12 (s, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 14.0, 20.3, 24.9, 33.2, 45.3, 83.7, 110.6, 114.4, 119.2, 121.8, 123.0, 128.0, 139.4.

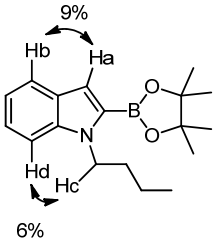
IR (ATR): 3051 w, 2976 w, 2930 w, 2873 w, 1610 w, 1569 w, 1523 s, 1480 w, 1468 w, 1386 m, 1354 m, 1321 s, 1304 s, 1263 s, 1237 w, 1200 m, 1134 s, 1114 m, 1072 m, 1008 w, 964 w, 926 m, 858 m, 834 m, 803 m, 753 m, 732 s, 707 m, 690 s, 666m.

MS m/z (% relative intensity): 299 (M+, 76), 257 (26), 256 (100), 156 (67), 130 (47).

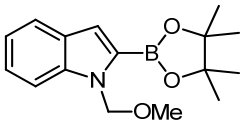
[12] Mertins, K.; Zapf, A.; Beller, M. *J. Mol. Cat. A Chem.* **2004**, *207*, 21.

HRMS (EI): Calcd for $C_{18}H_{26}BNO_2$ 299.2057, Found 299.2053.

The regiochemistry of the compound was determined by NOE experiments in $CDCl_3$. When signal H_a at δ = 7.12 (s, 1H, offset signal δ = 7.109, mixing time 3.43 s) was irradiated, H_b at δ = 7.63 (d, *J* = 7.6 Hz, 1H) exhibited an enhancement by 9%. When signal H_c at δ = 4.41 (t, *J* = 7.2 Hz, 1H, offset signal δ = 4.407, mixing time 0.443 s) was irradiated, H_d at δ = 7.36 (d, *J* = 8.4 Hz, 1H) exhibited an enhancement by 6%.



1-(Methoxymethyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 3, Table 3).



Method B was followed using 1-(methoxymethyl)-1*H*-indole (127 mg, 0.79 mmol) instead of 1-methylindole.

Rf 0.57 (hexane/EtOAc = 5/1). White solid (153 mg, 68%). Mp = 52-53 °C

^{1}H NMR (C_6D_6, 399.78 MHz): δ 1.07 (s, 12H), 3.04 (s, 3H), 5.71 (s, 2H), 7.13 (m, 1H), 7.23 (dt, *J* = 1.2, 6.0 Hz, 1H), 7.53 (dd, *J* = 8.0 Hz, 1H), 7.55 (s, 1H), 7.63 (d, 8.0 Hz, 1H).

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.37 (s, 12H), 3.22 (s, 3H), 5.81 (s, 2H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.18 (s, 1H), 7.28 (dt, *J* = 1.2, 7.4 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.65 (d, *J* = 7.8 Hz, 1H)

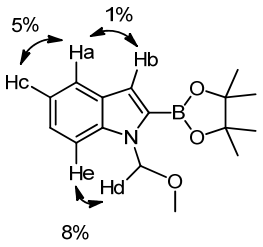
^{13}C NMR (C_6D_6, 100.53 MHz): δ 24.7, 55.0, 76.1, 83.7, 111.3, 117.0, 120.8, 122.1, 124.2, 129.3, 140.6.

IR (ATR): 2979 w, 2933 w, 2363 w, 1723 w, 1612 w, 1573 w, 1528 s, 1482 w, 1369 m, 1346 m, 1322 s, 1305 s, 1263 s, 1224 m, 1191 w, 1164 m, 1137 s, 1101 m, 1081 s, 1050 m, 1010 w, 962 m, 913 w, 857 m, 833 m, 812 m, 738 s, 704 w, 688 s, 670 w.

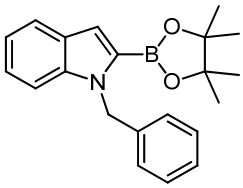
MS m/z (% relative intensity): 287 (M+, 100), 286 (27), 256 (77), 171 (27), 170 (20), 156 (68), 130 (39), 77 (20).

HRMS (EI): Calcd for $C_{16}H_{22}BNO_3$ 287.1693, Found 287.1692.

The regiochemistry of the compound was determined by NOE experiments in $CDCl_3$. When signal H_a at δ = 7.65 (d, *J* = 7.8 Hz, 1H, offset signal δ = 7.651, mixing time 0.8168 s) was irradiated, H_b at δ = 7.18 (s, 1H) was enhanced by 1% and H_c at δ = 7.13 (t, *J* = 7.6 Hz, 1H) by 5%. When signal H_d at δ = 5.81 (s, 2H, offset signal δ = 5.799, mixing time 0.8598 s) was irradiated, H_e δ = 7.53 (d, *J* = 8.0 Hz, 1H) was enhanced by 8%.



1-Benzyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 4, Table 3).



Method B was followed using 1-benzyl-1*H*-indole (150 mg, 0.73 mmol) instead of 1-methylindole.

Rf 0.43 (hexane/EtOAc = 5/1). White solid (199 mg, 82%). Mp = 125-127 °C.

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.26 (s, 12H), 5.66 (s, 2H), 7.03-7.09 (m, 3H), 7.17-7.22 (m, 5H), 7.30 (d, *J* = 8.4 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 1H).

^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 24.8, 49.0, 83.8, 110.4, 115.1, 119.6, 121.8, 123.5, 126.6, 126.9, 128.3, 128.4, 139.4, 139.9.

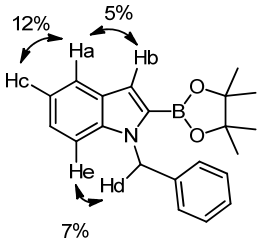
IR (ATR): 3030 w, 2979 w, 2925 w, 2852 w, 1609 w, 1523 m, 1494 w, 1480 w, 1452 w, 1387 m, 1360 m, 1324 s, 1300 s, 1267 s, 1192 m, 1136 s, 1114 m, 1087 m, 1031 w, 1005 w, 984 m, 960 w, 926 w, 855 m, 829 m, 799 m, 772 w, 726 s, 690 s, 672.

MS m/z (% relative intensity): 333 (M+, 54), 332 (30), 91 (100).

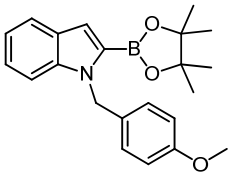
HRMS (EI): Calcd for $C_{21}H_{24}BNO_2$ 333.1900, Found 333.1899.

The regiochemistry of the compound was determined by NOE experiments in $CDCl_3$.

When signal H_a at δ = 7.66 (d, *J* = 8.0 Hz, 1H, offset signal δ = 7.673, mixing time 2.601 s) was irradiated, H_b at δ = 7.20 (s, 1H) exhibited an enhancement by 5% and H_c at δ = 7.06 (t, *J* = 7 Hz, 1H) enhanced by 12%. When signal H_d at δ = 5.66 (s, 2H, offset signal δ = 5.659, mixing time 0.8 s) was irradiated, H_e at δ = 7.30 (d, *J* = 8.4 Hz, 1H) was enhanced by 7%.



1-(4-Methoxybenzyl)-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 5, Table 3).



Method B was followed using 1-(4-methoxybenzyl)-1*H*-indole (174 mg, 0.73 mmol) instead of 1-methylindole.

Rf 0.54 (hexane/EtOAc = 5/1). White solid (201 mg, 76 %). Mp = 116 °C.

^{1}H NMR (C_6D_6, 399.78 MHz): δ 1.04 (s, 12H), 3.19 (s, 3H), 5.55 (s, 2H), 6.59 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 7.10 (t, *J* = 8.2 Hz, 1H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.25 (d, *J* = 8.4 Hz, 1H), 7.65 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 1H).

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.30 (s, 12H), 3.74 (s, 3H), 5.59 (s, 2H), 6.76 (d, J = 8.7 Hz, 2H), 7.02 (d, *J* = 8.8 Hz, 2H), 7.07 (t, *J* = 8.0 Hz, 1H), 7.18 (s, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.65 (d, J = 7.8 Hz, 1H).

^{13}C NMR (C_6D_6, 100.53 MHz): δ 24.6, 48.5, 54.5, 83.6, 110.8, 114.6, 116.1, 120.0, 122.2, 123.8, 127.8 (overlapped with the solvent peak), 129.0, 131.6, 140.4, 159.0.

^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 24.7, 48.3, 55.1, 83.7, 110.3, 113.6, 114.9, 119.4, 121.6, 123.2, 127.7, 128.1, 131.3, 139.6, 158.4.

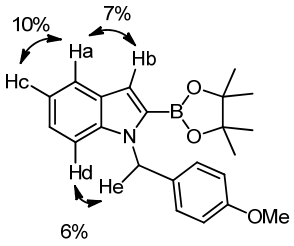
IR (ATR): 2977 w, 1613w, 1514 s, 1481 w, 1462 w, 1441 w, 1382 m, 1354 m, 1321 m, 1295 s, 1269 s, 1249 s, 1193 m, 1174 m, 1134 s, 1085 m, 1038 m, 985 w, 960 w, 856 m, 830 m, 805 737 s, 704 w, 688 s, 672 w.

MS m/z (% relative intensity): 363 (M+, 17), 121 (100).

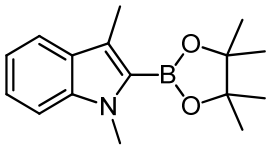
HRMS (EI): Calcd for $C_{22}H_{26}BNO_3$ 363.2006, Found 363.2005.

The regiochemistry of the compound was determined by NOE experiments in $CDCl_3$. When signal H_a at δ = 7.65 (d, *J* = 7.8 Hz, 1H, offset signal δ = 7.647, mixing time 2.379 s) was irradiated, H_b at δ = 7.18 (s, 1H) was enhanced by 7% and H_c at δ = 7.07 (t, *J* = 8.0 Hz, 1H) had

an enhancement of 10%. When signal H_d at δ = 7.32 (d, *J* = 8.4 Hz, 1H, offset signal δ = 7.317, mixing time 2.379 s) was irradiated, He at δ = 5.59 (s, 2H) had an NOE enhancement of 6%.



1,3-Dimethyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 6, Table 3).



Method B was followed using 1,3-dimethyl-1*H*-indole (116 mg, 0.80 mmol) instead of 1-methylindole.

Rf 0.54 (hexane/EtOAc = 5/1). White solid (187mg, 87%). Mp = 88-89 °C.

^{1}H NMR (C_6D_6, 399.78 MHz): δ 1.13 (s, 12H), 2.85 (s, 3H), 3.71 (s, 3H), 7.13 (d, *J* = 8.8 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.31 (dt, *J* = 0.8, 8.0 Hz, 1H), 7.74 (d, *J* = 7.6 Hz, 1H).

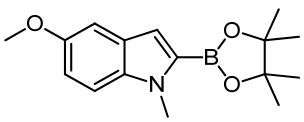
^{13}C NMR (C_6D_6, 100.53 MHz): δ 10.7, 24.7, 32.0, 83.0, 109.8, 119.0, 120.3, 123.8, 125.6, 129.3, 140.5.

IR (ATR): 3051 w, 2979 w, 2932 w, 2863 w, 1711 w, 1610 w, 1530 m, 1438 m, 1410 m, 1370 m, 1334 m, 1294 s, 1264 s, 1242 m, 1213 m, 1166 m, 1144 s, 1088 m, 1045 m, 1007 w, 962 m, 859 m, 835 m, 739 s, 714 w, 697 s, 672 w.

MS m/z (% relative intensity): 272 (16), 271 (M+, 100), 270 (36), 189 (22), 188 (41), 171 (20), 170 (32), 145 (10), 144 (44), 115 (12), 77 (14).

HRMS (EI): Calcd for $C_{16}H_{22}BNO_2$ 271.1744, Found 271.1745.

5-Methoxy-1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 7, Table3).



Method B was followed using 5-methoxy-1-methyl-1*H*-indole (127 mg, 0.79 mmol) instead of 1-methylindole.

Rf 0.46 (hexane/EtOAc = 5/1). White solid (172 mg, 76%). Mp = 100-101 °C.

^{1}H NMR ($CDCl_3$, 399.78 MHz): δ 1.35 (s, 12H), 3.84 (s, 3H), 3.93 (s, 3H), 6.93 (dd, *J* = 2.0 Hz, 9.2 Hz, 1H), 7.02 (s, 1H), 7.05 (d, *J* = 2.4 Hz, 1H), 7.23 (d, *J* = 9.2 Hz, 1H).

^{1}H NMR (C_6D_6, 399.78 MHz) δ 1.12 (s, 12H), 3.44 (s, 3H), 3.67 (s, 3H), 6.98 (d, *J* = 8.7 Hz, 1H), 7.06 (d, *J* = 2.3 Hz, 1H), 7.17 (dd, *J* = 2.4 Hz, 8.8 Hz, 1H), 7.56 (s, 1H)

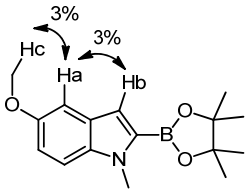
^{13}C NMR ($CDCl_3$, 100.53 MHz): δ 24.9, 32.5, 55.9, 83.7, 102.1, 110.6, 113.5, 114.5, 128.1, 135.9, 154.0.

IR (ATR): 2977 w, 1619 w, 1521 s, 1457 w, 1394 m, 1372 w, 1346 w, 1315 m, 1299 s, 1260 s, 1233 s, 1206 s, 1169 m, 1139 s, 1107 m, 1066 m, 1036 m, 963 m, 849 m, 832 s, 792 s, 706 w, 689 s, 677w.

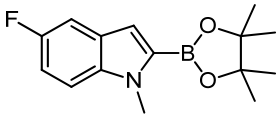
MS m/z (% relative intensity): 287 (M+, 100), 286 (25), 272 (41), 190 (17).

HRMS (EI): Calcd for $C_{16}H_{22}BNO_3$ 287.1693, Found 287.1695.

The regiochemistry of the compound was determined by NOE experiments in C_6D_6. When signal H_a at δ = 7.06 (d, *J* = 2.3 Hz, 1H, offset signal δ = 7.006, mixing time 1.52 s) was irradiated, H_b at δ = 7.56 (s, 1H) exhibited an enhancement by 9%, and H_c at δ = 3.44 (s, 3H) by 3 %.



5-Fluoro-1-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1*H*-indole (Entry 8, Table 3).



Method B was followed using 5-fluoro-1-methyl-1*H*-indole (115 mg, 0.77 mmol) and 1.2 equivalent of HBpin (120 mg, 0.94 mmol).

Rf 0.49 (hexane/EtOAc = 5/1). White solid (173 mg, 82%). Mp = 116 °C.

^{1}H NMR (C_6D_6, 395.88 MHz) δ 1.09 (s, 12H), 3.56 (s, 3H), 6.77 (dd, *J* = 4.0 Hz, 8.8 Hz, 1H), 6.99 (dt, *J* = 2.4 Hz, 8.8 Hz, 1H), 7.26 (dd, *J* = 2.4 Hz, 9.2 Hz, 1H), 7.37 (d, *J* = 1.2 Hz, 1H).

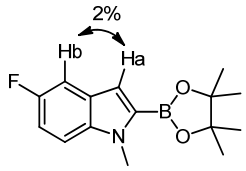
^{13}C NMR (C_6D_6, 99.54 MHz): δ 24.7, 32.1, 83.6, 106.3 (d, *J* = 23 Hz), 110.6 (d, *J* = 10 Hz), 112.2 (d, *J* = 27 Hz), 115.0 (d, *J* = 5 Hz), 128.6 (d, *J* = 10 Hz), 137.4, 158.4 (d, *J* = 235 Hz)

IR (ATR): 2982 m, 1523 s, 1458 w, 1390 m, 1372 m, 1317 m, 1295 m, 1254 s, 1181 m, 1137 s, 1065 m, 962 m, 861 m, 846 s, 787 s, 707 w, 690 s, 678 m.

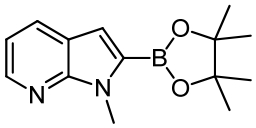
MS m/z (% relative intensity): 276 (16), 275 (M+, 100), 274 (28), 202 (20), 193 (20), 190 (32), 176 (19), 175 (43), 174 (30).

HRMS (APCI): Calcd for $[M+H]^+$ $C_{15}H_{20}BFNO_2$ 276.1568, Found 276.1569.

The regiochemistry of the compound was determined by NOE experiments in C_6D_6. When signal H_a at δ = 7.37 (d, J = 1.2 Hz, 1H, offset signal δ = 7.3116, mixing time 1.5 s) was irradiated, H_b at δ = 7.26 (dd, J = 2.4, 9.2 Hz, 1H) exhibited an enhancement of 2%.



1-Methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-7-aza-1*H*-indole (Entry 9, Table 3).



Method B was followed using 1-methyl-7-aza-1*H*-indole (105 mg, 0.80 mmol) and 1.2 equivalent of HBpin (120 mg, 0.94 mmol).

Rf 0.14 (hexane/EtOAc = 5/1). White solid (116 mg, 56%). Mp = 66-68 °C.

^{1}H NMR (C_6D_6, 399.78 MHz): δ 1.07 (s, 12H), 4.09 (s, 3H), 6.74 (dd, J = 4.6 Hz, 7.8 Hz, 1H), 7.38 (s, 1H), 7.59 (dd, J = 1.3 Hz, 7.8 Hz, 1H), 8.48 (dd, J = 1.3 Hz, 4.4 Hz, 1H).

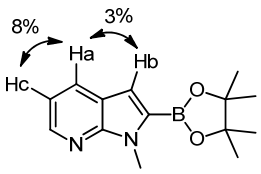
^{13}C NMR (C_6D_6, 100.53 MHz): δ 24.7, 30.9, 83.7, 112.9, 115.9, 120.2, 129.3, 145.3, 151.1.

IR (ATR): 2978 w, 1592 w, 1564 w, 1521 s, 1461 m, 1380 m, 1323 s, 1254 s, 1214 w, 1135 s, 1109 m, 1061 m, 961 w, 910 w, 855 m, 834 m, 816 m, 774 s, 724 w, 707 w, 690 s, 668 m.

MS m/z (% relative intensity): 258 (M+, 100), 257 (31), 185 (20), 173 (30), 159 (27), 158 (27) 157 (32), 132 (24), 131 (72).

HRMS (EI): Calcd for $C_{14}H_{19}BN_2O_2$ 258.1540, Found 258.1542.

The regiochemistry of the compound was determined by NOE experiments in C_6D_6. When signal H_a at δ = 7.59 (dd, J = 1.3, 7.8 Hz, 1H, offset signal δ = 7.547, mixing time 2.7 s) was irradiated, H_b at δ = 7.38 (s, 1H) was enhanced by 3% and H_c at δ = 6.74 (dd, J = 4.6, 7.8 Hz, 1H) by 8%.



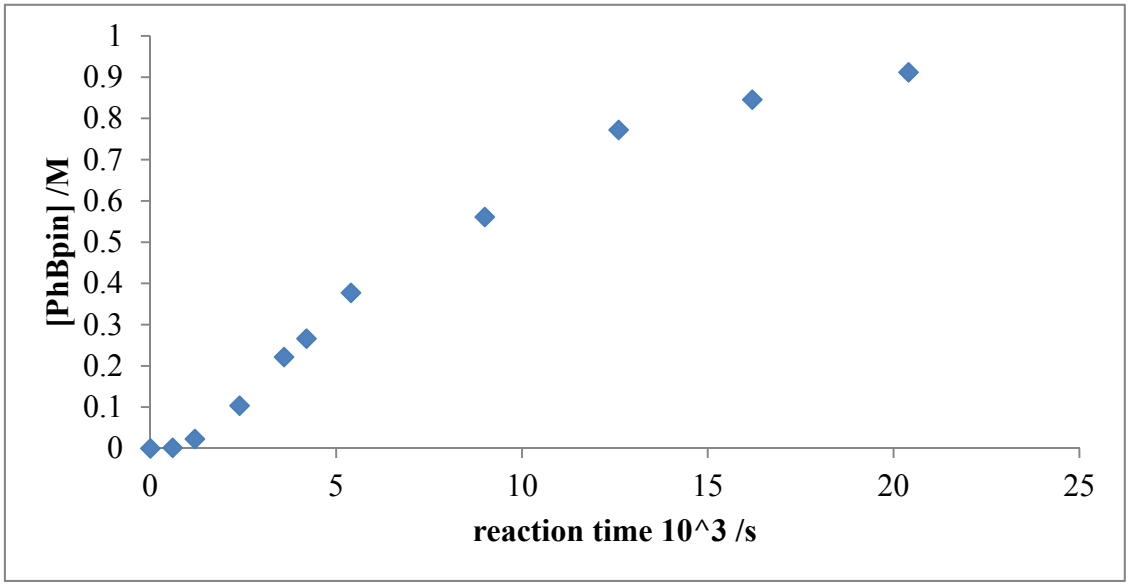
5. Mechanistic Studies

5-1. Kinetic Isotope Effect

The reaction progress was monitored by GC. Dodecane was added as an internal standard.

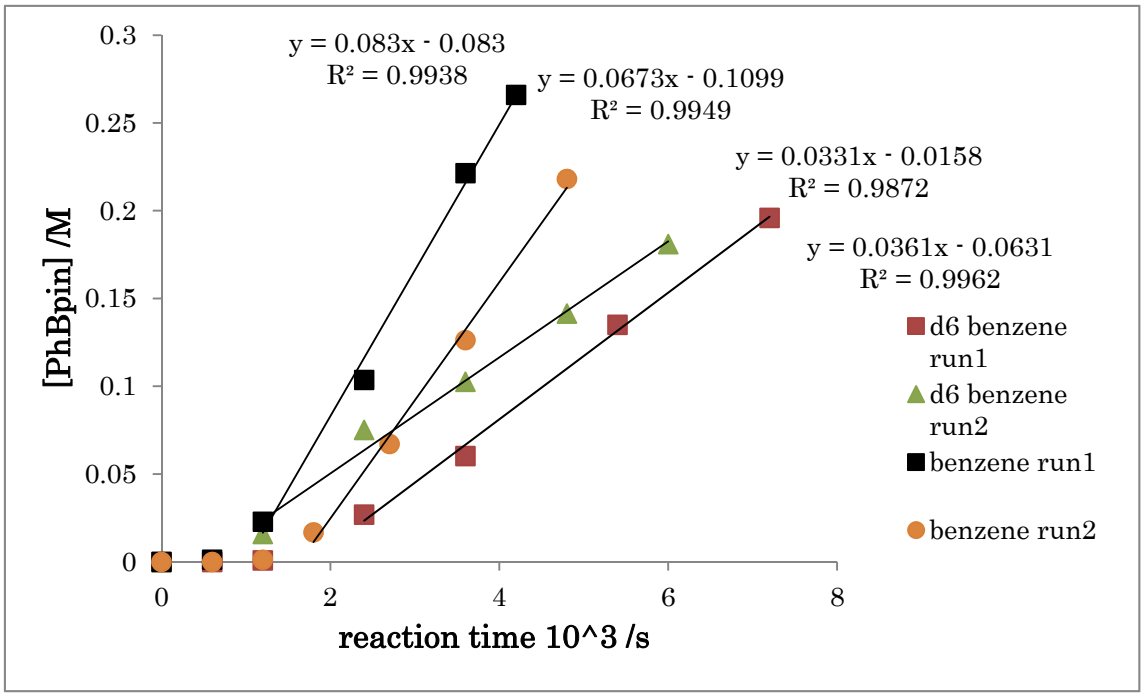
A typical procedure for kinetic studies. In a glovebox, $Ni(cod)_2$ (9.6 mg, 0.035 mmol), ICy•HCl (9.2 mg, 0.034 mmol), NaO^tBu (6.4 mg, 0.067 mmol), HBpin (161 mg, 1.3 mmol) and dodecane (60 mg as internal standard) were added to a 10 mL-sample vial with a Teflon-sealed screw cap. Benzene (1.0 mL) was then added and the cap was applied. The vial was heated at 80 °C with stirring over a hot plate. At the indicated times, the vial was rapidly cooled with running water and an aliquot of the reaction mixture was taken from the vial in a glovebox. The aliquot was diluted with AcOEt and analyzed by GC. After sampling, the reaction mixture was heated again until the next sampling.

The amount of PhBpin in the reaction mixture was determined over time, and the results are shown below.



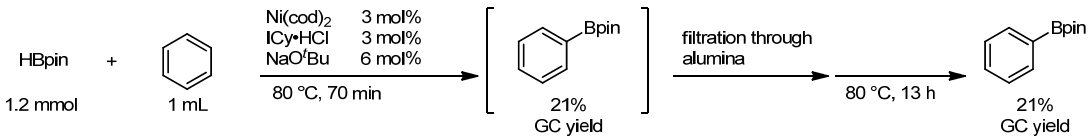
The total yield after 8 hours was 72%. These results showed that reaction monitoring by cooling did not deactivate the catalytic species. We observed that the reaction had an induction period of approximately 20 minutes at 80 °C.

Measurement of kinetic isotope effect. The initial rate up to 20% conversion was measured following the induction period, in which ca. 1 % of PhBpin was generated. The experiments were performed with benzene and benzene-d_6. Each experimental trial was performed in duplicate.



The average rate with benzene was determined to be 7.5×10^{-5} M/s, while the average rate with benzene-d_6 was determined to be 3.5×10^{-5} M/s. The KIE for this reaction was 2.1.

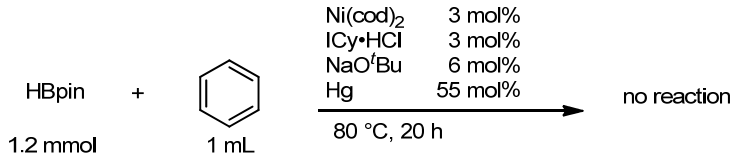
5-2. Test for Heterogeneous Catalyst



Filtration test. In a glovebox, $Ni(cod)_2$ (9.1 mg, 0.033 mmol), ICy•HCl (9.4 mg, 0.035 mmol), NaO^{t}Bu (6.8 mg, 0.070 mmol), HBpin (163 mg, 1.3 mmol) and dodecane (59 mg as internal standard) were added to a 10 mL-sample vial with a Teflon-sealed screw cap. Benzene (1.0 mL) was then added and the cap was applied. The vial was heated at 80 °C with stirring over a hot plate. After 70 minutes, the vial was rapidly cooled under running water. An aliquot was taken from the mixture in a glovebox for GC analysis, which indicated that PhBpin was formed in 21% yield. The remainder of the reaction mixture was passed through alumina using 1 mL of benzene as the eluent. The filtrate was then transferred to a new 10 mL-sample vial and heated

again for 13 h. The yield after 13 h was determined to be 21 %, thus demonstrating that the reaction did not proceed further following filtration.

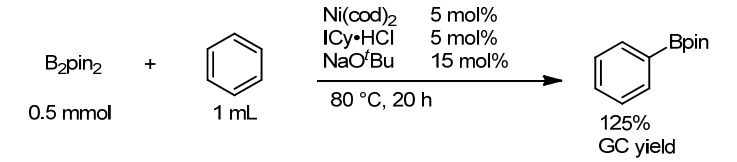
Note: When the reaction was performed under condition A with added alumina (40 mg), 63% GC yield was found following a 19 h reaction time span. This result indicated that alumina did not have a significant negative effect on this reaction.



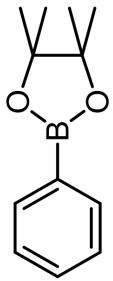
Hg drop test. In a glovebox, $Ni(cod)_2$ (10.5 mg, 0.038 mmol), ICy•HCl (10.2 mg, 0.038 mmol), NaO^tBu (6.4 mg, 0.067 mmol), HBpin (164 mg, 1.3 mmol), dodecane (64 mg as internal standard) and Hg (145 mg, 0.72 mmol) were added to a 10 mL-sample vial with a Teflon-sealed screw cap. Benzene (1.0 mL) was then added and the cap was applied. The vial was heated at 80 °C for 20 h with stirring over a hot plate. The resulting mixture was analyzed by GC and none of the target product was observed.

5-3. Diboron as a Borylating Reagent

This reaction was conducted with B_2pin_2 as the boron source instead of HBpin.



In a glovebox, $Ni(cod)_2$ (7.5 mg, 0.027 mmol), ICy•HCl (7.4 mg, 0.028 mmol), NaO^tBu (7.2 mg, 0.074 mmol) and B_2pin_2 (126 mg, 0.49 mmol) were added to a 10 mL-sample vial with a Teflon-sealed screw cap. Benzene (1.0 mL) was then added and the cap was applied. The vial was heated at 80 °C for 20 h with stirring over a hot plate. The resulting mixture was filtered through a silica gel column (eluting with 10 mL of hexane/AcOEt =5/1). The filtrate was analyzed by GC with dodecane as the internal standard and the target product was found in a 125% GC yield based on the molar amount of B_2pin_2.



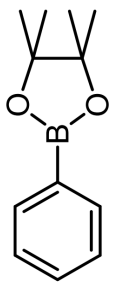
X : parts per Million : Proton

```
Filename          = TFP464_1H-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP464
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2014 19:16:24
Revision_Time     = 21-JAN-2015 11:54:26
Current_Time      = 21-JAN-2015 11:54:43

Comment           = TFP464_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 20.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

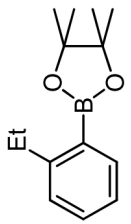


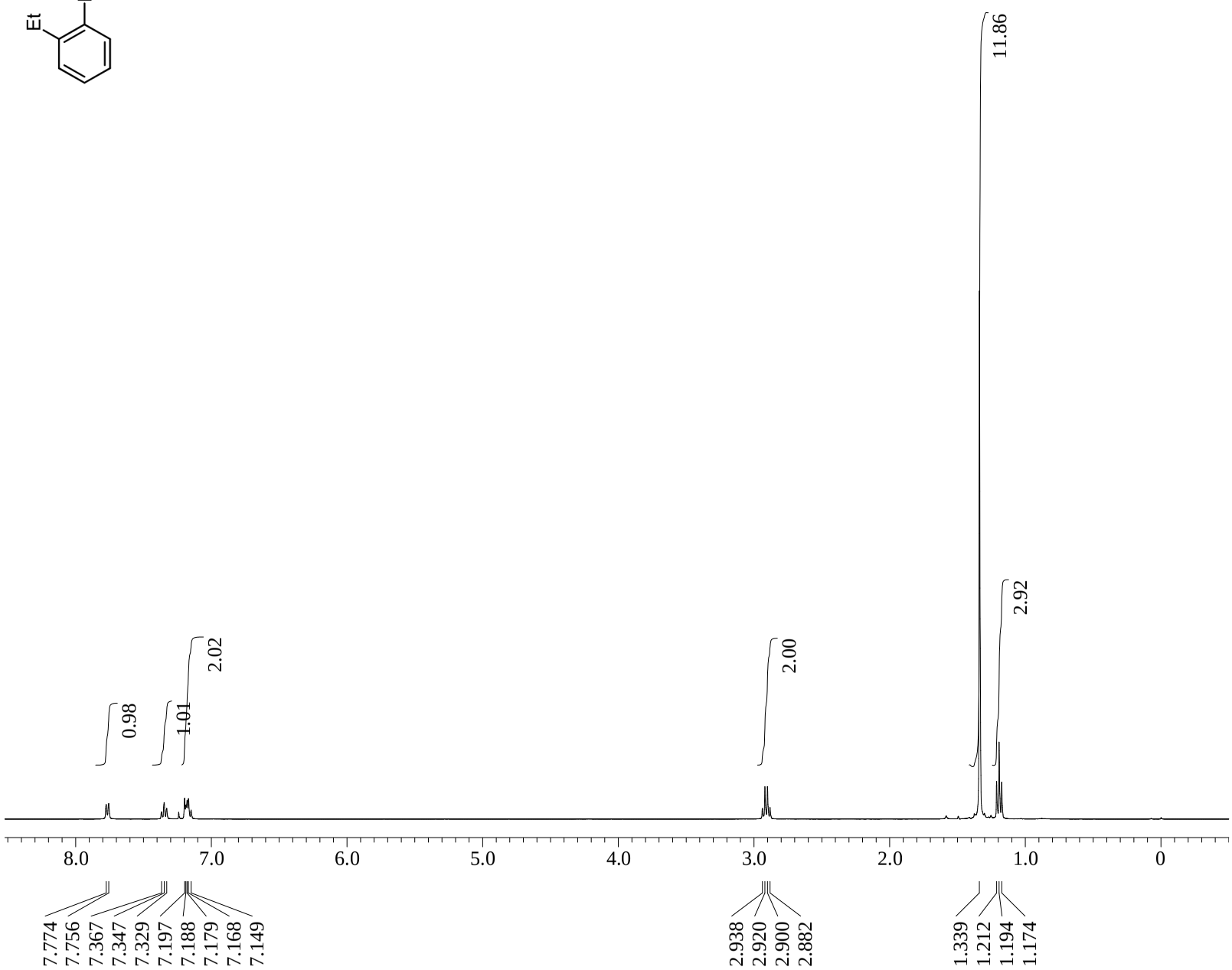

```
Filename          = TFP464_13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP464
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2014 19:17:53
Revision_Time     = 21-JAN-2015 11:58:07
Current_Time      = 21-JAN-2015 11:59:13

Comment           = TFP464_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.4[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```





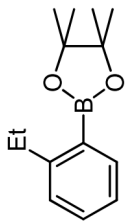
X : parts per Million : Proton

```
Filename          = TFPG88-1-7.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFPG88
Solvent           = CHLOROFORM-D
Creation_Time     = 28-JAN-2015 22:32:28
Revision_Time     = 13-FEB-2015 18:02:46
Current_Time      = 13-FEB-2015 18:03:11

Comment           = TFPG88
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 17.9[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



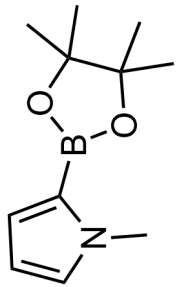
X : parts per Million : Carbon13

```
Filename          = TFPG88_13C-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFPG88
Solvent           = CHLOROFORM-D
Creation_Time     = 28-JAN-2015 22:36:09
Revision_Time     = 28-JAN-2015 22:53:09
Current_Time      = 13-FEB-2015 18:04:09

Comment           = TFPG88_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 18.1[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Atn_Noe       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



in $CDCl_3$

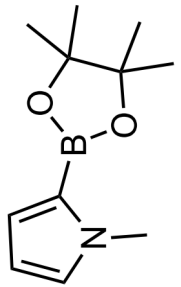

```
Filename          = TFP448-1_1H-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP448-1
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2014 23:20:37
Revision_Time     =  9-FEB-2015 18:04:56
Current_Time      =  9-FEB-2015 18:07:09

Comment           = TFP448-1_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 19.4[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

X : parts per Million : Proton



in C_6D_6

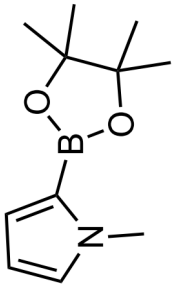
X : parts per Million : Proton

```
Filename          = TFP399-1_conc_1H-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP399-1_conc
Solvent           = BENZENE-D6
Creation_Time     =  1-OCT-2014 20:47:11
Revision_Time     =  9-FEB-2015 17:58:44
Current_Time      =  9-FEB-2015 17:59:04

Comment           = TFP399-1_conc_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 34
Temp_Get          = 20.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



in $CDCl_3$

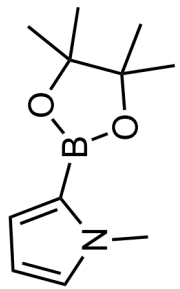
X : parts per Million : Carbon13

```
Filename          = TFP448-1_13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP448-1
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2014 23:22:08
Revision_Time     =  9-FEB-2015 18:09:53
Current_Time      =  9-FEB-2015 18:10:14

Comment           = TFP448-1_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 180
Total_Scans       = 180

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.4[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



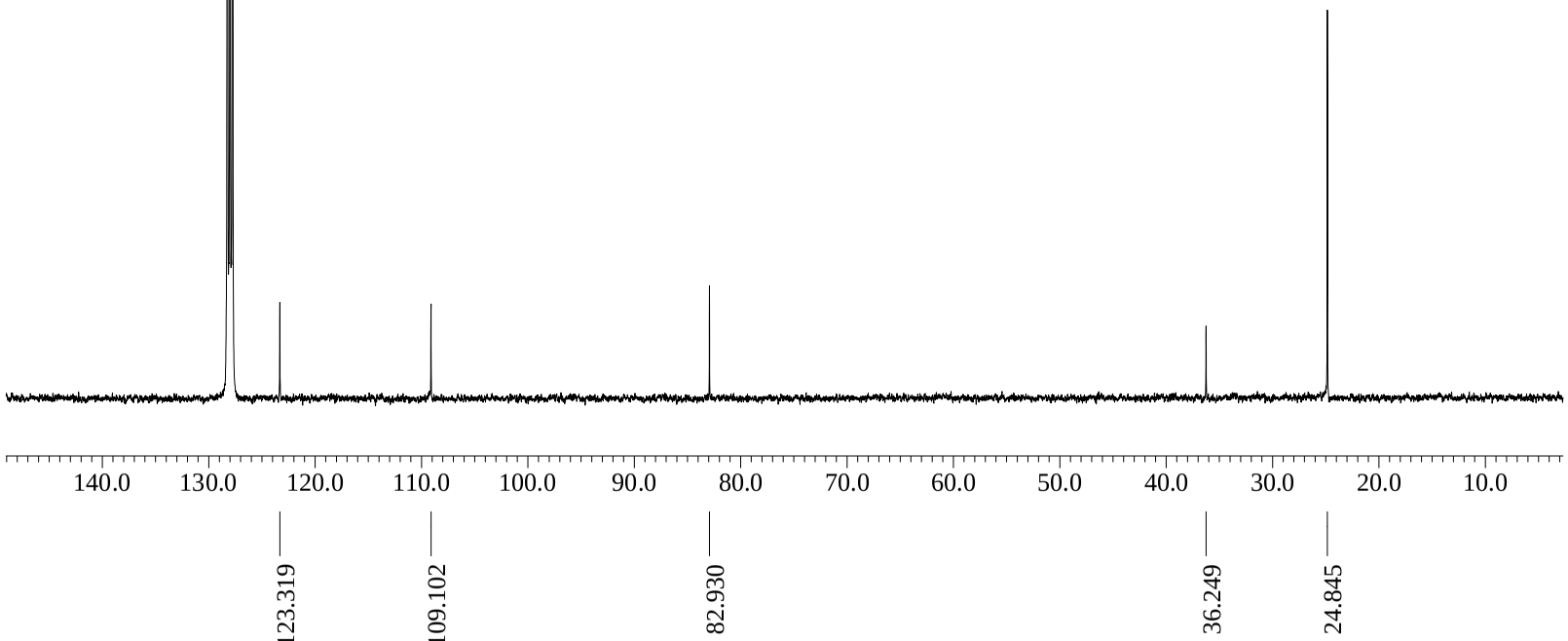
in C_6D_6

```
Filename          = TFP399-1_conc_13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP399-1_conc
Solvent           = BENZENE-D6
Creation_Time     =  1-OCT-2014 20:48:49
Revision_Time     =  9-FEB-2015 18:01:19
Current_Time      =  9-FEB-2015 18:02:00

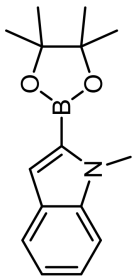
Comment           = TFP399-1_conc_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 21[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



X : parts per Million : Carbon13



X : parts per Million : Proton

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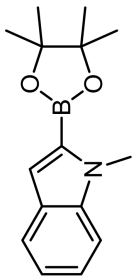
Filename          = TFP420_product_1H-1-7.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP420_product
Solvent           = CHLOROFORM-D
Creation_Time     = 14-OCT-2014 16:13:32
Revision_Time     = 21-JAN-2015 15:13:55
Current_Time      = 21-JAN-2015 15:14:07

Comment           = TFP420_product_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 19.1[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

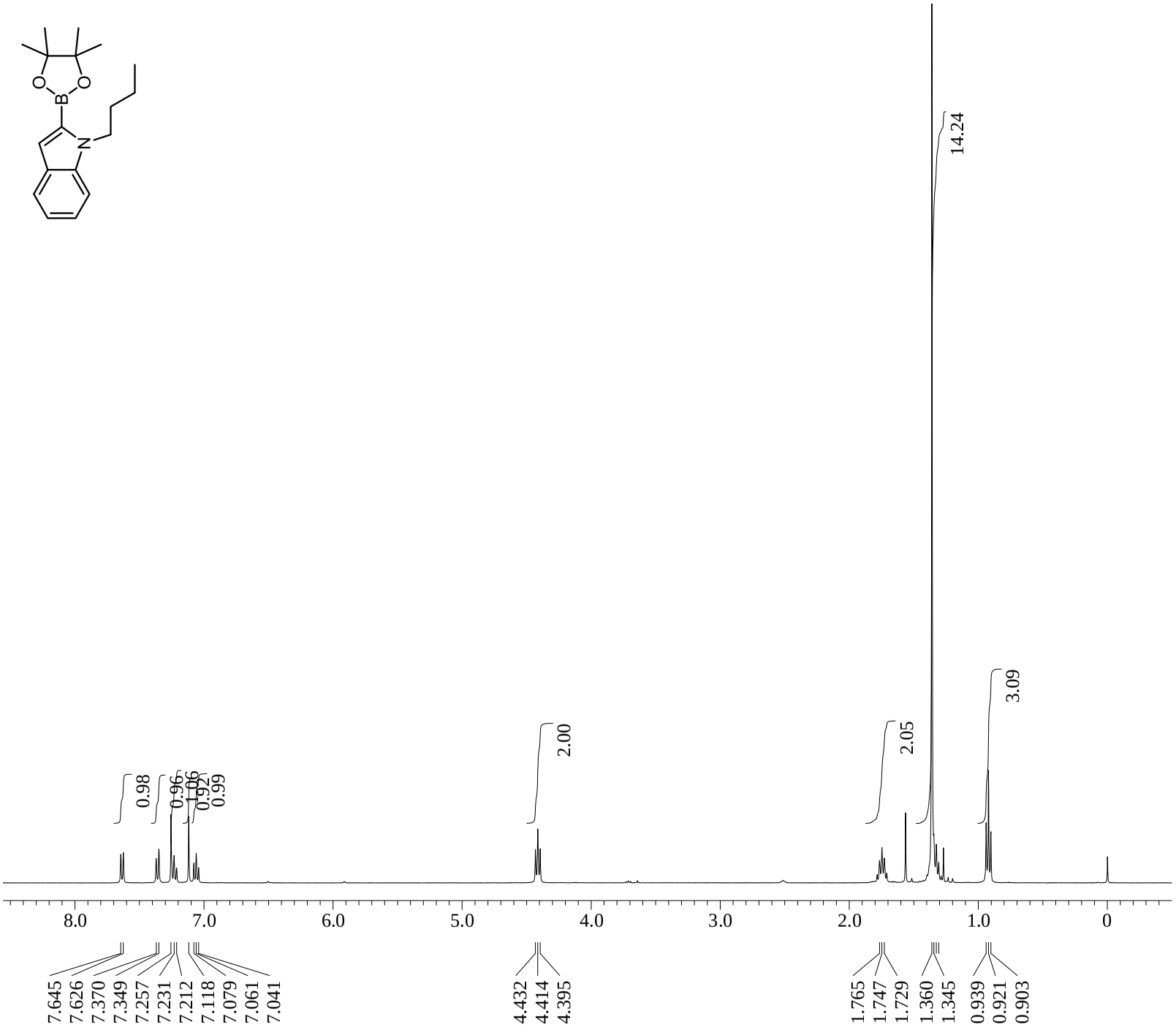



```
Filename          = TFP420_product_13C-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP420_product
Solvent           = CHLOROFORM-D
Creation_Time     = 14-OCT-2014 16:15:02
Revision_Time     = 14-OCT-2014 16:27:37
Current_Time      = 21-JAN-2015 15:16:56

Comment           = TFP420_product_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.3[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



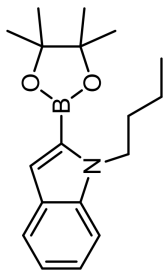
X : parts per Million : Proton

```
Filename          = TFPG_BuIndBpin-1H-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFPG_BuIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 14-JAN-2015 22:54:19
Revision_Time     = 21-JAN-2015 15:50:34
Current_Time      = 21-JAN-2015 15:51:53

Comment           = TFPG_BuIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 17.9[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

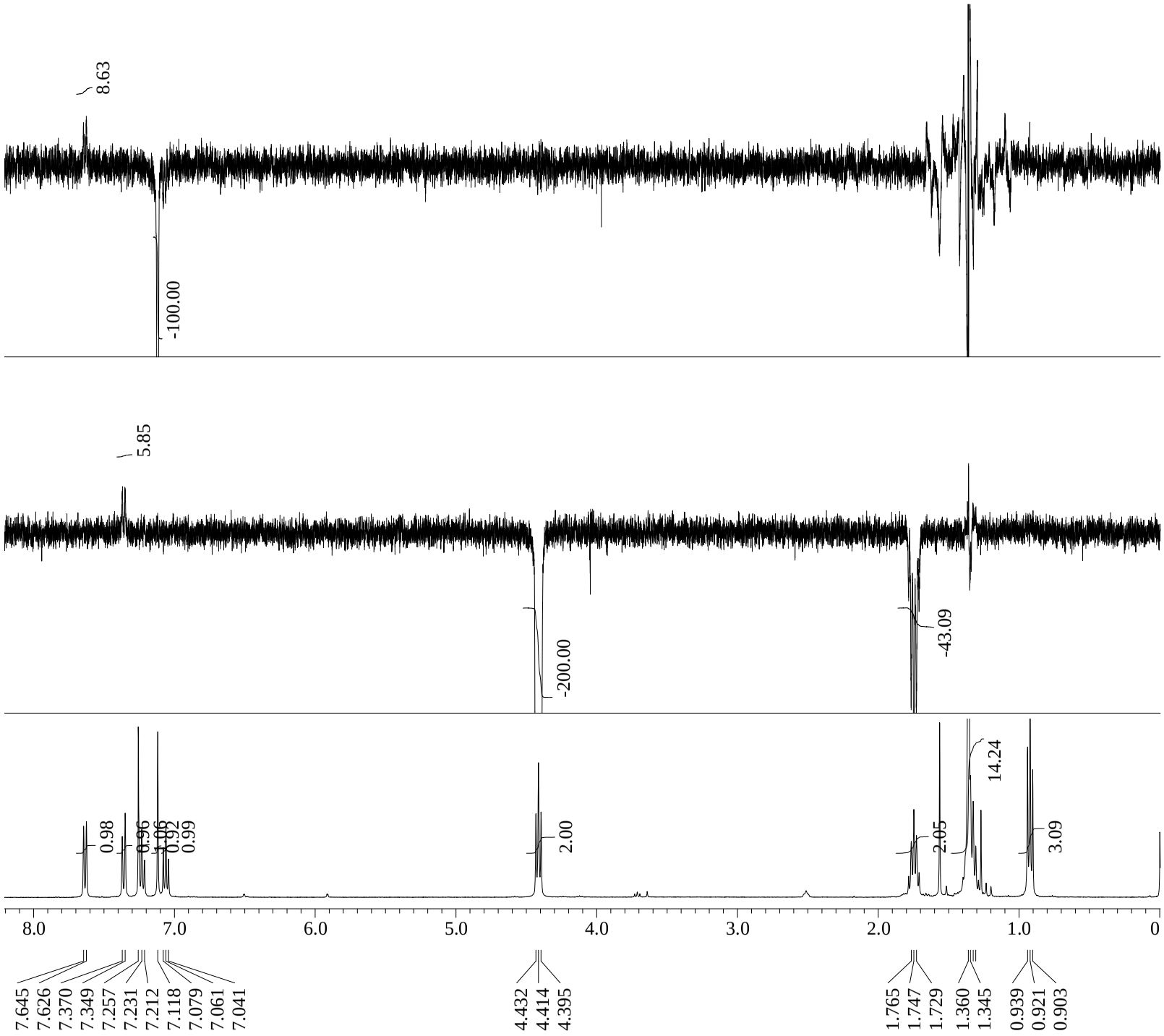



```
Filename          = TFP435-1_conc_13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP435-1_conc
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2014 18:55:51
Revision_Time     = 21-JAN-2015 15:56:18
Current_Time      = 21-JAN-2015 15:57:07

Comment           = TFP435-1_conc_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 180
Total_Scans       = 180

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.2[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



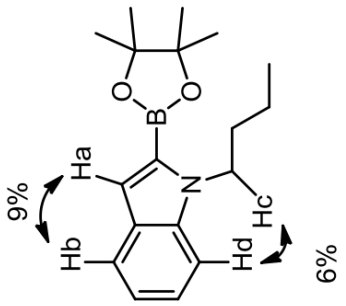
X : parts per Million : Proton

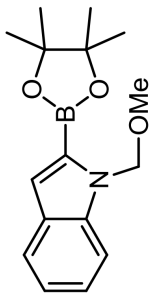
```
Filename          = TFPG_BuIndBpin-1H_an-1.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFPG_BuIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 14-JAN-2015 22:54:19
Revision_Time     = 21-JAN-2015 15:51:44
Current_Time      = 22-JAN-2015 15:29:43

Comment           = TFPG_BuIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 17.9[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```





in C_6D_6

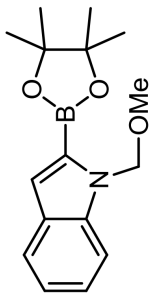
X : parts per Million : Proton

```
Filename          = TFPG527-2-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFPG527-2
Solvent           = BENZENE-D6
Creation_Time     = 14-JAN-2015 20:12:28
Revision_Time     = 21-JAN-2015 18:34:22
Current_Time      = 21-JAN-2015 18:35:08

Comment           = TFPG527-2
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 17.6[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



in $CDCl_3$

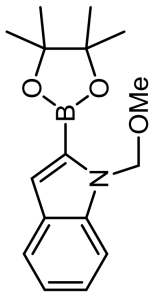
X : parts per Million : Proton

```
Filename          = TFP_MOMIndBpin-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_MOMIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2015 15:32:00
Revision_Time     = 21-JAN-2015 18:41:10
Current_Time      = 21-JAN-2015 18:48:12

Comment           = TFP_MOMIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 18[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



in C_6D_6

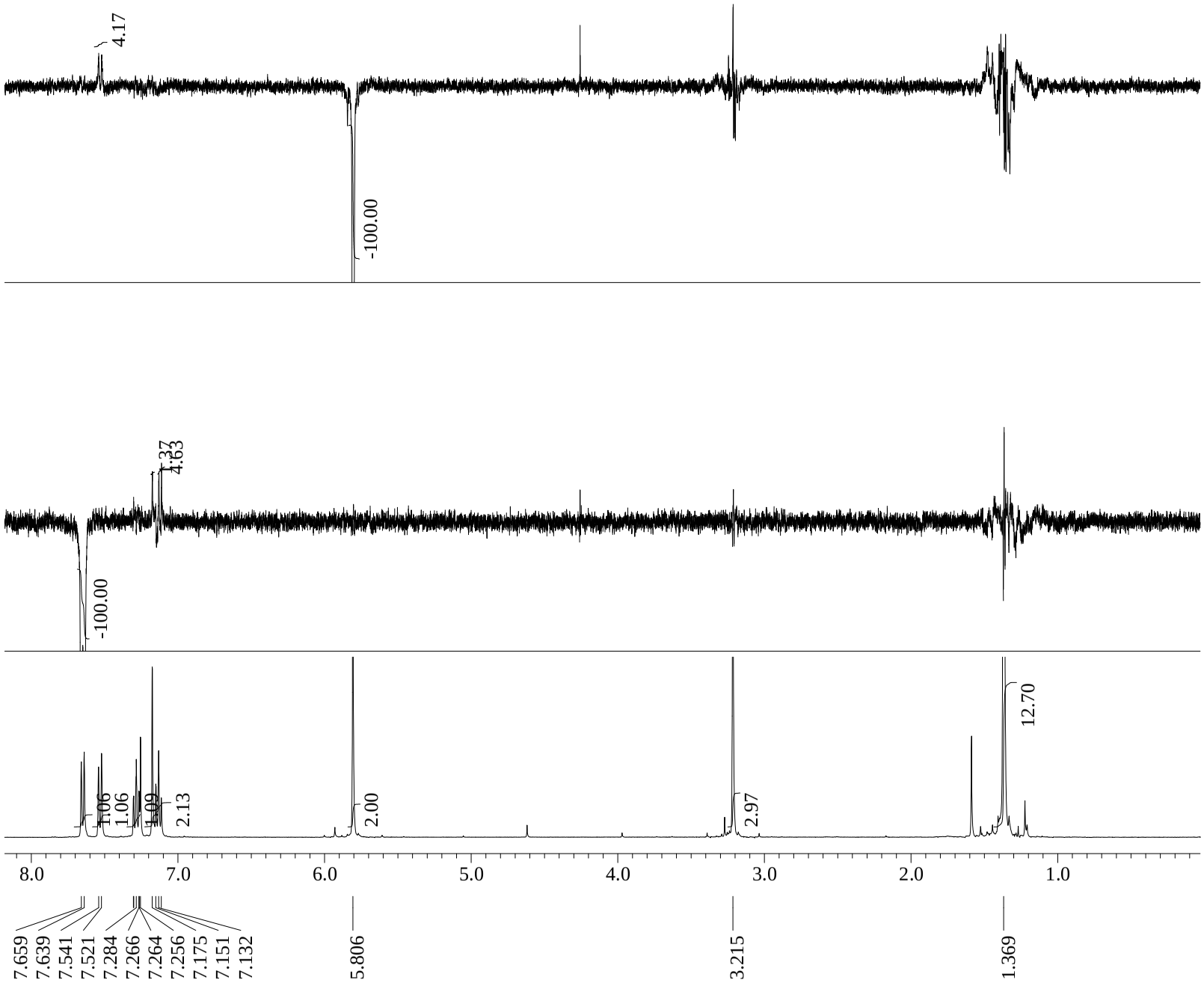
```
Filename          = TFP451_re_benz_13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP451_re_benz
Solvent           = BENZENE-D6
Creation_Time     =  7-NOV-2014 11:23:35
Revision_Time     = 21-JAN-2015 18:37:01
Current_Time      = 21-JAN-2015 18:37:42

Comment           = TFP451_re_benz_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 21.9[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```


X : parts per Million : Carbon13

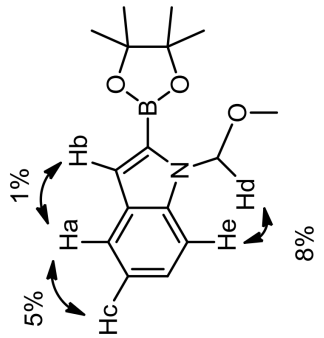


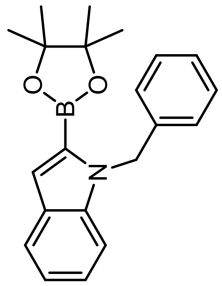

```
Filename          = TFP_MOMIndBpin-1_1H_CDCL3_
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_MOMIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2015 15:32:00
Revision_Time     = 21-JAN-2015 18:48:04
Current_Time      = 21-JAN-2015 19:02:01

Comment           = TFP_MOMIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 18[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```





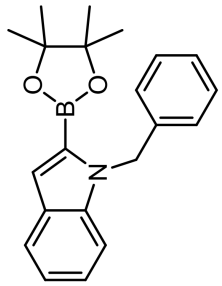
X : parts per Million : Proton

```
Filename          = TFP447_1H-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP447
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2014 16:41:24
Revision_Time     = 21-JAN-2015 17:56:05
Current_Time      = 21-JAN-2015 17:57:28

Comment           = TFP447_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 19.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



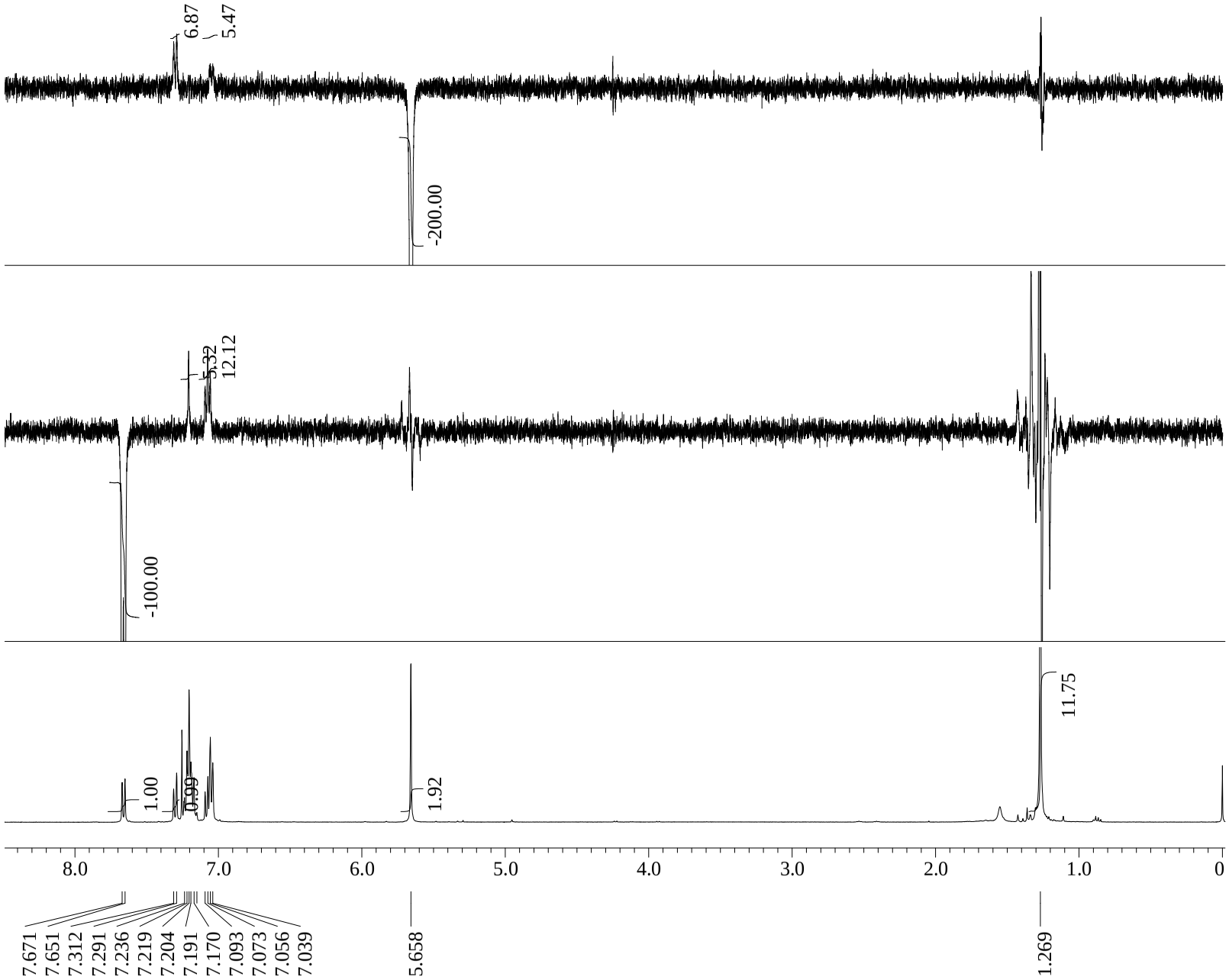
```
Filename          = TFP447_13C-1-6.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP447
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2014 16:42:55
Revision_Time     = 21-JAN-2015 17:58:51
Current_Time      = 21-JAN-2015 17:59:28

Comment           = TFP447_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 180
Total_Scans       = 180

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.6[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```


X : parts per Million : Carbon13



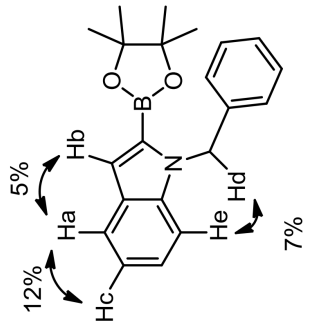
X : parts per Million : Proton

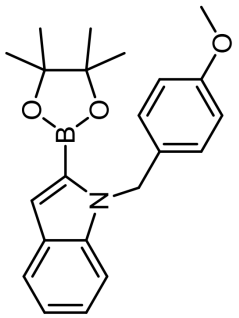
```
Filename          = TFP447_1H-1_ana-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP447
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2014 16:41:24
Revision_Time     = 21-JAN-2015 18:03:50
Current_Time      = 21-JAN-2015 18:04:31

Comment           = TFP447_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 19.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```





in C_6D_6

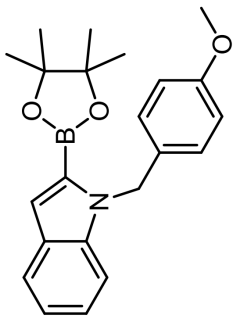
X : parts per Million : Proton

```
Filename          = TFP506_colum2-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP506_colum2
Solvent           = BENZENE-D6
Creation_Time     = 24-DEC-2014 16:21:43
Revision_Time     = 21-JAN-2015 19:40:44
Current_Time      = 21-JAN-2015 19:41:23

Comment           = TFP506_colum2
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 17.5[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



in $CDCl_3$

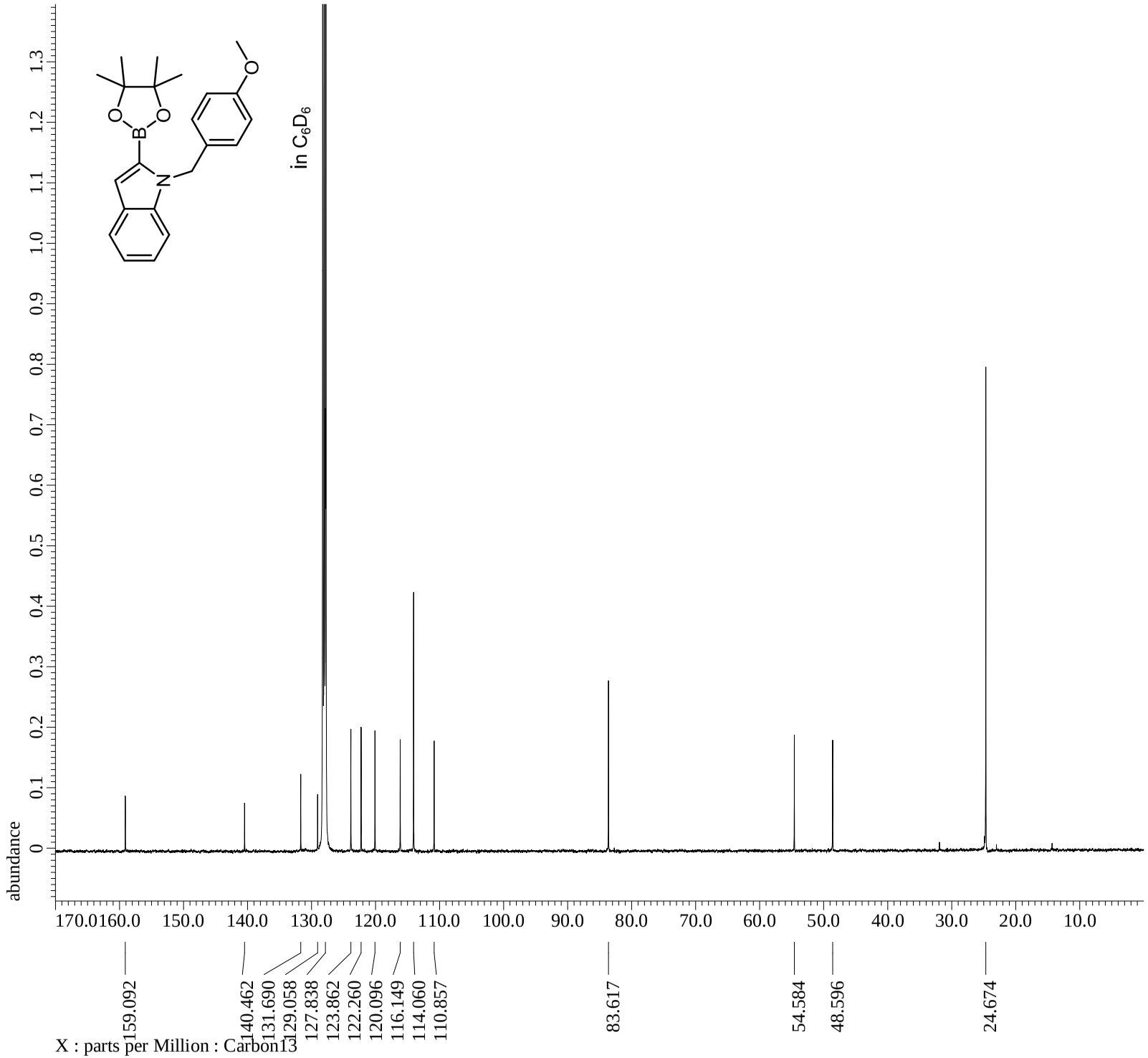
X : parts per Million : Proton

```
Filename          = TFP_MeOBnIndBpin-1-7.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_MeOBnIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2015 19:08:42
Revision_Time     = 21-JAN-2015 19:29:07
Current_Time      = 21-JAN-2015 19:37:00

Comment           = TFP_MeOBnIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 17.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

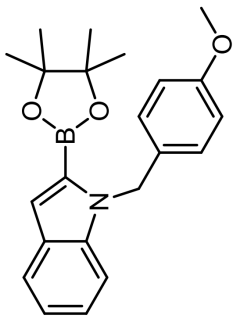



```
Filename          = TFP506_13C-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = S#275528
Solvent           = BENZENE-D6
Creation_Time     = 25-DEC-2014 07:39:38
Revision_Time     = 25-DEC-2014 10:20:11
Current_Time      = 21-JAN-2015 19:50:22

Comment           = _13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2000
Total_Scans       = 2000

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 17.9[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Atn_Noe       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



in $CDCl_3$

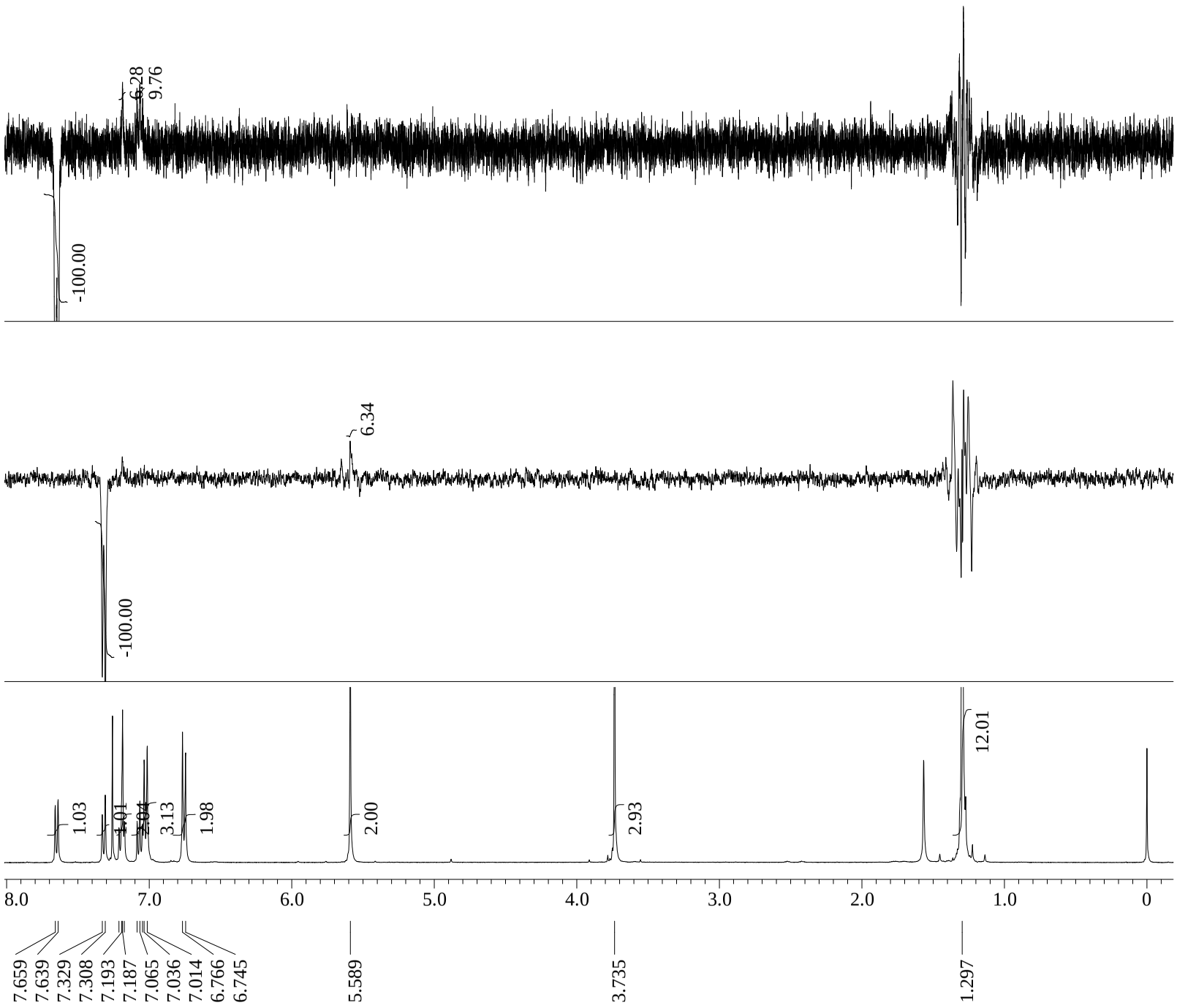
X : parts per Million : Carbon13

```
Filename          = TFP_MeOBnIndBpin_13C-1-5.j
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP_MeOBnIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2015 20:04:31
Revision_Time     = 17-JAN-2015 20:21:30
Current_Time      = 21-JAN-2015 19:51:09

Comment           = TFP_MeOBnIndBpin_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 18.2[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Atn_Noe       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

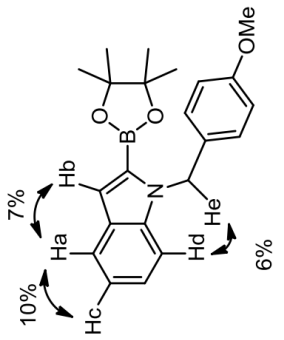


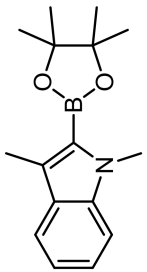

```
Filename          = TFP_MeOBnIndBpin-1_CDCL3_a
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_MeOBnIndBpin
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JAN-2015 19:08:42
Revision_Time     = 21-JAN-2015 19:36:37
Current_Time      = 21-JAN-2015 20:10:57

Comment           = TFP_MeOBnIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 17.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```





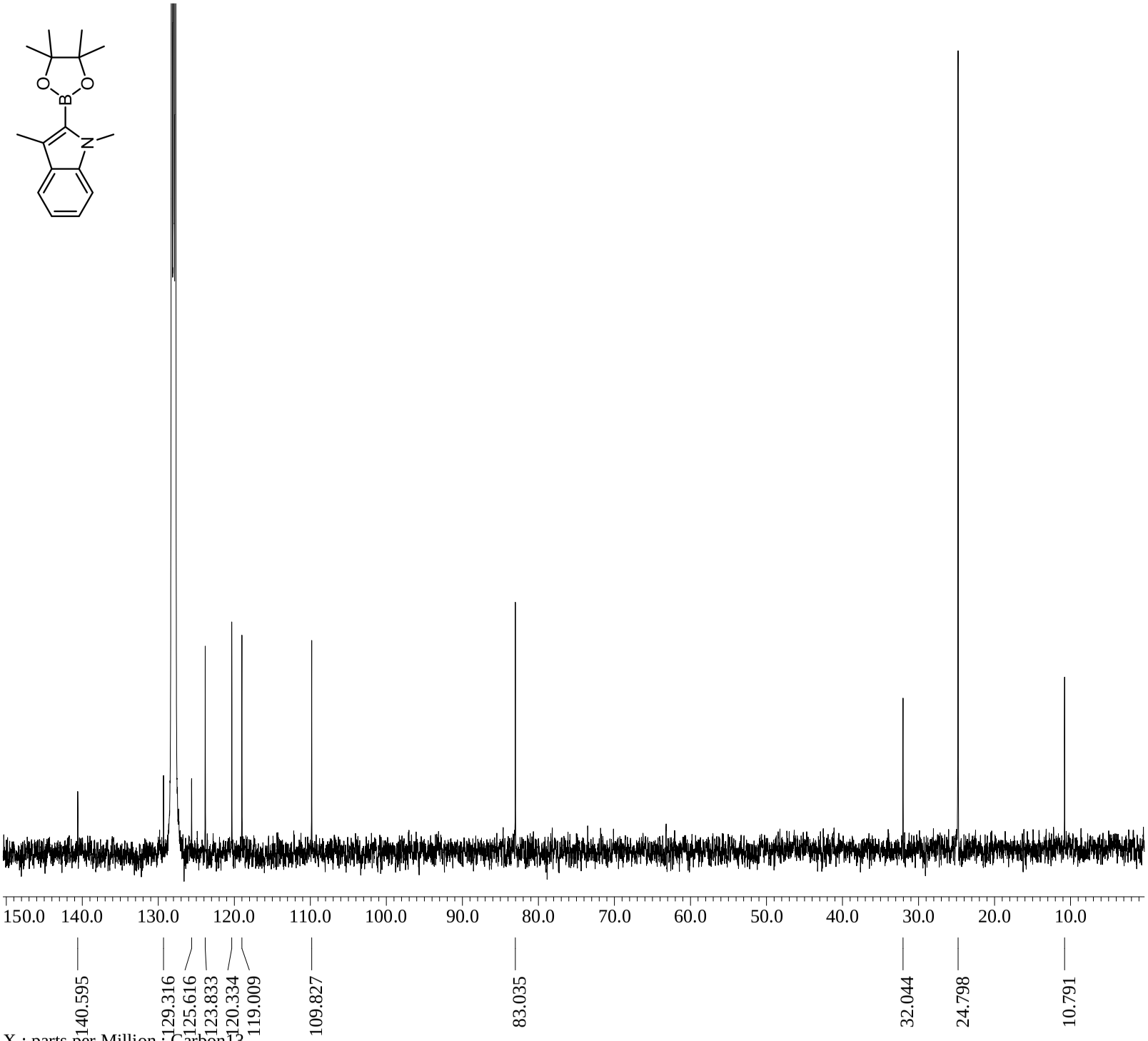
X : parts per Million : Proton

```
Filename          = TFP_1.3diMeindBpin-1-6.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_1.3diMeindBpin
Solvent           = BENZENE-D6
Creation_Time     = 16-JAN-2015 15:00:35
Revision_Time     = 21-JAN-2015 15:20:43
Current_Time      = 21-JAN-2015 15:22:41

Comment           = TFP_1.3diMeindBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 18.1[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

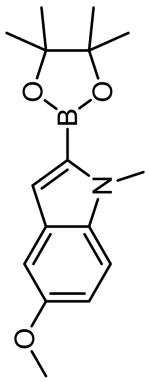



```
Filename          = TFP_1.3diMeindBpin_13C-1-3
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP_1.3diMeindBpin
Solvent           = BENZENE-D6
Creation_Time     = 16-JAN-2015 15:02:19
Revision_Time     = 21-JAN-2015 15:25:54
Current_Time      = 21-JAN-2015 15:27:00

Comment           = TFP_1.3diMeindBpin_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 18.5[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Atn_Noe       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



in $CDCl_3$

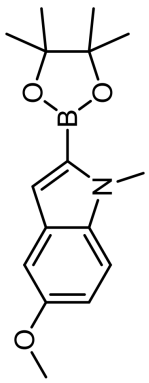
X : parts per Million : Proton

```
Filename          = TFP444_conc_1H-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP444_conc
Solvent           = CHLOROFORM-D
Creation_Time     = 22-OCT-2014 19:57:49
Revision_Time     = 21-JAN-2015 17:15:50
Current_Time      = 21-JAN-2015 17:16:27

Comment           = TFP444_conc_1H
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 20[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



in $CDCl_3$

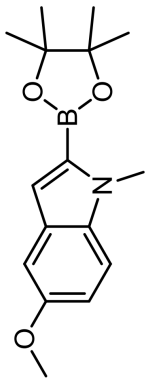
X : parts per Million : Carbon13

```
Filename          = TFP444_conc_13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP444_conc
Solvent           = CHLOROFORM-D
Creation_Time     = 22-OCT-2014 19:59:18
Revision_Time     = 21-JAN-2015 17:19:14
Current_Time      = 21-JAN-2015 17:19:50

Comment           = TFP444_conc_13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 180
Total_Scans       = 180

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.9[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```



in C_6D_6

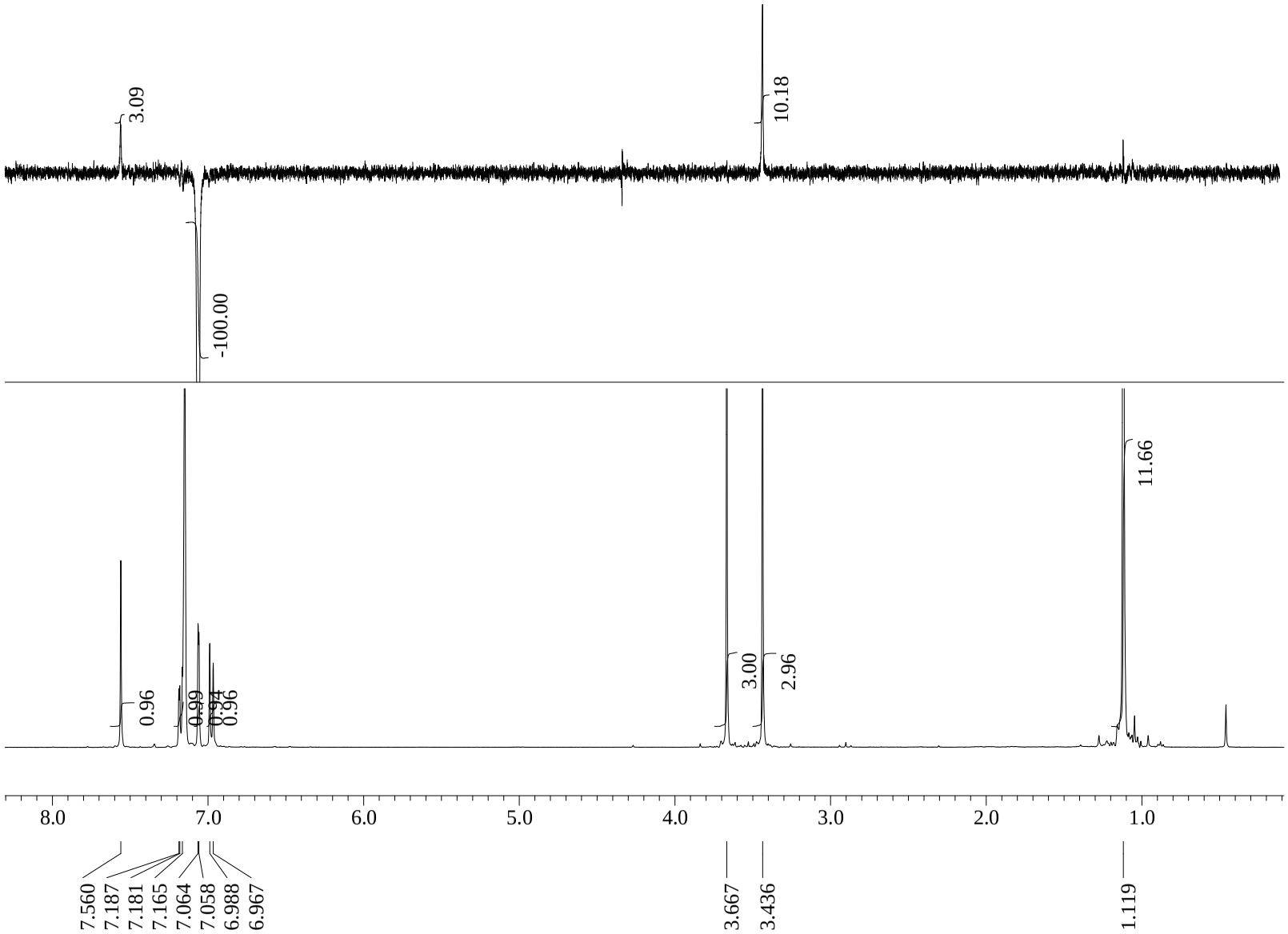
X : parts per Million : Proton

```
Filename          = TFP_5MeOIndBpin-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_5MeOIndBpin
Solvent           = BENZENE-D6
Creation_Time     = 18-JAN-2015 09:53:37
Revision_Time     = 21-JAN-2015 17:29:13
Current_Time      = 21-JAN-2015 17:33:16

Comment           = TFP_5MeOIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 18.1[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



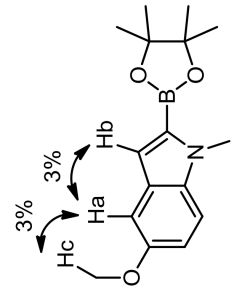
X : parts per Million : Proton

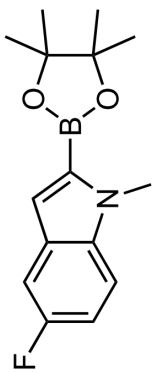
```
Filename          = TFP_5MeOIndBpin-d6benz_1H_
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP_5MeOIndBpin
Solvent           = BENZENE-D6
Creation_Time     = 18-JAN-2015 09:53:37
Revision_Time     = 21-JAN-2015 17:33:09
Current_Time      = 21-JAN-2015 17:37:52

Comment           = TFP_5MeOIndBpin
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 18.1[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```





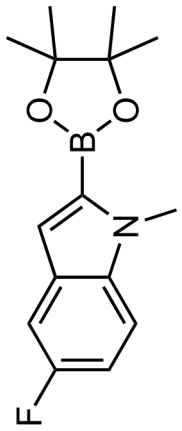
X : parts per Million : Proton

```
Filename          = Furukawa_TFP600_D_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = Furukawa_TFP600_D
Solvent           = BENZENE-D6
Creation_Time     = 12-FEB-2015 17:21:25
Revision_Time     = 12-FEB-2015 18:16:55
Current_Time      = 12-FEB-2015 18:19:38

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 2.20725248[s]
X_Domain          = 1H
X_Freq            = 395.88430144[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45305193[Hz]
X_Sweep           = 7.42280285[kHz]
X_Sweep_Clipped   = 5.93824228[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 395.88430144[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 22.9[dC]
X_90_Width        = 10.7[us]
X_Acq_Time        = 2.20725248[s]
X_Angle           = 45[deg]
X_Atn             = 2.2[dB]
X_Pulse           = 5.35[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.20725248[s]
```



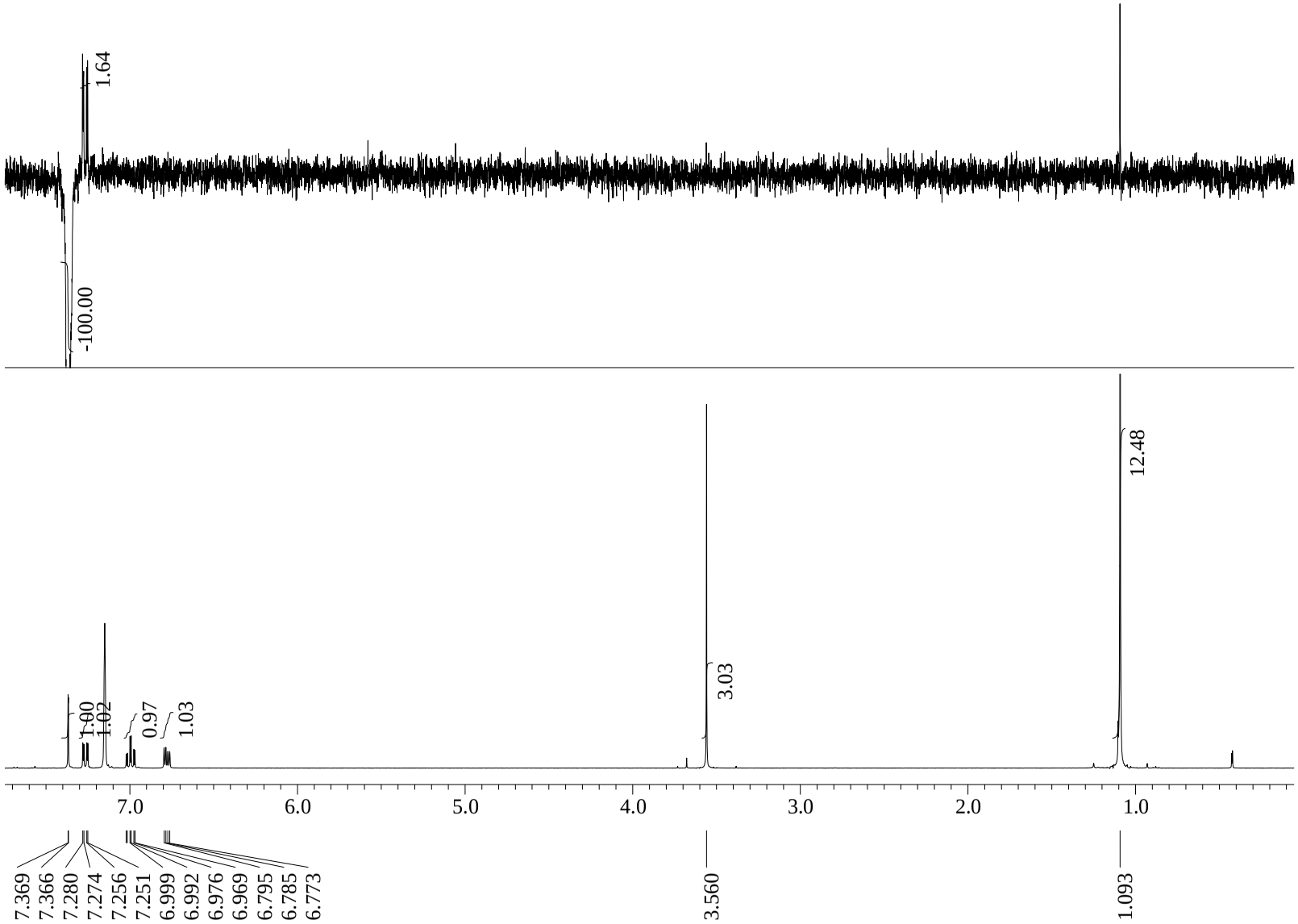
X : parts per Million : Carbon13

```
Filename          = Furukawa_TFP600_D_Carbon-1
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = Furukawa_TFP600_D
Solvent           = BENZENE-D6
Creation_Time     = 12-FEB-2015 17:26:48
Revision_Time     = 12-FEB-2015 18:22:27
Current_Time      = 12-FEB-2015 18:23:16

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 1.048576[s]
X_Domain          = 13C
X_Freq            = 99.54517646[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95367432[Hz]
X_Sweep           = 31.25[kHz]
X_Sweep_Clipped   = 25[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 400
Total_Scans       = 400

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 23.1[dC]
X_90_Width        = 9.45[us]
X_Acq_Time        = 1.048576[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.15[us]
Irr_Atn_Dec       = 22.826[dB]
Irr_Atn_Noe       = 22.826[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.048576[s]
```



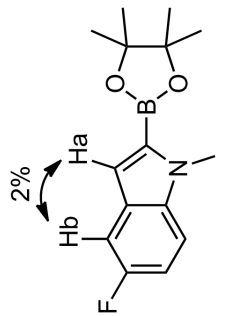
X : parts per Million : Proton

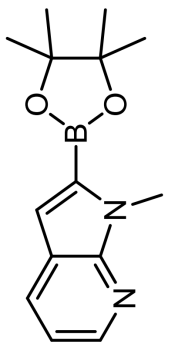
```
Filename          = Furukawa_TFP600_D_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = Furukawa_TFP600_D
Solvent           = BENZENE-D6
Creation_Time     = 12-FEB-2015 17:21:25
Revision_Time     = 12-FEB-2015 18:16:55
Current_Time      = 12-FEB-2015 18:29:00

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 2.20725248[s]
X_Domain          = 1H
X_Freq            = 395.88430144[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45305193[Hz]
X_Sweep           = 7.42280285[kHz]
X_Sweep_Clipped   = 5.93824228[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 395.88430144[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 22.9[dC]
X_90_Width        = 10.7[us]
X_Acq_Time        = 2.20725248[s]
X_Angle           = 45[deg]
X_Atn             = 2.2[dB]
X_Pulse           = 5.35[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.20725248[s]
```





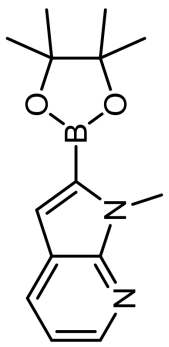
X : parts per Million : Proton

```
Filename          = TFP490-1-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP490-1
Solvent           = BENZENE-D6
Creation_Time     = 26-NOV-2014 16:22:02
Revision_Time     = 21-JAN-2015 19:10:00
Current_Time      = 21-JAN-2015 19:10:37

Comment           = TFP490-1
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 19.1[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```




```
Filename          = TFP490-1conc._13C-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = TFP490-1conc.
Solvent           = BENZENE-D6
Creation_Time     = 26-NOV-2014 16:39:12
Revision_Time     = 21-JAN-2015 19:12:49
Current_Time      = 21-JAN-2015 19:13:28

Comment           = TFP490-1conc._13C
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

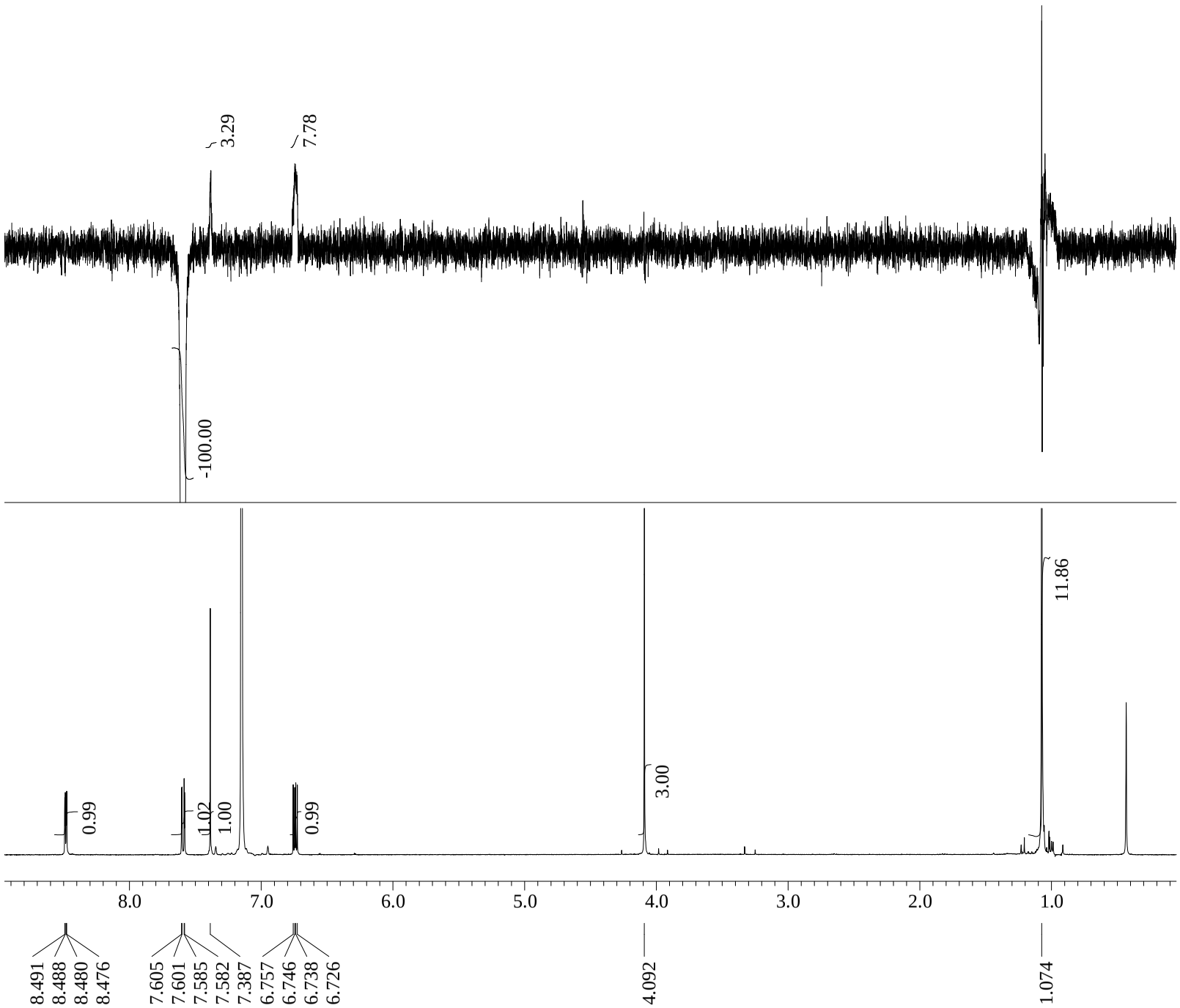
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 19.5[dC]
X_90_Width        = 9.2[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.06666667[us]
Irr_Atn_Dec       = 21.386[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = FALSE
Repetition_Time   = 3.04333312[s]
```

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

151.169 145.343 129.354 120.239 115.901 112.983 83.731 30.900 24.740

X : parts per Million : Carbon13



X : parts per Million : Proton

```
Filename          = TFP490-1-1_ana-1.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = TFP490-1
Solvent           = BENZENE-D6
Creation_Time     = 26-NOV-2014 16:22:02
Revision_Time     = 21-JAN-2015 19:10:32
Current_Time      = 21-JAN-2015 19:25:12

Comment           = TFP490-1
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 19.1[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```