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Electronic Supplementary Information

Fluorine-Controlled C–H Borylation of Arenes Catalyzed by PSiN-Pincer Platinum Complex

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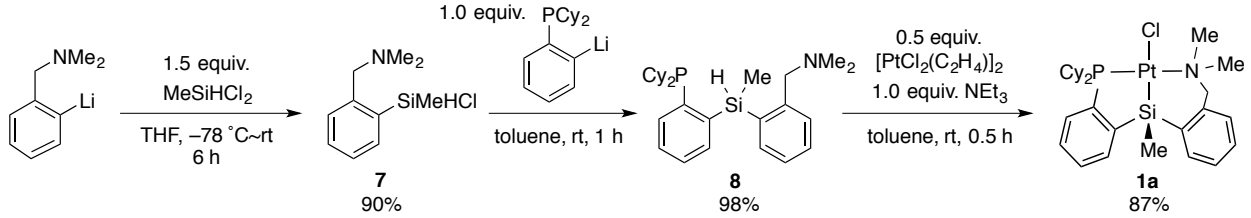
Table of Contents

2	General methods
2-4	Procedure for the preparation of PSiN-PtCl **1**
4	Procedure for the C–H borylation of arenes
4-11	Spectral data of borylation products
11	Iridium-catalyzed borylation of 1,2,4-trifluorobenzenes
12	Borylation of 1,2,4,5-tetrafluorobenzene with HBpin
12-13	References
14-72	NMR spectra

General: All operations were performed under an argon atmosphere. ^{1}H, ^{13}C, ^{19}F and ^{31}P-NMR spectra were recorded on a JEOL ECX-400 (400 MHz for ^{1}H, 100 MHz for ^{13}C, 160 MHz for ^{31}P and 368 MHz for ^{19}F) or a JEOL ECS-400 (400 MHz for ^{1}H, 100 MHz for ^{13}C, 160 MHz for ^{31}P and 368 MHz for ^{19}F) or a Bruker DRX-500 (500 MHz for ^{1}H, 125 MHz for ^{13}C) spectrometer. ^{1}H and ^{13}C{^{1}H} NMR chemical shifts are expressed in parts per million (ppm) downfield from tetramethylsilane and are referenced to residual solvents (δ_H 7.26 and δ_C 77.0 for chloroform, δ_H 7.15 and δ_C 128.6 for benzene). ^{31}P NMR chemical shifts are expressed in ppm downfield from an 85% H_3PO_4 external standard. ^{19}F NMR spectra are referenced relative to hexafluorobenzene (–162.9 ppm). IR spectra were recorded on an FT/IR-VIR-200 (JASCO Co., Ltd.). Silica Gel 60 (Kanto Chemical Co., Inc.) was used for flash column chromatography. Merck Kieselgel 60 F254 (0.25 mm thickness, coated on glass 20×20 cm^2) plate was used for analytical thin layer chromatography (TLC), and Wakogel B-5F coated on glass in a thickness of 0.9 mm was used for preparative TLC. High resolution mass analyses (FD$^+$ and EI$^+$) were performed on a JEOL JMS-T100 GSV mass spectrometer or a JEOL JMS-700 mass spectrometer. Elemental analyses were performed on an Elementar Vario MICRO. Unless otherwise noted, arenes, diboron, and mesitylene were used after distillation or recrystallization. THF, Et_2O, pentane and toluene were purified by solvent purification system of Glass-Contour. PSiP-platinum complex **1e** were prepared according to the reported procedures.[1]

Preparation of PSiN-PtCl complex **1a**



Synthesis of chlorosilane **7**

To a stirred solution of *o*-(*N*,*N*-dimethylaminomethyl)phenyllithium[2] (571 g, 4.05 mmol) in THF (20 ml) was added dichloromethylsilane (0.6 mL, 5.76 mmol) at –78 ˚C. The mixture was allowed to stand at room temperature and was stirred for 6 h. The solvent was removed under reduced pressure, and the mixture was filtered with toluene through a short pad of Celite. Evaporation of the solvent afforded a pale yellow solid, which was purified by recrystallization from Et_2O/pentane to give **7** as a white solid (0.774 g, 3.63 mmol) in 90% yield.[3]

^{1}H NMR ($CDCl_3$, 400 MHz) δ = 0.59 (s, 3H), 1.49 (s, 6H), 2.77 (d, *J* = 14.0 Hz, 1H), 2.95 (d, *J* = 14.0 Hz, 1H), 5.20-5.30 (m, 1H), 6.71 (d, *J* = 7.6 Hz, 1H), 7.10-7.50 (m, 2H), 8.66 (d, *J* = 6.8 Hz, 1H).

Synthesis of PN-mixed silane **8**

o-(Dicyclohexylphosphino)phenyllithium-diethylether (1.06 g, 3.0 mmol), which was prepared from (*o*-bromophenyl)dicyclohexylphosphine and *n*-BuLi, was added to a stirred solution of chlorosilane **7** (643 mg, 3.0 mmol) in toluene (5.0 mL) at room temperature. After 1 h, the mixture was filtered through a short pad of Celite to give P,N-mixed silane **8** (1.33 g, 2.94 mmol) in 98% yield, which was used for the next step without further purification.

IR (ATR) 2921, 2844, 2155, 1445 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 0.91 (s, 3H), 1.05-2.00 (m, 22H), 2.14 (s, 6H), 3.40 (d, *J* = 12.8 Hz, 1H), 3.90 (d, *J* = 12.8 Hz, 1H), 5.80-5.90 (m, 1H), 7.01-7.32 (m, 4H), 7.38-7.42 (m, 1H), 7.49-7.56 (m, 1H), 7.66-7.72 (m, 1H), 7.74-7.80 (m, 1H).; ^{13}C NMR ($CDCl_3$, 125 MHz) δ = 146.5 (d, *J* = 44.4 Hz), 145.5, 142.9 (d, *J* = 16.0 Hz), 136.9 (d, *J* = 3.9 Hz), 136.2 (d, *J* = 2.4 Hz), 135.3 (d, *J* = 14.8 Hz), 132.0 (d, *J* = 2.5 Hz), 128.4, 128.1, 127.9, 127.3, 125.6, 63.9, 44.4, 34.9 (d, *J* = 7.8 Hz), 34.8 (d, *J* = 7.6 Hz), 30.3 (d, *J* = 16.5 Hz),

30.1 (d, J = 15.6 Hz), 29.4 (d, J = 3.8 Hz), 29.3 (d, J = 3.8 Hz), 26.8-27.7 (m), 26.1 (d, J = 7.9 Hz), –3.2 (d, J = 9.6 Hz); ^{31}P NMR ($CDCl_3$, 160 MHz) = –7.48 (s); HRMS (FD^+) Calcd for $C_{28}H_{42}PNSi$, M 451.2824. Found m/z 451.2819.

Synthesis of PSiN-platinum complex **1a**

To a stirred solution of silane **8** (0.678 g, 1.50 mmol) in toluene (9 ml) was added $[PtCl_2(C_2H_4)]_2$ (0.44 g, 0.75 mmol) and NEt_3 (250 μL, 1.79 mmol) at room temperature. After 1 h, the mixture was filtered through a short pad of Celite, and the solvent was removed under reduced pressure. Extraction with Et_2O and reprecipitation from Et_2O/pentane afforded **1a** (0.676 g, 0.99 mmol) in 66% yield.

IR (ATR) 2928, 1466 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ = 0.73 (s, 3H), 0.82-1.84 (m, 18H), 2.25-2.40 (m, 4H), 2.56 (d, J = 1.5 Hz, 3H), 2.99 (d, J = 1.5 Hz, 3H), 3.15-3.25 (m, 1H), 4.29 (d, J = 12.3 Hz, 1H), 6.82 (d, J = 8.0 Hz, 1H), 6.95-7.20 (m, 3H), 7.25-7.30 (m, 1H), 7.43 (t, J = 7.1, 1H), 7.80 (d, J = 7.0 Hz, 1H), 7.87 (d, J = 7.3 Hz, 1H); ^{13}C NMR (CD_2Cl_2, 100 MHz) δ = 152.7 (d, J = 36.4 Hz), 141.8, 141.6, 141.2, 134.1 (d, J = 20.3 Hz), 131.7, 131.6, 131.1 (d, J = 6.4 Hz), 130.7, 129.4 (d, J = 7.5 Hz), 128.7, 128.4, 71.1, 51.8, 47.7, 36.3 (d, J = 7.5), 36.0 (d, J = 7.5 Hz), 30.0-26.7 (m), 2.3; ^{31}P NMR (C_6D_6, 160 MHz) = 52.4 (s, J_{PPt} = 4174 Hz); HRMS (FD^+) Calcd for $C_{28}H_{41}PPtSiNCl$, M 681.2082. Found m/z 680.2115. *Anal.* Calcd for $C_{28}H_{41}PPtSiNCl$: C, 49.37; H, 6.07; N, 2.06. Found: C, 48.70; H, 6.06; N, 2.06.

Formation of PSiN-pincer type structure was clearly supported by observation of the J_{PtP} coupling and separately observed two methyl protons on the nitrogen and benzylic methylene protons. Furthermore, the structure of a PSiN-platinum triflate complex **1a'**, derived from **1a** and AgOTf, was confirmed by X-ray analysis (Figure S1, CCDC 1419522).

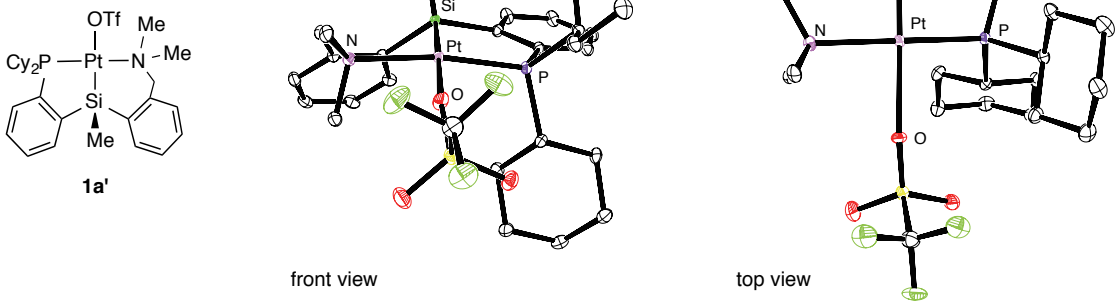
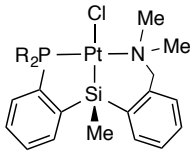


Figure S1. ORTEP drawings of the PSiN-platinum triflate complex **1a'** at 30% probability level. Hydrogen atoms are omitted for clarity.



Other platinum complexes were also synthesized according to the procedure for **1a**.

1b (R = *i*-Pr)

IR (ATR) 2961, 1448, 1234 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 0.79 (t, J_{PtH} = 27.2, 3H), 0.97 (dd, J = 16.4, 6.8 Hz, 3H), 1.04 (dd, J = 13.6, 6.8 Hz 3H), 1.22 (dd, J = 14.0, 6.8 Hz 3H), 1.65 (dd, J = 16.0, 6.8 Hz 3H), 2.64 (s, 3H), 3.01 (s, 3H), 3.25-3.40 (m, 1H), 4.23 (d , J = 12.0, 3H), 6.92 (d, J = 7.6 Hz, 1H), 7.12-7.37 (m, 5H), 7.83 (d, J = 8.0

Hz, 1H), 7.92 (d, J = 6.8 Hz, 1H); ^{13}C NMR (CD_2Cl_2, 125 MHz) δ = 151.6 (d, J = 35.0 Hz), 140.8, 140.6, 140.2, 133.3 (d, J = 19.3 Hz), 130.8, 130.7, 130.0, 129.9 (d, J = 3.6 Hz), 128.6 (d, J = 7.9 Hz), 127.8, 127.6, 70.2, 50.8, 46.7, 25.9 (d, J = 33.5 Hz), 25.5 (d, J = 34.6 Hz), 18.9 (d, J = 1.4 Hz), 18.7 (d, J = 2.3 Hz), 18.2 (d, J = 2.1 Hz), 17.2 (d, J = 3.6 Hz), 1.19; ^{31}P NMR (C_6D_6, 202 MHz) = 59.5 (s, J_{PPt} = 4177 Hz); HRMS (FD$^+$) Calcd for $C_{22}H_{33}ClNPPtSi$, M 600.1456. Found m/z 600.1466.

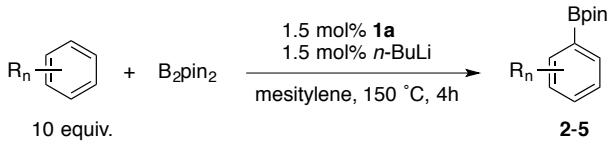
1c (R = *t*-Bu)

IR (ATR) 2890, 1473, 1174, 1107 cm^{-1}; ^{1}H NMR (CD_2Cl_2, 500 MHz) δ = 0.60 (s, 3H), 1.35 (d, J = 14.2 Hz, 9H), 1.43 (d, J = 14.5 Hz, 9H), 2.64 (s, 3H), 3.23 (s, 3H), 3.55 (dd, J = 12.6, 6.2 Hz, 1H), 4.56 (d, J = 12.5 Hz, 1H), 7.20-7.30 (m, 3H), 7.41-7.48 (m, 2H), 7.52-7.57 (m, 1H), 7.90 (d, J = 7.3 Hz, 1H), 7.94 (dd, J = 8.1, 5.1 Hz, 1H); ^{13}C NMR (CD_2Cl_2, 125 MHz) δ = 151.3 (d, J = 34.8 Hz), 141.1, 140.7 (d, J = 4.4 Hz), 140.6, 133.5 (d, J = 18.9 Hz), 133.1 (d, J = 5.5 Hz), 130.7, 130.5, 129.6 (d, J = 5.9 Hz), 127.7, 127.5, 127.4 (d, J = 6.9 Hz), 69.9, 50.8 (d, J = 2.3 Hz), 47.0 (d, J = 2.0 Hz), 36.9 (d, J = 26.3 Hz), 36.7 (d, J = 24.0 Hz), 30.5 (d, J = 7.9 Hz), 30.4 (d, J = 8.1 Hz), 1.1; ^{31}P NMR (C_6D_6, 202 MHz) = 76.4; HRMS (FD$^+$) Calcd for $C_{24}H_{37}ClNPPtSi$, M 628.1769. Found m/z 628.1753.

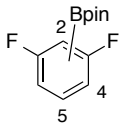
1d (R = Ph)

IR (ATR) 2360, 1431, 1103 cm^{-1}; ^{1}H NMR (C_6D_6, 400 MHz) δ = 0.73 (s, 3H), 2.57 (s, 3H), 2.69 (s, 3H), 2.90-3.10 (m, 1H), 4.20-4.40 (m, 1H), 6.80-7.40 (m, 14H), 7.60-8.20 (m, 6H); ^{31}P NMR (C_6D_6, 202 MHz) = 36.1 (s, J_{PPt} = 4159 Hz); HRMS (FD$^+$) Calcd for $C_{28}H_{29}ClNPPtSi$, M 668.1143. Found m/z 668.1148.

Procedure for the C–H borylation of arenes



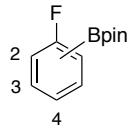
To a solution of **1a** (4.08 mg, 0.0059 mmol) in mesitylene (2 mL) was added a *n*-hexane solution of *n*-BuLi (1.56 M, 3.85 μL, 0.4 mmol) followed by addition of B_2pin_2 (101.3 mg, 0.4 mmol) and arene (4 mmol) at room temperature. The mixture was stirred at 150 ˚C for 4 h. After the solvent was removed under reduced pressure, the obtained crude material was purified by silica gel column chromatography to give arylboronic esters as an inseparable mixture of regioisomers. Silica gel column chromatography was carried out by cooling the column at –30 ˚C to avoid decomposition of the arylboronic ester on silica gel. The ratios of regioisomers were determined by ^{1}H NMR or ^{19}F NMR.



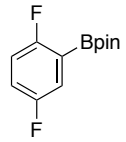
2a (82.9 mg, 0.345 mmol, 86%, 2-:4-:5- =74:22:4 (Entry 1, Table 1)). The ratio of regioisomers was determined by ^{19}F NMR of the isolated product, which was almost identical to that in the crude material. The ratio was slightly improved to 2-:4-:5- =78:18:4 by carrying out the reaction at 120 ˚C (Entry 2, Table 1).

^{1}H NMR ($CDCl_3$, 400 MHz) **2-2a**: δ = 1.38 (s, 12H), 6.81-6.87 (m, 2H), 7.31-7.39 (m, 1H); **4-2a**: δ = 1.35 (s, 12H), 6.73-6.80 (m, 1H), 6.83-6.90 (m, 1H), 7.69-7.76 (m, 1H); ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2a**: δ = –101.8; **4-2a**: δ = –99.8, –106.3; **5-2a**: δ = –112.0.

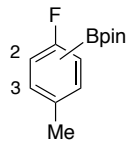
Spectral data were in good agreement with literature values.[4]



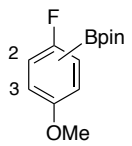
2b (45.7 mg, 0.206 mmol, 51%, 2-:3-:4- =80:17:3 (Entry 1, Table 2)). The ratio of regioisomers was determined by ^{19}F NMR of the isolated product, which was almost identical to that in the crude material. The ratio was slightly improved to 2-:3-:4- =84:13:3 by carrying out the reaction at 120 ˚C (Entry 2, Table 2).
^{1}H NMR ($CDCl_3$, 400 MHz) **2-2b**: δ = 7.71-7.76 (m, 1H), 7.40-7.60 (m, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.02 (br t, *J* = 9.4 Hz, 1H), 1.36 (s, 12H); **3-2a**: δ = 7.56 (d, *J* = 7.2 Hz, 1H), 7.50-7.55 (m, 1H), 7.30-7.37 (m, 1H), 1.35 (s, 12H) (one aromatic proton was overlapped); **4-2a**: δ = 7.77-7.81 (m, 2H), 1.34 (s, 12H) (two aromatic protons were overlapped); ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2b**: δ = –103.8; **3-2b**: δ = –115.4; **4-2b**: δ = –109.6.
Spectral data were in good agreement with literature values.[5]



2c (66.0 mg, 0.275 mmol, 69%)
^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.38 (q, *J* = 4.0 Hz, 1H), 7.05-7.12 (m, 1H), 6.95-7.00 (m, 1H), 1.36 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) δ = –110.6, –121.8.
Spectral data were in good agreement with literature values.[6]



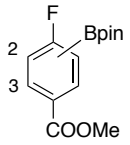
2d (79.4 mg, 0.33 mmol, 84%, 2-:3- =96:4). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material. The borylated position (2- or 3-) was determined by ^{1}H NMR, in which the *ortho*-proton to the Bpin appeared to be doublet of doublet with smaller J_{FH} (~5 Hz) in **2-2d** than that in **3-2d** (~10 Hz).
IR (ATR) 2983, 1613, 1492, 1409, 1347, 1216, 1144 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) **2-2d**: δ = 7.52 (dd, *J* = 5.5, 2.3 Hz, 1H), 7.19-7.24 (m, 1H), 6.91 (t, *J* = 8.7 Hz, 1H), 1.36 (s, 12H); **3-2d**: δ = 7.42 (dd, *J* = 9.4, 2.5 Hz, 1H), 7.11 (dd, *J* = 8.6, 4.9 Hz, 1H), 6.96-7.01 (m, 1H), 1.34 (s, 12H) ; ^{13}C NMR ($CDCl_3$, 100 MHz) **2-2d**: δ = 165.3 (d, *J* = 284 Hz), 136.9 (dd, *J* = 60.6, 20.9 Hz), 133.3 (m), 131.1 (d, *J* = 9.4 Hz), 114.9 (d, *J* = 24 Hz), 83.6 , 24.8, 20.4; ^{9}F NMR ($CDCl_3$, 368 MHz) **2-2d**: δ = –108.2; **3-2d**: δ = –119.6; HRMS (FD^{+}) Calcd for $C_{13}H_{18}BFO_2$, M 236.1384. Found m/z 236.1411.



2e (92.8 mg, 0.39 mmol, 92%, 2-:3- =87:13). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material. The borylated position (2- or 3-) was determined

by analyzing the J_{FH} value as described for **2d**.

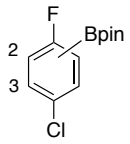
IR (KBr) 2979, 1492, 1418, 1349, 1207, 1145 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) **2-2e**: δ = 7.18-7.21 (m, 1H), 6.92-6.97 (m, 2H), 3.81 (s, 3H), 1.36 (s, 12H); **3-2e**: δ = 7.34 (dd, *J* = 12.0, 3.36 Hz, 1H), 7.02-7.09 (m, 1H), 6.78 (dd, *J* = 13.9, 4.03 Hz, 1H), 3.81 (s, 3H), 1.35 (s, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz) **2-2e**: δ = 161.6 (d, *J* = 242 Hz), 155.2, 119.8 (d, *J* = 8.4 Hz), 119.3 (d, *J* = 8.4 Hz), 116.0 (d, *J* = 26 Hz), 83.9, 55.8, 24.8; ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2e**: δ = –113.9; **3-2e**: δ = –125.2; HRMS (FD^+) Calcd for $C_{13}H_{18}BFO_3$, M 252.1333. Found m/z 252.1377.



2f (43.9 mg, 0.157 mmol, 78% (from 0.20 mmol B_2pin_2), 2- only). The selective borylation at the 2-position was confirmed by analyzing the J_{FH} value as described for **2d**.

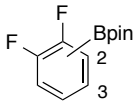
IR (KBr) 2982, 1352 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ = 8.44 (dd, *J* = 3.52, 2.34 Hz, 1H), 8.03-8.08 (m, 1H), 7.07 (t, *J* = 10.6 Hz, 1H), 3.91 (s, 3H), 1.37 (s, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 169.9 (d, *J* = 259 Hz), 166.2, 138.9 (d, *J* = 9.4 Hz), 135.0 (d, *J* = 10.5 Hz), 125.9 (d, *J* = 3.1 Hz), 115.6 (d, *J* = 25.2 Hz), 84.2, 52.1, 24.8; ^{19}F NMR ($CDCl_3$, 368 MHz) δ = –95.2.

HRMS (FD^+) Calcd for $C_{14}H_{18}BFO_4$, M 280.1282. Found m/z 280.1276.



2g (47 mg, 0.18 mmol, 91% (from 0.20 mmol B_2pin_2), 2-:3- =83:17). The ratio of regioisomers was determined by ^1H NMR of the isolated product, which was almost identical to that in the crude material. The borylated position (2- or 3-) was determined by analyzing the J_{FH} value as described for **2d**.

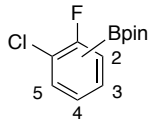
IR (KBr) 2982, 1352, 1299, 1134 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) **2-2g**: δ = 7.68 (dd, *J* = 5.0, 3.8 Hz, 1H), 7.34-7.40 (m, 1H), 6.97 (t, *J* = 8.8 Hz, 1H), 1.36 (s, 12H); **3-2g**: δ = 7.34-7.40 (m, 1H), 7.30 (dd, *J* = 9.2, 4.8 Hz, 1H), 7.00-7.05 (m, 1H), 1.35 (s, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz) **2-2g**: δ = 165.5 (d, *J* = 249 Hz), 136.2 (d, *J* = 5 Hz), 132.9 (d, *J* = 8.6 Hz), 116.8 (d, *J* = 36.3 Hz), 84.3, 24.8; ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2g**: δ = –105.8; **3-2g**: δ = –117.1; HRMS (FD^+) Calcd for $C_{12}H_{15}BFClO_2$, M 256.0838. Found m/z 256.0843.



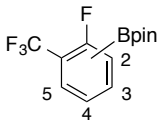
2h (71.5 mg, 0.29 mmol, 74%, 2-:3- =91:9). The ratio of regioisomers was determined by ^1H NMR of the isolated product, which was almost identical to that in the crude material.

^1H NMR ($CDCl_3$, 400 MHz) **2-2h**: δ = 7.43-7.48 (m, 1H), 7.19-7.28 (m, 1H), 7.03-7.10 (m, 1H), 1.36 (s, 12H); **3-2g**: δ = 7.50-7.61 (m, 2H), 7.10-7.20 (m, 1H), 1.36 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2h**: δ = –130.2, –140.2; **3-2h**: δ = –135.1, –140.8.

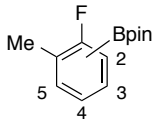
Spectral data were in good agreement with literature values.[7,8]



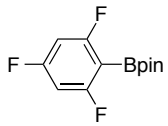
2i (35.2 mg, 0.13 mmol, 69% (from 0.2 mmol B_2pin_2), 2-:3-:4-:5-=65:6:8:21). The ratio of regioisomers was determined by ^{19}F NMR of the isolated product, which was almost identical to that in the crude material.
^1H NMR ($CDCl_3$, 400 MHz) **2-2i**: δ = 7.62 (d,t, *J* = 1.60, 8.38 Hz, 1H), 7.48 (dt, *J* = 1.20, 7.58 Hz, 1H), 7.08 (t, *J* = 7.98 Hz, 1H), 1.39 (s, 12H); **3-2i**: δ = 7.56 (d, *J* = 9.62 Hz, 1H), 7.5 (dd, *J* = 1.30, 7.80 Hz, 1H), 7.41 (t, *J* = 7.06 Hz, 1H), 1.36 (s, 12H); **4-2i**: δ = 7.88 (d, *J* = 12.6 Hz, 1H), 7.7-7.68 (m, 1H), 7.17-7.13 (m, 1H), 1.36 (s, 12H); **5-2i**: δ = 7.45-7.50 (m, 1H), 7.17-7.22 (m, 2H), 1.37 (s, 12H) (aromatic protons are overlapped); ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2i**: δ = –105.8; **3-2i**: δ = –117.1; **4-2i**: δ = –111.1; **5-2i**: δ = –114.4.
Spectral data were in good agreement with literature values of **2-2i**, **3-2i**, and **4-2i**[9,10] and those of authentic sample of **5-2i** derived from commercially available corresponding boronic acid.



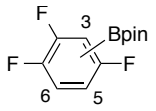
2j (73%, 2-:3-:4-:5-=75:16:9:–). The ratio of regioisomers was determined by ^{19}F NMR of the crude product due to partial decomposition of the products after column chromatography. The NMR yield of **2-2j** in the crude mixture was shown using 1,1,2,2-tetrachloroethane as an internal standard.
^1H NMR ($CDCl_3$, 400 MHz) **2-2j**: δ = 7.89 (t, *J* = 8.0 Hz, 1H), 7.68 (t, *J* = 6.0 Hz, 1H), 7.18-7.26 (m, 1H), 1.36 (s, 12H); **3-2j**: δ = 7.55-7.65 (m, 3H), 1.32 (m, 12H); **4-2j**: δ = 8.03 (d, *J* = 7.6 Hz, 1H), 7.88-7.96 (m, 1H), 7.14-7.18 (m, 1H), 1.35 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2j**: δ = –61.5, –104.0; **3-2j**: δ = –61.5, –116.0; **4-2j**: δ = –61.5, –110.3.
Spectral data were in good agreement with literature values.[11,12,13]



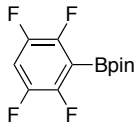
2k (23.4 mg, 0.99 mmol, 50% (from 0.2 mmol B_2pin_2), 2-:3-:4-:5-=71:16:7:6). The ratio of regioisomers was determined by ^{19}F NMR of the isolated product, which was almost identical to that in the crude material.
^1H NMR ($CDCl_3$, 400 MHz) **2-2k**: δ = 7.54 (t, *J* = 5.7 Hz, 1H), 7.27 (t, *J* = 7.8 Hz, 1H), 7.02 (t, *J* = 7.6 Hz, 1H), 2.25 (d, *J* = 2.0 Hz, 1H), 1.36 (s, 12H); **3-2k**: δ = 7.38-7.46 (m, 2H), 7.17 (t, *J* = 7.4 Hz, 1H), 2.28 (d, *J* = 1.8 Hz, 3H), 1.31 (s, 12H); **4-2k**: δ = 7.57-7.65 (m, 2H), 6.95-7.02 (m, 1H), 2.21 (s, 3H), 1.31 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) **2-2k**: δ = –106.8; **3-2k**: δ = –119.0; **4-2k**: δ = –112.8; **5-2k**: δ = –117.3.
Spectral data were in good agreement with literature values of **2-2k**, **3-2k**, and **4-2k**.[14,15] and those of authentic sample of **5-2k** derived from commercially available corresponding boronic acid.



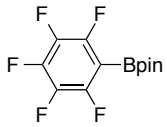
2l (116 mg, 0.449 mmol, 112%)
^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.55-6.65 (m, 2H), 1.36 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) δ = –98.4, –105.1. Spectral data were in good agreement with literature values.[16]



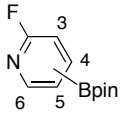
2m (83 mg, 0.32 mmol, 80%, 3-:5-:6-=73:19:8). The ratio of regioisomers was determined by ^{19}F NMR of the isolated product, which was almost identical to that in the crude material.
IR (KBr) 1634, 1482 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) **3-2m**: δ = 7.10-7.20 (m, 1H), 6.72-6.80 (m, 1H), 1.37 (s, 12H); **5-2m**: δ = 7.10-7.20 (m, 1H), 6.94-7.02 (m, 1H), 1.34 (s, 12H); **6-2m**: δ = 7.49 (ddd, *J* = 11.2, 9.7, 4.7 Hz, 1H), 6.82-6.90 (m, 1H), 1.33 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) **3-2m**: δ = –107.5 ~ –107.6 (m), –126.1 (dd, *J* = 21.0, 8.5 Hz), –144.8 ~ –145.0 (m); **5-2m**: δ = –117.4 ~ –117.5 (m), –126.1 (dd, *J* = 21.3, 8.5 Hz), –135.1 ~ –135.2 (m); **6-2m**: δ = –105.3 ~ –105.4 (m), –129.6 ~ –129.7 (m), –145.2 ~ –145.4 (m); *Anal.* Calcd for $C_{12}H_{14}BF_3O_2$: C, 55.85; H, 5.47. Found: C, 55.23; H, 5.22. The structure of **3-2m** was confirmed by NOE measurement, which demonstrated the two aromatic protons are next to each other. The identity of **5-2m** and **6-2m** was confirmed by independent synthesis of authentic samples by Pd-catalyzed borylation of 1-bromo-2,4,5-trifluorobenzene or 1-bromo-2,3,5-trifluorobenzene with B_2pin_2.



2n (74.4 mg, 0.270 mmol, 67%)
IR (KBr) 3057, 2985, 1479, 1379, 1336 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.05-7.15 (m, 1H), 1.41 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) δ = –110.5, –118.9; ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 148.7 (br d, *J* = 253 Hz), 145.5 (br d, *J* = 253 Hz), 108.5 (t, *J* = 22.5 Hz), 84.9, 24.7.

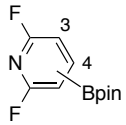


2o (77.9 mg, 0.26 mmol, 66%)
^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.37 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) δ = –130.6 ~ –130.7 (m, 2F), –150.1 ~ –150.9 (m, 1F), –162.3 ~ –163.0 (m, 2F);
Spectral data were in good agreement with literature values.[17] A small amount of 2,3,5,6-F_4C_6HBpin and 2,3,5,6-F_4-1,4-(Bpin)$_2$benzene were observed in ^{19}F NMR (<3%).

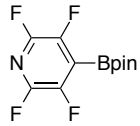


3a (77.4 mg, 0.344 mmol, 86%, 3-:4-:5-:6-=50:38:12:–). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product. The ^{1}H NMR of the crude mixture suggested the existence of an unidentified product, which might be **6-3a**, in less than 10% although it disappeared after column chromatography.

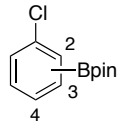
^{1}H NMR ($CDCl_3$, 400 MHz) **3-3a**: δ = 8.28-8.31 (m, 1H), 8.14 – 8.20 (m, 1H), 7.15-7.20 (m, 1H), 1.36 (s, 12H); **4-3a**: δ = 8.24 (d, *J* = 4.7 Hz, 1H), 7.47-7.51 (m, 1H), 7.28 (d, *J* = 2.0 Hz, 1H), 1.36 (s, 12H); **5-3a**: δ = 8.59 (s, 1H), 8.12-8.20 (m, 1H), 6.91 (dd, *J* = 4.1, 2.5 Hz, 1H), 1.35 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) **3-3a**: δ = –57.6; **4-3a**: δ = –68.7; **5-3a**: δ = –63.8;
Spectral data were in good agreement with literature values of **3-**, **4-**, and **5-3a**.[18,19]



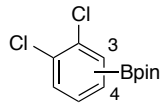
3b (69 mg, 0.28 mmol, 72%, 3-:4- = 69:31). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
IR (KBr) 1635, 1261 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) **3-3b**: δ = 8.24 (q, *J* = 8.2, 1H), 6.79-6.83 (m, 1H), δ = 1.34 (s, 12H); **4-3b**: δ = 7.14 (s, 2H), 1.34 (s, 12H); ^{19}F NMR ($CDCl_3$, 368 MHz) **3-3b**: δ = –57.0, –63.6; **4-3b**: δ = –69.2; ^{13}C NMR ($CDCl_3$, 100 MHz) **3-3b**: δ =165.6 (dd, *J* = 200, 11.3 Hz), 163.8 (dd, *J* = 200, 11.3 Hz), 152.4 (t, *J* = 7.8 Hz), 105.7 (dd, *J* = 32.8, 5.4 Hz), 84.5, 24.6; **4-3b**: δ = 161.4 (dd, *J* = 198, 10.8 Hz), 110.8 (dd, *J* = 21.5, 8.5 Hz), 85.1, 24.6; HRMS (EI$^+$) Calcd for $C_{11}H_{14}BF_2NO_2$, M 241.1086. Found m/z 241.1075.



3c (115mg, 0.41 mmol, 104%)
^{1}H NMR (C_6D_6, 400 MHz) δ = 1.01 (s, 12H); ^{19}F NMR (C_6D_6, 368 MHz) δ = –92.5, –132.6.
Spectral data were in good agreement with literature values.[20]

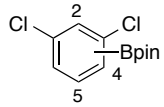


4a (41.2 mg, 0.17 mmol, 43%, 2-:3-:4-:=46:37:17). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
^{1}H NMR ($CDCl_3$, 400 MHz) **2-4a**: δ = 7.65 (d, *J* = 7.6 Hz, 1H), 7.30-7.36 (m, 2H), 7.19-7.24 (m, 1H), 1.36 (s, 12H); **3-4a**: δ = 7.77 (br s, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.38-7.43 (m, 1H), 7.26-7.32 (m, 1H), 1.33 (s, 12H); **4-4a**: δ = 7.71 (d, *J* = 8.4 Hz, 2H), 7.30-7.34 (m, 2H), 1.33 (s, 12H);
Spectral data were in good agreement with literature values.[21]

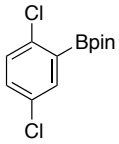


4b (82.8 mg, 0.30 mmol, 76%, 3-:4- = 60:40). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
^{1}H NMR ($CDCl_3$, 400 MHz) **3-4b**: δ = 7.55 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.50 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 1.37 (s, 12H); **4-4b**: δ = 7.86 (d, *J* = 1.4 Hz, 1H), 7.60 (d, *J* = 8.0, 1.4, 1H), 7.44 (d, *J* = 8.0, 1H), 1.34 (s,

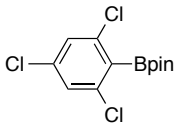
12H);
Spectral data were in good agreement with literature values.[22,23]



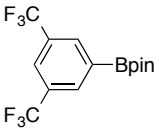
4c (99.7 mg, 0.36 mmol, 92%, 2-:4-:5- = 23:40:37). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
^{1}H NMR ($CDCl_3$, 400 MHz) **2-4c**: δ = 7.20-7.24 (m, 3H), 1.43 (s, 12H); **4-4c**: δ = 7.62 (d, *J* = 8.2 Hz, 1H), 7.36 (d, *J* = 2.0 Hz, 1H), 7.20-7.24 (m, 1H), 1.34 (s, 12H); **5-4c**: 7.64 (d, *J* = 2.0 Hz, 2H), 7.43 (t, *J* = 2.0 Hz, 1H), 1.36 (s, 12H);
Spectral data were in good agreement with literature values.[24,25,26]



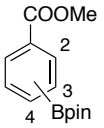
4d (93.7 mg, 0.34 mmol, 86%)
^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.65 (d, *J* = 2.5 Hz, 1H), 7.34-7.27 (m, 2H), 1.35 (s, 12H);
Spectral data were in good agreement with literature values.[27]



4e (70.8 mg, 0.23 mmol, 58%)
IR(ATR) 1577, 1535, 1323, 1109; ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 7.25 (s, 2H), 1.41 (s, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ =138.6, 136.2, 127.0, 85.2, 24.7; *Anal.* Calcd for $C_{12}H_{14}BCl_3O_2$: C, 46.89; H, 4.59. Found: C, 47.10; H, 4.77;

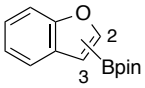


4f (83.0 mg, 0.244 mmol, 58%).
^{1}H NMR ($CDCl_3$, 400 MHz) δ = 8.23 (s, 1H), 7.93 (d, *J* = 14.8 Hz, 2H), 1.38 (s, 12H);
Spectral data were in good agreement with literature values.[28]

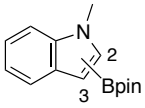


4g (49.6 mg, 0.189 mmol, 47%, 2-:3-:4- = –:58:42). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.

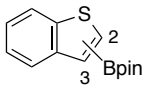
^{1}H NMR ($CDCl_3$, 400 MHz) **3-4g**: δ = 8.47 (s, 1H), 8.13 (dt, *J* = 8.0, 1.6 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 3.92 (s, 3H), 1.36 (s, 3H); **4-4g**: δ = 8.02 (d, *J* = 8.0 Hz, 2H), 7.87 (d, *J* = 8.0 Hz, 2H), 3.92 (s, 3H), 1.36 (s, 3H);
Spectral data were in good agreement with literature values.[29,30]



5a (91.9 mg, 0.37 mmol, 94%, 2-:3- = 86:14). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
^{1}H NMR ($CDCl_3$, 400 MHz) **2-5a**: δ = 7.63 (d, *J* = 7.8 Hz, 1H), 7.57 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.40 (d, *J* = 0.9 Hz, 1H), 7.32-7.36 (m, 1H), 7.20-7.26 (m, 1H), 1.39 (s, 12H); **3-5a**: δ = 7.95 (s, 1H), 7.91-7.94 (m, 1H), 7.47-7.52 (m, 1H), 7.23-7.29 (m, 2H), 1.37 (s, 12H);
Spectral data were in good agreement with literature values.[31]

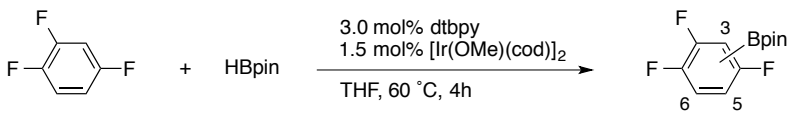


5b (69.7 mg, 0.27 mmol, 68%, 2-:3- = 91:9). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
^{1}H NMR ($CDCl_3$, 400 MHz) **2-5b**: δ = 7.64 (d, *J* = 8.3 Hz, 1H), 7.35 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.26 (td, *J* = 8.3, 0.9 Hz, 1H), 7.13 (s, 1H), 7.08 (td, *J* = 8.3, 0.9 Hz, 1H), 3.97 (s, 3H), 1.37 (s, 12H); **3-5a**: δ = 8.04 (br d, *J* = 7.6 Hz, 1H), 7.52 (s, 1H), 7.25-7.35 (m, 3H), 3.80 (s, 3H), 1.36 (s, 12H);
Spectral data were in good agreement with literature values.[32,33]



5c (79.2 mg, 0.304 mmol, 76%, 2-:3- = 84:16). The ratio of regioisomers was determined by ^{1}H NMR of the isolated product, which was almost identical to that in the crude material.
^{1}H NMR ($CDCl_3$, 400 MHz) **2-5c**: δ = 7.82-7.92 (m, 3H), 7.32-7.40 (m, 2H), 1.38 (s, 12H); **3-5c**: δ = 8.37 (d, *J* = 7.8 Hz, 1H), 8.07 (s, 1H), 7.88-7.92 (m, 1H), 7.36-7.42 (m, 2H), 1.38 (s, 12H);
Spectral data were in good agreement with literature values.[34,35]

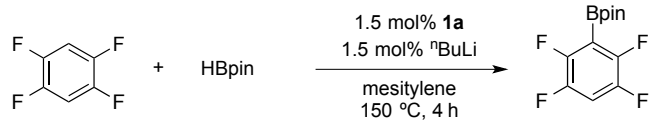
Iridium-catalyzed borylation of 1,2,4-trifluorobenzene (Concerning the footnote d in Figure 1)



To a solution of $[Ir(OMe)(cod)]_2$ (5.0 mg, 0.0075 mmol) and dtbpy (4.0 mg, 0.015 mmol) in THF (2 mL) was added HBpin (73 μL, 0.5 mmol) and 1,2,4-trifluorobenzene (208 μL, 2.0 mmol) at room temperature. After the

mixture was stirred at 60 ˚C for 4 h, the solvent was removed under reduced pressure to give a crude product. The yield and ratio of regioisomers were determined by ^{19}F NMR of the crude product using C_6F_6 (10 μL, 0.087 mmol) as an internal standard.

Borylation of 1,2,4,5-tetraflurorobenzene with HBpin instead of B_2pin_2 (Concerning the comment in ref 16.)



(10 equiv.)

2n
15% (NMR yield)

To a solution of **1a** (6.81 mg, 0.01 mmol) in mesitylene (2 mL) was added a *n*-hexane solution of *n*-BuLi (1.56 M, 6.4 μ L, 0.01 mmol) followed by addition of HBpin (25.9 mg, 0.2 mmol) and 1,2,4,5-tetrafuluorobenzene (2 mmol) at room temperature. The mixture was stirred at 150 ˚C for 4 h. After the solvent was removed under reduced pressure, formation of **2n** in 15% yield was confirmed by ^{19}F NMR. Therefore, it was demonstrated that HBpin instead of B_2pin_2 could be utilized as an atom economical boron source for this reaction, and further investigation on this is in progress.

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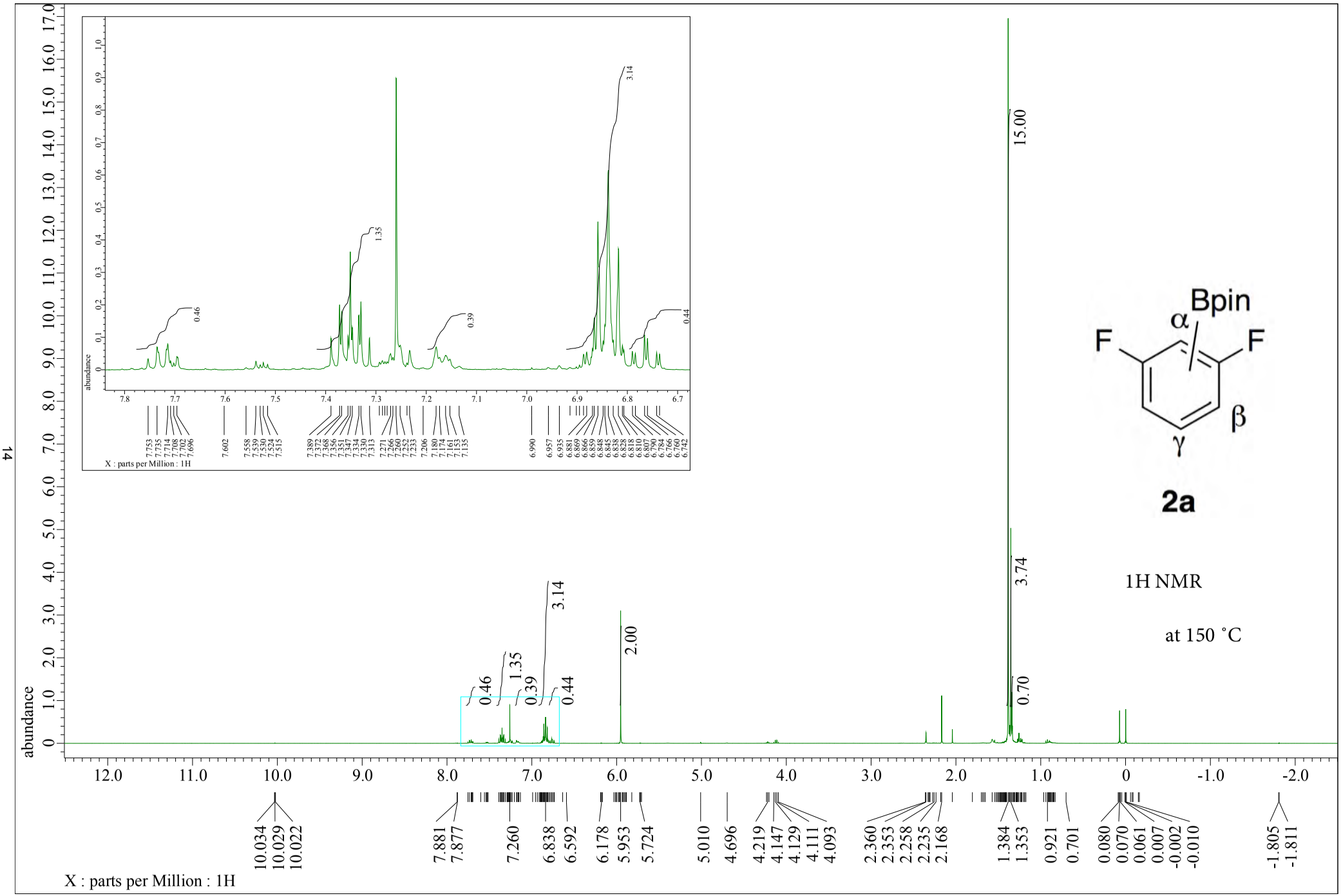
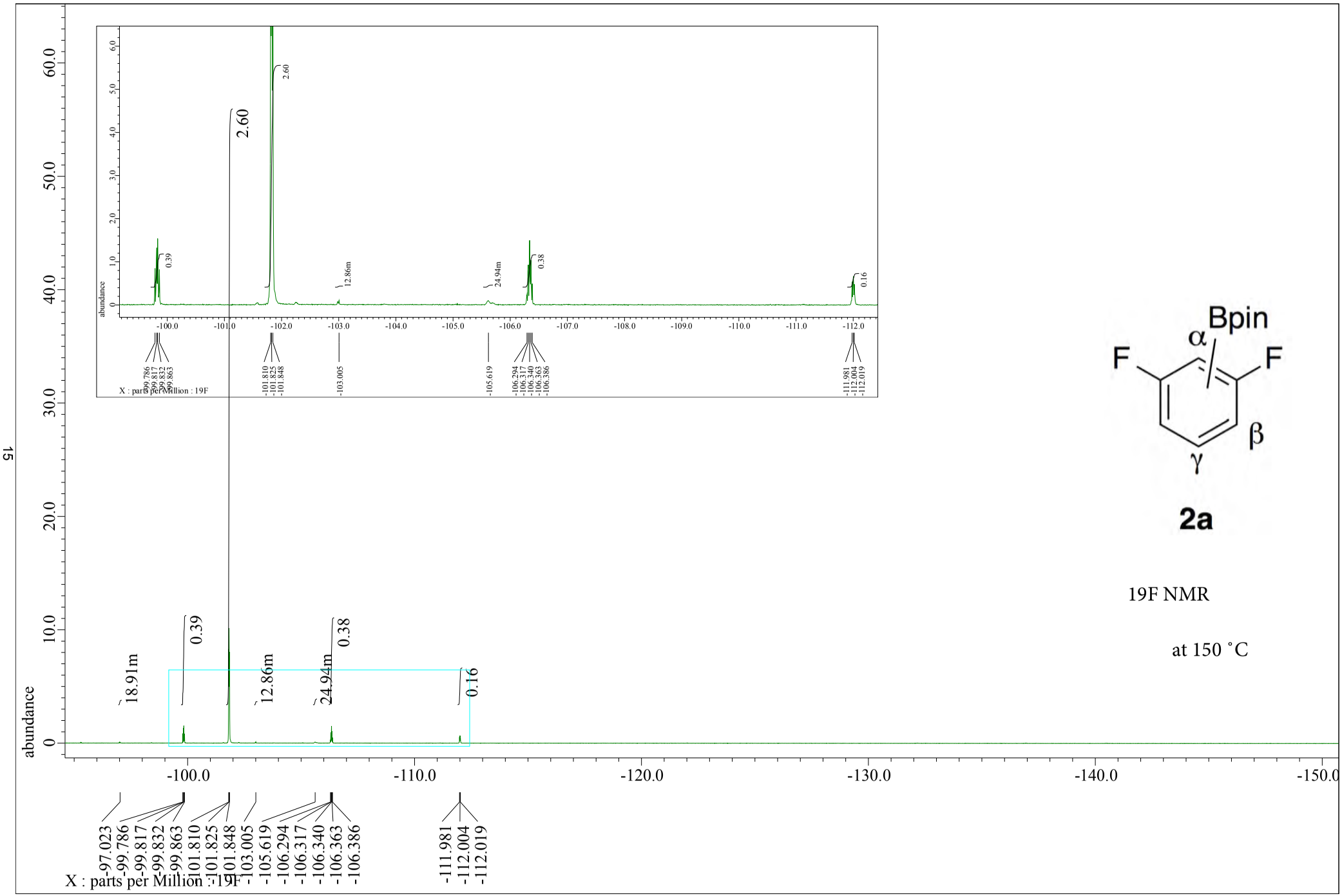
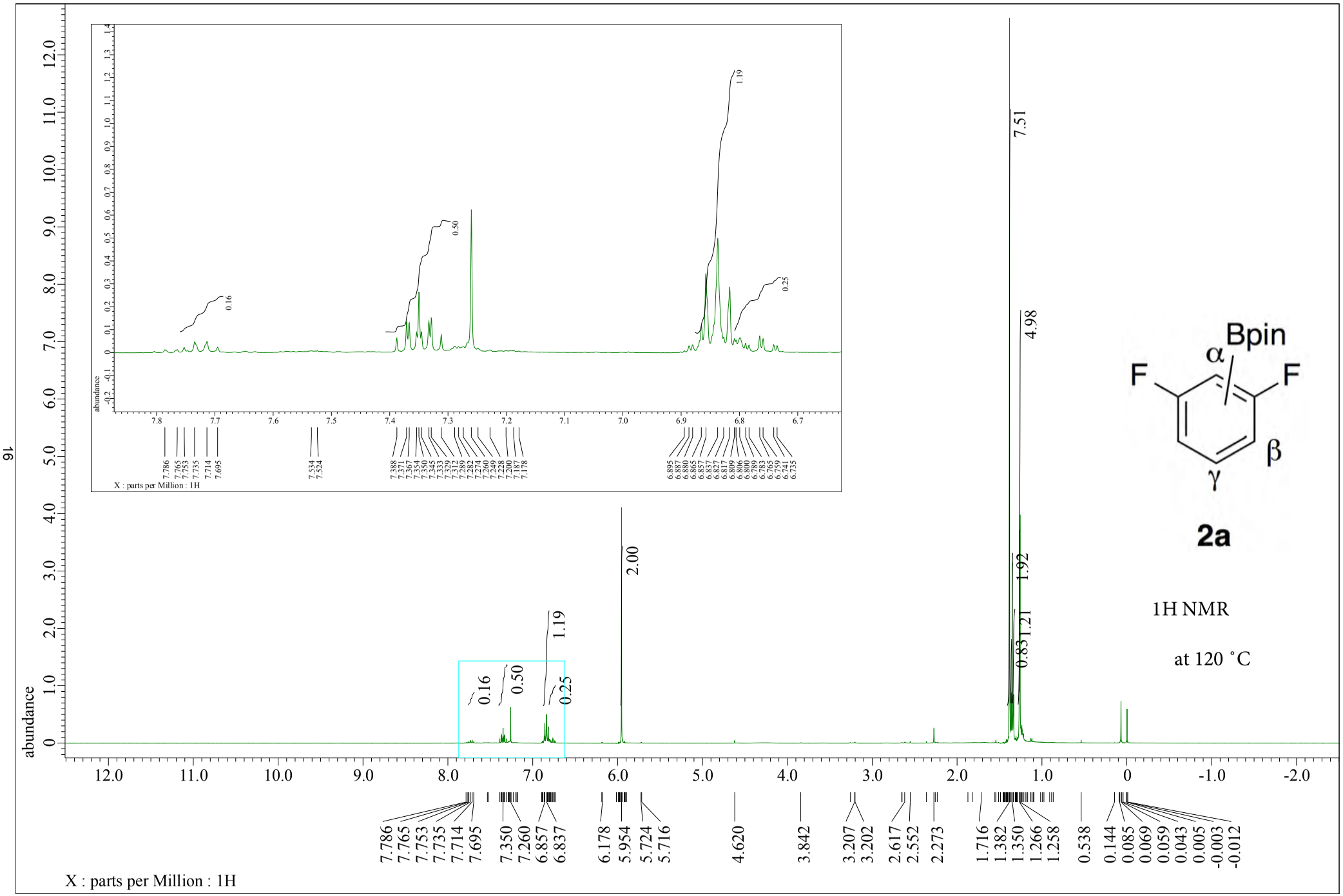
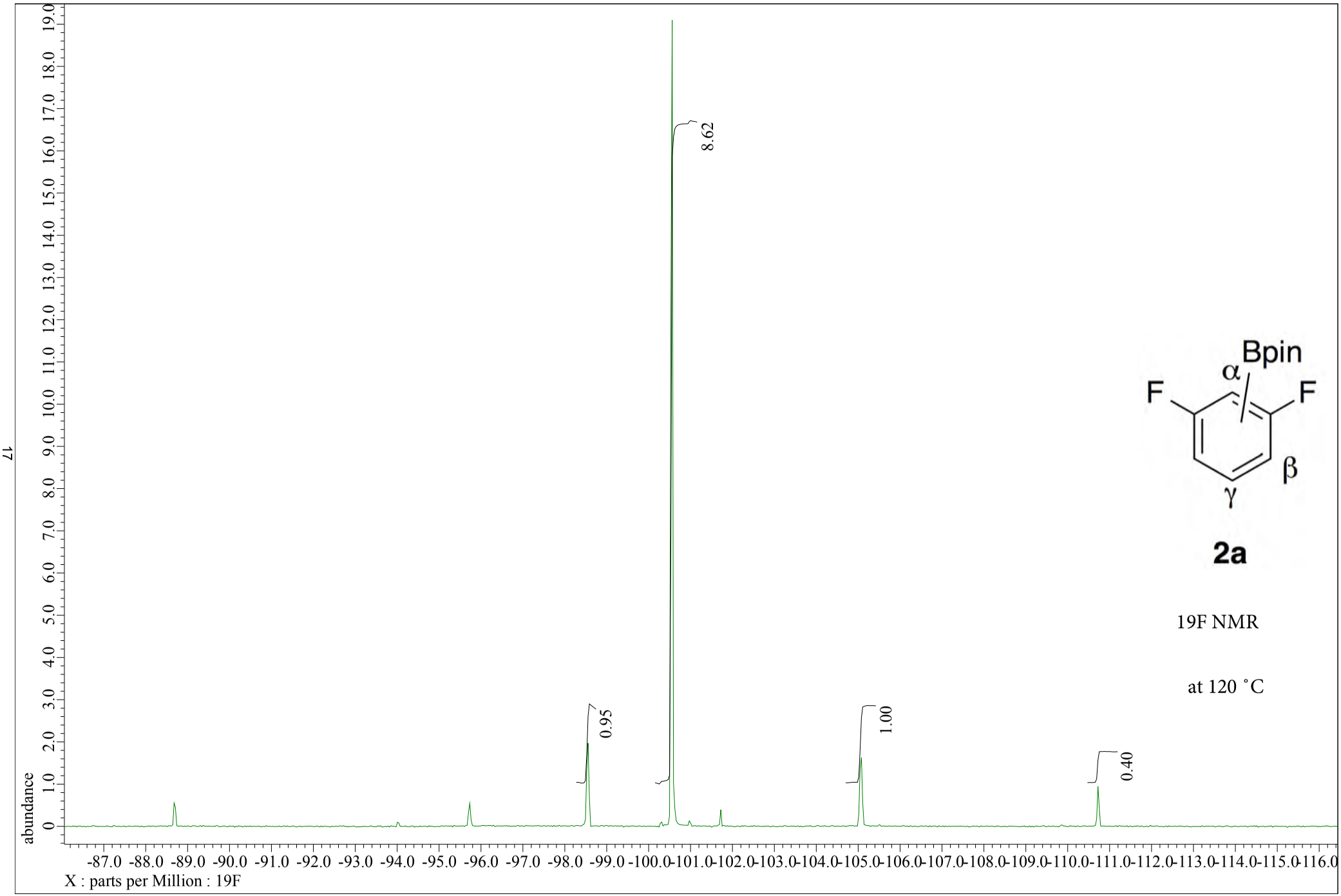


with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard


abundance
18.91m
0.39
2.60
12.86m
24.94m
0.38
0.16
-100.0
-110.0
-120.0
-130.0
-140.0
-150.0
X : parts per Million : 19F
-97.023
-99.786
-99.817
-99.832
-99.863
-101.810
-101.825
-101.848
-103.005
-105.619
-106.294
-106.317
-106.340
-106.363
-106.386
-111.981
-112.004
-112.019
Bpin
α
β
γ
F
F
2a
19F NMR
at 150 ˚C



with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard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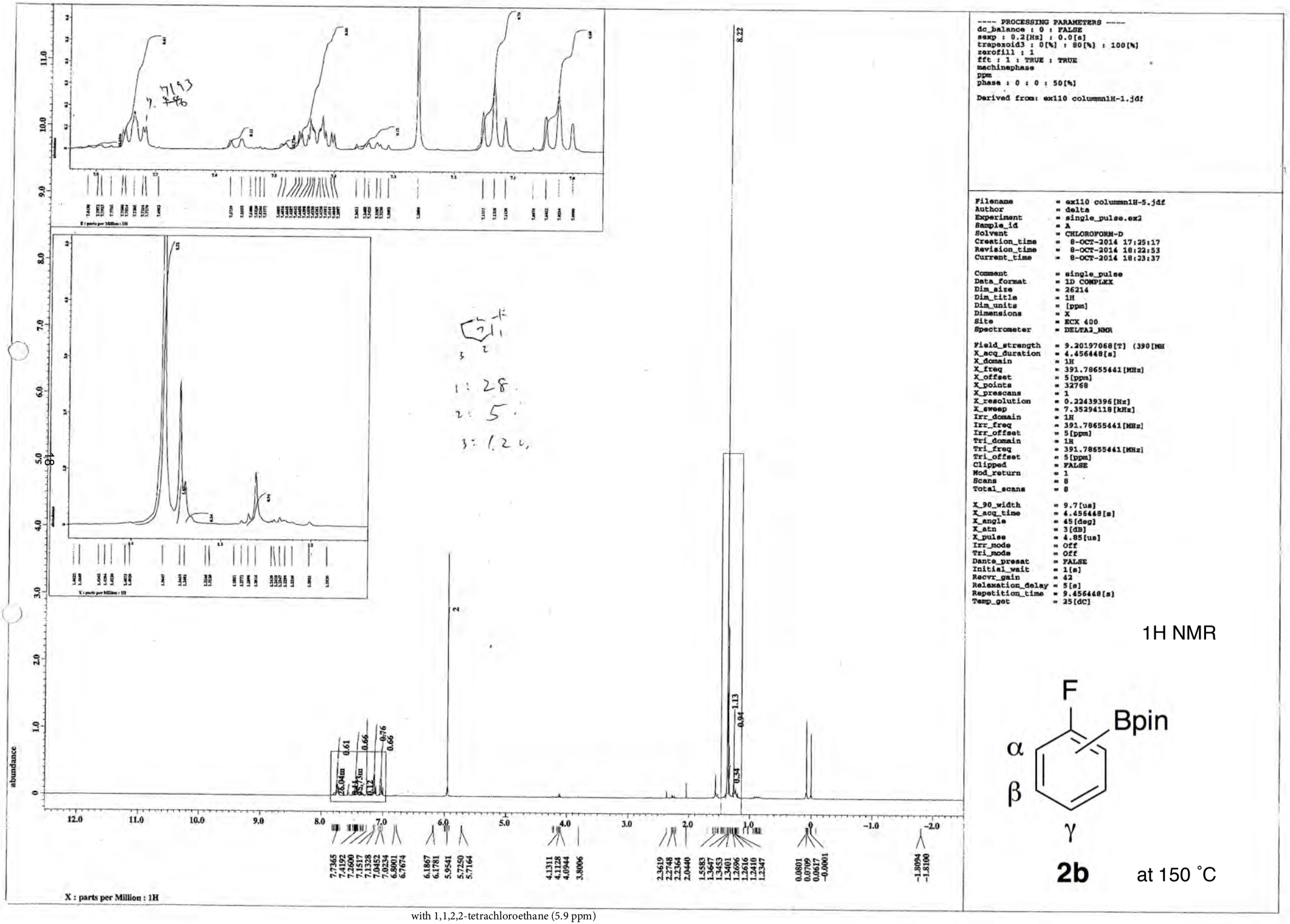
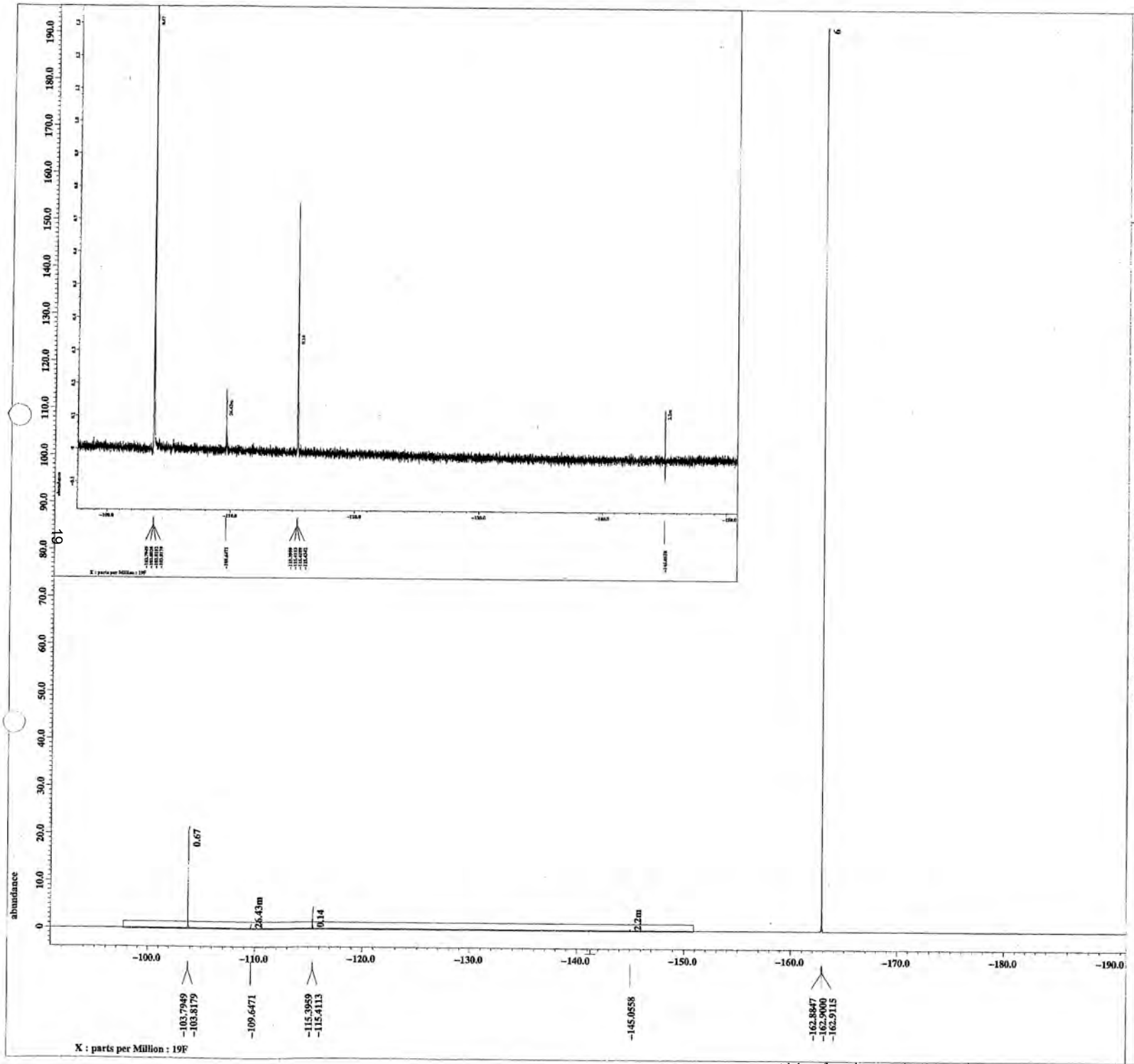
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
18.0
19.0
8.62
0.95
1.00
0.40
-87.0 -88.0 -89.0 -90.0 -91.0 -92.0 -93.0 -94.0 -95.0 -96.0 -97.0 -98.0 -99.0 -100.0 -101.0 -102.0 -103.0 -104.0 -105.0 -106.0 -107.0 -108.0 -109.0 -110.0 -111.0 -112.0 -113.0 -114.0 -115.0 -116.0
X : parts per Million : 19F
Bpin
α
F
F
β
γ
2a
19F NMR
at 120 ˚C



with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard




```
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sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: ex110 columnn 19F-1.jdf
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Revision_time     =  8-OCT-2014 18:17:00
Current_time      =  8-OCT-2014 18:17:09

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Dim_units         = [ppm]
Dimensions        = X
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X_offset          = -140[ppm]
X_points          = 32768
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X_sweep           = 46.2962963[kHz]
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Irr_freq          = 368.64763285[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 368.64763285[MHz]
Tri_offset        = 5[ppm]
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X_acq_time        = 0.7077888[s]
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X_atn             = 3.4[dB]
X_pulse           = 5.75[us]
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Dante_presat      = FALSE
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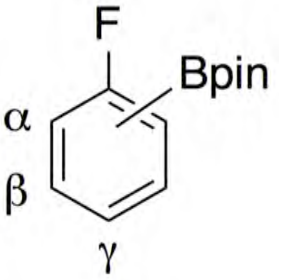
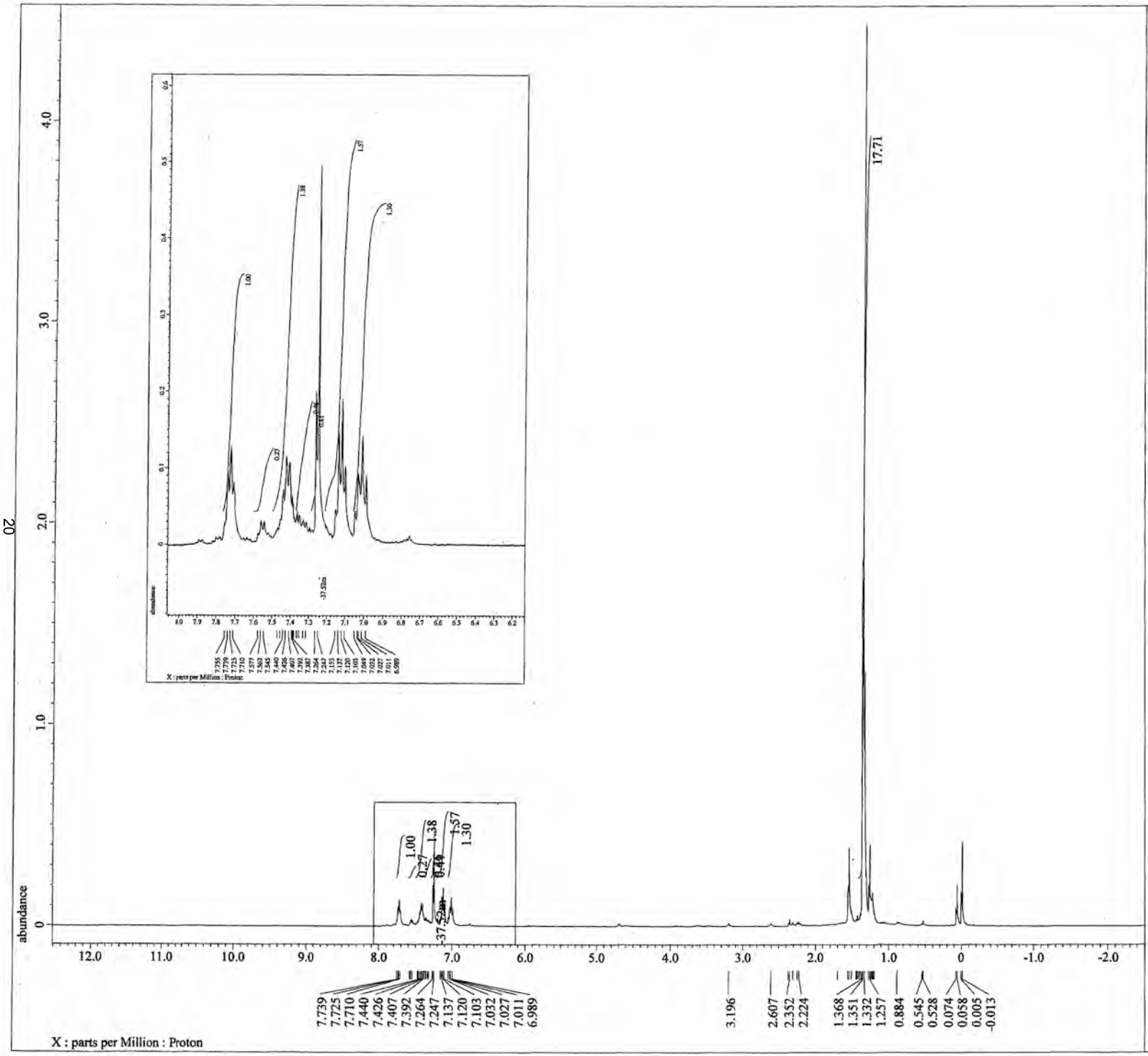
```

19F NMR



2b at 150 ˚C

with hexafluorobenzene (-162.9 ppm)
as an internal standard



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Spectrometer = DELTA2_NMR

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X_Points = 32768
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X_Sweep = 7.5030012[kHz]
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Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
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Total_Scans = 8

Relaxation_Delay = 5[s]
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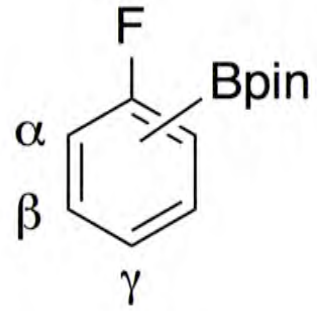
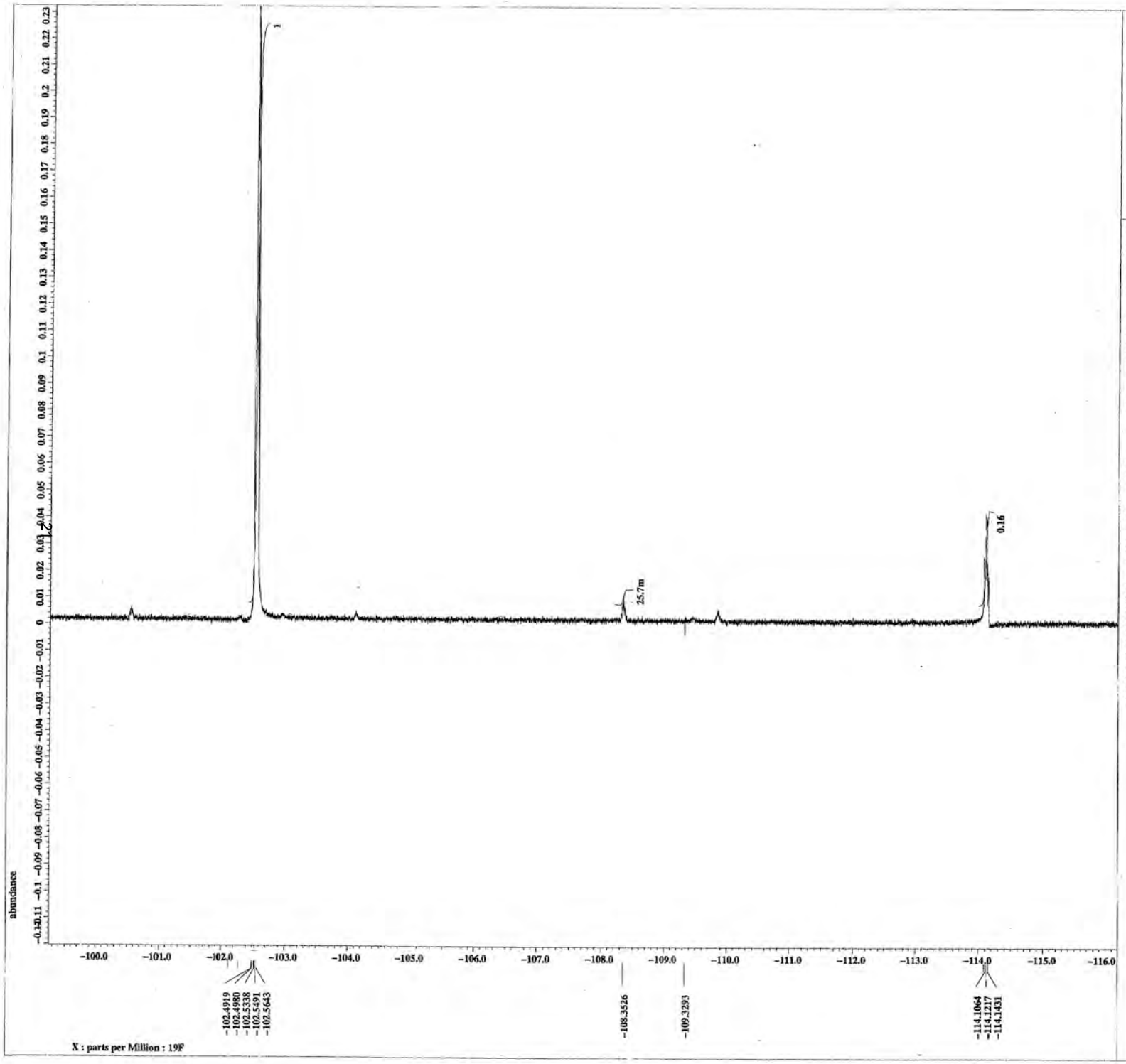
1H NMR



2b

at 120 ˚C

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fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: ex238 date 19F-1.jdf
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287

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Tri_domain        = 19F
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Clipped           = FALSE
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Total_scans       = 84

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X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
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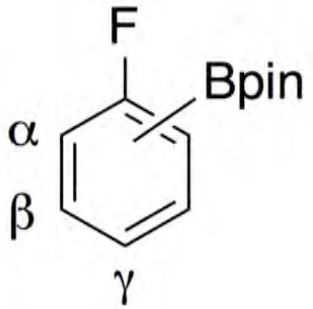
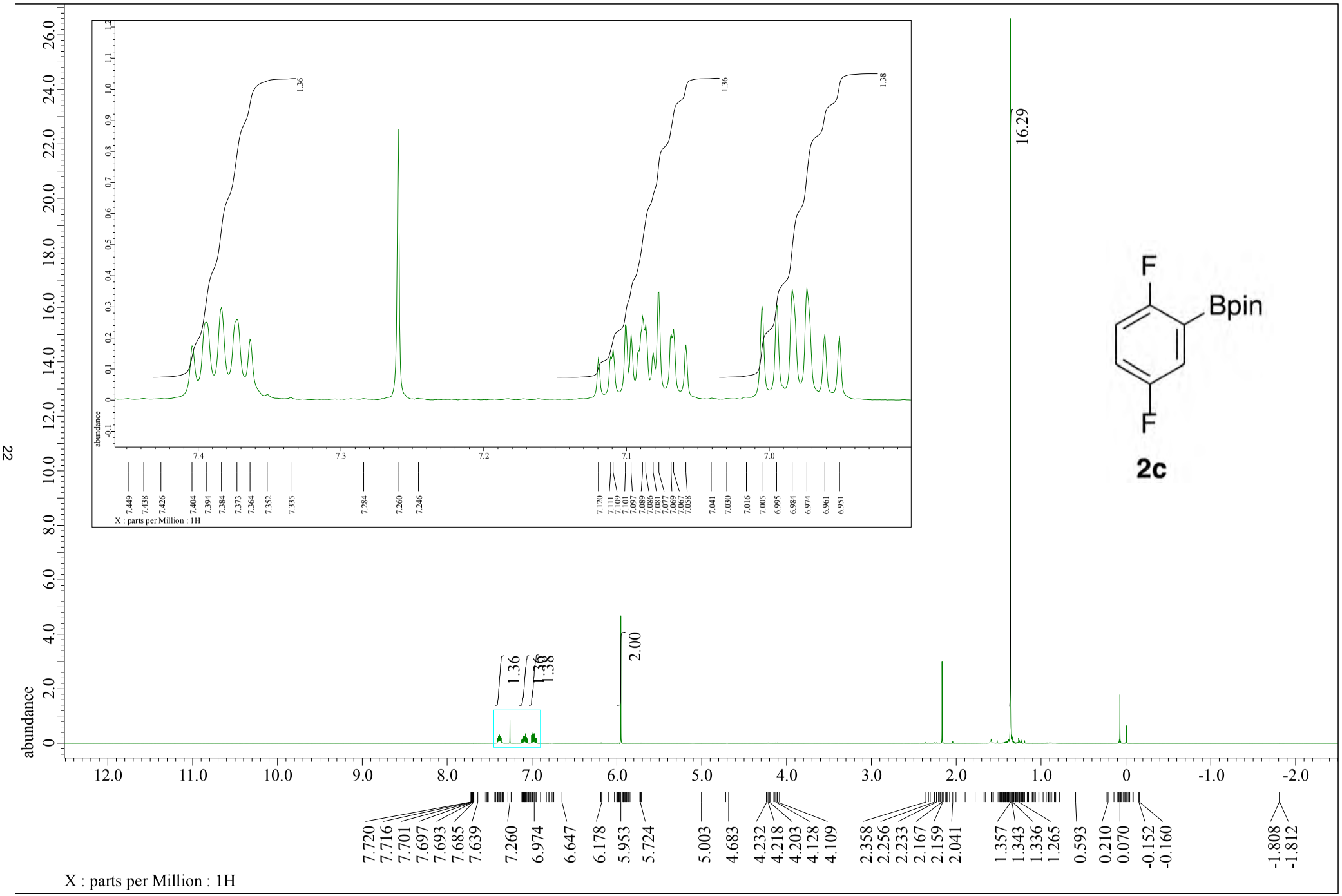
```

19F NMR

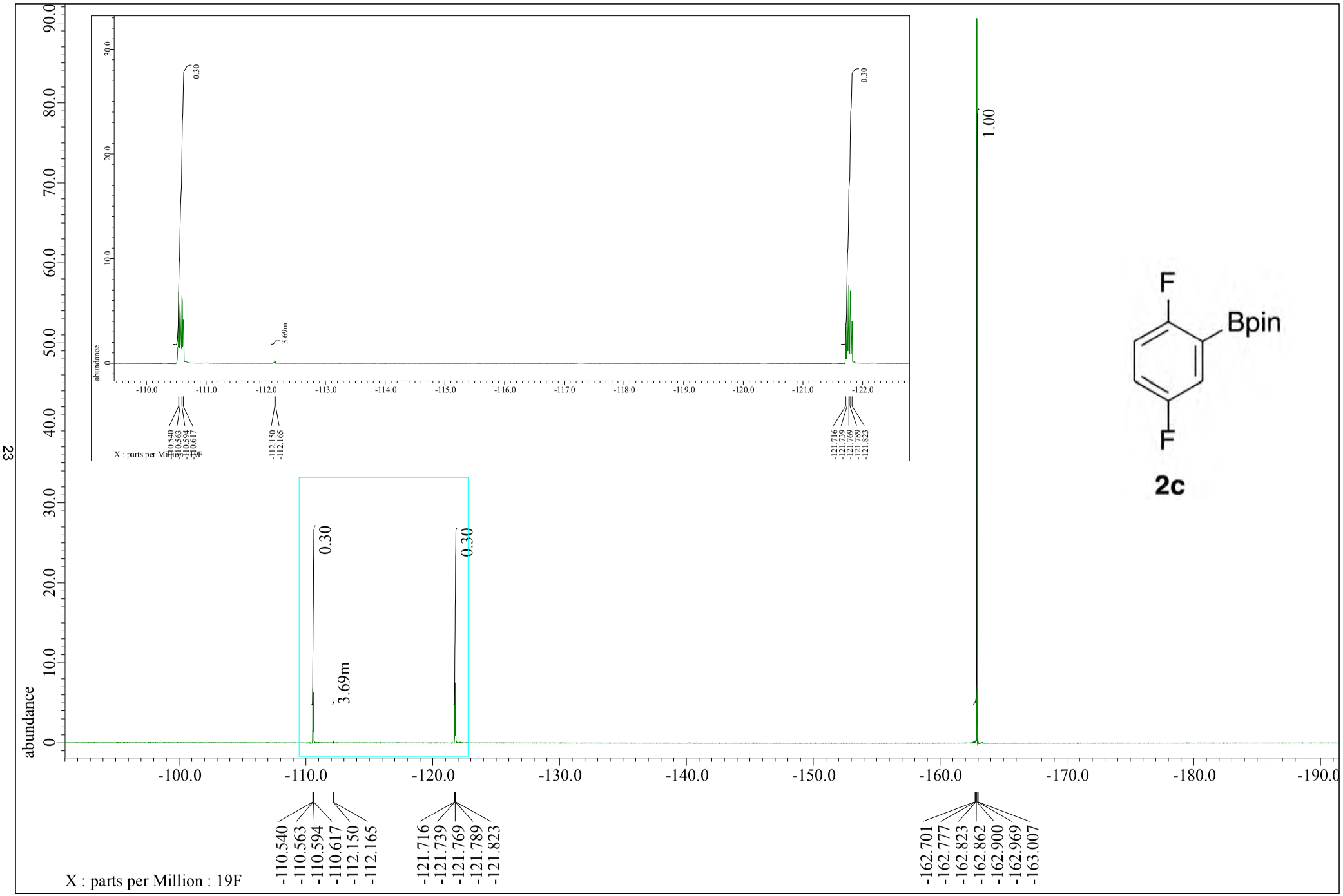


2b

at 120 ˚C

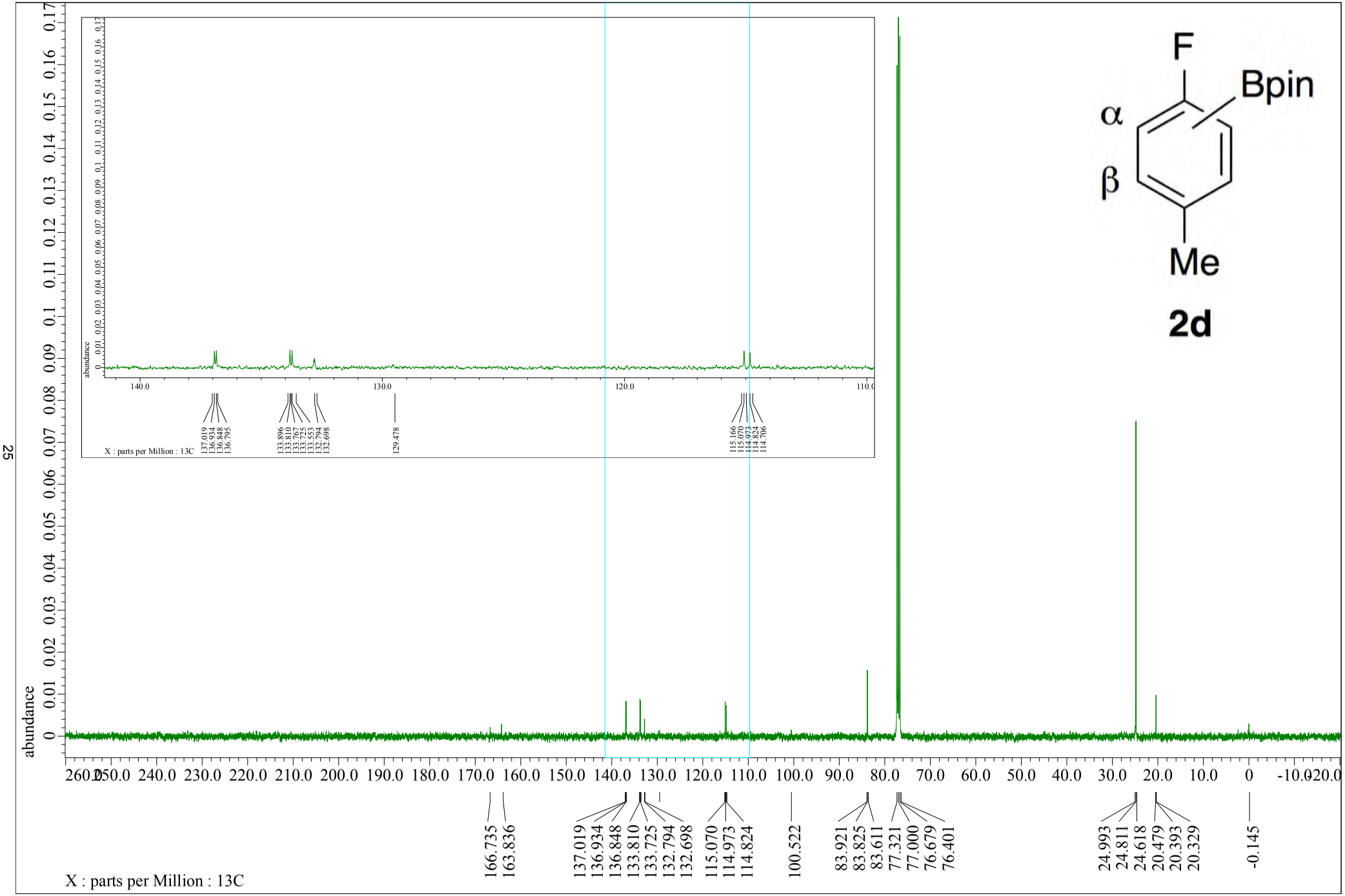


with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard



with hexafluorobenzene (-162.9 ppm)
as an internal

Bpin
F
Me
α
β
2d
11.49
3.07
0.55
X : parts per Million : 1H
abundance


F
Bpin
α
β
Me
2d
abundance
X : parts per Million : 13C
166.735
163.836
137.019
136.934
136.848
133.810
133.725
132.794
132.698
115.070
114.973
114.824
100.522
83.921
83.825
83.611
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76.679
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24.618
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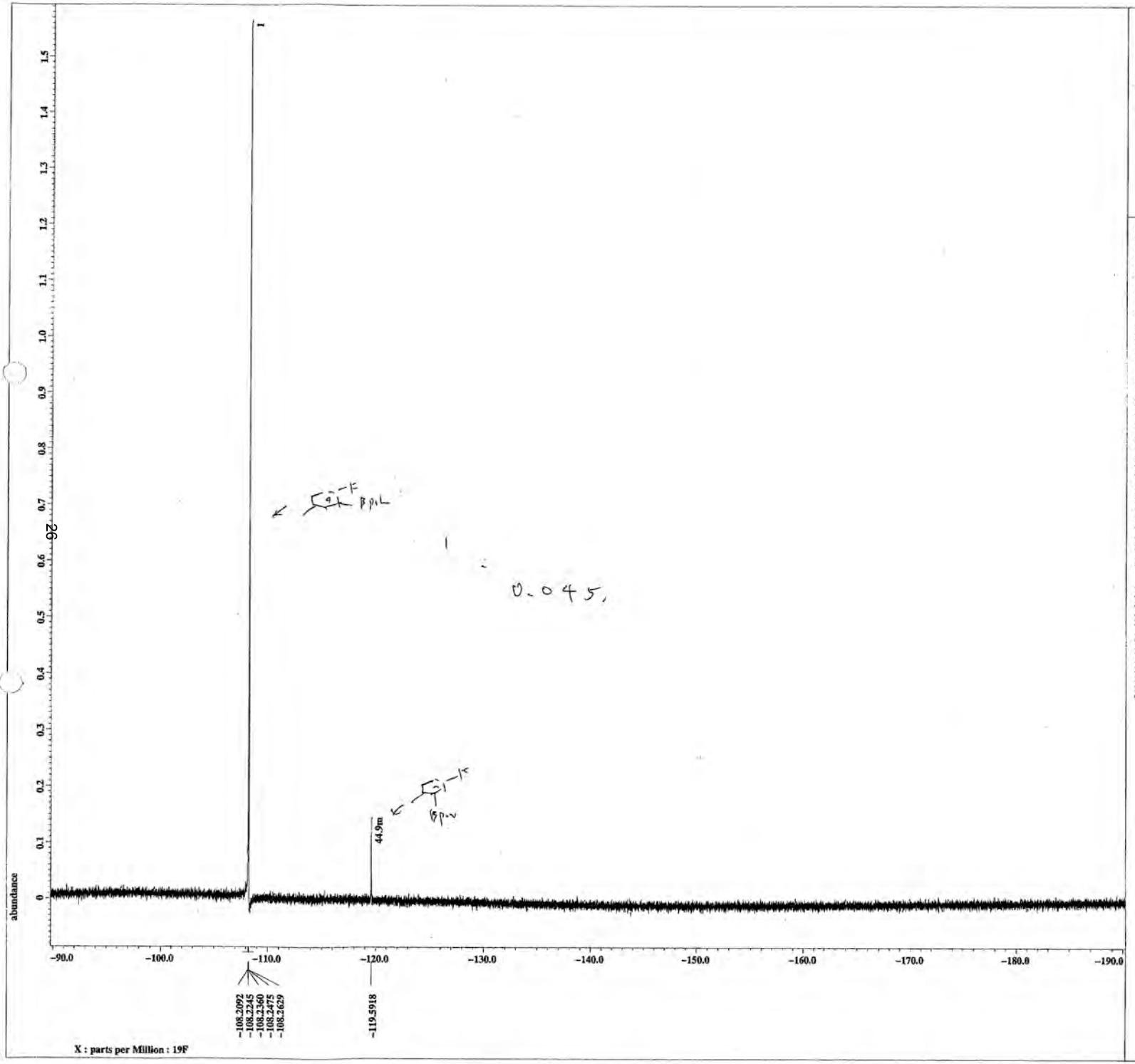
20.393
20.329
-0.145




```
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zerofill : 1
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machinephase
ppm
phase : 3.75 : 0 : 18.38331[%]

Derived from: ex166 crude 19F-14.jdf
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column

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X_points          = 12768
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X_resolution      = 1.41285084[Hz]
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Irr_freq          = 368.64763285[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 368.64763285[MHz]
Tri_offset        = 5[ppm]
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Mod_return        = 1
Scans             = 19
Total_scans       = 19

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Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 54
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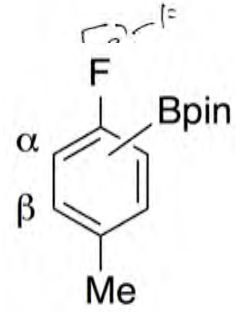
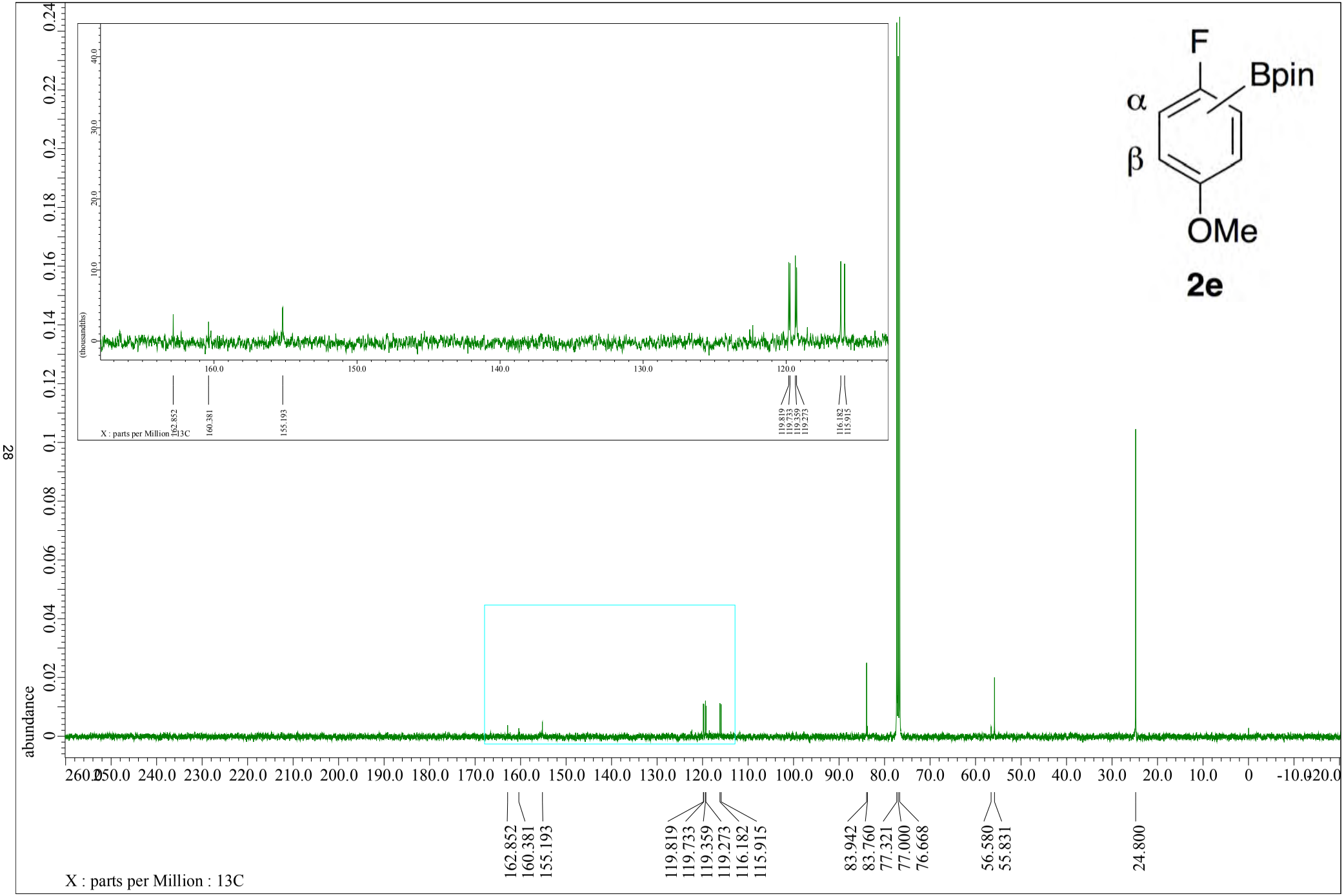
```

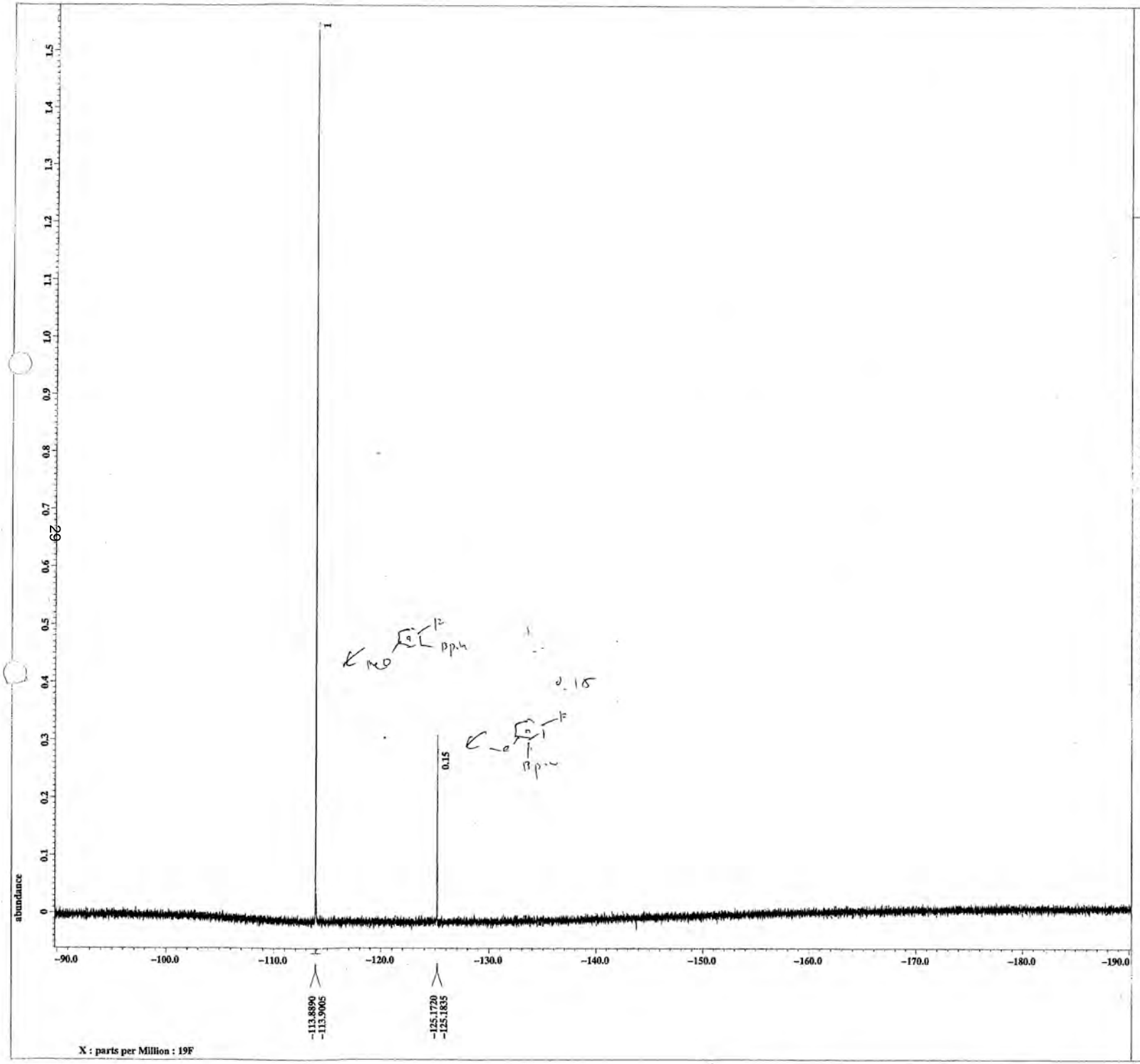
19F NMR



2d

F
Bpin
OMe
α
β
2e
14.83
3.80
7.520
7.256
6.952
6.949
6.945
6.935
6.931
5.294
3.829
3.798
3.787
3.777
3.766
3.204
3.200
2.616
2.609
2.360
2.040
1.562
1.356
1.347
0.876
0.535
0.066
-0.005
-0.157
1.97
0.17
0.14
0.15
1.00
X : parts per Million : 1H
abundance


F
Bpin
α
β
OMe
2e
abundance
X : parts per Million : 13C
162.852
160.381
155.193
119.819
119.733
119.359
119.273
116.182
115.915
83.942
83.760
77.321
77.000
76.668
56.580
55.831
24.800



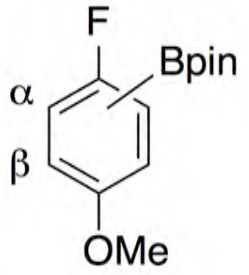

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: ex165 crude 19F-1.jdf
```

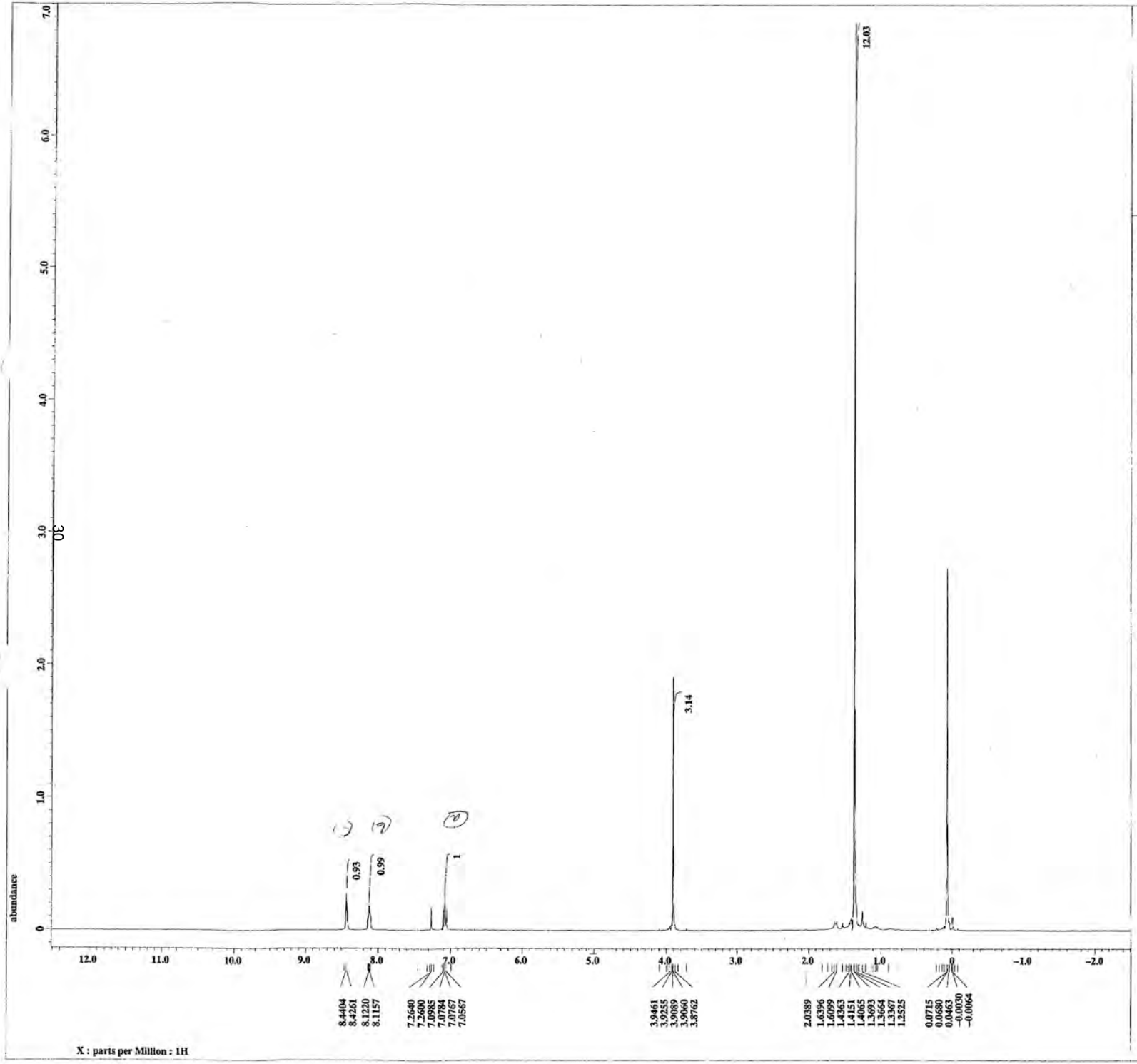
isolated

Filename	= ex165 crude 19F-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 15-JAN-2015 16:50:30
Revision_time	= 15-JAN-2015 17:56:50
Current_time	= 15-JAN-2015 17:57:02
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 19F
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 0.7077888[s]
X_domain	= 19F
X_freq	= 368.64763285[MHz]
X_offset	= -140[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 1.41285084[Hz]
X_sweep	= 46.2962963[kHz]
Irr_domain	= 19F
Irr_freq	= 368.64763285[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 19F
Tri_freq	= 368.64763285[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 31
Total_scans	= 31
X_90_width	= 11.5[us]
X_acq_time	= 0.7077888[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 56
Relaxation_delay	= 5[s]
Repetition_time	= 5.7077888[s]
Temp_get	= 22.1[dC]

19F NMR



2e

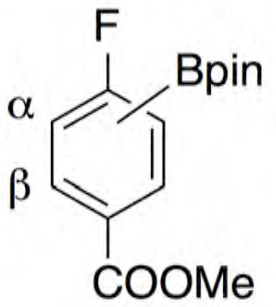



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]

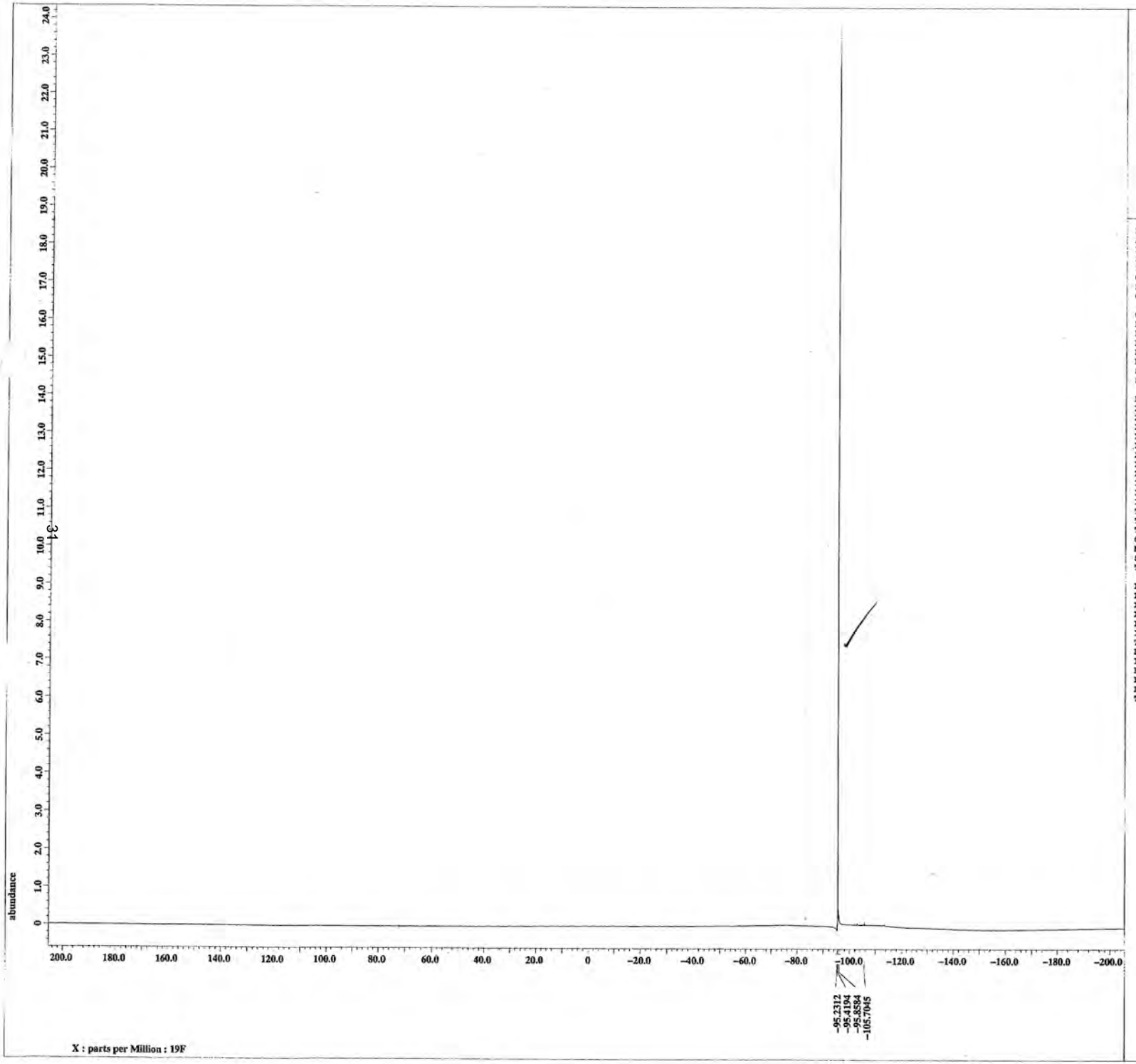
Derived from: ex247 date 1H-1.jdf
```

Filename	= ex247 date 1H-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 1-MAY-2015 16:48:23
Revision_time	= 1-MAY-2015 18:21:18
Current_time	= 1-MAY-2015 18:21:28
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.93[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.965[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 34
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 25[dC]

1H NMR



2f




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: ex247 date 19F-1.jdf
```

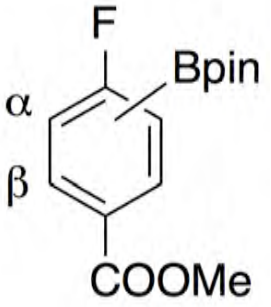
```
Filename          = ex247 date 19F-3.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = A
Solvent           = CHLOROFORM-D
Creation_time     =  1-MAY-2015 17:15:44
Revision_time     =  1-MAY-2015 18:28:02
Current_time      =  1-MAY-2015 18:28:16

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

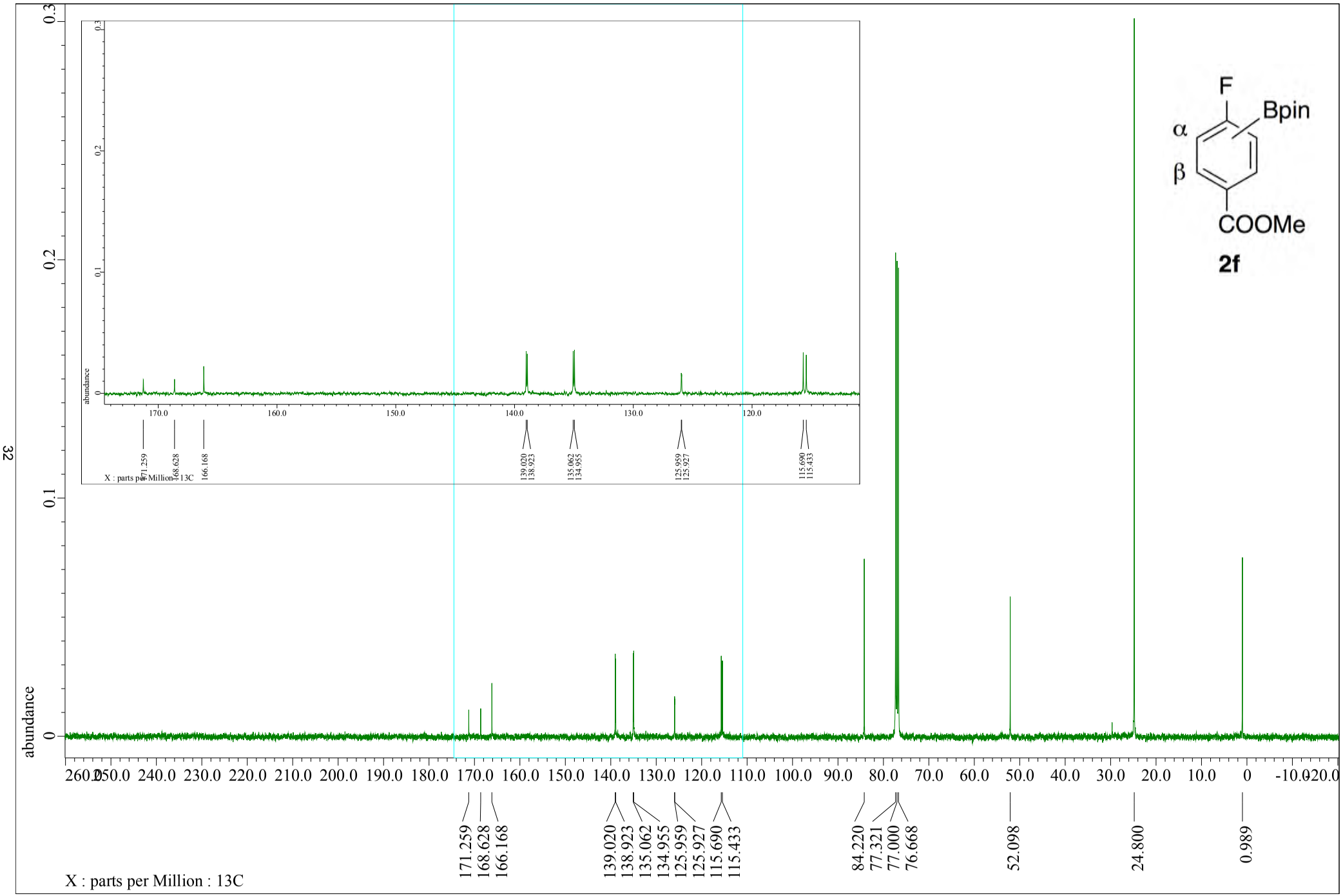
Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 86.50752[ms]
X_domain          = 19F
X_freq            = 368.64763285[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 11.55968868[Hz]
X_sweep           = 189.39393939[kHz]
Irr_domain        = 19F
Irr_freq          = 368.64763285[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 368.64763285[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 305
Total_scans       = 305

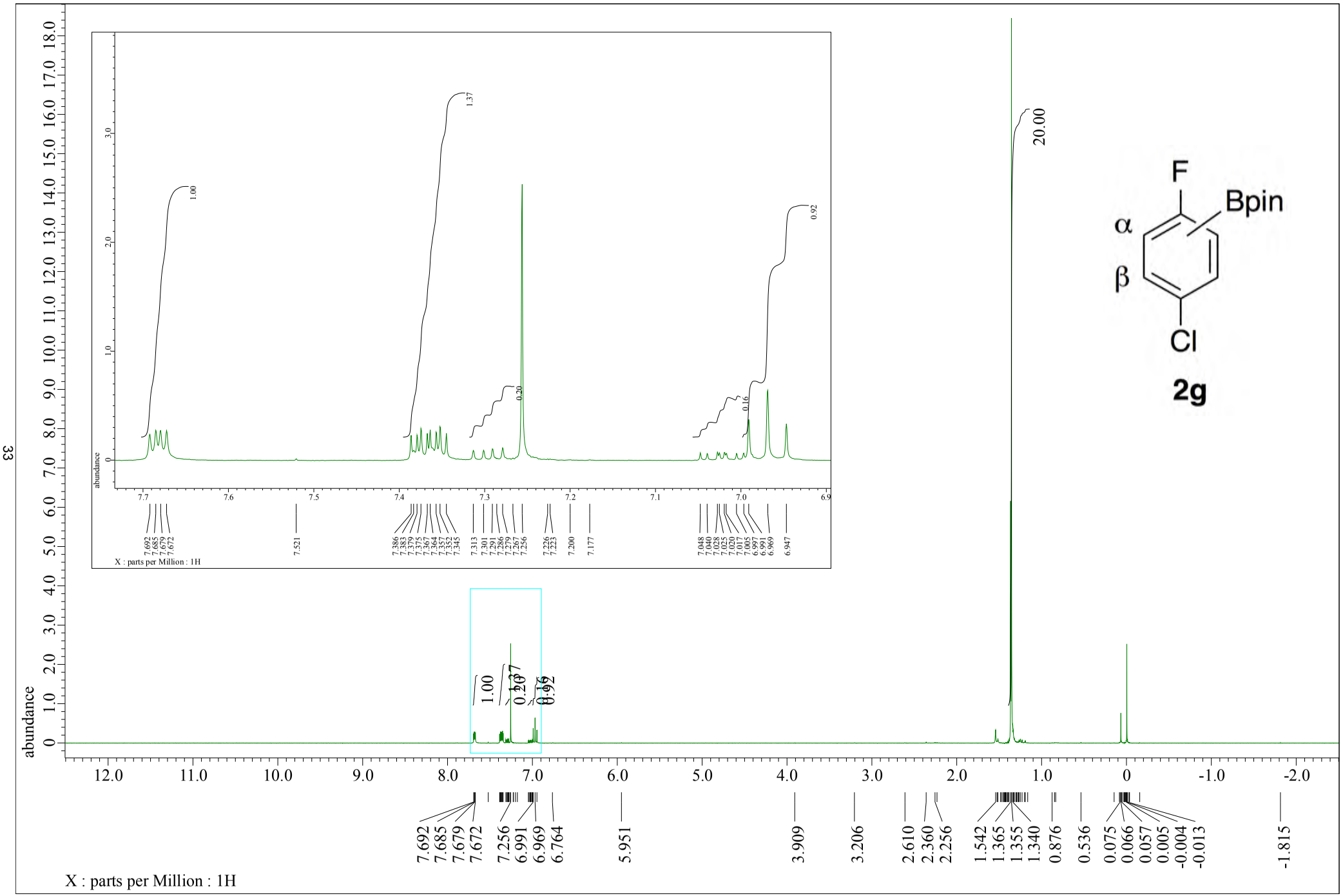
X_90_width        = 11.5[us]
X_acq_time        = 86.50752[ms]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 52
Relaxation_delay  = 5[s]
Repetition_time   = 5.08650752[s]
Temp_get          = 24.7[dC]
```

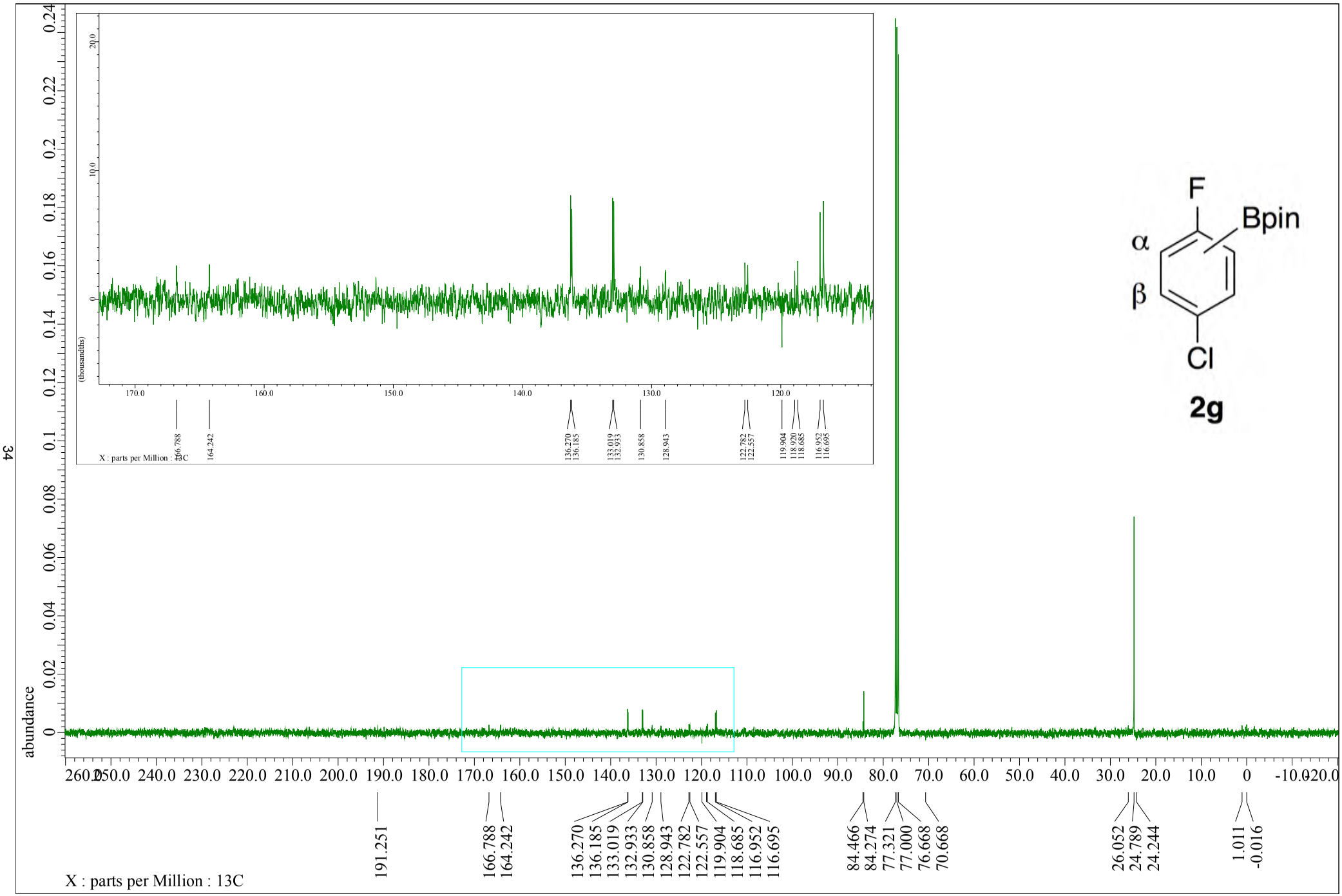
19F NMR

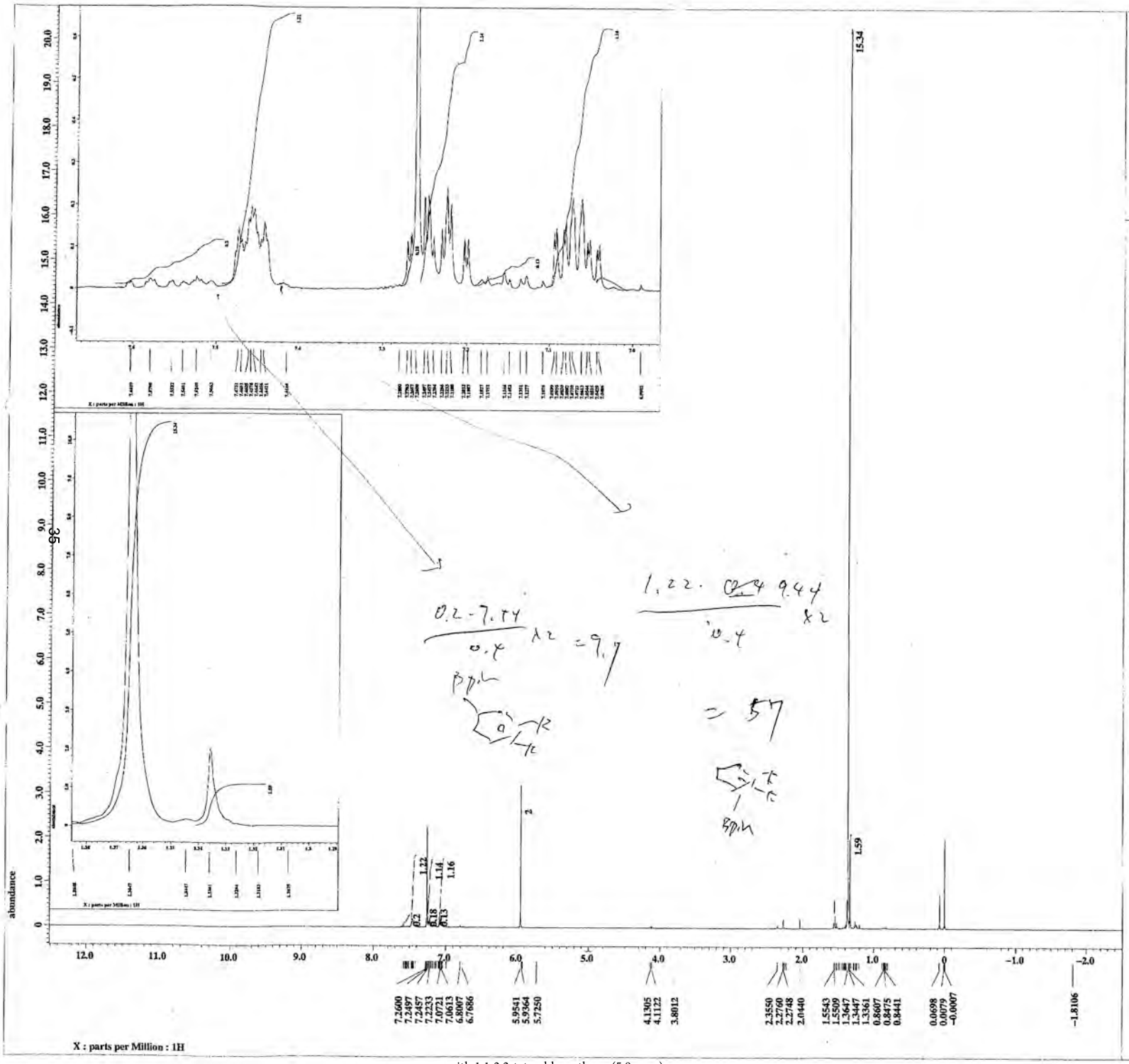


2f


F
Bpin
α
β
COOMe
2f
abundance
X : parts per Million : 13C
171.259
168.628
166.168
139.020
138.923
135.062
134.955
125.959
125.927
115.690
115.433
84.220
77.321
77.000
76.668
52.098
24.800
0.989


F
Bpin
α
β
Cl
2g
20.00
1.00
1.37
0.20
0.16
0.92
abundance
X : parts per Million : 1H
7.692
7.685
7.679
7.672
7.521
7.386
7.383
7.379
7.375
7.367
7.364
7.357
7.345
7.313
7.301
7.291
7.286
7.279
7.267
7.256
7.226
7.223
7.200
7.177
7.048
7.040
7.028
7.025
7.020
7.017
7.005
6.997
6.991
6.969
6.947
5.951
3.909
3.206
2.610
2.360
2.256
1.542
1.365
1.355
1.340
0.876
0.536
0.075
0.066
0.057
0.005
-0.004
-0.013
-1.815
6.764


F
Bpin
α
β
Cl
2g
abundance
X : parts per Million : 13C
(thousandths)
191.251
166.788
164.242
136.270
136.185
133.019
132.933
130.858
128.943
122.782
122.557
119.904
118.685
116.952
116.695
84.466
84.274
77.321
77.000
76.668
70.668
26.052
24.789
24.244
1.011
-0.016



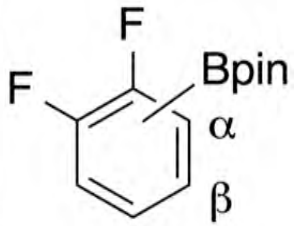

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]

Derived from: ex111 crude 1H-4.jdf
```

column

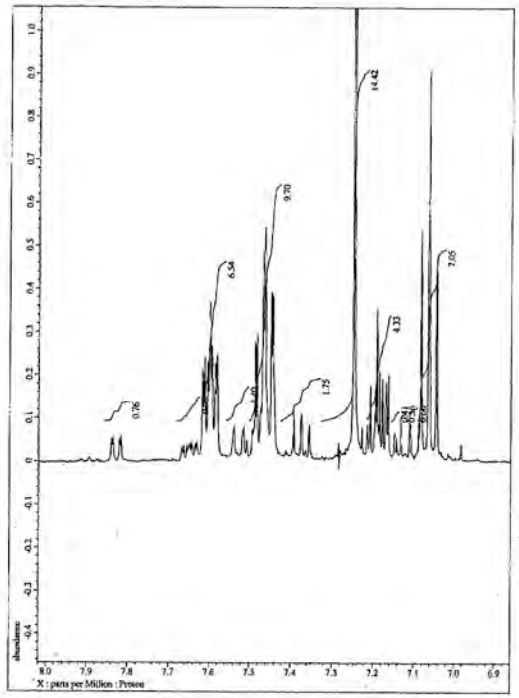
Filename	= ex111 crude 1H-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 10-OCT-2014 12:39:54
Revision_time	= 10-OCT-2014 13:37:46
Current_time	= 10-OCT-2014 13:38:30
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.7[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.85[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 24.8[dC]

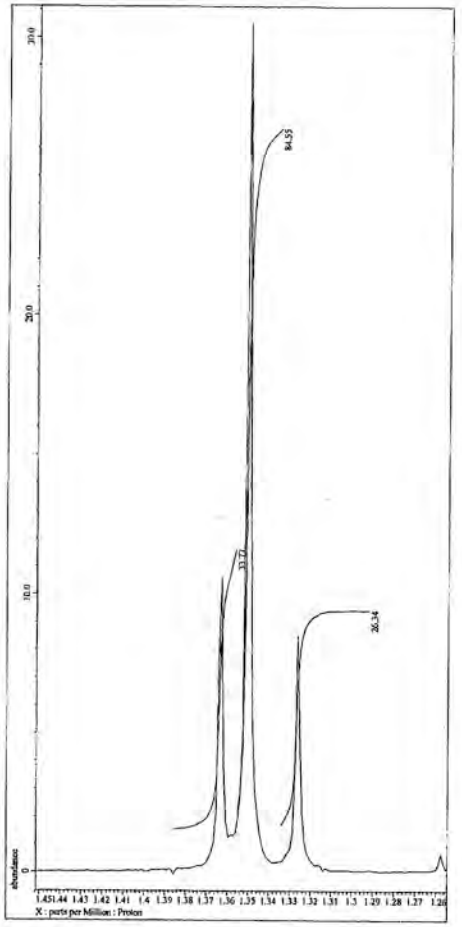
1H NMR



2h

with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard





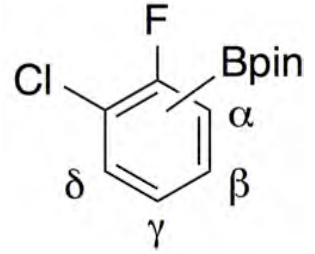

```
Filename          = ex276 columnn_Proton-1-3.j
Author            = iwasawa
Experiment        = proton.jxp
Sample_Id         = ex276 columnn
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JUL-2015 16:21:43
Revision_Time     = 15-JUL-2015 16:22:04
Current_Time      = 15-JUL-2015 16:22:41

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    = 9.389766[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      = 0.45796685[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

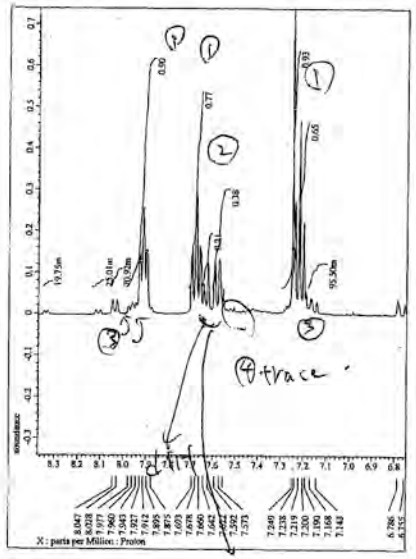
Relaxation_Delay  = 3[s]
Recvr_Gain        = 44
Temp_Get          = 26.5[dC]
X_90_Width        = 6.15[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.075[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 5.18365952[s]
```

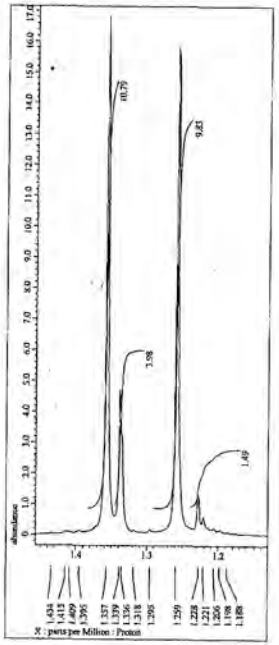
1H NMR



2i

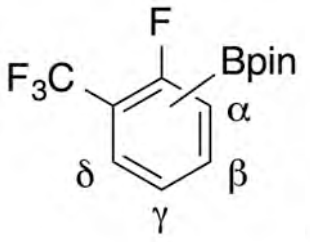
Bpin
α
β
F
γ
Cl
δ
2i
abundance
0
1.0
2.0
3.0
4.0
5.0
X : parts per Million : Fluorine19
-89.0
-91.0
-93.0
-95.0
-97.0
-99.0
-101.0
-103.0
-105.0
-107.0
-109.0
-111.0
-113.0
-115.0
-117.0
-119.0
-121.0
-123.0
-125.0
-127.0
-129.0
-100.825
-102.546
-104.482
-105.096
-106.848
-111.088
-114.407
-116.927
1.00
0.12
0.33
97.10m





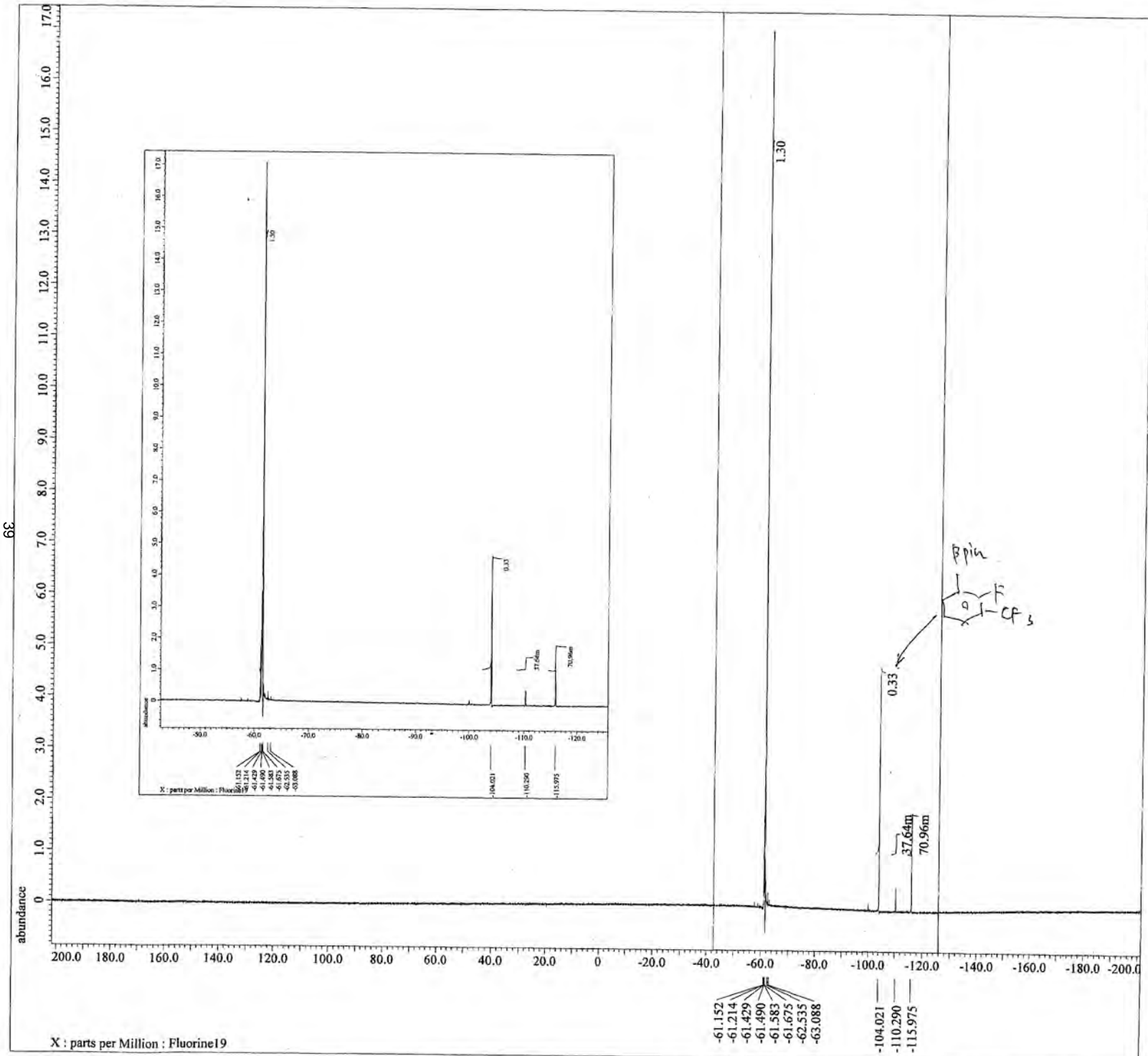
Filename	= ex280_Proton-1-3.jdf
Author	= iwasawa
Experiment	= proton.jxp
Sample_Id	= ex280
Solvent	= CHLOROFORM-D
Creation_Time	= 15-JUL-2015 15:30:45
Revision_Time	= 15-JUL-2015 15:34:43
Current_Time	= 15-JUL-2015 15:35:50
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[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	= 3[s]
Recvr_Gain	= 34
Temp_Get	= 26.6[dC]
X_90_Width	= 6.15[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 3.075[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 5.18365952[s]

1H NMR



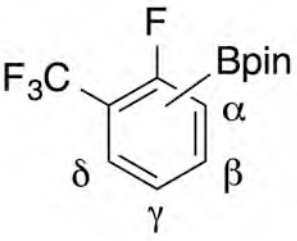
2j

with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard

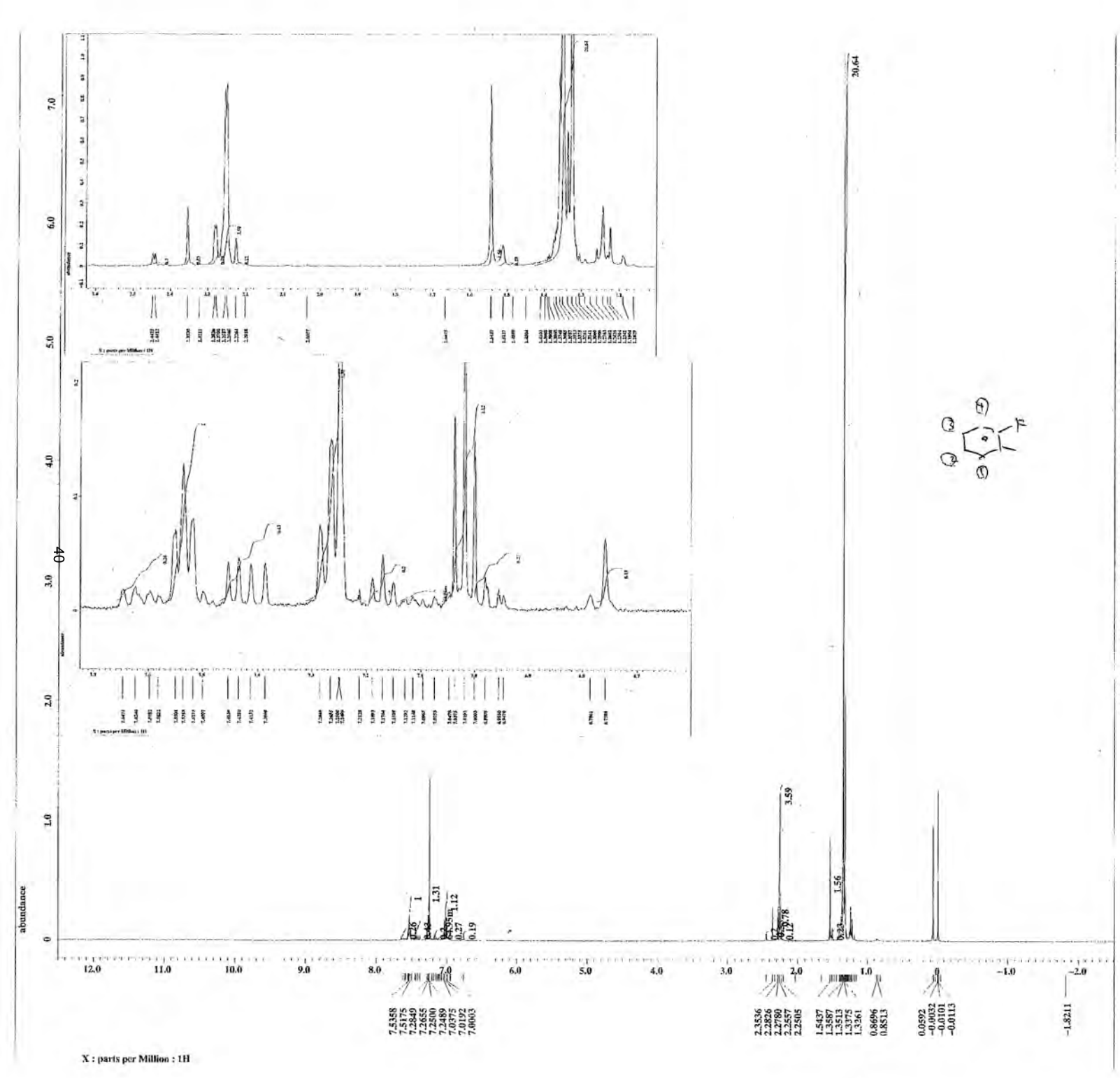


Filename	= ex280_Proton-1-5.jdf
Author	= iwasawa
Experiment	= proton.jxp
Sample_Id	= ex280
Solvent	= CHLOROFORM-D
Creation_Time	= 15-JUL-2015 15:34:06
Revision_Time	= 15-JUL-2015 15:36:41
Current_Time	= 15-JUL-2015 15:37:00
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Fluorine19
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 86.50752[ms]
X_Domain	= 19F
X_Freq	= 376.17105393[MHz]
X_Offset	= 0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 11.55958868[Hz]
X_Sweep	= 189.39393939[kHz]
X_Sweep_Clipped	= 151.51515152[kHz]
Irr_Domain	= Fluorine19
Irr_Freq	= 376.17105393[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Fluorine19
Tri_Freq	= 376.17105393[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 26.6[dC]
X_90_Width	= 7.12[us]
X_Acq_Time	= 86.50752[ms]
X_Angle	= 45[deg]
X_Atn	= 2.3[dB]
X_Pulse	= 3.56[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 5.08650752[s]

19F NMR



2j

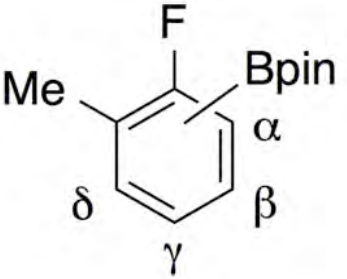



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]

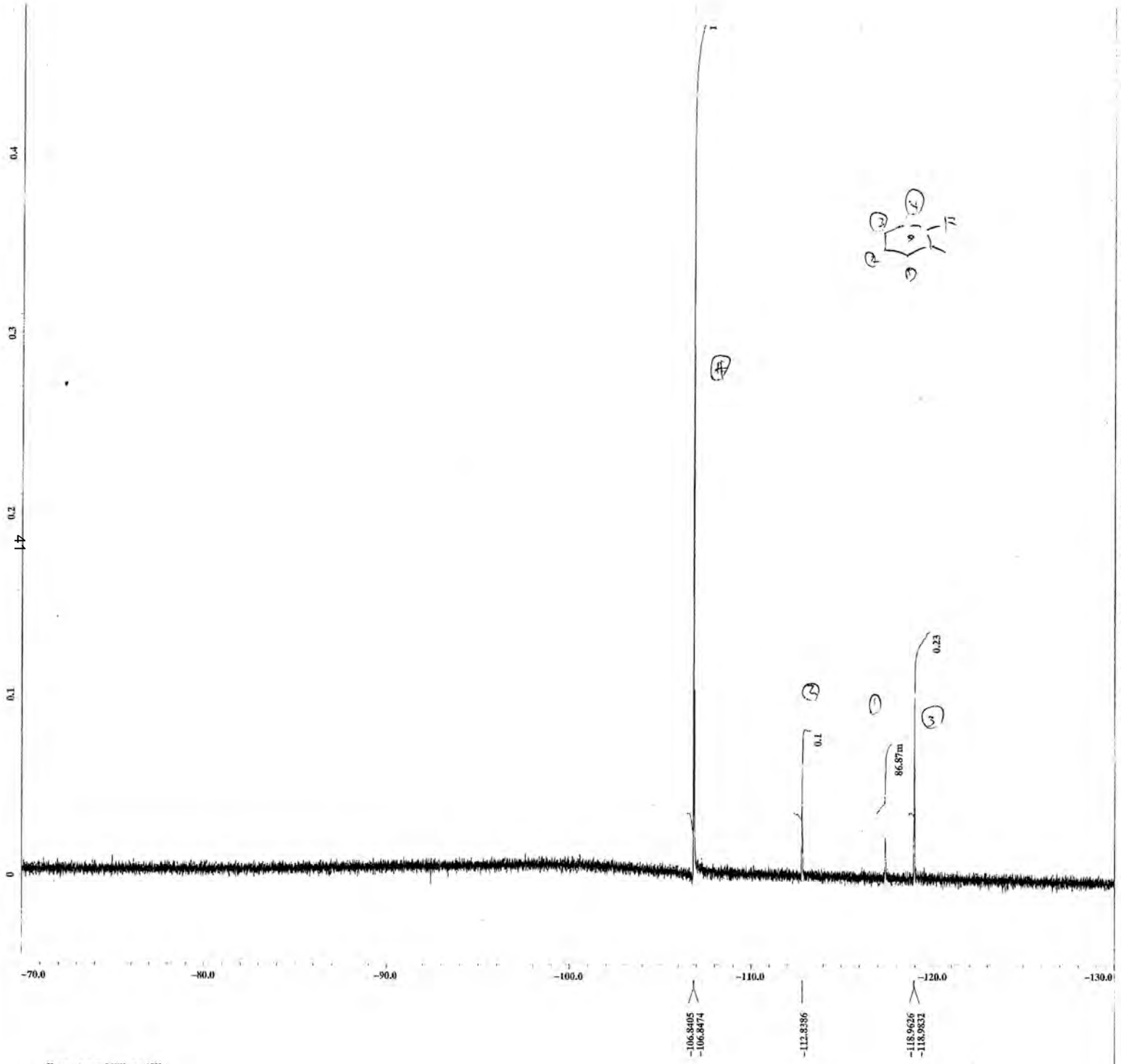
Derived from: ex277crude 1H-1.jdf
```

Filename	= ex277crude 1H-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 14-JUL-2015 13:04:49
Revision_time	= 14-JUL-2015 14:19:52
Current_time	= 14-JUL-2015 14:20:37
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.93[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.965[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 23[dC]

1H NMR



2k

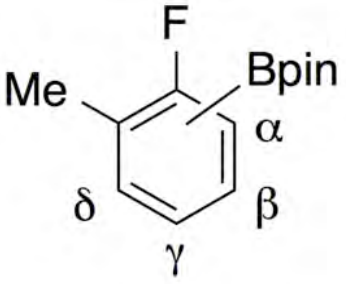



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : -15 : 0 : 50[%]

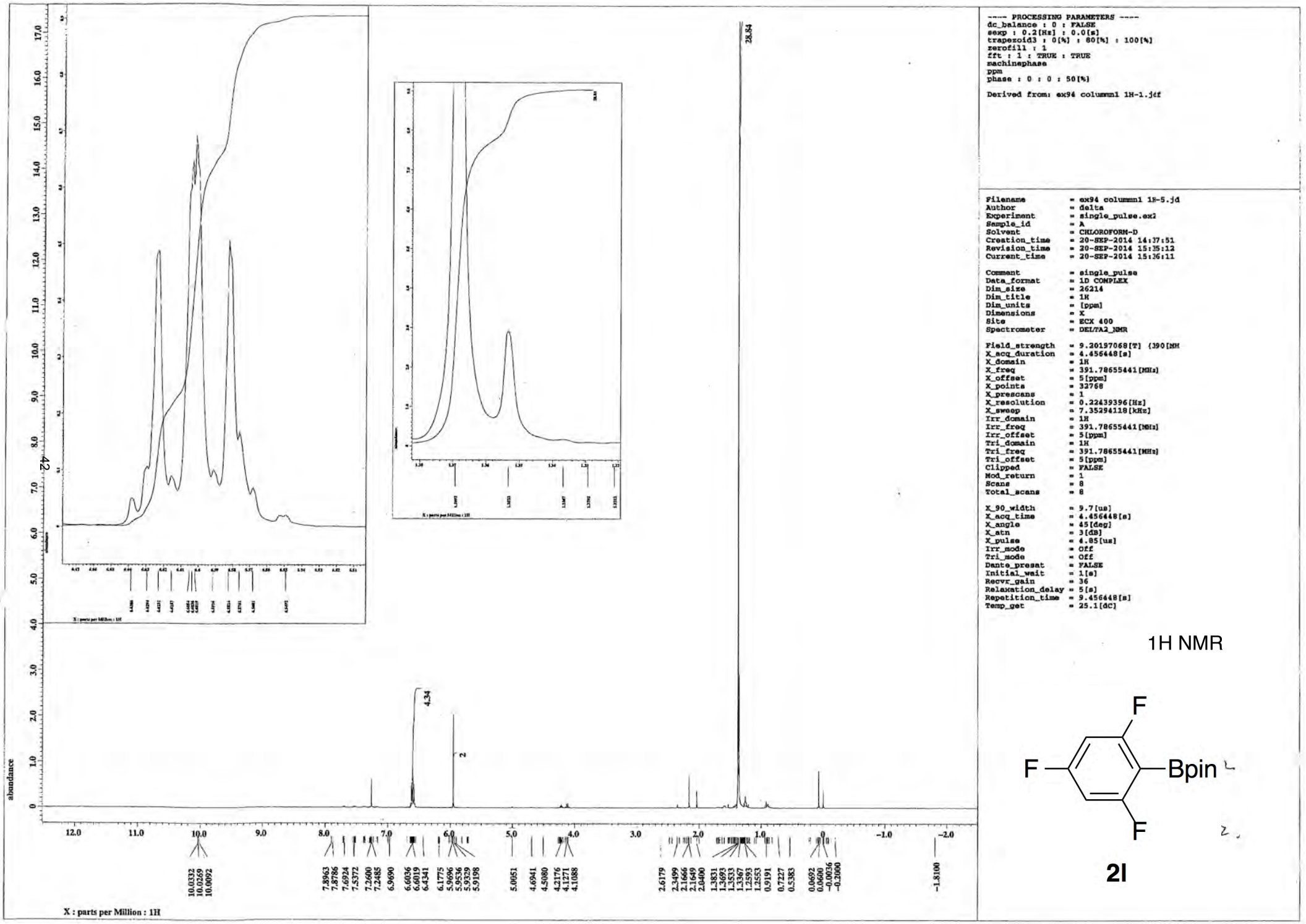
Derived from: ex277 crude 19F-1.jdf
```

Filename	= ex277 crude 19F-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 14-JUL-2015 13:11:04
Revision_time	= 14-JUL-2015 14:40:00
Current_time	= 14-JUL-2015 14:40:05
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 19F
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.18489088[s]
X_domain	= 19F
X_freq	= 368.64763285[MHz]
X_offset	= -100.0[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.84395957[Hz]
X_sweep	= 27.65486726[kHz]
Irr_domain	= 19F
Irr_freq	= 368.64763285[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 19F
Tri_freq	= 368.64763285[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 36
Total_scans	= 36
X_90_width	= 11.5[us]
X_acq_time	= 1.18489088[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 52
Relaxation_delay	= 5[s]
Repetition_time	= 6.18489088[s]
Temp_get	= 23[dC]

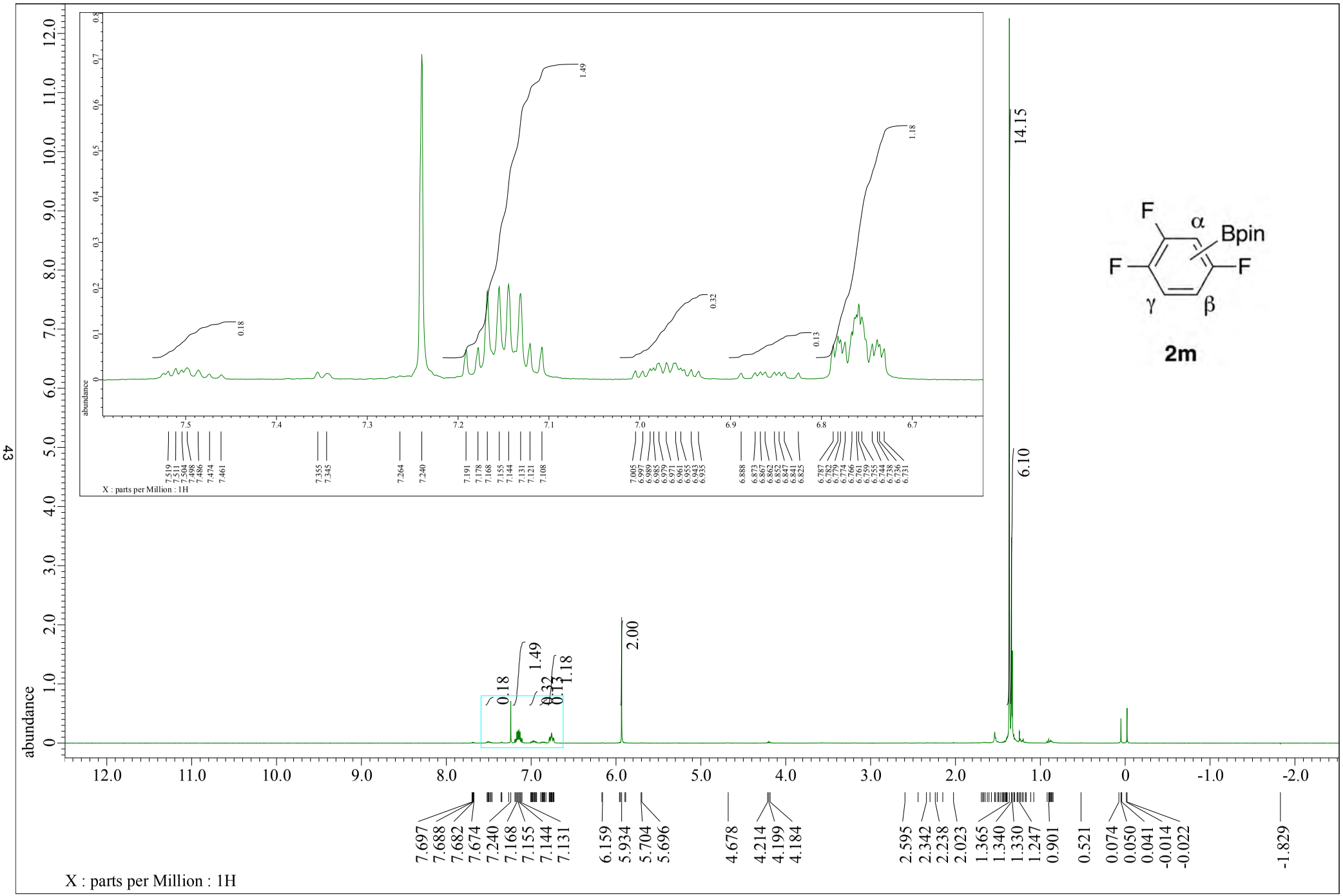
19F NMR



2k

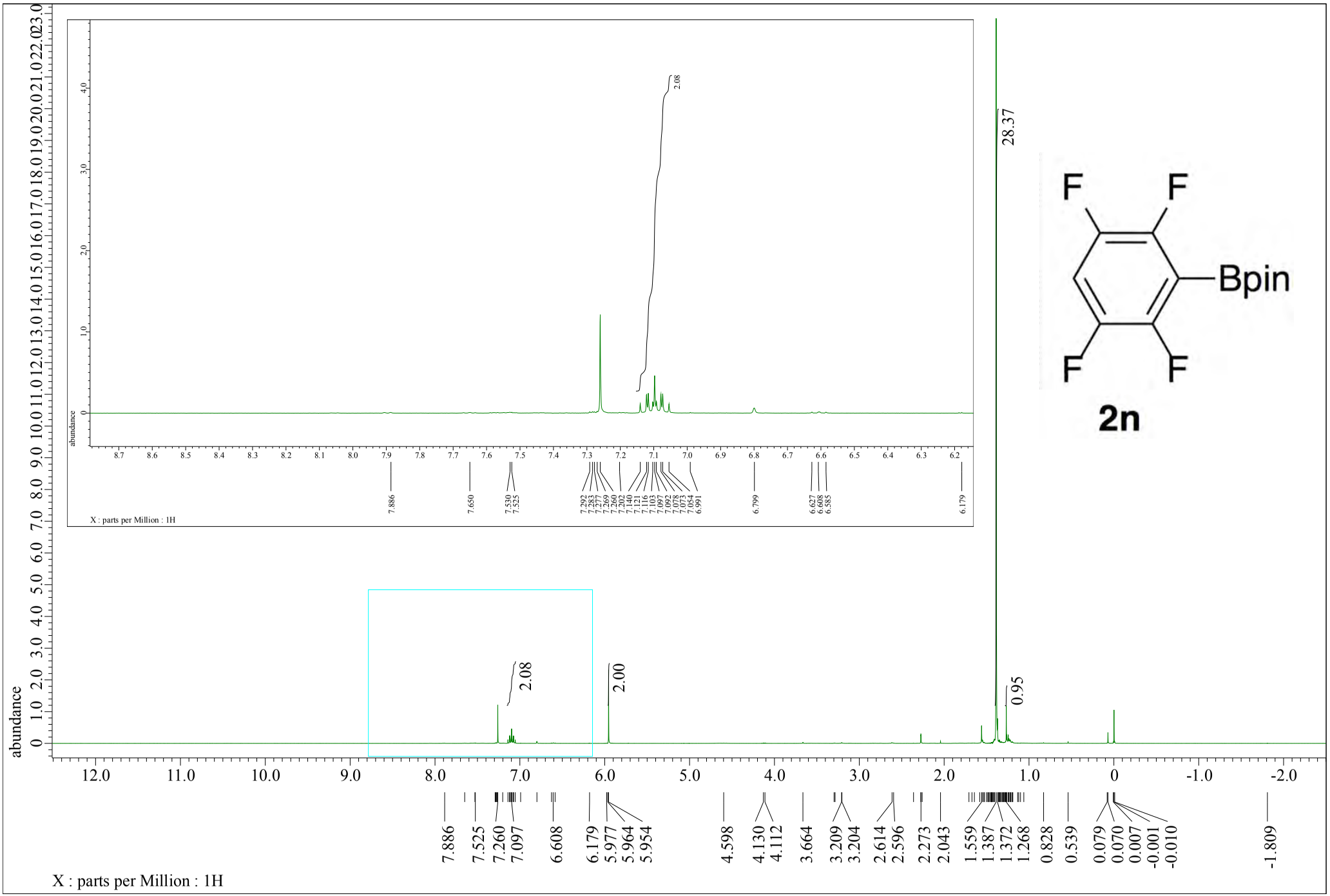


with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard

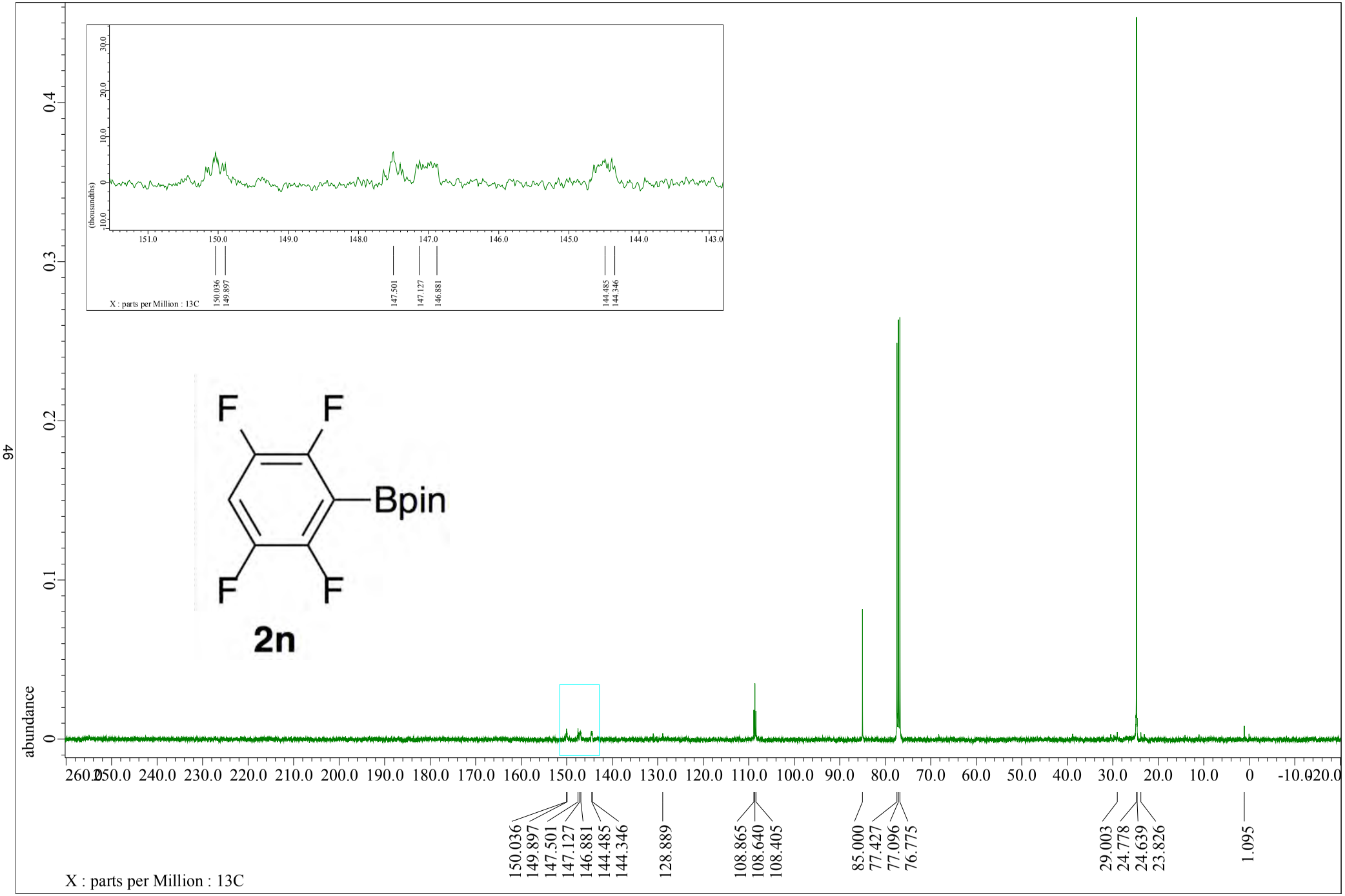


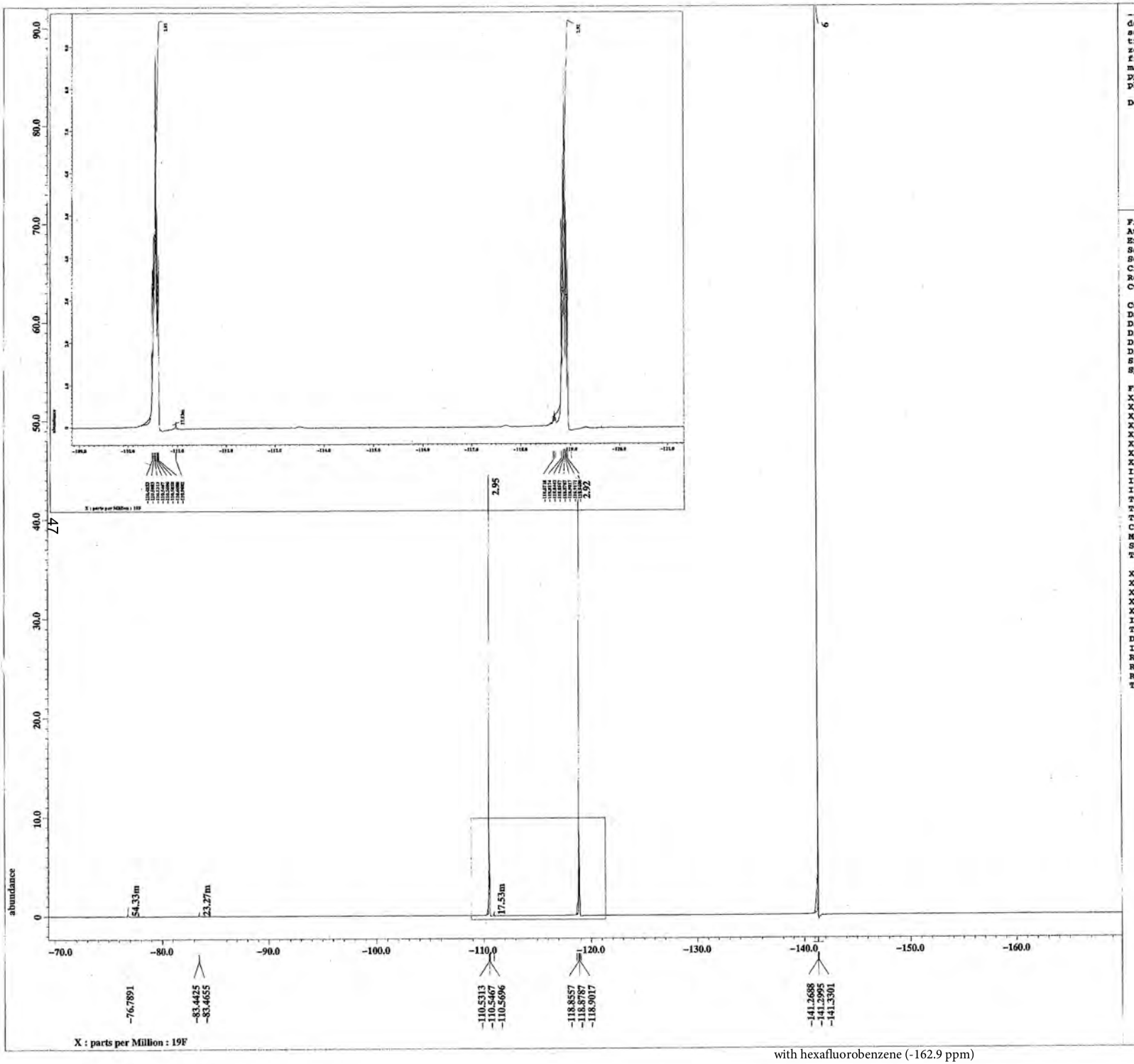
with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard

abundance
0
10.0
20.0
30.0
X : parts per Million : 19F
-100.0
-110.0
-120.0
-130.0
-140.0
-150.0
-160.0
-105.324
-105.339
-105.362
-105.382
-107.543
-107.558
-107.578
-107.585
-107.601
-107.620
-117.366
-117.377
-117.389
-117.400
-117.412
-117.423
-117.435
-117.446
-117.458
-126.012
-126.035
-126.069
-126.092
-129.638
-129.664
-129.695
-129.714
-134.700
-134.723
-134.754
-134.781
-135.076
-135.133
-135.141
-135.149
-144.765
-144.772
-144.791
-144.799
-144.807
-144.818
-144.834
-144.845
-144.857
-144.868
-144.876
-144.891
25.71m
0.23
60.47m
26.69m
0.23
61.93m
62.59m
26.71m
0.23
F
Bpin
F
α
β
γ
F
2m



with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard


46
abundance
0
0.1
0.2
0.3
0.4
(thousandths)
30.0
20.0
10.0
0
-10.0
151.0
150.0
149.0
148.0
147.0
146.0
145.0
144.0
143.0
150.036
149.897
147.501
147.127
146.881
144.485
144.346
X : parts per Million : 13C
F
F
Bpin
F
F
2n
260.0
250.0
240.0
230.0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
20.0
150.036
149.897
147.501
147.127
146.881
144.485
144.346
128.889
108.865
108.640
108.405
85.000
77.427
77.096
76.775
29.003
24.778
24.639
23.826
1.095
X : parts per Million : 13C

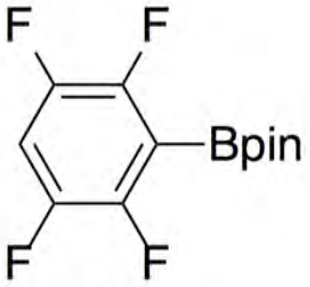



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : -6 : -3 : 71.58007[%]

Derived from: EX96 COLUMNN 19f-1.jdf
```

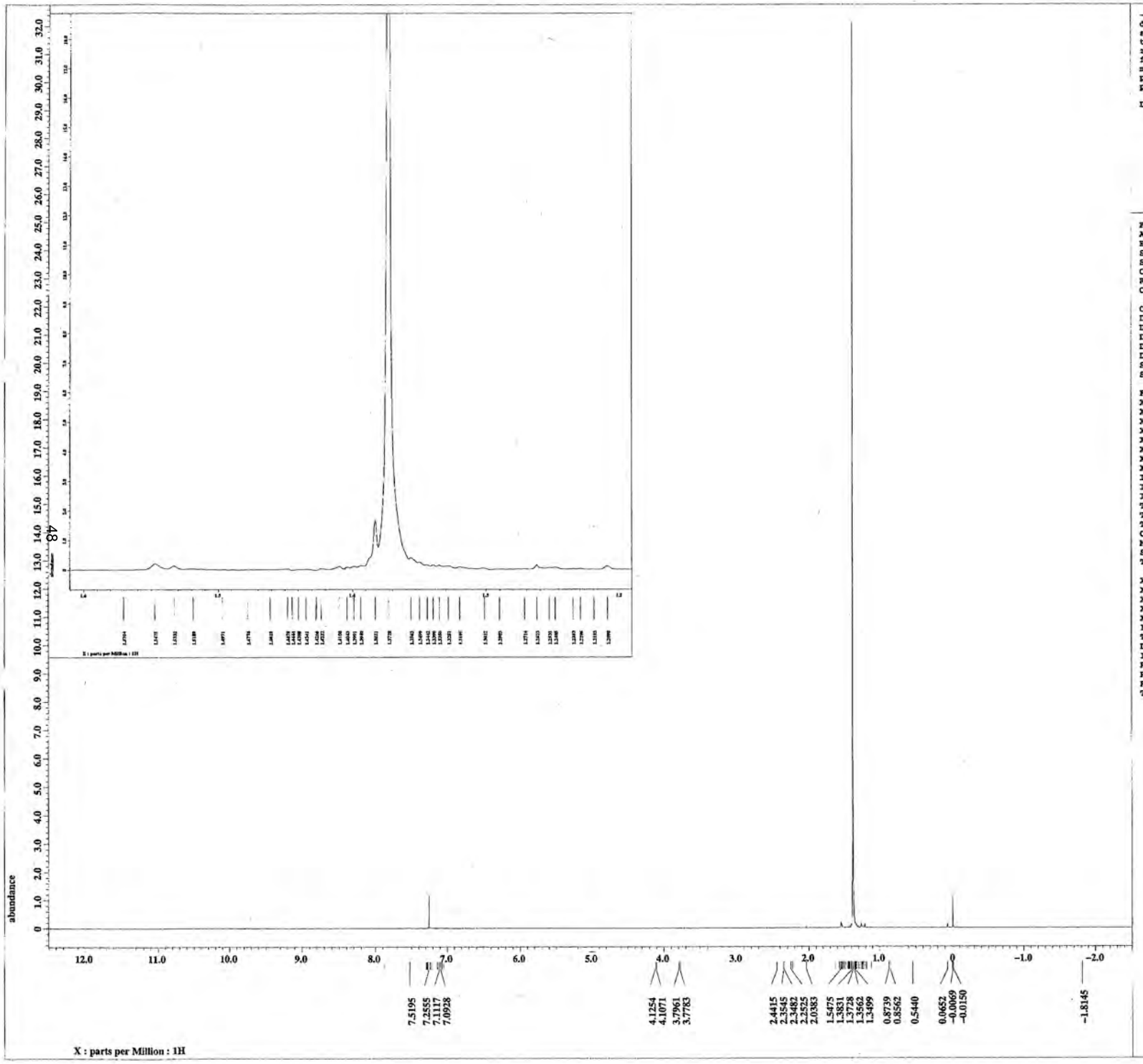
Filename	= EX96 COLUMNN 19f-5.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 24-SEP-2014 19:17:10
Revision_time	= 24-SEP-2014 20:12:52
Current_time	= 24-SEP-2014 20:13:17
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 19F
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 0.3538944[s]
X_domain	= 19F
X_freq	= 368.64763285[MHz]
X_offset	= -140[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 2.82570168[Hz]
X_sweep	= 46.2962963[kHz]
Irr_domain	= 19F
Irr_freq	= 368.64763285[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 19F
Tri_freq	= 368.64763285[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 11.5[us]
X_acq_time	= 0.3538944[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 5.3538944[s]
Temp_get	= 25.1[dC]

19F NMR



2n

with hexafluorobenzene (-162.9 ppm)
as an internal

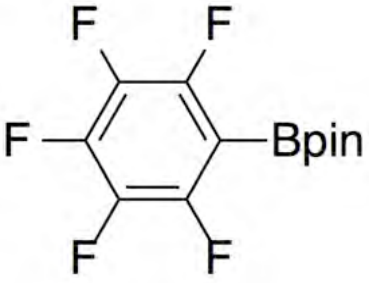



```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]

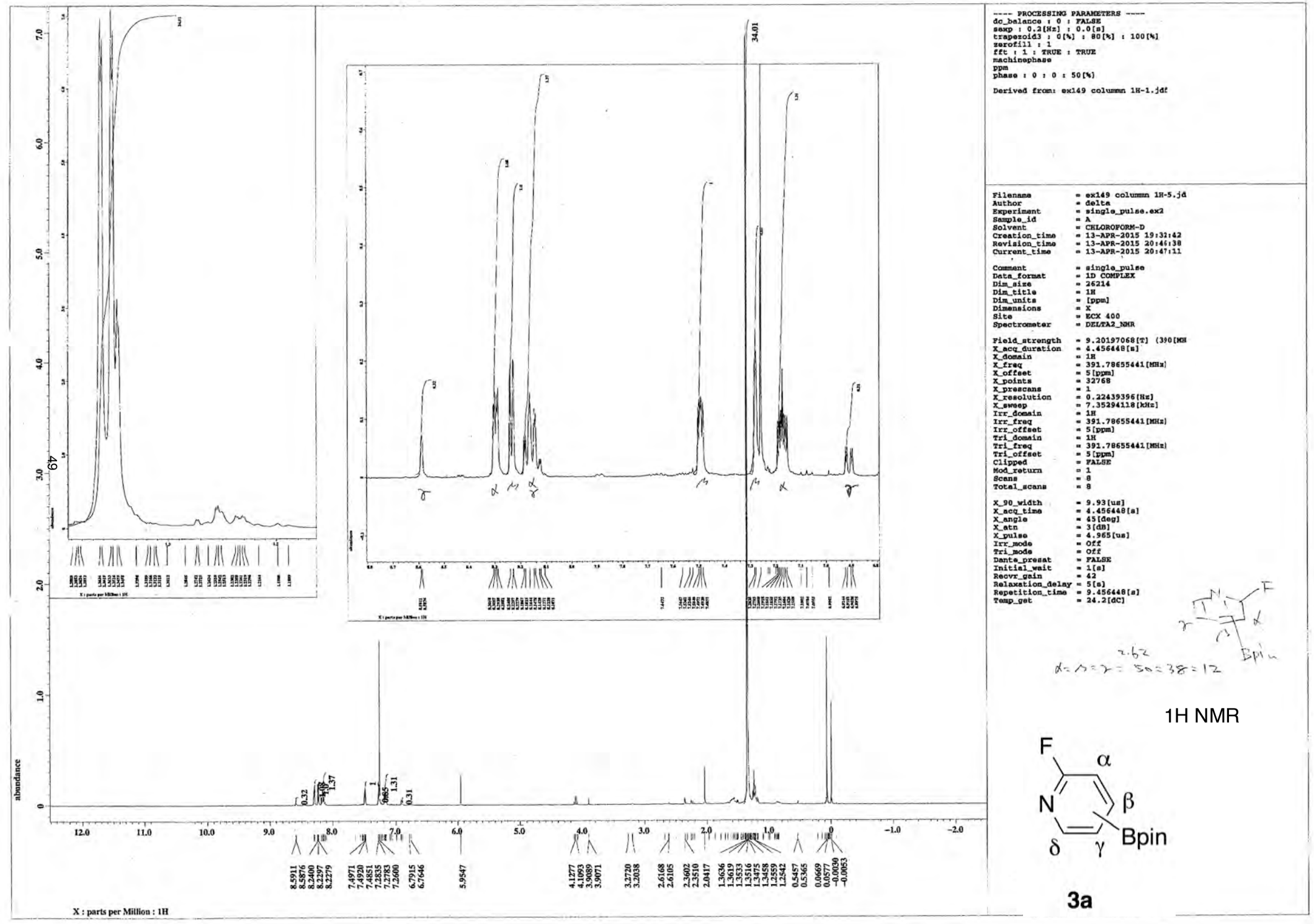
Derived from: ex 120 1H columnn-1.jdf
```

Filename	= ex 120 1H columnn-4.j
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 10-NOV-2014 17:56:09
Revision_time	= 10-NOV-2014 18:50:58
Current_time	= 10-NOV-2014 18:52:08
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.7[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.85[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 42
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 24.6[dC]

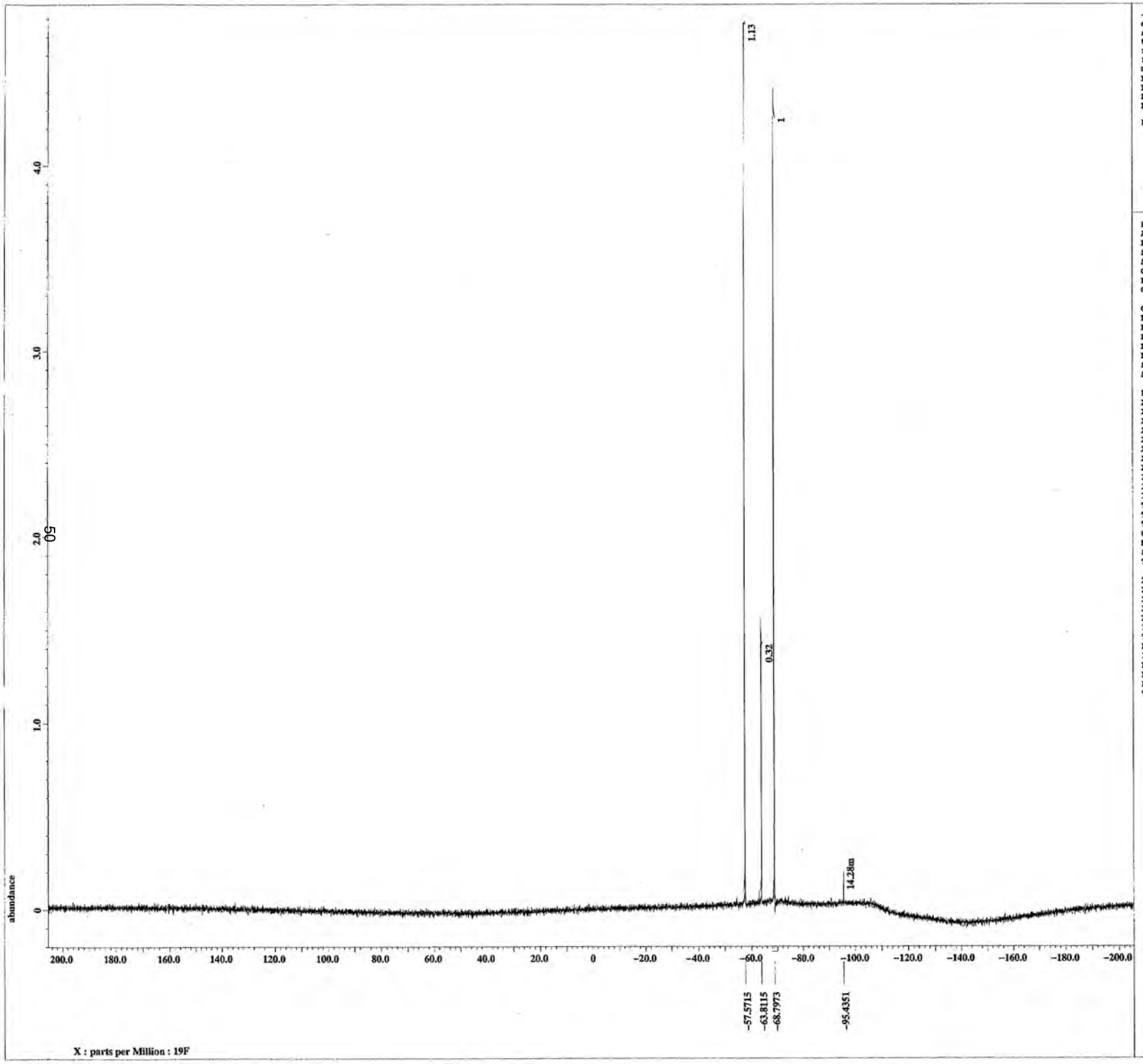
1H NMR



2o



with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 16.5 : 0 : 66.75059[%]
phase : 35 : 220 : 66.75059[%]
phase : -20 : -100 : 66.75059[%]

Derived from: ex149 columnn 19F-1.jdf
```

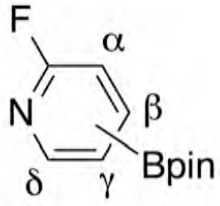
```
Filename          = ex149 columnn 19F-5.j
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = A
Solvent           = CHLOROFORM-D
Creation_time     = 13-APR-2015 19:36:44
Revision_time     = 13-APR-2015 20:49:47
Current_time      = 13-APR-2015 20:49:55

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

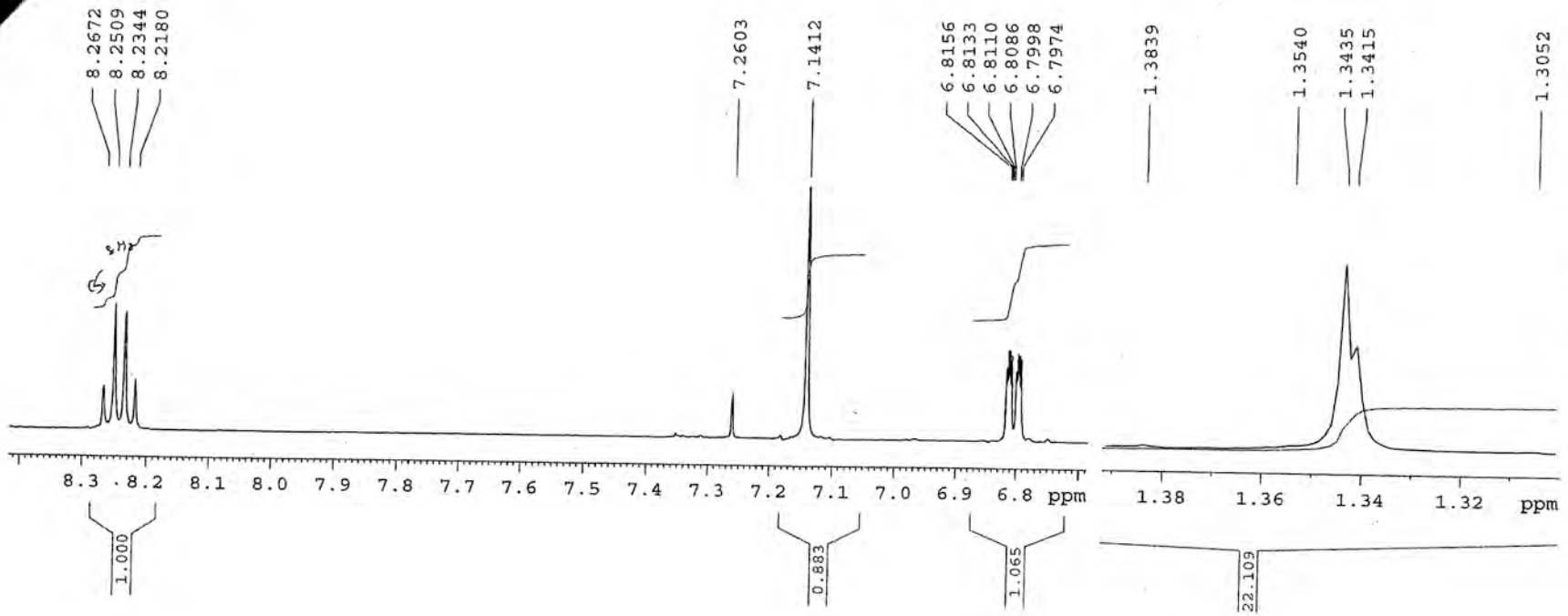
Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 86.50752[ms]
X_domain          = 19F
X_freq            = 368.64763285[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 11.55968868[Hz]
X_sweep           = 189.39393939[kHz]
Irr_domain        = 19F
Irr_freq          = 368.64763285[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 368.64763285[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 20
Total_scans       = 20

X_90_width        = 11.5[us]
X_acq_time        = 86.50752[ms]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.08650752[s]
Temp_get          = 24.2[dC]
```

19F NMR



3a



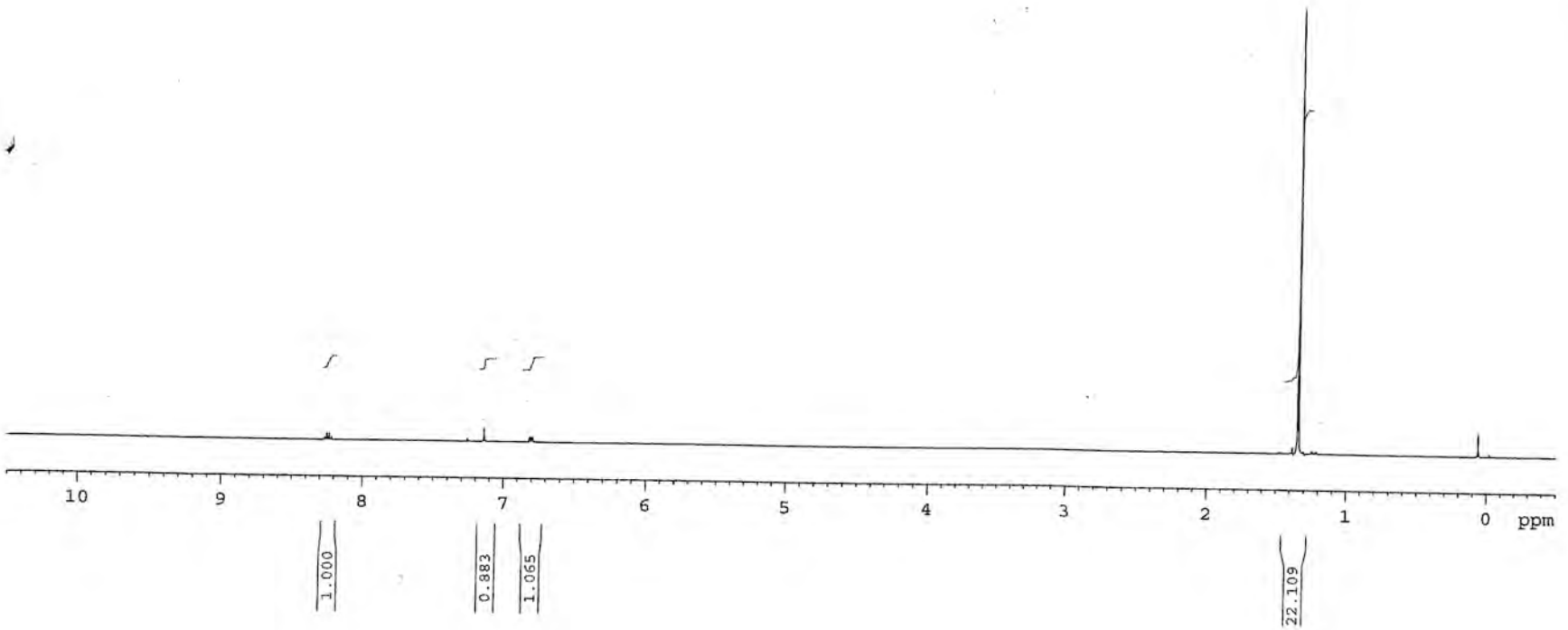

```
Current Data Parameters
NAME                  Shisei
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20150129
Time                 22.31
INSTRUM              spect
PROBHD    5 mm Multinucl
PULPROG                zg
TD                 159996
SOLVENT             CDCl3
NS                     10
DS                      2
SWH          20000.000 Hz
FIDRES           0.125003 Hz
AQ               3.9999750 sec
RG                   28.5
DW                 25.000 usec
DE                  6.00 usec
TE                 300.0 K
D1               3.00000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.00 usec
PL1                -3.50 dB
SFO1         500.1274993 MHz

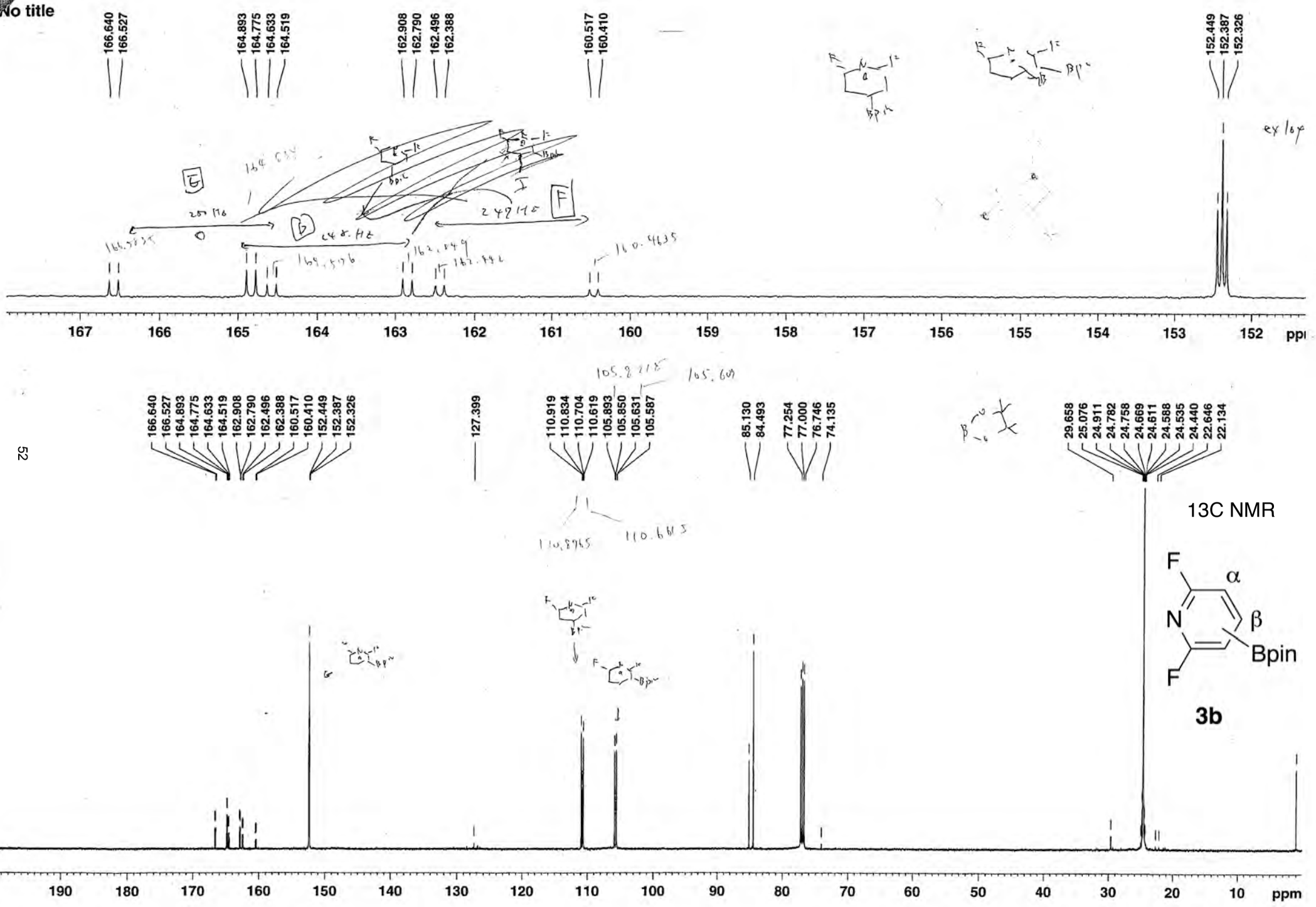
F2 - Processing parameters
SI                   32768
SF           500.1300128 MHz
WDW                    EM
SSB                     0
LB                 0.20 Hz
GB                      0
PC                   1.00
```

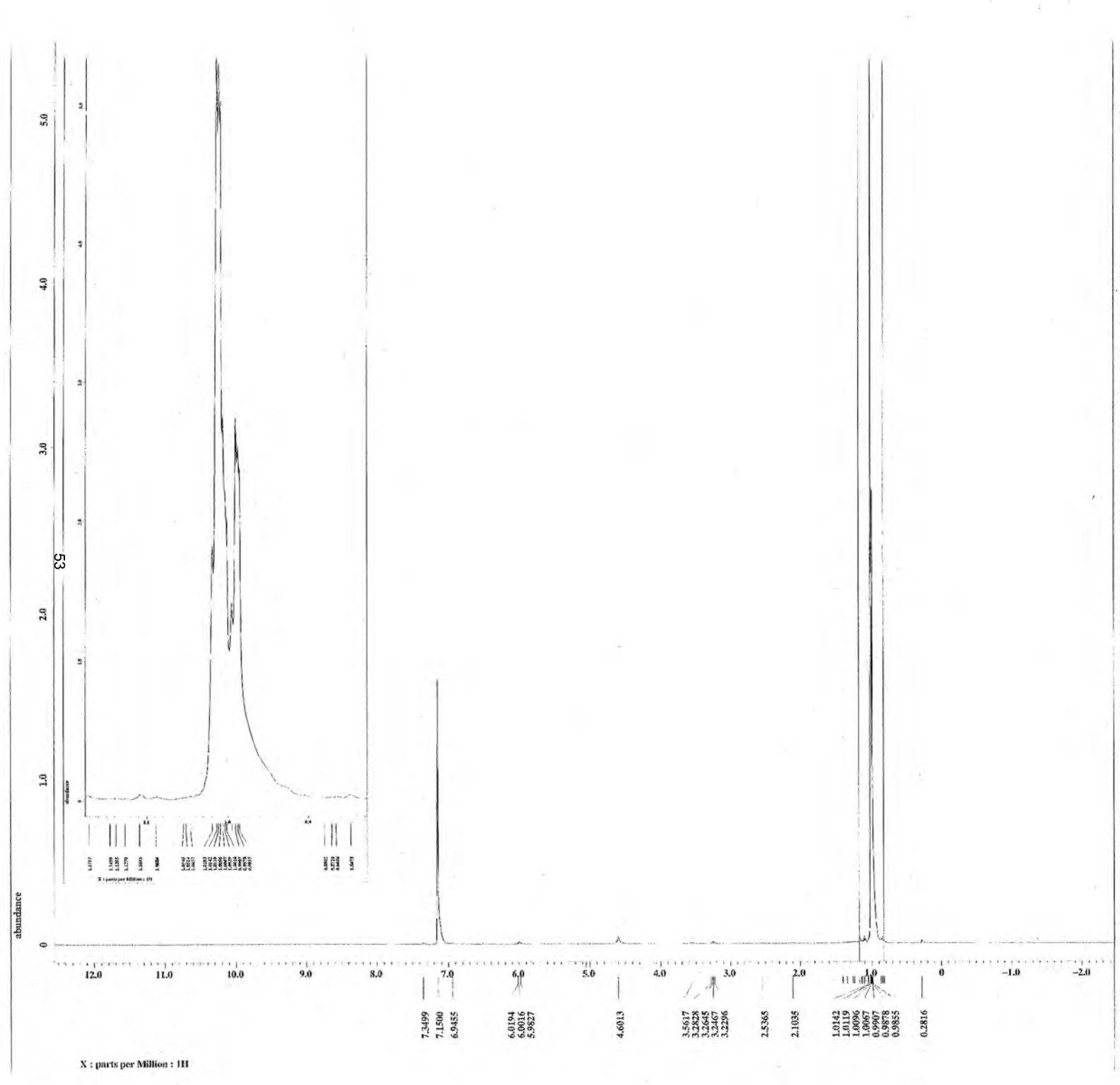
51



1H NMR

3b


166.640
166.527
164.893
164.775
164.633
164.519
162.908
162.790
162.496
162.388
160.517
160.410
152.449
152.387
152.326
127.399
110.919
110.834
110.704
110.619
105.893
105.850
105.631
105.587
85.130
84.493
77.254
77.000
76.746
74.135
29.658
25.076
24.911
24.782
24.758
24.669
24.611
24.588
24.535
24.440
22.646
22.134
13C NMR
F
α
N
β
Bpin
F
3b
ppm

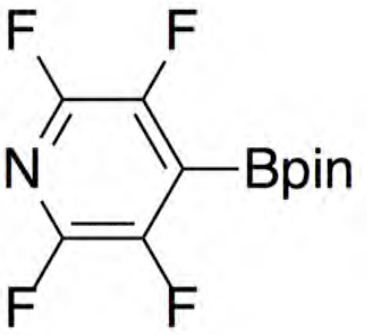



```
----- PROCESSING PARAMETERS -----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

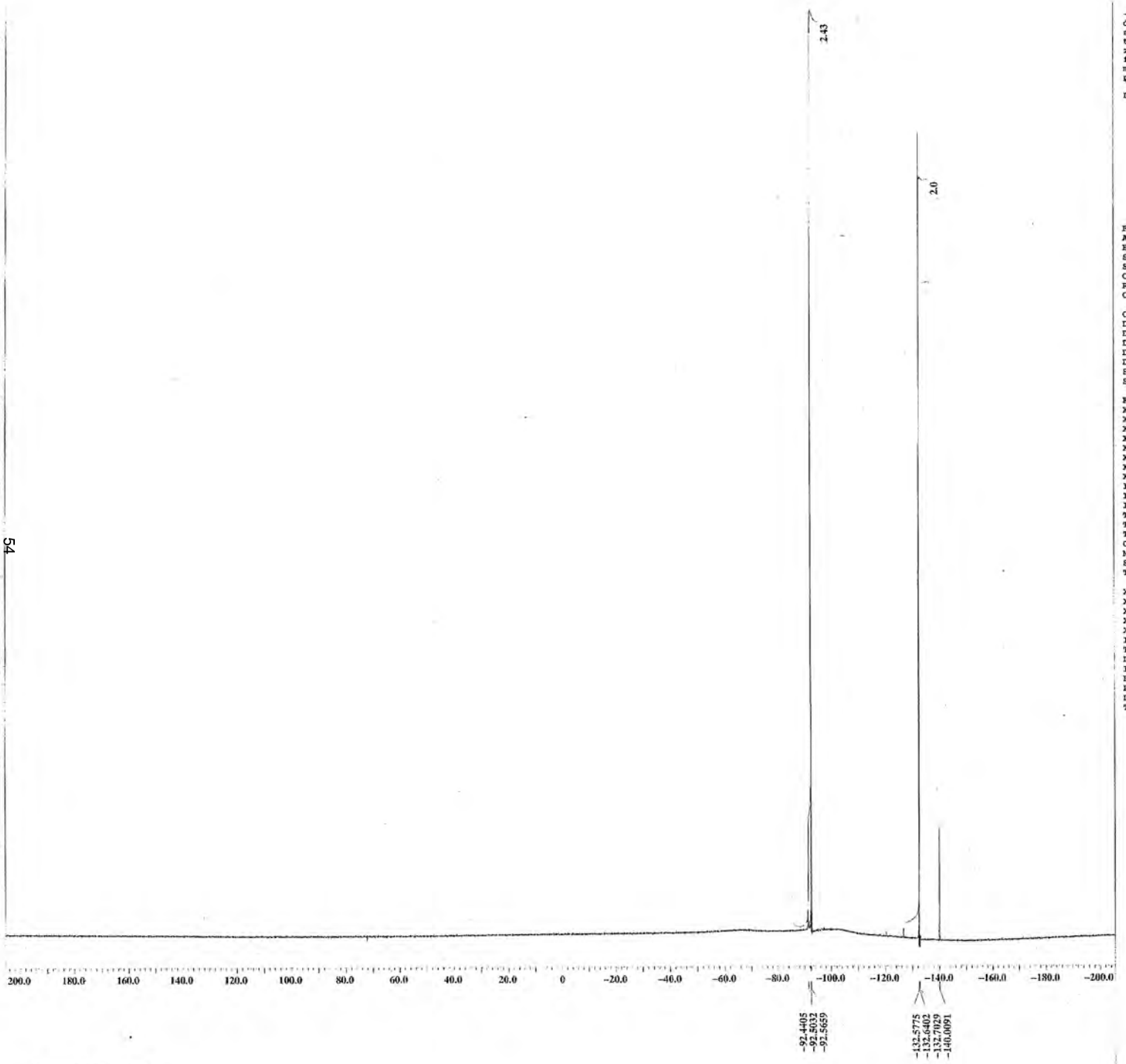
Derived from: N F4 BPIN -1.jdf
```

Filename	= N F4 BPIN -4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= BENZENE-D6
Creation_time	= 3-AUG-2015 18:39:31
Revision_time	= 3-AUG-2015 20:49:27
Current_time	= 3-AUG-2015 20:49:48
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DEL/FA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.93[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.965[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 23.5[dC]

1H NMR



3c




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: N F4 Bpin-1.jdf

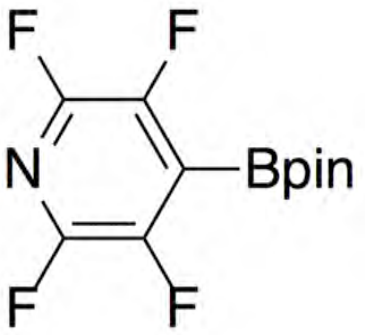
Filename          = N F4 Bpin-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = A
Solvent           = BENZENE-D6
Creation_time     =  3-AUG-2015 18:46:06
Revision_time     =  3-AUG-2015 20:56:39
Current_time      =  3-AUG-2015 20:56:45

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 19F
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

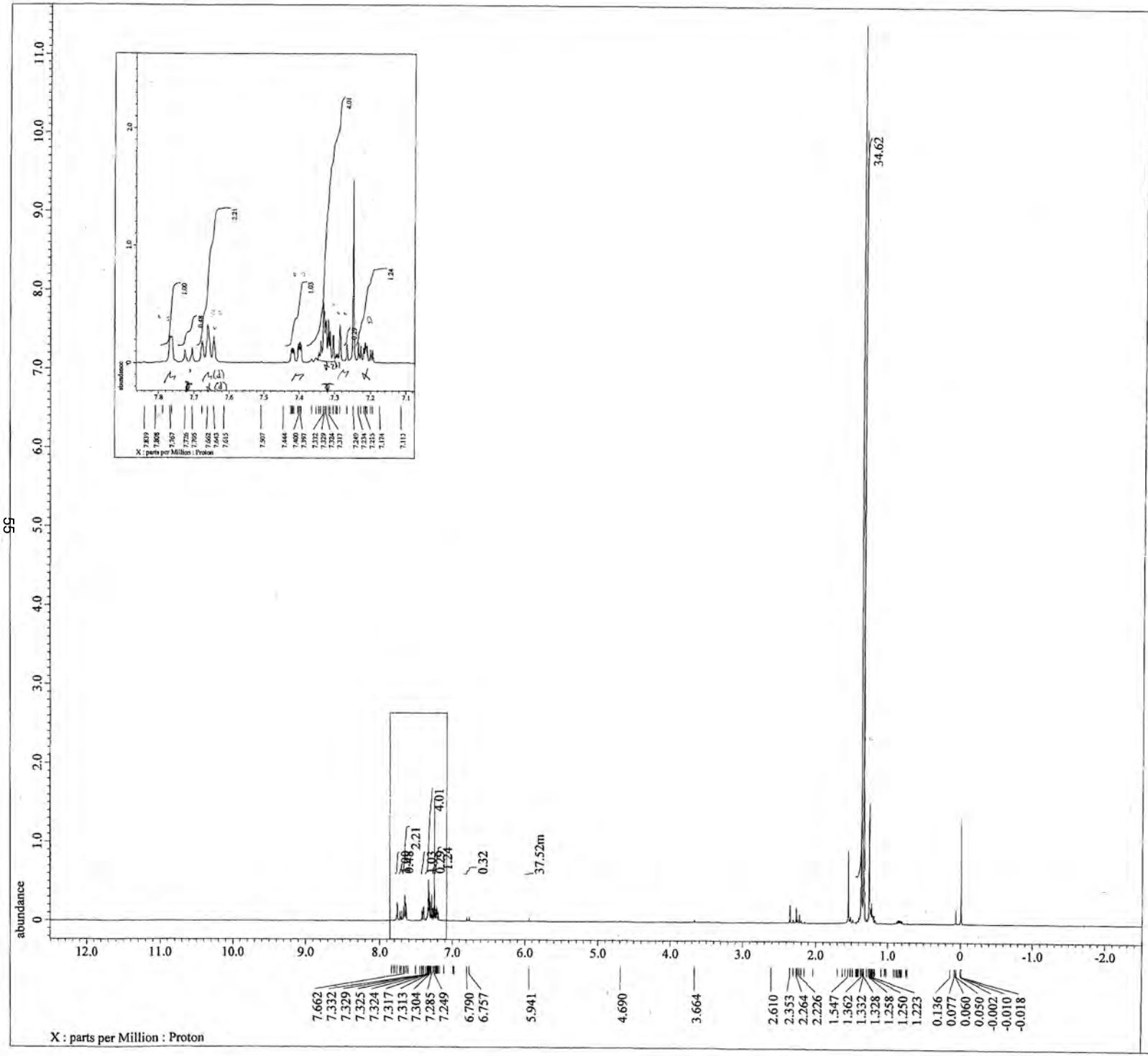
Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 86.50752[ms]
X_domain          = 19F
X_freq            = 368.64763285[MHz]
X_offset          = 0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 11.55968868[Hz]
X_sweep           = 189.39393939[kHz]
Irr_domain        = 19F
Irr_freq          = 368.64763285[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 19F
Tri_freq          = 368.64763285[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 51
Total_scans       = 51

X_90_width        = 11.5[us]
X_acq_time        = 86.50752[ms]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 5.08650752[s]
Temp_get          = 23.5[dC]
```

19F NMR

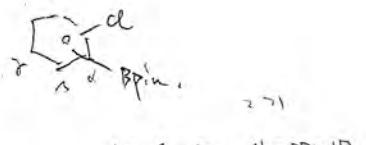


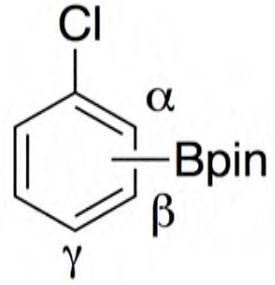
3c



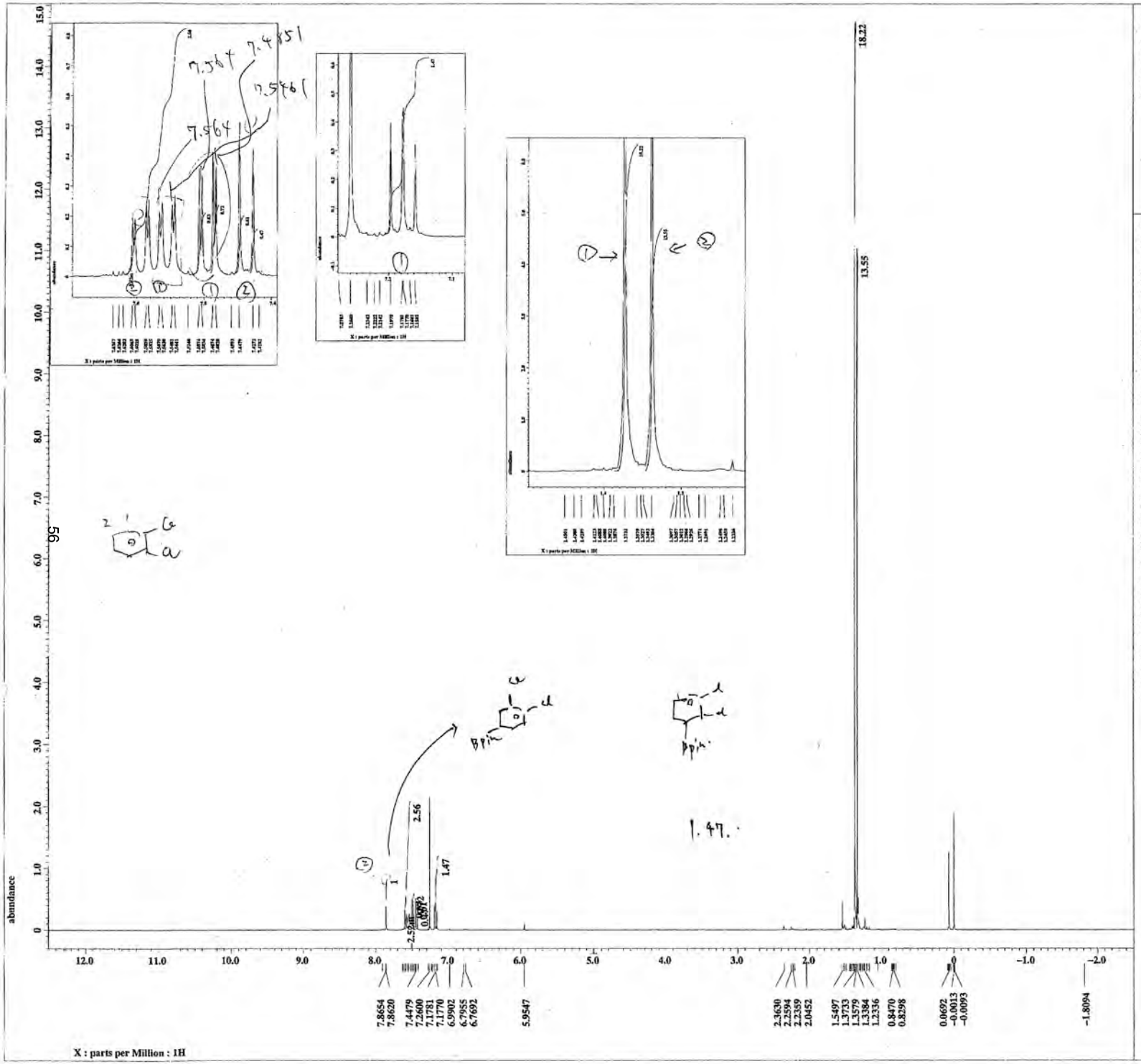
Filename	= ex274 solumnn_Proton-1-3.j
Author	= iwasawa
Experiment	= proton.jxp
Sample_Id	= ex274 solumnn
Solvent	= CHLOROFORM-D
Creation_Time	= 21-JUL-2015 13:24:52
Revision_Time	= 21-JUL-2015 13:29:37
Current_Time	= 21-JUL-2015 13:29:44
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-EC8400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 4.36731904[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.22897343[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	= 26.7[dC]
X_90_Width	= 6.15[us]
X_Acq_Time	= 4.36731904[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 3.075[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 9.36731904[s]

1H NMR





4a

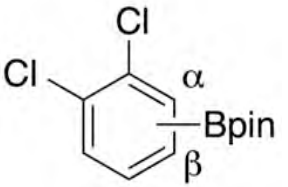



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

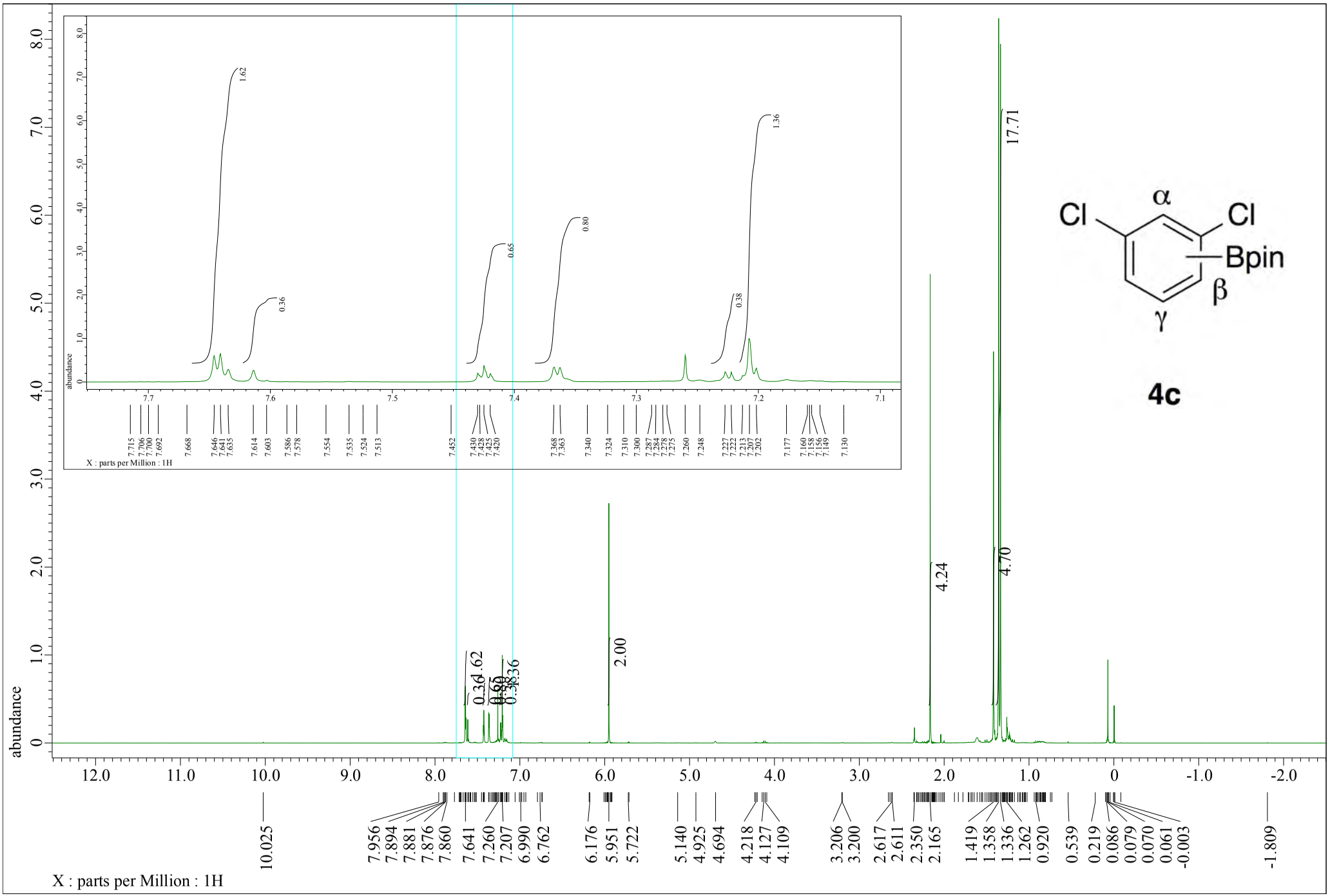
Derived from: ex148 columnn 1H-1.jdf
```

Filename	= ex148 columnn 1H-5.jd
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 18-DEC-2014 18:04:52
Revision_time	= 18-DEC-2014 19:26:26
Current_time	= 18-DEC-2014 19:27:02
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.7[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.85[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 44
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 22.4[dC]

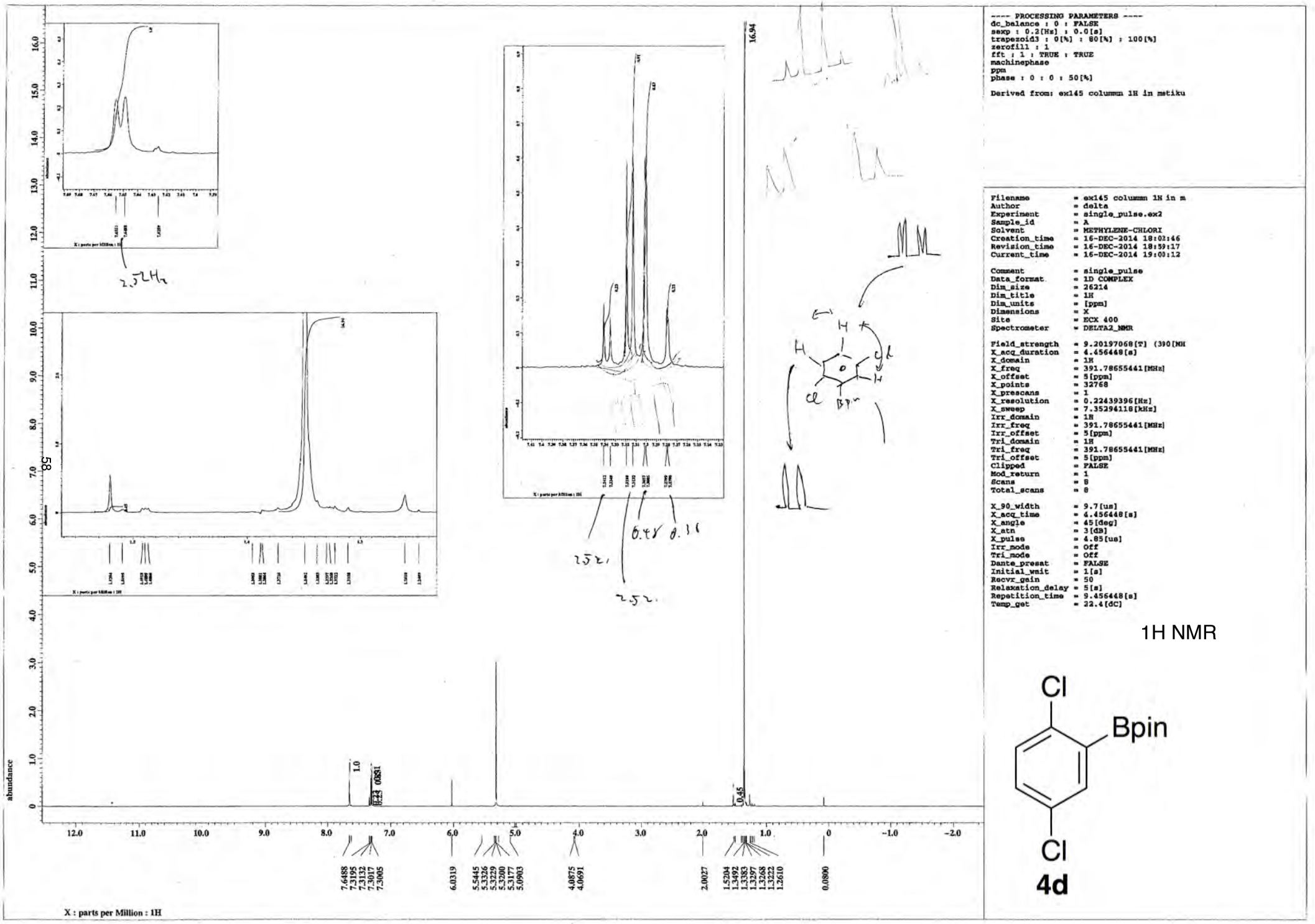
1H NMR

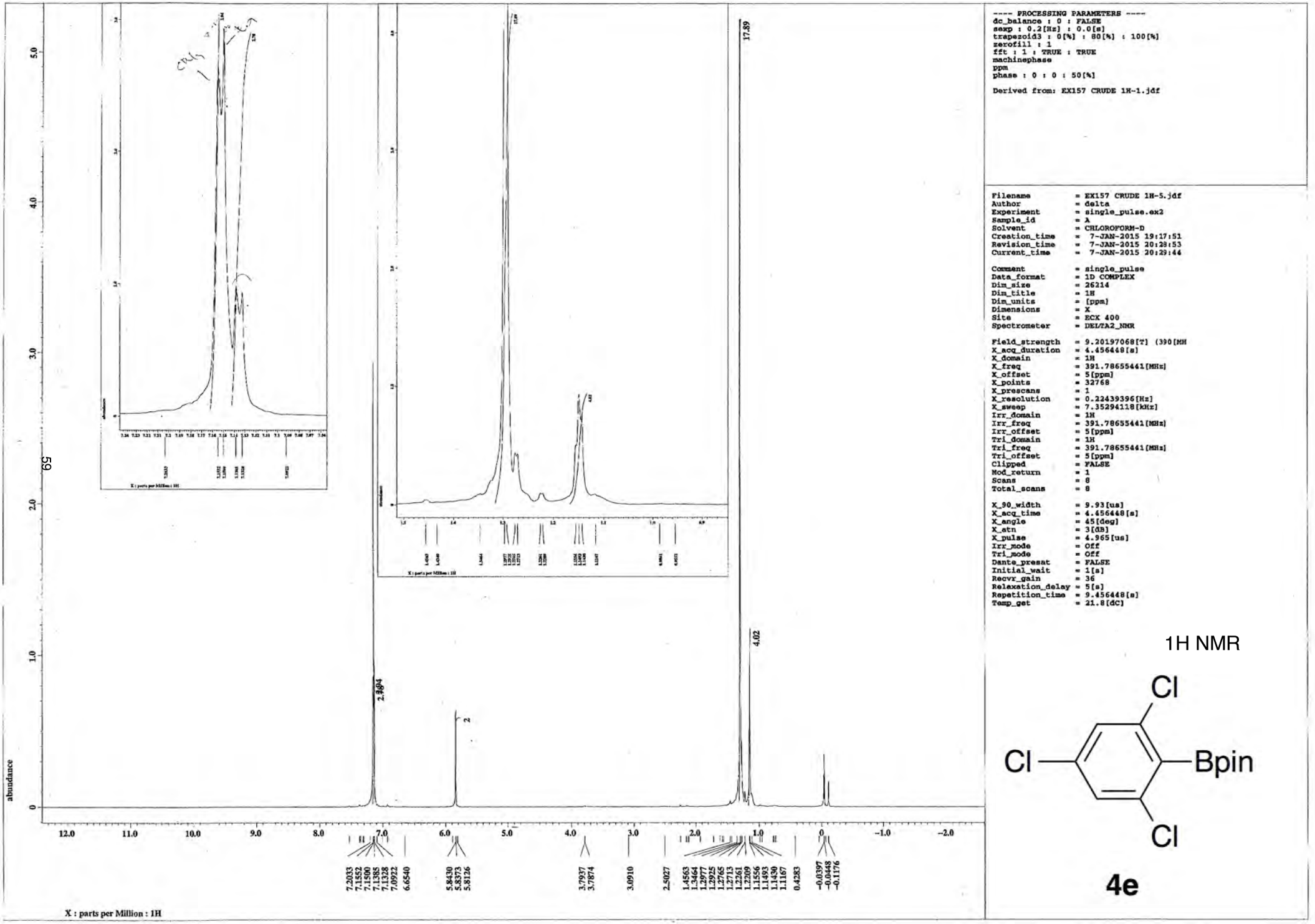


4b

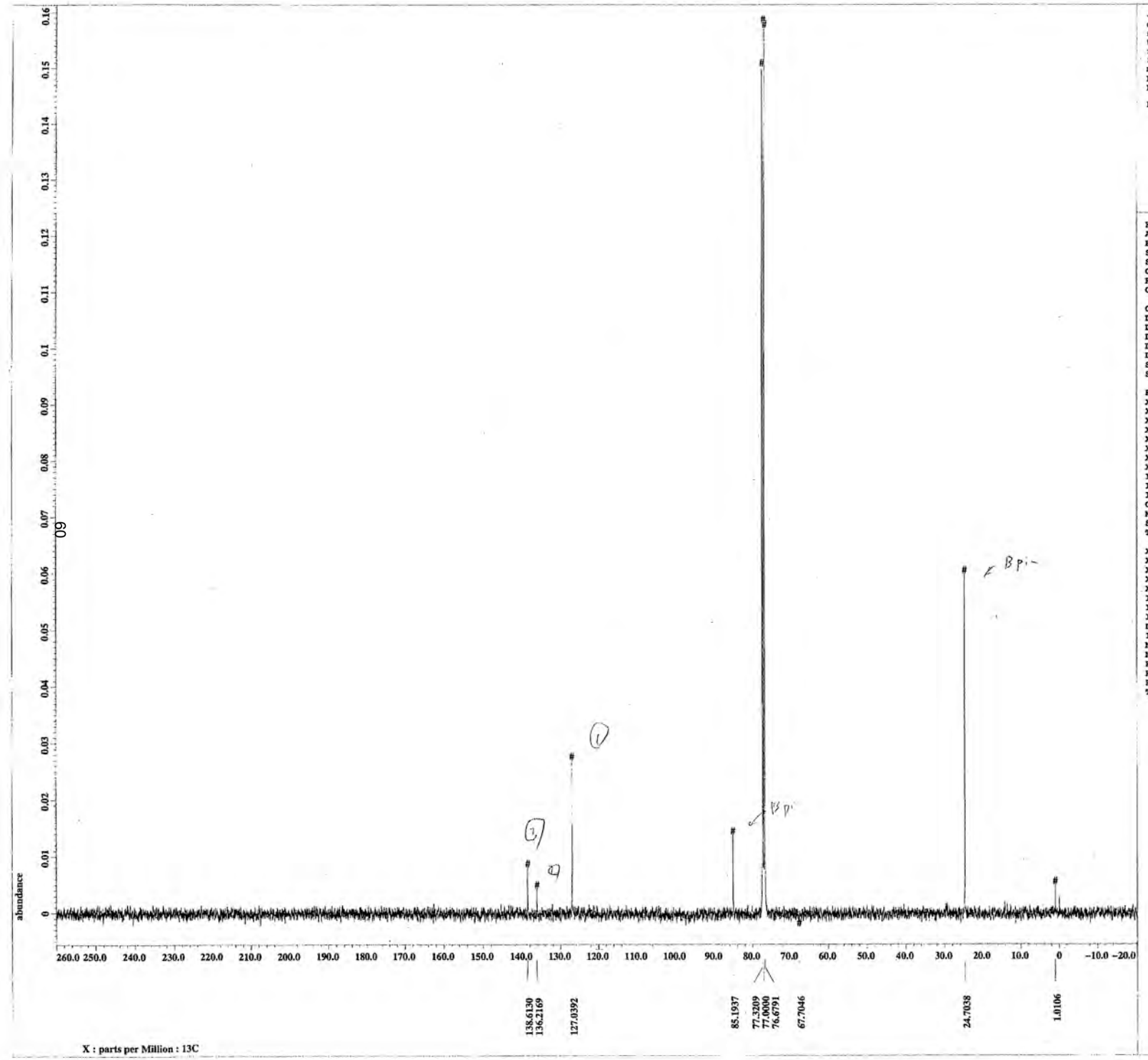


with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard


---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]
Derived from: ex145 columnn 1H in mstiku
Filename = ex145 columnn 1H in m
Author = delta
Experiment = single_pulse.ex2
Sample_id = A
Solvent = METHYLENE-CHLORI
Creation_time = 16-DEC-2014 18:02:46
Revision_time = 16-DEC-2014 18:59:17
Current_time = 16-DEC-2014 19:00:12
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 4.456448[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 0.22439396[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 9.7[us]
X_acq_time = 4.456448[s]
X_angle = 45[deg]
X_atn = 3[dB]
X_pulse = 4.85[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 9.456448[s]
Temp_get = 22.4[dC]
1H NMR
Cl
Bpin
Cl
4d
abundance
X : parts per Million : 1H
7.6488
7.3195
7.3132
7.3017
7.3005
6.0319
5.5445
5.3326
5.3229
5.3200
5.3177
5.0903
4.0875
4.0691
2.0027
1.5204
1.3492
1.3383
1.3297
1.3268
1.3222
1.2610
0.0890



with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]

Derived from: EX157 DATE 13C-1.jdf
```

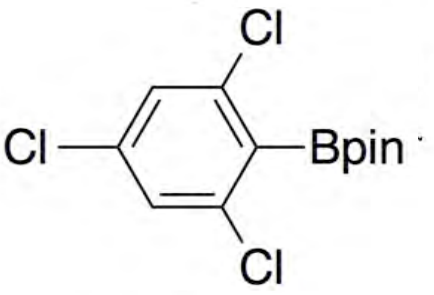
```
Filename          = EX157 DATE 13C-8.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = A
Solvent           = CHLOROFORM-D
Creation_time     =  7-MAY-2015 14:41:08
Revision_time     =  7-MAY-2015 15:55:15
Current_time      =  7-MAY-2015 15:56:26

Comment           = 13C_dec
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

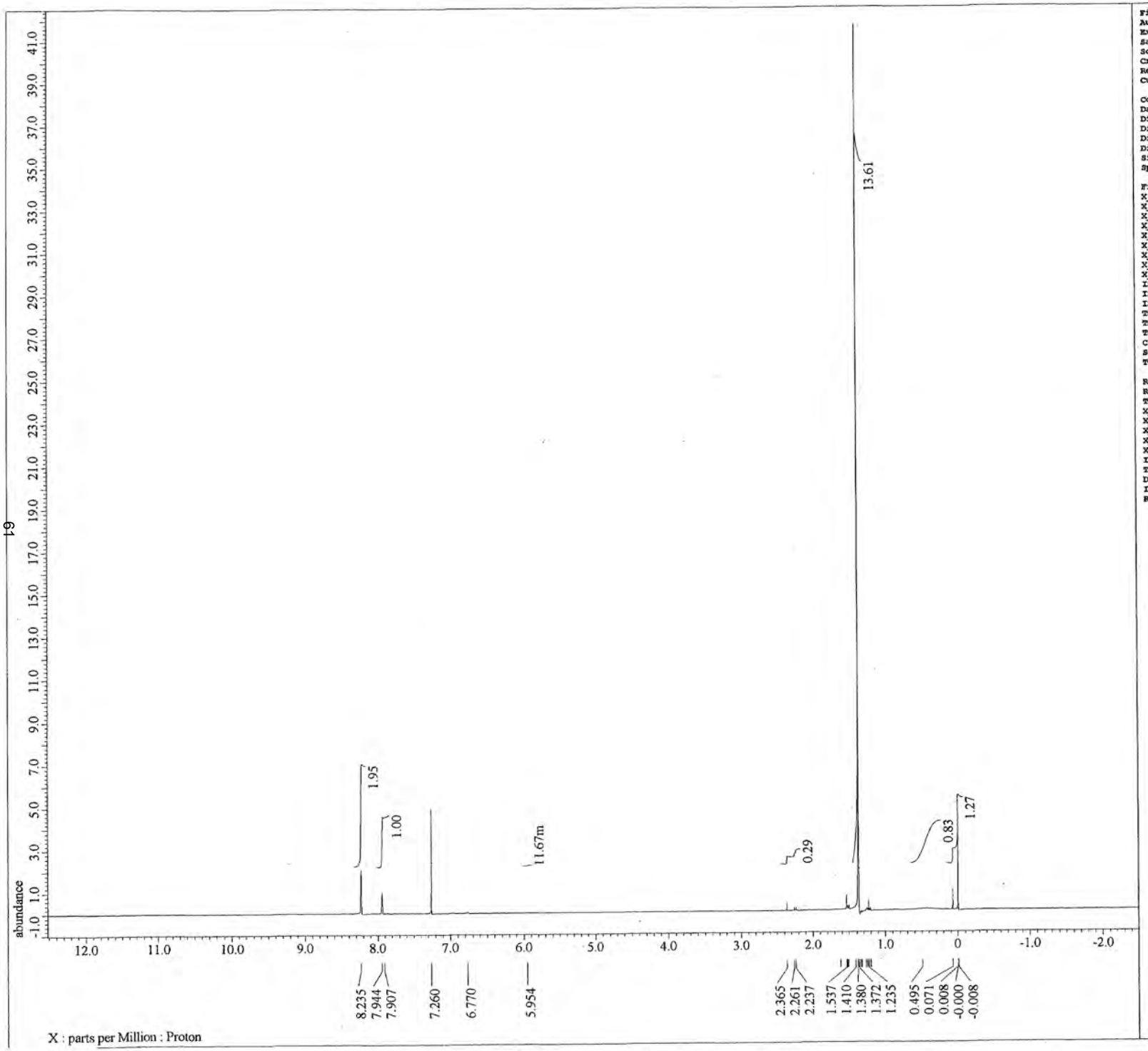
Field_strength    = 9.20197068[T]  (390[MH
X_acq_duration    = 0.94896128[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.05378378[Hz]
X_sweep           = 34.53038674[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 903.0
Total_scans       = 903.0

X_90_width        = 10.75[us]
X_acq_time        = 0.94896128[s]
X_angle           = 30[deg]
X_atn             = 9.7[dB]
X_pulse           = 3.58333333[us]
Irr_atn_dec       = 24.27[dB]
Irr_atn_noe       = 24.27[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 46
Relaxation_delay  = 2[s]
Repetition_time   = 2.94896128[s]
Temp_get          = 25.2[dC]
```

13C NMR



4e



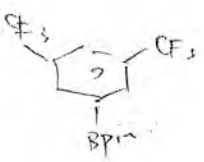

```
Filename          = ex152 columnn 1H_Proton-1-
Author            = iwasawa
Experiment        = proton.jxp
Sample_Id         = ex152 columnn 1H
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2014 17:29:31
Revision_Time     = 22-DEC-2014 17:34:25
Current_Time      = 22-DEC-2014 17:34:33

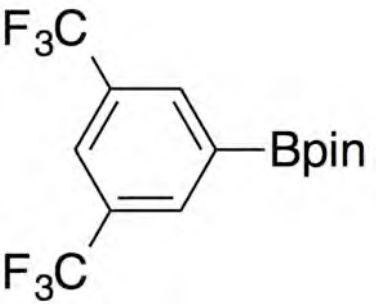
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[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

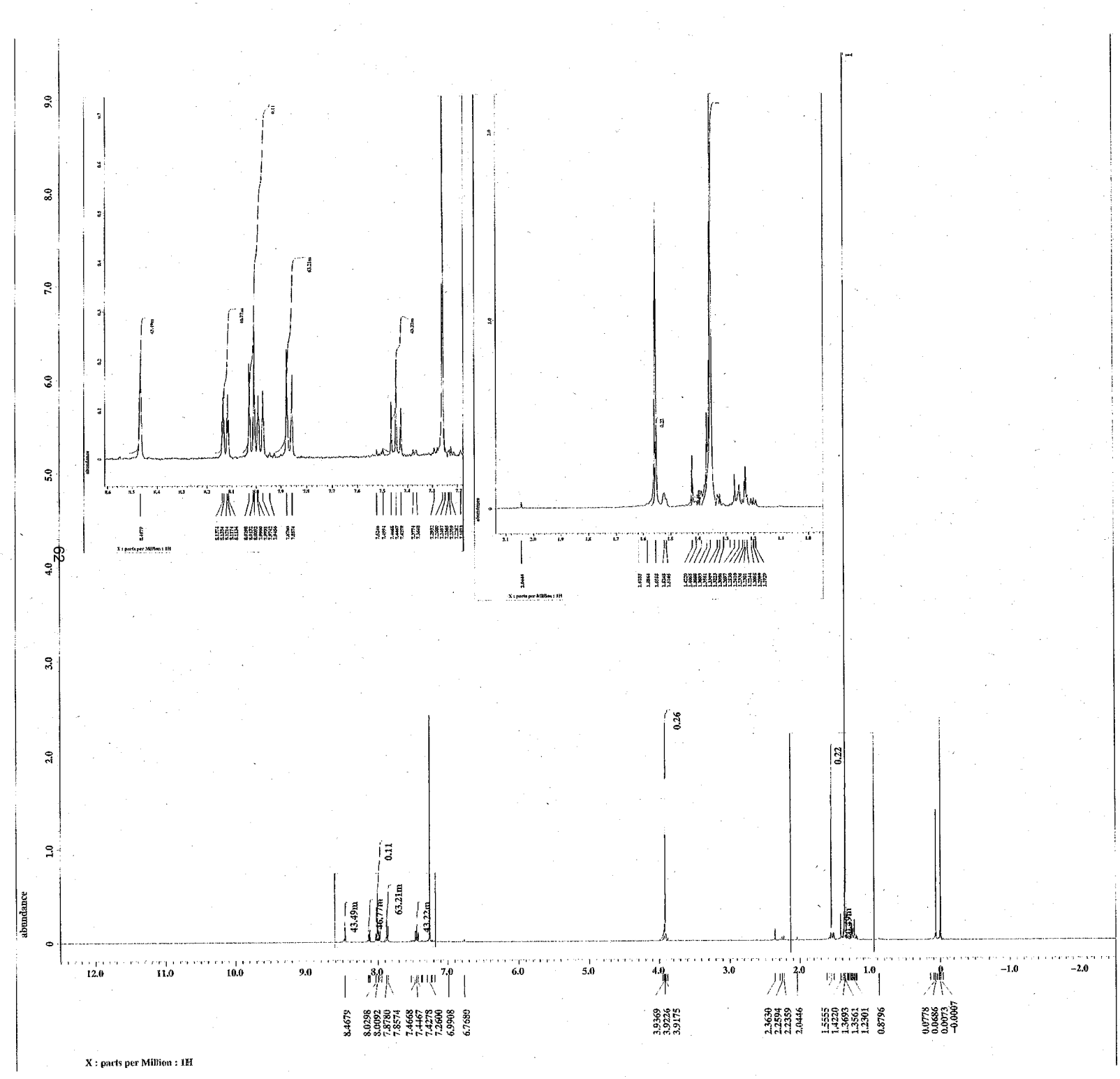
Relaxation_Delay  = 3[s]
Recvr_Gain        = 40
Temp_Get          = 25.5[dC]
X_90_Width        = 6.29[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 3.145[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 5.18365952[s]
```

1H NMR





4f



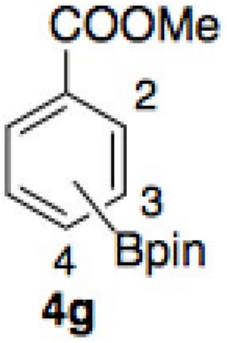

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]
phase : 0 : 0 : 50[%]
```

```
Filename          = ex275 crude 1H-18.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = A
Solvent           = CHLOROFORM-D
Creation_time     = 17-SEP-2015 20:37:59
Revision_time     = 17-SEP-2015 22:52:50
Current_time      = 17-SEP-2015 22:55:28

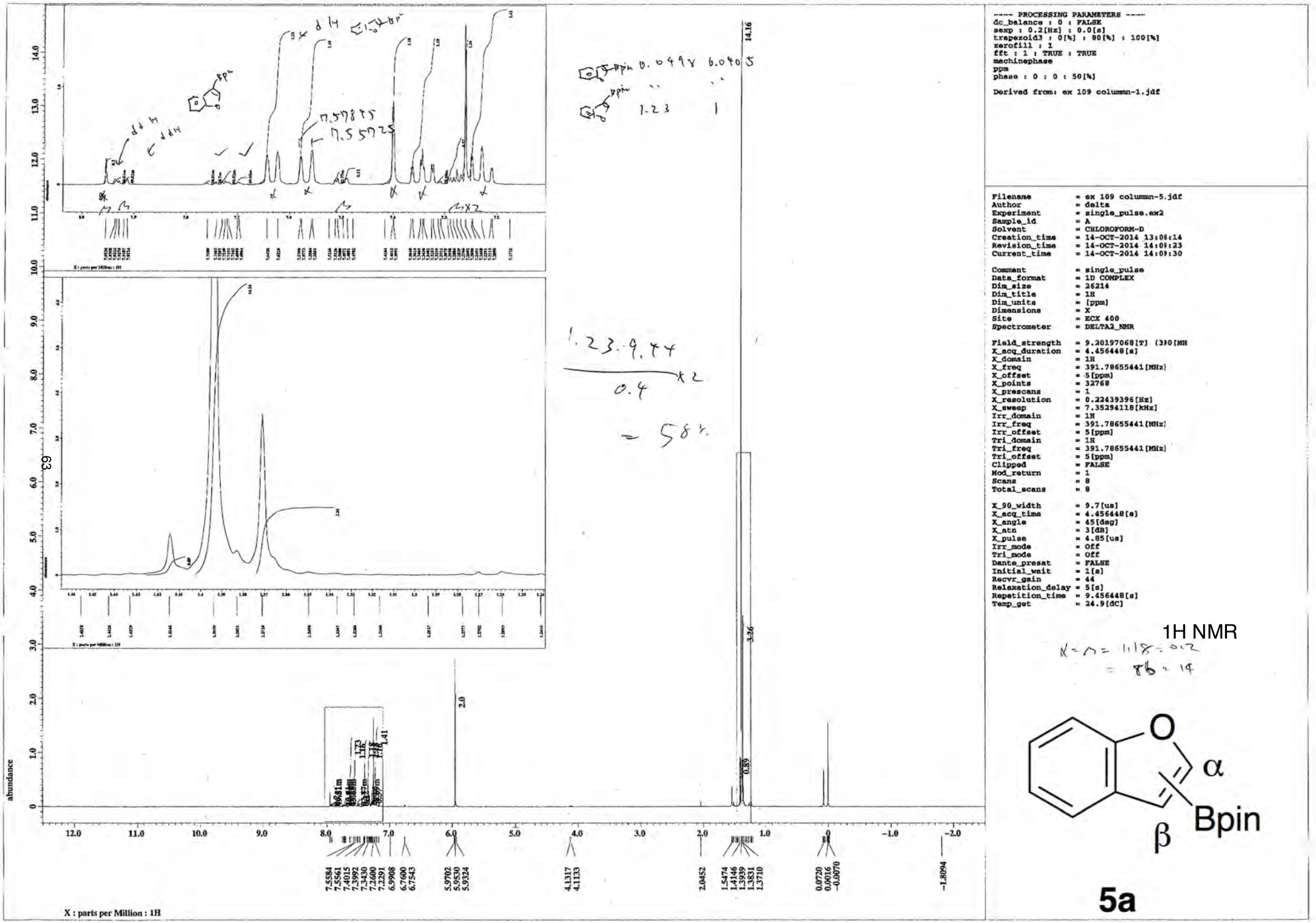
Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400
Spectrometer      = DELTA2_NMR

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 4.456448[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 32768
X_prescans        = 1
X_resolution      = 0.22439396[Hz]
X_sweep           = 7.35294118[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

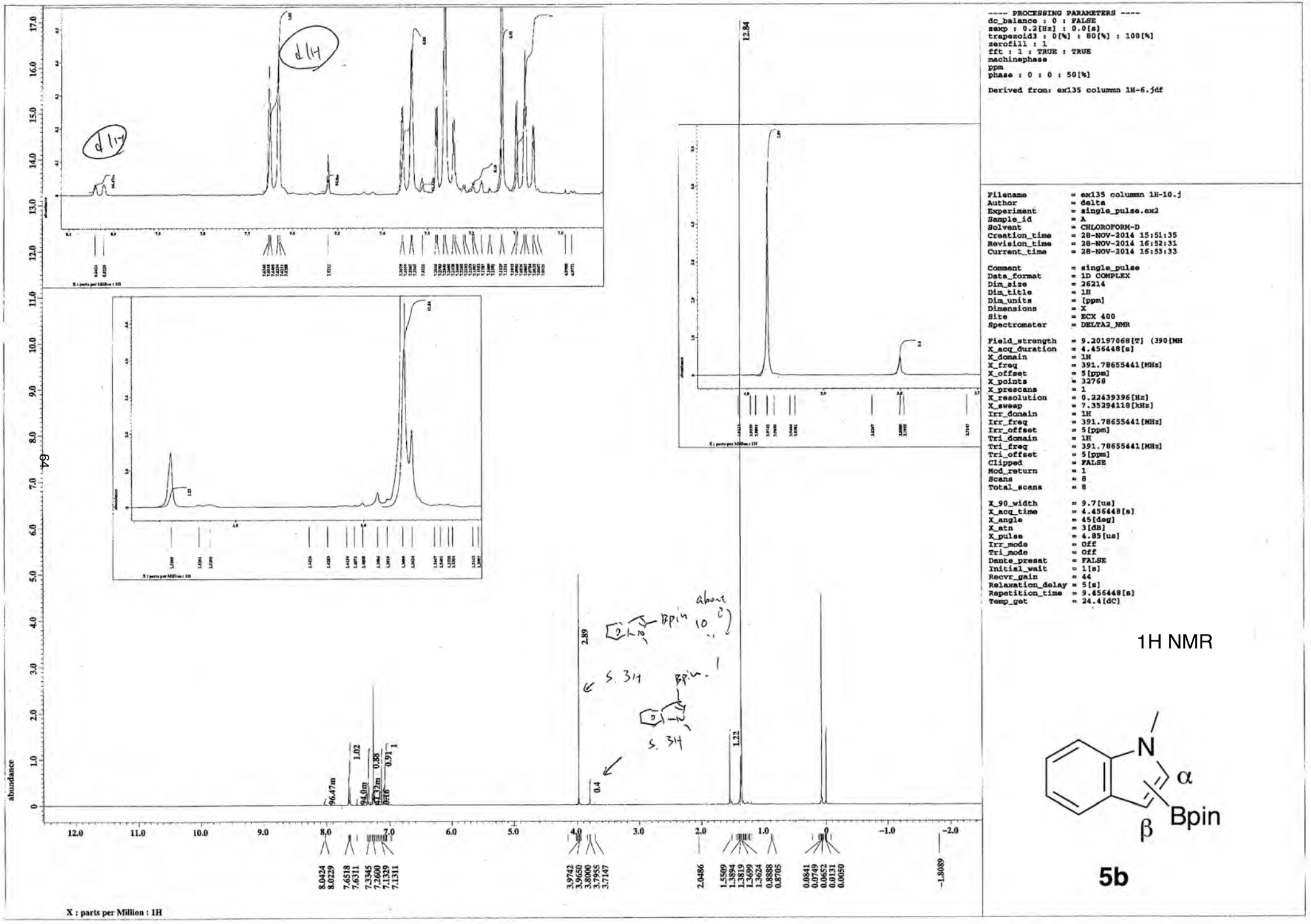
X_90_width        = 9.93[us]
X_acq_time        = 4.456448[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 4.965[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 9.456448[s]
Temp_get          = 23.3[dC]
```



4g



with 1,1,2,2-tetrachloroethane (5.9 ppm)
as an internal standard



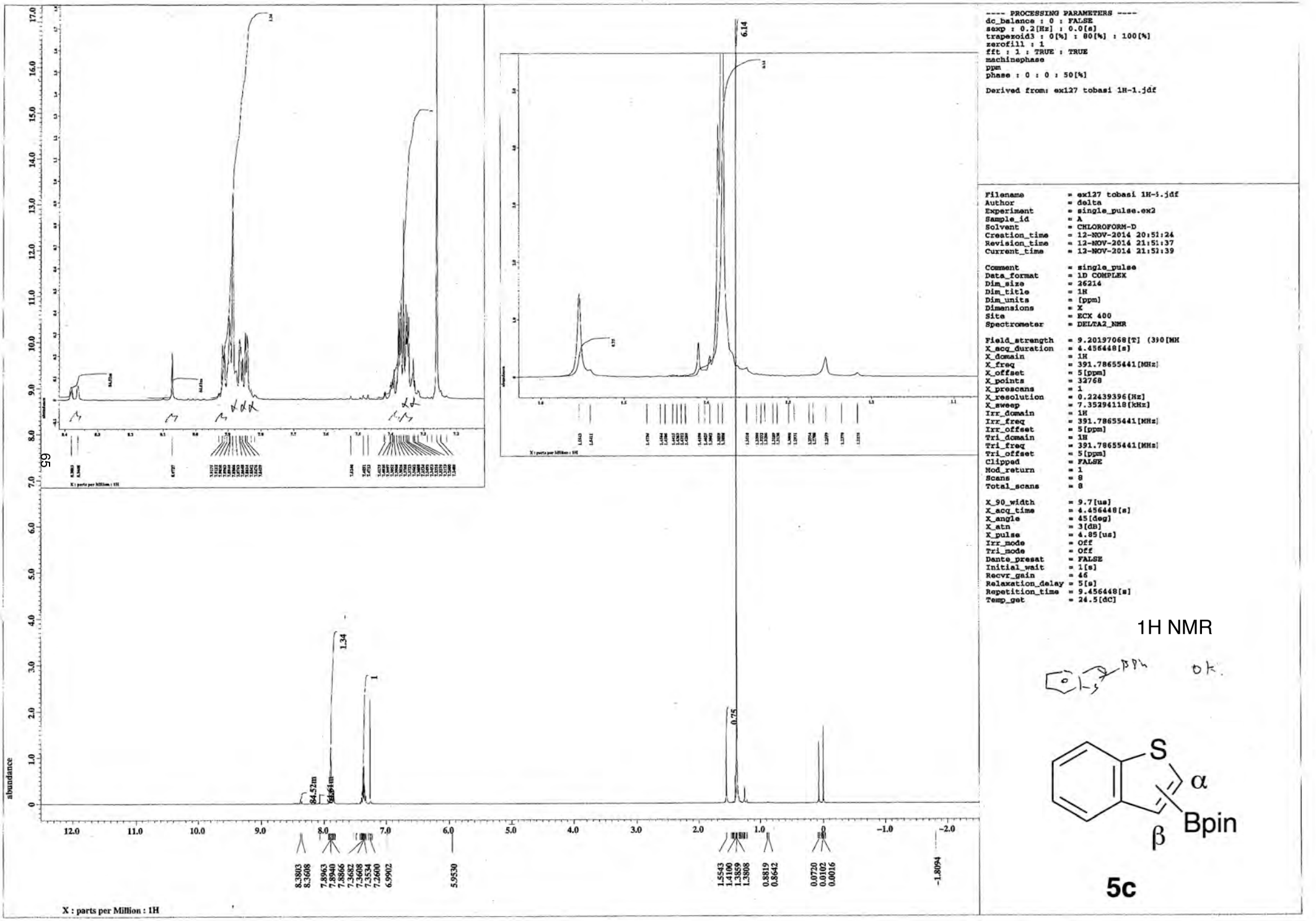

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]

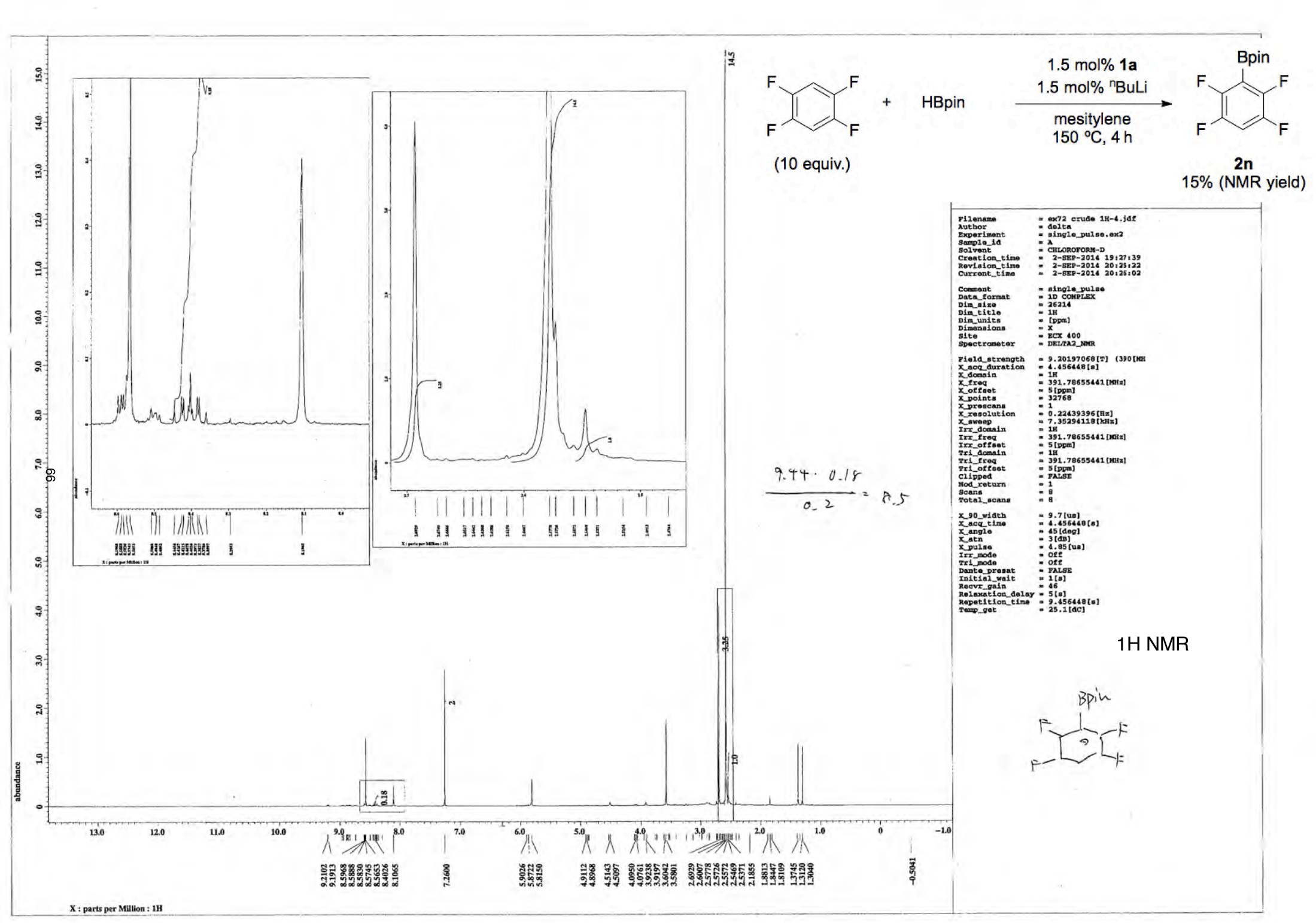
Derived from: ex135 columnn 1H-6.jdf
```

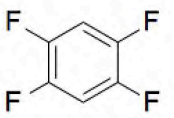
Filename	= ex135 columnn 1H-10.j
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 28-NOV-2014 15:51:35
Revision_time	= 28-NOV-2014 16:52:31
Current_time	= 28-NOV-2014 16:53:33
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 4.456448[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 0.22439396[Hz]
X_sweep	= 7.35294118[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 9.7[us]
X_acq_time	= 4.456448[s]
X_angle	= 45[deg]
X_atn	= 3[dB]
X_pulse	= 4.85[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 44
Relaxation_delay	= 5[s]
Repetition_time	= 9.456448[s]
Temp_get	= 24.4[dC]

1H NMR

5b


---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : 0 : 0 : 50[%]
Derived from: ex127 tobasi 1H-1.jdf
Filename = ex127 tobasi 1H-1.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = A
Solvent = CHLOROFORM-D
Creation_time = 12-NOV-2014 20:51:24
Revision_time = 12-NOV-2014 21:51:37
Current_time = 13-NOV-2014 21:52:39
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 4.456448[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 0.22439396[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 9.7[us]
X_acq_time = 4.456448[s]
X_angle = 45[deg]
X_atn = 3[dB]
X_pulse = 4.85[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 9.456448[s]
Temp_get = 24.5[dC]
1H NMR
ok.
S
α
Bpin
β
5c
abundance
X : parts per Million : 1H

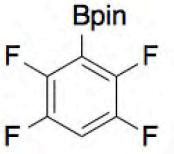

F
F
F
F
(10 equiv.)
+
HBpin
1.5 mol% 1a
1.5 mol% nBuLi
mesitylene
150 °C, 4 h
Bpin
F
F
F
F
2n
15% (NMR yield)
9.44 · 0.18 / 0.2 = 8.5
Filename = ex72 crude 1H-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = A
Solvent = CHLOROFORM-D
Creation_time = 2-SEP-2014 19:27:39
Revision_time = 2-SEP-2014 20:25:22
Current_time = 2-SEP-2014 20:25:02
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 400
Spectrometer = DELTA2_NMR
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 4.456448[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 32768
X_prescans = 1
X_resolution = 0.22439396[Hz]
X_sweep = 7.35294118[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 9.7[us]
X_acq_time = 4.456448[s]
X_angle = 45[deg]
X_atn = 3[dB]
X_pulse = 4.85[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 9.456448[s]
Temp_get = 25.1[dC]
1H NMR
Bpin
F
F
F
F
abundance
X : parts per Million : 1H



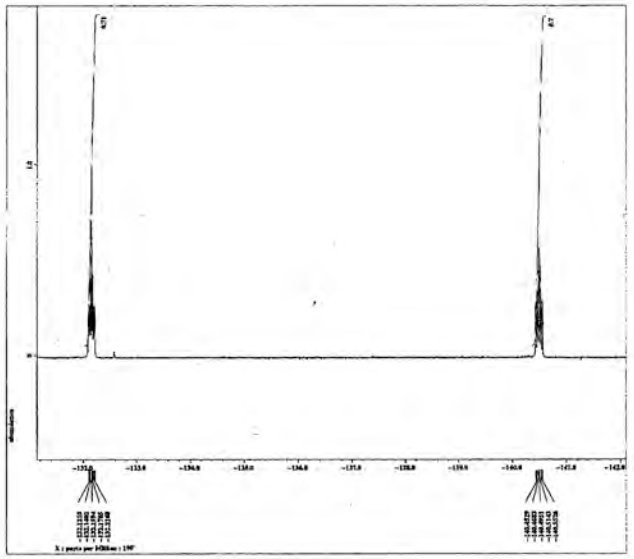
(10 equiv.)

+ HBpin

1.5 mol% **1a**
1.5 mol% ^{n}BuLi
mesitylene
150 °C, 4 h



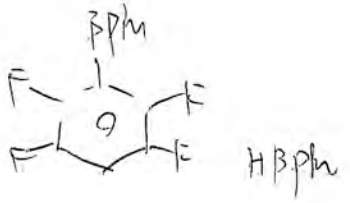
2n
15% (NMR yield)



$$\frac{\frac{0.7}{2} \cdot 8.622 \cdot 10^{-2}}{0.2} = 15\%$$

Filename	= ex72 crude 19F-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= A
Solvent	= CHLOROFORM-D
Creation_time	= 2-SEP-2014 19:23:37
Revision_time	= 2-SEP-2014 20:20:21
Current_time	= 2-SEP-2014 20:20:25
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 19F
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 0.7077888[s]
X_domain	= 19F
X_freq	= 368.64763285[MHz]
X_offset	= -140[ppm]
X_points	= 32768
X_prescans	= 1
X_resolution	= 1.41285084[Hz]
X_sweep	= 46.2962963[kHz]
Irr_domain	= 19F
Irr_freq	= 368.64763285[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 19F
Tri_freq	= 368.64763285[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 28
Total_scans	= 28
X_90_width	= 11.5[us]
X_acq_time	= 0.7077888[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 5.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 54
Relaxation_delay	= 5[s]
Repetition_time	= 5.7077888[s]
Temp_get	= 25.1[dC]

19F NMR

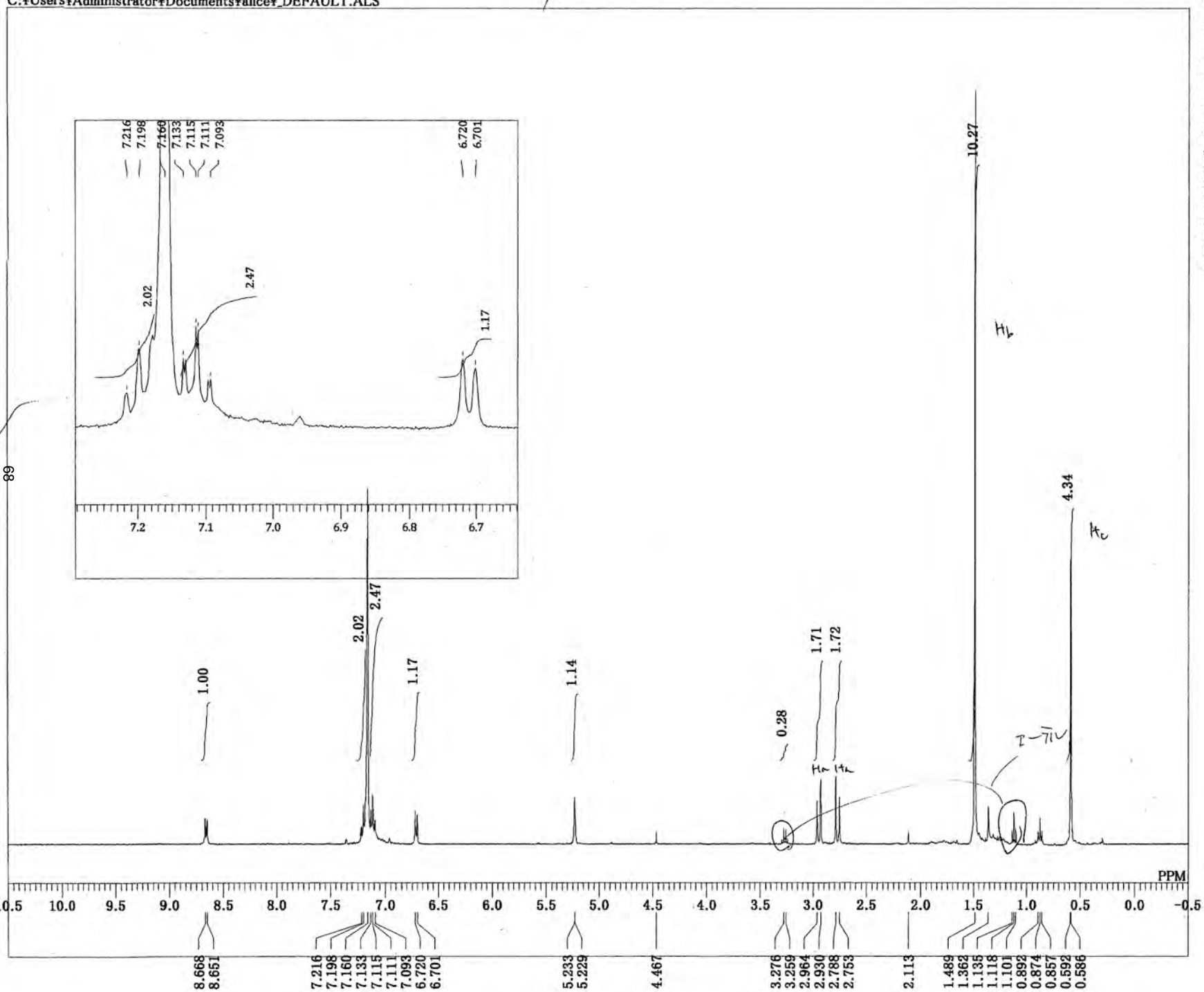


auto

C:¥Users¥Administrator¥Documents¥alice¥_DEFAULT.ALS

Ito ex3.

DFILE	_DEFAULT.ALS
COMNT	auto
DATIM	Mon Apr 21 19:18:29 :
OBNUC	1H
EXMOD	NON
OBFRQ	399.65 MHz
OBSET	124.00 KHz
OBFIN	10500.00 Hz
POINT	16384
FREQU	7992.01 Hz
SCANS	16
ACQTM	2.0500 sec
PD	1.5000 sec
PW1	6.80 usec
IRNUC	1H
CTEMP	26.9 c
SLVNT	C6D6
EXREF	7.16 ppm
BF	0.12 Hz
RGAIN	19

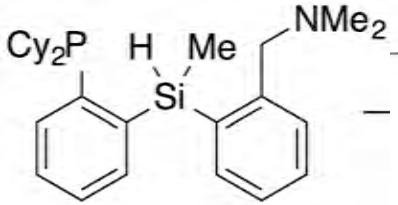


exp 3
の H NMR.

1H NMR

7

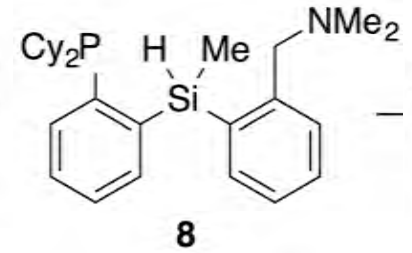
1H NMR


Cy2P
H
Me
NMe2
Si

8

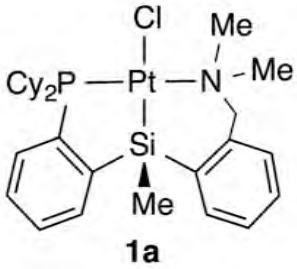
69
abundance
X : parts per Million : 1H
1.25
1.04
1.74
1.32
0.46
0.12
0.13
0.12
7.771
7.711
7.692
7.531
7.434
7.415
7.276
7.250
7.210
7.192
7.174
5.884
5.875
5.866
3.912
3.880
3.413
3.381
2.145
1.933
1.803
1.764
1.725
1.645
1.290
1.252
1.239
1.208
1.136
0.923
0.914

No title



8

13C NMR



1a

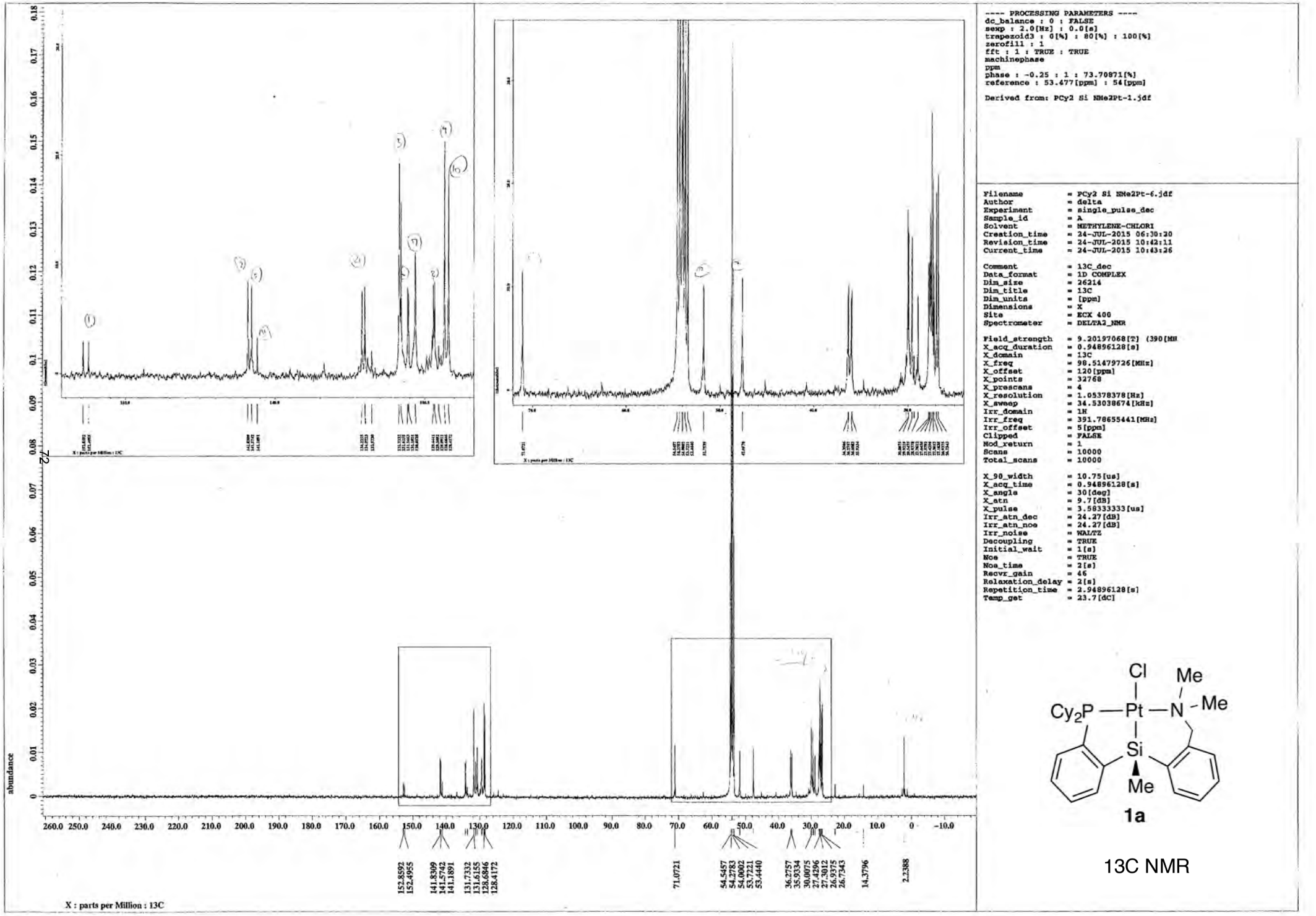
1H NMR

Current Data Parameters
NAME Shisei
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150630
Time 23.00
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 159996
SOLVENT CDCl3
NS 16
DS 2
SWH 20000.000 Hz
FIDRES 0.125003 Hz
AQ 3.9999750 sec
RG 143.7
DW 25.000 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -3.50 dB
SFO1 500.1274993 MHz

F2 - Processing parameters
SI 32768
SF 500.1300538 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
phase : -0.25 : 1 : 73.70871[%]
reference : 53.477[ppm] : 54[ppm]

Derived from: PCy2 Si NMe2Pt-1.jdf
```

Filename	= PCy2 Si NMe2Pt-6.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= A
Solvent	= METHYLENE-CHLORI
Creation_time	= 24-JUL-2015 06:30:30
Revision_time	= 24-JUL-2015 10:42:11
Current_time	= 24-JUL-2015 10:43:26
Comment	= 13C_dec
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400
Spectrometer	= DELTA2_NMR
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 0.94896128[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 120[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.05378378[Hz]
X_sweep	= 34.53038674[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 10000
Total_scans	= 10000
X_90_width	= 10.75[us]
X_acq_time	= 0.94896128[s]
X_angle	= 30[deg]
X_atn	= 9.7[dB]
X_pulse	= 3.58333333[us]
Irr_atn_dec	= 24.27[dB]
Irr_atn_noe	= 24.27[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 46
Relaxation_delay	= 2[s]
Repetition_time	= 2.94896128[s]
Temp_get	= 23.7[dC]

1a

13C NMR