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C-F Bond Formation with Fluoride Anion-Highly Selective Iodofluorination of Simple Allenes

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Supporting Information

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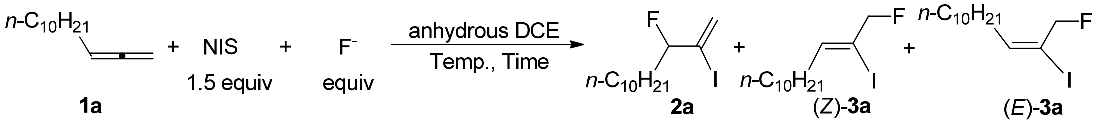
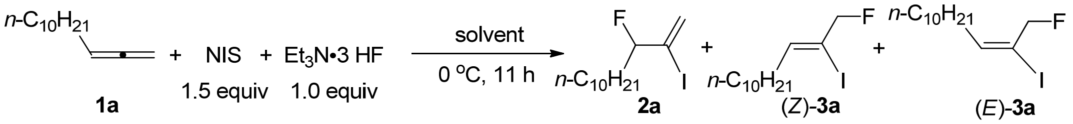
Table S1 Optimization of Reaction Conditions for the Iodofluorination of Trideca-1,2-diene **1a** with Nucleophilic Fluoride Anion.[a]



Entry	F^- (equiv)	Temp/ Time (°C/h)	Yield of **2a**+**3a** (%)[b]	**2a**: (*Z*)-**3a**: (*E*)-**3a**[c]	Recovery of **1a** (%)[b]
1	NH_4F (3.0)	0/18	0	N/A	36
2	$(NH_4)HF_2$ (1.5)	0/18	0	N/A	37
3	$NaHF_2$ (1.5)	0/18	0	N/A	65
4	KHF_2 (1.5)	0/18	0	N/A	66
5	Py·9HF (1.0)	0/15	53	88:8:4	0
6	$Et_3N\cdot 3HF$ (1.0)	0/15	58	90:9:1	0
7	$Et_3N\cdot 3HF$ (2.0)	0/5	60	91:8:1	0
8	$Et_3N\cdot 3HF$ (3.0)	0/5	58	90:9:1	0
9	$Et_3N\cdot 3HF$ (4.0)	0/5	55	90:9:1	0
10	$Et_3N\cdot 3HF$ (1.0)	-40/26	21	90:9:1	38
11	$Et_3N\cdot 3HF$ (1.0)	-20/26	35	91:8:1	27
12	$Et_3N\cdot 3HF$ (1.0)	rt/5	61	88:10:2	0

[a] Reactions were performed with 0.3 mmol of **1a**, 0.45 mmol of NIS, and nucleophilic fluoride sources under the given reaction conditions. [b] The yields were determined by the analysis of ^1H NMR spectra of crude products using mesitylene as the internal standard. [c] The ratios were determined by the analysis of ^{19}F NMR spectra of crude products.

Table S2 Effect of Solvent on the Iodofluorination of Trideca-1,2-diene **1a** with $Et_3N{\cdot}3HF$.[a]



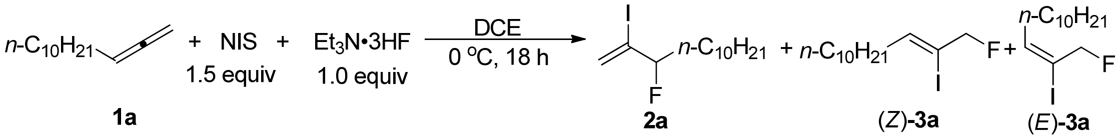
Entry	Solvent	NMR yield of **2a**+**3a** (%)[b]	**2a**: (*Z*)-**3a**: (*E*)-**3a**[c]	Recovery of **1a** (%)[b]
1	DMF	0	N/A	58
2	toluene	3	91:9:0	56
3	acetone	4	97:3:0	40
4	THF	6	95:5:0	23
5	dioxane	9	79:20:1	3
6[d]	CH_3CN	23	90:9:1	0
7	CH_3NO_2	28	86:12:2	0
8	CH_2Cl_2	57	91:8:1	0
9	$CHCl_3$	61	88:10:2	0

[a] Reactions were performed with 0.3 mmol of **1a**, 0.45 mmol of NIS, and 0.3 mmol of $Et_3N{\cdot}3HF$ under the given reaction conditions. [b] The yields were determined by the analysis of ^1H NMR spectra of crude products using mesitylene as the internal standard. [c] The ratios were determined by the analysis of ^{19}F NMR spectra of crude products. [d] The reaction time is 12 h.

General: Simple allenes[1,2] were prepared according to the literature procedures. DCE was distilled from CaH_2. Et_3N was distilled from KOH. The other commercially available chemicals were purchased and used without additional purification. The reactions were performed under an atmosphere of nitrogen using standard Schlenk tubes unless otherwise. Petroleum ether with a boiling point range of 30~60 °C was used. Flash-column chromatography was carried out on silica gel H (10–40 μ). ^1H, ^{13}C, and ^{19}F NMR spectra were recorded with a Bruker AN 300 MHz spectrometer. ^1H NMR spectra (300 MHz and 600 MHz) were recorded using TMS as an internal standard (δ 0 ppm). ^{13}C NMR spectra (75 MHz) were recorded using $CDCl_3$ as an internal standard (δ 77.00 ppm). ^{19}F NMR spectra (282 MHz) were recorded using $CFCl_3$ as an internal standard (δ 0 ppm). IR spectra were recorded with a Perkin-Elmer 983G instrument. Mass spectrometry was performed with an HP 5989A system. High-resolution mass spectrometry was determined with a Finnigan MAT 8430 or Bruker APEXIII instrument.

Iodofluorination Reactions of Simple Allenes with Nucleophilic Fluorides (Et_3N·3HF or Py·9HF)

1. 3-Fluoro-2-iodotridec-1-ene **(2a),** (*Z*)-1-fluoro-2-iodotridec-2-ene **((*Z*)-3a)**, and (*E*)-1-fluoro-2-iodotridec-2-ene **((*E*)-3a)** (xc-4-157)



Combined yield of **2a** +**3a**: 71%
Ratio of **2a** : (*Z*)-**3a**: (*E*)-**3a** = 87:11:2

Typical Procedure I: To a dried flask were added trideca-1,2-diene **1a** (1.0812 g, 6.01 mmol) and 20 mL of anhydrous 1,2-dichloroethane (DCE) at room temperature under N_2 atmosphere. The resulting mixture was then cooled to 0 °C with a cooling bath. After that *N*-iodosuccinimide (NIS) (2.0271 g, 9.01 mmol) and triethylamine trihydrofluoride ($Et_3N{\cdot}3HF$) (1.0 mL, 0.989 g/mL, 0.9890 g, 6.14 mmol) were added sequentially at 0 °C with 20 mL of DCE washing the vessel. After 18 h the reaction was complete as monitored by TLC, the mixture was quenched with 20 mL each of H_2O, sat. aq. $Na_2S_2O_3$ and aq. $NaHCO_3$, extracted with diethyl ether (20 mL×5), washed with brine and dried over anhydrous Na_2SO_4. After filtration and concentration, the ratio of **2a**/(*Z*)-**3a**/(*E*)-**3a** was 91:8:1 as determined by ^{19}F NMR analysis. Column chromatography on silica gel (eluent: petroleum ether) afforded a mixture of **2a** and **3a** (1.3837 g, 71%, ratio of **2a**/(*Z*)-**3a**/(*E*)-**3a** was 87:11:2) as a liquid.

2a: ^1H NMR (300 MHz, $CDCl_3$) δ 6.44-6.40 (m, 1 H, one proton of $=CH_2$), 5.98-5.94 (m, 1 H, one proton of $=CH_2$), 4.50 (dt, J_1 = 47.9 Hz, J_2 = 6.2 Hz, 1 H, CH), 1.86-1.66 (m, 2 H, CH_2), 1.40-1.20 (m, 16 H, 8 × CH_2), 0.88 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 127.0 (d, J = 9.1 Hz), 109.4 (d, J = 21.2 Hz), 96.2 (d, J = 177.8 Hz), 34.4 (d, J = 21.6 Hz), 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 24.2 (d, J = 4.4 Hz), 22.7, 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -165.7 (dt, J_1 = 47.6 Hz, J_2 = 21.6 Hz, 1 F); IR (neat) ν (cm^{-1}) 2923, 2854, 1615, 1465, 1413, 1378, 1169, 1060; GC-MS (EI, 70 ev) *m*/*z* (%) GC peak No. 1 (retention time: 7.3 min) 326 (M^+, 1.74), 186 (100); GC peak No. 2 (retention time: 7.4 min) 326 (M^+, 17.15), 43 (100);

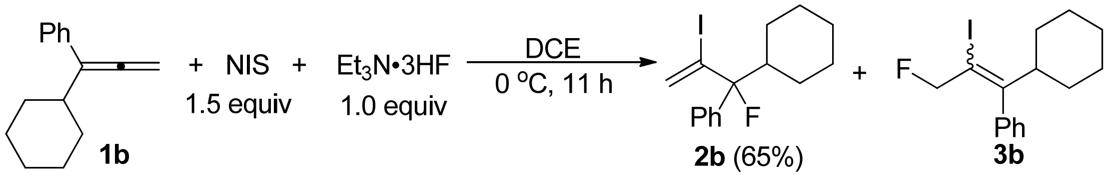
GC peak No. 3 (retention time: 7.5 min) 326 (M^+, 20.26), 43 (100); Elemental analysis calcd for $C_{13}H_{24}FI$: C, 47.86; H, 7.42; Found: C, 47.54; H, 7.28.

The following signals are discernible for (*E*)-**3a:** ^1H NMR (600 MHz, $CDCl_3$) δ 6.49 (td, J_1 = 11.4 Hz, J_2 = 3.0 Hz, 1 H, =CH), 4.98 (d, *J* = 47.4 Hz, 2 H, CH_2), 2.22-2.12 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.5 (d, *J* = 11.6 Hz), 83.3 (d, *J* = 169.3 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -201.7 ~ -202.3 (m, 1 F).

The following signals are discernible for (*Z*)-**3a:** ^1H NMR (600 MHz, $CDCl_3$) δ 6.02-5.98 (m, 1 H, =CH), 4.92 (d, *J* = 47.4 Hz, 2 H, CH_2), 2.22-2.12 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) 139.8 (d, *J* = 11.6 Hz), 99.6 (d, *J* = 16.4 Hz), 88.9 (d, *J* = 174.8 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -198.2 ~ -198.6 (m, 1 F).

The configuration of the C=C bond in **3a** was established by the NOE study (see page S42 in this document).

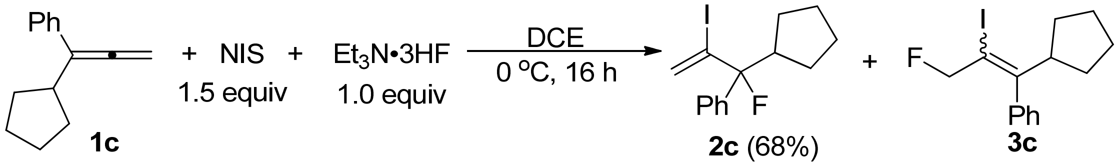
2. 3-Cyclohexyl-3-fluoro-2-iodo-3-phenylprop-1-ene **(2b)** (xc-4-194, 193)



Typical Procedure II: To a dried flask were added 1-cyclohexyl-1-phenylallene **1b** (0.1179 g, 0.60 mmol) and 1 mL of anhydrous 1,2-dichloroethane (DCE) at room temperature under N_2 atmosphere. The resulting mixture was then cooled to 0 °C with a cooling bath. After that *N*-iodosuccinimide (NIS) (0.2023 g, 0.90 mmol) and triethylamine trihydrofluoride ($Et_3N·3HF$) (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) were added sequentially at 0 °C with 1 mL of DCE washing the vessel. After

11 h the reaction was complete as monitored by TLC, the mixture was quenched with 5 mL each of H_2O, saturated aqueous $Na_2S_2O_3$ and Na_2CO_3, extracted with diethyl ether (10 mL×3), washed with brine, and dried over anhydrous Na_2SO_4. After filtration and concentration, the ratio of **2b**/**3b** was 99:1 as determined by ^{19}F NMR analysis. Column chromatography on silica gel (eluent: petroleum ether) afforded **2b** (0.1330 g, 65%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.38 (m, 2 H, ArH), 7.38-7.23 (m, 3 H, ArH), 6.59 (d, J = 1.8 Hz, 1 H, one proton of =CH_2), 5.93 (dd, J_1 = 3.5 Hz, J_2 = 2.0 Hz, 1 H, one proton of =CH_2), 2.48-2.24 (m, 1 H, CH), 1.97-1.78 (m, 2 H, CH_2), 1.75-1.60 (m, 2 H, CH_2), 1.52-1.02 (m, 6 H, 3 × CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.2 (d, J = 23.9 Hz), 127.9 (d, J = 1.6 Hz), 127.6 (d, J = 1.6 Hz), 126.0 (d, J = 9.7 Hz), 125.2 (d, J = 11.9 Hz), 114.0 (d, J = 27.0 Hz), 100.6 (d, J = 187.4 Hz), 42.6 (d, J = 21.1 Hz), 26.8 (d, J = 2.9 Hz), 26.5, 26.3 (d, J = 2.8 Hz), 26.2; ^{19}F NMR (282 MHz, $CDCl_3$) δ -157.9 (d, J = 30.2 Hz, 1 F); IR (neat) ν (cm^{-1}) 3090, 3059, 3027, 2933, 2852, 1606, 1493, 1447, 1402, 1332, 1245, 1218, 1137, 1069; MS (70 ev, EI) *m/z* (%) 344 (M^+, 23.37), 134 (100); HRMS calcd for $C_{15}H_{18}FI$ (M^+): 344.0437, Found: 344.0443.

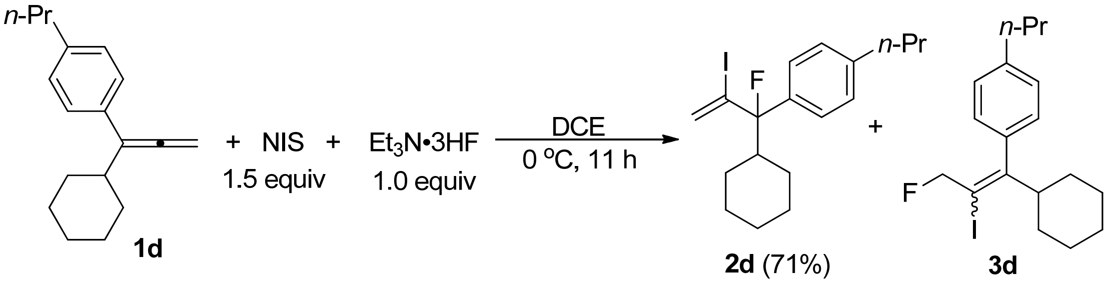
3. 3-Cyclopentyl-3-fluoro-2-iodo-3-phenylprop-1-ene **(2c)** (xc-5-042)



Following Typical Procedure I: The reaction of 1-cyclopentyl-1-phenylallene **1c** (1.1048 g, 6.00 mmol) with NIS (2.0259 g, 9.00 mmol) and $Et_3N \cdot 3HF$ (1.0 mL, 0.989 g/mL, 0.989 g, 6.14 mmol) in DCE (5 mL) at 0 °C for 16 h afforded a crude product. The ratio of **2c**/**3c** was 99:1 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2c** (1.3375 g, 68%) as a liquid: ^1H NMR (300 MHz, $CDCl_3$) δ 7.47-7.38 (m, 2 H, ArH), 7.38-7.27 (m, 3 H, ArH), 6.62 (d, J = 2.1 Hz, 1 H, one proton of $=CH_2$), 5.94 (t, J = 2.4 Hz, 1 H, one proton of $=CH_2$), 3.20-2.92 (m, 1 H, CH), 1.95-1.35 (m, 8 H, 4 × CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.4 (d, J = 23.4 Hz), 127.8, 127.7, 125.8 (d, J = 8.7 Hz), 124.9 (d, J = 9.8 Hz), 114.7 (d, J = 28.0 Hz), 100.6 (d, J = 185.5 Hz), 44.2 (d, J = 22.1 Hz), 27.8 (d, J = 2.4 Hz), 26.7 (d, J = 2.8 Hz), 26.3 (d, J = 5.4 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -159.8 (d, J = 34.1 Hz, 1 F); IR (neat) ν (cm^{-1}) 3058, 3027, 2954, 2868, 1603, 1493, 1450, 1368, 1270, 1241, 1157, 1091; MS (70 ev, EI) *m/z* (%) 330 (M^+, 18.47), 134 (100); HRMS calcd for $C_{14}H_{16}FI$ (M^+): 330.0281, Found: 330.0291.

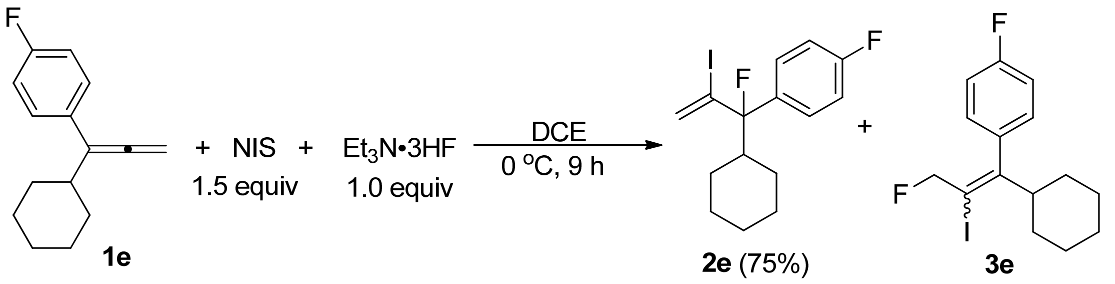
4. 3-Cyclohexyl-3-fluoro-2-iodo-3-(4'-propylphenyl)prop-1-ene **(2d)**

(xc-5-065,066)



Following Typical Procedure I: The reaction of 1-cyclohexyl-1-(4'-propylphenyl) allene **1d** (1.4419 g, 6.01 mmol) with NIS (2.0259 g, 9.00 mmol) and $Et_3N{\cdot}3HF$ (1.0 mL, 0.989 g/mL, 0.9890 g, 6.14 mmol) in DCE (5 mL) at 0 °C for 11 h afforded a crude product. The ratio of **2d**/**3d** was 99:1 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2d** (1.6440 g, 71%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (d, J = 8.1 Hz, 2 H, ArH), 7.14 (d, J = 8.1 Hz, 2 H, ArH), 6.58 (d, J = 1.8 Hz, 1 H, one proton of $=CH_2$), 5.91 (dd, J_1 = 3.3 Hz, J_2 = 1.8 Hz, 1 H, one proton of $=CH_2$), 2.58 (t, J = 7.7 Hz, 2 H, CH_2), 2.45-2.25 (m, 1 H, CH), 1.96-1.80 (m, 2 H, CH_2), 1.75-1.58 (m, 4 H, 2 × CH_2), 1.53-1.43 (m, 1 H, one proton of CH_2), 1.36-1.06 (m, 5 H, one proton of CH_2 and 2 × CH_2), 0.94 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.1 (d, J = 1.5 Hz), 136.4 (d, J = 23.6 Hz), 128.0 (d, J = 1.5 Hz), 125.9 (d, J = 8.8 Hz), 124.9 (d, J = 11.9 Hz), 114.4 (d, J = 26.9 Hz), 100.7 (d, J = 186.8 Hz), 42.6 (d, J = 20.2 Hz), 37.7, 26.9 (d, J = 2.9 Hz), 26.5, 26.3, 26.2, 24.3, 13.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -157.7 (d, J = 29.6 Hz, 1 F); IR (neat) ν (cm^{-1}) 3092, 3027, 2930, 2852, 2669, 1741, 1606, 1512, 1452, 1412, 1378, 1300, 1245, 1221, 1182, 1103, 1093, 1079; MS (70 ev, EI) *m/z* (%) 176 (100), 386 (M^+, 41.40); HRMS calcd for $C_{18}H_{24}FI$ (M^+): 386.0907, Found: 386.0912.

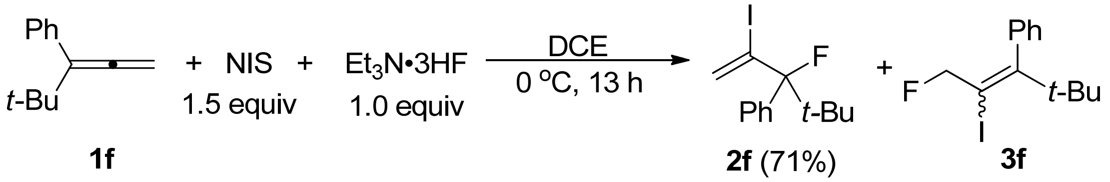
5. 3-Cyclohexyl-3-fluoro-2-iodo-3-(4'-fluorophenyl)prop-1-ene **(2e)** (xc-5-146)



Following Typical Procedure I: The reaction of 1-cyclohexyl-1-(4’-fluorophenyl)allene **1e** (1.2972 g, 6.01 mmol) with NIS (2.0271 g, 9.01 mmol) and $Et_3N{\cdot}3HF$ (1.0 mL, 0.989 g/mL, 0.9890 g, 6.14 mmol) in DCE (5 mL) at 0 ^{o}C for 9 h afforded a crude product. The ratio of **2e**/**3e** was 99:1 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2e** (1.6301 g, 75%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.33 (m, 2 H, ArH), 7.09-6.95 (t, J = 8.6 Hz, 2 H, ArH), 6.58 (d, J = 2.1 Hz, 1 H, one proton of $=CH_2$), 5.92 (dd, J_1 = 3.3 Hz, J_2 = 1.8 Hz, 1 H, one proton of $=CH_2$), 2.46-2.20 (m, 1 H, CH), 1.96-1.78 (m, 2 H, CH_2), 1.76-1.61 (m, 2 H, CH_2), 1.49-1.38 (m, 1 H, one proton of CH_2), 1.36-1.01 (m, 5 H, one proton of CH_2 and 2 × CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.1 (dd, J_1 = 245.4 Hz, J_2 = 1.2 Hz), 135.0 (dd, J_1 = 23.9 Hz, J_2 = 3.2 Hz), 127.9 (dd, J_1 = 9.7 Hz, J_2 = 8.3 Hz), 125.2 (d, J = 11.0 Hz), 114.8 (d, J = 21.4 Hz), 113.8 (d, J = 26.8 Hz), 100.4 (d, J = 188.3 Hz), 42.5 (d, J = 21.1 Hz), 26.7 (d, J = 3.1 Hz), 26.4, 26.2 (d, J = 2.4 Hz), 26.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -114.4~-114.6 (m, 1 F), -157.4 (d, J = 29.9 Hz, 1 F); IR (neat) ν (cm^{-1}) 3042, 2927, 2852, 1682, 1600, 1505, 1474, 1448, 1399, 1349, 1263, 1223, 1158, 1092, 1015; MS (70 ev, EI) *m/z* (%) 362 (M^{+}, 21.47), 152 (100); HRMS calcd for $C_{15}H_{17}F_2I$ (M^{+}): 362.0343, Found:

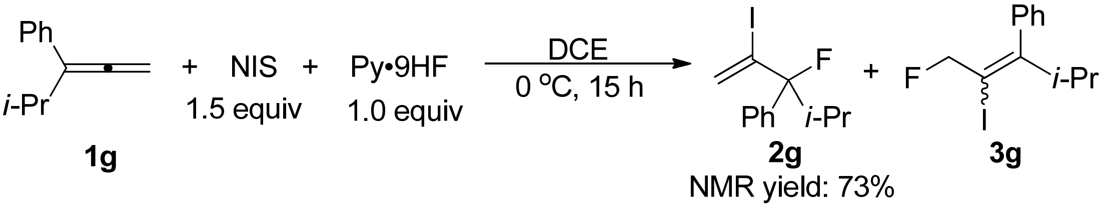
362.0340.

6. 3-Fluoro-2-iodo-4,4-dimethyl-3-phenylpent-1-ene **(2f)** (xc-5-192,6-022)



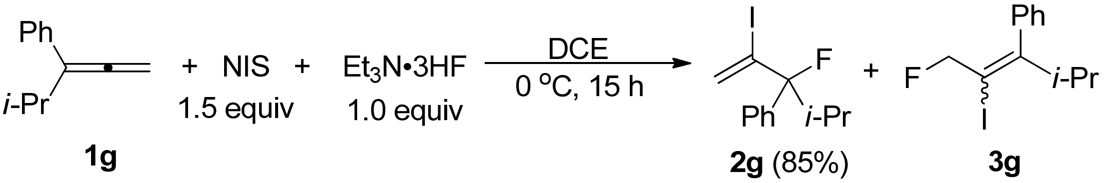
Following Typical Procedure II: The reaction of 4,4-dimethyl-3-phenylpenta-1,2-diene **1f** (0.1028 g, 0.60 mmol) with NIS (0.2031 g, 0.90 mmol) and $Et_3N{\cdot}3HF$ (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 13 h afforded a crude product. The ratio of **2f**/**3f** was > 98:2 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2f** (0.1340 g, 71%) as a liquid: ^1H NMR (300 MHz, $CDCl_3$) δ 7.45-7.27 (m, 5 H, ArH), 6.80 (t, *J* = 2.9 Hz, 1 H, one proton of $=CH_2$), 6.34 (d, *J* = 2.7 Hz, 1 H, one proton of $=CH_2$), 1.14 (d, *J* = 1.5 Hz, 9 H, 3 × CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.6 (d, *J* = 23.2 Hz), 128.9 (d, *J* = 7.3 Hz), 127.7, 127.2 (d, *J* = 1.4 Hz), 126.8 (d, *J* = 9.7 Hz), 114.7 (d, *J* = 28.6 Hz), 102.7 (d, *J* = 181.7 Hz), 38.6 (d, *J* = 24.4 Hz), 27.1 (d, *J* = 5.0 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -132.8 (s, 1 F); IR (neat) ν (cm^{-1}) 3090, 3059, 3027, 2988, 2939, 2913, 2878, 1615, 1492, 1482, 1465, 1447, 1395, 1370, 1222, 1193, 1134, 1045; MS (70 ev, EI) m/z (%) 318 (M^+, 6.37), 57 (100); HRMS calcd for $C_{13}H_{16}FI$ (M^+): 318.0281, Found: 318.0279.

7. 3-Fluoro-2-iodo-4-methyl-3-phenylpent-1-ene **(2g)** (xc-6-035)



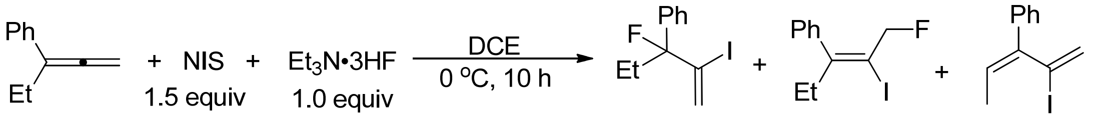
Following Typical Procedure II: The reaction of 4-methyl-3-phenylpenta-1,2-diene **1g** (0.0949 g, 0.60 mmol) with NIS (0.2029 g, 0.90 mmol) and Olah's Reagent (Py·9HF)[3,4] (0.06 mL, 1.100 g/mL, 0.0660 g, 0.67 mmol) in DCE (2 mL) at 0 °C for 15 h afforded **2g** and **3g**. The NMR yield of **2g** was 73% as determined by ^{1}H NMR analysis using mesitylene as the internal standard and the ratio of **2g**/**3g** was 99:1 as determined by ^{19}F NMR analysis of the crude product.

8. 3-Fluoro-2-iodo-4-methyl-3-phenylpent-1-ene **(2g)** (xc-6-034)



Following Typical Procedure II: The reaction of 4-methyl-3-phenylpenta-1,2-diene **1g** (0.0948 g, 0.60 mmol) with NIS (0.2027 g, 0.90 mmol) and $Et_3N{\cdot}3HF$ (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 15 h afforded a crude product. The ratio of **2g**/**3g** was 98:2 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2g** (0.1554 g, 85%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49-7.39 (m, 2 H, ArH), 7.38-7.24 (m, 3 H, ArH), 6.61 (d, J = 1.8 Hz, 1 H, one proton of $=CH_2$), 5.92 (dd, J_1 = 3.5 Hz, J_2 = 2.0 Hz, 1 H, one proton of $=CH_2$), 2.91-2.64 (m, 1 H, CH), 1.08 (d, J = 6.6 Hz, 3 H, CH_3), 0.86 (d, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.5 (d, J = 22.6 Hz), 127.9, 127.7, 125.9 (d, J = 9.2 Hz), 125.1 (d, J = 10.7 Hz), 114.2 (d, J = 27.3 Hz), 100.8 (d, J = 187.4 Hz), 32.8 (d, J = 21.9 Hz), 16.9 (d, J = 3.8 Hz), 16.3 (d, J = 3.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -162.8 (d, J = 31.3 Hz, 1 F); IR (neat) ν (cm^{-1}) 3089, 3060, 3027, 2974, 2935, 2877, 1607, 1493, 1462, 1447, 1388, 1369, 1219, 1179, 1136, 1099, 1076, 1032, 1003; MS (70 ev, EI) *m/z* (%) 304 (M$^+$, 68.83), 134 (100); HRMS calcd for $C_{12}H_{14}FI$ (M$^+$): 304.0124, Found: 304.0126.

9. 3-Fluoro-2-iodo-3-phenylpent-1-ene **(2h)**, 1-fluoro-2-iodo-3-phenylpent-1-ene (**3h**), and (*Z*)-2-iodo-3-phenylpenta-1,3-diene ((*Z*)-**5h**) (xc-4-064, 4-117, 8-168)



1h **2h** (67%) (*E*)-**3h** (4%) (*Z*)-**5h** (9%)

Following Typical Procedure I: The reaction of 3-phenylpenta-1,2-diene **1h** (0.4327 g, 3.00 mmol) with NIS (1.0137 g, 4.51 mmol) and $Et_3N{\cdot}3HF$ (0.5 mL, 0.989 g/mL, 0.4945 g, 3.07 mmol) in DCE (5 mL) at 0 °C for 10 h afforded a crude product. The ratio of **2h**/**3h** was 94:6 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2h** (0.5832 g, 67%), **3h** (0.0554 g, purity = 68% based on the ^{1}H NMR analysis with mesitylene as the internal standard, 4% from **1h**), and

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(*Z*)-**5h** (0.0698 g, 9%).

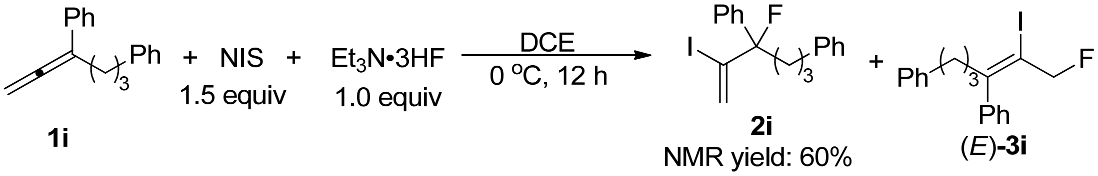
2h (more polar, liquid): ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.46-7.25 (m, 5 H, ArH), 6.51 (t, *J* = 1.5 Hz, 1 H, one proton of =CH$_2$), 6.04 (t, *J* = 1.8 Hz, 1 H, one proton of =CH$_2$), 2.55-2.33 (m, 1 H, one proton of CH$_2$), 2.32-2.10 (m, 1 H, one proton of CH$_2$), 0.94 (t, *J* = 7.2 Hz, 3 H, CH$_3$); ^{13}C NMR (75 MHz, CDCl$_3$) δ 139.7 (d, *J* = 22.8 Hz), 128.1, 126.6 (d, *J* = 9.2 Hz), 125.7, 125.6, 114.0 (d, *J* = 27.3 Hz), 99.5 (d, *J* = 181.9 Hz), 30.2 (d, *J* = 23.5 Hz), 7.7 (d, *J* = 3.8 Hz); ^{19}F NMR (282 MHz, CDCl$_3$) δ -145.6 (t, *J* = 23.0 Hz, 1 F); IR (neat) ν (cm^{-1}) 3090, 3061, 3030, 2978, 2938, 2882, 1609, 1494, 1448, 1407, 1380, 1292, 1219, 1158, 1124, 1078, 1065; MS (70 ev, EI) *m/z* (%) 290 (M$^+$, 90.58), 134 (100); HRMS calcd for $C_{11}H_{12}FI$ (M$^+$): 289.9968, Found: 289.9966.

The following signals are discernible for (*E*)-**3h** (most polar, liquid): ^{1}H NMR (300 MHz, CDCl$_3$) δ 4.86 (d, *J* = 47.7 Hz, 2 H, CH$_2$), 2.72-2.60 (m, 2 H, CH$_2$), 0.94 (t, *J* = 7.5 Hz, 3 H, CH$_3$); ^{13}C NMR (75 MHz, CDCl$_3$) δ 155.4 (d, *J* = 8.2 Hz), 138.4 (d, *J* = 3.0 Hz), 128.4, 127.9 (d, *J* = 2.0 Hz), 127.8, 96.5 (d, *J* = 17.2 Hz), 87.1 (d, *J* = 166.8 Hz), 37.8 (d, *J* = 1.1 Hz), 11.2 (d, *J* = 4.0 Hz); ^{19}F NMR (282 MHz, CDCl$_3$) δ -197.5 (tt, J_1 = 47.6 Hz, J_2 = 3.5 Hz, 1 F); MS (70 ev, EI) *m/z* (%) 290 (M$^+$, 37.66), 134 (100); HRMS calcd for $C_{11}H_{12}FI$ (M$^+$): 289.9968, Found: 289.9970.

(*Z*)-**5h** (less polar, liquid): ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.45-7.28 (m, 5 H, ArH), 6.23 (d, *J* = 1.2 Hz, 1 H, one proton of =CH$_2$), 6.19 (d, *J* = 0.9 Hz, 1 H, one proton of =CH$_2$), 5.99 (q, *J* = 6.9 Hz, 1 H, =CH), 1.85 (d, *J* = 7.2 Hz, 3 H, CH$_3$); ^{13}C NMR (75 MHz, CDCl$_3$) δ 144.6, 138.0, 130.5, 128.3, 127.5, 126.4, 124.2, 103.6, 15.6; IR (neat) ν (cm^{-1}) 3057, 3028, 2979, 2929, 1759, 1682, 1614, 1493, 1442, 1410, 1371, 1338, 1265, 1246, 1219, 1181, 1149, 1066, 1027, 1001; MS (70 ev, EI) *m/z* (%) 270 (M^+, 14.77), 128 (100); HRMS calcd for $C_{11}H_{11}I$ (M^+): 269.9906, Found: 269.9909.

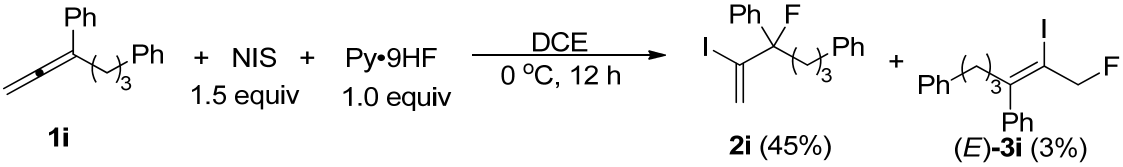
The configuration of the C=C bond in (*Z*)-**5h** was established by the NOE study (see page S84 in this document).

10. 3-Fluoro-2-iodo-3,6-diphenylhex-1-ene **(2i)** (xc-5-56)



Following Typical Procedure I: The reaction of 3,6-diphenylhexa-1,2-diene **1i** (1.4045 g, 6.00 mmol) with NIS (2.0261 g, 9.00 mmol) and $Et_3N{\cdot}3HF$ (1.0 mL, 0.989 g/mL, 0.9890 g, 6.14 mmol) in DCE (5 mL) at 0 °C for 12 h afforded **2i** and (*E*)-**3i**. The NMR yield of **2i** was 60% as determined by ^1H NMR analysis using mesitylene as the internal standard and the ratio of **2i**/(*E*)-**3i** was 91:9 as determined by ^{19}F NMR analysis of the crude product.

11. 3-Fluoro-2-iodo-3,6-diphenylhex-1-ene **(2i)** and (*E*)-1-fluoro-2-iodo-3,6-diphenylhex-2-ene **((*E*)-3i)** (xc-5-55)



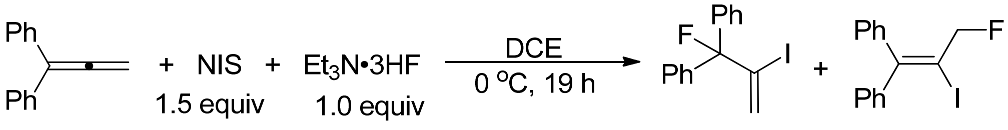
Following Typical Procedure I: The reaction of 3,6-diphenylhexa-1,2-diene **1i** (1.4042 g, 6.00 mmol) with NIS (2.0257 g, 9.00 mmol) and Py·9HF (0.6 mL, 1.100 g/mL, 0.6600 g, 6.67 mmol) in DCE (5 mL) at 0 °C for 12 h afforded a crude product. The ratio of **2i**/(*E*)-**3i** was 93:7 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2i** (1.0260 g, 45%) and (*E*)-**3i** (0.0682 g, 3%).

2i (less polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.21 (m, 7 H, ArH), 7.21-7.08 (m, 3 H, ArH), 6.44 (s, 1 H, one proton of $=CH_2$), 5.99 (t, *J* = 2.1 Hz, 1 H, one proton of $=CH_2$), 2.69-2.56 (m, 2 H, $=CH_2$), 2.51-2.05 (m, 2 H, CH_2), 1.90-1.51 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6, 139.7 (*J* = 22.7 Hz), 128.34, 128.29, 128.1, 126.6 (d, *J* = 9.3 Hz), 125.8, 125.7, 125.5, 114.0 (d, *J* = 27.2 Hz), 99.2 (d, *J* = 180.5 Hz), 36.7 (d, *J* = 22.4 Hz), 35.6, 24.9 (d, *J* = 2.9 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -143.1 (t, *J* = 23.8 Hz, 1 F); IR (neat) ν (cm^{-1}) 3085, 3061, 3026, 2955, 2855, 1610, 1495, 1448, 1404, 1312, 1229, 1211, 1156, 1062, 1030; MS (70 ev, EI) *m/z* (%) 91 (100), 380 (M$^+$, 1.80); HRMS calcd for $C_{18}H_{18}FI$ (M$^+$): 380.0437, Found: 380.0434.

(*E*)-**3i** (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.00 (m, 10 H, ArH), 4.87 (d, *J* = 47.7 Hz, 2 H, CH_2), 2.78-2.66 (m, 2 H, CH_2), 2.59 (t, *J* = 8.4 Hz, 2 H, CH_2), 1.74-1.59 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.0 (*J* = 8.2 Hz), 141.7, 138.5 (*J* = 2.3 Hz), 128.5, 128.34, 128.26, 128.0, 127.8 (d, *J*= 2.3 Hz), 125.8, 97.6 (d, *J* = 17.0 Hz), 87.1 (d, *J* = 166.9 Hz), 44.1 (d, *J* = 1.3 Hz), 35.5, 28.5 (d, *J* = 3.7 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -197.8 (tt, J_1 = 47.6 Hz, J_2= 3.5 Hz, 1 F); IR (neat) ν (cm^{-1}) 3060, 3025, 2927, 2858, 1602, 1574, 1495, 1455, 1442, 1368, 1280, 1242, 1157, 1099, 1074, 1045, 1029; MS (70 ev, EI) *m/z* (%) 91 (100), 380 (M^+, 0.60); HRMS calcd for $C_{18}H_{18}FI$ (M^+): 380.0437, Found: 380.0434.

The configuration of the C=C bond in (*E*)-**3i** was established by the NOE study (see page S94 in this document).

12. 3-Fluoro-2-iodo-3,3-diphenylprop-1-ene **(2j)** and 3-fluoro-2-iodo-1,1-diphenyl-prop-1-ene (**3j**) (xc-5-69, 8-156)



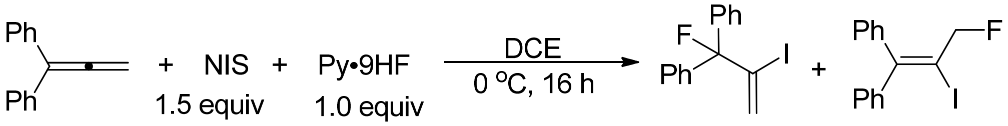
1j **2j** (72%) **3j** (15%)

Following Typical Procedure II: The reaction of 1,1-diphenylpropa-1,2-diene **1j** (0.1155 g, 0.60 mmol) with NIS (0.2027 g, 0.90 mmol) and $Et_3N{\cdot}3HF$ (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 19 h afforded a crude product. The ratio of **2j**/**3j** was 84:16 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography (low temperature column chromatography with a -18 ~ -16 °C circulating EtOH outside the column) on silica gel (eluent: petroleum ether) afforded **2j** (0.1469 g, 72%) and **3j** (0.0312 g, 15%).

2j (less polar, liquid): ^1H NMR (300 MHz, $CDCl_3$) δ 7.43-7.28 (m, 10 H, ArH), 6.25 (d, *J* = 2.4 Hz, 1 H, one proton of $=CH_2$), 5.93 (dd, $J_1 = 3.3$ Hz, $J_2 = 2.1$ Hz ,1 H, one proton of $=CH_2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4 (d, *J* = 24.3 Hz), 132.4 (d, *J* = 5.3 Hz), 128.6 (d, *J* = 1.7 Hz), 128.1, 127.4 (d, *J* = 6.7 Hz), 113.5 (d, *J* = 29.6 Hz), 101.6 (d, *J* = 176.9 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -123.3 (d, *J* = 3.4 Hz, 1 F).

3j (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.11 (m, 10 H, Ar-H), 5.13 (d, *J* = 47.7 Hz, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.8 (d, *J* = 8.7 Hz), 145.0 (d, *J* = 2.5 Hz), 139.0 (d, *J* = 2.5 Hz), 128.9 (d, *J* = 2.0 Hz), 128.6 (d, *J* = 2.8 Hz), 128.4, 128.32, 128.26, 128.1, 98.0 (d, *J* = 16.1 Hz), 87.1 (d, *J* = 167.5 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -197.2 (t, *J* = 47.4 Hz, 1 F).

3-Fluoro-2-iodo-3,3-diphenylprop-1-ene (**2j**) and 3-fluoro-2-iodo-1,1-diphenyl-prop-1-ene (**3j**) (xc-4-147)



1j **2j** (30%) **3j** (27%)

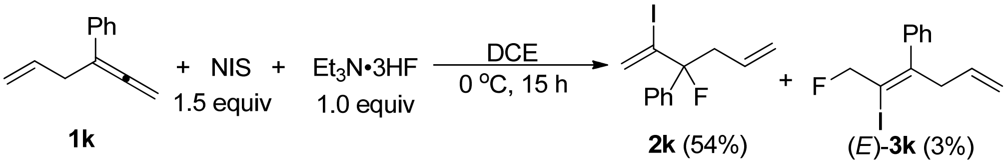
NMR Yield: 60% 5.5%

Following Typical Procedure II: The reaction of 1,1-diphenylpropa-1,2-diene **1j** (0.1147 g, 0.60 mmol) with NIS (0.2031 g, 0.90 mmol) and Py·9HF (0.06 mL, 1.100 g/mL, 0.0660 g, 0.67 mmol) in DCE (2 mL) at 0 °C for 16 h afforded a crude product. The NMR yields of **2j** (60%) and **3j** (5.5%) were determined by ^{1}H NMR analysis and the ratio of **2j**/**3j** was 84:16 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on basic alumina (eluent: petroleum ether) afforded pure **2j** (0.0608 g, 30%) and pure **3j** (0.0555 g, 27%).

2j (less polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.28 (m, 10 H, ArH), 6.25 (d, J = 1.8 Hz, 1 H, one proton of =CH_2), 5.93 (dd, J_1 = 3.0 Hz, J_2 = 2.1 Hz, 1 H, one proton of =CH_2); ^{19}F NMR (282 MHz, $CDCl_3$) δ -123.3 (d, J = 3.9 Hz, 1 F).

3j (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.27 (m, 6 H, ArH), 7.26-7.21 (m, 2 H, ArH), 7.20-7.13 (m, 2 H, ArH), 5.12 (d, J = 47.7 Hz, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.8 (d, J = 7.8 Hz), 145.0 (d, J = 1.9 Hz), 139.0 (d, J = 2.9 Hz), 128.9 (d, J = 2.1 Hz), 128.6 (d, J = 2.9 Hz), 128.4, 128.3, 128.2, 128.1, 98.0 (d, J = 16.7 Hz), 87.1 (d, J = 168.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -196.6 (t, J = 47.4 Hz, 1 F); IR (neat) ν (cm^{-1}) 3078, 3055, 3026, 1594, 1576, 1491, 1443, 1367, 1241, 1093, 1031, 1015; MS (70 ev, EI) *m/z* (%) 339 (M^++1, 17.49), 338 (M^+, 100); HRMS calcd for $C_{15}H_{12}FI$ (M^+): 337.9968, Found: 337.9964.

13. 3-Fluoro-2-iodo-3-phenylhexa-1,5-diene **(2k)** and 6-fluoro-5-iodo-4-phenyl-hexa-1,4-diene ((*E*)-**3k**) (xc-6-118, 8-160)



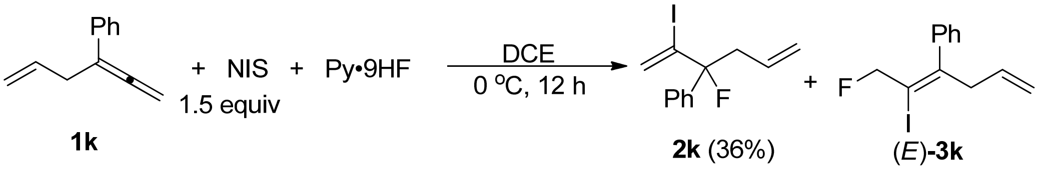
Following Typical Procedure II: The reaction of 3-phenylhexa-1,2,5-triene **1k** (0.0934 g, 0.60 mmol) with NIS (0.2028 g, 0.90 mmol) and $Et_3N·3HF$ (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 15 h afforded a crude product. The ratio of **2k**/(*E*)-**3k** was 88:12 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2k** (0.0973 g, 54%) and (*E*)-**3k** (0.0198 g, purity = 31% based on the ^{1}H NMR analysis with mesitylene as the internal standard, 3% from **1k**).

2k (less polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.25 (m, 5 H, ArH), 6.51 (s, one proton of $=CH_2$), 6.05 (t, *J* = 1.5 Hz, 1 H, one proton of $=CH_2$), 5.82-5.62 (m, 1 H, =CH), 5.23-5.05 (m, 2 H, $=CH_2$), 3.26-2.88 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4 (d, *J* = 22.6 Hz), 131.2 (d, *J* = 4.1 Hz), 128.3 (d, *J* = 1.3 Hz), 128.1, 127.1 (d, *J* = 9.4 Hz), 125.8 (d, *J* = 7.7 Hz), 119.2, 113.2 (d, *J* = 26.9 Hz), 98.3 (d, *J* = 181.9 Hz), 41.8 (d, *J* = 22.4 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -141.4 (t, *J* = 23.3 Hz, 1 F).

The following signals are discernable for (*E*)-**3k** (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.18-7.06 (m, 2 H, ArH), 4.90 (d, *J* = 47.7 Hz, 2 H, CH_2), 3.48-3.35 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.4 (d, *J* = 8.3 Hz), 86.7 (d, *J* = 167.4 Hz), 48.6; ^{19}F NMR (282 MHz, $CDCl_3$) δ -198.0 (tt, J_1 = 47.6 Hz, J_2 = 3.4 Hz, 1 F); MS (70 ev, EI) *m/z* (%) 303 (M^++1, 15.08), 302 (M^+, 100); HRMS calcd for $C_{12}H_{12}FI$ (M^+): 301.9968, Found: 301.9977.

The configuration of the C=C bond in (*E*)-**3k** was established by the NOE study (see page S114 in this document).

14. 3-Fluoro-2-iodo-3-phenylhexa-1,5-diene **(2k)** and 6-fluoro-5-iodo-4-phenylhexa-1,4-diene ((*E*)-**3k**) (xc-6-119)

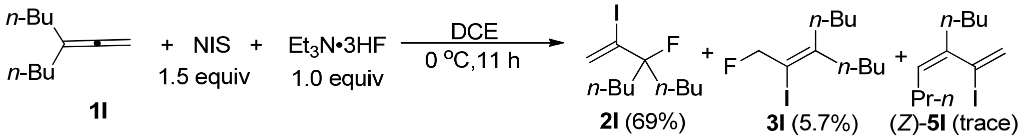


Following Typical Procedure II: The reaction of 3-phenylhexa-1,2,5-triene **1k**

(0.0935 g, 0.60 mmol) with NIS (0.2026 g, 0.90 mmol) and Py·9HF (0.06 mL, 1.100 g/mL, 0.0660 g, 0.67 mmol) in DCE (2 mL) at 0 °C for 12 h afforded a crude product. The ratio of **2k**/(*E*)-**3k** was 93:7 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2k** (0.0660 g, 36%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.30 (m, 5 H, ArH), 6.54 (dd, J_1 = 2.0 Hz, J_2 = 1.4 Hz, 1 H, one proton of $=CH_2$), 6.12-6.03 (m, 1 H, one proton of $=CH_2$), 5.85-5.67 (m, 1 H, =CH), 5.24-5.08 (m, 2 H, $=CH_2$), 3.29-2.90 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4 (d, *J* = 22.8 Hz), 131.2 (d, *J* = 3.1 Hz), 128.3 (d, *J* = 1.4 Hz), 128.2, 127.1 (d, *J* = 8.5 Hz), 125.8 (d, *J* = 7.6 Hz), 119.2, 113.2 (d, *J* = 26.6 Hz), 98.3 (d, *J* = 181.4 Hz), 41.8 (d, *J* = 24.1 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -141.4 (t, *J* = 23.4 Hz, 1 F); IR (neat) ν (cm^{-1}) 3078, 3029, 2981, 2951, 2917, 1642, 1610, 1494, 1448, 1432, 1405, 1315, 1294, 1268, 1213, 1157, 1132, 1095, 1057; MS (70 ev, EI) *m/z* (%) 302 (M^+, 2.18), 134 (100); HRMS calcd for $C_{12}H_{12}FI$ (M^+): 301.9968, Found: 301.9962.

15. 3-Butyl-3-fluoro-2-iodohept-1-ene **(2l)** and 3-butyl-1-fluoro-2-iodohept-2-ene **(3l)**

(xc-6-012)

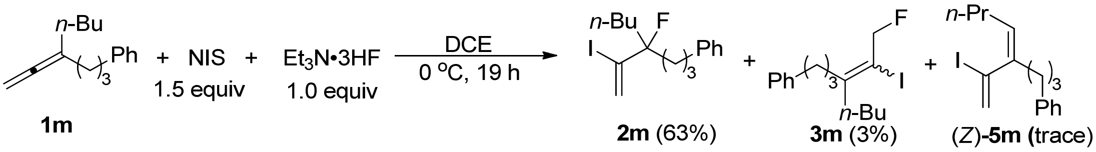


Following Typical Procedure II: The reaction of 3-butylhepta-1,2-diene **1l** (0.0917 g, 0.60 mmol) with NIS (0.2031 g, 0.90 mmol) and $Et_3N·3HF$ (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 11 h afforded a crude product. The ratio of **2l**/(*E*)-**3l** was 92:8 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2l** (0.1237 g, 69%) and **3l** (0.0103 g, 5.7%). Due to the trace amount of (*Z*)-**5l**, it has not been characterized.

2l (less polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.38 (s, 1 H, one proton of $=CH_2$), 5.92 (dd, J_1 = 4.4 Hz, J_2 = 1.4 Hz, 1 H, one proton of $=CH_2$), 2.10-1.80 (m, 2 H, CH_2), 1.78-1.60 (m, 2 H, CH_2), 1.42-1.18 (m, 8 H, 4 × CH_2), 0.91 (t, J = 6.9 Hz, 6 H, 2 × CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 126.2 (d, J = 13.3 Hz), 110.6 (d, J = 22.8 Hz), 100.0 (d, J = 183.2 Hz), 37.5 (d, J = 22.2 Hz), 24.7 (d, J = 2.5 Hz), 22.7, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -158.1 ~ -158.8 (m, 1 F); IR (neat) ν (cm^{-1}) 2957, 2872, 1613, 1466, 1398, 1380, 1341, 1129, 1089; MS (70 ev, EI) *m/z* (%) 298 (M^+, 0.97), 242 (100); HRMS calcd for $C_{11}H_{20}FI$ (M^+): 298.0594, Found: 298.0591.

3l (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.13 (d, J = 48.3 Hz, 2 H, CH_2), 2.34-2.21 (m, 4 H, 2 × CH_2), 1.47-1.24 (m, 8 H, 4 × CH_2), 0.99-0.88 (m, 6 H, 2 × CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.8 (d, J = 7.1 Hz), 94.1 (d, J = 16.4 Hz), 86.1 (d, J = 167.5 Hz), 42.2 (d, J = 2.3 Hz), 32.1 (d, J = 1.7 Hz), 31.5 (d, J = 2.7 Hz), 29.4 (d, J = 4.2 Hz), 22.8, 22.5, 13.9, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -197.4 ~ -198.0 (m, 1 F); IR (neat) ν (cm^{-1}) 2957, 2928, 2861, 1621, 1465, 1379, 1366, 1241, 1070, 1026; MS (70 ev, EI) *m/z* (%) 298 (M^+, 30.97), 109 (100); HRMS calcd for $C_{11}H_{20}FI$ (M^+): 298.0594, Found: 298.0602.

16. 3-Fluoro-2-iodo-3-(phenylpropyl)hept-1-ene **(2m)** (xc-5-064, 8-155)



Following Typical Procedure II: The reaction of 3-(phenylpropyl)hepta-1,2-diene **1m** (0.1288 g, 0.60 mmol) with NIS (0.2029 g, 0.90 mmol) and Et$_3$N·3HF (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 ^{o}C for 19 h afforded a crude product. The ratio of **2m**/**3m** was 92:8 as determined by ^{19}F NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether) afforded **2m** (0.1365 g, 63%) and **3m** (0.0177 g, a mixture of (*Z*)-**3m** and (*E*)-**3m**, the ratio of (*Z* or *E*)-**3m**/(*E* or *Z*)-**3m** was 43:57, purity = 39% based on the ^{1}H NMR analysis with mesitylene as the internal standard, 3% from **1m**).

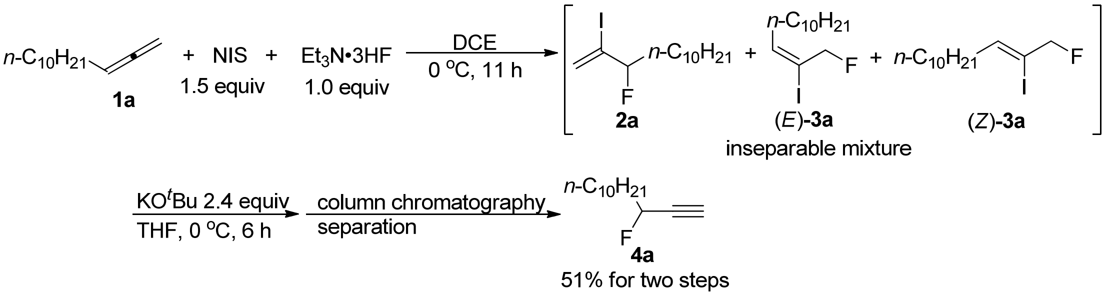
2m (less polar, liquid): ^{1}H NMR (300 MHz, CDCl$_3$) δ 7.31-7.22 (m, 2 H, ArH), 7.22-7.13 (m, 3 H, ArH), 6.37 (d, *J* = 0.6 Hz, 1 H, one proton of =CH$_2$), 5.89 (t, *J* = 2.1 Hz, 1 H, one proton of =CH$_2$), 2.71-2.51 (m, 2 H, CH$_2$), 2.08-1.60 (m, 6 H, 3 × CH$_2$), 1.38-1.20 (m, 4 H, 2 × CH$_2$), 0.89 (t, *J* = 6.3 Hz, 3 H, CH$_3$); ^{13}C NMR (75 MHz, CDCl$_3$) δ 141.9, 128.4, 128.3, 126.4 (d, *J*= 12.3 Hz), 125.8, 110.1 (d, *J* = 22.7 Hz), 100.0 (d, *J* = 183.7 Hz), 37.5 (d, *J* = 23.2 Hz), 37.3 (d, *J* = 21.7 Hz), 35.7, 24.6 (d, *J* = 2.4 Hz), 24.3 (d, *J* = 2.3 Hz), 22.6, 13.9; ^{19}F NMR (282 MHz, CDCl$_3$) δ -158.8 ~ -159.2 (m, 1 F); IR (neat) ν (cm^{-1}) 3085, 3062, 3026, 2954, 2870, 1613, 1496, 1454, 1397, 1379, 1080, 1031; MS (70 ev, EI) *m/z* (%) 360 (M$^+$, 2.18), 91 (100); HRMS calcd for $C_{16}H_{22}FI$ (M$^+$): 360.0750, Found: 360.0746.

The following signals are discernable for **3m** (less polar, liquid): ^{1}H NMR (300

MHz, $CDCl_3$) δ 5.12 (d, *J* = 48.3 Hz, 2 H, CH_2), 5.04 (d, *J* = 48.0 Hz, 2 H, CH_2); MS (70 ev, EI) *m/z* (%) 360 (M^+, 0.92), 91 (100).

Due to the amount obtained and the low purity of the isolated (*Z*)-**5m**, we were not able to fully characterize it.

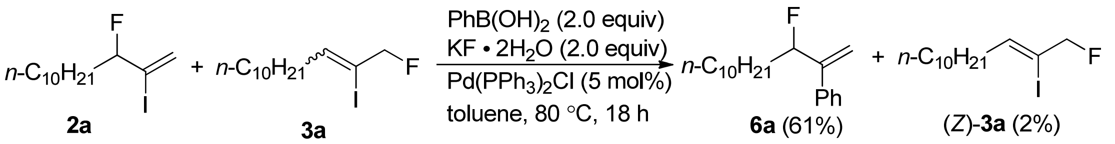
Iodofluorination Reaction of Trideca-1,2-diene (1a) and the Subsequent Elimination Reactions of the Allylic Fluorides with TBAF affording 3-Fluorotridec-1-yne (4a). (xc-6-131,156)



To a dried flask were added trideca-1,2-diene **1a** (1.0743 g, 5.97 mmol) and 3 mL of anhydrous 1,2-dichloroethane (DCE) at room temperature under N_2 atmosphere. The resulting mixture was then cooled to 0 °C in a cooling bath. After that NIS (2.0262 g, 9.01 mmol) and $Et_3N \cdot 3HF$ (1.0 mL, 0.989 g/mL, 0.9890 g, 6.14 mmol) were added sequentially at 0 °C with 2 mL of DCE washing the vessel. After 11 h the reaction was complete as monitored by TLC, the resulting mixture was quenched with 10 mL each of H_2O, sat. aq. $Na_2S_2O_3$, and aq. $NaHCO_3$, extracted with diethyl ether (20 mL×3), washed with brine, and dried over anhydrous Na_2SO_4. After filtration and concentration, a crude mixture of **2a** and **3a** was submitted directly to the next step without further characterization.

This mixture of **2a** and **3a** was dissolved in THF (10 mL) in a dried flask at the N_2 atmosphere, and then was cooled to 0 ^{o}C with a cooling bath. A solution of KO^{t}Bu (1.6135 g, 14.41 mmol) in THF (6 mL) was added slowly within 20 min with 4 mL of THF washing the vessel. As monitored by TLC, the reaction was complete after 6 h. The mixture was quenched with 20 mL of water at 0 ^{o}C, extracted with diethyl ether (20 mL×5), washed with brine and dried over anhydrous Na_2SO_4. After filtration and concentration, column chromatography on silica gel (eluent: petroleum ether, 30 ~ 60 ^{o}C) afforded **4a** (0.6007 g, 51% for two steps) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.07 (dtd, J_1 = 48.2 Hz, J_2 = 6.4 Hz, J_3 = 2.0 Hz, 1 H, CH), 2.65 (dd, J_1 = 5.6 Hz, J_2 = 2.3 Hz, 1 H, ≡CH), 2.00-1.70 (m, 2 H, CH_2), 1.50-1.40 (m, 2 H, CH_2), 1.40-1.15 (m, 14 H, 7 × CH_2), 0.88 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 82.4 (d, J = 166.2 Hz), 80.5 (d, J = 25.8 Hz), 76.0 (d, J = 10.9 Hz), 35.8 (d, J = 22.3 Hz), 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 24.3 (d, J = 4.1 Hz), 22.7, 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -175.6 (dtd, J_1 = 48.1 Hz, J_2 = 20.9 Hz, J_3 = 5.3 Hz, 1 F); IR (neat) ν (cm^{-1}) 3311, 2925, 2855, 2126, 1467, 1378, 1345, 1128, 1061; MS (EI, 70 ev) *m/z* (%) 178 (M^+-HF, 2.42), 43 (100); Elemental analysis calcd for $C_{13}H_{23}F$: C, 78.73; H, 11.69; Found: C, 78.83, H, 11.62.

Suzuki Coupling Reactions of 3-Fluoro-2-iodotridec-1-ene (2a), (*Z*)-1-Fluoro-2-iodotridec-2-ene ((*Z*)-3a), and (*E*)-1-Fluoro-2-iodotridec-2-ene ((*E*)-3a) with Phenylboronic Acid Affording 3-Fluoro-2-phenyltridec-1-ene (6a) (xc-7-022)



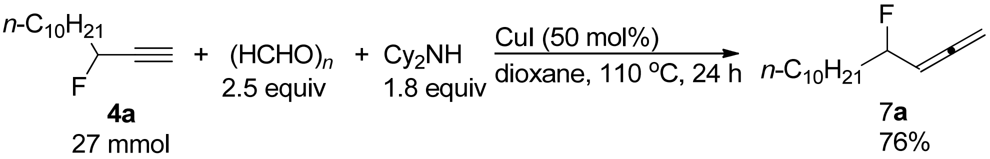
Ratio of **2a**:(*Z*)-**3a**:(*E*)-**3a** = 90:9:1

To a dried flask were added $PhB(OH)_2$ (0.0517 g, 0.40 mmol), $KF{\cdot}2H_2O$ (0.0380 g, 0.40 mmol), $Pd(PPh_3)_2Cl_2$ (0.0070 g, 0.01 mmol), a mixture of **2a** and **3a** (**2a**/(*Z*)-**3a**/(*E*)-**3a** = 90:9:1, 0.0650 g, 0.20 mmol) and 2 mL of toluene sequentially at room temperature under N_2 atmosphere. The resulting mixture then stirred in an oil bath preheated to 80 °C for 18 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel (eluent: Et_2O (60 mL)). After evaporation, the residue was purified by chromatography on silica gel (eluent: petroleum ether) afforded pure **6a** (0.0338 g, 61%) as a liquid: ^1H NMR (300 MHz, $CDCl_3$) δ 7.42-7.24 (m, 5 H, ArH), 5.44-5.20 (m, 3 H, $=CH_2$ and CH), 1.78-1.53 (m, 2 H, CH_2), 1.45-1.10 (m, 16 H, 8 × CH_2), 0.87 (t, *J* = 6.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.7 (d, *J* = 16.4 Hz), 138.8 (d, *J* = 2.8 Hz), 128.4, 127.8, 126.9, 114.3 (d, *J* = 10.8 Hz), 94.5 (d, *J* = 172.1 Hz), 34.4 (*d*, *J* = 22.5 Hz), 31.9, 29.6, 29.53, 29.47, 29.32, 29.26, 25.0 (*d*, *J* = 4.4 Hz), 22.7, 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -177.8~-177.4 (m, 1 F); IR (neat) ν (cm^{-1}) 3083, 3058, 3032, 2924, 2854, 1946, 1820, 1633, 1598, 1574, 1495, 1483, 1466, 1445, 1378, 1130, 1074, 1028; MS (EI, 70 ev) *m*/*z* (%) 276 (M^+, 2.06), 136 (100); HRMS calcd for $C_{19}H_{29}F$ (M^+): 276.2253, Found: 276.2255.

There is 2% of (*Z*)-**3a** left after the Suzuki coupling reaction with 1% of unknown product detected by the ^{19}F NMR analysis of the crude product.

Transformation from alkyne 4a to allene 7a:

Synthesis of 4-Fluorotetradeca-1,2-diene (**7a**) (xc-8-001,169)

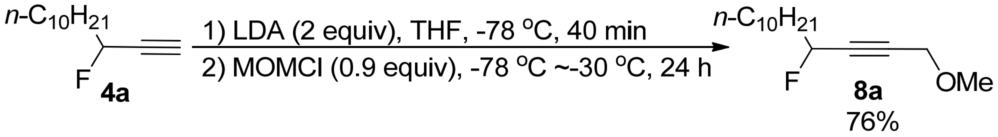


To a dried flask were added $(HCHO)_n$ (2.0291 g, 67.6 mmol), CuI (2.6530, 98%, 13.6 mmol), and 40 mL of anhydrous 1,4-dioxane at room temperature under N_2 atmosphere. Then Cy_2NH (9.9 mL, 0.91 g/mL, 98%, 8.8288 g, 48.8 mmol), 3-fluorotridec-1-yne **4a** (5.3452 g, 27.0 mmol), and 60 mL of anhydrous 1,4-dioxane were added to the vessel. The resulting mixture was then placed in a pre-heated oil bath of 110 ^{o}C with stirring for 24 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was concentrated and filtrated through a short pad of silica gel. After evaporation, 50 mL each of Et_2O and 5% HCl (aq.) were added. After filtration to remove the solid, the filtrate was then extracted with Et_2O (30 mL×5), washed with brine (30 mL×2) and dried over anhydrous Na_2SO_4. After evaporation, the residue was purified by chromatography on silica gel for twice (eluent: petroleum ether) to afford **7a** (4.3415 g, 76%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.36-5.20 (m, 1 H, =CH), 5.06-4.80 (m, 3 H, CH and $=CH_2$), 1.89-1.59 (m, 2 H, CH_2), 1.54-1.17 (m, 16 H, 8 × CH_2), 0.88 (t, $J = 6.6$ Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.8 (d, $J = 11.9$ Hz), 91.4 (d, $J = 167.3$ Hz), 91.0 (d, $J = 24.1$ Hz), 76.9 (d, $J = 3.1$ Hz), 35.3 (d, $J = 22.1$ Hz), 31.9, 29.6, 29.54, 29.50, 29.32, 29.31, 24.8 (d, $J = 4.5$ Hz), 22.7, 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -168.7~-169.2 (m, 1 F); IR (neat) ν (cm^{-1}) 2925, 2854, 1958, 1467, 1377, 1061; MS

(70 ev, EI) *m/z* (%) 212 (M^+, 1.59), 72 (100); HRMS calcd for $C_{14}H_{25}F$ (M^+): 212.1940, Found: 212.1945.

Transformation from alkyne 4a to alkyne 8a:

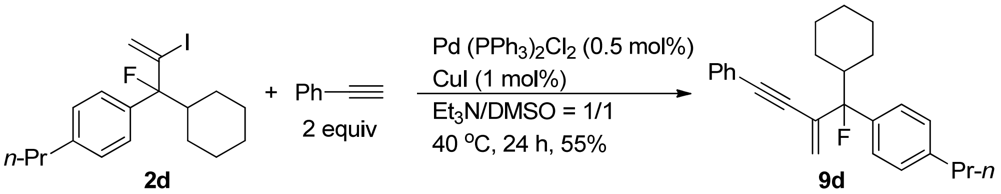
Synthesis of 4-Fluoro-1-methoxytetradec-2-yne (**8a**) (xc-8-094, 170)



To a dried flask were added 3-fluorotridec-1-yne **4a** (0.2981 g, 1.0 mmol) and 3 mL of anhydrous THF at room temperature under N_2 atmosphere. The vessel was then placed in the dry ice-acetone bath (-78 °C), which was followed by the addition of LDA (1.5 mL, 2.0 M in THF, 3.0 mmol) slowly within 5 min. After stirring for 30 min at -78 °C, MOMCl (100 μL, d = 1.06 g/mL, 0.1060 g, 1.35 mmol) was added. After 15 min, the resulting mixture was relocated to a -30 °C cooling bath. After being stirred for 24 h, the reaction was complete as monitored by TLC. The reaction was quenched with sat. NH_4Cl (10 mL) at -30 °C, extracted with Et_2O (10 mL×3), washed with brine (10 mL), and dried over anhydrous Na_2SO_4. After evaporation, the residue was purified by chromatography on silica gel (eluent: petroleum ether/地 diethyl ether = 100 / 1) to afford **8a** (0.2407 g, 76%) as a liquid: ^1H NMR (300 MHz, $CDCl_3$) δ 5.14 (dt, J_1 = 48.6 Hz, J_2 = 6.3 Hz, 1 H, CH), 4.17 (dd, J_1 = 6.3 Hz, J_2 = 1.2 Hz, 2 H, OCH_2), 3.39 (s, 3 H, OCH_3), 1.97-1.72 (m, 2 H, CH_2), 1.52-1.20 (m, 16 H, 8 × CH_2), 0.88 (t, J = 6.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 83.7 (d, J = 25.4 Hz), 83.6 (d, J = 61.4 Hz), 82.6 (d, J = 166.2 Hz), 59.7 (d, J = 2.6 Hz), 57.6, 35.8 (d, J = 21.8 Hz), 31.9, 29.55, 29.50, 29.4, 29.3, 29.1, 24.4 (d, J = 3.5 Hz), 22.7, 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -173.4 (dtt, J_1 = 48.8 Hz, J_2 = 23.0 Hz, J_3 = 6.4 Hz, 1 F); IR (neat) ν (cm^{-1}) 2924, 2854, 2239, 1998, 1724, 1688, 1466, 1377, 1348, 1279, 1188, 1149, 1102, 1049, 1001; Raman ν (cm^{-1}) 2908, 2852, 2824, 2243, 1441, 1359, 1303, 1081; MS (70 ev, EI) *m/z* (%) 243 (M^++1, 3.84), 43 (100), 41 (100); HRMS calcd for $C_{15}H_{27}OF$ (M^+): 242.2046, Found: 242.2047.

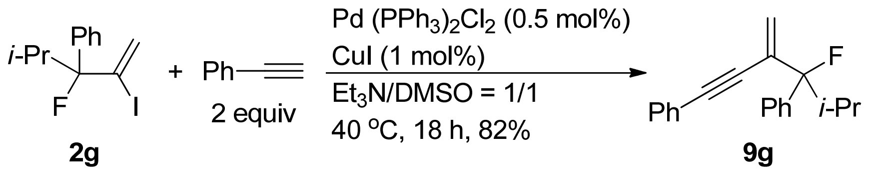
Sonogashira Coupling Reactions of Allylic Fluorides with Phenylacetylene.

1. 4-Cyclohexyl-4-fluoro-3-methylene-1-phenyl-4-(4'-propylphenyl)but-1-yne (**9d**) (xc-6-194)



Typical Procedure III: To a dried flask were added CuI (0.0020 g, 0.01 mmol), $Pd(PPh_3)_2Cl_2$ (0.0036 g, 0.005 mmol), 3-cyclohexyl-3-fluoro-2-iodo-3-(4'-propylphenyl)prop-1-ene **2d** (0.3854 g, 1.00 mmol), 4 mL of anhydrous Et_3N, phenylacetylene (0.2043 g, 2.00 mmol) and 4 mL of DMSO sequentially at room temperature under N_2 atmosphere. The resulting mixture was then stirred in an oil bath preheated to 40 °C. After 24 h, the reaction was completed as monitored by TLC, taken out and cooled to room temperature. The resulting mixture was quenched with 10 mL each of H_2O and HCl (aq., 5%), extracted with diethyl ether (15 mL×3), washed with brine and dried over anhydrous Na_2SO_4. After filtration and concentration, column chromatography on silica gel (eluent: petroleum ether) afforded **9d** (0.1992 g, 55%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60-7.46 (m, 4 H, ArH), 7.44-7.35 (m, 3 H, ArH), 7.26-7.18 (m, 2 H, ArH), 5.78 (s, 1 H, one proton of =CH_2), 5.55 (dd, J_1 = 5.4 Hz, J_2 = 1.2 Hz, 1 H, one proton of =CH_2), 2.56 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.48-2.23 (m, 1 H, CH), 2.04-1.79 (m, 2 H, CH_2), 1.75-1.53 (m, 4 H, 2 × CH_2), 1.48-1.10 (m, 6 H, 3 × CH_2), 0.93 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6, 138.7 (d, *J* = 22.6 Hz), 134.0 (d, *J* = 26.7 Hz), 131.4, 128.41, 128.35, 128.0 (d, *J* = 1.4 Hz), 124.6 (d, *J* = 9.9 Hz), 123.0, 120.1 (*d*, *J* = 11.9 Hz), 100.5 (*d*, *J* = 188.3 Hz), 91.4, 87.4 (*d*, *J* = 8.2 Hz), 43.4 (*d*, *J* = 20.7 Hz), 37.6, 26.7, 26.6 (*d*, *J* = 3.2 Hz), 26.5, 26.3, 26.2 (*d*, *J* = 2.9 Hz), 24.4, 13.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -173.2 (dd, J_1 = 31.4 Hz, J_2 = 4.7 Hz, 1 F); IR (neat) ν (cm^{-1}) 3081, 3057, 3031, 2930, 2852, 2669, 2198, 1604, 1512, 1489, 1464, 1444, 1412, 1380, 1339, 1297, 1247, 1223, 1189, 1142, 1107, 1082, 1069, 1011; MS (EI, 70 ev) *m*/*z* (%) 360 (M^+, 100); HRMS calcd for $C_{26}H_{29}F$ (M^+): 360.2253, Found: 360.2253.

2. 4-Fluoro-5-methyl-3-methylene-1,4-diphenylhex-1-yne (**9g**) (xc-6-187)



Following Typical Procedure III: The reaction of 3-fluoro-2-iodo-4-methyl-3-phenylpent-1-ene **2g** (0.0765 g, 0.25 mmol) with phenylacetylene (0.0522 g, 0.51 mmol), CuI (0.0006 g, 0.003 mmol), and

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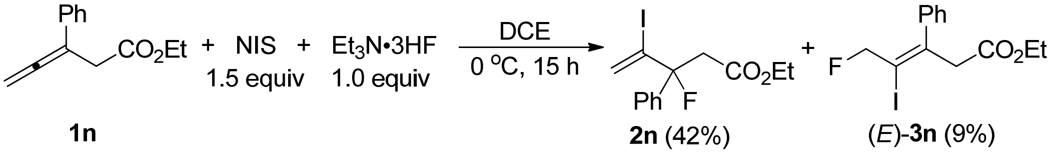
$Pd(PPh_3)_2Cl_2$ (0.0009 g, 0.0013 mmol) in Et_3N (1 mL) and DMSO (1 mL) at 40 °C for 18 h afforded **9g** (eluent: petroleum ether/ ethyl acetate = 200 : 1) (0.0573 g, 82%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63-7.52 (m, 2 H, ArH), 7.50-7.39 (m, 2 H, ArH), 7.39-7.22 (m, 6 H, ArH), 5.82 (s, 1 H, one proton of $=CH_2$), 5.56 (dd, J_1 = 5.6 Hz, J_2 = 1.1 Hz, 1 H, one proton of $=CH_2$), 2.95-2.64 (m, 1 H, CH), 1.14 (d, J = 6.9 Hz, 3 H, CH_3), 0.84 (d, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.7 (d, J = 23.5 Hz), 134.2 (d, J = 28.0 Hz), 131.4, 128.5, 128.4, 128.0 (d, J = 1.5 Hz), 127.3, 124.7 (d, J = 10.8 Hz), 122.9, 120.1 (*d*, J = 11.7 Hz), 100.6 (*d*, J = 189.4 Hz), 91.5, 87.2 (*d*, J = 8.3 Hz), 33.6 (*d*, J = 21.8 Hz), 16.5 (*d*, J = 3.0 Hz), 16.4 (*d*, J = 3.4 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -178.2 (dd, J_1 = 31.4 Hz, J_2 = 5.5 Hz, 1 F); IR (neat) ν (cm^{-1}) 3061, 3034, 2974, 2936, 2878, 1606, 1490, 1469, 1444, 1387, 1306, 1221, 1195, 1130, 1070, 1036; MS (EI, 70 ev) *m*/*z* (%) 278 (M$^+$, 100); HRMS calcd for $C_{20}H_{19}F$ (M$^+$): 278.1471, Found: 278.1475.

Iodofluorination reactions of functionalized allenes.

1. Ethyl 3-fluoro-4-iodo-3-phenylpent-4-enoate **(2n)** and (*E*)-ethyl 5-fluoro-4-iodo-3-phenylpent-3-enoate ((*E*)-**3n**) (xc-9-041)



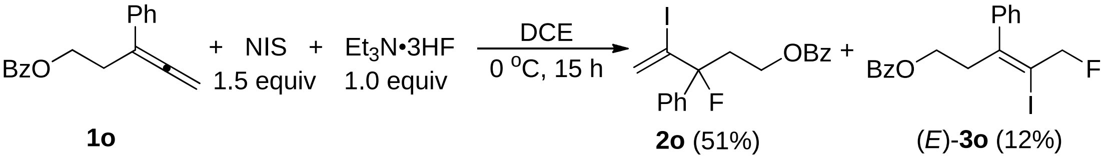
Following Typical Procedure II: The reaction of ethyl 3-phenyl-3,4-allenoate **1n** (0.1215 g, 0.60 mmol) with NIS (0.2028 g, 0.90 mmol) and Et_3N·3HF (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 15 h afforded a crude product. The ratio of **2n**/(*E*)-**3n** was 71:29 as determined by ^{19}F NMR analysis of the crude product. Column chromatography on silica gel for twice, the first time (eluent: petroleum ether/dichloromethane = 5/1) afforded a mixture of **2n** and (*E*)-**3n**, further purification (eluent: petroleum ether/ ethyl ether = 80/1) afforded pure **2n** (0.0880 g, 42%) and (*E*)-**3n** (0.0212 g, purity = 92% based on the ^{1}H NMR analysis with mesitylene as the internal standard, 9%).

2n (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.30 (m, 5 H, ArH), 6.49 (s, 1 H, one proton of $=CH_2$), 6.07 (s, 1 H, one proton of $=CH_2$), 4.06 (q, *J* = 7.1 Hz, 2 H, OCH_2), 3.55-3.13 (m, 2 H, CH_2), 1.13 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 138.5 (d, *J* = 23.0 Hz), 128.5 (d, *J* = 1.3 Hz), 128.2, 127.7 (d, *J* = 8.2 Hz), 125.5 (d, *J* = 7.8 Hz), 111.9 (d, *J* = 26.9 Hz), 96.9 (d, *J* = 185.1 Hz), 60.8, 43.0 (d, *J* = 23.4 Hz), 13.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -139.5 (t, *J* = 22.6 Hz, 1 F); IR (neat) ν (cm^{-1}) 3061, 3030, 2981, 2934, 1739, 1611, 1494, 1448, 1370, 1325, 1180, 1024; MS (ESI) *m/z* (%) 366 ($[M+NH_4]^+$); HRMS (ESI) calcd for $C_{13}H_{18}FINO_2$ ($[M+NH_4]^+$): 366.0361, Found: 366.0363.

(*E*)-**3n** (less polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.28 (m, 3 H, ArH), 7.24-7.12 (m, 2 H, ArH), 4.93 (d, *J* = 47.7 Hz, 2 H, CH_2F), 4.11 (q, *J* = 7.1 Hz, 2 H, OCH_2), 3.73 (d, *J* = 3.0 Hz, 2 H, CH_2), 1.20 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.9 (d, *J* = 4.1 Hz), 146.6 (d, *J* = 8.3 Hz), 138.2 (d, *J* = 3.0 Hz), 128.6, 128.3, 127.9 (d, *J* = 2.2 Hz), 102.2 (d, *J* = 18.5 Hz), 86.4 (d, *J* = 168.2 Hz), 61.1, 49.8 (d, *J* = 1.9 Hz), 14.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -198.5 (tt, J_1 = 47.5 Hz, J_2 = 3.1 Hz, 1 F); IR (neat) ν (cm^{-1}) 2980, 2917, 1737, 1607, 1491, 1443, 1368, 1326, 1243, 1175, 1095, 1030; MS (ESI) *m/z* (%) 366 ($[M+NH_4]^+$); HRMS (ESI) calcd for $C_{13}H_{18}FINO_2$ ($[M+NH_4]^+$): 366.0361, Found: 366.0366.

The configuration of the C=C bond in (*E*)-**3n** was established by the NOE study (see page 171 in this document).

2. 3-Fluoro-4-iodo-3-phenylpent-4-en-1-yl benzoate **(2o)** and (*E*)-5-fluoro-4-iodo-3-phenylpent-3-en-1-yl benzoate ((*E*)-**3o**) (xc-9-044)



Following Typical Procedure II: The reaction of 3-phenylpenta-3,4-dienyl benzoate **1o** (0.1588 g, 0.60 mmol) with NIS (0.2025 g, 0.90 mmol) and $Et_3N\cdot3HF$ (0.1 mL, 0.989 g/mL, 0.0989 g, 0.61 mmol) in DCE (2 mL) at 0 °C for 15 h afforded a crude product. The ratio of **2o**/(*E*)-**3o** was 79:21 as determined by ^{19}F NMR analysis of the crude product. Further purification on silica gel (eluent: petroleum ether/ethyl ether = 80/1) afforded pure **2o** (0.1252 g, 51% from **1o**) and (*E*)-**3o** (0.0370 g, purity = 82% based on the ^1H NMR analysis with mesitylene as the internal standard, 12%, the remaining is **2o**).

2o (less polar, liquid): ^1H NMR (300 MHz, $CDCl_3$) δ 8.05-7.88 (m, 2 H, ArH), 7.63-7.30 (m, 8 H, ArH), 6.59 (s, 1 H, one proton of $=CH_2$), 6.08 (s, 1 H, one proton of $=CH_2$), 4.58-4.29 (m, 2 H, OCH_2), 3.04-2.58 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 139.0 (d, *J* = 23.0 Hz), 132.9, 129.9, 129.6, 128.5 (d, *J* = 1.4 Hz), 128.4, 128.3, 127.1 (d, *J* = 9.4 Hz), 125.5 (d, *J* = 8.1 Hz), 113.0 (d, *J* = 26.9 Hz), 98.0 (d, *J* = 181.4 Hz), 60.4 (d, *J* = 4.2 Hz), 36.1 (d, *J* = 22.1 Hz); ^{19}F NMR (282

MHz, $CDCl_3$) δ -143.1 (dd, J_1 = 25.0 Hz, J_2 = 21.6 Hz, 1 F); IR (neat) ν (cm^{-1}) 1718, 1604, 1584, 1493, 1450, 1393, 1315, 1274, 1213, 1176, 1115, 1069, 1028, 1001; MS (ESI) *m/z* (%) 428 ($[M+NH_4]^+$); HRMS (ESI) calcd for $C_{18}H_{20}FI\ NO_2$ ($[M+NH_4]^+$): 428.0517, Found: 428.0519.

The following signals are discernable for (*E*)-**3o** (more polar, liquid): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02-7.88 (m, 2 H, ArH), 7.61-7.50 (m, 1 H, ArH), 7.49-7.29 (m, 5 H, ArH), 7.22-7.10 (m, 2 H, ArH), 4.90 (d, *J* = 47.7 Hz, 2 H, CH_2F), 4.30 (t, *J* = 6.6 Hz, 2 H, OCH_2), 3.28-3.12 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 150.1 (d, *J* = 7.8 Hz), 137.9 (d, *J* = 2.9 Hz), 132.9, 129.9, 129.6, 128.7, 128.3, 127.9 (d, *J* = 2.3 Hz), 100.1 (d, *J* = 17.3 Hz), 86.7 (d, *J* = 168.2 Hz), 61.8 (d, *J* = 3.9 Hz), 43.3 (d, *J* = 1.3 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ -198.3 (tt, J_1 = 47.7 Hz, J_2 = 3.4 Hz, 1 F); IR (neat) ν (cm^{-1}) 1720, 1601, 1491, 1452, 1372, 1314, 1271, 1176, 1113, 1069, 1026; MS (ESI) *m/z* (%) 428 ($[M+NH_4]^+$); HRMS (ESI) calcd for $C_{18}H_{20}FI\ NO_2$ ($[M+NH_4]^+$): 428.0517, Found: 428.0519.

The configuration of the C=C bond in (*E*)-**3o** was established by the NOE study (see page S180 in this document)).

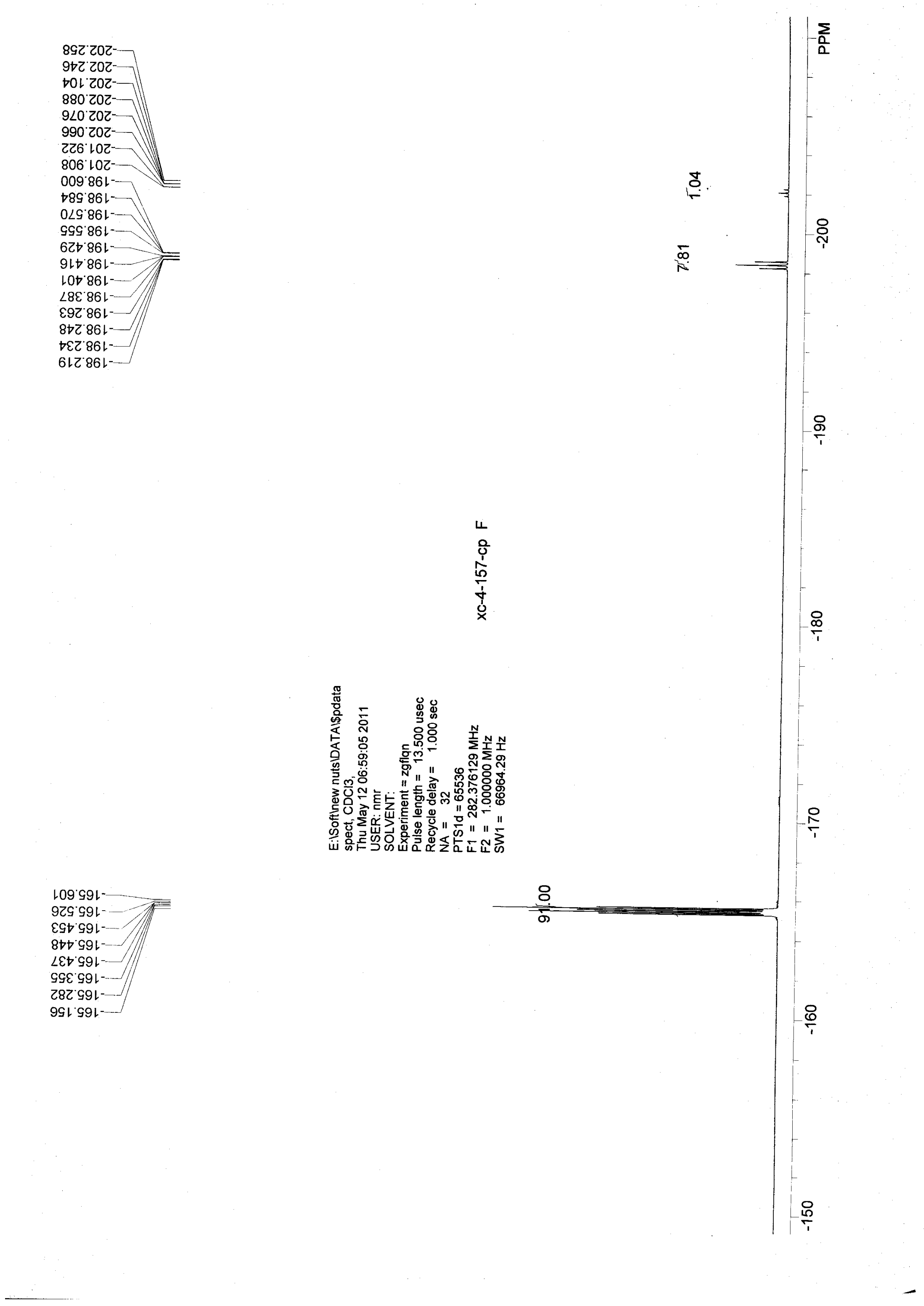
References:

[1] J.-L. Moreau and M. Gaudemar, *J. Organomet. Chem.*, 1976, **108**, 159.

[2] L. Brandsma, and H. D. Verkuijsse, *Synthesis of Acetylenes, Allenes and Cumulenes*, Elsevier, Amsterdam, 1981.

[3] G. A. Olah, J. T. Welch, Y. D. Vankar, M. Nojima, I. Kerekes and J. A. Olah, *J. Org. Chem.*, 1979, **44**, 3872.

[4] O. A. Wong and Y. A. Shi, *J. Org. Chem.*, 2009, **74**, 8377.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-198.570
-198.555
-198.429
-198.416
-198.401
-198.387
-198.263
-198.248
-198.234
-198.219
-165.601
-165.526
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7.81
91.00
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F2 = 1.000000 MHz
SW1 = 66964.29 Hz
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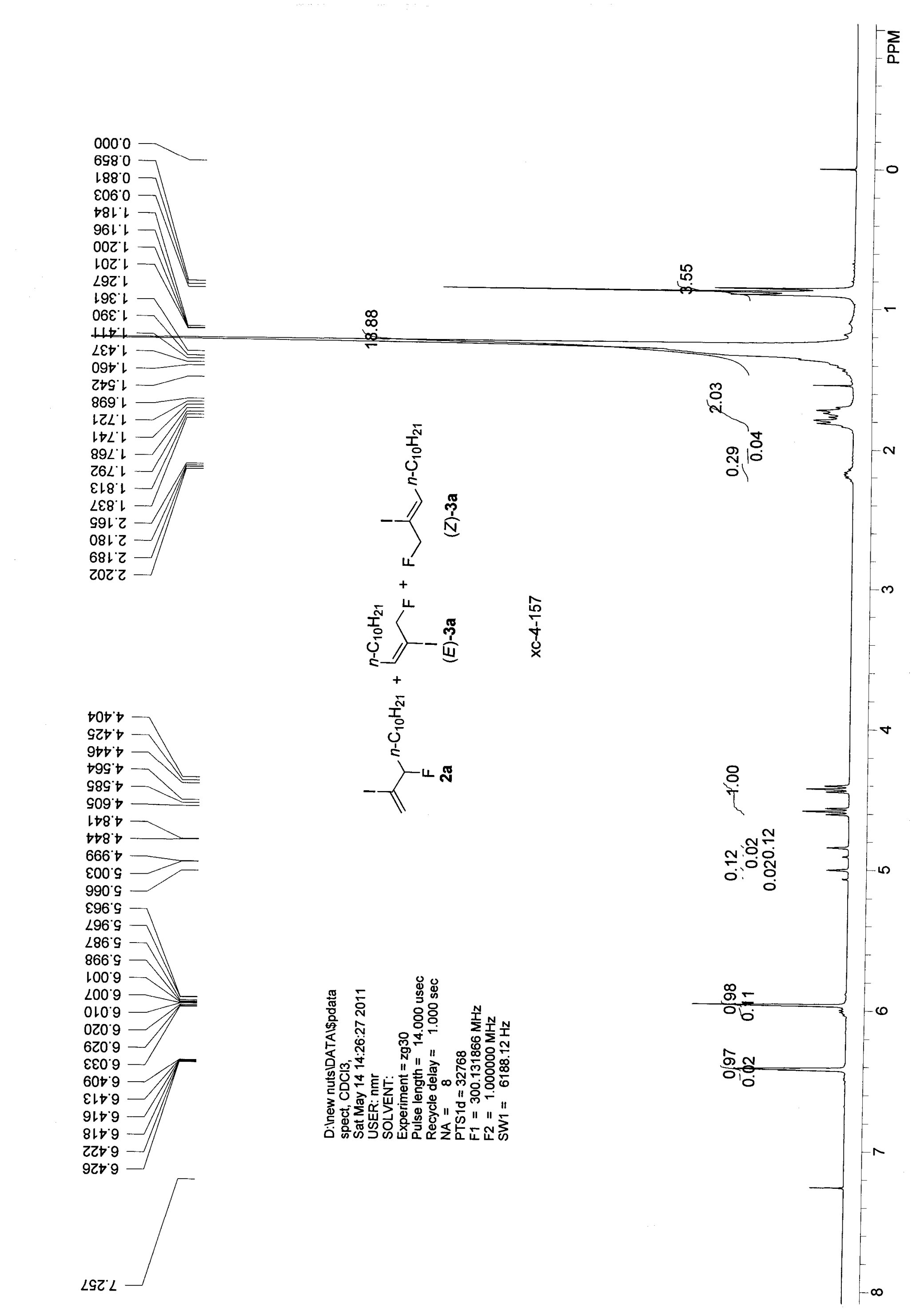
-170
-160
-150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1.437
1.460
1.542
1.698
1.721
1.741
1.768
1.792
1.813
1.837
2.165
2.180
2.189
2.202
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4.425
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4.564
4.585
4.605
4.841
4.844
4.999
5.003
5.066
5.963
5.967
5.987
5.998
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6.029
6.033
6.409
6.413
6.416
6.418
6.422
6.426
7.257
3.55
18.88
2.03
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0.04
1.00
0.12
0.02
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0.12
0.98
0.11
0.97
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F
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+
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(Z)-3a
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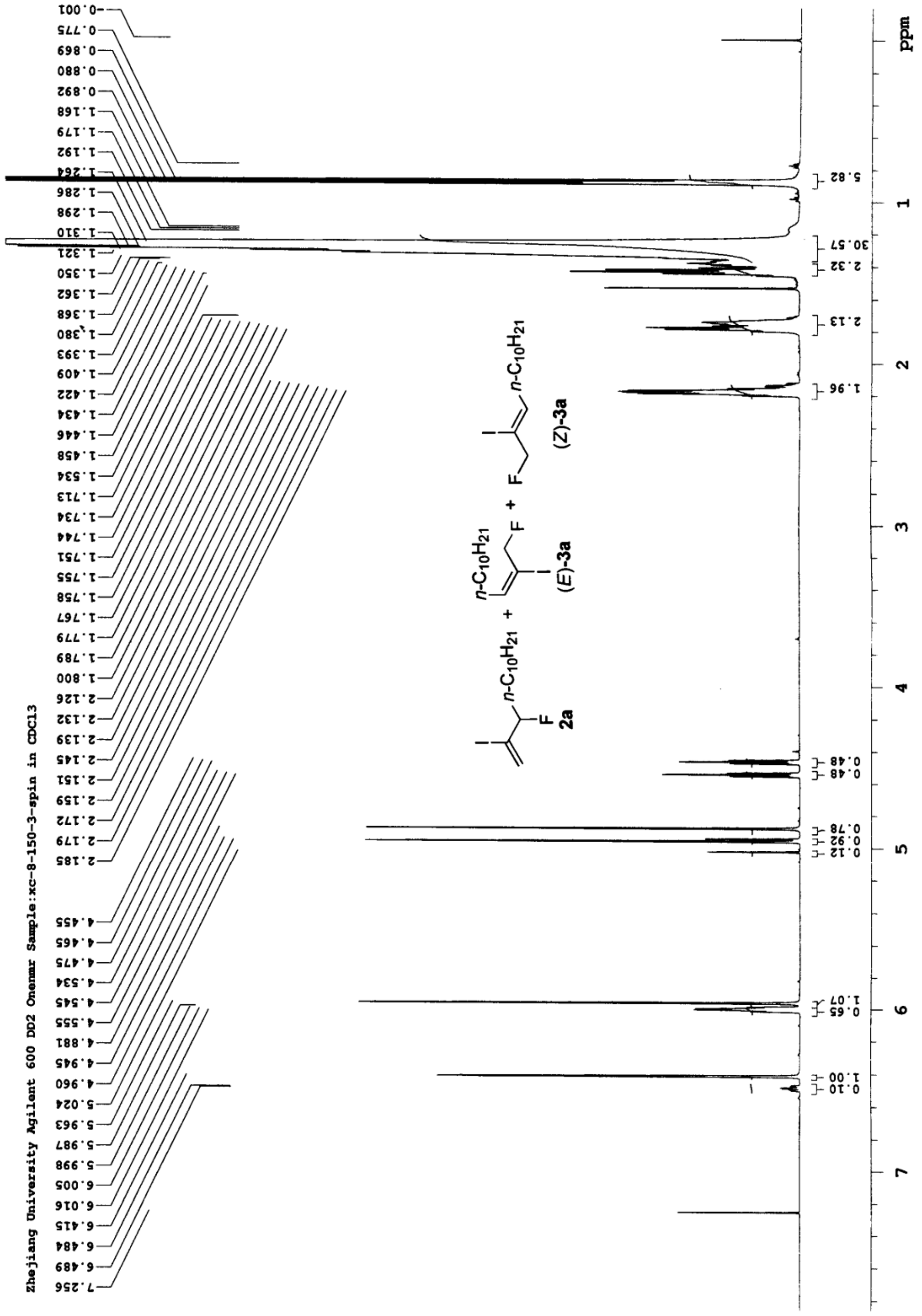
3
4
5
6
7
8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6.016
6.415
6.484
6.489
7.256
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+
n-C10H21
F
(E)-3a
+
F
n-C10H21
(Z)-3a
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2.32
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ppm
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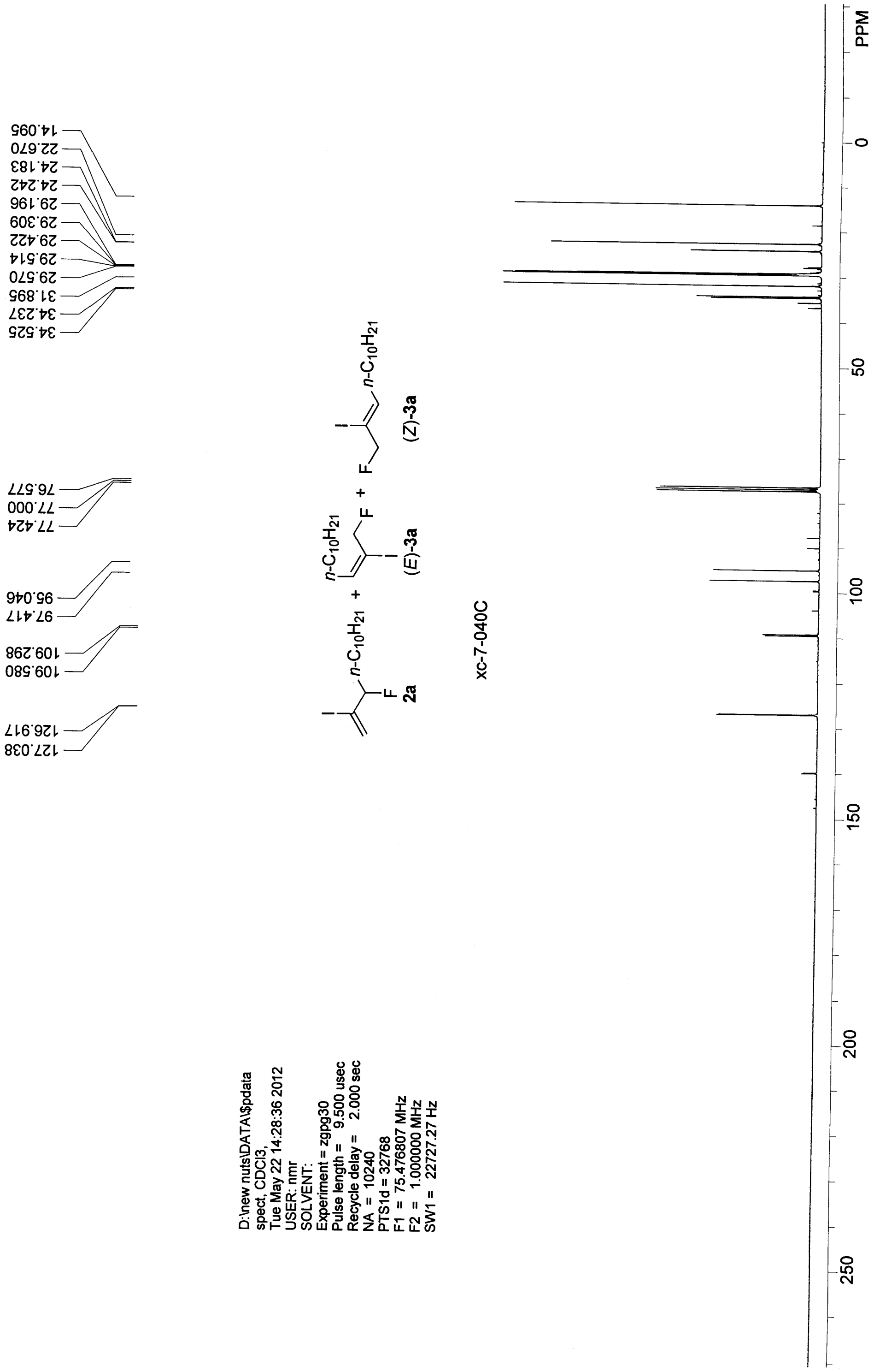
3
4
5
6
7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34.525
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77.000
77.424
95.046
97.417
109.298
109.580
126.917
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n-$C_{10}H_{21}$
F
2a
+
(E)-3a
+
(Z)-3a
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200
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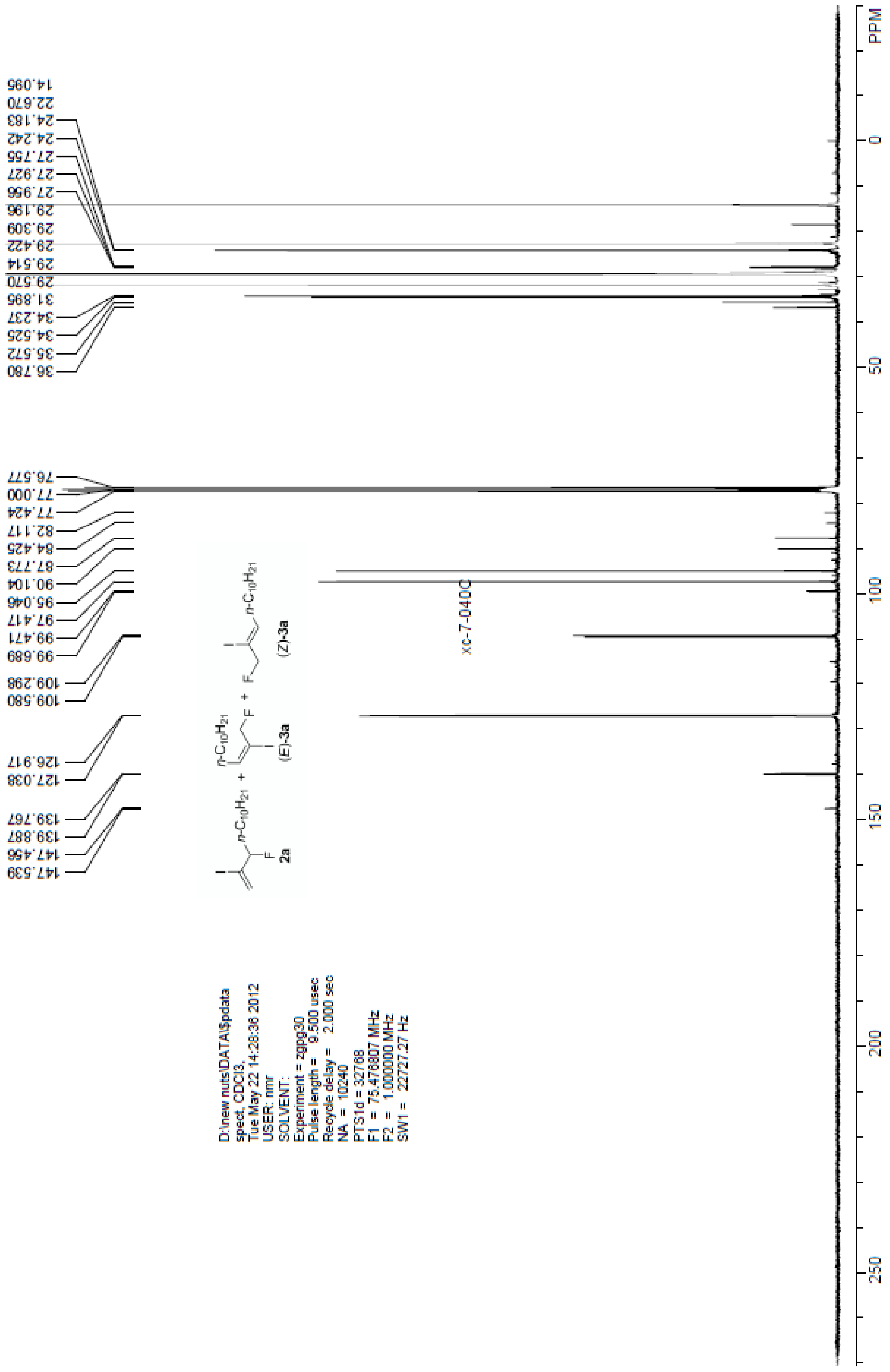
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77.000
77.424
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87.773
90.104
95.046
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126.917
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139.887
147.456
147.539
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F
2a
+
n-C$_{10}$H$_{21}$
F
(E)-3a
+
F
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Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10240
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F2 = 1.000000 MHz
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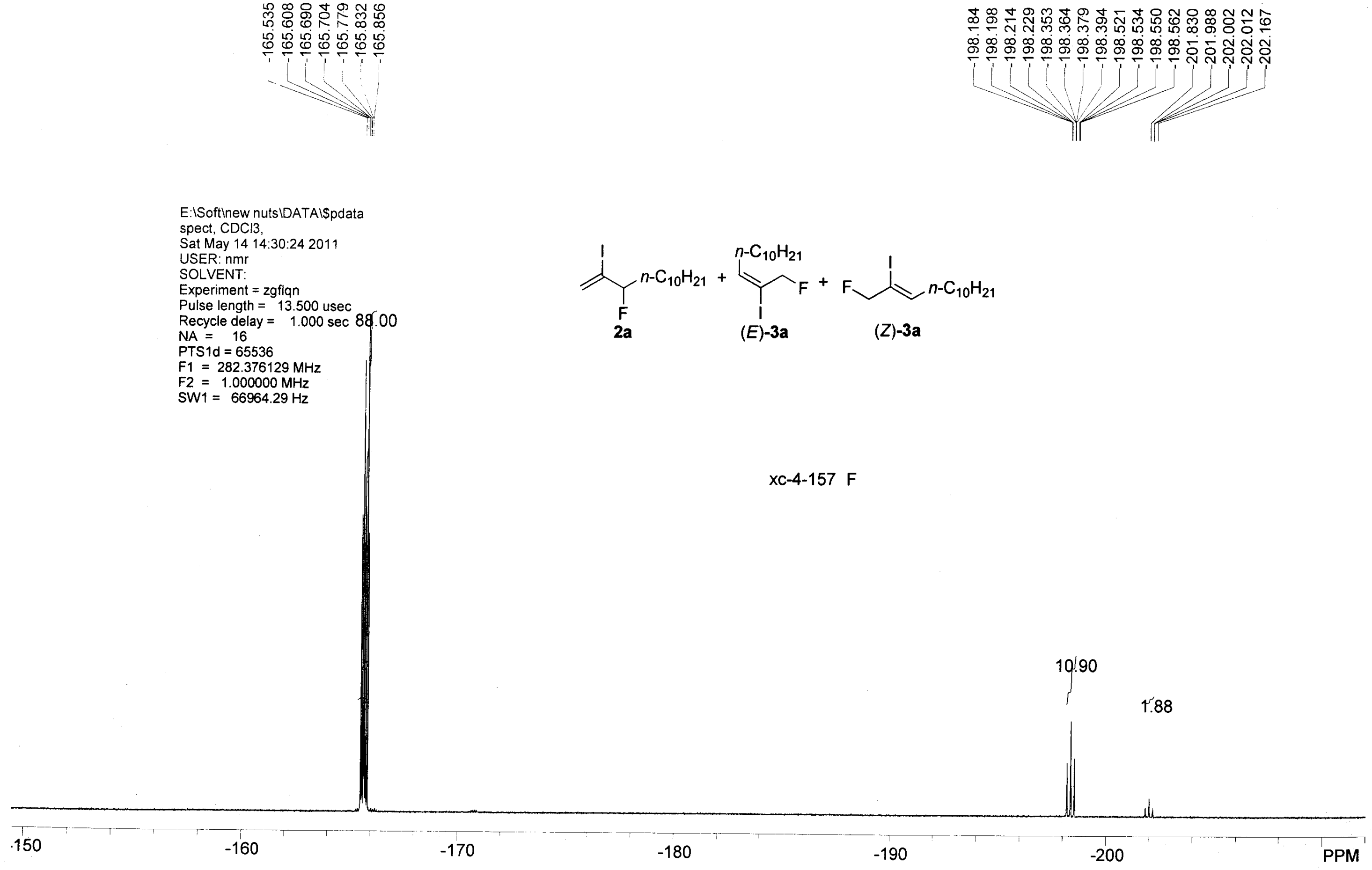
50
100
150
200
250



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F2 = 1.000000 MHz
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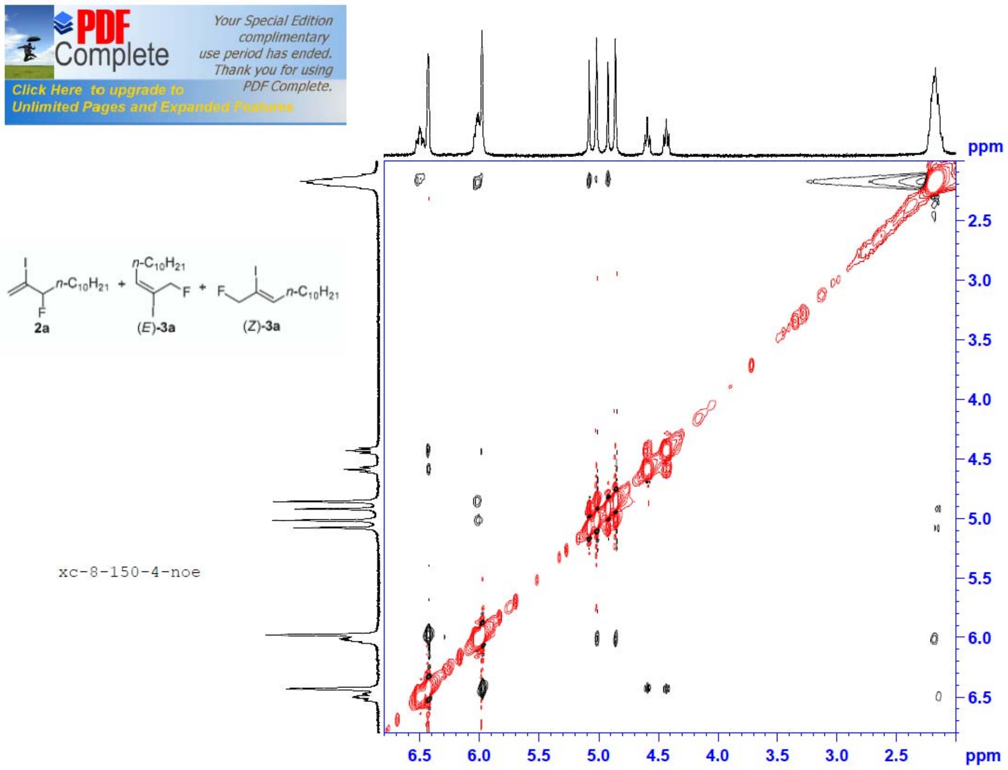
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F
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n-C10H21
F
(E)-3a
F
n-C10H21
(Z)-3a
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3.0
3.5
4.0
4.5
5.0
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3.0
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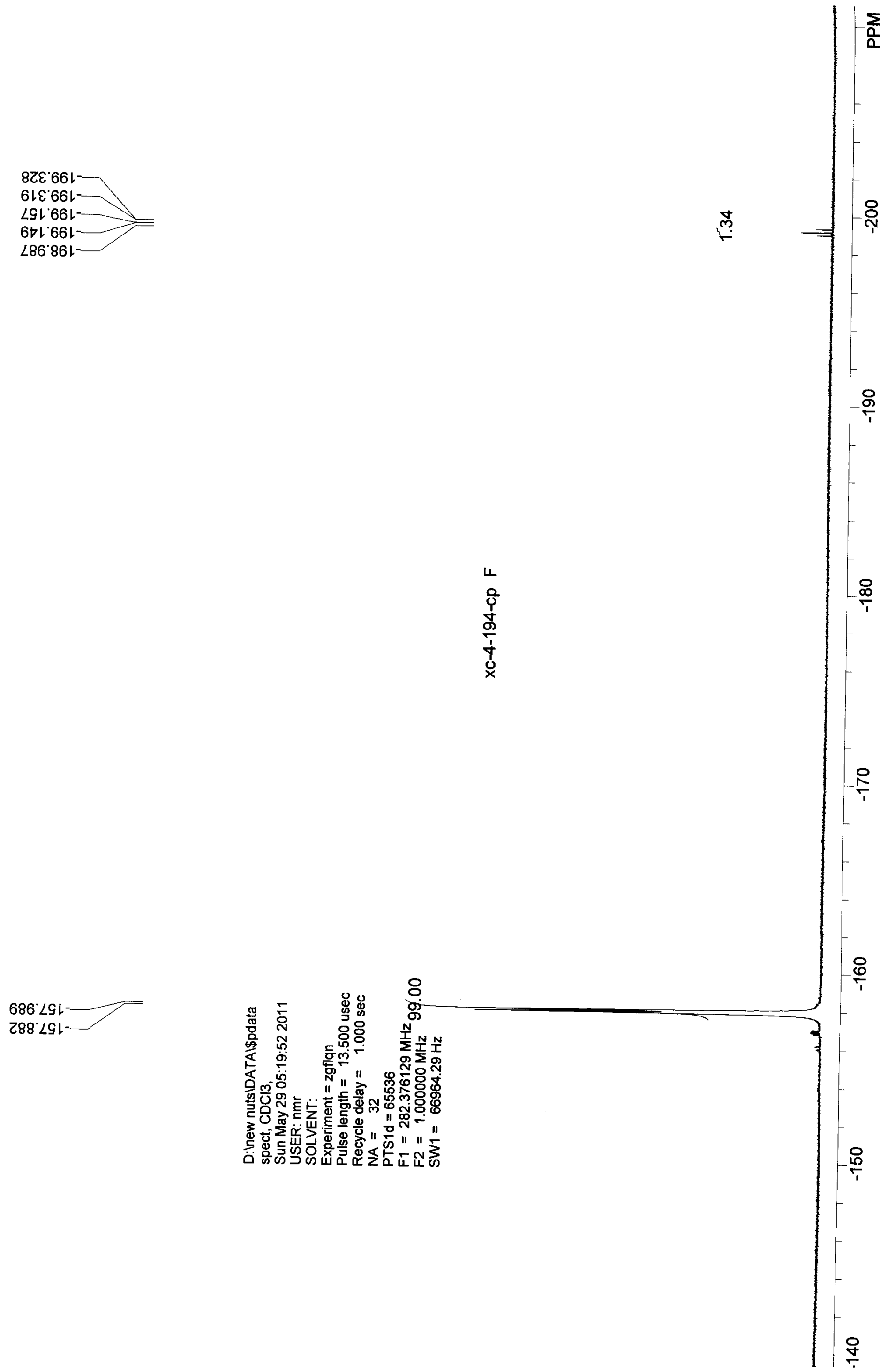
ppm



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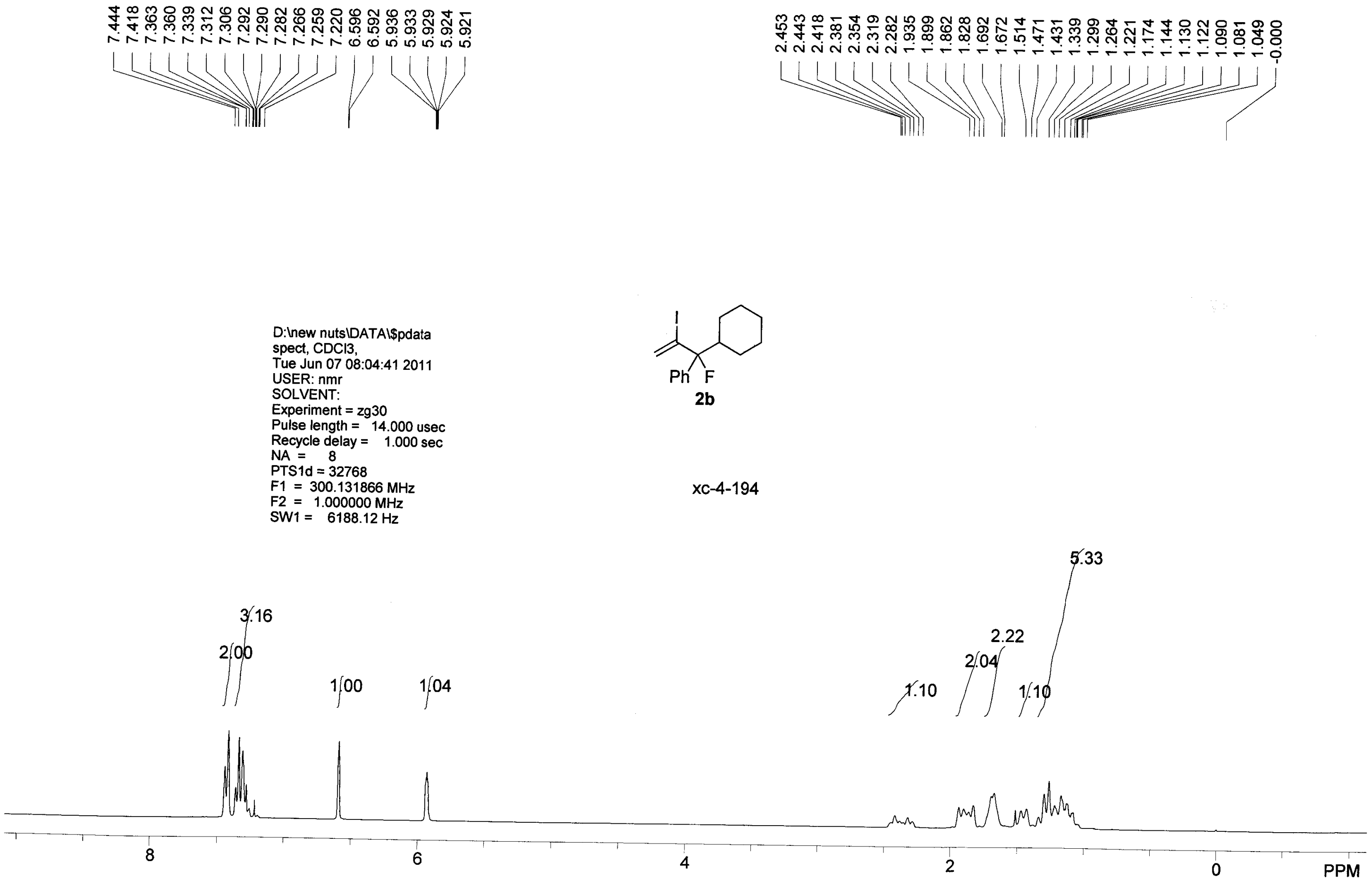

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xc-4-194

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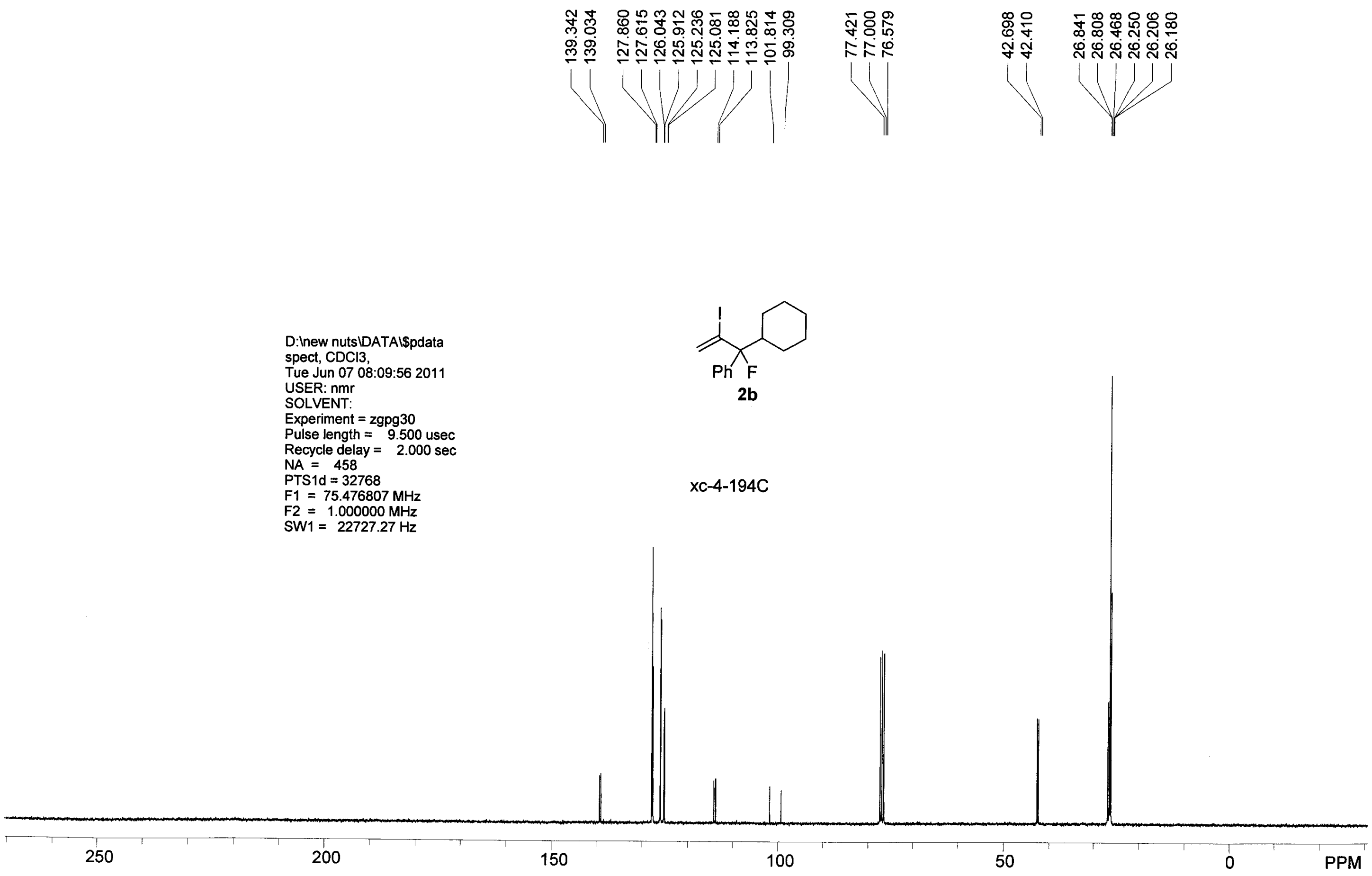

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xc-4-194C

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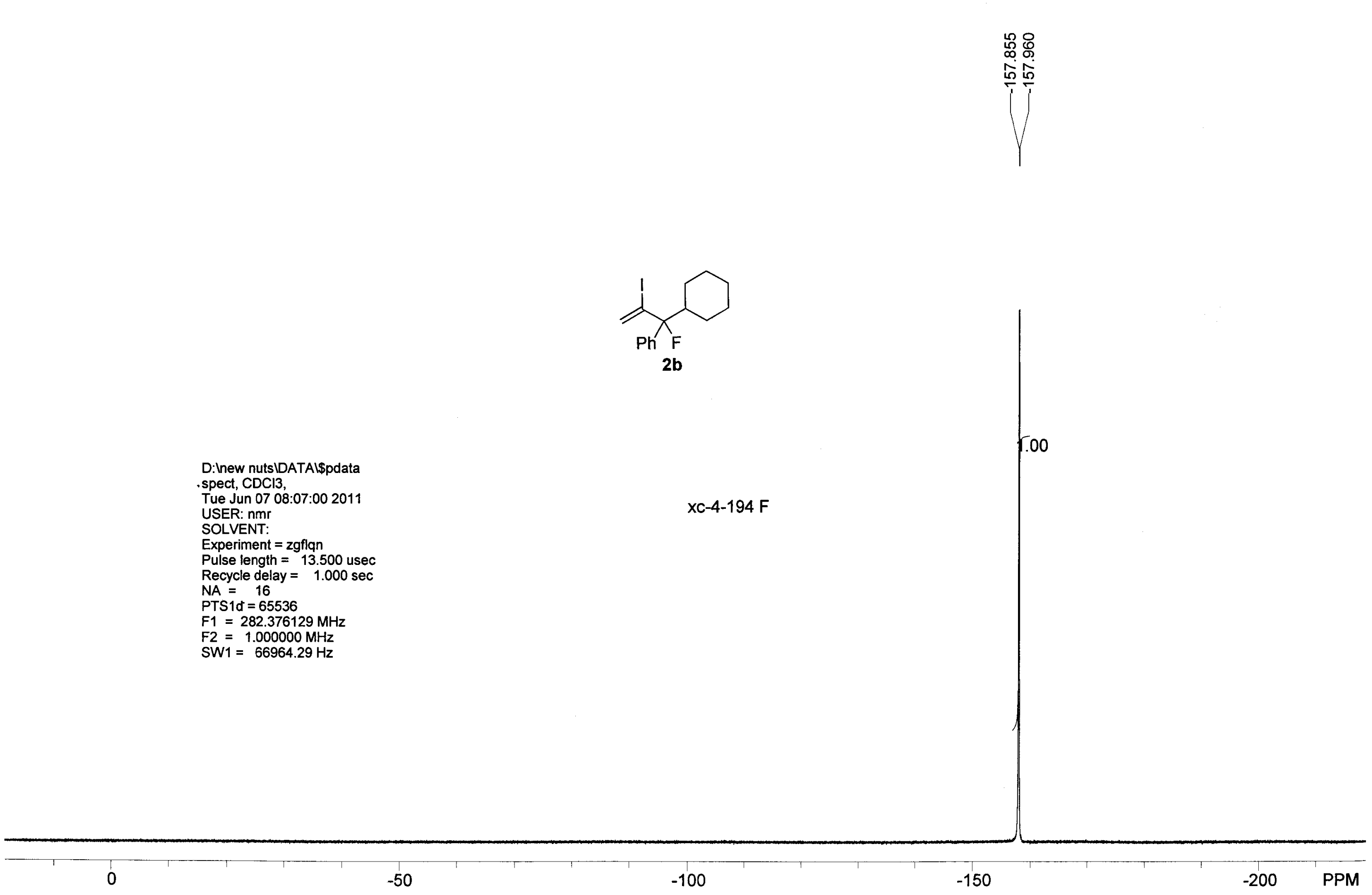

2b

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SW1 =   66964.29 Hz
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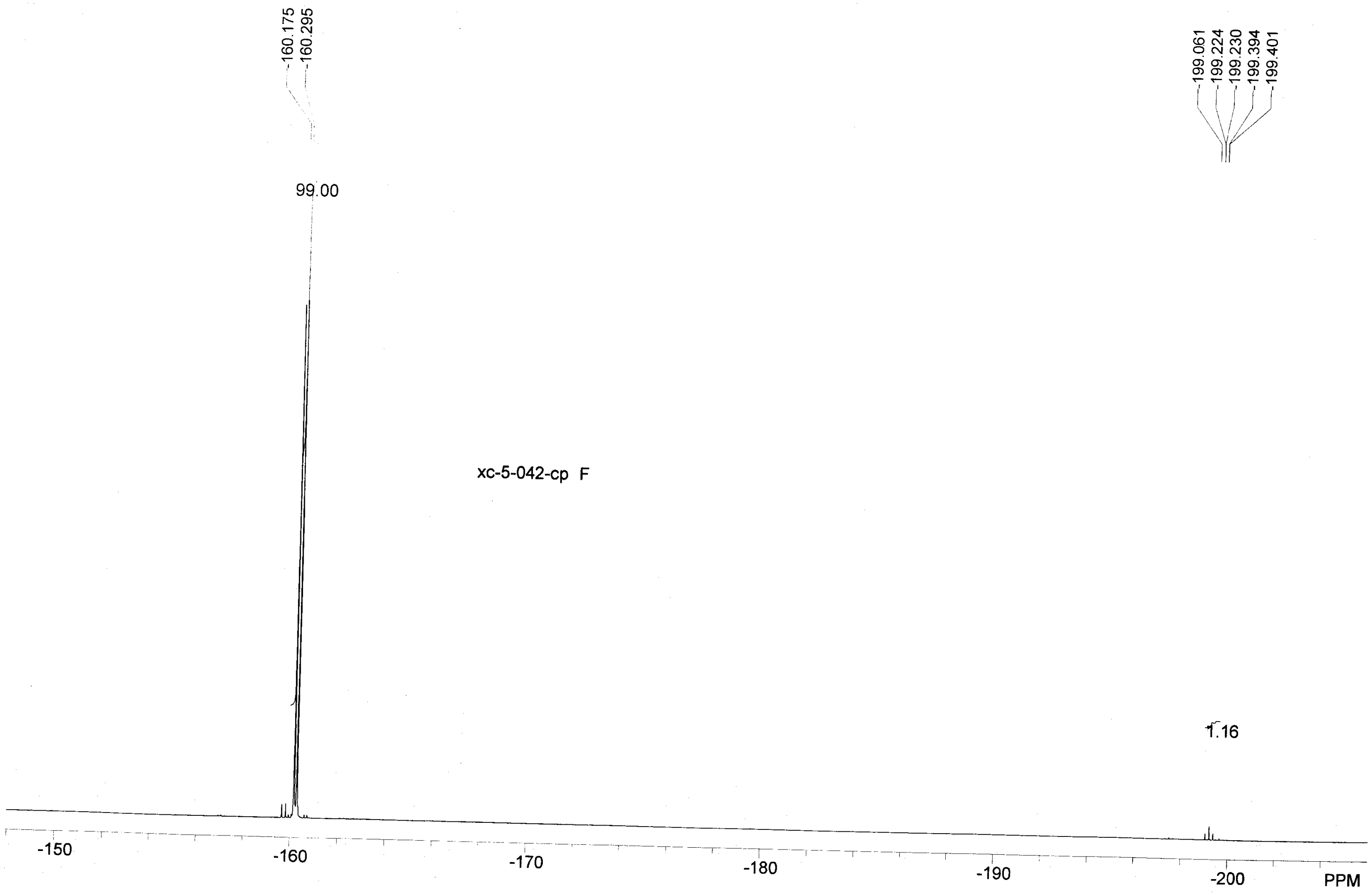
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-160
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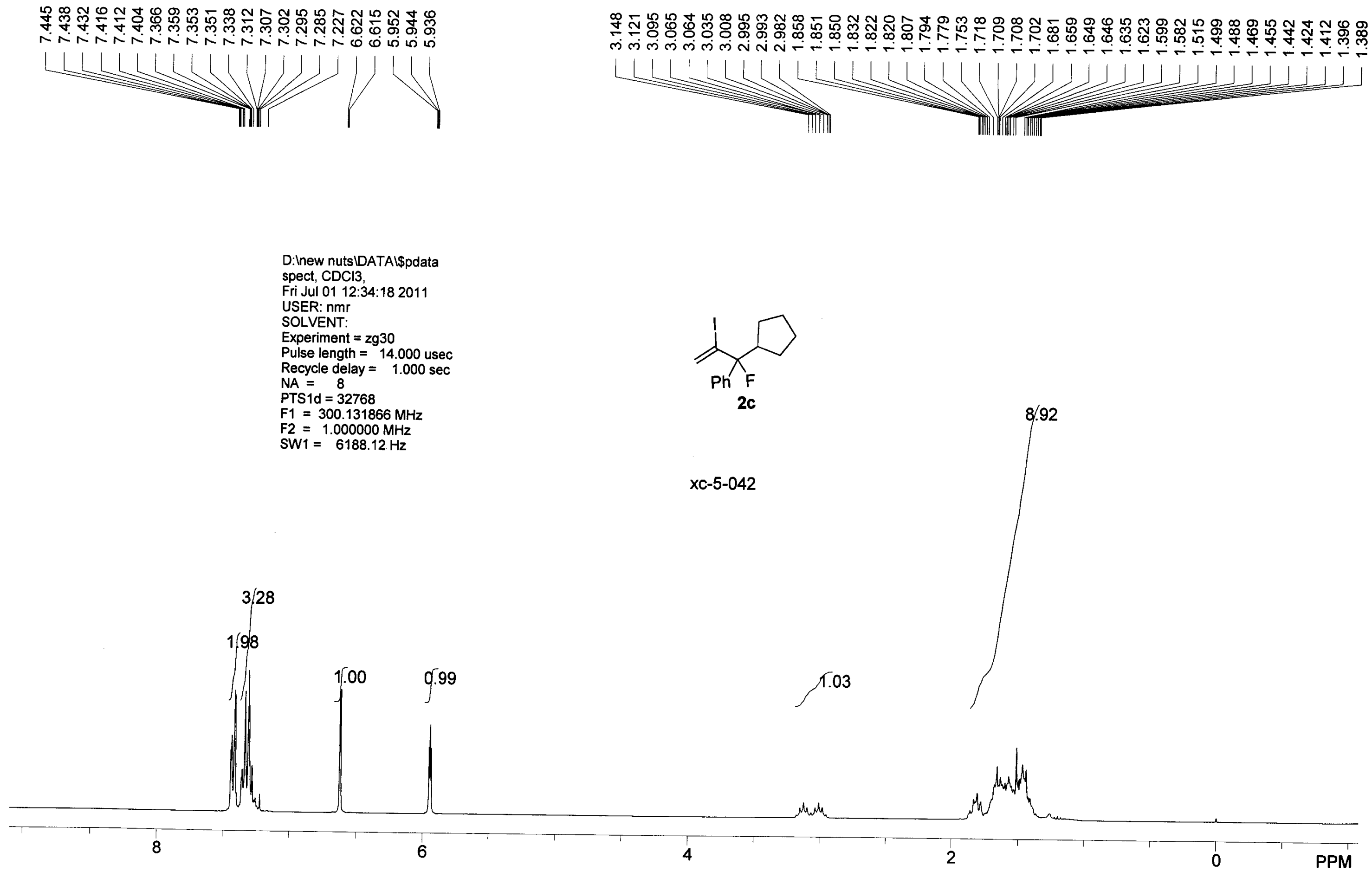
-190
-200
PPM



2c

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PTS1d = 32768
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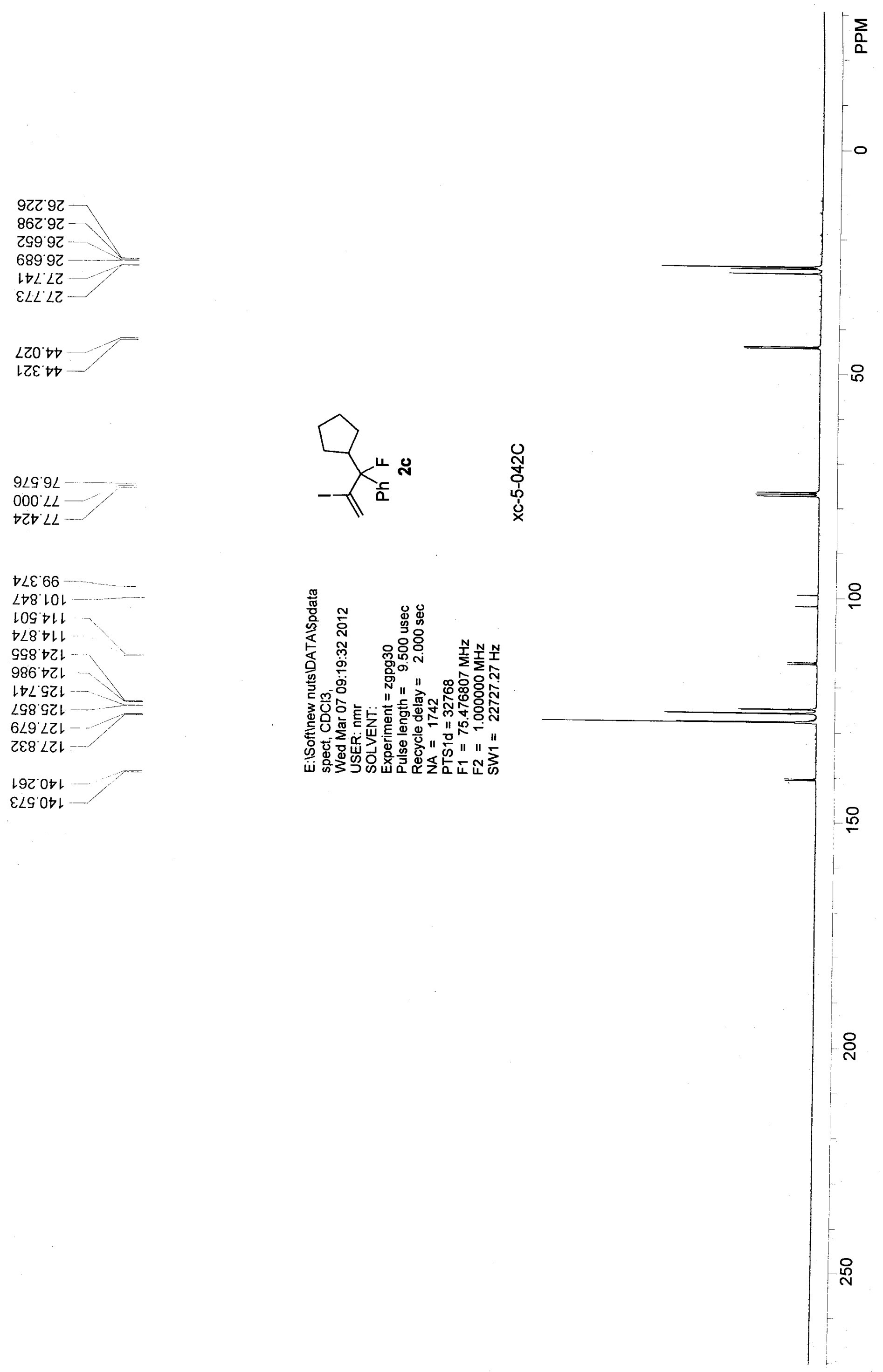
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

xc-5-042



2c

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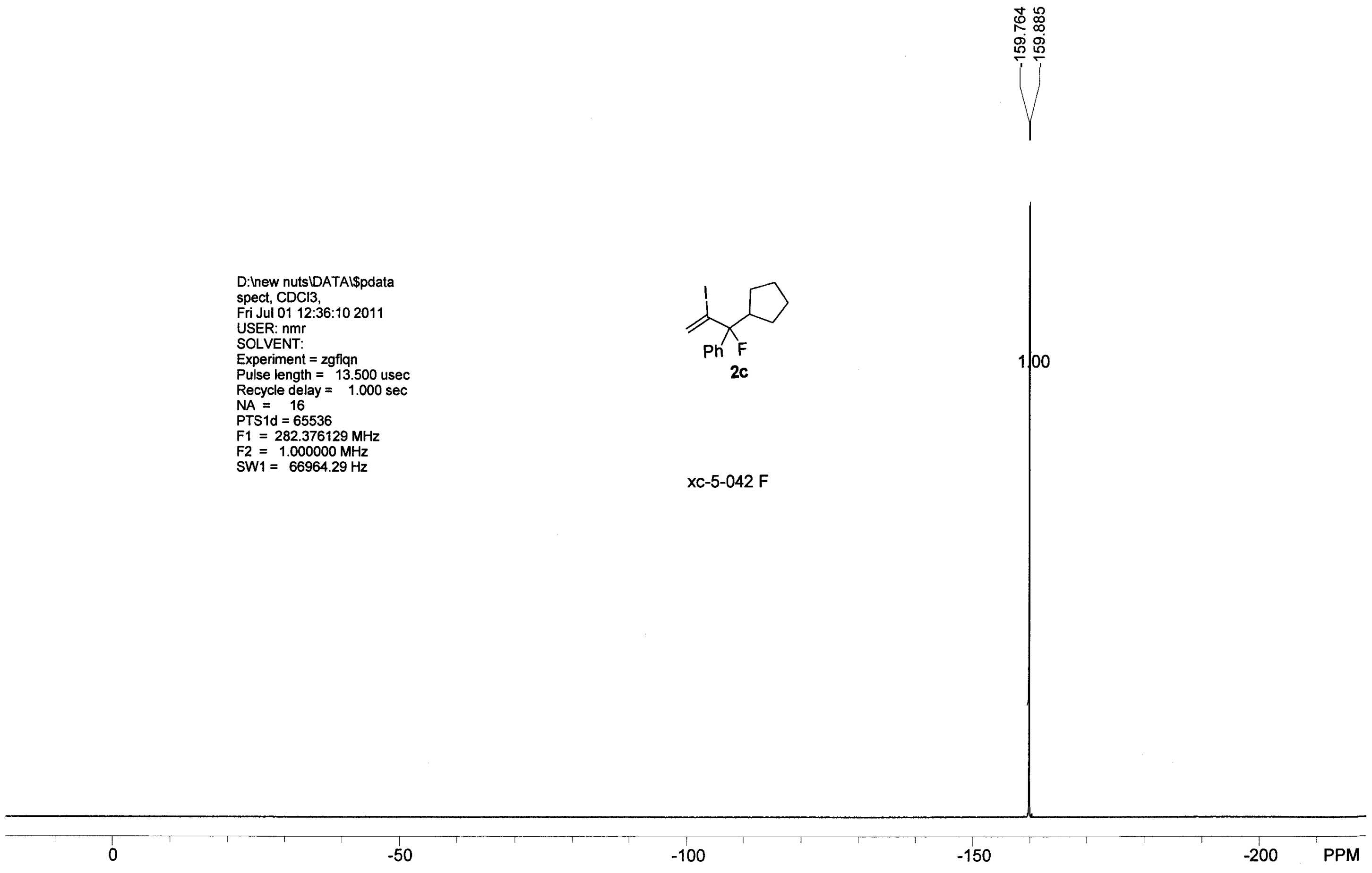
xc-5-042C



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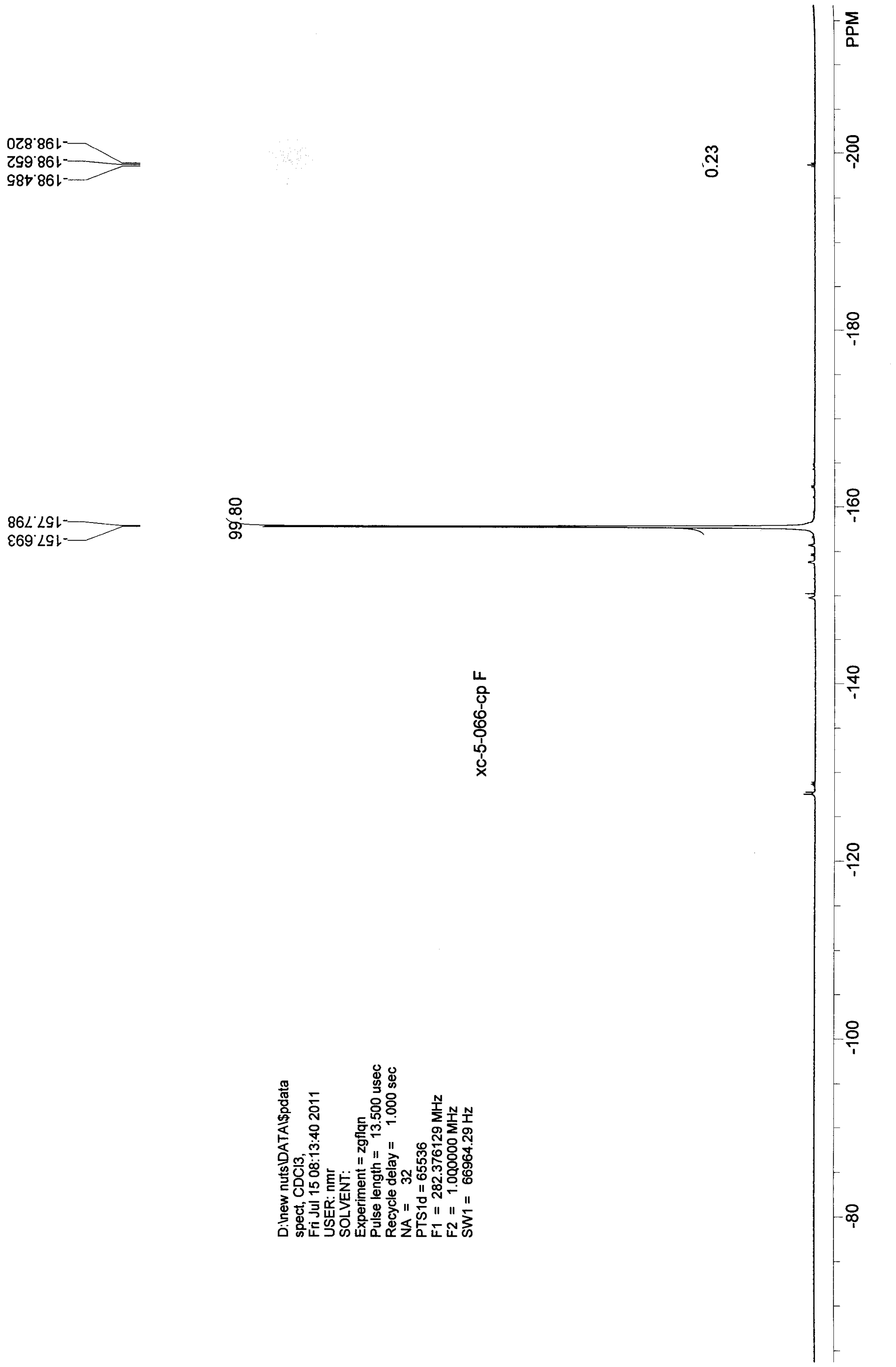
SW1 = 66964.29 Hz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99.80
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USER: nmr
SOLVENT:
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Recycle delay = 1.000 sec
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SW1 = 66964.29 Hz
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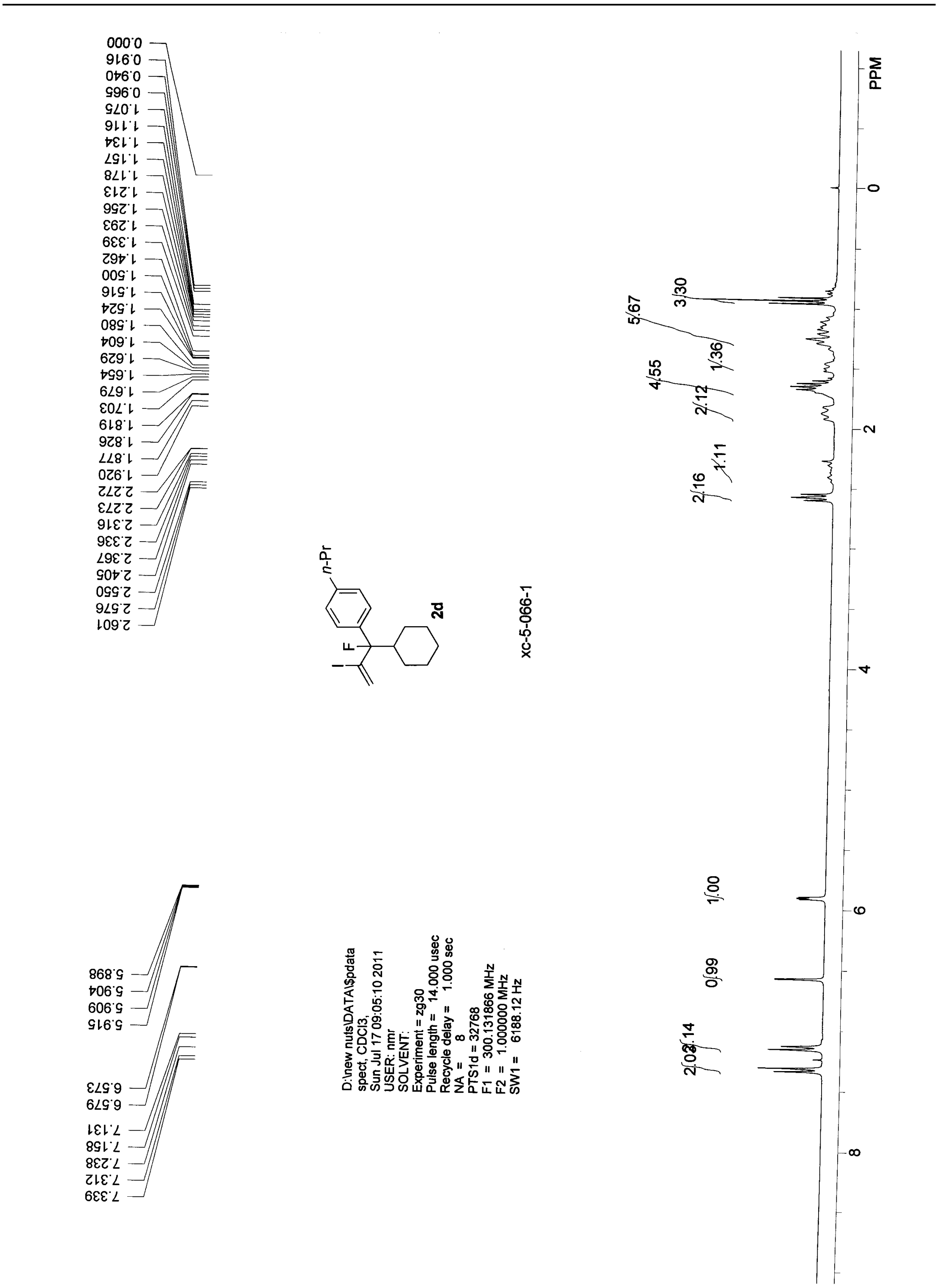
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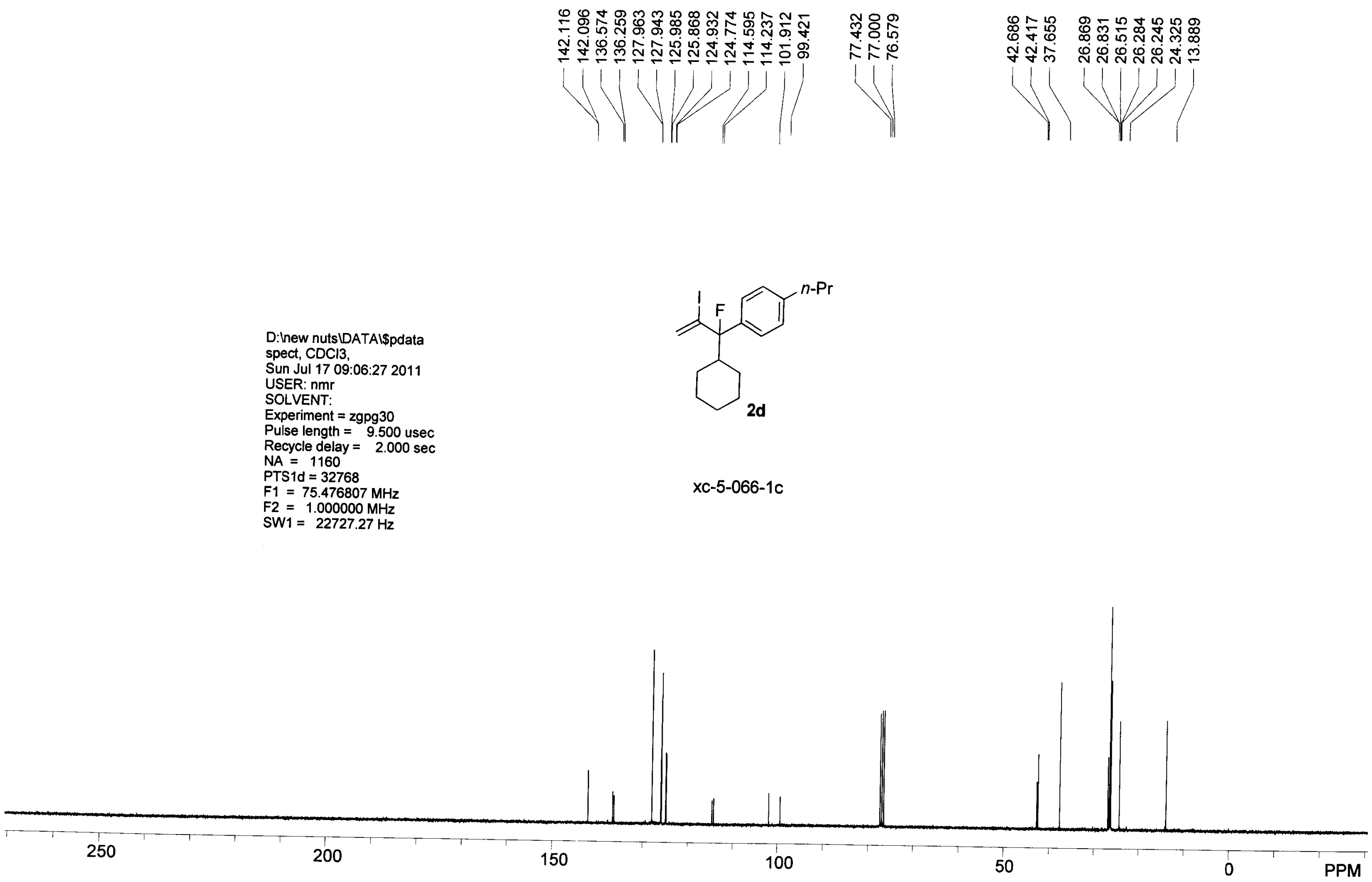
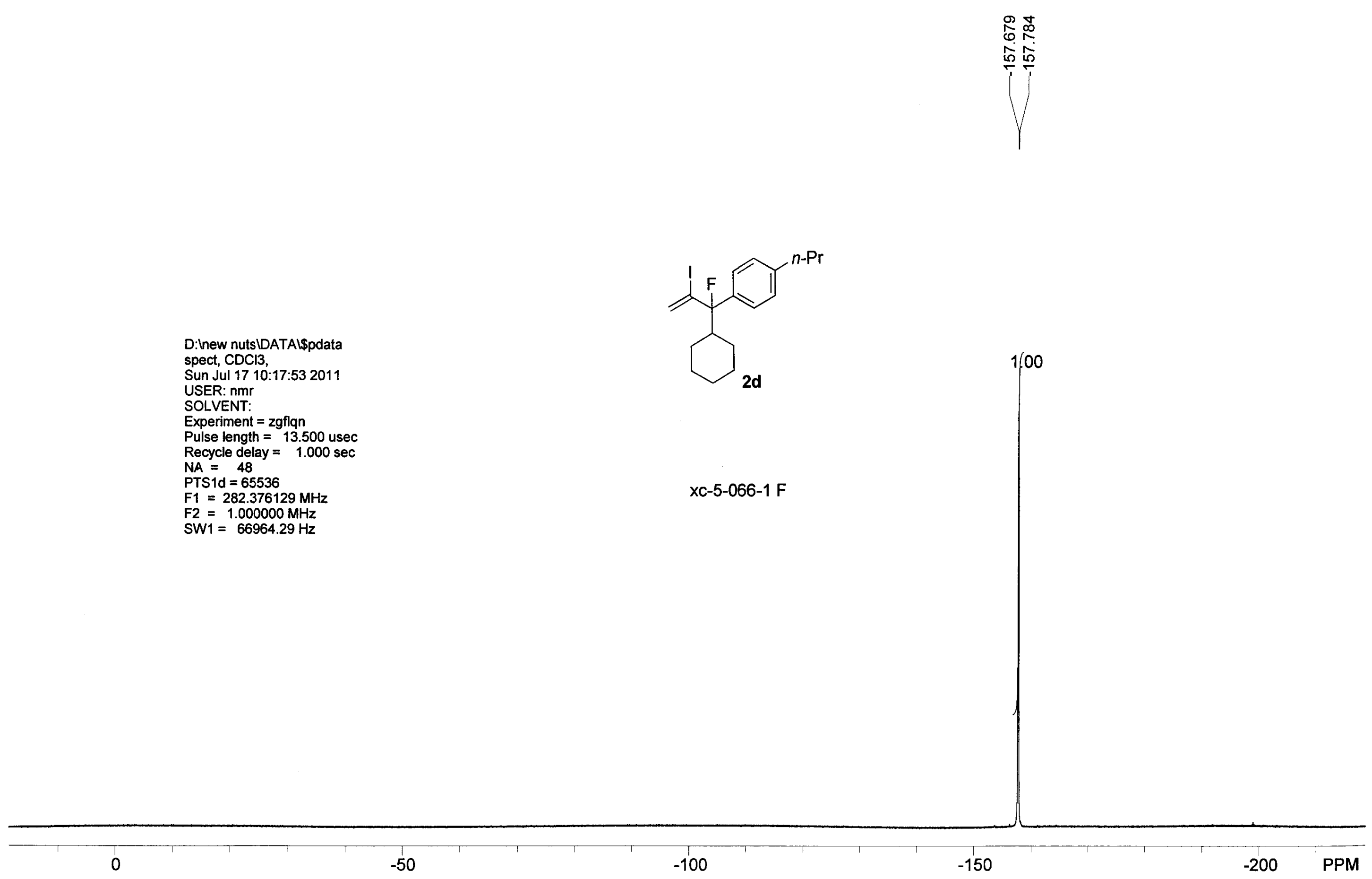
1.157
1.178
1.213
1.256
1.293
1.339
1.462
1.500
1.516
1.524
1.580
1.604
1.629
1.654
1.679
1.703
1.819
1.826
1.877
1.920
2.272
2.273
2.316
2.336
2.367
2.405
2.550
2.576
2.601
5.898
5.904
5.909
5.915
6.573
6.579
7.131
7.158
7.238
7.312
7.339
n-Pr
F
I
2d
xc-5-066-1
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Jul 17 09:05:10 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5.67
3.30
4.55
1.36
2.12
1.11
2.16
1.00
0.99
2.02
2.14
0
2
4
6
8
PPM



2d

xc-5-066-1c

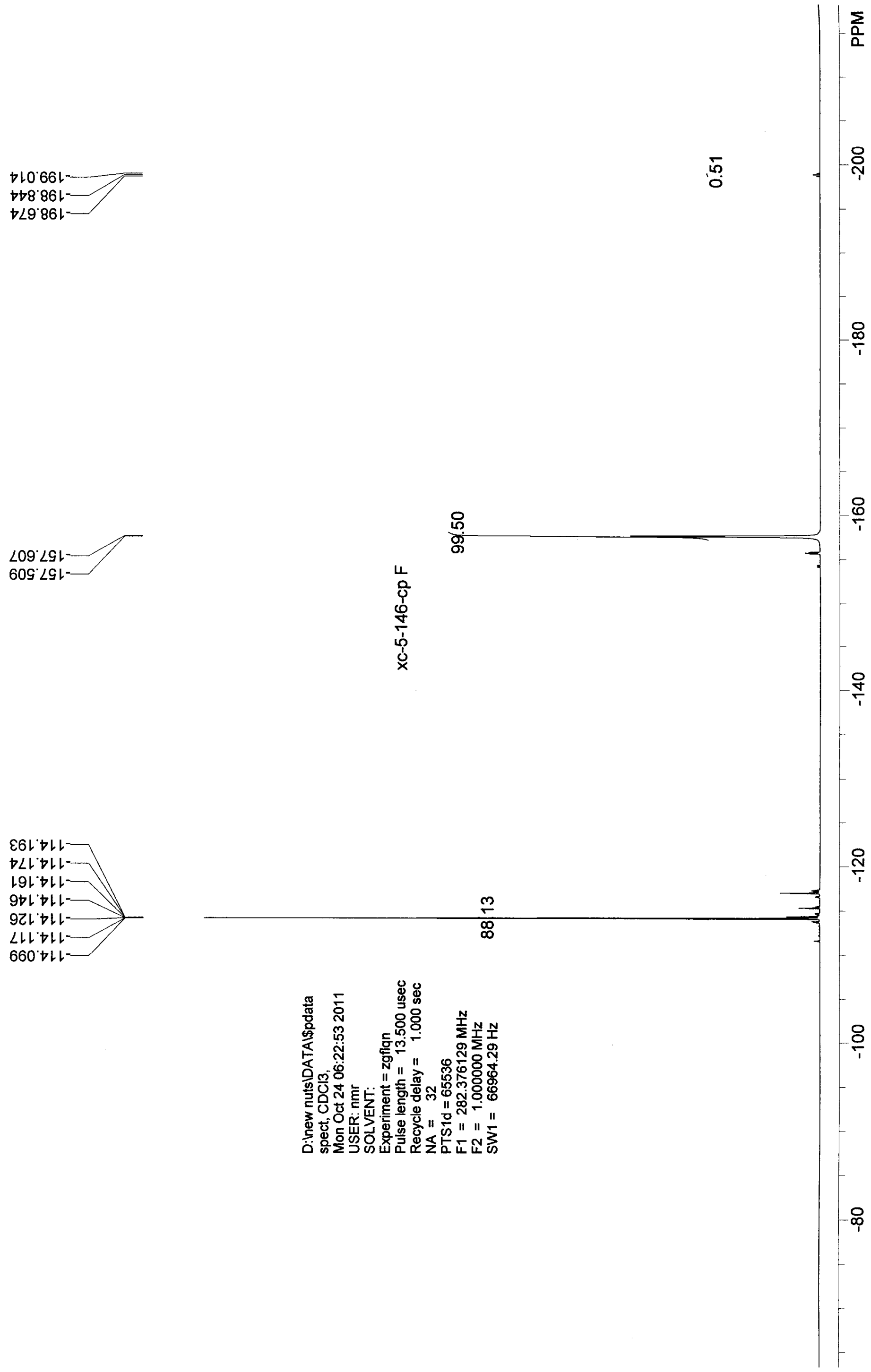
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Jul 17 09:06:27 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1160
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

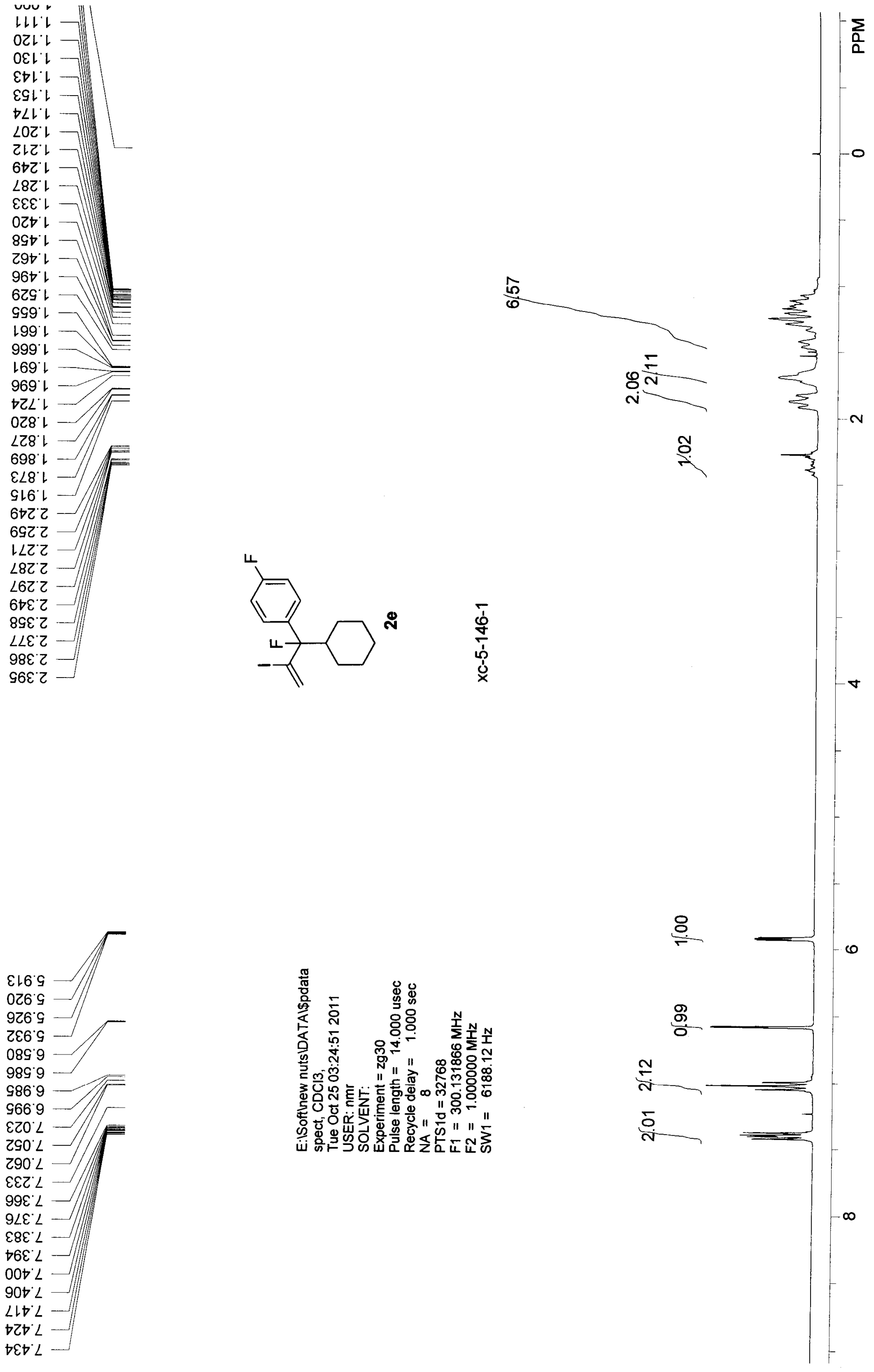


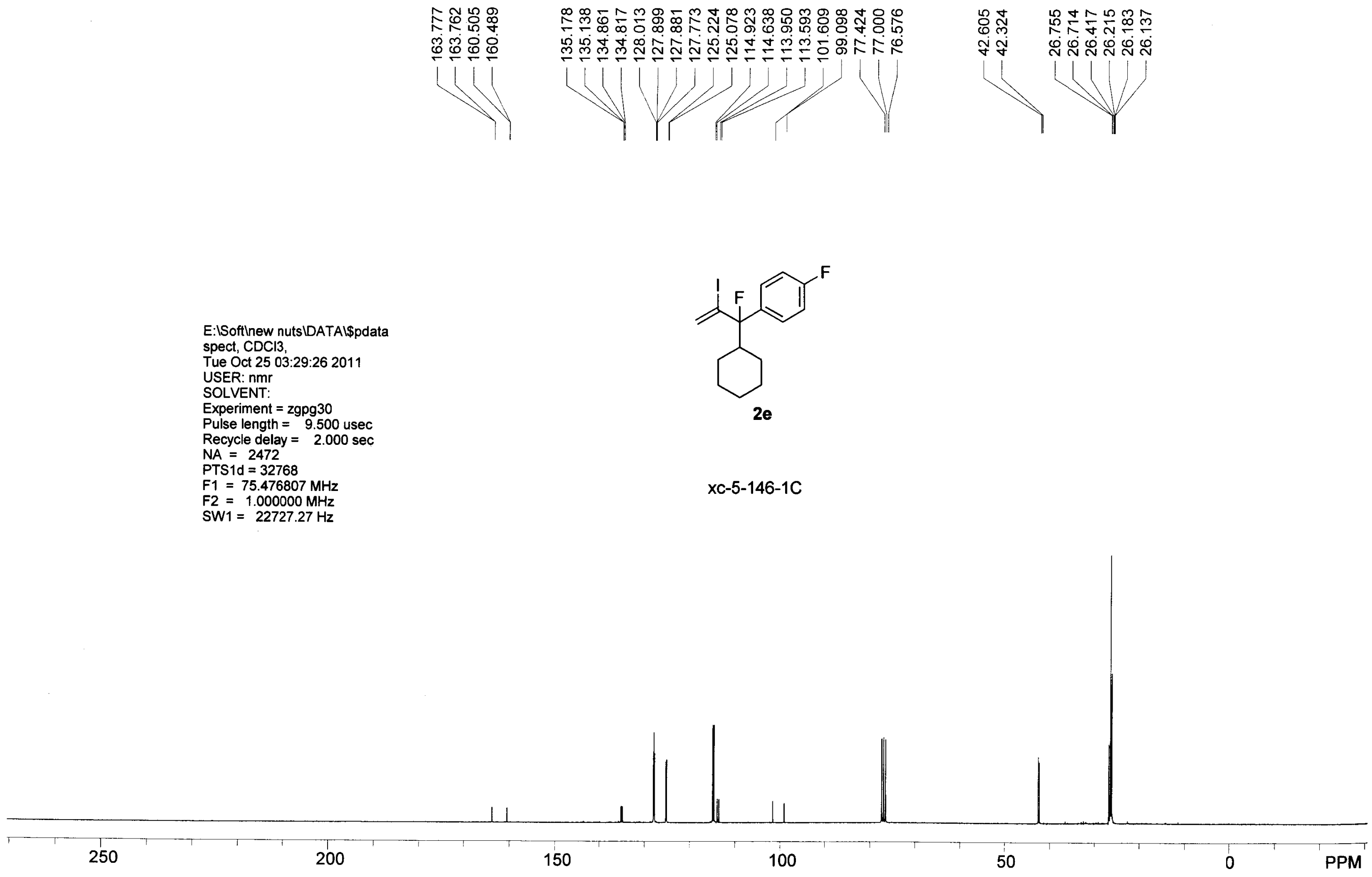
2d

xc-5-066-1 F

D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Jul 17 10:17:53 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 48
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz


xc-5-146-cp F
-199.014
-198.844
-198.674
-157.607
-157.509
-114.193
-114.174
-114.161
-114.146
-114.126
-114.117
-114.099
0.51
99.50
88.13
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Oct 24 06:22:53 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-200
-180
-160
-140
-120
-100
-80

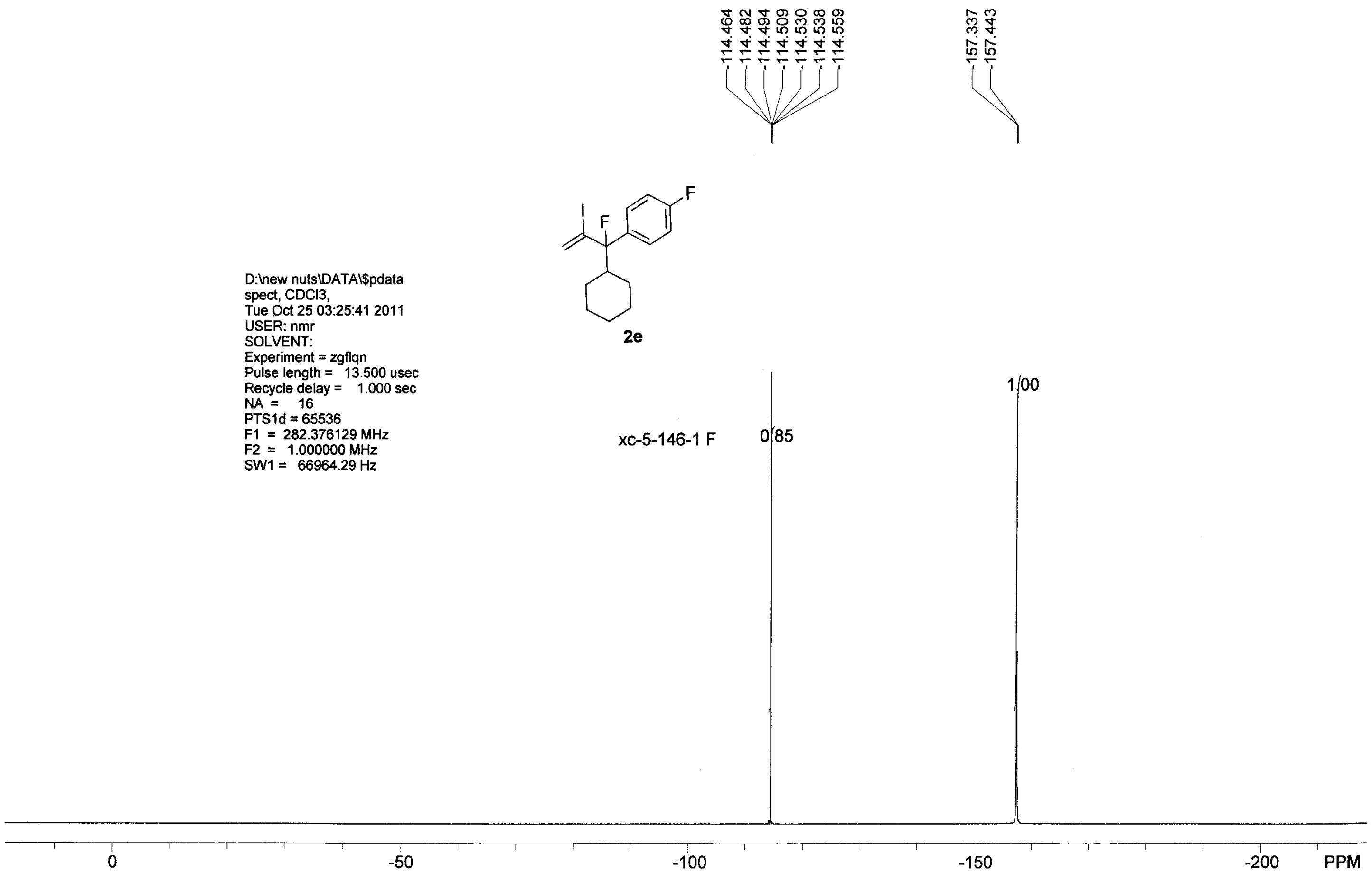

1.111
1.120
1.130
1.143
1.153
1.174
1.207
1.212
1.249
1.287
1.333
1.420
1.458
1.462
1.496
1.529
1.655
1.661
1.666
1.691
1.696
1.724
1.820
1.827
1.869
1.873
1.915
2.249
2.259
2.271
2.287
2.297
2.349
2.358
2.377
2.386
2.395
5.913
5.920
5.926
5.932
6.580
6.586
6.985
6.995
7.023
7.062
7.233
7.366
7.376
7.383
7.394
7.400
7.406
7.417
7.424
7.434
F
I
F
2e
xc-5-146-1
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Tue Oct 25 03:24:51 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.57
2.06
2.11
1.02
1.00
0.99
2.12
2.01
PPM
0
2
4
6
8



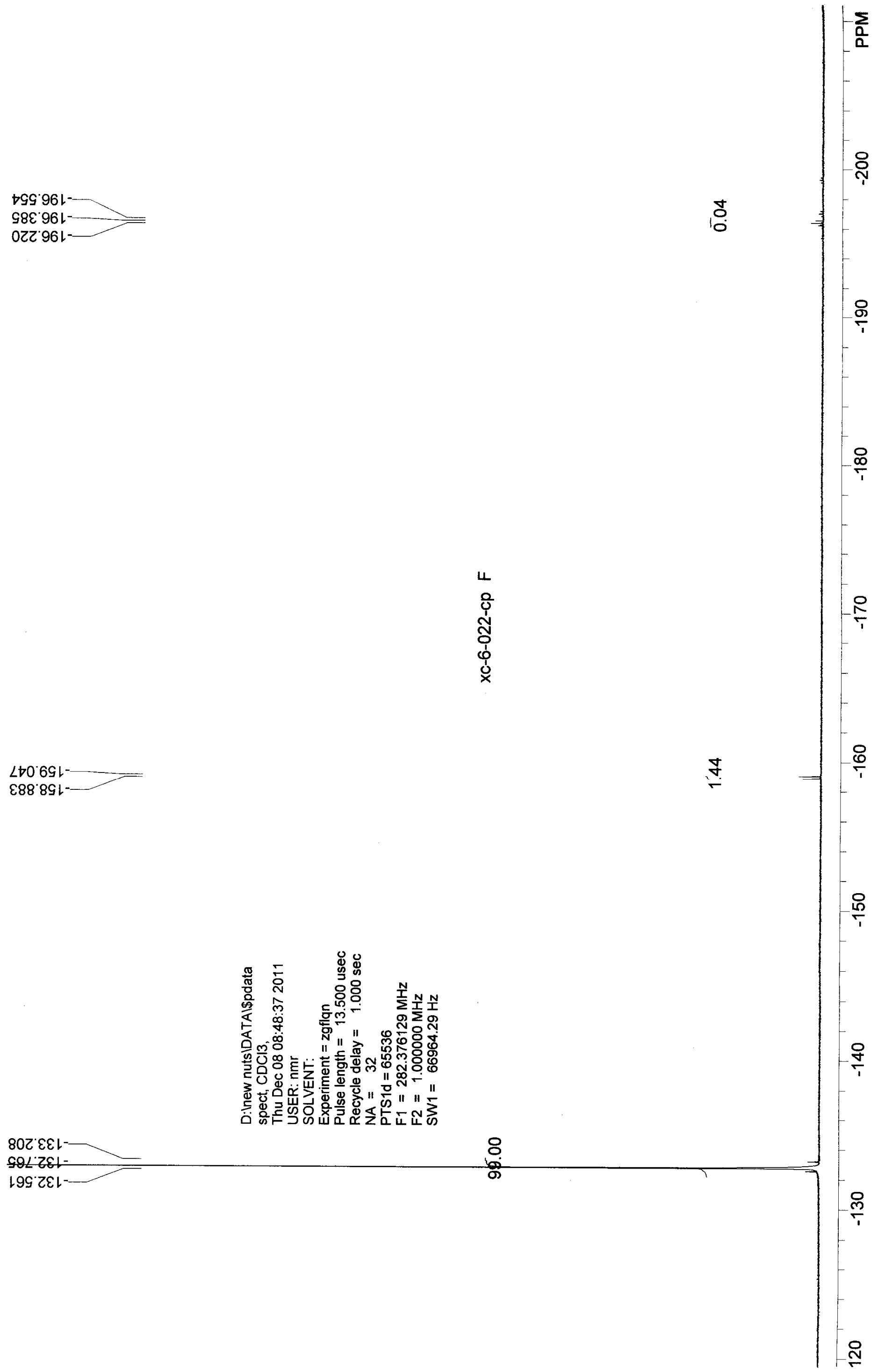
2e

xc-5-146-1C

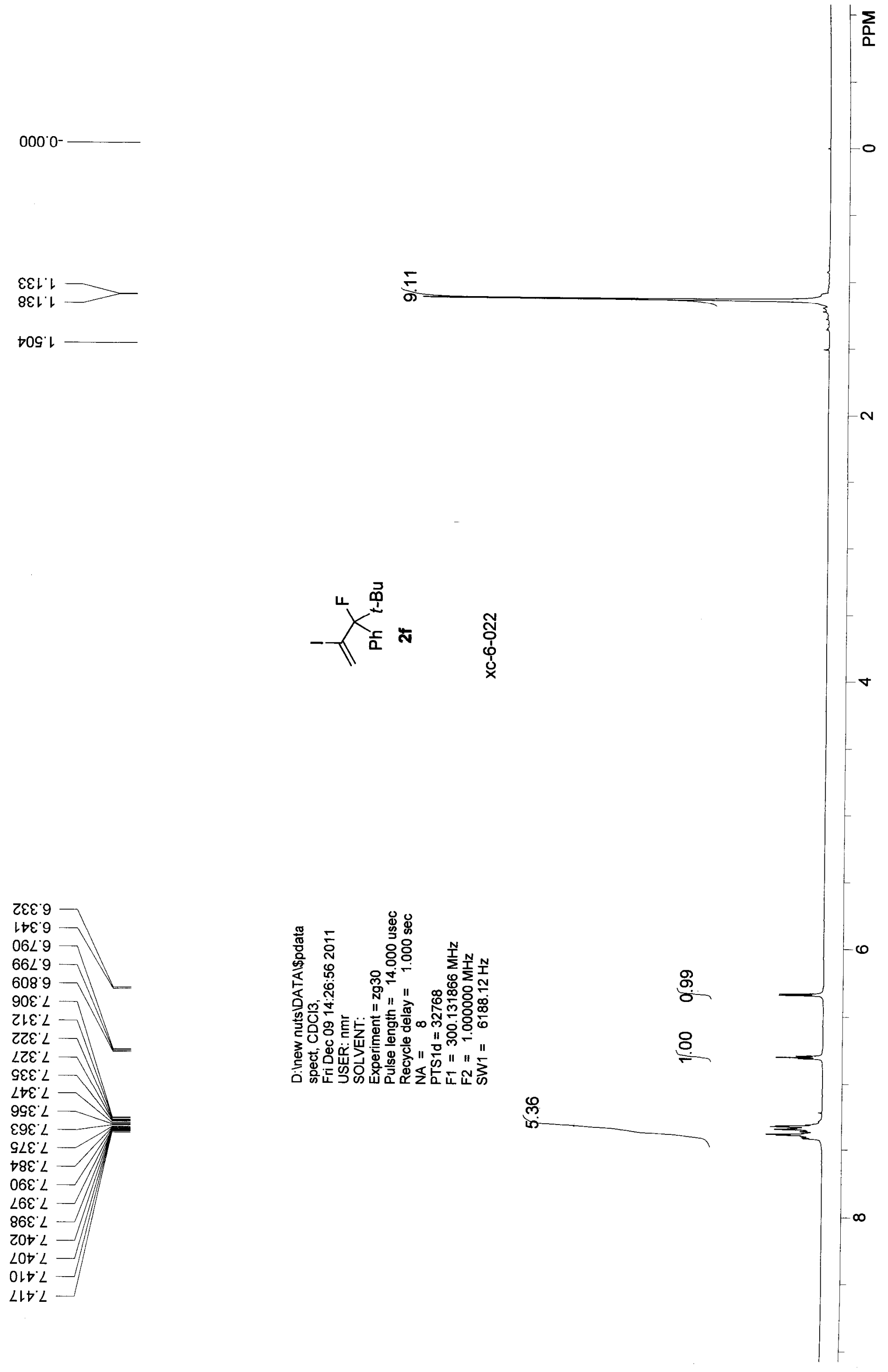
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Tue Oct 25 03:29:26 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2472
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

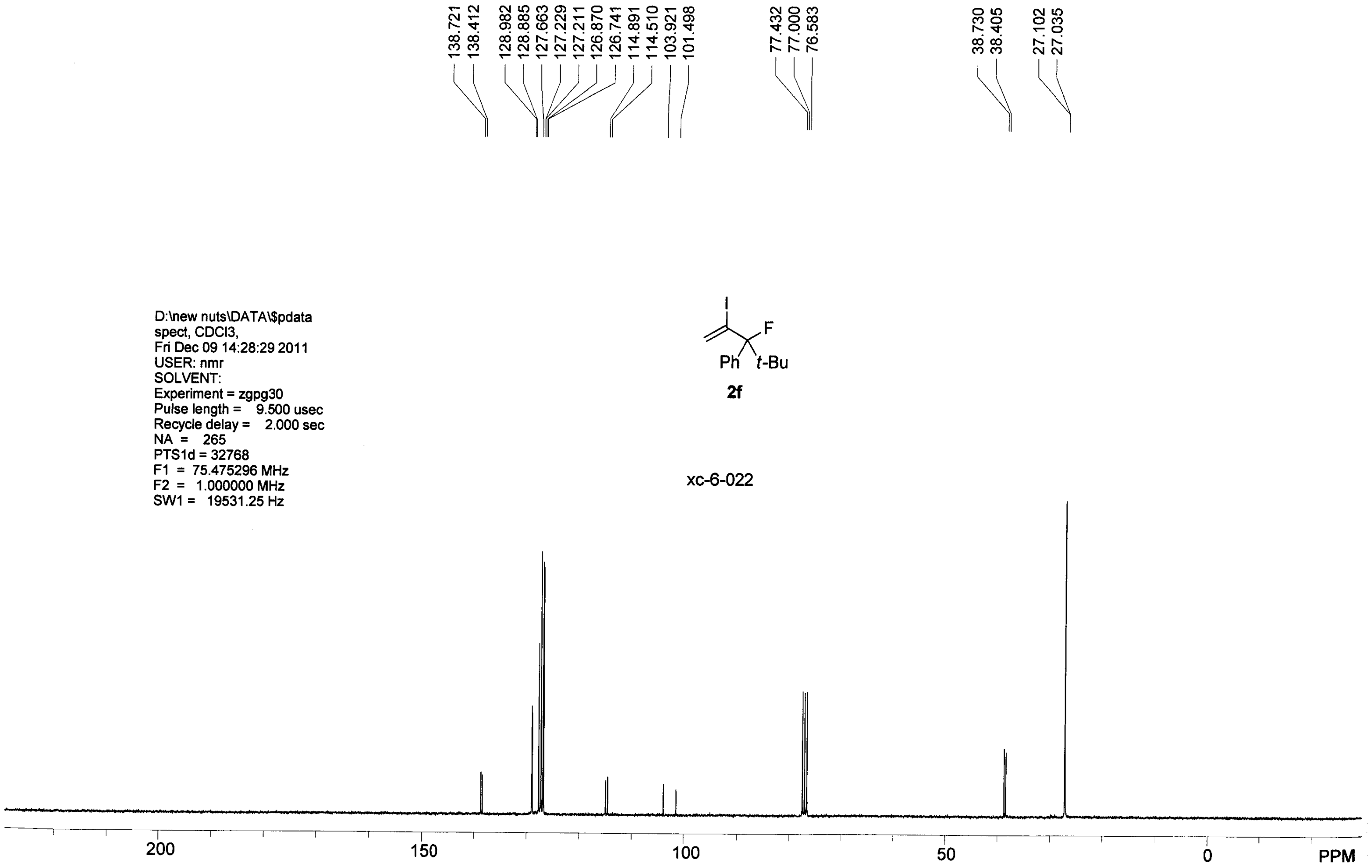


```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Oct 25 03:25:41 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =   13.500 usec
Recycle delay =    1.000 sec
NA =     16
PTS1d = 65536
F1  =  282.376129 MHz
F2  =   1.000000 MHz
SW1 =   66964.29 Hz
```



D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 08 08:48:37 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz

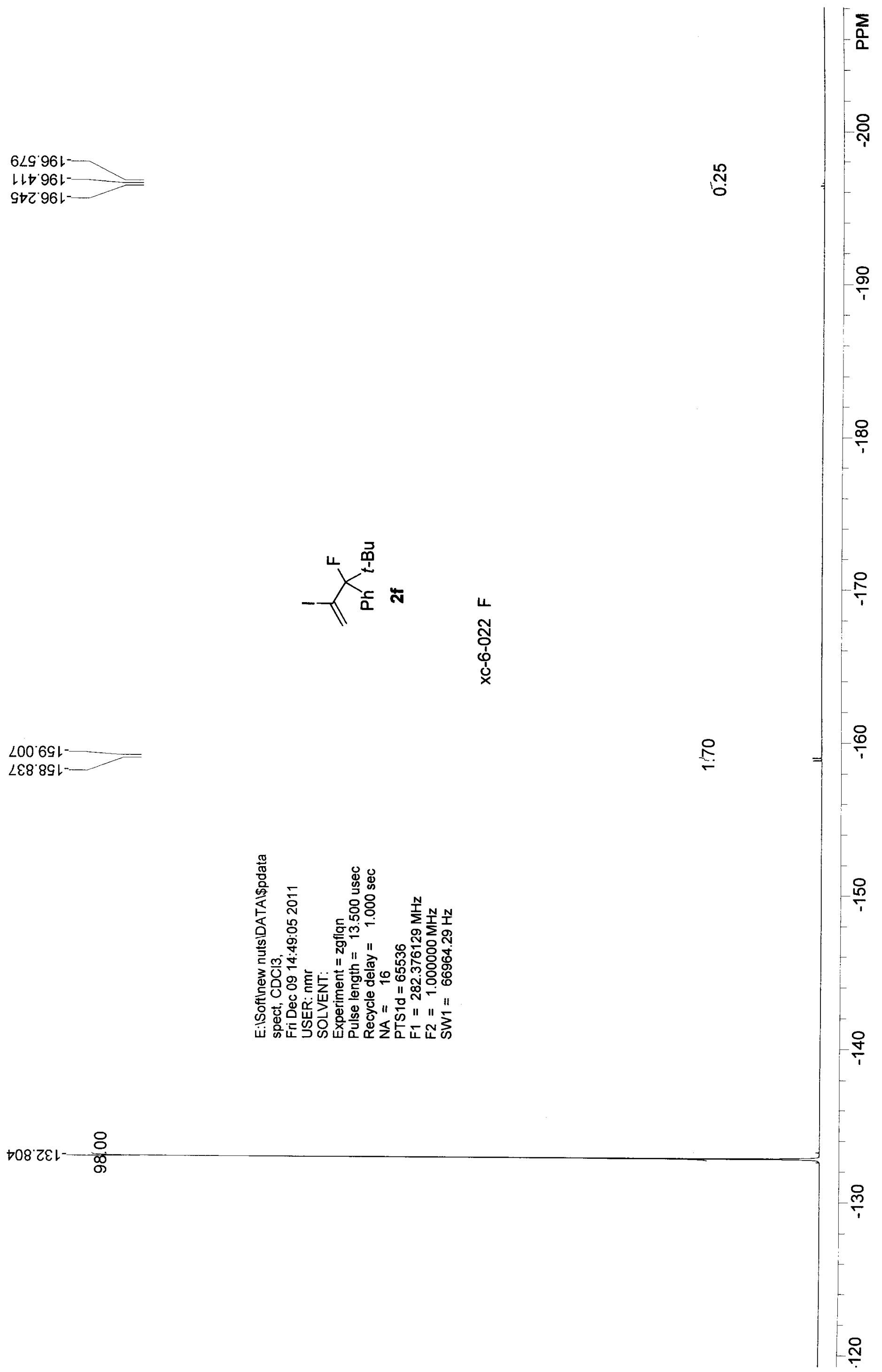

-0.000
1.133
1.138
1.504
6.332
6.341
6.790
6.799
6.809
7.306
7.312
7.322
7.327
7.335
7.347
7.356
7.363
7.375
7.384
7.390
7.397
7.398
7.402
7.407
7.410
7.417
9.11
5.36
1.00
0.99
F
Ph
t-Bu
I
2f
xc-6-022
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Dec 09 14:26:56 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
PPM
0
2
4
6
8



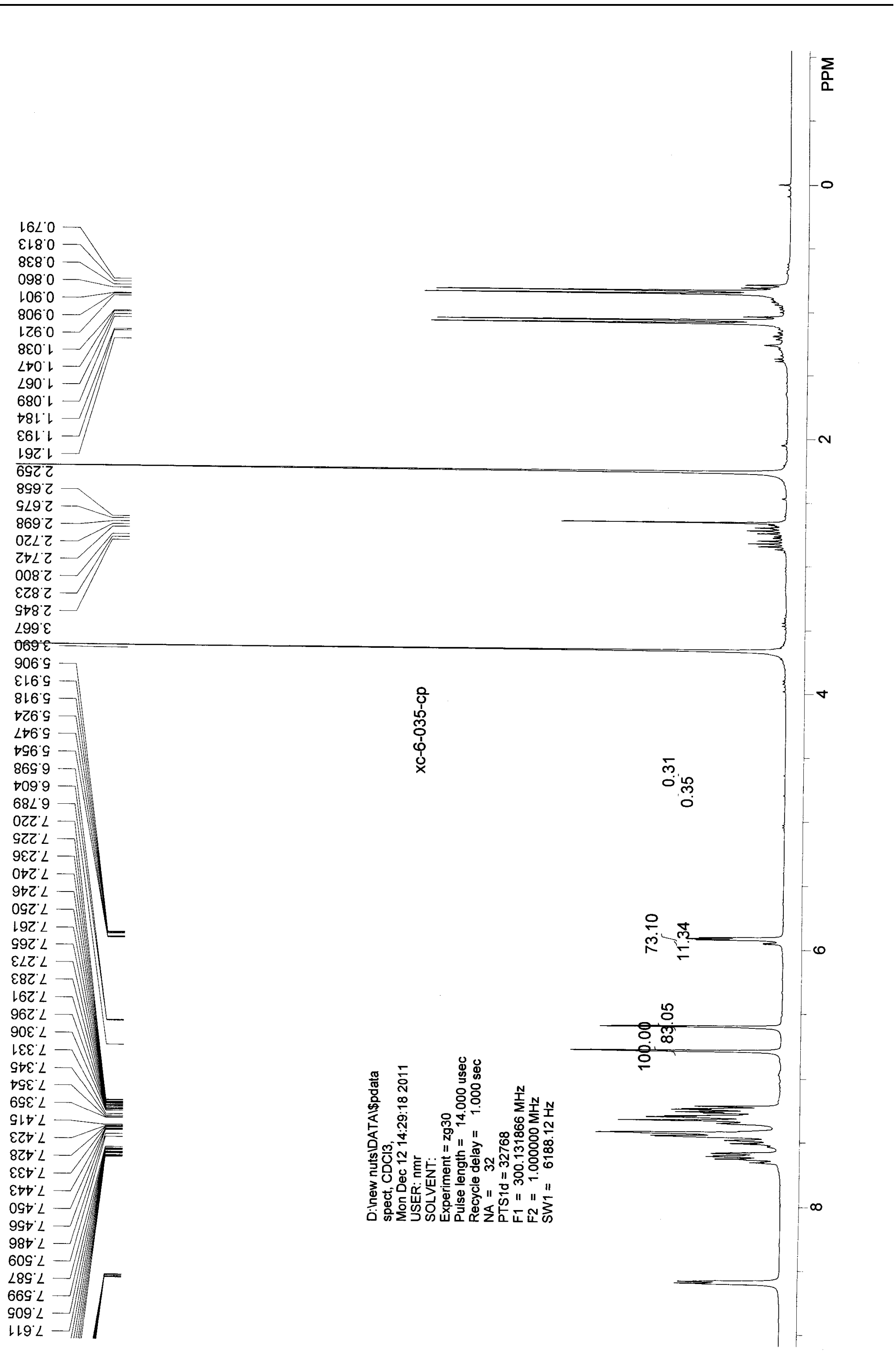
2f

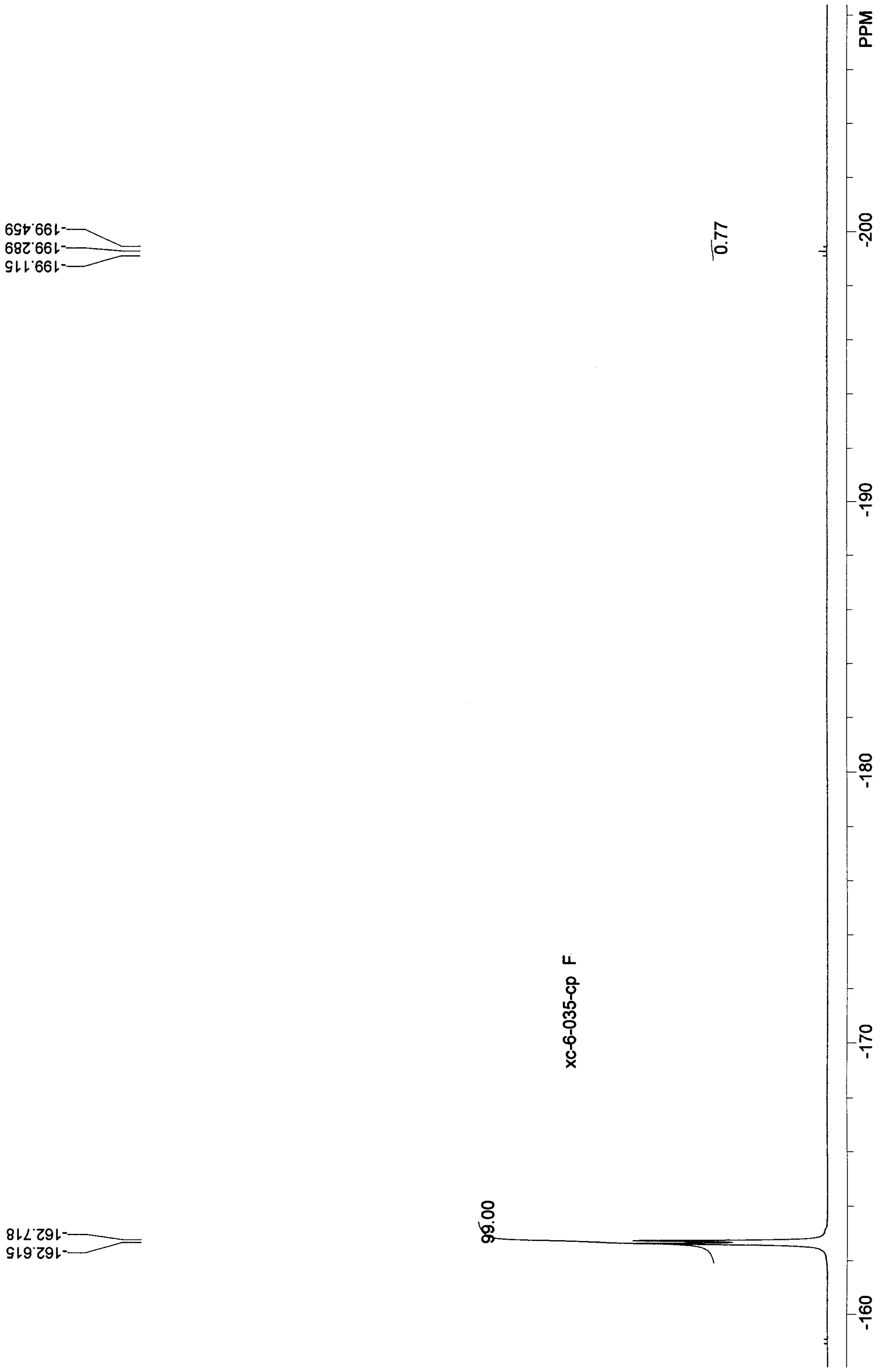
xc-6-022

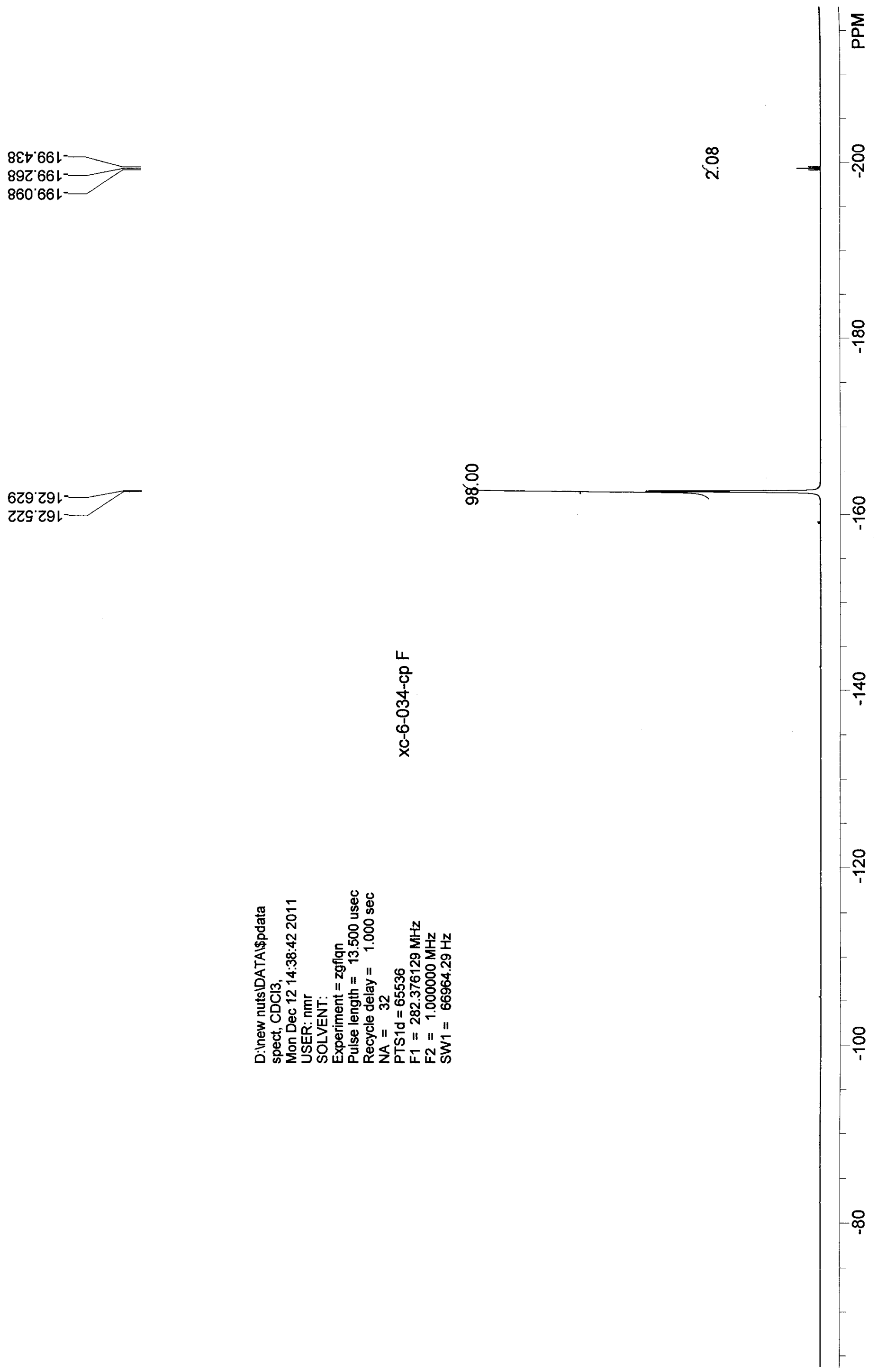
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Dec 09 14:28:29 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 265
PTS1d = 32768
F1 = 75.475296 MHz
F2 = 1.000000 MHz
SW1 = 19531.25 Hz

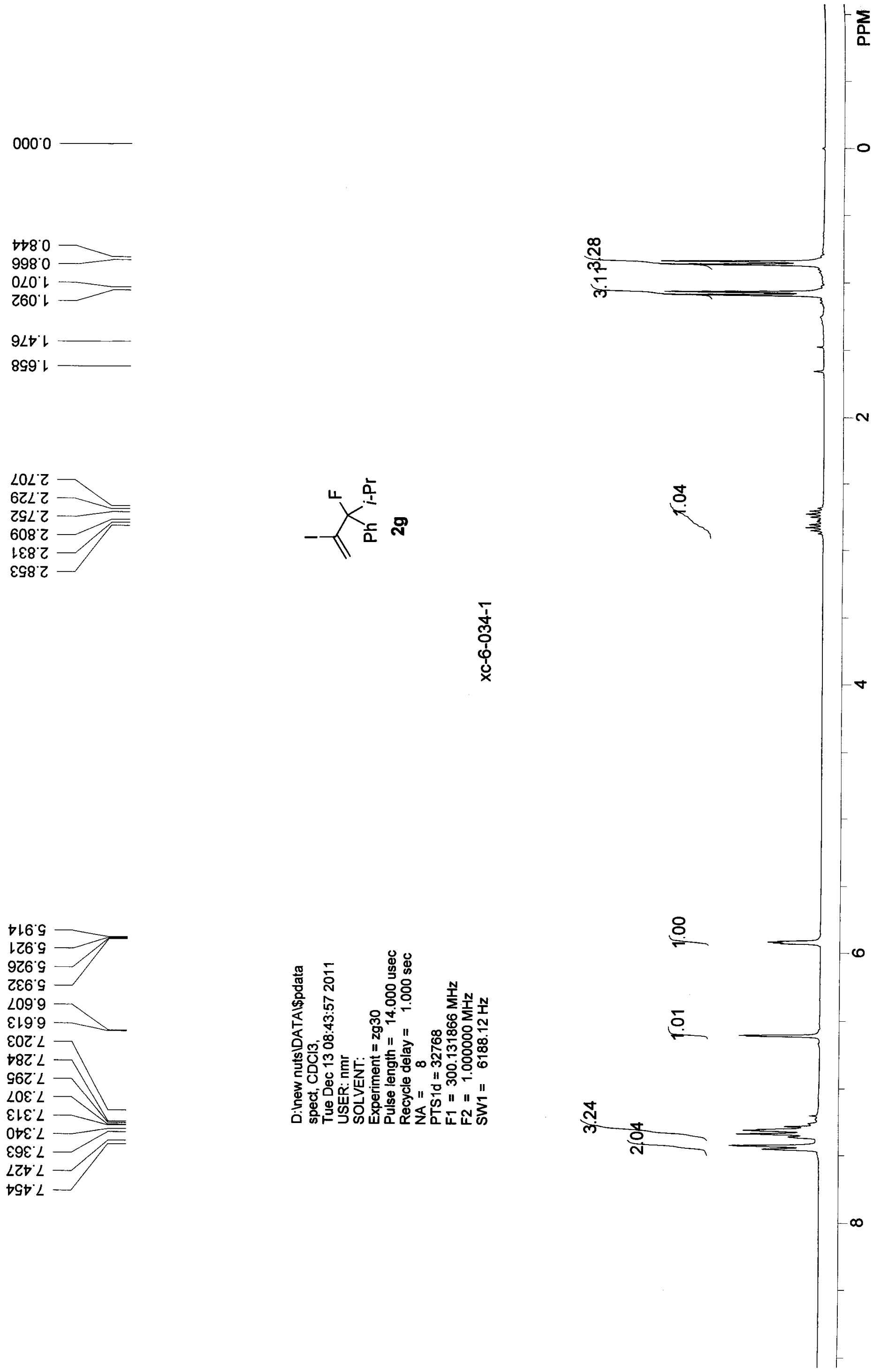


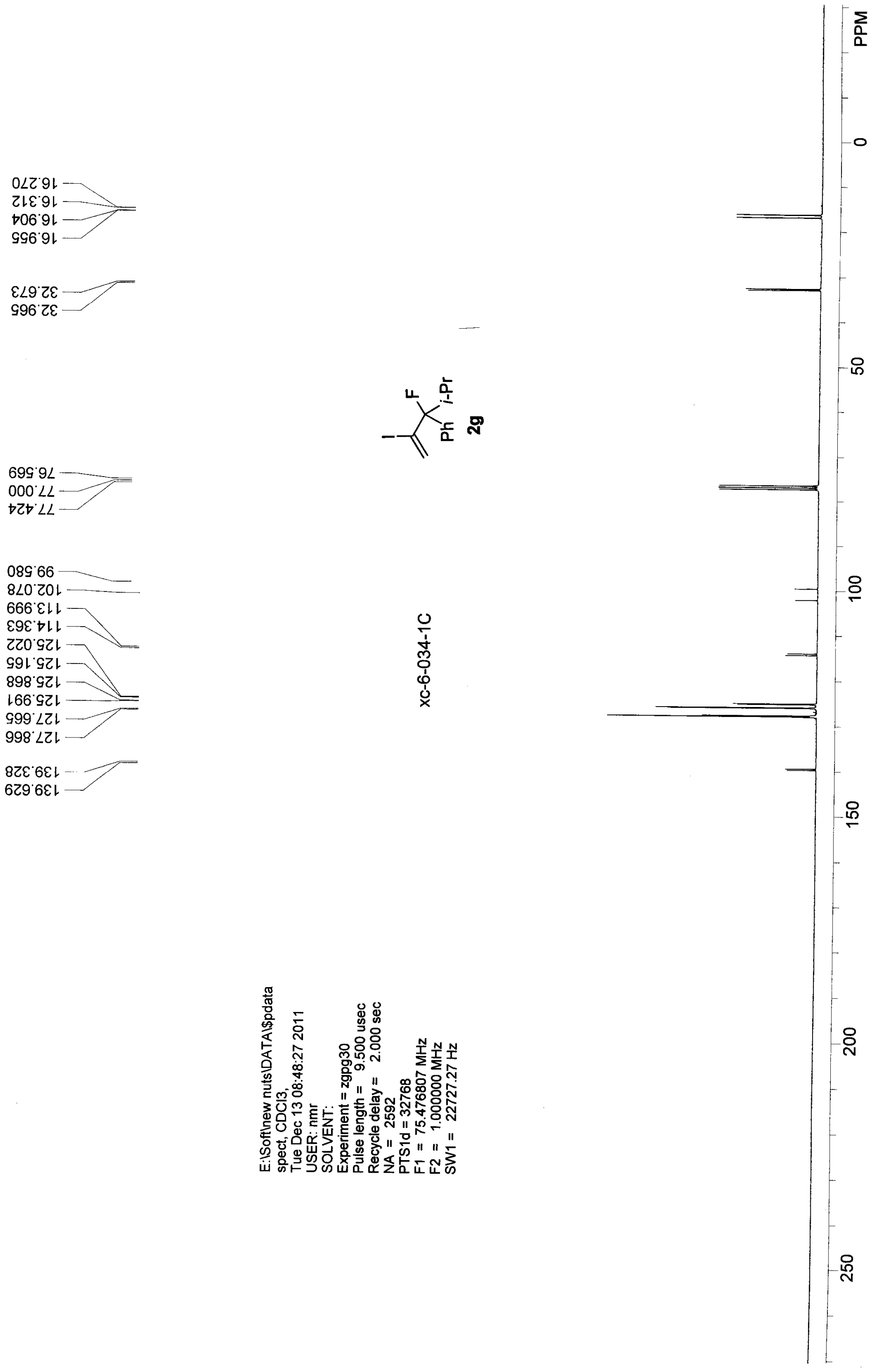
```
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Fri Dec 09 14:49:05 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =  13.500 usec
Recycle delay =   1.000 sec
NA =   16
PTS1d = 65536
F1  =  282.376129 MHz
F2  =   1.000000 MHz
SW1 =  66964.29 Hz
```


xc-6-035-cp
0.791
0.813
0.838
0.860
0.901
0.908
0.921
1.038
1.047
1.067
1.089
1.184
1.193
1.261
2.259
2.658
2.675
2.698
2.720
2.742
2.800
2.823
2.845
3.667
3.690
5.906
5.913
5.918
5.924
5.947
5.954
6.598
6.604
6.789
7.220
7.225
7.236
7.240
7.246
7.250
7.261
7.265
7.273
7.283
7.291
7.296
7.306
7.331
7.345
7.354
7.359
7.415
7.423
7.428
7.433
7.443
7.450
7.456
7.486
7.509
7.587
7.599
7.605
7.611
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Dec 12 14:29:18 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.31
0.35
73.10
11.34
100.00
83.05
PPM
0
2
4
6
8

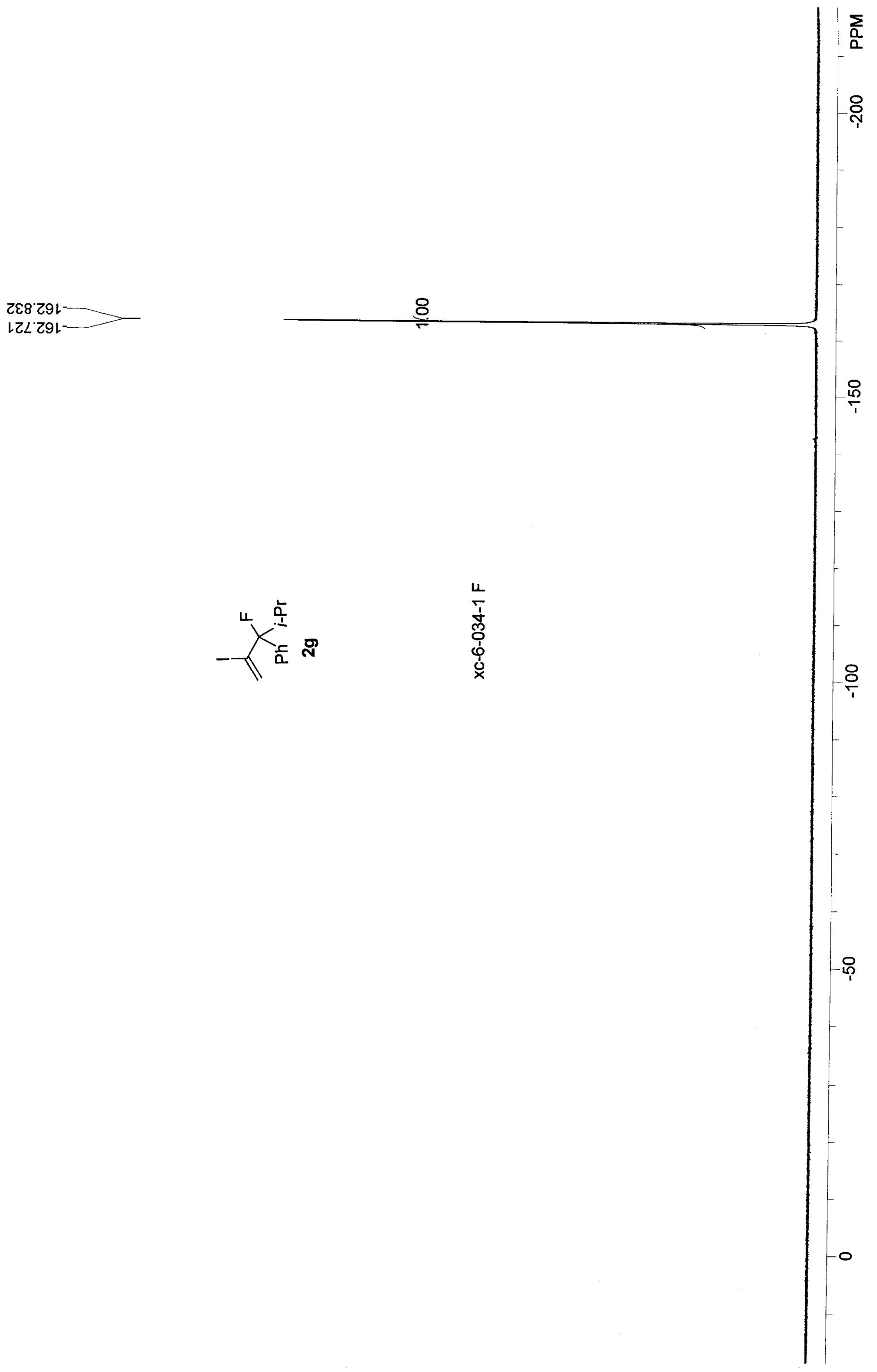

-199.459
-199.289
-199.115
0.77
xc-6-035-cp F
99.00
-162.718
-162.615
PPM
-200
-190
-180
-170
-160

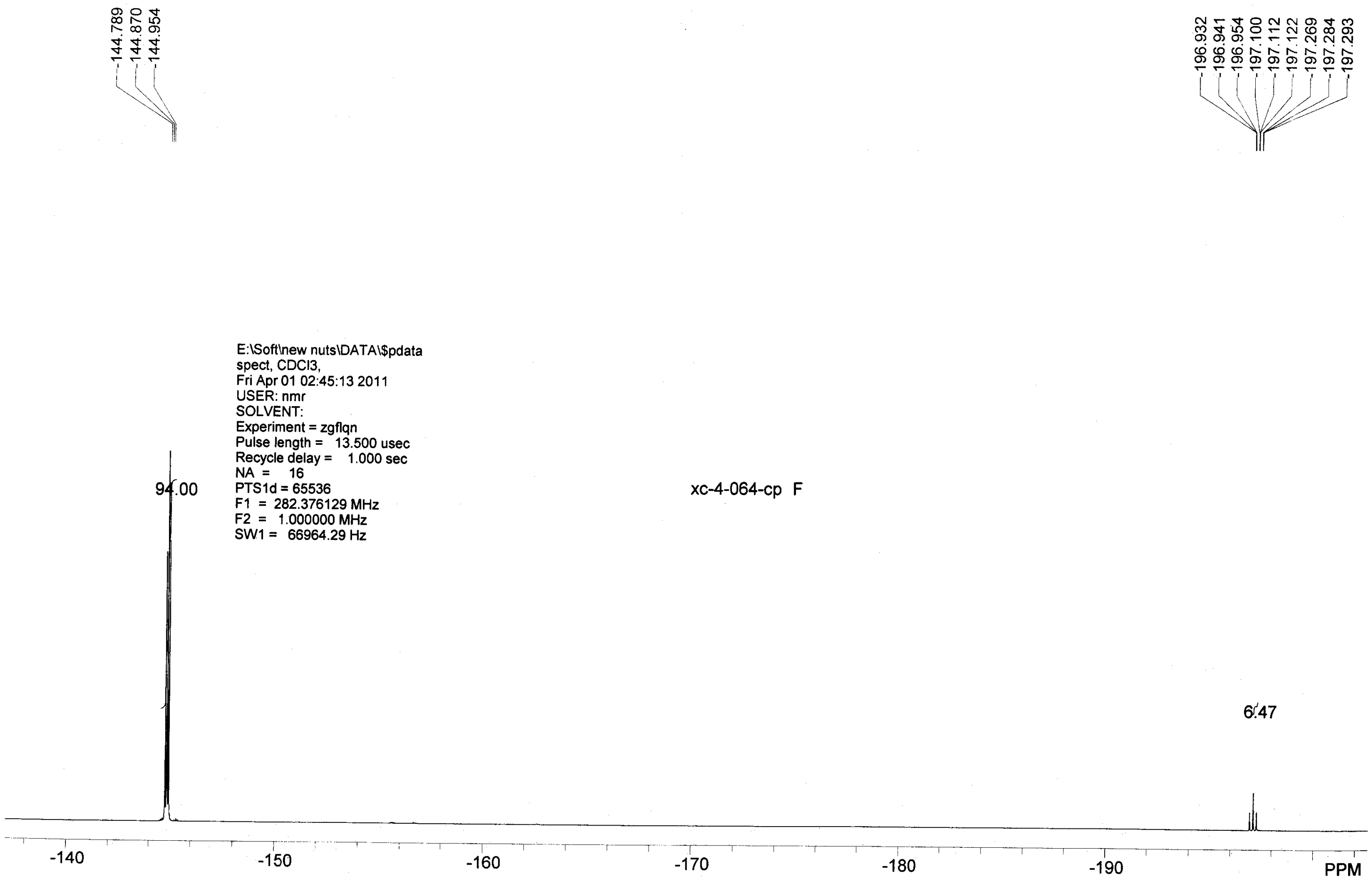

-199.438
-199.268
-199.098
-162.629
-162.522
2.08
98.00
xc-6-034-cp F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Dec 12 14:38:42 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-200
-180
-160
-140
-120
-100
-80


0.000
0.844
0.866
1.070
1.092
1.476
1.658
2.707
2.729
2.752
2.809
2.831
2.853
5.914
5.921
5.926
5.932
6.607
6.613
7.203
7.284
7.295
7.307
7.313
7.340
7.363
7.427
7.454
F
i-Pr
Ph
2g
xc-6-034-1
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Dec 13 08:43:57 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3.11
3.28
1.04
1.00
1.01
3.24
2.04
PPM
0
2
4
6
8



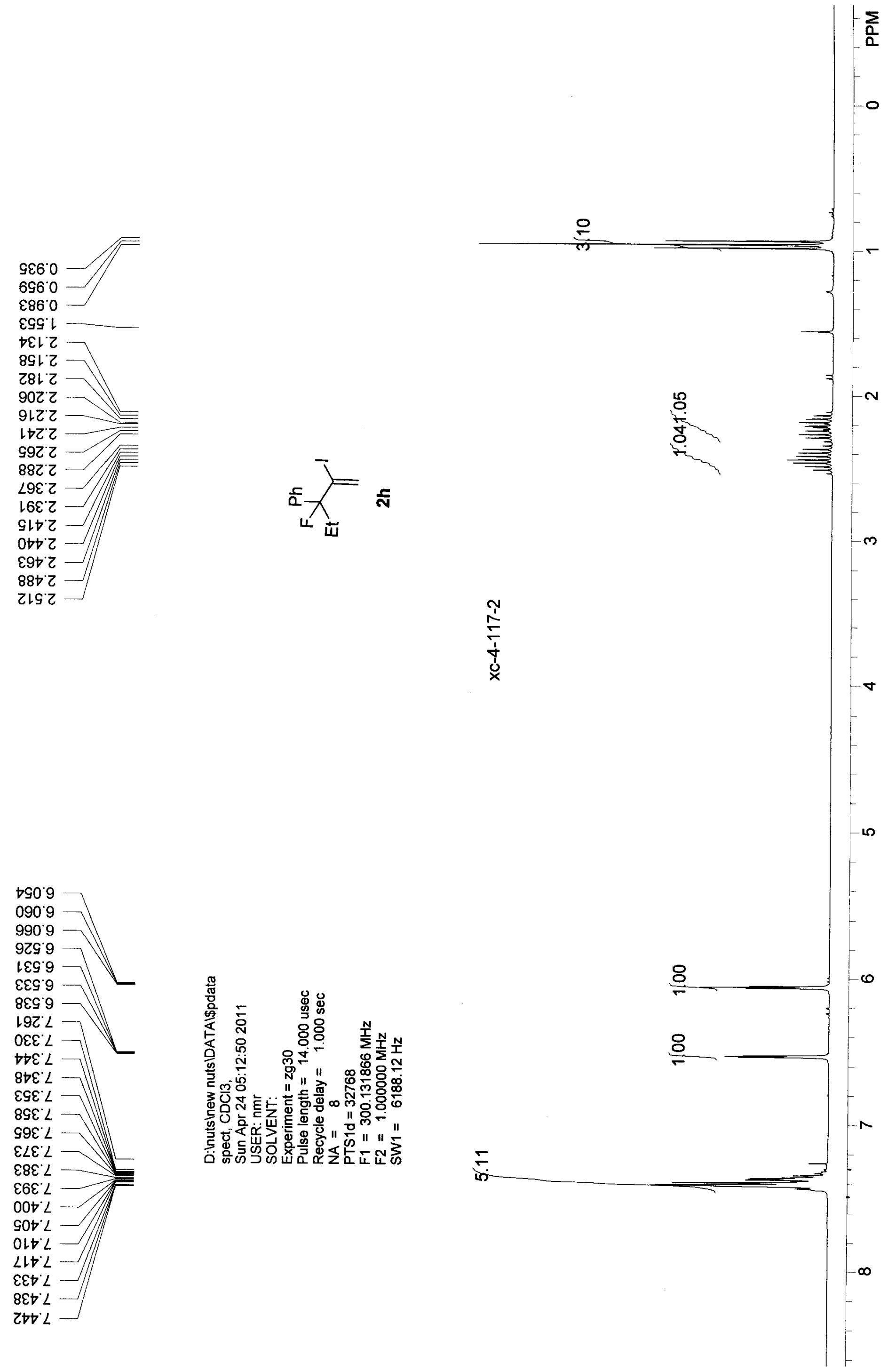
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Tue Dec 13 08:48:27 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2592
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

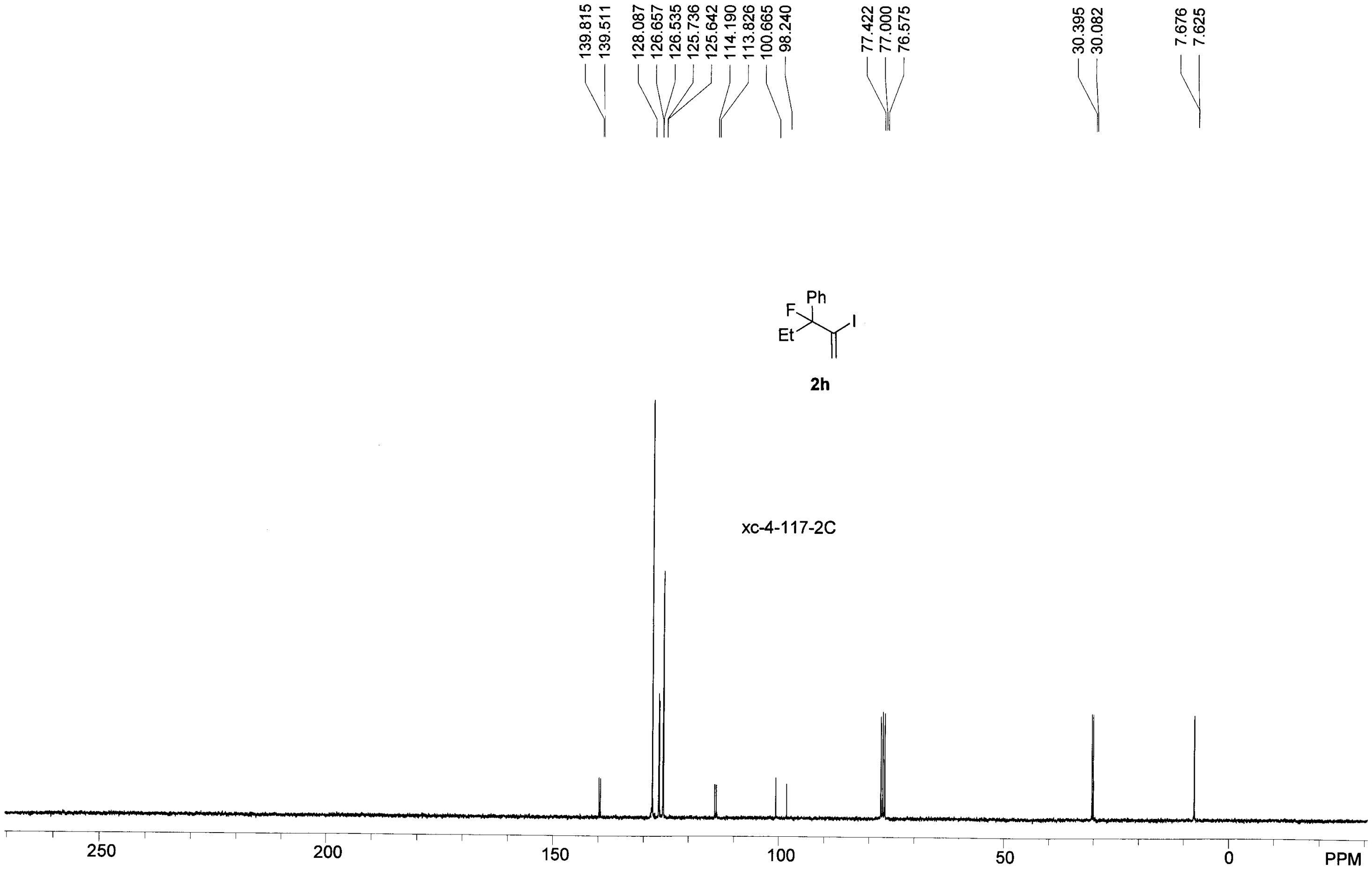

-162.721
-162.832
1.00
F
i-Pr
Ph
2g
xc-6-034-1 F
PPM
-200
-150
-100
-50
0

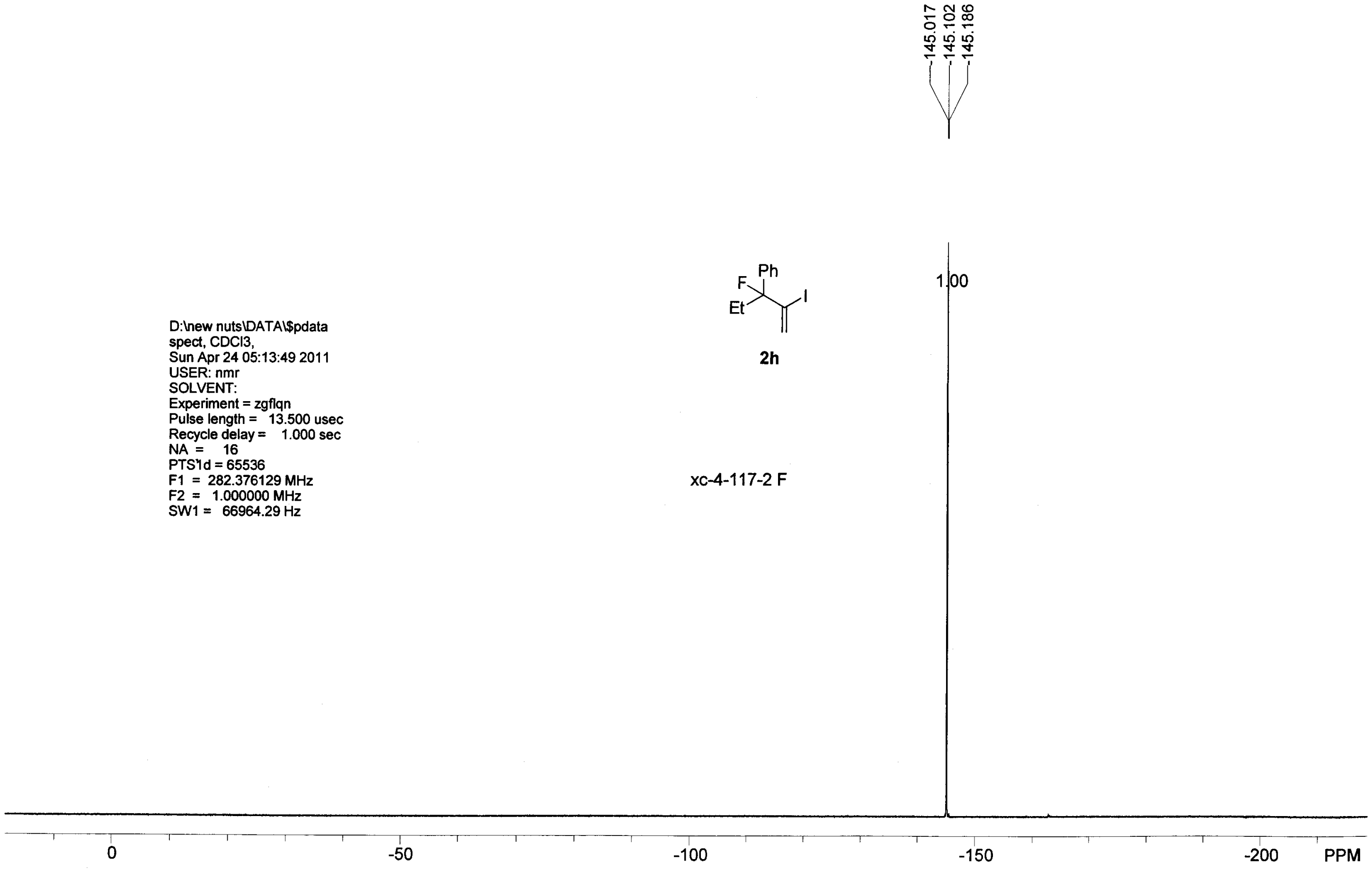


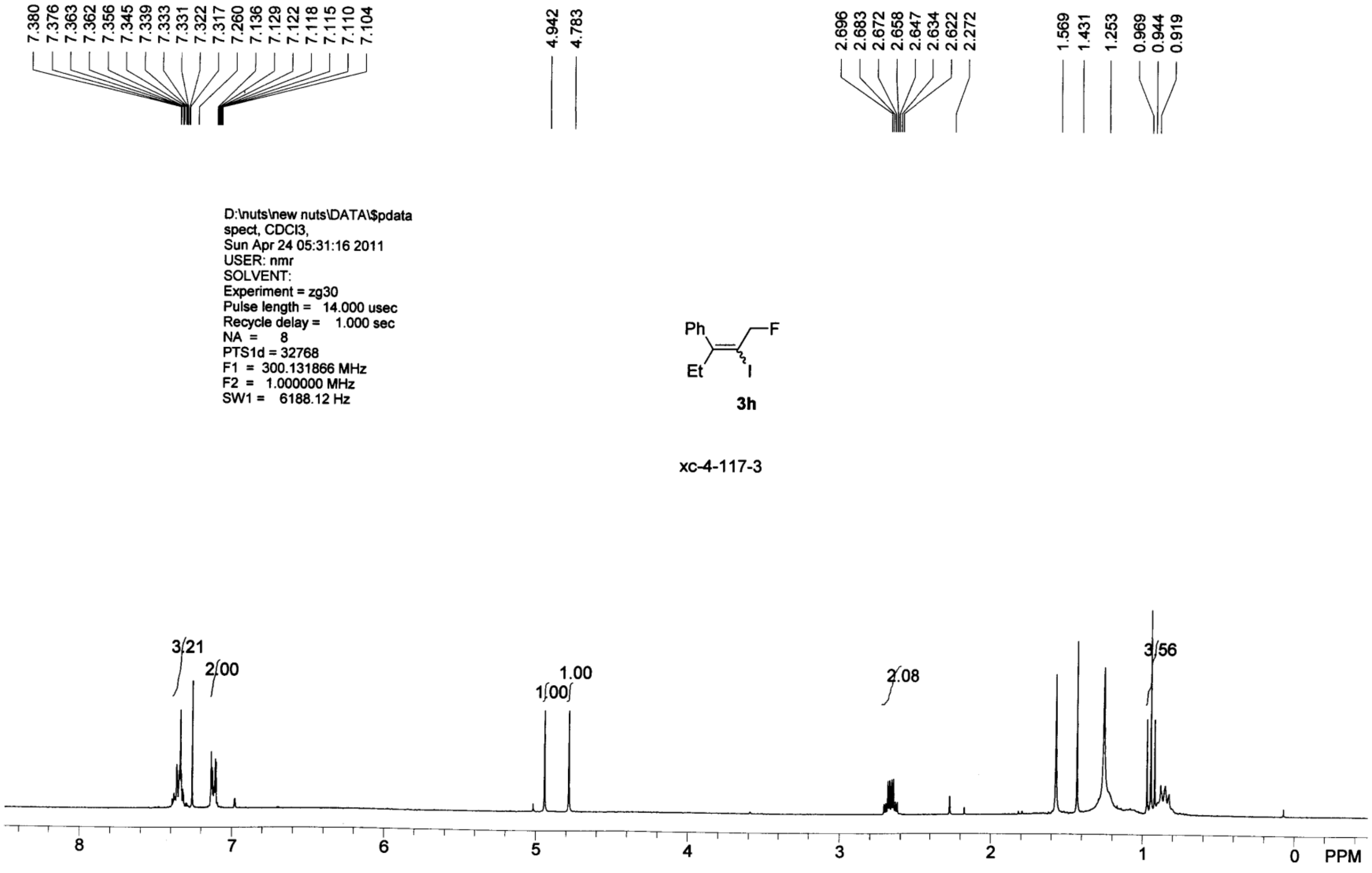
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Fri Apr 01 02:45:13 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz

xc-4-064-cp F


0.935
0.959
0.983
1.553
2.134
2.158
2.182
2.206
2.216
2.241
2.265
2.288
2.367
2.391
2.415
2.440
2.463
2.488
2.512
6.054
6.060
6.066
6.526
6.531
6.533
6.538
7.261
7.330
7.344
7.348
7.353
7.358
7.365
7.373
7.383
7.393
7.400
7.405
7.410
7.417
7.433
7.438
7.442
F
Ph
Et
I
2h
xc-4-117-2
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 24 05:12:50 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3.10
1.04
1.05
1.00
1.00
5.11
PPM
0
1
2
3
4
5
6
7
8


139.815
139.511
128.087
126.657
126.535
125.736
125.642
114.190
113.826
100.665
98.240
77.422
77.000
76.575
30.395
30.082
7.676
7.625
Ph
F
Et
I
2h
xc-4-117-2C
250
200
150
100
50
0
PPM

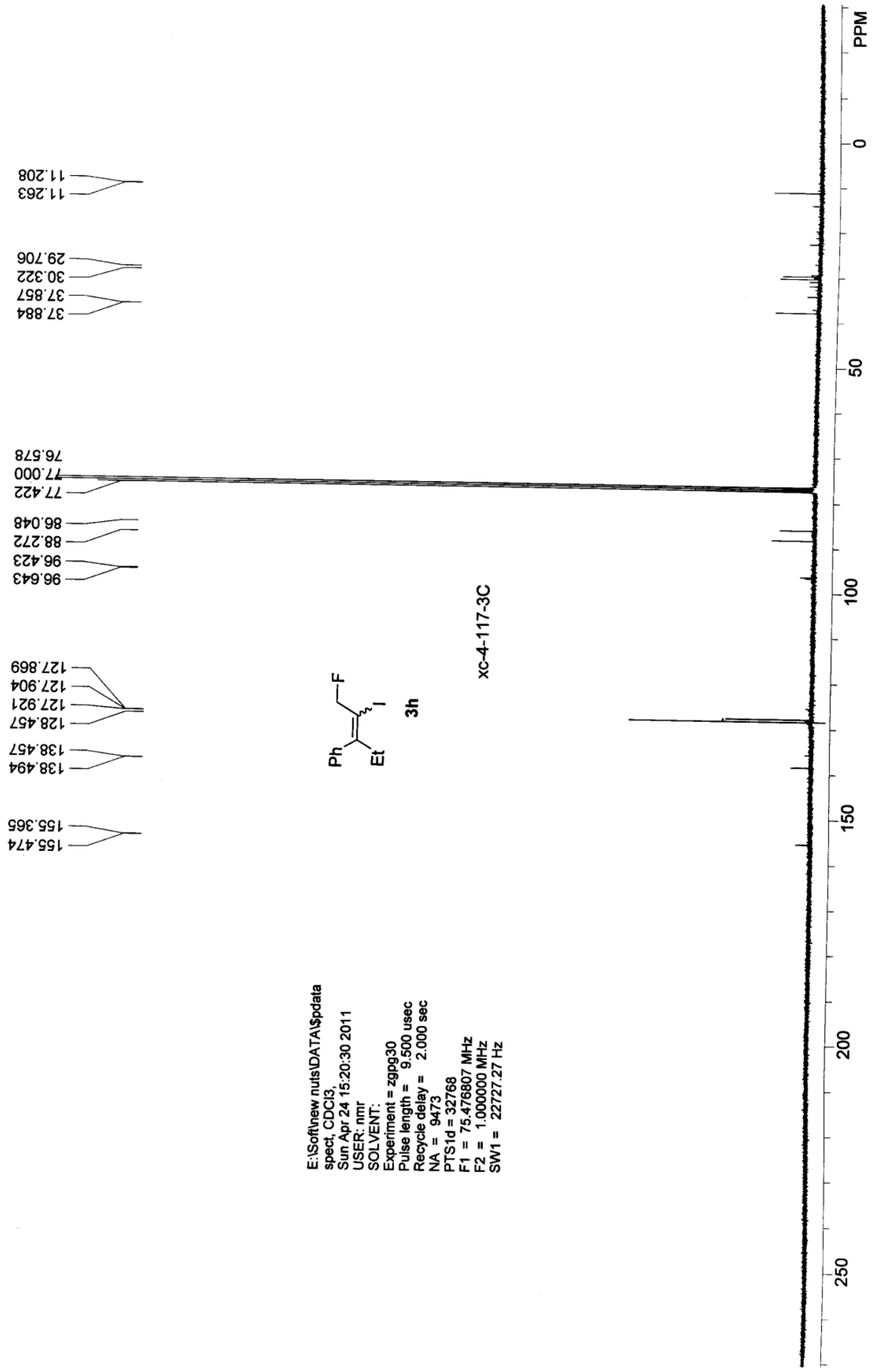

-145.017
-145.102
-145.186
1.00
F
Ph
Et
I
2h
xc-4-117-2 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 24 05:13:49 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
0
-50
-100
-150
-200
PPM



D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 24 05:31:16 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

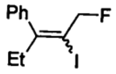
3h

xc-4-117-3



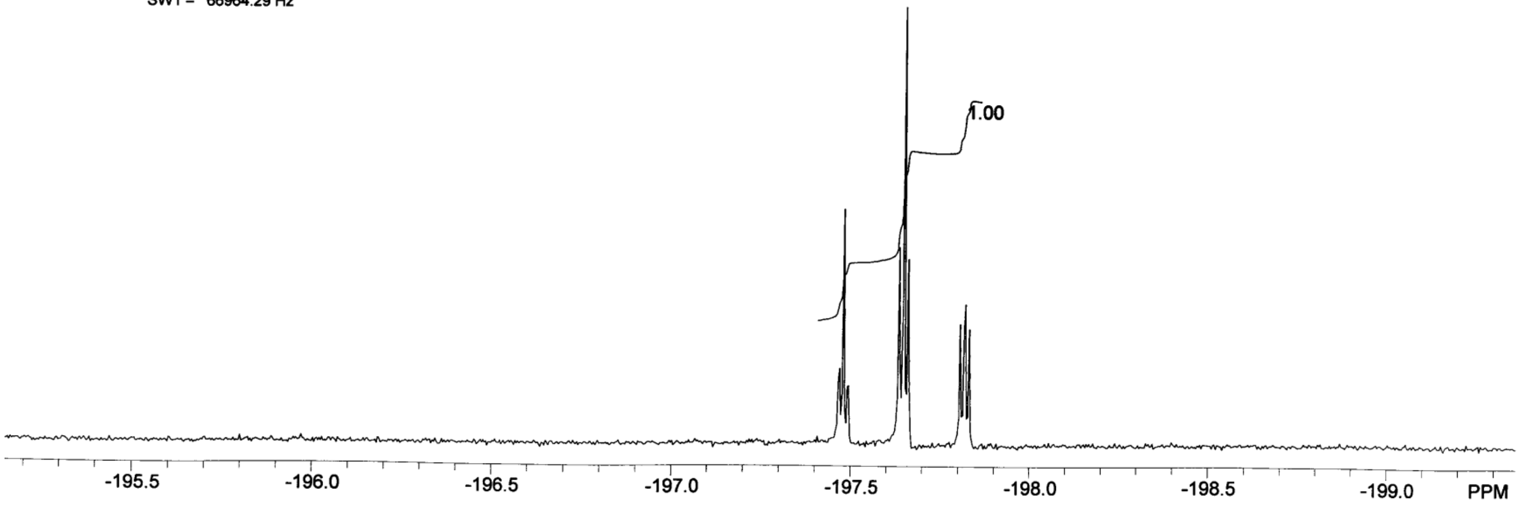
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 24 15:20:30 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 9473
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

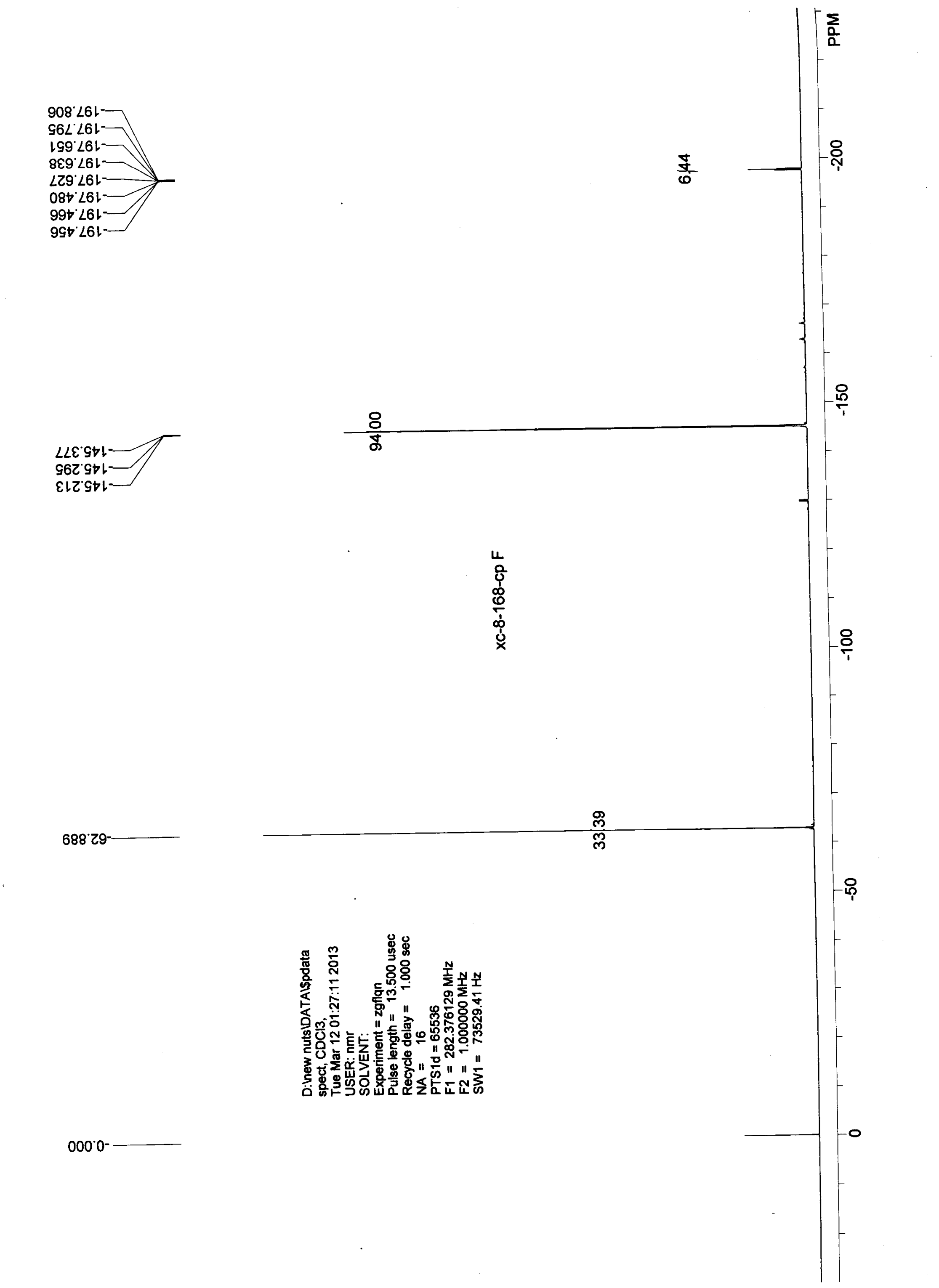
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 24 05:35:10 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz



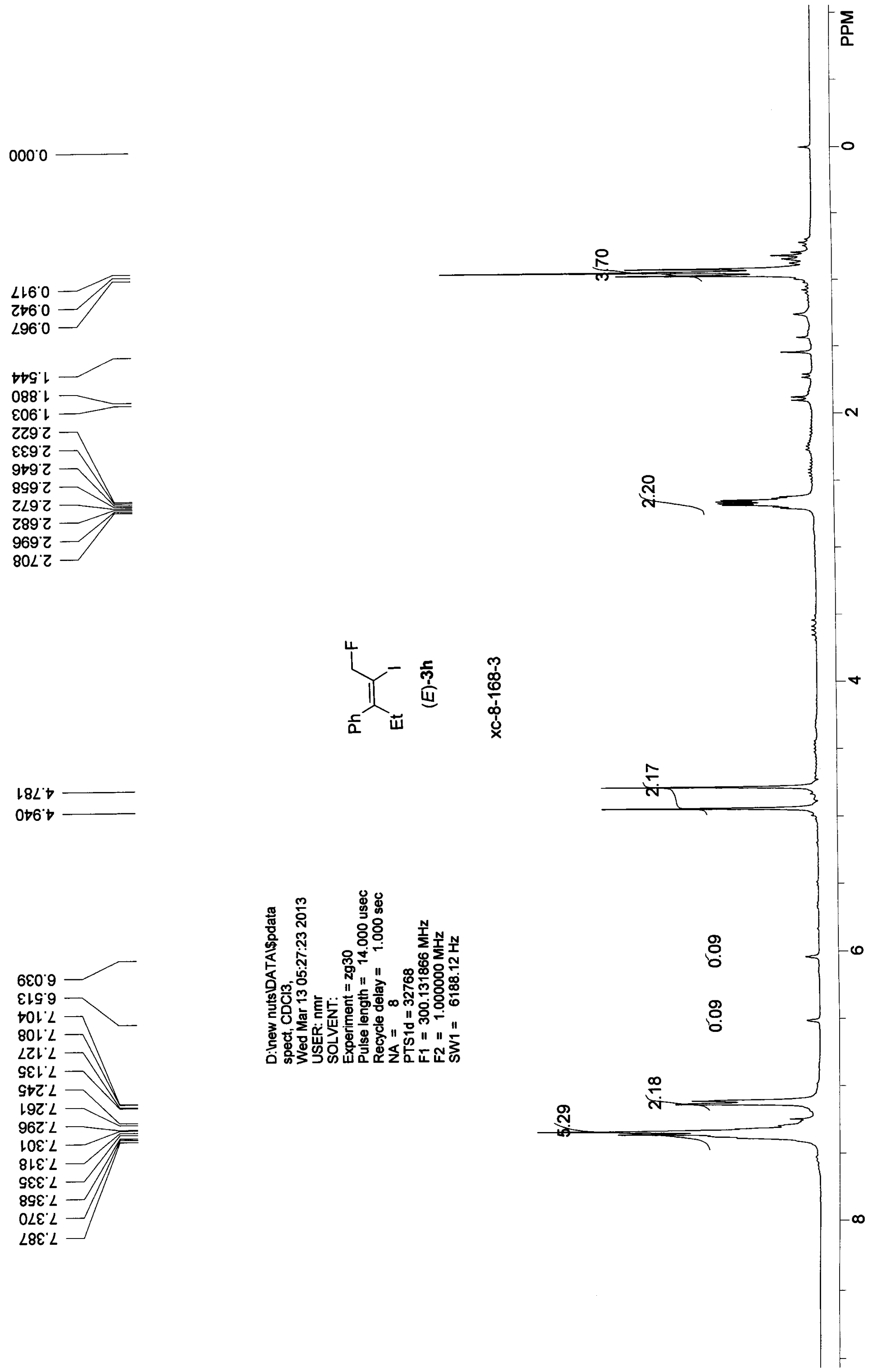
3h

xc-4-117-3 F





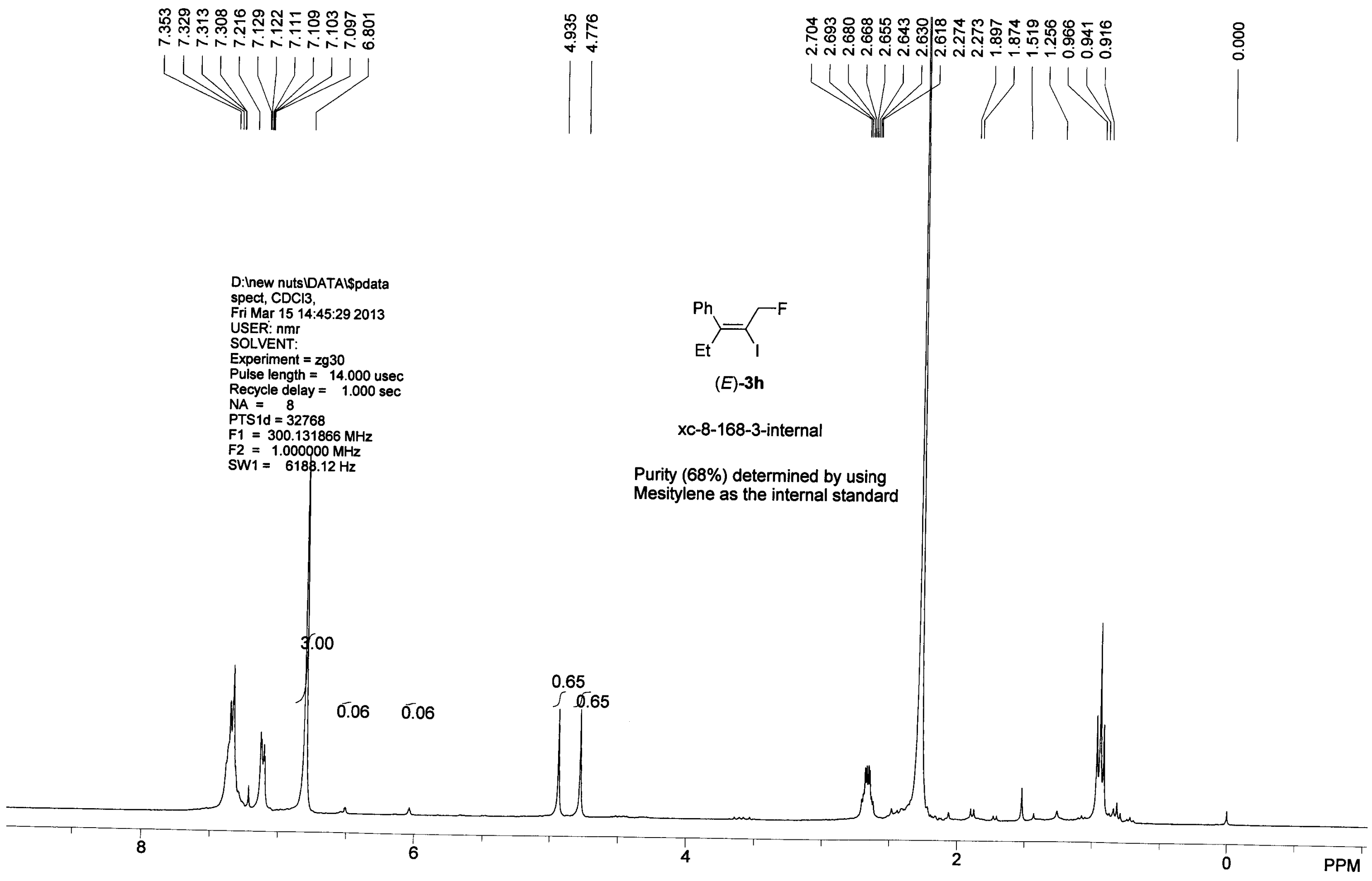
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Mar 12 01:27:11 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz



(E)-3h

xc-8-168-3

D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 13 05:27:23 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

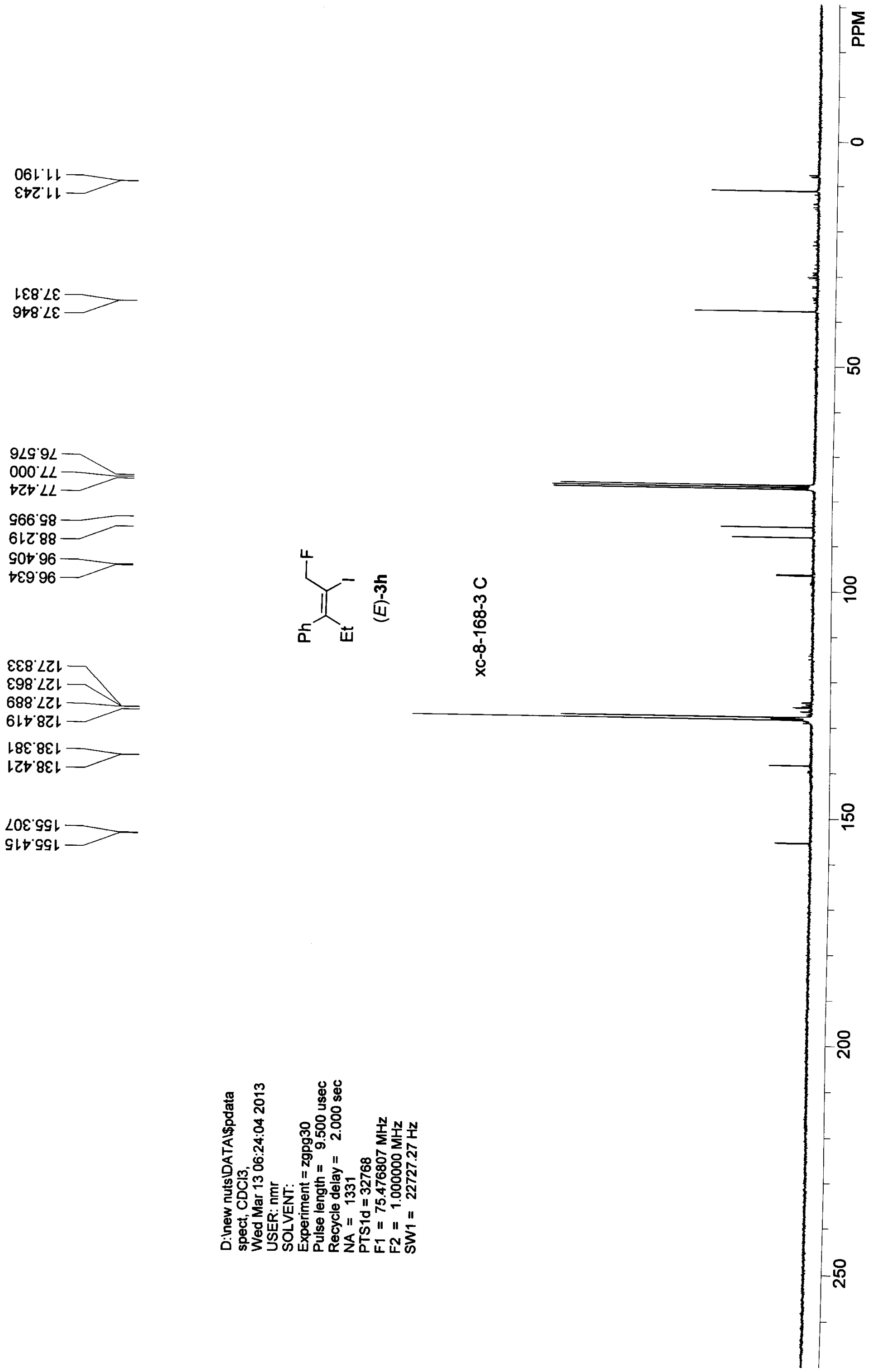


(*E*)-**3h**

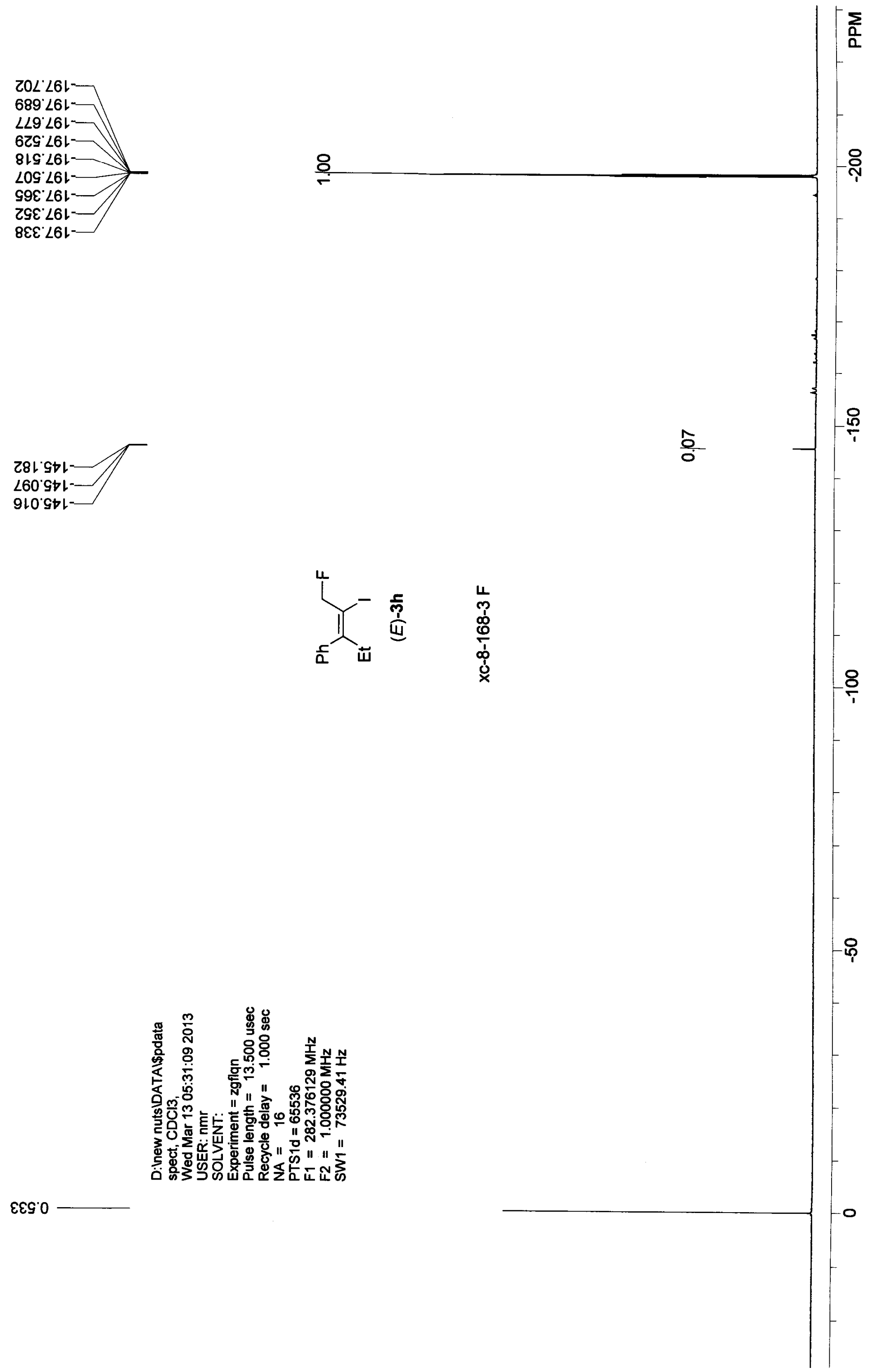
```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Mar 15 14:45:29 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length =   14.000 usec
Recycle delay =    1.000 sec
NA =     8
PTS1d = 32768
F1  =  300.131866 MHz
F2  =   1.000000 MHz
SW1 =    6188.12 Hz
```

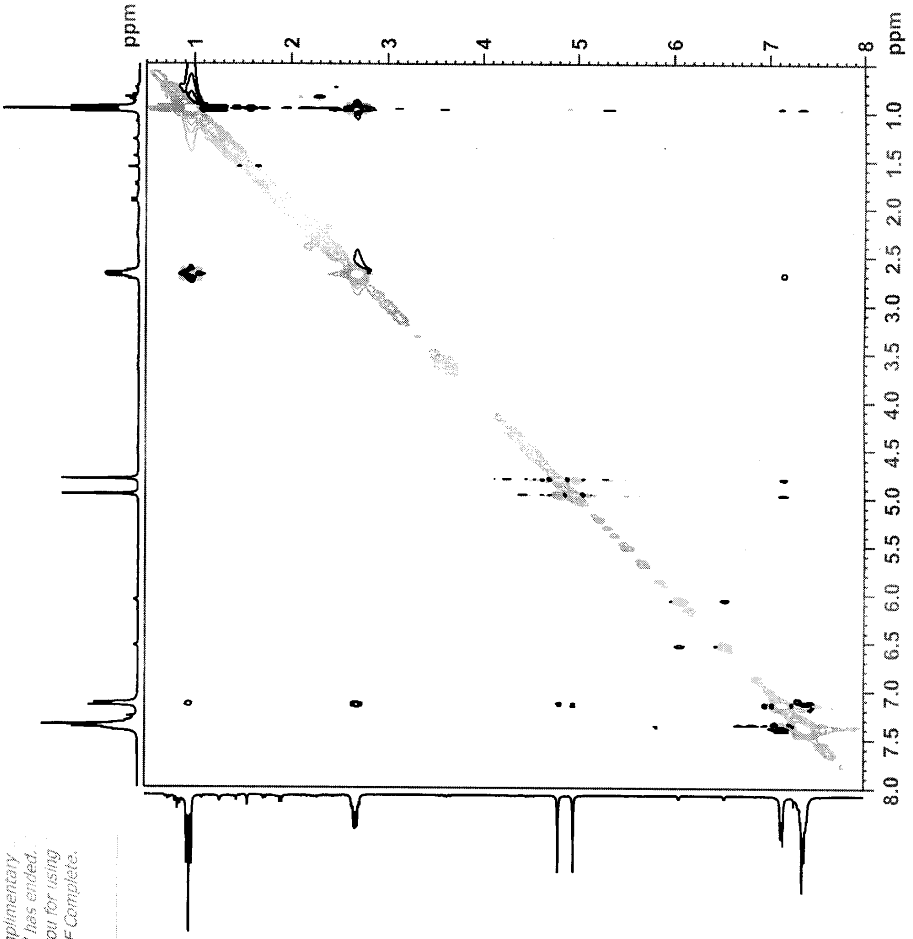
xc-8-168-3-internal

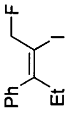
Purity (68%) determined by using
Mesitylene as the internal standard



D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 13 06:24:04 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1331
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

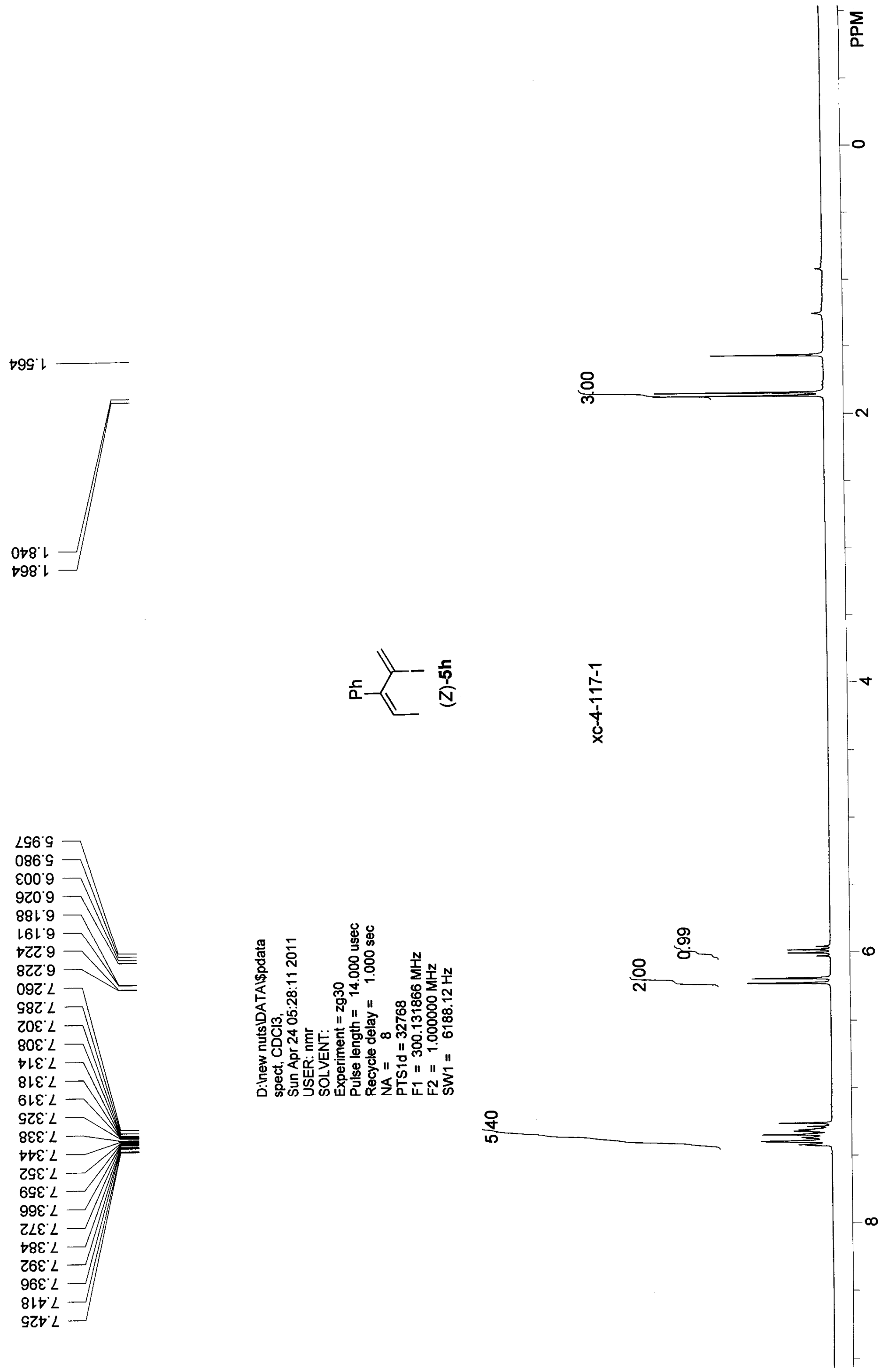

-197.702
-197.689
-197.677
-197.529
-197.518
-197.507
-197.365
-197.352
-197.338
-145.182
-145.097
-145.016
0.533
1.00
0.07
Ph
Et
F
I
(E)-3h
xc-8-168-3 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 13 05:31:09 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
PPM
-200
-150
-100
-50
0

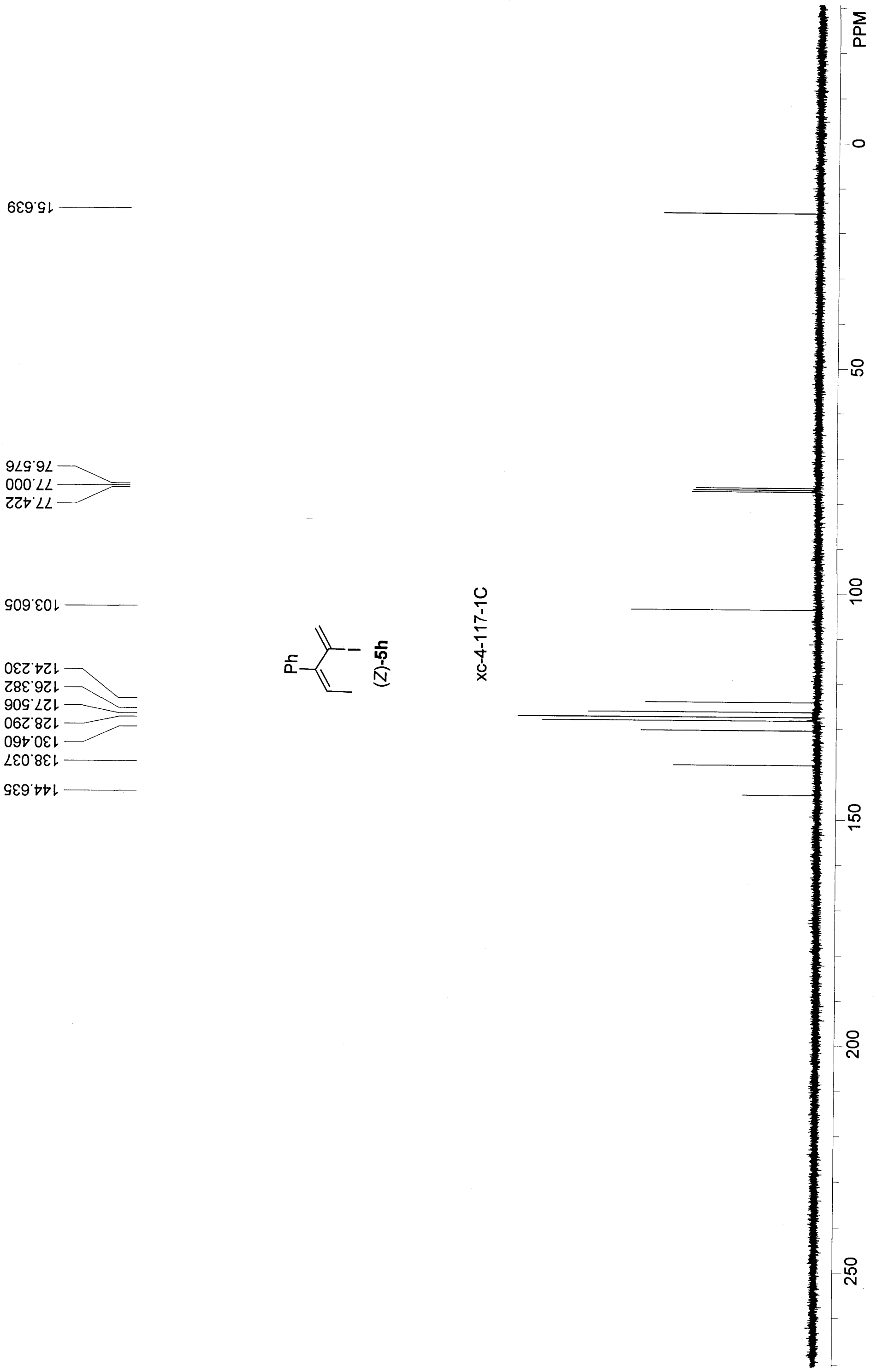


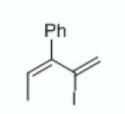


(*E*)-**3h**

xc-8-168-3-noe

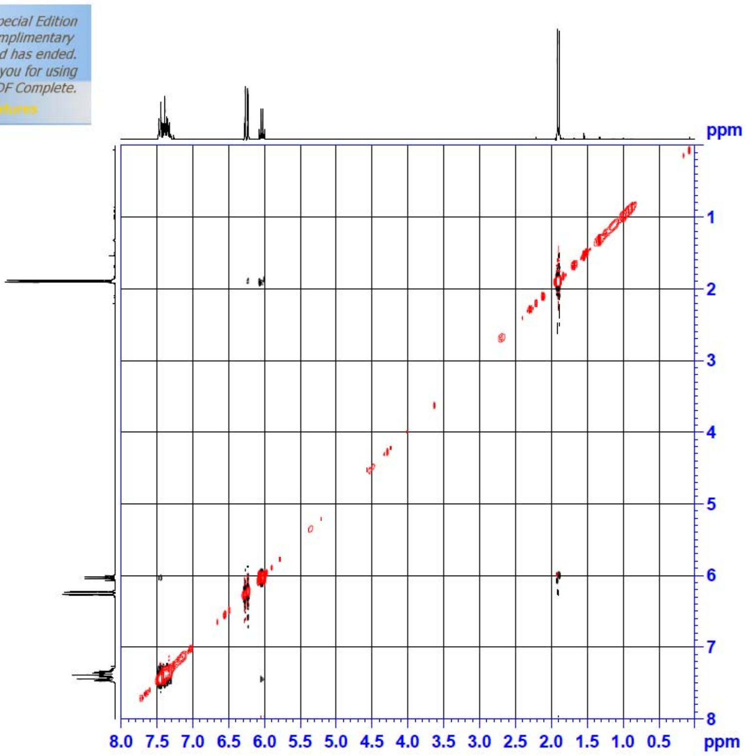

1.564
1.840
1.864
5.957
5.980
6.003
6.026
6.188
6.191
6.224
6.228
7.260
7.285
7.302
7.308
7.314
7.318
7.319
7.325
7.338
7.344
7.352
7.359
7.366
7.372
7.384
7.392
7.396
7.418
7.425
Ph
(Z)-5h
xc-4-117-1
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 24 05:28:11 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3.00
2.00
0.99
5.40
PPM
0
2
4
6
8

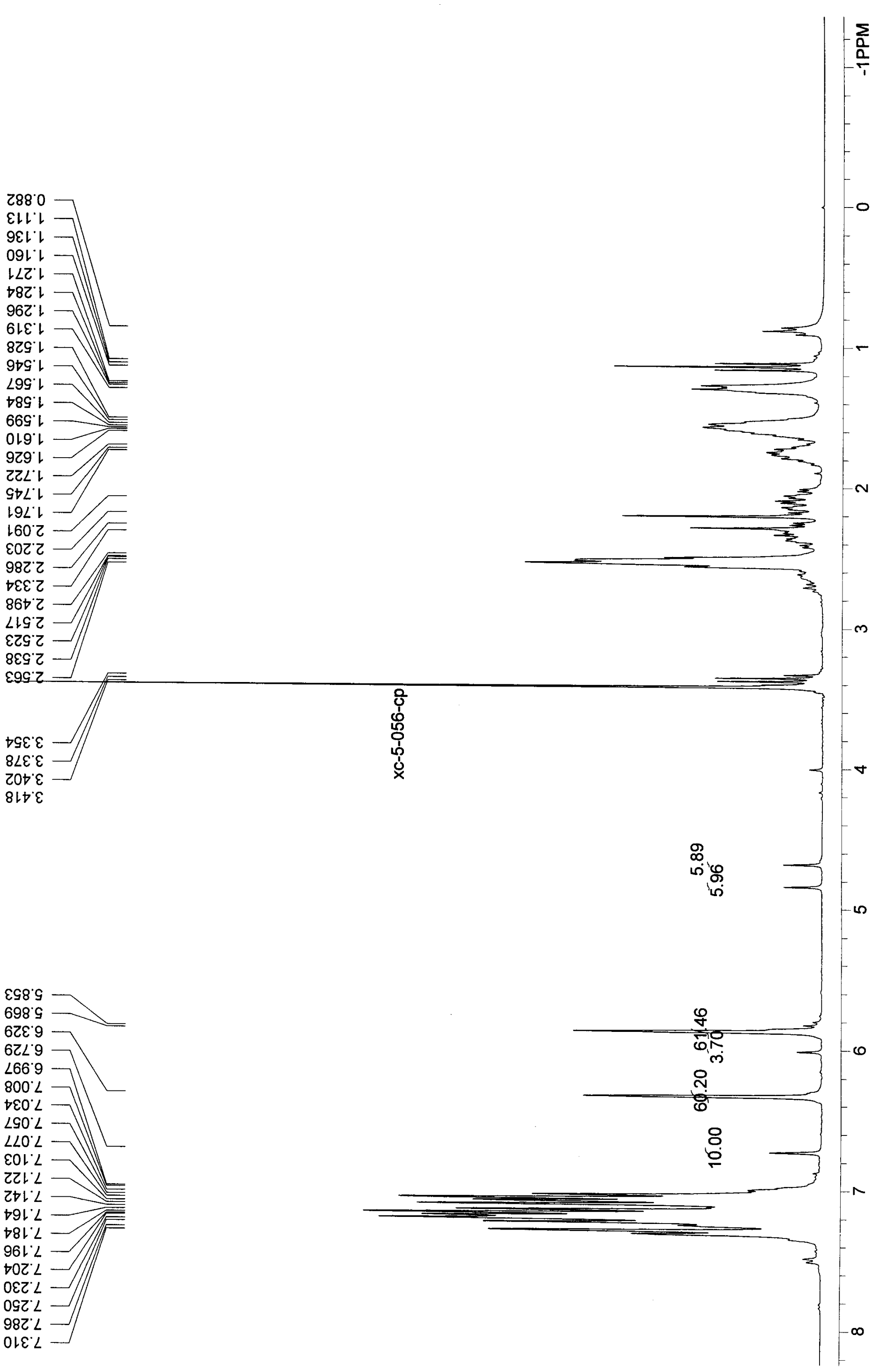

15.639
76.576
77.000
77.422
103.605
124.230
126.382
127.506
128.290
130.460
138.037
144.635
Ph
I
(Z)-5h
xc-4-117-1C
PPM
0
50
100
150
200
250

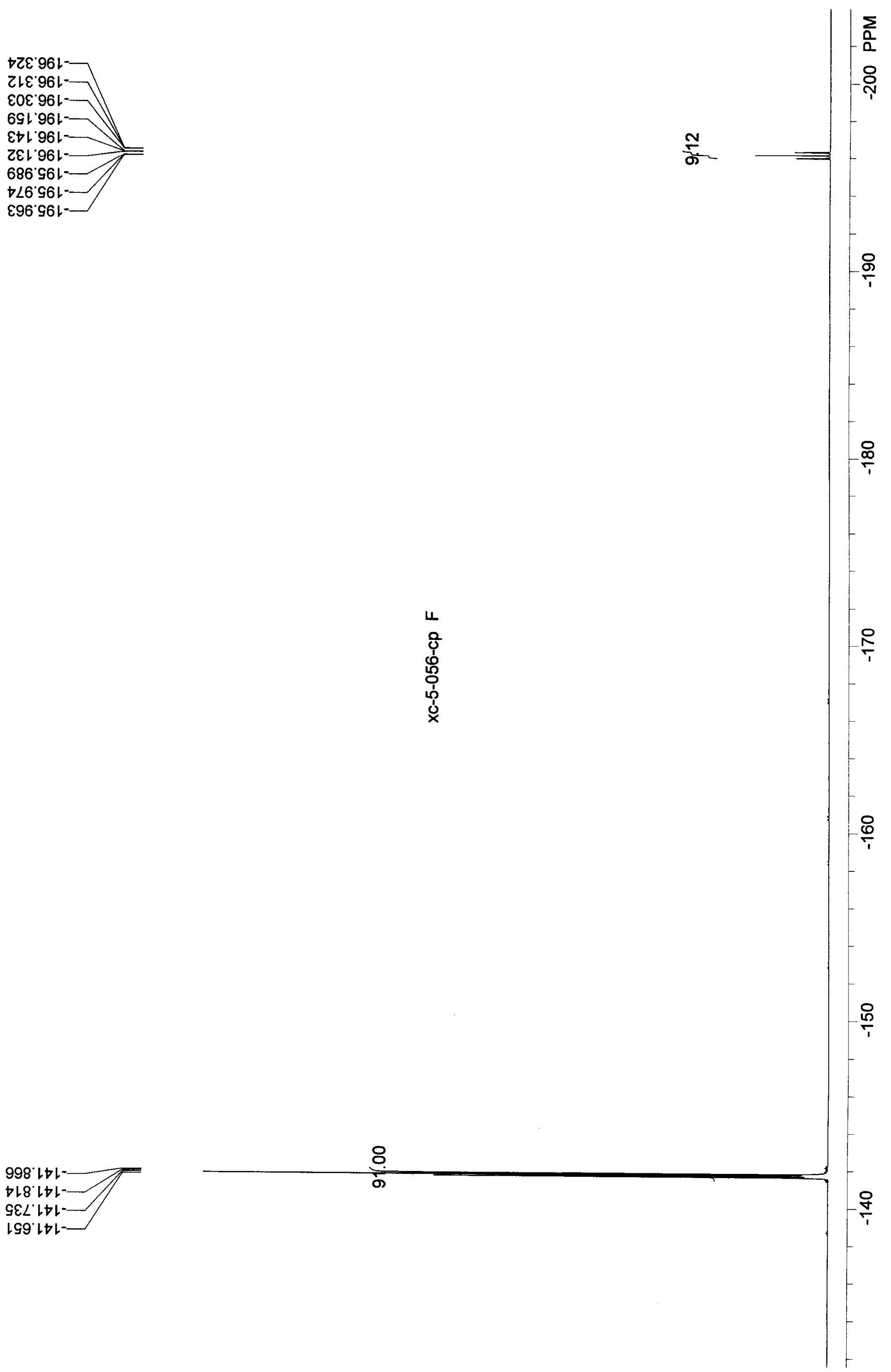


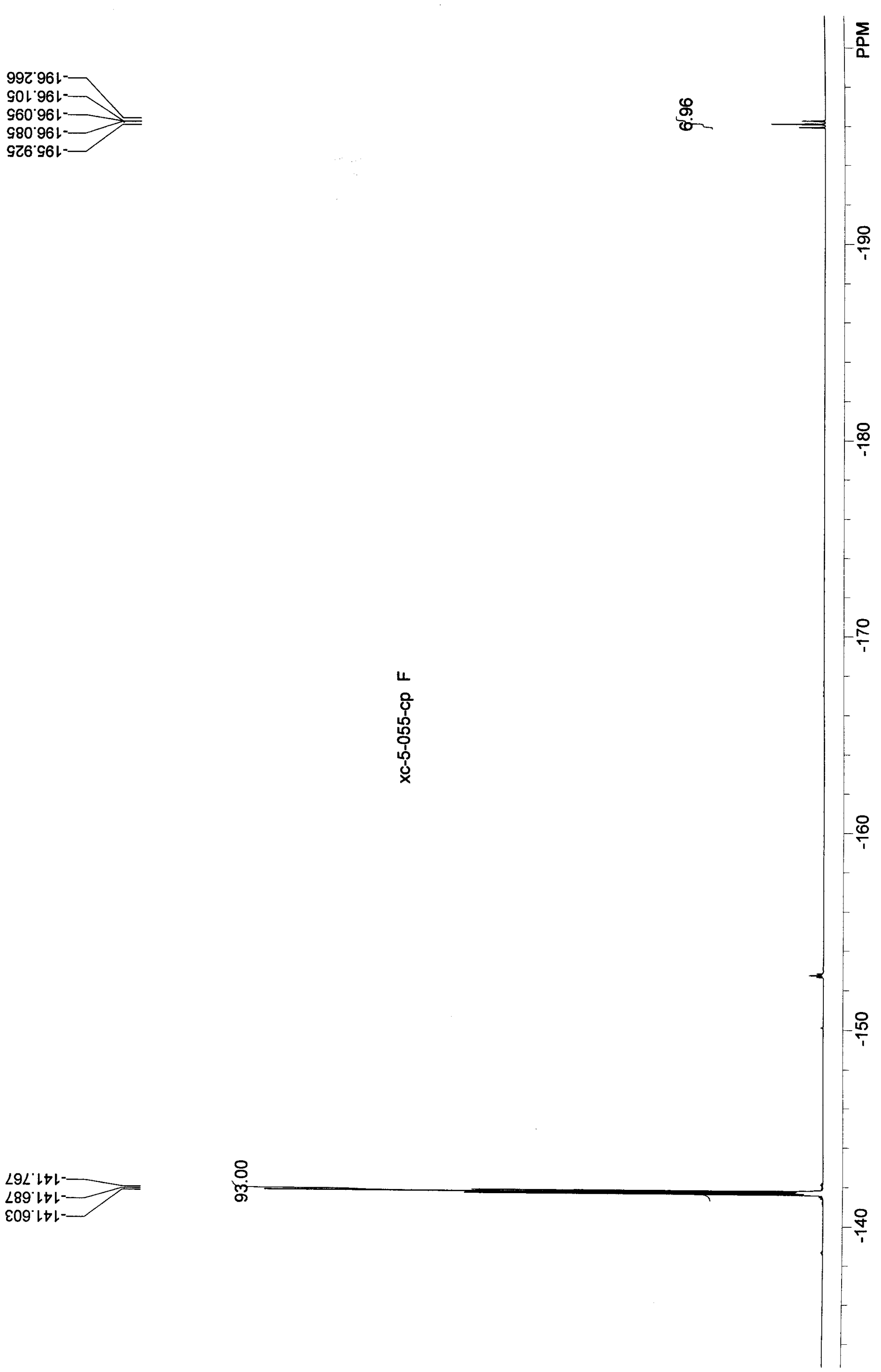
(*Z*)-**5h**

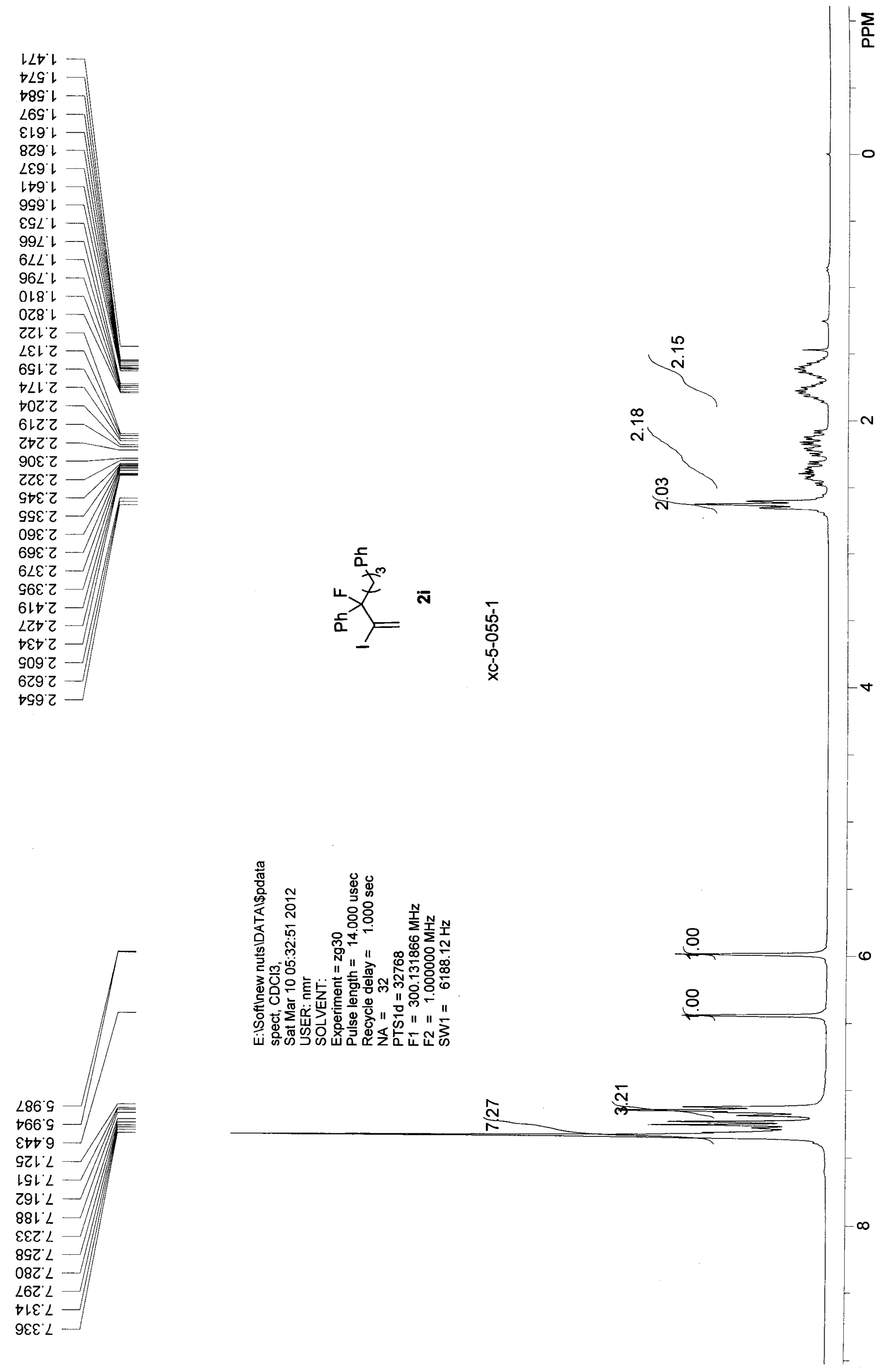
xc-4-117-1-noe




xc-5-056-cp
0.882
1.113
1.136
1.160
1.271
1.284
1.296
1.319
1.528
1.546
1.567
1.584
1.599
1.610
1.626
1.722
1.745
1.761
2.091
2.203
2.286
2.334
2.498
2.517
2.523
2.538
2.563
3.354
3.378
3.402
3.418
5.853
5.869
6.329
6.729
6.997
7.008
7.034
7.057
7.077
7.103
7.122
7.142
7.164
7.184
7.196
7.204
7.230
7.250
7.286
7.310
5.89
5.96
61.46
3.70
60.20
10.00
-1PPM
0
1
2
3
4
5
6
7
8


xc-5-056-cp F
-196.324
-196.312
-196.303
-196.159
-196.143
-196.132
-195.989
-195.974
-195.963
-141.866
-141.814
-141.735
-141.651
9.12
9.00
-200 PPM
-190
-180
-170
-160
-150
-140

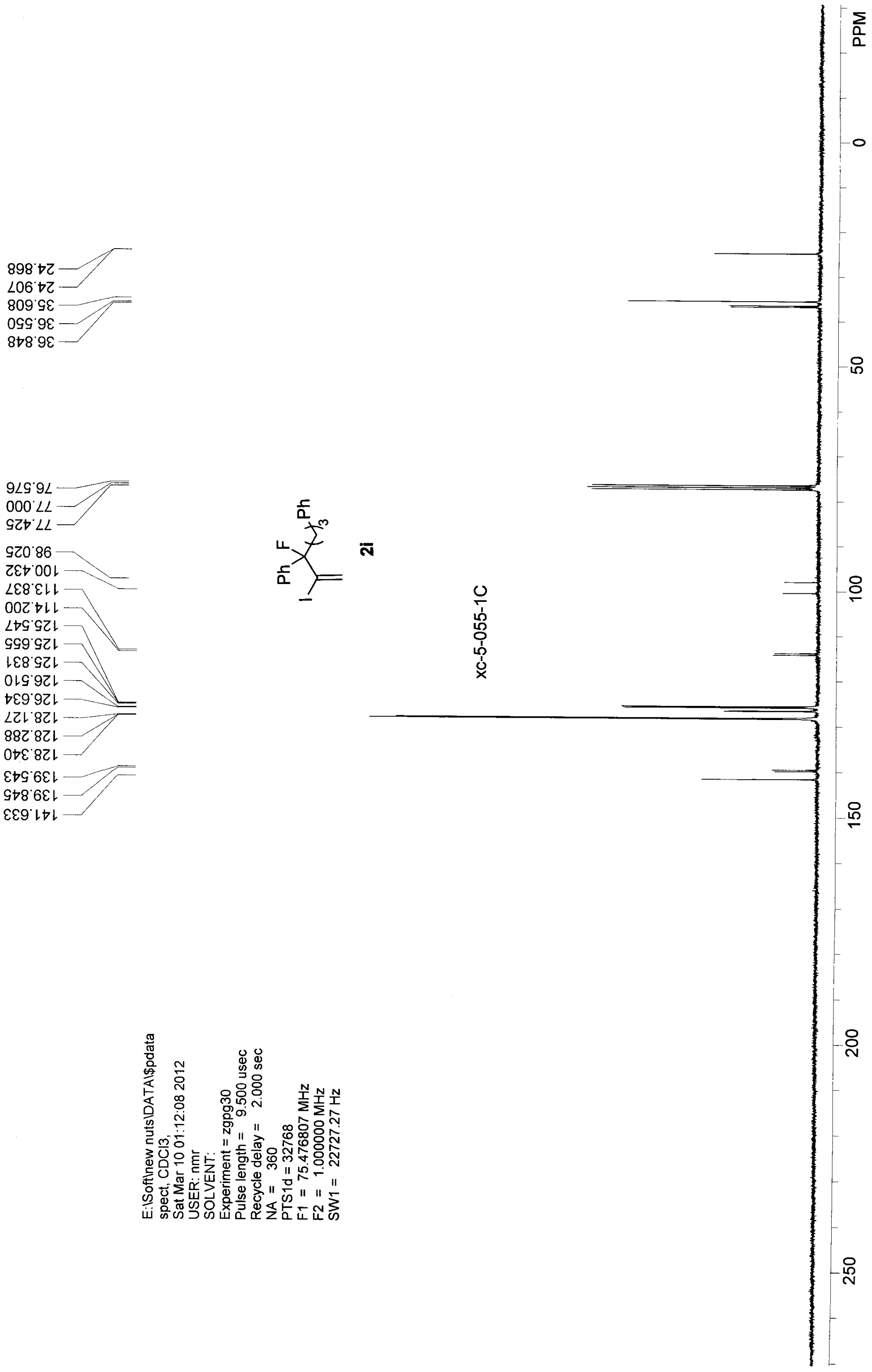

xc-5-055-cp F
-196.266
-196.105
-196.095
-196.085
-195.925
-141.767
-141.687
-141.603
6.96
93.00
PPM
-190
-180
-170
-160
-150
-140

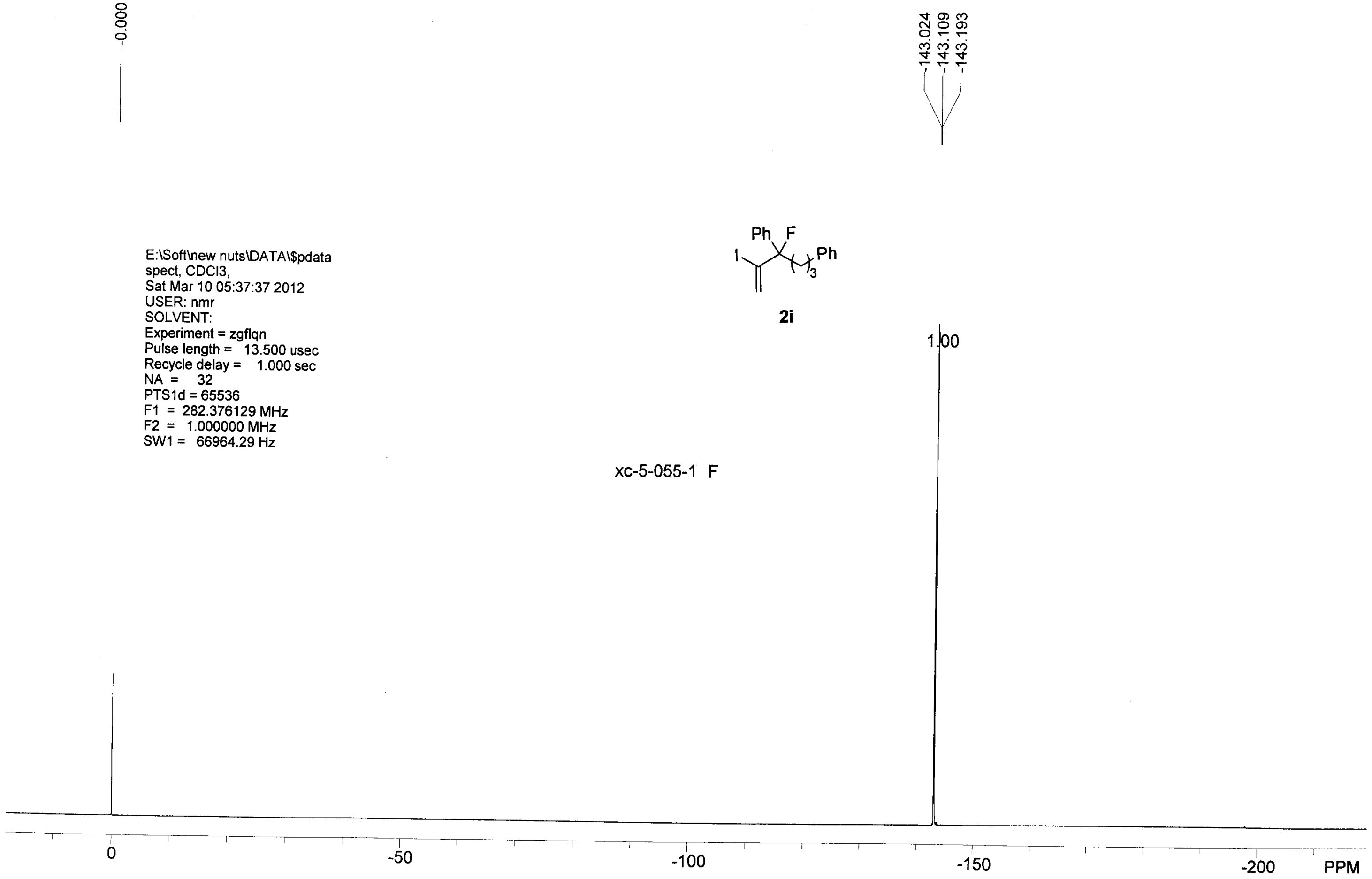


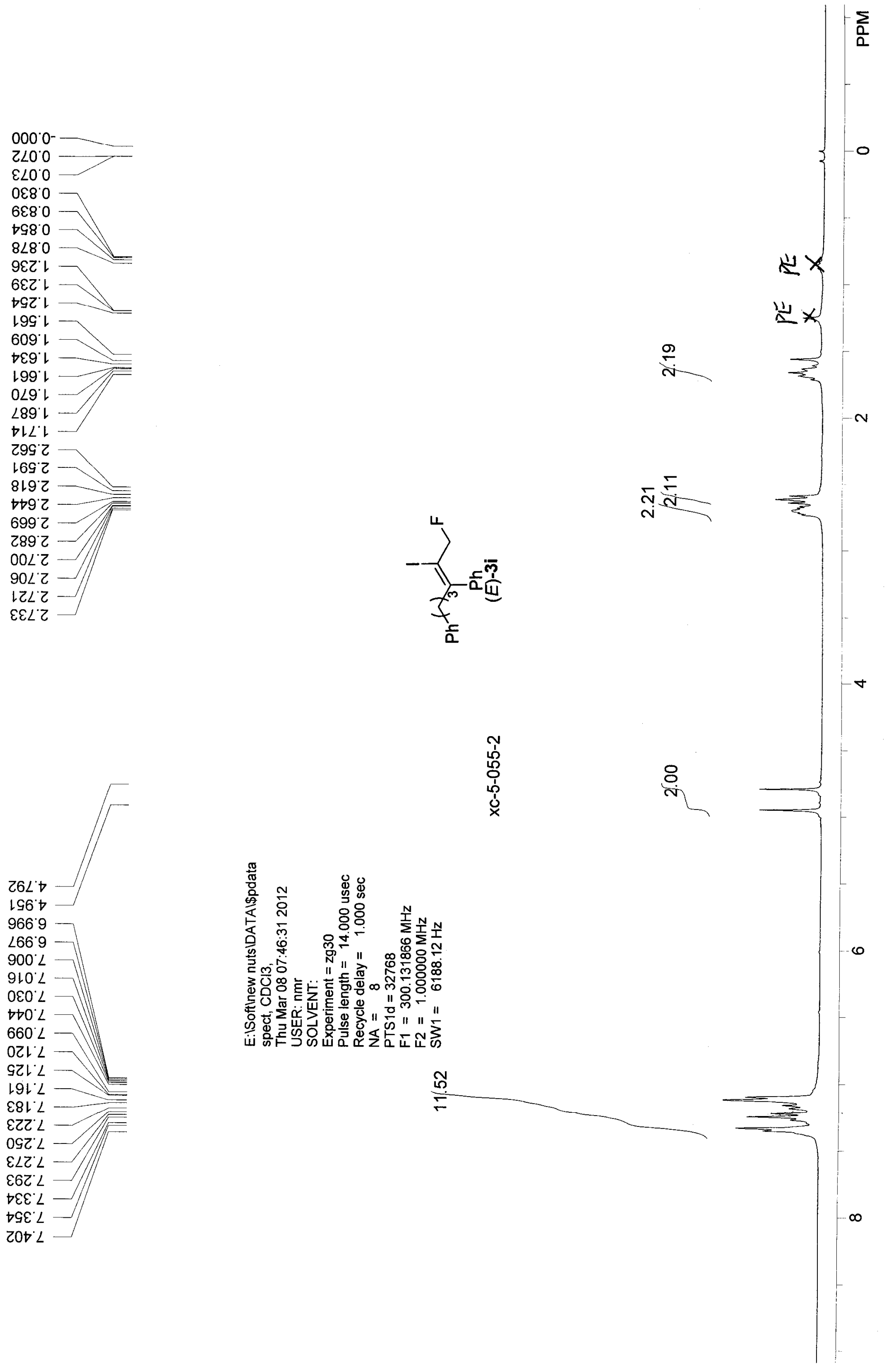
2i

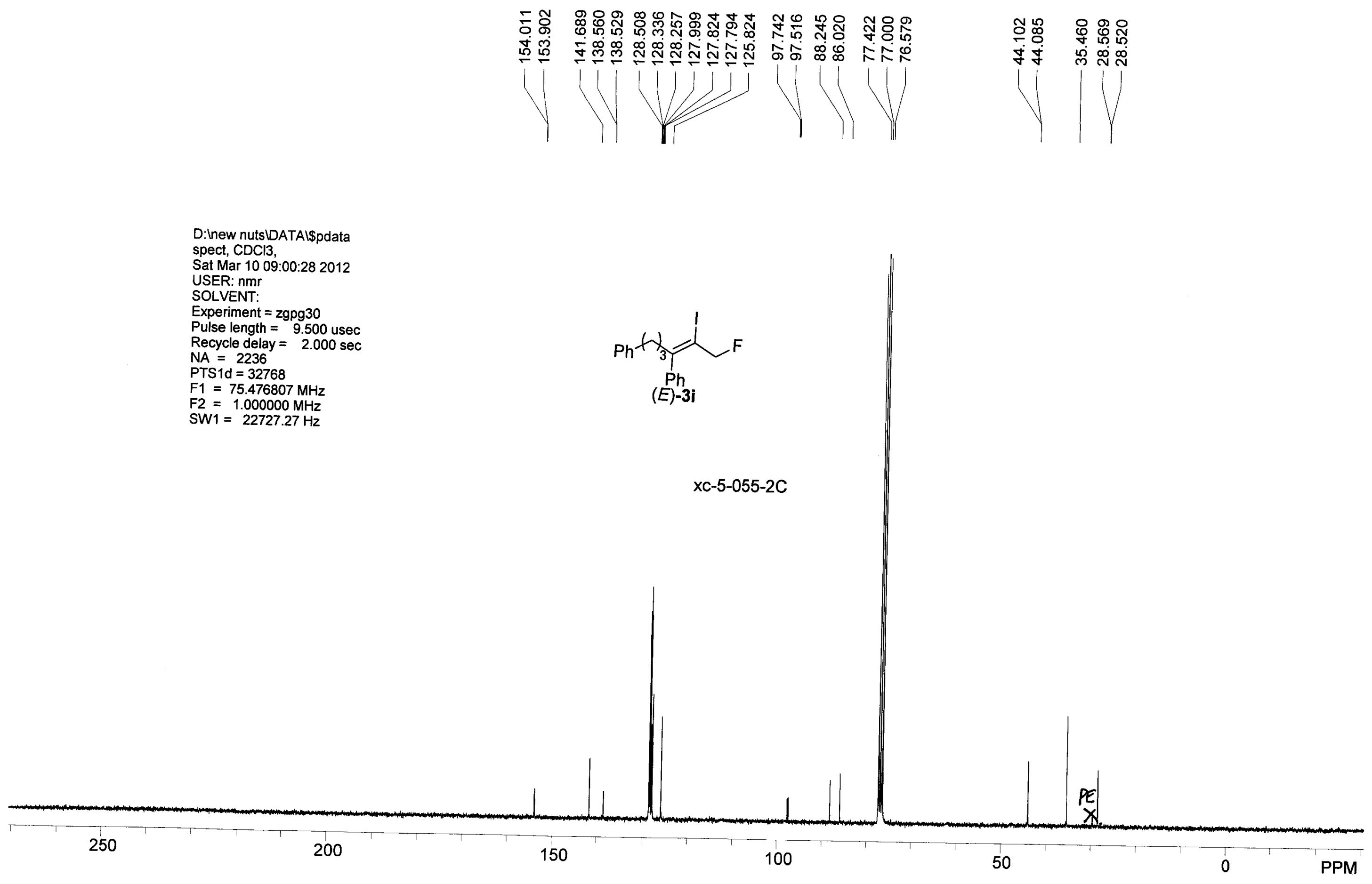
xc-5-055-1

E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 10 05:32:51 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz


24.868
24.907
36.608
36.550
36.848
76.576
77.000
77.425
98.025
100.432
113.837
114.200
125.547
125.655
125.831
126.510
126.634
128.127
128.288
128.340
139.543
139.845
141.633
Ph
F
Ph
I
3
2i
xc-5-055-1C
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 10 01:12:08 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 360
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250


-0.000
-143.024
-143.109
-143.193
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 10 05:37:37 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
Ph
F
I
Ph
3
2i
1.00
xc-5-055-1 F
0
-50
-100
-150
-200
PPM

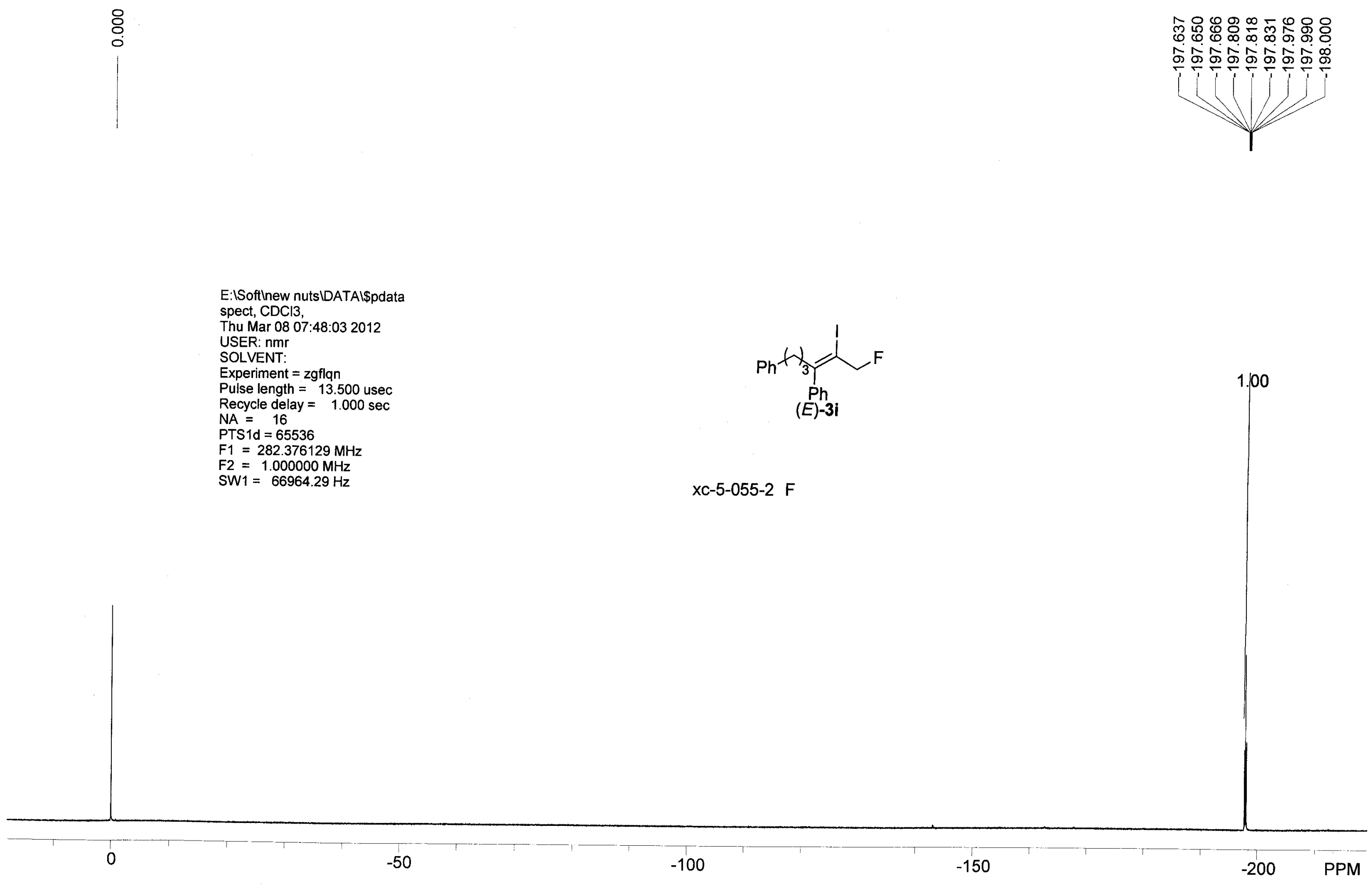

-0.000
0.072
0.073
0.830
0.839
0.854
0.878
1.236
1.239
1.254
1.561
1.609
1.634
1.661
1.670
1.687
1.714
2.562
2.591
2.618
2.644
2.669
2.682
2.700
2.706
2.721
2.733
4.792
4.951
6.996
6.997
7.006
7.016
7.030
7.044
7.099
7.120
7.125
7.161
7.183
7.223
7.250
7.273
7.293
7.334
7.354
7.402
Ph
3
I
F
Ph
(E)-3i
xc-5-055-2
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Thu Mar 08 07:46:31 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.19
2.21
2.11
2.00
11.52
PE
PE
PPM
0
2
4
6
8



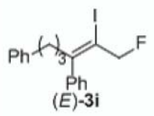
(*E*)-**3i**

xc-5-055-2C

```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 10 09:00:28 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length =    9.500 usec
Recycle delay =    2.000 sec
NA  =  2236
PTS1d = 32768
F1  =  75.476807 MHz
F2  =   1.000000 MHz
SW1 =   22727.27 Hz
```

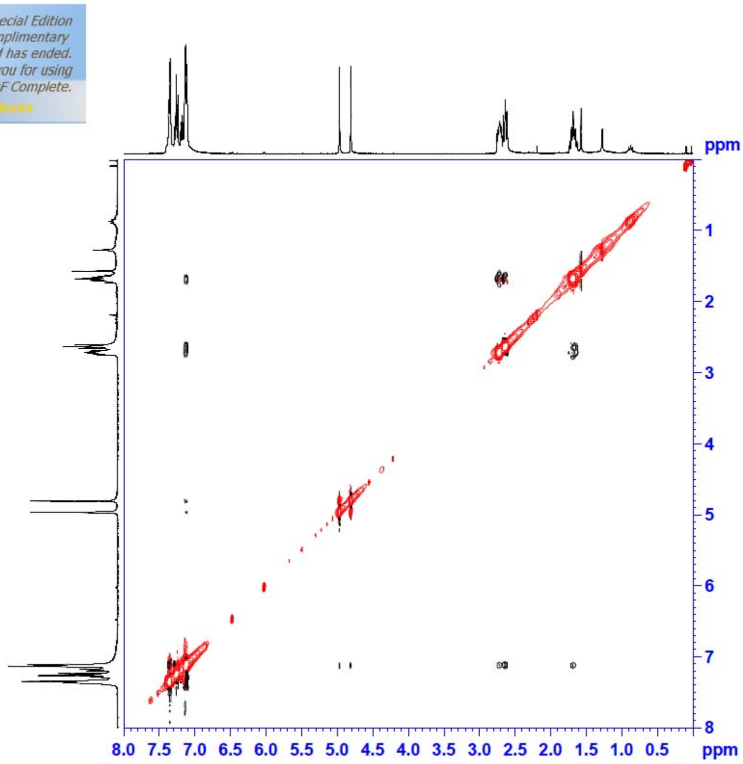


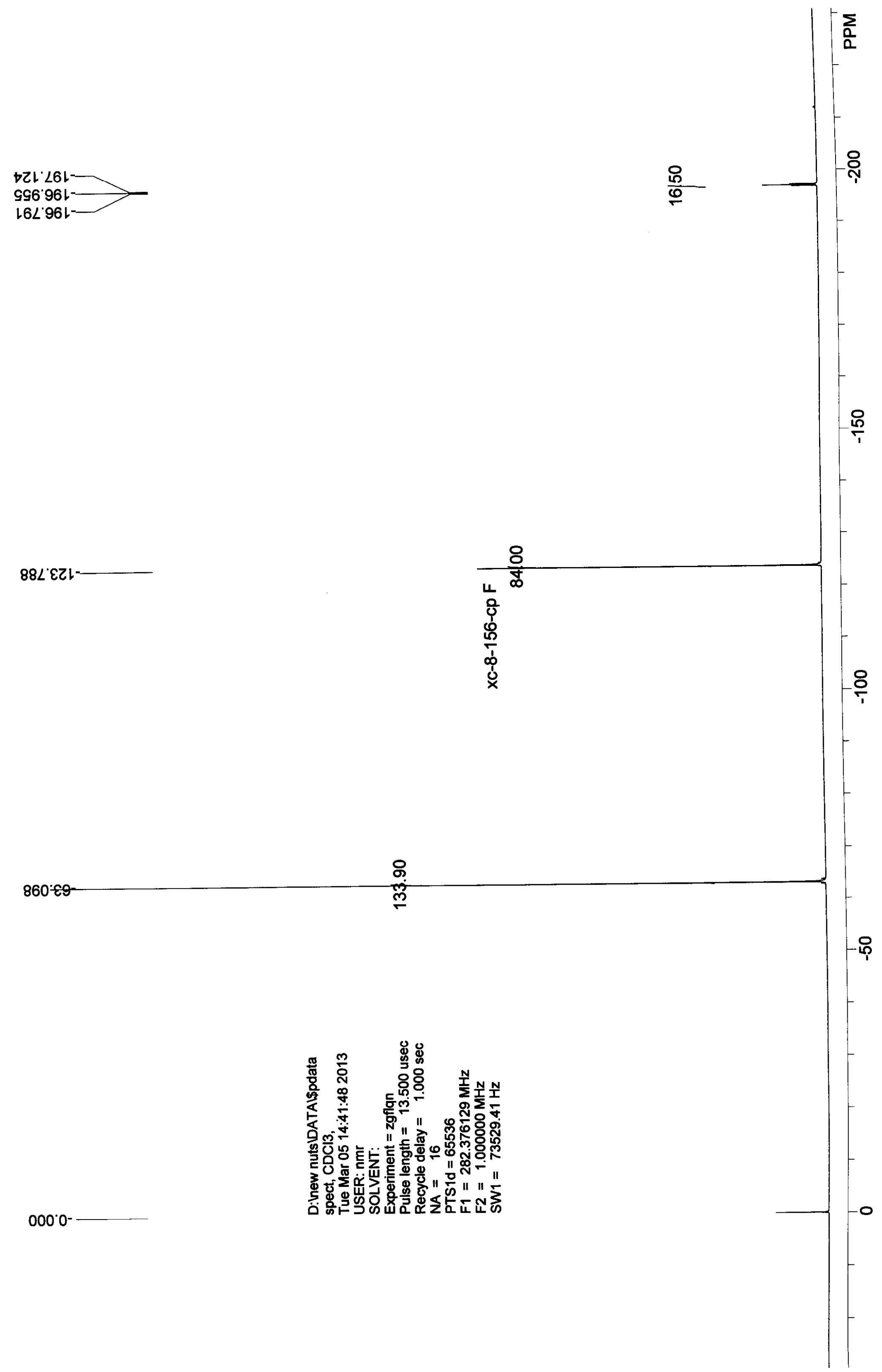
```
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Thu Mar 08 07:48:03 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =   13.500 usec
Recycle delay =    1.000 sec
NA =    16
PTS1d = 65536
F1  =  282.376129 MHz
F2  =   1.000000 MHz
SW1 =   66964.29 Hz
```

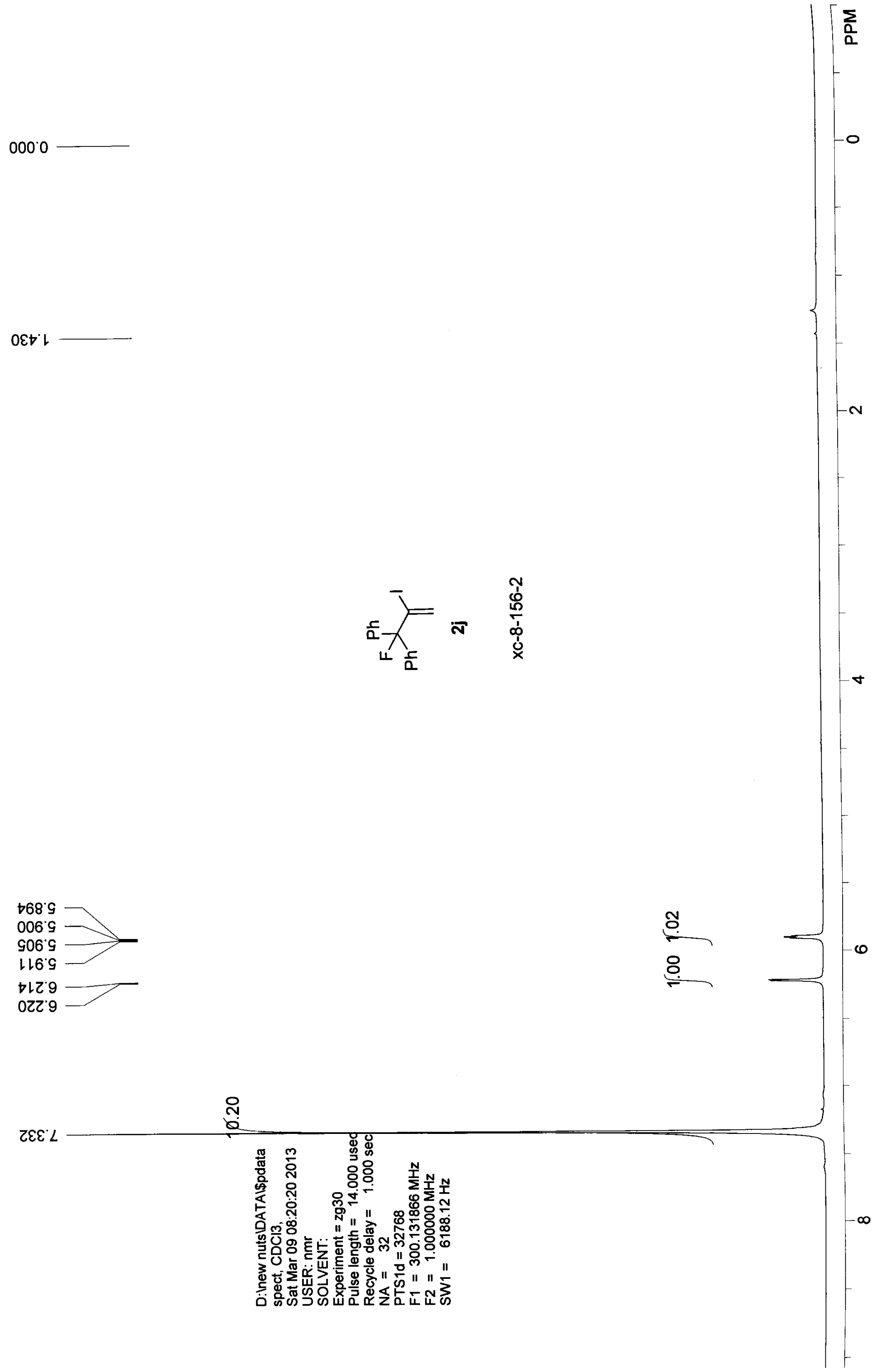


(*E*)-**3i**

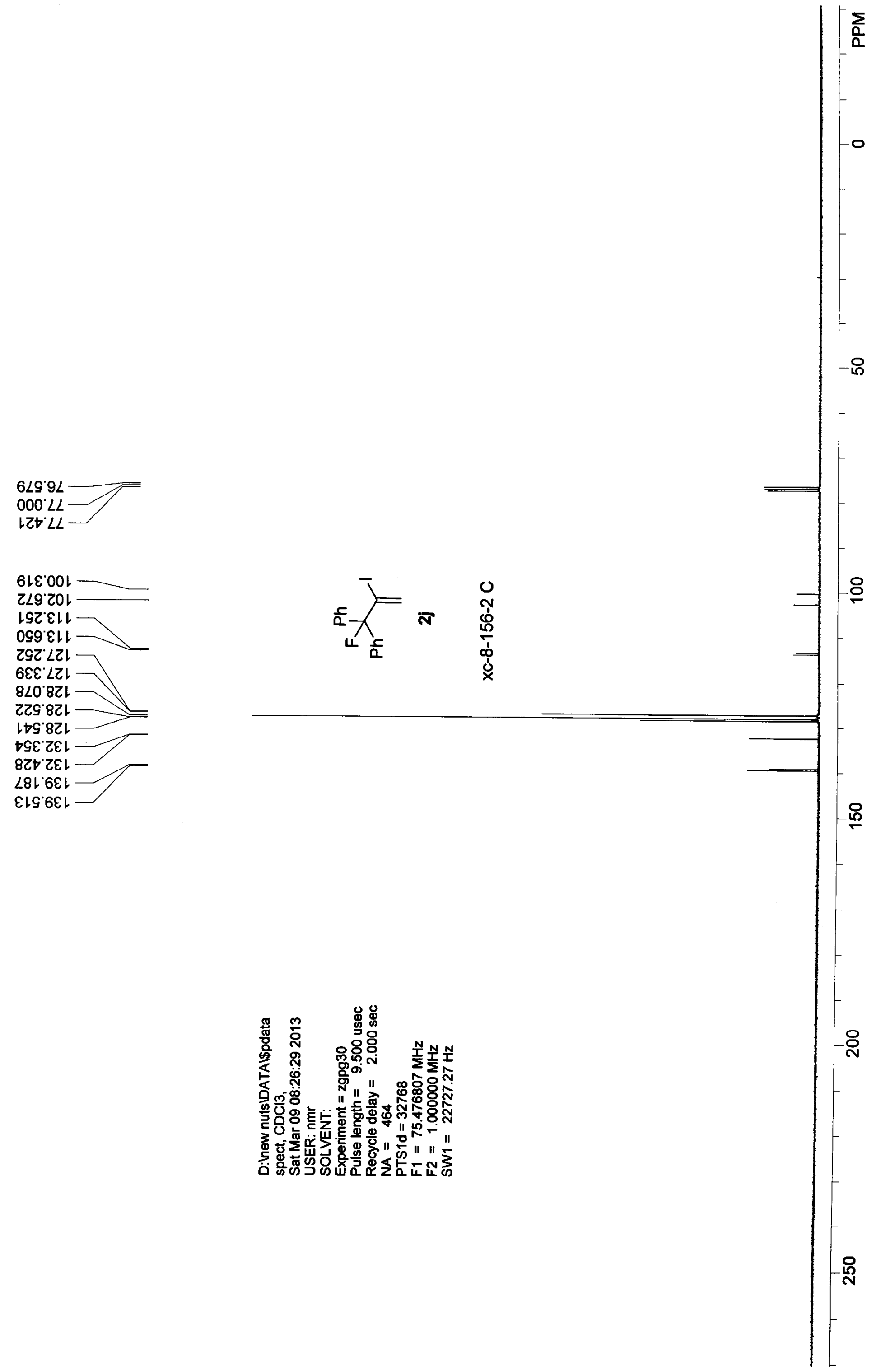
xc-5-055-2-noe

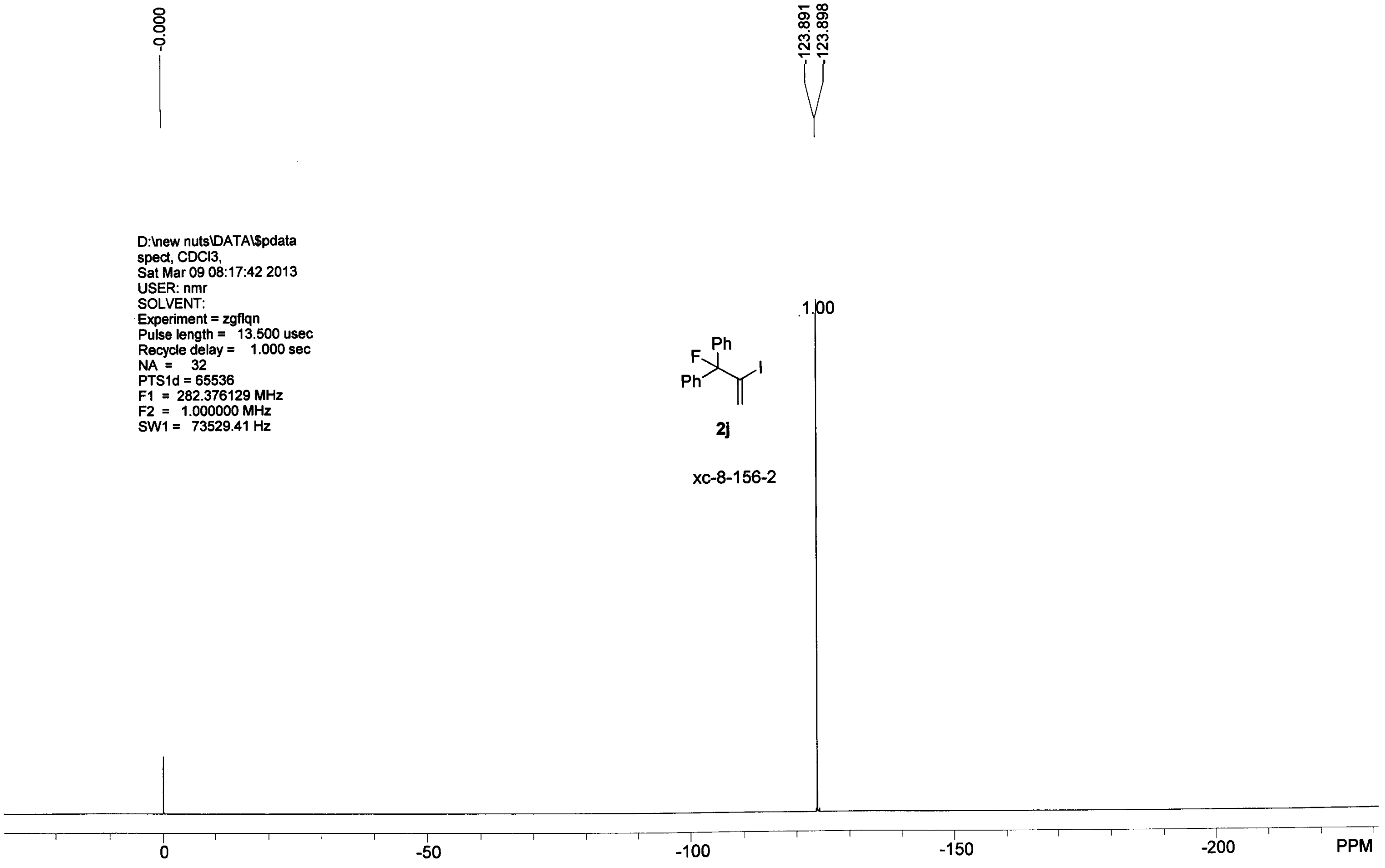



xc-8-156-cp F
-197.124
-196.955
-196.791
-123.788
-63.098
-0.000
16.50
84.00
133.90
PPM
-200
-150
-100
-50
0
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Mar 05 14:41:48 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz



D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 09 08:20:20 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

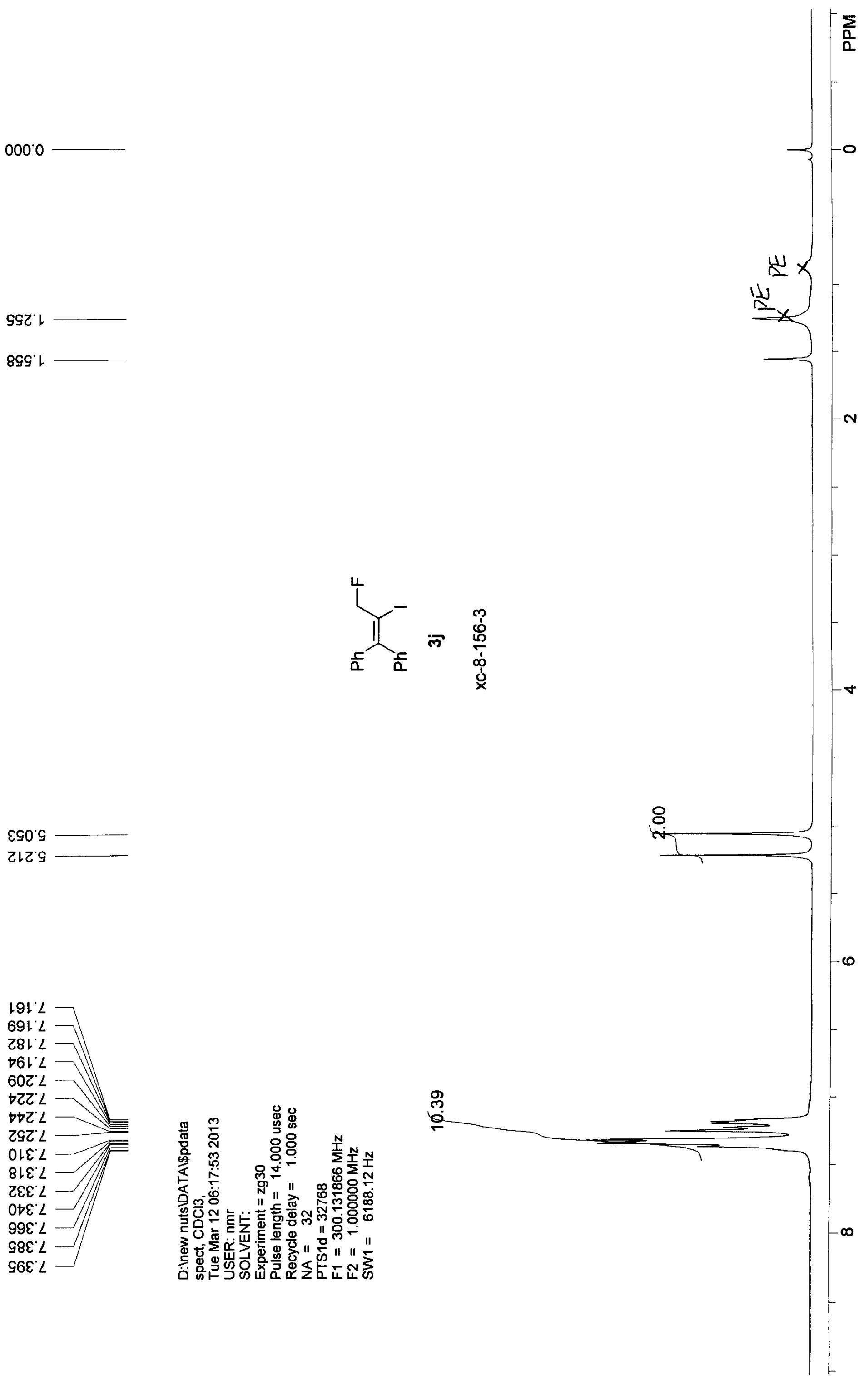

76.579
77.000
77.421
100.319
102.672
113.251
113.650
127.252
127.339
128.078
128.522
128.541
132.354
132.428
139.187
139.513
I
Ph
F
Ph
2j
xc-8-156-2 C
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 09 08:26:29 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 464
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250



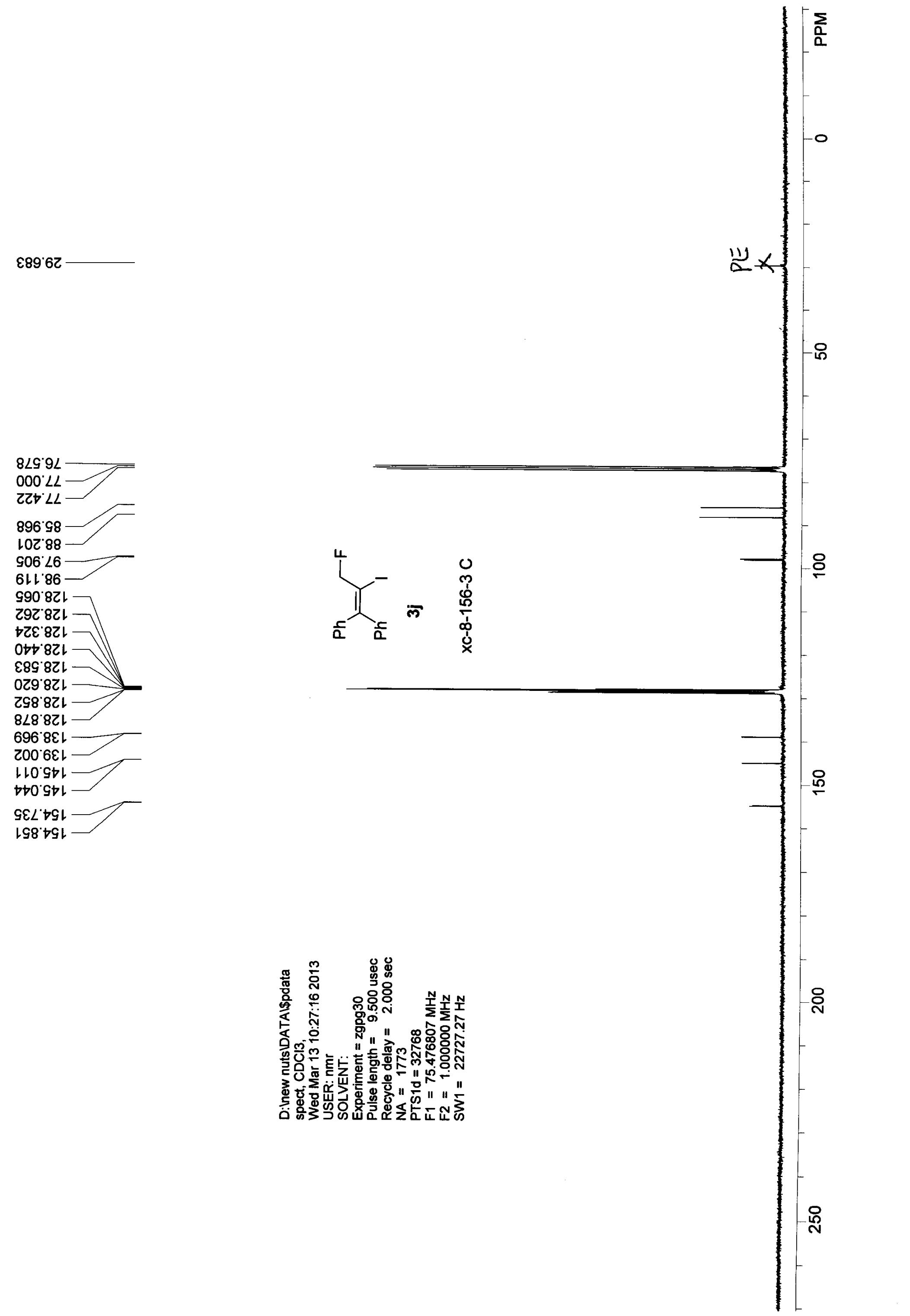
2j

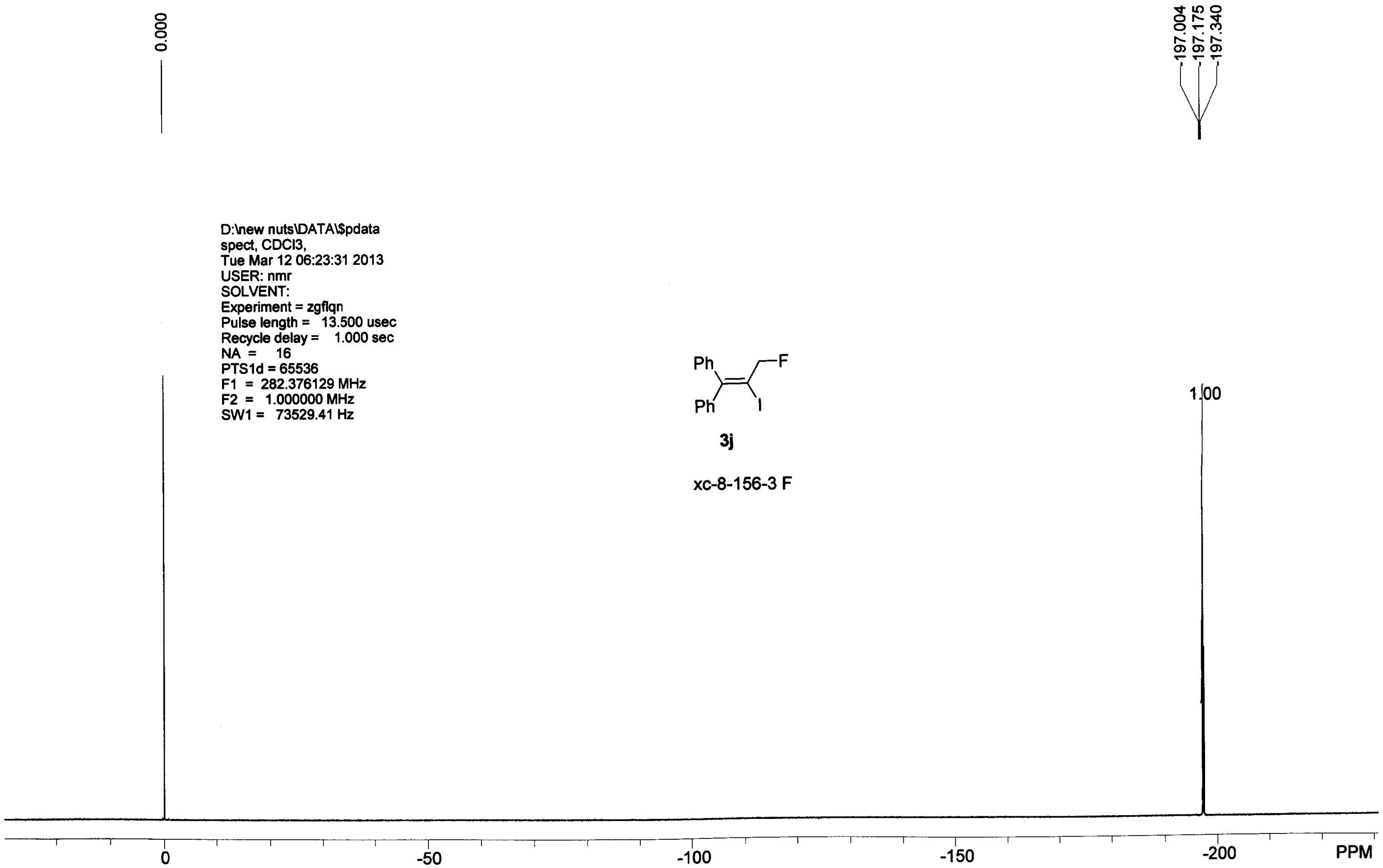
xc-8-156-2

D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 09 08:17:42 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz



```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Mar 12 06:17:53 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length =  14.000 usec
Recycle delay =   1.000 sec
NA =   32
PTS1d = 32768
F1 =  300.131866 MHz
F2 =   1.000000 MHz
SW1 =   6188.12 Hz
```

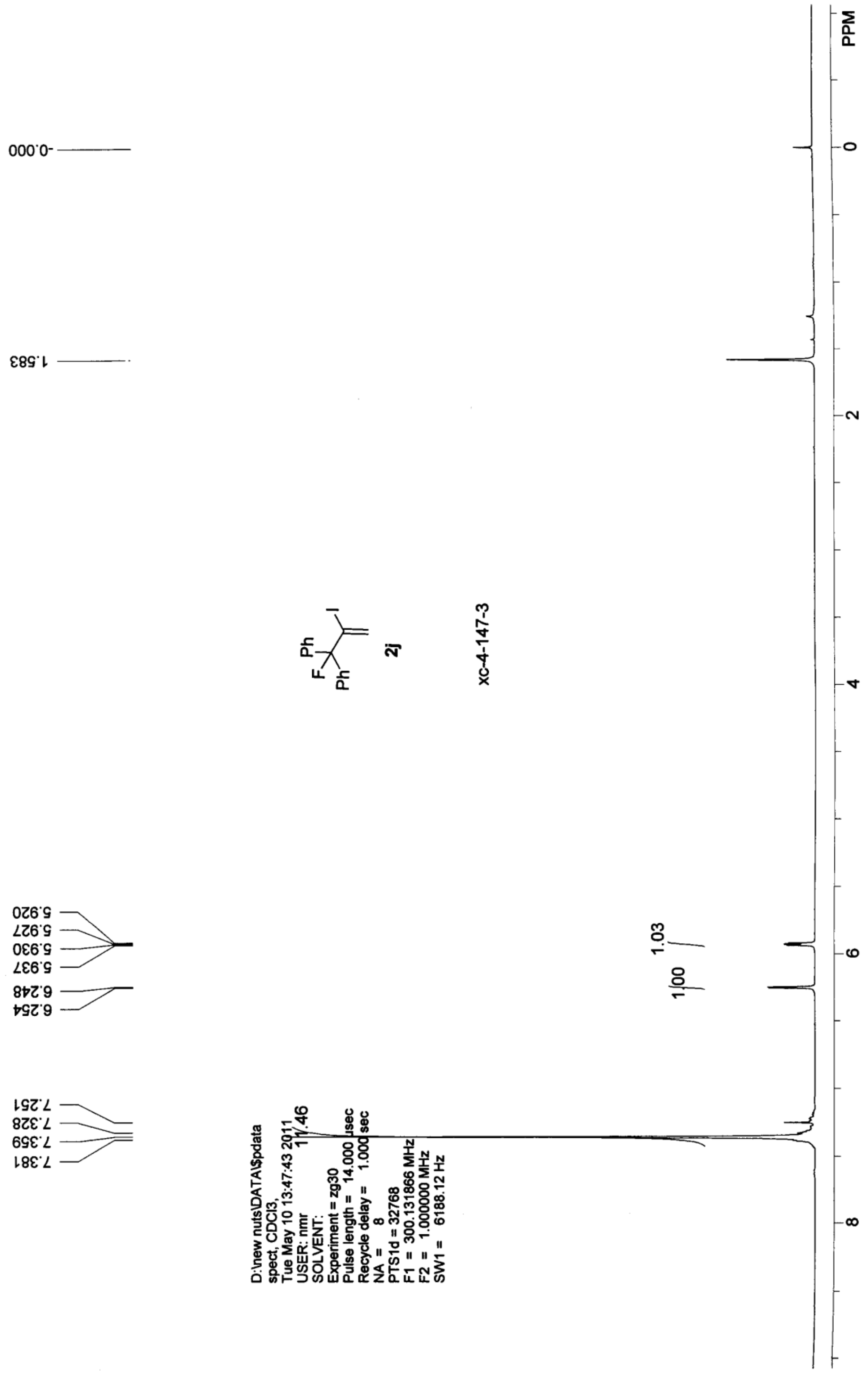

29.683
76.578
77.000
77.422
85.968
88.201
97.905
98.119
128.065
128.262
128.324
128.440
128.583
128.620
128.852
128.878
138.969
139.002
145.011
145.044
154.735
154.851
Ph
Ph
F
I
3j
xc-8-156-3 C
PE
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 13 10:27:16 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1773
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250



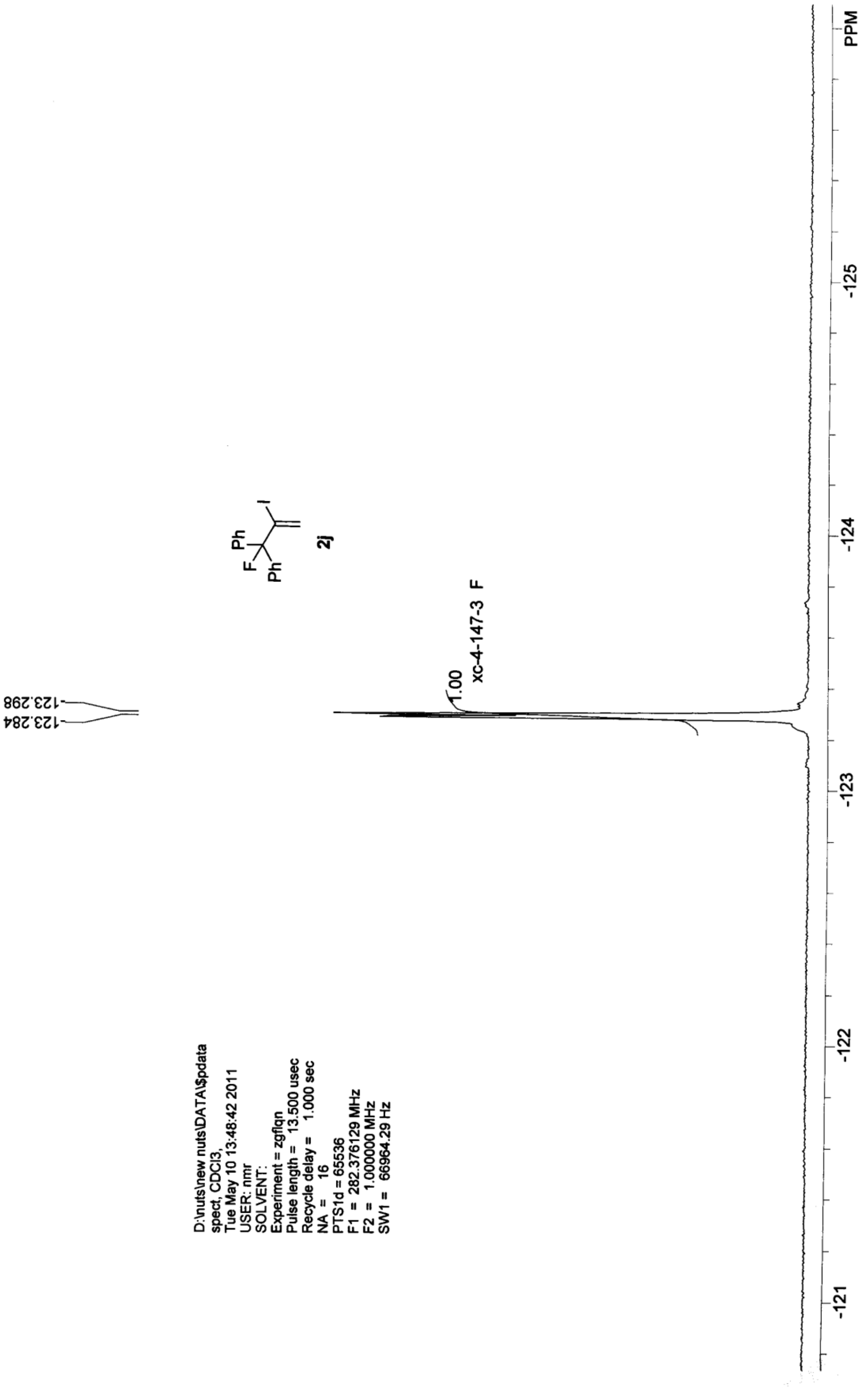
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Mar 12 06:23:31 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

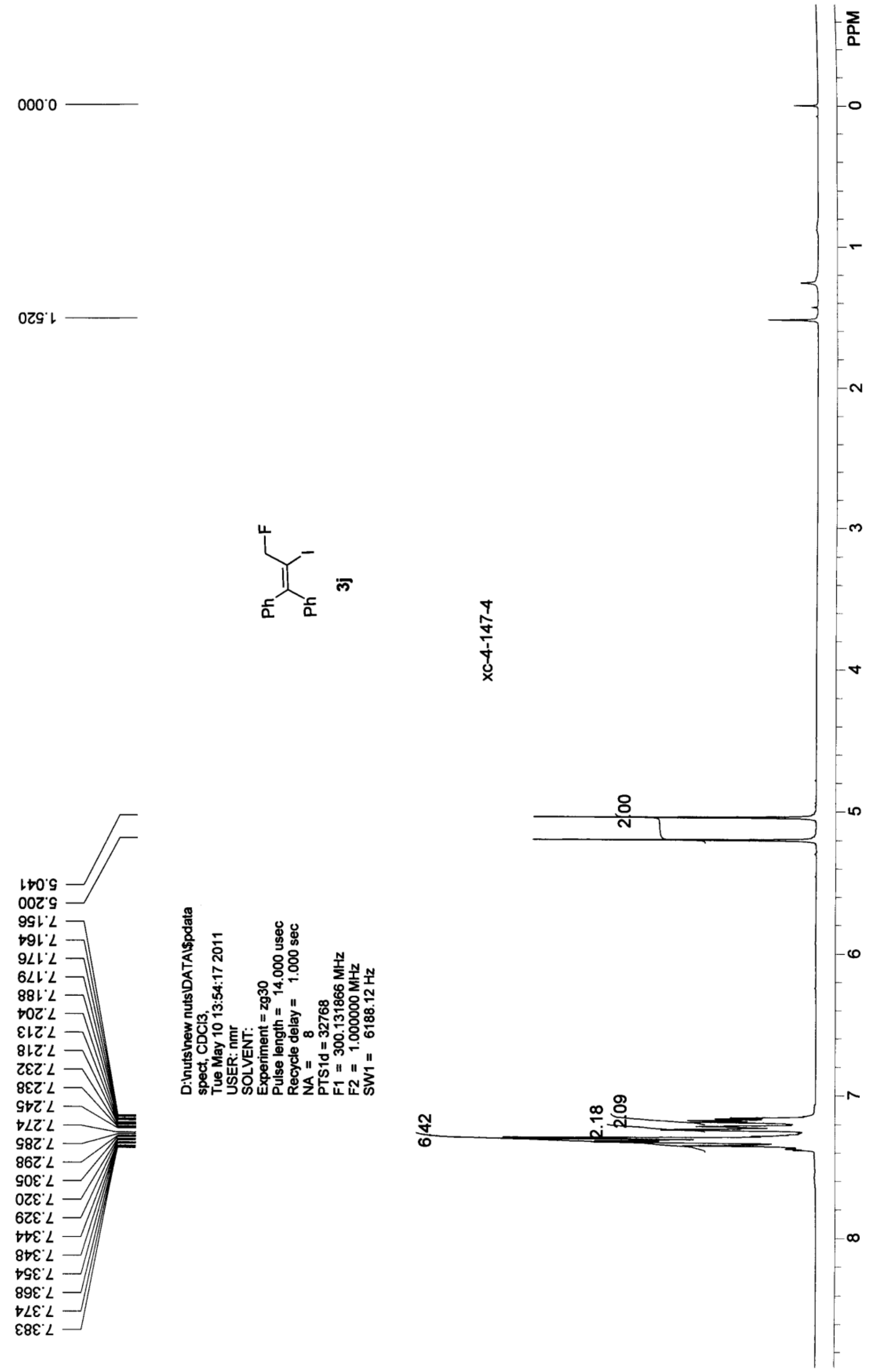
3j

xc-8-156-3 F



```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 10 13:47:43 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
```

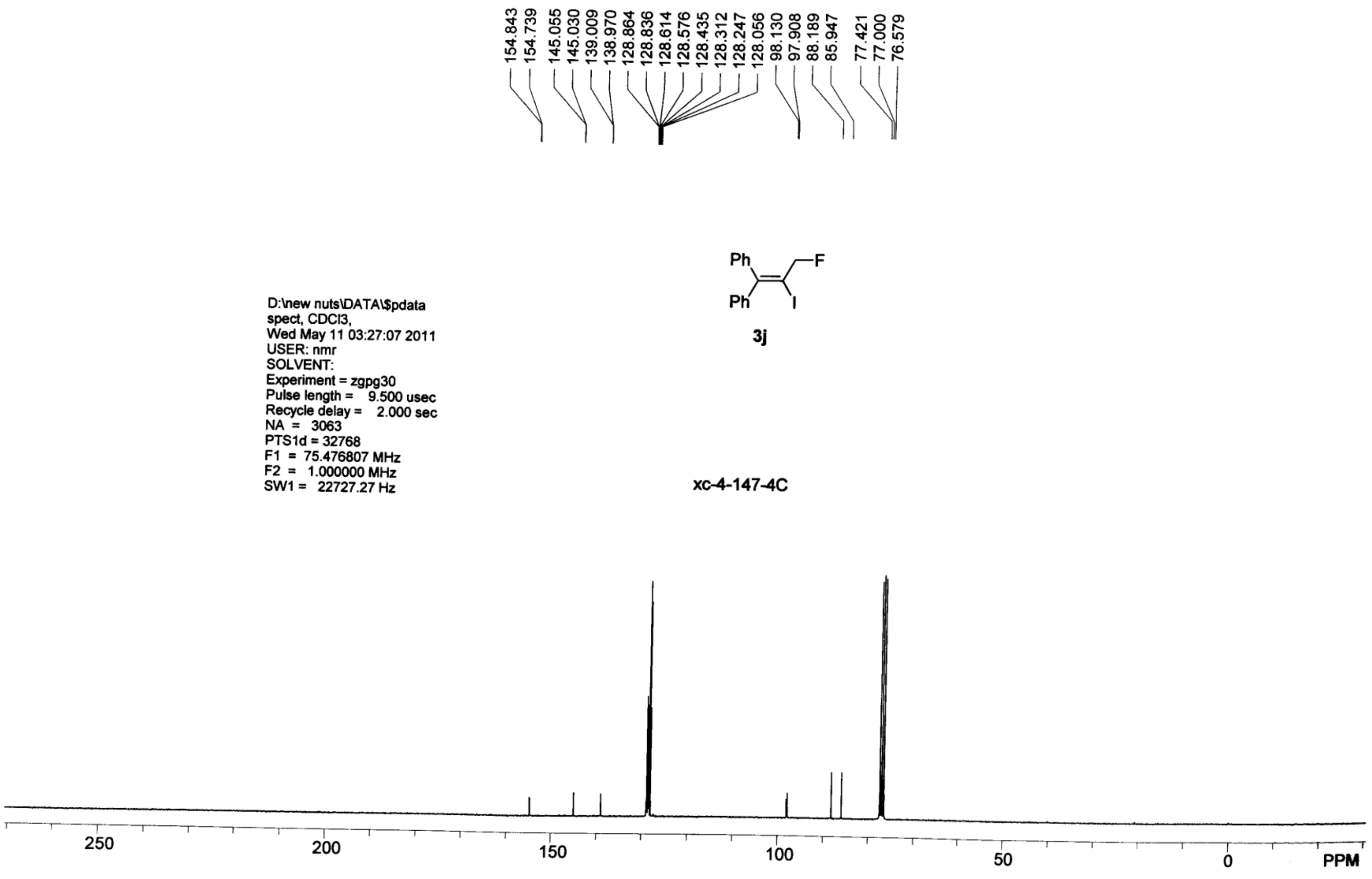

-123.298
-123.284
F
Ph
Ph
I
2j
1.00
xc-4-147-3 F
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 10 13:48:42 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-125
-124
-123
-122
-121



3j

xc-4-147-4

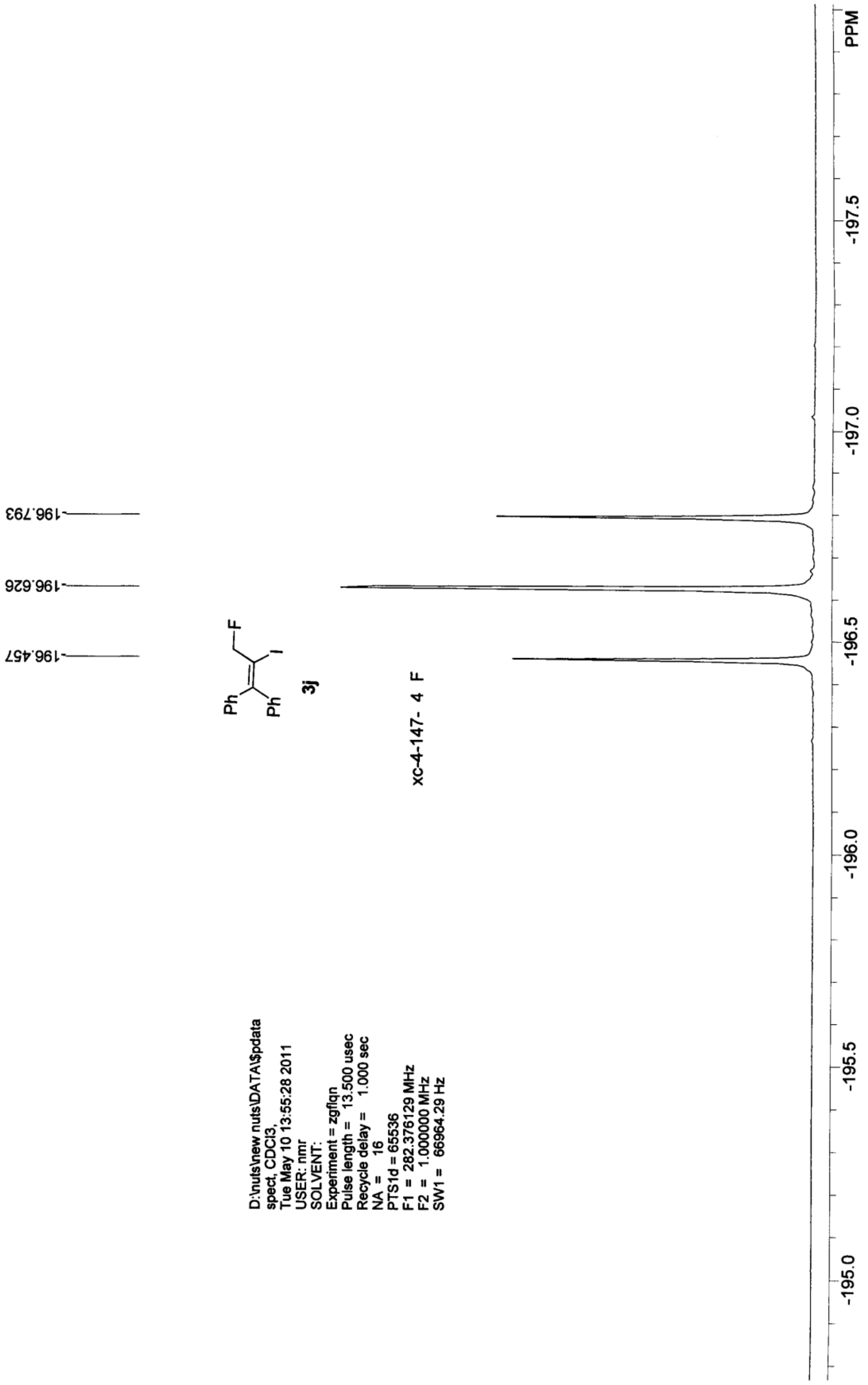
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 10 13:54:17 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

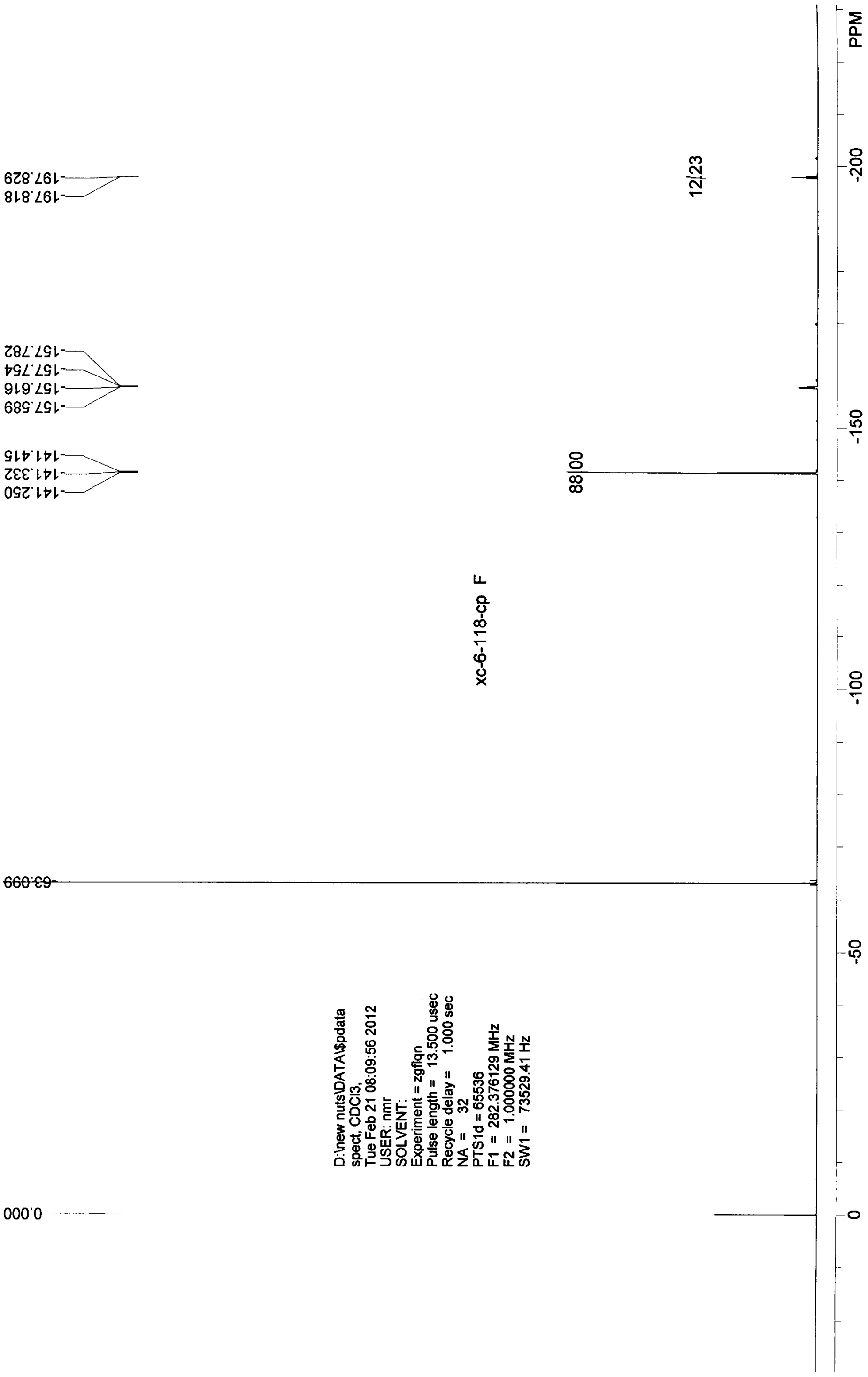


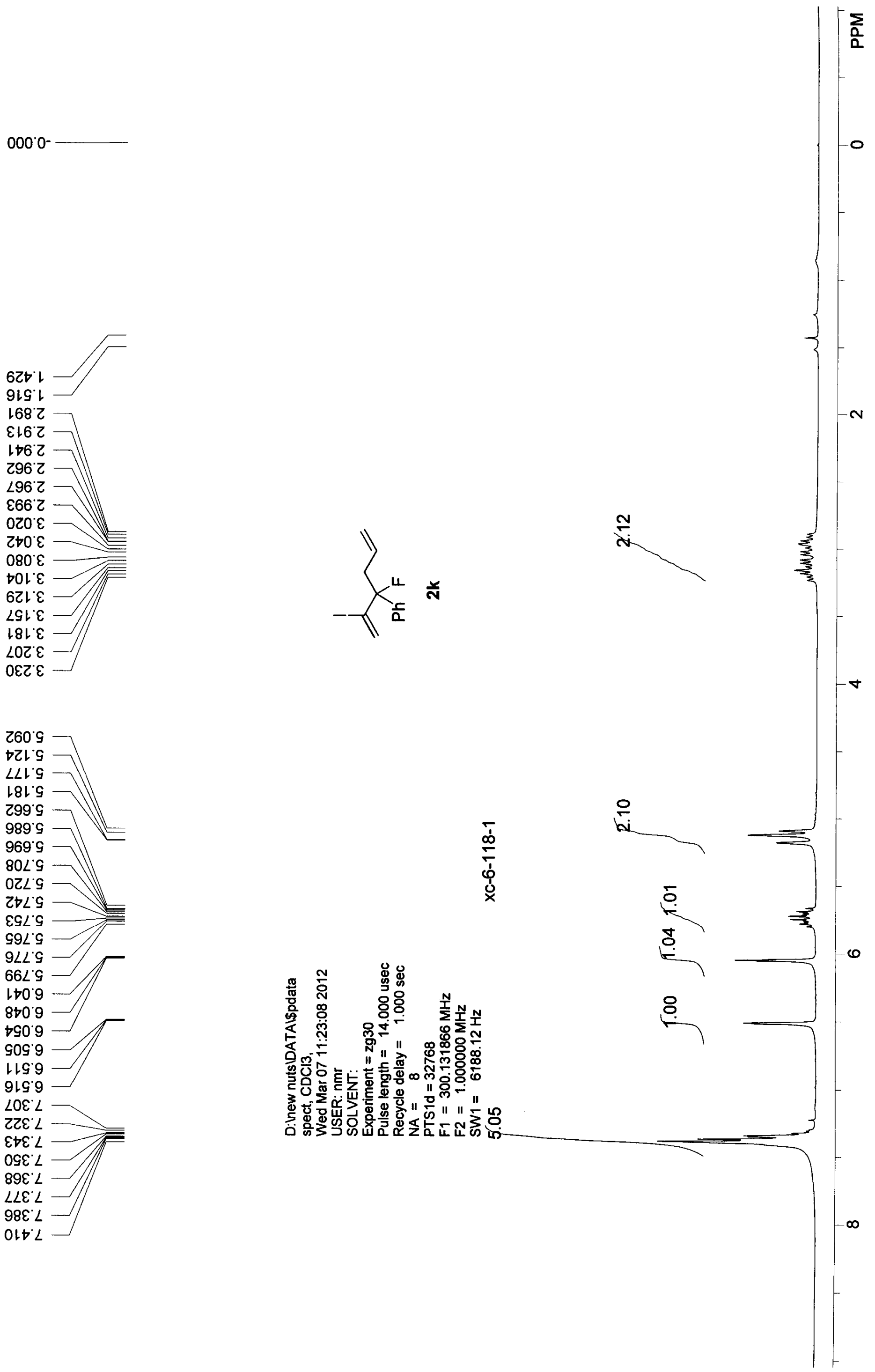
3j

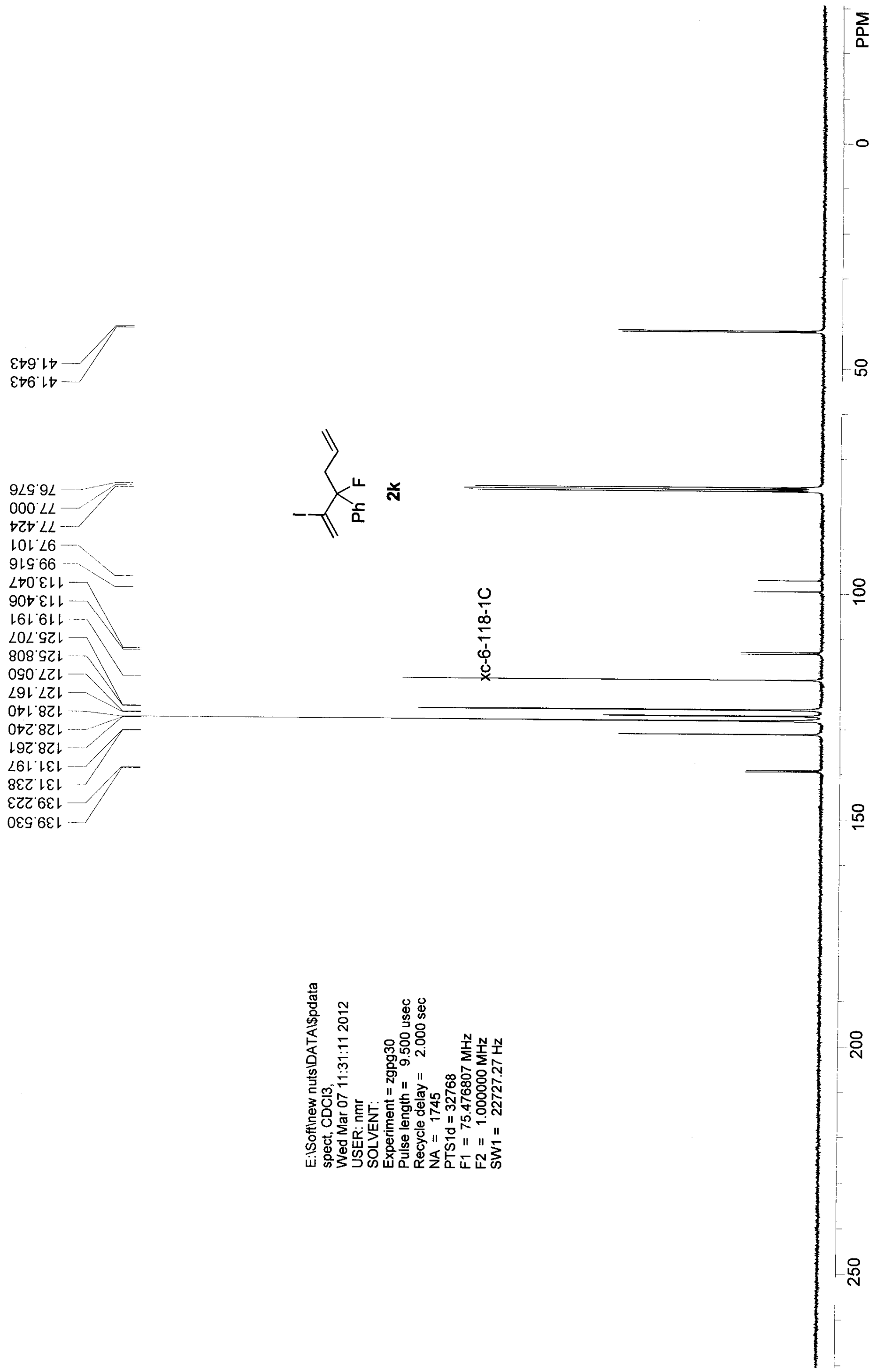
```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed May 11 03:27:07 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length =    9.500 usec
Recycle delay =    2.000 sec
NA  =  3063
PTS1d = 32768
F1  =  75.476807 MHz
F2  =   1.000000 MHz
SW1 =   22727.27 Hz
```

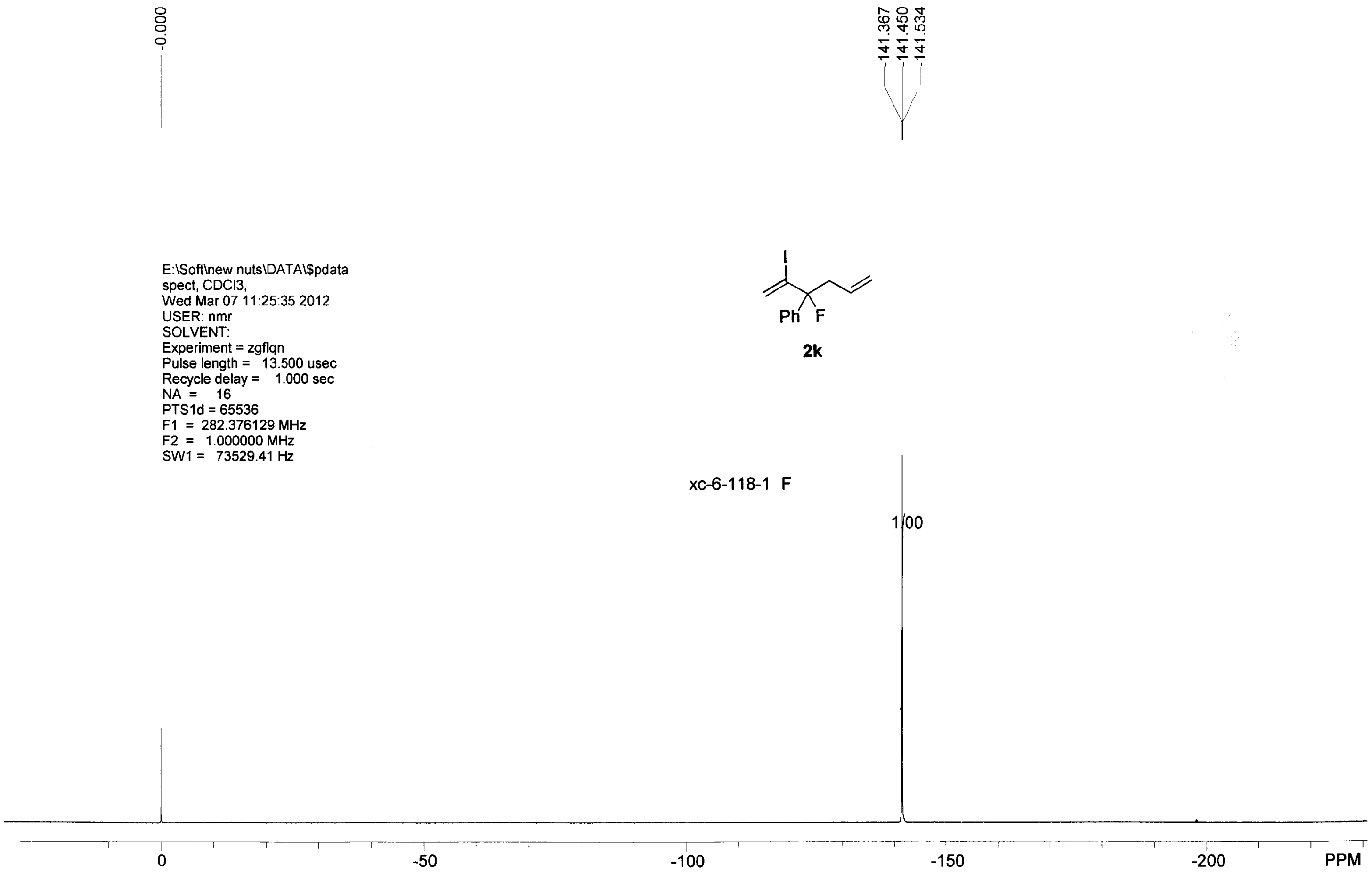
xc-4-147-4C


-196.793
-196.626
-196.457
Ph
Ph
F
I
3j
xc-4-147- 4 F
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 10 13:55:28 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-197.5
-197.0
-196.5
-196.0
-195.5
-195.0


-197.829
-197.818
-157.782
-157.754
-157.616
-157.589
-141.415
-141.332
-141.250
-63.099
0.000
12.23
88.00
xc-6-118-cp F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Feb 21 08:09:56 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
PPM
-200
-150
-100
-50
0

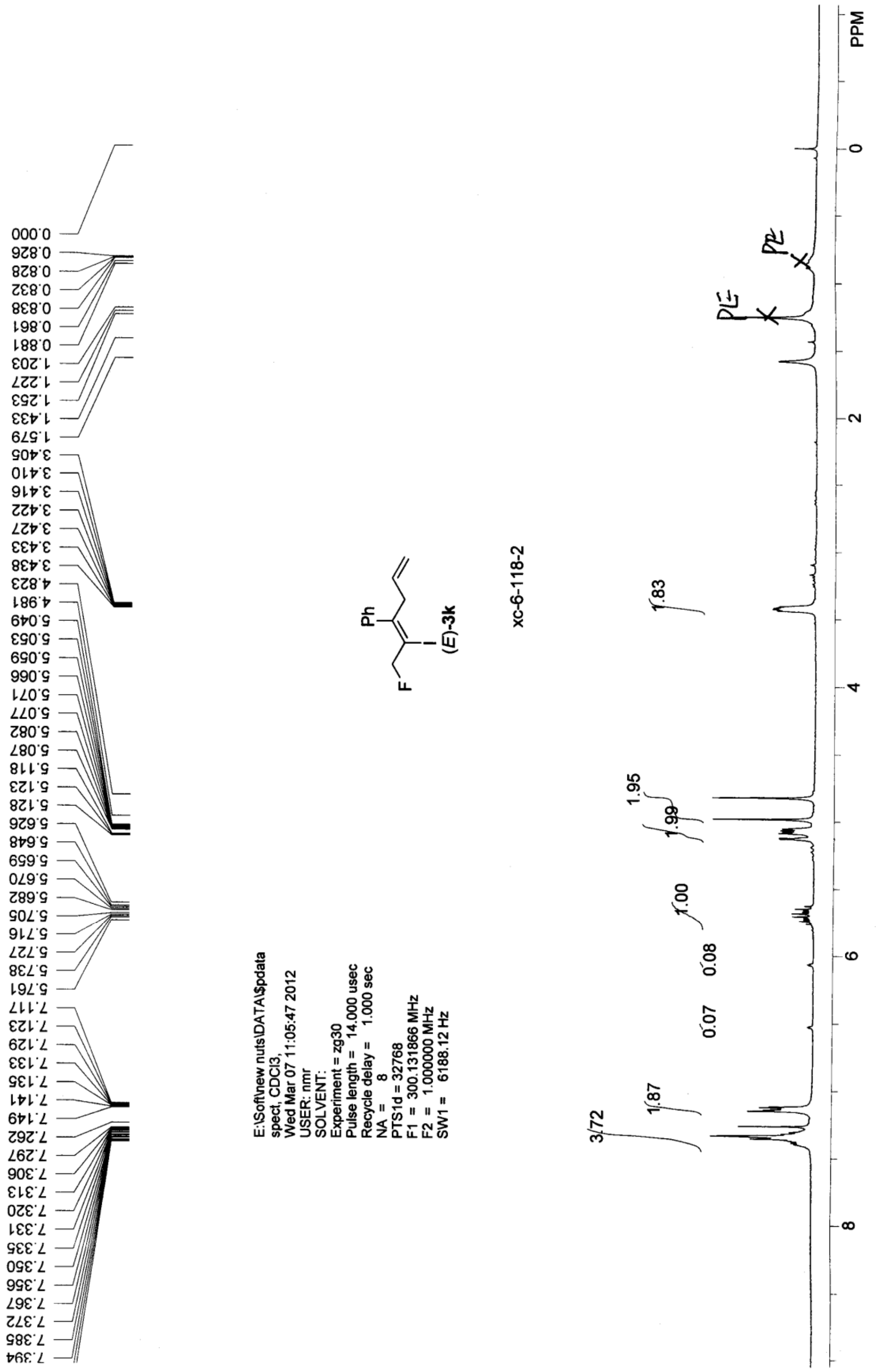

-0.000
1.429
1.516
2.891
2.913
2.941
2.962
2.967
2.993
3.020
3.042
3.080
3.104
3.129
3.157
3.181
3.207
3.230
5.092
5.124
5.177
5.181
5.662
5.686
5.696
5.708
5.720
5.742
5.753
5.765
5.776
5.799
6.041
6.048
6.054
6.505
6.511
6.516
7.307
7.322
7.343
7.350
7.368
7.377
7.386
7.410
Ph
F
2k
xc-6-118-1
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 07 11:23:08 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.12
2.10
1.01
1.04
1.00
5.05
PPM
0
2
4
6
8


41.643
41.943
76.576
77.000
77.424
97.101
99.516
113.047
113.406
119.191
125.707
125.808
127.050
127.167
128.140
128.240
128.261
131.197
131.238
139.223
139.530
Ph
F
2k
xc-6-118-1C
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 07 11:31:11 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1745
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

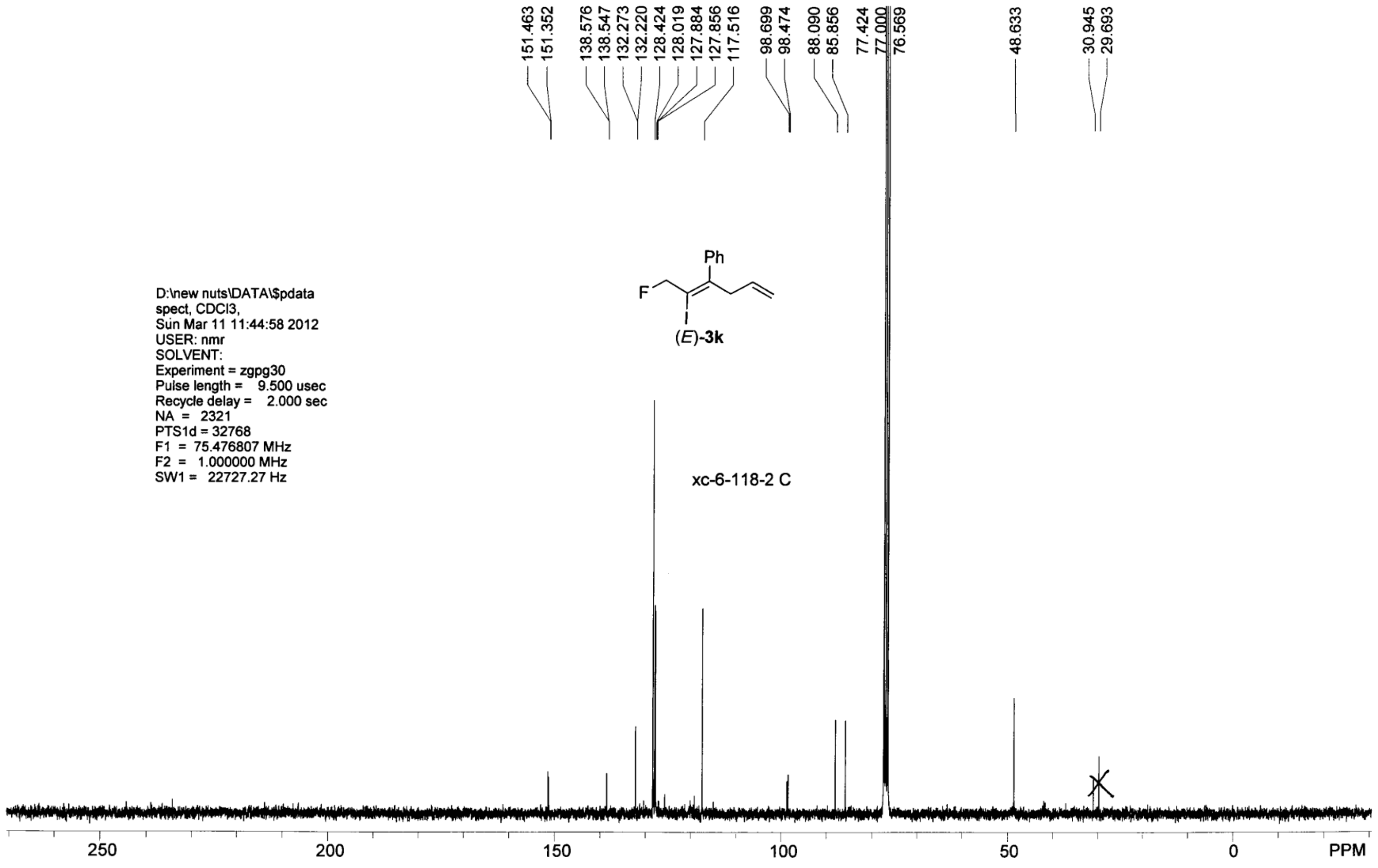


2k

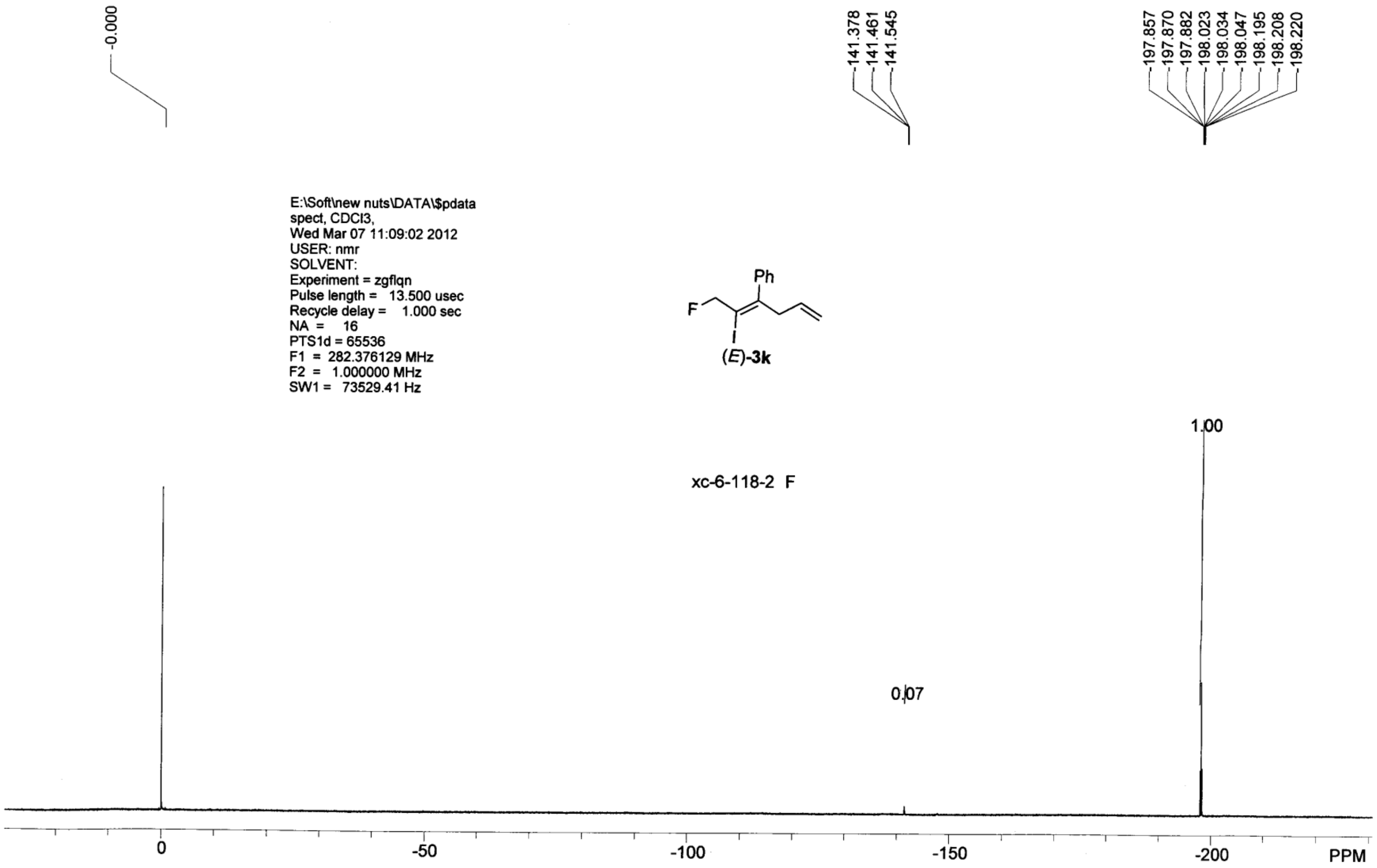
```
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 07 11:25:35 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =   13.500 usec
Recycle delay =    1.000 sec
NA =    16
PTS1d = 65536
F1  =  282.376129 MHz
F2  =   1.000000 MHz
SW1 =   73529.41 Hz
```



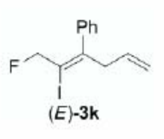
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 07 11:05:47 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Mar 11 11:44:58 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2321
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

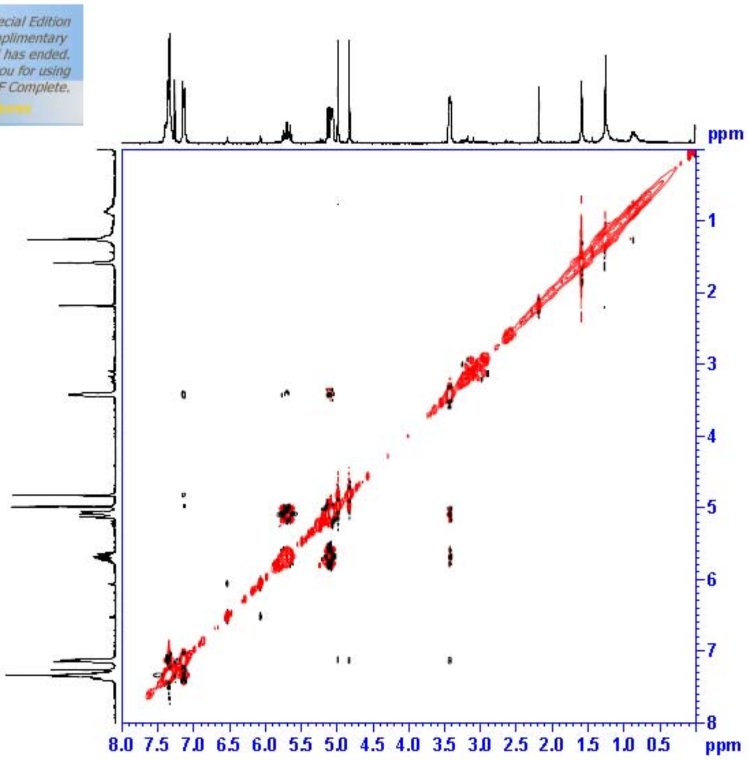


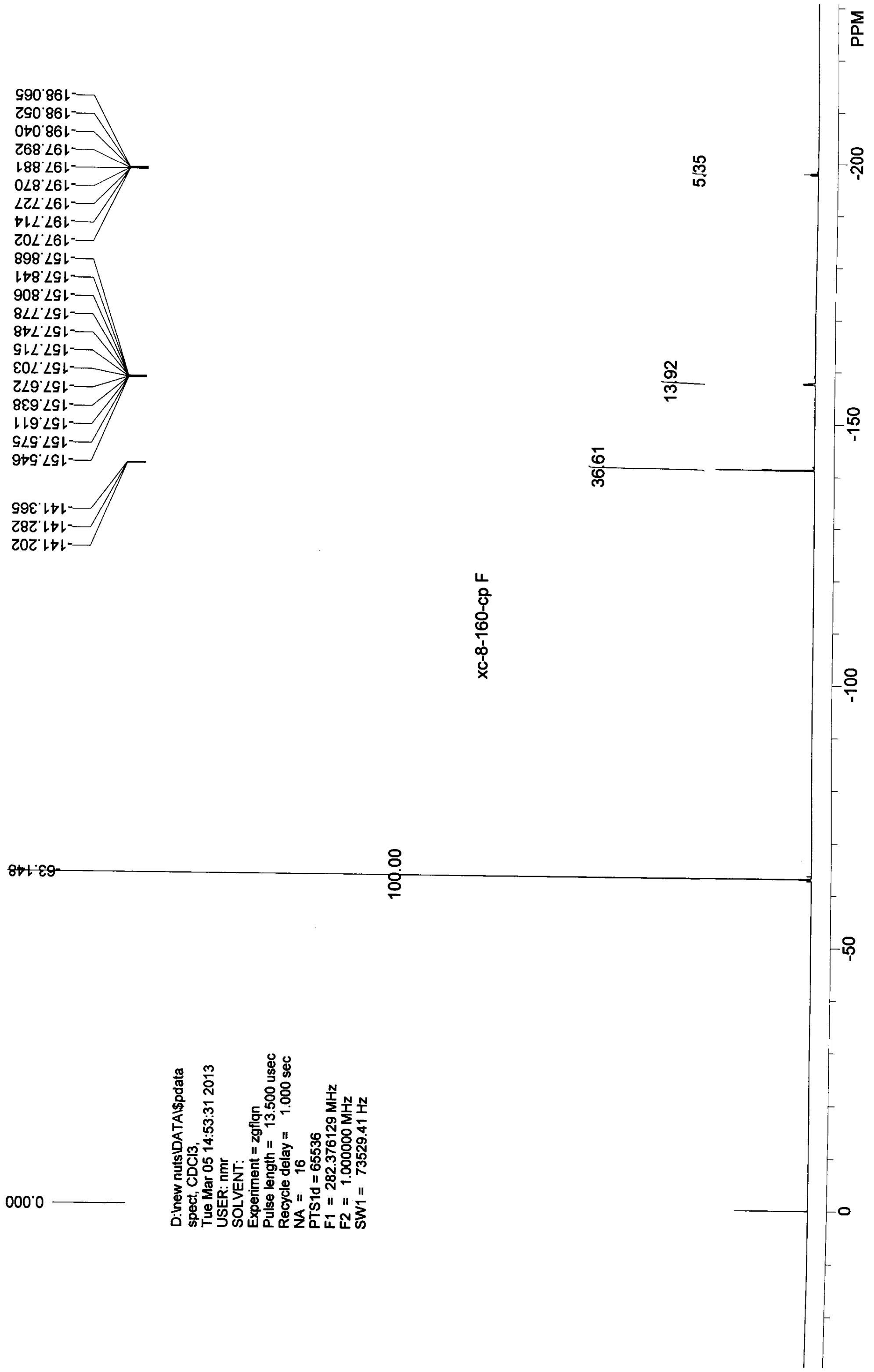
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Mar 07 11:09:02 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

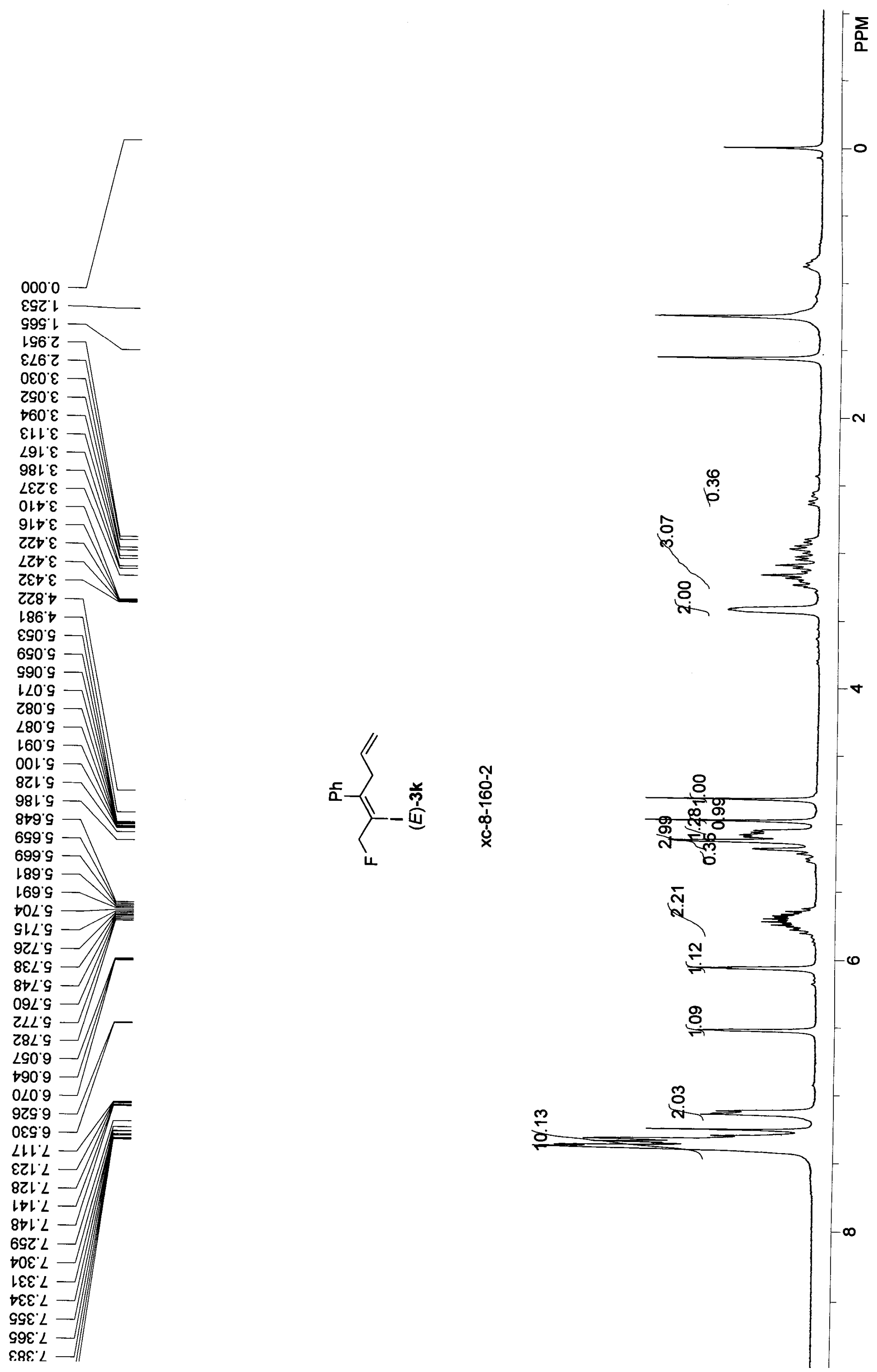


(*E*)-**3k**

xc-6-118-2-noe

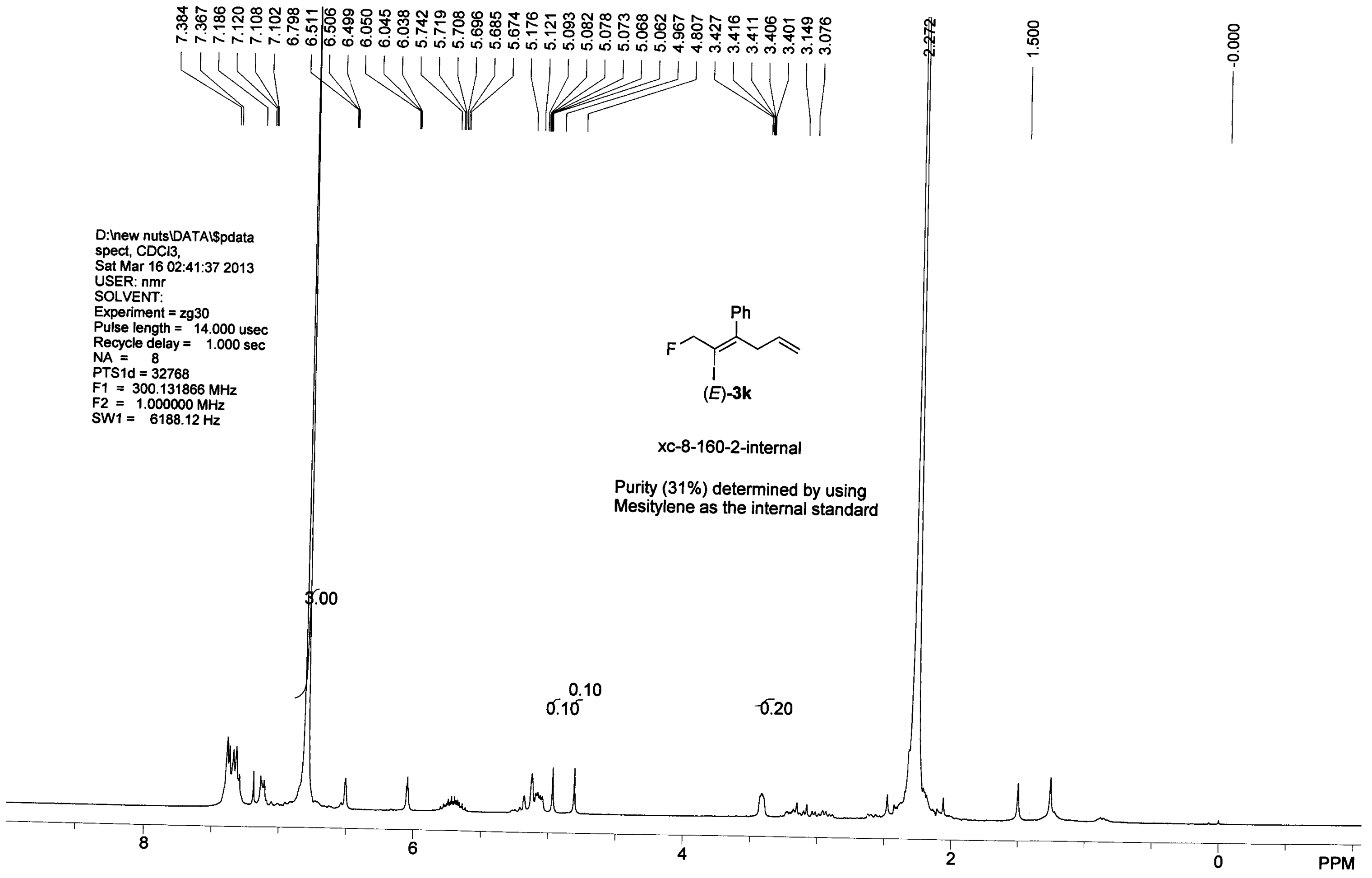



-198.065
-198.052
-198.040
-197.892
-197.881
-197.870
-197.727
-197.714
-197.702
-157.868
-157.841
-157.806
-157.778
-157.748
-157.715
-157.703
-157.672
-157.638
-157.611
-157.575
-157.546
-141.365
-141.282
-141.202
-63.148
0.000
5.35
13.92
36.61
100.00
xc-8-160-cp F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Mar 05 14:53:31 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
PPM
-200
-150
-100
-50
0



(E)-3k

0.000
1.253
1.565
2.951
2.973
3.030
3.052
3.094
3.113
3.167
3.186
3.237
3.410
3.416
3.422
3.427
3.432
4.822
4.981
5.053
5.059
5.065
5.071
5.082
5.087
5.091
5.100
5.128
5.186
5.648
5.659
5.669
5.681
5.691
5.704
5.715
5.726
5.738
5.748
5.760
5.772
5.782
6.057
6.064
6.070
6.526
6.530
7.117
7.123
7.128
7.141
7.148
7.259
7.304
7.331
7.334
7.355
7.365
7.383
Ph
F
I
xc-8-160-2
10.13
2.03
1.09
1.12
2.21
2.99
0.35
1.28
1.00
0.99
2.00
3.07
0.36
8
6
4
2
0
PPM

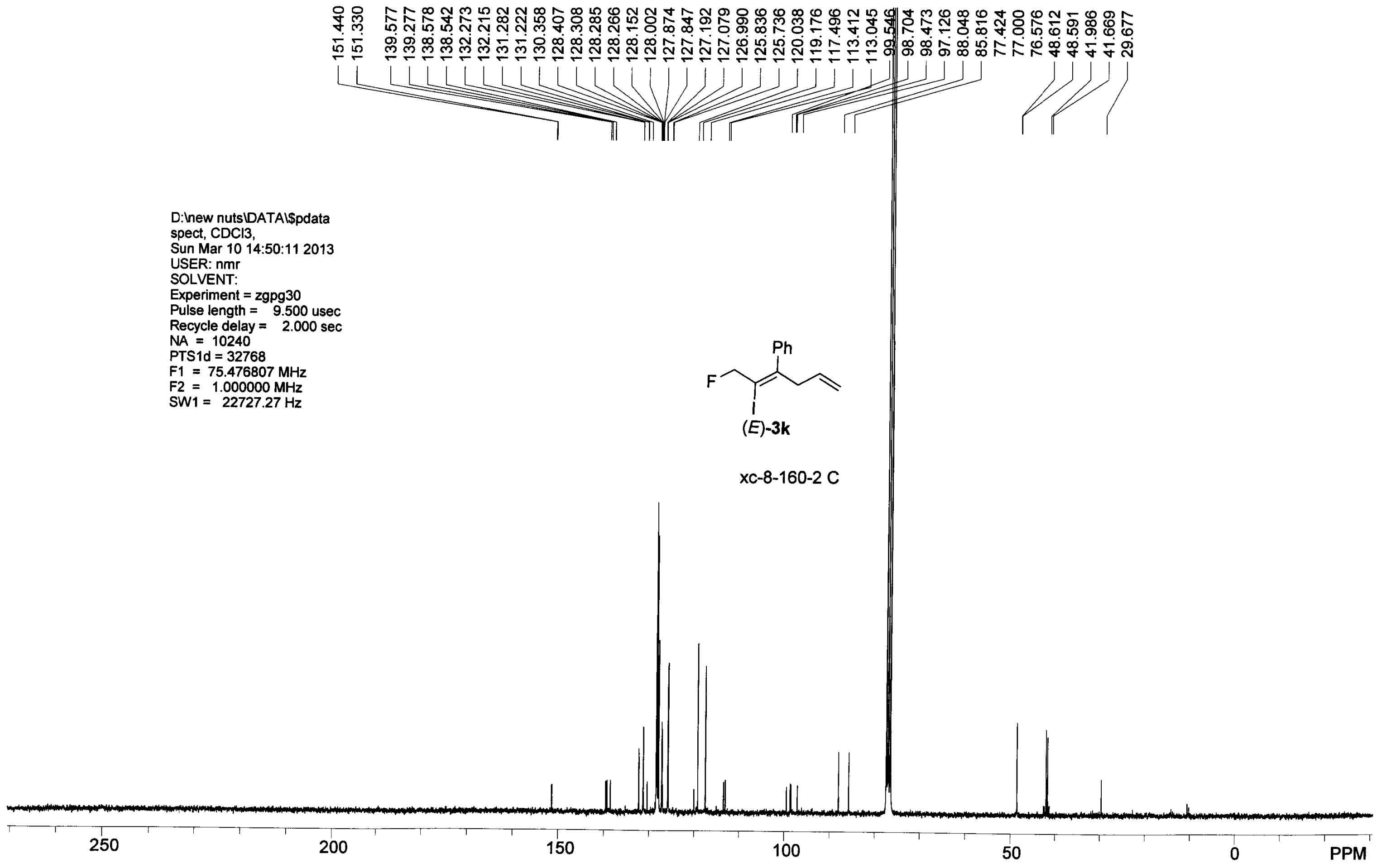


D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 16 02:41:37 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

(*E*)-**3k**

xc-8-160-2-internal

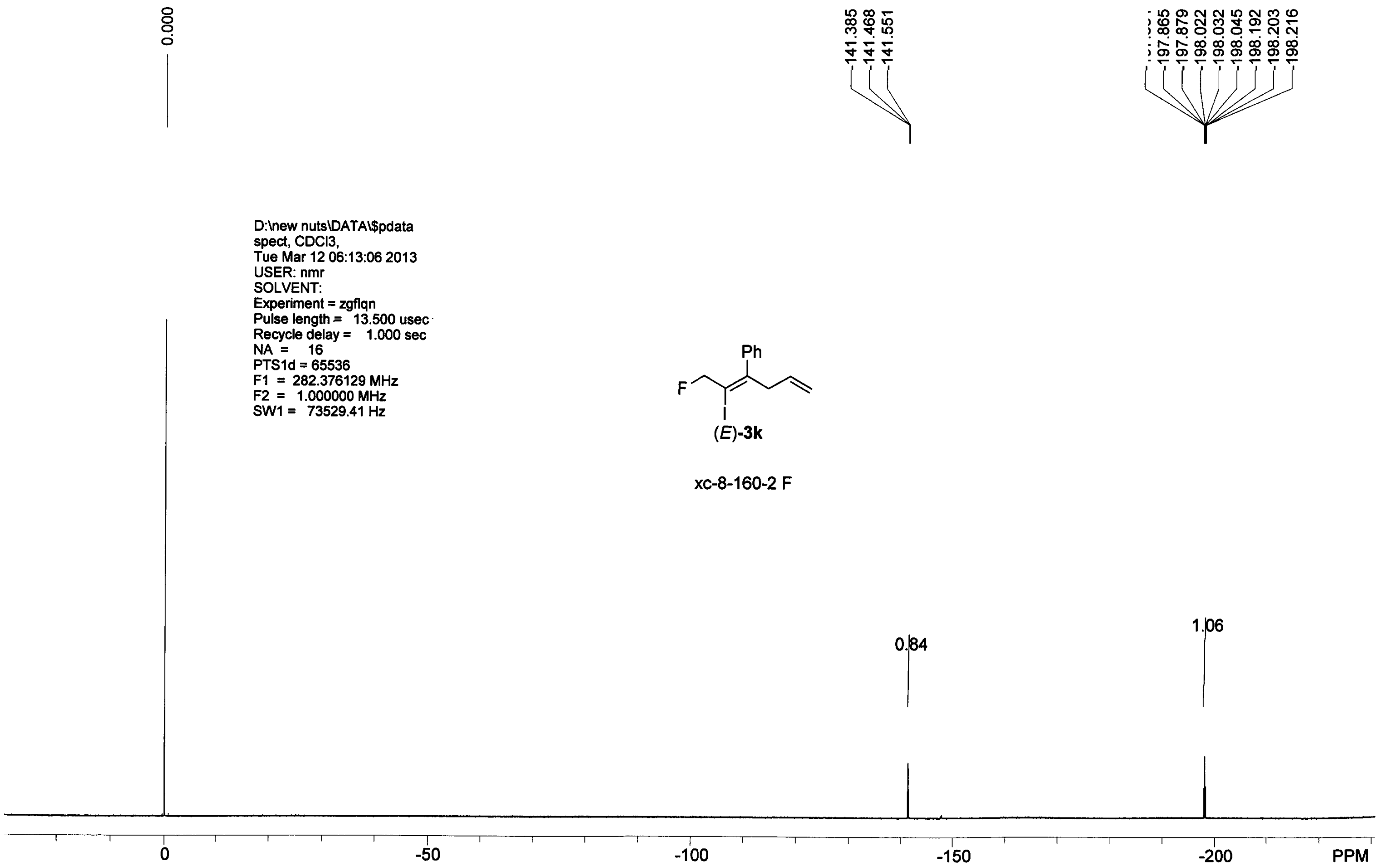
Purity (31%) determined by using
Mesitylene as the internal standard



```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Mar 10 14:50:11 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length =    9.500 usec
Recycle delay =    2.000 sec
NA  =  10240
PTS1d = 32768
F1  =  75.476807 MHz
F2  =   1.000000 MHz
SW1 =   22727.27 Hz
```

(*E*)-**3k**

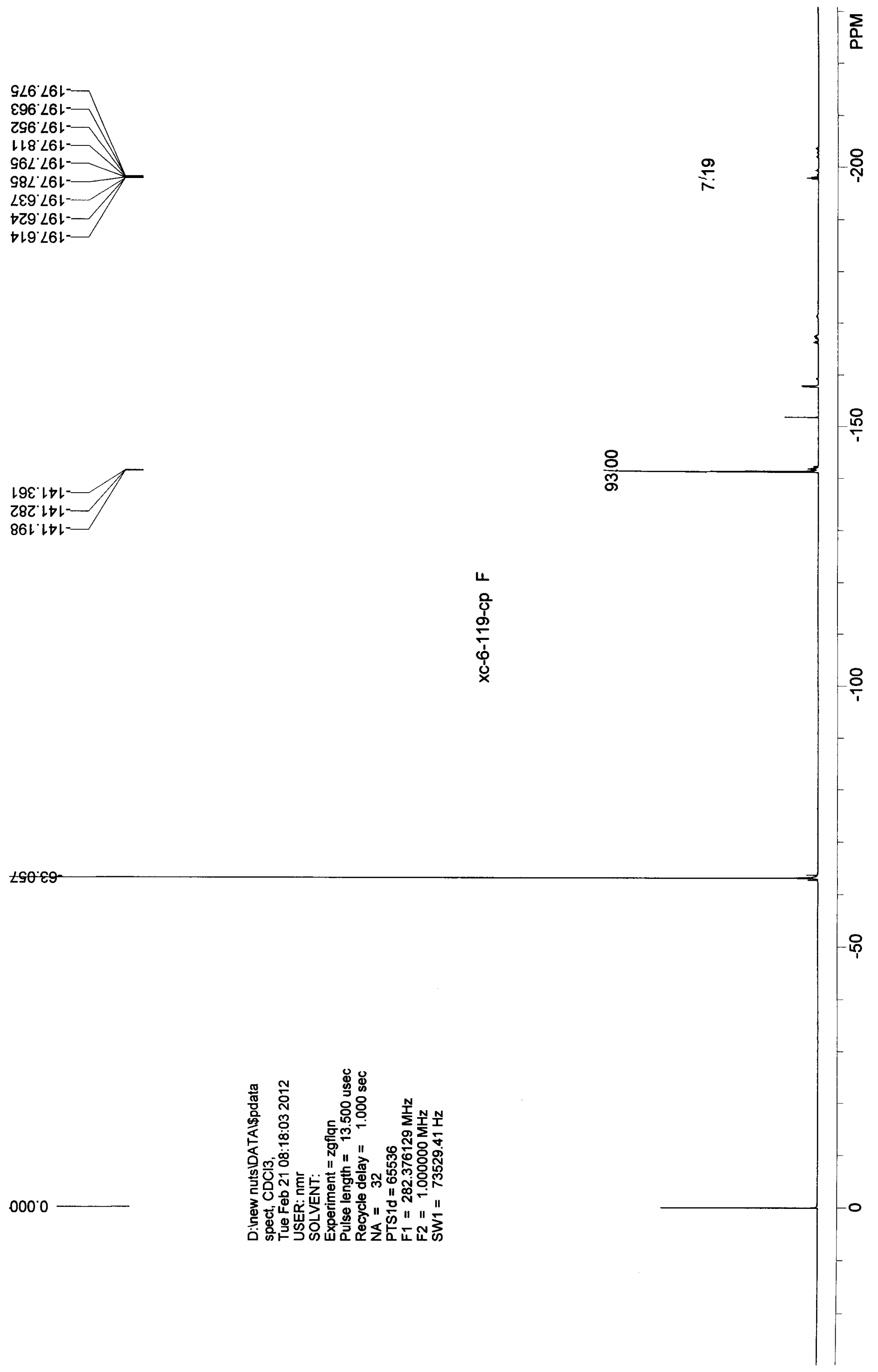
xc-8-160-2 C



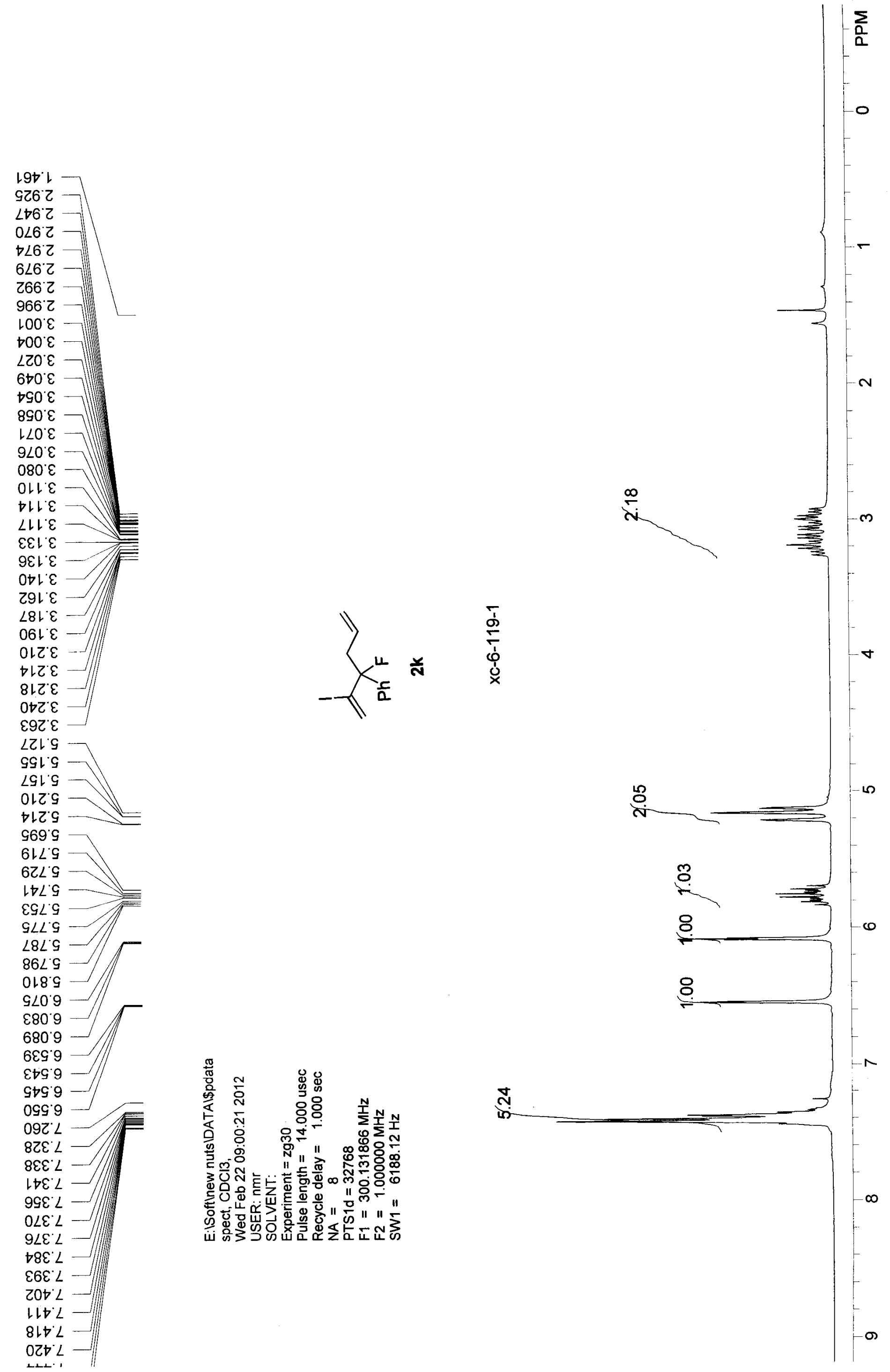
(*E*)-**3k**

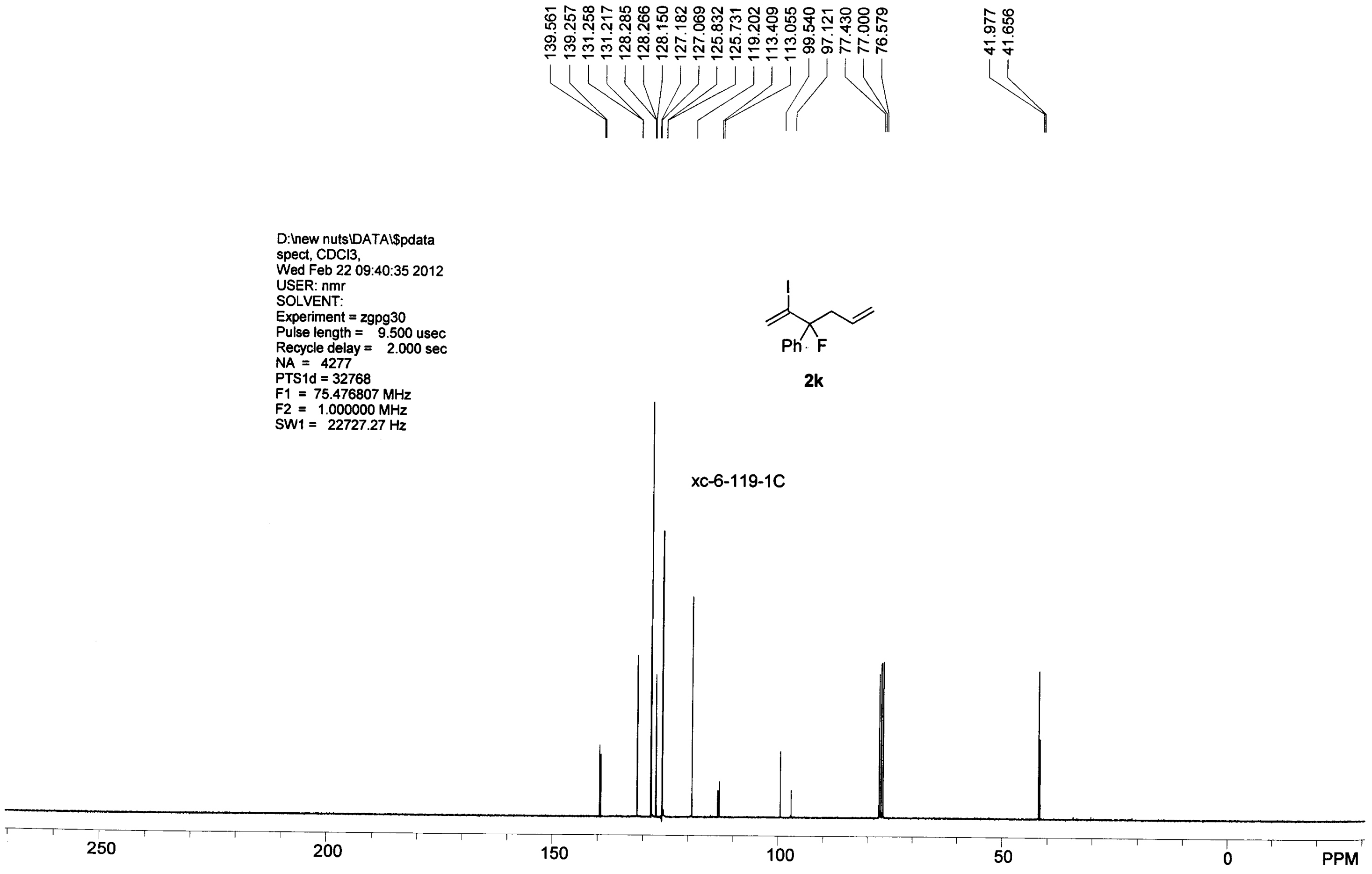
xc-8-160-2 F

```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Mar 12 06:13:06 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =   13.500 usec
Recycle delay =    1.000 sec
NA =    16
PTS1d = 65536
F1 =  282.376129 MHz
F2 =  1.000000 MHz
SW1 =  73529.41 Hz
```



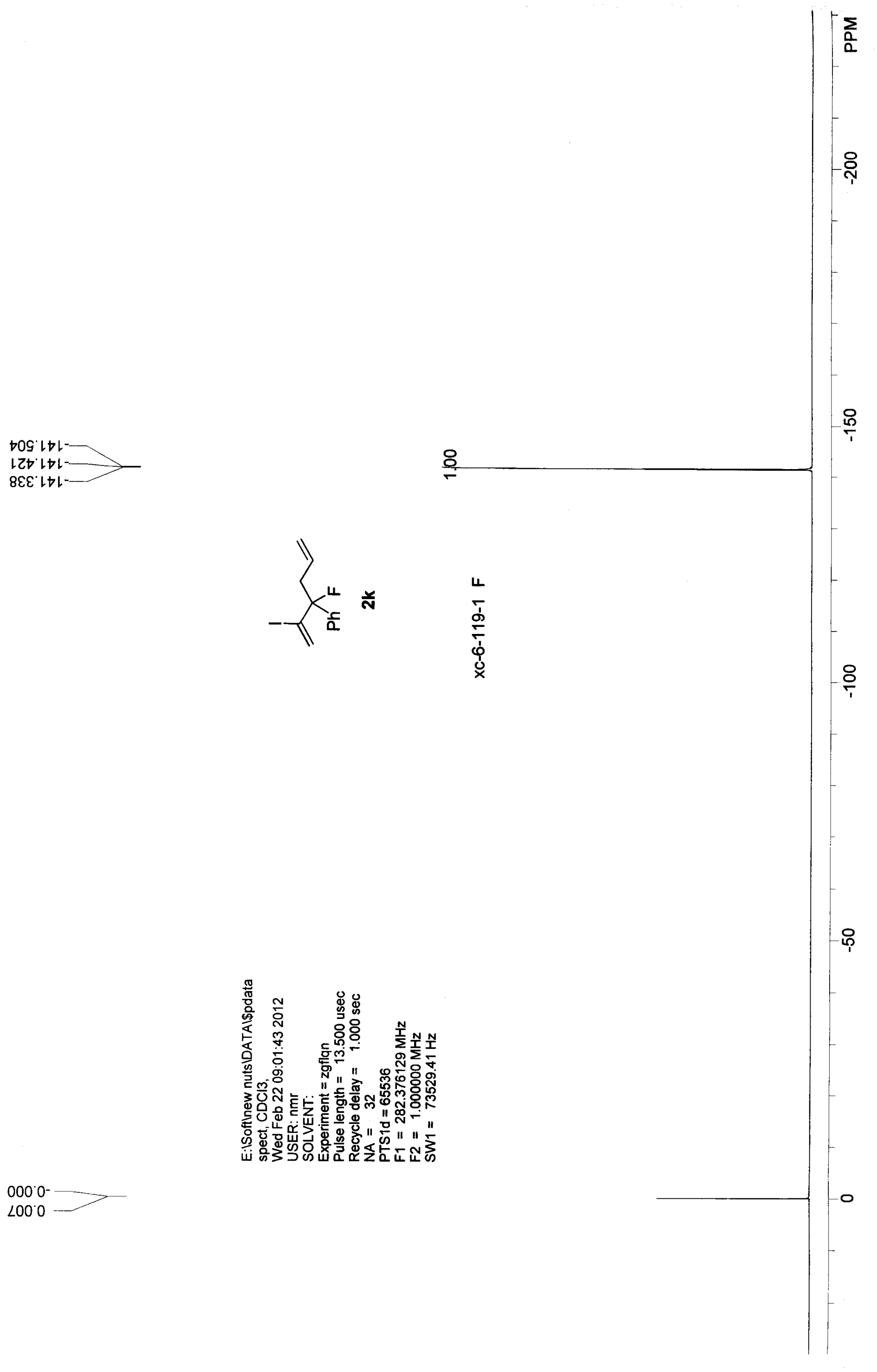
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Feb 21 08:18:03 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

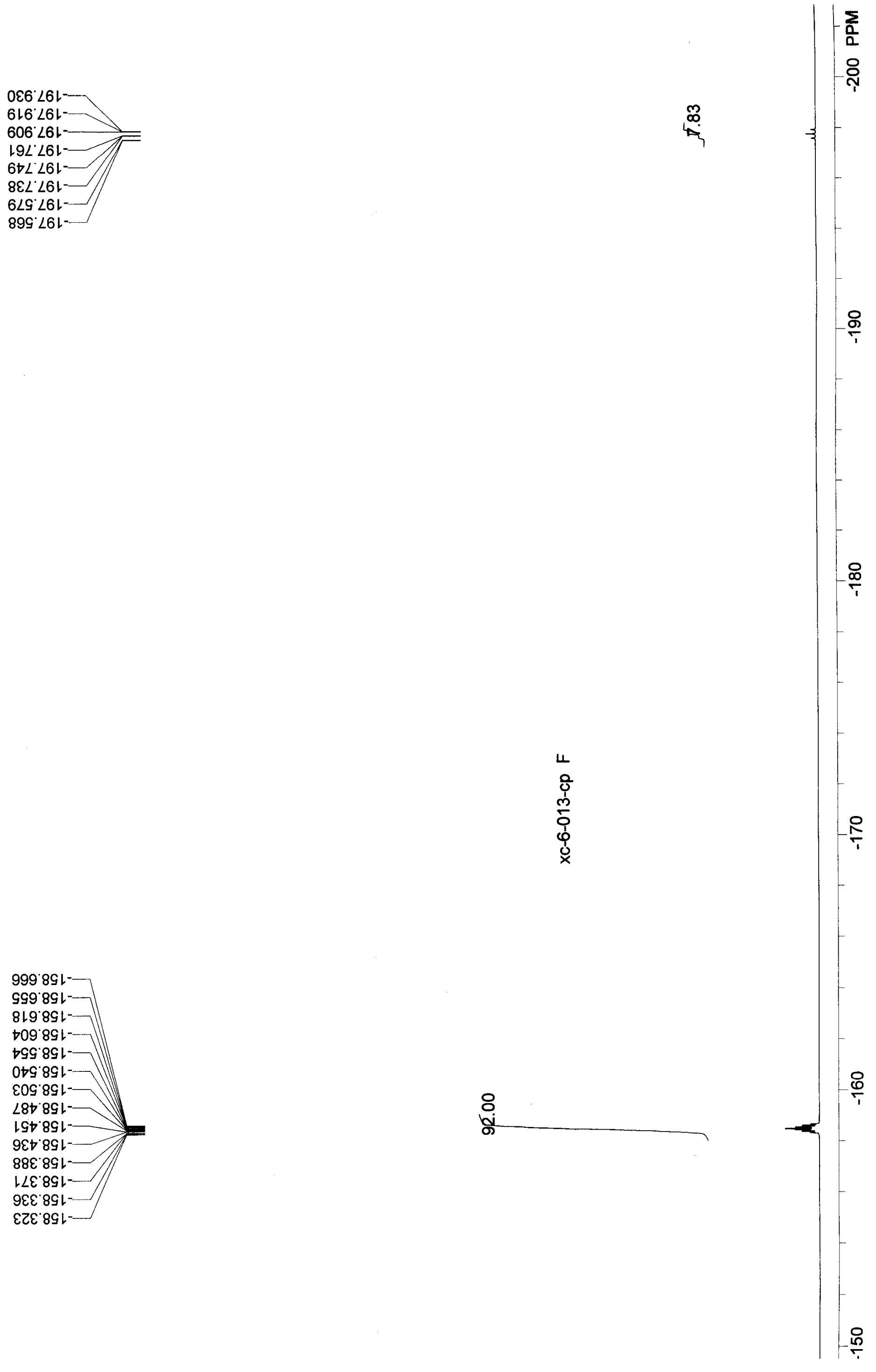

1.461
2.925
2.947
2.970
2.974
2.979
2.992
2.996
3.001
3.004
3.027
3.049
3.054
3.058
3.071
3.076
3.080
3.110
3.114
3.117
3.133
3.136
3.140
3.162
3.187
3.190
3.210
3.214
3.218
3.240
3.263
5.127
5.155
5.157
5.210
5.214
5.695
5.719
5.729
5.741
5.753
5.775
5.787
5.798
5.810
6.075
6.083
6.089
6.539
6.543
6.545
6.550
7.260
7.328
7.338
7.341
7.356
7.370
7.376
7.384
7.393
7.402
7.411
7.418
7.420
Ph
F
2k
xc-6-119-1
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Feb 22 09:00:21 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5.24
1.00
1.00
1.03
2.05
2.18
PPM
0
1
2
3
4
5
6
7
8
9

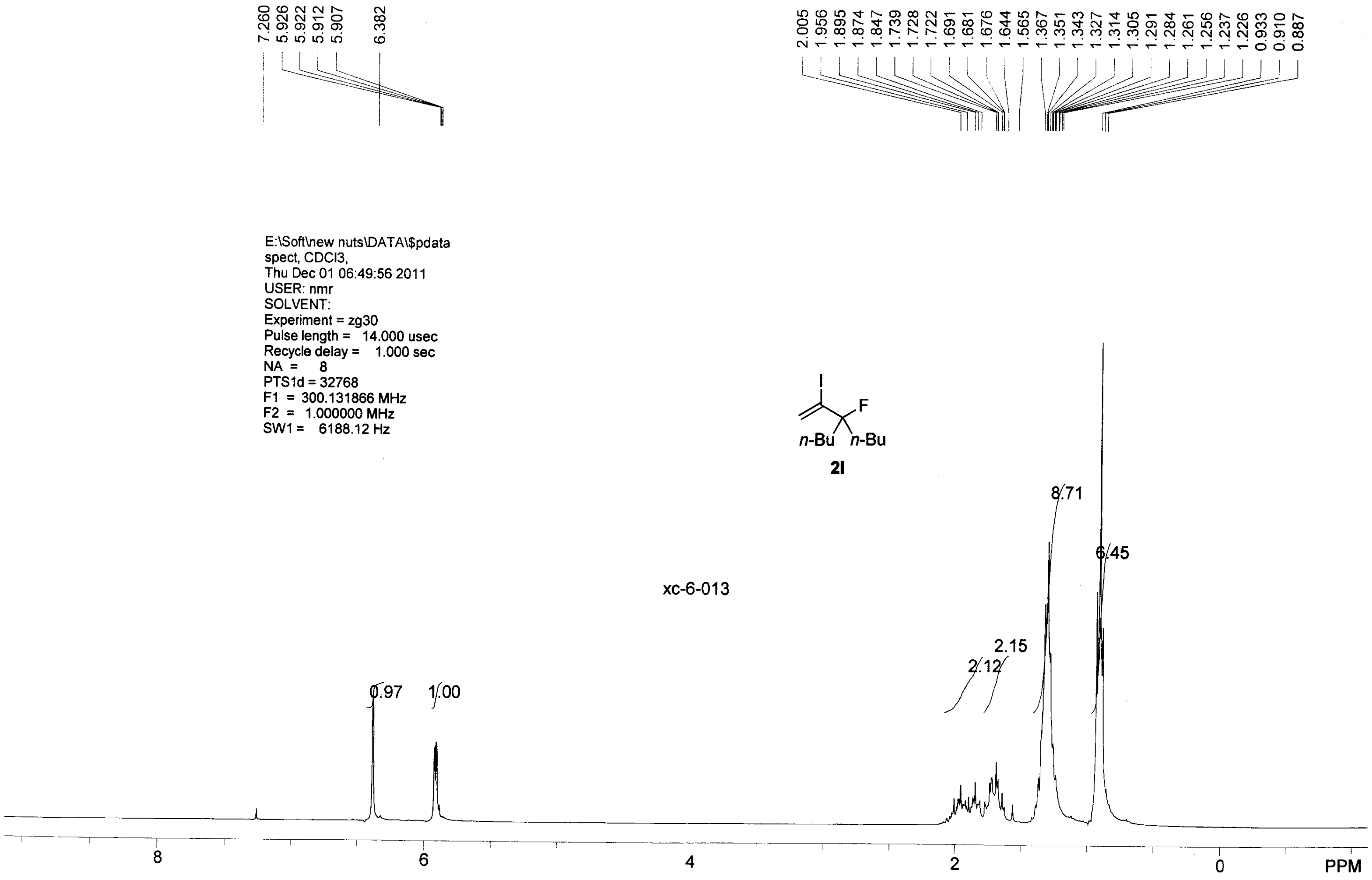


2k

D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Feb 22 09:40:35 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 4277
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

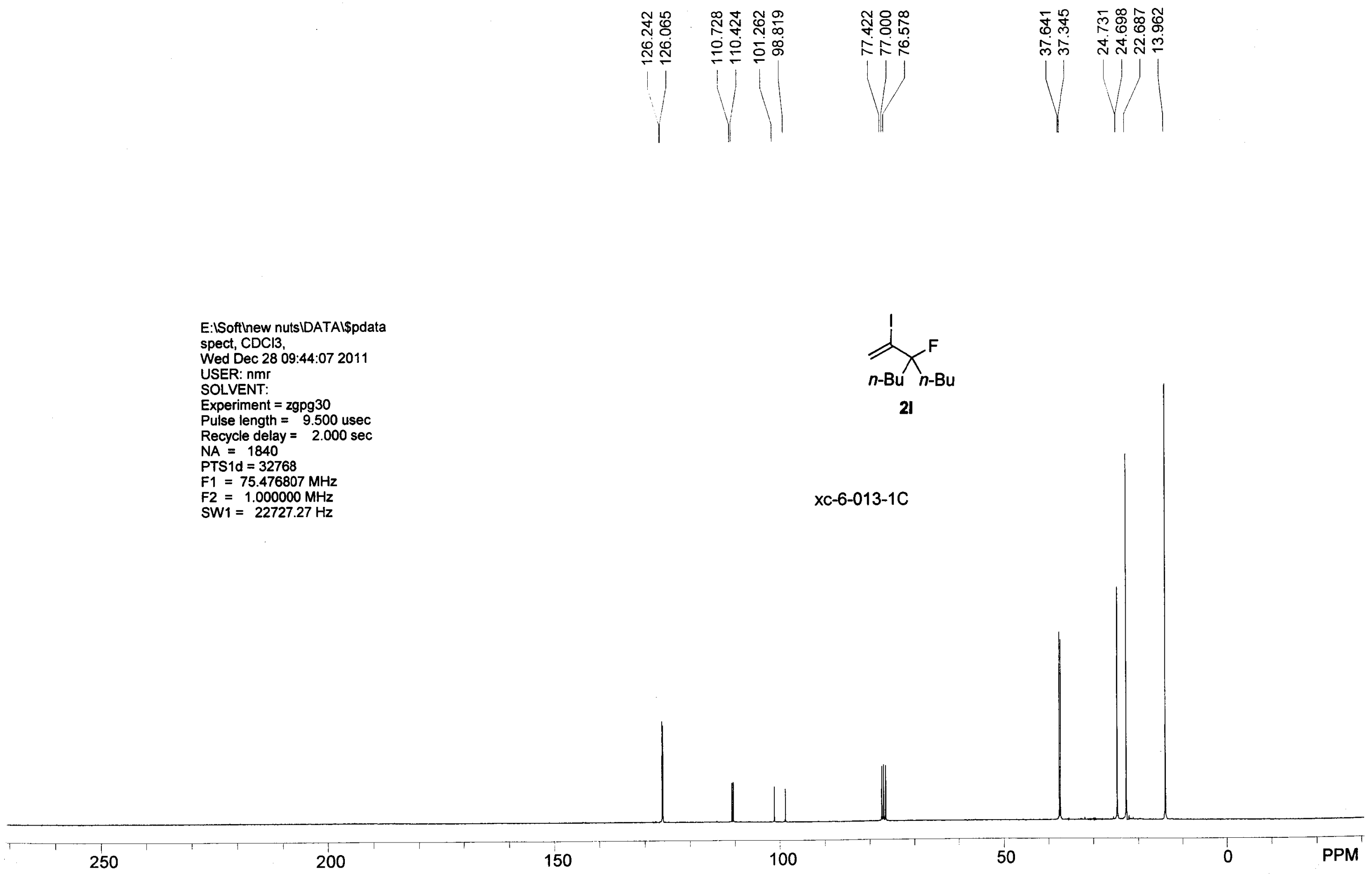

-141.504
-141.421
-141.338
-0.000
0.007
1.00
2k
Ph
F
xc-6-119-1 F
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Feb 22 09:01:43 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
PPM
-200
-150
-100
-50
0


-197.930
-197.919
-197.909
-197.761
-197.749
-197.738
-197.579
-197.568
-158.666
-158.655
-158.618
-158.604
-158.554
-158.540
-158.503
-158.487
-158.451
-158.436
-158.388
-158.371
-158.336
-158.323
7.83
92.00
xc-6-013-cp F
-200 PPM
-190
-180
-170
-160
-150

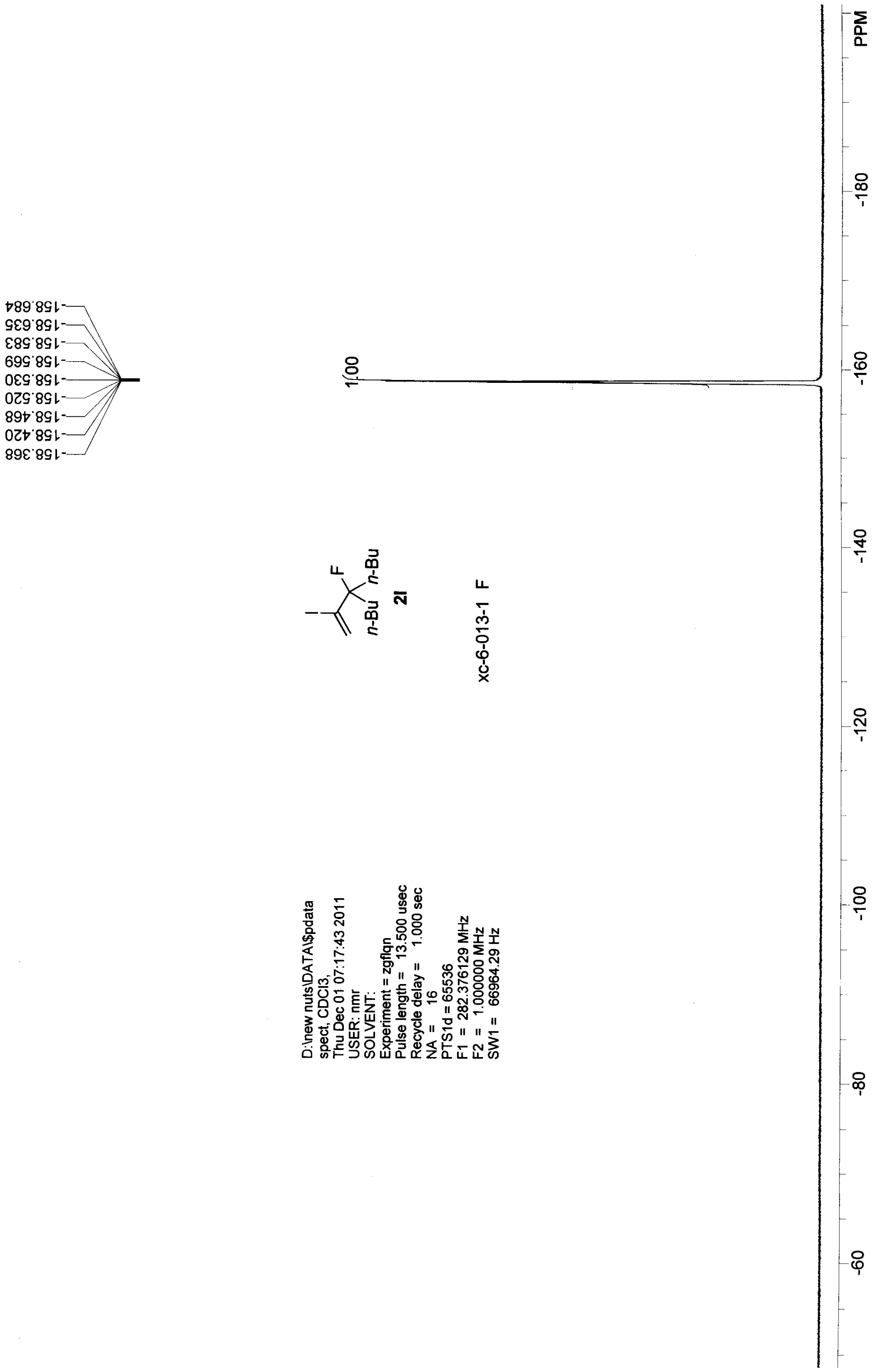


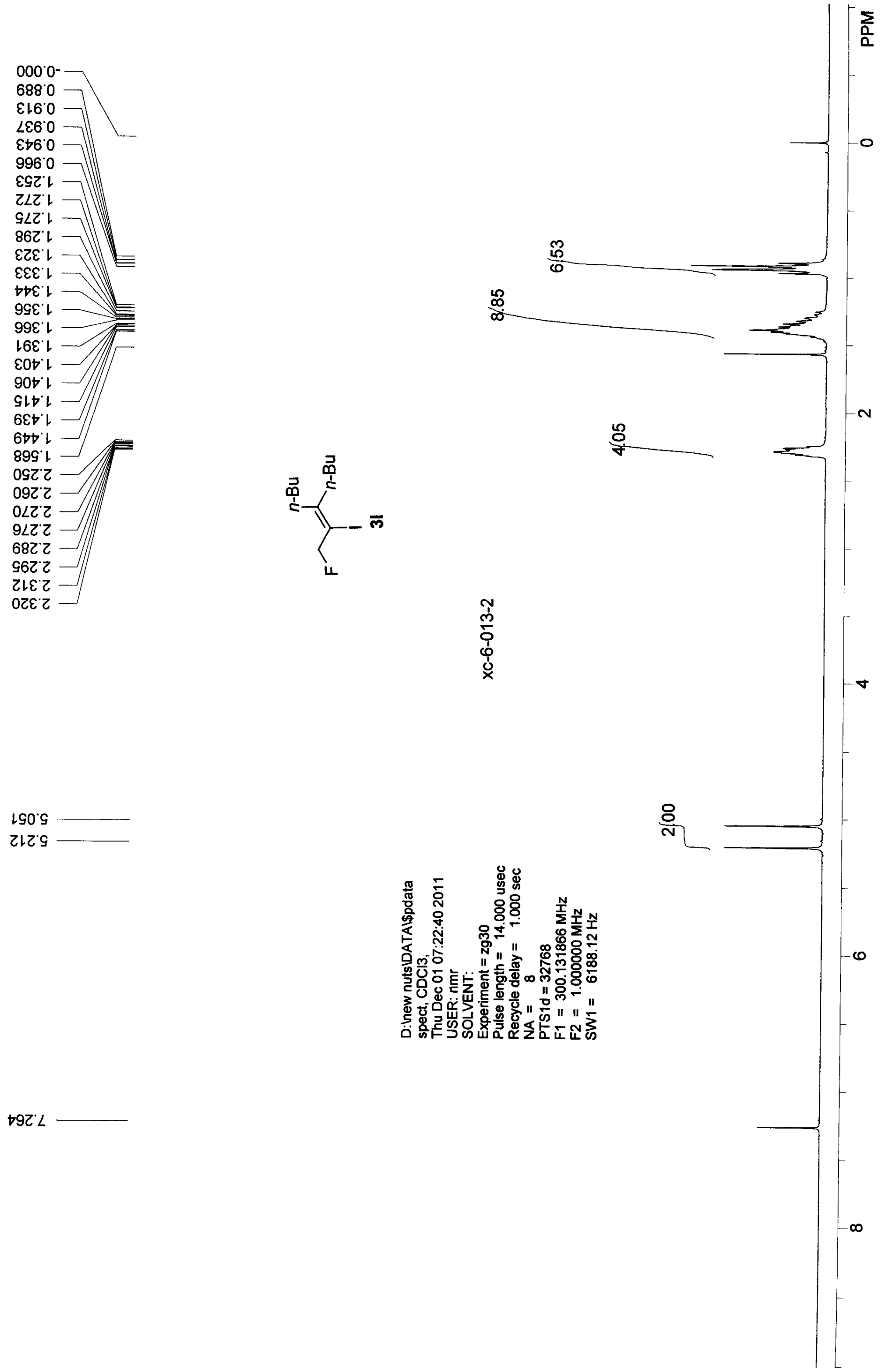
```
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 01 06:49:56 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length =   14.000 usec
Recycle delay =    1.000 sec
NA =     8
PTS1d = 32768
F1  =  300.131866 MHz
F2  =  1.000000 MHz
SW1 =    6188.12 Hz
```

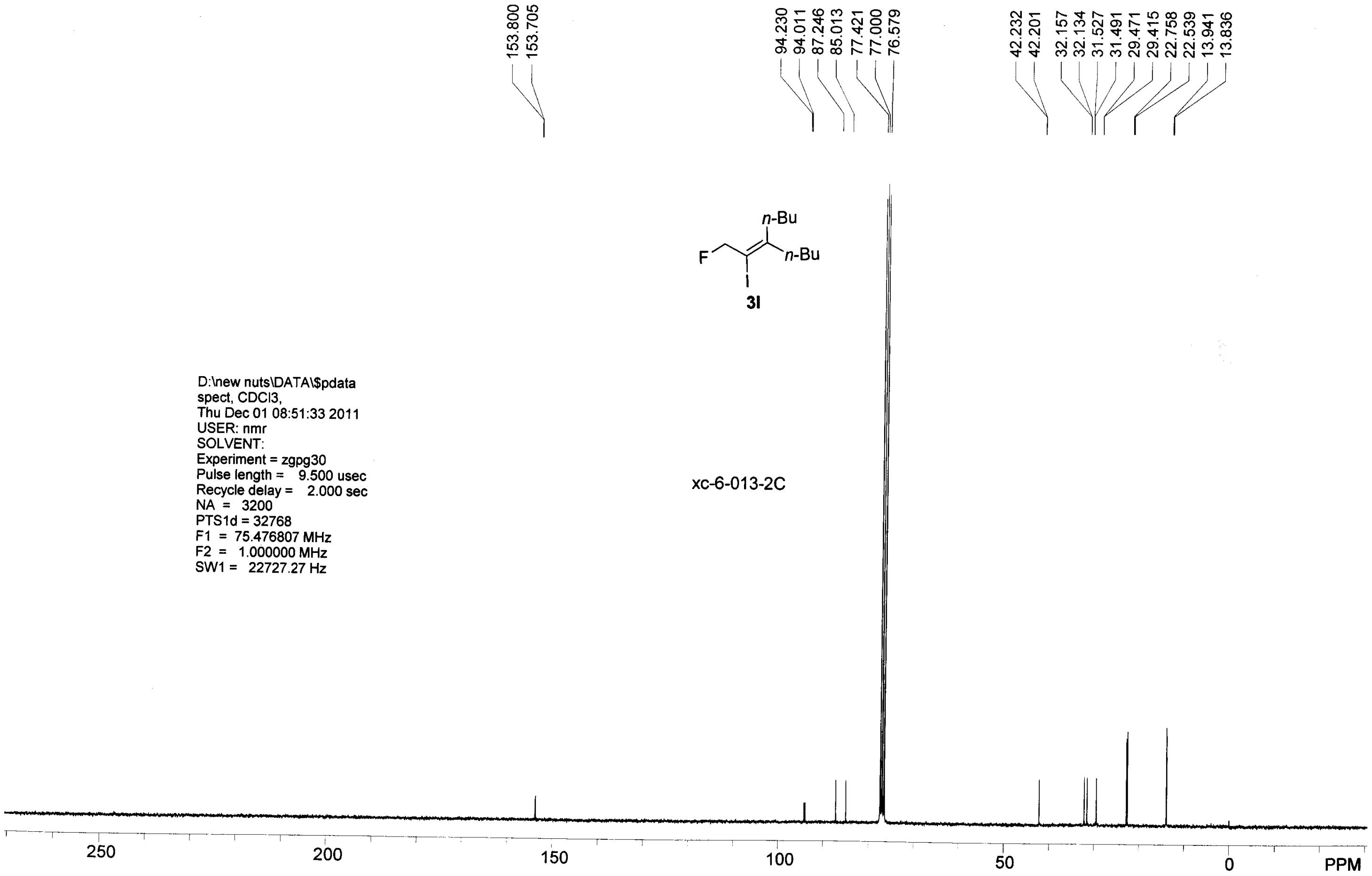
2l



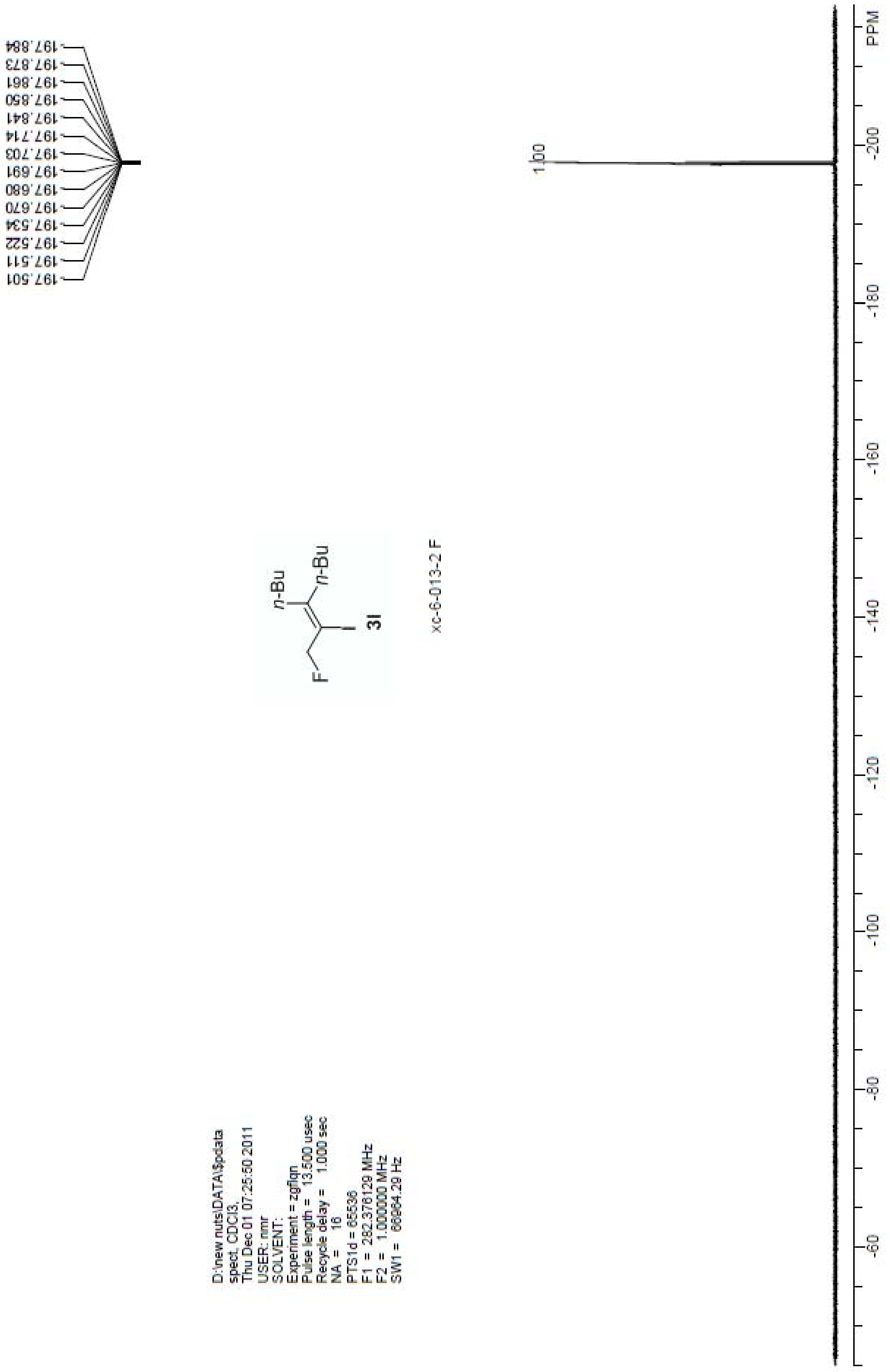
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Dec 28 09:44:07 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1840
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

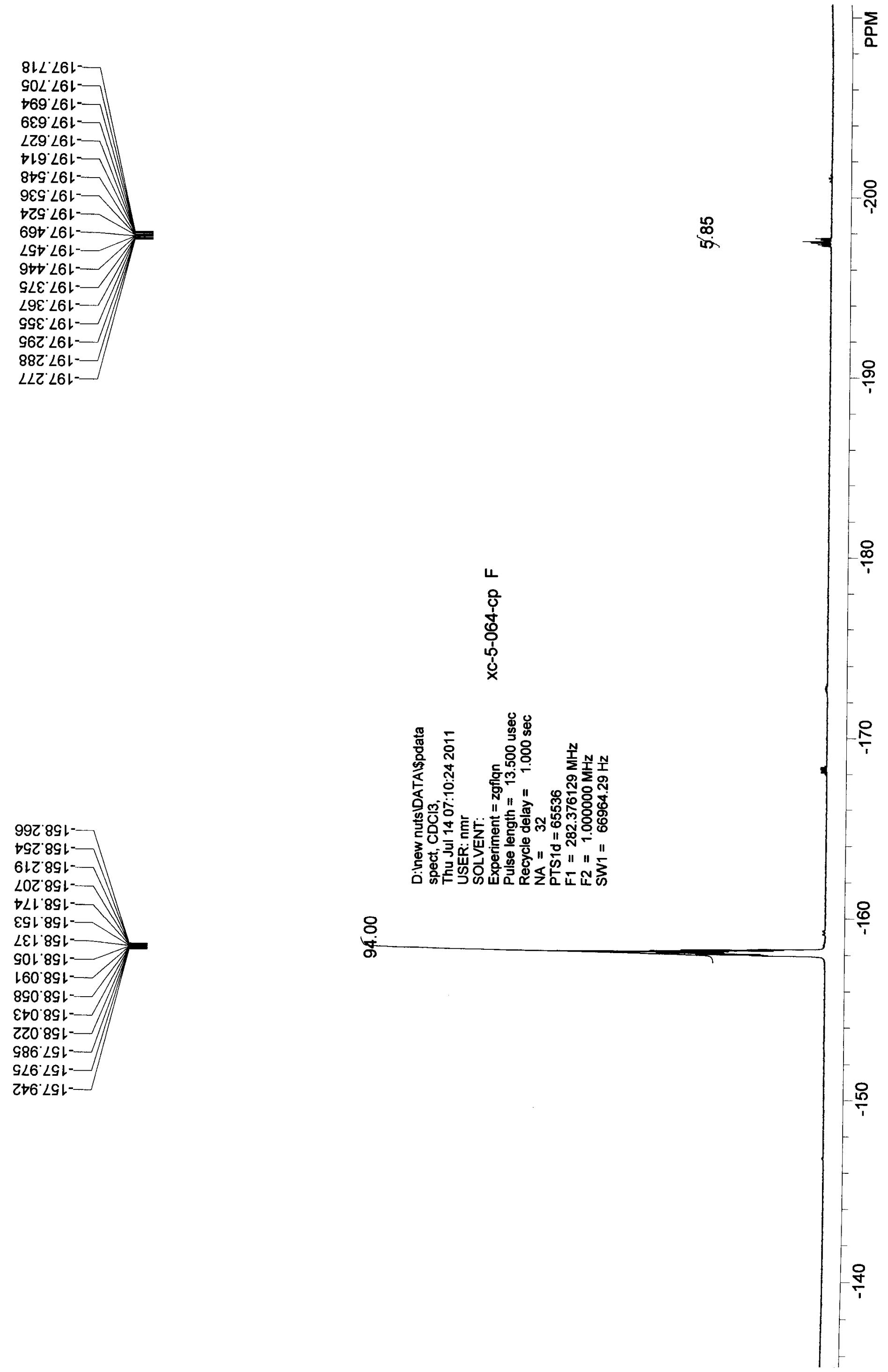

-158.684
-158.635
-158.583
-158.569
-158.530
-158.520
-158.468
-158.420
-158.368
1.00
F
n-Bu
n-Bu
2l
xc-6-013-1 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 01 07:17:43 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-180
-160
-140
-120
-100
-80
-60


-0.000
0.889
0.913
0.937
0.943
0.966
1.253
1.272
1.275
1.298
1.323
1.333
1.344
1.356
1.366
1.391
1.403
1.406
1.415
1.439
1.449
1.568
2.250
2.260
2.270
2.276
2.289
2.295
2.312
2.320
5.051
5.212
7.264
n-Bu
n-Bu
F
I
3l
xc-6-013-2
6.53
8.85
4.05
2.00
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 01 07:22:40 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
PPM
0
2
4
6
8



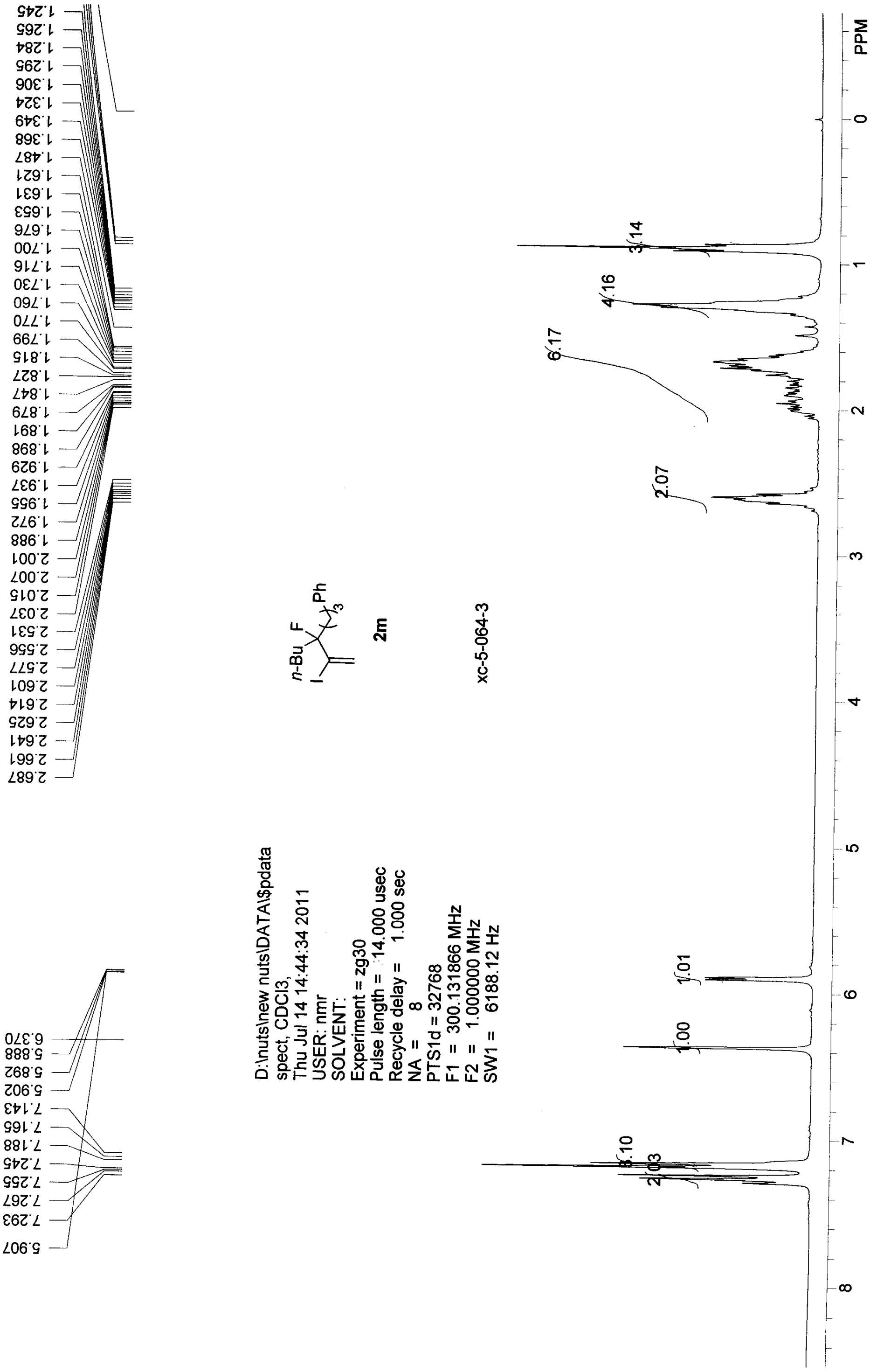
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 01 08:51:33 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 3200
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz


197.884
197.873
197.861
197.850
197.841
197.714
197.703
197.691
197.680
197.670
197.534
197.522
197.511
197.501
1.00
n-Bu
n-Bu
F
3l
xc-6-013-2 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 01 07:25:50 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-200
-180
-160
-140
-120
-100
-80
-60



xc-5-064-cp F

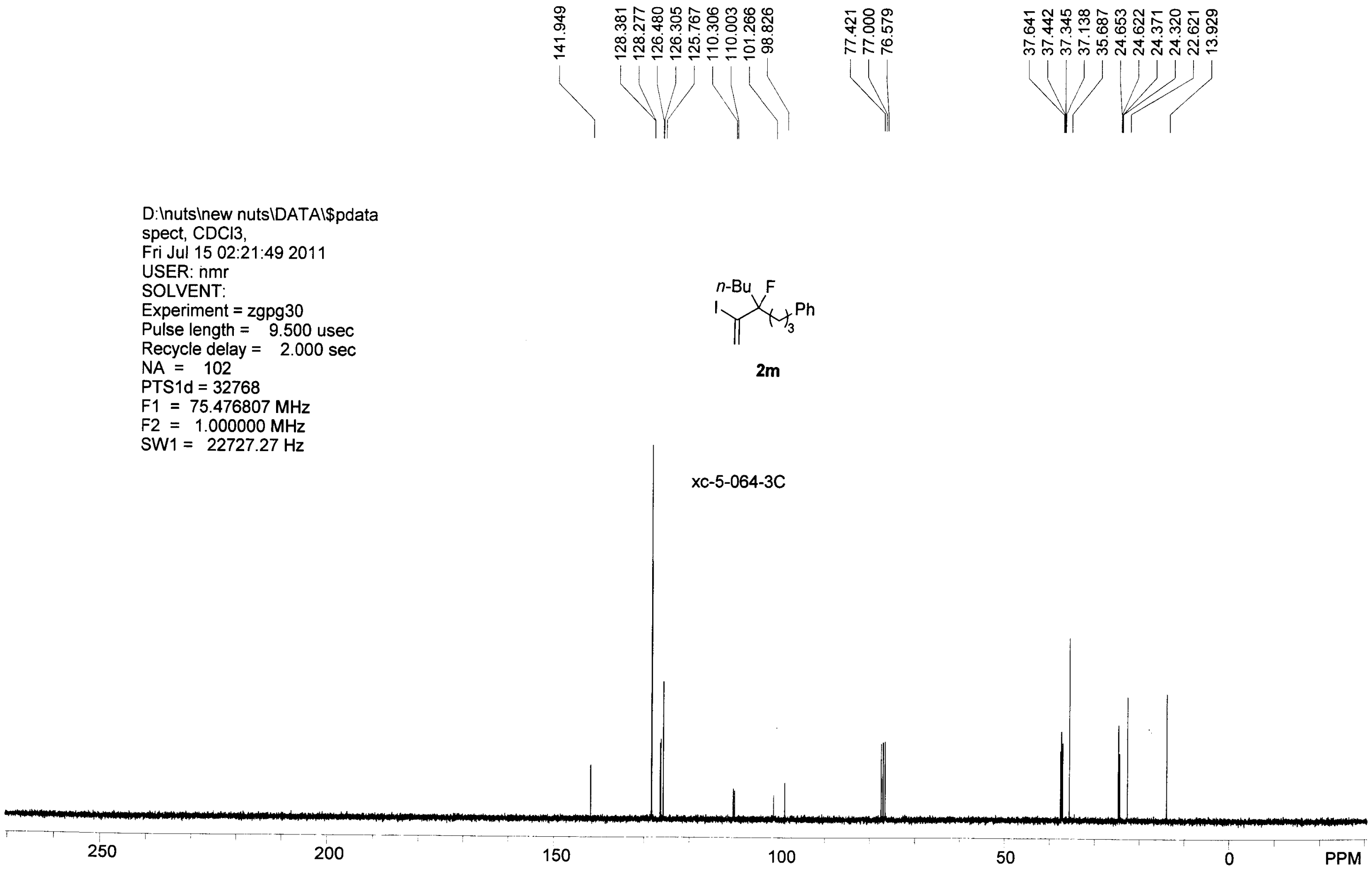
D:\new nuts\DATA\$pdata
spect, $CDCl_3$,
Thu Jul 14 07:10:24 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz



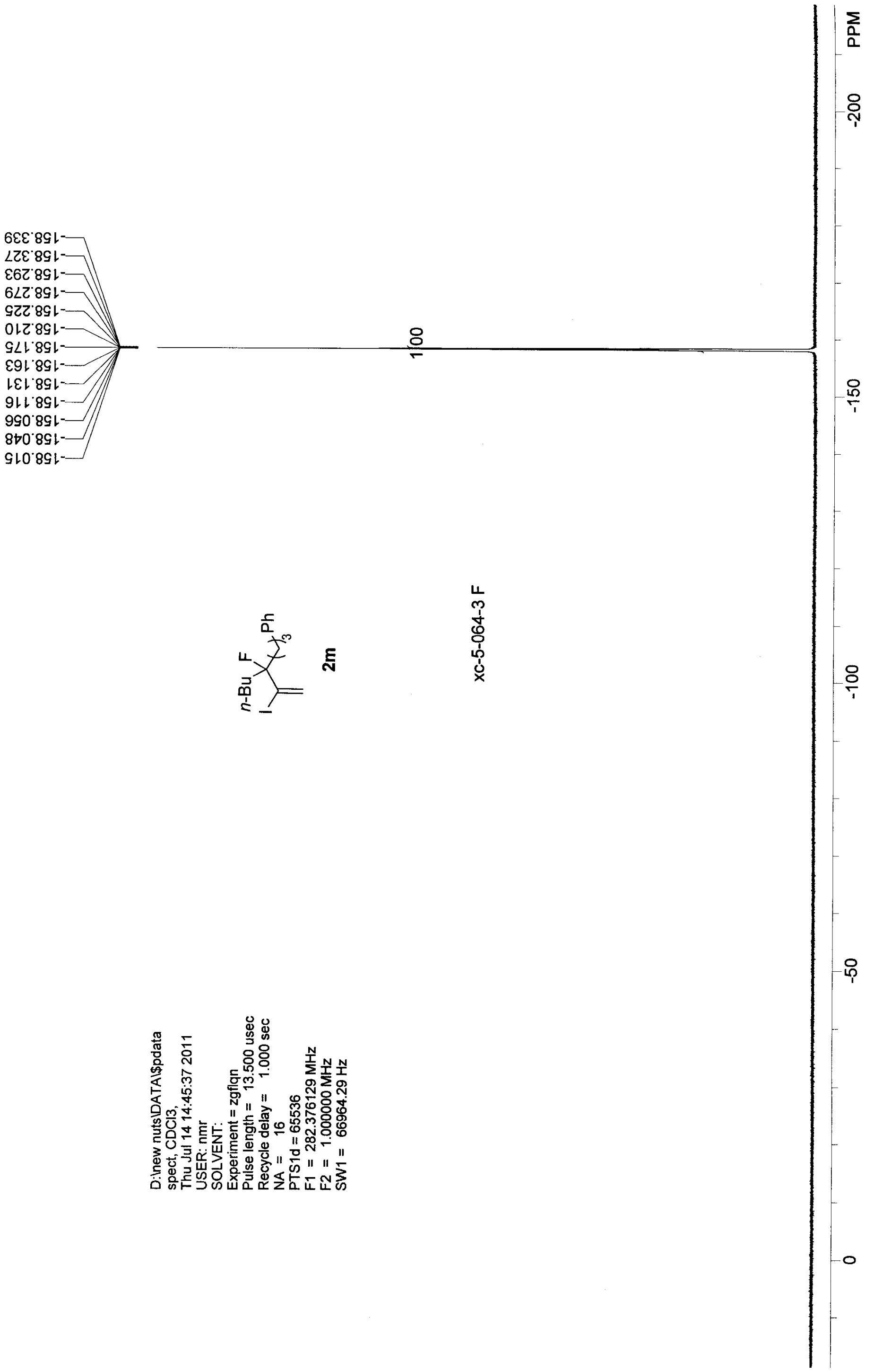
2m

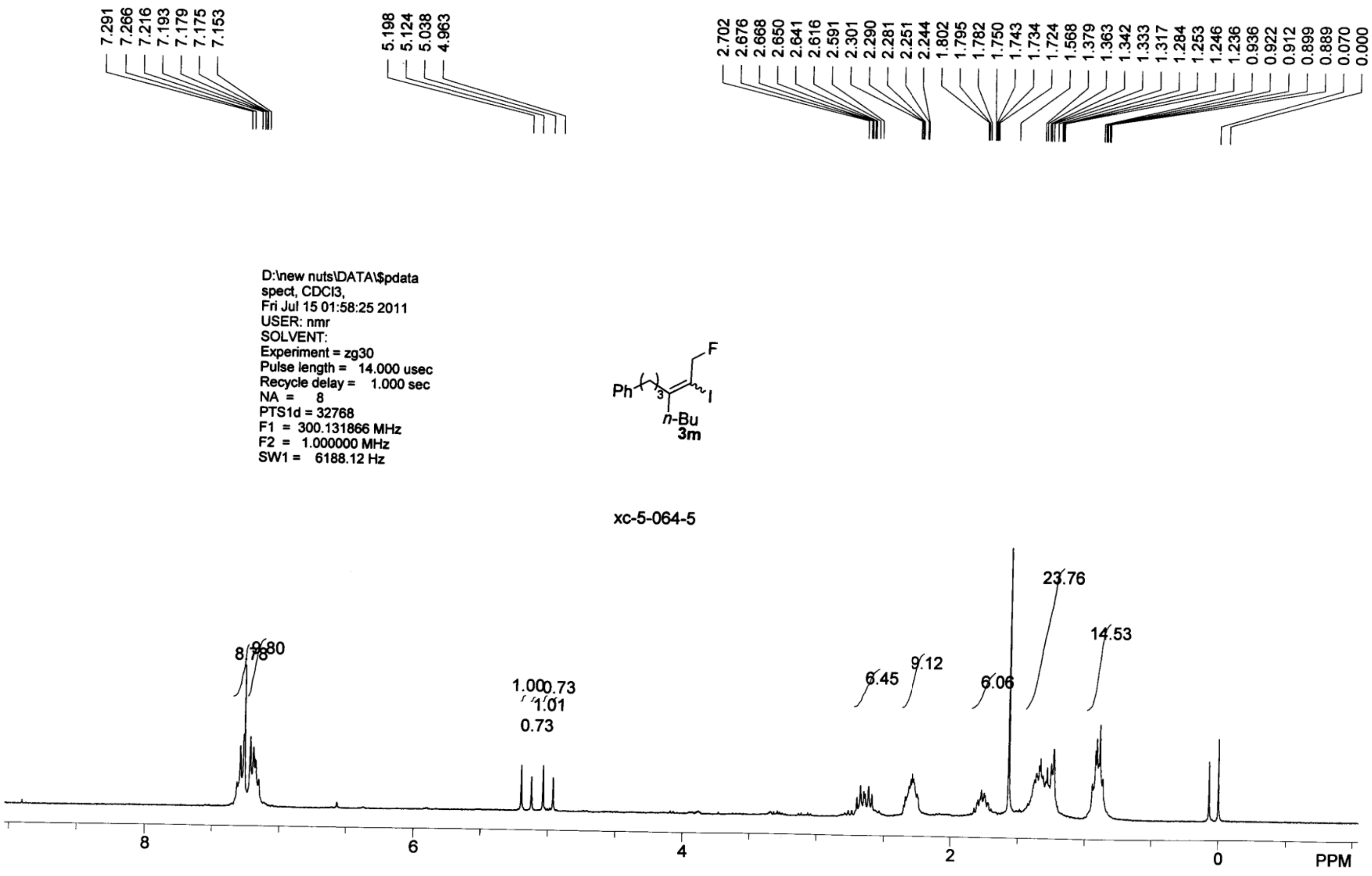
xc-5-064-3

D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Thu Jul 14 14:44:34 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

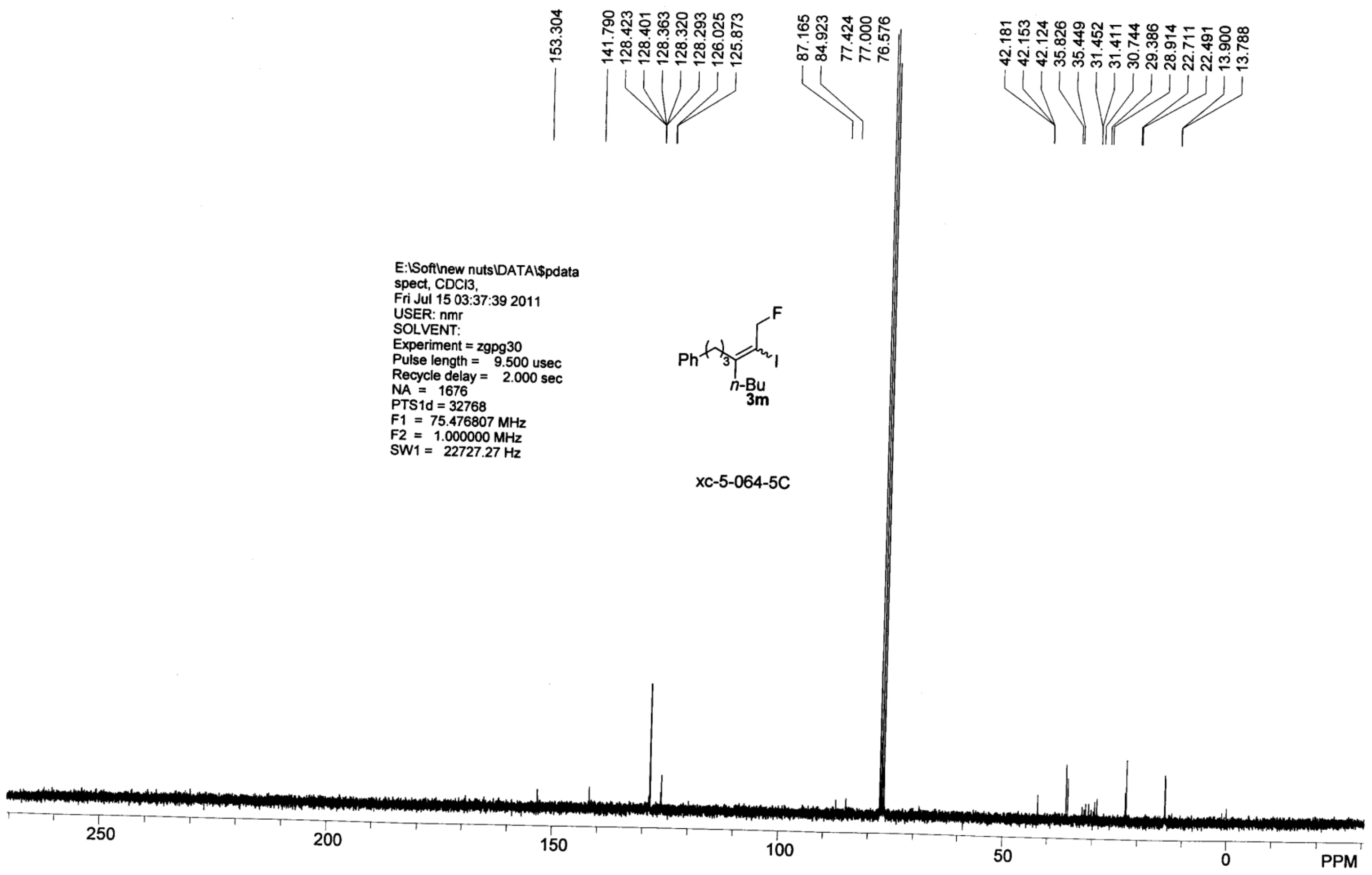


```
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 15 02:21:49 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length =    9.500 usec
Recycle delay =    2.000 sec
NA =    102
PTS1d = 32768
F1  =  75.476807 MHz
F2  =   1.000000 MHz
SW1 =   22727.27 Hz
```


-158.339
-158.327
-158.293
-158.279
-158.225
-158.210
-158.175
-158.163
-158.131
-158.116
-158.056
-158.048
-158.015
1.00
n-Bu
F
I
Ph
3
2m
xc-5-064-3 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Jul 14 14:45:37 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
-200
-150
-100
-50
0
PPM



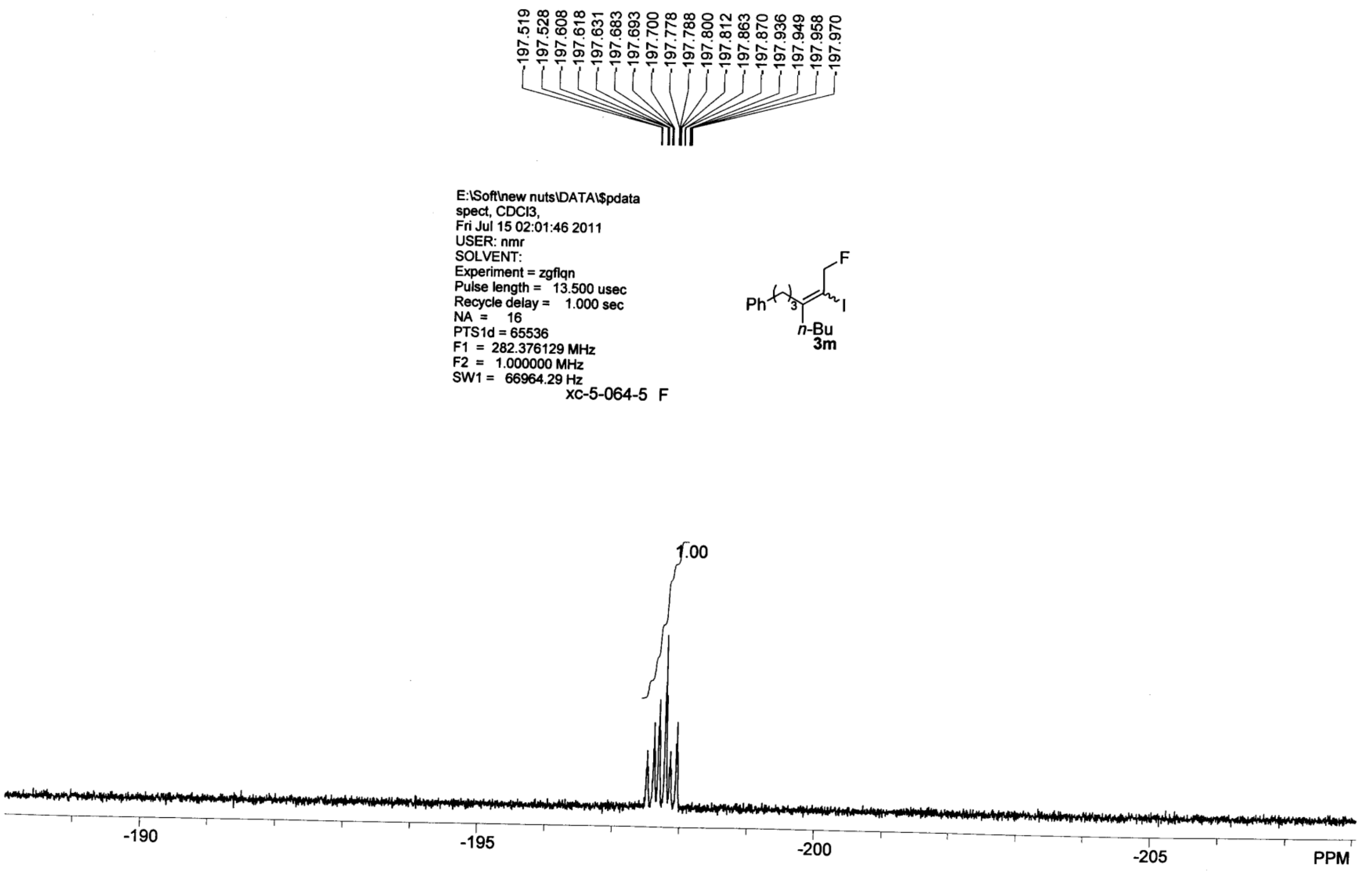
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 15 01:58:25 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



3m

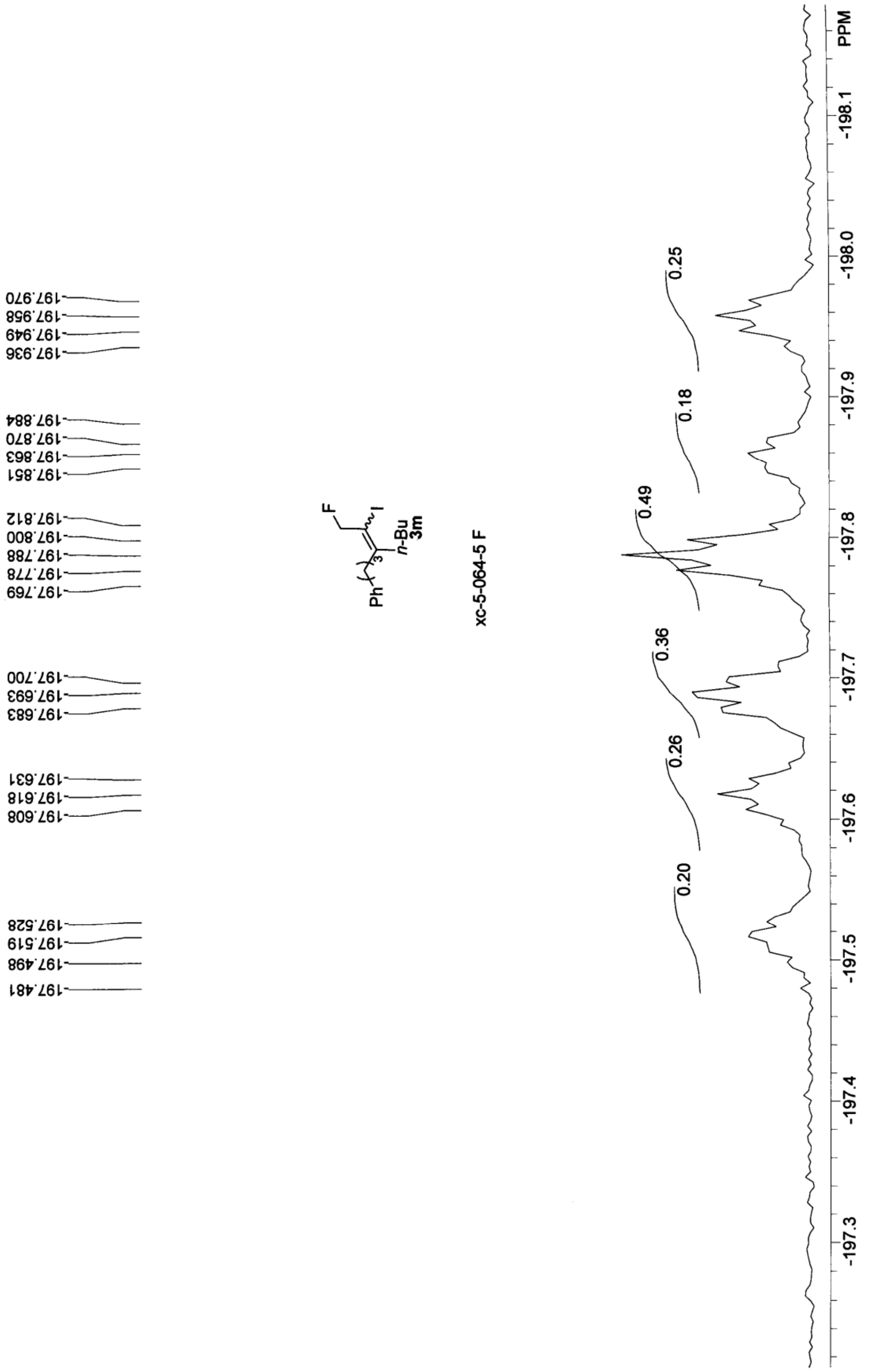
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 15 03:37:39 2011
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1676
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

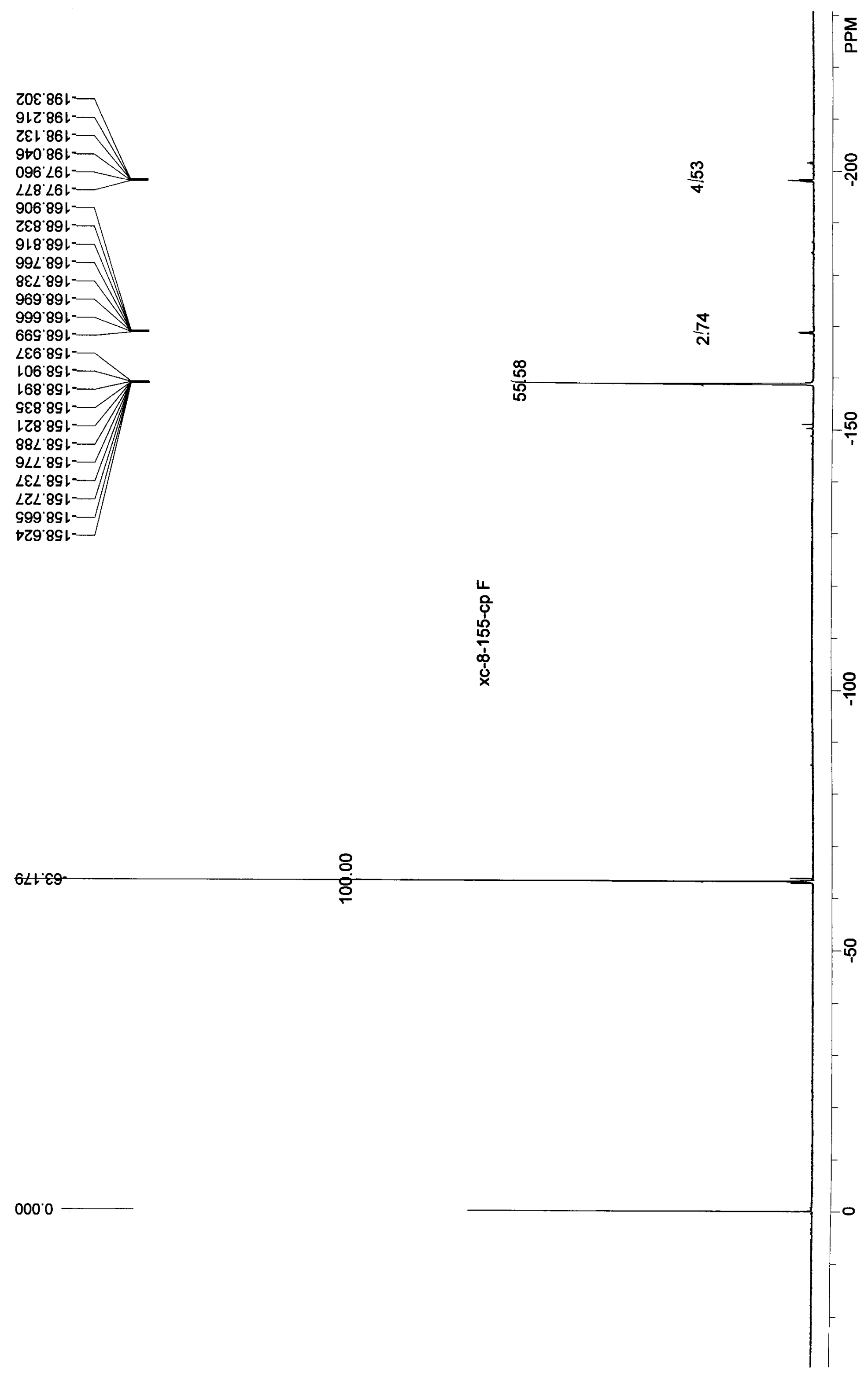
xc-5-064-5C

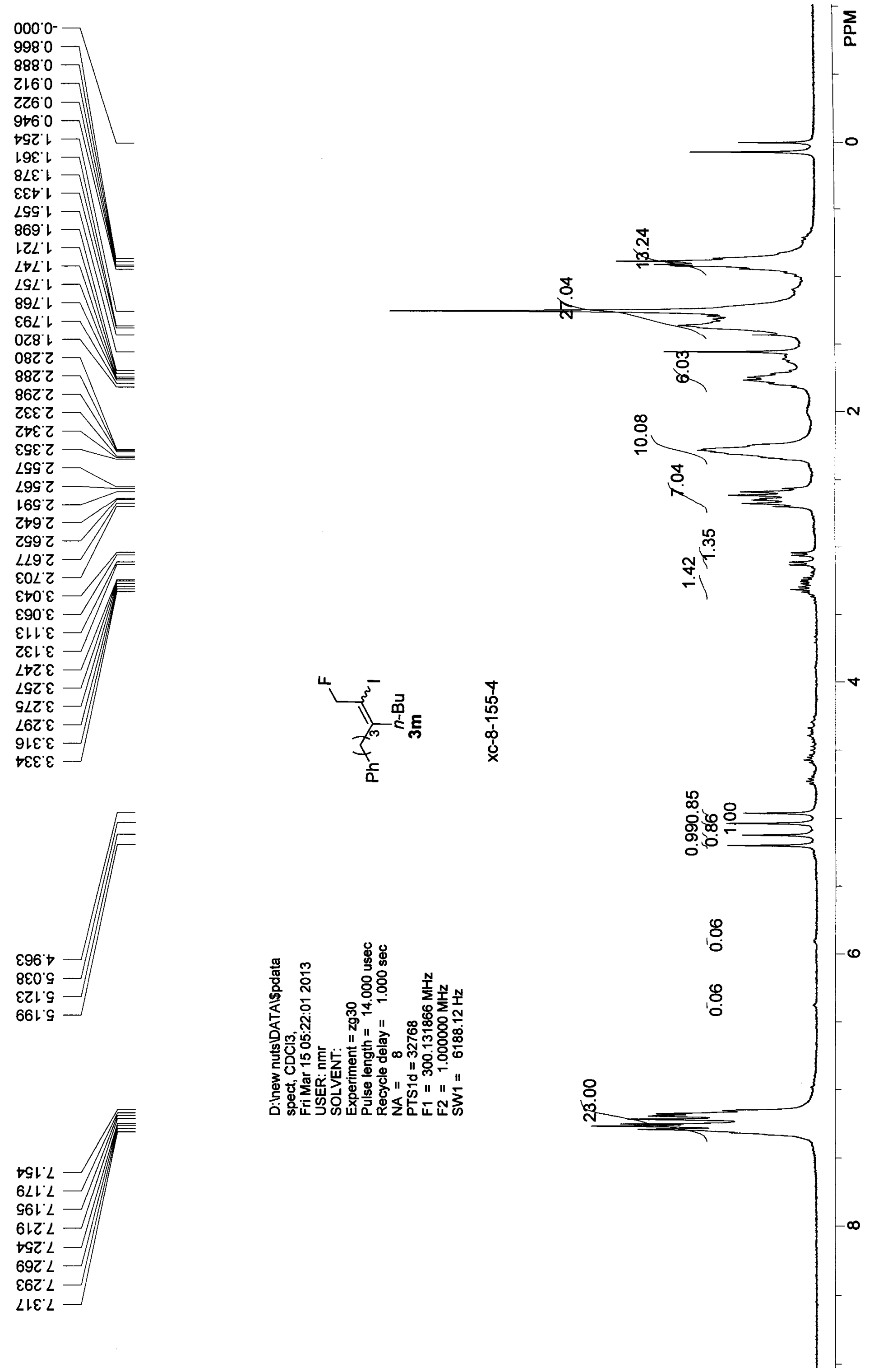


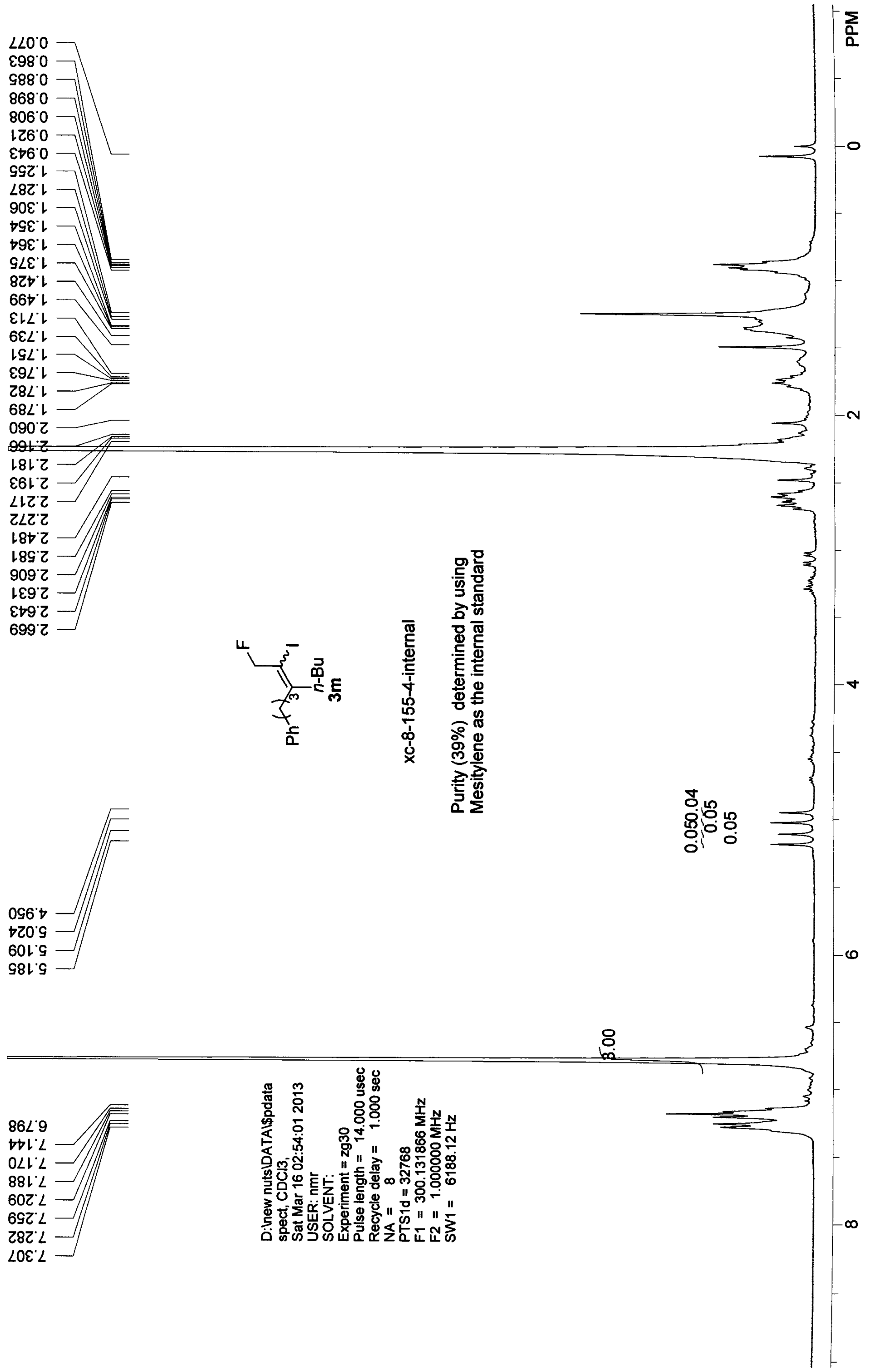
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 15 02:01:46 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
xc-5-064-5 F

3m


-197.970
-197.958
-197.949
-197.936
-197.884
-197.870
-197.863
-197.851
-197.812
-197.800
-197.788
-197.778
-197.769
-197.700
-197.693
-197.683
-197.631
-197.618
-197.608
-197.528
-197.519
-197.498
-197.481
F
I
Ph
3
n-Bu
3m
xc-5-064-5 F
0.25
0.18
0.49
0.36
0.26
0.20
PPM
-198.1
-198.0
-197.9
-197.8
-197.7
-197.6
-197.5
-197.4
-197.3


xc-8-155-cp F
-198.302
-198.216
-198.132
-198.046
-197.960
-197.877
-168.906
-168.832
-168.816
-168.766
-168.738
-168.696
-168.666
-168.599
-158.937
-158.901
-158.891
-158.835
-158.821
-158.788
-158.776
-158.737
-158.727
-158.665
-158.624
-63.179
0.000
4.53
2.74
55.58
100.00
PPM
-200
-150
-100
-50
0


-0.000
0.866
0.888
0.912
0.922
0.946
1.254
1.361
1.378
1.433
1.557
1.698
1.721
1.747
1.757
1.768
1.793
1.820
2.280
2.288
2.298
2.332
2.342
2.353
2.557
2.567
2.591
2.642
2.652
2.677
2.703
3.043
3.063
3.113
3.132
3.247
3.257
3.275
3.297
3.316
3.334
4.963
5.038
5.123
5.199
7.154
7.179
7.195
7.219
7.254
7.269
7.293
7.317
F
I
Ph
3
n-Bu
3m
xc-8-155-4
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Mar 15 05:22:01 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
13.24
27.04
6.03
10.08
7.04
1.42
1.35
0.99
0.85
0.86
1.00
0.06
0.06
23.00
PPM
0
2
4
6
8

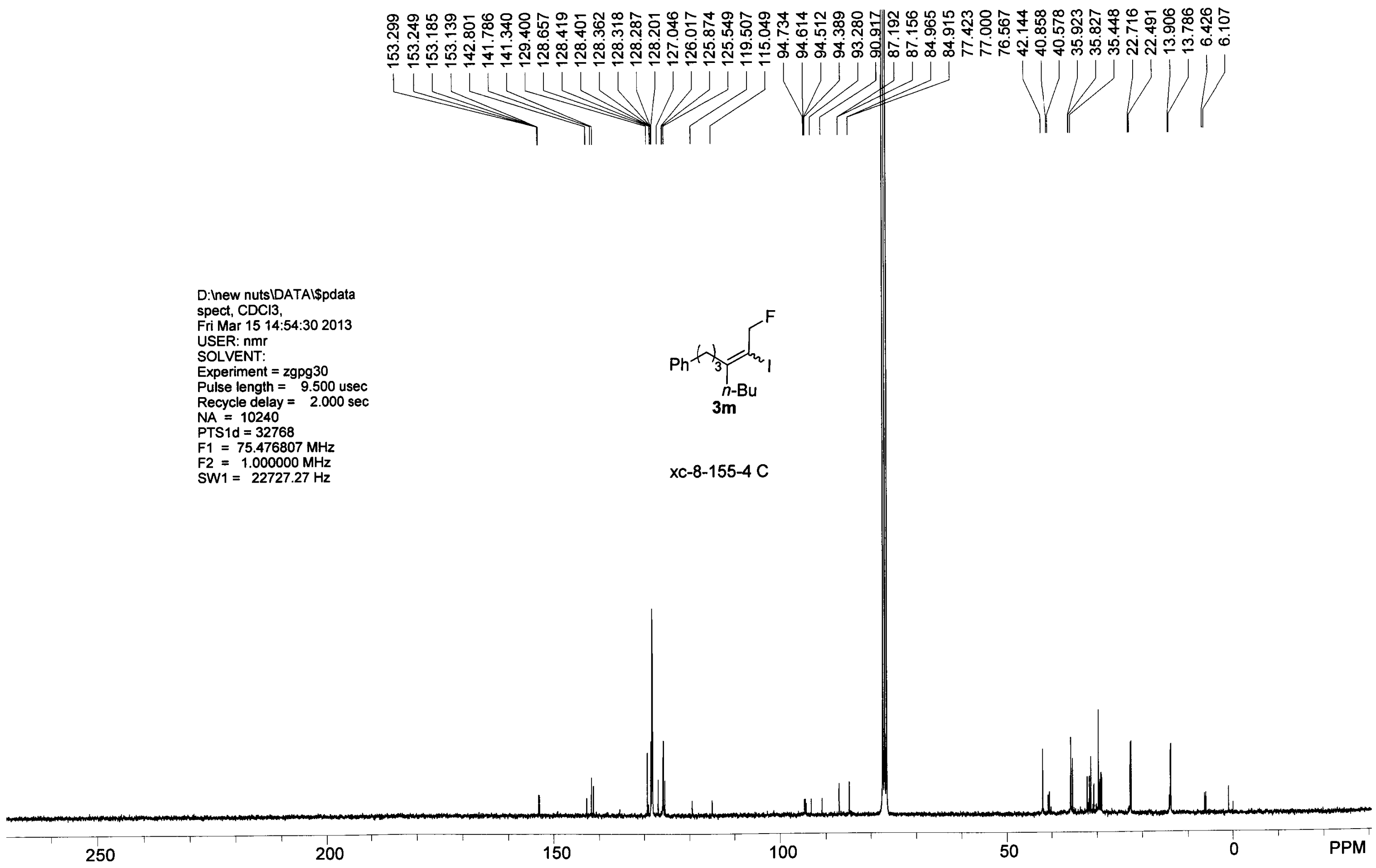


3m

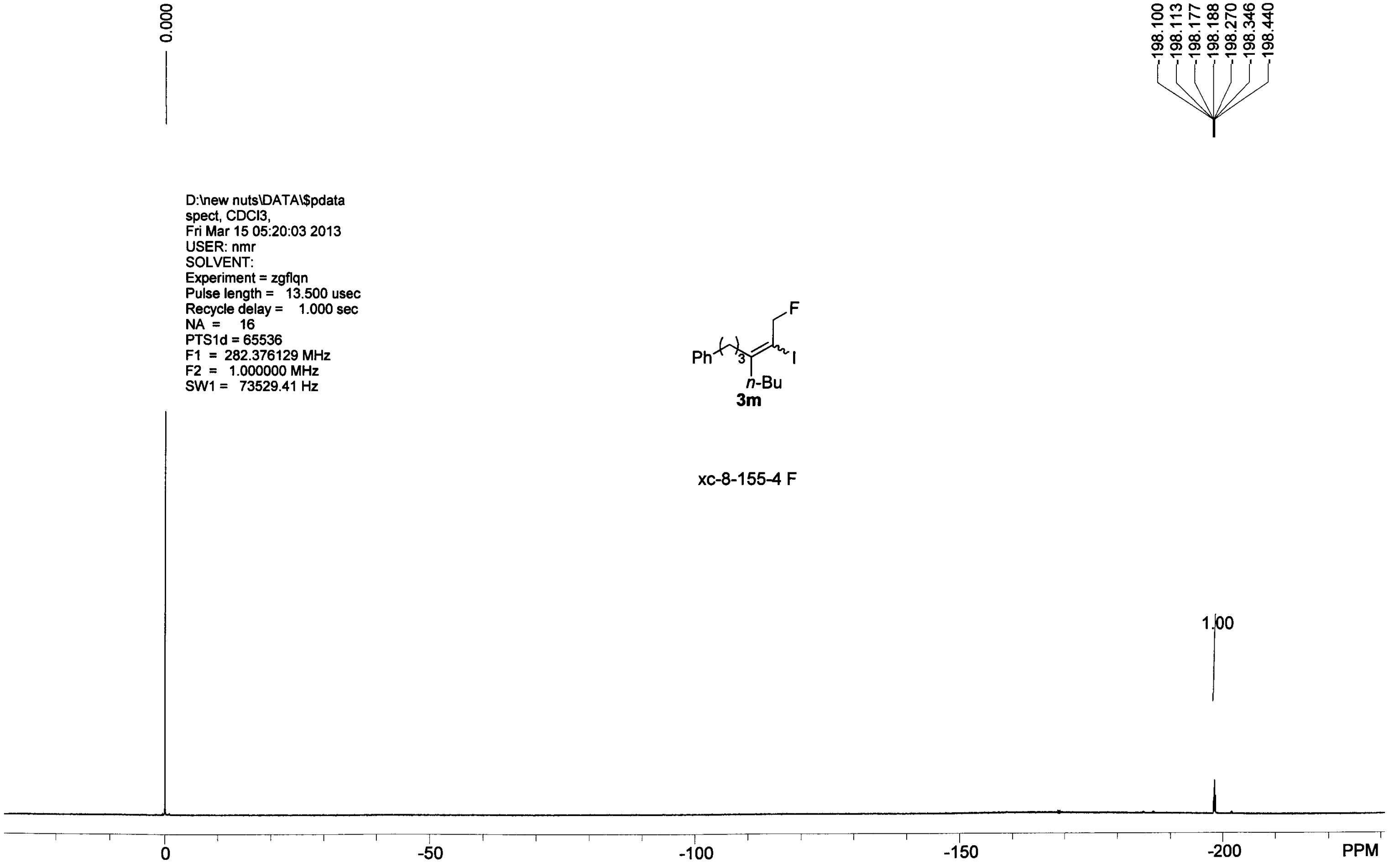
xc-8-155-4-internal

Purity (39%) determined by using
Mesitylene as the internal standard

D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Mar 16 02:54:01 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



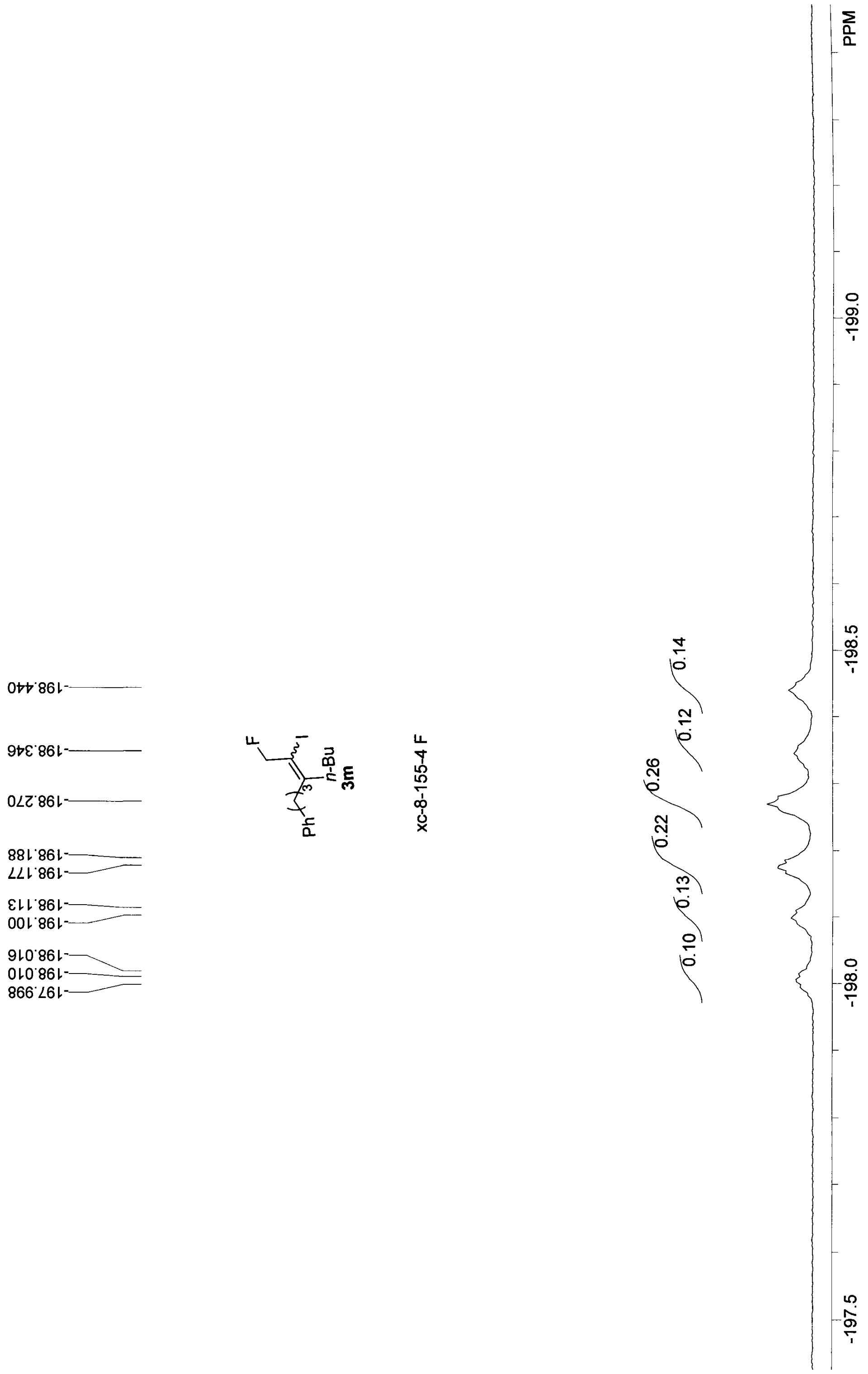
```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Mar 15 14:54:30 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length =    9.500 usec
Recycle delay =    2.000 sec
NA  =  10240
PTS1d = 32768
F1  =  75.476807 MHz
F2  =   1.000000 MHz
SW1 =   22727.27 Hz
```

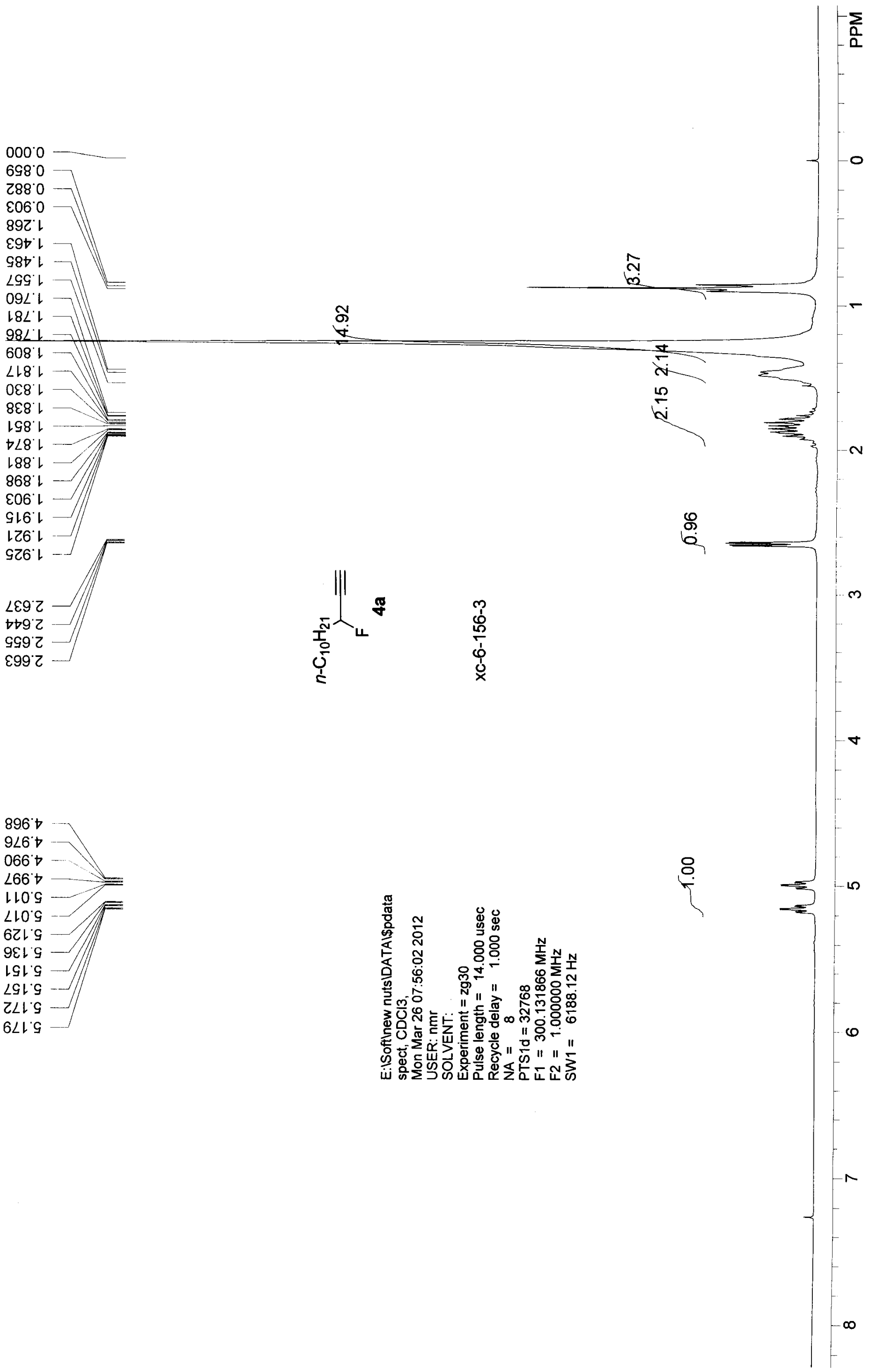


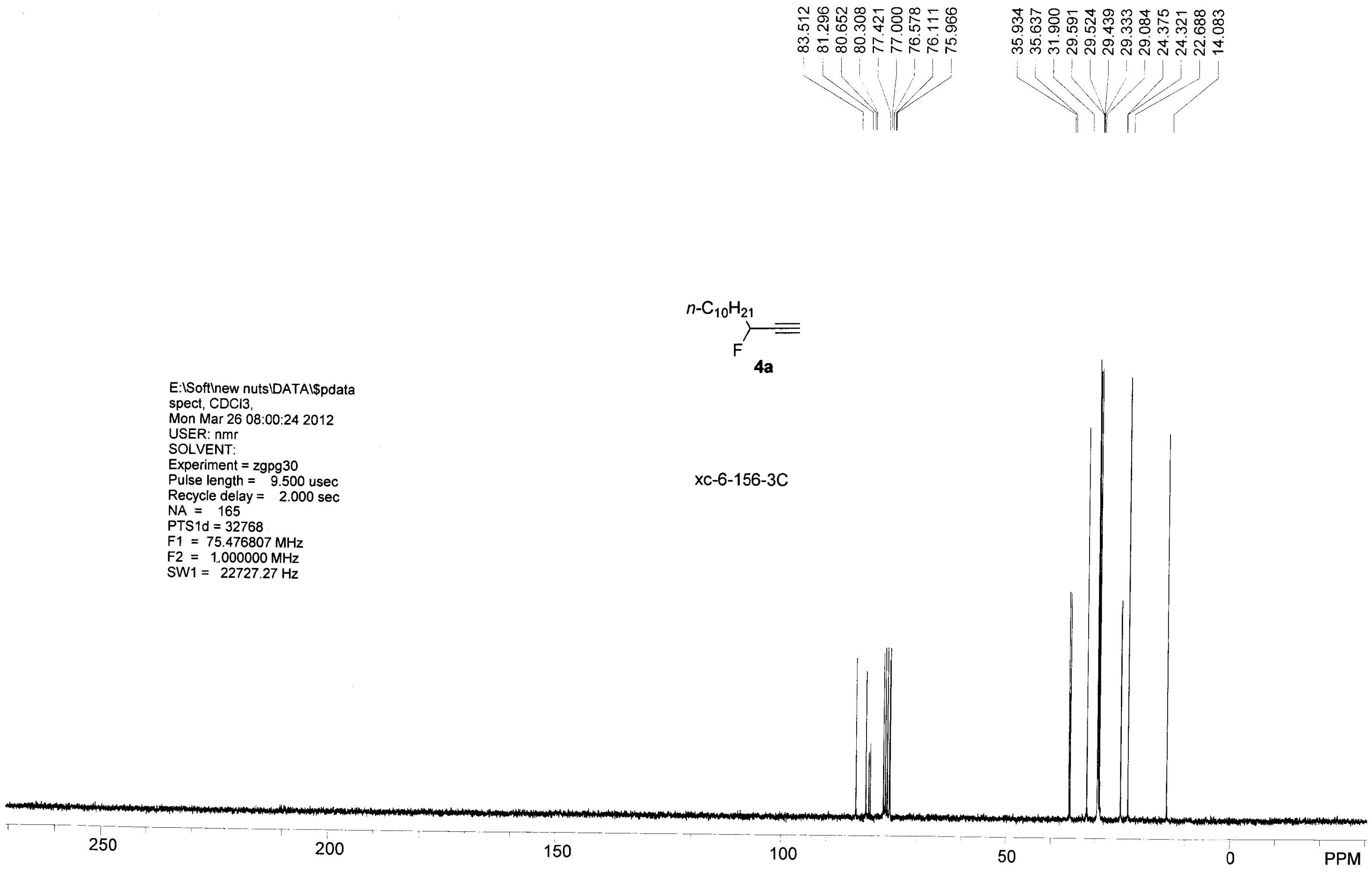
3m

xc-8-155-4 F

```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Mar 15 05:20:03 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =   13.500 usec
Recycle delay =    1.000 sec
NA =     16
PTS1d = 65536
F1  =  282.376129 MHz
F2  =  1.000000 MHz
SW1 =   73529.41 Hz
```


-198.440
-198.346
-198.270
-198.188
-198.177
-198.113
-198.100
-198.016
-198.010
-197.998
F
I
Ph
3
n-Bu
3m
xc-8-155-4 F
0.14
0.12
0.26
0.22
0.13
0.10
PPM
-199.0
-198.5
-198.0
-197.5

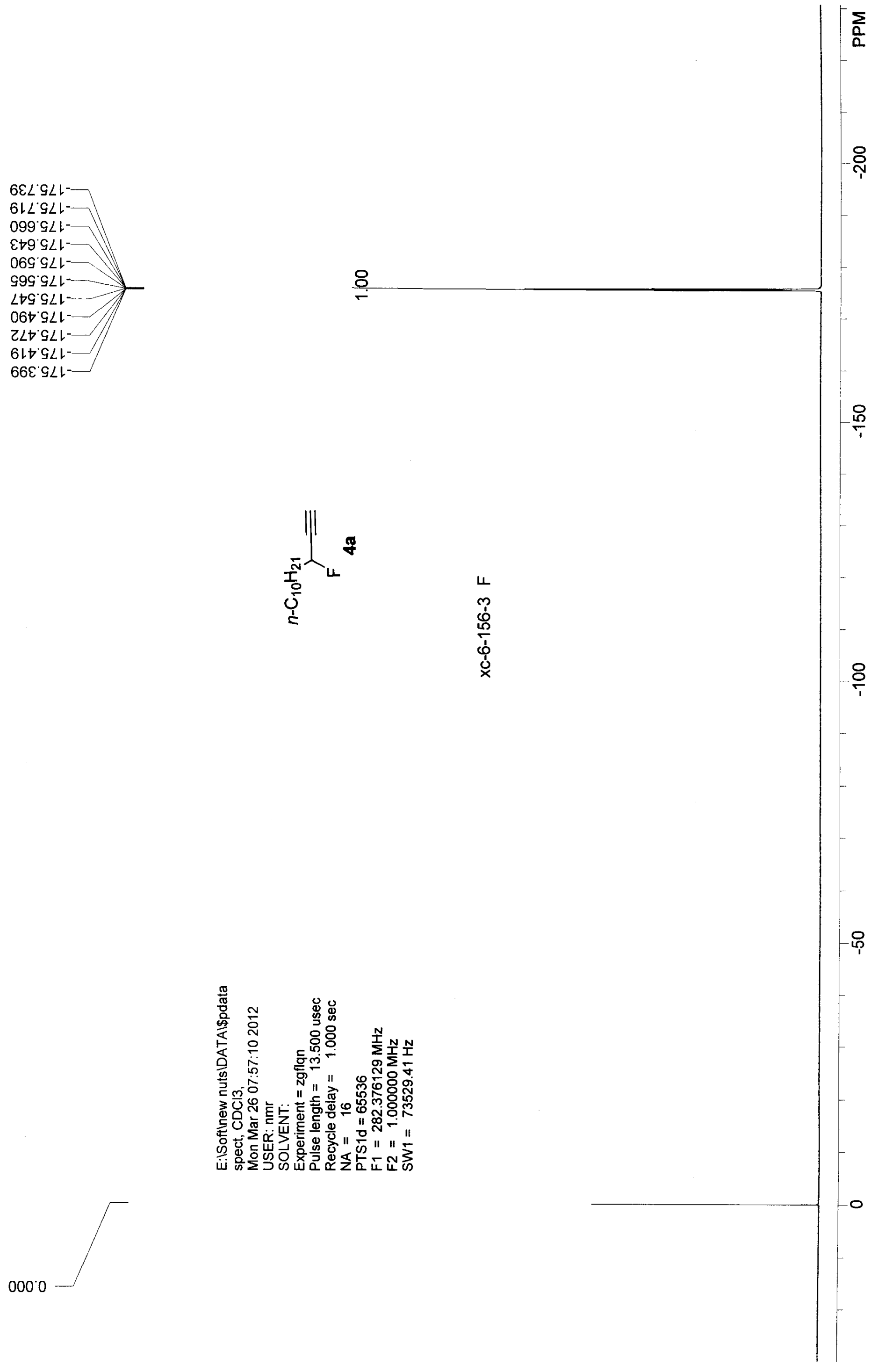

0.000
0.859
0.882
0.903
1.268
1.463
1.485
1.557
1.760
1.781
1.786
1.809
1.817
1.830
1.838
1.851
1.874
1.881
1.898
1.903
1.915
1.921
1.925
2.637
2.644
2.655
2.663
4.968
4.976
4.990
4.997
5.011
5.017
5.129
5.136
5.151
5.157
5.172
5.179
14.92
3.27
2.14
2.15
0.96
1.00
n-C10H21
F
4a
xc-6-156-3
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Mon Mar 26 07:56:02 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
PPM
0
1
2
3
4
5
6
7
8



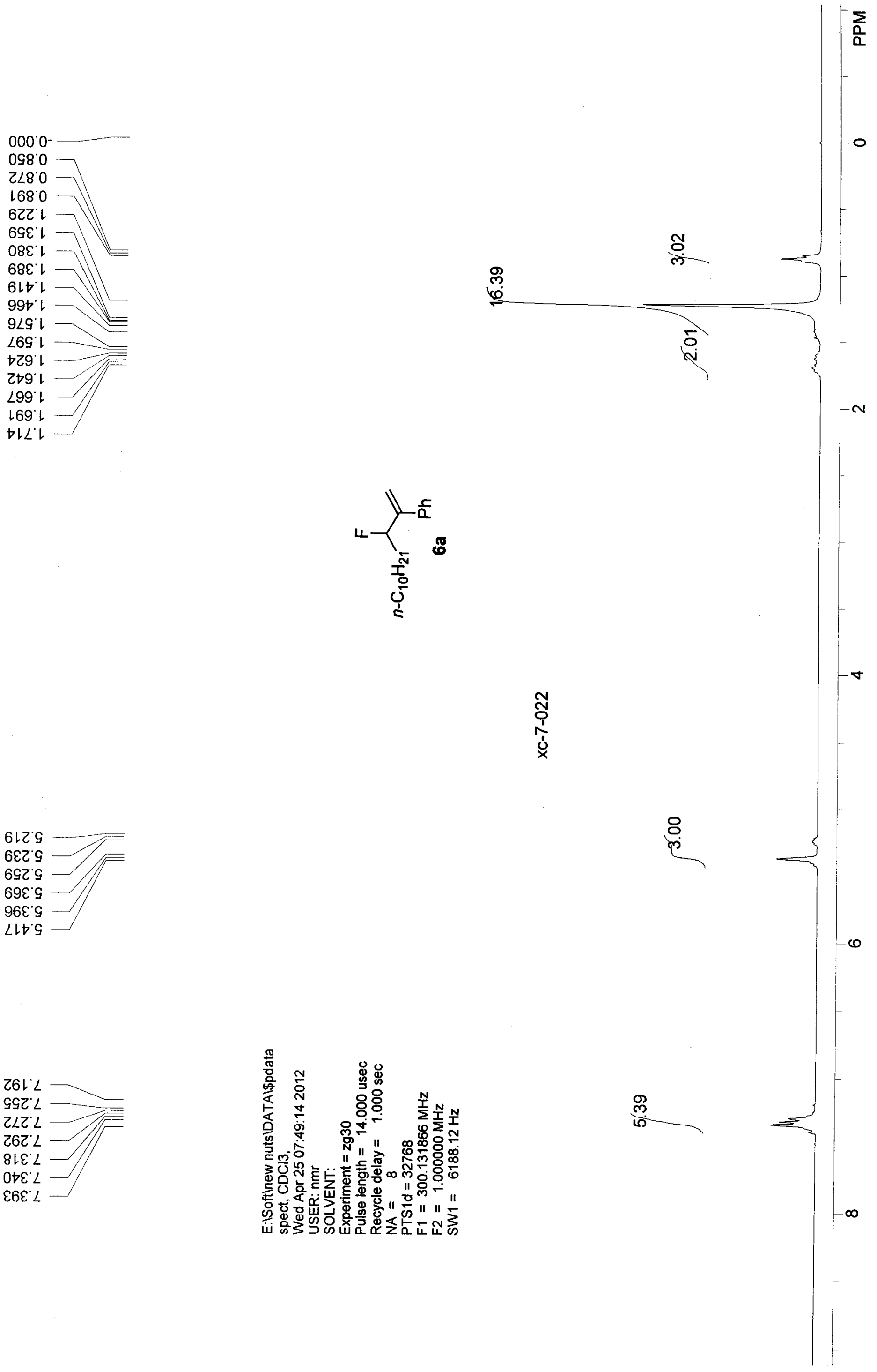
4a

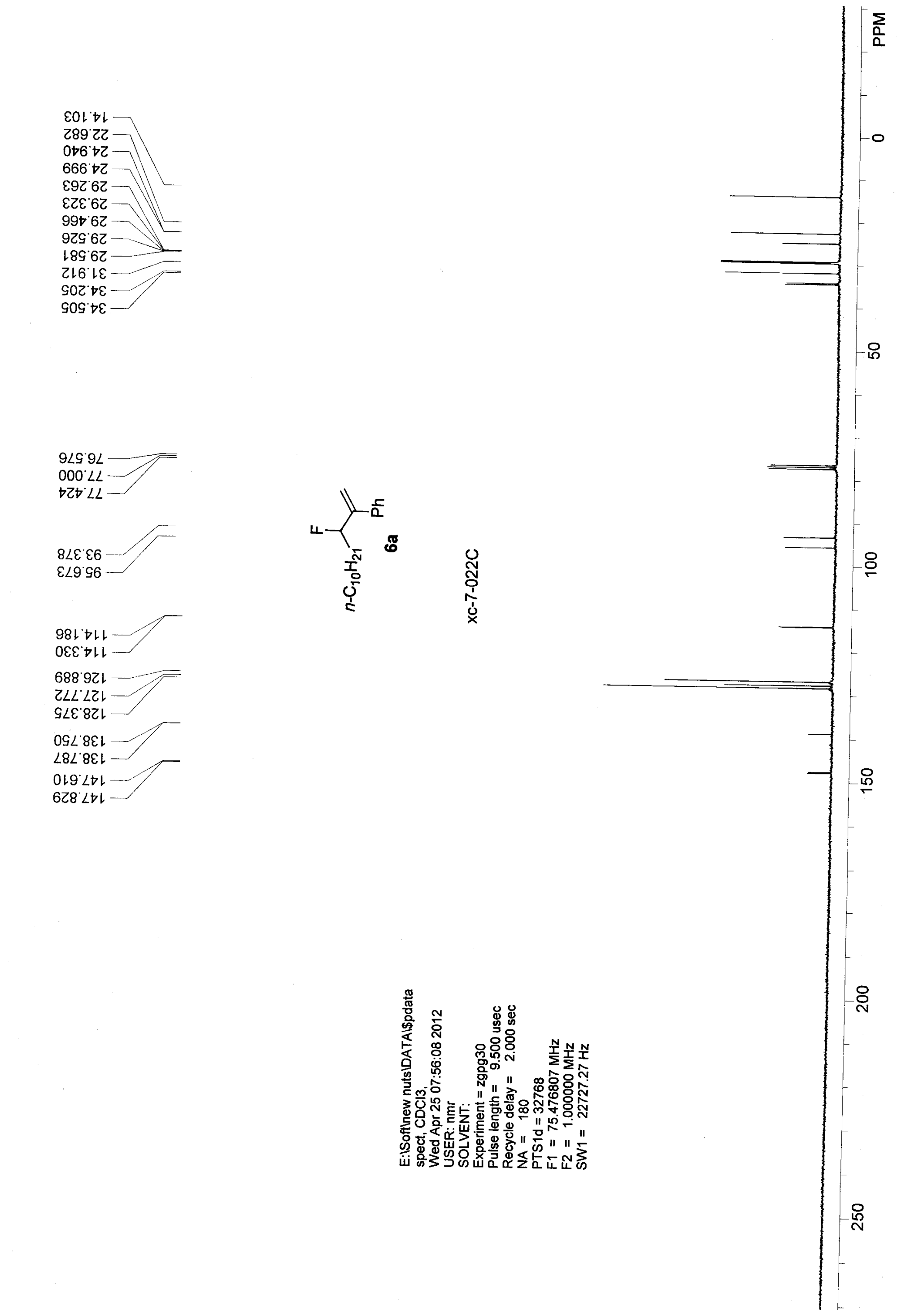
xc-6-156-3C

E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Mon Mar 26 08:00:24 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 165
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz



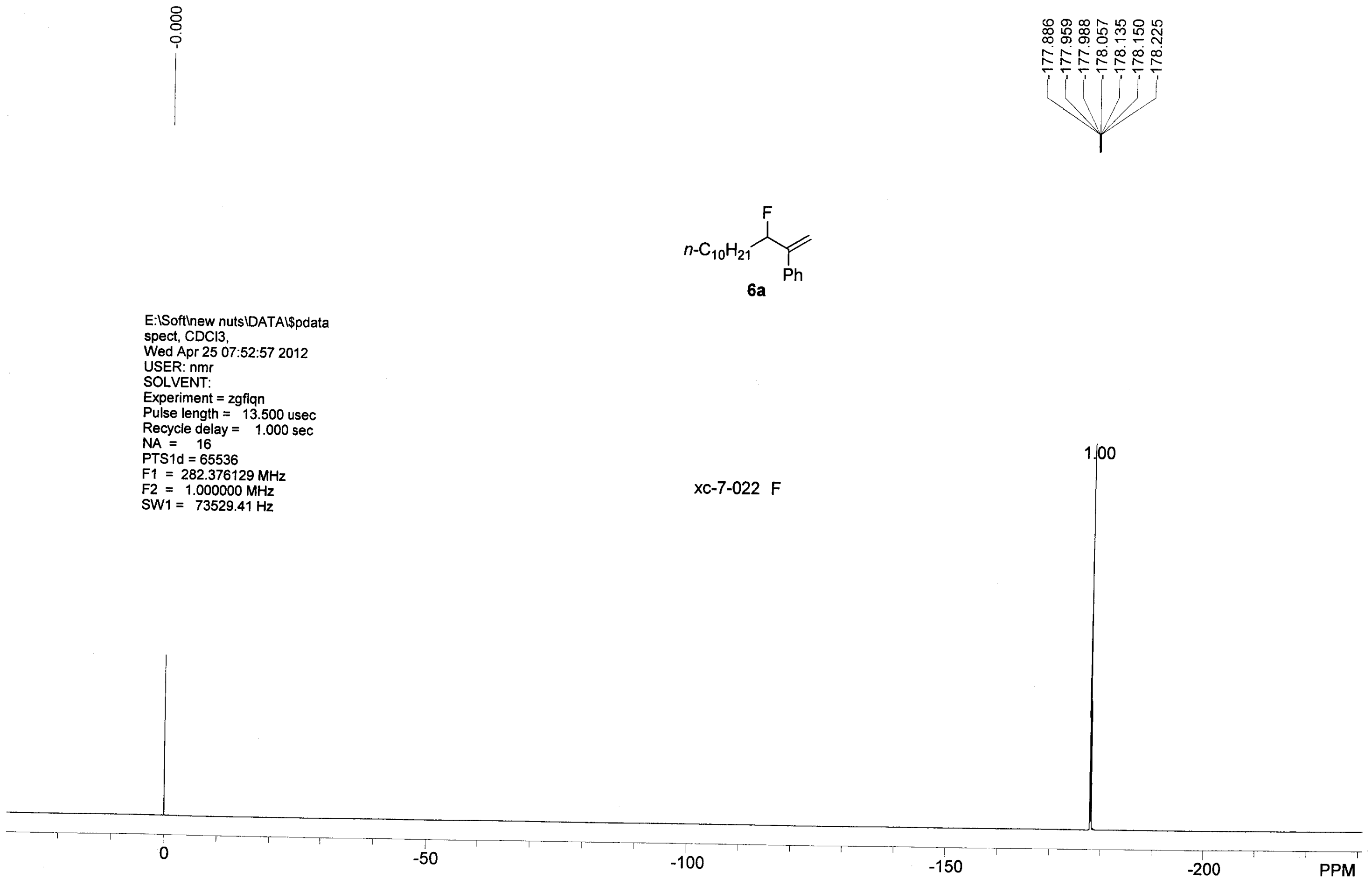
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Mon Mar 26 07:57:10 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz


-0.000
0.850
0.872
0.891
1.229
1.359
1.380
1.389
1.419
1.466
1.576
1.597
1.624
1.642
1.667
1.691
1.714
5.219
5.239
5.259
5.369
5.396
5.417
7.192
7.255
7.272
7.292
7.318
7.340
7.393
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 25 07:49:14 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
F
n-C10H21
Ph
6a
xc-7-022
16.39
3.02
2.01
3.00
5.39
PPM
0
2
4
6
8


14.103
22.682
24.940
24.999
29.263
29.323
29.466
29.526
29.581
31.912
34.205
34.505
76.576
77.000
77.424
93.378
95.673
114.186
114.330
126.889
127.772
128.375
138.750
138.787
147.610
147.829
F
n-C10H21
Ph
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 25 07:56:08 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 180
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

6a

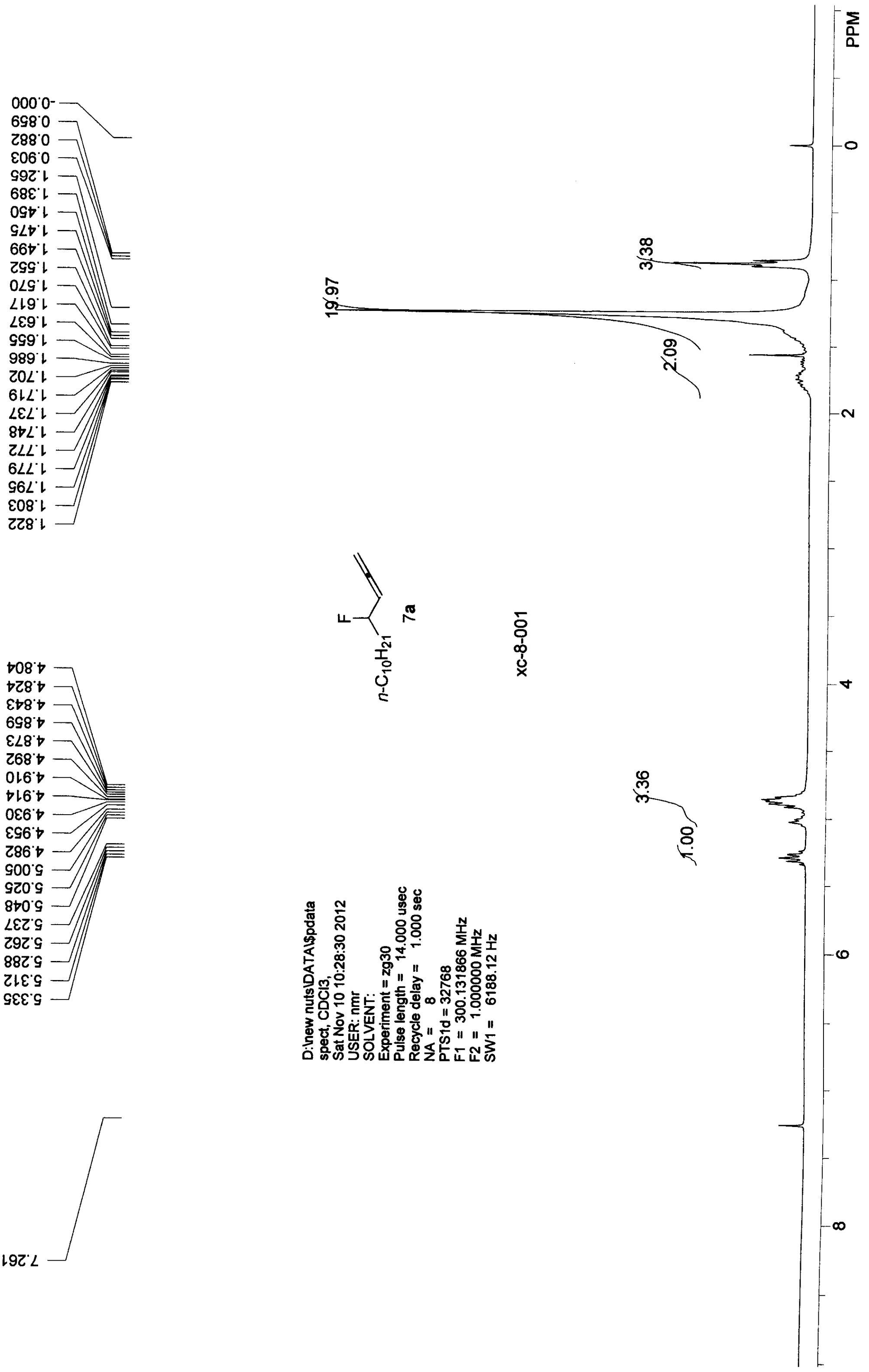
xc-7-022C



6a

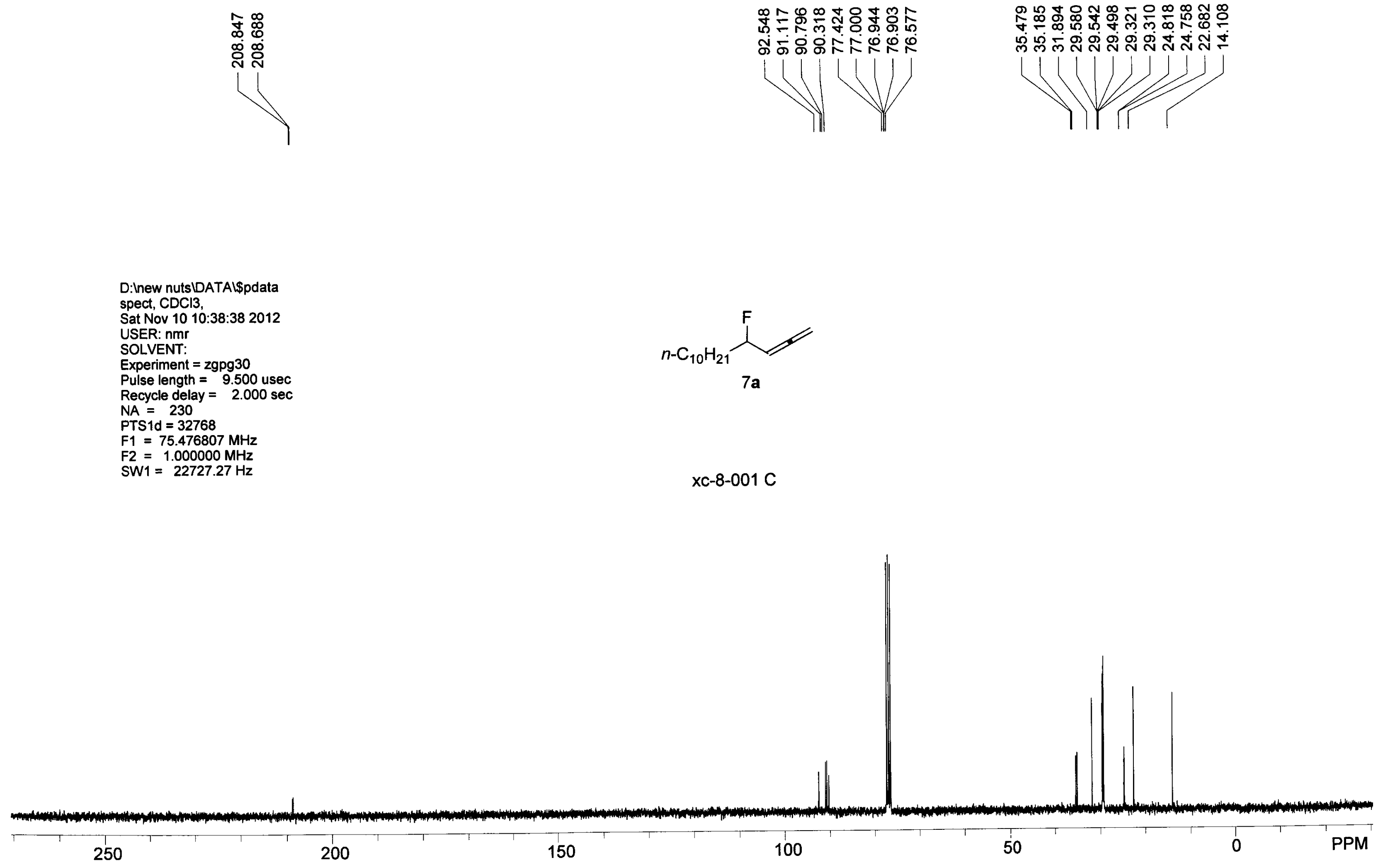
xc-7-022 F

E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 25 07:52:57 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz



7a

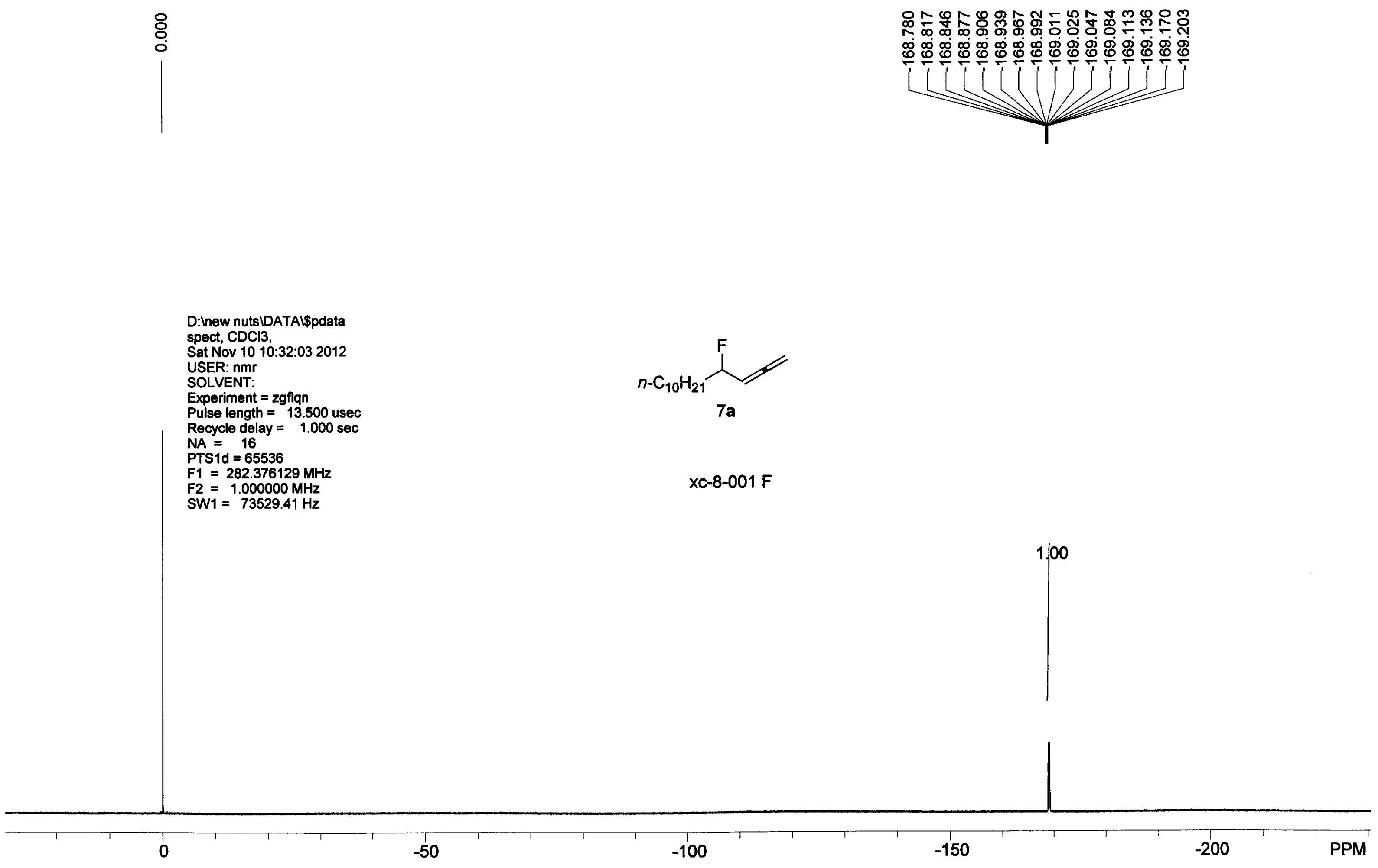
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Nov 10 10:28:30 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



7a

D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Nov 10 10:38:38 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 230
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

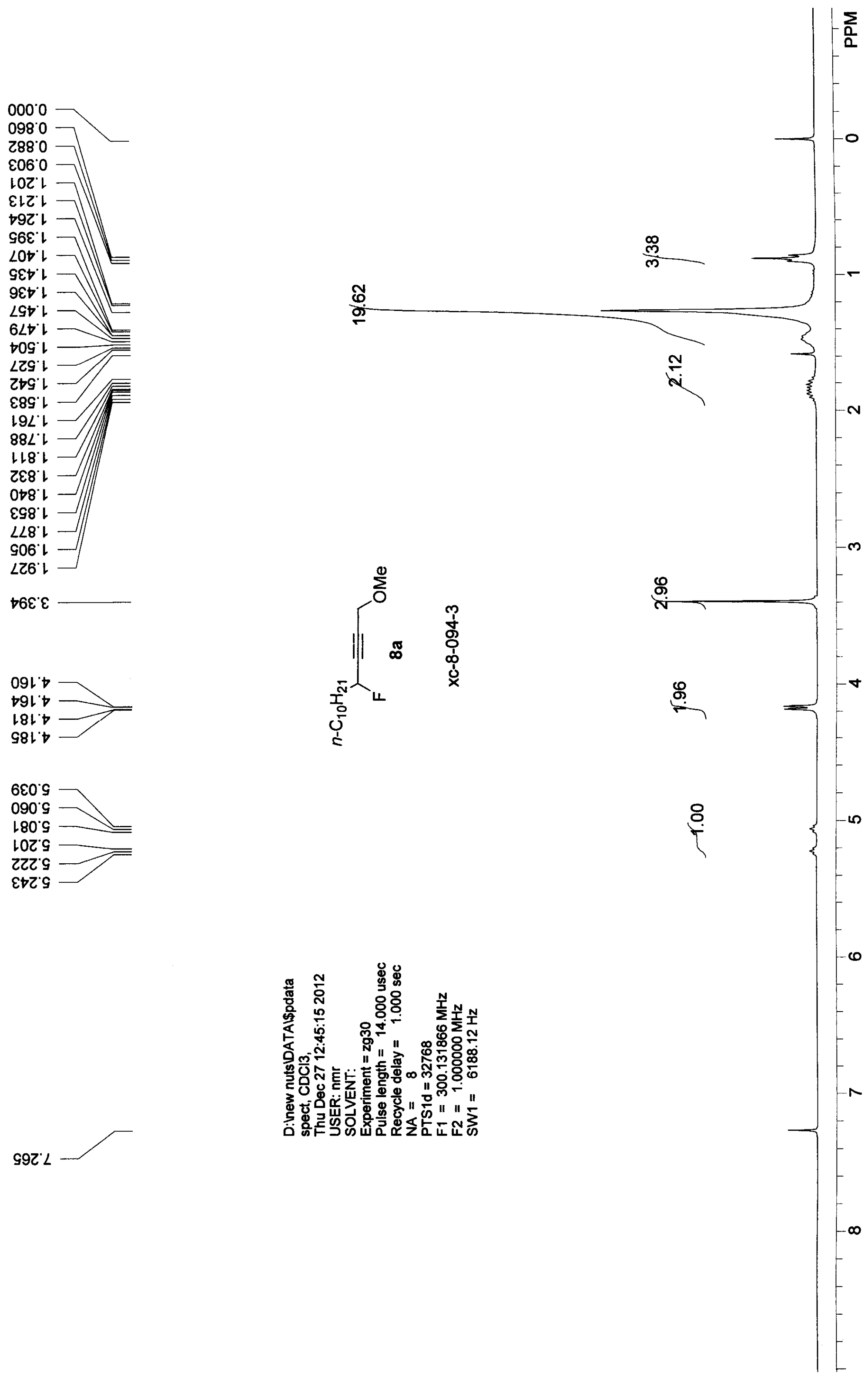
xc-8-001 C



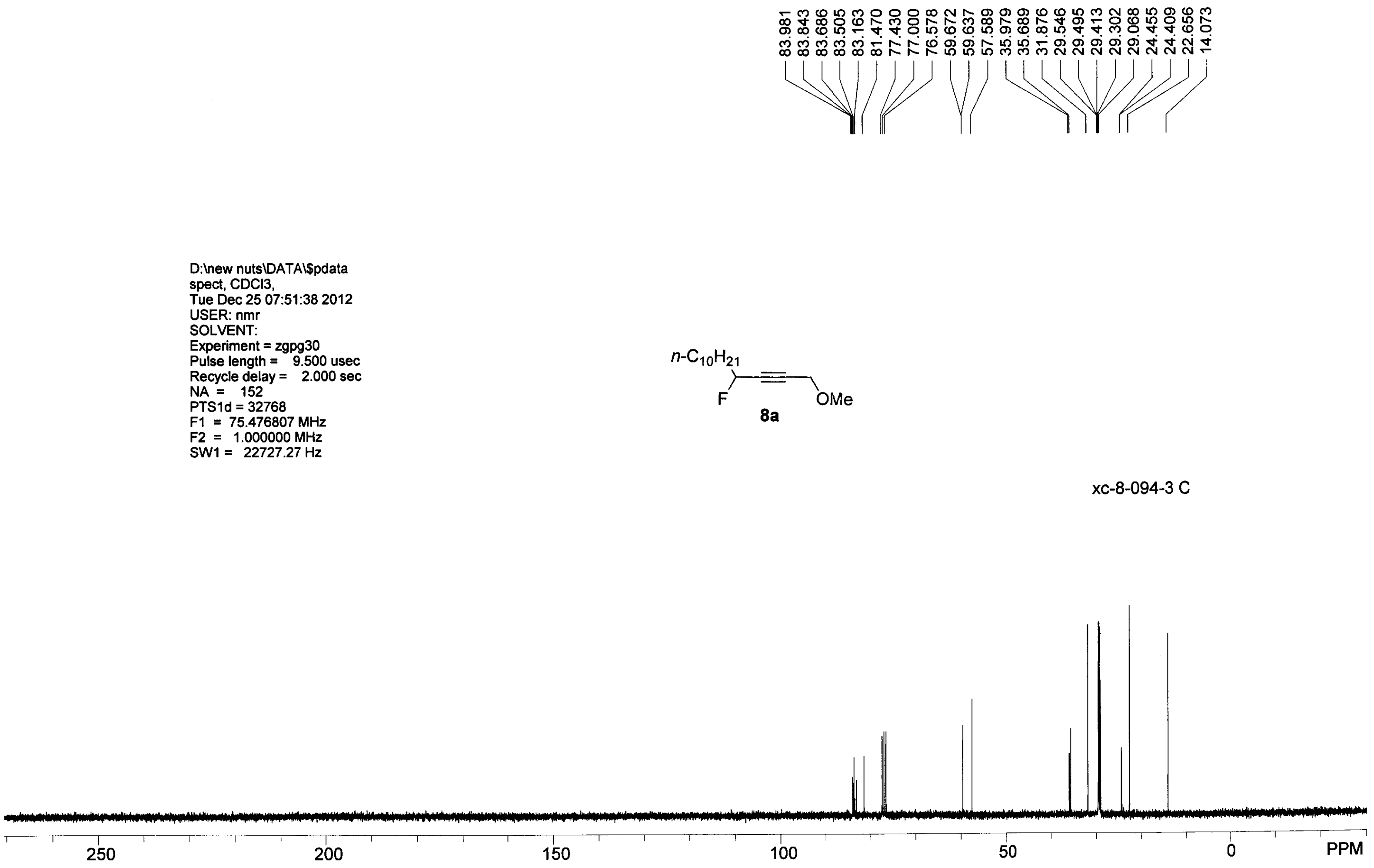
7a

xc-8-001 F

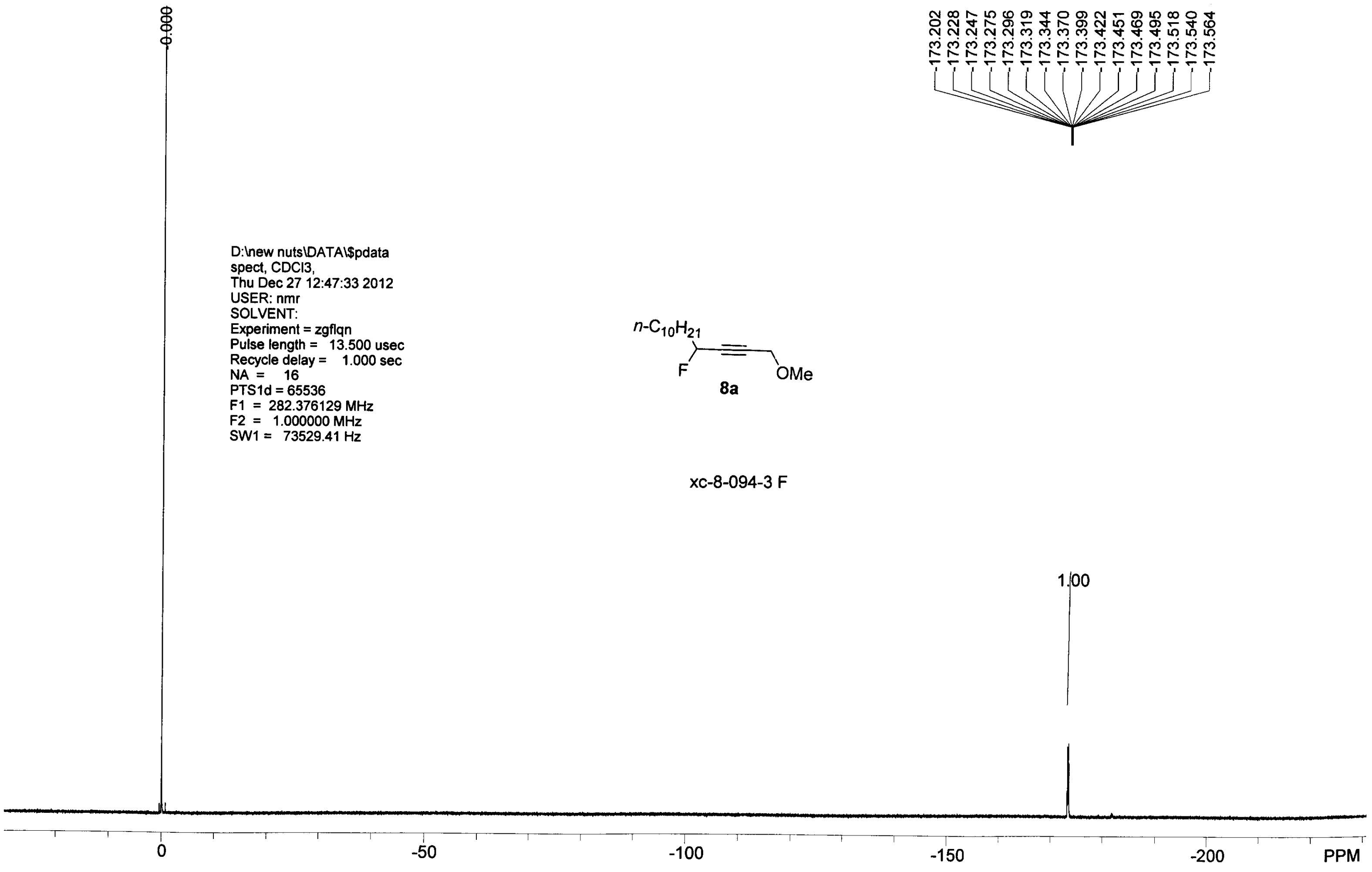
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Nov 10 10:32:03 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

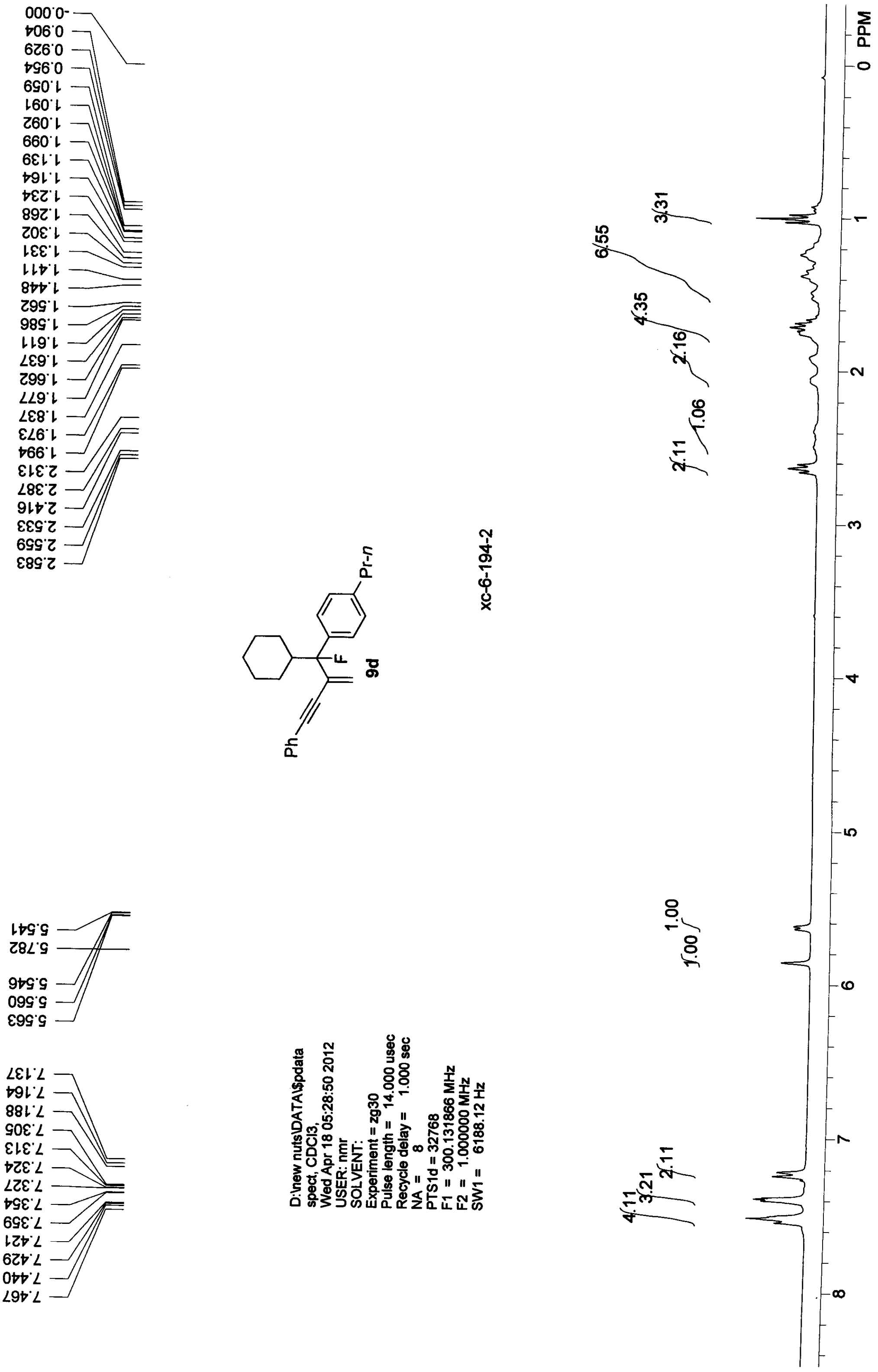


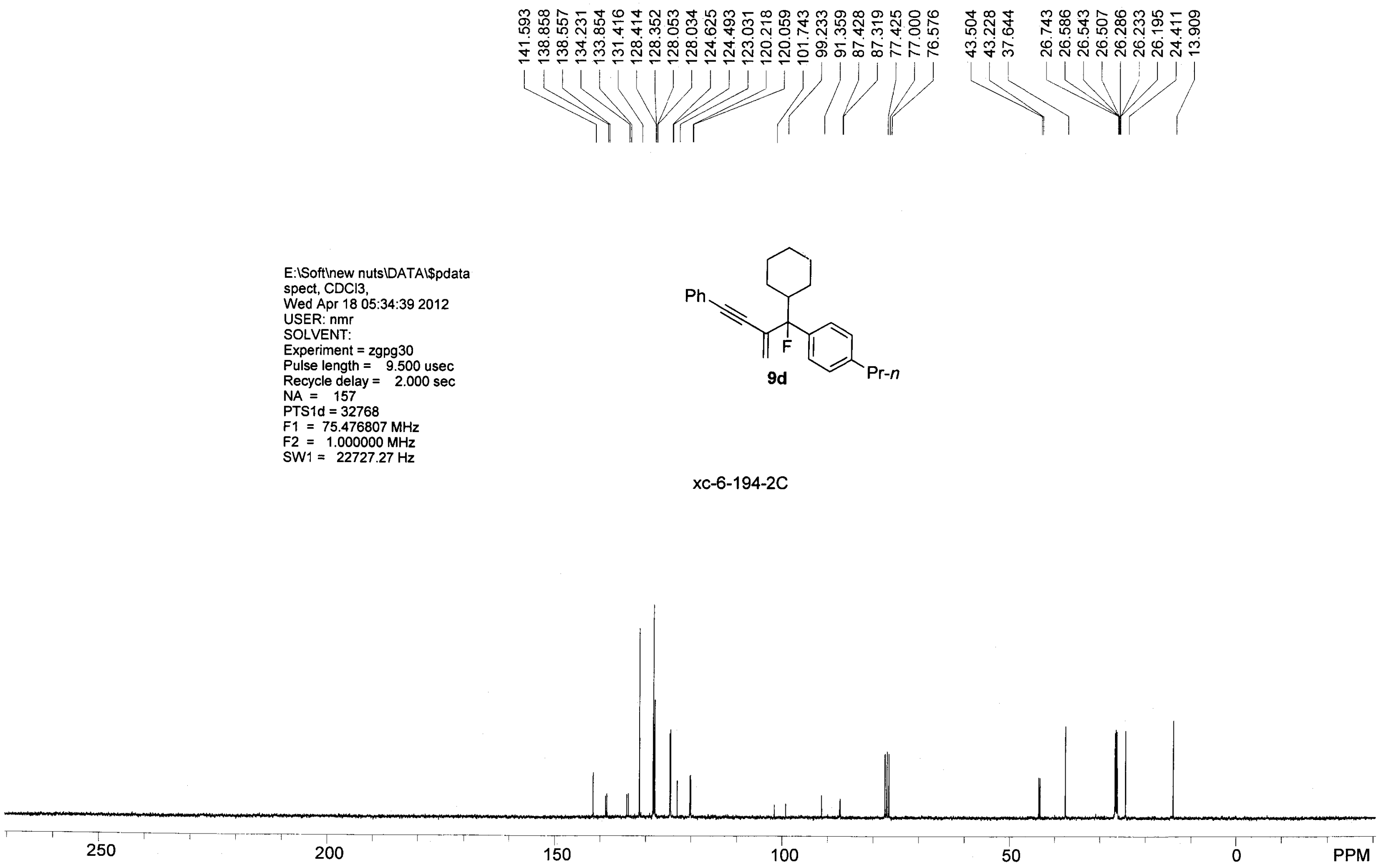
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 27 12:45:15 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue Dec 25 07:51:38 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 152
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz


0.000
-173.202
-173.228
-173.247
-173.275
-173.296
-173.319
-173.344
-173.370
-173.399
-173.422
-173.451
-173.469
-173.495
-173.518
-173.540
-173.564
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu Dec 27 12:47:33 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
n-C10H21
F
OMe
8a
xc-8-094-3 F
1.00
0
-50
-100
-150
-200
PPM

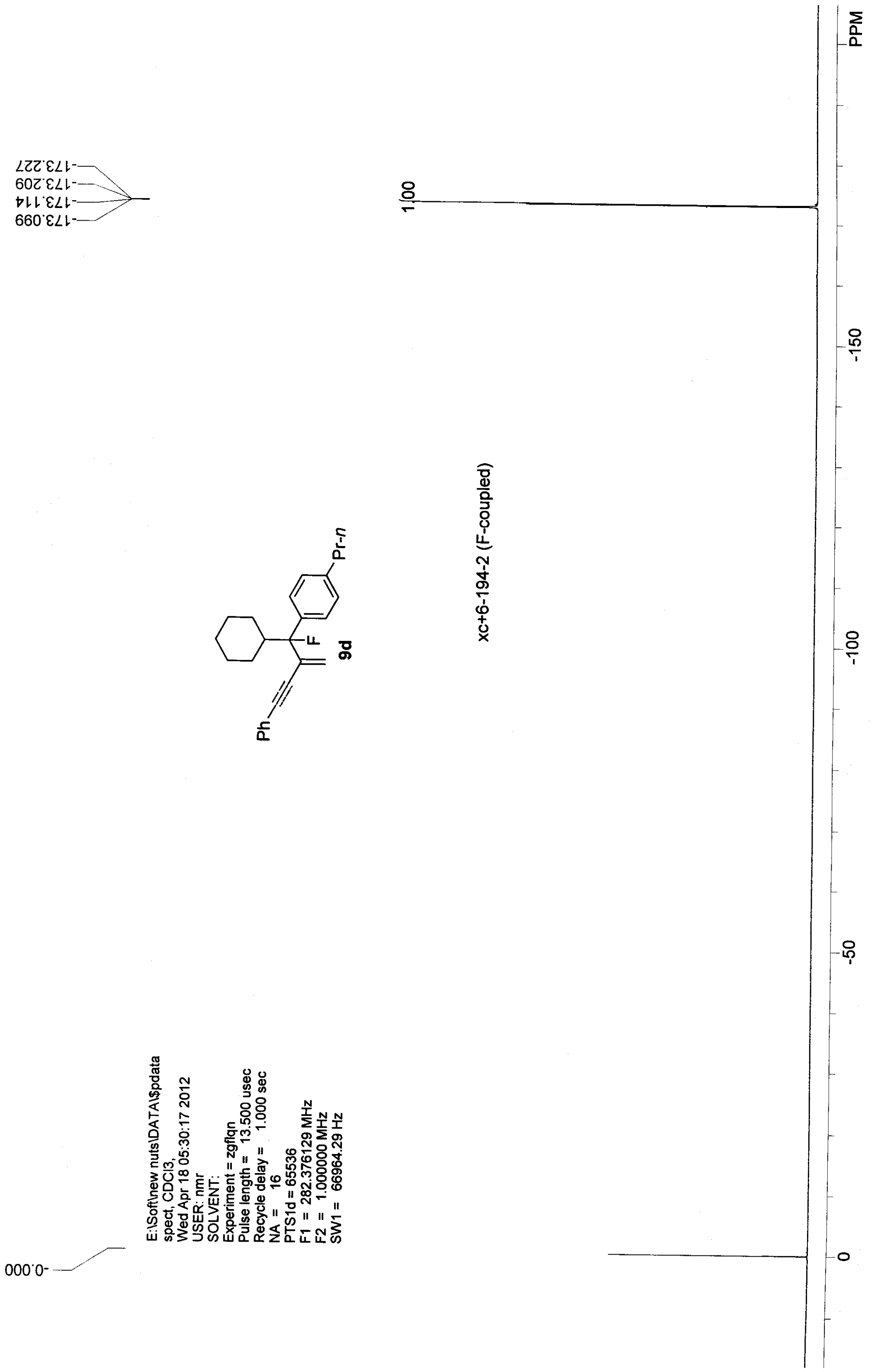

-0.000
0.904
0.929
0.954
1.059
1.091
1.092
1.099
1.139
1.164
1.234
1.268
1.302
1.331
1.411
1.448
1.562
1.586
1.611
1.637
1.662
1.677
1.837
1.973
1.994
2.313
2.387
2.416
2.533
2.559
2.583
5.541
5.782
5.546
5.560
5.563
7.137
7.164
7.188
7.305
7.313
7.324
7.327
7.354
7.359
7.421
7.429
7.440
7.467
Ph
F
Pr-n
9d
xc-6-194-2
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 18 05:28:50 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.55
3.31
4.35
2.16
1.06
2.11
1.00
1.00
2.11
3.21
4.11
0 PPM
1
2
3
4
5
6
7
8

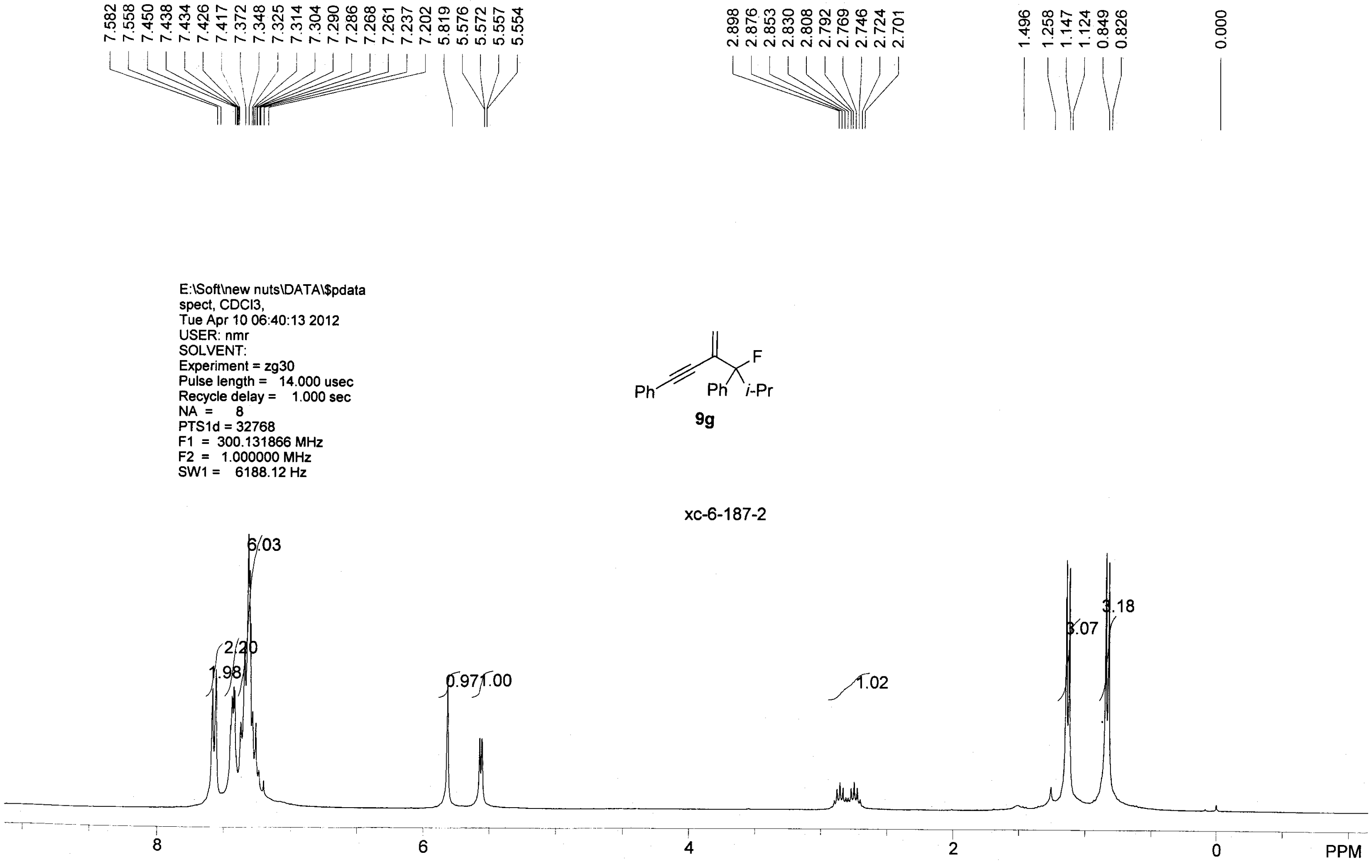


9d

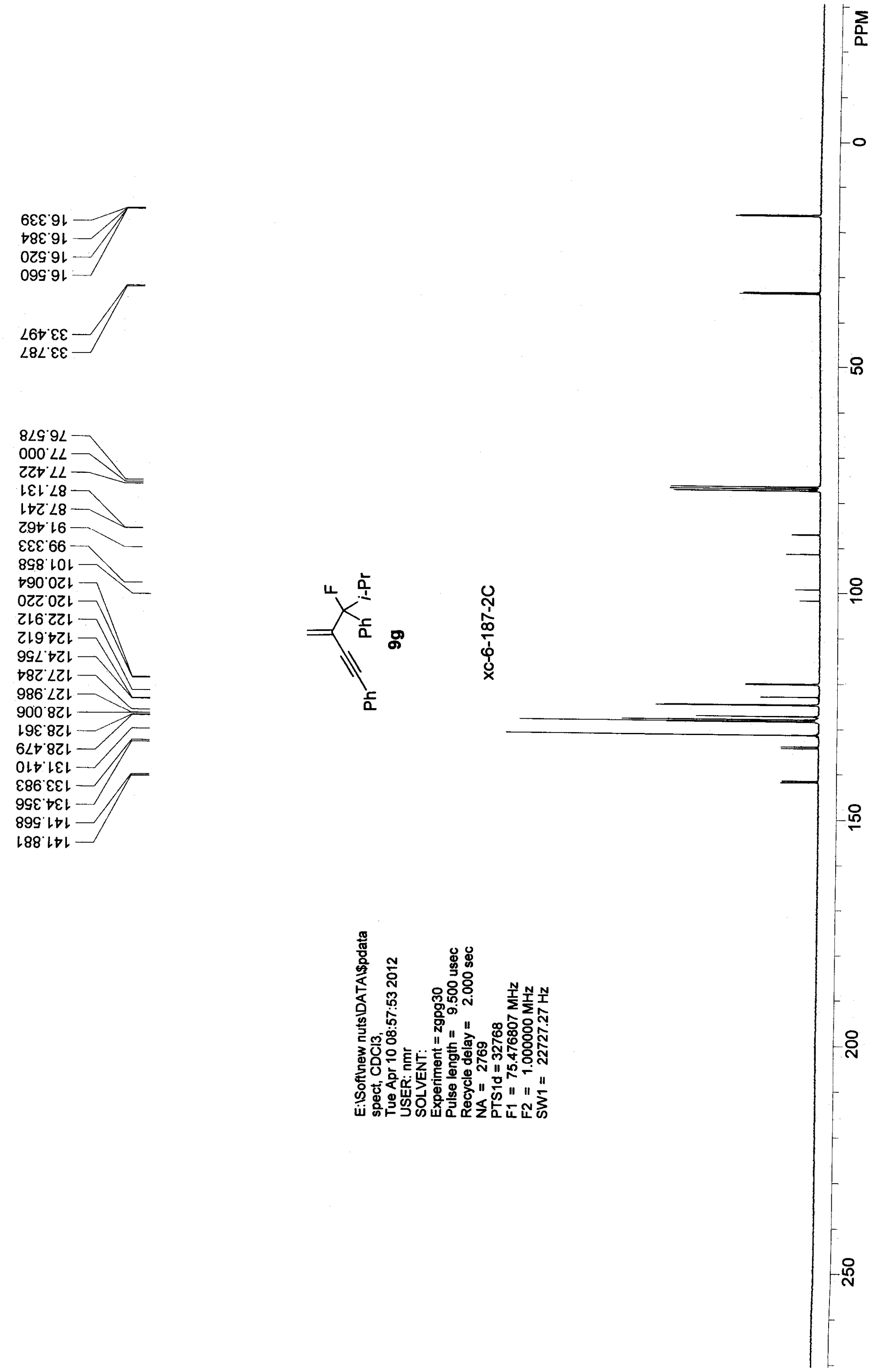
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 18 05:34:39 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 157
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

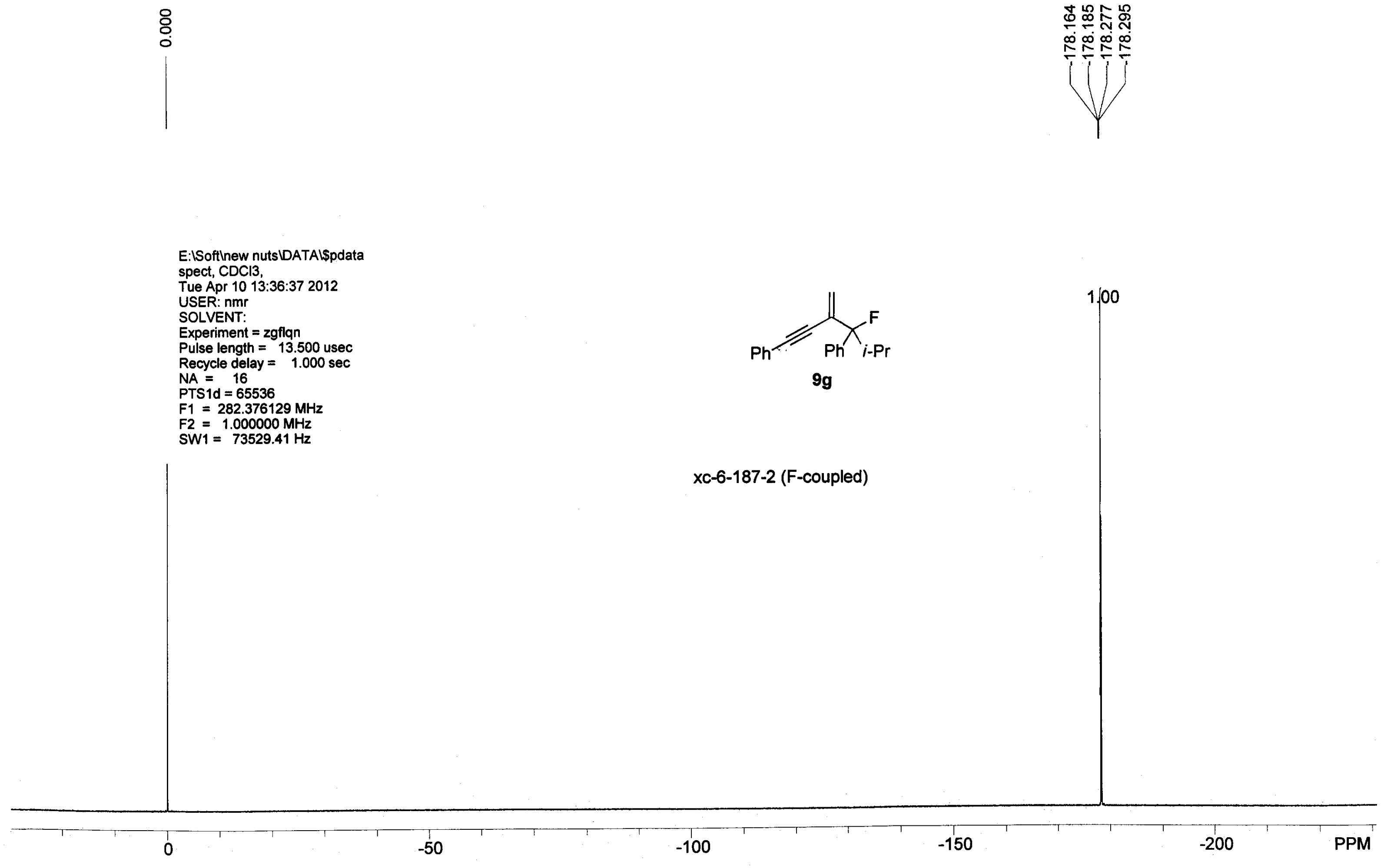
xc-6-194-2C


-173.099
-173.114
-173.209
-173.227
1.00
-0.000
xc+6-194-2 (F-coupled)
Ph
F
Pr-n
9d
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 18 05:30:17 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
PPM
-150
-100
-50
0



```
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Tue Apr 10 06:40:13 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length =   14.000 usec
Recycle delay =    1.000 sec
NA =     8
PTS1d = 32768
F1  =  300.131866 MHz
F2  =   1.000000 MHz
SW1 =    6188.12 Hz
```

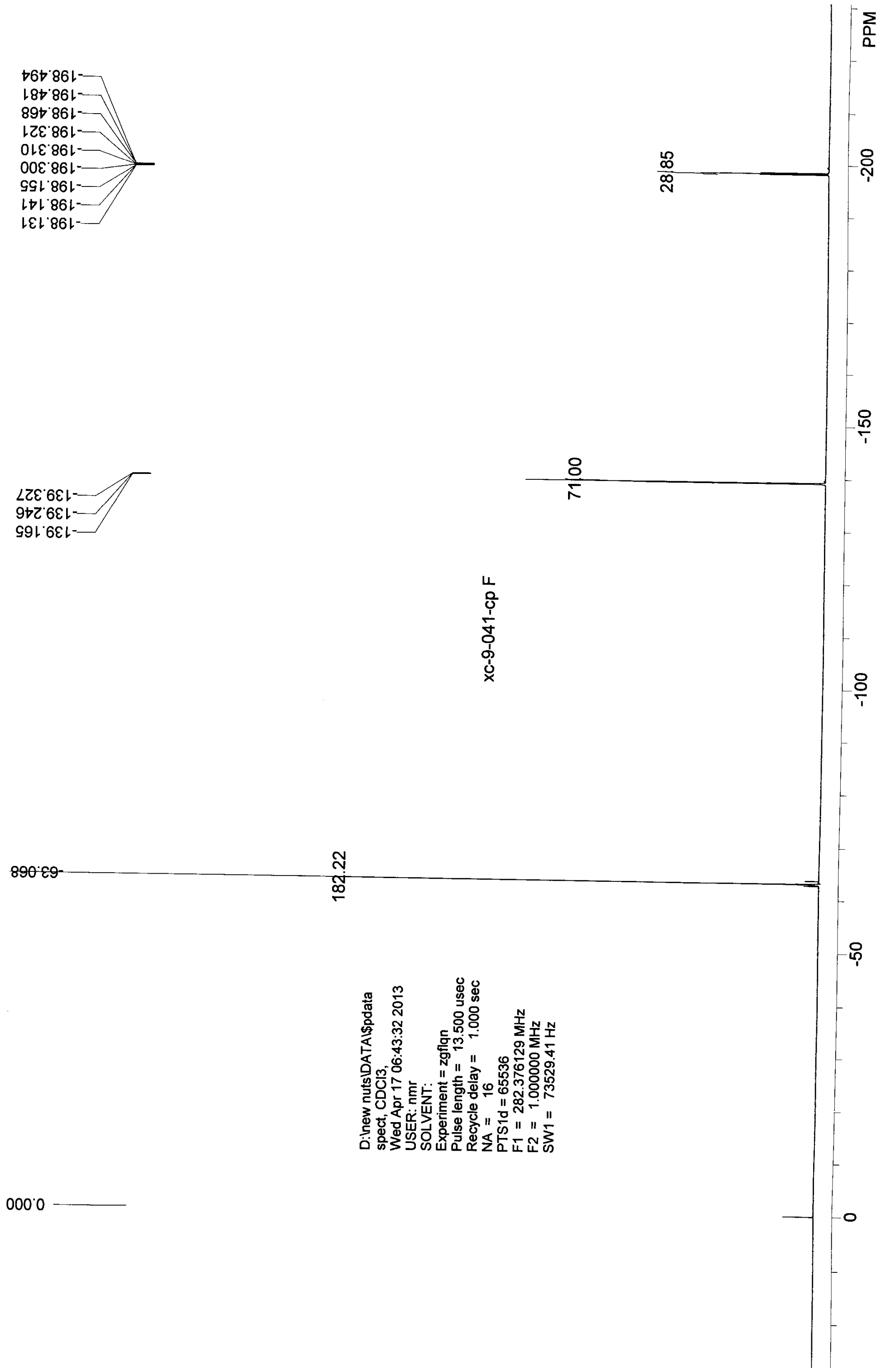

16.339
16.384
16.520
16.560
33.497
33.787
76.578
77.000
77.422
87.131
87.241
91.462
99.333
101.858
120.064
120.220
122.912
124.612
124.756
127.284
127.986
128.006
128.361
128.479
131.410
133.983
134.356
141.568
141.881
F
Ph
i-Pr
Ph
9g
xc-6-187-2C
E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Tue Apr 10 08:57:53 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2769
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

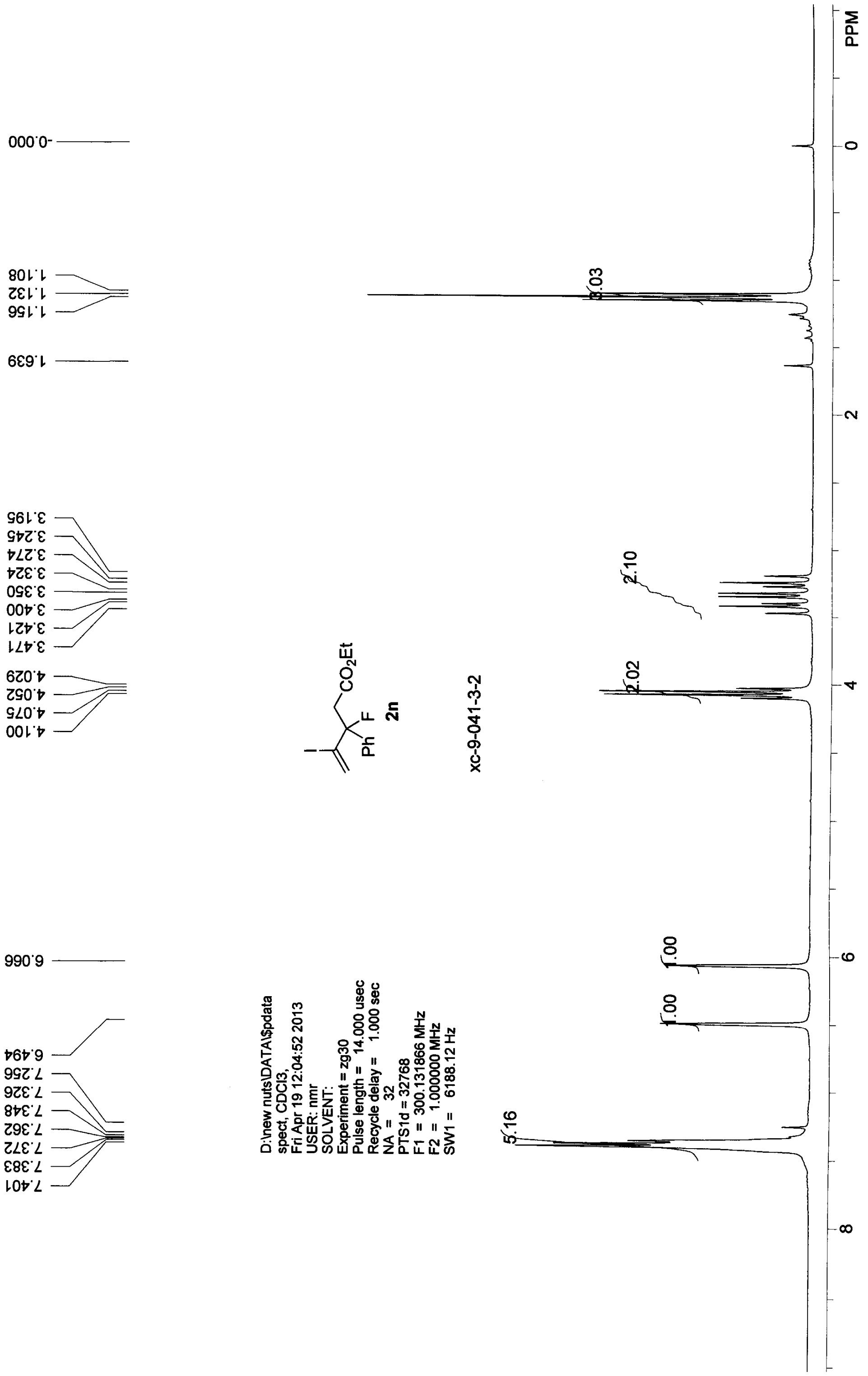


9g

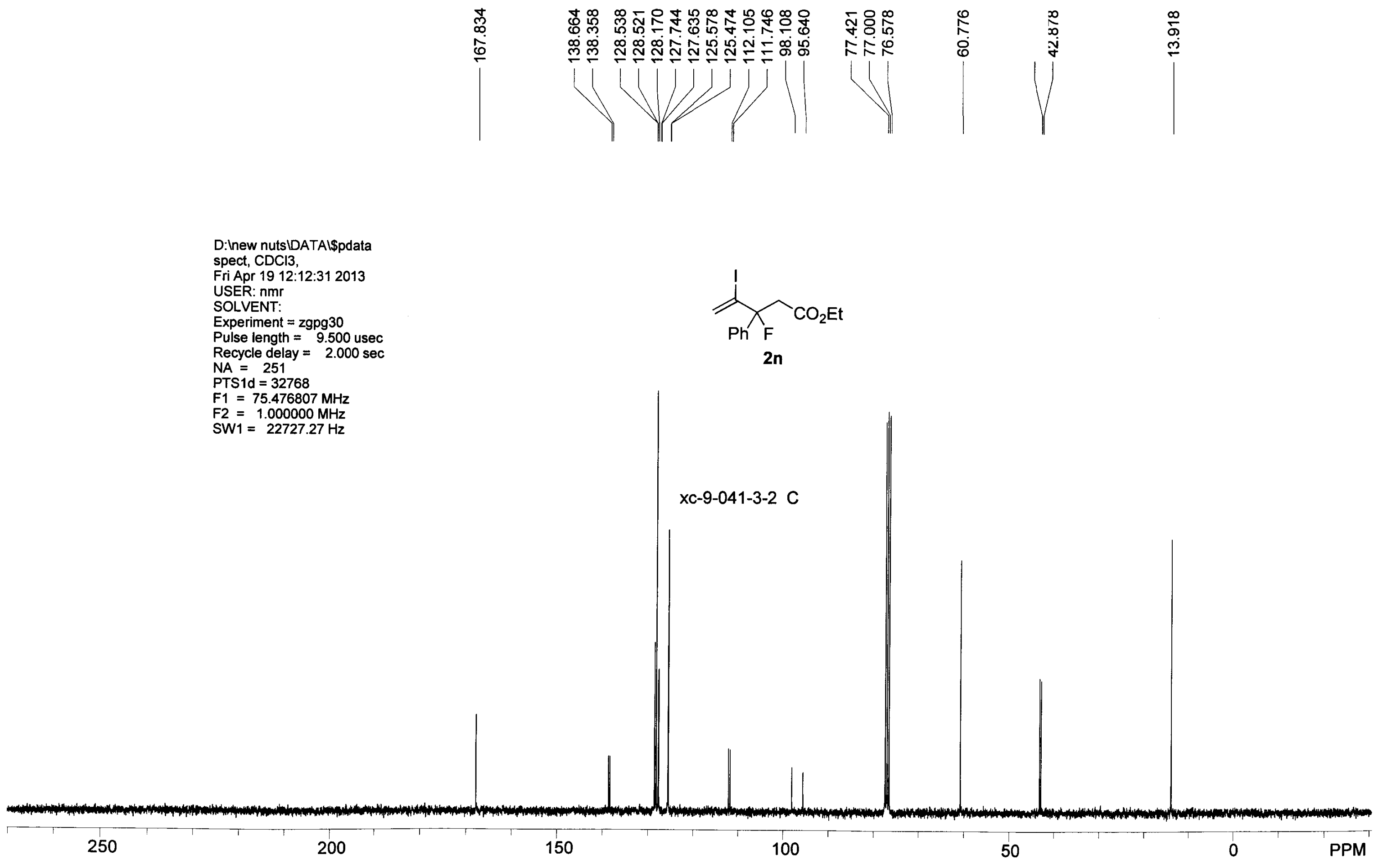
xc-6-187-2 (F-coupled)

E:\Soft\new nuts\DATA\$pdata
spect, CDCl3,
Tue Apr 10 13:36:37 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz

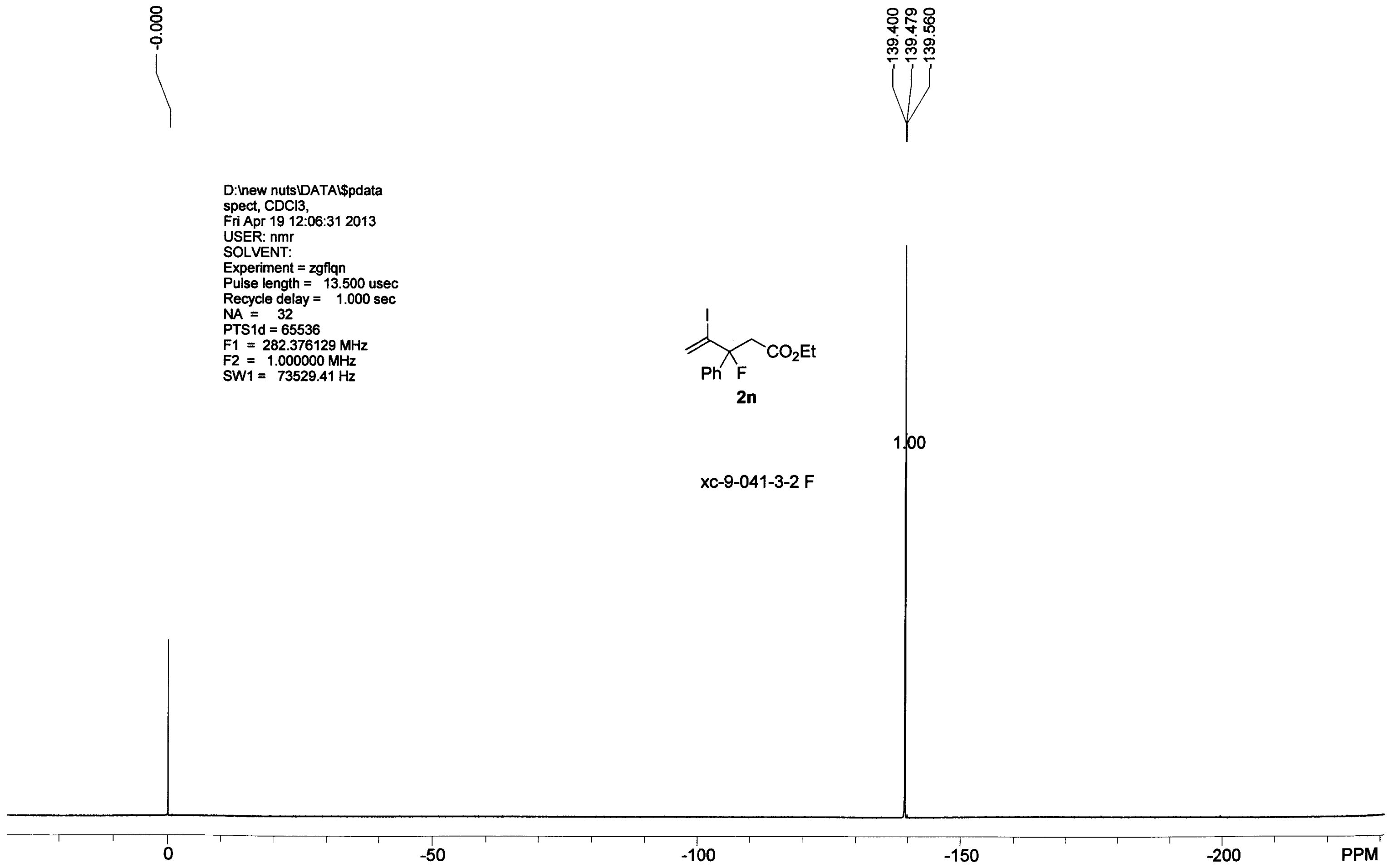

xc-9-041-cp F
-198.494
-198.481
-198.468
-198.321
-198.310
-198.300
-198.155
-198.141
-198.131
-139.327
-139.246
-139.165
-63.068
0.000
28.85
71.00
182.22
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed Apr 17 06:43:32 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
-200
-150
-100
-50
0
PPM

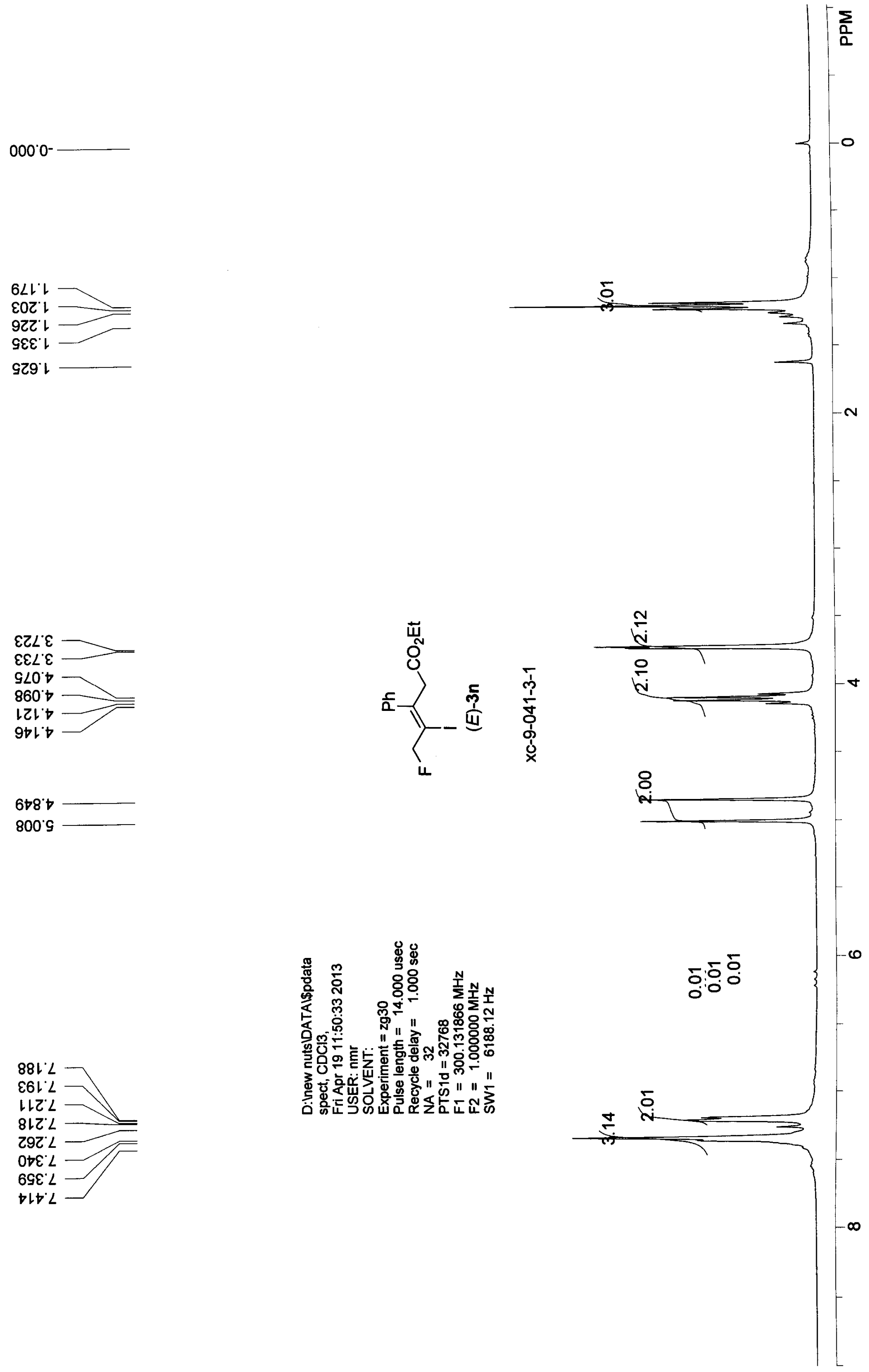


D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Apr 19 12:04:52 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Apr 19 12:12:31 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 251
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

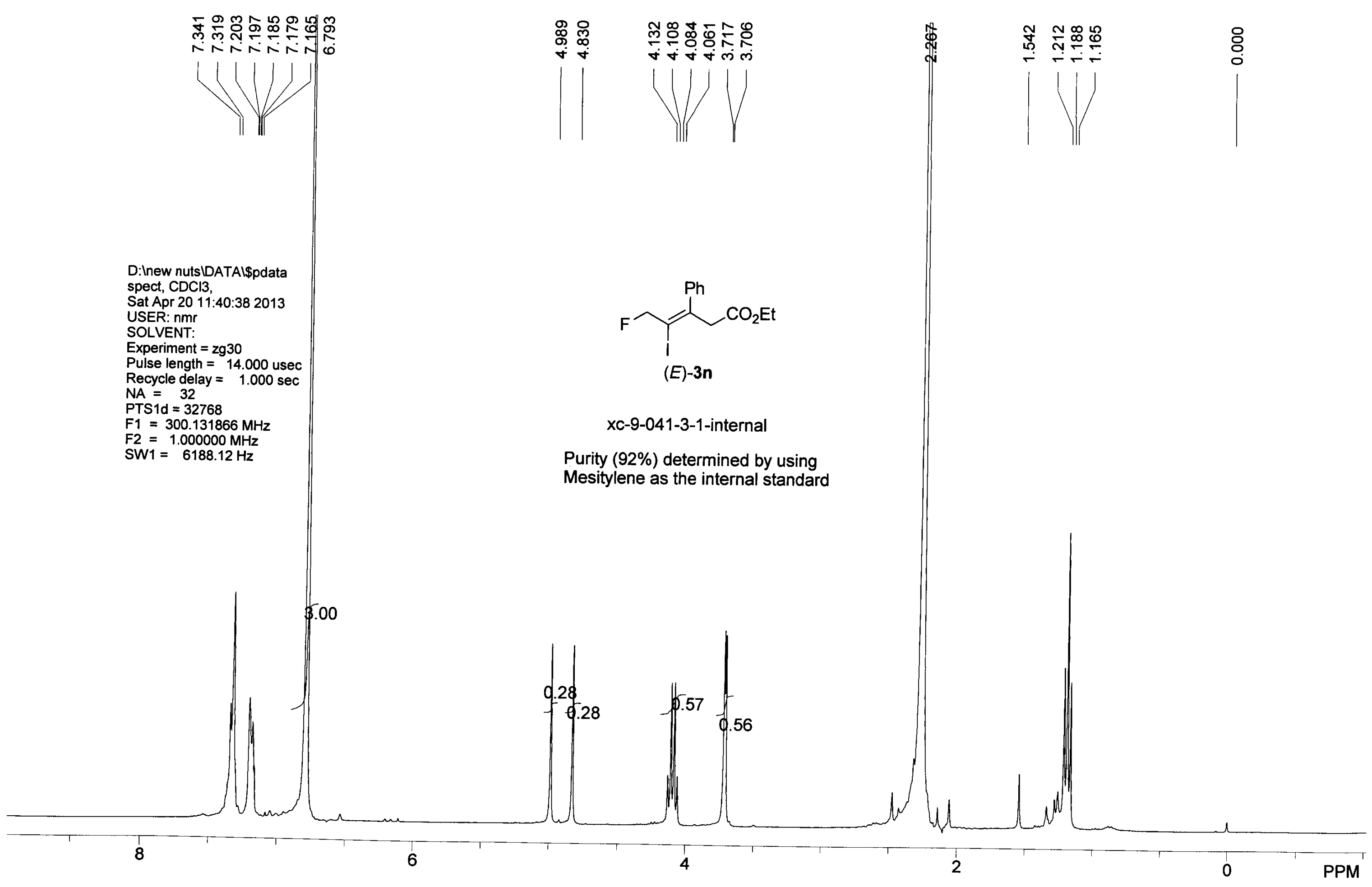

-0.000
139.400
139.479
139.560
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Apr 19 12:06:31 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
I
CO2Et
Ph
F
2n
1.00
xc-9-041-3-2 F
0
-50
-100
-150
-200
PPM

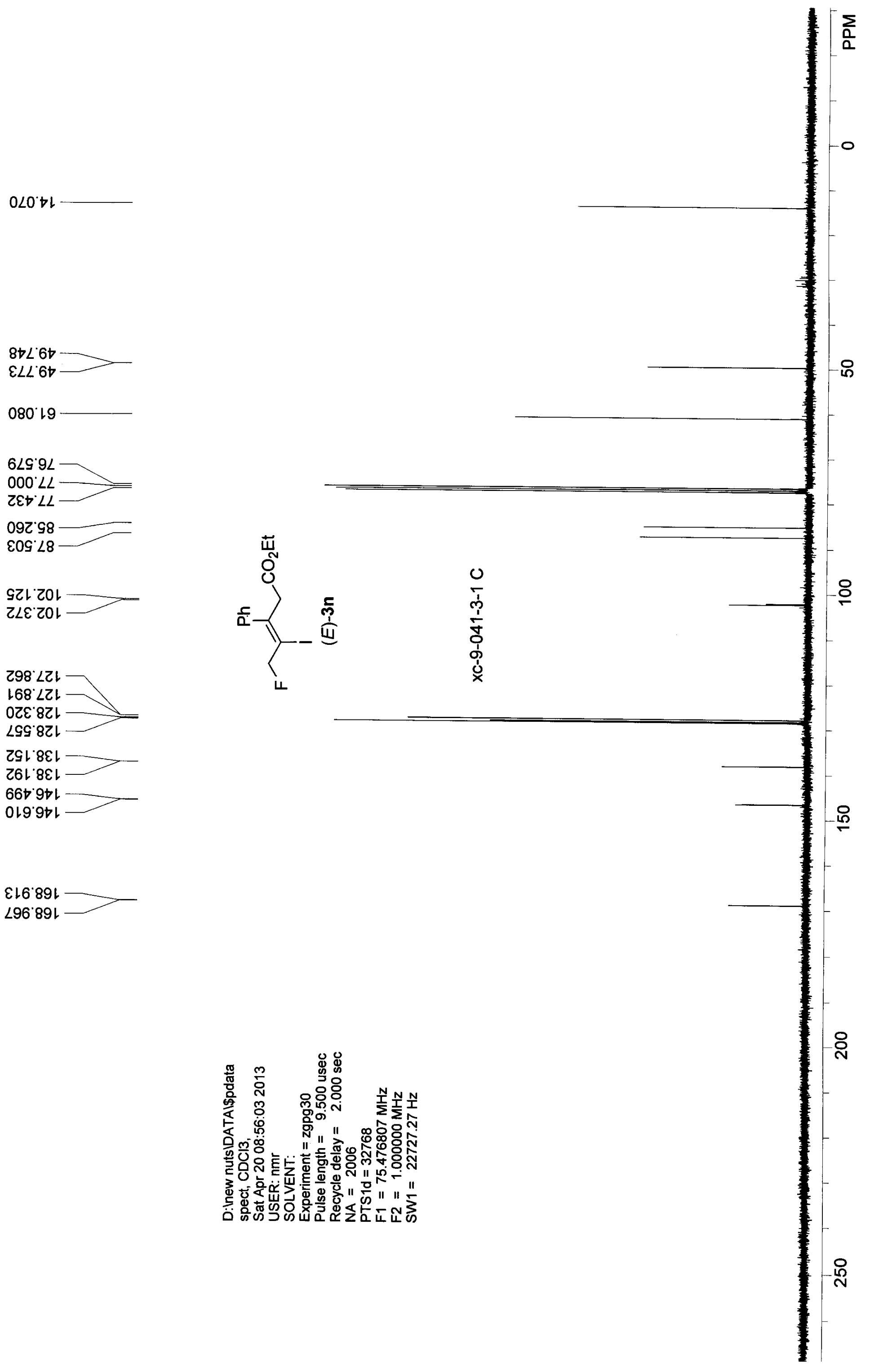


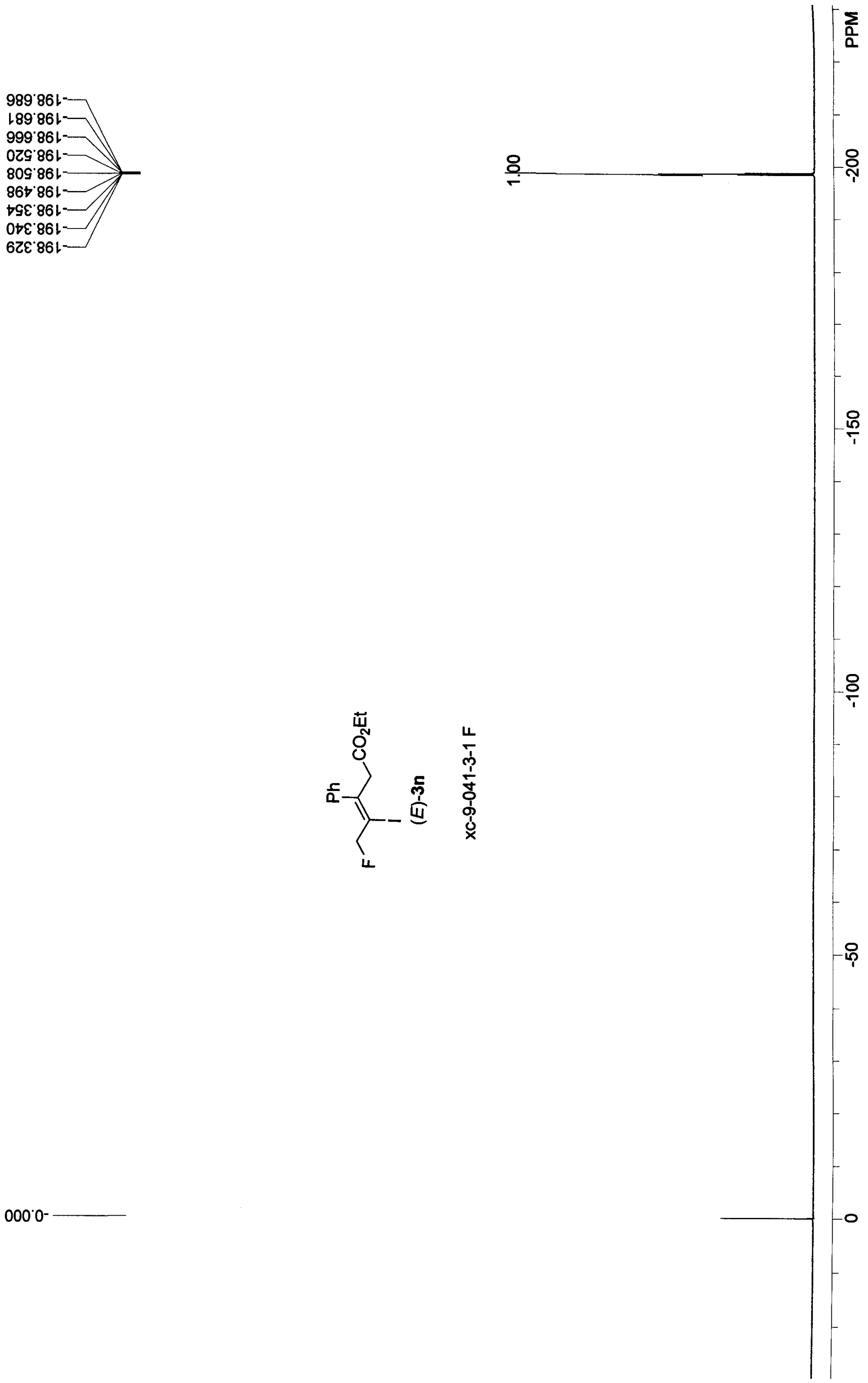
(E)-3n

xc-9-041-3-1

D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Apr 19 11:50:33 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz


7.341
7.319
7.203
7.197
7.185
7.179
7.165
6.793
4.989
4.830
4.132
4.108
4.084
4.061
3.717
3.706
2.267
1.542
1.212
1.188
1.165
0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 11:40:38 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Ph
F
CO2Et
I
(E)-3n
xc-9-041-3-1-internal
Purity (92%) determined by using
Mesitylene as the internal standard
3.00
0.28
0.28
0.57
0.56
8
6
4
2
0
PPM

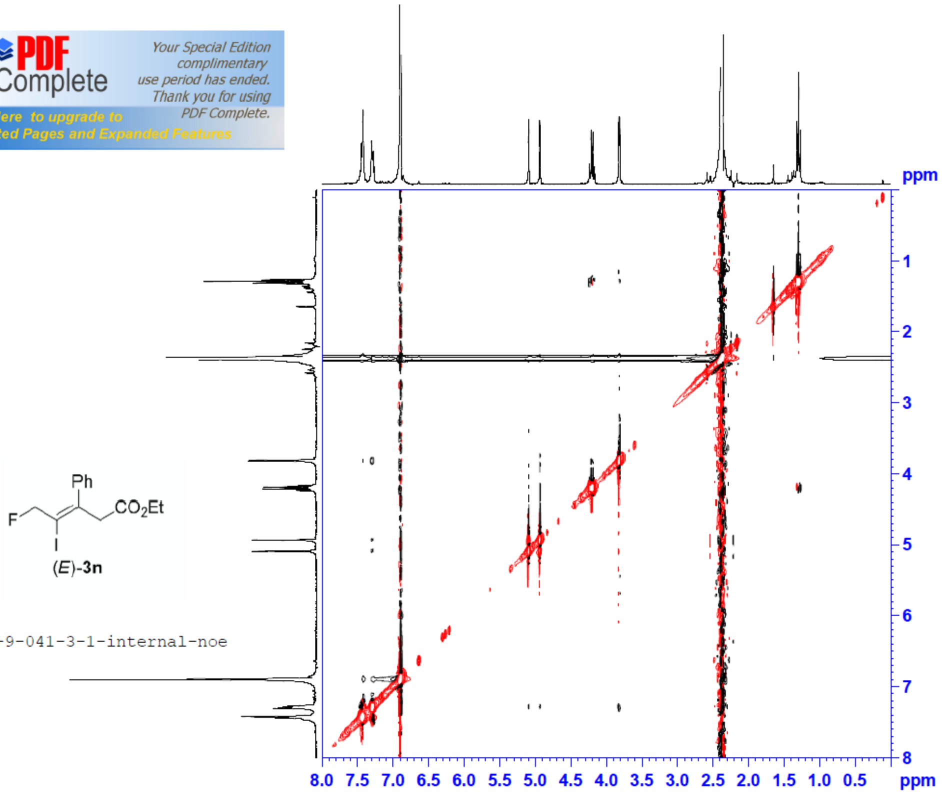

14.070
49.748
49.773
61.080
76.579
77.000
77.432
85.260
87.503
102.125
102.372
127.862
127.891
128.320
128.557
138.152
138.192
146.499
146.610
168.913
168.967
Ph
CO2Et
F
I
(E)-3n
xc-9-041-3-1 C
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 08:56:03 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2006
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250



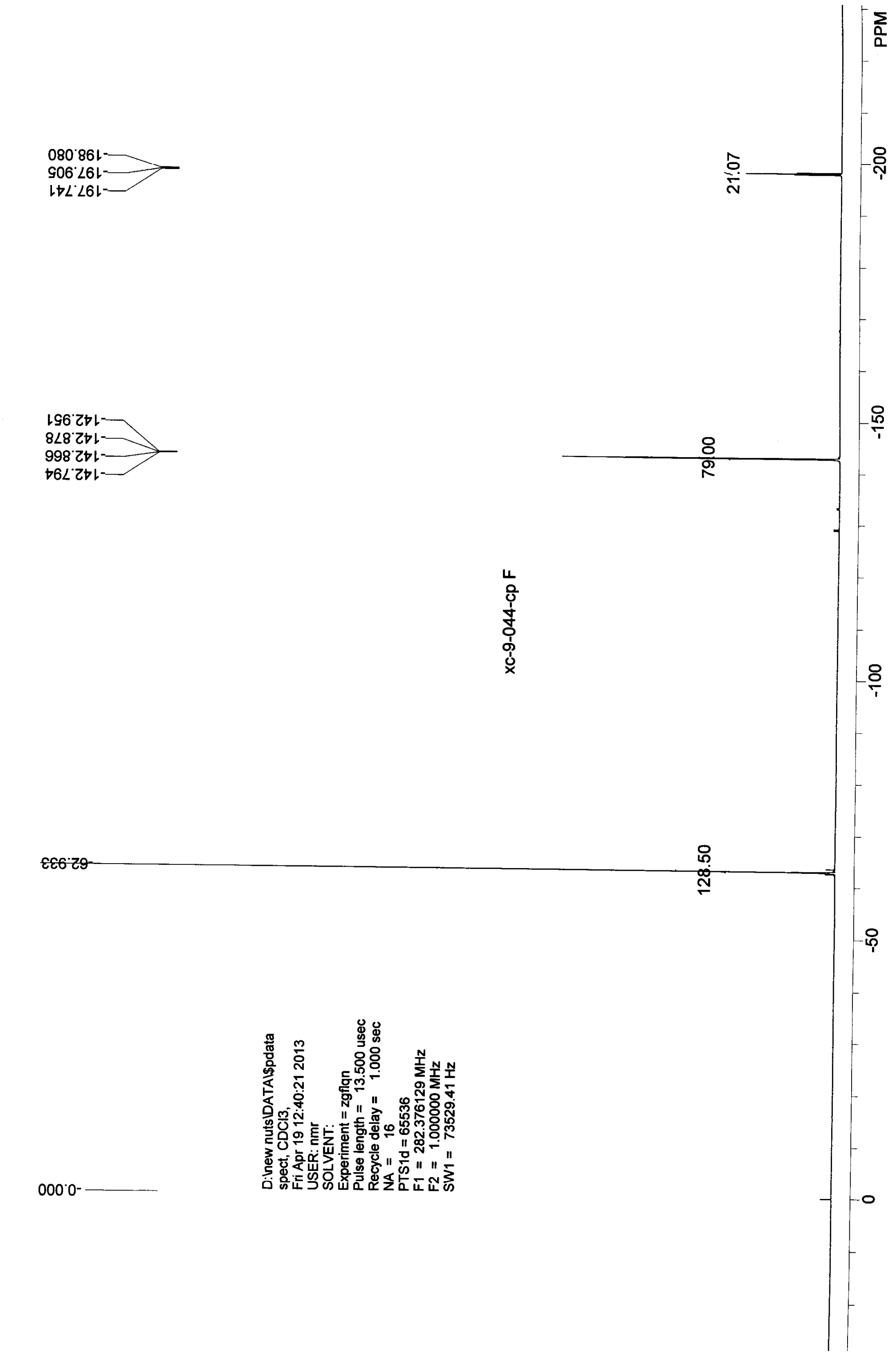
(*E*)-3n

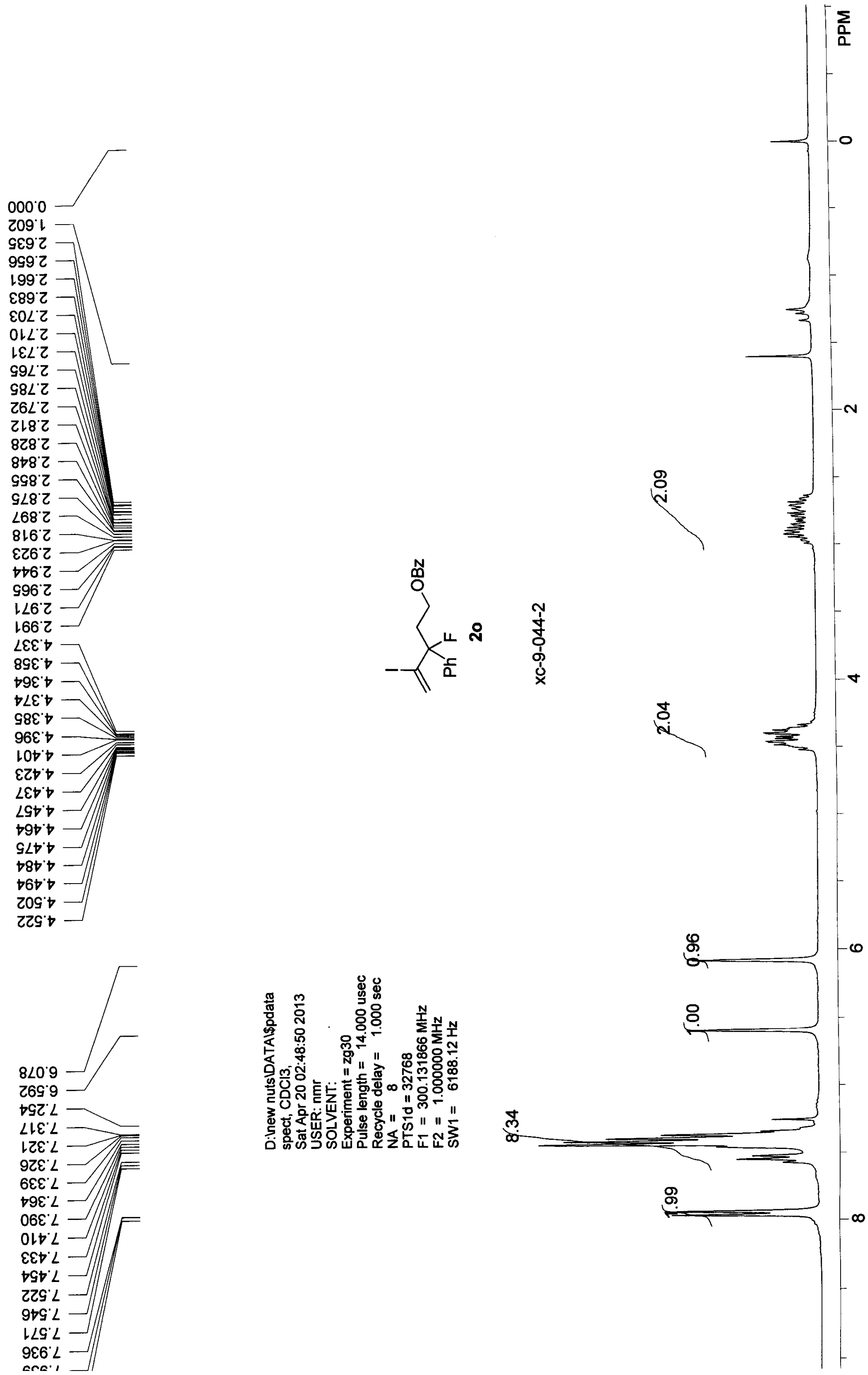
-198.686
-198.681
-198.666
-198.520
-198.508
-198.498
-198.354
-198.340
-198.329
-0.000
1.00
Ph
CO2Et
F
I
xc-9-041-3-1 F
PPM
-200
-150
-100
-50
0

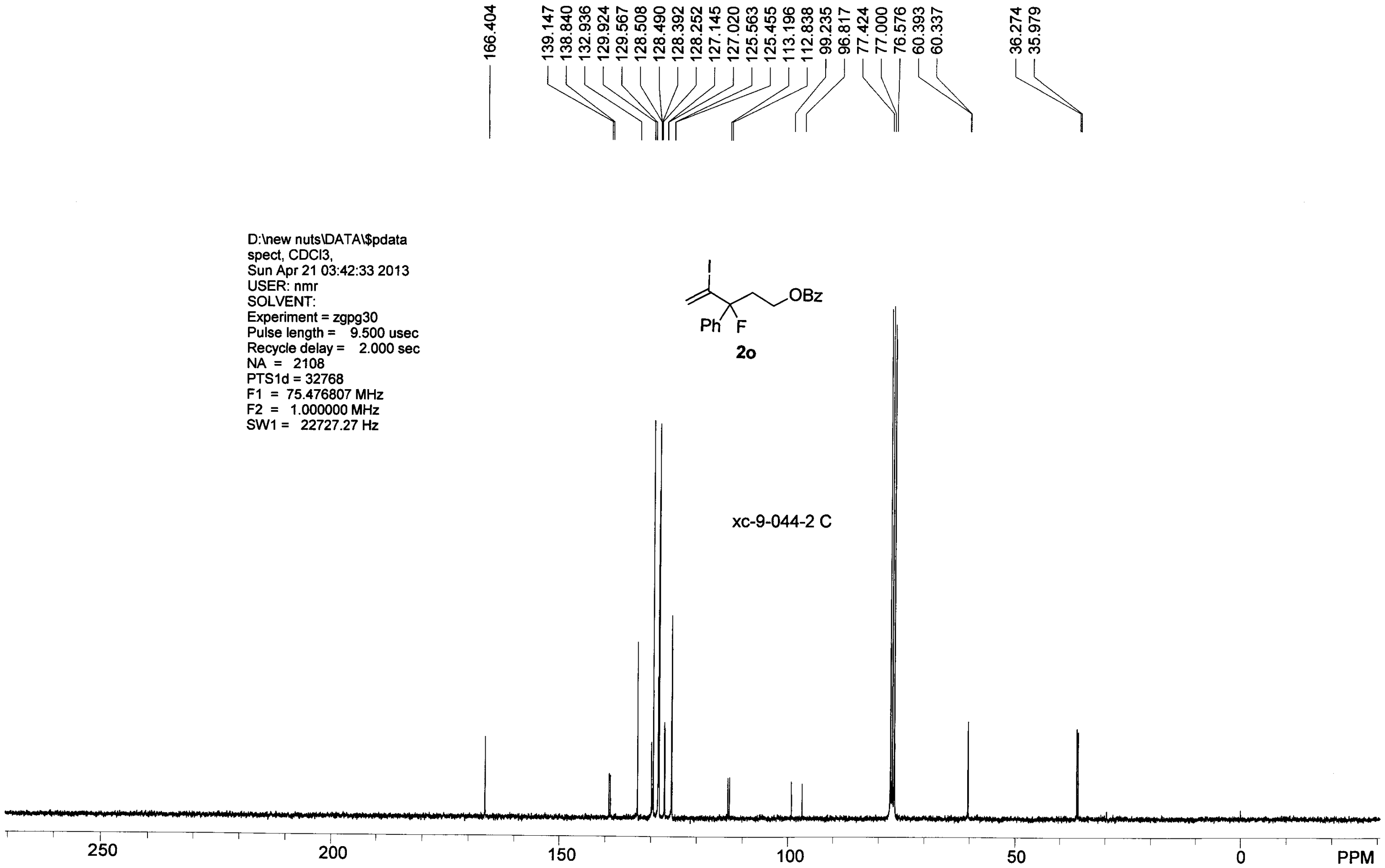
Use Mesitylene as the internal standard



(*E*)-**3n**


xc-9-044-cp F
-198.080
-197.905
-197.741
-142.951
-142.878
-142.866
-142.794
-62.933
-0.000
21.07
79.00
128.50
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Apr 19 12:40:21 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
PPM
-200
-150
-100
-50
0

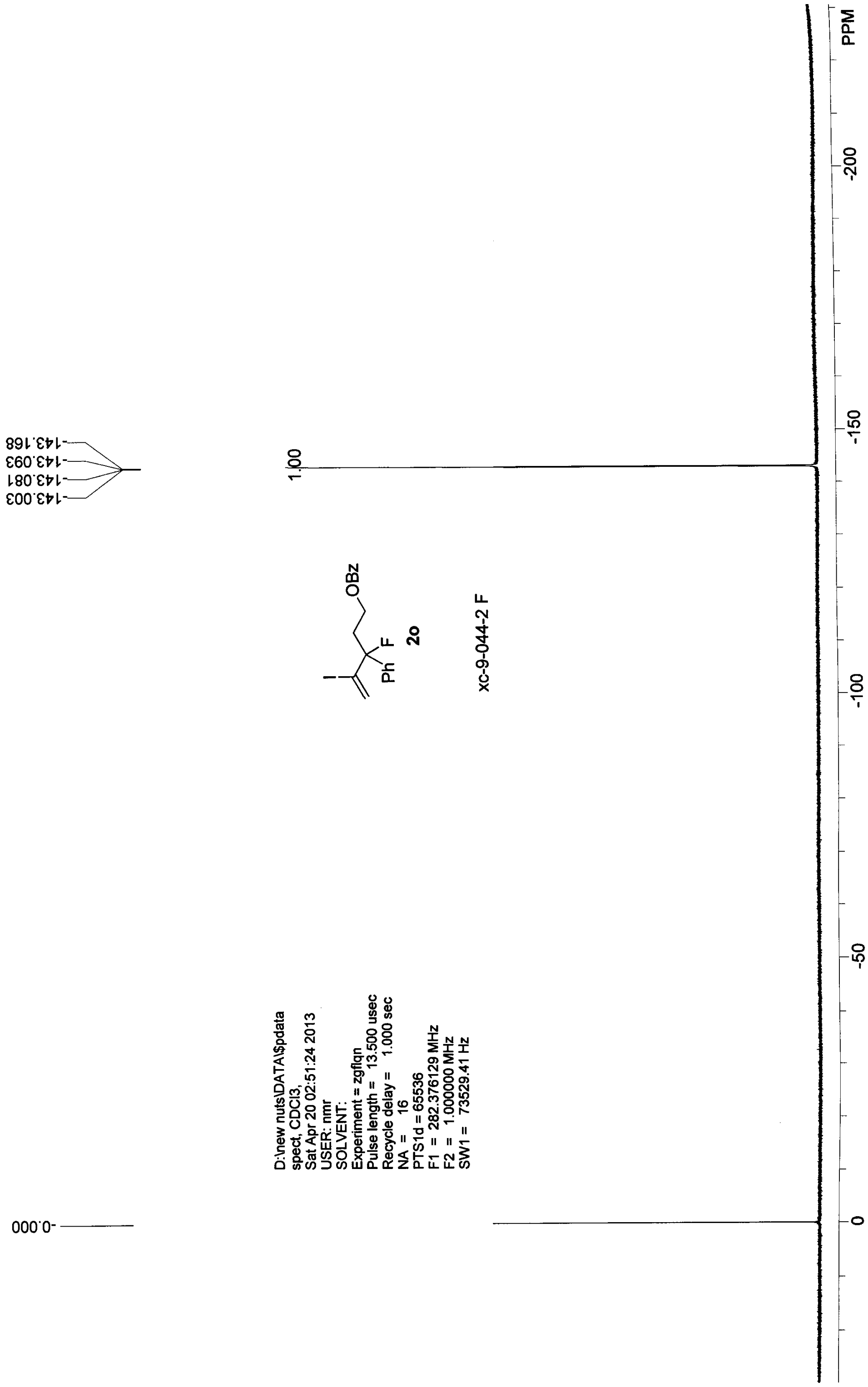

0.000
1.602
2.635
2.656
2.661
2.683
2.703
2.710
2.731
2.765
2.785
2.792
2.812
2.828
2.848
2.855
2.875
2.897
2.918
2.923
2.944
2.965
2.971
2.991
4.337
4.358
4.364
4.374
4.385
4.396
4.401
4.423
4.437
4.457
4.464
4.475
4.484
4.494
4.502
4.522
6.078
6.592
7.254
7.317
7.321
7.326
7.339
7.364
7.390
7.410
7.433
7.454
7.522
7.546
7.571
7.936
OBz
Ph
F
2o
xc-9-044-2
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 02:48:50 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.09
2.04
0.96
1.00
8.34
1.99
PPM
0
2
4
6
8

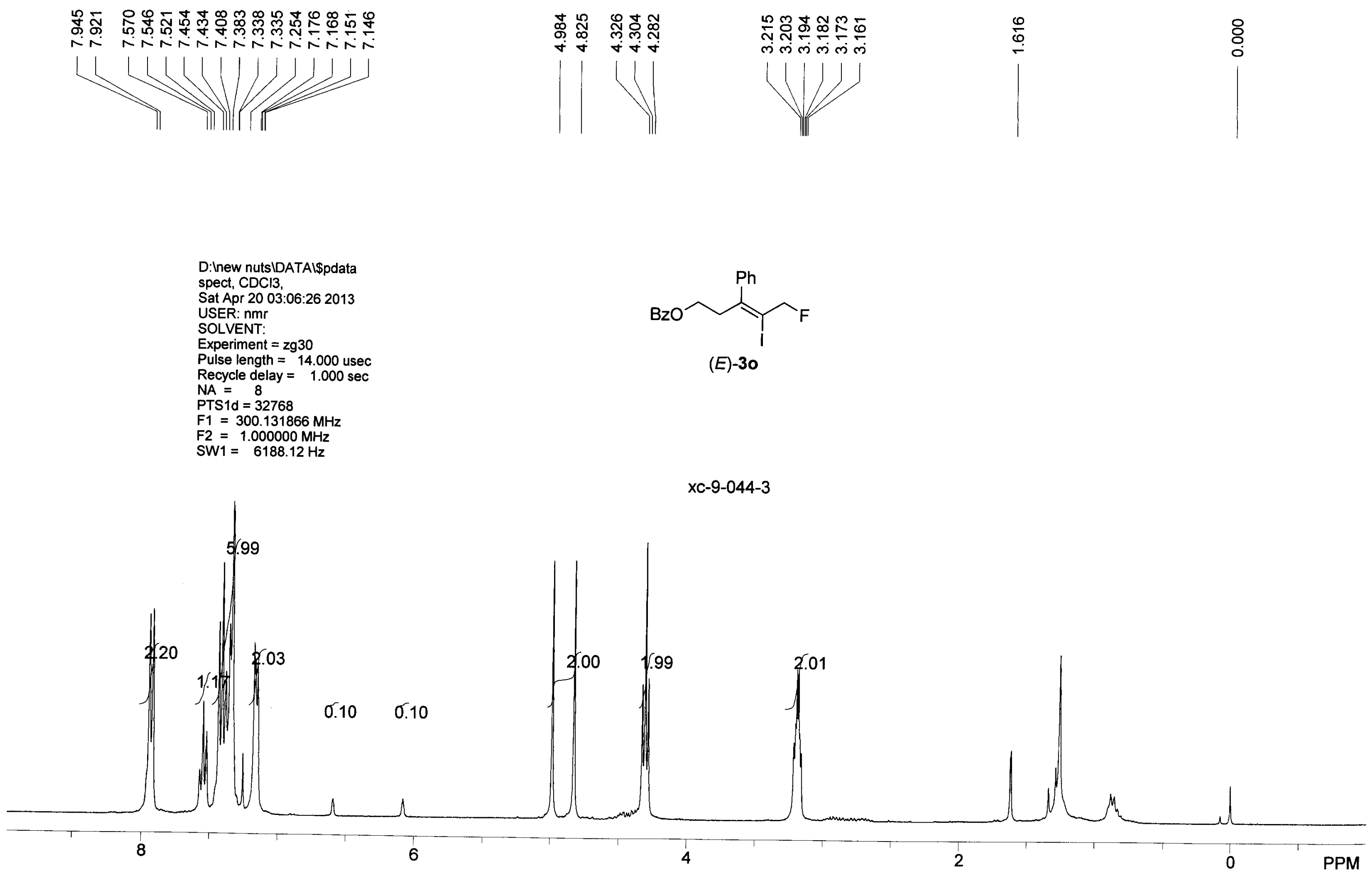


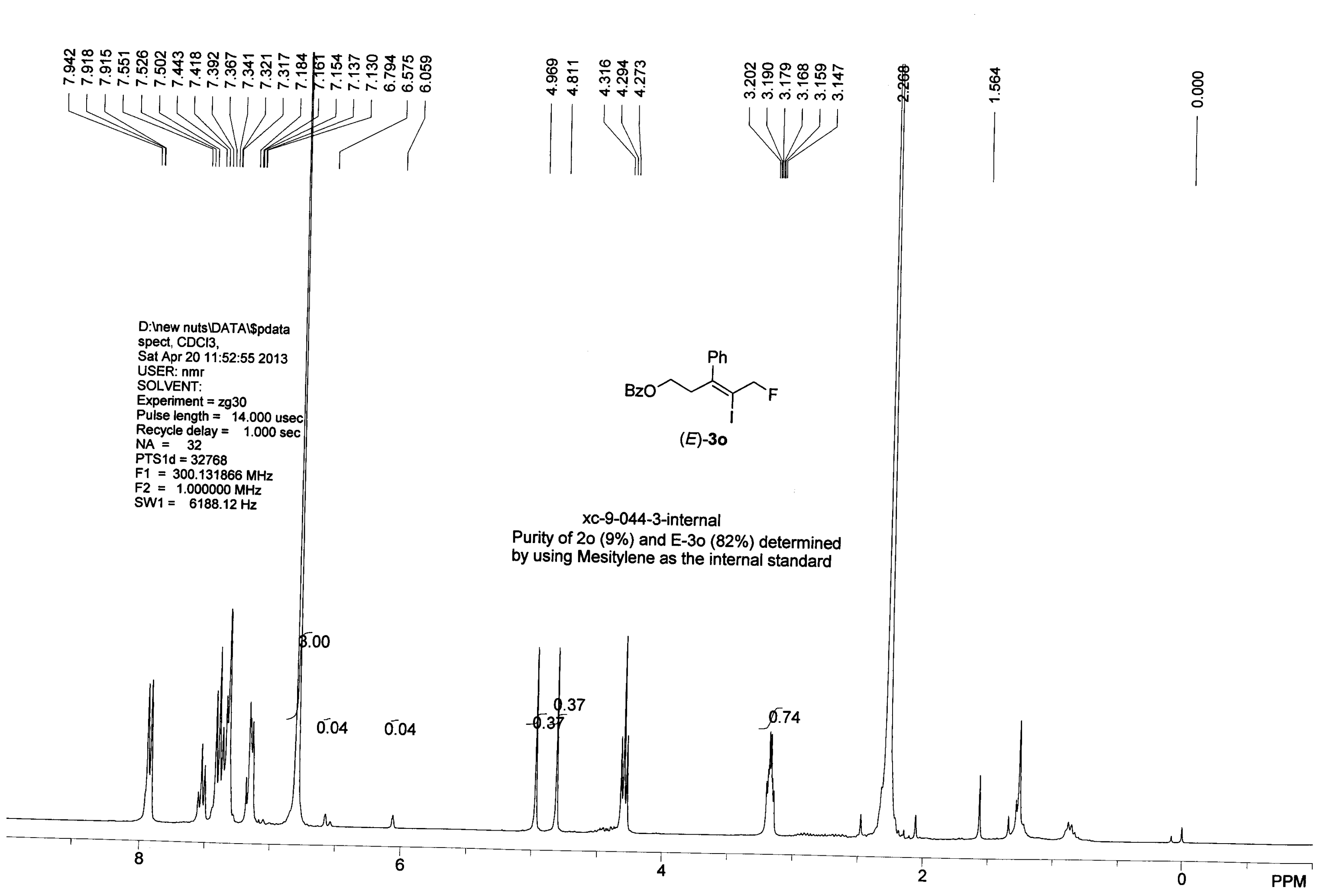
2o

xc-9-044-2 C

D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun Apr 21 03:42:33 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2108
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

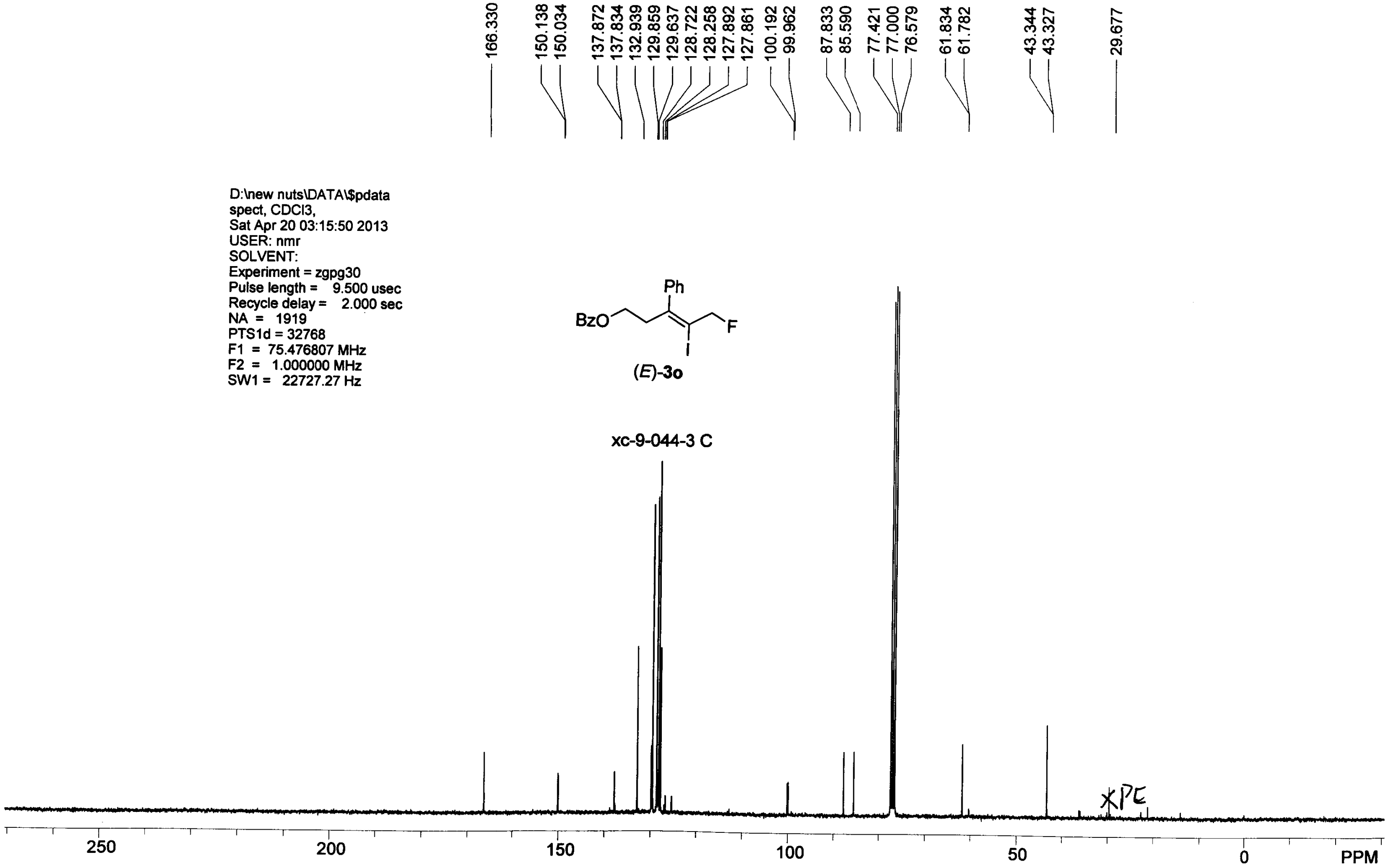

-143.168
-143.093
-143.081
-143.003
-0.000
1.00
OBz
Ph
F
2o
xc-9-044-2 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 02:51:24 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
PPM
-200
-150
-100
-50
0


(E)-**3o**
7.945
7.921
7.570
7.546
7.521
7.454
7.434
7.408
7.383
7.338
7.335
7.254
7.176
7.168
7.151
7.146
4.984
4.825
4.326
4.304
4.282
3.215
3.203
3.194
3.182
3.173
3.161
1.616
0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 03:06:26 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Ph
BzO
F
I
xc-9-044-3
5.99
2.20
1.17
2.03
0.10
0.10
2.00
1.99
2.01
8
6
4
2
0
PPM



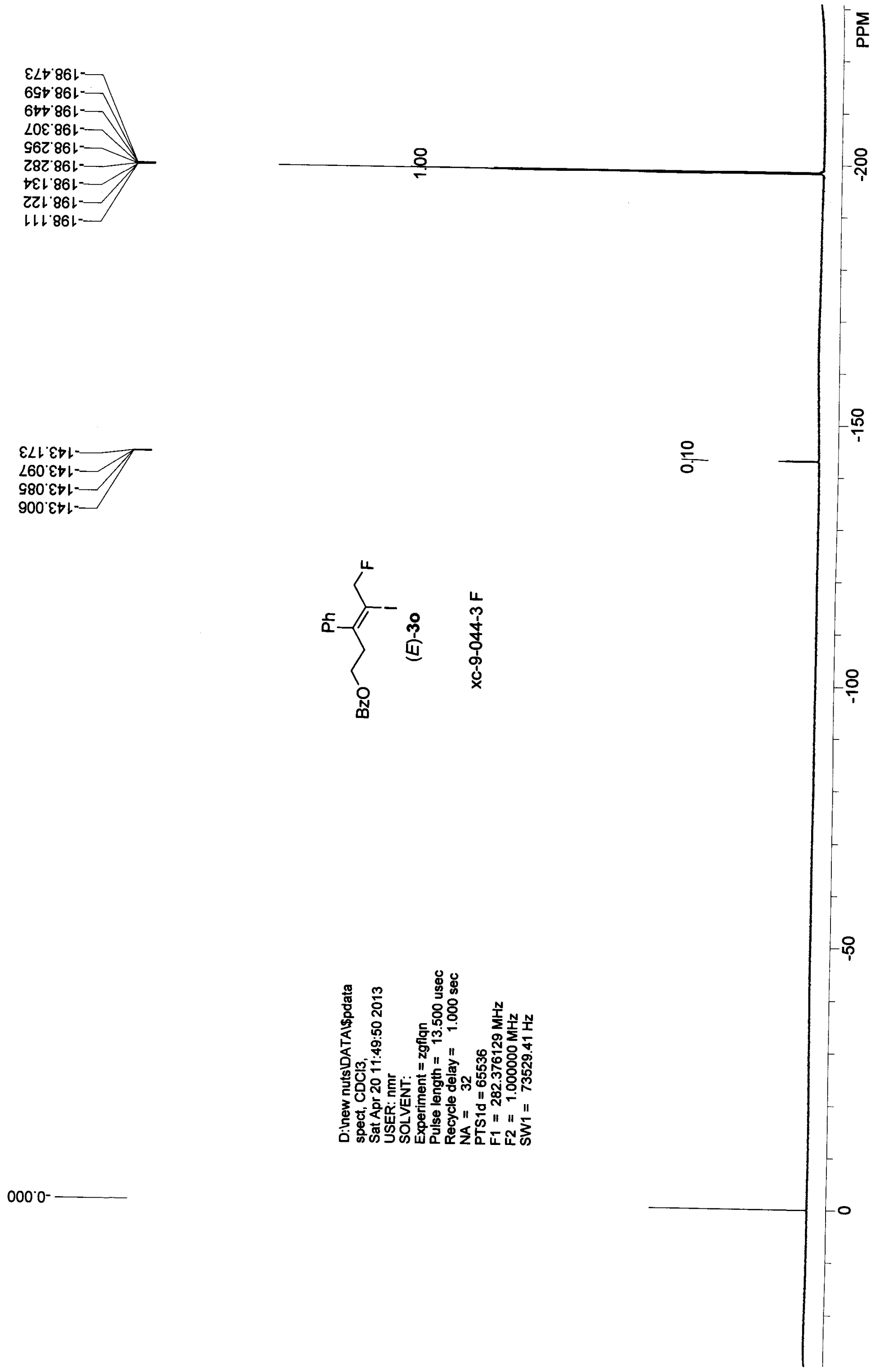
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 11:52:55 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

xc-9-044-3-internal
Purity of 2o (9%) and E-3o (82%) determined
by using Mesitylene as the internal standard



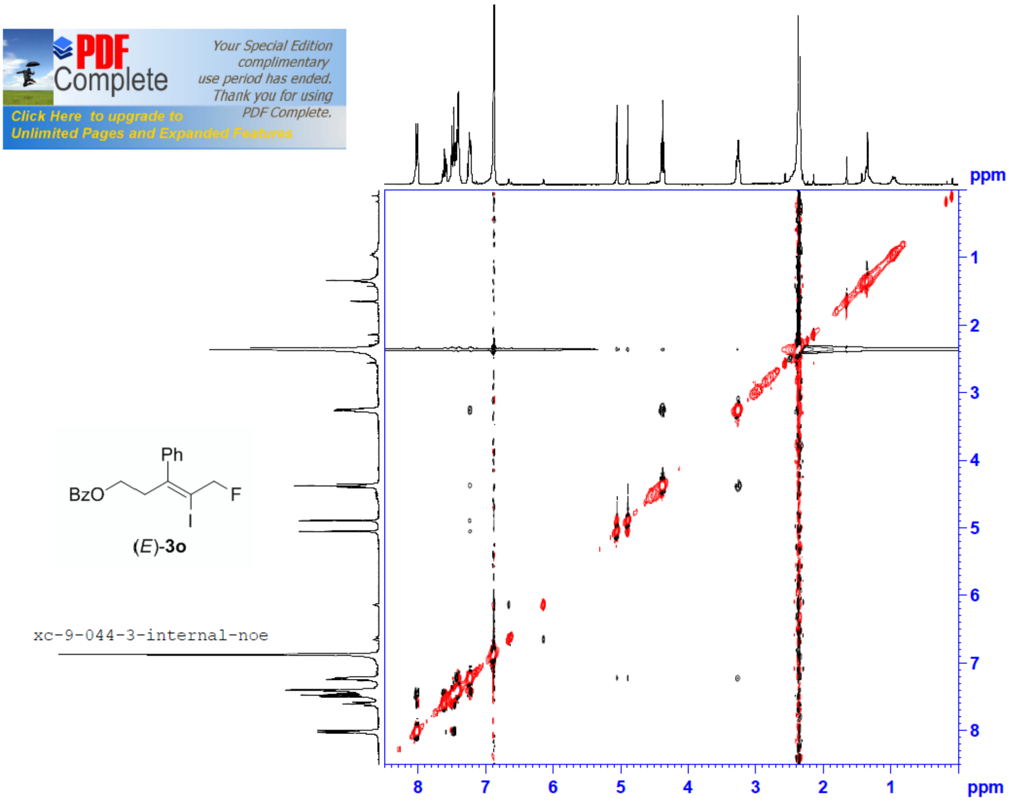
(E)-3o

166.330
150.138
150.034
137.872
137.834
132.939
129.859
129.637
128.722
128.258
127.892
127.861
100.192
99.962
87.833
85.590
77.421
77.000
76.579
61.834
61.782
43.344
43.327
29.677
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 03:15:50 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1919
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Ph
BzO
F
I
xc-9-044-3 C
X PE
250
200
150
100
50
0
PPM



```
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Apr 20 11:49:50 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length =  13.500 usec
Recycle delay =  1.000 sec
NA =   32
PTS1d = 65536
F1 =  282.376129 MHz
F2 =  1.000000 MHz
SW1 =  73529.41 Hz
```

Use Mesitylene as the internal standard



(*E*)-**3o**