Electronic Supplementary Material (ESI) for Chemical Communications.

TfOH-catalyzed domino cycloisomerization/hydrolytic defluorination of 2,3-allenyl perfluoroalkyl ketones

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Supporting Information

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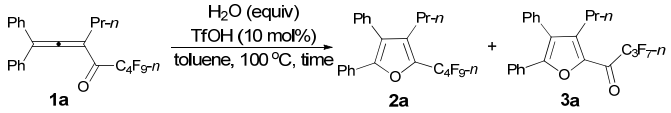
General： Perfluoroalkyl allenones were prepared according to the literature procedure.[1] Toluene was distilled from sodium wire/benzophenone. TfOH (Alfa) was stored in a glove box, and transferred with a micro-syringe. Other commercially available chemicals were used without additional purification. The reactions were performed under an atmosphere of nitrogen using standard Schlenk tubes unless otherwise stated. Petroleum ether with a boiling point ranges from 30 to 60 ^{o}C was used. Flash-column chromatography was carried out on silica gel H (10–40 μ). ^{1}H NMR spectra (300 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 taken with an HP 5989A system. High-resolution mass spectrometry was determined with a Finnigan MAT 8430 or Bruker APEXIII instrument. The structure of **3d-3f**, **3k** and **3m'** were established by the NOE study.

Table S1. Optimization on the selective synthesis of furyl perfluoropropyl ketone **3a**. [a]



Entry	H_2O (equiv)	Time (h)	NMR Yield (%)[b]	
			2a	**3a**
1	0	19.5	11	73
2	1.0	2.5	0	98
3[c]	1.0	17	88	12
4	1.4	2.5	0	96
5	1.8	2.5	0	99
6	2.8	3	0	97
7	5.6	3	0	94
8	10	2.5	0	93
9[d]	100	19.5	10	0

[a] The reactions were conducted on 0.2 mmol scale in toluene (2.0 mL). [b] Determined by the ^{1}H NMR spectra analysis with CH_2Br_2 as the internal standard. [c] The reaction was conducted at 60 °C with 5 mol% of TfOH. [d] Recovery of **1a** was 90%.

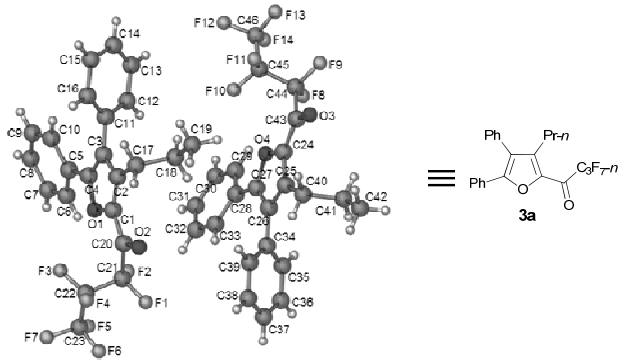
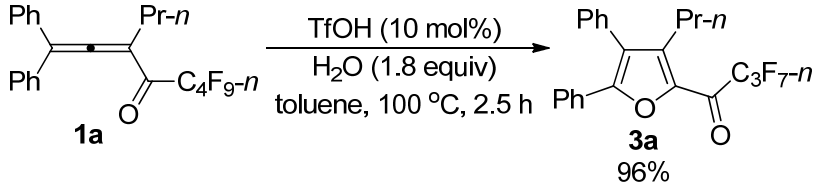


Figure S1. ORTEP representation of **3a**.

1. TfOH-catalyzed domino cycloisomerization/hydrolytic defluorination reaction of allenyl perfluorobutyl ketones 1a-1l.

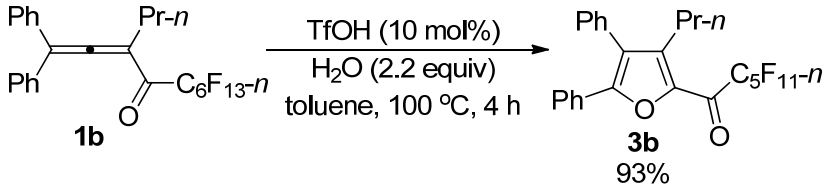
(1) Synthesis of (4,5-diphenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3a**) (xc-6-008)



Typical Procedure I: To a dried Schlenk tube were added 1,1-diphenylhexa-1,2-dien-3-yl perfluorobutyl ketone **1a** (0.0960 g, 0.20 mmol)/rinsed with toluene (2 mL), H_2O (6.7 μL, *d* = 1.00 g/mL, 0.0067 g, 0.37 mmol), and TfOH (1.8 μL, *d* = 1.695 g/mL, 0.0031 g, 0.02 mmol) at room temperature under a nitrogen atmosphere. The resulting reaction mixture was placed in a pre-heated oil bath of 100 ^{o}C and strirried for 2.5 h as monitored by TLC. After being cooled to the room temperature, the crude reaction mixture was transferred to a round bottom flask. After removing the solvent via rotary evaporation, column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100/1) afforded **3a** (0.0882 g, 96%) as a solid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56-7.40 (m, 5 H, ArH), 7.36-7.22 (m, 5 H, ArH), 2.70 (t, *J* = 7.8 Hz, 2 H, CH_2), 1.60-1.39 (m, 2 H, CH_2), 0.86 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5 (t, *J* = 25.8 Hz), 154.5, 146.0, 143.3, 131.4, 130.0, 129.7, 129.2, 128.9, 128.7, 128.5, 127.0, 126.8, 26.6, 22.6, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -80.5 (t, *J* = 8.7 Hz, 3 F), -117.4 (q, *J* = 9.4 Hz, 2 F), -125.7~-125.9 (m, 2 F).

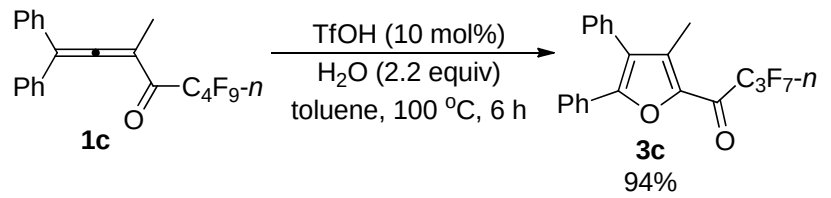
The following compounds **3b-3e** were prepared following **Typical Procedure I**.

(2) Synthesis of (4,5-diphenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropentyl ketone (**3b**) (xc-6-125)



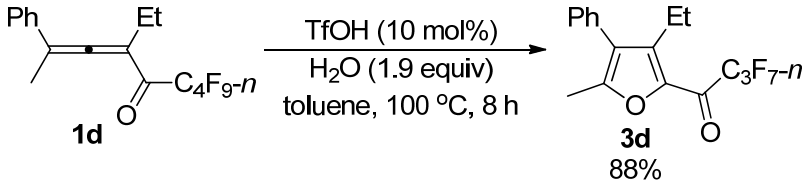
The reaction of 1,1-diphenylhexa-1,2-dien-3-yl perfluorohexyl ketone **1b** (0.2925 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol), and H_2O (20 μL, *d* = 1.0 g/mL, 0.0200 g, 1.1 mmol) in toluene (6 mL) at 100 °C for 4 h afforded **3b** (0.2604 g, 93%) (petroleum ether/ethyl acetate = 100/1) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54-7.22 (m, 10 H, ArH), 2.69 (t, *J* = 7.8 Hz, 2 H, CH_2), 1.56-1.41 (m, 2 H, CH_2), 0.86 (t, *J*= 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5 (t, *J* = 25.8 Hz), 154.5, 146.0, 143.3, 131.4, 130.0, 129.7, 129.2, 128.9, 128.7, 128.5, 127.0, 126.8, 26.6, 22.6, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.2 (t, *J* = 9.9 Hz, 3 F), -117.0 (t, *J* = 13.8 Hz), -121.8~-122.2 (m, 2 F), -122.6~-123.1 (m, 2 F), -126.4~-127.0 (m, 2 F); IR (neat) ν (cm^{-1}) 3068, 3034, 2965, 2934, 2875, 1682, 1569, 1531, 1477, 1446, 1414, 1381, 1348, 1239, 1087, 1074, 1057, 1029; MS (70 ev, EI) *m/z* (%) 558 (M^+, 14.93), 289 (100); HRMS calcd for $C_{25}H_{17}F_{11}O_2$ (M^+): 558.1053, Found: 558.1060.

(3) Synthesis of (4,5-diphenyl-3-methylfuran-2-yl) *n*-perfluoropropyl ketone (**3c**) (xc-6-165)



The reaction of 2-ethyl-4,4-diphenylbuta-2,3-dien-2-yl perfluorobutyl ketone **1c** (0.2265 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol), and H_2O (20 μL, *d* = 1.0 g/mL, 0.0200 g, 1.1 mmol) in toluene (6 mL) at 100 ^oC for 6 h afforded **3c** (0.2017 g, 94%) (petroleum ether/ethyl acetate = 100/1) as a solid: m.p. 55.3-56.7 ^oC (*n*-hexane/ethyl acetate); ^1H NMR (300 MHz, $CDCl_3$) δ 7.59-7.38 (m, 5 H, ArH), 7.36-7.22 (m, 5 H, ArH), 2.33 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.9 (t, *J* = 26.2 Hz), 154.5, 143.6, 141.1, 131.2, 129.9, 129.8, 129.2, 128.9, 128.7, 128.5, 127.2, 126.9, 10.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -80.9 (t, *J* = 8.9 Hz, 3 F), -117.7~-118.2 (m, 2 F), -126.0~-126.3 (m, 2 F); IR (KBr) ν (cm^{-1}) 3066, 3030, 1682, 1573, 1537, 1477, 1446, 1410, 1380, 1353, 1329, 1231, 1118, 1075; MS (70 ev, EI) *m/z* (%) 430 (M^+, 35.31), 261 (100); Elemental analysis calcd for $C_{21}H_{13}F_7O_2$: C, 58.61; H, 3.05; Found: C, 58.26; H, 3.41.

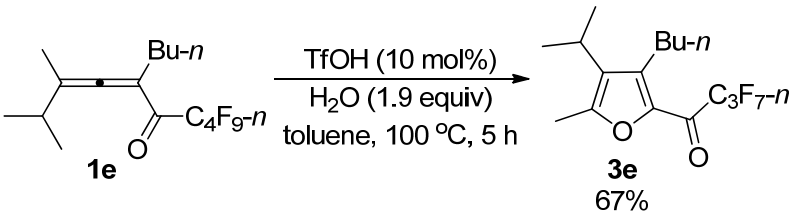
(4) Synthesis of (3-ethyl-5-methyl-4-phenylfuran-2-yl) *n*-perfluoropropyl ketone (**3d**) (xc-8-089)



The reaction of 2-phenylhexa-2,3-dien-4-yl perfluorobutyl ketone **1d** (0.2018 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.050 mmol), and H_2O (17 μL, *d* = 1.0 g/mL, 0.0170 g, 0.94 mmol) in toluene (5 mL) at 100 ^oC for 8 h afforded **3d** (0.1672 g, 88%) (petroleum ether/ethyl acetate = 300/1) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.52-7.36 (m, 3 H, ArH), 7.30-7.19 (m, 2 H, ArH), 2.78 (q, *J* = 7.5 Hz, 2 H, CH_2), 2.37 (s, 3 H, CH_3), 1.10 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR

(75 MHz, $CDCl_3$) δ 170.1 (t, *J* = 26.1 Hz), 156.9, 146.3, 143.4, 130.8, 129.5, 128.8, 128.0, 127.1, 18.3, 13.5, 13.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 8.9 Hz, 3 F), -117.9 (q, *J* = 9.1 Hz, 2 F), -126.2~-126.6 (m, 2 F); IR (neat) ν (cm^{-1}) 3061, 2978, 2938, 2878, 1674, 1612, 1591, 1533, 1492, 1463, 1442, 1424, 1376, 1337, 1317, 1231, 1119, 1081, 1013; MS (70 ev, EI) *m/z* (%) 382 (M^+, 33.00), 213 (100); HRMS calcd for $C_{17}H_{13}F_7O_2$ (M^+): 382.0804, Found: 382.0803.

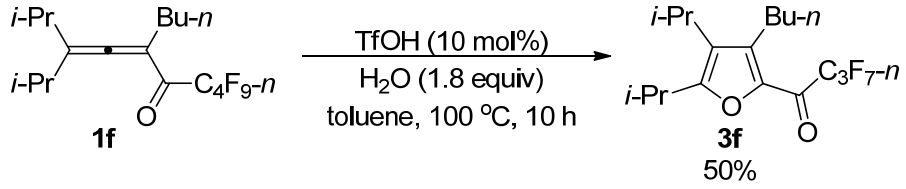
(5) Synthesis of (3-(*n*-butyl)-5-methyl-4-(*i*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3e**) (xc-8-035)



The reaction of 2,3-dimethylnona-3,4-dien-5-yl perfluorobutyl ketone **1e** (0.1995 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.050 mmol), and H_2O (16.7 μL, *d* = 1.0 g/mL, 0.0167 g, 0.93 mmol) in toluene (5 mL) at 100 ^oC for 5 h afforded **3e** (0.1263 g, 67%) (petroleum ether/ethyl acetate = 100/1) as a liquid: ^1H NMR (300 MHz, $CDCl_3$) δ 2.96-2.83 (m, 1 H, CH), 2.80 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.41 (s, 3 H, CH_3), 1.57-1.34 (m, 4 H, 2 × CH_2), 1.27 (d, *J* = 7.2 Hz, 6 H, 2 × CH_3) 0.95 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.5 (t, *J* = 25.2 Hz), 156.0, 145.5, 143.0, 130.0, 31.8, 24.7, 23.7, 22.9, 22.0, 14.0, 13.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.1 (t, *J* = 9.0 Hz, 3 F), -117.7 (q, *J* = 8.8 Hz, 2 F), -126.6~-126.2 (m, 2 F); IR (neat) ν (cm^{-1}) 2965, 2934, 2876, 1673, 1590, 1523, 1456, 1377, 1342, 1313, 1230, 1119, 1084, 1033; MS (70 ev, EI) *m/z* (%) 376 (M^+, 14.80), 137 (100);

HRMS calcd for $C_{16}H_{19}F_7O_2$ (M^+): 376.1273, Found: 376.1271.

(6) Synthesis of (3-(*n*-butyl)-4,5-di(*i*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3f**) (xc-10-142)

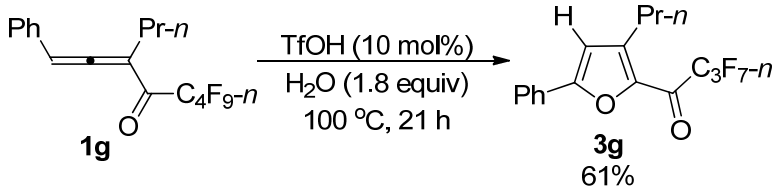


Typical Procedure II: To a dried Schlenk tube were added 2-methyl-3-(*i*-propyl)nona-3,4-dien-5-yl perfluorobutyl ketone **1f** (0.2138 g, 0.50 mmol)/rinsed with toluene (5 mL), TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.050 mmol), and H_2O (17.0 μL, *d* = 1.0 g/mL, 0.0170 g, 0.94 mmol) at room temperature under a nitrogen atmosphere. The resulting reaction mixture was placed in a pre-heated oil bath of 100 ^oC and strirried for 10 h as monitored by TLC. After being cooled to the room temperature, the crude reaction mixture was transferred to a round bottom flask. After removing the solvent via rotary evaporation, column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 500/1) afforded **3f** (0.1015 g, 50%) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 3.27-3.10 (heptet, *J* = 6.8 Hz, 1 H, CH), 3.00-2.85 (heptet, *J* = 7.1 Hz, 1 H, CH), 2.84-2.75 (m, 2 H, CH_2), 1.57-1.37 (m, 4 H, 2 × CH_2), 1.28 (d, *J* = 6.6 Hz, 6 H, 2 × CH_3), 1.27 (d, *J* = 7.2 Hz, 6 H, 2 × CH_3), 0.95 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.4 (t, *J* = 25.1 Hz), 164.1, 145.3, 142.9, 128.3, 31.8, 27.8, 24.9, 23.8, 23.0, 22.6, 20.8, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -80.8 (t, *J* = 9.2 Hz, 3 F), -117.6 (q, *J* = 9.3 Hz, 2 F), -125.9~-126.1 (m, 2 F); IR (neat) ν (cm^{-1}) 2968, 2936, 2876, 1673, 1584, 1524, 1455, 1385, 1368, 1344, 1308, 1230, 1119, 1086, 1060, 1028; MS (70 ev, EI) *m/z* (%) 404

(M^+, 22.64), 165 (100); HRMS calcd for $C_{18}H_{23}F_7O_2$ (M^+): 404.1586, Found: 404.1590.

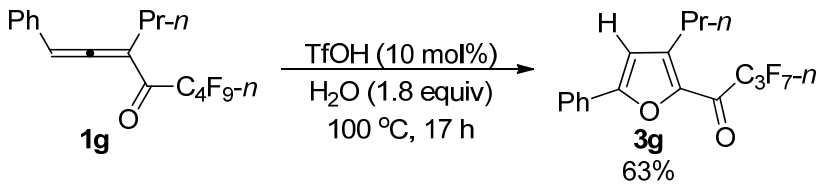
The following compounds **3g-3l** were prepared following **Typical Procedure II**.

(7) Synthesis of (5-phenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3g**) (xc-13-149)



The reaction of 1-phenylhexa-1,2-dien-3-yl *n*-perfluorobutyl ketone **1g** (0.2021 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol), and H_2O (16.2 μL, *d* = 1.00 g/mL, 0.0162 g, 0.90 mmol) in toluene (5 mL) at 100 ^oC for 21 h afforded **3g** (0.1168 g, 61%) (petroleum ether/ethyl acetate = 500/1) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.83-7.73 (m, 2 H, ArH), 7.52-7.39 (m, 3 H, ArH), 6.83 (s, 1 H, =CH), 2.91 (t, *J* = 7.7 Hz, 2 H, CH_2), 1.78-1.62 (m, 2 H, CH_2), 1.02 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4 (t, *J* = 26.1 Hz), 159.0, 146.8, 143.9, 130.2, 129.1, 128.5, 125.5, 110.3, 28.4, 22.3, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.0 Hz, 3 F), -117.9~-118.2 (m, 2 F), -126.2~-126.4 (m, 2 F); IR (neat) ν (cm^{-1}) 3071, 3040, 2966, 2936, 2877, 1667, 1588, 1573, 1520, 1475, 1454, 1410, 1352, 1316, 1269, 1231, 1119, 1088, 1072, 1028; MS (70 ev, EI) *m/z* (%) 382 (M^+, 36.86), 213 (100); HRMS calcd for $C_{17}H_{13}F_7O_2$ (M^+): 382.0804, Found: 382.0812.

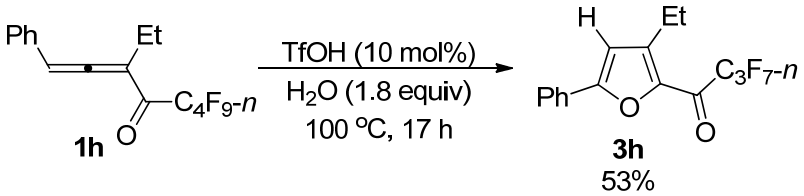
(8) Gram scale reaction for the synthesis of (5-phenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3g**) (xc-13-158)



The reaction of 1-phenylhexa-1,2-dien-3-yl *n*-perfluorobutyl ketone **1g** (1.2123 g, 3.00 mmol) with TfOH (99%, 27 μL, *d* = 1.695 g/mL, 0.0453 g, 0.30 mmol) and H_2O (97.2 μL, *d* = 1.0 g/mL, 0.0972 g, 5.40 mmol) at 100 °C for 17 h to afford **3g** (0.7228 g, 63%) (petroleum ether/ethyl acetate = 500/1) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83-7.74 (m, 2 H, ArH), 7.51-7.38 (m, 3 H, ArH), 6.82 (s, 1 H, =CH), 2.90 (t, *J* = 7.7 Hz, 2 H, CH_2), 1.79-1.62 (m, 2 H, CH_2), 1.01 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.4 (t, *J* = 25.8 Hz), 159.1, 146.8, 143.9, 130.2, 129.1, 128.5, 125.4, 110.3, 28.4, 22.3, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.0 Hz, 3 F), -117.9~-118.1 (m, 2 F), -126.2~-126.4 (s, 2 F).

Besides, we have also got an unidentified compound, which is studying in our laboratory.

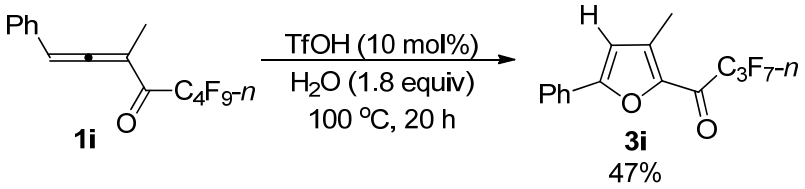
(9) Synthesis of (3-ethyl-5-phenylfuran-2-yl) *n*-perfluoropropyl ketone (**3h**) (xc-13-156)



The reaction of 1-phenylpenta-1,2-dien-3-yl perfluorobutyl ketone **1h** (0.1946 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol) and H_2O (16.2 μL, *d* = 1.0 g/mL, 0.0162 g, 0.90 mmol) at 100 °C for 17 h to afford **3h** (0.0966 g, 53%) (petroleum ether/ethyl acetate = 500/1) as a solid: m.p. 40.2-41.2 °C

(*n*-hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83-7.74 (m, 2 H, ArH), 7.52-7.39 (m, 3 H, ArH), 6.85 (s, 1 H, =CH), 2.95 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.29 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3 (t, *J* = 26.2 Hz), 159.2, 148.4, 143.5, 130.3, 129.1, 128.5, 125.4, 109.8, 20.0, 13.1; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.0 Hz, 3 F), -117.9~-118.2 (m, 2 F), -126.2~-126.4 (m, 2 F); IR (KBr) ν (cm^{-1}) 3071, 3039, 2977, 2939, 2884, 1671, 1587, 1572, 1523, 1475, 1464, 1453, 1411, 1353, 1332, 1271, 1230, 1123, 1083; MS (70 ev, EI) *m/z* (%) 368 (M^+, 26.51), 199 (100); Elemental analysis calcd for $C_{16}H_{11}F_7O_2$: C, 52.19; H, 3.01; Found: C, 52.46; H, 3.06.

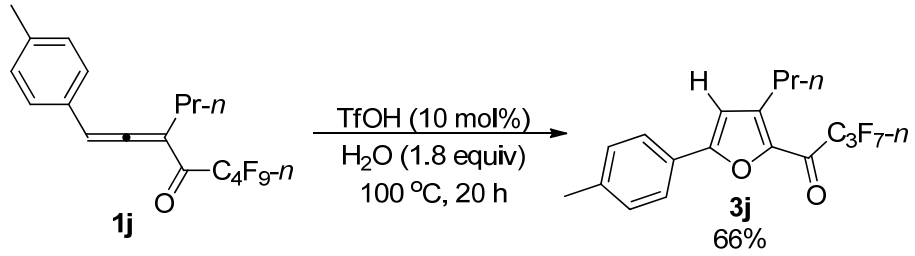
(10) Synthesis of (3-methyl-5-phenylfuran-2-yl) *n*-perfluoropropyl ketone (**3i**) (xc-13-151)



The reaction of 1-phenylbuta-1,2-dien-3-yl perfluorobutyl ketone **1i** (0.1877 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol) and H_2O (16.2 μL, *d* = 1.0 g/mL, 0.0162 g, 0.90 mmol) at 100 °C for 20 h to afford **3i** (0.0873 g, purity: 94.2% as determined by using dibromomethane as the internal standard, 47% yield) (petroleum ether/ethyl acetate = 500/1) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81-7.73 (m, 2 H, ArH), 7.52-7.40 (m, 3 H, ArH), 6.79 (s, 1 H, =CH), 2.51 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5 (t, *J* = 25.8 Hz), 158.9, 144.2, 141.9, 130.3, 129.1, 128.4, 125.4, 111.6, 12.6; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 8.9 Hz, 3 F), -118.0~-118.2 (m, 2 F), -126.2~-126.4 (m, 2 F); IR (neat) ν (cm^{-1}) 3071, 2928,

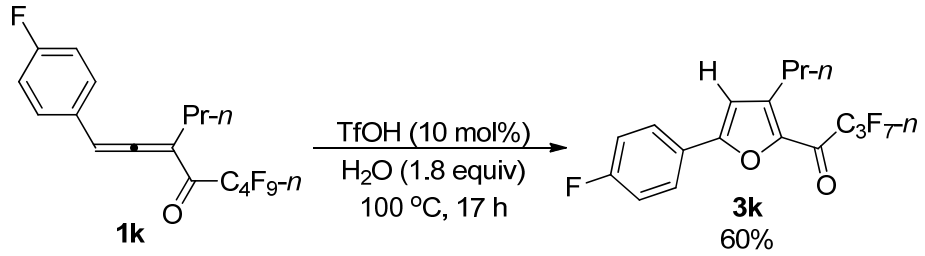
1672, 1574, 1524, 1475, 1453, 1407, 1379, 1352, 1292, 1270, 1230, 1118; MS (70 ev, EI) *m/z* (%) 354 (M^+, 76.54), 185 (100); HRMS calcd for $C_{15}H_9F_7O_2$ (M^+): 354.0491, Found: 354.0493.

(11) Synthesis of (5-(4'-methylphenyl)-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3j**) (xc-13-152)



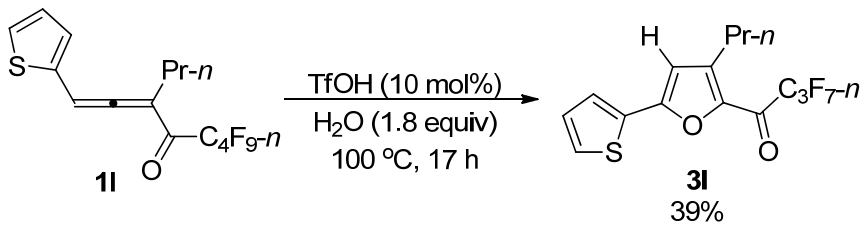
The reaction of 1-(4'-methylphenyl)hexa-1,2-dien-3-yl perfluorobutyl ketone **1j** (0.2085 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol) and H_2O (16.2 μL, *d* = 1.00 g/mL, 0.0162 g, 0.90 mmol) at 100 ^oC for 20 h to afford **3j** (0.1352 g, purity: 96.6% as determined by using dibromomethane as the internal standard, 66% yield) (petroleum ether/ethyl acetate = 500/1) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.72-7.63 (m, 2 H, ArH), 7.31-7.24 (m, 2 H, ArH), 6.78 (s, 1 H, =CH), 2.90 (t, *J* = 7.7 Hz, 2 H, CH_2), 2.41 (s, 3 H, CH_3), 1.78-1.62 (m, 2 H, CH_2), 1.01 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1 (t, *J* = 25.6 Hz), 159.4, 147.0, 143.7, 140.7, 129.8, 125.8, 125.5, 109.8, 28.4, 22.3, 21.5, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.0 Hz, 3 F), -117.9~118.1 (m, 2 F), -126.3~-126.4 (s, 2 F); IR (neat) ν (cm^{-1}) 3031, 2966, 2934, 2876, 1668, 1615, 1586, 1574, 1532, 1482, 1401, 1351, 1317, 1293, 1267, 1231, 1119, 1088, 1066; MS (70 ev, EI) *m/z* (%) 396 (M^+, 54.73), 227 (100); Elemental analysis calcd for $C_{18}H_{15}O_2F_7$: C, 54.55; H, 3.82; Found: C, 54.93; H, 3.80.

(12) Synthesis of (5-(4'-fluorophenyl)-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone (**3k**) (xc-13-153)



The reaction of 1-(4'-fluorophenyl)hexa-1,2-dien-3-yl perfluorobutyl ketone **1k** (0.2107 g, 0.50 mmol) with TfOH (99%, 4.5 μL, *d* = 1.695 g/mL, 0.0076 g, 0.05 mmol) and H_2O (16.2 μL, *d* = 1.0 g/mL, 0.0162 g, 0.90 mmol) at 100 ^oC for 17 h to afford **3k** (0.1263 g, purity: 95.1% as determined by using dibromomethane as the internal standard, 60% yield) (petroleum ether/ethyl acetate = 500/1) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.82-7.72 (m, 2 H, ArH), 7.21-7.10 (m, 2 H, ArH), 6.78 (s, 1 H, =CH), 2.90 (t, *J* = 7.7 Hz, 2 H, CH_2), 1.78-1.62 (m, 2 H, CH_2), 1.02 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3 (t, *J* = 25.7 Hz), 163.8 (d, *J* = 250.3 Hz), 158.0, 146.9, 143.8, 127.5 (d, *J* = 9.2 Hz), 124.9 (d, *J* = 3.2 Hz), 116.4 (t, *J* = 22.3 Hz), 110.1, 28.4, 22.3, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ 81.0 (t, *J* = 9.0 Hz, 3 F), -109.3~-109.5 (m, 1 F), -117.9~-118.2 (m, 2 F), -126.2~-126.4 (m, 2 F); IR (neat) ν (cm^{-1}) 2967, 2936, 2877, 1673, 1608, 1578, 1533, 1482, 1431, 1352, 1307, 1295, 1267, 1235, 1159, 1119, 1102; MS (70 ev, EI) *m/z* (%) 400 (M^+, 37.50), 231 (100); HRMS calcd for $C_{17}H_{12}F_8O_2$ (M^+): 400.0710, Found: 400.0710.

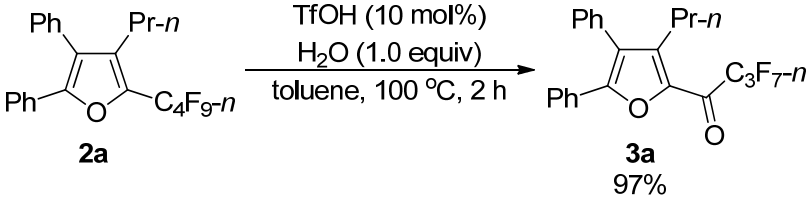
(13) Synthesis of (3-(*n*-propyl)-5-(2'-thienyl)furan-2-yl) *n*-perfluoropropyl ketone (**3l**) (xc-13-154)



The reaction of 1-(2'-thienyl)hexa-1,2-dien-3-yl perfluorobutyl ketone **1l** (0.0813 g, 0.20 mmol) with TfOH (99%, 1.8 μL, *d* = 1.695 g/mL, 0.0030 g, 0.02 mmol) and H_2O (6.5 μL, *d* = 1.0 g/mL, 0.0065 g, 0.36 mmol) at 100 ^oC for 17 h to afford **3l** (0.0298 g, 39%) (petroleum ether/ethyl acetate = 500/1) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.52 (dd, J_1 = 3.8 Hz, J_2 = 1.1 Hz, 1 H, ArH), 7.45 (dd, J_1 = 5.1 Hz, J_2 = 1.2 Hz, 1 H, ArH), 7.13 (dd, J_1 = 5.0 Hz, J_2 = 3.8 Hz, 1 H, ArH), 6.66 (s, 1 H, =CH), 2.89 (t, *J* = 7.8 Hz, 2 H, CH_2), 1.78-1.60 (m, 2 H, CH_2), 1.01 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1 (t, *J* = 26.1 Hz), 154.4, 147.0, 143.3, 131.4, 128.37, 128.35, 126.9, 110.1, 28.4, 22.2, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.1 (t, *J* = 8.9 Hz, 3 F), -117.9~-118.2 (m, 2 F), -126.3~-126.4 (m, 2 F); IR (neat) ν (cm^{-1}) 2968, 2934, 2876, 1667, 1588, 1538, 1479, 1441, 1418, 1351, 1266, 1216, 1119, 1086, 1053, 1031; MS (70 ev, EI) *m/z* (%) 388 (M^+, 44.60), 219 (100); HRMS calcd for $C_{15}H_{11}F_7O_2S$ (M^+): 388.0368, Found: 388.0372.

2. Mechanistic Studies.

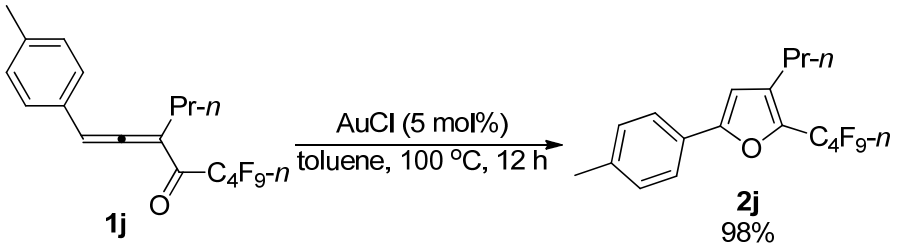
(1) TfOH-catalyzed transformation of 2-perfluorobutyl furan **2a** to (4,5-diphenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone **3a** (xc-4-169)



Typical Procedure III: To a dried Schlenk tube were sequentially added furan **2a** (0.0958 g, 0.20 mmol)/rinsed with toluene (1 mL), TfOH (1.8 μL, *d* = 1.695 g/mL, 0.0031 g, 0.02 mmol), and H_2O (3.6 μL, *d* = 1.0 g/mL, 0.0036 g, 0.2 mmol) in toluene (1 mL) at room temperature under a nitrogen atmosphere. The resulting reaction mixture was placed in a pre-heated oil bath of 100 ^oC and strirried for 2 h as monitored by TLC. After being cooled to the room temperature, the crude reaction mixture was transferred to a round bottom flask. After removing the solvent via rotary evaporation, column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100/1) afforded **3a** (0.0898 g, 97%) as a solid: m.p. 54.4-56.5 ^oC (*n*-hexane/ethyl acetate); ^1H NMR (300 MHz, $CDCl_3$) δ 7.59-7.39 (m, 5 H, ArH), 7.39-7.16 (m, 5 H, ArH), 2.70 (t, *J* = 7.7 Hz, 2 H, CH_2), 1.61-1.35 (m, 2 H, CH_2), 0.86 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.6 (t, *J* = 26.0 Hz), 154.5, 145.9, 143.4, 131.5, 130.0, 129.7, 129.2, 129.0, 128.7, 128.5, 127.0, 126.9, 26.6, 22.6, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -80.5 (t, *J* = 8.9 Hz, 3 F), -117.3 (q, *J* = 9.4 Hz, 2 F), -125.6~-125.8 (m, 2 F); IR (neat) ν (cm^{-1}) 3067, 2964, 2933, 2874, 1673, 1570, 1531, 1477, 1447, 1414, 1329, 1235, 1119, 1029; MS (70 ev, EI) *m/z* (%) 458 (M^+, 37.17), 289 (100); Elemental analysis calcd for $C_{23}H_{17}F_7O_2$: C, 60.27; H, 3.74; Found: C, 60.07; H, 3.93.

(2) AuCl catalyzed cycloisomerization of **1j** to form

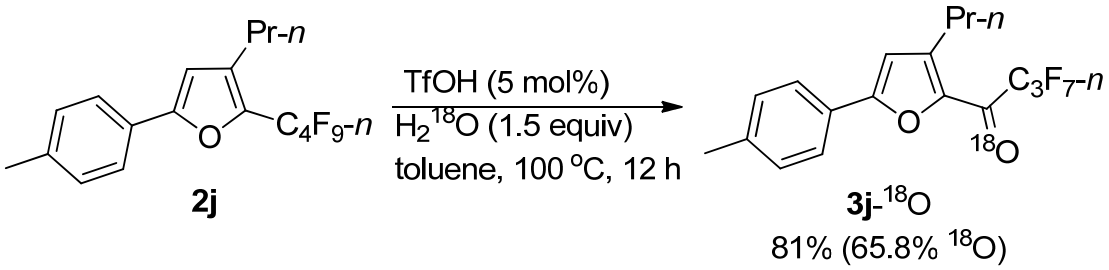
5-(4'-methylphenyl)-2-(*n*-perfluorobutyl)-3-(*n*-propyl)furan (**2j**) (xc-8-148)



To a dried Schlenk tube were added anhydrous AuCl (97%, 0.0023 g, 0.010 mmol), 1-(4'-methylphenyl)hexa-1,2-dien-3-yl perfluorobutyl ketone **1j** (0.0832 g, 0.20 mmol), and anhydrous toluene (2 mL) at room temperature under N_2 atmosphere. The resulting mixture was then placed in a pre-heated oil bath of 100 ^{o}C and stirred for 12 h as monitored by TLC. After being cooled to room temperature, the crude reaction mixture was filtrated through a short column of silica gel eluted with 30 mL of Et_2O. After removing the solvent via rotary evaporation, column chromatography on silica gel (eluent: petroleum ether) afforded **2j** (0.0812 g, 98%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.1 Hz, 2 H, ArH), 7.20 (d, *J* = 8.1 Hz, 2 H, ArH), 6.56 (s, 1 H, =CH), 2.55 (t, *J* = 7.7 Hz, 2 H, CH_2), 2.37 (s, 3 H, CH_3), 1.74-1.54 (m, 2 H, CH_2), 0.97 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.1, 138.7, 134.0 (t, *J* = 32.3 Hz), 132.6, 129.5, 126.7, 124.3, 107.2, 26.3, 23.2, 21.3, 13.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.5 (t, *J* = 9.4 Hz, 3 F), -110.8 (t, *J* = 11.6 Hz, 2 F), -124.0~-124.3 (m, 2 F), -126.6~-126.9 (m, 2 F); IR (neat) ν (cm^{-1}) 3029, 2967, 2935, 2876, 1906, 1621, 1581, 1557, 1498, 1468, 1417, 1393, 1350, 1315, 1235, 1135, 1094, 1053; MS (70 ev, EI) *m/z* (%) 418 (M^+, 20.78), 249 (100); HRMS calcd for $C_{18}H_{15}F_9O$ (M^+): 418.0979, Found: 418.0978.

(3) TfOH-catalyzed transformation of 2-perfluorobutyl furan **2j** to

^{18}O-(5-(4'-methylphenyl)-3-(*n*-propyl)furan-2-yl) (*n*-perfluoropropyl) ketone **3j**-^{18}O in the presence of $H_2{}^{18}O$. (xc-8-149)



Following Typical Procedure III, the reaction of 2-(perfluorobutyl)-3-propyl-5-(*p*-tolyl)furan **2j** (0.0812 g, 0.19 mmol) with TfOH (99%, 0.9 μL, *d* = 1.695 g/mL, 0.0015 g, 0.010 mmol) and $H_2{}^{18}O$ (97%, 5.6 μL, *d* = 1.1 g/mL, 0.0062 g, 0.30 mmol) (transferred with a syringe from a glove box) at 100 ^{o}C for 12 h to afford **3j**-^{18}O (0.0625 g, 81%) (petroleum ether) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.1 Hz, 2 H, ArH), 7.26 (d, *J* = 7.5 Hz, 2 H, ArH), 6.77 (s, 1 H, =CH), 2.89 (t, *J* = 7.7 Hz, 2 H, CH_2), 2.40 (s, 3 H, CH_3), 1.78-1.60 (m, 2 H, CH_2), 1.01 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1 (t, *J* = 26.1 Hz, 3 F), 159.4, 147.0, 143.7, 140.7, 129.8, 125.8, 125.4, 109.8, 28.4, 22.3, 21.5, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 8.9 Hz, 3 F), -117.7~-118.3 (m, 2 F), -126.0~-126.6 (m, 2 F); MS (70 ev, EI) *m/z* (%) 396 (M (^{16}O)$^{+}$, 22.56), 398 (M (^{18}O)$^{+}$, 45.41), 229 (100); HRMS (EI) Cacld for $C_{18}H_{15}{}^{16}O_2F_7$ (M^{+}): 396.0960; Found: 396.0962; HRMS (EI) Cacld for $C_{18}H_{15}{}^{16}O^{18}OF_7$ (M^{+}): 398.1003; Found : 398.1002.

The ^{18}O% incorporation of **3j**-^{18}O was determined via the analysis of MS spectrum. The natural abundances of the stable isotopes of C, H, and O are known. The naturally occurring isotopic ^{18}O will also produce [M(^{18}O)]$^{+}$ peak. According to the natural abundance of ^{18}O, the ratio $C_{18}H_{15}{}^{16}O_2F_7$: $C_{18}H_{15}{}^{16}O^{18}OF_7$ is 99.762:0.2.

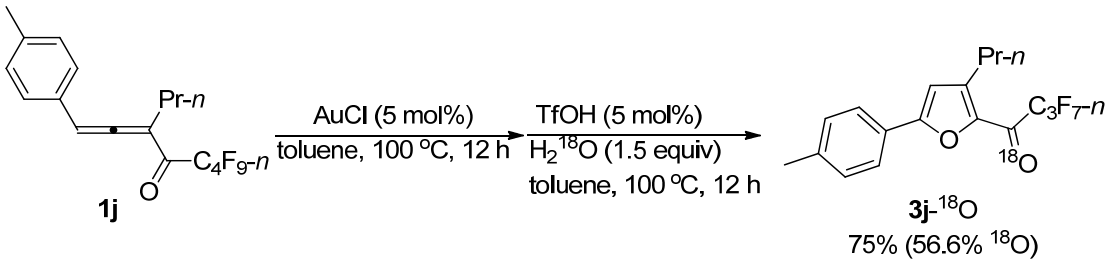
Thus, the intensity of $[M(^{18}O)^+]$ $(C_{18}H_{15}{}^{16}O^{18}OF_7)^+$ peak will be 0.2% of the intensity of the molecular peak $[M(^{16}O)^+]$ $(C_{18}H_{15}{}^{16}O_2F_7)$. According to the MS spectrum of **3j**-^{18}O, the relative abundances of **3j**-^{16}O 396 $[M(^{16}O)^+]$ and **3j**-^{18}O 398 $[M(^{18}O)^+]$ are 22.56, 45.41, respectively. The ^{18}O% of **3j**-^{18}O can be calculated as follows: $([M(^{18}O)^+] - [M(^{16}O)^+] \times 0.2\%)/([M(^{18}O)^+] - [M(^{16}O)^+] \times 0.2\% + [M(^{16}O)^+]) = 45.41\text{-}22.56\times0.002/(45.41\text{-}22.56\times0.002+ 22.56) \approx 66.79\%$.

In addition, the contributions to the isotope peak intensities from background peaks or from impurities in the sample must be considered. According to the MS spectrum of **3j**, such contribution of **3j** to $[M(^{16}O)+2]^+$ is $(1.03\text{-}33.70\times0.002)\%$ ($\approx$ 0.96%).

So the ^{18}O% of **3j**-^{18}O is 66.79% - 0.96% $\approx$ 65.83%.

(4) One-pot reaction of allenyl perfluorobutyl ketone **1j** sequentially catalyzed by AuCl (5 mol%) and TfOH (5 mol%) in the presence of $H_2{}^{18}O$.

^{18}O-(5-(4'-methylphenyl)-3-(*n*-propyl)furan-2-yl) (*n*-perfluoropropyl) ketone (**3j**-^{18}O) (xc-8-147)



To a dried Schlenk tube were added anhydrous AuCl (97%, 0.0025 g, 0.010 mmol), 1-(4'-methylphenyl)hexa-1,2-dien-3-yl perfluorobutyl ketone **1j** (0.0831 g, 0.20 mmol), and anhydrous toluene (2 mL) at room temperature under N_2 atmosphere. The resulting mixture was then placed in a pre-heated oil bath of 100 ^{o}C and stirred.

After 12 h as monitored by TLC, the cycloisomerization reaction was over, TfOH (99%, 0.9 μL, *d* = 1.695 g/mL, 0.0015 g, 0.010 mmol) and $H_2{}^{18}O$ (97%, 5.6 μL, *d* = 1.1 g/mL, 0.0062 g, 0.30 mmol) (transferred with a syringe from a glove box) were added to the above crude reaction mixture, and the resulting mixture was stirred at 100 ^oC for another 12 h as monitored by TLC. After being cooled to the room temperature, the reaction mixture was transferred to a round bottom flask. After removing the solvent via rotary evaporation, column chromatography on silica gel (eluent: petroleum ether) afforded **3j**-^{18}O (0.0590 g, 75%) as a liquid: ^1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.1 Hz, 2 H, ArH), 7.26 (d, *J* = 8.1 Hz, 2 H, ArH), 6.77 (s, 1 H, =CH), 2.89 (t, *J* = 7.8 Hz, 2 H, CH_2), 2.40 (s, 3 H, CH_3), 1.78-1.59 (m, 2 H, CH_2), 1.01 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.0 (t, *J* = 9.0 Hz, 3 F), -117.8~-118.2 (m, 2 F), -126.2~-126.4 (m, 2 F); IR (neat) ν (cm^{-1}) 3031, 2965, 2876, 1667, 1644, 1574, 1532, 1481, 1381, 1351, 1317, 1231, 1119, 1020; MS (70 ev, EI) *m/z* (%) 396 (M (^{16}O)$^+$, 30.22), 398 (M (^{18}O)$^+$, 41.10), 229 (100).

The ^{18}O% incorporation of **3j**-^{18}O was determined via the analysis of the MS spectrum. According to the MS spectrum of **3j**-^{18}O, the relative abundances of **3j**-^{16}O 396 [M(^{16}O)$^+$], **3j**-^{18}O 398 [M(^{18}O)$^+$] are 30.22, 41.10, respectively. The ^{18}O% of **3j**-^{18}O can be calculated as follows: ([M(^{18}O)$^+$] - [M(^{16}O)$^+$] × 0.2%)/([M(^{18}O)$^+$] - [M(^{16}O)$^+$] × 0.2% + [M(^{16}O)$^+$]) = 41.10-30.22×0.002/(41.10-30.22×0.002+30.22) ≈ 57.59%.

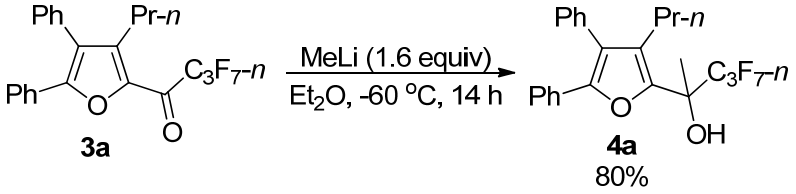
In addition, the contributions to the isotope peak intensities from background peaks or from impurities in the sample must be considered. According to the MS

spectrum of **3j**, such contribution of **3j** to $[M(^{16}O)+2]^+$ is (1.03-33.70×0.002)% (≈ 0.96%).

So the ^{18}O% of **3j**-^{18}O is 57.59% - 0.96% ≈ 56.63%.

3. Synthetic application of 3a.

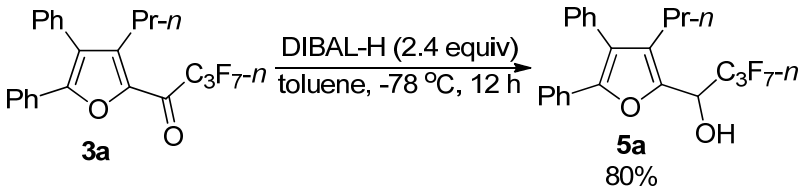
(1) 2-(4,5-Diphenyl-3-propylfuran-2-yl)-3,3,4,4,5,5,5-heptafluoropentan-2-ol (**4a**) (wsz-7-055)



To a dried Schlenk tube were added (4,5-diphenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone **3a** (0.0915 g, 0.20 mmol) and Et_2O (2 mL) at room temperature under a nitrogen atmosphere. The resulting mixture was cooled to -60 °C with a cooling bath. A solution of MeLi (0.11 mL, 3.0 M in diethoxymethane, 0.32 mmol) was added over 10 min at -60 °C and stirred for 14 h as monitored by TLC. The mixture was quenched with 5 mL of a saturated aqueous NH_4Cl solution at -60 °C. After warming up to rt naturally, the mixture was extracted with 20 mL × 3 of Et_2O. The combined organic extract was washed with brine and dried over anhydrous Na_2SO_4. Filtration, evaporation, and chromatography on silica gel (eluent: petroleum ether/ethyl acetate/triethylamine = 15/1/0.1) afforded **4a** (0.0758 g, 80%) as an oil: ^1H NMR (300 MHz, $CDCl_3$) δ 7.48-7.25 (m, 7 H, ArH), 7.24-7.11 (m, 3 H, ArH), 3.05 (s, 1 H, OH), 2.58-2.31 (m, 2 H, CH_2), 1.94 (s, 3 H, CH_3), 1.37-1.22 (m, 2 H, CH_2), 0.74 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.7, 143.2, 133.5, 130.4,

130.1, 128.8, 128.3, 127.6, 127.3, 126.6, 125.4, 125.0, 75.4 (t, J = 24.7 Hz), 25.7, 23.7, 22.3, 14.2; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.3 (t, J = 11.1 Hz, 3 F), -118.8~-121.3 (m, 2 F), -124.7~-125.1 (m, 2 F); IR (neat) ν (cm^{-1}) 3548, 3060, 3029, 2963, 2933, 2873, 1603, 1503, 1482, 1446, 1339, 1228, 1191, 1120, 1071; MS (70 ev, EI) *m/z* (%) 474 (M^+, 10.18), 305 (100); HRMS calcd for $C_{24}H_{21}F_7O_2$ (M^+): 474.1430, Found: 474.1432.

(2) 1-(4,5-Diphenyl-3-propylfuran-2-yl)-2,2,3,3,4,4,4-heptafluorobutan-1-ol (**5a**) (wsz-7-073)

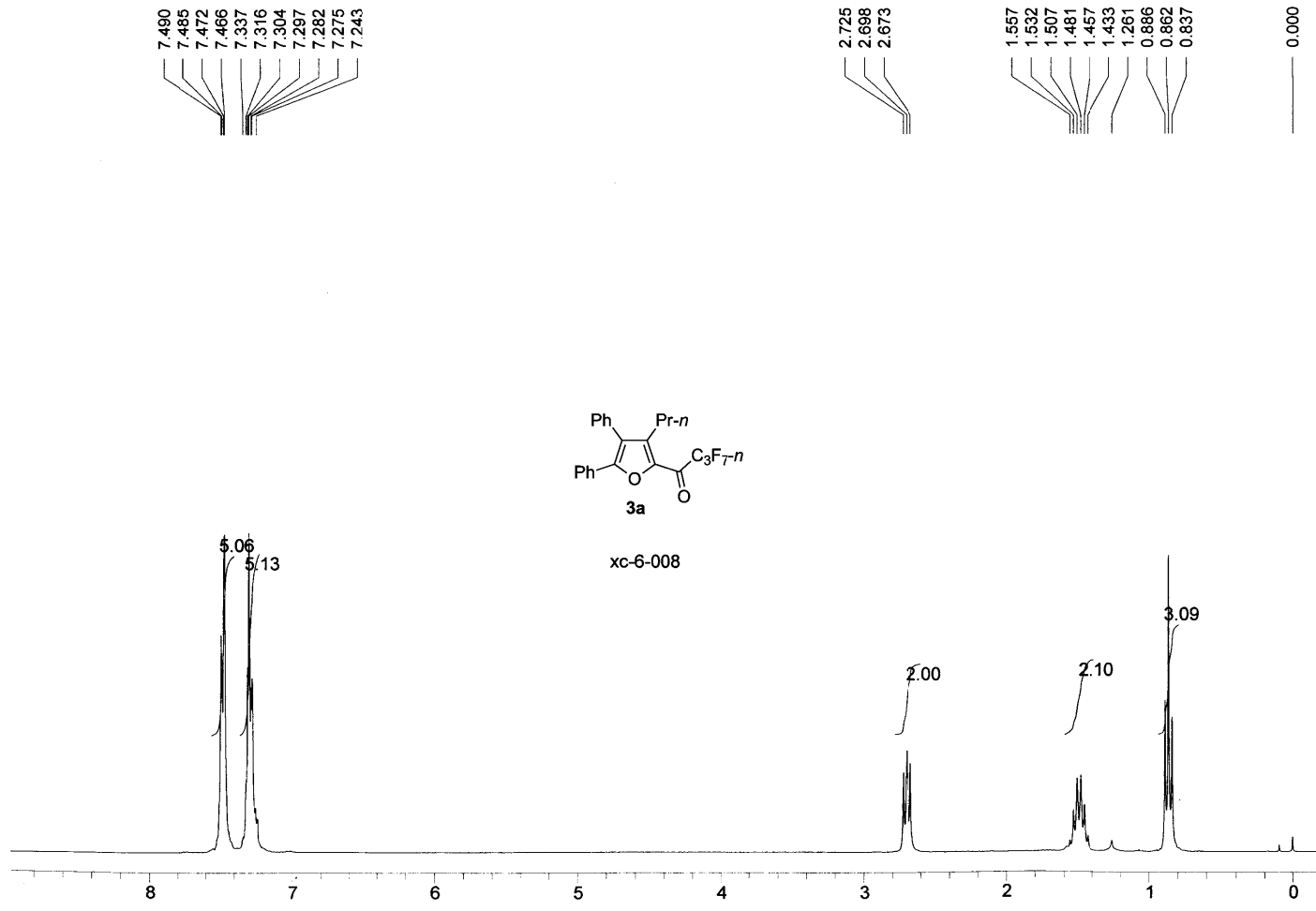


To a dried Schlenk tube were added (4,5-diphenyl-3-(*n*-propyl)furan-2-yl) *n*-perfluoropropyl ketone **3a** (0.0915 g, 0.20 mmol) and toluene (1 mL) at room temperature under a nitrogen atmosphere. The resulting mixture was cooled to -78 ^{o}C with a cooling bath. A solution of DIBAL-H (0.48 mL, 1.0 M in toluene, 0.48 mmol) was added over 10 min at -78 ^{o}C and the resulting mixture was stirred for 12 h as monitored by TLC. After warming up to rt naturally, the mixture was quenched with 5 mL of EtOH with an ice-water bath followed by another 10 mL of HCl (5%, aq.). The mixture was extracted with 10 mL × 5 of Et_2O. The combined organic extract was washed with brine and dried over anhydrous Na_2SO_4. Filtration, evaporation, and chromatography on silica gel (eluent: petroleum ether/ethyl acetate/ triethylamine = 15/1/0.1) afforded **5a** (0.0733 g, 80%) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ

7.49-7.32 (m, 5 H, ArH), 7.32-7.26 (m, 2 H, ArH), 7.25-7.14 (m, 3 H, ArH), 5.25 (dd, J_1 = 18.2 Hz, J_2 = 5.9 Hz, 1 H, CH), 2.94 (brs, 1 H, OH), 2.34 (t, J = 7.8 Hz, 2 H, CH_2), 1.36-1.22 (m, 2 H, CH_2), 0.78 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.5, 140.6, 133.2, 130.3, 129.9, 128.9, 128.4, 128.3, 127.69, 127.66, 125.7, 123.7, 64.7 (dd, J_1 = 28.0 Hz, J_2 = 23.1 Hz), 25.2, 23.2, 13.8; ^{19}F NMR (282 MHz, $CDCl_3$) δ -81.2 (dd, J_1 = 11.3 Hz, J_2 = 9.0 Hz, 3 F), -118.8 (dm, J = 281.4 Hz, one fluorine of CF_2), -125.4 (dm, J = 281.3 Hz, one fluorine of CF_2), -126.1 (dm, J = 290.6 Hz, one fluorine of CF_2), -127.4 (dm, J = 290.5 Hz, one fluorine of CF_2); IR (neat) ν (cm^{-1}) 3482, 3054, 3024, 2956, 2929, 2878, 1605, 1507, 1447, 1382, 1343, 1233, 1177, 1120, 1070; MS (70 ev, EI) *m/z* (%) 460 (M^+, 67.19), 291 (100); HRMS calcd for $C_{23}H_{19}F_7O_2$ (M^+): 460.1273; Found: 460.1272.

Reference

[1] G. He, C. Xue, C. Fu, S. Ma, *Synlett* **2010**, *2*, 281-285.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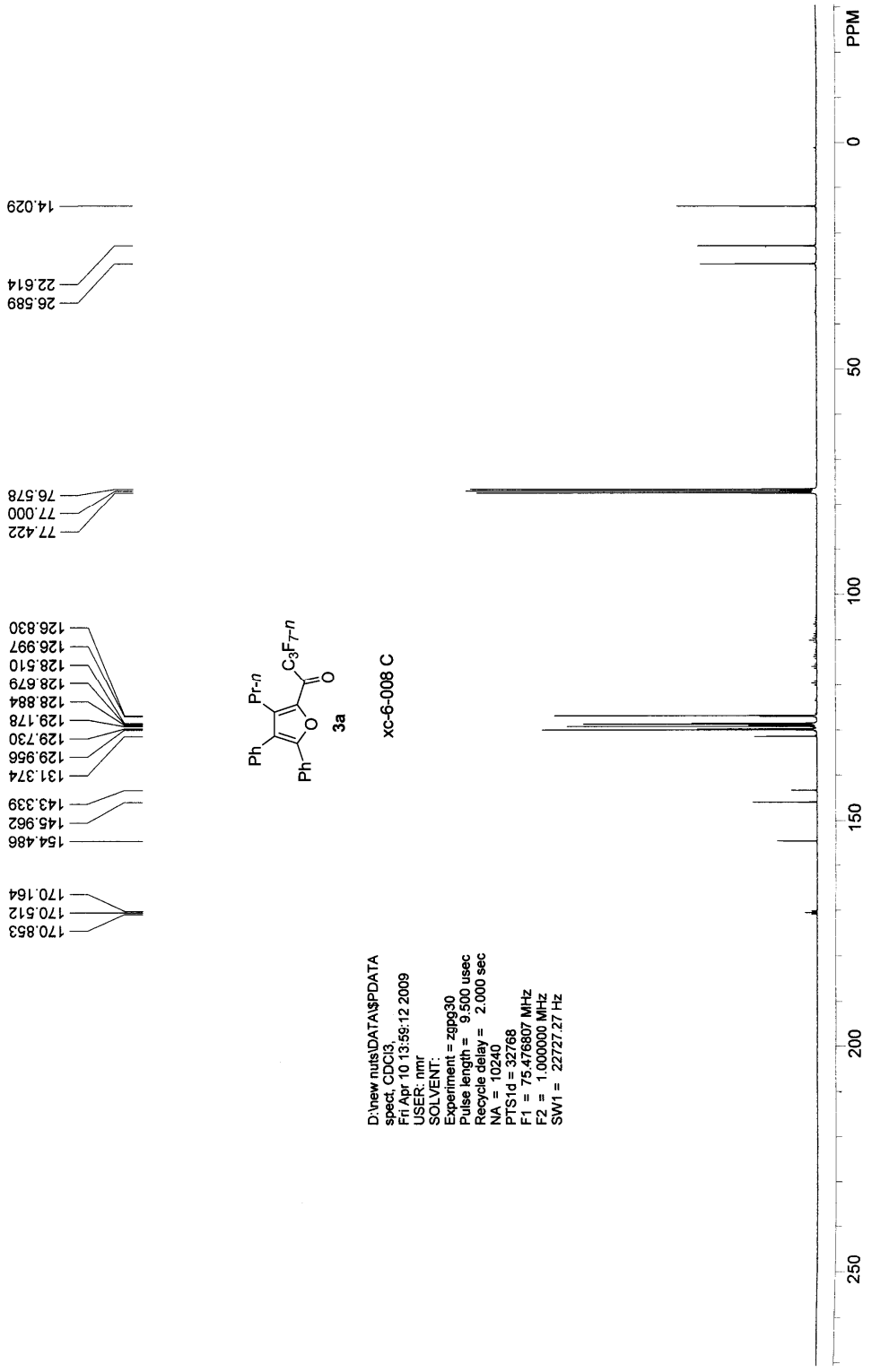
7.472
7.466
7.337
7.316
7.304
7.297
7.282
7.275
7.243
2.725
2.698
2.673
1.557
1.532
1.507
1.481
1.457
1.433
1.261
0.886
0.862
0.837
0.000
Ph
Pr-n
Ph
O
C3F7-n
O
3a
xc-6-008
5.06
5.13
2.00
2.10
3.09
8
7
6
5
4
3
2
1
0
PPM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22.614
26.589
76.578
77.000
77.422
126.830
126.997
128.510
128.679
128.884
129.178
129.730
129.956
131.374
143.339
145.962
154.486
170.164
170.512
170.853
PPM
0
50
100
150
200
250
Pr-n
C3F7-n
Ph
Ph
O
O
3a
xc-6-008 C
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Fri Apr 10 13:59:12 2009
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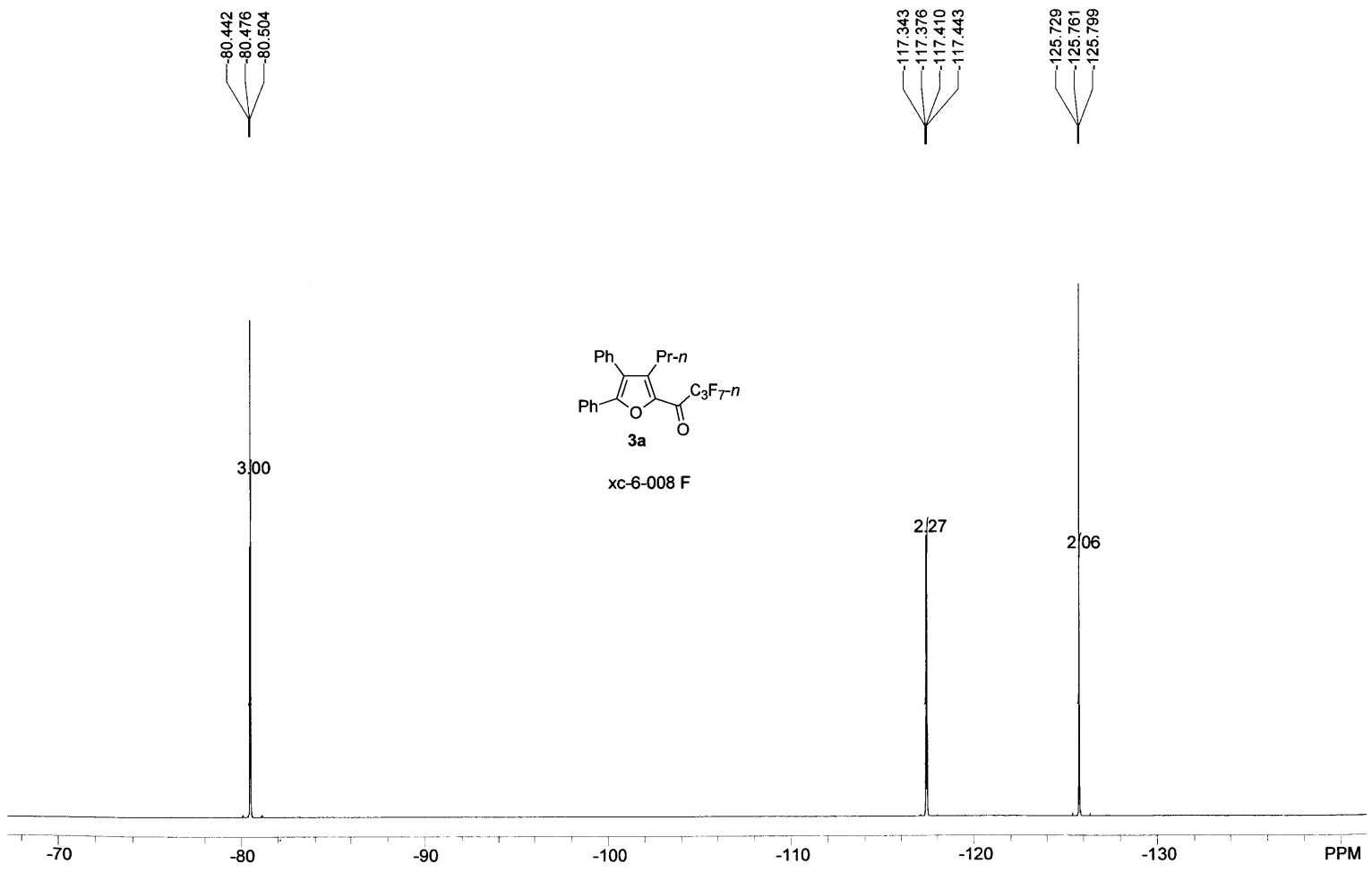
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10240
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz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-117.443
-125.729
-125.761
-125.799
Ph
Pr-n
Ph
O
C3F7-n
O
3a
xc-6-008 F
3.00
2.27
2.06
-70
-80
-90
-100
-110
-120
-130
PPM

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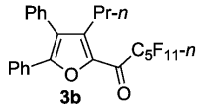
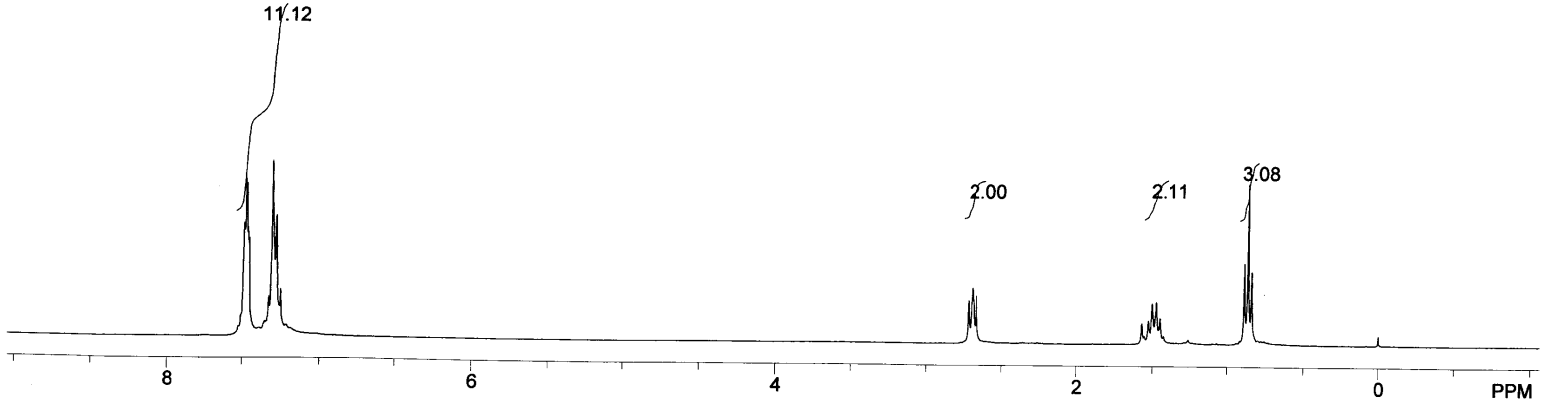
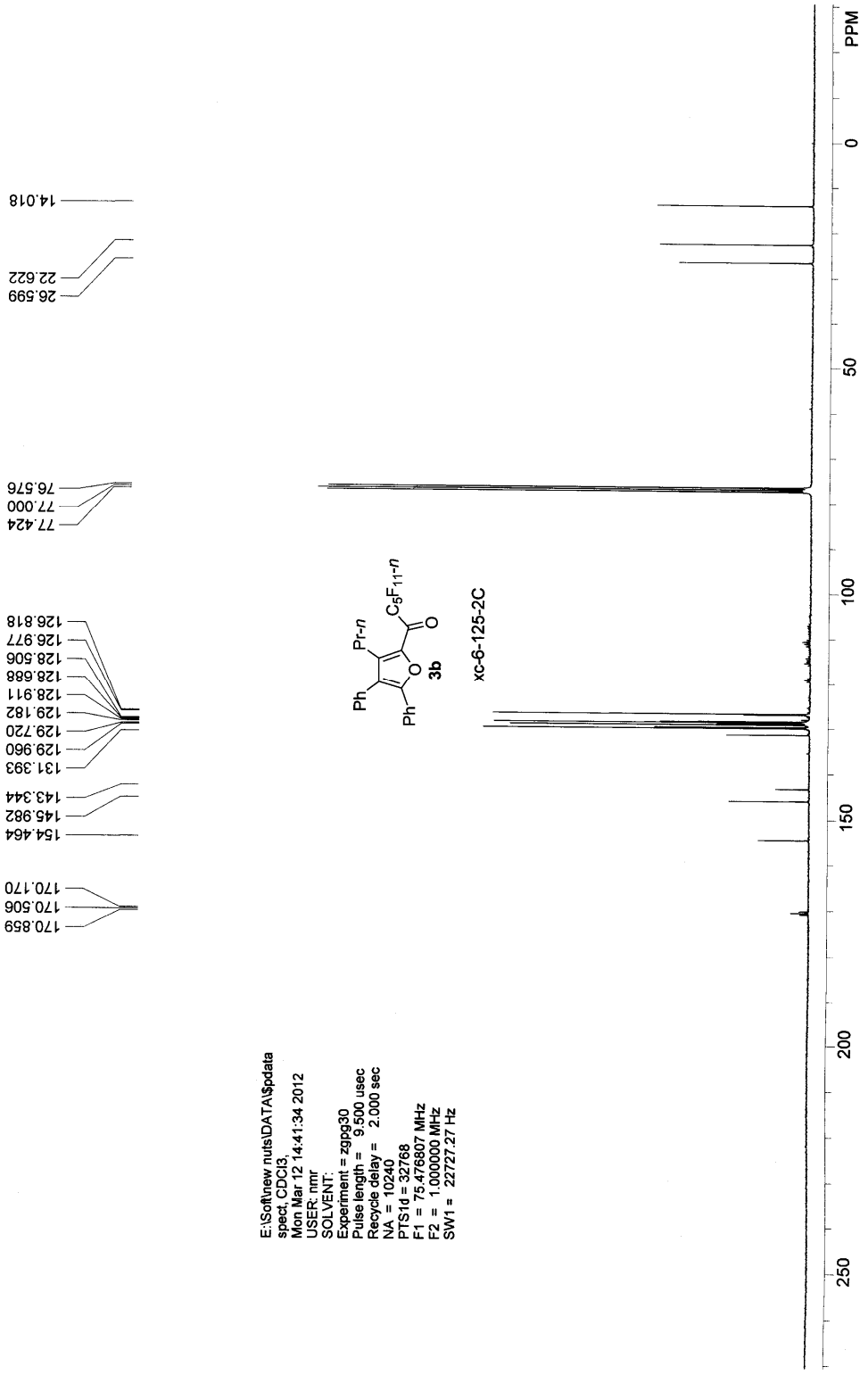
spect, CDCl3,
Mon Mar 12 08:18:16 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



3b

xc-6-125-2

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76.576
77.000
77.424
126.818
126.977
128.506
128.688
128.911
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129.720
129.960
131.393
143.344
145.982
154.464
170.170
170.506
170.859
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USER: nmr
SOLVENT:
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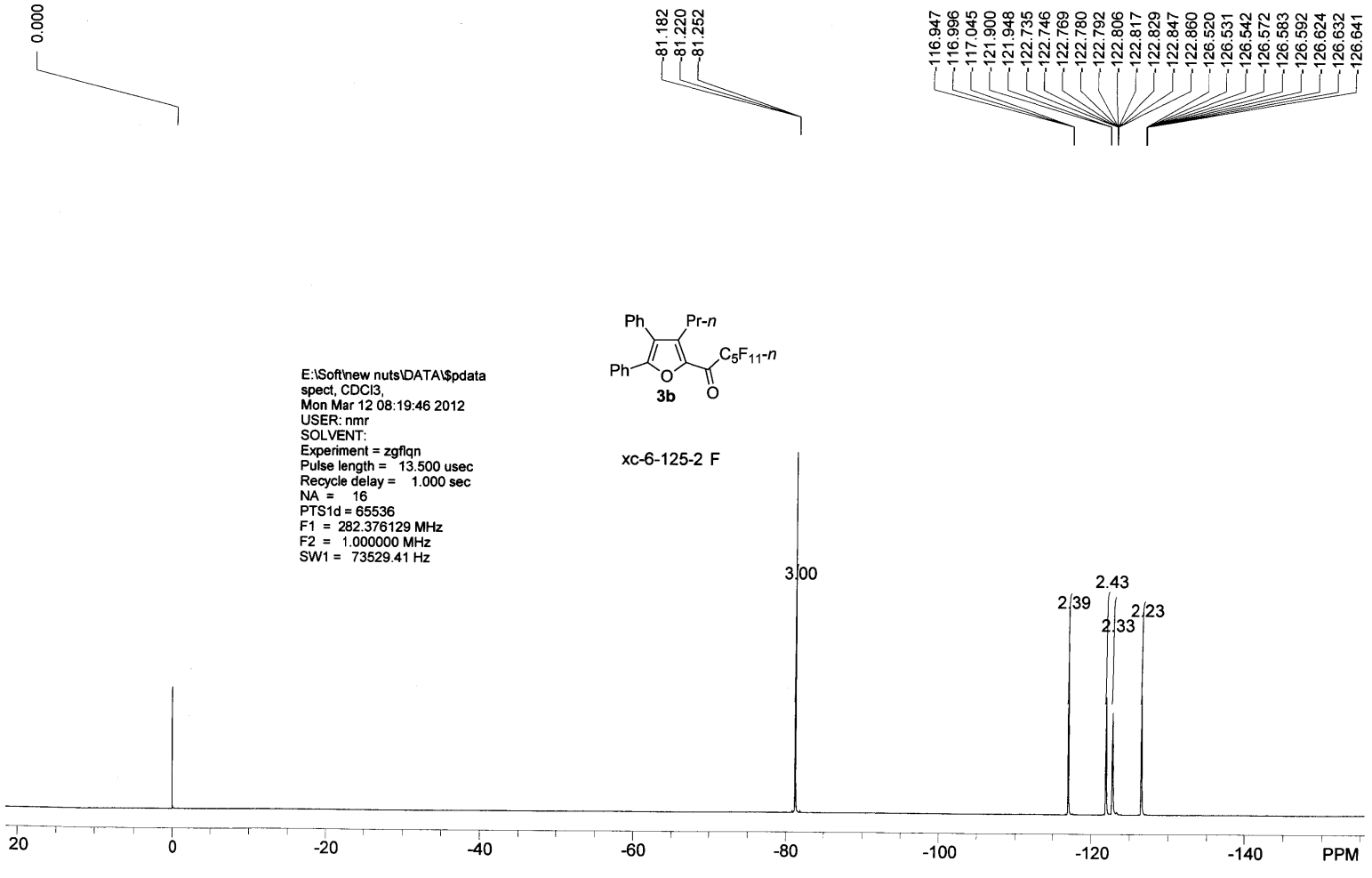
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10240
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Ph
Pr-n
O
C5F11-n
Ph
3b
xc-6-125-2C
PPM
0
50
100
150
200
250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-81.220
-81.252
116.947
116.996
117.045
121.900
121.948
122.735
122.746
122.769
122.780
122.792
122.806
122.817
122.829
122.847
122.860
126.520
126.531
126.542
126.572
126.583
126.592
126.624
126.632
126.641
Ph
Pr-n
Ph
O
C5F11-n
O
3b
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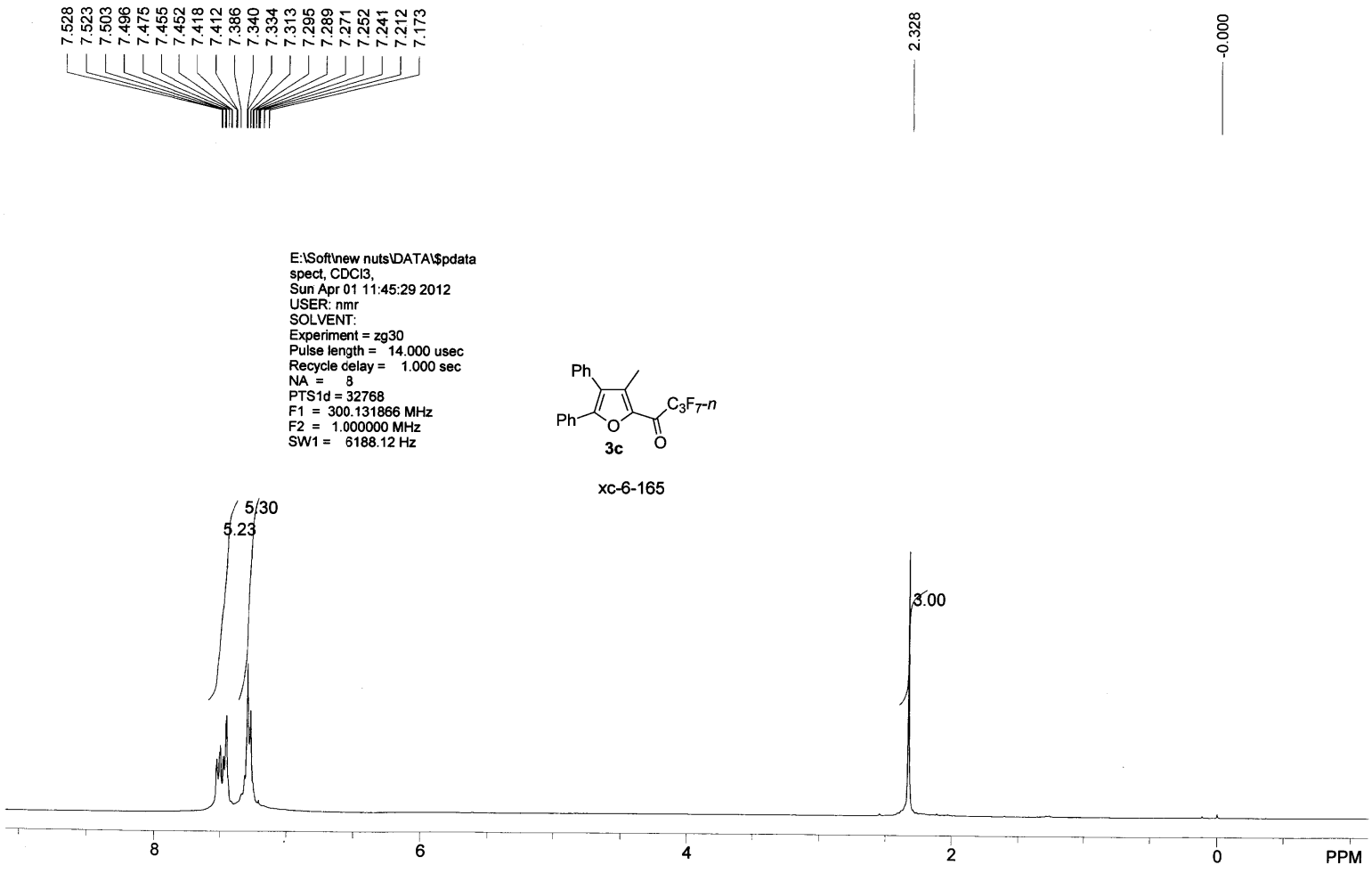
spect, CDCl3,
Mon Mar 12 08:19:46 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
xc-6-125-2 F
3.00
2.43
2.39
2.33
2.23
20
0
-20
-40
-60
-80
-100
-120
-140
PPM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7.523
7.503
7.496
7.475
7.455
7.452
7.418
7.412
7.386
7.340
7.334
7.313
7.295
7.289
7.271
7.252
7.241
7.212
7.173
2.328
-0.000
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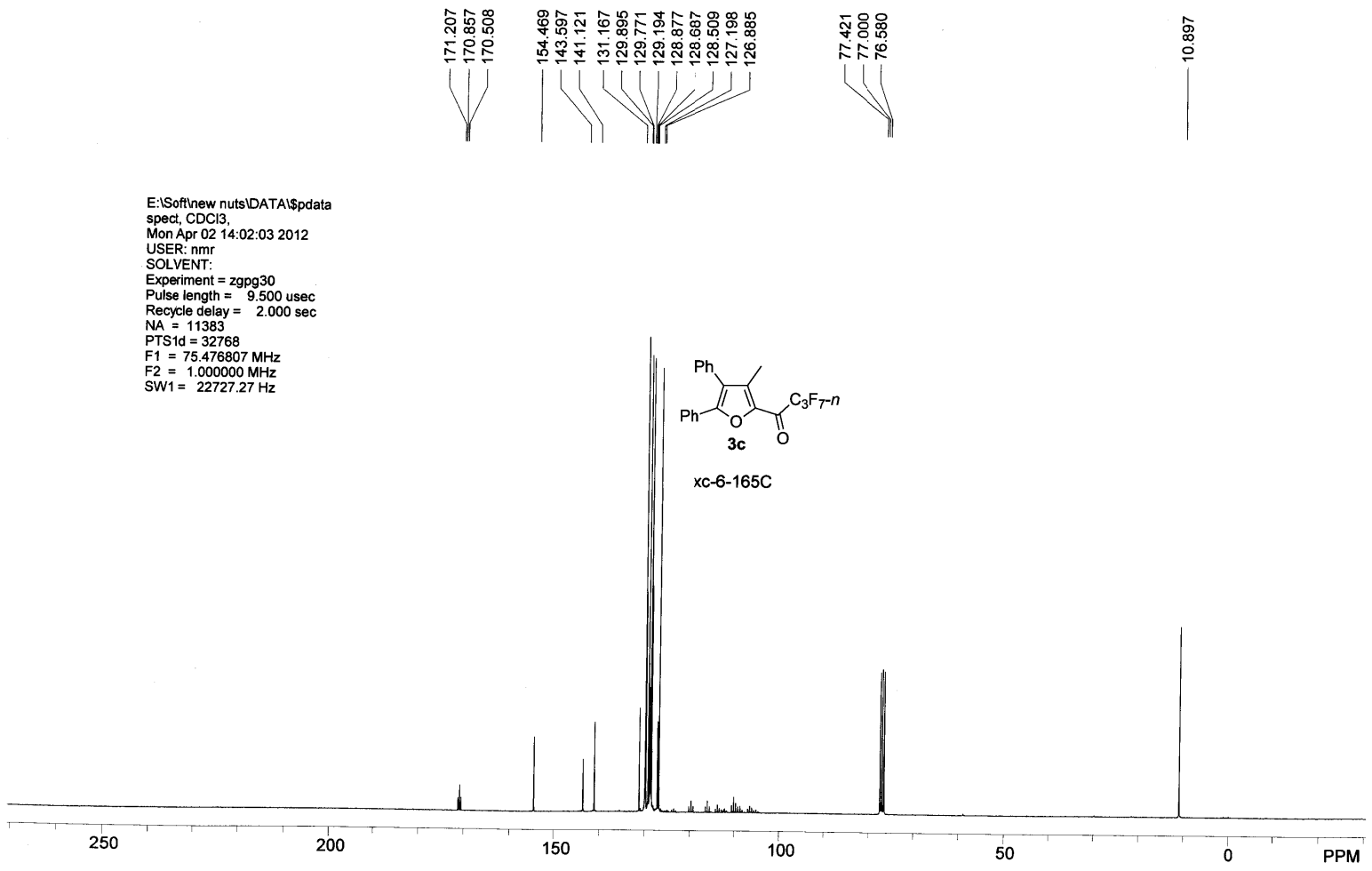
Sun Apr 01 11:45:29 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Ph
Ph
O
C3F7-n
O
3c
xc-6-165
5.30
5.23
3.00
8
6
4
2
0
PPM


171.207
170.857
170.508
154.469
143.597
141.121
131.167
129.895
129.771
129.194
128.877
128.687
128.509
127.198
126.885
77.421
77.000
76.580
10.897
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spect, CDCl3,
Mon Apr 02 14:02:03 2012
USER: nmr
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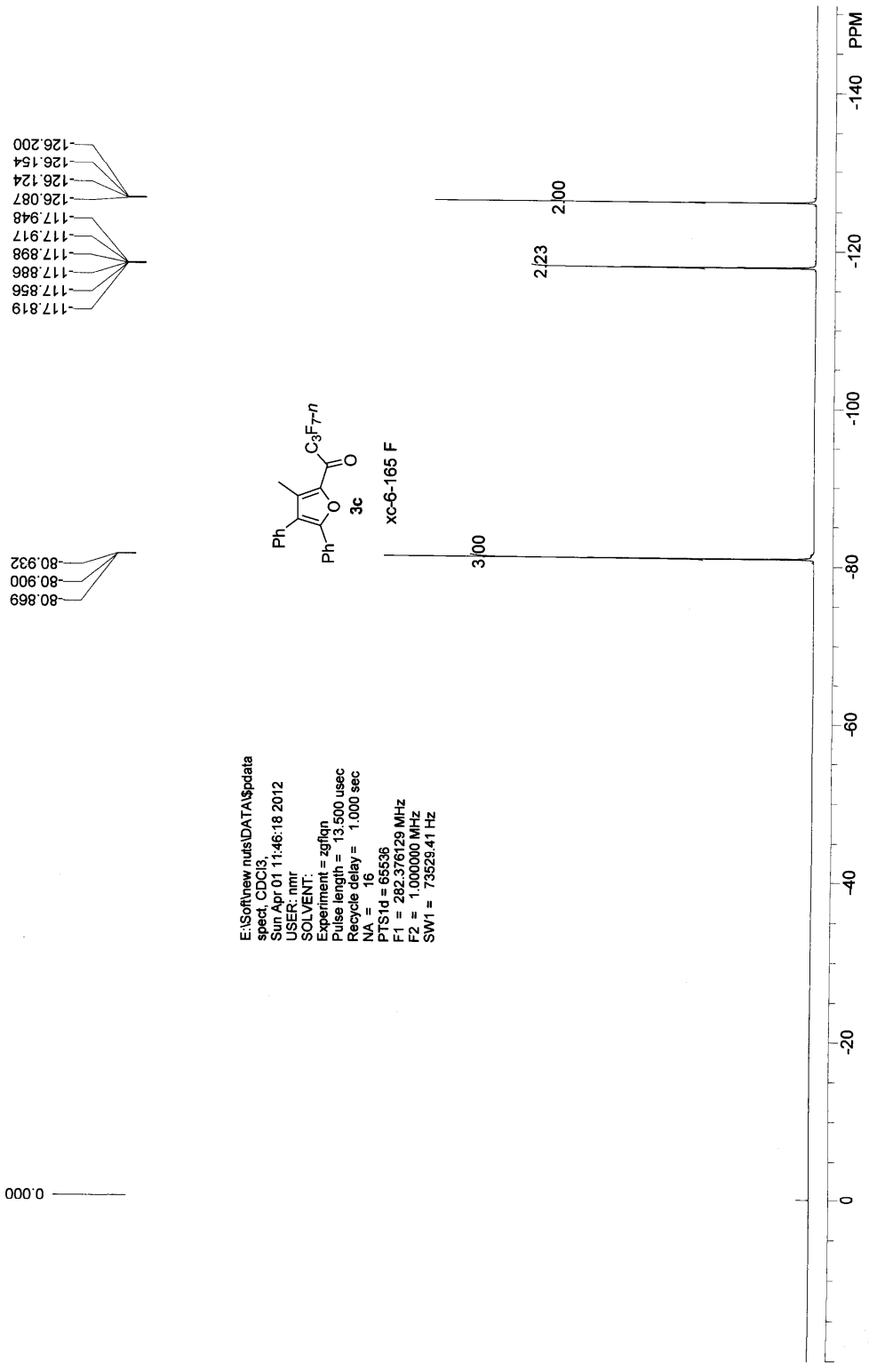
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 11383
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Ph
Ph
O
C3F7-n
O
3c
xc-6-165C
250
200
150
100
50
0
PPM


0.000
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-80.900
-80.932
-117.819
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-117.948
-126.087
-126.124
-126.154
-126.200
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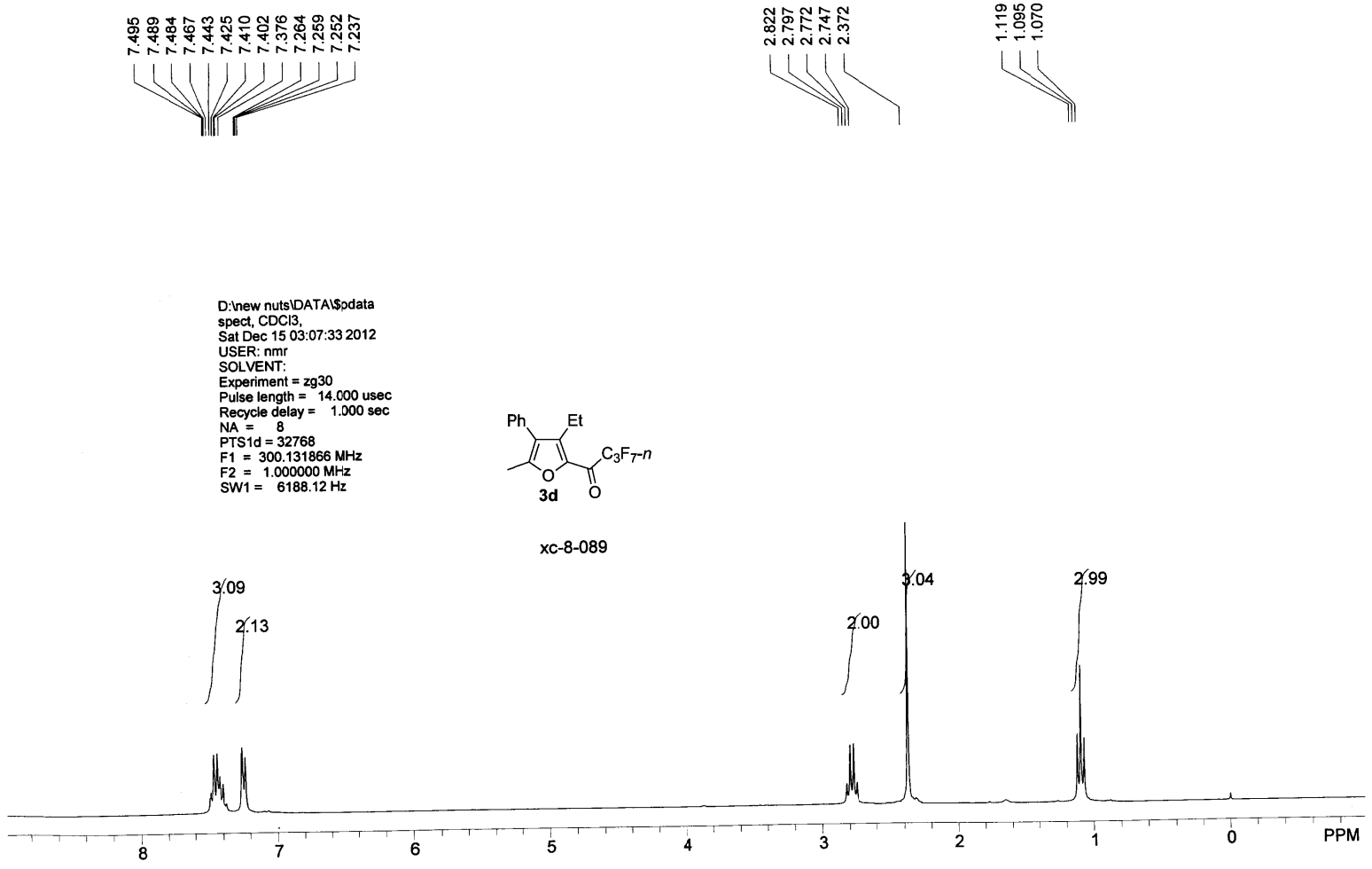
spect, CDCl3,
Sun Apr 01 11:46:18 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
Ph
Ph
O
O
C_3F_{7-n}
3c
xc-6-165 F
3.00
2.23
2.00
0
-20
-40
-60
-80
-100
-120
-140
PPM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7.489
7.484
7.467
7.443
7.425
7.410
7.402
7.376
7.264
7.259
7.252
7.237
2.822
2.797
2.772
2.747
2.372
1.119
1.095
1.070
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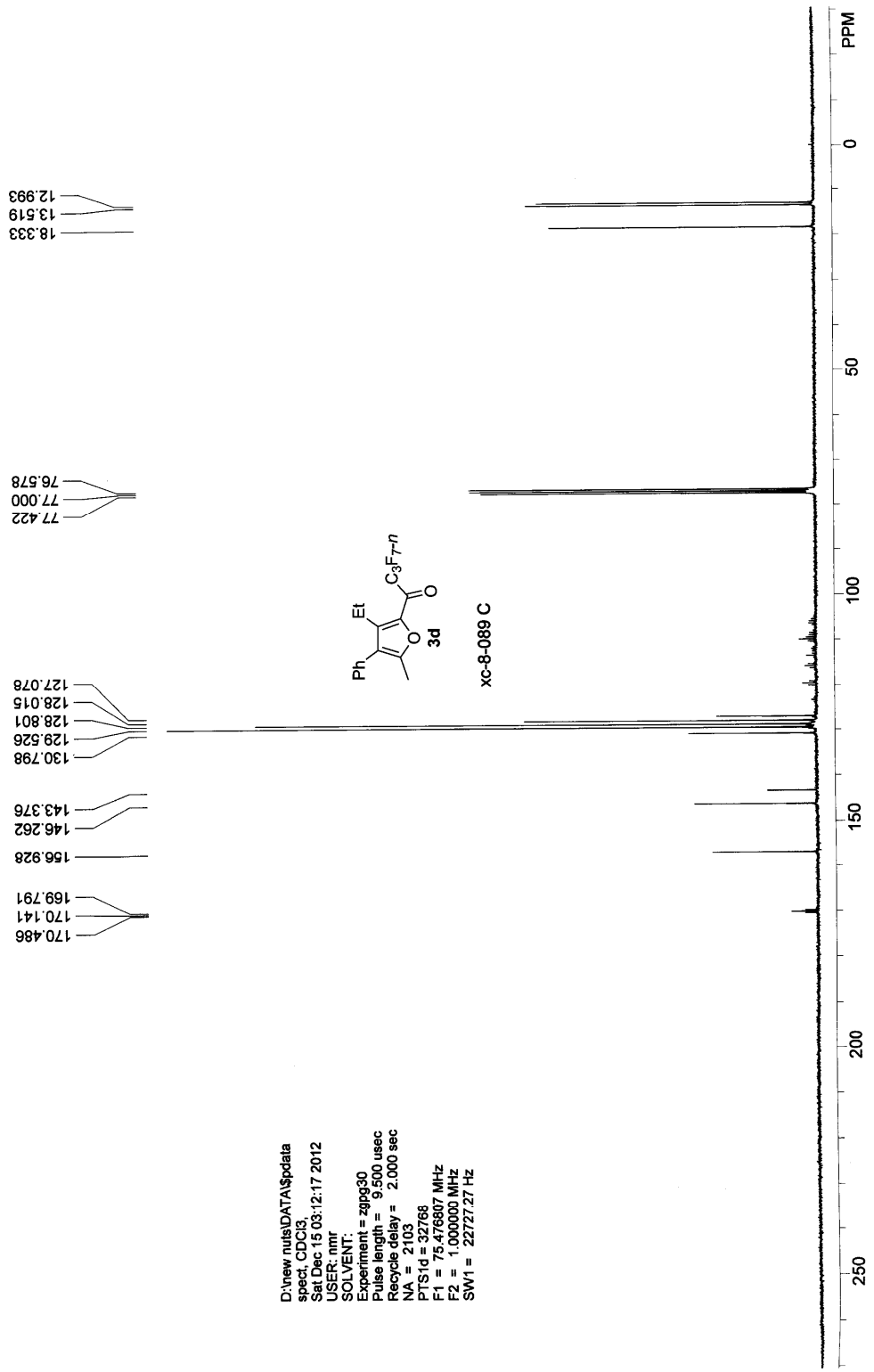
spect, CDCl3,
Sat Dec 15 03:07:33 2012
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Ph
Et
C3F7-n
O
O
3d
xc-8-089
3.09
2.13
2.00
3.04
2.99
8
7
6
5
4
3
2
1
0
PPM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170.141
169.791
156.928
146.262
143.376
130.798
129.526
128.801
128.015
127.078
77.422
77.000
76.578
18.333
13.519
12.993
Ph
Et
C_3F_{7-n}
O
O
3d
xc-8-089 C
PPM
0
50
100
150
200
250
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Sat Dec 15 03:12:17 2012
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2103
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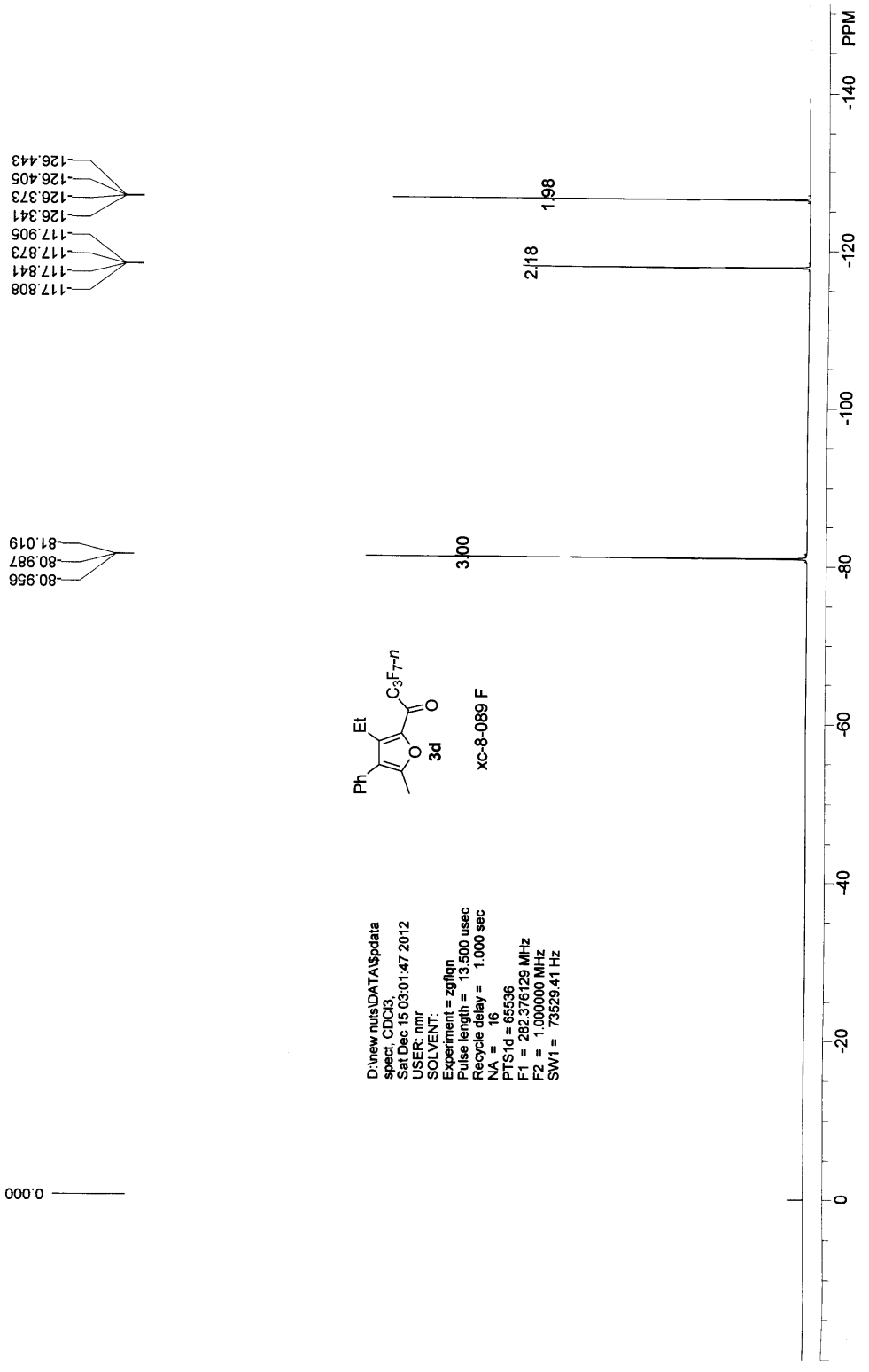
F2 = 1.000000 MHz
SW1 = 22727.27 Hz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117.841
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126.373
126.405
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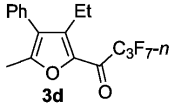
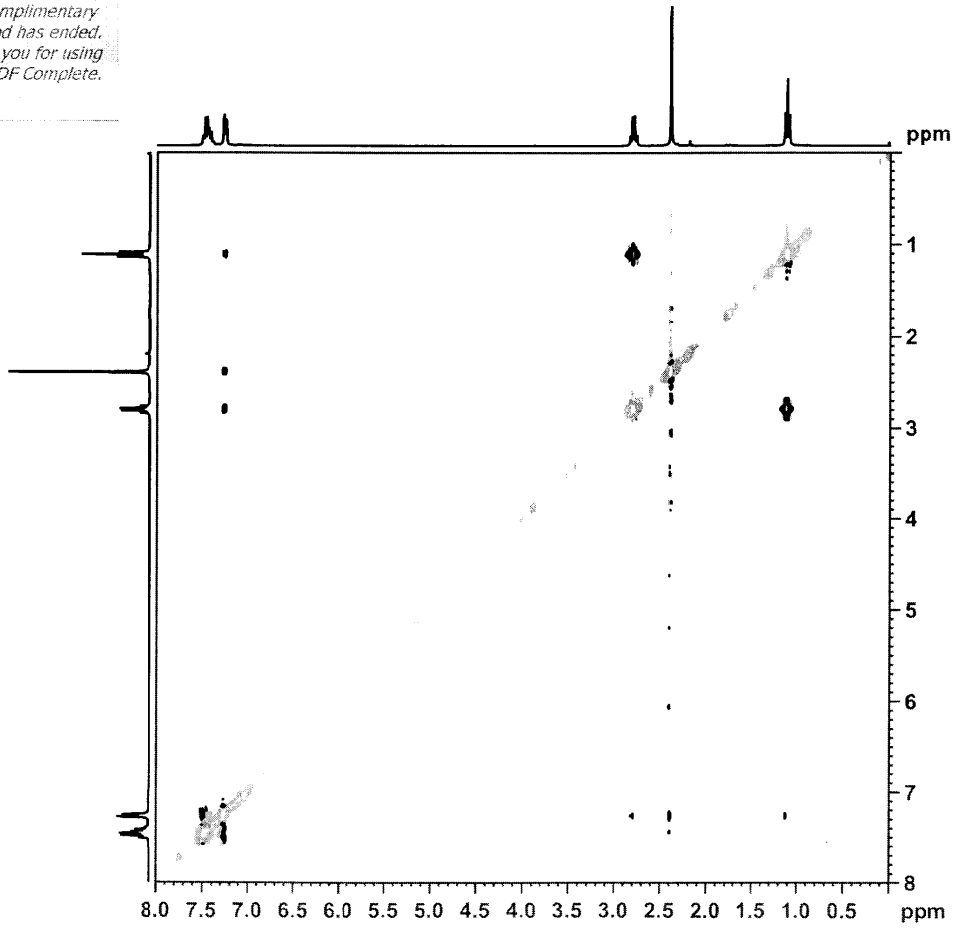
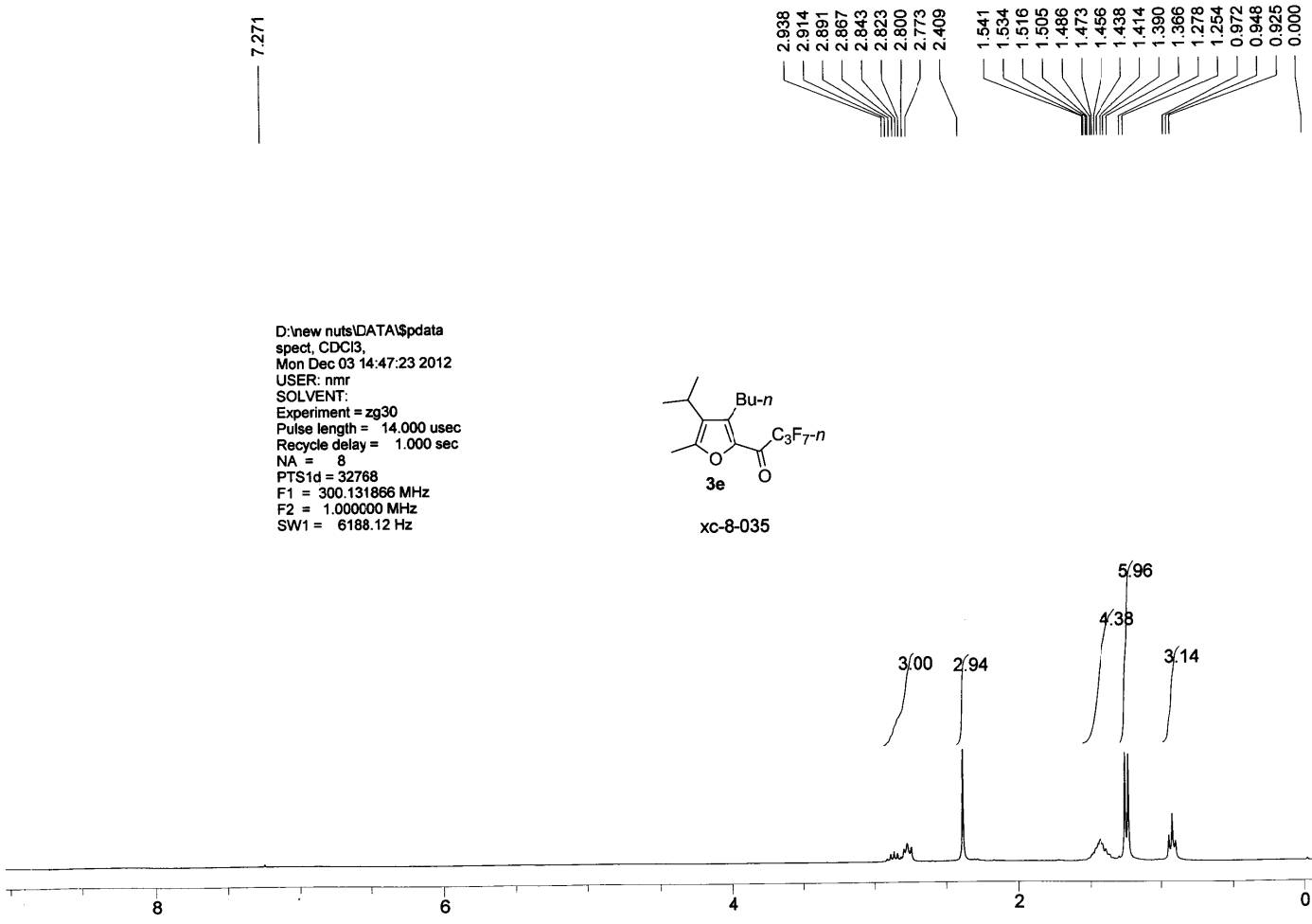
3.00
2.18
1.98
Ph
Et
C_3F_{7-n}
O
3d
xc-8-089 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat Dec 15 03:01:47 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0
-20
-40
-60
-80
-100
-120
-140
PPM



3d

xc-8-089-noe

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2.891
2.867
2.843
2.823
2.800
2.773
2.409
1.541
1.534
1.516
1.505
1.486
1.473
1.456
1.438
1.414
1.390
1.366
1.278
1.254
0.972
0.948
0.925
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Mon Dec 03 14:47:23 2012
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SOLVENT:
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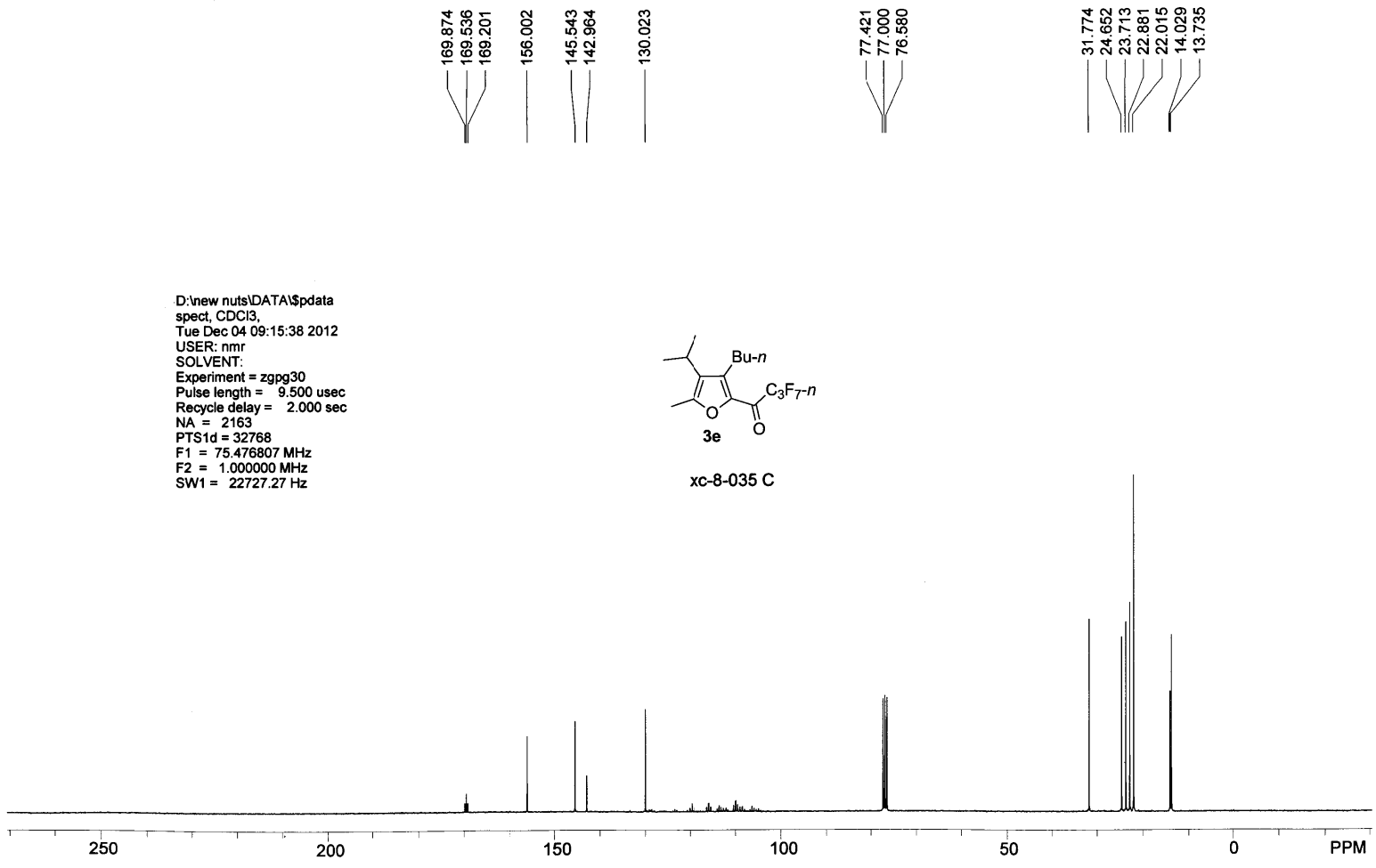
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Bu-n
C3F7-n
O
O
3e
xc-8-035
5.96
4.38
3.14
3.00
2.94
8
6
4
2
0
PPM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76.580
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22.881
22.015
14.029
13.735
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spect, CDCl3,
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USER: nmr
SOLVENT:
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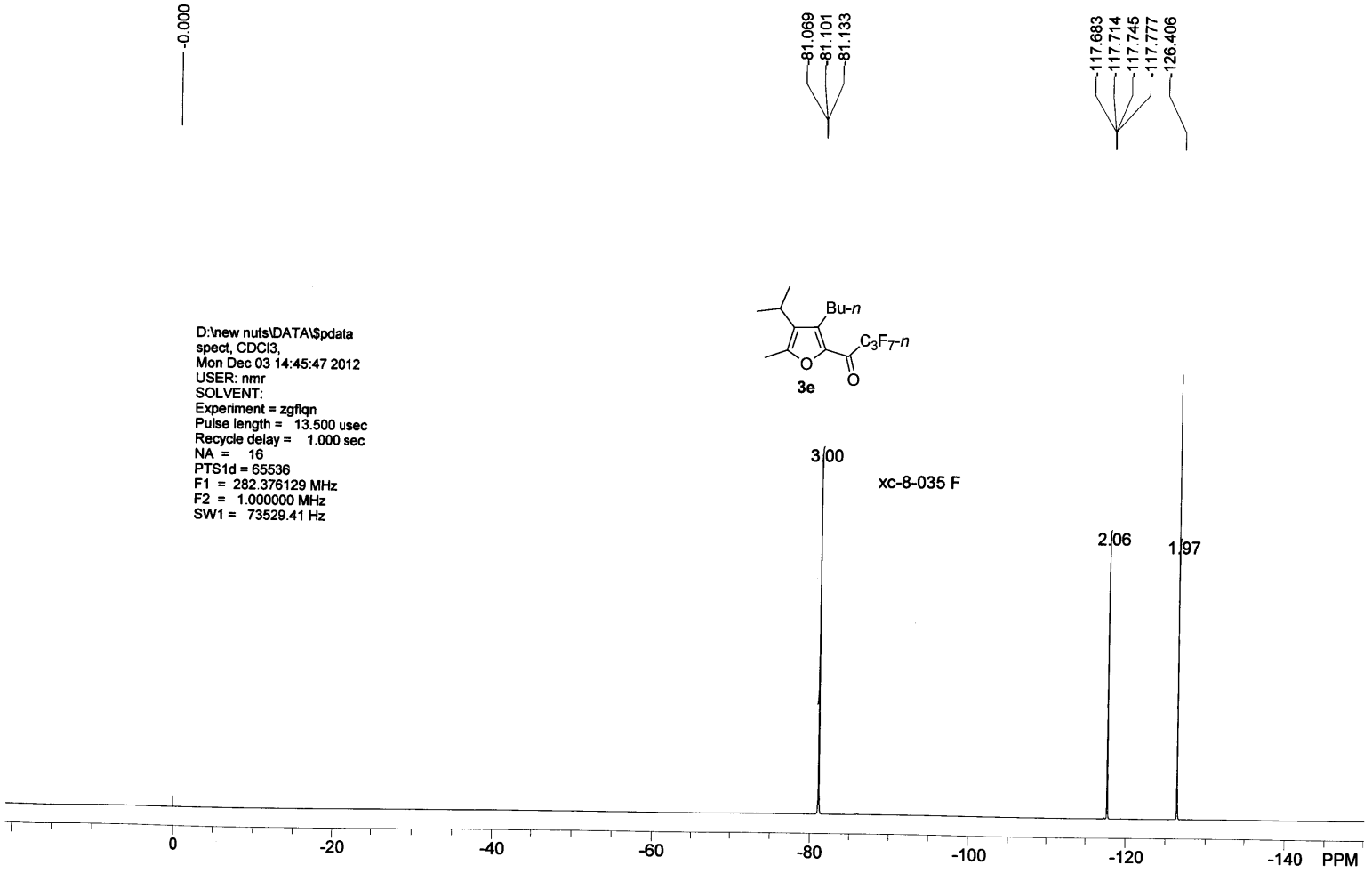
Recycle delay = 2.000 sec
NA = 2163
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Bu-n
C3F7-n
O
O
3e
xc-8-035 C
250
200
150
100
50
0
PPM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spect, CDCl3,
Mon Dec 03 14:45:47 2012
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
Bu-n
C3F7-n
O
O
3e
xc-8-035 F
3.00
2.06
1.97
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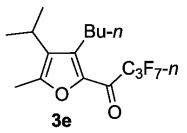
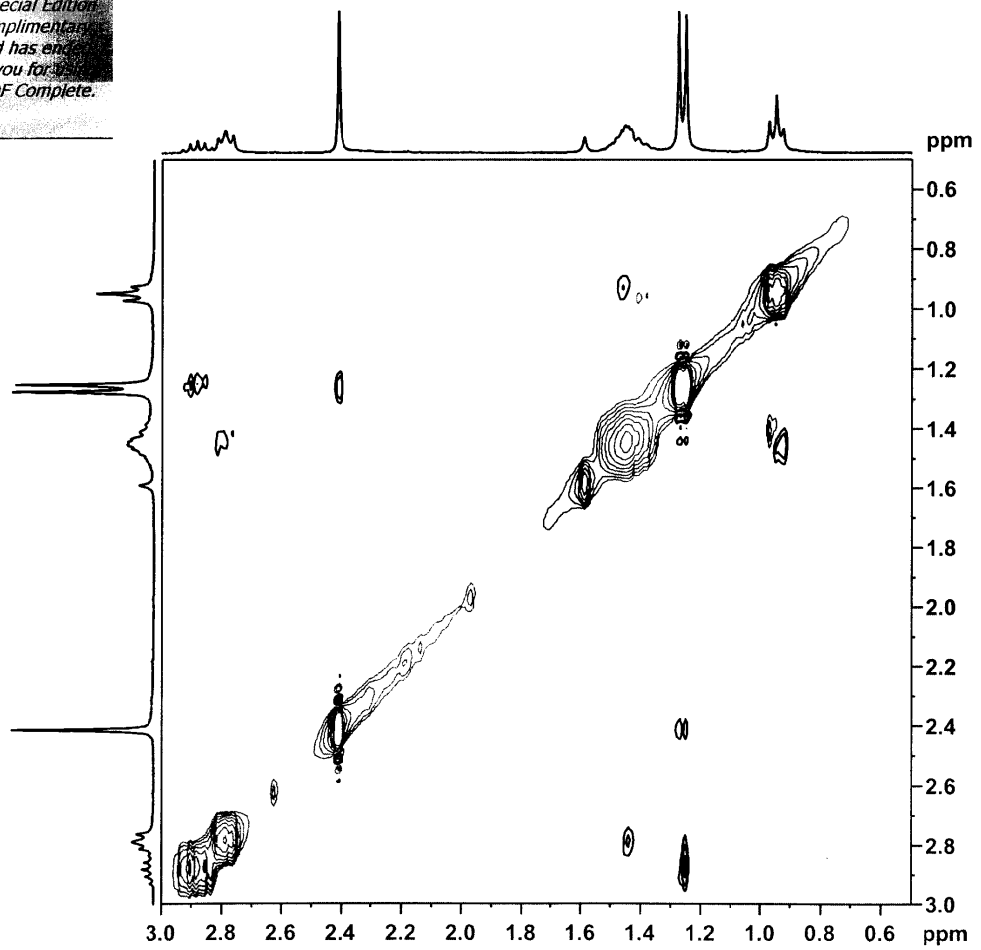
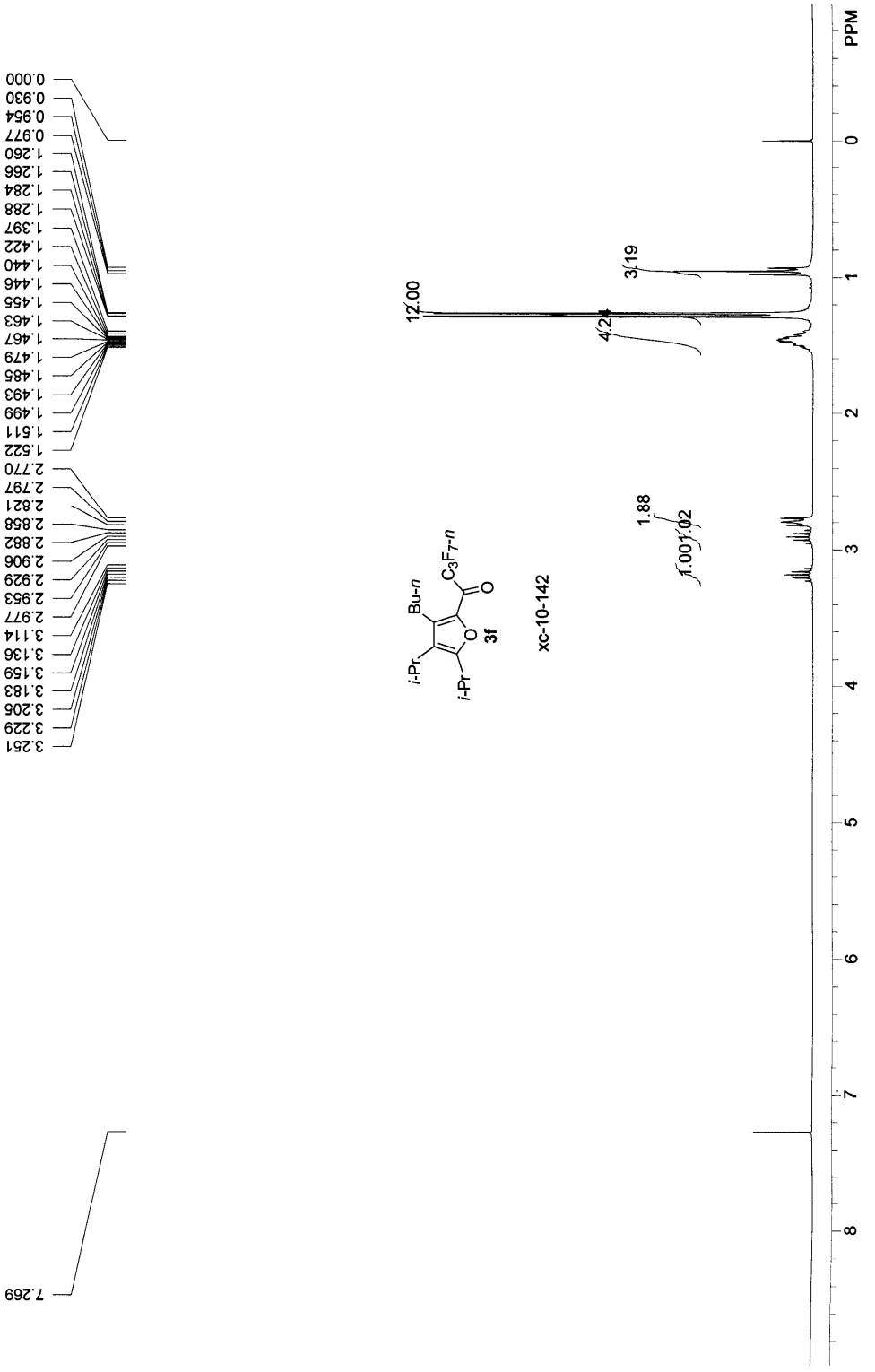
-60
-80
-100
-120
-140
PPM



3e

xc-8-026-noe

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1.288
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1.422
1.440
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2.929
2.953
2.977
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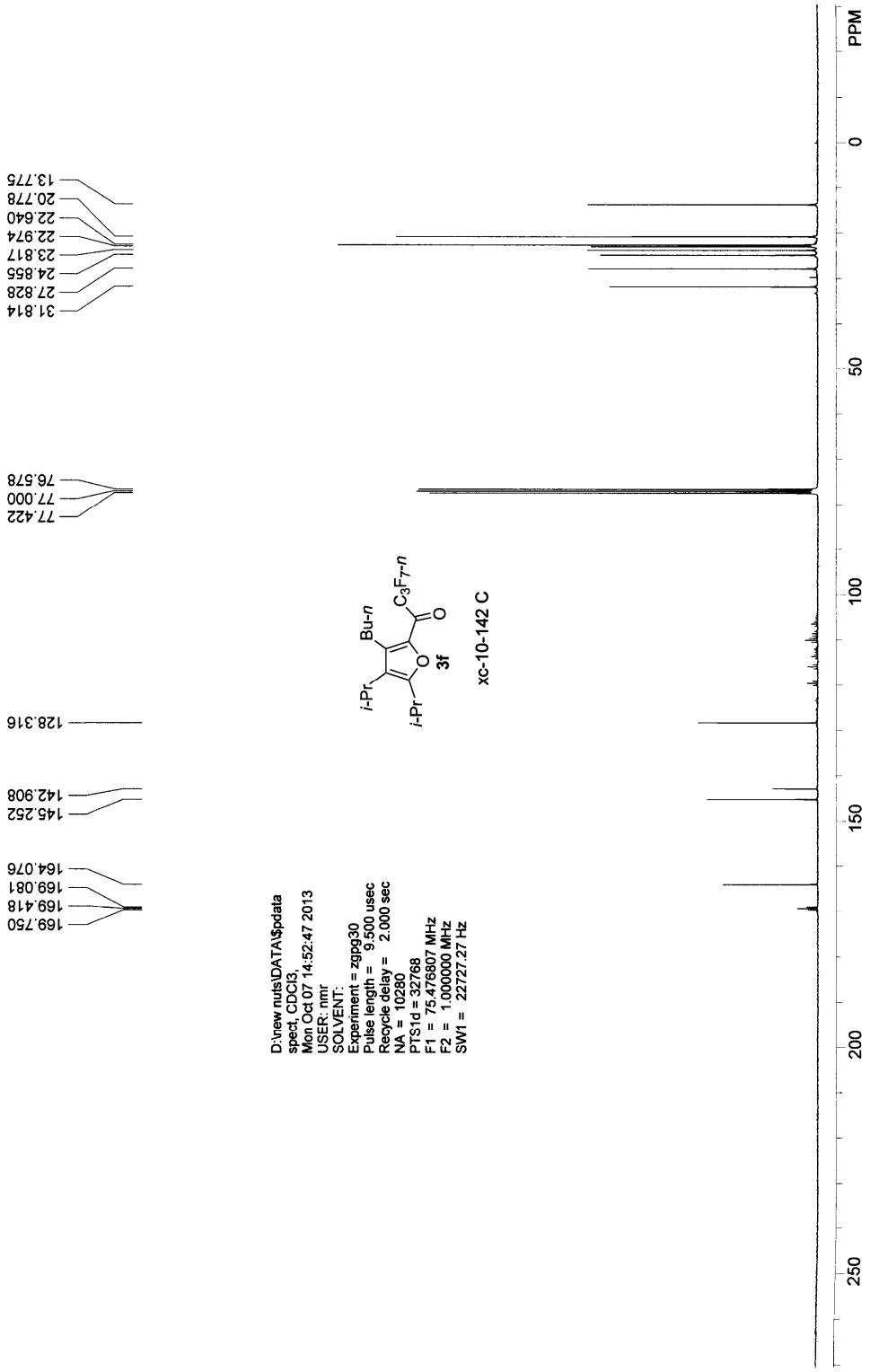
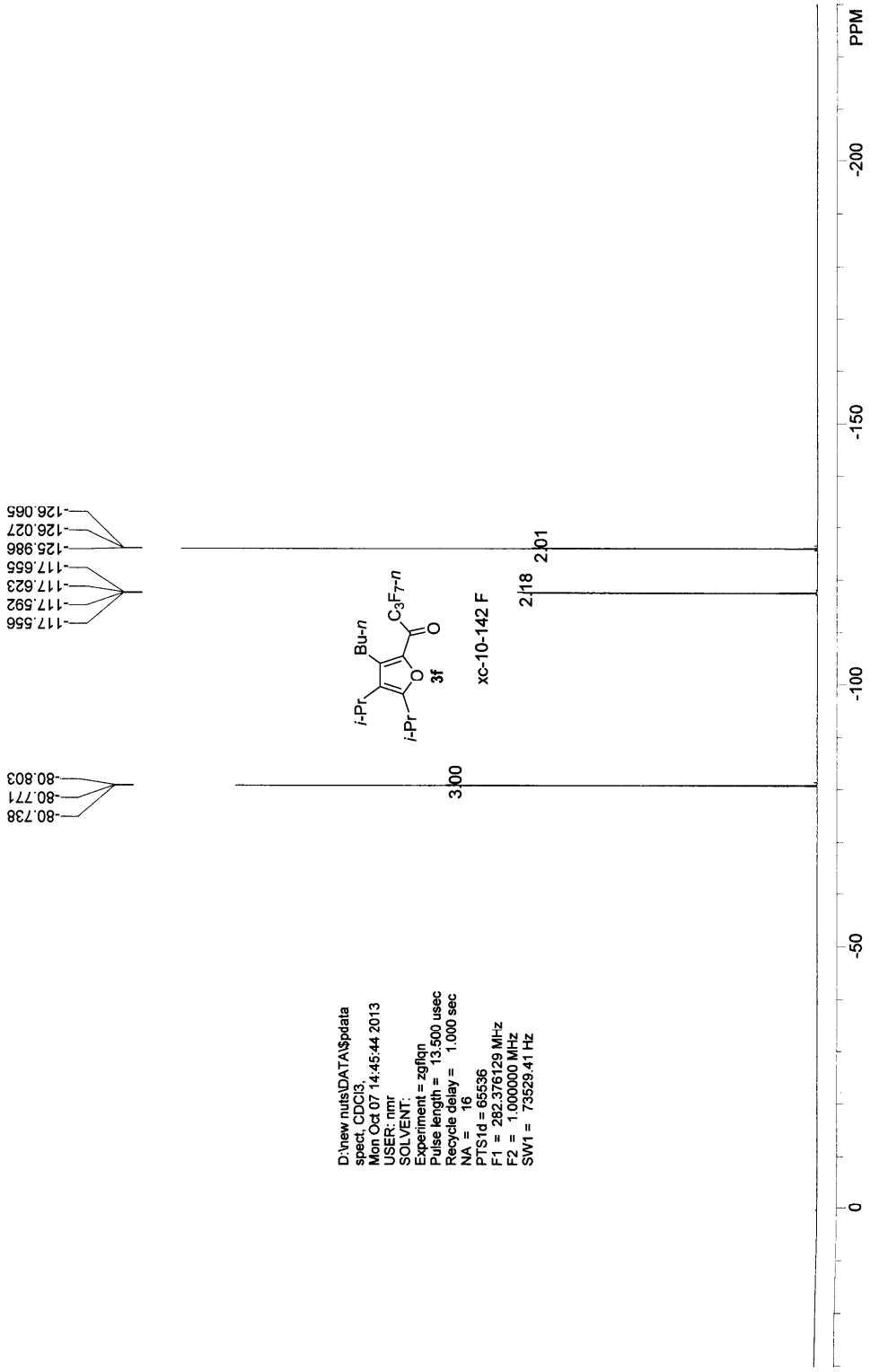
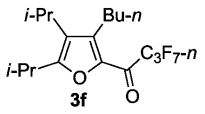
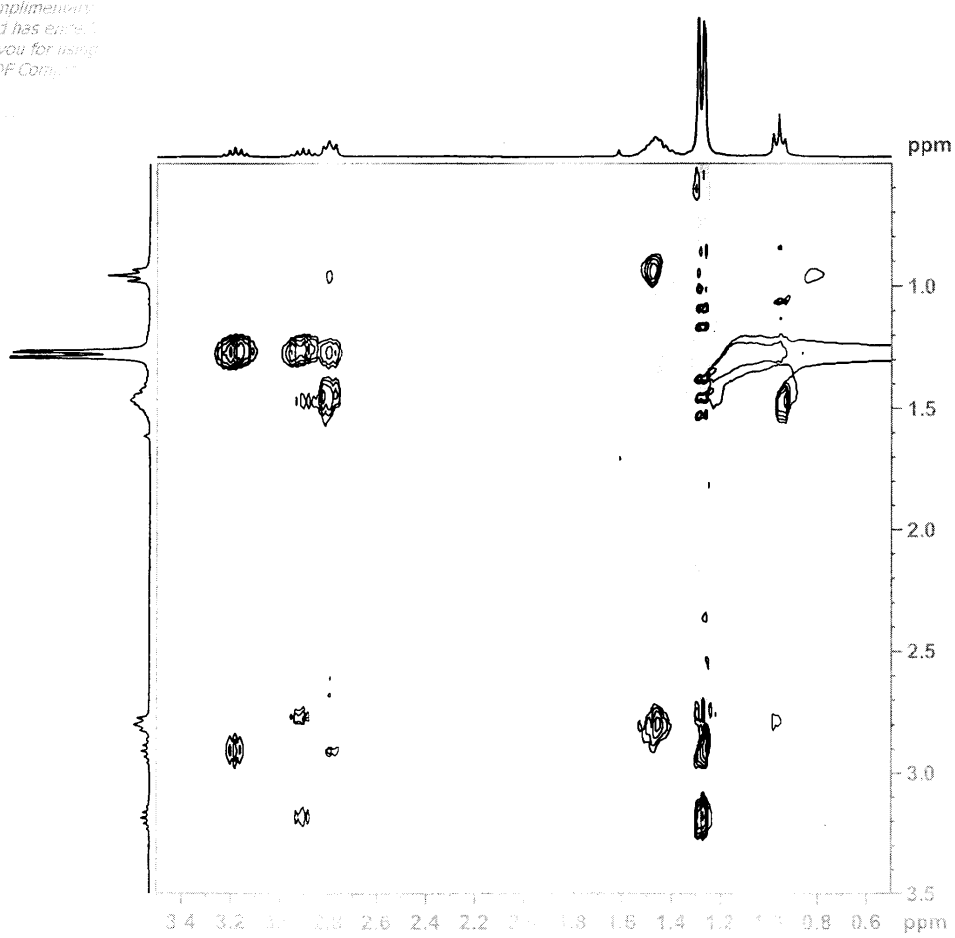
3.136
3.159
3.183
3.205
3.229
3.261
7.269
12.00
4.24
3.19
1.88
1.00
1.02
Bu-n
C3F7-n
i-Pr
i-Pr
O
O
3f
xc-10-142
PPM
0
1
2
3
4
5
6
7
8


169.750
169.418
169.081
164.076
145.252
142.908
128.316
77.422
77.000
76.578
31.814
27.828
24.855
23.817
22.974
22.640
20.778
13.775
PPM
0
50
100
150
200
250
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Oct 07 14:52:47 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10280
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
i-Pr
Bu-n
C3F7-n
i-Pr
O
O
3f
xc-10-142 C

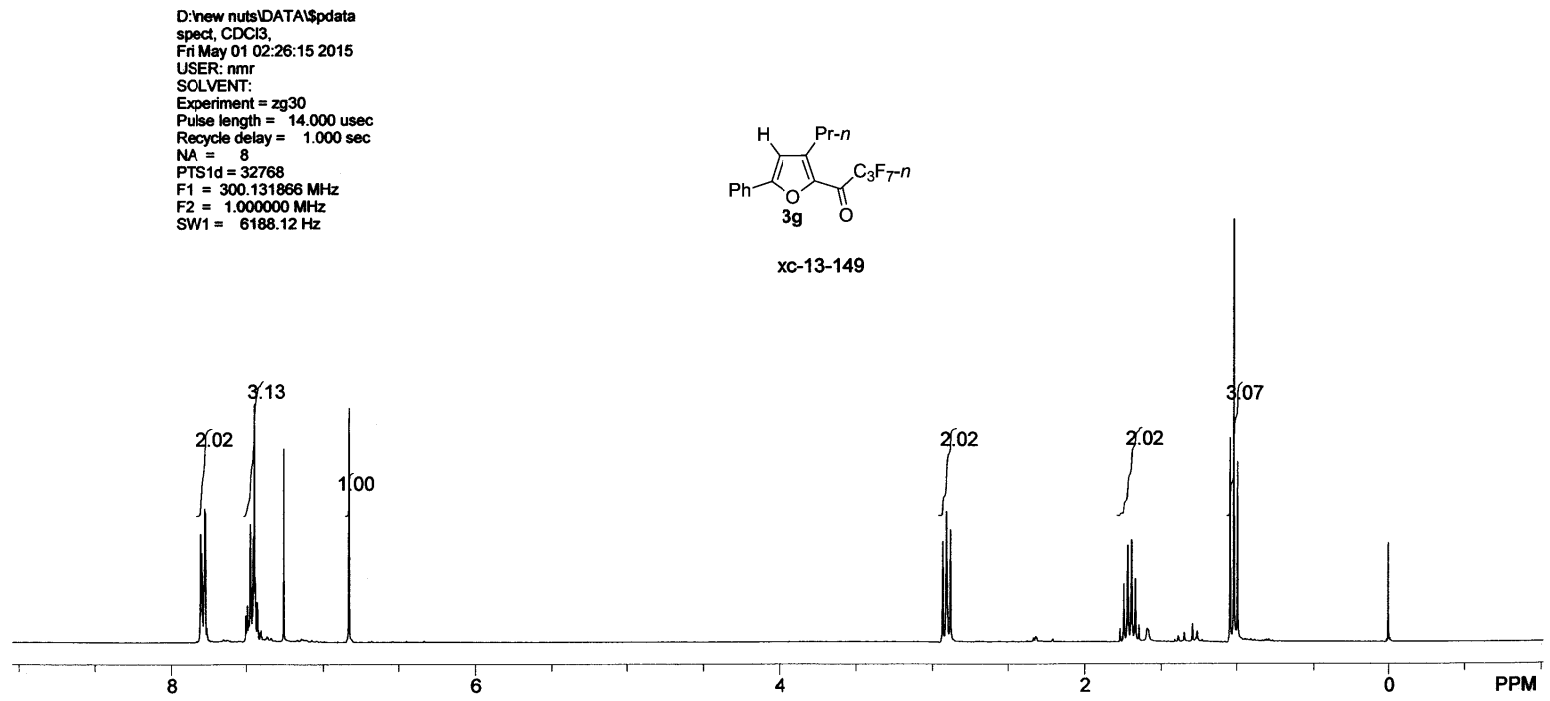

-80.738
-80.771
-80.803
-117.556
-117.592
-117.623
-117.655
-126.986
-126.027
-126.065
3.00
2.18
2.01
Bu-n
i-Pr
i-Pr
C3F7-n
O
O
3f
xc-10-142 F
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Oct 07 14:45:44 2013
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.3776129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0
-50
-100
-150
-200
PPM

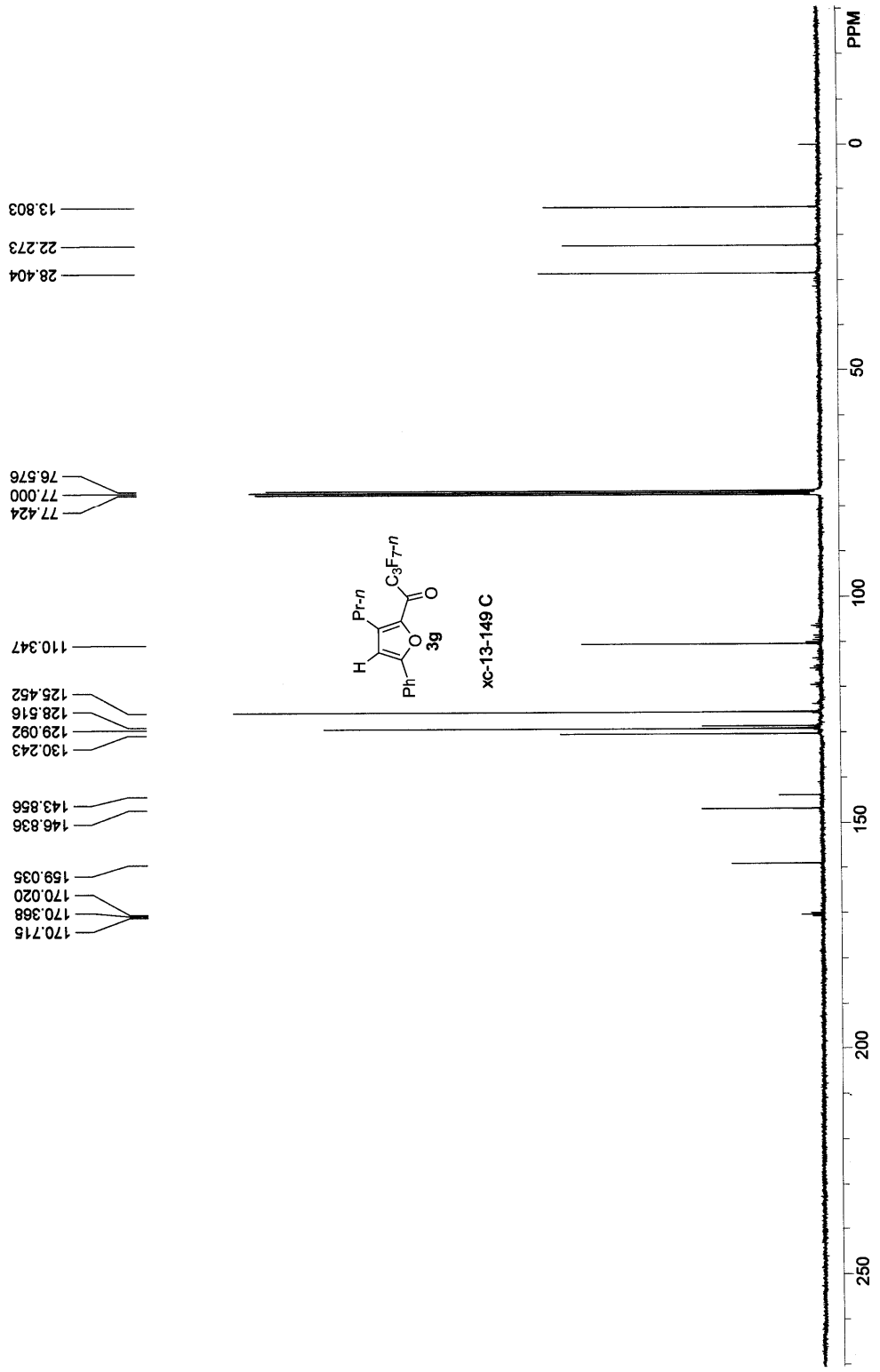


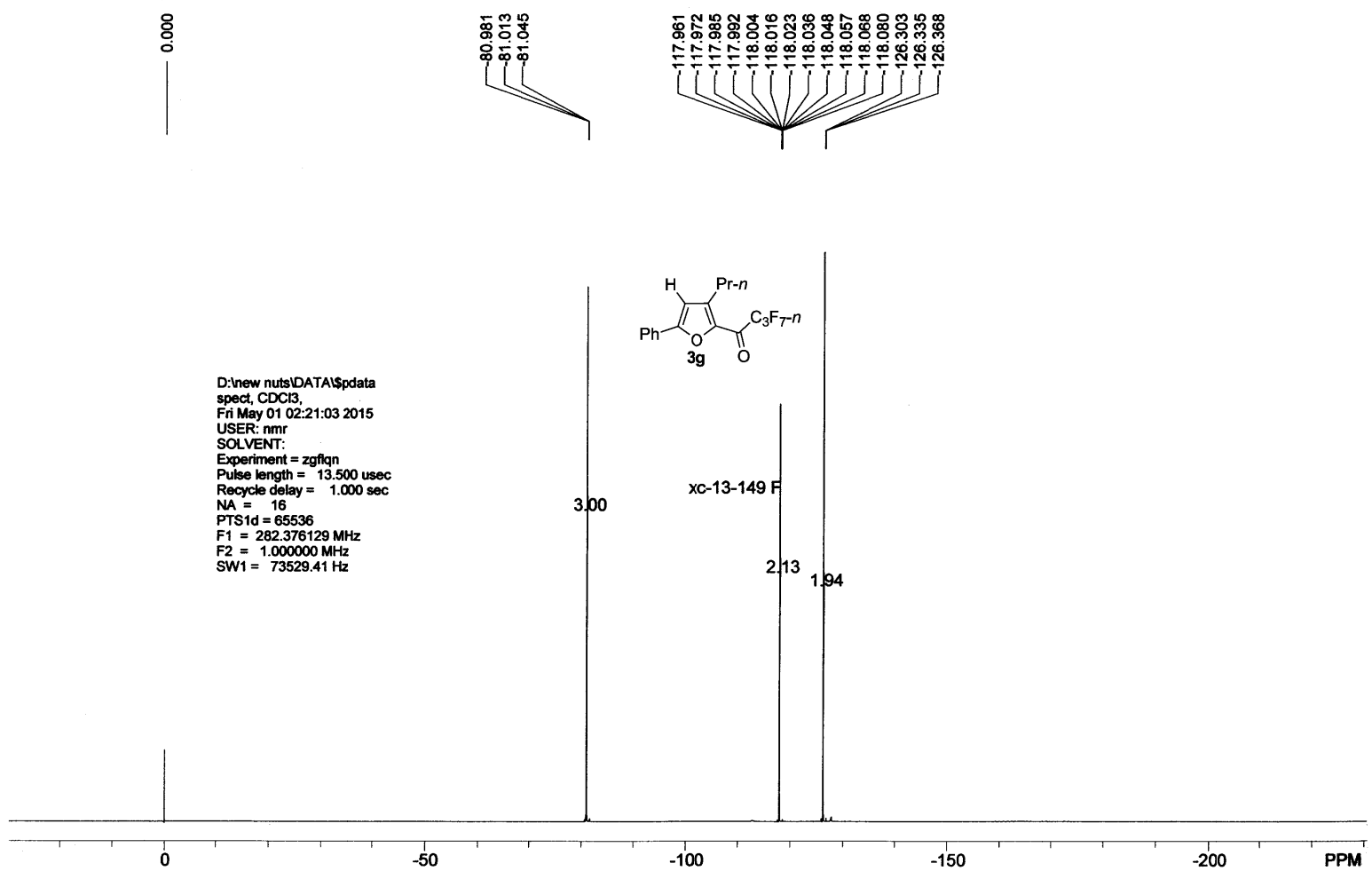
xc-10-142-noe

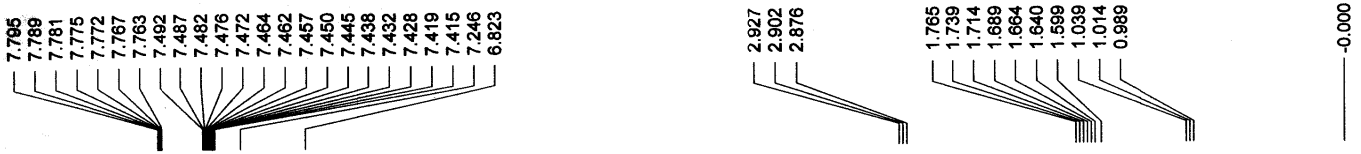



7.800
7.794
7.787
7.780
7.777
7.773
7.768
7.759
7.502
7.497
7.493
7.487
7.483
7.473
7.468
7.461
7.455
7.449
7.443
7.439
7.430
7.426
7.256
6.832
2.932
2.907
2.881
1.768
1.744
1.719
1.693
1.668
1.644
1.591
1.042
1.018
0.993
0.000

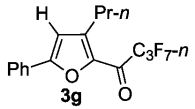

D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri May 01 02:26:15 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
H
Pr-n
Ph
O
C3F7-n
O
3g
xc-13-149
2.02
3.13
1.00
2.02
2.02
3.07
8
6
4
2
0
PPM


13.803
22.273
28.404
76.576
77.000
77.424
110.347
125.452
128.516
129.092
130.243
143.856
146.836
159.035
170.020
170.368
170.715
Pr-n
C3F7-n
O
H
O
Ph
3g
xc-13-149 C
PPM
0
50
100
150
200
250


0.000
80.981
81.013
81.045
117.961
117.972
117.985
117.992
118.004
118.016
118.023
118.036
118.048
118.057
118.068
118.080
126.303
126.335
126.368
H
Pr-n
Ph
O
C3F7-n
O
3g
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri May 01 02:21:03 2015
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
3.00
xc-13-149 F
2.13
1.94
0
-50
-100
-150
-200
PPM

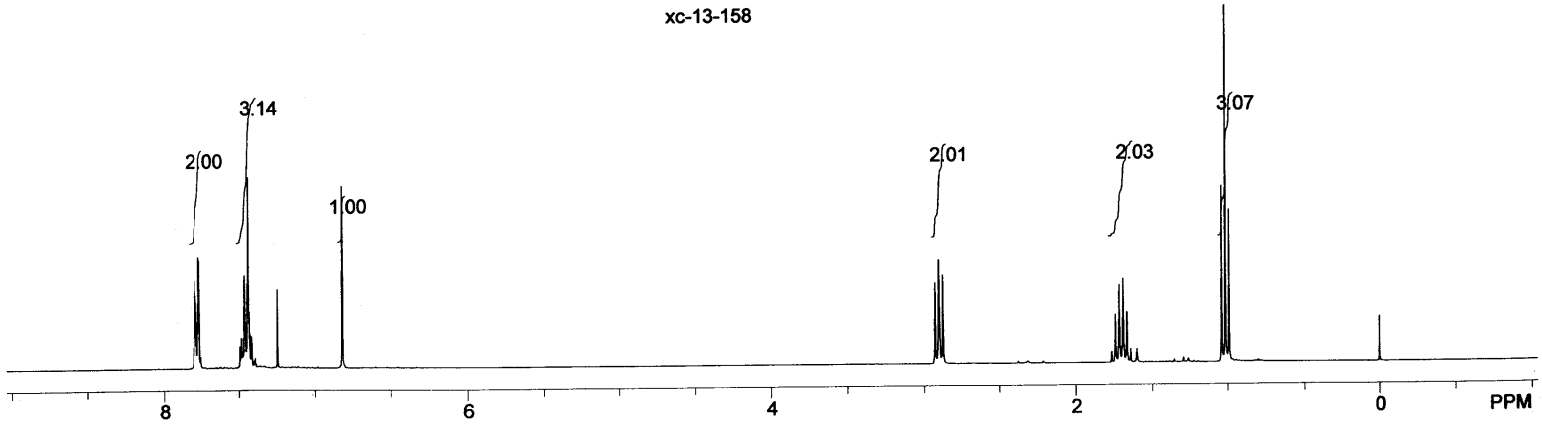


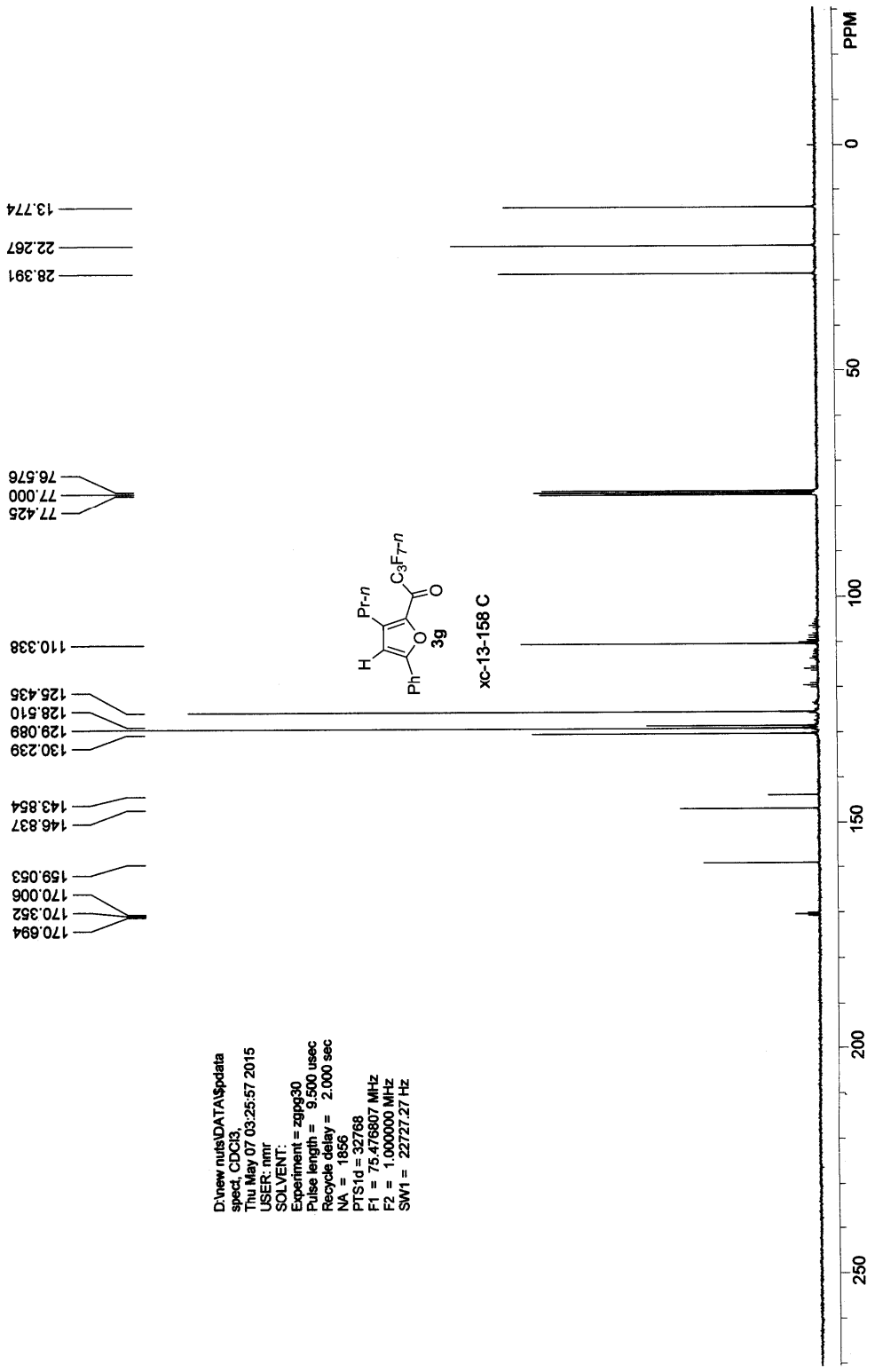
D:\new nuts\DATA\$pdata
spect, CDCl3,
Wed May 06 14:38:40 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

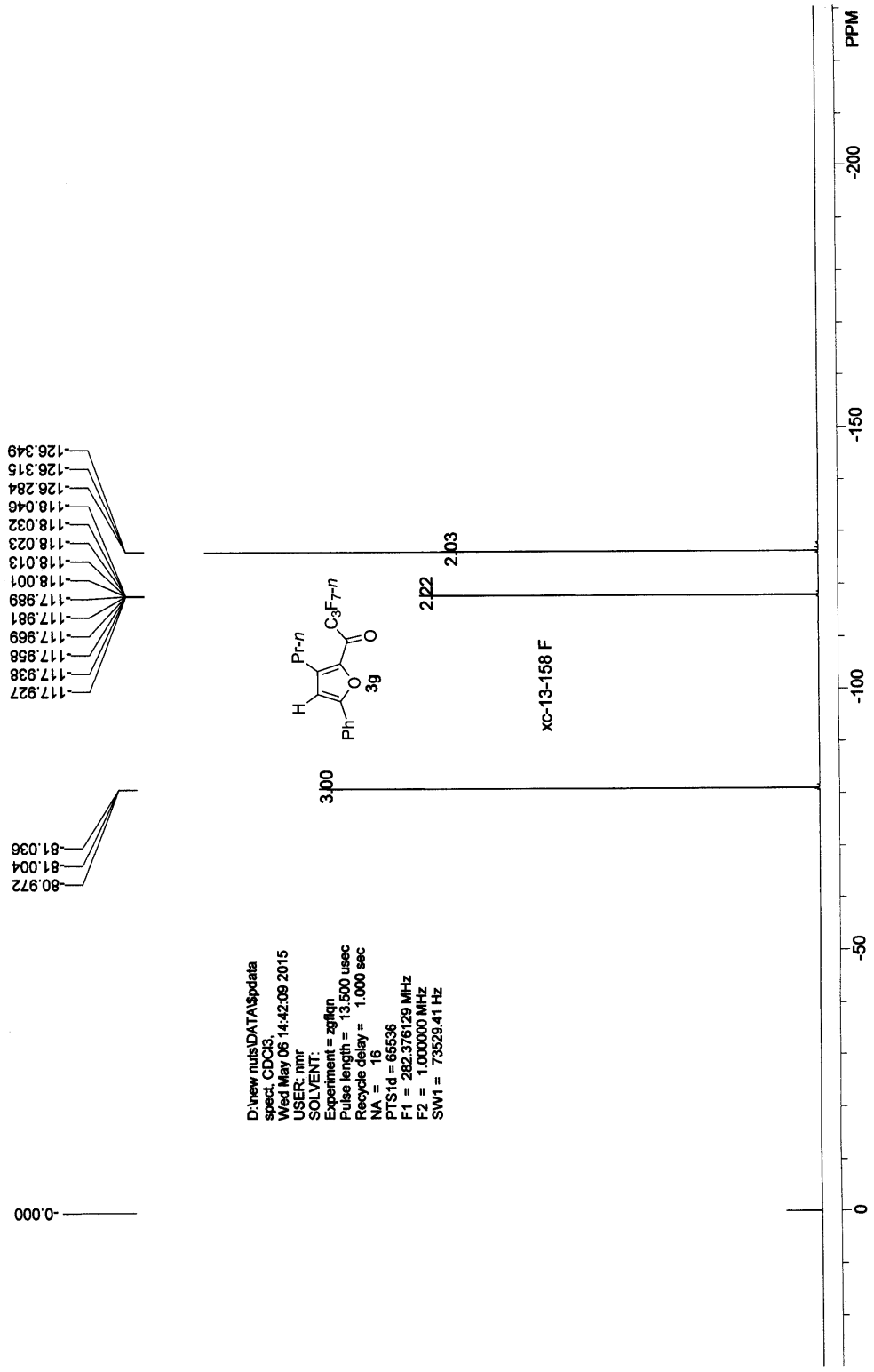


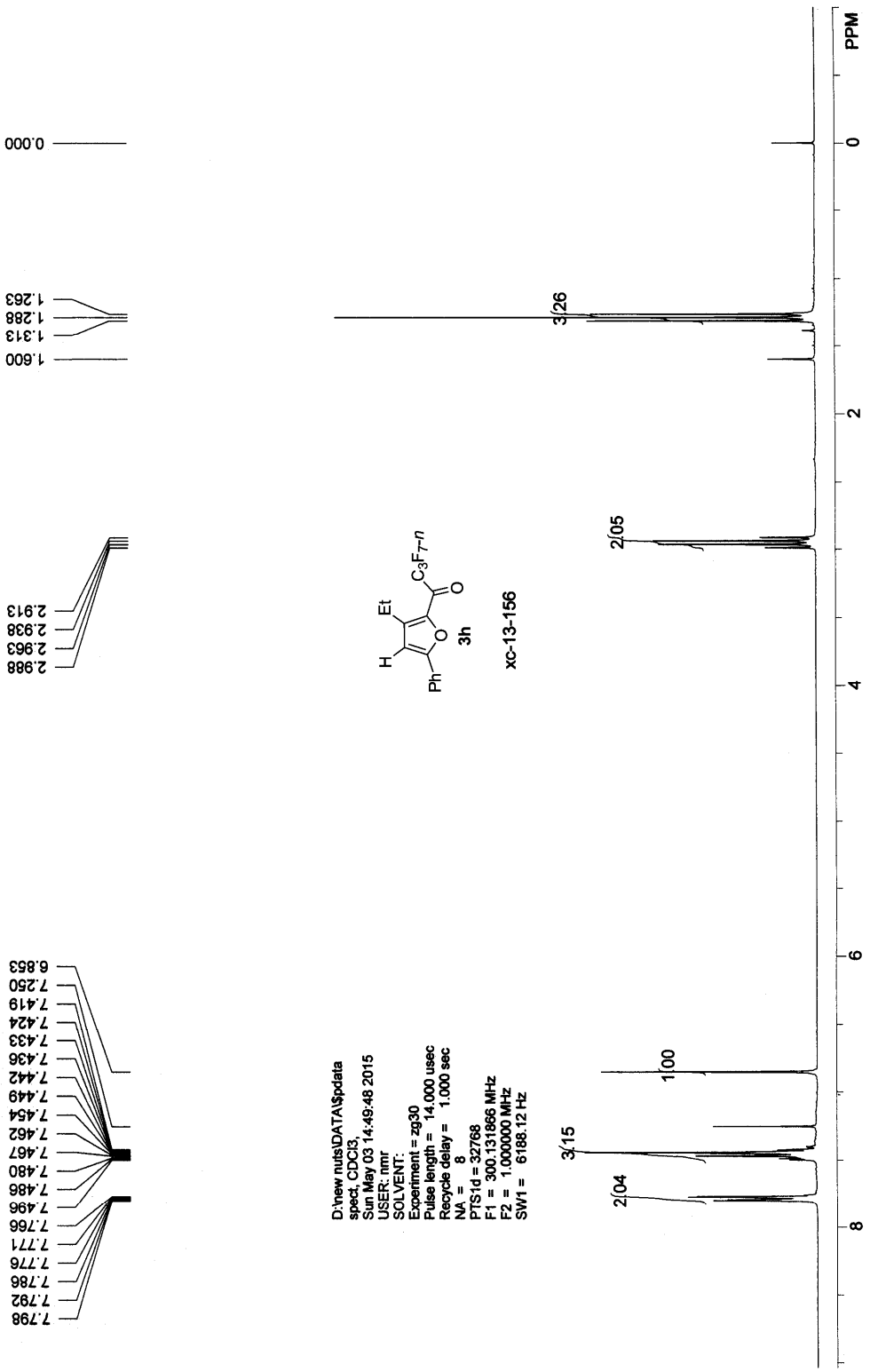
3g

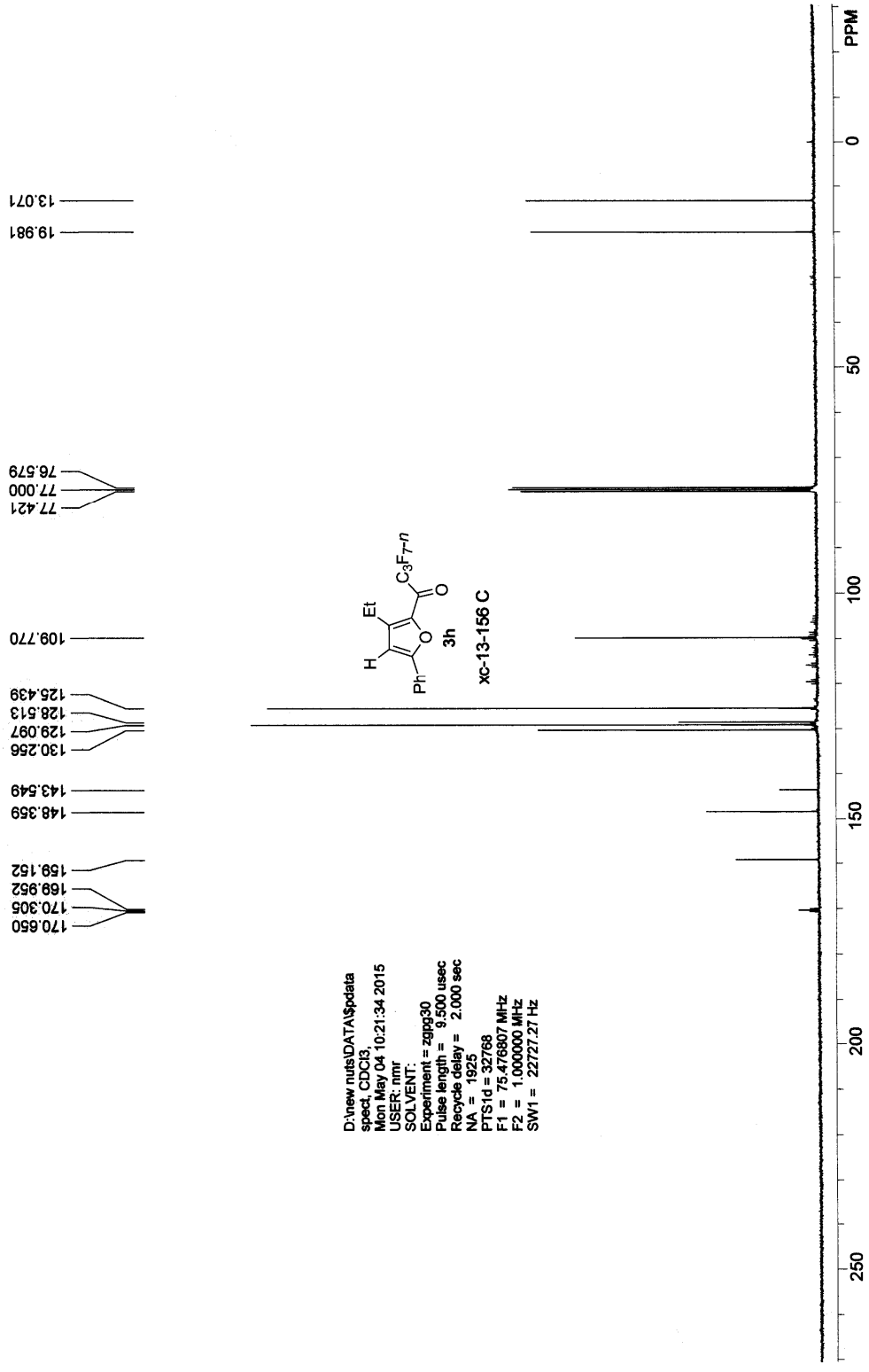
xc-13-158

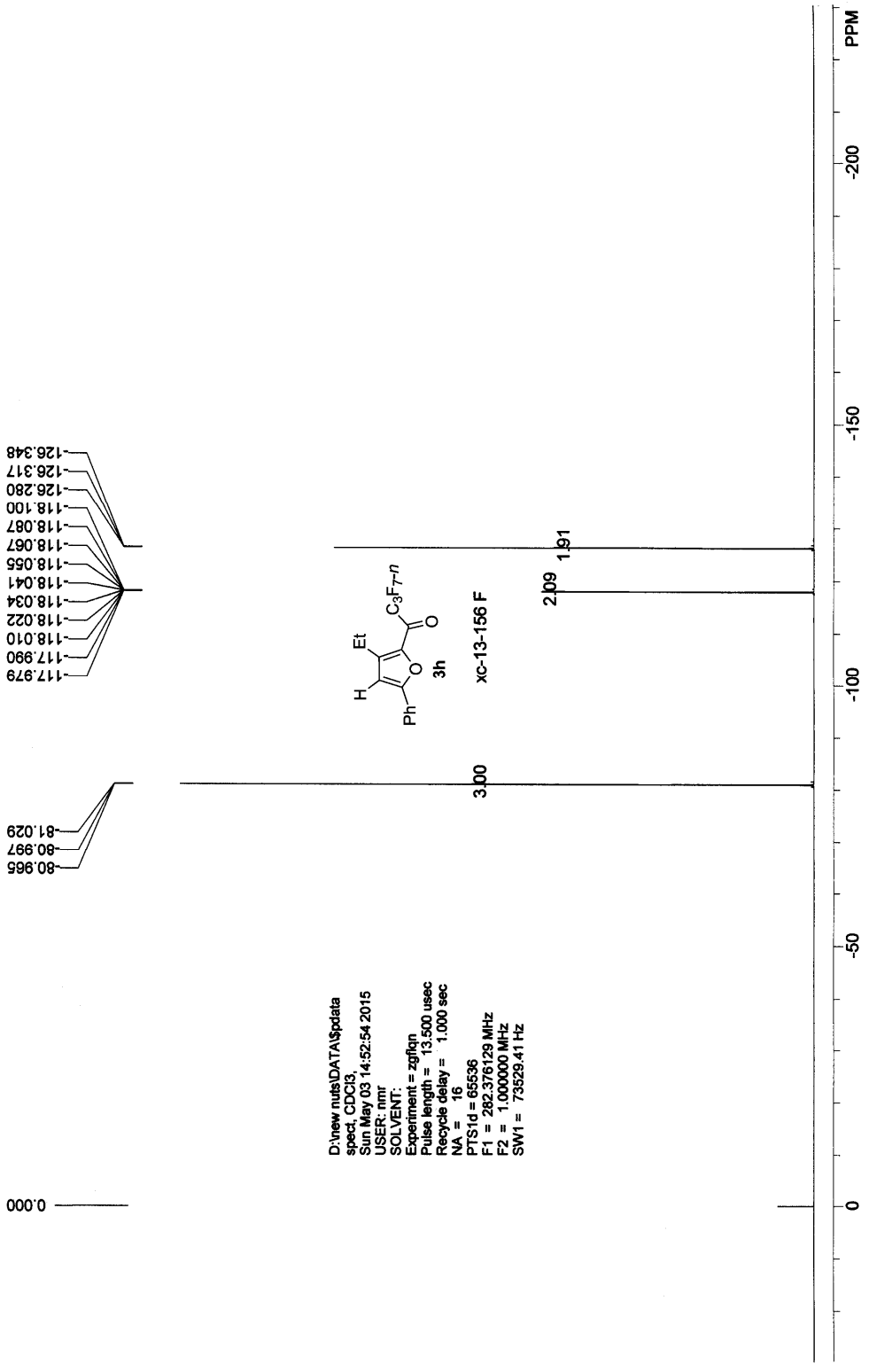


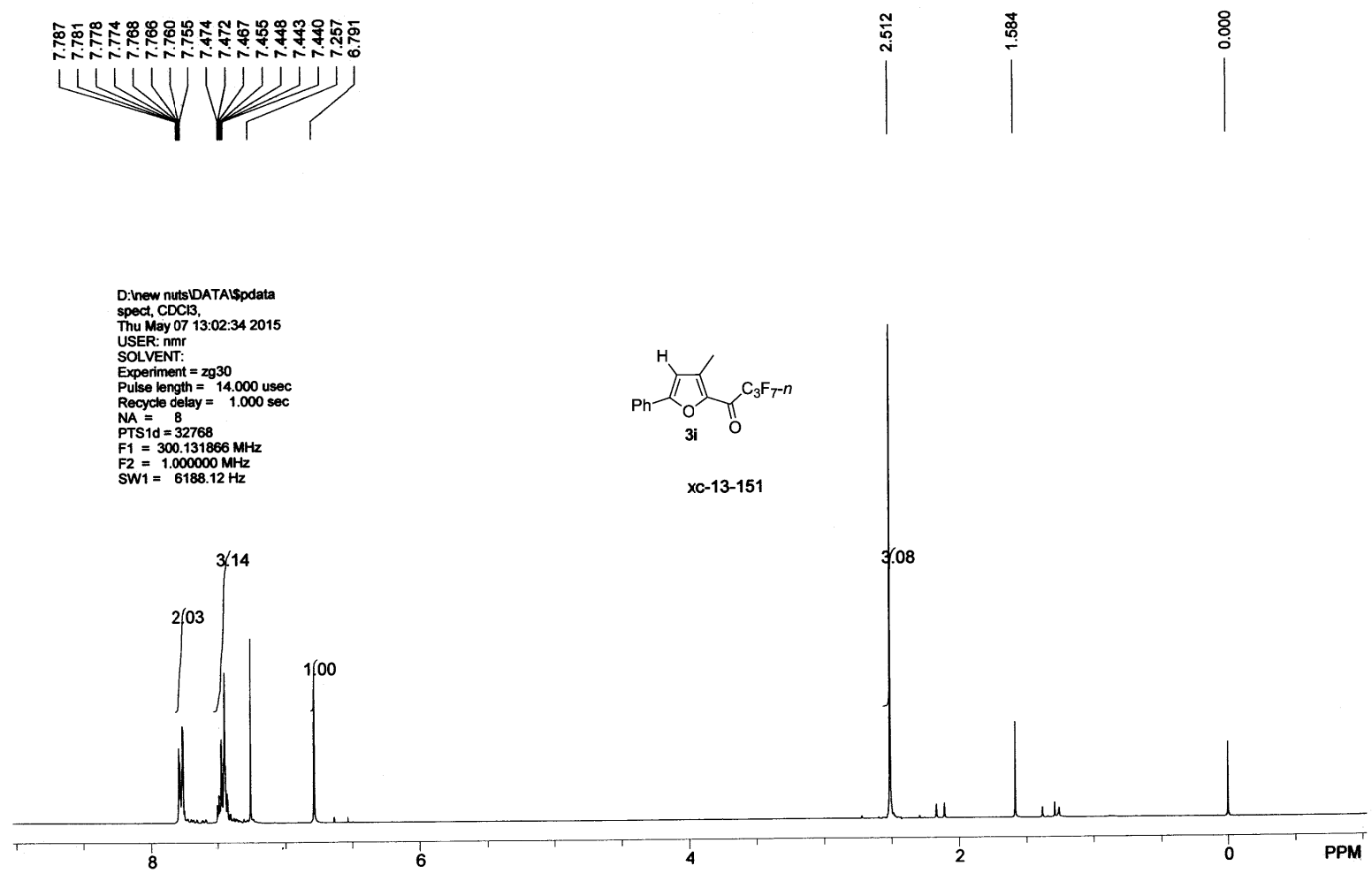

13.774
22.267
28.391
76.576
77.000
77.425
110.338
125.435
128.510
129.089
130.239
143.854
146.837
159.053
170.006
170.352
170.694
PPM
0
50
100
150
200
250
Pr-n
C3F7-n
H
Ph
O
3g
xc-13-158 C
D:\new nuts\DATA\$pdata
sped, CDCl3,
Thu May 07 03:25:57 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1856
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

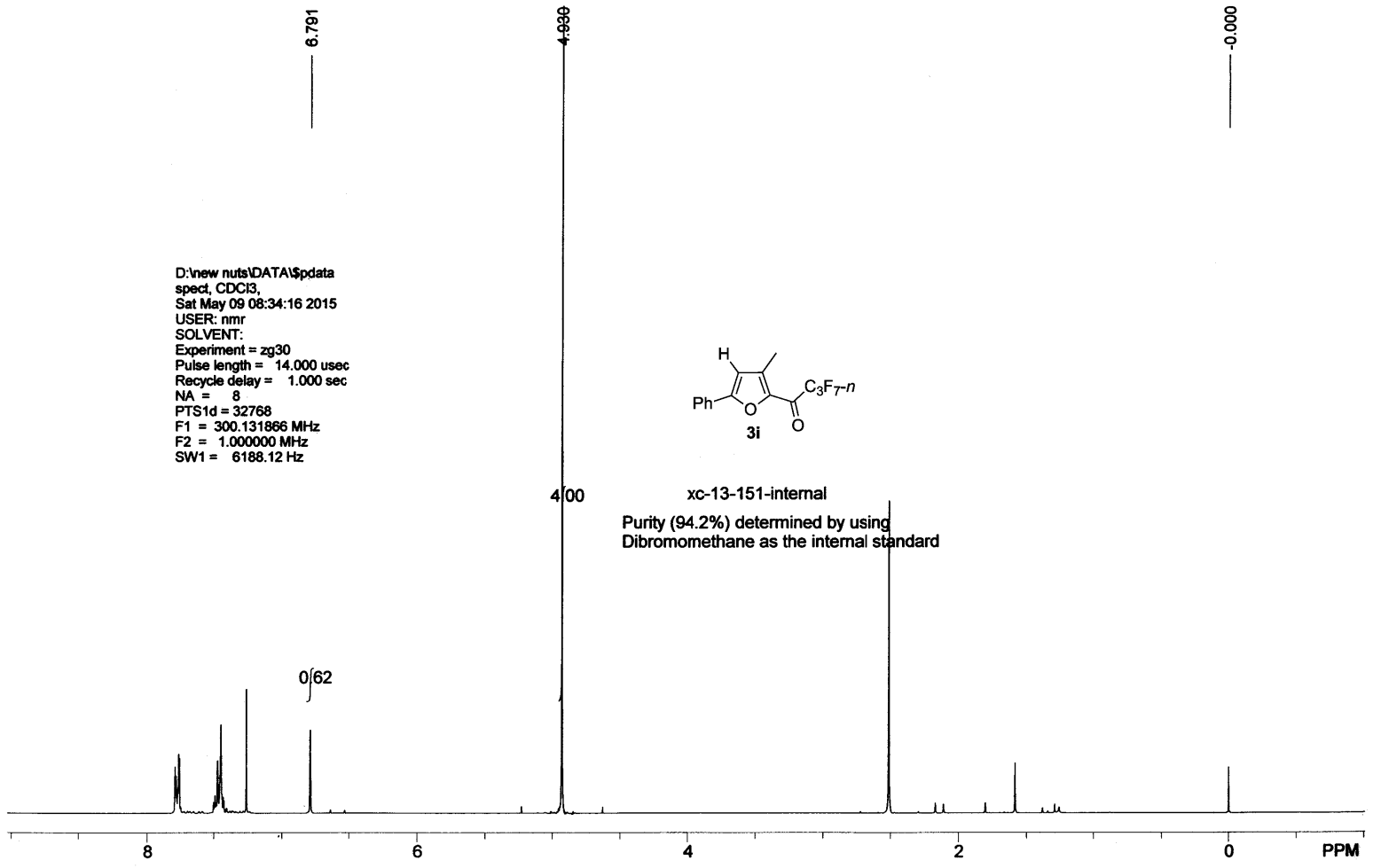

-0.000
80.972
81.004
81.036
117.927
117.938
117.958
117.969
117.981
117.989
118.001
118.013
118.023
118.032
118.046
126.284
126.315
126.349
3.00
2.22
2.03
Pr-n
C3F7-n
O
H
O
Ph
3g
xc-13-158 F
D:\new nuts\DATA\$pdata
sped, CDCl3,
Wed May 06 14:42:09 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 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
-50
-100
-150
-200
PPM

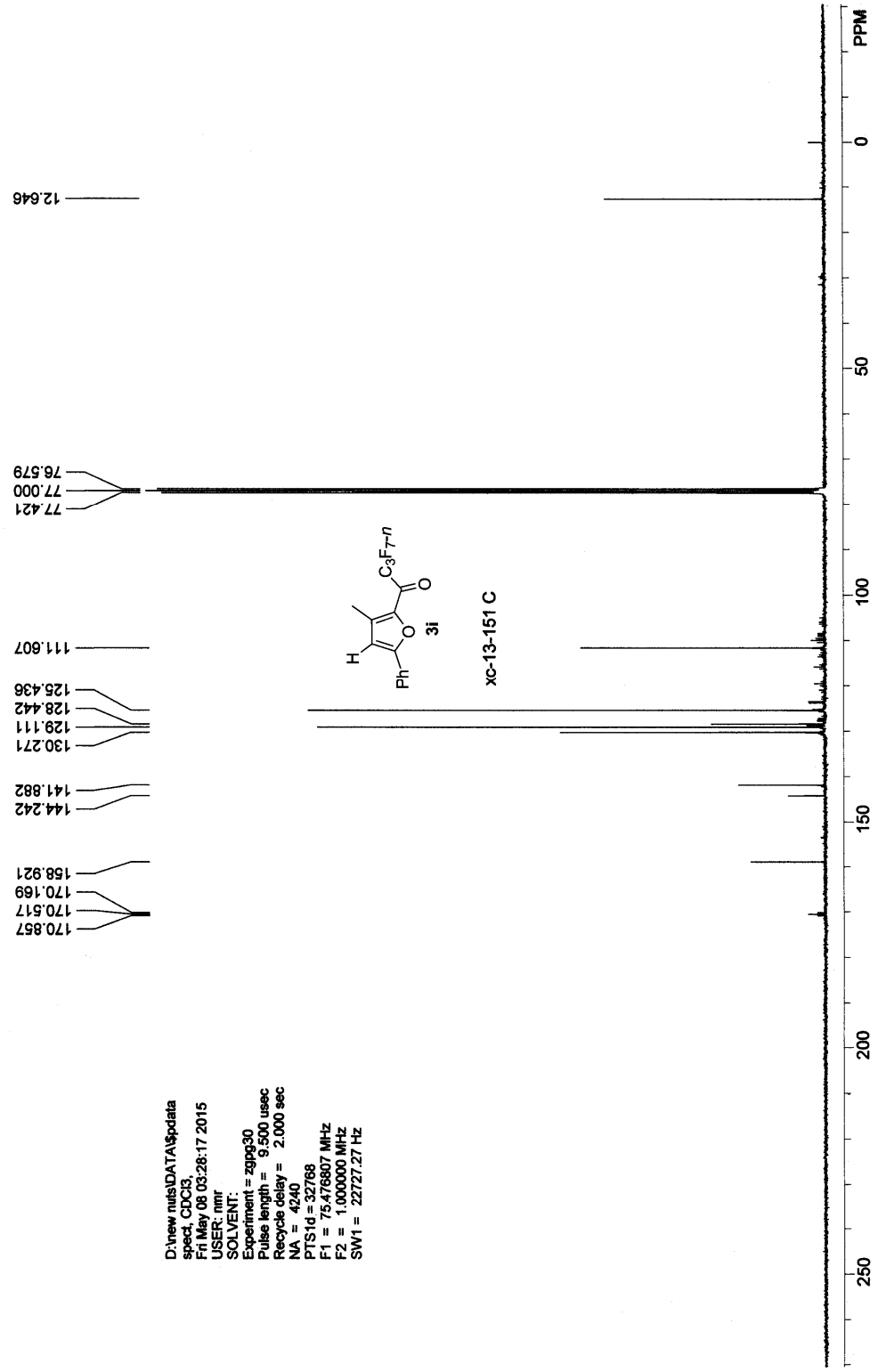

0.000
1.263
1.288
1.313
1.600
2.913
2.938
2.963
2.988
6.853
7.250
7.419
7.424
7.433
7.436
7.442
7.449
7.454
7.462
7.467
7.480
7.486
7.496
7.766
7.771
7.776
7.786
7.792
7.798
D:\new nuts\DATA\$pdata
sped, CDCl3,
Sun May 03 14:49:48 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Et
H
Ph
O
O
C_3F_{7-n}
3h
xc-13-156
3.15
2.04
1.00
2.05
3.26
PPM
8
6
4
2
0

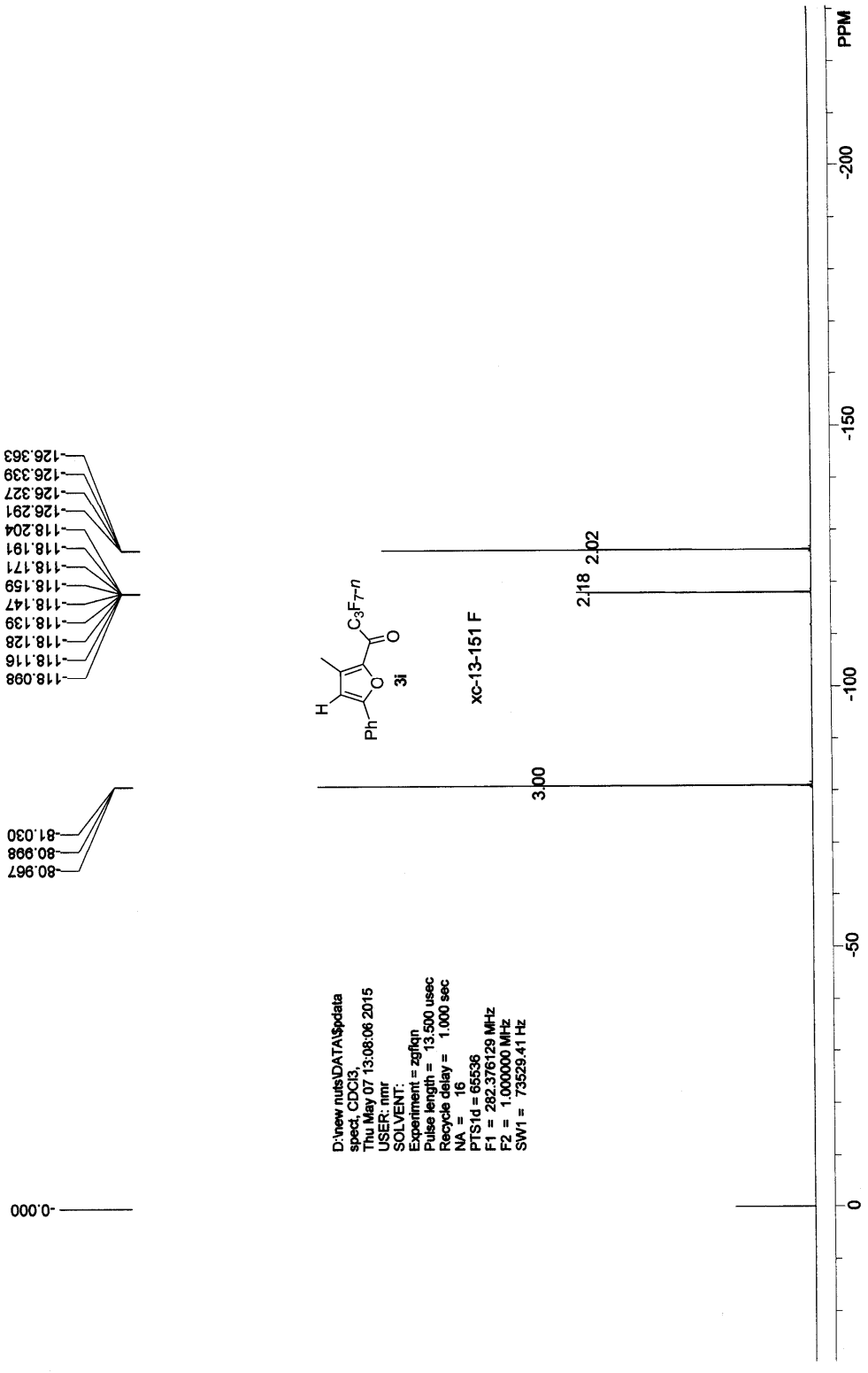

13.071
19.981
76.579
77.000
77.421
109.770
125.439
128.513
129.097
130.256
143.549
148.359
158.152
169.952
170.305
170.650
D:\new nuts\DATA\5pdata
spect, CDCl3,
Mon May 04 10:21:34 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1925
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Et
H
Ph
O
O
C3F7-n
3h
xc-13-156 C
PPM
0
50
100
150
200
250

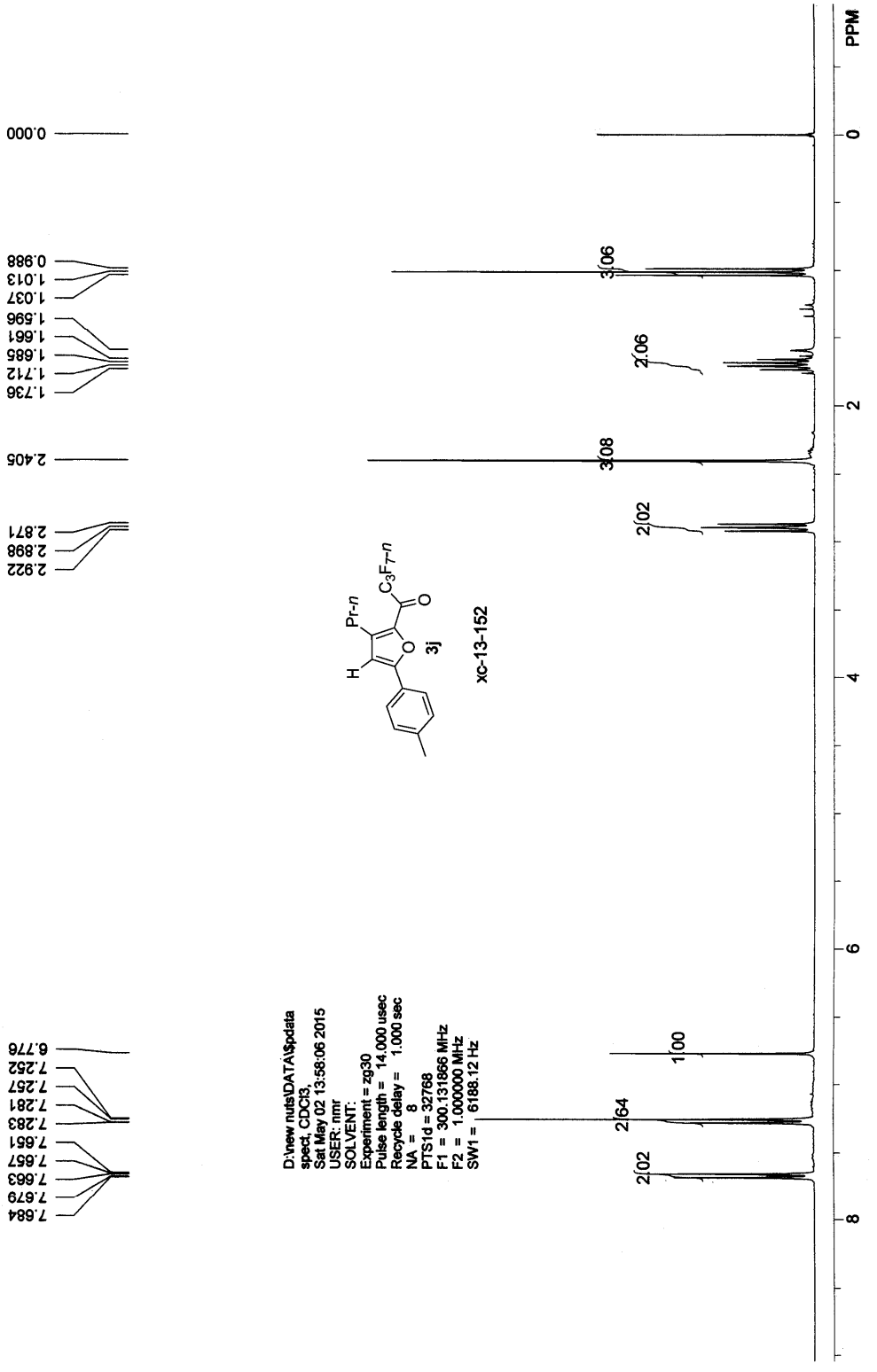

0.000
80.965
80.997
81.029
117.979
117.990
118.010
118.022
118.034
118.041
118.055
118.067
118.087
118.100
126.280
126.317
126.348
3.00
2.09
1.91
xc-13-156 F
3h
Et
H
Ph
O
O
C_3F_{7-n}
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sun May 03 14:52:54 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 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
-50
-100
-150
-200
PPM

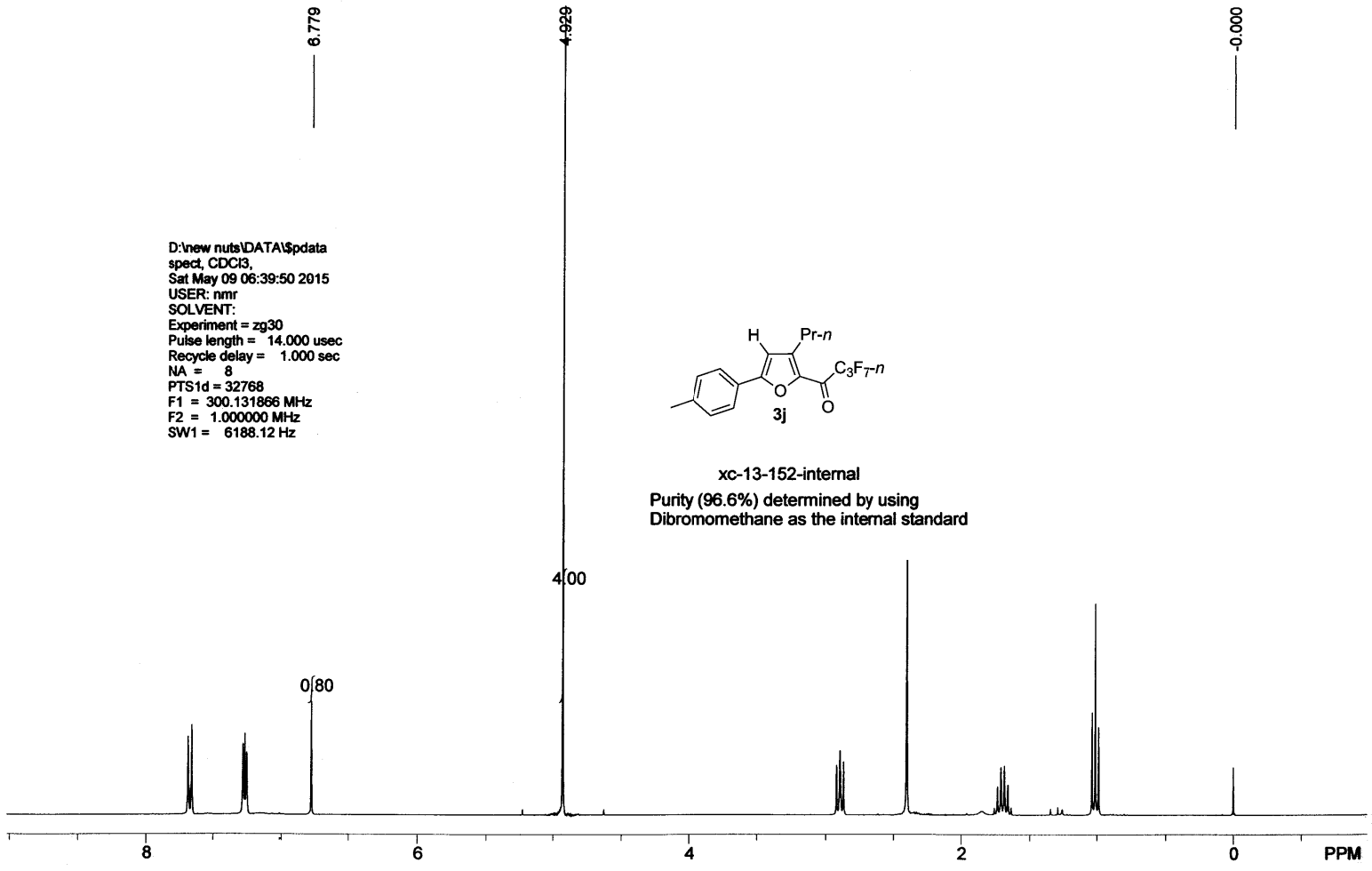

7.787
7.781
7.778
7.774
7.768
7.766
7.760
7.755
7.474
7.472
7.467
7.455
7.448
7.443
7.440
7.257
6.791
2.512
1.584
0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Thu May 07 13:02:34 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
H
Ph
O
O
C_3F_7-n
3i
xc-13-151
2.03
3.14
1.00
3.08
8
6
4
2
0
PPM

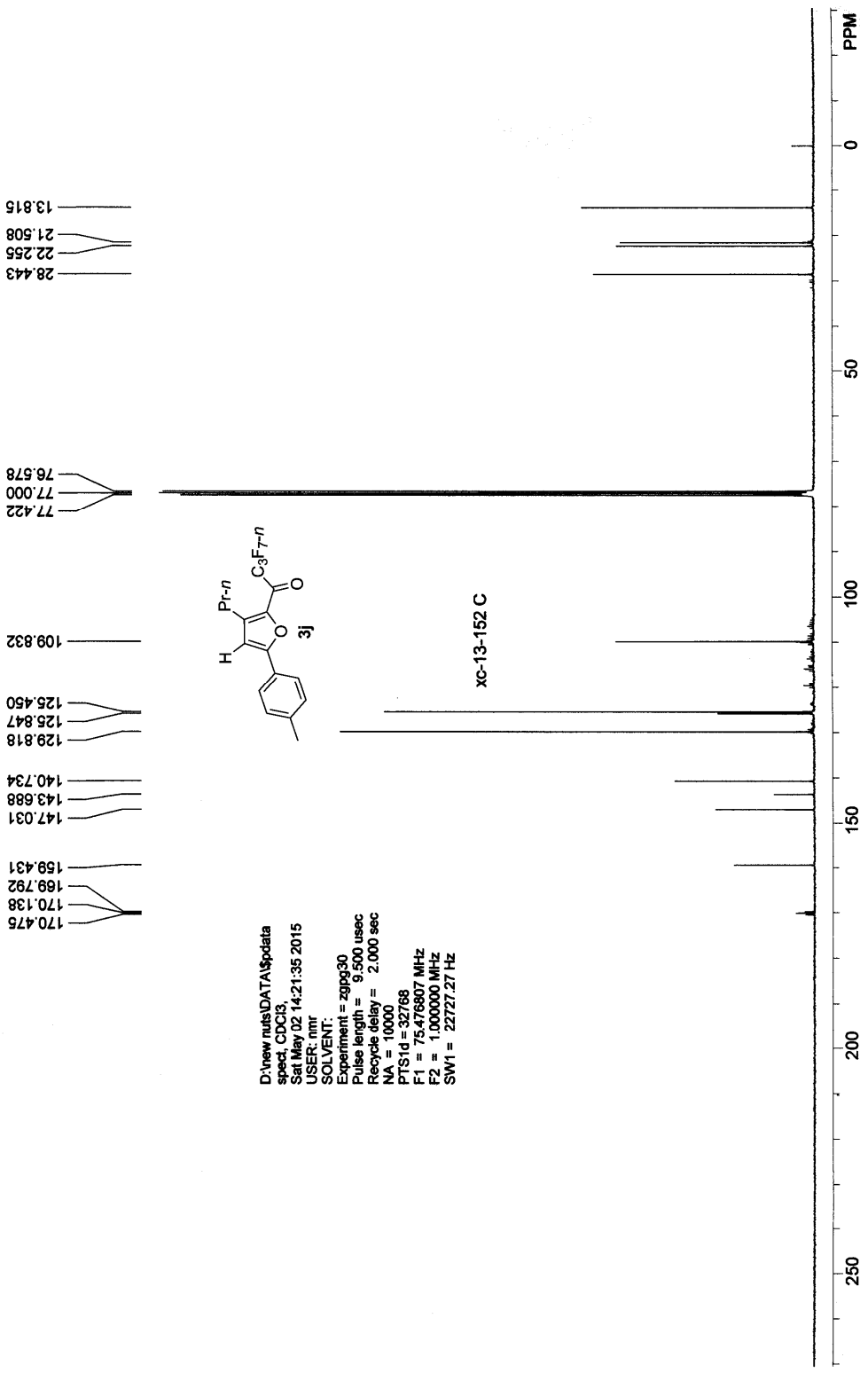

6.791
4.000
0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat May 09 08:34:16 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
H
Ph
O
C3F7-n
O
3i
4.00
xc-13-151-internal
Purity (94.2%) determined by using
Dibromomethane as the internal standard
0.62
8
6
4
2
0
PPM

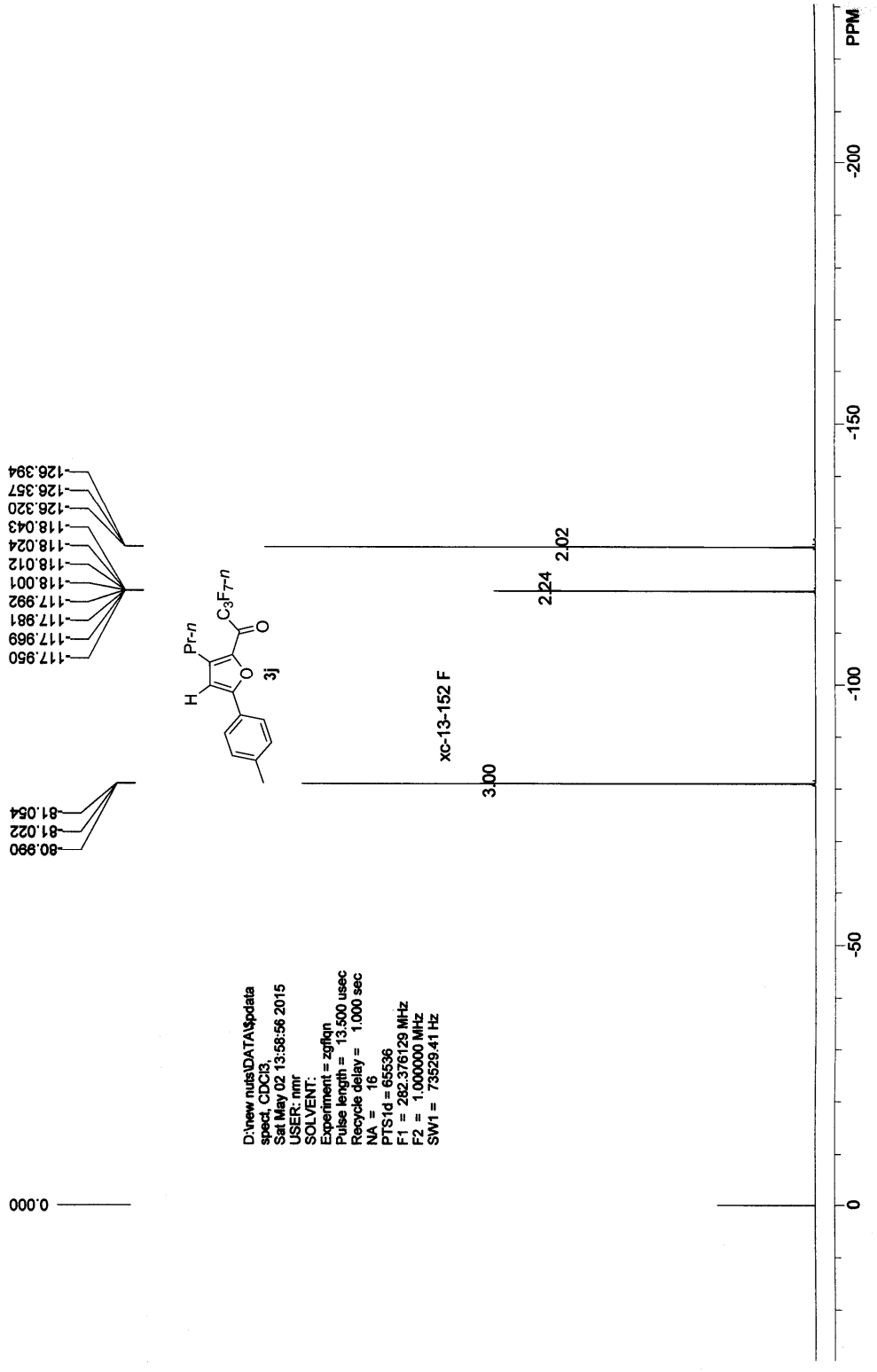

12.646
76.579
77.000
77.421
111.607
125.436
128.442
129.111
130.271
141.882
144.242
158.921
170.169
170.517
170.857
D:\new nuts\DATA\5pdata
sped, CDCl3,
Fri May 08 03:28:17 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 4240
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
xc-13-151 C
3i
PPM
0
50
100
150
200
250

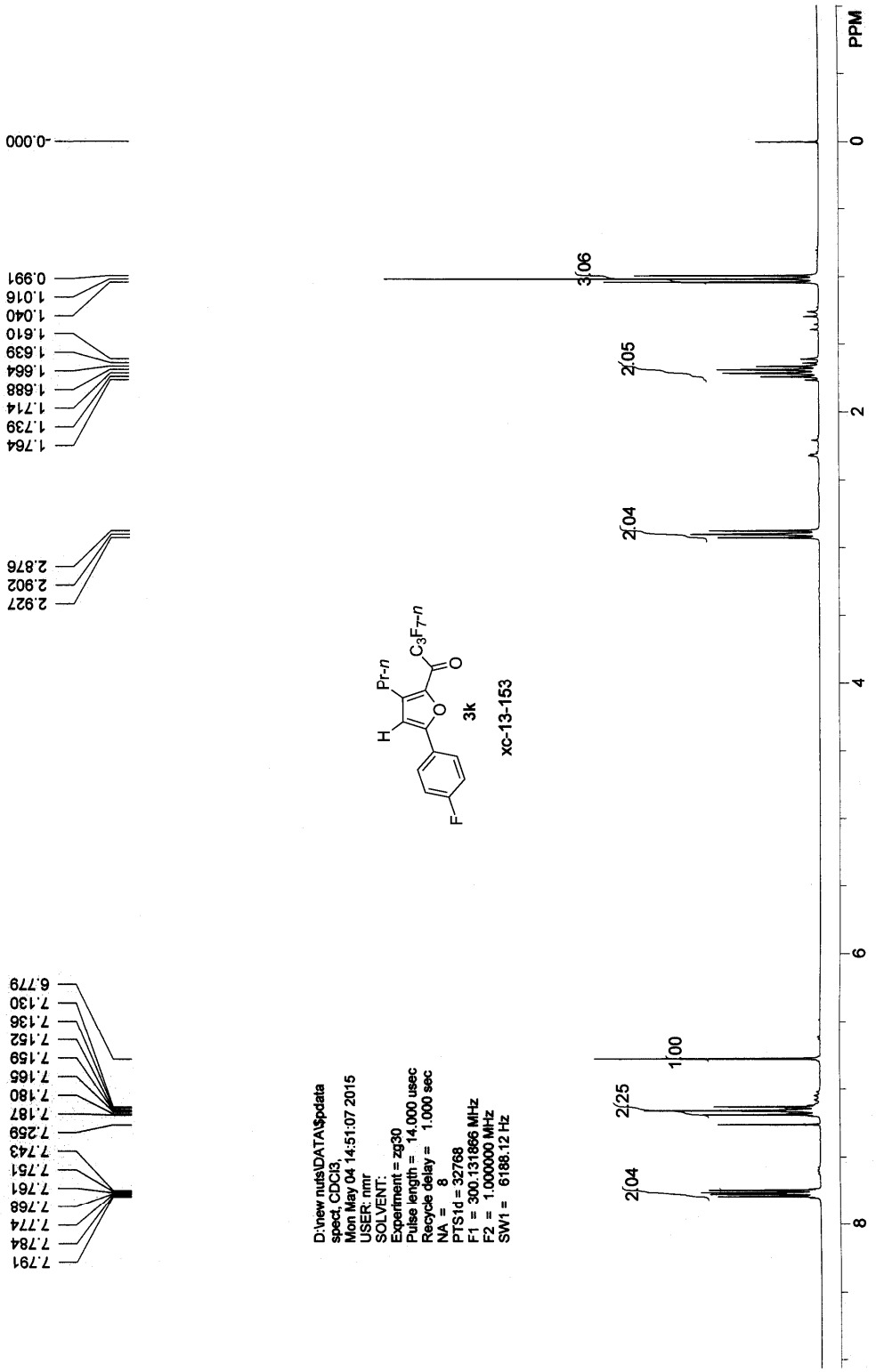

126.363
126.339
126.327
126.291
118.204
118.191
118.171
118.159
118.147
118.139
118.128
118.116
118.098
81.030
80.998
80.967
0.000
D:\new nuts\DATA\$pdata
sped, CDCl3,
Thu May 07 13:08:06 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
3.00
2.18
2.02
xc-13-151 F
3i
Ph
H
O
O
C3F7-n
PPM
0
-50
-100
-150
-200

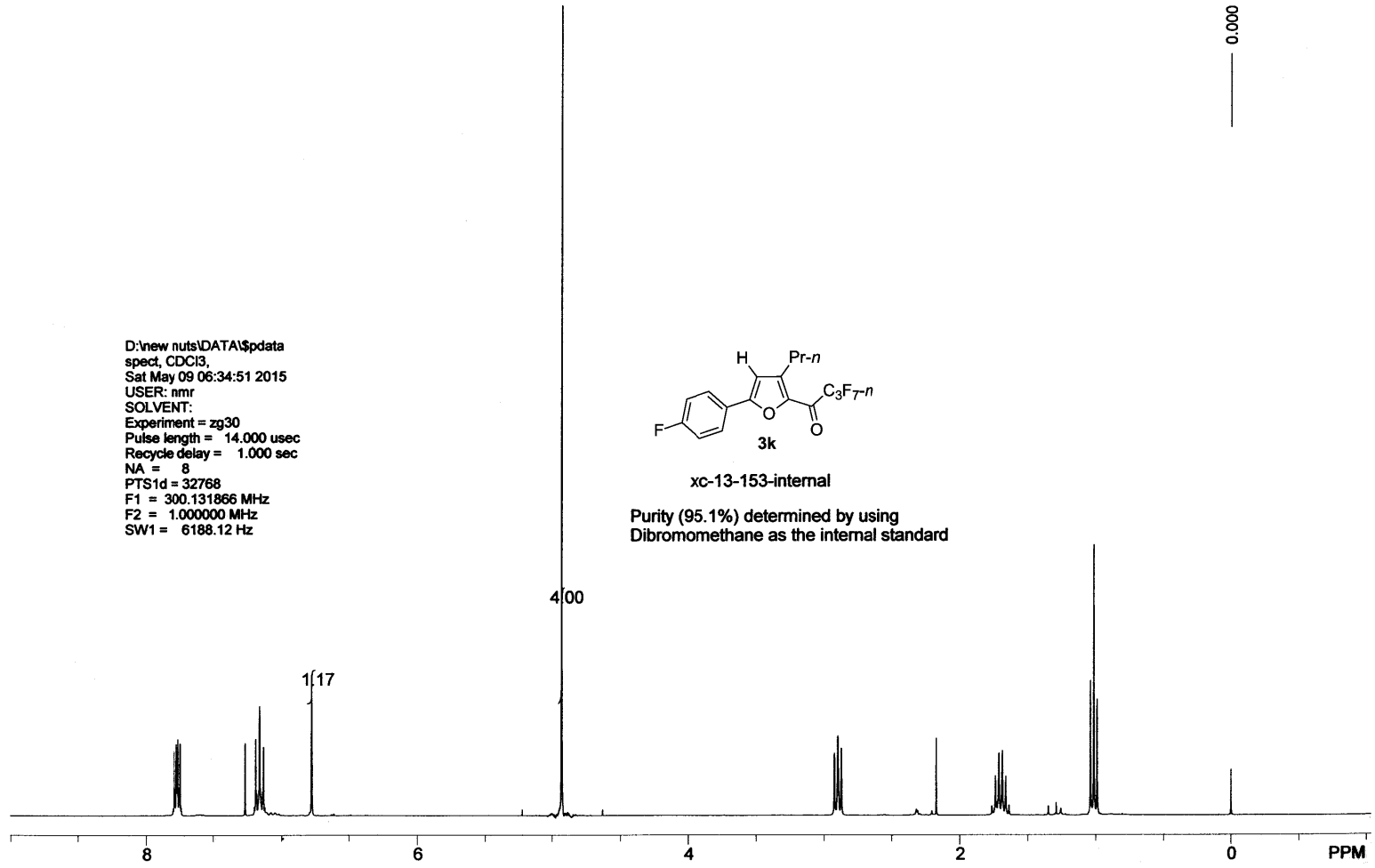

D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat May 02 13:58:06 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
xc-13-152
3j
Pr-n
C3F7-n
H
O
O
PPM
0
2
4
6
8
3.06
2.06
3.08
2.02
2.64
1.00
2.02
0.000
0.988
1.013
1.037
1.696
1.661
1.685
1.712
1.736
2.405
2.871
2.898
2.922
6.776
7.252
7.257
7.281
7.283
7.651
7.657
7.663
7.679
7.684

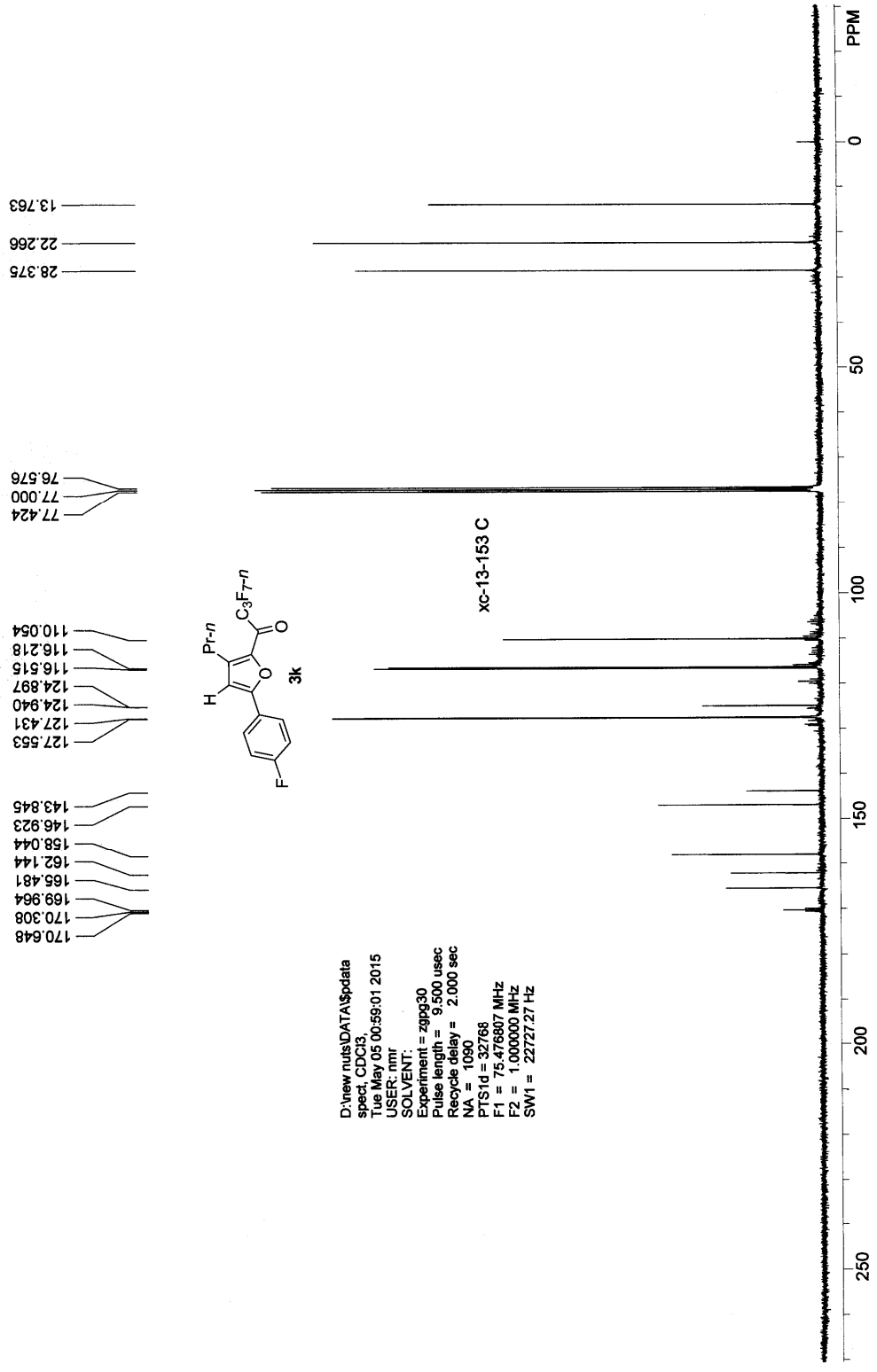

6.779
4.929
-0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat May 09 06:39:50 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
H
Pr-n
C3F7-n
O
O
3j
xc-13-152-internal
Purity (96.6%) determined by using
Dibromomethane as the internal standard
4.00
0.80
8
6
4
2
0
PPM

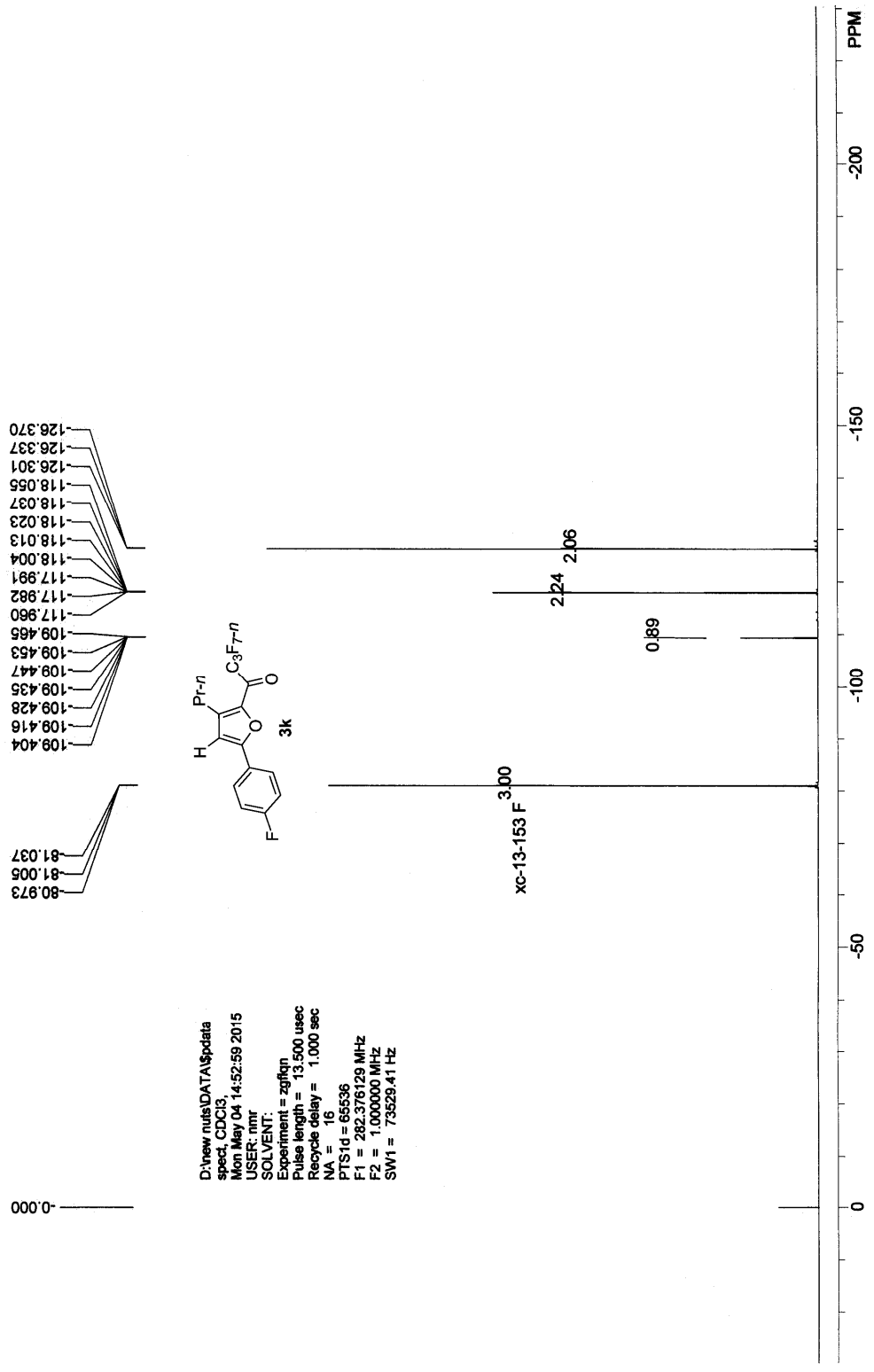

13.815
21.508
22.255
28.443
76.578
77.000
77.422
109.832
125.450
125.847
129.818
140.734
143.688
147.031
159.431
169.792
170.138
170.475
Pr-n
C3F7-n
H
O
O
3j
xc-13-152 C
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat May 02 14:21:35 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 10000
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

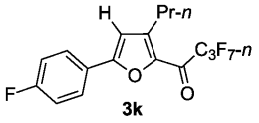

126.394
126.357
126.320
118.043
118.024
118.012
118.001
117.992
117.981
117.969
117.950
81.054
81.022
80.990
0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat May 02 13:58:56 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
xc-13-152 F
3.00
2.24
2.02
Pr-n
C3F7-n
O
O
H
3j
PPM
0
-50
-100
-150
-200


D:\new nuts\DATA\$pdata
spec4, CDCl3,
Mon May 04 14:51:07 2015
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-13-153
3k
Pr-n
C3F7-n
H
O
F
PPM
0
2
4
6
8
3.06
2.05
2.04
1.00
2.25
2.04
0.000
0.991
1.016
1.040
1.610
1.639
1.664
1.688
1.714
1.739
1.764
2.876
2.902
2.927
6.779
7.130
7.136
7.152
7.159
7.165
7.180
7.187
7.259
7.743
7.751
7.761
7.768
7.774
7.784
7.791


D:\new nuts\DATA\$pdata
spect, CDCl3,
Sat May 09 06:34:51 2015
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
H
Pr-n
C3F7-n
F
O
O
3k
xc-13-153-internal
Purity (95.1%) determined by using
Dibromomethane as the internal standard
0.000
4.00
1.17
8
6
4
2
0
PPM

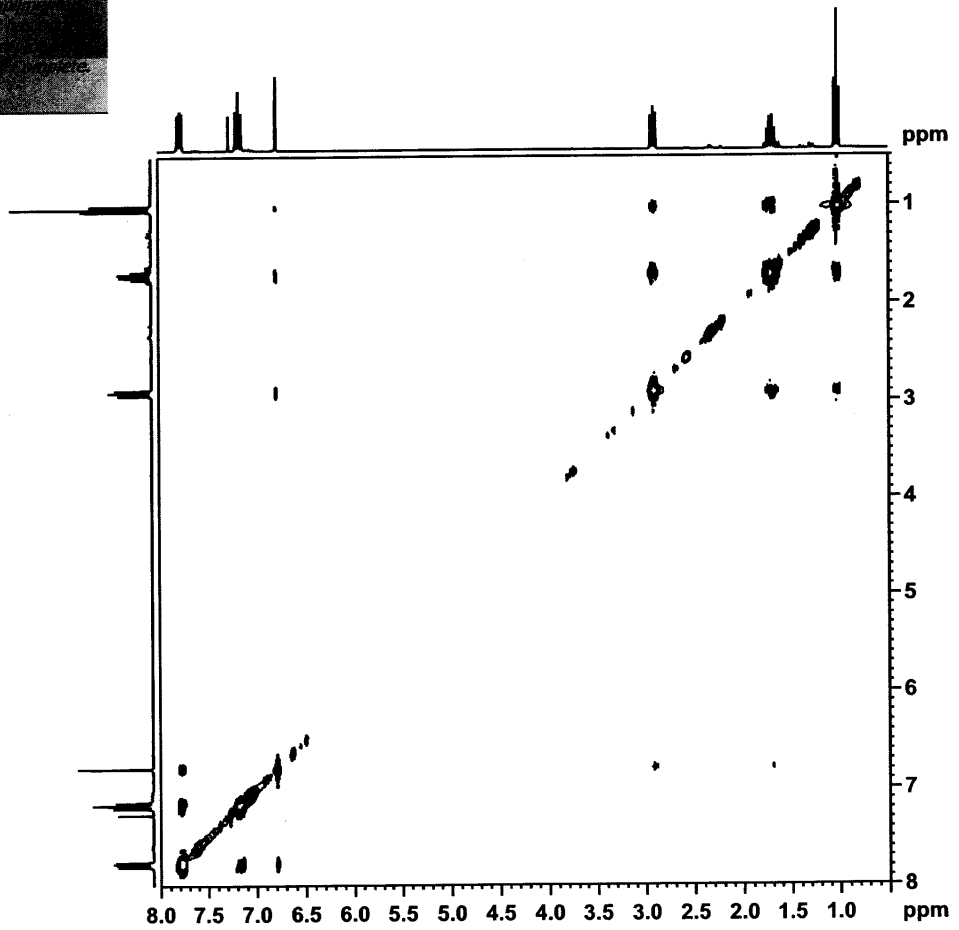

13.763
22.266
28.375
76.576
77.000
77.424
110.054
116.218
116.515
124.897
124.940
127.431
127.553
143.845
146.923
158.044
162.144
165.481
169.964
170.308
170.648
Pr-n
C3F7-n
O
H
O
F
3k
xc-13-153 C
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 05 00:59:01 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 1090
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

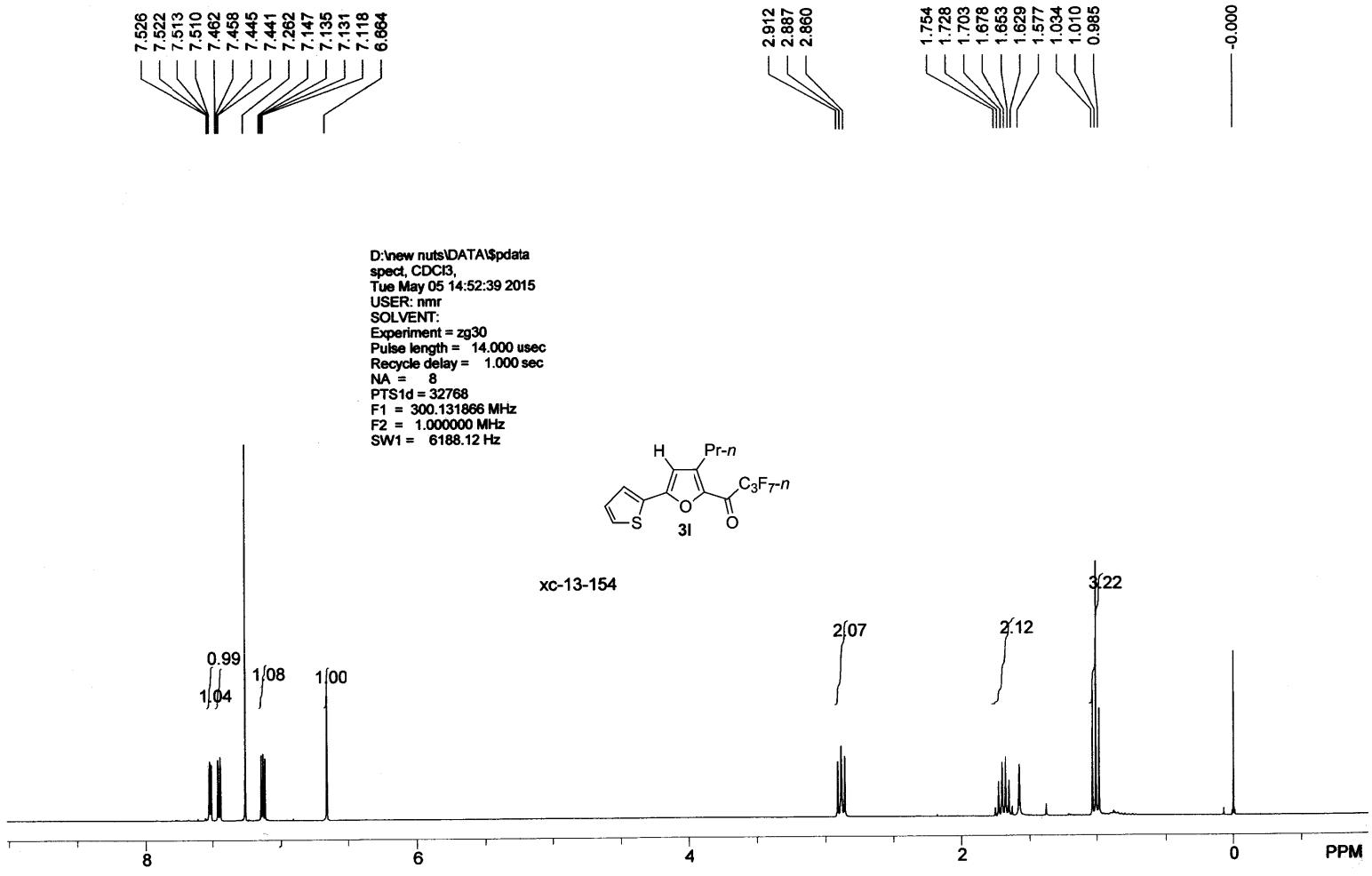

xc-13-153 F
3.00
2.24
2.06
0.89
3k
Pr-n
C3F7-n
O
H
F
D:\new nuts\DATA\5pdata
spect, CDCl3,
Mon May 04 14:52:59 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0.000
80.973
81.005
81.037
109.404
109.416
109.428
109.435
109.447
109.453
109.465
117.960
117.982
117.991
118.004
118.013
118.023
118.037
118.055
126.301
126.337
126.370
PPM
0
-50
-100
-150
-200

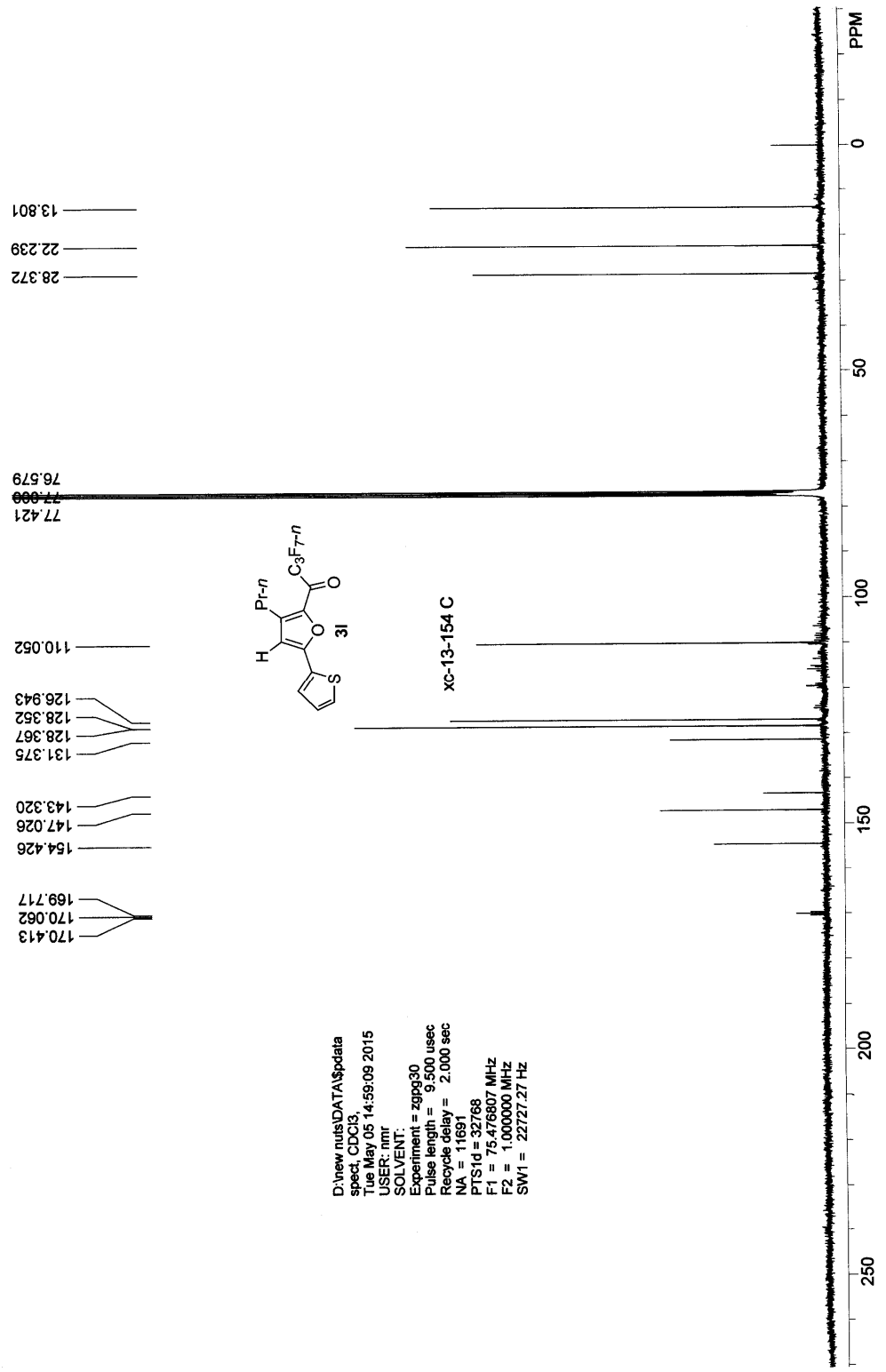


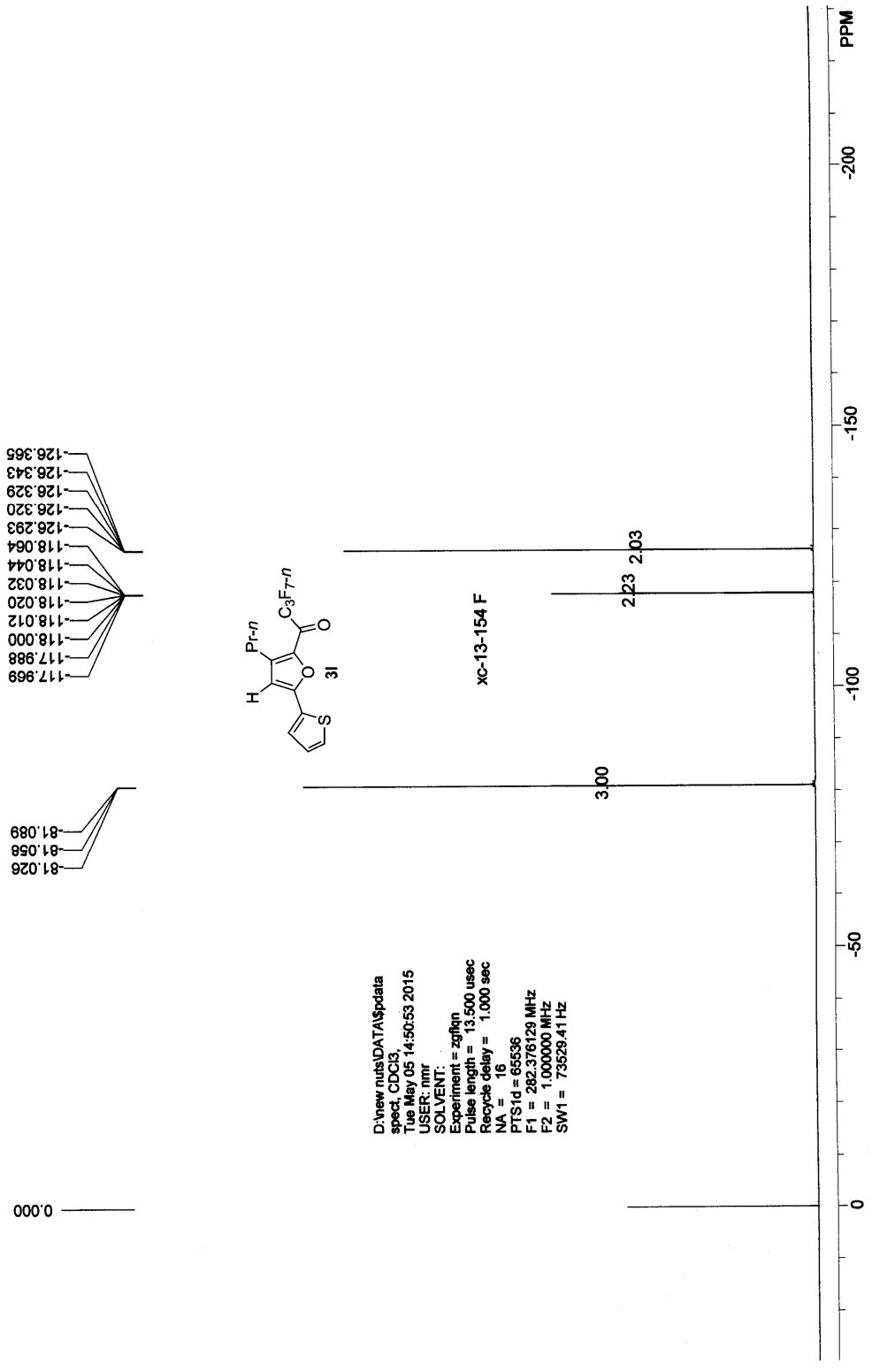
3k

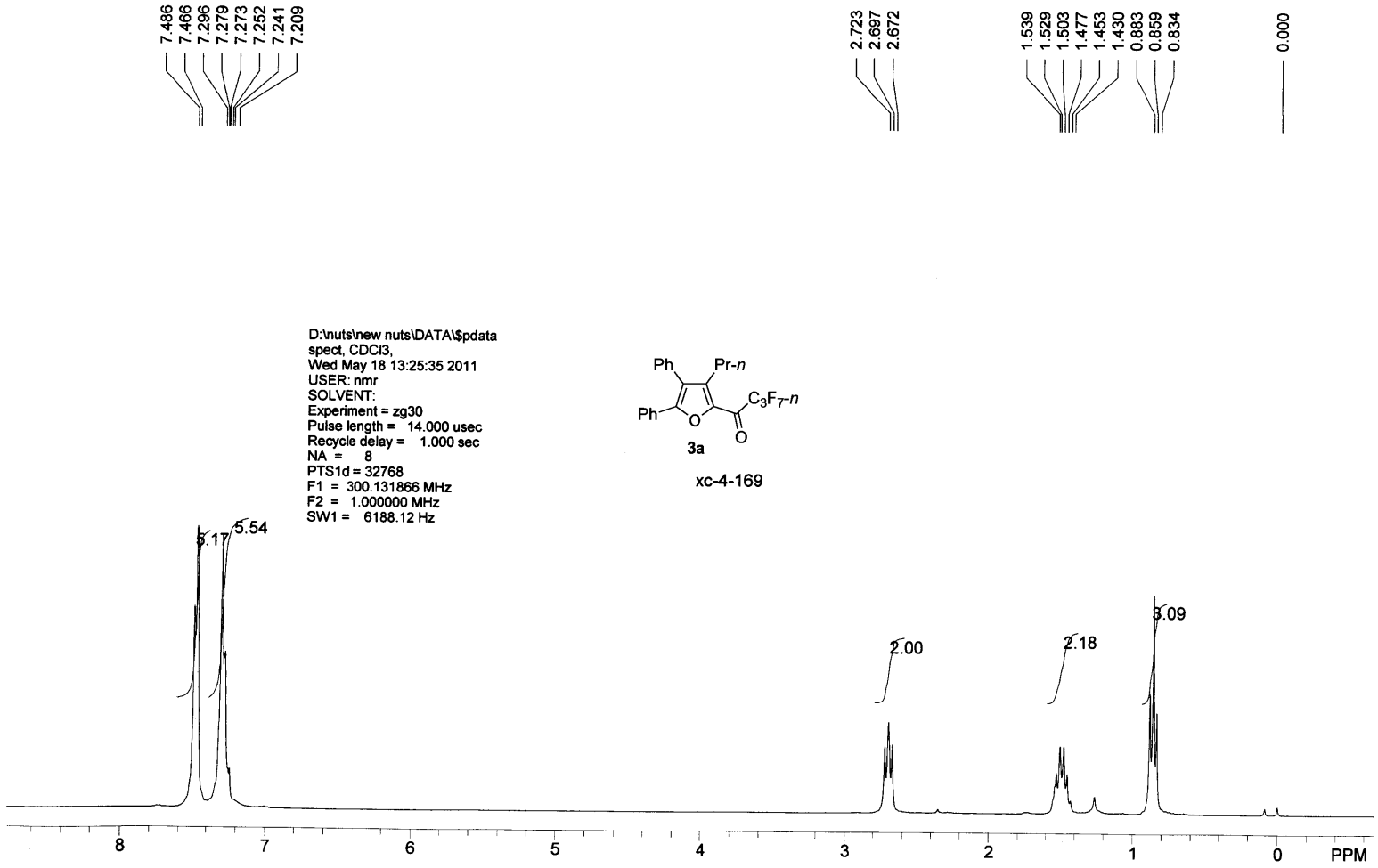
xc-13-153-noe

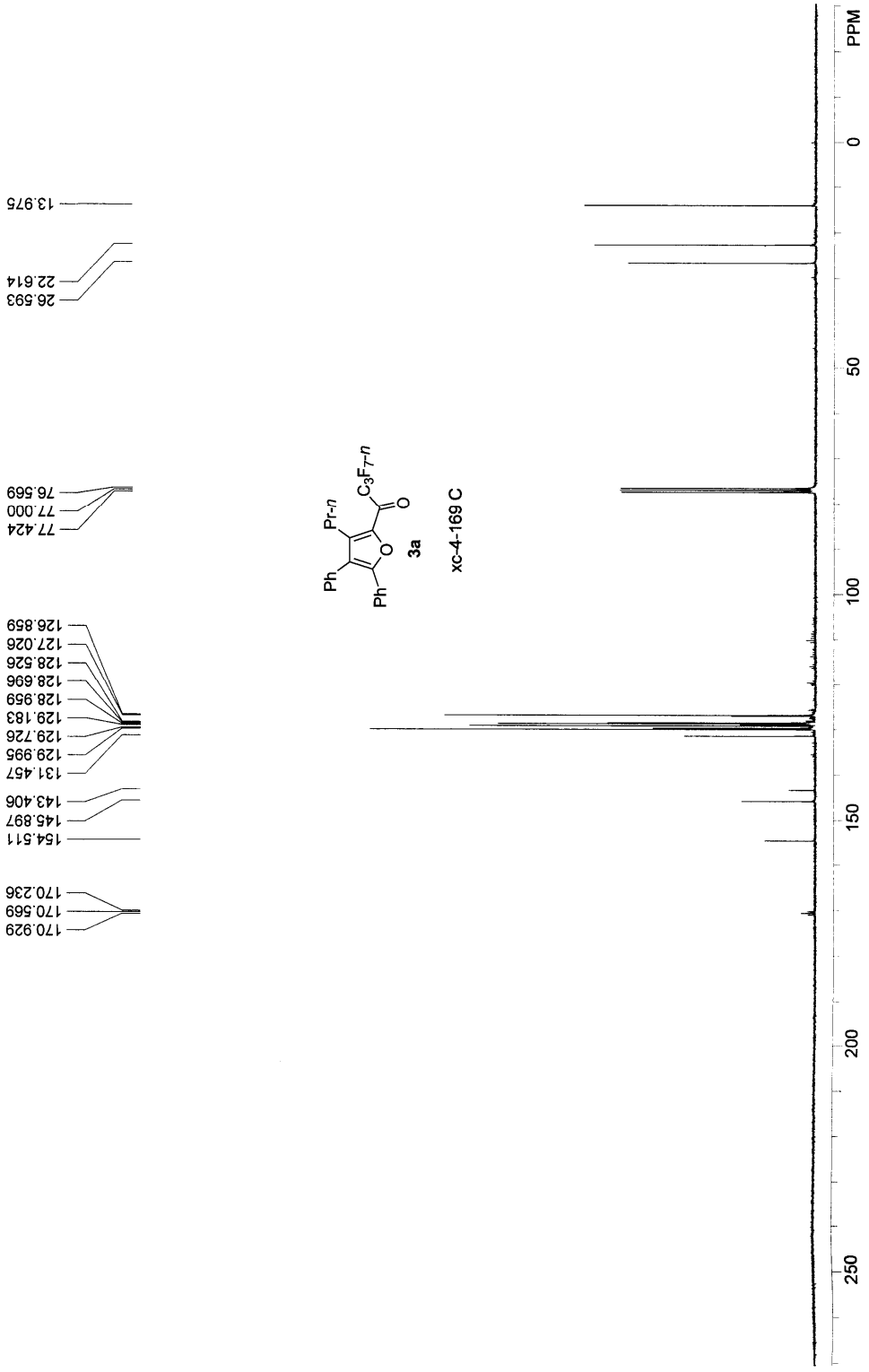


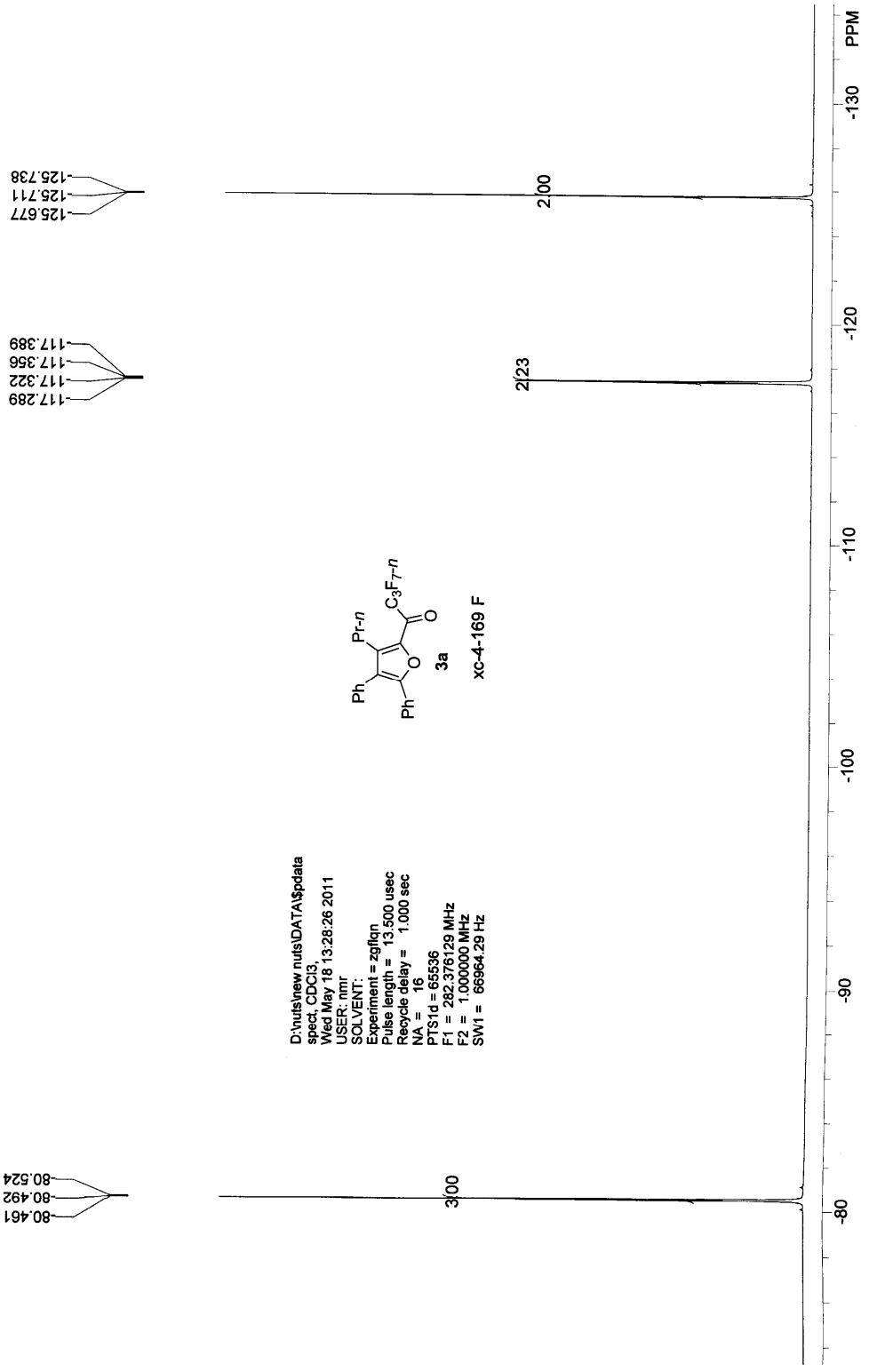

7.526
7.522
7.513
7.510
7.462
7.458
7.445
7.441
7.262
7.147
7.135
7.131
7.118
6.864
2.912
2.887
2.860
1.754
1.728
1.703
1.678
1.653
1.629
1.577
1.034
1.010
0.985
-0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 05 14:52:39 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
H
Pr-n
C3F7-n
S
O
O
3l
xc-13-154
0.99
1.04
1.08
1.00
2.07
2.12
3.22
8
6
4
2
0
PPM

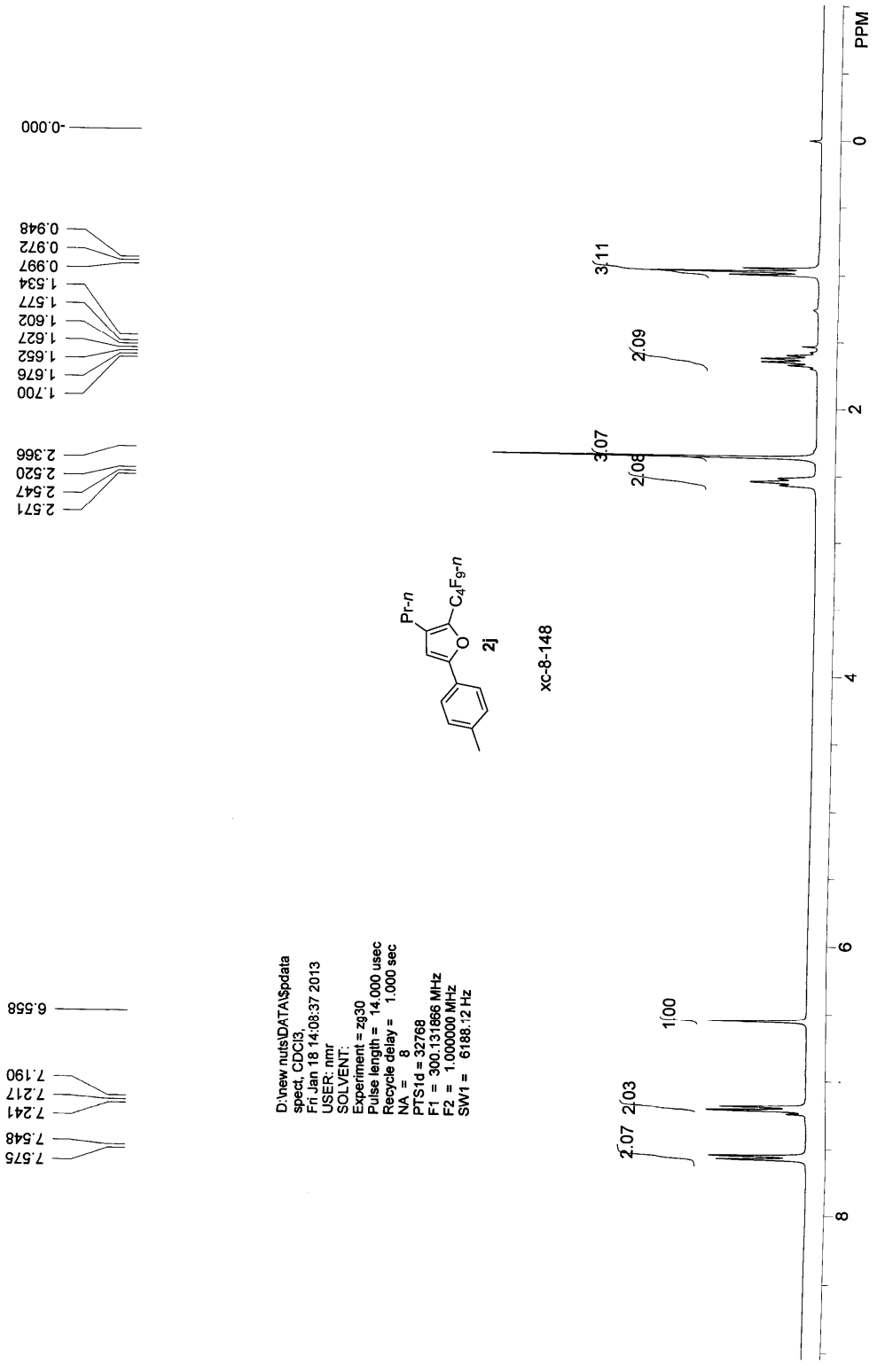

13.801
22.239
28.372
76.579
77.421
110.052
126.943
128.362
128.367
131.375
143.320
147.026
154.426
169.717
170.062
170.413
xc-13-154 C
3l
Pr-n
C3F7-n
H
O
S
D:\new nuts\DATA\$pdata
sped, CDCl3,
Tue May 05 14:59:09 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 11691
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

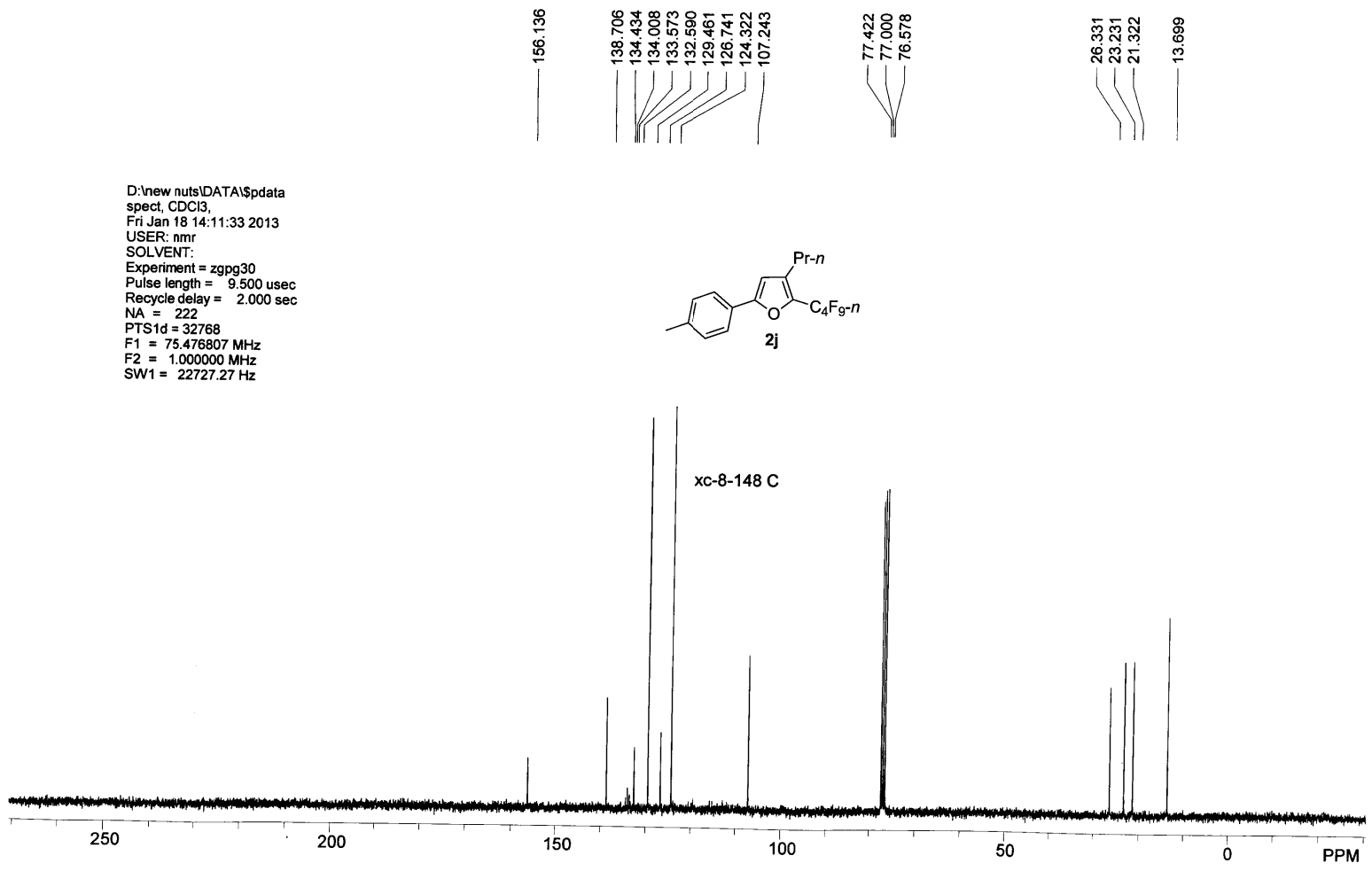

PPM
0
-50
-100
-150
-200
3.00
2.23
2.03
xc-13-154 F
3l
Pr-n
C3F7-n
O
O
S
H
D:\new nuts\DATA\$pdata
spect, CDCl3,
Tue May 05 14:50:53 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.3761129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0.000
81.026
81.056
81.089
117.969
117.988
118.000
118.012
118.020
118.032
118.044
118.064
126.293
126.320
126.329
126.343
126.365

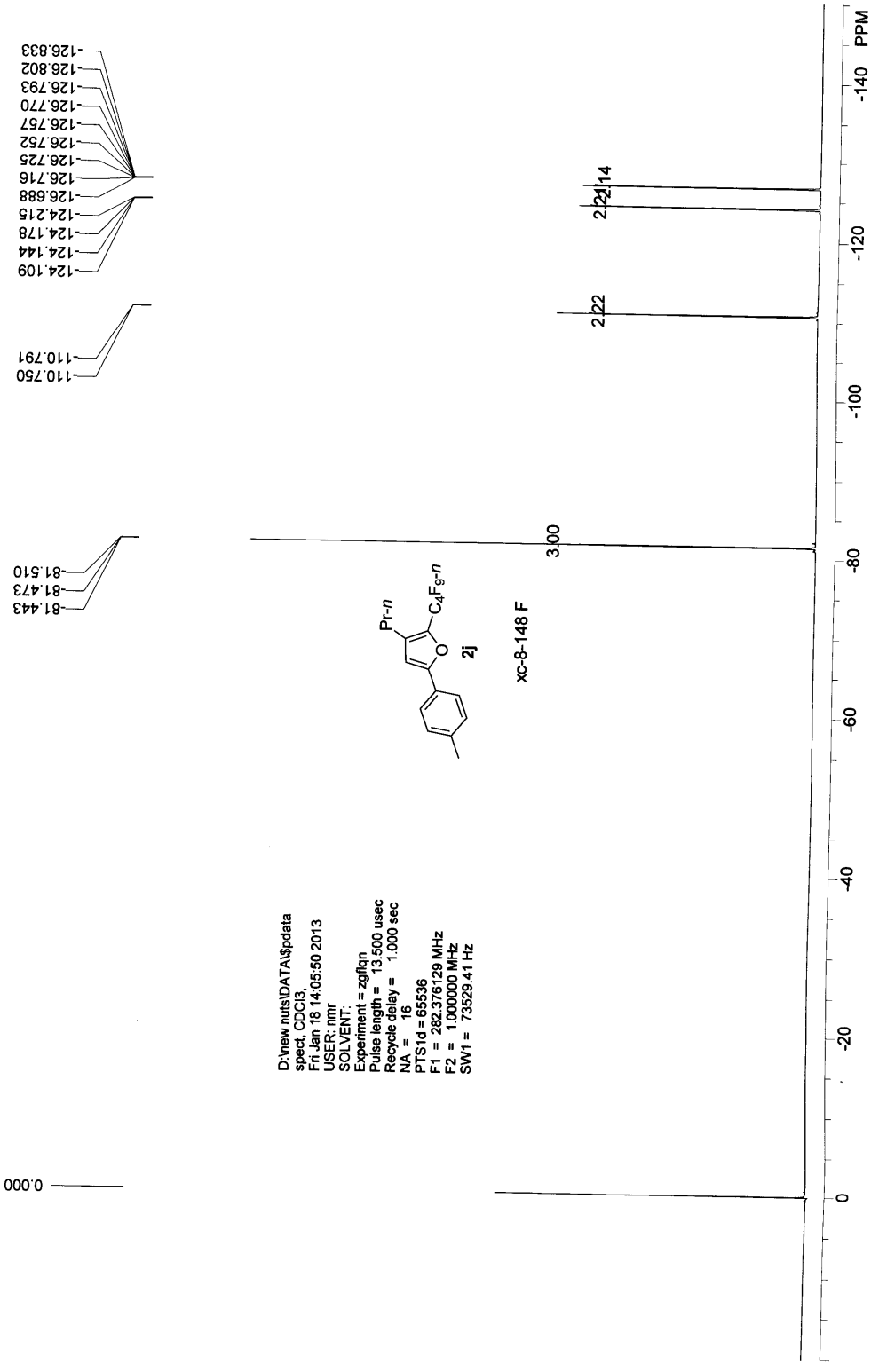

7.486
7.466
7.296
7.279
7.273
7.252
7.241
7.209
2.723
2.697
2.672
1.539
1.529
1.503
1.477
1.453
1.430
0.883
0.859
0.834
0.000
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3,
Wed May 18 13:25:35 2011
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Ph
Pr-n
Ph
O
C3F7-n
O
3a
xc-4-169
5.17
5.54
2.00
2.18
3.09
8
7
6
5
4
3
2
1
0
PPM

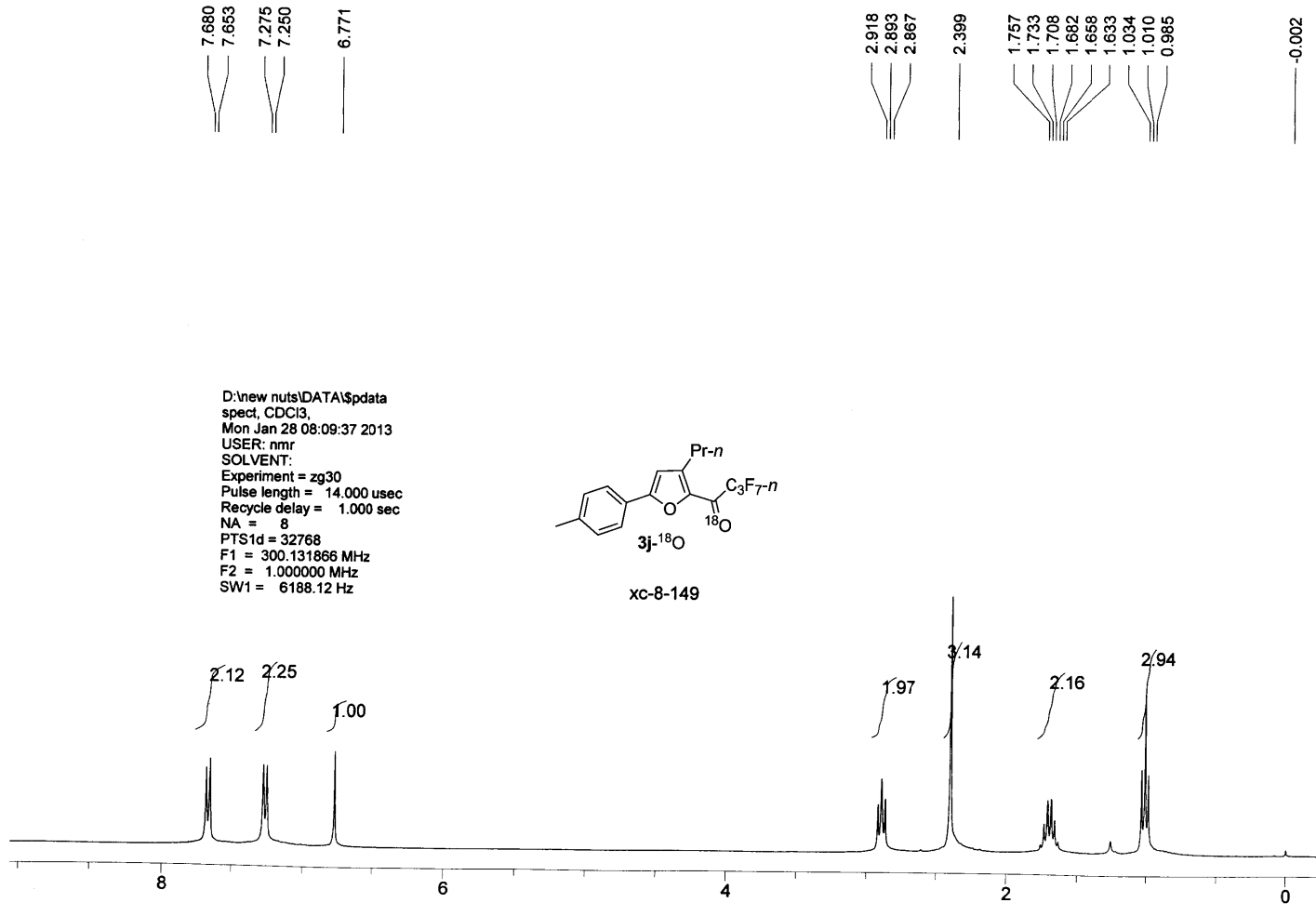

13.975
22.614
26.593
76.569
77.000
77.424
126.859
127.026
128.526
128.696
128.959
129.183
129.726
129.995
131.457
143.406
145.897
154.511
170.236
170.569
170.929
Pr-n
C3F7-n
O
Ph
O
Ph
3a
xc-4-169 C
PPM
0
50
100
150
200
250

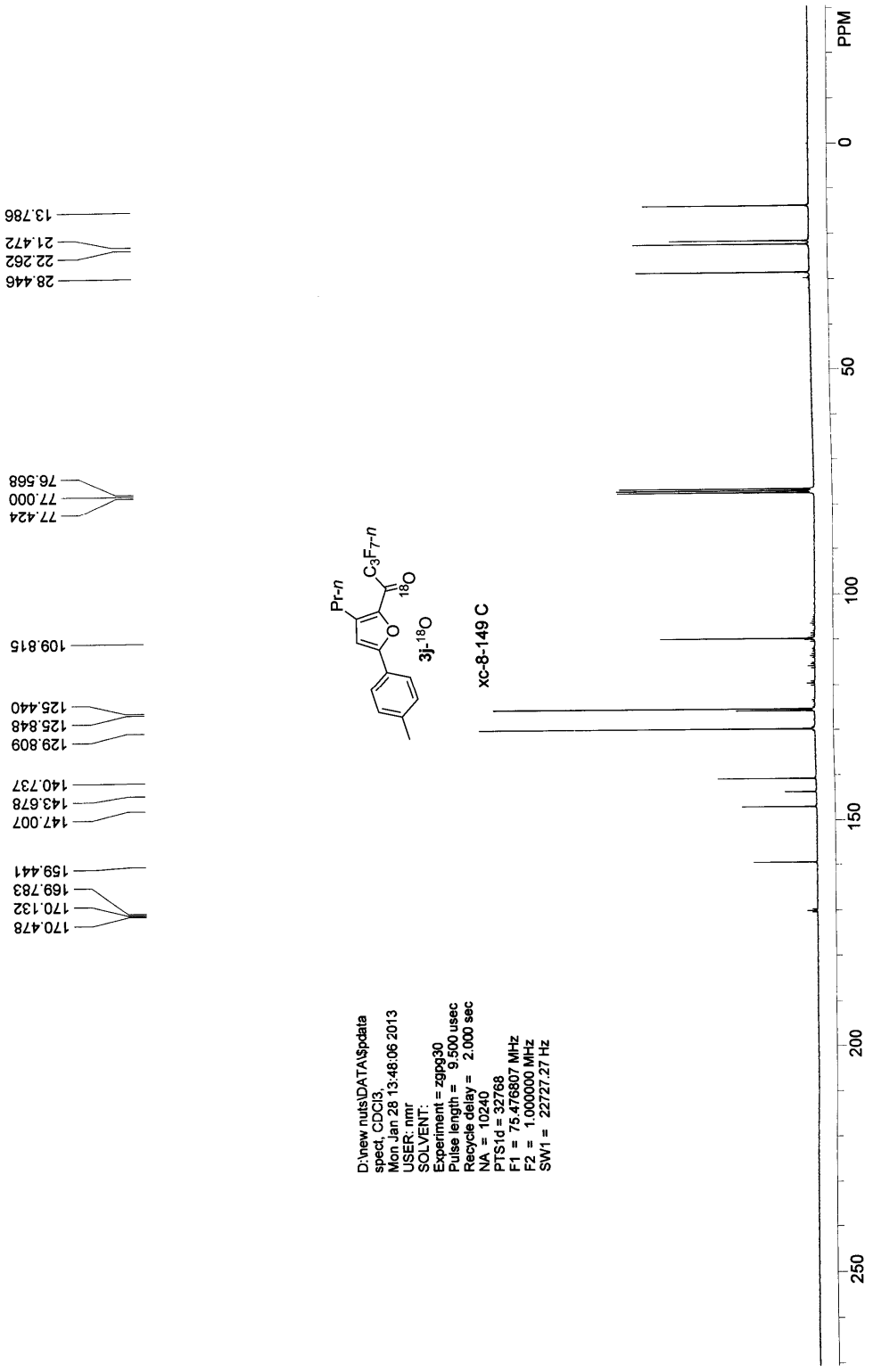

-80.461
-80.492
-80.524
-117.289
-117.322
-117.356
-117.389
-125.677
-125.711
-125.738
3.00
2.23
2.00
-80
-90
-100
-110
-120
-130
PPM
Ph
Pr-n
C3F7-n
O
Ph
3a
xc-4-169 F
D:\nuts\new nuts\DATA\$pdata
spect, CDCl3
Wed May 18 13:28:26 2011
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz

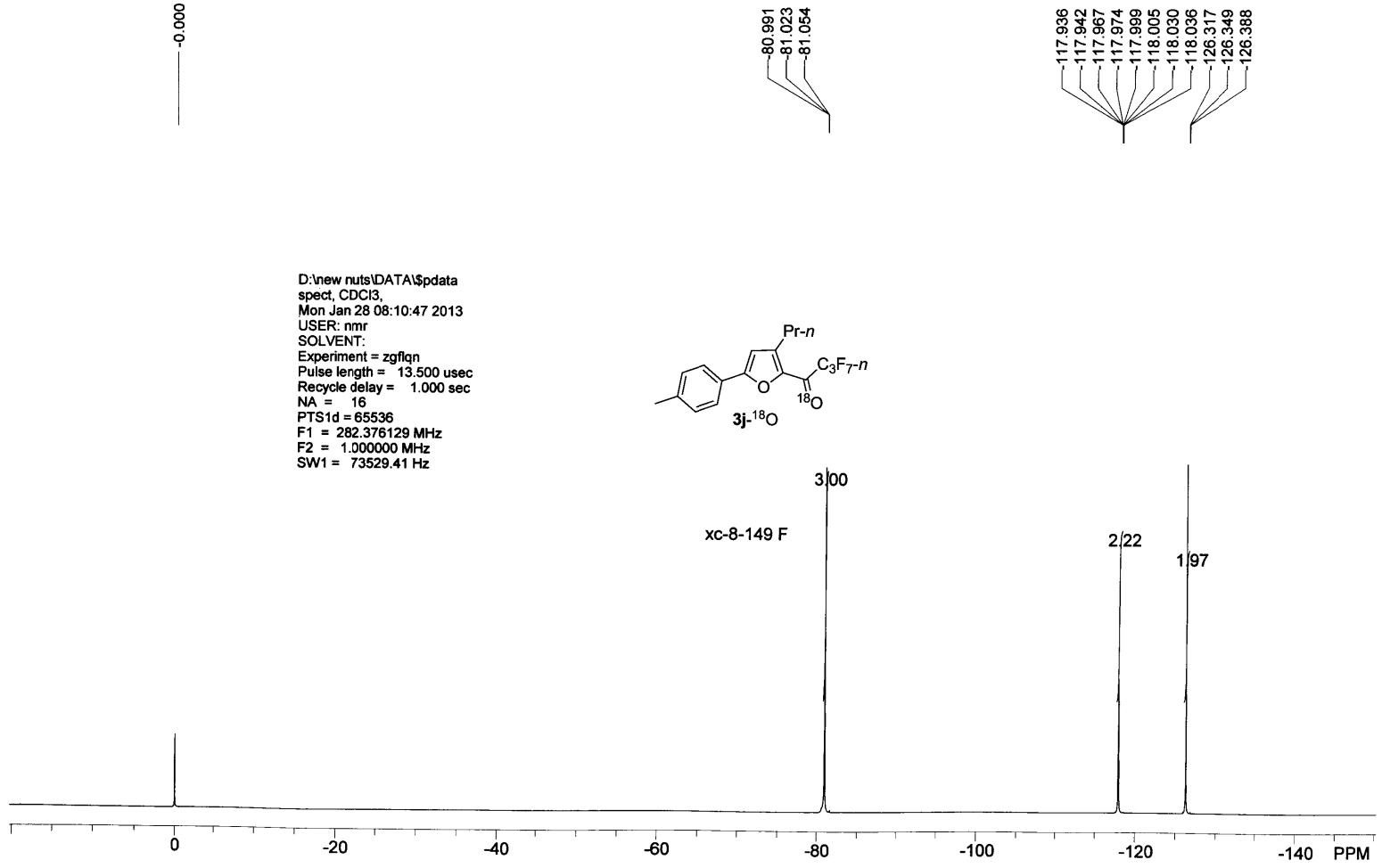

-0.000
0.948
0.972
0.997
1.534
1.577
1.602
1.627
1.652
1.676
1.700
2.366
2.520
2.547
2.571
6.558
7.190
7.217
7.241
7.548
7.575
D:\new nuts\DATA\5pdata
spect, CDCl3,
Fri Jan 18 14:08:37 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Pr-n
C4F9-n
O
2j
xc-8-148
3.11
2.09
3.07
2.08
2.07
2.03
1.00
8
6
4
2
0
PPM


156.136
138.706
134.434
134.008
133.573
132.590
129.461
126.741
124.322
107.243
77.422
77.000
76.578
26.331
23.231
21.322
13.699
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jan 18 14:11:33 2013
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 222
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Pr-n
C4F9-n
O
2j
xc-8-148 C
250
200
150
100
50
0
PPM


PPM
-140
-120
-100
-80
-60
-40
-20
0
2.22
2.21
2.14
3.00
xc-8-148 F
2j
Pr-n
C4F9-n
O
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jan 18 14:05:50 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
0.000
81.510
81.473
81.443
110.791
110.750
126.833
126.802
126.793
126.770
126.757
126.752
126.725
126.716
126.688
124.215
124.178
124.144
124.109

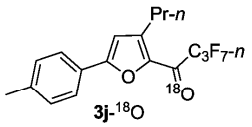

7.680
7.653
7.275
7.250
6.771
2.918
2.893
2.867
2.399
1.757
1.733
1.708
1.682
1.658
1.633
1.034
1.010
0.985
-0.002
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Jan 28 08:09:37 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Pr-n
C3F7-n
O
18O
3j-18O
xc-8-149
2.12
2.25
1.00
1.97
3.14
2.16
2.94
8
6
4
2
0
PPM


13.786
21.472
22.262
28.446
76.568
77.000
77.424
109.815
125.440
125.848
129.809
140.737
143.678
147.007
159.441
169.783
170.132
170.478
Pr-n
C3F7-n
18O
O
3j-18O
xc-8-149 C
D:\new nuts\DATA\5pdata
spect, CDCl3,
Mon Jan 28 13:48:06 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
PPM
0
50
100
150
200
250


-0.000
80.991
81.023
81.054
117.936
117.942
117.967
117.974
117.999
118.005
118.030
118.036
126.317
126.349
126.388
D:\new nuts\DATA\$pdata
spect, CDCl3,
Mon Jan 28 08:10:47 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
Pr-n
C3F7-n
O
18O
3j-18O
xc-8-149 F
3.00
2.22
1.97
0
-20
-40
-60
-80
-100
-120
-140
PPM

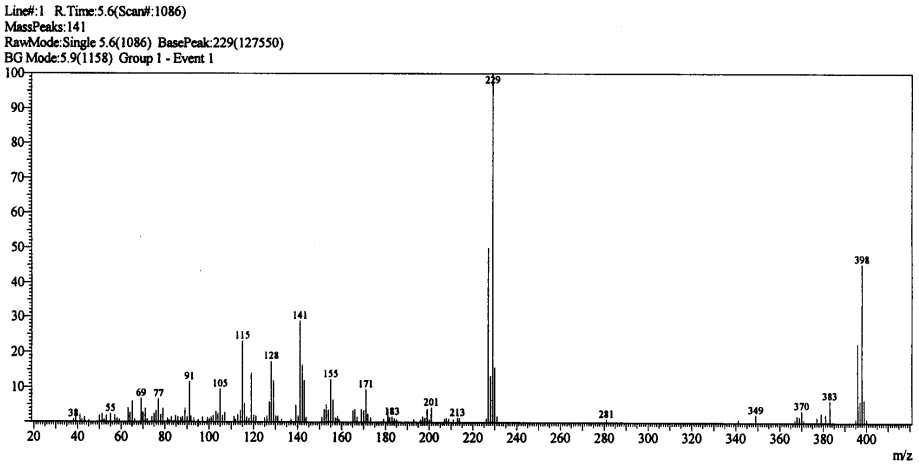
Sample Information

E:\msm\xc-8-149_2013-1-25_5.qgd



3j-^{18}O

Spectrum



Mass Table

Line#:1 R.Time:5.6(Scan#:1086)

MassPeaks:141

RawMode:Single 5.6(1086) BasePeak:229(127550)

BG Mode:5.9(1158) Group 1 - Event 1

#	m/z	Abs. Int.	Rel. Int.	#	m/z	Abs. Int.	Rel. Int.
1	38.05	1073	0.84	25	73.20	244	0.19
2	39.05	4456	3.49	26	74.00	2020	1.58
3	41.05	2707	2.12	27	75.00	3136	2.46
4	41.95	930	0.73	28	75.95	4165	3.27
5	43.05	1902	1.49	29	77.00	8519	6.68
6	45.05	696	0.55	30	78.10	2641	2.07
7	48.95	268	0.21	31	79.05	5130	4.02
8	49.95	2355	1.85	32	81.10	1420	1.11
9	51.10	2951	2.31	33	82.00	883	0.69
10	52.10	1092	0.86	34	82.95	1928	1.51
11	53.15	2339	1.83	35	84.00	512	0.40
12	55.05	3028	2.37	36	84.85	2360	1.85
13	57.10	2477	1.94	37	85.90	1983	1.55
14	58.10	1489	1.17	38	87.05	1702	1.33
15	59.10	1002	0.79	39	87.90	2058	1.61
16	63.10	5182	4.06	40	89.05	5041	3.95
17	63.95	3368	2.64	41	90.00	1924	1.51
18	65.00	7690	6.03	42	91.05	14851	11.64
19	66.10	1098	0.86	43	91.85	2245	1.76
20	67.10	134	0.11	44	93.10	1572	1.23
21	69.00	8646	6.78	45	95.15	894	0.70
22	69.90	3615	2.83	46	97.00	1962	1.54
23	70.90	4980	3.90	47	99.20	1790	1.40
24	71.95	1085	0.85	48	100.15	1385	1.09

#	m/z	Abs. Int.	Rel. Int.	#	m/z	Abs. Int.	Rel. Int.
49	100.95	1834	1.44	96	171.05	12074	9.47
50	101.90	2449	1.92	97	171.90	3044	2.39
51	103.05	3913	3.07	98	173.15	1731	1.36
52	104.00	3242	2.54	99	179.10	1175	0.92
53	105.00	12119	9.50	100	181.10	4964	3.89
54	106.00	2366	1.85	101	181.95	1875	1.47
55	107.10	3569	2.80	102	183.05	2012	1.58
56	111.20	2084	1.63	103	184.00	1380	1.08
57	111.95	1055	0.83	104	185.00	1060	0.83
58	112.90	2762	2.17	105	192.90	1066	0.84
59	114.05	4245	3.33	106	196.00	1002	0.79
60	115.00	29578	23.19	107	197.00	2084	1.63
61	116.00	6756	5.30	108	198.00	1649	1.29
62	117.00	1937	1.52	109	199.00	4938	3.87
63	118.05	1427	1.12	110	200.00	1118	0.88
64	119.05	18063	14.16	111	200.95	5449	4.27
65	120.10	2660	2.09	112	206.95	1272	1.00
66	121.10	2314	1.81	113	207.80	1610	1.26
67	125.00	1585	1.24	114	208.85	1270	1.00
68	126.05	2394	1.88	115	211.00	1092	0.86
69	127.10	7460	5.85	116	212.85	1702	1.33
70	128.10	22327	17.50	117	213.90	1540	1.21
71	129.05	15242	11.95	118	226.05	1346	1.06
72	130.05	2260	1.77	119	227.00	63844	50.05
73	131.05	2399	1.88	120	228.05	17091	13.40
74	132.75	1190	0.93	121	229.00	127550	100.00
75	137.00	1265	0.99	122	230.00	20202	15.84
76	139.10	6289	4.93	123	231.05	2313	1.81
77	140.15	2322	1.82	124	280.95	1282	1.01
78	141.10	36804	28.85	125	341.05	1115	0.87
79	142.10	21047	16.50	126	349.05	2835	2.22
80	143.05	15337	12.02	127	366.95	1146	0.90
81	143.90	1841	1.44	128	367.90	2538	1.99
82	150.95	1822	1.43	129	368.95	2175	1.71
83	152.00	4695	3.68	130	370.00	4343	3.40
84	153.05	6553	5.14	131	371.00	1054	0.83
85	154.00	4567	3.58	132	377.00	1962	1.54
86	155.00	15710	12.32	133	379.00	3479	2.73
87	156.10	8260	6.48	134	381.00	3050	2.39
88	157.10	1674	1.31	135	383.00	7998	6.27
89	158.10	2212	1.73	136	395.05	1233	0.97
90	158.95	1025	0.80	137	396.00	28772	22.56
91	165.05	4239	3.32	138	397.05	7760	6.08
92	166.05	4645	3.64	139	398.00	57924	45.41
93	166.95	1999	1.57	140	399.00	8356	6.55
94	169.05	4873	3.82	141	400.10	1278	1.00
95	170.05	4332	3.40				

xc-8-149

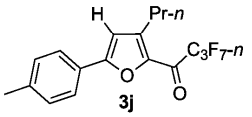
The naturally occurring isotopic ^{18}O will also produce $[M+2]^+$ peak. According to the theoretical calculations, the ratio $C_{18}H_{15}{}^{16}O_2F_7$: $C_{18}H_{15}{}^{16}O^{18}OF_7$ = 99.762:0.2. Thus, the intensity of $[M+2]^+$ $(C_{18}H_{15}{}^{16}O^{18}OF_7)^+$ peak will be 0.2% of the intensity of the molecular peak M ($C_{18}H_{15}{}^{16}O_2F_7$). According to the MS spectrum of xc-8-149, the relative abundances of **3j**-^{16}O 396 $[M(^{16}O)^+]$ and **3j**-^{18}O 398 $[M(^{18}O)^+]$ are 22.56, 45.41, respectively. The ^{18}O% of **3j**-^{18}O can be calculated as follows: $([M(^{18}O)^+] - [M(^{16}O)^+] \times 0.2\%)/([M(^{18}O)^+] - [M(^{16}O)^+] \times 0.2\% + [M(^{16}O)^+]) = 45.41-22.56\times0.002/(45.41-22.56\times0.002+ 22.56) \approx 66.79\%$.

xc-8-138

The spurious contribution of **3j** to M+2: $(1.03-33.70\times0.002)\% \approx 0.96\%$.

Sample Information

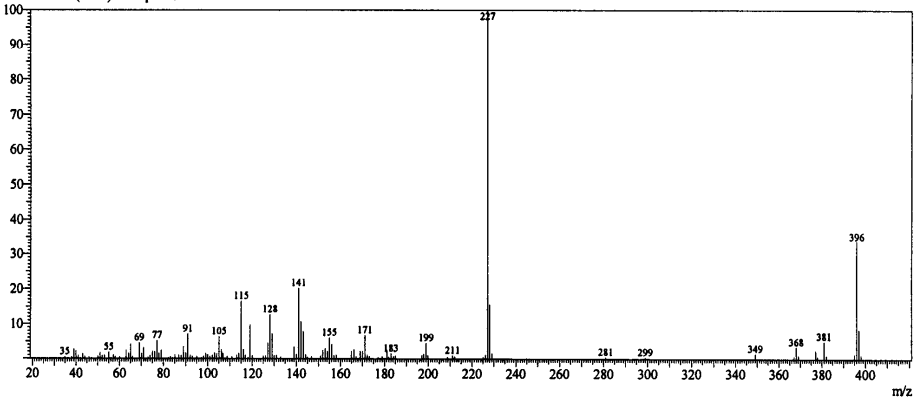
E:\msm\xc-8-138_2013-1-25_3.qgd



3j

Spectrum

Line#:1 R.Time:5.6(Scan#:1086)
MassPeaks:132
RawMode:Single 5.6(1086) BasePeak:227(228017)
BG Mode:5.7(1109) Group 1 - Event 1



Mass Table

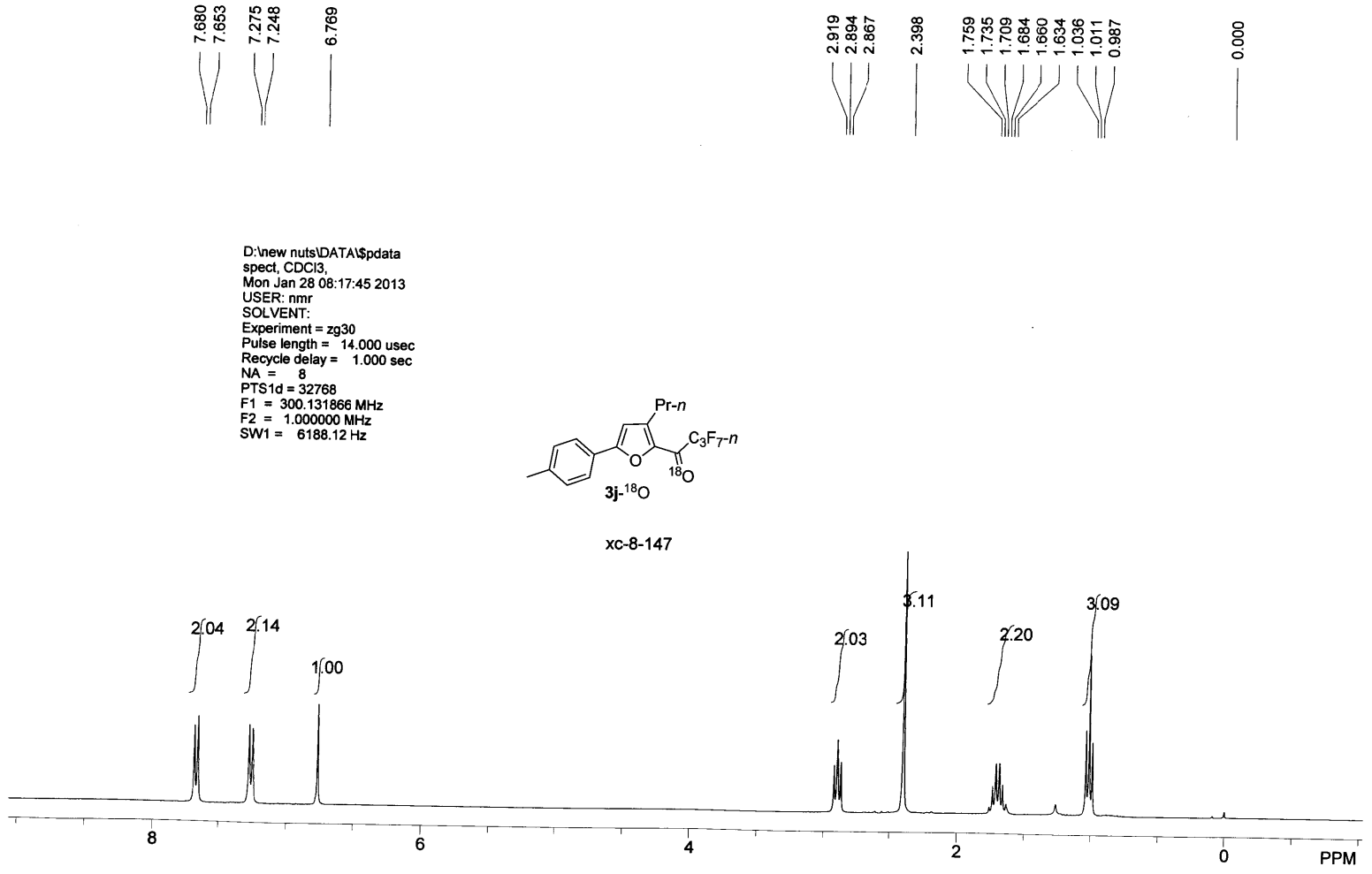
Line#:1 R.Time:5.6(Scan#:1086)
MassPeaks:132
RawMode:Single 5.6(1086) BasePeak:227(228017)
BG Mode:5.7(1109) Group 1 - Event 1

#	m/z	Abs. Int.	Rel. Int.	#	m/z	Abs. Int.	Rel. Int.
1	34.95	1217	0.53	25	70.05	3299	1.45
2	37.95	1086	0.48	26	71.05	7305	3.20
3	39.05	6292	2.76	27	72.95	1010	0.44
4	40.00	5222	2.29	28	73.90	2164	0.95
5	41.05	2012	0.88	29	75.05	4795	2.10
6	42.05	434	0.19	30	76.05	4690	2.06
7	43.05	3240	1.42	31	77.10	11943	5.24
8	44.05	1482	0.65	32	77.95	3652	1.60
9	45.90	1006	0.44	33	79.05	5541	2.43
10	46.95	381	0.17	34	82.15	709	0.31
11	50.05	1648	0.72	35	83.10	1413	0.62
12	51.00	3991	1.75	36	84.10	825	0.36
13	52.00	1959	0.86	37	85.10	2784	1.22
14	53.00	2286	1.00	38	86.05	238	0.10
15	55.00	4199	1.84	39	86.85	2366	1.04
16	56.10	560	0.25	40	88.00	2068	0.91
17	57.15	2616	1.15	41	89.00	7937	3.48
18	58.05	1468	0.64	42	90.05	3878	1.70
19	60.05	1141	0.50	43	91.00	16295	7.15
20	63.00	5652	2.48	44	92.00	2484	1.09
21	64.15	3487	1.53	45	92.95	1837	0.81
22	65.10	9569	4.20	46	95.05	1735	0.76
23	65.95	1431	0.63	47	97.15	815	0.36
24	69.00	10458	4.59	48	98.05	1837	0.81

#	m/z	Abs. Int.	Rel. Int.	#	m/z	Abs. Int.	Rel. Int.
49	99.00	3572	1.57	91	158.10	2670	1.17
50	100.00	2855	1.25	92	165.00	5006	2.20
51	101.00	1626	0.71	93	166.10	6190	2.71
52	102.15	2447	1.07	94	167.05	1858	0.81
53	103.10	3937	1.73	95	169.05	5184	2.27
54	104.05	3364	1.48	96	170.05	5063	2.22
55	105.10	14604	6.40	97	171.10	15482	6.79
56	106.10	6049	2.65	98	172.15	2660	1.17
57	106.90	3591	1.57	99	173.10	1607	0.70
58	108.80	1684	0.74	100	177.20	1031	0.45
59	110.95	1332	0.58	101	178.95	1715	0.75
60	113.05	2376	1.04	102	181.10	5934	2.60
61	114.00	3783	1.66	103	181.95	1403	0.62
62	115.05	37780	16.57	104	182.95	3484	1.53
63	116.10	6164	2.70	105	184.05	1730	0.76
64	117.00	2465	1.08	106	185.05	2156	0.95
65	118.15	1118	0.49	107	197.05	2544	1.12
66	119.10	22404	9.83	108	198.00	3322	1.46
67	120.10	2202	0.97	109	199.00	10478	4.60
68	125.05	1796	0.79	110	199.90	2497	1.10
69	126.05	2162	0.95	111	208.80	1146	0.50
70	127.10	10483	4.60	112	211.10	2254	0.99
71	128.10	29088	12.76	113	212.00	1575	0.69
72	129.05	16539	7.25	114	225.05	1285	0.56
73	130.00	2350	1.03	115	226.05	2713	1.19
74	131.05	2412	1.06	116	227.05	228017	100.00
75	133.00	1191	0.52	117	228.05	35837	15.72
76	139.05	8038	3.53	118	229.15	3652	1.60
77	140.10	3079	1.35	119	281.00	1249	0.55
78	141.05	46203	20.26	120	299.00	1127	0.49
79	142.05	24504	10.75	121	349.10	3476	1.52
80	143.10	18042	7.91	122	367.05	1789	0.78
81	144.15	2934	1.29	123	368.05	7856	3.45
82	145.10	1178	0.52	124	369.05	2395	1.05
83	146.90	1550	0.68	125	377.10	5818	2.55
84	150.95	1915	0.84	126	378.00	1735	0.76
85	151.95	5593	2.45	127	381.00	11495	5.04
86	153.10	6830	3.00	128	382.10	2279	1.00
87	154.10	4961	2.18	129	395.05	3188	1.40
88	155.05	13819	6.06	130	396.05	76845	33.70
89	156.05	9784	4.29	131	397.05	19325	8.48
90	157.00	2247	0.99	132	398.00	2356	1.03

xc-8-138

The spurious contribution of **3j** to M+2: (1.03-33.70×0.002)% ≈ 0.96%.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1.735
1.709
1.684
1.660
1.634
1.036
1.011
0.987
0.000
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spect, CDCl3,
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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
Pr-n
C3F7-n
O
18O
3j-18O
xc-8-147
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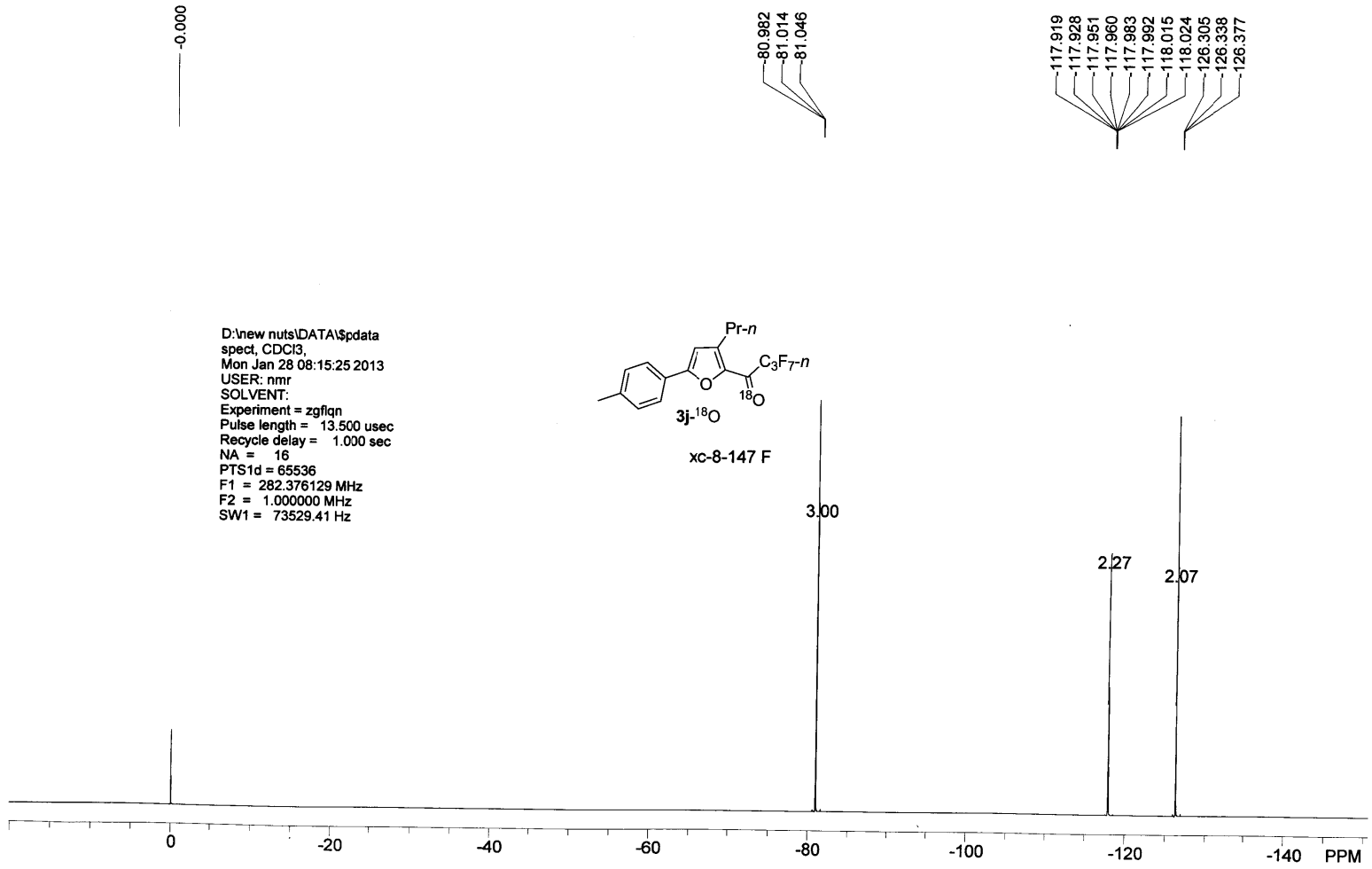
2.14
1.00
2.03
3.11
2.20
3.09
8
6
4
2
0
PPM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-117.928
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-117.960
-117.983
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-118.024
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-126.338
-126.377
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spect, CDCl3,
Mon Jan 28 08:15:25 2013
USER: nmr
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Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
Pr-n
C3F7-n
O
18O
3j-18O
xc-8-147 F
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2.27
2.07
0
-20
-40
-60
-80
-100
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-140
PPM

Sample Information

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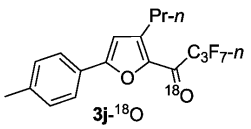
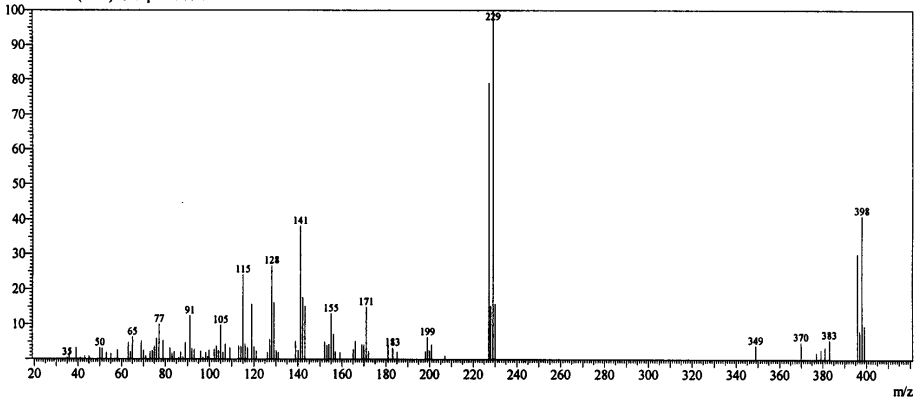


3j-^{18}O

Spectrum

Line#:1 R.Time:5.6(Scan#:1088)
MassPeaks:103
RawMode:Single 5.6(1088) BasePeak:229(52639)
BG Mode:5.8(1143) Group 1 - Event 1



Mass Table

Line#:1 R.Time:5.6(Scan#:1088)
MassPeaks:103
RawMode:Single 5.6(1088) BasePeak:229(52639)
BG Mode:5.8(1143) Group 1 - Event 1

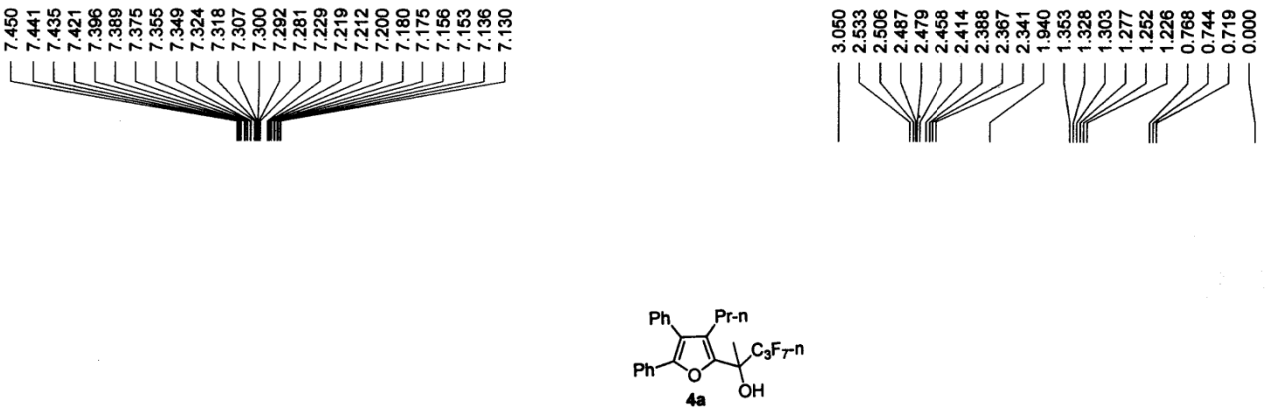
#	m/z	Abs. Int.	Rel. Int.	#	m/z	Abs. Int.	Rel. Int.
1	35.05	295	0.56	25	75.95	3115	5.92
2	36.00	1007	1.91	26	77.05	5277	10.02
3	39.05	1751	3.33	27	78.90	2786	5.29
4	41.00	259	0.49	28	82.05	1752	3.33
5	42.05	152	0.29	29	83.05	913	1.73
6	43.05	477	0.91	30	84.00	1153	2.19
7	44.95	510	0.97	31	86.00	262	0.50
8	47.05	19	0.04	32	86.90	1103	2.10
9	49.95	1700	3.23	33	87.90	355	0.67
10	51.05	1634	3.10	34	89.05	2522	4.79
11	53.05	1012	1.92	35	91.05	6554	12.45
12	55.00	863	1.64	36	92.00	1622	3.08
13	57.00	53	0.10	37	93.10	1519	2.89
14	58.10	1404	2.67	38	96.00	1263	2.40
15	63.05	2565	4.87	39	97.05	329	0.63
16	64.05	1104	2.10	40	98.20	1026	1.95
17	65.00	3318	6.30	41	99.00	416	0.79
18	67.20	84	0.16	42	99.80	1404	2.67
19	69.00	2746	5.22	43	102.10	1513	2.87
20	70.05	1372	2.61	44	103.10	2018	3.83
21	71.05	510	0.97	45	104.05	1296	2.46
22	73.10	1150	2.18	46	105.00	5161	9.80
23	74.00	1308	2.48	47	106.05	1062	2.02
24	75.00	1974	3.75	48	107.05	2306	4.38

#	m/z	Abs. Int.	Rel. Int.	#	m/z	Abs. Int.	Rel. Int.
49	109.10	1737	3.30	77	166.00	2774	5.27
50	113.05	2052	3.90	78	169.05	2240	4.26
51	114.05	1928	3.66	79	170.00	2198	4.18
52	115.00	12706	24.14	80	171.05	7882	14.97
53	116.00	2306	4.38	81	172.10	1257	2.39
54	117.10	1782	3.39	82	180.95	2125	4.04
55	119.00	8286	15.74	83	183.15	1796	3.41
56	120.05	1908	3.62	84	185.10	1193	2.27
57	121.10	1276	2.42	85	198.05	1212	2.30
58	126.05	1051	2.00	86	199.05	3378	6.42
59	127.10	3059	5.81	87	200.00	1327	2.52
60	128.05	14062	26.71	88	200.90	2268	4.31
61	129.05	8530	16.20	89	207.00	583	1.11
62	130.15	1348	2.56	90	227.05	41596	79.02
63	131.10	1103	2.10	91	228.00	8105	15.40
64	138.95	2774	5.27	92	229.05	52639	100.00
65	140.10	1378	2.62	93	229.95	8358	15.88
66	141.10	20105	38.19	94	348.90	2102	3.99
67	142.10	9346	17.75	95	369.95	2642	5.02
68	143.10	8028	15.25	96	377.05	1098	2.09
69	152.05	2657	5.05	97	379.15	1486	2.82
70	153.10	2153	4.09	98	381.00	1839	3.49
71	154.05	2317	4.40	99	383.00	2940	5.59
72	155.05	6913	13.13	100	396.00	15906	30.22
73	156.10	3766	7.15	101	396.95	4258	8.09
74	156.90	1193	2.27	102	398.00	21634	41.10
75	159.05	1042	1.98	103	399.10	5033	9.56
76	165.05	1552	2.95				

xc-8-147

The ^{18}O% incorporation of **3j**-^{18}O was determined by MS spectrum. According to the MS spectrum of xc-8-147, the relative abundances of **3j**-^{16}O 396 [$M(^{16}O)^+$], **3j**-^{18}O 398 [$M(^{18}O)^+$] are 30.22, 41.10, respectively. The ^{18}O% of **3j**-^{18}O can be calculated as follows: ([$M(^{18}O)^+$] - [$M(^{16}O)^+$] × 0.2%)/([$M(^{18}O)^+$] - [$M(^{16}O)^+$] × 0.2% + [$M(^{16}O)^+$])

$= 41.10\text{-}30.22 \times 0.002/(41.10\text{-}30.22 \times 0.002 + 30.22) \approx 57.59\%$.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7.281
7.229
7.219
7.212
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7.180
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7.156
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7.136
7.130
3.050
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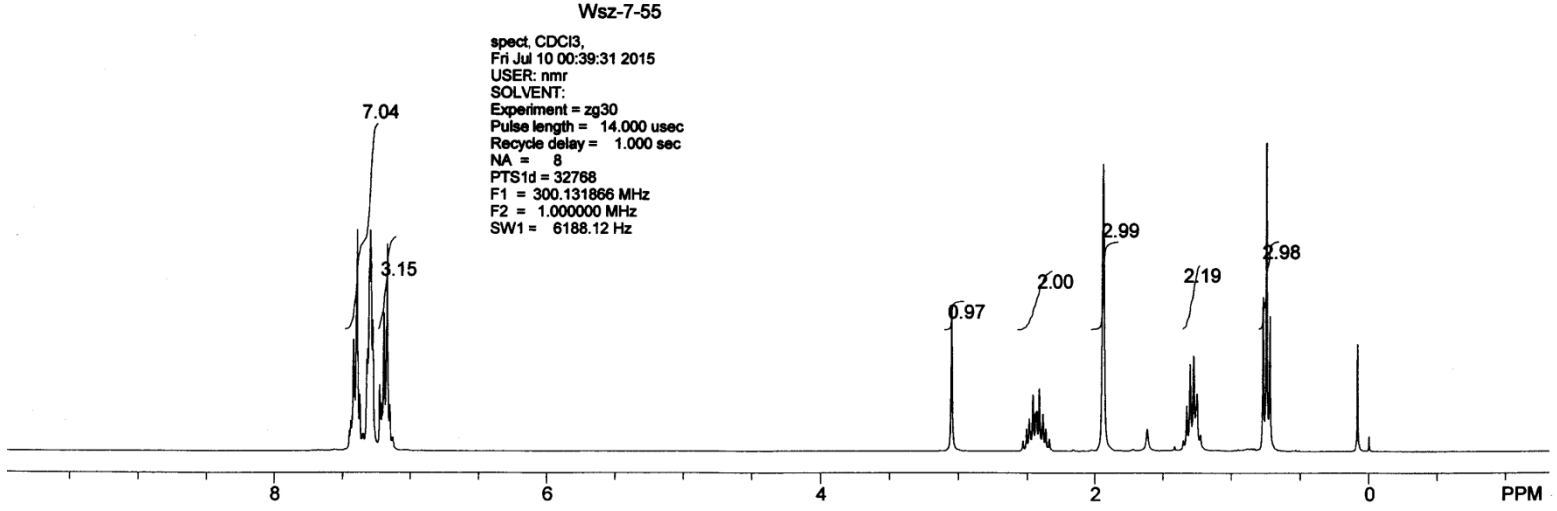
2.479
2.458
2.414
2.388
2.367
2.341
1.940
1.353
1.328
1.303
1.277
1.252
1.226
0.768
0.744
0.719
0.000

Ph
Pr-n
Ph
O
C_3F_7-n
OH

4a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Recycle delay = 1.000 sec
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2.98
8
6
4
2
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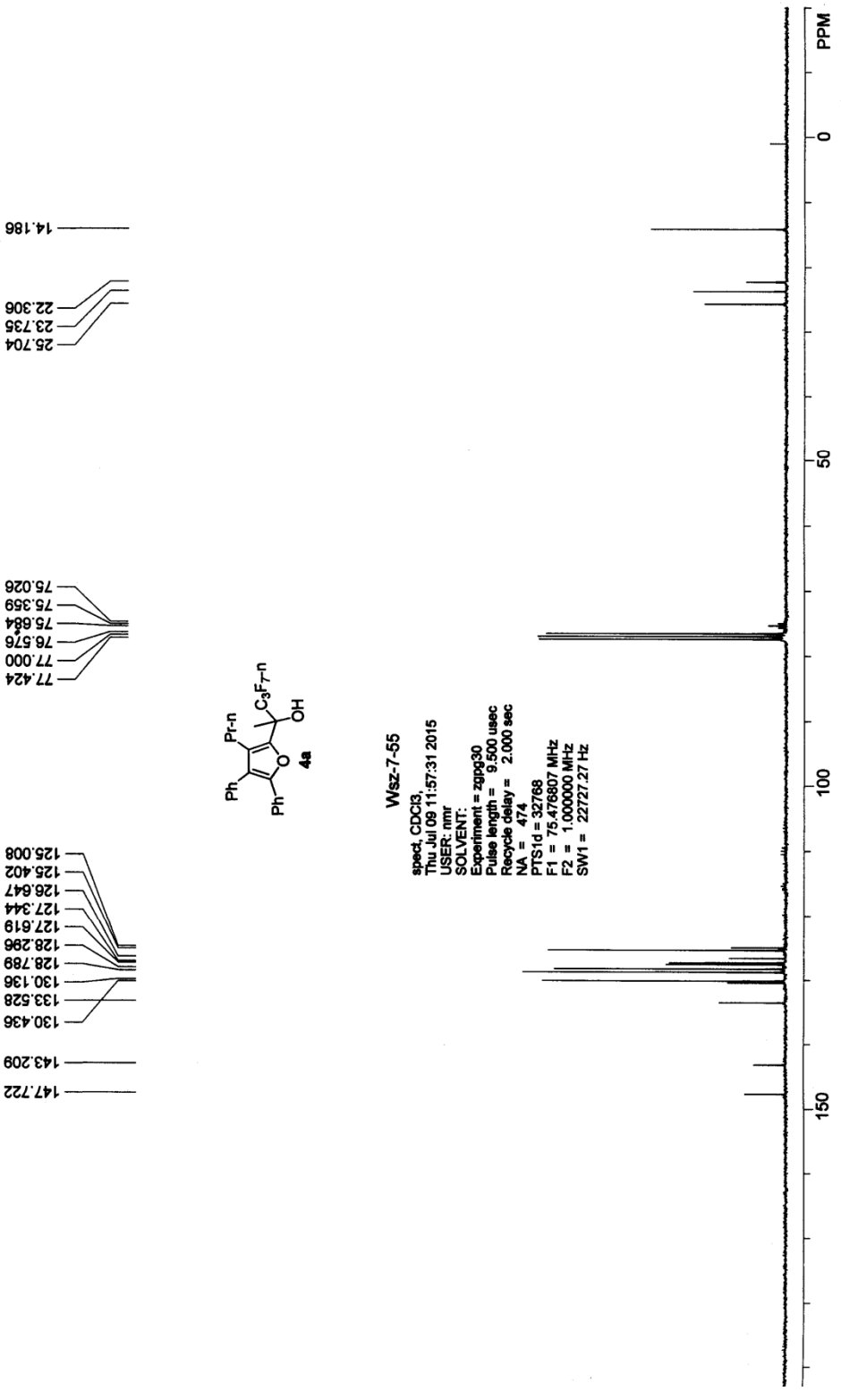
PPM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Ph
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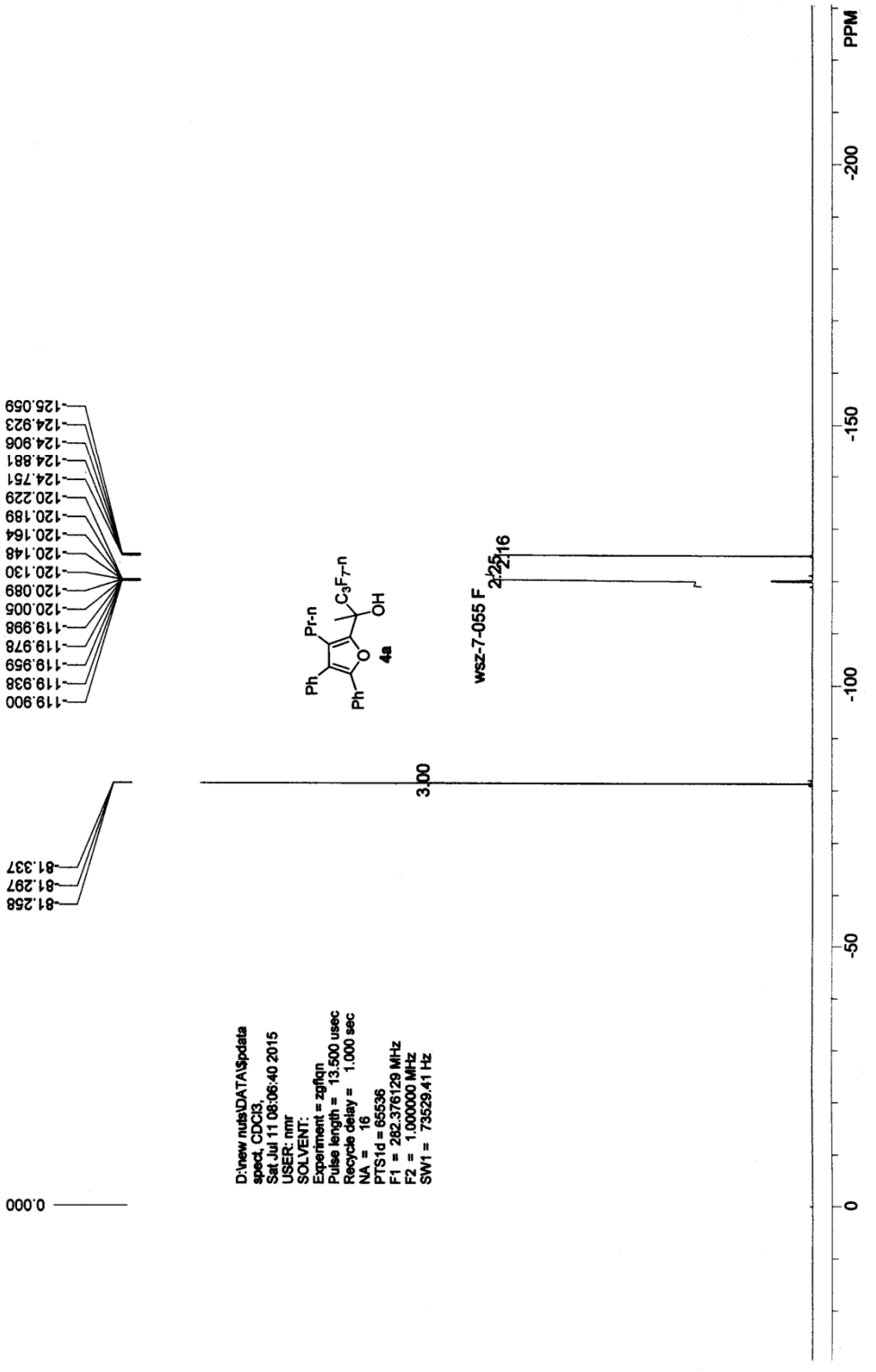
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120.164
120.189
120.229
124.751
124.881
124.906
124.923
125.059
3.00
2.25
2.16
wsz-7-055 F
Pr-n
C_3F_7-n
OH
Ph
Ph
4a
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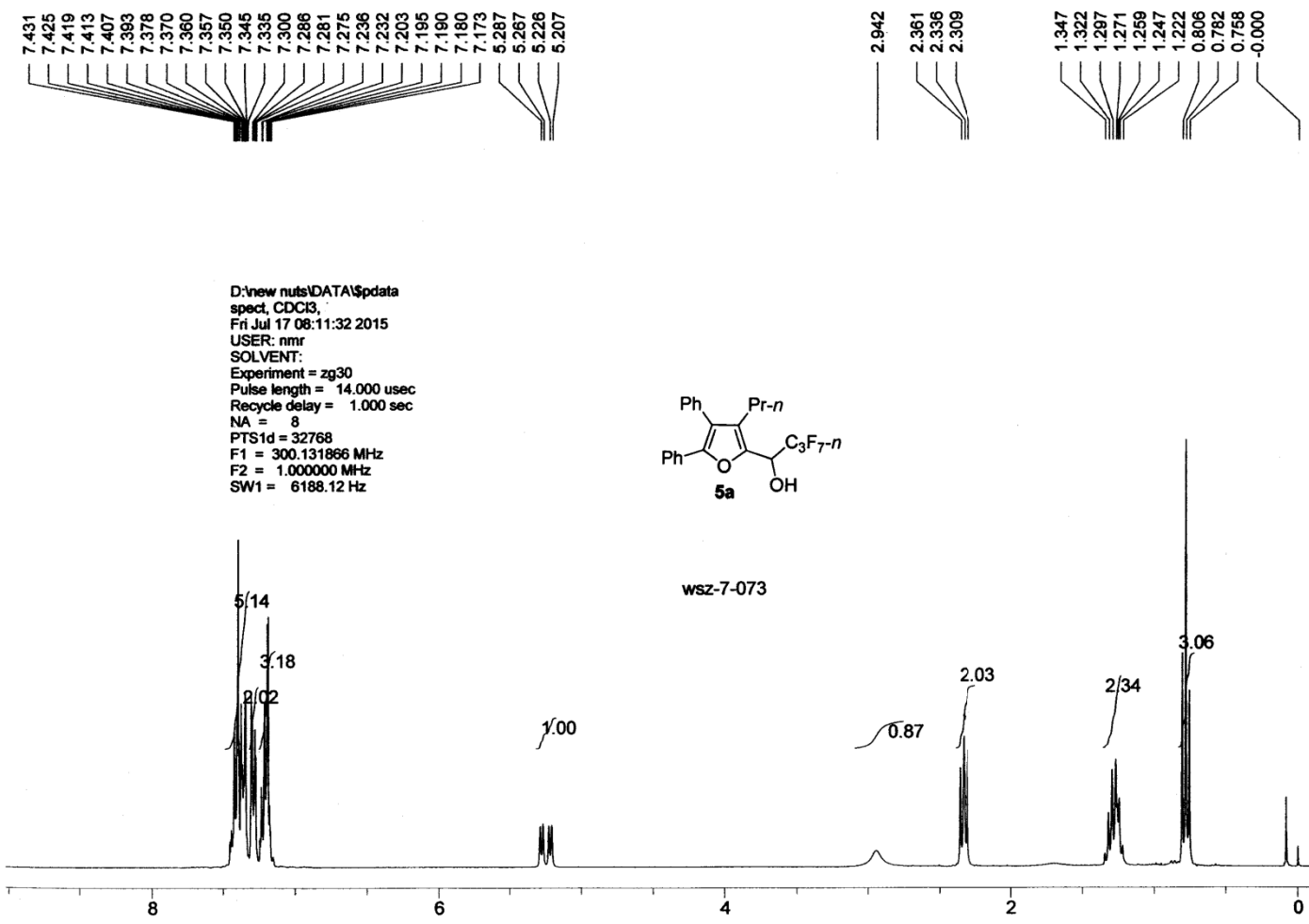
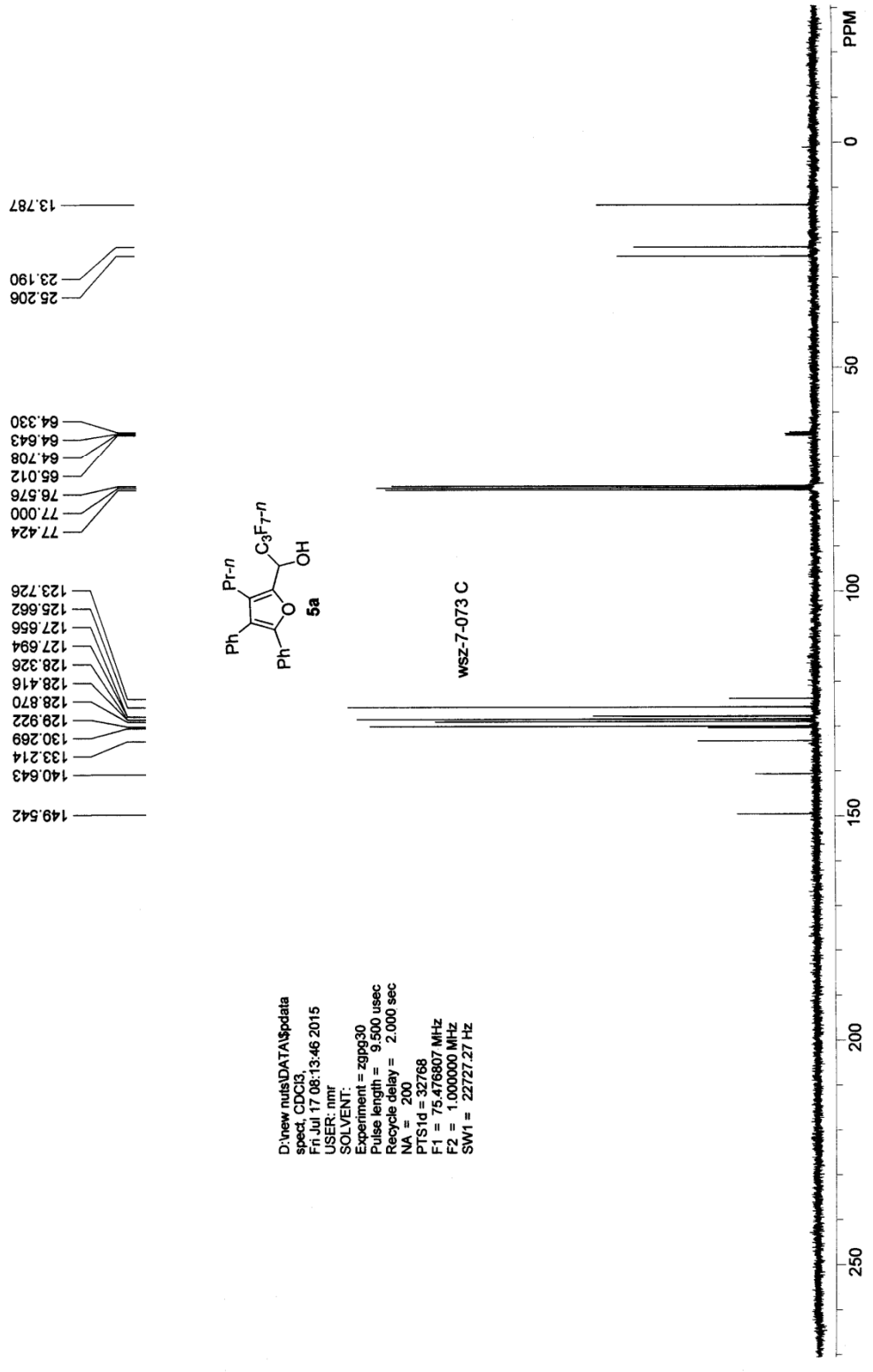
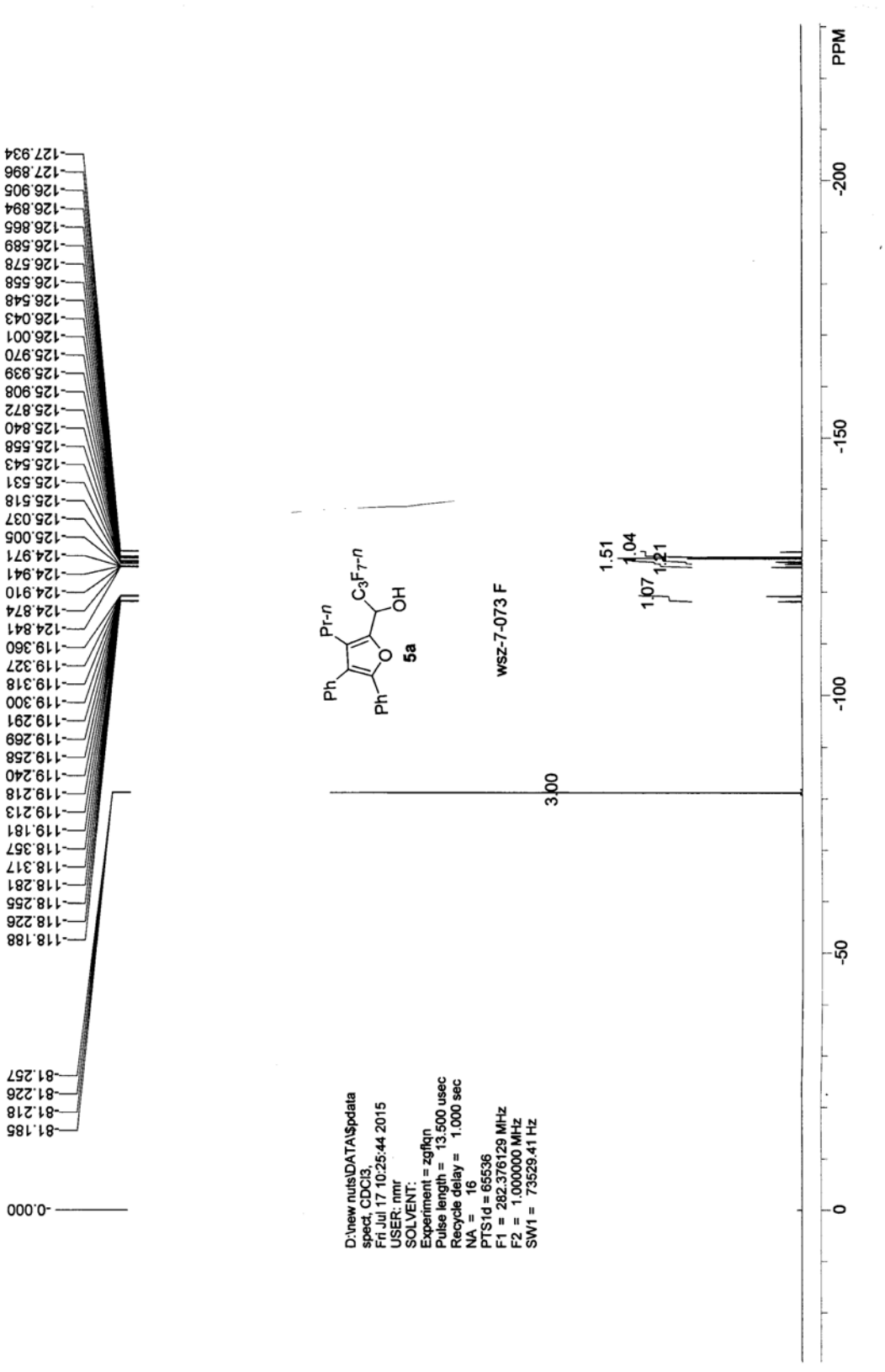
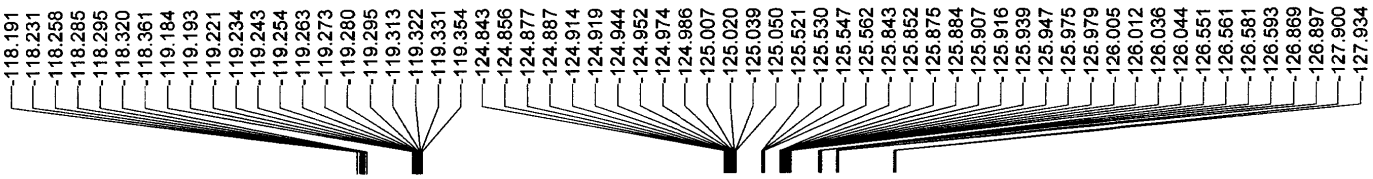
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTS1d = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0
-50
-100
-150
-200
PPM

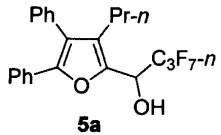

7.431
7.425
7.419
7.413
7.407
7.393
7.378
7.370
7.360
7.357
7.350
7.345
7.335
7.300
7.286
7.281
7.275
7.236
7.232
7.203
7.195
7.190
7.180
7.173
5.287
5.267
5.226
5.207
2.942
2.361
2.336
2.309
1.347
1.322
1.297
1.271
1.259
1.247
1.222
0.806
0.782
0.758
-0.000
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 17 08:11:32 2015
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
Pr-n
Ph
O
C3F7-n
OH
5a
wsz-7-073
5.14
3.18
2.02
1.00
0.87
2.03
2.34
3.06
8
6
4
2
0
PPM


13.787
23.190
25.206
64.330
64.643
64.708
65.012
76.576
77.000
77.424
123.726
125.662
127.656
127.694
128.326
128.416
128.870
128.922
130.269
133.214
140.643
149.542
Ph
Pr-n
C3F7-n
OH
Ph
O
5a
wsz-7-073 C
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 17 08:13:46 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 200
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM


127.934
127.896
126.905
126.894
126.865
126.589
126.578
126.558
126.548
126.043
126.001
125.970
125.939
125.908
125.872
125.840
125.558
125.543
125.531
125.518
125.037
125.005
124.971
124.941
124.910
124.874
124.841
119.360
119.327
119.318
119.300
119.291
119.269
119.258
119.240
119.218
119.213
119.181
118.357
118.317
118.281
118.255
118.226
118.188
81.257
81.226
81.218
81.185
0.000
Ph
Pr-n
O
C3F7-n
OH
5a
wsz-7-073 F
3.00
1.51
1.04
1.07
1.21
D:\new nuts\DATA\5pdata
spect, CDCl3,
Fri Jul 17 10:25:44 2015
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
0
-50
-100
-150
-200



wsz-7-073-F
spect, CDCl3,
Fri Jul 17 10:24:26 2015
USER: nmr
SOLVENT:
Experiment = zgflqn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 20



5a