Supporting Information

Highly Diastereroselective Synthesis of Dihydrofurans and Dihydropyrroles via Pyridine Catalyzed Formal [4+1] Annulation

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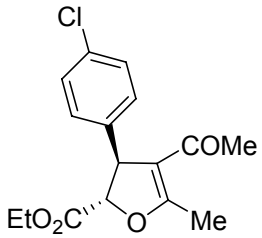
General Information. All reaction flasks were dried by flame. And all reactions were carried out under N_2 unless otherwise noted. All solvents were purified according to standard methods unless otherwise noted.

^{1}H NMR and ^{13}C NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer or VARIAN Mercury 400 MHz spectrometer in chloroform-d_3 unless otherwise noted. ^{1}H NMR chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard unless otherwise noted. The data is being reported as s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration. ^{13}C NMR chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard unless otherwise noted.

Content

General procedure for synthesis of dihydrofurans A: (**3a** as an example) Fe(TCP)Cl (2.0 mg, 2 μmol), **1a** (44 mg, 0.20 mmol), pyridine (1.0 μL, 0.01 mmol) was mixed in toluene (1.0 mL). The reaction mixture was heated to 40 °C, and EDA (46 mg, 0.4 mmol) in toluene (1.0 mL) was added within 3 h via a syringe pump. After the reaction was complete, the resulting mixture was cooled to room temperature, and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel) to give **3a** as a pure product, dr > 50/1. Yield: 59.2 mg (96%).

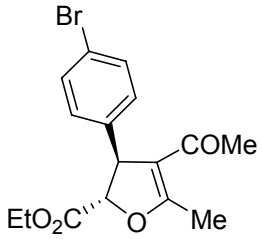
4-Acetyl-3-(4-chlorophenyl)-5-methyl-2,3-dihydrofuran-2-carboxylate (3a):



3a

Solid. Yield: 96%. Dr > 50/1. ^{1}HNMR (300 MHz, $CDCl_3$/TMS) for trans- isomer: δ 7.33 (d, *J* = 8.7 Hz, 2 H), 7.19 (d, *J* = 8.1 Hz, 2 H), 4.74 (d, *J* = 4.8 Hz, 1 H), 4.48 (d, *J* = 4.5 Hz, 1 H), 4.34-4.27 (m, 2 H), 2.44 (s, 3 H), 2.00 (s, 3 H), 1.34 (t, *J* = 6.9 Hz, 3 H).

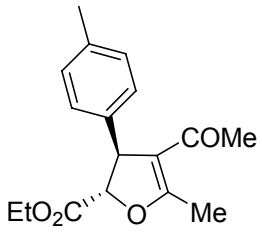
Ethyl 4-acetyl-3-(4-bromophenyl)-2,3-dihydro-5-methylfuran-2-carboxylate (3b):



3b

Solid.. Yield: 66.4 mg (94%). Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.47 (d, *J* = 8.7 Hz, 2 H), 7.11 (d, *J* = 8.4 Hz, 2 H), 4.72 (d, *J* = 4.8 Hz, 1 H), 4.45 (d, *J* = 4.5 Hz, 1 H), 4.31-4.24 (m, 2 H), 2.42 (s, 3 H), 1.99 (s, 3 H), 1.34 (t, *J* = 6.6 Hz, 3 H).

Ethyl-4-acetyl-2,3-dihydro-5-methyl-3-p-tolylfuran-2-carboxylate (3c):

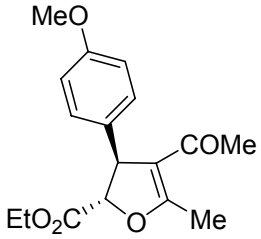


3c

Liquid. Yield: 50.7 mg (88%). Dr > 50/1. ^{1}H NMR (300 MHz,

$CDCl_3$/TMS) for trans isomer: δ 7.18-7.11 (m, 4 H), 4.75 (d, *J* = 5.1 Hz, 1 H), 4.44 (d, *J* = 4.8 Hz, 1 H), 4.31-4.26 (m, 2 H), 2.43 (s, 3 H), 2.34(s, 3 H), 1.95 (s, 3 H), 1.32 (t, *J* = 6.6 Hz, 3H).

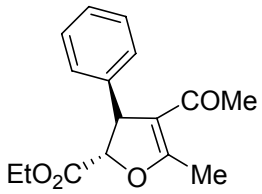
Ethyl-4-acetyl-2,3-dihydro-3-(4-methoxyphenyl)-5-methylfuran-2-carboxylate (3d):



3d

Solid. Yield: 51.7 mg (85%). Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.15 (d, *J* = 8.4 Hz, 2 H), 6.88 (d, *J* = 8.4 Hz, 2 H), 4.74 (d, *J* = 5.1 Hz, 1H), 4.44(d, *J*= 4.5 Hz, 1 H), 4.31-4.26 (m, 2 H), 3.80 (s, 3 H), 2.43 (s, 3 H), 1.95 (s, 3 H), 1.32 (t, *J* = 7.0 Hz, 3 H).

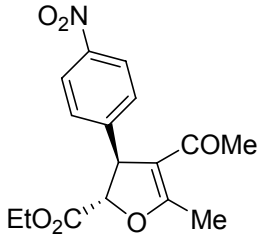
Ethyl 4-acetyl-2,3-dihydro-5-methyl-3-phenylfuran-2-carboxylate (3e):



3e

Solid. Yield: 51.5 mg (94%). Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans- isomer: δ 7.39-7.23 (m, 5 H), 4.78 (d, *J* = 5.1 Hz, 1H), 4.49 (d, *J* = 4.5 Hz, 1 H), 4.32-4.28 (m, 2H), 2.45 (s, 3H), 1.97 (s, 3H), 1.34 (t, *J* = 7.2 Hz, 3H).

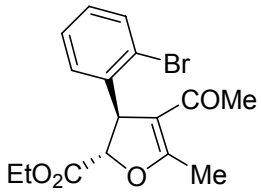
Ethyl 4-acetyl-2,3-dihydro-5-methyl-3-(4-nitrophenyl)furan-2-carboxylate (3f):



3f

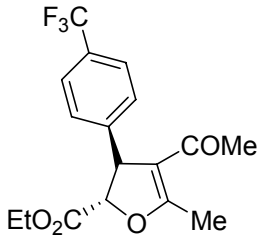
Solid. Yield: 59.6 mg (95%). Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans- isomer: δ 8.21 (d, *J* = 9.0 Hz, 2 H), 7.43 (d, *J* = 8.7 Hz, 2 H), 4.77 (d, *J* = 5.1 Hz, 1 H), 4.61 (d, *J* = 5.1Hz, 1 H), 4.35-4.27 (m, 2 H), 2.47 (s, 3 H), 2.10 (s, 3 H), 1.35 (t, *J* = 7.2 Hz, 3 H).

Ethyl 4-acetyl-3-(2-bromophenyl)-2,3-dihydro-5-methylfuran-2-carboxylate (3g):



3g Solid. Yield: 67.1 mg (95%). Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.63 (m, 1 H), 7.33 (m, 1 H), 7.20-7.16 (m, 2 H), 5.10 (d, *J* = 4.5 Hz, 1 H), 4.75 (d, *J* = 5.4 Hz, 1 H), 4.34-4.30 (m, 2 H), 2.47 (s, 3 H), 1.95 (s, 3 H), 1.36 (t, *J* = 7.2 Hz, 3 H).

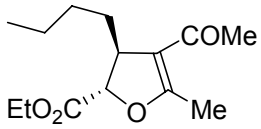
Ethyl4-acetyl-3-(4-(trifluoromethyl)phenyl)-2,3-dihydro-5-methylfuran-2-carboxylate (3i):



3i Liquid. Yield: 63.0 mg (92%)。Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.63 (d, *J* = 8.1 Hz, 2 H), 7.38 (d, *J* = 8.4 Hz, 2 H), 4.77 (d, *J* = 5.1 Hz, 1 H), 4.57 (d, *J* = 3.9 Hz, 1 H), 4.34-4.28 (m, 2 H), 2.46 (d, *J* = 0.9 Hz, 3 H), 2.05 (s, 3 H), 1.35 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (400 MHz, $CDCl_3$/TMS): δ 193.4, 169.4, 168.7, 146.2, 129.5 (q, *J* = 31.7 Hz), 128.0, 127.5, 126.0, 125.9, 125.3, 122.6, 115.3, 85.3, 62.0, 52.94, 29.5, 15.0, 14.0, 13.9; ^{19}F NMR (282 MHz, $CDCl_3$/TMS): δ -62.95; IR (film) *v*/cm^{-1} 3062 (w), 2925(w), 2853 (w), 1755 (s), 1621 (s), 1602 (s), 1323 (s); MS (EI) *m*/*z* (% rel intensity) 342 (17.6) M^+, 43 (100); HRMS (EI) calcd for $C_{17}H_{17}F3O_4$ (M^+) 342.1079, found 342.1078.

General Procedure for synthesis of dihydrofurans B: Fe(TCP)Cl (2.0 mg, 0.002 mmol), **1j** (1.0 mmol), were mixed in a Schlenk tube. The tube was evacuated and backfilled with nitrogen. Pyridine (4.0 μL, 0.05 mmol) was added, followed by toluene (1.0 mL) . The reaction mixture was heated to 40 °C, and 2.0 equiv of EDA (228 mg, 2.0 mmol) in toluene (1.0 mL) was added within 8 h via a syringe pump. After the reaction was complete, the resulting mixture was cooled to room temperature, and concentrated. The residue was purified by flash chromatography (silica gel) to give the product **3j**.

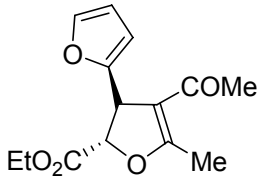
Ethyl 4-acetyl-3-butyl-2,3-dihydro-5-methylfuran-2-carboxylate (3j):



3j Liquid. Yield: 216.1 mg (85%). Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 4.65 (d, *J* = 3.6 Hz, 1 H), 4.23 (q, *J* = 7.2 Hz, 2 H), 3.32-3.29 (m, 1 H), 2.32 (s, 3 H), 2.26 (s, 3 H), 1.78-1.70 (m, 2 H), 1.50-1.26 (m, 7H), 0.95-0.90 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (300 MHz, $CDCl_3$/TMS): δ 193.6, 170.6, 167.1, 116.6, 82.3, 61.5, 47.6, 36.2, 35.9, 29.3, 19.3, 15.2, 14.0, 13.7; IR (film) *v*/cm^{-1} 3062 (w), 2925(w), 2853 (w), 1753(s), 1624 (s), 1600 (s), 1384 (m), 1192 (s); MS (EI) *m*/*z* (% rel intensity) 353 (1.7) $[M-H]^+$, 84 (100); HRMS (EI) calcd for $C_{14}H_{22}O_4$ (M^+) 254.1518, found 254.1514.

General Procedure for synthesis of dihydrofurans C: Fe(TCP)Cl (2.0 mg, 0.002 mmol), **1h** (1.0 mmol), were mixed in a Schlenktube. The tube was evacuated and backfilled with nitrogen. Pyridine (8.0 μL, 0.10 mmol) was added, followed by toluene (1.0 mL) . The reaction mixture was heated to 40 °C, and 2.0 equiv of EDA (228 mg, 2.0 mmol) in toluene (1.0 mL) was added within 16 h via a syringe pump. After the reaction was complete, the resulting mixture was cooled to room temperature, and concentrated. The residue was purified by flash chromatography (silica gel) to give the product **3h**.

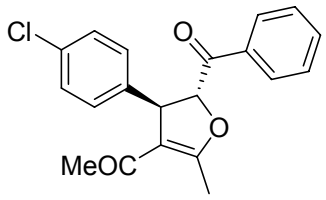
Ethyl 4-acetyl-3-(furan-2-yl)-2,3-dihydro-5-methylfuran-2-carboxylate (3h):



3h Solid. Yield: 224.4 mg (85%). Dr > 50/1. ^{1}HNMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.37 (m, 1 H), 6.33 (m, 1 H), 6.17 (d, *J* = 3.0 Hz, 1 H), 4.94 (d, *J* = 4.5 Hz, 1 H), 4.62 (d, *J* = 4.5 Hz, 1 H), 4.30 (q, *J* = 7.2 Hz, 2 H), 2.39 (d, *J* = 1.5 Hz, 1 H), 2.08 (s, 3 H), 1.33 (t, *J* = 7.2 Hz, 3 H).

General procedure for synthesis of dihydrofurans D: Fe(TCP)Cl (2.0 mg, 0.002 mmol), **1a** (0.20 mmol), were mixed in a Schlenk tube. The tube was evacuated and backfilled with nitrogen. Pyridine (1.0 μL, 0.01 mmol) was added, followed by toluene (1.0 mL). The reaction mixture was heated to 40 °C, and 2.0 equivalents of diazoacetophenone **4** (58mg, 0.4 mmol) in toluene (1.0 mL) was added within 3 h via a syringe pump. After the reaction was complete, the resulting mixture was cooled to room temperature, and concentrated. The residue was purified by flash chromatography (silica gel) to give **5**.

2-Benzoyl-4-acetyl-3-(4-chlorophenyl)-5-methyl-2,3-dihydrofuran (5) :

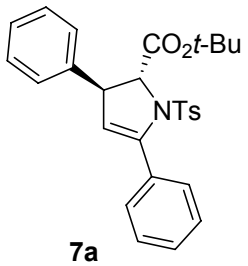


5

Solid. Yield: 62.6 mg (92%); Dr > 50/1. ^{1}H NMR (300 MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.88-8.50 (m, 2 H), 7.67-7.62 (m, 1 H), 7.52-7.46 (m, 2 H), 7.37-7.34 (m, 2 H), 7.22-7.19 (m, 2 H), 5.61 (d, J = 4.5 Hz, 1 H), 4.53 (dd, J = 4.8, 0.9 Hz, 1 H), 2.47 (d, J = 0.9 Hz, 3 H), 2.00 (s, 3 H).

General Procedure for synthesis of dihydropyrroles: (**7a** as an example). Fe(TCP)Cl (2.0 mg, 2 μmol), **6a** (180 mg, 0.50mmol), and 4-methylpyridine (2.5 μL, 0.025 mmol) was mixed in toluene (1.0 mL). The reaction mixture was heated to 40 °C, and BDA (150 mg, 1.0 mmol) in toluene (1.0 mL) was added within 20 h via a syringe pump. After the reaction was complete, the resulting mixture was cooled to room temperature, and concentrated under reduced pressure. The residue was purified by flash chromatography (silica gel) to give **7a** as a pure product.

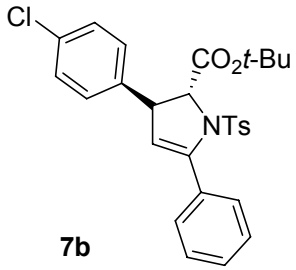
Tert-butyl 2,3-dihydro-3,5-diphenyl-1-tosyl-1H-pyrrole-2-carboxylate(7a):



7a

Solid. Yield: 218.5 mg (92%). Dr > 50/1. ^{1}H NMR (300MHz, $CDCl_3$/TMS) for trans-isomer: δ 7.58-7.55 (m, 2H), 7.32-7.30 (m, 3 H), 7.22-7.18（m，2 H），7.09-6.96 (m, 5H), 6.63 (m，2 H), 5.22 (d, J = 3.3 Hz, 1 H)，4.50 (d, J = 3.6 Hz, 1 H), 3.91 (t，J = 3.6 Hz, 1 H) , 2.32 (s, 3 H) ,1.52 (s, 9 H); ^{13}C NMR (300 MHz, $CDCl_3$/TMS): δ 170.0, 145.8, 143.7, 141.8, 133.7, 132.2, 129.4, 129.0, 128.4, 127.8, 127.7, 126.9, 126.6, 114.9, 82.3, 71.1, 50.3, 28.0, 21.5; IR (film) v/cm^{-1} 1748(m), 1361(m),1258(s), 1149(s), 1090(s),1021(s); MS (EI) m/z (% rel intensity) 475 (0.9) M^+, 57 (100); HRMS (EI) calcd for $C_{28}H_{29}NO_4S$ (M^+) 475.1817, found 475.1816.

Tert-butyl 3-(4-chlorophenyl)-2,3-dihydro-5-phenyl-1-tosyl-1H-pyrrole-2-carboxylate (7b):

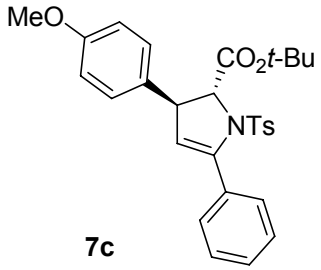


7b

Solid. Yield: 236.7 mg (93 %). ^{1}H NMR (300 MHz,

$CDCl_3$/TMS): δ 7.66-7.63 (m, 2 H), 7.41-7.39 (m, 3 H), 7.28-7.25 (m, 2 H)，7.08-7.04 (m, 4 H), 6.68 (d，*J* = 8.1 Hz，2 H)， 5.30 (d, *J* = 3.9 Hz，1 H)，4.55 (d, *J* = 3.0 Hz, 1 H), 3.96 (t, *J* = 3.0 Hz, 1 H)， 2.42 (s, 3 H) , 1.59 (s, 9 H); ^{13}C NMR (300 MHz, $CDCl_3$/TMS): δ 169.8, 146.3, 144.0, 140.5, 133.7, 132.4, 132.1, 129.3, 129.2, 128.5, 128.3, 128.1, 127.8, 127.7, 114.2, 82.5, 71.2, 49.4, 28.0, 21.5; IR (film) *v*/cm^{-1} 1719(m), 1361(m),1167(s), 1088(s); MS (EI) *m*/*z* (% rel intensity) 509 (1.4) M^+, 57 (100); HRMS (EI) calcd for $C_{28}H_{28}NO_4SCl$ (M^+) 509.1428, found 509.1431.

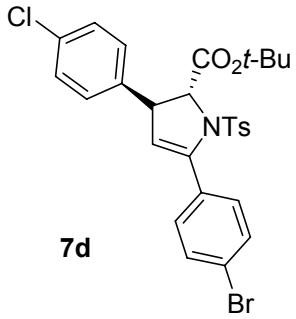
Tert-butyl-2,3-dihydro-3-(4-methoxyphenyl)-5-phenyl-1-tosyl-1H-pyrrole-2-carb oxylate(7c):



7c

Solid. Yield: 207.3 mg (82 %). ^{1}H NMR (400MHz, $CDCl_3$/TMS): δ 7.63-7.60 (m, 2 H), 7.39-7.29 (m, 5 H), 7.10 (m, 2 H), 6.63 (s，4 H), 5.25 (d，*J* = 3.3 Hz，1 H)，4.52 (d, *J* = 4.2 Hz, 1 H), 3.94 (t，*J* = 3.6 Hz，1 H), 3.79 (s, 3 H) , 2.42 (s, 3 H) , 1.59 (s，9 H); ^{13}C NMR (400 MHz, $CDCl_3$/TMS): δ 170.1, 158.4, 145.4, 143.6, 134.1, 132.3, 129.4, 129.0, 128.4, 128.0, 127.9, 127.7, 127.6, 115.2, 113.8, 82.2, 71.3, 55.2, 49.8, 28.0, 21.5; IR (film) *v*/cm^{-1} 1724 (m), 1488 (m), 1362(m), 1291(m), 1258 (m), 1243 (m), 1167(s), 1087 (s),1069 (m), 1069 (m); MS (EI) *m*/*z* (% rel intensity) 404 (11.2) [M-CO_2*t*-Bu]$^+$, 57 (100); HRMS (EI) calcd for $C_{29}H_{31}NO_5S$(M^+) 505.1923, found 505.1928.

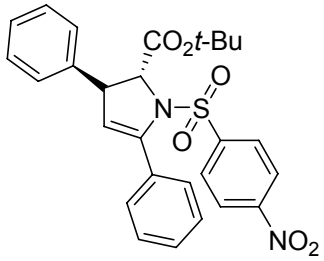
Tert-butyl-5-(4-bromophenyl)-3-(4-chlorophenyl)-2,3-dihydro-1-tosyl-1H-pyrrole -2-carboxylate(7d):



7d

Solid. Yield: 270.5 mg (92 %). ^{1}H NMR (400MHz, $CDCl_3$/TMS): δ 7.52 (s, 4 H), 7.25 (d, *J* = 8.7 Hz，2H), 7.06 (m, 4 H), 6.65 (d，*J* = 3.3 Hz，2 H), 5.33 (d，*J* = 3.6 Hz，1 H)，4.53 (d, *J* = 3.3 Hz, 1 H), 3.95 (t，*J* = 3.3 Hz, 1 H), 2.43 (s, 3 H) , 1.59 (s, 9 H); ^{13}C NMR (300 MHz, $CDCl_3$/TMS): δ 169.6, 145.3, 144.3, 140.2, 133.5, 132.5, 131.1, 129.9, 129.4, 128.5, 128.0, 127.8, 127.7, 123.4, 117.8, 82.7, 71.1, 49.5, 28.0, 21.5; IR (film) *v*/cm^{-1} 1723 (m), 1363 (m), 1166(s),1088 (m), 1070 (m), 1010(m); MS (EI) *m*/*z* (% rel intensity) 587 (0.34) M^+, 57 (100); HRMS (EI) calcd for $C_{28}H_{27}NO_4SClBr$(M^+) 587.0533, found 587.0537.

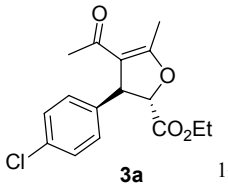
Tert-butyl-2,3-dihydro-3,5-diphenyl-1-(4'-nitrobenzene-sulfonyl)-1H-pyrrole-2-carboxylate (7e):



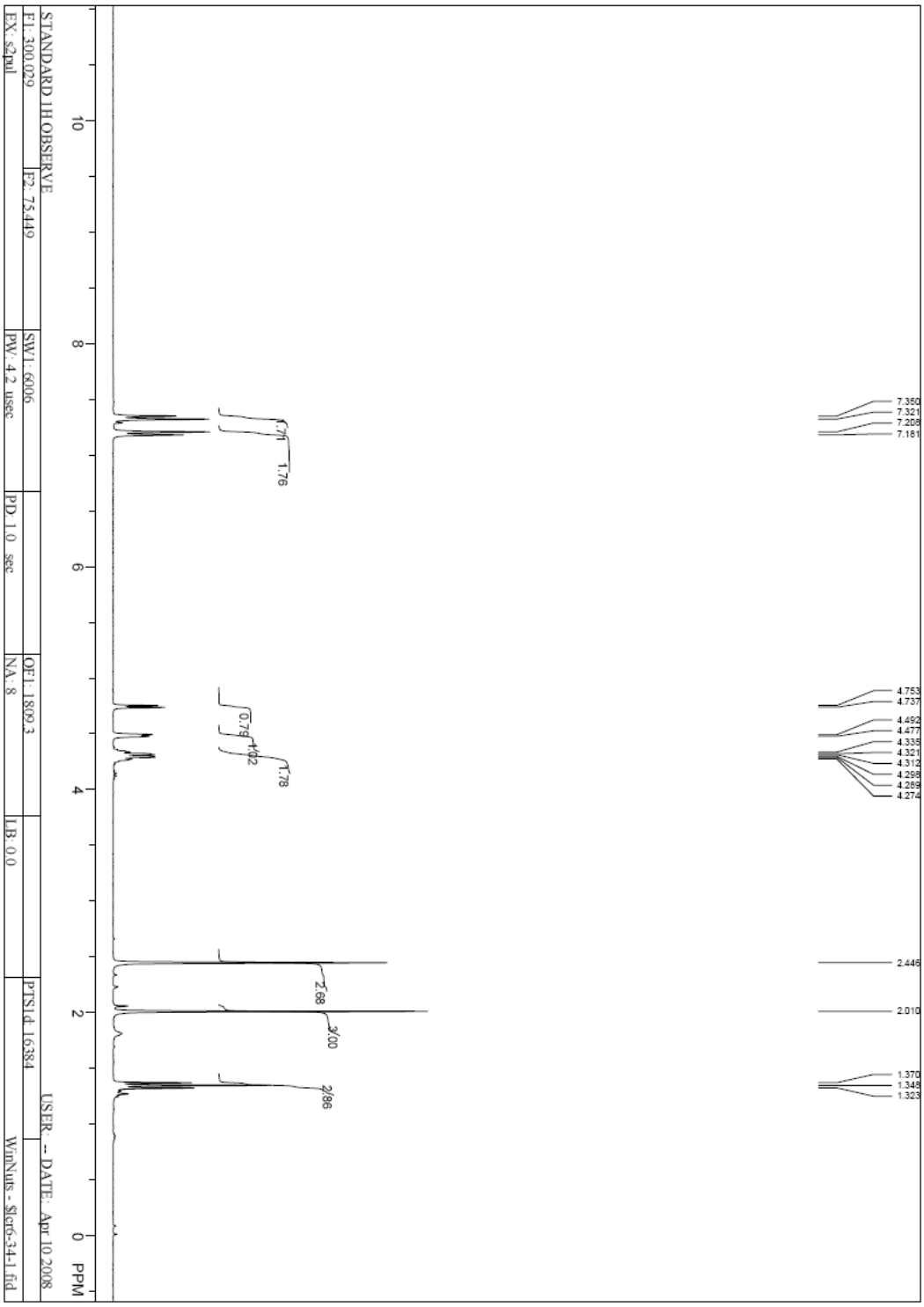
7e

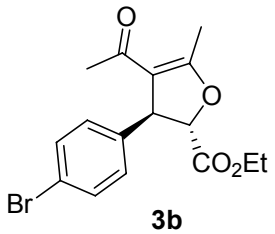
Solid. Yield: 230.1 mg (91 %). ^{1}H NMR (400 MHz, $CDCl_3$/TMS): δ 8.00-7.97 (m, 2 H), 7.65-7.62 (m, 2 H), 7.53-7.50 (m, 2 H), 7.44-7.40 (m, 3 H), 7.17-7.07 (m, 3 H), 6.82 (d，J = 7.2 Hz，2 H), 5.49 (d，J = 3.6 Hz，1 H)，4.71 (d, J = 3.3 Hz, 1 H), 4.07 (t，J = 3.0 Hz，1 H), 1.63 (s，9 H). ^{13}C NMR (300 MHz, $CDCl_3$/TMS): δ 169.3, 150.1, 145.1, 142.3, 141.6, 131.5, 129.5, 128.8, 128.6, 128.4, 128.0, 127.0, 126.4, 123.7, 115.6, 82.9, 71.5, 49.6, 28.0. IR (film) v/cm^{-1} 1725 (m), 1522 (m),1364(s), 1364(m),1304 (m), 1770 (s), 1087 (m); MS (EI) m/z (% rel intensity) 506 (0.85) M^+, 219 (100); HRMS (EI) calcd for $C_{27}H_{26}N_2O_6S$ (M^+) 506.1512, found 506.1517.

NMR Data of Compounds:

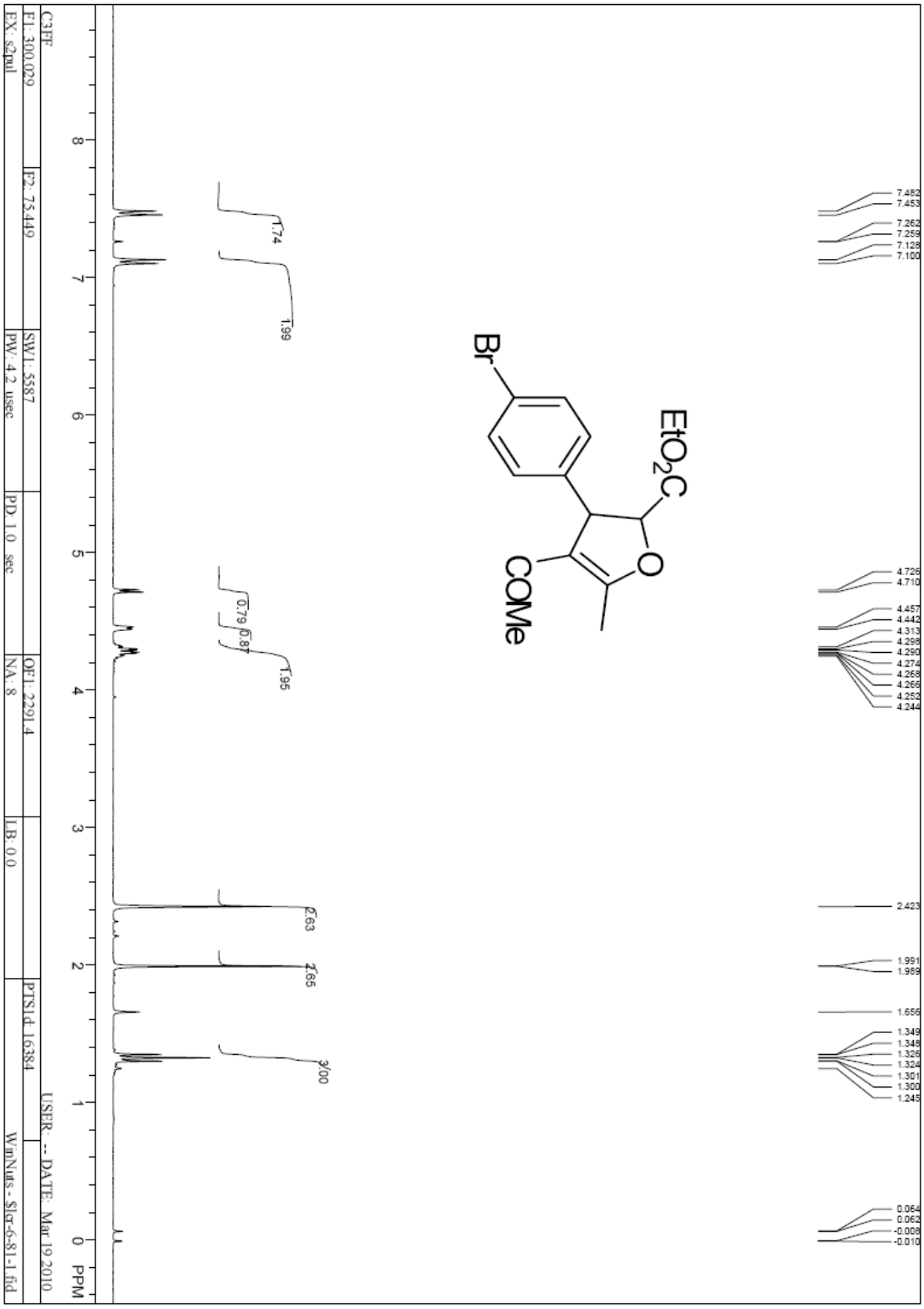


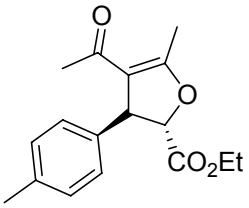
3a ^{1}H NMR (300 M Hz in CD_3Cl)





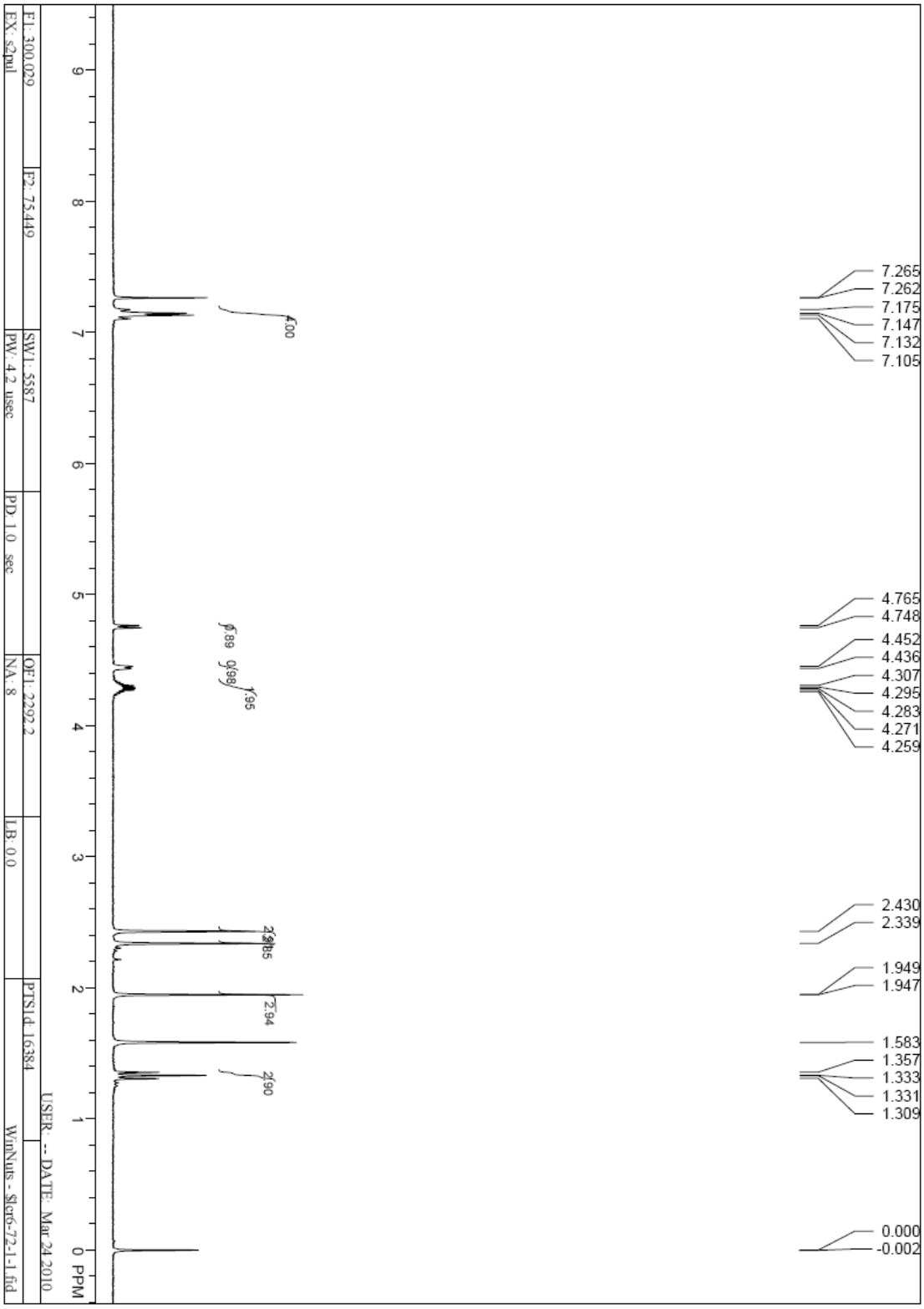
3b ^{1}H NMR (300 M Hz in CD_3Cl)

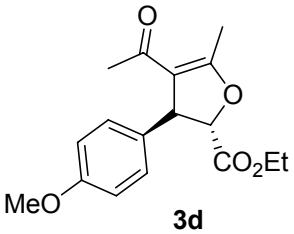




3c

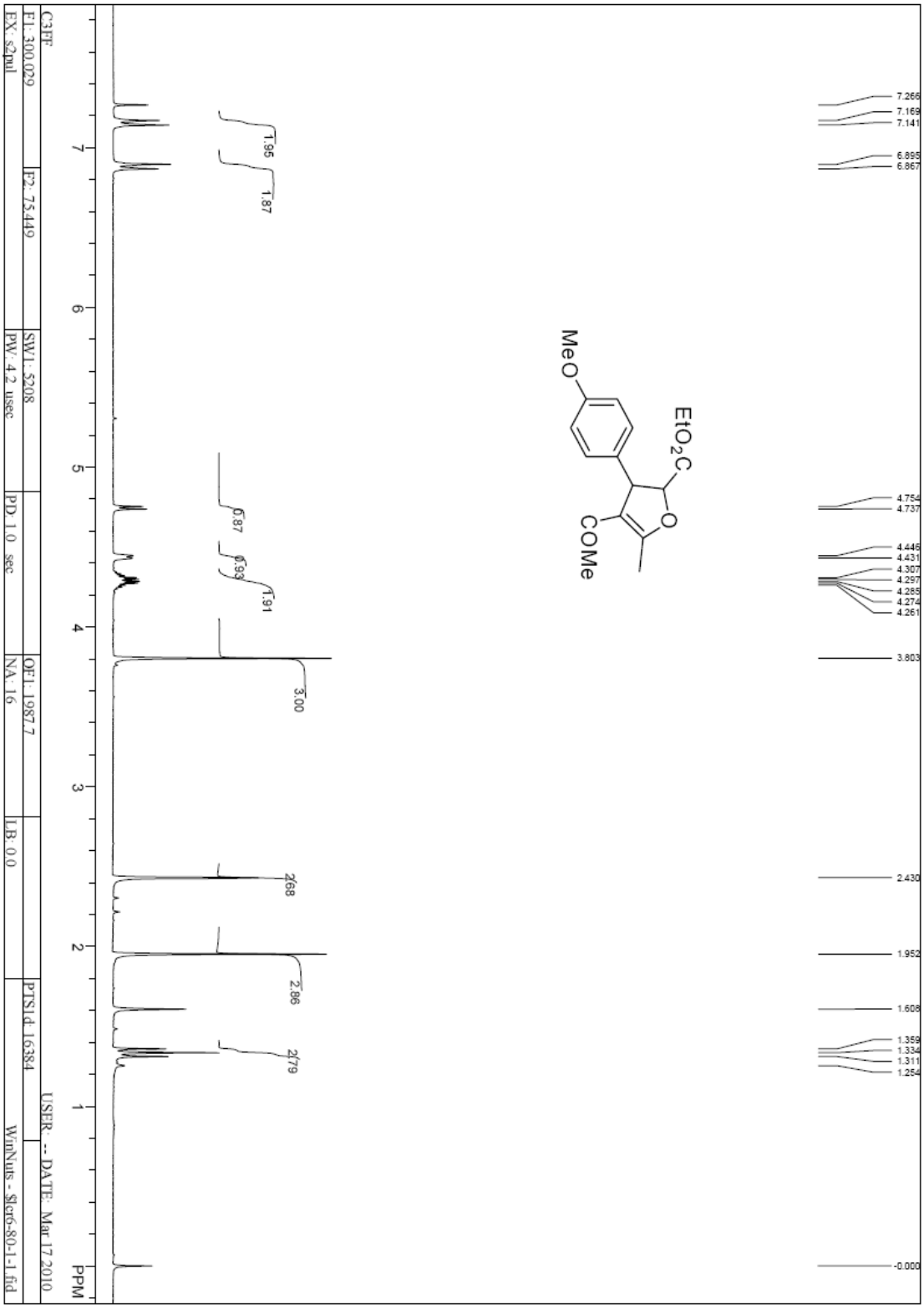
^{1}H NMR (300 M Hz in CD_3Cl)

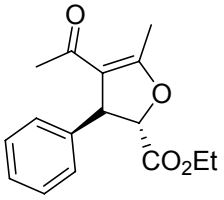




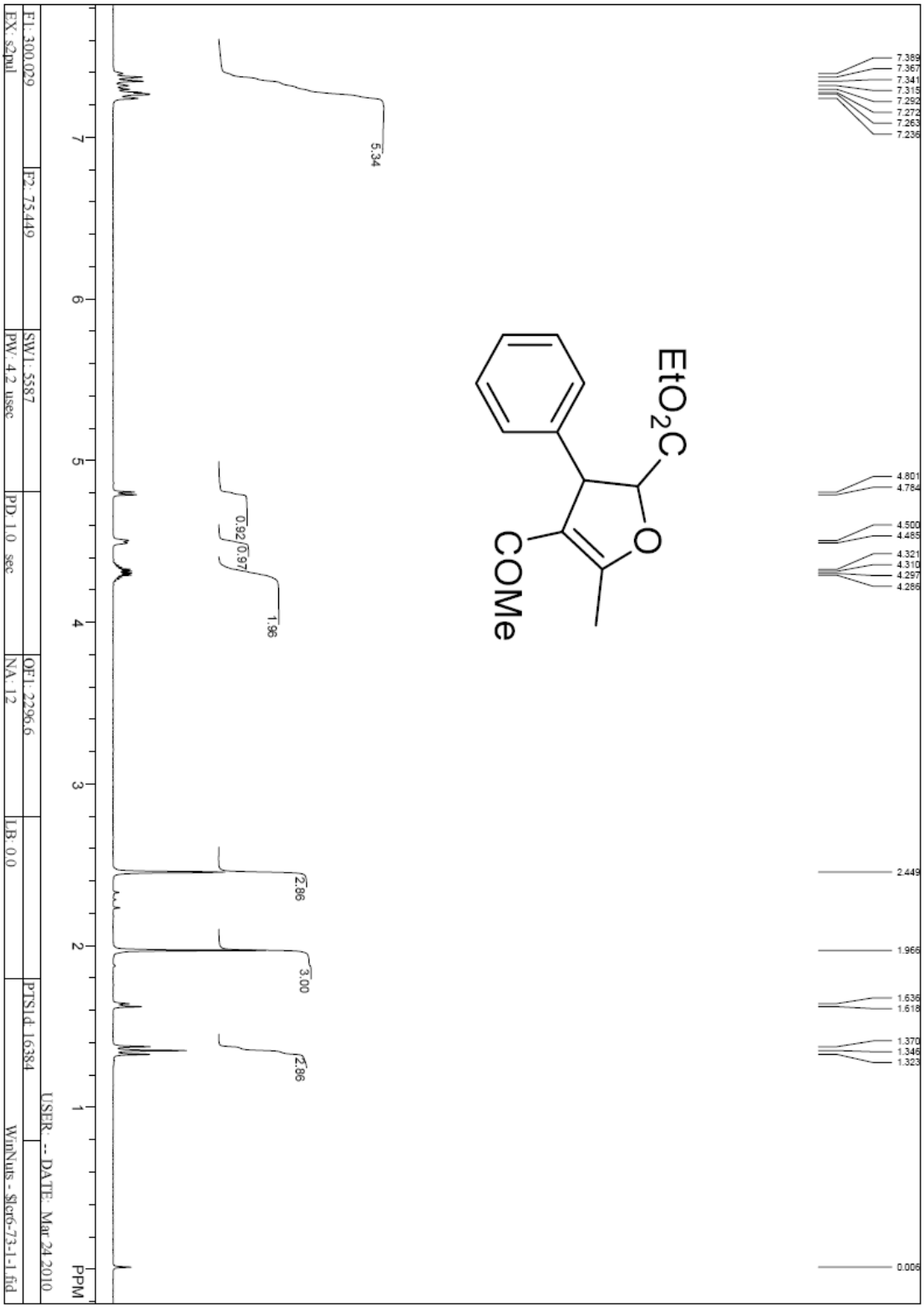
3d

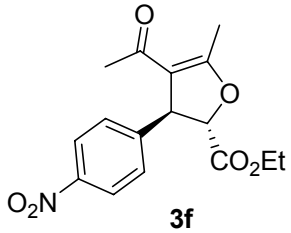
^{1}H NMR (300 M Hz in CD_3Cl)





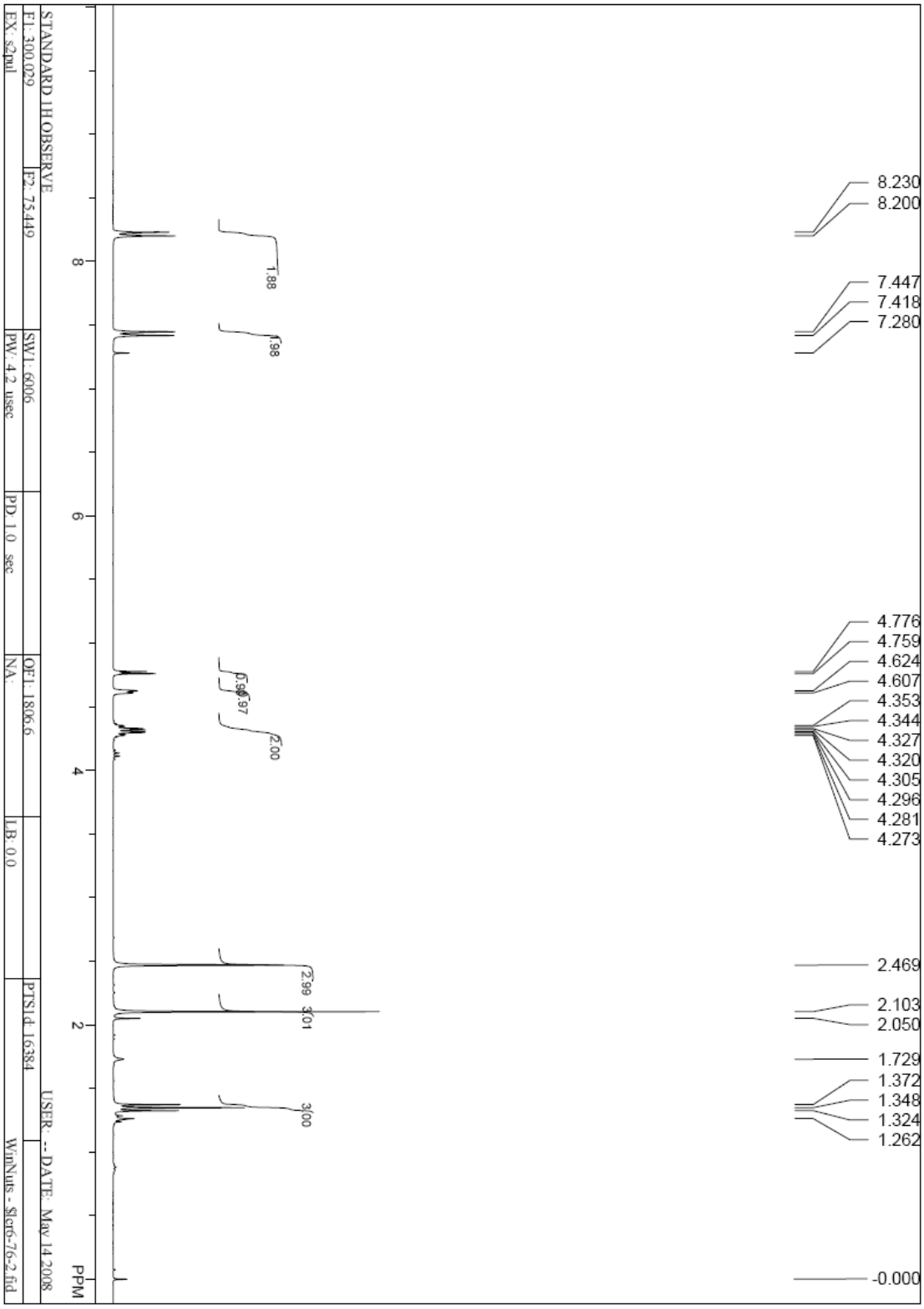
3e ^{1}H NMR (300 M Hz in CD_3Cl)

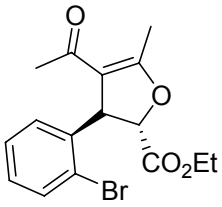




3f

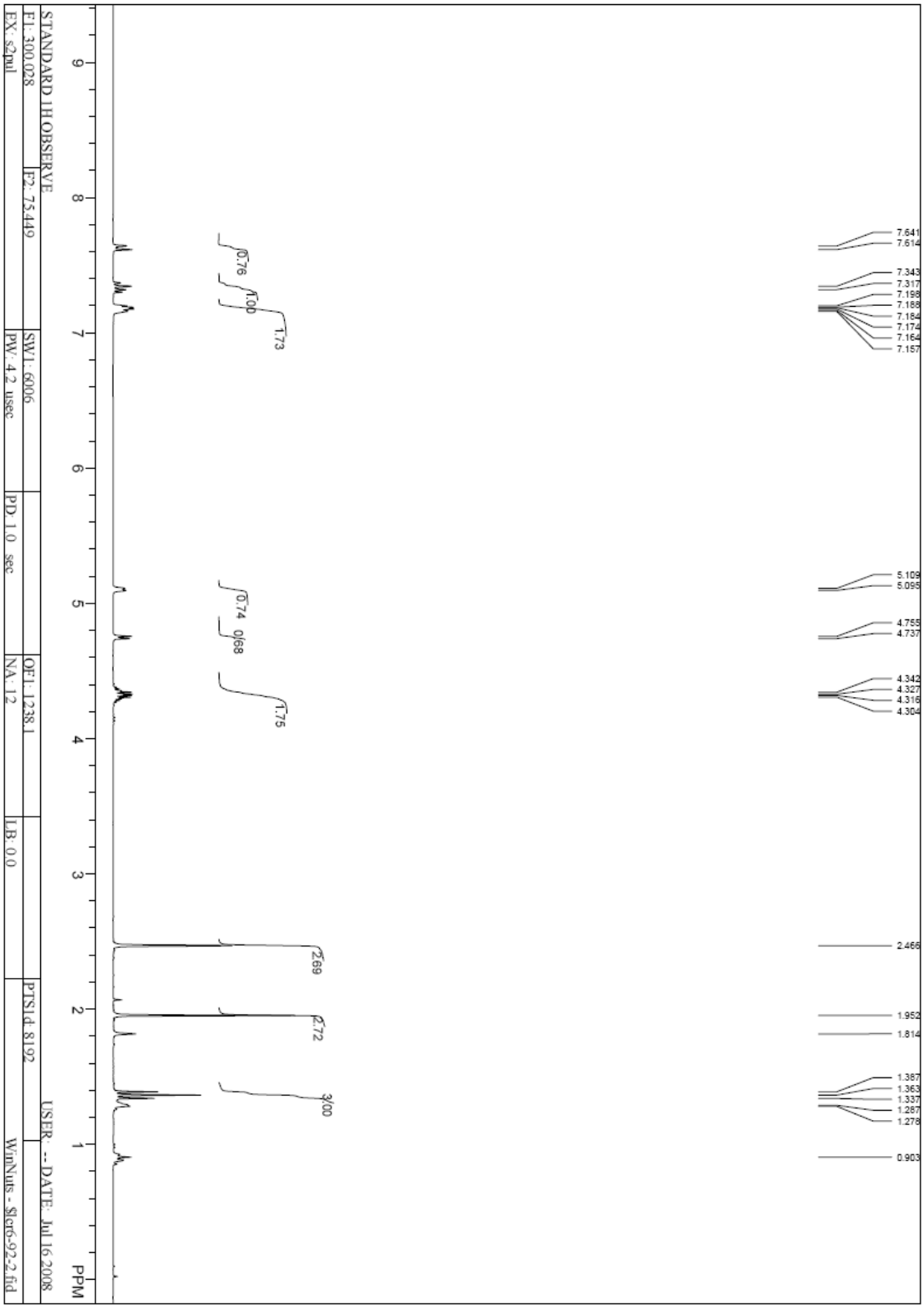
^{1}H NMR (300 M Hz in CD_3Cl)

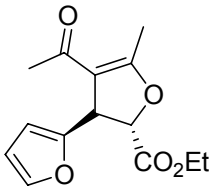




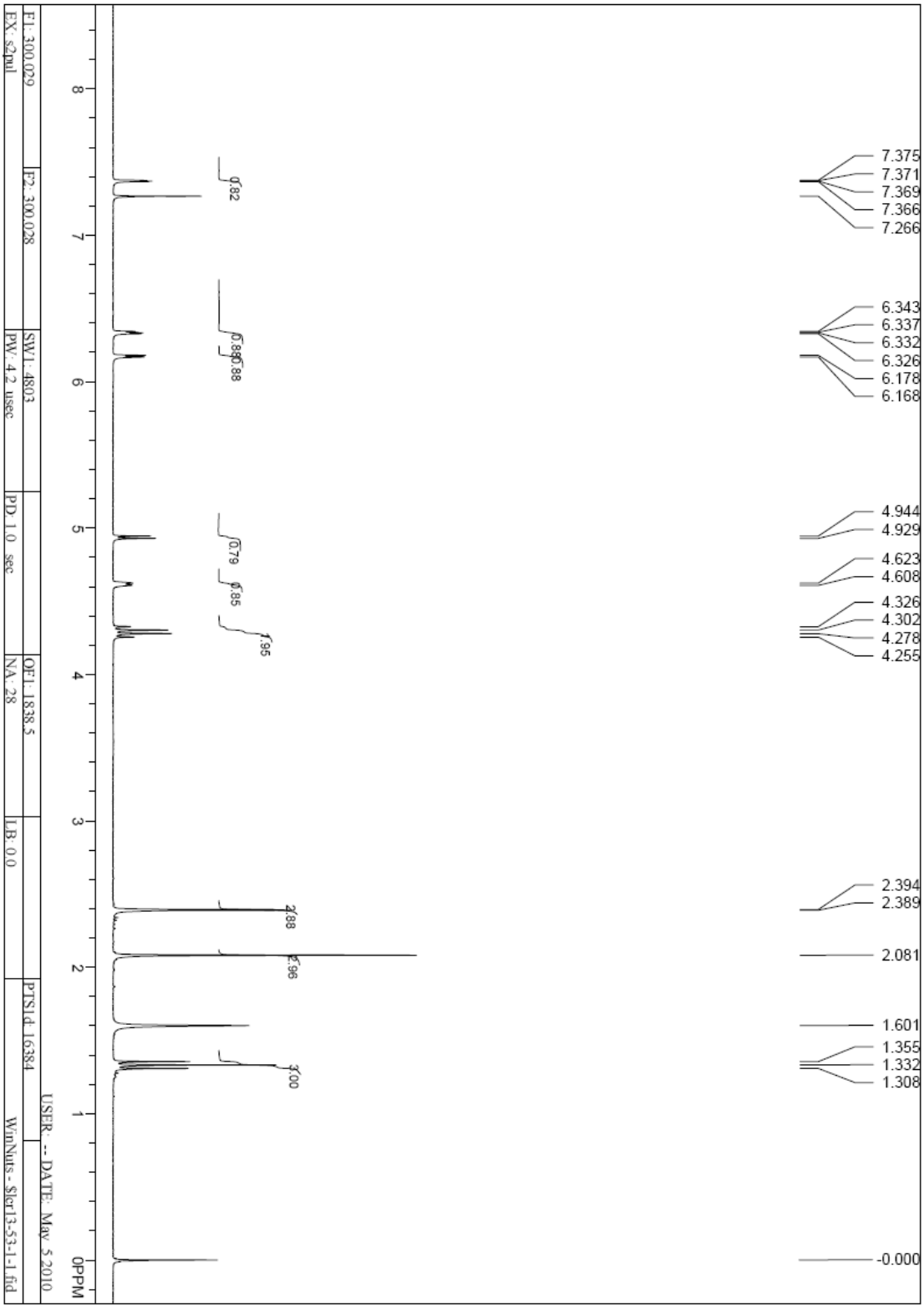
3g

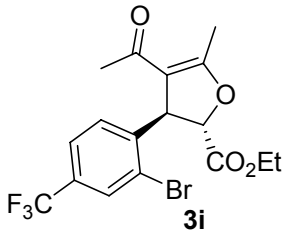
^{1}H NMR (300 M Hz in CD_3Cl)





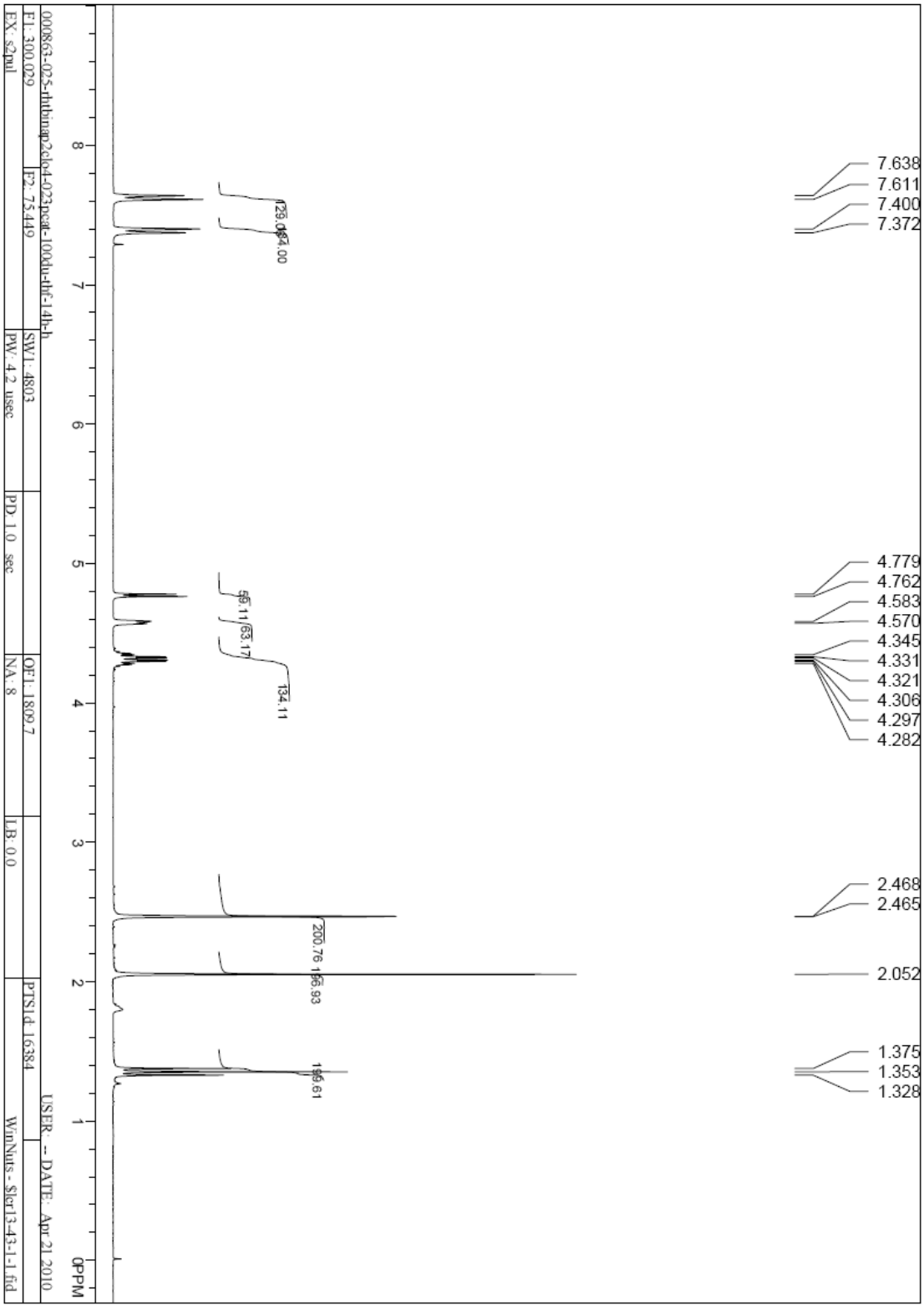
3h ^{1}H NMR (300 M Hz in CD_3Cl)

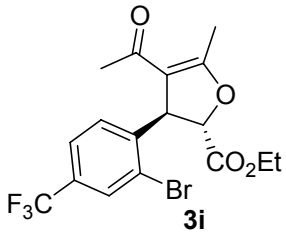




3i

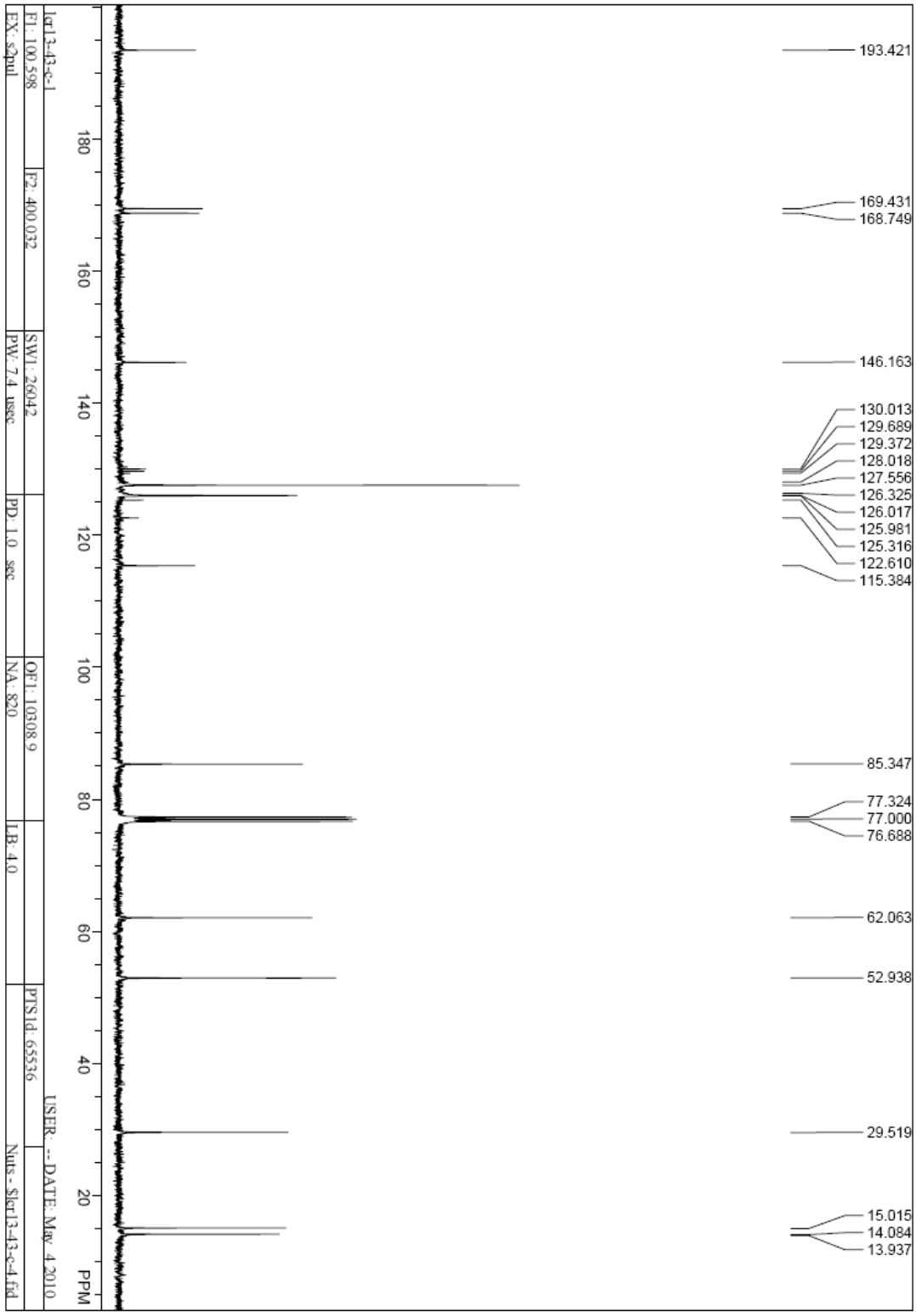
^{1}H NMR (300 M Hz in CD_3Cl)

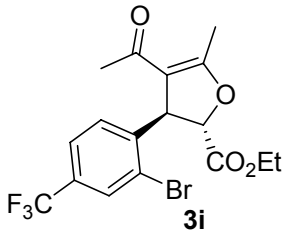




3i

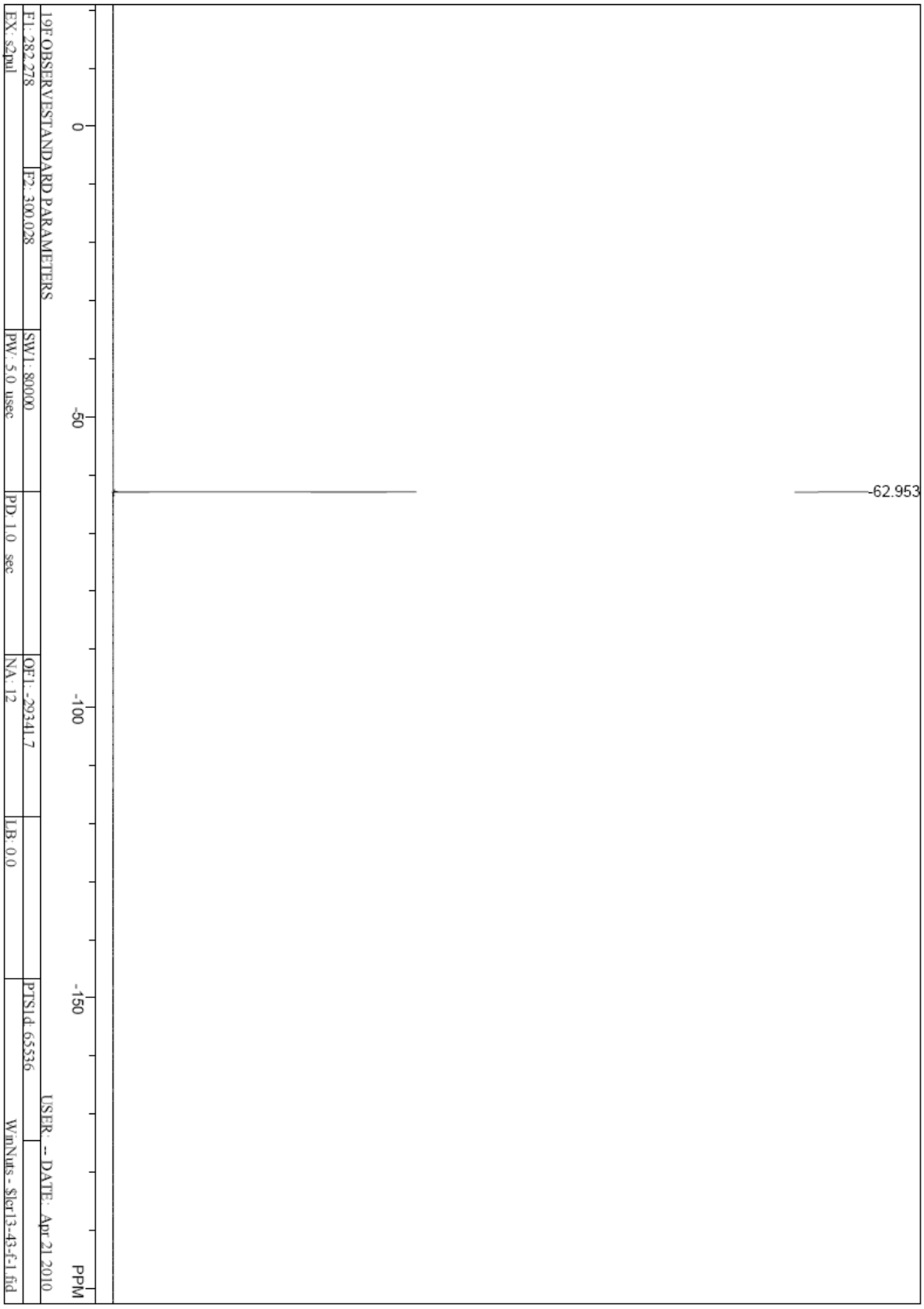
^{13}C NMR (100 M Hz in CD_3Cl)

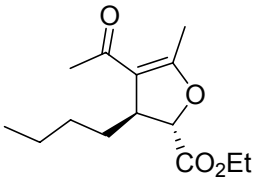




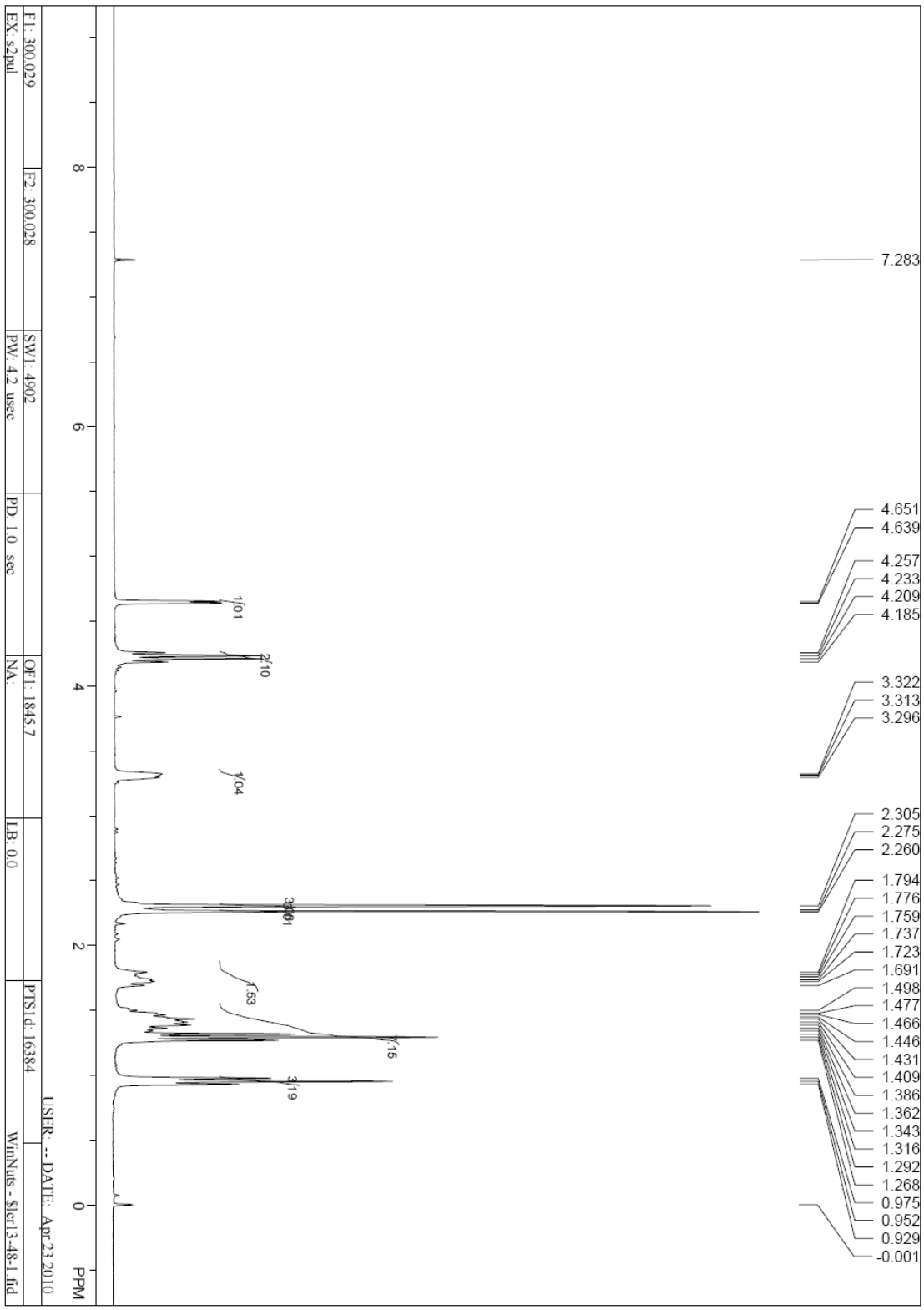
3i

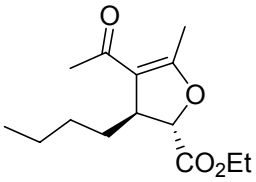
^{19}F NMR (282 M Hz in CD_3Cl)



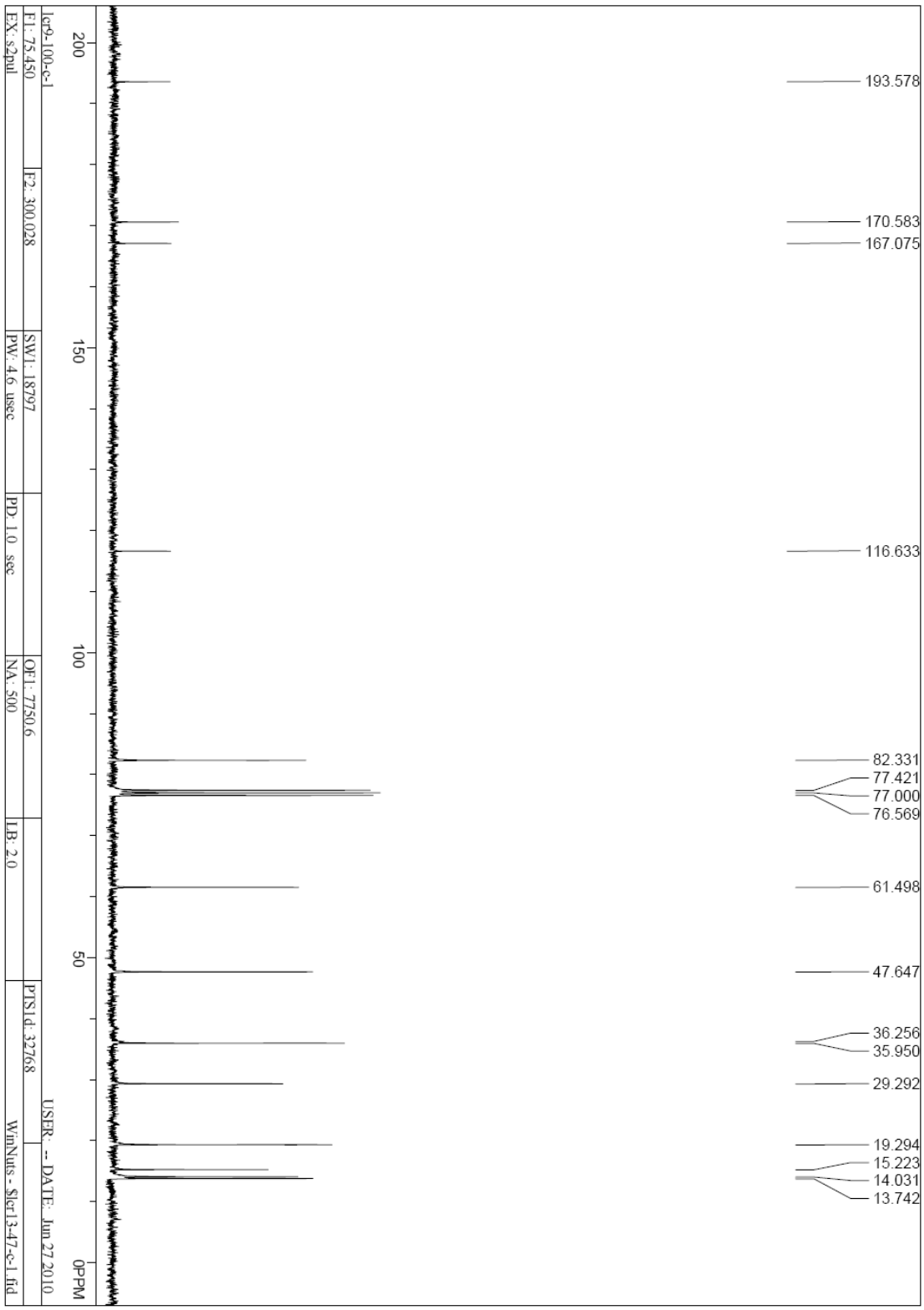


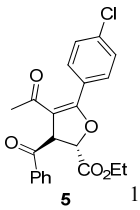
3j ^{1}H NMR (300 M Hz in CD_3Cl)



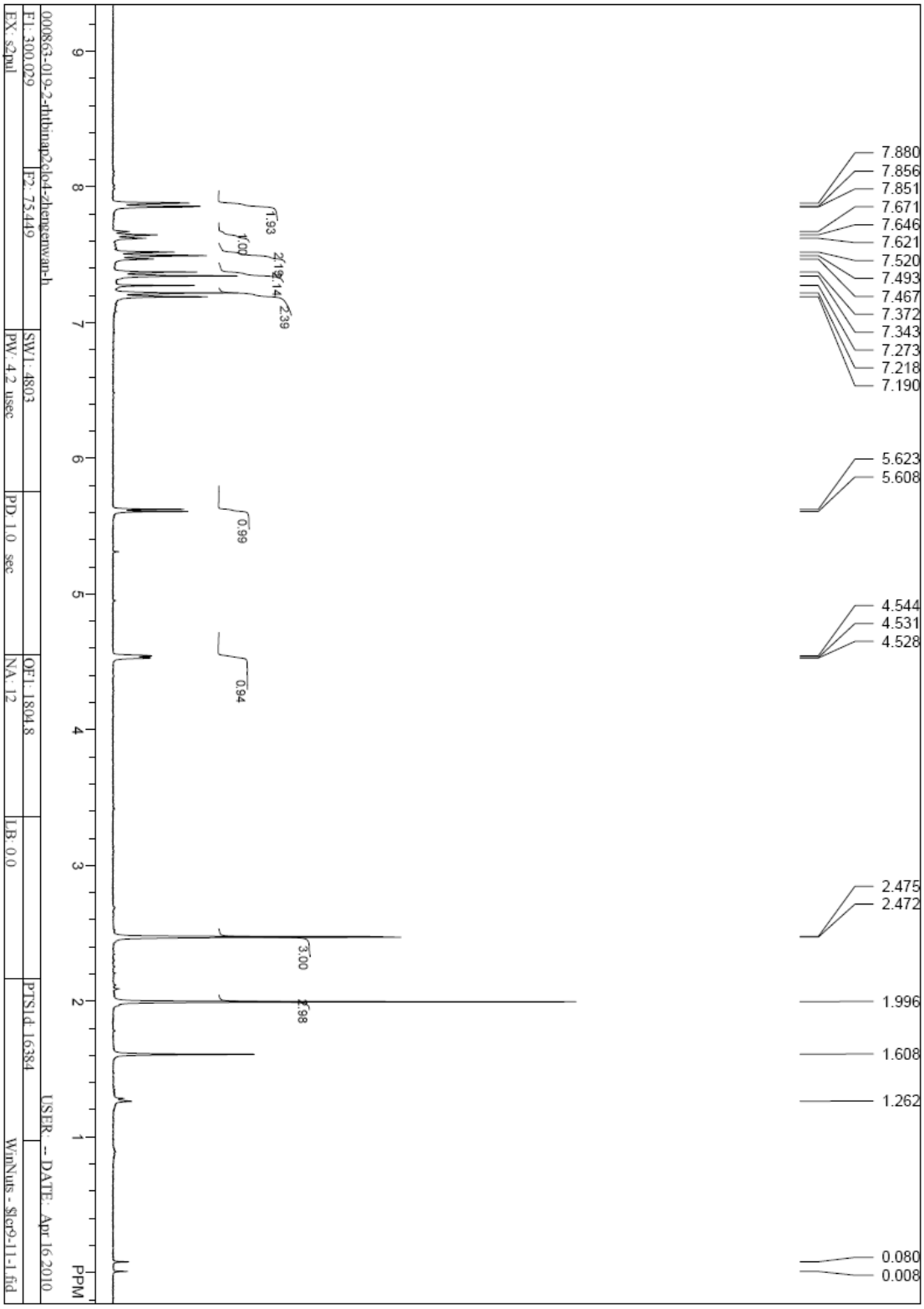


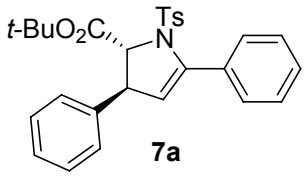
3j ^{13}C NMR (300 M Hz in CD_3Cl)





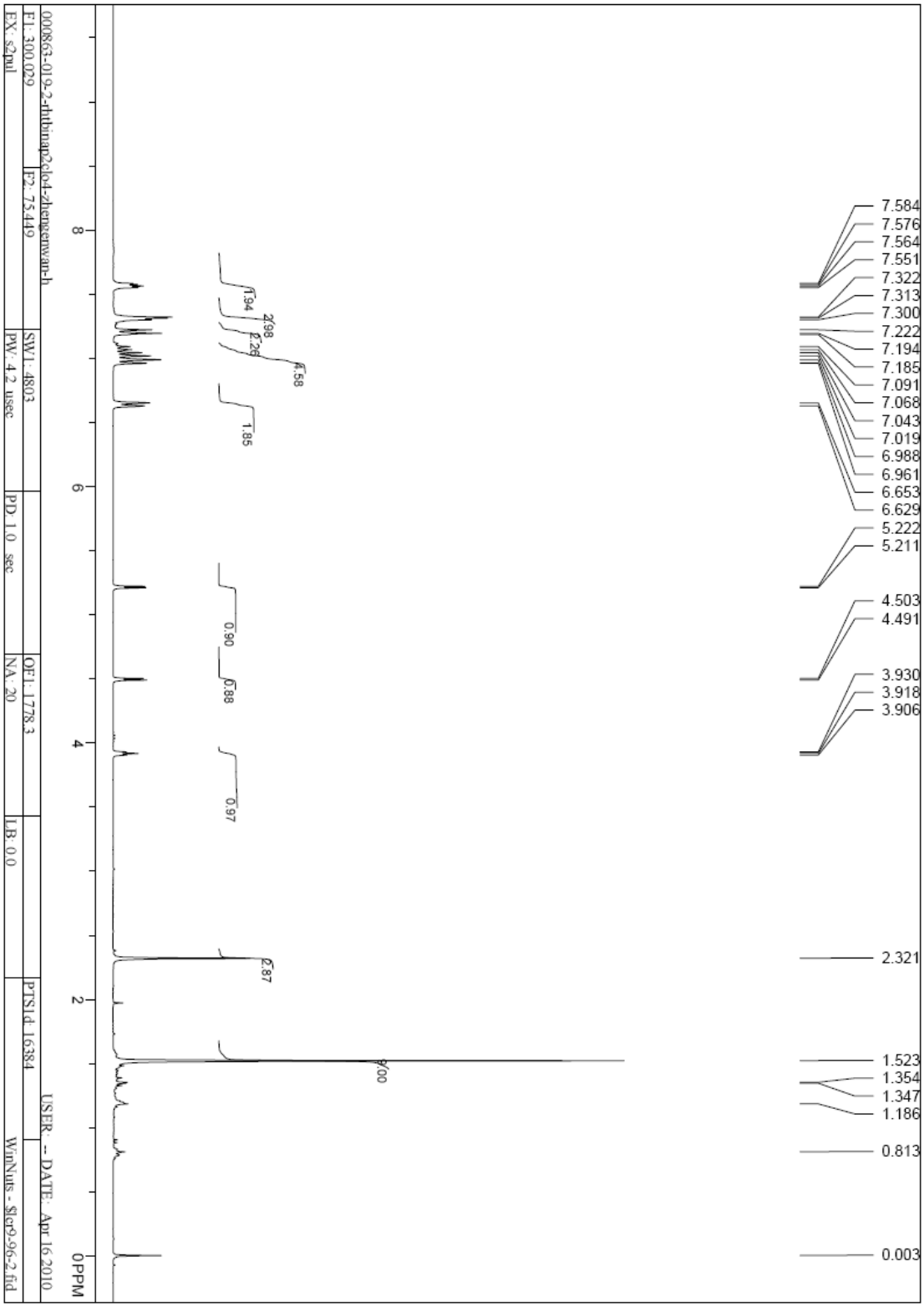
5 ^{1}H NMR (300 M Hz in CD_3Cl)

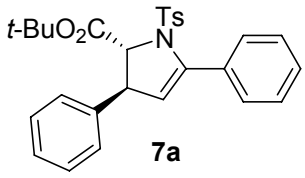




7a

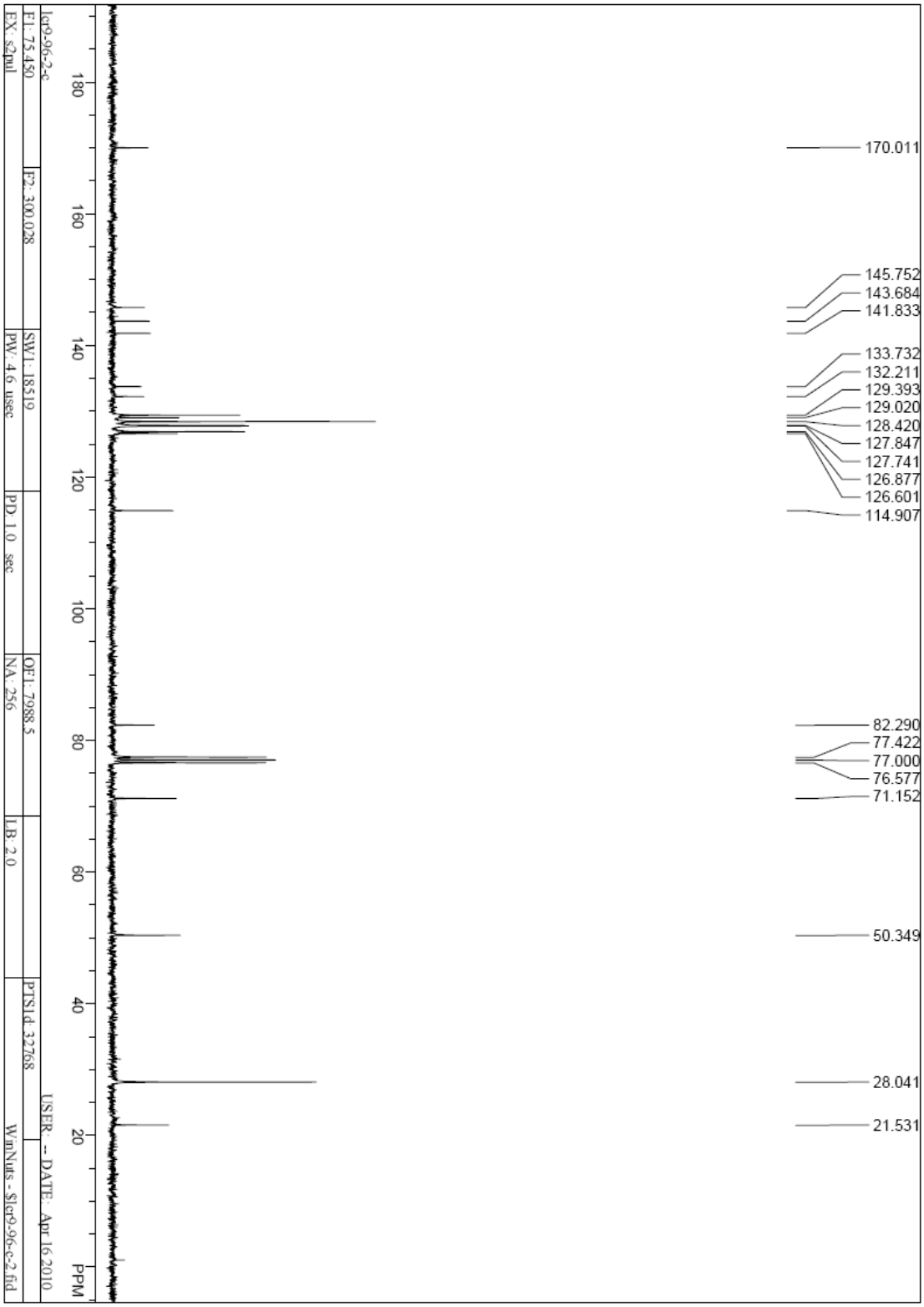
^{1}H NMR (300 M Hz in CD_3Cl)

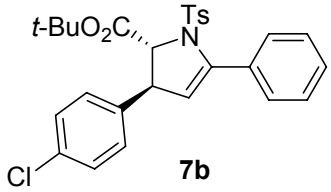




7a

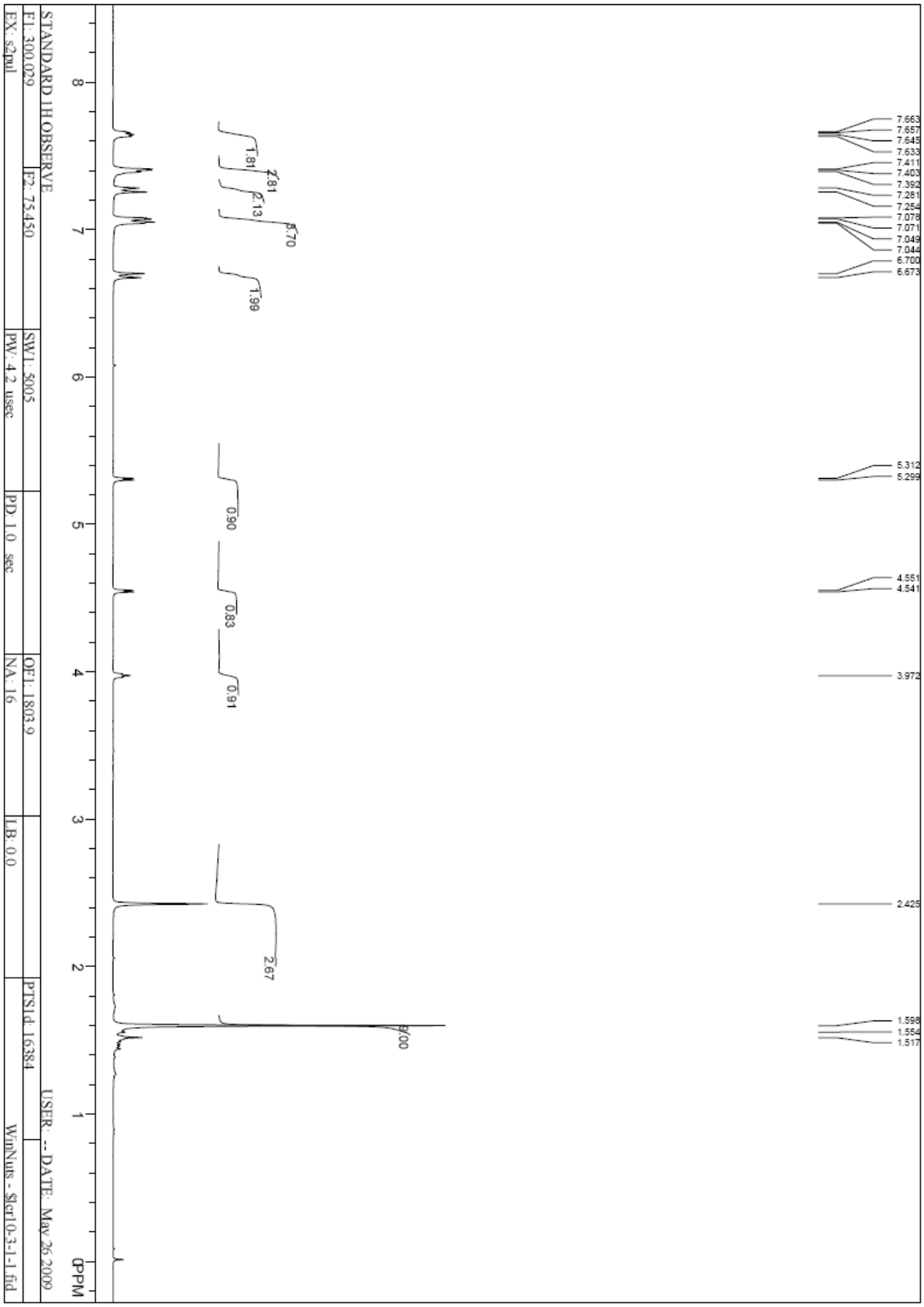
^{13}C NMR (300 M Hz in CD_3Cl)

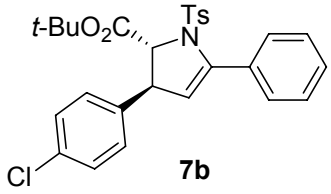




7b

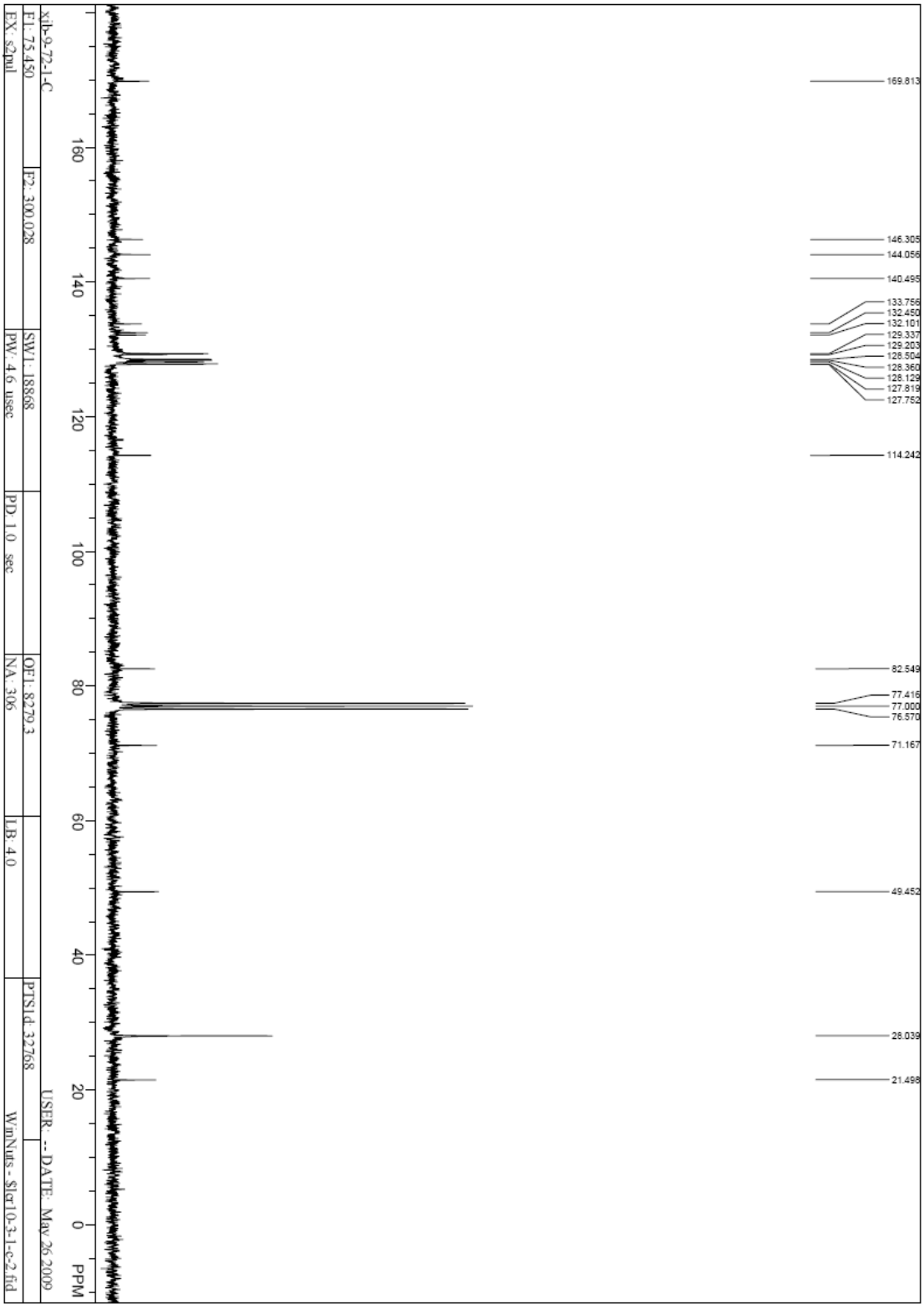
^{1}H NMR (300 M Hz in CD_3Cl)

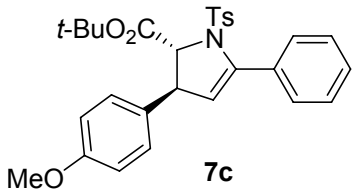




7b

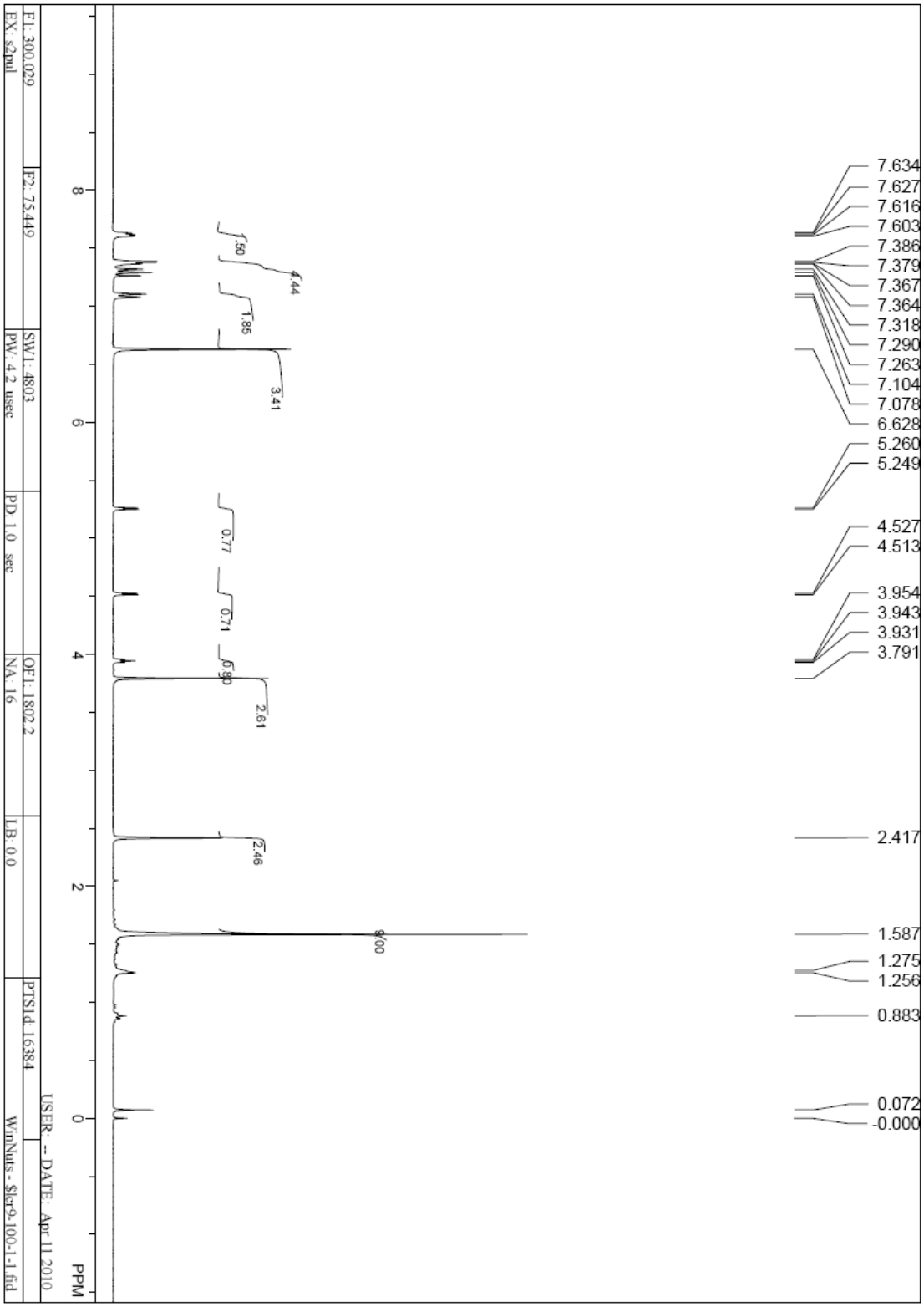
^{13}C NMR (300 M Hz in CD_3Cl)

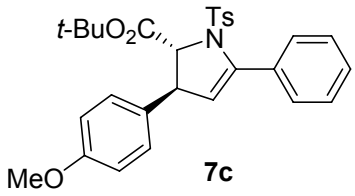




7c

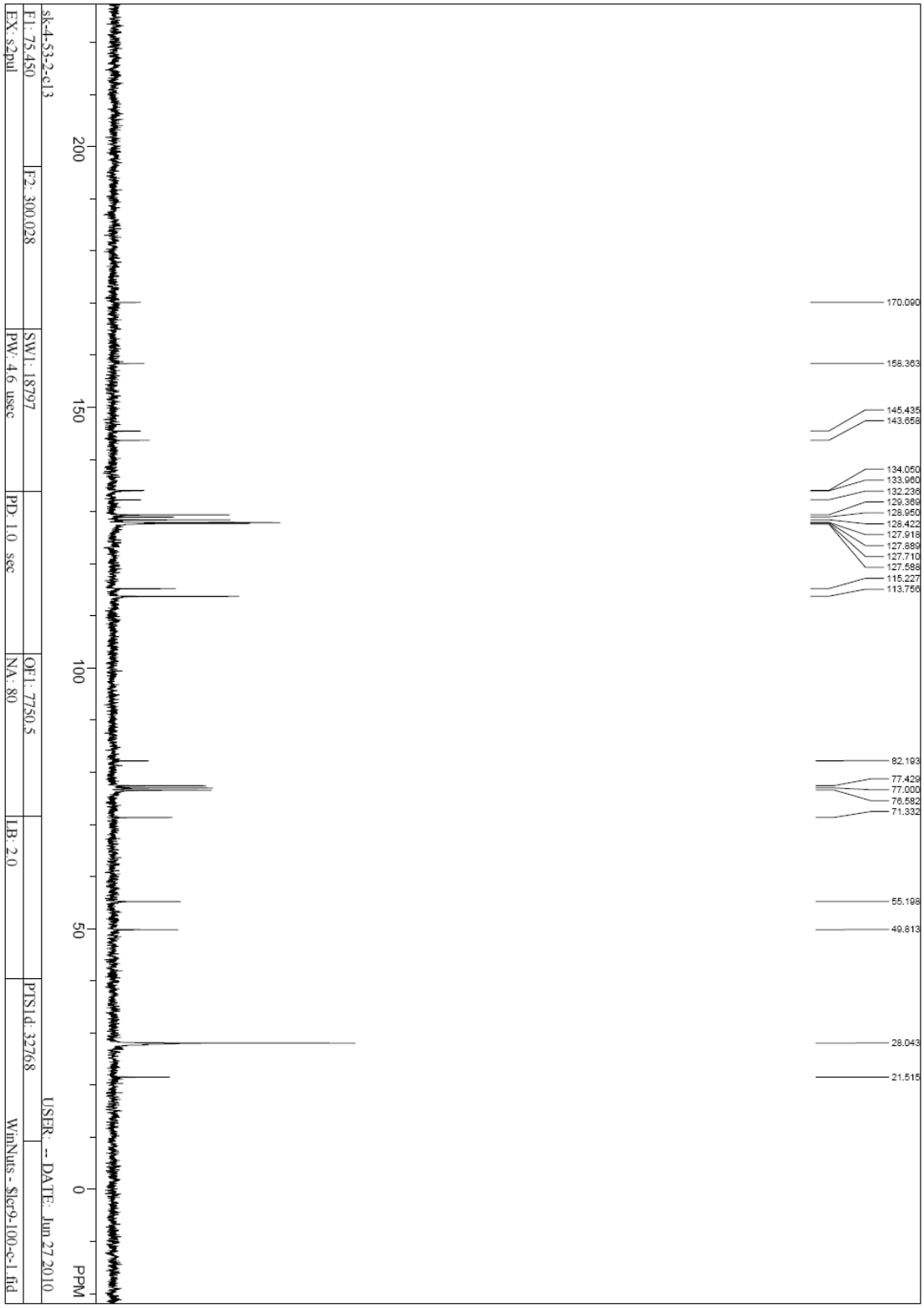
^{1}H NMR (300 M Hz in CD_3Cl)

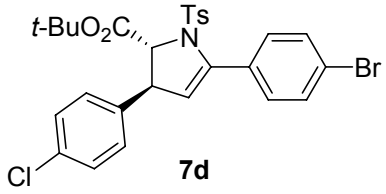




7c

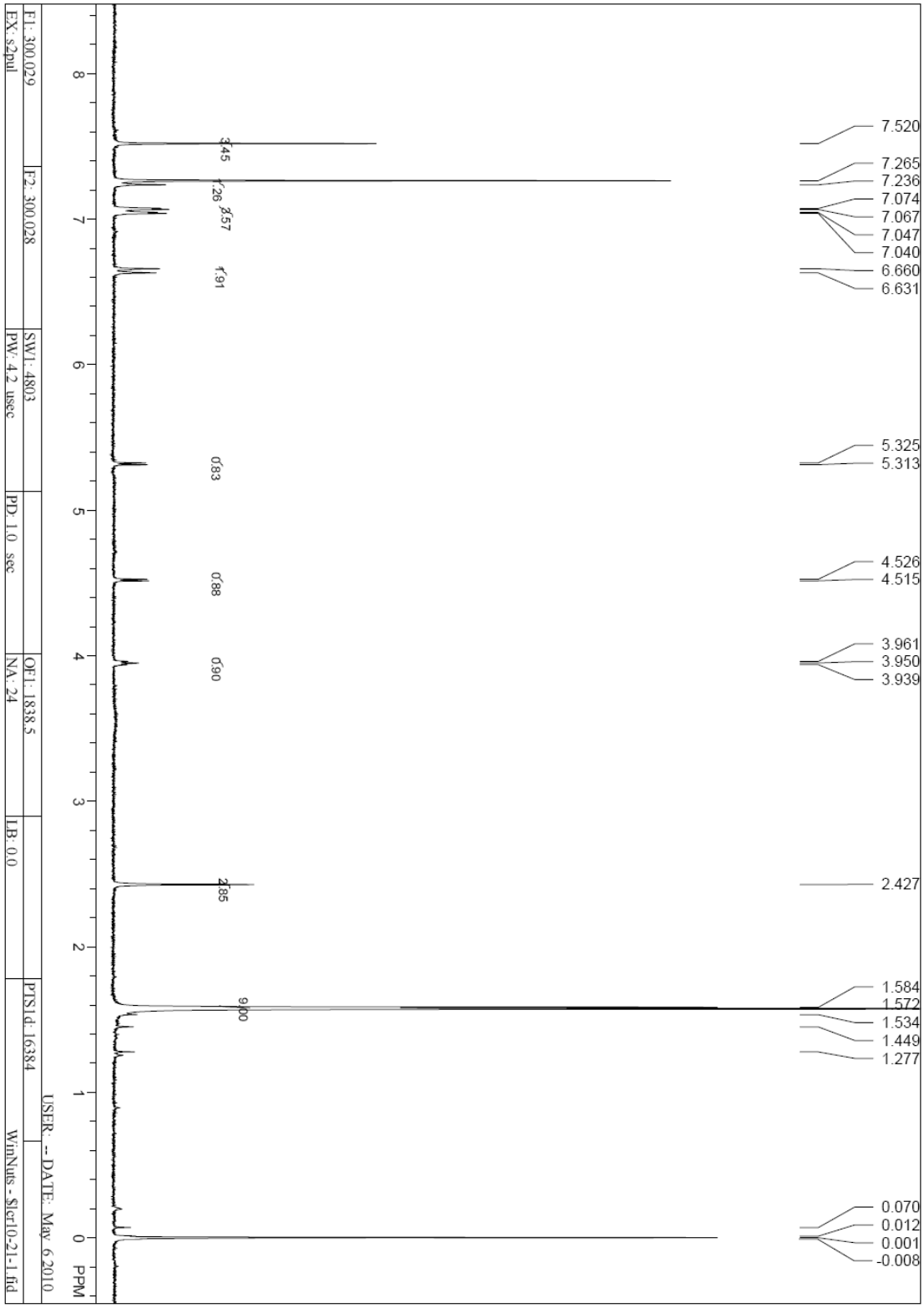
^{13}C NMR (400 M Hz in CD_3Cl)

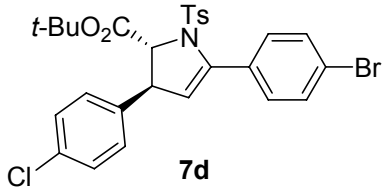




7d

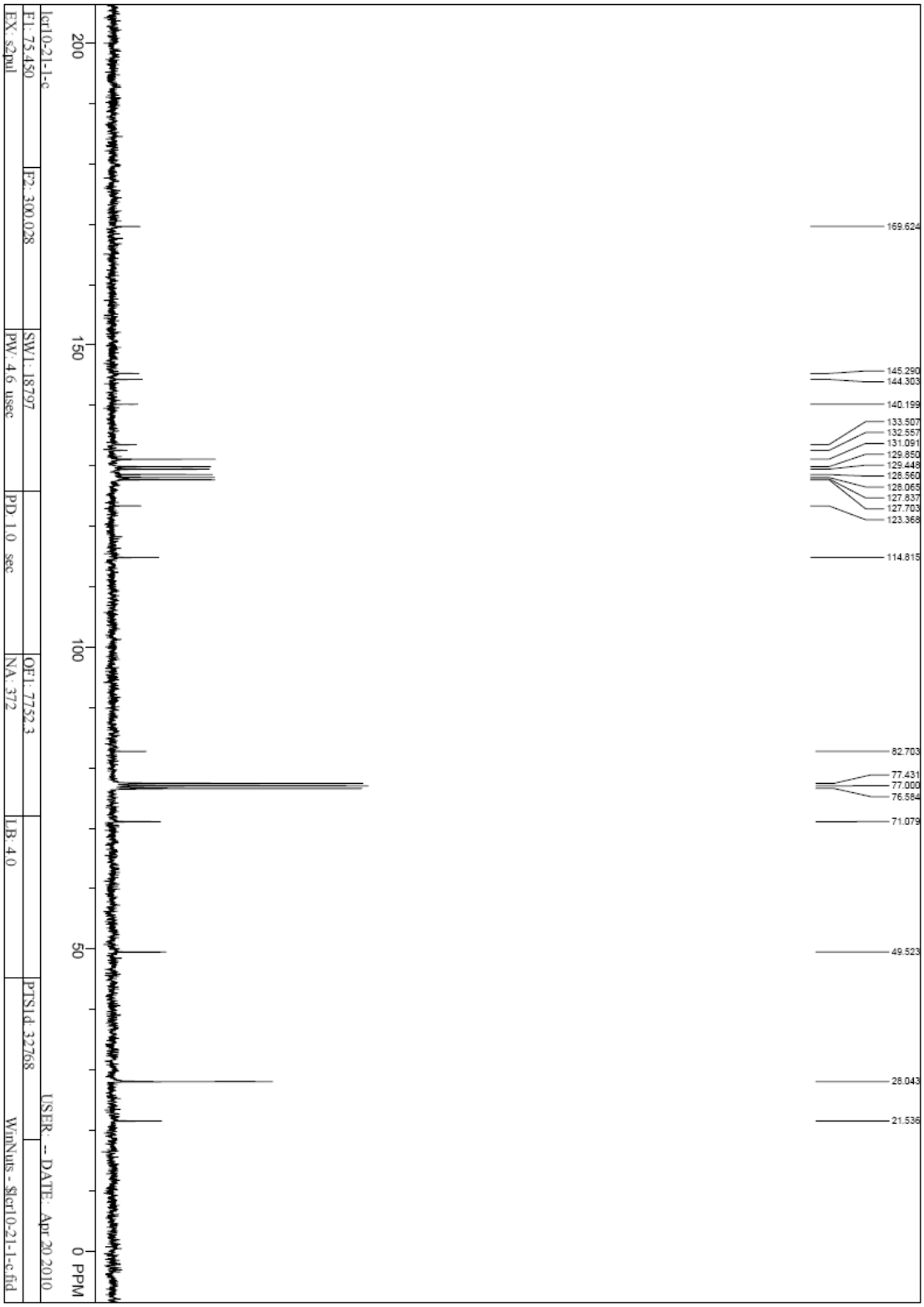
^{1}H NMR (300 M Hz in CD_3Cl)

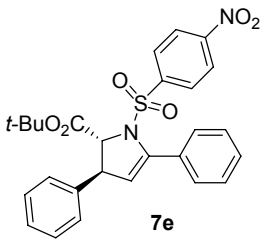




7d

^{13}C NMR (300 M Hz in CD_3Cl)





7e

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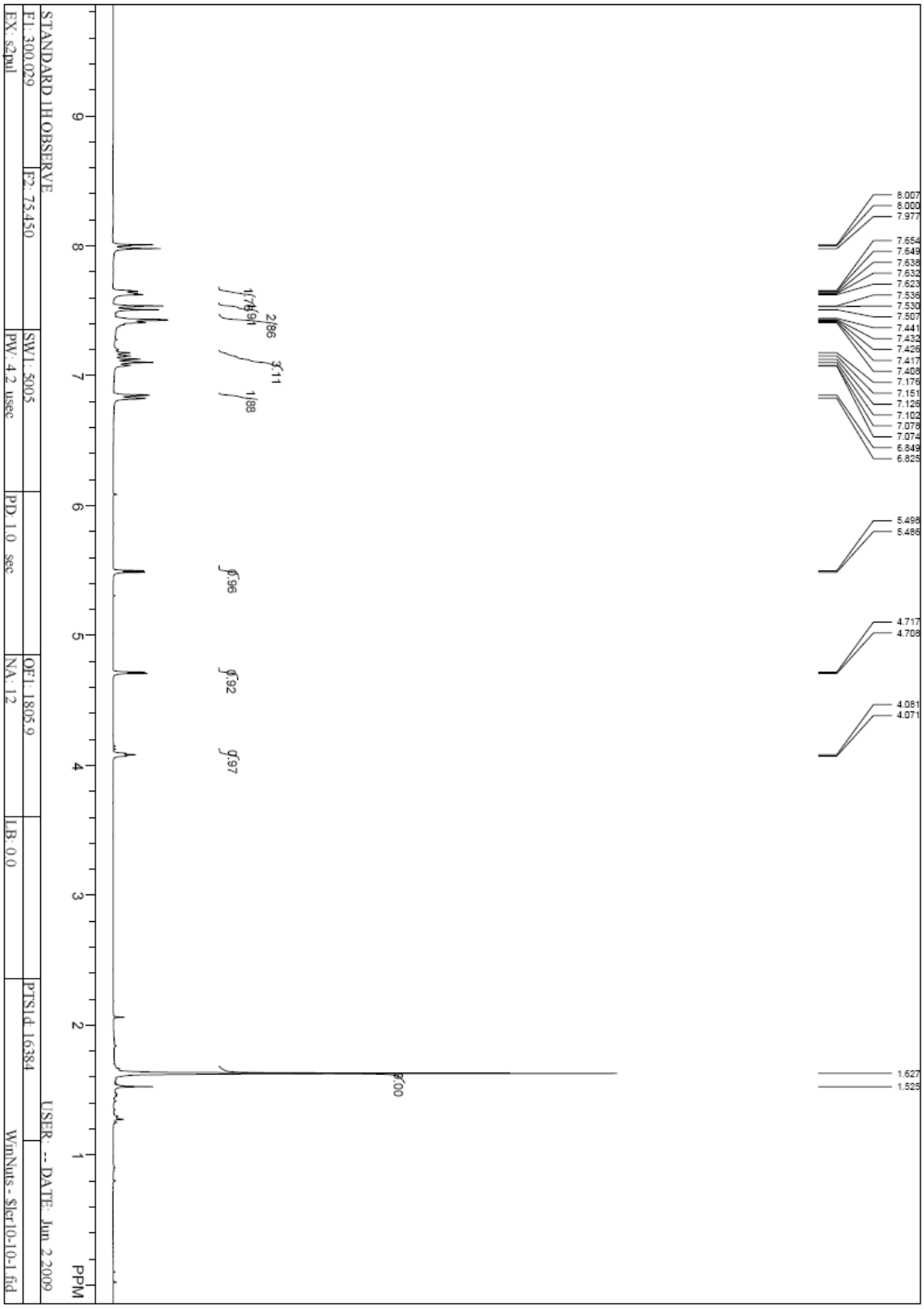

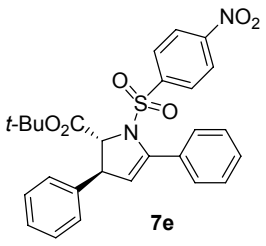

7e

^{13}C NMR (300 M Hz in CD_3Cl)