Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Stereospecific Synthesis of Highly Functionalized Benzo[3.1.0] Bicycloalkanes via Multistep Casecade Reaction

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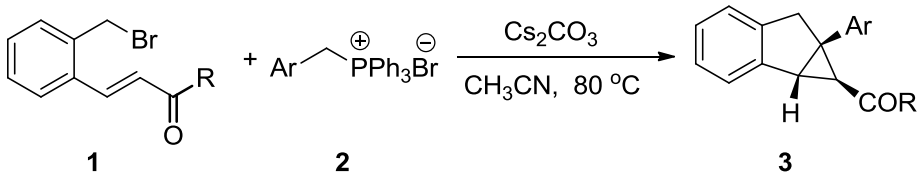
Supporting Information

1. General Information

All reactions were carried out under N_2 unless otherwise noted. All solvents were purified according to standard methods unless otherwise noted.

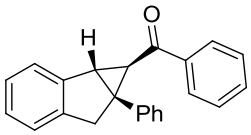
^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz or VARIAN Mercury 400 MHz spectrometer in chloroform-d. All signals are reported in ppm with the internal TMS signal at 0.0 ppm or chloroform signal at 7.26 ppm as a standard. The data are reported as (s = singlet, d = doublet, t = triplet, q = quadruplet, m = multiplet or unresolved, coupling constant(s) in Hz, integration). ^{13}C NMR spectra were recorded on a VARIAN Mercury 75.5 MHz or 100 MHz spectrometer in chloroform-d. All signals are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. IR spectra were recorded on a Perkin–Elmer 983, Digital FT–IR spectrometer or Bruker–Tensor 27; frequencies are given in reciprocal centimeters (cm^{-1}) and only selected absorbance is reported; Mass spectra were determined on an Agilent 5973N MSD (EI) and Shimadzu LCMS-2010EV (ESI) mass spectrometer or Agilent G6100 LC/MSD (ESI) single Quand mass spectrometer. High resolution mass spectra were recorded on Waters Micromass GCT Premier (EI) and Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS (ESI) mass spectrometers.

2. General Procedure for the Tandem Reactions



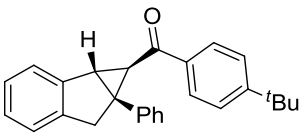
1 **2** **3**

A mixture of substrate **1** (0.3 mmol), phosphonium salt **2** (0.6 mmol), Cs_2CO_3 (586.4 mg, 1.8 mmol) and CH_3CN (4 mL) was stirred for 15 min under a nitrogen atmosphere at room temperature, then warmed to 80 ^oC. After the completion of the reaction (monitored by TLC), the resulting mixture was cooled to room temperature, filtered rapidly through a funnel with a thin layer of silica gel and eluted with DCM. The filtrate was concentrated and the residue was purified by chromatography on silica gel to afford the desired product **3**.



3a

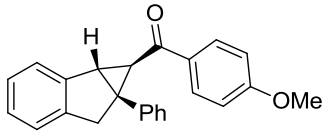
3a, white solid, 82% yield; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.87-7.85 (m, 2H), 7.52-7.48 (m, 1H), 7.44-7.38 (m, 3H), 7.29-7.15 (m, 8H), 3.87 (d, J = 2.4 Hz, 1H), 3.79 (ABd, J_{AB} = 17.6 Hz, 1H), 3.48 (ABd, J_{BA} = 18.0 Hz, 1H), 2.70 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.5, 144.6, 141.4, 138.2, 132.6, 128.9, 128.4, 128.1, 127.9, 126.9, 126.6, 126.3, 125.1, 123.6, 46.5, 44.5, 43.7, 37.9; IR ν/ cm^{-1} 3026 (m), 2970 (m), 1739 (m), 1218 (m), 760 (m); HRMS (positive ESI) calcd for $C_{23}H_{19}O^+$ ($[M+H]^+$): 311.1430; Found: 311.1427.



3b

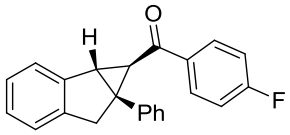
3b, white solid, 78% yield; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.80 (d, J = 8.4 Hz, 2H), 7.42-7.40 (m, 3H), 7.30-7.15 (m, 8H), 3.85 (d, J = 2.8 Hz, 1H), 3.77 (ABd,

J_{AB} = 18.0 Hz, 1H), 3.47 (ABd, J_{BA} = 18.0 Hz, 1H), 2.69 (d, J = 3.2 Hz, 1H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.1, 156.2, 144.7, 141.5, 138.3, 135.7, 129.0, 128.1, 127.8, 126.8, 126.6, 126.2, 125.3, 125.0, 123.6, 46.3, 44.6, 43.7, 38.0, 35.0, 31.0; IR ν/ cm^{-1} 3025 (m), 2968 (m), 1739 (m), 1357 (m), 1220 (m), 756 (m); HRMS (positive ESI) calcd for $C_{27}H_{27}O^+$ ($[M+H]^+$): 367.2056; Found: 367.2074.



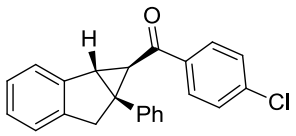
3c

3c, white solid, 85% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.87-7.84 (m, 2H), 7.43-7.41 (m, 1H), 7.27-7.13 (m, 8H), 6.90-6.86 (m, 2H), 3.85-3.82 (m, 4H), 3.77 (ABd, J_{AB} = 17.6 Hz, 1H), 3.46 (ABd, J_{BA} = 18.0 Hz, 1H), 2.65 (d, J = 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 163.1, 144.8, 141.5, 138.4, 131.3, 130.2, 128.9, 128.1, 126.8, 126.6, 126.2. 125.0, 123.6, 113.5, 55.3, 45.8, 44.5, 43.4, 37.4; IR ν/ cm^{-1} 3027 (m), 2897 (m), 1739 (m), 1361 (m), 1225 (m), 750 (m); HRMS (positive ESI) calcd for $C_{24}H_{21}O_2^+$ ($[M+H]^+$): 341.1536; Found: 341.1535.



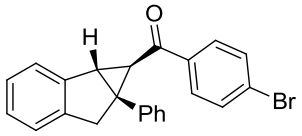
3d

3**d**, white solid, 82% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.90-7.85 (m, 2H), 7.43-7.41 (m, 1H), 7.27-7.14 (m, 8H), 7.08-7.03 (m, 2H), 3.86 (d, J = 3.2 Hz, 1H), 3.78 (ABd, J_{AB} = 17.6 Hz, 1H), 3.47 (ABd, J_{BA} = 17.6 Hz, 1H), 2.64 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 165.4 (d, $J_{C\text{-}F}$ = 252.8 Hz), 144.5, 141.4, 138.1, 134.6 (d, $J_{C\text{-}F}$ = 3.1 Hz), 130.5 (d, $J_{C\text{-}F}$ = 9.4 Hz), 128.9, 128.2, 127.0, 126.7, 126.4, 125.1, 123.6, 115.5 (d, $J_{C\text{-}F}$ = 21.9 Hz), 46.5, 44.5, 43.6, 37.9; IR ν/ cm^{-1} 3026 (m), 1739 (m), 1352 (m), 1219 (m), 762 (m); HRMS (positive ESI) calcd for $C_{23}H_{18}FO^+$ ($[M+H]^+$): 329.1336; Found: 329.1350.



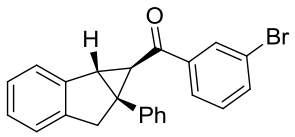
3e

3e, white solid, 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.79 (d, *J* = 8.4 Hz, 2H), 7.44-7.42 (m, 1H), 7.37 (d, *J* = 8.8 Hz, 2H), 7.26-7.15 (m, 8H), 3.87 (d, *J* = 2.8 Hz, 1H), 3.78 (ABd, J_{AB} = 17.6 Hz, 1H), 3.48 (ABd, J_{BA} = 18.0 Hz, 1H), 2.64 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.2, 144.4, 141.3, 139.0, 138.0, 136.4, 129.3, 128.9, 128.7, 128.2, 127.0, 126.7, 126.4, 125.1, 123.6, 46.7, 44.4, 43.6, 37.9; IR ν/ cm^{-1} 3025 (m), 2970 (m), 1739 (m), 1366 (m), 1217 (m), 760 (m); HRMS (positive ESI) calcd for $C_{23}H_{18}ClO^+$ ($[M+H]^+$): 345.1041; Found: 345.1039.



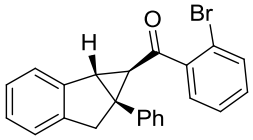
3f

3f, white solid, 65% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.70 (d, *J* = 8.8 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.43-7.41 (m, 1H), 7.26-7.14 (m, 8H), 3.86 (d, *J* = 2.4 Hz, 1H), 3.78 (ABd, J_{AB} = 18.0 Hz, 1H), 3.47 (ABd, J_{BA} = 17.6 Hz, 1H), 2.62 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.4, 144.4, 141.4, 137.9, 136.8, 131.7, 129.5, 128.9, 128.2, 127.8, 127.0, 126.7, 126.4, 125.1, 123.6, 46.7, 44.5, 43.6, 38.0; IR ν/ cm^{-1} 3025 (m), 2970 (m), 1739 (m), 1215 (m), 760 (m); HRMS (positive ESI) calcd for $C_{23}H_{18}BrO^+$ ($[M+H]^+$): 389.0536; Found: 389.0531.



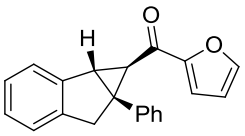
3g

3g, white solid, 64% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.96 (s, 1H), 7.77 (d, *J* =7.6 Hz, 1H), 7.62 (dd, *J* = 8.0 and 1.2 Hz, 1H), 7.44-7.42 (m, 1H), 7.30-7.16 (m, 9H), 3.87 (d, *J* = 2.8 Hz, 1H), 3.80 (ABd, J_{AB} = 17.6 Hz, 1H), 3.48 (ABd, J_{BA} = 18.0 Hz, 1H), 2.64 (d, *J* = 2.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.1, 144.3, 141.4, 139.9, 137.9, 135.5, 131.0, 130.0, 128.9, 128.2, 127.1, 126.7, 126.5(0), 126.4(8), 125.1, 123.7, 122.8, 47.0, 44.5, 43.7, 38.4; IR ν/ cm^{-1} 3022 (m), 1739 (m), 1353 (m), 1206 (m), 756 (m); HRMS (positive ESI) calcd for $C_{23}H_{18}BrO^+$ ($[M+H]^+$): 389.0536; Found: 389.0529.



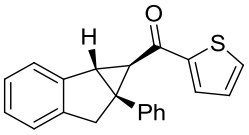
3h

3h, brown oil, 73% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.54 (d, *J* = 8.0 Hz, 1H), 7.46-7.42 (m, 3H), 7.34-7.27 (m, 4H), 7.25-7.15 (m, 5H), 3.84 (d, *J* = 3.2 Hz, 1H), 3.79 (ABd, J_{AB} = 17.6 Hz, 1H), 3.49 (ABd, J_{BA} = 17.6 Hz, 1H), 2.60 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.8, 144.2, 141.4(4), 141.3(9), 137.9, 133.6, 131.8, 129.8, 129.3, 128.1, 127.3, 127.0, 126.6, 126.4, 125.1, 123.6, 119.7, 47.9, 47.6, 44.4, 40.4; IR ν/ cm^{-1} 3025 (m), 1711 (m), 1352 (m), 1217 (m), 754 (m); HRMS (positive ESI) calcd for $C_{23}H_{18}BrO^{+}$ ($[M+H]^{+}$): 389.0536; Found: 389.0533.



3i

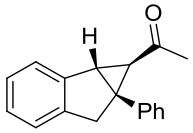
3i, brown solid, 70% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.52-7.51 (m, 1H), 7.42-7.40 (m, 1H), 7.29-7.17 (m, 8H), 7.07 (dd, *J* = 3.6 and 0.4 Hz, 1H), 6.48 (dd, *J* = 3.6 and 1.6 Hz, 1H), 3.80 (dd, *J* = 3.2 and 0.8 Hz, 1H), 3.74 (ABd, J_{AB} = 18.0 Hz, 1H), 3.44 (ABd, J_{BA} = 18.0 Hz, 1H), 2.65 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.2, 153.7, 146.0, 144.5, 141.5, 138.1, 129.1, 128.1, 126.9, 126.6, 126.4, 125.0, 123.6, 116.2, 112.1, 46.3, 44.7, 42.8, 38.0; IR ν/ cm^{-1} 3026 (m), 2970 (m), 1739 (m), 1365 (m), 1217 (m), 776 (m); HRMS (positive ESI) calcd for $C_{21}H_{17}O_2^{+}$ ($[M+H]^{+}$): 301.1223; Found: 301.1220.



3j

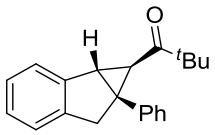
3j, yellow solid, 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.67 (d, *J* = 3.6, 1H), 7.53 (d, *J* = 4.8, 1H), 7.43-7.41 (m, 1H), 7.28-7.16 (m, 8H), 7.08-7.06 (m, 1H), 3.81 (d, *J* = 2.8 Hz, 1H), 3.72 (ABd, J_{AB} = 18.0 Hz, 1H), 3.44 (ABd, J_{BA} = 18.0 Hz, 1H), 2.58 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.8, 145.4, 144.4, 141.4, 138.1, 133.1, 131.3, 128.9, 128.1, 127.9, 126.9, 126.6, 126.4, 125.0,

123.6, 46.1, 44.7, 43.8, 38.1; IR ν/ cm^{-1} 3021 (m), 2970 (m), 1739 (m), 1354 (m), 1228 (m), 727 (m); HRMS (positive ESI) calcd for $C_{21}H_{17}OS^+$ ($[M+H]^+$): 317.0995; Found: 317.0993.



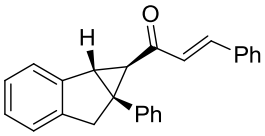
3k

3k, brown solid, 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.38-7.36 (m, 1H), 7.32-7.27 (m, 4H), 7.25-7.13 (m, 4H), 3.63 (ABd, J_{AB} = 18.0 Hz, 1H), 3.59 (dd, J = 2.8 and 1.2 Hz, 1H), 3.36 (ABd, J_{BA} = 18.0 Hz, 1H), 2.04 (s, 3H), 2.02 (d, J = 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.5, 144.2, 141.3, 138.3, 128.8, 128.2, 127.0, 126.5, 126.2, 125.0, 123.4, 46.8, 45.2, 44.5, 37.6, 31.3; IR ν/ cm^{-1} 3024 (m), 2908 (m), 1689 (m), 1356 (m), 1165 (m), 751 (m); HRMS (positive ESI) calcd for $C_{18}H_{17}O^+$ ($[M+H]^+$): 249.1274; Found: 249.1270.



3l

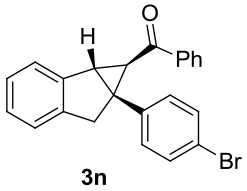
3l, white solid, 83% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.39-7.37 (m, 1H), 7.28-7.27 (m, 4H), 7.22-7.14 (m, 4H), 3.68 (ABd, J_{AB} = 18.0 Hz, 1H), 3.64 (d, J = 3.2 Hz, 1H), 3.41 (ABd, J_{BA} = 17.6 Hz, 1H), 2.24 (d, J = 3.6 Hz, 1H), 1.01 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.4, 144.7, 141.5, 138.2, 128.8, 127.9, 126.7, 126.5, 126.1, 125.0, 123.5, 45.3, 43.9, 43.7, 43.5, 37.3, 26.0; IR ν/ cm^{-1} 2970 (m), 1739 (m), 1366 (m), 1090 (m), 765 (m); HRMS (positive ESI) calcd for $C_{21}H_{23}O^+$ ($[M+H]^+$): 291.1743; Found: 291.1739.



3m

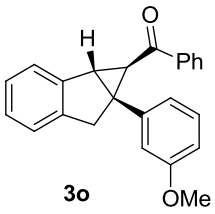
3m, yellow solid, 57% yield; ^{1}H NMR (300 MHz, $CDCl_3$, TMS) δ 7.49-7.40 (m, 4H), 7.36-7.16 (m, 11H), 6.73 (d, J = 16.2 Hz, 1H), 3.74-3.68 (m, 2H), 3.43 (d, J = 17.7 Hz, 1H), 2.33 (d, J = 2.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.0, 144.7,

142.0, 141.5, 138.4, 134.5, 130.3, 129.1, 128.8, 128.2(0), 128.1(7), 127.1, 126.9, 126.6, 126.3, 125.0, 123.6, 46.2, 45.4, 44.8, 38.3; IR ν/ cm^{-1} 3026 (m), 2970 (m), 1738 (m), 1357 (m), 1175 (m), 767 (m); HRMS (positive ESI) calcd for $C_{25}H_{21}O^+$ ($[M+H]^+$): 337.1587; Found: 337.1583.



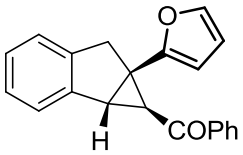
3n

3n, white solid, 82% yield; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.86-7.84 (m, 2H), 7.54-7.50 (m, 1H), 7.43-7.40 (m, 3H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.26-7.14 (m, 5H), 3.83 (d, *J* = 3.2 Hz, 1H), 3.77 (ABd, J_{AB} = 17.2 Hz, 1H), 3.44 (ABd, J_{BA} = 18.0 Hz, 1H), 2.72 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.3, 144.2, 141.1, 138.0, 137.3, 132.8, 131.3, 130.6, 128.5, 127.9, 126.7, 126.5, 125.1, 123.6, 120.9, 45.7, 44.2, 43.6, 38.1; IR ν/ cm^{-1} 3022 (m), 1739 (m), 1352 (m), 1218 (m), 755 (m); HRMS (positive ESI) calcd for $C_{23}H_{18}BrO^+$ ($[M+H]^+$): 389.0536; Found: 389.0521.



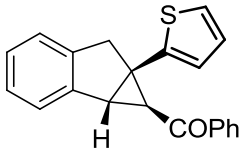
3o

3o, white solid, 90% yield; ^1H NMR (400 MHz, $CDCl_3$, TMS) δ 7.89-7.86 (m, 2H), 7.52-7.49 (m, 1H), 7.41-7.39 (m, 3H), 7.26-7.12 (m, 4H), 6.86 (d, *J* = 7.2 Hz, 1H), 6.82-6.80 (m, 1H), 6.71 (dd, *J* = 8.0 and 2.4 Hz, 1H), 3.85 (d, *J* = 3.2 Hz, 1H), 3.76 (ABd, J_{AB} = 17.6 Hz, 1H), 3.68 (s, 3H), 3.47 (ABd, J_{AB} = 18.0 Hz, 1H), 2.69 (d, *J* = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.4, 159.2, 144.5, 141.4, 139.8, 138.2, 132.6, 129.1, 128.4, 127.9, 126.6, 126.3, 125.1, 123.6, 121.3, 114.6, 112.5, 54.9, 46.4, 44.6, 43.6, 37.9; IR ν/ cm^{-1} 3020 (m), 2912 (m), 1739 (m), 1354 (m), 1217 (m), 754 (m); HRMS (positive ESI) calcd for $C_{24}H_{21}O_2^+$ ($[M+H]^+$): 341.1536; Found: 341.1535.



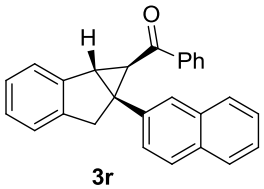
3p

3p, at 20 ^{o}C, brown solid, 75% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.90 (dd, *J* = 8.0 and 1.2 Hz, 2H), 7.54-7.50 (m, 1H), 7.44-7.39 (m, 3H), 7.27-7.18 (m, 4H), 6.23 (dd, *J* = 7.2 and 2.0 Hz, 1H), 6.12 (dd, *J* = 3.2 and 0.8 Hz, 1H), 3.80 (dd, *J* = 3.6 and 0.8 Hz, 1H), 3.72 (ABd, J_{AB} = 17.6 Hz, 1H), 3.57 (ABd, J_{BA} = 17.6 Hz, 1H), 2.65 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 151.7, 143.6, 141.8, 141.1, 137.8, 132.7, 128.4, 128.0, 126.7, 126.5, 125.2, 123.7, 110.1, 107.7, 43.2, 41.9, 38.2, 37.9; IR ν/ cm^{-1} 2905 (m), 1738 (m), 1375 (m), 1217 (m), 727 (m); HRMS (positive ESI) calcd for $C_{21}H_{17}O_2^+$ ([M+H]$^+$): 301.1223; Found: 301.1216.



3q

3q, at 40 ^{o}C, brown solid, 52% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.53-7.49 (m, 1H), 7.43-7.39 (m, 3H), 7.26-7.17 (m, 3H), 7.08-7.06 (m, 1H), 6.85-6.83 (m, 2H), 3.89 (d, *J* = 3.2 Hz, 1H), 3.78 (d, *J* = 17.2 Hz, 1H), 3.57 (d, *J* = 17.6 Hz, 1H), 2.71 (d, *J* = 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 143.9, 141.4, 141.1, 137.9, 132.8, 128.4, 128.0, 126.7, 126.5, 126.3, 125.1, 124.5, 123.6, 44.8, 44.6, 40.4, 39.1; IR ν/ cm^{-1} 2925 (m), 1738 (m), 1381 (m), 1218 (m), 760 (m); HRMS (positive ESI) calcd for $C_{21}H_{17}OS^+$ ([M+H]$^+$): 317.0995; Found: 317.0994.

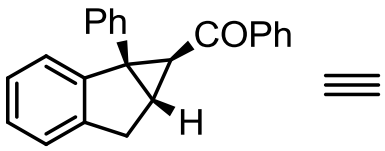


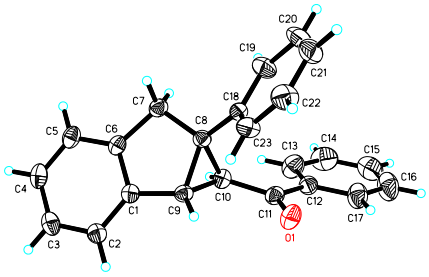
3r

3r, white solid, 81% yield; ^{1}H NMR (400 MHz, $CDCl_3$, TMS) δ 7.86-7.84 (m, 2H), 7.75-7.68 (m, 4H), 7.50-7.45 (m, 2H), 7.42-7.35 (m, 5H), 7.28-7.18 (m, 3H),

3.99 (d, J = 2.4 Hz, 1H), 3.87 (ABd, J_{AB} = 17.6 Hz, 1H), 3.53 (ABd, J_{BA} = 18.0 Hz, 1H), 2.78 (d, J = 3.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.4, 144.6, 141.4, 138.2, 135.7, 133.1, 132.6, 132.4, 128.4, 127.9, 127.8, 127.7(9), 127.7(6), 127.5, 127.0, 126.7, 126.4, 125.9, 125.6, 125.1, 123.7, 46.6, 44.6, 43.8, 38.2; IR ν/ cm^{-1} 3023 (m), 2928 (m), 1738 (m), 1353 (m), 1220 (m), 709 (m); HRMS (positive ESI) calcd for $C_{27}H_{21}O^+$ ($[M+H]^+$): 361.1587; Found: 361.1573.

3. X-Ray structure of 3a (CCDC 962017)





Bond precision: C-C = 0.0025 A Wavelength=0.71073

Cell: a=6.1046(6) b=15.4141(15) c=17.5871(17)

alpha=90 beta=90 gamma=90

Temperature: 293 K

	Calculated	Reported
Volume	1654.9(3)	1654.9(3)
Space group	P 21 21 21	P2(1)2(1)2(
Hall group	P 2ac 2ab	?
Moiety formula	C23 H18 O	?
Sum formula	C23 H18 O	C23 H18 O
Mr	310.37	310.37
Dx,g cm-3	1.246	1.246
Z	4	4
Mu (mm-1)	0.075	0.075
F000	656.0	656.0
F000'	656.27	
h,k,lmax	7,19,21	7,19,21
Nref	1899[3261]	3260
Tmin,Tmax	0.984,0.992	0.380,1.000
Tmin'	0.983	

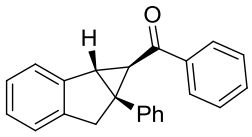
Correction method= EMPIRICAL

Data completeness= 1.72/1.00 Theta(max)= 25.990

R(reflections)= 0.0362(2837) wR2(reflections)=0.0936(3260)

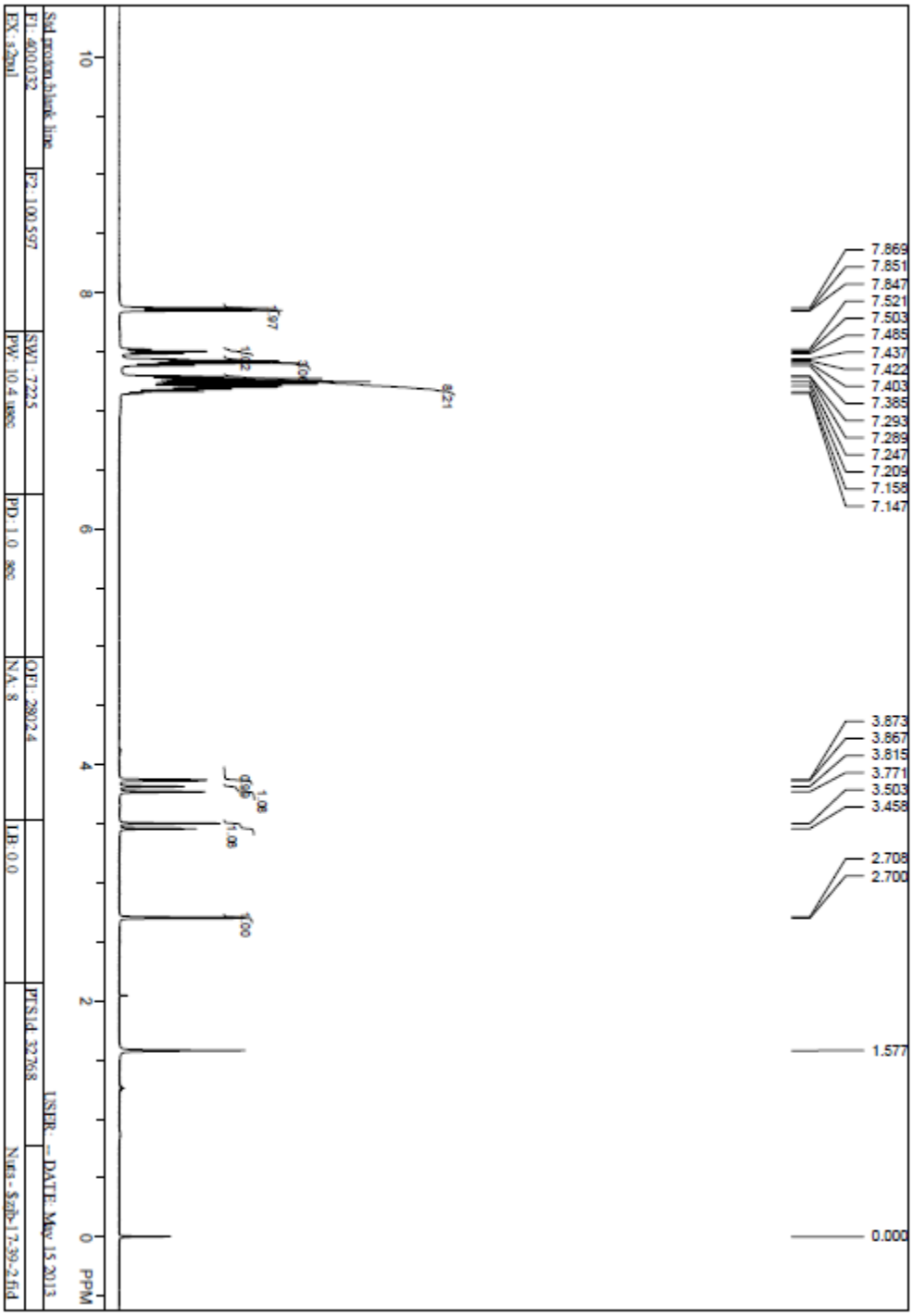
S = 1.050 Npar= 217

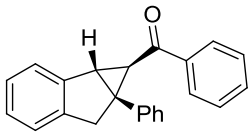
4. NMR Spectra



3a

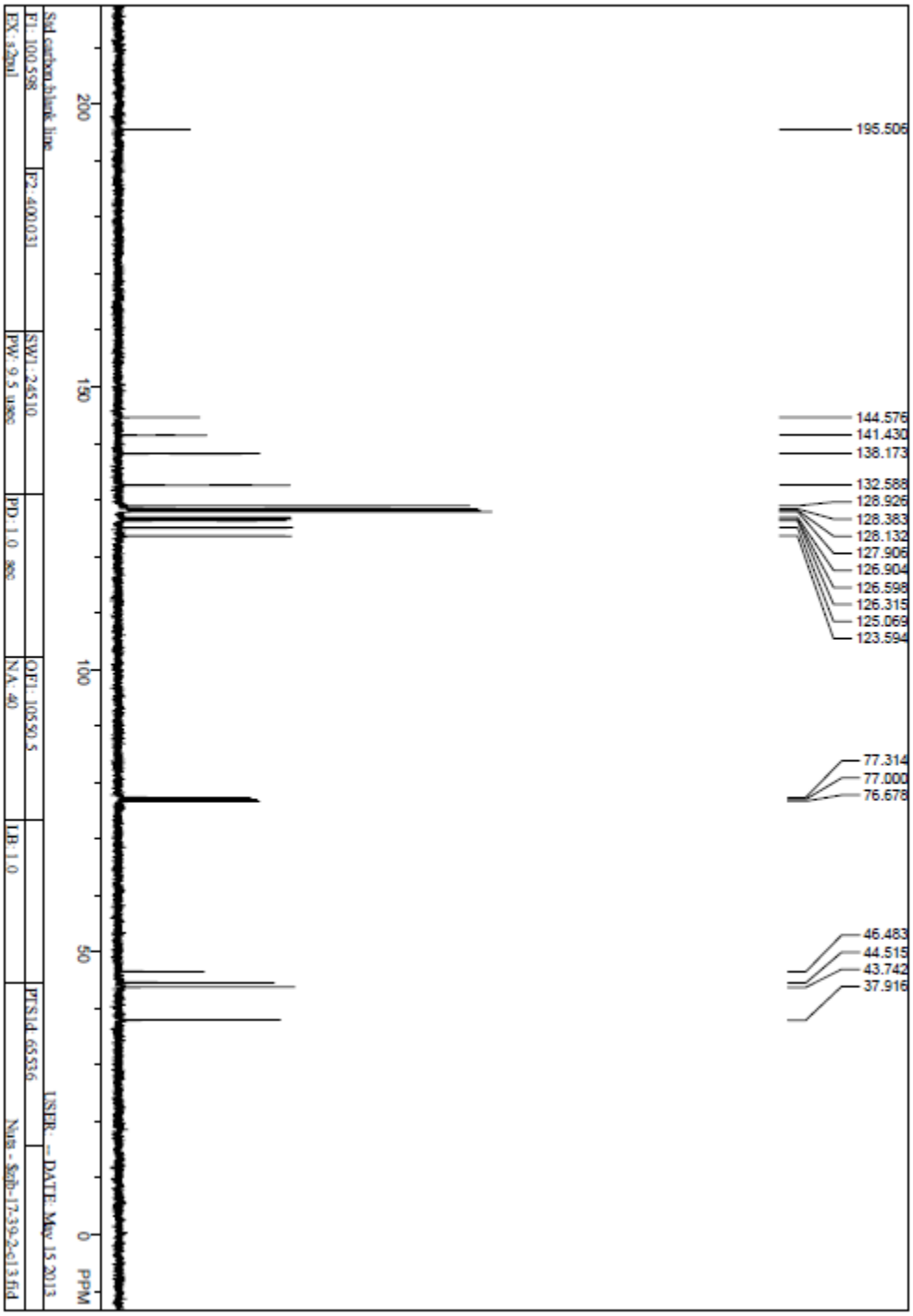
^{1}H NMR (400 M Hz in $CDCl_3$)

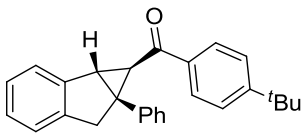




3a

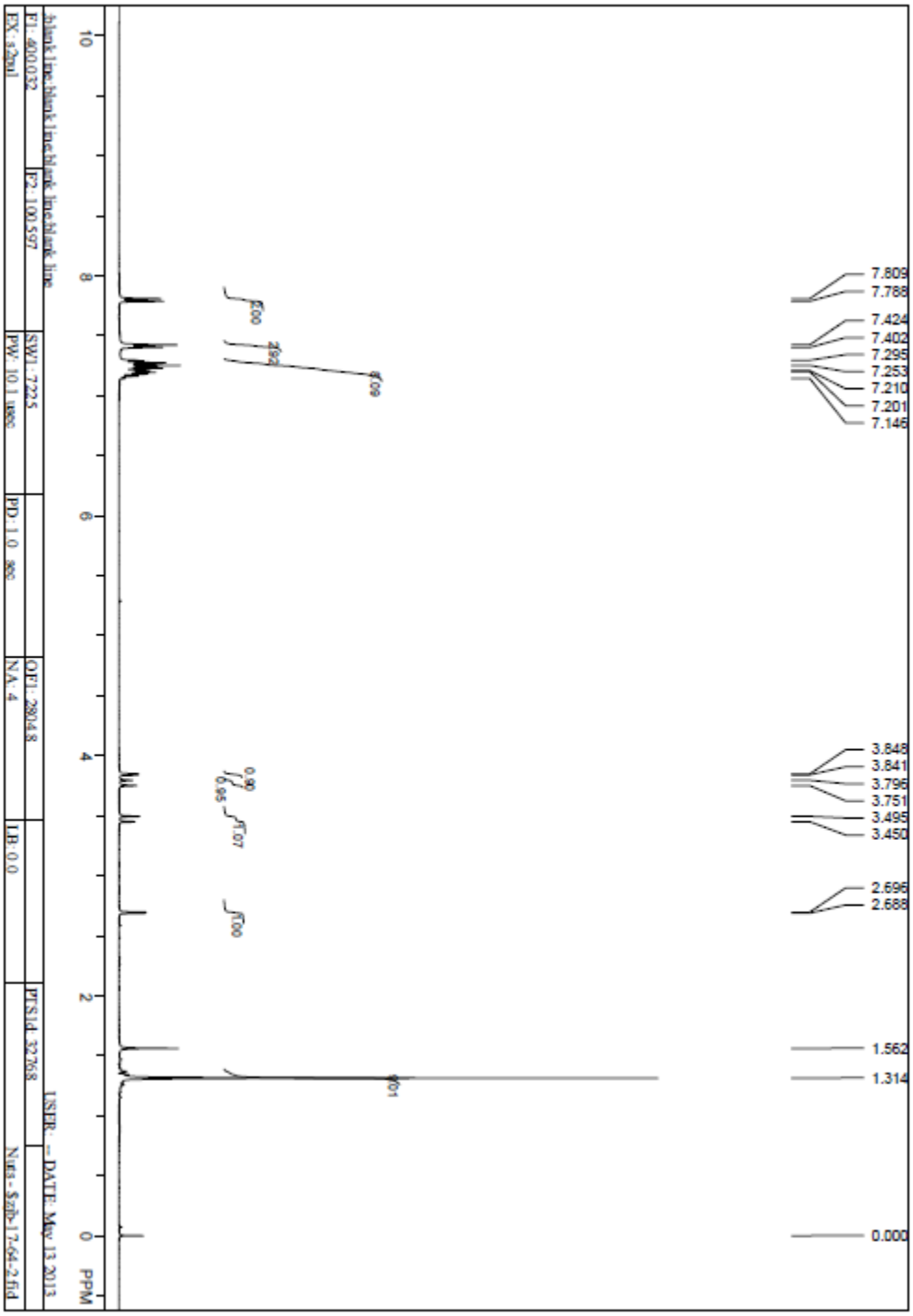
^{13}C NMR (100 M Hz in $CDCl_3$)

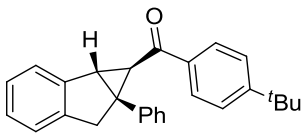




3b

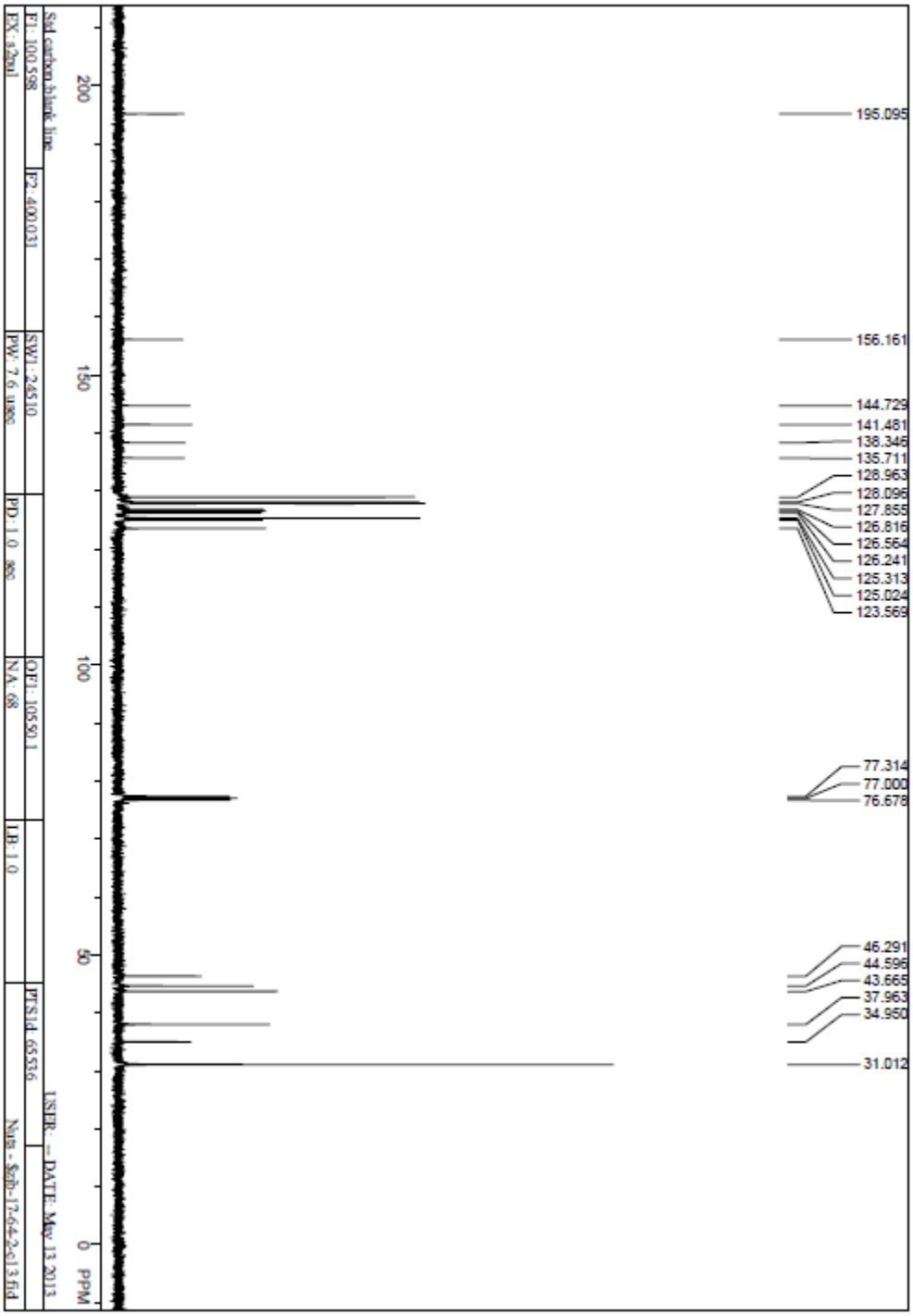
^{1}H NMR (400 M Hz in $CDCl_3$)

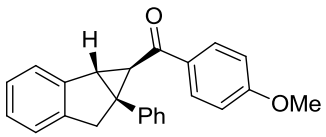




3b

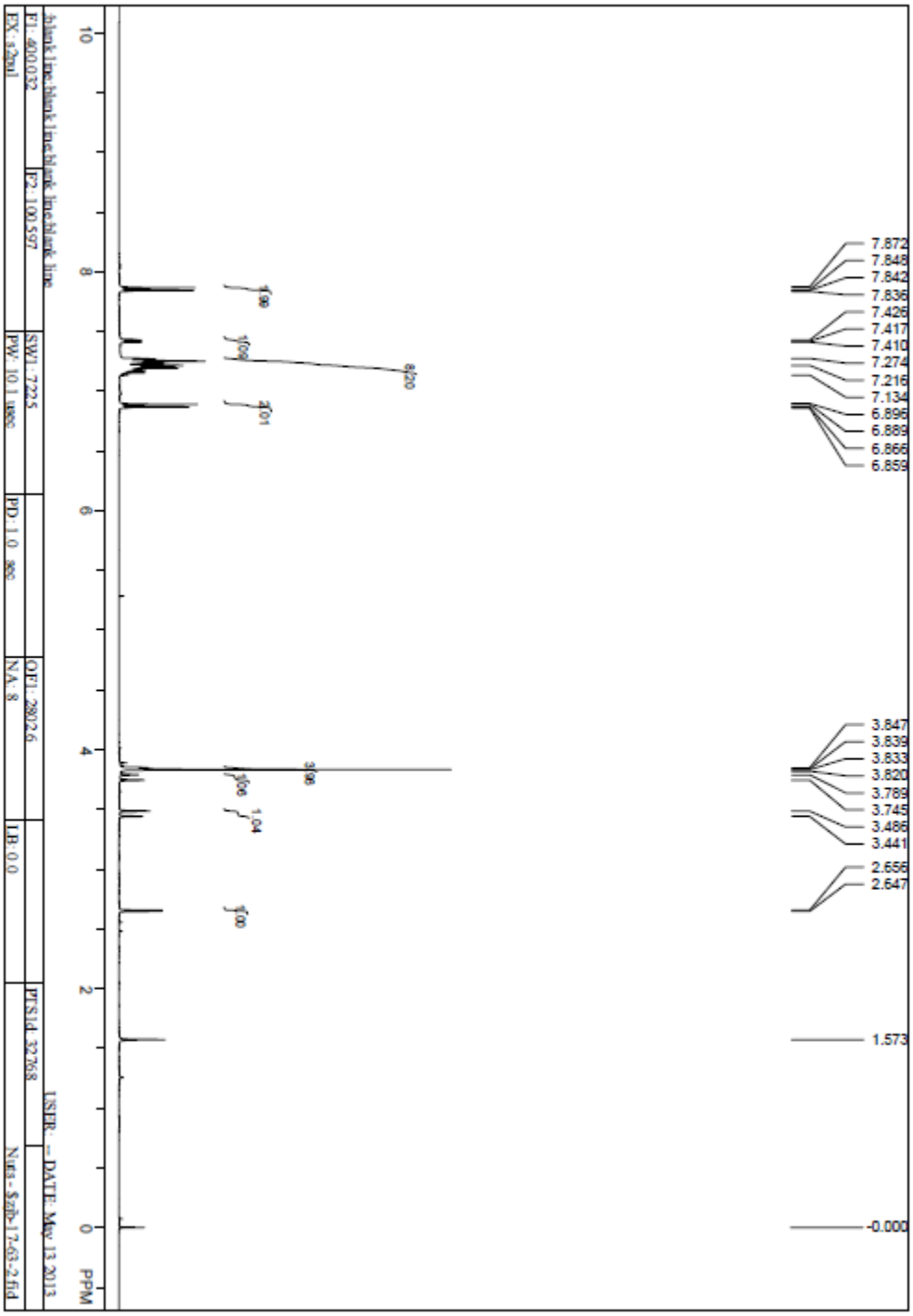
^{13}C NMR (100 M Hz in $CDCl_3$)

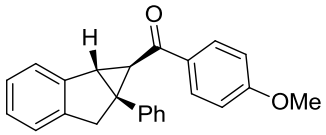




3c

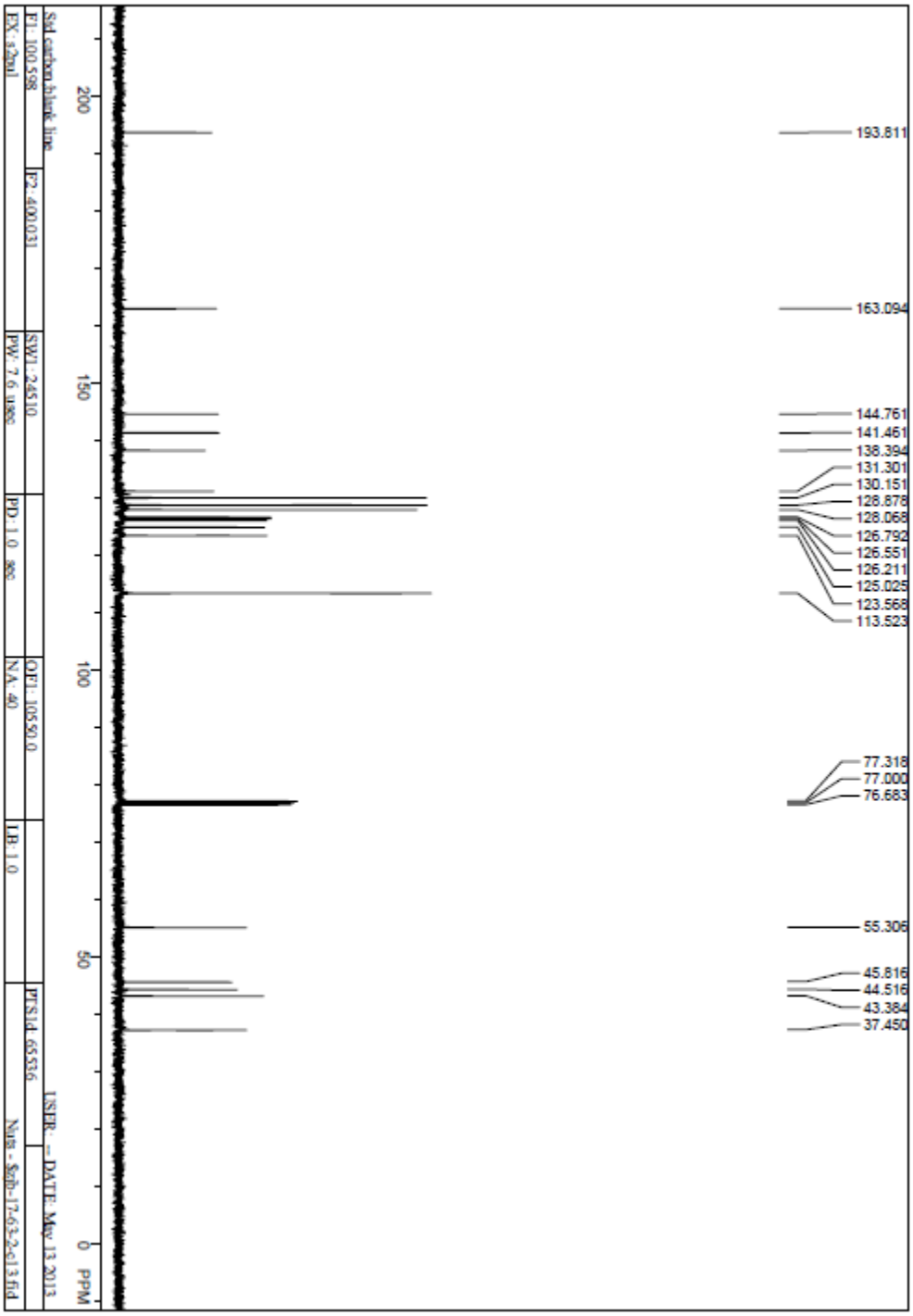
^{1}H NMR (400 M Hz in $CDCl_3$)

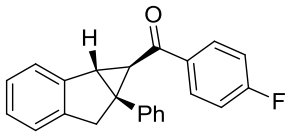




3c

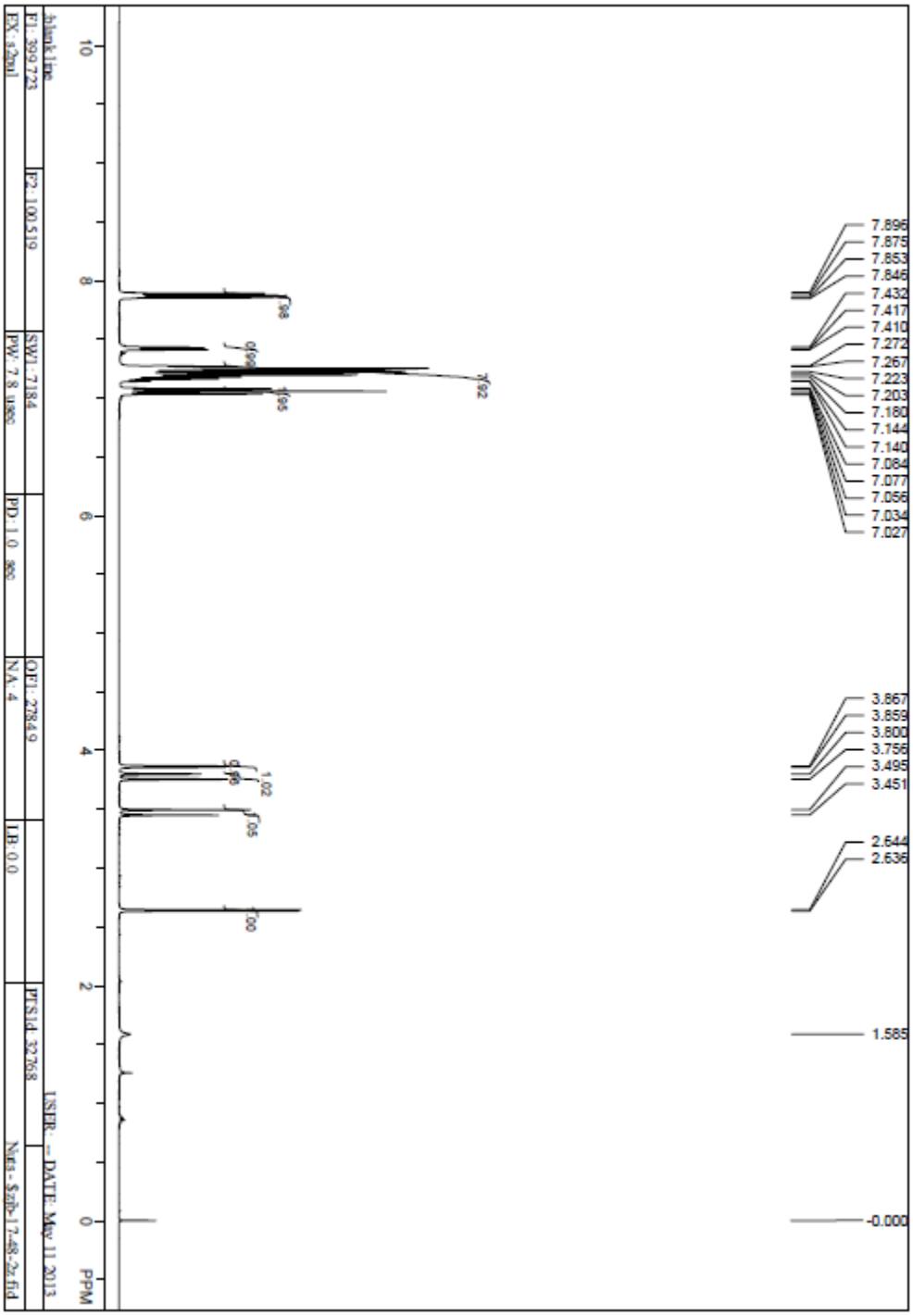
^{13}C NMR (100 M Hz in $CDCl_3$)

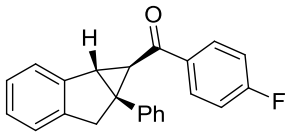




3d

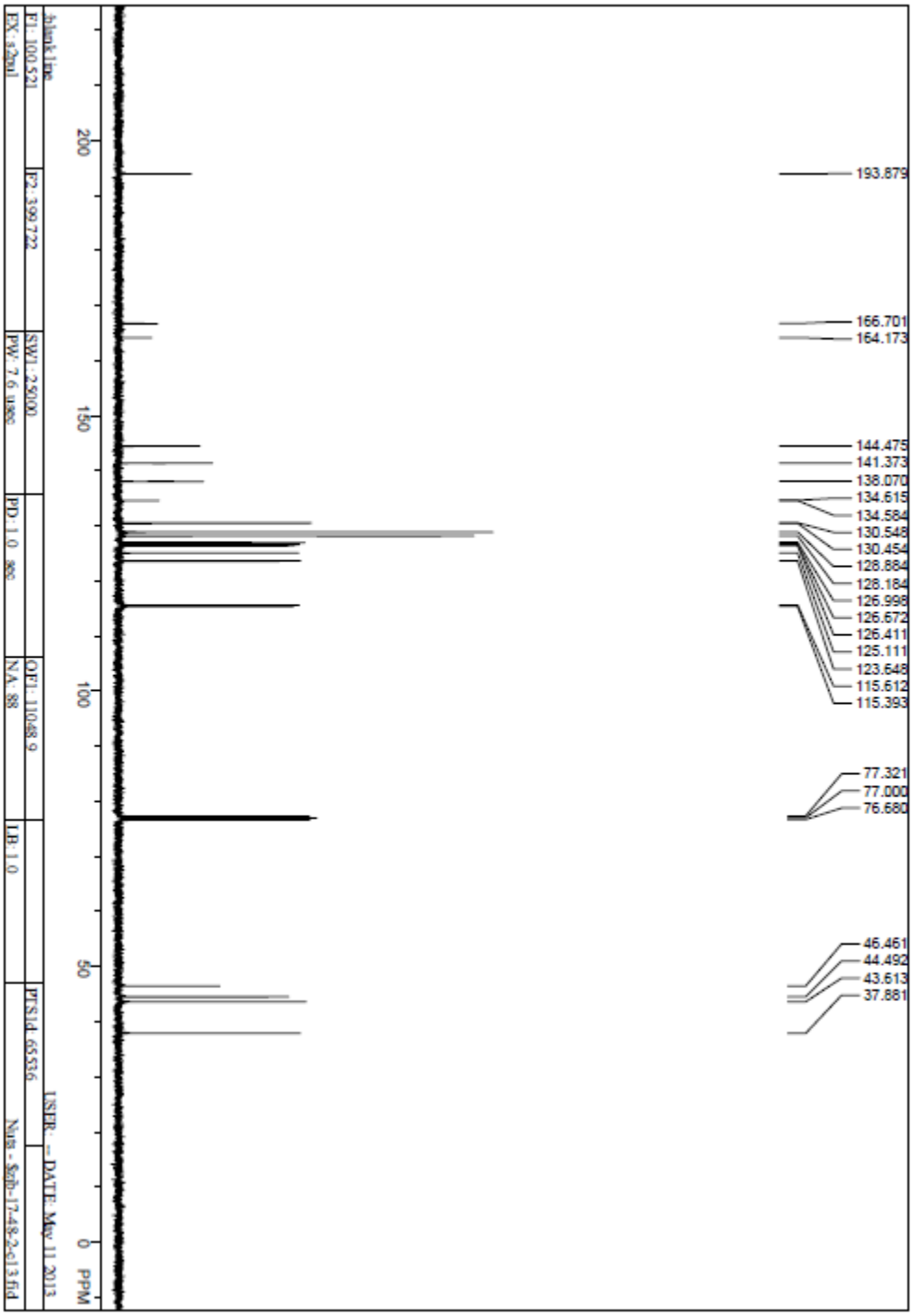
^{1}H NMR (400 M Hz in $CDCl_3$)

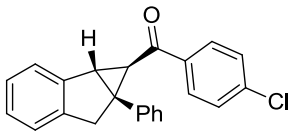




3d

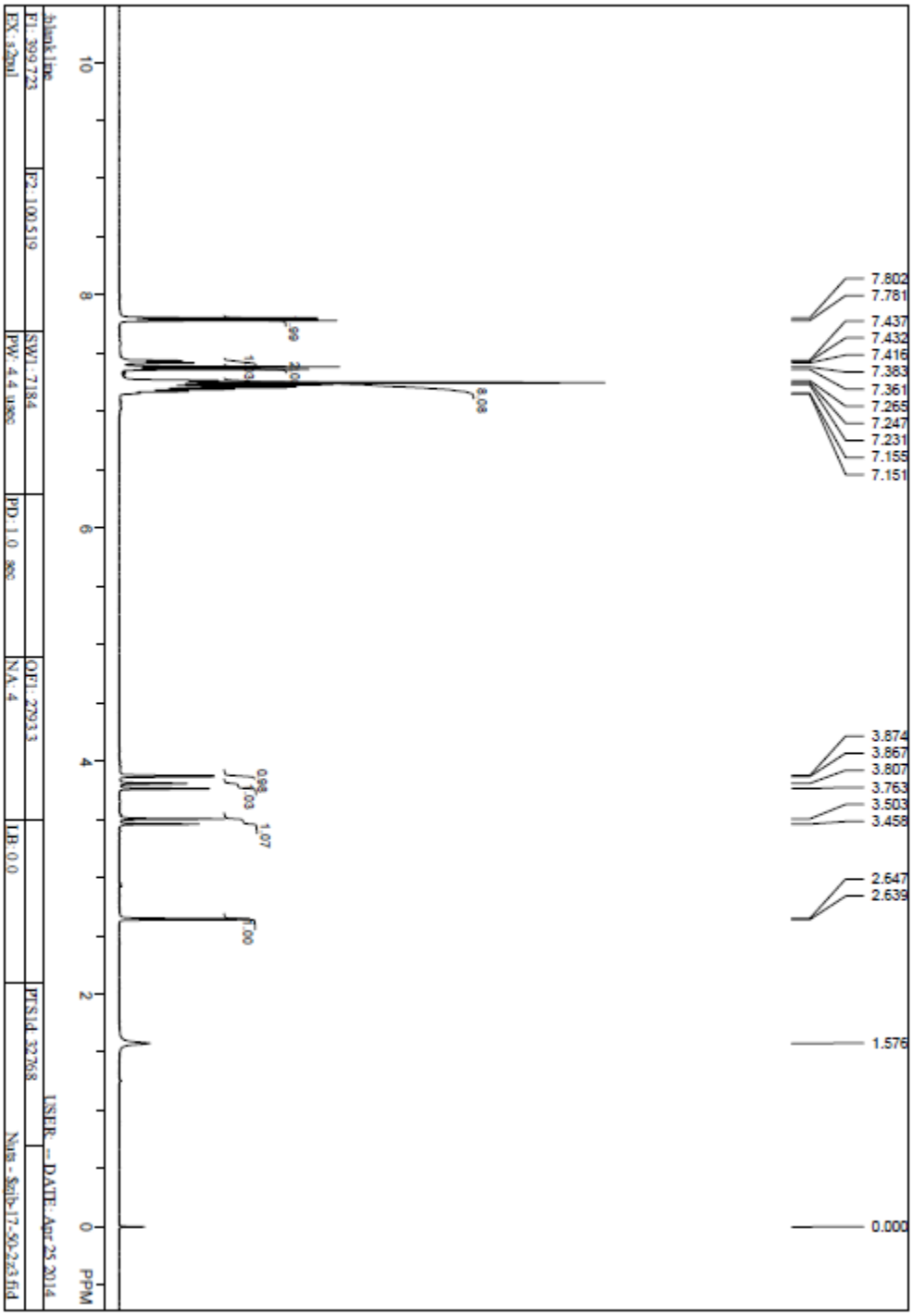
^{13}C NMR (100 M Hz in $CDCl_3$)

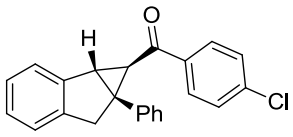




3e

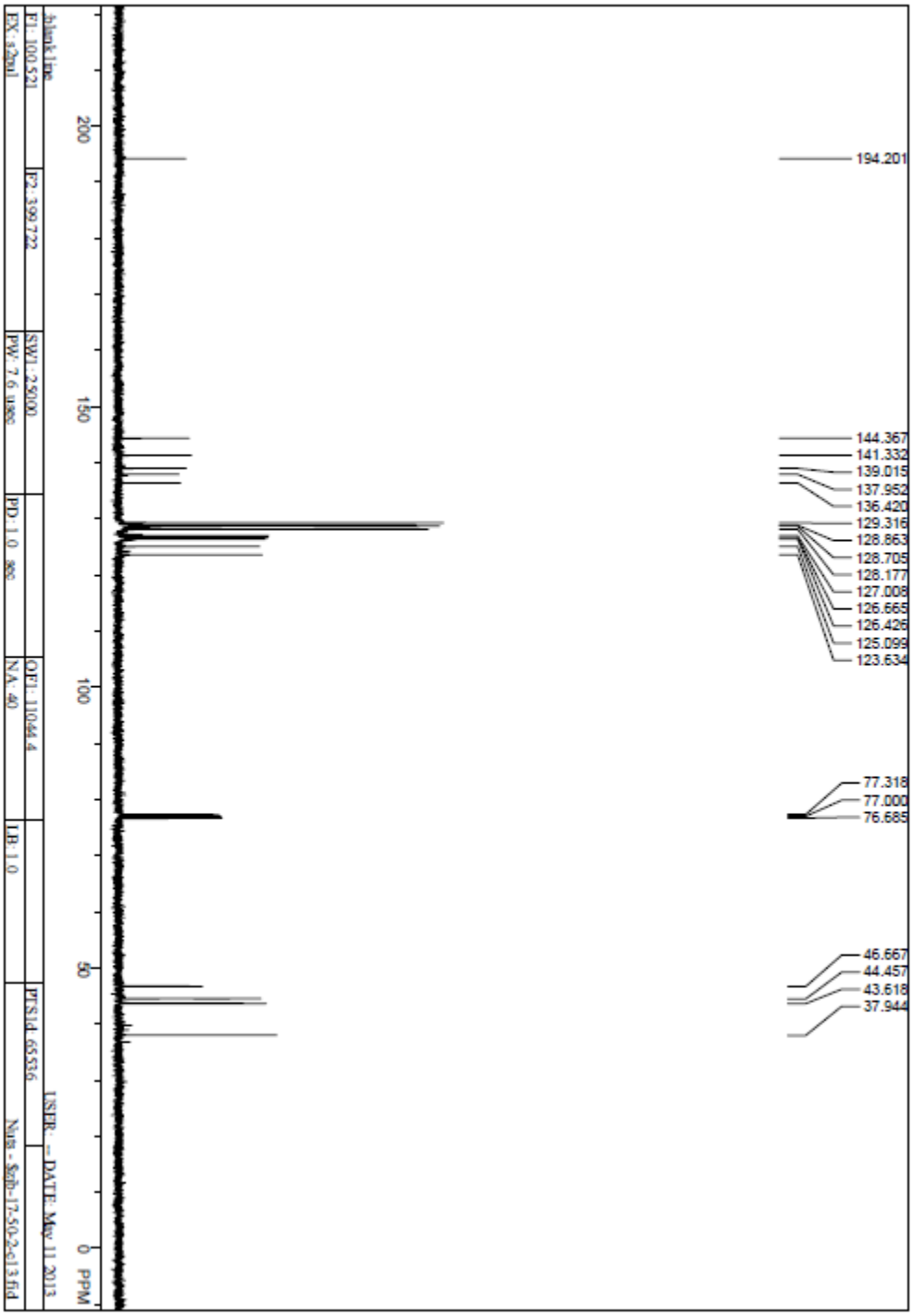
^{1}H NMR (400 M Hz in $CDCl_3$)

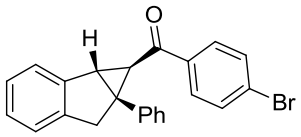




3e

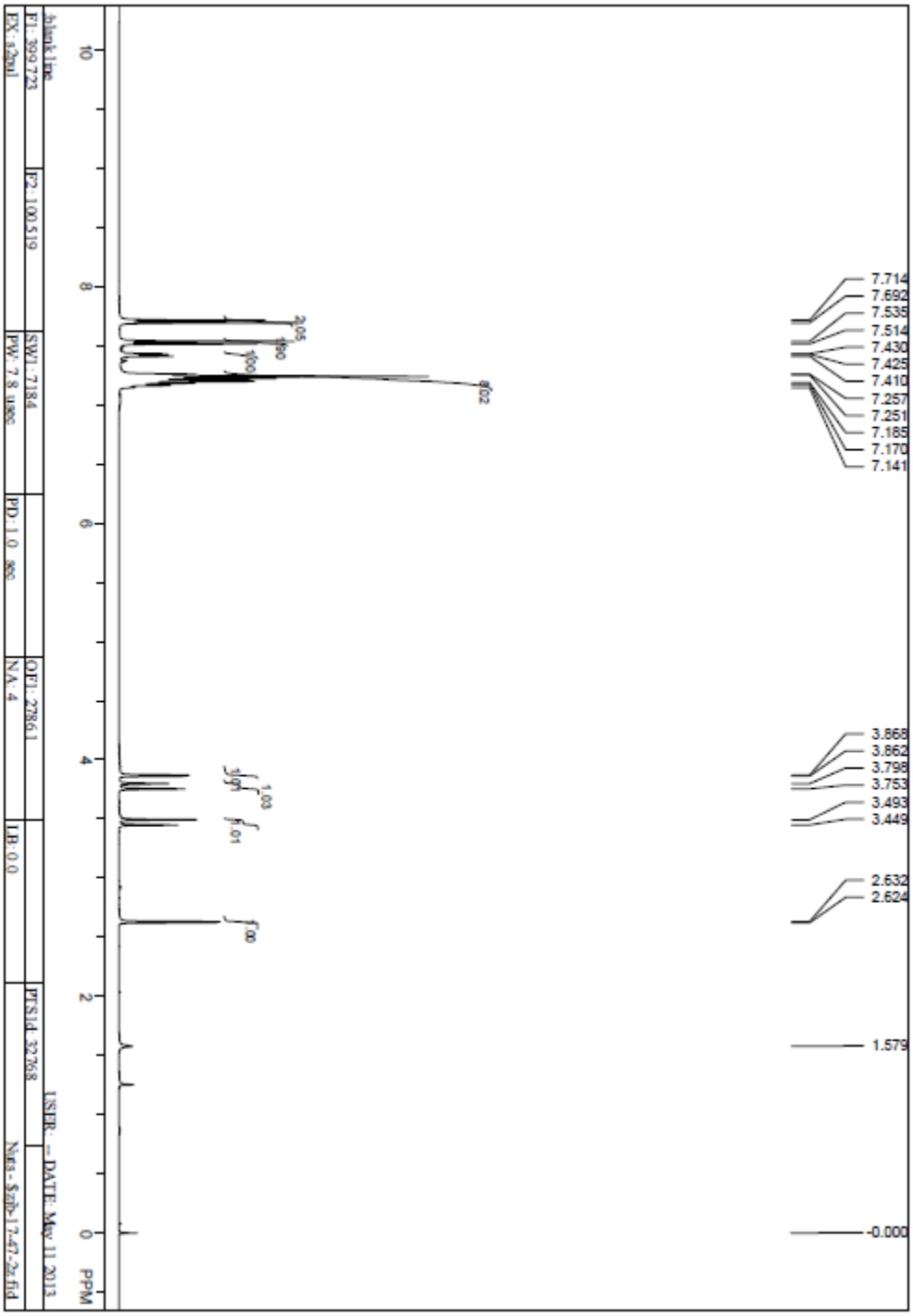
^{13}C NMR (100 M Hz in $CDCl_3$)

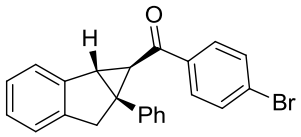




3f

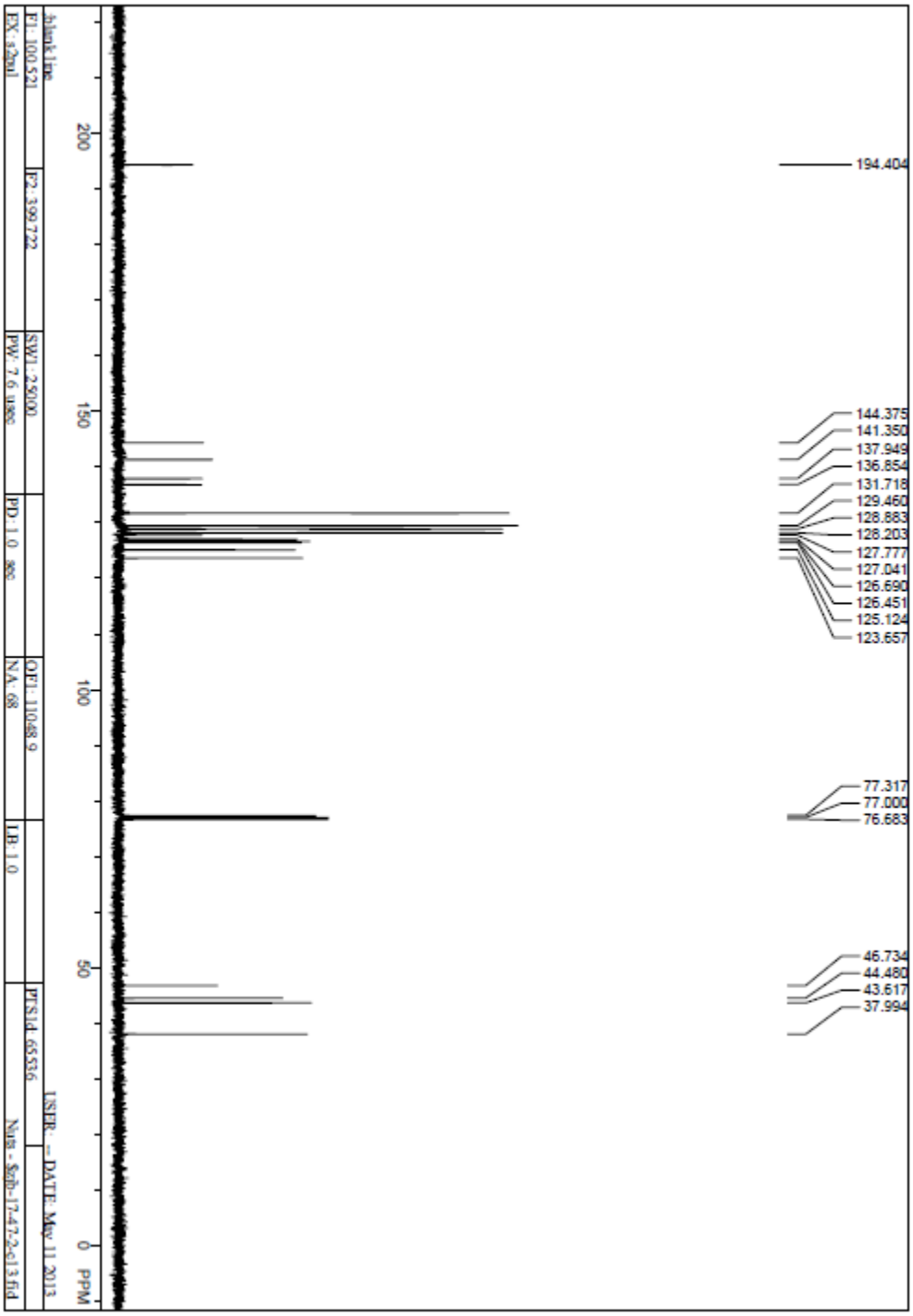
^{1}H NMR (400 M Hz in $CDCl_3$)

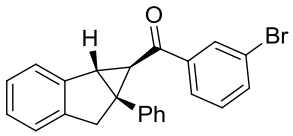




3f

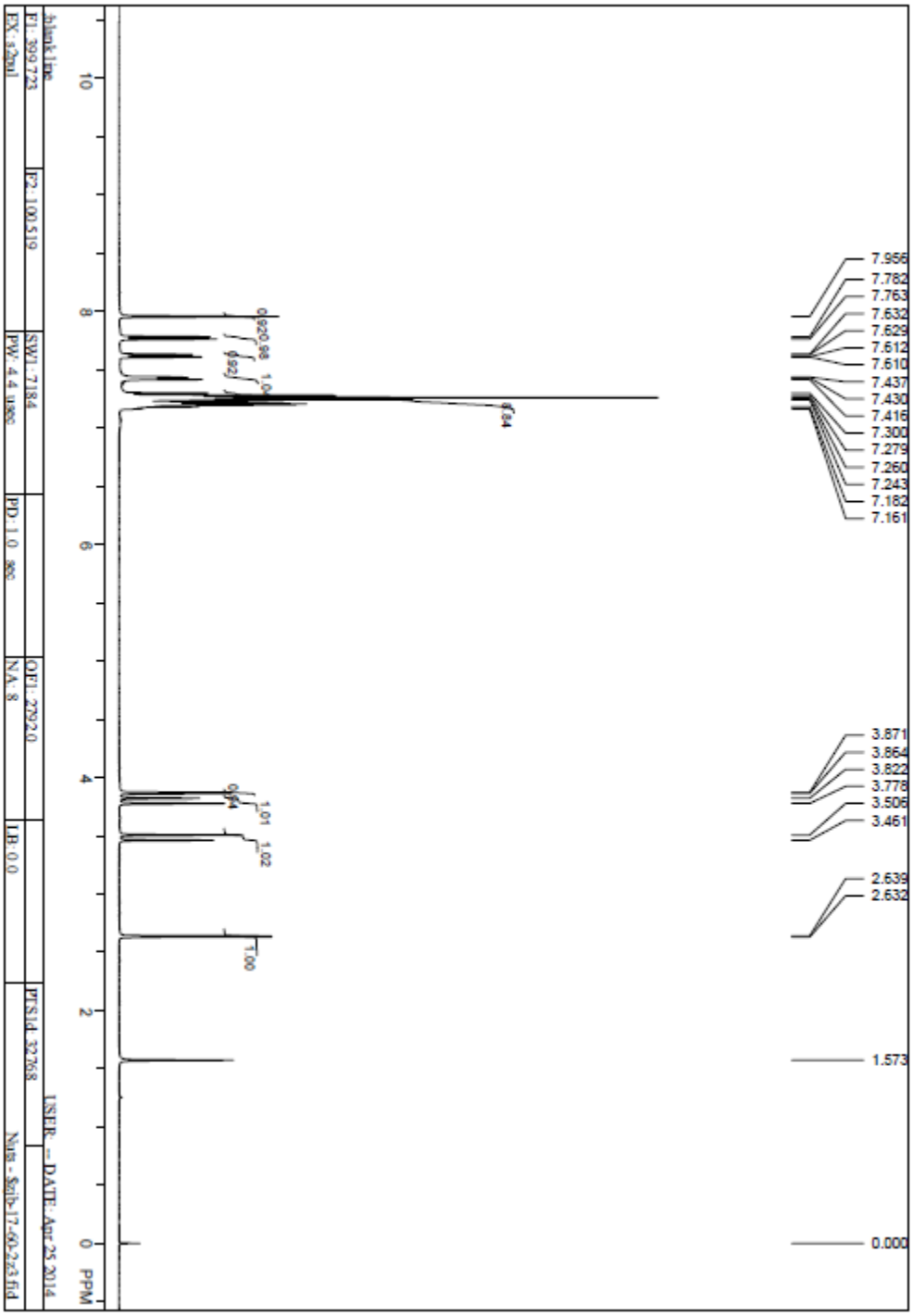
^{13}C NMR (100 M Hz in $CDCl_3$)

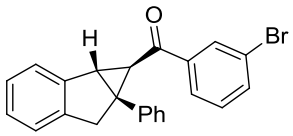




3g

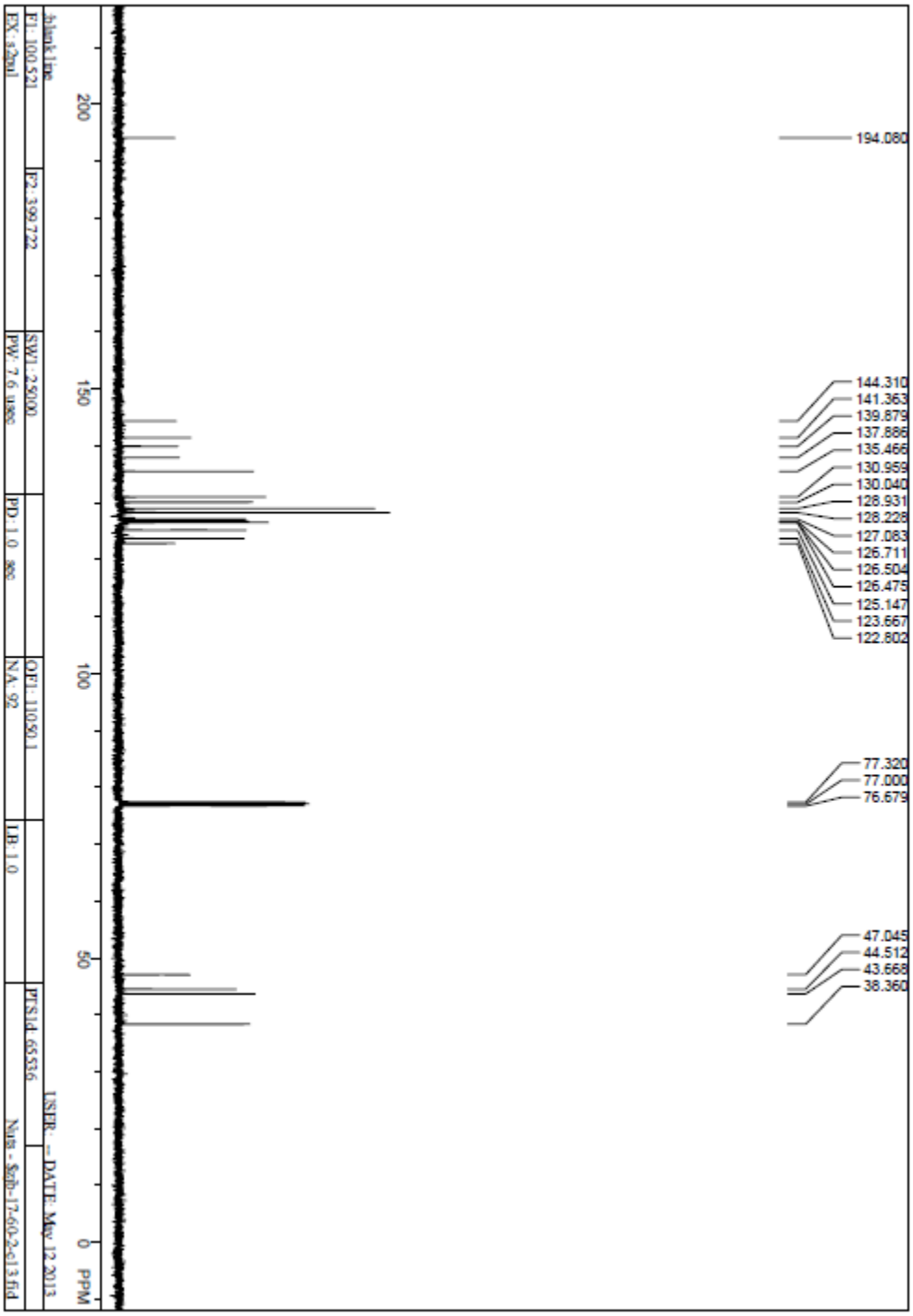
^{1}H NMR (400 M Hz in $CDCl_3$)

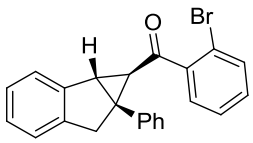




3g

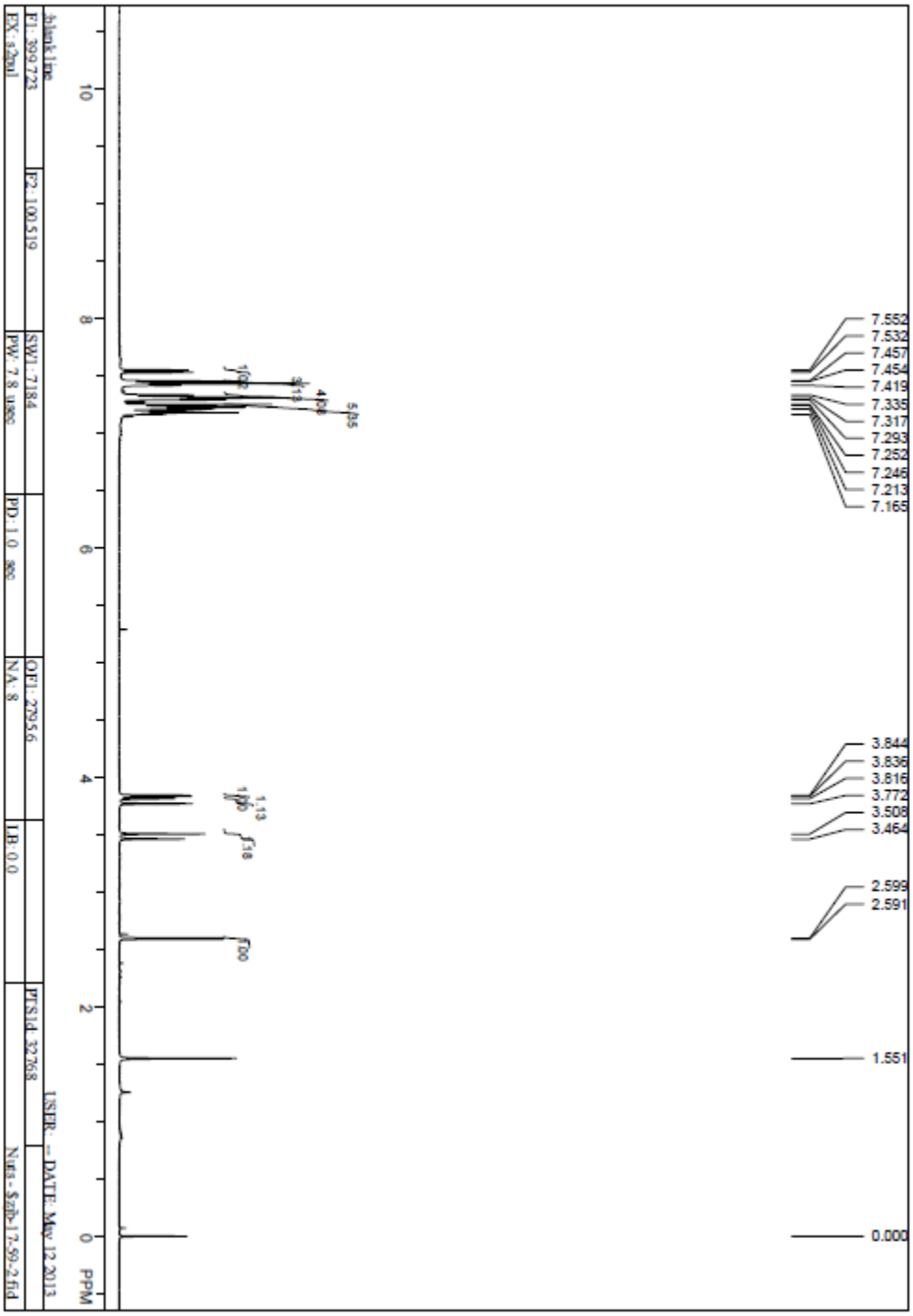
^{13}C NMR (100 M Hz in $CDCl_3$)

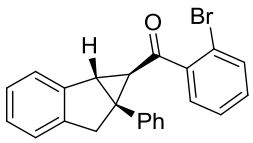




3h

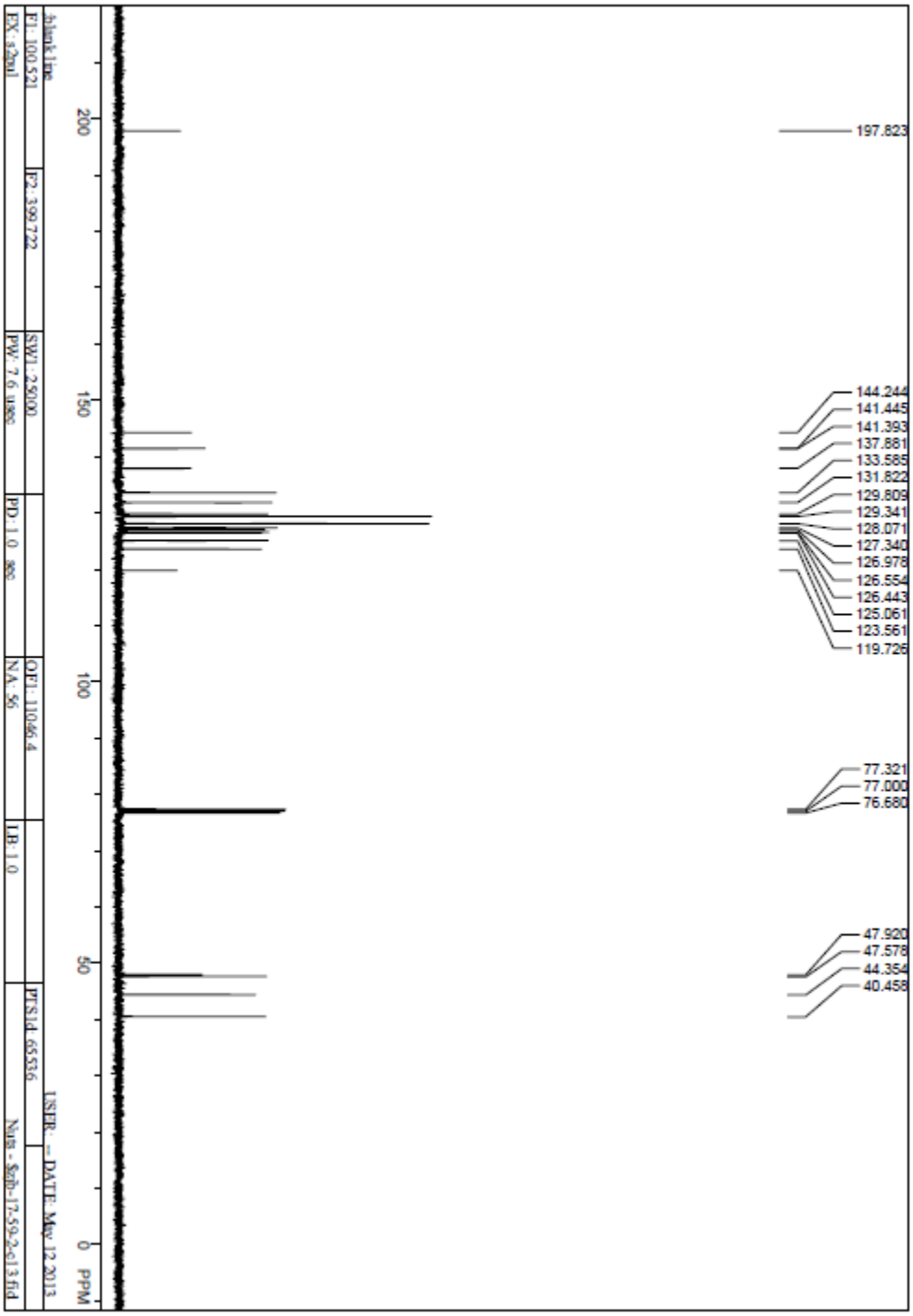
^{1}H NMR (400 M Hz in $CDCl_3$)

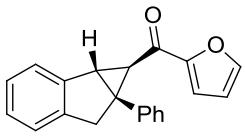




3h

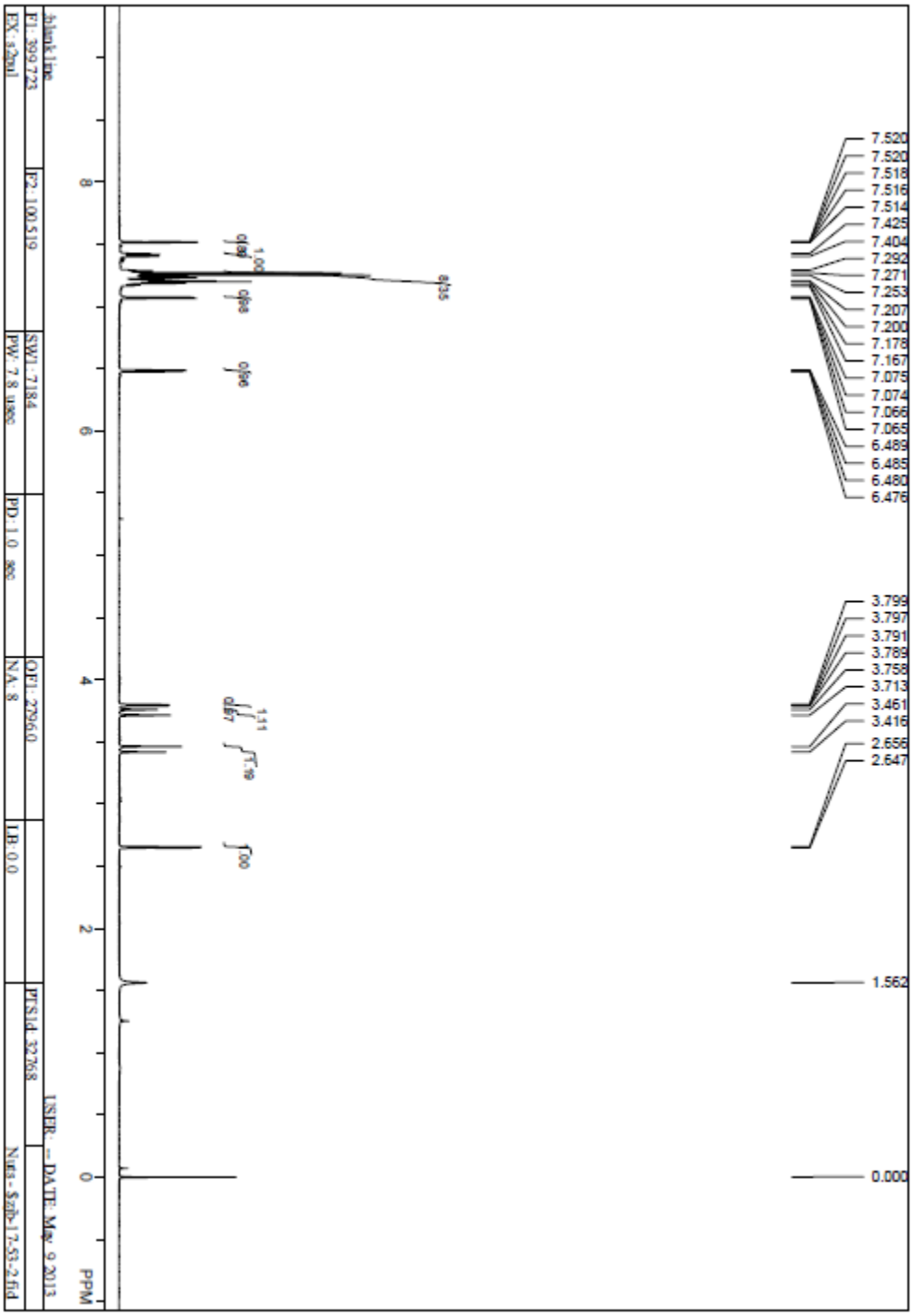
^{13}C NMR (100 M Hz in $CDCl_3$)

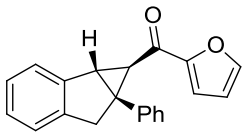




3i

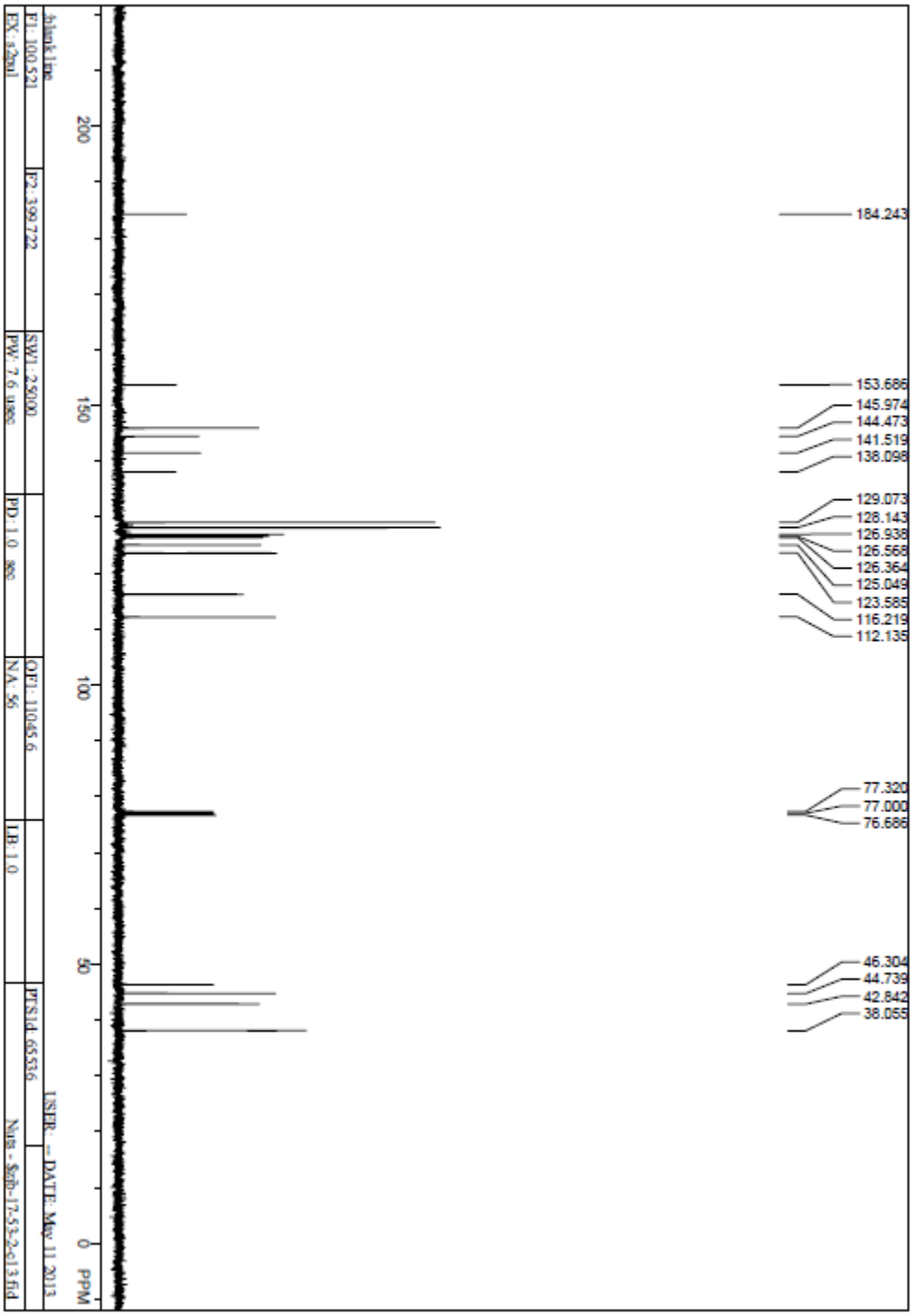
^{1}H NMR (400 M Hz in $CDCl_3$)

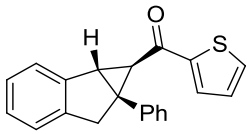




3i

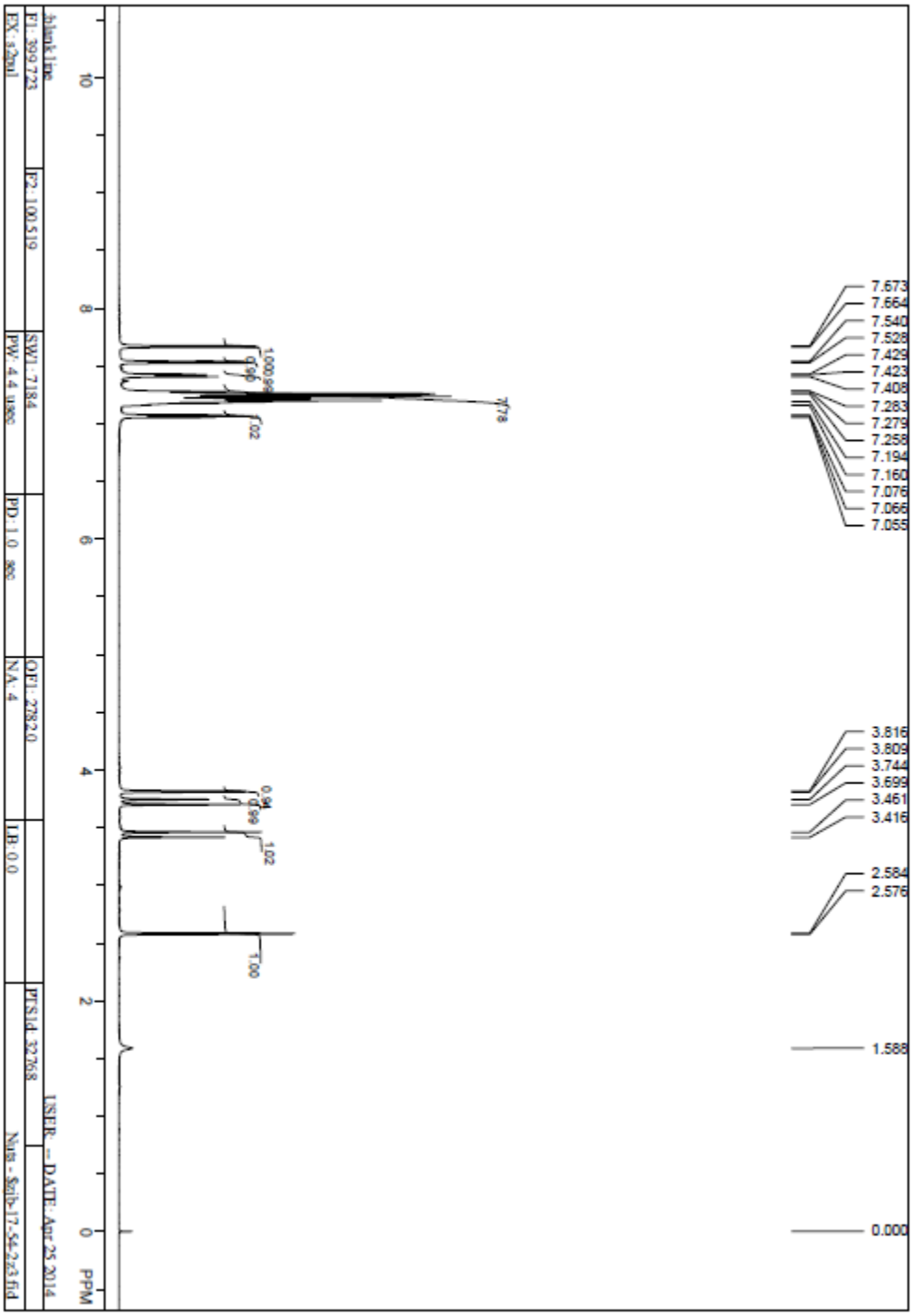
^{13}C NMR (100 M Hz in $CDCl_3$)

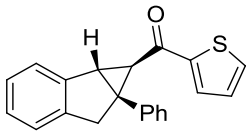




3j

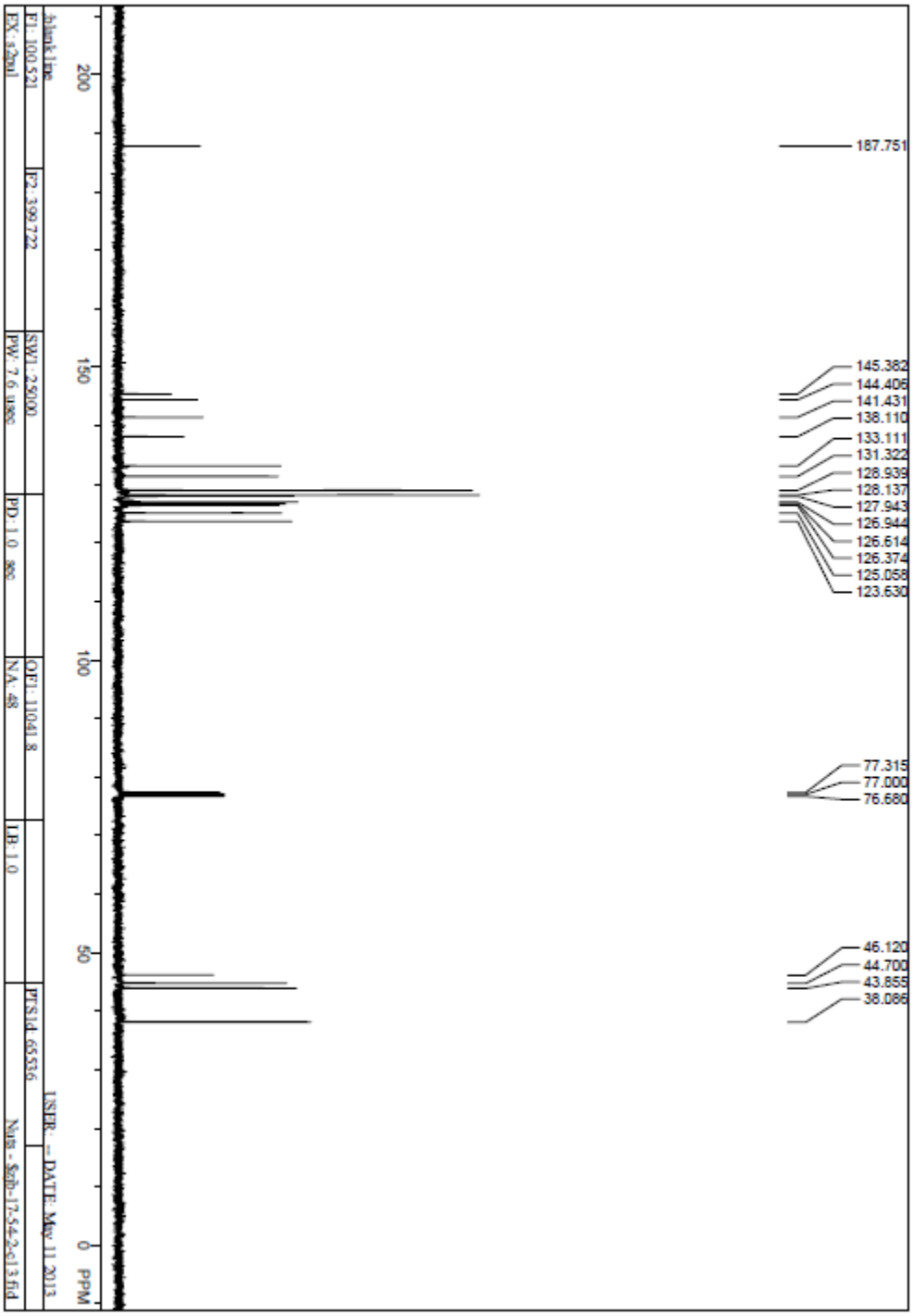
^{1}H NMR (400 M Hz in $CDCl_3$)

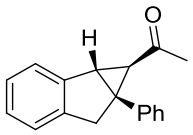




3j

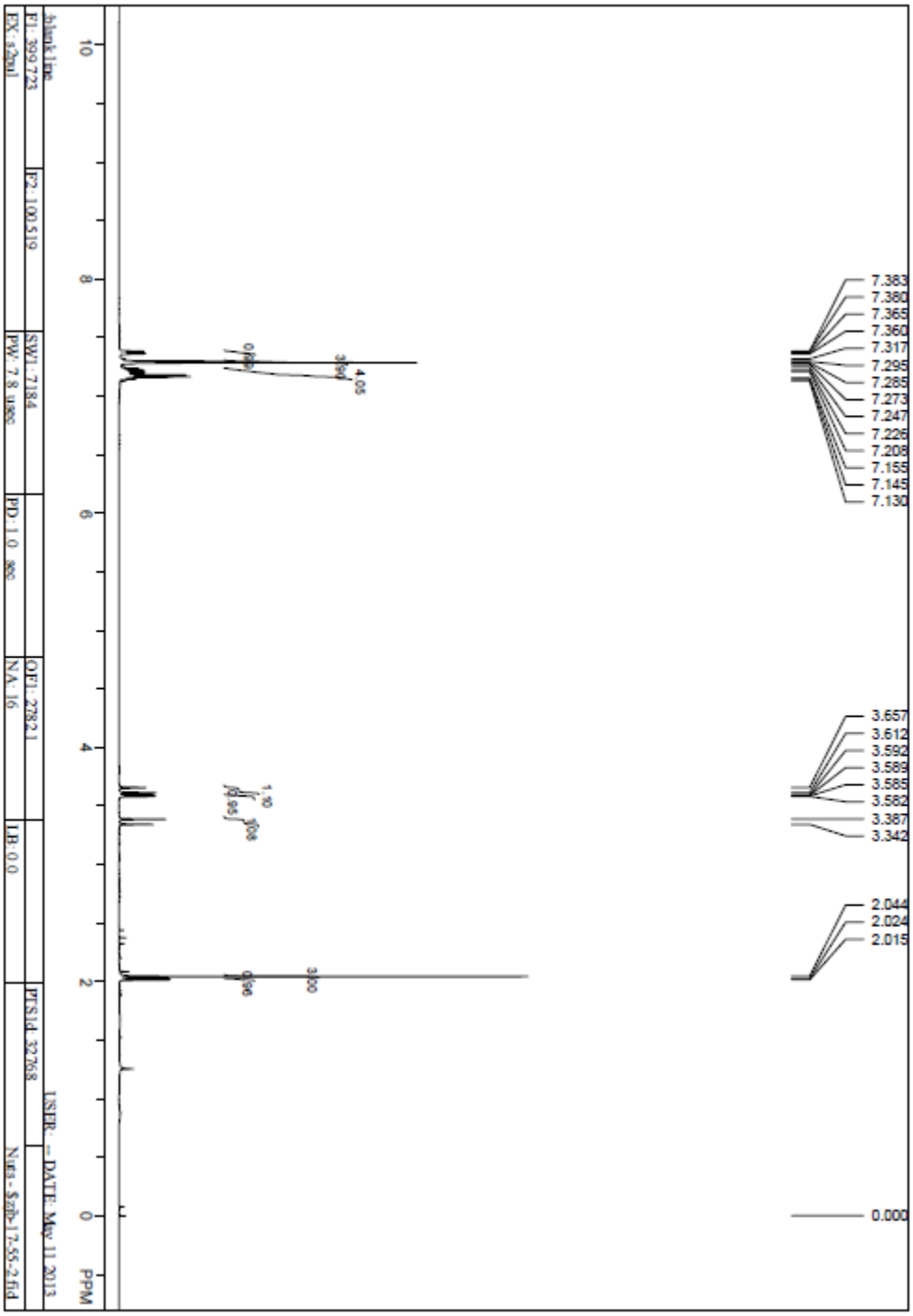
^{13}C NMR (100 M Hz in $CDCl_3$)

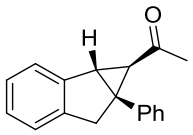




3k

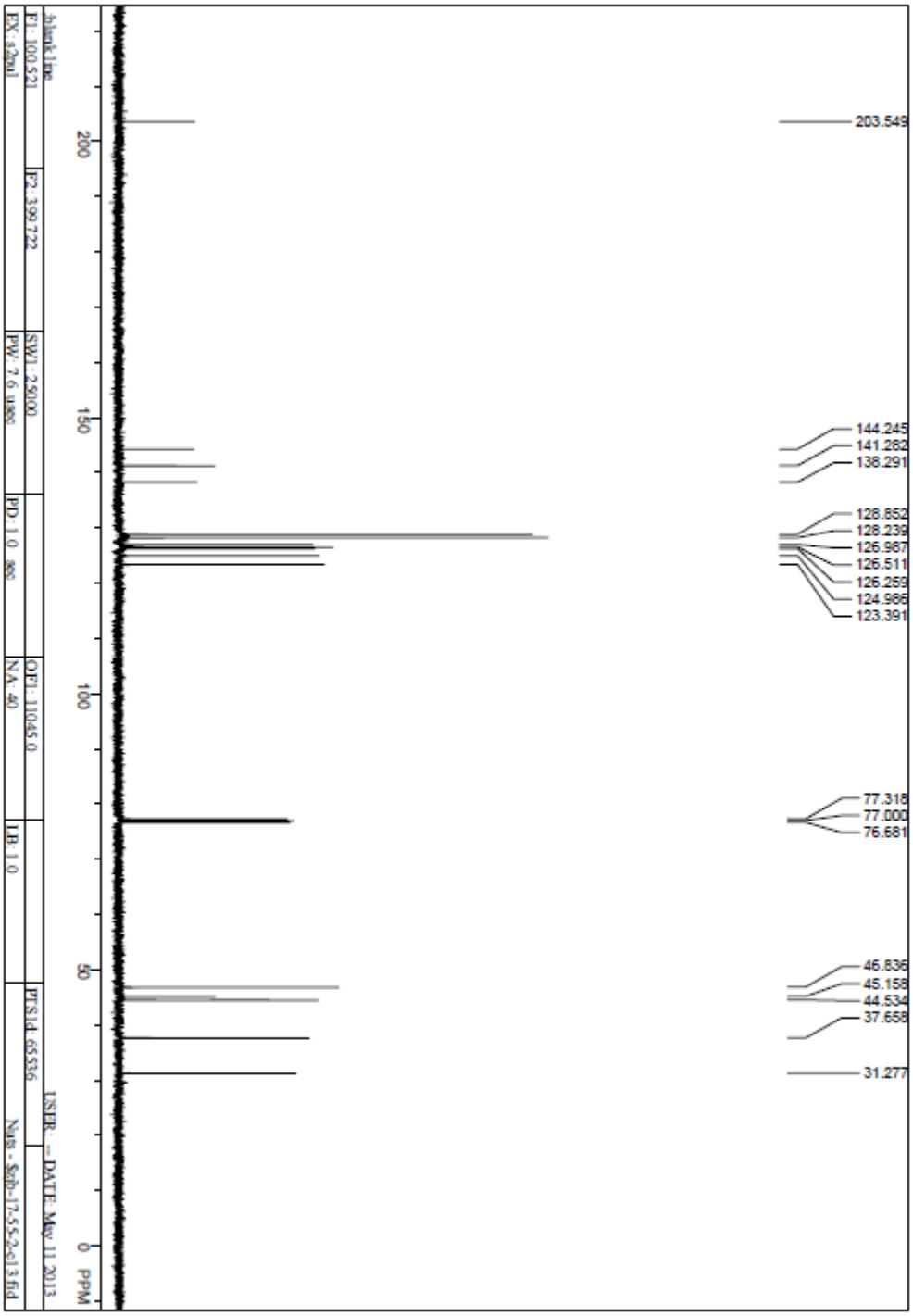
^{1}H NMR (400 M Hz in $CDCl_3$)

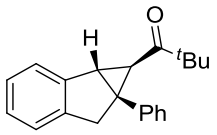




3k

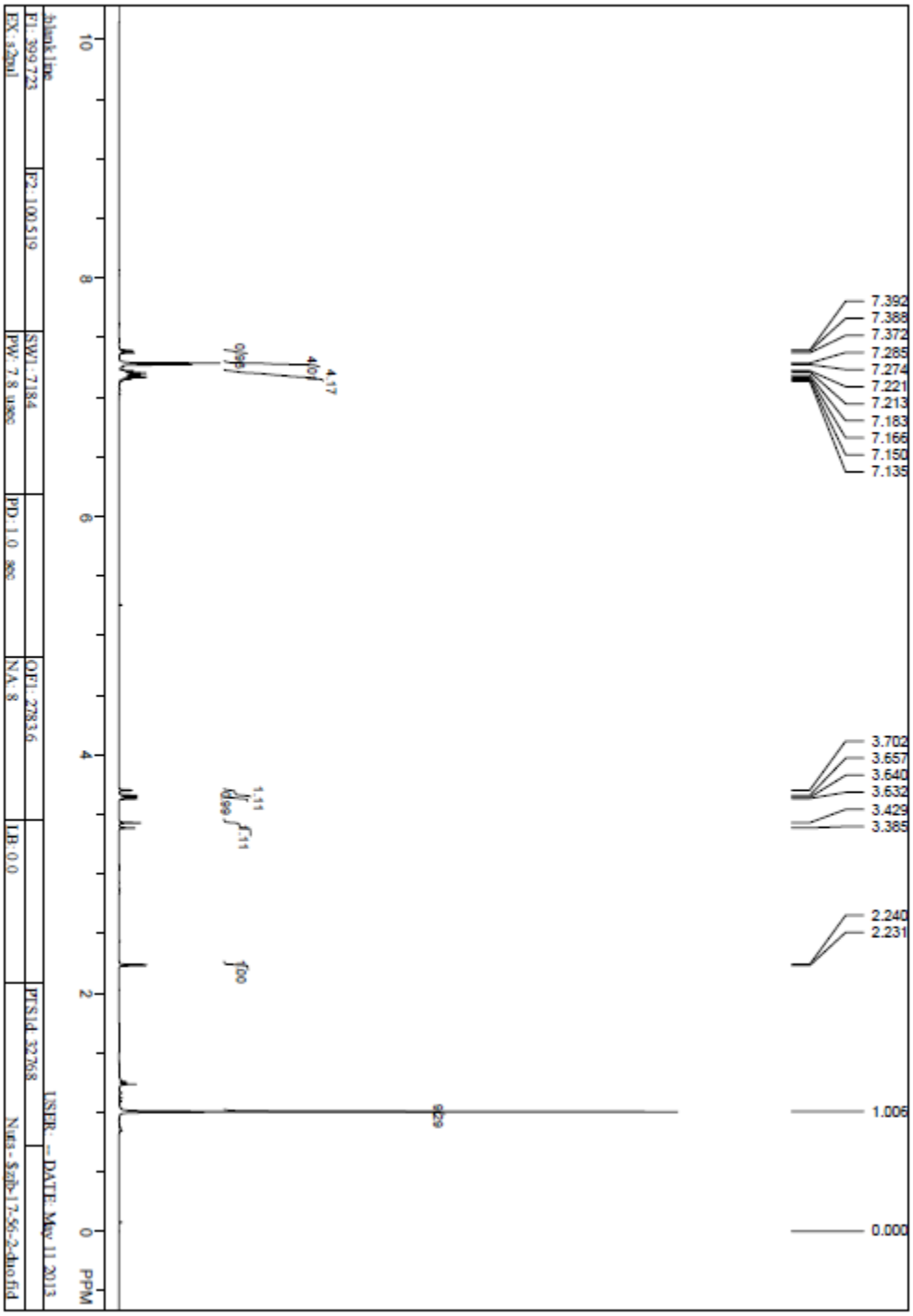
^{13}C NMR (100 M Hz in $CDCl_3$)

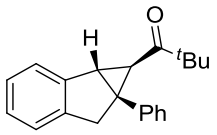




3l

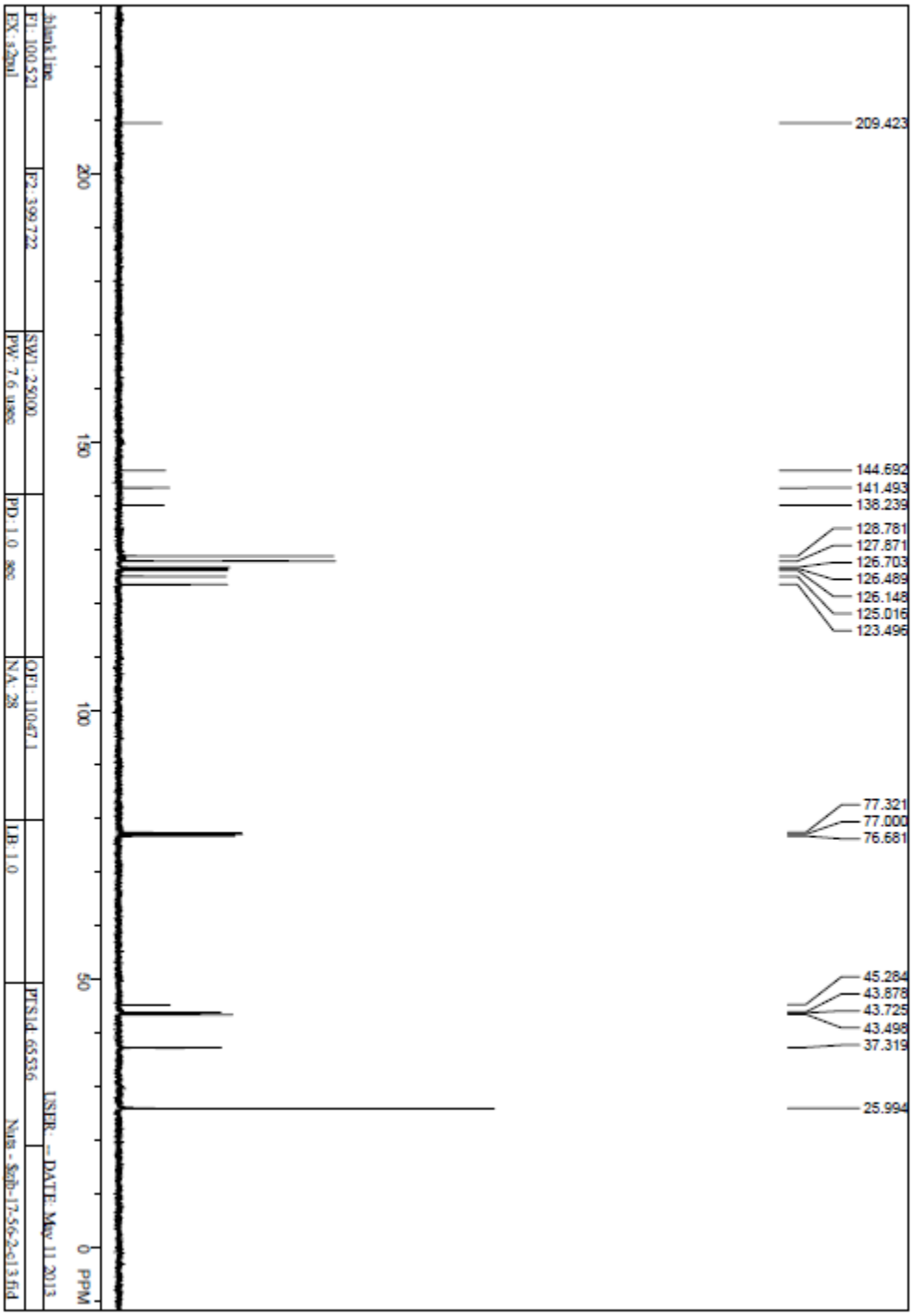
^{1}H NMR (400 M Hz in $CDCl_3$)

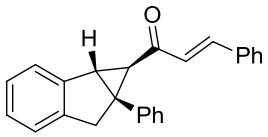




3l

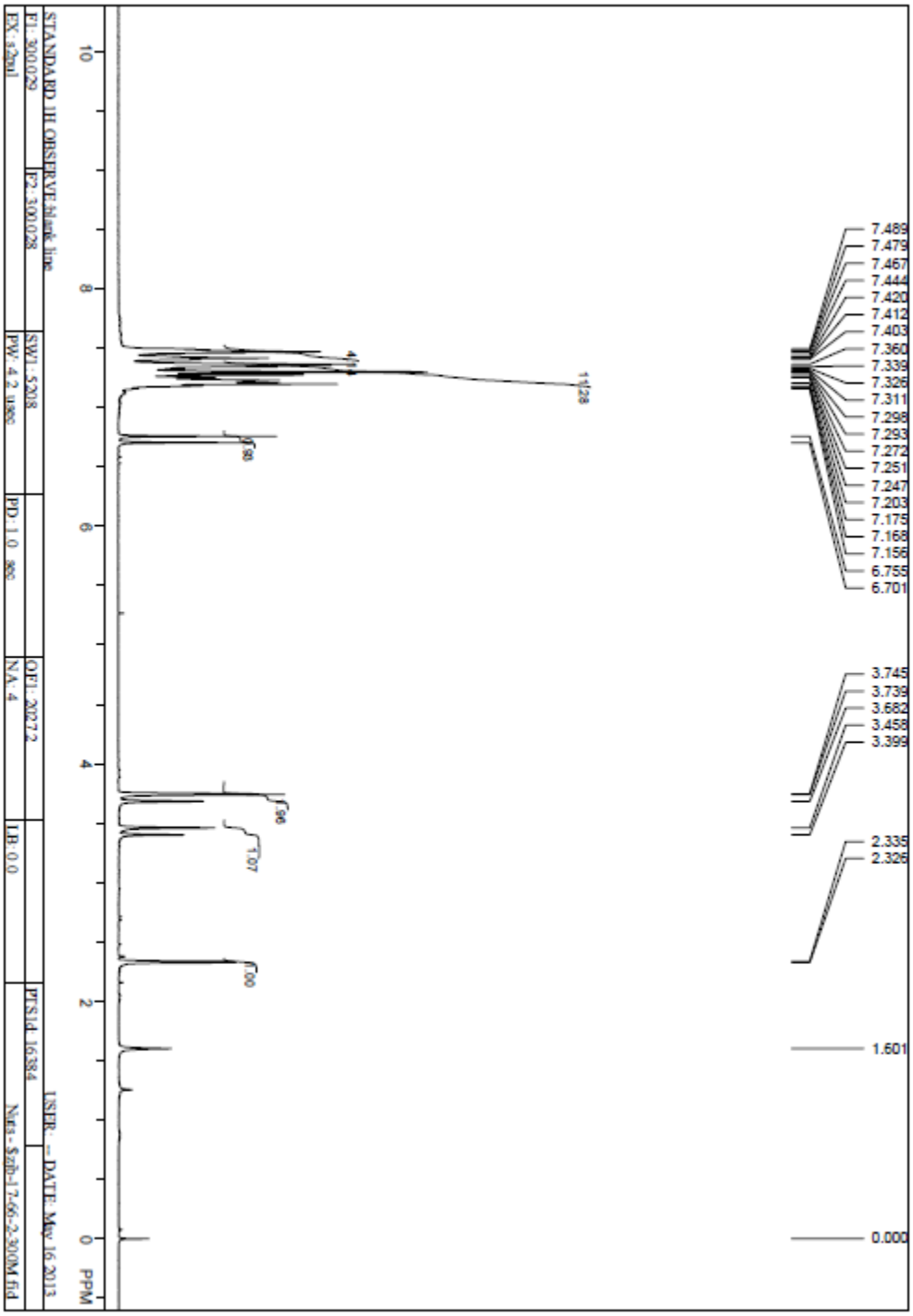
^{13}C NMR (100 M Hz in $CDCl_3$)

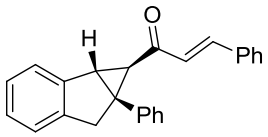




3m

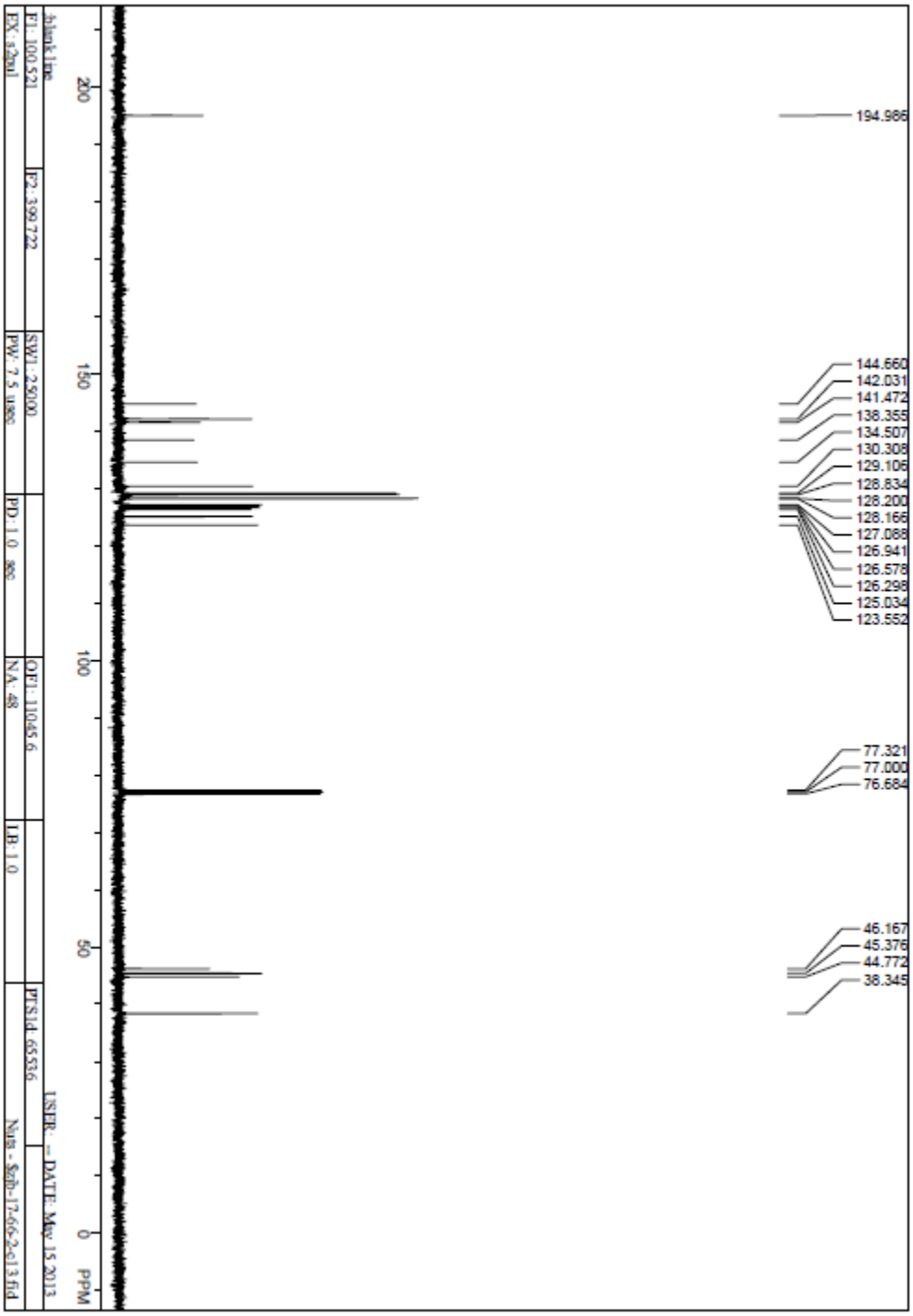
^{1}H NMR (300 M Hz in $CDCl_3$)

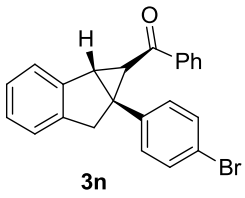




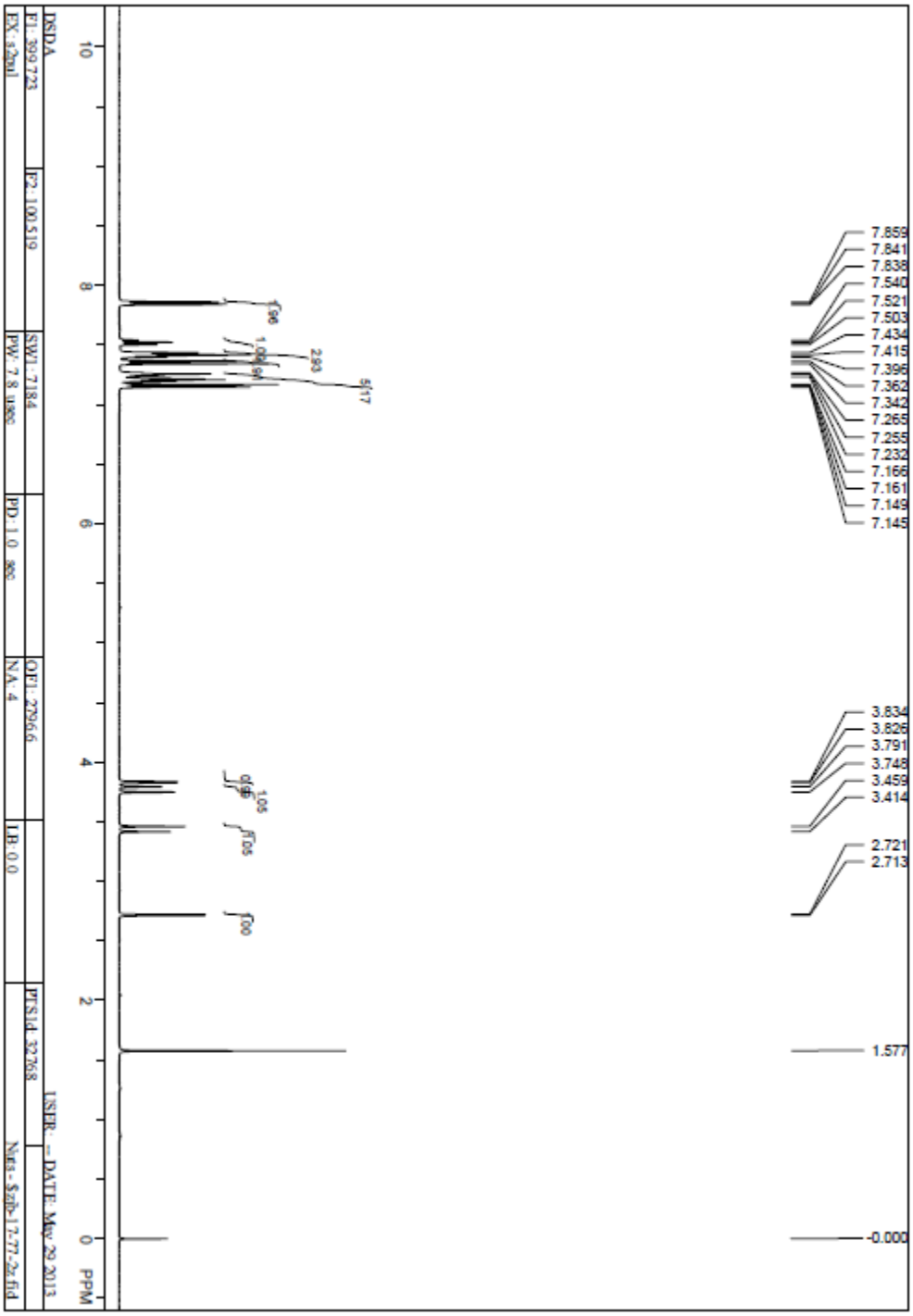
3m

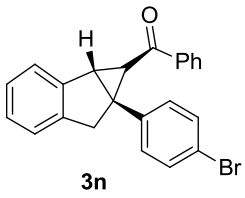
^{13}C NMR (100 M Hz in $CDCl_3$)



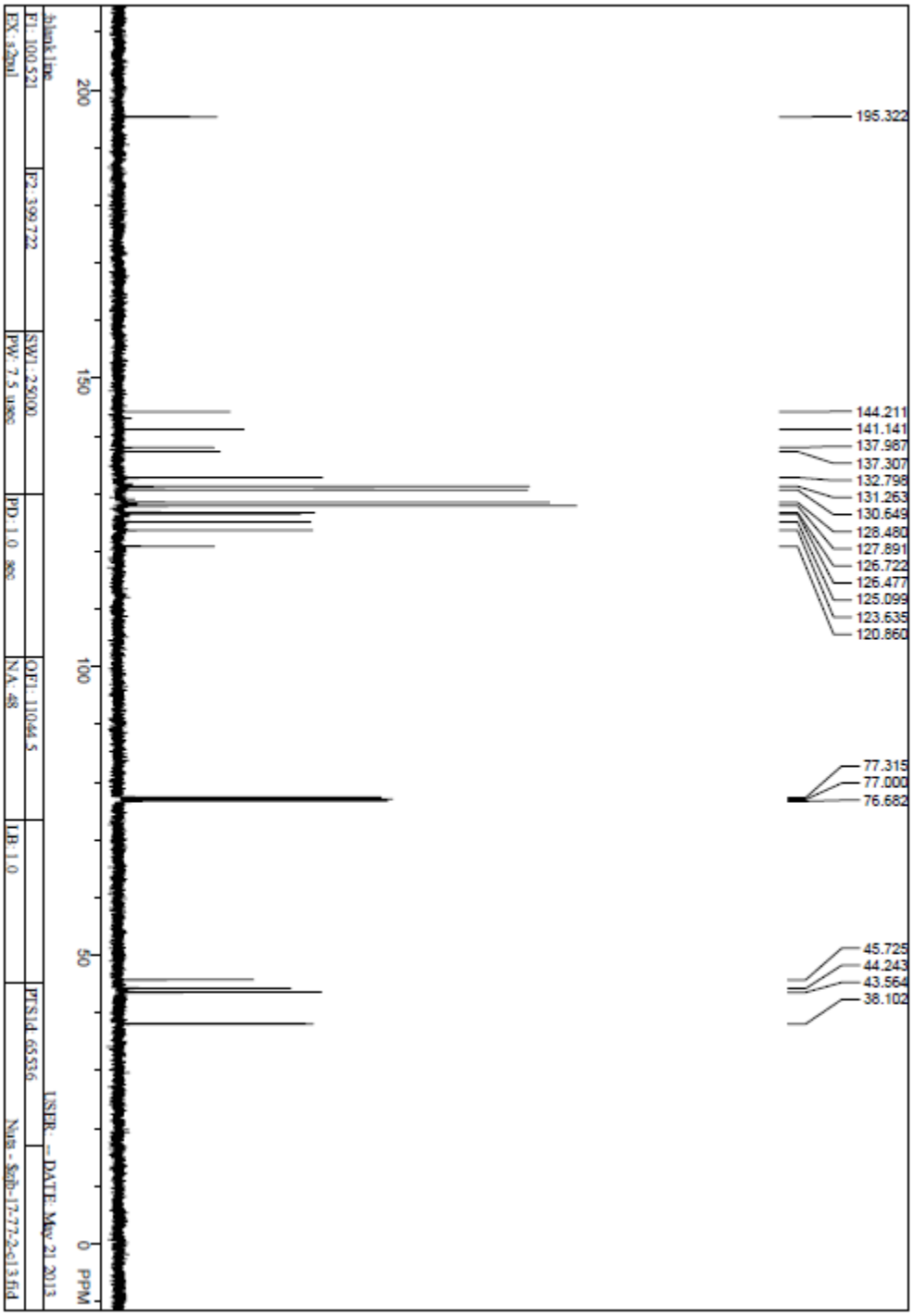


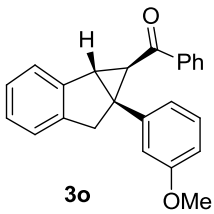
3n ^{1}H NMR (400 M Hz in $CDCl_3$)



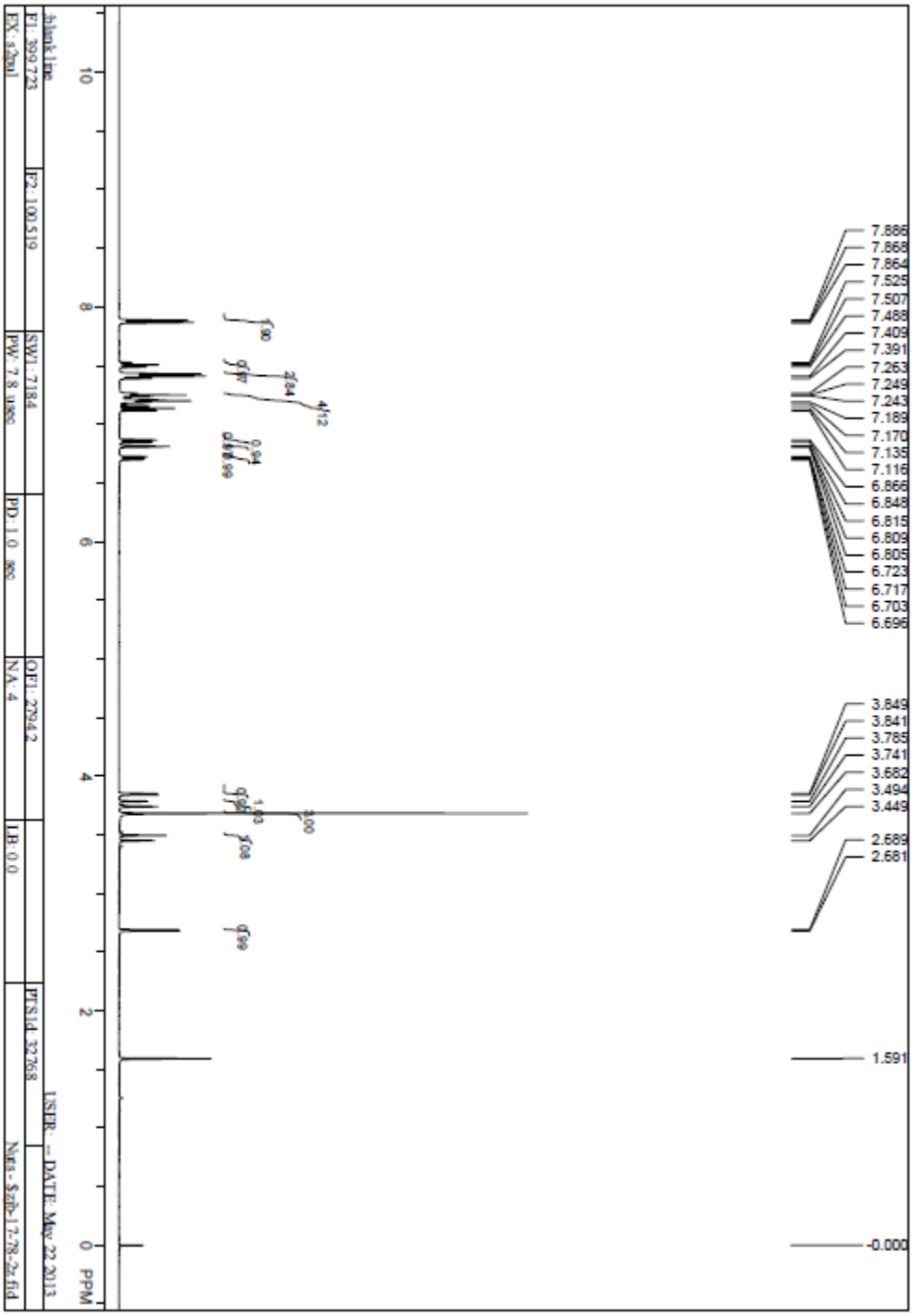


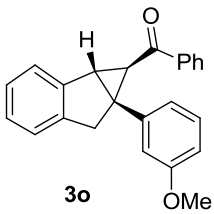
3n ^{13}C NMR (100 M Hz in $CDCl_3$)



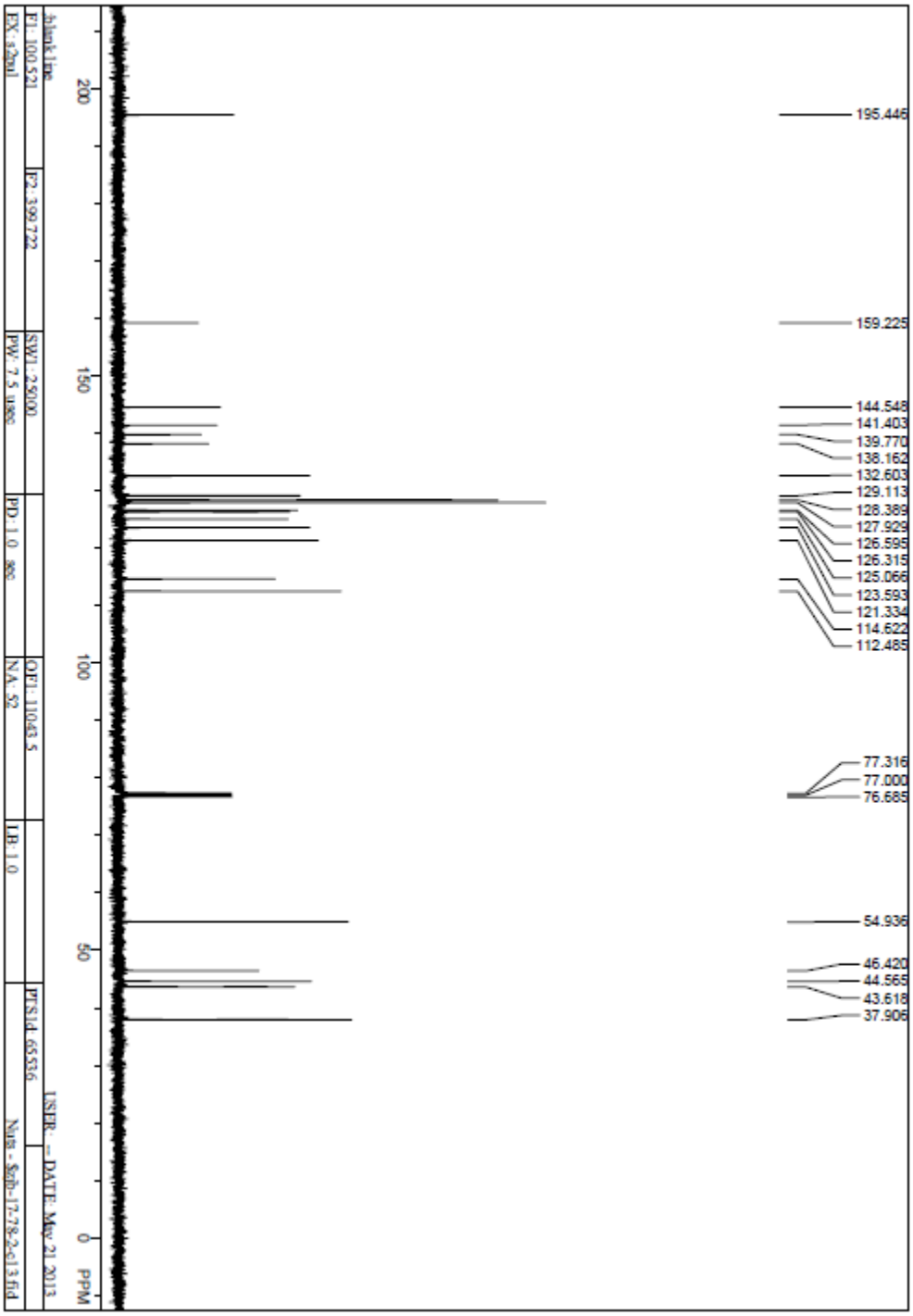


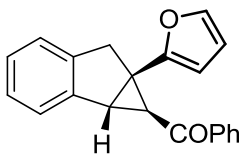
3o ^{1}H NMR (400 M Hz in $CDCl_3$)





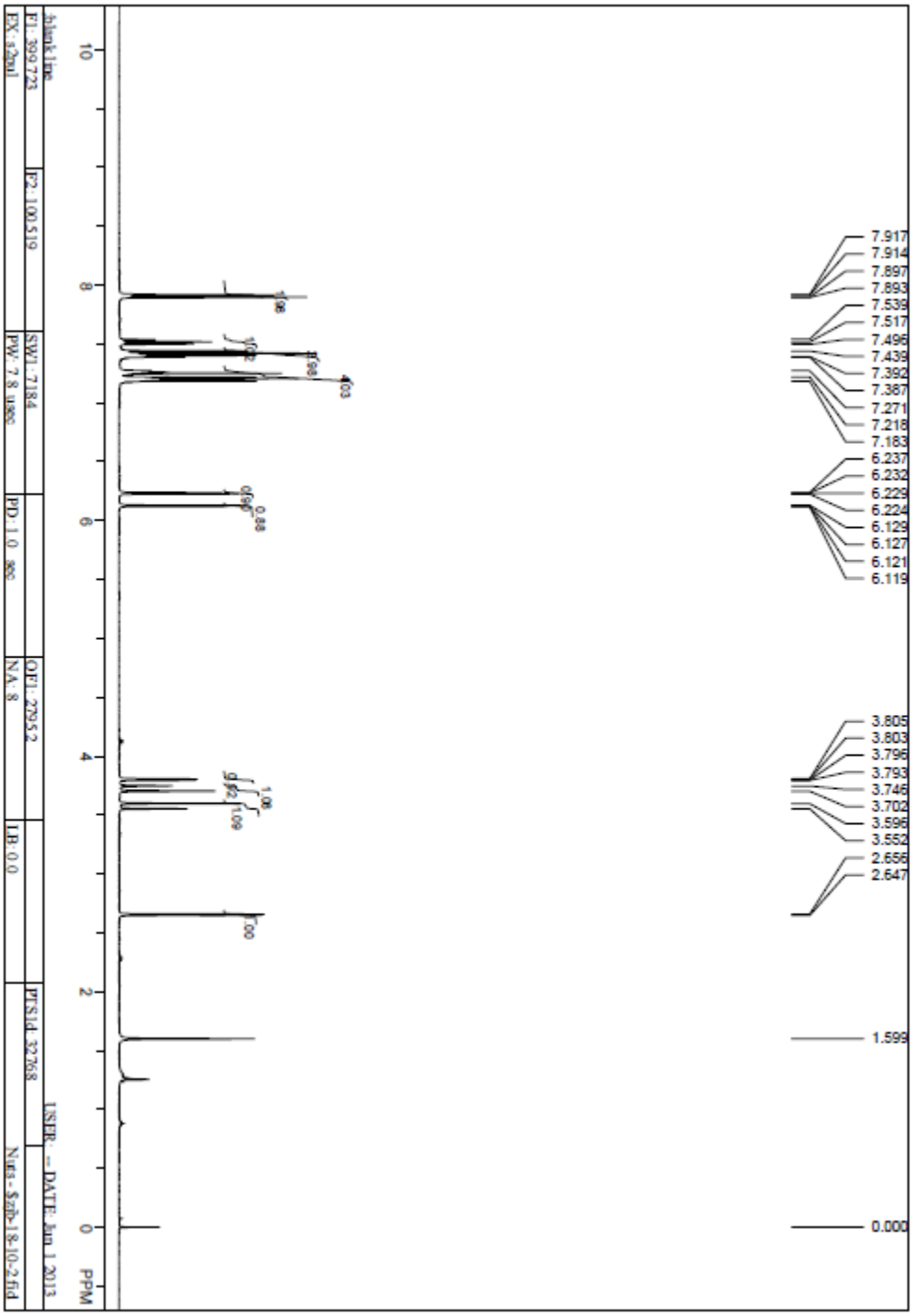
3o ^{13}C NMR (100 M Hz in $CDCl_3$)

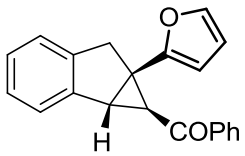




3p

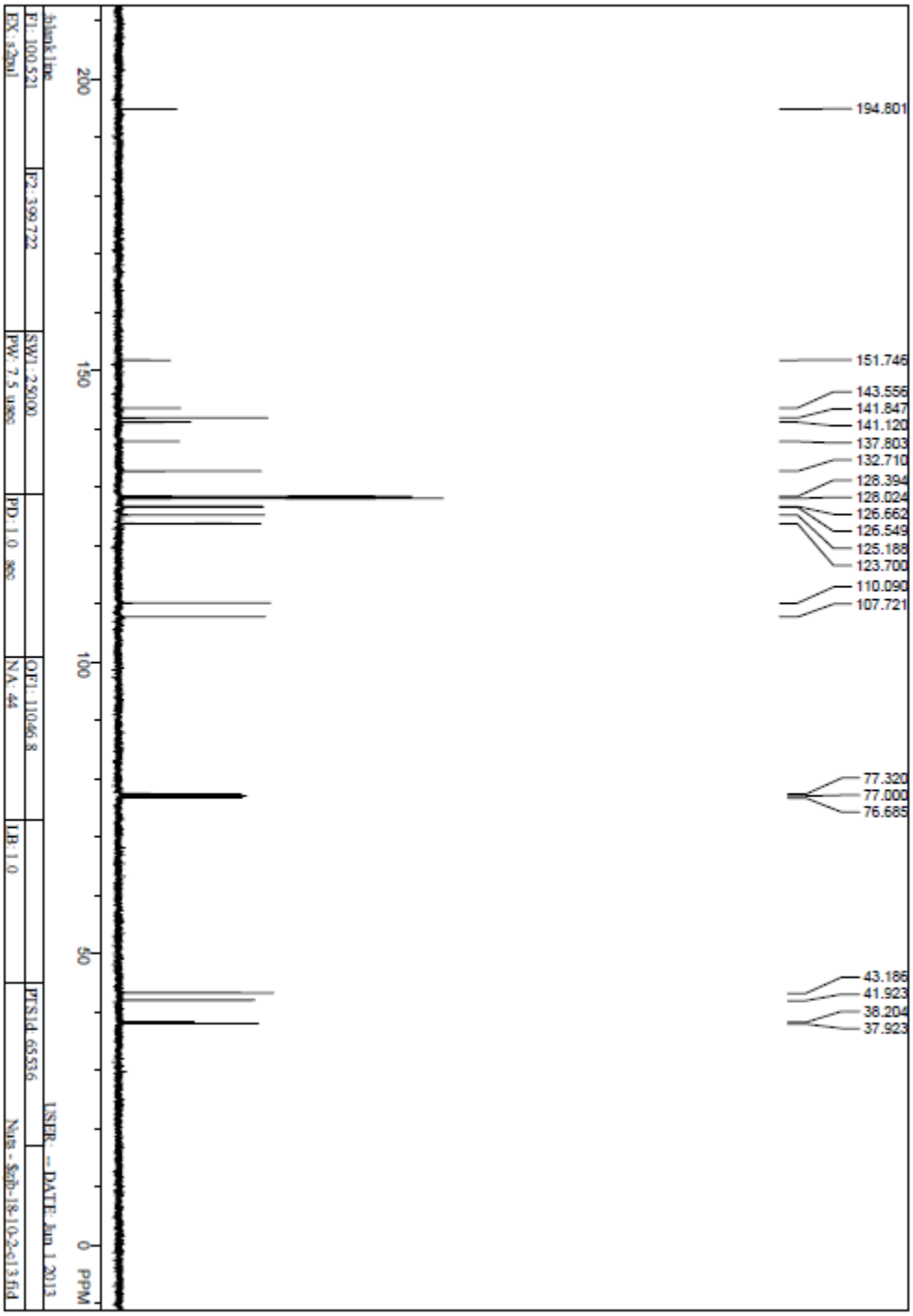
^{1}H NMR (400 M Hz in $CDCl_3$)

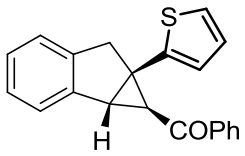




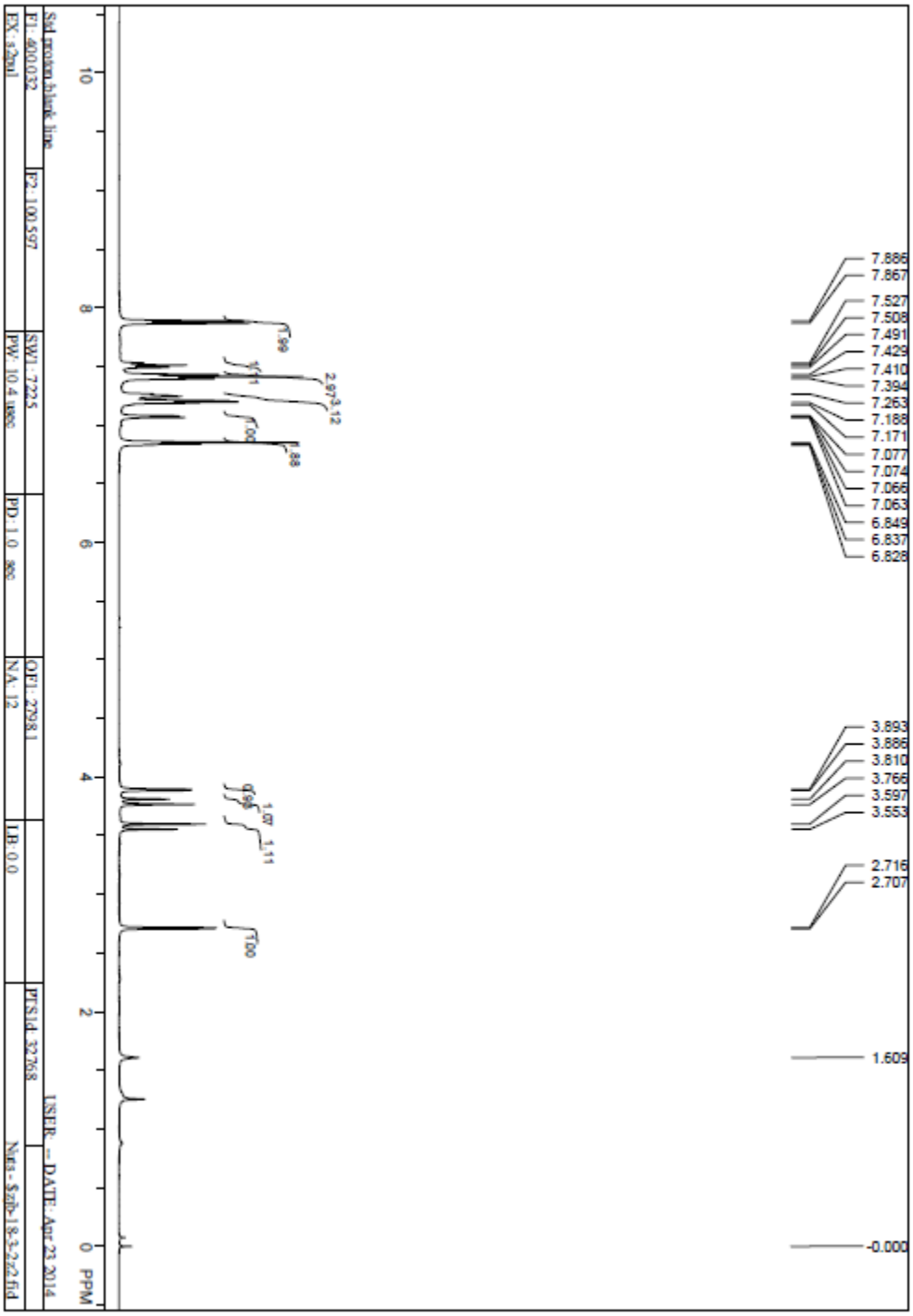
3p

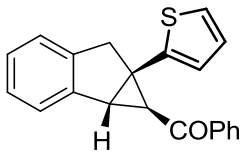
^{13}C NMR (100 M Hz in $CDCl_3$)



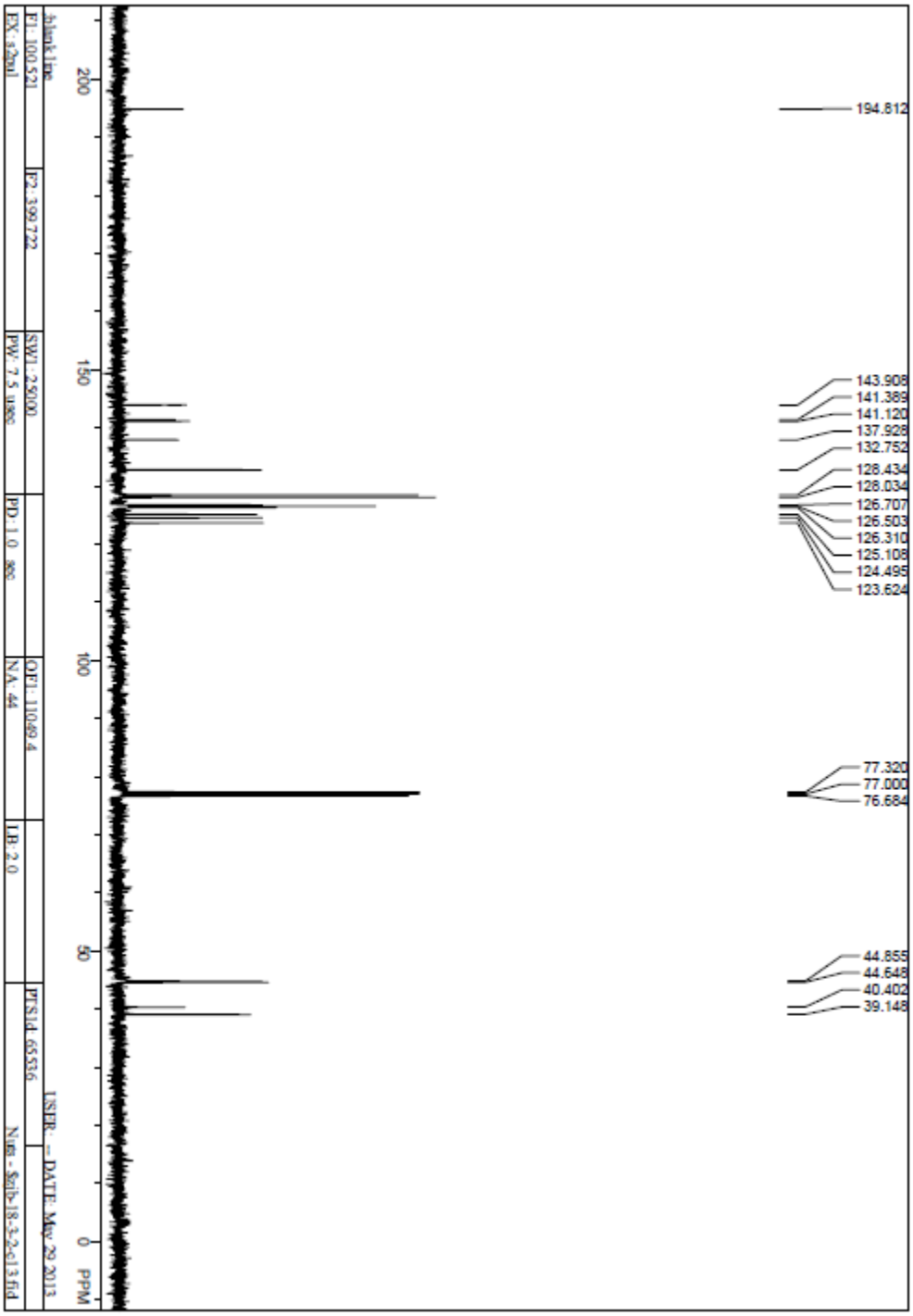


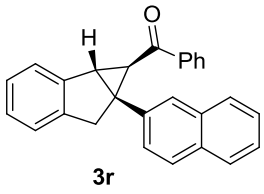
3q ^{1}H NMR (400 M Hz in $CDCl_3$)





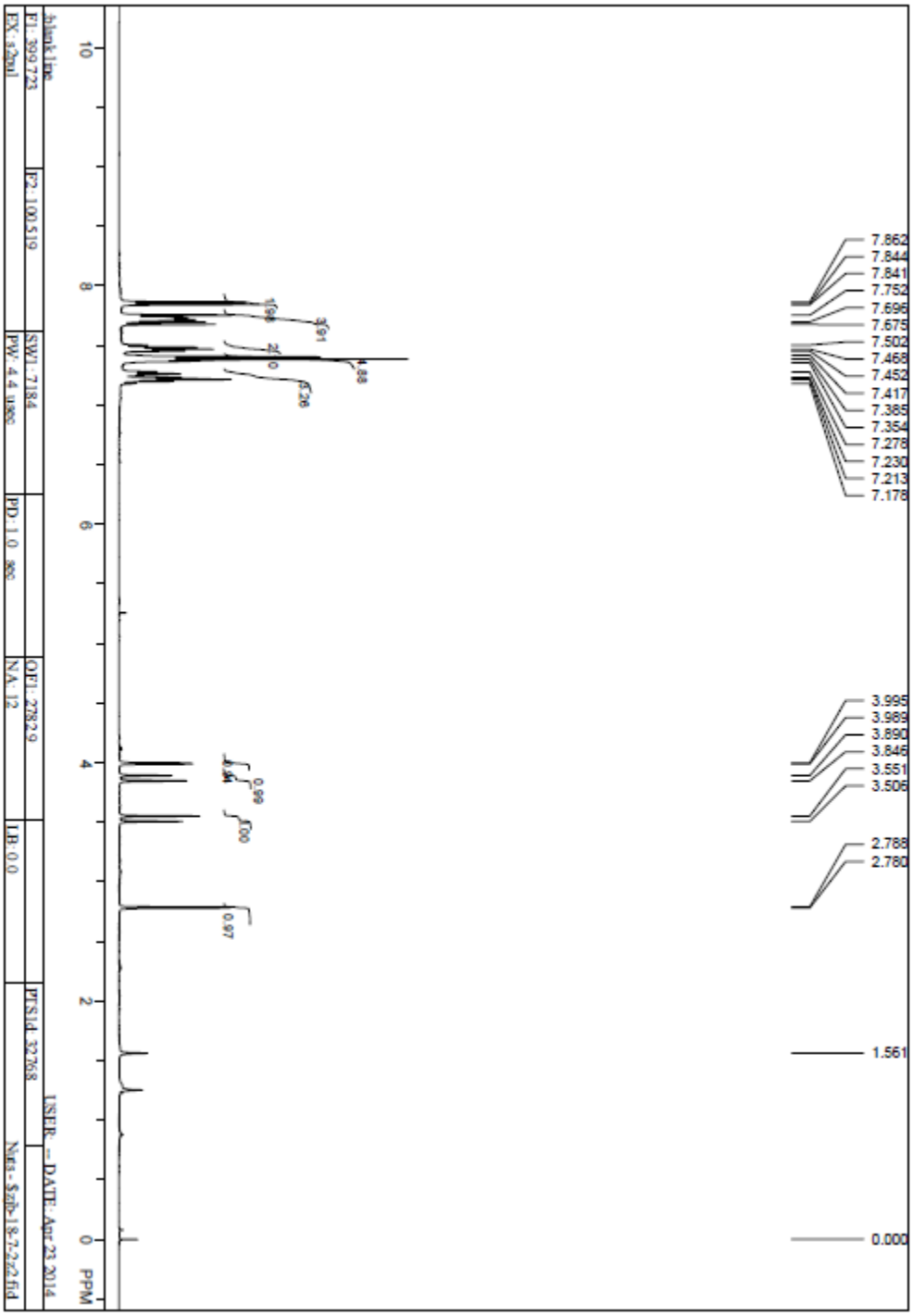
3q ^{13}C NMR (100 M Hz in $CDCl_3$)

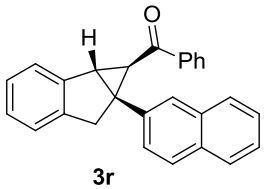




3r

^{1}H NMR (400 M Hz in $CDCl_3$)





3r

^{13}C NMR (100 M Hz in $CDCl_3$)