

A Zn(II) Catalyzed Synthesis of Piperidines from Propargyl Amines and Cyclopropanes

Supporting Information

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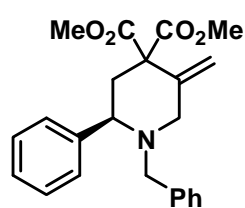
Experimental

General

Infrared spectra were obtained as thin films on NaCl plates using a Bruker Vector 33 FT-IR instrument. NMR experiments were performed on Varian Mercury 400, Varian Inova 600 and Inova 400 instruments and samples were obtained in CDCl₃ (referenced to 7.26 ppm for ¹H and 77.0 ppm for ¹³C). ¹⁹F spectra were externally referenced to neat trifluorotoluene (referenced to -63.9 ppm). Coupling constants (J) are in Hz. The multiplicities of the signals are described using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, sept = septuplet, m = multiplet, br = broad. High resolution mass spectra (HRMS) were obtained on a Finnigan MAT 8200 spectrometer at 70 eV. Optical rotations were recorded in cells of 10 cm path length using a Perkin-Elmer 241 digital polarimeter.

All reagents and solvents were used as purchased from Aldrich, Strem, Caledon or VWR. Reaction progress was followed by thin layer chromatography (TLC) (EM Science, silica gel 60 F₂₅₄) visualizing with UV light, and the plates developed using acidic anisaldehyde. Flash chromatography was performed using silica gel purchased from Silicycle Chemical Division Inc. (230-400 mesh).

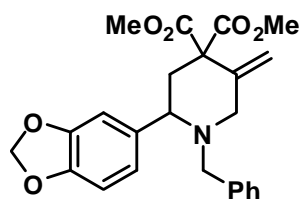
General Experimental Procedure A: 1,1-cyclopropane diester (1 equivalent), N-benzylprop-2-yn-1-amine (1.5 equivalent) and zinc(II) bistriflamide (0.1 equivalent) were dissolved in 3 ml benzene. A reflux condenser was attached and the vessel purged with Argon and the reaction was brought to reflux. Upon completion by TLC analysis a small amount of Li₂CO₃ was added to the reaction mixture which was then preabsorbed on silica and purified by flash chromatography (EtOAc/Hexanes) to yield the desired piperidines.



Piperidine 4a

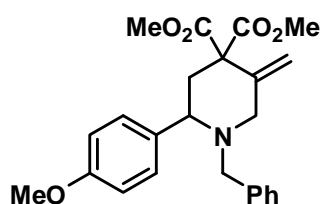
Piperidine **4a** was prepared using general experimental procedure A. Reagents employed: (R)-dimethyl 2-phenylcyclopropane-1,1-dicarboxylate (106 mg, 0.453 mmol, 98% *ee*), N-benzylprop-2-yn-1-amine (99 mg, 0.682 mmol) and zinc(II) bistriflamide (28 mg, 0.0447 mmol). Piperidine **4a** (165 mg, 0.435 mmol, 96%, 96% *ee*) was obtained as a pale yellow oil: *R*_f = 0.44, 20% EtOAc in hexanes; ¹H-NMR (400 MHz, CDCl₃): δ = 7.51-7.47 (m, 2H), 7.39-7.33 (m, 2H), 7.32-7.20 (m, 6H), 5.06 (d, *J* = 1.2 Hz, 1H), 4.79 (s, 1H), 3.86 (s, 3H), 3.81 (d, *J* = 13.2 Hz, 1H), 3.77 (s, 3H), 3.43 (d, *J* = 13.2 Hz, 1H), 3.30 (X of ABX system, *J* = 12.1, 2.3 Hz, 1H), 2.86 (d, *J* = 13.2 Hz, 1H), 2.83 (d, *J* = 13.2 Hz, 1H), 2.54 (A of ABX system, *J* = 13.7, 2.3 Hz, 1H), 2.44 (B of ABX system, *J* = 13.7, 12.1 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ = 170.2, 170.1, 142.7, 140.5, 138.7, 128.7, 128.5, 128.2, 127.5, 126.8, 113.4, 65.0, 61.3, 58.9, 57.5, 52.9, 52.6, 41.2 (note: 1 *sp*² carbon is missing presumably due to overlap); IR (thin film): 3086, 3063, 3029, 3004, 2952, 2838, 2795, 1735, 1654, 1604, 1495, 1453, 1436, 1373, 1361, 1321, 1307, 1262, 1244, 1213, 1139, 1116, 1086, 1061, 1030, 1019, 983, 914770, 741, 701; HRMS calc'd for C₂₃H₂₅NO₄ = 379.1784, found = 379.1774; [*α*]_D = -70.4 (*c* = 0.746, CH₂Cl₂).

The enantiomeric excess was determined to be 96% by chiral HPLC, Chiralcel OD-H, 250 x 4.6 mm², Diacel Chemical Industries; 96:4 Hexanes / *i*PrOH at 1.5 mL/min; 220 nm; The retention time for the enantiomers were *r*_t(+) = 3.89 min, *r*_t(-) = 5.03 min.



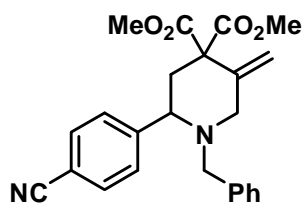
Piperidine 4b

Piperidine **4b** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-(benzo[d][1,3]dioxol-5-yl)cyclopropane-1,1-dicarboxylate (100 mg, 0.359 mmol), N-benzylprop-2-yn-1-amine (78 mg, 0.537 mmol), and zinc(II) bistriflamide (22 mg, 0.0352 mmol). Piperidine **4b** (144 mg, 0.340 mmol, 95%) was obtained as a colorless foam: *R*_f = 0.33, 20% EtOAc in hexanes; ¹H-NMR (400 MHz, CDCl₃): δ = 7.31-7.19 (m, 5H), 7.03 (d, *J* = 1.6 Hz, 1H), 6.87 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.76 (d, *J* = 8.0 Hz, 1H), 5.94 (AB, 2H), 5.03 (d, *J* = 0.8 Hz, 1H), 4.75 (s, 1H), 3.83 (s, 3H), 3.82 (d partially obscured by s at 3.83 ppm, *J* = 12.8 Hz, 1H), 3.76 (s, 3H), 3.38 (d, *J* = 12.8 Hz, 1H), 3.19 (X of ABX system, *J* = 12.2, 2.5 Hz, 1H), 2.80 (d, *J* = 13.2 Hz, 1H), 2.79 (d, *J* = 13.2 Hz, 1H), 2.49 (A of ABX system, *J* = 13.6, 2.5 Hz, 1H), 2.37 (B of ABX system, 13.6, 12.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ = 170.2, 170.1, 148.0, 146.9, 140.5, 138.7, 136.7, 128.5, 128.2, 126.8, 120.9, 113.4, 108.2, 107.5, 101.0, 64.8, 61.3, 58.8, 57.6, 52.9, 52.7, 41.3; IR (thin film): 3086, 3063, 3027, 3002, 2952, 2894, 2840, 2794, 1734, 1654, 1609, 1503, 1486, 1439, 1385, 1324, 1246, 1208, 1145, 1113, 1078, 1064, 1039, 1020, 983, 935, 915, 870, 812, 795, 740, 780, 700; HRMS calc'd for C₂₄H₂₅NO₆ = 423.1682, found = 423.1687.



Piperidine 4c

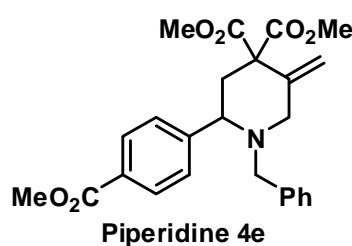
Piperidine **4c** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-(4-methoxyphenyl)cyclopropane-1,1-dicarboxylate (116 mg, 0.439 mmol), N-benzylprop-2-yn-1-amine (96 mg, 0.661 mmol), and zinc(II) bistriflamide (27 mg, 0.0432 mmol). Piperidine **4c** (171 mg, 0.418 mmol, 95%) was obtained as a pale yellow sticky solid: *R*_f = 0.33, 20% EtOAc in hexanes; ¹H-NMR (400 MHz, CDCl₃): δ = 7.40-7.36 and 6.90-6.86 (m, AA'BB', 4H), 7.30-7.18 (m, 5H) 7.03 (d, *J* = 1.2 Hz, 1H) 4.76 (s, 1H), 3.84 (s, 3H), 3.79 (s, 3H), 3.78 (d obscured by s at 3.79 and 3.76 ppm, *J* = 12.8, 1H), 3.76 (s, 3H), 3.39 (d, *J* = 12.8 Hz, 1H), 3.23 (X of ABX system, *J* = 11.9, 2.5 Hz, 1H), 2.82 (d, *J* = 12.4 Hz, 1H), 2.78 (d, *J* = 12.4 Hz, 1H), 2.50 (A of ABX system, *J* = 13.6, 2.5 Hz, 1H), 2.40 (B of ABX system, *J* = 13.6, 11.9 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ = 170.3, 170.2, 158.9, 140.6, 138.8, 134.7, 128.53, 128.47, 128.1, 126.8, 114.0, 113.3, 64.3, 61.3, 58.7, 57.6, 55.2, 52.9, 52.6, 41.2; IR (thin film): 3088, 3062, 3029, 3000, 2953, 2910, 2837, 2802, 1735, 1654, 1612, 1586, 1513, 1495, 1454, 1437, 1373, 1322, 1302, 1245, 1179, 1139, 1110, 1078, 1064, 1033, 915, 835, 773, 742, 699; HRMS calc'd for C₂₄H₂₇NO₅ = 409.1889, found = 409.1880.



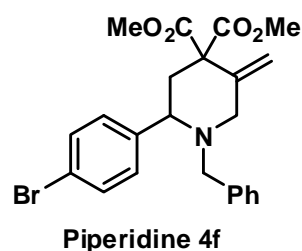
Piperidine 4d

Piperidine **4d** was prepared using general experimental procedure A with modifications to the equivalency of reagents. Reagents employed: dimethyl 2-(4-cyanophenyl)cyclopropane-1,1-dicarboxylate (150 mg, 0.579 mmol), N-benzylprop-2-yn-1-amine (252 mg, 1.735 mmol), and

zinc(II) bistriflamide (109 mg, 0.174 mmol). Piperidine **4d** (187 mg, 0.462 mmol, 80%) was obtained as a yellow sticky solid: R_f = 0.19, 20% EtOAc in hexanes; $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.67-7.59 (m, AA'BB', 4H), 7.32-7.21 (m, 5H), 5.07 (s, 1H), 4.80 (s, 1H), 3.85 (s, 3H), 3.76 (s, 3H), 3.66 (d, J = 13.4 Hz, 1H), 3.42 (d, J = 13.4 Hz, 1H), 3.37 (X of ABX system, J = 13.1, 2.5 Hz, 1H), 2.87 (d, J = 10.0 Hz, 1H), 2.83 (d, J = 10.0 Hz, 1H), 2.49 (A of ABX system, J = 13.3, 2.5 Hz, 1H), 2.33 (B of ABX, J = 13.3, 13.1 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 170.0, 169.8, 148.5, 139.8, 137.9, 132.6, 128.34, 128.32, 127.1, 118.7, 113.9, 111.5, 64.7, 61.0, 59.1, 57.3, 53.1, 52.8, 41.0 (note: 1 carbon is missing presumably due to overlap); IR (thin film): 3087, 3062, 3030, 3003, 2953, 2841, 2803, 2228, 1735, 1656, 1608, 1495, 1453, 1436, 1376, 1360, 1322, 1270, 1245, 1213, 1138, 1115, 1077, 1065, 1020, 916, 843, 785, 741, 699; HRMS calc'd for $\text{C}_{24}\text{H}_{24}\text{N}_2\text{O}_4$ = 404.1736, found = 404.1720.

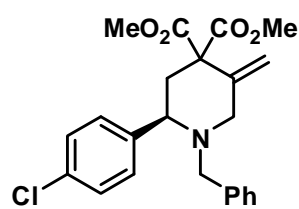


Piperidine **4e** was prepared using general experimental procedure A with modifications to the equivalency of reagents. Reagents employed: dimethyl 2-(4-(methoxycarbonyl)phenyl)cyclopropane-1,1-dicarboxylate (150 mg, 0.513 mmol), N-benzylprop-2-yn-1-amine (224 mg, 1.543 mmol), and zinc(II) bistriflamide (96 mg, 0.153 mmol). Piperidine **4e** (209 mg, 0.478 mmol, 93%) was obtained as a pale yellow sticky solid: R_f = 0.23, 20% EtOAc in hexanes; $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 8.05-8.01 and 7.59-7.55 (m, AA'BB', 4H), 7.31-7.20 (m, 5H), 5.06 (d, J = 1.2 Hz, 1H), 4.79 (s, 1H), 3.91 (s, 3H), 3.86 (s, 3H), 3.76 (s, 3H), 3.73 (d, J = 13.2 Hz, 1H), 3.42 (d, J = 13.2 Hz, 1H), 3.35 (X of ABX system, J = 11.8, 2.5 Hz, 1H), 2.88 (d, J = 13.2 Hz, 1H), 2.84 (d, J = 13.2 Hz, 1H), 2.51 (A of ABX system, J = 13.6, 2.5 Hz, 1H), 2.39 (B of ABX system, J = 13.6, 11.8 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 170.1, 170.0, 166.8, 148.2, 140.2, 138.3, 130.1, 129.5, 128.5, 128.3, 127.6, 127.0, 113.7, 64.8, 61.2, 59.1, 57.4, 53.0, 52.7, 52.1, 41.0; IR (thin film): 3088, 3063, 3029, 3001, 2953, 2842, 2803, 1726, 1657, 1611, 1495, 1452, 1435, 1416, 1374, 1358, 1309, 1277, 1244, 1214, 1196, 1177, 1139, 1113, 1077, 1065, 1019, 970, 915, 859, 820, 776, 741, 700; HRMS calc'd for $\text{C}_{25}\text{H}_{27}\text{NO}_6$ = 437.1838, found = 437.1826.



Piperidine **4f** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-(4-bromophenyl)cyclopropane-1,1-dicarboxylate (100 mg, 0.319 mmol), N-benzylprop-2-yn-1-amine (69 mg, 0.475 mmol), and zinc(II) bistriflamide (20 mg, 0.0320 mmol). Piperidine **4f** (139 mg, 0.303 mmol, 95%) was obtained as a pale yellow oil: R_f = 0.43, 20% EtOAc in hexanes; $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.49-7.46 and 7.38-7.34 (m, AA'BB', 4H), 7.31-7.20 (m, 5H), 5.05 (d, J = 1.2 Hz, 1H), 4.78 (s, 1H), 3.84 (s, 3H), 3.76 (s, 3H), 3.73 (d, J = 13.2, 1H), 3.40 (d, J = 13.2 Hz, 1H), 3.26 (X of ABX system, J = 12.1, 2.7 Hz, 1H), 2.82 (d, J = 13.2 Hz, 1H), 2.81 (d, J = 13.2 Hz, 1H), 2.49 (A of ABX system, J = 13.6, 2.7 Hz, 1H), 2.35 (B of ABX system, J = 13.6, 12.1 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 170.1, 170.0, 141.9, 140.2, 138.4, 131.9, 129.3, 128.4, 128.3, 127.0, 121.2, 113.6, 64.4, 61.2, 58.9, 57.4, 53.0, 52.7, 41.1; IR (thin film): 3086, 3062, 3028, 3001, 2951, 2795, 1730, 1655, 1590, 1484, 1450, 1434, 1406,

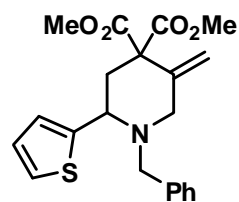
1372, 1322, 1268, 1242, 1212, 1180, 1136, 1115, 1063, 1009, 981, 912, 830, 782, 733, 698; HRMS calc'd for $C_{23}H_{24}BrNO_4$ = 457.0889, found = 457.0895.



Piperidine 4g

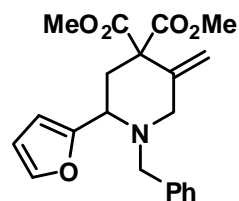
Piperidine **4g** was prepared using general experimental procedure A. Reagents employed: (R)-dimethyl 2-(4-chlorophenyl)cyclopropane-1,1-dicarboxylate (133 mg, 0.495 mmol, 98% *ee*), N-benzylprop-2-yn-1-amine (108 mg, 0.744 mmol), and zinc(II) bistriflamide (31 mg, 0.0495 mmol). Piperidine **4g** (194 mg, 0.469 mmol, 95%, 96% *ee*) was obtained as a pale yellow oil: R_f = 0.43, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.44-7.40 (m, 2H), 7.34-7.19 (m, 7H), 5.05 (d, J = 1.2 Hz, 1H), 4.77 (s, 1H), 3.84 (s, 3H), 3.76 (s, 3H), 3.73 (d, 12.8 Hz, 1H), 3.40 (d, J = 12.8 Hz, 1H), 3.27 (X of ABX system, J = 11.6, 2.4, 1H), 2.82 (d, J = 12.8, 1H), 2.81 (d, J = 12.8, 1H), 2.49 (A of ABX system, J = 13.7, 2.4 Hz, 1H) 2.36 (B of ABX system, J = 13.7, 11.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 170.1, 169.9, 141.3, 140.2, 138.3, 133.1, 128.8, 128.4, 128.2, 126.9, 113.6, 64.3, 61.1, 58.8, 57.4, 53.0, 52.7, 41.1 (note: 1 sp^2 carbon is missing presumably due to overlap); IR (thin film): 3088, 3064, 3031, 3003, 2954, 2904, 2801, 1735, 1656, 1598, 1494, 1453, 1436, 1410, 1374, 1360, 1321, 1268, 1244, 1213, 1182, 1140, 1117, 1078, 1065, 1015, 983, 914, 834, 801, 784, 736, 703; HRMS calc'd for $C_{23}H_{24}ClNO_4$ = 413.1394, found = 413.1390. $[\alpha]_D$ = -42.8 (c = 0.594, CH_2Cl_2).

The enantiomeric excess was determined to be 96% by chiral HPLC, Chiralcel OD-H, 250 x 4.6 mm², Diacel Chemical Industries; 98:2 Hexanes / *i*PrOH at 1.5 mL/min; 220 nm; The retention time for the enantiomers were r_t (+) = 4.46 min, r_t (-) = 5.36 min.



Piperidine 4h

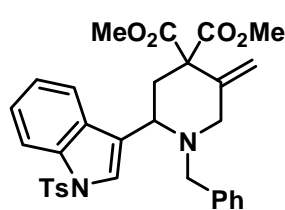
Piperidine **4h** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-(thiophen-2-yl)cyclopropane-1,1- (104 mg, 0.432 mmol), N-benzylprop-2-yn-1-amine (94 mg, 0.647 mmol), and zinc(II) bistriflamide (26 mg, 0.0416 mmol). Piperidine **4h** (153 mg, 0.397 mmol, 92%) was obtained as yellow sticky solid: R_f = 0.42, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.32-7.20 (m, 6H), 7.03 (dd, J = 3.6, 0.6 Hz, 1H), 6.94 (dd, J = 5.2, 3.6 Hz, 1H), 5.04 (d, J = 1.2 Hz, 1H), 4.81 (s, 1H), 3.87, (dd, J = 13.2 Hz, 1H), 3.84 (s, 3H), 3.78 (s, 3H), 3.76 (X of ABX system, 11.8, 3.0 Hz, 1H), 3.38 (d, J = 13.2 Hz, 1H), 2.95 (d, J = 13.2 Hz, 2H), 2.67 (A of ABX system, J = 13.8, 3.0 Hz, 1H), 2.56 (B of ABX system, J = 13.8, 11.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 170.1, 169.9, 146.7, 139.7, 138.6, 128.6, 128.2, 126.9, 126.2, 125.0, 113.9, 61.2, 59.7, 57.6, 57.0, 53.0, 52.7, 40.6 (note: 1 sp^2 carbon is missing presumably due to overlap); IR (thin film): 3087, 3065, 3029, 3004, 2953, 2841, 2796, 1735, 1654, 1605, 1495, 1453, 1436, 1373, 1321, 1265, 1245, 1181, 1114, 1077, 1064, 1030, 1017, 980, 915, 860, 834, 788, 739, 700; HRMS calc'd for $C_{21}H_{23}NO_4S$ = 385.1348, found = 385.1342.



Piperidine 4i

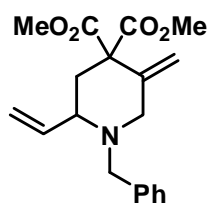
Piperidine **4i** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-(furan-2-yl)cyclopropane-1,1-dicarboxylate (100 mg, 0.446 mmol), N-benzylprop-2-yn-1-amine (194 mg, 1.336 mmol),

and zinc(II) bistriflamide (56 mg, 0.0894 mmol). Piperidine **4i** (139 mg, 0.376 mmol, 84%) was obtained as a pale yellow sticky solid: R_f = 0.36, 20% EtOAc in hexanes; $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.39 (dd, J = 2.0, 0.8 Hz, 1H), 7.30-7.19 (m, 5H), 6.35-6.31 (m, 2H), 5.04 (d, J = 0.8 Hz, 1H), 4.82 (s, 1H), 3.81 (s, 3H), 3.78 (s, 3H), 3.66 (d, J = 13.6 Hz, 1H), 3.65 (X of ABX system, J = 11.9, 3.3 Hz, 1H), 3.36 (d, J = 13.6 Hz, 1H), 3.08 (d, J = 13.2 Hz, 1H), 3.01 (d, J = 13.2 Hz, 1H), 2.68 (A of ABX system, J = 13.8, 11.9 Hz, 1H), 2.60 (B of ABX system, J = 13.8, 3.3 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 170.1, 169.9, 154.3, 141.8, 139.6, 138.3, 128.8, 128.1, 126.9, 113.8, 110.1, 107.6, 60.9, 57.0, 56.2, 52.9, 52.7, 36.0 (note: 1 sp^3 carbon is missing presumably due to overlap); IR (thin film): 3288, 3147, 3116, 3087, 3063, 3029, 3004, 2953, 2844, 2817, 1734, 1603, 1586, 1496, 1453, 1436, 1349, 1311, 1270, 1206, 1150, 1119, 1074, 1028, 1012, 970, 941, 916, 885, 865, 814, 739, 700, 599; HRMS calc'd for $\text{C}_{21}\text{H}_{23}\text{NO}_5$ = 369.1576, found = 369.1567.



Piperidine 4j

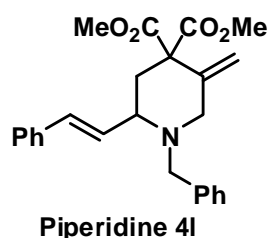
Piperidine **4j** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-(1-tosyl-1H-indol-3-yl)cyclopropane-1,1-dicarboxylate (150 mg, 0.351 mmol), N-benzylprop-2-yn-1-amine (76 mg, 0.523 mmol), and zinc(II) bistriflamide (22 mg, 0.0352 mmol). Piperidine **4j** (199 mg, 0.347 mmol, 99%) was obtained as a pale yellow solid: R_f = 0.22, 20% EtOAc in hexanes; $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.97 (d, J = 8.4 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.72-7.68 (m, 2H), 7.58 (s, 1H), 7.40-6.98 (m, 9H), 5.05 (s, 1H), 4.82 (s, 1H), 3.85 (s, 3H), 3.75 (s, 3H), 3.67 (d, J = 13.4 Hz, 1H), 3.66 (X of ABX system, J = 12.1, 2.7 Hz, 1H), 3.39 (d, J = 13.4, 1H), 2.89 (d, J = 12.0 Hz, 1H), 2.86 (d, J = 12.0 Hz, 1H), 2.69 (A of ABX system, J = 13.8, 12.1 Hz, 1H), 2.55 (B of ABX system, J = 13.8, 2.7 Hz, 1H), 2.25 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 170.3, 170.0, 144.9, 139.9, 138.5, 135.7, 135.0, 129.8, 129.3, 128.6, 128.2, 126.9, 126.7, 125.0, 124.0, 123.8, 123.2, 121.1, 114.0, 113.8, 61.3, 57.6, 57.3, 57.0, 53.1, 52.7, 38.2, 21.5; IR (thin film): 3107, 3086, 3064, 3030, 3004, 2955, 2846, 2797, 1735, 1654, 1598, 1495, 1447, 1372, 1269, 1243, 1213, 1189, 1175, 1132, 1119, 1100, 1076, 1019, 986, 915, 813, 748, 705, 664; HRMS calc'd for $\text{C}_{32}\text{H}_{32}\text{N}_2\text{O}_6\text{S}$ = 572.1981, found = 572.1993.



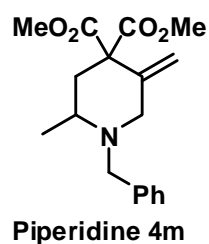
Piperidine 4k

Piperidine **4k** was prepared using general experimental procedure A. Reagents employed: dimethyl 2-vinylcyclopropane-1,1-dicarboxylate (117 mg, 0.635 mmol), N-benzylprop-2-yn-1-amine (138 mg, 0.950 mmol), and zinc(II) bistriflamide (40 mg, 0.0639 mmol). Piperidine **4k** (175 mg, 0.531 mmol, 84%) was obtained as a pale yellow oil: R_f = 0.29, 20% EtOAc in hexanes; $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.33-7.21 (m, 5H), 5.83 (ddd, J = 17.2, 10.0, 8.0 Hz, 1H), 5.28 (d, J = 17.2 Hz, 1H), 5.19 (dd, 10.0, 0.8 Hz, 1H), 4.98 (s, 1H), 4.73 (s, 1H), 4.05 (d, J = 13.6 Hz, 1H), 3.80 (s, 3H), 3.77 (s, 3H), 3.28 (d, J = 12.8 Hz, 1H), 3.03 (d, J = 13.6 Hz, 1H), 2.85 (ddd, X of ABX system, J = 11.3, 8.0, 2.7 Hz, 1H), 2.79 (d, J = 12.8 Hz, 1H), 2.42 (A of ABX system, J = 13.8, 2.7 Hz, 1H), 2.28 (B of ABX system, J = 13.8, 11.3 Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ = 170.25, 170.16, 140.3, 140.0, 138.5, 128.8, 128.2, 126.9, 117.5, 113.4, 62.7, 60.7, 58.3, 56.5, 52.9, 52.6, 38.1; IR (thin film): 3086, 3064, 3029, 3001, 2978, 2953, 2910, 2841, 2794, 1736, 1654, 1495, 1453, 1437, 1373, 1359, 1323,

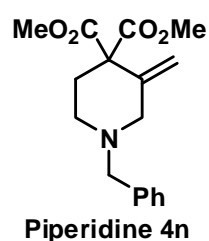
1262, 1242, 1204, 1162, 1129, 1069, 1029, 1022, 995, 918, 822, 784, 742, 700; HRMS calc'd for $C_{19}H_{23}NO_4$ = 329.1627, found = 329.1638.



Piperidine **4l** was prepared using general experimental procedure A. Reagents employed: (E)-dimethyl 2-styrylcyclopropane-1,1-dicarboxylate (120 mg, 0.461 mmol), N-benzylprop-2-yn-1-amine (100 mg, 0.689 mmol), and zinc(II) bistriflamide (29 mg, 0.0463 mmol). Piperidine **4l** (174 mg, 0.429 mmol, 93%) was obtained as a yellow gummy solid: R_f = 0.29, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.39-7.36 (m, 2H), 7.34-7.22 (m, 8H), 6.63 (d, J = 16.0 Hz, 1H), 6.21 (dd, J = 16.0, 8.0 Hz, 1H), 5.04 (s, 1H), 4.78 (m, 1H), 4.11 (d, J = 13.2 Hz, 1H), 3.84 (s, 3H), 3.78 (s, 3H), 3.35 (d, J = 13.2 Hz, 1H), 3.10 (d, J = 13.2 Hz, 1H), 3.03 (ddd, X of ABX system, J = 11.1, 8.0, 2.9 Hz, 1H), 2.84 (d, J = 13.2 Hz, 1H), 2.52 (A of ABX system, J = 13.5, 2.9 Hz, 1H), 2.39 (B of ABX system, J = 13.5, 11.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 170.3, 170.2, 140.0, 138.6, 136.6, 132.3, 131.4, 128.8, 128.5, 128.2, 127.6, 126.9, 126.3, 113.4, 62.2, 60.7, 58.8, 56.8, 52.9, 52.7, 38.4; IR (thin film): 3085, 3062, 3028, 3003, 2953, 2840, 2795, 1735, 1654, 1600, 1495, 1450, 1436, 1361, 1321, 1264, 1243, 1200, 1178, 1155, 1110, 1068, 1029, 1016, 971, 914, 784, 749, 697; HRMS calc'd for $C_{25}H_{27}NO_4$ = 405.1940, found = 405.1937.

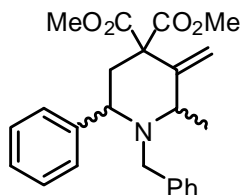


Piperidine **4m** was prepared using general experimental procedure A with modifications to the equivalency of reagents. Reagents employed: dimethyl 2-methylcyclopropane-1,1-dicarboxylate (106 mg, 0.616 mmol), N-benzylprop-2-yn-1-amine (268 mg, 1.846 mmol), and zinc(II) bistriflamide (77 mg, 0.123 mmol). Piperidine **4m** (115 mg, 0.362 mmol, 59%) was obtained as a yellow oil: R_f = 0.23, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.32-7.22 (m, 5H), 4.96 (s, 1H), 4.74 (s, 1H), 3.94 (d, J = 13.4 Hz, 1H), 3.78 (s, 6H), 3.23 (d, J = 7.8 Hz, 1H), 3.20 (d, J = 7.8 Hz, 1H), 2.88 (d, J = 13.4 Hz, 1H), 2.53 (ddq, X of ABX system, J = 11.3, 6.4, 2.7 Hz, 1H), 2.37 (A of ABX system, J = 13.6, 2.7 Hz, 1H), 2.18 (B of ABX system, J = 13.6, 11.3 Hz, 1H), 1.23 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 170.5, 170.4, 140.5, 138.8, 128.9, 128.2, 126.8, 113.2, 61.1, 56.7, 55.8, 53.4, 52.8, 52.6, 39.1, 19.8; IR (thin film): 3086, 3063, 3028, 2953, 2841, 2794, 1737, 1653, 1495, 1453, 1436, 1375, 1360, 1330, 1245, 1213, 1158, 1143, 1119, 1088, 1073, 1028, 963, 911, 783, 741, 668; HRMS calc'd for $C_{18}H_{23}NO_4$ = 317.1627, found = 317.1624.



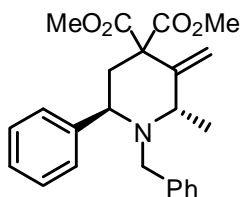
Piperidine **4n** was prepared using general experimental procedure A with modifications to the equivalency of reagents. Toluene was also used in replace of benzene. Reagents employed: dimethyl cyclopropane-1,1-dicarboxylate (108 mg, 0.683 mmol), N-benzylprop-2-yn-1-amine (149 mg, 1.026 mmol), and zinc(II) bistriflamide (64 mg, 0.102 mmol). Piperidine **4n** (151 mg, 0.498 mmol, 73%) was obtained as a yellow oil: R_f = 0.20, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.32-7.23 (m, 5H), 5.10 (s, 1H), 4.76 (s, 1H), 3.78 (s, 6H), 3.50 (s, 2H), 3.09 (s, 2H), 2.50-2.44 (m, 2H), 2.37-2.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 170.4, 140.8, 137.8, 129.0, 128.2, 127.1, 113.5, 62.2, 60.9, 58.9, 52.7, 49.8,

31.9 ; IR (thin film): 3088, 3064, 3029, 3000, 2953, 2909, 2840, 2801, 2757, 2718, 1735, 1653, 1496, 1455, 1437, 1364, 1340, 1286, 1259, 1213, 1194, 1179, 1137, 1105, 1076, 1059, 1029, 1015, 957, 918, 818, 780, 742, 700; HRMS calc'd for $C_{17}H_{21}NO_4$ = 303.1471, found = 303.1479.



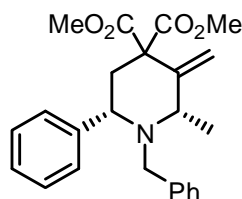
Piperidine 7
(equimolar mix of 4 isomers)

Piperidine **7**(equimolar mix of 4 isomers) was prepared using general experimental procedure A with modifications to the equivalency of reagents. Reagents employed: dimethyl 2-phenylcyclopropane-1,1-dicarboxylate (100 mg, 0.427 mmol), N-benzylbut-3-yn-2-amine (204 mg, 1.281 mmol), and zinc(II) bistriflamide (27 mg, 0.0432 mmol). Piperidine **7**(equimolar mix of 4 isomers) (165 mg, 0.419 mmol, 98%) was obtained as a pale yellow oil: R_f = 0.40, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): Spectral data is consistent with a 1:1 mixture of **7**(2*S*,6*R*) and **7**(2*S*,6*S*) (*vide infra*).



Piperidine 7(2*S*,6*R*)

Piperidine **7**(2*S*,6*R*) was prepared using general experimental procedure A with modifications to the equivalency of reagents. Reagents employed: (*S*)-dimethyl 2-phenylcyclopropane-1,1-dicarboxylate (48 mg, 0.205 mmol, 95% *ee*), (*S*)-N-benzylbut-3-yn-2-amine (66 mg, 0.414 mmol), and zinc(II) bistriflamide (17 mg, 0.0272 mmol). Piperidine **7**(2*S*,6*R*) (78 mg, 0.198 mmol, 96%, diastereomeric purity >97%) was obtained as a very pale yellow oil: R_f = 0.41, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.52-7.48 (m, 2H), 7.36-7.18 (m, 8H), 5.12 (s, 1H), 4.97 (s, 1H), 4.21 (X of ABX system, J = 10.7, 3.7 Hz, 1H), 3.81 (s, 3H), 3.74 (s, 3H), 3.63 (q, J = 7.2 Hz, 1H), 3.55 (d, J = 14.0 Hz, 1H), 3.34 (d, J = 14.0 Hz, 1H), 2.76 (A of ABX system, J = 13.5, 3.7 Hz, 1H), 2.57 (B of ABX system, J = 13.5, 10.7 Hz, 1H), 1.21 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 171.1, 171.0, 143.2, 142.3, 139.6, 128.4, 128.2, 128.1, 127.7, 127.1, 126.6, 114.7, 60.0, 56.8, 55.2, 52.7, 51.3, 35.4, 15.8 (note: 1 sp^3 carbon is missing presumably due to overlap); IR (thin film): 3086, 3062, 3029, 2979, 2951, 2841, 1731, 1645, 1603, 1494, 1451, 1434, 1376, 1267, 1243, 1211, 1148, 1121, 1088, 1076, 1062, 1029, 1009, 969, 912, 862, 818, 736, 699; HRMS calc'd for $C_{24}H_{27}NO_4$ = 393.1940, found = 393.1923. $[\alpha]_D^{25}$ = +33.9 (c = 0.327, CH_2Cl_2).

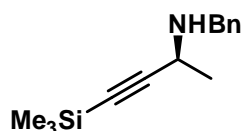


Piperidine 7(2*S*,6*S*)

Piperidine **7**(2*S*,6*S*) was prepared using general experimental procedure A. Reagents employed: (*R*)-dimethyl 2-phenylcyclopropane-1,1-dicarboxylate (60 mg, 0.256 mmol), (*S*)-N-benzylbut-3-yn-2-amine (61 mg, 0.383 mmol, 98% *ee*), and zinc(II) bistriflamide (16 mg, 0.0256 mmol). Piperidine **7**(2*S*,6*S*) (96 mg, 0.244 mmol, 95%, diastereomeric purity >97%) was obtained as a very pale yellow oil: R_f = 0.39, 20% EtOAc in hexanes; 1H -NMR (400 MHz, $CDCl_3$): δ = 7.53-7.49 (m, 2H), 7.34-7.29 (m, 2H), 7.26-7.19 (m, 5H), 7.17-7.12 (m, 1H), 5.23 (s, 1H), 5.05 (s, 1H), 3.85-3.77 (m obscured by s at 3.83 and 3.79 ppm, 1 H), 3.83 (s, 3H), 3.79 (s, 3H), 3.66 (d, J = 15.6 Hz, 1H), 3.50 (q, J = 6.8 Hz, 1H), 3.38 (d, J = 15.6 Hz, 1H), 2.60 (A of ABX system, J = 14.0, 12.9 Hz, 1H), 2.53 (B of ABX

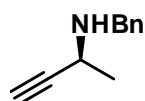
system, $J = 14.0, 2.3$ Hz, 1H), 1.13 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 171.0, 170.7, 143.4, 143.3, 141.4, 128.3, 127.8, 127.7, 127.2, 126.2, 113.9, 109.7, 63.5, 61.0, 60.0, 54.8, 52.9, 52.8, 37.8, 21.3$; IR (thin film): 3087, 3062, 3028, 2981, 2952, 2881, 2844, 2808, 1735, 1644, 1603, 1495, 1454, 1435, 1370, 1314, 1244, 1208, 1151, 1124, 1094, 1066, 1029, 971, 915, 765, 733, 700; HRMS calc'd for $\text{C}_{24}\text{H}_{27}\text{NO}_4 = 393.1940$, found = 393.1927. $[\alpha]_{\text{D}} = -14.2$ ($c = 0.562$, CH_2Cl_2).

(R)-4-(trimethylsilyl)but-3-yn-2-yl methanesulfonate was prepared according to the procedure of Marshall and Chobanian.¹



(R)-4-(trimethylsilyl)but-3-yn-2-yl methanesulfonate (463 mg, 2.101 mmol) was dissolved in benzyl amine (1.30 ml, 11.913 mmol) and the reaction stirred until TLC showed complete consumption of the starting material.

The reaction was poured into an aqueous NaHCO_3 solution and extracted 4 times with ethyl acetate. The combined organics were washed with water then brine, and dried with MgSO_4 . The solvent was removed under reduced pressure to produce a crude oil that was purified by column chromatography on silica gel (ethyl acetate / hexanes as eluent) to yield (S)-N-benzyl-4-(trimethylsilyl)but-3-yn-2-amine (0.446 g, 1.927 mmol, 92%) as a colorless oil: $R_f = 0.47$, 20% EtOAc in hexanes; ^1H -NMR (400 MHz, CDCl_3): $\delta = 7.37\text{--}7.30$ (m, 4H), 7.28-7.23 (m, 1H), 4.00 (d, $J = 12.8$ Hz, 1H), 3.80 (d, $J = 12.8$ Hz, 1H), 3.49 (q, $J = 6.8$ Hz, 1H), 1.37-1.34 (br.s obscured by d at 1.35 ppm, 1H) 1.35 (d, $J = 6.8$ Hz, 3H), 0.19 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 140.0, 128.40, 128.39, 127.0, 108.6, 87.1, 51.4, 45.1, 22.2, 0.14$; IR (thin film): 3086, 3065, 3028, 2959, 2933, 2897, 2158, 1496, 1454, 1370, 1303, 1250, 1137, 1113, 1070, 884, 841, 760, 733, 698; HRMS calc'd for $\text{C}_{14}\text{H}_{21}\text{NSi} = 231.1443$, found = 231.1438.



(S)-N-benzyl-4-(trimethylsilyl)but-3-yn-2-amine (230 mg, 0.994 mmol) was dissolved in MeOH (3.0 ml). K_2CO_3 (206 mg, 1.490 mmol) was then added and the reaction stirred until TLC showed complete consumption of the starting material.

The reaction was poured into water and extracted 4 times with ethyl acetate. The combined organics were washed with water then brine, and dried with MgSO_4 . The solvent was removed under reduced pressure to yield (S)-N-benzylbut-3-yn-2-amine (144 mg, 0.992 mmol, 100%, >99% ee) as a light yellow oil: $R_f = 0.32$, 20% EtOAc in hexanes; ^1H -NMR (400 MHz, CDCl_3): $\delta = 7.38\text{--}7.30$ (m, 4H), 7.28-7.23 (m, 1H), 4.02 (d, $J = 12.8$ Hz, 1H), 3.81 (d, $J = 12.8$ Hz, 1H), 3.50 (dq, $J = 7.2, 2.0$ Hz, 1H), 2.32 (d, $J = 2.0$ Hz, 1H), 1.44-1.38 (br.s, 1H), 1.39 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 139.9, 128.40, 128.37, 127.0, 86.3, 70.7, 51.4, 44.3, 22.3$; IR (thin film): 3292, 3087, 3064, 3029, 2974, 2933, 2843, 1496, 1453, 1372, 1308, 1137, 1067, 1028, 980, 909, 838, 734, 698; HRMS calc'd for $\text{C}_{11}\text{H}_{13}\text{N} = 159.1048$, found = 159.1053; $[\alpha]_{\text{D}} = -118.5$ ($c = 5.462$, CH_2Cl_2).

The enantiomeric excess was determined to be >99% by chiral HPLC, Chiralcel OD-H, 250 x 4.6 mm², Diacel Chemical Industries; 98:2 Hexanes / *i*PrOH at 1.0 mL/min; 220 nm; The retention time for the enantiomers with the nitrogen protected with an acetyl group were $r_t(+)$ = 16.59 min, $r_t(-)$ = 17.89 min.

Zn(NTf₂)₂ ZnO (292 mg, 3.589 mmol) and trifluoromethane-sulfonimide (2.020 g, 7.185 mmol) were added to distilled H₂O (15 ml) and the reaction brought to reflux. After 22 hours the solvent was removed under reduced pressure to yield zinc(II) bistriflamide (2.570 g, presumably as the hydrate) as an off-white crystalline solid: ¹⁹F-NMR (400 MHz, Acetonitrile-d₃): δ = -79.635

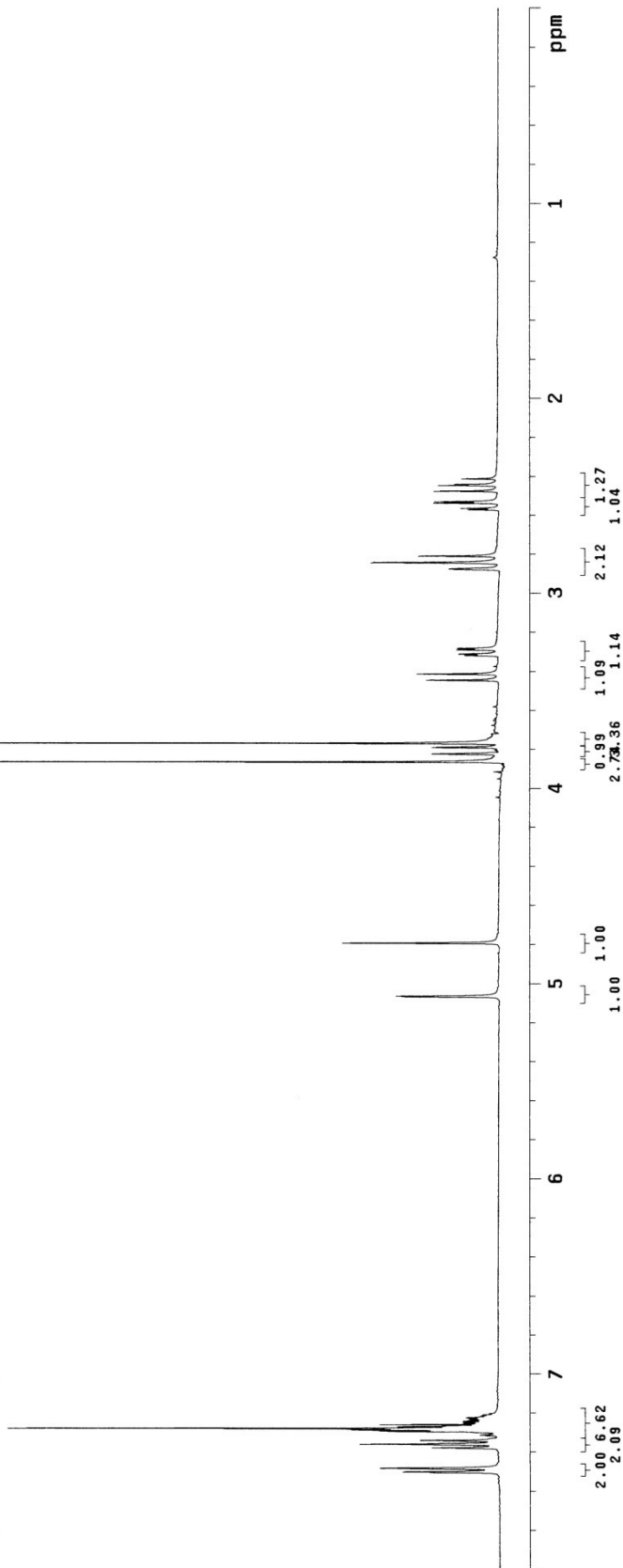
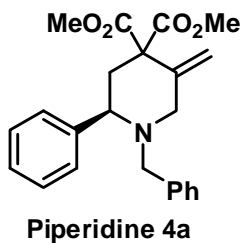
References:

1 Marshall, J. A.; Chobanian, H. *Organic Syntheses*, **2005**, 82, 43.

ABL-14-15-CARBON
STANDARD 1H OBSERVE

exp3 s2pul

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ACQUISITION	alfa	20.000	
SW	6402.0	FLAGS	
at	4.000	il	n
np	51216	in	n
fb	not used	dp	y
bs	16	hs	nn
d1	1.000	fn	not used
nt	8	fn	not used
ct	8	DISPLAY	
TRANSMITTER		SP	-0.6
tn	H1	WD	3204.3
sfrq	400.083	rf1	3702.9
tof	424.3	rfp	2904.6
tpwr	58	rp	-168.5
pw	6.750	lp	-69.1
DECOUPLER		C13	WC
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dof	0	VS	0
dm	nnn	VS	38
dmm	C	TH	54
dpwr	44	AI	cdc
dmf	18500	PH	

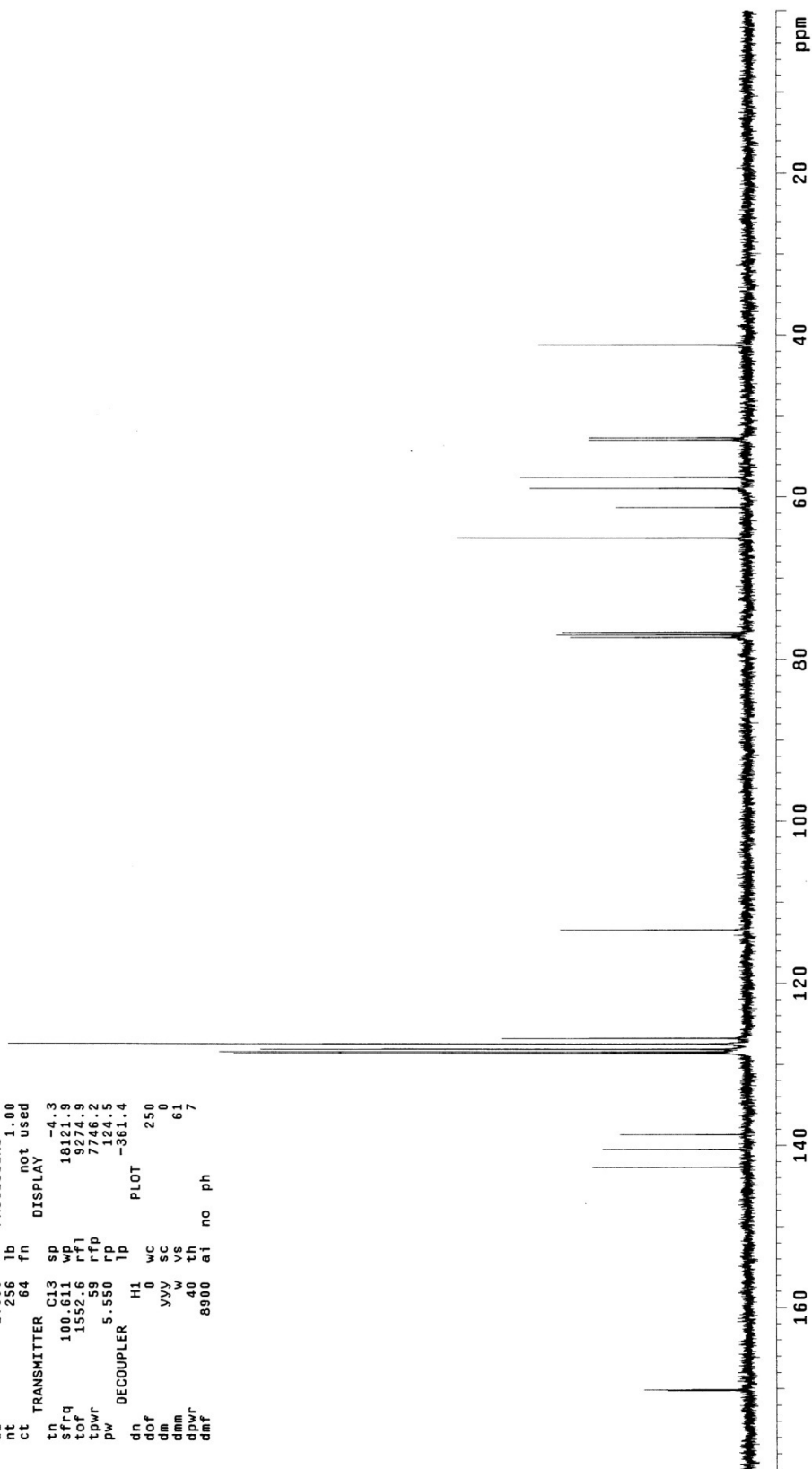
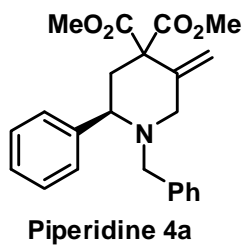


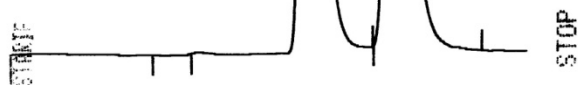
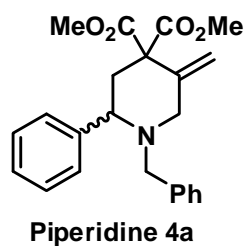
ABL-14-15-CARBON
STANDARD 1H OBSERVE

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solvent cdc13 gain not used
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ACQUISITION alfa 20.000
sw 25125.6
at 1.199 fl n
np 60270 tn n
fb 13800 dp y
bs 64 hs mn
dl 1.000 lb 1.00
nt 256 fn not used
ct TRANSMITTER C13 DISPLAY -4.3
tn C13 sp 18121.8
sfrq 100.611 wpr 19271.8
tot 1552.6 rfl 7146.2
tpwr 5.59 rfp 124.5
pw DECOUPLER 1p -361.4
dn H1 PLOT
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dm yyv sc 0
dnn w vs 61
dpwr 40 th 7
dmf 8900 al no ph

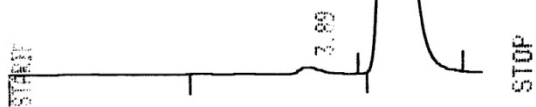
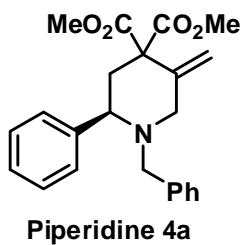
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3.89	3.89	136730	PB	0.315	49.182
5.03	5.03	141280	PB	0.376	50.818

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 MUL FACTOR= 1.0000E+00



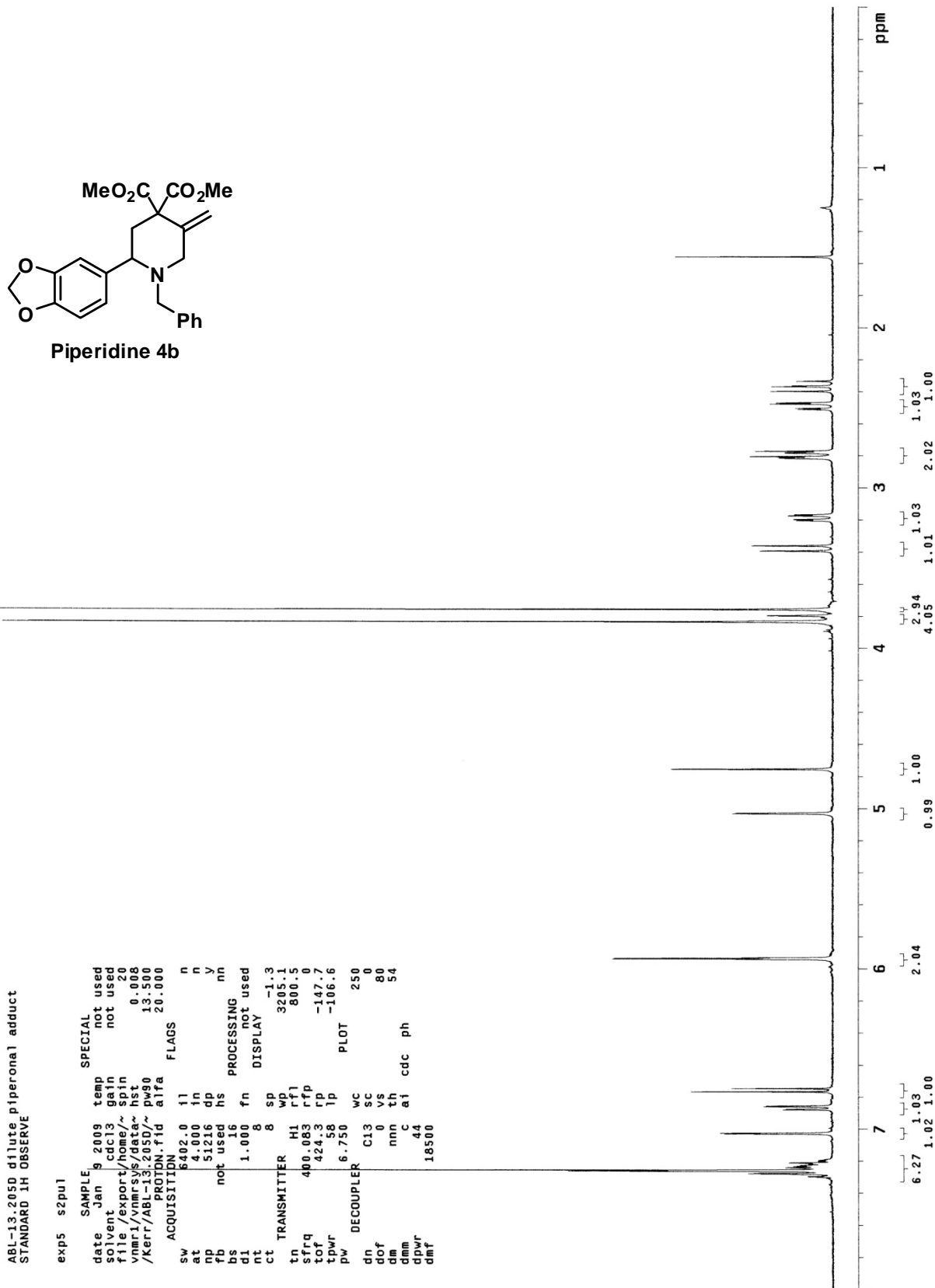
AREA%	RT	AREA	TYPE	AR/NT	AREA%
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RUN # 7
 TOTAL AREA= 273390
 MUL FACTOR= 1.0000E+00

ABL-13.205D dilute piperonal adduct
STANDARD 1H OBSERVE

exp5 s2pul

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/Kerr/ABL-13.205D/~ pw90		pw90	13.500
PROTON.fid		alpha	20.000
ACQUISITION			
sw	6402.0	l1	n
at	4.000	in	n
np	51216	dp	y
fb	not used	hs	nn
bs	16		
di	1.000	fn	not used
nt	8		
ct	8	sp	-1.3
TRANSMITTER			
tn	H1	wp	3205.1
sfrq	400.083	rfl	800.5
tof	424.3	rpf	0
tpwr	58	lp	-147.7
pw	6.750		-106.6
DECOUPLER			
dn	C13	sc	250
dof	0	vs	0
dm	nnn	th	80
dmm	c	al	54
dpwr	44		
daf	18500		

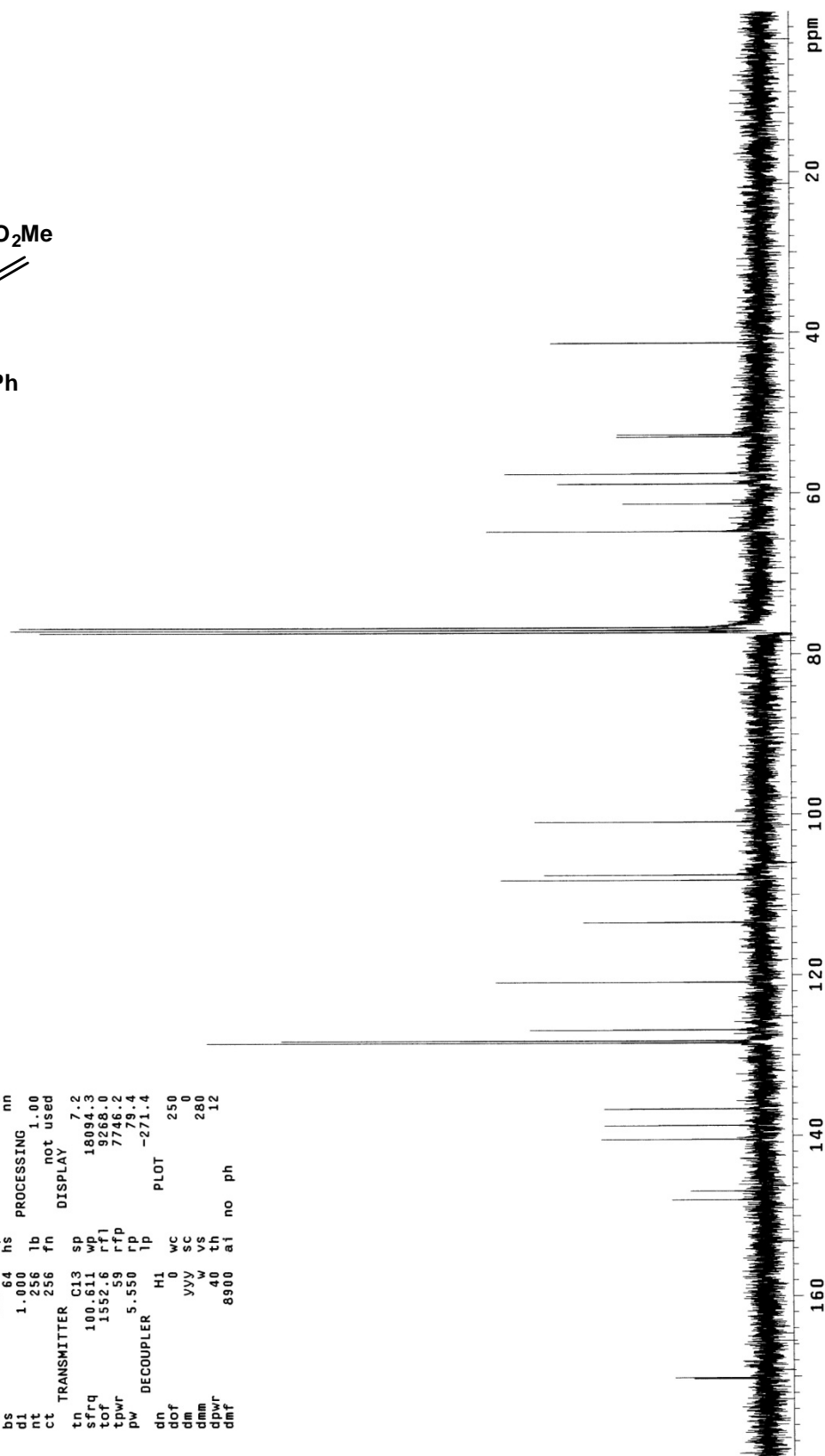
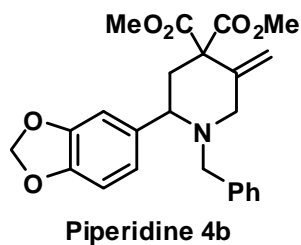


ABU-13.205C Carbon of piperonal cyclo
STANDARD 1H OBSERVE

```

exp1 s2pul
date Jan 8 2009 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/walkup/autoc~ hst 0.008
08.01.09/0206 pw90 11.100
ACQUISITION alfa 20.000
sw 25125.6
at 1.199 11 n
np 60270 1n n
fb 13800 dp y
bs 64 hs PROCESSING
d1 1.000 1b 1.00
nt 256 1b not used
ct 256 1n DISPLAY 7.2
tn TRANSMITTER C13 sp 7.2
sfrq 100.611 wp1 18094.3
tor 1552.6 rfp 19284.0
tpwr 7748.2 rfp 7748.2
pw 5.550 1p -271.4
DECOUPLER H1 PLOT
dn 0 wc 250
dof 0 yyv sc 0
dm 40 vs 280
dpm 40 th 12
dmf 8900 at no ph

```

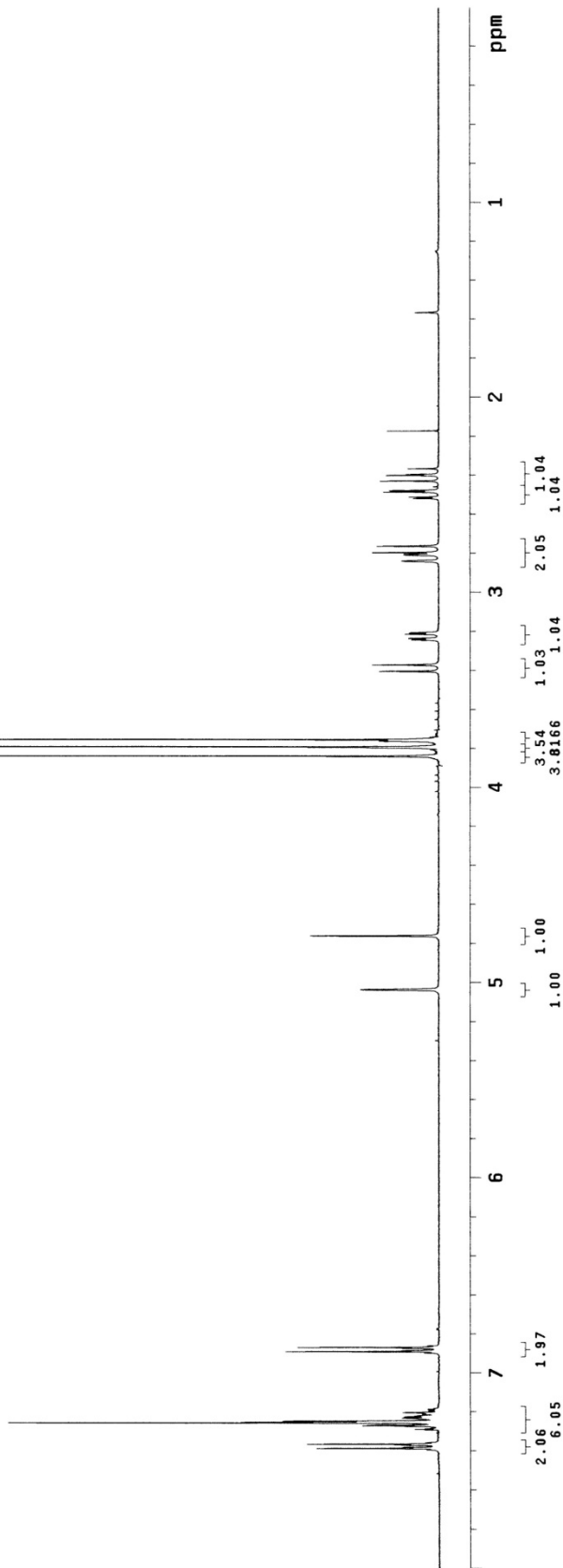
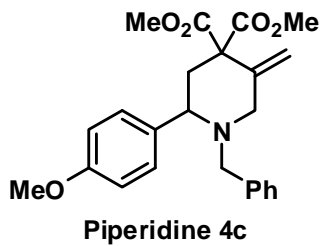


TPL-P-163-a0
STANDARD 1H OBSERVE

```

exp1 s2pu1
SAMPLE
date Jan 9 2009 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin 20
vnmr1/wakup/auto/~ hst 0.008
09-01-09/0215 pw90 13.500
ACQUISITION alfa 26.000
SW 6402.0
at 4.000 il n
np 51216 in n
fb not used dp y
bs 16 hs
d1 1.000
nt 8 fn not used
ct 8 DISPLAY
TRANSMITTER H1 sp 1.7
tn H1 wp 3200.8
sfrq 400.083 rf1 800.5
tof 424.3 rfp 0
tpwr 58 rp -152.2
pw 6.750 lp -96.1
DECOUPLER C13 wc 250
dn 0 sc 0
dof 0 sc 0
dm nnn vs 55
dmm c th 54
dpwr 44 ai cdc ph
dmf 18500

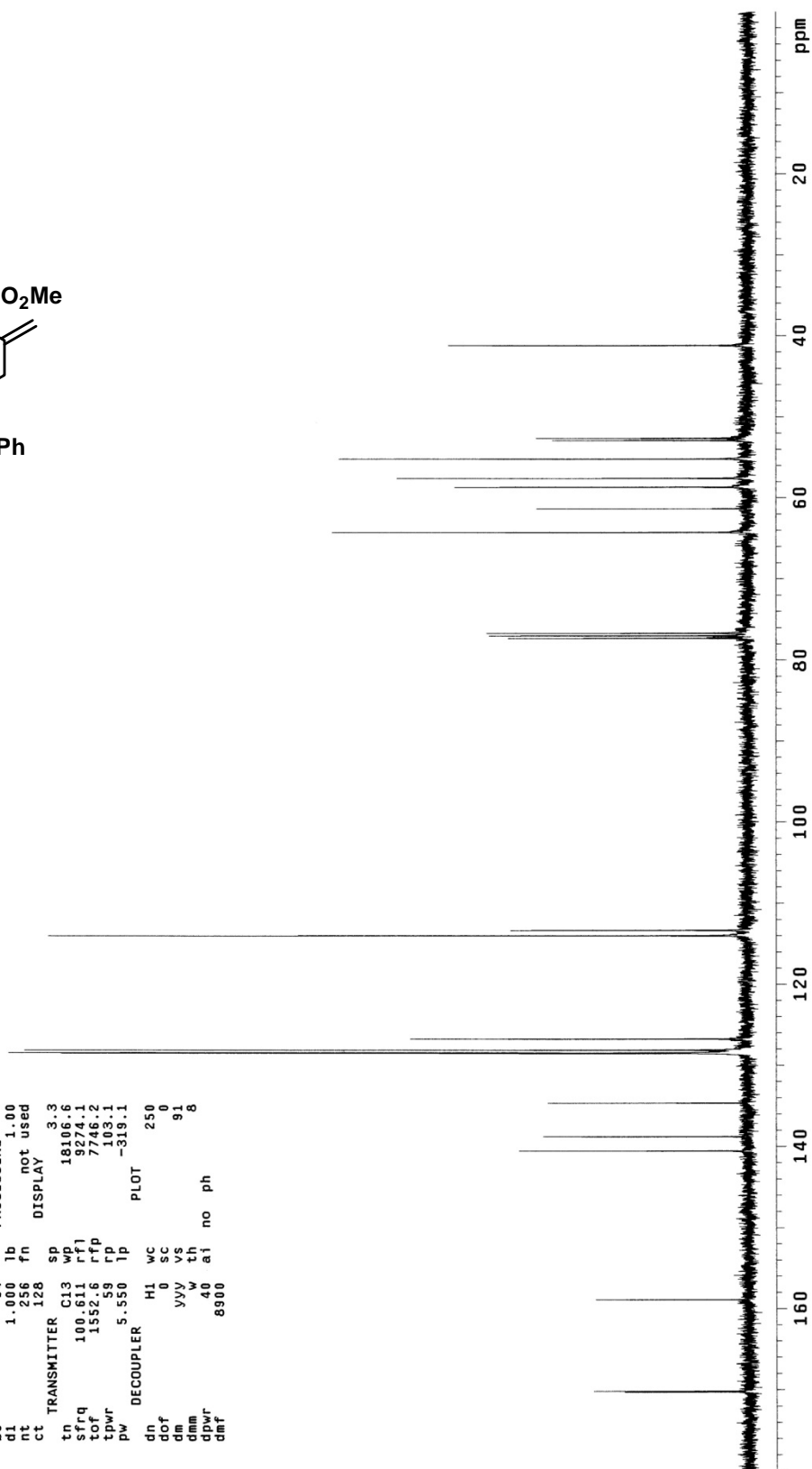
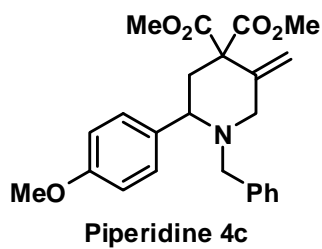
```



TPL-P-163-carb
STANDARD 1H OBSERVE

exp1 s2pu1

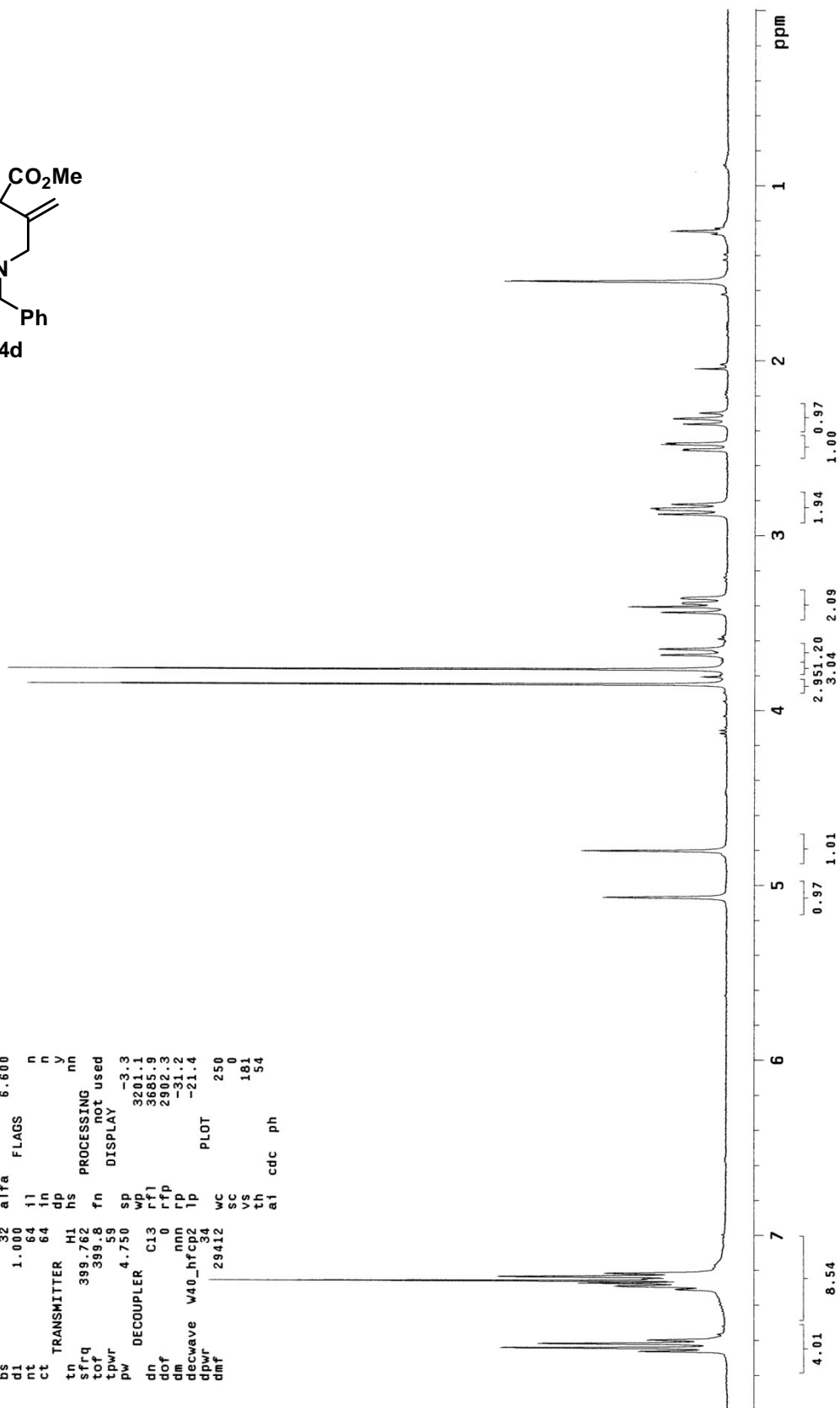
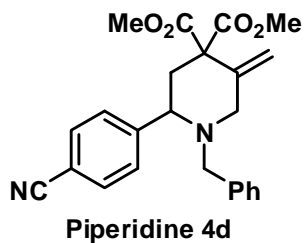
SAMPLE		SPECIAL	
date	Jan 9 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
nmr1/vnmr	sys/data/~	hst	0.008
/Kerr/TPL-P-163-carb	pw90	11.100	
rb/CARBON.fid	alfa	20.000	
ACQUISITION		FLAGS	
sw	25125.6	il	n
at	1.199	in	n
np	60270	dp	y
fb	13800	hs	nn
bs	64		
di	1.000	lb	1.00
nt	236	fn	not used
ct	128		
TRANSMITTER C13		SP	
tn	18106.6	wp	3.3
sfreq	100.611	rf1	18106.6
tofr	1552.6	rf2	924.1
tpwr	5.59	rfp	7746.2
pw	5.350	lp	103.1
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yvy	vs	91
dmm	w	th	8
dpwr	40	ai	no
dmf	8900		ph



TPL-Q-53-H

exp1 PROTON

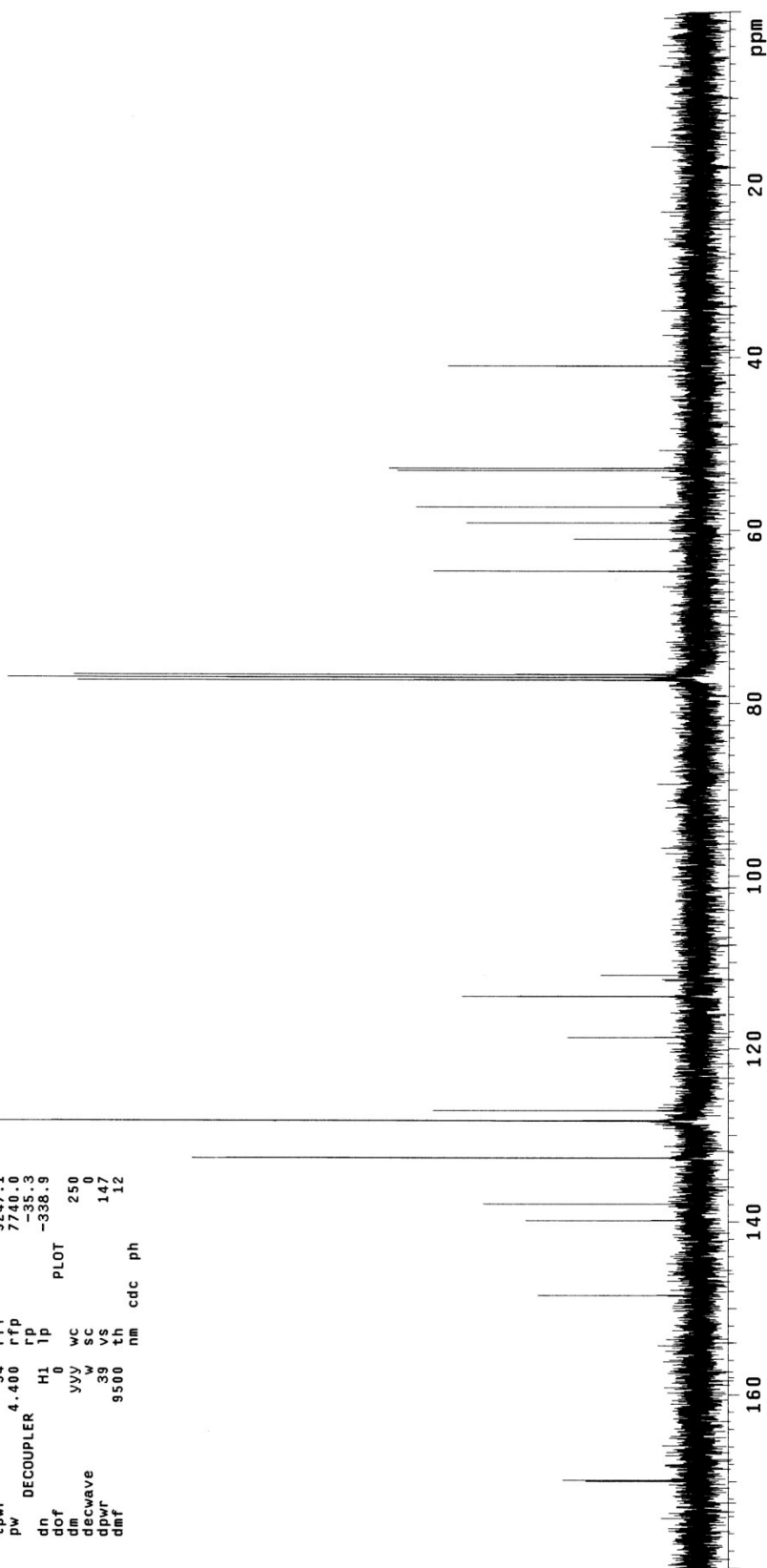
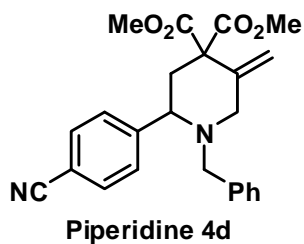
SAMPLE		PRESATURATION	
date	Feb 11 2009	satmode	n
solvent	cdcl3	wet	n
file	cdcl3	exp	26.0
ACQUISITION		SPECIAL	
sw	6395.9	gain	not used
at	2.562	spn	20
np	32768	hst	0.008
fb	4000	pw90	9.500
bs	32	alfa	6.600
d1	1.000	il	FLAGS
nt	64	il	n
ct	64	in	n
tn	399.762	hs	nn
sfrq	399.762	fn	not used
tof	399.8	fn	not used
tpwr	59	sp	-3.3
pw	4.750	wp	3201.1
dn	DECOUPLER	rf1	3685.9
dof	C13	rfp	2902.3
dm	nnn	rp	-31.2
decwave	w40_hfcp2	lp	-21.4
dpwr	34	PLOT	
dmf	29412	wc	250
		sc	0
		vs	181
		th	54
		ai	cdc ph



TPL-Q-53-carb

exp1 CARBON

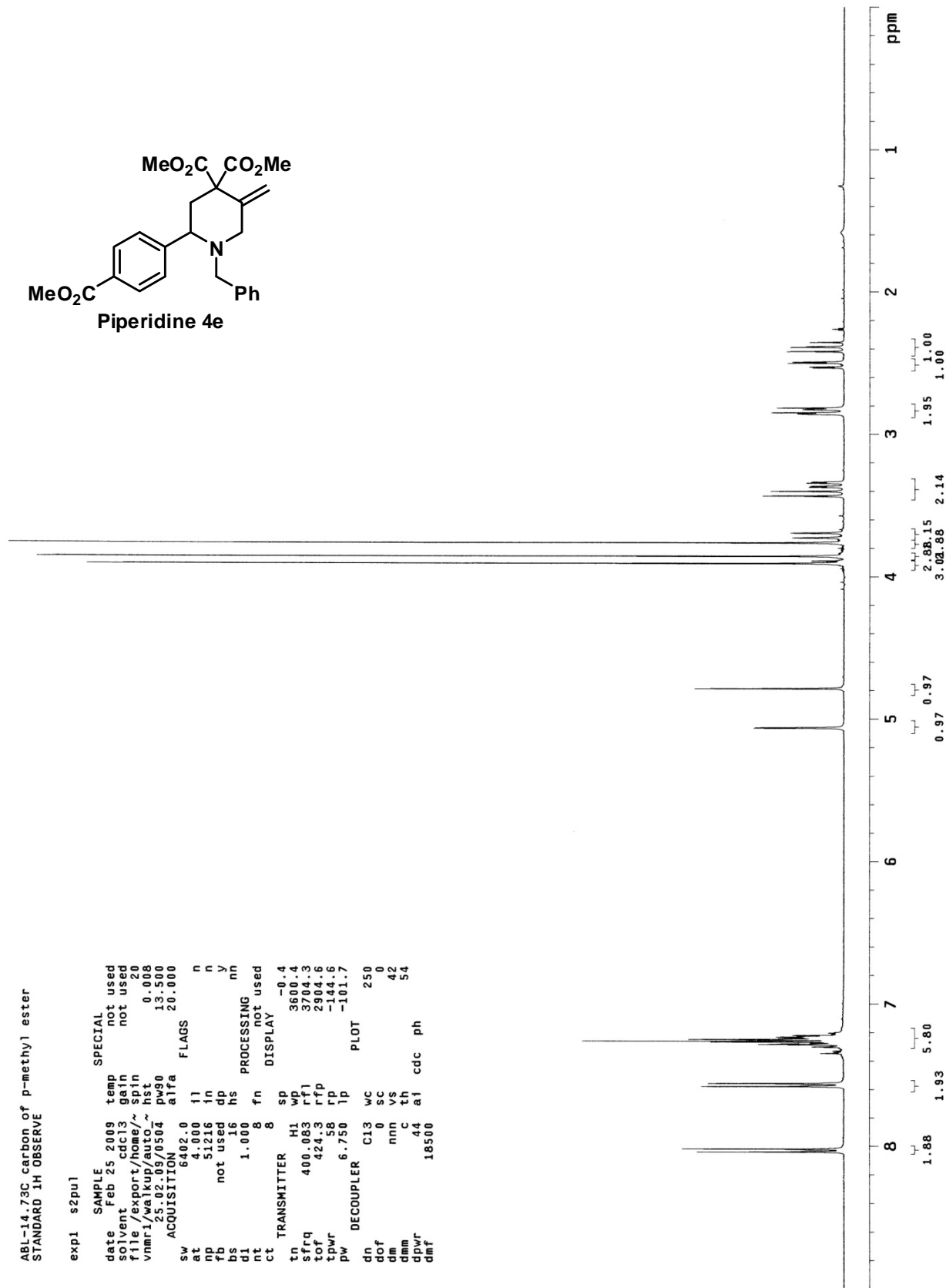
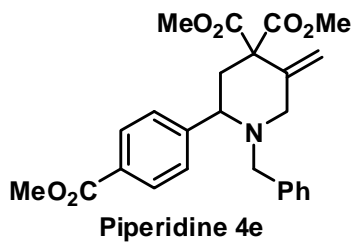
SAMPLE		PRESATURATION	
date	Feb 11 2009	satmode	n
solvent	cdcl3	wet	SPECIAL
file	/home/kerr/vn~	temp	26.0
mrssys/data/terr/T~		gain	not used
PL-Q-53-carb TPL-Q~		spin	20
-53-carb_01/CARBON~		hst	0.008
01		pw90	8.800
ACQUISITION	25133.5	alpha	10.000
at	1.304	FLAGS	
np	65536	l1	n
fb	14000	l1n	n
bs	8	dp	y
dl	1.000	hs	nn
nt	1000	lb	0.50
ct	184	fn	not used
tn	TRANSMITTER C13	DISPLAY	-0.7
sfrq	100.531	sp	18102.3
tof	1530.6	wp	9247.1
tpwr	54	rfl	7740.0
pw	4.400	rtp	-35.3
dn	DECOUPLER H1	lp	-338.9
dof	0	WC	250
dm	yy	SC	147
decwave	30	SS	12
dpwr	9500	tn	nm
dmr		cdc	ph



ABI-14.73C carbon of p-methyl ester
STANDARD 1H OBSERVE

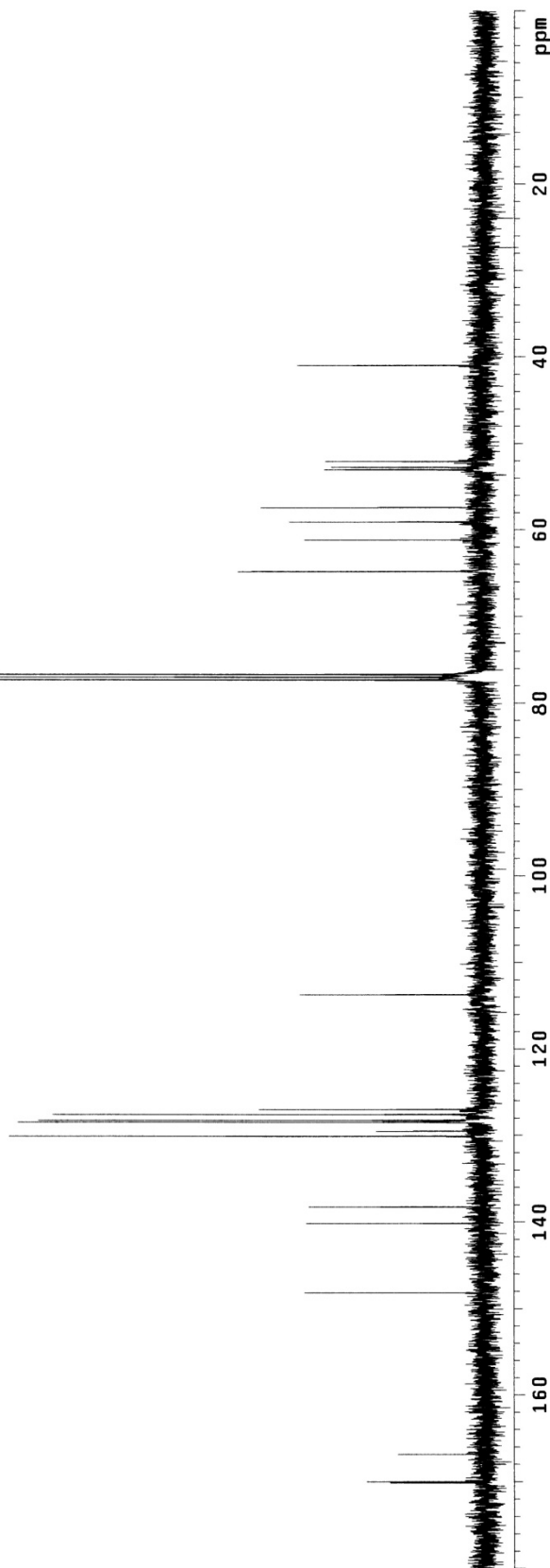
exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 25 2009	temp	not used
solvent	Feb 25 cdc13	gain	not used
file	export/home/~	spin	not used
nmr1	2502090504	ns	0.008
ACQUISITION	alpha	ps0	13.500
sw	6402.0	alpha	20.000
at	4.000	il	
ns	51216	in	n
fb	not used	dp	y
bs	1.000	hs	
di		fn	not used
nt	8	fn	not used
ct	TRANSMITTER	8	DISPLAY
tn	H1	sp	-0.4
sfreq	400.083	wp	3600.4
tof	424.3	rfl	3704.3
tdwr	58	rfp	2904.6
pw	6.750	rp	-144.6
DECOUPLER	C13	lp	-101.7
dn	0	wc	250
dof	0	sc	42
dm	nnn	vs	54
dmm	c	th	
dpwr	44	ai	cdc
dmf	18500	ph	



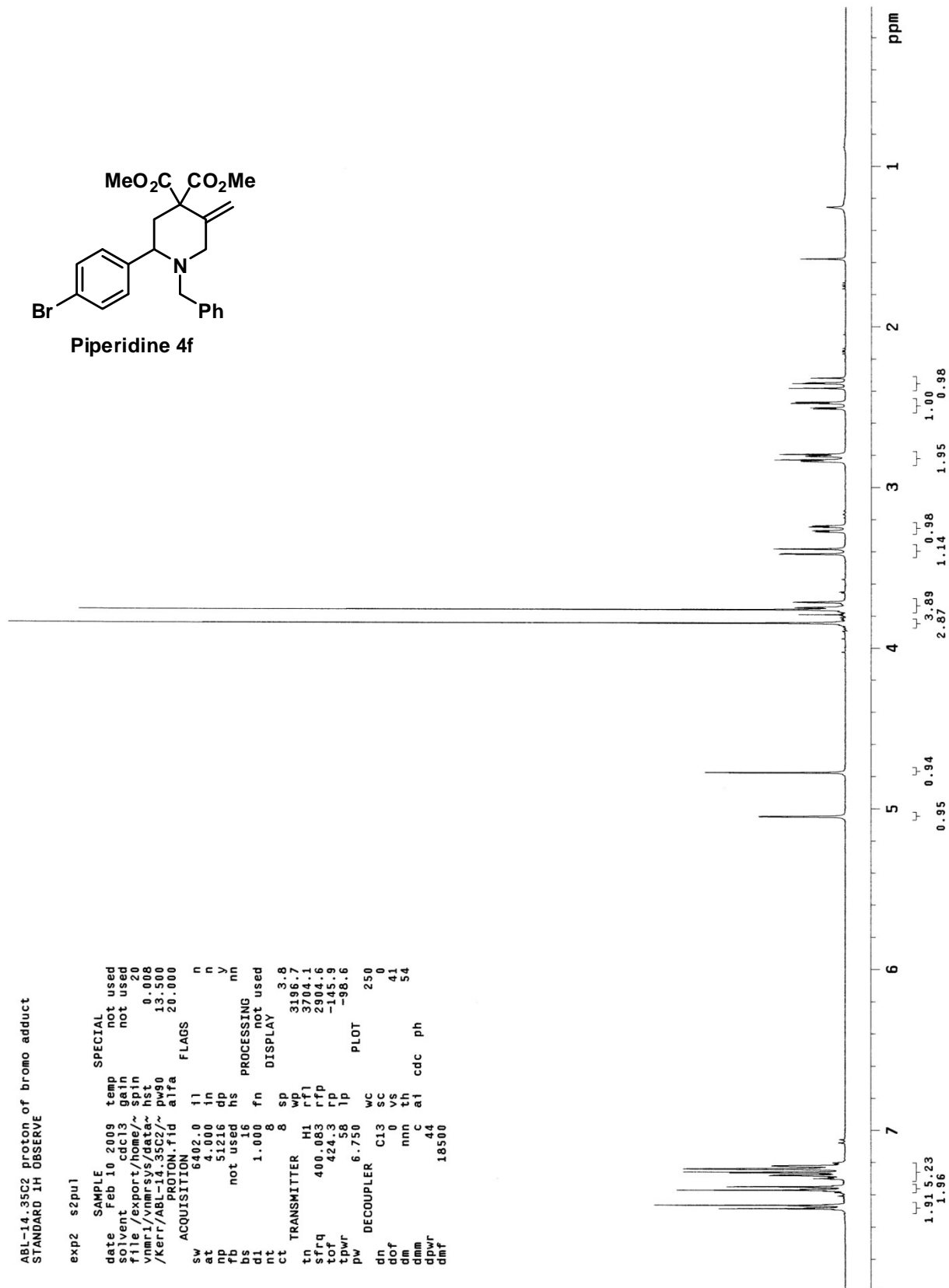
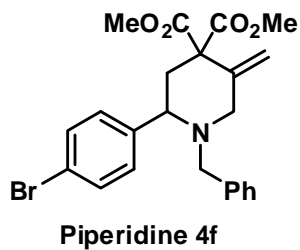
exp1 s2pu1

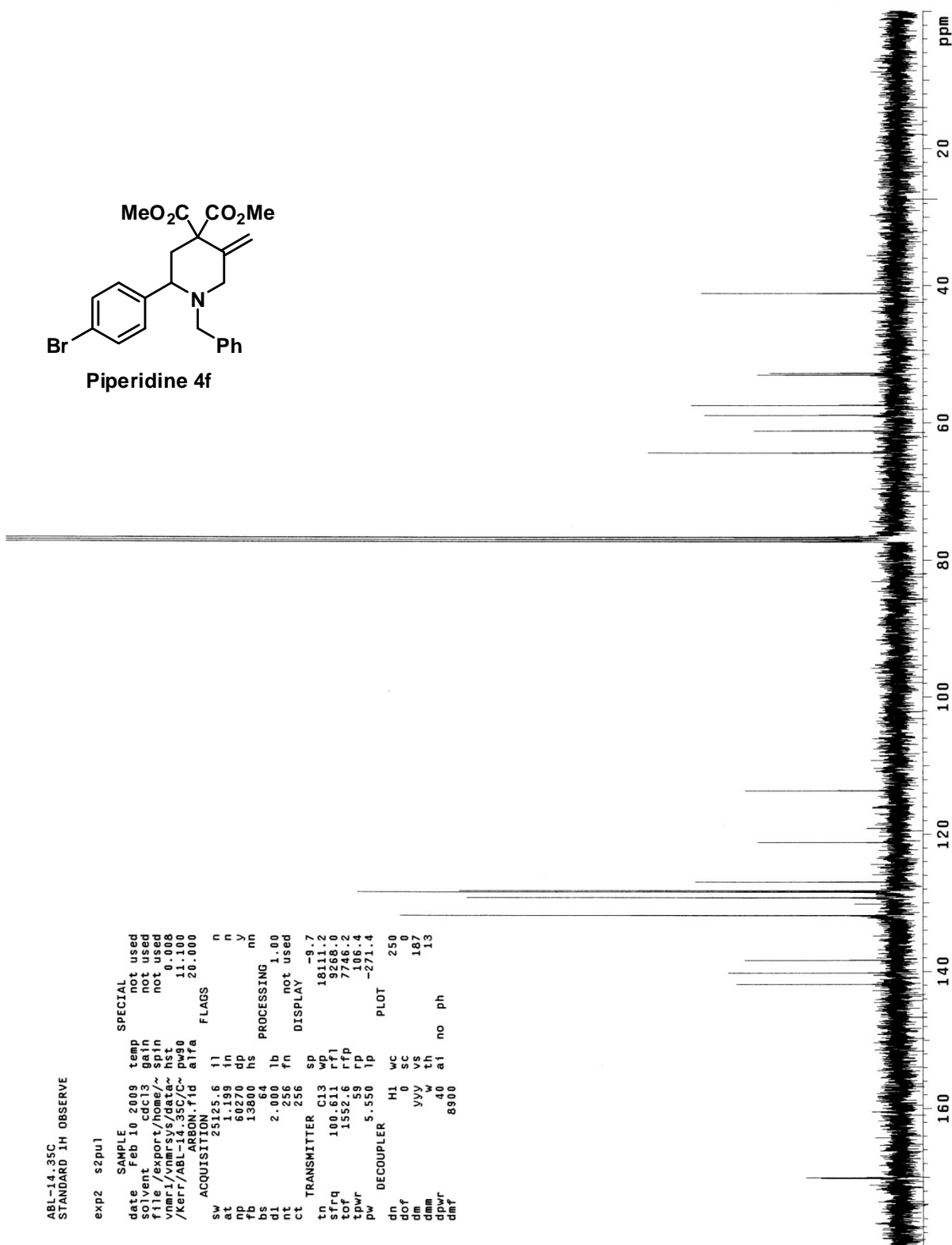
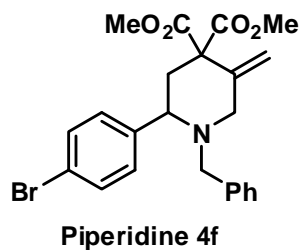
Piperidine 4e



ABL-14_35C2 proton of bromo adduct
STANDARD 1H OBSERVE

```
exp2 s2pu1
SAMPLE
date Feb 10 2009 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin 20
nmr1/vnmrSYS/data~ hst 0.008
/Kerr/ABL-14_35C2/~ pw90 13.500
PROTON.fid alfa 20.000
ACQUISITION
sw 6402.0 il n
at 4.000 in n
np 51216 dp y
fb not used hs nn
bs 16 PROCESSING
dl 1.000 fn not used
nt 8 DISPLAY
ct 8 sp 3.8
TRANSMITTER
tn H1 wp 3196.7
sfrq 400.083 rfl 3704.1
tof 424.3 rfp 2904.6
tpwr 58 lp -145.9
pw 6.750 PLOT -98.6
DECOUPLER
dn C13 wc 250
dof 0 sc 0
dm nnn th 41
dmm c a1 54
dpwr 44
dmf 18500
```



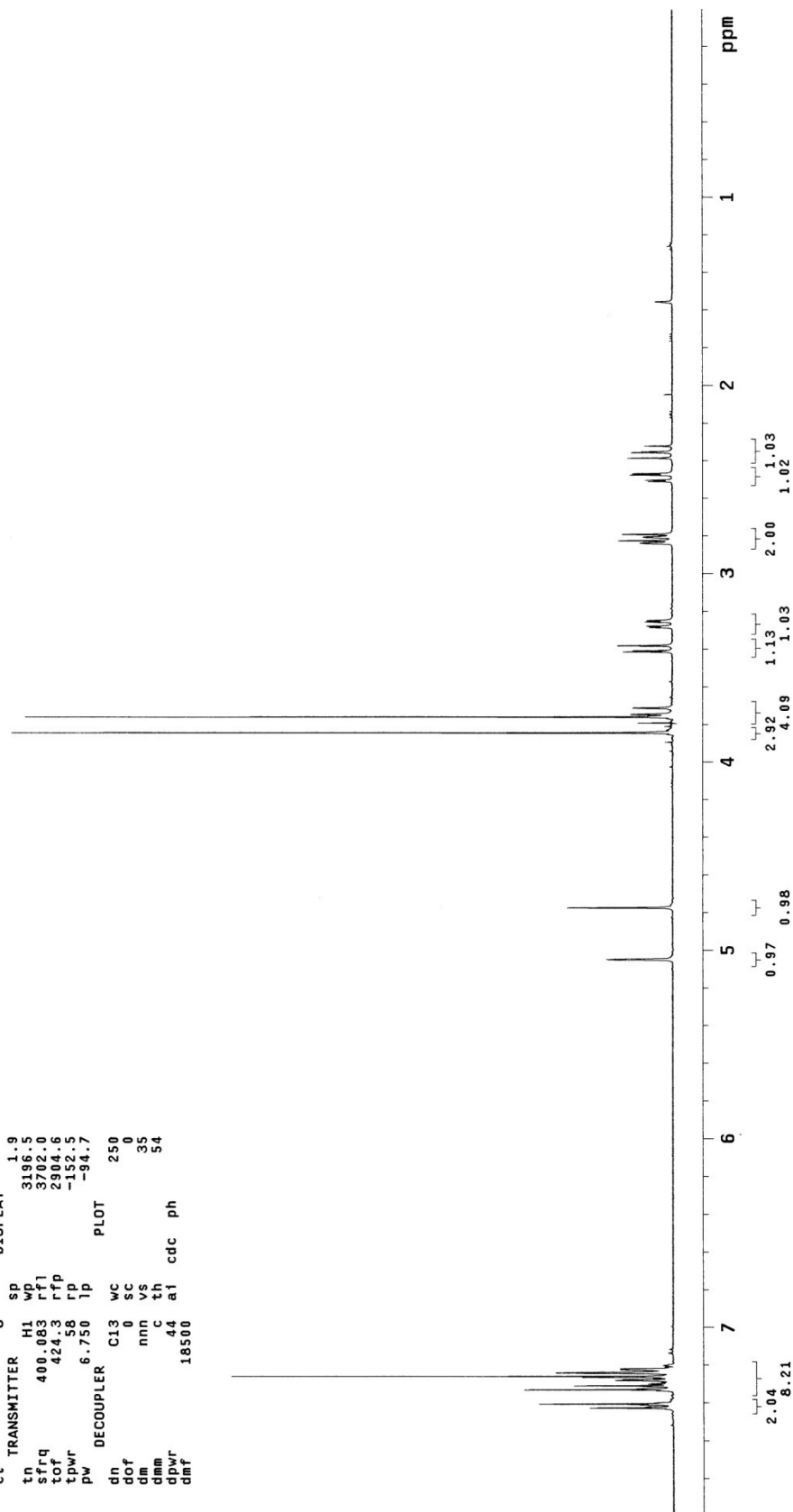
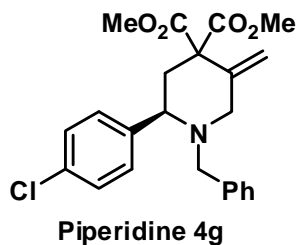


TPI-Q-15-ag
STANDARD 1H OBSERVE

```

exp4 s2pu1
SAMPLE
date Jan 19 2009 temp not used
solvent cdc13 gain not used
file/export/home/~ hst 20
vnari/walkup/auto/~ hst 0.008
19.01.09/0223 pw90 13.500
ACQUISITION alfa 20.000
SW 6402.0
at 4.000 il n
np 51216 in n
fb not used dp y
bs 16 hs
d1 1.000 PROCESSING nn
nt 8 fn not used
ct 8 DISPLAY
TRANSMITTER SP 1.9
tn H1 WD 3196.5
sfrq 400.083 rf1 3702.0
tof 424.3 rfp 2904.6
tpwr 58 rp -152.5
pw 6.750 lp -94.7
DECOUPLER C13 WC 250
dn 0 SC 0
dm nnn vs 35
dmm c th 54
dpwr 44 a1 cdc ph
dmf 18500

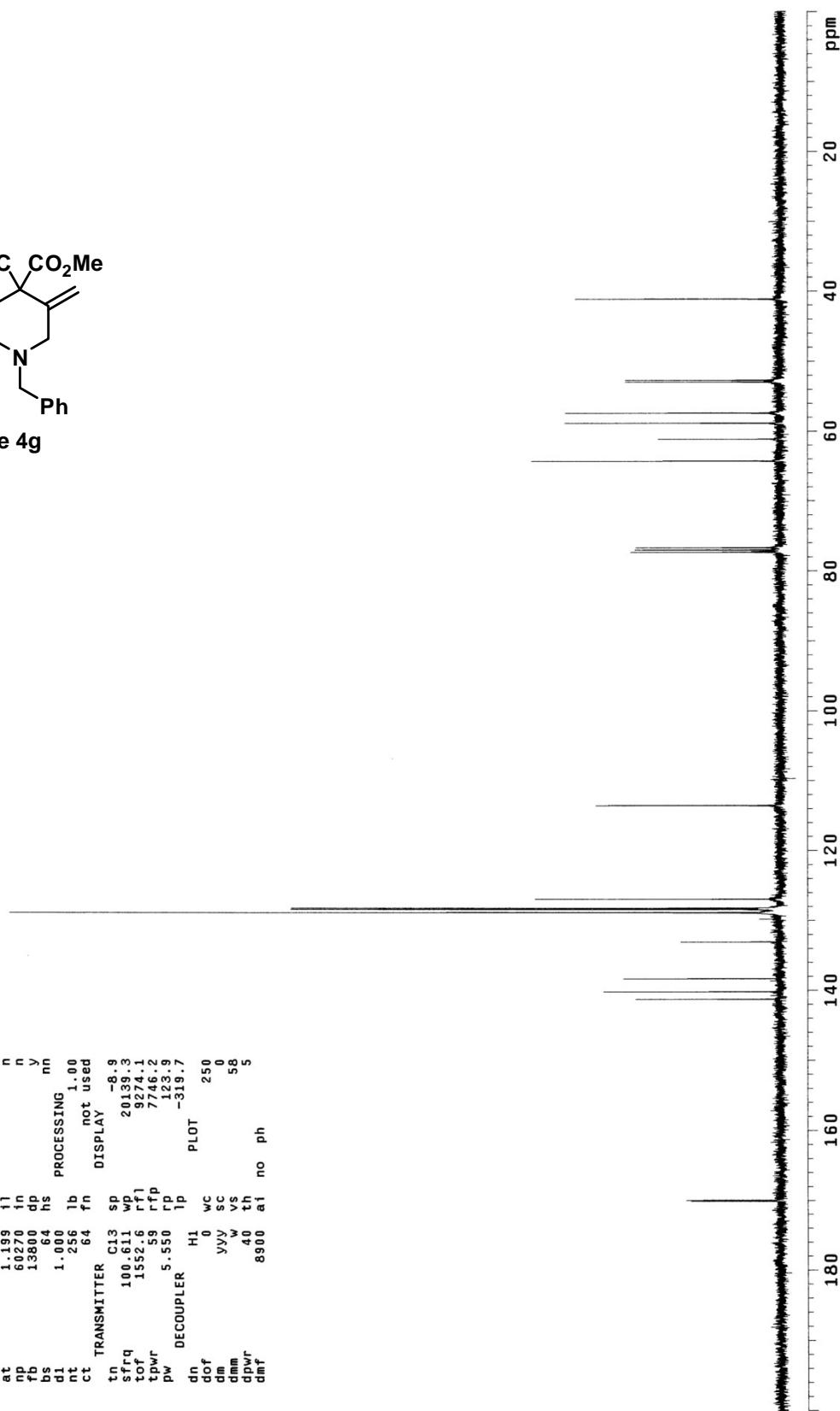
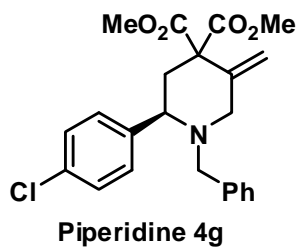
```

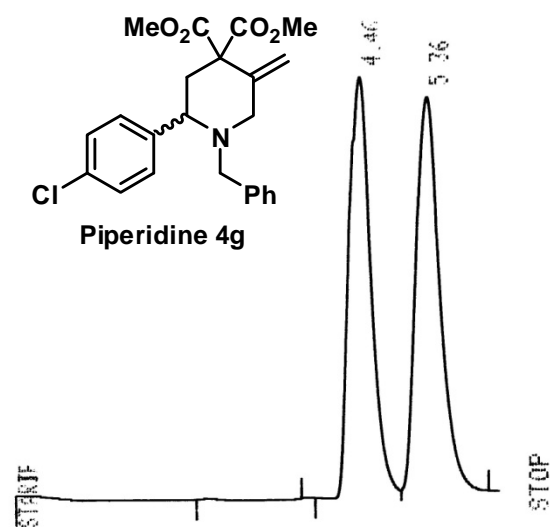


TPL-Q-15-carbon
STANDARD 1H OBSERVE

exp4 s2pu1

date	Jan 19 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
nmr1/wakeup/auto~	hst	0.008	
19.01.09/0222	pw90	11.100	
ACQUISITION	alpha	20.000	
sw	25125.6		
at	1.199	tl	n
tp	60270	tn	y
fb	135800	dp	
bs	64	hs	mn
ds	1.004		
dt	256	lb	1.00
nt	64	fn	not used
ct	TRANSMITTER	C13	DISPLAY
tn	100.611	sp	-8.9
sfreq	1552.6	wp	20139.3
tof	59	rf1	9274.1
tpwr	5.550	rfp	7746.2
pw	DECOUPLER	rp	123.9
dn	H1	lp	-319.7
dof	0	wc	250
dm	yyv	sc	0
dmm	w	vs	58
dpwr	40	th	5
dmf	8900	ai	no ph

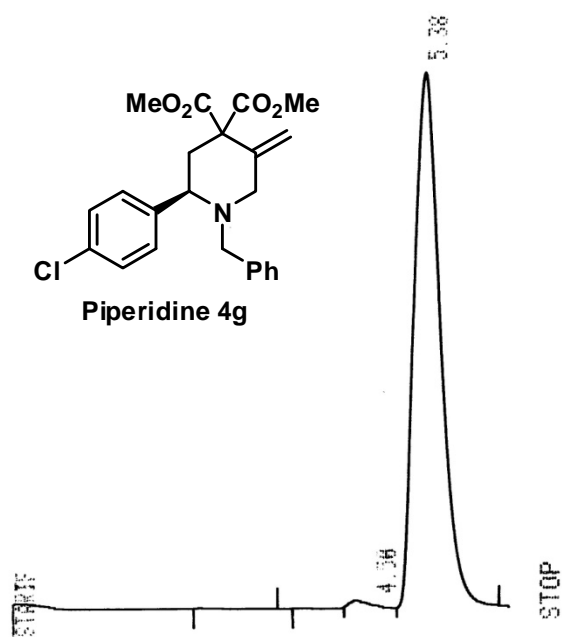




AREA%	RT	AREA	TYPE	AR/HT	AREA%
4.46	240470	48.467	PV	0.322	48.467
5.36	255680	51.533	VB	0.362	51.533

TOTAL AREA= 496150
MUL FACTOR= 1.0000E+00

RUN # 10



AREA%	RT	AREA	TYPE	AR/HT	AREA%
4.50	4655	1.237	PP	0.318	1.237
5.38	371590	98.763	PB	0.389	98.763

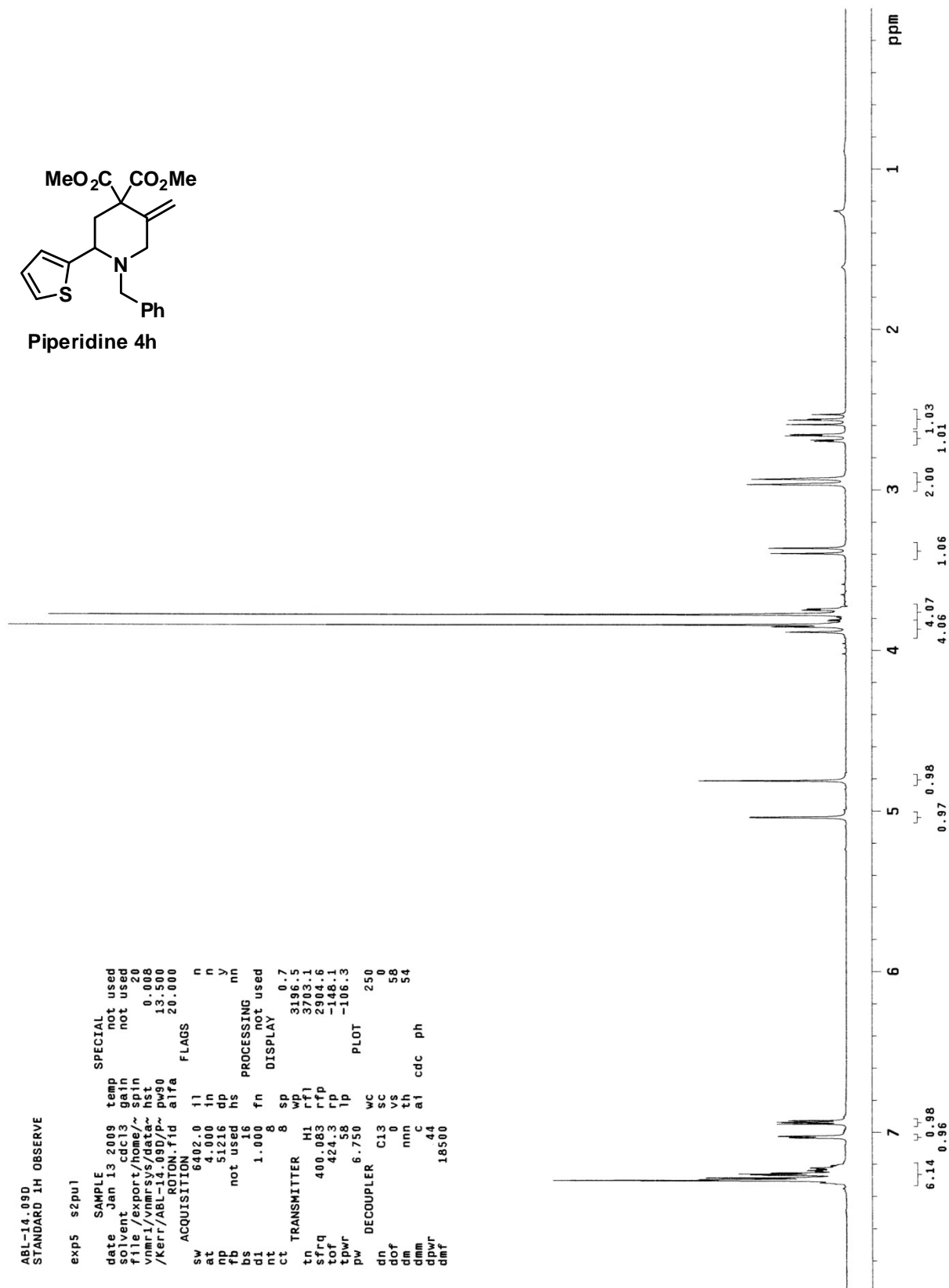
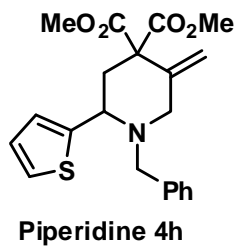
TOTAL AREA= 376250
MUL FACTOR= 1.0000E+00

RUN # 7

ABL-14.09D
STANDARD 1H OBSERVE

```

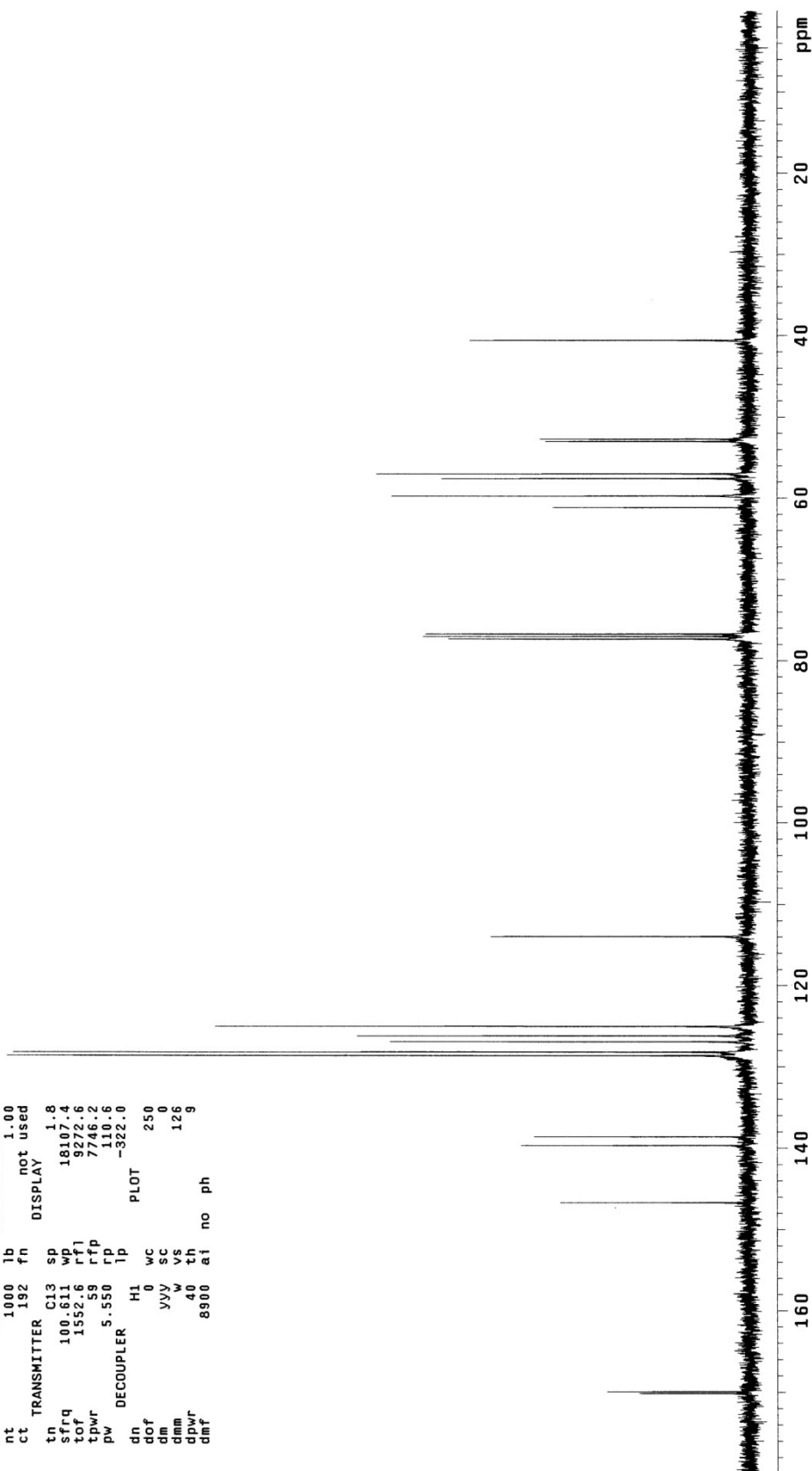
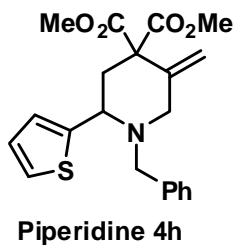
exp5 s2pul
SAMPLE
date Jan 13 2009 temp not used
solvent cdc13 gain not used
file /export/home/~spin 20
nmr1/vnmrsvs/data~ hst 0.008
/Kerr/ABL-14.09D/p~ pw90 13.500
ROTON.fid alfa 20.000
ACQUISITION
sw 6402.0 11 n
at 4.000 1n n
np 51216 dp y
fb not used hs
bs 16
d1 1.000 fn not used
nt 8
ct 8 sp
TRANSMITTER H1 wp 3196.5
rf1 3703.1
strq 400.083 rfp 2904.6
tof 424.3 rp -148.1
tpwr 58 lp -106.3
pw 6.750 PLOT
DECOUPLER C13 wc 250
dn 0 sc 0
dof 0 vs 58
dm nnn th cdc ph
dmm c
dpwr 44
dnt 18500
  
```



ABL-14.09C
STANDARD 1H OBSERVE

exp1 s2pu1

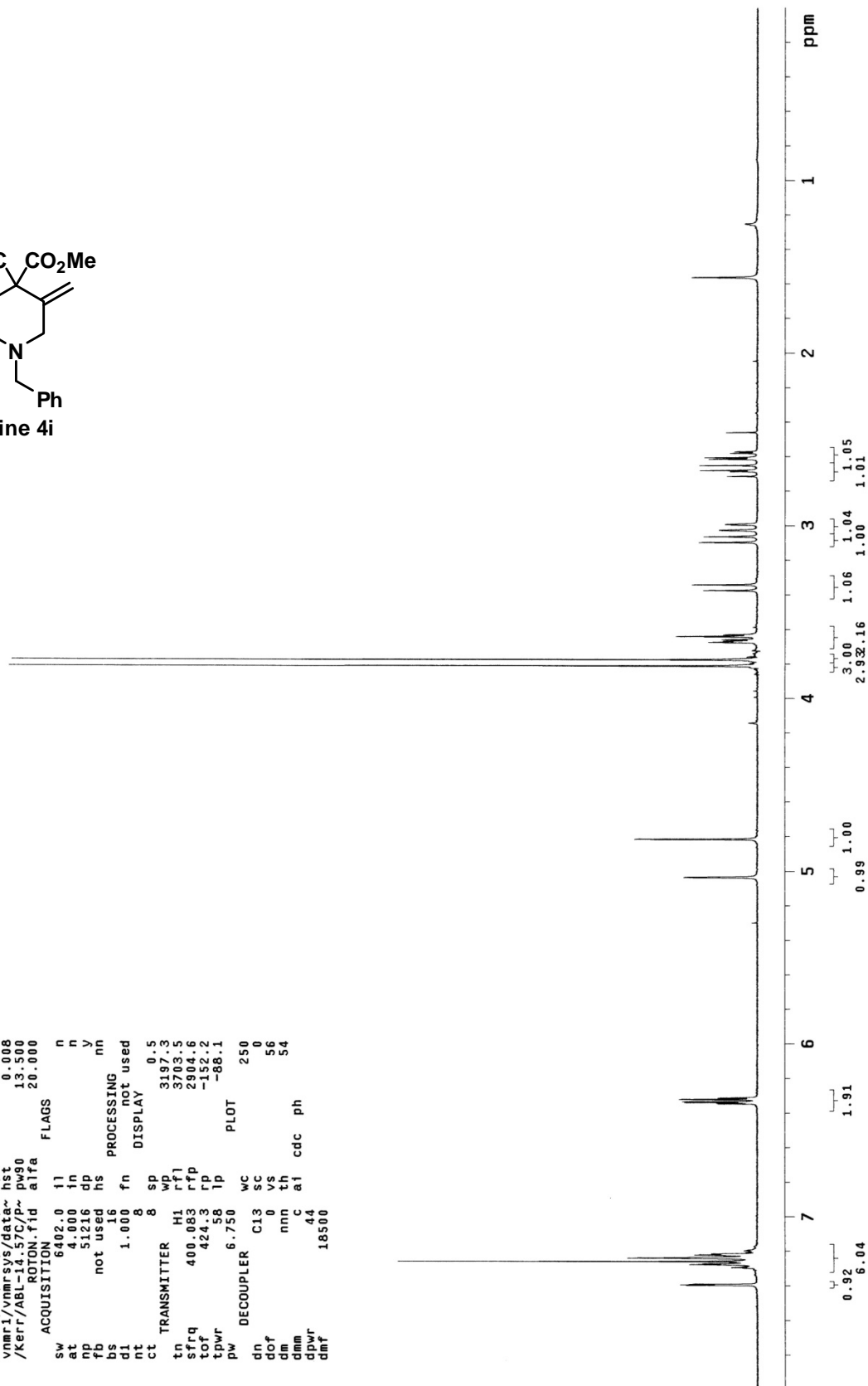
SAMPLE		SPECIAL	
date	Jan 12 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
nmr1	/walkup/autot~	hst	0.008
	12.01.09/0221	pw90	11.100
		alpha	20.000
ACQUISITION			
sw	25125.6	flags	
at	1.199	l1	n
np	60270	in	n
fb	13800	dp	y
bs	64	hs	nn
d1	1.000		
nt	1000	lb	1.00
ct	192	fn	not used
TRANSMITTER			
tn	c13	sp	1.8
sfreq	100.611	wp	18107.4
tor	1552.6	rfl	7272.6
tpwr	5.550	rfp	7746.2
pw	5.550	lp	1210.6
DECOUPLER			
dn	H1	pl	-322.0
dof	0	wc	250
dnn	yyv	sc	0
dmm	ys	vs	126
dpr	40	th	9
dpr	8900	at	no
dmf		ph	



exp7 s2pu1

SAMPLE			SPECIAL		
date	Jan 30 2009	temp	gain	spin	not used
solvent	cdcl3				0.20
file	/export/home/~				0.008
nmr1/vnmr2/data/~	hst				13.500
/Kerr/ABF~14.57C/p~	pw50				20.000
ROTOR/ABF	alpha				FLAGS
ACQUISITION					
sw	6402.0	1			n
np	4.000	fn			n
at	5126	dp			y
bs	not used	hs			nn
fs	16				nn
rd	1.000	fn			not used
nt	8				DISPLAY
ct	8	sp			0.5
TRANSMITTER					
tn	H1	wp			3197.3
srq	400.063	rfl			3703.5
tof	424.3	rfp			2904.6
tpwr	58	lp			-152.2
pw	6.750	rp			-88.1
DECOUPLER					
dn	C13	wc			250
dof	0	sc			0
dm	nnn	vs			56
dmm	c	th			54
dppr	44	ai			cdc
dmp	18500	ph			

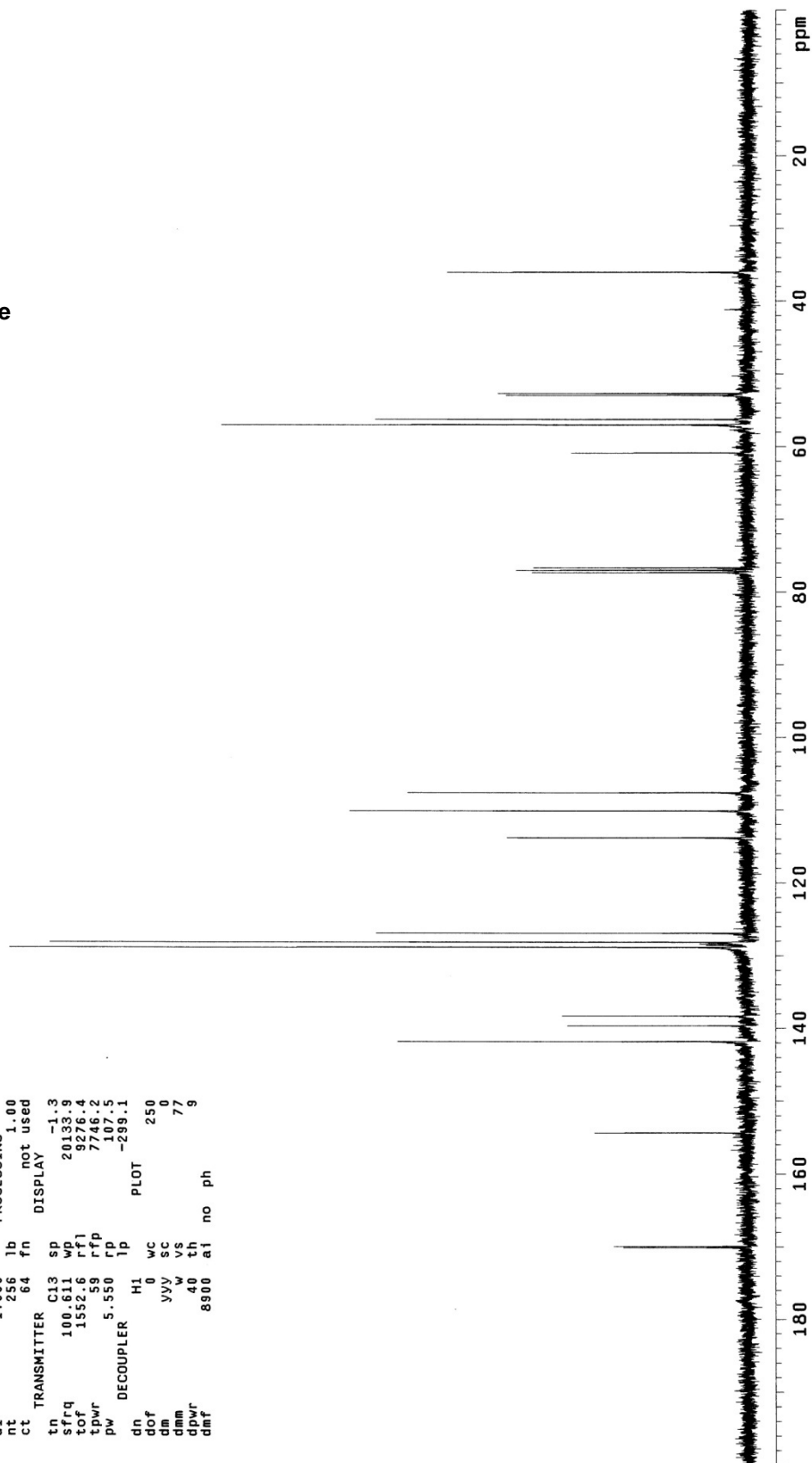
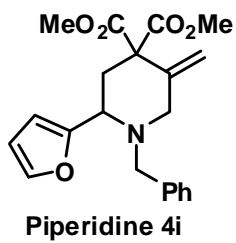
Piperidine 4i



ABL-14-57-C
STANDARD 1H OBSERVE

exp17 s2pul

SAMPLE		SPECIAL	
date	Jan 30 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
vnarl	/walkup/auto/~	hst	0.008
	30.01.09/0217	pw90	11.100
		alpha	20.000
ACQUISITION		FLAGS	
sw	25125.6		
at	1.199	fl	n
np	60270	in	n
fb	13800	dp	y
bs	64	hs	nn
d1	1.000		
nt	256	lb	1.00
ct	64	fn	not used
TRANSMITTER		DISPLAY	
tn	C13	sp	-1.3
sfrq	100.611	wp	20133.9
tof	1552.6	rfl	9276.4
tpwr	59	rfp	7746.2
pw	5.550	rp	107.5
DECOUPLER		lp	-299.1
dn	H1		
dof	0	wc	250
dm	yvy	sc	0
dmm	w	vs	77
dpr	40	th	9
dmt	8900	al	no ph

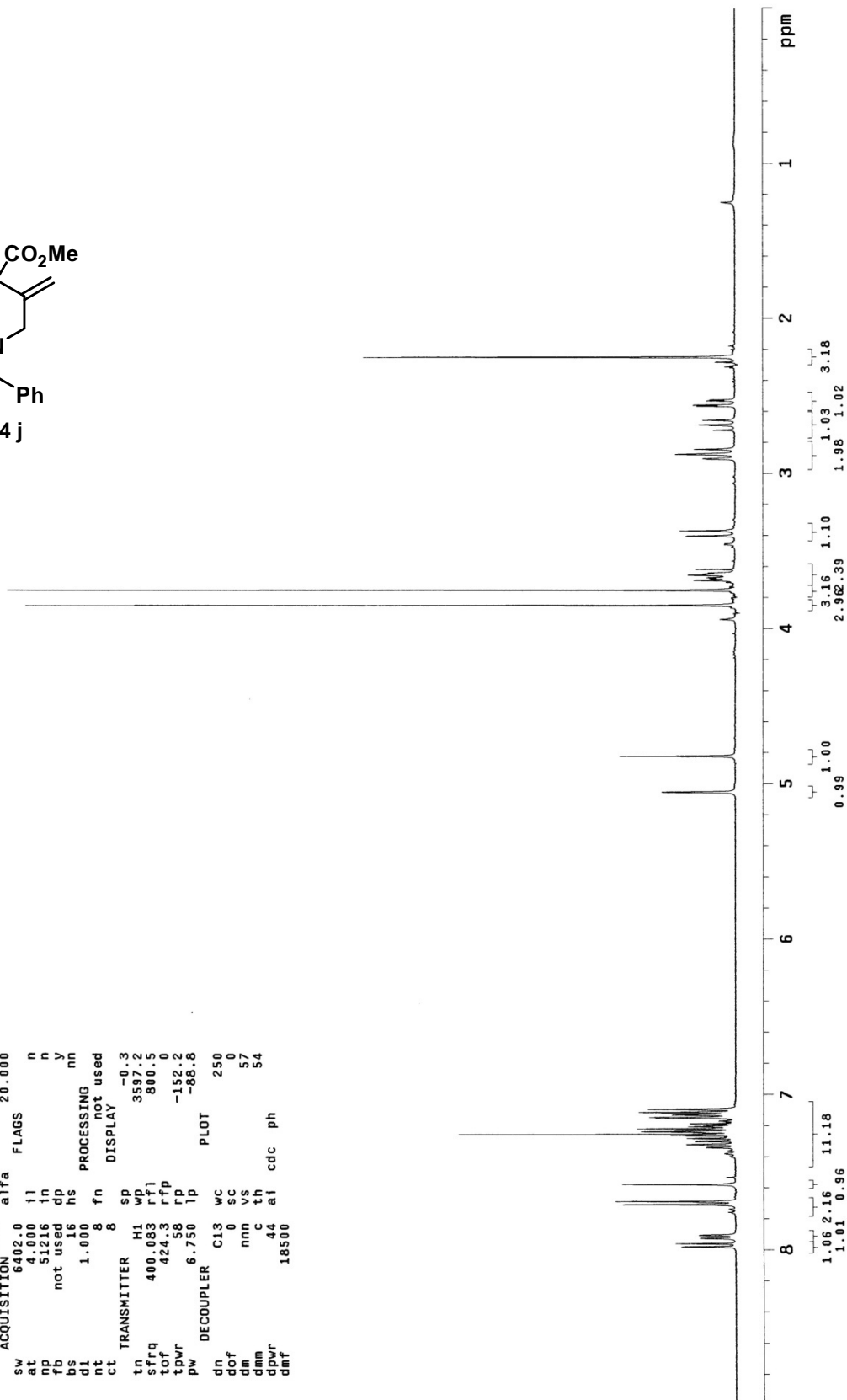
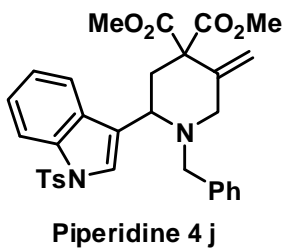


ABL-14.11C
STANDARD 1H OBSERVE

```

exp1 s2pul
date Jan 12 2009 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin 0.008
nmr1/walkup/auto~ hst 13.500
12.01.05/0405 pw50 20.000
ACQUISITION alfa 20.000
sw 6402.0 il n
at 4.000 in n
fp 31216 tn n
fb not used dp y
bs 16 hs
dl 1.000 fn not used
nt 8 not used
Ct TRANSMITTER H1 SP
tn 3597.2
sfreq 400.083 rf1 800.5
tof 424.3 rfp -152.2
tpwr 6.750 rp -86.8
pw DECOUPLER C13 wc 250
dn dof 0 sc 57
dm nnn vs 54
dmm c th
dpwr 44 a1 cdc ph
dmf 18500

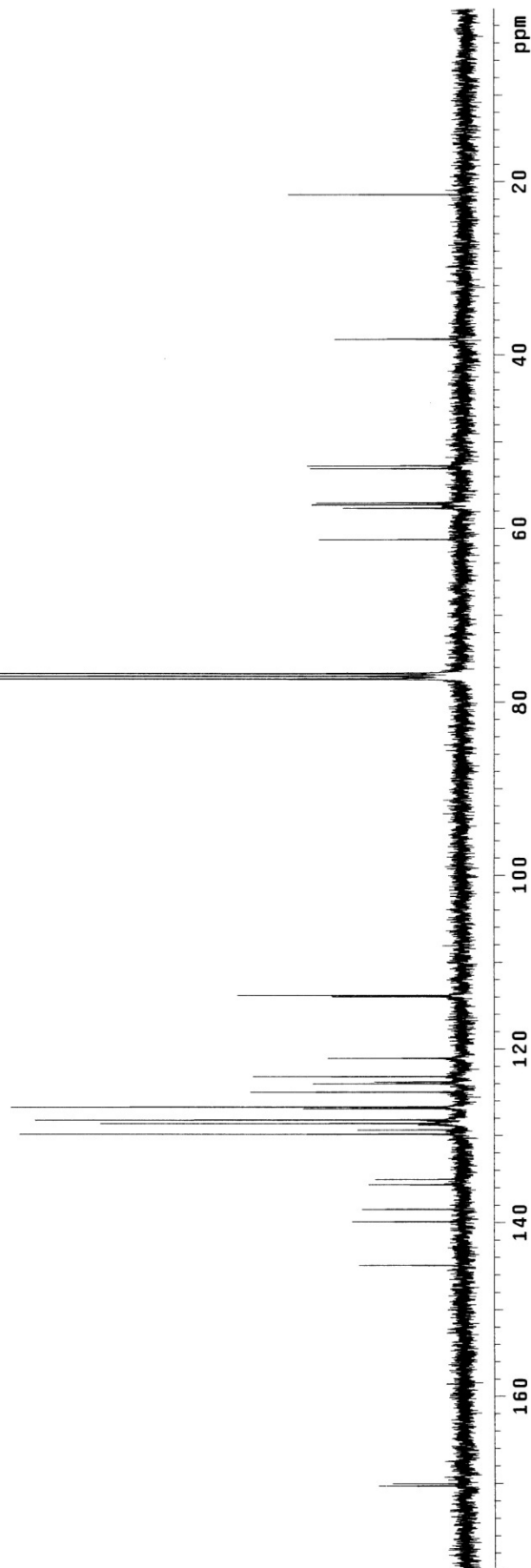
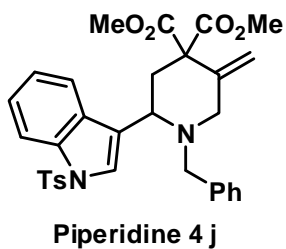
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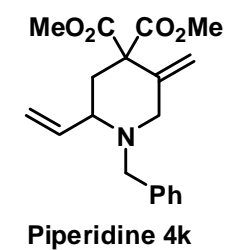


ABL-14.11C
STANDARD 1H OBSERVE

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 12 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
nmr1	walkup/autoc~	hst	0.008
12.01.09/0410	pw90	11.100	
ACQUISITION	alpha	20.000	
sw	25125.6	FLAGS	
at	1.199	l	n
np	60270	in	n
fb	13800	dp	y
bs	64	hs	
d1	1.000	lb	
nt	1000	fn	1.00
ct	1000	not used	
TRANSMITTER C13		SP	7.2
tn	100.611	wf	18107.4
sfrq	155216	rf	3267.2
tofr	155216	rfp	7746.2
tpwr	5.550	rp	106.4
DECOUPLER H1		lp	-323.1
dn	0	wc	250
dof	0	vy	sc
dm	0	vs	323
dmm	40	th	14
dpwr	8900	ai	no
dmf		ph	

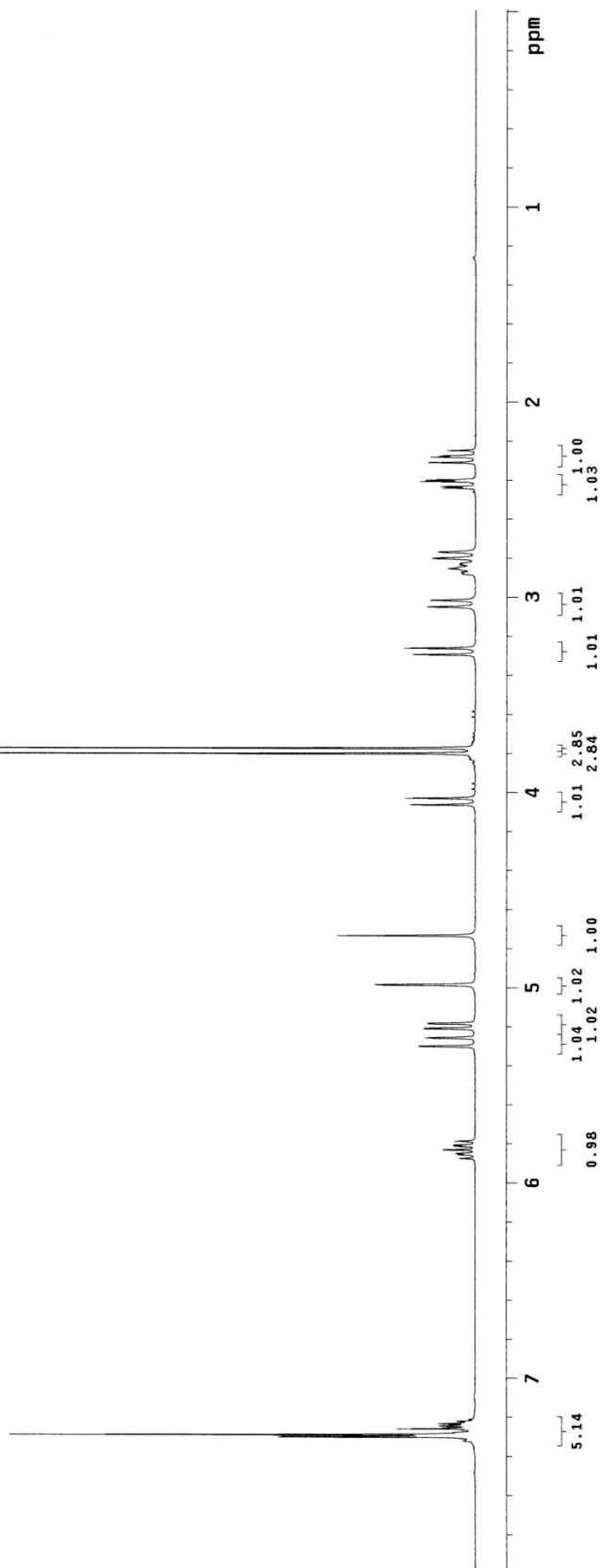




TPL-P-143

exp1 PROTON

date	May 19 2009	PRESATURATION	n
solvent	cdc13	satmode	n
file	cdc13	wet	SPECIAL
sw	6396.4	temp	25.0
at	2.561	gain	not used
np	32768	spin	20
fb	4000	hst	0.008
bs	32	pw90	9.500
d1	1.000	alfa	6.600
nt	8	il	FLAGS
ct	8	in	n
tn	8	dp	n
tn	8	hs	y
sfrq	399.762	fn	nn
tof	399.8	fn	not used
tpwr	59	DISPLAY	-3.1
pw	4.750	sp	3197.8
dn	C13	wp	3686.2
dof	0	rfl	2902.3
dm	nnn	rp	-131.3
decwave	w40_hfcp2	lp	-20.8
dpwr	34	PLOT	
dmf	29412	wc	250
		sc	0
		vs	33
		th	54
		ai	cdc
			ph

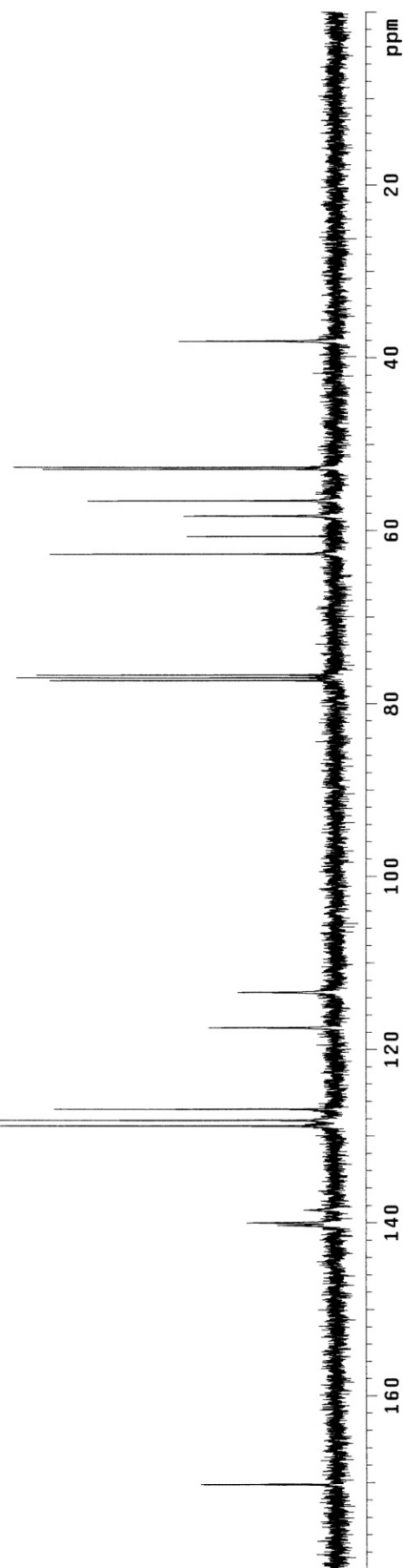
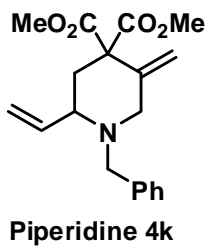


TPL-P-137
STANDARD 1H OBSERVE

```

exp1  s2pu1
=====
date  Jan 6 2009    temp  not used
solvent  Jan 6 2009    gain  not used
file  /export/home/~  spin  not used
nmr1/vnmrsvs/data/~  hst    0.008
/kerr/TPL-P-137/CA~ pw90   11.100
                        R80N.fid alfa  20.000
=====
ACQUISITION          FLAGS
sw  25125.6  l  n
at  1.199  in  n
np  60270  dp  y
fb  13800  hs  n
bs  64     lb  1.00
dl  1.000  fb  not used
nt  256    fn  not used
ct  64     sp  DISPLAY -2.0
=====
tn  TRANSMITTER C13  sp  18106.1
sfreq  100.611  wf  8274.1
rf  15526  rfp  774.2
tpwr  114.6  rp  114.6
pw  5.530  lp  -307.7
=====
dn  DECOUPLER H1  wc  250
dof  0  sc  90
dm  yyy  vs  8
dmm  w  th  40
dpwr  40  ai  no  ph
dmf  8900

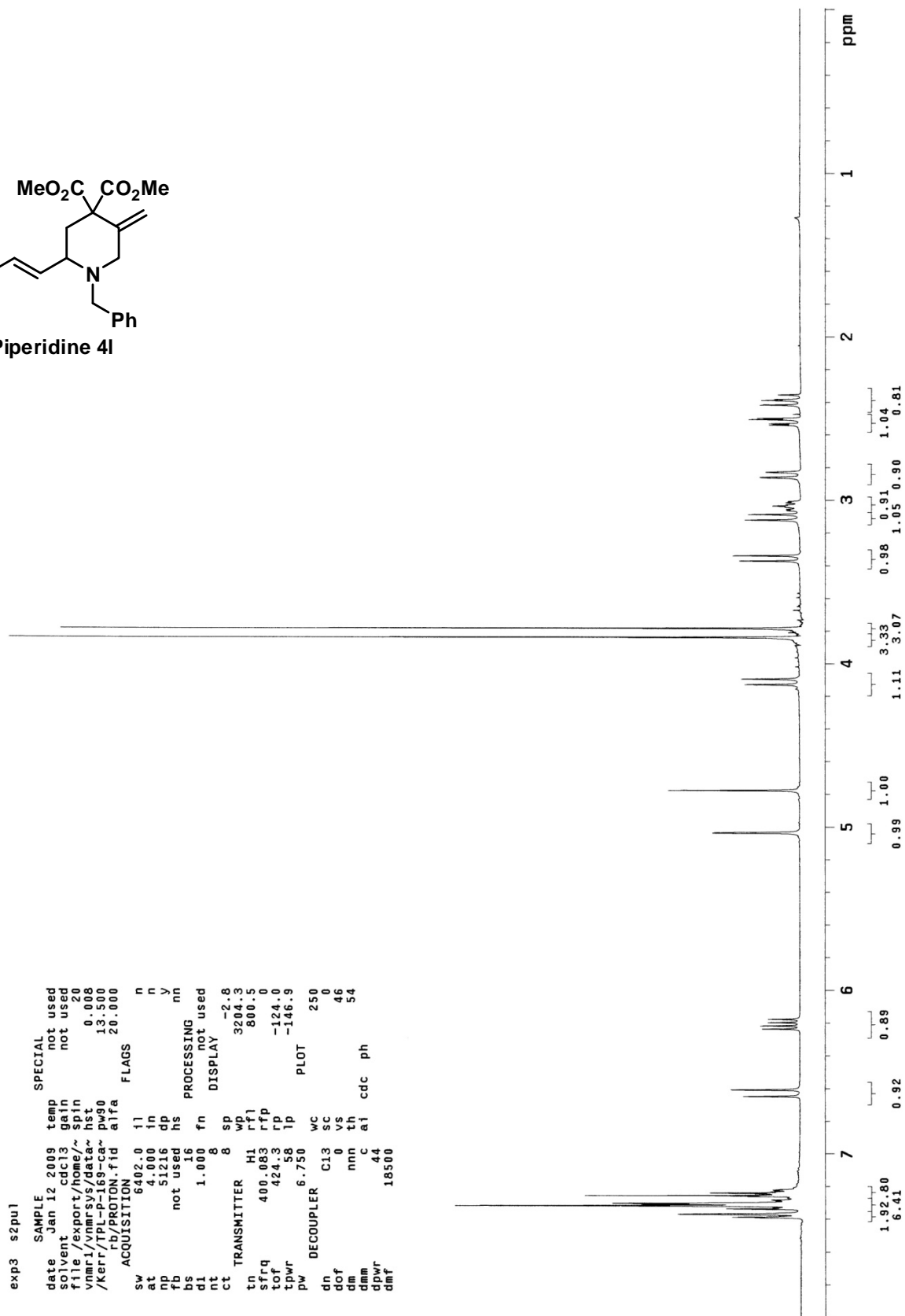
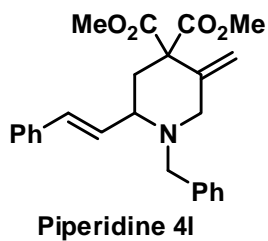
```



TPL-P-169-carb
STANDARD 1H OBSERVE

exp3 szpul

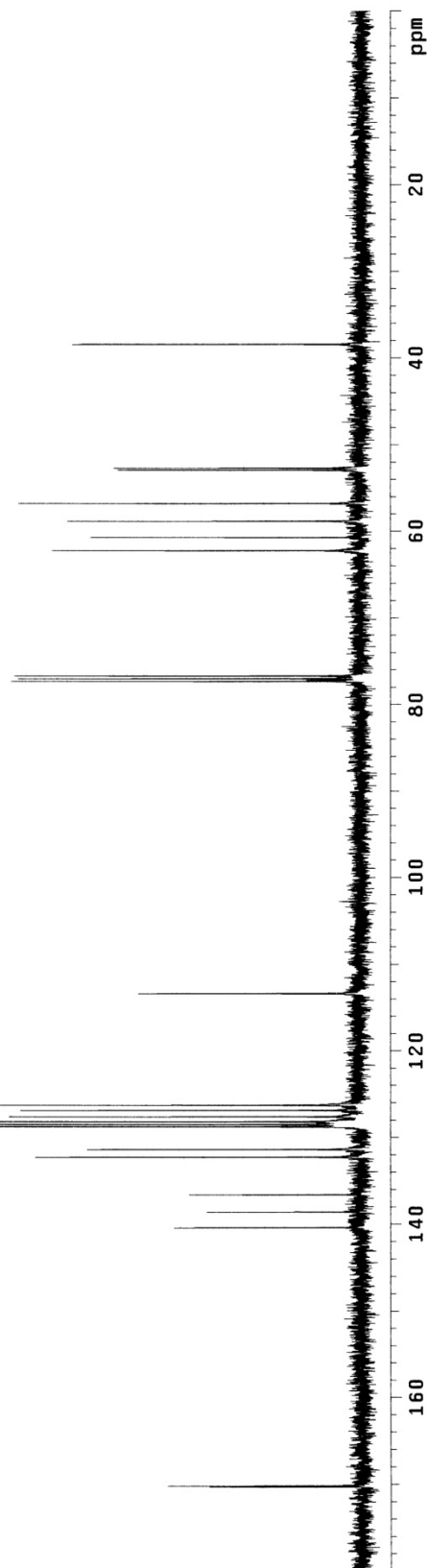
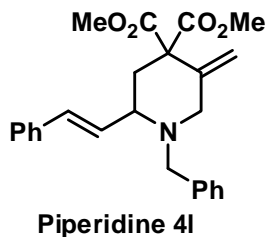
SAMPLE		SPECIAL	
date	Jan 12 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
nmr1/vmrsvs/data-	bst	0.008	
/Kerr/TPL-P-169-car-	pw90	13.500	
rb/PROTON.fid	alfa	20.000	
ACQUISITION			
sw	6402.0	fl	n
at	4.000	in	n
np	51216	dp	y
fb	not used	hs	nn
bs	16	PROCESSING	nn
d1	1.000	fn	not used
nt	8	DISPLAY	
ct	8	SP	-2.8
TRANSMITTER			
tn	H1	wp	3204.3
rf1		rf1	800.5
sfrq	400.083	rfp	0
tof	424.3	rp	-124.0
tpwr	58	lp	-146.9
pw	6.750	WC	250
DECOUPLER			
C13	sc		0
dn	0	vs	46
dm	nn	th	54
dam	C	ai	cdc
dpr	44	ph	
dnf	18500		



TPL-P-169-carb
STANDARD 1H OBSERVE

exp3 s2pu1

date	Jan 12 2009	temp	not used
file	export/cdcl3	gain	not used
file	export/hdata-	not used	
nmr	vsrv/deta-	pt	not used
/kerr	/TPL-P-169-ca-	pw90	11.100
	rb/CARBON.fid	alpha	20.000
		flags	
sw	25125.6	l1	n
at	1.199	in	n
np	60270	dp	y
fb	13800	hs	nn
bs	64		
d1	1.000	lb	1.00
nt	256	fn	not used
ct	128		DISPLAY
		sp	-4.3
tn	C13	wp	18115.8
sfrq	100.611	rfl	9274.1
tof	1552.6	rpf	7746.2
tpwr	59	rp	108.3
pw	5.550	lp	-353.9
			PLOT
dn	H1	wc	250
dof	0	sc	0
dm	yyv	vs	109
dmm	w	th	8
dpwr	40	a1	no
dmy	8900	ph	

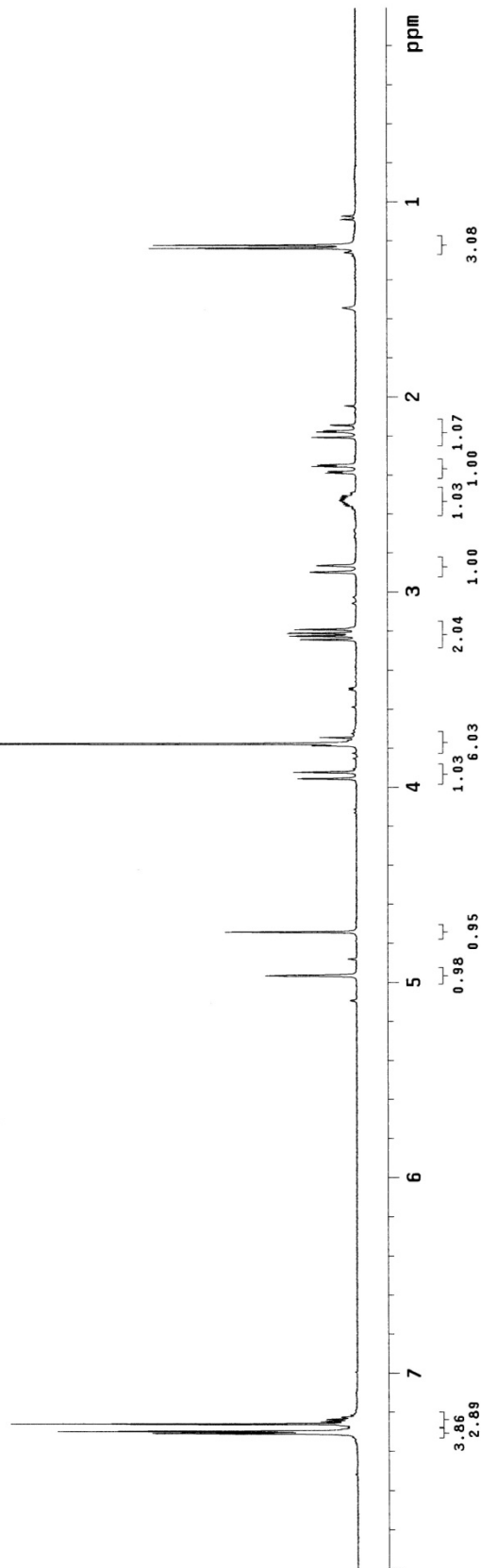
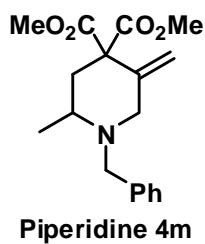


TPL-Q-55-2

```

exp1  PROTON
date   SAMPLE
Feb 24 2009
solvent cdc13
file   ACQUISITION exp
sw     6396.4
at     2.561
np     32768
fb     4000
bs     32
d1     1.000
nt     16
ct     16
tn     399.762
sfrq   399.8
tof     59
tpwr   4.750
pw     DECOUPLER C13
dn     0
dof     0
dm     nnn
decwave w40_hfcp2
dpwr   34
dmf    29412
wc     250
sc     0
vs     76
th     27
ai     cdc
ph     ph

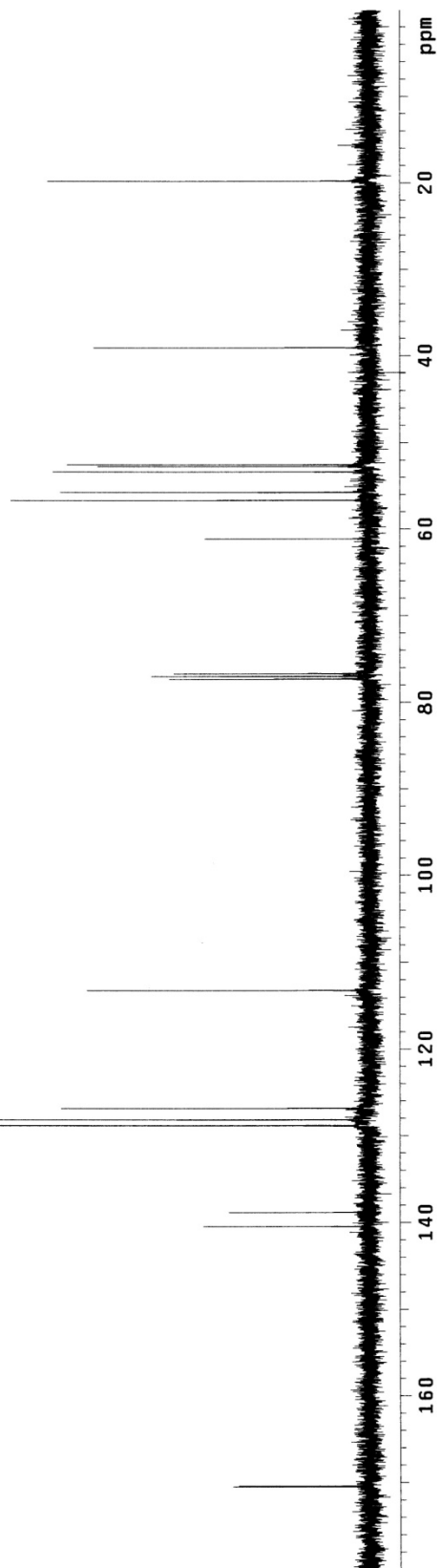
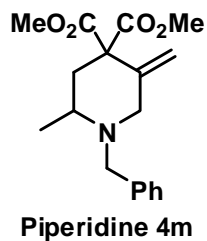
```

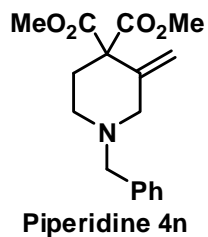


TPL-Q-55-carb

exp1 CARBON

date	Feb 24 2009	satmode	n
solvent	cdcl3	wet	n
file	/home/kerr/vn~	SPECIAL	
mrsys/data/Terry/T~	temp	25.0	
PL-Q-55-carb	gain	not used	
-55-carb_01/CARBON~	spin	not used	
01.fid	hst	0.008	
pw90	8.800		
sw	25133.5	alpha	10.000
at	1.304	fl	FLAGS
np	6536	in	n
rb	1400	in	n
ds	4	dp	y
dl	1.000	hs	PROCESSING
nt	236	lb	0.50
ct	36	fn	not used
tn	TRANSMITTER	C13	DISPLAY
sfrq	100.531	sp	7.7
tof	1530.6	wd	18082.4
tpwr	4.400	rfl	9252.5
pw	DECOUPLER	rfd	7740.0
dn	H1	lp	1.8
dof	0	lp	-308.7
dm	YVY	wc	250
decwave	w	sc	0
dpwr	39	vs	121
dmf	9500	th	68
		nm	cdc
		ph	

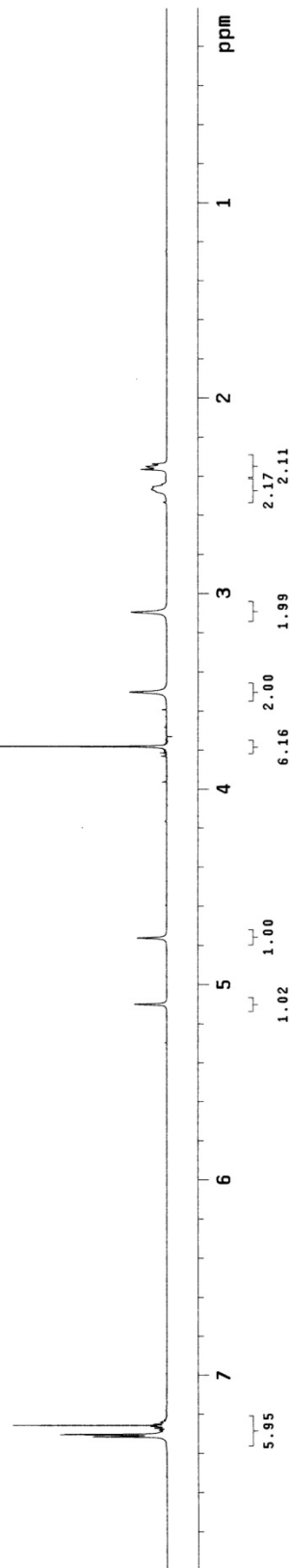




TPL-P-147-ag
 STANDARD 1H OBSERVE

```

exp1 s2pu1
SAMPLE
date Jan 8 2009 temp not used
solvent cdc13 gain not used
file /export/home/~spin 20
nmr1/vnmrSYS/data~ hst 0.008
/kerr/TPL-P-147-ag~ pw90 13.500
/PROTON.fid alfa 20.000
ACQUISITION
sw 6402.0 ll n
at 4.000 tn n
np 51216 dp y
bs not used hs PROCESSING nn
ds 16 fn not used
dt 1.008 f1 DISPLAY
Ct 8 SP 1.7
tn TRANSMITTER H1 WD 3200.8
sfrq 400.083 rfl 800.5
tof 424.3 rfp -149.8
tpwr 58 lp -103.5
pw 6.750 PLOT
DECOUPLER C13 sc 250
dn 0 vs 21
dm nnn th 3
dmm C a1 cdc ph
dpwr 44
dmf 18500
  
```

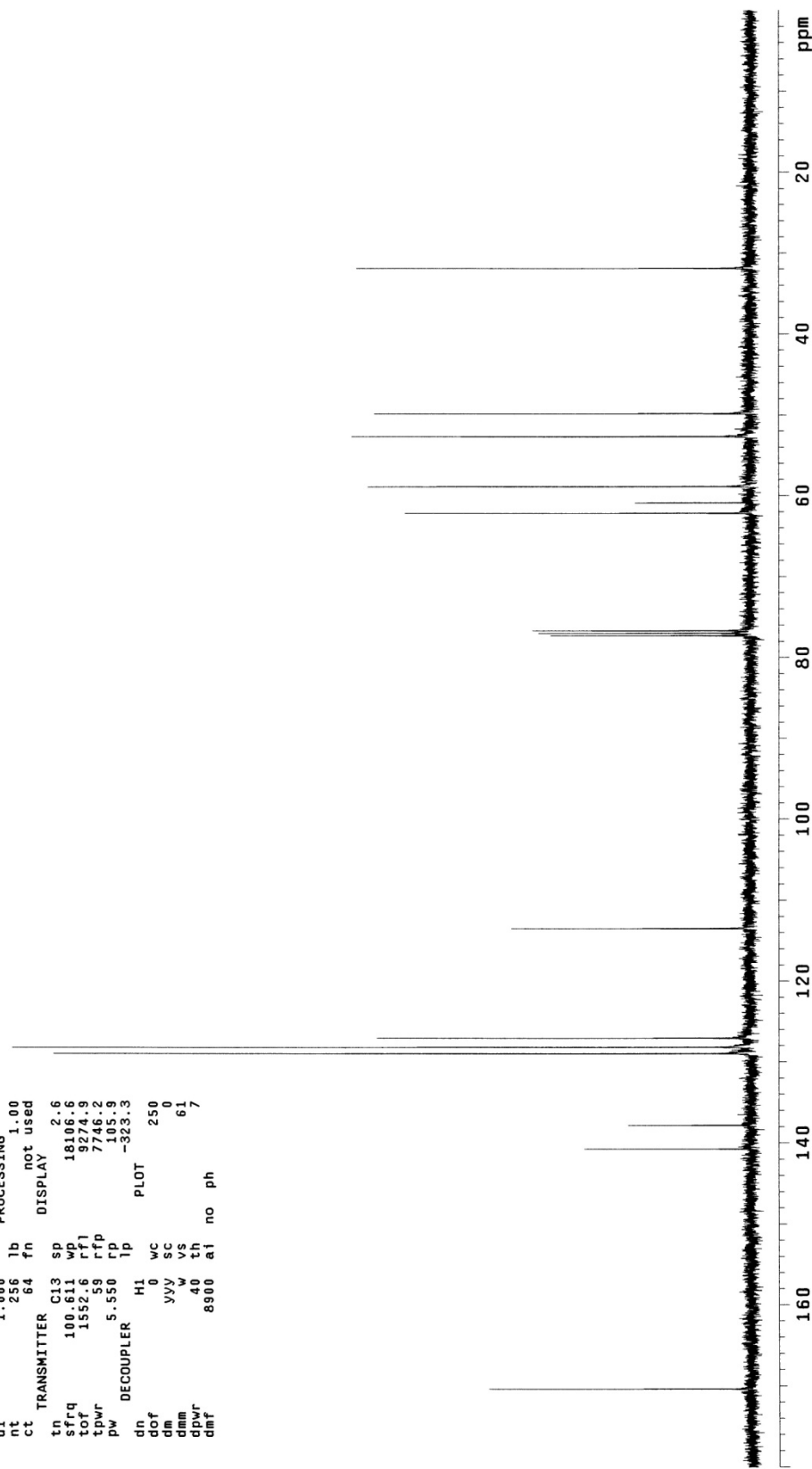
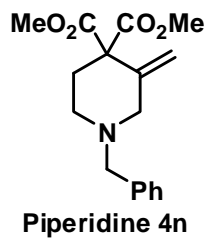


TPL-P-147-carb
STANDARD 1H OBSERVE

```

exp1  s2pu1
SAMPLE
date   Jan  8 2009    temp  not used
solvent Jan  8 2009    gain  not used
file   /export/home/~h1n  not used
nmr1   /usr/local/nmr/0008
vnmr1  08-01-09/0217  p1a0  0.008
                                11.100
ACQUISITION
sw      25125.6  f1  a1fa  20.000
at      1.199  f1  n
np      60270  in  n
fb      13800  dp  v
bs      64     hs  n
d1      1.000  lb  PROCESSING  nn
nt      256    1.00
ct      64     fn  not used
                                1.00
TRANSMITTER C13 SP DISPLAY
tn      100.611  wp  18106.6  2.6
sfrq    1552.6  rfl  9274.9
tof     59     rfp  7746.2
tpwr    5.550  rfp  105.9
pw      5.550  lp  -323.3
DECOUPLER H1 PLOT
dn      0      wc  250
dof     0      sc  0
dm      yyy   vs  61
dmm     w     vs  61
dpwr    40    th  7
dmf     8900  a1  no ph

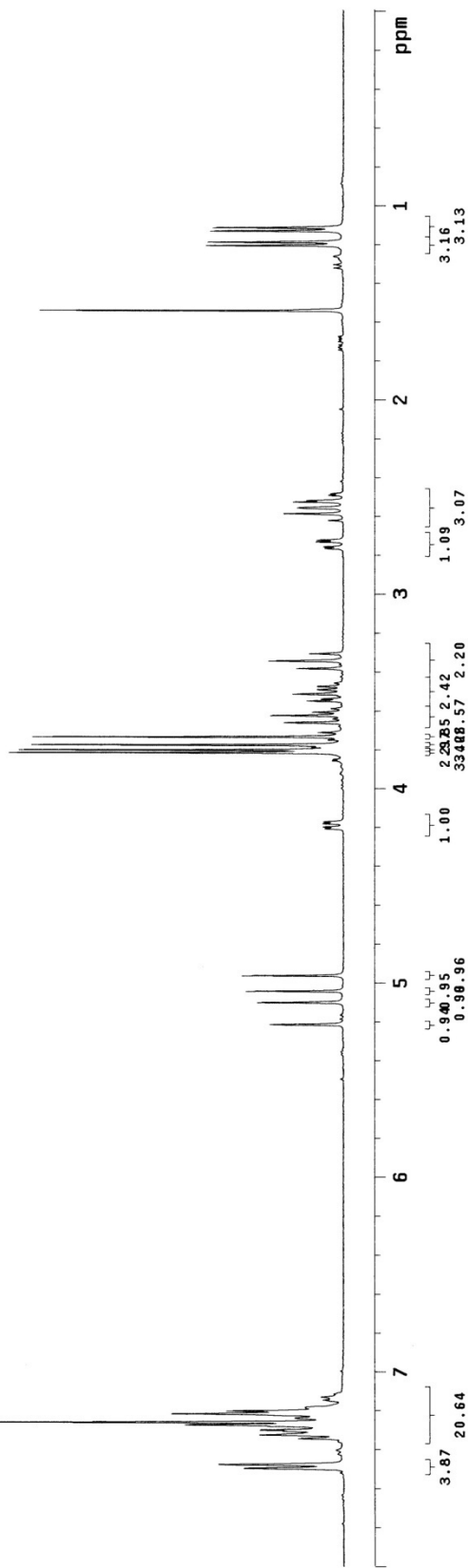
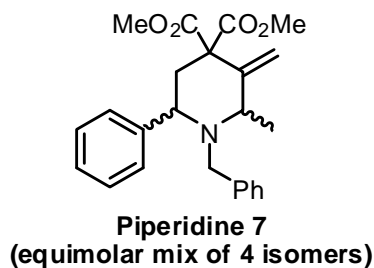
```



TPL-Q-49-agg

exp1 PROTON

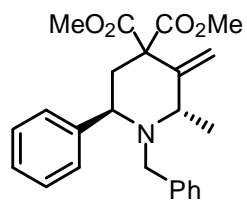
date	Feb 17 2009	PRESATURATION	n
solvent	cdcl3	satmode	n
file	/home/kerr/vn~	wet	SPECIAL
mrsvs/data/Terrv/T~	temp	not used	26.0
PL-Q-49-agg.TPL-Q-~	gain	0.008	20
49-agg.01/PROTON03	spin	9.500	6.600
ACQUISITION	hst	0.008	6.600
sw	6395.9	pw90	6.600
at	2.562	alpha	6.600
np	32768	fl	6.600
fb	4000	in	6.600
bs	4	in	6.600
d1	1.000	dp	6.600
nt	64	hs	6.600
ct	64	fn	6.600
TRANSMITTER	H1	not used	6.600
tn	399.762	sp	6.600
sfrq	399.762	wp	6.600
tof	399.8	rf1	6.600
tpwr	59	rfp	6.600
pw	4.750	rfp	6.600
DECOUPLER	C13	lp	6.600
dn	0	plot	6.600
dof	nnn	wc	6.600
dm	nnn	sc	6.600
decwave	w40_hfcp2	vs	6.600
dpwr	34	th	6.600
dmf	29412	at	6.600
		cdc	6.600
		ph	6.600



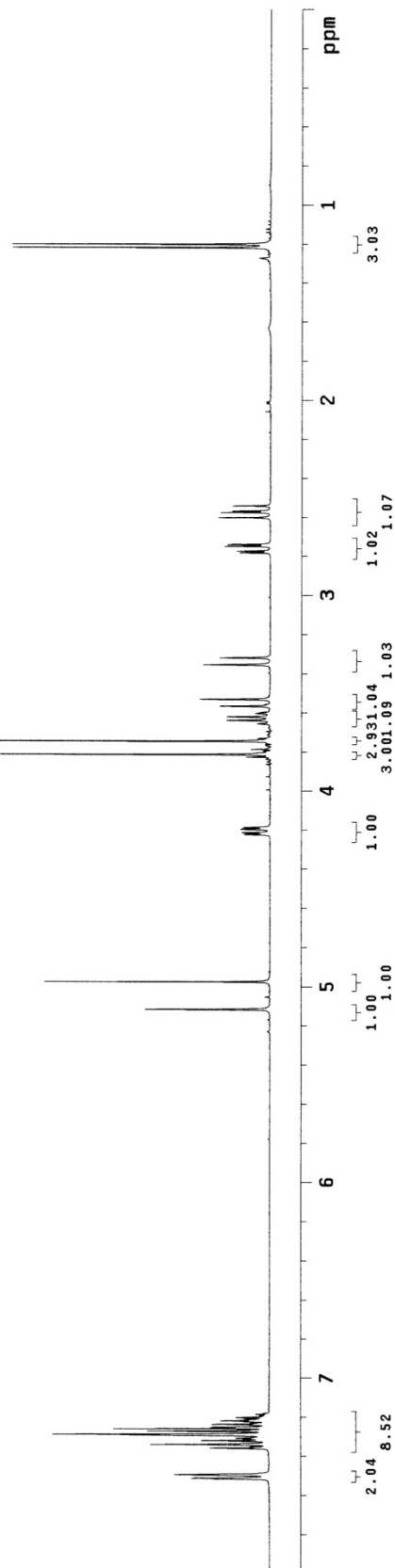
TPI-Q-207
STANDARD 1H OBSERVE

```

exp1 s2pul
SAMPLE
date Apr 14 2009 temp not used
solvent cdcl3 gain not used
file/export/home/~ spin 20
nmr1/wakeup/auto~ hst 0.008
14.04.09/0123 pw90 13.800
ACQUISITION alfa 20.000
sw 6402.0
at 4.000 il n
np 51216 in n
fb not used dp v
bs 16 hs
d1 1.000
ct 8 fn not used
nm PROCESSING
ct 8 DISPLAY
TRANSMITTER H1 SP -0.1
tn wp 3196.5
sfrq 400.083 rf1 3703.9
tof 424.3 rfp 2904.6
tpwr 58 tp -151.9
pw 6.900 lp -95.3
DECOUPLER C13 WC 250
dn 0 SC 0
dm nnn vs 40
dmm c th 54
dpwr 44 ai cdc ph
dmt 18500
  
```



Piperidine 7(2S,6R)

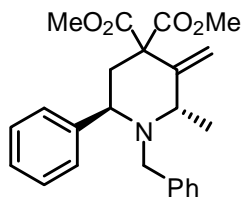


TPL-Q-207
STANDARD 1H OBSERVE

```

exp1 s2pu1
SAMPLE
date Apr 14 2009 temp not used
solvent cdcl3 gain not used
file/export/home/~ spin not used
vnmr1/walkup/autoc~ hst 0.008
14.04.09/0124 pw90 12.600
ACQUISITION alfa 20.000
sw 25125.6
at 1.199 l1 n
np 60270 in n
fb 13800 dp y
bs 64 hs nn
d1 1.000
nt 256 lb 1.00
ct 128 fn not used
TRANSMITTER C13 sp -5.9
tn 100.611 wp 18125.0
sfrq 1552.6 rf1 9271.1
tpwr 59 rfp 7746.2
pw 6.300 rp 116.8
DECOUPLER H1 lp -283.7
dn 0 wc 250
dof yvy sc 0
dm w vs 121
dmm 42 tn no ph
dmf 11100 at

```



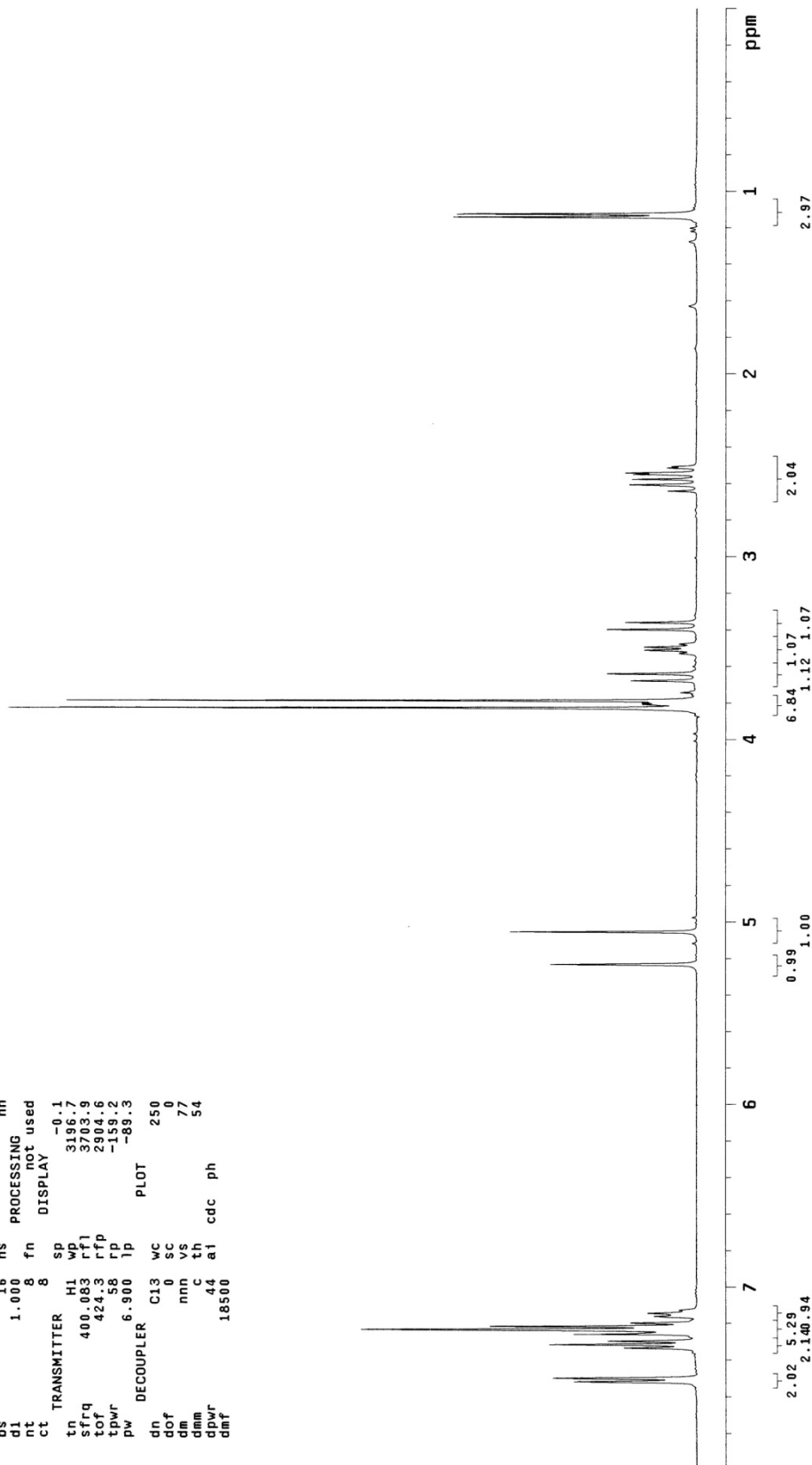
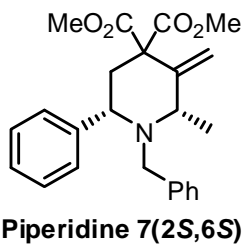
Piperidine 7(2S,6R)



TPL-Q-199
STANDARD 1H OBSERVE

```

exp1  s2pu1
SAMPLE
date   Apr 11 2009    temp not used
solvent cdc13        gain not used
file    /export/home/~ spin 0.20
nmr1/walkup/autoc~ hst 0.008
11.04.09/0202~ pw90 13.800
ACQUISITION 2.000 a1ra FLADS 20.000
sw      6402.0      il      n
at      45000      in      n
ap      51248      in      n
pe      not used   dp      y
bs      1.000      te      y
d1      1.000      fn      not used
nt      8          DISPLAY
ct      8          SP      -0.1
tn      H1         wp      3196.7
sfrq    400.083    rfl      3703.9
tof      424.3     rfp      2904.6
tpwr     58        rp      -159.2
pw      6.900     lp      -89.3
DECOUPLER C13      wc      250
dn        0        sc      0
dof       0        vs      77
dm        0        th      54
dmm       44      ai      cdc
dpwr      18500    ph
dmf
  
```

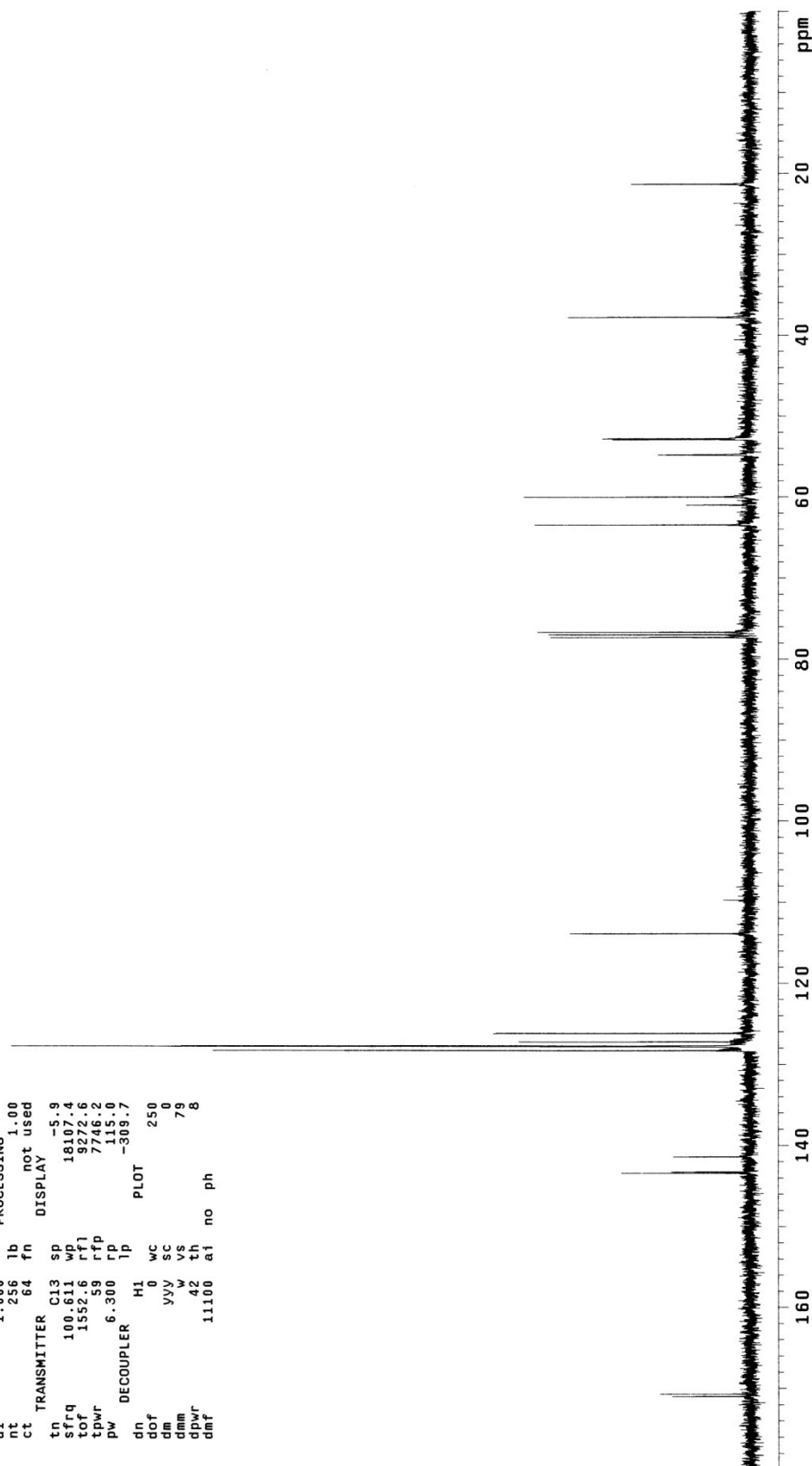
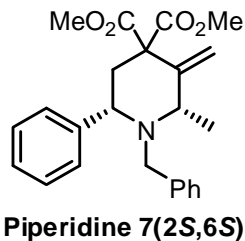


TPL-Q-199
STANDARD 1H OBSERVE

```

exp1 s2pul
SAMPLE
date Apr 11 2009 temp not used
solvent cdcl3 gain not used
file /export/home/~ hst not used
nmr1/valkup/auto/~ hst 0.008
11.04.05/0203 pw90 12.600
ACQUISITION a1fa 26.000
SW 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 64 hs nn
d1 1.000 PROCESSING
nt 256 lb 1.00
ct 64 fn not used
DISPLAY
TRANSMITTER C13 sp -5.9
tn 100.611 wp 18107.4
sfrq 1552.6 rfl 9272.6
tof 59 rfp 7746.2
tpwr 6.300 rp 115.0
pw DECOUPLER lp -309.7
dn H1 PLOT
dn dof 0 wc 250
dm yyv sc 0
dmm w vs 79
dpwr 42 th 8
dnt 11100 ai no ph

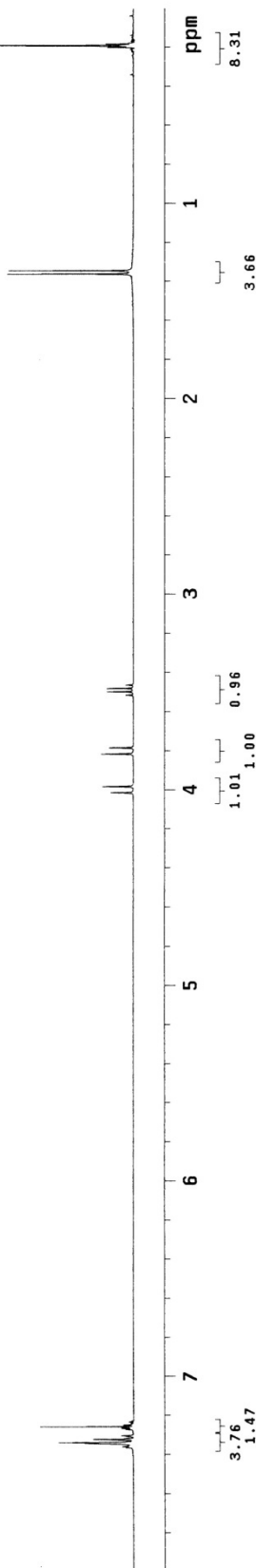
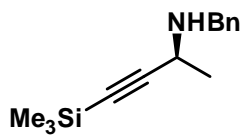
```



TPL-R-33-ag9
STANDARD 1H OBSERVE

exp3 s2pu1

date	Apr 30 2009	temp	not used	SPECIAL
solvent	cdcl3	gain	not used	
file	/export/home/~	spin	20	
nmr1	walnut/autoc~	hst	0.008	
30.04.09/0120	pw90	13.800		
ACQUISITION	alif	20.000		
sw	6402.0	il	FLAGS	
at	4.000	in	n	
np	51216	dp	y	
fb	not used	hs	nn	
bs	16	fn	not used	
dl	1.000	sp	DISPLAY	
nt	8	tp	1.5	
ct	TRANSMITTER	wf	3186.9	
tn	H1	rf	3784.3	
sf	400.083	rfp	2984.6	
tof	424.3	p	-188.2	
tpwr	58	lp	-83.7	
pw	6.900	plot		
DECOUPLER	C13	wc	250	
dn	0	sc	0	
dof	nnn	vs	16	
dm	c	th	3	
dmm	44	al	cdc	ph
dpwr				
dmf	18500			

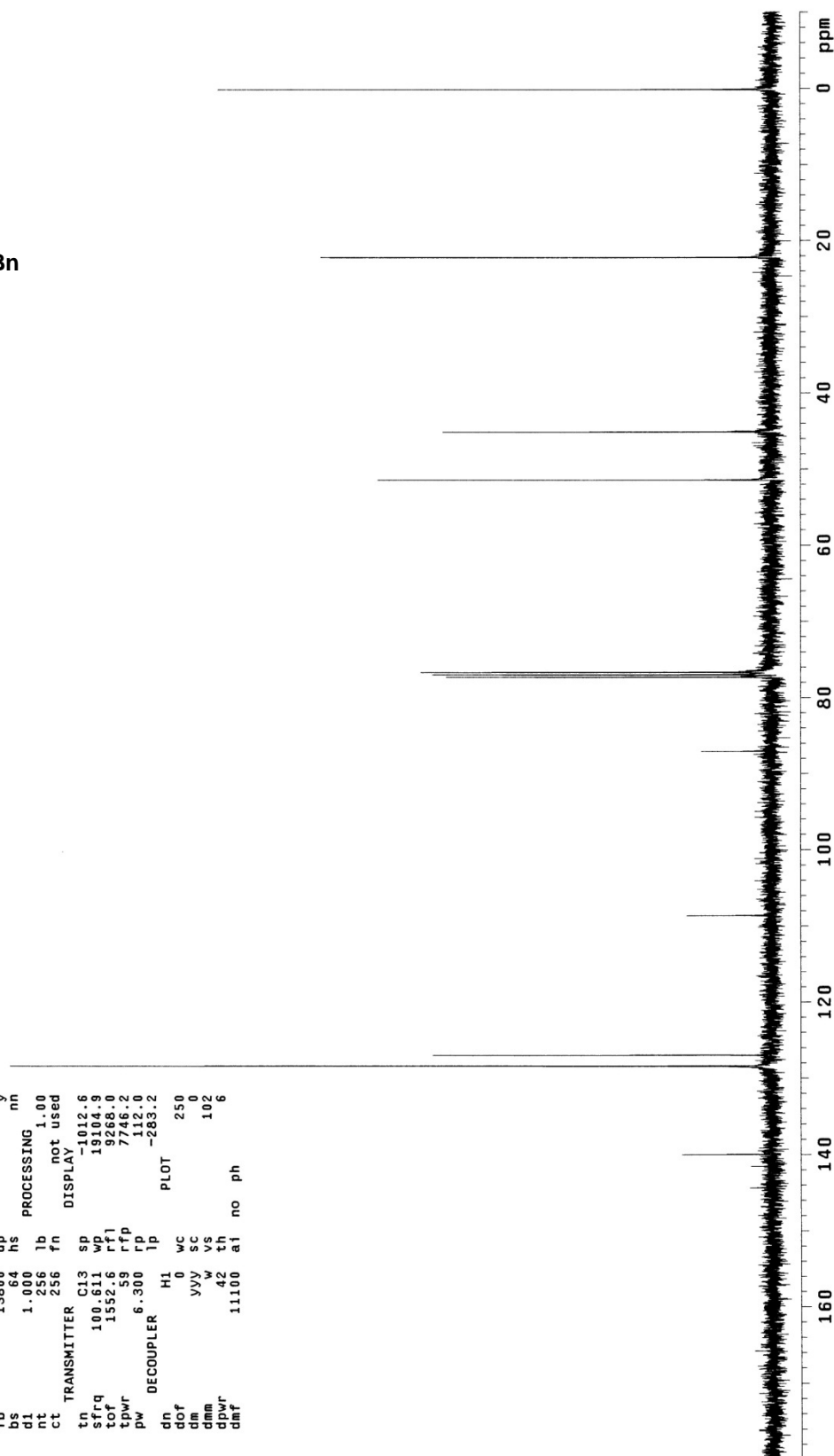
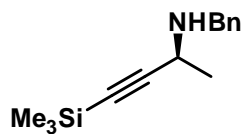


TPL-R-33-28
STANDARD 1H OBSERVE

```

exp3  szpul
SAMPLE
date   Apr 30 2009    temp    not used
solvent cdc13         gain    not used
file   /export/home/~ spin    not used
nmr1/walkup/auto/~ hst      0.008
30.04.09/0214 pw90      12.600
ACQUISITION alfa      20.000
sw      25125.6      FLAGS
at      1.199      fl      n
np      60270      in      n
fb      13800      dp      y
bs      64      hs      nn
dl      1.000      nt      1.00
nt      256      lb      not used
ct      256      fn      not used
PROCESSING
TRANSMITTER C13 sp      -1012.6
tn      100.611      wp      19104.9
sfrq      1552.6      rf1      9268.0
tof      59      rfp      7746.2
tpwr      6.300      rp      112.0
pw      DECOUPLER      lp      -283.2
dn      H1      PLOT
dof      0      wc      250
dm      yvy      sc      0
dmm      w      vs      102
dpwr      42      th      6
dnt      11100      al      no      ph

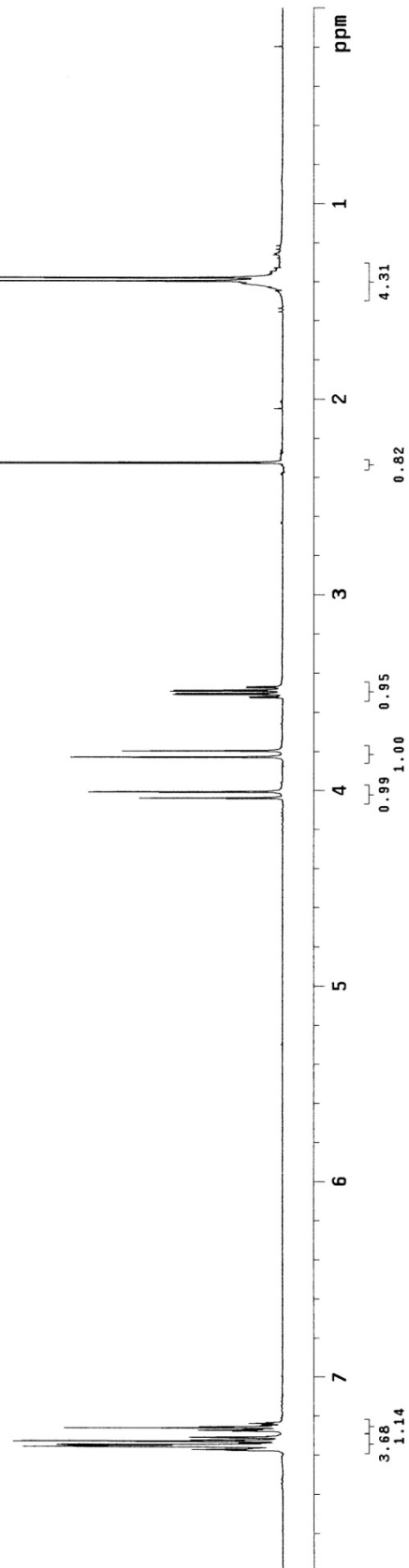
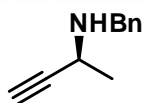
```



TPL-R-35-carbon
STANDARD 1H OBSERVE

exp3 s2pu1

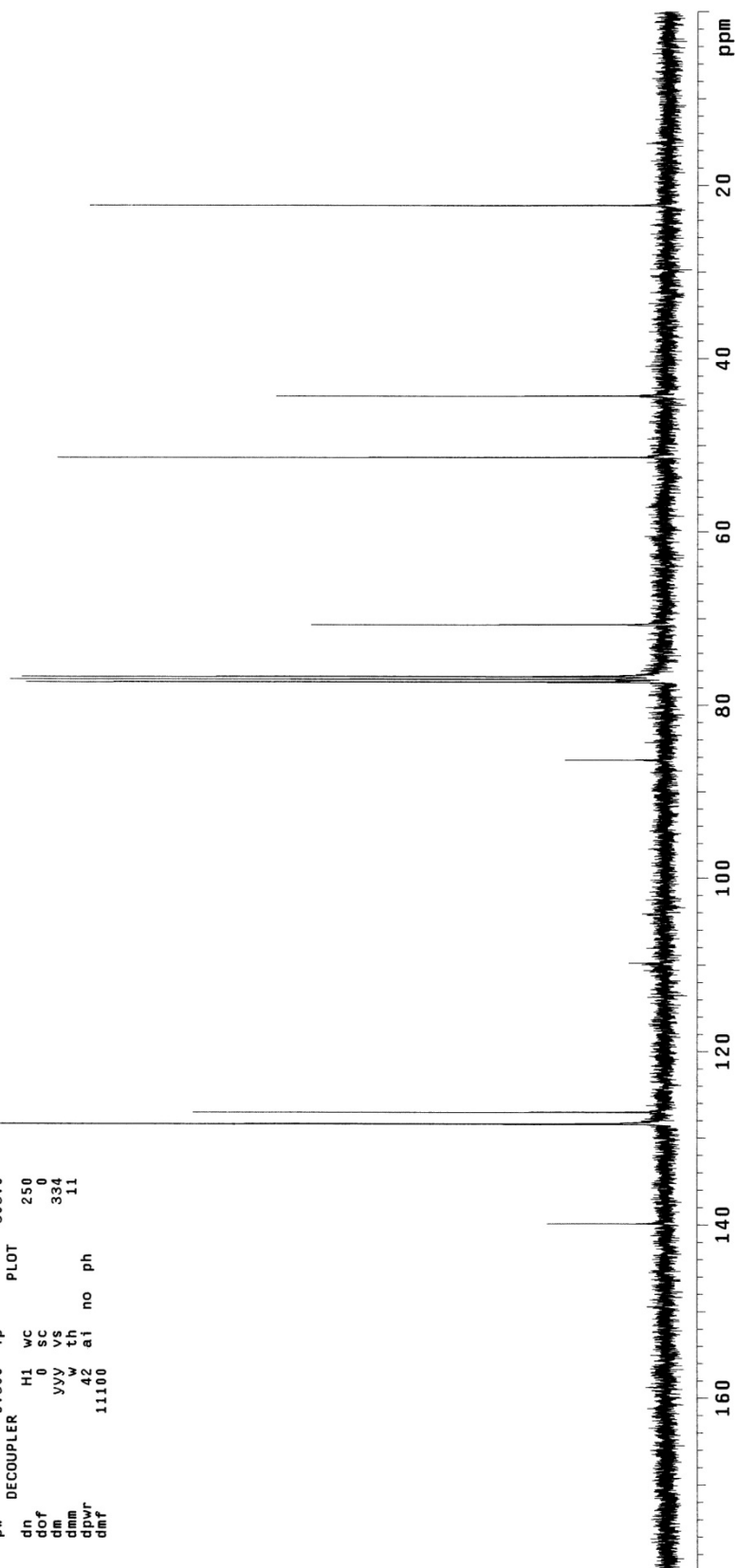
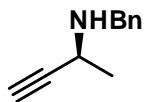
SAMPLE		SPECIAL	
date	May 4 2009	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
nmr1/vnmrsvs/data/~	hst	0.008	
/Kerr/TPL-R-35-car~	pw90	13.800	
bon/PROTON.fid	alfa	20.000	
ACQUISITION		FLAGS	
sw	6402.0	il	n
at	4.000	in	n
np	51216	dp	y
fb	not used	hs	nn
bs	16		
di	1.000	fn	not used
nt	8		
ct	8	sp	-0.3
tn	3196.5	wp	3704.1
sfreq	400.083	rfl	2904.6
tof	424.3	rfp	-163.5
tpwr	58	lp	-87.0
pw	6.900		
DECOUPLER		WC	250
dn	C13	SC	0
dof	0	VS	40
dm	nnn	th	54
dmm	c	ai	cdc
dpwr	44		ph
dmf	18500		

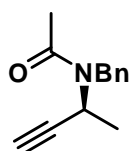
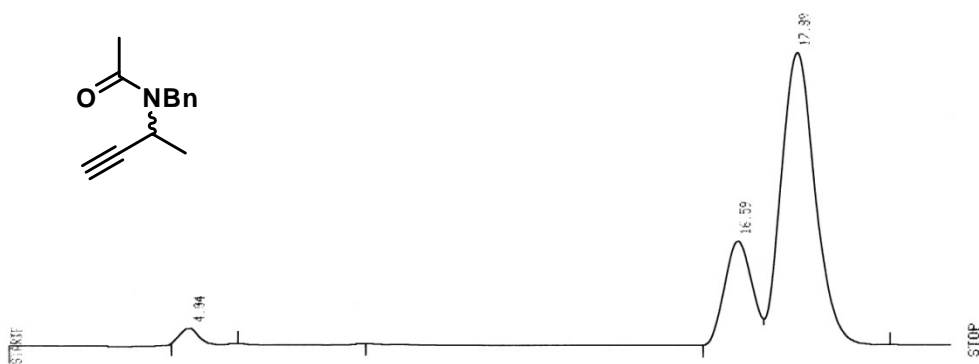


TPL-R-35-carbon
STANDARD 1H OBSERVE

exp3 s2pu1

SAMPLE		SPECIAL	
date	May 4 2009	temp	not used
solvent	cdc13	gain	not used
file	/export/home/~	spin	not used
nmr1/vnmrSYS/data/~	hst	0.008	
/Kerr/TPL-R-35-car~	pw90	12.600	
bon/CARBON.fid	alfa	20.000	
ACQUISITION			
sw	25125.6	fl	n
at	1.199	in	n
np	60270	dp	y
fb	13800	hs	nn
bs	64		
di	1.000	lb	1.00
nt	1000	fn	not used
ct	832	display	-1.3
TRANSMITTER			
tn	c13	wp	18082.0
strq	100.611	rfl	9266.5
tor	1352.6	rfp	7746.2
tpwr	59	rp	111.1
pw	6.300	lp	-303.0
DECOUPLER			
dn	H1	wc	250
dof	0	sc	0
dm	yyv	vc	334
dmm	42	th	11
dpr	42	at	no
dpr	11100	ph	

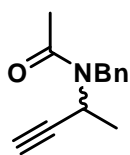
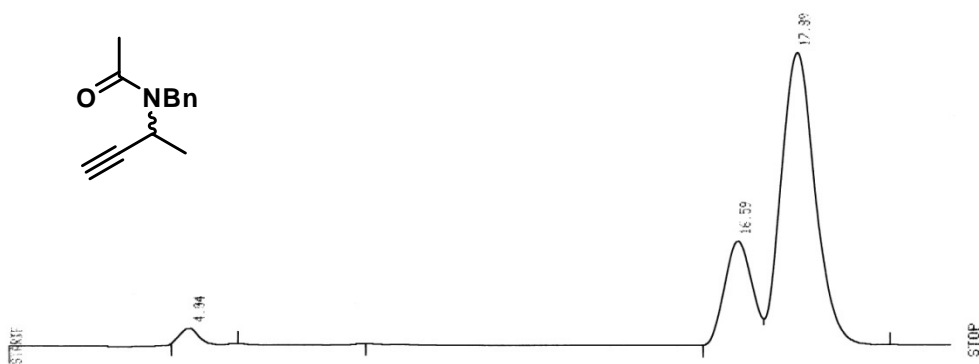




RUN # 12

AREA%	RT	AREA	TYPE	AR/HT	AREA%
4.85	4.85	5413	PB	0.492	2.362
17.83	17.83	389186	PB	0.855	97.638

TOTAL AREA= 394600
MUL FACTOR= 1.0000E+00



RUN # 13

AREA%	RT	AREA	TYPE	AR/HT	AREA%
4.94	4.94	12676	BB	0.485	2.394
16.39	16.39	112768	PV	0.731	22.095
17.39	17.39	398928	VB	0.887	75.501

TOTAL AREA= 528350
MUL FACTOR= 1.0000E+00