Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Diastereoselective Building up Polycyclic Tetrahydrofurans via Tandem Annulation of 1,*n*-Enynes with Aliphatic Acids

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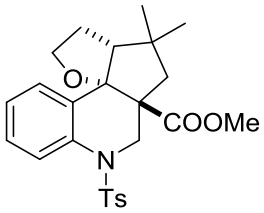
Contents

1. General information

^{1}H NMR spectra were recorded on Bruker 400 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for $CDCl_3$. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; q, quartet; m, multiplet. The coupling constants, *J*, are reported in Hertz (Hz). ^{13}C NMR spectra were obtained at Bruker 100 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in $CDCl_3$). $CDCl_3$ was used as the NMR solvent. APEX II (Bruker Inc.) was used for HR-MS and ESI-MS. IR spectra were recorded by a Nicolet 5MX-S infrared spectrometer. Flash column chromatography was performed over silica gel 200-300. All reagents were weighed and handled in air at room temperature. All reagents were purchased from Alfa, Acros, Aldrich, and TCI and used without further purification.

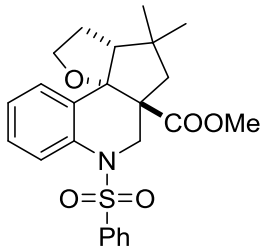
2. General procedure and characterization data for product 3 and 5

To a mixture of enyne **1** (0.2 mmol), alkyl carboxylic acid **2** or **4** (0.4 mmol), $AgNO_3$ (10.2 mg, 30 mol %) and $K_2S_2O_8$ (108 mg, 0.4 mmol), MeCN (2.0 mL) and H_2O (1.0 mL) were added under air at room temperature. The resulting mixture was stirred at 100 ℃ for 1 hour. After the mixture was cooled to room temperature, EtOAc (15.0 mL) and H_2O (5.0 mL) were added sequentially. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated in vacuo to give the crude products. The residue was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether) to give the pure product **3** or **5**.

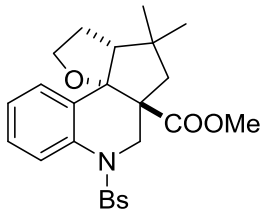


Methyl 4,4-dimethyl-7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3aa). (59 mg, 65%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2872, 1736, 1483, 1452, 1352, 1233, 1165, 1084, 1032, 934 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 1H), 7.44 (d, *J* = 8.2 Hz, 2H), 7.22-7.17 (m, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.05-6.99 (m, 2H), 4.46 (d, *J* = 10.6 Hz, 1H), 3.77 (d, *J* = 10.6 Hz, 1H), 3.41 (s, 3H), 3.31-3.25 (m, 1H), 2.97-2.91 (m, 2H), 2.32 (s, 3H), 2.19-2.12 (m, 2H), 1.91 (d, *J* = 13.8 Hz, 1H), 1.87 (d, *J* = 13.8 Hz, 1H), 1.04 (s, 3H), 0.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 143.0, 135.4, 135.0, 131.2, 128.7, 128.1, 127.5, 124.0, 123.9, 123.3, 91.0, 64.7, 56.0,

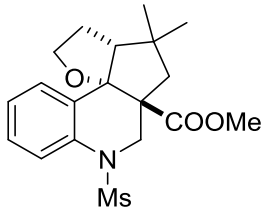
52.0, 51.8, 51.6, 48.7, 41.4, 31.7, 29.3, 28.6, 21.4; HRMS (ESI) calcd for $C_{25}H_{29}NNaO_5S$ [M + Na^+], 478.1659; found: 478.1641.



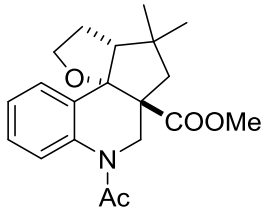
Methyl 4,4-dimethyl-7-(phenylsulfonyl)-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ba). (59 mg, 67%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 3067, 3036, 2951, 2872, 1734, 1483, 1447, 1352, 1231, 1167, 1082, 1032, 933 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.2 Hz, 1H), 7.57-7.54 (m, 2H), 7.48-7.43 (m, 1H), 7.35-7.31 (m, 2H), 7.26-7.21 (m, 1H), 7.06-7.04 (m, 2H), 4.48 (d, J = 10.6 Hz, 1H), 3.78 (d, J = 10.6 Hz, 1H), 3.42 (s, 3H), 3.25 (q, J = 8.4 Hz, 1H), 2.98 (dd, J = 7.0, 1.2 Hz, 1H), 2.93-2.86 (m, 1H), 2.22-2.09 (m, 2H), 1.93 (d, J = 13.2 Hz, 1H), 1.88 (d, J = 13.2 Hz, 1H), 1.05 (s, 3H), 0.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 137.7, 135.3, 132.3, 131.4, 128.1, 127.5, 124.2, 124.0, 123.6, 91.0, 64.8, 56.1, 51.9, 51.8, 51.7, 48.7, 41.4, 31.7, 29.3, 28.5; HRMS (ESI) calcd for $C_{24}H_{27}NNaO_5S$ [M + Na^+], 464.1502; found: 464.1502.



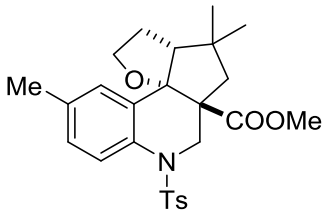
Methyl 7-((4-bromophenyl)sulfonyl)-4,4-dimethyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ca). (56 mg, 54%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2870, 1734, 1472, 1389, 1358, 1231, 1169, 1088, 1032 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.2 Hz, 1H), 7.47 (d, J = 8.6 Hz, 2H), 7.37 (d, J = 8.6 Hz, 2H), 7.27-7.24 (m, 1H), 7.11-7.04 (m, 2H), 4.45 (d, J = 10.6 Hz, 1H), 3.69 (d, J = 10.6 Hz, 1H), 3.42 (s, 3H), 3.28 (q, J = 8.4 Hz, 1H), 2.98 (dd, J = 6.8, 1.8 Hz, 1H), 2.92-2.86 (m, 1H), 2.23-2.11 (m, 2H), 1.93 (d, J = 13.2 Hz, 1H), 1.87 (d, J = 13.2 Hz, 1H), 1.06 (s, 3H), 0.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 136.4, 135.0, 131.9, 131.3, 129.0, 128.3, 127.2, 124.8, 124.1, 124.0, 91.1, 64.8, 56.2, 51.9, 51.7, 48.8, 41.4, 31.7, 29.3, 28.5; HRMS (ESI) calcd for $C_{24}H_{26}BrNNaO_5S$ [M + Na^+], 542.0607; found: 542.0619.



Methyl 4,4-dimethyl-7-(methylsulfonyl)-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3da). (42 mg, 56%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.3); IR (neat): ν_{max} 2951, 2870, 1734, 1480, 1456, 1352, 1230, 1168, 1090 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 8.4, 1.0 Hz, 1H), 7.29-7.24 (m, 1H), 7.20 (dd, J = 7.6, 1.6 Hz, 1H), 7.12 (td, J = 7.6, 1.0 Hz, 1H), 4.40 (d, J = 10.6 Hz, 1H), 3.85 (q, J = 8.4 Hz, 1H), 3.83 (d, J = 10.6 Hz, 1H), 3.67-3.61 (m, 1H), 3.44 (s, 3H), 3.13 (d, J = 7.0 Hz, 1H), 2.76 (s, 3H), 2.45-2.31 (m, 2H), 2.04 (d, J = 13.6 Hz, 1H), 1.99 (d, J = 13.6 Hz, 1H), 1.13 (s, 3H), 1.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 135.3, 131.7, 128.5, 124.3, 124.1, 123.9, 91.8, 65.4, 56.3, 52.0, 51.9, 51.7, 48.8, 41.5, 35.5, 31.8, 29.3, 28.8; HRMS (ESI) calcd for $C_{19}H_{26}NO_5S$ [M + H^+], 380.1526; found: 380.1518.

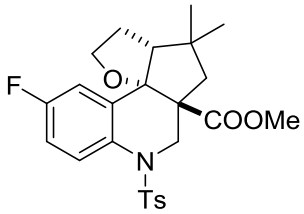


Methyl 7-acetyl-4,4-dimethyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ea). (46 mg, 68%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:3, R_f = 0.2); IR (neat): ν_{max} 2949, 2870, 1732, 1653, 1485, 1371, 1329, 1290, 1215, 1150, 1032 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.50-7.26 (br, 1H), 7.27-7.23 (m, 1H), 7.22-7.19 (m, 1H), 7.13-7.08 (m, 1H), 4.31 (d, J = 11.8 Hz, 1H), 3.95 (d, J = 11.8 Hz, 1H), 3.84 (q, J = 8.4 Hz, 1H), 3.67-3.60 (m, 1H), 3.54 (s, 3H), 3.12 (d, J = 7.0 Hz, 1H), 2.39-2.28 (m, 2H), 2.25 (s, 3H), 2.06 (d, J = 13.2 Hz, 1H), 2.01 (d, J = 13.2 Hz, 1H), 1.13 (s, 3H), 1.08 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3, 170.7, 127.7, 125.4, 123.9, 92.6, 65.9, 56.9, 51.6, 51.5, 50.5, 49.1, 31.7, 29.4, 28.3, 23.7; HRMS (ESI) calcd for $C_{20}H_{25}NNaO_4$ [M + Na^+], 366.1676; found: 366.1677.

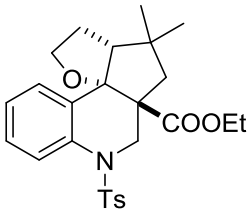


Methyl 4,4,10-trimethyl-7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate

(3ga). (67 mg, 72%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2870, 1736, 1490, 1350, 1233, 1209, 1165, 1084, 1034, 935 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 1H), 7.44 (d, *J* = 8.2 Hz, 2H), 7.12 (d, *J* = 8.2 Hz, 2H), 7.01 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.76 (dd, *J* = 8.4, 3.0 Hz, 1H), 4.43 (d, *J* = 10.8 Hz, 1H), 3.72 (d, *J* = 10.8 Hz, 1H), 3.46 (s, 3H), 3.25 (q, *J* = 8.4 Hz, 1H), 2.93-2.87 (m, 2H), 2.35 (s, 3H), 2.29 (s, 3H), 2.16-2.11 (m, 2H), 1.92 (dd, *J* = 13.6, 2.2 Hz, 1H), 1.87 (d, *J* = 13.6 Hz, 1H), 1.04 (s, 3H), 0.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 142.8, 134.9, 132.8, 131.1, 128.7, 128.6, 127.5, 124.6, 123.9, 91.1, 64.6, 56.1, 51.9, 51.8, 51.5, 48.7, 41.3, 31.7, 29.3, 28.6, 21.4, 20.9; HRMS (ESI) calcd for $C_{26}H_{31}NNaO_5S$ [M + Na^+], 492.1815; found: 492.1806.

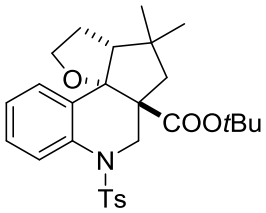


Methyl 10-fluoro-4,4-dimethyl-7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ha). (53 mg, 56%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2953, 2872, 1734, 1458, 1352, 1165, 1034 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.87 (dd, *J* = 9.2, 5.0 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 7.02 (td, *J* = 8.6, 3.0 Hz, 1H), 6.76 (dd, *J* = 8.4, 3.0 Hz, 1H), 4.43 (d, *J* = 10.8 Hz, 1H), 3.72 (d, *J* = 10.8 Hz, 1H), 3.46 (s, 3H), 3.25 (q, *J* = 8.4 Hz, 1H), 2.93-2.87 (m, 2H), 2.35 (s, 3H), 2.16-2.11 (m, 2H), 1.92 (dd, *J* = 13.6, 2.2 Hz, 1H), 1.87 (d, *J* = 13.6 Hz, 1H), 1.04 (s, 3H), 0.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 158.9 (d, $J_{C\text{-}F}$ = 243.5 Hz), 143.1, 134.4, 133.7 (d, $J_{C\text{-}F}$ = 6.8 Hz), 131.3, 128.7, 127.6, 126.0 (d, $J_{C\text{-}F}$ = 7.8 Hz), 114.6 (d, $J_{C\text{-}F}$ = 22.0 Hz), 111.1 (d, $J_{C\text{-}F}$ = 22.8 Hz), 90.6, 64.9, 56.1, 52.0, 51.5, 48.6, 41.3, 31.7, 29.2, 28.4, 21.4; HRMS (ESI) calcd for $C_{25}H_{28}FNNaO_5S$ [M + Na^+], 496.1564; found: 496.1554.



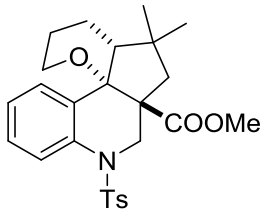
Ethyl 4,4-dimethyl-7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ia). (58 mg, 62%, dr = 9:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 3067, 3034, 2957, 2870, 1728, 1483, 1458, 1352, 1229, 1206, 1165, 1084, 1032, 935 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.2 Hz, 1H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.24-7.19 (m, 1H), 7.12 (d, *J* = 8.2 Hz, 2H), 7.06-7.00 (m, 2H), 4.49 (d, *J* = 10.4 Hz, 1H), 3.95-3.80 (m, 2H), 3.79 (d, *J* = 10.4 Hz, 1H), 3.30 (q, *J* = 8.4 Hz, 1H), 2.99-2.92 (m, 2H), 2.34 (s, 3H), 2.22-2.12 (m, 2H), 1.93 (d, *J* = 13.2 Hz, 1H), 1.89 (d, *J* = 13.2 Hz, 1H), 1.07 (s,

3H), 0.98 (s, 3H), 0.95 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 142.9, 135.6, 135.0, 131.3, 128.7, 128.0, 127.5, 124.0, 123.2, 91.1, 64.7, 60.7, 56.0, 52.1, 51.7, 48.7, 41.5, 31.8, 29.4, 28.6, 21.4, 13.6; HRMS (ESI) calcd for $C_{26}H_{31}NNaO_5S$ [M + Na^+], 492.1815; found: 492.1816.



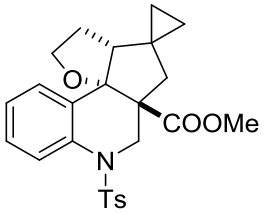
***Tert*-butyl**

4,4-dimethyl-7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ja). (57 mg, 58%, dr = 2:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 3067, 3034, 2974, 2870, 1718, 1458, 1352, 1165, 1030, 935 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 8.2 Hz, 1H), 7.43 (d, J = 8.4 Hz, 2H), 7.21-7.17 (m, 1H), 7.10 (d, J = 8.4 Hz, 2H), 7.03-6.98 (m, 2H), 4.45 (d, J = 10.2 Hz, 1H), 3.71 (d, J = 10.2 Hz, 1H), 3.29-3.23 (m, 1H), 2.94-2.88 (m, 2H), 2.32 (s, 3H), 2.20-2.09 (m, 2H), 1.88 (d, J = 13.4 Hz, 1H), 1.83 (d, J = 13.4 Hz, 1H), 1.11 (s, 3H), 1.09 (s, 9H), 0.95 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.3, 142.9, 135.8, 135.2, 131.6, 128.7, 128.0, 127.5, 124.1, 123.1, 91.3, 81.5, 64.6, 56.7, 52.4, 52.1, 48.8, 41.6, 31.9, 29.5, 28.6, 27.3, 21.4. HRMS (ESI) calcd for $C_{28}H_{35}NNaO_5S$ [M + Na^+], 520.2128; found: 520.2128.

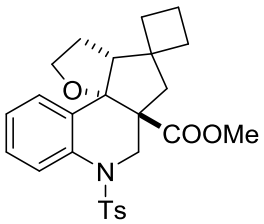


Methyl

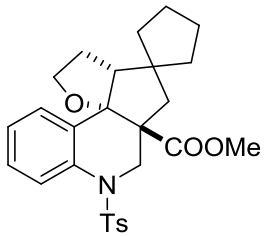
(12bR)-5,5-dimethyl-8-tosyl-2,3,4,4a,5,6,7,8-octahydro-6aH-pyrano[2',3':2,3]cyclopenta[1,2-c]quinoline-6a-carboxylate (3ma). (52 mg, 56%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:7, R_f = 0.5); IR (neat): ν_{max} 2951, 2854, 1734, 1352, 1296, 1258, 1165, 1095 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (dd, J = 8.4, 1.0 Hz, 1H), 7.50 (d, J = 8.4 Hz, 2H), 7.31 (dd, J = 7.6, 1.2 Hz, 1H), 7.27-7.24 (m, 1H), 7.15 (d, J = 8.4 Hz, 2H), 7.07 (td, J = 7.6, 1.0 Hz, 1H), 4.35 (d, J = 10.4 Hz, 1H), 3.84 (d, J = 10.4 Hz, 1H), 3.39 (s, 3H), 2.76 (d, J = 5.6 Hz, 1H), 2.69 (td, J = 13.6, 3.2 Hz, 1H), 2.41 (dd, J = 11.8, 5.8 Hz, 1H), 2.34 (s, 3H), 2.13-2.03 (m, 1H), 1.96 (d, J = 13.2 Hz, 1H), 1.92 (d, J = 13.2 Hz, 1H), 1.84-1.68 (m, 2H), 1.23 (s, 3H), 1.17-1.13 (m, 1H), 1.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 142.9, 135.8, 135.5, 131.5, 129.0, 128.1, 127.5, 127.3, 124.4, 122.9, 81.6, 59.6, 58.4, 51.8, 50.0, 48.1, 44.3, 42.5, 31.8, 30.0, 21.4, 21.3, 19.9; HRMS (ESI) calcd for $C_{26}H_{31}NNaO_5S$ [M + Na^+], 492.1815; found: 492.1815.



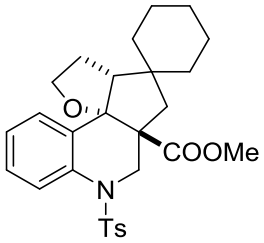
Methyl 7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclopropane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3ab). (14 mg, 16%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2949, 2874, 1732, 1485, 1456, 1350, 1230, 1200, 1165, 1090, 1038 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.4 Hz, 1H), 7.50 (d, J = 8.2 Hz, 2H), 7.21-7.17 (m, 1H), 7.14 (d, J = 8.2 Hz, 2H), 7.03-6.98 (m, 2H), 4.54 (d, J = 10.6 Hz, 1H), 3.85 (d, J = 10.6 Hz, 1H), 3.49-3.43 (m, 1H), 3.43 (s, 3H), 3.34-3.29 (m, 1H), 2.97 (d, J = 6.8 Hz, 1H), 2.34 (s, 3H), 2.21 (d, J = 12.4 Hz, 1H), 2.18-2.08 (m, 1H), 1.86-1.81 (m, 1H), 1.47 (d, J = 12.4 Hz, 1H), 0.58-0.51 (m, 3H), 0.49-0.43 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.0, 143.0, 135.5, 135.2, 131.2, 128.8, 128.1, 127.6, 124.0, 123.4, 123.3, 90.7, 66.6, 57.9, 51.9, 50.5, 48.9, 43.8, 32.2, 25.6, 21.4, 19.0, 7.6; HRMS (ESI) calcd for $C_{25}H_{27}NNaO_5S$ [M + Na^+], 476.1502; found: 476.1485.



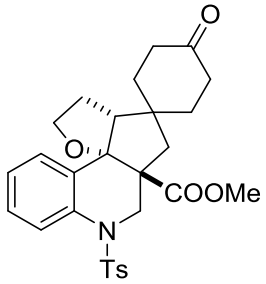
Methyl 7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclobutane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3ac). (39 mg, 42%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2928, 2870, 1734, 1485, 1458, 1350, 1233, 1165, 1051, 1016, 920 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.2 Hz, 1H), 7.49 (d, J = 8.2 Hz, 2H), 7.21-7.16 (m, 1H), 7.14 (d, J = 8.2 Hz, 2H), 7.06-6.99 (m, 2H), 4.54 (d, J = 10.6 Hz, 1H), 3.82 (d, J = 10.6 Hz, 1H), 3.40 (s, 3H), 3.24 (td, J = 8.4, 3.6 Hz, 1H), 3.16 (q, J = 8.4 Hz, 1H), 3.10 (d, J = 6.8 Hz, 1H), 2.36 (d, J = 12.4 Hz, 1H), 2.34 (s, 3H), 2.26-2.21 (m, 1H), 2.19-2.11 (m, 2H), 1.99-1.92 (m, 1H), 1.91 (d, J = 12.4 Hz, 1H), 1.88-1.80 (m, 1H), 1.77-1.66 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 143.0, 135.4, 135.1, 131.5, 128.8, 127.9, 127.6, 123.9, 123.4, 123.3, 90.2, 66.4, 56.8, 52.3, 51.7, 51.0, 49.0, 47.0, 38.7, 31.2, 30.2, 21.4, 16.6; HRMS (ESI) calcd for $C_{26}H_{29}NNaO_5S$ [M + Na^+], 490.1659; found: 490.1645.



Methyl 7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclopentane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3ad). (54 mg, 56%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2872, 1734, 1481, 1456, 1350, 1233, 1165, 1090, 1022 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 8.2 Hz, 1H), 7.44 (d, J = 8.2 Hz, 2H), 7.21-7.17 (m, 1H), 7.11 (d, J = 8.2 Hz, 2H), 7.06-6.99 (m, 2H), 4.46 (d, J = 10.6 Hz, 1H), 3.78 (d, J = 10.6 Hz, 1H), 3.41 (s, 3H), 3.33 (q, J = 8.4 Hz, 1H), 3.07-3.02 (m, 2H), 2.32 (s, 3H), 2.26-2.16 (m, 1H), 2.10-2.03 (m, 1H), 1.94 (d, J = 12.9 Hz, 1H), 1.86 (d, J = 12.9 Hz, 1H), 1.61-1.50 (m, 6H), 1.45-1.42 (m, 1H), 1.35-1.31 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 143.0, 135.5, 135.0, 131.4, 128.7, 128.0, 127.6, 123.9, 123.8, 123.4, 90.3, 65.3, 56.4, 52.8, 51.8, 51.5, 50.7, 46.4, 41.4, 37.7, 29.7, 23.2, 21.4; HRMS (ESI) calcd for $C_{27}H_{31}NNaO_5S$ [M + Na^+], 504.1815; found: 504.1801.

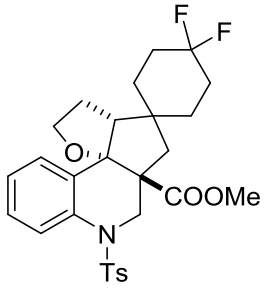


Methyl 7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclohexane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3ae). (57 mg, 58%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 3065, 3034, 2930, 2855, 1734, 1487, 1456, 1352, 1233, 1167, 1026 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, J = 8.2 Hz, 1H), 7.43 (d, J = 8.2 Hz, 2H), 7.22-7.17 (m, 1H), 7.10 (d, J = 8.2 Hz, 2H), 7.06-6.99 (m, 2H), 4.47 (d, J = 10.6 Hz, 1H), 3.79 (d, J = 10.6 Hz, 1H), 3.38 (s, 3H), 3.20 (q, J = 8.4 Hz, 1H), 2.94-2.88 (m, 1H), 2.85 (d, J = 7.4 Hz, 1H), 2.32 (s, 3H), 2.27-2.10 (m, 3H), 1.69 (d, J = 13.4 Hz, 1H), 1.59-1.49 (m, 4H), 1.36-1.08 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 143.0, 135.5, 135.0, 131.4, 128.7, 128.0, 127.6, 124.0, 123.4, 89.8, 64.7, 56.0, 53.4, 51.9, 51.8, 45.1, 43.0, 38.2, 38.0, 28.1, 25.7, 23.8, 23.3, 21.4; HRMS (ESI) calcd for $C_{28}H_{33}NNaO_5S$ [M + Na^+], 518.1972; found: 518.1955.

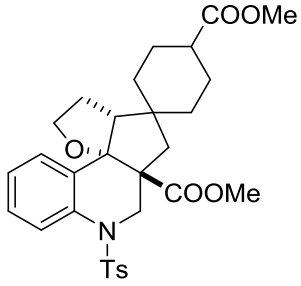


Methyl 4-oxo-7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclohexane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3af). (43 mg, 42%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5); IR (neat): ν_{max} 2951, 2928, 2872, 1734, 1717, 1489, 1456, 1350, 1234, 1165, 1092, 1036 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.2 Hz, 1H), 7.42 (d, J = 8.2 Hz, 2H), 7.25-7.21 (m, 1H), 7.11 (d, J = 8.2

Hz, 2H), 7.05-7.01 (m, 2H), 4.51 (d, *J* = 10.6 Hz, 1H), 3.81 (d, *J* = 10.6 Hz, 1H), 3.40 (s, 3H), 3.23 (q, *J* = 8.4 Hz, 1H), 3.05 (d, *J* = 7.0Hz, 1H), 2.95-2.88 (m, 1H), 2.39 (d, *J* = 13.2 Hz, 1H), 2.32 (s, 3H), 2.35-2.29 (m, 4H), 2.27-2.12 (m, 2H), 2.02-1.98 (m, 1H), 1.96 (d, *J* = 13.2 Hz, 1H), 1.81-1.68 (m, 2H), 1.65-1.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.8, 173.5, 143.1, 135.5, 134.7, 130.5, 128.8, 128.5, 127.5, 124.4, 123.9, 123.6, 90.2, 64.4, 56.1, 52.1, 51.4, 44.3, 42.0, 38.9, 38.5, 37.8, 37.5, 28.2, 21.4; HRMS (ESI) calcd for $C_{28}H_{31}NNaO_6S$ [M + Na^+], 532.1764; found: 532.1748.

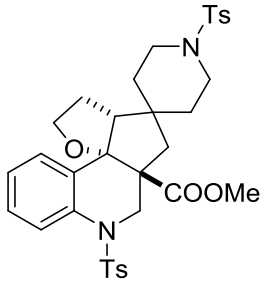


Methyl 4,4-difluoro-7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclohexane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3ag). (60 mg, 56%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 3067, 3034, 2955, 2874, 1734, 1483, 1449, 1375, 1352, 1235, 1167, 1117, 1090, 1038, 989 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.2 Hz, 1H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.25-7.20 (m, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.05-7.01 (m, 2H), 4.47 (d, *J* = 10.6 Hz, 1H), 3.77 (d, *J* = 10.6 Hz, 1H), 3.39 (s, 3H), 3.21 (q, *J* = 8.4 Hz, 1H), 2.30-2.96 (m, 1H), 2.92-2.87 (m, 1H), 2.32 (s, 3H), 2.21-2.16 (m, 3H), 2.05-1.94 (m, 2H), 1.80-1.52 (m, 6H), 1.35-1.32 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 143.1, 135.5, 134.8, 130.6, 128.7, 128.4, 127.5, 124.3, 123.9, 123.5, 122.6 (t, $J_{C\text{-}F}$ = 238.6 Hz), 90.3, 64.5, 56.1, 52.3, 52.1, 51.4, 44.0, 41.8, 33.9-33.7 (m), 31.8-30.8 (m), 28.2, 21.4; HRMS (ESI) calcd for $C_{28}H_{31}F_2NNaO_5S$ [M + Na^+], 554.1783; found: 554.1761.

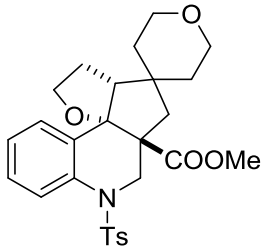


Dimethyl 7'-tosyl-3',3a',6',7'-tetrahydro-2'H-spiro[cyclohexane-1,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-4,5a'(5'H)-dicarboxylate (3ah). (72 mg, 65%, dr = 2.5:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5); IR (neat): ν_{max} 2951, 2872, 1734, 1732, 1597, 1478, 1456, 1350, 1238, 1165, 1078, 1036 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.2 Hz, 1H), 7.43 (d, *J* = 8.2 Hz, 2H), 7.22-7.18 (m, 1H), 7.10 (d, *J* = 8.2 Hz, 2H), 7.02-6.99 (m, 2H), 4.47 (d, *J* = 10.6 Hz, 1H), 3.77 (d, *J* = 10.6 Hz, 1H), 3.65 (s, 3H), 3.39 (s, 3H), 3.20 (q, *J* = 8.4 Hz, 1H), 2.93-2.87 (m, 2H), 2.32 (s, 3H), 2.24-2.16 (m, 4H), 1.88-1.79 (m, 2H),

1.69-1.63 (m, 2H), 1.55-1.40 (m, 2H), 1.38-1.32 (m, 2H), 1.27-1.20 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.0, 173.6, 143.0, 135.5, 134.9, 130.9, 128.7, 128.2, 127.5, 124.1, 124.0, 123.4, 89.8, 64.6, 56.0, 53.2, 51.9, 51.6, 44.4, 42.4, 42.2, 37.0, 36.7, 28.0, 26.3, 25.7, 21.4; HRMS (ESI) calcd for $C_{30}H_{35}NNaO_7S$ [M + Na^+], 576.2026; found: 576.2005.

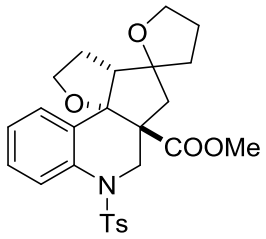


Methyl 1',7-ditosyl-3,3a,6,7-tetrahydro-2H-spiro[furo[2',3':2,3]cyclopenta[1,2-c]quinoline-4,4'-piperidine]-5a(5H)-carboxylate (3ai). (67 mg, 52%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:3, R_f = 0.4); IR (neat): ν_{max} 3067, 3032, 2951, 2878, 2853, 1734, 1597, 1483, 1350, 1237, 1165, 1092, 1040, 1020, 950, 914 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 8.2 Hz, 1H), 7.60 (d, J = 8.2 Hz, 2H), 7.36 (d, J = 8.2 Hz, 2H), 7.32 (d, J = 8.2 Hz, 2H), 7.23-1.19 (m, 1H), 7.07 (d, J = 8.2 Hz, 2H), 7.02-6.95 (m, 2H), 4.37 (d, J = 10.6 Hz, 1H), 3.64 (d, J = 10.6 Hz, 2H), 3.57 (d, J = 12.4 Hz, 1H), 3.29 (s, 3H), 3.13 (q, J = 8.4 Hz, 1H), 2.94 (d, J = 5.6 Hz, 1H), 2.85-2.80 (m, 1H), 2.44 (s, 3H), 2.30 (s, 3H), 2.27-2.21 (m, 2H), 2.18-2.13 (m, 2H), 1.85 (d, J = 13.6 Hz, 1H), 1.78-1.71 (m, 1H), 1.69-1.58 (m, 2H), 1.49 (d, J = 13.6 Hz, 1H), 1.30 (d, J = 12.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 143.6, 143.1, 135.6, 134.7, 132.6, 130.4, 129.7, 128.7, 128.5, 127.6, 127.4, 124.5, 123.9, 123.6, 90.1, 64.5, 56.3, 53.0, 52.0, 51.1, 44.0, 43.5, 43.1, 42.0, 36.8, 36.5, 27.8, 21.5, 21.4; HRMS (ESI) calcd for $C_{34}H_{38}N_2NaO_7S_2$ [M + Na^+], 673.2013; found: 673.1989.

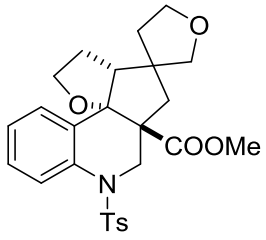


Methyl 7-tosyl-2',3,3a,3',5',6,6',7-octahydro-2H-spiro[furo[2',3':2,3]cyclopenta[1,2-c]quinoline-4,4'-pyran]-5a(5H)-carboxylate (3aj). (56 mg, 57%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:3, R_f = 0.5); IR (neat): ν_{max} 2951, 2870, 1736, 1479, 1458, 1352, 1242, 1165, 1072 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, J = 8.2 Hz, 1H), 7.42 (d, J = 8.2 Hz, 2H), 7.24-7.19 (m, 1H), 7.09 (d, J = 8.2 Hz, 2H), 7.04-6.99 (m, 2H), 4.49 (d, J = 10.6 Hz, 1H), 3.85-3.73 (m, 2H), 3.77 (d, J = 10.6 Hz, 1H), 3.52-3.40 (m, 2H), 3.36 (s, 3H), 3.18 (q, J = 8.4 Hz, 1H), 2.96-2.88 (m, 2H), 2.31 (s, 3H), 2.29 (d, J = 13.2 Hz, 1H), 2.24-2.20 (m, 2H), 1.77 (d, J = 13.2 Hz, 1H), 1.77-1.70 (m, 1H), 1.66-1.58 (m, 1H), 1.51-1.46 (m, 1H), 1.20-1.17 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ

173.6, 143.0, 135.6, 134.9, 130.7, 128.7, 128.3, 127.5, 124.3, 123.9, 123.5, 90.1, 65.6, 64.9, 64.6, 56.2, 53.3, 52.0, 51.4, 43.1, 43.0, 38.3, 38.0, 28.0, 21.4; HRMS (ESI) calcd for $C_{27}H_{31}NNaO_6S$ [M + Na^+], 520.1764; found: 520.1751.

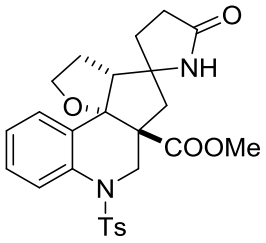


Methyl 7'-tosyl-3',3a',4,5,6',7'-hexahydro-2'H,3H-spiro[furan-2,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3ak). (62 mg, 64%, dr = 1:1). **Isomer 1:** Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:7, R_f = 0.6); IR (neat): ν_{max} 3065, 2976, 2949, 2878, 1738, 1485, 1458, 1350, 1233, 1165, 1051, 920 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.2 Hz, 1H), 7.44 (d, J = 8.2 Hz, 2H), 7.26-7.21 (m, 1H), 7.12 (d, J = 8.2 Hz, 2H), 7.06-6.99 (m, 2H), 4.49 (d, J = 10.6 Hz, 1H), 3.89-3.83 (m, 1H), 3.80 (d, J = 10.6 Hz, 1H), 3.76-3.70 (m, 1H), 3.41 (q, J = 8.4 Hz, 1H), 3.40 (s, 3H), 3.13 (d, J = 7.4 Hz, 1H), 3.05-3.00 (m, 1H), 2.33 (s, 3H), 2.26-2.20 (m, 1H), 2.19 (d, J = 13.4 Hz, 1H), 2.13-2.06 (m, 1H), 2.07 (d, J = 13.4 Hz, 1H), 1.95-1.83 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 143.0, 135.7, 134.7, 130.9, 128.8, 128.3, 127.6, 124.3, 123.7, 123.5, 90.4, 89.6, 67.8, 66.3, 55.7, 52.9, 51.9, 50.7, 47.4, 41.0, 28.4, 25.9, 21.4; HRMS (ESI) calcd for $C_{26}H_{29}NNaO_6S$ [M + Na^+], 506.1608; found: 506.1591. **Isomer 2:** Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:7, R_f = 0.5); IR (neat): ν_{max} 3065, 3032, 2950, 2872, 1734, 1483, 1458, 1350, 1231, 1165, 1053, 1018 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 8.2 Hz, 1H), 7.48 (d, J = 8.2 Hz, 2H), 7.23-7.19 (m, 1H), 7.14 (d, J = 8.2 Hz, 2H), 7.06-7.01 (m, 2H), 4.47 (d, J = 10.6 Hz, 1H), 3.85 (d, J = 10.6 Hz, 1H), 3.84-3.78 (m, 1H), 3.71-3.65 (m, 1H), 3.41 (s, 3H), 3.33 (q, J = 8.0 Hz, 1H), 3.24-3.19 (m, 2H), 2.40-2.32 (m, 5H), 2.13-2.07 (m, 1H), 1.94-1.82 (m, 4H), 1.69-1.63 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 143.1, 135.3, 135.1, 130.8, 128.8, 127.9, 127.5, 124.1, 123.2, 123.1, 92.6, 89.5, 66.1, 65.3, 55.6, 51.8, 51.6, 44.9, 33.7, 30.2, 24.9, 21.4; HRMS (ESI) calcd for $C_{26}H_{29}NNaO_6S$ [M + Na^+], 506.1608; found: 506.1591.

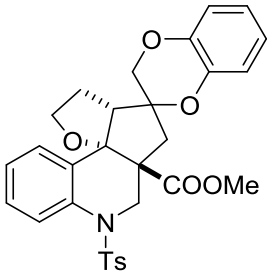


Methyl 7'-tosyl-3',3a',4,5,6',7'-hexahydro-2H,2'H-spiro[furan-3,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoline]-5a'(5'H)-carboxylate (3al). (46 mg, 48%, dr = 1:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5); IR (neat): ν_{max} 2951, 2870, 1734, 1598, 1458, 1352, 1240, 1072 cm^{-1}; **Isomer 1**: ^1H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.2 Hz, 1H), 7.42 (d, J = 8.2 Hz, 2H), 7.25-7.21 (m, 1H), 7.11 (d, J = 8.2 Hz, 2H),

7.05-7.01 (m, 2H), 4.48 (d, *J* = 10.6 Hz, 1H), 3.92-3.87 (m, 1H), 3.81-3.45 (m, 1H), 3.73 (d, *J* = 10.6 Hz, 1H), 3.63 (d, *J* = 8.8 Hz, 1H), 3.41 (s, 3H), 3.35 (d, *J* = 8.8 Hz, 1H), 3.33-3.27 (m, 1H), 3.10-3.08 (m, 1H), 3.02-2.96 (m, 1H), 2.33 (s, 3H), 2.24-2.17 (m, 2H), 2.12-2.06 (m, 1H), 2.00 (d, *J* = 13.2 Hz, 1H), 1.94 (d, *J* = 13.2 Hz, 1H), 1.84-1.77 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.2, 143.1, 135.7, 134.8, 130.5, 128.8, 128.5, 127.5, 124.3, 123.8, 123.6, 90.6, 75.3, 67.8, 65.2, 56.6, 52.0, 51.9, 51.2, 50.8, 42.6, 42.2, 28.8, 21.4; HRMS (ESI) calcd for $C_{26}H_{29}NNaO_6S$ [M + Na^+], 506.1608; found: 506.1585. **Isomer 2**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.2 Hz, 1H), 7.45 (d, *J* = 8.2 Hz, 2H), 7.24-7.20 (m, 1H), 7.12 (d, *J* = 8.2 Hz, 2H), 7.02-7.01 (m, 2H), 4.50 (d, *J* = 10.6 Hz, 1H), 3.90-3.85 (m, 1H), 3.78 (d, *J* = 10.6 Hz, 1H), 3.77-3.73 (m, 1H), 3.54 (s, 2H), 3.39 (s, 3H), 3.38-3.32 (m, 1H), 3.19 (d, *J* = 7.2 Hz, 1H), 3.10-3.05 (m, 1H), 2.33 (s, 3H), 2.32-2.26 (m, 1H), 2.16 (d, *J* = 13.2 Hz, 1H), 2.13-2.08 (m, 1H), 2.01-1.94 (m, 1H), 1.91 (d, *J* = 13.2 Hz, 1H), 1.68-1.66 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 143.1, 135.7, 135.0, 130.5, 128.8, 128.5, 127.6, 124.2, 123.8, 123.5, 90.6, 79.5, 67.1, 65.2, 56.5, 52.6, 52.1, 50.9, 48.2, 45.0, 37.6, 29.7, 21.4; HRMS (ESI) calcd for $C_{26}H_{29}NNaO_6S$ [M + Na^+], 506.1608; found: 506.1585.

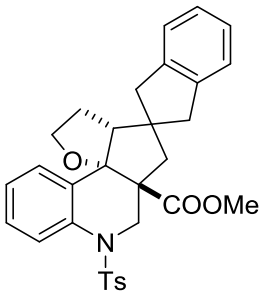


Methyl 5'-oxo-7-tosyl-3,3a,6,7-tetrahydro-2H-spiro[furo[2',3':2,3]cyclopenta[1,2-c]quinoline-4,2'-pyrrolidine]-5a(5H)-carboxylate (3am). (51 mg, 52%, dr = 1:1). Isolated by flash column chromatography (methanol/dichloromethane = 1:20, R_f = 0.5); IR (neat): ν_{max} 3412, 3069, 3034, 2951, 2884, 1732, 1680, 1601, 1483, 1456, 1394, 1350, 1229, 1165, 1087, 1030, 914 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.82 (m, 1H), 7.39-7.35 (m, 2H), 7.24-7.18 (m, 1H), 7.07 (d, *J* = 8.2 Hz, 2H), 6.70-6.85 (m, 2H), 6.56 (br, 0.63H), 6.11 (br, 0.37H), 4.42-4.34 (m, 1H), 3.68-3.59 (m, 1H), 3.32-3.29 (m, 3H), 3.28-3.20 (m, 2H), 2.94-2.86 (m, 1H), 2.31-1.90 (m, 11H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 175.8, 174.1, 172.8, 143.2, 143.1, 136.2, 135.9, 134.8, 134.7, 129.1, 128.9, 128.8, 128.7, 127.4, 124.7, 124.4, 123.9, 123.6, 123.5, 91.1, 90.1, 69.4, 69.0, 65.2, 64.8, 56.7, 55.6, 53.5, 53.3, 52.5, 52.1, 50.2, 49.7, 48.4, 48.0; HRMS (ESI) calcd for $C_{26}H_{28}N_2NaO_6S$ [M + Na^+], 519.1560; found: 519.1552.

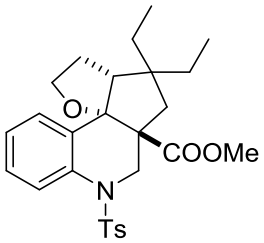


Methyl 7'-tosyl-3',3a',6',7'-tetrahydro-2'H,3H-spiro[benzo[b][1,4]dioxine-2,4'-furo[2',3':2,3]cyclopenta[1,2-c]quinoli

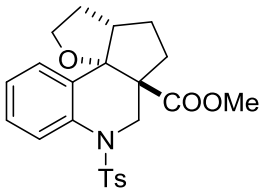
ne]-5a'(5'H)-carboxylate (3an). (93 mg, 62%, dr = 1:1). **Isomer 1:** Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:7, R_f = 0.6); IR (neat): ν_{max} 3067, 3038, 2953, 2878, 1732, 1595, 1495, 1460, 1352, 1265, 1232, 1167, 1024, 928 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 8.2 Hz, 1H), 7.48 (d, J = 8.2 Hz, 2H), 7.29-7.25 (m, 1H), 7.16 (d, J = 8.2 Hz, 2H), 7.05 (t, J = 7.6 Hz, 1H), 7.01 (t, J = 7.6 Hz, 1H), 6.92-6.85 (m, 4H), 4.52 (d, J = 10.6 Hz, 1H), 4.14 (d, J = 11.2 Hz, 1H), 4.00 (d, J = 11.2 Hz, 1H), 3.90 (d, J = 10.6 Hz, 1H), 3.53 (q, J = 8.4 Hz, 1H), 3.41 (s, 3H), 3.37 (d, J = 7.2 Hz, 1H), 3.05-3.00 (m, 1H), 2.38 (d, J = 13.6 Hz, 1H), 2.37 (s, 3H), 2.21 (d, J = 13.6 Hz, 1H), 2.21-2.06 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 143.1, 143.0, 142.7, 135.8, 134.8, 130.1, 128.8, 128.7, 127.6, 124.5, 123.7, 123.6, 121.9, 121.2, 117.1, 117.0, 90.0, 83.4, 70.3, 66.1, 56.1, 52.3, 50.4, 50.2, 43.0, 27.9, 21.5; HRMS (ESI) calcd for $C_{30}H_{29}NNaO_7S$ [M + Na^+], 570.1557; found: 570.1534. **Isomer 2:** Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:7, R_f = 0.5); IR (neat): ν_{max} 2947, 2872, 1732, 1597, 1493, 1456, 1435, 1350, 1265, 1230, 1167, 1038 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.2 Hz, 1H), 7.47 (d, J = 8.2 Hz, 2H), 7.28-7.24 (m, 1H), 7.15 (d, J = 8.2 Hz, 2H), 7.06 (t, J = 7.6 Hz, 1H), 7.02 (t, J = 7.6 Hz, 1H), 6.91-6.81 (m, 3H), 6.70-6.67 (m, 1H), 4.52 (d, J = 10.6 Hz, 1H), 4.03 (d, J = 11.2 Hz, 1H), 3.97 (d, J = 11.2 Hz, 1H), 3.87 (d, J = 10.6 Hz, 1H), 3.51 (s, 3H), 3.41 (q, J = 8.4 Hz, 1H), 3.34 (d, J = 7.2 Hz, 1H), 3.20-3.14 (m, 1H), 2.39 (d, J = 13.6 Hz, 1H), 2.37 (s, 3H), 2.35-2.27 (m, 2H), 2.15 (d, J = 13.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 143.2, 142.5, 141.3, 135.2, 134.8, 129.8, 128.9, 128.4, 127.5, 124.1, 123.7, 123.5, 121.7, 121.6, 118.0, 116.8, 89.8, 83.9, 68.7, 64.7, 55.5, 52.0, 51.9, 51.3, 42.7, 27.6, 21.5; HRMS (ESI) calcd for $C_{30}H_{29}NNaO_7S$ [M + Na^+], 570.1557; found: 570.1534.



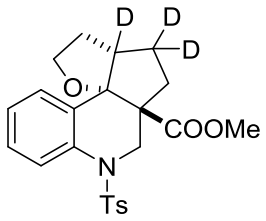
Methyl 7-tosyl-1',3,3a,3',6,7-hexahydro-2H-spiro[furo[2',3':2,3]cyclopenta[1,2-c]quinoline-4,2'-indene]-5a(5H)-carboxylate (3ao). (38 mg, 36%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2878, 2841, 1734, 1483, 1458, 1352, 1230, 1165, 1090, 1055, 1024, 930 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.2 Hz, 1H), 7.48 (d, J = 8.2 Hz, 2H), 7.26-7.14 (m, 7H), 7.04 (d, J = 4.2 Hz, 2H), 4.50 (d, J = 10.6 Hz, 1H), 3.81 (d, J = 11.2 Hz, 1H), 3.48 (s, 3H), 3.40 (q, J = 8.4 Hz, 1H), 3.24 (d, J = 7.2 Hz, 1H), 3.09-3.05 (m, 1H), 3.05 (d, J = 15.4 Hz, 1H), 2.97 (d, J = 15.4 Hz, 1H), 2.85 (d, J = 15.4 Hz, 1H), 2.77 (d, J = 15.4 Hz, 1H), 2.37 (s, 3H), 2.28-2.21 (m, 1H), 2.20 (d, J = 13.6 Hz, 1H), 2.12 (d, J = 13.6 Hz, 1H), 2.08-2.01 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 143.0, 142.5, 142.3, 135.6, 134.9, 131.1, 128.8, 128.3, 127.6, 126.4, 124.4, 124.1, 123.8, 123.7, 123.5, 90.8, 65.4, 56.5, 54.7, 52.0, 51.5, 51.2, 49.2, 47.2, 44.5, 30.8, 21.4; HRMS (ESI) calcd for $C_{31}H_{31}NNaO_5S$ [M + Na^+], 552.1815; found: 552.1767.



Methyl 4,4-diethyl-7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3ap). (53 mg, 55%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 3067, 3034, 2965, 2876, 1736, 1600, 1483, 1458, 1352, 1232, 1167, 1090, 1038, 934, 920 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.2 Hz, 1H), 7.45 (d, J = 8.2 Hz, 2H), 7.24-7.19 (m, 1H), 7.15-7.09 (m, 3H), 7.04 (td, J = 7.6, 0.8 Hz, 1H), 4.48 (d, J = 10.6 Hz, 1H), 3.79 (d, J = 10.6 Hz, 1H), 3.42 (s, 3H), 3.26-3.20 (m, 1H), 3.02 (d, J = 7.6 Hz, 1H), 2.93-2.87 (m, 1H), 2.34 (s, 3H), 2.25-2.11 (m, 2H), 1.93 (d, J = 13.2 Hz, 1H), 1.86 (d, J = 13.2 Hz, 1H), 1.65-1.58 (m, 1H), 1.46-1.33 (m, 2H), 1.01-0.92 (m, 1H), 0.82 (t, J = 7.2 Hz, 3H), 0.76 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 143.0, 135.3, 134.8, 131.5, 128.7, 128.0, 127.5, 124.1, 124.0, 123.3, 90.4, 64.6, 55.5, 52.0, 51.8, 50.3, 47.5, 46.9, 30.4, 28.6, 28.1, 21.4, 8.8, 8.7; HRMS (ESI) calcd for $C_{27}H_{33}NNaO_5S$ [M + Na^+], 506.1972; found: 506.1951.

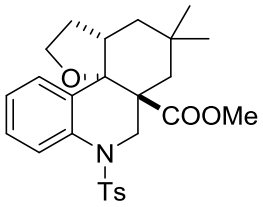


Methyl 7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate (3aq). (30 mg, 35%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.3); IR (neat): ν_{max} 3063, 3028, 2951, 2872, 1732, 1485, 1454, 1350, 1325, 1227, 1165, 1045, 1024 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.2 Hz, 1H), 7.49 (d, J = 8.2 Hz, 2H), 7.21-7.17 (m, 1H), 7.13 (d, J = 8.2 Hz, 2H), 7.02-6.97 (m, 2H), 4.53 (d, J = 10.6 Hz, 1H), 3.80 (d, J = 10.6 Hz, 1H), 3.47-3.40 (m, 1H), 3.37 (s, 3H), 3.38-3.33 (m, 1H), 3.26 (td, J = 8.6, 3.8 Hz, 1H), 2.33 (s, 3H), 2.41-2.23 (m, 2H), 1.91-1.80 (m, 3H), 1.51-1.44 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 143.0, 135.9, 135.2, 130.7, 128.8, 128.3, 127.6, 123.8, 123.6, 123.3, 90.4, 66.2, 59.5, 51.8, 50.6, 42.1, 35.0, 32.7, 32.5, 21.4; HRMS (ESI) calcd for $C_{23}H_{25}NNaO_5S$ [M + Na^+], 450.1346; found: 450.1332.

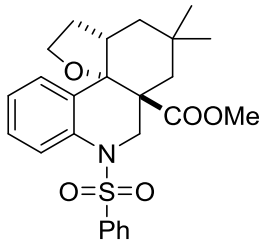


Methyl 7-tosyl-3,3a,4,5,6,7-hexahydrofuro[2',3':2,3]cyclopenta[1,2-c]quinoline-5a(2H)-carboxylate-3a,4,4-d3

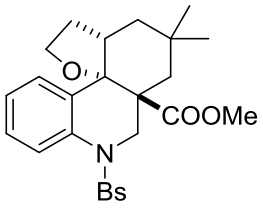
(3aq-D3). (20 mg, 23%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.3); IR (neat): ν_{max} 3067, 3036, 2951, 2872, 1732, 1483, 1456, 1348, 1232, 1165, 1090, 1041, 1030 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.2 Hz, 1H), 7.49 (d, J = 8.2 Hz, 2H), 7.21-7.17 (m, 1H), 7.13 (d, J = 8.2 Hz, 2H), 7.02-6.97 (m, 2H), 4.53 (d, J = 10.6 Hz, 1H), 3.80 (d, J = 10.6 Hz, 1H), 3.47-3.40 (m, 1H), 3.37 (s, 3H), 3.26 (td, J = 8.6, 3.8 Hz, 1H), 2.33 (s, 3H), 2.30-2.23 (m, 1H), 1.89-1.80 (m, 3H); HRMS (ESI) calcd for $C_{23}H_{22}D_3NNaO_5S$ [M + Na^+], 453.1534; found: 453.1524.



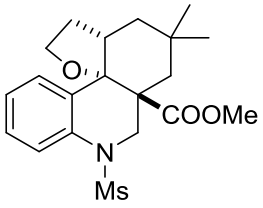
Methyl 5,5-dimethyl-8-tosyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5a). (64 mg, 68%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2955, 2866, 1734, 1481, 1456, 1352, 1227, 1207, 1165, 1090, 1057, 1036 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, J = 8.2, 0.8 Hz, 1H), 7.46 (d, J = 8.2 Hz, 2H), 7.22-7.16 (m, 2H), 7.12 (d, J = 8.2 Hz, 2H), 7.02 (td, J = 7.6, 1.0 Hz, 1H), 4.21 (d, J = 10.8 Hz, 1H), 3.68 (d, J = 10.8 Hz, 1H), 3.36 (s, 3H), 3.28-3.22 (m, 1H), 2.99-2.93 (m, 1H), 2.62-2.56 (m, 1H), 2.40-2.34 (m, 1H), 2.33 (s, 3H), 1.80 (dd, J = 13.6, 2.2 Hz, 1H), 1.57-1.50 (m, 1H), 1.51 (d, J = 13.6 Hz, 1H), 1.46-1.41 (m, 1H), 1.05-0.99 (m, 1H), 0.88 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 143.0, 135.4, 134.8, 133.1, 128.8, 127.7, 127.6, 125.1, 124.6, 123.2, 78.7, 62.0, 53.6, 51.6, 50.8, 43.5, 42.5, 34.3, 33.6, 33.4, 30.1, 23.6, 21.4; HRMS (ESI) calcd for $C_{26}H_{31}NNaO_5S$ [M + Na^+], 492.1815; found: 492.1795.



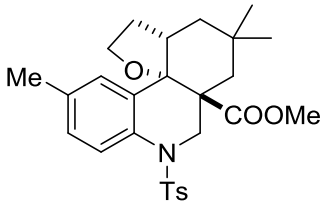
Methyl 5,5-dimethyl-8-(phenylsulfonyl)-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5b). (61 mg, 67%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2953, 2886, 1736, 1576, 1483, 1358, 1238, 1225, 1171, 1042, 1010 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.94 (dd, J = 8.6, 1.0 Hz, 1H), 7.60-7.56 (m, 2H), 7.50-7.46 (m, 1H), 7.37-7.33 (m, 2H), 7.25-7.20 (m, 2H), 7.08-7.04 (m, 1H), 4.24 (d, J = 10.6 Hz, 1H), 3.70 (d, J = 10.6 Hz, 1H), 3.38 (s, 3H), 3.25-3.19 (m, 1H), 3.01-2.95 (m, 1H), 2.57-2.51 (m, 1H), 2.40-2.31 (m, 1H), 1.82 (dd, J = 13.6, 2.2 Hz, 1H), 1.57-1.52 (m, 1H), 1.52 (d, J = 13.6 Hz, 1H), 1.47-1.42 (m, 1H), 1.06-0.99 (m, 1H), 0.90 (s, 3H), 0.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 137.6, 135.3, 133.3, 132.3, 128.2, 127.7, 127.6, 125.2, 124.8, 123.4, 78.6, 62.1, 53.6, 51.6, 50.9, 43.4, 42.5, 34.3, 33.6, 33.3, 30.1, 23.6; HRMS (ESI) calcd for $C_{25}H_{29}NNaO_5S$ [M + Na^+], 478.1659; found: 478.1645.



Methyl 8-((4-bromophenyl)sulfonyl)-5,5-dimethyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5c). (77 mg, 72%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2951, 2886, 1736, 1485, 1447, 1354, 1270, 1252, 1171, 1088, 1044 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.2 Hz, 1H), 7.49 (d, J = 8.8 Hz, 2H), 7.42 (d, J = 8.8 Hz, 2H), 7.25-7.21 (m, 2H), 7.08 (td, J = 7.6, 1.2 Hz, 1H), 4.21 (d, J = 10.6 Hz, 1H), 3.62 (d, J = 10.6 Hz, 1H), 3.37 (s, 3H), 3.31-3.25 (m, 1H), 3.01-2.95 (m, 1H), 2.59-2.53 (m, 1H), 2.42-2.34 (m, 1H), 1.81 (dd, J = 13.6, 2.2 Hz, 1H), 1.60-1.54 (m, 1H), 1.50 (d, J = 13.6 Hz, 1H), 1.47-1.43 (m, 1H), 1.06-1.00 (m, 1H), 0.90 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 136.4, 134.9, 133.6, 131.4, 129.1, 127.8, 127.3, 125.3, 125.0, 123.8, 78.7, 62.1, 53.7, 51.7, 51.0, 43.3, 42.5, 34.3, 33.6, 33.3, 30.0, 23.5; HRMS (ESI) calcd for $C_{25}H_{28}BrNNaO_5S$ [M + Na^+], 556.0764; found: 556.0757.

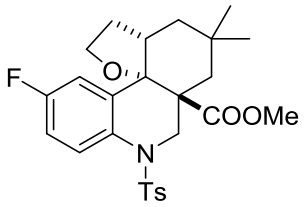


Methyl 5,5-dimethyl-8-(methylsulfonyl)-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5d). (44 mg, 56%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5); IR (neat): ν_{max} 2953, 2878, 1736, 1485, 1346, 1240, 1159, 1040, 962 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, J = 8.2, 1.0 Hz, 1H), 7.38 (dd, J = 7.8, 1.4 Hz, 1H), 7.23 (td, J = 7.8, 1.6 Hz, 1H), 7.13 (dd, J = 7.6, 1.2 Hz, 1H), 4.17 (d, J = 11.2 Hz, 1H), 3.95-3.89 (m, 1H), 3.74 (d, J = 11.2 Hz, 1H), 3.51-3.46 (m, 1H), 3.39 (s, 3H), 3.19-3.13 (m, 1H), 2.77 (s, 3H), 2.69-2.60 (m, 1H), 1.89 (dd, J = 13.6, 2.2 Hz, 1H), 1.83-1.77 (m, 1H), 1.66 (d, J = 13.6 Hz, 1H), 1.63-1.56 (m, 1H), 1.25-1.19 (m, 1H), 0.96 (s, 3H), 0.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 135.3, 133.6, 128.0, 125.3, 125.1, 123.8, 79.5, 62.7, 53.8, 51.7, 51.3, 43.3, 42.7, 35.4, 34.5, 33.7, 33.5, 30.1, 23.6; HRMS (ESI) calcd for $C_{20}H_{27}NNaO_5S$ [M + Na^+], 416.1502; found: 416.1493.

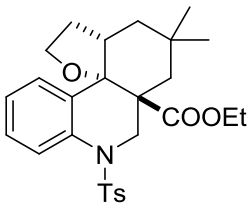


Methyl 5,5,11-trimethyl-8-tosyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5e). (65 mg, 67%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2949, 2878, 1728, 1491, 1346, 1254, 1161, 1090, 1051, 1038 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.76

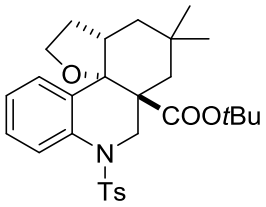
(d, J = 8.2 Hz, 1H), 7.43 (d, J = 8.2 Hz, 2H), 7.11 (d, J = 8.2 Hz, 2H), 6.98-6.96 (m, 2H), 4.17 (d, J = 10.6 Hz, 1H), 3.62 (d, J = 10.6 Hz, 1H), 3.35 (s, 3H), 3.23-3.16 (m, 1H), 2.96-2.90 (m, 1H), 2.59-2.53 (m, 1H), 2.39-2.33 (m, 1H), 2.32 (s, 3H), 2.28 (s, 3H), 1.76 (dd, J = 13.6, 2.2 Hz, 1H), 1.54-1.48 (m, 1H), 1.48 (d, J = 13.6 Hz, 1H), 1.43-1.38 (m, 1H), 1.02-0.96 (m, 1H), 0.86 (s, 3H), 0.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 142.8, 134.7, 132.9, 132.8, 132.5, 128.7, 128.3, 127.6, 125.7, 124.4, 78.6, 61.8, 53.5, 51.6, 50.8, 43.4, 42.4, 34.3, 33.5, 33.4, 30.0, 23.6, 21.4, 21.1; HRMS (ESI) calcd for $C_{27}H_{33}NNaO_5S$ [M + Na^+], 506.1972; found: 506.1957.



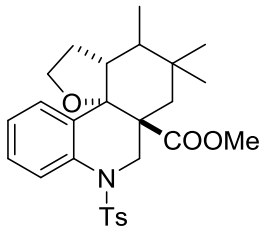
Methyl 11-fluoro-5,5-dimethyl-8-tosyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5f). (55 mg, 57%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2953, 2882, 1730, 1599, 1352, 1339, 1246, 1218, 1167, 1042, 941 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.85 (m, 1H), 7.41 (d, J = 8.6 Hz, 2H), 7.13 (d, J = 8.6 Hz, 2H), 6.92-6.88 (m, 2H), 4.16 (d, J = 11.0 Hz, 1H), 3.62 (d, J = 11.0 Hz, 1H), 3.38 (s, 3H), 3.24-3.18 (m, 1H), 2.90-2.84 (m, 1H), 2.56-2.58 (m, 1H), 2.34 (s, 3H), 2.32-2.26 (m, 1H), 1.79 (dd, J = 13.6, 2.2 Hz, 1H), 1.56-1.561(m, 1H), 1.47 (d, J = 13.6 Hz, 1H), 1.44-1.40 (m, 1H), 1.01-0.95 (m, 1H), 0.87 (s, 3H), 0.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 159.0 (d, $J_{C\text{-}F}$= 242.2 Hz), 143.1, 135.6 (d, $J_{C\text{-}F}$= 6.6 Hz), 134.3, 131.3, 128.8, 127.6, 126.4 (d, $J_{C\text{-}F}$= 8.0 Hz), 114.4 (d, $J_{C\text{-}F}$= 22.2 Hz), 112.1 (d, $J_{C\text{-}F}$= 23.4 Hz), 78.5, 62.1, 53.4, 51.7, 51.0, 43.4, 42.4, 34.2, 33.7, 33.1, 30.0, 23.5, 21.4; HRMS (ESI) calcd for $C_{26}H_{30}FNNaO_5S$ [M + Na^+], 510.1721; found: 510.1709.



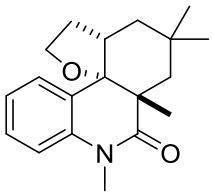
Ethyl 5,5-dimethyl-8-tosyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5g). (56 mg, 58%, dr = 13:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2953, 2893, 1734, 1489, 1430, 1352, 1229, 1213, 1167, 1090 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, J = 8.2, 1.0 Hz, 1H), 7.45 (d, J = 8.2 Hz, 2H), 7.20-7.11 (m, 2H), 7.12 (d, J = 8.2 Hz, 2H), 7.01 (td, J = 7.6, 1.2 Hz, 1H), 4.22 (d, J = 10.6 Hz, 1H), 3.85-3.71 (m, 2H), 3.66 (d, J = 10.6 Hz, 1H), 3.27-3.21 (m, 1H), 2.98-2.92 (m, 1H), 2.61-2.55 (m, 1H), 2.41-2.33 (m, 1H), 2.32 (s, 3H), 1.78 (dd, J = 13.6, 2.2 Hz, 1H), 1.56-1.50 (m, 1H), 1.49 (d, J = 13.6 Hz, 1H), 1.46-1.40 (m, 1H), 1.05-0.99 (m, 1H), 0.92 (t, J = 7.2 Hz, 3H), 0.88 (s, 3H), 0.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.6, 142.9, 135.5, 134.8, 133.2, 128.8, 127.6, 125.2, 124.6, 123.1, 78.7, 61.9, 60.7, 53.8, 50.8, 43.5, 42.5, 34.3, 33.7, 33.4, 30.1, 23.8, 21.4, 13.5; HRMS (ESI) calcd for $C_{27}H_{33}NNaO_5S$ [M + Na^+], 506.1972; found: 506.1958.



***Tert*-butyl 5,5-dimethyl-8-tosyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5h)**. (53 mg, 52%, dr = 10:1). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2955, 2891, 1717, 1485, 1352, 1250, 1167, 1088, 1042, 943 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.45 (d, *J* = 8.2 Hz, 2H), 7.20-7.16 (m, 2H), 7.11 (d, *J* = 8.2 Hz, 2H), 7.01 (td, *J* = 7.8, 1.0 Hz, 1H), 4.21 (d, *J* = 10.6 Hz, 1H), 3.61 (d, *J* = 11.0 Hz, 1H), 3.26-3.20 (m, 1H), 2.94-2.88 (m, 1H), 2.60-2.54 (m, 1H), 2.39-2.30 (m, 1H), 2.32 (s, 3H), 1.72 (dd, *J* = 13.6, 2.2 Hz, 1H), 1.56-1.50 (m, 1H), 1.44 (d, *J* = 13.6 Hz, 1H), 1.45-1.40 (m, 1H), 1.07 (s, 9H), 1.06-0.99 (m, 1H), 0.92 (s, 3H), 0.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.6, 142.9, 135.7, 135.0, 133.4, 128.8, 127.6, 125.4, 124.5, 123.0, 81.6, 78.9, 61.8, 54.2, 51.5, 43.6, 42.5, 34.5, 33.8, 33.5, 30.1, 27.2, 24.8, 21.4. HRMS (ESI) calcd for $C_{29}H_{37}NNaO_5S$ [M + Na^+], 534.2285; found: 534.2285.

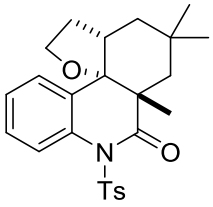


Methyl 4,5,5-trimethyl-8-tosyl-2,3,3a,4,5,6,7,8-octahydro-6aH-furo[3,2-l]phenanthridine-6a-carboxylate (5i). (25 mg, 26%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2945, 2868, 1736, 1458, 1352, 1165, 1020 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 8.4, 1.0 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 7.15-7.11 (m, 2H), 7.02-6.98 (m, 1H), 4.12 (d, *J* = 11.4 Hz, 1H), 3.73 (d, *J* = 10.2 Hz, 1H), 3.56 (s, 3H), 3.54-3.49 (m, 1H), 3.18 (q, *J* = 8.2 Hz, 1H), 3.05-2.99 (m, 1H), 2.35 (s, 3H), 2.13-2.06 (m, 2H), 1.97 (d, *J* = 14.2 Hz, 1H), 1.87-1.84 (m, 1H), 1.84 (d, *J* = 14.2 Hz, 1H), 0.92 (d, *J* = 7.6 Hz, 3H), 0.91 (s, 3H), 0.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 143.3, 136.1, 135.3, 130.9, 129.2, 127.5, 127.2, 124.7, 122.5, 120.8, 81.1, 62.9, 52.3, 51.9, 48.9, 43.0, 41.8, 36.2, 33.1, 31.1, 28.8, 26.3, 21.5, 13.3. HRMS (ESI) calcd for $C_{27}H_{33}NNaO_5S$ [M + Na^+], 506.1972; found: 506.1956.

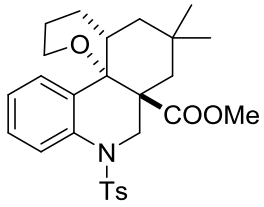


5,5,6a,8-tetramethyl-3,3a,4,5,6,6a-hexahydro-2H-furo[3,2-l]phenanthridin-7(8H)-one (5j). (28 mg, 48%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5); IR (neat): ν_{max} 2967, 2880, 1738, 1485, 1460, 1449, 1352, 1248, 1169, 1090, 1051, 1040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.28 (td, *J* = 7.8, 1.4 Hz, 1H), 7.21 (dd, *J* = 7.4, 1.0 Hz, 1H), 7.02-6.98 (m, 2H), 3.94-3.88 (m, 1H), 3.37 (s, 3H), 3.41-3.35 (m, 1H),

2.80-2.75 (m, 1H), 2.50-2.41 (m, 1H), 1.95 (d, *J* = 13.6 Hz, 1H), 1.74-1.69 (m, 1H), 1.67 (d, *J* = 13.6 Hz, 1H), 1.53-1.47 (m, 1H), 1.22-1.16 (m, 1H), 1.04 (s, 3H), 1.01 (s, 3H), 0.99 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.1, 140.4, 128.6, 128.3, 124.1, 121.6, 114.8, 83.2, 64.1, 43.1, 43.0, 40.4, 34.9, 33.2, 32.8, 30.1, 29.7, 27.6, 24.3; HRMS (ESI) calcd for $C_{19}H_{25}NNaO_2$ [M + Na^+], 322.1778; found: 322.1768.

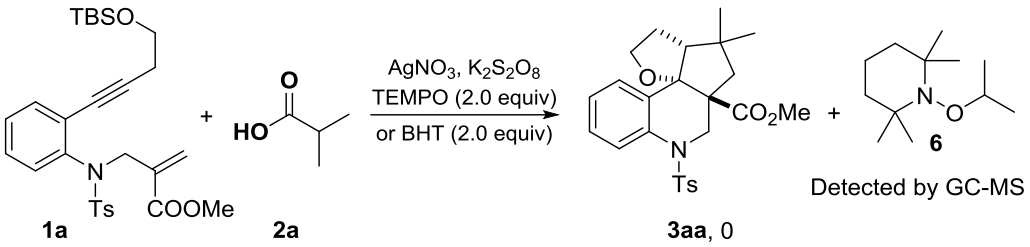


5,5,6a-trimethyl-8-tosyl-3,3a,4,5,6,6a-hexahydro-2H-furo[3,2-l]phenanthridin-7(8H)-one (5k). (30 mg, 34%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:5, R_f = 0.5); IR (neat): ν_{max} 2951, 2884, 1680, 1599, 1495, 1458, 1346, 1270, 1136, 1098, 1045 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.2 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.40-7.36 (m, 1H), 7.27-7.21 (m, 4H), 3.76 (q, *J* = 8.4 Hz, 1H), 3.56-3.50 (m, 1H), 2.76-2.70 (m, 1H), 2.47-2.38 (m, 1H), 2.42 (s, 3H), 1.71-1.58 (m, 2H), 1.43-1.36 (m, 2H), 1.07-1.04 (m, 1H), 0.94 (s, 3H), 0.89 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 144.2, 136.0, 135.7, 132.0, 129.5, 128.6, 128.1, 125.5, 125.0, 123.8, 82.8, 64.1, 45.9, 42.1, 40.7, 34.4, 32.7, 32.3, 30.3, 26.7, 23.4, 21.7; HRMS (ESI) calcd for $C_{25}H_{29}NNaO_4S$ [M + Na^+], 462.1710; found: 462.1696.



Methyl (4a,13b)-6,6-dimethyl-9-tosyl-3,4,4a,5,6,7,8,9-octahydropyrano[3,2-l]phenanthridine-7a(2H)-carboxylate (5l). (31 mg, 32%). Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:10, R_f = 0.5); IR (neat): ν_{max} 2955, 2922, 1717, 1597, 1485, 1454, 1360, 1296, 1229, 1146, 1086, 1042, 966 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.45 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.23 (td, *J* = 7.8, 1.6 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.08 (td, *J* = 7.8, 1.2 Hz, 1H), 4.06 (d, *J* = 10.6 Hz, 1H), 3.67 (d, *J* = 11.0 Hz, 1H), 3.35 (s, 3H), 2.79-2.76 (m, 1H), 2.52-2.43 (m, 2H), 2.33 (s, 3H), 2.25-2.20 (m, 1H), 1.90 (t, *J* = 13.2 Hz, 1H), 1.81 (dd, *J* = 13.6, 2.2 Hz, 1H), 1.75-1.69 (m, 1H), 1.57 (d, *J* = 13.6 Hz, 1H), 1.41-1.37 (m, 1H), 1.18-1.13 (m, 1H), 1.04-1.00 (m, 1H), 0.93 (s, 3H), 0.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 143.0, 135.6, 132.7, 129.5, 129.2, 127.8, 127.6, 124.5, 122.7, 72.3, 61.3, 53.5, 52.1, 51.5, 43.0, 40.7, 34.6, 30.0, 28.4, 26.0, 24.0, 21.4, 20.1. HRMS (ESI) calcd for $C_{27}H_{33}NNaO_5S$ [M + Na^+], 506.1972; found: 506.1972.

3. The radical trapping experiment



To a mixture of enyne **1a** (106 mg, 0.2 mmol), isobutyric acid **2a** (38 μL, 0.4 mmol), $AgNO_3$ (10.2 mg, 30 mol %), $K_2S_2O_8$ (108 mg, 0.4 mmol) and TEMPO (0.2 mmol) or BHT (0.2 mol), MeCN (2.0 mL) and H_2O (1.0 mL) were added under air at room temperature. The resulting mixture was stirred at 100 ℃ for 1 hour. After the mixture was cooled to room temperature, EtOAc (15.0 mL) and H_2O (5.0 mL) were added sequentially. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated in vacuo to give the crude products. ^{1}H NMR did not dectect the formation of **3aa**. Besides, the TEMPO-isopropyl adduct **6** was detected by GC-MS when TEMPO was added.

4. X-ray crystallography data for 3aa

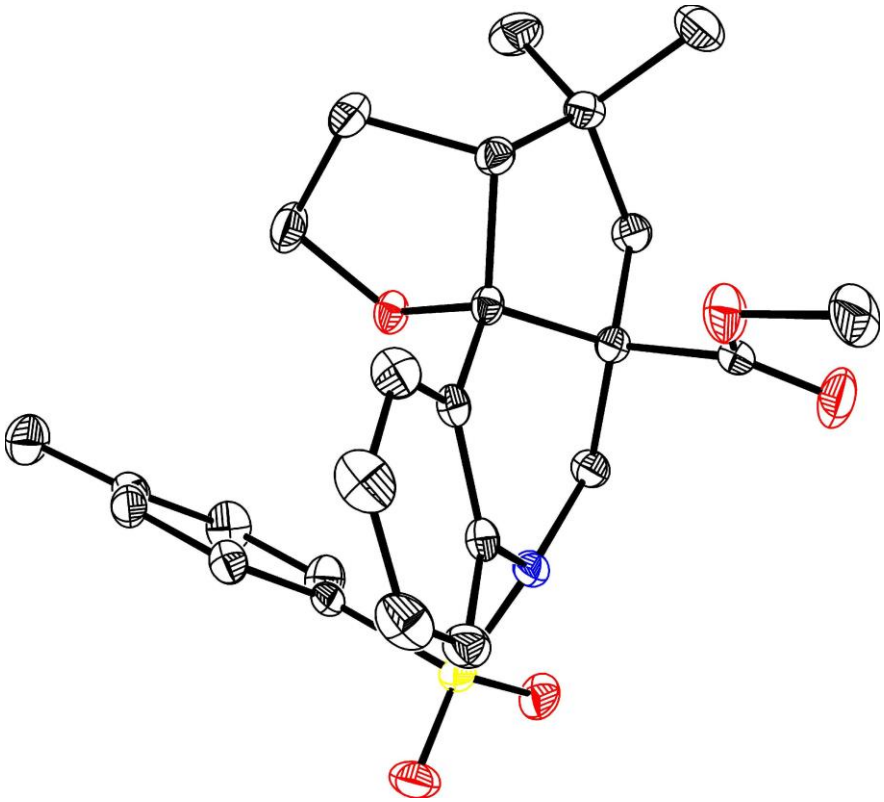


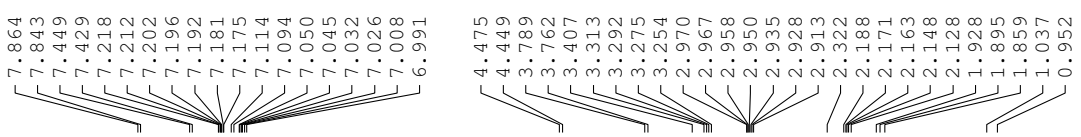
Figure S1 X-ray diffraction of major isomer of **3aa** (50% thermal ellipsoid plot). CCDC (1554749) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

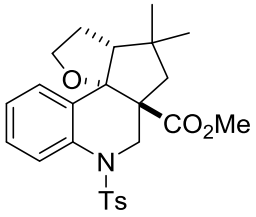
Table S1. Crystal data and structure refinement for **3aa**

Identification code	**3aa**

Empirical formula	C25 H29 N O5 S	
Formula weight	455.55	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 11.7381(4) Å	α= 90 °.
	b = 13.2058(5) Å	β= 98.487(2) °.
	c = 14.4879(6) Å	γ = 90 °.
Volume	2221.19(15) $Å^3$	
Z	4	
Density (calculated)	1.362 Mg/m^3	
Absorption coefficient	0.184 mm^{-1}	
F(000)	968	
Crystal size	0.40 x 0.20 x 0.20 mm^3	
Theta range for data collection	2.34 to 26.39 °.	
Index ranges	-14<=h<=14, -14<=k<=16, -18<=l<=18	
Reflections collected	18581	
Independent reflections	4521 [R(int) = 0.0323]	
Completeness to theta = 26.39 °	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9642 and 0.9302	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4521 / 0 / 293	
Goodness-of-fit on F^2	1.027	
Final R indices [I>2sigma(I)]	R1 = 0.0347, wR2 = 0.0876	
R indices (all data)	R1 = 0.0408, wR2 = 0.0914	
Largest diff. peak and hole	0.325 and -0.436 $e.Å^{-3}$	

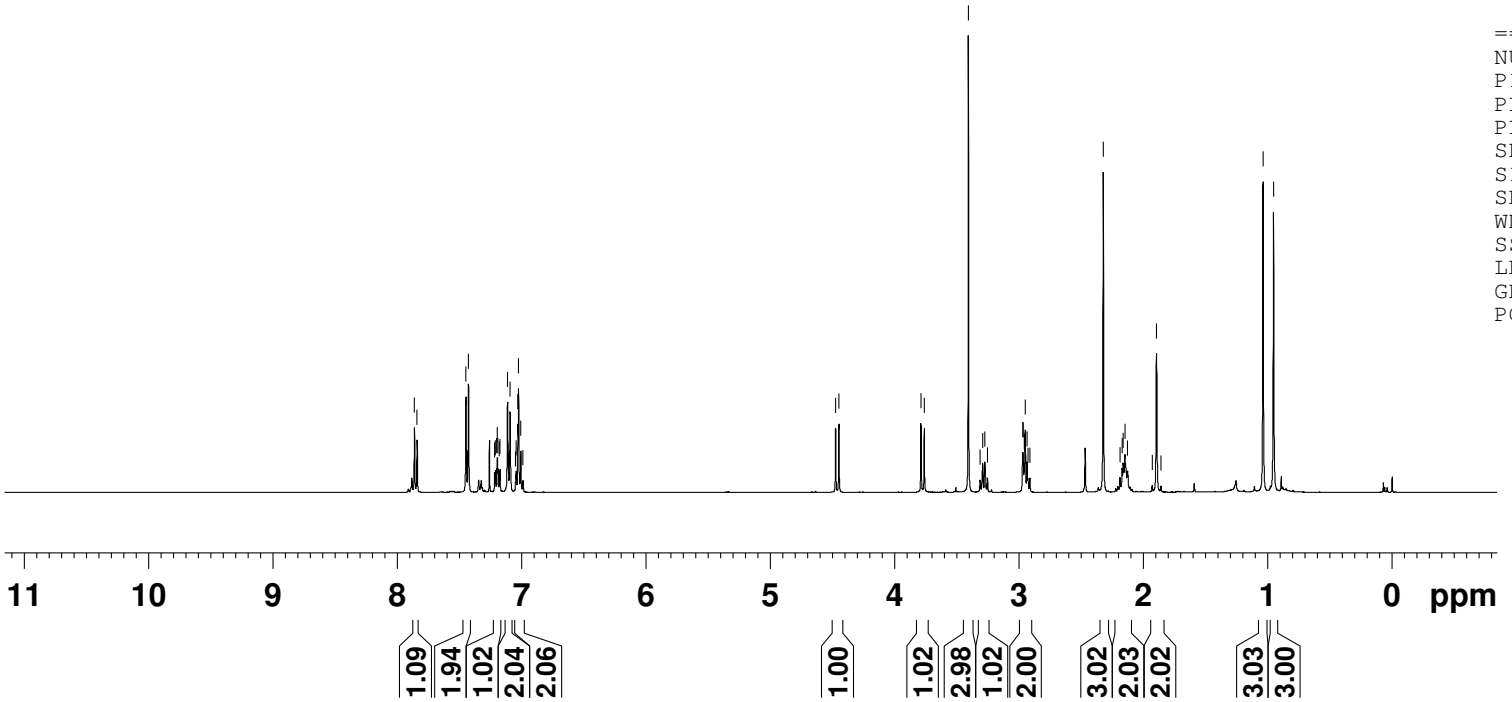
5. Copies of ^{1}H NMR and ^{13}C NMR spectra for new compounds

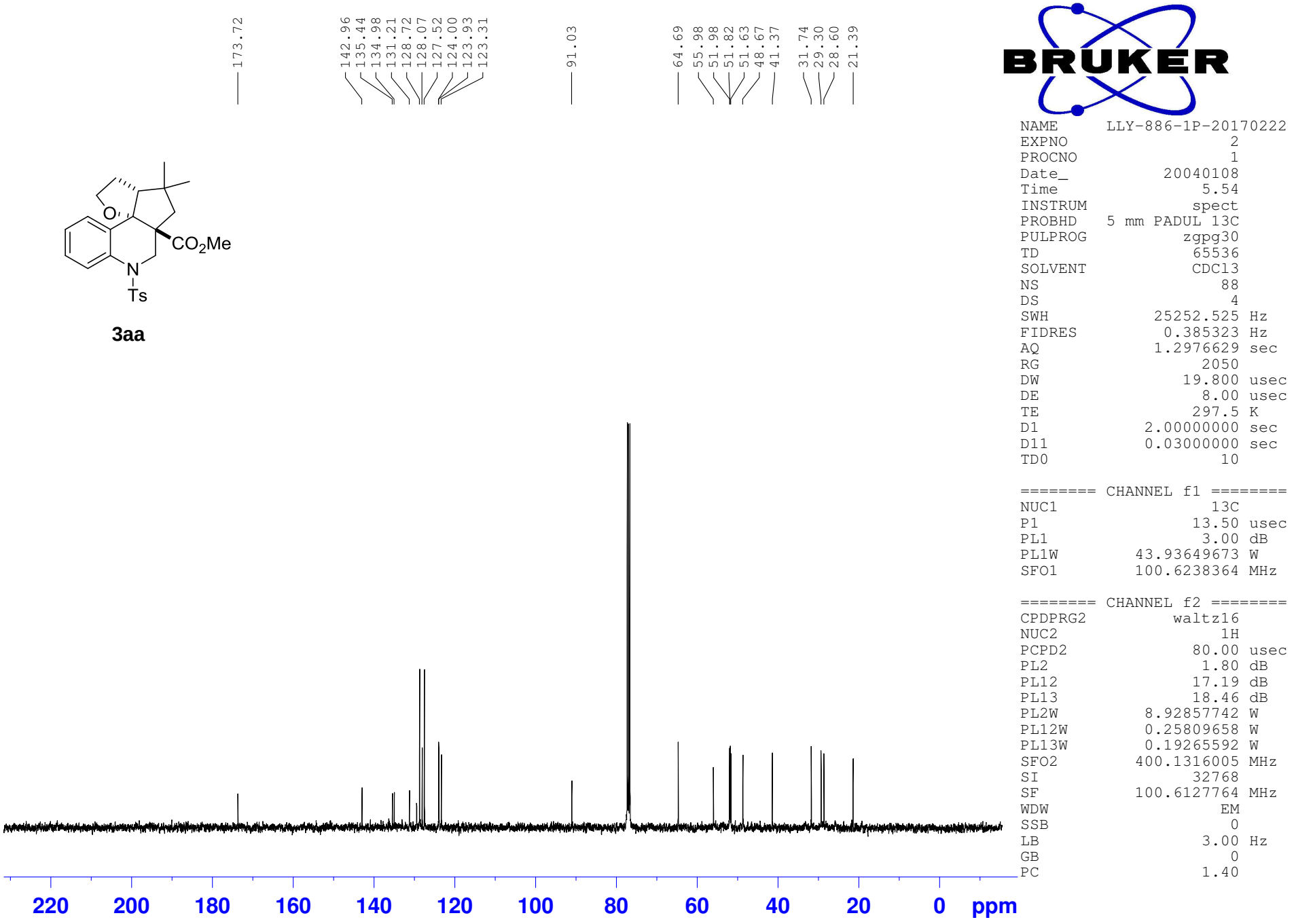




3aa

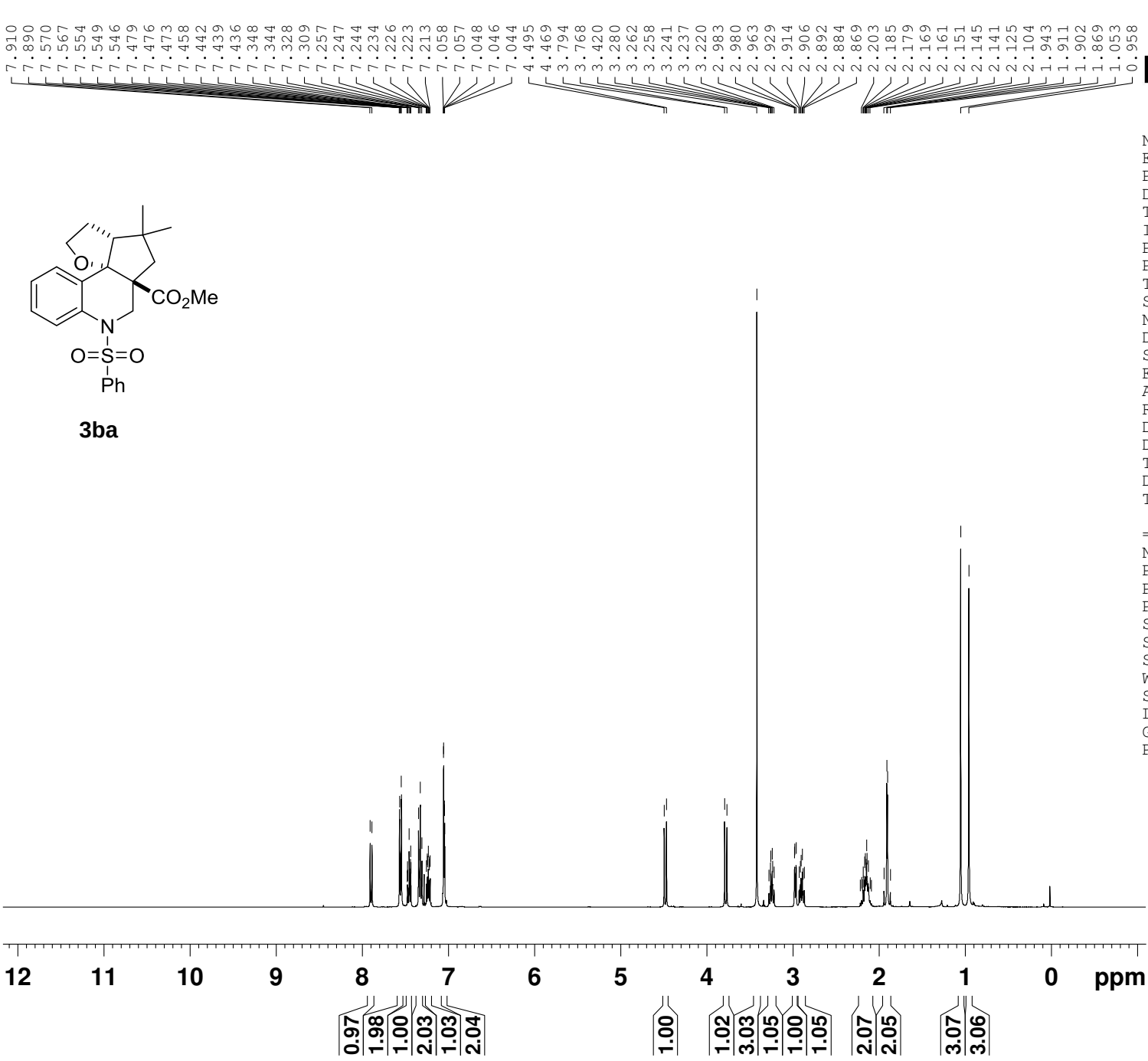
```
NAME      LLY-886-1P-20170222
EXPNO                       1
PROCNO                      1
Date_              20040108
Time                    5.37
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                          8
DS                          0
SWH                6393.862 Hz
FIDRES            0.195125 Hz
AQ                2.5625076 sec
RG                        144
DW                   78.200 usec
DE                    6.50 usec
TE                   296.4 K
D1               1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W            8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300096 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00
```



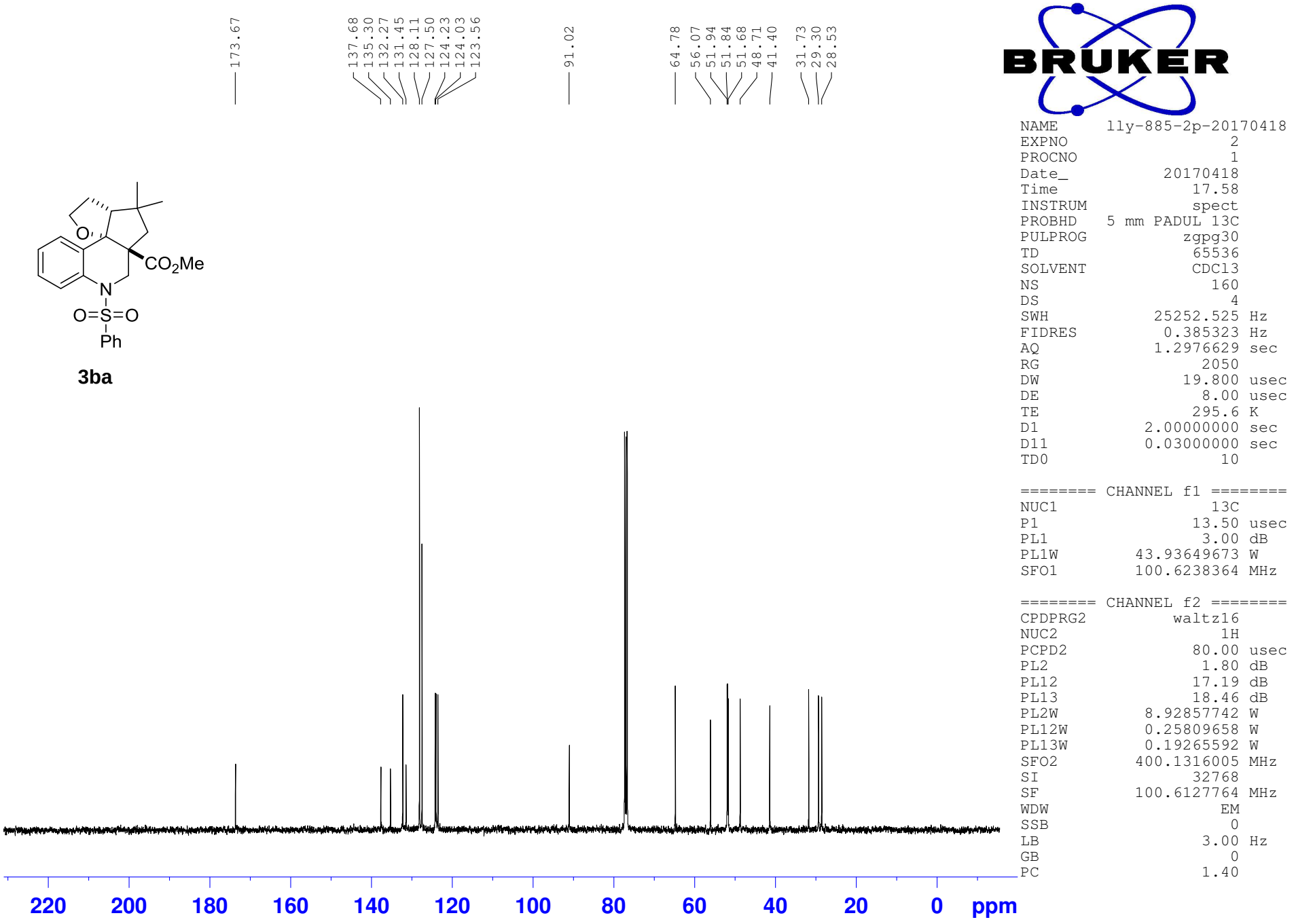


BRUKER

NAME	LLY-886-1P-20170222
EXPNO	2
PROCNO	1
Date_	20040108
Time	5.54
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	88
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	8.00 usec
TE	297.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	13.50 usec
PL1	3.00 dB
PL1W	43.93649673 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.80 dB
PL12	17.19 dB
PL13	18.46 dB
PL2W	8.92857742 W
PL12W	0.25809658 W
PL13W	0.19265592 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127764 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

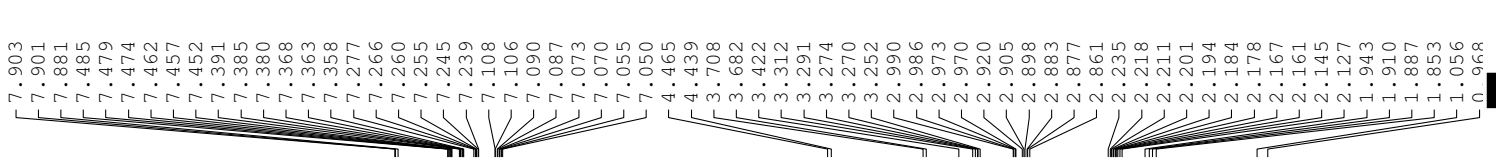


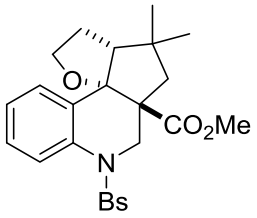

```
NAME      lly-885-2p-20170418
EXPNO                       1
PROCNO                      1
Date_              20170418
Time                   16.25
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       101
DW                    78.200 usec
DE                     6.50 usec
TE                    294.8 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



BRUKER

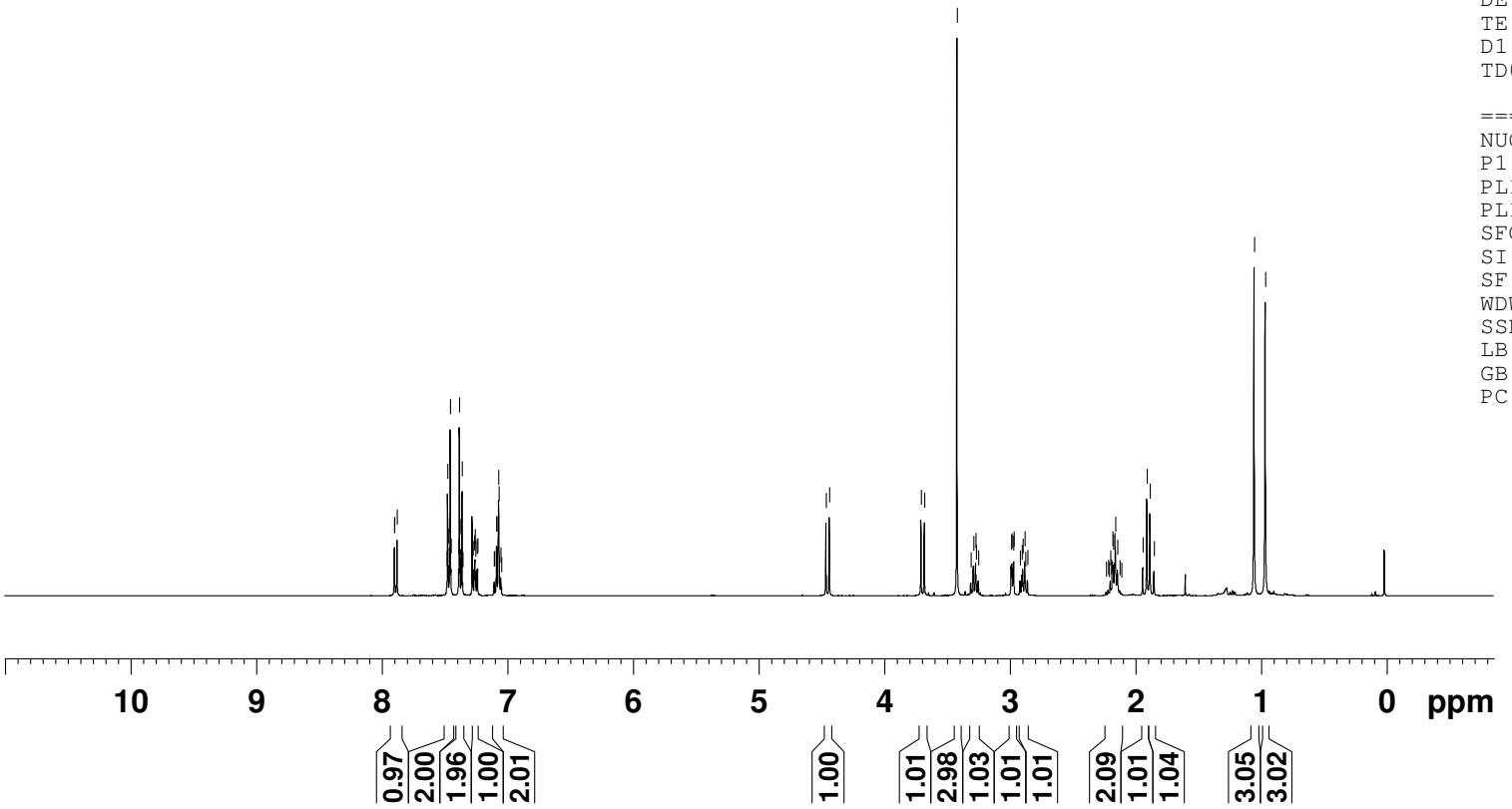
NAME	lly-885-2p-20170418
EXPNO	2
PROCNO	1
Date_	20170418
Time	17.58
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	160
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	8.00 usec
TE	295.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	13.50 usec
PL1	3.00 dB
PL1W	43.93649673 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.80 dB
PL12	17.19 dB
PL13	18.46 dB
PL2W	8.92857742 W
PL12W	0.25809658 W
PL13W	0.19265592 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127764 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

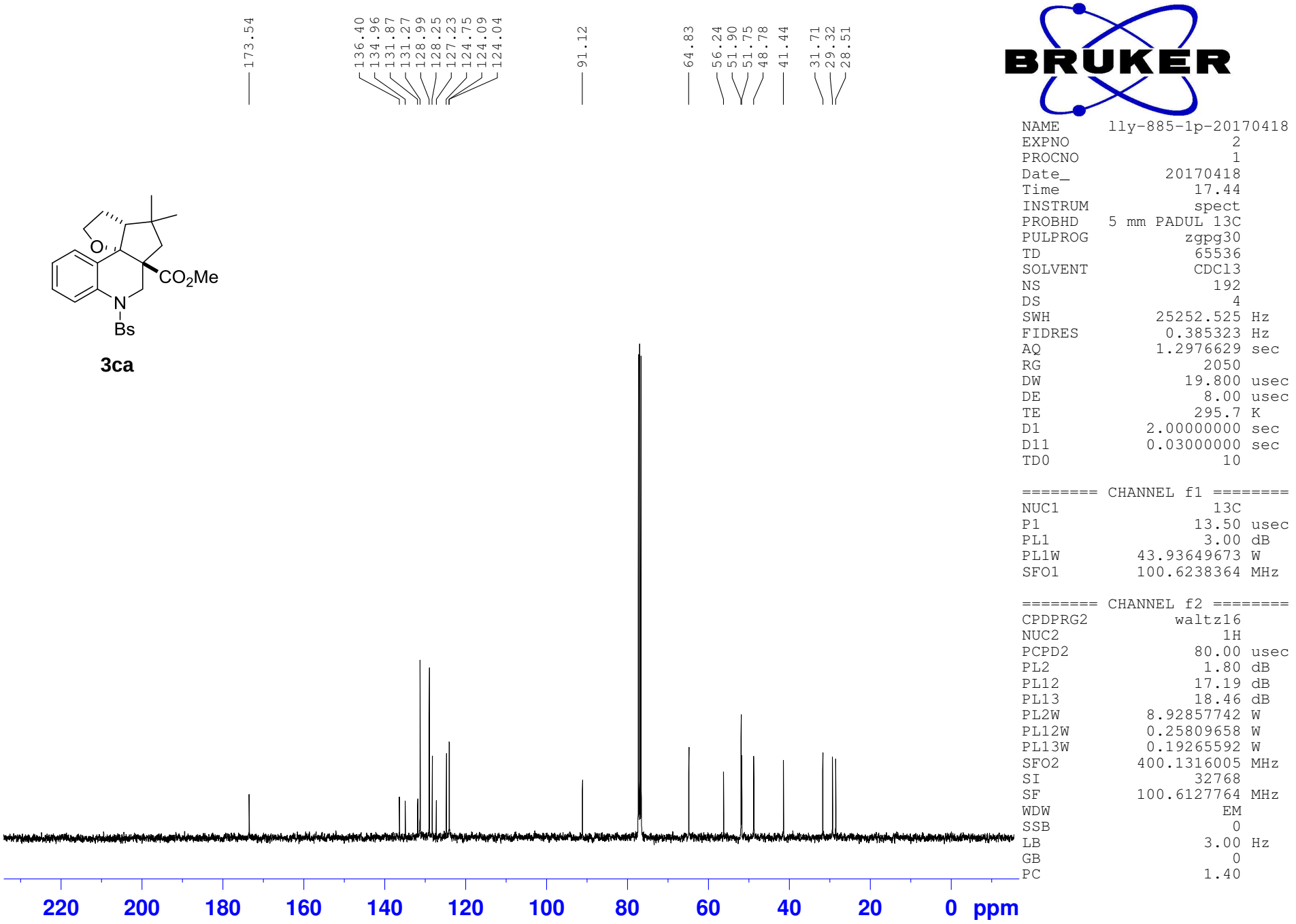




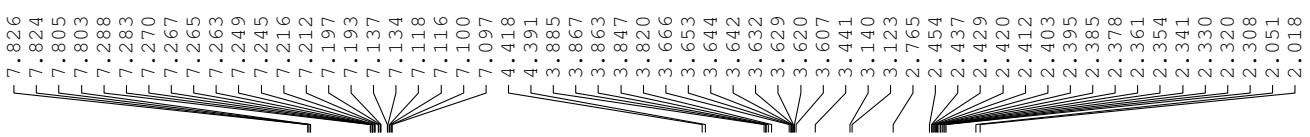
3ca

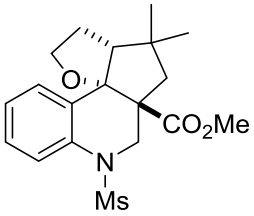
```
NAME      lly-885-1p-20170418
EXPNO                       1
PROCNO                      1
Date_                20170418
Time                    16.29
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                   2.5625076 sec
RG                        181
DW                     78.200 usec
DE                      6.50 usec
TE                     294.7 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W               8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300005 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```





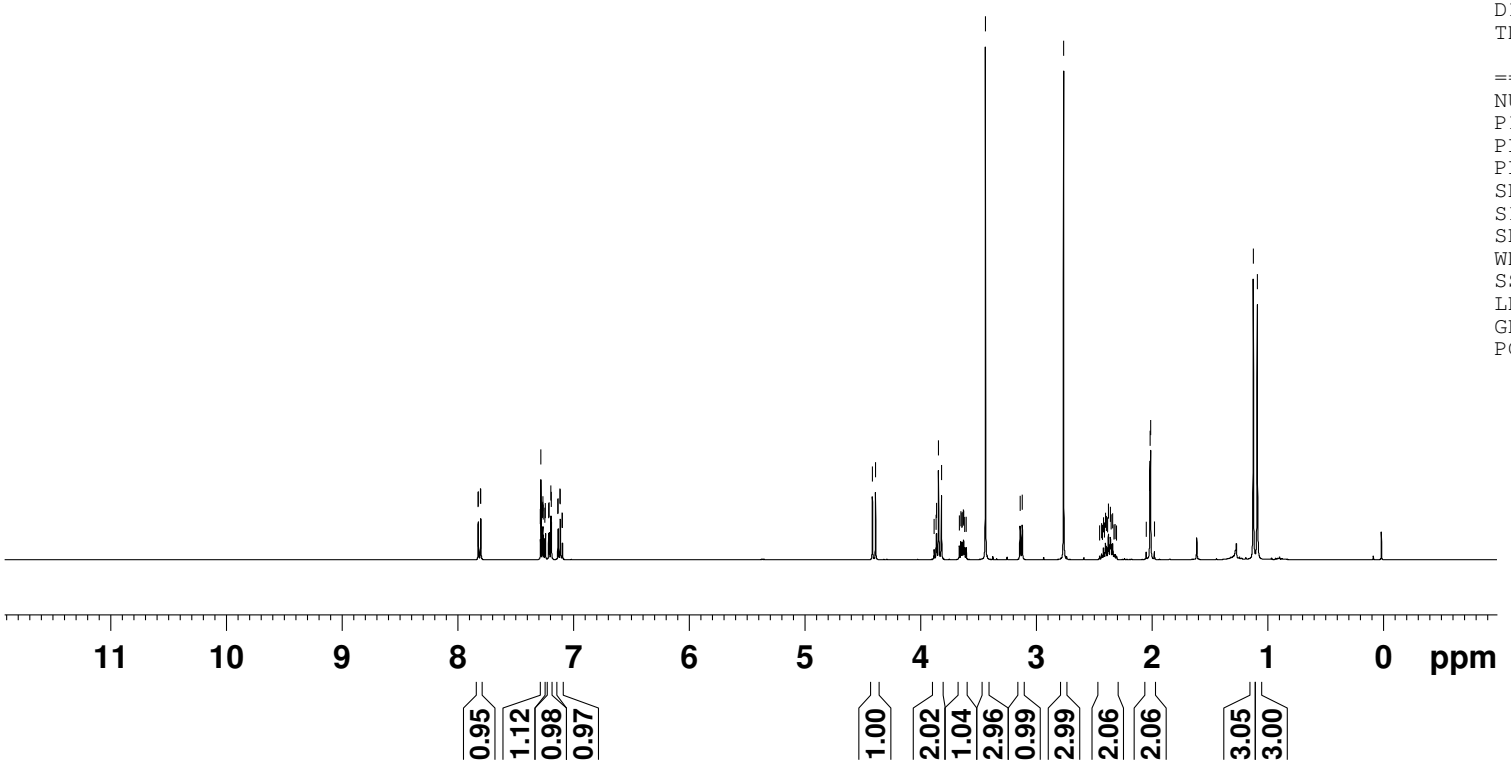

```
NAME          lly-885-1p-20170418
EXPNO                           2
PROCNO                          1
Date_                    20170418
Time                        17.44
INSTRUM                     spect
PROBHD       5 mm PADUL 13C
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           192
DS                             4
SWH                    25252.525 Hz
FIDRES                 0.385323 Hz
AQ                     1.2976629 sec
RG                          2050
DW                        19.800 usec
DE                         8.00 usec
TE                        295.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            10

======== CHANNEL f1 ========
NUC1                          13C
P1                         13.50 usec
PL1                         3.00 dB
PL1W                43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         1.80 dB
PL12                       17.19 dB
PL13                       18.46 dB
PL2W                 8.92857742 W
PL12W                0.25809658 W
PL13W                0.19265592 W
SFO2               400.1316005 MHz
SI                         32768
SF                 100.6127764 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.40
```

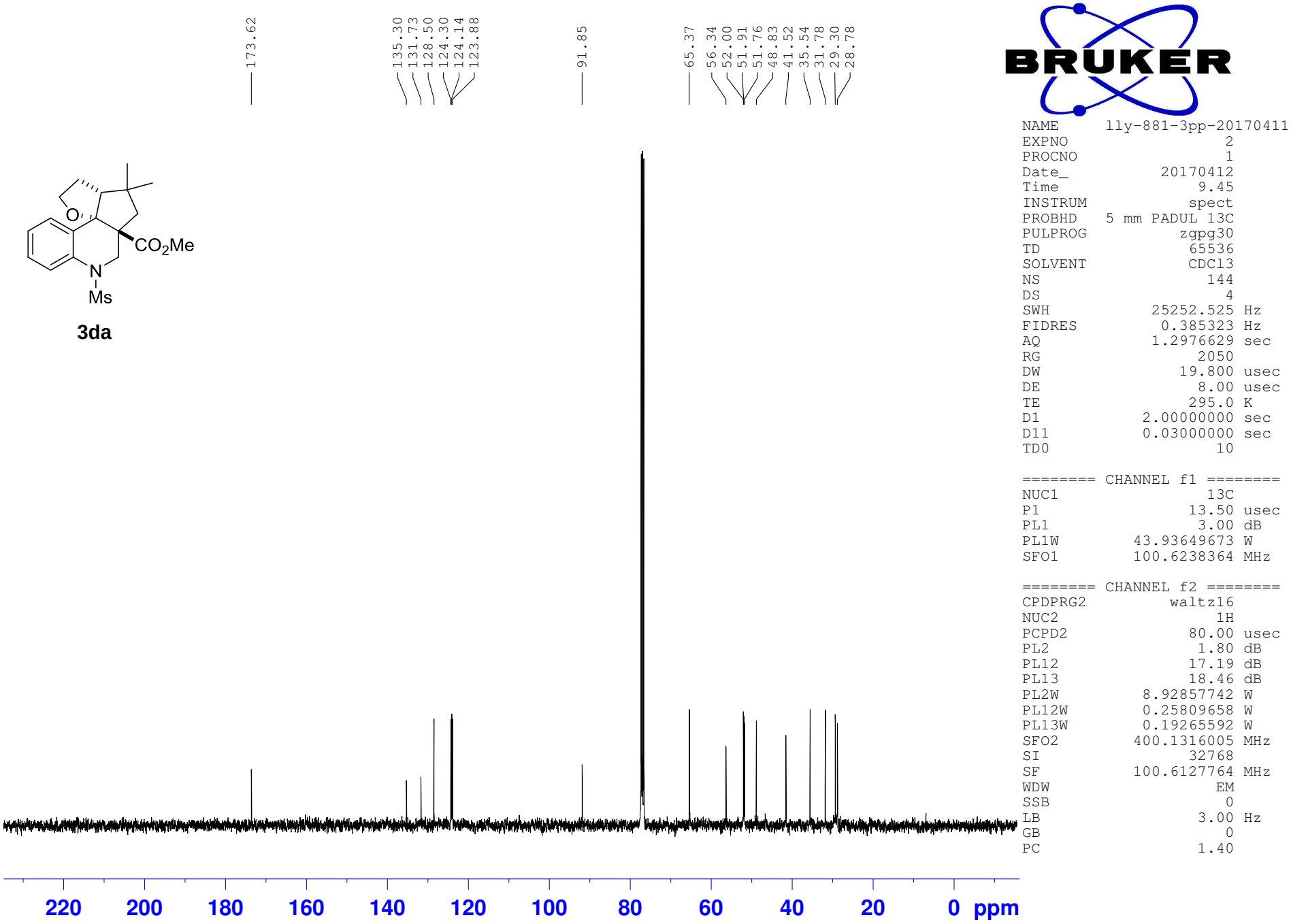




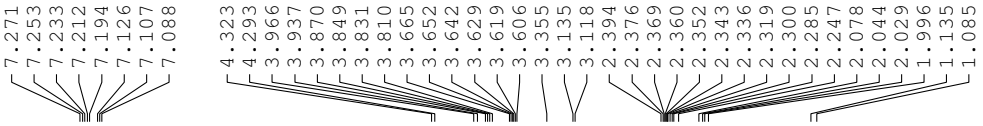
3da

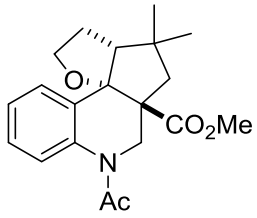
```
NAME      lly-881-3pp-20170412
EXPNO                        1
PROCNO                       1
Date_             20170412
Time                      9.42
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                         203
DW                    78.200 usec
DE                     6.50 usec
TE                     294.4 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300005 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      1.00
```





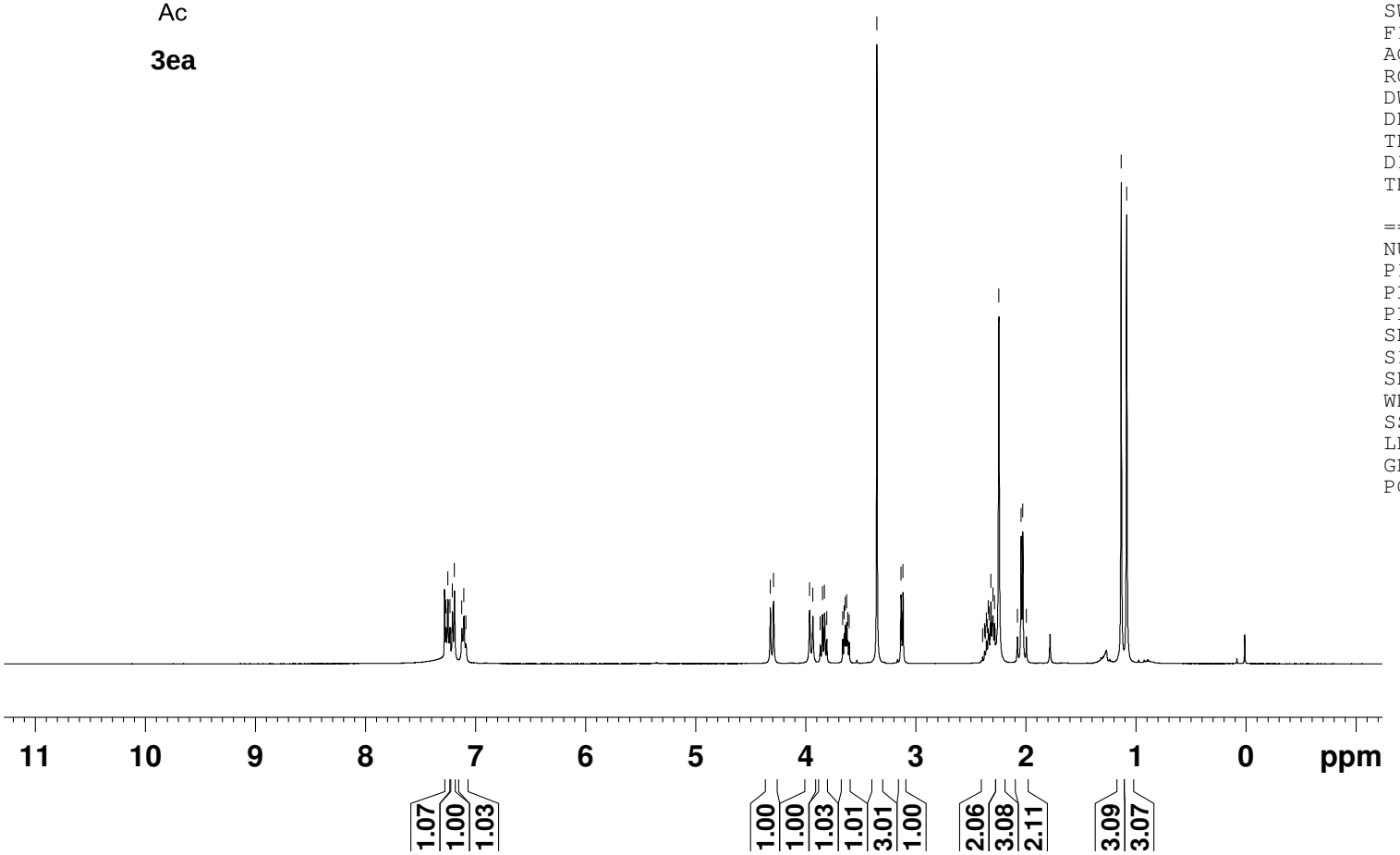

```
NAME      lly-881-3pp-20170411
EXPNO                        2
PROCNO                       1
Date_             20170412
Time                      9.45
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        144
DS                          4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                 1.2976629 sec
RG                       2050
DW                     19.800 usec
DE                      8.00 usec
TE                     295.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        10

======== CHANNEL f1 ========
NUC1                      13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                     32768
SF              100.6127764 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.40
```

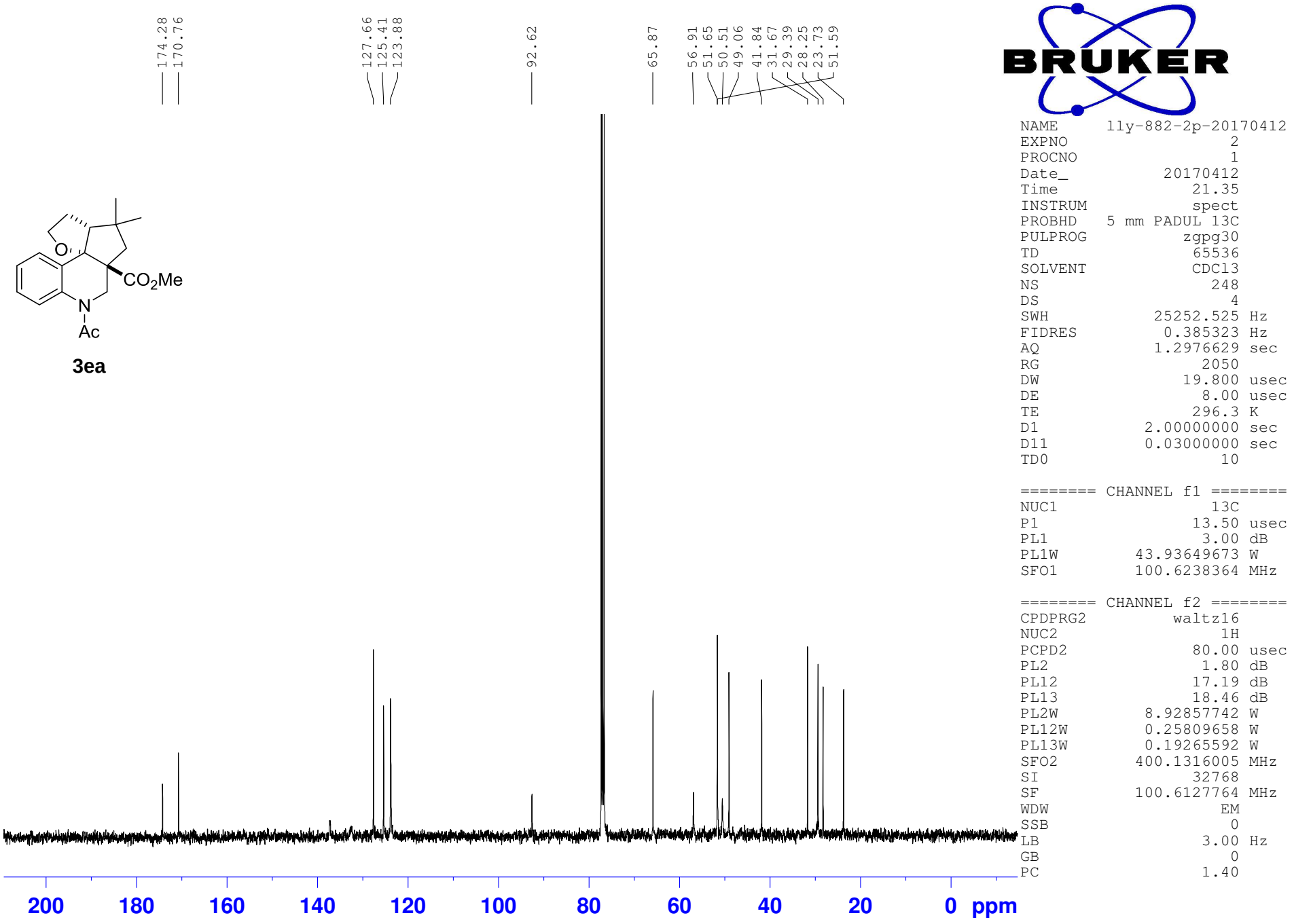




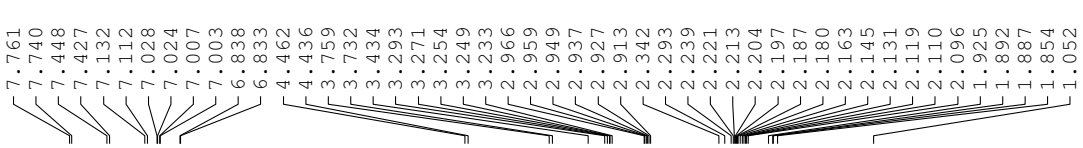
3ea

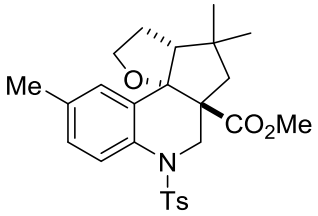
```
NAME      lly-882-2p-20170412
EXPNO                       1
PROCNO                      1
Date_                20170412
Time                    21.31
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                   2.5625076 sec
RG                        161
DW                     78.200 usec
DE                      6.50 usec
TE                     295.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300005 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```





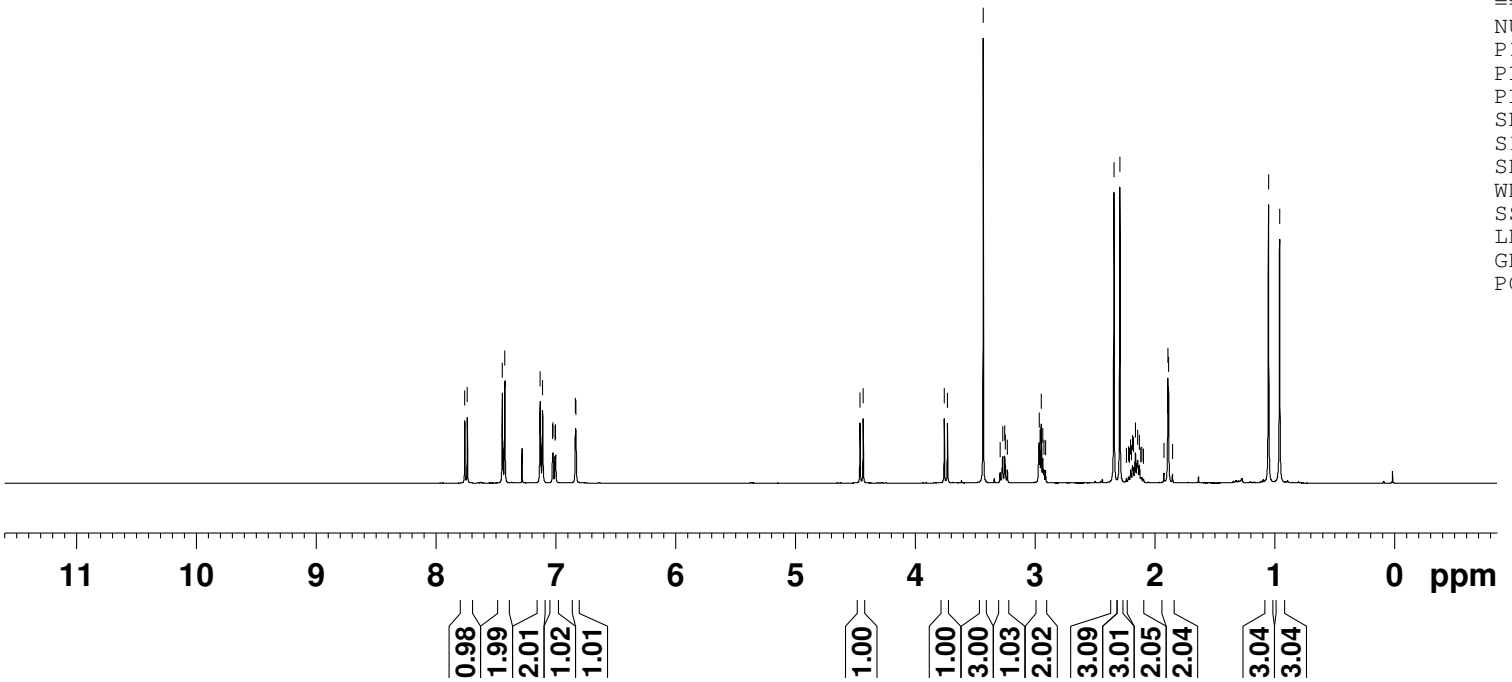

```
NAME        lly-882-2p-20170412
EXPNO                         2
PROCNO                        1
Date_                  20170412
Time                      21.35
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         248
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      296.3 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

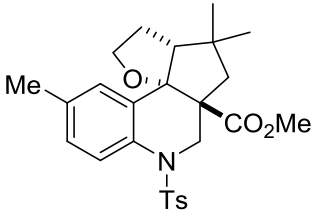




3ga

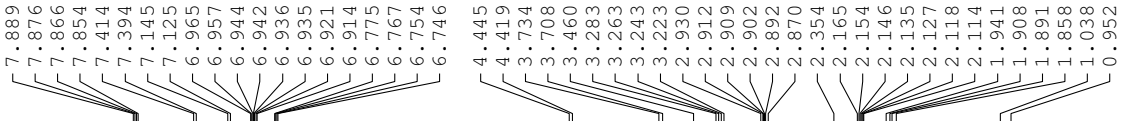
```
NAME      lly-887-4p-20170505
EXPNO                       1
PROCNO                      1
Date_              20170505
Time                   20.07
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       101
DW                    78.200 usec
DE                     6.50 usec
TE                    296.2 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

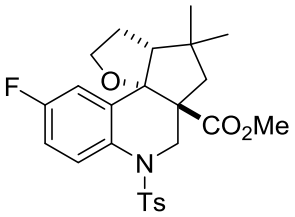




3ga

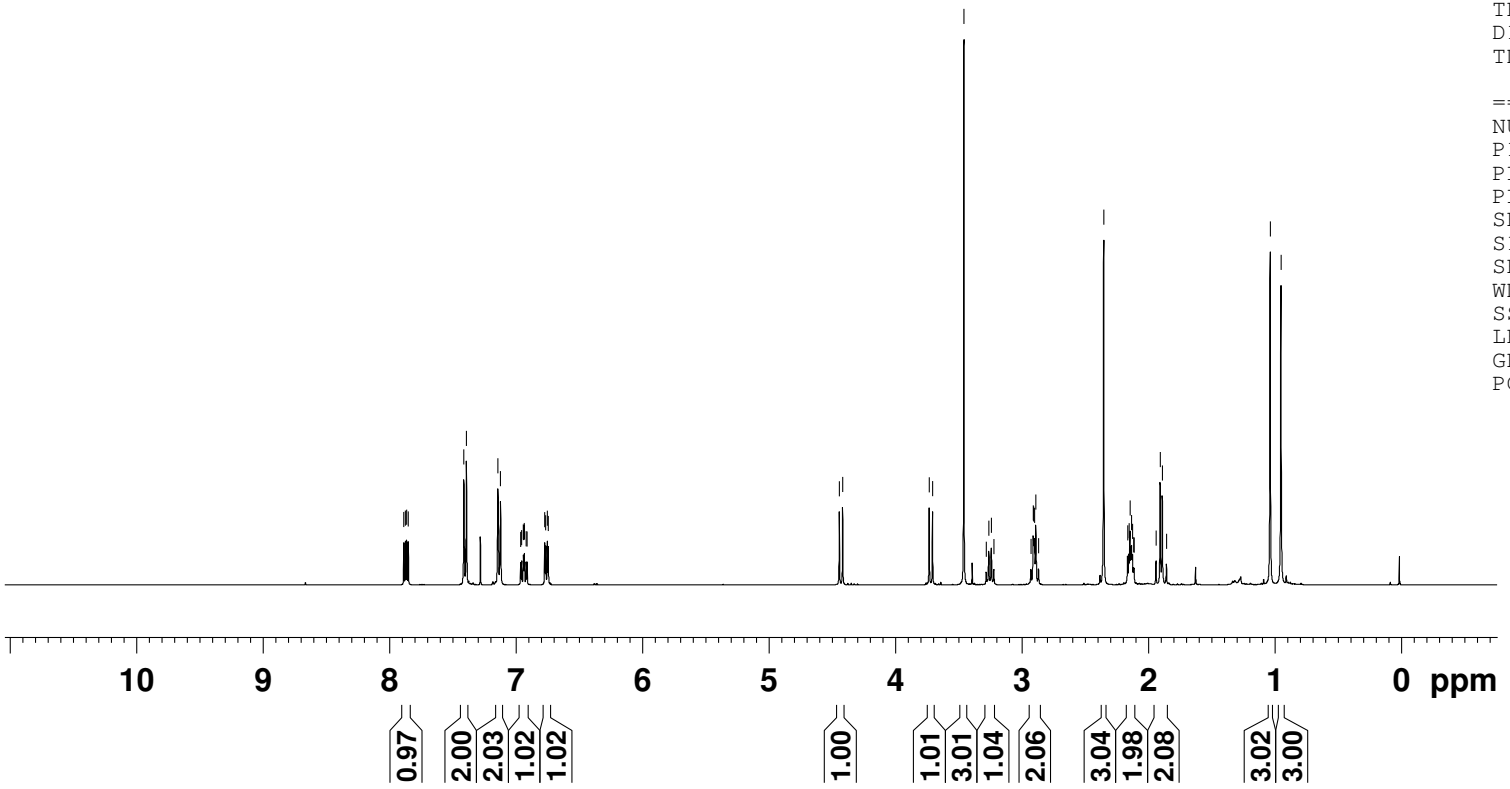

```
NAME        lly-887-4p-20170505
EXPNO                         2
PROCNO                        1
Date_                  20170505
Time                      20.13
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          80
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                         2050
DW                       19.800 usec
DE                        8.00 usec
TE                       297.3 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                        13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

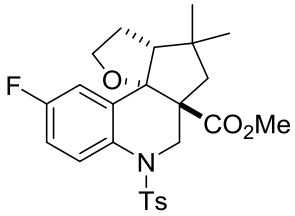




3ha

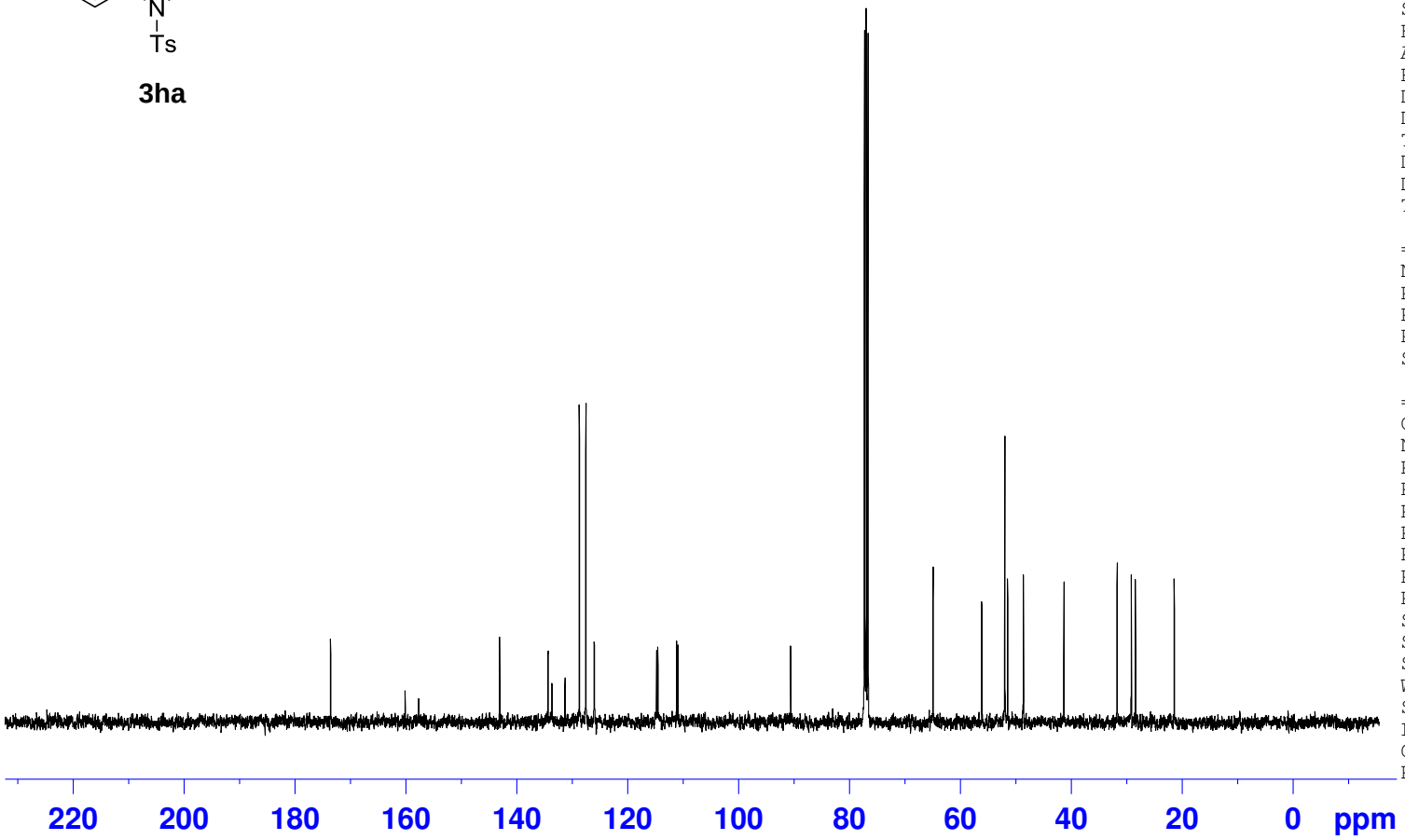
```
NAME      lly-885-4p-20170420
EXPNO                       1
PROCNO                      1
Date_              20170420
Time                   16.22
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       128
DW                    78.200 usec
DE                     6.50 usec
TE                    293.6 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300005 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00
```



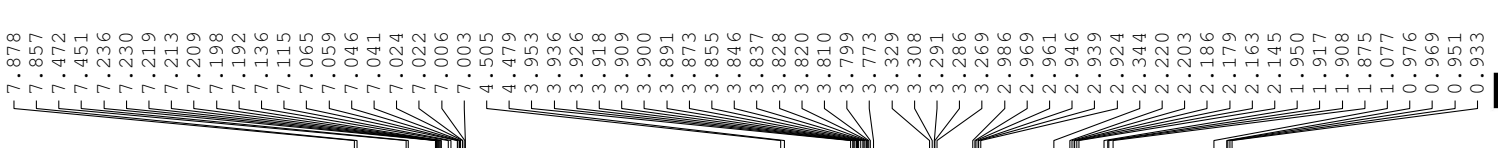


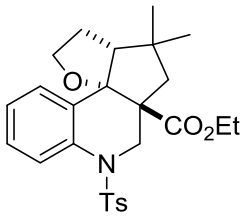
3ha

173.60
160.15
157.71
143.12
134.38
133.71
133.65
131.32
128.73
127.56
126.07
125.99
114.77
114.55
111.17
110.95
90.62
64.91
56.13
51.97
51.47
48.62
41.30
31.71
29.15
28.41
21.40



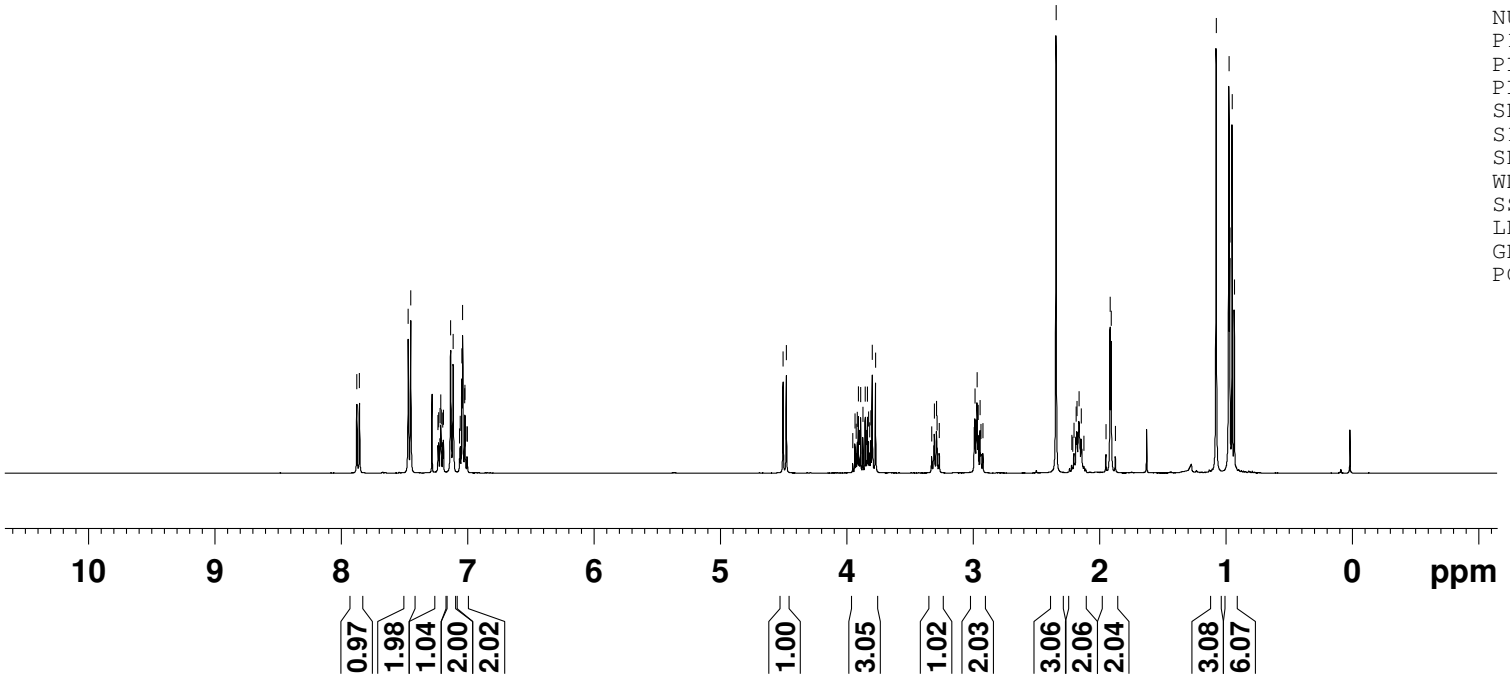

```
NAME          lly-885-4p-20170420
EXPNO                           2
PROCNO                          1
Date_                    20170420
Time                        16.27
INSTRUM                     spect
PROBHD          5 mm PADUL 13C
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            120
DS                              4
SWH                     25252.525 Hz
FIDRES                   0.385323 Hz
AQ                       1.2976629 sec
RG                           2050
DW                         19.800 usec
DE                          8.00 usec
TE                         294.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            10

======== CHANNEL f1 ========
NUC1                          13C
P1                         13.50 usec
PL1                         3.00 dB
PL1W                43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         1.80 dB
PL12                       17.19 dB
PL13                       18.46 dB
PL2W                 8.92857742 W
PL12W                0.25809658 W
PL13W                0.19265592 W
SFO2               400.1316005 MHz
SI                         32768
SF                 100.6127764 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.40
```

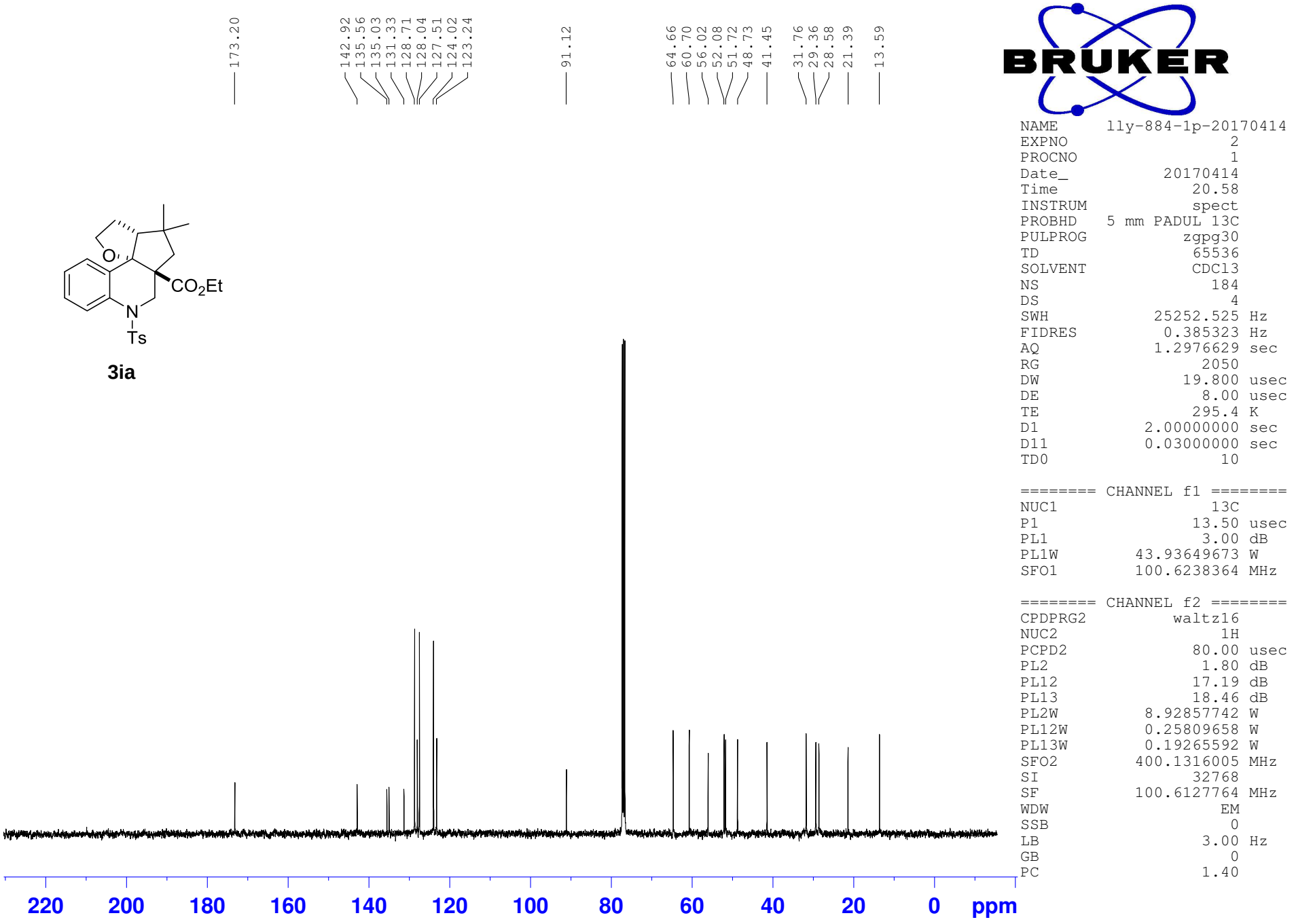




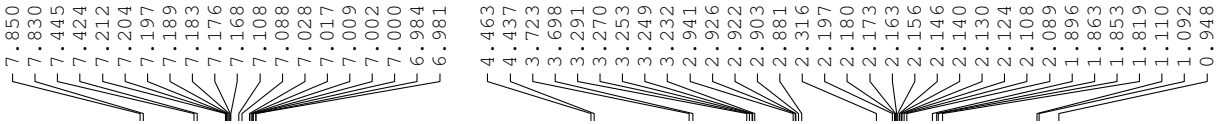
3ia

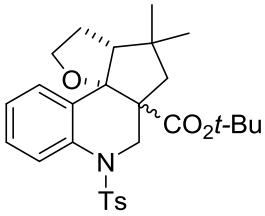
```
NAME      lly-884-1p-20170414
EXPNO                       1
PROCNO                      1
Date_              20170414
Time                   20.55
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       181
DW                    78.200 usec
DE                     6.50 usec
TE                    294.5 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```





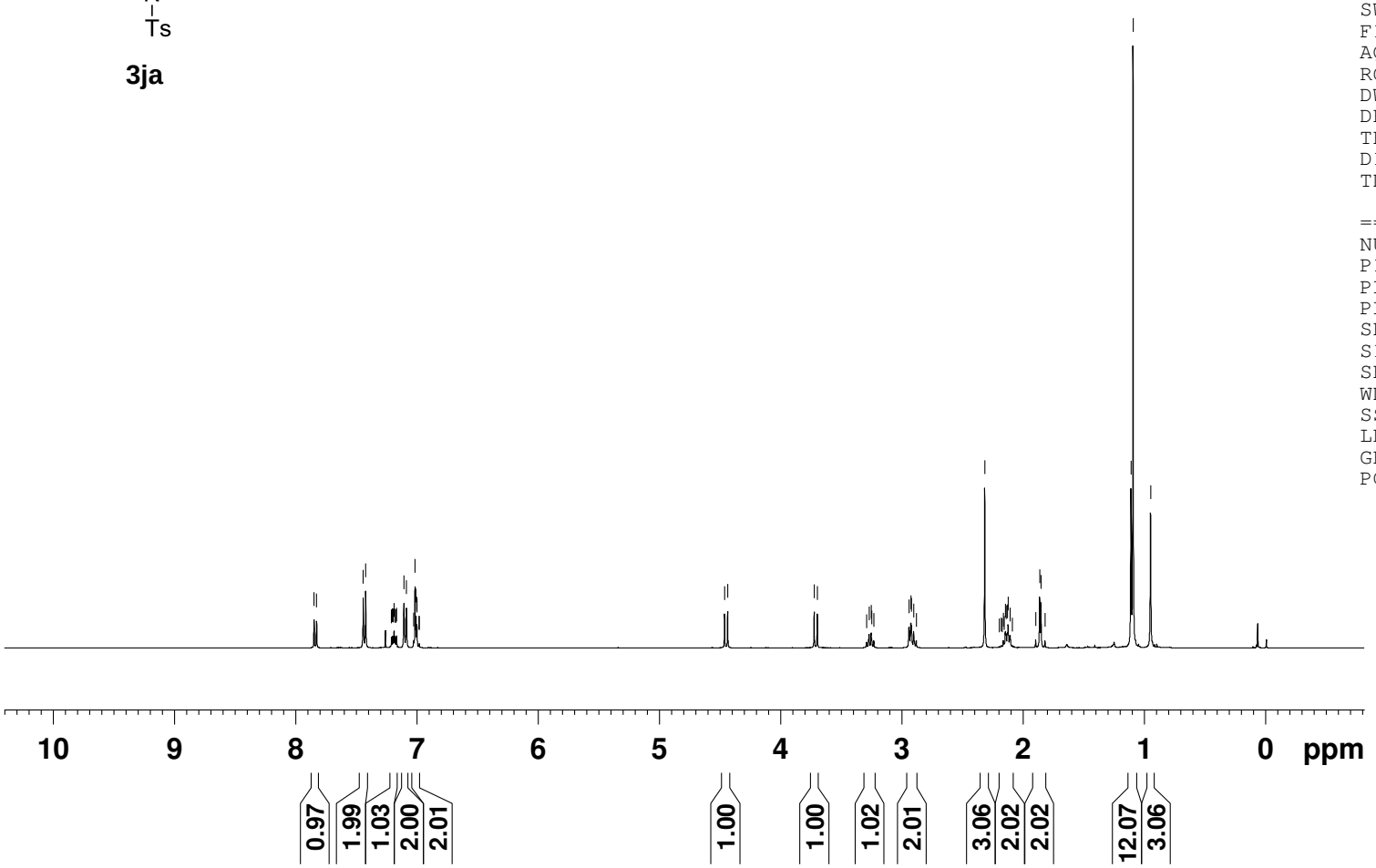

```
NAME      lly-884-1p-20170414
EXPNO                       2
PROCNO                      1
Date_              20170414
Time                   20.58
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        184
DS                          4
SWH                25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                     295.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        10

======== CHANNEL f1 ========
NUC1                      13C
P1                      13.50 usec
PL1                      3.00 dB
PL1W             43.93649673 W
SFO1             100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2             400.1316005 MHz
SI                      32768
SF               100.6127764 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

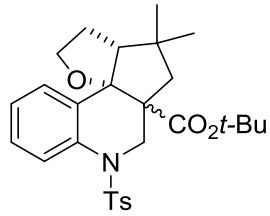




3ja

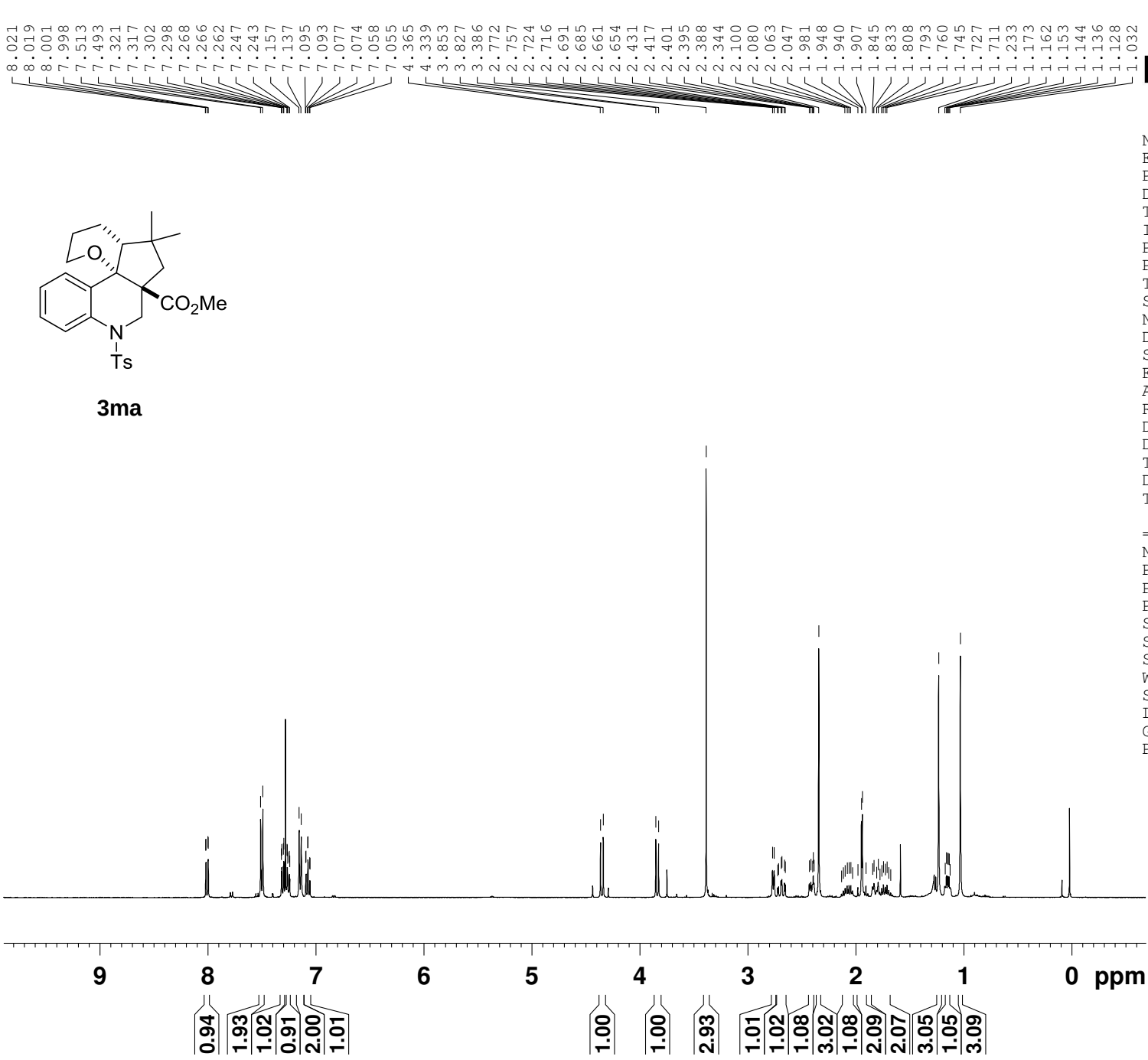
```
NAME      lly-904-1p-20170602
EXPNO                       1
PROCNO                      1
Date_                20170602
Time                    20.46
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                2.5625076 sec
RG                       90.5
DW                     78.200 usec
DE                      6.50 usec
TE                     293.5 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



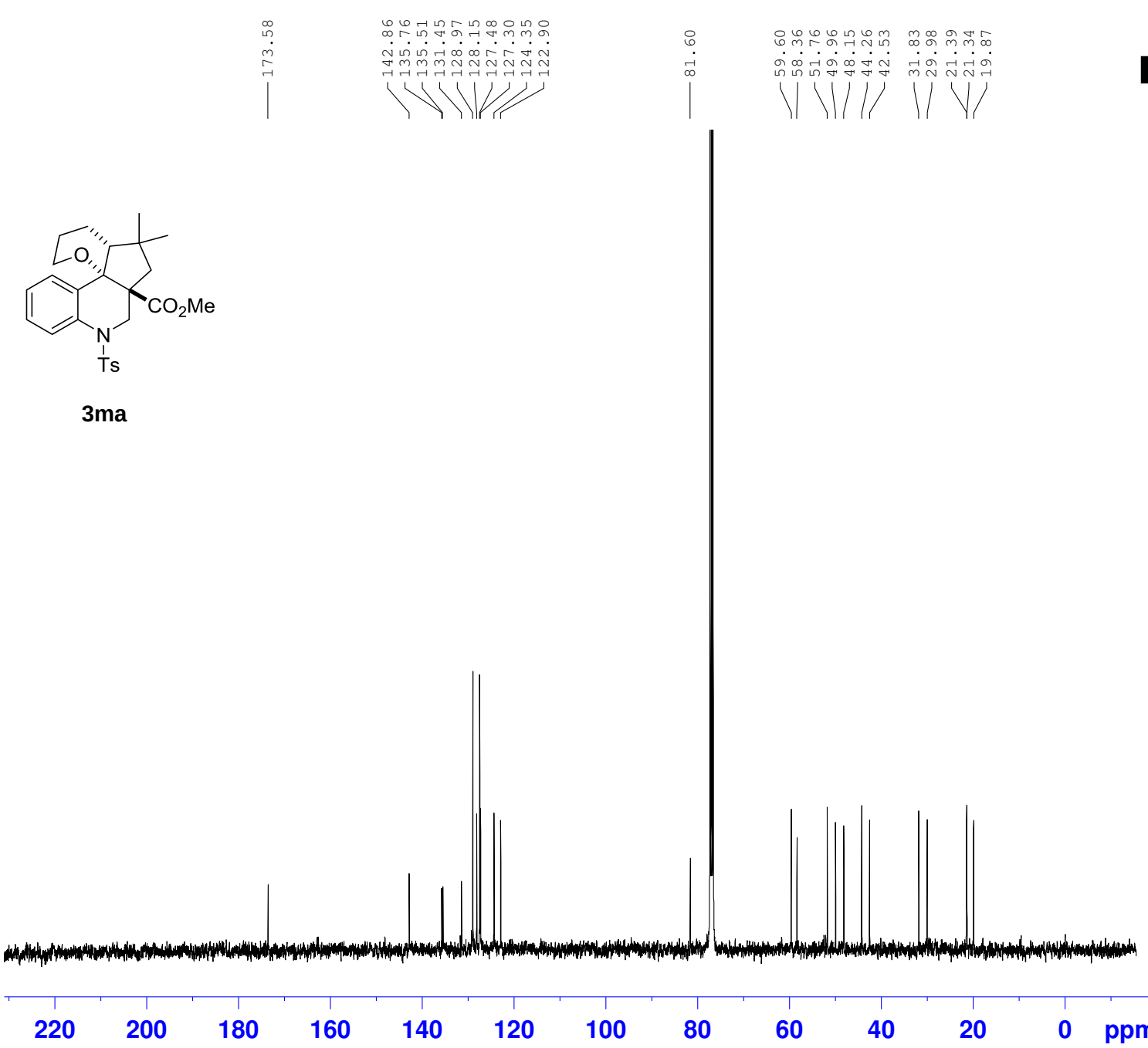


3ja


```
NAME          lly-904-1p-20170602
EXPNO                           2
PROCNO                          1
Date_                    20170602
Time                        20.49
INSTRUM                     spect
PROBHD       5 mm PADUL 13C
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           376
DS                             4
SWH                    25252.525 Hz
FIDRES                 0.385323 Hz
AQ                     1.2976629 sec
RG                          2050
DW                        19.800 usec
DE                         8.00 usec
TE                        294.4 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            10

======== CHANNEL f1 ========
NUC1                          13C
P1                         13.50 usec
PL1                         3.00 dB
PL1W                43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         1.80 dB
PL12                       17.19 dB
PL13                       18.46 dB
PL2W                 8.92857742 W
PL12W                0.25809658 W
PL13W                0.19265592 W
SFO2               400.1316005 MHz
SI                         32768
SF                 100.6127750 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.40
```

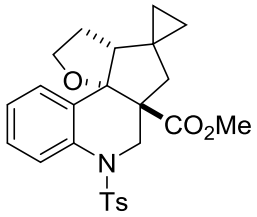



```
NAME      lly-882-1p-20170412
EXPNO                       1
PROCNO                      1
Date_              20170412
Time                   16.32
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       203
DW                    78.200 usec
DE                     6.50 usec
TE                    294.5 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



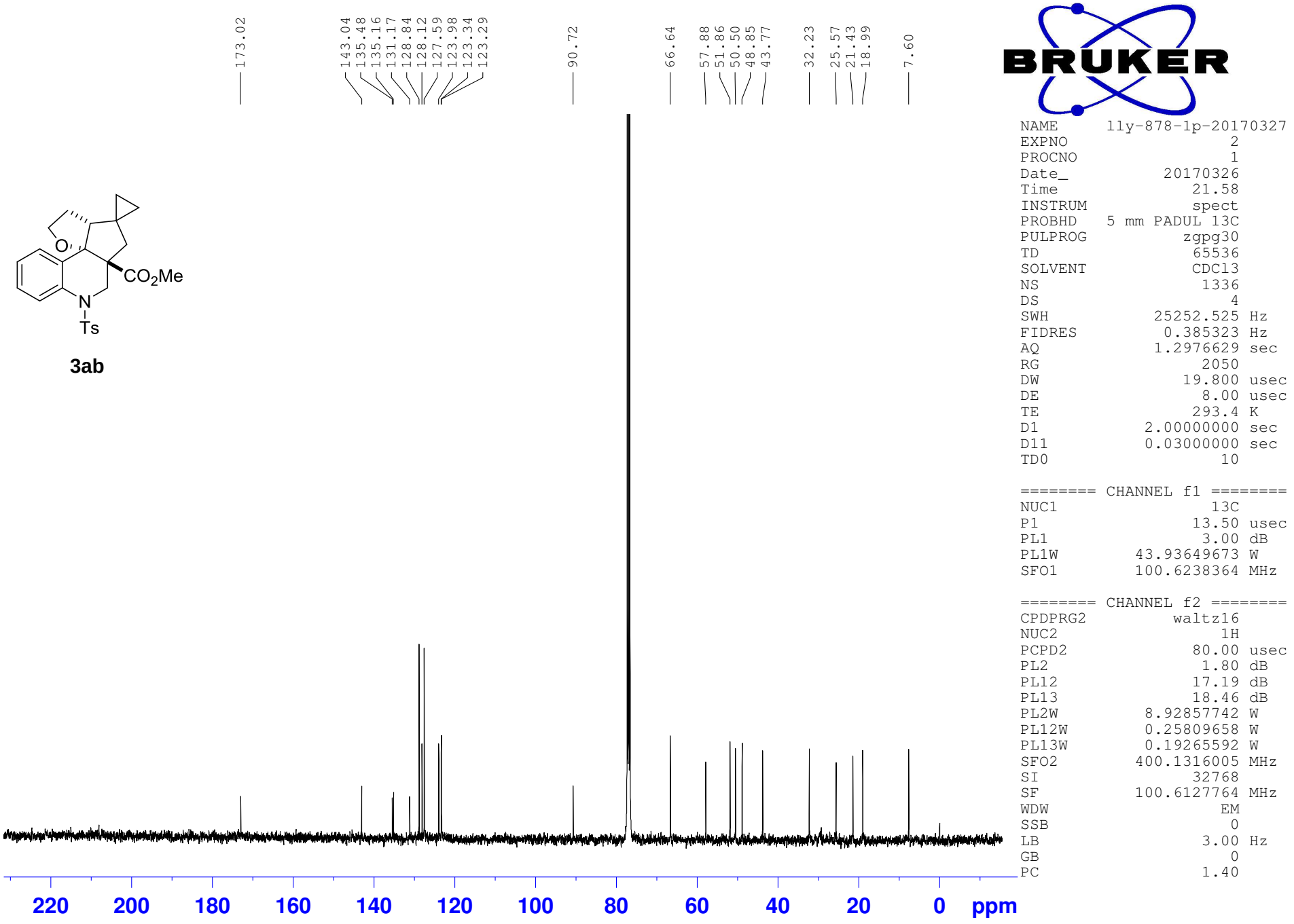
3ma

```
NAME      lly-882-1p-c-20170412
EXPNO                         2
PROCNO                        1
Date_                  20170412
Time                      18.37
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         864
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                   1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      295.3 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W                43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                        1.80 dB
PL12                      17.19 dB
PL13                      18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

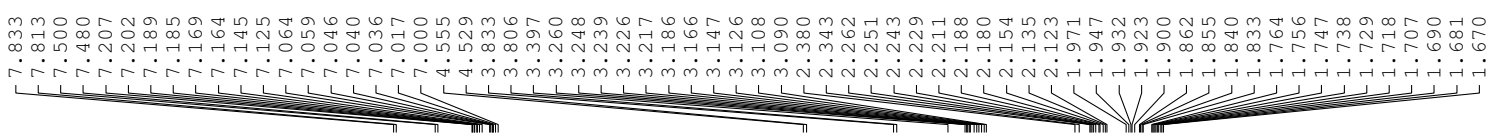


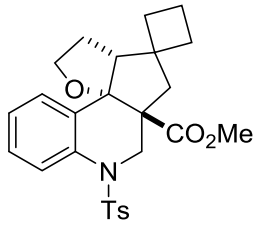
3ab


```
NAME      lly-878-1p-20170327
EXPNO                       1
PROCNO                      1
Date_              20170326
Time                   21.48
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       144
DW                    78.200 usec
DE                     6.50 usec
TE                    292.4 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



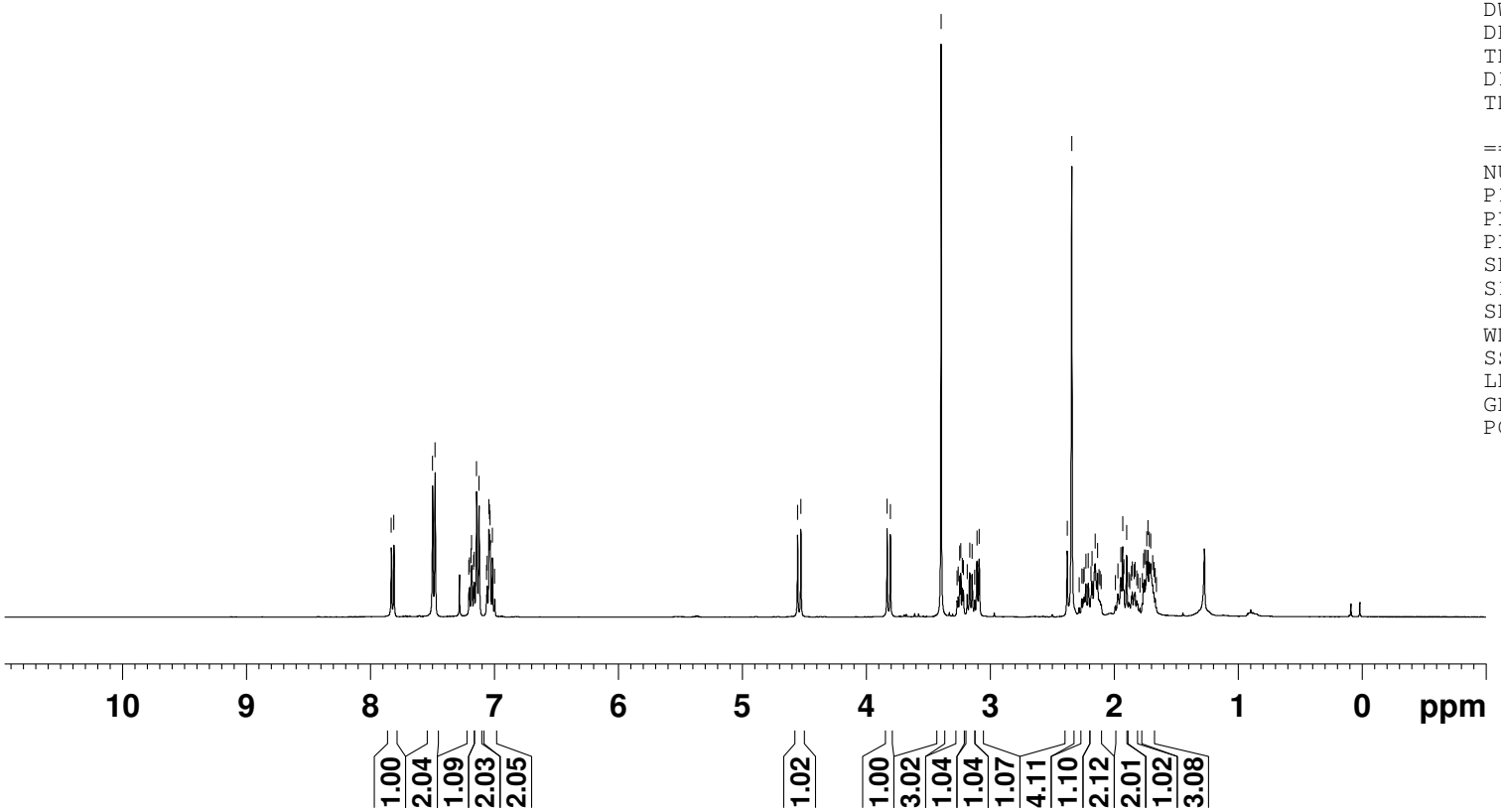

```
NAME      lly-878-1p-20170327
EXPNO                       2
PROCNO                      1
Date_                20170326
Time                    21.58
INSTRUM                 spect
PROBHD   5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1336
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                   1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      293.4 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                       3.00 Hz
GB                           0
PC                        1.40
```

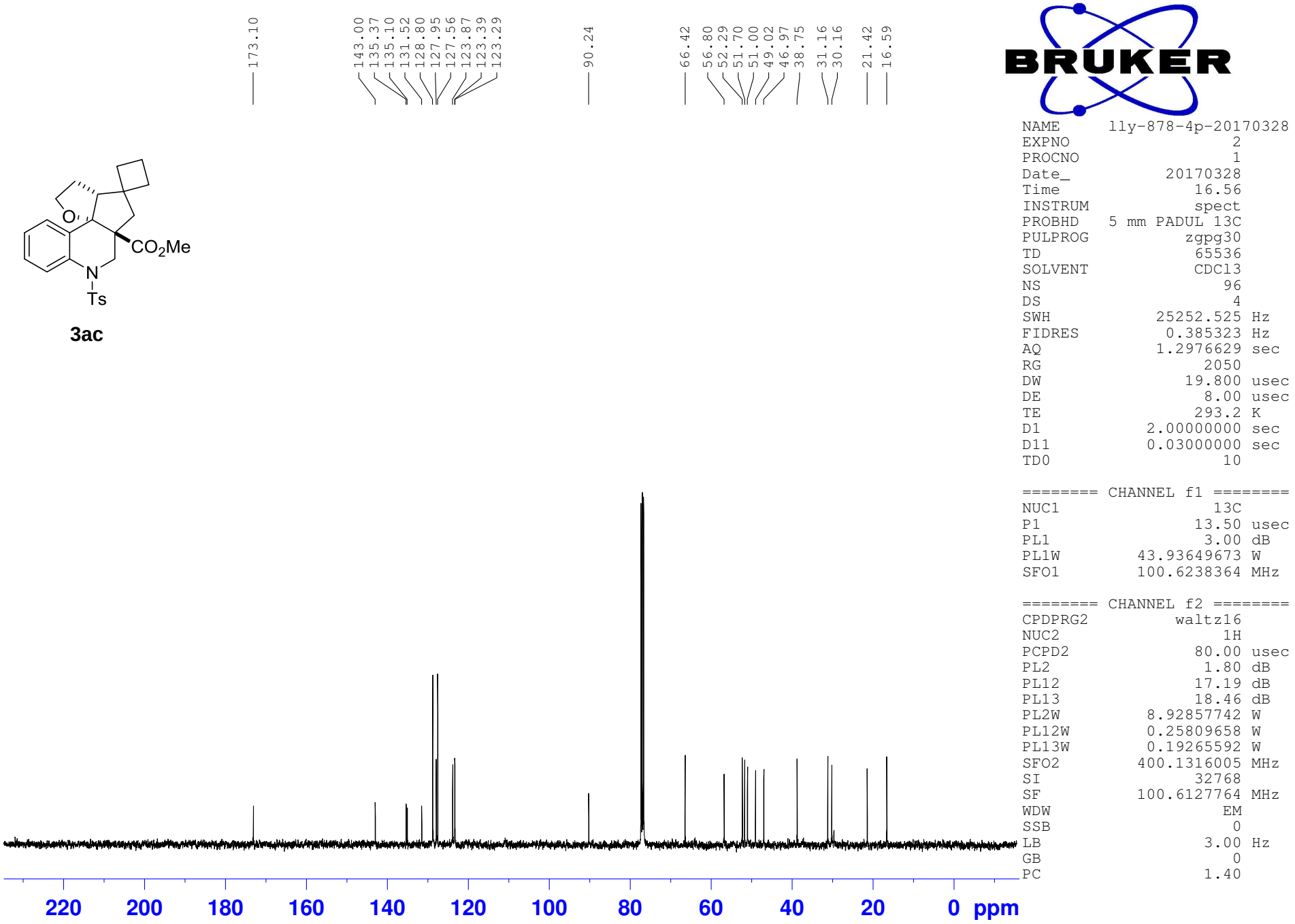


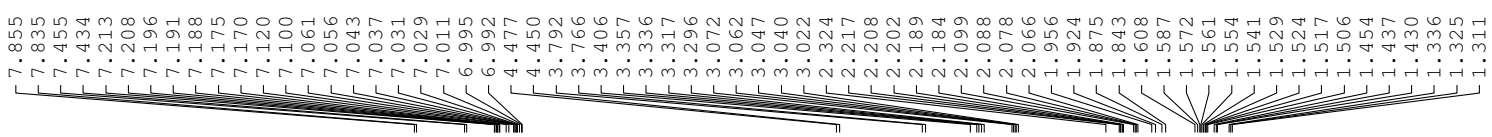


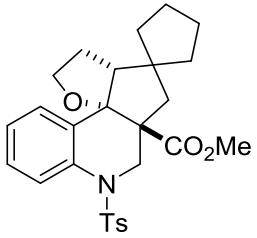
3ac

```
NAME      lly-878-4p-20170328
EXPNO                       1
PROCNO                      1
Date_              20170328
Time                   16.52
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       114
DW                    78.200 usec
DE                     6.50 usec
TE                    292.4 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



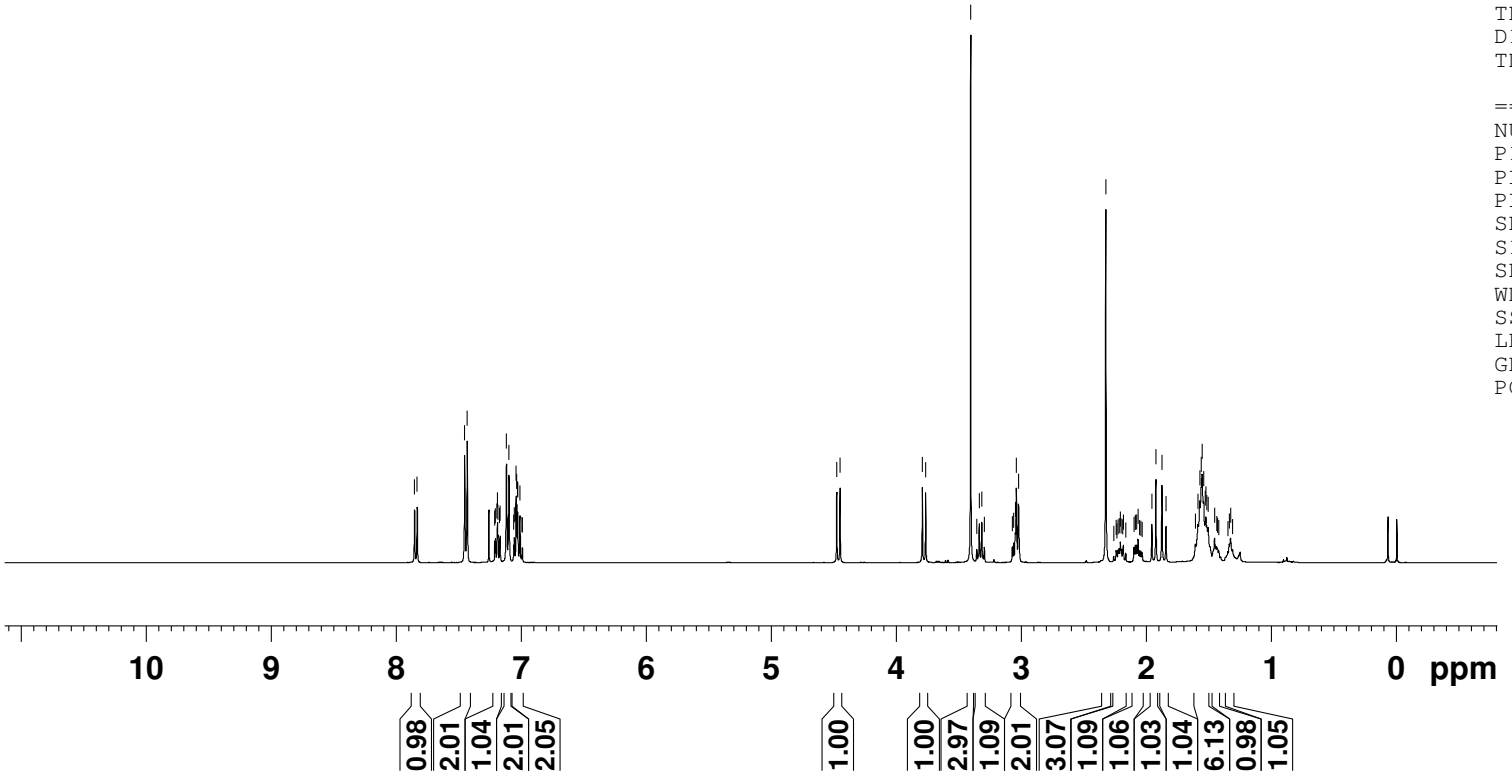

173.10
143.00
135.37
135.10
131.52
128.80
127.95
127.56
123.87
123.39
123.29
90.24
66.42
56.80
52.29
51.70
51.00
49.02
46.97
38.75
31.16
30.16
21.42
16.59
O
CO2Me
N
Ts
3ac
NAME lly-878-4p-20170328
EXPNO 2
PROCNO 1
Date_ 20170328
Time 16.56
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 2050
DW 19.800 usec
DE 8.00 usec
TE 293.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 10
======== CHANNEL f1 ========
NUC1 13C
P1 13.50 usec
PL1 3.00 dB
PL1W 43.93649673 W
SFO1 100.6238364 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.80 dB
PL12 17.19 dB
PL13 18.46 dB
PL2W 8.92857742 W
PL12W 0.25809658 W
PL13W 0.19265592 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127764 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
0 ppm

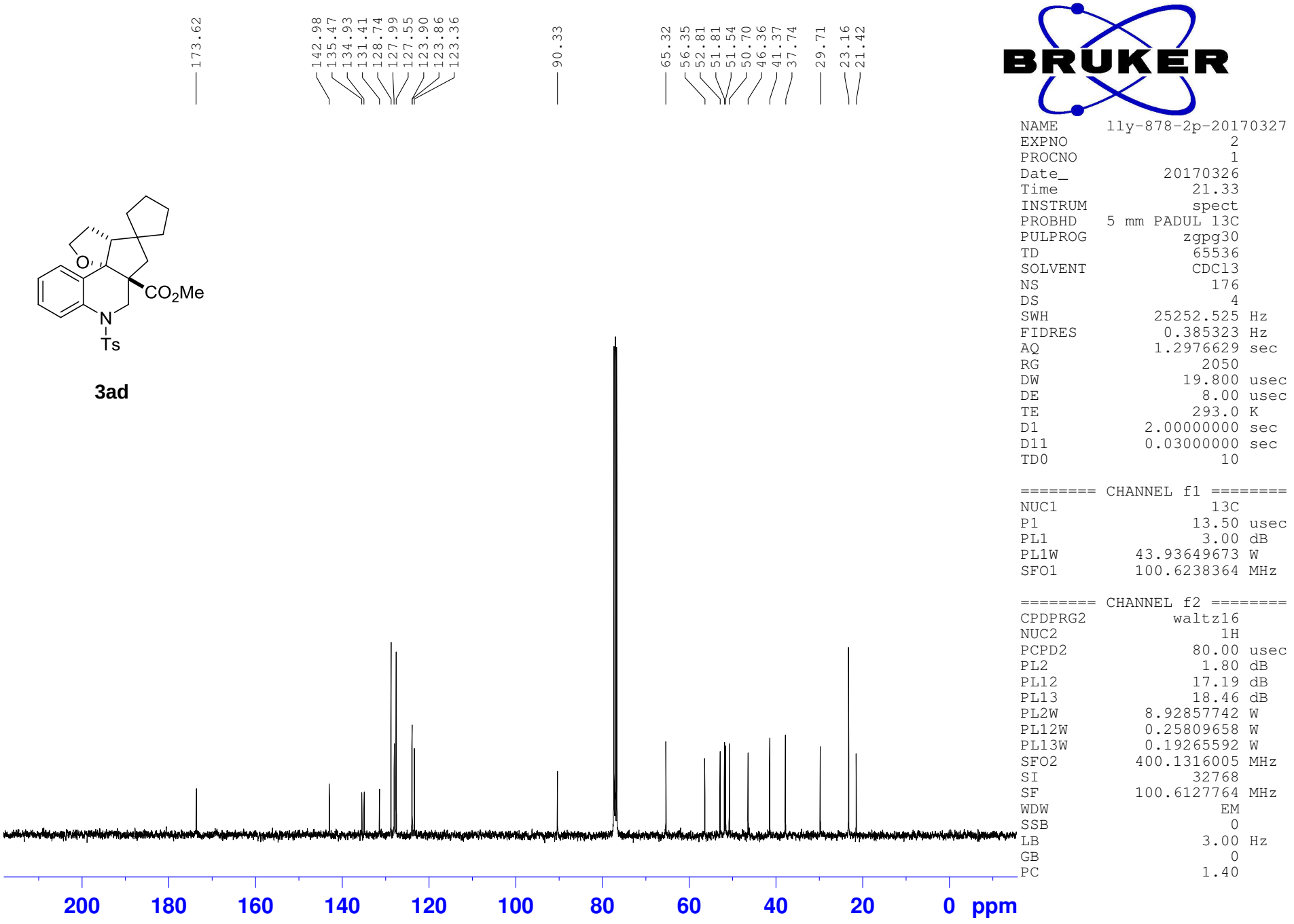




3ad

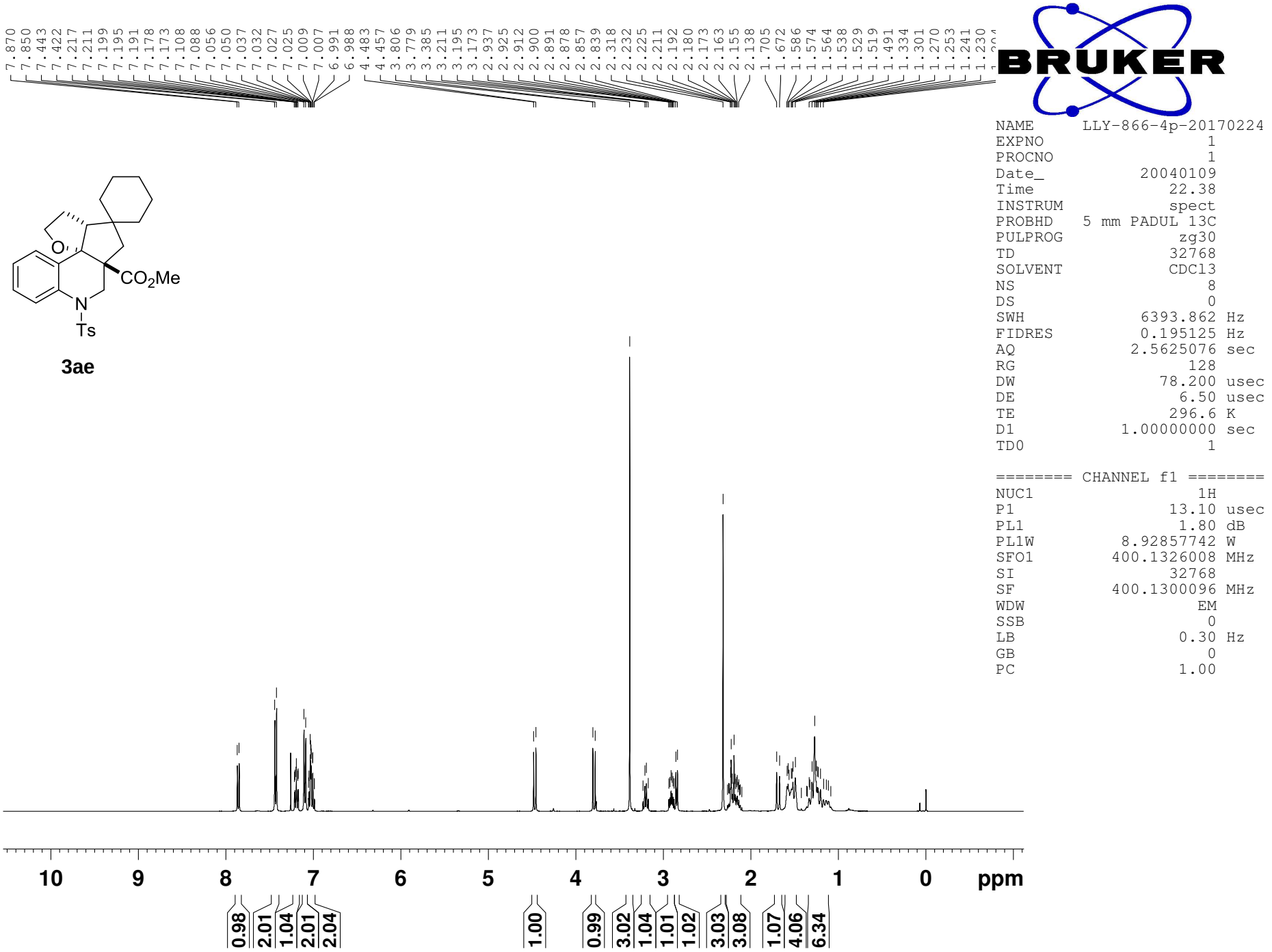
```
NAME      lly-878-2p-20170327
EXPNO                       1
PROCNO                      1
Date_              20170326
Time                   21.29
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       144
DW                    78.200 usec
DE                     6.50 usec
TE                    292.0 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

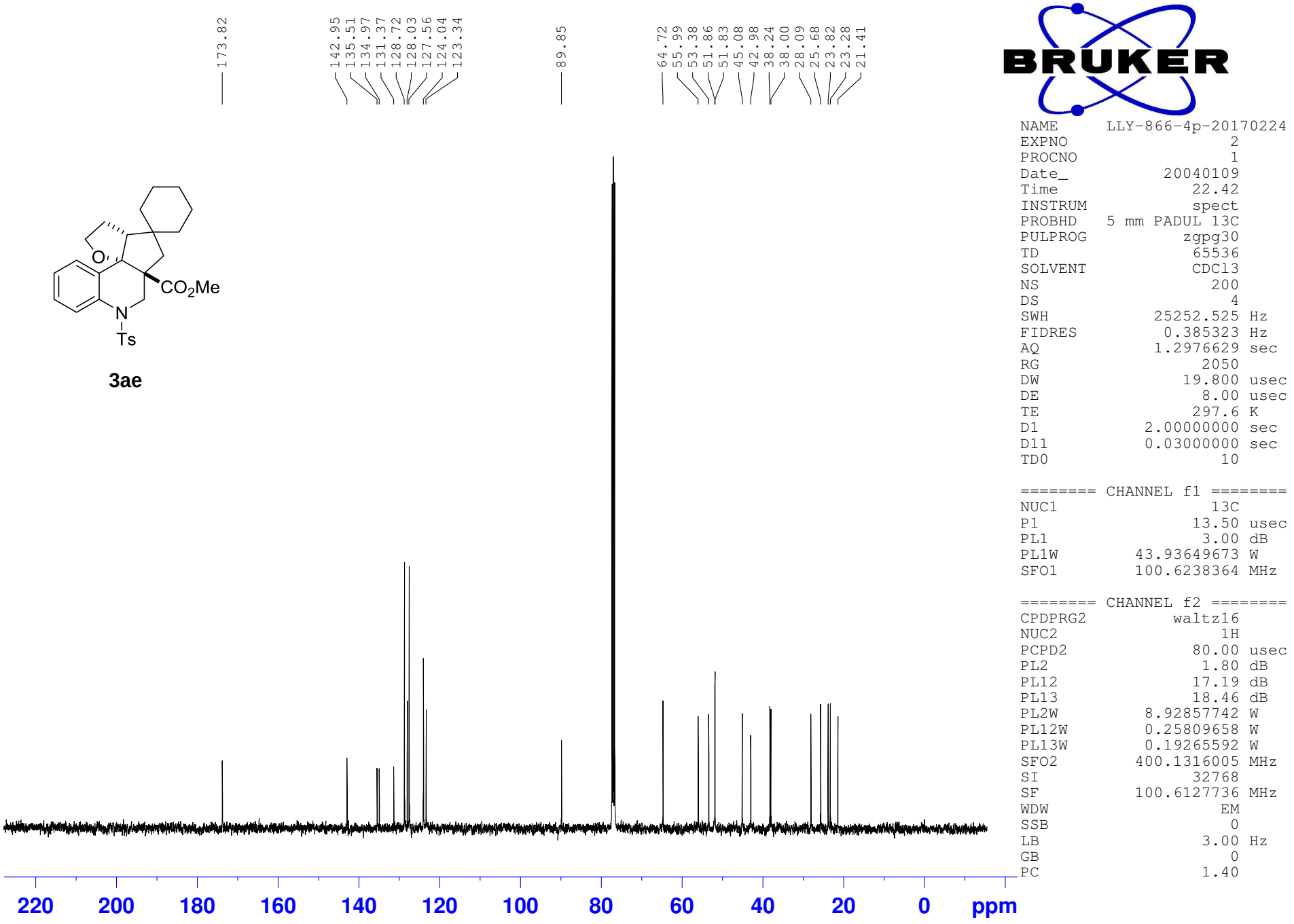




BRUKER

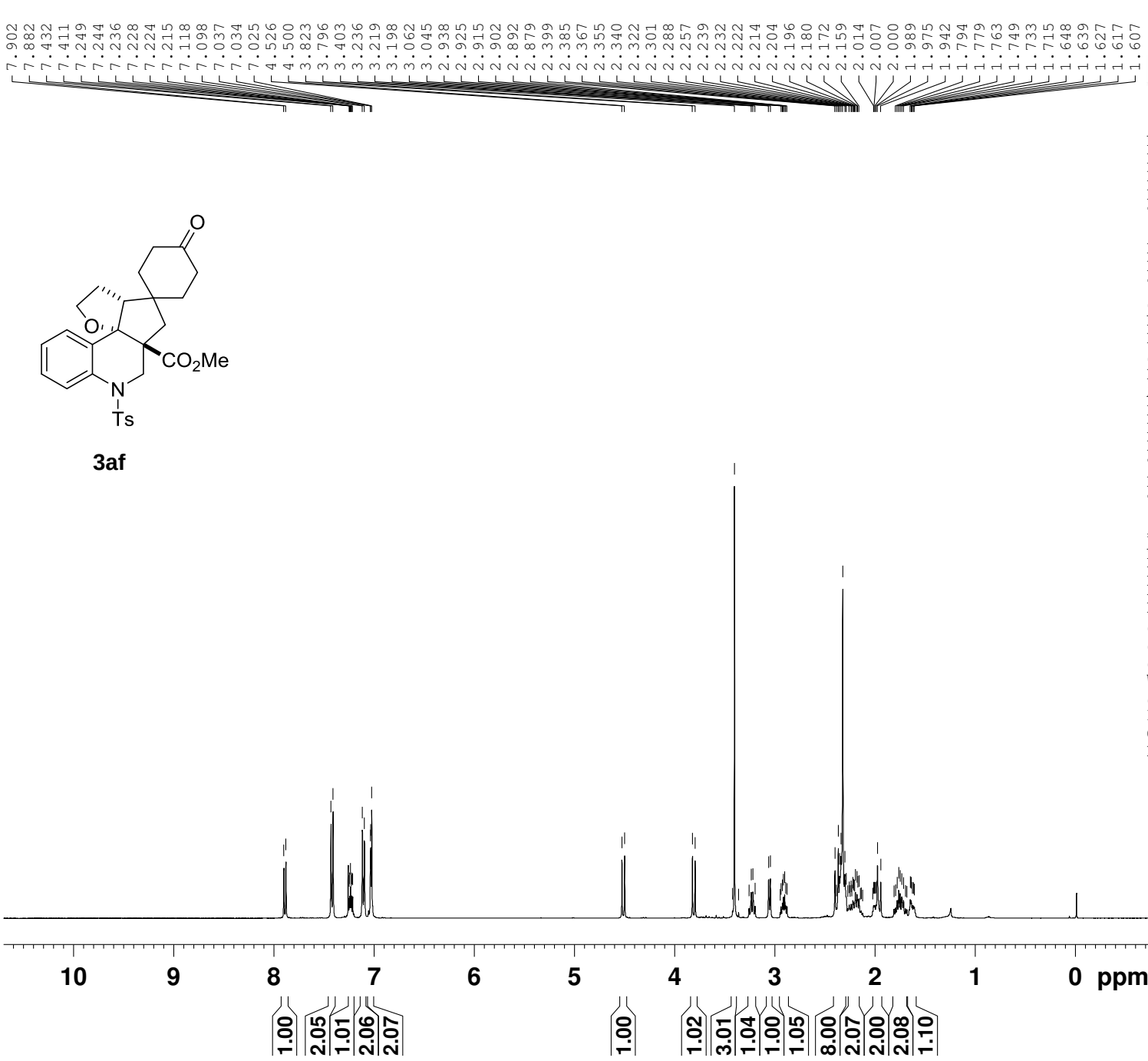
NAME	lly-878-2p-20170327
EXPNO	2
PROCNO	1
Date_	20170326
Time	21.33
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	176
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	8.00 usec
TE	293.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	13.50 usec
PL1	3.00 dB
PL1W	43.93649673 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.80 dB
PL12	17.19 dB
PL13	18.46 dB
PL2W	8.92857742 W
PL12W	0.25809658 W
PL13W	0.19265592 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127764 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40


7.870 7.850 7.443 7.422 7.217 7.211 7.199 7.195 7.191 7.178 7.173 7.108 7.088 7.056 7.050 7.037 7.032 7.027 7.025 7.009 7.007 6.991 6.988 4.483 4.457 3.806 3.779 3.385 3.211 3.195 3.173 2.937 2.925 2.912 2.900 2.891 2.878 2.857 2.839 2.318 2.232 2.225 2.211 2.192 2.180 2.173 2.163 2.155 2.138 1.705 1.672 1.586 1.574 1.564 1.538 1.529 1.519 1.491 1.334 1.301 1.270 1.253 1.241 1.230
BRUKER
O
CO2Me
N
Ts
3ae
NAME LLY-866-4p-20170224
EXPNO 1
PROCNO 1
Date_ 20040109
Time 22.38
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 6393.862 Hz
FIDRES 0.195125 Hz
AQ 2.5625076 sec
RG 128
DW 78.200 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.10 usec
PL1 1.80 dB
PL1W 8.92857742 W
SFO1 400.1326008 MHz
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10 9 8 7 6 5 4 3 2 1 0 ppm
0.98 2.01 1.04 2.01 2.04 1.00 0.99 3.02 1.04 1.01 1.02 3.03 3.08 1.07 4.06 6.34

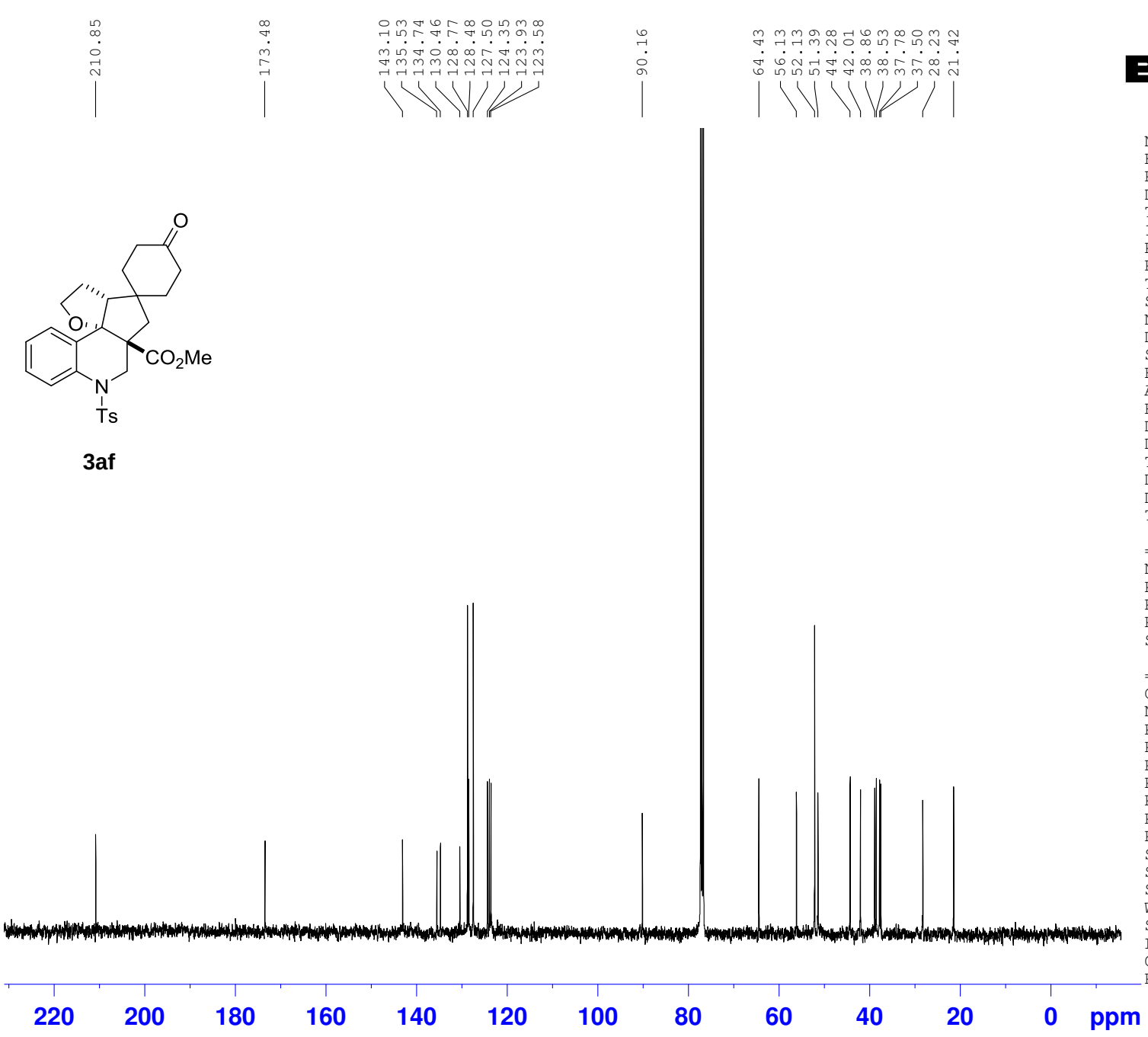


3ae

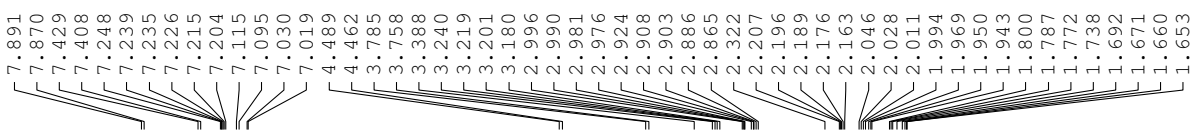
```
NAME        LLY-866-4p-20170224
EXPNO                         2
PROCNO                        1
Date_                  20040109
Time                      22.42
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                         65536
SOLVENT                  CDCl3
NS                           200
DS                             4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                          2050
DW                        19.800 usec
DE                         8.00 usec
TE                        297.6 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           10

======== CHANNEL f1 ========
NUC1                         13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                        1.80 dB
PL12                      17.19 dB
PL13                      18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127736 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.40
```

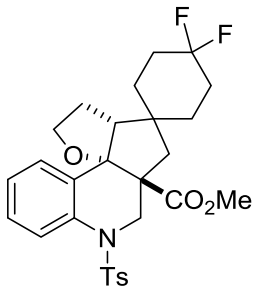



```
NAME      lly-877-2p-20170324
EXPNO                       1
PROCNO                      1
Date_              20170325
Time                   21.27
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       161
DW                    78.200 usec
DE                     6.50 usec
TE                    292.1 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



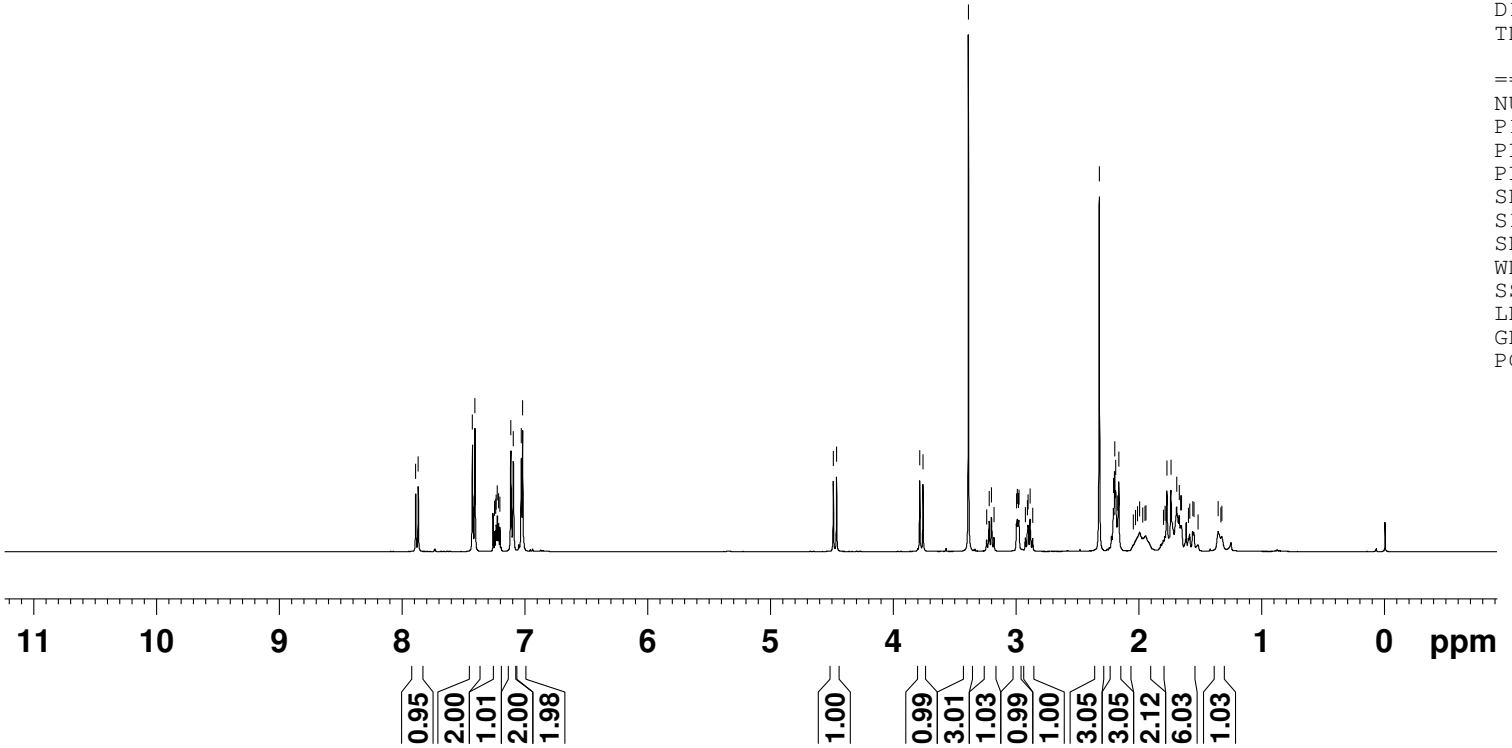

```
NAME        lly-877-2p-20170324
EXPNO                         2
PROCNO                        1
Date_                  20170325
Time                      21.30
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                         65536
SOLVENT                   CDCl3
NS                          216
DS                            4
SWH                   25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                         2050
DW                       19.800 usec
DE                        8.00 usec
TE                       292.9 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           10

======== CHANNEL f1 ========
NUC1                         13C
P1                         13.50 usec
PL1                         3.00 dB
PL1W                43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                     80.00 usec
PL2                        1.80 dB
PL12                      17.19 dB
PL13                      18.46 dB
PL2W                 8.92857742 W
PL12W                0.25809658 W
PL13W                0.19265592 W
SFO2               400.1316005 MHz
SI                         32768
SF                 100.6127764 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.40
```

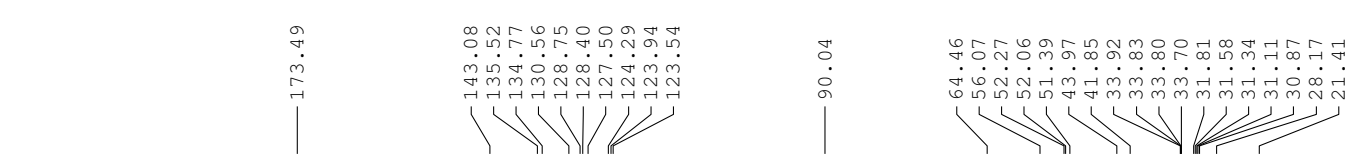


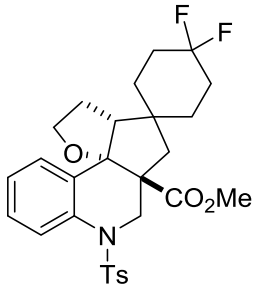


3ag

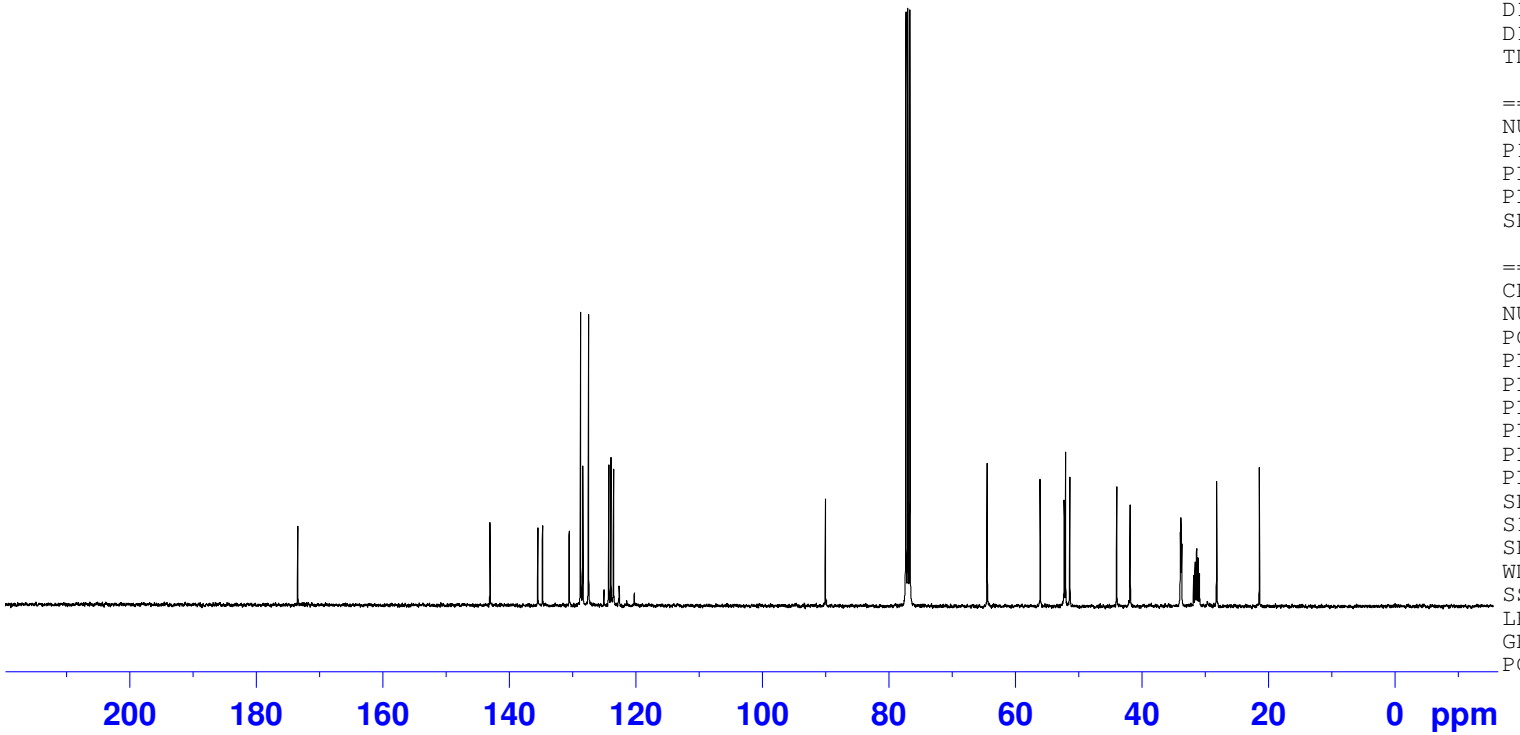
```
NAME      lly-877-3p-20170324
EXPNO                       1
PROCNO                      1
Date_              20170325
Time                   21.46
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       101
DW                    78.200 usec
DE                     6.50 usec
TE                    292.4 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



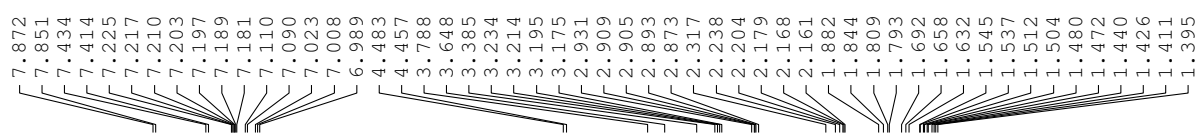


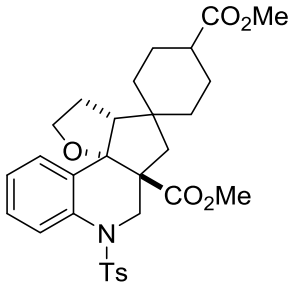


3ag



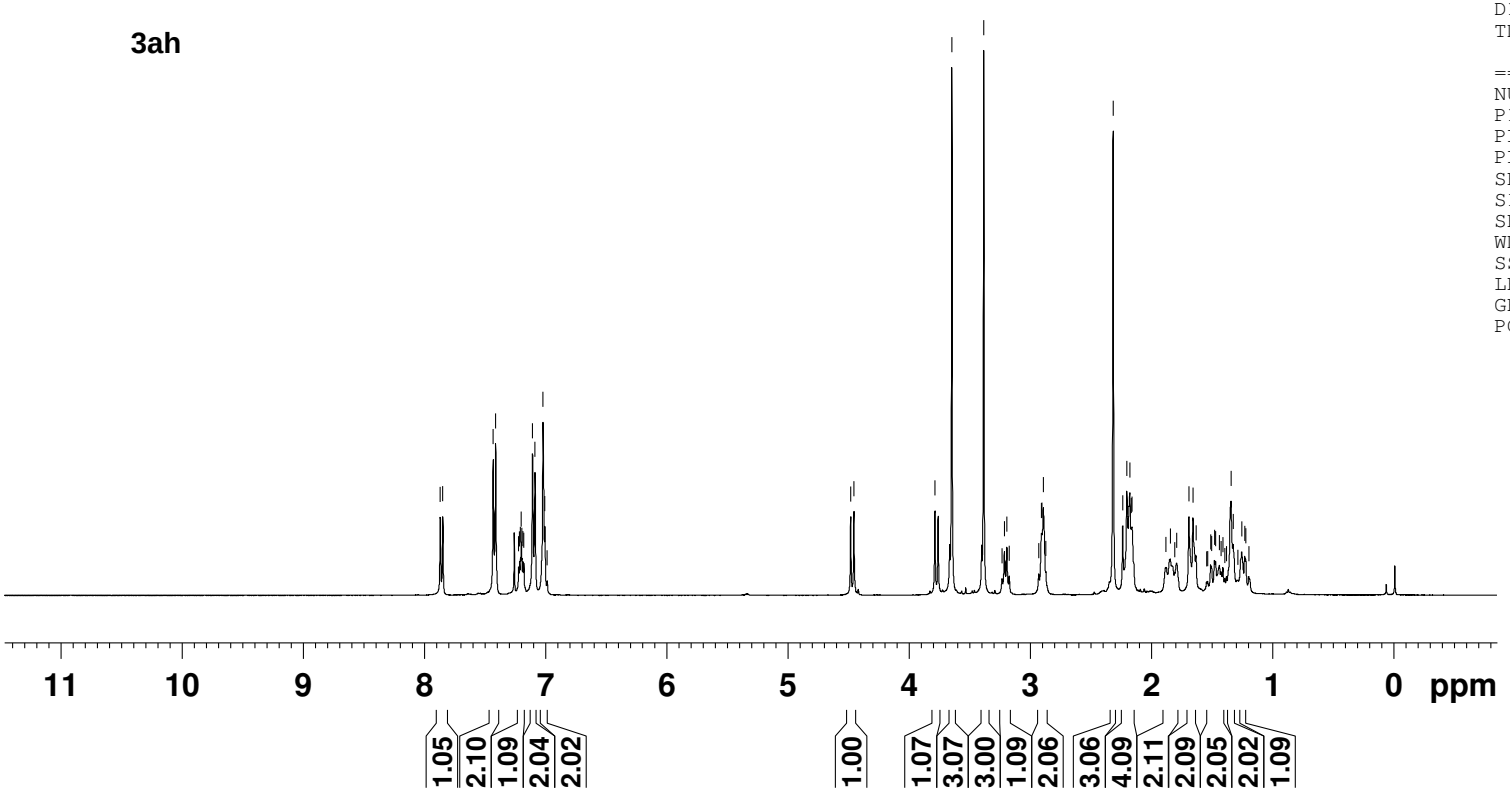

```
NAME        lly-877-3p-20170324
EXPNO                         2
PROCNO                        1
Date_                  20170325
Time                      21.49
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1248
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      293.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1              100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2              400.1316005 MHz
SI                       32768
SF                100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

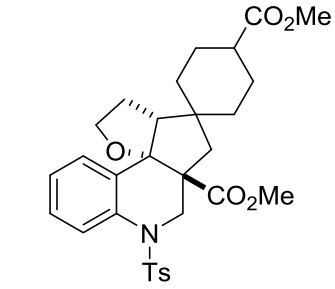




3ah

```
NAME        lly-878-1bp-a-20170329
EXPNO                            1
PROCNO                           1
Date_                     20170329
Time                         22.49
INSTRUM                      spect
PROBHD    5 mm PADUL 13C
PULPROG                       zg30
TD                           32768
SOLVENT                      CDCl3
NS                               8
DS                               0
SWH                       6393.862 Hz
FIDRES                    0.195125 Hz
AQ                     2.5625076 sec
RG                             144
DW                          78.200 usec
DE                           6.50 usec
TE                          292.6 K
D1                     1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                            1H
P1                          13.10 usec
PL1                          1.80 dB
PL1W                 8.92857742 W
SFO1                400.1326008 MHz
SI                           32768
SF                  400.1300096 MHz
WDW                             EM
SSB                              0
LB                           0.30 Hz
GB                               0
PC                           1.00
```

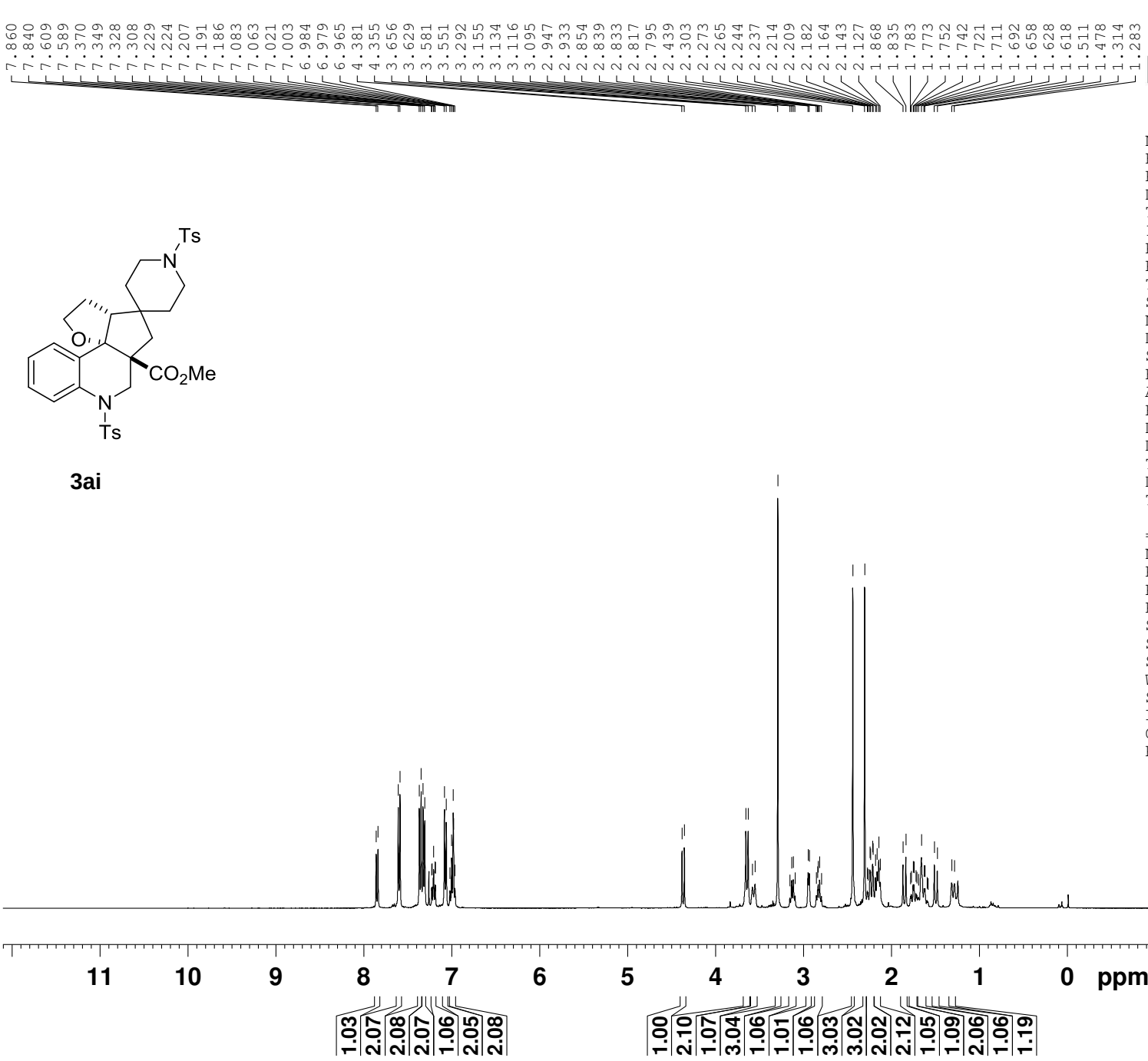




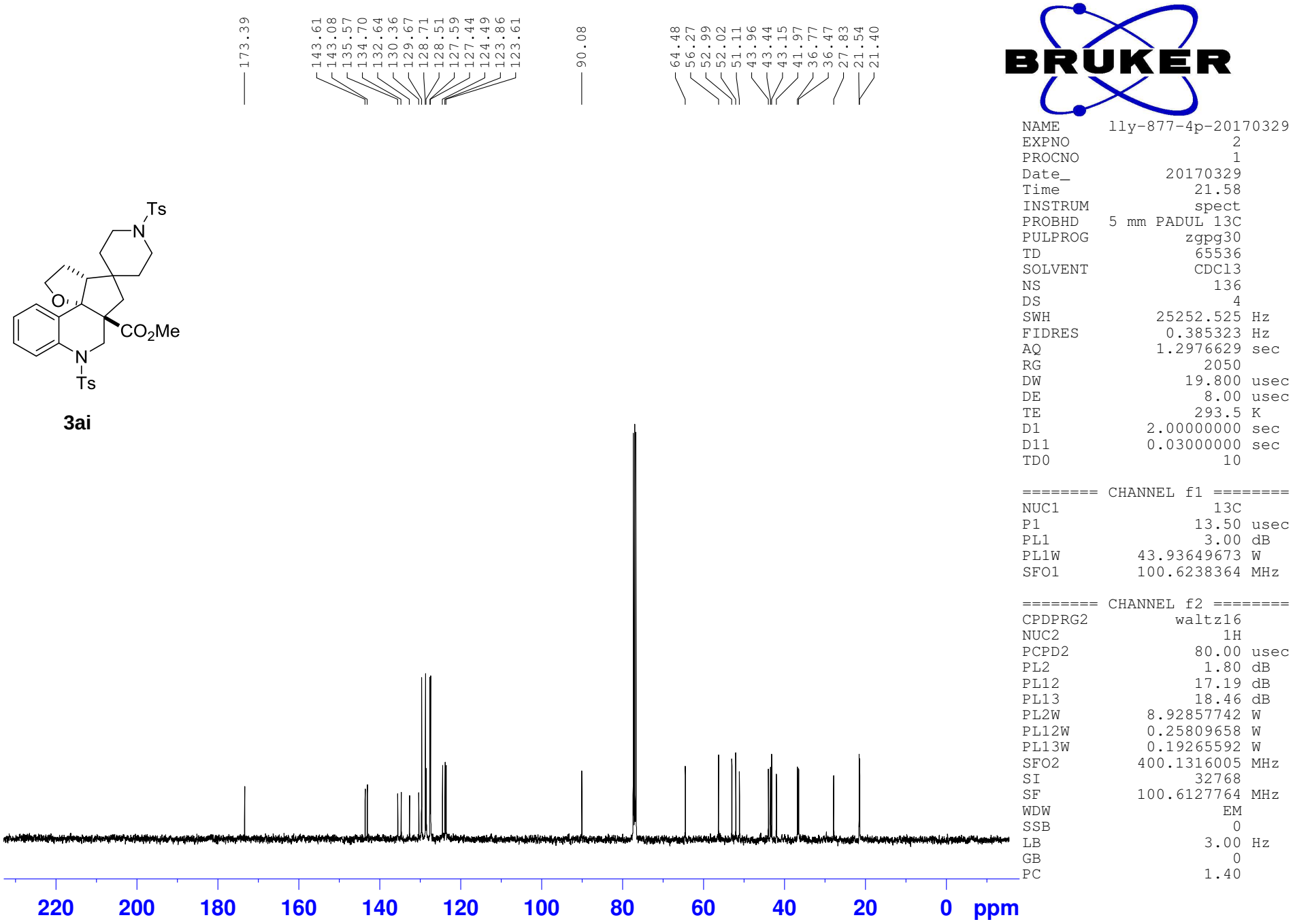
3ah

176.04
173.59
142.99
135.50
134.86
130.94
128.73
128.18
127.51
124.11
123.98
123.40
89.82
64.59
56.03
53.24
51.93
51.56
44.45
42.39
42.21
36.96
36.71
28.00
26.27
25.68
21.41

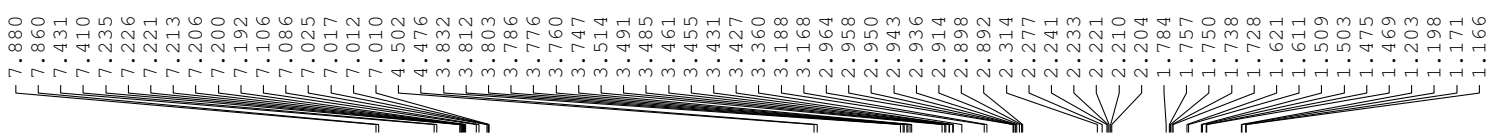

```
NAME      lly-878-1bp-a-20170329
EXPNO                          2
PROCNO                         1
Date_               20170329
Time                     22.54
INSTRUM                  spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         320
DS                           4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      293.8 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                       13C
P1                      13.50 usec
PL1                      3.00 dB
PL1W              43.93649673 W
SFO1              100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      1.80 dB
PL12                    17.19 dB
PL13                    18.46 dB
PL2W               8.92857742 W
PL12W              0.25809658 W
PL13W              0.19265592 W
SFO2              400.1316005 MHz
SI                       32768
SF                100.6127764 MHz
WDW                         EM
SSB                          0
LB                       3.00 Hz
GB                           0
PC                       1.40
```

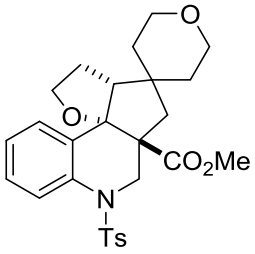



```
NAME      lly-877-4p-20170329
EXPNO                       1
PROCNO                      1
Date_              20170329
Time                  21.53
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                6393.862 Hz
FIDRES            0.195125 Hz
AQ                2.5625076 sec
RG                      101
DW                   78.200 usec
DE                    6.50 usec
TE                   292.5 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   13.10 usec
PL1                   1.80 dB
PL1W           8.92857742 W
SFO1          400.1326008 MHz
SI                    32768
SF            400.1300096 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



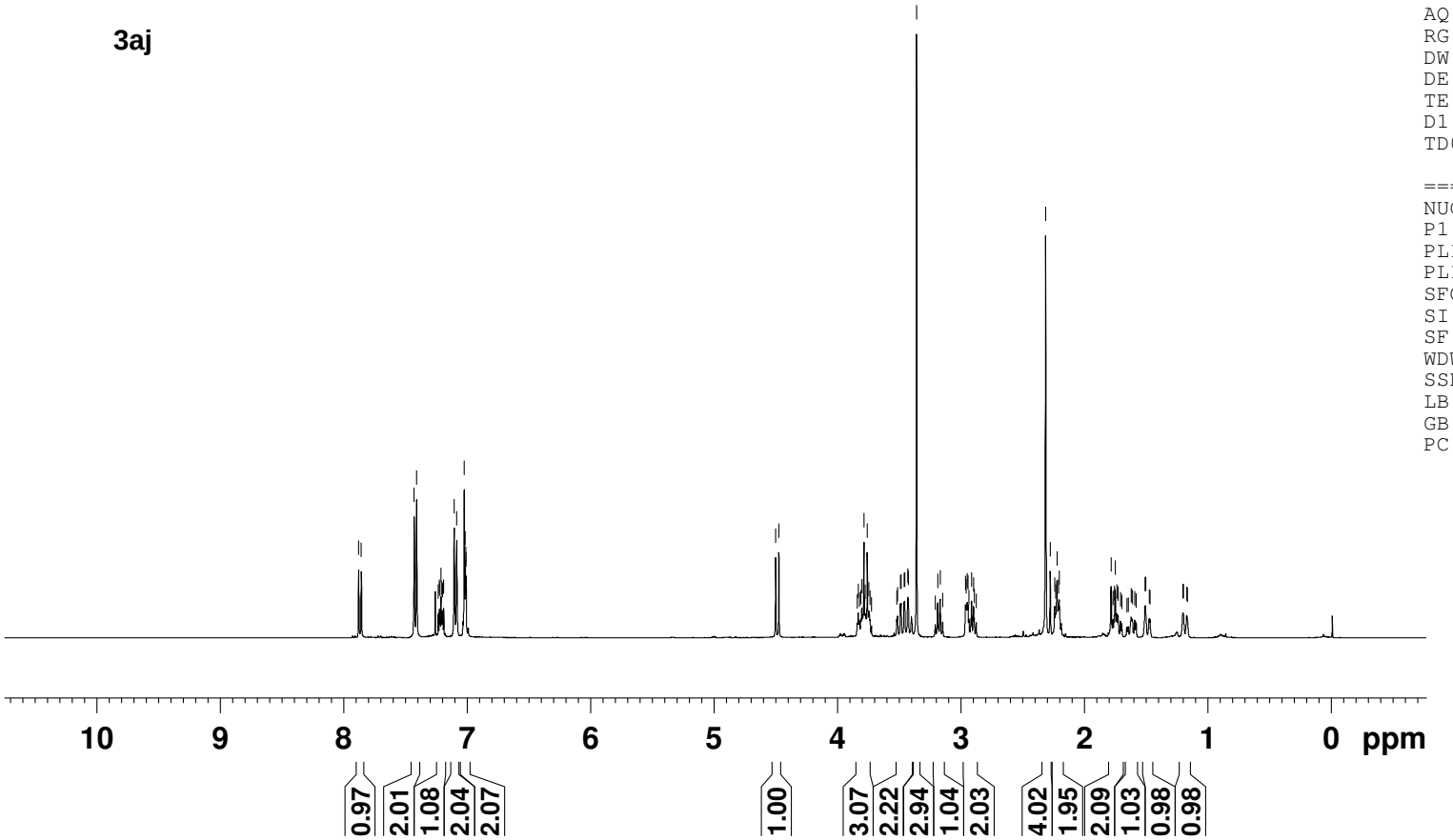

```
NAME        lly-877-4p-20170329
EXPNO                         2
PROCNO                        1
Date_                  20170329
Time                      21.58
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         136
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      293.5 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     80.00 usec
PL2                        1.80 dB
PL12                      17.19 dB
PL13                      18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.40
```

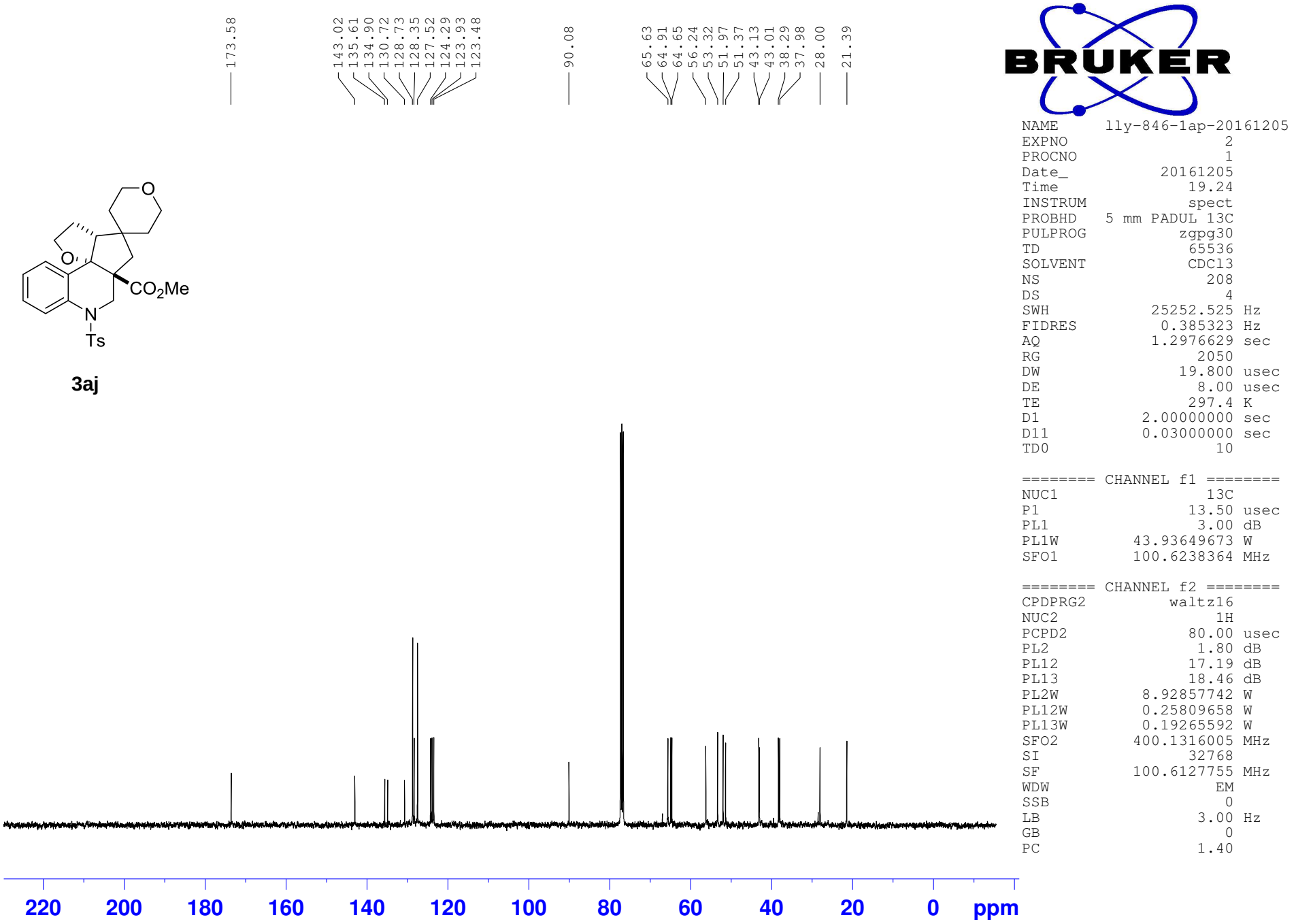




3aj

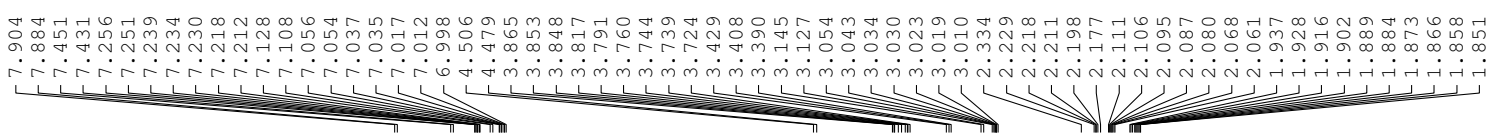
```
NAME      lly-846-1ap-20161205
EXPNO                        1
PROCNO                       1
Date_             20161205
Time                    19.19
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                2.5625076 sec
RG                        144
DW                     78.200 usec
DE                      6.50 usec
TE                     296.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

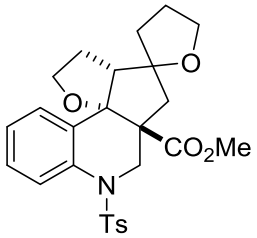




BRUKER

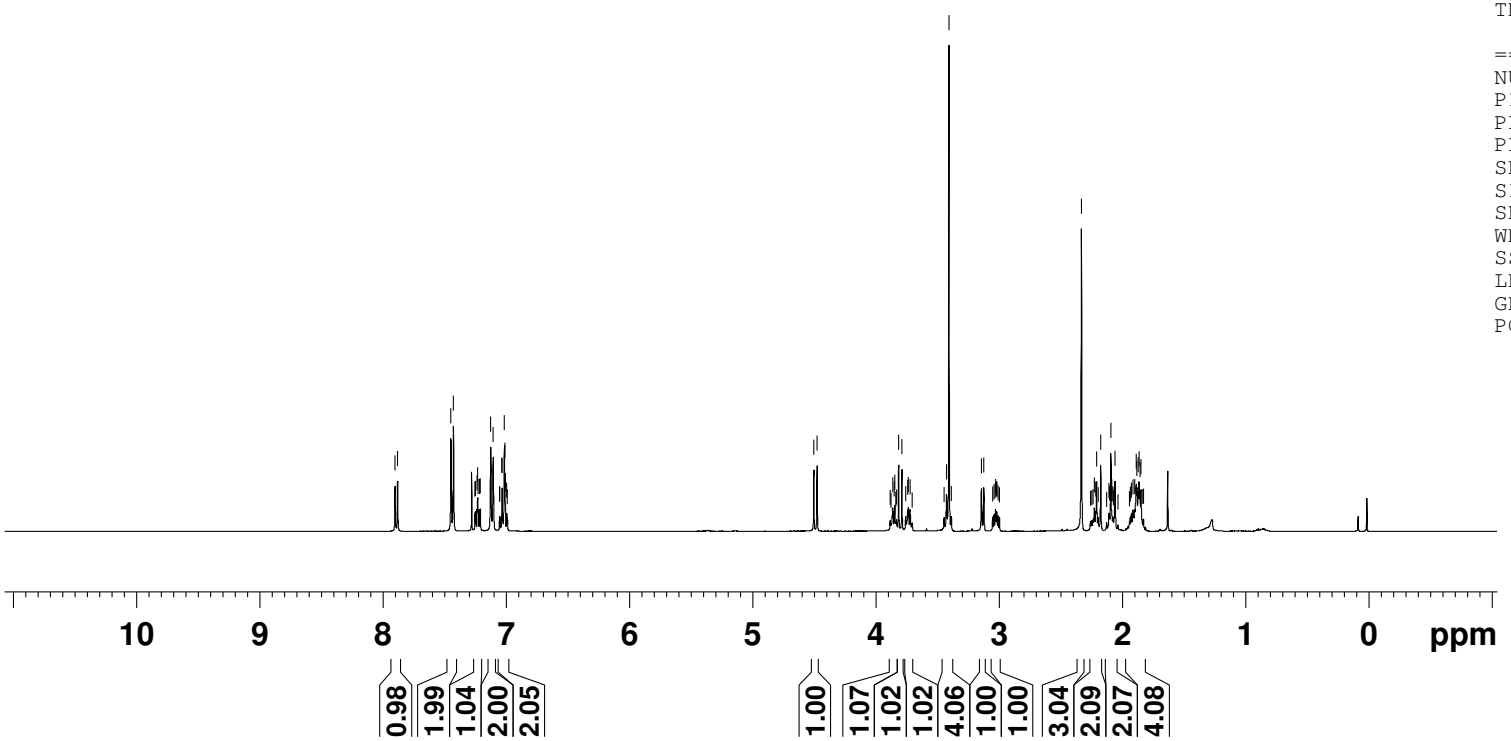
NAME	lly-846-1ap-20161205
EXPNO	2
PROCNO	1
Date_	20161205
Time	19.24
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	208
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	8.00 usec
TE	297.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	13.50 usec
PL1	3.00 dB
PL1W	43.93649673 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.80 dB
PL12	17.19 dB
PL13	18.46 dB
PL2W	8.92857742 W
PL12W	0.25809658 W
PL13W	0.19265592 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127755 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

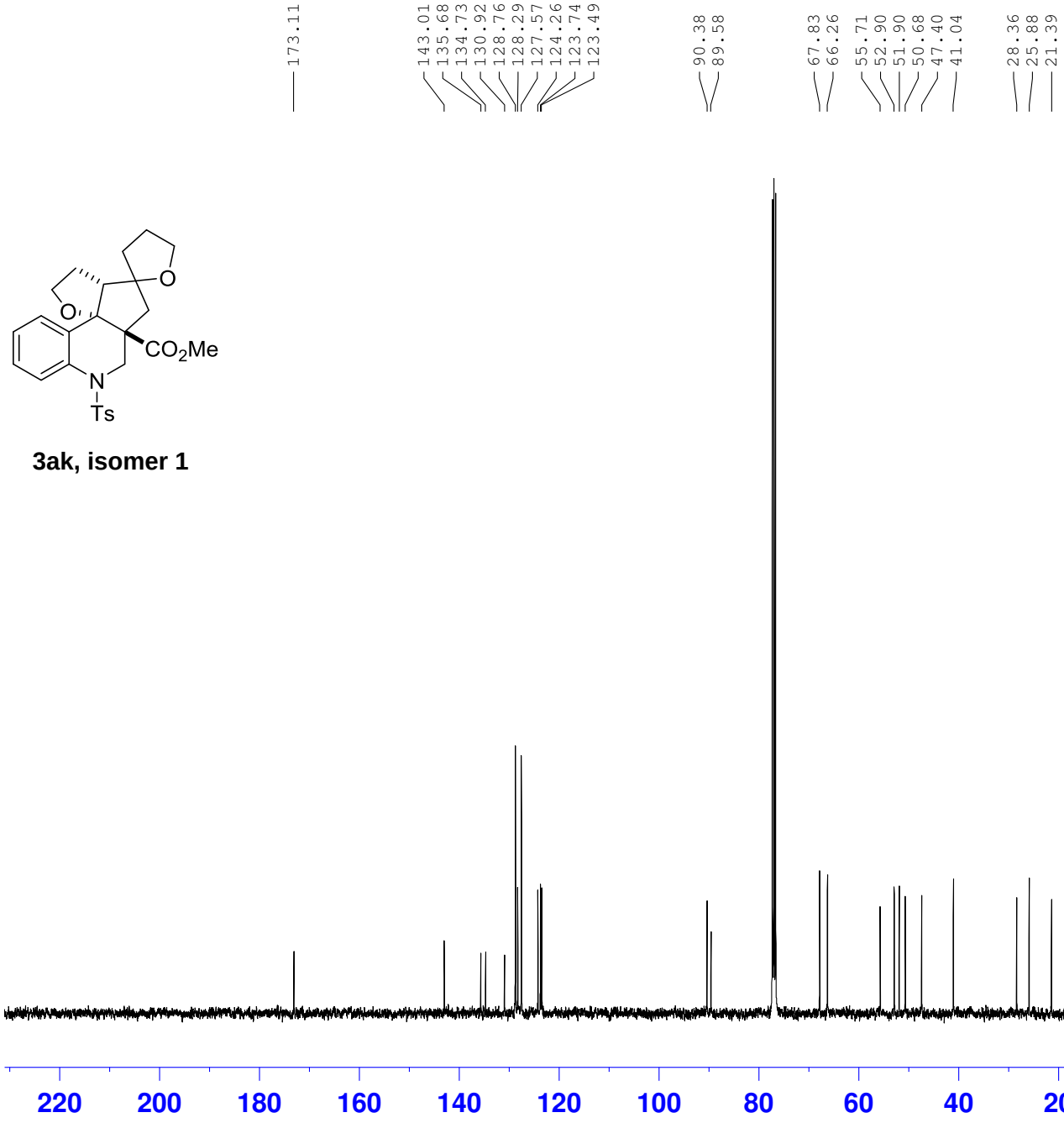




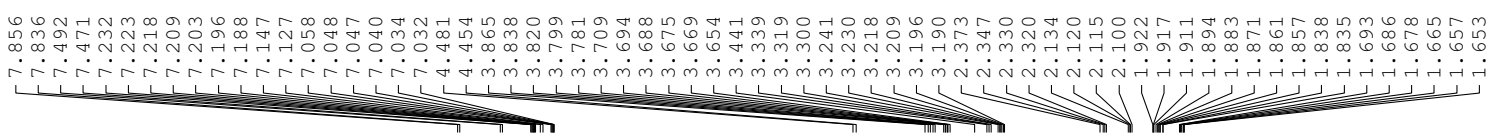
3ak, isomer 1

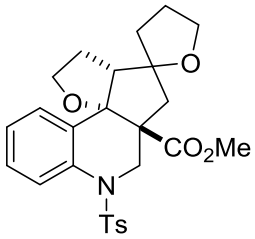
```
NAME      lly-880-3ap-20170406
EXPNO                        1
PROCNO                       1
Date_             20170406
Time                    17.29
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                 2.5625076 sec
RG                        181
DW                     78.200 usec
DE                      6.50 usec
TE                     293.6 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300005 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```





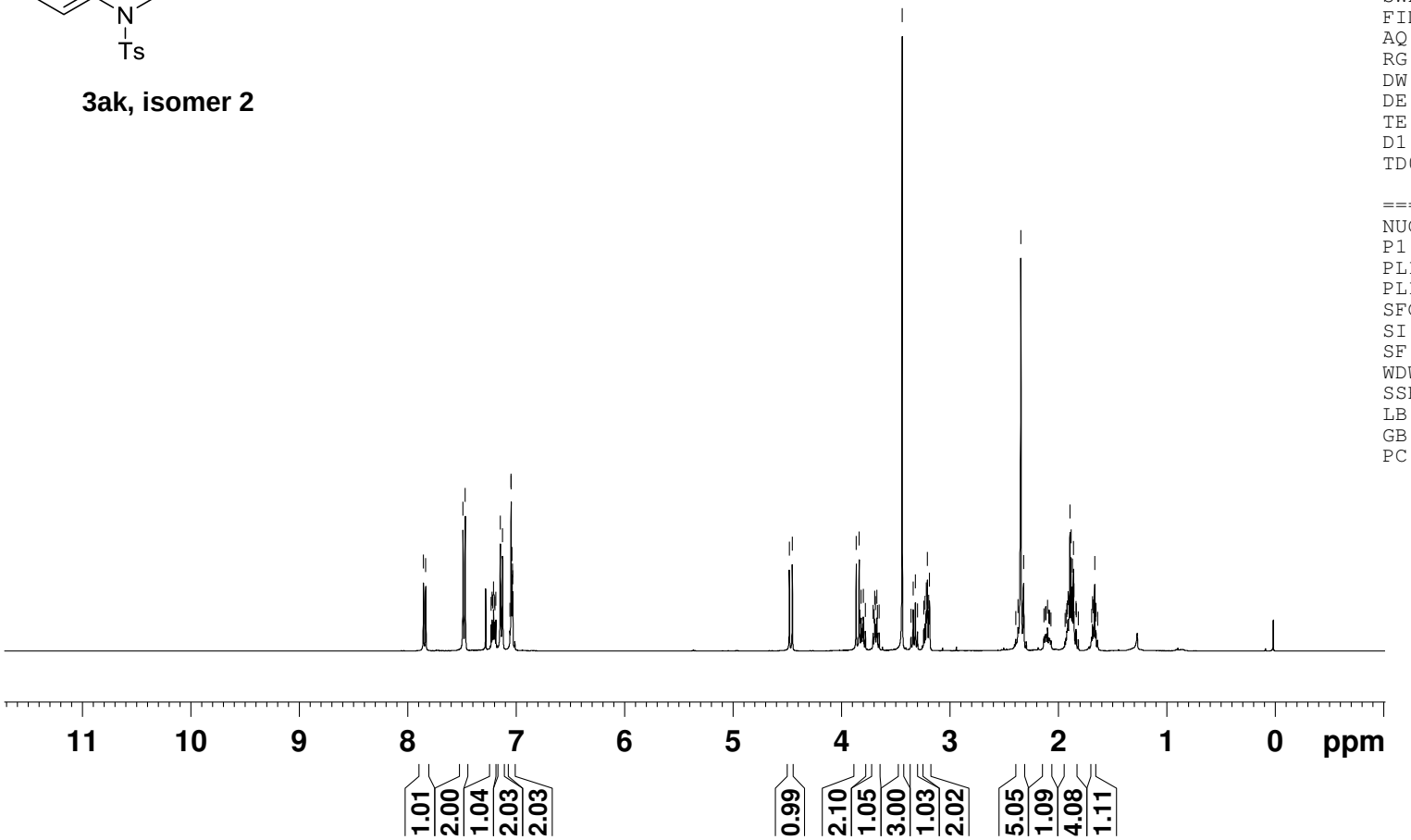

```
NAME      lly-880-3ap-20170406
EXPNO                        2
PROCNO                       1
Date_             20170406
Time                    17.32
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       368
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                    294.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127764 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

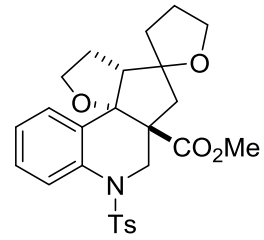




3ak, isomer 2

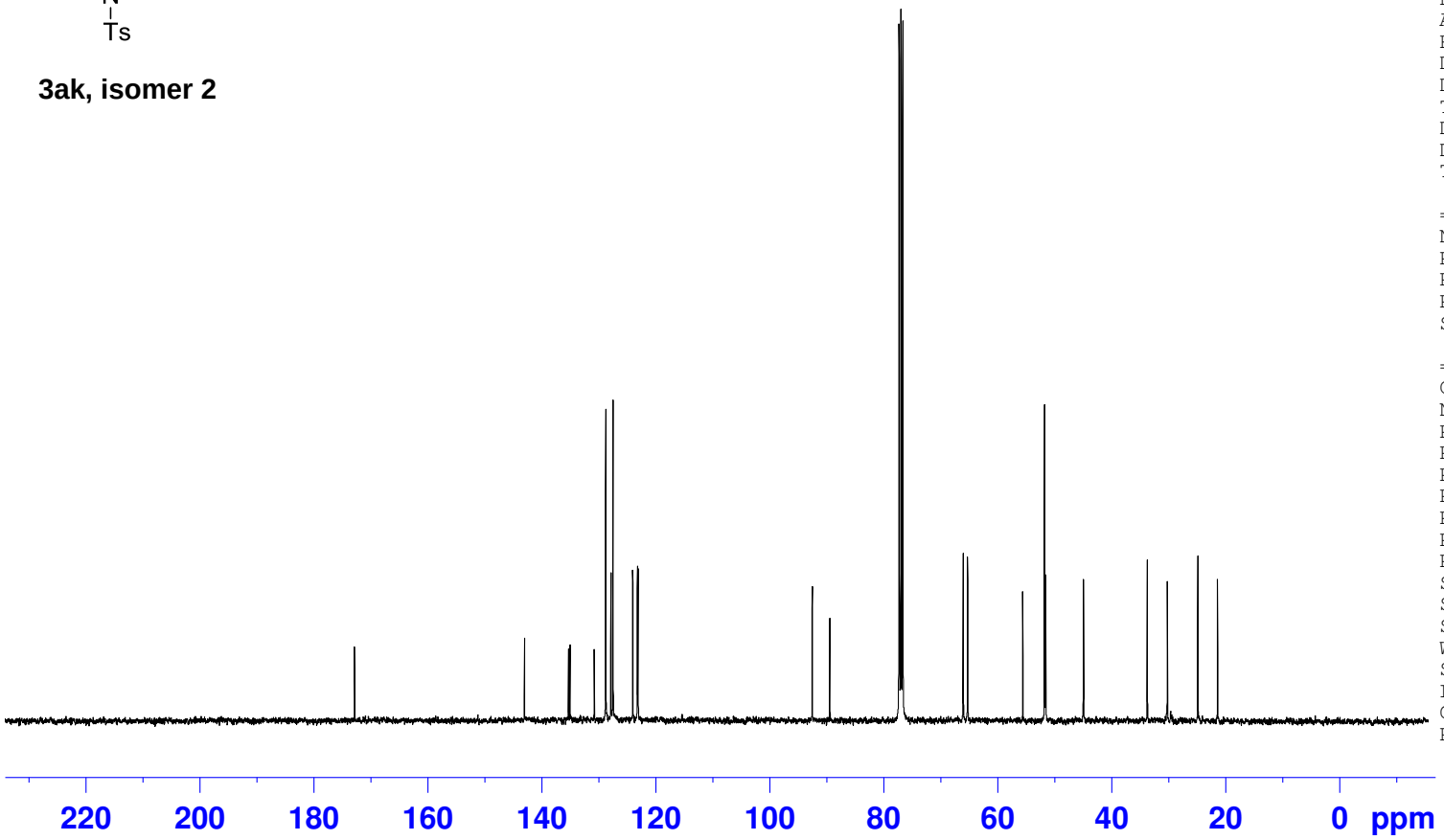
```
NAME      lly-880-3bp-20170406
EXPNO                        1
PROCNO                       1
Date_             20170406
Time                    17.56
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                 2.5625076 sec
RG                         181
DW                    78.200 usec
DE                     6.50 usec
TE                     293.9 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W              8.92857742 W
SFO1            400.1326008 MHz
SI                      32768
SF              400.1300005 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```



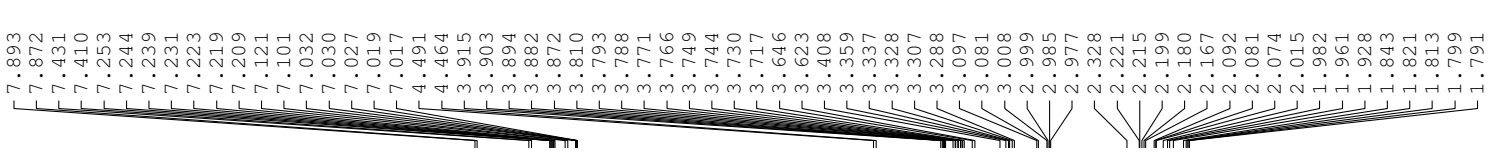


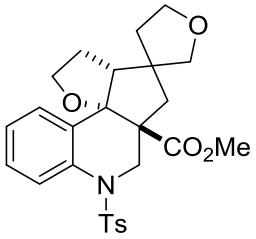
3ak, isomer 2

172.90
143.06
135.33
135.07
130.84
128.81
127.90
127.53
124.06
123.25
123.13
92.56
89.48
66.06
65.28
55.60
51.80
51.59
44.95
33.74
30.23
24.88
21.41



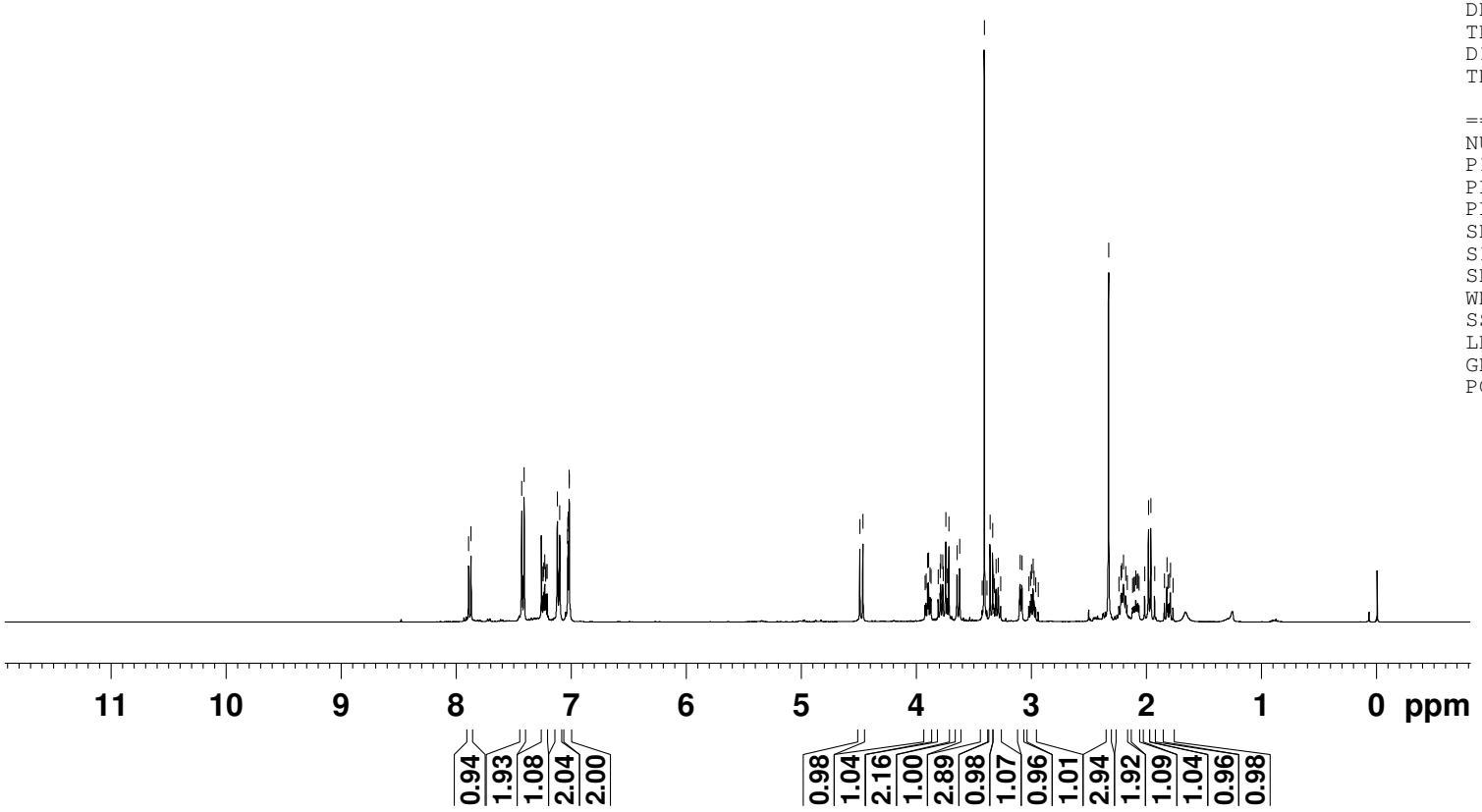

```
NAME      lly-880-3bp-20170406
EXPNO                        2
PROCNO                       1
Date_             20170406
Time                    18.00
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       800
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                     294.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127764 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

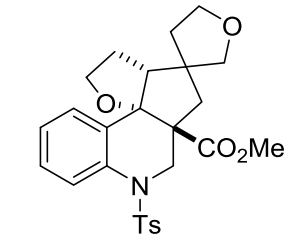




3al, isomer 1

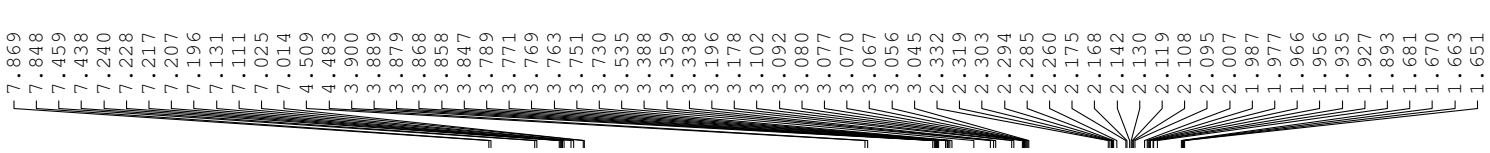
```
NAME      lly-905-2ap-20170606
EXPNO                        1
PROCNO                       1
Date_             20170606
Time                    16.14
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                         128
DW                    78.200 usec
DE                     6.50 usec
TE                     293.6 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      1.00
```

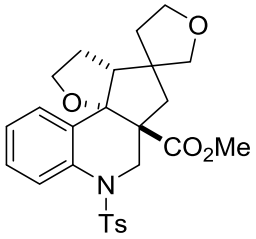




3al, isomer 1

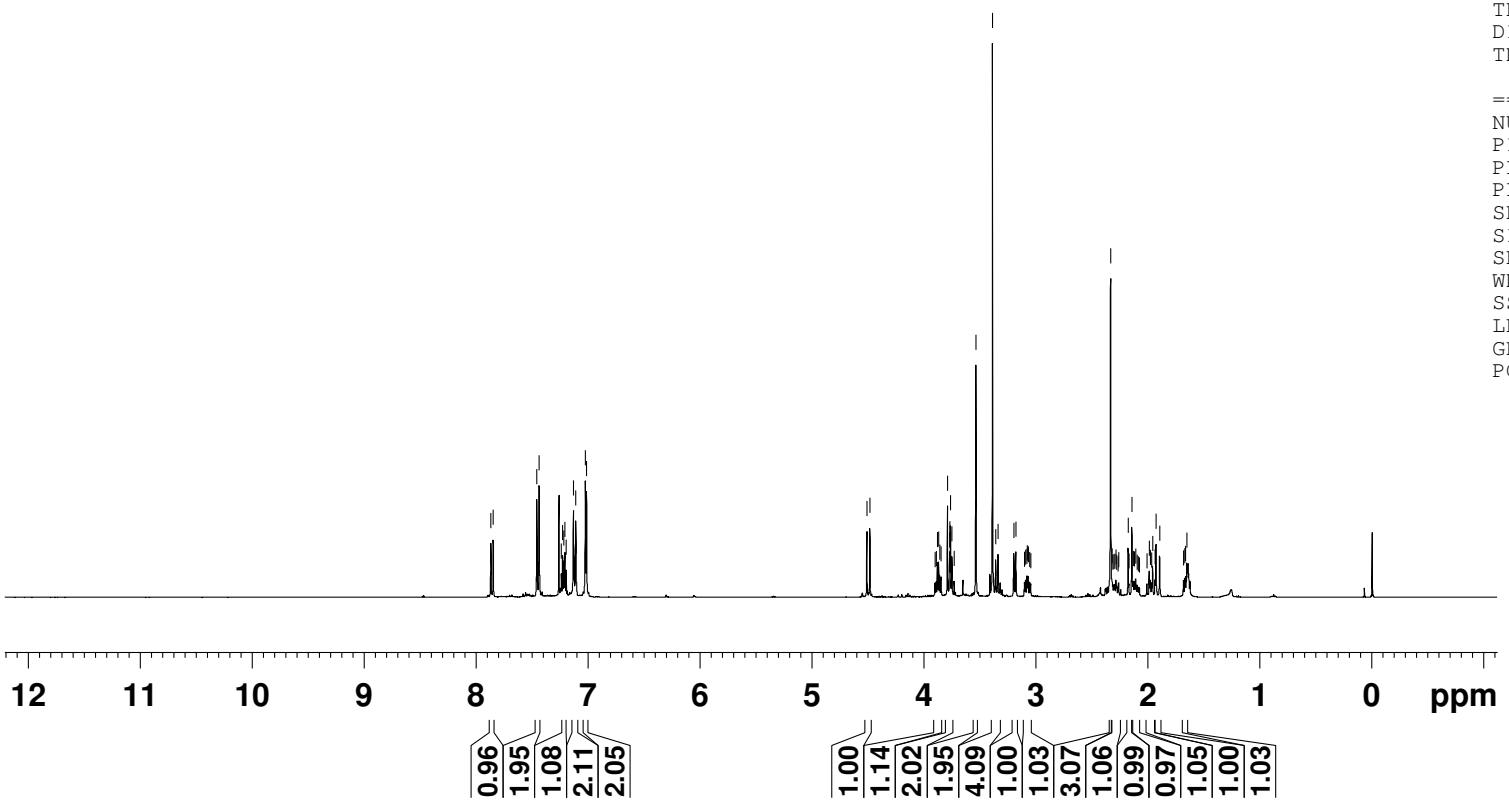

```
NAME      lly-905-2ap-20170606
EXPNO                        2
PROCNO                       1
Date_             20170606
Time                    16.19
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       200
DS                         4
SWH                25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                     294.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127743 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

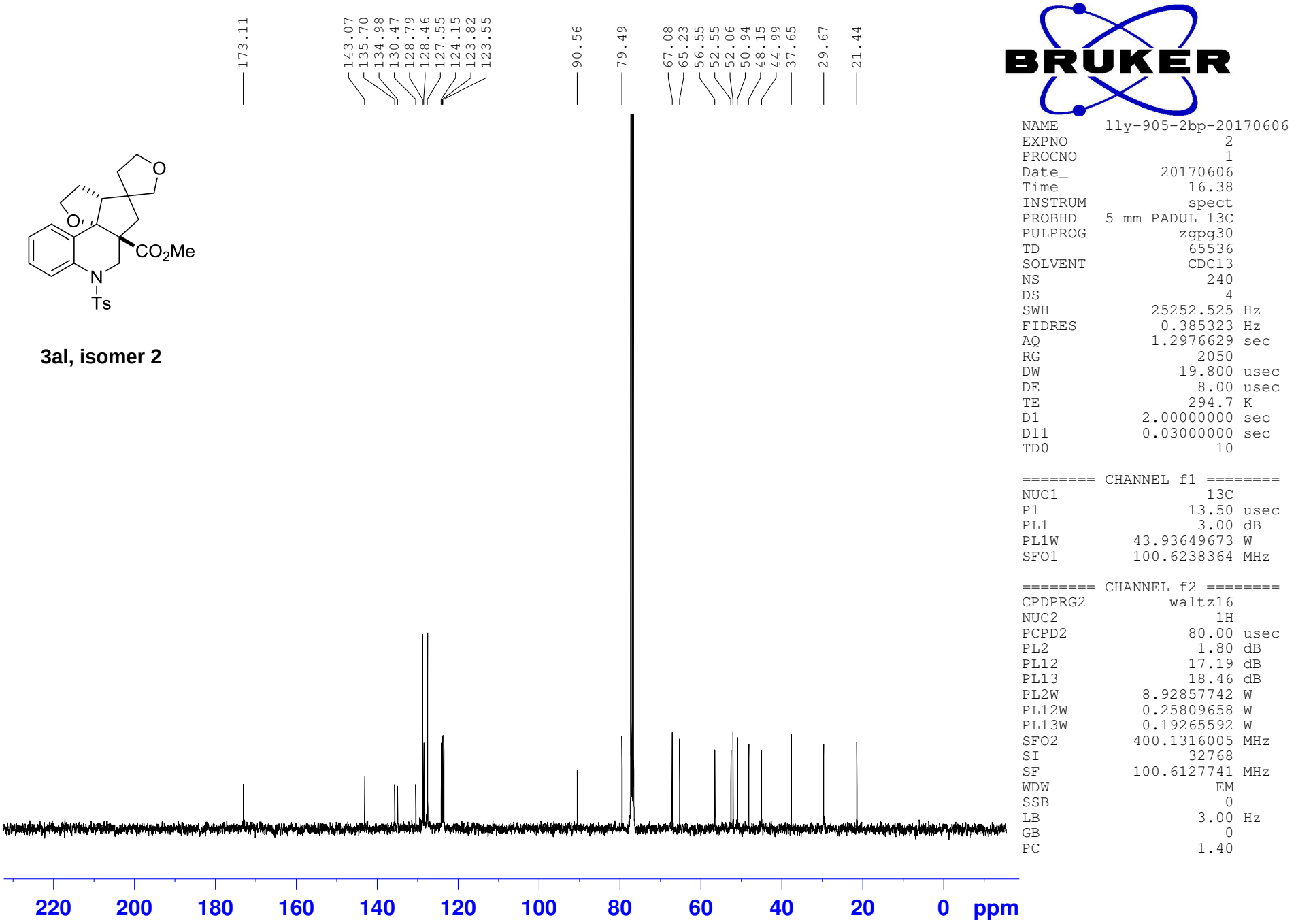




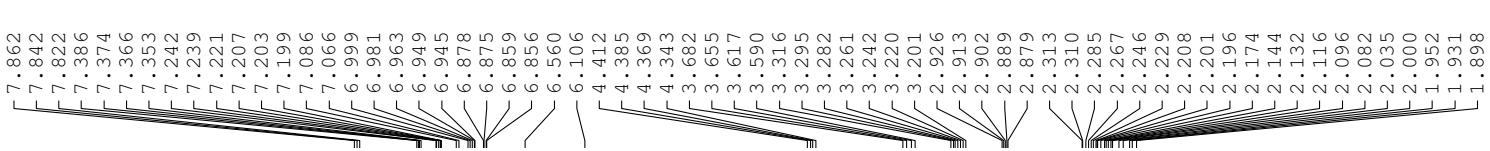
3al, isomer 2

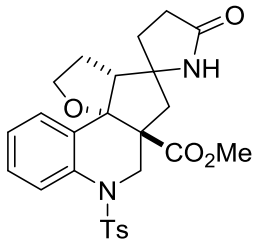
```
NAME      lly-905-2bp-20170606
EXPNO                        1
PROCNO                       1
Date_             20170606
Time                    16.32
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                         128
DW                    78.200 usec
DE                     6.50 usec
TE                     293.7 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                     1.00
```





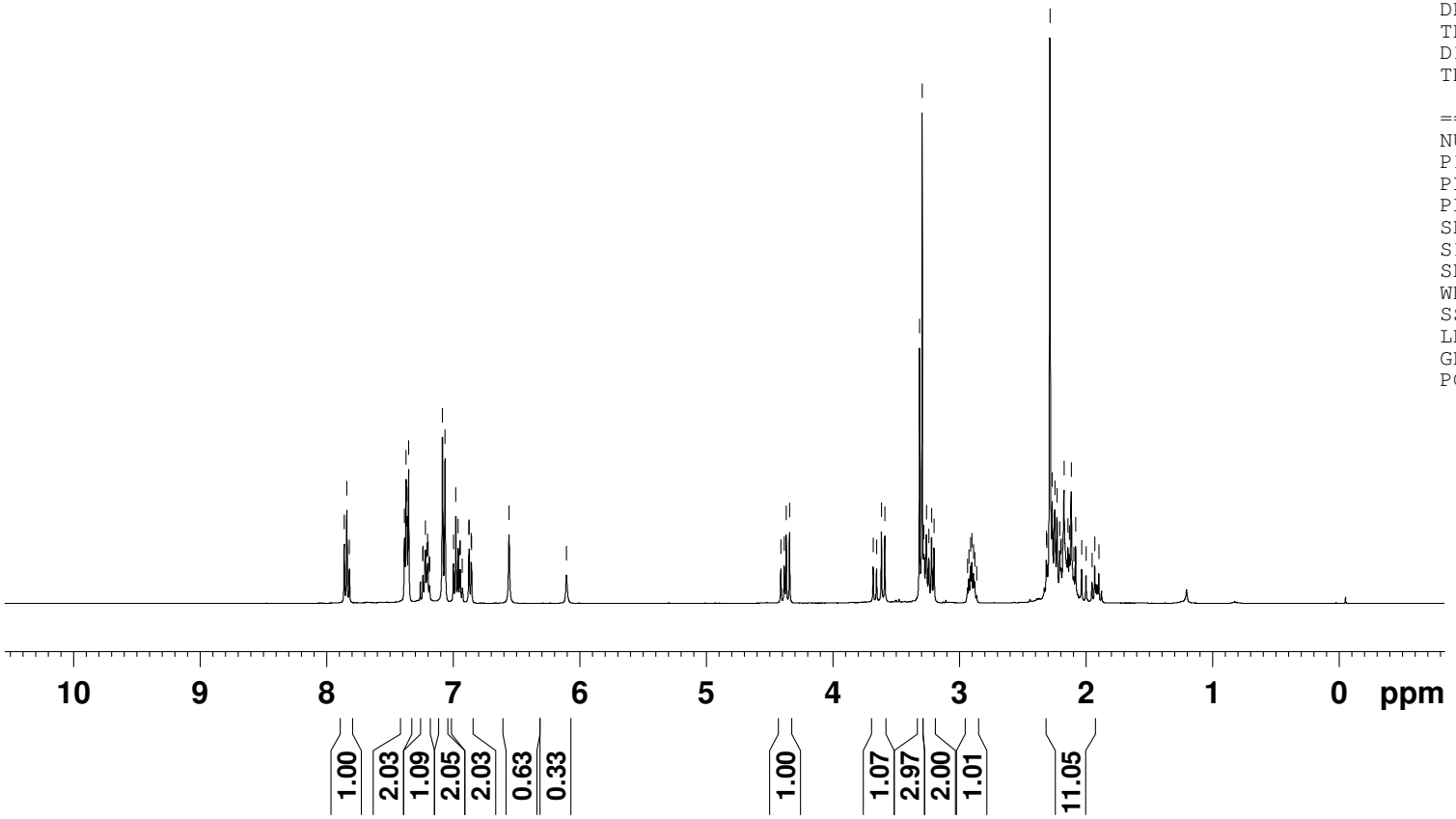

```
NAME      lly-905-2bp-20170606
EXPNO                        2
PROCNO                       1
Date_             20170606
Time                    16.38
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       240
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                    294.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127741 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

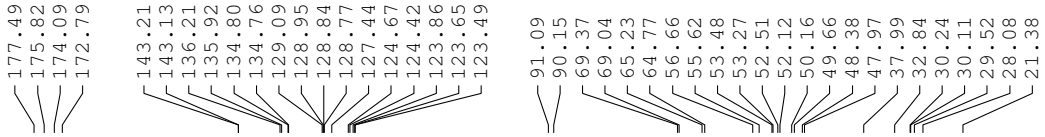


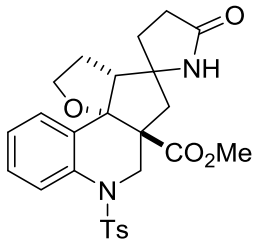


3am

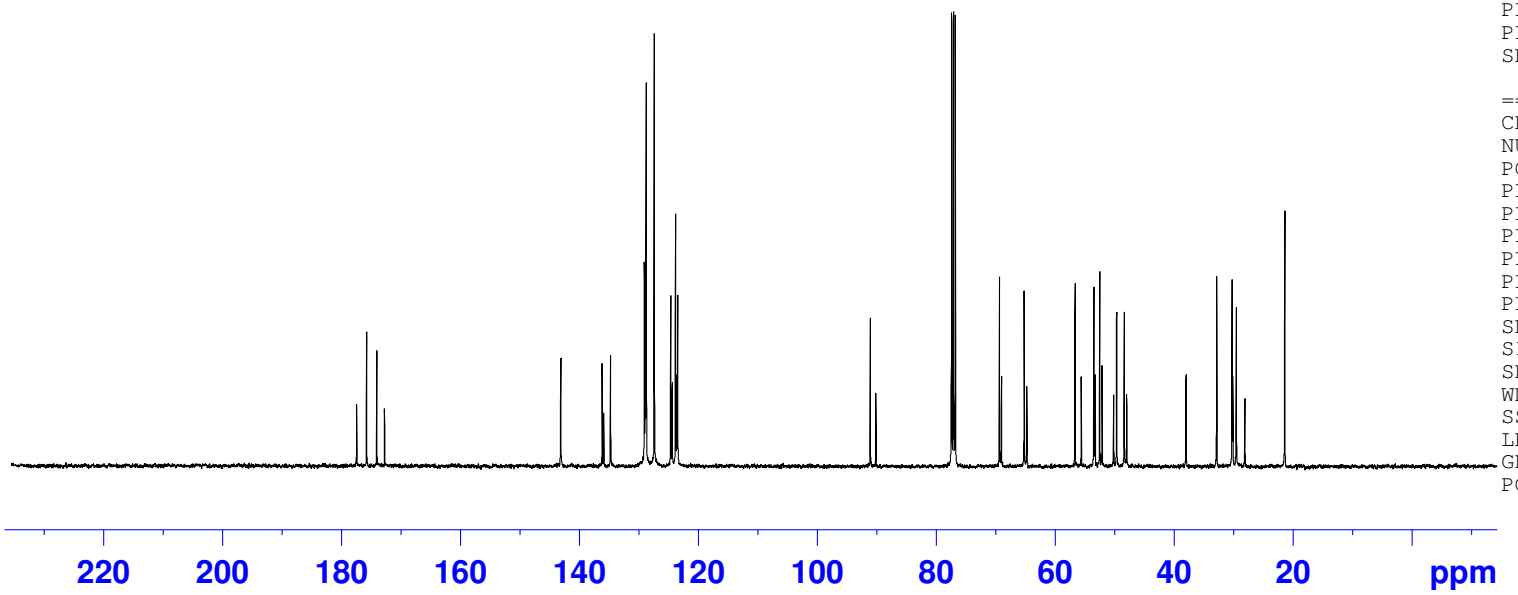
```
NAME      lly-900-1ap-20170519
EXPNO                        1
PROCNO                       1
Date_             20170521
Time                    22.21
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                 2.5625076 sec
RG                       45.2
DW                     78.200 usec
DE                      6.50 usec
TE                     295.1 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                        1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W               8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



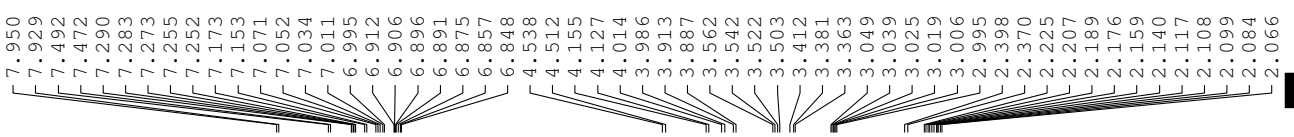


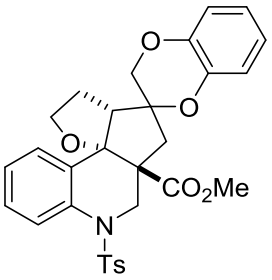


3am



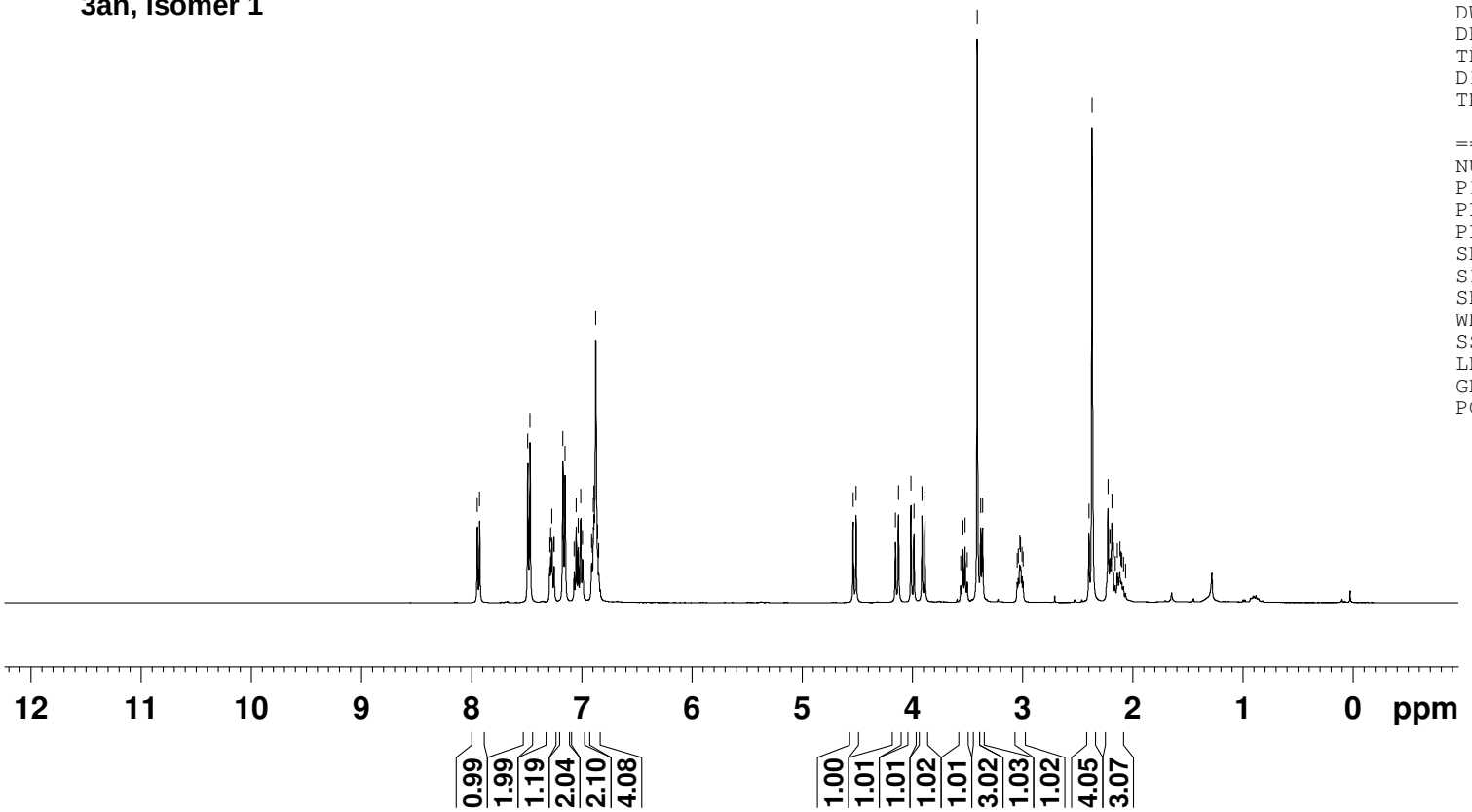

```
NAME      lly-900-1ap-20170519
EXPNO                        2
PROCNO                       1
Date_                 20170521
Time                     22.25
INSTRUM                  spect
PROBHD    5 mm PADUL 13C
PULPROG                zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         784
DS                           4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                 1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      296.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W              43.93649673 W
SFO1             100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W               8.92857742 W
PL12W              0.25809658 W
PL13W              0.19265592 W
SFO2             400.1316005 MHz
SI                       32768
SF               100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

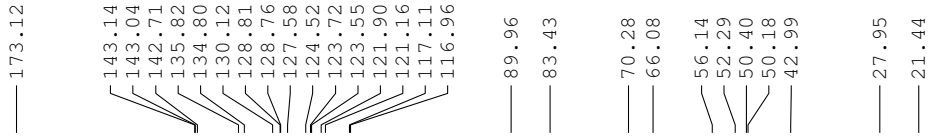


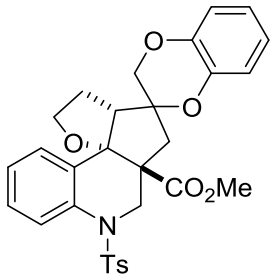


3an, isomer 1

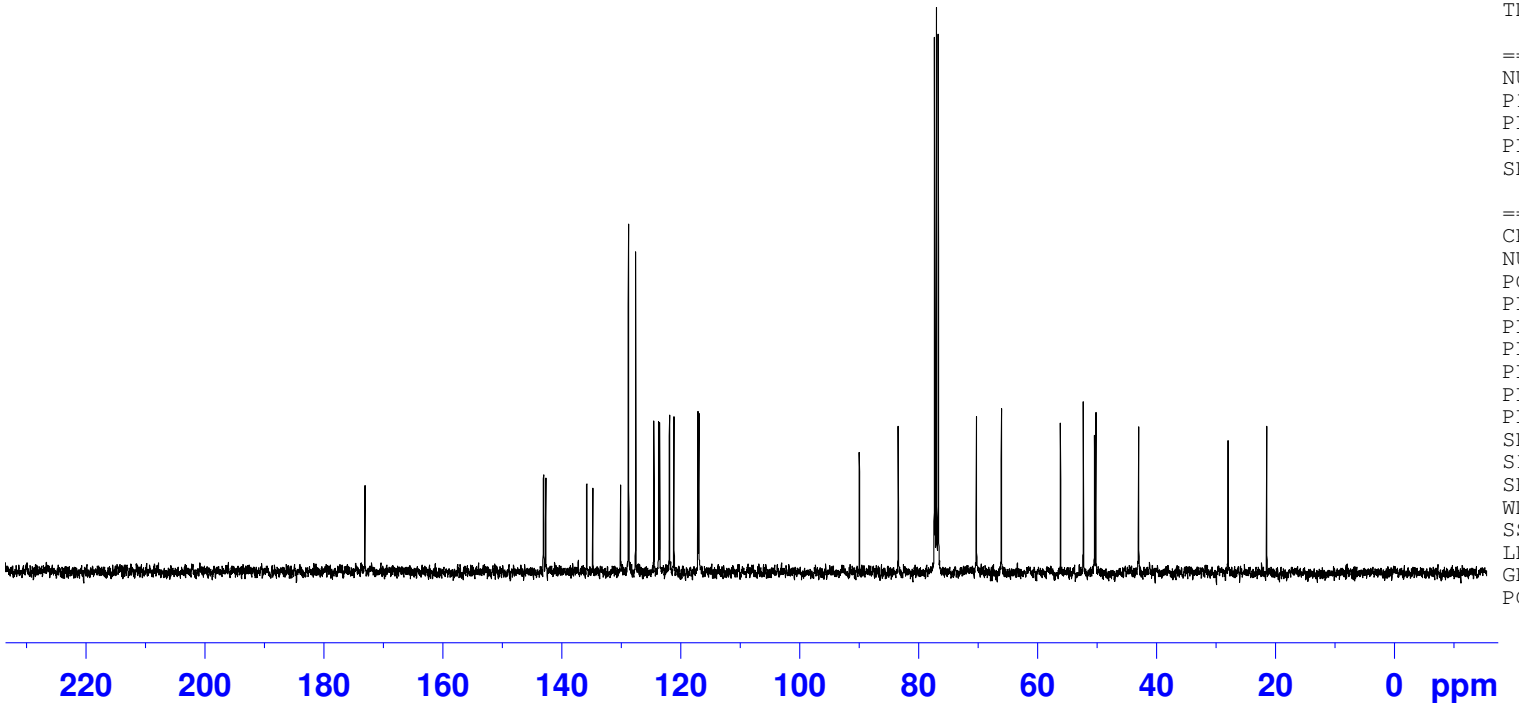
```
NAME      lly-879-3ap-20170331
EXPNO                        1
PROCNO                       1
Date_             20170331
Time                    22.34
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                 2.5625076 sec
RG                        144
DW                     78.200 usec
DE                      6.50 usec
TE                     293.2 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W              8.92857742 W
SFO1            400.1326008 MHz
SI                      32768
SF              400.1300005 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



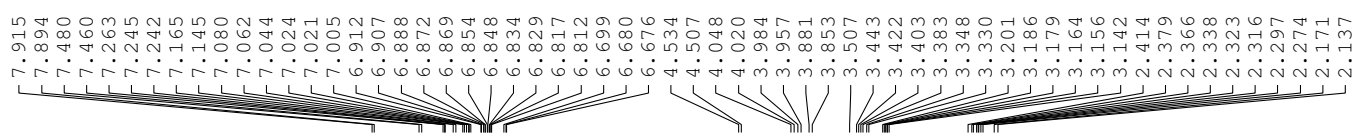


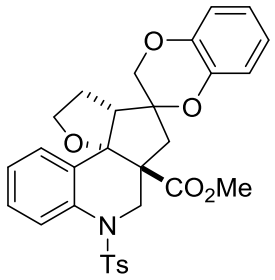


3an, isomer 1



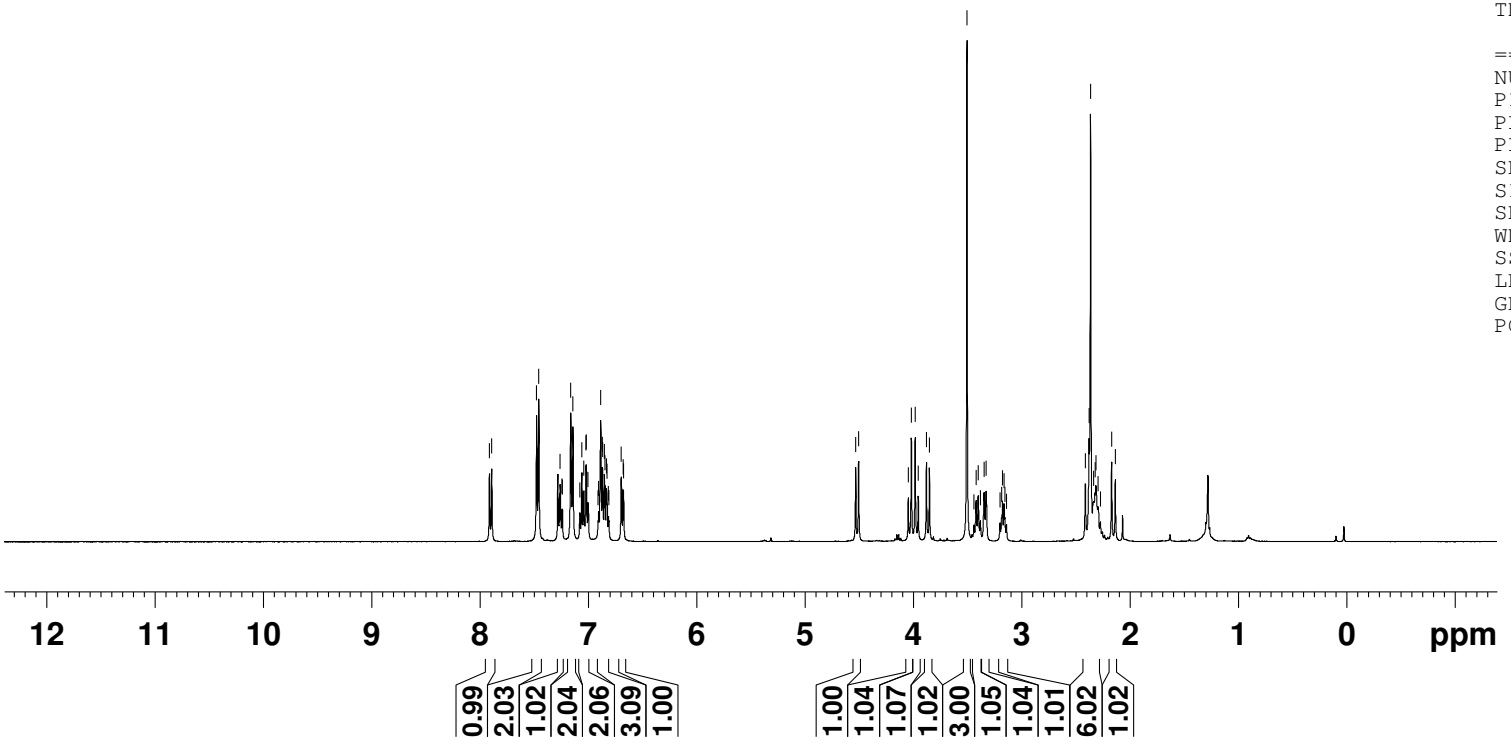

```
NAME      lly-879-3ap-20170331
EXPNO                        2
PROCNO                       1
Date_                 20170331
Time                     22.37
INSTRUM                  spect
PROBHD    5 mm PADUL 13C
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        112
DS                          4
SWH                 25252.525 Hz
FIDRES               0.385323 Hz
AQ                  1.2976629 sec
RG                       2050
DW                     19.800 usec
DE                      8.00 usec
TE                     293.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W               43.93649673 W
SFO1              100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2              400.1316005 MHz
SI                       32768
SF                100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

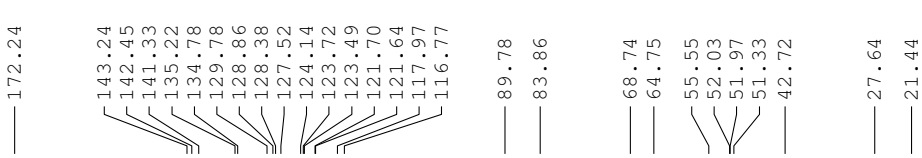


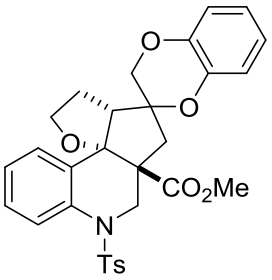


3an, isomer 2

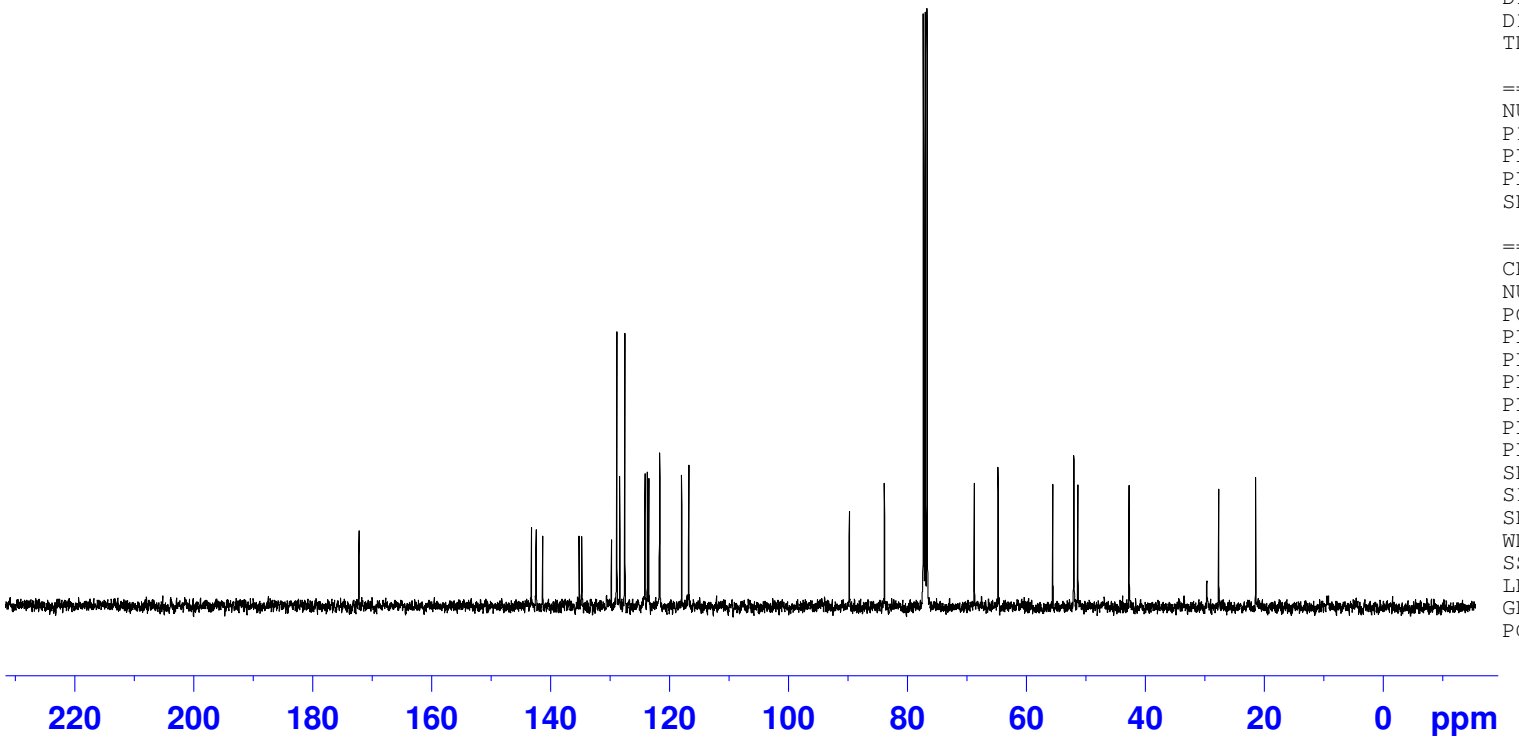
```
NAME        lly-879-3bp-20170331
EXPNO                          1
PROCNO                         1
Date_               20170331
Time                      22.47
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                            8
DS                            0
SWH                    6393.862 Hz
FIDRES                 0.195125 Hz
AQ                     2.5625076 sec
RG                          144
DW                       78.200 usec
DE                        6.50 usec
TE                       293.1 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                         1H
P1                       13.10 usec
PL1                       1.80 dB
PL1W                8.92857742 W
SFO1               400.1326008 MHz
SI                        32768
SF                 400.1300005 MHz
WDW                          EM
SSB                           0
LB                        0.30 Hz
GB                            0
PC                        1.00
```



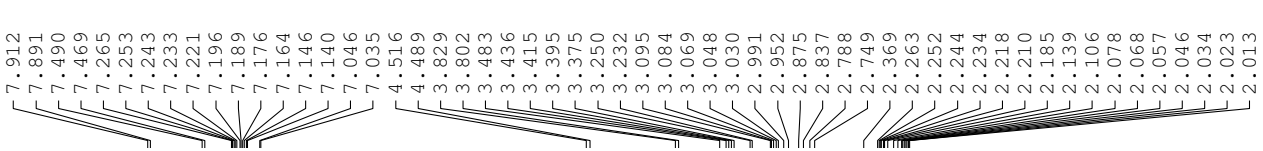


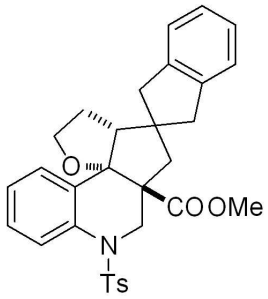


3an, isomer 2



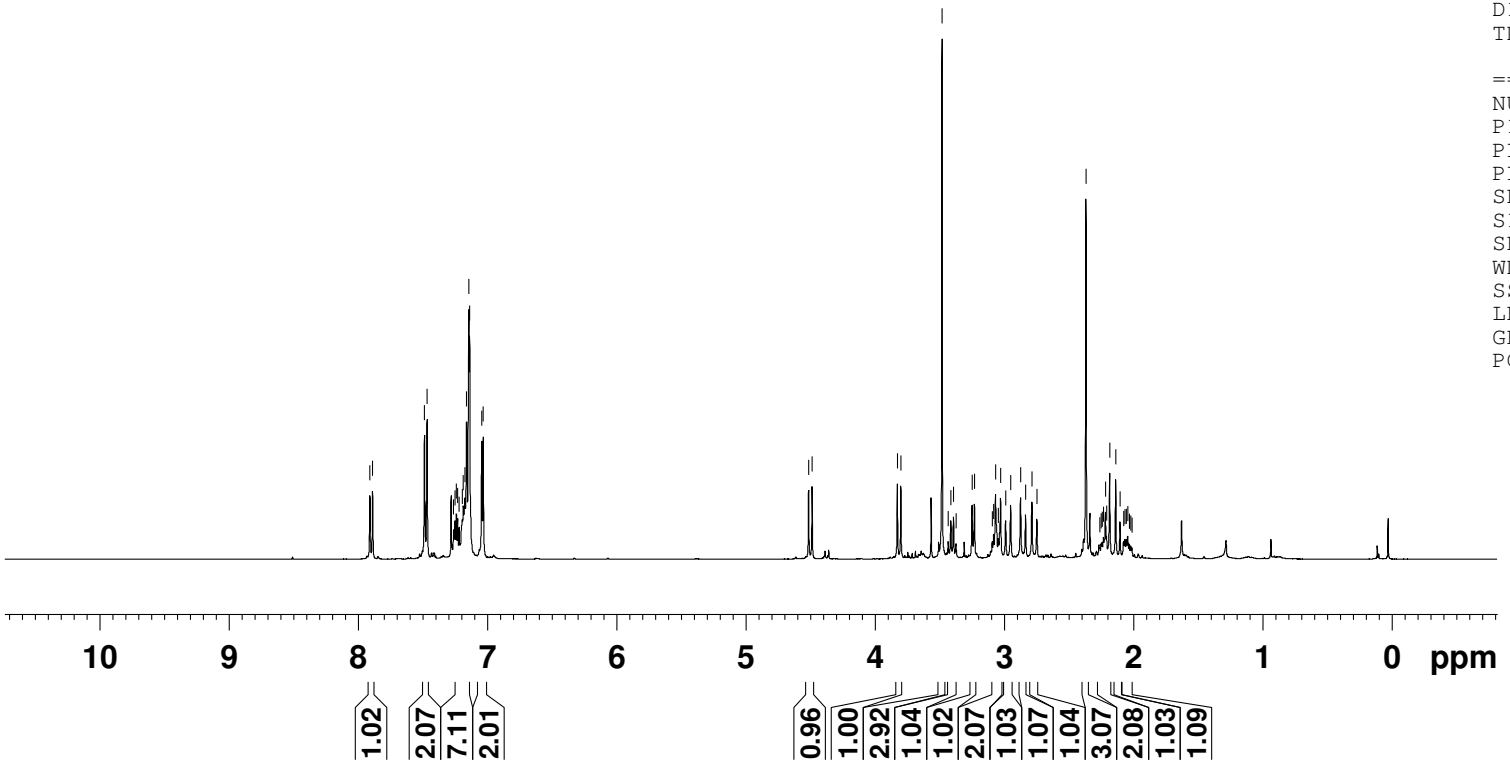

```
NAME      lly-879-3bp-20170331
EXPNO                        2
PROCNO                       1
Date_                 20170331
Time                     22.50
INSTRUM                  spect
PROBHD    5 mm PADUL 13C
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        160
DS                          4
SWH                 25252.525 Hz
FIDRES               0.385323 Hz
AQ                  1.2976629 sec
RG                       2050
DW                     19.800 usec
DE                      8.00 usec
TE                     293.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W              43.93649673 W
SFO1             100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W               8.92857742 W
PL12W              0.25809658 W
PL13W              0.19265592 W
SFO2             400.1316005 MHz
SI                       32768
SF               100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

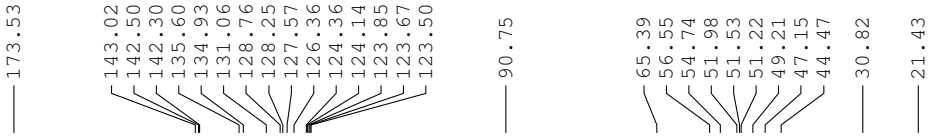


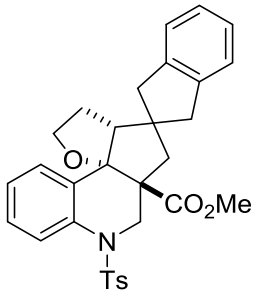


3ao

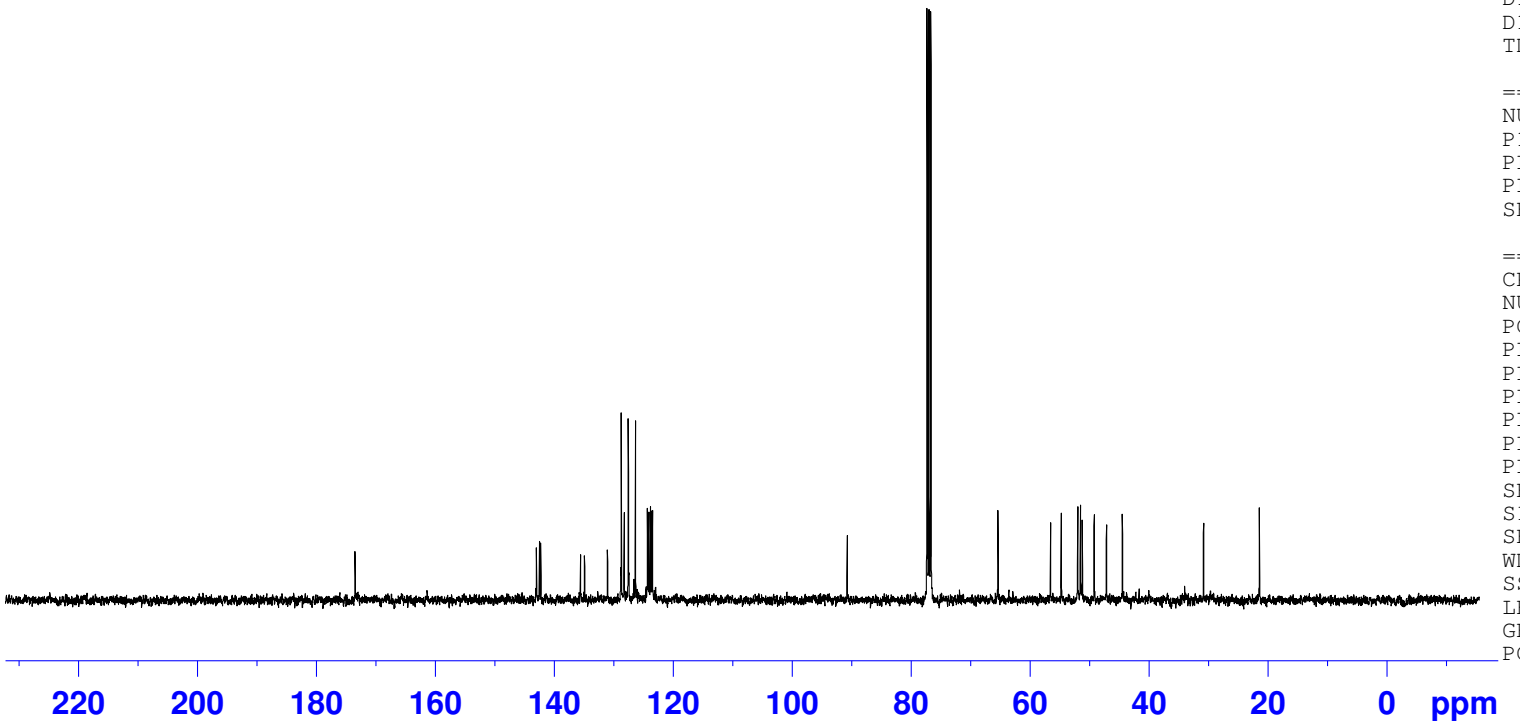
```
NAME      lly-880-1p-20170405
EXPNO                       1
PROCNO                      1
Date_              20170405
Time                   19.20
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       128
DW                    78.200 usec
DE                     6.50 usec
TE                    293.8 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300005 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.00
```



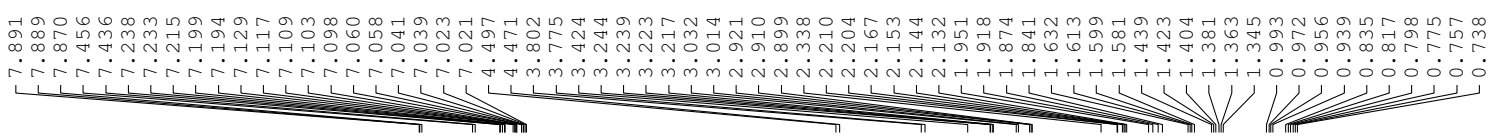


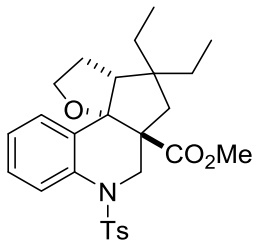


3ao



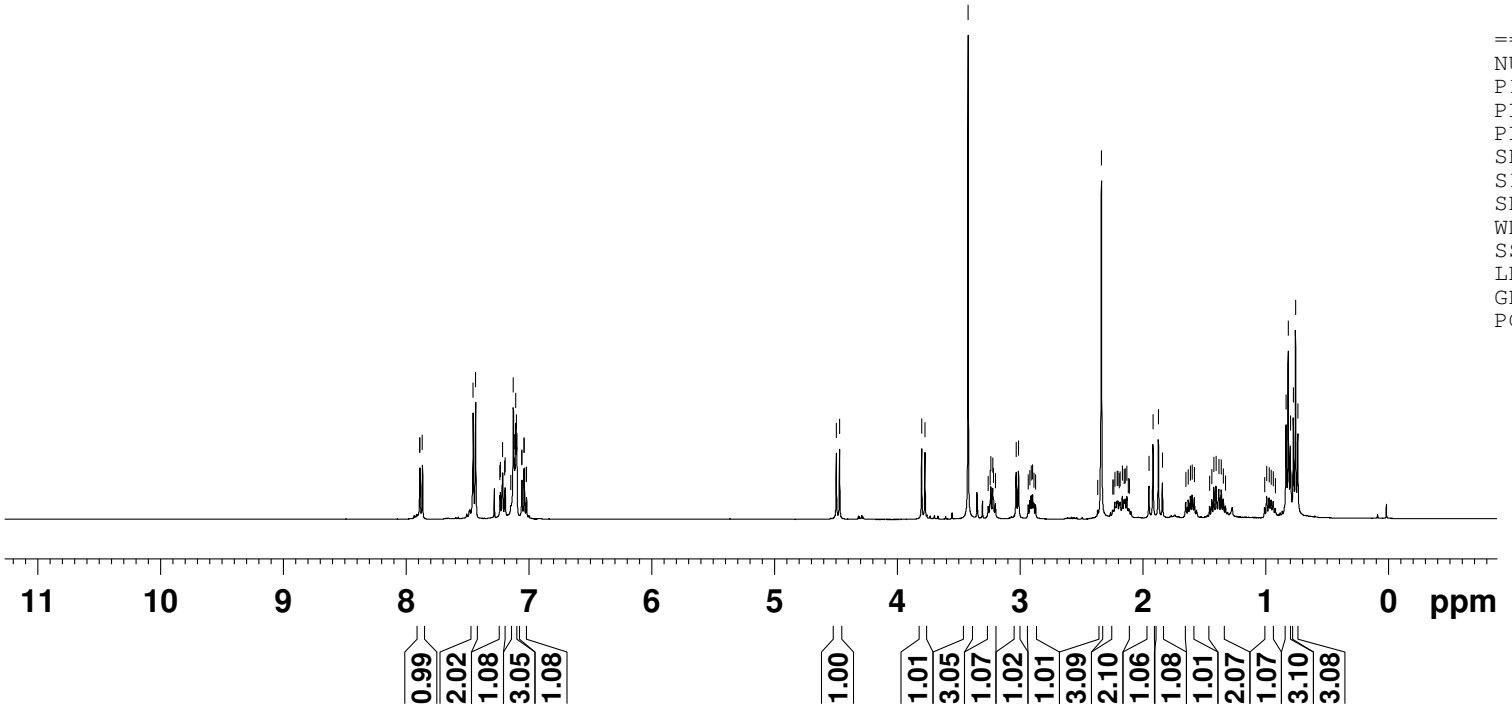

```
NAME        lly-880-1p-20170405
EXPNO                         2
PROCNO                        1
Date_                  20170405
Time                      19.24
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         160
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      294.8 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

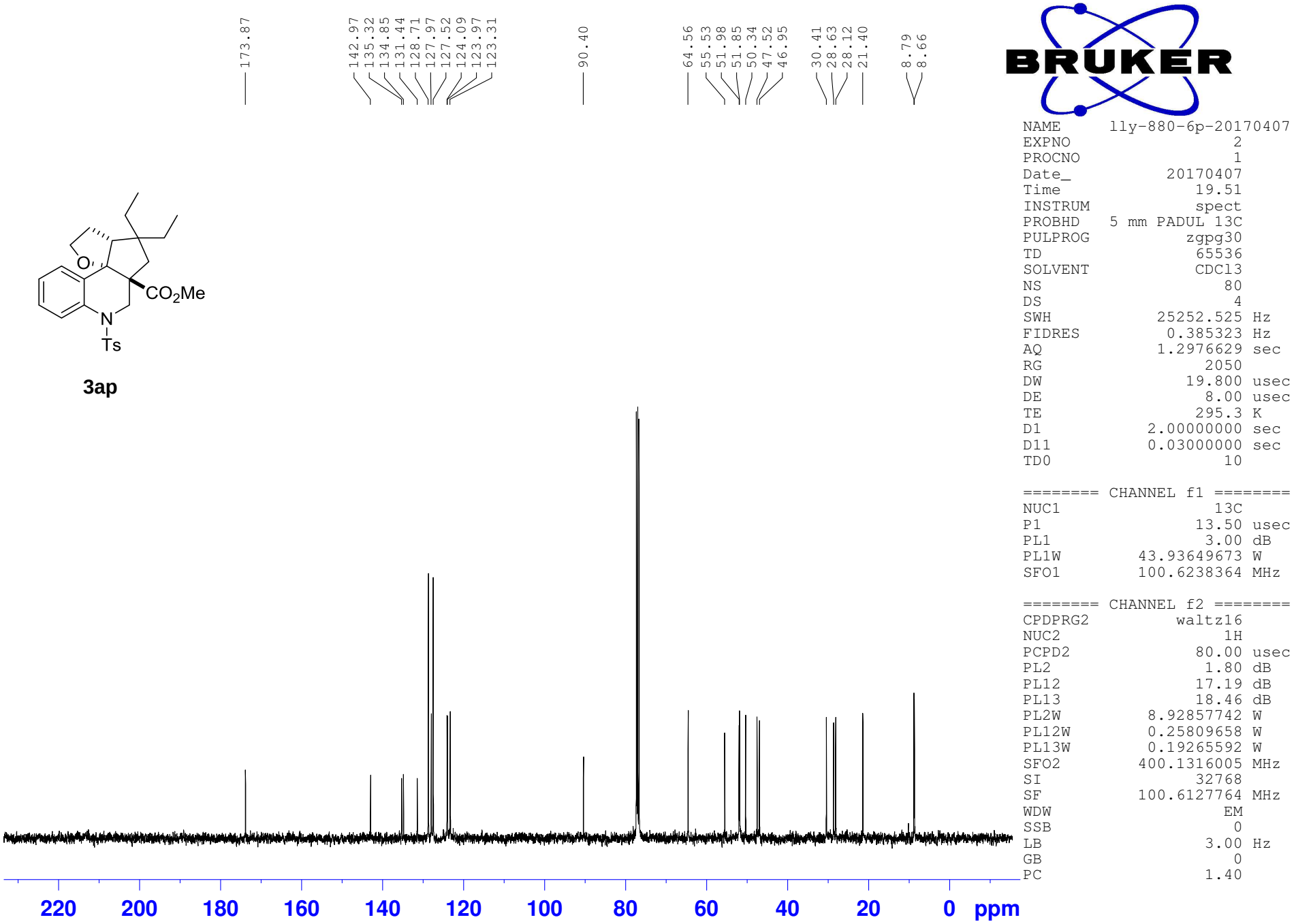




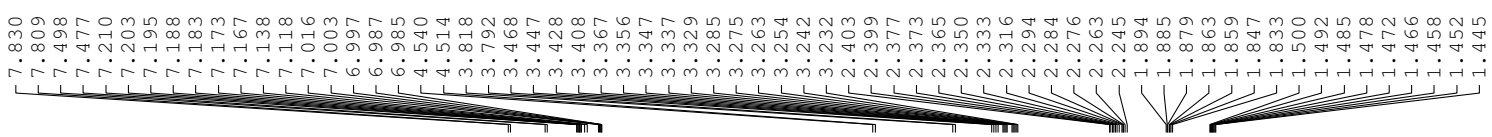
3ap

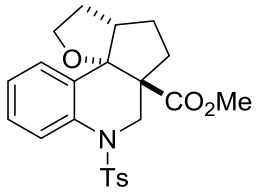
```
NAME      lly-880-6p-20170407
EXPNO                       1
PROCNO                      1
Date_              20170407
Time                   19.46
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                      71.8
DW                    78.200 usec
DE                     6.50 usec
TE                    294.2 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```





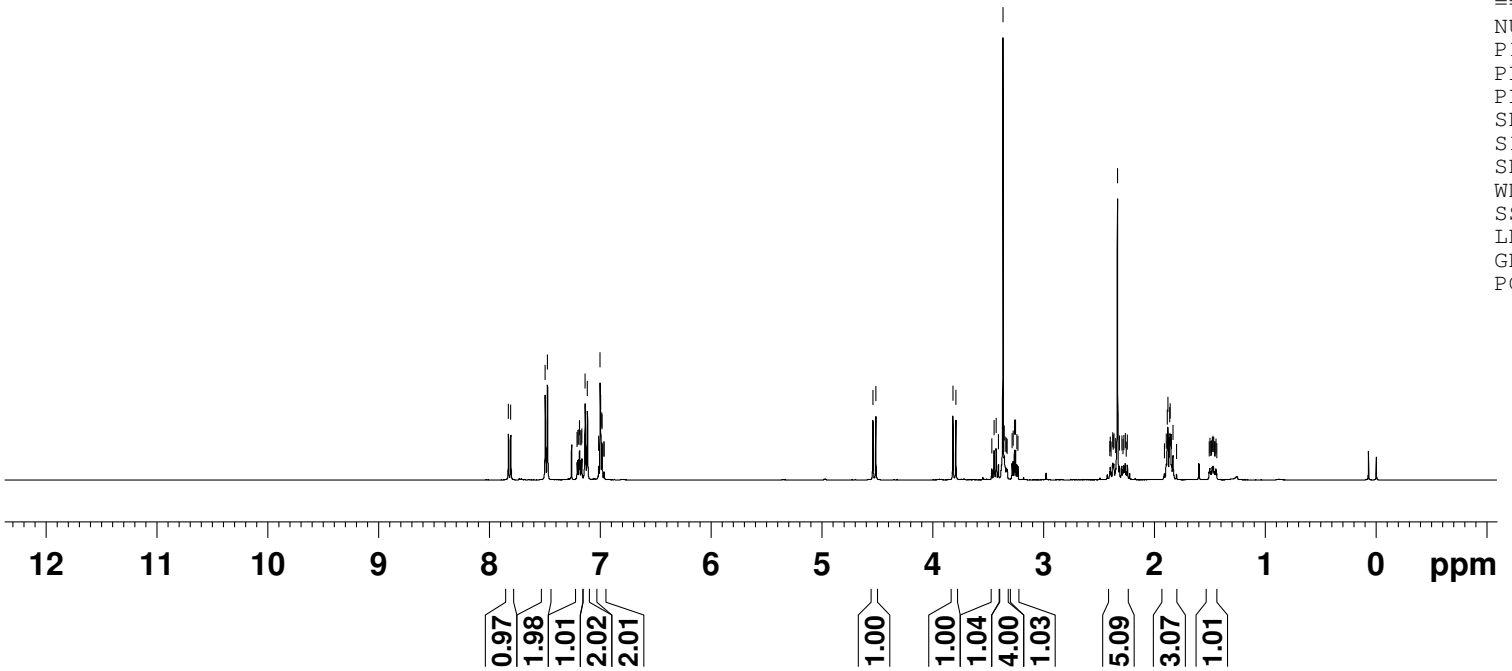
NAME	lly-880-6p-20170407
EXPNO	2
PROCNO	1
Date_	20170407
Time	19.51
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	80
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	8.00 usec
TE	295.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	13.50 usec
PL1	3.00 dB
PL1W	43.93649673 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.80 dB
PL12	17.19 dB
PL13	18.46 dB
PL2W	8.92857742 W
PL12W	0.25809658 W
PL13W	0.19265592 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127764 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

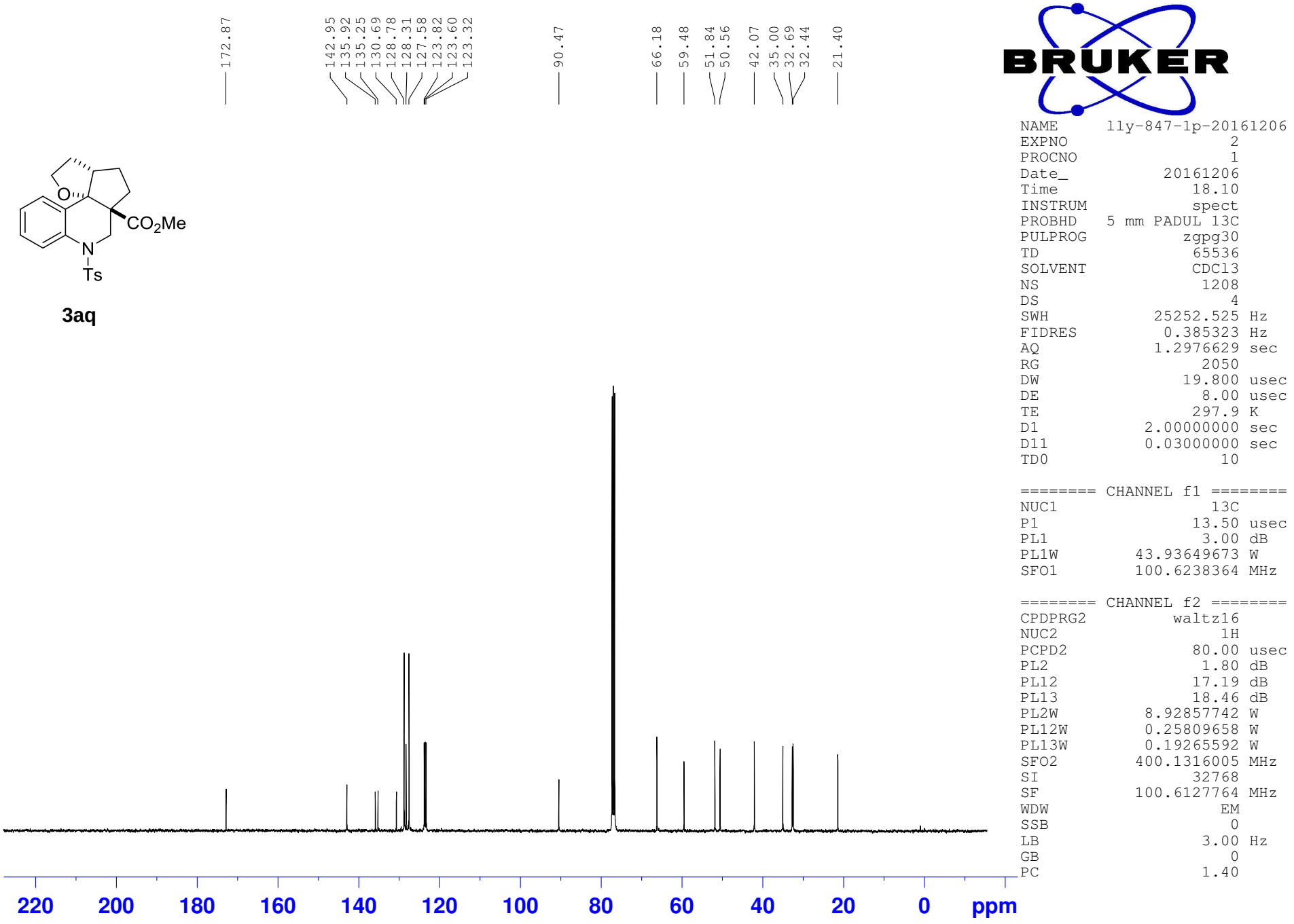




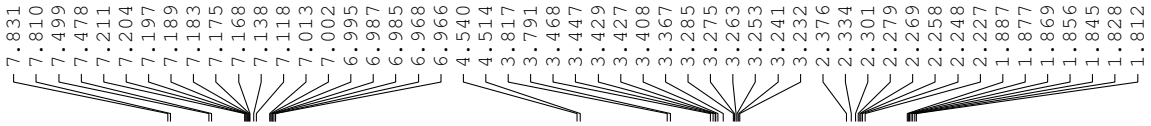
3aq

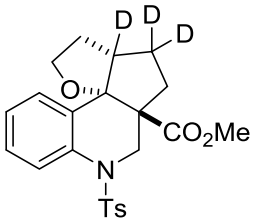
```
NAME      lly-847-1p-20161206
EXPNO                       1
PROCNO                      1
Date_                20161206
Time                    16.31
INSTRUM                 spect
PROBHD   5 mm PADUL 13C
PULPROG                 zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                   2.5625076 sec
RG                        161
DW                     78.200 usec
DE                      6.50 usec
TE                     296.3 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W               8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```



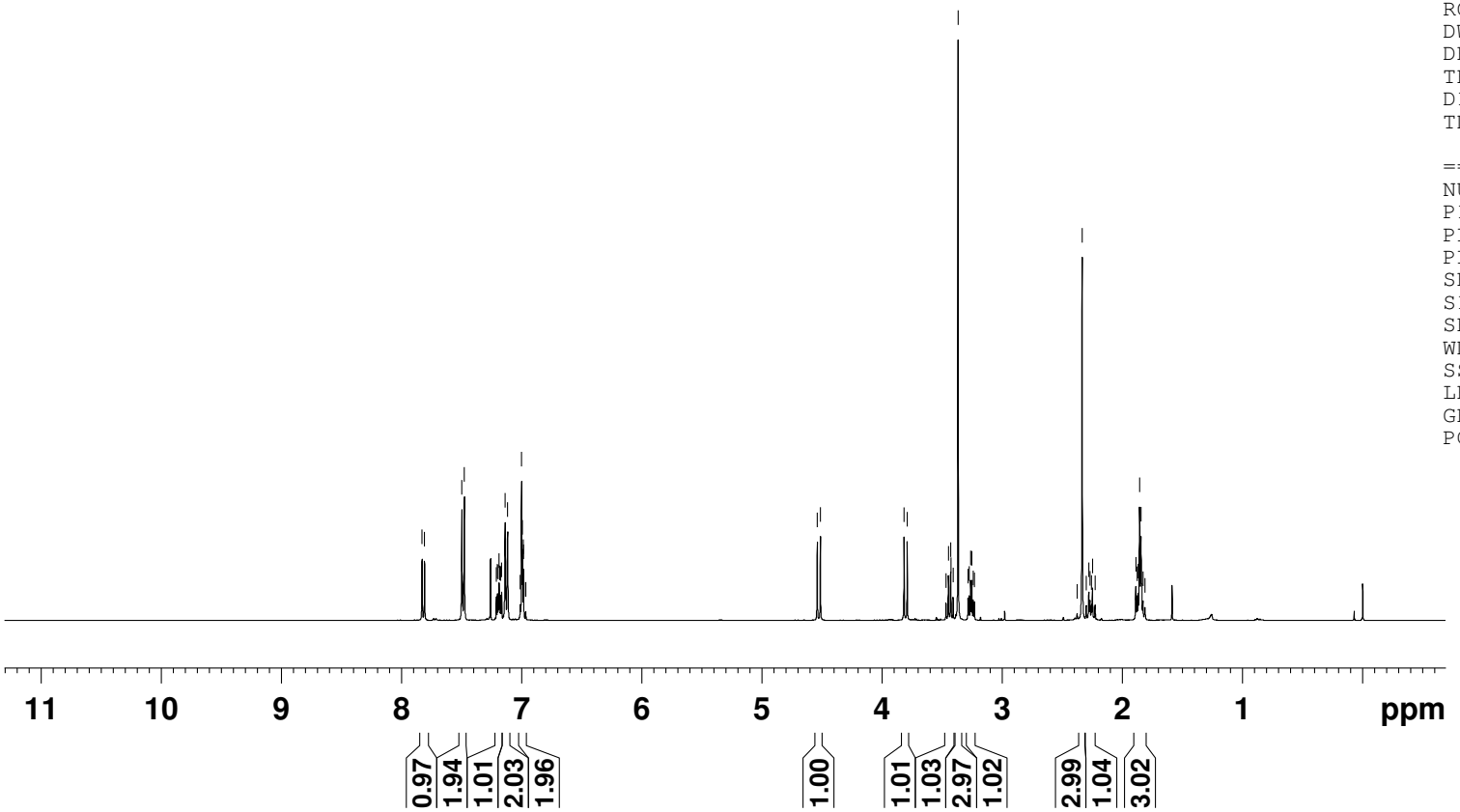



```
NAME      lly-847-1p-20161206
EXPNO                       2
PROCNO                      1
Date_              20161206
Time                   18.10
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1208
DS                          4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                       2050
DW                     19.800 usec
DE                      8.00 usec
TE                     297.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        10

======== CHANNEL f1 ========
NUC1                      13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                       32768
SF              100.6127764 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

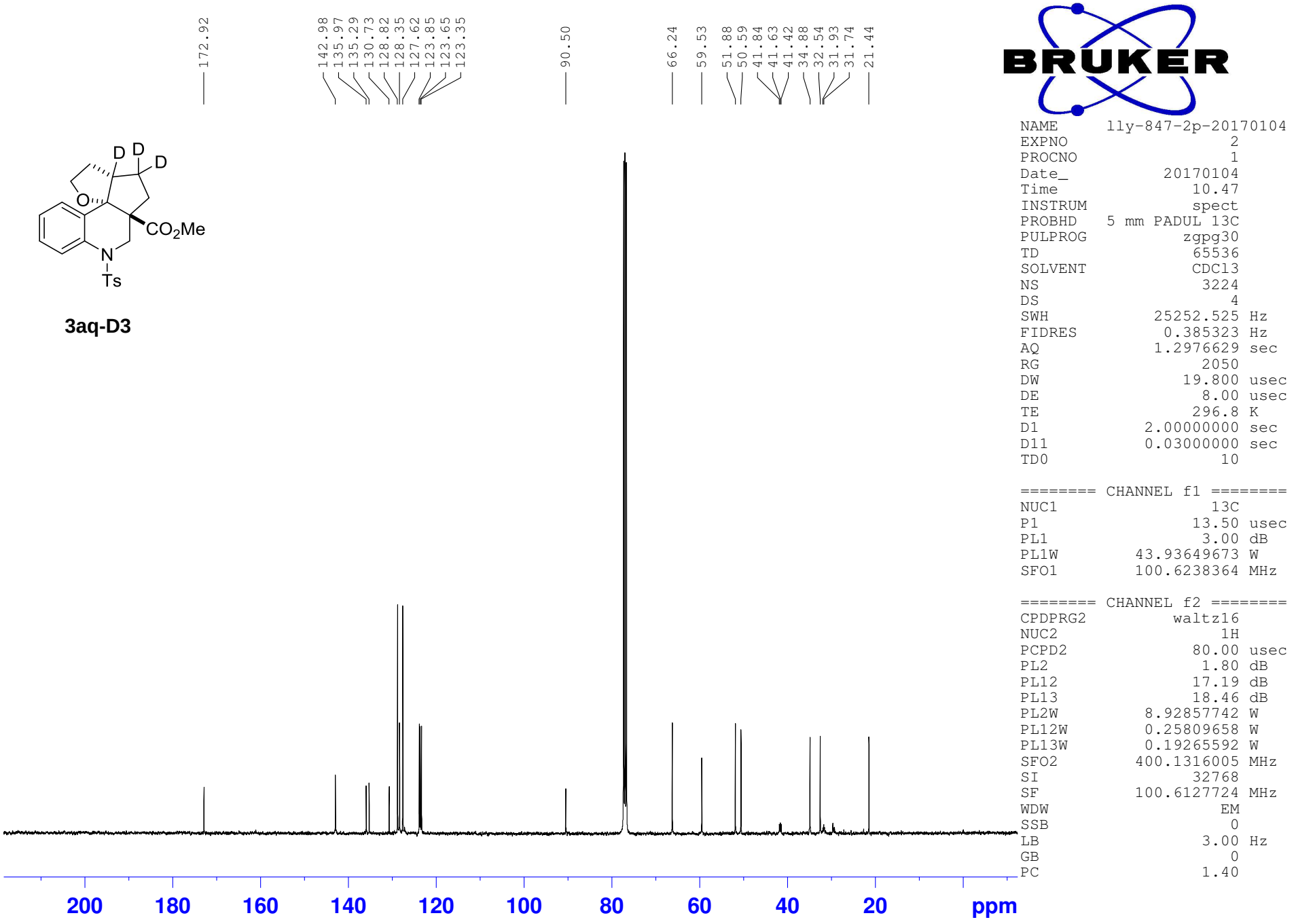




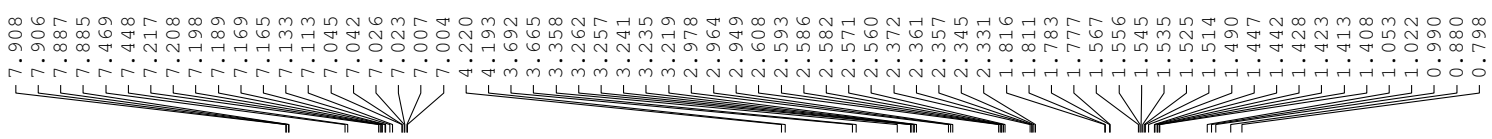
3aq-D3

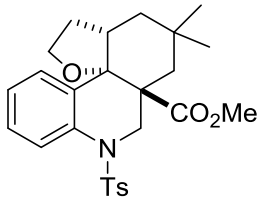



```
NAME      lly-847-2p-20161206
EXPNO                       1
PROCNO                      1
Date_              20161206
Time                   16.36
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       161
DW                    78.200 usec
DE                     6.50 usec
TE                    296.4 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



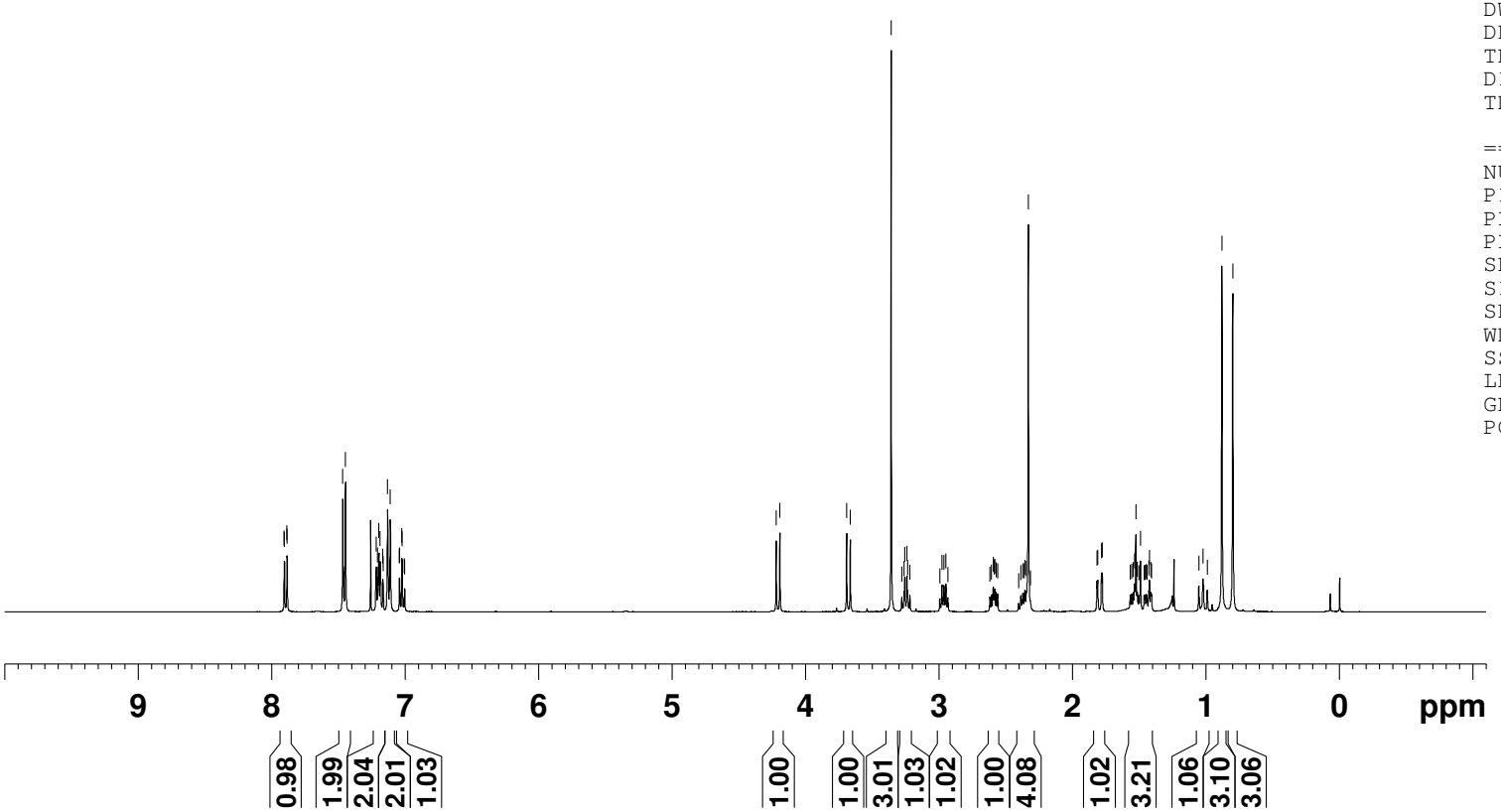

```
NAME      lly-847-2p-20170104
EXPNO                       2
PROCNO                      1
Date_            20170104
Time                   10.47
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                     65536
SOLVENT              CDCl3
NS                      3224
DS                         4
SWH              25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                      2050
DW                   19.800 usec
DE                    8.00 usec
TE                   296.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                    13.50 usec
PL1                    3.00 dB
PL1W          43.93649673 W
SFO1          100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                   1.80 dB
PL12                 17.19 dB
PL13                 18.46 dB
PL2W           8.92857742 W
PL12W          0.25809658 W
PL13W          0.19265592 W
SFO2          400.1316005 MHz
SI                    32768
SF            100.6127724 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40
```

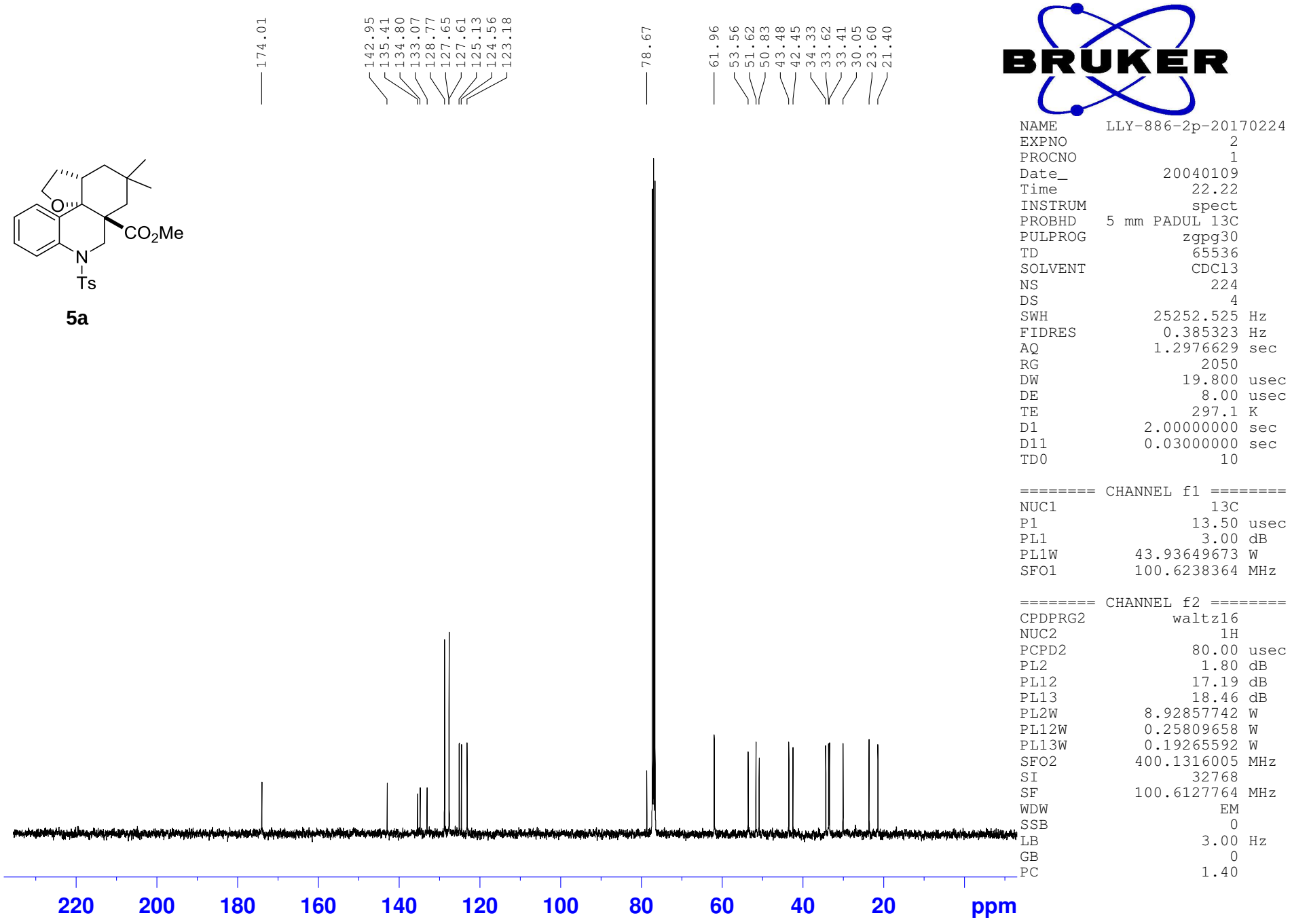




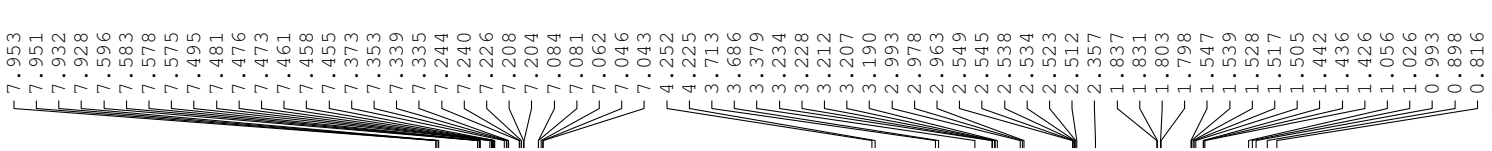
5a

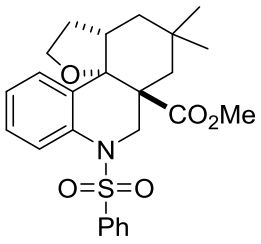
```
NAME      LLY-886-2p-20170224
EXPNO                       1
PROCNO                      1
Date_            20040109
Time                  22.19
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        0
SWH               6393.862 Hz
FIDRES           0.195125 Hz
AQ               2.5625076 sec
RG                      128
DW                  78.200 usec
DE                   6.50 usec
TE                  296.3 K
D1             1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   13.10 usec
PL1                   1.80 dB
PL1W          8.92857742 W
SFO1         400.1326008 MHz
SI                    32768
SF           400.1300096 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                   1.00
```





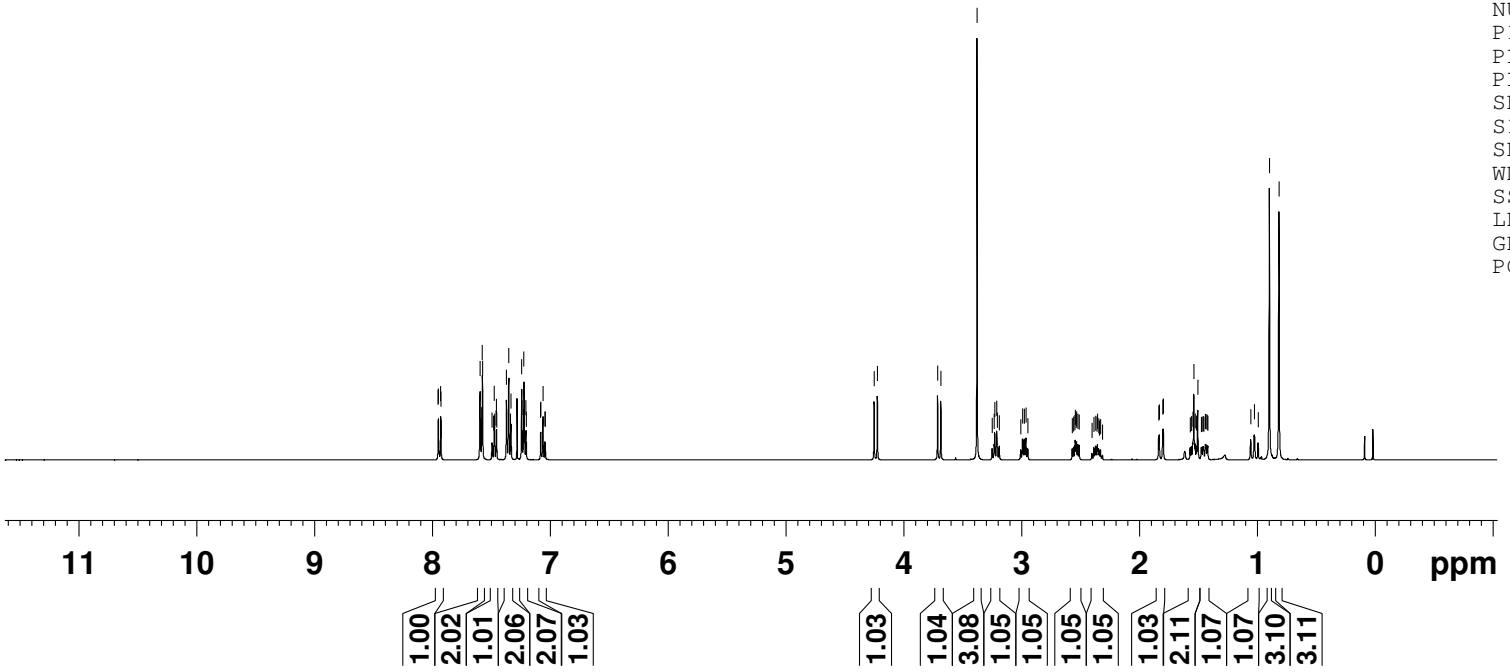

```
NAME      LLY-886-2p-20170224
EXPNO                       2
PROCNO                      1
Date_                20040109
Time                    22.22
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         224
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      297.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                       3.00 Hz
GB                           0
PC                        1.40
```

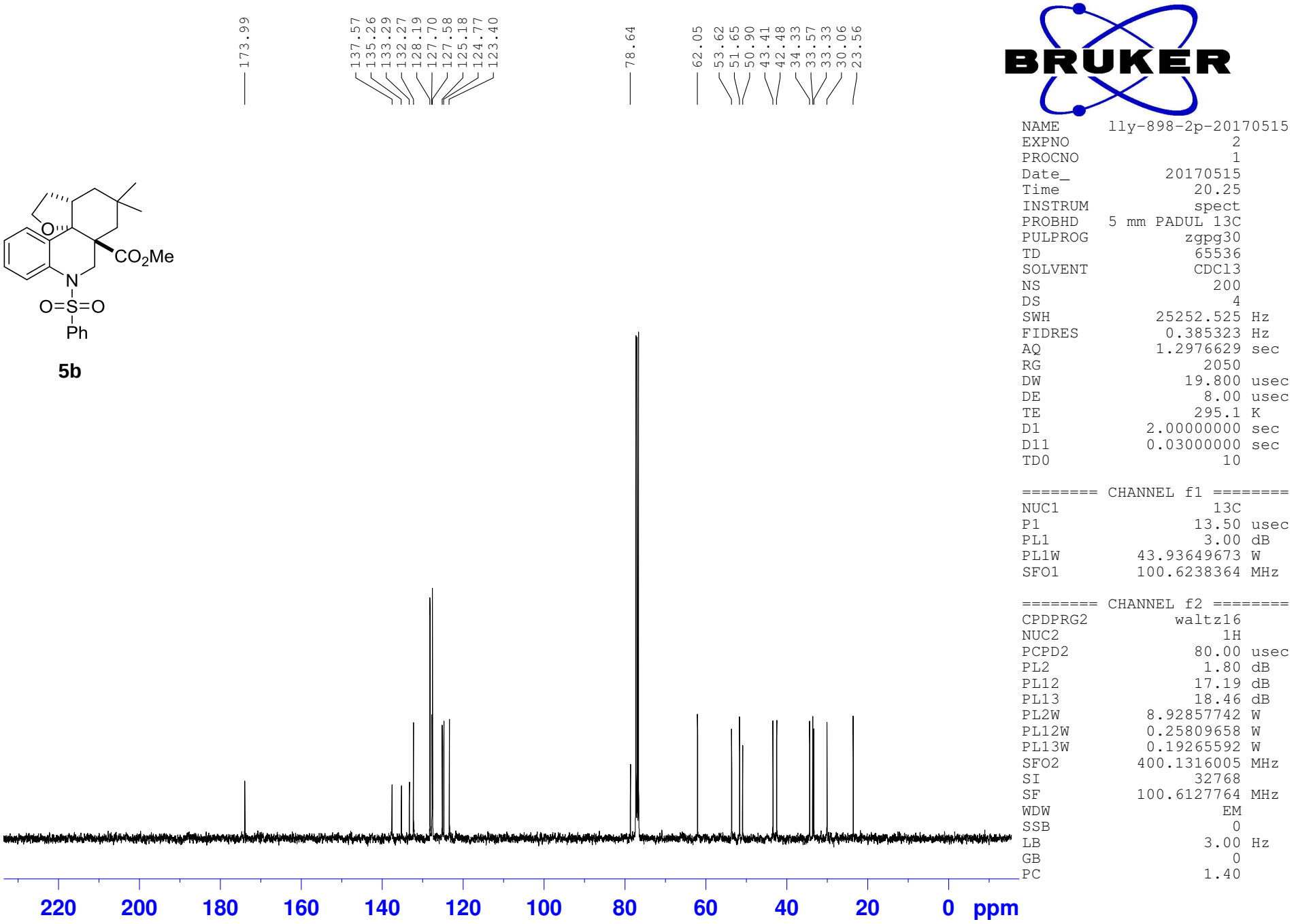




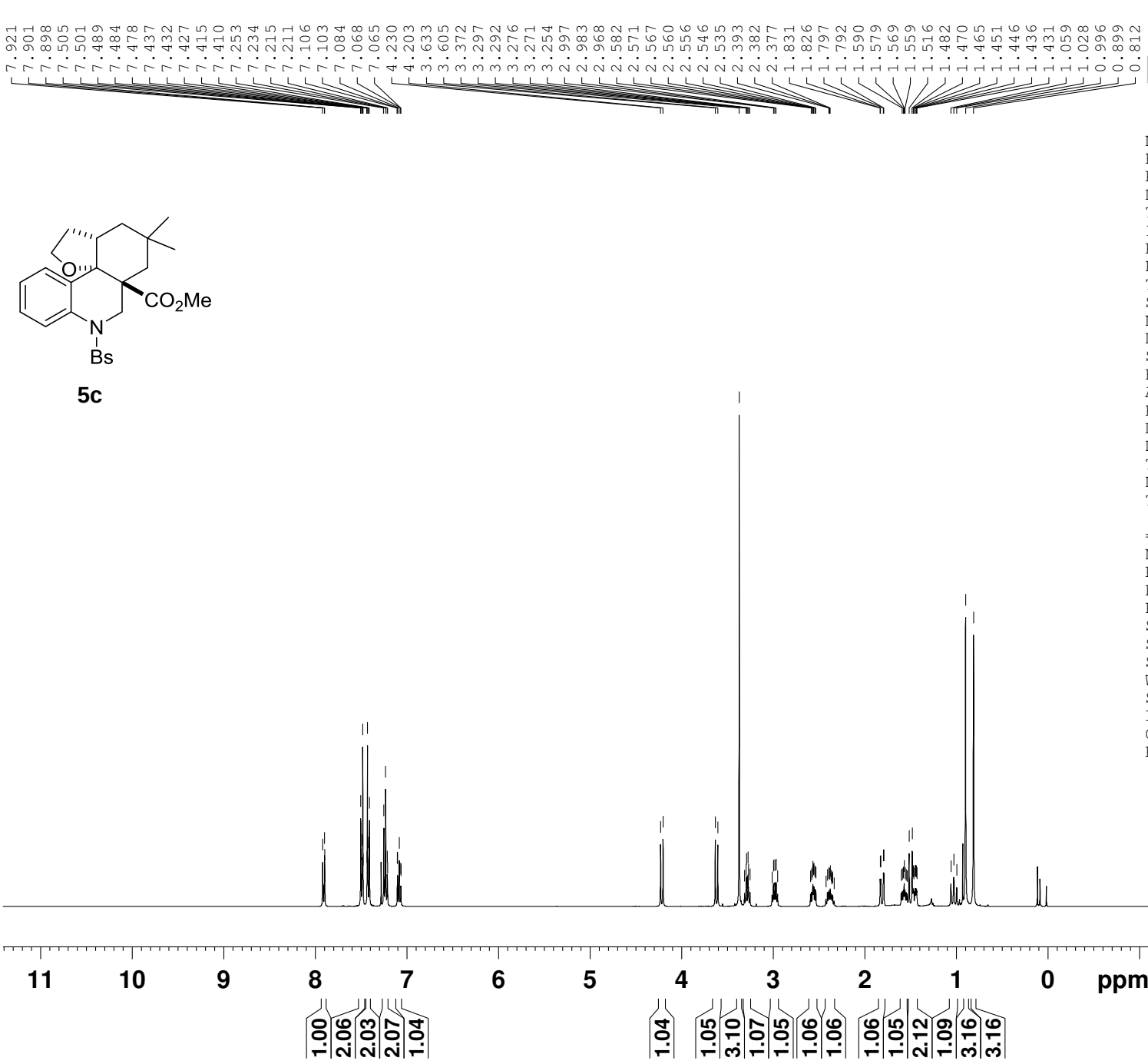
5b

```
NAME      lly-898-2p-20170515
EXPNO                       1
PROCNO                      1
Date_              20170515
Time                   20.22
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                6393.862 Hz
FIDRES            0.195125 Hz
AQ                2.5625076 sec
RG                      161
DW                   78.200 usec
DE                    6.50 usec
TE                   294.4 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W            8.92857742 W
SFO1           400.1326008 MHz
SI                    32768
SF             400.1300005 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

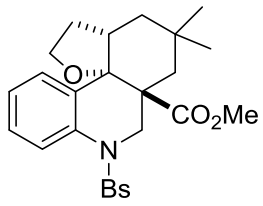





```
NAME      lly-898-2p-20170515
EXPNO                       2
PROCNO                      1
Date_              20170515
Time                   20.25
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       200
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                     295.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        10

======== CHANNEL f1 ========
NUC1                      13C
P1                      13.50 usec
PL1                      3.00 dB
PL1W             43.93649673 W
SFO1            100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                     1.80 dB
PL12                   17.19 dB
PL13                   18.46 dB
PL2W              8.92857742 W
PL12W             0.25809658 W
PL13W             0.19265592 W
SFO2            400.1316005 MHz
SI                      32768
SF              100.6127764 MHz
WDW                        EM
SSB                         0
LB                      3.00 Hz
GB                          0
PC                      1.40
```

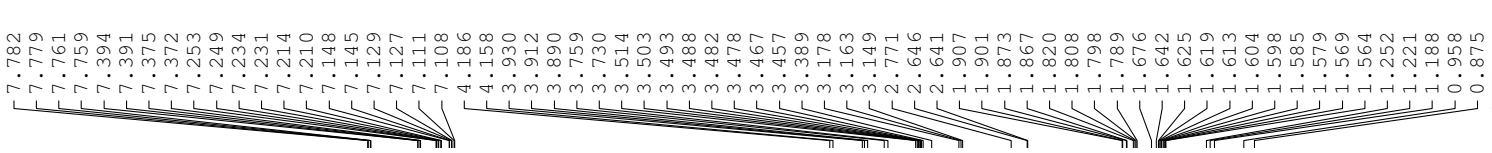


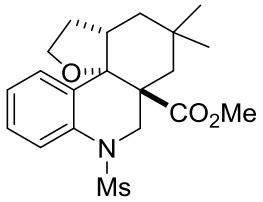

```
NAME      lly-898-3p-20170515
EXPNO                       1
PROCNO                      1
Date_              20170515
Time                   20.39
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       101
DW                    78.200 usec
DE                     6.50 usec
TE                    294.4 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



5c

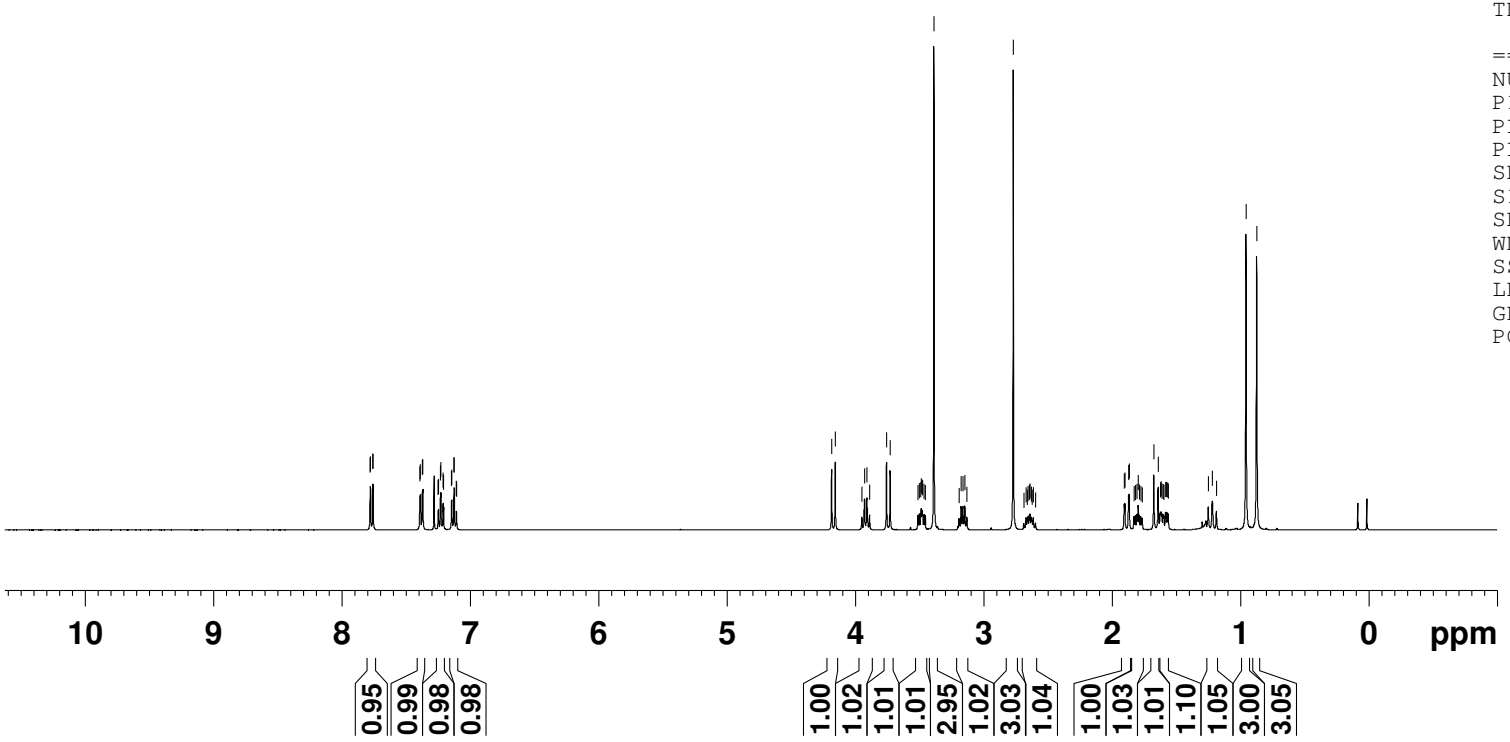

```
NAME          lly-898-3p-20170515
EXPNO                           2
PROCNO                          1
Date_                    20170515
Time                        20.54
INSTRUM                     spect
PROBHD          5 mm PADUL 13C
PULPROG                    zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            240
DS                              4
SWH                     25252.525 Hz
FIDRES                   0.385323 Hz
AQ                       1.2976629 sec
RG                           2050
DW                         19.800 usec
DE                          8.00 usec
TE                         295.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             10

======== CHANNEL f1 ========
NUC1                           13C
P1                          13.50 usec
PL1                          3.00 dB
PL1W                43.93649673 W
SFO1                100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                           1H
PCPD2                       80.00 usec
PL2                          1.80 dB
PL12                        17.19 dB
PL13                        18.46 dB
PL2W                 8.92857742 W
PL12W                0.25809658 W
PL13W                0.19265592 W
SFO2                400.1316005 MHz
SI                         32768
SF                  100.6127764 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.40
```

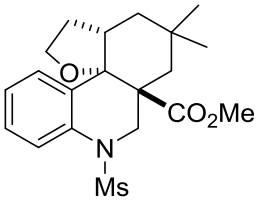




5d

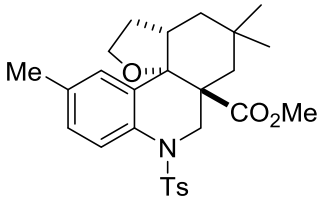
```
NAME      lly-898-1p-20170515
EXPNO                       1
PROCNO                      1
Date_              20170515
Time                   19.46
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       161
DW                    78.200 usec
DE                     6.50 usec
TE                    294.1 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300005 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



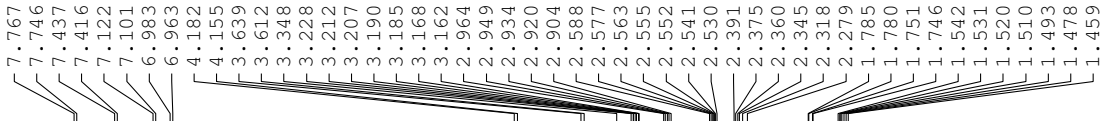


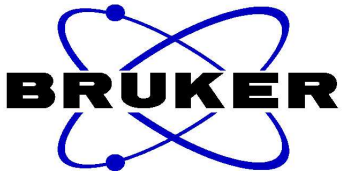
5d


```
NAME        lly-898-1p-20170515
EXPNO                         2
PROCNO                        1
Date_                  20170515
Time                      19.49
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         528
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      295.1 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

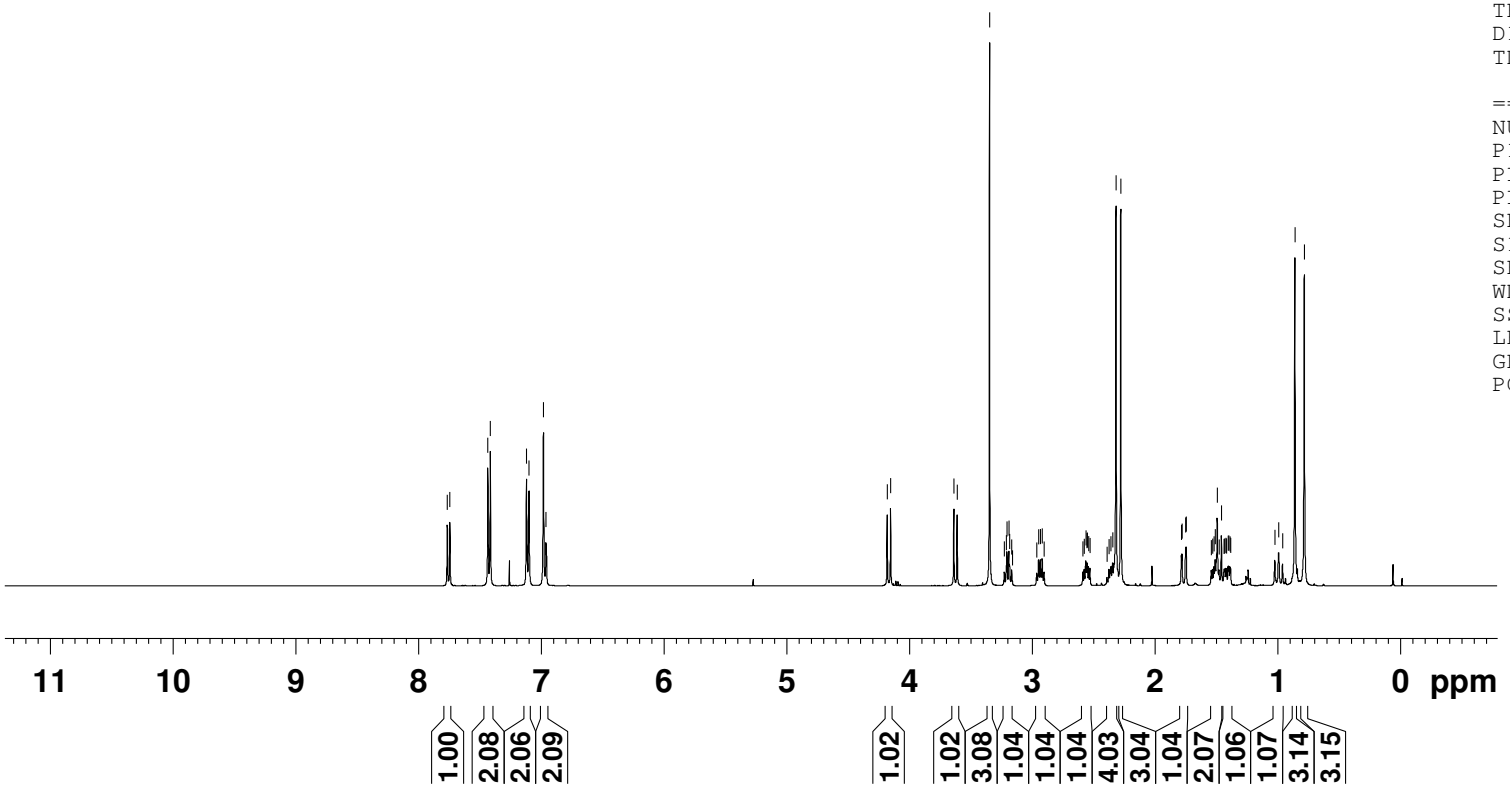


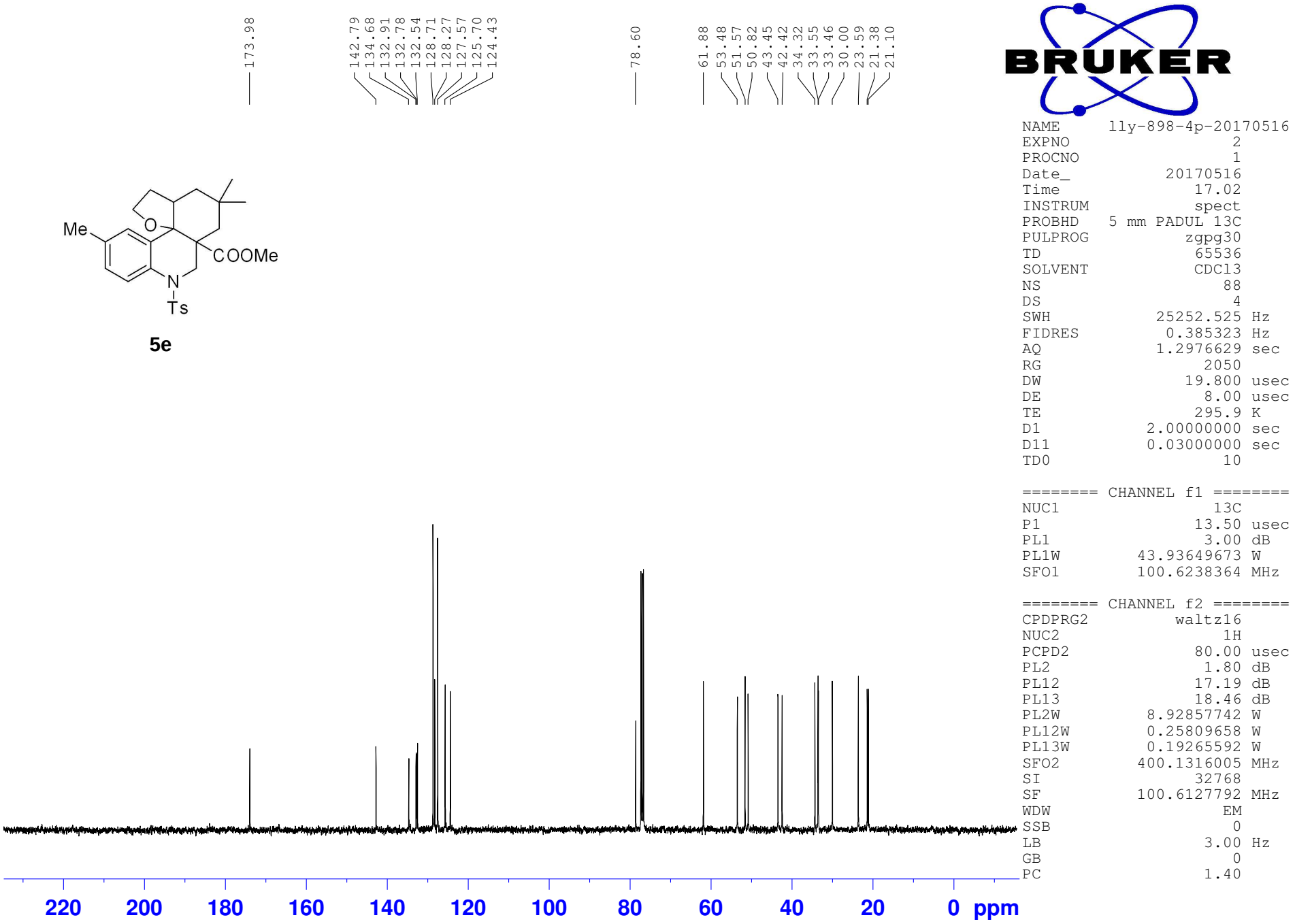
5e



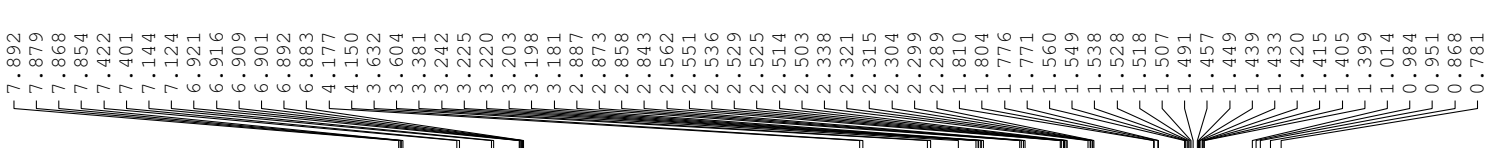


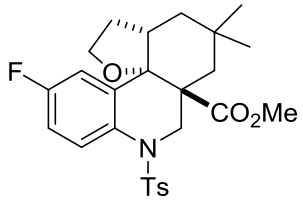

```
NAME      lly-898-4p-20170516
EXPNO                       1
PROCNO                      1
Date_              20170516
Time                   16.55
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                        57
DW                   78.200 usec
DE                    6.50 usec
TE                   294.7 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300096 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00
```





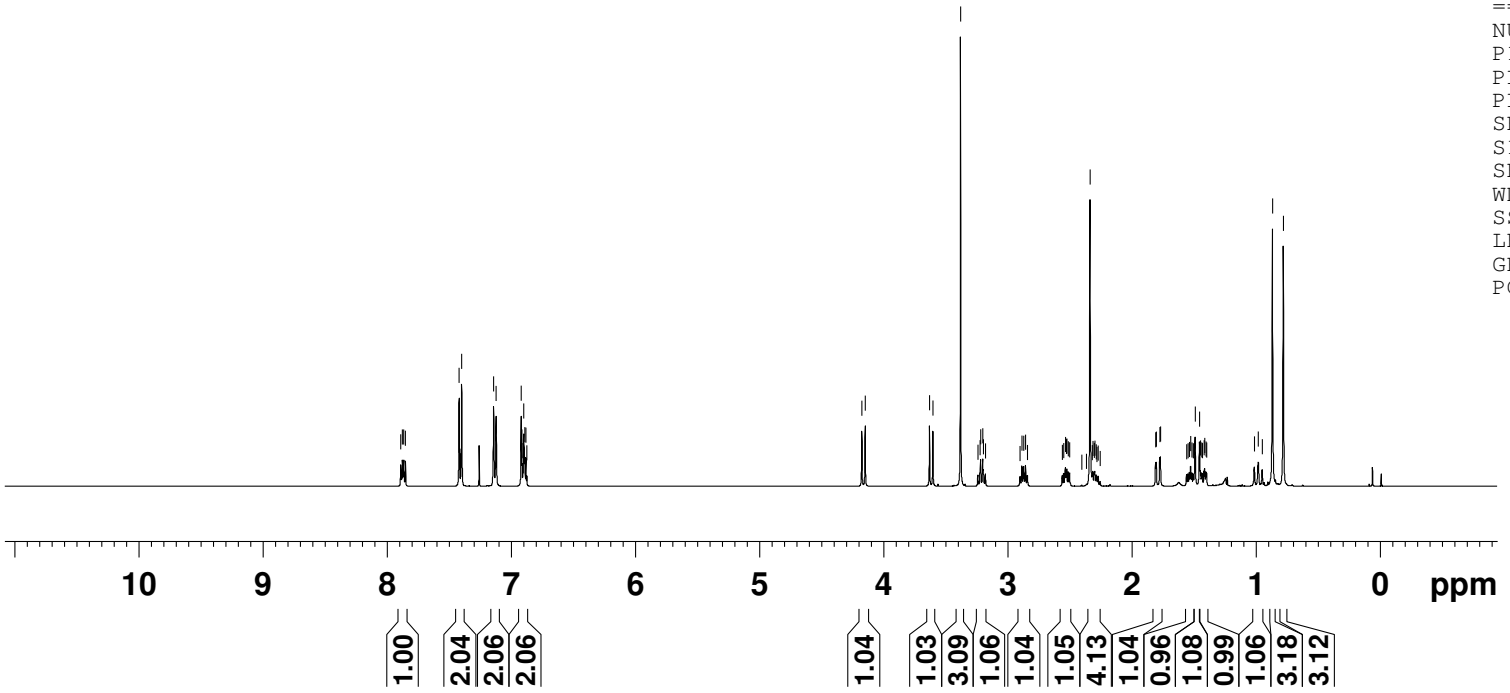

```
NAME      lly-898-4p-20170516
EXPNO                       2
PROCNO                      1
Date_                20170516
Time                    17.02
INSTRUM                 spect
PROBHD   5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          88
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      295.9 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                      13.50 usec
PL1                      3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      1.80 dB
PL12                    17.19 dB
PL13                    18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127792 MHz
WDW                         EM
SSB                          0
LB                       3.00 Hz
GB                           0
PC                        1.40
```

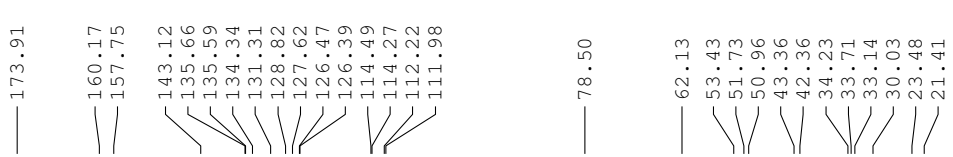


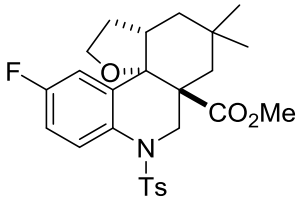


5f

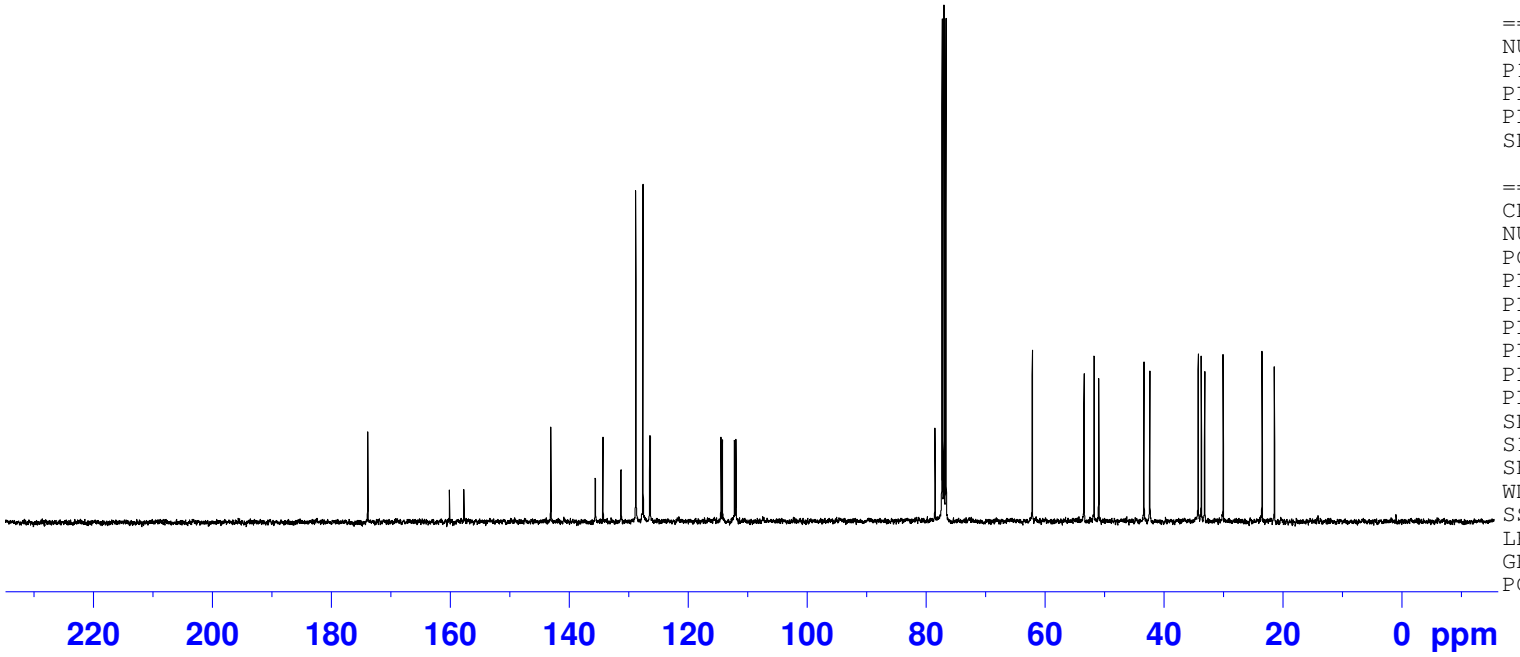
```
NAME      lly-898-6p-20170516
EXPNO                       1
PROCNO                      1
Date_                20170516
Time                    17.32
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  6393.862 Hz
FIDRES               0.195125 Hz
AQ                   2.5625076 sec
RG                       90.5
DW                     78.200 usec
DE                      6.50 usec
TE                     294.8 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      13.10 usec
PL1                      1.80 dB
PL1W              8.92857742 W
SFO1             400.1326008 MHz
SI                      32768
SF               400.1300096 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```



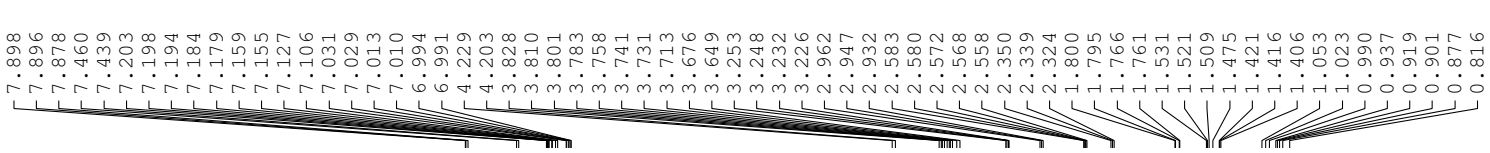


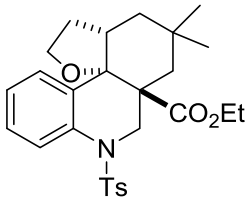


5f



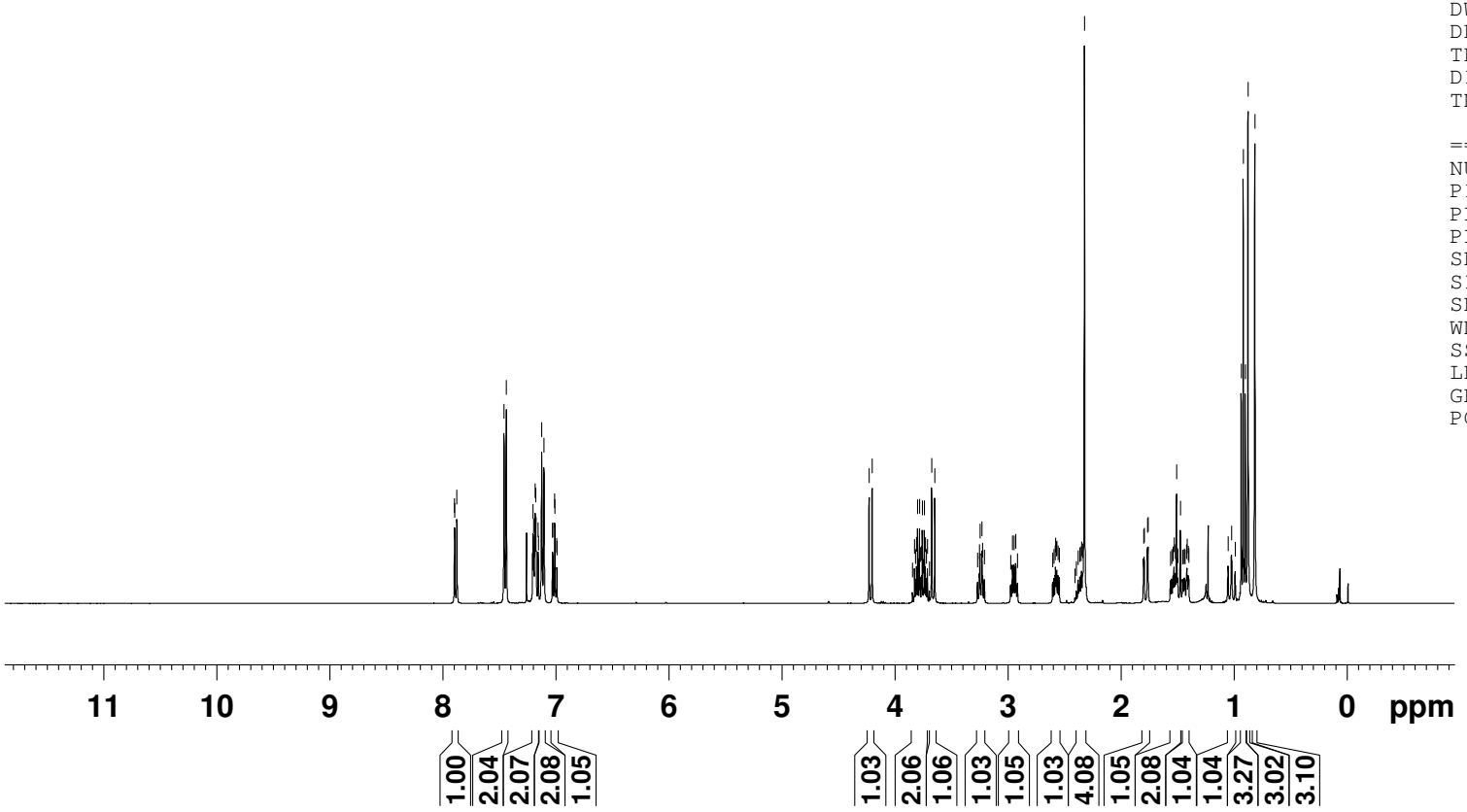

```
NAME        lly-898-6p-20170516
EXPNO                         2
PROCNO                        1
Date_                  20170516
Time                      17.35
INSTRUM                   spect
PROBHD    5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         536
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      295.5 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          10

======== CHANNEL f1 ========
NUC1                        13C
P1                        13.50 usec
PL1                        3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127764 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

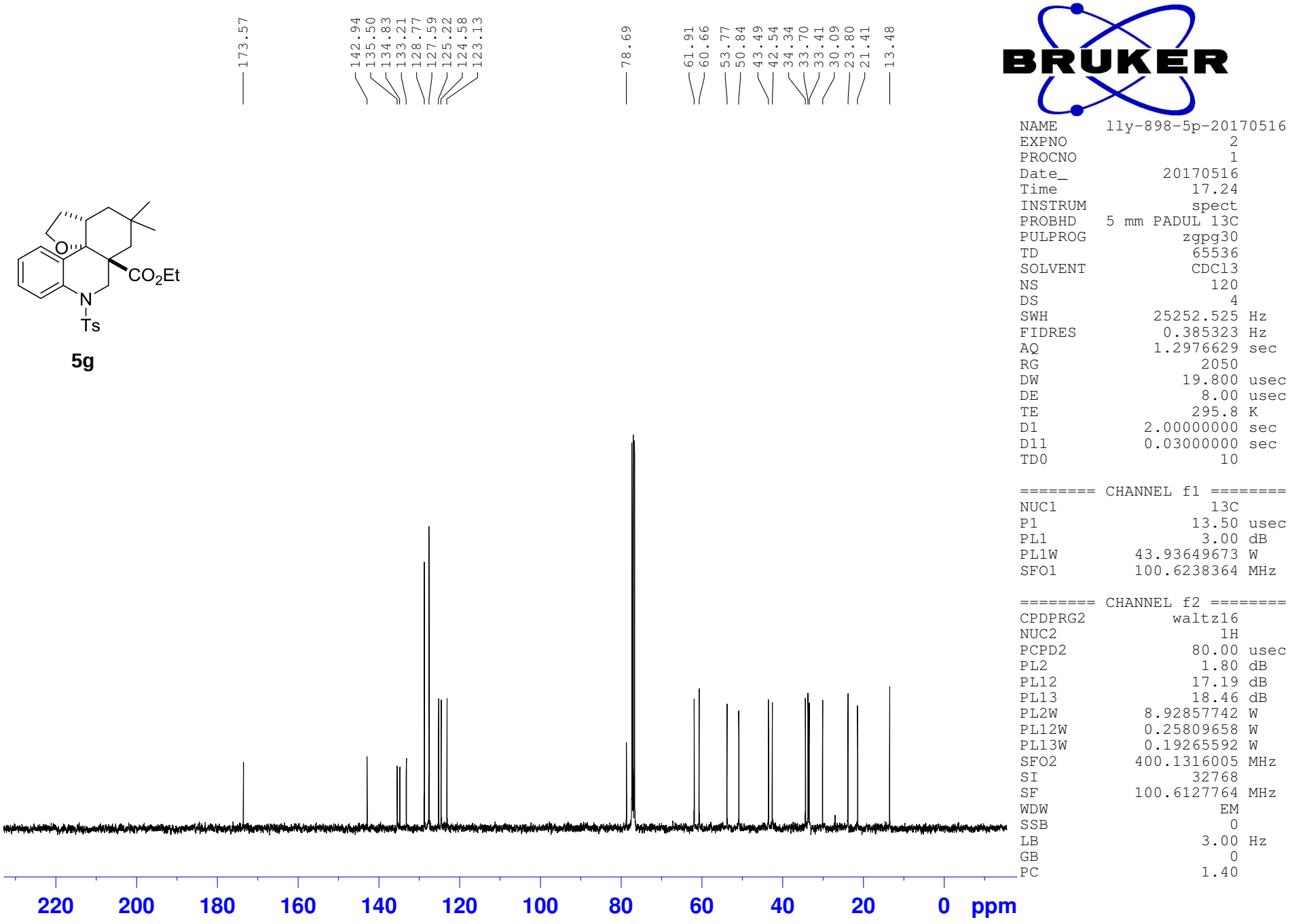




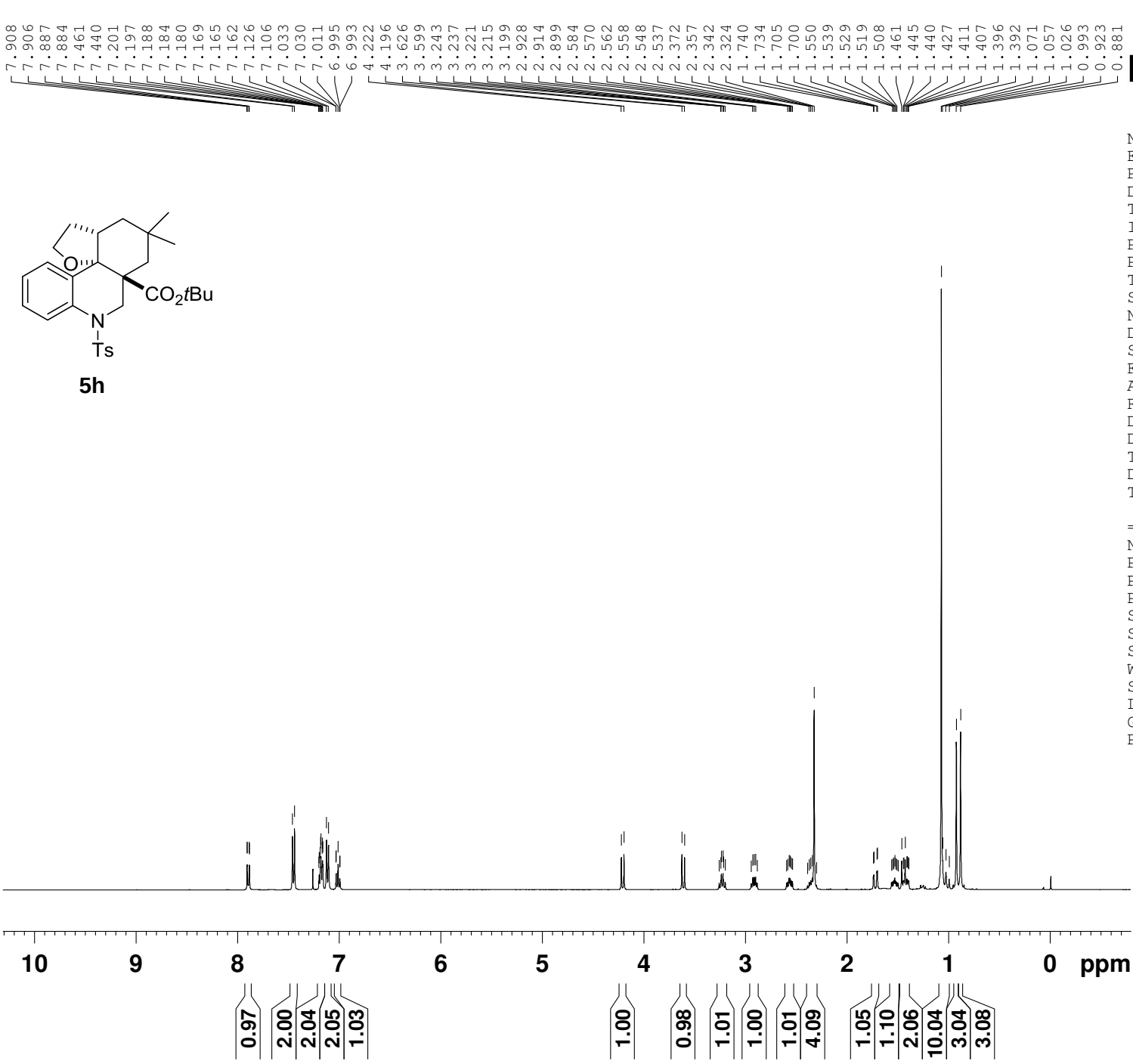
5g

```
NAME      lly-898-5p-20170516
EXPNO                       1
PROCNO                      1
Date_              20170516
Time                   17.11
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                  2.5625076 sec
RG                      90.5
DW                    78.200 usec
DE                     6.50 usec
TE                    294.7 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300096 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.00
```

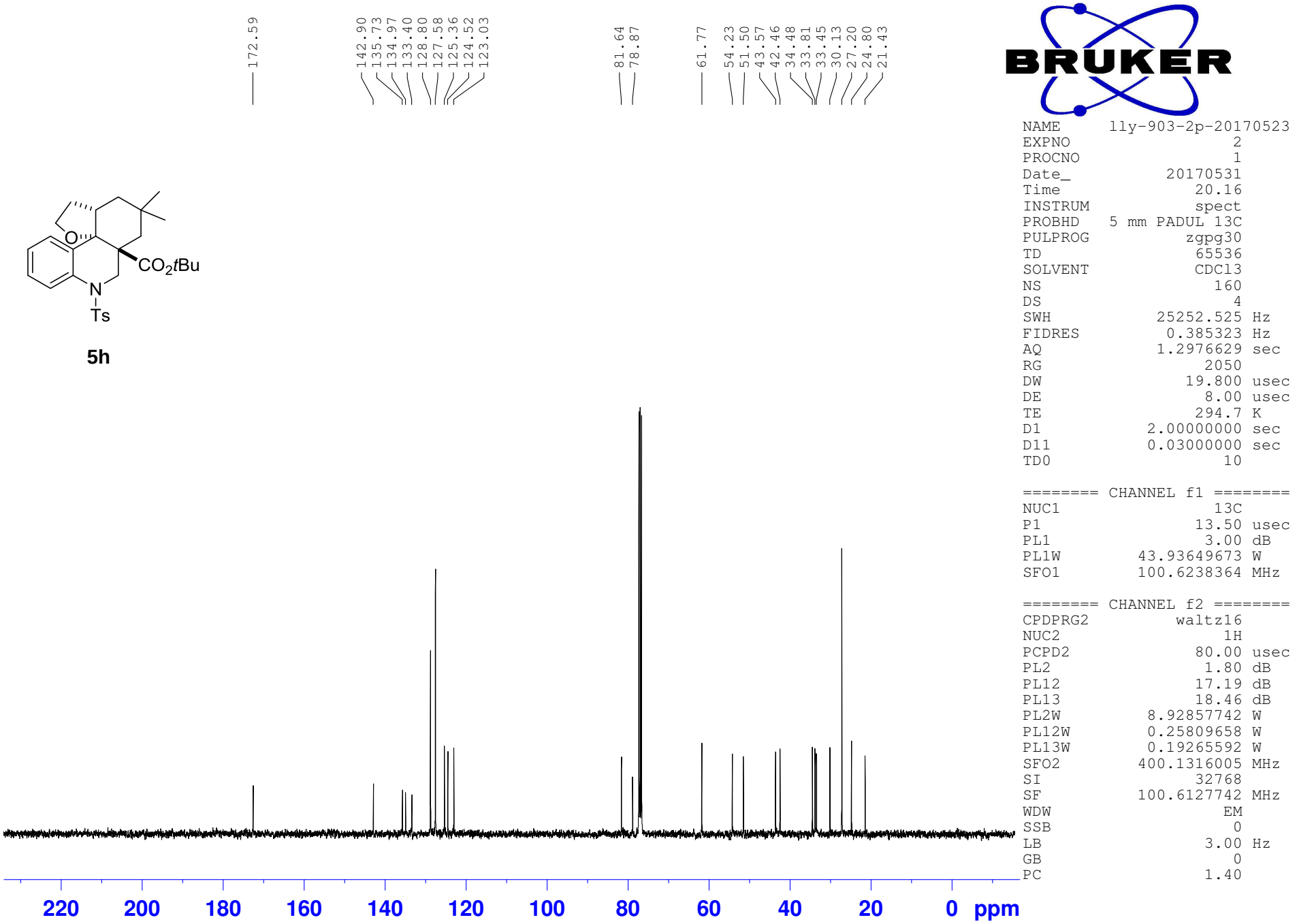




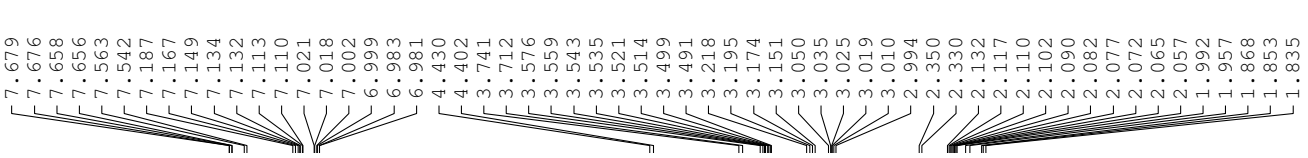

```
NAME      lly-898-5p-20170516
EXPNO                       2
PROCNO                      1
Date_              20170516
Time                   17.24
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      120
DS                        4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                     2050
DW                   19.800 usec
DE                    8.00 usec
TE                   295.8 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W           43.93649673 W
SFO1           100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                   1.80 dB
PL12                 17.19 dB
PL13                 18.46 dB
PL2W            8.92857742 W
PL12W           0.25809658 W
PL13W           0.19265592 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127764 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40
```

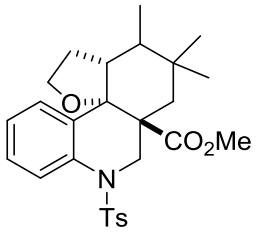



```
NAME      lly-903-2p-20170523
EXPNO                       1
PROCNO                      1
Date_              20170531
Time                   20.12
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       101
DW                    78.200 usec
DE                     6.50 usec
TE                    293.7 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



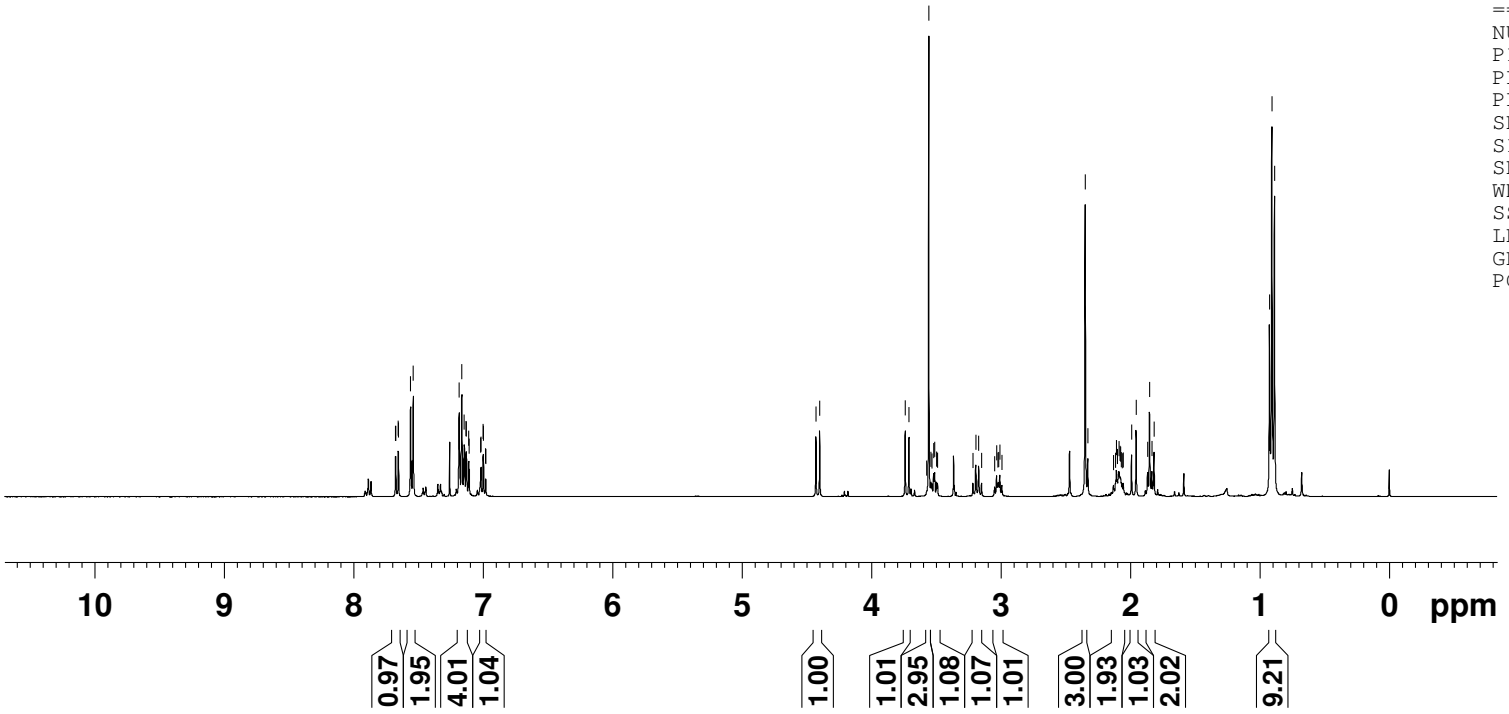

```
NAME      lly-903-2p-20170523
EXPNO                       2
PROCNO                      1
Date_                20170531
Time                    20.16
INSTRUM                 spect
PROBHD   5 mm PADUL 13C
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         160
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                        2050
DW                      19.800 usec
DE                       8.00 usec
TE                      294.7 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                         10

======== CHANNEL f1 ========
NUC1                       13C
P1                       13.50 usec
PL1                       3.00 dB
PL1W               43.93649673 W
SFO1               100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    80.00 usec
PL2                       1.80 dB
PL12                     17.19 dB
PL13                     18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2               400.1316005 MHz
SI                        32768
SF                 100.6127742 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.40
```

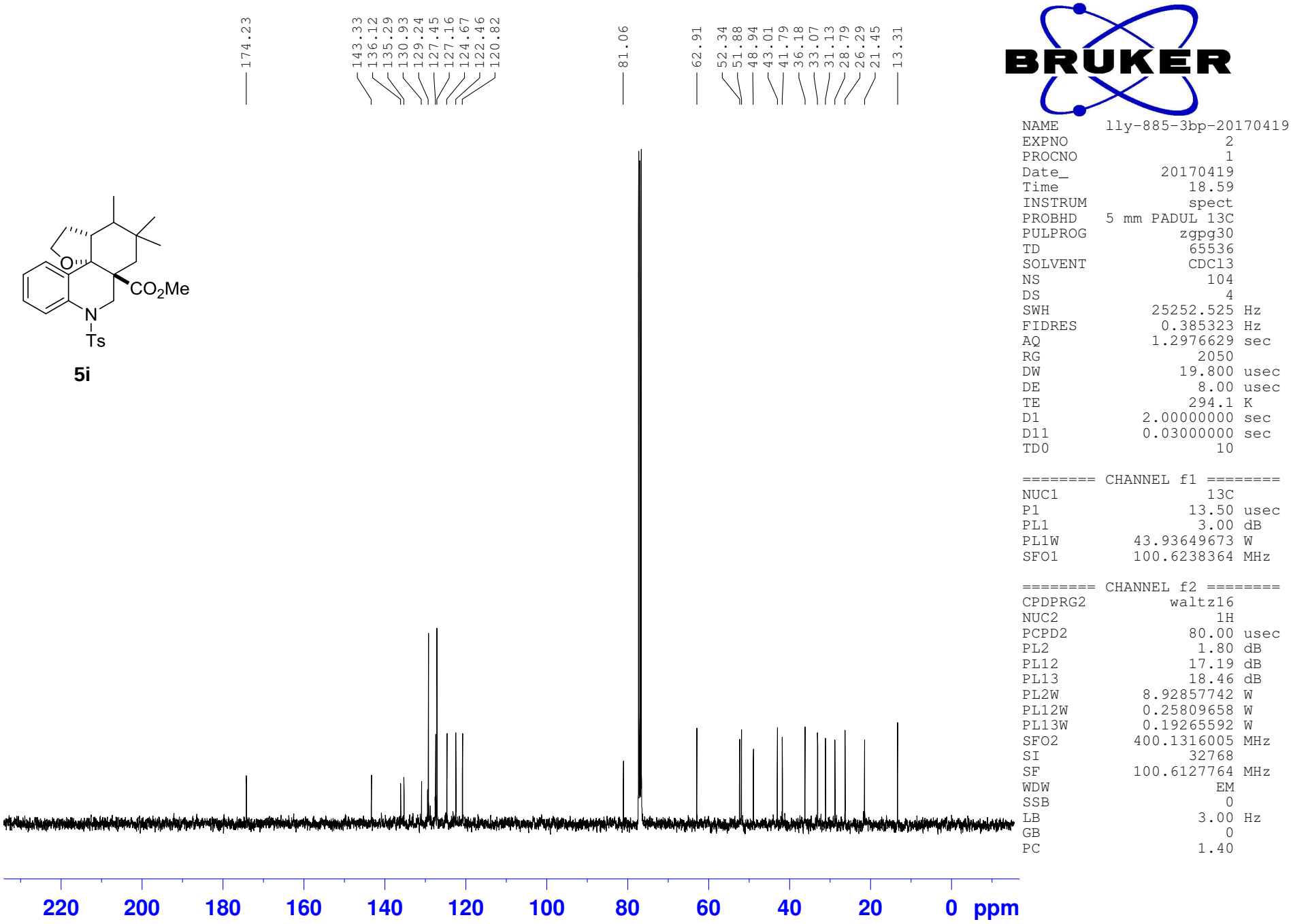




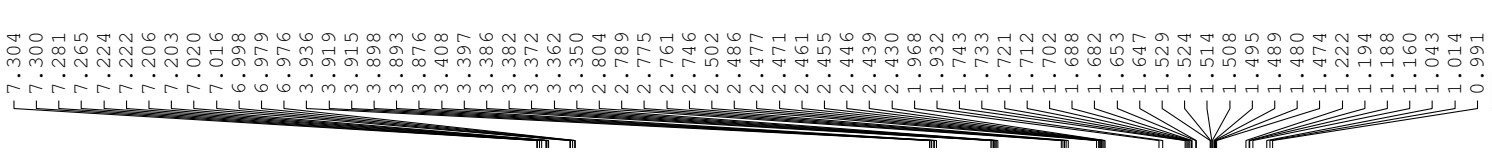
5i

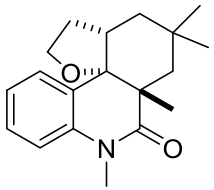
```
NAME      lly-885-3bp-20170419
EXPNO                        1
PROCNO                       1
Date_             20170419
Time                    18.54
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                  zg30
TD                      32768
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                         128
DW                    78.200 usec
DE                     6.50 usec
TE                     293.2 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W              8.92857742 W
SFO1            400.1326008 MHz
SI                       32768
SF              400.1300096 MHz
WDW                         EM
SSB                          0
LB                     0.30 Hz
GB                           0
PC                      1.00
```





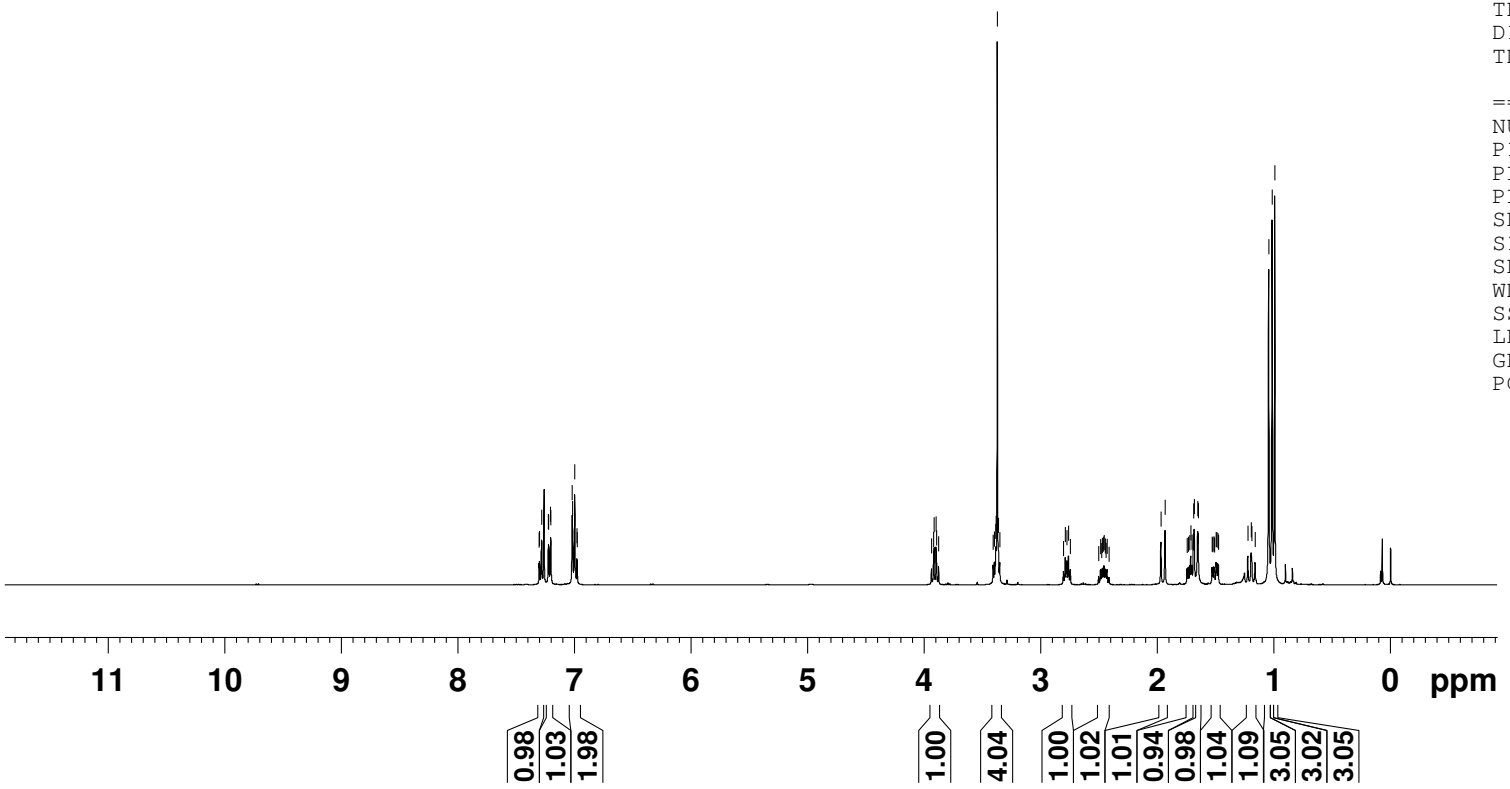

```
NAME      lly-885-3bp-20170419
EXPNO                        2
PROCNO                       1
Date_             20170419
Time                    18.59
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       104
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                1.2976629 sec
RG                      2050
DW                    19.800 usec
DE                     8.00 usec
TE                    294.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       10

======== CHANNEL f1 ========
NUC1                     13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W            43.93649673 W
SFO1           100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                    1.80 dB
PL12                  17.19 dB
PL13                  18.46 dB
PL2W            8.92857742 W
PL12W           0.25809658 W
PL13W           0.19265592 W
SFO2           400.1316005 MHz
SI                    32768
SF             100.6127764 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40
```

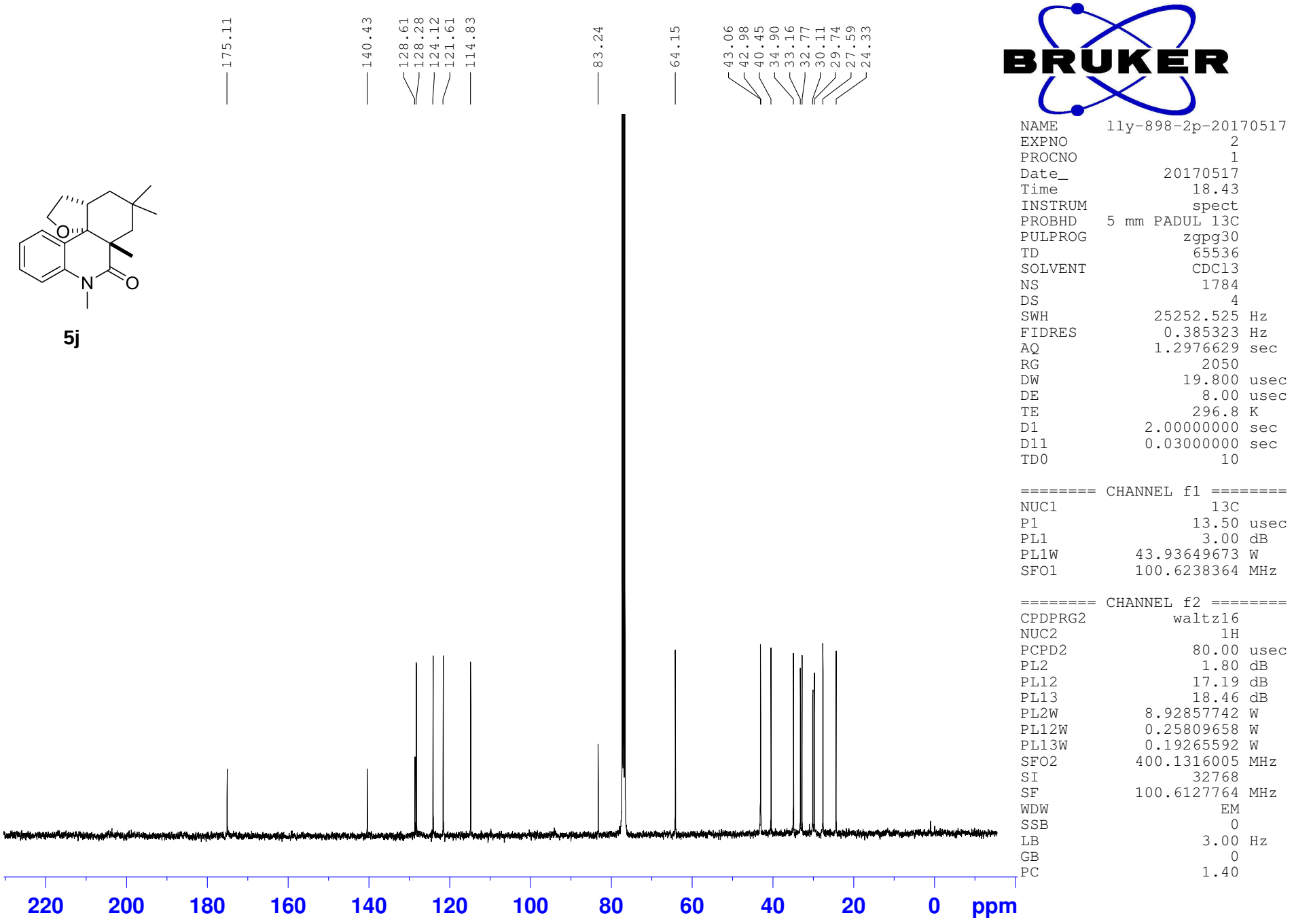




5j

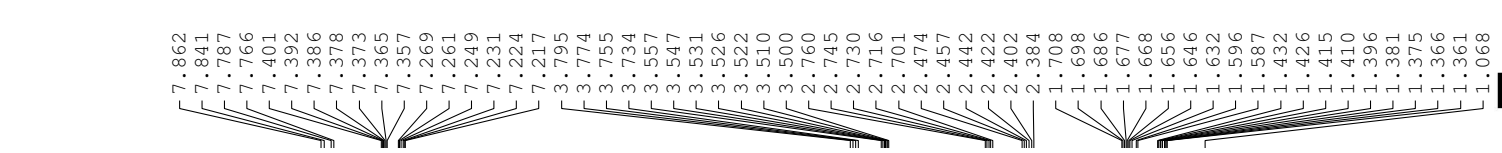
```
NAME      lly-899-2p-20170517
EXPNO                       1
PROCNO                      1
Date_              20170517
Time                   16.51
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       256
DW                    78.200 usec
DE                     6.50 usec
TE                    295.1 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300096 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00
```

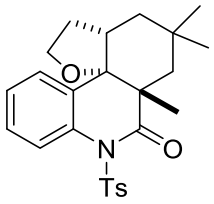




5j

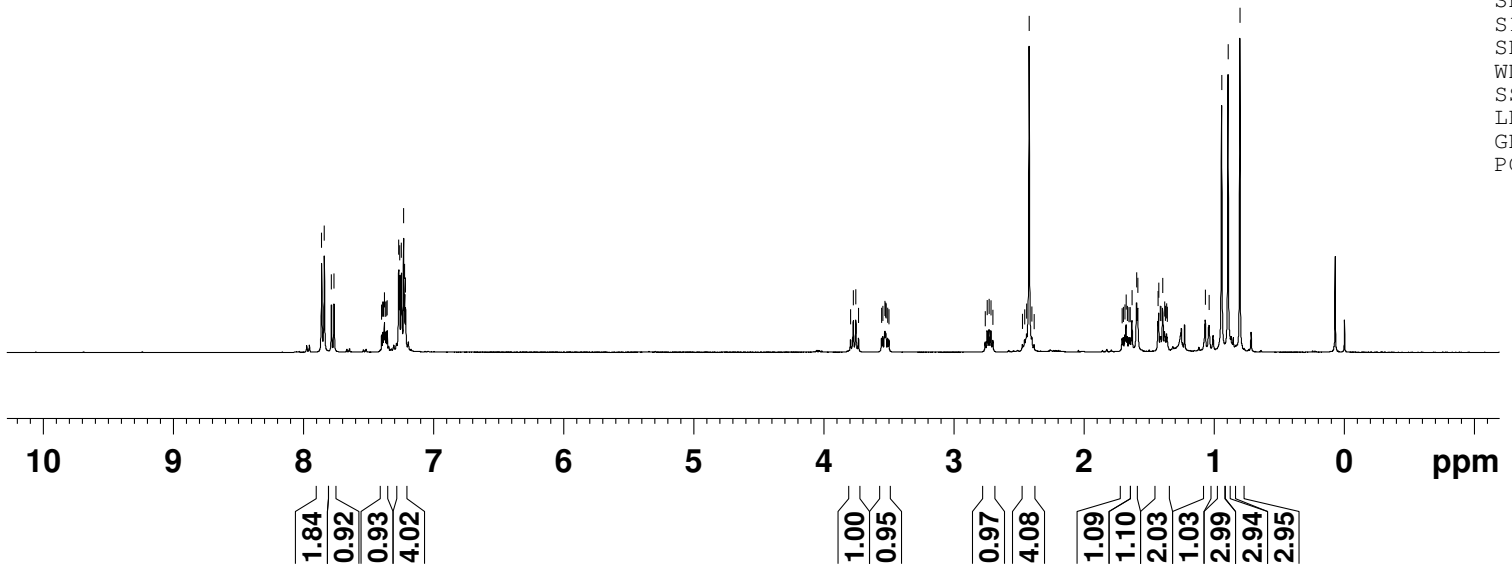
NAME	lly-898-2p-20170517
EXPNO	2
PROCNO	1
Date_	20170517
Time	18.43
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1784
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	2050
DW	19.800 usec
DE	8.00 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	13.50 usec
PL1	3.00 dB
PL1W	43.93649673 W
SFO1	100.6238364 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.80 dB
PL12	17.19 dB
PL13	18.46 dB
PL2W	8.92857742 W
PL12W	0.25809658 W
PL13W	0.19265592 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127764 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

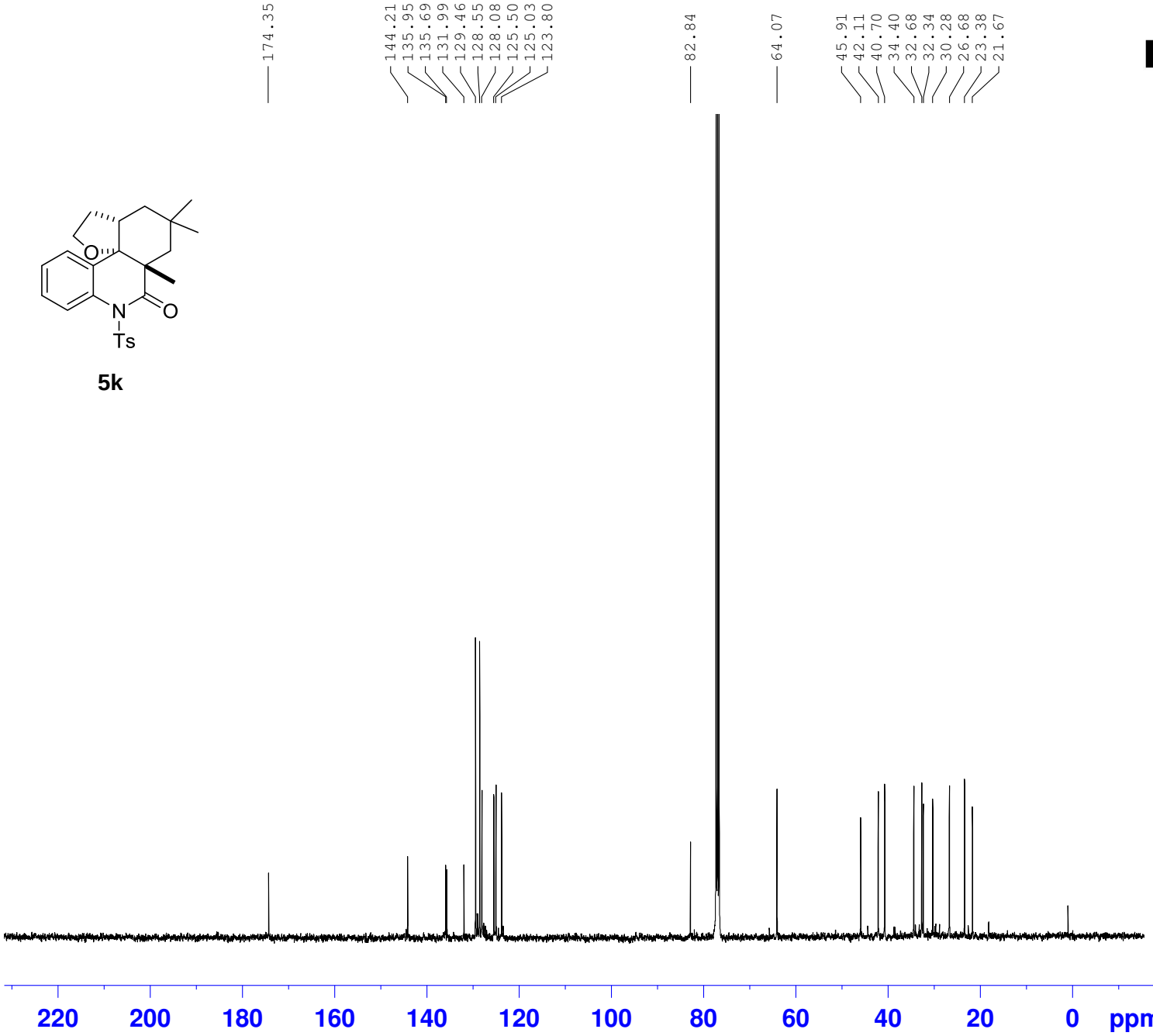




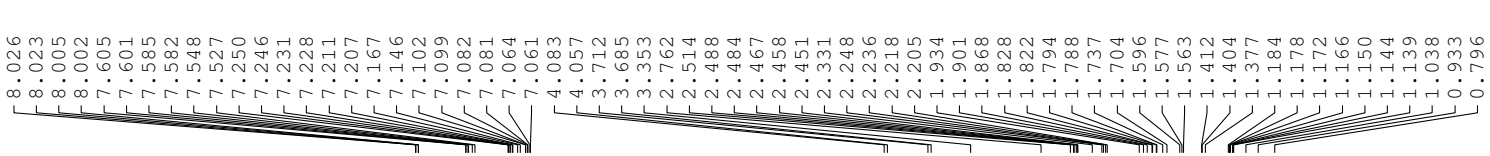
5k

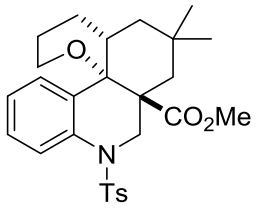
```
NAME      lly-899-3p-20170517
EXPNO                       1
PROCNO                      1
Date_              20170517
Time                   16.46
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       256
DW                    78.200 usec
DE                     6.50 usec
TE                    295.1 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1           400.1326008 MHz
SI                     32768
SF             400.1300096 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00
```





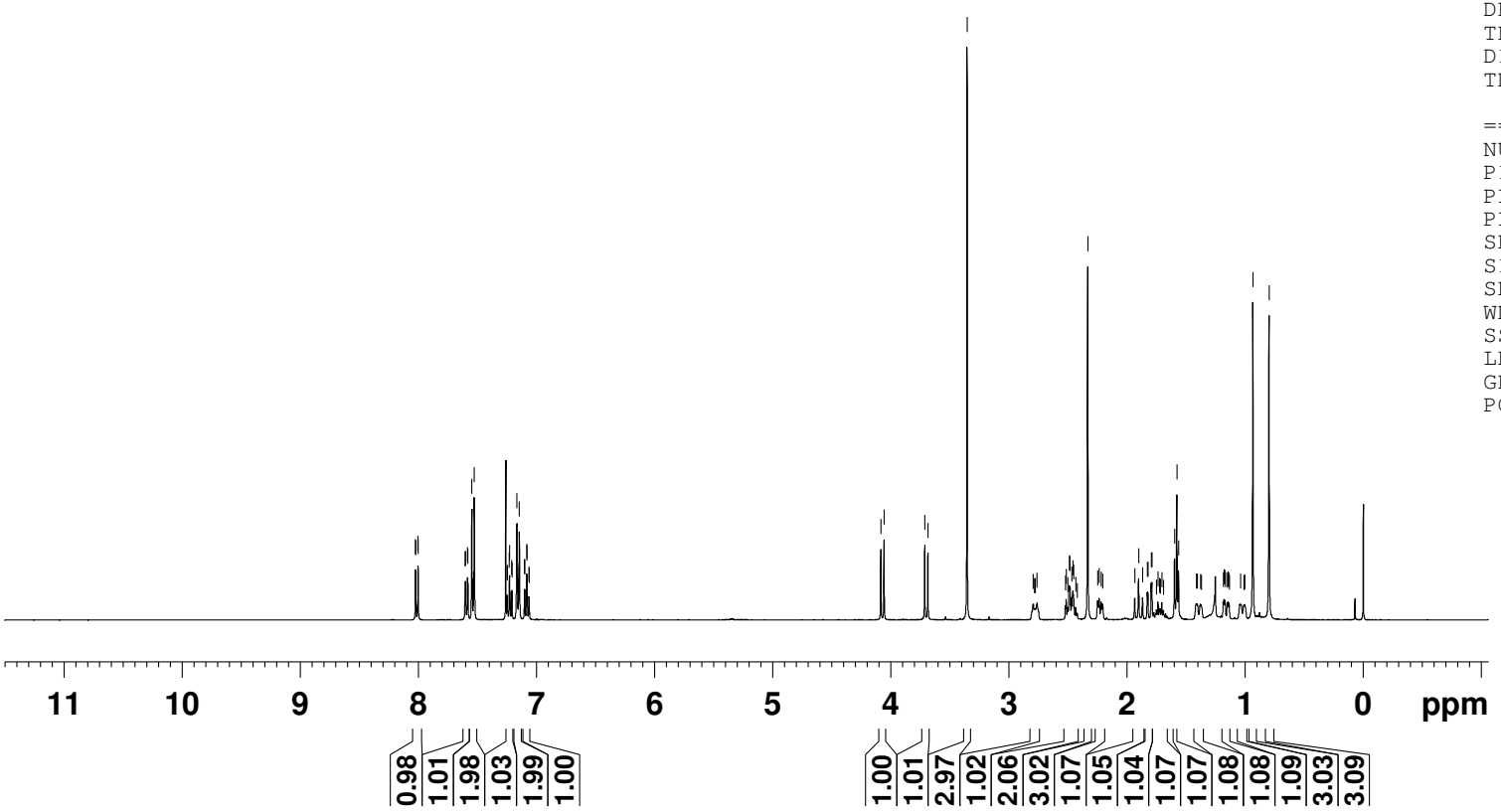

```
NAME      lly-899-3pa-20170517
EXPNO                        2
PROCNO                       1
Date_                 20170517
Time                     19.30
INSTRUM                  spect
PROBHD    5 mm PADUL 13C
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1592
DS                          4
SWH                 25252.525 Hz
FIDRES               0.385323 Hz
AQ                  1.2976629 sec
RG                       2050
DW                     19.800 usec
DE                      8.00 usec
TE                      296.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        10

======== CHANNEL f1 ========
NUC1                      13C
P1                      13.50 usec
PL1                      3.00 dB
PL1W               43.93649673 W
SFO1              100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                      1.80 dB
PL12                    17.19 dB
PL13                    18.46 dB
PL2W                8.92857742 W
PL12W               0.25809658 W
PL13W               0.19265592 W
SFO2              400.1316005 MHz
SI                       32768
SF                100.6127764 MHz
WDW                         EM
SSB                          0
LB                       3.00 Hz
GB                           0
PC                       1.40
```

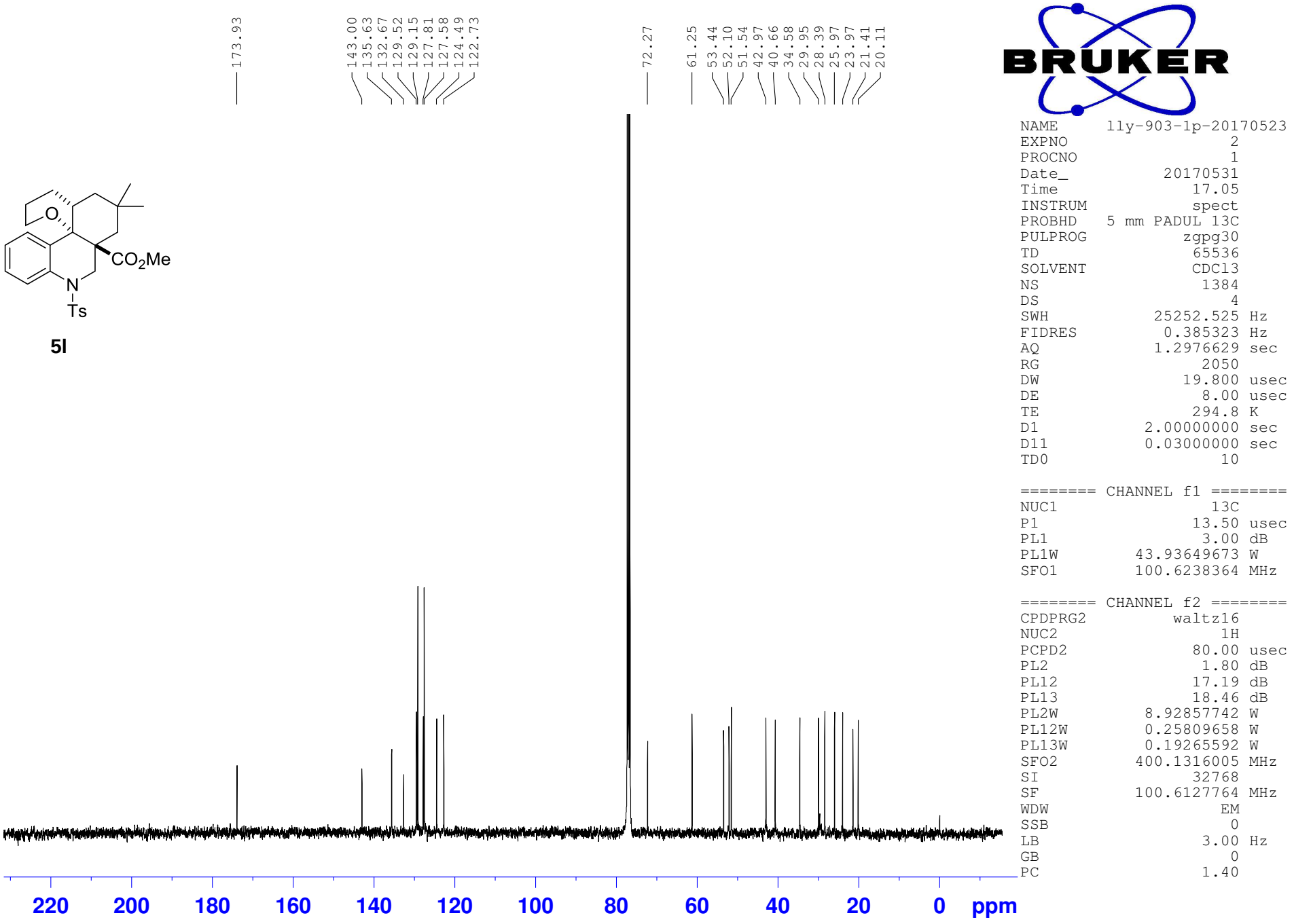




5l

```
NAME      lly-903-1p-20170523
EXPNO                       1
PROCNO                      1
Date_              20170531
Time                   17.00
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 6393.862 Hz
FIDRES              0.195125 Hz
AQ                2.5625076 sec
RG                       203
DW                    78.200 usec
DE                     6.50 usec
TE                    293.8 K
D1               1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     13.10 usec
PL1                     1.80 dB
PL1W             8.92857742 W
SFO1            400.1326008 MHz
SI                     32768
SF              400.1300096 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```






```
NAME      lly-903-1p-20170523
EXPNO                       2
PROCNO                      1
Date_              20170531
Time                   17.05
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1384
DS                          4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                1.2976629 sec
RG                       2050
DW                   19.800 usec
DE                    8.00 usec
TE                    294.8 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        10

======== CHANNEL f1 ========
NUC1                      13C
P1                     13.50 usec
PL1                     3.00 dB
PL1W            43.93649673 W
SFO1           100.6238364 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                    1.80 dB
PL12                  17.19 dB
PL13                  18.46 dB
PL2W             8.92857742 W
PL12W            0.25809658 W
PL13W            0.19265592 W
SFO2           400.1316005 MHz
SI                       32768
SF             100.6127764 MHz
WDW                        EM
SSB                         0
LB                     3.00 Hz
GB                          0
PC                     1.40
```