Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Cesium carbonate promoted cascade reaction involving DMF as a reactant for the synthesis of dihydropyrrolizino[3,2-*b*]indol-10-ones

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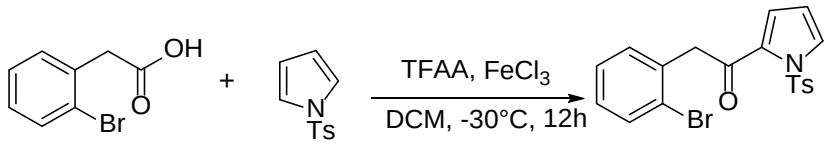
Contents:

1. General remarks

Melting points were determined on a XT4A hot-stage apparatus and are uncorrected. IR spectra were obtained using an PerkinElmer FT/IR spectrophotometer. ^{1}H and ^{13}C NMR spectra were obtained on a Agilent AV400 instrument. High-resolution mass spectra were recorded on a Micromass Q-TOF mass spectrometer. X-ray diffraction (XRD) patterns of the samples were recorded on a Rigaku B/Max-RB diffractometer.

2. Synthesis and characterization data of compounds 1, 1h–1n, 9a, 3a–3n, 9b and 8

2.1 Synthesis of 2-(2-bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one 1[1]

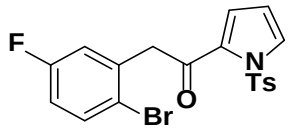


1

A mixture of 1-tosyl-1*H*-pyrrole (221 mg, 1 mmol), 2-(2-bromophenyl)acetic acid (258 mg, 1.2 mmol), TFAA (705 μL, 5 mmol), $FeCl_3$ (80 mg, 0.5 mmol) and DCM (10 mL) under nitrogen was stirred at -30 °C for 12 h. Saturated $NaHCO_3$ (20 mL) was added and the resulting mixture was extracted with EtOAc (3 × 20 mL), then dried (Na_2SO_4), filtered, and evaporated *in vacuo*. The residue was purified by column chromatography on silica gel (5% EtOAc in petroleum ether) to afford the title compound **1** (284 mg, 68%) as a colorless solid; mp 121–122 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.87 (d, J = 8.4 Hz, 2H), 7.80 (dd, J = 3.2, 1.7 Hz, 1H), 7.51 (dd, J = 8.0, 1.1 Hz, 1H), 7.27 (d, J = 8.1 Hz, 2H), 7.21 (td, J = 7.5, 1.2 Hz, 1H), 7.17 (dd, J = 3.8, 1.7 Hz, 1H), 7.14 (dd, J = 7.6, 1.7 Hz, 1H), 7.09 (td, J = 7.8, 1.8 Hz, 1H), 6.33 (t, J = 3.2 Hz, 1H), 4.14 (s, 2H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 184.8, 144.9, 135.8, 134.8, 132.9, 132.8, 131.6, 130. 6, 129.5, 128.8, 128.5, 127.7, 125.0, 124.1, 110.6, 46.4, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1634, 1622, 1427, 1396, 1375, 1140, 1047, 1015; HRMS (ESI): *m/z* calcd for $C_{19}H_{16}{}^{79}BrNO_3SNa$ $[M+Na]^+$: 439.9926; found 439.9943.

Substrates **1h**–**1n**, **9a** were synthesized according to the procedure as describe for compound **1**.

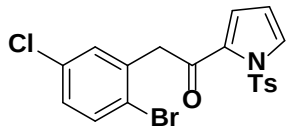
2-(2-Bromo-5-fluorophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1h)



301 mg, 69% as a colorless solid; mp 131–132 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (d, J = 8.4 Hz, 2H), 7.83 (dd, J = 3.2, 1.7 Hz, 1H), 7.46 (m, 1H), 7.28 (d, J = 8.2 Hz, 2H), 7.18 (dd, J = 3.8, 1.7 Hz, 1H), 6.85 – 6.81 (m, 2H), 6.35 (t, J = 3.5 Hz, 1H), 4.11 (s, 2H), 2.41 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 184.0, 161.9 (d, J_{F-C} = 245.6 Hz), 145.1, 136.7 (d, J_{F-C} = 8.0 Hz), 135.64, 133.9 (d, J_{F-C} = 8.1 Hz), 132.6, 130.9, 129.5, 128.5, 124.3, 119.1 (d, J_{F-C} = 3.2 Hz), 118.6 (d, J_{F-C} = 23.1 Hz), 116.1

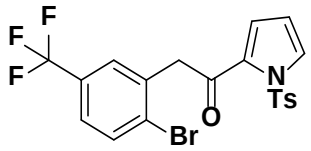
(d, $J_{F\text{-}C}$ = 22.4 Hz), 110.6, 46.2 (d, $J_{F\text{-}C}$ = 1.5 Hz), 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1670, 1248, 1172, 1140, 1043; HRMS (ESI): *m/z* calcd for $C_{19}H_{15}{}^{79}BrFNO_3SNa$ $[M+Na]^+$: 457.9832; found 457.9822.

2-(2-Bromo-5-chlorophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1i)



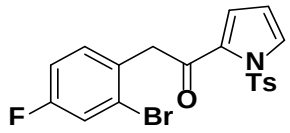
308 mg, 68% as a colorless solid; mp 141–142 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.85 (d, *J* = 8.4 Hz, 2H), 7.83 (dd, *J* = 3.1, 1.8 Hz, 1H), 7.42 (d, *J* = 8.3 Hz, 1H), 7.28 (d, *J* = 8.3 Hz, 2H), 7.18 (dd, *J* = 3.7, 1.6 Hz, 1H), 7.10 – 7.06 (m, 2H), 6.36 (t, *J* = 3.5 Hz, 1H), 4.11 (s, 2H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 183.8, 145.1, 136.4, 135.6, 133.8, 133.5, 132.6, 131. 6, 130.9, 129.5, 128.9, 128.5, 124.2, 122.9, 110.6, 46.1, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1671, 1661, 1169, 1140, 1067, 1043; HRMS (ESI): *m/z* calcd for $C_{19}H_{15}{}^{81}BrClNO_3SNa$ $[M+Na]^+$: 475.9516; found 475.9510.

2-[2-Bromo-5-(trifluoromethyl)phenyl]-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1j)



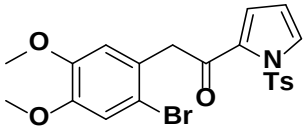
340 mg, 70% as a colorless solid; mp 125–126 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (m, 1H), 7.85–7.84 (m, 2H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.38–7.35 (m, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 7.21 (dd, *J* = 3.8, 1.7 Hz, 1H), 6.37 (t, *J* = 3.6 Hz, 1H), 4.21 (s, 2H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 183.5, 145.1, 135.8, 135.6, 133.4, 132.5, 130.9, 129.5, 129.1 (q, $J_{F\text{-}C}$ = 1.5 Hz), 128.6 (q, $J_{F\text{-}C}$ = 3.8 Hz), 128.5, 126.5 (q, $J_{F\text{-}C}$ = 270.8 Hz), 125.6 (q, $J_{F\text{-}C}$ = 3.8 Hz), 124.2, 110.6, 46.2, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1678, 1330, 1169, 1143, 1129, 1079, 1042; HRMS (ESI): *m/z* calcd for $C_{20}H_{15}{}^{79}BrF_3NO_3SNa$ $[M+Na]^+$: 507.9800; found 507.9809.

2-(2-Bromo-4-fluorophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1k)



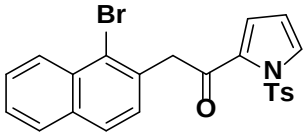
297 mg, 68% as a colorless solid; mp 141–142 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (d, *J* = 8.4 Hz, 2H), 7.81 (dd, *J* = 3.1, 1.7 Hz, 1H), 7.29 – 7.25 (m, 3H), 7.17 (dd, *J* = 3.8, 1.7 Hz, 1H), 7.13 (dd, *J* = 8.5, 6.0 Hz, 1H), 6.94 (td, *J* = 8.3, 2.6 Hz, 1H), 6.35 (t, *J* = 3.5 Hz, 1H), 4.11 (s, 2H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 184.5, 161.5 (d, $J_{F\text{-}C}$ = 248.5 Hz), 145.0, 135.7, 132.7, 132.4 (d, $J_{F\text{-}C}$ = 8.4 Hz), 130.7, 130.6 (d, $J_{F\text{-}C}$ = 3.6 Hz), 129.5, 128.5, 124.9 (d, $J_{F\text{-}C}$ = 9.5 Hz), 124.0, 120.0 (d, $J_{F\text{-}C}$ = 24.3 Hz), 114.8 (d, $J_{F\text{-}C}$ = 20.9 Hz), 110.8, 45.5, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1684, 1489, 1183, 1165, 1142, 1068, 1039; HRMS (ESI): *m/z* calcd for $C_{19}H_{15}{}^{79}BrFNO_3SNa$ $[M+Na]^+$: 457.9832; found 457.9823.

2-(2-Bromo-4,5-dimethoxyphenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1l)



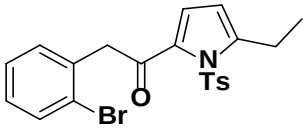
339 mg, 71% as a colorless solid; mp 135–136 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.90 (d, J = 8.4 Hz, 2H), 7.78 (dd, J = 3.2, 1.7 Hz, 1H), 7.29 (d, J = 8.1 Hz, 2H), 7.16 (dd, J = 3.8, 1.7 Hz, 1H), 6.98 (s, 1H), 6.71 (s, 1H), 6.32 (t, J = 3.6 Hz, 1H), 4.06 (s, 2H), 3.83 (s, 3H), 3.79 (s, 3H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 185.6, 148.7, 148.6, 144. 9, 136.0, 132.9, 130.5, 129.5, 128.5, 126.6, 124.0, 115.4, 114.5, 113.5, 110.625, 56.2, 56.1, 46.0, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1658, 1292, 1157, 1136, 1032; HRMS (ESI): *m/z* calcd for $C_{21}H_{20}{}^{81}BrNO_5SNa$ $[M+Na]^+$: 502.0117; found 502.0111.

2-(1-Bromonaphthalen-2-yl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1m)



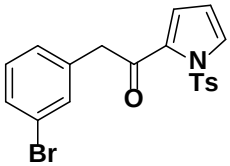
327 mg, 70% as a colorless solid; mp 186–188 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.25 (d, J = 8.6 Hz, 1H), 7.86 (d, J = 8.4 Hz, 2H), 7.77 – 7.80 (m, 2H), 7.70 (d, J = 8.4 Hz, 1H), 7.56 (m, 1H), 7.48 (m, 1H), 7.25 – 7.21 (m, 4H), 6.32 (t, J = 3.6 Hz, 1H), 4.39 (s, 2H), 2.37 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 185.0, 144.9, 135.8, 133.7, 133.0, 132.9, 132.6, 130.6, 129.5, 128.5, 128.4, 128.2, 127.8, 127.6, 127.5, 126.5, 124.9, 124.2, 110.6, 47.7, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1687, 1362, 1312, 1276, 1028; HRMS (ESI): *m/z* calcd for $C_{23}H_{18}{}^{79}BrNO_3SNa$ $[M+Na]^+$: 490.0083; found 490.0086.

2-(2-Bromophenyl)-1-(5-ethyl-1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (1n)



312 mg, 70% as a colorless solid; mp 156–158 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.96 (d, J = 8.4 Hz, 2H), 7.54 (dd, J = 8.0, 1.1 Hz, 1H), 7.34 – 7.25 (m, 4H), 7.12 (td, J = 7.8, 1.8 Hz, 1H), 6.96 (d, J = 3.7 Hz, 1H), 6.05 (dt, J = 3.7, 1.0 Hz, 1H), 4.26 (s, 2H), 2.91 (q, J = 7.3 Hz, 2H), 2.40 (s, 3H), 1.23 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.7, 147.0, 144.9, 136.6, 135.9, 134.9, 132.8, 132.1, 129.7, 128.8, 127.9, 127.7, 125.1, 121.6, 110.5, 48.1, 22.2, 21.8, 13.1 ppm; IR (neat, cm^{-1}) ν_{max} 1674, 1420, 1303, 1174, 1016; HRMS (ESI): *m/z* calcd for $C_{21}H_{20}{}^{79}BrNO_3SNa$ $[M+Na]^+$: 468.0239; found 468.0238.

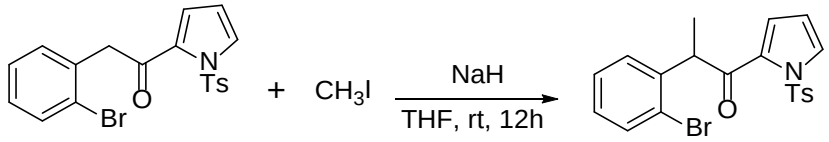
2-(3-Bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)ethan-1-one (9a)



301 mg, 72% as a colorless solid; mp 122–123 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.85 (d, J = 8.4 Hz, 2H), 7.82 (dd, J = 3.1, 1.7 Hz, 1H), 7.34 (d, J = 7.8 Hz, 1H), 7.29 (d, J = 8.2 Hz, 2H), 7.25 (s, 1H), 7.12

(t, J = 7.7 Hz, 1H), 7.09 – 7.06 (m, 2H), 6.34 (t, J = 3.5 Hz, 1H), 3.93 (s, 2H), 2.41 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 185.1, 145.0, 136.8, 135.7, 132.8, 132.3, 130.9, 130.2, 130.2, 129.5, 128.5, 128.0, 124.4, 122.7, 110.6, 45.8, 21.9 ppm; IR (neat, cm^{-1}) ν_{max} 1673, 1354, 1174, 1144, 1063, 1036; HRMS (ESI): *m/z* calcd for $C_{19}H_{16}{}^{81}BrNO_3SNa$ $[M+Na]^+$: 441.9906; found 441.9901.

2.2 Synthesis of 2-(2-bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one 3a[2]

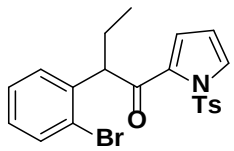


1 **3a**

A mixture of **1** (418 mg, 1 mmol), CH_3I (75 μL, 1.2 mmol), NaH (40 mg, 60% dispersion in mineral oil, 1 mmol) and THF (10 mL) under nitrogen was stirred at rt for 12 h. H_2O (10 mL) was added. The mixture was extracted with EtOAc (3 × 20 mL), then dried (Na_2SO_4), filtered, and evaporated *in vacuo*. The residue was purified by column chromatography on silica gel (3% EtOAc in petroleum ether) to afford **3a** (406 mg, 94%) as a colorless solid; mp 175–176 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.92 (d, J = 8.4 Hz, 2H), 7.75 (dd, J = 3.1, 1.7 Hz, 1H), 7.55 (dd, J = 7.9, 1.2 Hz, 1H), 7.35 (d, J = 8.1 Hz, 2H), 7.14 (td, J = 7.5, 1.2 Hz, 1H), 7.07–7.06 (m, 2H), 6.93 (dd, J = 7.7, 1.7 Hz, 1H), 6.26 (t, J = 3.5 Hz, 1H), 4.78 (q, J = 6.8 Hz, 1H), 2.47 (s, 3H), 1.34 (d, J = 6.8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 188.3, 144.8, 141.4, 136.1, 133.1, 132.9, 130.3, 129.5, 128.6, 128.5, 128.4, 128.2, 123.7, 123.6, 110.6, 47.8, 21.9, 17.7 ppm; IR (neat, cm^{-1}) ν_{max} 1673, 1425, 1174, 1141, 1067, 1023; HRMS (ESI): *m/z* calcd for $C_{29}H_{18}{}^{81}BrNO_3SNa$ $[M+Na]^+$: 456.0063; found 456.0055.

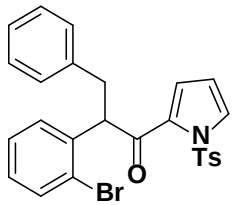
Substrates **3b**–**3n**, **9b** were synthesized according to the procedure given above for compound **3a**.

2-(2-Bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)butan-1-one (3b)



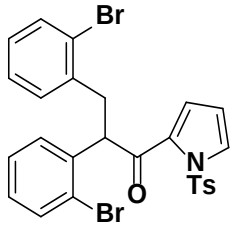
428 mg, 96% as a colorless solid; mp 126–128 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (d, J = 8.3 Hz, 2H), 7.73 (dd, J = 3.1, 1.7 Hz, 1H), 7.52 (dd, J = 7.9, 1.0 Hz, 1H), 7.30 (d, J = 8.1 Hz, 2H), 7.17 (dd, J = 3.8, 1.6 Hz, 1H), 7.10 (t, J = 7.0 Hz, 1H), 7.02 (td, J = 7.7, 1.6 Hz, 1H), 6.96 (dd, J = 7.7, 1.6 Hz, 1H), 6.25 (t, J = 3.5 Hz, 1H), 4.62 (t, J = 7.2 Hz, 1H), 2.43 (s, 3H), 1.98 (m, 1H), 1.64 (m, 1H), 0.81 (t, J = 7.4 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.7, 144.7, 139.6, 136.0, 133.5, 133.0, 130.5, 129.4, 128.7, 128.5, 128.4, 128.0, 124.6, 123.7, 110.5, 54.2, 26.7, 21.8, 12.0 ppm; IR (neat, cm^{-1}) ν_{max} 1666, 1430, 1172, 1139, 1034; HRMS (ESI): *m/z* calcd for $C_{21}H_{21}{}^{81}BrNO_3S$ $[M+H]^+$: 448.0400; found 448.0393.

2-(2-Bromophenyl)-3-phenyl-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3c)



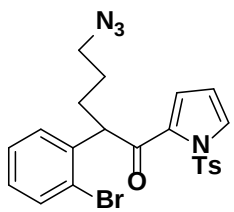
488 mg, 98% as a colorless solid; mp 134–135 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.84 (d, J = 8.4 Hz, 2H), 7.71 (dd, J = 3.1, 1.7 Hz, 1H), 7.48 (dd, J = 8.0, 1.1 Hz, 1H), 7.29 (d, J = 8.1 Hz, 2H), 7.16 – 7.09 (m, 5H), 7.06 – 7.00 (m, 4H), 6.18 (t, J = 3.6 Hz 1H), 4.96 (dd, J = 8.4, 5.7 Hz, 1H), 3.33 (dd, J = 13.6, 8.4 Hz, 1H), 2.79 (dd, J = 13.6, 5.7 Hz, 1H), 2.45 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 186.4, 144.7, 139.2, 139.1, 136.0, 133.2, 133.1, 130.7, 129.4, 129.3, 128.8, 128.7, 128.4, 128.2, 128.1, 126.2, 124.5, 124.1, 110.5, 54.8, 39.0, 21.9 ppm; IR (neat, cm^{-1}) ν_{max} 1670, 1433, 1372, 1172, 1143, 1016; HRMS (ESI): *m/z* calcd for $C_{26}H_{22}{}^{81}BrNO_3SNa$ $[M+Na]^+$: 532.0376; found 532.0368.

2,3-Bis(2-bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3d)



575 mg, 98% as a colorless solid; mp 156–158 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (d, J = 8.4 Hz 2H), 7.73 (dd, J = 3.2, 1.7 Hz, 1H), 7.45 (dd, J = 7.9, 1.3 Hz, 1H), 7.37 (dd, J = 8.0, 1.1 Hz, 1H), 7.31 (d, J = 8.0 Hz, 2H), 7.21 – 7.15 (m, 3H), 7.03 – 6.96 (m, 2H), 6.92 (td, J = 7.4, 1.4 Hz, 1H), 6.59 (dd, J = 7.5, 1.8 Hz, 1H), 6.23 (t, J = 3.6 Hz, 1H), 5.16 (dd, J = 8.8, 5.9 Hz, 1H), 3.46 (dd, J = 13.6, 5.9 Hz, 1H), 2.96 (dd, J = 13.6, 8.8 Hz, 1H), 2.46 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 186.9, 144.8, 138.3, 137.7, 136.0, 133.0, 133.0, 132.7, 131.7, 130.78, 129.5, 129.4, 128.8, 128.4, 128.1, 128.0, 127.0, 125.4, 125.2, 124.1, 110.7, 51.7, 39.9, 21.9 ppm; IR (neat, cm^{-1}) ν_{max} 1665, 1372, 1178, 1141, 1023; HRMS (ESI): *m/z* calcd for $C_{26}H_{21}{}^{81}Br_2NO_3SNa$ $[M+Na]^+$: 609.9481; found 609.9489.

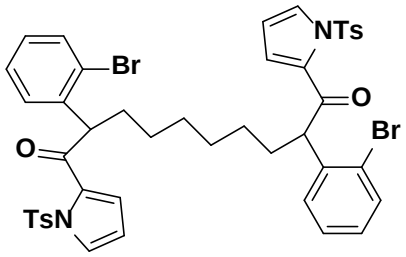
5-Azido-2-(2-bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)pentan-1-one (3e)



496 mg, 99% as a yellow oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (d, J = 8.4 Hz, 2H), 7.74 (dd, J = 3.2, 1.7 Hz, 1H), 7.54 (dd, J = 7.9, 1.3 Hz, 1H), 7.32 (d, J = 8.1 Hz, 2H), 7.14 – 7.10 (m, 2H), 7.04 (td, J = 7.7, 1.8 Hz, 1H), 6.96 (dd, J = 7.7, 1.7 Hz, 1H), 6.26 (t, J = 3.6 Hz, 1H), 4.71 (t, J = 6.8 Hz, 1H), 3.20 (td, J = 6.9, 2.3 Hz, 2H), 2.45 (s, 3H), 2.02 (m, 1H), 1.67 (m, 1H), 1.55 (m, 1H), 1.40 (m, 1H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.1, 144.9, 139.2, 135.9, 133.2, 133.1, 130.7, 129.5, 128.8,

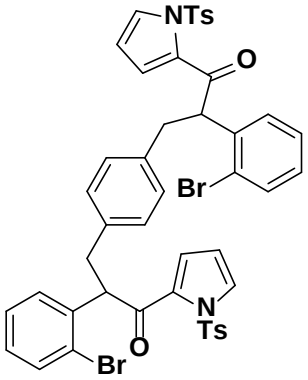
128.7, 128.4, 128.3, 124.4, 123.8, 110.6, 52.2, 51.4, 30.5, 26.6, 21.9 ppm; IR (neat, cm^{-1}) ν_{max} 2094, 1673, 1433, 1365, 1142, 1019; HRMS (ESI): *m/z* calcd for $C_{22}H_{22}{}^{81}BrN_4O_3S$ $[M+H]^+$: 503.0570; found 503.0583.

2,9-Bis(2-bromophenyl)-1,10-bis(1-tosyl-1*H*-pyrrol-2-yl)decane-1,10-dione (3f)



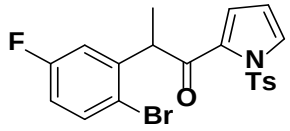
588 mg, 64% as a colorless solid; mp 135–136 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.84 (d, *J* = 7.7 Hz, 4H), 7.74 – 7.73 (m, 2H), 7.50 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 4H), 7.16 (dd, *J* = 3.6, 1.4 Hz, 2H), 7.08 (t, *J* = 7.0 Hz, 2H), 7.00 (td, *J* = 7.7, 1.6 Hz, 2H), 6.95 (d, *J* = 7.7 Hz, 2H), 6.25 (t, *J* = 3.4 Hz, 2H), 4.65 (t, *J* = 7.1 Hz, 2H), 2.42 (s, 6H), 1.93 –1.87 (m, 2H), 1.56 – 1.49 (m, 2H), 1.14 – 1.02 (m, 8H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.7, 144.7, 139.7, 136.0 133.5, 133.0, 130.5, 129.4, 128.8, 128.5, 128.4, 128.0, 124.5, 123.7, 110.5, 52.6, 33.5, 29.4, 27.2, 27.2, 21.8 ppm; IR (neat, cm^{-1}) ν_{max} 1676, 1433, 1364, 1171, 1142, 1018; HRMS (ESI): *m/z* calcd for $C_{44}H_{42}{}^{79}Br^{81}BrN_2O_6S_2Na$ $[M+Na]^+$: 941.0723; found 941.0718.

2-(2-Bromophenyl)-3-(4-(2-(2-bromophenyl)-3-oxo-3-(1-tosyl-1*H*-pyrrol-2-yl)propyl)phenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3g)



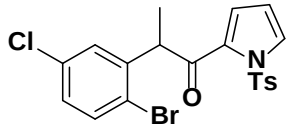
563 mg, 60 % as a colorless solid; mp 185–186 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.82 (dd, *J* = 8.3, 3.4 Hz, 4H), 7.70 – 7.69 (m, 2H), 7.43 (dd, *J* = 7.8, 2.1 Hz, 2H), 7.28 (d, *J* = 7.5 Hz, 4H), 7.13 – 7.06 (m, 4H), 7.03 – 7.00 (m, 4H), 6.74 (d, *J* = 2.4 Hz, 4H), 6.17 (t, *J* = 2.9 Hz, 2H), 4.86 (ddd, *J* = 8.6, 5.7, 3.2 Hz, 2H), 3.23 (dd, *J* = 13.5, 8.4 Hz, 2H), 2.69 (dt, *J* = 13.5, 5.4 Hz, 2H), 2.45 (s, 3H), 2.44 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 186.5, 186.5, 144.7, 144.7, 139.0, 138.9, 137.0, 136.9, 136.0, 135.9, 133.2, 133.1, 133.0, 130.7, 130.6, 129.4, 129.1, 128.8, 128.6, 128.4, 128.0, 127.9, 124.6, 124.5, 124.0, 123.9, 110.5, 110.4, 54.9, 54.8, 38.8, 38.7, 21.9 ppm; IR (neat, cm^{-1}) ν_{max} 1677, 1428, 1364, 1170, 1142, 1062; HRMS (ESI): *m/z* calcd for $C_{46}H_{38}{}^{79}Br^{81}BrN_2O_6S_2Na$ $[M+Na]^+$: 961.0410; found 961.0392.

2-(2-Bromo-5-fluorophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3h)



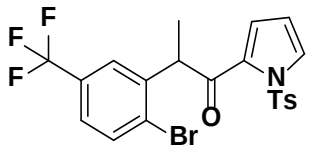
432 mg, 96% as a colorless solid; mp 108–109 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.84 (d, J = 8.4 Hz, 2H), 7.78 (dd, J = 3.2, 1.7 Hz, 1H), 7.47 (dd, J = 8.8, 5.4 Hz, 1H), 7.32 (d, J = 8.1 Hz, 2H), 7.08 (dd, J = 3.8, 1.7 Hz, 1H), 6.76 (m, 1H), 6.48 (dd, J = 9.6, 3.0 Hz, 1H), 6.27 (t, J = 3.6 Hz, 1H), 4.73 (q, J = 7.6, 6.8 Hz, 1H), 2.44 (s, 3H), 1.30 (d, J = 6.8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.4, 162.2 (d, $J_{F\text{-}C}$ = 246.3 Hz), 145.2, 143.2 (d, $J_{F\text{-}C}$ = 7.6 Hz), 135.6, 134.2 (d, $J_{F\text{-}C}$ = 8.1 Hz), 132.5, 130.8, 129.5, 128.3, 123.9, 117.7 (d, $J_{F\text{-}C}$ = 3.2 Hz), 115.9 (d, $J_{F\text{-}C}$ = 22.4 Hz), 115.4 (d, $J_{F\text{-}C}$ = 23.3 Hz), 110.6, 47.6 (d, $J_{F\text{-}C}$ = 1.7 Hz), 21.8, 17.7 ppm; IR (neat, cm^{-1}) ν_{max} 1672, 1246, 1176, 1140, 1043; HRMS (ESI): *m/z* calcd for $C_{20}H_{17}{}^{79}BrFNO_3SNa$ $[M+Na]^+$: 471.9989; found 471.9988.

2-(2-Bromo-5-chlorophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3i)



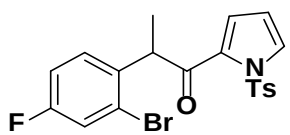
438 mg, 94% as a colorless solid; mp 162–163 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.86 (d, J = 8.4 Hz, 2H), 7.78 (dd, J = 3.2, 1.7 Hz, 1H), 7.45 (d, J = 8.5 Hz, 1H), 7.33 (d, J = 8.1 Hz, 2H), 7.06 (dd, J = 3.8, 1.7 Hz, 1H), 7.02 (dd, J = 8.5, 2.5 Hz, 1H), 6.85 (d, J = 2.5 Hz, 1H), 6.27 (t, J = 3.6 Hz, 1H), 4.72 (q, J = 6.8 Hz, 1H), 2.44 (s, 3H), 1.31 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.4, 145.0, 143.0, 135.8, 134.1, 134.1, 132.5, 130.8, 129.6, 128.8, 128.5, 128.3, 123.9, 121.5, 110.6, 47.6, 21. 9, 17.8 ppm; IR (neat, cm^{-1}) ν_{max} 1669, 1366, 1171, 1142, 1085, 1065, 1022; HRMS (ESI): *m/z* calcd for $C_{20}H_{17}{}^{81}Br{}^{35}ClNO_3SNa$ $[M+Na]^+$: 489.9673; found 489.9663.

2-(2-Bromo-5-(trifluoromethyl)phenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3j)



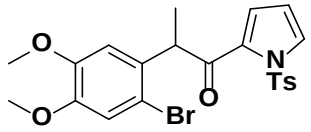
460 mg, 92% as a colorless solid; mp 134–135 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.87 (d, J = 8.3 Hz, 2H), 7.78 (dd, J = 3.0, 1.7 Hz, 1H), 7.65 (d, J = 8.3 Hz, 1H), 7.32 – 7.27 (m, 3H), 7.24 (m, 1H), 7.08 (dd, J = 3.8, 1.6 Hz, 1H), 6.27 (t, J = 3.5 Hz, 1H), 4.83 (q, J = 6.9 Hz, 1H), 2.43 (s, 3H), 1.36 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 187.4, 145.1, 142.2, 135.8, 133.7, 132.5, 130.9, 130.6 (q, $J_{F\text{-}C}$ = 32.7 Hz), 129.5, 128.3, 125.4 (q, $J_{F\text{-}C}$ = 3.8 Hz), 125.3 (q, $J_{F\text{-}C}$ = 3.6 Hz), 123.8, 123.6 (q, $J_{F\text{-}C}$ = 270.9 Hz), 110.6, 47.5, 21. 8, 18.0 ppm; IR (neat, cm^{-1}) ν_{max} 1680, 1369, 1329, 1162, 1132, 1083, 1067, 1023; HRMS (ESI): *m/z* calcd for $C_{21}H_{18}{}^{81}BrF_3NO_3S$ $[M+H]^+$: 502.0117; found 502.0120.

2-(2-Bromo-4-fluorophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3k)



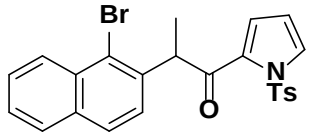
423 mg, 94% as a colorless solid; mp 150–151 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.89 (d, J = 8.4 Hz, 2H), 7.73 (dd, J = 3.2, 1.7 Hz, 1H), 7.33 (d, J = 8.1 Hz, 2H), 7.27 (m, 1H), 7.03 (dd, J = 3.8, 1.7 Hz, 1H), 6.94 (dd, J = 8.7, 6.0 Hz, 1H), 6.86 (td, J = 8.3, 2.6 Hz, 1H), 6.25 (t, J = 3.6 Hz, 1H), 4.72 (q, J = 6.8 Hz, 1H), 2.45 (s, 3H), 1.30 (d, J = 6.8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 188.2, 161.2 (d, J_{F-C} = 248.8 Hz), 144.9, 137.2 (d, J_{F-C} = 3.6 Hz), 136.0, 132.8, 130.4, 129.5 (d, J_{F-C} = 8.4 Hz), 129.5, 128.4, 123.6 (d, J_{F-C} = 9.3 Hz), 123.4, 120.2 (d, J_{F-C} = 24.2 Hz), 115.5 (d, J_{F-C} = 20.8 Hz), 110.6, 47.0, 21.8, 17.8 ppm; IR (neat, cm^{-1}) ν_{max} 1673, 1487, 1365, 1175, 1141, 1026; HRMS (ESI): *m/z* calcd for $C_{20}H_{17}{}^{81}BrFNO_3SNa$ $[M+Na]^+$: 573.9968; found 573.9970.

2-(2-Bromo-4,5-dimethoxyphenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3l)



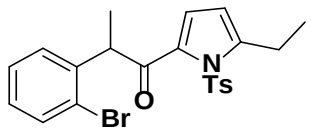
463 mg, 94% as a colorless solid; mp 168–169 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.94 (d, J = 8.4 Hz, 2H), 7.68 (dd, J = 3.2, 1.7 Hz, 1H), 7.33 (d, J = 8.1 Hz, 2H), 7.04 (dd, J = 3.8, 1.7 Hz, 1H), 6.98 (s, 1H), 6.60 (s, 1H), 6.23 (t, J = 3.6 Hz, 1H), 4.67 (q, J = 6.8 Hz, 1H), 3.82 (s, 3H), 3.75 (s, 3H), 2.43 (s, 3H), 1.31 (d, J = 6.8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 189.0, 149.1, 148.5, 144.8, 136.4, 133.4, 133.1, 130.0, 129.5, 128.3, 123.1, 115.5, 113.1, 110.7, 110.6, 56.2, 56.0, 47.6, 21.8, 17.4 ppm; IR (neat, cm^{-1}) ν_{max} 1668, 1435, 1362, 1247, 1165, 1143, 1003; HRMS (ESI): *m/z* calcd for $C_{22}H_{22}{}^{81}BrNO_5SNa$ $[M+Na]^+$: 516.0274; found 516.0275.

2-(1-Bromonaphthalen-2-yl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3m)



443 mg, 92% as a colorless solid; mp 182–183 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.31 (d, J = 8.5 Hz, 1H), 7.90 (d, J = 8.3 Hz, 2H), 7.74 (d, J = 7.6 Hz, 1H), 7.69 (dd, J = 3.0, 1.7 Hz, 1H), 7.61 – 7.56 (m, 2H), 7.48 (t, J = 7.4 Hz, 1H), 7.33 (d, J = 8.2 Hz, 2H), 7.07 (dd, J = 3.8, 1.6 Hz, 1H), 7.00 (d, J = 8.5 Hz, 1H), 6.17 (t, J = 3.5 Hz, 1H), 5.09 (q, J = 6.8 Hz, 1H), 2.45 (s, 3H), 1.39 (d, J = 6.8 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 188.4, 144.8, 139.7, 136.1, 133.6, 132.9, 132.7, 130.3, 129.5, 128.6, 128.4, 128.3, 127.8, 127.7, 126.8, 125.2, 123.8, 123.3, 110.6, 49.1, 21.9, 17.3 ppm; IR (neat, cm^{-1}) ν_{max} 1672, 1362, 1170, 1146, 1030; HRMS (ESI): *m/z* calcd for $C_{24}H_{20}{}^{81}BrNO_3SNa$ $[M+Na]^+$: 506.0219; found 506.0207.

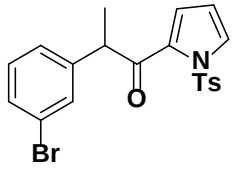
2-(2-Bromophenyl)-1-(5-ethyl-1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (3n)



432 mg, 94% as a colorless solid; mp 104–105 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.95 (d, J = 8.3 Hz, 2H), 7.54 (dd, J = 8.0, 1.1 Hz, 1H), 7.32 – 7.27 (m, 3H), 7.23 (td, J = 7.6, 1.1 Hz, 1H), 7.06 (td, J = 7.6,

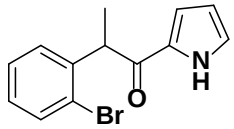
1.8 Hz, 1H), 6.88 (d, *J* = 3.7 Hz, 1H), 5.97 (d, *J* = 3.8 Hz, 1H), 4.85 (q, *J* = 6.8 Hz, 1H), 2.89 (q, *J* = 7.4 Hz, 2H), 2.42 (s, 3H), 1.42 (d, *J* = 6.9 Hz, 3H), 1.19 (t, *J* = 7.4 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 190.0, 147.4, 144.7, 141.1, 137.1, 135.8, 132.9, 129.7, 129.4, 128.5, 128.2, 127.7, 124.2, 121.7, 110.4, 48.8, 22.3, 21.8, 17.9, 13.0 ppm; IR (neat, cm^{-1}) ν_{max} 1673, 1362, 1174, 1146, 1067; HRMS (ESI): *m/z* calcd for $C_{24}H_{20}{}^{79}BrNO_3SNa$ $[M+Na]^+$: 504.0239; found 504.0236.

2-(3-Bromophenyl)-1-(1-tosyl-1*H*-pyrrol-2-yl)propan-1-one (9b)



406 mg, 94% as a colorless solid; mp 108–109 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.84 (d, *J* = 8.4 Hz, 2H), 7.75 (dd, *J* = 3.2, 1.7 Hz, 1H), 7.31 – 7.29 (m, 3H), 7.21 (t, *J* = 1.8 Hz, 1H), 7.09 (t, *J* = 7.8 Hz, 1H), 7.02 (m, 1H), 6.96 (dd, *J* = 3.8, 1.7 Hz, 1H), 6.27 (t, *J* = 3.6 Hz 1H), 4.24 (q, *J* = 6.9 Hz, 1H), 2.43 (s, 3H), 1.37 (d, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 188.0, 144.9, 143.8, 135.8, 132.7, 130.5, 130.5, 130.4, 130.1, 129.5, 128.3, 126.2, 123.8, 122.8, 110.5, 48.5, 21.9, 19.0 ppm; IR (neat, cm^{-1}) ν_{max} 1673, 1357, 1168, 1147, 1066; HRMS (ESI): *m/z* calcd for $C_{20}H_{18}{}^{81}BrNO_3SNa$ $[M+Na]^+$: 456.0063; found 456.0057.

2-(2-Bromophenyl)-1-(1*H*-pyrrol-2-yl)propan-1-one (8)



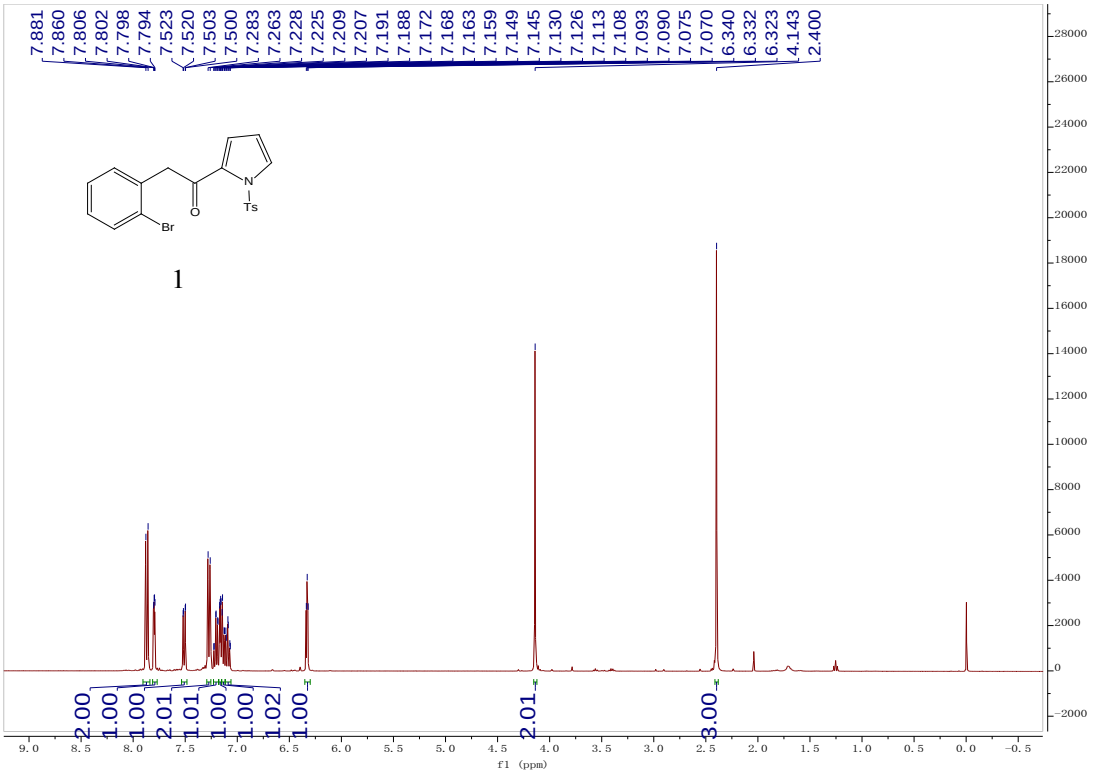
A mixture of **1** (418 mg, 1 mmol), NaOH (639 mg, 16 mmol) and CH_3OH (10 mL) was stirred at 70 °C for 10 h, before being cooled and concentrated in vacuo. The residue was partitioned between EtOAc (20 mL) and H_2O (10 mL). The separated aqueous phase was extracted with EtOAc (2 × 20 mL), and then dried (Na_2SO_4), filtered, and evaporated *in vacuo*. The residue was purified by column chromatography on silica gel (3% EtOAc in petroleum ether) to afford **8** (261 mg, 94%) as a colorless solid; mp 115–116 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 9.64 (s, 1H), 7.57 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.34 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.22 (td, *J* = 7.6, 1.1 Hz, 1H), 7.06 (td, *J* = 7.9, 1.7 Hz, 1H), 6.98 (m, 1H), 6.96 (m, 1H), 6.21 (dt, *J* = 3.8, 2.5 Hz, 1H), 4.93 (q, *J* = 6.9 Hz, 1H), 1.48 (d, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 190.4, 141.5, 133.2, 131.4, 128.8, 128.5, 128.1, 124.9, 124.0, 117.0, 111.0, 46.5, 18.2 ppm; IR (neat, cm^{-1}) ν_{max} 3265, 1630, 1287, 1109, 1045, 1016; HRMS (ESI): *m/z* calcd for $C_{13}H_{13}{}^{79}BrNO$ $[M+H]^+$: 278.0175; found 278.0172.

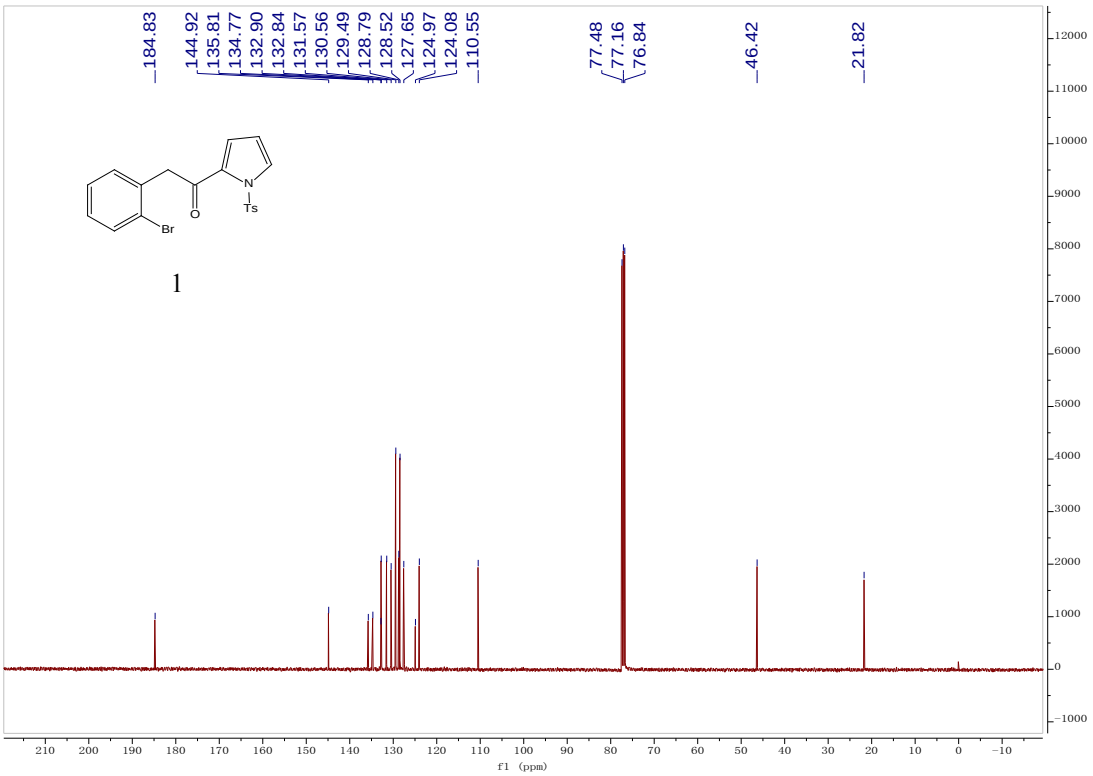
3. References:

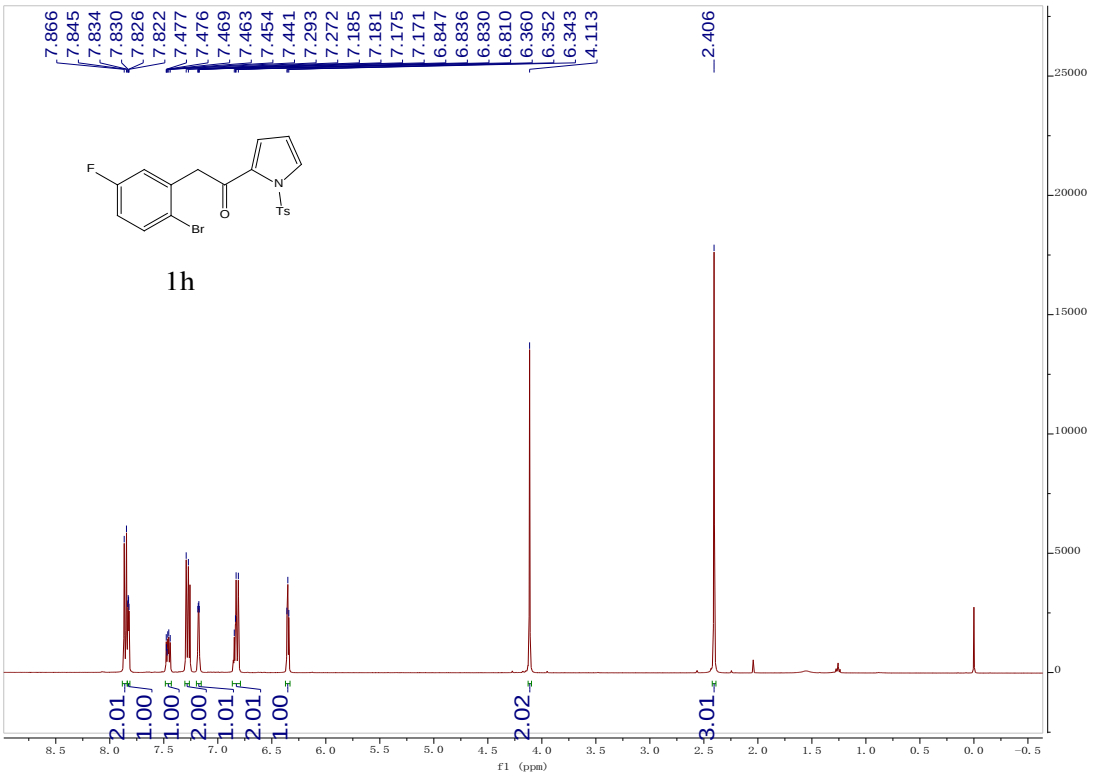
[1] C. Song, D. W. Knight and M. A. Whatton, *Tetrahedron Lett.*, 2004, **45**, 9573–9576.

[2] M. L. McIntosh, C. M. Moore and T. B. Clark, *Org. Lett.*, 2010, **12**, 1996–1999.

4. ^{1}H and ^{13}C NMR spectra



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7.454
7.441
7.293
7.272
7.185
7.181
7.175
7.171
6.847
6.836
6.830
6.810
6.360
6.352
6.343
4.113
2.406
F
Br
N
Ts
O
1h
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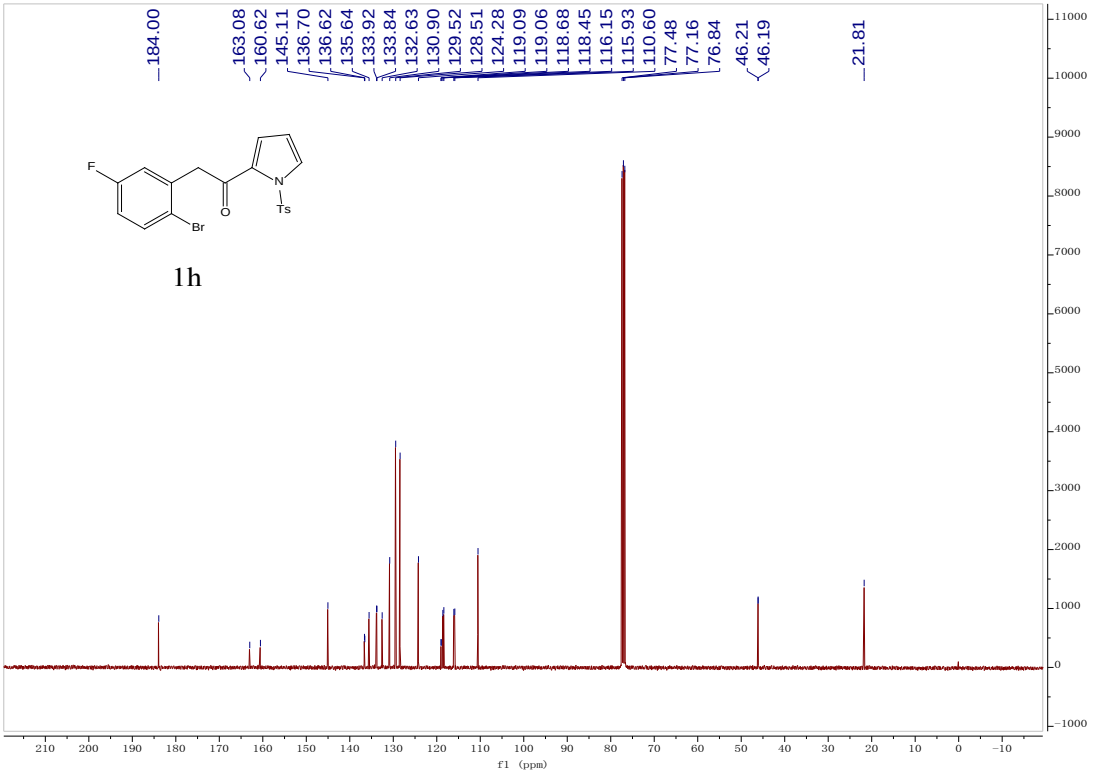
1.00
1.00
2.00
1.01
2.01
1.00
2.02
3.01
f1 (ppm)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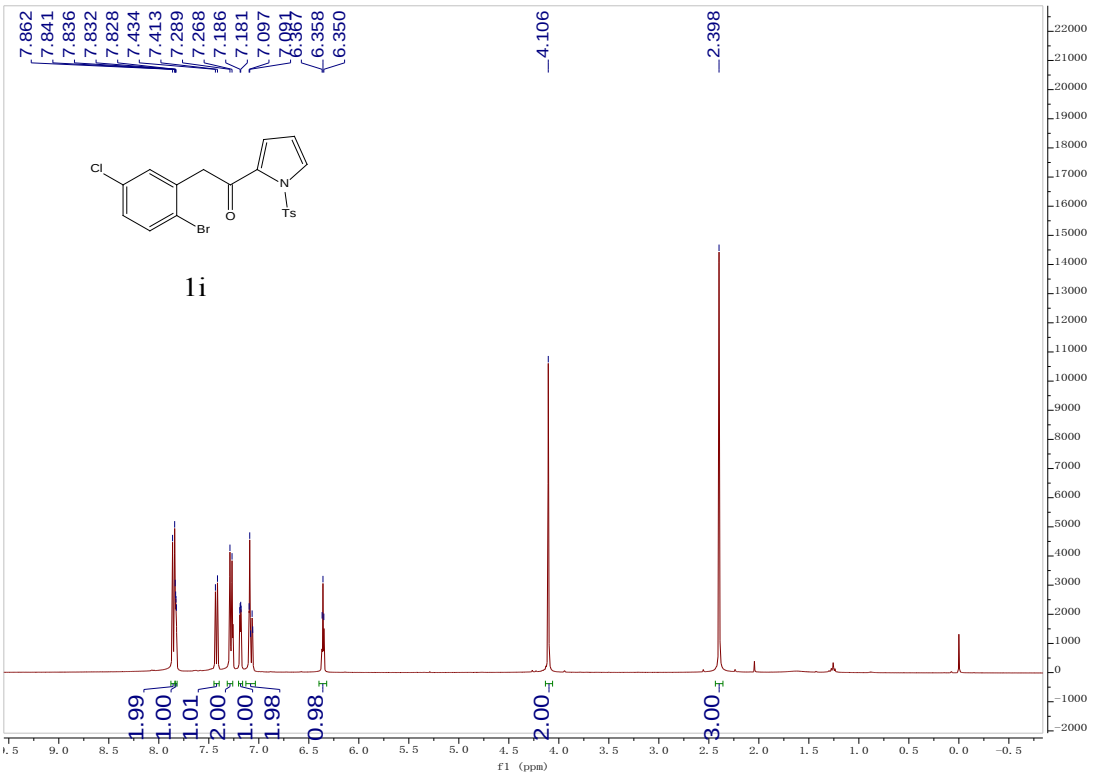
160.62
145.11
136.70
136.62
135.64
133.92
133.84
132.63
130.90
129.52
128.51
124.28
119.09
119.06
118.68
118.45
116.15
115.93
110.60
77.48
77.16
76.84
46.21
46.19
21.81
F
Br
N
Ts
O
1h
f1 (ppm)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7.434
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7.181
7.097
7.091
6.367
6.358
6.350
4.106
2.398
Cl
Br
O
N
Ts
1i
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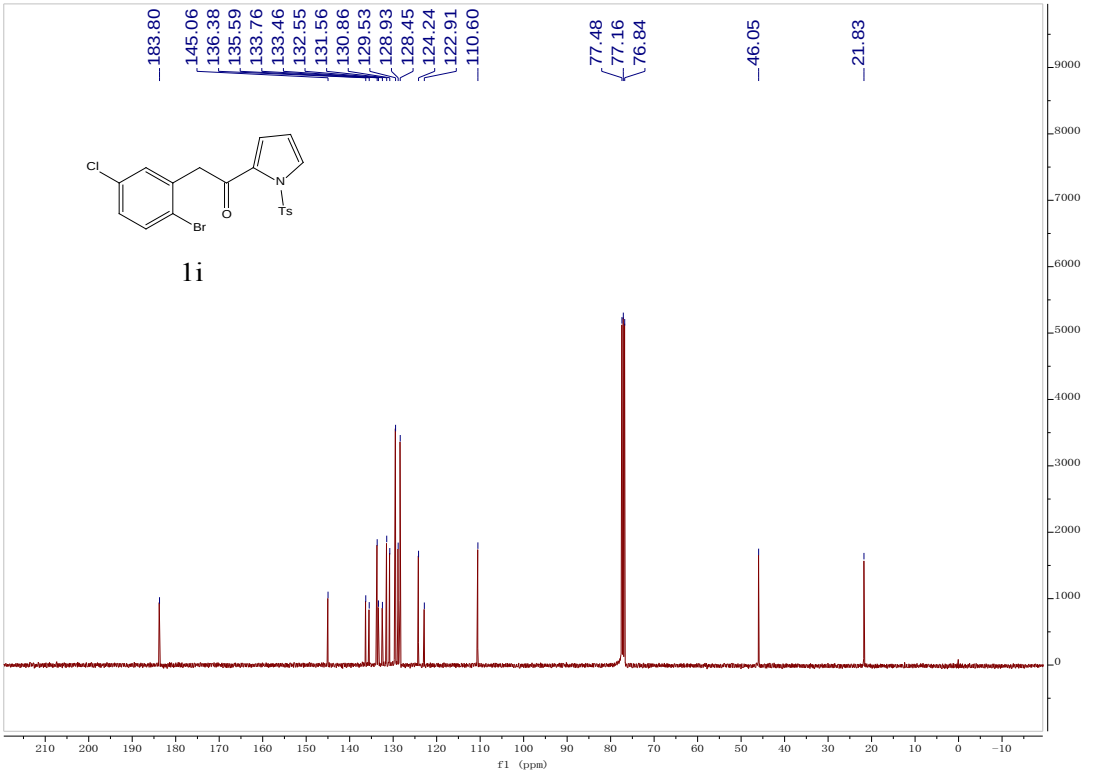
1.00
1.01
2.00
1.00
1.98
0.98
2.00
3.00
f1 (ppm)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131.56
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128.93
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124.24
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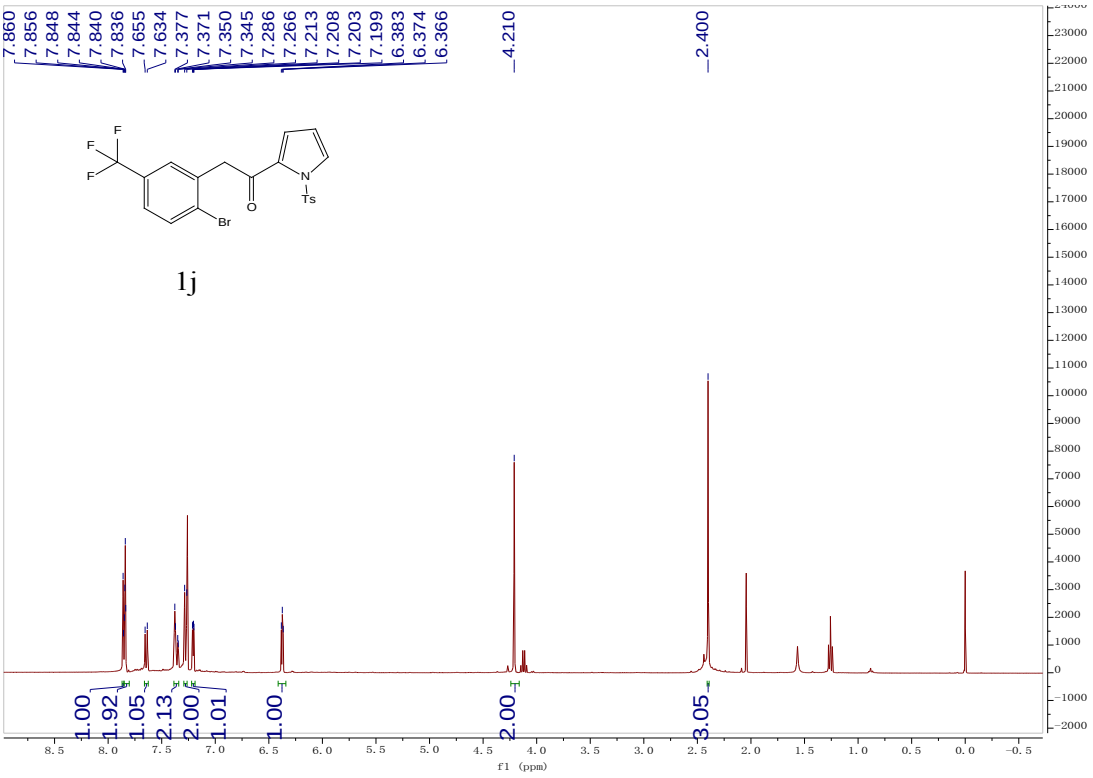
77.16
76.84
46.05
21.83
Cl
Br
O
N
Ts
1i
f1 (ppm)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7.345
7.286
7.266
7.213
7.208
7.203
7.199
6.383
6.374
6.366
4.210
2.400
F
F
F
N
Ts
O
Br
1j
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1.92
1.05
2.13
2.00
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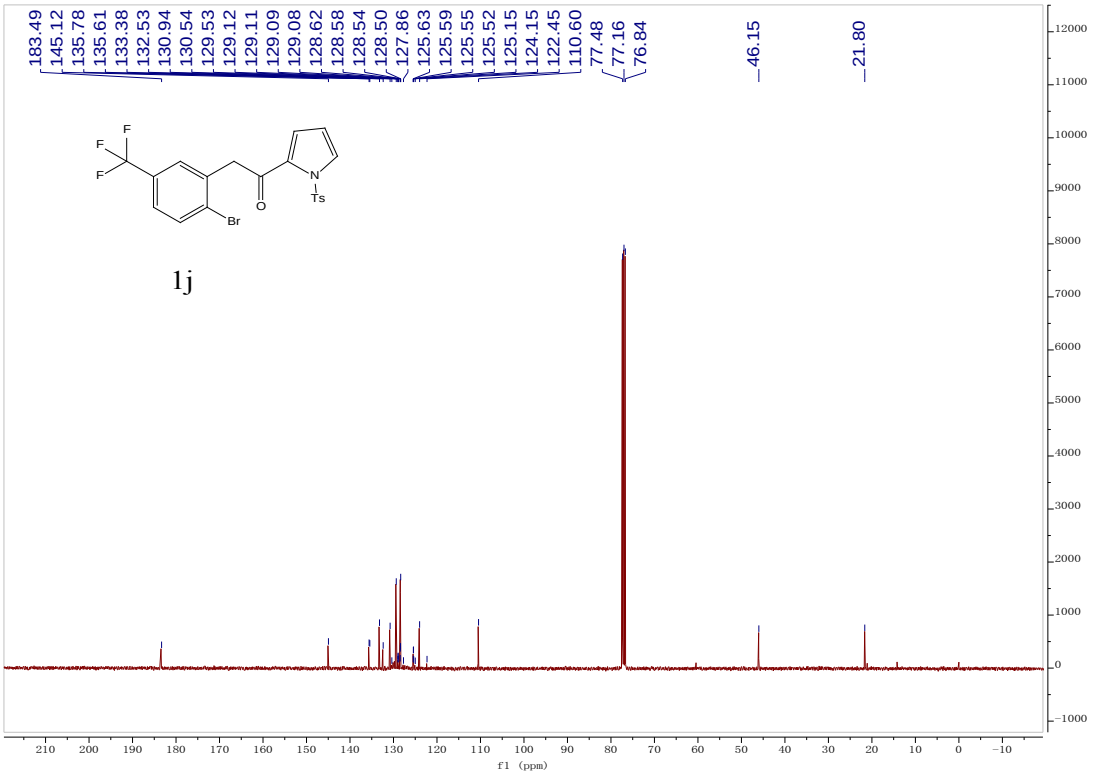
1.00
2.00
3.05
f1 (ppm)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128.62
128.58
128.54
128.50
127.86
125.63
125.59
125.55
125.52
125.15
124.15
122.45
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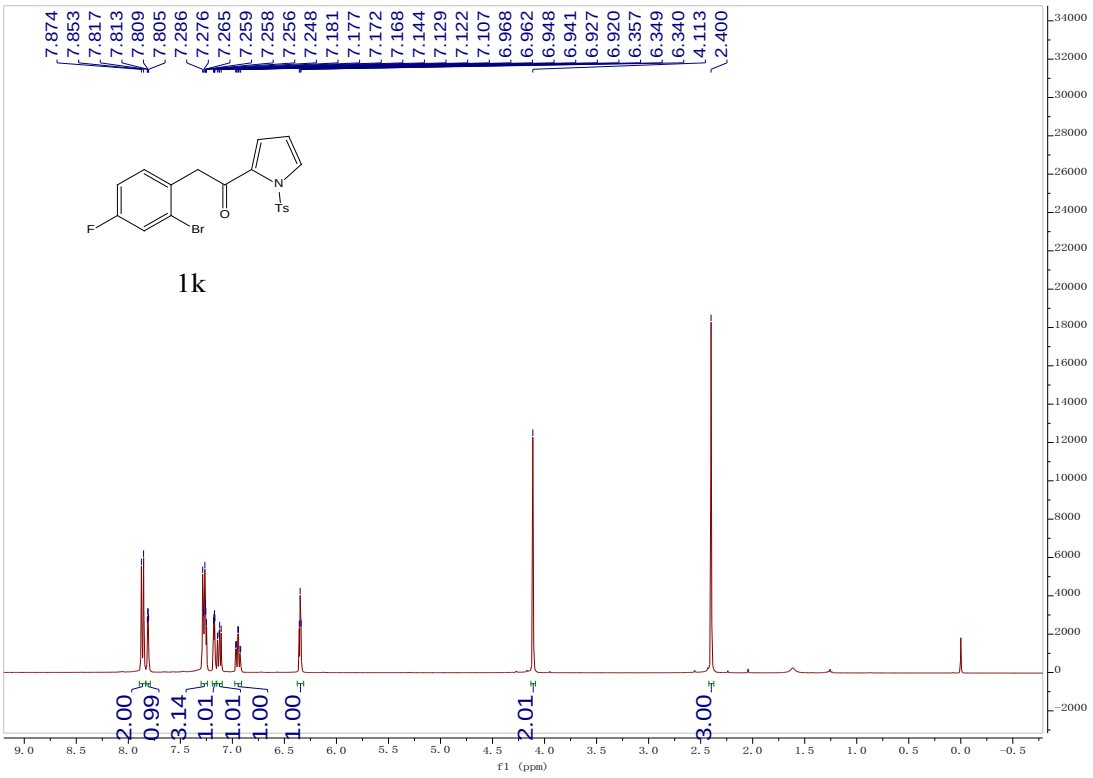
76.84
46.15
21.80
F
F
F
N
Ts
O
Br
1j
f1 (ppm)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7.276
7.265
7.259
7.258
7.256
7.248
7.181
7.177
7.172
7.168
7.144
7.129
7.122
7.107
6.968
6.962
6.948
6.941
6.927
6.920
6.357
6.349
6.340
4.113
2.400
F
Br
O
N
Ts
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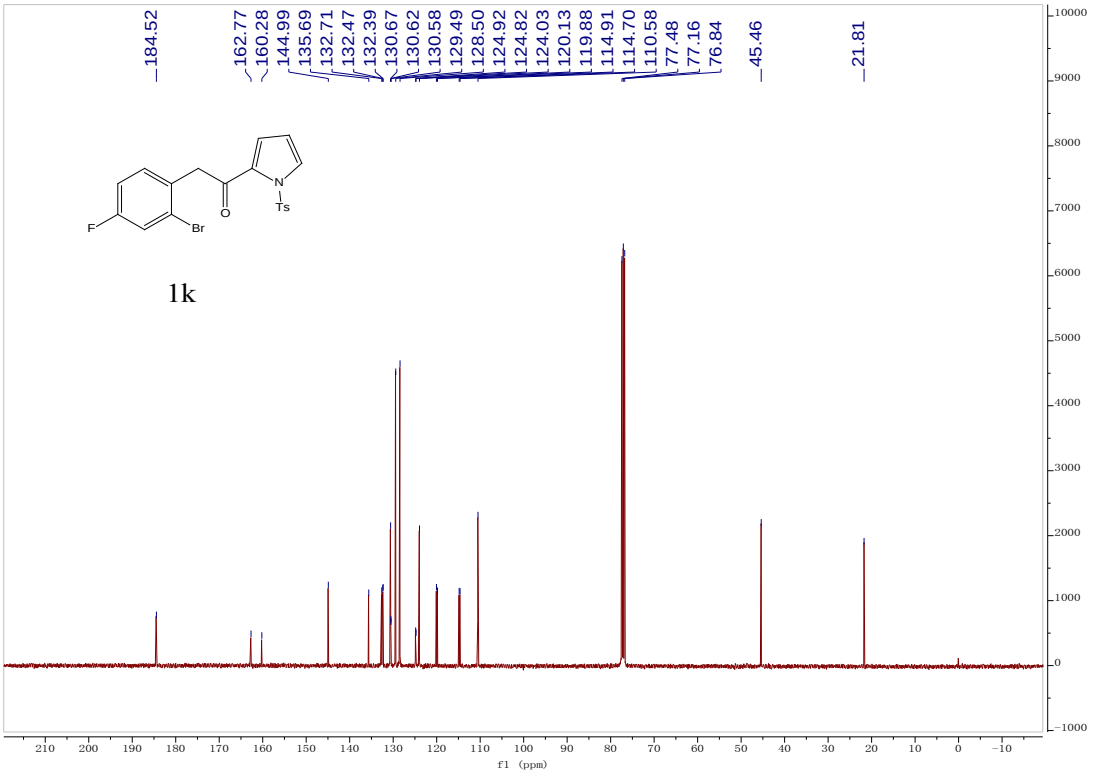
3.14
1.01
1.01
1.00
1.00
2.01
3.00
f1 (ppm)

1k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130.62
130.58
129.49
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124.82
124.03
120.13
119.88
114.91
114.70
110.58
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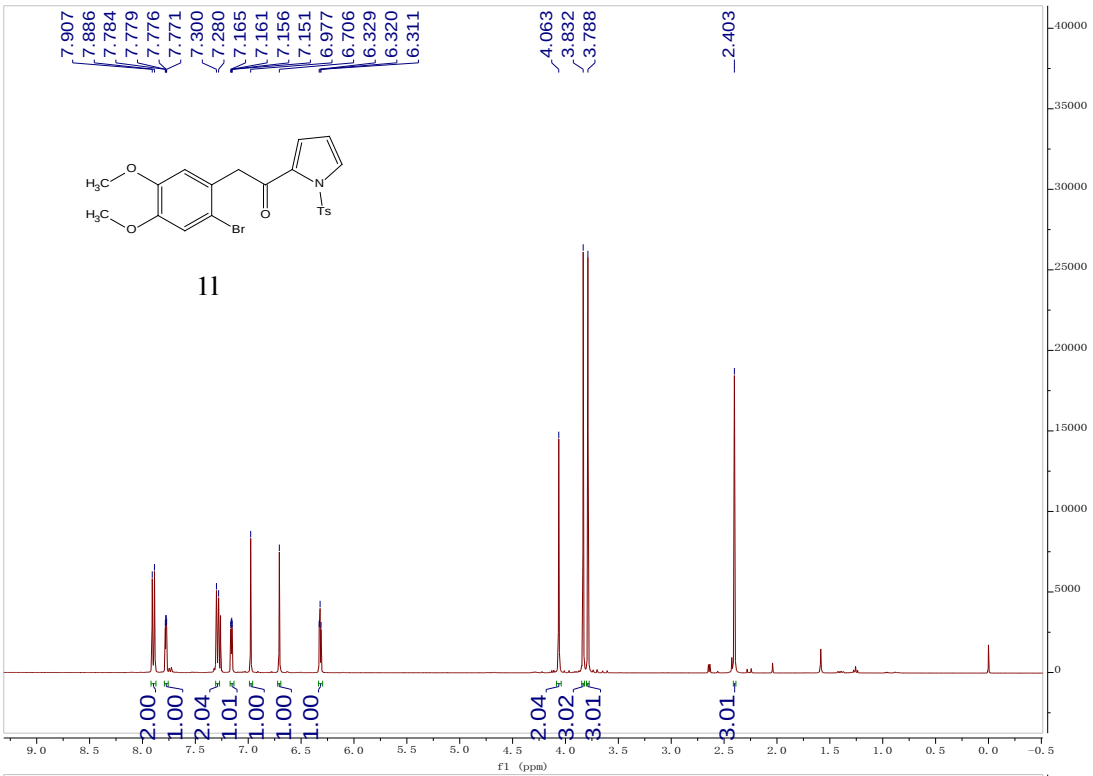
77.16
76.84
45.46
21.81
F
Br
O
N
Ts
f1 (ppm)

1k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7.280
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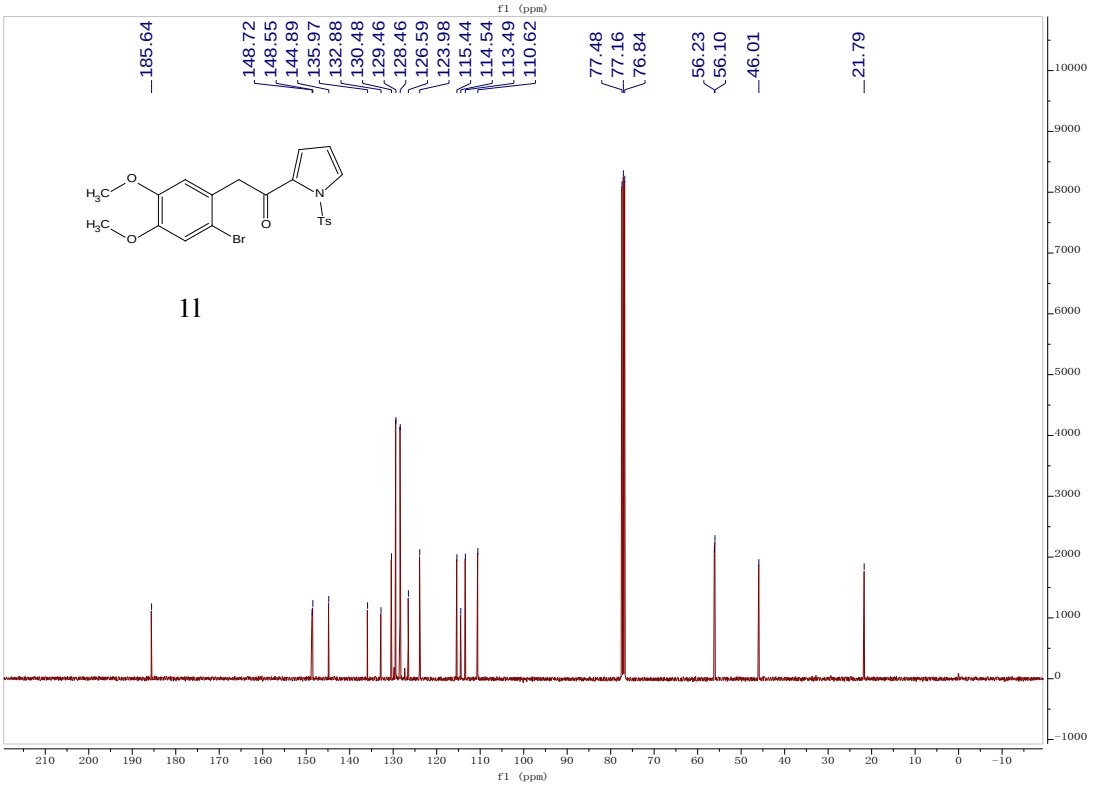
7.161
7.156
7.151
6.977
6.706
6.329
6.320
6.311
4.063
3.832
3.788
2.403
2.00
1.00
2.04
1.01
1.00
1.00
1.00
2.04
3.02
3.01
3.01
f1 (ppm)

11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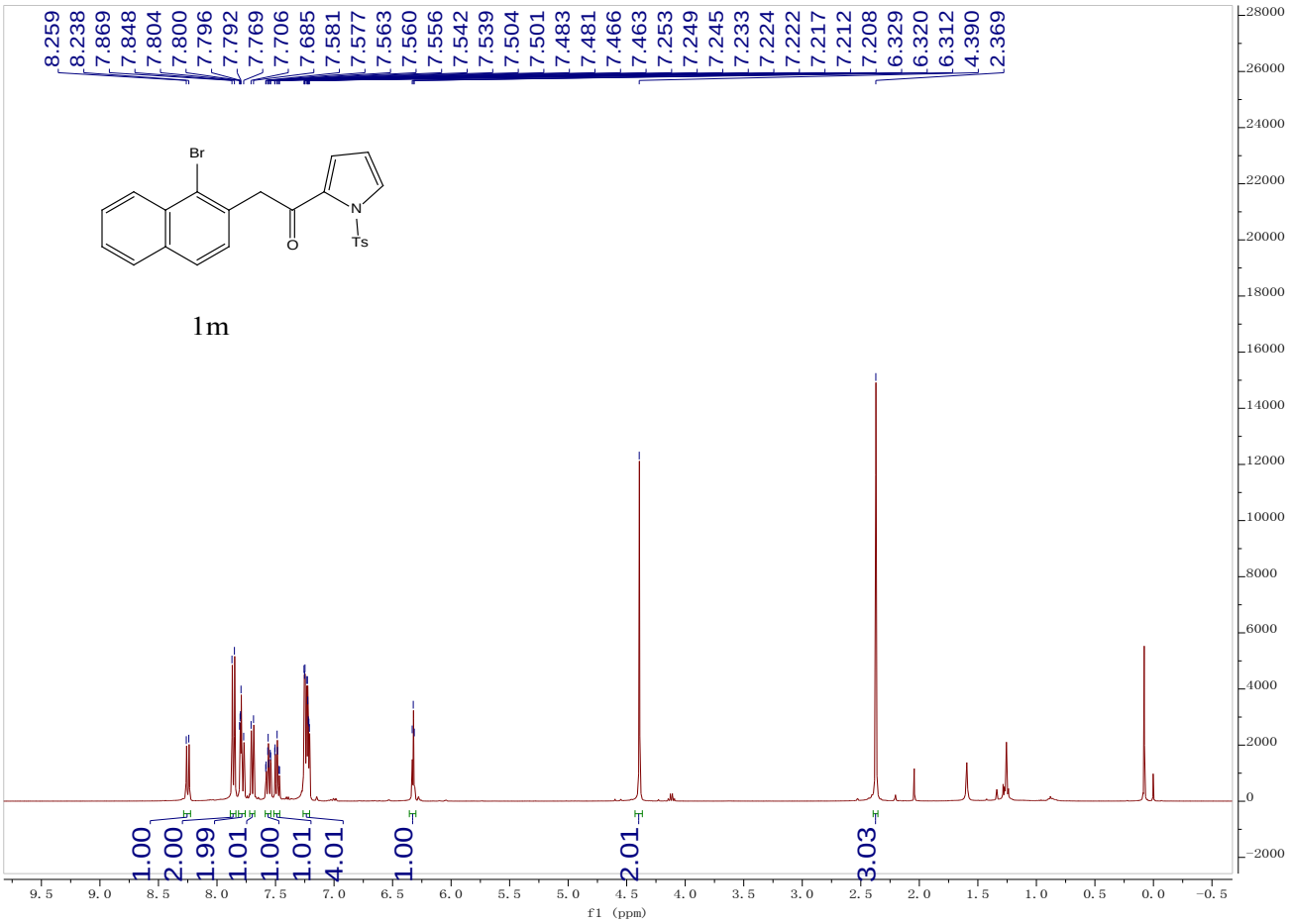
148.55
144.89
135.97
132.88
130.48
129.46
128.46
126.59
123.98
115.44
114.54
113.49
110.62
77.48
77.16
76.84
56.23
56.10
46.01
21.79
f1 (ppm)

11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7.792
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7.706
7.685
7.581
7.577
7.563
7.560
7.556
7.542
7.539
7.504
7.501
7.483
7.481
7.466
7.463
7.253
7.249
7.245
7.233
7.224
7.222
7.217
7.212
7.208
6.329
6.320
6.312
4.390
2.369
Br
N
Ts
O
1m
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1.99
1.01
1.00
1.01
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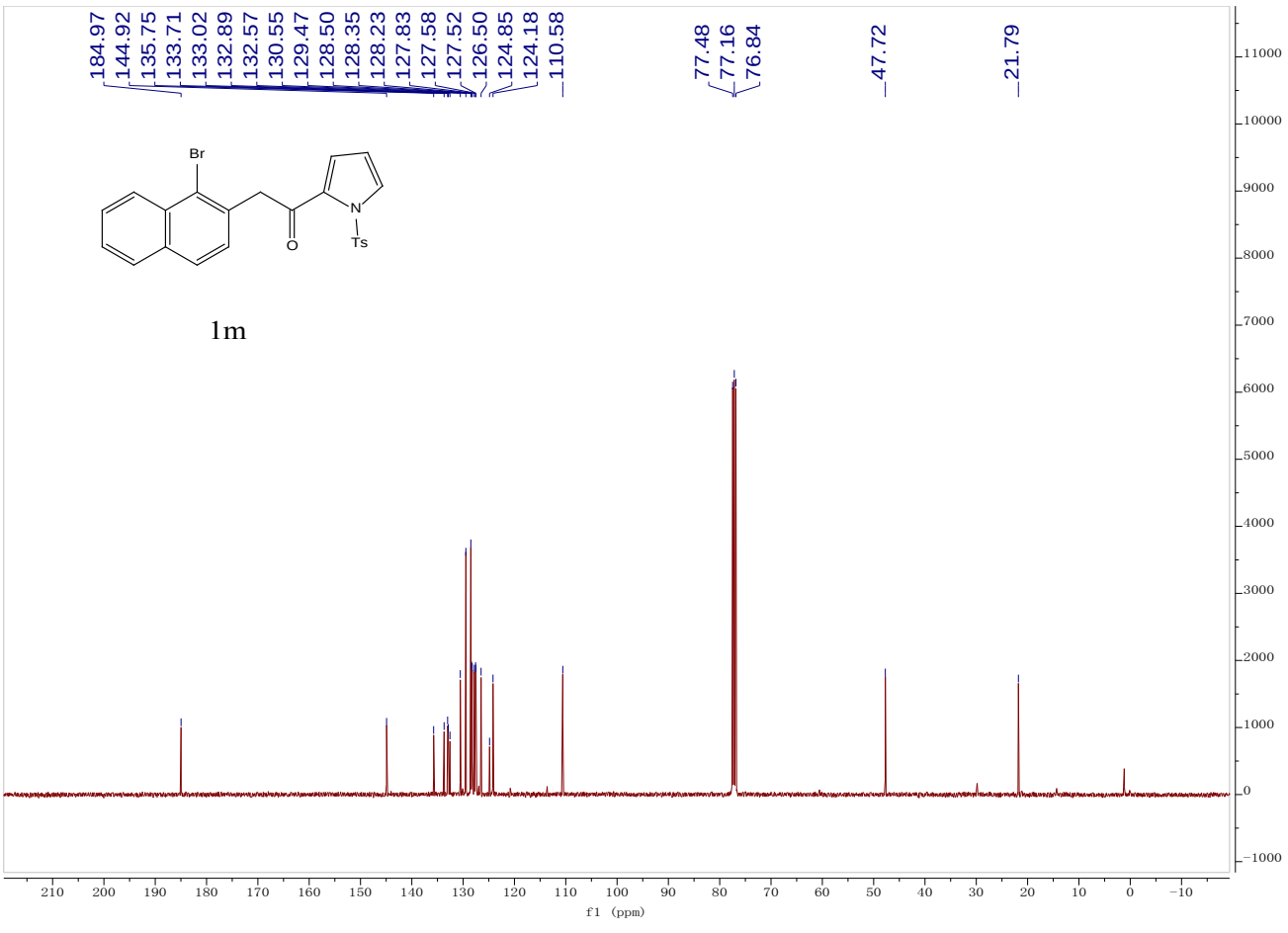
1.00
2.01
3.03
f1 (ppm)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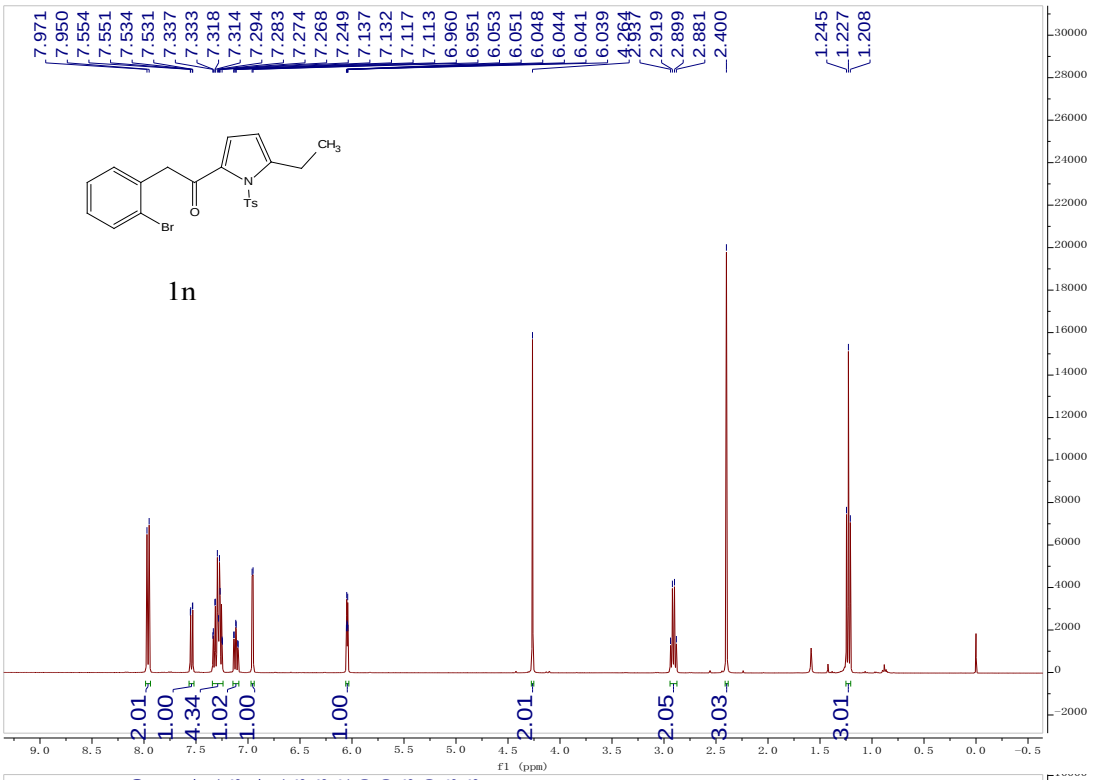
128.50
128.35
128.23
127.83
127.58
127.52
126.50
124.85
124.18
110.58
77.48
77.16
76.84
47.72
21.79
Br
N
Ts
O
1m
f1 (ppm)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7.314
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7.283
7.274
7.268
7.249
7.137
7.132
7.117
7.113
6.960
6.951
6.053
6.051
6.048
6.044
6.041
6.039
4.264
4.257
2.919
2.899
2.881
2.400
1.245
1.227
1.208
CH3
N
Ts
O
Br
1n
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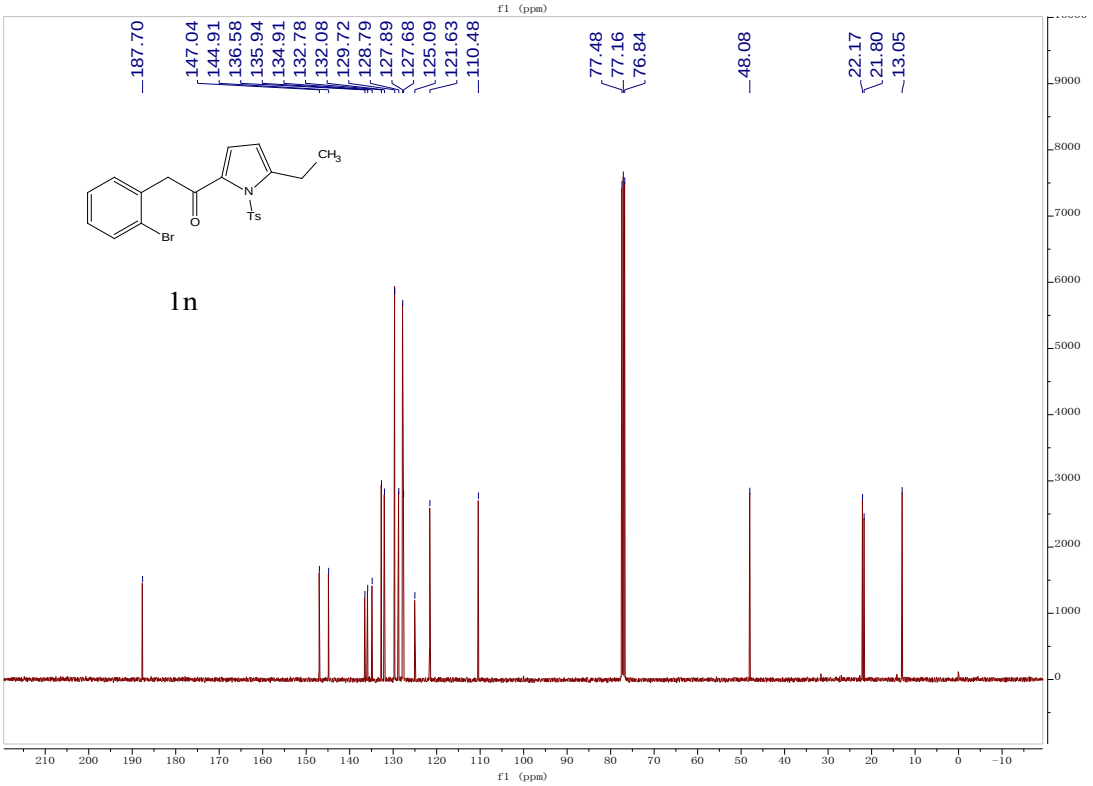
1.00
4.34
1.02
1.00
1.00
2.01
2.05
3.03
3.01
f1 (ppm)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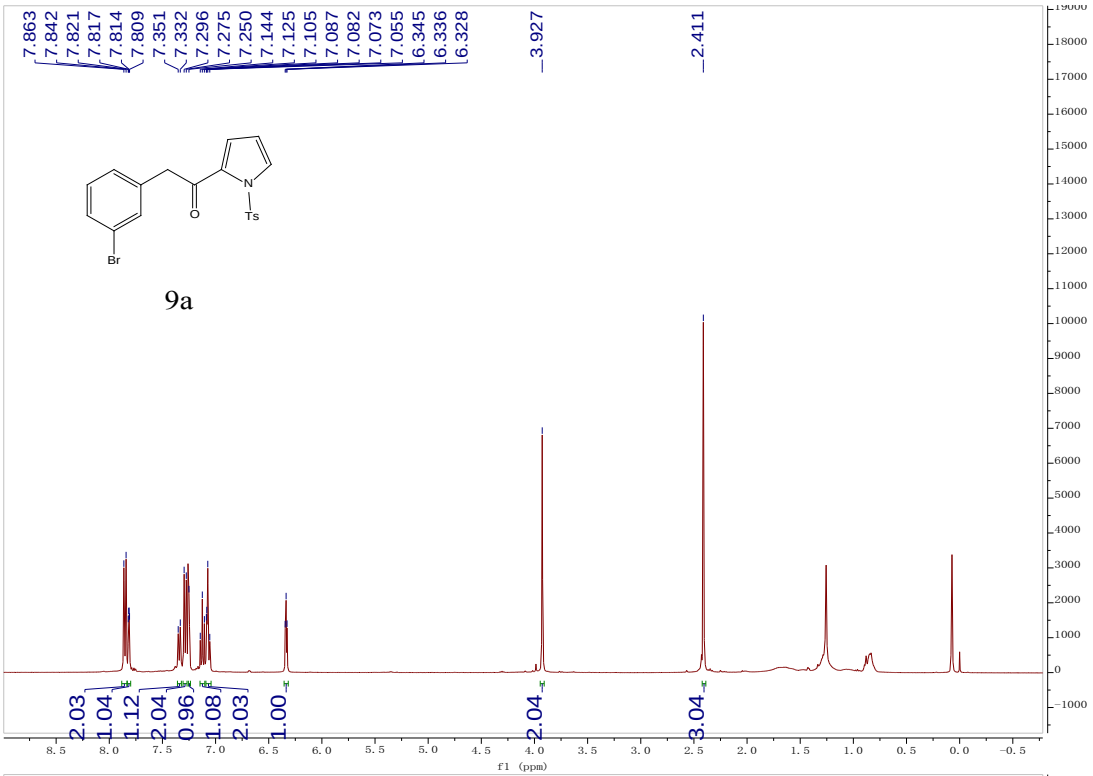
132.08
129.72
128.79
127.89
127.68
125.09
121.63
110.48
77.48
77.16
76.84
48.08
22.17
21.80
13.05
CH3
N
Ts
O
Br
1n
f1 (ppm)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7.125
7.105
7.087
7.082
7.073
7.055
6.345
6.336
6.328
3.927
2.411
N
O
Ts
Br
9a
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1.12
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1.00
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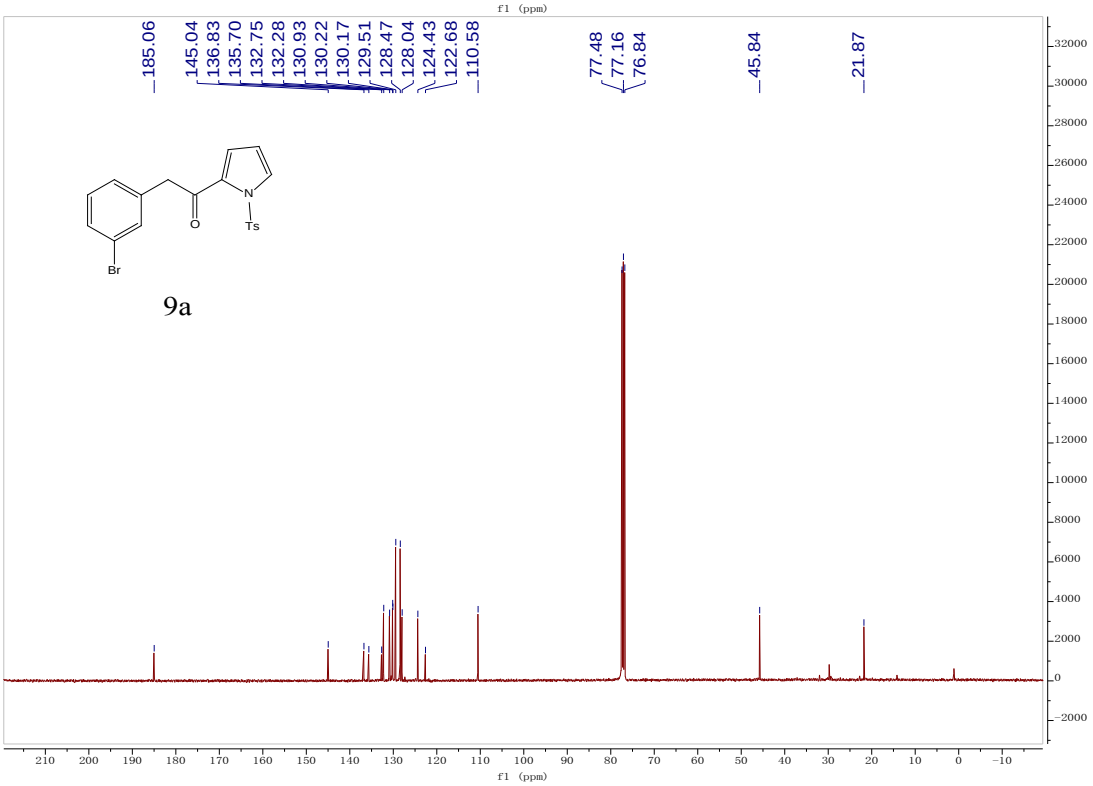
f1 (ppm)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129.51
128.47
128.04
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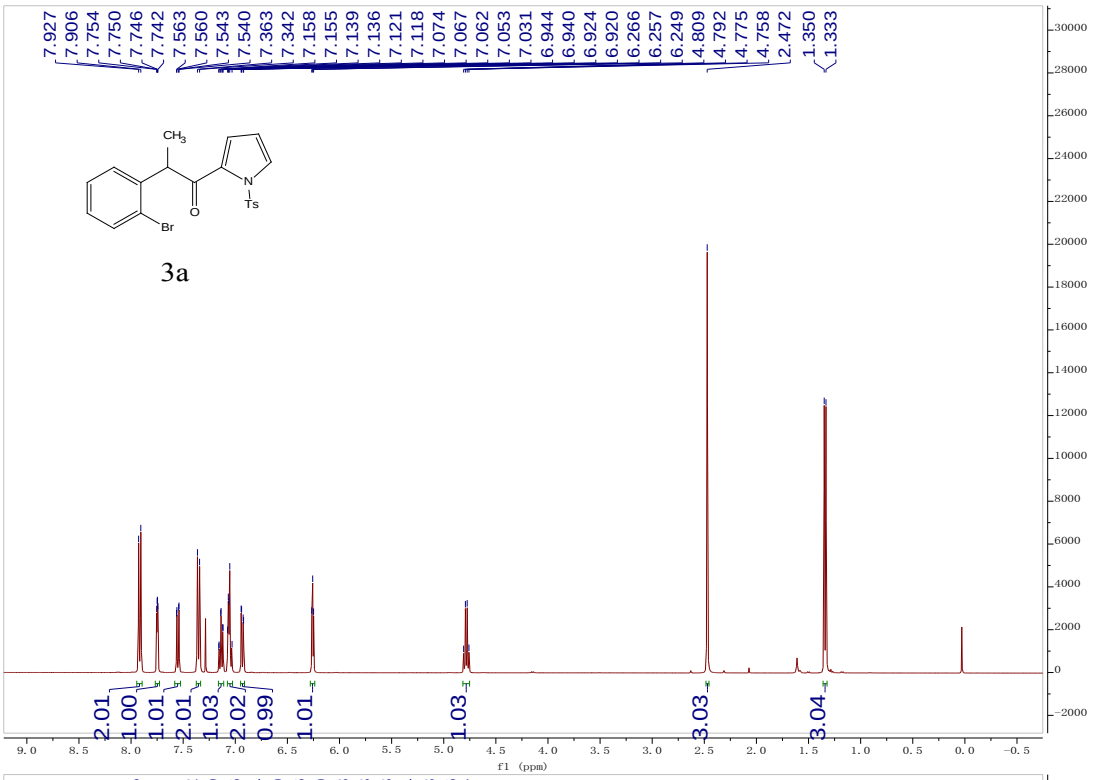
77.16
76.84
45.84
21.87
N
O
Ts
Br
9a
f1 (ppm)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7.155
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7.121
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6.920
6.266
6.257
6.249
4.809
4.792
4.775
4.758
2.472
1.350
1.333
CH3
N
Ts
O
Br
3a
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2.01
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2.02
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1.03
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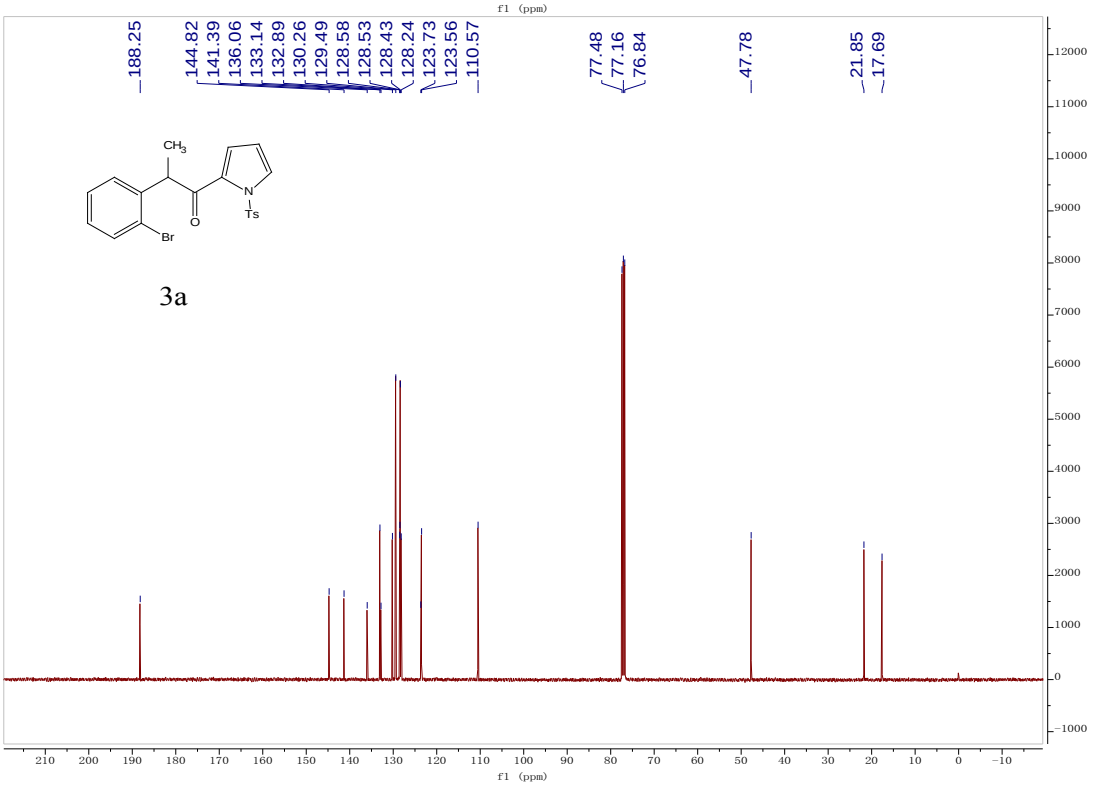
3.04
f1 (ppm)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128.53
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128.24
123.73
123.56
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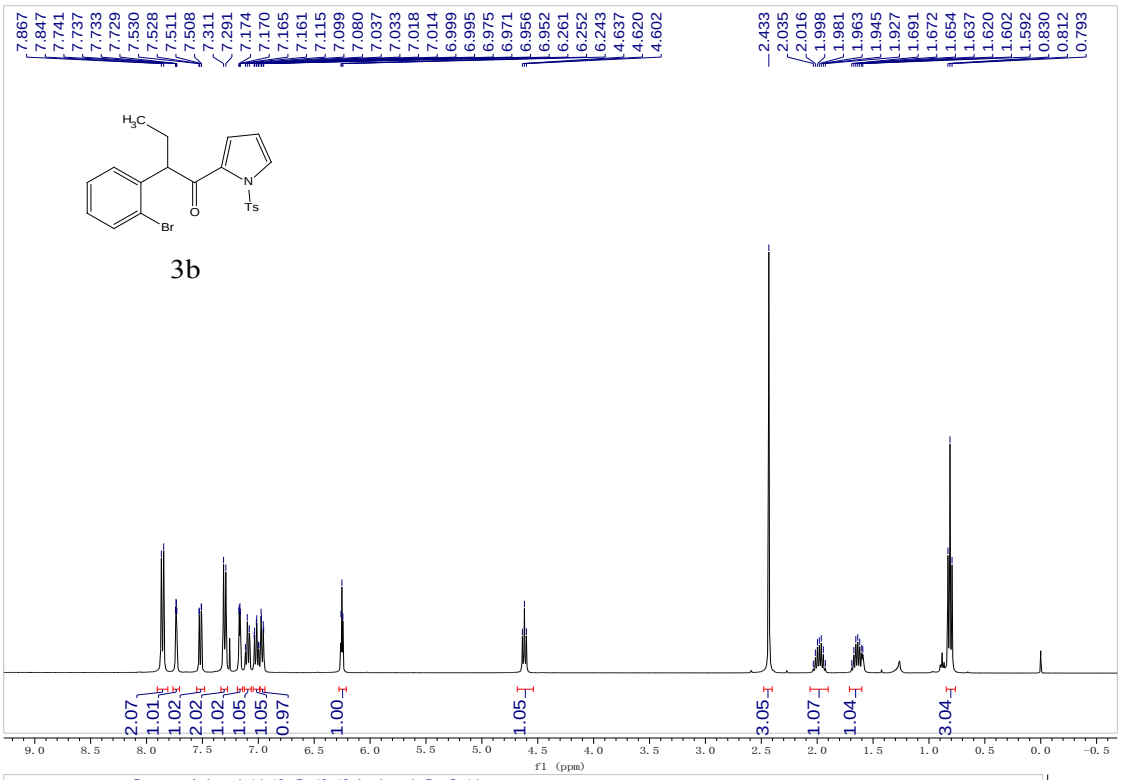
76.84
47.78
21.85
17.69
CH3
N
Ts
O
Br
3a
f1 (ppm)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7.161
7.115
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7.033
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1.592
0.830
0.812
0.793
H3C
N
Ts
O
Br
3b
2.07
1.01
1.02
2.02
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1.05
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1.00
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1.07
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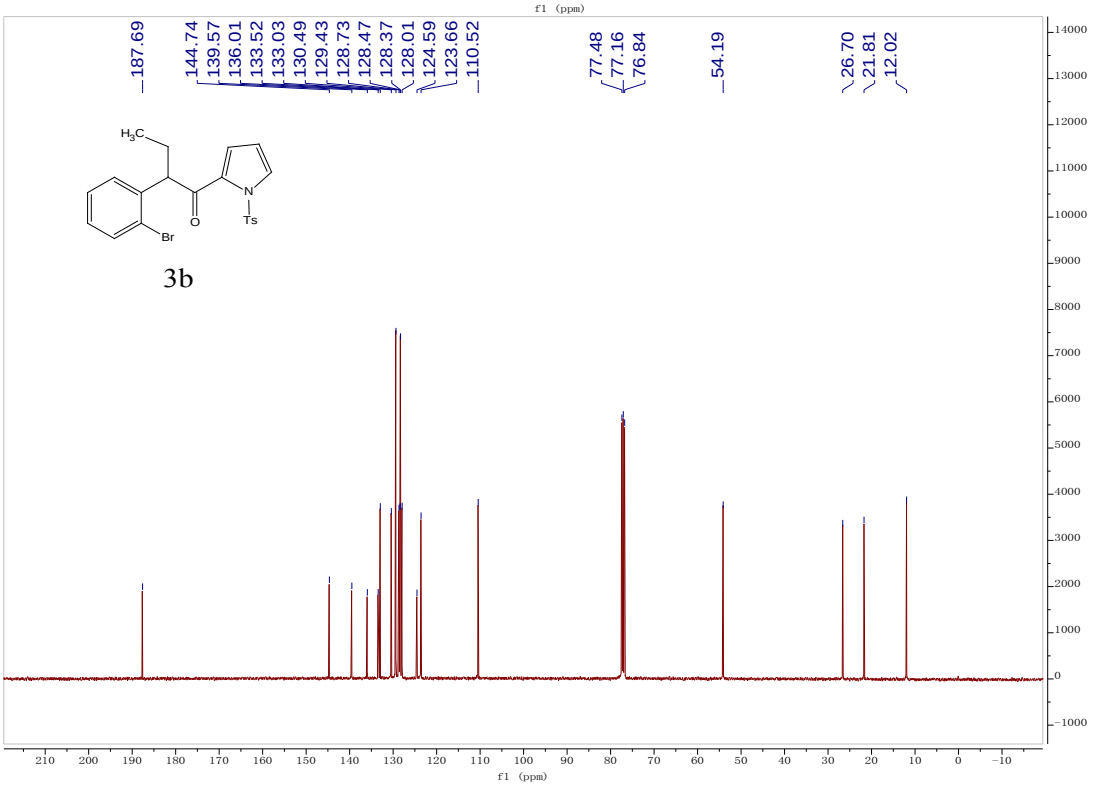
3.04
f1 (ppm)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133.03
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129.43
128.73
128.47
128.37
128.01
124.59
123.66
110.52
77.48
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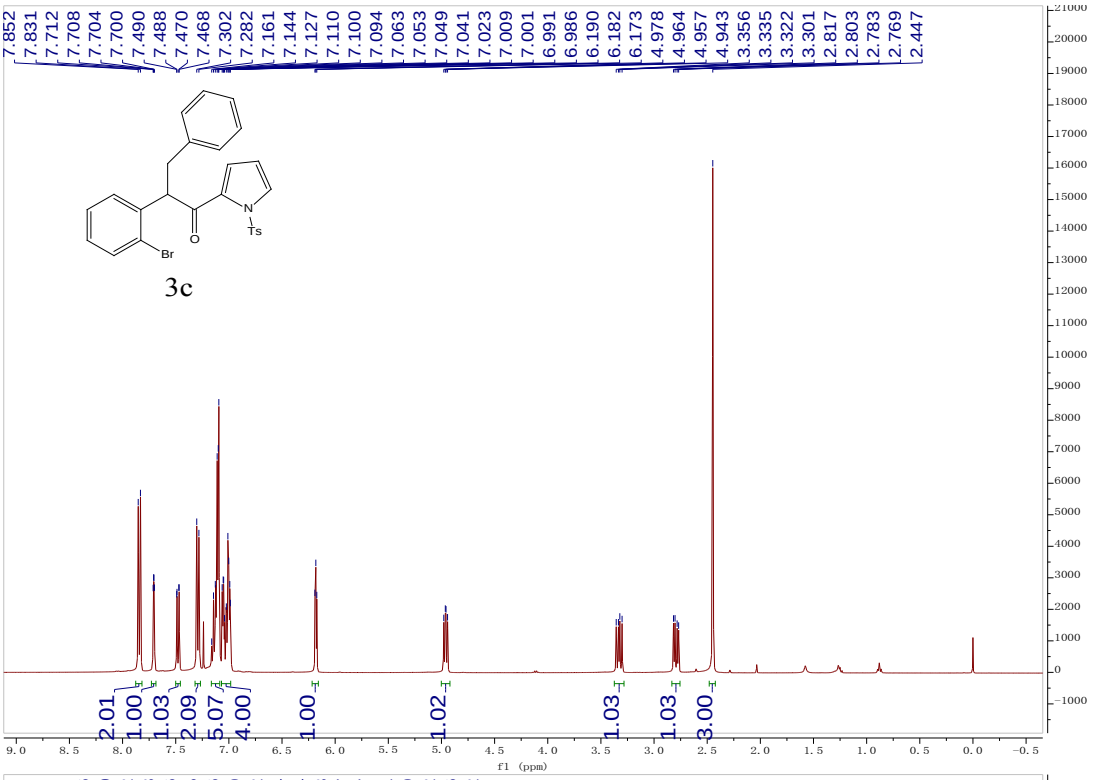
54.19
26.70
21.81
12.02
H3C
N
Ts
O
Br
3b
f1 (ppm)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7.144
7.127
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7.100
7.094
7.063
7.053
7.049
7.041
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7.009
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6.182
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2.803
2.783
2.769
2.447
Br
O
N
Ts
3c
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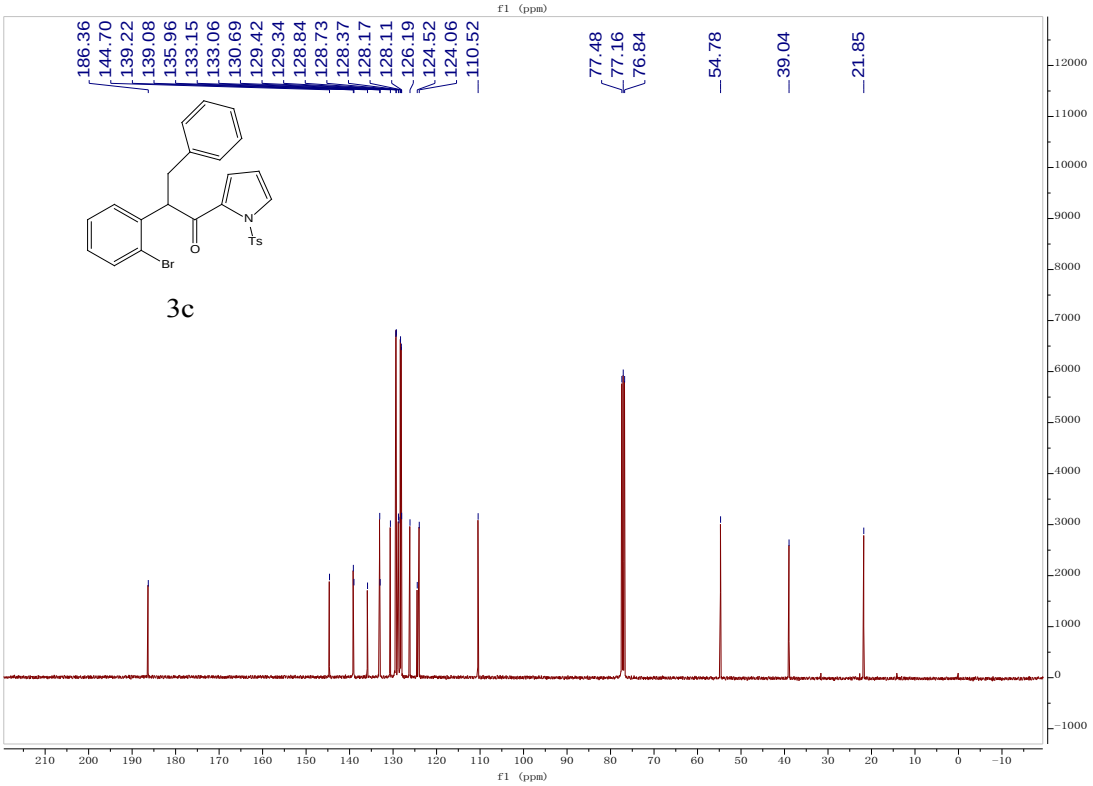
2.09
5.07
4.00
1.00
1.02
1.03
1.03
3.00
f1 (ppm)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130.69
129.42
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128.84
128.73
128.37
128.17
128.11
126.19
124.52
124.06
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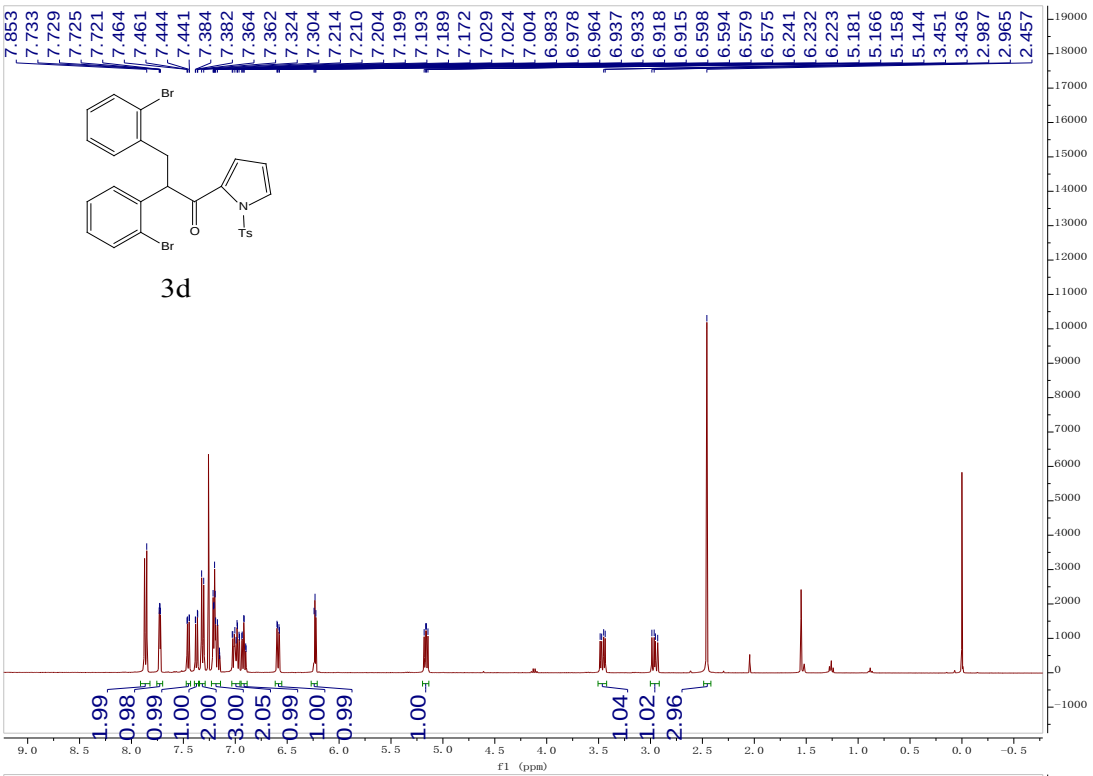
77.16
76.84
54.78
39.04
21.85
Br
O
N
Ts
3c
f1 (ppm)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7.210
7.204
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7.193
7.189
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7.029
7.024
7.004
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6.964
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6.933
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5.181
5.166
5.158
5.144
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3.436
2.987
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Br
N
Ts
O
Br
3d
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0.99
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1.00
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1.04
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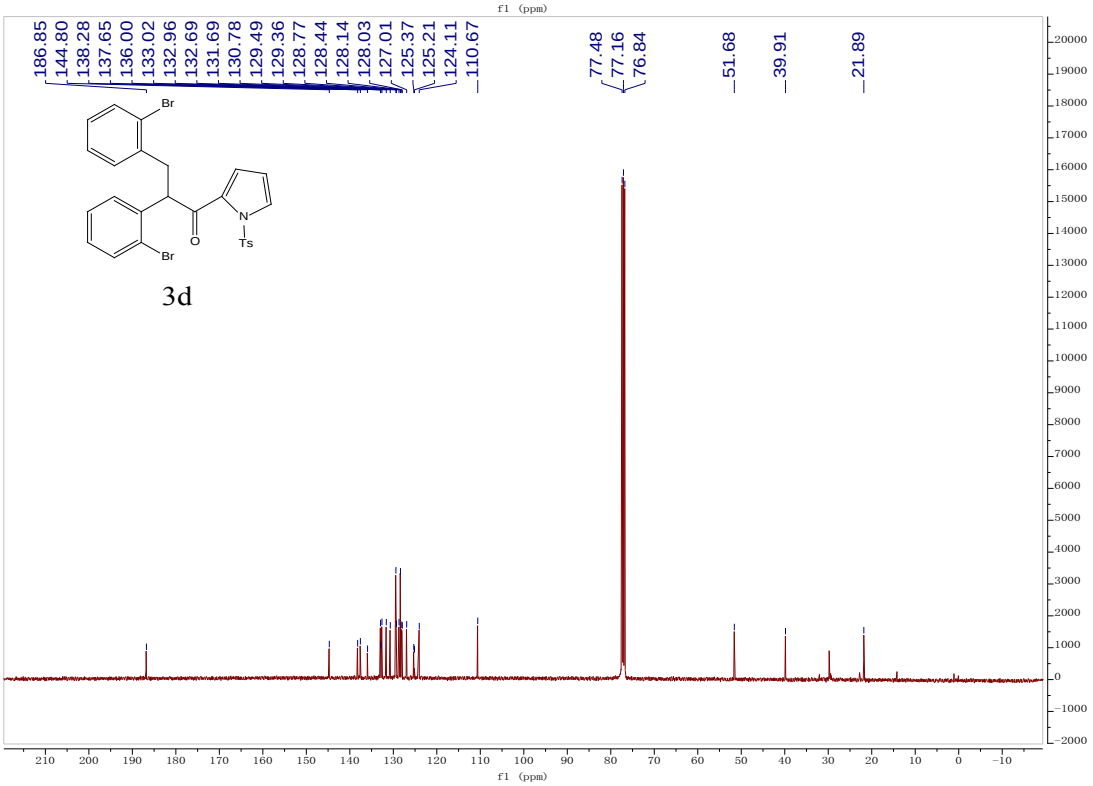
f1 (ppm)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130.78
129.49
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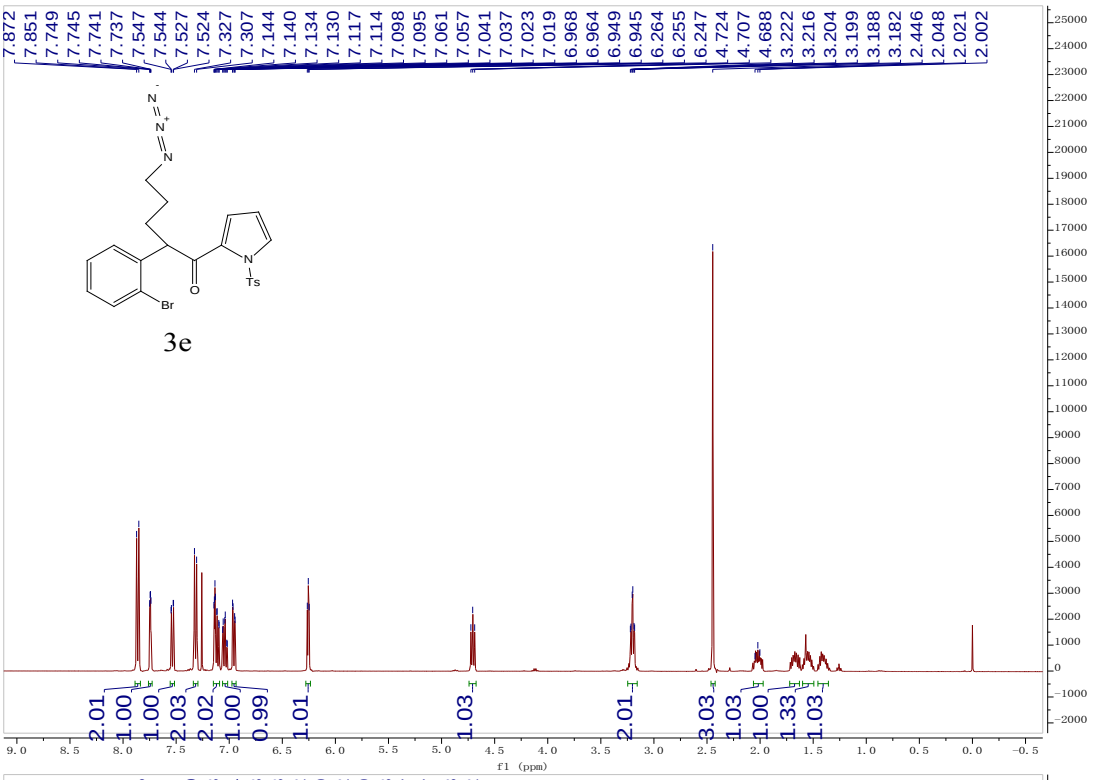
128.44
128.14
128.03
127.01
125.37
125.21
124.11
110.67
77.48
77.16
76.84
51.68
39.91
21.89
Br
N
Ts
O
Br
3d
f1 (ppm)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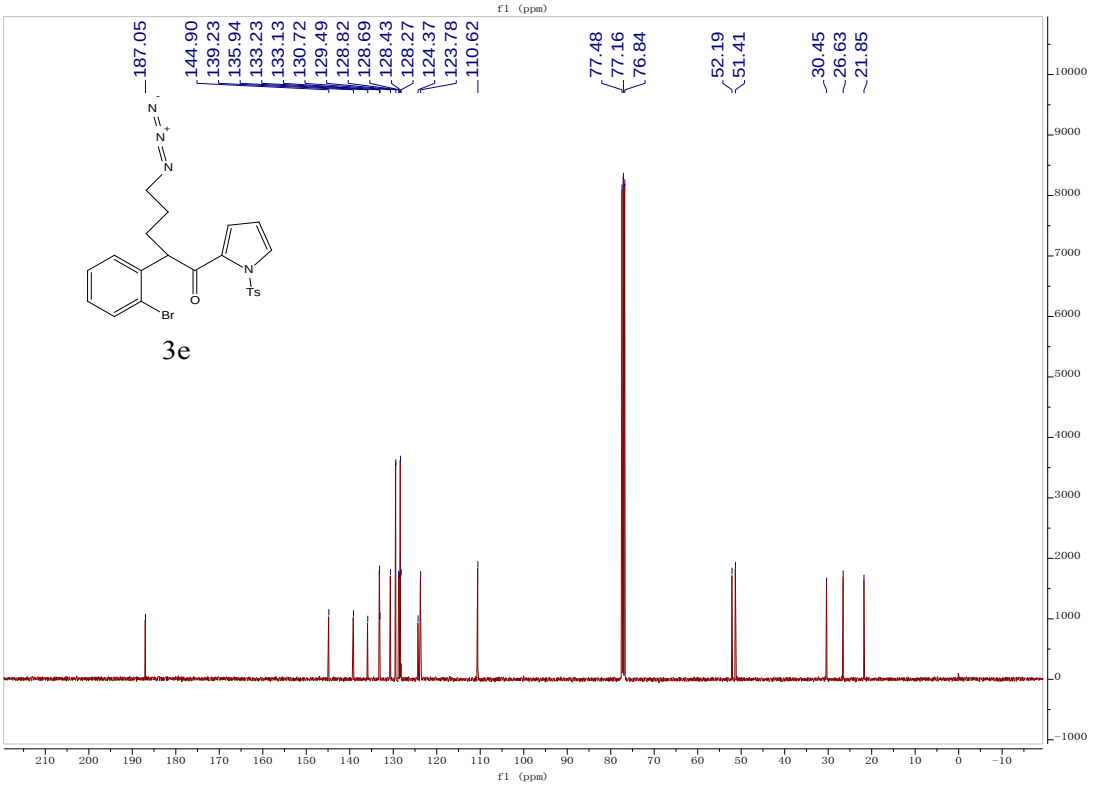
7.741
7.737
7.547
7.544
7.527
7.524
7.327
7.307
7.144
7.140
7.134
7.130
7.117
7.114
7.098
7.095
7.061
7.057
7.041
7.037
7.023
7.019
6.968
6.964
6.949
6.945
6.264
6.255
6.247
4.724
4.707
4.688
3.222
3.216
3.204
3.199
3.188
3.182
2.446
2.048
2.021
2.002
2.01
1.00
1.00
2.03
2.02
1.00
0.99
1.01
1.03
2.01
3.03
1.03
1.00
1.33
1.03
f1 (ppm)

3e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139.23
135.94
133.23
133.13
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129.49
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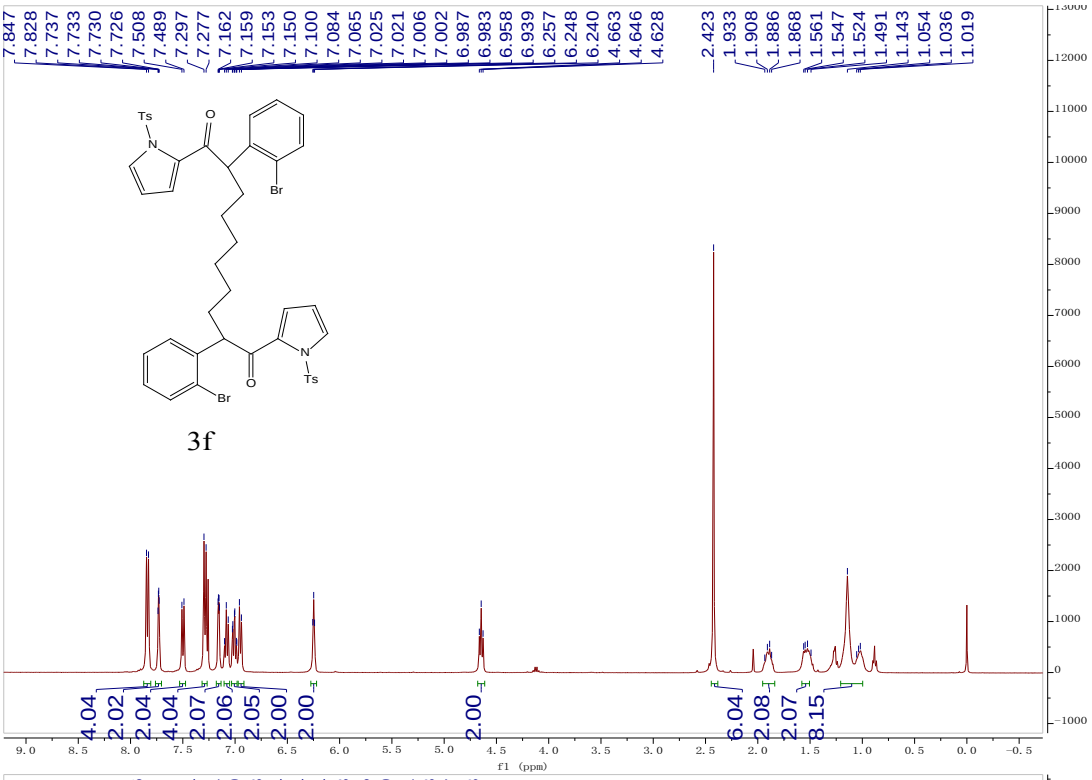
128.69
128.43
128.27
124.37
123.78
110.62
77.48
77.16
76.84
52.19
51.41
30.45
26.63
21.85
f1 (ppm)

3e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7.828
7.737
7.733
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7.726
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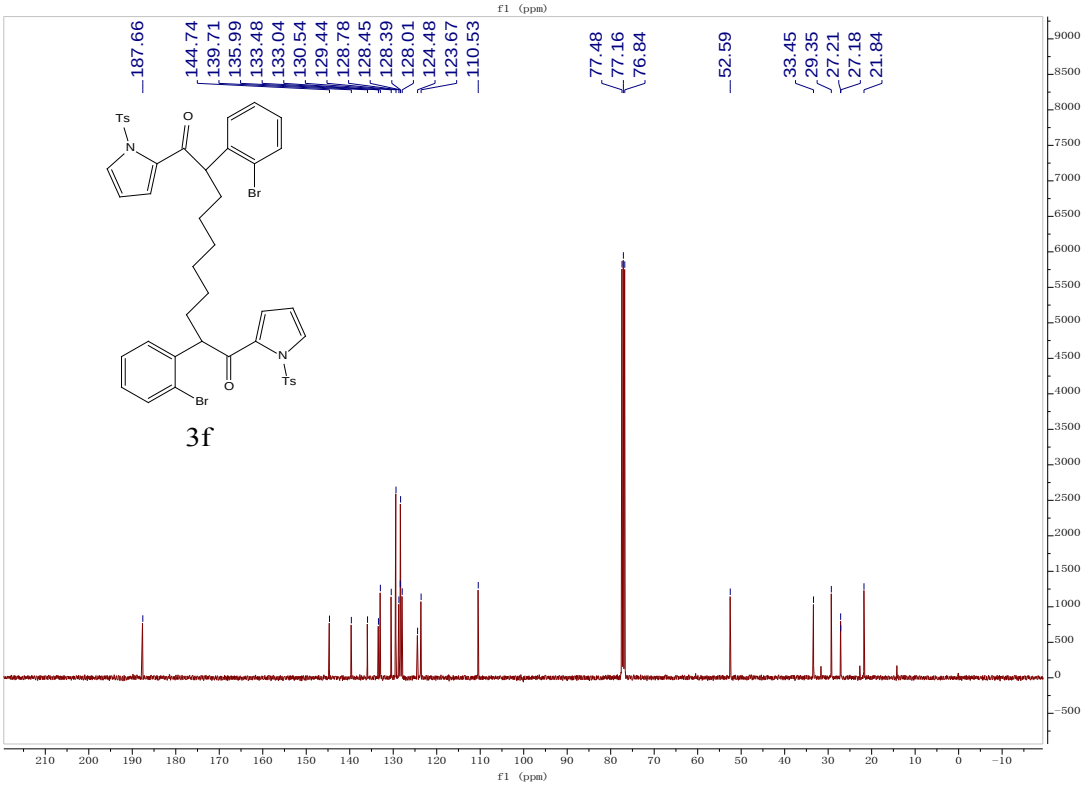
7.489
7.297
7.277
7.162
7.159
7.153
7.150
7.100
7.084
7.065
7.025
7.021
7.006
7.002
6.987
6.983
6.958
6.939
6.257
6.248
6.240
4.663
4.646
4.628
2.423
1.933
1.908
1.886
1.868
1.561
1.547
1.524
1.491
1.143
1.054
1.036
1.019
Ts
O
N
Br
Br
O
N
Ts
3f
4.04
2.02
2.04
4.04
2.07
2.06
2.05
2.00
2.00
2.00
6.04
2.08
2.07
8.15
f1 (ppm)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139.71
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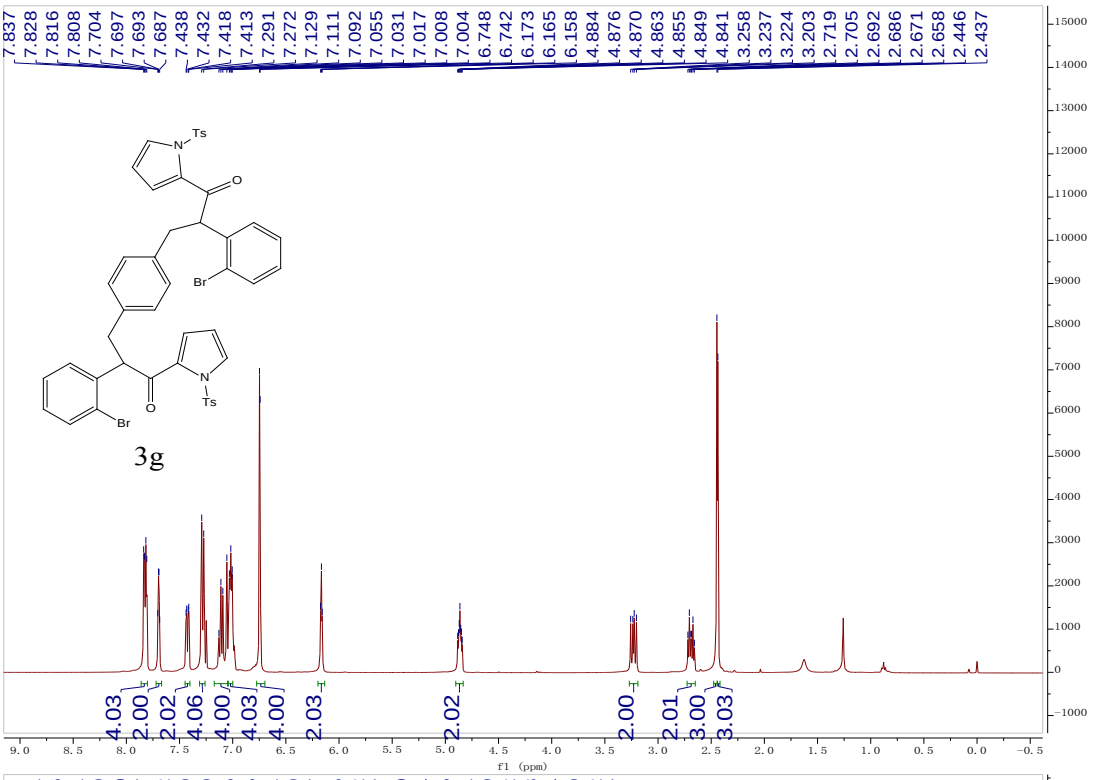
133.04
130.54
129.44
128.78
128.45
128.39
128.01
124.48
123.67
110.53
77.48
77.16
76.84
52.59
33.45
29.35
27.21
27.18
21.84
Ts
O
N
Br
Br
O
N
Ts
3f
f1 (ppm)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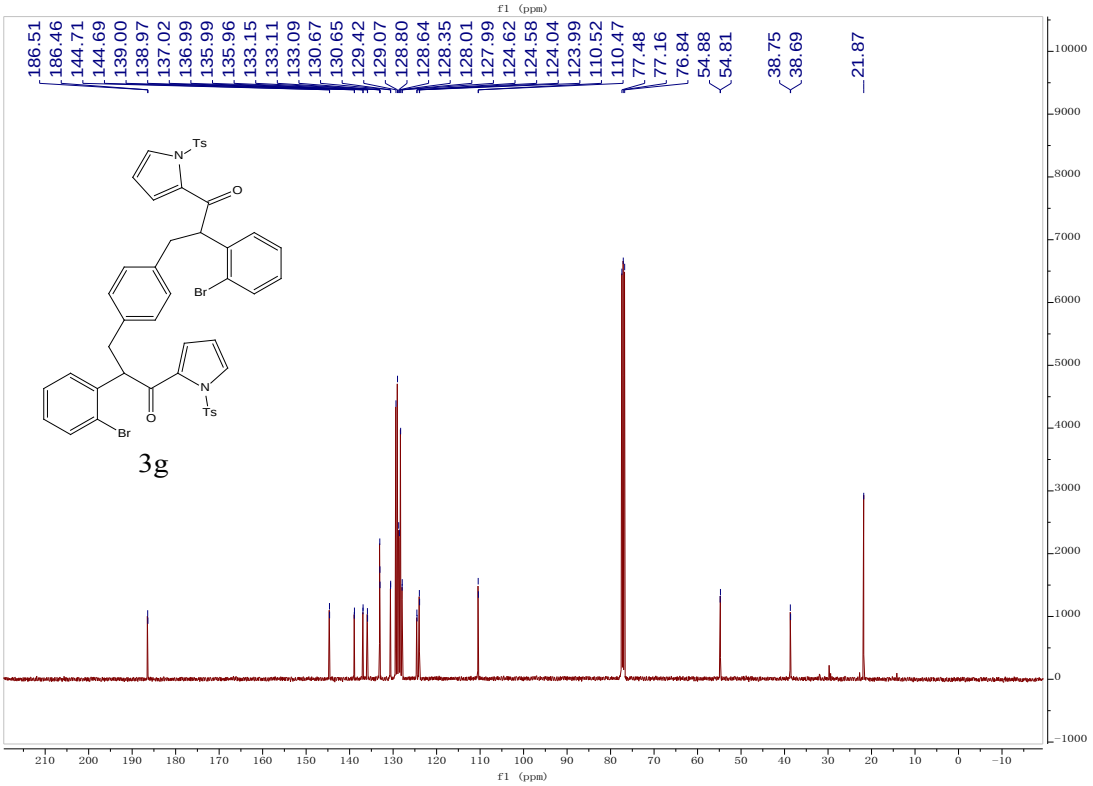
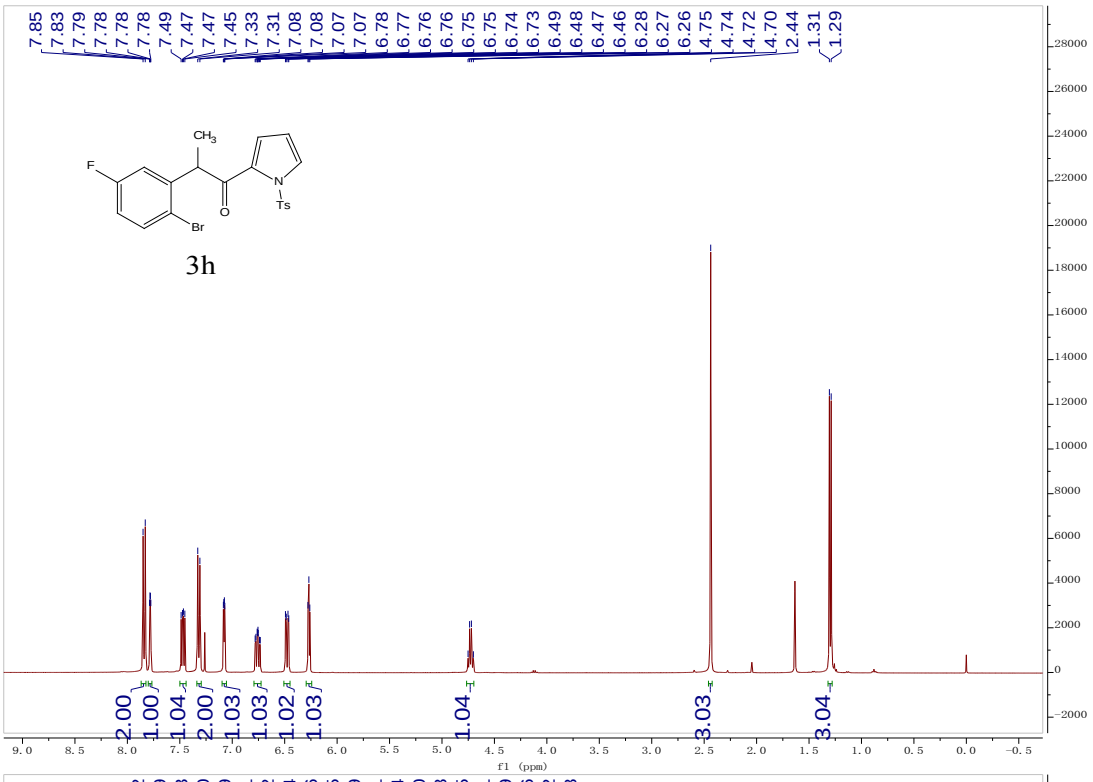
7.808
7.704
7.697
7.693
7.687
7.438
7.432
7.418
7.413
7.291
7.272
7.129
7.111
7.092
7.055
7.031
7.017
7.008
7.004
6.748
6.742
6.173
6.165
6.158
4.884
4.876
4.870
4.863
4.855
4.849
4.841
3.258
3.237
3.224
3.203
2.719
2.705
2.692
2.686
2.671
2.658
2.446
2.437
Ts
N
O
Br
Br
O
N
Ts
3g
4.03
2.00
2.02
4.06
4.00
4.03
4.00
2.03
2.02
2.00
2.01
3.00
3.03
f1 (ppm)


186.51
186.46
144.71
144.69
139.00
138.97
137.02
136.99
135.99
135.96
133.15
133.11
133.09
130.67
130.65
129.42
129.07
128.80
128.64
128.35
128.01
127.99
124.62
124.58
124.04
123.99
110.52
110.47
77.48
77.16
76.84
54.88
54.81
38.75
38.69
21.87
Ts
N
O
Br
Br
O
N
Ts
3g
f1 (ppm)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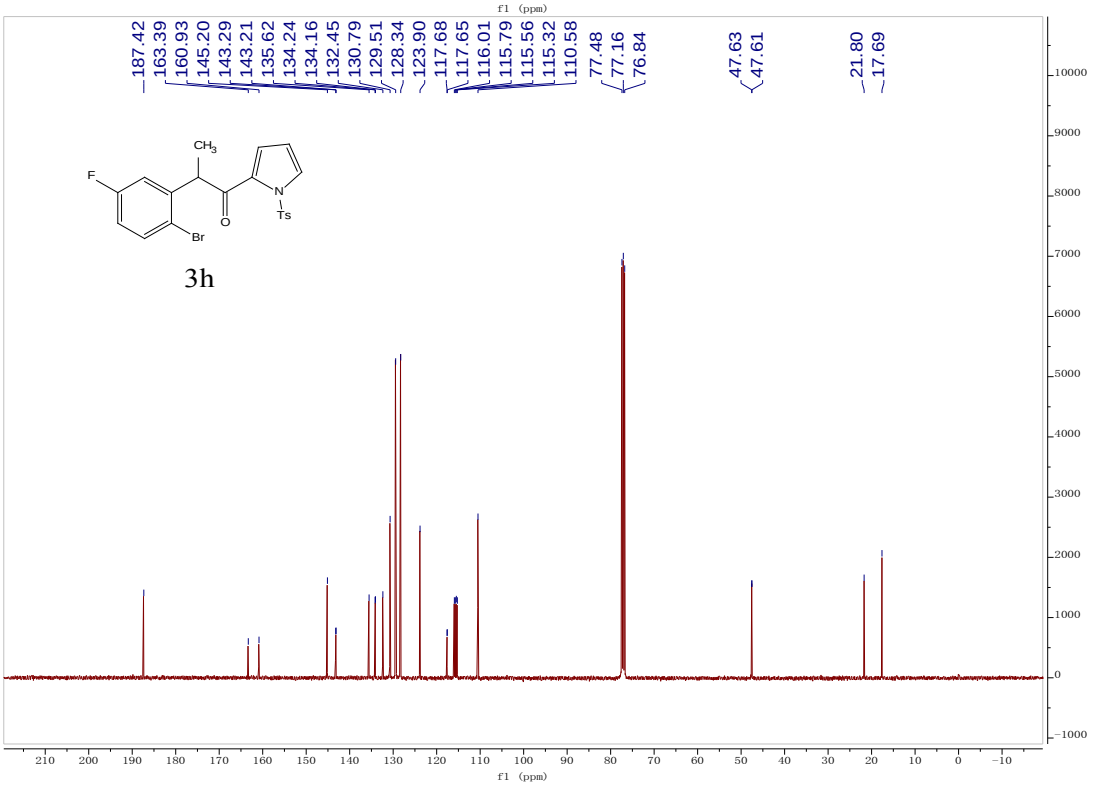
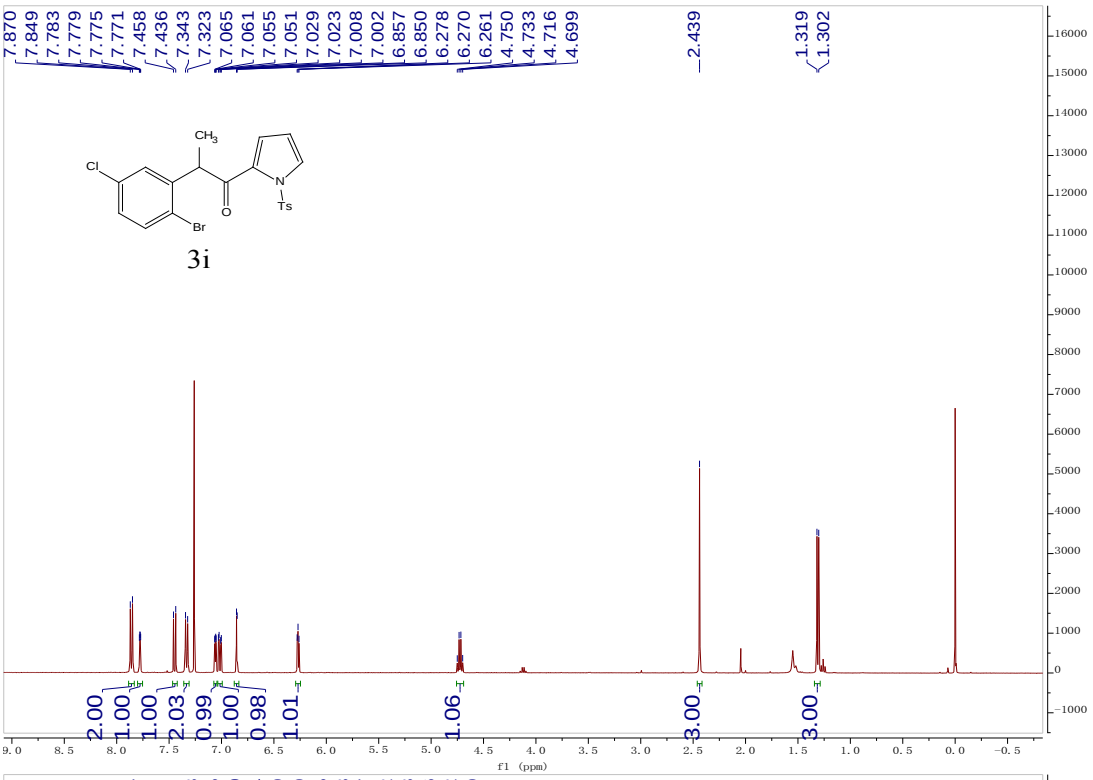
7.83
7.79
7.78
7.78
7.78
7.78
7.49
7.47
7.47
7.45
7.33
7.31
7.08
7.08
7.07
7.07
6.78
6.77
6.76
6.76
6.76
6.75
6.75
6.74
6.73
6.49
6.48
6.47
6.46
6.28
6.27
6.26
4.75
4.74
4.72
4.70
2.44
1.31
1.29
CH3
F
N
Ts
O
Br
3h
2.00
1.00
1.04
2.00
1.03
1.03
1.02
1.03
1.04
3.03
3.04
f1 (ppm)


187.42
163.39
160.93
145.20
143.29
143.21
135.62
134.24
134.16
132.45
130.79
129.51
128.34
123.90
117.68
117.65
116.01
115.79
115.56
115.32
110.58
77.48
77.16
76.84
47.63
47.61
21.80
17.69
CH3
F
N
Ts
O
Br
3h
f1 (ppm)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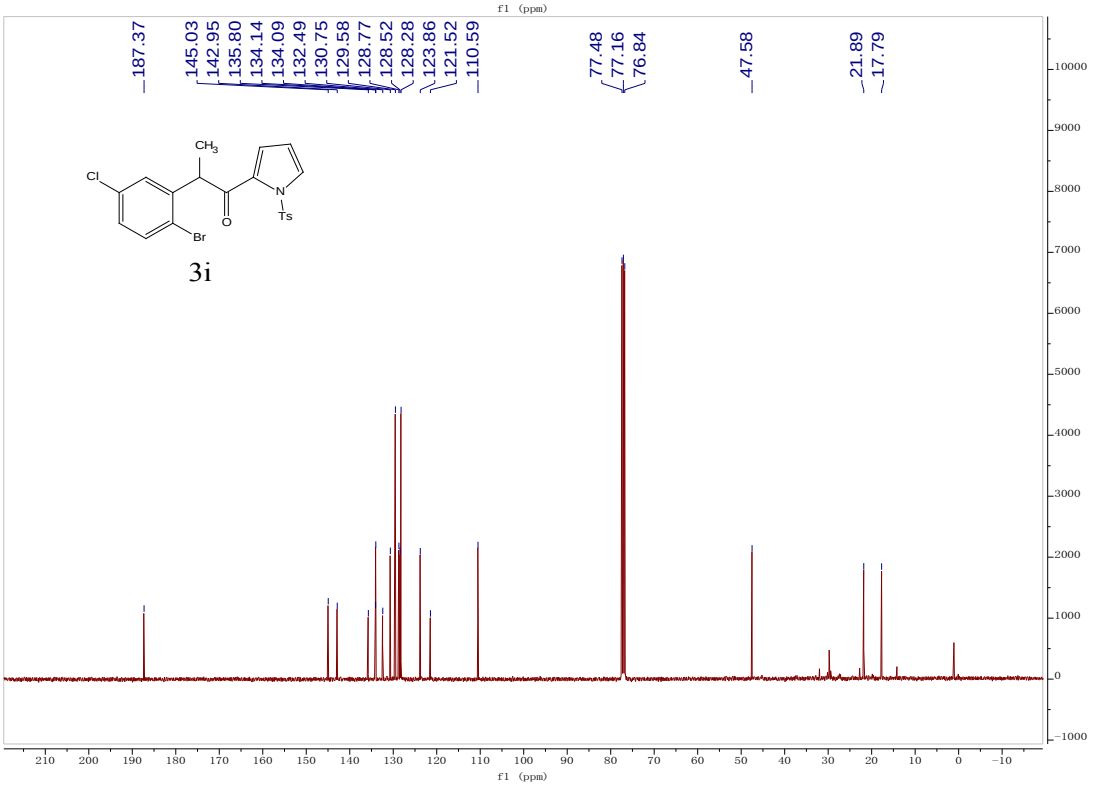
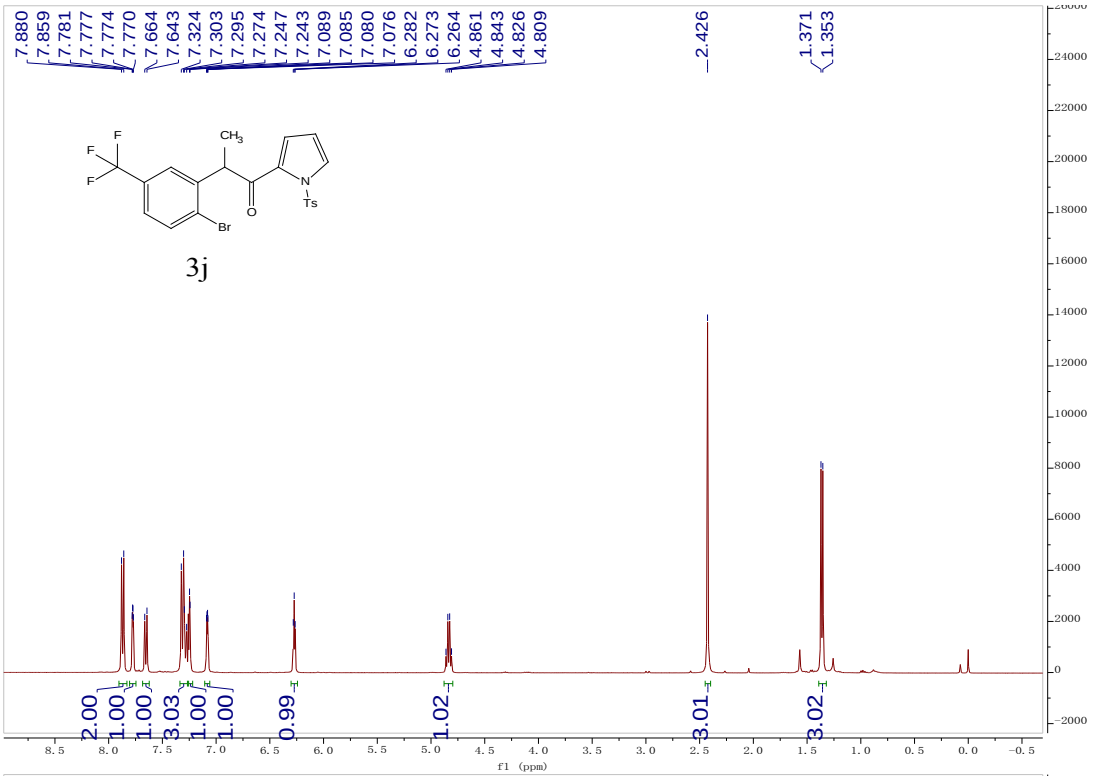
7.849
7.783
7.779
7.775
7.771
7.458
7.436
7.343
7.323
7.065
7.061
7.055
7.051
7.029
7.023
7.008
7.002
6.857
6.850
6.278
6.270
6.261
4.750
4.733
4.716
4.699
2.439
1.319
1.302
CH3
Cl
N
Ts
O
Br
3i
2.00
1.00
1.00
2.03
0.99
1.00
0.98
1.01
1.06
3.00
3.00
f1 (ppm)


187.37
145.03
142.95
135.80
134.14
134.09
132.49
130.75
129.58
128.77
128.52
128.28
123.86
121.52
110.59
77.48
77.16
76.84
47.58
21.89
17.79
CH3
Cl
N
Ts
O
Br
3i
f1 (ppm)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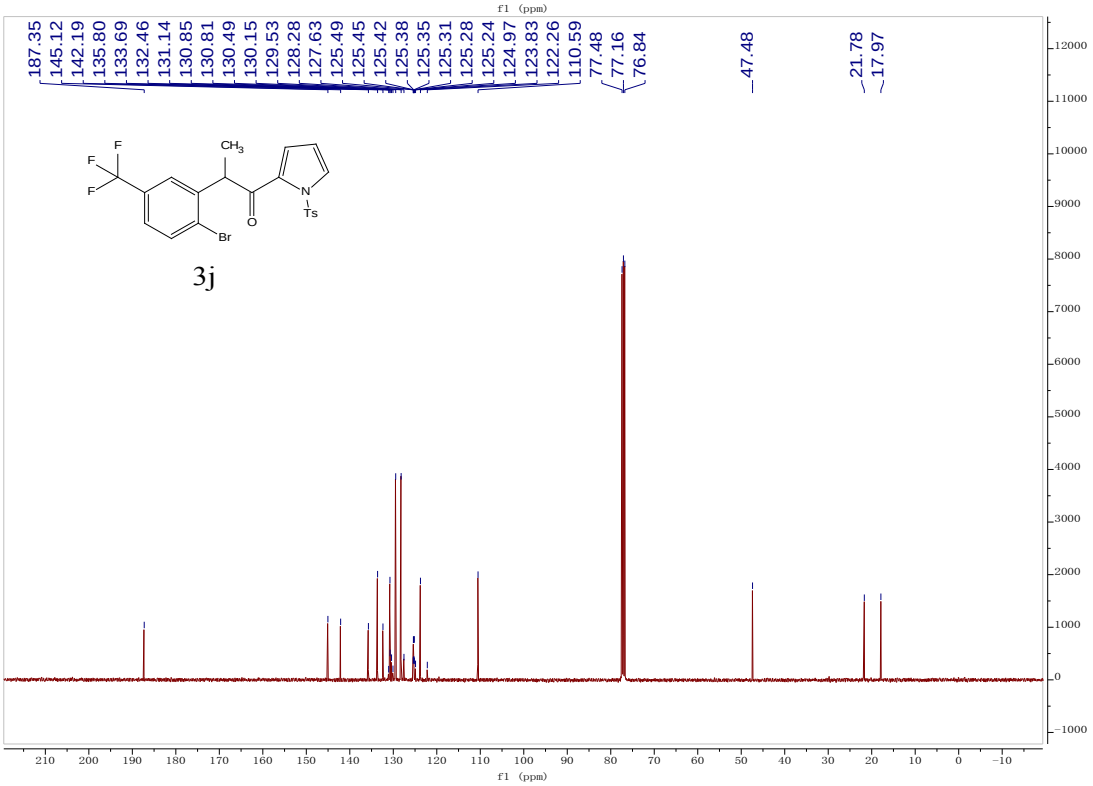
7.643
7.324
7.303
7.295
7.274
7.247
7.243
7.089
7.085
7.080
7.076
6.282
6.273
6.264
4.861
4.843
4.826
4.809
2.426
1.371
1.353
F
F
F
CH3
N
Ts
O
Br
3j
2.00
1.00
1.00
3.03
1.00
1.00
0.99
1.02
3.01
3.02
f1 (ppm)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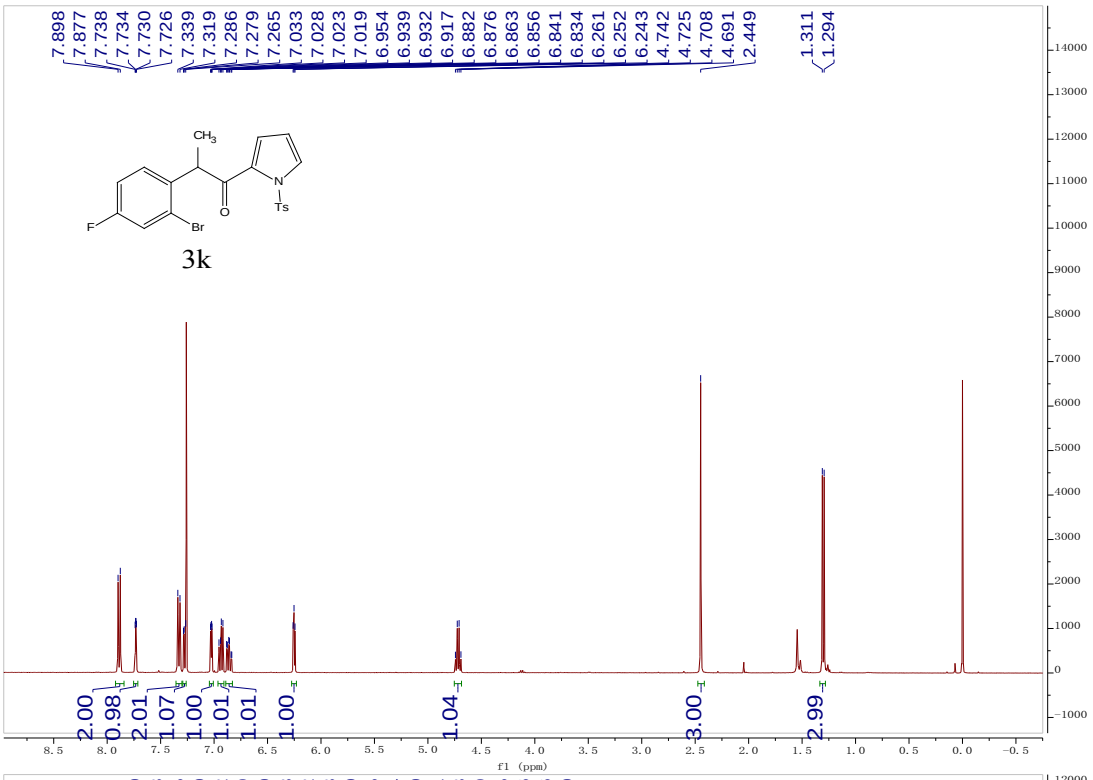
145.12
142.19
135.80
133.69
132.46
131.14
130.85
130.81
130.49
130.15
129.53
128.28
127.63
125.49
125.45
125.42
125.38
125.35
125.31
125.28
125.24
124.97
123.83
122.26
110.59
77.48
77.16
76.84
47.48
21.78
17.97
F
F
F
CH3
N
Ts
O
Br
3j
f1 (ppm)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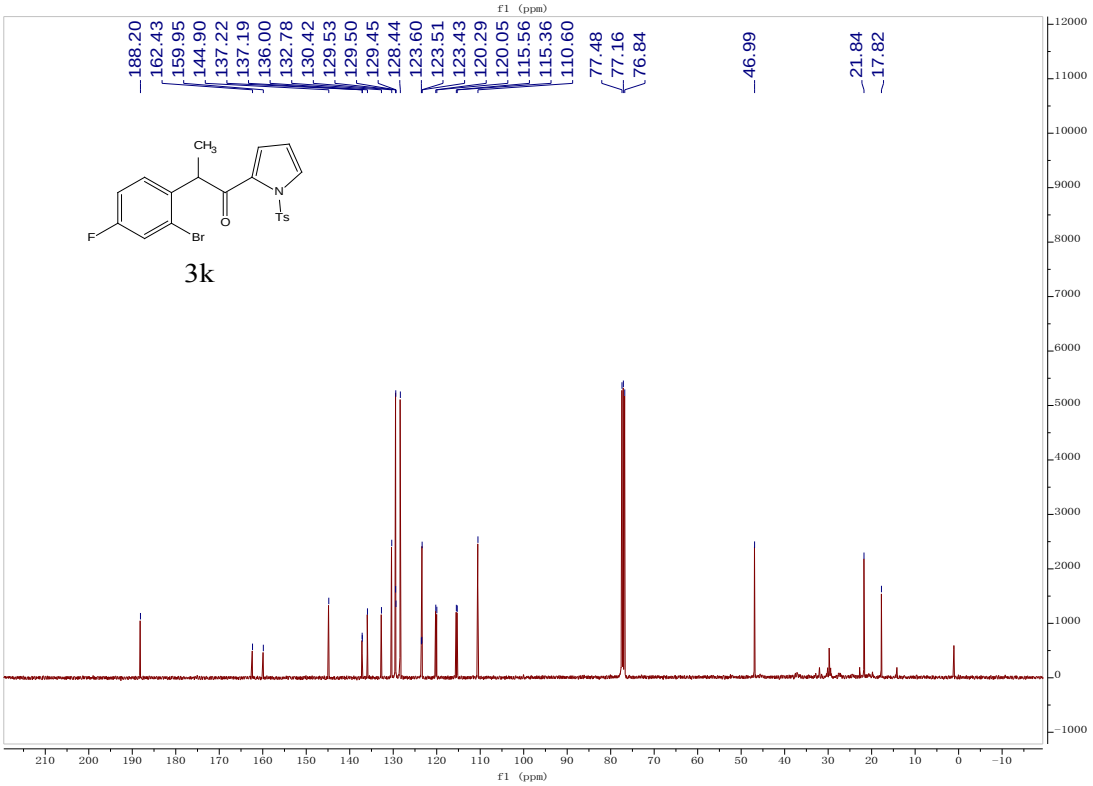
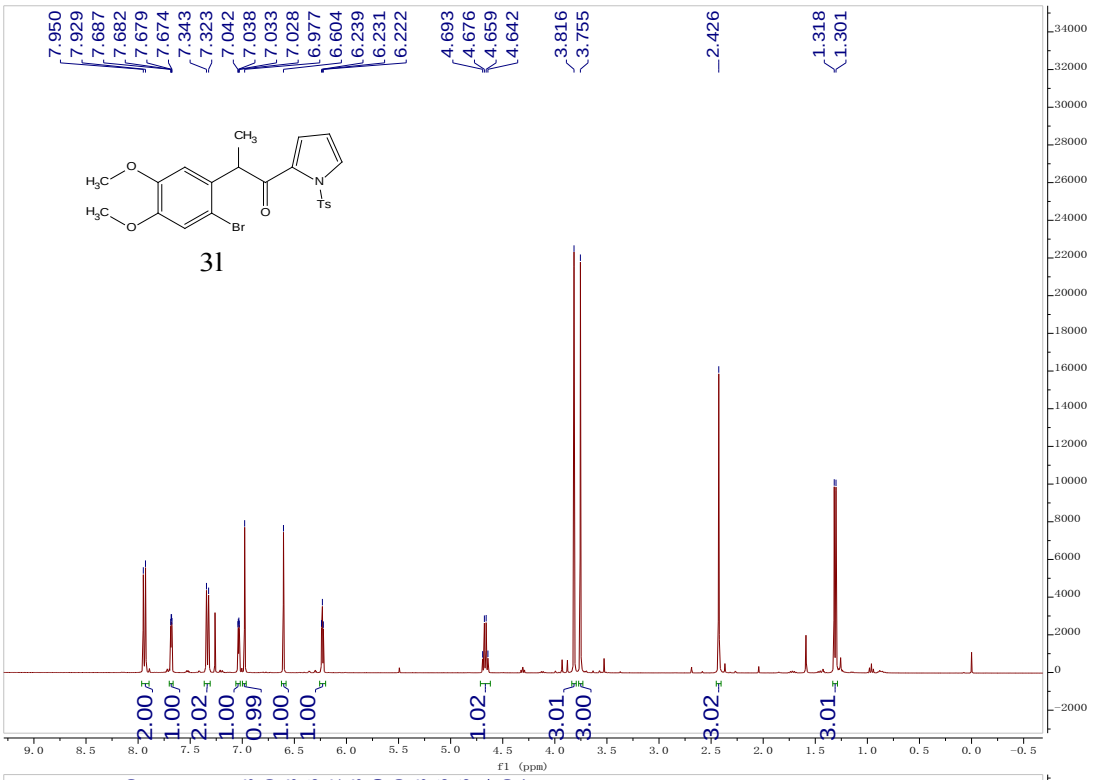
7.734
7.730
7.726
7.339
7.319
7.286
7.279
7.265
7.033
7.028
7.023
7.019
6.954
6.939
6.932
6.917
6.882
6.876
6.863
6.856
6.841
6.834
6.261
6.252
6.243
4.742
4.725
4.708
4.691
2.449
1.311
1.294
CH3
N
Ts
O
F
Br
2.00
0.98
2.01
1.07
1.00
1.01
1.01
1.00
1.04
3.00
2.99
f1 (ppm)

3k


188.20
162.43
159.95
144.90
137.22
137.19
136.00
132.78
130.42
129.53
129.50
129.45
128.44
123.60
123.51
123.43
120.29
120.05
115.56
115.36
110.60
77.48
77.16
76.84
46.99
21.84
17.82
CH3
N
Ts
O
F
Br
f1 (ppm)

3k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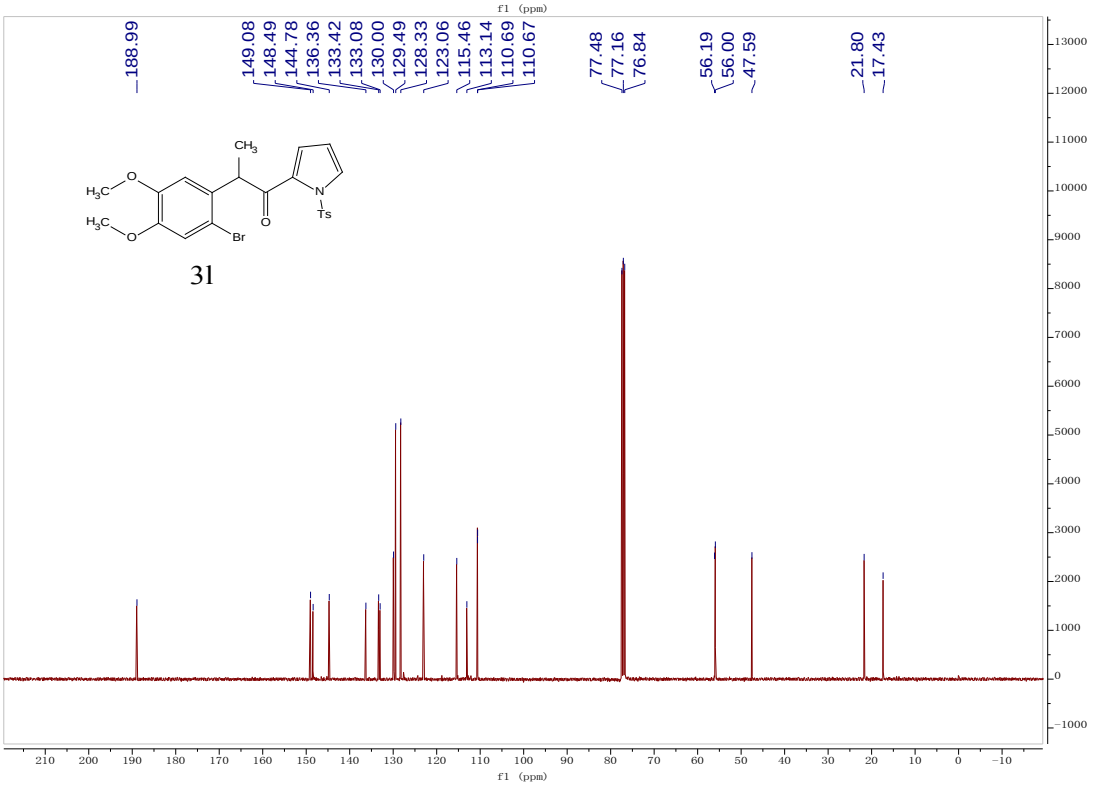
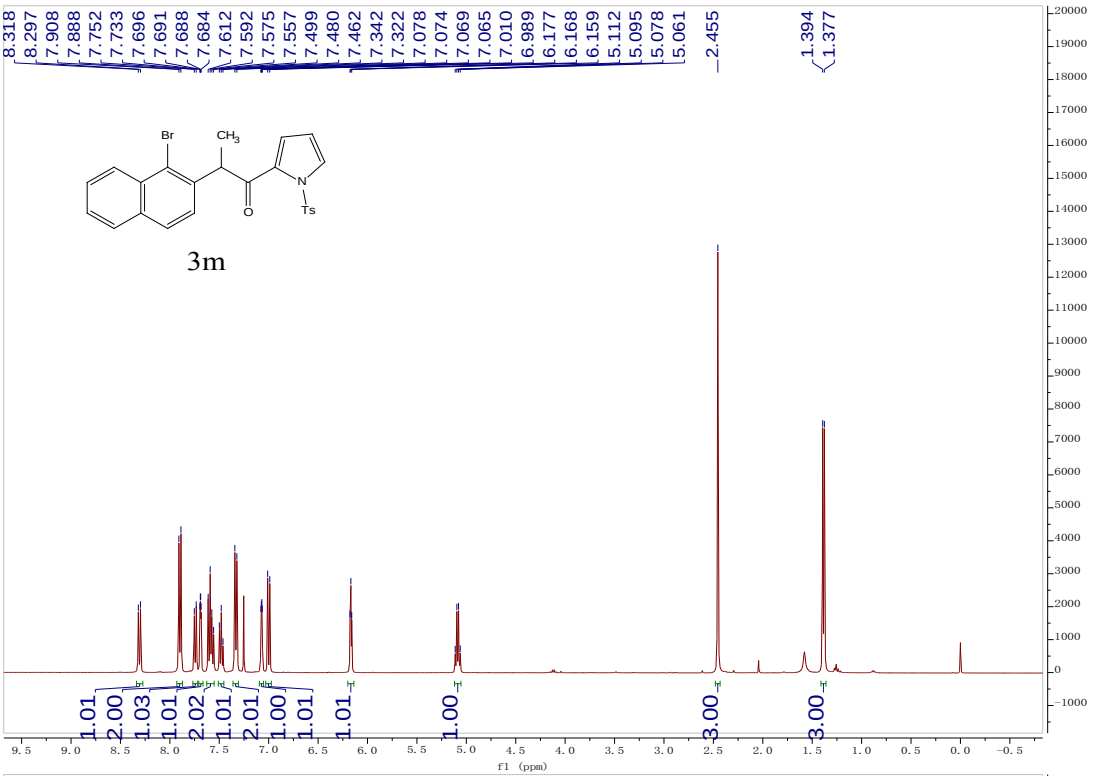
7.674
7.343
7.323
7.042
7.038
7.033
7.028
6.977
6.604
6.239
6.231
6.222
4.693
4.676
4.659
4.642
3.816
3.755
2.426
1.318
1.301
CH3
H3C
O
H3C
O
Br
O
N
Ts
2.00
1.00
2.02
1.00
0.99
1.00
1.00
1.02
3.01
3.00
3.02
3.01
f1 (ppm)

31


188.99
149.08
148.49
144.78
136.36
133.42
133.08
130.00
129.49
128.33
123.06
115.46
113.14
110.69
110.67
77.48
77.16
76.84
56.19
56.00
47.59
21.80
17.43
CH3
H3C
O
H3C
O
Br
O
N
Ts
f1 (ppm)

31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7.752
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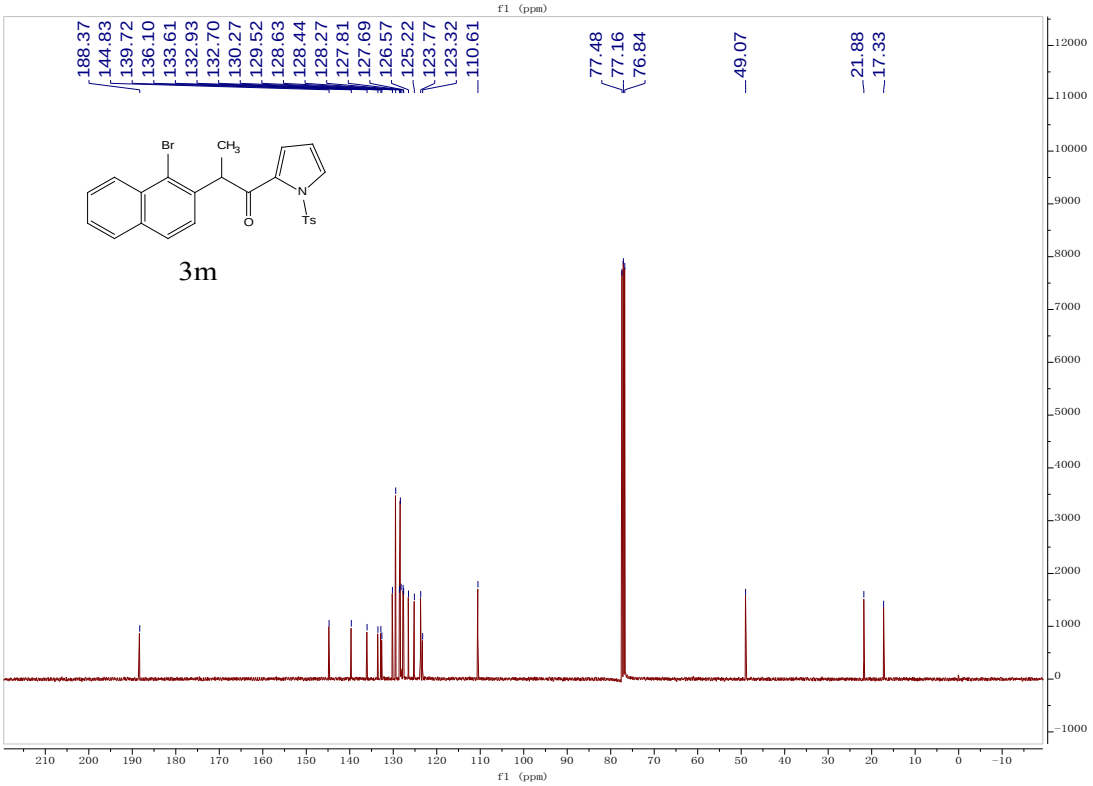
7.691
7.688
7.684
7.612
7.592
7.575
7.557
7.499
7.480
7.462
7.342
7.322
7.078
7.074
7.069
7.065
7.010
6.989
6.177
6.168
6.159
5.112
5.095
5.078
5.061
2.455
1.394
1.377
Br
CH3
N
Ts
O
3m
1.01
2.00
1.03
1.01
2.02
1.01
2.01
1.00
1.01
1.01
1.00
3.00
3.00
f1 (ppm)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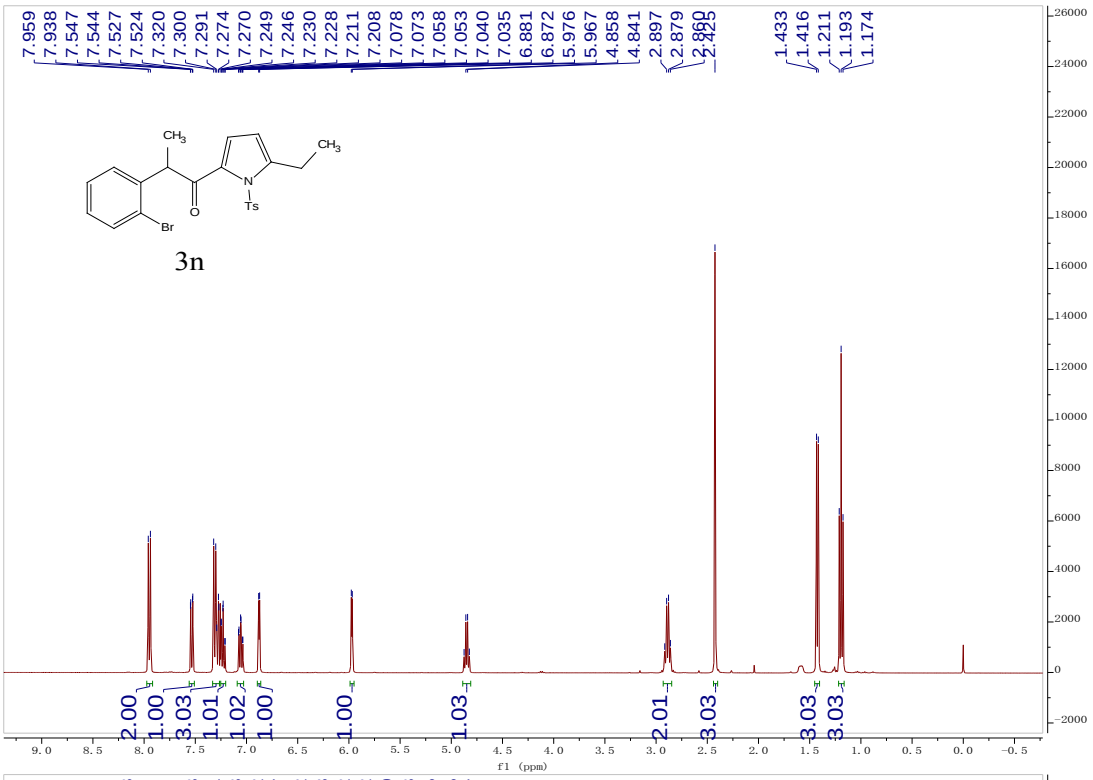
144.83
139.72
136.10
133.61
132.93
132.70
130.27
129.52
128.63
128.44
128.27
127.81
127.69
126.57
125.22
123.77
123.32
110.61
77.48
77.16
76.84
49.07
21.88
17.33
Br
CH3
N
Ts
O
3m
f1 (ppm)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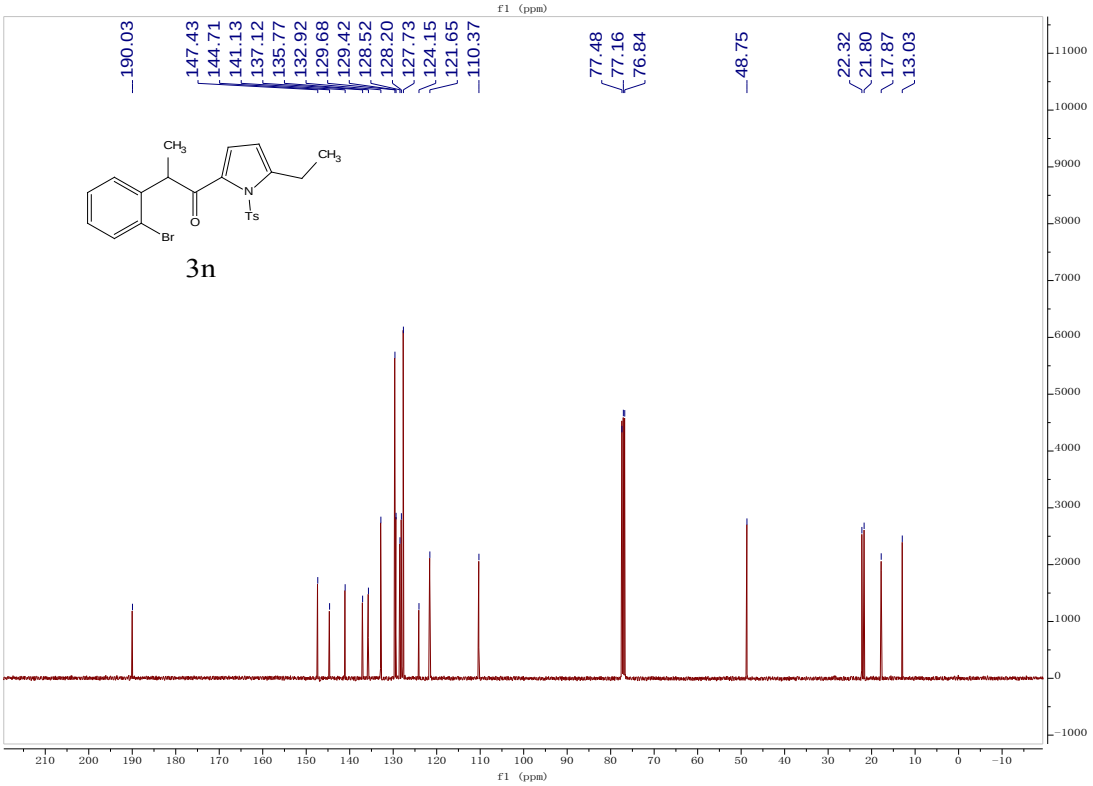
7.544
7.527
7.524
7.320
7.300
7.291
7.274
7.270
7.249
7.246
7.230
7.228
7.211
7.208
7.078
7.073
7.058
7.053
7.040
7.035
6.881
6.872
5.976
5.967
4.858
4.841
2.897
2.879
2.860
2.425
1.433
1.416
1.211
1.193
1.174
CH3
CH3
N
Ts
O
Br
3n
2.00
1.00
3.03
1.01
1.02
1.00
1.00
1.03
2.01
3.03
3.03
3.03
f1 (ppm)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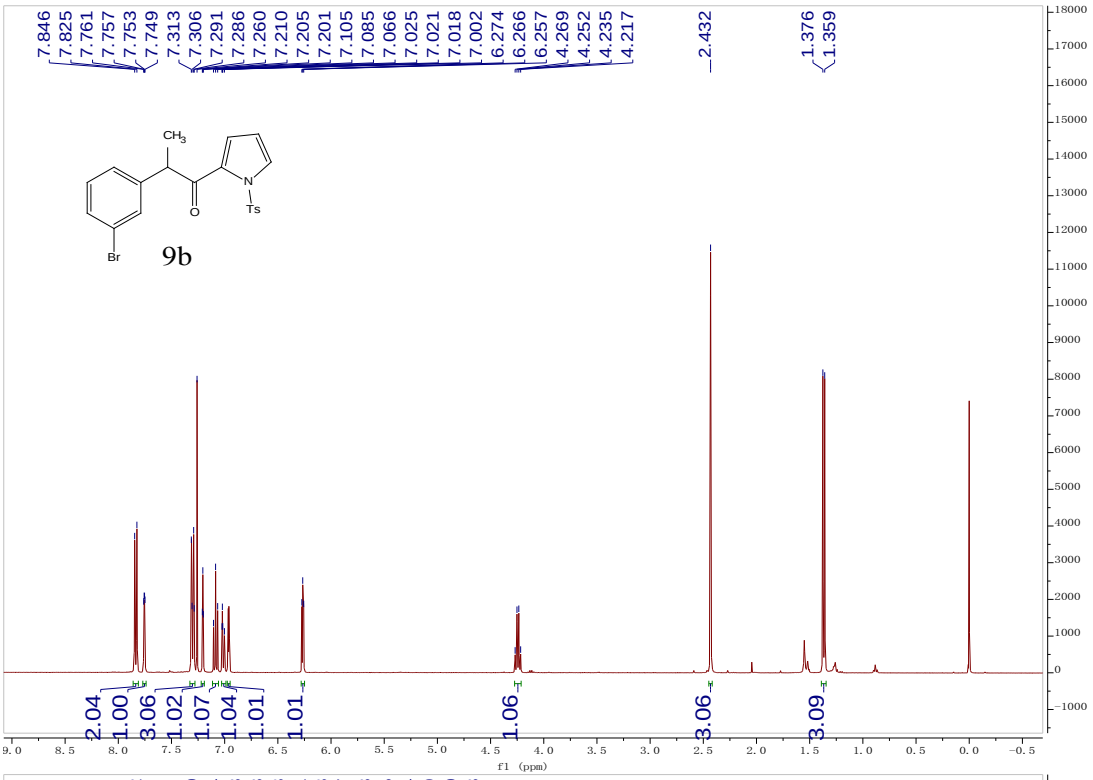
147.43
144.71
141.13
137.12
135.77
132.92
129.68
129.42
128.52
128.20
127.73
124.15
121.65
110.37
77.48
77.16
76.84
48.75
22.32
21.80
17.87
13.03
CH3
CH3
N
Ts
O
Br
3n
f1 (ppm)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7.749
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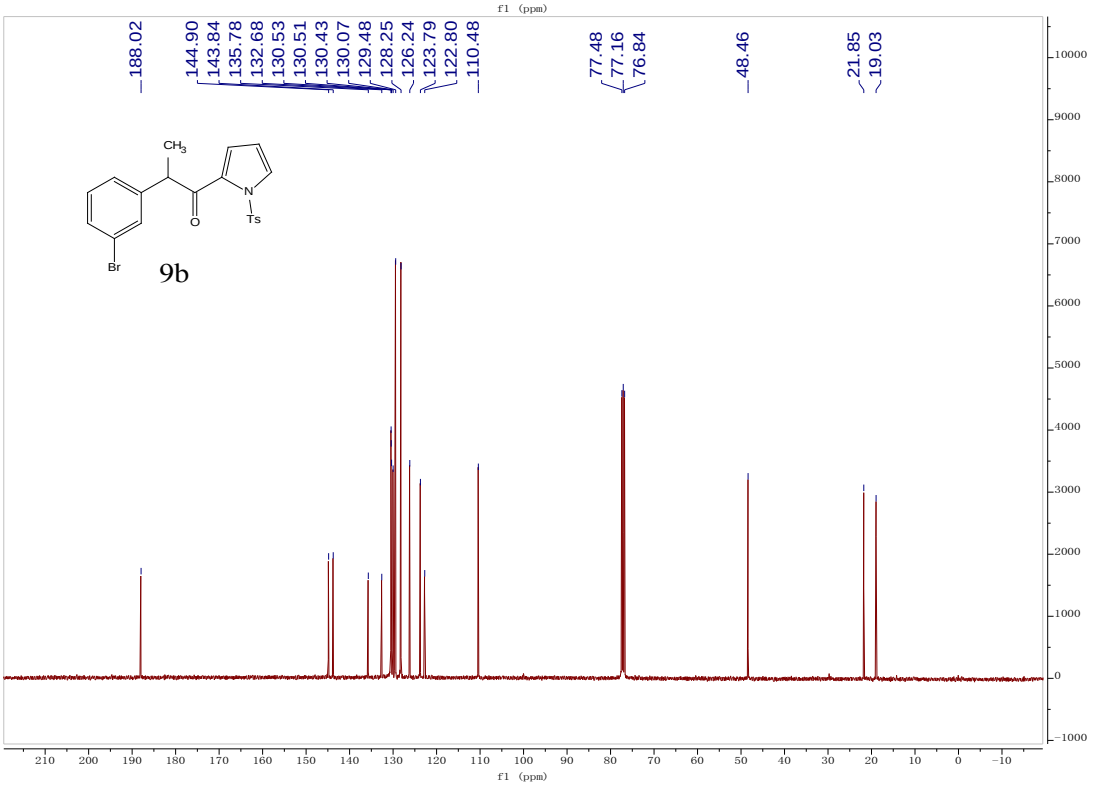
7.306
7.291
7.286
7.260
7.210
7.205
7.201
7.105
7.085
7.066
7.025
7.021
7.018
7.002
6.274
6.266
6.257
4.269
4.252
4.235
4.217
2.432
1.376
1.359
CH3
N
Ts
O
Br
9b
2.04
1.00
3.06
1.02
1.07
1.04
1.01
1.01
1.06
3.06
3.09
f1 (ppm)


188.02
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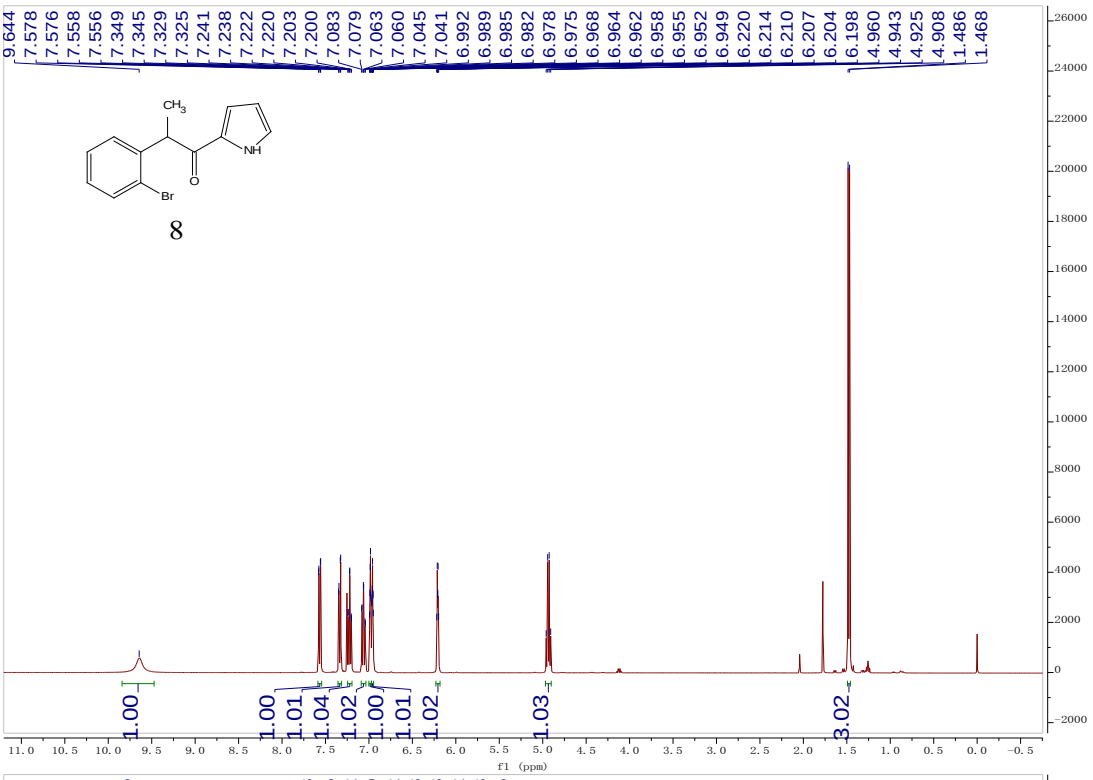
143.84
135.78
132.68
130.53
130.51
130.43
130.07
129.48
128.25
126.24
123.79
122.80
110.48
77.48
77.16
76.84
48.46
21.85
19.03
CH3
N
Ts
O
Br
9b
f1 (ppm)


9.644
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7.349
7.345
7.329
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7.241
7.238
7.222
7.220
7.203
7.200
7.083
7.079
7.063
7.060
7.045
7.041
6.992
6.989
6.985
6.982
6.978
6.975
6.968
6.964
6.962
6.958
6.955
6.952
6.949
6.220
6.214
6.210
6.207
6.204
6.198
4.960
4.943
4.925
4.908
1.486
1.468
CH3
NH
O
Br
8
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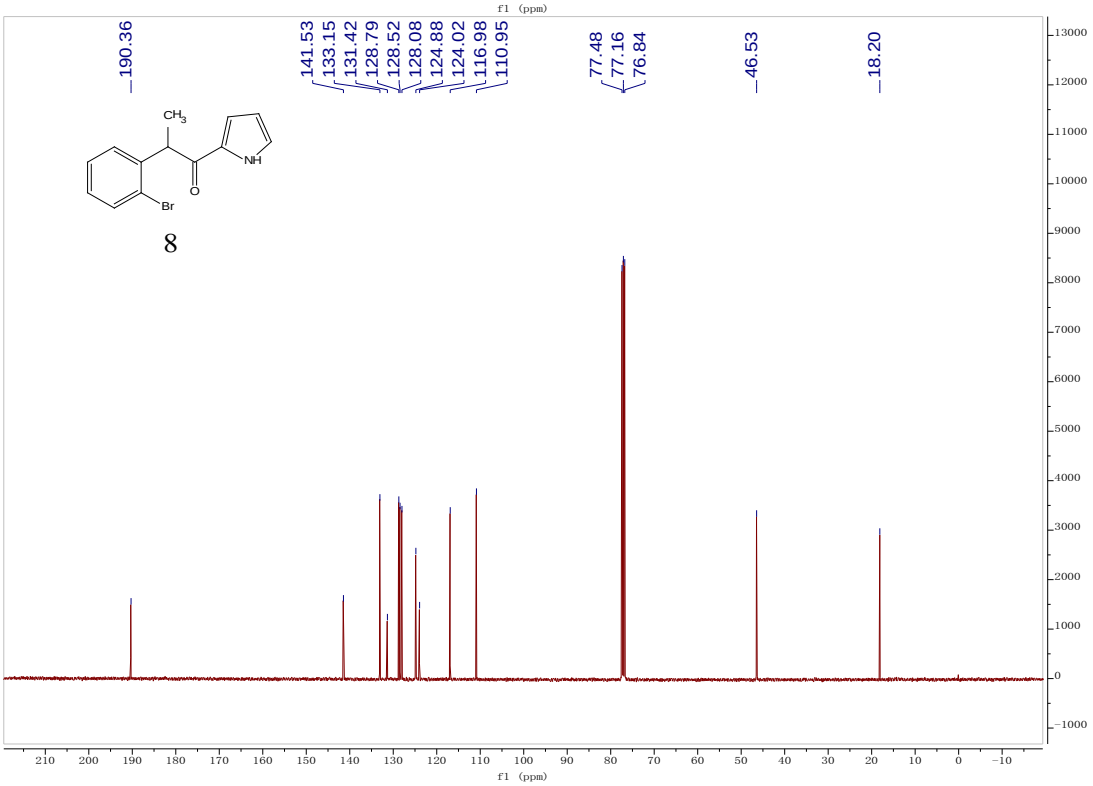
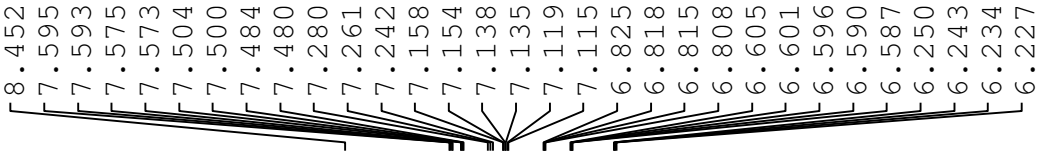
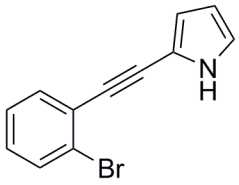
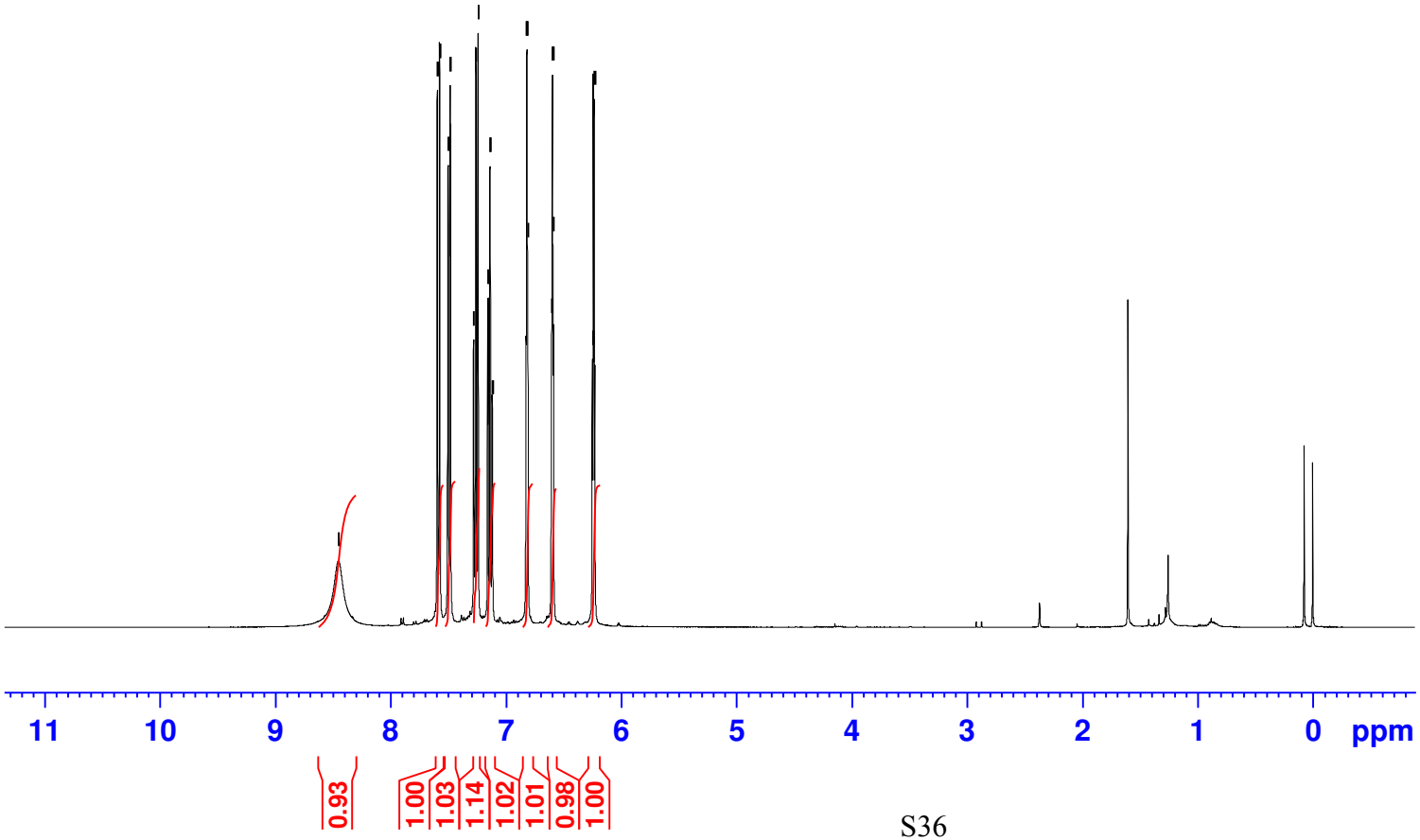
1.00
1.01
1.04
1.02
1.00
1.01
1.02
1.03
3.02
f1 (ppm)


190.36
141.53
133.15
131.42
128.79
128.52
128.08
124.88
124.02
116.98
110.95
77.48
77.16
76.84
46.53
18.20
CH3
NH
O
Br
8
f1 (ppm)





2

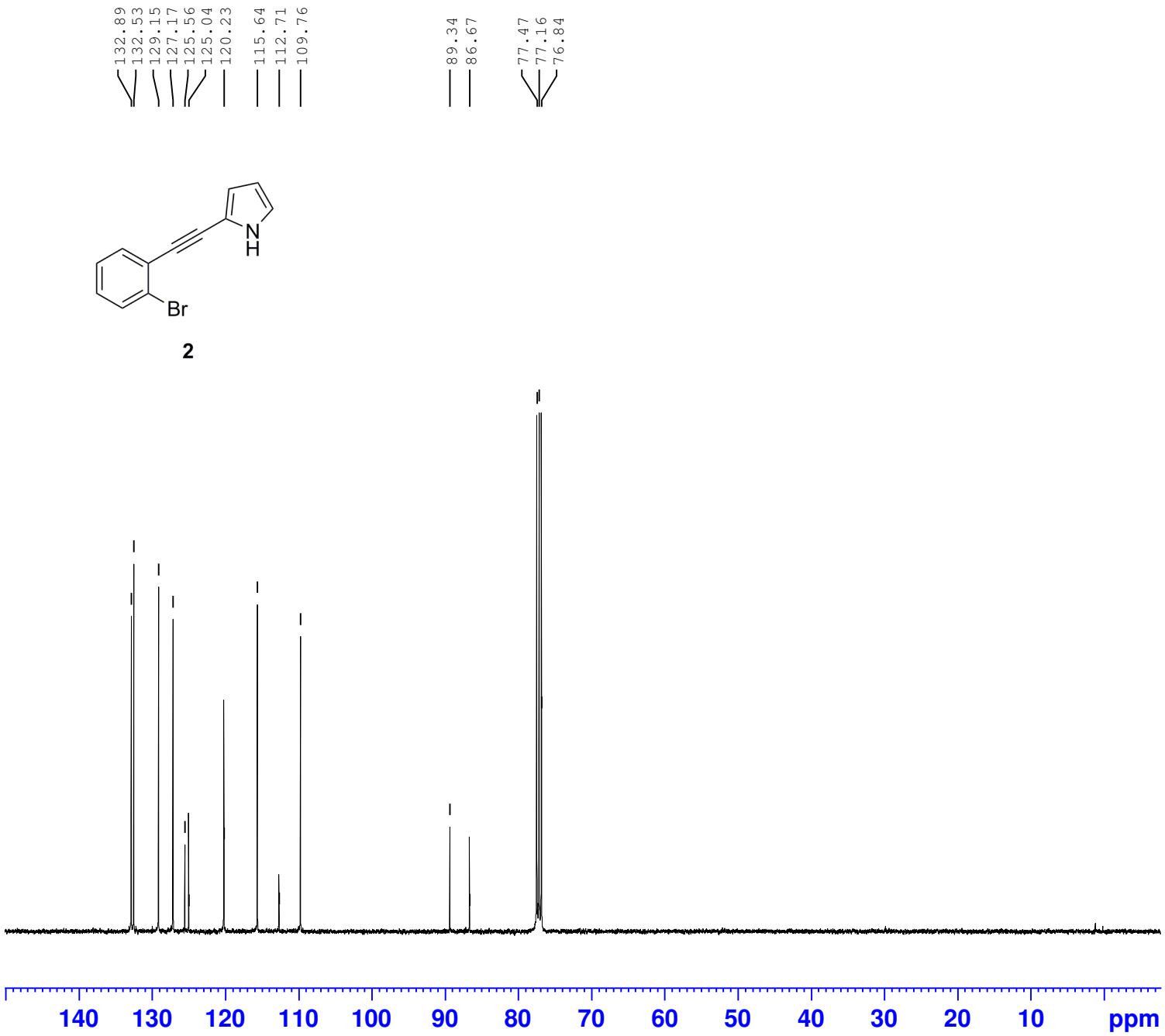


Current Data Parameters
NAME K-G17
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170322
Time 13.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 114.84
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.09 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300171 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



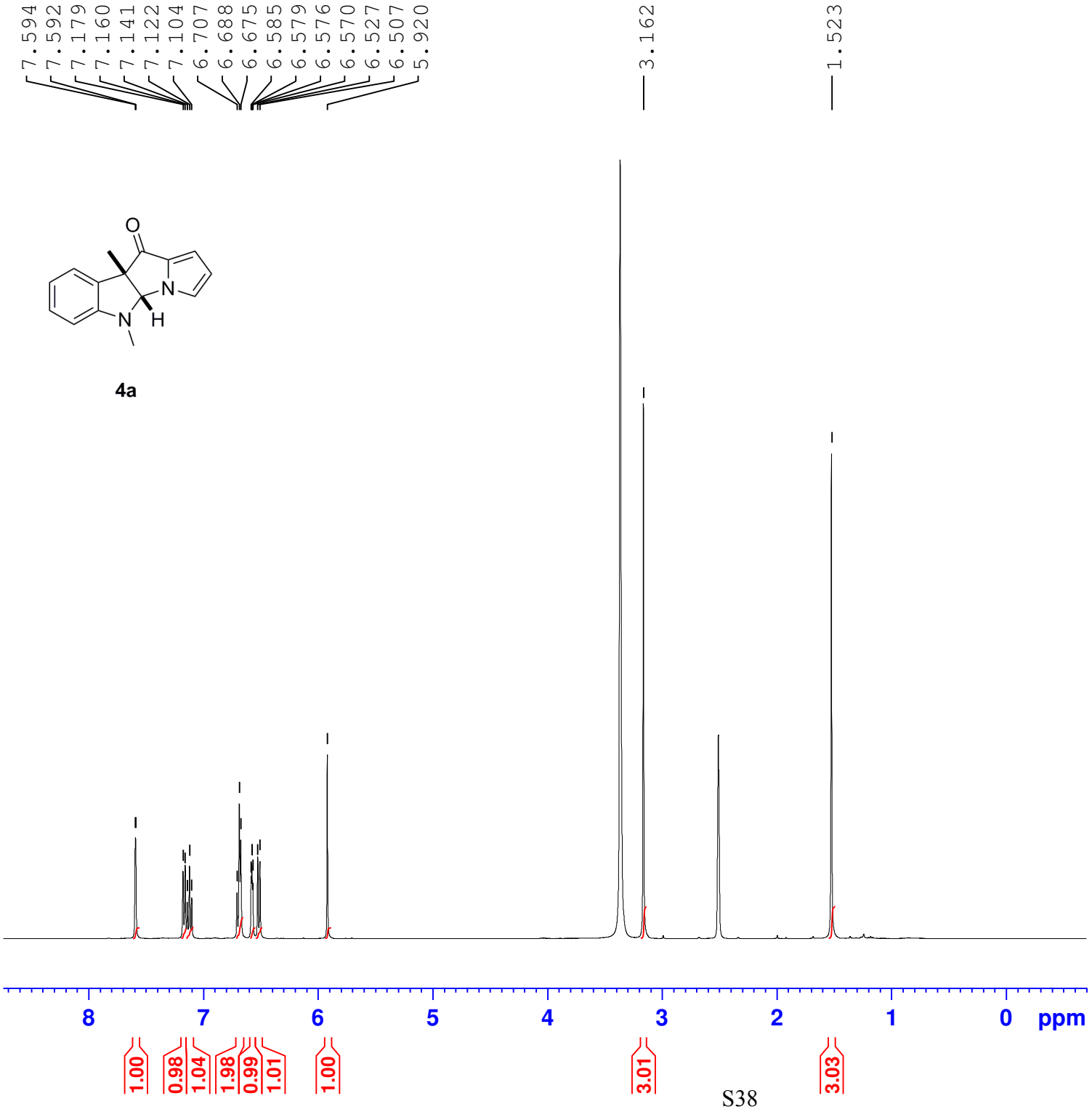
Current Data Parameters
NAME K-G17
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170322
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127593 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

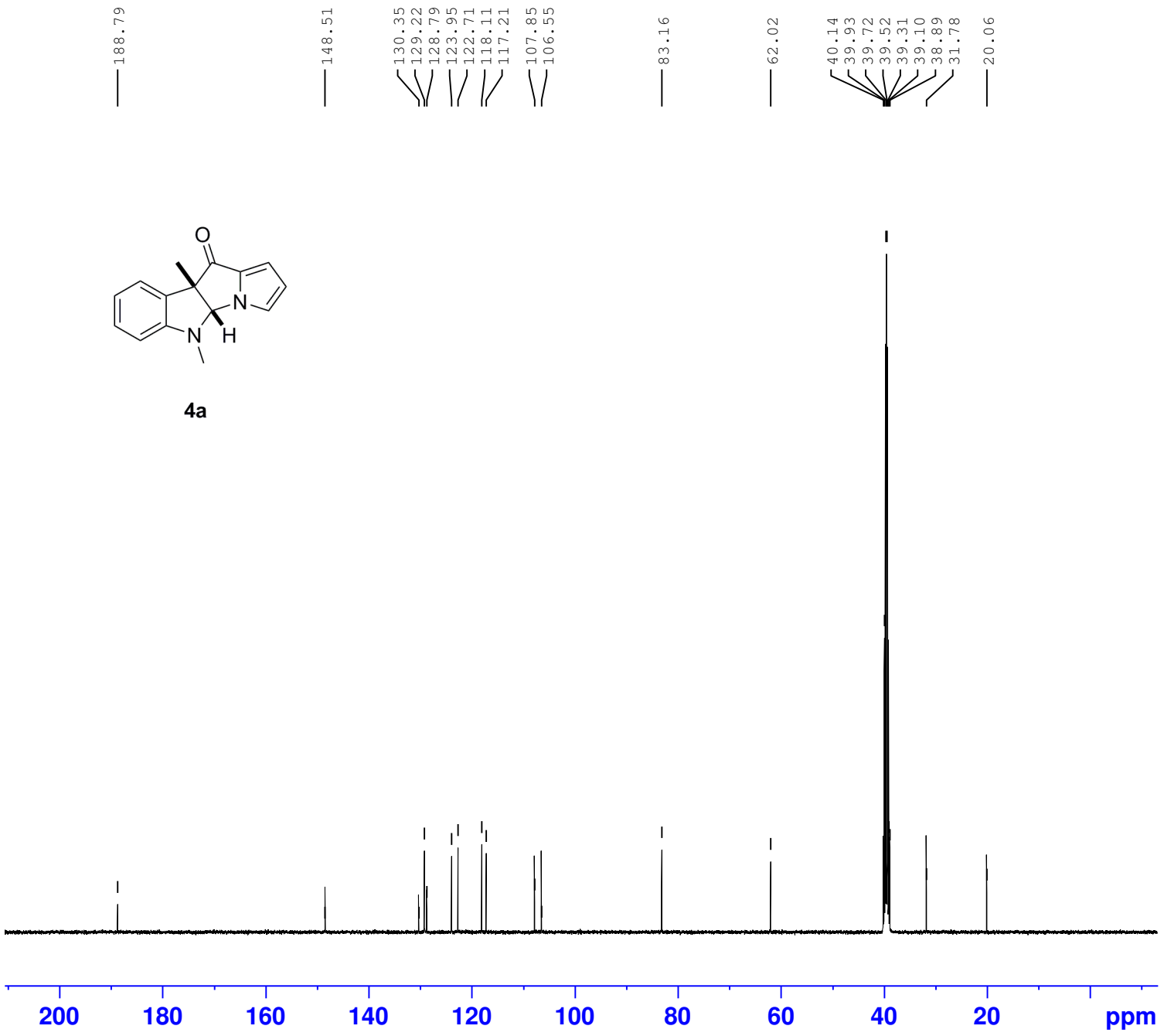



```
Current Data Parameters
NAME            zqq-73iv
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161024
Time               21.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 114.84
DW                 62.400 usec
DE                  6.50 usec
TE                 298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.31 usec
PLW1         16.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



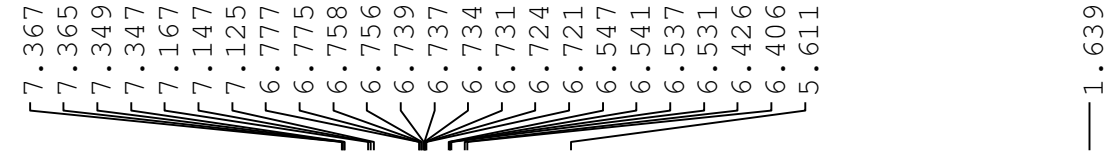
Current Data Parameters
NAME zqq-73iv
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20161024
Time 22.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

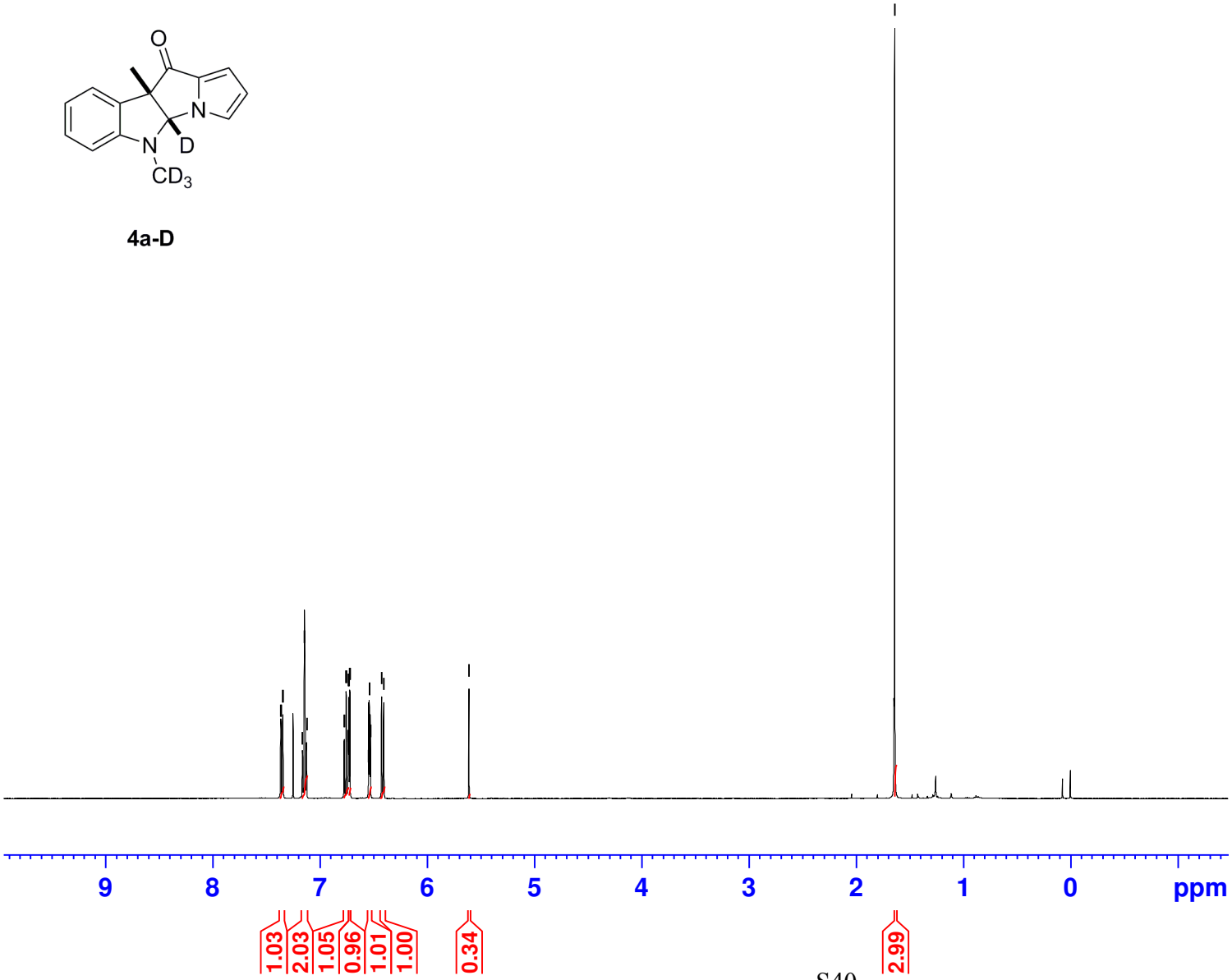
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.09 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.17121001 W
PLW13 0.13868000 W

F2 - Processing parameters
SI 32768
SF 100.6128145 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



4a-D

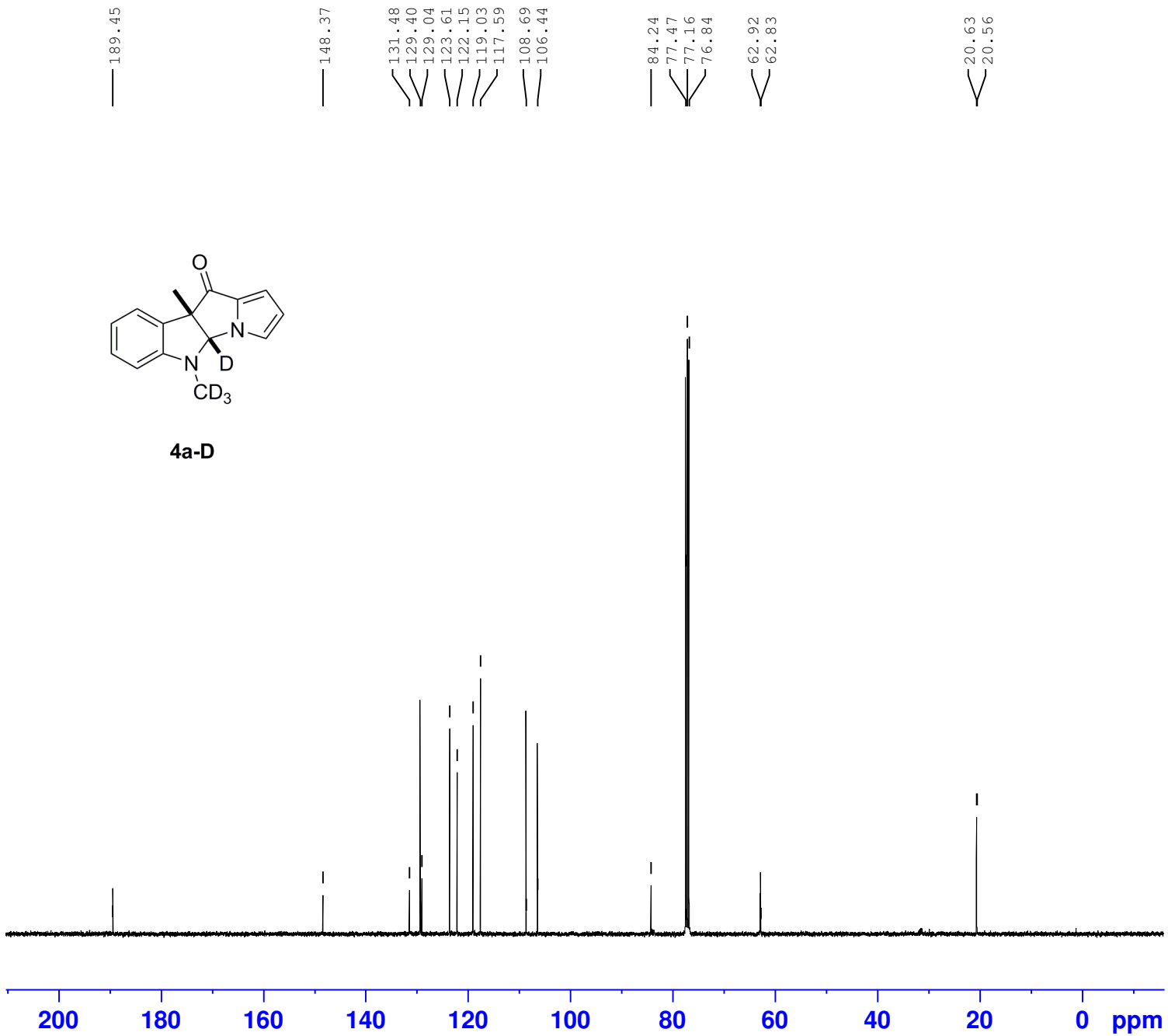



```
Current Data Parameters
NAME                  ZQQ-126
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20161217
Time                    12.09
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                     125.55
DW                     62.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                      9.31 usec
PLW1             16.00000000 W

F2 - Processing parameters
SI                     65536
SF               400.1300128 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



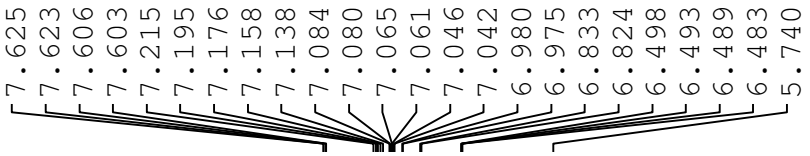
Current Data Parameters
NAME ZQQ-126-C
EXPNO 10
PROCNO 1

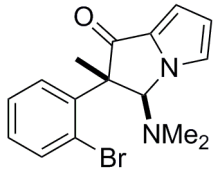
F2 - Acquisition Parameters
Date_ 20161219
Time 10.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.09 usec
PLW1 73.00000000 W

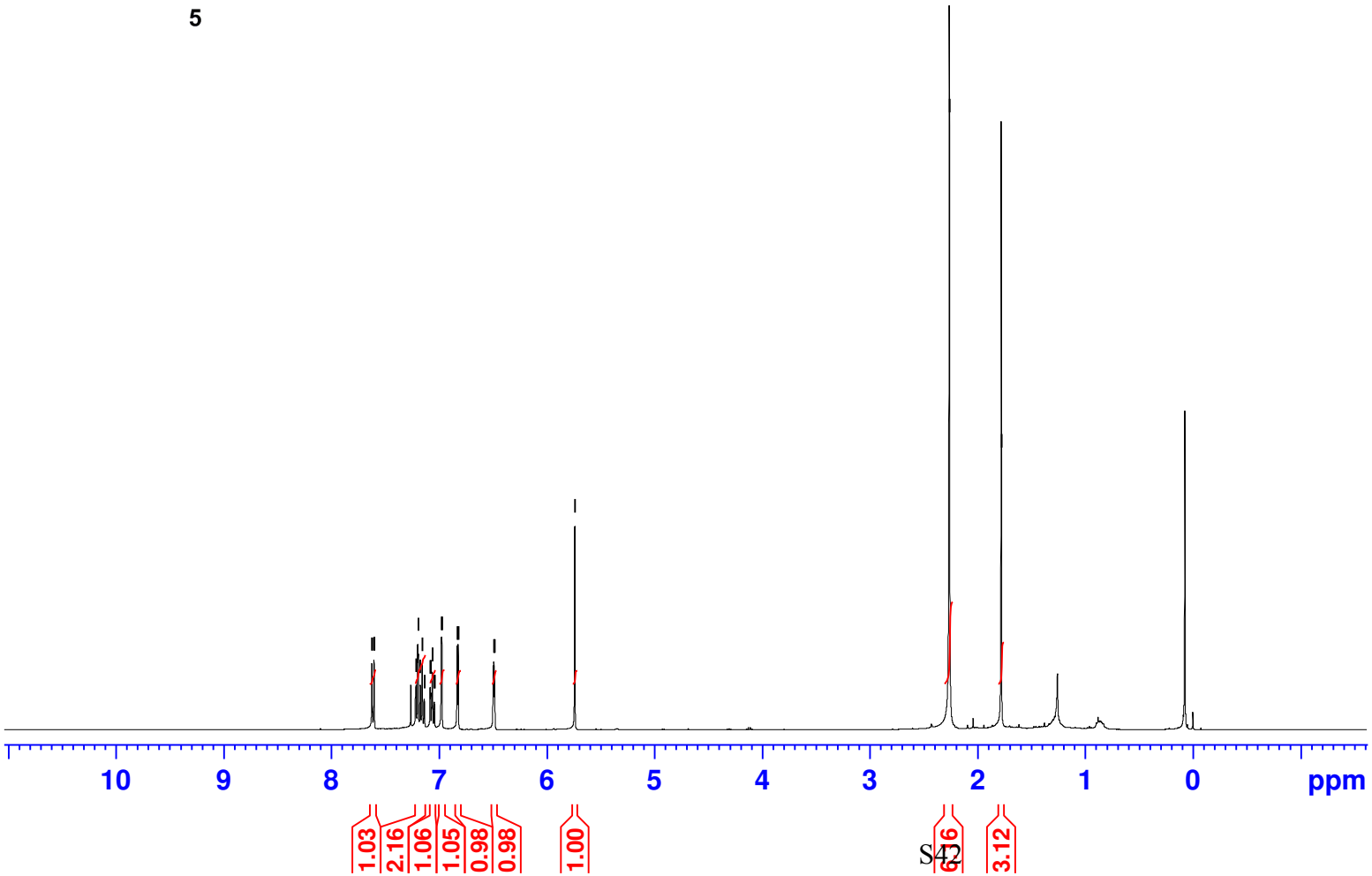
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.17121001 W
PLW13 0.13868000 W

F2 - Processing parameters
SI 32768
SF 100.6127587 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





5

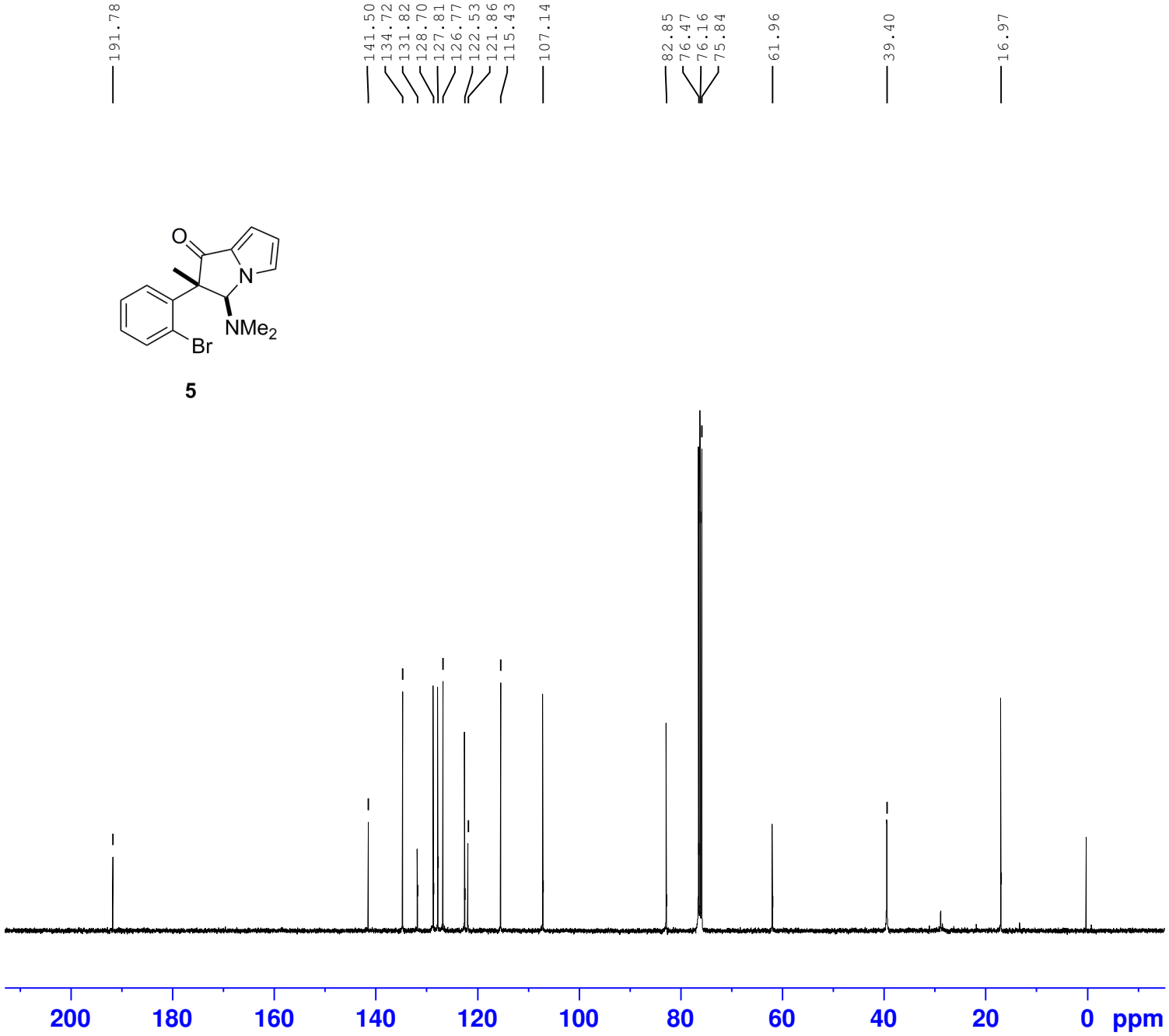


Current Data Parameters
NAME ZQQ-83B
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170402
Time 12.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 69.41
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.09 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



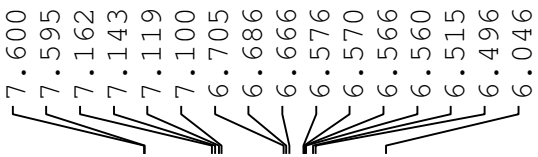
Current Data Parameters
NAME ZQQ-83B
EXPNO 11
PROCNO 1

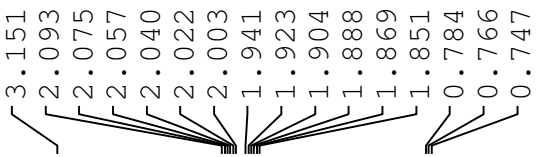
F2 - Acquisition Parameters
Date_ 20170402
Time 13.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

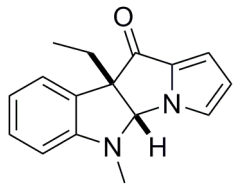
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

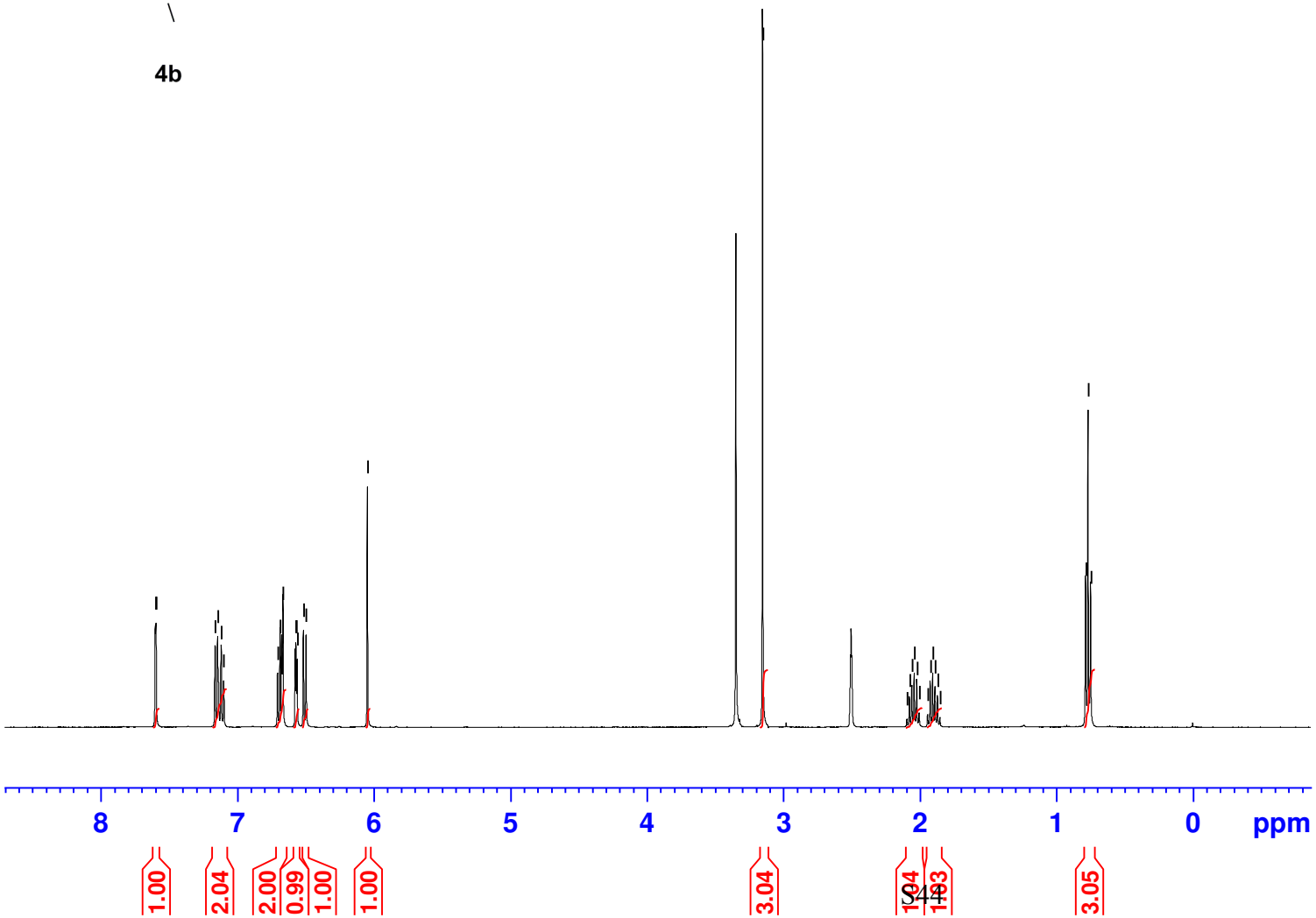
F2 - Processing parameters
SI 32768
SF 100.6128588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







4b

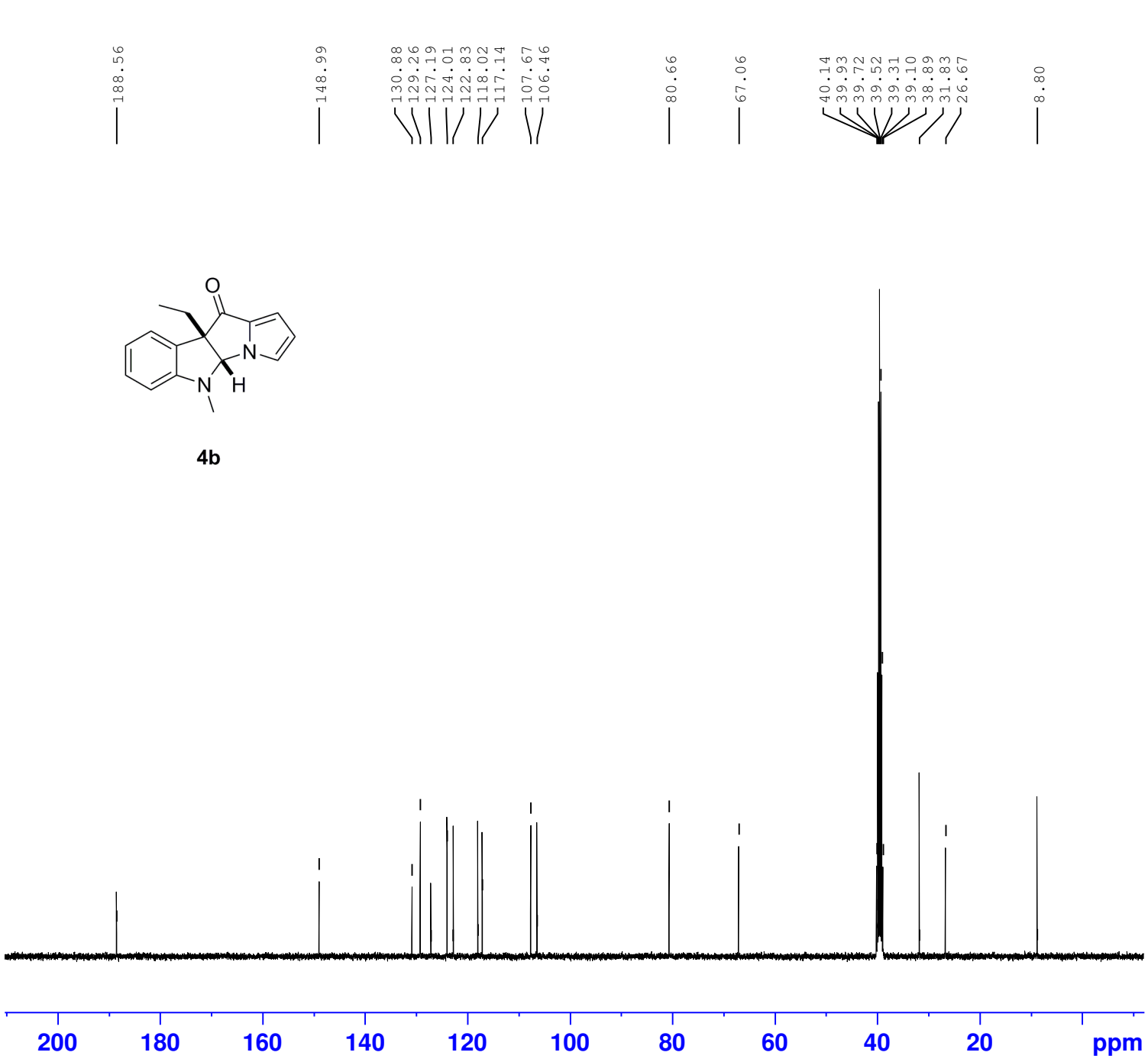



```
Current Data Parameters
NAME                  ZQQ-86B
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_                20161103
Time                    10.33
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                 DMSO
NS                         16
DS                          2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                 4.0894465 sec
RG                      91.76
DW                     62.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                       9.31 usec
PLW1             16.00000000 W

F2 - Processing parameters
SI                     65536
SF               400.1300024 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



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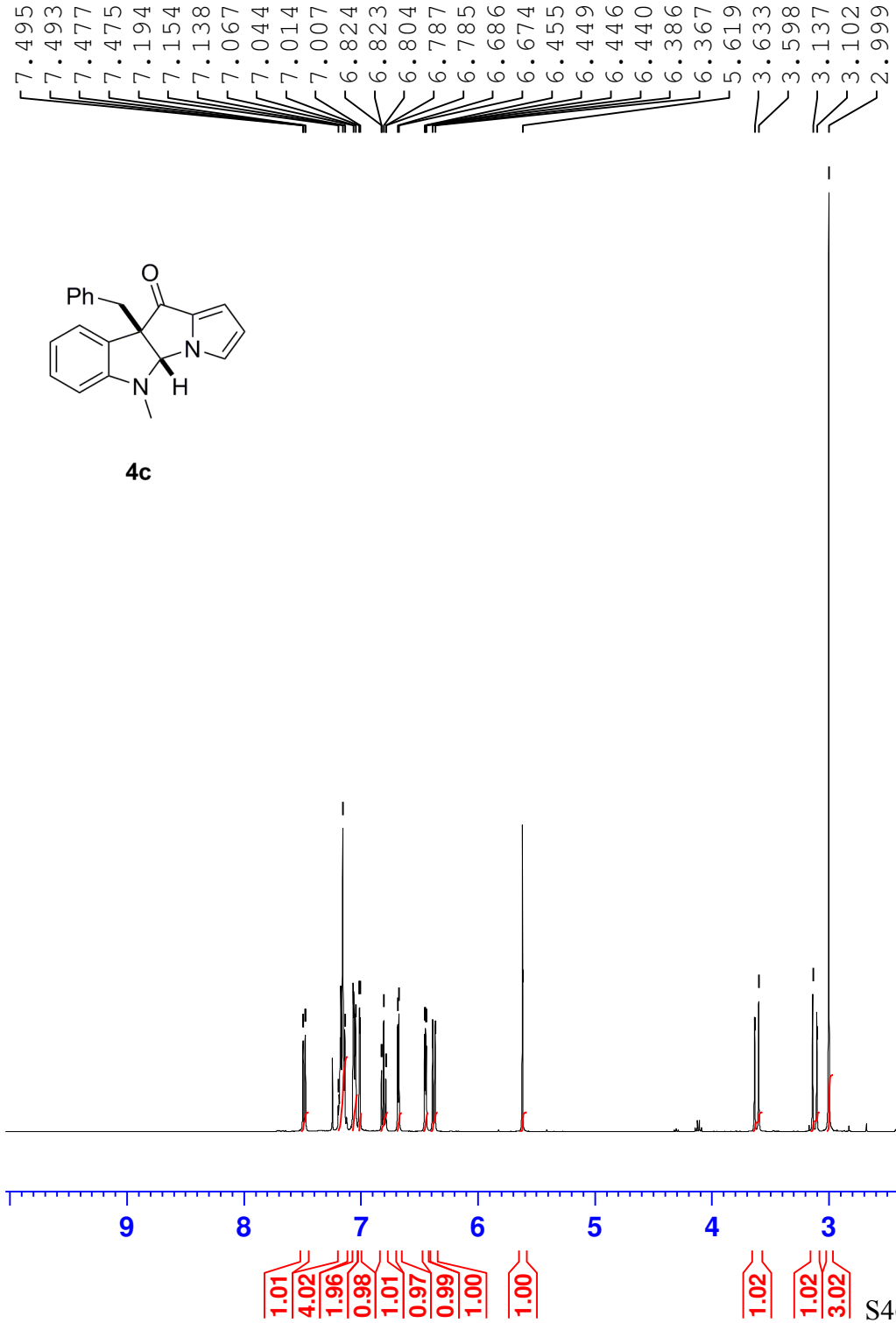
```
Current Data Parameters
NAME                  ZQQ-86B
EXPNO                      11
PROCNO                      1

F2 - Acquisition Parameters
Date_               20161103
Time                   10.45
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                  DMSO
NS                         199
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     207.09
DW                     20.800 usec
DE                      6.50 usec
TE                     298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1             100.6228293 MHz
NUC1                     13C
P1                      9.09 usec
PLW1             73.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                   90.00 usec
PLW2             16.00000000 W
PLW12             0.17121001 W
PLW13             0.13868000 W

F2 - Processing parameters
SI                       32768
SF               100.6128163 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

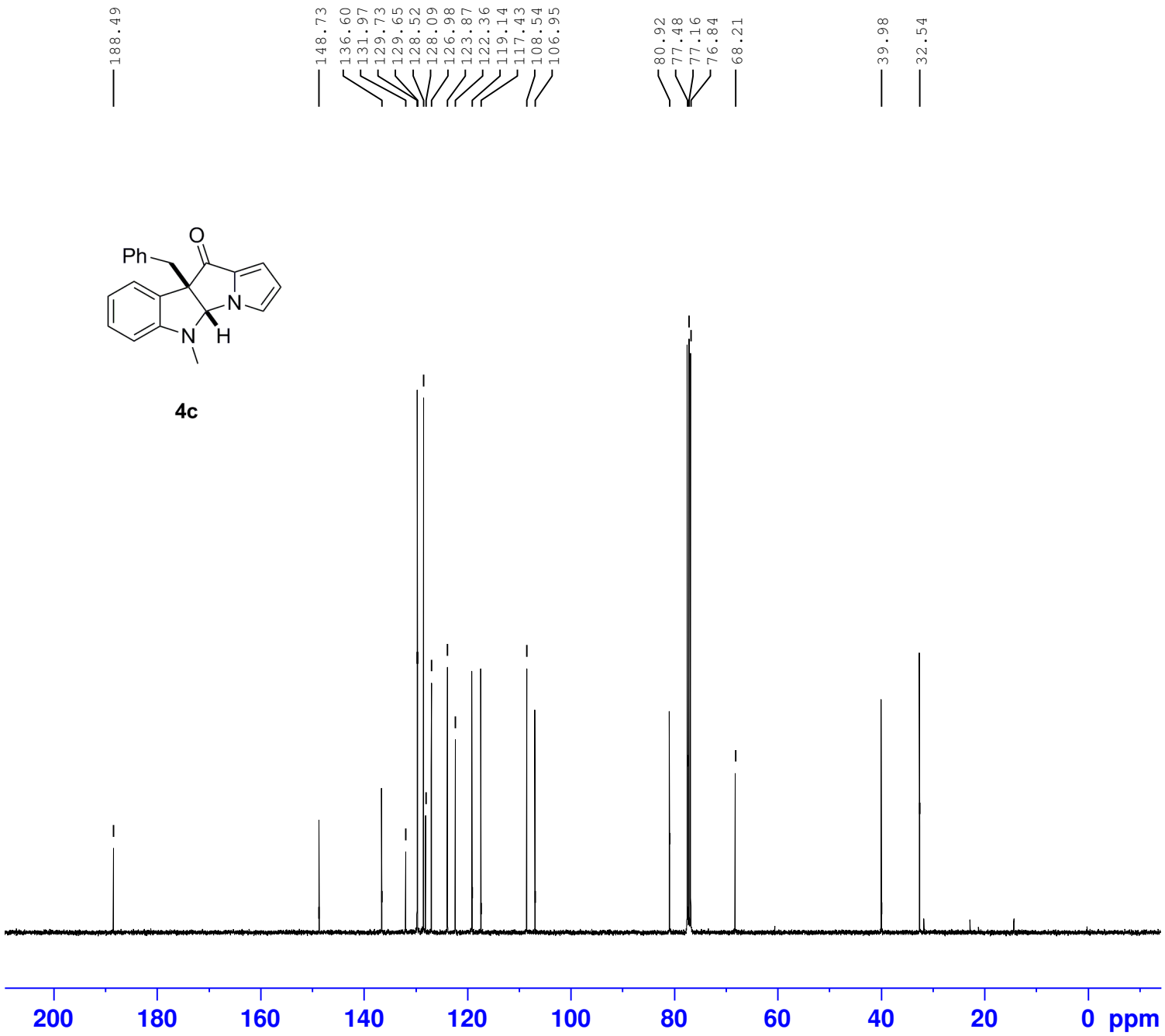



```
Current Data Parameters
NAME                  ZQQ-142
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20161230
Time                    9.00
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                     82.33
DW                     62.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                      9.31 usec
PLW1            16.00000000 W

F2 - Processing parameters
SI                     65536
SF               400.1300159 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



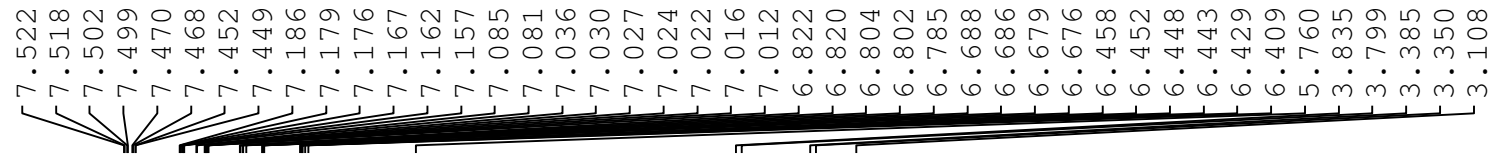
Current Data Parameters
NAME ZQQ-142
EXPNO 11
PROCNO 1

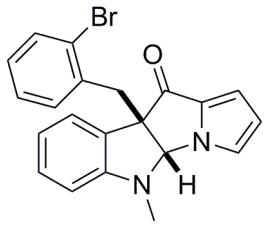
F2 - Acquisition Parameters
Date_ 20161230
Time 9.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.09 usec
PLW1 73.00000000 W

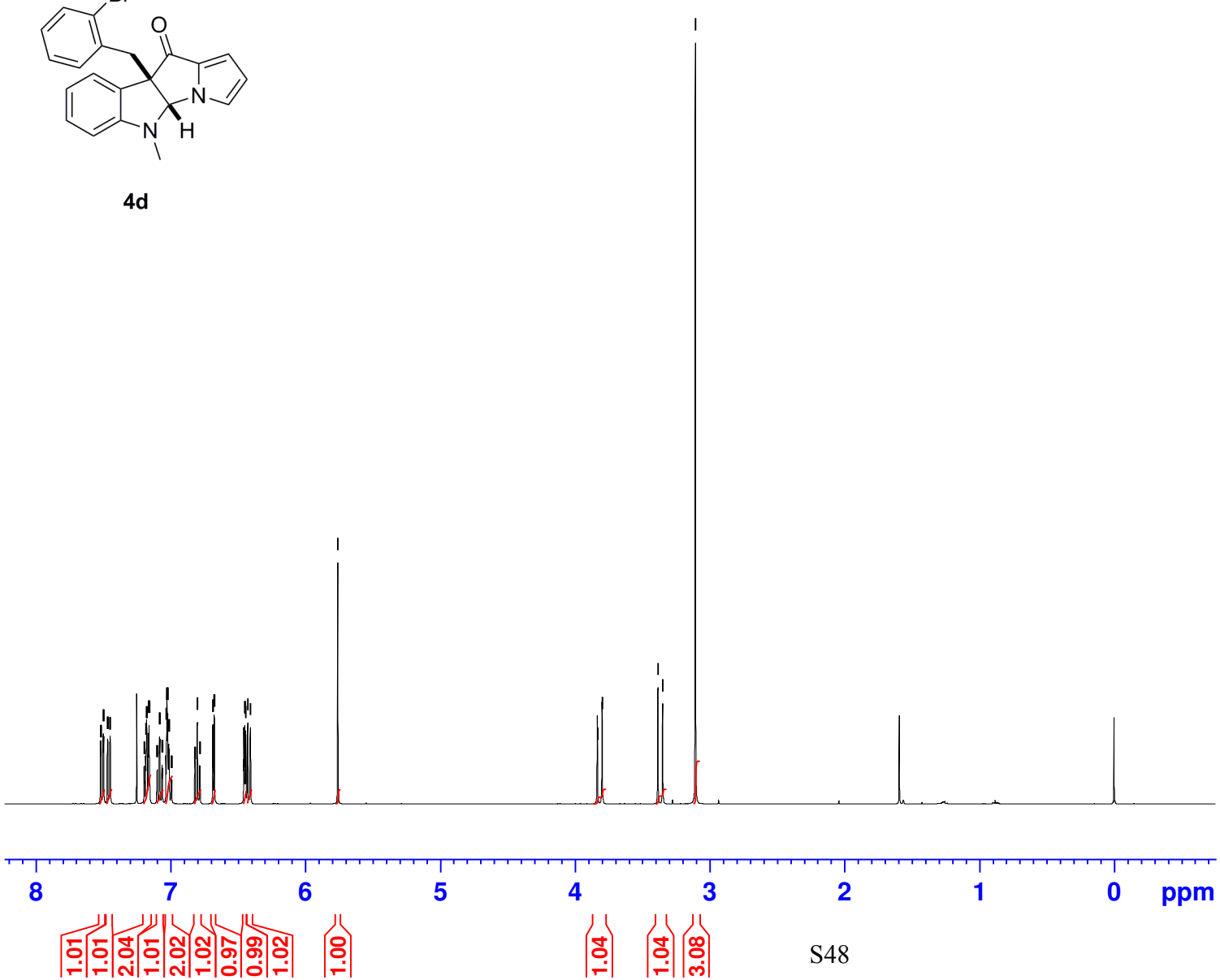
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.17121001 W
PLW13 0.13868000 W

F2 - Processing parameters
SI 32768
SF 100.6127597 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





4d

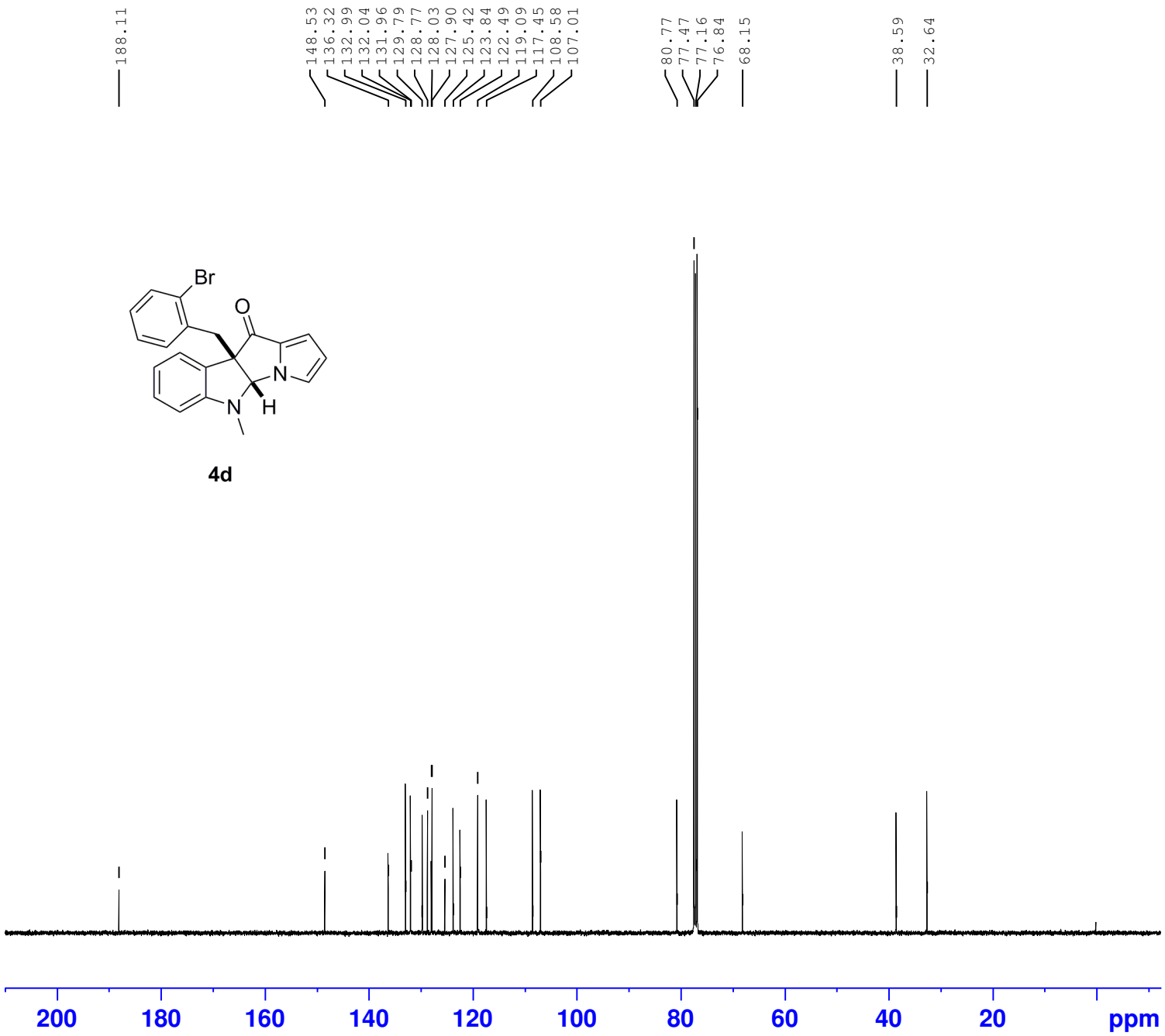



```
Current Data Parameters
NAME                  ZQQ-154B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170108
Time                     21.50
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                      125.55
DW                      62.400 usec
DE                       6.50 usec
TE                      298.0 K
D1                  1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                       1H
P1                       9.31 usec
PLW1           16.00000000 W

F2 - Processing parameters
SI                      65536
SF              400.1300122 MHz
WDW                        EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```



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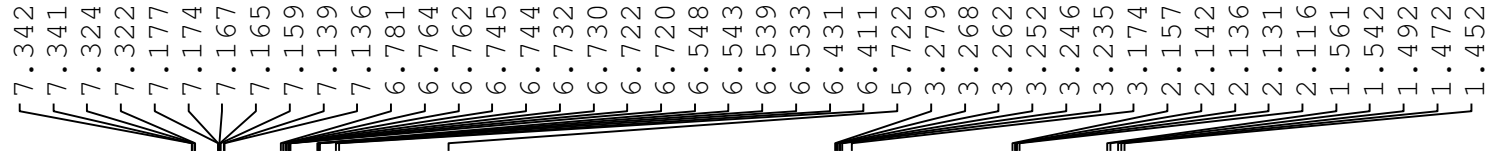
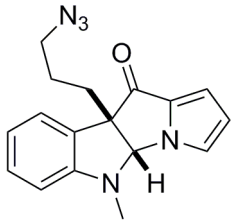
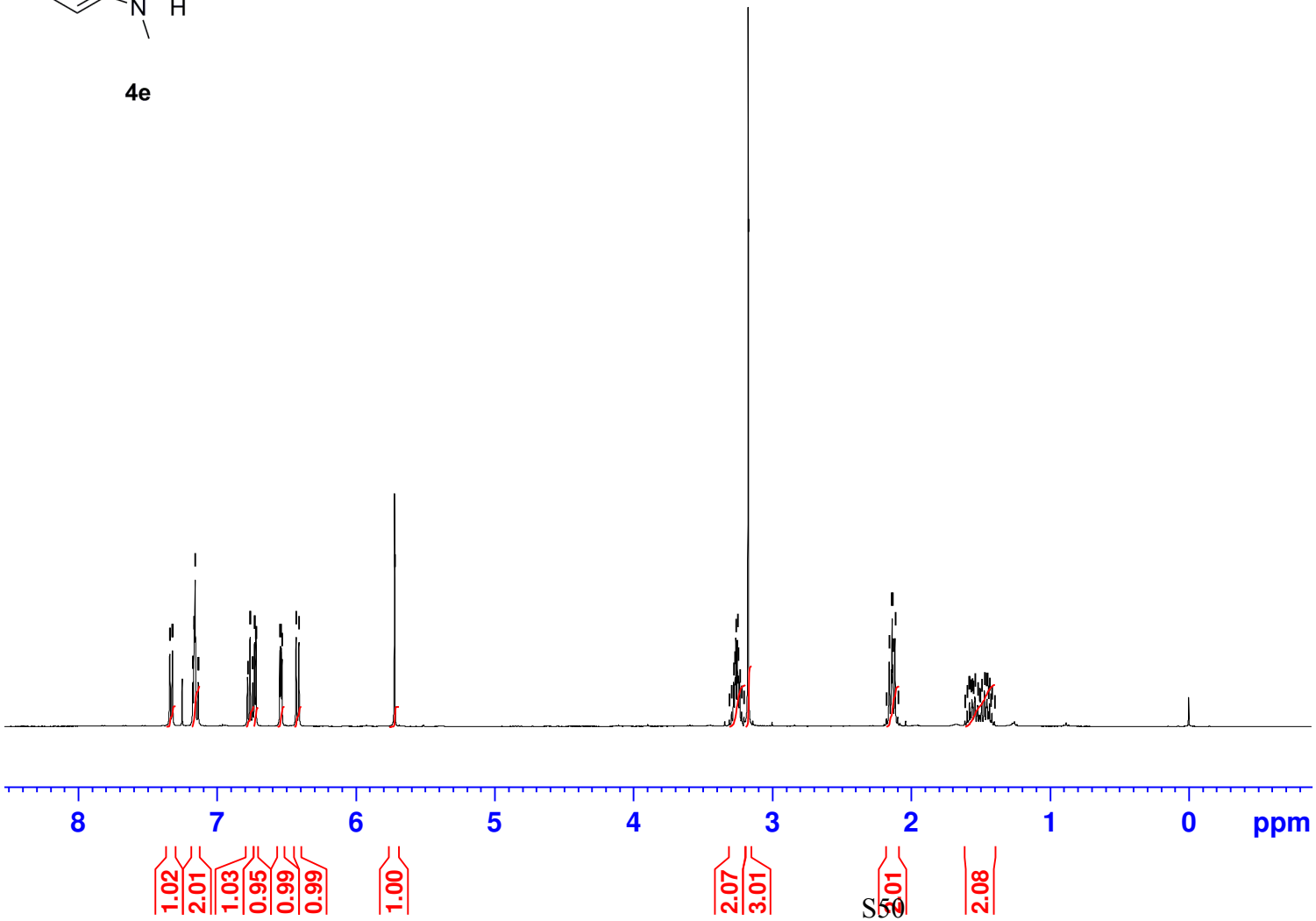
```
Current Data Parameters
NAME            ZQQ-154B
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170108
Time               22.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                207.09
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                  9.09 usec
PLW1         73.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2         16.00000000 W
PLW12         0.17121001 W
PLW13         0.13868000 W

F2 - Processing parameters
SI                  32768
SF           100.6127571 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

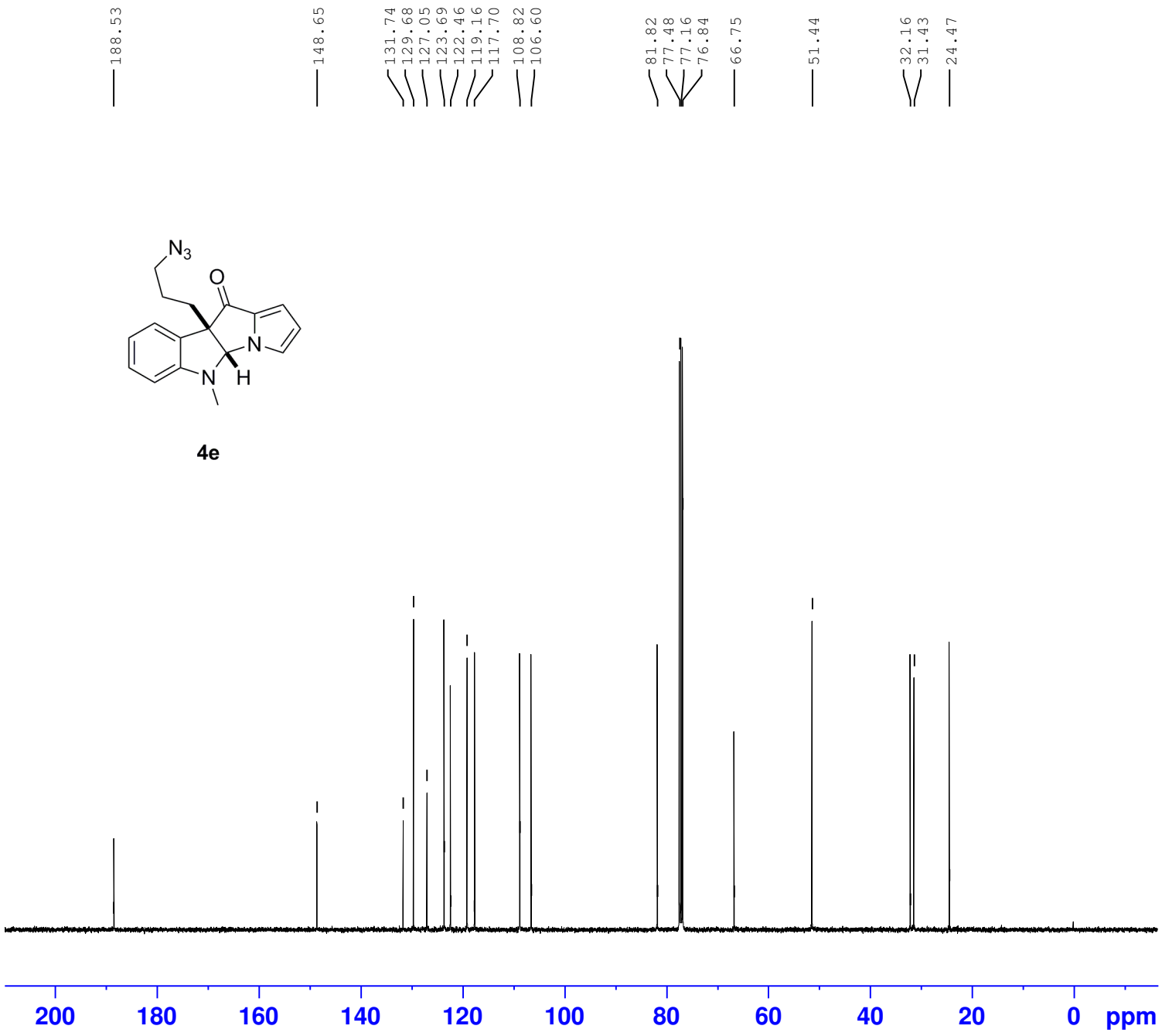




4e




```
Current Data Parameters
NAME            ZQQ-185B
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170428
Time               20.32
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 82.33
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.09 usec
PLW1        16.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.1300130 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



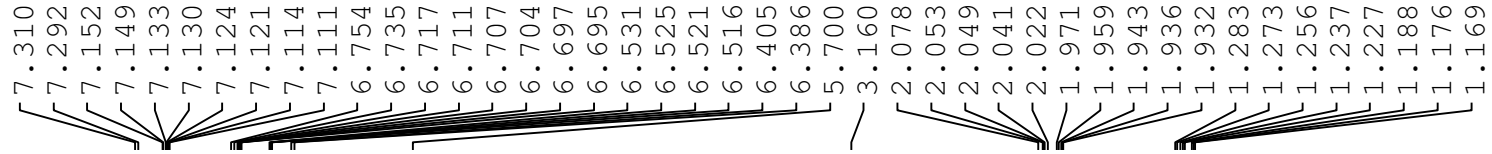
Current Data Parameters
NAME ZQQ-185B
EXPNO 11
PROCNO 1

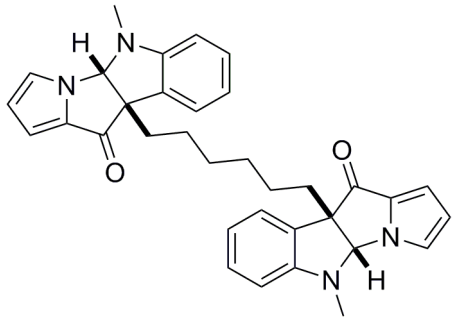
F2 - Acquisition Parameters
Date_ 20170428
Time 21.32
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

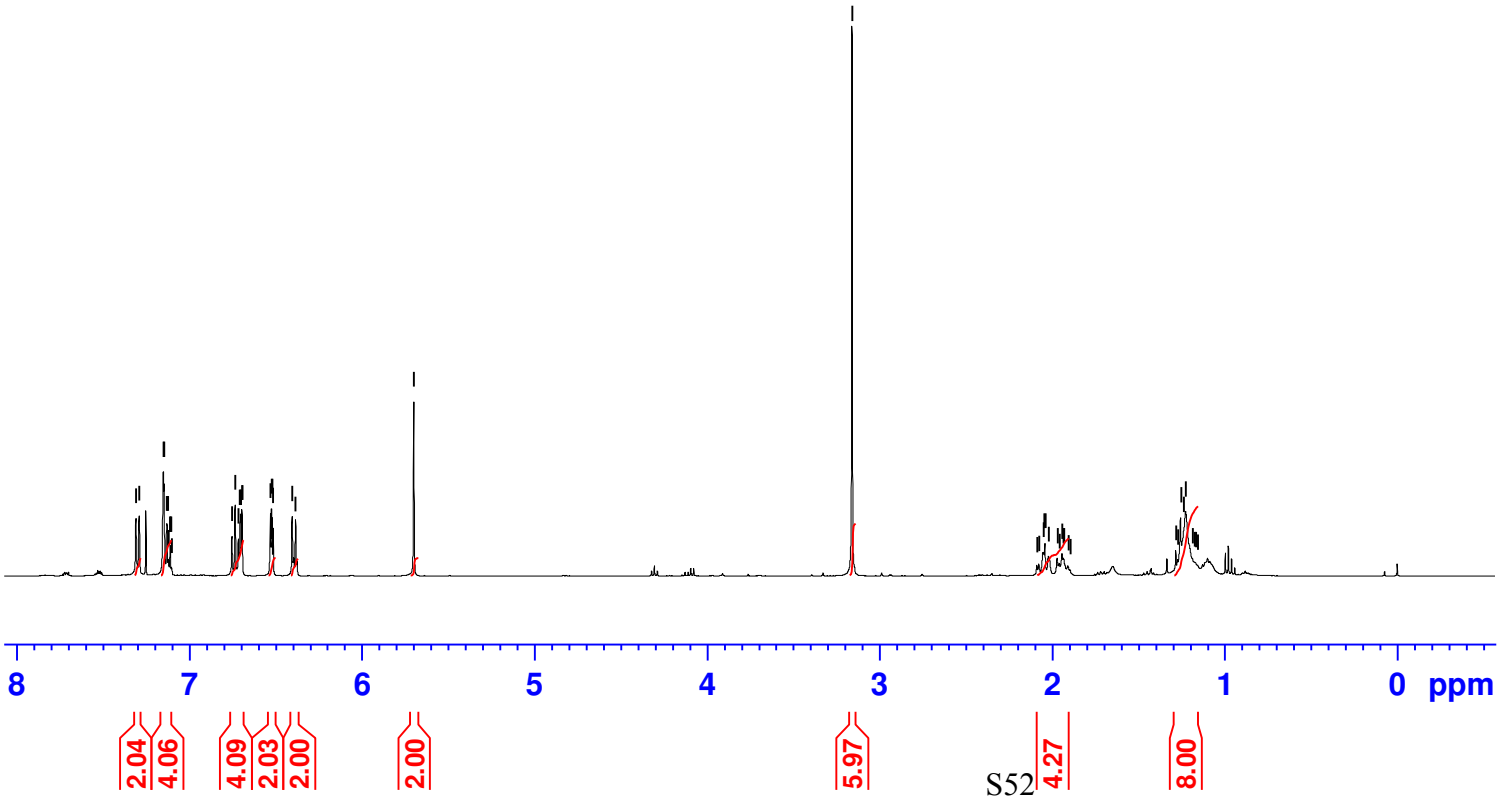
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127600 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

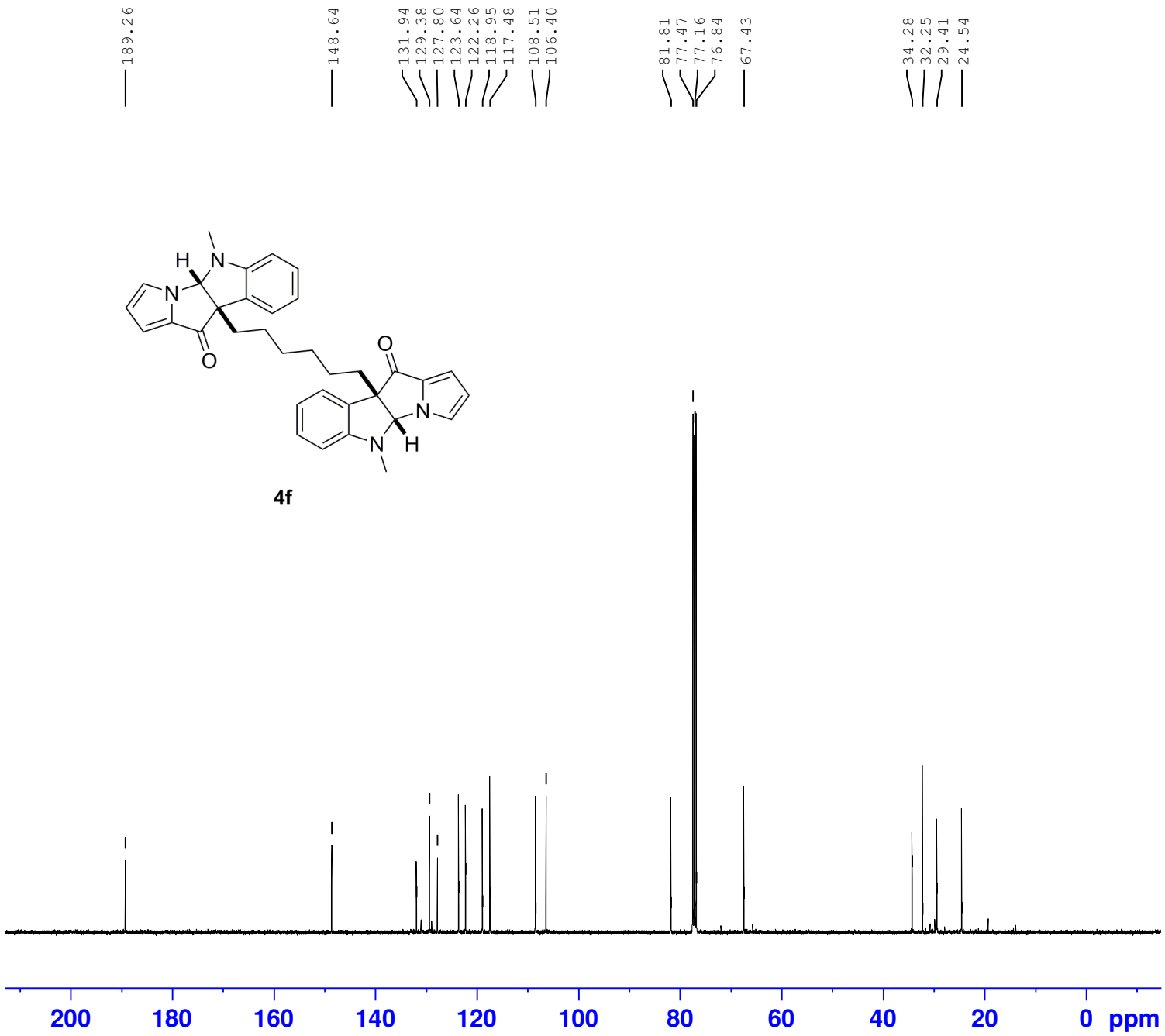




4f




```
Current Data Parameters
NAME                  ZQQ-172B
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170215
Time                     12.01
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                       91.76
DW                     62.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1              400.1324710 MHz
NUC1                        1H
P1                       9.09 usec
PLW1             16.00000000 W

F2 - Processing parameters
SI                      65536
SF                400.1300125 MHz
WDW                        EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```



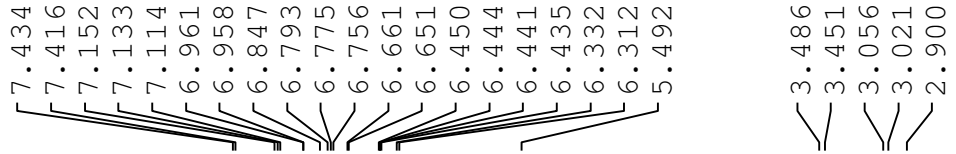
Current Data Parameters
NAME ZQQ-172B
EXPNO 11
PROCNO 1

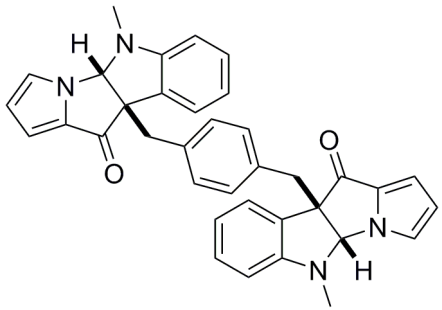
F2 - Acquisition Parameters
Date_ 20170215
Time 13.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

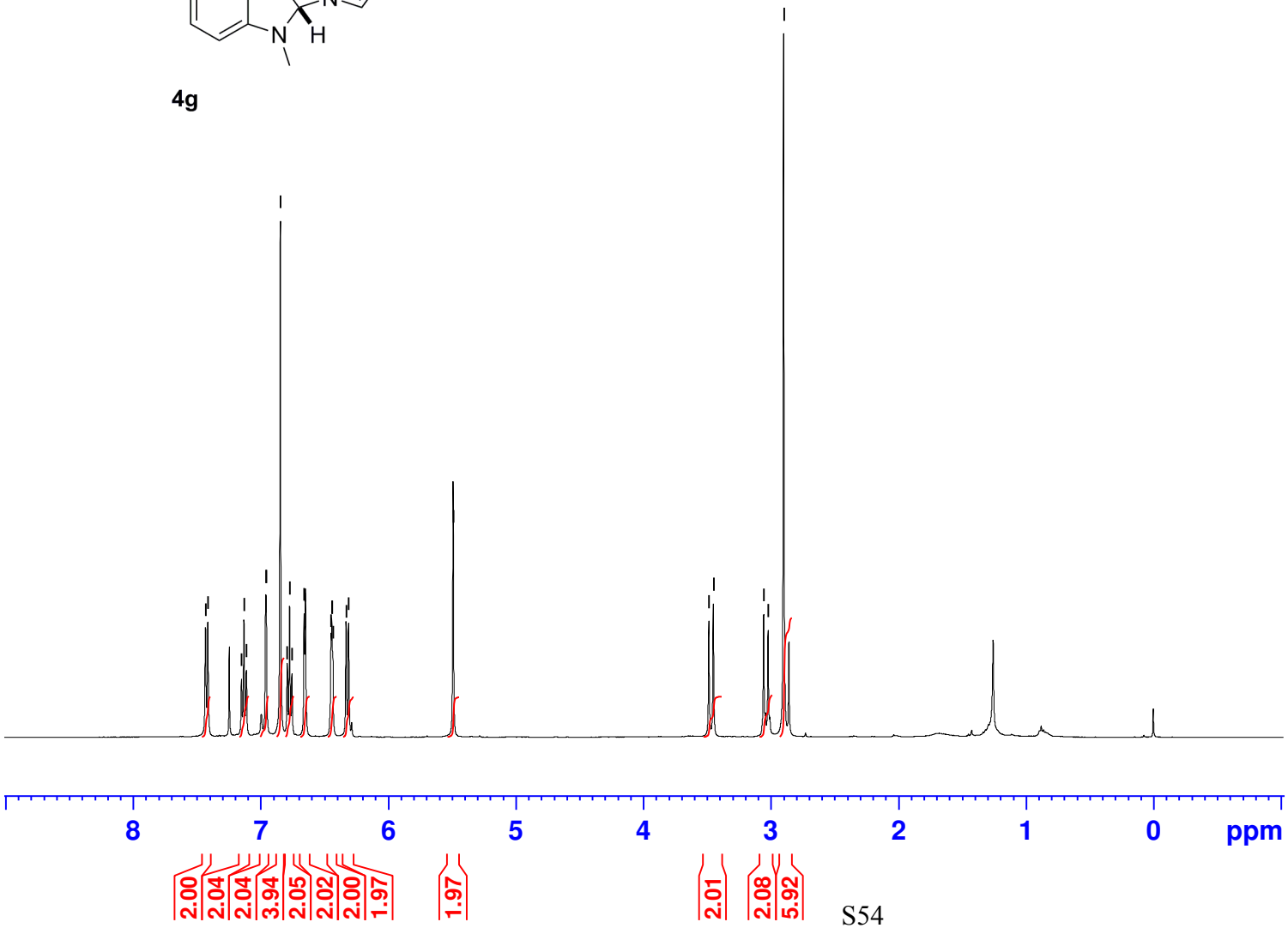
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127585 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

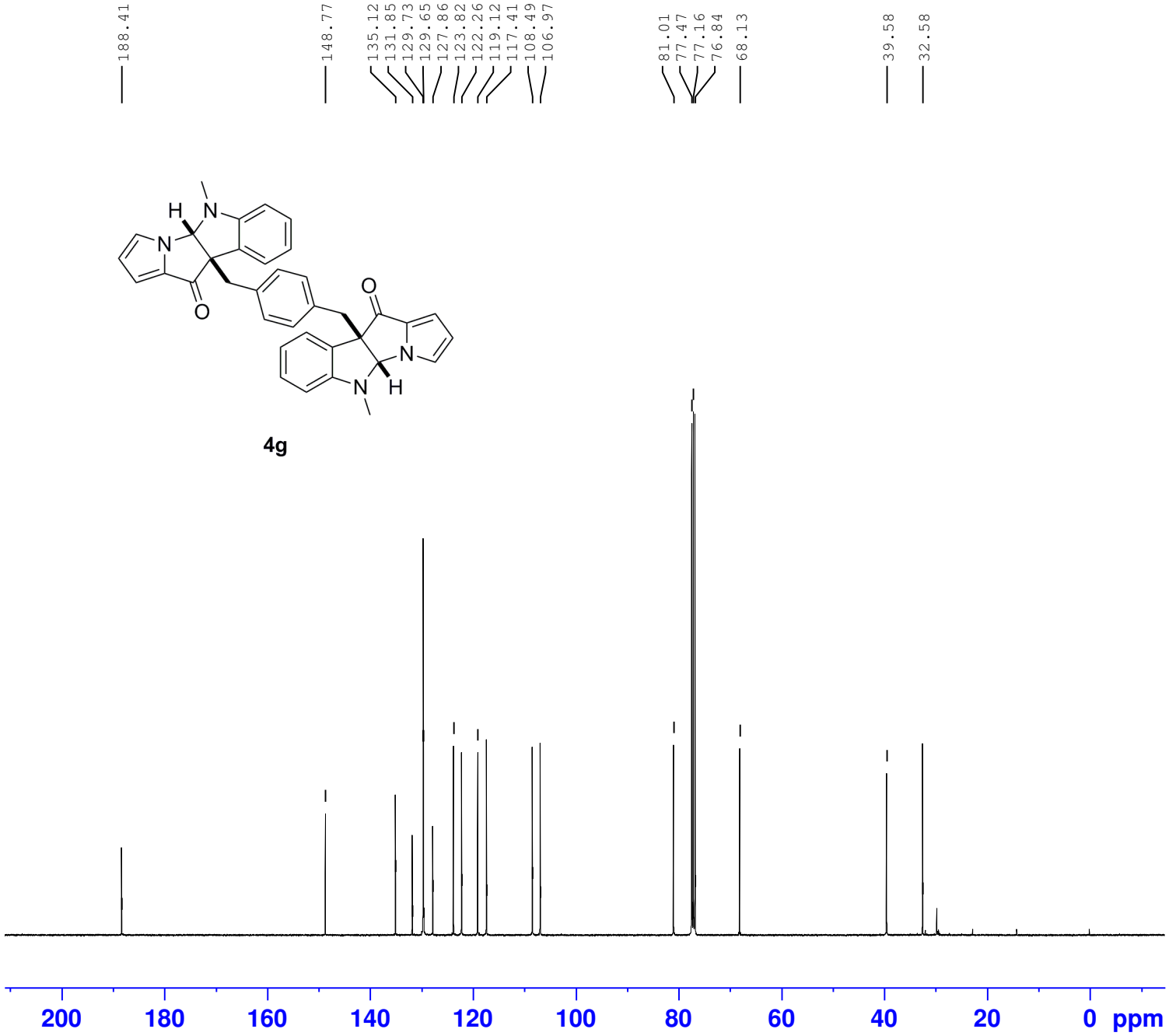




4g




```
Current Data Parameters
NAME                  ZQQ-201D
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170418
Time                      2.51
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                       82.33
DW                      62.400 usec
DE                       6.50 usec
TE                      300.3 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1              400.1324710 MHz
NUC1                        1H
P1                       9.09 usec
PLW1             16.00000000 W

F2 - Processing parameters
SI                      65536
SF                400.1300155 MHz
WDW                        EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```



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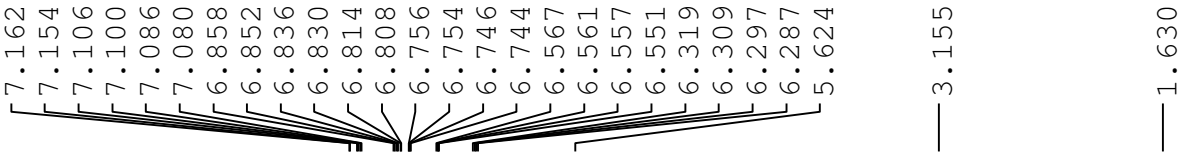
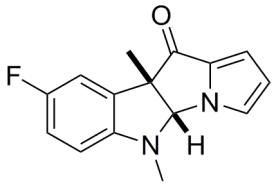
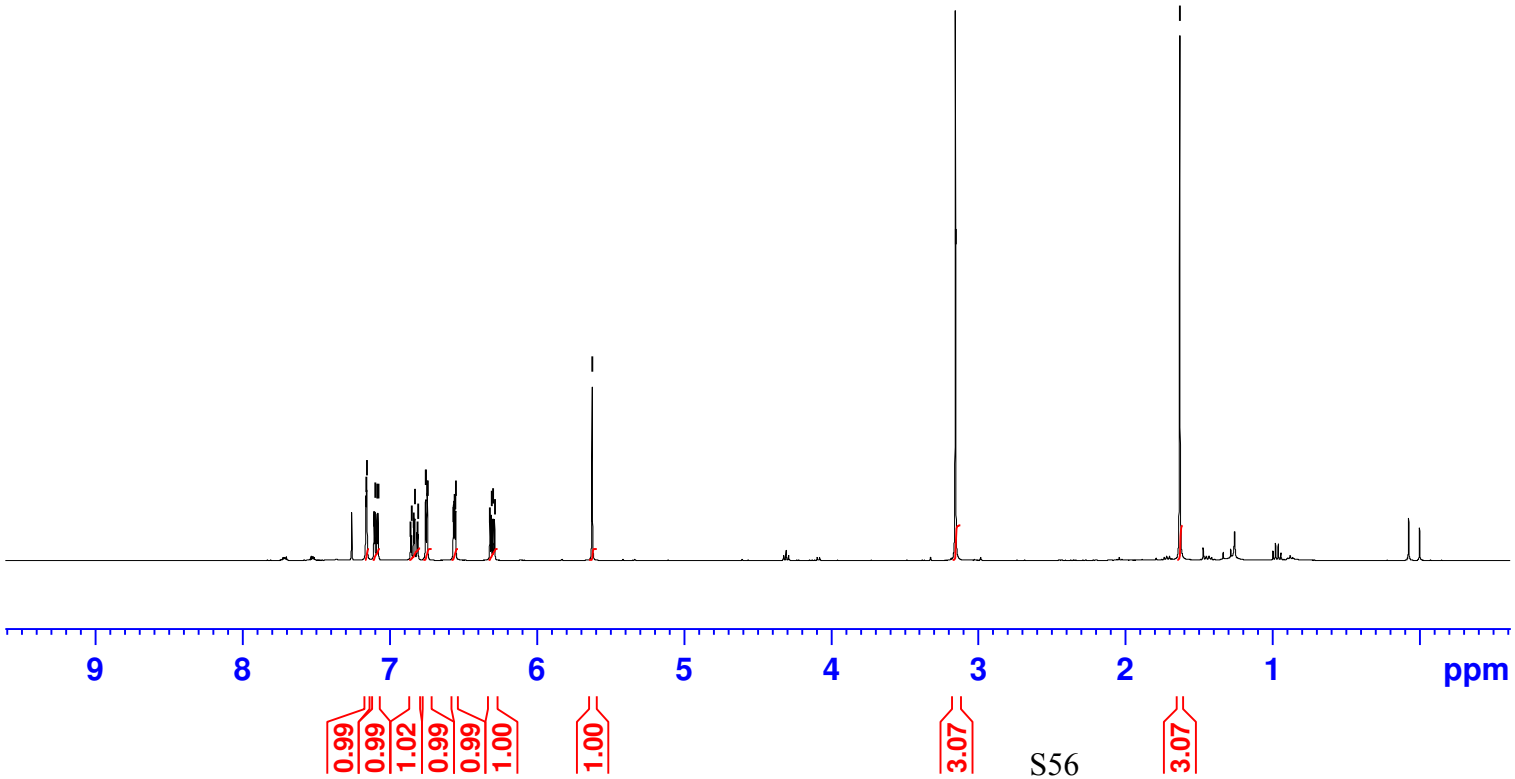
```
Current Data Parameters
NAME                  ZQQ-201D
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170418
Time                     11.24
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       8973
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     207.09
DW                     20.800 usec
DE                      6.50 usec
TE                     301.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               100.6228293 MHz
NUC1                       13C
P1                        9.65 usec
PLW1               73.00000000 W

======== CHANNEL f2 ========
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               16.00000000 W
PLW12               0.16322000 W
PLW13               0.13220000 W

F2 - Processing parameters
SI                        32768
SF                 100.6127595 MHz
WDW                          EM
SSB             0
LB                        1.00 Hz
GB              0
PC                        1.40
```





4h

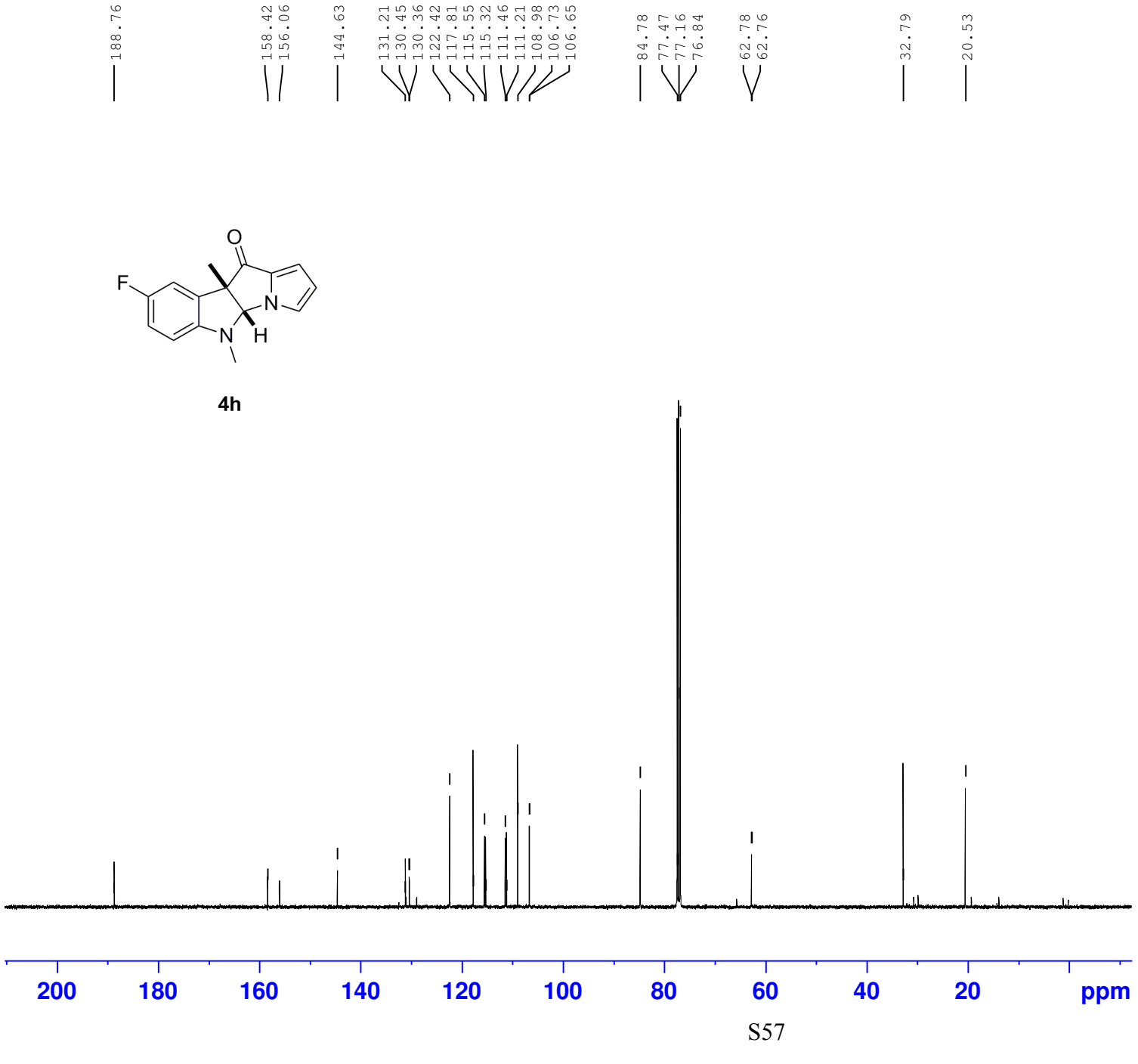


Current Data Parameters
NAME ZQQ-162
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170114
Time 19.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 114.84
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.31 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300103 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4h

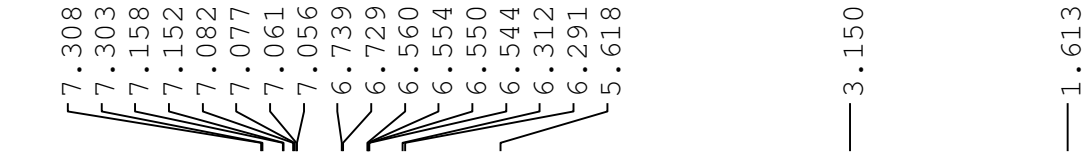
Current Data Parameters
NAME ZQQ-162
EXPNO 11
PROCNO 1

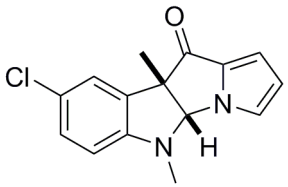
F2 - Acquisition Parameters
Date_ 20170114
Time 20.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.09 usec
PLW1 73.00000000 W

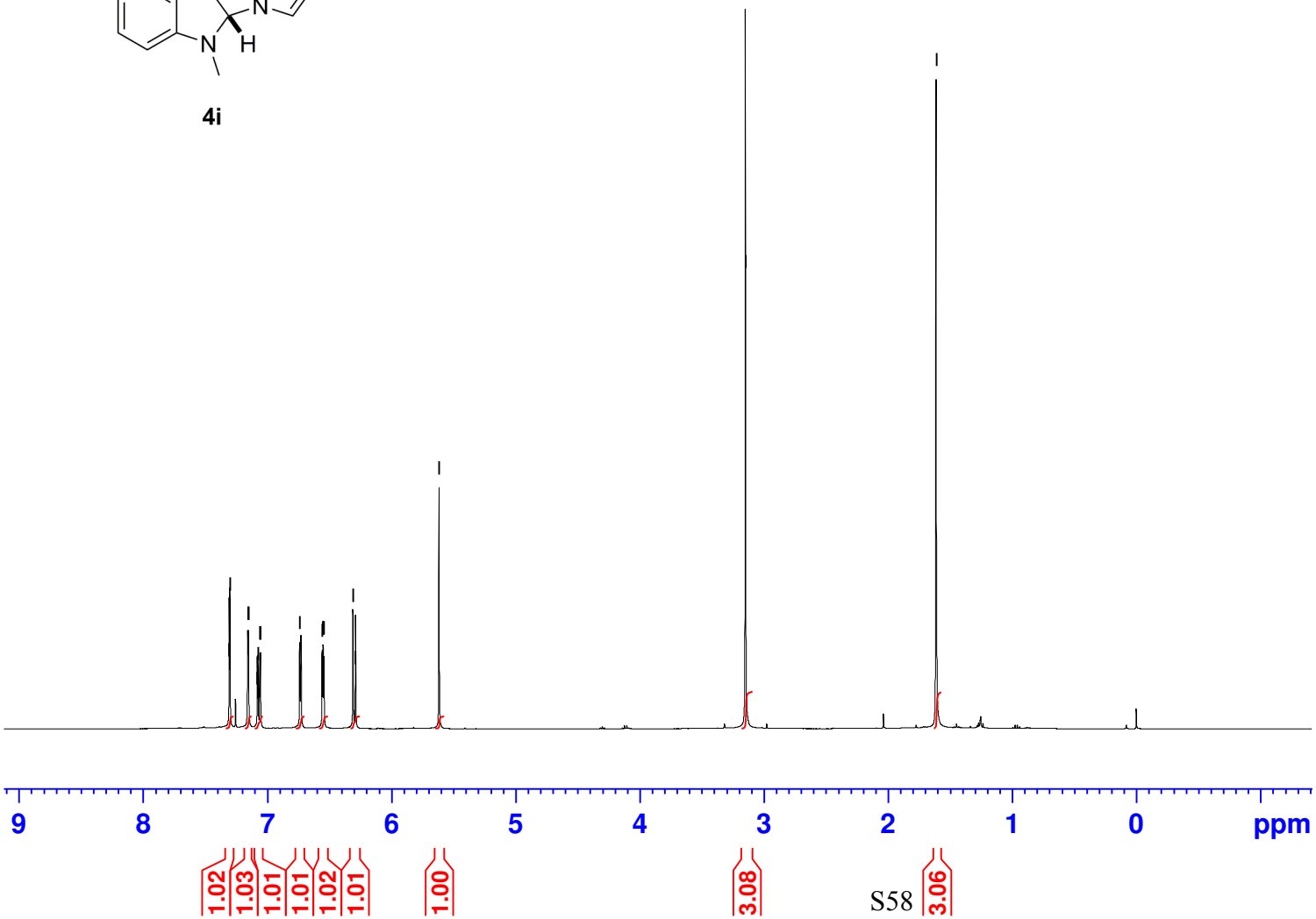
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.17121001 W
PLW13 0.13868000 W

F2 - Processing parameters
SI 32768
SF 100.6127575 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

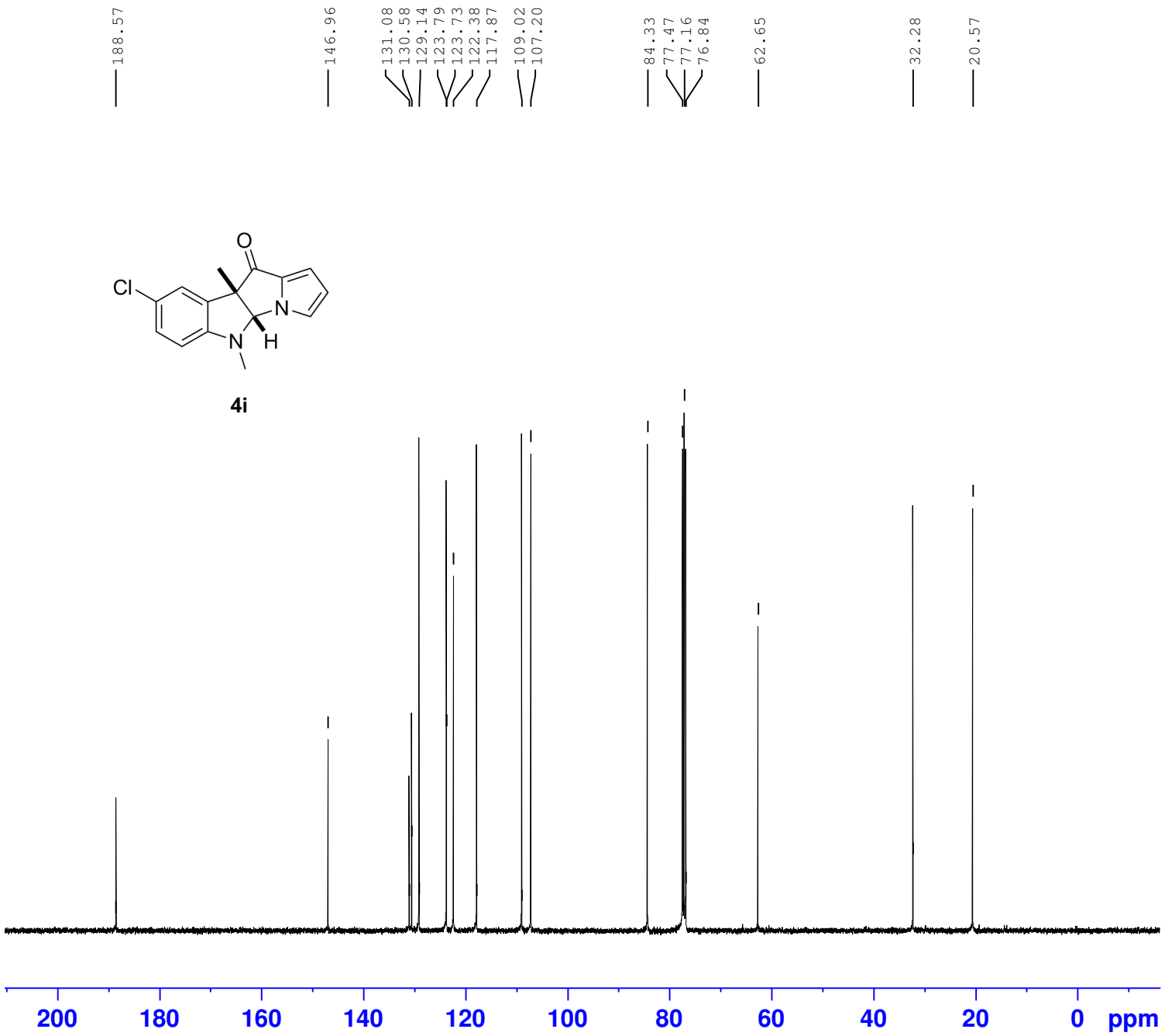




4i




```
Current Data Parameters
NAME                  ZQQ-163
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170402
Time                   20.11
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                    69.41
DW                  62.400 usec
DE                   6.50 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                     1H
P1                    9.09 usec
PLW1           16.00000000 W

F2 - Processing parameters
SI                    65536
SF             400.1300114 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



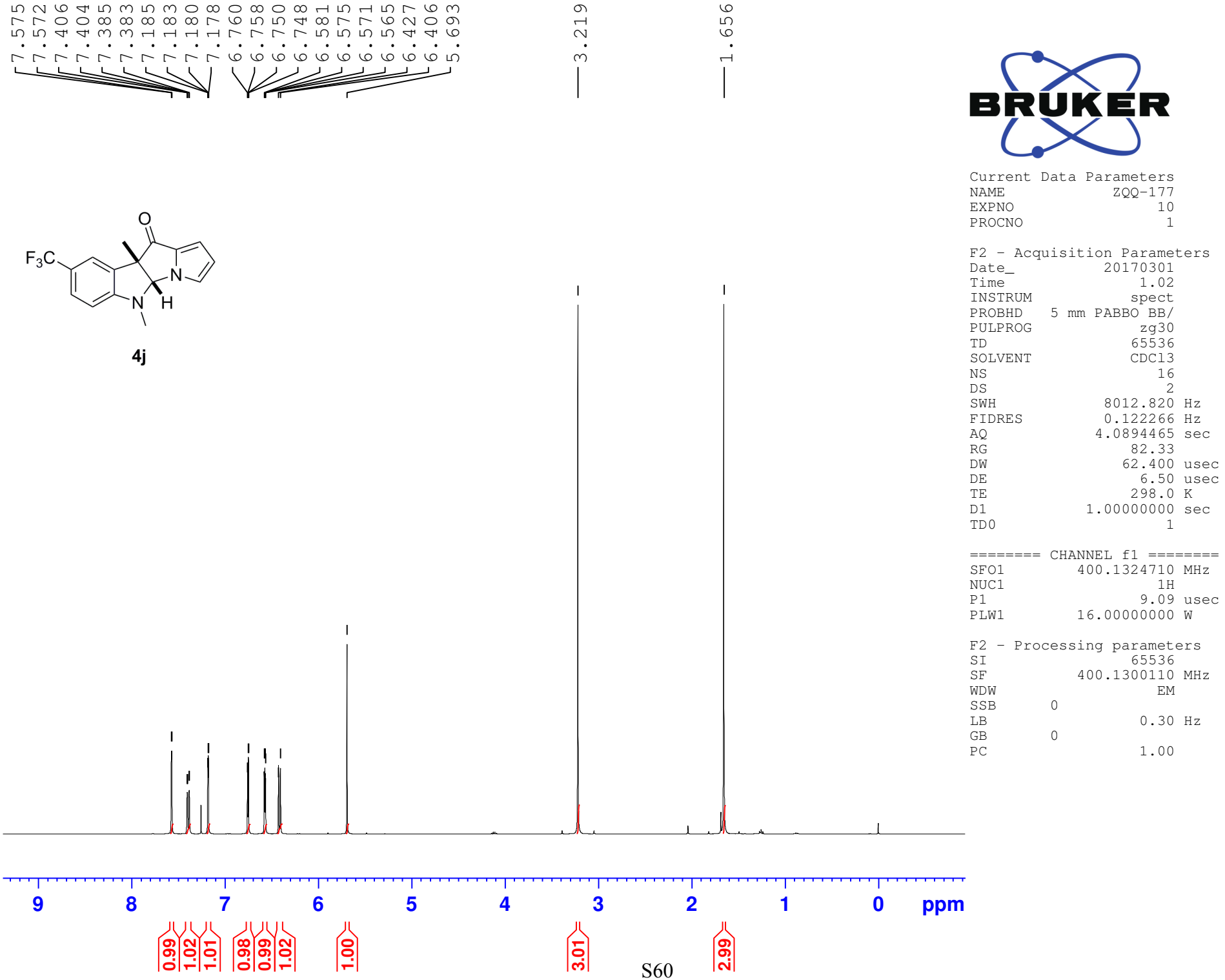
Current Data Parameters
NAME ZQQ-163
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170402
Time 21.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127625 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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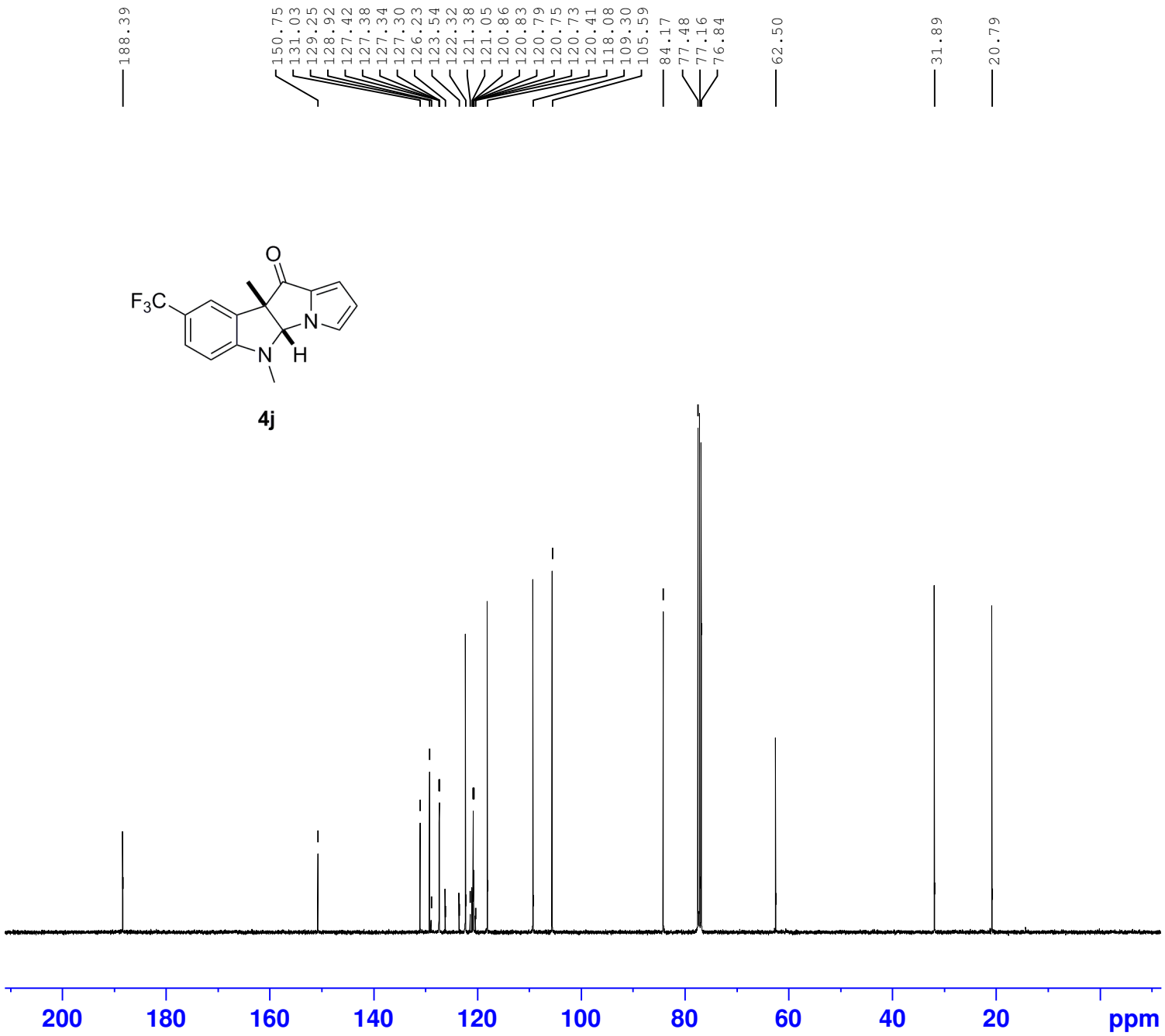
Current Data Parameters
NAME ZQQ-177
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170301
Time 1.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 82.33
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.09 usec
PLW1 16.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300110 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



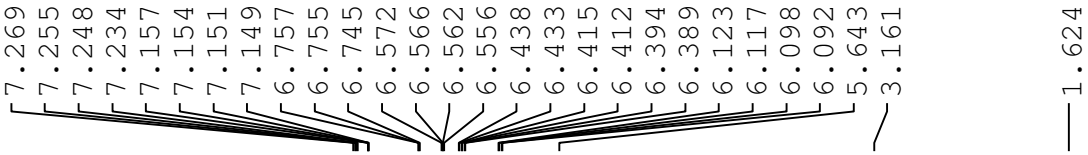
Current Data Parameters
NAME ZQQ-177
EXPNO 11
PROCNO 1

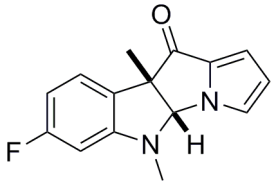
F2 - Acquisition Parameters
Date_ 20170301
Time 2.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

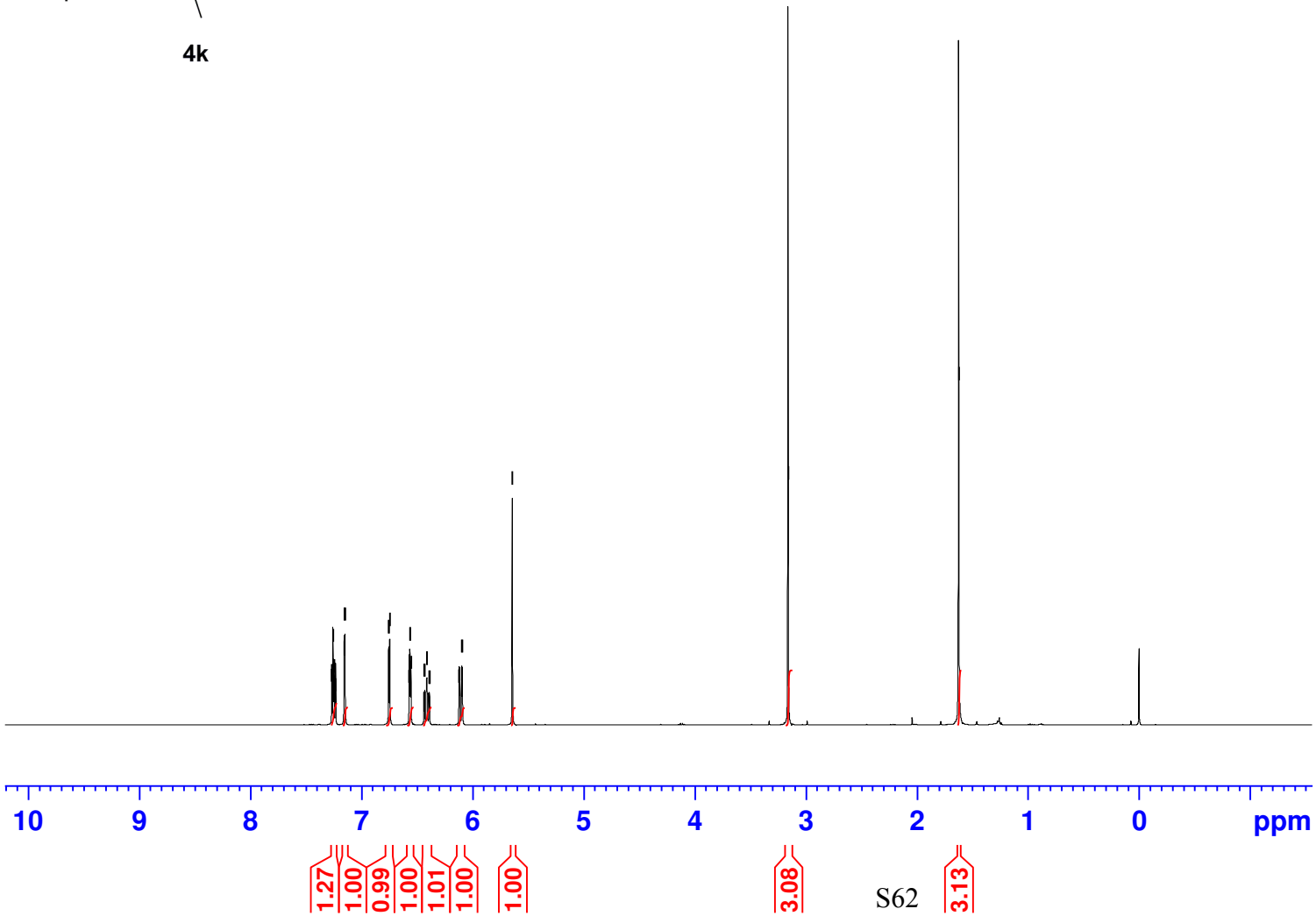
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127589 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





4k

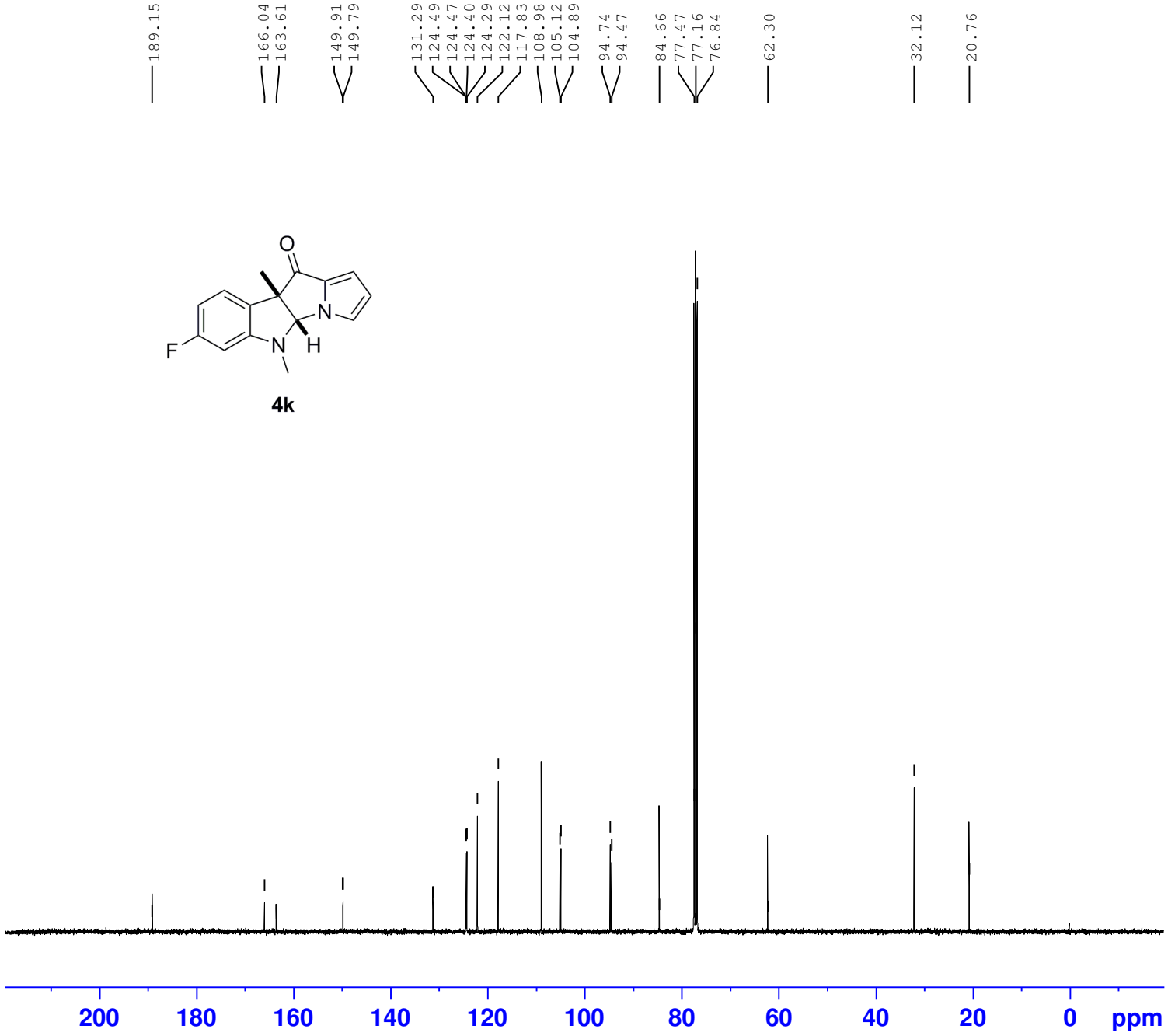



```
Current Data Parameters
NAME                  ZQQ-164
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170116
Time                   14.57
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                     140.62
DW                     62.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                  1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                       9.31 usec
PLW1            16.00000000 W

F2 - Processing parameters
SI                     65536
SF               400.1300102 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



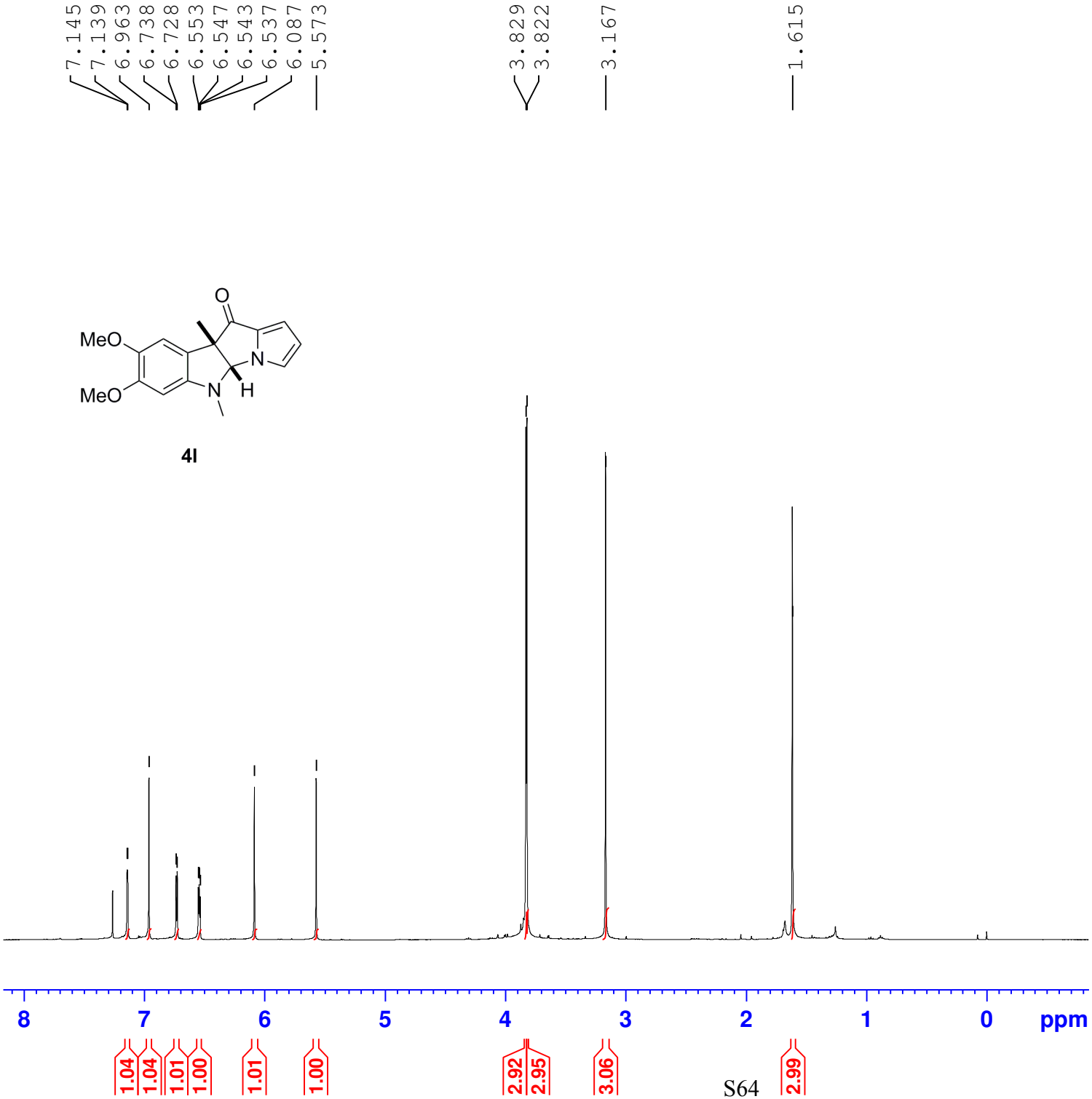
Current Data Parameters
NAME ZQQ-164
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170116
Time 15.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.09 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.17121001 W
PLW13 0.13868000 W

F2 - Processing parameters
SI 32768
SF 100.6127567 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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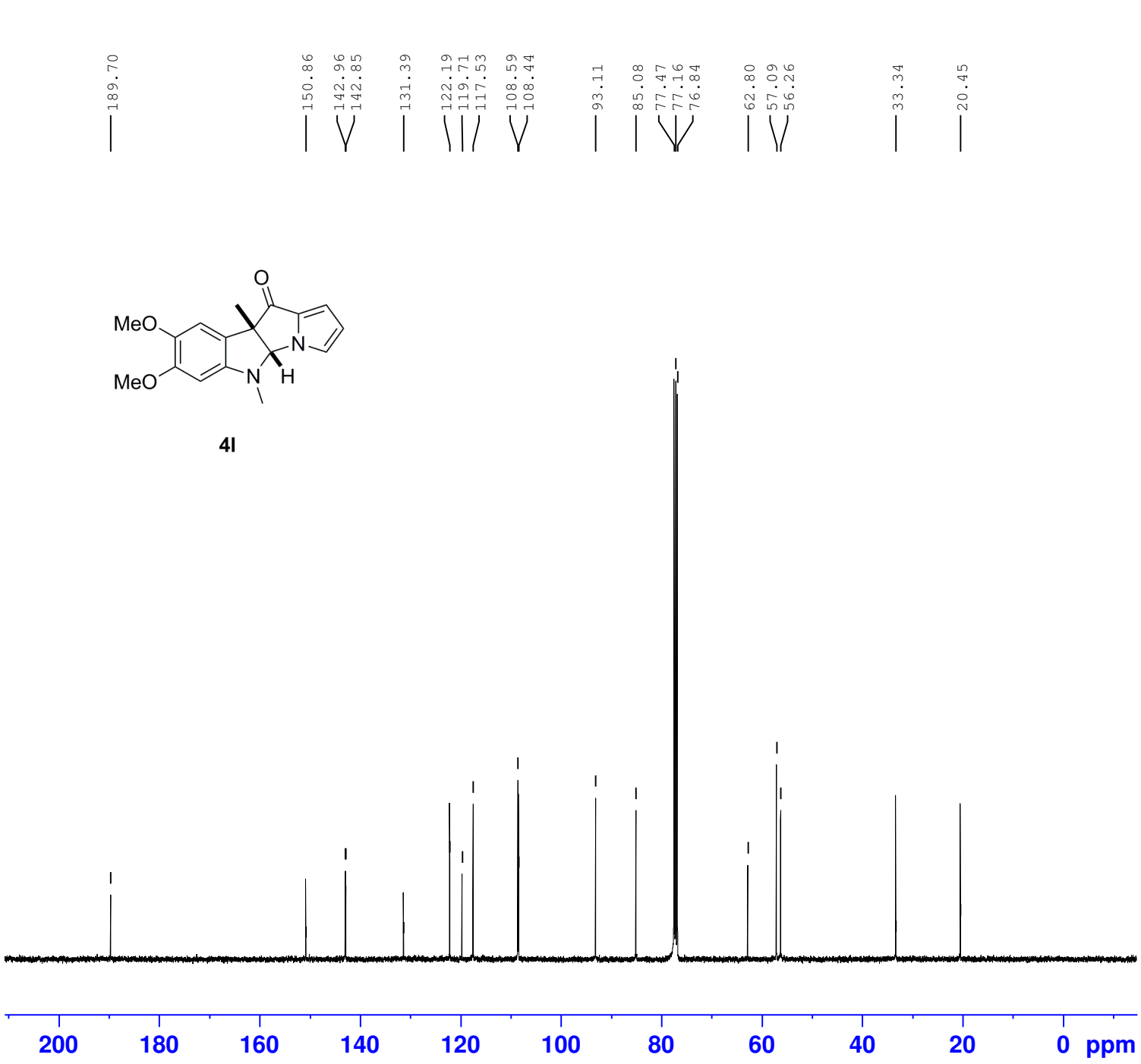
```
Current Data Parameters
NAME                  ZQQ-196C
EXPNO                       10
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170328
Time                     14.43
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                      100.89
DW                      62.400 usec
DE                       6.50 usec
TE                      298.0 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                        1H
P1                       9.09 usec
PLW1             16.00000000 W

F2 - Processing parameters
SI                      65536
SF                400.1300079 MHz
WDW                        EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```



4l

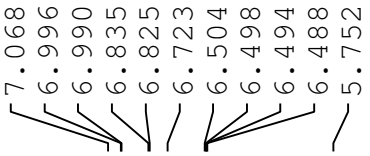
Current Data Parameters
NAME ZQQ-196C
EXPNO 11
PROCNO 1

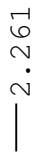
F2 - Acquisition Parameters
Date_ 20170328
Time 15.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

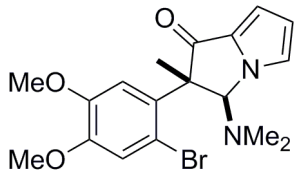
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127590 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40







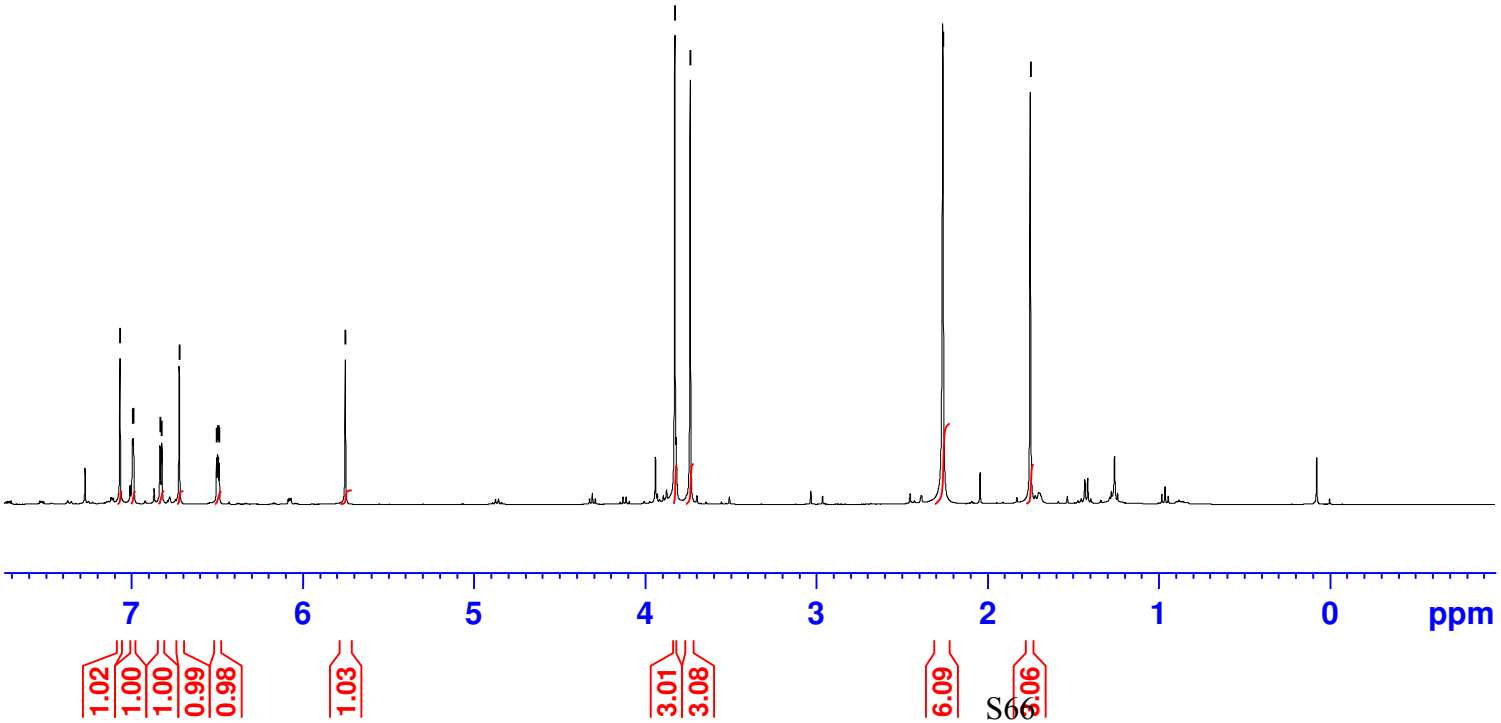
6

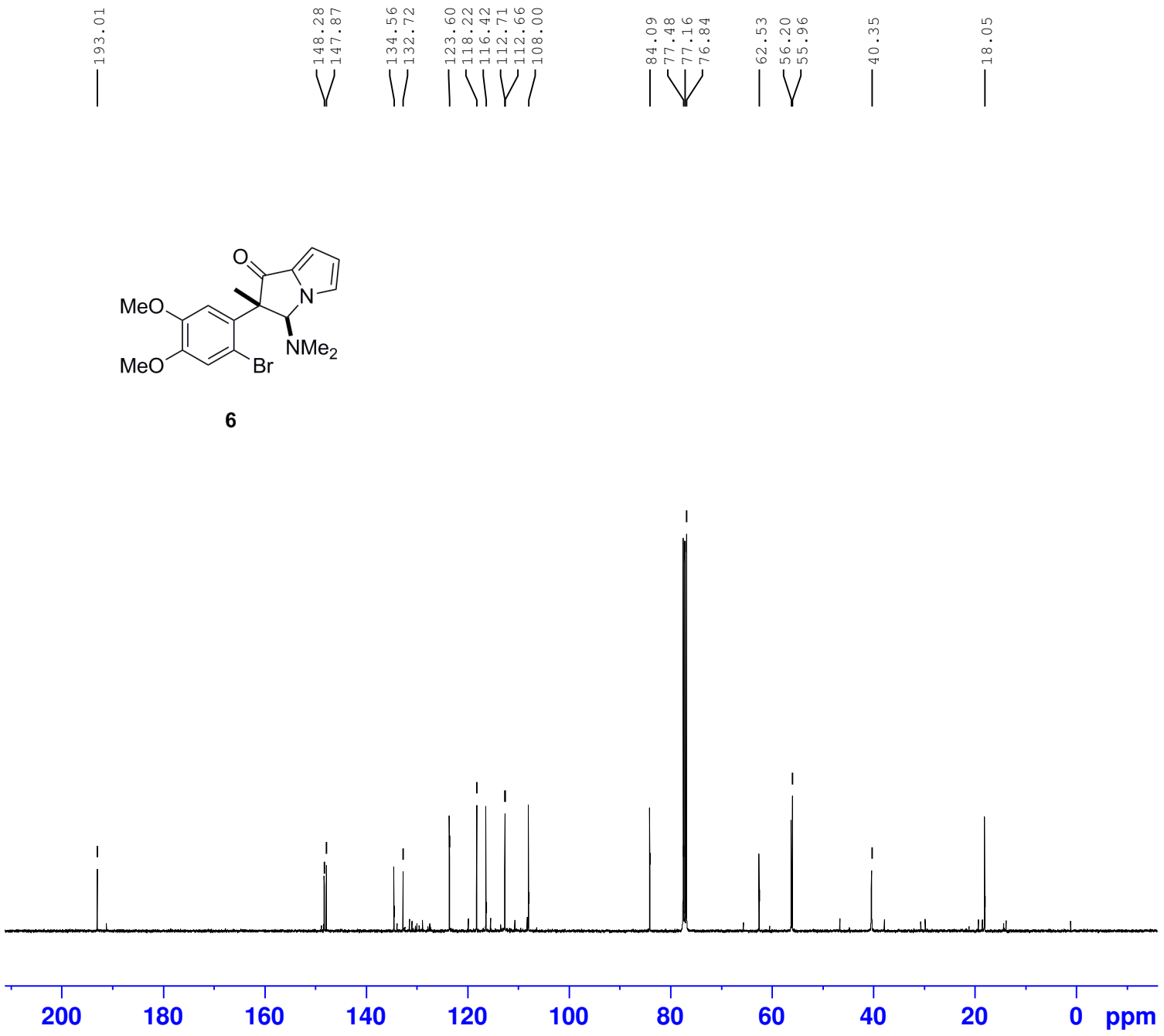
Current Data Parameters
NAME ZQQ-196B
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170328
Time 9.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 61.85
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.09 usec
PLW1 16.00000000 W

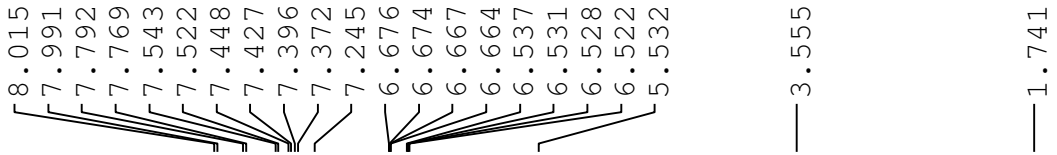
F2 - Processing parameters
SI 65536
SF 400.1300049 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

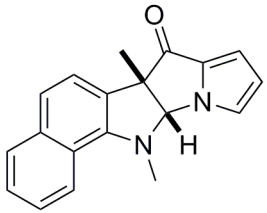




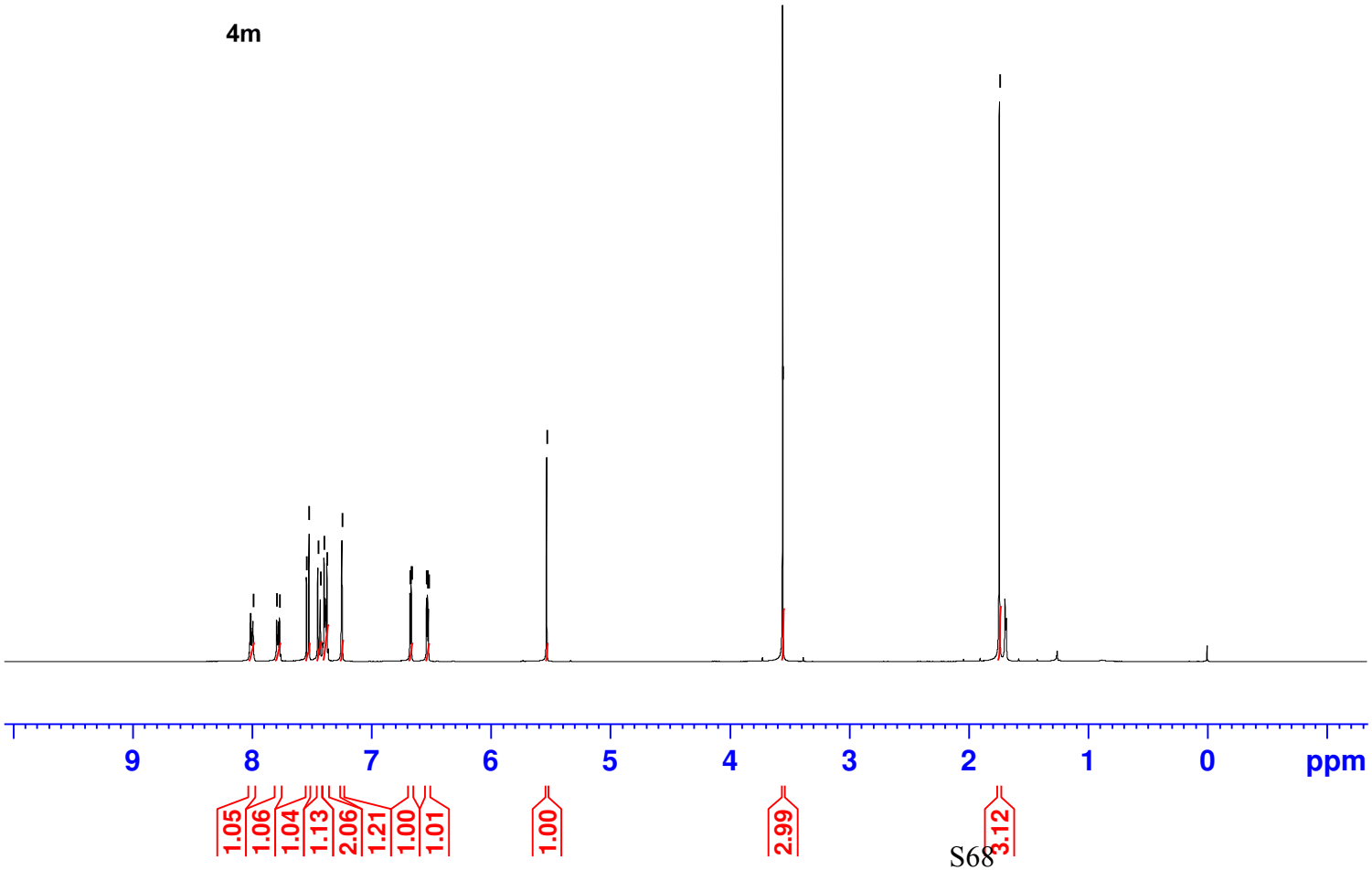
6

```
Current Data Parameters
NAME            ZQQ-196B
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170328
Time               10.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               207.09
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                 9.65 usec
PLW1        73.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        16.00000000 W
PLW12        0.16322000 W
PLW13        0.13220000 W

F2 - Processing parameters
SI                 32768
SF          100.6127629 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

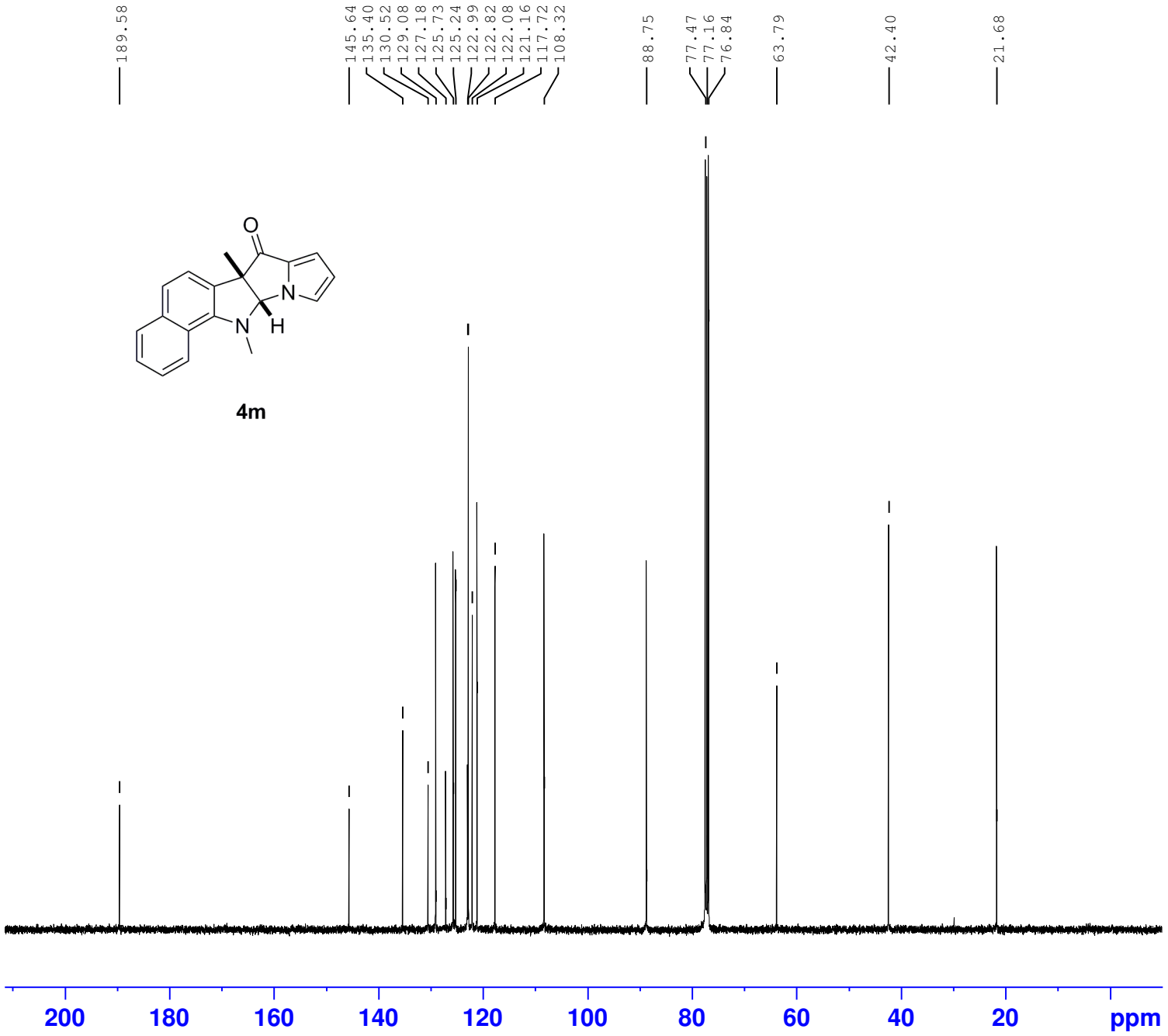




4m




```
Current Data Parameters
NAME                  H-287
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170330
Time                   9.15
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894465 sec
RG                    100.89
DW                    62.400 usec
DE                     6.50 usec
TE                    298.0 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                      1H
P1                     9.09 usec
PLW1           16.00000000 W

F2 - Processing parameters
SI                    65536
SF              400.1300157 MHz
WDW                      EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```



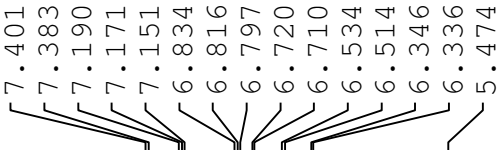
Current Data Parameters
NAME H-287-C
EXPNO 10
PROCNO 1

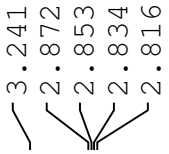
F2 - Acquisition Parameters
Date_ 20170405
Time 7.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

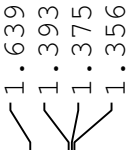
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

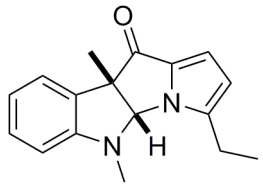
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127607 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

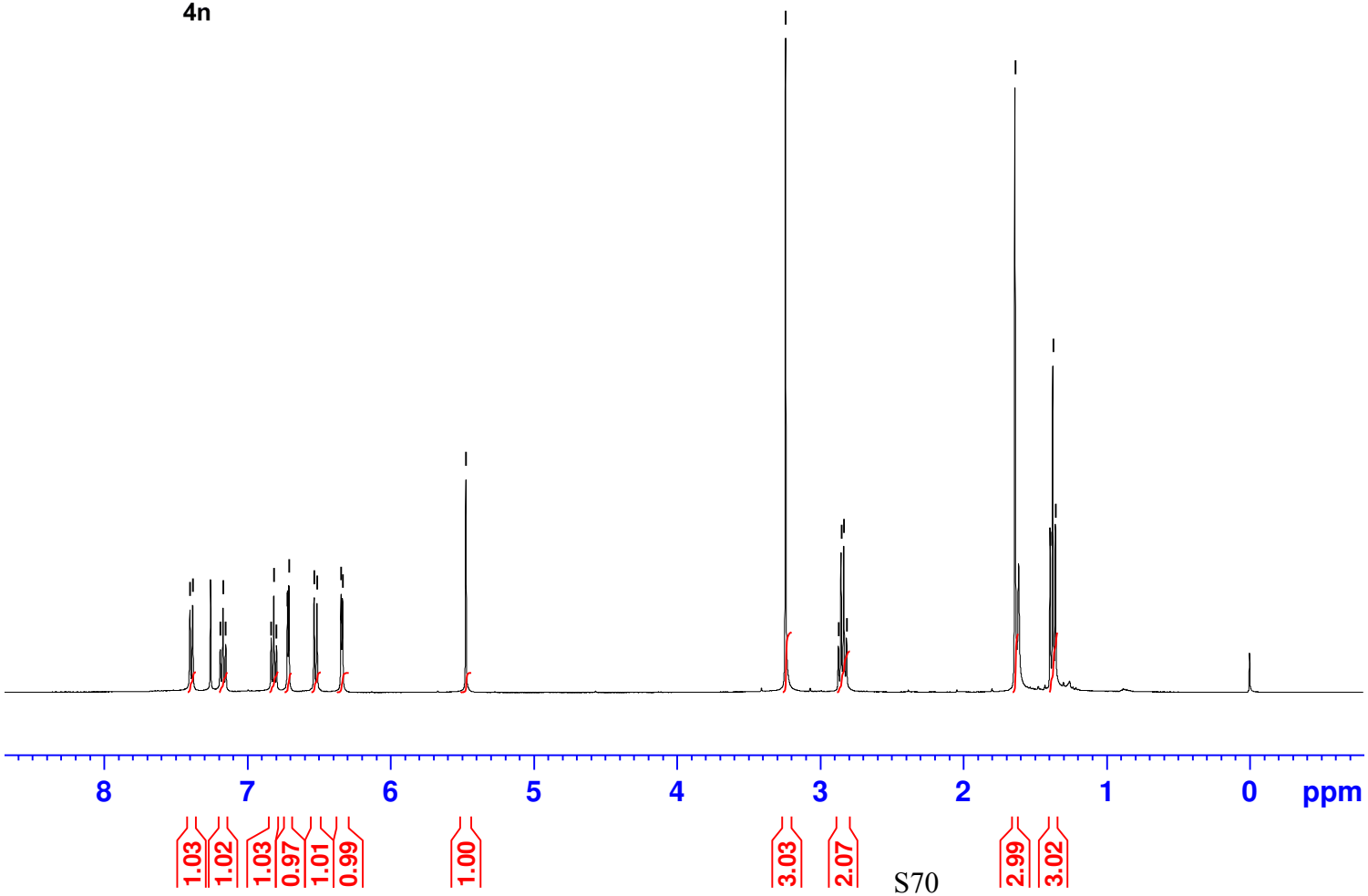




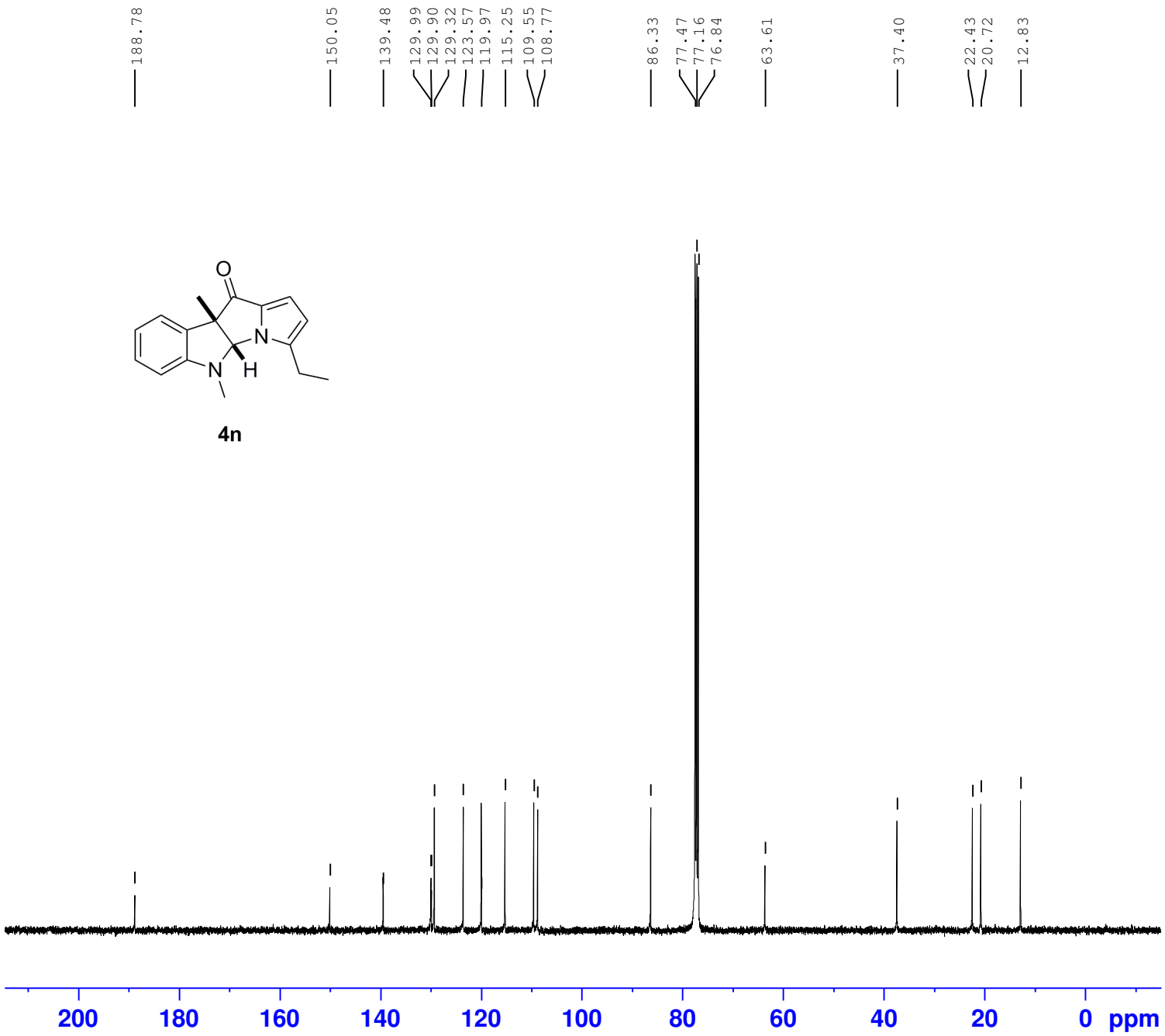




4n




```
Current Data Parameters
NAME            ZQQ-228H
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170522
Time               18.04
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                161.54
DW                62.400 usec
DE                 6.50 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.09 usec
PLW1         16.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300114 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



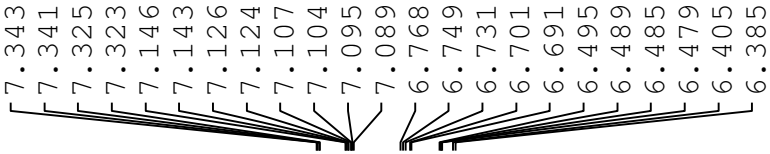
Current Data Parameters
NAME ZQQ-228
EXPNO 11
PROCNO 1

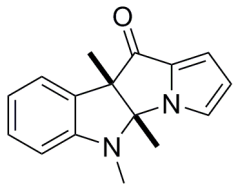
F2 - Acquisition Parameters
Date_ 20170520
Time 2.37
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5120
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

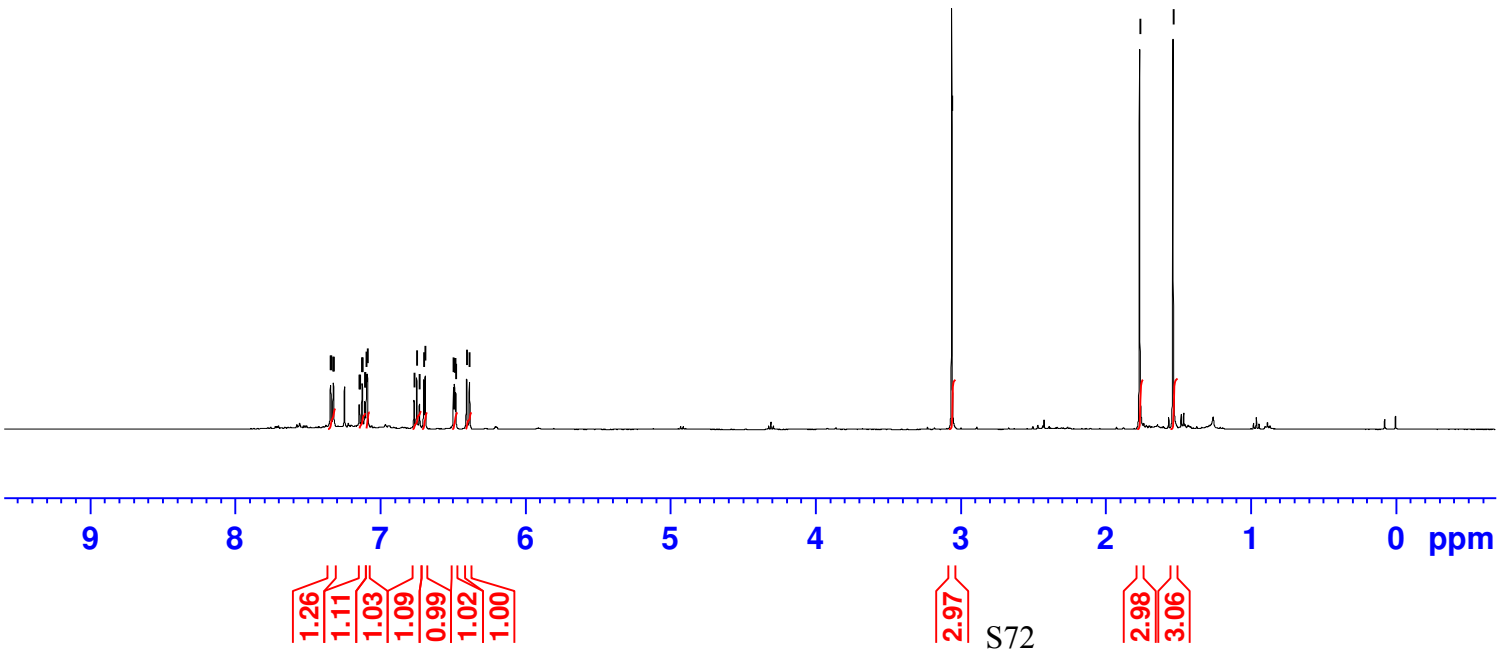
F2 - Processing parameters
SI 32768
SF 100.6127571 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

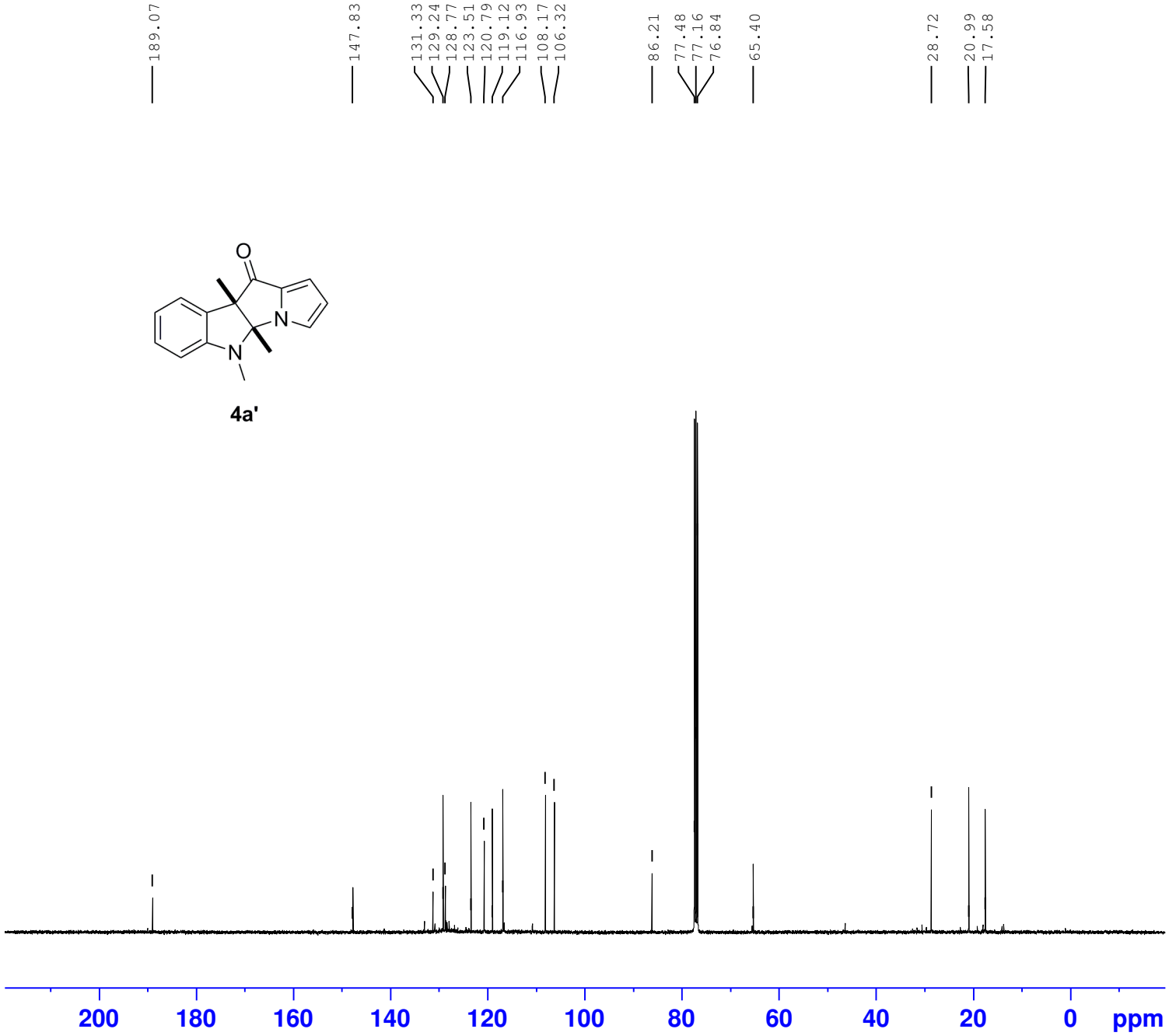




4a'

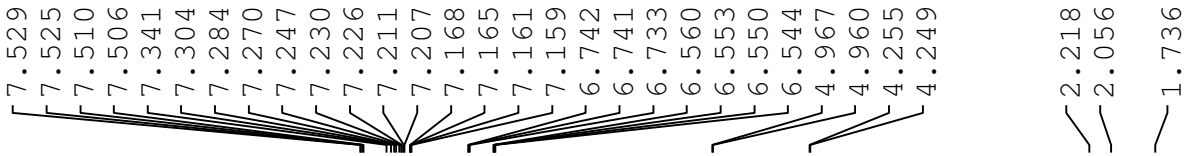
```
Current Data Parameters
NAME            ZQQ-145D
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20170326
Time               23.19
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 91.76
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.09 usec
PLW1        16.00000000 W

F2 - Processing parameters
SI                 65536
SF          400.1300151 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

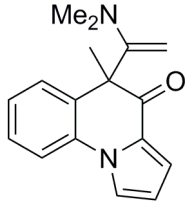




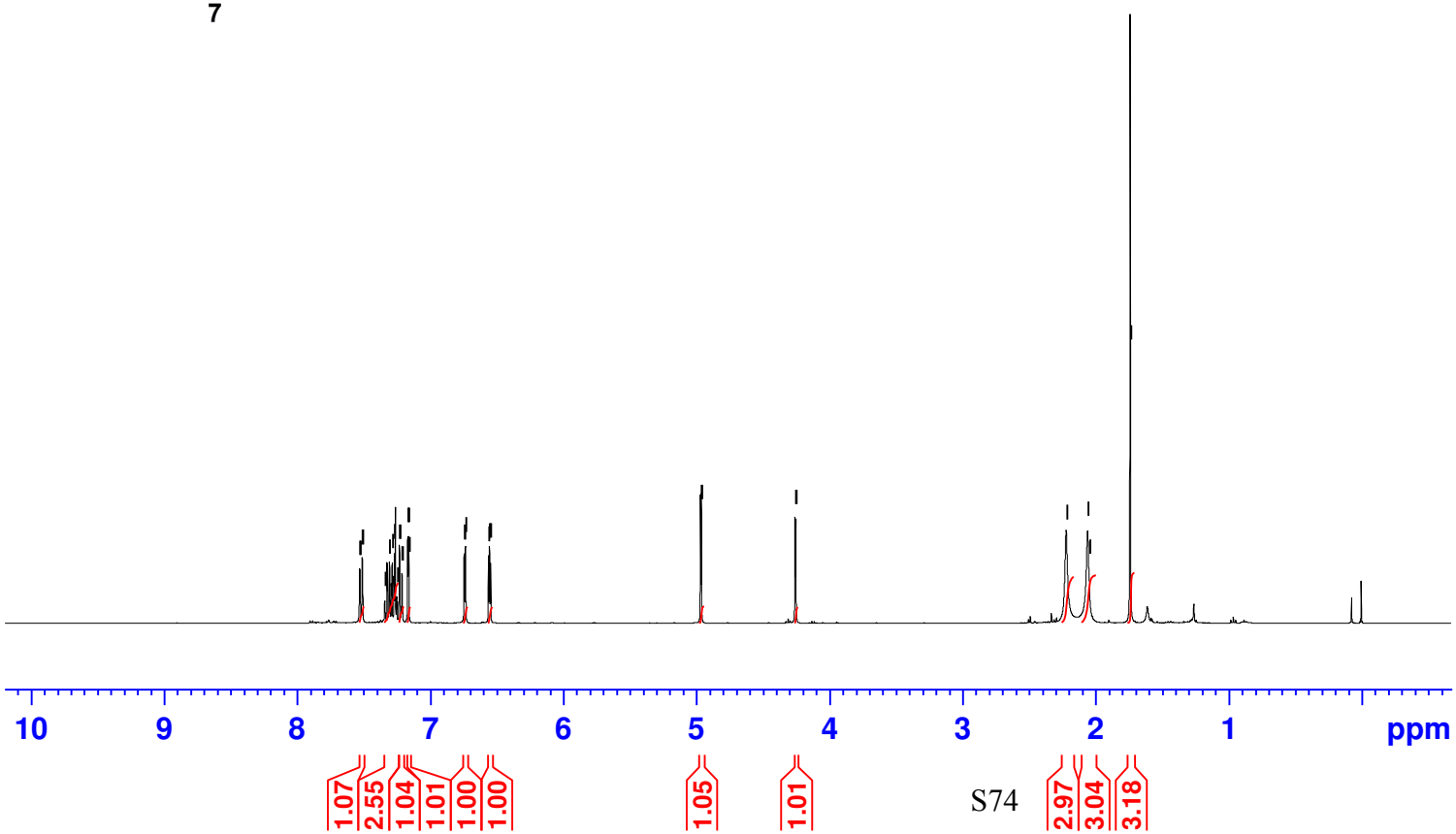
BRUKER

```
Current Data Parameters
NAME                  ZQQ-145D
EXPNO                       11
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20170327
Time                      0.19
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631488 sec
RG                      207.09
DW                     20.800 usec
DE                      6.50 usec
TE                      298.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6228293 MHz
NUC1                       13C
P1                        9.65 usec
PLW1               73.00000000 W

======== CHANNEL f2 ========
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               16.00000000 W
PLW12               0.16322000 W
PLW13               0.13220000 W

F2 - Processing parameters
SI                       32768
SF                 100.6127676 MHz
WDW                         EM
SSB             0
LB                        1.00 Hz
GB              0
PC                        1.40
```

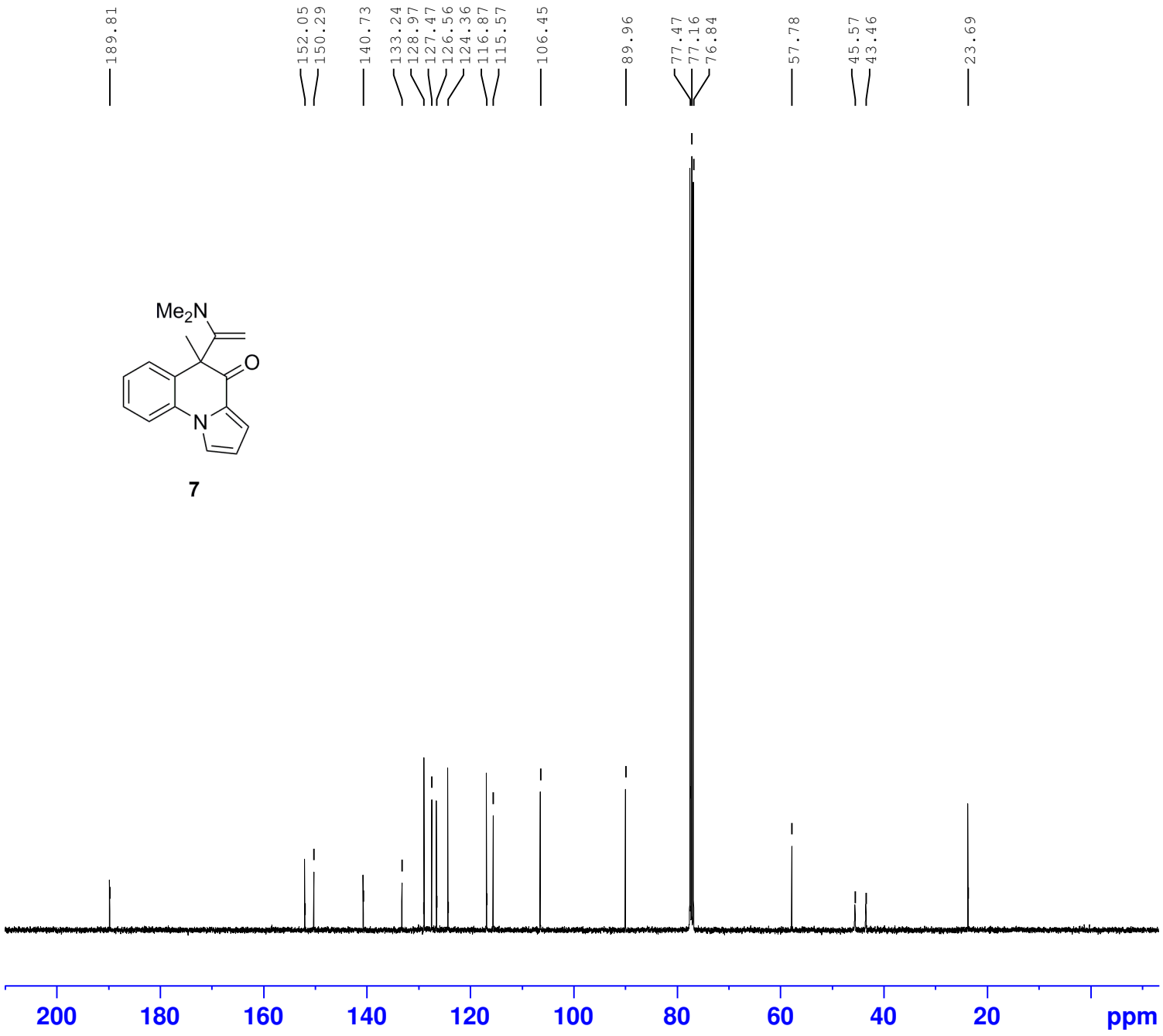




7




```
Current Data Parameters
NAME            ZQQ-145A
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161231
Time               13.23
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                140.62
DW                62.400 usec
DE                 6.50 usec
TE                297.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  9.31 usec
PLW1         16.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300099 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



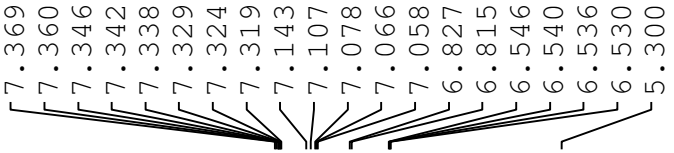
Current Data Parameters
NAME ZQQ-145A
EXPNO 20
PROCNO 1

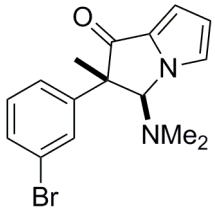
F2 - Acquisition Parameters
Date_ 20161231
Time 13.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.09 usec
PLW1 73.00000000 W

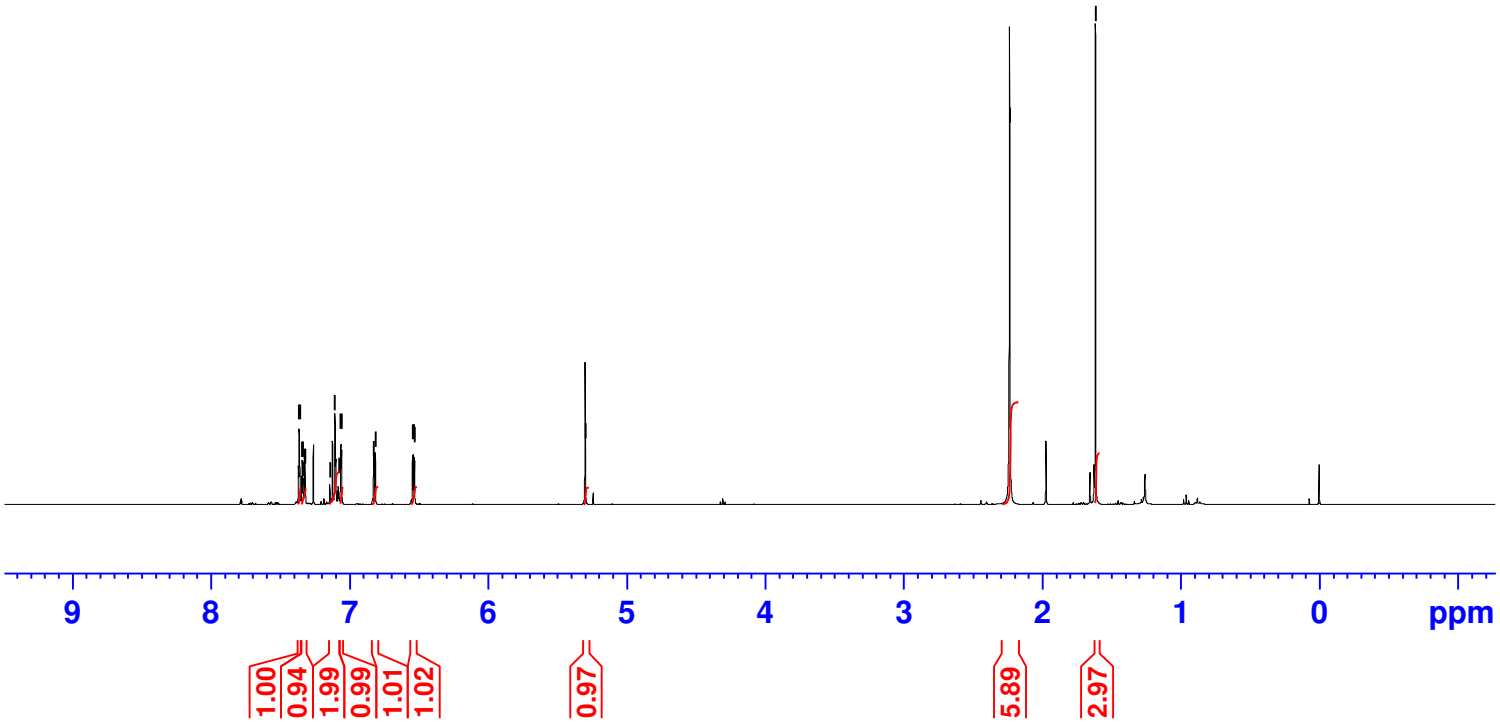
======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.17121001 W
PLW13 0.13868000 W

F2 - Processing parameters
SI 32768
SF 100.6127561 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

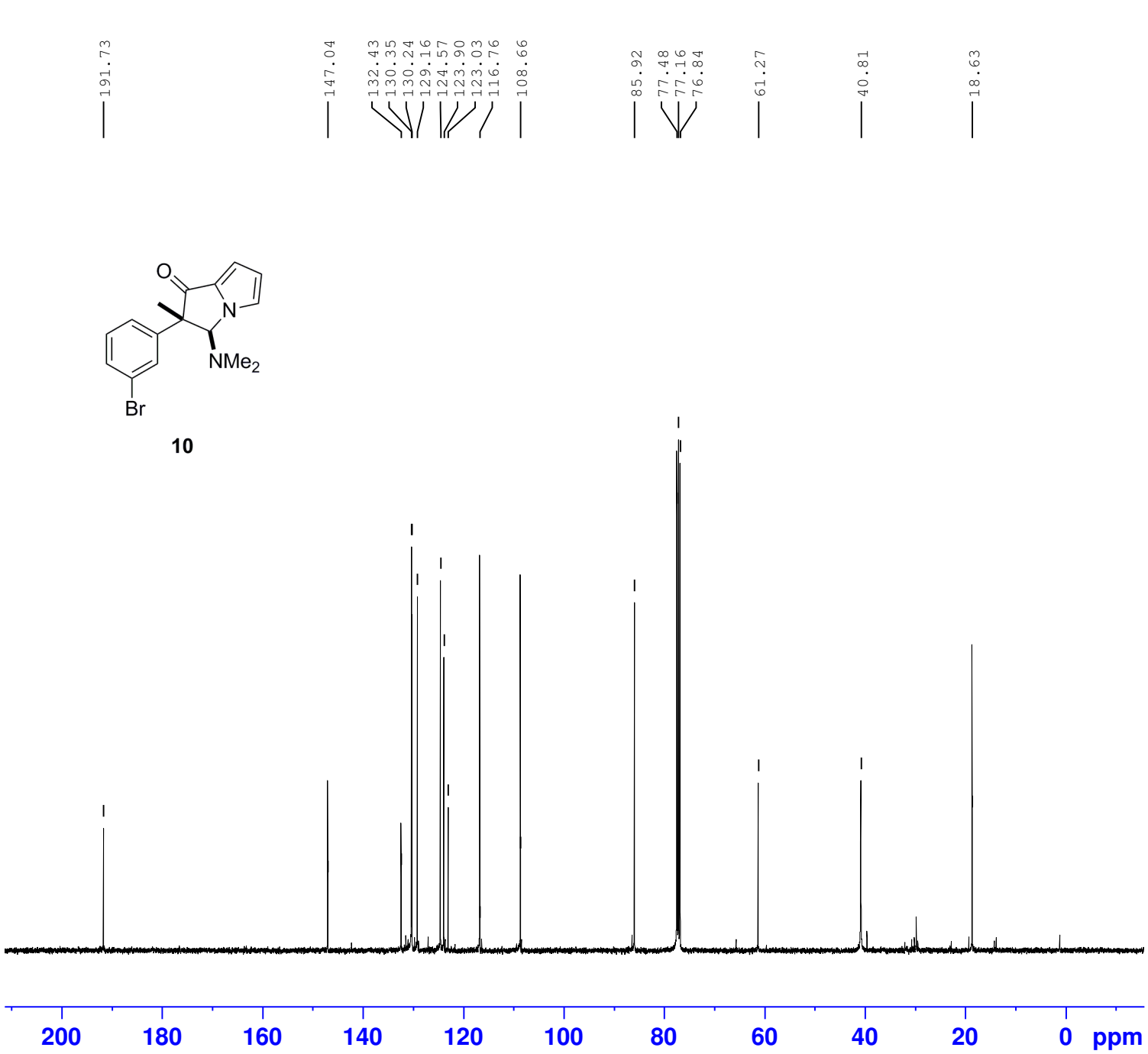




10




```
Current Data Parameters
NAME                  ZQQ-148
EXPNO                      10
PROCNO                      1

F2 - Acquisition Parameters
Date_               20170105
Time                    1.07
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                     100.89
DW                     62.400 usec
DE                      6.50 usec
TE                     298.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                      9.31 usec
PLW1             16.00000000 W

F2 - Processing parameters
SI                     65536
SF               400.1300087 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



Current Data Parameters
NAME ZQQ-148
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170403
Time 21.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 207.09
DW 20.800 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.65 usec
PLW1 73.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.16322000 W
PLW13 0.13220000 W

F2 - Processing parameters
SI 32768
SF 100.6127603 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

5. NOE Spectra of compound 5

Batch NO:201704003

Sample Name:
huanganlvdaqinna
Data Collected on:
Agilent-NMR-vnmrs400
Archive directory:

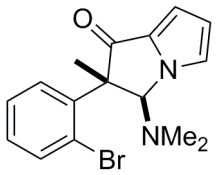
Sample directory:

FidFile: NOESY

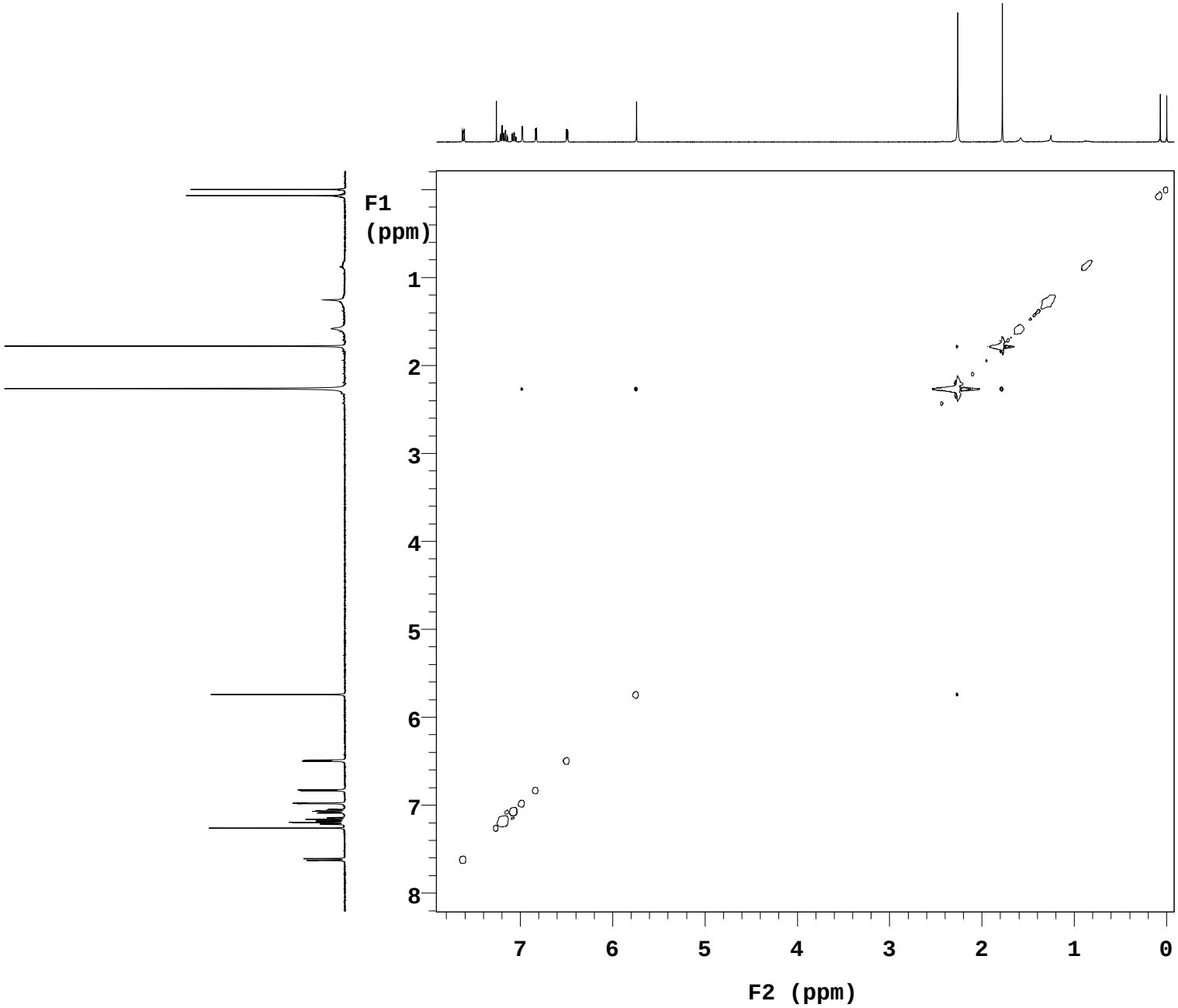
Pulse Sequence: NOESY
Solvent: cdcl3
Data collected on: Apr 14 2017

Operator: lyj

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4006.4 Hz
2D Width 4006.4 Hz
8 repetitions
2 x 200 increments
OBSERVE H1, 399.7902626 MHz
DATA PROCESSING
Gauss apodization 0.069 sec
F1 DATA PROCESSING
Gauss apodization 0.046 sec
FT size 2048 x 2048
Total time 1 hr, 30 min



5



6. X-ray crystal structure of 4a and 4d

5,9*b*-Dimethyl-5,9*b*-dihydropyrrolizino[3,2-*b*]indol-10(4*aH*)-one (4a)

The structure of **4a** was determined by X-ray diffraction. The X-ray data have been deposited at the Cambridge Crystallographic Data Center (CCDC1542588).

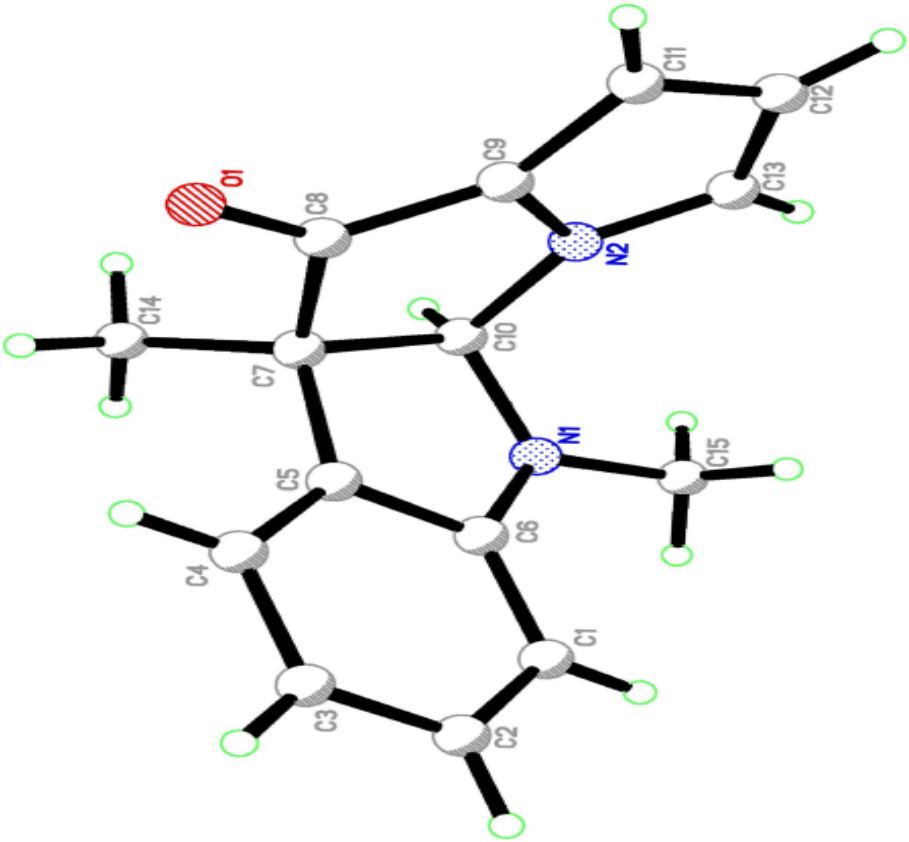


Table 1.

Crystal data	
Chemical formula	$C_{30}H_{28}N_4O_2$
Mr	476.56
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	292(2)
a, b, c (Å)	9.8465 (5), 26.3558 (14), 9.9725 (5)
α (°)	90
β (°)	104.267(2)
γ (°)	90
V ($Å^3$)	2508.2(2)
Z	4
Absorption coefficient (mm^-1)	0.640
F(000)	1008
Crystal size (mm)	0.20 × 0.18 × 0.16
Theta range for data collection	3.35 to 68.42
Limiting indices	-11<=h<=11, -31<=k<=31, -11<=l<=11

Reflections collected / unique	27797 / 4559 [R(int) = 0.0304]
Completeness to theta = 68.42	99.2 %
Max. and min. transmission	0.9045 and 0.8826
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4559 / 0 / 329
Goodness-of-fit on F^2	1.033
Final R indices [I>2sigma(I)]	R1 = 0.0420, wR2 = 0.1109
R indices (all data)	R1 = 0.0474, wR2 = 0.1155
Largest diff. peak and hole (A^-3)	0.128 and -0.236 e.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for a. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(14)	200(2)	427(1)	1970(2)	64(1)
O(1)	3133(1)	73(1)	2954(1)	70(1)
O(2)	2361(2)	7632(1)	8329(1)	85(1)
N(1)	1251(1)	1673(1)	2200(1)	57(1)
N(2)	2603(1)	1254(1)	4285(1)	49(1)
N(3)	3368(2)	9235(1)	7134(2)	71(1)
N(4)	3321(1)	8861(1)	9362(1)	56(1)
C(1)	2066(2)	1809(1)	43(2)	66(1)
C(2)	2518(2)	1563(1)	-995(2)	78(1)
C(3)	2640(2)	1045(1)	-1019(2)	75(1)
C(4)	2306(2)	752(1)	14(2)	60(1)
C(5)	1869(1)	991(1)	1067(1)	46(1)
C(6)	1742(1)	1517(1)	1080(1)	49(1)
C(7)	1481(1)	772(1)	2323(1)	45(1)
C(8)	2774(1)	496(1)	3215(1)	48(1)
C(9)	3420(1)	827(1)	4326(1)	48(1)
C(10)	1316(1)	1260(1)	3149(1)	49(1)
C(11)	4563(2)	873(1)	5456(2)	59(1)
C(12)	4406(2)	1333(1)	6080(2)	68(1)
C(13)	3189(2)	1560(1)	5351(2)	63(1)
C(15)	1179(2)	2197(1)	2598(2)	78(1)
C(16)	1451(2)	9260(1)	4955(2)	83(1)
C(17)	566(2)	8957(1)	3990(2)	95(1)
C(18)	569(2)	8441(1)	4108(2)	87(1)
C(19)	1482(2)	8201(1)	5220(2)	66(1)
C(20)	2381(1)	8497(1)	6190(1)	51(1)
C(21)	2367(2)	9022(1)	6068(2)	59(1)
C(22)	3475(1)	8340(1)	7473(1)	50(1)
C(23)	2746(2)	8070(1)	8467(2)	57(1)

C(24)	2621(2)	8427(1)	9521(2)	58(1)
C(25)	2096(2)	8487(1)	10683(2)	77(1)
C(26)	2504(2)	8964(1)	11215(2)	83(1)
C(27)	3267(2)	9190(1)	10397(2)	73(1)
C(28)	3583(2)	9770(1)	7354(3)	96(1)
C(29)	4659(2)	8023(1)	7163(2)	70(1)
C(30)	3945(1)	8862(1)	8156(2)	57(1)

Table 3. Bond lengths [A] and angles [deg] for a.

C(14)-C(7)	1.5244(18)	C(11)-C(12)	1.390(2)
C(14)-H(14A)	0.9600	C(11)-H(11)	0.9300
C(14)-H(14B)	0.9600	C(12)-C(13)	1.376(2)
C(14)-H(14C)	0.9600	C(12)-H(12)	0.9300
O(1)-C(8)	1.2174(16)	C(13)-H(13)	0.9300
O(2)-C(23)	1.2133(17)	C(15)-H(15A)	0.9600
N(1)-C(6)	1.3841(18)	C(15)-H(15B)	0.9600
N(1)-C(10)	1.4320(17)	C(15)-H(15C)	0.9600
N(1)-C(15)	1.4427(19)	C(16)-C(17)	1.381(3)
N(2)-C(13)	1.3461(18)	C(16)-C(21)	1.395(2)
N(2)-C(9)	1.3785(16)	C(16)-H(16)	0.9300
N(2)-C(10)	1.4769(17)	C(17)-C(18)	1.365(3)
N(3)-C(21)	1.379(2)	C(17)-H(17)	0.9300
N(3)-C(30)	1.429(2)	C(18)-C(19)	1.395(2)
N(3)-C(28)	1.436(2)	C(18)-H(18)	0.9300
N(4)-C(27)	1.3587(19)	C(19)-C(20)	1.379(2)
N(4)-C(24)	1.3652(19)	C(19)-H(19)	0.9300
N(4)-C(30)	1.4797(19)	C(20)-C(21)	1.389(2)
C(1)-C(2)	1.385(3)	C(20)-C(22)	1.5121(19)
C(1)-C(6)	1.389(2)	C(22)-C(29)	1.5281(19)
C(1)-H(1)	0.9300	C(22)-C(23)	1.5346(19)
C(2)-C(3)	1.372(3)	C(22)-C(30)	1.5521(19)
C(2)-H(2)	0.9300	C(23)-C(24)	1.438(2)
C(3)-C(4)	1.390(2)	C(24)-C(25)	1.390(2)
C(3)-H(3)	0.9300	C(25)-C(26)	1.385(3)
C(4)-C(5)	1.381(2)	C(25)-H(25)	0.9300
C(4)-H(4)	0.9300	C(26)-C(27)	1.373(3)
C(5)-C(6)	1.3904(19)	C(26)-H(26)	0.9300
C(5)-C(7)	1.5111(17)	C(27)-H(27)	0.9300
C(7)-C(8)	1.5441(17)	C(28)-H(28A)	0.9600
C(7)-C(10)	1.5571(18)	C(28)-H(28B)	0.9600
C(8)-C(9)	1.4307(19)	C(28)-H(28C)	0.9600
C(9)-C(11)	1.3872(19)	C(29)-H(29A)	0.9600
C(10)-H(10)	0.9800	C(29)-H(29B)	0.9600

C(29)-H(29C)	0.9600	O(1)-C(8)-C(9)	128.83(12)
C(30)-H(30)	0.9800	O(1)-C(8)-C(7)	123.42(12)
C(7)-C(14)-H(14A)	109.5	C(9)-C(8)-C(7)	107.75(10)
C(7)-C(14)-H(14B)	109.5	N(2)-C(9)-C(11)	107.74(12)
H(14A)-C(14)-H(14B)	109.5	N(2)-C(9)-C(8)	109.26(11)
C(7)-C(14)-H(14C)	109.5	C(11)-C(9)-C(8)	142.99(13)
H(14A)-C(14)-H(14C)	109.5	N(1)-C(10)-N(2)	113.96(11)
H(14B)-C(14)-H(14C)	109.5	N(1)-C(10)-C(7)	05.73(11)
C(6)-N(1)-C(10)	109.84(10)	N(2)-C(10)-C(7)	102.43(9)
C(6)-N(1)-C(15)	123.84(13)	N(1)-C(10)-H(10)	111.4
C(10)-N(1)-C(15)	122.79(13)	N(2)-C(10)-H(10)	111.4
C(13)-N(2)-C(9)	109.40(12)	C(7)-C(10)-H(10)	111.4
C(13)-N(2)-C(10)	135.98(11)	C(9)-C(11)-C(12)	106.36(13)
C(9)-N(2)-C(10)	114.59(10)	C(9)-C(11)-H(11)	126.8
C(21)-N(3)-C(30)	110.60(12)	C(12)-C(11)-H(11)	126.8
C(21)-N(3)-C(28)	124.68(16)	C(13)-C(12)-C(11)	108.75(14)
C(30)-N(3)-C(28)	123.35(16)	C(13)-C(12)-H(12)	125.6
C(27)-N(4)-C(24)	109.31(14)	C(11)-C(12)-H(12)	125.6
C(27)-N(4)-C(30)	136.01(14)	N(2)-C(13)-C(12)	107.74(13)
C(24)-N(4)-C(30)	114.68(11)	N(2)-C(13)-H(13)	126.1
C(2)-C(1)-C(6)	118.14(15)	C(12)-C(13)-H(13)	126.1
C(2)-C(1)-H(1)	120.9	N(1)-C(15)-H(15A)	109.5
C(6)-C(1)-H(1)	120.9	N(1)-C(15)-H(15B)	109.5
C(3)-C(2)-C(1)	121.67(15)	H(15A)-C(15)-H(15B)	109.5
C(3)-C(2)-H(2)	119.2	N(1)-C(15)-H(15C)	109.5
C(1)-C(2)-H(2)	119.2	H(15A)-C(15)-H(15C)	109.5
C(2)-C(3)-C(4)	120.17(16)	H(15B)-C(15)-H(15C)	109.5
C(2)-C(3)-H(3)	119.9	C(17)-C(16)-C(21)	117.88(18)
C(4)-C(3)-H(3)	119.9	C(17)-C(16)-H(16)	121.1
C(5)-C(4)-C(3)	118.95(15)	C(21)-C(16)-H(16)	121.1
C(5)-C(4)-H(4)	120.5	C(18)-C(17)-C(16)	121.86(18)
C(3)-C(4)-H(4)	120.5	C(18)-C(17)-H(17)	119.1
C(4)-C(5)-C(6)	120.54(13)	C(16)-C(17)-H(17)	119.1
C(4)-C(5)-C(7)	130.16(12)	C(17)-C(18)-C(19)	120.45(19)
C(6)-C(5)-C(7)	109.30(11)	C(17)-C(18)-H(18)	119.8
N(1)-C(6)-C(1)	128.73(13)	C(19)-C(18)-H(18)	119.8
N(1)-C(6)-C(5)	110.73(11)	C(20)-C(19)-C(18)	118.59(18)
C(1)-C(6)-C(5)	120.52(14)	C(20)-C(19)-H(19)	120.7
C(5)-C(7)-C(14)	113.59(11)	C(18)-C(19)-H(19)	120.7
C(5)-C(7)-C(8)	108.46(10)	C(19)-C(20)-C(21)	120.64(14)
C(14)-C(7)-C(8)	111.69(11)	C(19)-C(20)-C(22)	129.78(14)
C(5)-C(7)-C(10)	101.74(10)	C(21)-C(20)-C(22)	109.58(12)
C(14)-C(7)-C(10)	115.28(11)	N(3)-C(21)-C(20)	110.40(13)
C(8)-C(7)-C(10)	105.27(10)	N(3)-C(21)-C(16)	129.00(16)

C(20)-C(21)-C(16)	120.57(16)	N(4)-C(27)-H(27)	126.1
C(20)-C(22)-C(29)	113.47(12)	C(26)-C(27)-H(27)	126.1
C(20)-C(22)-C(23)	108.90(11)	N(3)-C(28)-H(28A)	109.5
C(29)-C(22)-C(23)	112.19(12)	N(3)-C(28)-H(28B)	109.5
C(20)-C(22)-C(30)	101.75(11)	H(28A)-C(28)-H(28B)	109.5
C(29)-C(22)-C(30)	114.43(12)	N(3)-C(28)-H(28C)	109.5
C(23)-C(22)-C(30)	105.29(11)	H(28A)-C(28)-H(28C)	109.5
O(2)-C(23)-C(24)	128.67(15)	H(28B)-C(28)-H(28C)	109.5
O(2)-C(23)-C(22)	123.47(14)	C(22)-C(29)-H(29A)	109.5
C(24)-C(23)-C(22)	107.86(11)	C(22)-C(29)-H(29B)	109.5
N(4)-C(24)-C(25)	107.67(14)	H(29A)-C(29)-H(29B)	109.5
N(4)-C(24)-C(23)	109.06(13)	C(22)-C(29)-H(29C)	109.5
C(25)-C(24)-C(23)	143.19(16)	H(29A)-C(29)-H(29C)	109.5
C(26)-C(25)-C(24)	106.86(17)	H(29B)-C(29)-H(29C)	109.5
C(26)-C(25)-H(25)	126.6	N(3)-C(30)-N(4)	114.09(12)
C(24)-C(25)-H(25)	126.6	N(3)-C(30)-C(22)	105.86(12)
C(27)-C(26)-C(25)	108.39(16)	N(4)-C(30)-C(22)	102.56(11)
C(27)-C(26)-H(26)	125.8	N(3)-C(30)-H(30)	111.3
C(25)-C(26)-H(26)	125.8	N(4)-C(30)-H(30)	111.3
N(4)-C(27)-C(26)	107.75(16)	C(22)-C(30)-H(30)	111.3

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for a. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(14)	50(1)	62(1)	80(1)	5(1)	12(1)	-10(1)
O(1)	72(1)	44(1)	85(1)	-1(1)	4(1)	13(1)
O(2)	107(1)	51(1)	95(1)	4(1)	23(1)	-16(1)
N(1)	63(1)	43(1)	62(1)	5(1)	9(1)	12(1)
N(2)	50(1)	54(1)	45(1)	-1(1)	14(1)	8(1)
N(3)	83(1)	51(1)	82(1)	7(1)	25(1)	-13(1)
N(4)	50(1)	58(1)	55(1)	-10(1)	3(1)	0(1)
C(1)	66(1)	62(1)	61(1)	20(1)	-4(1)	-9(1)
C(2)	78(1)	103(1)	47(1)	21(1)	7(1)	-16(1)
C(3)	79(1)	100(1)	45(1)	1(1)	15(1)	-6(1)
C(4)	60(1)	66(1)	51(1)	-6(1)	7(1)	0(1)
C(5)	40(1)	49(1)	45(1)	4(1)	3(1)	-1(1)
C(6)	43(1)	50(1)	49(1)	8(1)	-1(1)	0(1)
C(7)	40(1)	43(1)	51(1)	4(1)	10(1)	1(1)
C(8)	47(1)	42(1)	55(1)	9(1)	14(1)	4(1)
C(9)	47(1)	50(1)	47(1)	9(1)	13(1)	7(1)
C(10)	41(1)	52(1)	55(1)	4(1)	15(1)	7(1)

C(11)	55(1)	68(1)	49(1)	10(1)	6(1)	7(1)
C(12)	72(1)	83(1)	44(1)	-5(1)	5(1)	0(1)
C(13)	74(1)	68(1)	49(1)	-11(1)	19(1)	7(1)
C(15)	79(1)	47(1)	108(1)	-2(1)	22(1)	14(1)
C(16)	84(1)	94(1)	79(1)	39(1)	37(1)	18(1)
C(17)	80(1)	146(2)	56(1)	26(1)	15(1)	24(1)
C(18)	66(1)	136(2)	53(1)	-13(1)	5(1)	12(1)
C(19)	53(1)	86(1)	57(1)	-18(1)	12(1)	6(1)
C(20)	43(1)	64(1)	50(1)	-1(1)	17(1)	4(1)
C(21)	58(1)	66(1)	59(1)	11(1)	26(1)	1(1)
C(22)	44(1)	49(1)	56(1)	-6(1)	9(1)	-2(1)
C(23)	56(1)	50(1)	60(1)	6(1)	5(1)	-3(1)
C(24)	57(1)	64(1)	50(1)	5(1)	7(1)	2(1)
C(25)	76(1)	100(1)	55(1)	15(1)	17(1)	11(1
C(26)	83(1)	111(2)	52(1)	-12(1)	9(1)	22(1)
C(27)	67(1)	75(1)	66(1)	-23(1)	-5(1)	10(1)
C(28)	93(1)	54(1)	149(2)	2(1)	46(1)	-19(1)
C(29)	50(1)	73(1)	83(1)	-14(1)	8(1)	9(1)
C(30)	46(1)	56(1)	70(1)	-9(1)	13(1)	-9(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for a.

	x	y	z	U(eq)
H(14A)	-571	606	1383	97
H(14B)	-45	328	2806	97
H(14C)	410	131	1499	97
H(1)	1981	2161	45	79
H(2)	2745	1754	-1694	93
H(3)	2947	889	-1728	90
H(4)	2376	400	-3	72
H(10)	473	1245	3503	58
H(11)	5290	641	5739	70
H(12)	5022	1466	6862	82
H(13)	2836	1870	5557	76
H(15A)	2111	2330	2917	117
H(15B)	701	2219	3327	117
H(15C)	679	2390	1815	117
H(16)	1437	9611	4865	99
H(17)	-50	9109	3239	113
H(18)	-42	8248	3442	104
H(19)	1484	7850	5306	79

H(25)	1572	8252	11037	92
H(26)	2296	9108	11993	100
H(27)	3674	9510	10529	88
H(28A)	3618	9931	6499	143
H(28B)	4451	9827	8029	143
H(28C)	2825	9911	7682	143
H(29A)	4292	7703	6771	105
H(29B)	5363	7964	8005	105
H(29C)	5067	8201	6519	105
H(30)	4969	8887	8446	69

Table 6. Torsion angles [deg] for a.

C(6)-C(1)-C(2)-C(3)	0.4(2)	C(10)-N(2)-C(9)-C(8)	0.10(15)
C(1)-C(2)-C(3)-C(4)	0.0(3)	O(1)-C(8)-C(9)-N(2)	-175.42(14)
C(2)-C(3)-C(4)-C(5)	-0.8(2)	C(7)-C(8)-C(9)-N(2)	5.23(14)
C(3)-C(4)-C(5)-C(6)	1.1(2)	O(1)-C(8)-C(9)-C(11)	2.8(3)
C(3)-C(4)-C(5)-C(7)	-178.23(13)	C(7)-C(8)-C(9)-C(11)	-176.56(17)
C(10)-N(1)-C(6)-C(1)	-168.82(13)	C(6)-N(1)-C(10)-N(2)	94.94(13)
C(15)-N(1)-C(6)-C(1)	-9.6(2)	C(15)-N(1)-C(10)-N(2)	-64.58(17)
C(10)-N(1)-C(6)-C(5)	12.44(15)	C(6)-N(1)-C(10)-C(7)	-16.77(14)
C(15)-N(1)-C(6)-C(5)	171.70(14)	C(15)-N(1)-C(10)-C(7)	-176.28(13)
C(2)-C(1)-C(6)-N(1)	-178.73(14)	C(13)-N(2)-C(10)-N(1)	63.36(19)
C(2)-C(1)-C(6)-C(5)	-0.1(2)	C(9)-N(2)-C(10)-N(1)	-118.88(12)
C(4)-C(5)-C(6)-N(1)	178.21(12)	C(13)-N(2)-C(10)-C(7)	177.05(15)
C(7)-C(5)-C(6)-N(1)	-2.35(14)	C(9)-N(2)-C(10)-C(7)	-5.20(13)
C(4)-C(5)-C(6)-C(1)	-0.64(19)	C(5)-C(7)-C(10)-N(1)	14.29(12)
C(7)-C(5)-C(6)-C(1)	178.79(12)	C(14)-C(7)-C(10)-N(1)	-109.09(13)
C(4)-C(5)-C(7)-C(14)	-63.62(18)	C(8)-C(7)-C(10)-N(1)	127.36(11)
C(6)-C(5)-C(7)-C(14)	117.01(12)	C(5)-C(7)-C(10)-N(2)	-105.31(10)
C(4)-C(5)-C(7)-C(8)	61.19(17)	C(14)-C(7)-C(10)-N(2)	131.31(12)
C(6)-C(5)-C(7)-C(8)	-118.17(11)	C(8)-C(7)-C(10)-N(2)	7.76(12)
C(4)-C(5)-C(7)-C(10)	171.86(13)	N(2)-C(9)-C(11)-C(12)	0.00(16)
C(6)-C(5)-C(7)-C(10)	-7.50(13)	C(8)-C(9)-C(11)-C(12)	-178.23(17)
C(5)-C(7)-C(8)-O(1)	-79.32(15)	C(9)-C(11)-C(12)-C(13)	0.42(18)
C(14)-C(7)-C(8)-O(1)	46.61(18)	C(9)-N(2)-C(13)-C(12)	0.69(17)
C(10)-C(7)-C(8)-O(1)	172.41(13)	C(10)-N(2)-C(13)-C(12)	178.53(14)
C(5)-C(7)-C(8)-C(9)	100.08(12)	C(11)-C(12)-C(13)-N(2)	-0.69(19)
C(14)-C(7)-C(8)-C(9)	-133.99(12)	C(21)-C(16)-C(17)-C(18)	0.2(3)
C(10)-C(7)-C(8)-C(9)	-8.19(13)	C(16)-C(17)-C(18)-C(19)	-0.1(3)
C(13)-N(2)-C(9)-C(11)	-0.43(15)	C(17)-C(18)-C(19)-C(20)	-0.3(2)
C(10)-N(2)-C(9)-C(11)	-178.78(11)	C(18)-C(19)-C(20)-C(21)	0.6(2)
C(13)-N(2)-C(9)-C(8)	178.44(11)	C(18)-C(19)-C(20)-C(22)	-179.31(14)

C(30)-N(3)-C(21)-C(20)	-9.74(17)	C(30)-N(4)-C(24)-C(23)	2.63(16)
C(28)-N(3)-C(21)-C(20)	-176.74(15)	O(2)-C(23)-C(24)-N(4)	173.89(16)
C(30)-N(3)-C(21)-C(16)	172.22(15)	C(22)-C(23)-C(24)-N(4)	-6.44(16)
C(28)-N(3)-C(21)-C(16)	5.2(3)	O(2)-C(23)-C(24)-C(25)	-2.4(3)
C(19)-C(20)-C(21)-N(3)	-178.80(13)	C(22)-C(23)-C(24)-C(25)	177.3(2)
C(22)-C(20)-C(21)-N(3)	1.16(15)	N(4)-C(24)-C(25)-C(26)	-0.29(18)
C(19)-C(20)-C(21)-C(16)	-0.6(2)	C(23)-C(24)-C(25)-C(26)	176.0(2)
C(22)-C(20)-C(21)-C(16)	179.39(13)	C(24)-C(25)-C(26)-C(27)	-0.3(2)
C(17)-C(16)-C(21)-N(3)	178.00(16)	C(24)-N(4)-C(27)-C(26)	-0.91(18)
C(17)-C(16)-C(21)-C(20)	0.1(2)	C(30)-N(4)-C(27)-C(26)	179.68(15)
C(19)-C(20)-C(22)-C(29)	63.39(19)	C(25)-C(26)-C(27)-N(4)	0.7(2)
C(21)-C(20)-C(22)-C(29)	-116.56(13	C(21)-N(3)-C(30)-N(4)	-98.19(14)
C(19)-C(20)-C(22)-C(23)	-62.34(17)	C(28)-N(3)-C(30)-N(4)	69.02(19)
C(21)-C(20)-C(22)-C(23)	117.71(12)	C(21)-N(3)-C(30)-C(22)	13.81(16)
C(19)-C(20)-C(22)-C(30)	-173.19(14)	C(28)-N(3)-C(30)-C(22)	-178.99(15)
C(21)-C(20)-C(22)-C(30)	6.86(14)	C(27)-N(4)-C(30)-N(3)	-64.4(2)
C(20)-C(22)-C(23)-O(2)	78.83(17)	C(24)-N(4)-C(30)-N(3)	116.21(14)
C(29)-C(22)-C(23)-O(2)	-47.63(19)	C(27)-N(4)-C(30)-C(22)	-178.37(15)
C(30)-C(22)-C(23)-O(2)	-172.70(15)	C(24)-N(4)-C(30)-C(22)	2.24(15)
C(20)-C(22)-C(23)-C(24)	-100.87(13)	C(20)-C(22)-C(30)-N(3)	-12.09(14)
C(29)-C(22)-C(23)-C(24)	132.67(13)	C(29)-C(22)-C(30)-N(3)	110.68(14)
C(30)-C(22)-C(23)-C(24)	7.60(15)	C(23)-C(22)-C(30)-N(3)	-125.66(12)
C(27)-N(4)-C(24)-C(25)	0.74(17)	C(20)-C(22)-C(30)-N(4)	107.77(11)
C(30)-N(4)-C(24)-C(25)	-179.70(12)	C(29)-C(22)-C(30)-N(4)	-129.46(13)
C(27)-N(4)-C(24)-C(23)	-176.93(12)	C(23)-C(22)-C(30)-N(4)	5.80(13)

9*b*-(2-Bromobenzyl)-5-methyl-5,9*b*-dihydropyrrolizino[3,2-*b*]indol-10(4*aH*)-one (4d)

The structure of **4d** was determined by X-ray diffraction. The X-ray data have been deposited at the Cambridge Crystallographic Data Center (CCDC1543830).

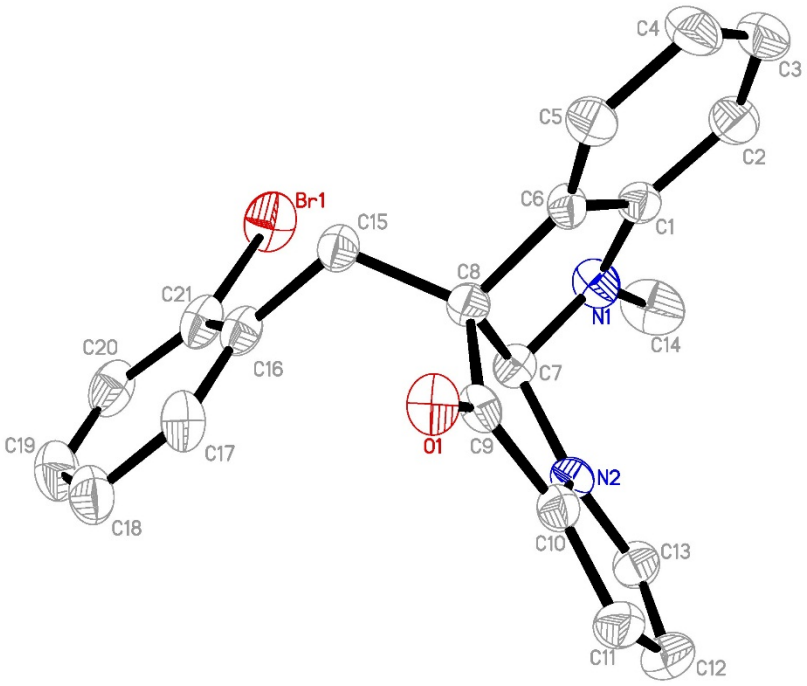


Table 1.

Crystal data	
Chemical formula	$C_{21}H_{17}BrN_2O$
Mr	393.28
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293(2)
a, b, c (Å)	8.7889 (18), 8.9182 (18), 22.444 (5)
α (°)	90
β (°)	91.02(3)
γ (°)	90
V ($Å^3$)	1758.9(6)
Z	8
Absorption coefficient(mm^-1)	2.348
F(000)	800
Crystal size (mm)	0.18 × 0.15 × 0.11
Theta range for data collection	2.46 to 28.02
Limiting indices	-11<=h<=11, -11<=k<=11, -27<=l<=29
Reflections collected / unique	20845 / 4152 [R(int) = 0.0557]
Completeness to theta = 68.42	97.6 %

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4152 / 0 / 227
Goodness-of-fit on F^2	1.095
Final R indices [I>2sigma(I)]	R1 = 0.0742, wR2 = 0.2136
R indices (all data)	R1 = 0.1190, wR2 = 0.2675
Largest diff. peak and hole (A^-3)	0.591 and -0.711 e.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for a. U(eq) is defined as one third of the trace of the orthogonalizedUij tensor.

	x	y	z	U(eq)
Br(1)	4163(1)	11229(1)	1002(1)	72(1)
O(1)	6074(4)	4762(4)	764(1)	48(1)
N(1)	2794(5)	7463(5)	1932(2)	48(1)
N(2)	5261(4)	6305(4)	2138(2)	40(1)
C(1)	1968(5)	6476(5)	1578(2)	40(1)
C(2)	483(6)	5970(6)	1626(3)	55(1)
C(3)	-74(6)	5000(7)	1197(3)	61(1)
C(4)	780(6)	4546(7)	722(3)	59(1)
C(5)	2260(6)	5036(6)	676(2)	50(1)
C(6)	2855(5)	5995(5)	1098(2)	38(1)
C(7)	4382(5)	7408(5)	1784(2)	41(1)
C(8)	4401(5)	6701(5)	1145(2)	39(1)
C(9)	5582(5)	5440(5)	1184(2)	38(1)
C(10)	5927(5)	5200(5)	1813(2)	42(1)
C(11)	6727(6)	4259(7)	2192(2)	54(1)
C(12)	6514(6)	4829(7)	2769(2)	59(1)
C(13)	5613(6)	6085(6)	2723(2)	50(1)
C(14)	2285(7)	8006(9)	2494(3)	76(2)
C(15)	4684(5)	7785(5)	630(2)	42(1)
C(16)	6067(6)	8753(5)	681(2)	43(1)
C(17)	7497(6)	8162(6)	573(2)	52(1)
C(18)	8804(7)	9023(7)	595(3)	60(1)
C(19)	8702(7)	10554(7)	694(3)	64(1)
C(20)	7335(8)	11191(6)	807(2)	60(2)
C(21)	6024(6)	10284(6)	810(2)	52(1)

Table 3. Bond lengths [A] and angles [deg] for a.

Br(1)-C(21)	1.897(6)	N(1)-C(1)	1.384(6)
O(1)-C(9)	1.206(5)	N(1)-C(14)	1.430(7)

N(1)-C(7)	1.442(6)	C(4)-C(3)-C(2)	122.3(5)
N(2)-C(13)	1.357(6)	C(3)-C(4)-C(5)	119.6(5)
N(2)-C(10)	1.365(6)	C(6)-C(5)-C(4)	119.7(5)
N(2)-C(7)	1.474(6)	C(5)-C(6)-C(1)	120.6(4)
C(1)-C(2)	1.387(7)	C(5)-C(6)-C(8)	130.1(4)
C(1)-C(6)	1.408(6)	C(1)-C(6)-C(8)	109.3(4)
C(2)-C(3)	1.378(8)	N(1)-C(7)-N(2)	113.5(4)
C(3)-C(4)	1.375(8)	N(1)-C(7)-C(8)	104.6(4)
C(4)-C(5)	1.378(7)	N(2)-C(7)-C(8)	102.2(3)
C(5)-C(6)	1.372(6)	C(6)-C(8)-C(15)	111.8(4)
C(6)-C(8)	1.500(6)	C(6)-C(8)-C(9)	107.9(4)
C(7)-C(8)	1.566(6)	C(15)-C(8)-C(9)	112.8(4)
C(8)-C(15)	1.531(6)	C(6)-C(8)-C(7)	102.1(3)
C(8)-C(9)	1.532(6)	C(15)-C(8)-C(7)	116.2(4)
C(9)-C(10)	1.454(6)	C(9)-C(8)-C(7)	105.2(3)
C(10)-C(11)	1.379(7)	O(1)-C(9)-C(10)	127.8(4)
C(11)-C(12)	1.407(8)	O(1)-C(9)-C(8)	125.1(4)
C(12)-C(13)	1.374(8)	C(10)-C(9)-C(8)	107.0(4)
C(15)-C(16)	1.493(6)	N(2)-C(10)-C(11)	109.2(4)
C(16)-C(17)	1.388(7)	N(2)-C(10)-C(9)	109.2(4)
C(16)-C(21)	1.396(6)	C(11)-C(10)-C(9)	141.6(5)
C(17)-C(18)	1.382(8)	C(10)-C(11)-C(12)	105.9(5)
C(18)-C(19)	1.386(8)	C(13)-C(12)-C(11)	108.1(4)
C(19)-C(20)	1.356(9)	N(2)-C(13)-C(12)	108.2(4)
C(20)-C(21)	1.408(8)	C(16)-C(15)-C(8)	116.7(4)
C(1)-N(1)-C(14)	123.6(5)	C(17)-C(16)-C(21)	115.8(5)
C(1)-N(1)-C(7)	110.2(3)	C(17)-C(16)-C(15)	120.3(4)
C(14)-N(1)-C(7)	122.3(4)	C(21)-C(16)-C(15)	123.8(5)
C(13)-N(2)-C(10)	108.7(4)	C(18)-C(17)-C(16)	122.5(5)
C(13)-N(2)-C(7)	136.6(4)	C(17)-C(18)-C(19)	119.8(6)
C(10)-N(2)-C(7)	114.7(4)	C(20)-C(19)-C(18)	120.2(5)
N(1)-C(1)-C(2)	130.4(5)	C(19)-C(20)-C(21)	119.2(5)
N(1)-C(1)-C(6)	110.0(4)	C(16)-C(21)-C(20)	122.4(5)
C(2)-C(1)-C(6)	119.6(4)	C(16)-C(21)-Br(1)	120.5(4)
C(3)-C(2)-C(1)	118.3(5)	C(20)-C(21)-Br(1)	117.1(4)

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for a. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12

Br(1)	85(1)	53(1)	78(1)	2(1)	16(1)	14(1)
O(1)	58(2)	40(2)	46(2)	-7(1)	10(2)	-1(1)
N(1)	46(2)	53(2)	45(2)	-11(2)	11(2)	4(2)
N(2)	36(2)	42(2)	42(2)	-1(2)	-3(2)	-5(2)
C(1)	36(2)	43(2)	42(2)	4(2)	1(2)	5(2)
C(2)	39(3)	70(3)	57(3)	7(2)	6(2)	6(2)
C(3)	38(2)	66(3)	78(4)	13(3)	-8(3)	-2(2)
C(4)	48(3)	59(3)	71(3)	-1(3)	-16(3)	-15(2)
C(5)	50(3)	51(3)	48(3)	-1(2)	-6(2)	-10(2)
C(6)	36(2)	41(2)	38(2)	4(2)	5(2)	-2(2)
C(7)	45(2)	38(2)	40(2)	-1(2)	0(2)	0(2)
C(8)	40(2)	39(2)	39(2)	1(2)	-1(2)	-2(2)
C(9)	35(2)	37(2)	43(2)	-4(2)	5(2)	-8(2)
C(10)	38(2)	44(2)	43(2)	5(2)	6(2)	0(2)
C(11)	43(3)	64(3)	55(3)	14(2)	4(2)	11(2)
C(12)	61(3)	67(3)	46(3)	10(2)	-12(2)	0(3)
C(13)	55(3)	63(3)	32(2)	-1(2)	-3(2)	-8(2)
C(14)	64(4)	97(5)	68(4)	-31(3)	13(3)	16(3)
C(15)	48(2)	39(2)	38(2)	3(2)	2(2)	-5(2)
C(16)	59(3)	35(2)	35(2)	2(2)	1(2)	-6(2)
C(17)	62(3)	43(3)	50(3)	-4(2)	16(2)	-7(2)
C(18)	59(3)	71(4)	52(3)	0(2)	13(3)	-11(3)
C(19)	69(4)	55(3)	68(4)	2(3)	12(3)	-21(3)
C(20)	90(4)	40(3)	49(3)	3(2)	-3(3)	-21(3)
C(21)	76(3)	40(2)	42(2)	3(2)	5(2)	-5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for a.

	x	y	z	U(eq)
H(2)	-121	6277	1939	67
H(3)	-1062	4639	1229	73
H(4)	361	3913	433	71
H(5)	2853	4718	361	60
H(7)	4854	8403	1794	49
H(11)	7291	3420	2087	65
H(12)	6913	4426	3120	70
H(13)	5300	6680	3037	60
H(14A)	1250	8340	2454	114
H(14B)	2917	8827	2622	114
H(14C)	2346	7214	2784	114

H(15A)	4755	7202	266	50
H(15B)	3803	8432	586	50
H(17)	7579	7148	483	6
H(18)	9749	8578	543	72
H(19)	9572	11145	683	76
H(20)	7265	12214	881	72