

Supporting Information for

**Tandem Prins/Pinacol reaction for the synthesis of
oxaspiro[4.5]decan-1-one scaffolds**

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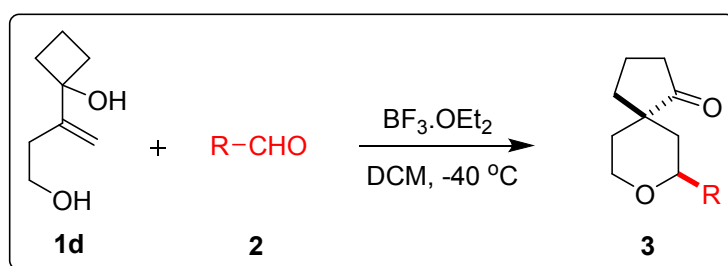
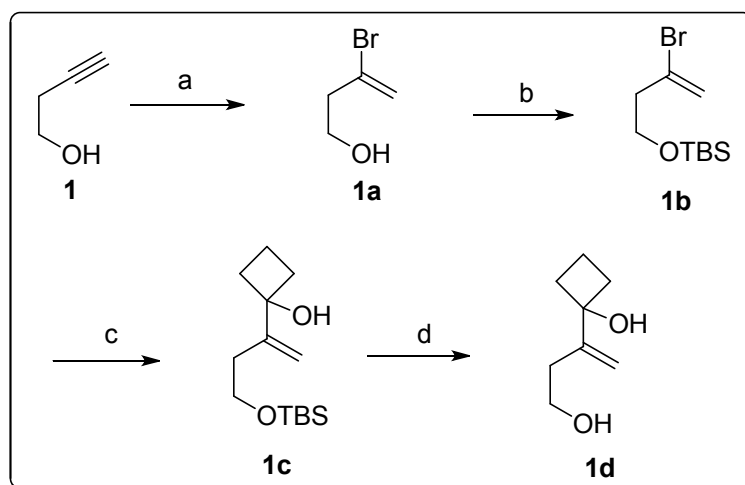


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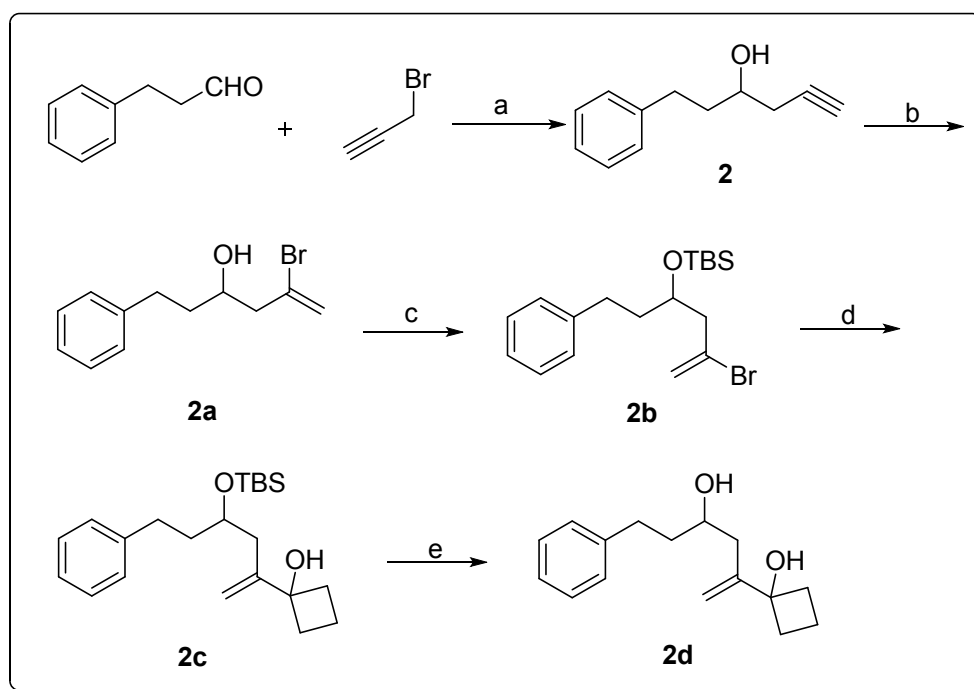
General procedure:

Scheme 1. Synthetic procedure for **1d**



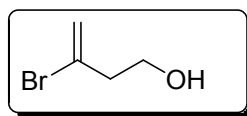
Reagents & conditions: (a) PBr_3 , H_2O , TEAB, DCM, -40°C (b) TBSCl, imidazole, DCM, 0°C to rt (c) *n*-BuLi, THF, cyclobutanone, -78°C (d) TBAF, THF.

Scheme 2. Synthetic procedure for **2d**



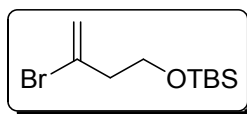
Reagents & conditions: (a) Zn, aq. NH_4Cl , THF (b) PBr_3 , H_2O , TEAB, DCM, -40°C (c) TBSCl, imidazole, DCM, 0°C to rt (d) *n*-BuLi, THF, cyclobutanone, -78°C (e) TBAF, THF.

General procedure for 3-bromobut-3-en-1-ol (**1a**):



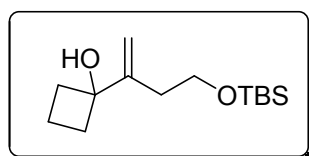
HBr gas was produced by adding PBr_3 (1.46 mL, 11 mmol) dropwise to water (0.59 mL, 33mmol). The HBr gas thus produced was bubbled through tetraethyl ammonium bromide (6.3 g) in 40 mL of dichloromethane at 0 °C after which the dichloromethane was weighed to find 2.25g of HBr (25 mmol) was absorbed by tetraethyl ammonium bromide solution. To this solution inject 3-Butyn-1 ol (1.89 mL, 12 mmol) the reaction mixture was heated at 40 °C for 5 hrs. Cooled to 0 °C and extracted with ether, dried over Na_2SO_4 , solvent was removed in *vacuo*. The crude product was used as such for further step.

General procedure for (3-bromobut-3-enyloxy)(*tert*-butyl)dimethylsilane (**1b**):



3-bromobut-3-en-1-ol **1a** (5.0 g, 33.3 mmol) was taken in to dry DCM and added imidazole (2.49 g, 36.6 mmol) at 0 °C. After ten minutes added *tert*-Butyldimethylsilyl chloride (5.0g, 33.3 mmol) and stirred at room temperature for 30 min. Ice pieces was added at 0 °C to quench the reaction. The combined organic layers were dried over anhydrous Na_2SO_4 and solvent was removed under reduced pressure. The crude residue was then purified by flash chromatography on silica gel column with hexane-ethyl acetate to give compound **1b** as a liquid.

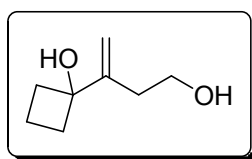
General procedure for 1-(4-(*tert*-butyldimethylsilyloxy)but-1-en-2-yl)cyclobutanol (**1c**):



n-BuLi (1.9 M solution in pentane, 2.0 equiv.) was slowly added to a solution of **1b** (260 mg, 1.0 equiv.) in anhydrous THF at -78 °C over a period of 10 min. The resultant solution was

stirred at -78 °C for 0.5 h. Cyclobutanone (75mg, 1.0 equiv.) was then added and the mixture was stirred at -78 °C for 0.5 h. The mixture was allowed to slowly reach ambient temperature. sat.NH₄Cl was added to quench the reaction, and the aqueous layer was extracted with DCM. The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was then purified by flash chromatography on silica gel column with hexane-ethyl acetate to give compound **1c** (55% yield) as a liquid.

General procedure for 1-(4-hydroxybut-1-en-2-yl)cyclobutanol (1d**):**



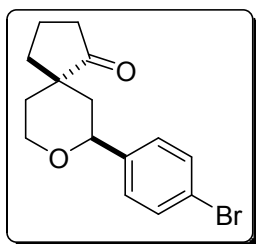
To a solution of **1c** (145 mg, 1.02 mmol) in THF (15 mL) at 0 °C was added TBAF (1.0 N in THF, 1.02 mL, 1.02 mmol). After stirring for 10 min, the reaction was quenched with sat. NH₄Cl solution and extracted with Ethyl acetate for thrice. The combined organic layers were washed with brine, dried over Na₂SO₄, and then filtered and evaporated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc) to afford **1** (95% yield) as a liquid.

liquid; ¹H NMR (500 MHz, CDCl₃): δ 5.20 (d, *J*= 0.9 Hz, 1H), 5.02 (d, *J*= 0.9 Hz, 1H), 3.81 (t, *J*= 5.8 Hz, 2H), 2.44-2.33 (m, 3H), 2.17-2.08 (m, 2H), 1.94-1.87 (m, 1H) ppm; ¹³C NMR (75 MHz, CDCl₃): δ 134.3, 111.3, 75.9, 63.1, 35.2, 34.8, 13.2 ppm; MS (*APCI*): *m/z* 165 (M+Na)⁺; HRMS (*APCI*) calculated for C₈H₁₄O₂Na: 165.0882 (M+Na)⁺, Found 165.0882.

Typical procedure for the Prins/Pinacol cyclization: To a stirred solution of homoallylic diol (**1**; 0.5 mmol) and aldehyde (0.6 mmol) in dry dichloromethane (5 mL) was added 10 mol% BF₃.OEt₂ at -40 °C. The resulting mixture was stirred at same temperature under nitrogen atmosphere for the specified time. After completion of the reaction, as indicated by TLC, the mixture was quenched with sat. NaHCO₃ solution (1.0 mL) and extracted with dichloromethane (2x5 mL). The organic layers were combined, washed with brine (5 mL), dried over anhydrous Na₂SO₄, and concentrated in *vacuo*. The resulting crude product was purified by silica gel column chromatography (100–200 mesh) using ethyl acetate/hexane as eluent to afford the pure product. This reaction was repeated in 1 gram scale to demonstrate its suitability for large scale synthesis. The results are consistent with small scale reaction (0.5 mmol).

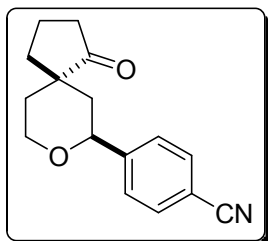
Characterization data of products:

(5*S*,7*S*)-7-(4-Bromophenyl)-8-oxaspiro[4.5]decan-1-one (3a):



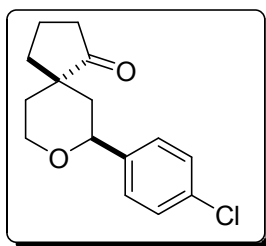
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.45 (d, J = 8.4 Hz, 2H), 7.22 (d, J = 8.2 Hz, 1H), 4.95 (dd, J = 2.3 Hz, J = 11.5 Hz, 1H), 4.08 (dt, J = 3.0 Hz, J = 11.8 Hz, 1H), 3.98 (ddd, J = 1.5 Hz, J = 4.9 Hz, J = 11.7 Hz, 1H), 2.44-2.32 (m, 2H), 2.00-1.97 (m, 2H), 1.87-1.78 (m, 3H), 1.73-1.66 (m, 2H), 1.41-1.36 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 225.8, 141.4, 130.9, 127.1, 120.7, 74.0, 45.8, 40.4, 38.3, 32.4, 18.6 ppm; MS (APCI): m/z 309 (M+H) $^+$; HRMS (APCI) calculated for $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Br}$: 309.0476 (M+H) $^+$, Found 309.0476.

4-((5*S*,7*S*)-1-Oxo-8-oxaspiro[4.5]decan-7-yl)benzotrile (3b):



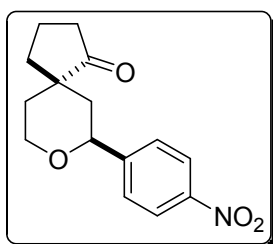
liquid; ^1H NMR (300 MHz, CDCl_3): δ 7.62 (d, J = 8.2 Hz, 2H), 7.43 (d, J = 8.1 Hz, 2H), 5.05 (dd, J = 2.1 Hz, J = 11.4 Hz, 1H), 4.10-3.98 (m, 2H), 2.44-2.10 (m, 2H), 2.01-1.90 (m, 2H), 1.88-1.70 (m, 3H), 1.48-1.37 (m, 1H) ppm; ^{13}C NMR (75 MHz, CDCl_3): δ 220.8, 147.7, 131.8, 126.0, 118.6, 110.8, 73.9, 64.0, 45.7, 40.3, 40.2, 38.3, 32.3, 18.6 ppm; MS (APCI): m/z 256 (M+H) $^+$; HRMS (APCI) calculated for $\text{C}_{16}\text{H}_{18}\text{NO}_2$: 256.1336 (M+H) $^+$, Found 256.1336.

(5*S*,7*S*)-7-(4-Chlorophenyl)-8-oxaspiro[4.5]decan-1-one (3c):



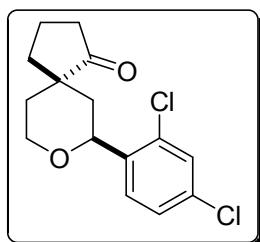
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.31-7.25 (m, 4H), 4.95 (dd, $J= 2.3$ Hz, $J= 11.5$ Hz, 1H), 4.13-3.91 (m, 2H), 2.40-2.29 (m, 2H), 1.98-1.74 (m, 4H), 1.74-1.60 (m, 2H), 1.54-1.41 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 221.9, 140.9, 132.5, 128.0, 126.8, 74.0, 64.1, 45.9, 40.4, 40.3, 38.3, 32.4, 18.6 ppm; MS (APCI): m/z 265 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Cl}$: 265.0980 ($\text{M}+\text{H}$) $^+$, Found 265.0980.

(5S,7S)-7-(4-Nitrophenyl)-8-oxaspiro[4.5]decan-1-one (3d):



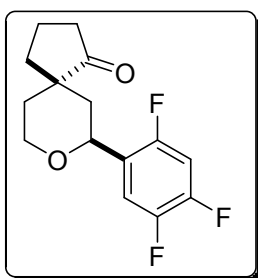
liquid; ^1H NMR (500 MHz, CDCl_3): δ 8.21 (d, $J= 8.6$ Hz, 2H), 7.53 (d, $J= 8.6$ Hz, 2H), 5.14 (dd, $J= 1.9$ Hz, $J= 11.5$ Hz, 1H), 4.14-3.98 (m, 2H), 2.49-2.30 (m, 2H), 2.05-1.94 (m, 2H), 1.91-1.70 (m, 4H), 1.54-1.43 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 220.8, 149.8, 146.6, 126.0, 123.2, 73.8, 64.0, 45.7, 40.4, 40.2, 38.2, 32.3, 18.6 ppm; MS (APCI): m/z 276 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{15}\text{H}_{18}\text{NO}_4$: 276.1235 ($\text{M}+\text{H}$) $^+$, Found 276.1235.

(5S,7S)-7-(2,4-Dichlorophenyl)-8-oxaspiro[4.5]decan-1-one (3e):



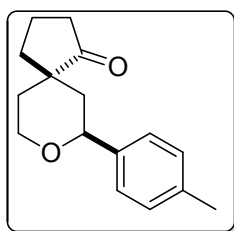
liquid; ^1H NMR (300 MHz, CDCl_3): δ 7.53-7.25 (m, 3H), 5.31 (dd, $J=2.1$ Hz, $J=11.3$ Hz, 1H), 4.26-3.96 (m, 2H), 2.50-2.34 (m, 2H), 2.13-1.62 (m, 5H), 1.38-1.24 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 220.3, 138.8, 132.7, 131.5, 128.4, 127.4, 126.9, 71.3, 64.3, 45.8, 40.1, 38.8, 38.1, 32.5, 18.6 ppm; MS (APCI): m/z 299 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{15}\text{H}_{17}\text{O}_2\text{Cl}_2$: 299.0604 ($\text{M}+\text{H}$) $^+$, Found 299.0603.

(5*S*,7*S*)-7-(2,4,5-Trifluorophenyl)-8-oxaspiro[4.5]decan-1-one (3f):



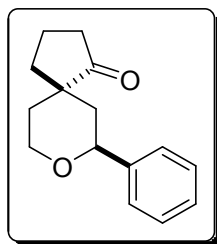
liquid; ^1H NMR (300 MHz, CDCl_3): δ 7.39-7.25 (m, 1H), 6.98-6.85 (m, 1H), 5.29-5.20 (m, 1H), 4.17-3.92 (m, 2H), 2.51-2.35 (m, 2H), 2.07-1.56 (m, 6H), 1.48-1.38 (m, 1H) ppm; ^{13}C NMR (75 MHz, CDCl_3): δ 220.4, 147.3, 126.4, 114.8, 104.9, 68.1, 64.2, 45.6, 40.2, 39.2, 38.2, 32.4, 18.6 ppm; MS (APCI): m/z 285 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{15}\text{H}_{16}\text{O}_2\text{F}_3$: 285.1089 ($\text{M}+\text{H}$) $^+$, Found 285.1088.

(5*S*,7*S*)-7-(*p*-tolyl)-8-oxaspiro[4.5]decan-1-one (3g):



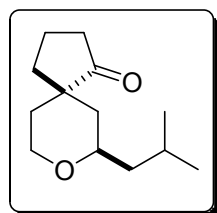
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.25-7.21 (d, 2H), δ 7.15-7.11 (d, 2H), 4.89 (dd, $J=2.2$ Hz, $J=11.5$ Hz, 1H), 4.15-4.05 (m, 1H), 3.98-3.93 (m, 1H), 2.39-2.38 (m, 5H), 1.99-1.74 (m, 5H), 1.71-1.63 (m, 2H), 1.57-1.49 (m, 1H), ppm; ^{13}C NMR (75 MHz, CDCl_3): δ 221.0, 139.3, 136.5, 128.6, 125.4, 74.6, 64.1, 45.9, 40.4, 38.2, 32.4, 21.4, 18.5 ppm; MS (APCI): m/z 244 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{16}\text{H}_{20}\text{O}_2$: 244.1461 ($\text{M}+\text{H}$) $^+$, Found 244.1459.

(5*S*,7*S*)-7-Phenyl-8-oxaspiro[4.5]decan-1-one (3h):



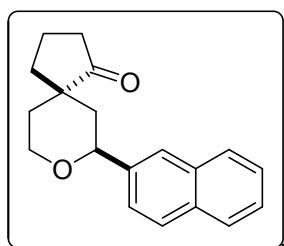
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.39-7.19 (m, 5H), 4.93 (dd, $J= 3.0$ Hz, $J= 12.0$ Hz, 1H), 4.16-3.91 (m, 2H), 2.36 (t, $J= 6.7$ Hz, 2H), 2.04-1.65 (m, 7H), 1.61-1.48 (m, 1H), ppm; ^{13}C NMR (75 MHz, CDCl_3): δ 221.0, 142.3, 127.9, 127.0, 125.4, 74.7, 64.1, 45.9, 40.3, 38.2, 32.4, 18.5 ppm; MS (APCI): m/z 231 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{15}\text{H}_{19}\text{O}_2$: 231.1385 ($\text{M}+\text{H}$) $^+$, Found 231.1387.

(5S,7R)-7-Isobutyl-8-oxaspiro[4.5]decan-1-one (3i):



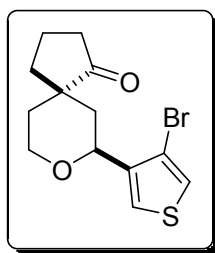
liquid; ^1H NMR (500 MHz, CDCl_3): δ 4.00-3.77 (m, 3H), 2.41-2.28 (m, 2H), 2.03-1.89 (m, 2H), 1.85-1.74 (m, 2H), 1.68-1.57 (m, 3H), 1.38-1.09 (m, 2H), 0.92 (d, $J= 6.5$ Hz, 6H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 220.9, 70.7, 63.7, 46.0, 45.6, 40.5, 38.9, 38.2, 32.7, 24.7, 23.6, 22.8, 18.5 ppm; MS (APCI): m/z 211 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{13}\text{H}_{23}\text{O}_2$: 211.1688 ($\text{M}+\text{H}$) $^+$, Found 211.1687.

(5S,7S)-7-(Naphthalen-2-yl)-8-oxaspiro[4.5]decan-1-one (3j):



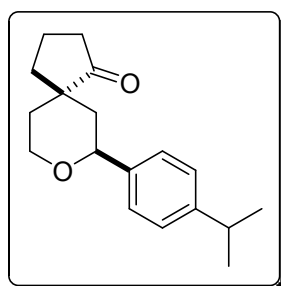
liquid; ^1H NMR (500 MHz, CDCl_3): δ 8.05 (d, $J = 7.9$ Hz, 1H), 7.91-7.63 (m, 3H), 7.53-7.48 (m, 2H), 5.84 (dd, $J = 1.7$ Hz, $J = 11.3$ Hz, 1H), 4.32-4.05 (m, 2H), 2.54-2.38 (m, 2H), 2.18-1.63 (m, 6H) ppm; ^{13}C NMR (75 MHz, CDCl_3): δ 224.0, 153.3, 144.1, 132.3, 129.7, 128.7, 127.1, 126.6, 126.4, 124.2, 123.5, 73.1, 66.1, 60.2, 47.7, 41.7, 41.5, 39.7, 34.2, 20.0 ppm; MS (APCI): m/z 281 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{19}\text{H}_{21}\text{O}_2$: 281.1527 ($\text{M}+\text{H}$) $^+$, Found 281.1527.

(5*S*,7*S*)-7-(4-Bromothiophen-3-yl)-8-oxaspiro[4.5]decan-1-one (3k):



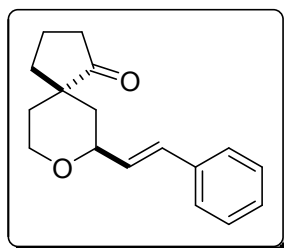
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.14 (d, $J = 1.4$ Hz, 1H), 6.88-6.86 (m, 1H), 5.20 (dd, $J = 2.4$ Hz, $J = 11.4$ Hz, 1H), 4.08 (dt, $J = 3.3$ Hz, $J = 11.7$ Hz, 1H), 3.97-3.93 (m, 2H), 2.44-2.30 (m, 2H), 2.01-1.93 (m, 1H), 1.87-1.82 (m, 2H), 1.74-1.65 (m, 2H), 1.47-1.40 (m, 1H) ppm; ^{13}C NMR (75 MHz, CDCl_3): δ 212.0, 154.2, 132.0, 130.4, 128.4, 125.6, 121.3, 70.4, 68.2, 64.2, 39.9, 32.2, 30.0, 29.2, 24.1, 18.6 ppm; MS (APCI): m/z 315 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{13}\text{H}_{16}\text{BrO}_2\text{S}$: 315.0055 ($\text{M}+\text{H}$) $^+$, Found 315.0053.

(5*S*,7*S*)-7-(4-Isopropylphenyl)-8-oxaspiro[4.5]decan-1-one (3l):



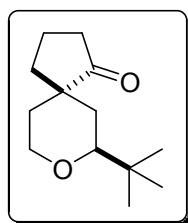
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.29-7.25 (m, 2H), 7.23-7.19 (m, 2H), 4.91 (dd, $J = 2.3$ Hz, $J = 11.5$ Hz, 1H), 4.15-4.09 (m, 1H), 3.99-3.94 (m, 1H), 2.94-2.82 (m, 1H), 2.40-2.36 (m, 2H), 1.98-1.79 (m, 4H), 1.74-1.70 (m, 2H), 1.24 (d, $J = 6.9$ Hz, 6H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 213.1, 147.2, 139.3, 126.8, 125.7, 125.3, 74.7, 64.2, 46.1, 40.6, 40.4, 38.4, 34.3, 32.7, 24.6, 18.8 ppm; MS (APCI): m/z 273 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{18}\text{H}_{25}\text{O}_2$: 273.1854 ($\text{M}+\text{H}$) $^+$, Found 273.1857.

(5*S*,7*S*)-7-Styryl-8-oxaspiro[4.5]decan-1-one (3m):



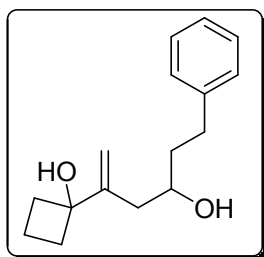
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.45-7.15 (m, 5H), 6.66 (d, J = 15.9 Hz, 1H), 6.14 (dd, J = 6.0 Hz, J = 15.9 Hz, 1H), 4.61 (dd, J = 6.2 Hz, J = 10.7 Hz, 1H), 4.05 (dt, J = 3.1 Hz, J = 11.5 Hz, 1H), 3.96-3.91 (m, 1H), 2.38 (t, J = 7.5 Hz, 1H), 2.03-1.80 (m, 5H), 1.78-1.68 (m, 2H), 1.46 (t, J = 12.4 Hz, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 220.8, 136.4, 129.9, 129.7, 128.0, 127.1, 126.0, 73.0, 63.6, 45.5, 40.2, 38.4, 38.2, 32.4, 18.5 ppm; MS (APCI): m/z 257 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{17}\text{H}_{21}\text{O}_2$: 257.1528 ($\text{M}+\text{H}$) $^+$, Found 257.1528.

(5*S*,7*S*)-7-*Tert*-butyl-8-oxaspiro[4.5]decan-1-one (3n):



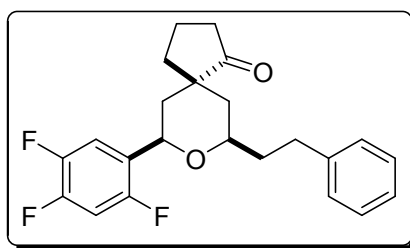
liquid; ^1H NMR (500 MHz, CDCl_3): δ 3.92-3.80 (m, 2H), 3.48 (d, J = 13.6 Hz, 1H), 2.33 (t, J = 7.7 Hz, 2H), 1.94 (m, 2H), 1.83-1.77 (m, 2H), 1.66-1.60 (m, 2H), 1.55-1.49 (m, 1H), 1.32-1.23 (m, 1H), 0.89 (s, 9H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 221.0, 79.8, 64.1, 45.6, 40.8, 38.2, 34.1, 32.8, 32.5, 26.2, 18.5 ppm; MS (APCI): m/z 211 ($\text{M}+\text{H}$) $^+$; HRMS (APCI) calculated for $\text{C}_{13}\text{H}_{23}\text{O}_2$: 211.1698 ($\text{M}+\text{H}$) $^+$, Found 211.1693.

1-(4-Hydroxy-6-phenylhex-1-en-2-yl)cyclobutanol (2d):



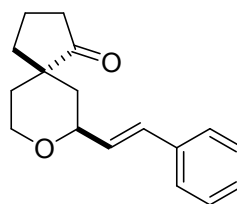
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.34-7.14 (m, 5H), 5.21 (s, 1H), 4.99 (s, 1H), 3.83-3.75 (m, 1H), 2.86-2.62 (m, 4H), 2.47-2.35 (m, 2H), 2.29-2.20 (m, 4H), 1.92-1.80 (m, 3H), 1.58-1.51 (m, 1H), 0.96-0.92 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 148.2, 141.4, 127.9, 125.4, 112.2, 77.5, 71.6, 39.9, 39.4, 35.9, 34.8, 32.5, 13.2 ppm; MS (*ESI*): m/z 247 ($\text{M}+\text{H}$) $^+$; HRMS (*ESI*) calculated for $\text{C}_{16}\text{H}_{23}\text{O}_2$: 247.1698 ($\text{M}+\text{H}$) $^+$, Found 247.1698.

(5R,7R,9R)-7-Phenethyl-9-(2,4,5-trifluorophenyl)-8-oxaspiro[4.5]decan-1-one (4):



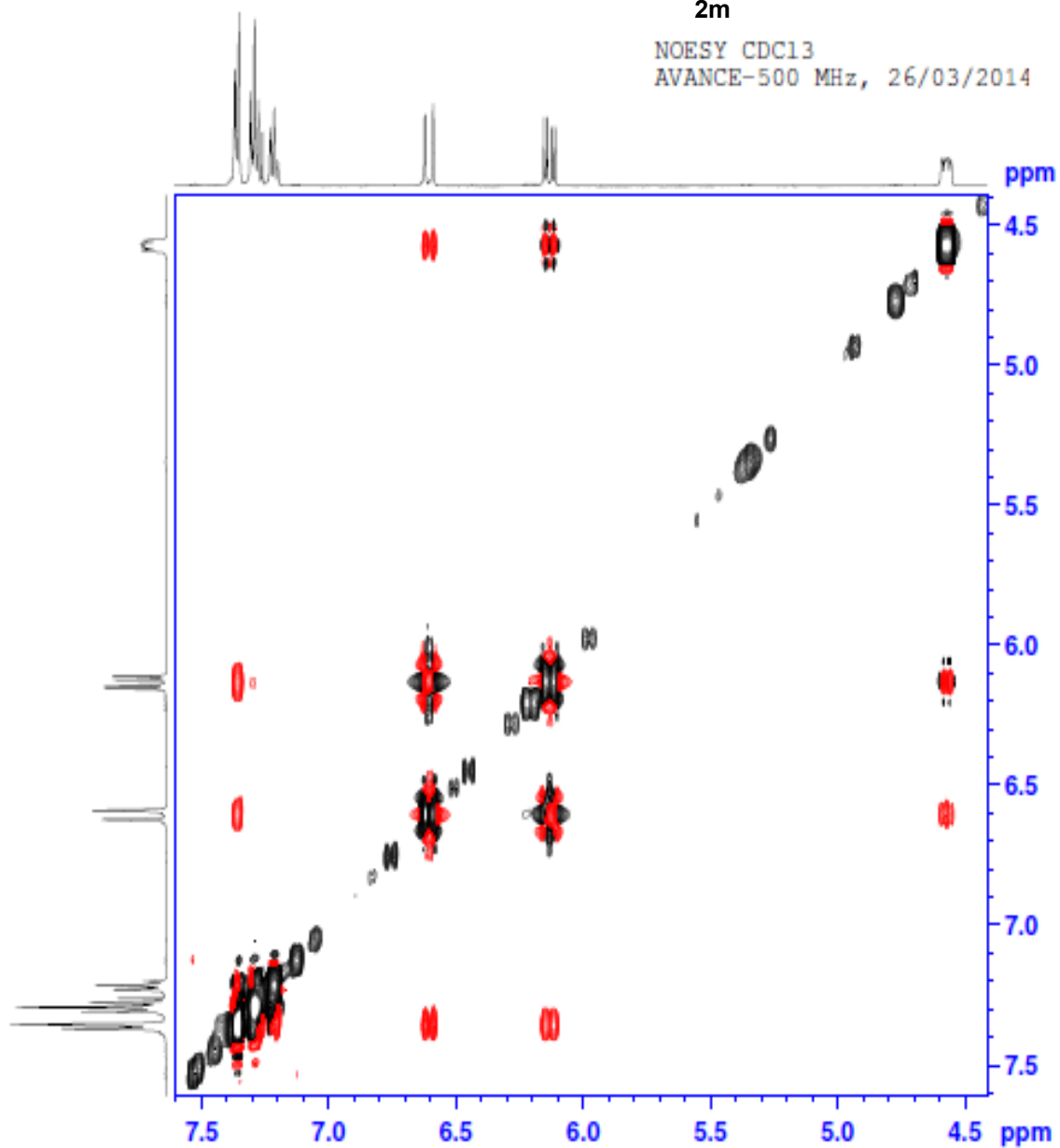
liquid; ^1H NMR (500 MHz, CDCl_3): δ 7.37-7.27 (m, 3H), 7.20-7.16 (m, 3H), 6.90-6.85 (m, 1H), 5.23 (d, $J=10.8$ Hz, 1H), 4.13-4.08 (m, 1H), 2.88-2.79 (m, 1H), 2.72-2.65 (m, 1H), 2.40-2.34 (m, 2H), 2.00-1.72 (m, 7H), 1.35-1.27 (m, 3H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 220.4, 141.7, 127.9, 125.3, 114.8 (dd), 104.8 (q), 72.5, 67.9, 46.4, 40.2, 38.9, 38.4, 38.2, 37.9, 32.1, 18.6 ppm; MS (*APCI*): m/z 389 ($\text{M}+\text{H}$) $^+$; HRMS (*APCI*) calculated for $\text{C}_{23}\text{H}_{24}\text{F}_3\text{O}_2$: 389.1723 ($\text{M}+\text{H}$) $^+$, Found 389.1724.

2D-NOESY spectrum of (5*S*,7*S*)-7-styryl-8-oxaspiro[4.5]decan-1-one (3m):

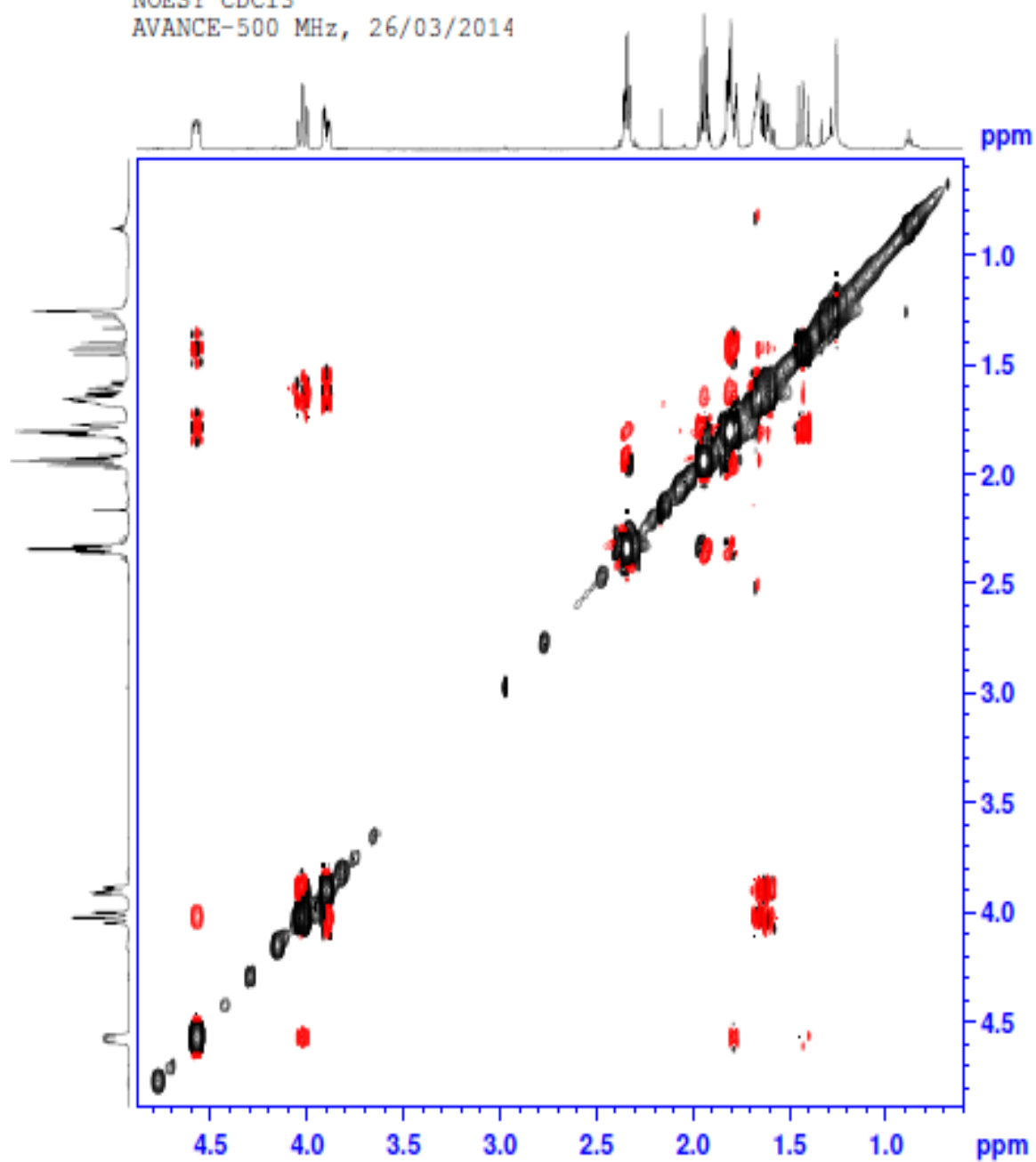


2m

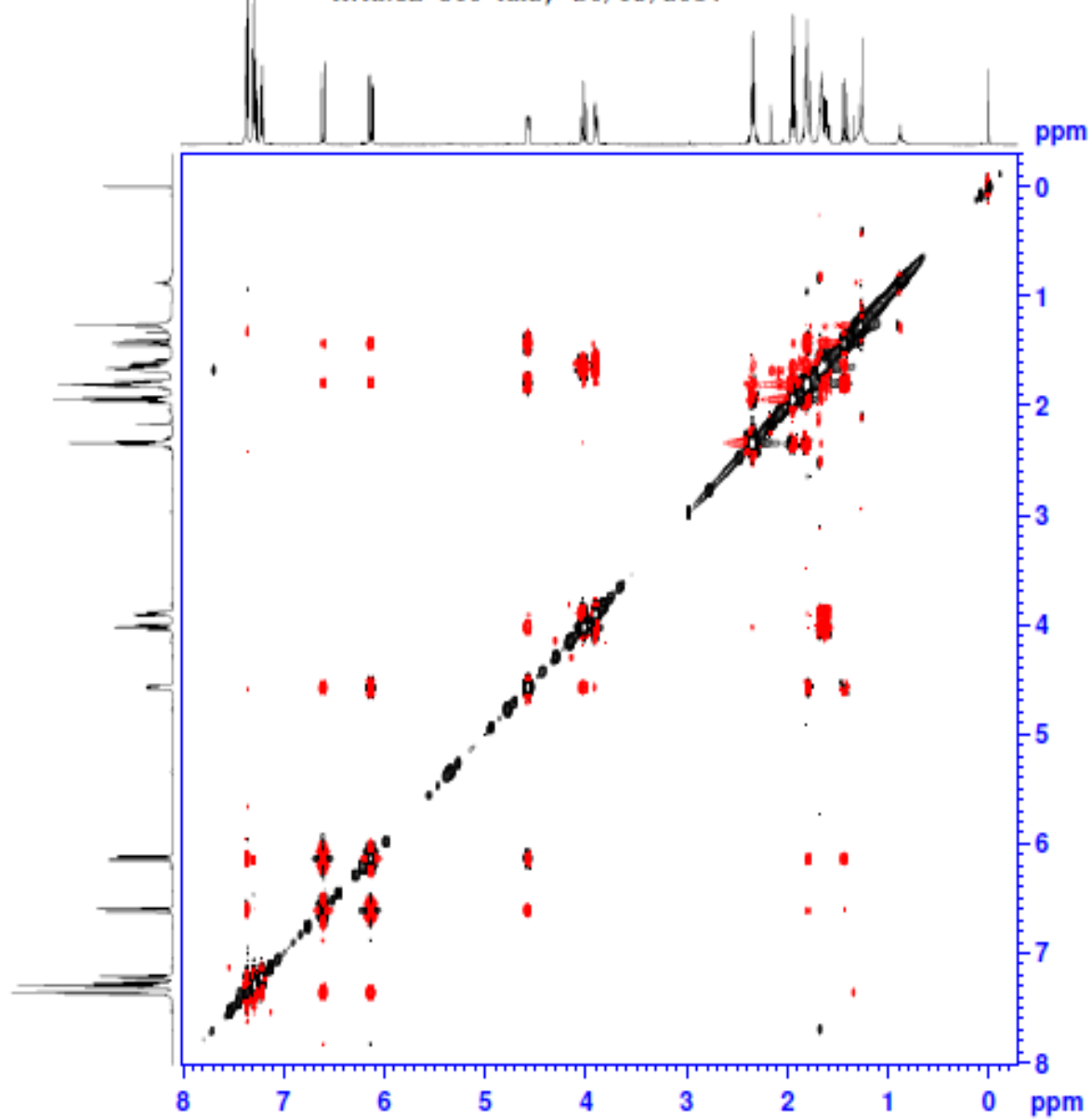
NOESY CDC13
AVANCE-500 MHz, 26/03/2014



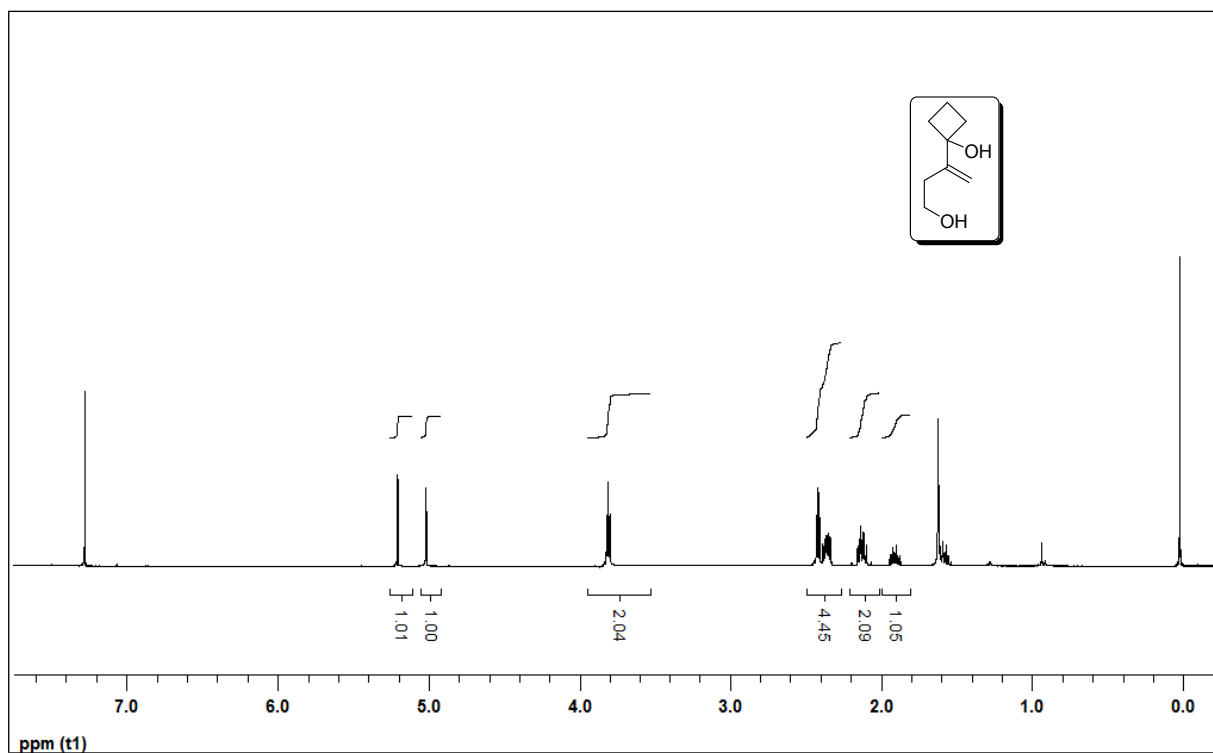
NOESY CDC13
AVANCE-500 MHz, 26/03/2014



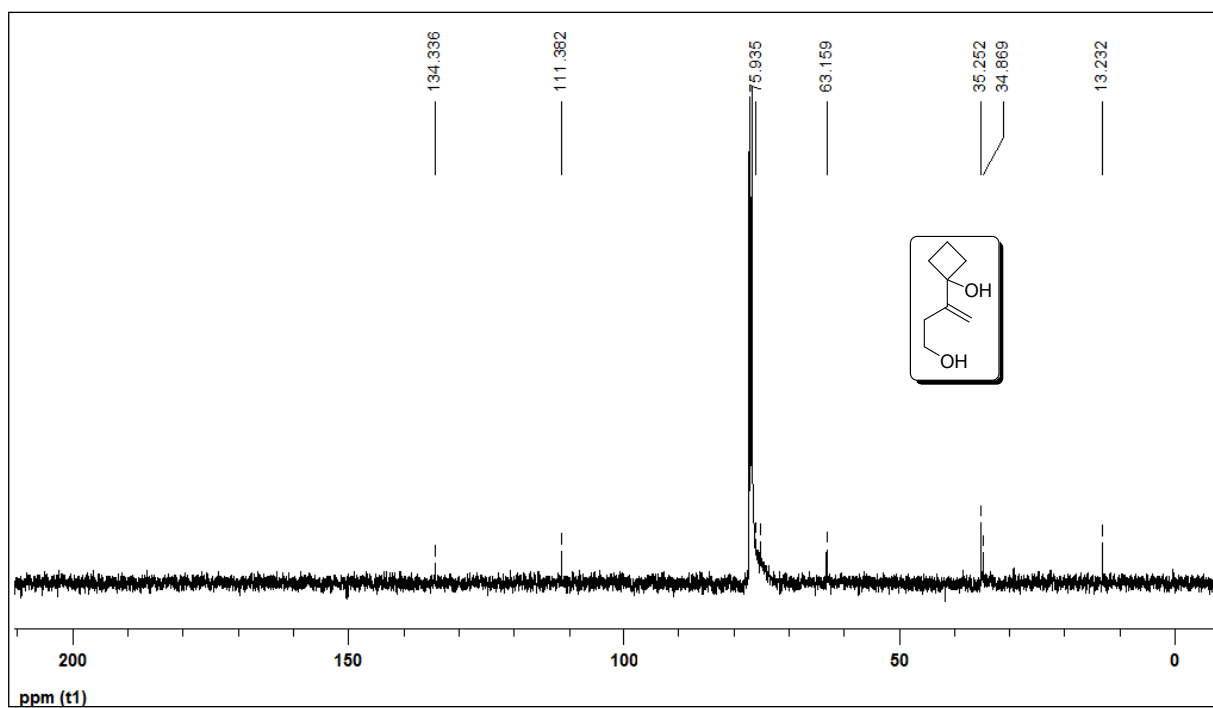
NOESY CDC13
AVANCE-500 MHz, 26/03/2014



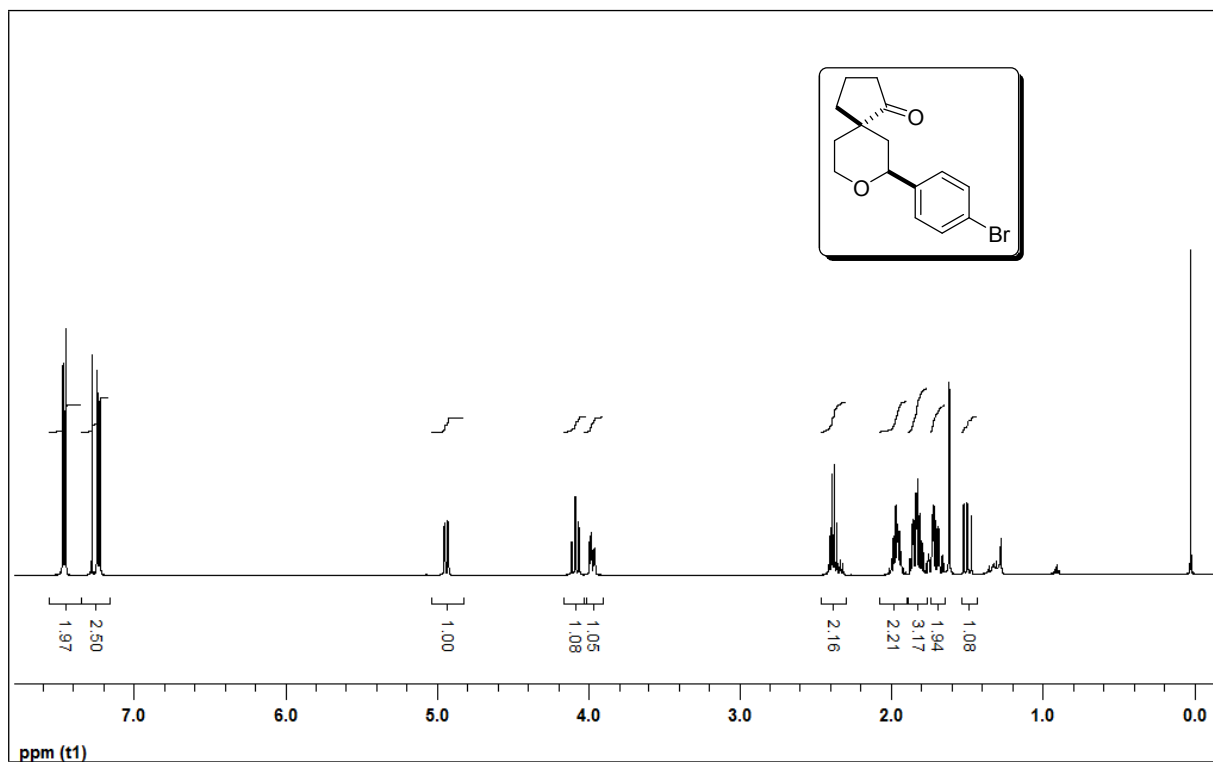
Copies of ^1H and ^{13}C NMR spectra:



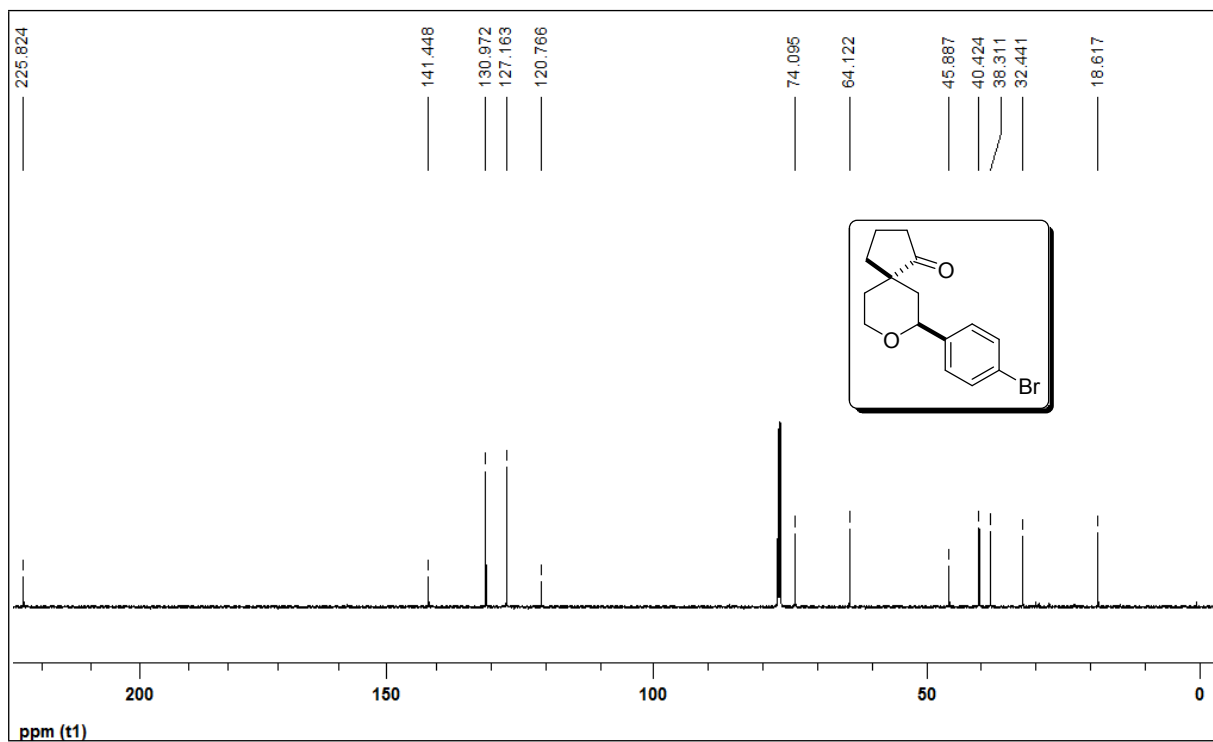
^1H NMR (500 MHz, CDCl_3) spectrum of compound 1d



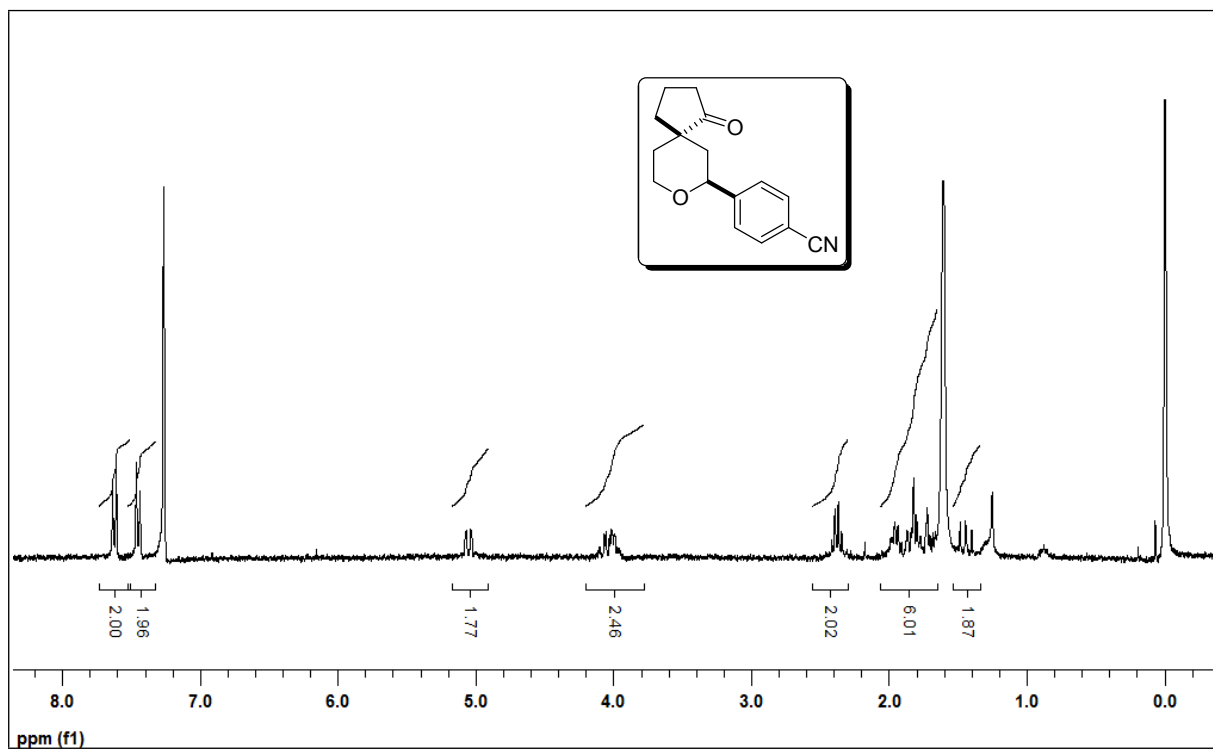
^{13}C NMR (75 MHz, CDCl_3) spectrum of compound 1d



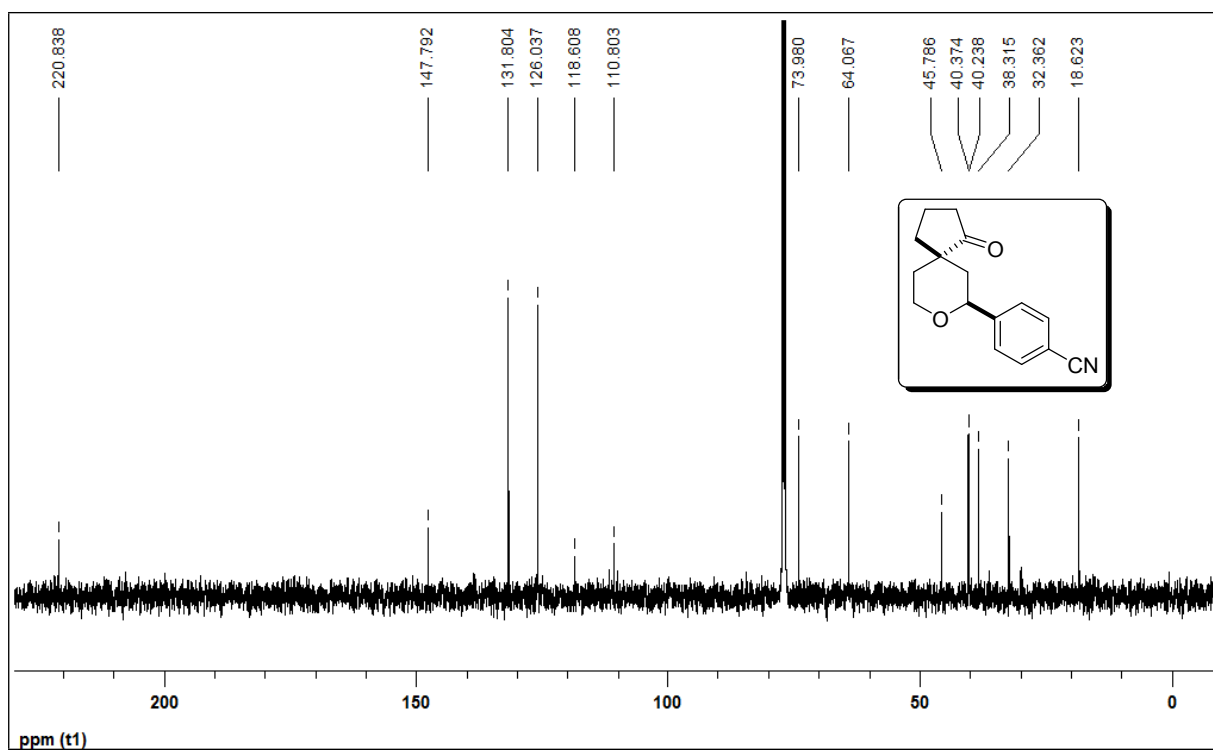
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3a



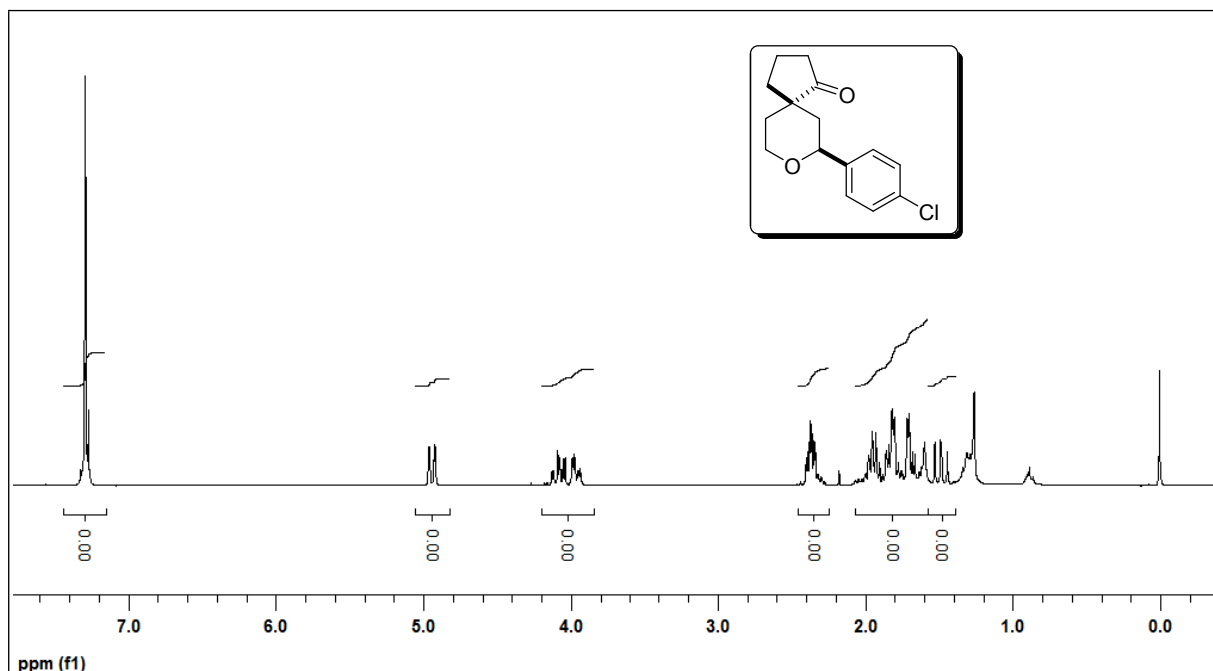
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3a



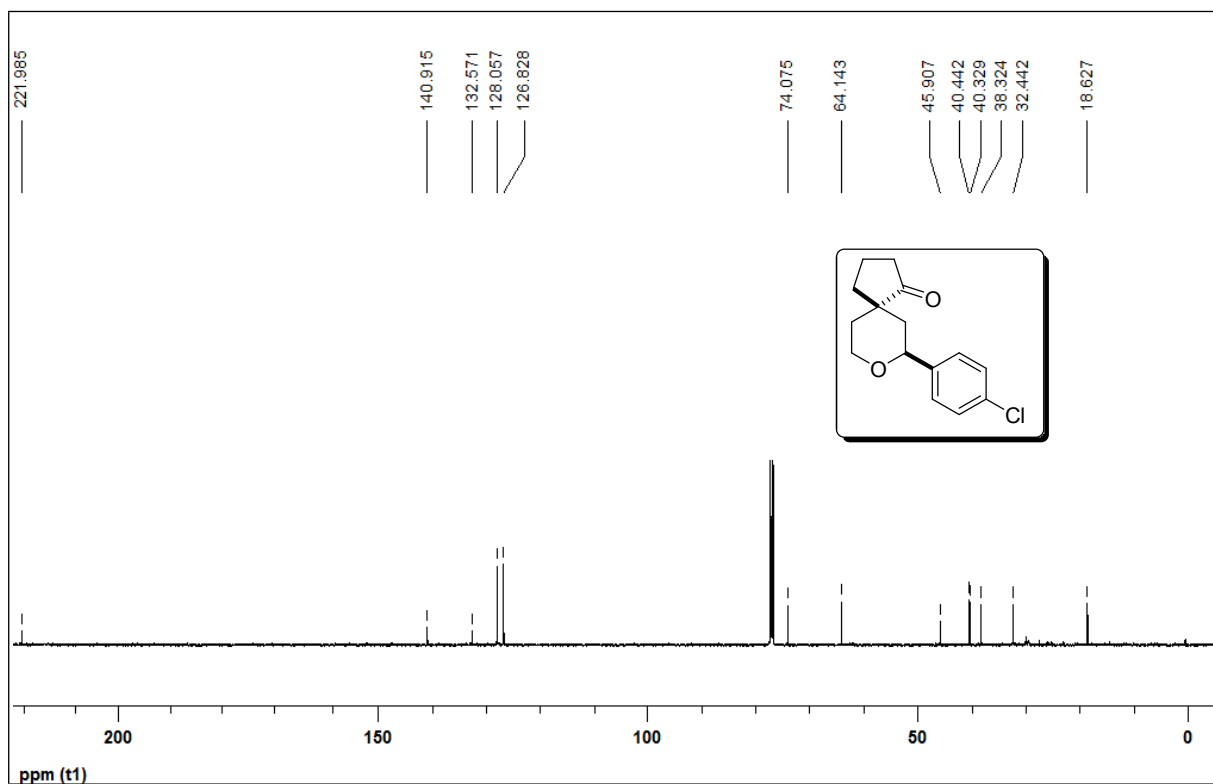
¹H NMR (300 MHz, CDCl₃) spectrum of compound 3b



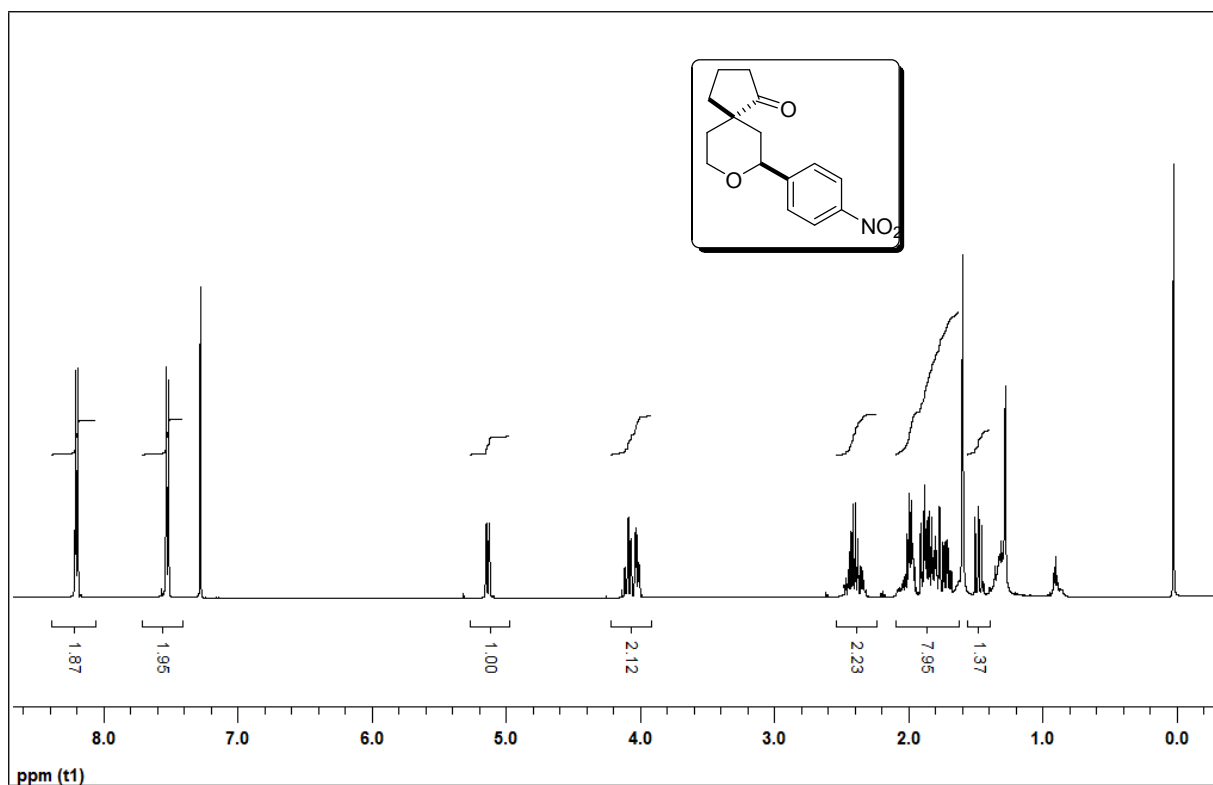
¹³C NMR (75 MHz, CDCl₃) spectrum of compound 3b



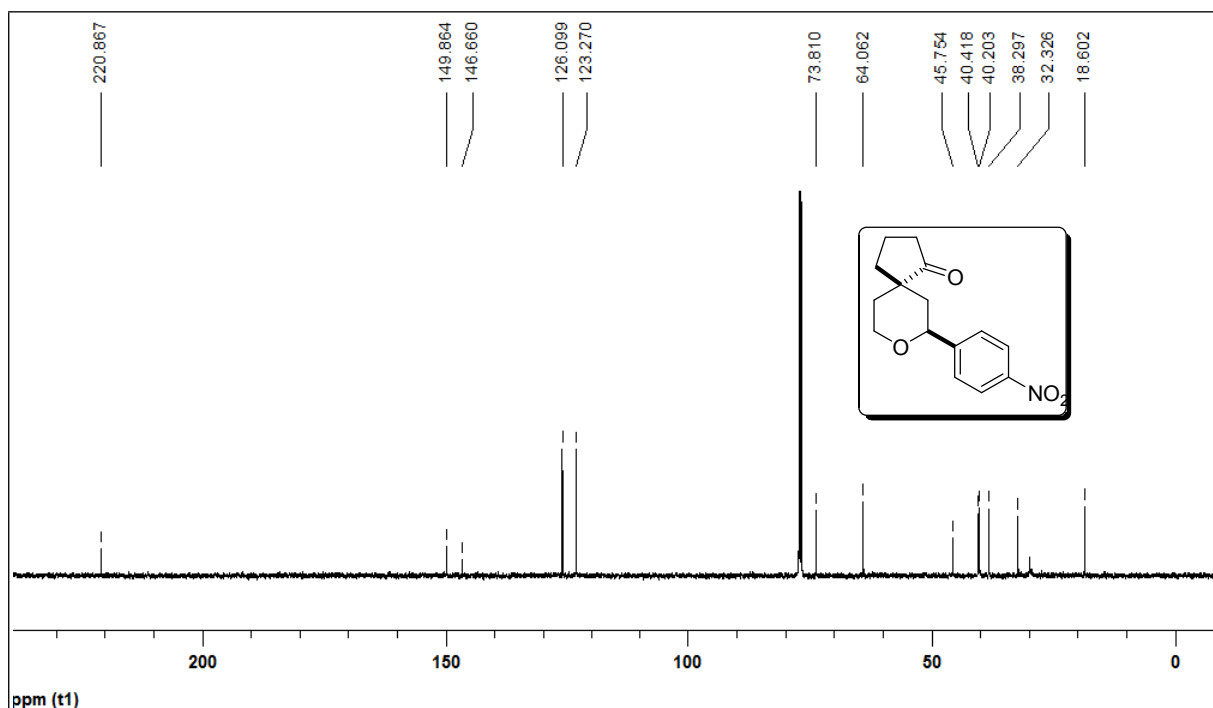
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3c



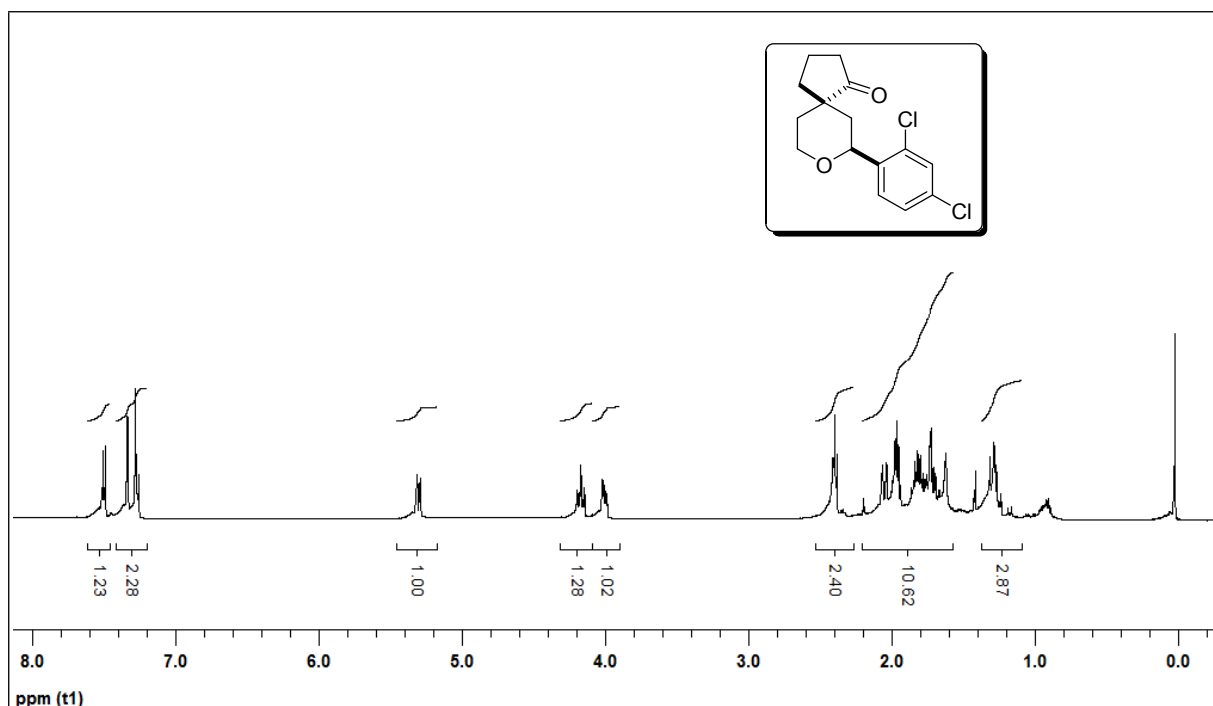
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3c



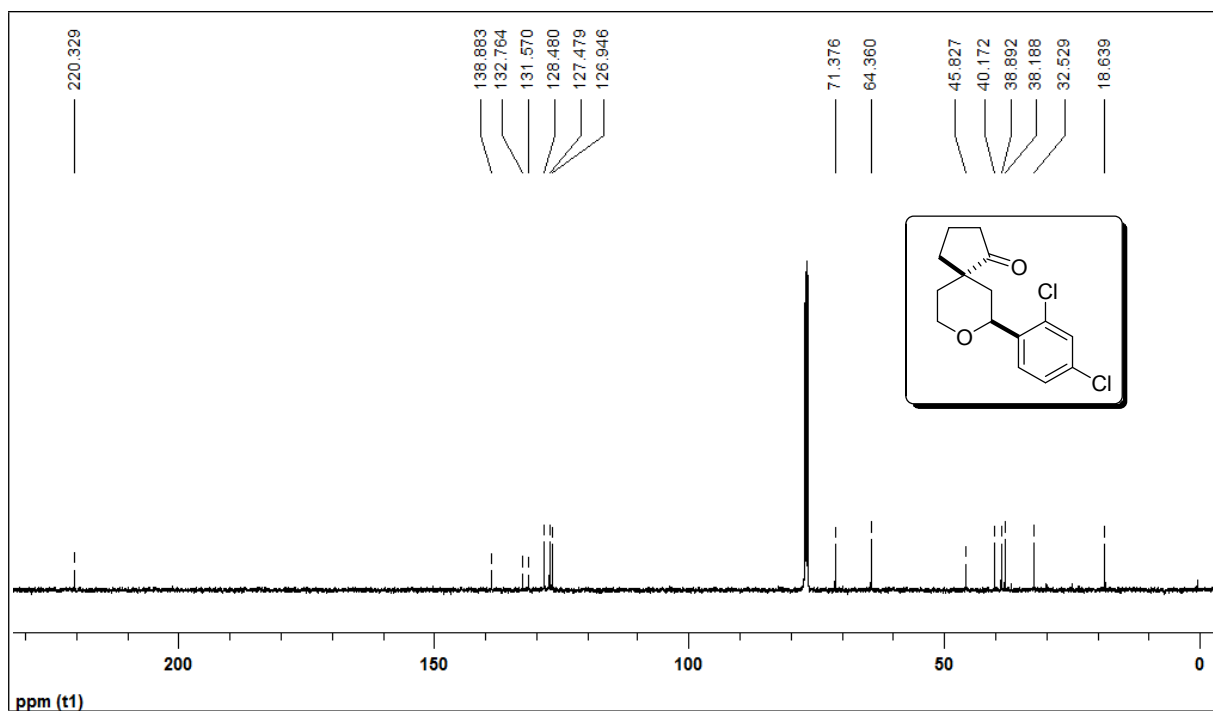
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3d



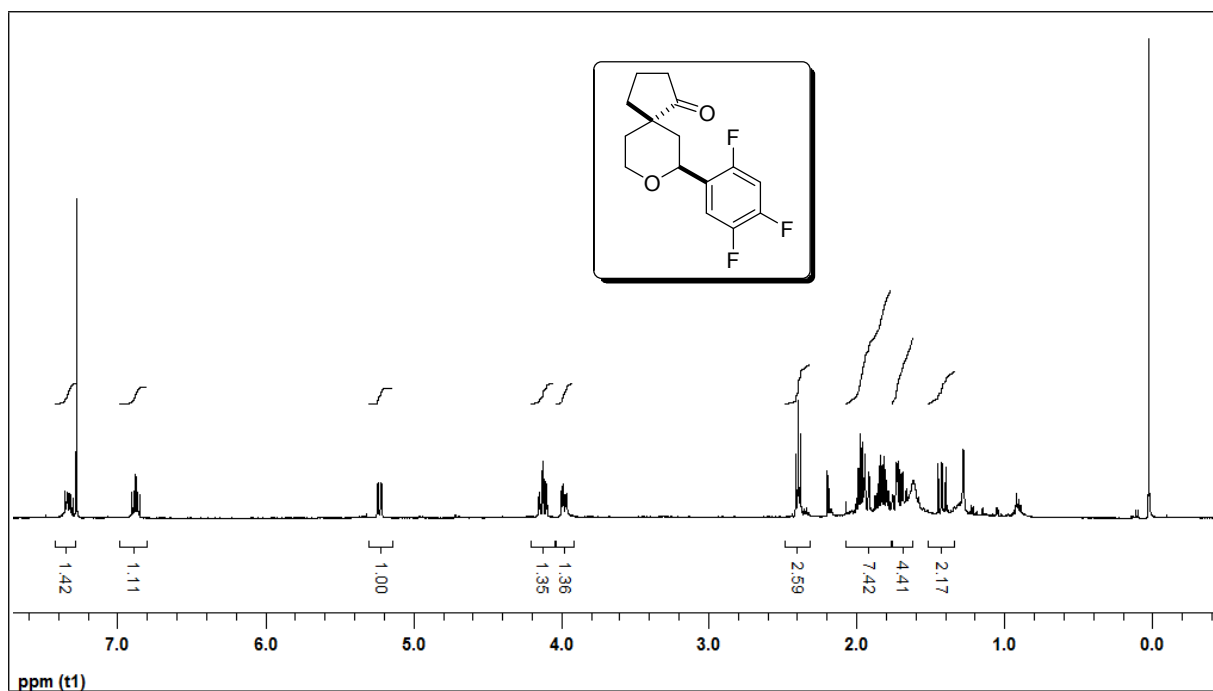
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3d



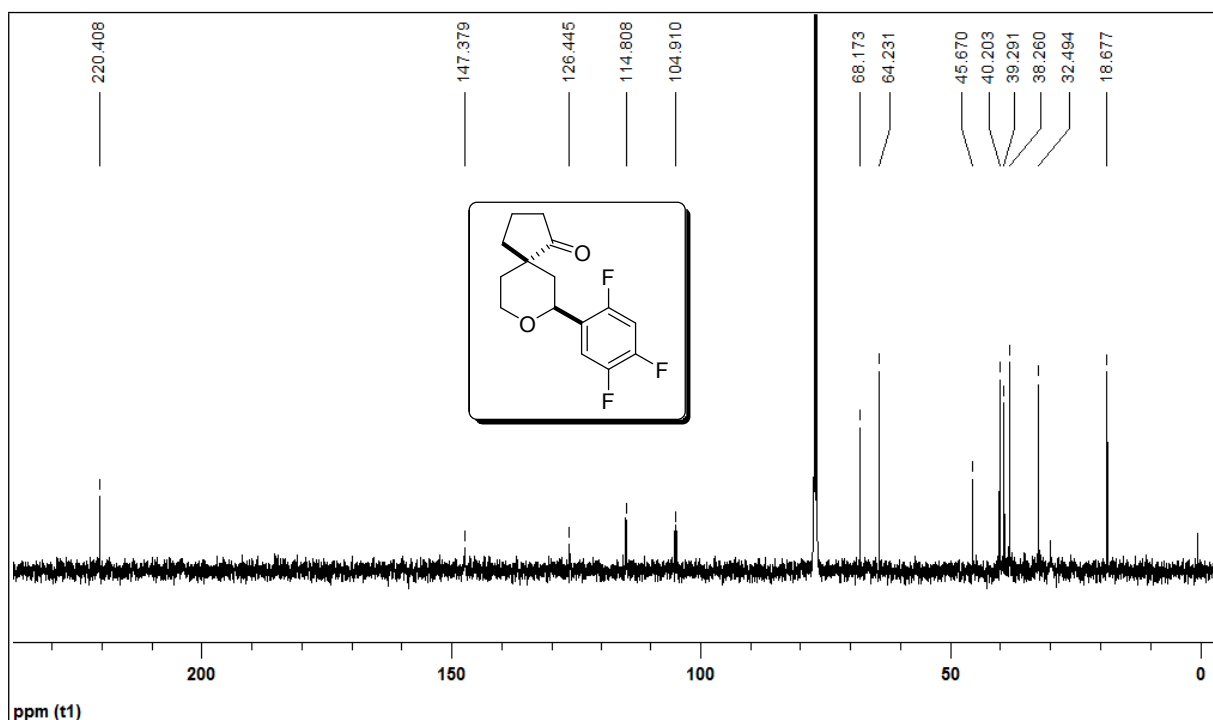
¹H NMR (300 MHz, CDCl₃) spectrum of compound 3e



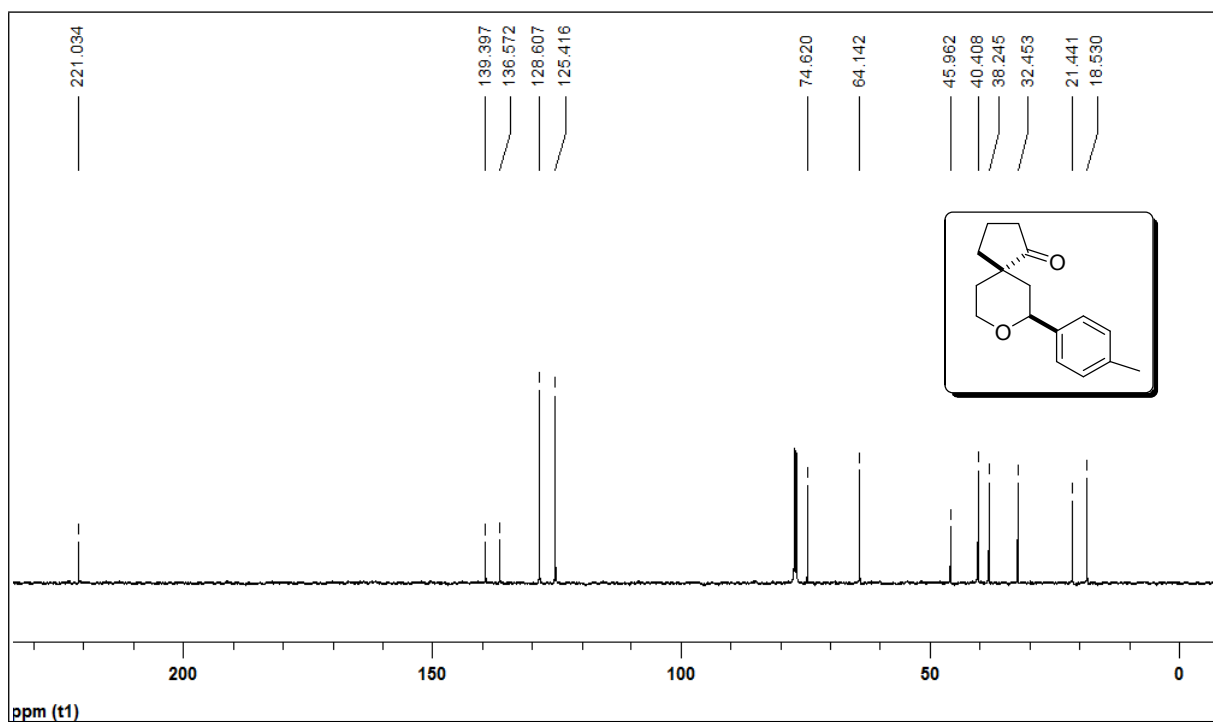
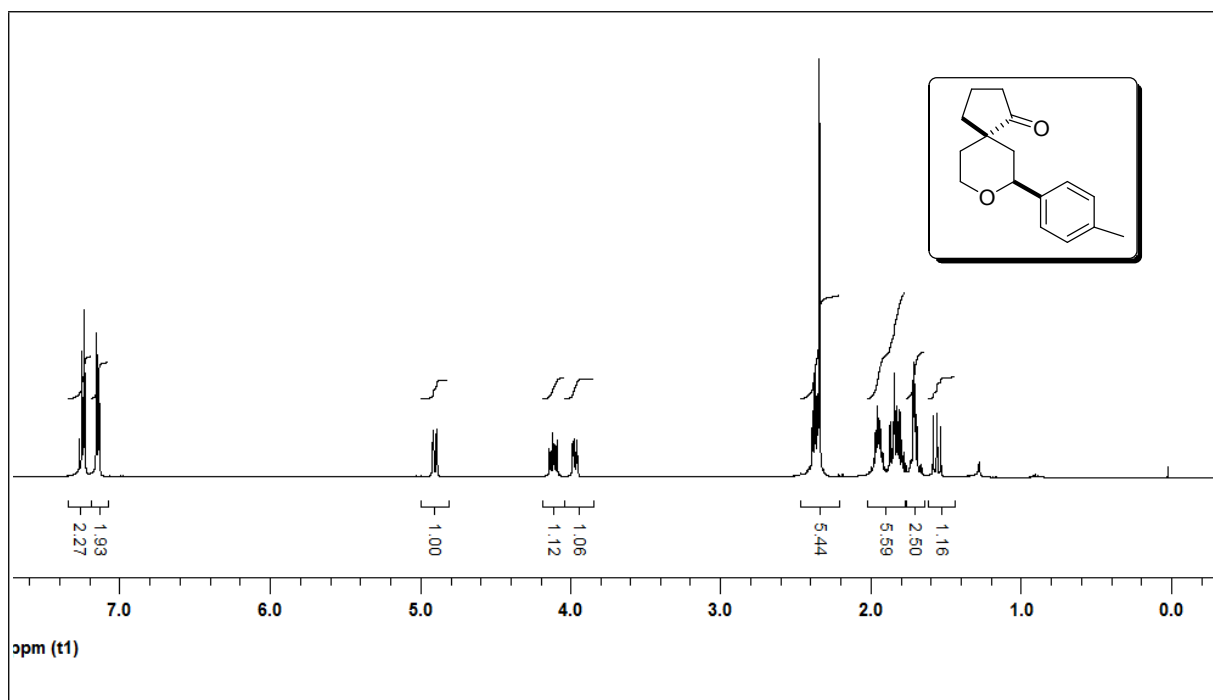
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3e

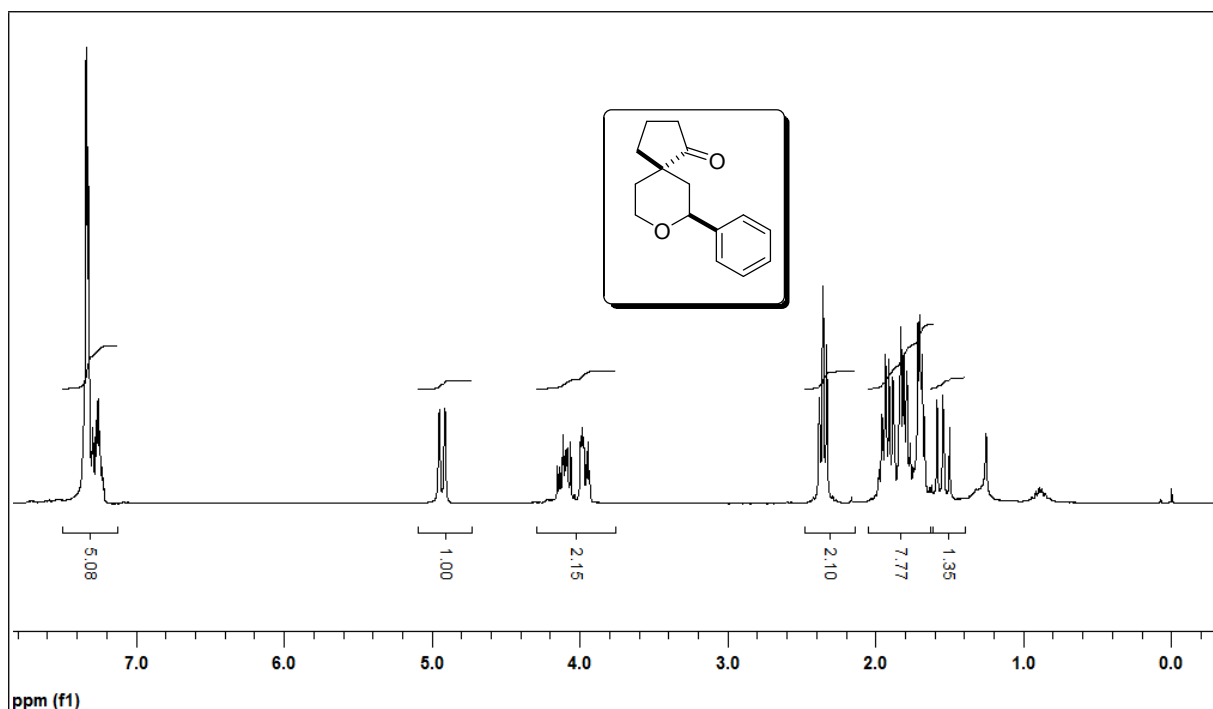


¹H NMR (300 MHz, CDCl₃) spectrum of compound 3f

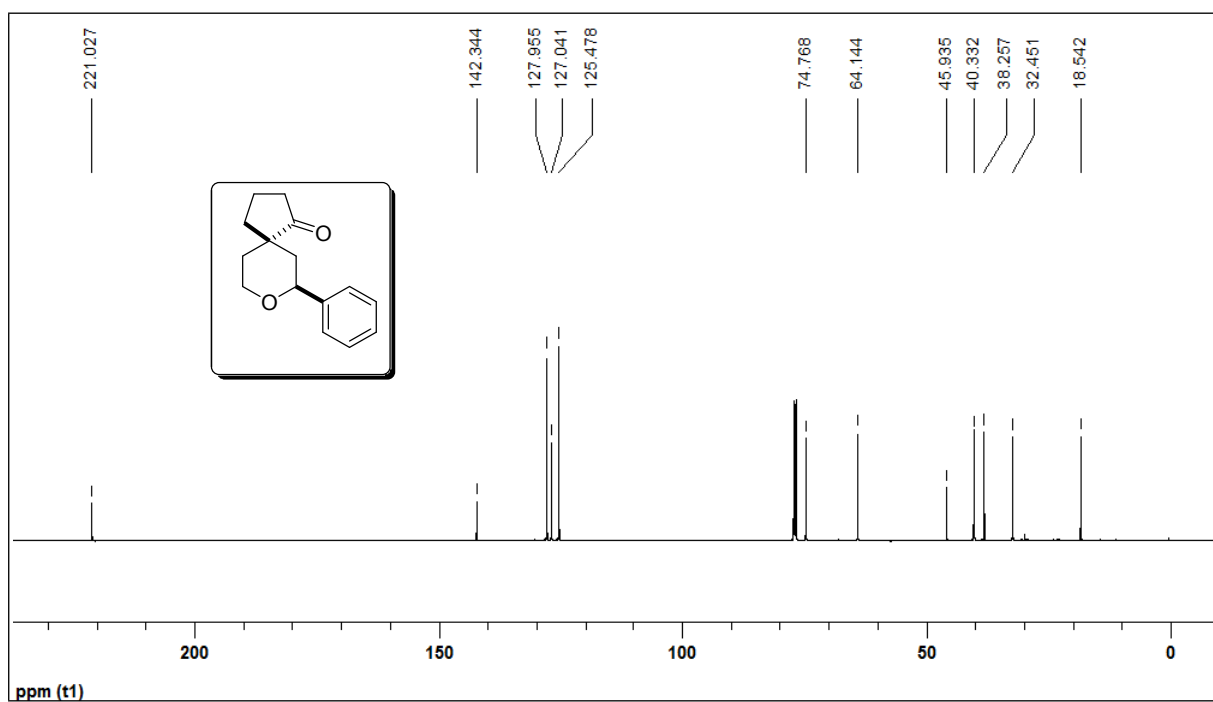


¹³C NMR (75 MHz, CDCl₃) spectrum of compound 3f

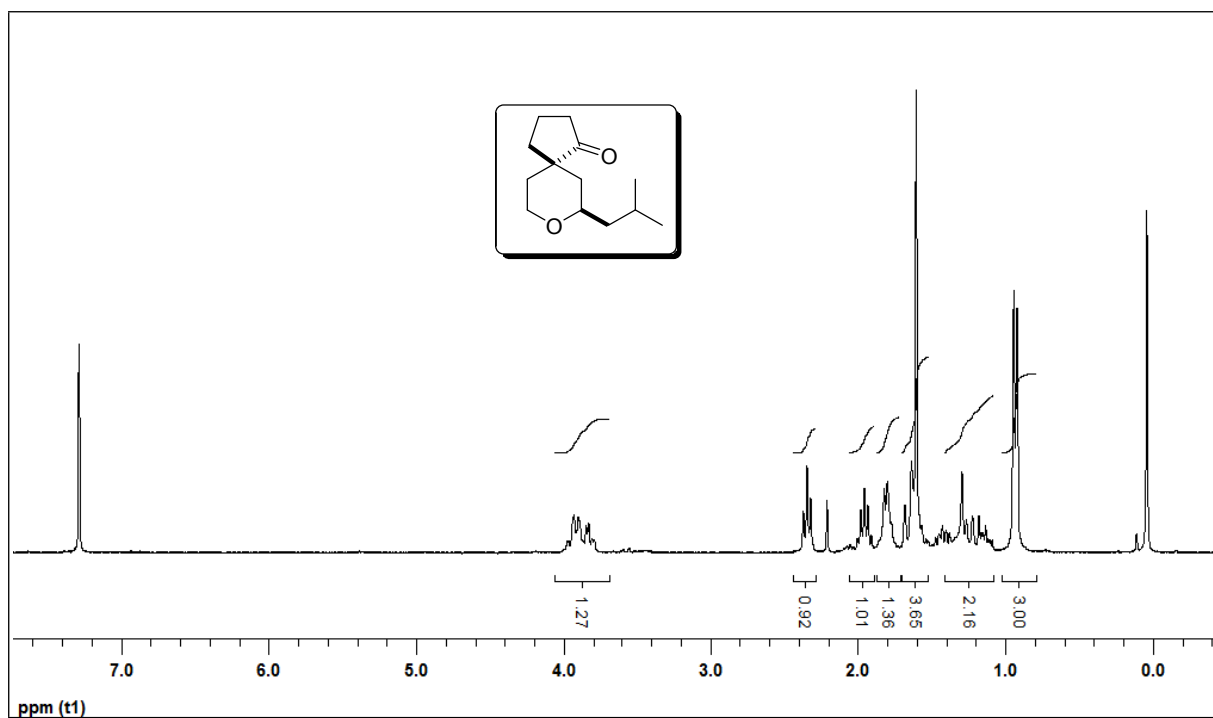




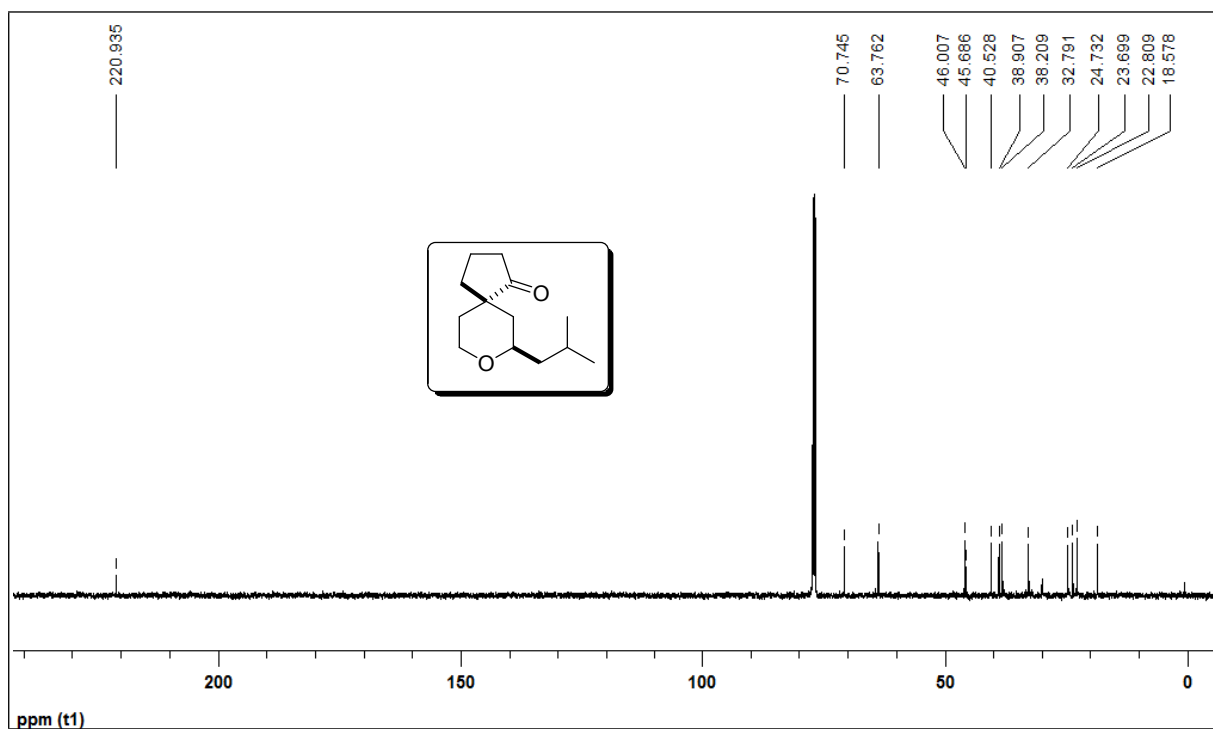
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3h



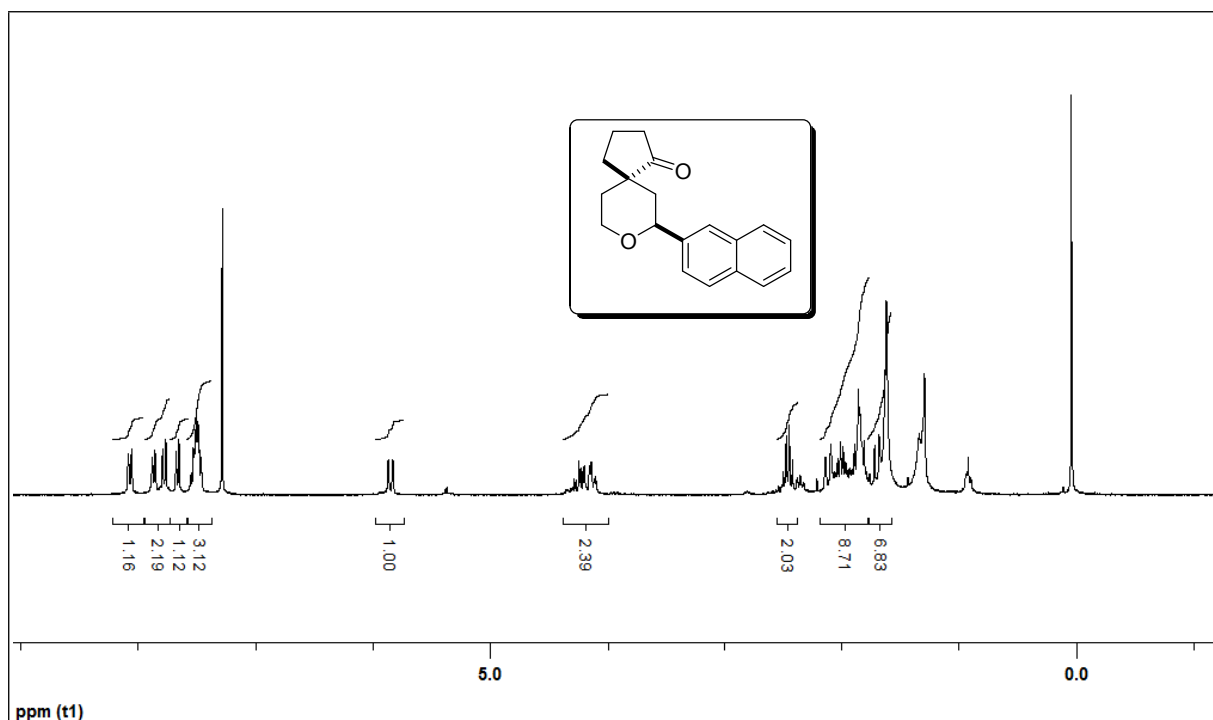
¹³C NMR (75 MHz, CDCl₃) spectrum of compound 3h



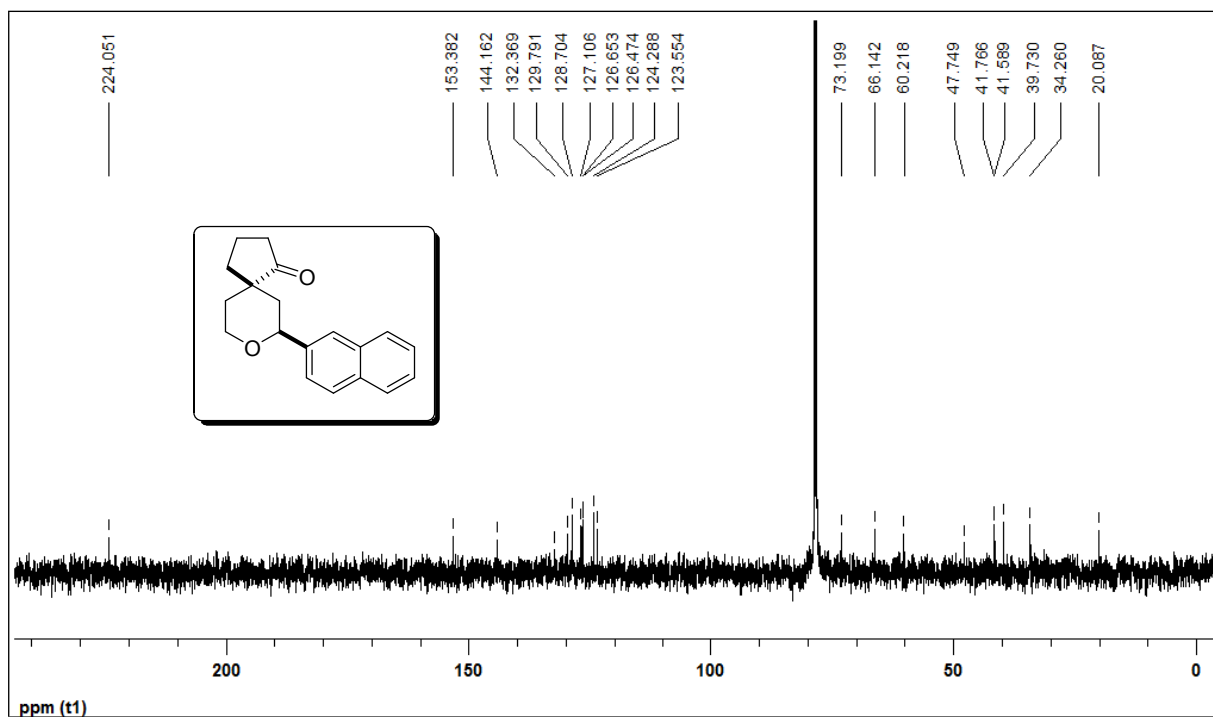
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3i



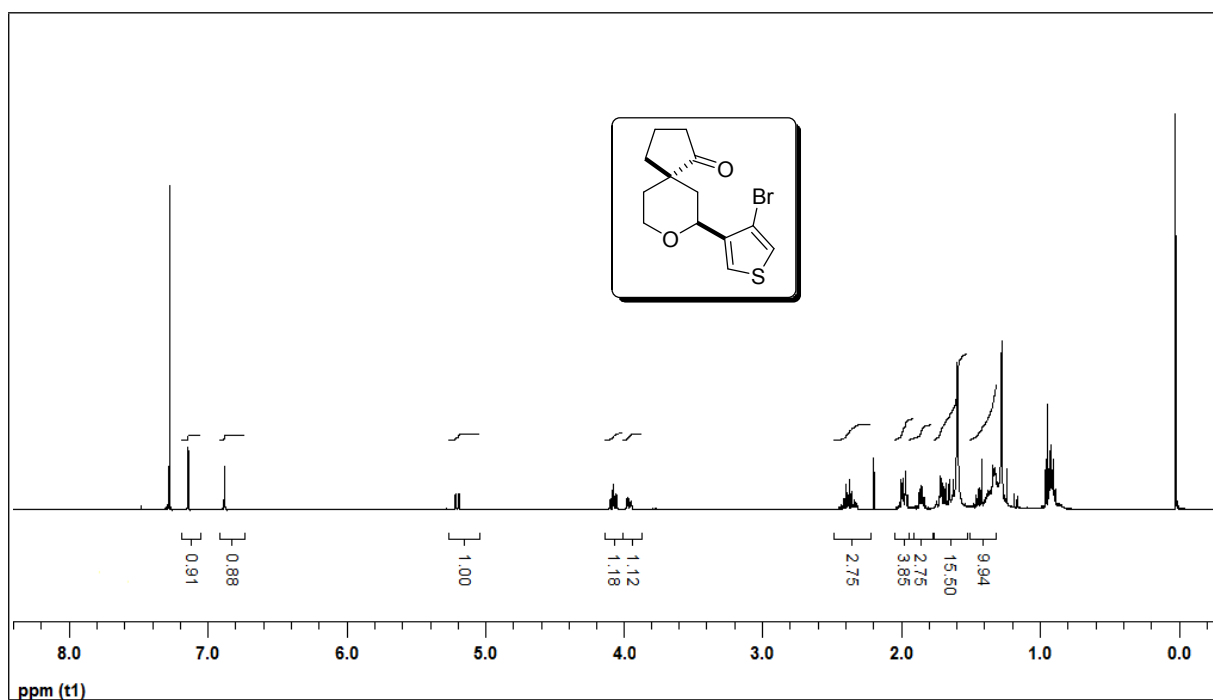
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3i



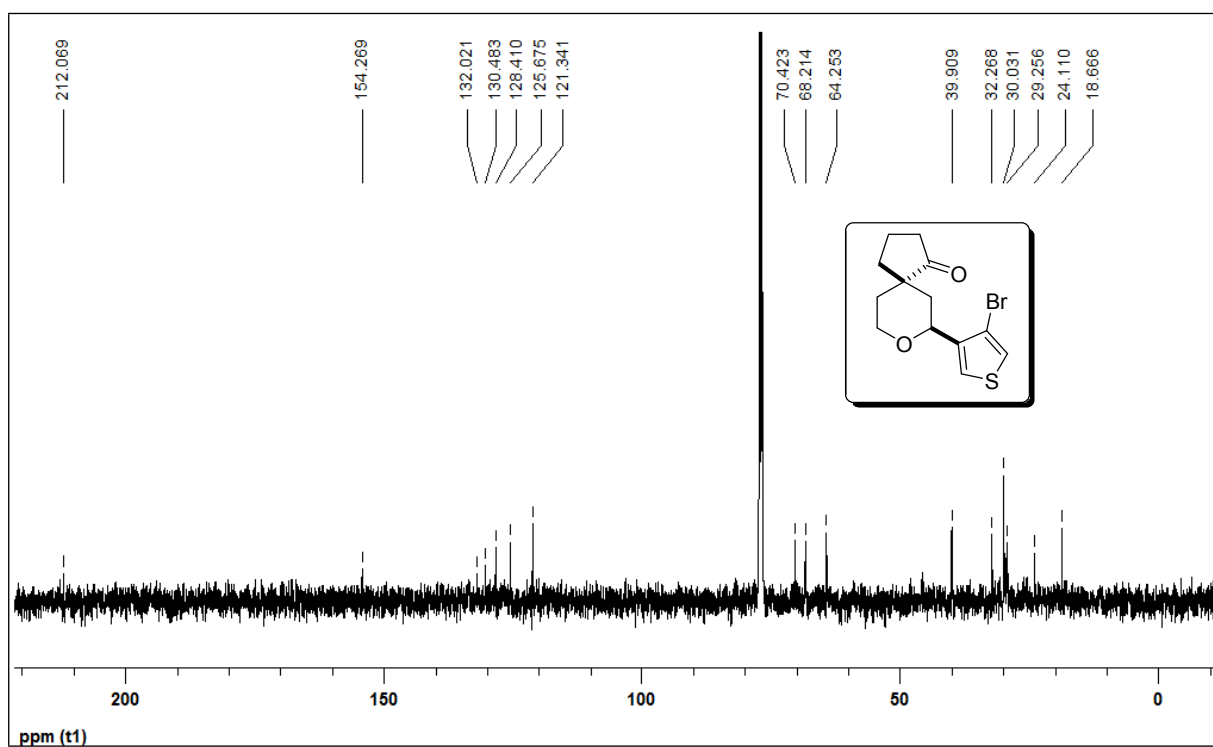
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3j



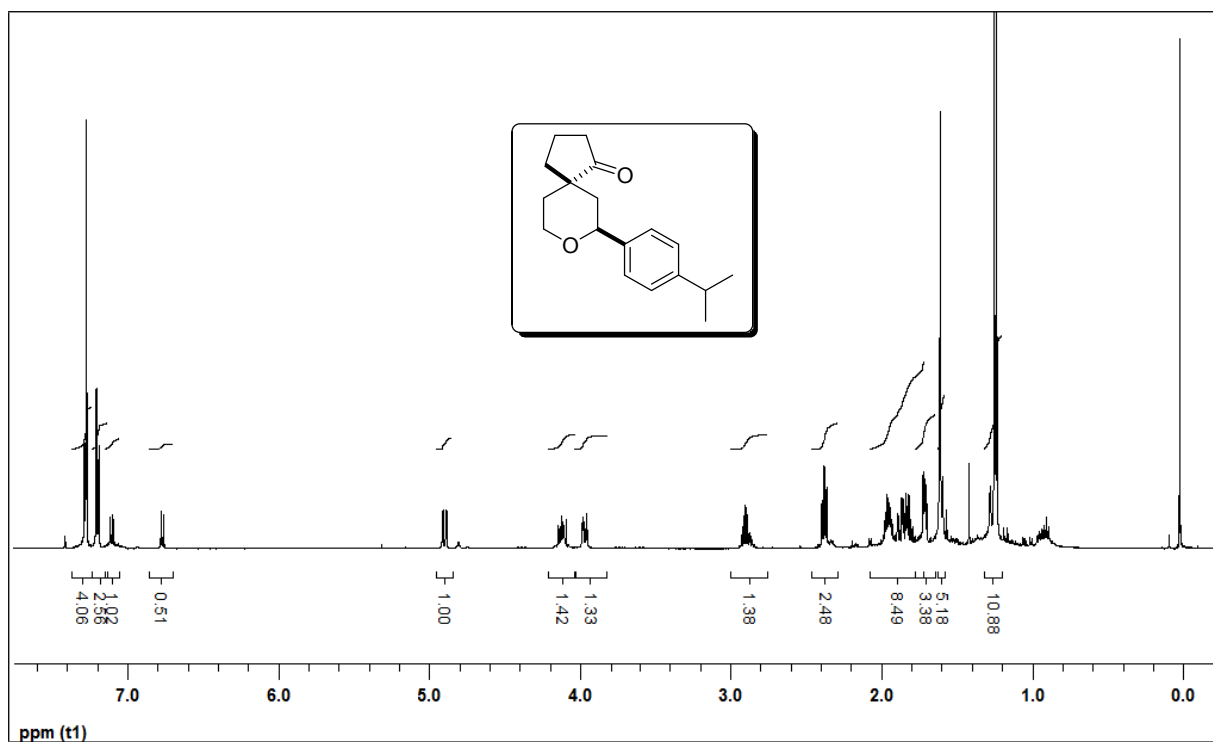
¹³C NMR (75 MHz, CDCl₃) spectrum of compound 3j



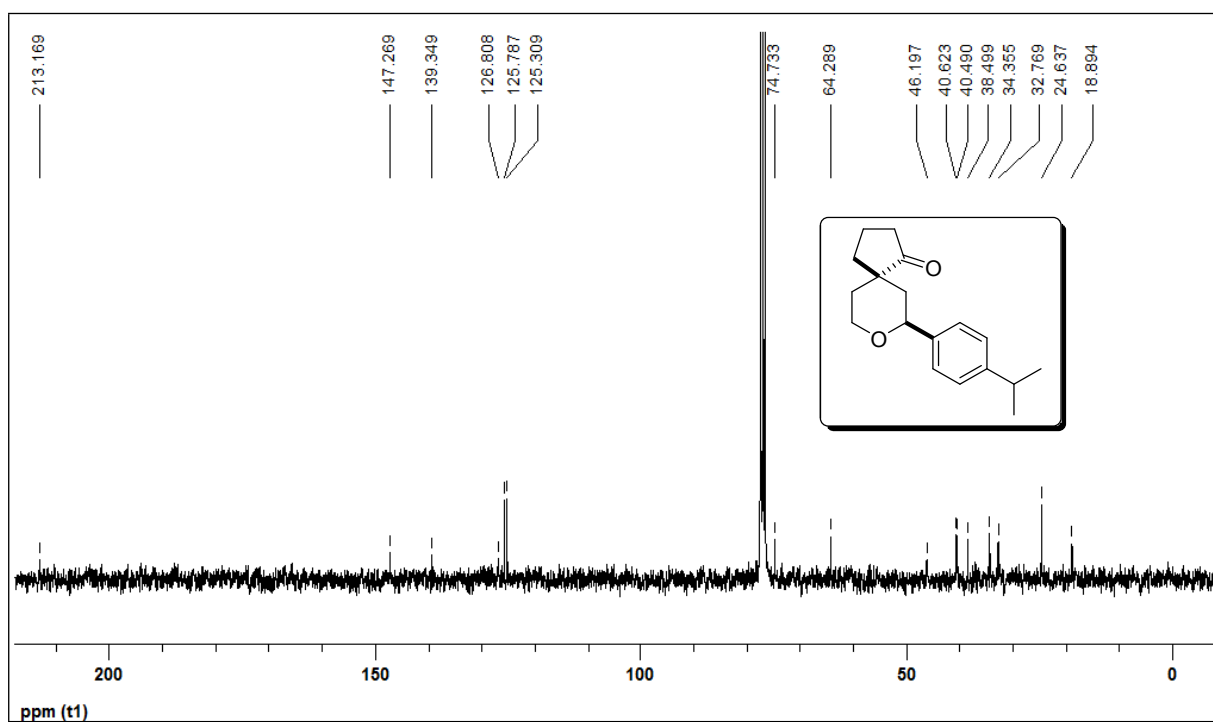
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3k



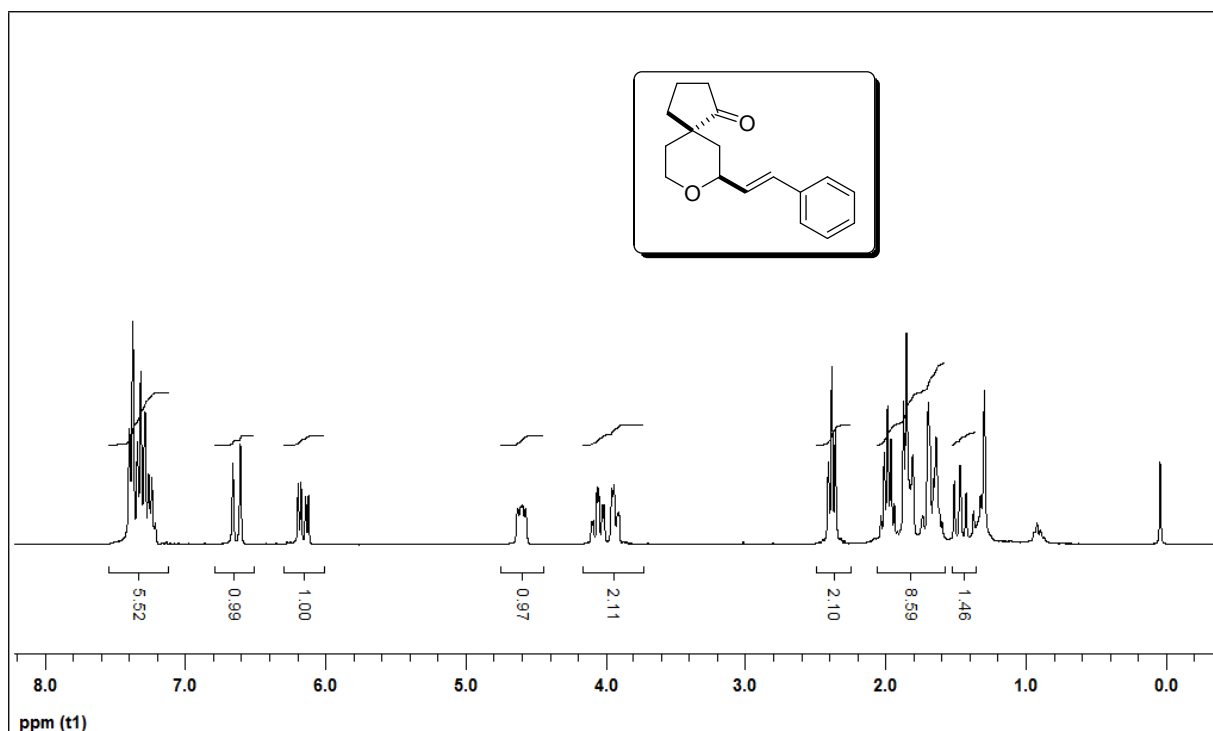
¹³C NMR (75 MHz, CDCl₃) spectrum of compound 3k



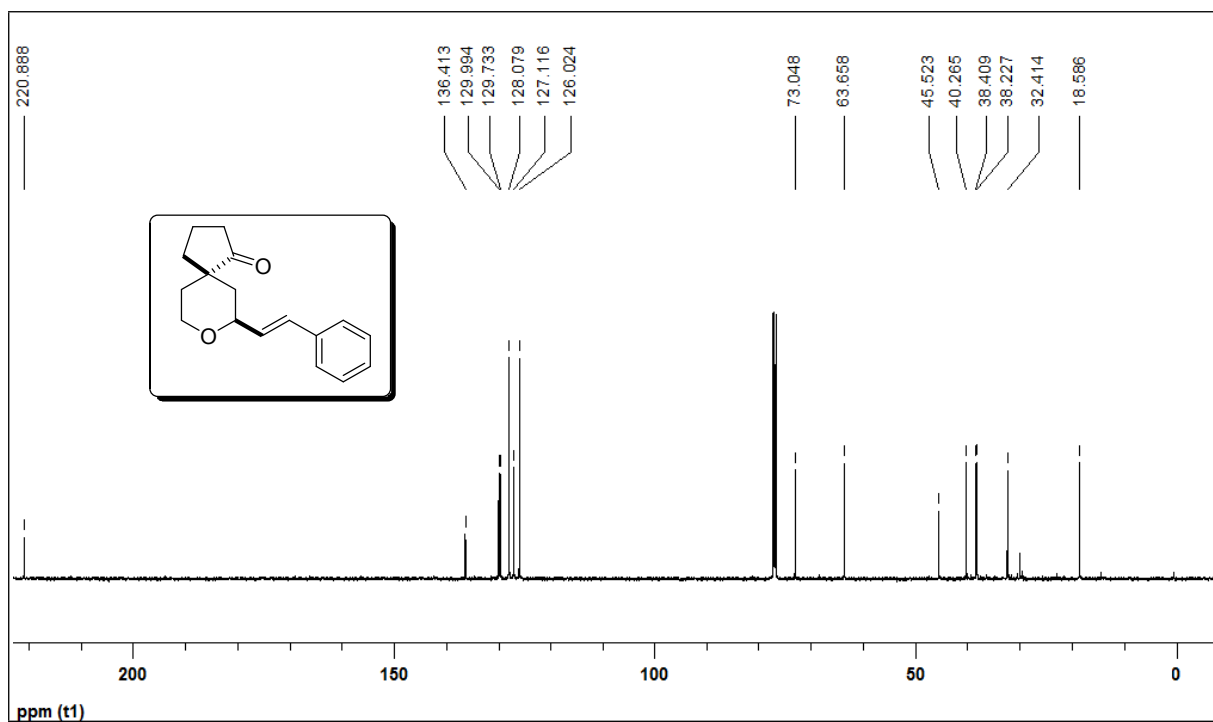
^1H NMR (500 MHz, CDCl_3) spectrum of compound 31



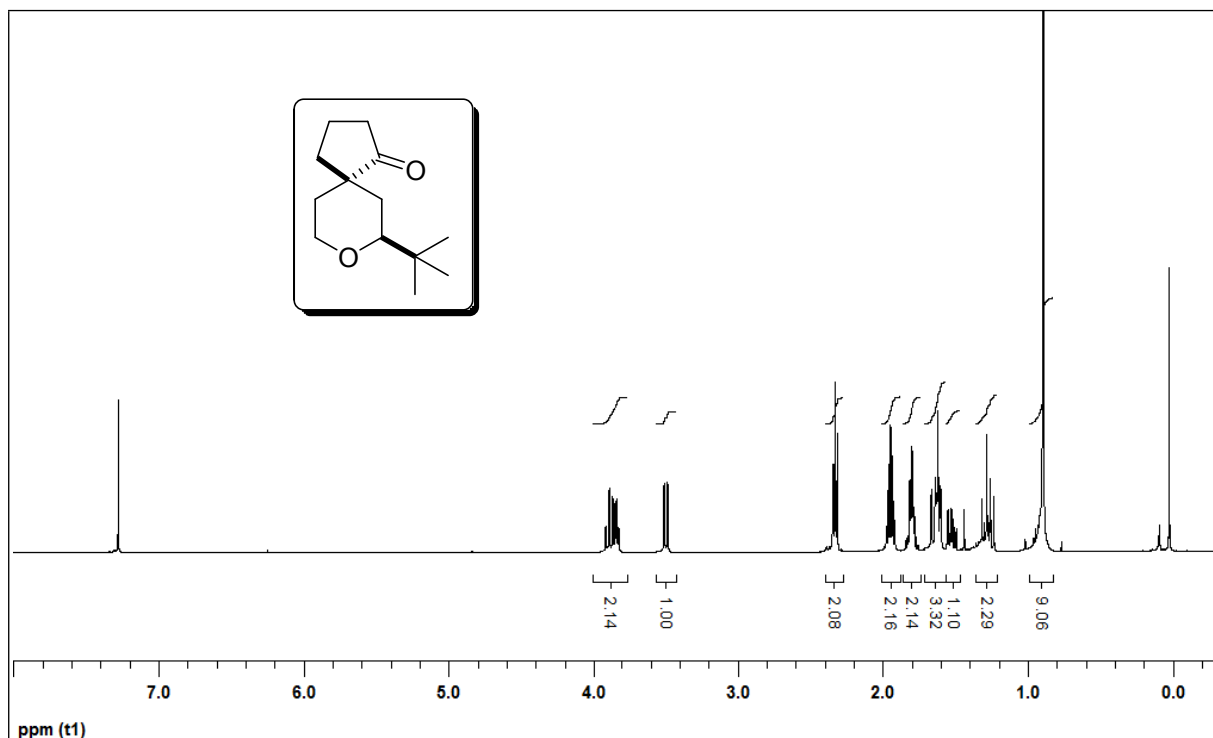
^{13}C NMR (125 MHz, CDCl_3) spectrum of compound 31



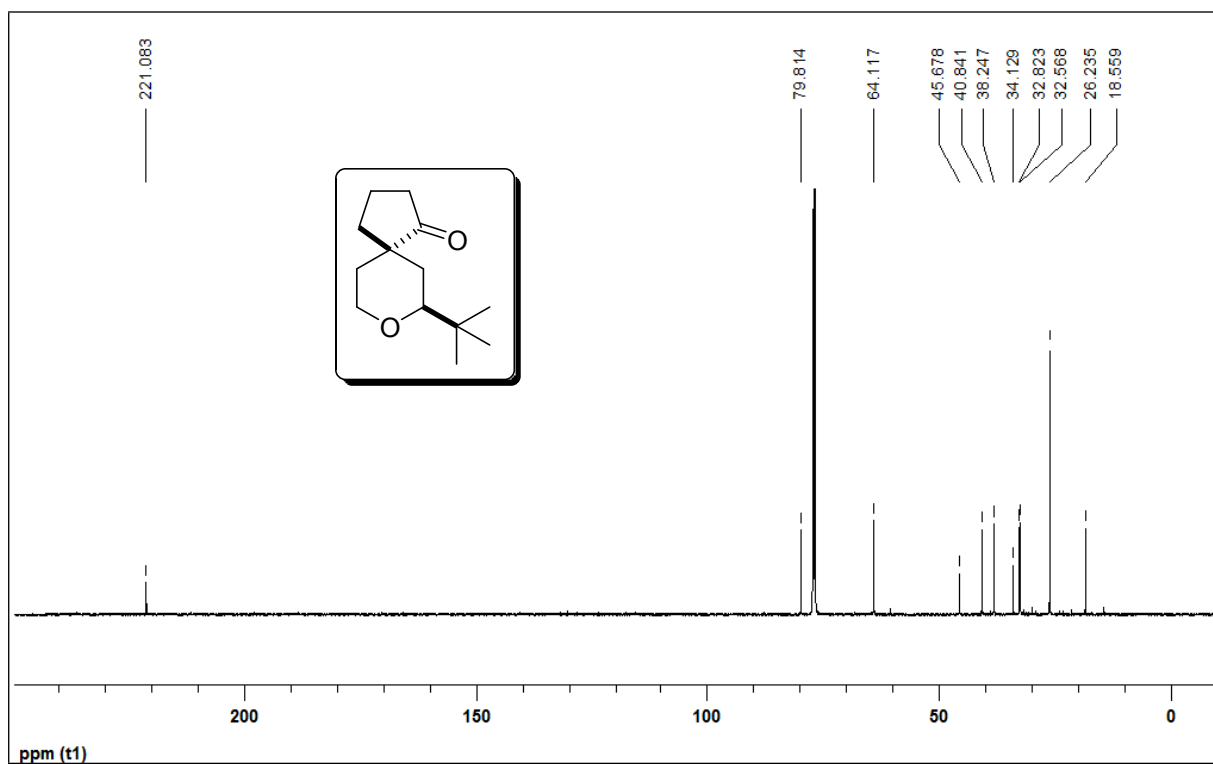
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3m



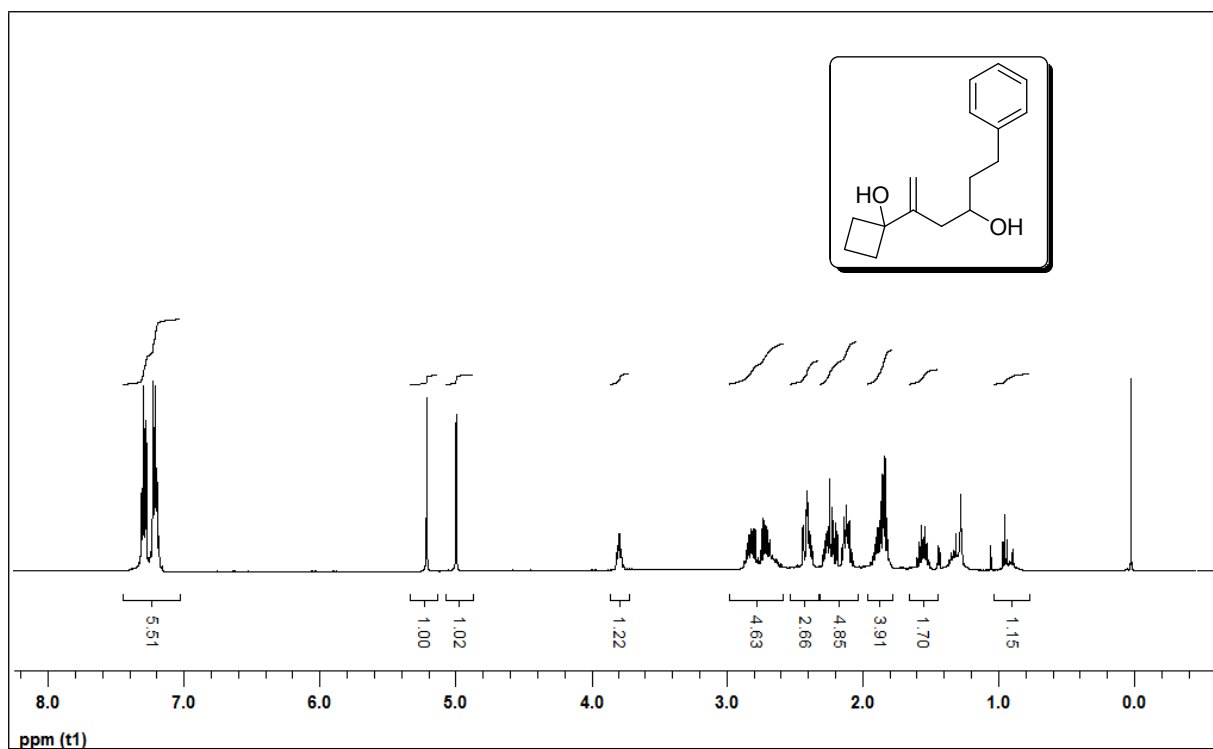
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3m



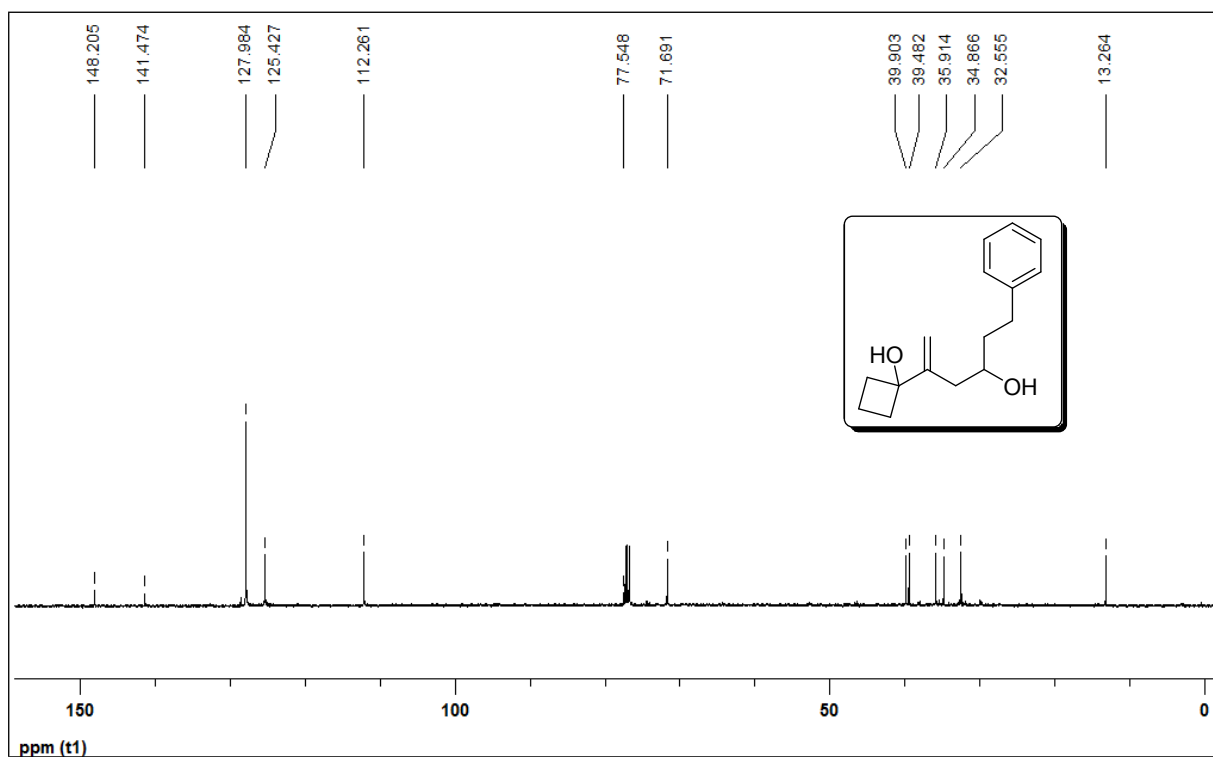
¹H NMR (500 MHz, CDCl₃) spectrum of compound 3n



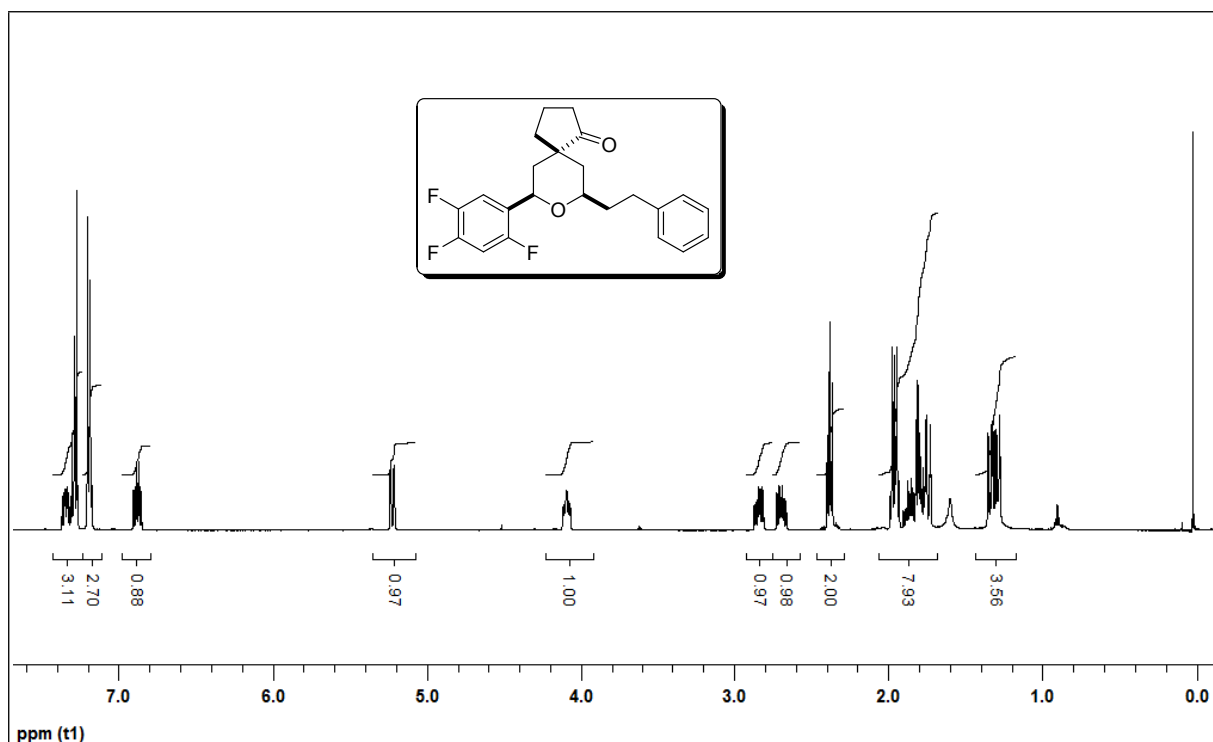
¹³C NMR (125 MHz, CDCl₃) spectrum of compound 3n



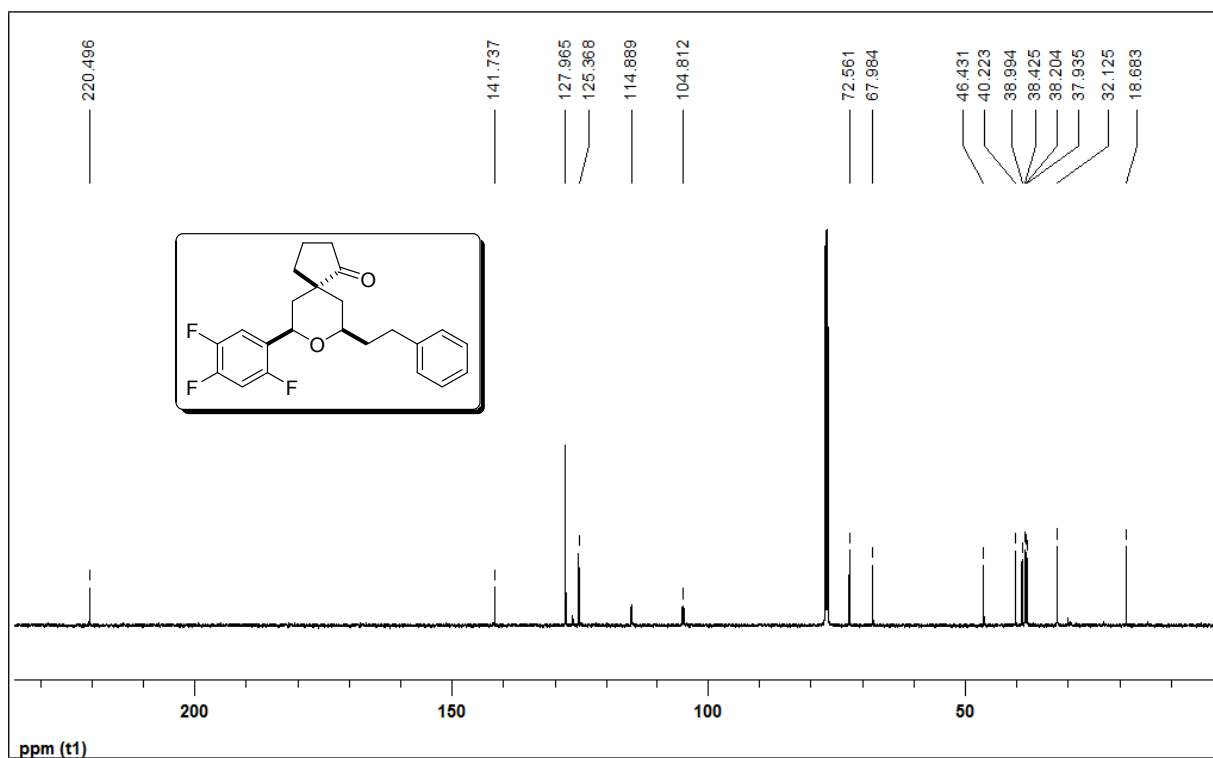
$^1\text{H NMR}$ (500 MHz, CDCl_3) spectrum of compound 2d



$^{13}\text{C NMR}$ (125 MHz, CDCl_3) spectrum of compound 2d



¹H NMR (500 MHz, CDCl₃) spectrum of compound 4



¹³C NMR (125 MHz, CDCl₃) spectrum of compound 4