Amide-assisted intramolecular [3+2] annulation of cyclopropane ring-opening: A facile and diastereoselective access to the tricyclic core of (±)-scandine

Jun-An Xiao[a], Peng-Ju Xia[a], Xing-Yu Zhang[b], Xiao-Qing Chen[a]*, Guang-Chuan Ou[b], Hua Yang[a]*

[a]*College of Chemistry and Chemical Engineering, Central South University, Changsha 410083, P. R. China*
[b]*Key Laboratory of Comprehensive Utilization of Advantage Plants Resources in Hunan South/Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, P. R. China*

E-mail:hyangchem@csu.edu.cn

Supporting information

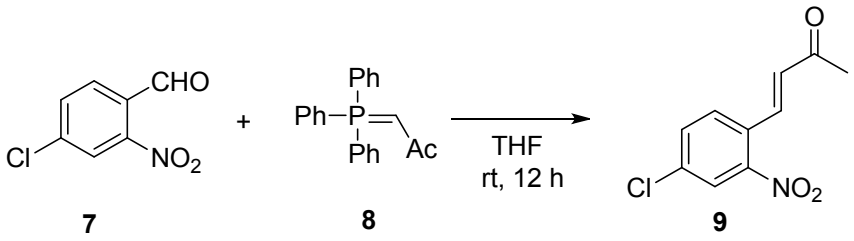
Table of Contents

1. General Information

Unless otherwise noted, all the reagents were purchased from commercial suppliers and used without further purification. ^{1}H NMR spectra were recorded at 400 MHz. The chemical shifts were recorded in ppm relative to tetramethylsilane and with the solvent resonance as the internal standard. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. ^{13}C NMR data were collected at 100 M Hz with complete proton decoupling. Chemical shifts were reported in ppm from the tetramethylsilane with the solvent resonance as internal standard. Infrared spectra (IR) were measured by FT-IR apparatus. High resolution mass spectroscopy (HRMS) was recorded on TOF MS mass spectrometer and acetonitrile was used to dissolve the sample. Column chromatography was carried out on silica gel (200-300 mesh). All solvents and commercially available reagents were either purified via literature procedures or used without further purification. *o*-Nitro-*α,β*-unsaturated enones **10**[1] and cyclopropane monoester **12**[2] were prepared according to the reported procedures respectively.

2. Experimental Procedures and characterization data of 1a-1y, 2a-2x, 3-6, 9.

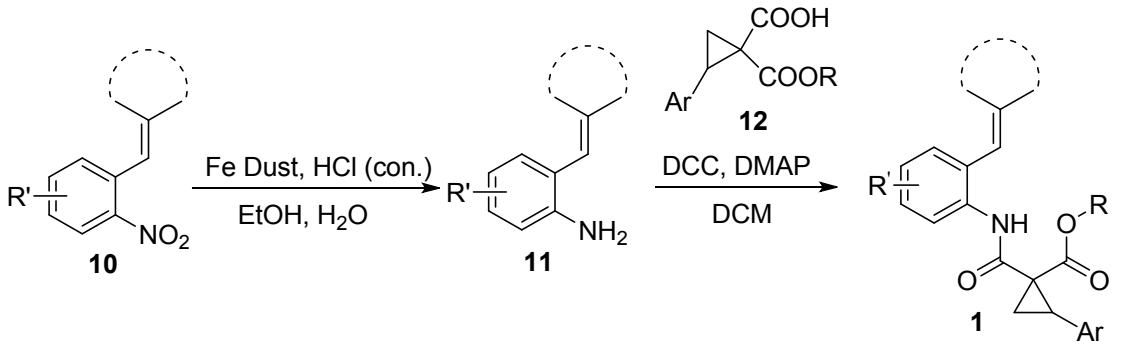
2.1 Experimental procedure for the synthesis of (*E*)-4-(4-chloro-2-nitrophenyl)but-3-en-2-one (**9**).



7 **8** **9**

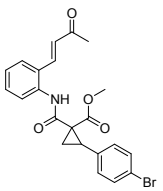
To a solution of 4-chloro-2-nitro-benzaldehyde (**7**, 3.0 g, 16.2 mmol, 1.0 equiv.) in THF (40 mL) was added ylide **8** (5.2 g, 16.2 mmol, 1.0 equiv.). The mixture was stirred at room temperature for 12 hours. Then solvent was removed under vacuum and the residual was purified through column chromatography (eluenting with EtOAc/PE = 1:5) to afford enone **9** as a white solid (3.2 g, 14.1 mmol, 87% yield, *inseparable two stereoisomers, trans/cis = 10:1*); m.p. 119-120°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07 (d, *J* = 8.8 Hz, 1H), 7.95 (d, *J* = 16.4 Hz, 1H), 7.62 (s, 1H), 7.53 (d, *J* = 8.8 Hz, 1H), 6.57 (d, *J* = 16.4 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 197.6, 146.3, 140.3, 137.7, 132.8, 130.3, 129.1, 126.6, 27.4; IR (KBr) ν 3104, 3041, 1921, 1690, 1618, 1516, 1180, 971 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{10}H_9NO_3Cl$ 226.0271, found 226.0259.

2.2 General procedure for the synthesis of **amide 1**.

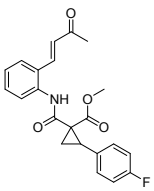


To a solution of *o*-nitro-*α,β*-unsaturated enones **10** (5.0 mmol) in anhydrous ethanol (40 mL) was added water (10 mL) and a drop of concentrated hydrochloric acid. The mixture was refluxed at 80℃ for 3 hours. Then ethanol was evaporated and 100 mL of dichloromathane was added and filtered. The residue was washed with brine three times and the auquous layer was washed with EtOAc (100 mL × 2). The combined organic layer was dried and concentrated to afford the crude *o*-amino-*α, β*-unsaturated enones **11**, which was used directely without further purification.

To a solution of *o*-amino-*α, β*-unsaturated enones **11** (1.0 equiv.) and cyclopropane monoester **12** (1.2 equiv.) in dichloromathane were added DCC (1.2 equiv.) and DMAP (0.3 equiv.) respectively. After being stirred at room temperature for 24-72 hours, the resulting mixutre was filtered, concentrated, and purified through column chromatogrophy (eluenting with EtOAc/PE = 1:9-1:5) to yield amide **1** .

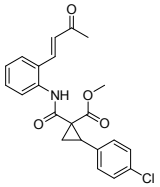


Amide 1a: White solid (3.4 g, 7.7 mmol, 71% yield); m.p. 123-124°C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.25 (s, 1H), 7.84 (d, J = 7.6 Hz, 1H), 7.76 (d, J = 16.0 Hz, 1H), 7.52-7.44 (m, 4H), 7.32-7.24 (m, 3H), 6.82 (d, J = 16.4 Hz, 1H), 3.34 (s, 3H), 3.23 (t, J = 8.6 Hz, 1H), 2.36 (s, 3H), 2.21 (t, J = 6.0 Hz, 1H), 1.83 (q, J = 4.8 Hz, 1H) ; ^{13}C NMR (DMSO-d_6, 100 MHz) δ 198.6, 168.8, 166.7, 139.1, 137.3, 135.1, 131.7, 131.3, 131.1, 129.5, 128.4, 127.2, 127.1, 126.6, 120.8, 52.7, 38.6, 32.0, 27.9, 18.8; IR (KBr) ν 3301, 3059, 1709, 1665, 1255, 1132, 971, 834 cm^{-1}; HRMS (TOF-ES+) m/z: [M+Na]$^+$ calcd for $C_{22}H_{20}NO_4BrNa$ 464.0473, found 464.0454.

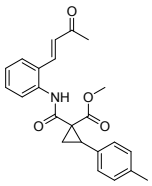


Amide 1b: White solid (2.1 g, 5.4 mmol, 62% yield); m.p. 97-98°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.84 (s, 1H), 8.06 (d, J = 8.0 Hz, 1H), 7.86 (d, J = 16.0 Hz, 1H), 7.59 (d, J = 7.6 Hz, 1H), 7.44-7.40 (m, 1H), 7.28-7.25 (m, 2H), 7.20 (t, J = 7.6 Hz, 1H), 7.01 (t, J = 8.8 Hz, 2H), 6.68 (d, J = 16.0 Hz, 1H), 3.32-3.30 (m, 1H), 3.29 (s, 3H), 2.43 (s, 3H), 2.40-2.34 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 171.8, 166.4, 162.2 (d, $^1J_{C-F}$ = 245 Hz), 138.3, 136.5, 131.0, 130.8 (d, $^3J_{C-F}$ = 8.0 Hz), 130.74, 130.71, 129.9, 127.1, 126.5, 125.3, 123.3, 52.0, 38.3, 35.7, 27.2, 27.1, 20.0; IR (KBr) ν 3276, 3012, 1708, 1666, 1513, 1254, 1139,

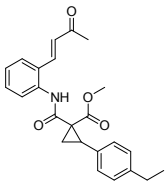
971, 844 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{22}H_{21}NO_4F$ 382.1455, found 382.1442.



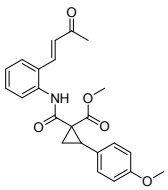
Amide 1c: White solid (2.9 g, 7.3 mmol, 62% yield); m.p. 120-121°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.83 (s, 1H), 8.06 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 16.0 Hz, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.27-7.30 (m, 2H), 7.18-7.24 (m, 3H), 6.68 (d, *J* = 16.0 Hz, 1H), 3.27-3.31 (m, 4H), 2.43 (s, 3H), 2.34-2.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 171.7, 166.3, 138.3, 136.5, 133.5, 131.1, 130.6, 129.9, 128.4, 127.1, 126.5, 125.3, 123.3, 52.1, 38.2, 35.7, 27.2, 27.1, 19.9; IR (KBr) ν 3303, 3061, 1710, 1582, 1539, 1256, 1133, 971, 856 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4ClNa$ 420.0979, found 420.0961.



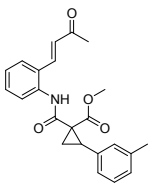
Amide 1d: White solid (2.7 g, 7.1 mmol, 67% yield); m.p. 132-133°C; ^1H NMR (DMSO-d_6, 400 MHz) δ 10.23 (s, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.77 (d, *J* = 16.4 Hz, 1H), 7.54 (d, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.11-7.18 (m, 4H), 6.81 (d, *J* = 16.4 Hz, 1H), 3.38 (s, 3H), 3.22 (t, *J* = 8.8 Hz, 1H), 2.36 (s, 3H), 2.28 (s, 3H), 2.19-2.22 (m, 1H), 1.81 (q, *J* = 4.8 Hz, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 198.5, 169.0, 167.0, 139.1, 137.4, 136.7, 132.4, 131.1, 129.4, 129.3, 129.0, 128.5, 127.2, 126.9, 126.5, 52.5, 38.5, 32.8, 27.8, 21.2, 18.7; IR (KBr) ν 3282, 3027, 1708, 1666, 1541, 1336, 1248, 998, 764 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1712.



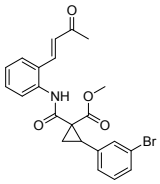
Amide 1e: White solid (1.9 g, 4.9 mmol, 70% yield); m.p. 130-131 °C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.89 (s, 1H), 8.10 (d, *J* = 7.6 Hz, 1H), 7.89 (d, *J* = 16.4 Hz, 1H), 7.60 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.42-7.46 (m, 1H), 7.20-7.23 (m, 3H), 7.14-7.16 (m, 2H), 6.69 (d, *J* = 16.0 Hz, 1H), 3.32 (t, *J* = 9.0 Hz, 1H), 3.26 (s, 3H), 2.65 (q, *J* = 7.6 Hz, 2H), 2.46 (s, 3H), 2.36-2.45 (m, 2H), 1.24 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 172.1, 166.7, 143.8, 138.4, 136.6, 132.0, 131.0, 130.0, 129.2, 127.7, 127.1, 126.5, 125.1, 123.3, 51.8, 39.2, 35.9, 28.5, 26.9, 19.7, 15.6; IR (KBr) ν 3327, 2951, 2351, 1669, 1537, 1343, 1252, 1140, 978 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{24}H_{26}NO_5$ 392.1862, found 392.1850.



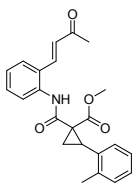
Amide 1f: White solid (2.7 g, 6.8 mmol, 53% yield); m.p. 143-144°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.87 (s, 1H), 8.09 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.88 (d, *J* = 16.0 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.40-7.45 (m, 1H), 7.18-7.22 (m, 3H), 6.83-6.87 (m, 1H), 6.68 (d, *J* = 16.0 Hz, 1H), 3.81 (s, 3H), 3.26-3.30 (m, 4H), 2.45 (s, 3H), 2.34-2.42 (m, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 198.5, 169.1, 167.0, 158.8, 139.1, 137.4, 131.0, 129.4, 128.6, 127.23, 127.18, 126.9, 126.5, 113.9, 55.5, 52.5, 38.4, 32.8, 27.8, 18.8; IR (KBr) ν 3005, 2840, 2062, 1706, 1666, 1542, 1253, 1140, 998 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_5$ 394.1654, found 394.1638.



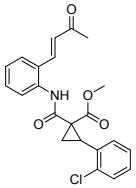
Amide 1g: White solid (1.6 g, 4.1 mmol, 53% yield); m.p. 71-72°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.89 (s, 1H), 8.08 (d, J = 8.0 Hz, 1H), 7.88 (d, J = 16.0 Hz, 1H), 7.58-7.61 (m, 1H), 7.40-7.44 (m, 1H), 7.17-7.22 (m, 2H), 7.06-7.10 (m, 3H), 6.68 (d, J = 16.0 Hz, 1H), 3.30 (t, J = 8.8 Hz, 1H), 3.24 (s, 3H), 2.44 (s, 3H), 2.40-2.43 (m, 1H), 2.36-2.37 (m, 1H), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.6, 172.1, 166.7, 138.5, 137.8, 136.6, 134.8, 131.0, 129.9(7), 129.9(4), 128.3, 128.1, 127.1, 126.5, 126.2, 125.2, 123.4, 51.8, 39.3, 35.7, 27.0, 21.3, 19.7; IR (KBr) ν 3280, 3005, 1960, 1703, 1605, 1322, 1139, 981 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1693.



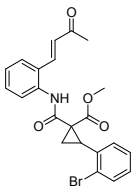
Amide 1h: White solid (2.1 g, 4.8 mmol, 61% yield); m.p. 98-99°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.84 (s, 1H), 8.07 (d, J = 8.0 Hz, 1H), 7.87 (d, J = 16.0 Hz, 1H), 7.61 (d, J = 8.0 Hz, 1H), 7.42-7.46 (m, 3H), 7.18-7.29 (m, 3H), 6.69 (d, J = 16.0 Hz, 1H), 3.34 (s, 3H), 3.30-3.33 (m, 1H), 2.46 (s, 3H), 2.35-2.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.4, 171.6, 166.2, 138.2, 137.4, 136.5, 132.2, 131.0, 130.7, 130.0, 129.7, 128.0, 127.1, 126.6, 125.3, 123.4, 122.2, 52.0, 38.1, 35.7, 27.1, 19.8; IR (KBr) ν 3286, 2958, 2348, 1693, 1591, 1542, 1329, 1145, 984 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4BrNa$ 464.0473, found 464.0479.



Amide 1i: White solid (2.0 g, 5.3 mmol, 72% yield); m.p. 92-93°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.93 (s, 1H), 8.02 (d, J = 8.4 Hz, 1H), 7.85 (d, J = 16.0 Hz, 1H), 7.60 (d, J = 7.6 Hz, 1H), 7.43 (t, J = 7.6 Hz, 1H), 7.16-7.26 (m, 5H), 6.68 (d, J = 16.0 Hz, 1H), 3.24 (t, J = 8.8 Hz, 1H), 3.18 (s, 3H), 2.48-2.51 (m, 1H), 2.39-2.41 (m, 4H), 2.33 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 172.3, 166.9, 138.3, 136.6, 133.3, 131.0, 129.9, 129.8, 128.8, 127.7, 127.0, 126.8, 125.5, 125.3, 123.7, 51.9, 38.7, 35.4, 27.0, 19.9, 19.4; IR (KBr) ν 3266, 3019, 1952, 1702, 1666, 1542, 1337, 1252, 1147, 976 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1710.

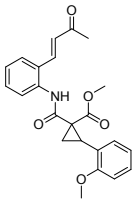


Amide 1j: colorless oil (3.7 g, 9.2 mmol, 77% yield); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.96 (s, 1H), 7.99 (d, J = 8.4 Hz, 1H), 7.86 (d, J = 16.0 Hz, 1H), 7.59 (dd, J = 8.0, 1.2 Hz, 1H), 7.39-7.43 (m, 1H), 7.35-7.37 (m, 1H), 7.29-7.32 (m, 1H), 7.18-7.27 (m, 3H), 6.66 (d, J = 16.4 Hz, 1H), 3.30 (t, J = 8.8 Hz, 1H), 3.23 (s, 3H), 2.39-2.45 (m, 5H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 198.6, 171.6, 166.6, 138.5, 136.6, 136.0, 133.5, 130.9, 130.6, 129.9, 129.1, 128.9, 127.04, 127.00, 126.4, 125.4, 123.9, 51.9, 37.8, 35.1, 26.9, 20.1; IR (KBr) ν 3294, 3006, 1938, 1706, 1667, 1438, 1332, 1137, 977 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{22}H_{21}NO_4Cl$ 398.1159, found 398.1135.

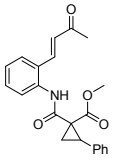


Amide 1k: White solid (1.5 g, 3.4 mmol, 68% yield); m.p. 107-108°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 11.01 (s, 1H), 7.99 (d, J = 8.0 Hz, 1H), 7.89 (d, J = 16.0 Hz, 1H), 7.61 (d, J = 8.0 Hz, 1H), 7.55 (d, J = 8.0 Hz, 1H), 7.40-7.45 (m, 1H), 7.30-7.31 (m, 2H), 7.14-7.23 (m, 2H), 6. 68 (d, J = 16.4 Hz, 1H), 3.30 (t, J = 8.8 Hz, 1H), 3.23 (s, 3H), 2.40-2.47 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.7, 171.6, 166.6, 138.6, 136.6, 135.3, 132.3, 131.0, 130.8,

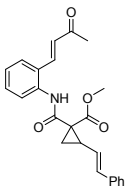
129.9, 129.2, 127.04, 127.00, 126.7, 126.5, 125.4, 124.0, 51.9, 40.3, 35.3, 27.0, 20.7; IR (KBr) ν 3309, 3058, 1687, 1610, 1509, 1328, 1140, 993, 748 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{22}H_{21}NO_4Br$ 442.0654, found 442.0663.



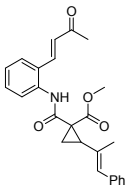
Amide 1l: White solid (3.9 g, 9.8 mmol, 74% yield); m.p. 115-116°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.96 (s, 1H), 8.06 (d, *J* = 7.6 Hz, 1H), 7.94 (d, *J* = 16.0 Hz, 1H), 7.63 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.43-7.47 (m, 1H), 7.20-7.29 (m, 3H), 6.92-6.96 (m, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.71 (d, *J* = 16.0 Hz, 1H), 3.80 (s, 3H), 3.18-3.22 (m, 4H), 2.44 (s, 3H), 2.33-2.42 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 172.1, 167.2, 158.8, 138.6, 136.9, 131.0, 129.8, 129.7, 128.8, 127.0, 126.8, 125.1, 124.0, 123.7, 120.0, 109.9, 55.5, 51.6, 35.5, 35.0, 27.0, 19.6; IR (KBr) ν 3433, 2953, 2353, 1701, 1662, 1538, 1330, 1141, 763 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+ Na]^+$ calcd for $C_{23}H_{23}NO_5Na$ 416.1474, found 416.1475.



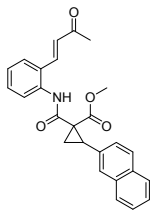
Amide 1m: White solid (3.0 g, 8.4 mmol, 77% yield); m.p. 90-91°C; ^1H NMR (DMSO-d_6, 400 MHz) δ 10.23 (s, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 16.4 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.32-7.26 (m, 6H), 6.80 (d, *J* = 16.0 Hz, 1H), 3.27 (t, *J* = 8.6 Hz, 1H), 2.36 (s, 3H), 2.24 (t, *J* = 7.6 Hz, 1H), 1.82-1.85 (m, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 198.5, 169.0, 166.9, 139.1, 137.3, 135.5, 131.1, 129.4, 128.5, 128.4, 127.6, 127.2, 127.0, 126.6, 52.5, 38.5, 33.0, 27.8, 18.7; IR (KBr) ν 3239, 3035, 1953, 1726, 1650, 1533, 1275, 974, 749, 695 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{22}H_{22}NO_4$ 364.1549, found 364.1533.



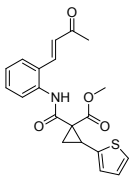
Amide 1n: White solid (2.36 g, 6.0 mmol, 44% yield); m.p. 154-155°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.83 (s, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 7.84 (d, *J* = 16.0 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.16-7.33 (m, 6H), 6.73 (d, *J* = 15.6 Hz, 1H), 6.66 (d, *J* = 16.0 Hz, 1H), 6.09 (dd, *J* = 16.0, 8.0 Hz, 1H), 3.83 (s, 3H), 3.87 (q, *J* = 8.4 Hz, 1H), 2.44 (s, 3H), 2.33 (q, *J* = 4.6 Hz, 1H), 2.15 (q, *J* = 4.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 172.5, 166.3, 138.4, 136.6, 135.5, 131.0, 129.9, 128.7, 127.9, 127.0, 126.5, 126.1, 125.1, 124.3, 123.3, 52.6, 39.0, 35.5, 27.1, 22.8; IR (KBr) ν 3226, 3049, 2353, 1665, 1509, 1543, 1451, 1256, 1147, 976 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{24}H_{23}NO_4Na$ 412.1525, found 412.1505.



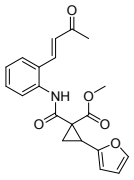
Amide 1o: colorless oil (0.58 g, 1.4 mmol, 76% yield); ^1H NMR ($CDCl_3$, 400 MHz) δ 10.84 (s, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 16.0 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.32-7.36 (m, 2H), 7.16-7.29 (m, 5H), 6.66 (d, *J* = 16.0 Hz, 1H), 6. 48 (s, 1H), 3.69 (s, 3H), 2.84 (t, *J* = 8.4 Hz, 1H), 2.43 (s, 3H), 2.28-2.31 (m, 1H), 2.19-2.22 (m, 1H), 1.92 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 172.6, 166.8, 138.4, 137.2, 136.6, 131.5, 131.0, 129.9, 128.7, 128.3, 127.1, 126.8, 126.6, 125.2, 123.5, 52.5, 43.7, 34.9, 26.9, 20.2, 18.6; IR (KBr) ν 2952, 1736, 1703, 1668, 1528, 1436, 1252, 1144, 982 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+ Na]^+$ calcd for $C_{25}H_{25}NO_4Na$ 426.1681, found 426.1672.



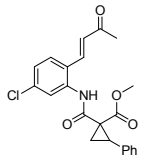
Amide 1p: White solid (1.4 g, 2.1 mmol, 62% yield); m.p. 231-232°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.93 (s, 1H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.92 (d, *J* = 16.0 Hz, 1H), 7.85-7.78 (m, 4H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.40-7.53 (m, 4H), 7.22 (t, *J* = 7.2 Hz, 1H), 6.71 (d, *J* = 16.4 Hz, 1H), 3.52 (t, *J* = 8.8 Hz, 1H), 3.16 (s, 3H), 2.57-2.60 (m, 1H), 2.47-2.50 (m, 1H), 2.46 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.5, 171.9, 166.6, 138.4, 136.6, 133.1, 132.7, 132.4, 131.1, 131.0, 130.0, 128.2, 127.9, 127.72, 127.69, 127.1, 126.54, 126.51, 126.4, 126.2, 125.2, 123.4, 51.9, 39.4, 36.0, 27.0, 20.0; IR (KBr) ν 3329, 2928, 2854, 1626, 1575, 1312, 1240, 1092, 895 cm^{-1}; HRMS (TOF-ES+) m/z: [M+ Na]$^+$ calcd for $C_{26}H_{23}NO_4Na$ 436.1525, found 436.1520.



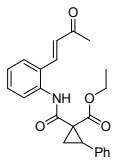
Amide 1q: White solid (2.6 g, 7.1 mmol, 78% yield); m.p. 109-110°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.80 (s, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 16.0 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.40-7.44 (m, 1H), 7.18-7.21 (m, 2H), 6.93-6.97 (m, 1H), 6.67 (d, *J* = 16.0 Hz, 1H), 3.41 (s, 3H), 3.33 (t, *J* = 8.4 Hz, 1H), 2.46 (s, 3H), 2.39-2.44 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.6, 171.5, 165.9, 138.4, 138.3, 136.4, 131.0, 130.0, 127.5, 127.1, 126.7, 126.5, 125.4, 125.3, 123.4, 52.3, 36.6, 33.3, 27.0, 21.4; IR (KBr) ν 3272, 3009, 1707, 1664, 1584, 1335, 1254, 1141, 974 cm^{-1}; HRMS (TOF-ES+) m/z: [M+Na]$^+$ calcd for $C_{20}H_{19}NO_4SNa$ 392.0932, found 392.0918.



Amide 1r: White solid (4.0 g, 11.3 mmol, 75% yield); m.p. 102-103°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.81 (s, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 16.4 Hz, 1H), 7.58 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.39-7.43 (m, 1H), 7.33 (d, *J* = 1.2 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 16.0 Hz, 1H), 6.23-6.34 (m, 2H), 3.49 (s, 3H), 3.17 (t, *J* = 8.4 Hz, 1H), 2.44 (s, 3H), 2.35-2.38 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.6, 171.6, 165.8, 149.7, 142.3, 138.4, 136.4, 131.0, 127.1, 126.6, 125.3, 123.4, 110.5, 109.0, 52.6, 35.0, 31.2, 27.0, 19.8; IR (KBr) ν 3131, 2952, 1781, 1709, 1668, 1543, 1258, 1143, 975 cm^{-1}; HRMS (TOF-ES+) m/z: [M+ Na]$^+$ calcd for $C_{20}H_{19}NO_5Na$ 376.1161, found 376.1156.

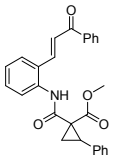


Amide 1s: White solid (1.3 g, 3.1 mmol, 83% yield); m.p. 105-106 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.95 (s, 1H), 8.11 (d, *J* = 8.8 Hz, 1H), 7.80 (d, *J* = 16.4 Hz, 1H), 7.56 (d, *J* = 2.4 Hz, 1H), 7.39 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.29-7.35 (m, 5H), 6.69 (d, *J* = 16.0 Hz, 1H), 3.36 (t, *J* = 8.8 Hz, 1H), 3.25 (s, 3H), 2.47 (s, 3H), 2.43-2.46 (m, 1H), 2.34-2.40 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.0, 172.0, 166.7, 136.8, 135.2, 134.8, 130.8, 130.7, 130.3, 129.2, 128.2, 127.9, 127.7, 126.8, 124.4, 51.9, 39.5, 35.7, 27.3, 19.9; IR (KBr) ν 3431, 3295, 2354, 1672, 1528, 1342, 1256, 1147, 981 cm^{-1}; HRMS (TOF-ES+) m/z: [M+Na]$^+$ calcd for $C_{22}H_{20}NO_4ClNa$ 420.0979, found 420.0978.

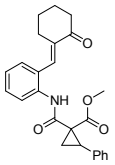


Amide 1t: White solid (1.93 g, 9.3 mmol, 82% yield); m.p. 90-92°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 11.03 (s, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.88 (d, *J* = 16.0 Hz, 1H), 7.59 (d, *J* = 7.6 Hz, 1H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.26-7.31 (m, 5H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.67 (d, *J* = 16.0 Hz, 1H), 3.77-3.85 (m, 1H), 3.60-3.68 (m, 1H), 3.36 (t, *J* = 8.8 Hz, 1H), 2.44 (s, 3H), 2.39-2.42 (m, 1H), 2.33-2.36 (m, 1H), 0.72 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.6, 171.8,

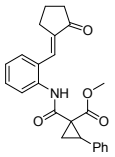
166.9, 138.5, 136.7, 135.2, 131.0, 130.0, 129.5, 128.2, 127.6, 127.1, 126.5, 125.1, 123.3, 61.6, 39.2, 35.1, 26.9, 20.0, 13.2; IR (KBr) ν 3319, 2930, 1667, 1589, 1544, 1325, 1256, 1147, 990 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1705.



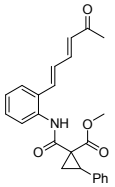
Amide 1u: White solid (1.2 g, 2.9 mmol, 62% yield); m.p. 137-138°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.79 (s, 1H), 7.99-8.11 (m, 4H), 7.72-7.74 (m, 1H), 7.14-7.58 (m, 5H), 7.21-7.31 (m, 6H), 3.33 (t, *J* = 8.8 Hz, 1H), 3.22 (s, 3H), 2.32-2.41 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 190.5, 171.5, 167.0, 139.6, 138.1, 136.9, 135.0, 132.8, 131.0, 129.3, 128.7, 128.6, 128.2, 127.6, 127.51, 127.47, 125.4, 124.7, 124.2, 51.9, 39.1, 35.7, 19.9; IR (KBr) ν 3433, 3324, 2930, 2353, 1668, 1590, 1543, 1254, 1146, 991 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{27}H_{24}NO_4$ 426.1705, found 426.1686.



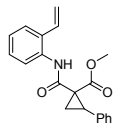
Amide 1v: White solid (1.1 g, 2.7 mmol, 61% yield); m.p. 128-129°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.48 (s, 1H), 8.25 (d, *J* = 8.4 Hz, 1H), 7.45 (s, 1H), 7.32-7.37 (m, 1H), 7.23-7.30 (m, 5H), 7.19-7.22 (m, 1H), 7.11-7.15 (m, 1H), 3.29 (t, *J* = 8.8 Hz, 1H), 3.18 (s, 3H), 2.59-2.64 (m, 2H), 2.54-2.58 (m, 2H), 2.29-2.38 (m, 2H), 1.90-1.96 (m, 2H), 1.71-1.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 201.5, 171.1, 166.4, 140.7, 136.6, 135.1, 130.3, 129.2, 129.1, 128.1, 127.5, 126.7, 123.9, 121.7, 51.8, 40.6, 38.8, 35.9, 29.0, 24.0, 23.8, 19.6; IR (KBr) ν 3270, 3224, 2940, 2352, 1696, 1539, 1448, 1329, 1142, 754 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{25}H_{26}NO_4$ 404.1862, found 404.1854.



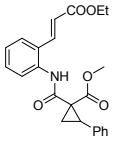
Amide 1w: White solid (2.2 g, 5.6 mmol, 83% yield); m.p. 98-99°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.61 (s, 1H), 8.10 (d, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 2.4 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.36-7.41 (m, 1H), 7.23-7.32 (m, 5H), 7.19 (t, *J* = 7.2 Hz, 1H), 3.32 (t, *J* = 8.8 Hz, 1H), 3.24 (s, 3H), 2.85-2.91 (m, 2H), 2.43 (t, *J* = 7.6 Hz, 2H), 2.31-2.40 (m, 2H), 2.00-2.05 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 207.3, 171.2, 166.7, 138.9, 137.3, 135.0, 129.8, 129.3, 129.1, 128.1, 127.5, 127.4, 126.6, 124.5, 123.2, 51.9, 39.0, 38.1, 35.9, 29.3, 20.3, 19.7; IR (KBr) ν 3195, 3063, 2353, 1728, 1674, 1494, 1240, 867, 760 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{24}H_{24}NO_4$ 390.1705, found 390.1724.



Amide 1x: White solid (1.6 g, 8.2 mmol, 61% yield); m.p. 124-125°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 10.73 (s, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 7.2 Hz, 1H), 7.21-7.39 (m, 8H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.84-6.91 (m, 1H), 6.29 (d, *J* = 15.2 Hz, 1H), 3.33 (t, *J* = 8.8 Hz, 1H), 3.22 (s, 3H), 2.33-2.43 (m, 2H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 198.3, 171.9, 166.6, 143.18, 143.15, 135.69, 135.66, 135.1, 130.8, 129.7, 129.4, 129.2, 128.2, 127.6, 126.6, 125.2, 123.7, 51.8, 39.1, 35.6, 27.8, 19.8; IR (KBr) ν 3438, 2959, 2353, 1694, 1542, 1139, 1090, 797 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{24}H_{23}NO_4Na$ 412.1525, found 412.1532.

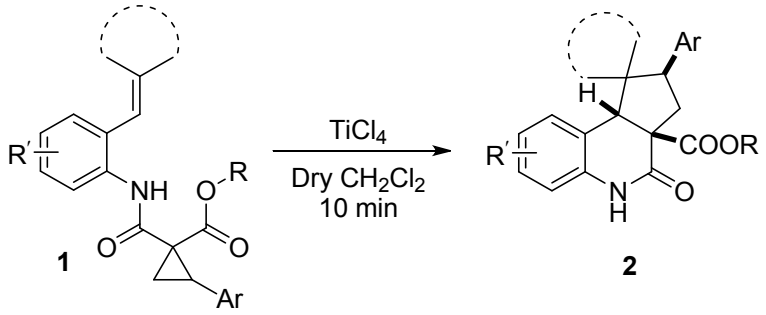


Amide 1y: White solid (1.3 g, 4.1 mmol, 63% yield); m.p. 78-79°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.61 (s, 1H), 8.05 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.49 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.24-7.32 (m, 6H), 7.12-7.16 (m, 1H), 6.97-7.04 (m, 1H), 5.73 (dd, *J* = 17.2, 1.2 Hz, 1H), 5.46 (dd, *J* = 10.8, 1.2 Hz, 1H), 3.31 (t, *J* = 8.8 Hz, 1H), 3.21 (s, 3H), 2.33-2.40 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 171.7, 166.4, 135.2, 135.0, 132.0, 129.9, 129.2, 128.4, 128.1, 127.5, 126.6, 124.9, 122.8, 117.8, 51.7, 38.8, 35.7, 19.5; IR (KBr) ν 3429, 3306, 3043, 2354, 1706, 1542, 1448, 1335, 1140, 923, 759 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{20}H_{19}NO_3Na$ 344.1263, found 344.1274.

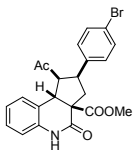


Amide 5: White solid (1.0 g, 2.6 mmol, 57% yield); m.p. 74-75°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.75 (s, 1H), 7.97-8.02 (m, 2H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 7.2 Hz, 1H), 7.23-7.30 (m, 5H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.44 (d, *J* = 15.6 Hz, 1H), 4.26 (q, *J* = 7.2 Hz, 2H), 3.33 (t, *J* = 9.2 Hz, 1H), 3.24 (s, 3H), 2.33-2.42 (m, 2H), 1.31 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 171.5, 166.8, 166.6, 139.2, 136.4, 135.1, 130.7, 129.3, 128.1, 127.5, 127.1, 127.0, 125.4, 124.0, 120.7, 60.5, 51.9, 39.1, 35.7, 19.7, 14.3; IR (KBr) ν 3315, 2986, 2351, 1707, 1542, 1450, 1273, 1146, 980 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_5$ 394.1654, found 394.1663.

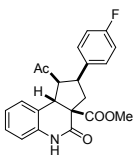
2.3 General procedure for the synthesis of cycloadduct **2**.



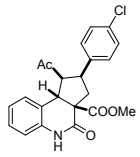
To a solution of *α,β*-unsaturated enone **1** (0.2 mmol) in anhydrous CH_2Cl_2 (2.0 mL) was added titanium tetrachloride (45.5 mg, 26.3 μL, 0.24 mmol) at room temperature. The solution was stirred for 10 mimutes, and brine (10 mL) was then added to it. The resulting mixture was stirred for another 10 minutes. The aqueous phase was extracted with EtOAc(3 mL × 2). The combined organic layer was dried, concentrated, and purified *via* column chromatography (eluenting with with EtOAc/PE = 1:3-1:2) to afford **tricycle 2**.



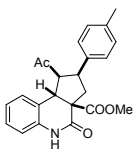
Tricycle 2a: White solid (60.9 mg, 0.14 mmol, 69% yield); m.p. 208-209°C; ^{1}H NMR (DMSO-*d*$_6$, 400 MHz) δ 10.70 (s, 1H), 7.52 (d, *J* = 8.4 Hz, 2H), 7.20 (d, *J* = 8.8 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 7.2 Hz, 1H), 6.89-6.95 (m, 2H), 4.19 (d, *J* = 10.8 Hz, 1H), 3.80 (q, *J* = 10.0 Hz, 1H), 3.58 (s, 3H), 3.37-3.43 (m, 1H), 3.08-3.14 (m, 1H), 2.32-2.38 (m, 1H), 1.52 (s, 3H); ^{13}C NMR (DMSO-*d*$_6$, 100 MHz) δ 206.7, 171.5, 166.9, 141.3, 137.0, 131.7, 131.2, 129.0, 128.4, 123.1, 122.9, 120.6, 115.9, 62.3, 57.8, 53.4, 47.9, 44.1, 41.3, 31.5; IR (KBr) ν 3208, 2921, 1725, 1666, 1491, 1388, 1241, 829, 757 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4BrNa$ 464.0473, found 464.0475.



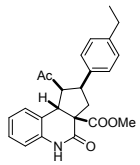
Tricycle 2b: White solid (58.7 mg, 0.15 mmol, 77% yield); m.p. 212-213°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.91 (s, 1H), 7.18-7.24 (m, 4H), 6.96-7.02 (m, 3H), 6.86 (d, J = 8.0 Hz, 1H), 4.49 (d, J = 10.4 Hz, 1H), 3.75-3.83 (m, 1H), 3.65 (s, 1H), 3.29-3.35 (m, 2H), 2.52-2.58 (m, 1H), 1.52 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.1, 170.9, 168.5, 161.9 (d, $^1J_{C\text{-}F}$ = 245 Hz), 136.49, 136.46, 135.4, 130.1 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 129.3, 128.3, 123.8, 123.3, 115.6 (d, $^2J_{C\text{-}F}$ = 21 Hz), 62.7, 58.2, 53.2, 48.0, 44.8, 42.0, 31.3; IR (KBr) ν 3067, 2928, 1739, 1678, 1510, 1391, 1232, 1163, 843, 762 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4FNa$ 404.1274, found 404.1265.



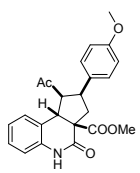
Tricycle 2c: White solid (66.7 mg, 0.17 mmol, 84% yield); m.p. 130-131°C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.71 (s, 1H), 7.38 (d, J = 8.4 Hz, 2H), 7.27 (d, J = 8.4 Hz, 2H), 7.15-7.19 (m, 1H), 7.08 (d, J = 7.2 Hz, 1H), 6.86-6.95 (m, 2H), 4.20 (d, J = 10.8 Hz, 1H), 3.81 (q, J = 10.0 Hz, 1H), 3.58 (s, 3H), 3.38-3.43 (m, 1H), 3.08-3.14 (m, 1H), 2.33-2.38 (m, 1H), 1.51 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 206.7, 171.5, 166.9, 140.9, 137.0, 132.0, 130.8, 129.0, 128.8, 128.4, 123.1, 122.9, 115.9, 62.3, 57.8, 53.4, 47.9, 44.1, 41.3, 31.5; IR (KBr) ν 3201, 2922, 1728, 1668, 1493, 1239, 1094, 832, 756 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4ClNa$ 420.0979, found 420.0973.



Tricycle 2d: White solid (57.3 mg, 0.15 mmol, 76% yield); m.p. 174-175°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.78 (s, 1H), 7.26-7.28 (m, 1H), 7,19 (t, J = 7.6 Hz, 1H), 7.09-7.11 (m, 4H), 6.98 (t, J = 7.4 Hz, 1H), 6.85 (d, J = 7.6 Hz, 1H), 4.51 (d, J = 19.8 Hz, 1H), 3.73-3.80 (m, 1H), 3.65 (s, 3H), 3.27-3.33 (m, 2H), 2.54-2.60 (m, 1H), 2.31 (s, 3H), 1.49 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.2, 171.0, 168.5, 137.7, 137.0, 135.4, 129.42, 129.36, 128.5, 128.2, 123.7, 123.6, 115.5, 62.8, 58.2, 53.1, 47.9, 45.2, 41.9, 31.2, 21.0; IR (KBr) ν 3057, 2919, 1729, 1675, 1494, 1389, 1234, 814, 762 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{23}H_{23}NO_4Na$ 400.1525, found 400.1511.

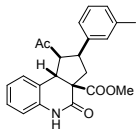


Tricycle 2e: White solid (63.3 mg, 0.16 mmol, 81% yield); m.p. 193-194°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.36 (s, 1H), 7.27 (d, J = 6.4 Hz, 1H), 7.17-7.21 (m, 1H), 7.11-7.14 (m, 4H), 6.97 (t, J = 7.4 Hz, 1H), 6.90 (d, J = 8.0 Hz, 1H), 4.52 (d, J = 10.4 Hz, 1H), 3.75-3.82 (m, 1H), 3.65 (s, 3H), 3.29-3.35 (m, 2H), 2.56-2.64 (m, 3H), 1.48 (s, 3H), 1.21 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.4, 171.1, 168.9, 143.3, 137.9, 135.5, 129.3, 128.5, 128.2, 123.7, 123.5, 115.7, 62.9, 58.2, 53.1, 47.9, 45.2, 41.9, 31.2, 28.4, 15.5; IR (KBr) ν 3204, 3069, 2963, 2353, 1687, 1496, 1382, 1234, 836, 759 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{24}H_{26}NO_4$ 392.1862, found 392.1880.

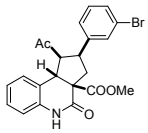


Tricycle 2f: White solid (43.2 mg, 0.11 mmol, 55% yield); m.p. 119-120°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.65 (s, 1H), 7.25-7.27 (m, 1H), 7.12-7.21 (m, 2H), 6.96-6.99 (m, 1H), 6.83-6.85 (m, 3H), 4.50 (d, J = 10.8 Hz, 1H), 3.79 (s, 3H), 3.72-3.77 (m, 1H), 3.65 (s, 3H), 3.26-3.32 (m, 1H), 2.51-2.57 (m, 1H), 1.51 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.4, 171.0, 168.5,

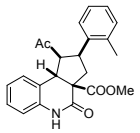
158.7, 135.4, 132.7, 129.6, 129.4, 128.2, 123.7, 123.6, 115.5, 114.0, 62.7, 58.2, 55.3, 53.1, 47.8, 44.8, 42.0, 31.3; IR (KBr) ν 3199, 2935, 2353, 1683, 1508, 1244, 1039, 837, 761 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_5$ 394.1654, found 394.1664.



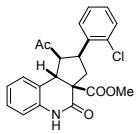
Tricycle 2g: White solid (53.5 mg, 0.14 mmol, 71% yield); m.p. 113-114°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.54 (s, 1H), 7.29-7.27 (m, 1H), 7.19 (t, *J* = 7.8 Hz, 2H), 6.97-7.06 (m, 4H), 6.83 (d, *J* = 7.6 Hz, 1H), 4.52 (d, *J* = 10.4 Hz, 1H), 3.71-3.79 (m, 1H), 3.66 (s, 3H), 3.27-3.32 (m, 2H), 2.54-2.60 (m, 1H), 2.33 (s, 3H), 1.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.1, 171.0, 168.4, 140.7, 138.3, 135.4, 129.5, 129.3, 128.6, 128.2, 128.1, 125.7, 123.7, 123.6, 115.4, 62.9, 58.2, 53.1, 47.9, 45.5, 41.9, 31.1, 21.4; IR (KBr) ν 3201, 2952, 1724, 1673, 1492, 1384, 1235, 825, 757 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1700.



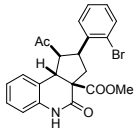
Tricycle 2h: White solid (54.7 mg, 0.12 mmol, 62% yield); m.p. 182-183°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 9.06 (s, 1H), 7.37-7.39 (m, 2H), 7.25-7.27 (m, 1H), 7.15-7.22 (m, 3H), 6.70-7.00 (m, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 4.48 (d, *J* = 10.8 Hz, 1H), 3.70-3.78 (m, 1H), 3.66 (s, 3H), 3.30-3.36 (m, 1H), 2.52-2.58 (m, 1H), 1.55 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 205.8, 170.8, 168.5, 143.2, 135.4, 131.8, 130.5, 130.3, 129.3, 128.3, 127.1, 123.8, 123.2, 122.6, 115.7, 62.7, 58.2, 53.2, 48.0, 45.1, 41.7, 31.4; IR (KBr) ν 3064, 2926, 2369, 1735, 1680, 1595, 1491, 1233, 788 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{22}H_{21}NO_4Br$ 442.0654, found 442.0674.



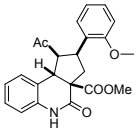
Tricycle 2i: White solid (40.0 mg, 0.11 mmol, 53% yield); m.p. 232-233°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.74 (s, 1H), 7.23-7.24 (m, 1H), 7.16-7.21 (m, 3H), 7.12-7.13 (m, 2H), 6.96-7.00 (m, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 4.55 (d, *J* = 10.0 Hz, 1H), 3.99-4.06 (m, 1H), 3.66 (s, 3H), 3.35-3.30 (m, 1H), 3.20 (dd, *J* = 13.2, 7.2 Hz, 1H), 2.64 (t, *J* = 12.2 Hz, 1H), 2.38 (s, 3H), 1.42 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.5, 171.0, 168.4, 138.5, 135.8, 135.4, 130.4, 129.2, 128.2, 127.7, 127.0, 126.7, 123.8, 123.7, 115.5, 62.1, 58.3, 53.1, 48.1, 41.2, 40.6, 30.5, 20.2; IR (KBr) ν 3066, 2989, 2928, 1735, 1679, 1494, 1436, 770 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{23}H_{23}NO_4Na$ 400.1525, found 400.1507.



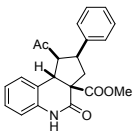
Tricycle 2j: White solid (52.4 mg, *inseparable two diastereomers, dr = 1.4:1*, 0.13 mmol, 66% yield); m.p. 206-208°C; ^1H NMR ($CDCl_3$, 500 MHz,) δ 8.66 (s, 1.0H), 8.58 (s, 0.73H), 7.60-7.57 (m, 1.8H), 7.40-7.36 (m, 1.5H), 7.35-7.30 (m, 3.0H), 7.27-7.24 (m, 2.1H), 7.22-7.16 (m, 3.0H), 6.02 (t, *J* = 6.5 Hz, 0.81H), 5.98-5.95 (m, 1.0H), 4.29 (dd, *J* = 10, 3 Hz, 1.1H), 4.14-4.12 (m, 0.87H), 3.51 (s, 0.47H), 3.49 (s, 2.8H), 3.21 (s, 2.3H), 3.02-3.00 (m, 0.8H), 2.88-2.80 (m, 2.1H), 2.72-2.58 (m, 4.9H), 2.05 (s, 2.9H), 2.02 (s, 2.6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 205.2, 205.0, 169.7, 169.3, 168.7, 168.5, 139.6, 138.4, 132.6, 129.8, 128.7, 127.4, 126.4, 115.9, 115.8, 55.7, 55.4, 54.9, 54.4, 52.9, 52.5, 42.8, 42.6, 40.0, 38.7, 30.8, 30.7; IR (KBr) ν 3205, 3073, 2922, 2372, 1726, 1679, 1491, 1371, 1216, 758 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{22}H_{21}NO_4Cl$ 398.1159, found 398.1145.



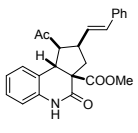
Tricycle 2k: White solid (60.9 mg, *two diastereomers, dr = 1.5:1*, 0.14 mmol, 69% yield); m.p. 170-173°C; ^{1}H NMR (DMSO-d_6, 400 MHz, *major isomer*) δ 10.56 (s, 1H), 7.66-7.56 (m, 2H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.28-7.23 (m, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 7.00-6.85 (m, 2H), 4.06-4.00 (m, 1H), 3.87-3.81 (m, 1H), 2.97-2.71 (m, 6H), 2.05 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, *major isomer*) δ 205.1, 169.2, 168.4, 140.1, 134.5, 133.0, 129.9, 129.4, 128.9, 128.3, 128.0, 123.9, 123.4, 115.8, 56.9, 55.2, 52.6, 42.6, 38.6, 30.8; IR (KBr) ν 3434, 2956, 2353, 1604, 1496, 1084, 873, 758 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4BrNa$ 464.0473, found 464.0465.



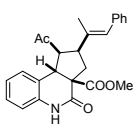
Tricycle 2l: White solid (68.4 mg, *inseparable two diastereomers, dr = 1.5:1*, 0.17 mmol, 87% yield); m.p. 197-199°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.46 (s, 1H), 9.24 (s, 1H), 7.15-7.27 (m, 8H), 6.81-6.98 (m, 8H), 4.45 (d, *J* = 9.6 Hz, 1H), 4.10-4.16 (m, 2.5H), 3.80 (s, 3H), 3.77 (s, 3H), 3.65 (s, 3H), 3.64 (s, 3H), 3.36-3.43 (m, 2H), 3.15-3.09 (m, 2H), 3.01-3.03 (m, 1H), 2.78 (t, *J* = 12.2 Hz, 1H), 1.65 (s, 3H), 1.53 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 208.7, 206.9, 171.8, 171.2, 169.9, 168.9, 157.2, 156.9, 135.5, 135.4, 129.4, 129.1, 129.0, 128.9, 128.8, 128.30, 128.26, 128.1, 123.9, 123.6, 123.5, 122.0, 121.0, 120.9, 115.72, 115.66, 110.7, 110.2, 63.7, 61.8, 61.8, 58.2, 55.1, 53.1, 53.0, 50.6, 48.2, 44.1, 39.4, 39.1, 38.8, 31.9, 30.7; IR (KBr) ν 3437, 3067, 2950, 2353, 1683, 1494, 1378, 1239, 758 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+ Na]^+$ calcd for $C_{23}H_{23}NO_5Na$ 416.1474, found 416.1470.



Tricycle 2m: White solid (54.5 mg, 0.15 mmol, 75% yield); m.p. 220-221°C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.70 (s, 1H), 7.30-7.34 (m, 2H), 7.22-7.25 (m, 3H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.2 Hz, 1H), 6.88-6.95 (m, 2H), 4.24 (d, *J* = 10.8 Hz, 1H), 3.79 (q, *J* = 10.0 Hz, 1H), 3.58 (s, 3H), 3.36-3.42 (m, 1H), 3.07-3.13 (m, 1H), 2.36-2.42 (m, 1H), 1.45 (s, 3H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 206.7, 171.6, 167.0, 141.8, 137.0, 129.1, 128.92, 128.85, 128.4, 127.4, 123.4, 122.9, 115.9, 62.6, 57.8, 53.3, 47.9, 44.9, 41.5, 31.2; IR (KBr) ν 3059, 2921, 2376, 1675, 1494， 1441, 1392, 1239, 871, 759 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{21}NO_4Na$ 386.1368, found 386.1349.

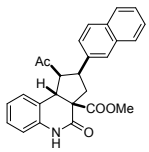


Tricycle 2n: White solid (49.8 mg, 0.13 mmol, 64% yield); m.p. 203-204°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.91 (s, 1H), 7.18-7.34 (m, 7H), 6.96-6.98 (m, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 6.44 (d, *J* = 15.2 Hz, 1H), 6.03 (dd, *J* = 15.6, 10.0 Hz, 1H), 4.32 (d, *J* = 10.8 Hz, 1H), 3.63 (s, 3H), 3.31-3.38 (m, 1H), 3.16-3.26 (m, 2H), 2.27-2.36 (m, 1H), 1.99 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.2, 170.9, 168.6, 136.5, 135.4, 132.0, 129.3, 129.0, 128.6, 128.2, 127.8, 126.4, 123.7, 123.2, 115.6, 61.5, 58.3, 53.1, 47.5, 43.0, 40.1, 31.9; IR (KBr) ν 3197, 3065, 2927, 2354, 1681, 1495, 1386, 1233, 756 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{24}H_{23}NO_4Na$ 412.1525, found 412.1511.

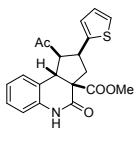


Tricycle 2o: colorless oil (62.1 mg, *inseparable two diastereomers, dr = 1:1*, 0.15 mmol, 77% yield); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.18 (s, 1H), 7.26-7.34 (m, 4H), 7.17-7.23 (m, 4H), 6.96-7.00 (m, 1H), 6.88 (d, *J* = 7.6 Hz, 1H),

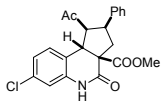
6.46 (s, 1H), 4.32 (d, *J* = 10.8 Hz, 1H), 3.63 (s, 3H), 3.44-3.52 (m, 1H), 3.23 (t, *J* = 11.2 Hz, 1H), 3.12 (dd, *J* = 13.2, 8.0 Hz, 1H), 2.55 (dd, *J* = 13.2, 10.0 Hz, 1H), 2.06 (s, 3H), 1.86 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.6, 171.1, 168.8, 137.3, 137.0, 135.4, 129.6, 128.8, 128.2, 126.7, 123.7, 123.3, 115.7, 61.8, 58.2, 53.1, 49.6, 48.2, 37.4, 31.5, 16.0; IR (KBr) ν 3058, 2925, 1732, 1675, 1596, 1490, 1361, 1234, 938, 735 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{25}H_{25}NO_4Na$ 426.1681, found 426.1664.



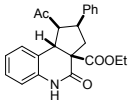
Tricycle 2p: White solid (65.3 mg, 0.16 mmol, 79% yield); m.p. 173-174°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.17 (*br* s, 1H), 7.80-7.82 (m, 3H), 7.68 (s, 1H), 7.44-7.50 (m, 2H), 7.30-7.36 (m, 2H), 7.18-7.23 (m, 1H), 6.97-7.01 (m, 1H), 6.90-6.92 (m, 1H), 4.62 (d, *J* = 10.8 Hz, 1H), 3.98 (q, *J* = 10.8 Hz, 1H), 3.68 (s, 3H), 3.37-3.44 (m, 1H), 2.72 (t, *J* = 12.4 Hz, 1H), 1.46 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.2, 171.0, 168.6, 138.2, 135.5, 133.2, 132.5, 129.4, 128.6, 128.3, 127.8, 127.7, 127.5, 126.4, 126.1, 123.8, 123.5, 115.7, 62.9, 58.3, 53.2, 48.1, 45.7, 41.9, 31.3; IR (KBr) ν 3198, 3059, 2934, 2353, 1677, 1495, 1383, 1228, 751 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{26}H_{23}NO_4Na$ 436.1525, found 436.1520.



Tricycle 2q: White solid (47.2 mg, 0.13 mmol, 64% yield); m.p. 196-197°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.07 (*br* s, 1H), 7.16-7.24 (m, 3H), 6.94-6.99 (m, 2H), 6.86-6.91 (m, 2H), 4.49 (d, *J* = 10.4 Hz, 1H), 4.06-4.13 (m, 1H), 3.66 (s, 3H), 3.39 (dd, *J* = 13.6, 8.0 Hz, 1H), 3.31 (t, *J* = 10.8 Hz, 1H), 2.63 (dd, *J* = 13.2, 10.4 Hz, 1H), 1.69 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.0, 170.7, 168.3, 143.3, 135.4, 129.3, 128.3, 127.2, 126.0, 124.3, 123.8, 123.1, 115.6, 62.4, 57.9, 53.2, 47.5, 42.6, 40.4, 31.3; IR (KBr) ν 3203, 3073, 2363, 1733, 1677, 1494, 1250, 823, 742 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{20}H_{20}NO_4S$ 370.1113, found 370.1107.

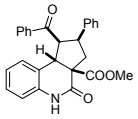


Tricycle 2r: White solid (59.6 mg, 0.15 mmol, 75% yield); m.p. 240-241°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 10.81 (s, 1H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.22-7.26 (m, 4H), 7.12 (d, *J* = 2.0 Hz, 1H), 6.96 (d, *J* = 8.8 Hz, 1H), 4.32 (d, *J* = 11.2 Hz, 1H), 3.79 (q, *J* = 9.6 Hz, 1H), 3.60 (s, 3H), 3.43 (t, *J* = 11.2 Hz, 1H), 3.10-3.15 (m, 1H), 2.36-2.41 (m, 1H), 1.47 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 205.8, 170.6, 168.4, 140.6, 134.1, 129.4, 128.8, 128.7, 128.6, 127.5, 125.2, 116.8, 62.6, 58.0, 53.3, 47.6, 45.5, 41.8, 31.0; IR (KBr) ν 3193, 3071, 2962, 2354, 1680, 1491, 1371, 1237, 844 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{22}H_{20}NO_4ClNa$ 420.0979, found 420.1023.

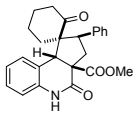


Tricycle 2s: colorless oil (61.8 mg, 0.16 mmol, 82% yield); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.52 (s, 1H), 7.17-7.33 (m, 7H), 6.97 (t, *J* = 7.4 Hz, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 4.49 (d, *J* = 10.8 Hz, 1H), 4.04-4.17 (m, 2H), 3.77-3.85 (m, 1H), 3.32-3.38 (m, 2H), 2.62 (dd, *J* = 13.2, 10.8 Hz, 1H), 1.48 (s, 3H), 1.10 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.3, 170.4, 169.1, 141.0, 135.7, 129.3, 128.7, 128.6, 128.2, 127.3, 123.6, 123.5, 115.7, 62.8, 61.9, 58.3, 48.1,

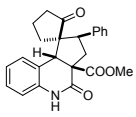
45.5, 41.5, 31.1, 13.9; IR (KBr) ν 3061, 2983, 1729, 1677, 1492, 1365, 1229, 861, 732 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1699.



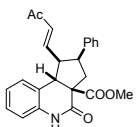
Tricycle 2t: White solid (62.9 mg, *inseparable two diastereomers, dr = 13:1*, 0.15 mmol, 74% yield); m.p. 271-272°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.72 (*br* s, 1H), 7.40-7.45 (m, 3H), 7.33-7.36 (m, 1H), 7.16-7.21 (m, 3H), 6.94-7.04 (m, 6H), 6.86-6.88 (m, 1H), 4.76 (d, *J* = 11.2 Hz, 1H), 4.11 (t, *J* = 11.2 Hz, 1H), 3.87-3.94 (m, 1H), 3.67 (s, 3H), 3.48 (dd, *J* = 13.6, 8.4 Hz, 1H), 2.70 (dd, *J* = 13.6, 9.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 197.8, 171.2, 168.8, 140.8, 137.1, 135.5, 132.7, 129.9, 128.8, 128.3, 128.1, 128.0, 126.7, 123.8, 123.3, 115.6, 58.4, 57.5, 53.2, 48.3, 46.7, 42.1; IR (KBr) ν 3194, 3062, 2353, 1729, 1681, 1493, 1237, 1070, 760, 686 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{27}H_{24}NO_4$ 426.1705, found 426.1700.



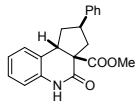
Tetracycle 2u: White solid (70.1 mg, *inseparable two diastereomers, dr = 9:1*, 0.17 mmol, 87% yield); m.p. 258-259°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.55 (s, 1H), 7.28-7.35 (m, 3H), 7.10-7.25 (m, 4H), 7.00 (t, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.66 (s, 3H), 3.40-3.51 (m, 2H), 2.51 (dd, *J* = 12.8, 8.8 Hz, 1H), 2.00-2.07 (m, 1H), 1.87-1.91 (m, 1H), 1.69-1.82 (m, 2H), 1.48-1.52 (m, 1H), 1.16-1.32 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 210.2, 171.8, 169.1, 142.5, 137.2, 130.1, 129.0, 128.8, 128.2, 127.2, 123.8, 122.3, 115.3, 66.4, 57.3, 55.0, 53.2, 50.3, 42.2, 41.2, 35.7, 26.3, 20.8; IR (KBr) ν 3205, 3074, 2937, 2353, 1686, 1492, 1226, 767 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{25}H_{25}NO_4Na$ 426.1681, found 426.1665.



Tetracycle 2v: White solid (59.1 mg, *inseparable two diastereomers, dr = 1.8:1*, 0.15 mmol, 76% yield); m.p. 174-175 °C; ^1H NMR ($CDCl_3$, 400 MHz, *major isomer*) δ 9.22 (s, 1H), 7.19-7.31 (m, 9H), 6.91-6.97 (m, 4H), 3.67 (s, 3H), 3.21-3.30 (m, 2H), 2.65-2.70 (m, 1H), 1.64-1.88 (m, 4H), 1.54-1.57 (m, 1H), 1.26-1.41 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, *major isomer*) δ 218.5, 171.2, 169.2, 140.2, 136.7, 129.0, 128.9, 128.7, 128.6, 128.5(1), 128.4(7), 128.2, 127.9, 127.5, 127.3, 123.7, 121.8, 120.1, 115.9, 115.6, 66.2, 65.0, 58.3, 56.6, 55.8, 55.3, 53.2, 53.1, 52.3, 51.5, 40.7, 39.3, 37.0, 36.1, 34.6, 24.4, 18.6, 17.9; IR (KBr) ν 3300, 2956, 2353, 1709, 1533, 1449, 1331, 1147, 755 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{24}H_{24}NO_4$ 390.1705, found 390.1693.

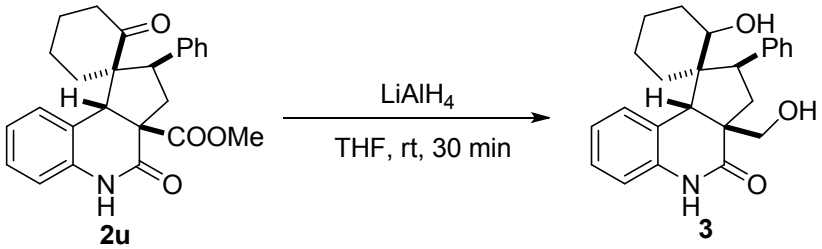


Tricycle 2w: White solid (31.9 mg, 0.082 mmol, 41% yield); m.p. 93-94°C; ^1H NMR ($CDCl_3$, 400 MHz) δ 9.16 (s, 1H), 7.34 (d, *J* = 7.4 Hz, 2H), 7.26-7.27 (m, 2H), 7.18-7.25 (m, 2H), 6.89-6.96 (m, 3H), 6.11 (dd, *J* = 16.0, 10.0 Hz, 1H), 5.57 (d, *J* = 16.0 Hz, 1H), 3.65-3.73 (m, 5H), 3.44 (dd, *J* = 13.6, 8.0 Hz, 1H), 2.86-2.97 (m, 2H), 1.91 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 197.9, 171.6, 168.6, 146.5, 140.3, 135.5, 132.8, 128.7, 128.60, 128.56, 128.45, 127.0, 123.4, 121.9, 115.8, 59.1, 53.6, 53.3, 52.5, 47.1, 39.5, 26.4; IR (KBr) ν 3061, 2993, 2931, 2354, 1729, 1679, 1495, 1243, 759 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{24}H_{23}NO_4Na$ 412.1525, found 412.1518.



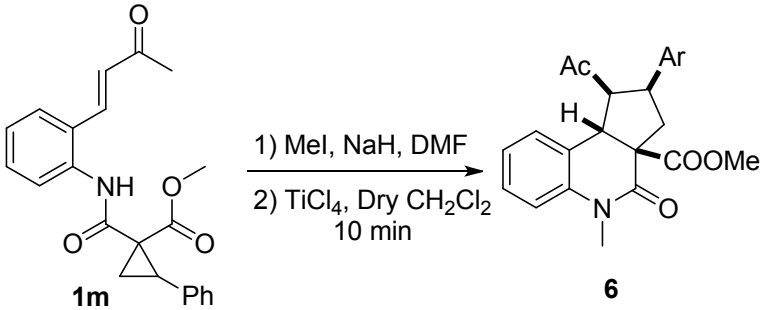
Tricycle 2x: White solid (27.6 mg, *inseparable two diastereomers, dr = 2.0:1*, 0.086 mmol, 43% yield); m.p. 99-100°C; ^{1}H NMR ($CDCl_3$, 400 MHz, *major isomer*) δ 9.51 (s, 1H), 7.38-7.41 (m, 1H), 7.26-7.30 (m, 4H), 7.16-7.18 (m, 1H), 7.03-7.12 (m, 2H), 6.60 (dd, *J* = 17.6, 10.8 Hz, 1H), 5.61 (dd, *J* = 17.6, 1.2 Hz, 1H), 5.29 (dd, *J* = 10.8, 1.2 Hz, 1H), 3.84 (s, 3H), 3.79 (d, *J* = 2.4 Hz, 1H), 3.18 (t, *J* = 9.0 Hz, 1H), 2.59-2.63 (m, 1H), 2.12 (dd, *J* = 9.2, 4.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz, *major isomer*) δ 172.7, 163.0, 134.6, 134.2, 132.1, 130.7, 129.2, 128.2, 128.2, 127.6, 126.1, 125.1, 123.9, 116.9, 52.8, 37.7, 36.8, 30.3, 29.7, 18.5; IR (KBr) ν 3262, 3042, 2949, 2353, 1736, 1658, 1272, 1157, 760 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{20}H_{19}NO_3Na$ 344.1263, found 344.1257.

2.4 Experimental procedure for the synthesis of **alcohol 3**.



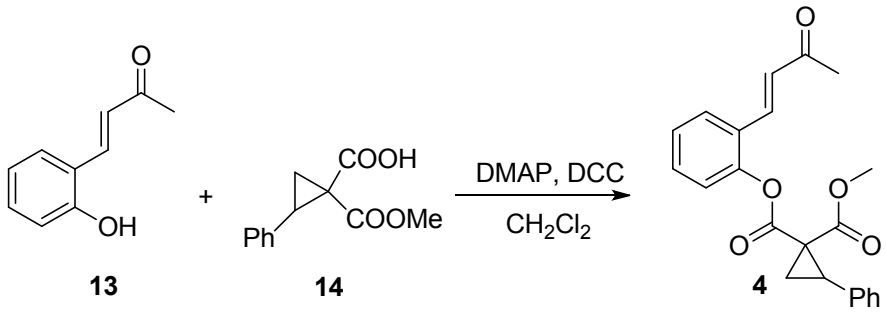
To a solution of **2u** (80.6 mg, 0.2 mmol, 1.0 equiv.) in anhydrous THF (2 mL) was added LAH (22.8 mg, 0.6 mmol, 3.0 equiv.) in small portions. The mixture was stirred at room temperature for 30 minutes. Then excess LAH was quenched by water carefully at 0℃, and extracted with EtOAc (10 mL × 2). The combined organic layer was washed with brine, dried over $MgSO_4$, concentrated, and purified through column chromatography (eluenting with EtOAc/PE = 1:5) to afford alcohol **3** as a white solid (47.5 mg, 0.13 mmol, 63% yield); m.p. 271-272°C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.07 (s, 1H), 7.59 (d, *J* = 6.0 Hz, 2H), 7.12-7.24 (m, 5H), 6.86-6.97 (m, 2H), 5.02 (t, *J* = 5.4 Hz, 1H), 4.24 (d, *J* = 4.8 Hz, 2H), 4.14 (s, 1H), 3.41-3.46 (m, 1H), 3.31-3.34 (m, 1H), 3.02 (t, *J* = 8.6 Hz, 1H), 2.66-2.72 (m, 1H), 2.17 (t, *J* = 11.2 Hz, 1H), 1.46-1.55 (m, 1H), 1.27-1.37 (m, 2H), 1.18-1.20 (m, 2H), 0.83-1.01 (m, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 173.7, 145.2, 139.0, 131.8, 129.9, 127.6, 126.1, 123.6, 122.3, 115.0, 73.1, 64.9, 55.9, 52.6, 52.2, 47.48, 47.45, 35.5, 31.2, 25.3, 21.3; IR (KBr) ν 3412, 2934, 2870, 2354, 1664, 1493, 1339, 1060, 766 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{24}H_{28}NO_3$ 378.2069, found 378.2065.

2.5 Experimental procedure for the synthesis of **tricycle 6.**



To a solution of *α,β*-unsaturated enone **1m** (145.2 mg, 0.4 mmol) in anhydrous DMF (2.0 mL) was added sodium hydride (20.0 mg, 0.5 mmol, 60% dispersion in mineral oil, 1.25 equiv.) in a ice bath. After 10 minutes, iodomethane (71.0 mg, 31.1 μL, 0.5 mmol, 1.25 equiv.) was added slowly. The mixture was stirred at 0℃ for 10 minutes and then 30 minutes at room temperature. Subsequently, water (20 mL) was added and extracted with EtOAc (10 mL ×2). The combined organic phases was washed with brine (10 mL × 2), dried over $MgSO_4$, concentrated *in vacuo* to afford the crude product which was used in the next step without further purification. To the solution of the crude product in anhydrous CH_2Cl_2 (2.0 mL) was added titanium tetrachloride (45.5 mg, 26.3 μL, 0.24 mmol) at room temperature. The solution was stirred for 10 minutes, and brine (10 mL) was then added to it. The resulting mixture was stirred for another 10 minutes. The aqueous phase was extracted with EtOAc (3 mL × 2). The combined organic layer was dried, concentrated, and purified *via* column chromatography (eluenting with EtOAc/PE = 1:3) to afford **tricycle 6** as a white solid (66.4 mg, *inseparable two diastereoisomers, dr = 3:1*, 0.18 mmol, 44% yield over two steps); m.p. 85-87°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.24-7.34 (m, 6.5H), 7.20-7.23 (m, 3.5H), 7.00-7.04 (m, 2.7H), 4.45-4.48 (m, 1.0H), 3.72-3.79 (m, 1.0H), 3.60 (s, 3.5H), 3.52 (s, 3.5H), 3.35-3.41 (m, 1.5H), 3.22-3.28 (m, 1.1H), 2.50-2.56 (m, 1.3H), 1.46 (s, 2.9H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 206.0, 171.1, 167.3, 141.0, 129.6, 128.8, 128.7, 128.6, 128.2, 127.3, 124.7, 123.4, 114.9, 62.3, 58.3, 52.9, 47.4, 45.4, 43.0, 31.0, 30.5, 6.8; IR (KBr) ν 3437, 2947, 2355, 1726, 1674, 1367, 1247, 761 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+H]^+$ calcd for $C_{23}H_{24}NO_4$ 378.1705, found 378.1711.

2.6 Experimental procedure for the synthesis of ester **4**.



13 **14** **4**

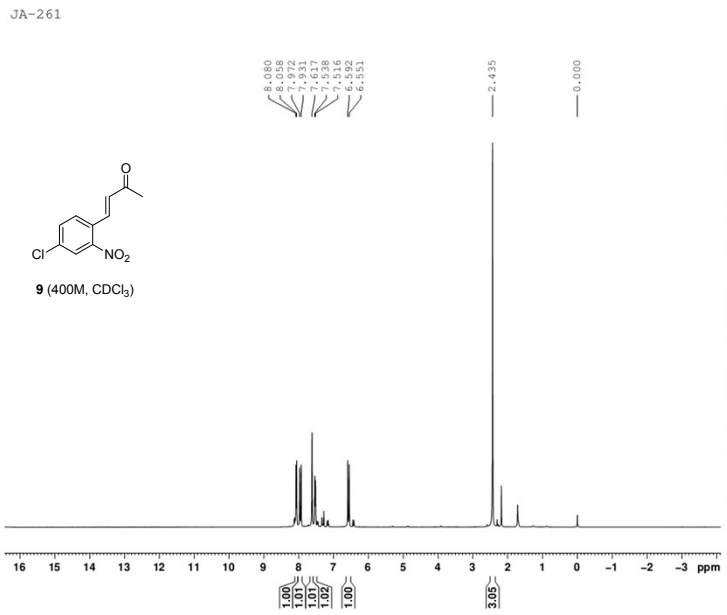
To a solution of *o*-hydroxyl-*α,β*-unsaturated enone **13** (1.0 g, 6.17 mmol, 1.0 equiv.) and cyclopropane dicarboxylic monoester **14** (1.49 g, 6.79 mmol, 1.2 equiv.) in dichloromathane (60 mL) were added DCC (1.53 g, 7.40 mmol, 1.2 equiv.) and DMAP (0.23 g, 1.85 mmol, 0.3 equiv.) respectively. The resulting mixture was stirred at room temperature for 48 hours, filtered, and concentrated. The residue was purified through column chromatogrophy (eluenting with EtOAc/PE = 1:9) to yield **4** (1.80 g, 4.96 mmol, 74% yield); m.p. 76-77°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.79 (d, *J* = 16.4 Hz, 1H), 7.69 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.41-7.46 (m, 1H), 7.25-7.34 (m, 6H), 7.20 (dd, *J* = 8.0, 0.8 Hz, 1H), 6.74 (d, *J* = 16.4 Hz, 1H), 3.46 (s, 3H), 3.39 (t, *J* = 8.8 Hz, 1H), 2.37-2.44 (m, 4H), 1.92 (dd, *J* = 9.6, 5.6 Hz, 1H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 198.4, 168.4, 166.6, 149.3, 136.5, 134.0, 131.3, 129.1, 128.6, 128.3, 127.8, 127.4, 126.8, 122.9, 52.5, 37.2, 33.3, 27.5, 20.1; IR (KBr) ν 3061, 3001, 1736, 1671, 1438, 1273, 1214, 974, 765 cm^{-1}; HRMS (TOF-ES+) m/z: $[M+ Na]^+$ calcd for $C_{22}H_{20}O_5Na$ 378.1208, found 378.1212.

References:

1 (a) D. Alonso, E. Caballero, M. Medarde and F. Tomé, *Tetrahedron Lett.*, 2005, **46**, 4839; (b) U. Das, A. Doroudi, S. Das, B. Bandy, J. Balzarini, E. De Clercq and J. R. Dimmock, *Bioorg. & Med. Chem.*, 2008, **16**, 6261.

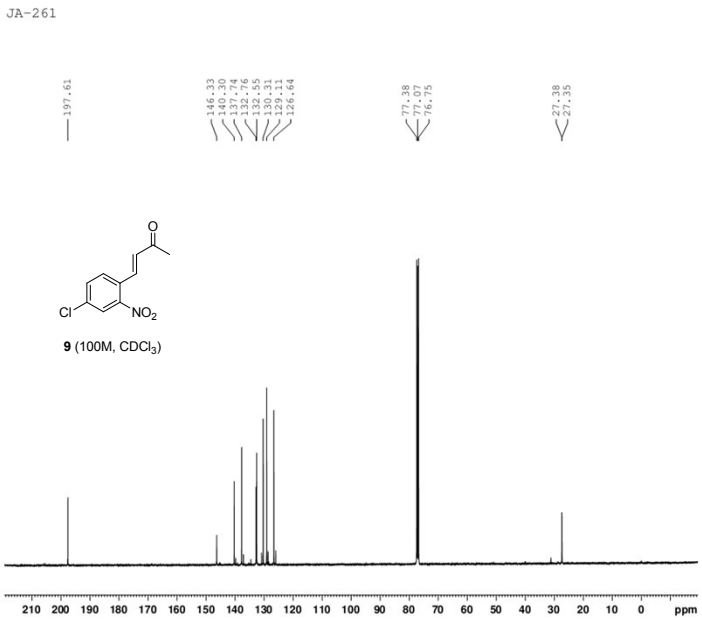
2 X. R. He, G. P. Qiu, J. Yang, Y. L. Xiao, Z. Y. Wu, G. F. Qiu and X. M. Hu, *Eur. J. Med. Chem.*, 2010, **45**, 3818.

3. NMR Spectra of 1a-1y, 2a-2x, 3-6, 9.



9 (400M, $CDCl_3$)

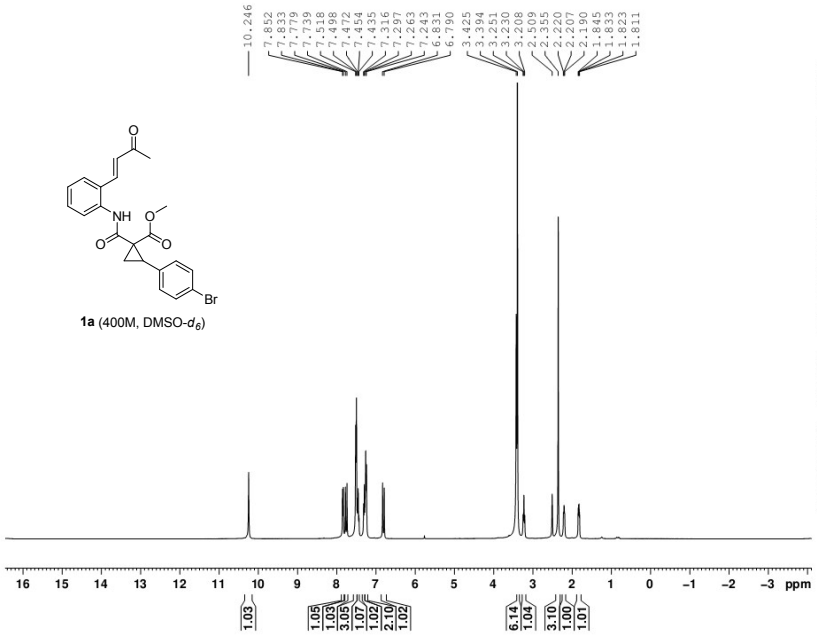

```
Current Data Parameters
NAME                  06-05-2015
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20150605
Time                      13.29
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  CDC13
NS                          52
DS                           2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                       138.14
DW                     60.800 usec
DE                      6.50 usec
TE                      296.9 K
D1                 1.00000000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                      14.00 usec
PLW1                 9.72999954 W
SFO1               400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF                 400.1300034 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```



9 (100M, $CDCl_3$)


```
Current Data Parameters
NAME                  06-05-2015
EXPNO                          7
PROCNO                         1

F2 - Acquisition Parameters
Date_                  20150609
Time                      13.38
INSTRUM                   spect
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDC13
NS                        2188
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                       198.33
DW                     20.800 usec
DE                      6.50 usec
TE                      296.3 K
D1                 2.00000000 sec
D11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                        13C
P1                       9.00 usec
PLW1                50.09999847 W
SFO1               100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                   90.00 usec
PLW2                 9.36999989 W
PLW12                0.22673000 W
PLW13                0.18365000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127690 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

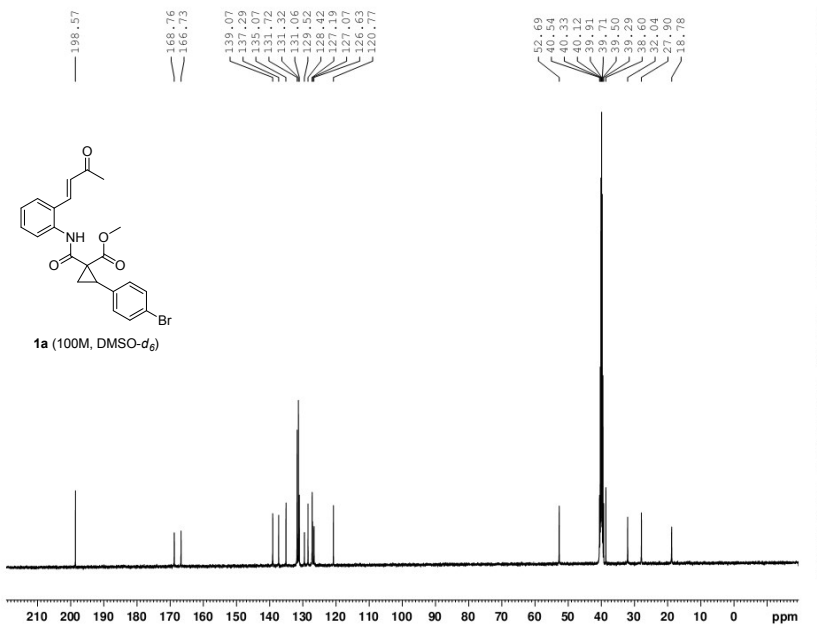
JA-223



1a (400M, DMSO-d_6)

```
Current Data Parameters
NAME          03-17-2015
EXPNO                 18
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150317
Time               16.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                    64
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 87.39
DW                60.800 usec
DE                 6.50 usec
TE                291.9 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

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

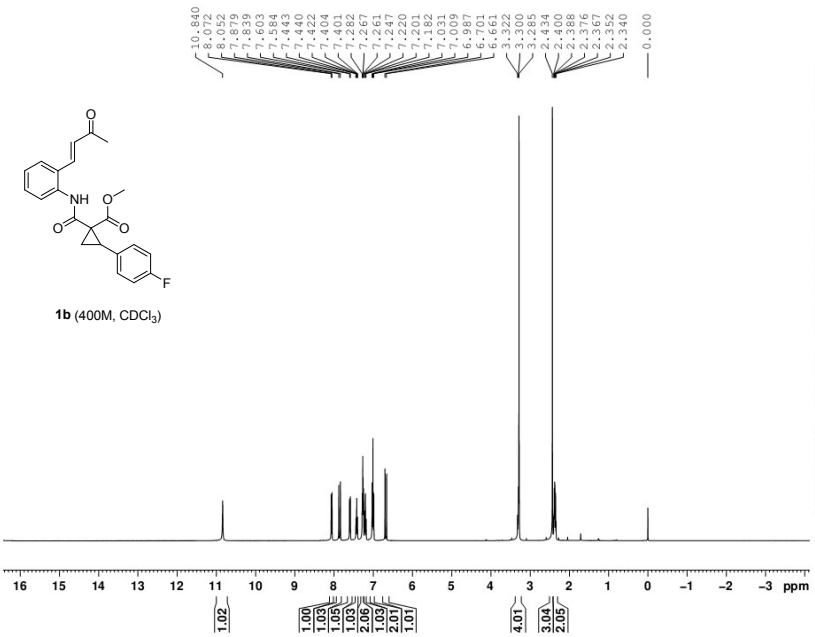
JA-223



1a (100M, DMSO-d_6)

```
Current Data Parameters
NAME          03-17-2015
EXPNO                 26
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150319
Time               11.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                   596
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                106.96
DW                20.800 usec
DE                 6.50 usec
TE                291.3 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

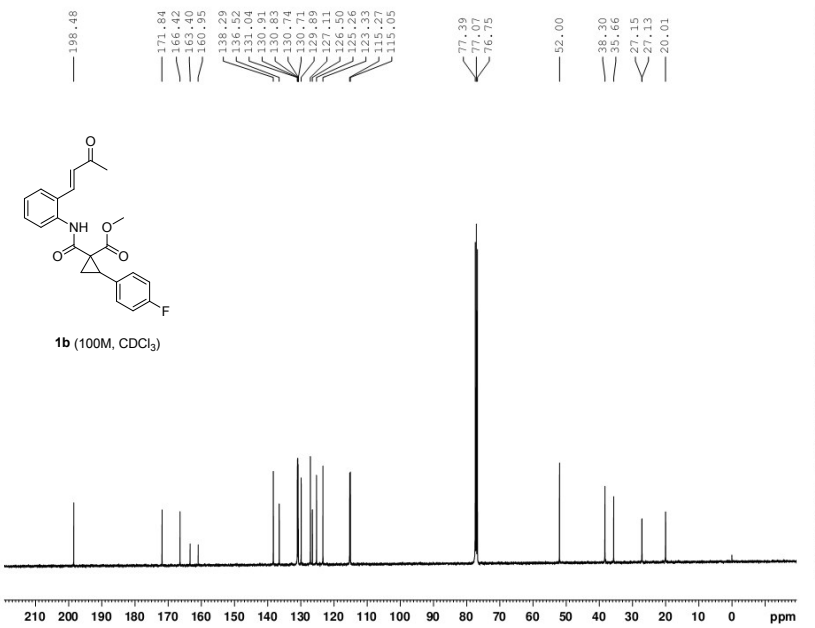
JA-238



1b (400M, $CDCl_3$)

```
Current Data Parameters
NAME          04-23-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150423
Time               15.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                106.96
DW                60.800 usec
DE                 6.50 usec
TE                294.2 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300073 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

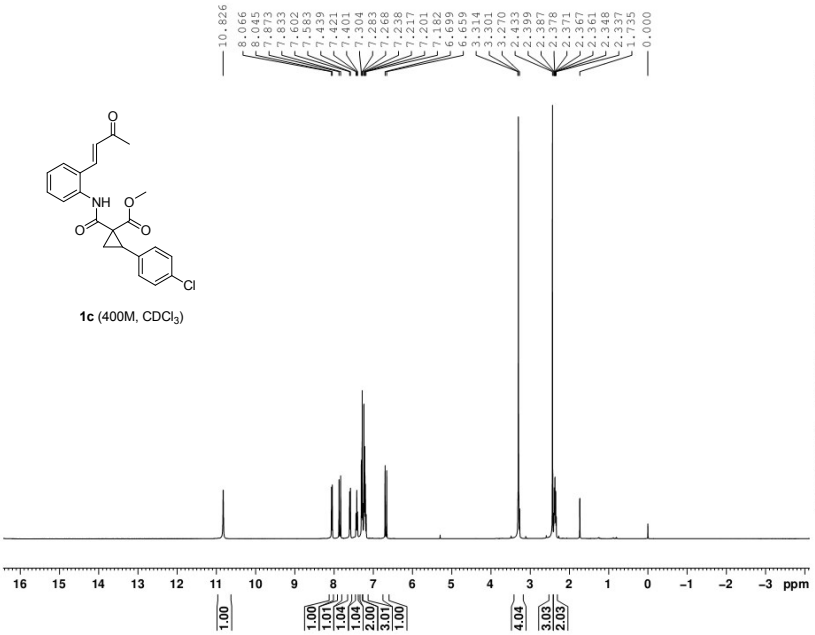
JA-238



1b (100M, $CDCl_3$)

```
Current Data Parameters
NAME          04-23-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150424
Time               13.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  1672
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                168.03
DW                20.800 usec
DE                 6.50 usec
TE                294.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

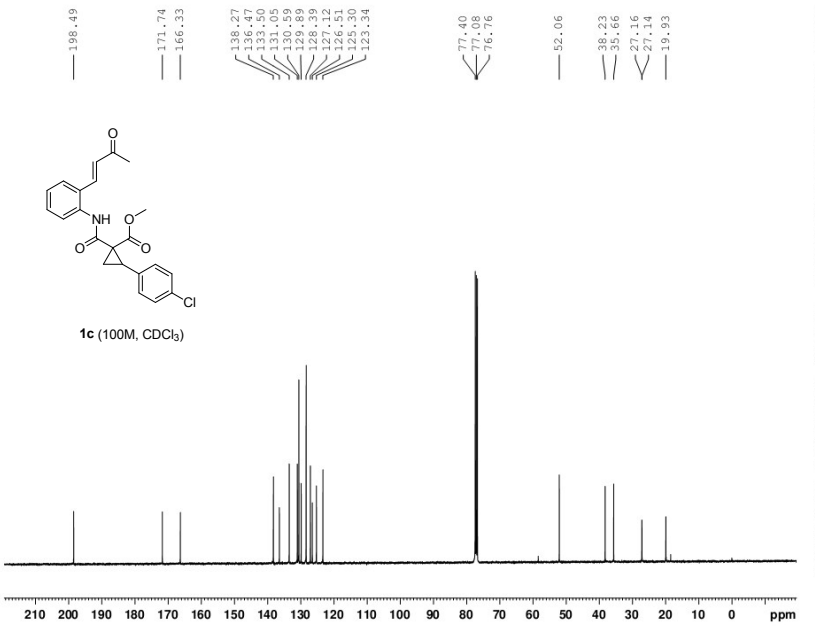
JA-237



1c (400M, $CDCl_3$)

```
Current Data Parameters
NAME          04-23-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150423
Time               14.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                294.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300071 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

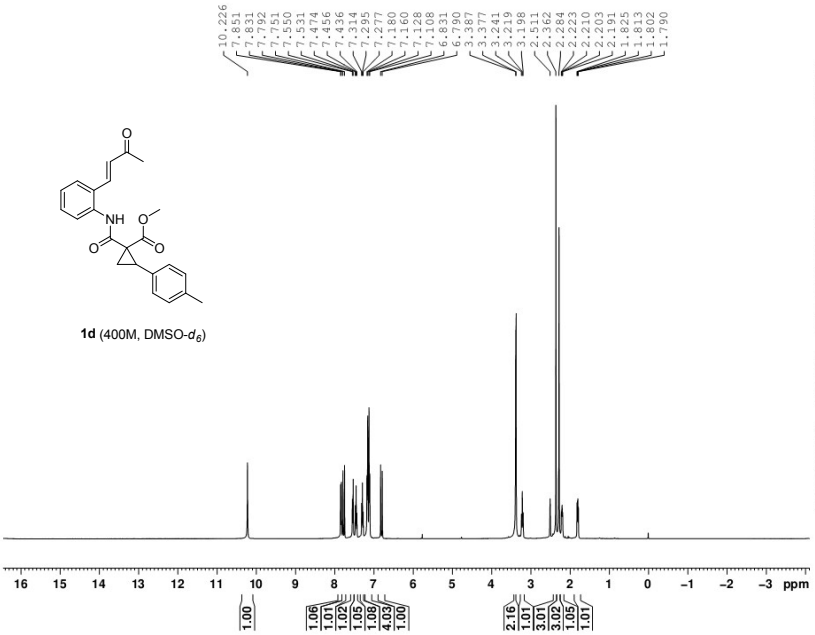
JA-237



1c (100M, $CDCl_3$)

```
Current Data Parameters
NAME          04-23-2015
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150424
Time               12.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                 1788
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                168.03
DW                20.800 usec
DE                 6.50 usec
TE                294.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

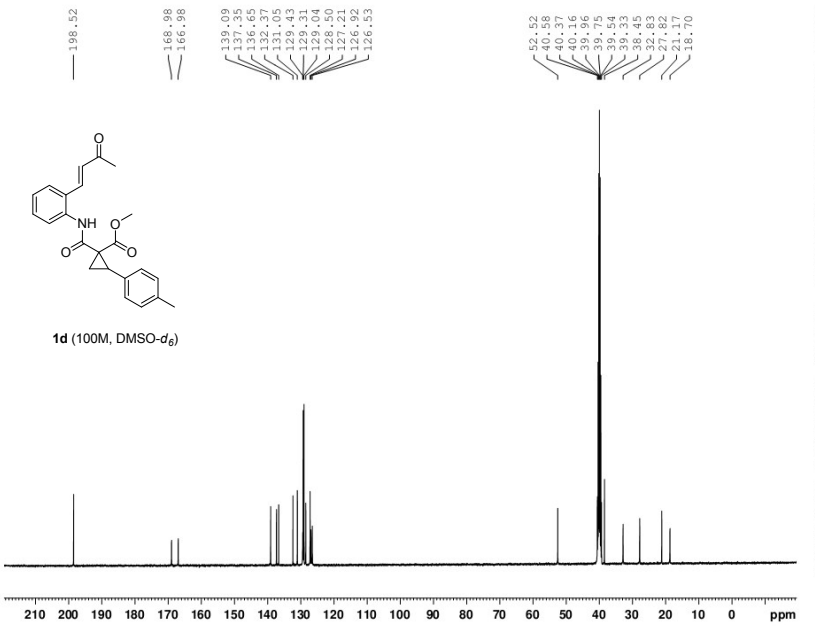
JA-227



1d (400M, DMSO-d_6)

```
Current Data Parameters
NAME          03-24-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150324
Time               11.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 67.01
DW                60.800 usec
DE                 6.50 usec
TE                292.3 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

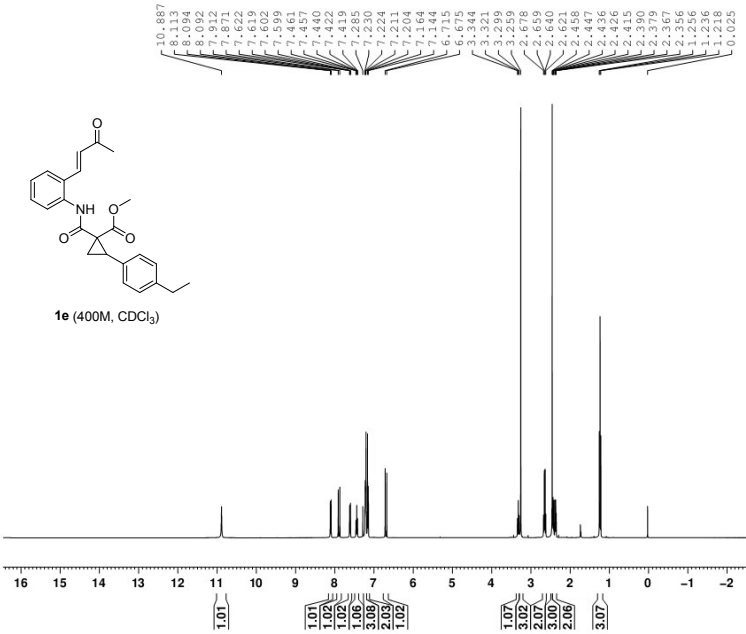
JA-227



1d (100M, DMSO-d_6)

```
Current Data Parameters
NAME          03-24-2015
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150324
Time               18.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                  1316
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                152.51
DW                20.800 usec
DE                 6.50 usec
TE                293.6 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

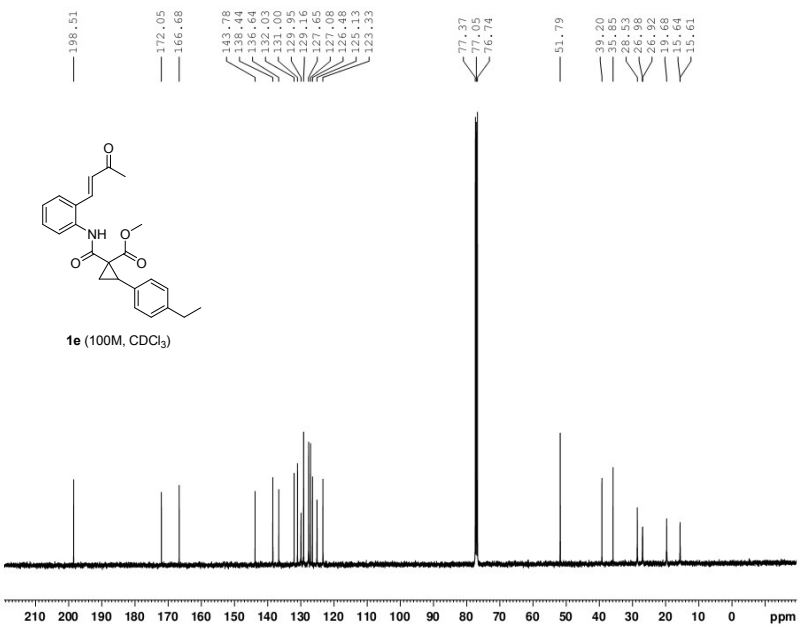
JA-303



1e (400M, $CDCl_3$)

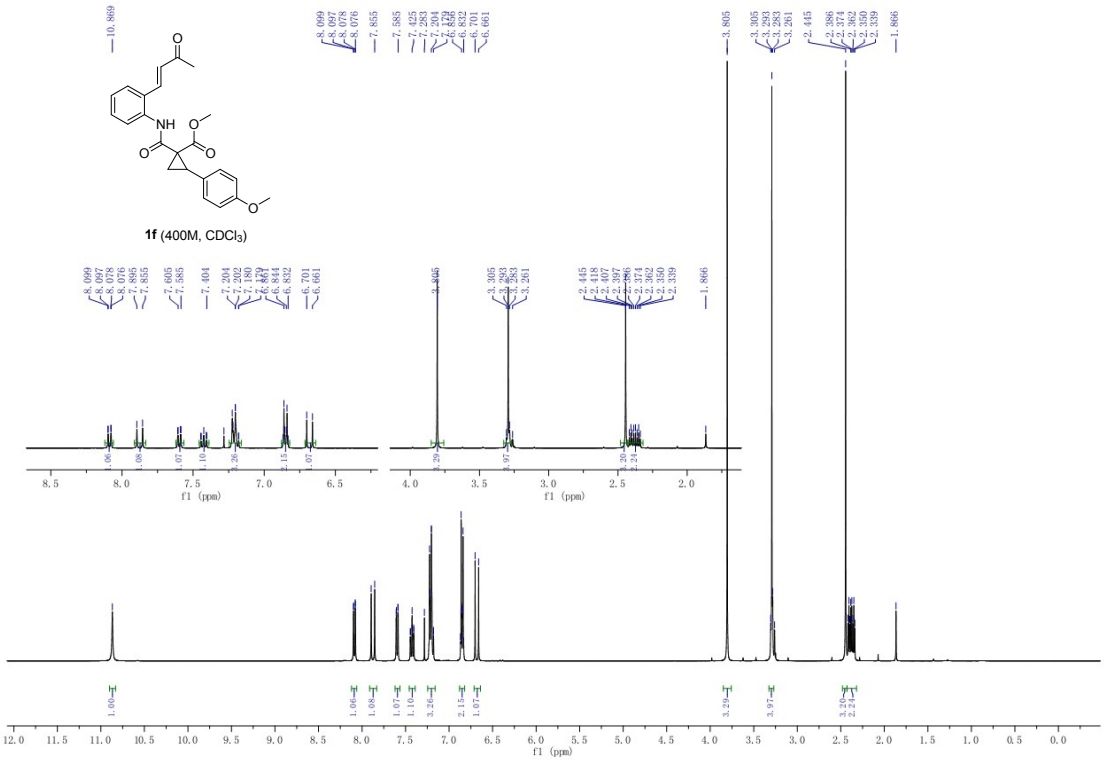
```
Current Data Parameters
NAME          08-04-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time               10.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   60
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                94.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

JA-303

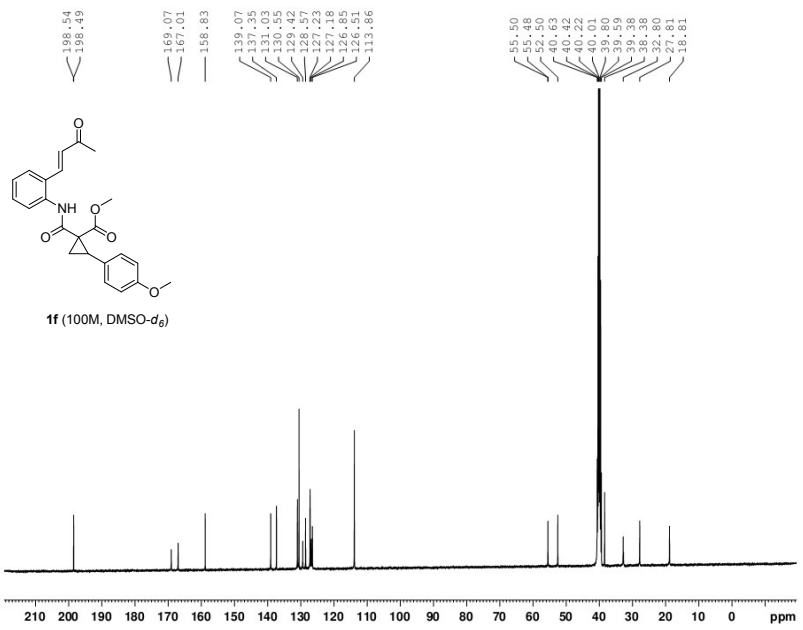


1e (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time               17.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  640
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



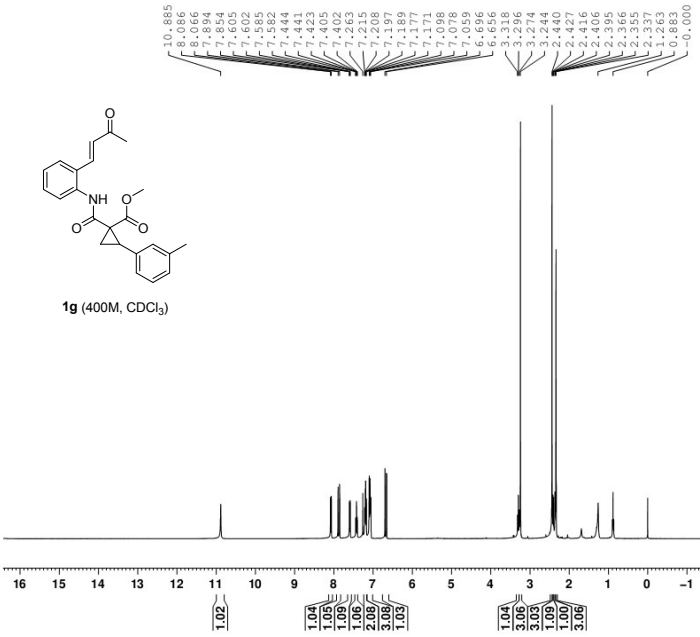
JA-229



BRUKER

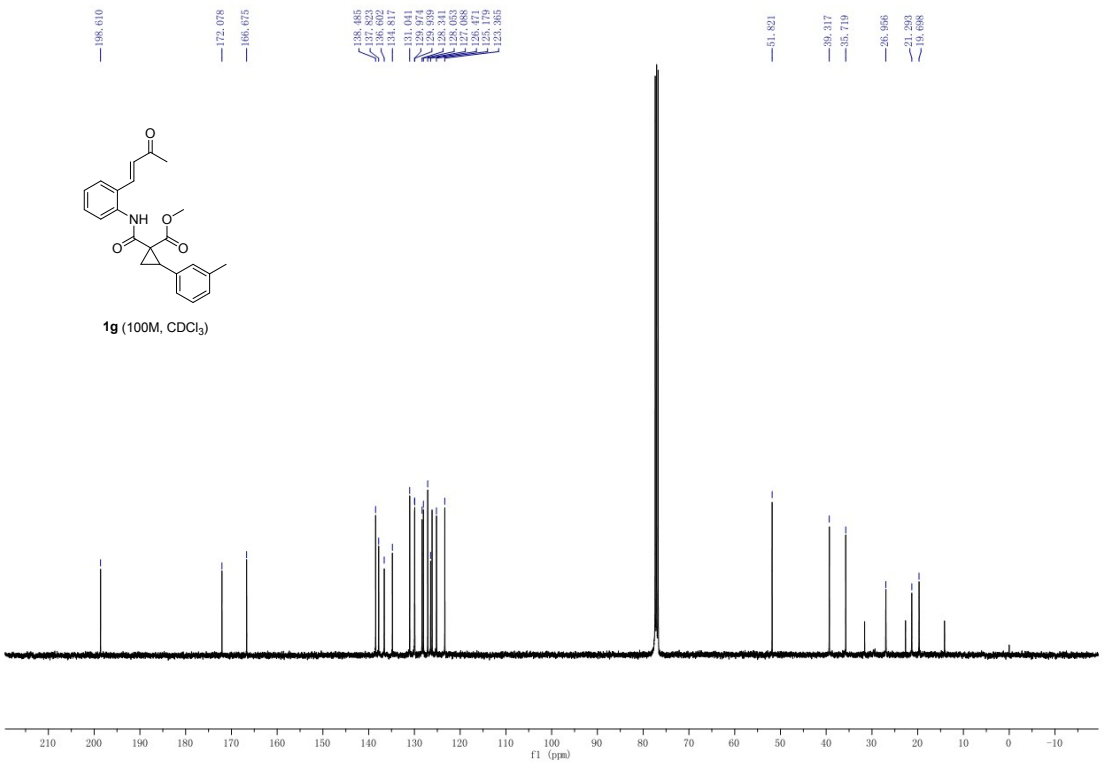
```
Current Data Parameters
NAME              03-27-2015
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20150330
Time                  18.27
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                    4384
DS                       4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                   152.51
DW                  20.800 usec
DE                   6.50 usec
TE                  297.5 K
D1                 2.00000000 sec
D11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.00 usec
PLW1          50.09999847 W
SFO1         100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           9.36999989 W
PLW12          0.22673000 W
PLW13          0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                     32768
SP            100.6127690 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

JA-251



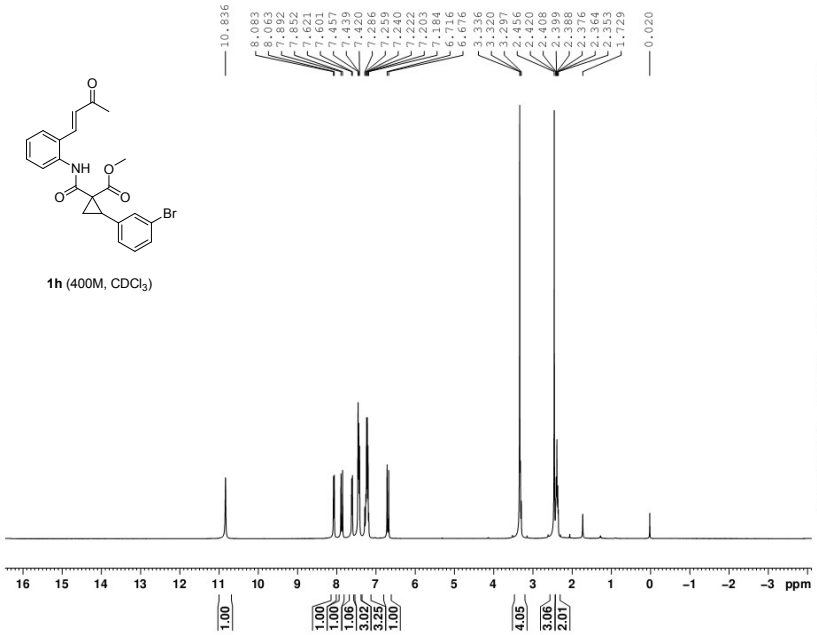
1g (400M, $CDCl_3$)

```
Current Data Parameters
NAME          05-19-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150519
Time               14.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                106.96
DW                60.800 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300092 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1g (100M, $CDCl_3$)

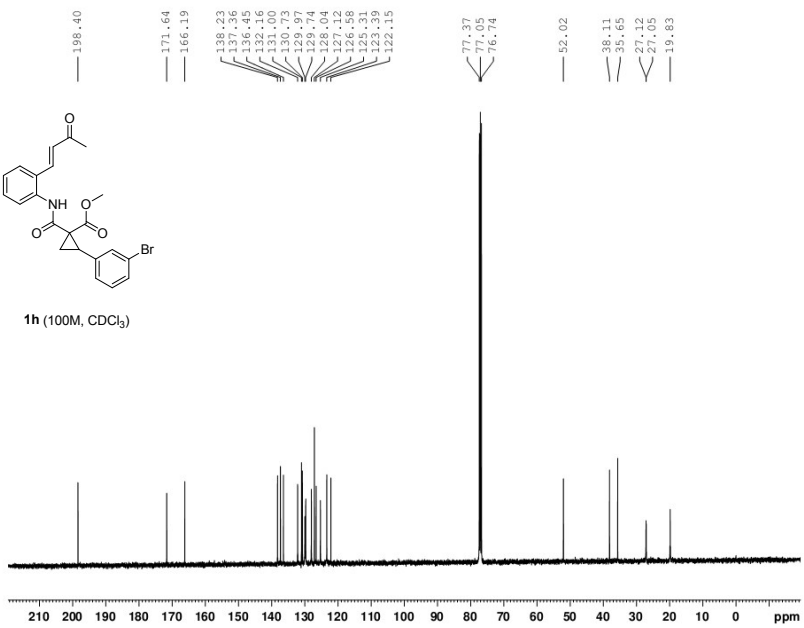
JA-301



1h (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time                9.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   32
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ             3.9846387 sec
RG                94.96
DW               60.800 usec
DE                6.50 usec
TE               518.1 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

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

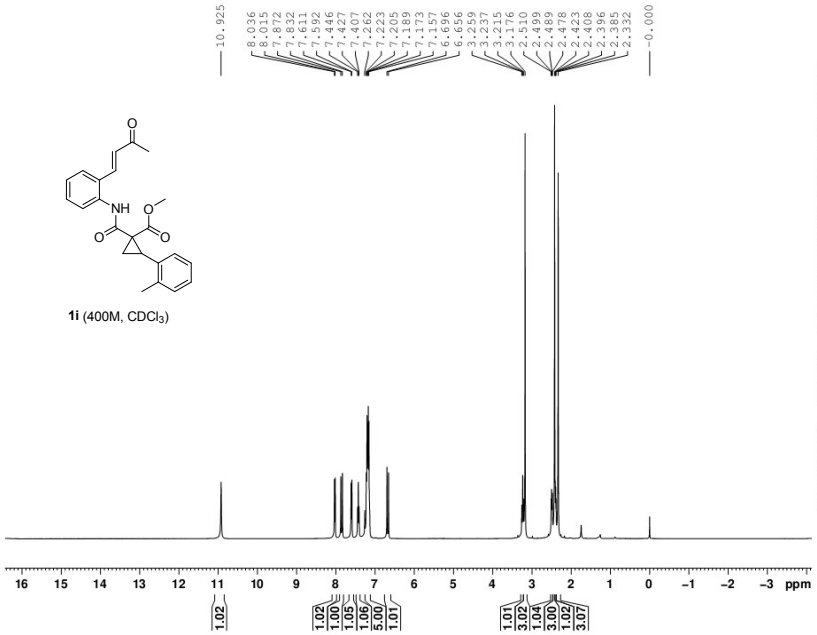
JA-301



1h (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time               15.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                  736
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ             1.3631988 sec
RG                198.33
DW               20.800 usec
DE                6.50 usec
TE               518.1 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF         100.6127690 MHz
WDW                    EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

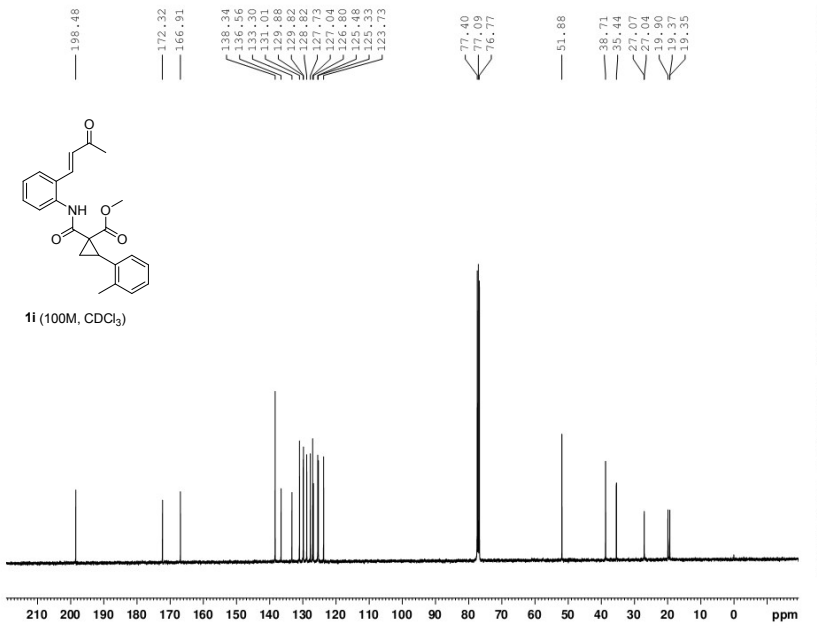
JA-248



1i (400M, $CDCl_3$)

```
Current Data Parameters
NAME          05-14-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150514
Time               11.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   36
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                87.39
DW                60.800 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300095 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

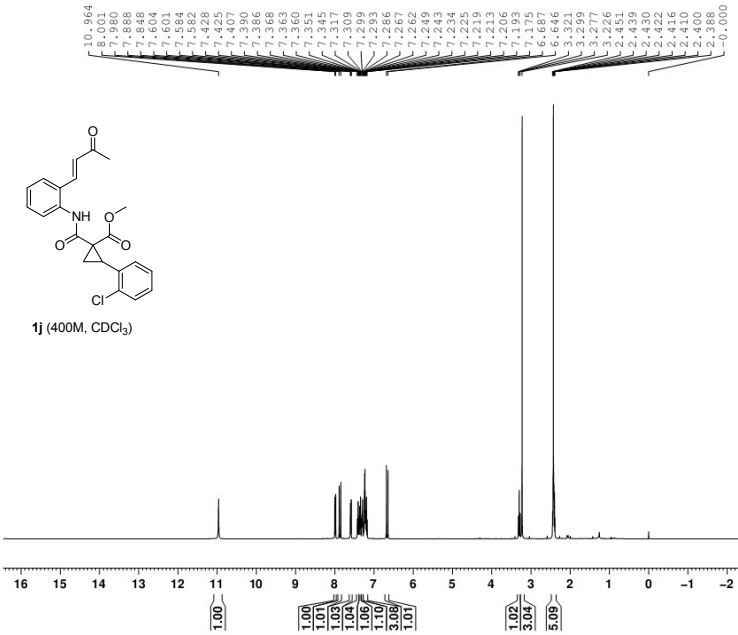
JA-248



1i (100M, $CDCl_3$)

```
Current Data Parameters
NAME          05-14-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150515
Time               10.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  840
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

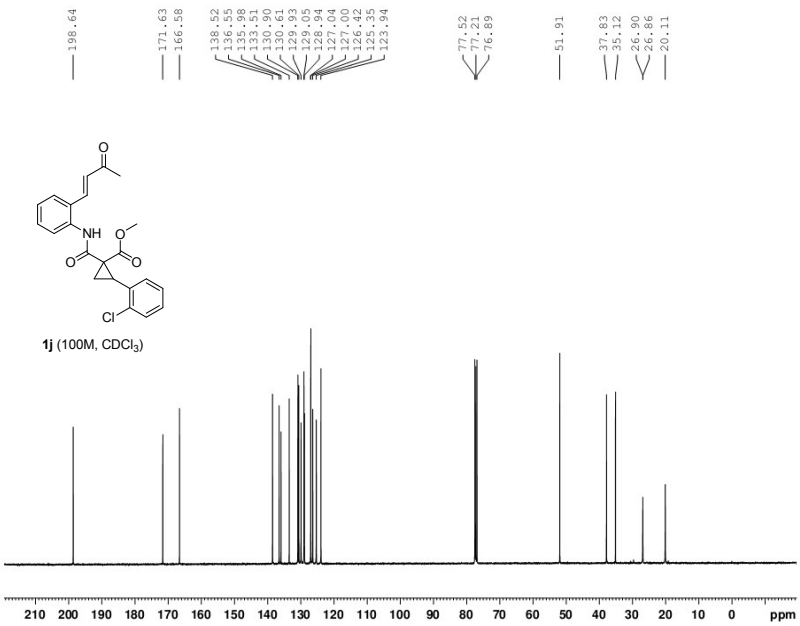
JA-296



1j (400M, $CDCl_3$)

```
Current Data Parameters
NAME          07-30-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150730
Time               11.06
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    36
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 33.09
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF           400.1299993 MHz
WDW                      EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

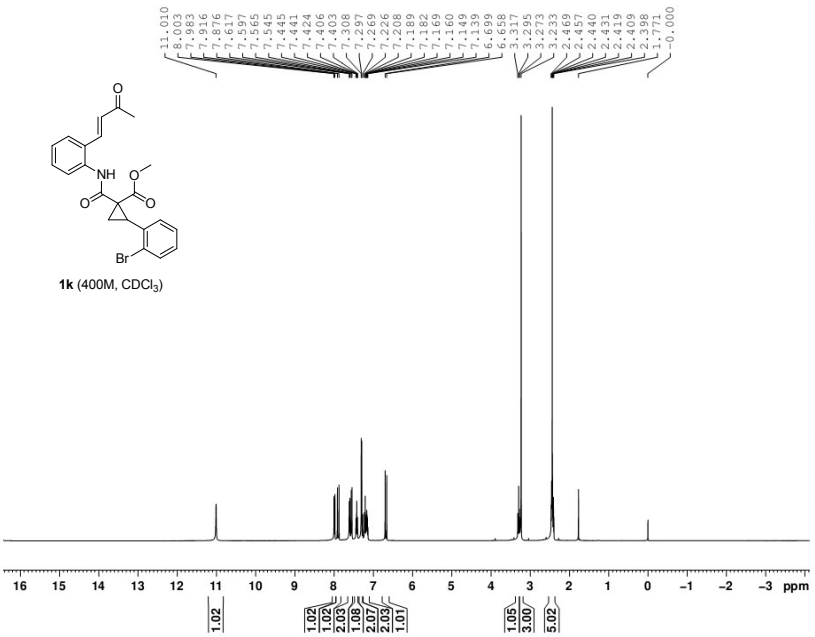
JA-296



1j (100M, $CDCl_3$)

```
Current Data Parameters
NAME          07-30-2015
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150731
Time               13.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  1132
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                  9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127690 MHz
WDW                      EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

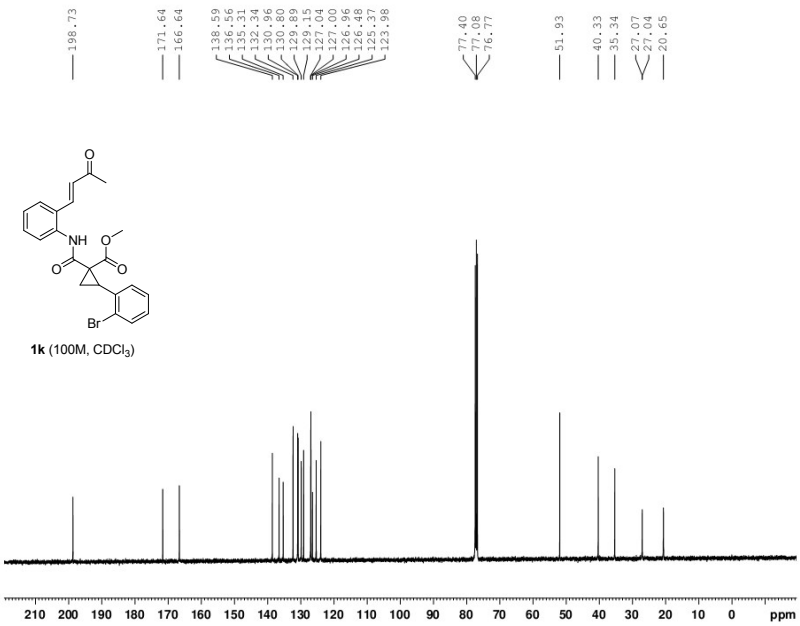
JA-258



1k (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-01-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150601
Time               13.41
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                295.9 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300066 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

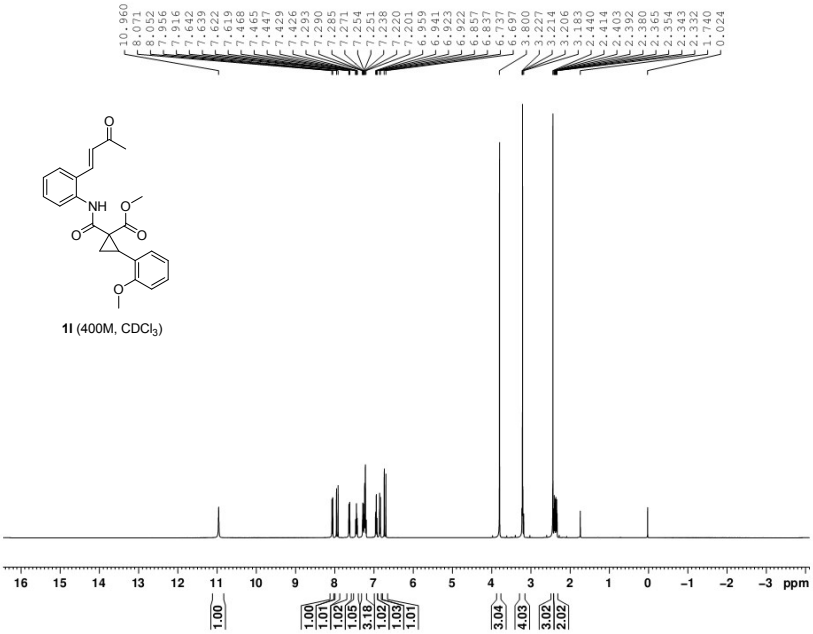
JA-258



1k (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-01-2015
EXPNO                 15
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150609
Time                9.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   636
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.6 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

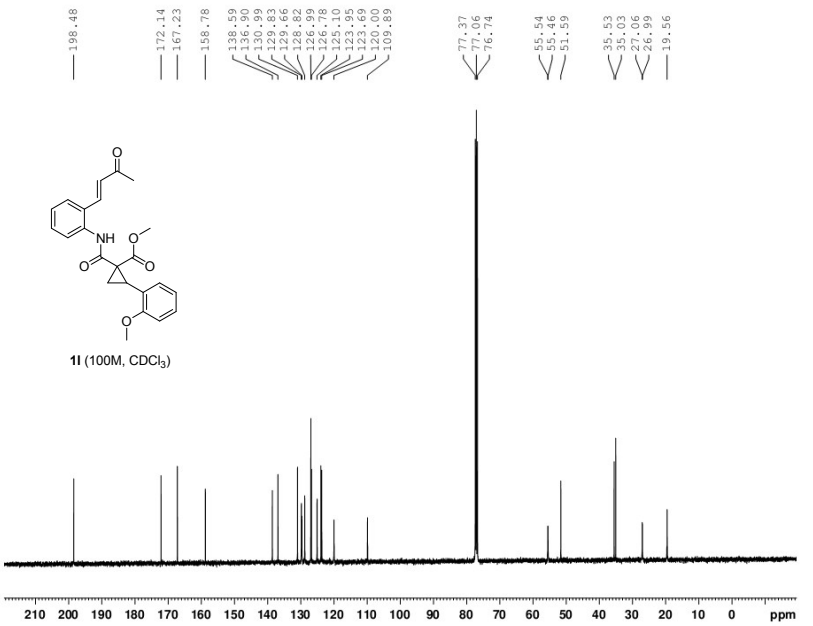
JA-302



1l (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time               10.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

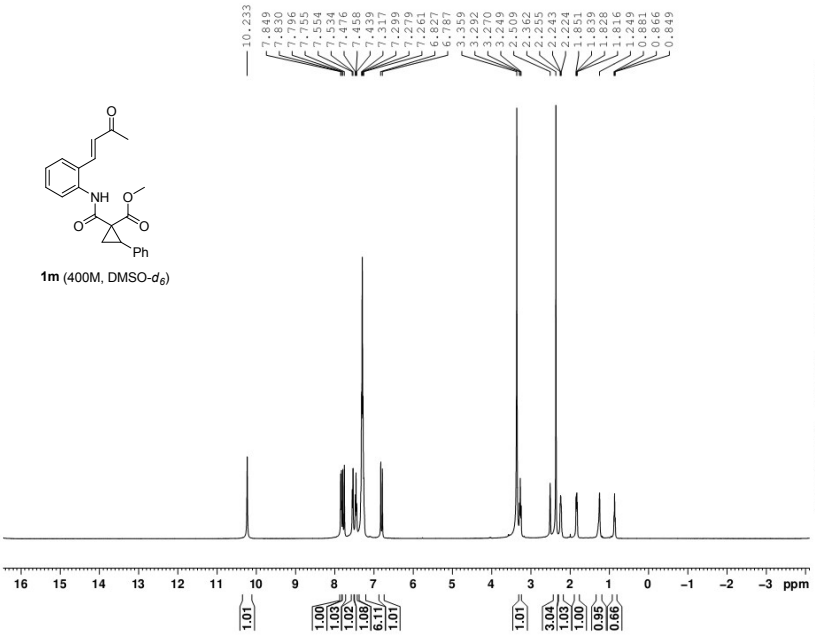
JA-302



1l (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time               16.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   696
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

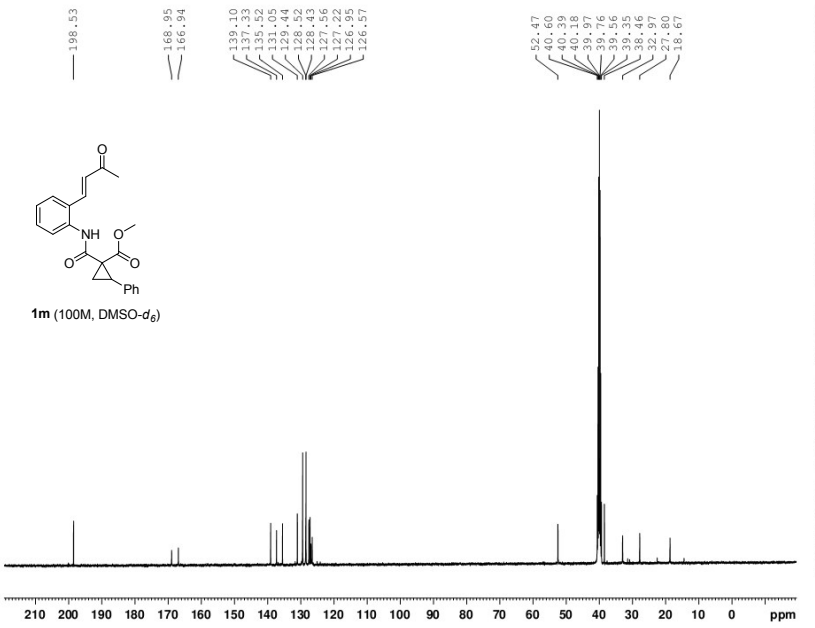
JA-219



1m (400M, DMSO-d_6)

```
Current Data Parameters
NAME          03-09-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150309
Time               13.43
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   16
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 79.3
DW                60.800 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

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

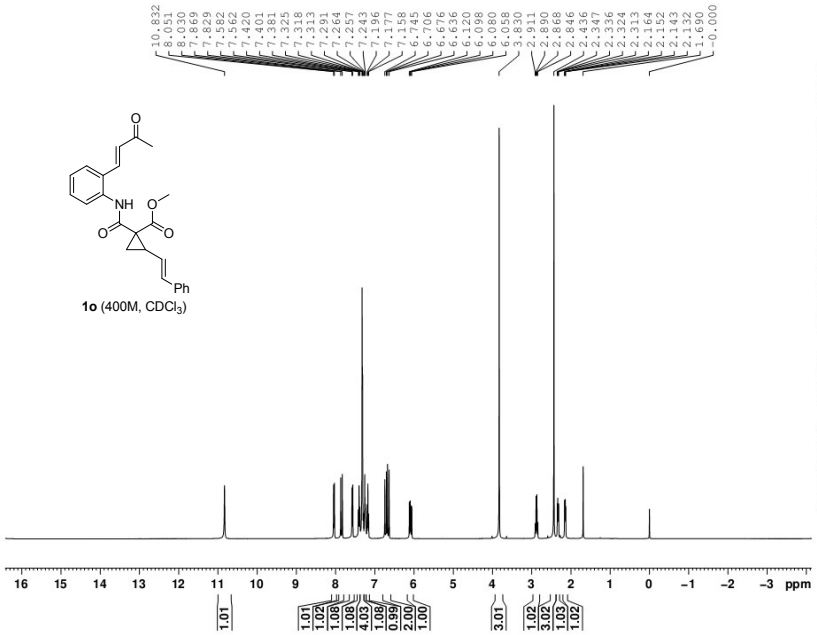
JA-219



1m (100M, DMSO-d_6)

```
Current Data Parameters
NAME          03-09-2015
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150311
Time               12.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                  944
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                152.51
DW                20.800 usec
DE                 6.50 usec
TE                295.2 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

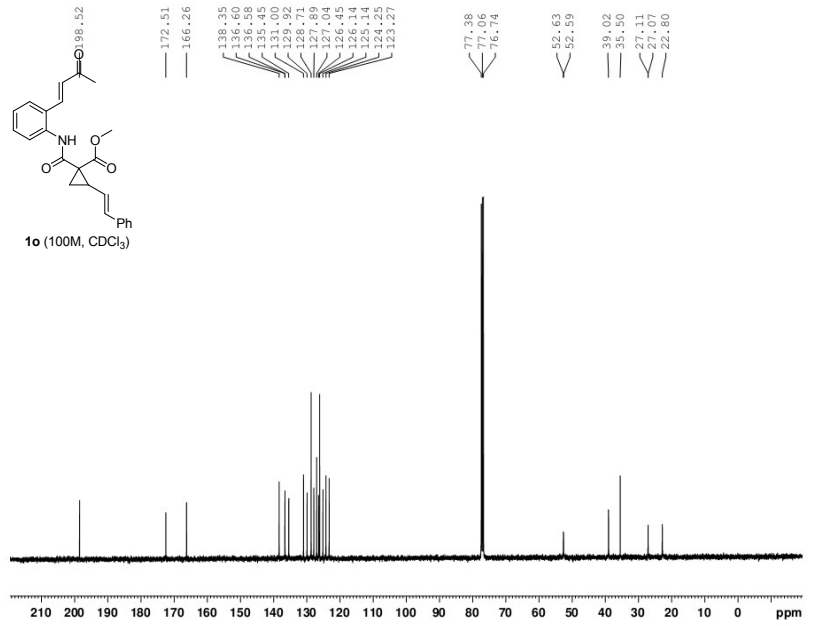
JA-313



1o (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               15.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                106.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300120 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

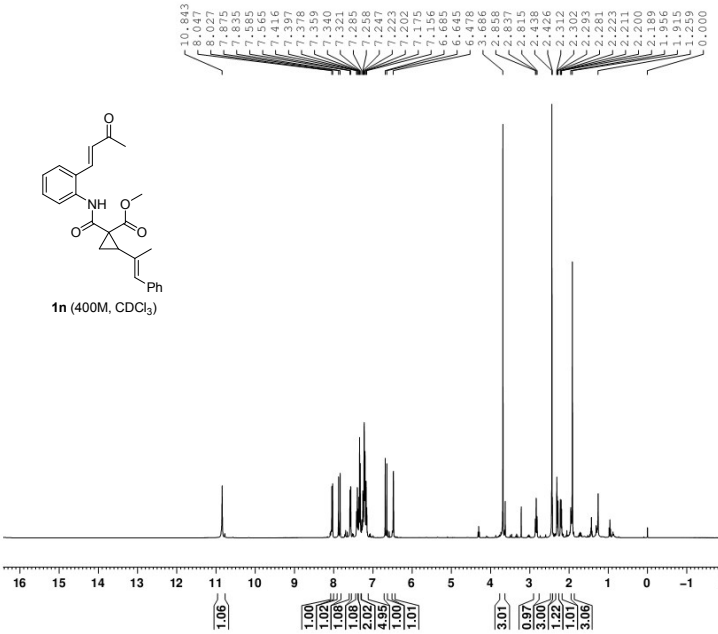
JA-313



1o (100M, $CDCl_3$)

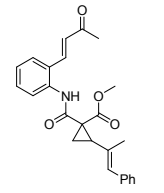
```
Current Data Parameters
NAME          08-20-2015
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               16.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  404
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

JA-297

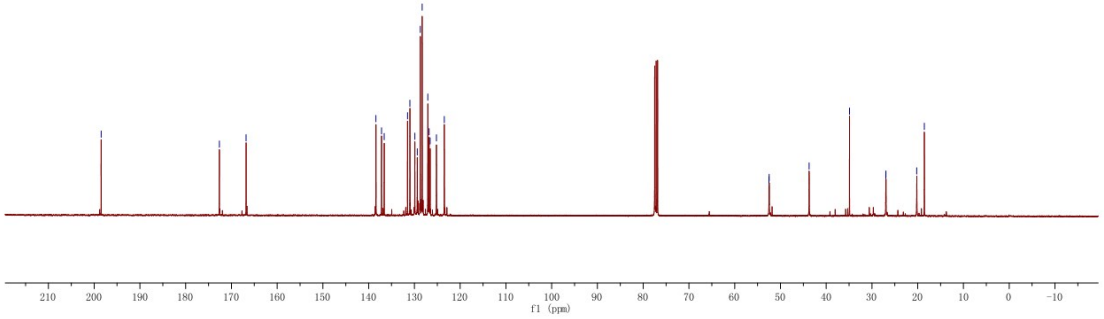


1n (400M, $CDCl_3$)

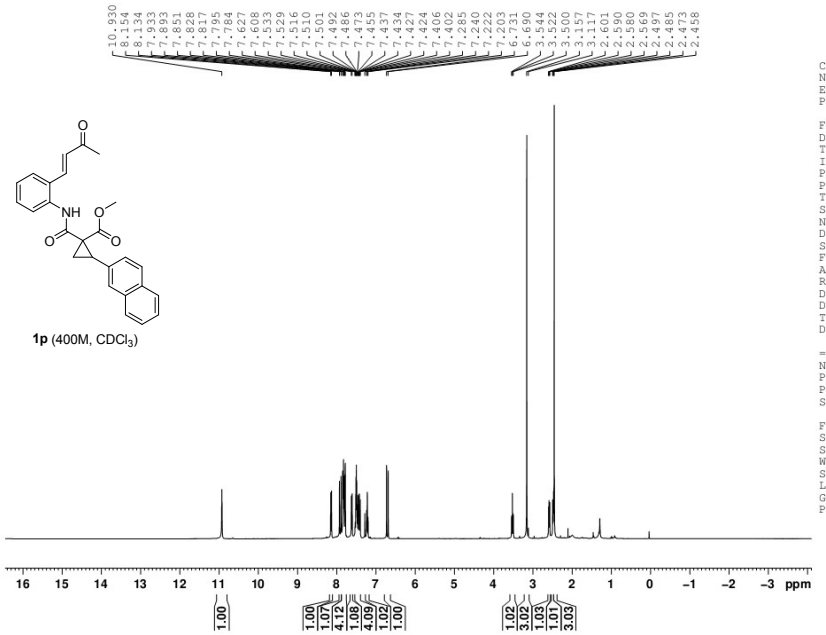
```
Current Data Parameters
NAME          07-30-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150730
Time               11.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                33.09
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300111 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



1n (100M, $CDCl_3$)



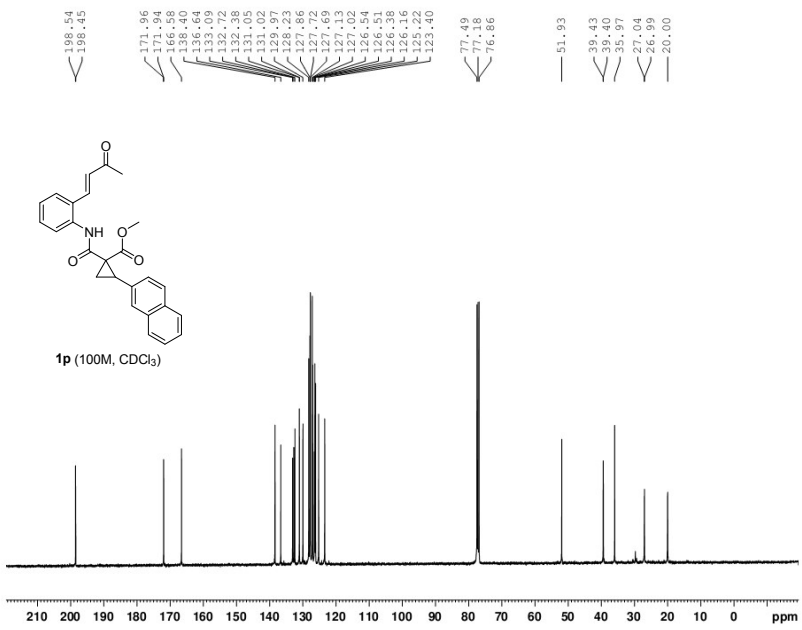
JA-314



1p (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-24-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150824
Time               14.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 33.09
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

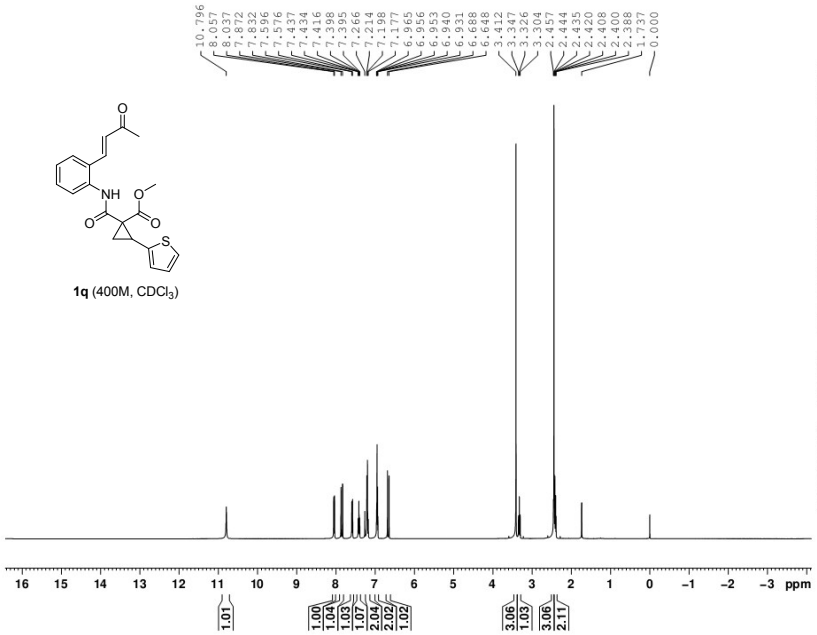
JA-314



1p (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-24-2015
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150825
Time               13.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  2212
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

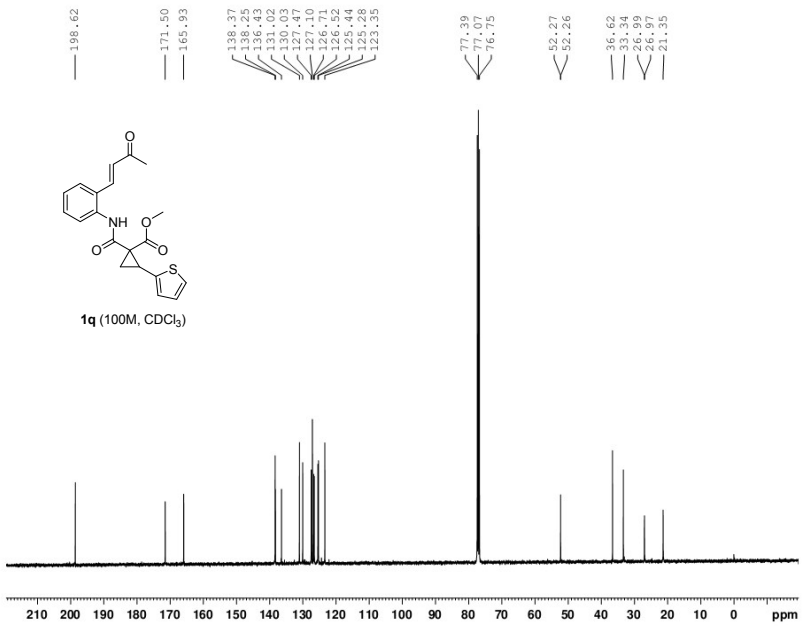
JA-255



1q (400M, $CDCl_3$)

```
Current Data Parameters
NAME          05-25-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150525
Time               10.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    44
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                106.96
DW                60.800 usec
DE                 6.50 usec
TE                295.9 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300080 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

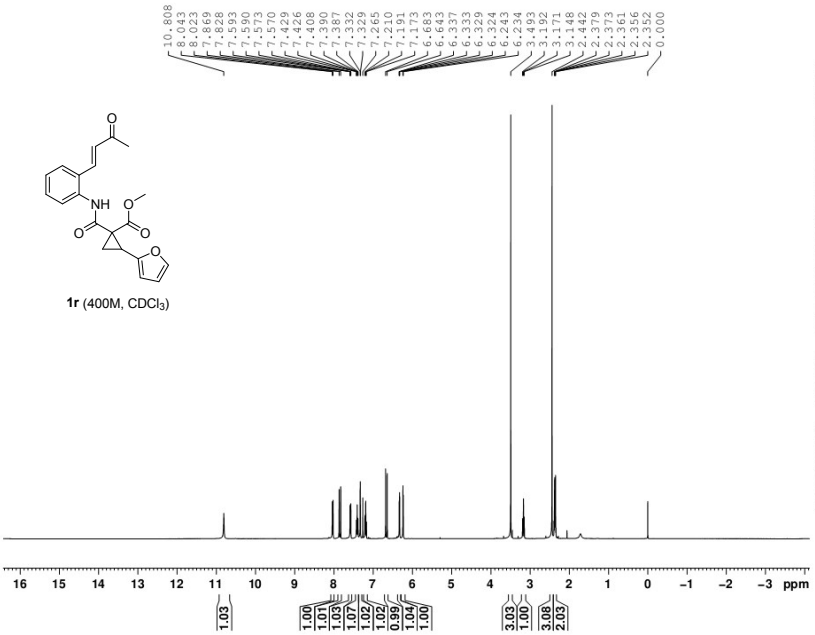
JA-255



1q (100M, $CDCl_3$)

```
Current Data Parameters
NAME          05-25-2015
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150527
Time               12.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1396
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.2 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

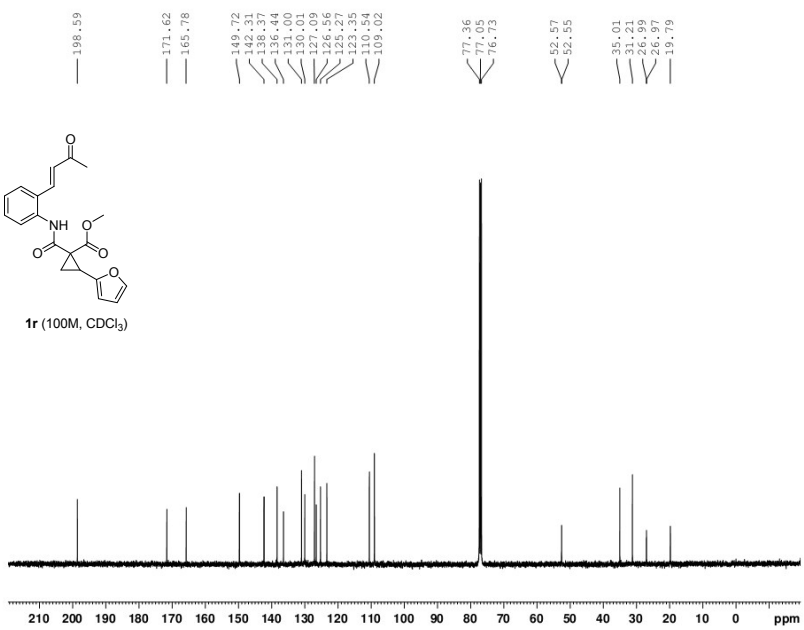
JA-310



1r (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               14.50
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ             3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300080 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

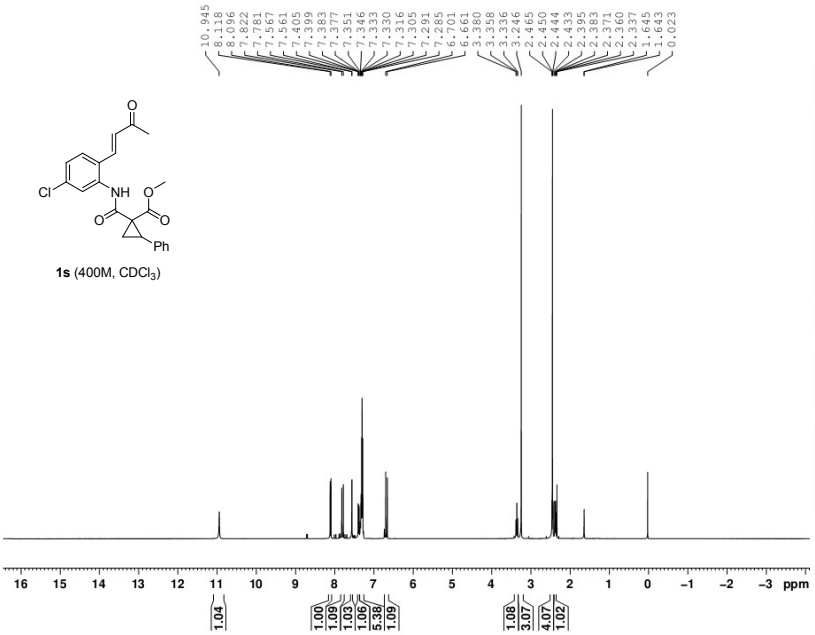
JA-310



1r (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150821
Time               10.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  552
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ             1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1           2.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

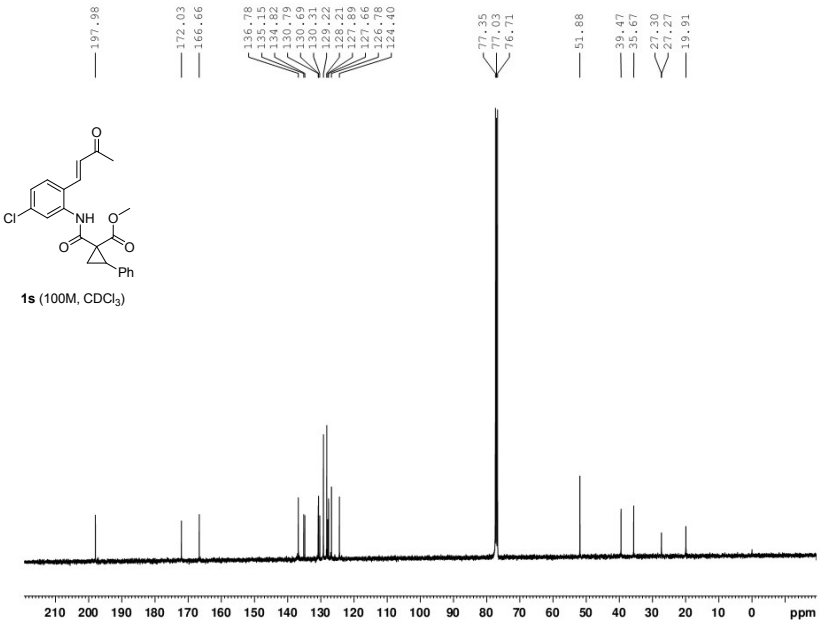
JA-305



1s (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150804
Time               10.35
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    40
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                152.51
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

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

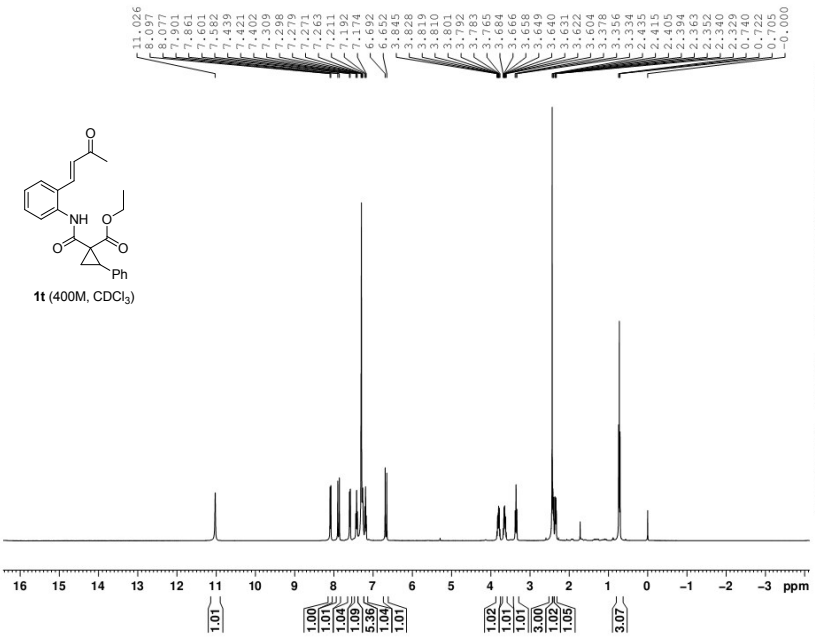
JA-305



1s (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-04-2015
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150805
Time               11.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  1020
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127690 MHz
WDW                      EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

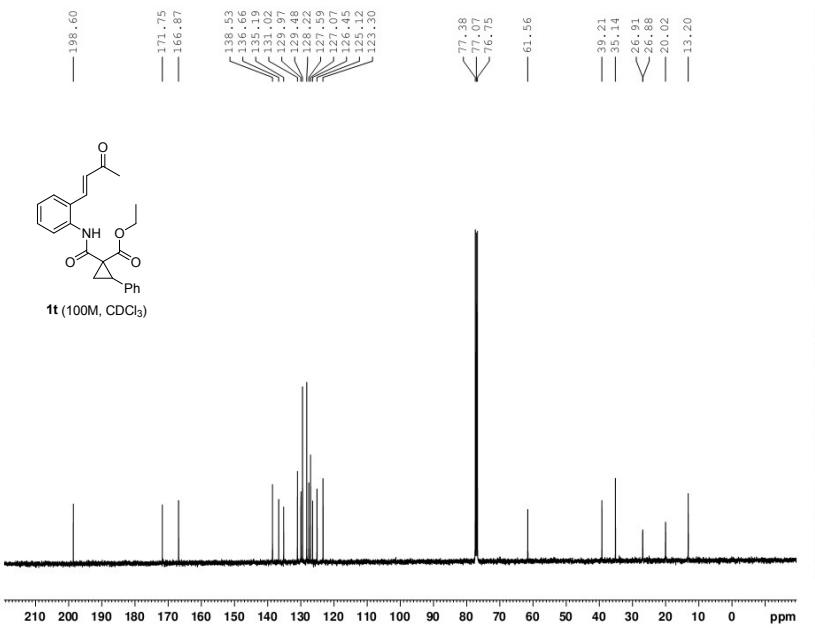
JA-264



1t (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-11-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150611
Time               10.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    36
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                295.8 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

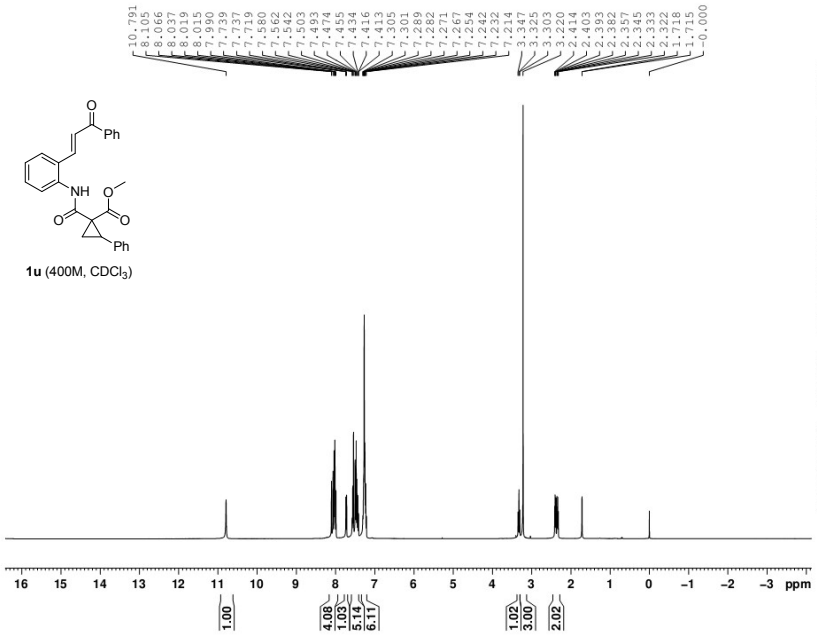
JA-264



1t (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-11-2015
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150611
Time               14.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   328
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                297.0 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

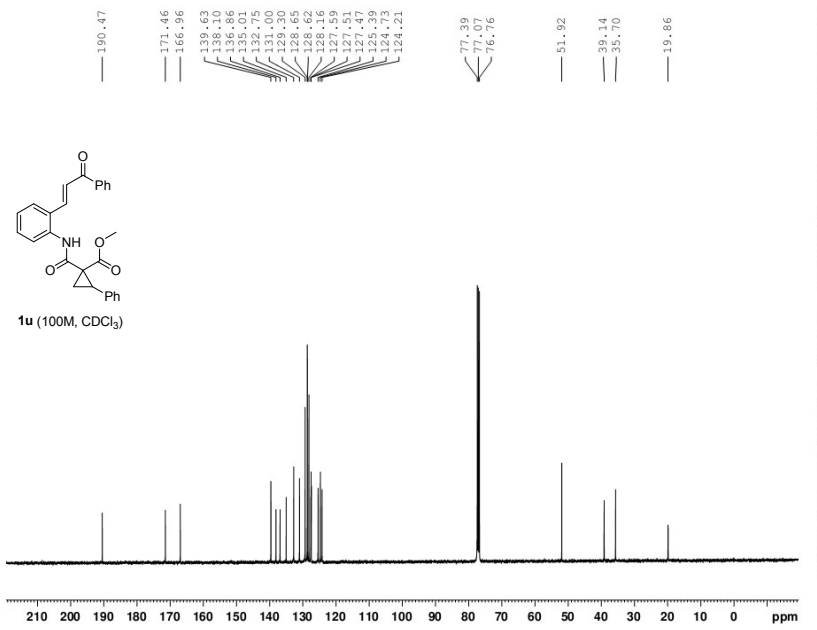
JA-311



1u (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               15.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    80
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300123 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

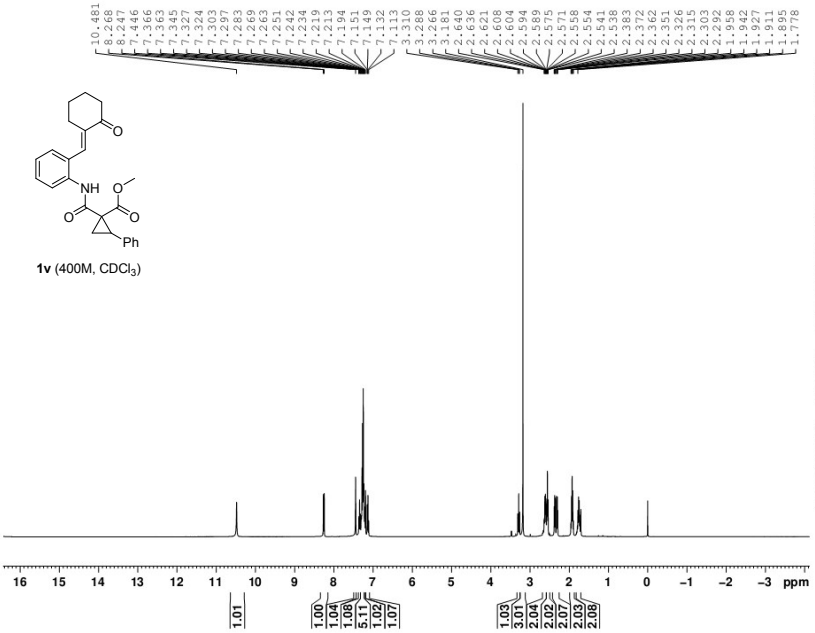
JA-311



1u (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150821
Time               10.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   940
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

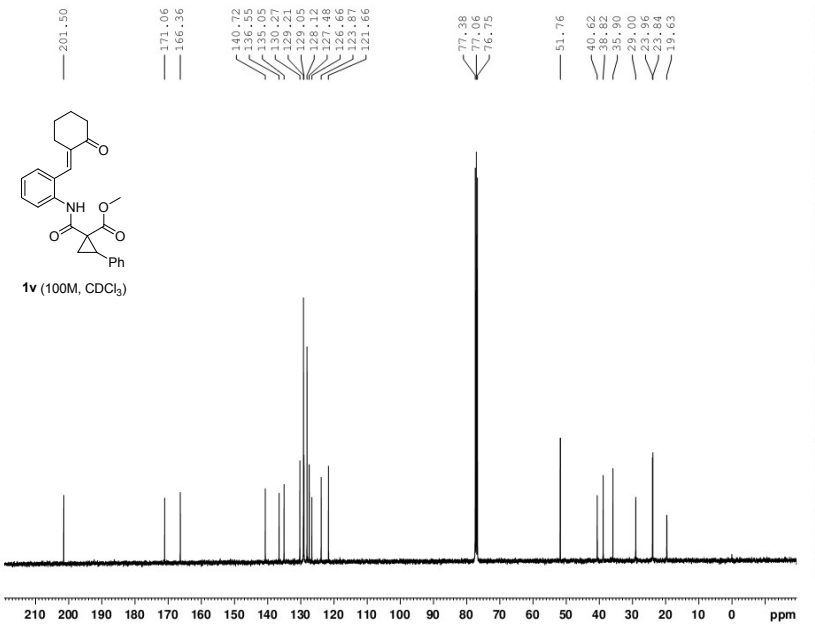
JA-266



1v (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-11-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150611
Time               11.12
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    68
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                295.8 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

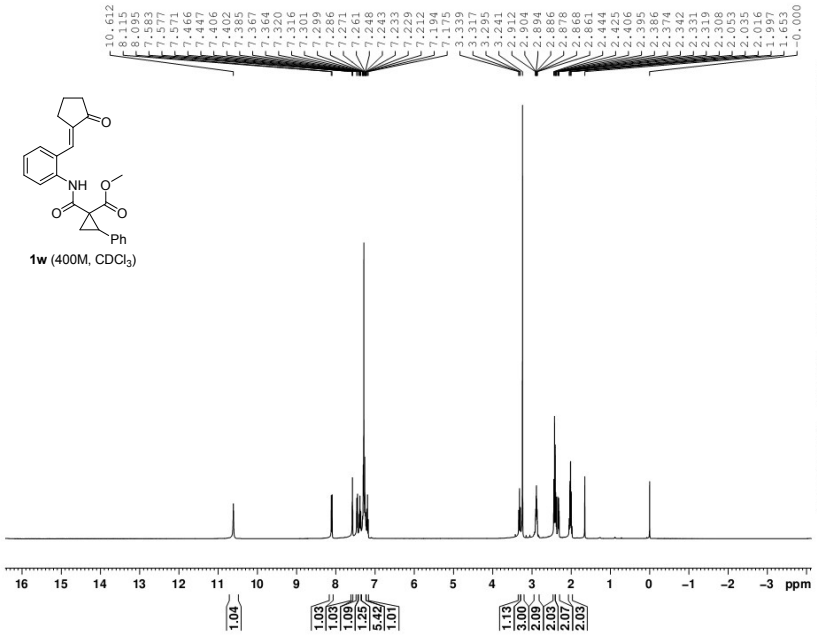
JA-266



1v (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-11-2015
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150611
Time               16.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   608
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.8 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

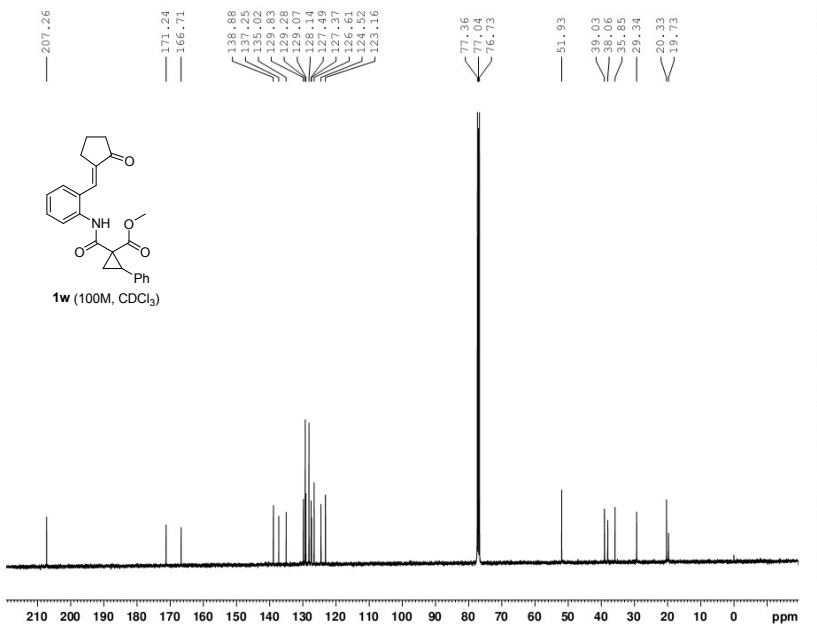
JA-272



1w (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-23-2015
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150623
Time               14.26
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE        0 K
D1          1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300096 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

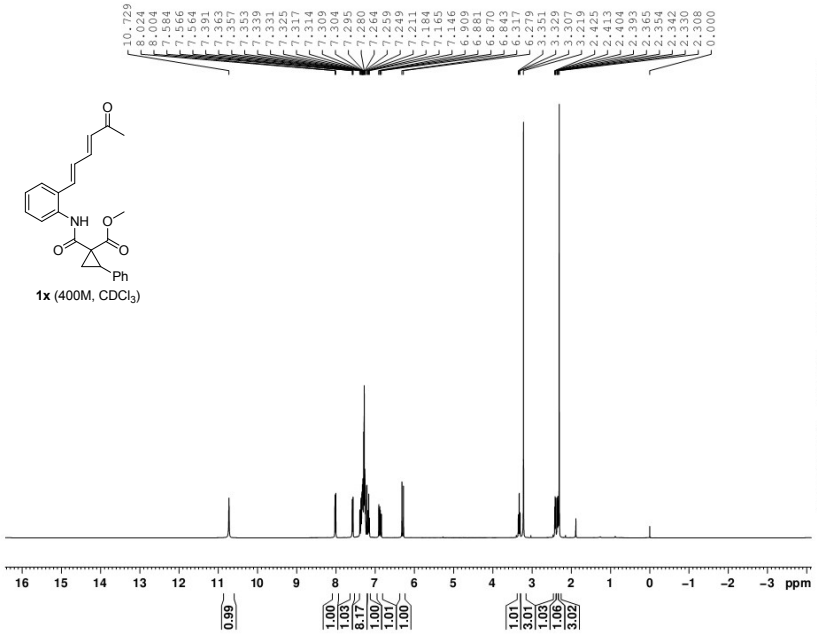
JA-272



1w (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-23-2015
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150625
Time                9.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1136
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE        0 K
D1          2.00000000 sec
D11         0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

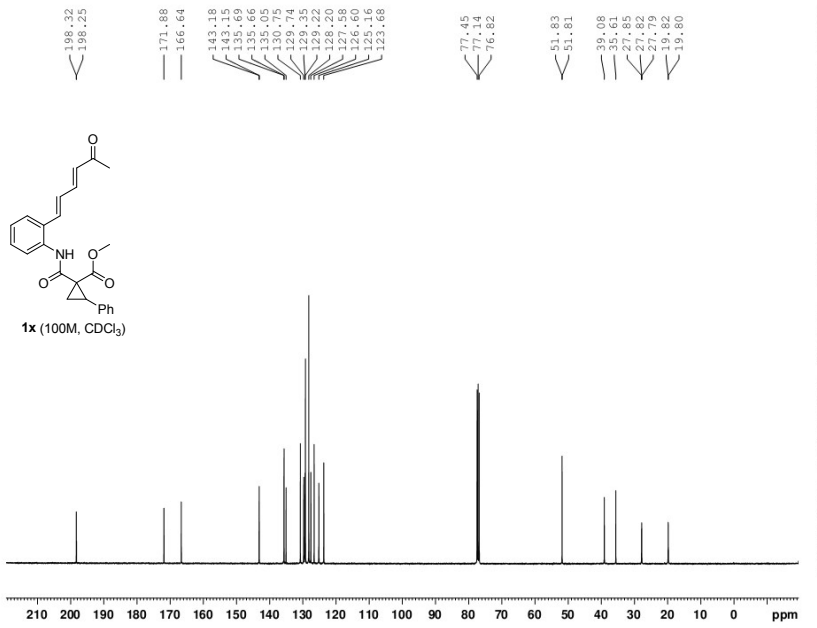
JA-312



1x (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               15.16
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    80
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 55.68
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300083 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

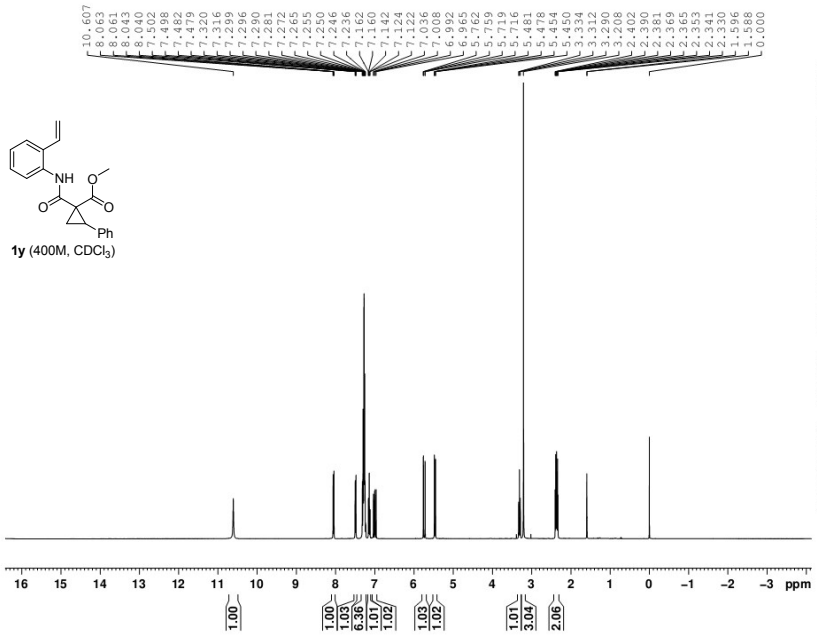
JA-312



1x (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               18.18
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  2048
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

JA-277

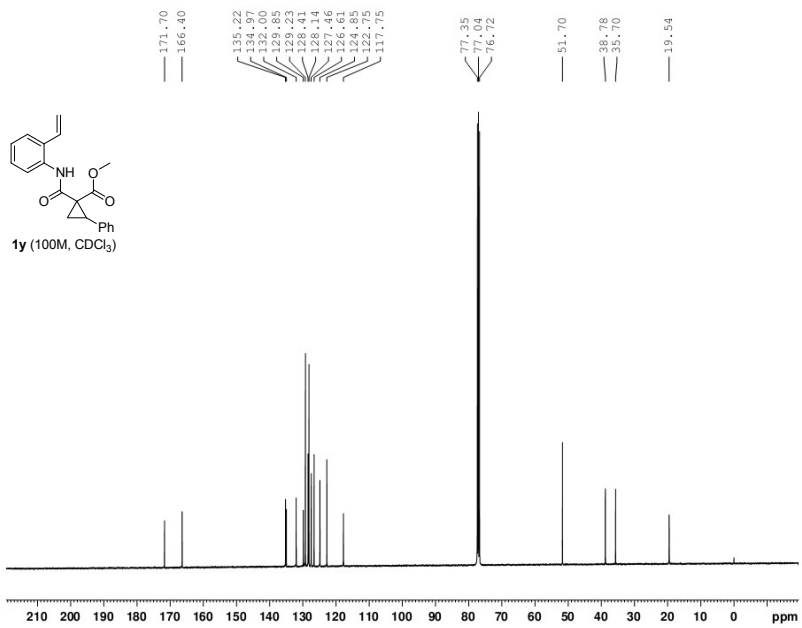


1y (400M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          07-01-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150701
Time               15.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    64
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300123 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

JA-277

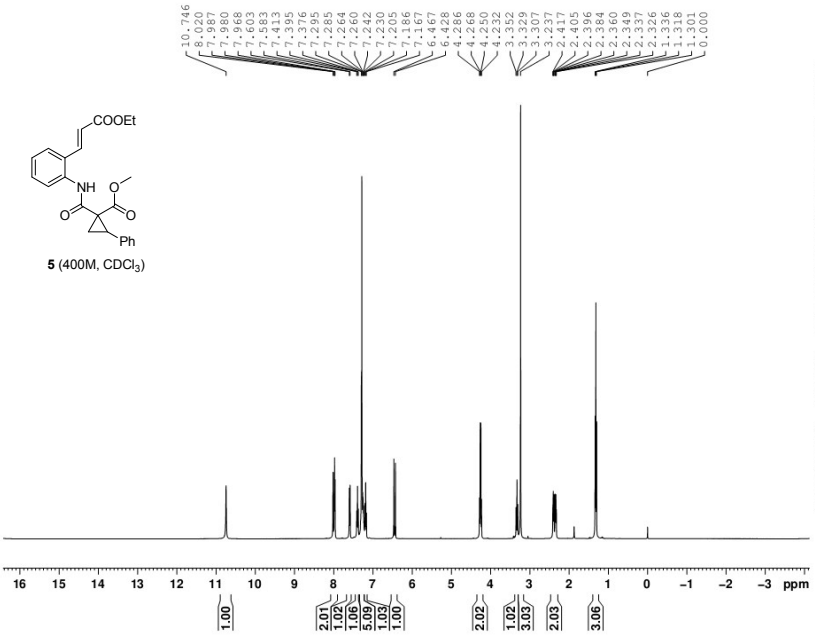


1y (100M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          07-01-2015
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150707
Time               1.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                  8600
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

JA-269

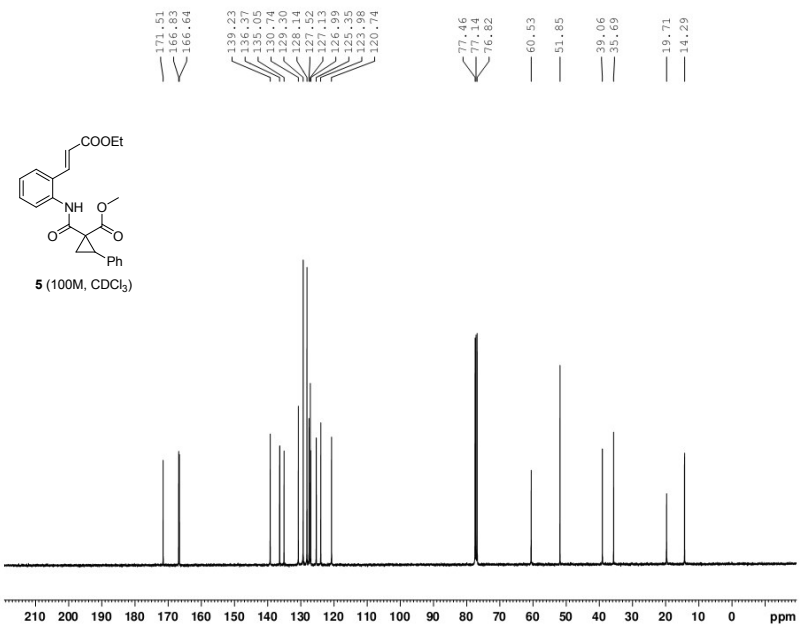


5 (400M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          06-23-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150623
Time               13.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   56
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                52.15
DW                60.800 usec
DE                 6.50 usec
TE        0 K
D1          1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF          400.1300104 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

JA-269

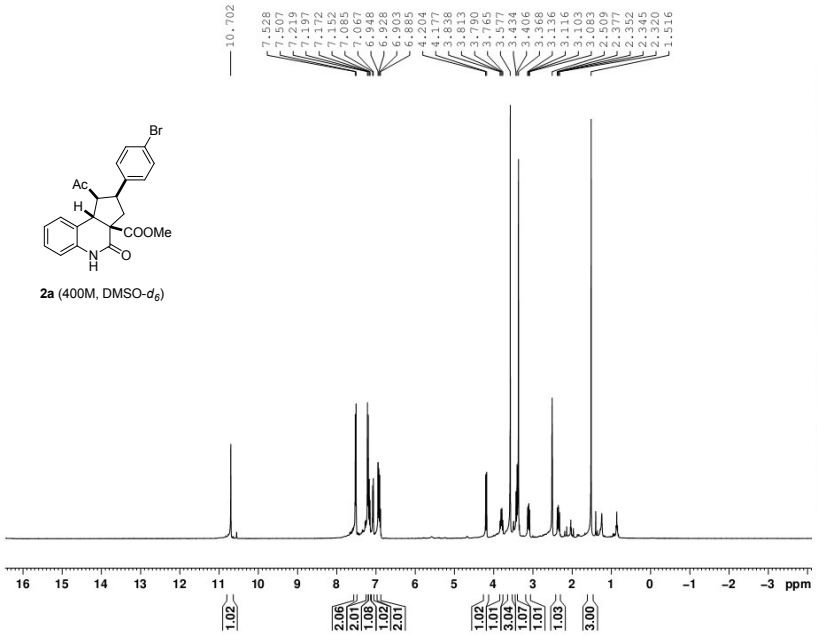


5 (100M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          06-23-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150624
Time               14.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  736
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE        0 K
D1          2.00000000 sec
D11         0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF         100.6127690 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

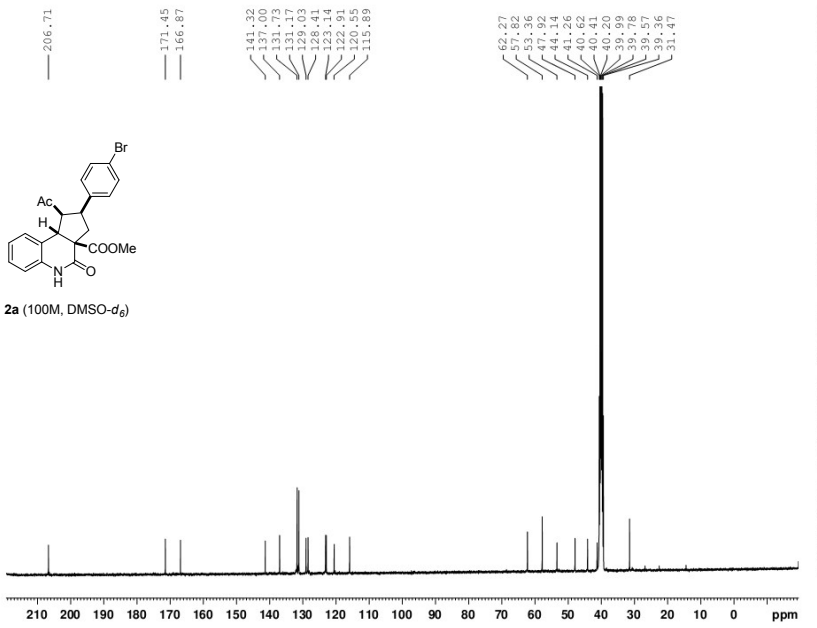
JA-233



2a (400M, DMSO-d_6)

```
Current Data Parameters
NAME          04-07-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150407
Time               13.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   48
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE                291.4 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

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

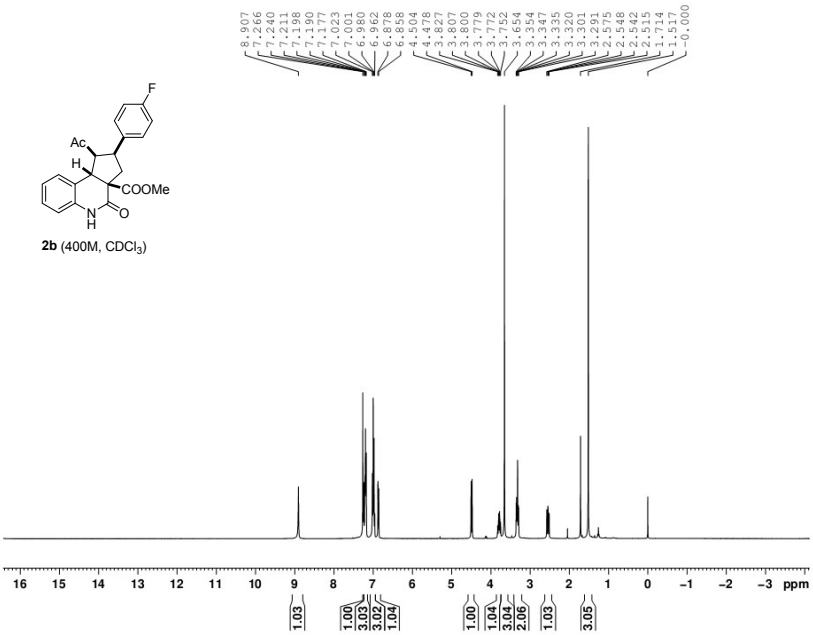
JA-233



2a (100M, DMSO-d_6)

```
Current Data Parameters
NAME          04-07-2015
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150411
Time               1.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 8600
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                106.96
DW                20.800 usec
DE                 6.50 usec
TE                294.5 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

JA-298



2b (400M, $CDCl_3$)

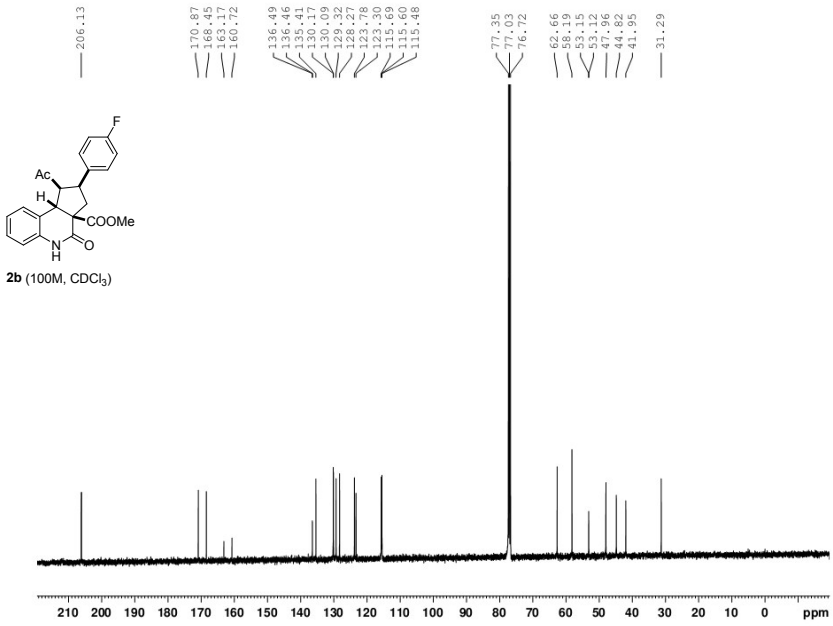
Current Data Parameters
NAME 07-30-2015
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150730
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 138.14
DW 60.800 usec
DE 6.50 usec
TE 518.1 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300080 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

JA-298



2b (100M, $CDCl_3$)

Current Data Parameters
NAME 07-30-2015
EXPNO 11
PROCNO 1

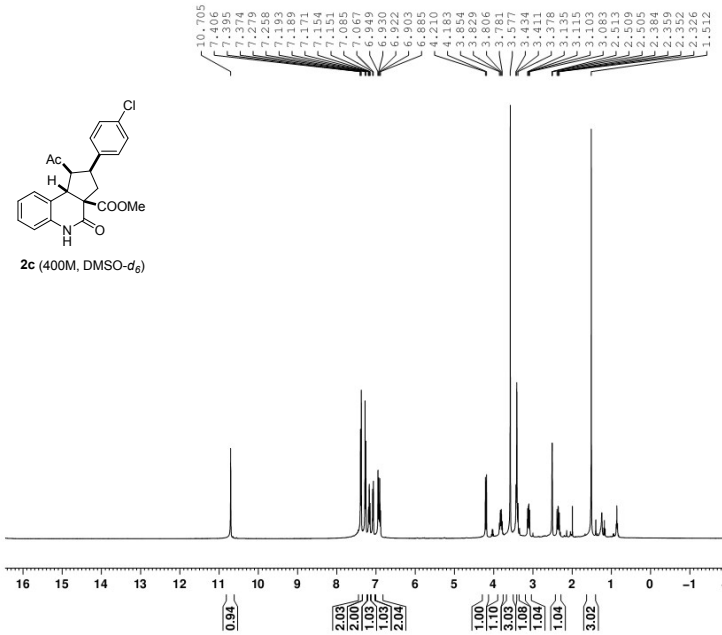
F2 - Acquisition Parameters
Date_ 20150731
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 900
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 518.1 K
D1 2.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

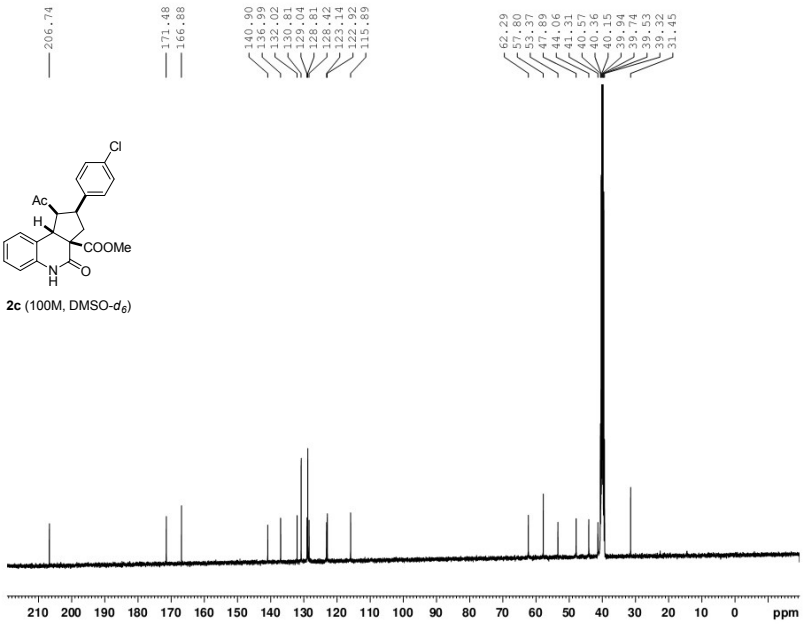
JA-235



2c (400M, DMSO-d_6)

```
Current Data Parameters
NAME          04-07-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150407
Time               13.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   48
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                124.55
DW                60.800 usec
DE                 6.50 usec
TE                291.2 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

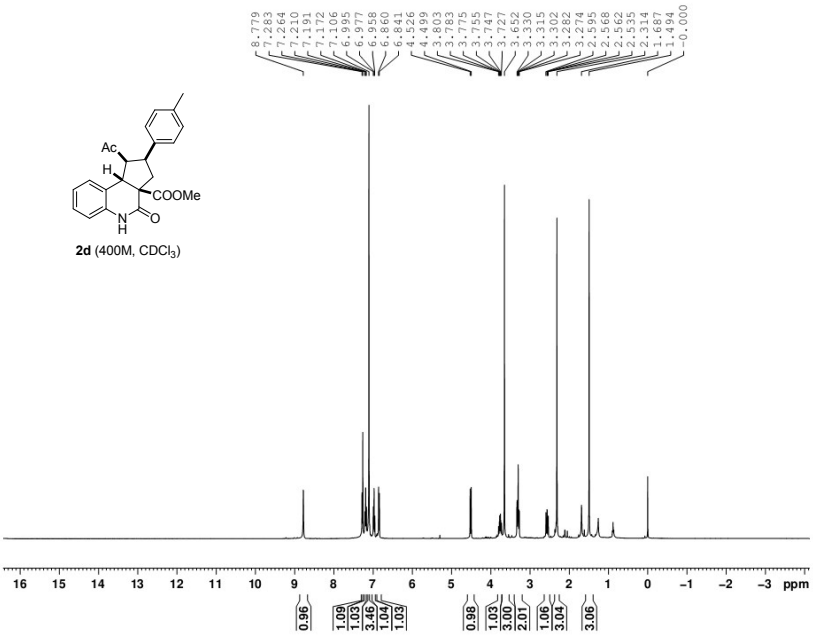
JA-235



2c (100M, DMSO-d_6)

```
Current Data Parameters
NAME          04-07-2015
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150414
Time               11.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 1672
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                138.14
DW                20.800 usec
DE                 6.50 usec
TE                294.2 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

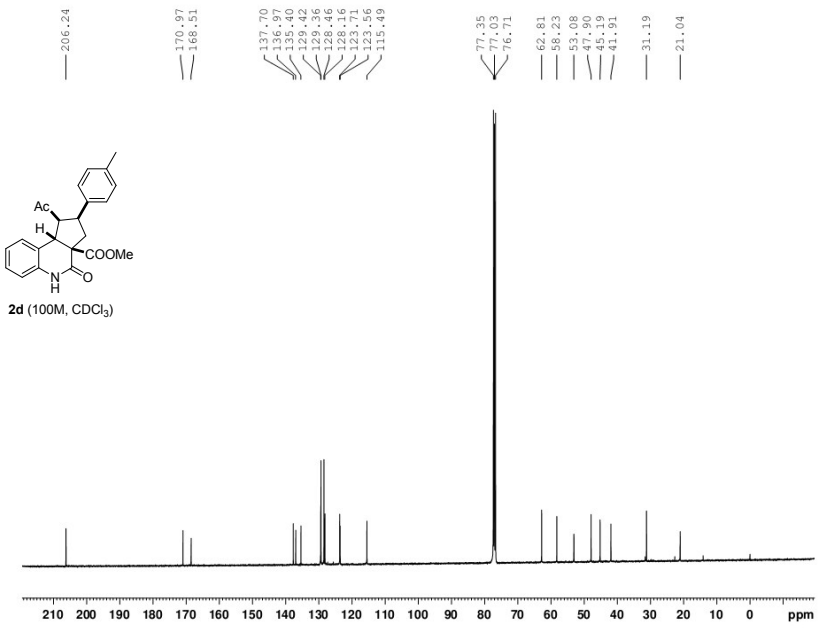
JA-240



2d (400M, $CDCl_3$)

```
Current Data Parameters
NAME          05-04-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150504
Time               13.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    64
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE                294.4 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF           400.1300089 MHz
WDW                      EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

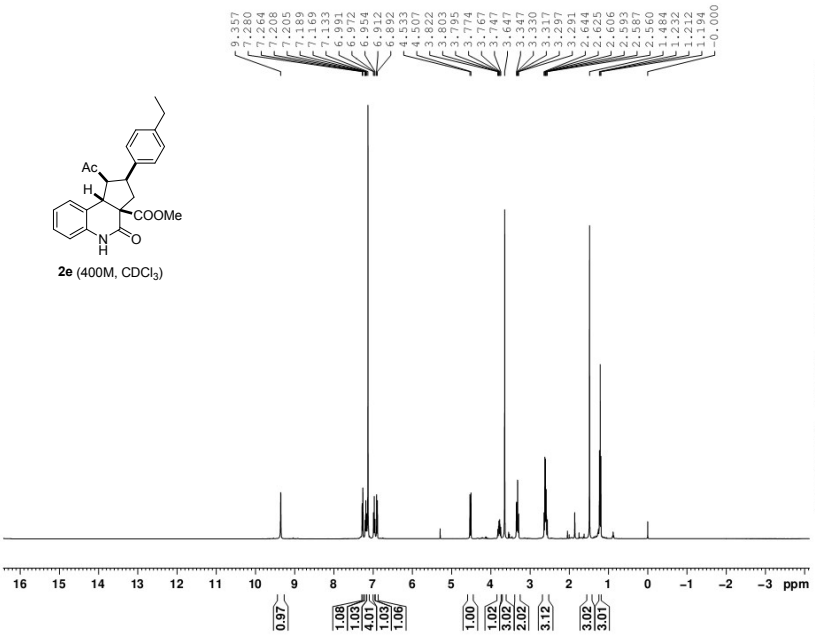
JA-240



2d (100M, $CDCl_3$)

```
Current Data Parameters
NAME          05-04-2015
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150506
Time                2.38
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  9800
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                168.03
DW                20.800 usec
DE                 6.50 usec
TE                296.5 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127690 MHz
WDW                      EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

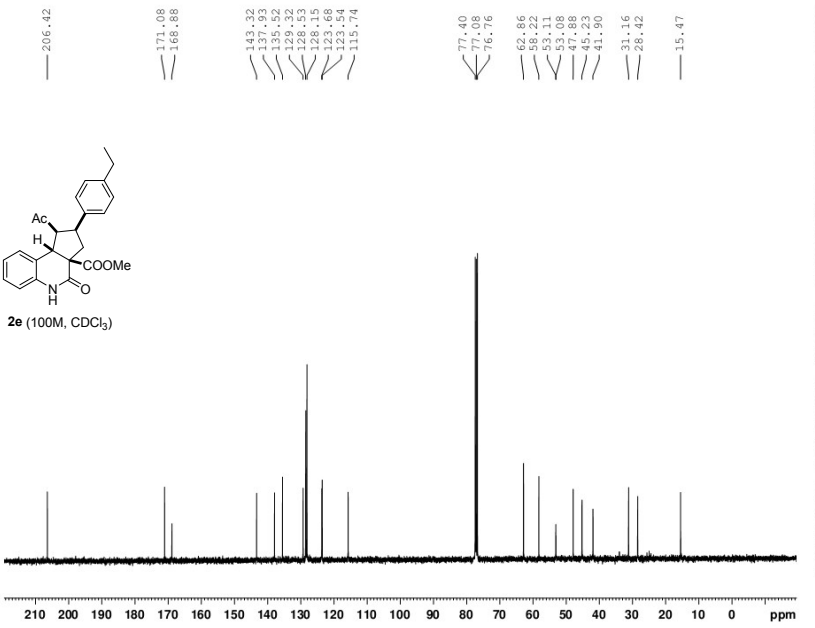
JA-260



2e (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-01-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150601
Time               13.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    64
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                  79.3
DW                60.800 usec
DE                 6.50 usec
TE                296.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300085 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

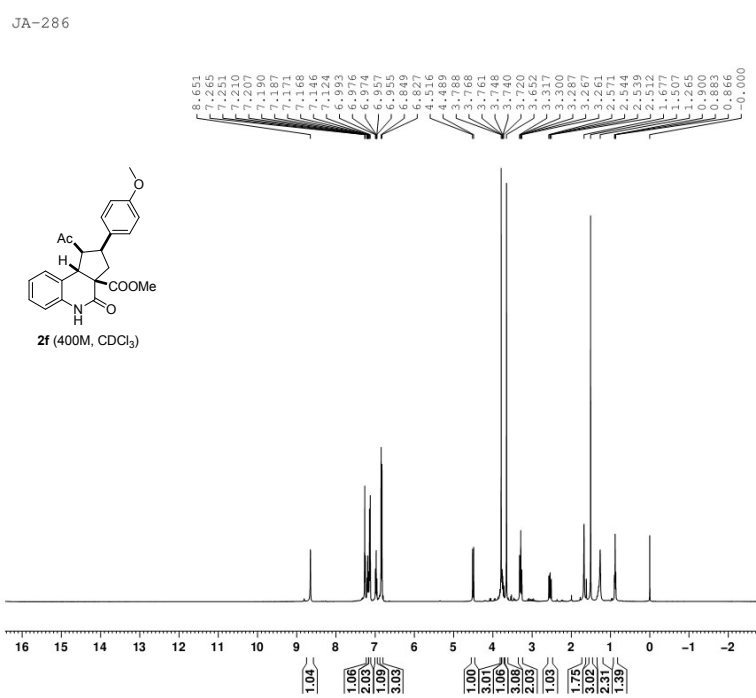
JA-260



2e (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-01-2015
EXPNO                 17
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150609
Time               10.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDC13
NS                   200
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                 198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.4 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

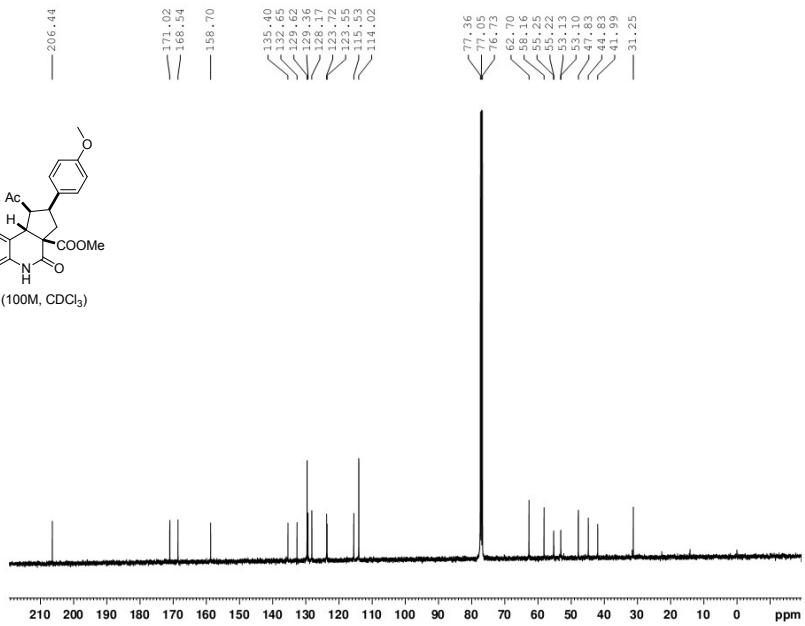
JA-286



2f (400M, $CDCl_3$)

```
Current Data Parameters
NAME          07-14-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150714
Time               10.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDCl3
NS                    64
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                152.51
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300086 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

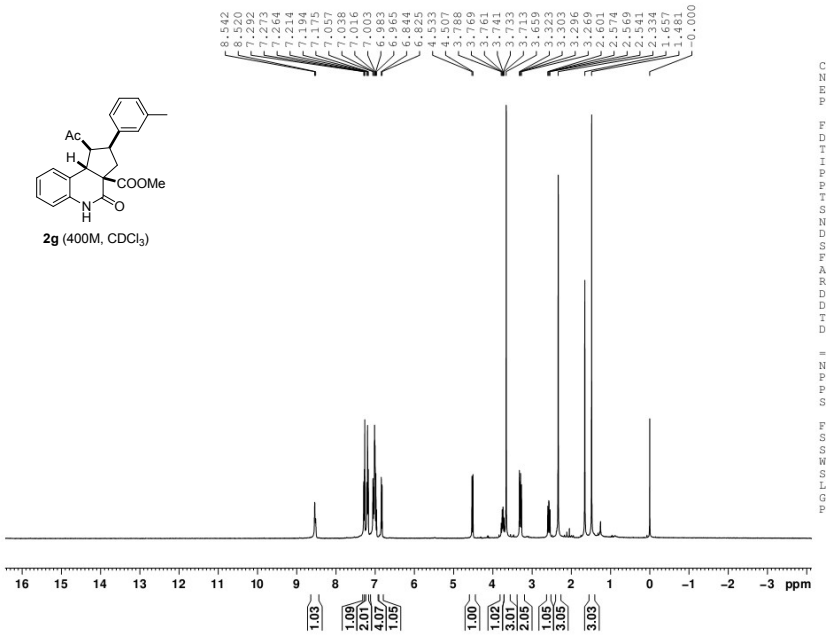
JA-249



2f (100M, $CDCl_3$)

```
Current Data Parameters
NAME          05-14-2015
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150515
Time               13.05
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                  1056
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                168.03
DW                20.800 usec
DE                 6.50 usec
TE                295.5 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

JA-244



2g (400M, $CDCl_3$)

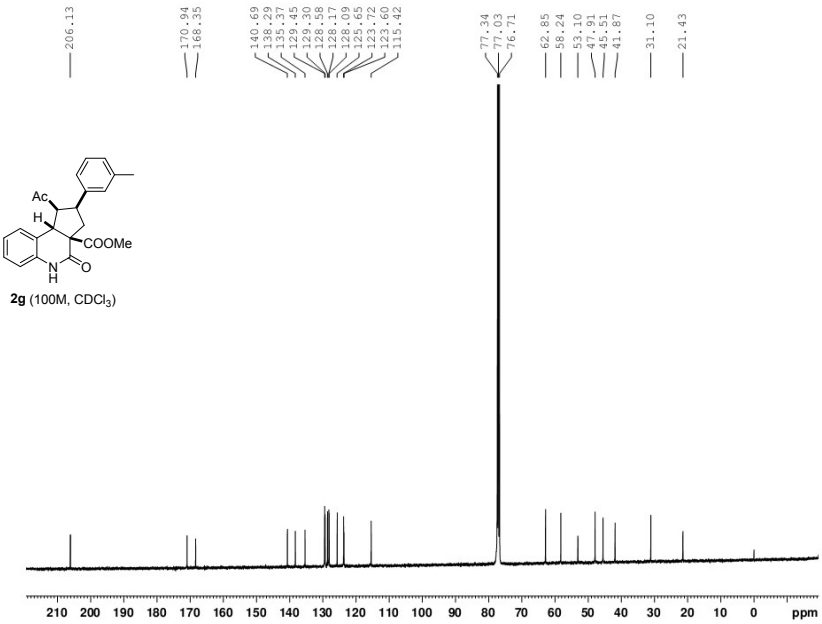
Current Data Parameters
NAME 05-04-2015
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150504
Time 14.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 168.03
DW 60.800 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

JA-244



2g (100M, $CDCl_3$)

Current Data Parameters
NAME 05-04-2015
EXPNO 8
PROCNO 1

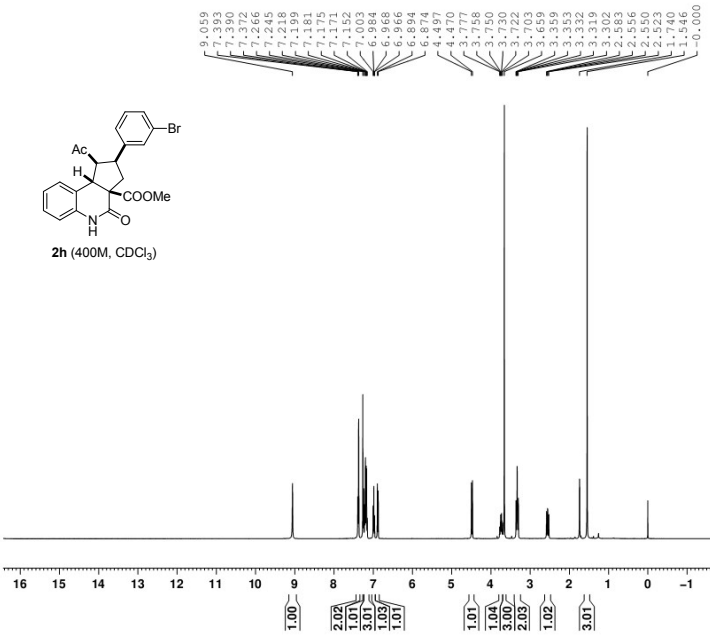
F2 - Acquisition Parameters
Date_ 20150506
Time 23.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 9800
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 168.03
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

JA-289

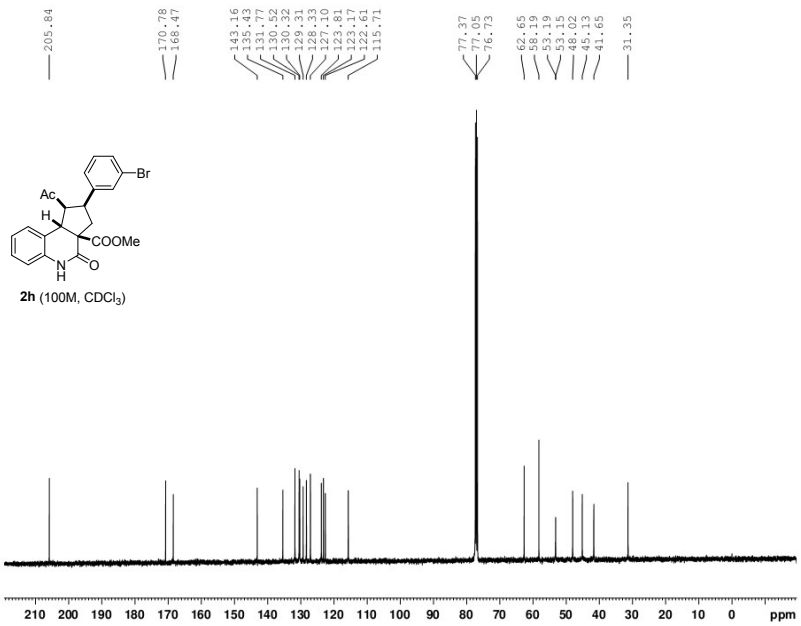


2h (400M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          07-24-2015
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150724
Time               16.09
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    26
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                124.55
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF           400.1300077 MHz
WDW                      EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

JA-289

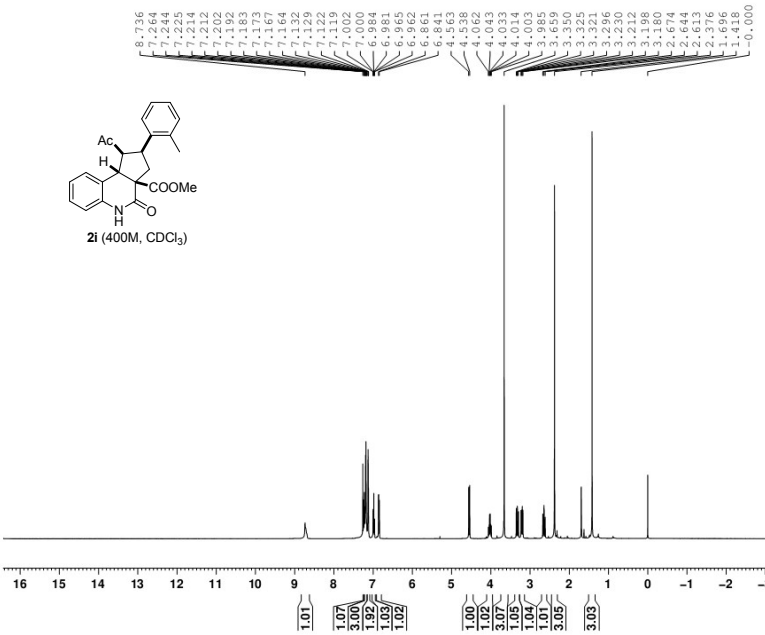


2h (100M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          07-24-2015
EXPNO                 15
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150727
Time               15.31
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   816
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127690 MHz
WDW                      EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

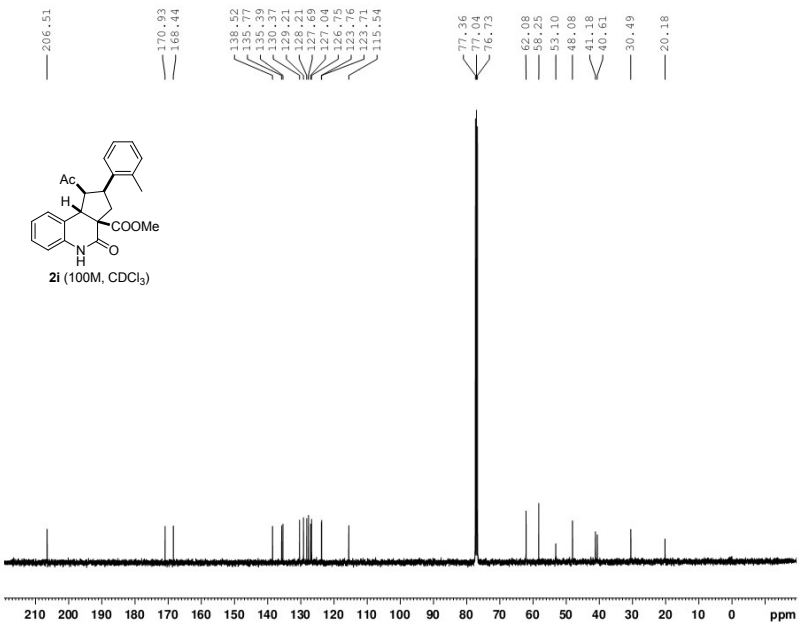
JA-253



2i (400M, $CDCl_3$)

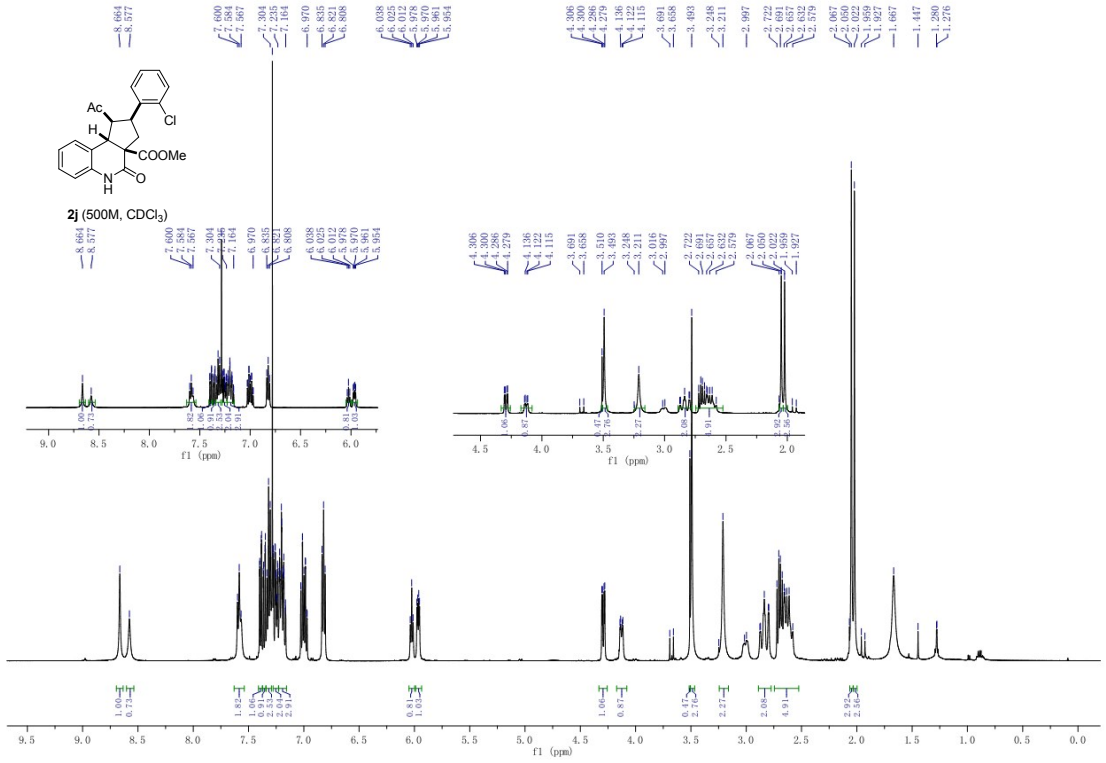
```
Current Data Parameters
NAME          05-19-2015
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150519
Time               14.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    32
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE                295.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300088 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

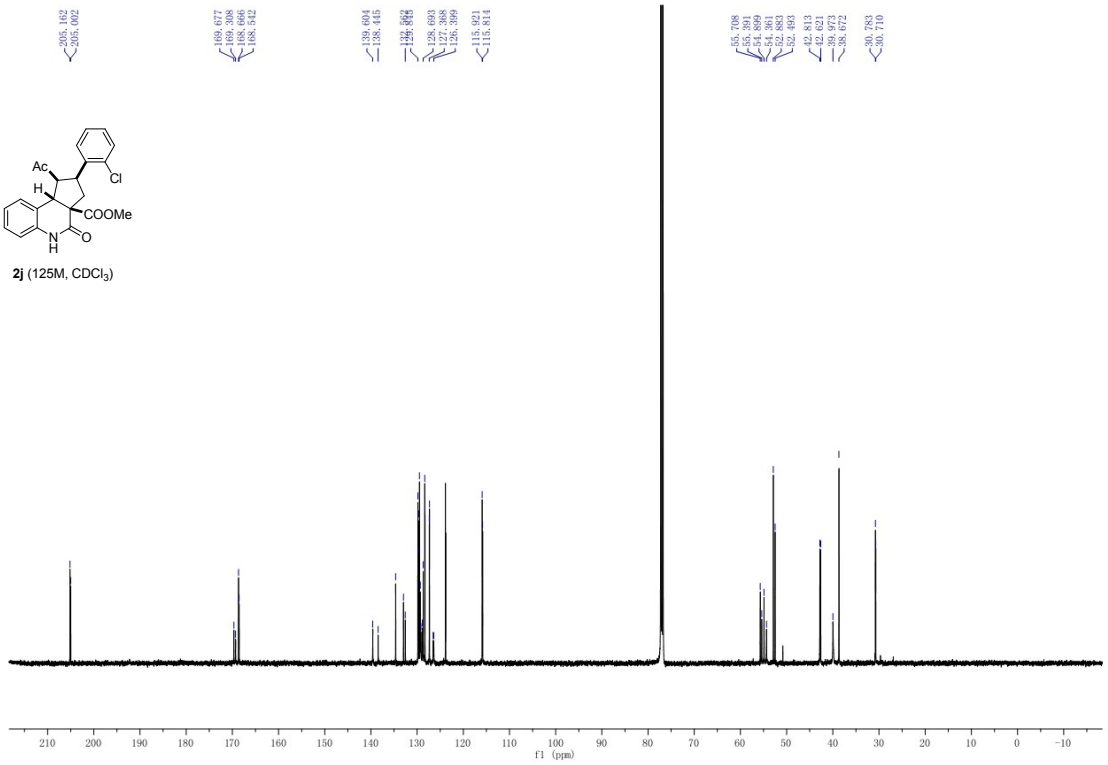
JA-253



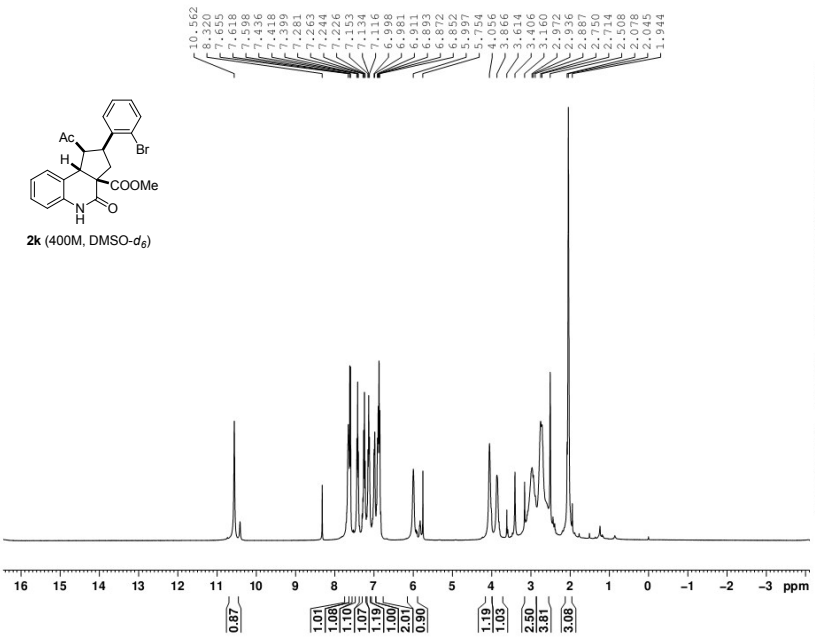
2i (100M, $CDCl_3$)

```
Current Data Parameters
NAME          05-19-2015
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150522
Time               14.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   360
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.3 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```


Ac
H
Cl
COOMe
N
H
O
2j (500M, CDCl3)
f1 (ppm)


Ac
H
Cl
COOMe
N
H
O
2j (125M, CDCl3)
f1 (ppm)

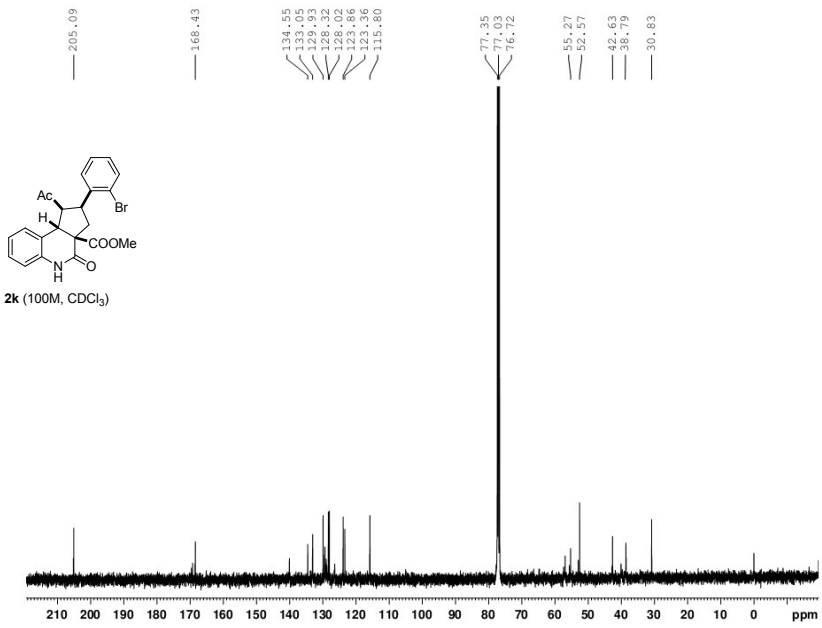
JA-299



2k (400M, DMSO-d_6)

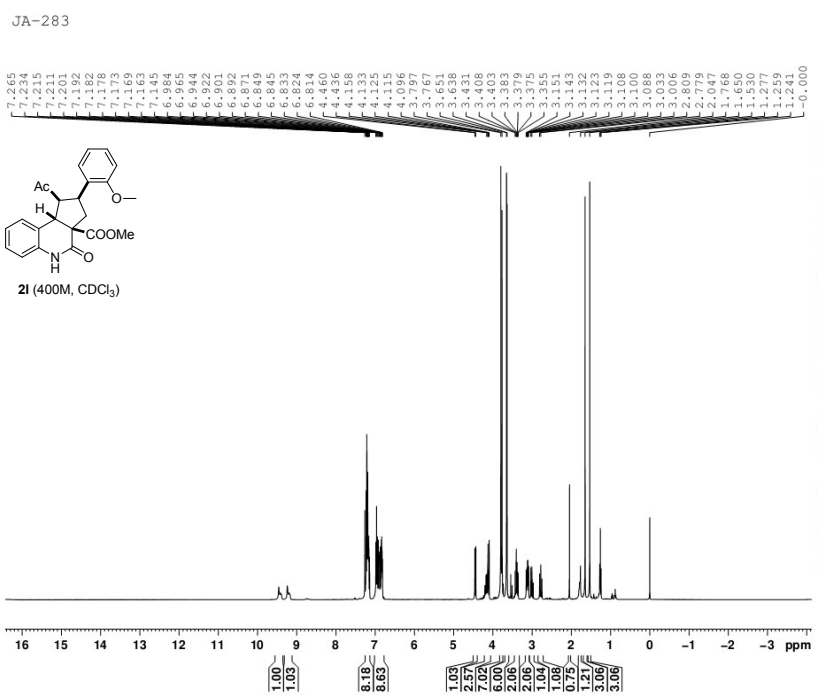
```
Current Data Parameters
NAME          07-30-2015
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150730
Time              11.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT           DMSO
NS                  68
DS                   2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                94.96
DW               60.800 usec
DE                6.50 usec
TE               518.1 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

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

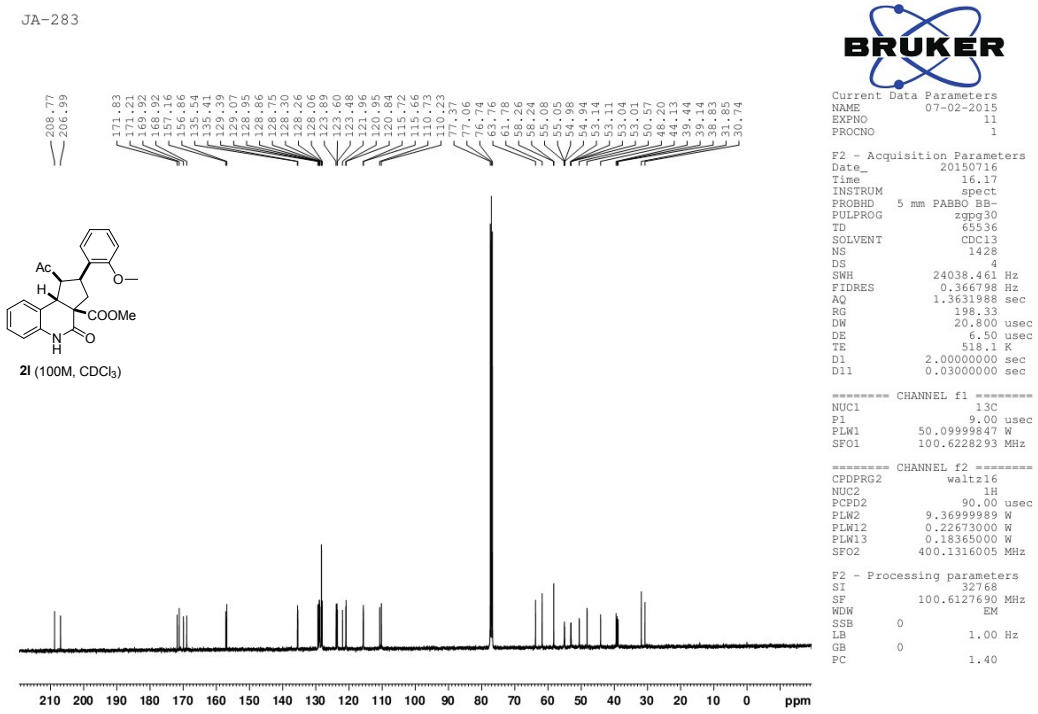
JA-254



2k (100M, $CDCl_3$)

```
Current Data Parameters
NAME          05-19-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150522
Time              15.35
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD               65536
SOLVENT           CDC13
NS                1700
DS                   4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                198.33
DW               20.800 usec
DE                6.50 usec
TE               296.3 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```





Current Data Parameters
NAME 07-02-2015
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150702
Time 14.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 106.96
DW 60.800 usec
DE 6.50 usec
TE 518.1 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME 07-02-2015
EXPNO 11
PROCNO 1

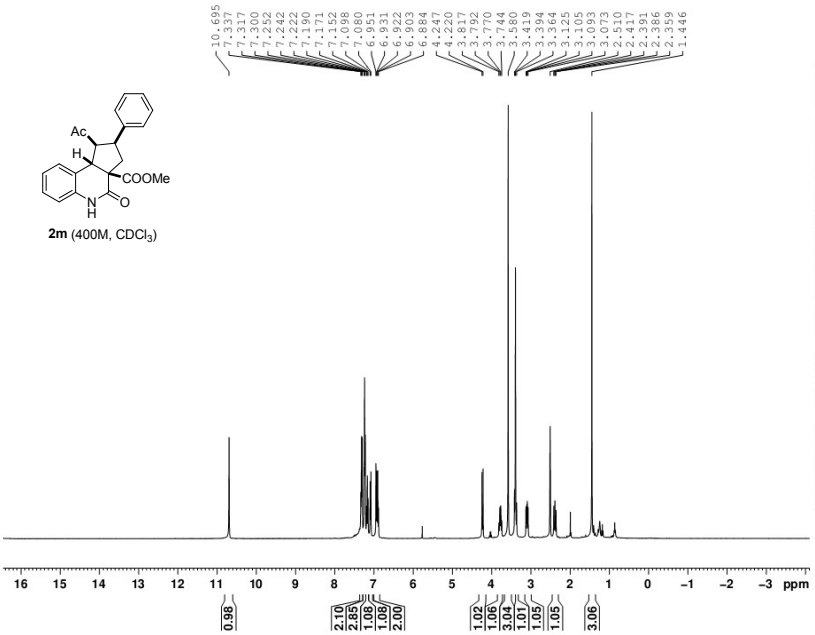
F2 - Acquisition Parameters
Date_ 20150716
Time 16.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1428
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 518.1 K
D1 2.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

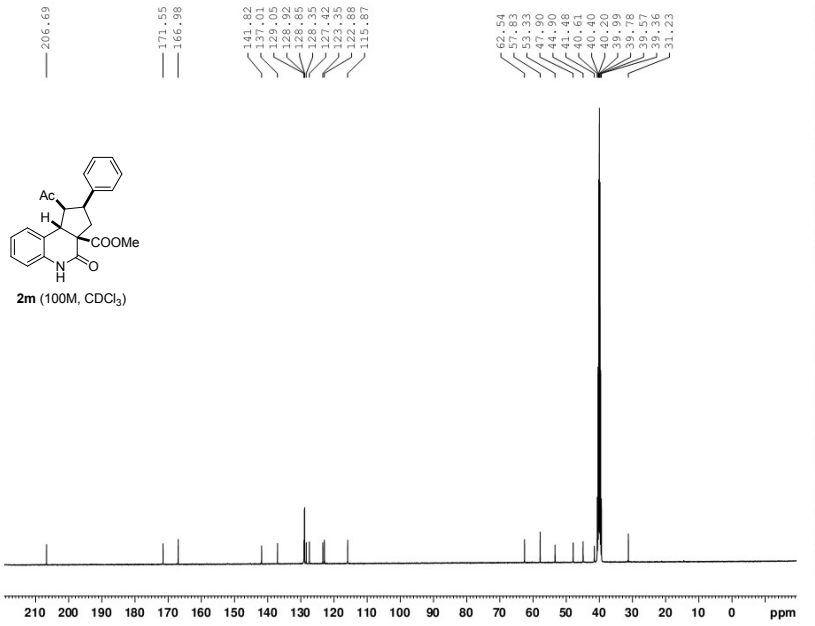
JA-234



2m (400M, $CDCl_3$)

```
Current Data Parameters
NAME          04-07-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150407
Time               13.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   48
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                124.55
DW                60.800 usec
DE                 6.50 usec
TE                291.3 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

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

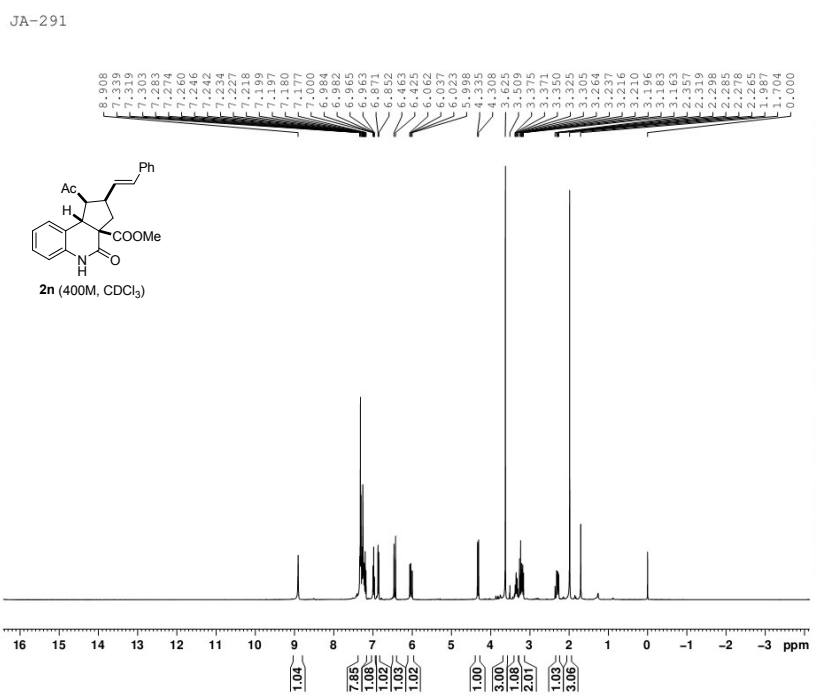
JA-234



2m (100M, $CDCl_3$)

```
Current Data Parameters
NAME          04-07-2015
EXPNO                 21
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150414
Time               1.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 8600
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                138.14
DW                20.800 usec
DE                 6.50 usec
TE                294.5 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

JA-291

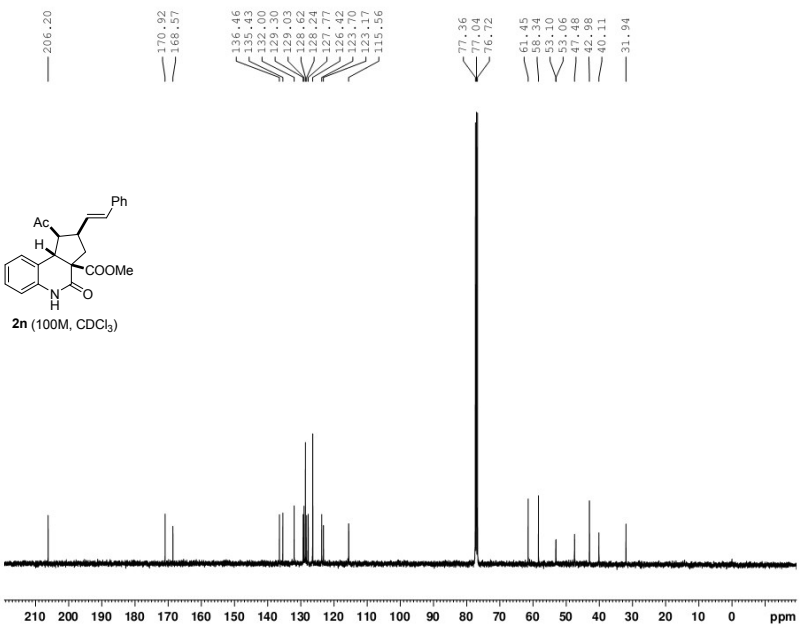


2n (400M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          07-24-2015
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150724
Time               16.23
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    52
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                138.14
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

JA-291

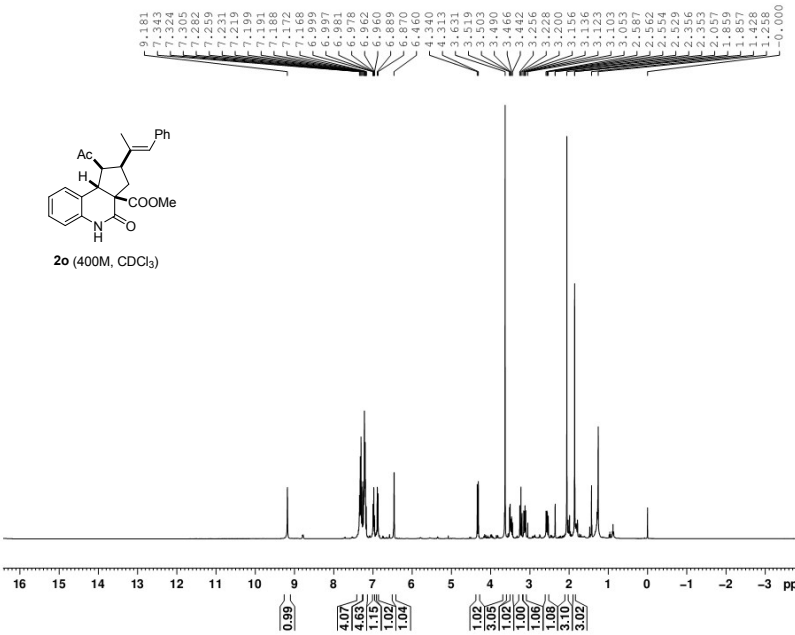


2n (100M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          07-24-2015
EXPNO                 17
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150727
Time               16.59
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   712
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

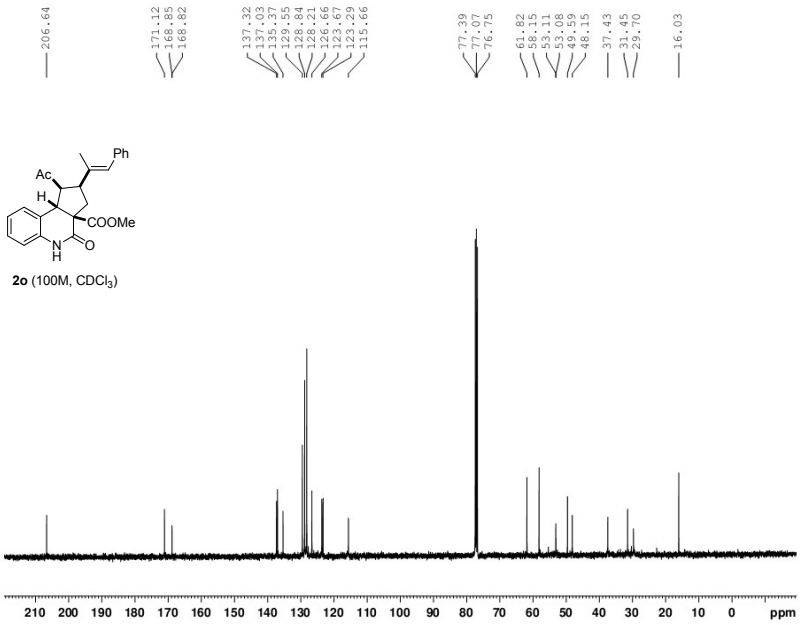
JA-306



2o (400M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               13.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ             3.9846387 sec
RG                 87.39
DW               60.800 usec
DE                6.50 usec
TE                518.1 K
D1            1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF          400.1300107 MHz
WDW                     EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

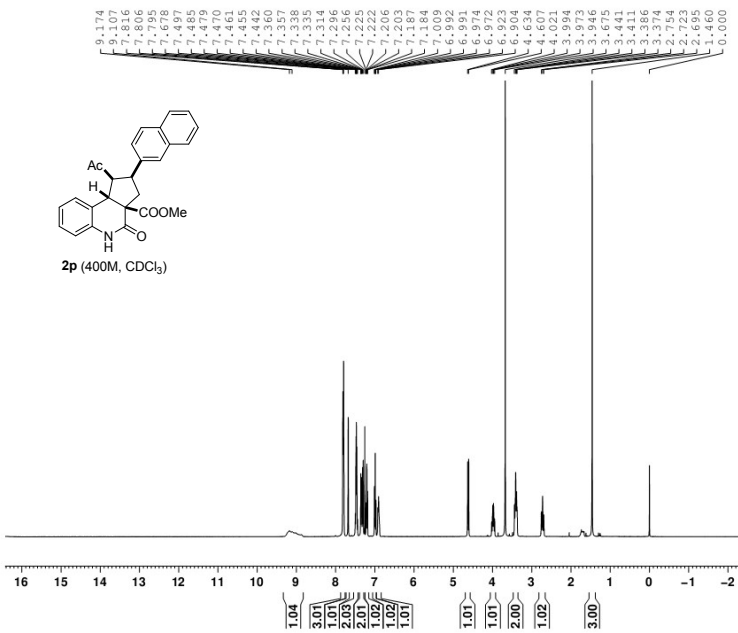
JA-306



2o (100M, $CDCl_3$)

```
Current Data Parameters
NAME          08-20-2015
EXPNO                 14
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150821
Time               14.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  504
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ             1.3631988 sec
RG                198.33
DW               20.800 usec
DE                6.50 usec
TE                518.1 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF          100.6127690 MHz
WDW                     EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

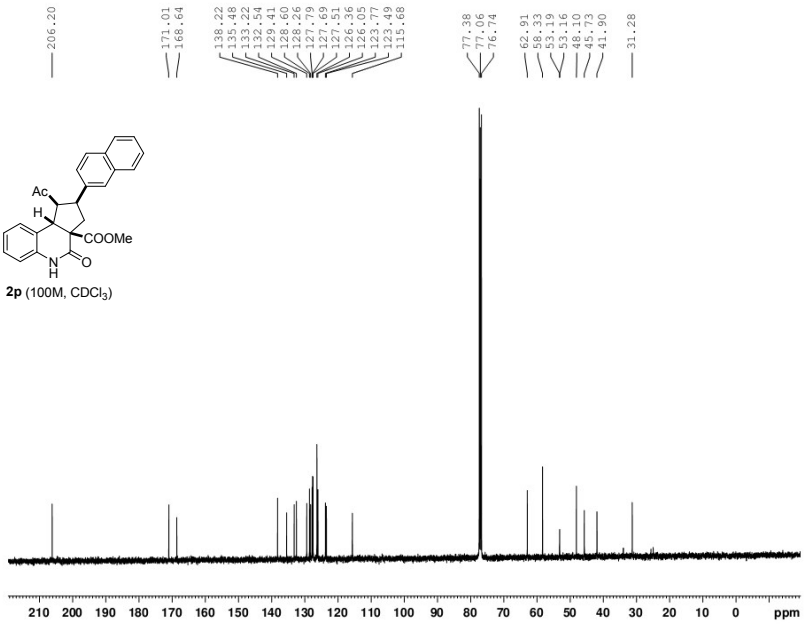
JA-295



2p (400M, $CDCl_3$)

```
Current Data Parameters
NAME          07-24-2015
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150724
Time               17.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDCl3
NS                    40
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                124.55
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300119 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

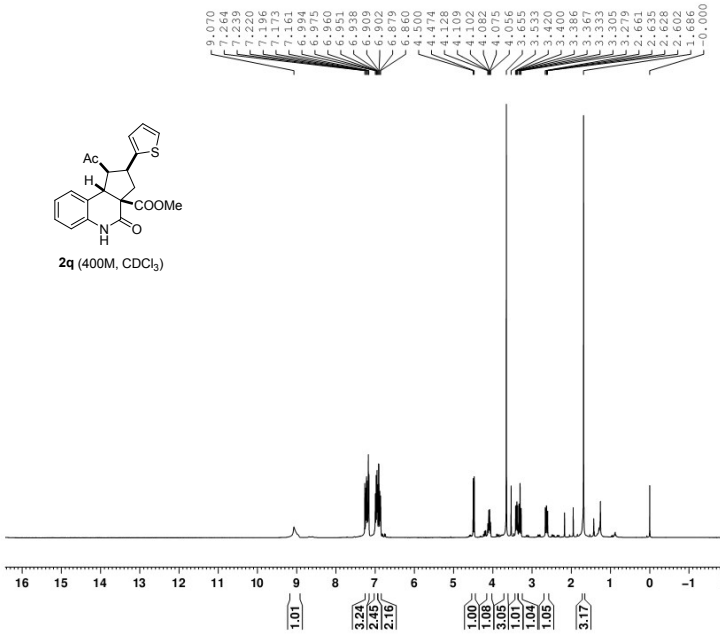
JA-259



2p (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-01-2015
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150609
Time                9.47
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   400
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.5 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

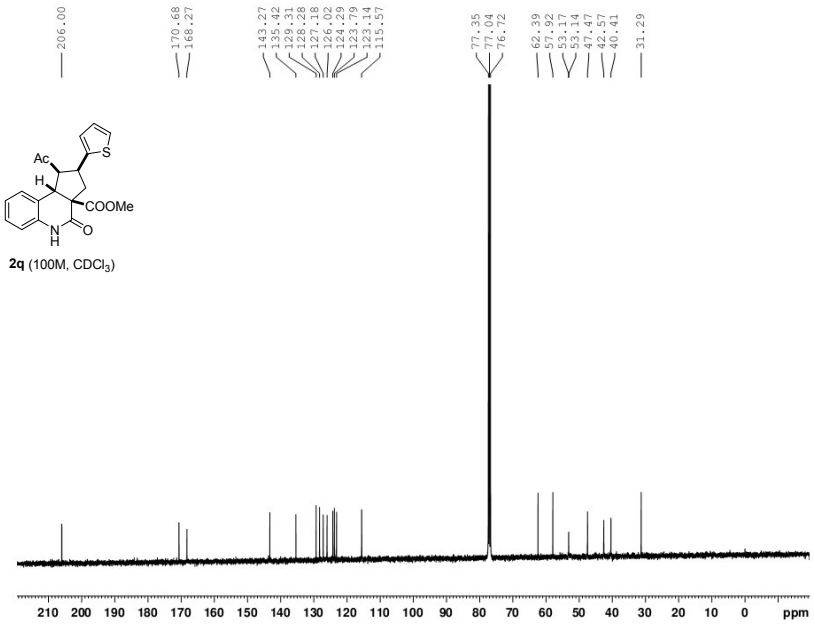
JA-294



2q (400M, $CDCl_3$)

```
Current Data Parameters
NAME          07-24-2015
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150724
Time               16.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    64
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                124.55
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300088 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

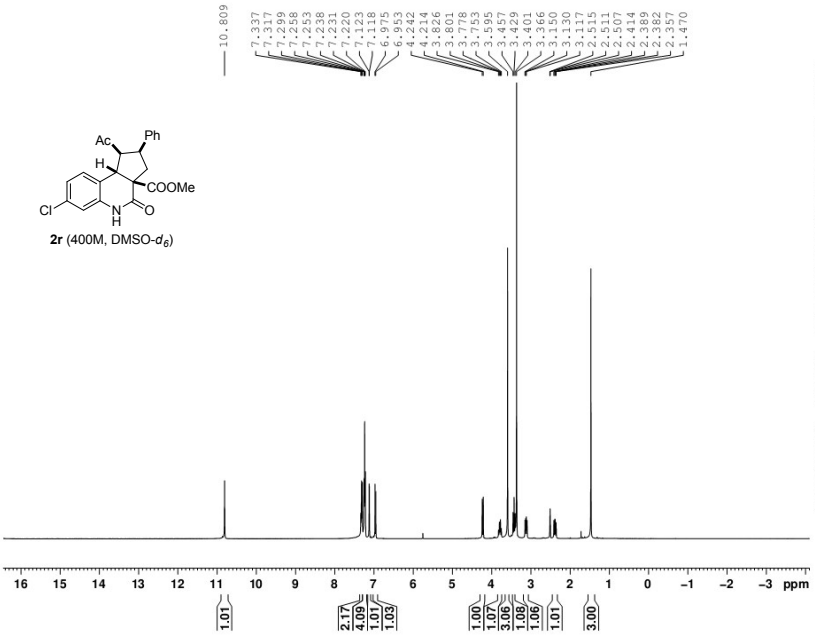
JA-284



2q (100M, $CDCl_3$)

```
Current Data Parameters
NAME          07-02-2015
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150716
Time               15.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   868
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

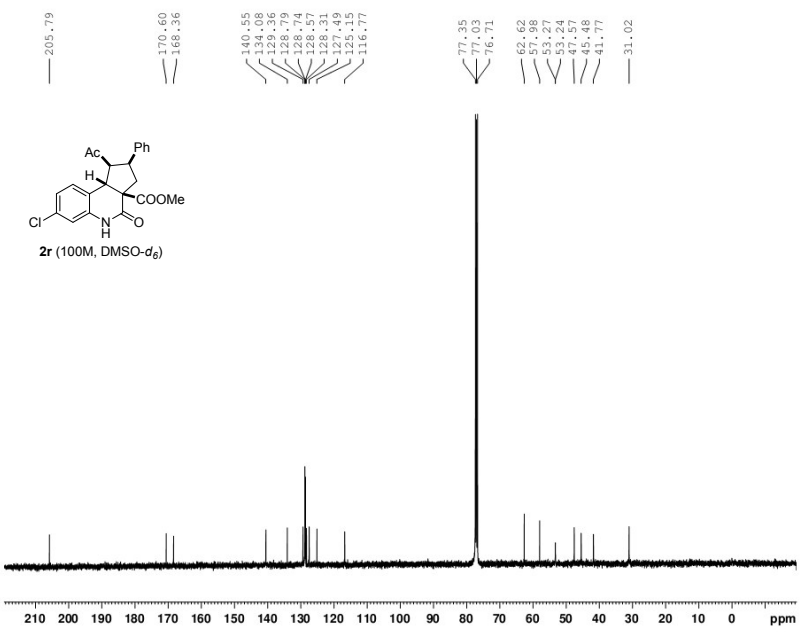
JA-293



2r (400M, DMSO-d_6)

```
Current Data Parameters
NAME          07-24-2015
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150724
Time               15.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   24
DS                    2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                94.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

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

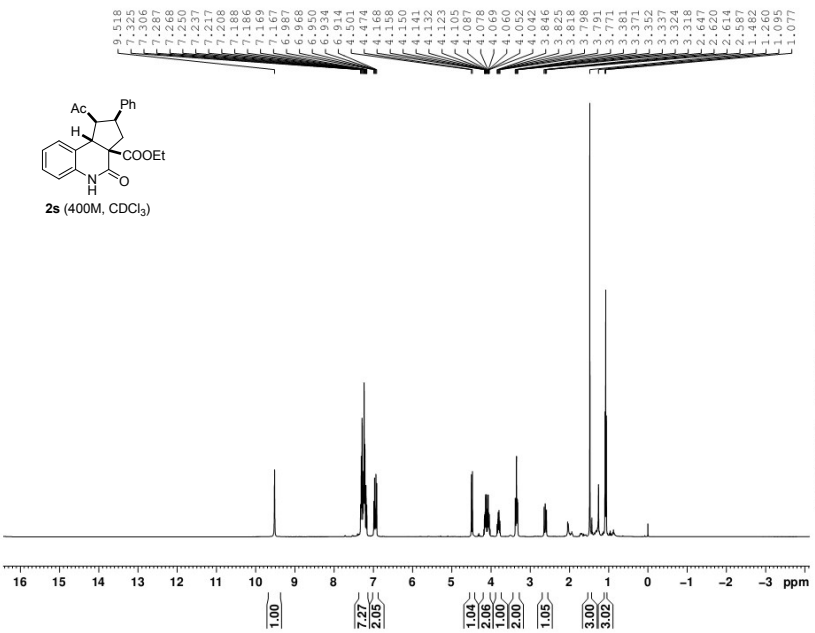
JA-270



2r (100M, DMSO-d_6)

```
Current Data Parameters
NAME          06-23-2015
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150624
Time               15.17
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  808
DS                    4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                 0 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             90.00 usec
PLW2         9.36999989 W
PLW12        0.22673000 W
PLW13        0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

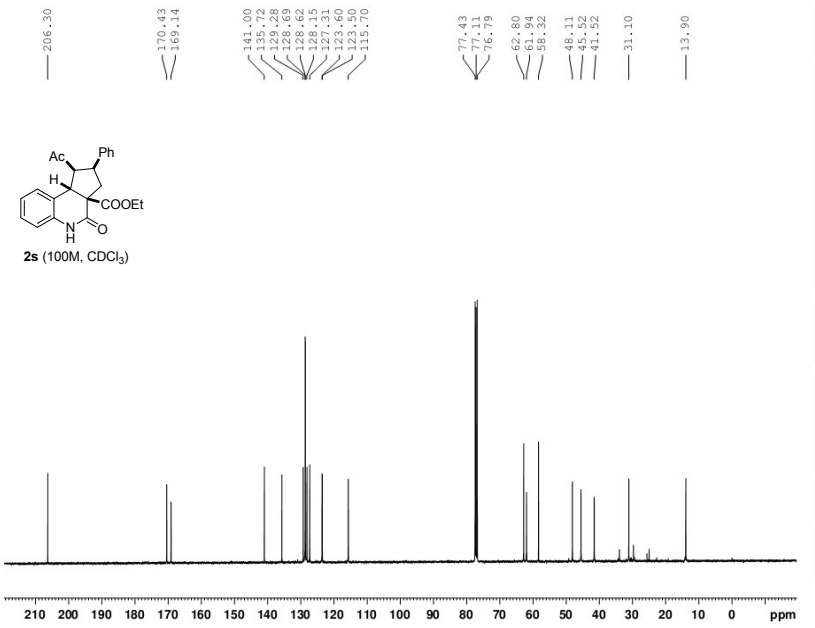
JA-267



2s (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-23-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150623
Time               13.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    44
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                55.68
DW                60.800 usec
DE                 6.50 usec
TE        0 K
D1          1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300066 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

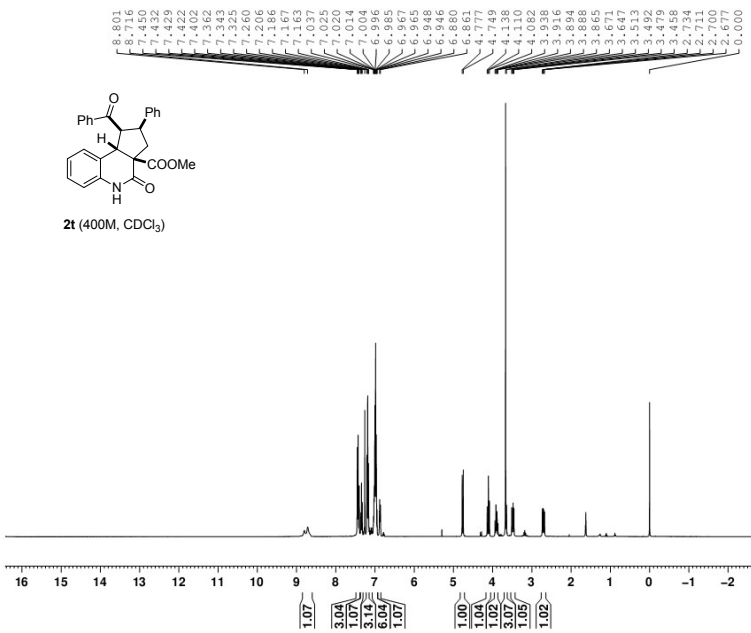
JA-267



2s (100M, $CDCl_3$)

```
Current Data Parameters
NAME          06-23-2015
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150624
Time               13.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  1004
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE        0 K
D1          2.00000000 sec
D11         0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

JA-265

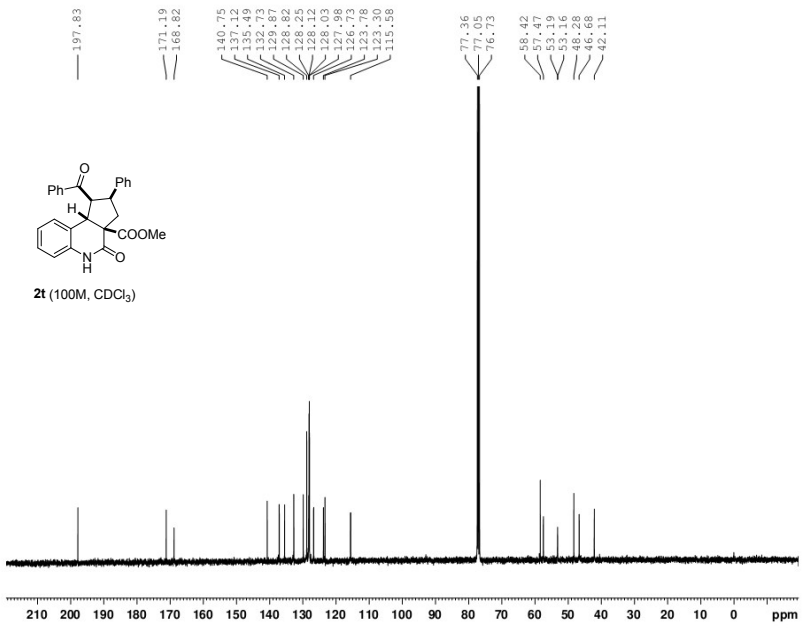


2t (400M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          06-11-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150611
Time               10.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                   120
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                168.03
DW                60.800 usec
DE                 6.50 usec
TE                295.8 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300104 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

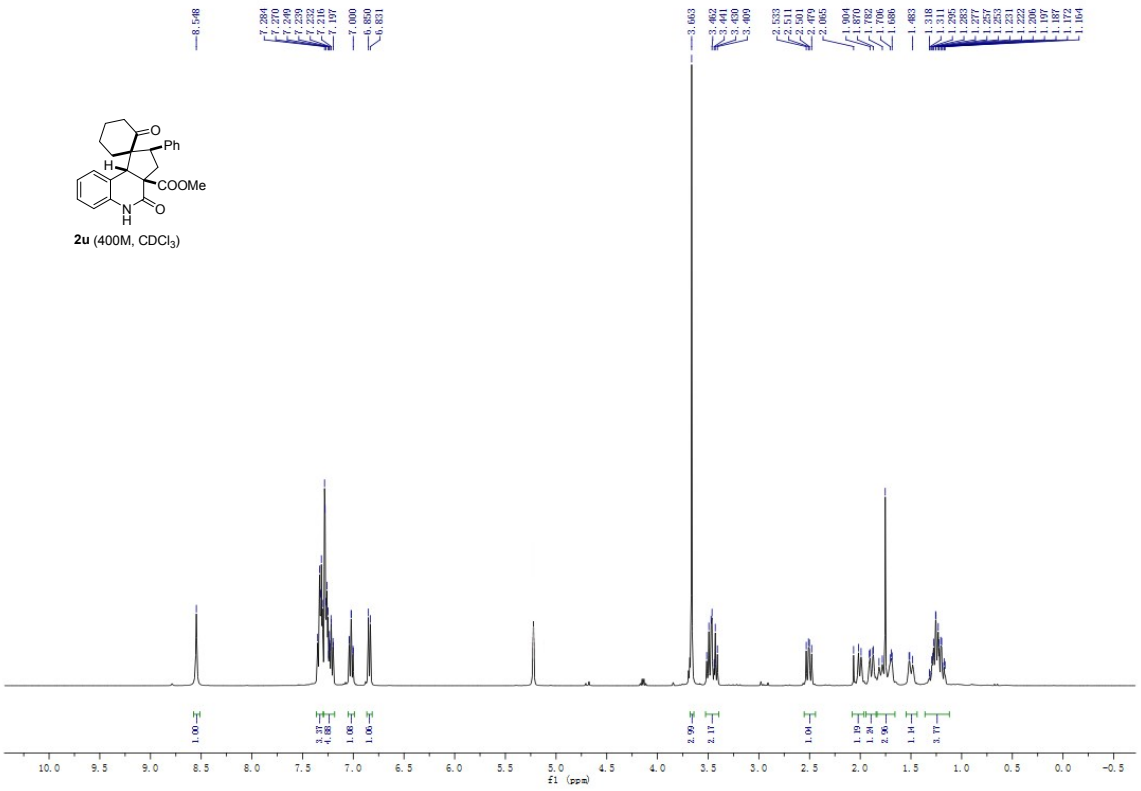
JA-265



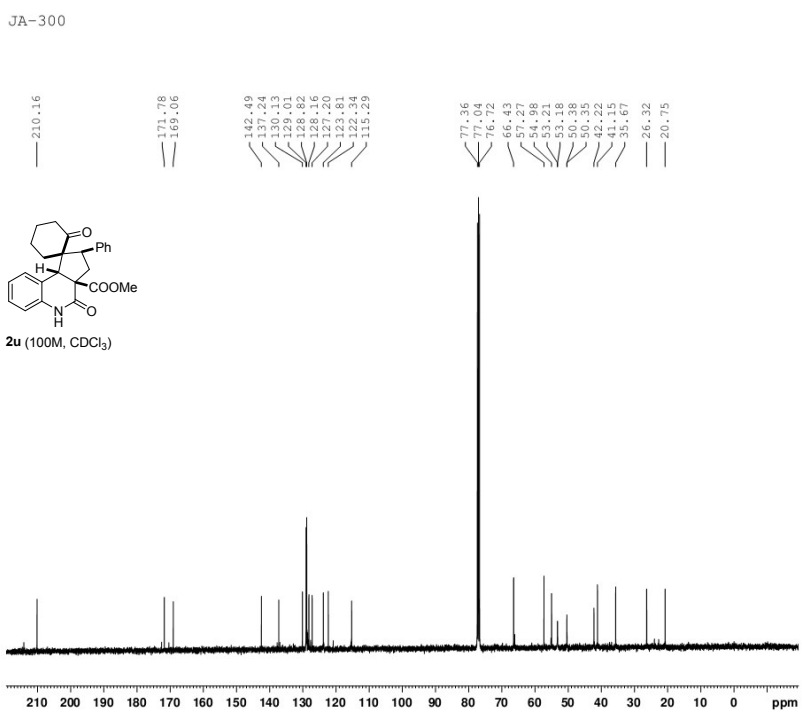
2t (100M, $CDCl_3$)

BRUKER

```
Current Data Parameters
NAME          06-11-2015
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150611
Time               15.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   600
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.9 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



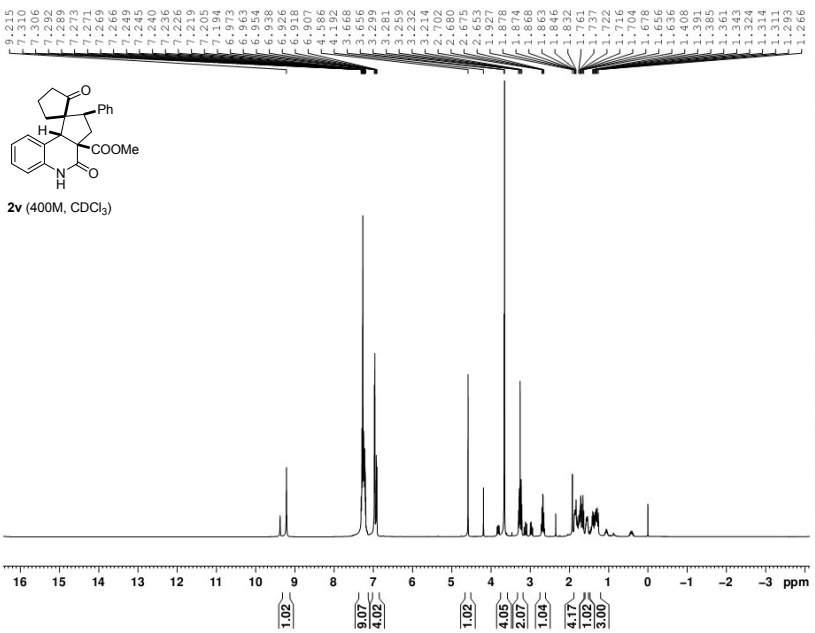
2u (400M, $CDCl_3$)



2u (100M, $CDCl_3$)

```
Current Data Parameters
NAME            07-30-2015
EXPNO                   13
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150803
Time                 10.39
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT             CDC13
NS                    980
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF          100.6127690 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

JA-307



2v (400M, $CDCl_3$)

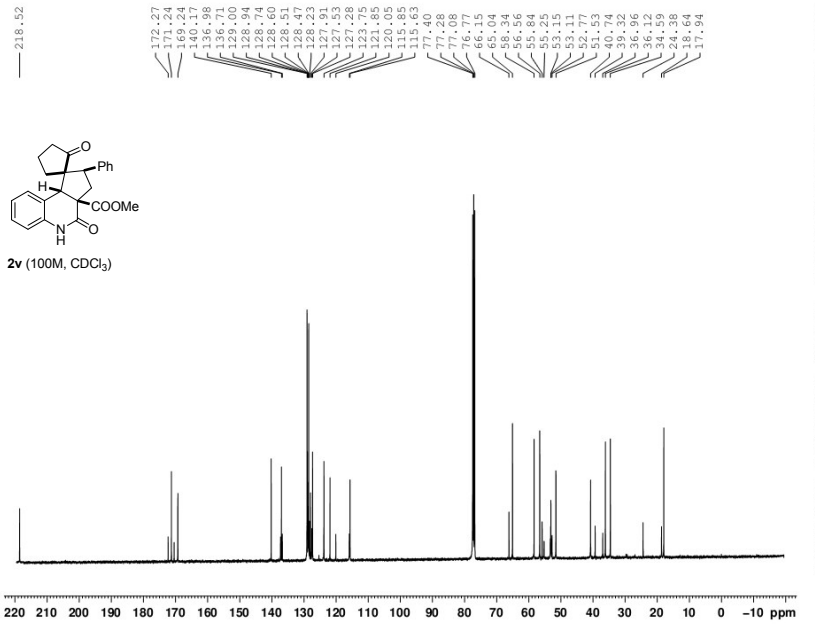
Current Data Parameters
NAME 08-20-2015
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150820
Time 14.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 128
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 67.01
DW 60.800 usec
DE 6.50 usec
TE 518.1 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PLW1 9.72999954 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

JA-307



2v (100M, $CDCl_3$)

Current Data Parameters
NAME 08-20-2015
EXPNO 13
PROCNO 1

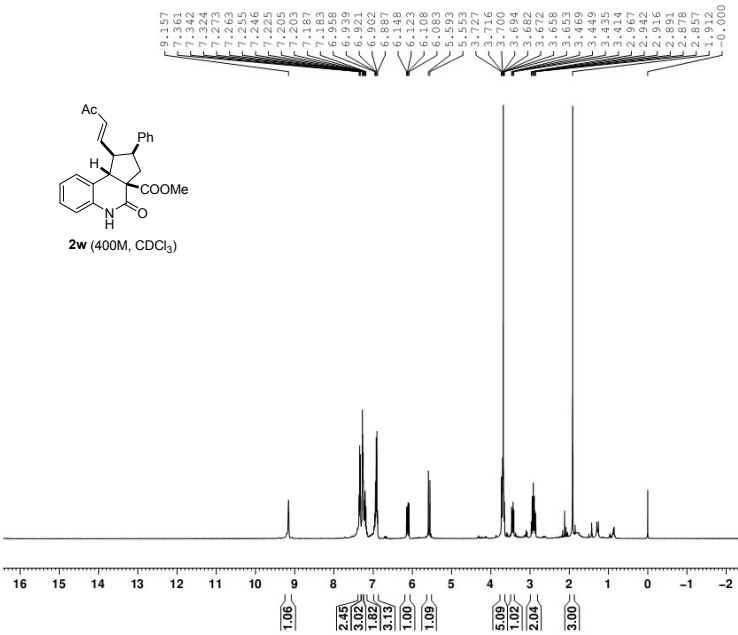
F2 - Acquisition Parameters
Date_ 20150821
Time 14.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 3036
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 518.1 K
D1 2.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 9.00 usec
PLW1 50.09999847 W
SFO1 100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.36999989 W
PLW12 0.22673000 W
PLW13 0.18365000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

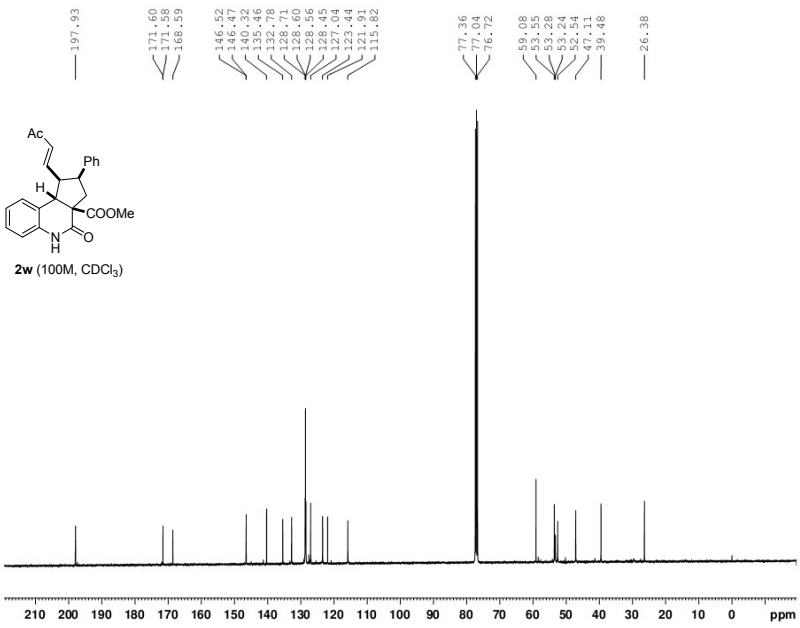
JA-292



2w (400M, $CDCl_3$)

```
Current Data Parameters
NAME          07-24-2015
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150724
Time               16.40
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    40
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                106.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.00 usec
PLW1          9.72999954 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF           400.1300088 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

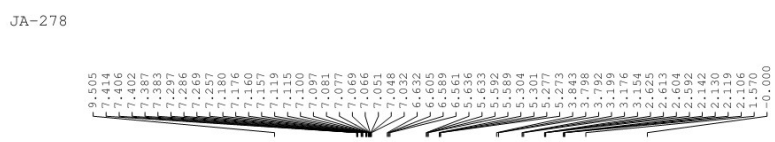
JA-292

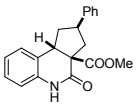


2w (100M, $CDCl_3$)

```
Current Data Parameters
NAME          07-24-2015
EXPNO                 18
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150727
Time               21.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                  4600
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

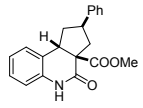
JA-278





2x (400M, $CDCl_3$)

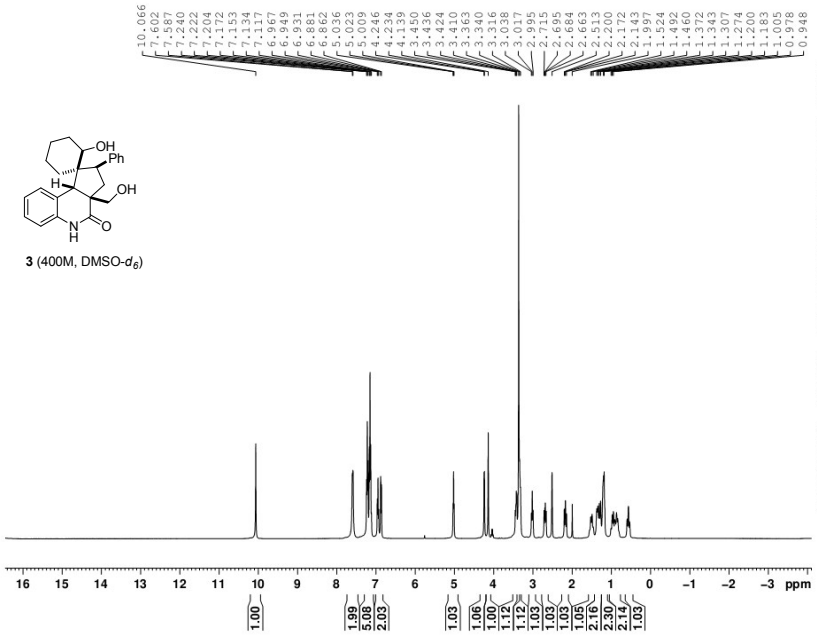

```
Current Data Parameters
NAME          07-02-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150702
Time              13.01
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT           CDCl3
NS                   52
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG               198.33
DW               60.800 usec
DE                6.50 usec
TE                518.1 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

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



2x (125M, $CDCl_3$)

S68

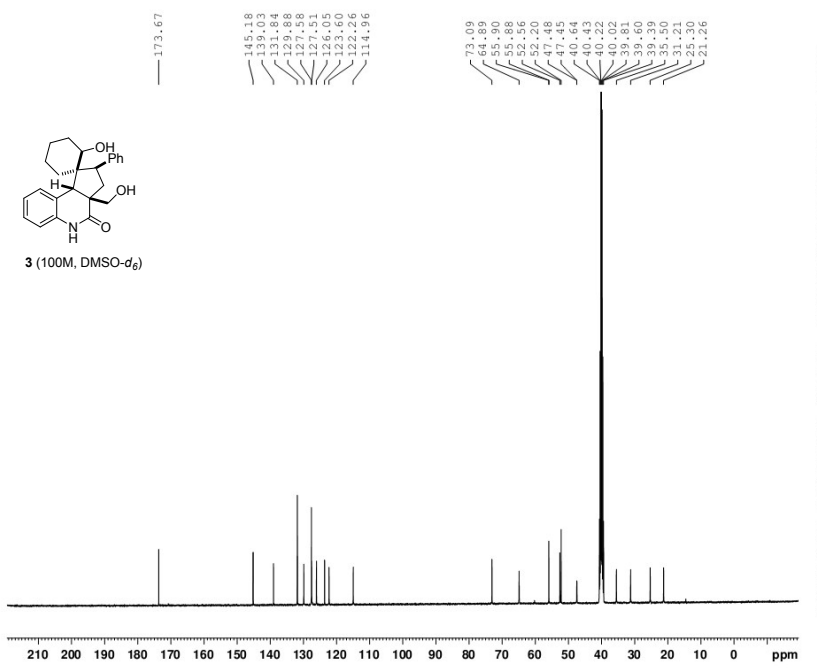
JA-315



3 (400M, DMSO-d_6)

```
Current Data Parameters
NAME          08-24-2015
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150824
Time               14.48
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   128
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

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

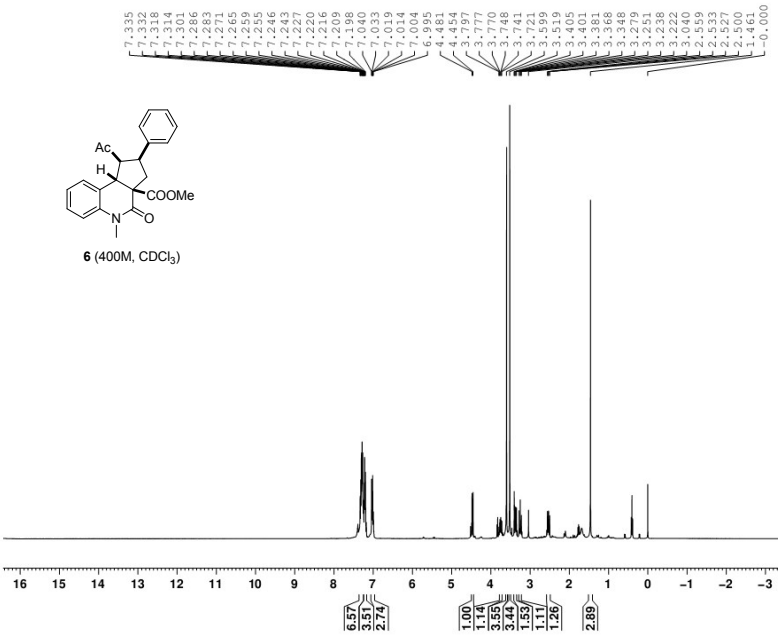
JA-315



3 (100M, DMSO-d_6)

```
Current Data Parameters
NAME          08-24-2015
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150824
Time               16.34
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                 4600
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                168.03
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF          100.6127690 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

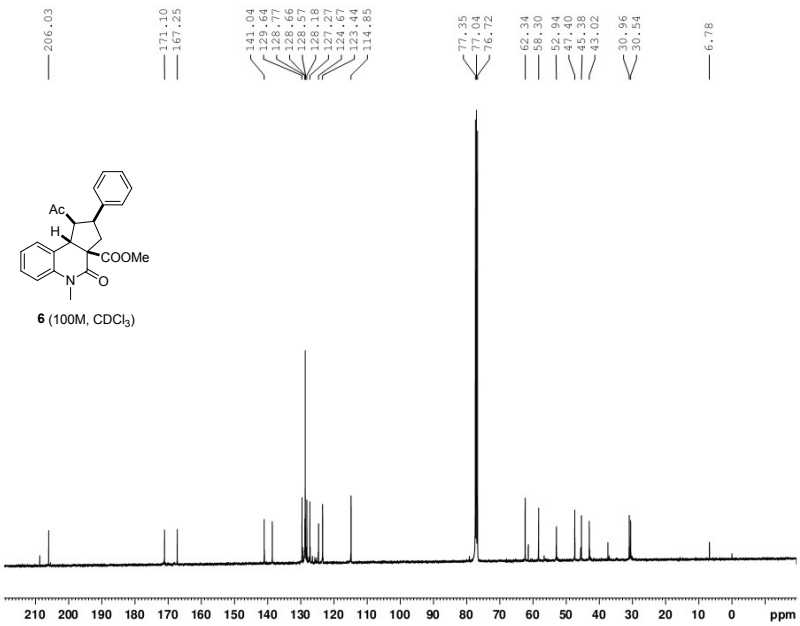
JA-285



6 (400M, $CDCl_3$)

```
Current Data Parameters
NAME          07-14-2015
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150714
Time                9.44
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   64
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                124.55
DW                60.800 usec
DE                 6.50 usec
TE                518.1 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1         9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300081 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

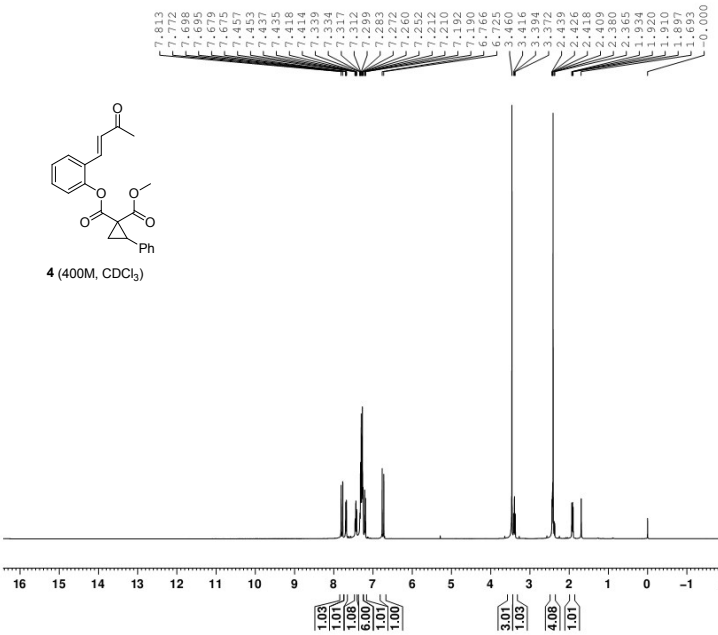
JA-285



6 (100M, $CDCl_3$)

```
Current Data Parameters
NAME          07-14-2015
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150715
Time               23.24
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 6400
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                518.1 K
D1           2.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1        50.09999847 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF         100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

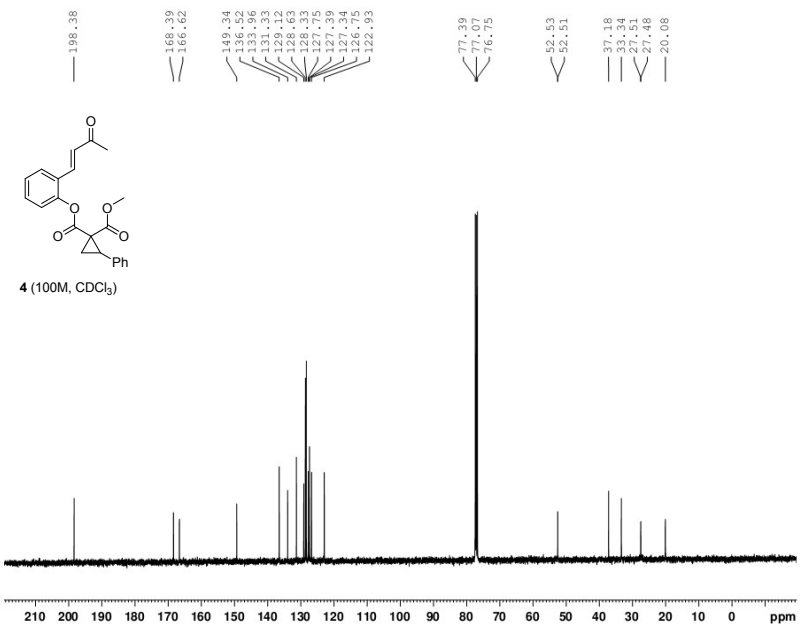
JA-262



4 (400M, $CDCl_3$)

```
Current Data Parameters
NAME          06-05-2015
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150605
Time               13.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT            CDC13
NS                    56
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 94.96
DW                60.800 usec
DE                 6.50 usec
TE                297.0 K
D1             1.00000000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                14.00 usec
PLW1          9.72999954 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300101 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

JA-262

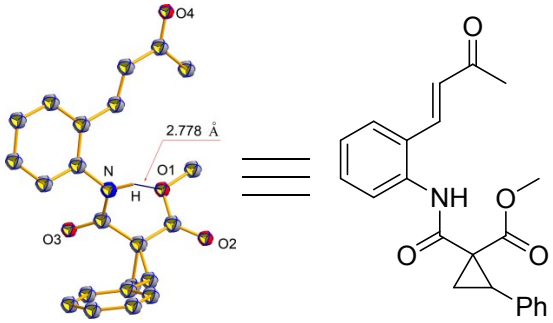


4 (100M, $CDCl_3$)

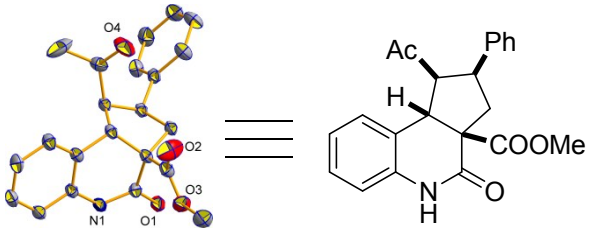
```
Current Data Parameters
NAME          06-05-2015
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150609
Time               13.58
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            CDC13
NS                   260
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                198.33
DW                20.800 usec
DE                 6.50 usec
TE                296.3 K
D1             2.00000000 sec
D11            0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.00 usec
PLW1         50.09999847 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2          9.36999989 W
PLW12         0.22673000 W
PLW13         0.18365000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

4. X-ray single crystal structure for 1m, 2m, and 3.

X-ray structure of 1m:



X-ray structure of 2m:



X-ray structure of 3: