

Supporting Information

for

Additive-assisted regioselective 1,3-dipolar cycloaddition of azomethine ylides with benzylideneacetone

Chuqin Peng, Jiwei Ren, Jun-An Xiao, Honggang Zhang, Hua Yang*[§], Yiming Luo*[¶]

Address: School of Chemistry and Chemical Engineering, Central South

University, Changsha, Hunan 410083, P. R. China

Email: Yiming Luo - ymluo@csu.edu.cn; Hua Yang - hyangchem@csu.edu.cn

*Corresponding author

§Corresponding author Tel.: +86-731-88830833

¶Corresponding author Tel.: +86-731-88864409

Experimental procedures, characterization data and

copies of ¹H and ¹³C NMR spectra

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General information

¹H 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.

¹³C NMR data were collected at 100 MHz with complete proton decoupling.

Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as internal standard. Mass spectra (MS) were measured by 3200 Q TRAP LC-MS/MS utilizing electrospray ionization (ESI) and acetonitrile was used to dissolve the sample.

All chemicals were obtained from commercial sources and used without further purification. Column chromatography was carried out on silica gel (300–400 mesh).

Thin-layer chromatography (TLC) was performed on TLC silica gel 60 F254 plates.

Benzylideneacetone derivatives **3a–g** were prepared according to literature procedure [1].

Typical procedure for the synthesis of spirooxindole derivatives

A mixture of isatin (**1a**, 110.3 mg, 0.75 mmol, 1.5 equiv), benzylamine (**2**, 107.1 mg, 1.0 mmol, 2.0 equiv), benzylideneacetone (**3a**, 73.1 mg, 0.5 mmol, 1.0 equiv) and additives (Conditions A: H₂O, 45 mg, 5.0 equiv; Conditions B: 4-nitrobenzoic acid, 167.1 mg, 2.0 equiv) in THF (5.0 mL) was stirred at rt for the indicated time. After completion of the reaction as monitored by TLC, Conditions A: the solvent was removed in vacuo. Conditions B: saturated NaHCO₃ solution was added, the organic phase was separated and the aqueous layer was extracted with CH₂Cl₂. The combined organic phases were dried over Na₂SO₄, concentrated under reduced pressure and the

crude mixture was purified via flash silica gel chromatography (eluent: dichloromethane/ethyl acetate 97:3 to 95:5) to give cycloadducts **4a**, white solid, conditions A: 144.5 mg, yield 76%; conditions B: 45.8 mg, yield 27%; **5a**, white solid, conditions A: 23.5 mg, yield 12%; conditions B: 120.3 mg, yield 63%.

Characterization data for spiropyrrolidine oxindoles

Crystal data for **4e** and **5e**

Crystal data for **4e**: formula: C₂₅H₂₁Br₁N₂O₂, unit cell parameters: *a* 7.382(10), *b* 9.93110(10), *c* 16.9977(3), *P*1. Crystallographic data for this structure have been deposited at the Cambridge Crystallographic Data Centre and allocated deposition number CCDC 967232.

Crystal data for **5e**: formula: C₂₅H₂₁Br₁N₂O₂, unit cell parameters: *a* 10.3391(7), *b* 10.6015(6), *c* 22.2259(16), *P*21/*c*. Crystallographic data for this structure have been deposited at the Cambridge Crystallographic Data Centre and allocated deposition number CCDC 967233.

Spectral characterization data for spiropyrrolidine oxindoles

3'-Acetyl-4',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (**4a**)

White solid (conditions A: 144.5 mg, yield 76%; conditions B: 45.8 mg, yield 27%); m.p. 170-172°C; IR (KBr) ν 3332, 3030, 1712, 1619, 1471, 1188, 753, 700 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.40 (*br s*, 1H), 6.93-7.34 (*m*, 15H), 4.97-5.02 (*m*, 1H), 3.96-4.03 (*m*, 2H), 1.57 (*s*, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 204.4, 182.9, 140.4, 140.3, 138.7, 130.0, 129.7, 128.6, 128.4, 128.3, 127.7, 127.0, 126.0, 123.6, 110.3, 68.3, 67.9, 55.1, 30.8, 25.4; HRMS (TOF-ES+) *m/z*: [M+Na]⁺ calcd for C₂₅H₂₂N₂O₂Na 405.1576, found 405.1598.

4'-Acetyl-3',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (5a)

White solid (conditions A: 23.5 mg, yield 12%; conditions B: 120.3 mg, yield 63%);
m.p. 205-206°C; IR (KBr) ν 3391, 3061, 1709, 1620, 1471, 1187, 753, 699 cm^{-1} ; ^1H
NMR (400 MHz, CDCl_3) δ 7.70-7.72 (m, 1H), 7.54 (d, $J = 7.2$ Hz, 2H), 7.35 (t, $J =$
7.2 Hz, 2H), 6.99-7.29 (m, 9H), 6.59-6.61 (m, 1H), 5.65 (d, $J = 10.4$ Hz, 1H), 4.74 (t,
 $J = 11.2$ Hz, 1H), 4.32 (d, $J = 11.6$ Hz, 1H), 2.58 (s, 1H), 1.65 (s, 3H); ^{13}C NMR (100
MHz, CDCl_3) δ 205.8, 180.2, 147.3, 142.9, 141.0, 140.4, 129.7, 128.9, 128.8, 128.5,
128.3, 124.0, 123.39, 123.33, 109.9, 72.4, 61.0, 58.9, 54.6, 31.2; HRMS (TOF-ES+)
m/z: $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{25}\text{H}_{23}\text{N}_2\text{O}_3$ 383.1760, found 383.1752.

3'-Acetyl-5-fluoro-4',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (4b)

White solid (conditions A: 117.0 mg, yield 59%; conditions B: 69.9 mg, yield 35%);
m.p. 122-123°C; IR (KBr) ν 3426, 3062, 3031, 2923, 1714, 1630, 1484, 1186, 816,
755, 699 cm^{-1} ; ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.62 (*br s*, 1H), 7.17-7.24 (m, 10H),
7.07 (d, $J = 8.0$ Hz, 2H), 6.84-6.87 (m, 1H), 4.79 (dd, $J = 10.0, 5.2$ Hz, 1H), 4.04 (d, J
 $= 5.2$ Hz, 1H), 3.85 (d, $J = 10.8$ Hz, 1H), 3.76 (d, $J = 10.4$ Hz, 1H), 1.53 (s, 3H); ^{13}C
NMR (100 MHz, $\text{DMSO}-d_6$) δ 204.9, 182.3, 159.7, 157.3, 141.7, 139.6, 138.5, 133.1,
128.8, 128.7, 128.4, 127.8, 127.7, 127.2, 116.1, 115.8, 114.0, 113.7, 110.9, 110.8, 67.9,
67.67, 67.66, 67.61, 54.8, 30.8; HRMS (TOF-ES+) m/z: $[\text{M}+\text{Na}]^+$ calcd for
 $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_2\text{NaF}$ 423.1485, found 423.1480.

4'-Acetyl-5-fluoro-3',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (5b)

White solid (conditions A: 41.1 mg, yield 21%; conditions B: 114.1 mg, yield 57%);
m.p. 243-245°C; IR (KBr) ν 3372, 3331, 1715, 1637, 1490, 1171, 809, 750, 700 cm^{-1} ;
 ^1H NMR (400 MHz, DMSO- d_6) δ 9.90 (s, 1H), 7.55-7.60 (m, 3H), 7.34 (t, $J = 7.6$ Hz
2H), 7.25 (t, $J = 7.6$ Hz, 1H), 6.95-7.12 (m, 6H), 6.55 (dd, $J = 8.4, 4.4$ Hz, 1H), 5.46
(dd, $J = 10.6, 5.8$ Hz, 1H), 4.80 (t, $J = 11.6$ Hz, 1H), 4.14-4.18 (m, 2H), 1.65 (s, 3H);
 ^{13}C NMR (100 MHz, DMSO- d_6) δ 206.6, 180.8, 160.0, 157.6, 143.4, 138.8, 136.0,
133.1, 133.0, 129.0, 128.4, 128.3, 127.8, 127.6, 115.5, 115.3, 112.3, 112.1, 110.3,
110.2, 72.8, 60.2, 58.3, 54.2, 31.5; HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for
 $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_2\text{NaF}$ 423.1485, found 423.1503.

3'-Acetyl-5-methyl-4',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (4c)

White solid (conditions A: 118.5 mg, yield 60%; conditions B: 54.7 mg, yield 28%);
m.p. 183-184°C; IR (KBr) ν 3328, 3185, 3030, 2918, 1706, 1624, 1489, 1204, 819,
754, 699 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6) δ 10.48 (s, 1H), 7.04-7.28 (m, 11H),
6.75 (d, $J = 8.4$ Hz, 1H), 4.80 (dd, $J = 10.0, 5.2$ Hz, 1H), 3.71-3.87 (m, 3H), 2.28 (s,
3H), 1.47 (s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 204.7, 182.4, 142.0, 140.0,
139.9, 131.2, 131.1, 129.8, 128.8, 128.7, 128.5, 127.8, 127.6, 127.2, 126.8, 109.8,
67.9, 67.7, 67.4, 55.0, 30.7, 21.3; HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for
 $\text{C}_{26}\text{H}_{24}\text{N}_2\text{O}_2\text{Na}$ 419.1735, found 419.1742.

4'-Acetyl-5-methyl-3',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (5c)

White solid (conditions A: 55.8 mg, yield 28%; conditions B: 121.6 mg, yield 61%);
m.p. 186-187°C; IR (KBr) ν 3342, 3316, 3027, 2917, 2870, 1711, 1625, 1491, 1162,

818, 757, 700, 699 cm^{-1} ; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 9.77 (s, 1H), 7.58 (d, $J = 7.2$ Hz, 2H), 7.50 (s, 1H), 7.34 (t, $J = 7.2$ Hz, 2H), 7.27 (t, $J = 7.2$ Hz, 1H), 6.94-7.10 (m, 6H), 6.46 (d, $J = 7.6$ Hz, 1H), 5.48 (dd, $J = 10.4, 5.6$ Hz, 1H), 4.79 (t, $J = 11.2$ Hz, 1H), 4.12 (d, $J = 12.0$ Hz, 1H), 4.03 (d, $J = 6.0$ Hz, 1H), 2.37 (s, 3H), 1.65 (s, 3H); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 206.8, 180.7, 143.5, 140.3, 136.4, 131.0, 130.9, 129.4, 128.9, 128.3, 172.7, 127.4, 124.9, 109.3, 72.4, 60.2, 58.5, 54.2, 31.6, 21.3; HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{24}\text{N}_2\text{O}_2\text{Na}$ 419.1735, found 419.1736.

3'-Acetyl-5-chloro-4',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (4d)

White powder (conditions A: 104.8 mg, yield 50%; conditions B: 38.8 mg, yield 21%); m.p 130-132°C; IR (KBr) ν 3420, 3031, 2924, 1713, 1619, 1474, 1183, 817, 748, 700 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 8.61 (s, 1H), 7.19-7.35 (m, 12H), 6.88 (d, $J = 8.4$ Hz, 1H), 4.95 (d, $J = 10.0$ Hz, 1H), 4.01 (d, $J = 10.8$ Hz, 1H), 3.90 (t, $J = 10.4$ Hz, 1H), 2.55 (s, 1H), 1.62 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 203.9, 182.5, 140.0, 138.7, 138.3, 132.0, 129.6, 128.9, 128.6, 128.39, 128.34, 127.8, 127.1, 126.9, 126.6, 111.5, 68.2, 67.8, 67.6, 55.1, 30.8; HRMS (TOF-ES+) m/z : $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_2\text{Cl}$ 417.1370, found 417.1359.

4'-Acetyl-5-chloro-3',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (5d)

White solid (conditions A: 38.8 mg, yield 19%; conditions B: 94.8 mg, yield 46%); m.p. 240-241°C; IR (KBr) ν 3442, 3353, 3282, 1714, 1622, 1480, 1176, 815, 749, 701 cm^{-1} ; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.01 (s, 1H), 7.71 (d, $J = 2.0$ Hz, 1H), 7.58 (d, $J = 7.2$ Hz, 1H), 6.97-7.36 (m, 9H), 6.57 (d, $J = 8.4$, 1H), 5.47 (dd, $J = 16.8, 6.0$

Hz, 1H), 4.77 (t, $J = 11.2$ Hz, 1H), 4.14-4.20 (m, 2H), 1.63 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 206.5, 180.5, 143.3, 141.6, 135.9, 133.2, 129.0, 128.9, 128.4, 128.28, 128.24, 127.7, 127.6, 126.2, 124.5, 110.9, 72.5, 60.2, 58.3, 54.2, 31.4; HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_2\text{NaCl}$ 439.1168, found 439.1189.

3'-Acetyl-6-bromo-4',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (4e)

White solid (conditions A: 141.7 mg, yield 62%; conditions B: 88.3 mg, yield 19%); m.p. 176-178°C; IR (KBr) ν 3325, 3182, 3029, 1708, 1612, 1452, 1325, 699 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6) δ 10.74 (s, 1H), 7.63-7.17 (m, 12H), 7.016-7.020 (m, 1H), 4.80 (dd, $J = 10.0, 4.8$ Hz, 1H), 4.03 (d, $J = 4.8$ Hz, 1H), 3.83 (d, $J = 10.8$ Hz, 1H), 3.74 (t, $J = 10.0$ Hz, 1H), 1.53 (s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 204.8, 182.2, 144.0, 141.8, 139.6, 130.6, 128.9, 128.7, 128.5, 128.1, 127.8, 127.6, 127.2, 125.0, 122.1, 112.9, 67.8, 67.4, 55.4, 55.0, 30.9; HRMS (TOF-ES+) m/z : $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_2\text{Br}$ 461.0865, found 461.0861.

4'-Acetyl-6-bromo-3',5'-diphenylspiro[indoline-3,2'-pyrrolidin]-2-one (5e)

White solid (conditions A: 35.4 mg, yield 15%; conditions B: 139.8 mg, yield 61%); m.p. 232-234°C; IR (KBr) ν 3390, 3193, 3030, 1709, 1610, 1452, 1326, 700 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6) δ 10.02 (s, 1H), 7.64 (d, $J = 8.0$ Hz, 1H), 7.57 (d, $J = 7.6$ Hz, 2H), 7.25-7.35 (m, 4H), 7.12-7.13 (m, 3H), 6.96-6.98 (m, 2H), 6.70-6.71 (m, 1H), 5.46 (dd, $J = 10.8, 6.0$ Hz, 1H), 4.79 (t, $J = 11.2$ Hz, 1H), 4.12-4.18 (m, 2H), 1.65 (s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 206.5, 180.6, 144.4, 143.4, 136.0, 130.4, 128.9, 128.4, 128.3, 127.8, 127.6, 126.4, 124.8, 121.6, 112.3, 72.2, 60.1, 58.2, 54.0,

31.5; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{25}H_{21}N_2O_2NaBr$ 483.0684, found 483.0681.

3'-Acetyl-4'-(2-hydroxyphenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one (4f)

White solid (conditions A: 145.5 mg, yield 73%; conditions B: 17.5 mg, yield 9%); m.p. 210-211°C; IR (KBr) ν 3381, 3310, 1694, 1621, 1470, 1352, 1251, 1225, 752, 704 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 8.63-8.72 (m, 1H), 7.49 (d, $J = 6.8$ Hz, 1H), 7.07-7.41 (m, 10H), 6.98 (d, $J = 7.6$ Hz, 1H), 6.86-6.92 (m, 2H), 5.20 (d, $J = 7.2$ Hz, 1H), 4.19 (t, $J = 10.8$ Hz, 1H), 4.03 (dd, $J = 10.8, 2.4$ Hz, 1H), 2.53 (s, 1H), 1.61 (s, 3H); ^{13}C NMR (100 MHz, $DMSO-d_6$) δ 205.3, 182.1, 156.0, 142.7, 142.3, 131.4, 129.9, 129.3, 128.3, 127.8, 127.5, 127.4, 126.1, 125.5, 122.2, 119.5, 115.8, 109.9, 68.0, 66.5, 66.2, 49.4, 30.6; HRMS (TOF-ES+) m/z: $[M+Na]^+$ calcd for $C_{25}H_{22}N_2O_3Na$ 421.1528, found 421.1518.

4'-Acetyl-3'-(2-hydroxyphenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one (5f)

White solid (conditions A: 25.7 mg, yield 13%; conditions B: 141.7 mg, yield 71%); m.p. 213-215°C; IR (KBr) ν 3376, 3256, 3189, 3078, 1702, 1673, 1471, 1455, 1361, 1257, 753, 704 cm^{-1} ; 1H NMR (400 MHz, $DMSO-d_6$) δ 9.91 (s, 1H), 8.96 (s, 1H), 7.62 (d, $J = 7.2$ Hz, 1H), 7.58 (d, $J = 7.2$ Hz, 2H), 7.29-7.36 (m, 3H), 7.25 (t, $J = 7.2$ Hz, 1H), 7.09 (td, $J = 7.6, 1.2$ Hz, 1H), 6.99 (t, $J = 7.6$ Hz, 1H), 6.87 (td, $J = 8.0, 1.6$ Hz, 1H), 6.62 (t, $J = 7.2$ Hz, 1H), 6.51-6.55 (m, 2H), 5.45 (dd, $J = 10.4, 5.6$ Hz, 1H), 4.83 (d, $J = 12.0$ Hz, 1H), 4.62 (t, $J = 10.8$ Hz, 1H), 3.98 (d, $J = 6.0$ Hz, 1H), 1.53 (s,

3H); ^{13}C NMR (100MHz, DMSO- d_6) δ 207.2, 181.5, 156.3, 143.6, 142.6, 130.9, 128.7, 128.6, 128.2, 127.9, 127.6, 125.5, 122.6, 121.6, 118.9, 115.2, 109.1, 72.1, 60.5, 60.3, 45.1, 30.9; HRMS (TOF-ES+) m/z: $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{25}\text{H}_{23}\text{N}_2\text{O}_3$ 399.1709, found 399.1700.

3'-Acetyl-5'-phenyl-4'-(pyridin-2-yl)spiro[indoline-3,2'-pyrrolidin]-2-one (4g)

White powder, combined yield of **4g** and **5g** (conditions A: 172.3 mg, yield 90%; conditions B: 160.9 mg, yield 84%); IR (KBr) ν 3334, 3083, 3060, 1712, 1619, 1471, 1187, 753, 701 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6) δ 9.12 (s, 1H), 8.58 (s, 1H), 7.07-7.55 (m, 12H), 6.96 (d, $J = 7.6$ Hz, 1H) 6.90 (d, $J = 7.6$ Hz, 1H), 5.03 (d, $J = 10.0$ Hz, 1H), 4.58 (d, $J = 11.2$ Hz, 1H), 4.08 (dd, $J = 10.8, 8.0$ Hz, 1H), 1.61 (s, 3H); HRMS (TOF-ES+) m/z: $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{24}\text{H}_{21}\text{N}_3\text{O}_2\text{Na}$ 406.1531, found 406.1538.

*Note: Compounds **4g** and **5g** cannot be separated by column chromatography. Pure **4g** was obtained by recrystallization and the regioisomeric ratio was determined by ^1H NMR spectroscopy of the mixture of **4g** and **5g**.*

4'-Acetyl-5'-phenyl-3'-(pyridin-2-yl)spiro[indoline-3,2'-pyrrolidin]-2-one (5g)

White crystals; m.p. 209-211 $^\circ\text{C}$; IR (KBr) ν 3420, 3339, 3191, 3084, 1707, 1618, 1472, 1190, 754 cm^{-1} ; ^1H NMR (400 MHz, DMSO- d_6) δ 9.91 (s, 1H), 8.35 (d, $J = 4.4$ Hz, 1H), 7.69 (d, $J = 7.2$ Hz, 1H), 7.08-7.58 (m, 9H), 6.65 (d, $J = 7.6$ Hz, 1H), 6.49 (d, $J = 8.0$ Hz, 1H), 5.43 (dd, $J = 10.4, 5.6$ Hz, 1H), 5.09 (t, $J = 11.2$ Hz, 1H), 4.32 (d, $J = 11.2$ Hz, 1H), 4.08 (d, $J = 5.6$ Hz, 1H), 1.71 (s, 3H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 206.9, 180.2, 156.2, 149.0, 143.7, 142.9, 136.2, 131.2, 129.3, 128.9, 128.2, 127.6,

124.3, 122.58, 122.50, 122.2, 109.6, 71.0, 59.8, 57.3, 55.5, 31.3; HRMS (TOF-ES+) m/z: [M+Na]⁺ calcd for C₂₄H₂₁N₃O₂Na 406.1531, found 406.1516.

3'-Acetyl-4'-(2-nitrophenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one (4h)

White solid (conditions A: 131.6 mg, yield 62%; conditions B: 99.8 mg, yield 47%); m.p. 213-214°C; IR (KBr) ν 3393, 3321, 1715, 1620, 1484, 1440, 1347, 1185, 844, 750, 702 cm⁻¹; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.65 (s, 1H), 8.13 (d, *J* = 8.4 Hz, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.20-7.30 (m, 7H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.89 (d, *J* = 7.6 Hz, 1H), 4.88 (t, *J* = 4.4 Hz, 1H), 4.06 (d, *J* = 4.8 Hz, 1H), 3.91-3.92 (m, 2H), 1.47 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 204.5, 182.2, 148.1, 146.9, 142.4, 141.3, 130.9, 130.1, 129.7, 128.6, 128.0, 127.5, 126.1, 124.0, 122.4, 110.1, 67.7, 67.33, 67.26, 54.9, 30.4; HRMS (TOF-ES+) m/z: [M+H]⁺ calcd for C₂₅H₂₂N₃O₄ 428.1610, found 428.1606.

4'-Acetyl-3'-(2-nitrophenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one (5h)

White solid (conditions A: 64.8 mg, yield 30%; conditions B: 81.7 mg, yield 38%); m.p. 213-214°C; IR (KBr) ν 3339, 3155, 3074, 3033, 2891, 1701, 1622, 1521, 1471, 1347, 753, 703 cm⁻¹; ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.97 (s, 1H), 7.98 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 7.2 Hz, 1H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.09-7.37 (m, 7H), 6.59 (d, *J* = 7.6 Hz, 1H), 5.52 (dd, *J* = 10.4, 5.6 Hz, 1H), 4.90 (t, *J* = 11.2 Hz, 1H), 4.31 (d, *J* = 11.6 Hz, 1H), 4.21 (d, *J* = 5.6 Hz, 1H), 1.68 (s, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 206.4, 180.4, 147.1, 144.5, 143.4, 142.5, 130.2, 129.7, 129.5, 129.0, 128.3, 127.9, 124.6, 123.4, 122.5, 109.7, 72.3, 60.2, 58.6, 53.8, 31.4; HRMS (TOF-ES+) m/z:

$[M+H]^+$ calcd for $C_{25}H_{22}N_3O_4$ 428.1610, found 428.1599.

3'-Acetyl-4'-(4-nitrophenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one (4i)

White solid (conditions A: 139.0 mg, yield 65%; conditions B: 104.1 mg, yield 49%); m.p. 267-268°C; IR (KBr) ν 3393, 3321, 2940, 2882, 1715, 1513, 1470, 1346, 1186, 750, 702 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, $J = 8.8$ Hz, 2H), 7.70 (*br s*, 1H), 7.51 (d, $J = 8.4$ Hz, 2H), 7.11-7.34 (m, 8H), 7.13 (t, $J = 7.6$ Hz, 1H), 6.93 (d, $J = 7.6$ Hz, 1H), 5.05 (d, $J = 10.4$ Hz, 1H), 4.12 (t, $J = 10.0$ Hz, 1H), 3.96 (d, $J = 10.8$ Hz, 1H), 1.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 180.3, 147.4, 143.0, 141.1, 140.5, 129.8, 129.0, 128.8, 128.6, 128.3, 124.1, 123.4, 123.3, 110.0, 72.5, 61.0, 58.9, 54.6, 31.3; HRMS (TOF-ES+) m/z : $[M+Na]^+$ calcd for $C_{25}H_{21}N_3O_4Na$ 450.1430, found 450.1448.

4'-Acetyl-3'-(4-nitrophenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one (5i)

White solid (conditions A: 59.6 mg, yield 28%; conditions B: 75.3 mg, yield 35%); m.p. 217-218°C; IR (KBr) ν 3411, 3340, 3074, 2891, 1698, 1621, 1521, 1471, 1346, 849, 752, 702 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, $J = 8.8$ Hz, 2H), 7.71 (d, $J = 6.8$ Hz, 1H), 7.53 (d, $J = 7.6$ Hz, 2H), 7.38 (t, $J = 7.2$ Hz, 2H), 7.18-7.33 (m, 6H), 6.65 (d, $J = 7.6$ Hz, 1H), 5.68 (d, $J = 10.8$ Hz, 1H), 4.75 (t, $J = 11.8$ Hz, 1H), 4.44 (d, $J = 11.2$ Hz, 1H), 1.68 (s, 3H); ^{13}C NMR (100 MHz, $DMSO-d_6$) δ 206.3, 180.3, 147.1, 144.5, 143.3, 142.5, 130.2, 129.7, 129.5, 128.9, 128.3, 127.9, 124.5, 123.3, 122.4, 109.8, 72.4, 60.3, 58.7, 53.9, 31.4; HRMS (TOF-ES+) m/z : $[M+H]^+$ calcd for $C_{25}H_{22}N_3O_4$ 428.1610, found 428.1626.

3'-Acetyl-5'-phenyl-4'-(*o*-tolyl)spiro[indoline-3,2'-pyrrolidin]-2-one (4j)

White solid (conditions A: 142.1 mg, yield 72%; conditions B: 110.5 mg, yield 56%);
m.p. 200-202°C; IR (KBr) ν 3346, 3195, 1710, 1623, 1471, 1357, 1180, 743, 702 cm^{-1} ;
 ^1H NMR (400 MHz, CDCl_3) δ 9.20-9.28 (m, 1H), 7.67 (d, $J = 8.0$ Hz, 1H), 7.39 (d, $J = 7.6$ Hz, 1H), 7.19-7.31 (m, 7H), 7.13 (t, $J = 7.6$ Hz, 1H), 7.07 (t, $J = 7.6$ Hz, 1H), 6.99 (t, $J = 8.0$ Hz, 2H), 4.90 (d, $J = 8.0$ Hz, 1H), 4.27 (t, $J = 10.8$ Hz, 1H), 4.08 (d, $J = 10.4$ Hz, 1H), 2.60 (s, 1H), 1.98 (s, 3H), 1.55 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 204.7, 183.5, 140.54, 140.48, 140.45, 137.9, 137.2, 130.3, 130.2, 129.7, 128.3, 127.7, 126.7, 126.6, 126.3, 126.1, 123.6, 110.4, 69.5, 68.2, 68.0, 50.3, 30.7, 19.6;
HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{24}\text{N}_2\text{O}_2\text{Na}$ 419.1735, found 419.1739.

4'-Acetyl-5'-phenyl-3'-(*o*-tolyl)spiro[indoline-3,2'-pyrrolidin]-2-one (5j)

White solid (conditions A: 40.1 mg, yield 20%; conditions B: 73.7 mg, yield 37%);
m.p. 206-207°C; IR (KBr) ν 3380, 3203, 1709, 1620, 1471, 1360, 750, 700 cm^{-1} ; ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.98 (s, 1H), 7.64 (d, $J = 7.2$ Hz, 1H), 7.59 (d, $J = 6.8$ Hz, 2H), 7.51 (d, $J = 7.6$ Hz, 1H), 7.35 (t, $J = 7.2$ Hz, 2H), 7.26 (t, $J = 7.2$ Hz, 1H), 6.96-7.14 (m, 4H), 6.89 (d, $J = 7.2$ Hz, 1H), 6.55 (d, $J = 7.2$ Hz, 1H), 5.57 (dd, $J = 8.8$, 6.0 Hz, 1H), 4.55-4.63 (m, 2H), 4.08 (d, $J = 5.6$ Hz, 1H), 2.03 (s, 3H), 1.55 (s, 3H); ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 206.9, 181.7, 143.5, 142.6, 137.7, 135.2, 130.8, 130.4, 129.2, 128.9, 128.3, 127.8, 127.4, 127.0, 126.1, 124.8, 121.6, 109.6, 72.4, 61.8, 60.5, 48.9, 31.6, 20.1; HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{26}\text{H}_{24}\text{N}_2\text{O}_2\text{Na}$ 419.1735, found 419.1714.

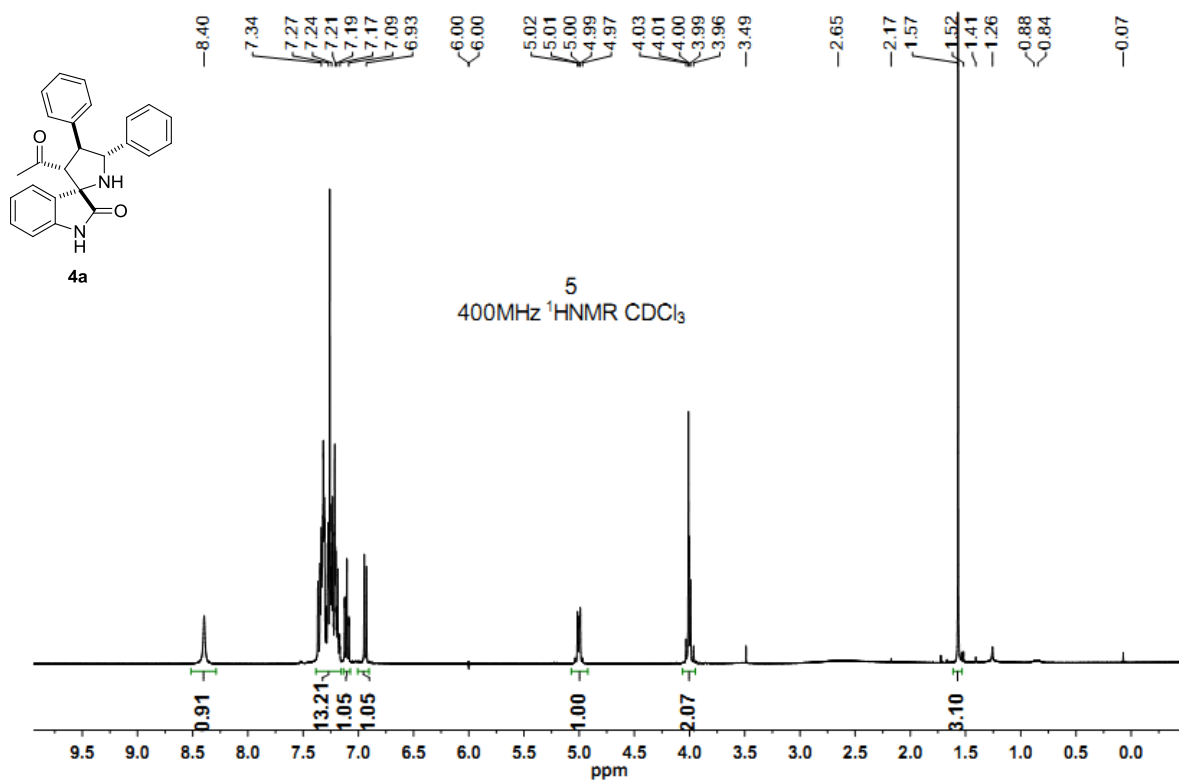
**3'-Acetyl-4'-(4-hydroxyphenyl)-5'-phenylspiro[indoline-3,2'-pyrrolidin]-2-one
(4k)**

White solid (conditions A: 187.1 mg, yield 94%; conditions B: 162.4 mg, yield 82%);
m.p. 230-231°C; IR (KBr) ν 3332, 3060, 3030, 1717, 1619, 1470, 1187, 752, 699 cm^{-1} ;
 ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.64 (s, 1H), 9.30 (s, 1H), 7.39-7.41 (m, 7H),
7.05-7.21 (m, 3H), 6.95 (d, $J = 7.6$ Hz, 1H), 6.73 (d, $J = 8.4$ Hz, 2H), 4.84 (dd, $J =$
10.0, 5.2 Hz, 1H), 3.93 (d, $J = 5.2$ Hz, 1H), 3.83 (d, $J = 10.8$ Hz, 1H), 3.74 (t, $J = 10.4$
Hz, 1H), 1.56 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 204.9, 182.5, 156.5, 142.3,
131.3, 129.8, 129.5, 129.4, 128.4, 127.6, 127.5, 126.2, 122.3, 115.6, 110.0, 67.8, 67.7,
67.3, 54.3, 30.8; HRMS (TOF-ES+) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_3\text{Na}$ 421.1528,
found 421.1547.

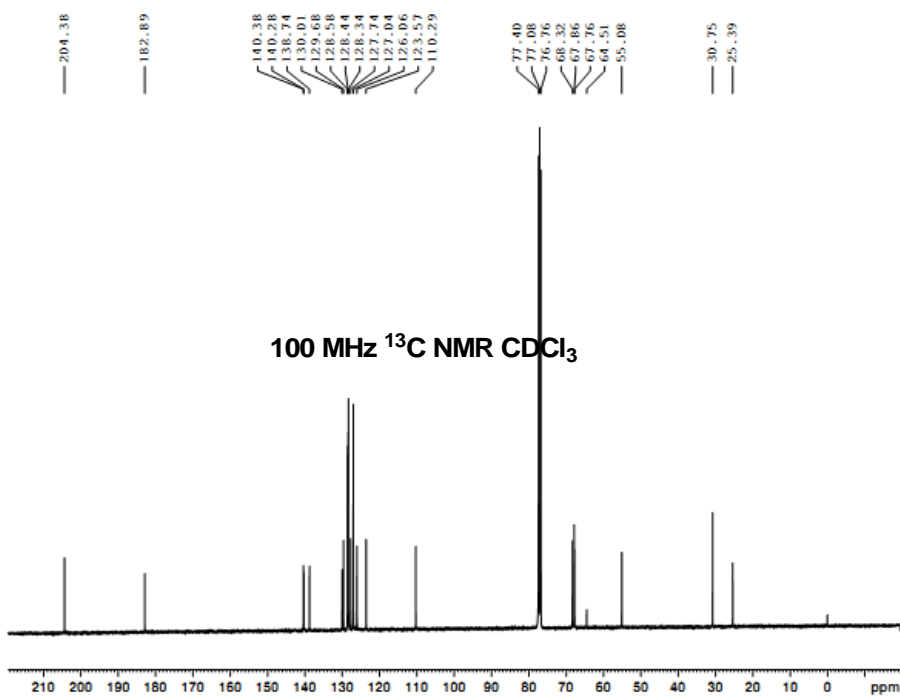
References

(1) Titu, D.; Chadha, A. *Tetrahedron: Asymmetry*, **2008**, *19*, 1698-1701.

Copies of NMR spectra for spiropyrrolidine oxindoles



NO. 7-zhongnan



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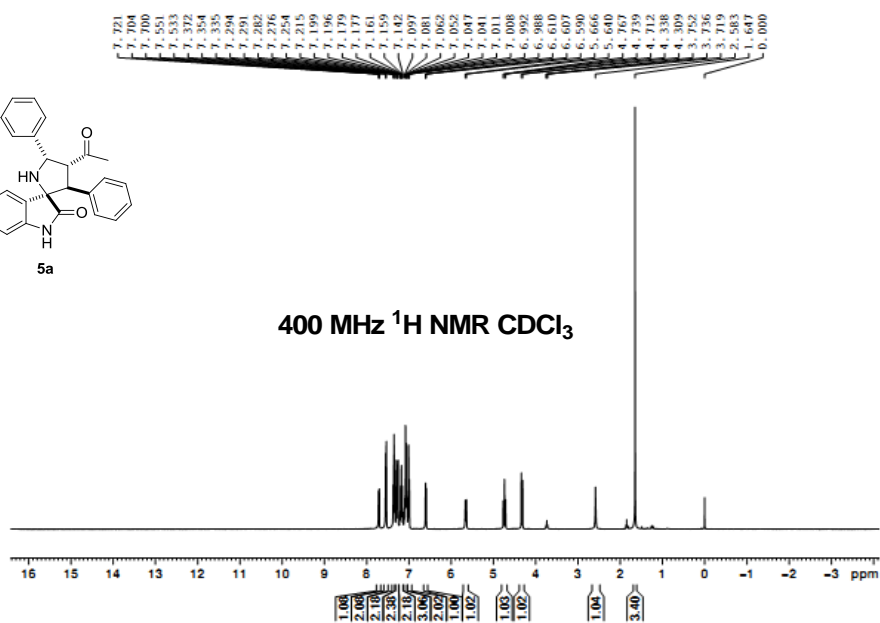
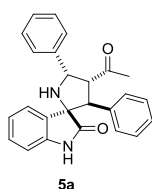
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EXPNO         7
PROCNO        1

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PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2160
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            198.33
DE            20.800 usec
TE            289.9 K
D1            2.0000000 sec
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===== 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         9.26999989 W
PLW12        0.22673000 W
PLW13        0.18945000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127690 MHz
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Current Data Parameters
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EXPNO    7
PROCNO   1

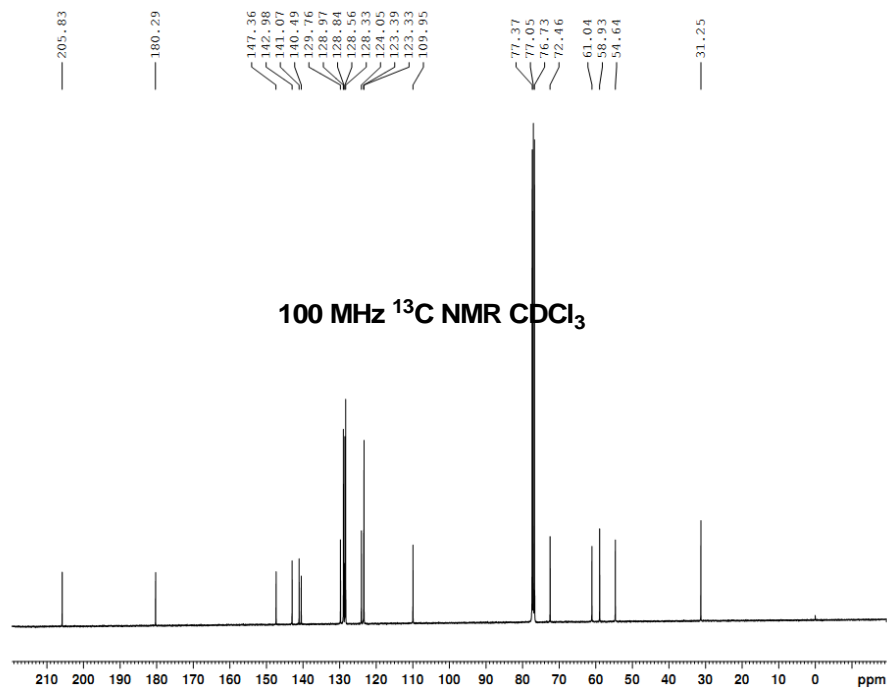
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PULPROG zg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH      8223.685 Hz
FIDRES   0.125492 Hz
AQ        3.9846287 sec
RG        106.96
DM        60.800 usec
DE        6.50 usec
TE        297.8 K
D1        1.00000000 sec

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

F2 - Processing parameters
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WDW       EM
SSB       0
LB        0.30 Hz
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PC        1.00

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NO. 4



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Current Data Parameters
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EXPNO    7
PROCNO   1

F2 - Acquisition Parameters
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Time     3.12
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
NS        10240
DS        4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ        1.3631998 sec
RG        198.33
DW        20.800 usec
DE        6.50 usec
TE        295.7 K
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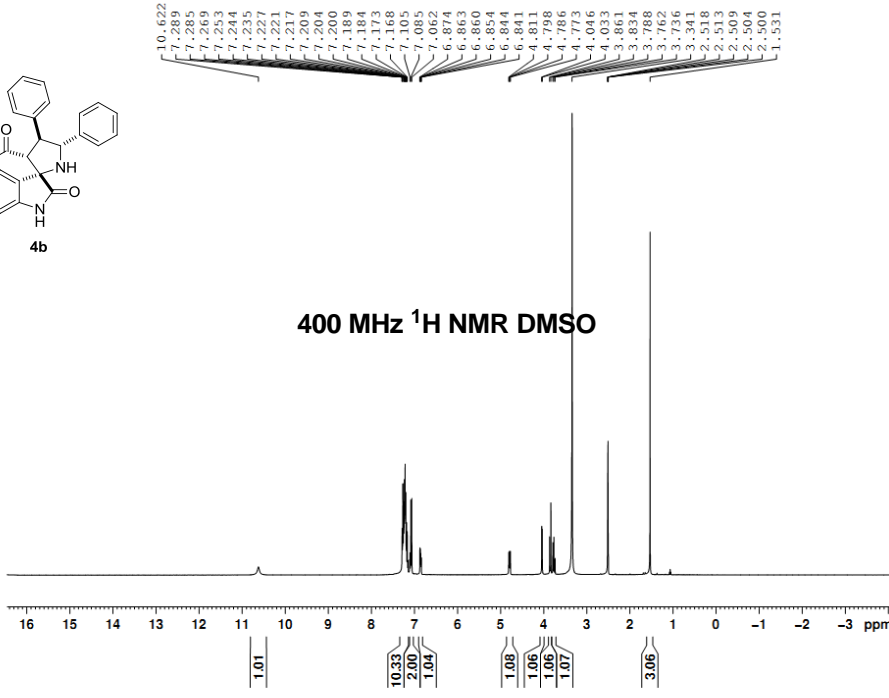
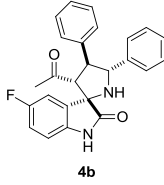
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PLW1      50.09999847 W
SFO1      100.6228293 MHz

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CPDPRG2  waltz16
NUC2      1H
PCPD2    90.00 usec
PLM2     9.36999989 W
PLW12    0.22673000 W
PLW13    0.18365000 W
SFO2     400.1316005 MHz

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

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PC-10



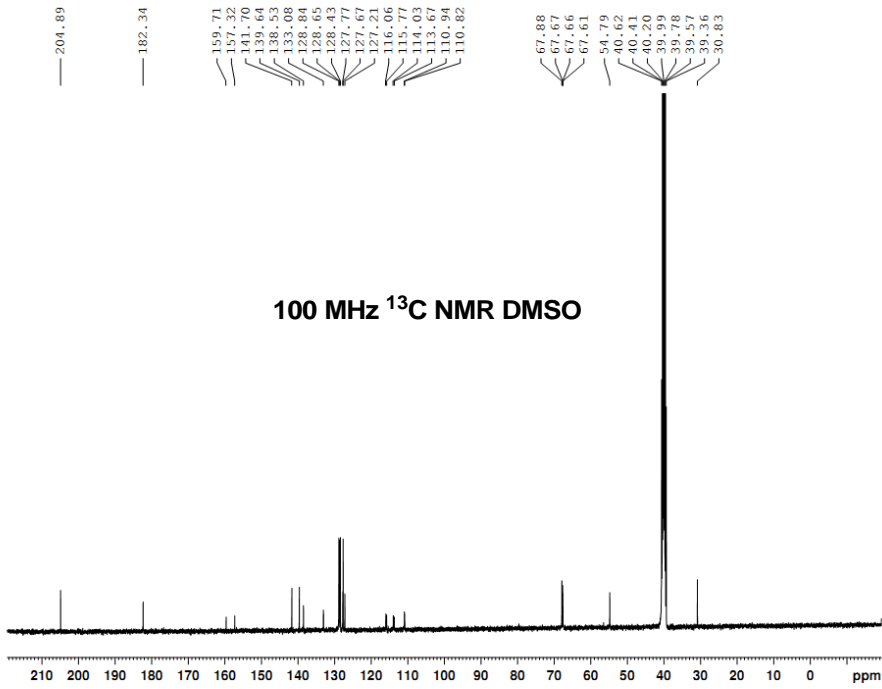
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 NAME 07-22-2013
 EXPNO 9
 PROCNO 1

F2 - Acquisition Parameters
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 40
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 198.33
 DM 60.800 usec
 DE 6.50 usec
 TE 297.4 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

PC-10



Current Data Parameters
 NAME 07-22-2013
 EXPNO 15
 PROCNO 1

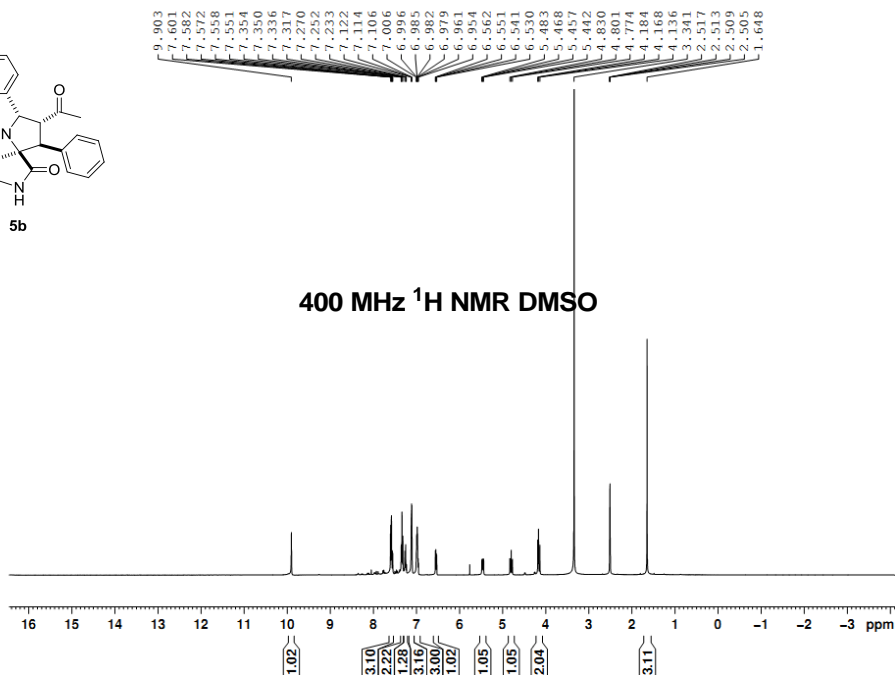
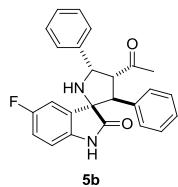
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 Date_ 20130724
 Time 4.46
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 198.33
 DM 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 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 9.36999959 W
 PLW12 0.22673000 W
 PLW13 0.18365000 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 2766
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

PC-7



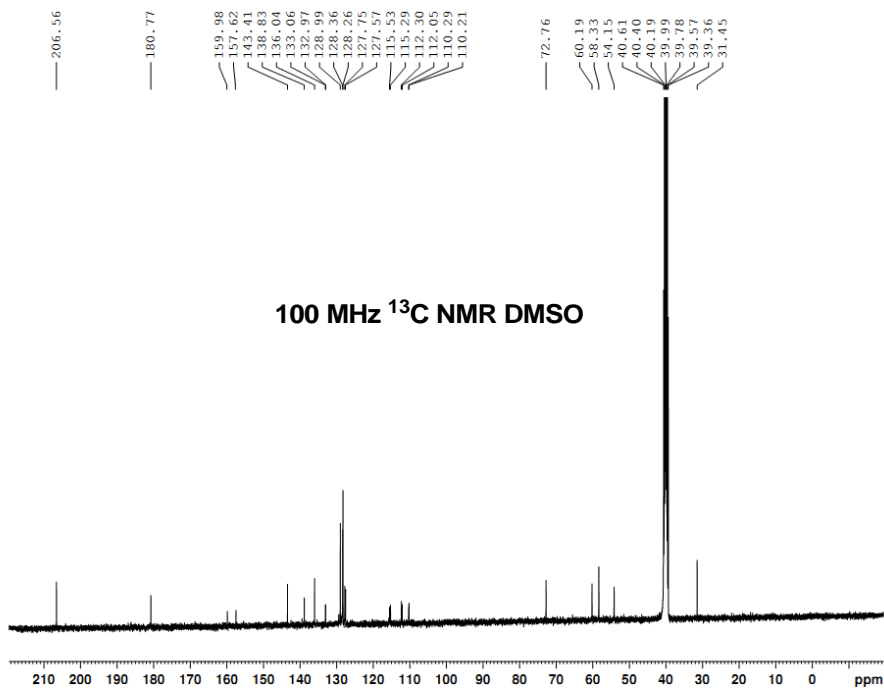
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EXPNO    3
PROCNO   1

F2 - Acquisition Parameters
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Time     15.28
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PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       64
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       198.33
DM       60.800 usec
DE       6.50 usec
TE       297.5 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
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PC-7



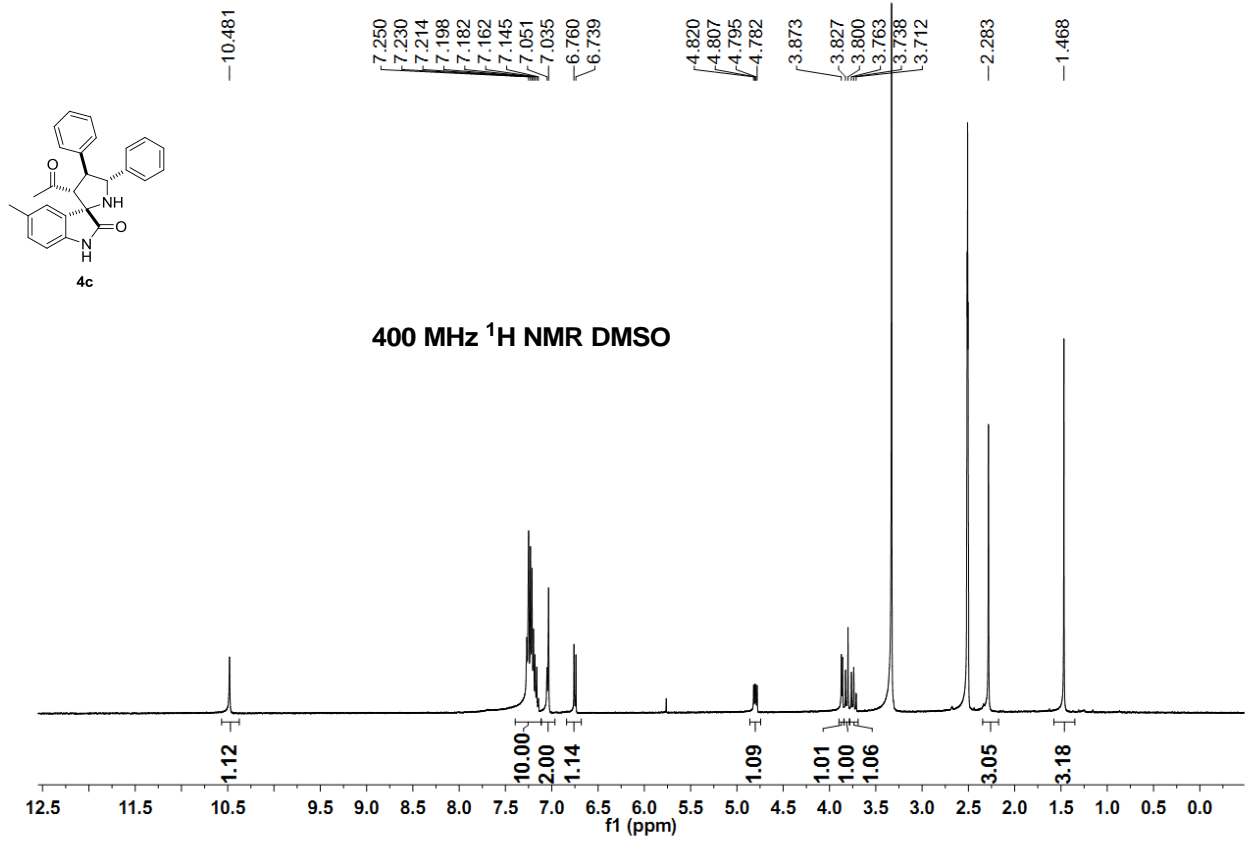
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EXPNO    13
PROCNO   1

F2 - Acquisition Parameters
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Time     19.11
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       3000
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       168.03
DM       20.800 usec
DE       6.50 usec
TE       297.9 K
D1       2.00000000 sec
D11      0.03000000 sec

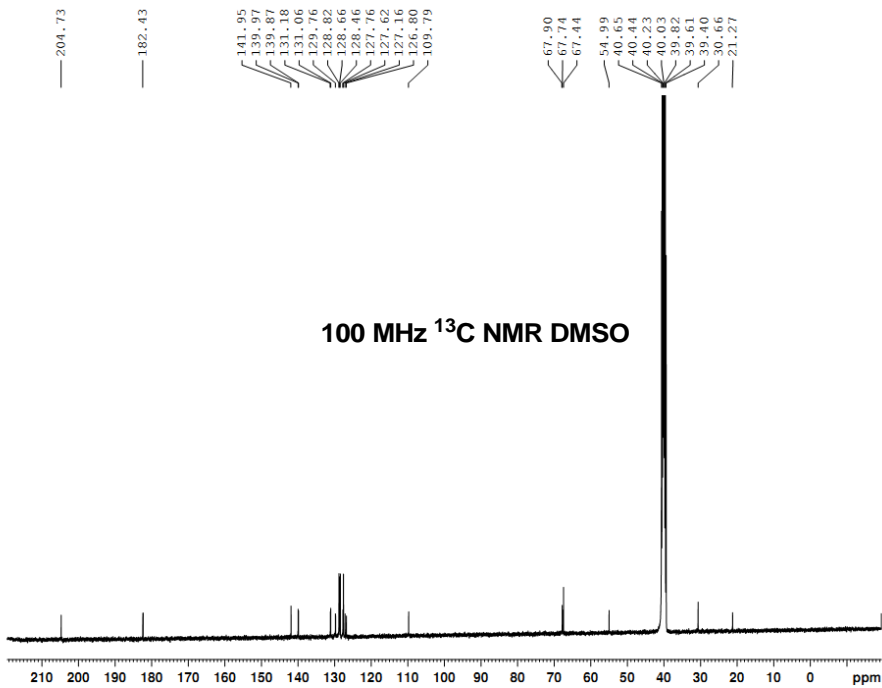
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PLW1    50.09999847 W
SFO1    100.6228293 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2   90.00 usec
PLW2    9.36999959 W
PLW12   0.22673000 W
PLW13   0.18365000 W
SFO2    400.1316005 MHz

F2 - Processing parameters
SI       30766
SF       100.6127690 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
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PC-16



Current Data Parameters
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 EXPNO 6
 PROCNO 1

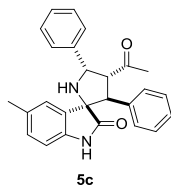
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 Time 9.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 14352
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 152.51
 DW 20.800 usec
 DE 6.50 usec
 TE 298.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec

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

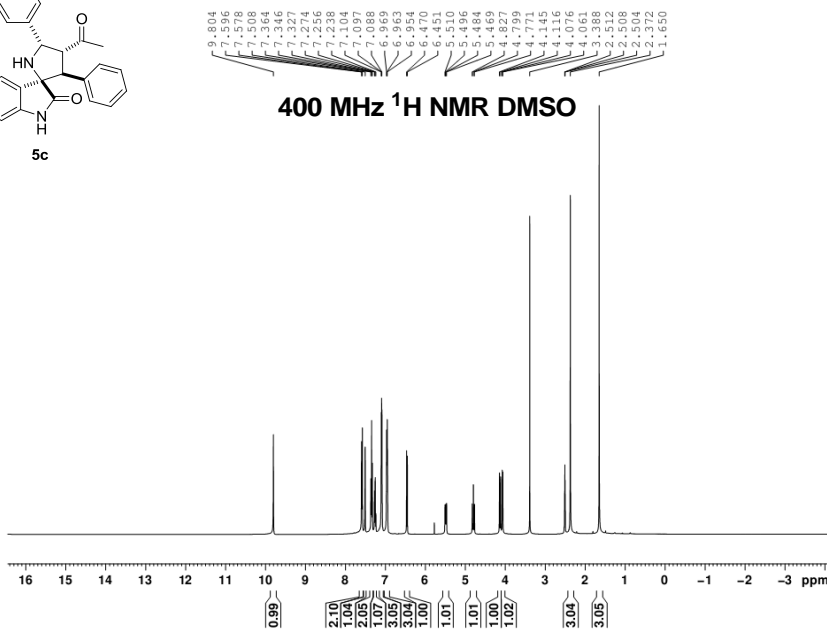
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 NUC2 1H
 PCPD2 90.00 usec
 PLW2 9.3699989 W
 PLW12 0.22673000 W
 PLW13 0.18365000 W
 SFO2 400.1316005 MHz

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

PC



400 MHz ¹H NMR DMSO



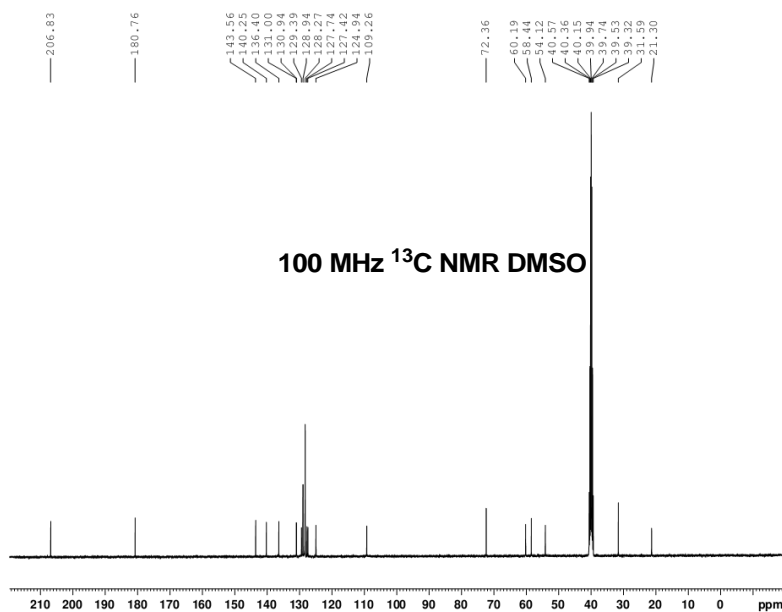
Current Data Parameters
 NAME 01-02-2014
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20140102
 Time 21.39
 INSTRUM spect
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 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 128
 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 290.4 K
 D1 1.0000000 sec

==== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PLW1 9.7299954 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

PC

100 MHz ¹³C NMR DMSO



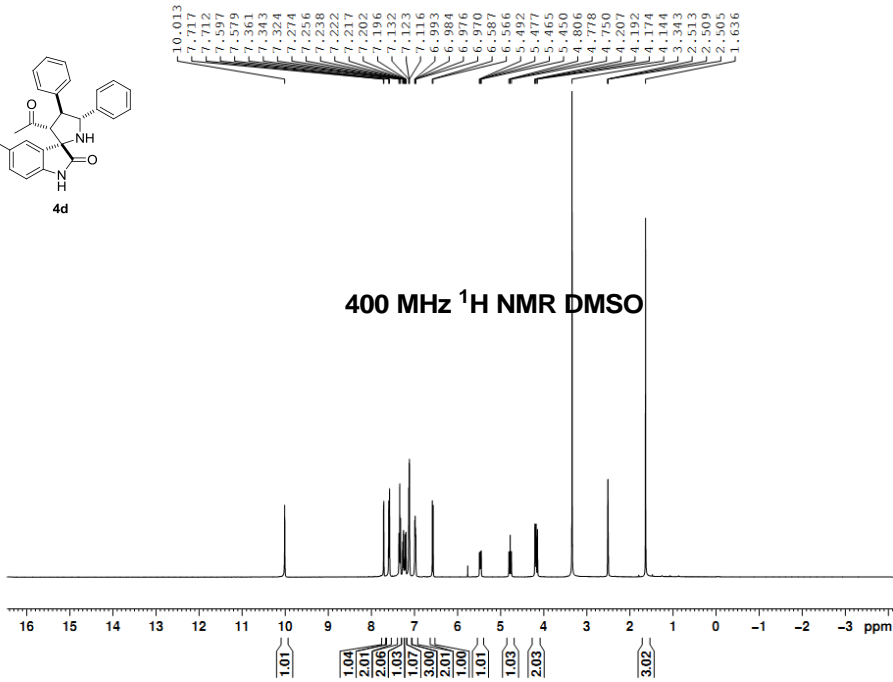
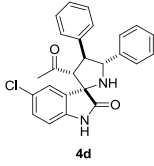
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 EXPNO 7
 PROCNO 1

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 Date_ 20140103
 Time 16.15
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 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 712
 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 292.5 K
 D1 2.0000000 sec
 D11 0.0300000 sec

==== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PLW1 50.0999847 W
 SFO1 100.6228293 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 FCFD2 90.00 usec
 PLW2 9.3699989 W
 PLW12 0.22673000 W
 PLW13 0.18365000 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
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 SF 100.6127690 MHz
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 SSB 0
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PC-11



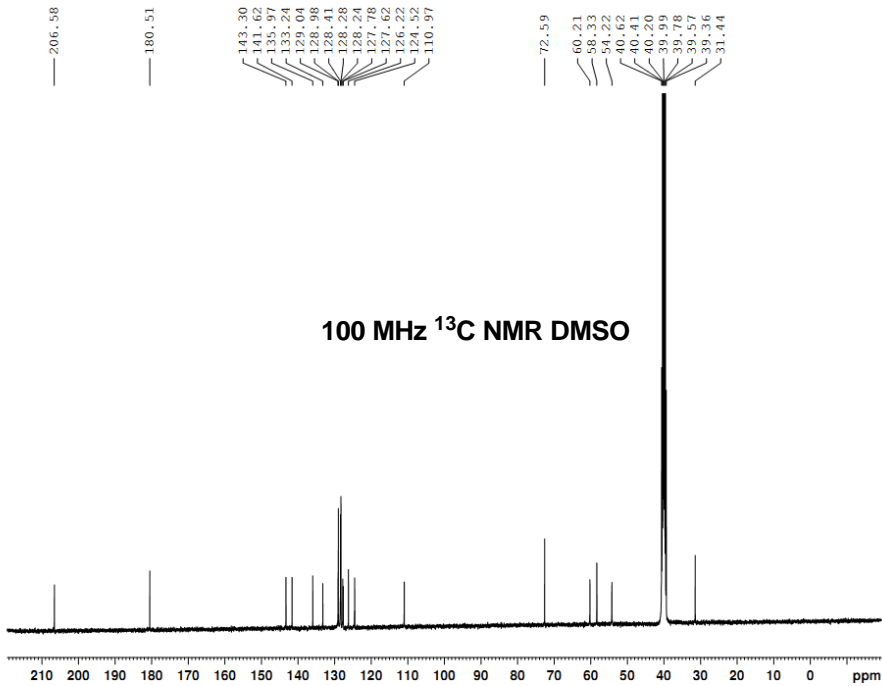
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 EXPNO 1
 PROCNO 1

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 PULPROG zg30
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 SOLVENT DMSO
 NS 32
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 168.03
 DM 60.800 usec
 DE 6.50 usec
 TE 297.6 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
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PC-11



Current Data Parameters
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 PROCNO 1

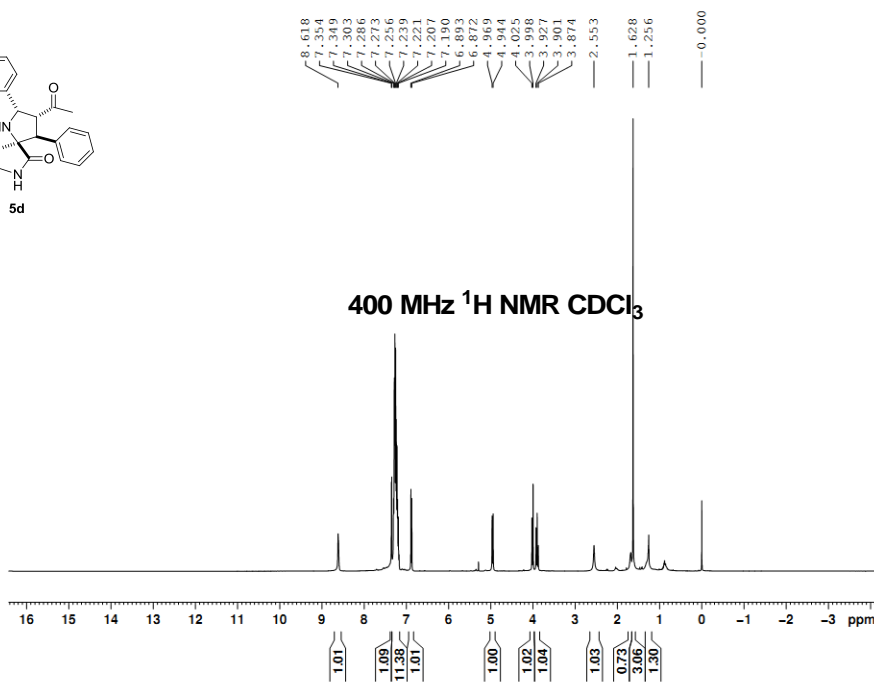
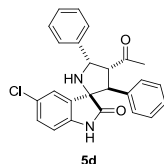
F2 - Acquisition Parameters
 Date_ 20130724
 Time 9.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 4668
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 168.03
 DM 20.800 usec
 DE 6.50 usec
 TE 298.1 K
 D1 2.00000000 sec
 D11 0.03000000 sec

----- 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 9.36999959 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
 FC 1.40

PC-40



```

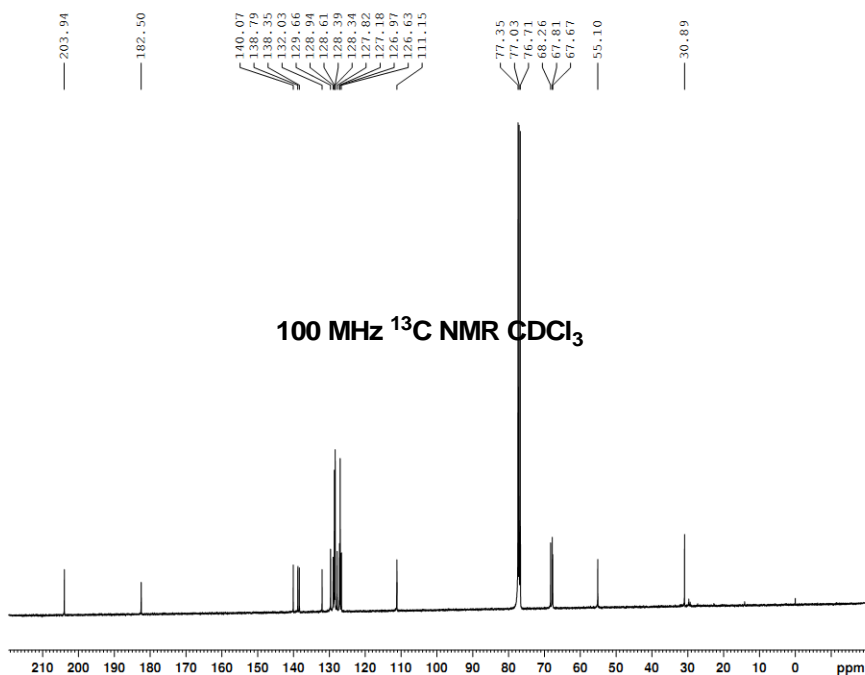
Current Data Parameters
NAME      10-11-2013
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20131011
Time     15.45
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
DM        60.800 usec
DE        6.50 usec
TE        296.5 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
    
```

PC-40



```

Current Data Parameters
NAME      10-11-2013
EXPNO    2
PROCNO   1

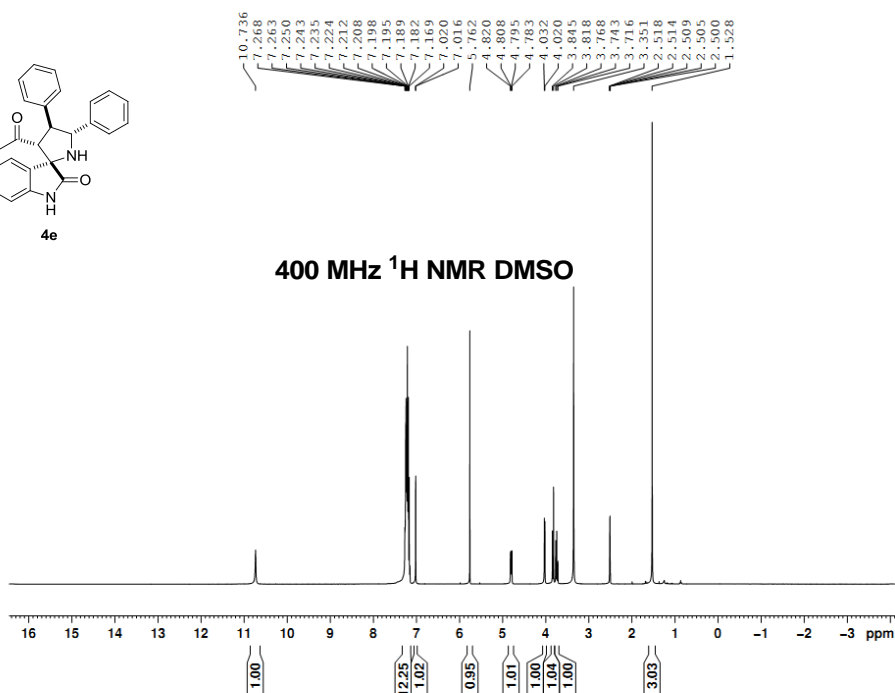
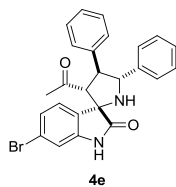
F2 - Acquisition Parameters
Date_    20131013
Time     8.24
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        14248
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        152.51
DM        20.800 usec
DE        6.50 usec
TE        297.8 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
    
```

PC-15



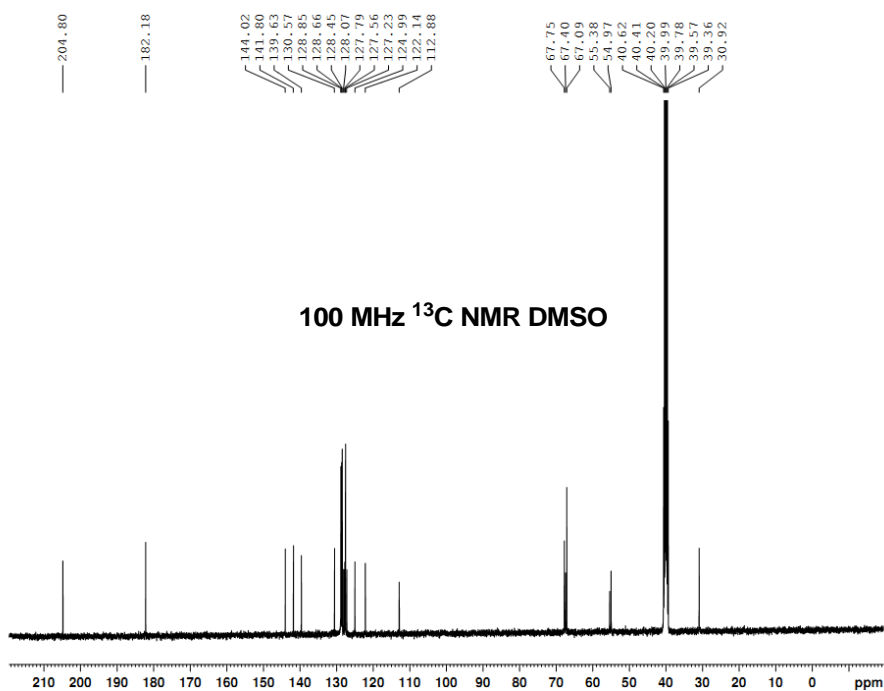
Current Data Parameters
 NAME 07-22-2013
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130722
 Time 19.16
 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 124.55
 DM 60.800 usec
 DE 6.50 usec
 TE 297.6 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
 FC 1.00

PC-15



Current Data Parameters
 NAME 07-22-2013
 EXPNO 18
 PROCNO 1

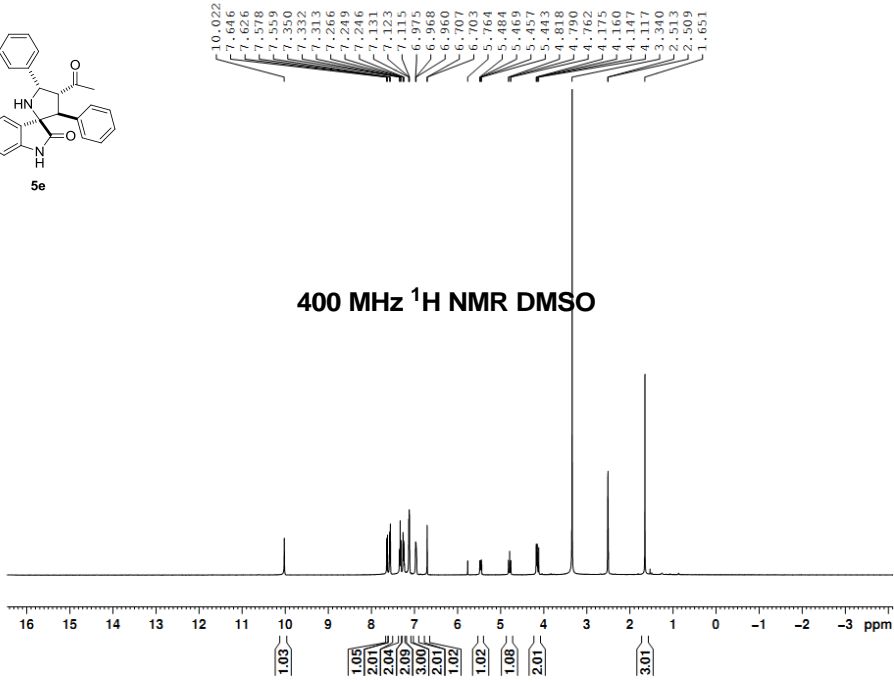
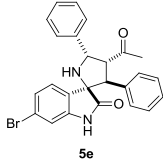
F2 - Acquisition Parameters
 Date_ 20130722
 Time 15.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 1228
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 198.33
 DM 20.800 usec
 DE 6.50 usec
 TE 297.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec

===== 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 9.36999959 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
 FC 1.40

PC-8



```

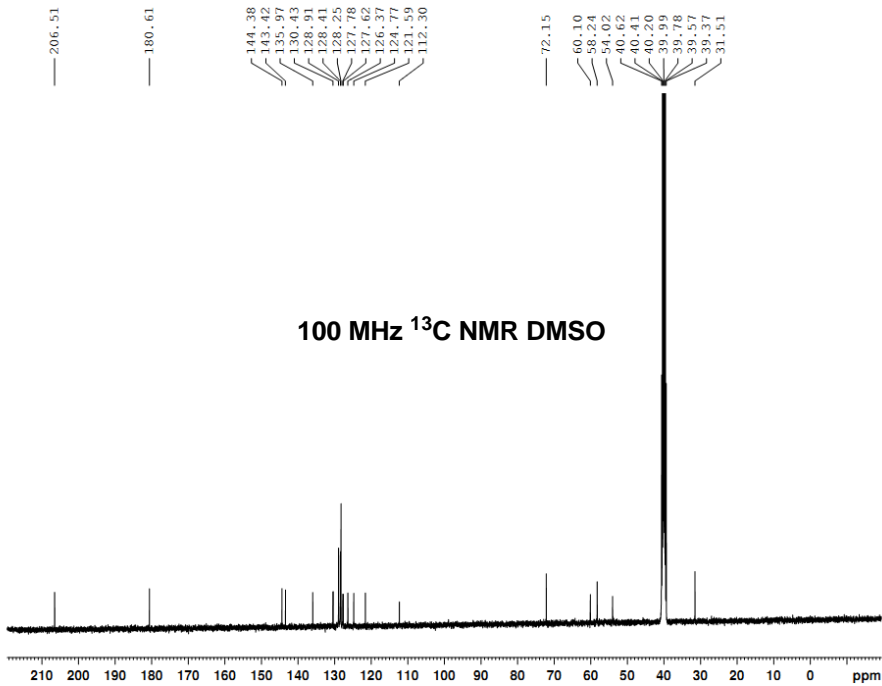
Current Data Parameters
NAME      07-22-2013
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20130722
Time     15.37
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       198.33
DM       60.800 usec
DE       6.50 usec
TE       297.3 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
    
```

PC-8



```

Current Data Parameters
NAME      07-22-2013
EXPNO    19
PROCNO   1

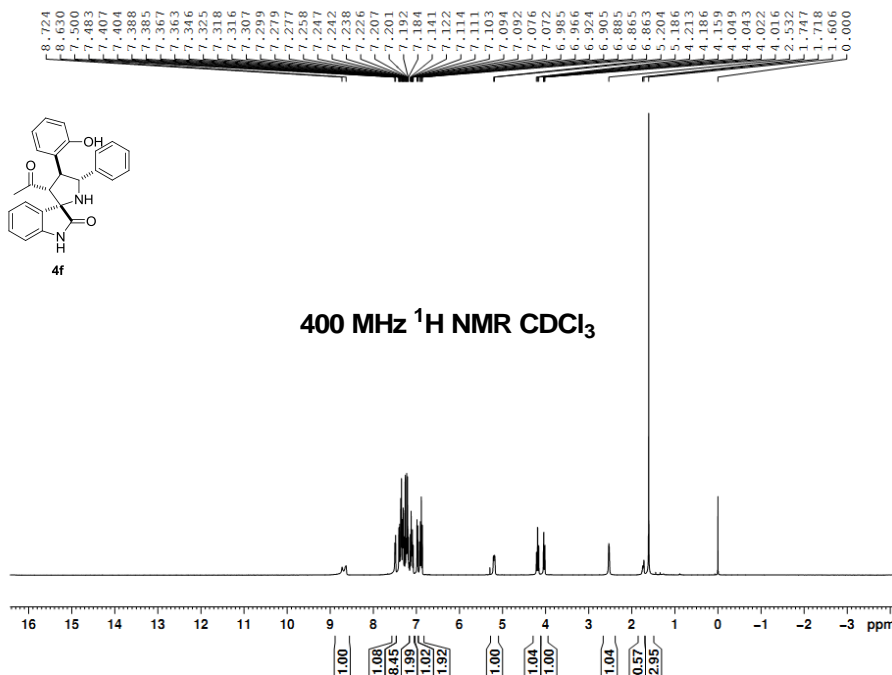
F2 - Acquisition Parameters
Date_    20130724
Time     14.51
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zgpg30
TD       65536
SOLVENT  DMSO
NS       1992
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       168.03
DM       20.800 usec
DE       6.50 usec
TE       298.4 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    9.36999959 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
    
```

NO. 2



```

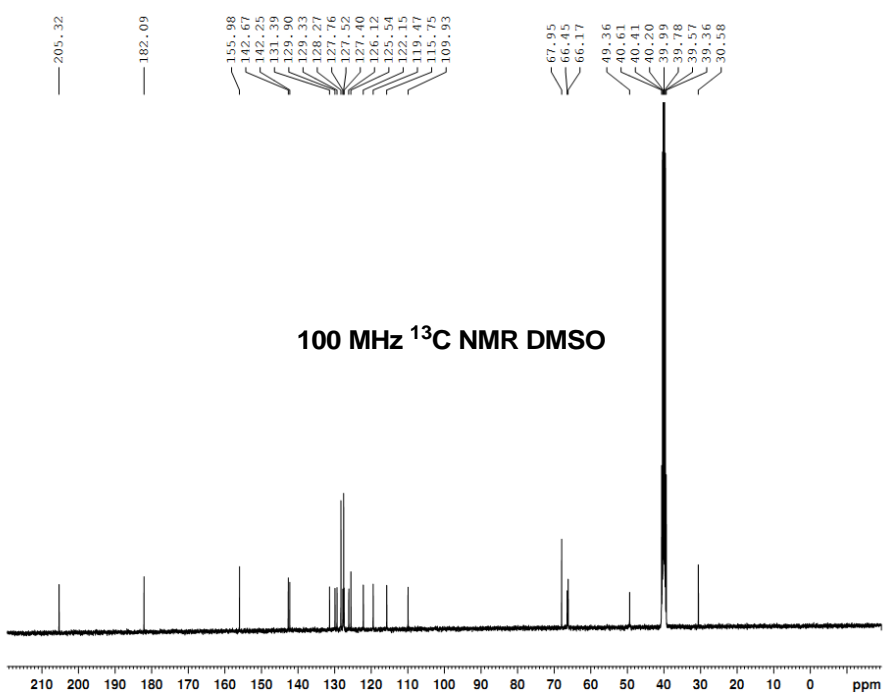
Current Data Parameters
NAME      03-30-2013
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20130330
Time     11.09
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       128.14
DM       60.800 usec
DE       6.50 usec
TE       291.7 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
  
```

PC-17



```

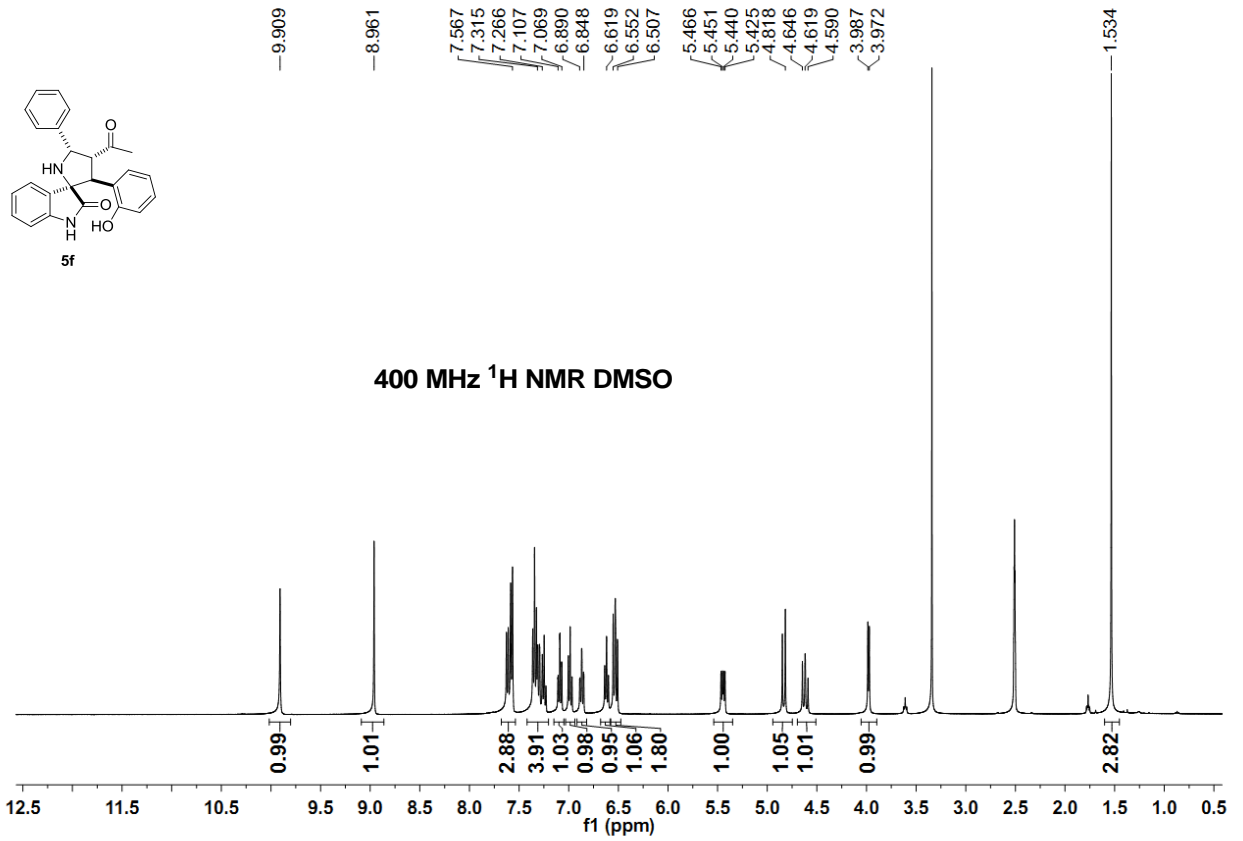
Current Data Parameters
NAME      08-20-2013
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20130823
Time     9.48
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       1568
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       198.33
DM       20.800 usec
DE       6.50 usec
TE       298.3 K
D1       2.00000000 sec
D11      0.03000000 sec

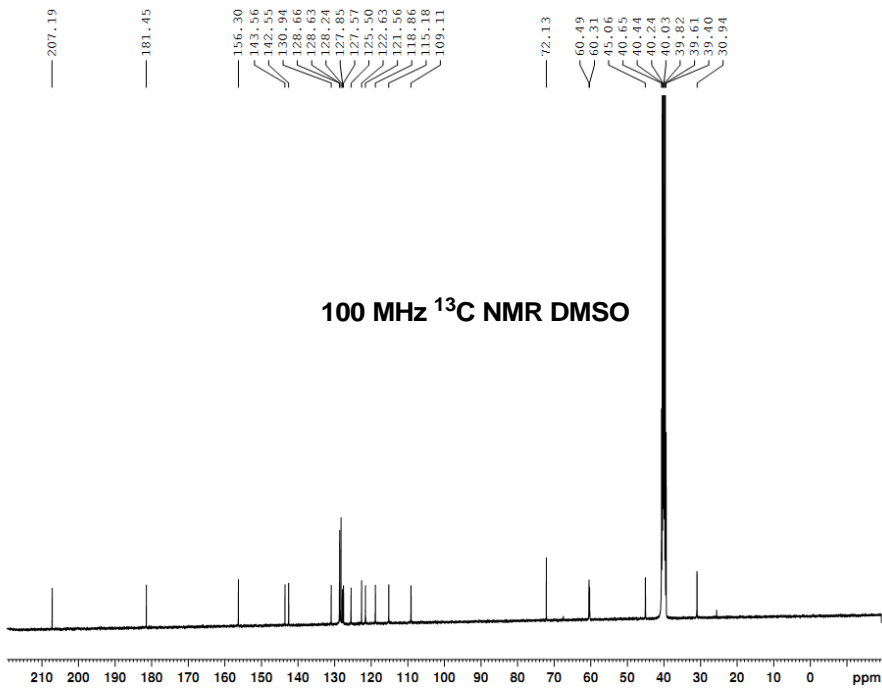
===== CHANNEL f1 =====
NUC1     13C
P1       9.00 usec
PLW1     50.09999847 W
SFO1     100.6228293 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    90.00 usec
PLW2     9.36999959 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
  
```

PC-1



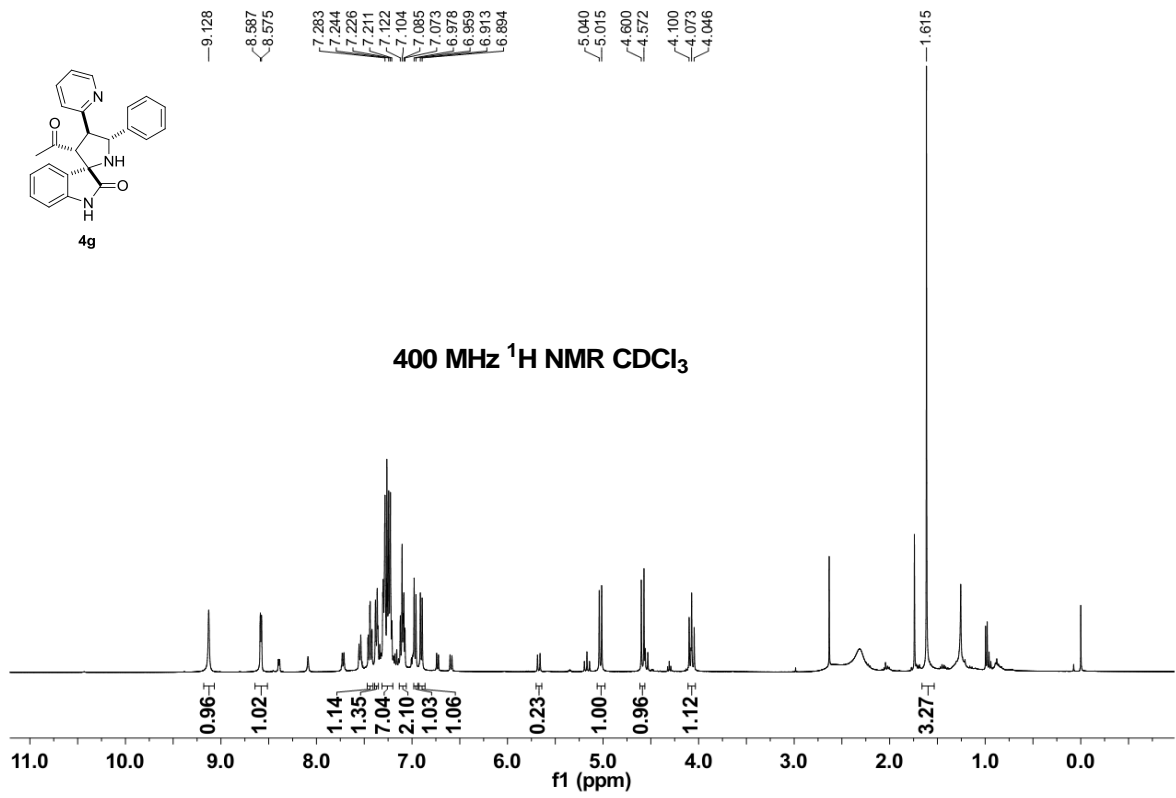
Current Data Parameters
 NAME 07-02-2013
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130704
 Time 3.16
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 10240
 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 299.5 K
 D1 2.0000000 sec
 D11 0.0300000 sec

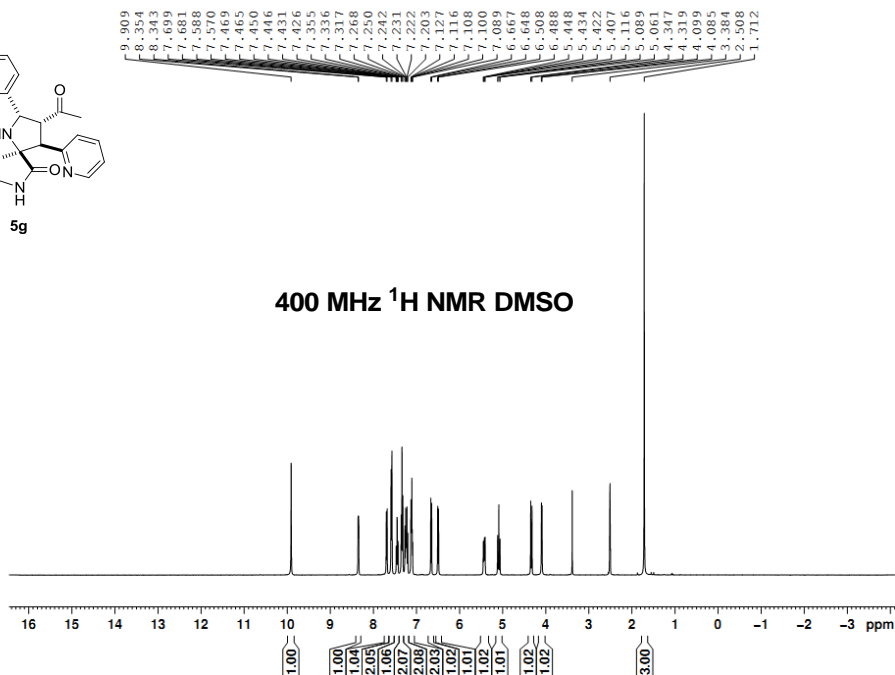
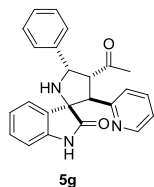
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PLW1 50.09999847 W
 SFO1 100.6228293 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PLW2 9.3699989 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
 GB 0 1.00 Hz
 PC 1.40



NO. 3



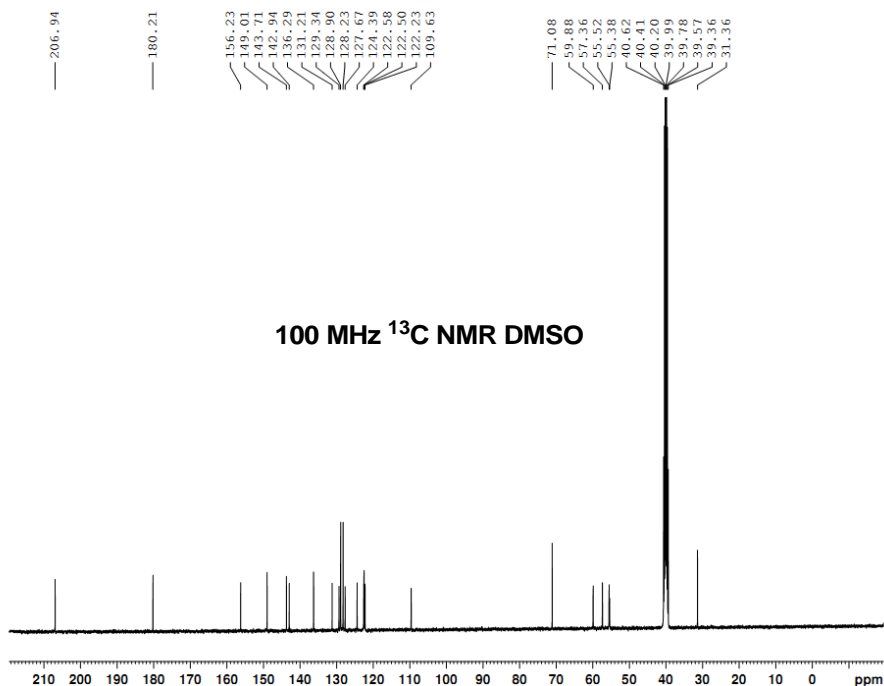
Current Data Parameters
 NAME 04-06-2013
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130408
 Time 13.57
 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 27.39
 DM 60.800 usec
 DE 6.50 usec
 TE 290.0 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 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

PC-9



Current Data Parameters
 NAME 07-22-2013
 EXPNO 14
 PROCNO 1

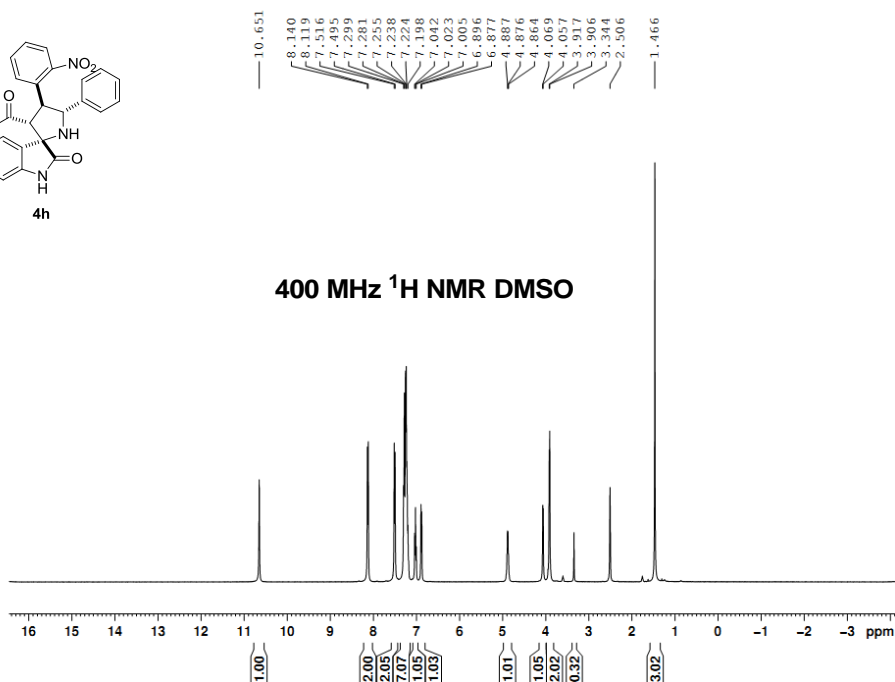
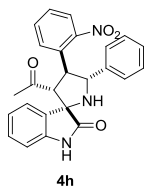
F2 - Acquisition Parameters
 Date_ 20130724
 Time 1.47
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 3072
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 198.33
 DM 20.800 usec
 DE 6.50 usec
 TE 298.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec

----- 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 9.36999899 W
 PLW12 0.22673000 W
 PLW13 0.18365000 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32766
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

NO. 5



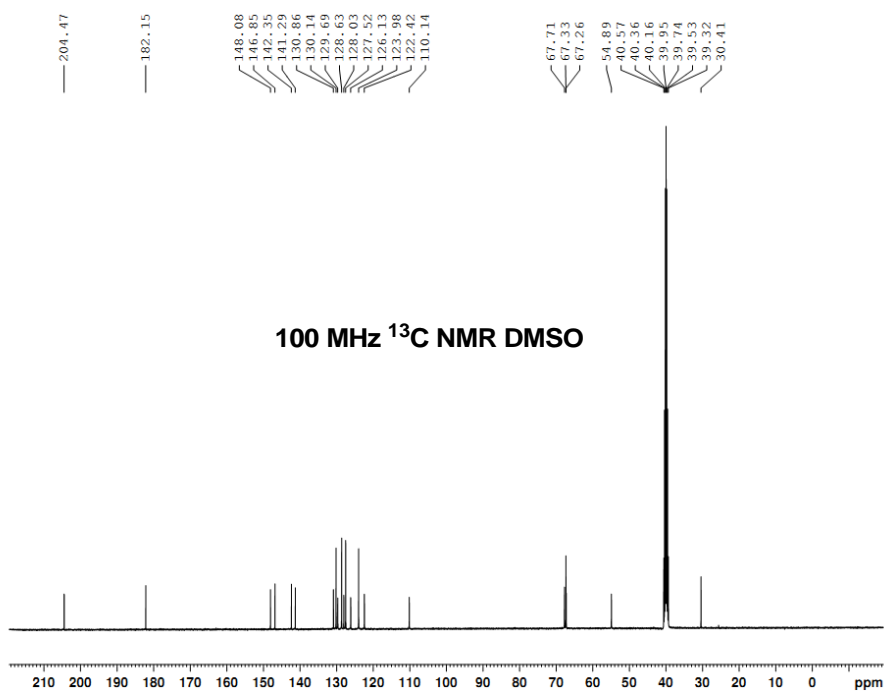
Current Data Parameters
 NAME 03-30-2013
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130330
 Time 14.28
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SMH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 24.96
 DM 60.800 usec
 DE 6.50 usec
 TE 297.8 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 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 FC 1.00

NO. 5



Current Data Parameters
 NAME 03-31-2013
 EXPNO 3
 PROCNO 1

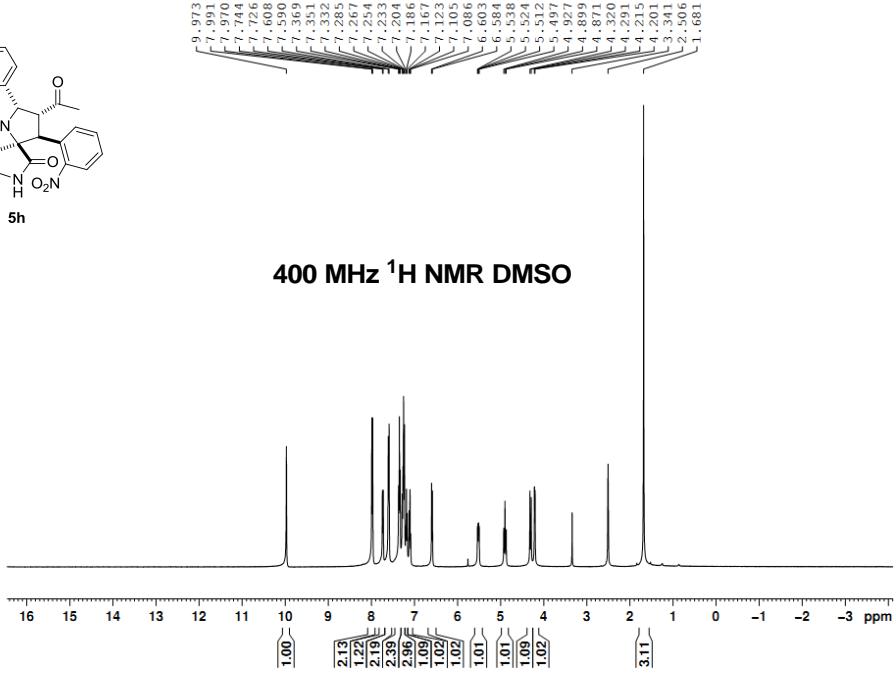
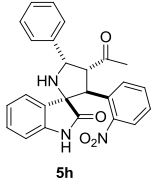
F2 - Acquisition Parameters
 Date_ 20130331
 Time 0.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 199.33
 DM 20.800 usec
 DE 6.50 usec
 TE 292.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec

===== 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 9.36999959 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
 FC 1.40

NO. 4



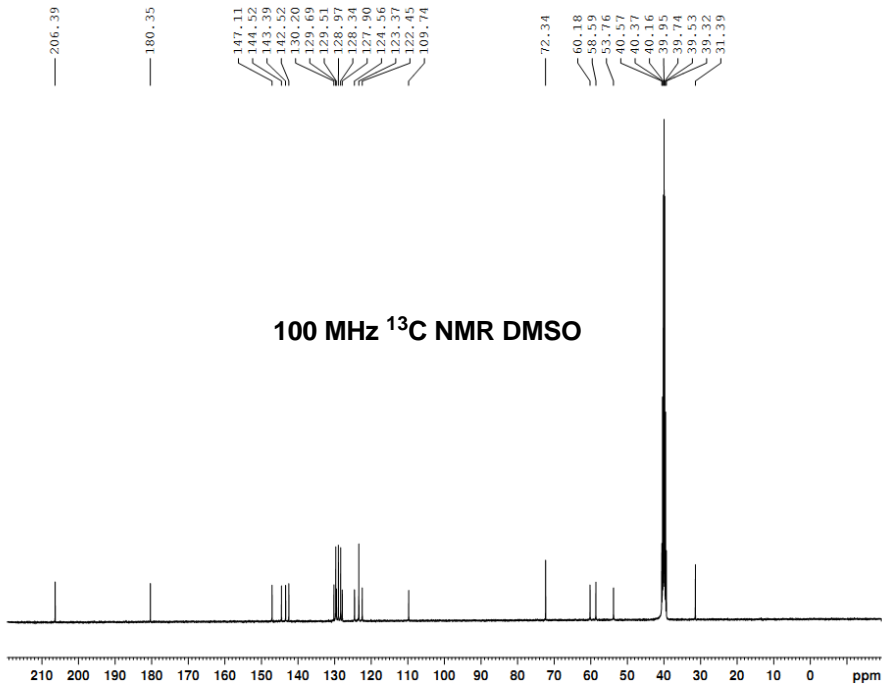
Current Data Parameters
 NAME 03-30-2013
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130330
 Time 14.23
 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 24.96
 DM 60.800 usec
 DE 6.50 usec
 TE 297.9 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
 FC 1.00

NO. 4



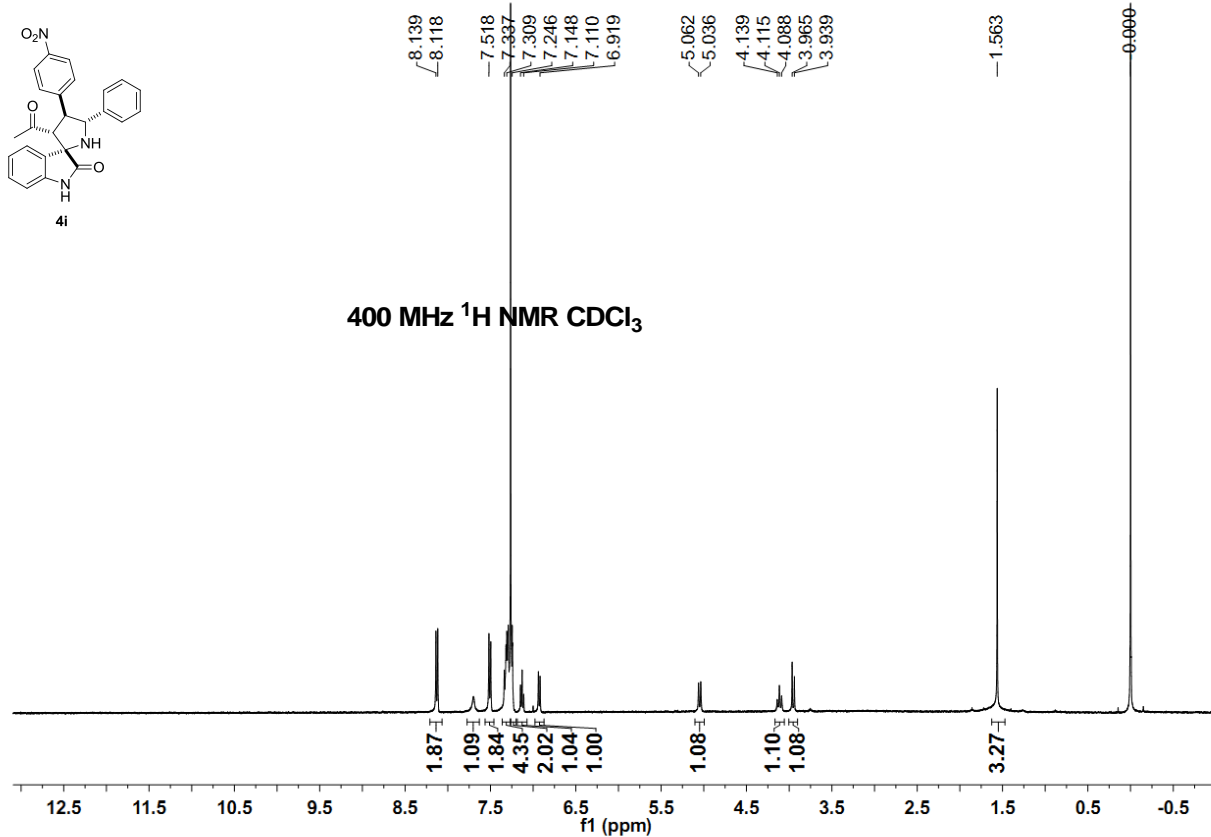
Current Data Parameters
 NAME 03-31-2013
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130330
 Time 22.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 198.33
 DM 20.800 usec
 DE 6.50 usec
 TE 292.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec

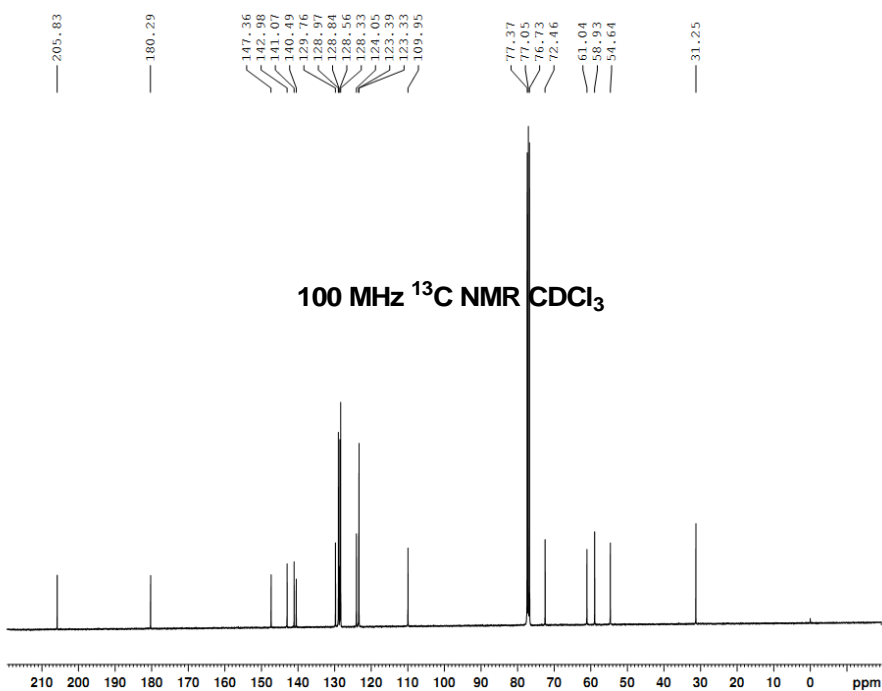
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PLW1 50.09999847 W
 SFO1 100.6228293 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PLW2 9.36999959 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
 FC 1.40



NO. 4



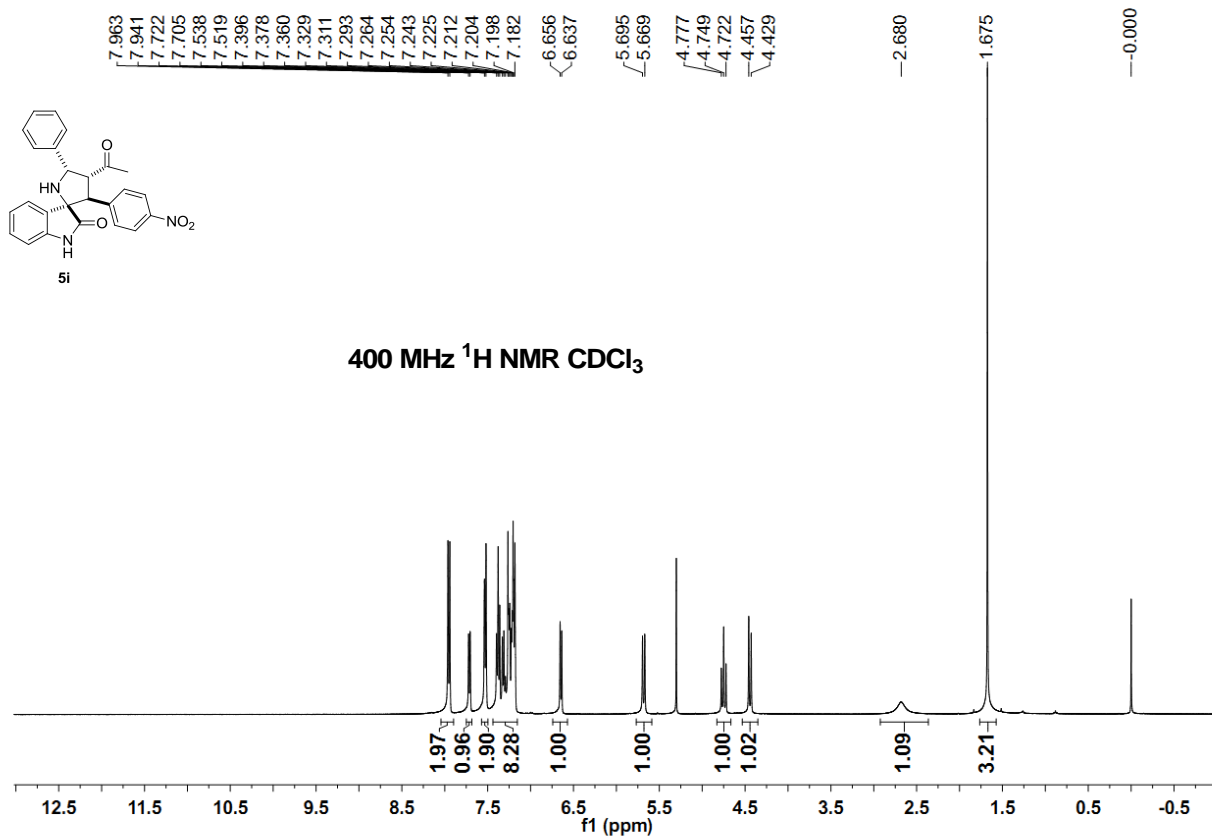
Current Data Parameters
 NAME 04-16-2013
 EXPNO 7
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130417
 Time 3.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 10240
 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 295.7 K
 D1 2.00000000 sec
 D11 0.03000000 sec

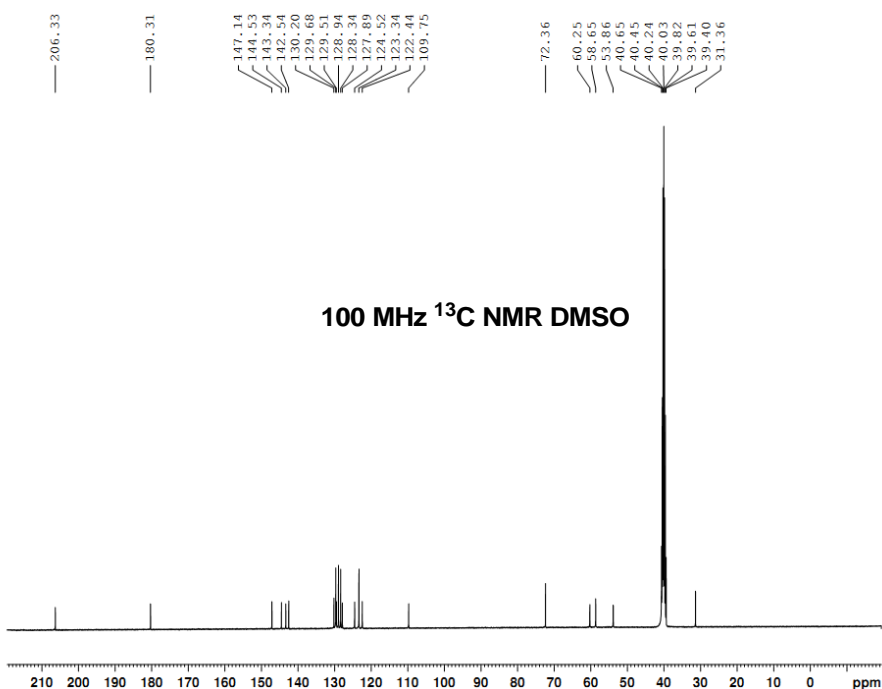
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PLW1 50.09999847 W
 SFO1 100.6228293 MHz

===== 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
 GB 0 1.00 Hz
 PC 1.40



PC-27



Current Data Parameters
NAME 09-04-2013
EXPNO 6
PROCNO 1

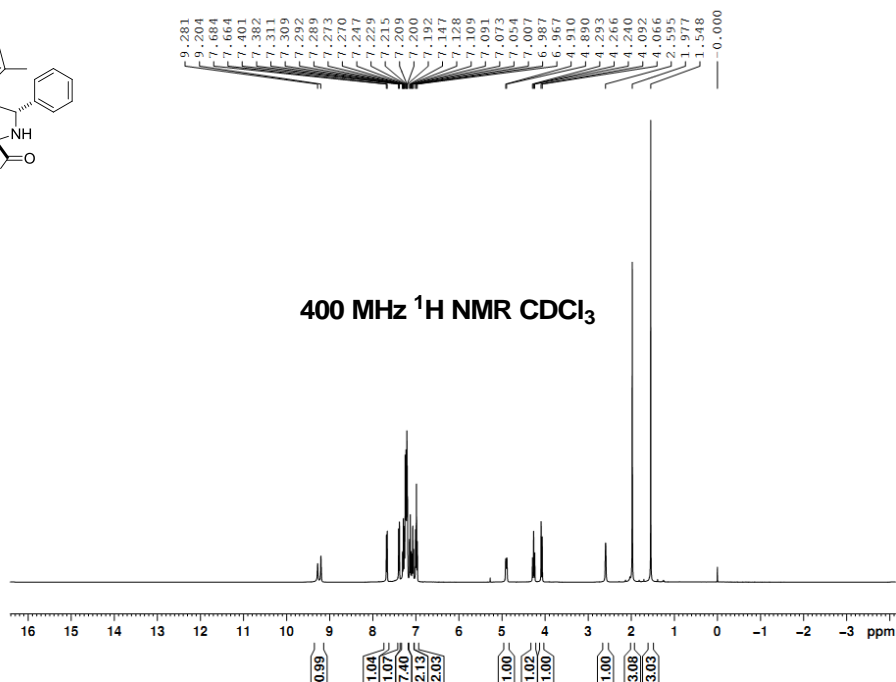
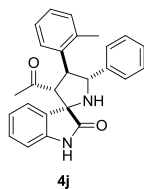
F2 - Acquisition Parameters
Date_ 20130905
Time 8.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 10240
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 198.33
DW 20.800 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
D11 0.03000000 sec

===== 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 9.36999899 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

NO. 2-ZN



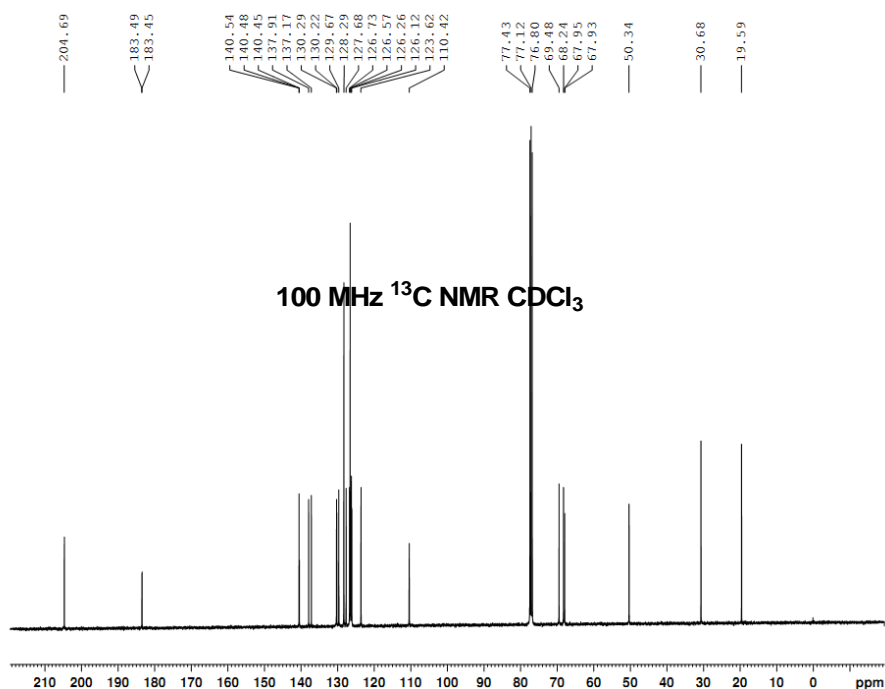
Current Data Parameters
NAME 01-16-2013
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130116
Time 11.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 60.33
DM 60.800 usec
DE 6.50 usec
TE 286.5 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 400.1300150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NO. 2-ZN



Current Data Parameters
NAME 01-16-2013
EXPNO 3
PROCNO 1

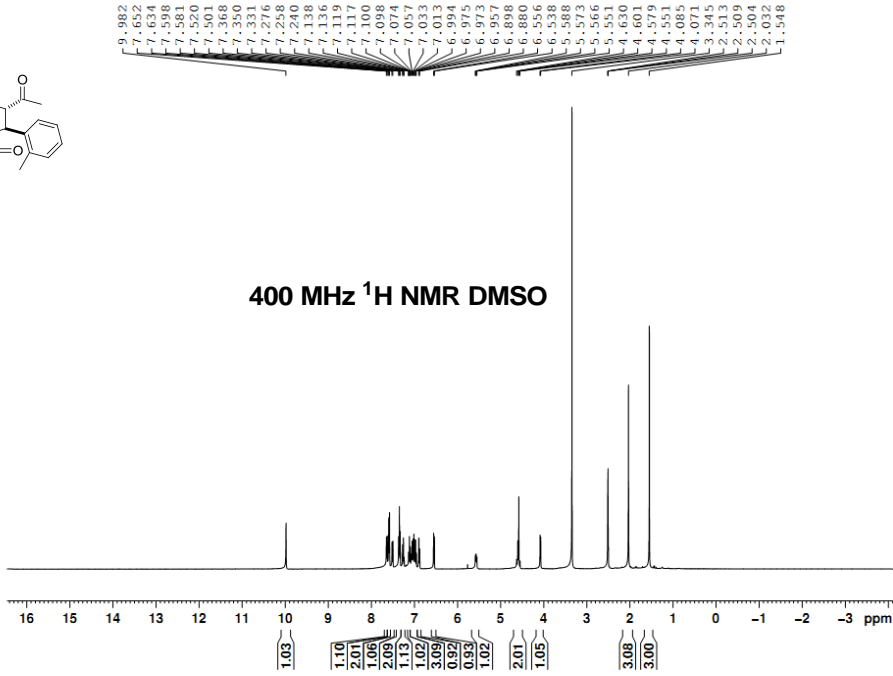
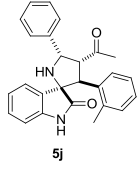
F2 - Acquisition Parameters
Date_ 20130116
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1656
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 138.14
DM 20.800 usec
DE 6.50 usec
TE 287.0 K
D1 2.00000000 sec
D11 0.03000000 sec

----- 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

NO. 4



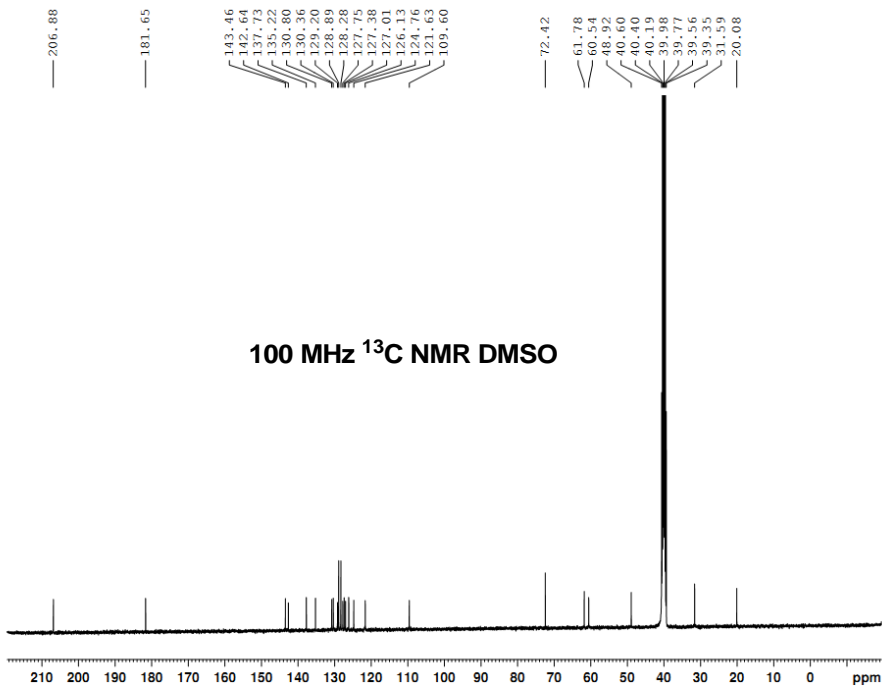
Current Data Parameters
 NAME 05-06-2013
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130508
 Time 14.48
 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 168.03
 DM 60.800 usec
 DE 6.50 usec
 TE 295.7 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
 FC 1.00

NO. 4



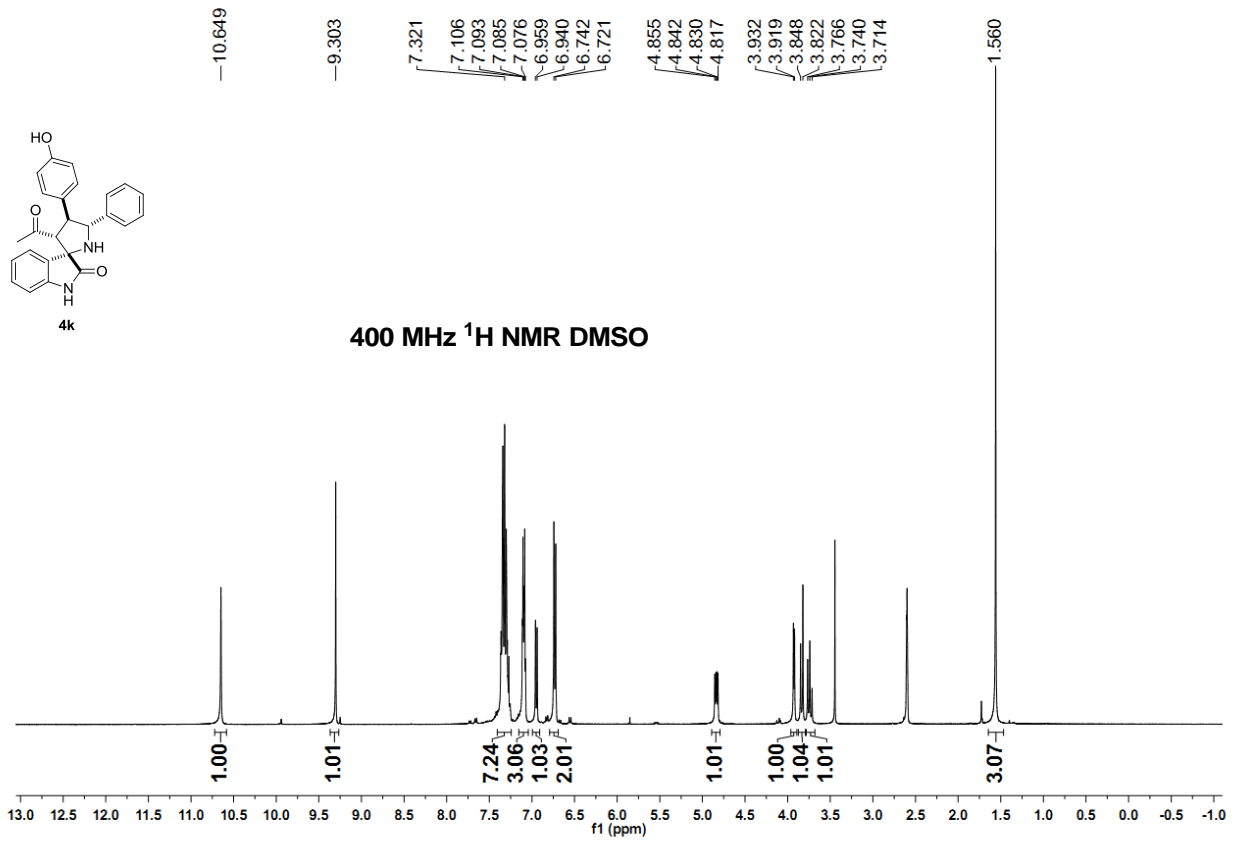
Current Data Parameters
 NAME 05-06-2013
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130510
 Time 2.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 5120
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 168.03
 DM 20.800 usec
 DE 6.50 usec
 TE 296.5 K
 D1 2.00000000 sec
 D11 0.03000000 sec

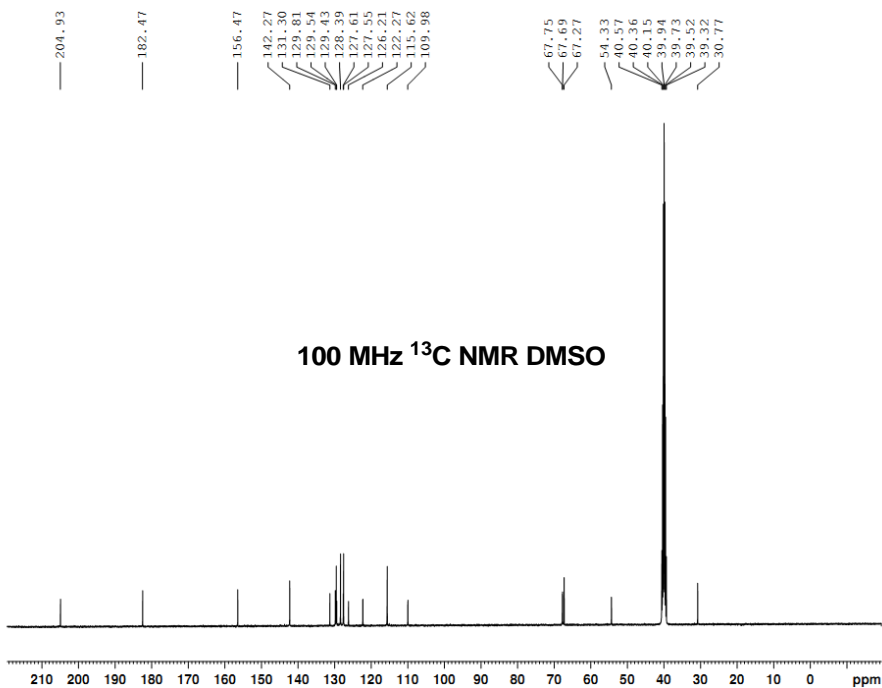
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PLW1 50.09999847 W
 SFO1 100.6228293 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PLW2 9.36999959 W
 PLW12 0.22673000 W
 PLW13 0.18365000 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 27768
 SF 100.6127690 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40



NO. 3



Current Data Parameters
 NAME 03-31-2013
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130320
 Time 20.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 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 292.5 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
 MDW EM
 SSB 0
 GB 0 1.00 Hz
 PC 1.40