Supporting Information

N-Heterocyclic Carbene-Catalyzed Synthesis of Spirocyclopentene-oxindoles from Bromoenals

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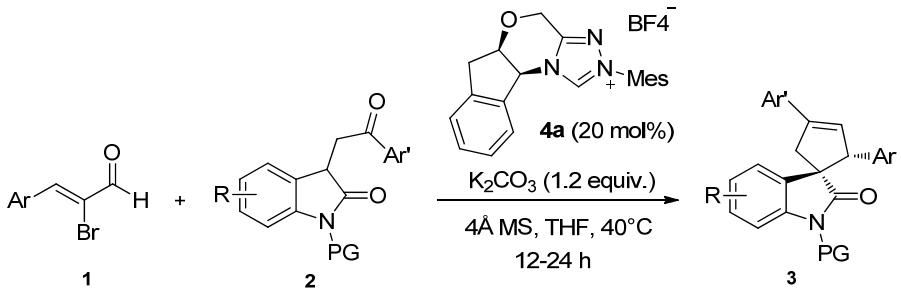
Part I General information

Unless otherwise indicated, all reactions were carried out under N_2 atmosphere at room temperature with magnetic stirring. Anhydrous THF and toluene were distilled from sodium and benzophenone. Anhydrous CH_2Cl_2 was distilled from CaH_2. Chiral triazolium salts **4-5**[1], oxindoles[2] and α-bromoenals[3] were prepared according to literatures. Column chromatography was performed on silica gel 200~300 mesh. All ^1H NMR (400 and 500 MHz), ^{13}C NMR (100 and 125 MHz) spectra were recorded on a Bruker Avance 400 and Bruker Avance 500 spectrometer in $CDCl_3$, with tetramethylsilane as an internal standard and reported in parts per million (ppm, δ). ^1H NMR Spectroscopy splitting patterns were designated as singlet (s), doublet (d), triplet (t), quartet (q). Splitting patterns that could not be interpreted or easily visualized were designated as multiplet (m) or broad (br). Infrared spectra were recorded on a JASCO FT/IR-480 spectrophotometer and reported as wave number (cm^{-1}). Optical rotations were measured on Perkin Elmer/Model-343 digital polarimeter operating at the sodium D line with a 100 mm path cell, and are reported as follows: $[\alpha]_D^T$ (concentration (g/100 mL), solvent).

Part II Experimental part

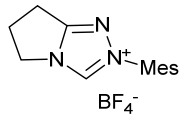
1. NHC-catalyzed reaction of bromoenals and oxindoles (Scheme 2)



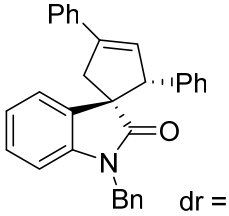
General procedure: An oven-dried 50 mL Schlenk tube was charged with bromoenal **1** (0.24 mmol), oxindole **2** (0.2 mmol), NHC precursor **4a** (16.8 mg, 0.04 mmol), K_2CO_3 (33.1 mg, 0.24 mmol), 4Å MS (100 mg) and THF (2 mL). The reaction mixture was stirred at 40 °C until the full consumption of the α-bromoenal (typically,

12-24 h). The reaction mixture was concentrated under reduced pressure and the residue was purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent, typically 5:1 to 20:1) to furnish the corresponding adduct **3**.

Racemic samples for the chiral phase HPLC analysis were prepared using triazolium **S1** as the NHC pre-catalyst under the same conditions.



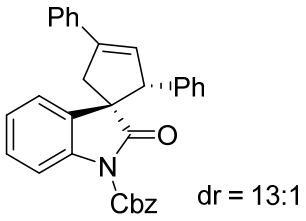
S1



3a zzf-398-b

(*1R,2R*)-1'-benzyl-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

53 mg, 63% yield, 6:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 82% ee [Daicel CHIRALPAK AD-H column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 12.3 min (minor), 20.7 min (major)]. ^1H NMR (400 MHz, CDCl3) δ 7.57 (d, J = 7.3 Hz, 2H), 7.49 (d, J = 6.6 Hz, 1H), 7.40-7.36 (t, 9.5 Hz, 2H), 7.34-7.27 (m, 2H), , 7.27-7.23 (m, 1H), 7.18-7.11 (m, 4H), 7.11-7.08 (m, 1H), 7.08-7.03 (m, 2H), 6.67-6.61 (m, 3H), 6.55 (d, J = 7.5 Hz, 1H), 6.38 (d, J = 1.6 Hz, 1H), 4.97 (d, J = 15.7 Hz, 1H), 4.63 (d, J = 1.9 Hz, 1H), 4.22 (d, J = 15.7 Hz, 1H), 3.48 (dt, J = 16.0, 2.2 Hz, 1H), 3.26 (dt, J = 16.0, 2.2 Hz, 1H); ^{13}C NMR (101 MHz, CDCl3) δ 177.82, 142.73, 142.21, 138.65, 135.80, 135.46, 134.49, 128.58, 128.52, 128.14, 128.02, 127.82, 127.33, 127.14, 127.04, 126.30, 125.96, 122.66, 122.41, 108.78, 63.07, 58.55, 44.04, 43.52. IR (KBr) 1712, 1858, 1611, 1171, 753, 652; HRMS (ESI) calcd for $C_{31}H_{25}NO$ $C_{31}H_{26}NO$ $[M+H]^+$: 428.2009, found 428.2008.

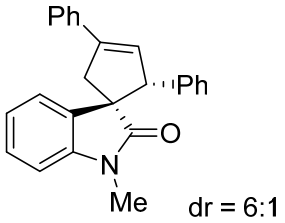


3b zzf-363

benzyl

(1R,2R)-2'-oxo-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-3-ene-1'-carboxylate

32 mg, 35% yield, 13:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 85% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 15.5 min (major), 28.2 min (minor)]. ^1H NMR (400 MHz, CDCl3) δ 7.84 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 7.4 Hz, 2H), 7.48 (d, J = 7.3 Hz, 1H), 7.39 (t, J = 7.5 Hz, 3H), 7.32 (dd, J = 9.8, 6.1 Hz, 5H), 7.28 (d, J = 6.8 Hz, 2H), 7.17-7.08 (m, 3H), 6.98-6.88 (m, 2H), 6.28 (d, J = 1.6 Hz, 1H), 5.20 (q, J = 12.6 Hz, 2H), 4.50 (d, J = 1.6 Hz, 1H), 3.54 (dt, J = 16.1, 2.0 Hz, 1H), 3.24 (dt, J = 16.1, 2.0 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 175.95, 150.53, 142.56, 138.70, 137.86, 135.16, 135.09, 133.74, 128.60, 128.49, 128.47, 128.19, 128.14, 128.06, 127.99, 127.65, 127.53, 125.98, 125.17, 124.92, 122.12, 114.96, 77.30, 77.05, 76.80, 67.91, 65.45, 59.10, 43.54. IR (KBr) 1771, 1731, 1108, 754, 657; HRMS (ESI) calcd for $C_{32}H_{26}NO_3$ $[M+H]^+$: 472.1907, found: 472.1902.

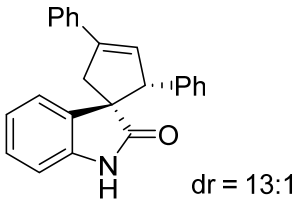


3c zzf-421

(1R,2R)-1'-methyl-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

25 mg, 36% yield, 6:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 84% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 6.3 min (major), 9.3 min (minor)]. ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.3 Hz, 2H), 7.45 (d, *J* = 7.3 Hz, 1H), 7.38 (t,

J = 7.4 Hz, 2H), 7.34-7.26 (m, 2H), 7.19-7.18 (m, 3H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.97-6.96 (d, *J* = 3.6 Hz, 2H), 6.74 (d, *J* = 7.7 Hz, 1H), 6.35 (d, *J* = 14,8 Hz 1H), 4.49 (d, *J* = 2.4 Hz, 1H), 3.46 (dt, *J* = 16.0, 2.0 Hz), 3.16 (dt, *J* = 16.0, 2.0 Hz, 1H), 2.85 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.66, 143.37, 142.50, 138.76, 135.45, 128.52, 128.06, 128.03, 127.85, 127.83, 127.21, 125.98, 125.86, 122.65, 122.09, 107.64, 63.34, 58.21, 43.11, 25.78. IR (KBr) 1710, 1635, 1077, 697, 507. 351.1623; HRMS (ESI) calcd for $C_{25}H_{22}NO$ $[M+H]^+$: 352.1696, found: 352.1687.



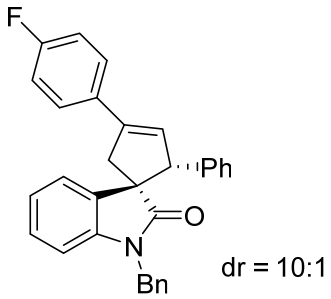
dr = 13:1

3d

zzf-423

(1R,2R)-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

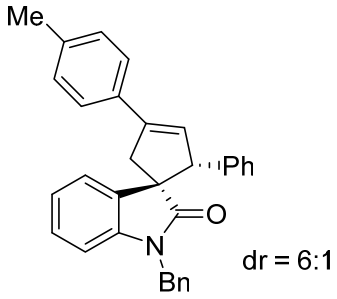
34 mg, 51% yield, 13:1 dr. White solid, mp. = 187-188^{o}C; R_f = 0.2 (petroleum ether/ethyl acetate 5:1); $[\alpha]_D^{25}$ = +132 (*c* 0.14, $CHCl_3$); HPLC analysis: 62% ee [Daicel CHIRALPAK IB column, 20 ^{o}C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 6.7 min (major), 11.7 min (minor)]. ^{1}H NMR (500 MHz, CDCl3) δ 7.57 (d, J = 7.3 Hz, 2H), 7.44 (d, J = 7.6 Hz, 2H), 7.39 (t, J = 7.6 Hz, 2H), 7.31 (t, J = 7.3 Hz, 1H), 7.22 (dd, J = 8.6, 7.9 Hz, 1H), 7.21-7.19 (m, 3H), 7.09 (t, J = 7.5 Hz, 1H), 7.04-6.99 (m, 2H), 6.76 (d, J = 7.7 Hz, 1H). 6.35 (dd, J = 3.9, 1.8 Hz, 1H), 4.52 (t, J = 4.4 Hz, 1H), 3.47 (dt, J = 16.1, 2.0 Hz, 1H), 3.16 (dt, J = 16.1, 2.0 Hz,, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 179.78, 142.41, 140.23, 138.77, 135.86, 135.27, 128.55, 128.17, 128.01, 127.90, 127.27, 125.97, 125.79, 122.73, 122.54, 109.40, 63.31, 58.46, 43.32, 29.72. IR (KBr) 1707, 1633, 1075, 753, 698; HRMS (ESI) calcd for $C_{24}H_{20}NO$ $[M+H]^+$: 338.1539, found: 338.1534.



3e zzf-399

(1R,2R)-1'-benzyl-4-(4-fluorophenyl)-2-phenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

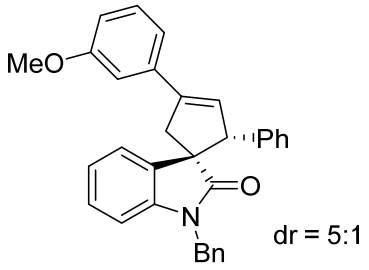
54 mg, 61% yield, 10:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 81% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 7.6 min (major), 12.4 min (minor)]. ^1H NMR (500 MHz, CDCl3) δ 7.53 (dd, J = 8.5, 5.5 Hz, 2H), 7.49 (d, J = 7.1 Hz, 1H), 7.25 (d, J = 8.9 Hz, 1H), 7.21 (t, J = 7.3 Hz, 2H), 7.18-7.10 (m, 4H), 7.10-7.07 (m, 1H), 7.07 (s, 1H), 7.06-7.02 (m, 2H), 6.61 (d, J = 6.7 Hz, 2H), 6.54 (d, J = 7.6 Hz, 1H). 6.30 (d, J = 1.2 Hz, 1H), 4.96 (d, J = 15.7 Hz, 1H), 4.63 (s, 1H), 4.21 (d, J = 15.7 Hz, 1H), 3.43 (dt, J = 15.9, 2.1 Hz , J = 15.9 Hz, 1H), 3.24 (dt, J = 15.9, 2.1 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.81, 162.56 (d, J = 247.70 Hz) 142.76, 141.10, 138.50, 135.71, 134.12, 131.70, 131.67, 128.53 (d, J = 1.24 Hz), 128.17, 128.10, 127.82, 127.61, 127.55, 127.38, 127.15, 126.99, 126.02, 122.70, 122.43, 115.41 (d, J = 21.62 Hz), 108.84, 77.31, 77.05, 76.80, 63.00, 58.68, 44.15, 43.51. IR (KBr) 1713, 1611, 1078, 908, 507; HRMS (ESI) calcd for $C_{31}H_{25}FNO$ $[M+H]^+$: 446.1915, found: 446.1907.



3f zzf-439

(1R,2R)-1'-benzyl-2-phenyl-4-(p-tolyl)spiro[cyclopentane-1,3'-indolin]-3-en-2'-one

40 mg, 45% yield, 6:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 77% ee [Daicel CHIRALPAK AD-H column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 14.1 min (major), 30.0 min (minor)]. ^1H NMR (500 MHz, CDCl3) δ 7.47 (m, 3H), 7.24 (t, J = 2.4 Hz, 4H), 7.23-7.18 (m, 2H), 7.17-7.11 (m, 3H), 7.06 (t, J = 2.4 Hz, 2H), 6.66 (d, J = 3.9 Hz, 2H), 6.55 (d, J = 7.6 Hz, 1H), 6.32 (d, J = 7.3 Hz, 1H), 4.97 (d, J = 15.7 Hz, 1H), 4.61 (d, J = 4.4 Hz, 1H), 4.23 (d, J = 15.7 Hz, 1H), 3.47 (dt, J = 15.9, 2.0 Hz, 1H), 3.23 (dt, J = 15.9, 2.0 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.87, 142.67, 142.08, 138.79, 137.64, 135.81, 134.68, 132.65, 129.19, 128.58, 128.51, 128.11, 127.95, 127.28, 127.13, 127.04, 125.87, 125.27, 122.64, 122.38, 108.74, 63.09, 58.49, 44.06, 43.51, 21.28. IR (KBr) 1714, 1611, 1168, 767, 659; HRMS (ESI) calcd for $C_{32}H_{28}NO$ $[M+H]^+$: 442.2165, found: 442.2159.

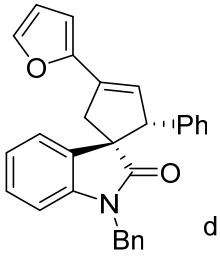


3g zzf-403

(1R,2R)-1'-benzyl-4-(3-methoxyphenyl)-2-phenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

53 mg, 58% yield, 5:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 89% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 10.7 min (minor), 13.9 min (major)]. ^1H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.2 Hz, 1H), 7.29 (dd, *J* = 14.0, 6.2 Hz, 2H), 7.22 (t, *J* = 7.3 Hz, 2H), 7.15 (dd, *J* = 13.0, 7.2 Hz, 5H), 7.09 (d, *J* = 8.2 Hz, 2H), 7.07-7.02 (m, 3H), 6.89-6.84 (m, 1H), 6.64 (d, *J* = 6.5 Hz, 2H), 6.55 (d, *J* = 7.6 Hz, 1H), 6.37 (d, J = 7.3 Hz, 1H), 4.97 (d, J = 15.7 Hz, 1H), 4.62 (d, J = 4.4 Hz, 1H), 4.22 (d, J = 15.7 Hz, 1H), 3.84 (s, 3H), 3.47 (dt, J = 15.9, 2.0 Hz, 1H), 3.25 (dt, J = 15.9, 2.0 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.80, 159.73, 142.70, 142.11, 138.57,

136.86, 135.77, 134.44, 129.48, 128.57, 128.52, 128.14, 128.02, 127.34, 127.14, 127.02, 126.71, 122.67, 122.40, 118.59, 113.38, 111.57, 108.78, 63.00, 58.51, 55.30, 44.08, 43.51. IR (KBr) 1712, 1611, 1106, 753, 698; HRMS (ESI) calcd for $C_{32}H_{28}NO_2$ $[M+H]^+$: 458.2115, found: 458.2106.



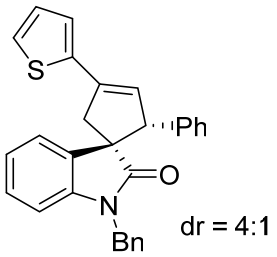
dr = 1.2:1

3h

zzf-413

(1R,2R)-1'-benzyl-4-(furan-2-yl)-2-phenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

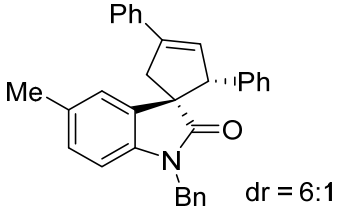
54 mg, 65% yield, 1.2:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 58% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 9.6 min (minor), 17.6 min (major)]. ^1H NMR (400 MHz, CDCl3) δ 7.46 (t, J = 7.1 Hz, 2H), 7.30 (dd, J = 12.3, 5.0 Hz, 2H), 7.21 (dd, J = 14.8, 7.2 Hz, 3H), 7.14 (t, J = 6.9 Hz, 2H), 7.01 (dd, J = 11.4, 6.6 Hz, 6H), 6.91 (t, J = 7.4 Hz, 1H), 6.71 (t, J = 7.5 Hz, 1H), 6.61 (d, J = 6.2 Hz, 1H), 6.48 (d, J = 7.8 Hz, 1H), 6.43 (dd, J = 4.8, 2.9 Hz, 1H), 6.35 (s, 1H), 6.30 (d, J = 3.2 Hz, 1H), 6.28 (d, J = 7.3 Hz, 1H). 5.07 (d, *J* = 15.6 Hz, 1H), 4.87 (d, J = 1.9 Hz , 1H), 4.76 (d, *J* = 15.6 Hz, 1H), 4.20 (d, *J* = 15.7 Hz, 1H), 3.49 (dt, *J* = 15.3, 2.6 Hz, 1H), 2.98 (dt, *J* = 15.3, 2.6 Hz,1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.86, 151.01, 142.51, 141.80, 138.55, 135.88, 132.44, 128.66, 128.52, 128.14, 127.76, 127.54, 127.39, 126.99, 126.83, 125.03, 124.18, 121.97, 111.29, 108.54, 107.58, 63.03, 59.55, 43.92, 43.50. IR (KBr) 1709, 1612, 1078, 739, 532; HRMS (ESI) calcd for $C_{29}H_{24}NO_2$ $[M+H]^+$: 418.1802, found: 418.1793.



3i zzf-411

(1R,2R)-1'-benzyl-2-phenyl-4-(thiophen-2-yl)spiro[cyclopentane-1,3'-indolin]-3-en-2'-one

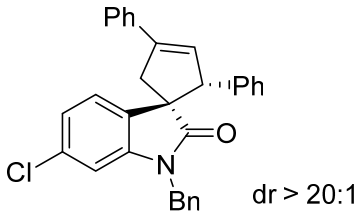
63 mg, 73% yield, 4:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 77% ee [Daicel CHIRALPAK AD-H column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 13.5 min (minor), 20.1 min (major)]. ^1H NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 7.2, 0.7 Hz, 1H), 7.25 (d, *J* = 2.0 Hz, 2H), 7.21 (t, *J* = 7.2 Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 3H), 7.08 (td, *J* = 7.6, 0.9 Hz, 1H), 7.05 (d, *J* = 3.1 Hz, 1H), 7.03-7.01 (m, 4H), 6.62 (d, *J* = 6.4 Hz, 2H), 6.54 (d, *J* = 7.6 Hz, 1H), 6.21 (d, *J* = 1.6 Hz, 1H), 4.97 (d, *J* = 15.7 Hz, 1H), 4.97 (d, *J* = 15.7 Hz, 1H), 4.62 (d, *J* = 1.9 Hz, 1H), 4.62 (d, *J* = 1.9 Hz, 1H), 4.20 (d, *J* = 15.7 Hz, 1H), 4.20 (d, *J* = 15.7 Hz, 1H), 3.51-3.42 (m, 1H), 3.51-3.40 (m, 1H), 3.25 (dt, *J* = 15.8, 2.3 Hz, 1H), 3.25 (dt, *J* = 15.8, 2.3 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.85, 142.43, 142.15, 138.56, 135.37, 134.82, 134.17, 131.78, 131.64, 129.07, 128.75, 128.56, 128.52, 128.17, 128.07, 127.85, 127.35, 126.21, 125.93, 122.86, 122.60, 121.05, 108.57, 63.04, 58.65, 43.98, 42.91. IR (KBr) 1716, 1608, 1101, 753, 574; HRMS (ESI) calcd for $C_{29}H_{24}NOS$ $[M+H]^+$: 434.1573, found: 434.1565.



3j zzf-405

(1R,2R)-1'-benzyl-5'-methyl-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

64 mg, 73% yield, 6:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 84% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 5.4 min (major), 18.5 min (minor)]. ^1H NMR (500 MHz, $CDCl_3$) δ 7.61-7.54 (m, 2H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.33-7.29 (m, 2H), 7.26- 7.24 (t, *J* = 1.6 Hz, 1H), 7.23 (d, *J* = 7.3 Hz, 2H), 7.16-7.12 (m, 2H), 7.09-7.05 (m, 2H), 6.94 (dd, *J* = 7.9, 0.7 Hz, 1H), 6.68-6.64 (m, 2H), 6.44 (d, *J* = 7.9 Hz, 1H), 6.37 (d, *J* = 1.9 Hz, 1H), 4.94 (d, *J* = 15.7 Hz, 1H), 4.61 (d, *J* = 2.1 Hz, 1H), 4.21 (d, *J* = 15.7 Hz, 1H), 3.52-3.45 (dt, *J* = 15.9, 2.2 Hz, 1H, 1H), 3.24 (dt, *J* = 15.9, 2.2 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.81, 142.19, 140.27, 138.76, 135.92, 135.48, 134.71, 132.19, 128.60, 128.50, 128.48, 128.24, 128.20, 128.10, 127.78, 127.37, 127.29, 127.09, 127.05, 126.31, 125.97, 125.91, 123.17, 108.51, 63.11, 58.50, 44.07, 43.53, 21.22. IR (KBr) 1705, 1558, 1249, 909, 659; HRMS (ESI) calcd for $C_{32}H_{28}NO$ $[M+H]^+$: 442.2162, found: 442.2165.

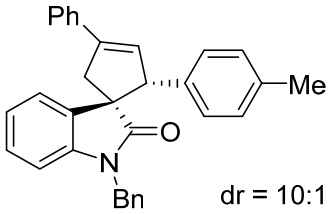


3k zzf-415

(1R,2R)-1'-benzyl-6'-chloro-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

68 mg, 74% yield, > 20:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +23 (*c* 0.39, $CHCl_3$); HPLC analysis: 81% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 8.9 min (major), 27.8 min (minor)]. ^1H NMR (500 MHz, $CDCl_3$) δ 7.59-7.54 (m, 2H), 7.43-7.36 (m, 3H), 7.32 (d, *J* = 7.4 Hz, 1H), 7.28 (ddd, *J* = 6.0, 4.1, 1.2 Hz, 1H), 7.25-7.21 (m, 1H), 7.19-7.13 (m, 3H), 7.09-7.02 (m, 3H), 6.64-6.59 (m, 2H), 6.54 (d, *J* = 1.8 Hz, 1H), 6.39-6.33 (m, 1H), 5.05 (d, J = 15.7 Hz, 1H), 4.94 (d, J = 15.7 Hz, 1H), 4.59 (d, J = 2.1 Hz, 1H), 4.18 (d, J = 15.7 Hz, 1H), 3.52-3.42 (dt, J = 15.9, 2.3 Hz,, 1H), 3.22 (dt, J = 15.9, 2.3 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.71,

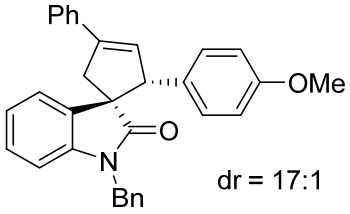
143.92, 142.12, 138.29, 135.24, 135.17, 133.66, 132.86, 128.68, 128.55, 128.25, 127.94, 127.50, 127.37, 126.96, 126.14, 125.94, 123.34, 122.54, 109.33, 77.30, 77.04, 76.79, 63.04, 58.24, 43.98, 43.59. IR (KBr) 1706, 1646, 1176, 807, 652; HRMS (ESI) calcd for $C_{31}H_{25}NOCl$ $[M+H]^+$: 462.1619, found: 462.1609.



3l zzf-387

(1R,2S)-1'-benzyl-4-phenyl-2-(p-tolyl)spiro[cyclopentane-1,3'-indolin]-3-en-2'-one

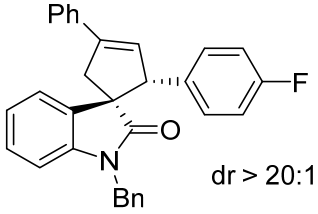
54 mg, 61% yield, 10:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +133 (*c* = 0.15, $CHCl_3$); HPLC analysis: 86% ee [Daicel CHIRALPAK AD-H column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL /min, 254 nm, 12.7 min (minor), 26.0 min (major)]. ^1H NMR (400 MHz, CDCl3) δ 7.56 (d, J = 7.3 Hz, 2H), 7.48 (d, J = 7.1 Hz, 1H), 7.38 (t, J = 7.4 Hz, 2H), 7.34-7.27 (m, 1H), 7.15 (t, J = 8.2 Hz, 4H), 7.10-7.05 (m, 1H), 7.03 (d, J = 7.8 Hz, 2H), 6.95 (d, J = 7.8 Hz, 2H), 6.67 (d, J = 6.9 Hz, 2H), 6.54 (d, J = 7.6 Hz, 1H), 6.36 (d, J = 7.3 Hz 1H), 5.01 (d, J = 15.7 Hz, 1H), 4.60 (d, *J* = 14,8 Hz, 1H), 4.23 (d, J = 15.7 Hz, 1H), 3.48 (dt, *J* = 15.9, 2.0 Hz, 1H), 3.24 (dt, *J* = 15.9, 2.0 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (101 MHz, CDCl3) δ 177.91, 142.71, 141.95, 136.79, 135.82, 135.61, 135.49, 134.65, 128.83, 128.49, 128.39, 127.93, 127.75, 127.13, 127.07, 126.59, 125.94, 122.62, 122.37, 108.74, 62.78, 58.53, 44.00, 43.51, 21.28. IR (KBr) 1715, 1352, 1076, 751, 653. 441.2093; HRMS (ESI) calcd for $C_{32}H_{28}NO$ $[M+H]^+$: 442.2165, found 427.2162.



3m zzf-377

(1R,2R)-1'-benzyl-2-(4-methoxyphenyl)-4-phenylspiro[cyclopentane-1,3' indolin]-3-en-2'-one.

62 mg, 68% yield, 17:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); HPLC analysis: 86% ee [Daicel CHIRALPAK IB column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 10.8 min (minor), 31.2 min (major)]. ^1H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.6 Hz, 2H), 7.47 (d, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.30 (dd, *J* = 14.5, 7.1 Hz, 1H), 7.18-7.11 (m, 4H), 7.07 (dd, *J* = 13.7, 6.3 Hz, 1H), 6.96 (d, *J* = 8.5 Hz, 2H), 6.74 (d, *J* = 8.5 Hz, 2H), 6.64 (d, *J* = 6.0 Hz, 2H), 6.55 (d, *J* = 7.6 Hz, 1H), 6.34 (s, 1H), 5.01 (d, *J* = 15.7 Hz, 1H), 4.59 (d, J = 1.6 Hz 1H), 4.21 (d, *J* = 15.7 Hz, 1H), 3.77 (s, 3H), 3.46 (dt, J = 15.9, 2.0 Hz, 1H), 3.24 (dt, J = 15.9, 2.0 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.99, 158.96, 142.79, 141.85, 135.79, 135.52, 134.41, 130.68, 129.63, 128.51, 128.43, 127.98, 127.77, 127.21, 127.05, 126.68, 125.95, 122.63, 122.43, 113.49, 108.71, 77.32, 77.07, 76.81, 62.48, 58.68, 55.13, 43.85, 43.50. IR (KBr) 1712, 1611, 1031, 831, 576; HRMS (ESI) calcd for $C_{32}H_{28}NO_2$ $[M+H]^+$: 458.2115, found: 458.2107.

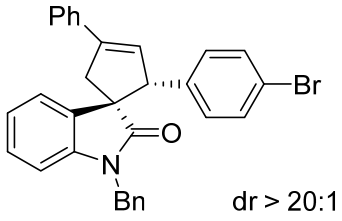


3n zzf-382

(1R,2R)-1'-benzyl-2-(4-fluorophenyl)-4-phenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one.

53 mg, 60% yield, >20:1 dr. White solid; mp. = 96-97 ^oC; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +100 (*c* = 0.08, $CHCl_3$); HPLC analysis: 86% ee [Daicel CHIRALPAK AD-H column, 20 ^oC, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL

/min, 254 nm, 8.9 min (major), 27.8 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.3 Hz, 2H), 7.48 (d, *J* = 7.2 Hz, 1H), 7.42-7.35 (m, 2H), 7.34-7.28 (m, 1H), 7.21-7.13 (m, 4H), 6.99 (dd, *J* = 8.5, 5.6 Hz, 1H), 6.88 (t, *J* = 8.7 Hz, 2H), 6.70-6.64 (m, 2H), 6.60 (d, *J* = 7.7 Hz, 1H), 6.33 (d, *J* = 1.6 Hz, 1H), 4.98 (d, J = 15.6 Hz, 1H), 4.60 (d, J = 1.8 Hz, 1H), 4.23 (d, J = 15.6 Hz, 1H), 3.47 (d, J = 16.0 Hz, 1H), 3.26 (dt, J = 16.0, 2.2 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.75, 162.29 (d, JC-F = 243.6 Hz), 142.73, 142.39, 135.71, 135.29, 9134.33, 134.13, 130.09, 130.03, 128.66 (d, JC-F = 7.3 Hz), 128.13, 127.92, 127.03, 127.05, 125.96, 114.9344 (d, JC-F = 8.2 Hz), 108.77, 62.24, 58.51, 43.90, 43.51. IR (KBr) 1712, 1608, 840, 753, 551; HRMS (ESI) calcd for $C_{31}H_{25}FNO$ $[M+H]^+$: 442.1915, found: 446.1906.

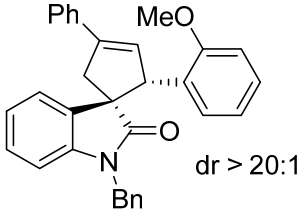


3o zzf-373

(1R,2R)-1'-benzyl-2-(4-bromophenyl)-4-phenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

65 mg, 65% yield, > 20:1 dr. Light yellow solid, mp. = 106-107 °C; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +121 (*c* 0.09, $CHCl_3$); HPLC analysis: 83% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL /min, 254 nm, 11.1 min (major), 24.4 min (minor)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58-7.54 (m, 2H), 7.48 (dd, *J* = 7.3, 0.8 Hz, 1H), 7.39 (dd, *J* = 10.4, 4.7 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 3H), 7.22 (d, *J* = 7.0 Hz, 2H), 7.16 (td, *J* = 7.7, 1.2 Hz, 1H), 7.09 (td, *J* = 7.6, 0.9 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 2H), 6.66 (dd, *J* = 8.5, 6.5 Hz, 2H), 6.59 (d, *J* = 7.7 Hz, 1H), 6.31 (d, *J* = 1.6 Hz, 1H), 5.01 (d, J = 15.6 Hz, 1H), 4.58 (d, J = 2.1 Hz, 1H), 4.21 (d, J = 15.6 Hz, 1H), 3.52-3.41 (dt, J = 16.0, 2.4 Hz,, 1H), 3.26 (dt, J = 16.0, 2.4 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.66, 142.76, 142.67, 137.71, 135.63, 135.22, 133.89, 131.24, 130.28, 128.63, 128.55, 128.21, 127.98, 127.39, 127.03, 125.97, 125.59, 122.79, 122.48, 121.34, 108.86, 62.28, 58.46, 44.08, 43.60.

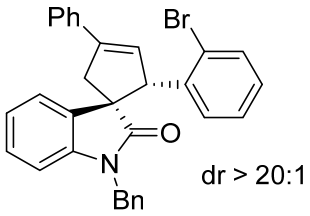
IR (KBr) 1712, 1611, 1171, 1029, 802; HRMS (ESI) calcd for $C_{31}H_{25}BrNO$ $[M+H]^+$: 506.1114, found: 506.1103.



3p zzf-371

(1R,2R)-1'-benzyl-2-(2-methoxyphenyl)-4-phenylspiro[cyclopentane-1,3'-indolin]-3-en-2'-one

58.0 mg, 64% yield, > 20:1 dr. Colorless oil; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +63 (*c* 0.15, $CHCl_3$); HPLC analysis: 92% ee [Daicel CHIRALPAK IB column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 7.0 min (major), 16.1 min (minor)]. ^{1}H NMR (500 MHz, CDCl3) δ 7.57 (d, J = 7.5 Hz, 2H), 7.46 (d, 1H), 7.43-7.35 (q, 3H), 7.30 (t, J = 7.3 Hz, 1H), 7.20 (m, 4H), 7.14-7.08 (t, J = 7.6 Hz 1H), 7.04 (t, J = 7.7 Hz,1H), 6.95 (t, J = 7.4 Hz 1H), 6.90-6.85 (m, 2H), 6.68 (d, J = 3.1 Hz 1H), 6.62 (d, J = 7.7 Hz 1H), 6.33 (d, J = 1.8 Hz, 1H), 4.97 (d, J = 1.5 Hz, 1H), 4.92 (d, J = 15.5 Hz, 1H), 4.43 (d, J = 15.5 Hz, 1H), 3.49 (dt, J = 15.9, 2.3 Hz, 1H), 3.13 (dt, J = 15.9, 2.3 Hz, 1H), 3.14 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.98, 157.33, 142.33, 142.18, 136.75, 136.31, 135.57, 129.79, 128.54, 128.51, 128.08, 127.70, 127.59, 127.39, 127.30, 127.19, 126.65, 125.92, 122.40, 122.17, 120.31, 109.87, 108.21, 57.11, 55.29, 54.60, 44.61, 43.64. IR (KBr) 1712,1578, 1106, 849, 753; HRMS (ESI) calcd for $C_{32}H_{28}NO_2$ $[M+H]^+$: 458.2115, found: 458.2107.



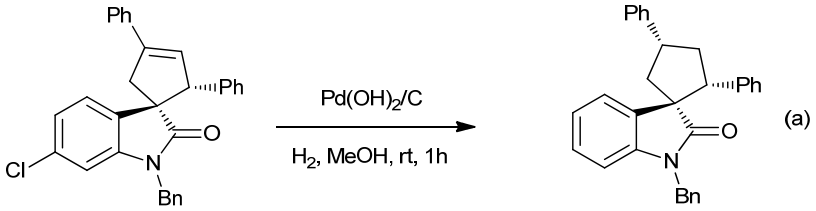
3q zzf-375

(1R,2S)-1'-benzyl-2-(2-bromophenyl)-4-phenylspiro[cyclopentane-1,3'-indolin]-3

-en-2'-one

61 mg, 61% yield, > 20:1 dr. White solid, mp. = 120-121°C; R_f = 0.2 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +75 (*c* 0.15, $CHCl_3$); HPLC analysis: 88% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm, hexane/*i*-PrOH = 80:20, 1.0 mL /min, 254 nm, 10.5 min (minor), 15.2 min (major)]. ^{1}H NMR (400 MHz, CDCl3) δ 7.58 (d, J = 7.4 Hz, 2H), 7.46 (d, J = 7.0 Hz, 2H), 7.40 (t, J = 7.4 Hz, 2H), 7.36-7.26 (m, 2H), 7.22 (d, J = 5.6 Hz, 2H), 7.18-7.08 (m, 2H), 7.03 (d, J = 7.0 Hz, 2H), 6.67 (d, J = 7.7 Hz, 1H), 6.27 (d, *J* = 7.6 Hz 1H), 4.98 (s, 1H), 4.80 (d, J = 15.5 Hz, 1H), 4.64 (d, J = 15.6 Hz, 1H), 3.56 (dt, J = 16.0, 2.0 Hz, 1H), 3.09 (dt, J = 16.0, 2.0 Hz, 1H); ^{13}C NMR (126 MHz, CDCl3) δ 177.34, 143.30, 142.25, 138.43, 136.50, 136.01, 135.12, 132.42, 131.29, 128.79, 128.64, 128.55, 128.07, 127.89, 127.45, 127.33, 127.01, 126.12, 126.02, 125.06, 122.77, 122.16, 108.70, 77.29, 77.04, 76.78, 61.05, 56.74, 44.61, 43.95. IR (KBr) 1712, 1611, 1525, 1217, 753; HRMS (ESI) calcd for $C_{31}H_{25}BrNO$ $[M+H]^+$: 506.1114, found: 506.1106.

2. Hydrogenation of spirocyclopentene-2-oxindoles (Scheme 3)



3k, 81% ee

4a, 89%, >20:1 dr, 80% ee

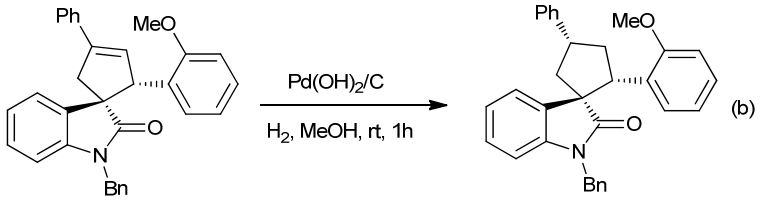
zzf-438-b

A suspension of $Pd(OH)_2/C$ (15 mg) and spirocyclopentene-2-oxindole **3k** (30 mg) in methanol (5 mL) was stirred at room temperature under 1 atm hydrogen atmosphere. After stirring for 1 h, the solvent was filtered through a plug of celite, evaporated. The residue was purified by column chromatography to afford spirocyclopentane-2-oxindole **4a** with the loss of chlorine.

(1R,2R,4R)-1'-benzyl-2,4-diphenylspiro[cyclopentane-1,3'-indolin]-2'-one

29 mg, 89% yield, > 20:1 dr. Colorless oil; R_f = 0.3 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +81 (*c* 0.15, $CHCl_3$); HPLC analysis: 80% ee [Daicel CHIRALPAK IB

column, 20 °C, 254 nm, hexane/*i*-PrOH = 90:10, 1.0 mL /min, 254 nm, 6.2 min (major), 8.7 min (minor)]. ^{1}H NMR (500 MHz, CDCl3) δ 7.61 (d, *J* = 7.3 Hz, 2H), 7.51-7.48 (m, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.27- 7.24 (m, 1H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.12 (dt, *J* = 6.2, 4.0 Hz, 4H), 7.08 (dt, *J* = 10.9, 4.2 Hz, 3H), 6.97 (d, *J* = 7.5 Hz, 2H), 6.45 (d, *J* = 7.4 Hz, 2H), 6.41 (d, *J* = 7.2 Hz, 1H), 4.99 (d, J = 15.9 Hz, 1H), 4.21 (d, J = 15.9 Hz, 1H), 3.72 (dd, J = 13.5, 5.0 Hz, 1H), 3.69–3.58 (m, 1H), 3.23 (dd, J = 25.4, 12.1 Hz, 1H), 2.67–2.58 (m, 2H), 2.39 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.98, 144.53, 142.90, 137.50, 135.45, 134.41,128.58, 128.52, 128.11, 127.93, 127.77, 127.11, 127.00, 126.58, 126.45, 122.70, 122.30, 108.87, 58.69, 57.94, 45.37, 45.20, 43.51, 39.43. IR (KBr) 1705, 1612, 909, 659; HRMS (ESI) calcd for $C_{31}H_{27}NONa$ $[M+Na]^+$ 452.1985, found 452.1988.



3p, 92% ee **4p**, 99%, >20:1 dr, 91% ee zzf-438-a

(1R,2R,4R)-1'-benzyl-2-(2-methoxyphenyl)-4-phenylspiro[cyclopentane-1,3'-indolin]-2'-one

The same procedure as the reaction of **3k** but using **3p** (60 mg), $Pd(OH)_2$ (5 mg) to give **4p** (61 mg, 99% yield). > 20:1 dr. White solid, mp. = 86-87°C; R_f = 0.3 (petroleum ether/ethyl acetate 20:1); $[\alpha]_D^{25}$ = +187 (*c* 0.046, $CHCl_3$); HPLC analysis: 91% ee [Daicel CHIRALPAK IB column, 20 °C, 254 nm, hexane/*i*-PrOH = 95:5, 1.0 mL /min, 254 nm, 7.9 min (minor),13.7 min (major)]. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 (dd, *J* = 10.8, 4.3 Hz, 3H), 7.51 (dd, *J* = 7.3, 0.6 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.27-7.23 (m, 1H), 7.21-7.16 (m, 1H), 7.15-7.06 (m, 4H), 7.01 (td, *J* = 7.7, 1.2 Hz, 1H), 6.91 (dd, *J* = 11.0, 4.0 Hz, 1H), 6.59 (d, *J* = 7.7 Hz, 1H), 6.51 (d, *J* = 7.1 Hz, 2H), 6.39 (d, *J* = 7.6 Hz, 1H), 5.06 (d, *J* = 15.8 Hz, 1H), 4.33 (dd, *J* = 13.7, 5.2 Hz, 1H), 4.18 (d, *J* = 15.8 Hz, 1H), 3.73-3.61 (m, 1H), 3.11 (dd, *J* = 23.8, 10.3 Hz, 1H), 2.99 (s, 3H), 2.69-2.54 (m, 2H), 2.28 (dt, *J* = 10.8, 5.2 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$)

δ 180.12, 157.72, 144.37, 142.40, 135.84, 135.76, 129.02, 128.54, 128.50, 127.69, 126.94, 126.89, 126.78, 126.59, 126.41, 122.92, 122.14, 120.51, **1**10.13, 108.28, 77.30, 77.04, 76.79, 58.10, 54.52, 49.31, 45.97, 45.56, 43.52, 40.09. IR (KBr)1706, 1646, 1112, 758, 652; HRMS (ESI) calcd for $C_{32}H_{30}NO_2$ $[M+H]^+$ 460.2271, found 460.2267.

3. X-ray structure of compound 3q

The crystal of compound **3q** was prepared from its solution in Et_2O by slow evaporation of the solvent. The structure of compound **3q** was established by the X-ray analysis of its crystal (**Figure S1**).

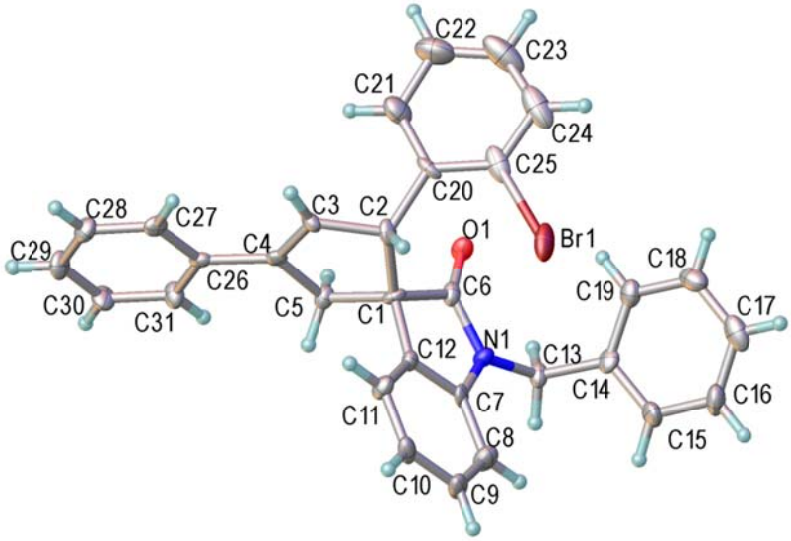
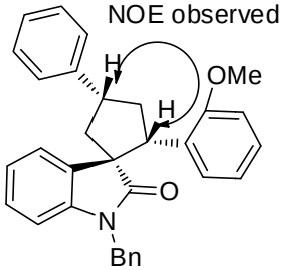


Figure S1. X-ray structure of **3q**.

4. NOE spectrum of compound 4p

The cis-configuration of compound **4p** were assigned by its NOE spectra.



4p

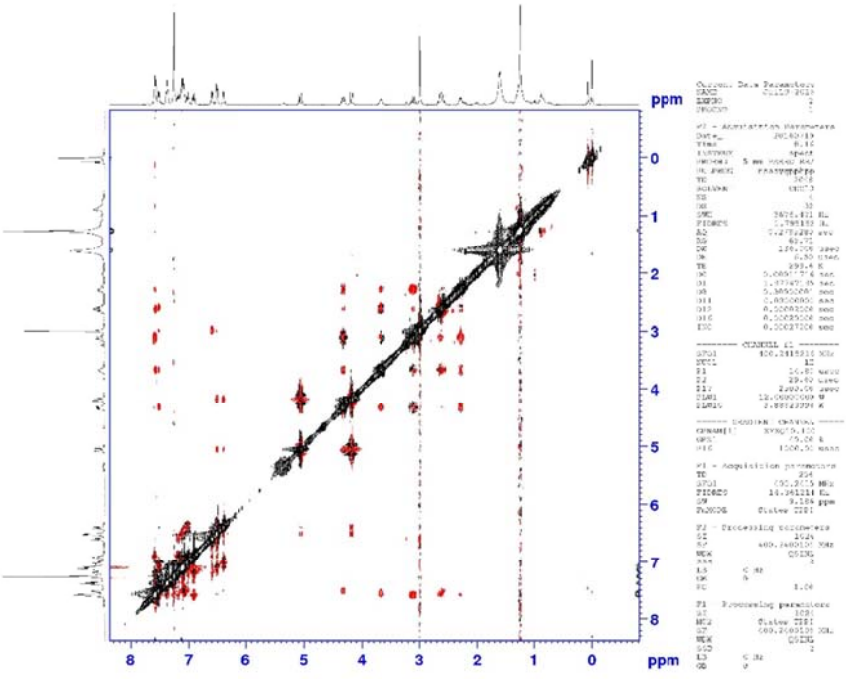


Figure S2. NOESY spectrum of **4p** (400 MHz, CDCl3)

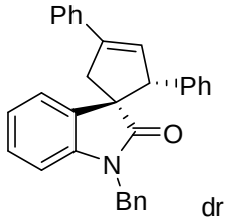
5. References

1 (a) M. He, J. R. Struble and J. W. Bode, *J. Am. Chem. Soc.*, 2006, **128**, 8418; (b) Y.-R. Zhang, L. He, X. Wu, P.-L. Shao and S. Ye, *Org. Lett.*, 2008, **10**, 277; (c) H.-M. Zhang, Z.-H. Gao and S. Ye, *Org. Lett.*, 2014, **16**, 3079.

2 (a) B. Tan, N. R. Candeias and C. F. Barbas III, *Nat. Chem.*, 2011, **3**, 473; (b) K. Albertshofer, B. Tan and C. F. Barbas III, *Org. Lett.*, 2009, **11**, 1297.

3 (a) F.-G. Sun, L.-H. Sun and S. Ye, *Adv. Synth. Catal.*, 2011, **353**, 3134; (b) Y.-H. Wu, W.-J. Yao, L.-J. Pan, Y.-P. Zhang and C. Ma, *Org. Lett.*, 2010, **12**, 640.

Part III NMR Spectra



dr = 6:1

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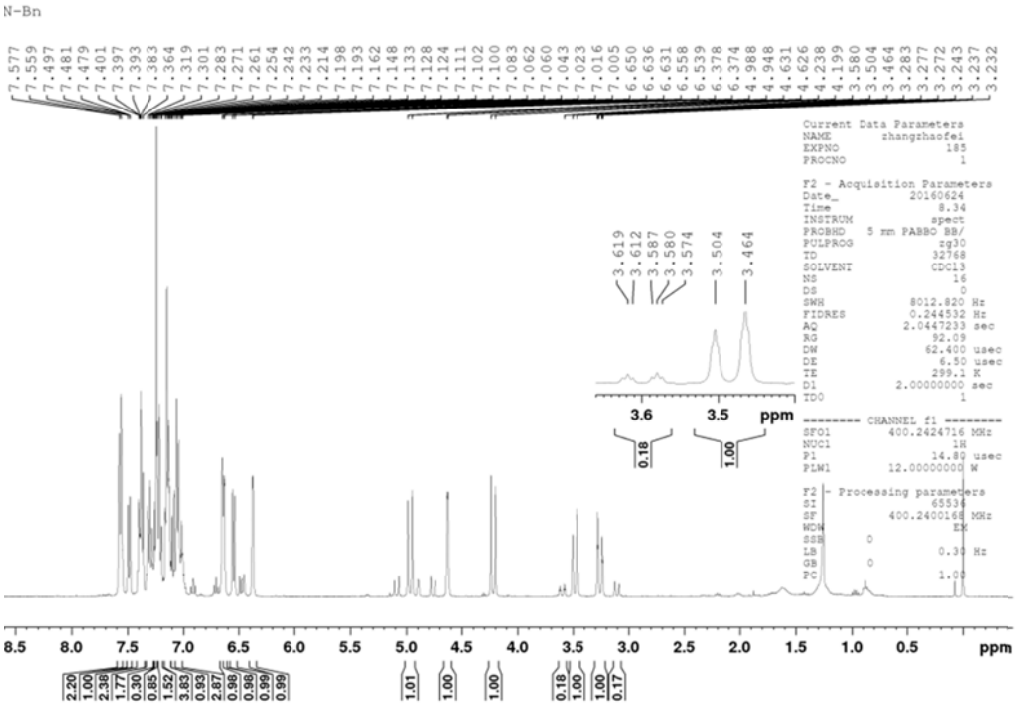
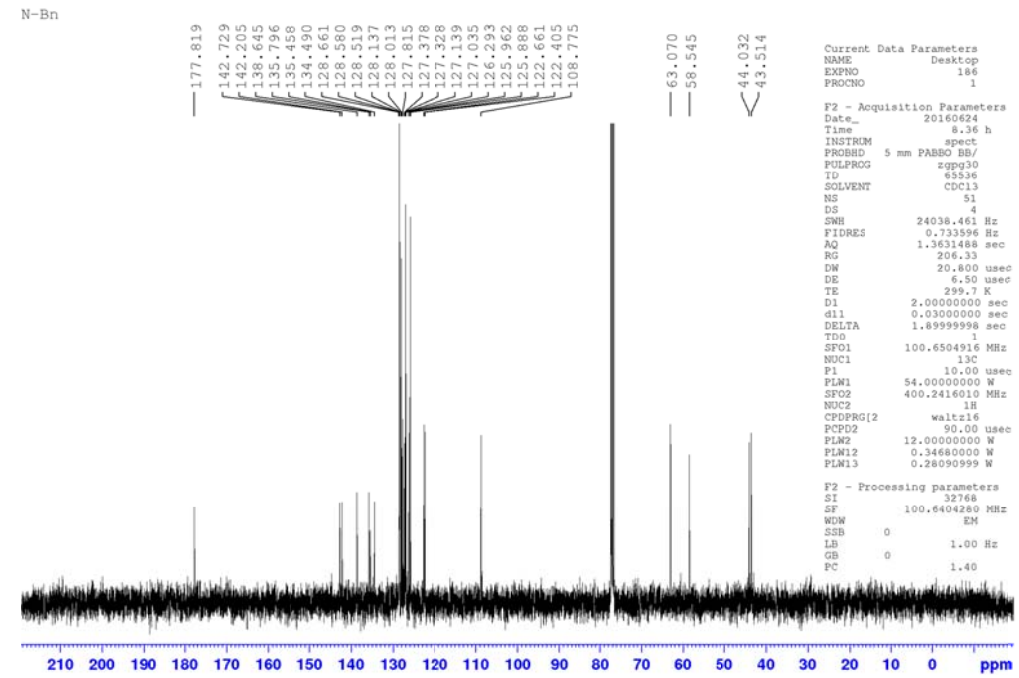
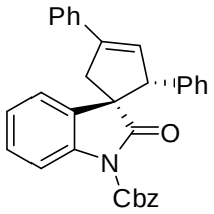
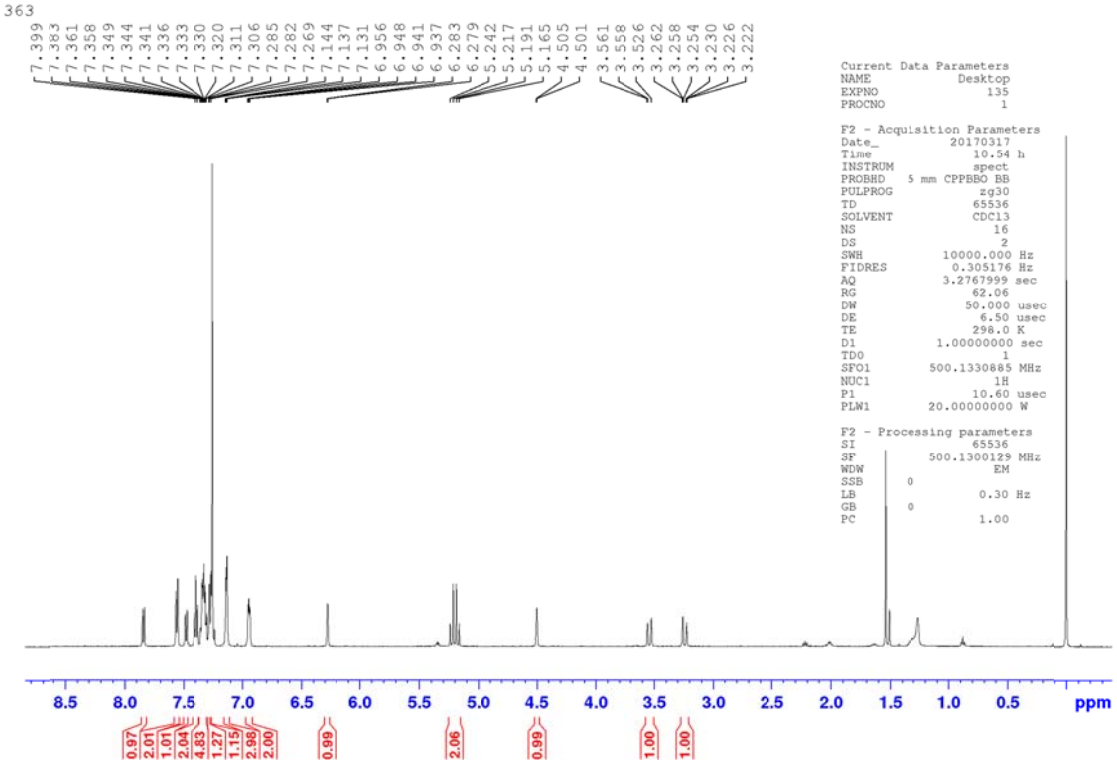





Ph
Ph
O
N
Cbz

3b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7.358
7.349
7.344
7.341
7.336
7.333
7.330
7.320
7.311
7.306
7.285
7.282
7.269
7.144
7.137
7.131
6.956
6.948
6.941
6.937
6.283
6.279
5.242
5.217
5.191
5.165
4.505
4.501
3.561
3.558
3.526
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3.258
3.254
3.230
3.226
3.222
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RG 62.06
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
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TD0 1
SFO1 500.1330885 MHz
NUC1 1H
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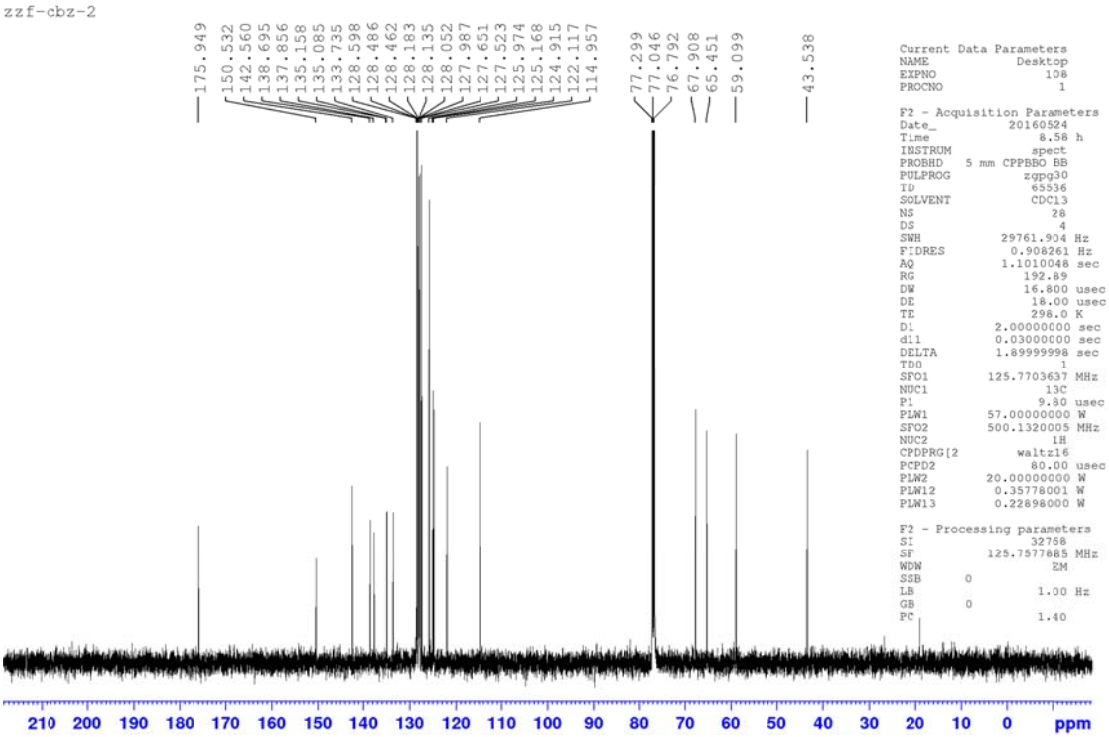
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.97 2.01 1.01 2.04 4.83 1.27 1.15 2.98 2.00 0.99 2.06 0.99 1.00 1.00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128.183
128.135
128.052
127.987
127.651
127.523
125.974
125.168
124.915
122.117
114.957
77.299
77.046
76.792
67.908
65.451
59.099
43.538
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RG 192.89
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DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
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NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
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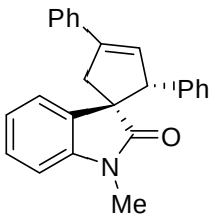
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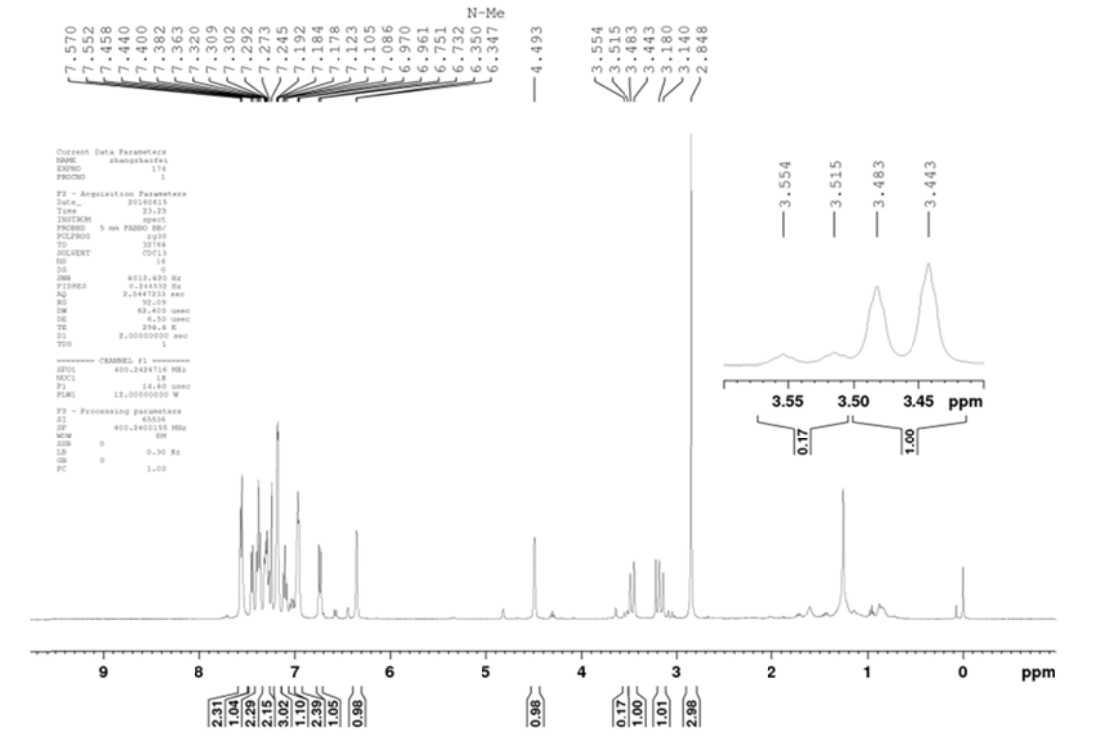
Ph
O
N
Me

dr = 6:1

3c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7.363
7.320
7.309
7.302
7.292
7.273
7.245
7.192
7.184
7.178
7.123
7.105
7.086
6.970
6.961
6.751
6.732
6.350
6.347
4.493
3.554
3.515
3.483
3.443
3.180
3.140
2.848
3.55
3.50
3.45
ppm
0.17
1.00
9
8
7
6
5
4
3
2
1
0
ppm
2.31
1.04
2.29
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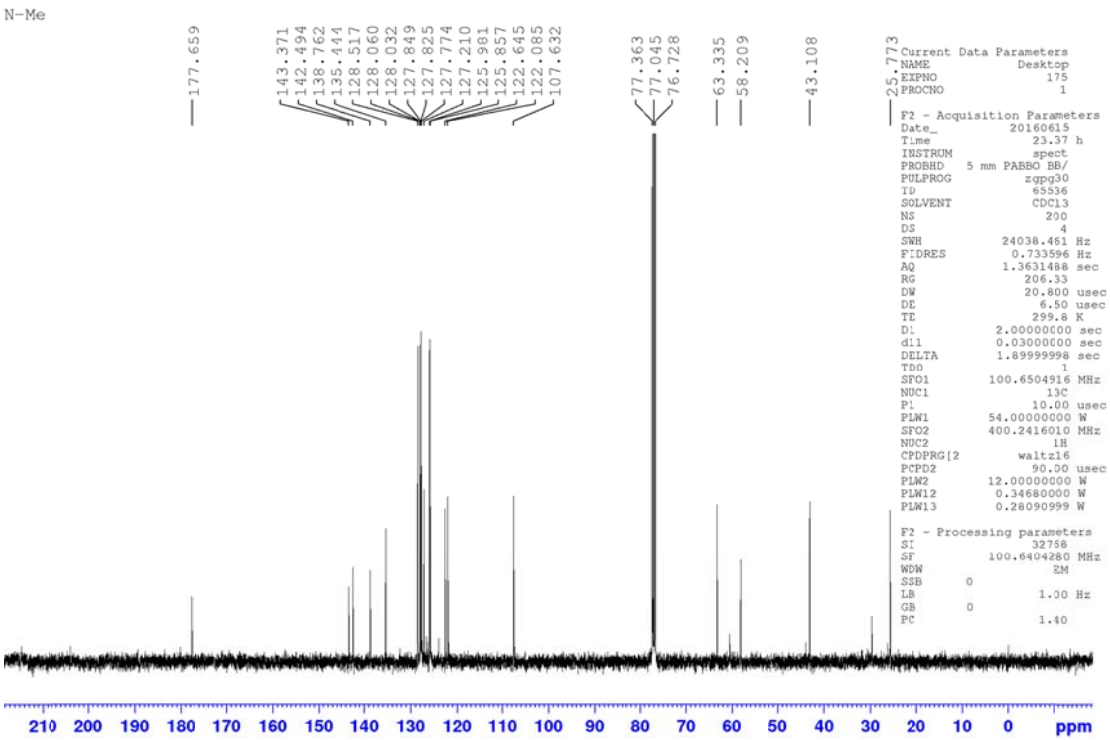
3.02
1.10
2.39
1.05
0.98
0.98
0.17
1.00
1.01
2.98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128.060
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127.825
127.774
127.210
125.981
125.857
122.645
122.085
107.632
77.363
77.045
76.728
63.335
58.209
43.108
25.773
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PULPROG zgpg30
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RG 206.33
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PLW1 54.00000000 W
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NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
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WDW EM
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LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
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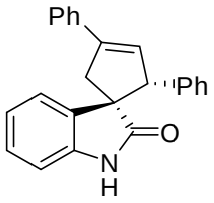
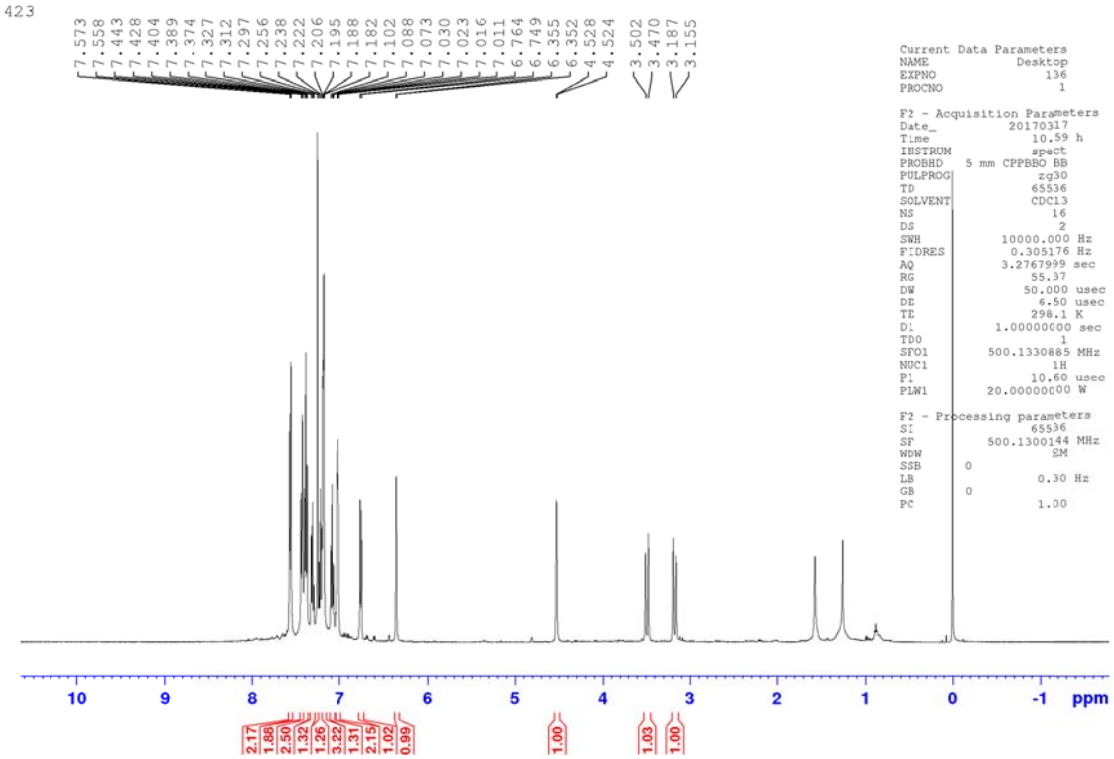
60
50
40
30
20
10
0
ppm


Ph
Ph
O
N
H

3d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7.404
7.389
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7.312
7.297
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7.238
7.222
7.206
7.195
7.188
7.182
7.102
7.088
7.073
7.030
7.023
7.016
7.011
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6.749
6.355
6.352
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3.502
3.470
3.187
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RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
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PLW1 20.00000000 W
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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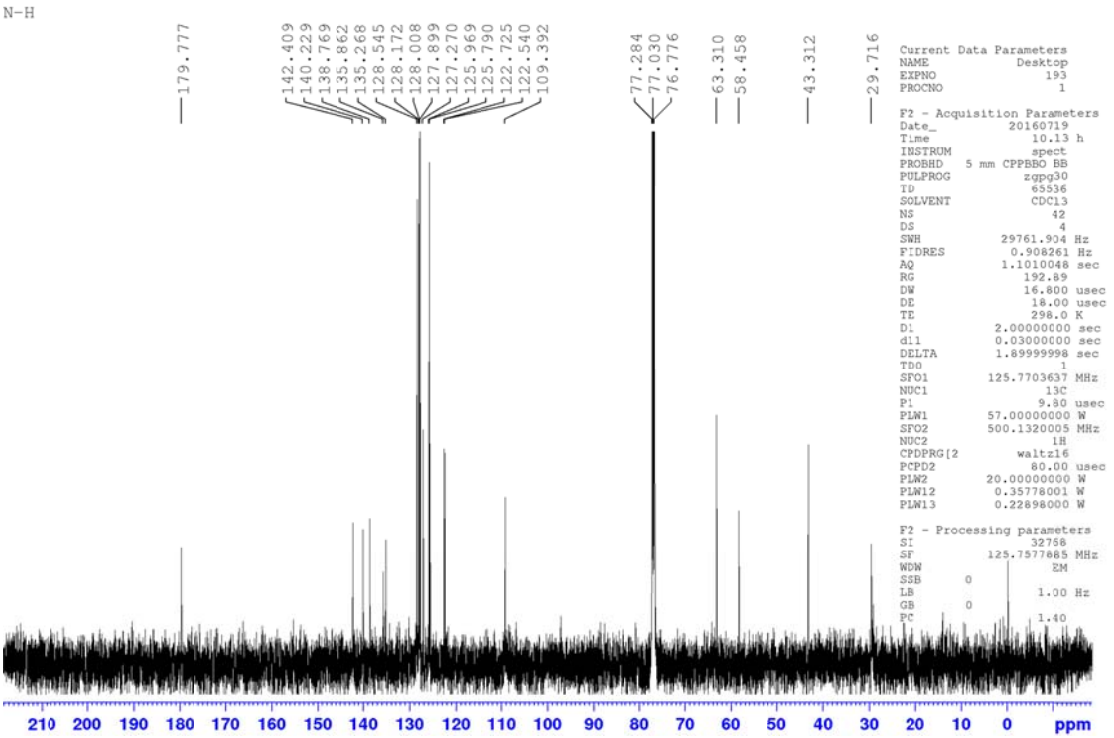
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122.725
122.540
109.392
77.284
77.030
76.776
63.310
58.458
43.312
29.716
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
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NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
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PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
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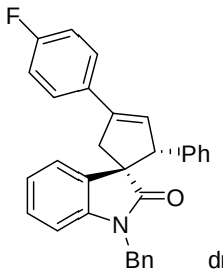
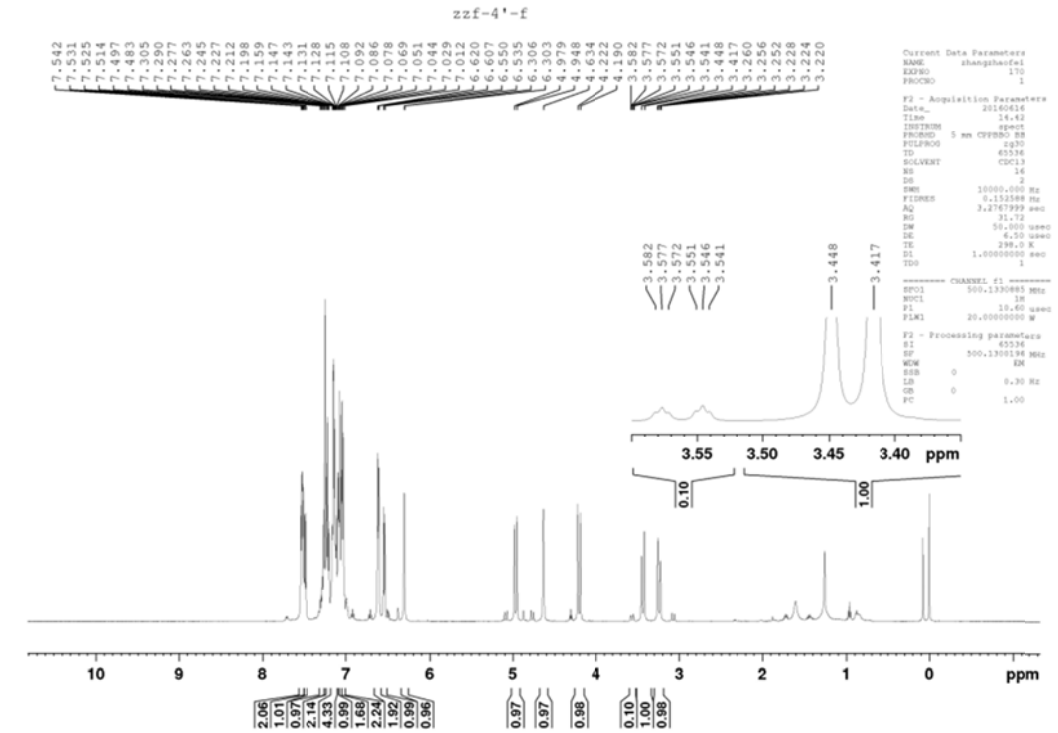

F
Ph
O
N
Bn

dr = 10:1

3e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3.45
3.40
ppm
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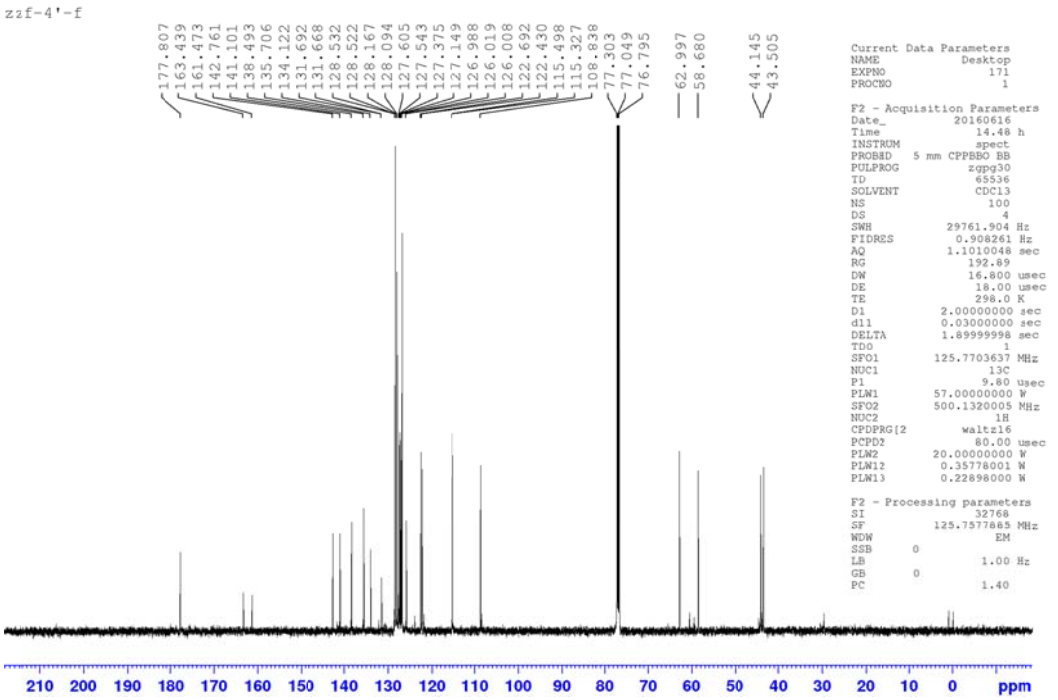
1.00
10
9
8
7
6
5
4
3
2
1
0
ppm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TD 65536
SOLVENT CDCl3
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DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
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PLW13 0.22898000 W
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210
200
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60
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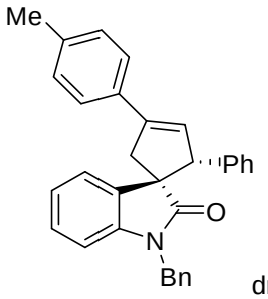
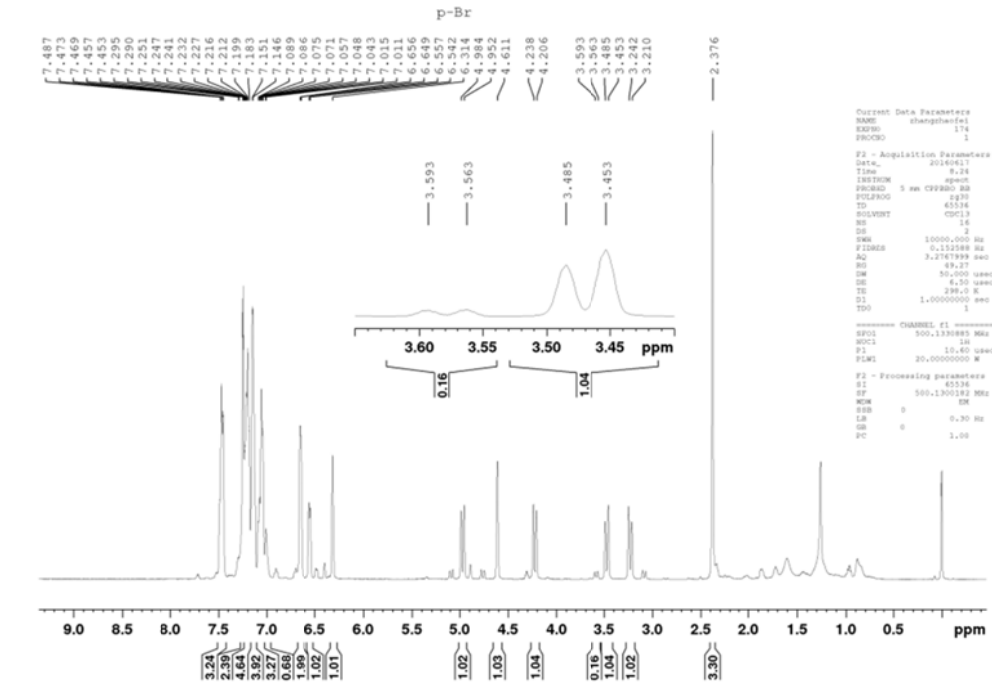
0
ppm


Me
Ph
O
N
Bn

dr = 6:1

3f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3.45
ppm
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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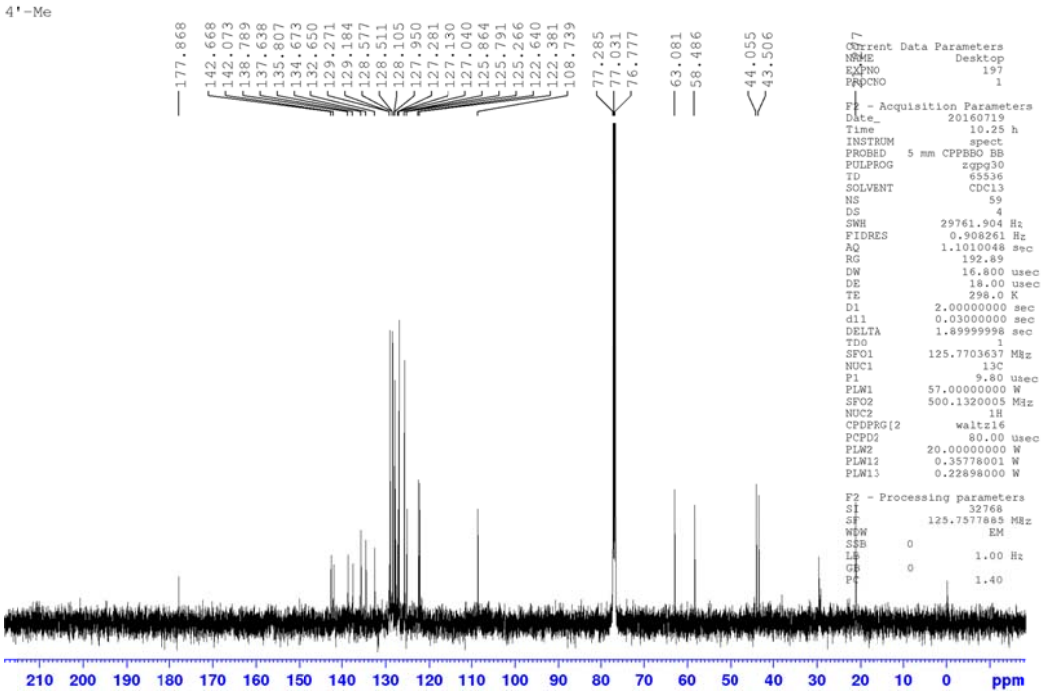
2.0
1.5
1.0
0.5
ppm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210
200
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180
170
160
150
140
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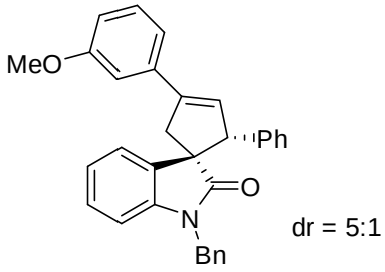
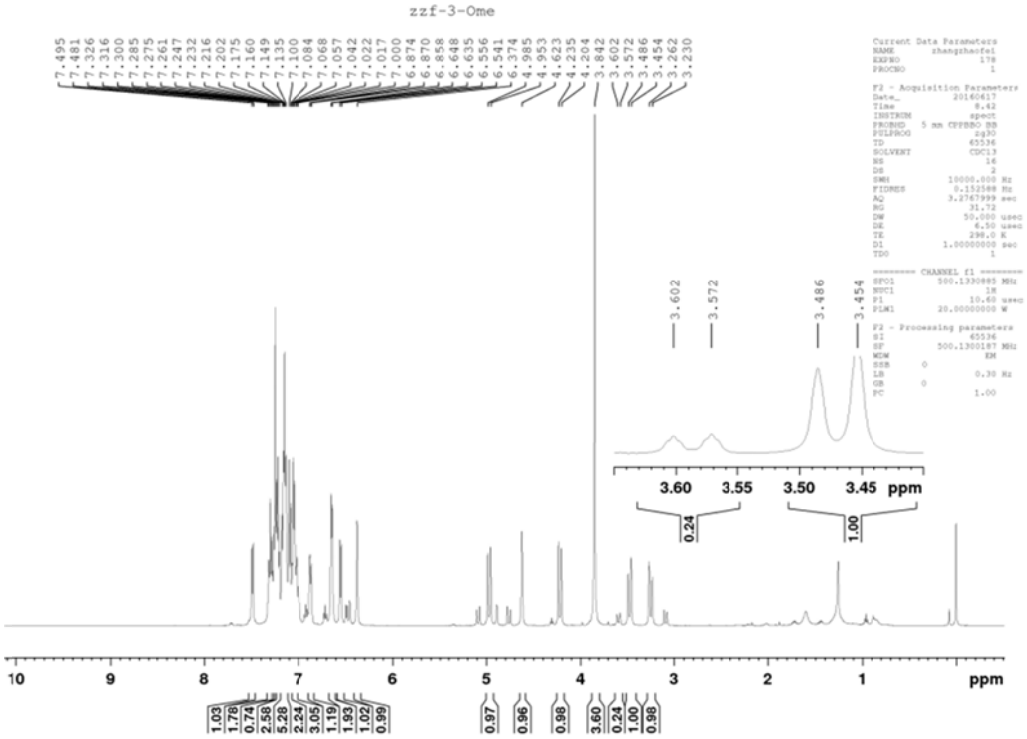
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


MeO
Ph
O
N
Bn

dr = 5:1

3g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7.316
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7.261
7.247
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7.216
7.202
7.175
7.160
7.149
7.135
7.100
7.084
7.068
7.057
7.042
7.022
7.017
7.000
6.874
6.870
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6.648
6.635
6.556
6.541
6.374
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4.953
4.623
4.235
4.204
3.842
3.602
3.572
3.486
3.454
3.262
3.230
3.60
3.55
3.50
3.45 ppm
0.24
1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.03
1.78
0.74
2.58
5.28
2.24
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0.96
0.98
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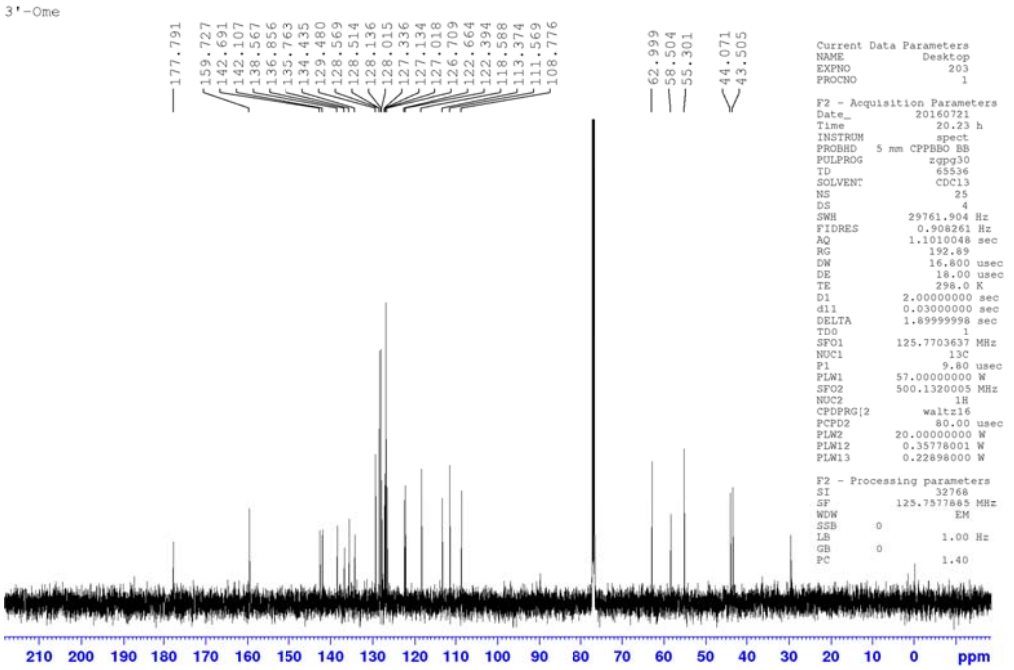
1.00
0.98


3'-Ome
177.791
159.727
142.691
142.107
138.567
136.856
135.763
134.435
129.480
128.569
128.514
128.136
128.015
127.336
127.134
127.018
126.709
122.664
122.394
118.588
113.374
111.569
108.776
62.999
58.504
55.301
44.071
43.505
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200
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180
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140
130
120
110
100
90
80
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60
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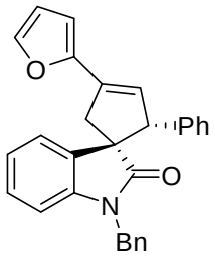
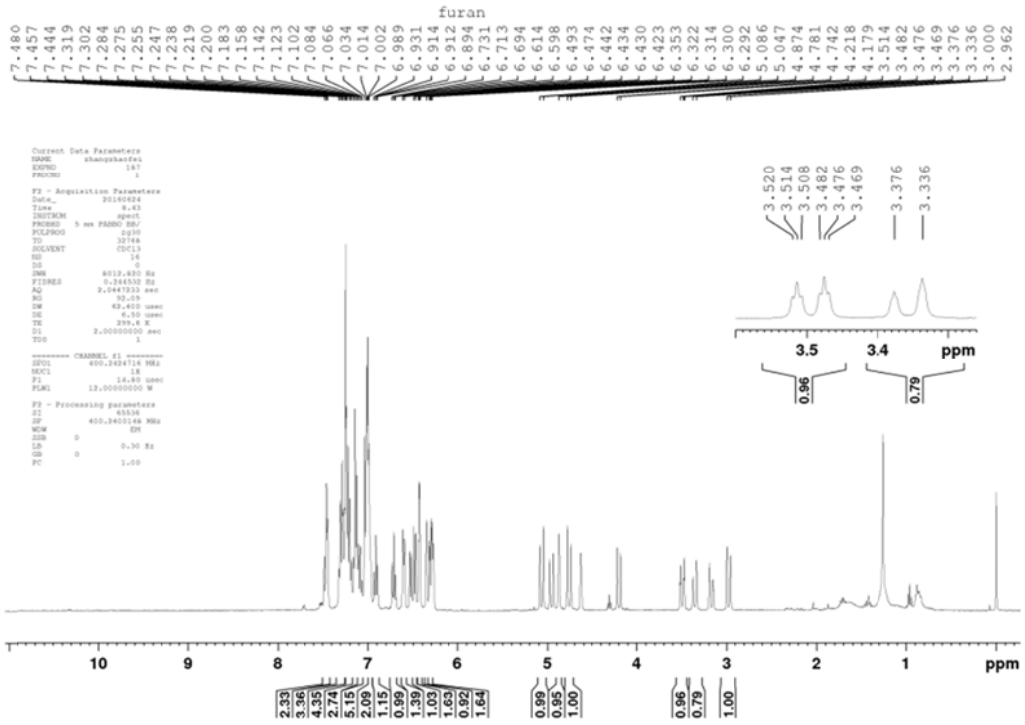
30
20
10
0
ppm


O
Ph
O
N
Bn

dr = 1.2:1

3h


furan
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7.457
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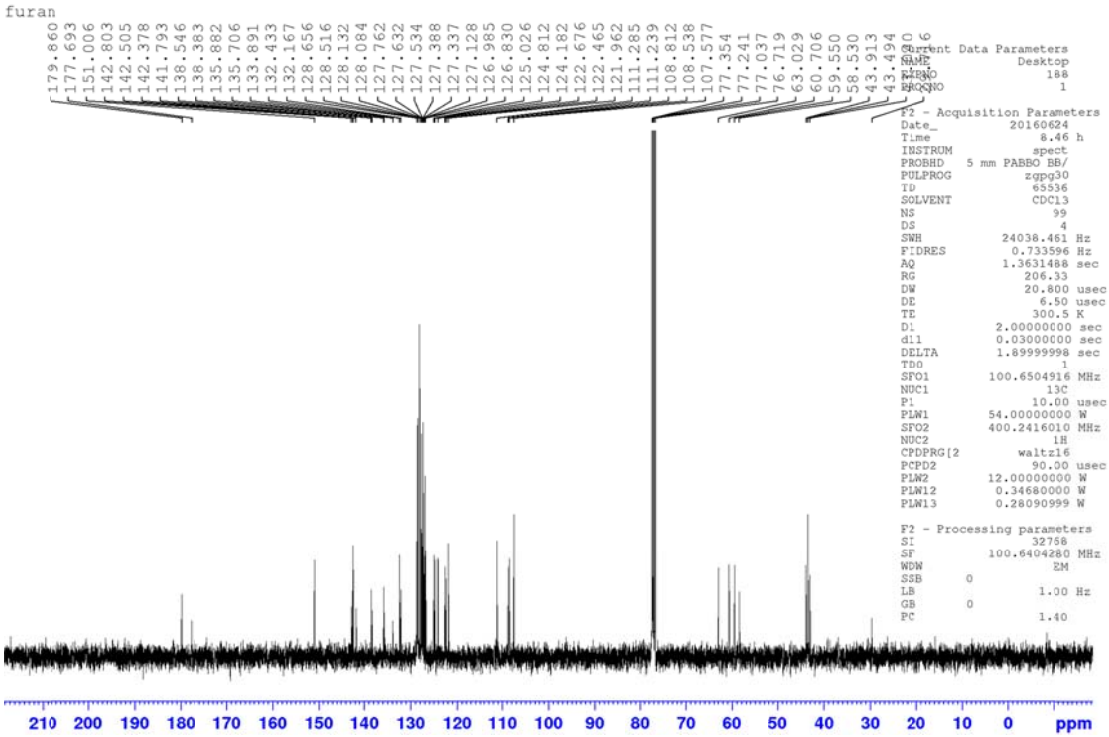
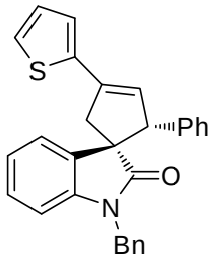
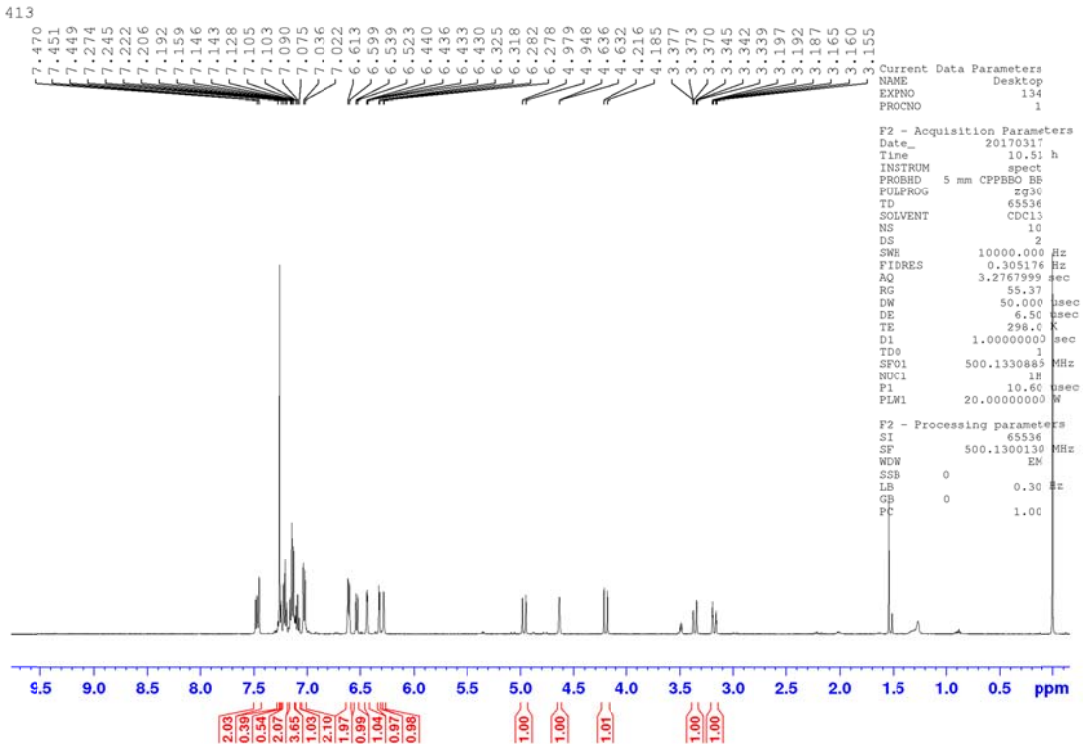
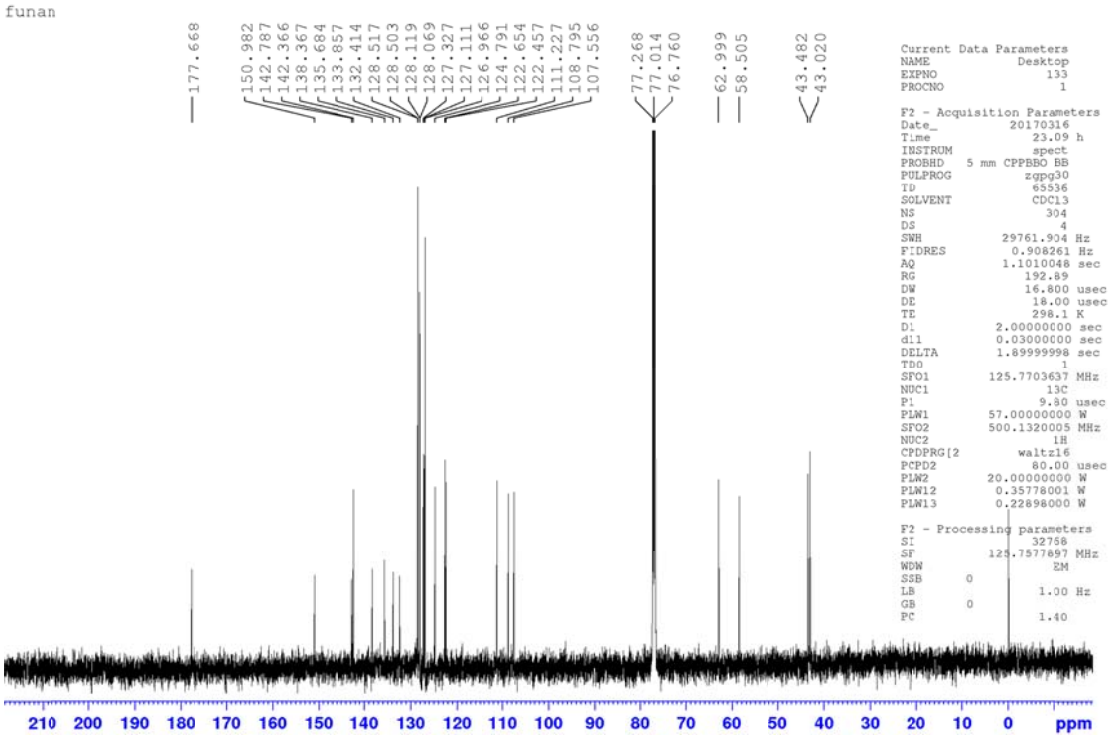
7.319
7.302
7.284
7.275
7.255
7.247
7.238
7.219
7.200
7.183
7.158
7.142
7.123
7.102
7.084
7.066
7.034
7.014
7.002
6.989
6.931
6.914
6.912
6.894
6.731
6.713
6.694
6.614
6.598
6.493
6.474
6.442
6.434
6.430
6.423
6.353
6.322
6.314
6.300
6.292
5.086
5.047
4.874
4.781
4.742
4.218
4.179
3.514
3.482
3.476
3.469
3.376
3.336
3.000
2.962
3.520
3.514
3.508
3.482
3.476
3.469
3.376
3.336
3.5
3.4
ppm
0.96
0.79
10
9
8
7
6
5
4
3
2
1
ppm
2.33
3.36
4.35
2.74
5.15
2.09
1.15
0.99
1.39
1.03
1.63
0.92
1.64
0.99
0.95
1.00
0.96
0.79
1.00

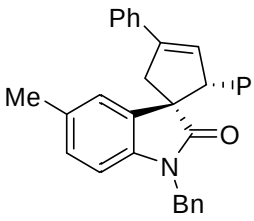

furan
179.860
177.693
151.006
142.803
142.505
142.378
141.793
138.546
138.383
135.882
135.706
133.891
132.433
132.167
128.656
128.516
128.132
128.084
127.762
127.632
127.534
127.388
127.337
127.128
126.985
126.830
125.026
124.812
124.182
122.676
122.465
121.962
111.285
111.239
108.812
108.538
107.577
77.354
77.241
77.037
76.719
63.029
60.706
59.550
58.530
43.913
43.494
Current Data Parameters
NAME Desktop
EXPNO 188
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160624
Time 8.46 h
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 99
DS 4
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 1.3631488 sec
RG 206.33
DW 20.800 usec
DE 6.50 usec
TE 300.5 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 100.6504916 MHz
NUC1 13C
P1 10.00 usec
PLW1 54.00000000 W
SFO2 400.2416010 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 12.00000000 W
PLW12 0.34680000 W
PLW13 0.28090999 W
F2 - Processing parameters
SI 32768
SF 100.6404280 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


S
Ph
O
N
Bn

3i

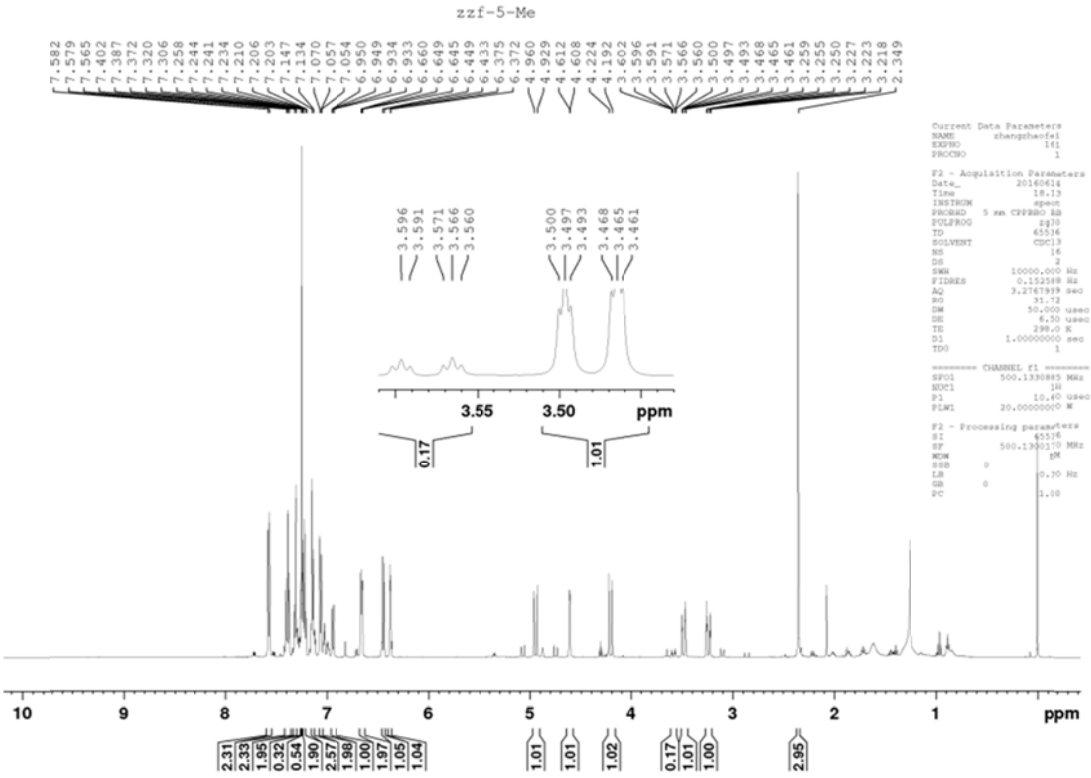

413
7.470 7.451 7.449 7.274 7.245 7.222 7.206 7.192 7.159 7.146 7.143 7.128 7.105 7.103 7.090 7.075 7.036 7.022 6.613 6.599 6.539 6.523 6.440 6.436 6.433 6.430 6.325 6.318 6.282 6.278 4.979 4.948 4.636 4.632 4.216 4.185 3.377 3.373 3.370 3.345 3.342 3.339 3.197 3.192 3.187 3.165 3.160 3.155
Current Data Parameters
NAME Desktop
EXPNO 134
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170317
Time 10.51 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 55.37
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.03 0.39 0.54 2.07 3.65 1.03 2.10 1.97 0.99 1.04 0.97 0.98 1.00 1.00 1.01 1.00 1.00

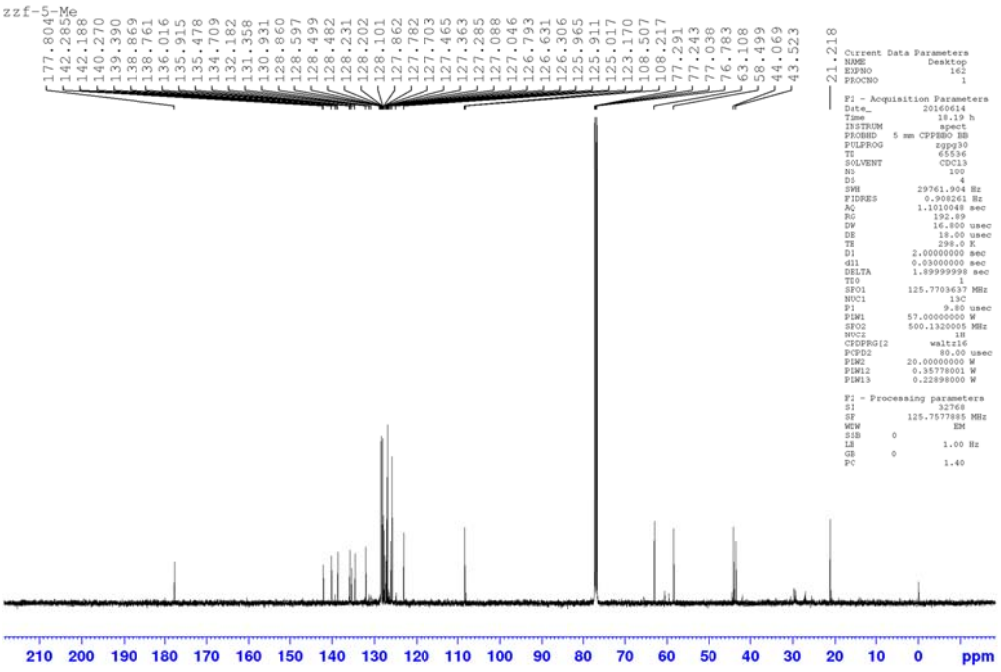

funan
177.668 150.982 142.787 142.366 138.367 135.684 133.857 132.414 128.517 128.503 128.119 128.069 127.327 127.111 126.966 124.791 122.654 122.457 111.227 108.795 107.556 77.268 77.014 76.760 62.999 58.505 43.482 43.020
Current Data Parameters
NAME Desktop
EXPNO 133
PROCNO 1
F2 - Acquisition Parameters
Date_ 20170316
Time 23.09 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 304
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577697 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

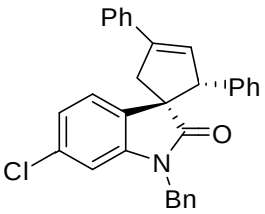


dr = 6:1

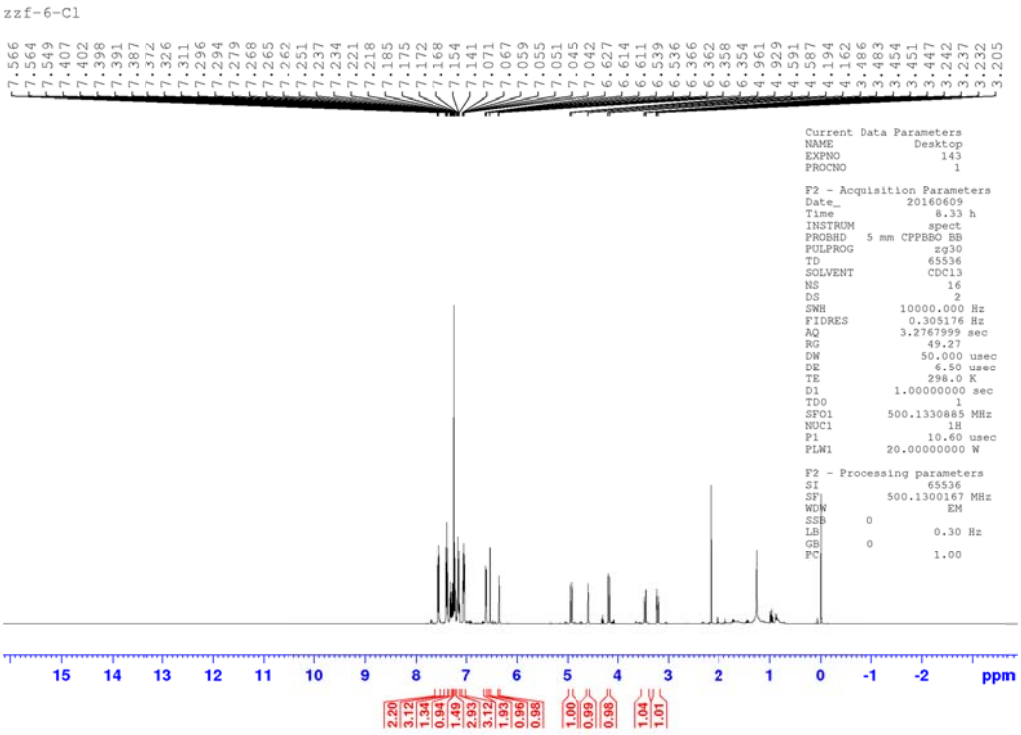
3j

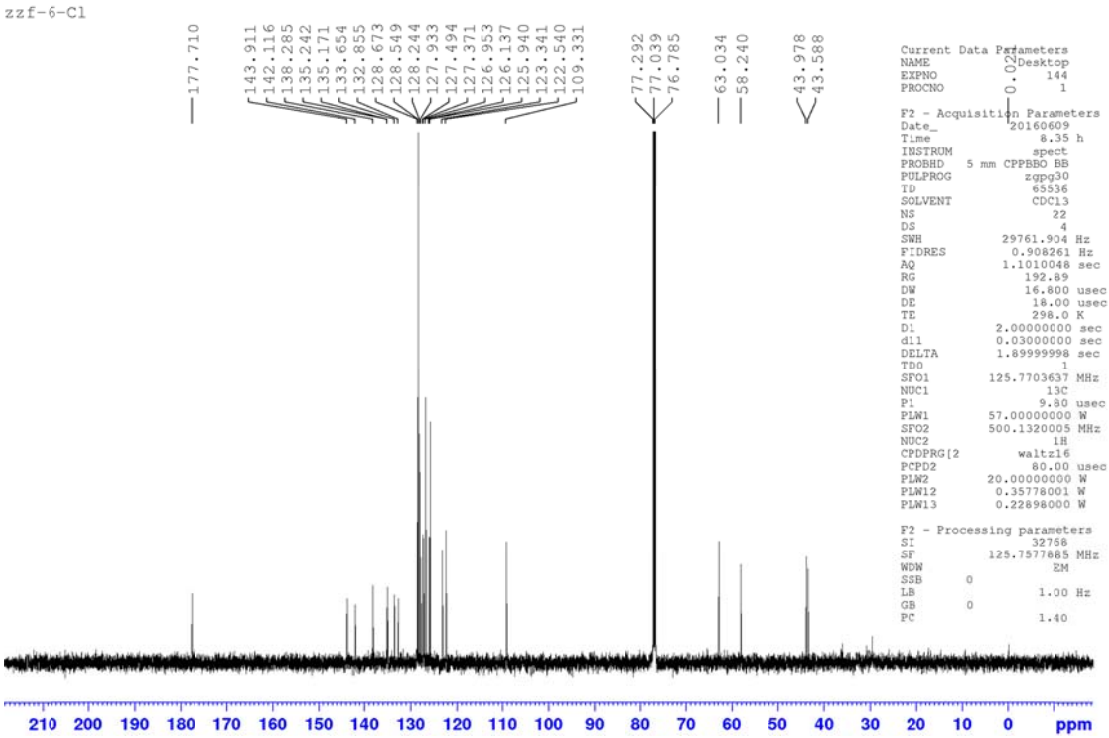


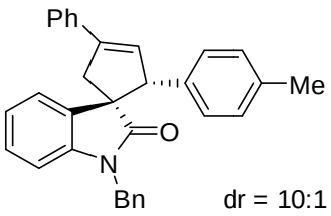



Ph
Ph
O
N
Cl
Bn

3k

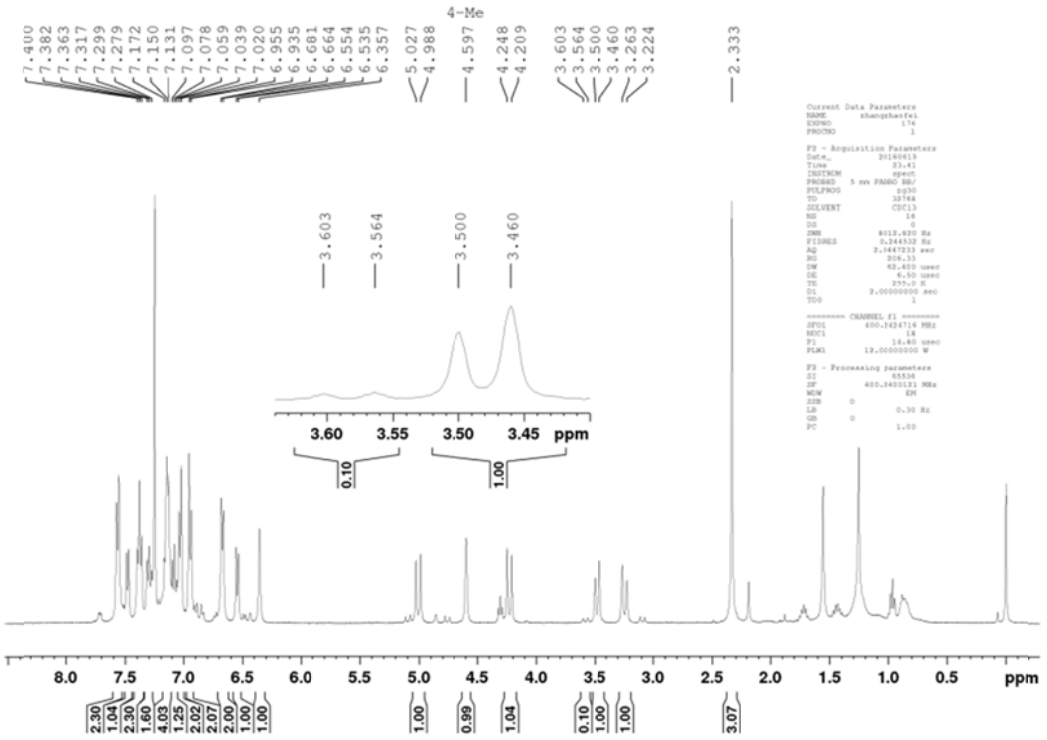

zzf-6-Cl
7.566
7.564
7.549
7.407
7.402
7.398
7.391
7.387
7.372
7.326
7.311
7.296
7.294
7.279
7.268
7.265
7.262
7.251
7.237
7.234
7.221
7.218
7.185
7.175
7.172
7.168
7.154
7.141
7.071
7.067
7.059
7.055
7.051
7.045
7.042
6.627
6.614
6.611
6.539
6.536
6.366
6.362
6.358
6.354
4.961
4.929
4.591
4.587
4.194
4.162
3.486
3.483
3.454
3.451
3.447
3.242
3.237
3.232
3.205
Current Data Parameters
NAME Desktop
EXPNO 143
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160609
Time 8.33 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300167 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
2.20 3.12 1.34 0.94 1.49 2.93 3.12 1.93 0.96 0.98
1.00 0.99 0.98
1.04 1.01

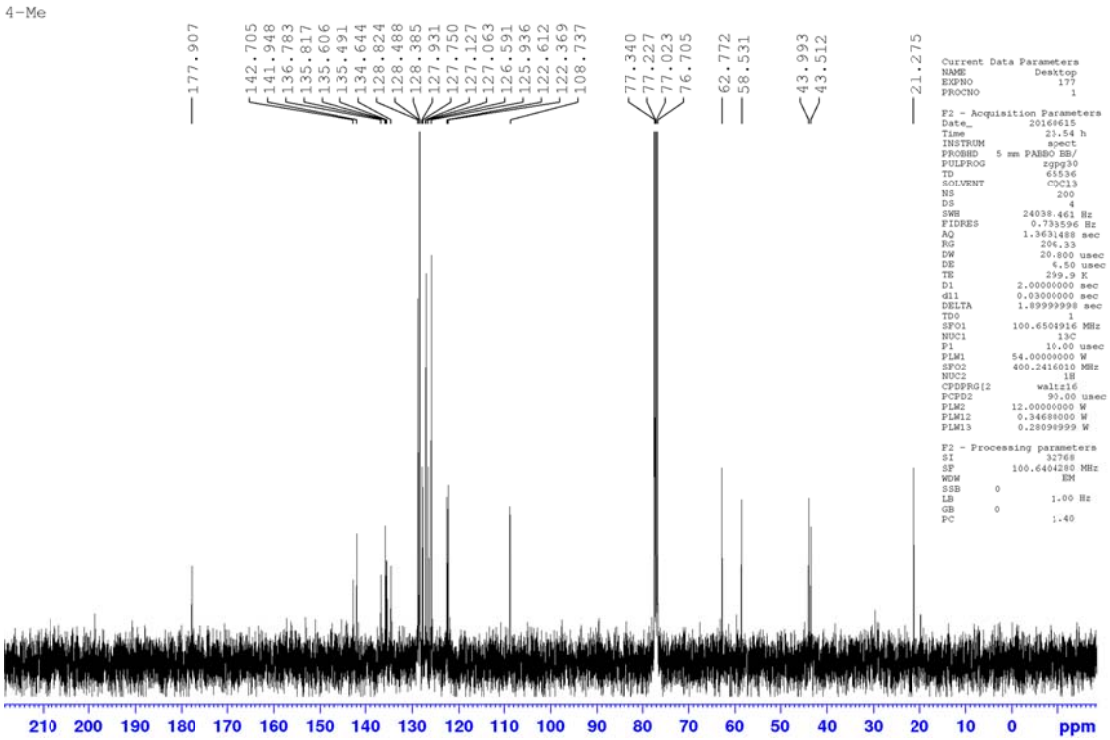

zzf-6-Cl
177.710
143.911
142.116
138.285
135.242
135.171
133.654
132.855
128.673
128.549
128.244
127.933
127.494
127.371
126.953
126.137
125.940
123.341
122.540
109.331
77.292
77.039
76.785
63.034
58.240
43.978
43.588
Current Data Parameters
NAME Desktop
EXPNO 144
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160609
Time 8.35 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 22
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32758
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

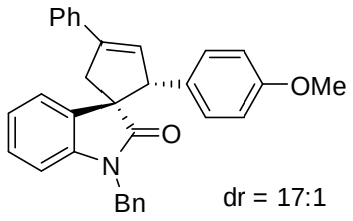

Ph
Me
O
N
Bn

dr = 10:1

3l

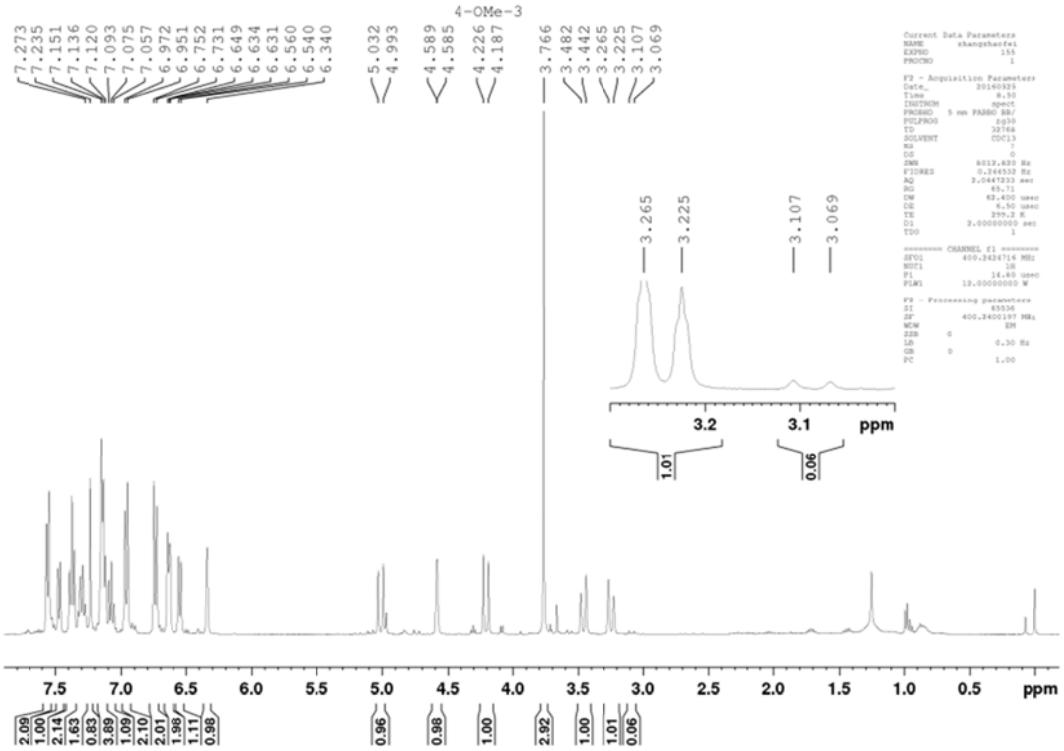

4-Me
7.400
7.382
7.363
7.317
7.299
7.279
7.172
7.150
7.131
7.097
7.078
7.059
7.039
7.020
6.955
6.935
6.681
6.664
6.554
6.535
6.357
5.027
4.988
4.597
4.248
4.209
3.603
3.564
3.500
3.460
3.263
3.224
2.333
3.60
3.55
3.50
3.45 ppm
0.10
1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.30
1.04
2.30
1.60
4.03
1.25
2.02
2.07
2.00
1.00
1.00
1.00
0.99
1.04
0.10
1.00
1.00
3.07

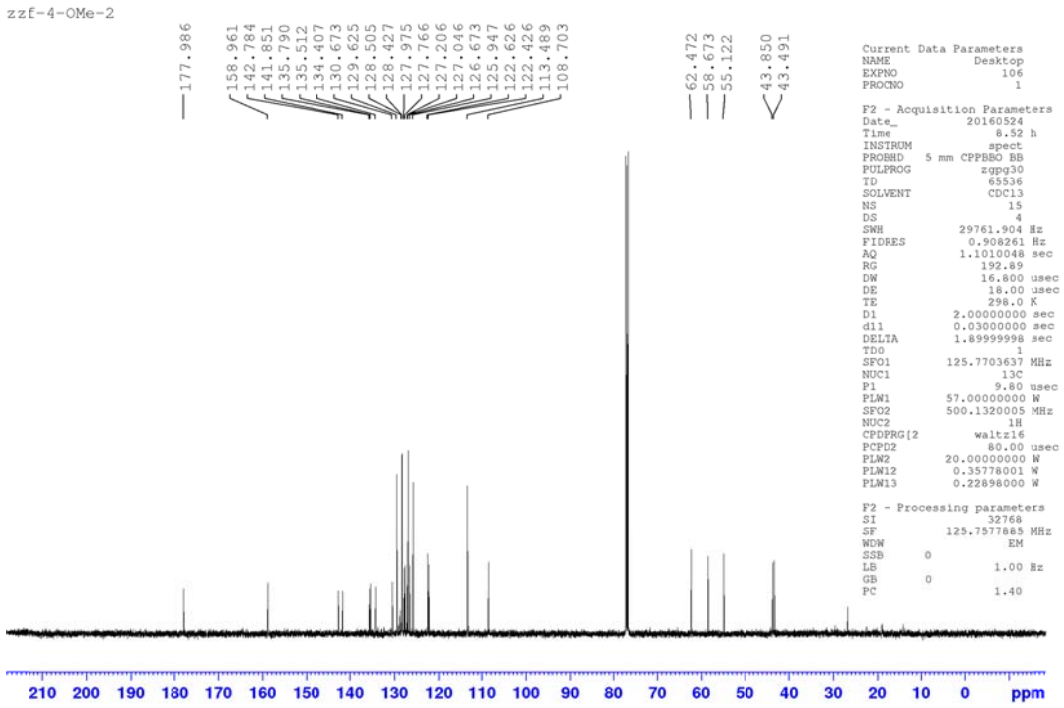

4-Me
177.907
142.705
141.948
136.783
135.817
135.606
135.491
134.644
128.824
128.488
128.385
127.931
127.750
127.127
127.063
126.591
125.936
122.612
122.369
108.737
77.340
77.227
77.023
76.705
62.772
58.531
43.993
43.512
21.275
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



dr = 17:1

3m

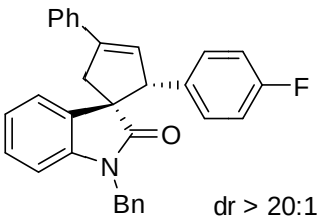




Current Data Parameters
NAME Desktop
EXPNO 106
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160524
Time 8.52 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W

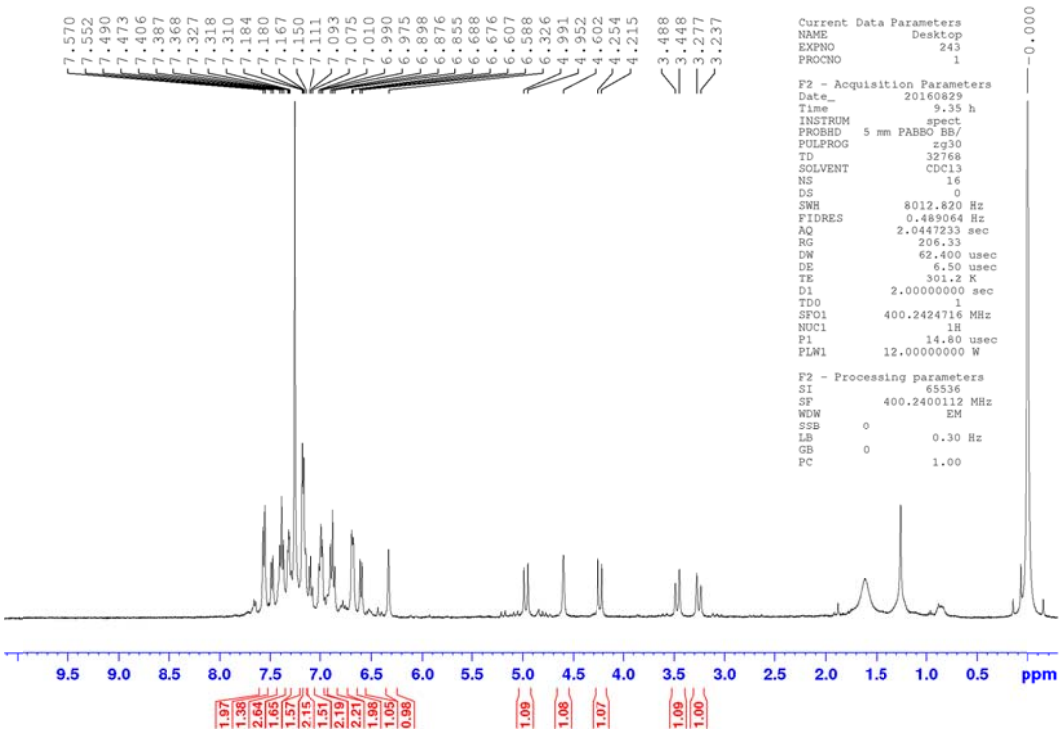
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



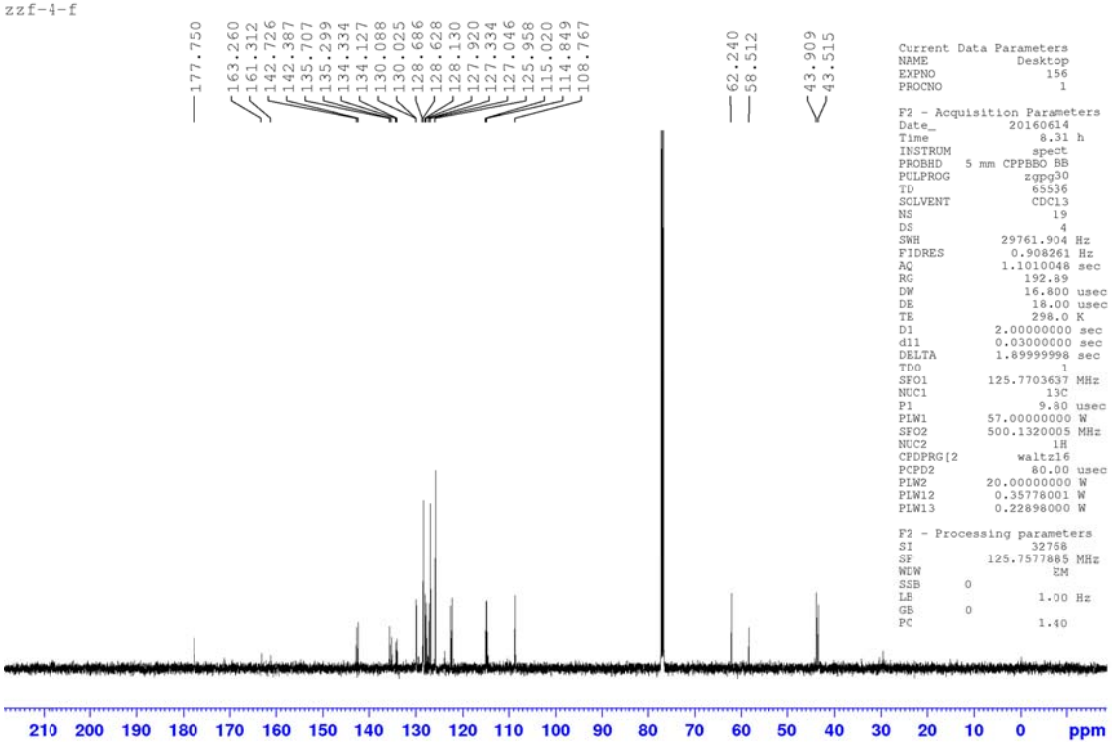
dr > 20:1

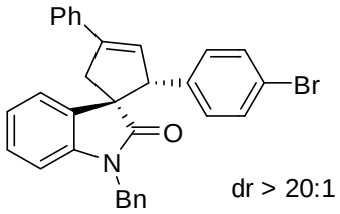
3n

4-f-cat



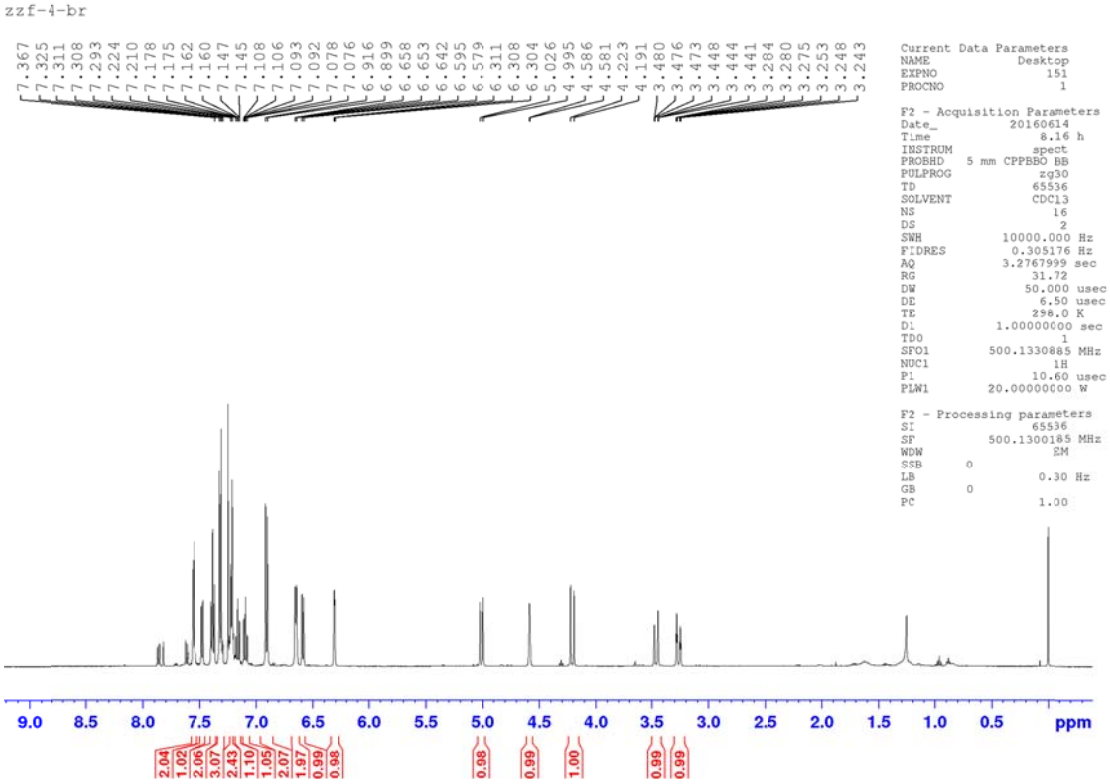
zzf-4-f

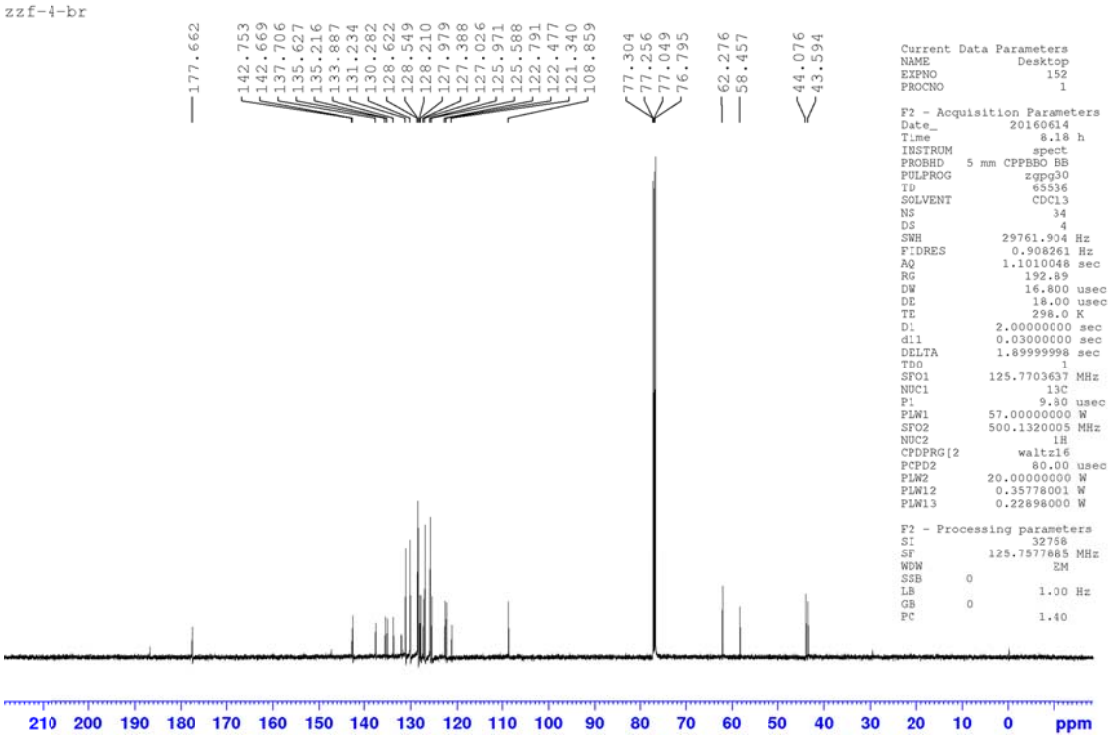


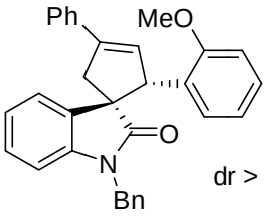

Ph
Br
O
N
Bn

dr > 20:1

3o

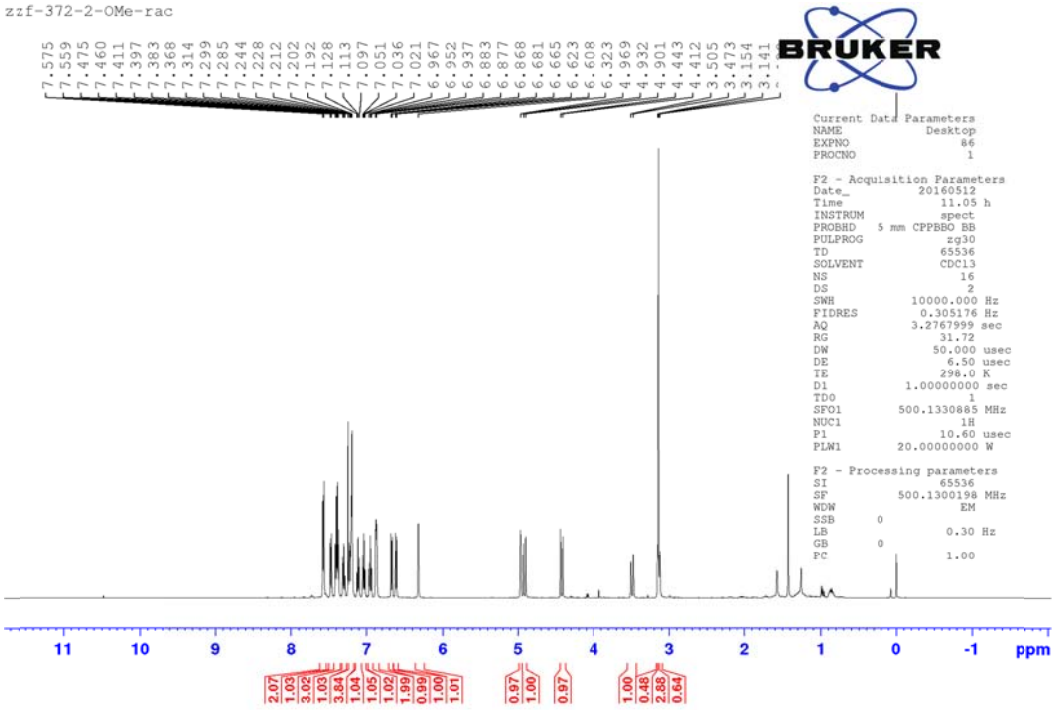

zzf-4-br
7.367 7.325 7.311 7.308 7.293 7.224 7.210 7.178 7.175 7.162 7.160 7.147 7.145 7.108 7.106 7.093 7.092 7.078 7.076 6.916 6.899 6.658 6.653 6.642 6.595 6.579 6.311 6.308 6.304 5.026 4.995 4.586 4.581 4.223 4.191 3.480 3.476 3.473 3.448 3.444 3.441 3.284 3.280 3.275 3.253 3.248 3.243
Current Data Parameters
NAME Desktop
EXPNO 151
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160614
Time 8.16 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300185 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.04 1.02 2.06 3.07 2.43 1.10 1.05 2.07 1.97 0.99 0.98
0.98 0.99 1.00 0.99 0.99

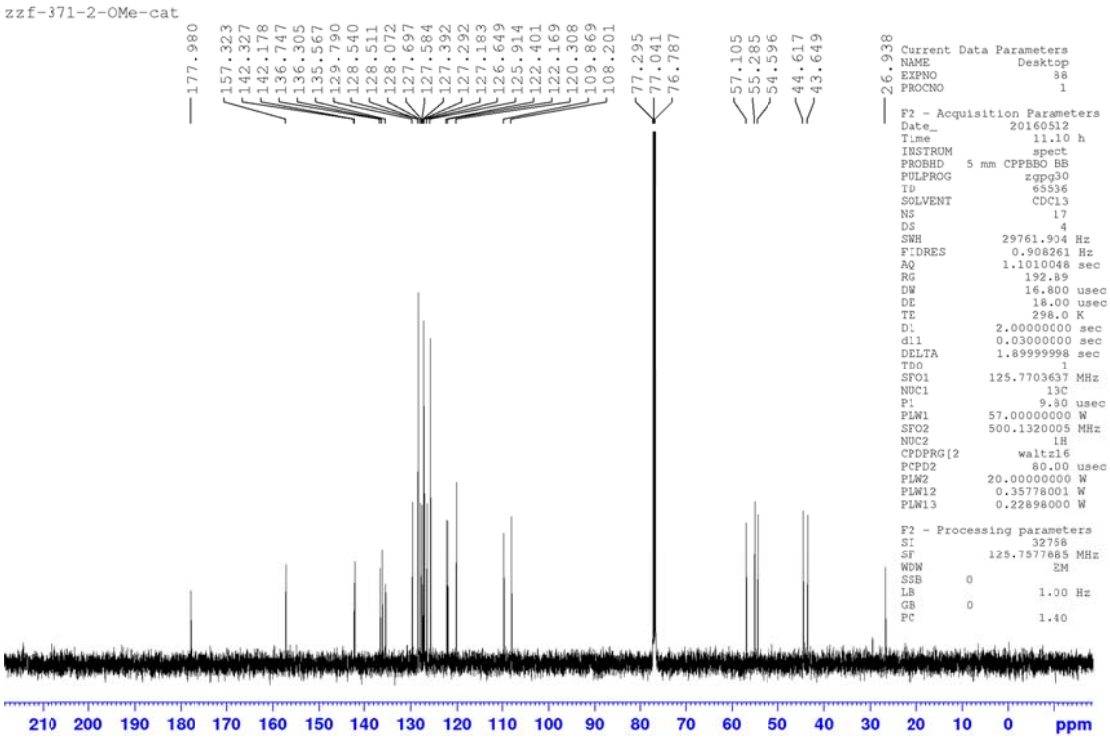

zzf-4-br
177.662
142.753 142.669 137.706 135.627 135.216 133.887 131.234 130.282 128.622 128.549 128.210 127.979 127.388 127.026 125.971 125.588 122.791 122.477 121.340 108.859
77.304 77.256 77.049 76.795
62.276 58.457
44.076 43.594
Current Data Parameters
NAME Desktop
EXPNO 152
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160614
Time 8.18 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 34
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

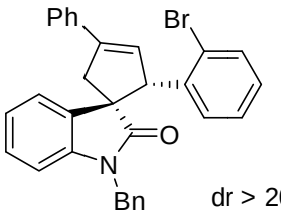

Ph
MeO
O
N
Bn

dr > 20:1

3p

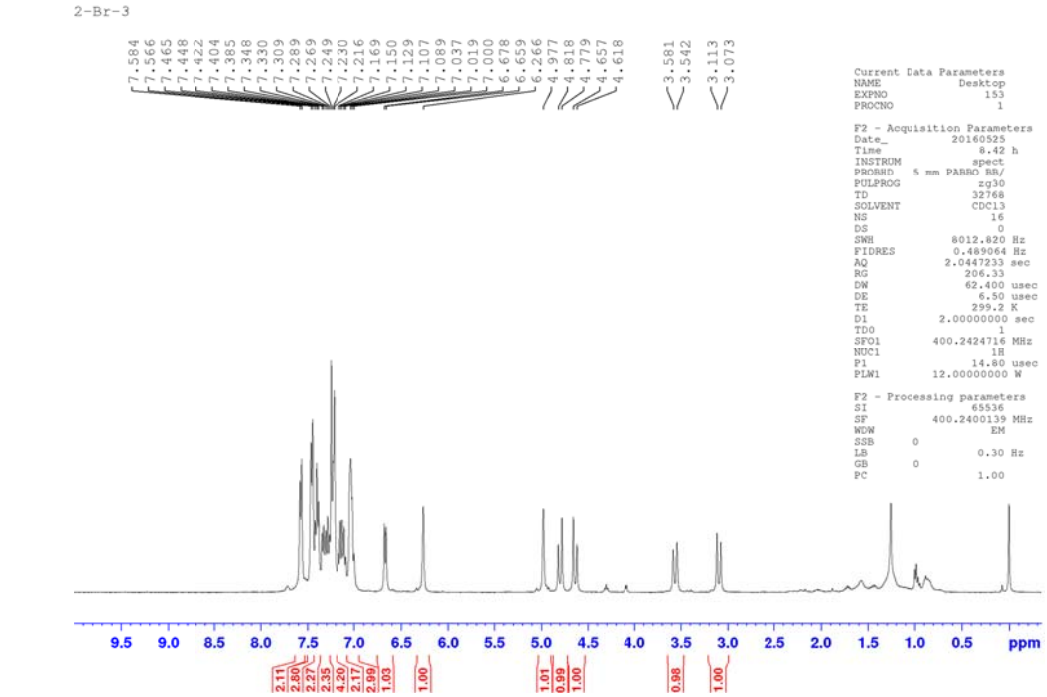

zzf-372-2-OMe-rac
BRUKER
Current Data Parameters
NAME Desktop
EXPNO 86
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160512
Time 11.05 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300198 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

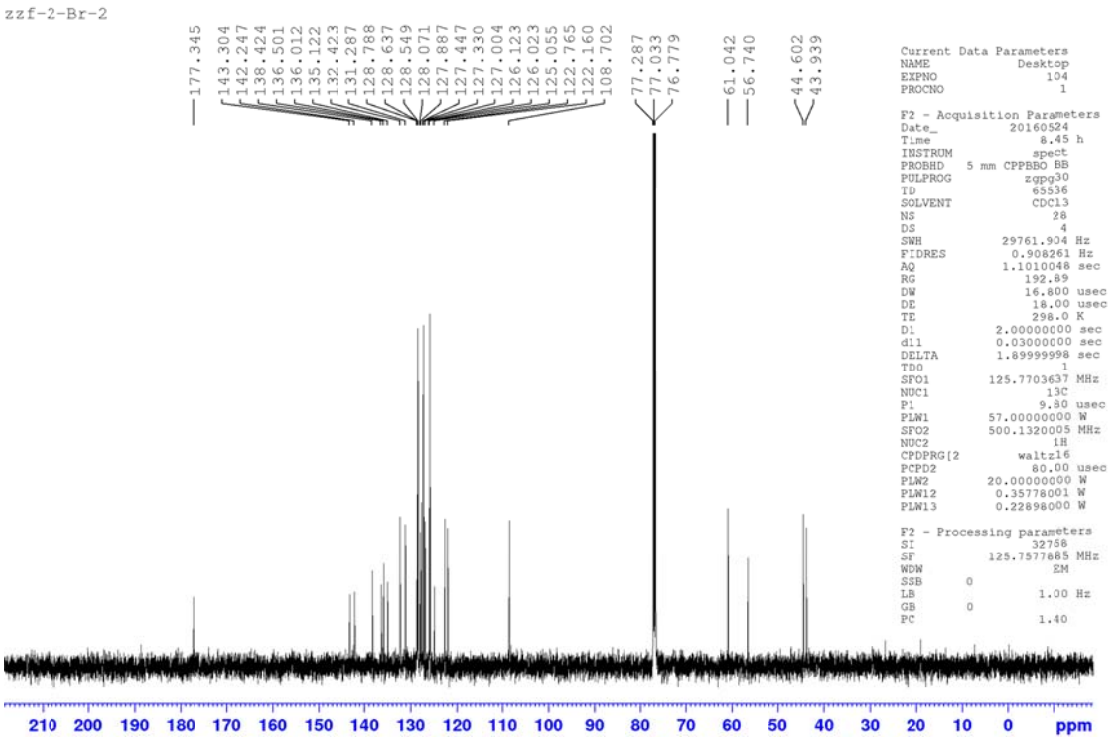

zzf-371-2-OMe-cat
Current Data Parameters
NAME Desktop
EXPNO 88
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160512
Time 11.10 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 17
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

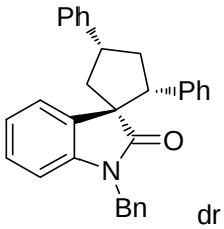

Ph
Br
O
N
Bn

dr > 20:1

3q

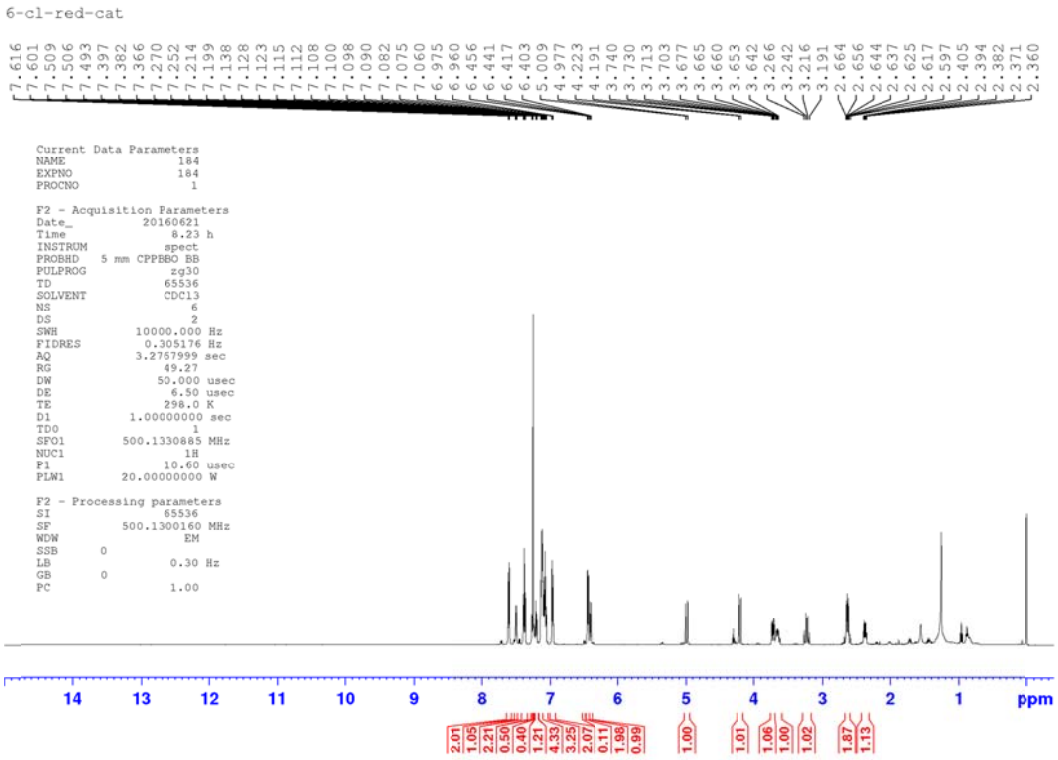

2-Br-3
7.584 7.566 7.465 7.448 7.422 7.404 7.385 7.348 7.330 7.309 7.289 7.269 7.249 7.230 7.216 7.169 7.150 7.129 7.107 7.089 7.037 7.019 7.000 6.678 6.659 6.266 4.977 4.818 4.779 4.657 4.618
3.581 3.542 3.113 3.073
Current Data Parameters
NAME Desktop
EXPNO 153
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160525
Time 8.42 h
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.489064 Hz
AQ 2.0447233 sec
RG 206.33
DW 62.400 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
SFO1 400.2424716 MHz
NUC1 1H
P1 14.80 usec
PLW1 12.00000000 W
F2 - Processing parameters
SI 65536
SF 400.2400139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.11 2.80 2.27 2.35 4.20 2.17 2.99 1.03 1.00 1.01 0.99 1.00 0.98 1.00

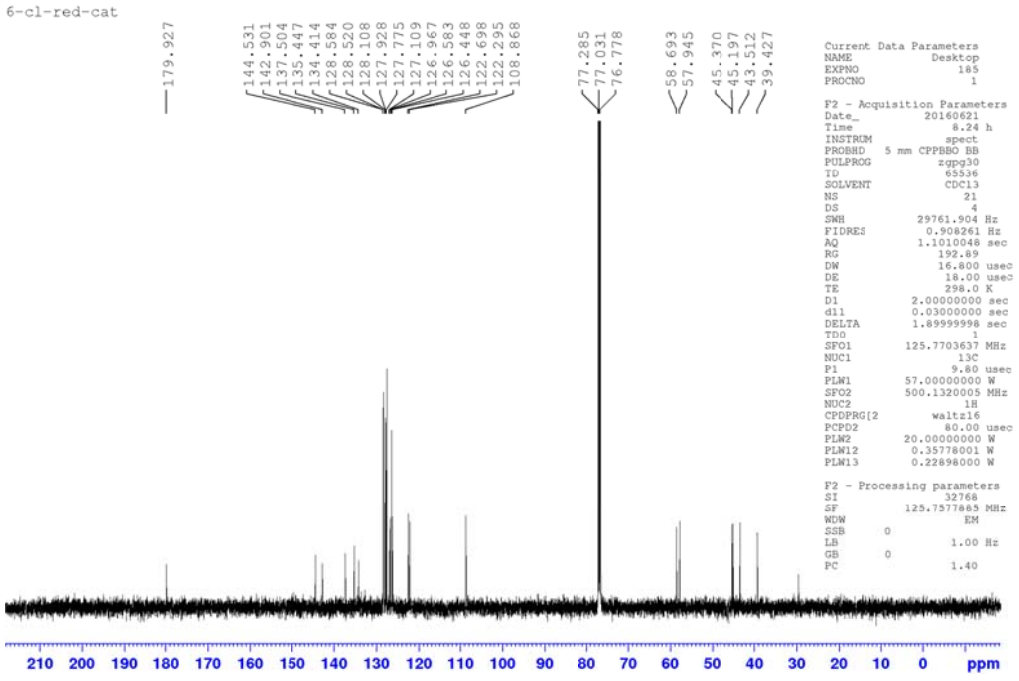

zzf-2-Br-2
177.345 143.304 142.247 138.424 136.501 136.012 135.122 132.423 131.287 128.788 128.637 128.549 128.071 127.887 127.447 127.330 127.004 126.123 126.023 125.055 122.765 122.160 108.702
77.287 77.033 76.779
61.042 56.740
44.602 43.939
Current Data Parameters
NAME Desktop
EXPNO 104
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160524
Time 8.45 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 28
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.30 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

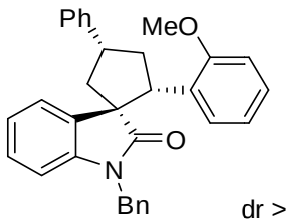

Ph
Ph
O
N
Bn

dr > 20:1

4a

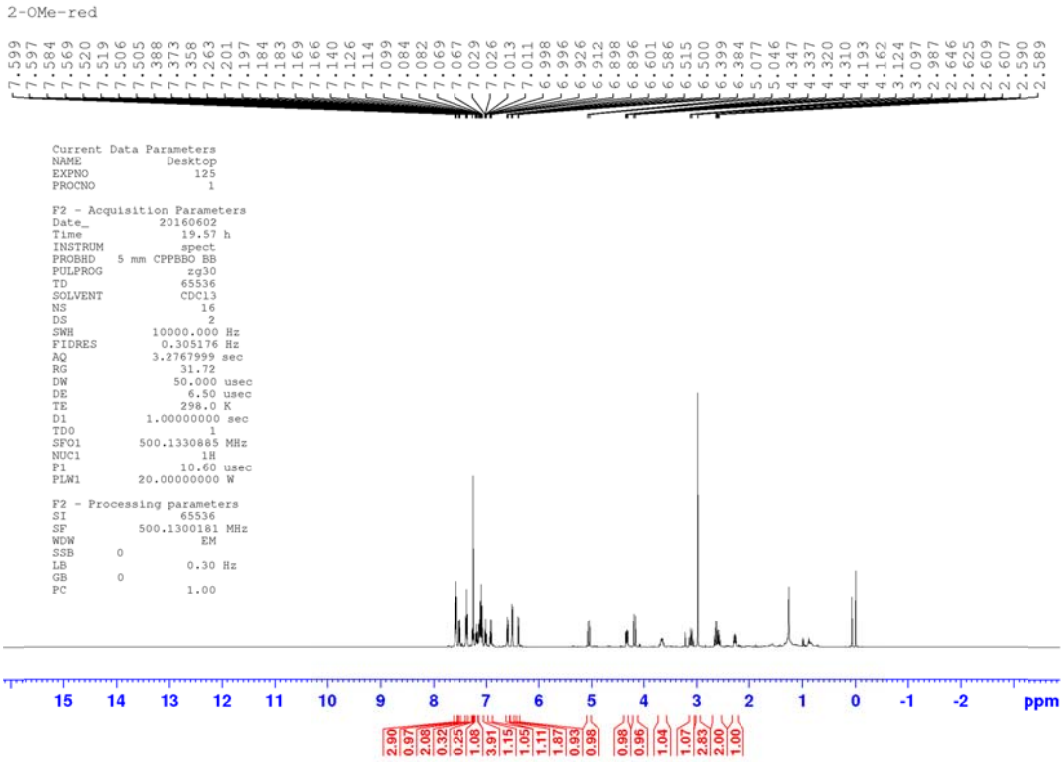

6-cl-red-cat
7.616
7.601
7.509
7.506
7.493
7.397
7.382
7.366
7.270
7.252
7.214
7.199
7.138
7.128
7.123
7.115
7.112
7.108
7.100
7.098
7.090
7.082
7.075
7.060
6.975
6.960
6.456
6.441
6.417
6.403
5.009
4.977
4.223
4.191
3.740
3.730
3.713
3.703
3.677
3.665
3.660
3.653
3.642
3.266
3.242
3.216
3.191
2.664
2.656
2.644
2.637
2.625
2.617
2.597
2.405
2.394
2.382
2.371
2.360
Current Data Parameters
NAME 184
EXPNO 184
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160621
Time 8.23 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 6
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2757999 sec
RG 49.27
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300160 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
2.01 1.05 2.21 0.50 0.40 1.21 4.33 3.25 2.07 0.11 1.98 0.99 1.00 1.01 1.06 1.00 1.02 1.87 1.13

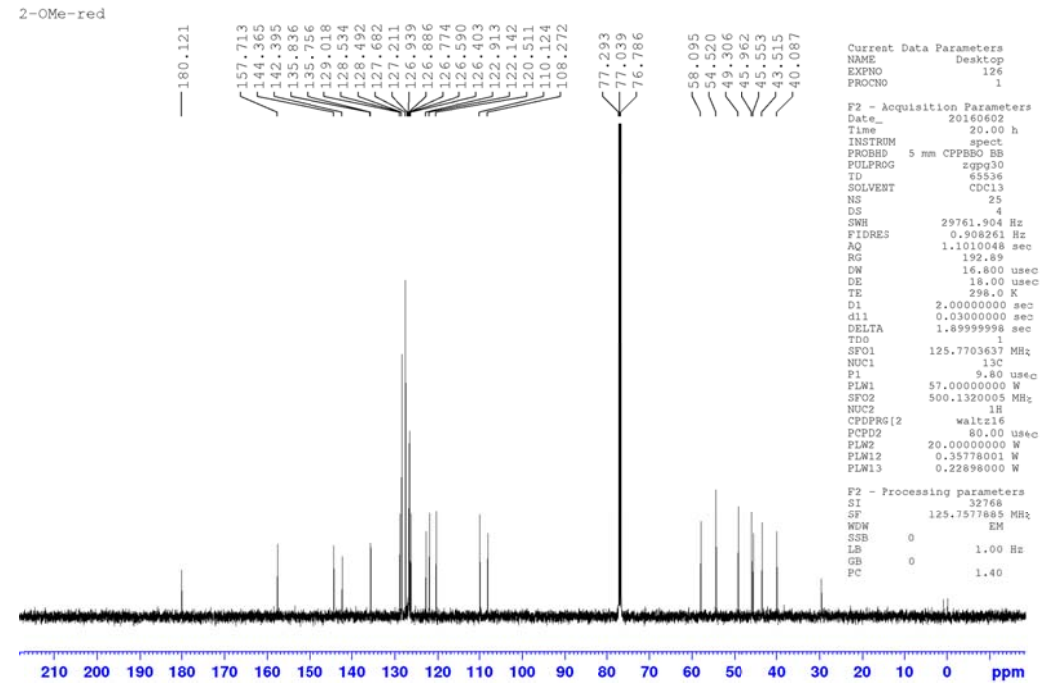

6-cl-red-cat
179.927
144.531
142.901
137.504
135.447
134.414
128.584
128.520
128.108
127.928
127.775
127.109
126.967
126.583
126.448
122.698
122.295
108.868
77.285
77.031
76.778
58.693
57.945
45.370
45.197
43.512
39.427
Current Data Parameters
NAME Desktop
EXPNO 185
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160621
Time 8.24 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 21
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577865 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


Ph
MeO
O
N
Bn

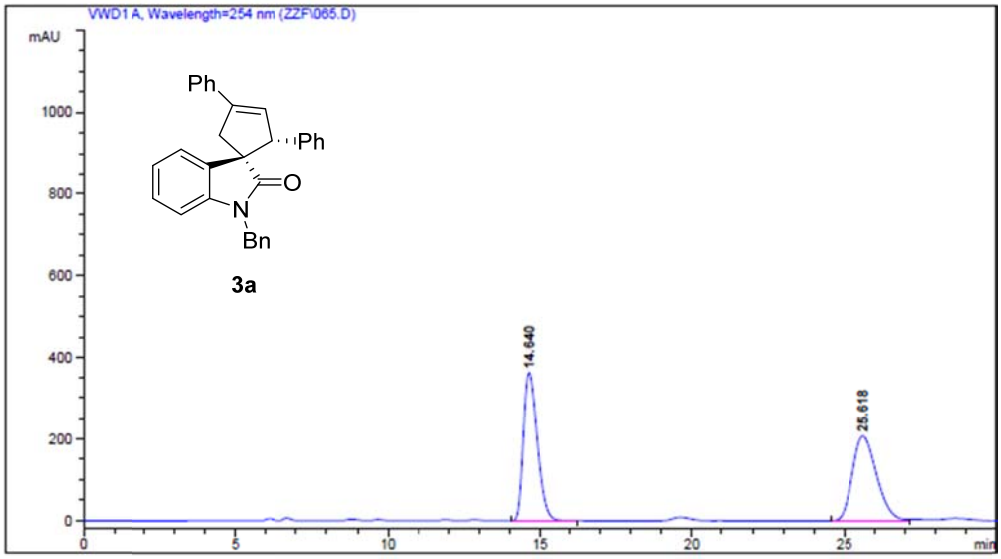
dr > 20:1

4p


2-OMe-red
Current Data Parameters
NAME Desktop
EXPNO 125
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160602
Time 19.57 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 31.72
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
SFO1 500.1330885 MHz
NUC1 1H
P1 10.60 usec
PLW1 20.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300181 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm

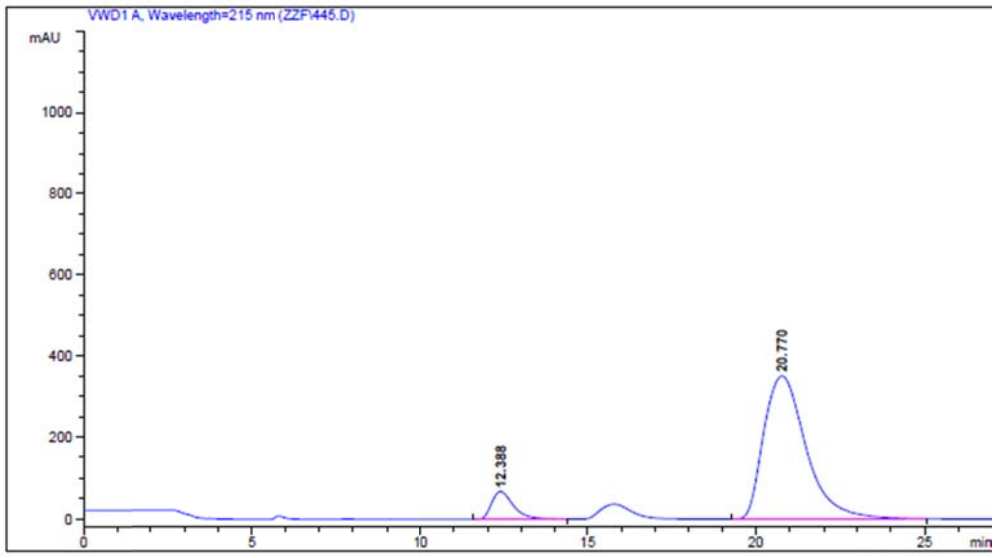

2-OMe-red
Current Data Parameters
NAME Desktop
EXPNO 126
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160602
Time 20.00 h
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 25
DS 4
SWH 29761.904 Hz
FIDRES 0.908261 Hz
AQ 1.1010048 sec
RG 192.89
DW 16.800 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
SFO1 125.7703637 MHz
NUC1 13C
P1 9.80 usec
PLW1 57.00000000 W
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 20.00000000 W
PLW12 0.35778001 W
PLW13 0.22898000 W
F2 - Processing parameters
SI 32768
SF 125.7577885 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

Part IV HPLC Spectra

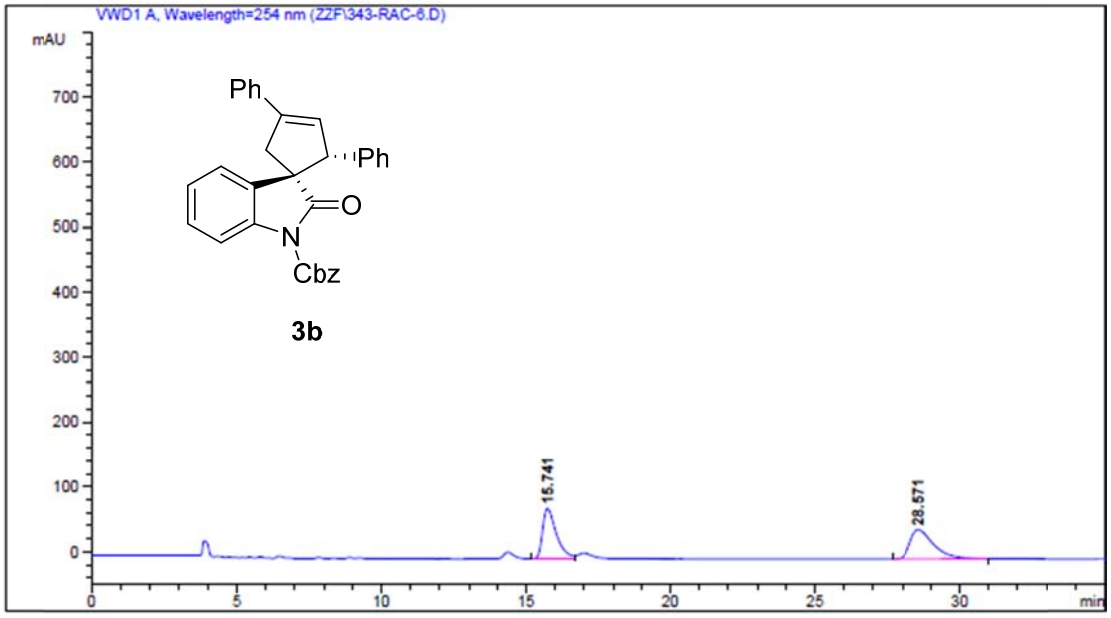


3a

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.640	BB	0.4949	1.14886e4	362.52884	49.9686
2	25.618	BV	0.8765	1.15030e4	206.66826	50.0314
Totals :				2.29916e4	569.19710	

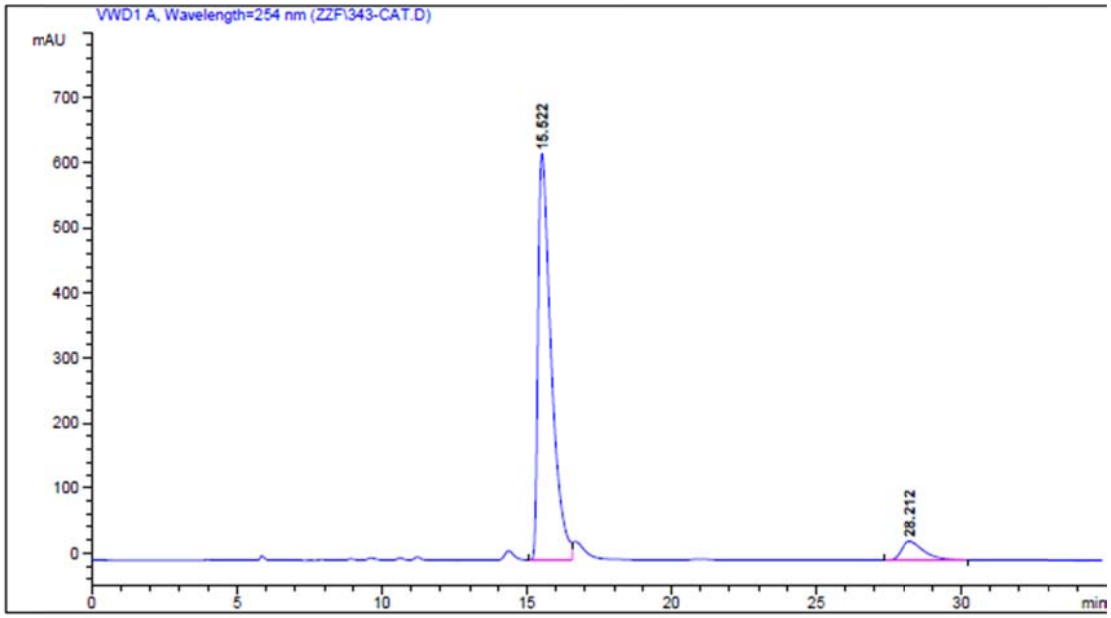


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.388	BB	0.6955	3047.28833	67.51313	9.0986
2	20.770	BB	1.3646	3.04445e4	351.91202	90.9014
Totals :				3.34918e4	419.42515	

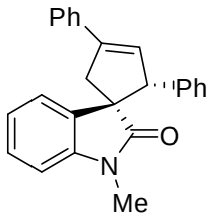


3b

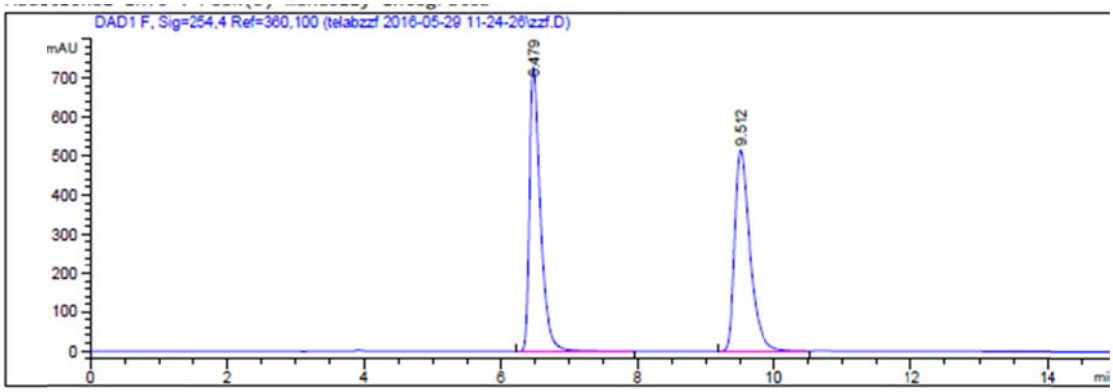
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.741	BV	0.4952	2544.44385	77.19134	49.7412
2	28.571	BB	0.8670	2570.91724	44.83544	50.2588
Totals :				5115.36108	122.02678	



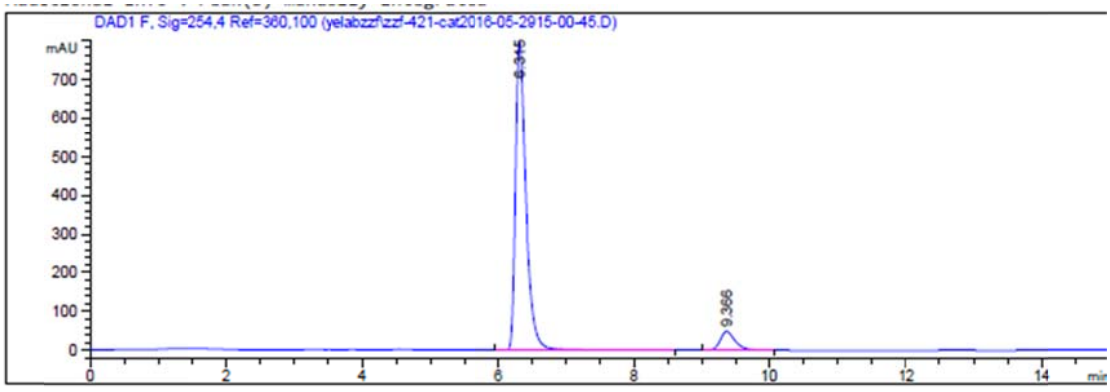
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.522	VV	0.4680	1.95613e4	623.39819	92.5699
2	28.212	BB	0.8194	1570.07825	28.80434	7.4301
Totals :				2.11314e4	652.20253	



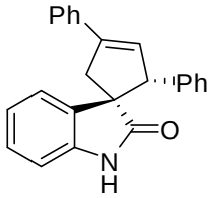
3c



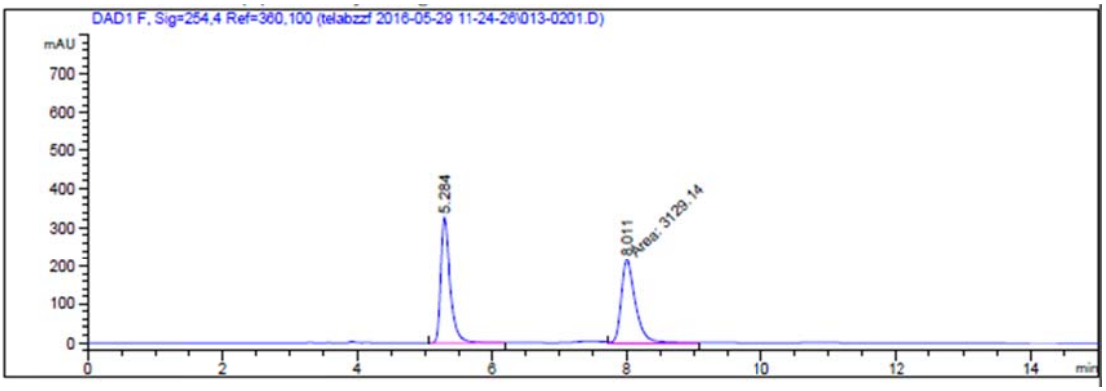
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.479	BB	0.1652	8066.03076	728.26813	50.1292
2	9.512	BB	0.2360	8024.46338	514.27850	49.8708
Totals :				1.60905e4	1242.54663	



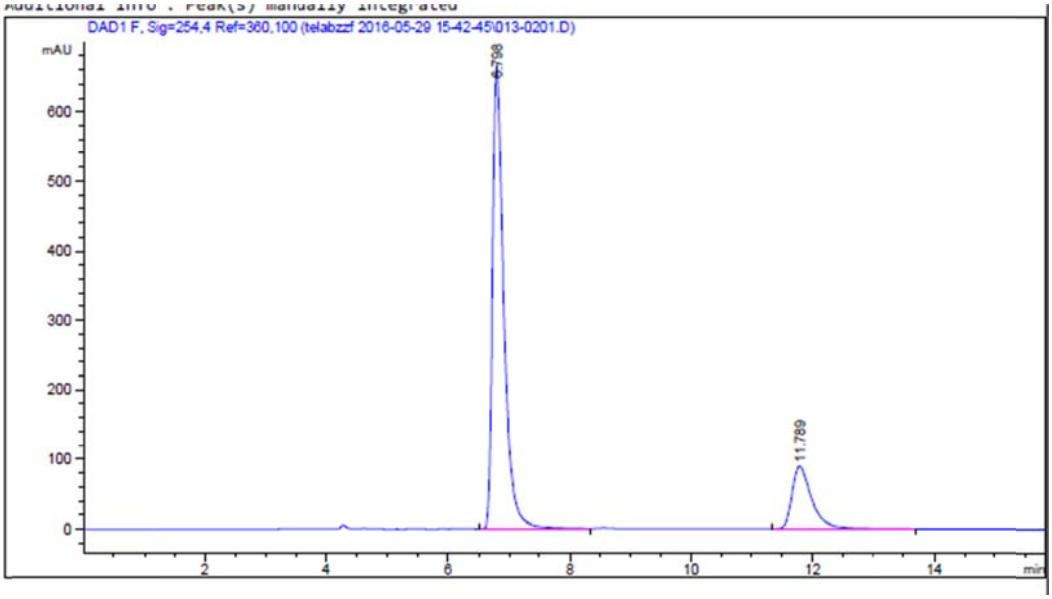
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.315	BB	0.1573	8401.71582	794.67609	91.9655
2	9.366	BB	0.2281	734.01001	48.62725	8.0345
Totals :				9135.72583	843.30334	



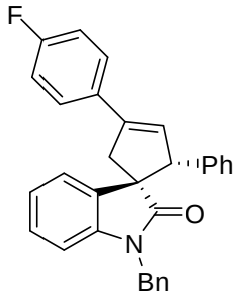
3d



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.284	BB	0.1407	3097.66724	326.36298	49.7473
2	8.011	MM	0.2416	3129.14209	215.82080	50.2527
Totals :				6226.80933	542.18378	

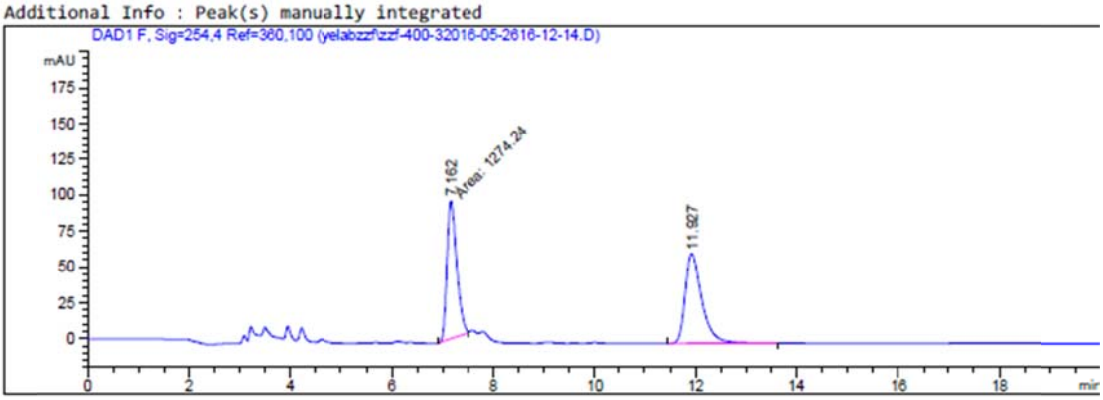


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.798	BB	0.1900	8564.04395	666.16522	81.1841
2	11.789	BB	0.3336	1984.87939	89.20419	18.8159
Totals :				1.05489e4	755.36942	



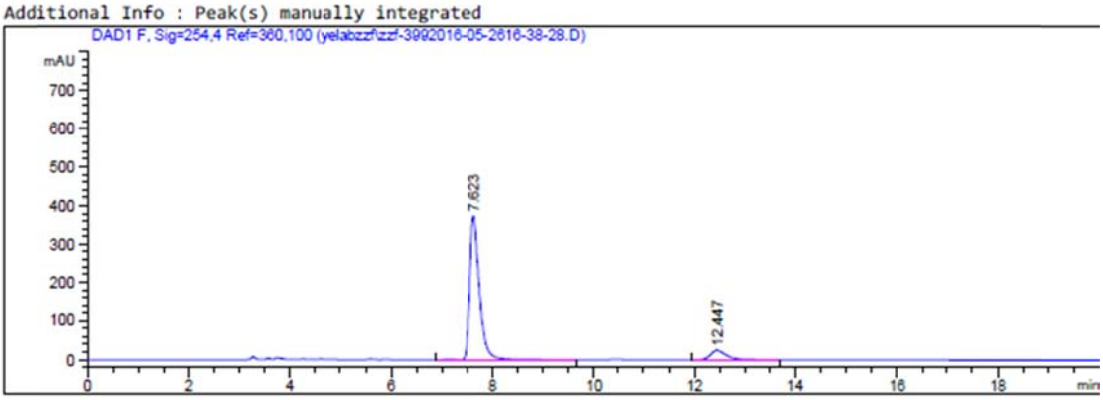
3e

Additional Info : Peak(s) manually integrated

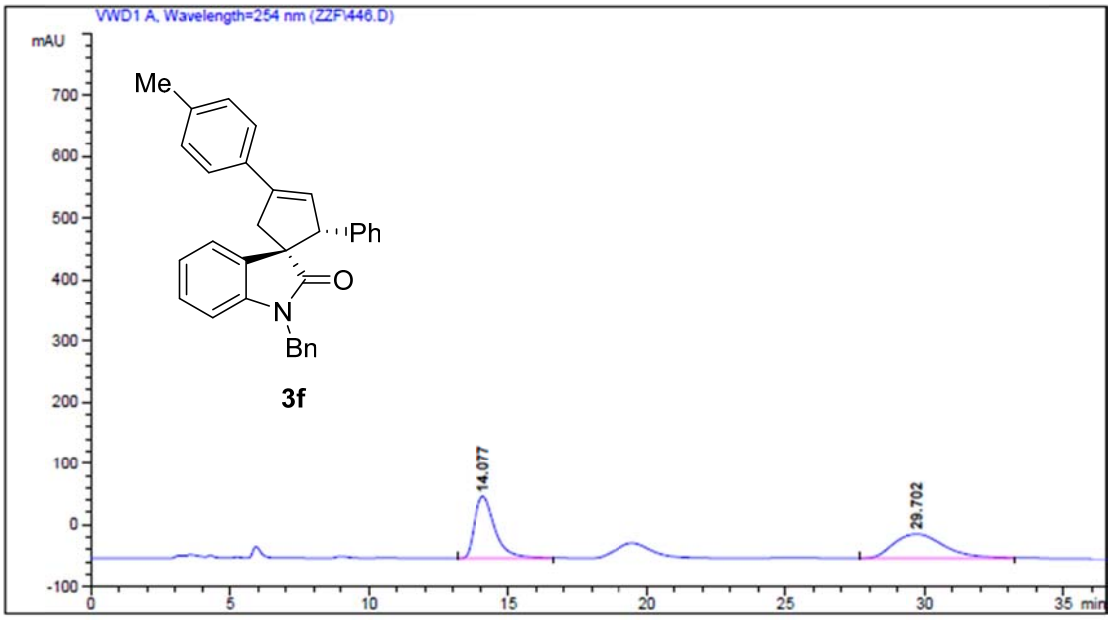


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.162	MM	0.2221	1274.24329	95.63833	47.1992
2	11.927	BB	0.3449	1425.47266	62.31913	52.8008
Totals :				2699.71594	157.95745	

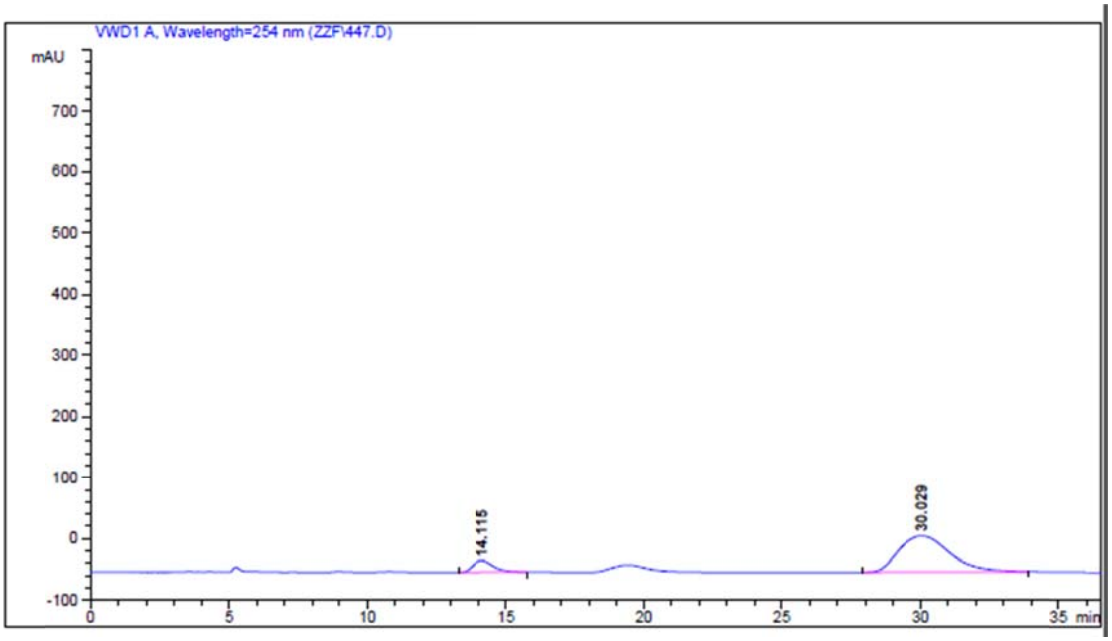
Additional Info : Peak(s) manually integrated



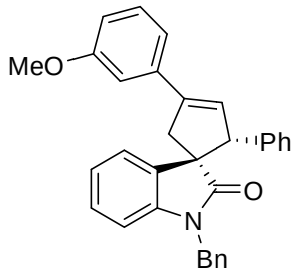
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.623	VB R	0.1996	5004.88965	374.08563	90.4411
2	12.447	BB	0.3264	528.97681	24.44916	9.5589
Totals :				5533.86646	398.53479	



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.077	BB	0.7955	5146.71826	100.01515	51.0659
2	29.702	BB	1.9813	4931.85889	38.94323	48.9341
Totals :				1.00786e4	138.95838	

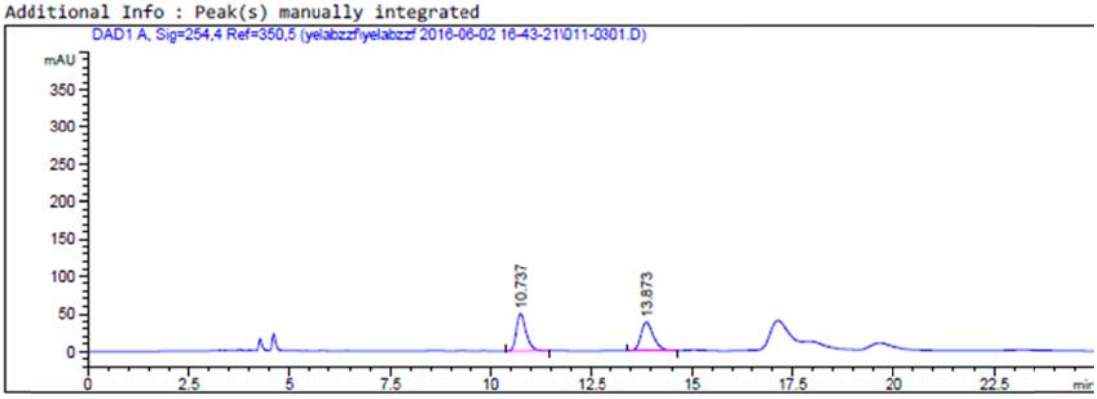


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.115	BB	0.7903	954.66626	18.71306	11.1696
2	30.029	BB	2.0633	7592.36328	58.77311	88.8304
Totals :				8547.02954	77.48616	



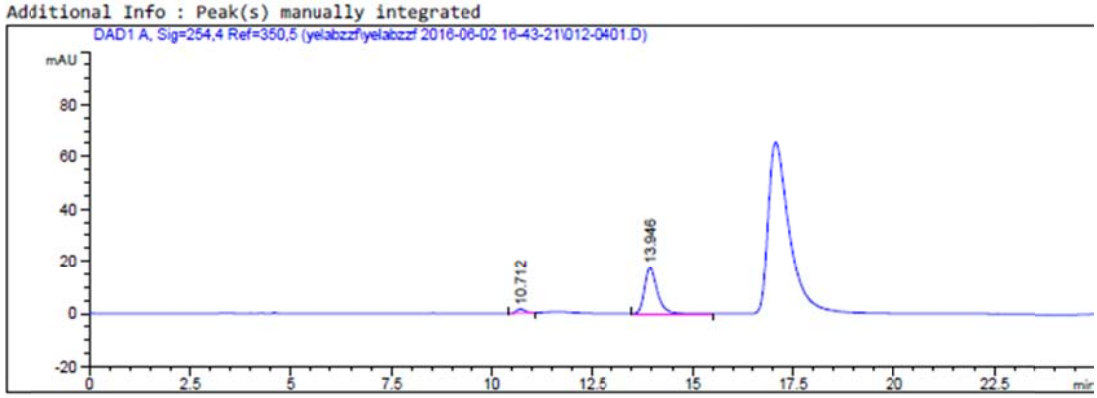
3g

Additional Info : Peak(s) manually integrated

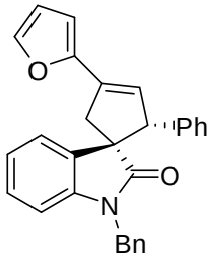


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.737	BB	0.2519	824.84314	49.63707	50.4256
2	13.873	BB	0.3263	810.91827	37.79843	49.5744

Additional Info : Peak(s) manually integrated

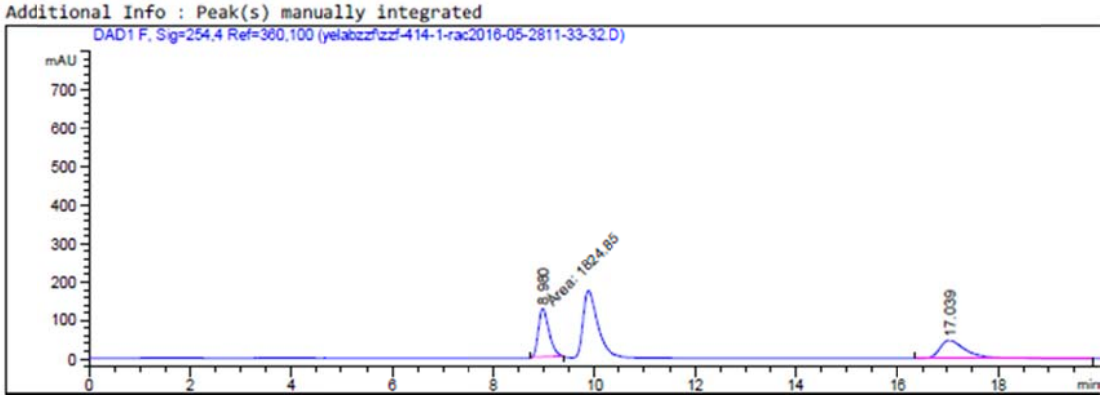


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.712	BB	0.2312	23.49962	1.54720	5.7904
2	13.946	BB	0.3317	382.33890	17.44171	94.2096
Totals :				405.83852	18.98891	



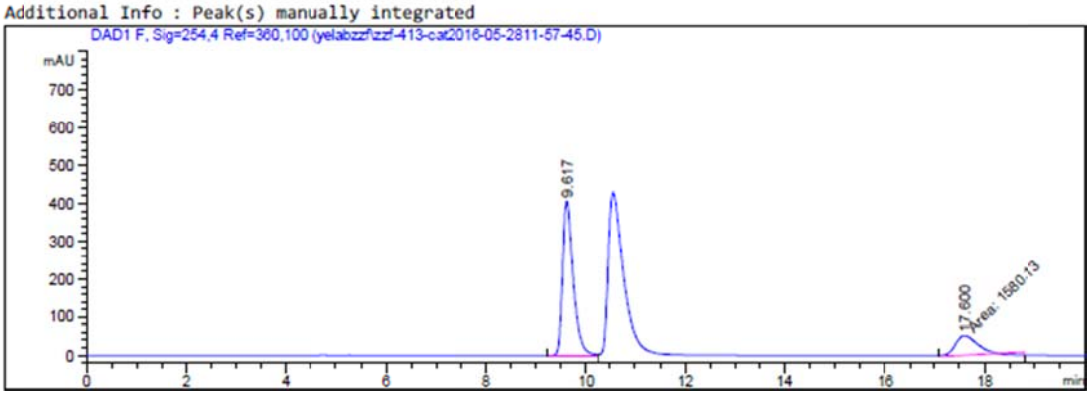
3h

Additional Info : Peak(s) manually integrated

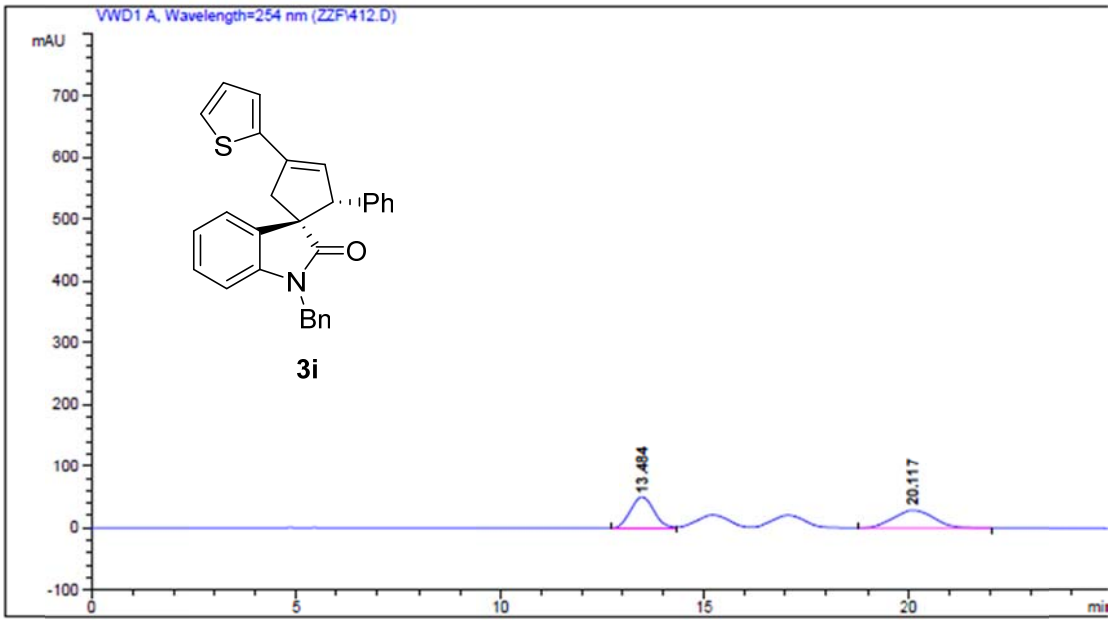


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.980	MM	0.2428	1824.85376	125.24522	52.9069
2	17.039	BB	0.5225	1624.32532	46.38968	47.0931
Totals :				3449.17908	171.63490	

Additional Info : Peak(s) manually integrated

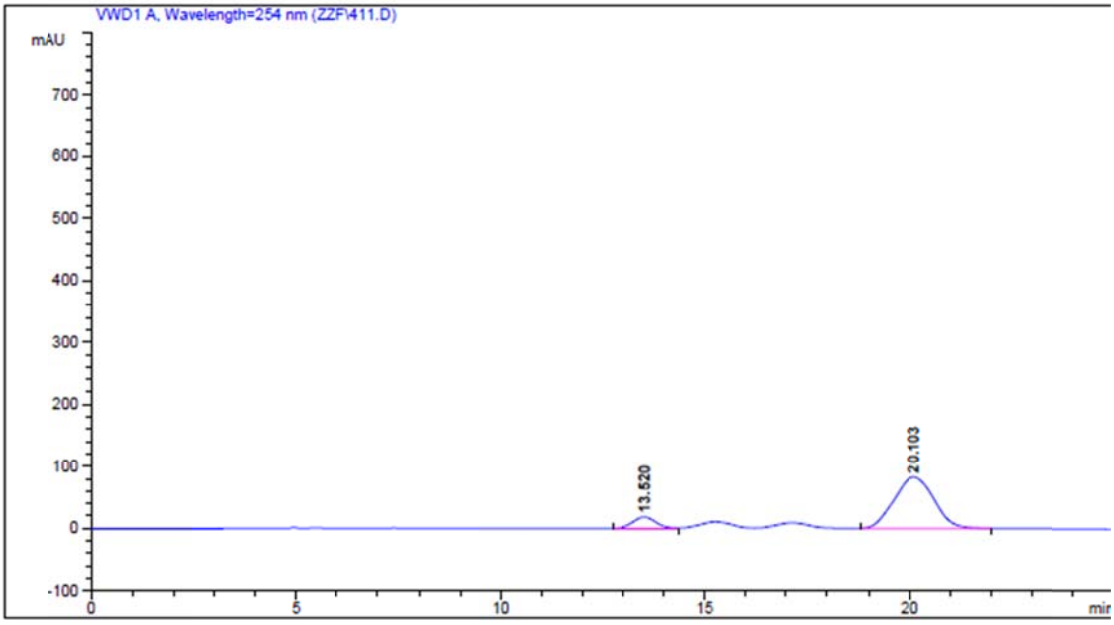


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.617	BV	0.2220	5983.06836	405.77206	79.1077
2	17.600	MM	0.5112	1580.12976	51.51407	20.8923
Totals :				7563.19812	457.28613	

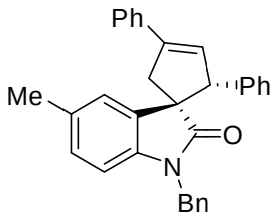


3i

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.484	BV	0.6194	1967.92139	49.67483	50.0761
2	20.117	BB	1.0951	1961.93628	28.37099	49.9239
Totals :				3929.85767	78.04582	

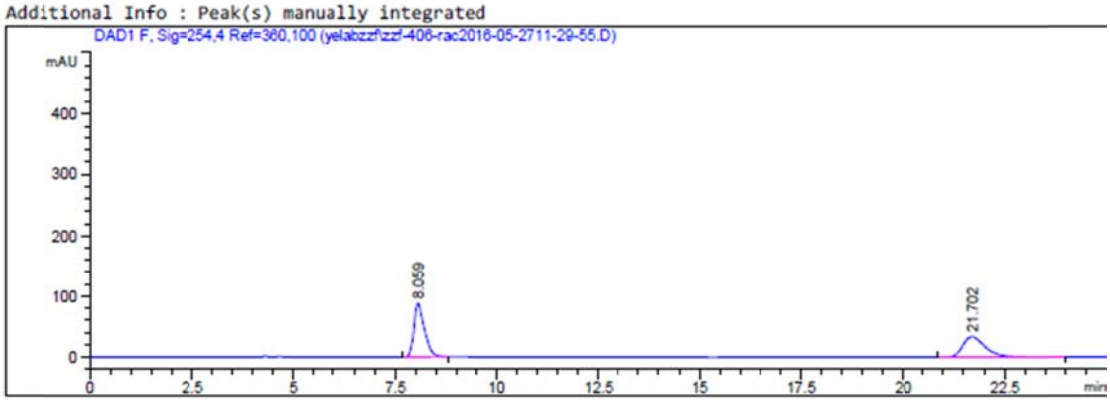


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.520	BV	0.6132	703.20068	18.10942	11.1705
2	20.103	BB	1.0555	5591.95557	82.53015	88.8295
Totals :				6295.15625	100.63957	



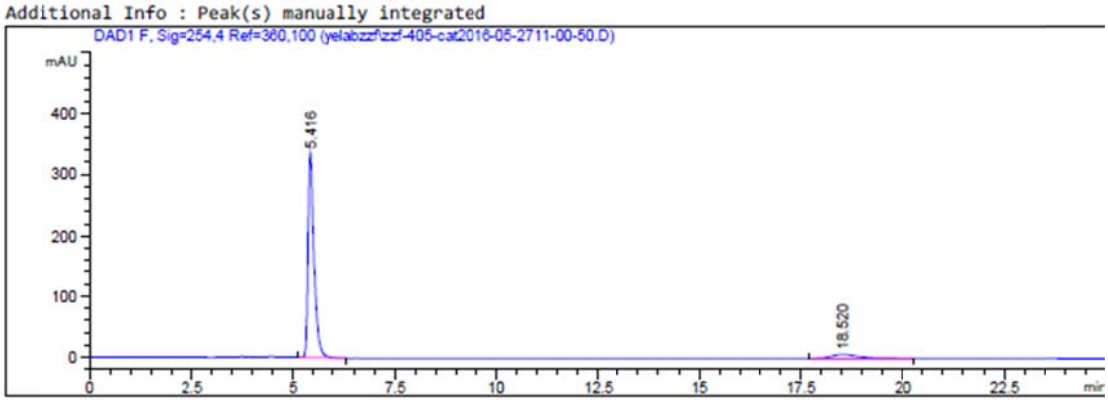
3j

Additional Info : Peak(s) manually integrated

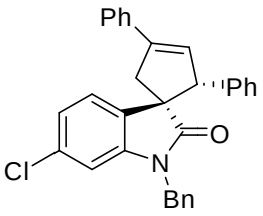


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.059	BB	0.2507	1548.21790	88.21850	54.9224
2	21.702	BB	0.5865	1270.69971	32.61982	45.0776

Additional Info : Peak(s) manually integrated

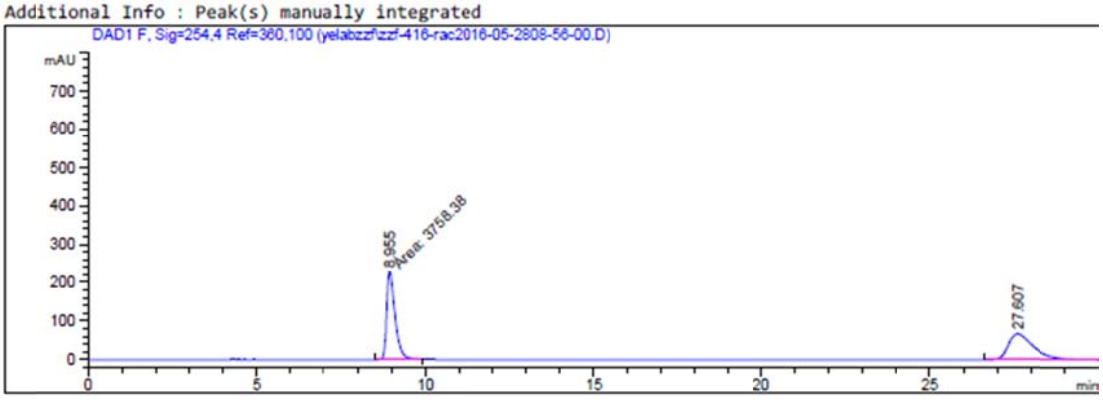


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.416	BB	0.1487	3425.68896	336.75458	92.1263
2	18.520	BB	0.6241	292.78308	6.77576	7.8737
Totals :				3718.47205	343.53033	



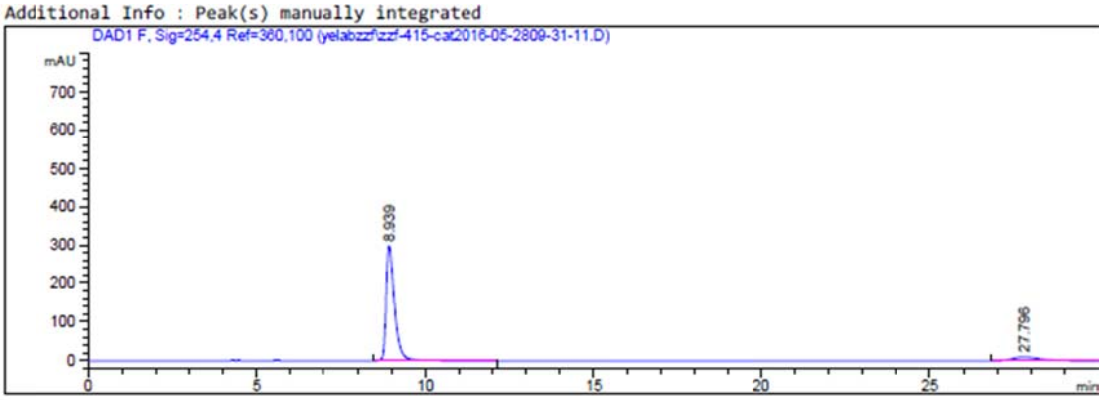
3k

Additional Info : Peak(s) manually integrated

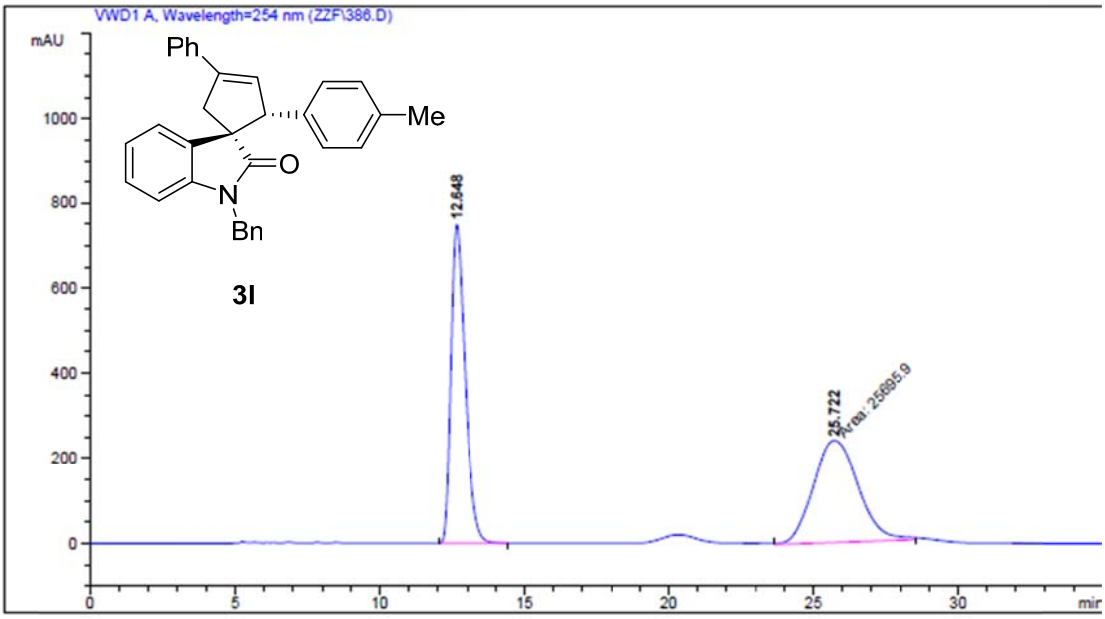


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.955	MM	0.2785	3758.38428	224.89830	51.6038
2	27.607	BB	0.7890	3524.76733	66.43863	48.3962
Totals :				7283.15161	291.33693	

Additional Info : Peak(s) manually integrated

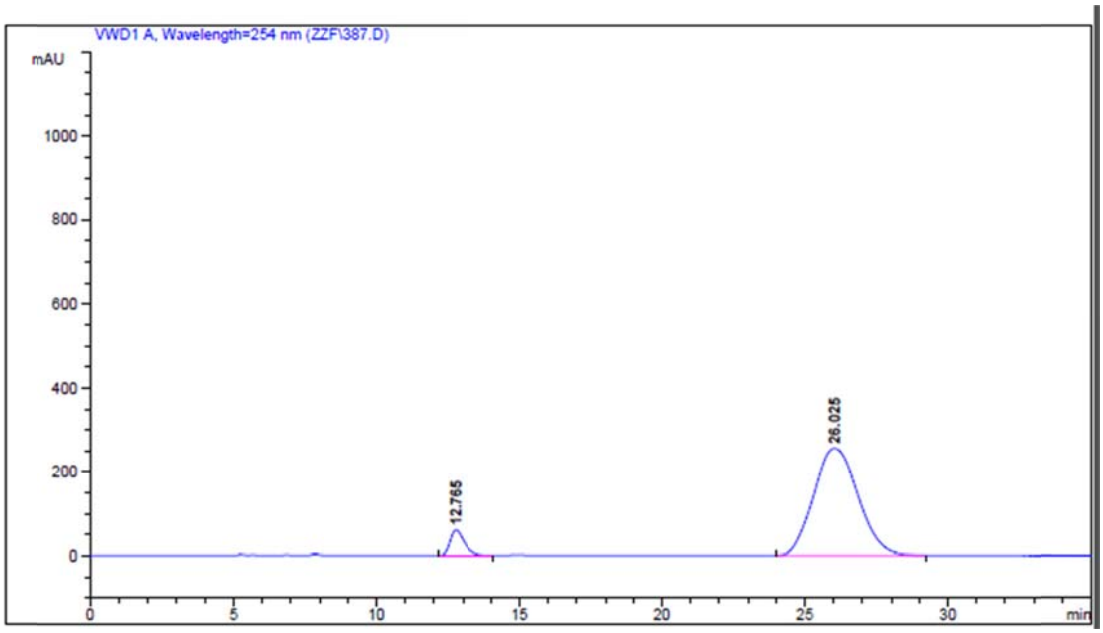


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.939	BB	0.2502	5068.80029	298.39084	90.6839
2	27.796	BB	0.7814	520.72736	9.74581	9.3161
Totals :				5589.52765	308.13665	

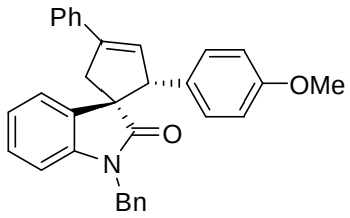


3l

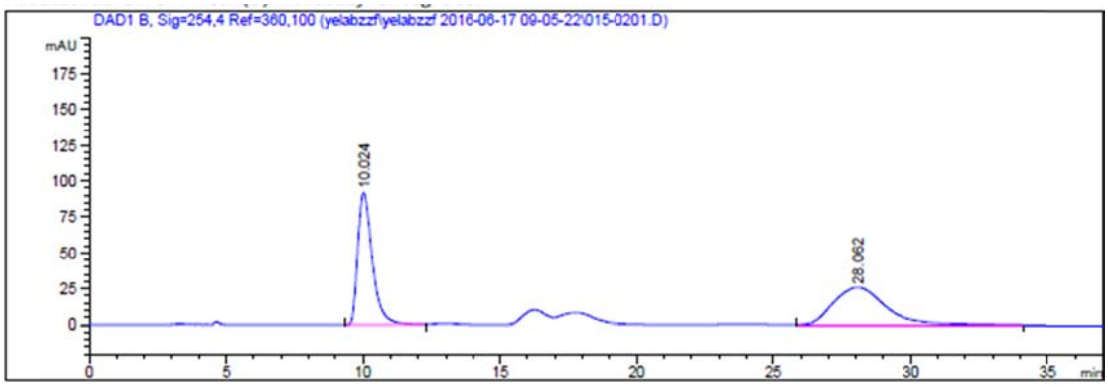
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.648	BB	0.5244	2.56217e4	748.81219	49.9277
2	25.722	MM	1.7851	2.56959e4	239.90872	50.0723



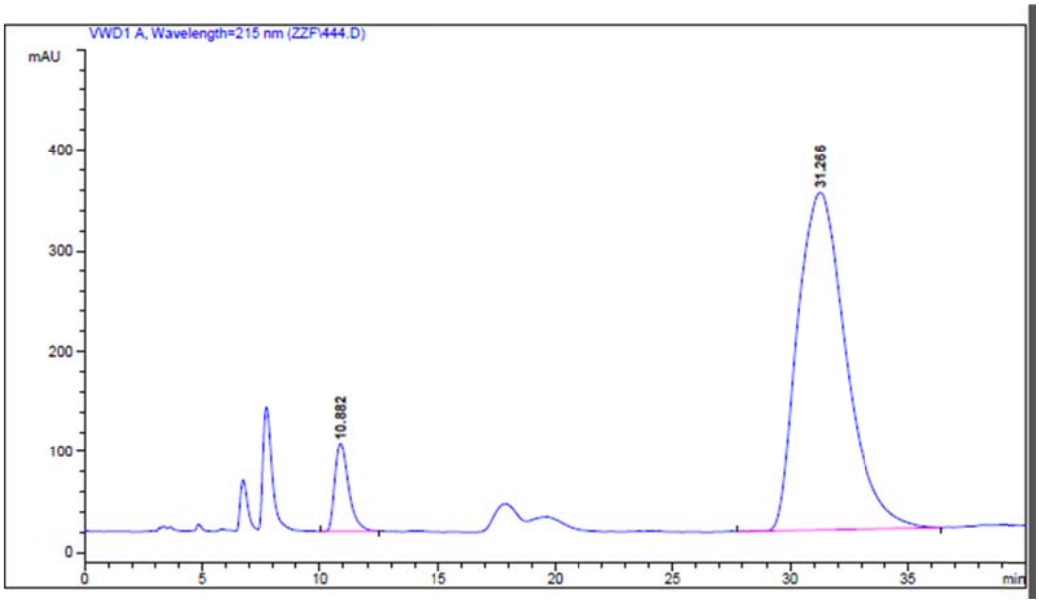
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.765	BB	0.5282	2106.38354	60.98278	7.1291
2	26.025	BB	1.6509	2.74400e4	255.71130	92.8709
Totals :				2.95464e4	316.69409	



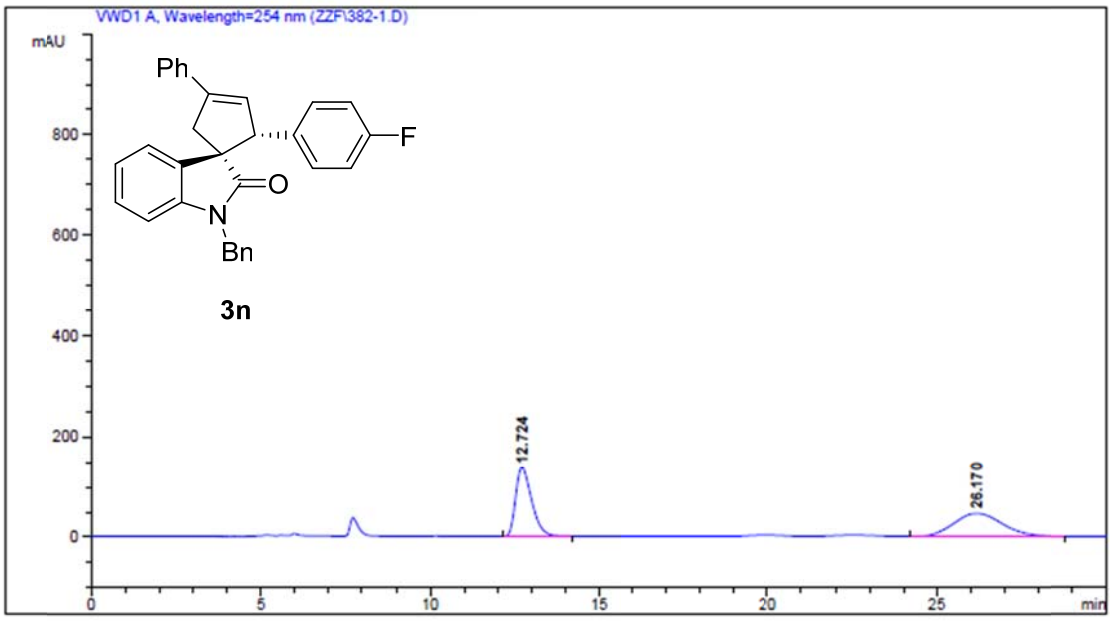
3m



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.024	BB	0.5647	3362.83521	91.52627	49.9575
2	28.062	BB	1.7421	3368.56006	25.81898	50.0425
Totals :				6731.39526	117.34525	

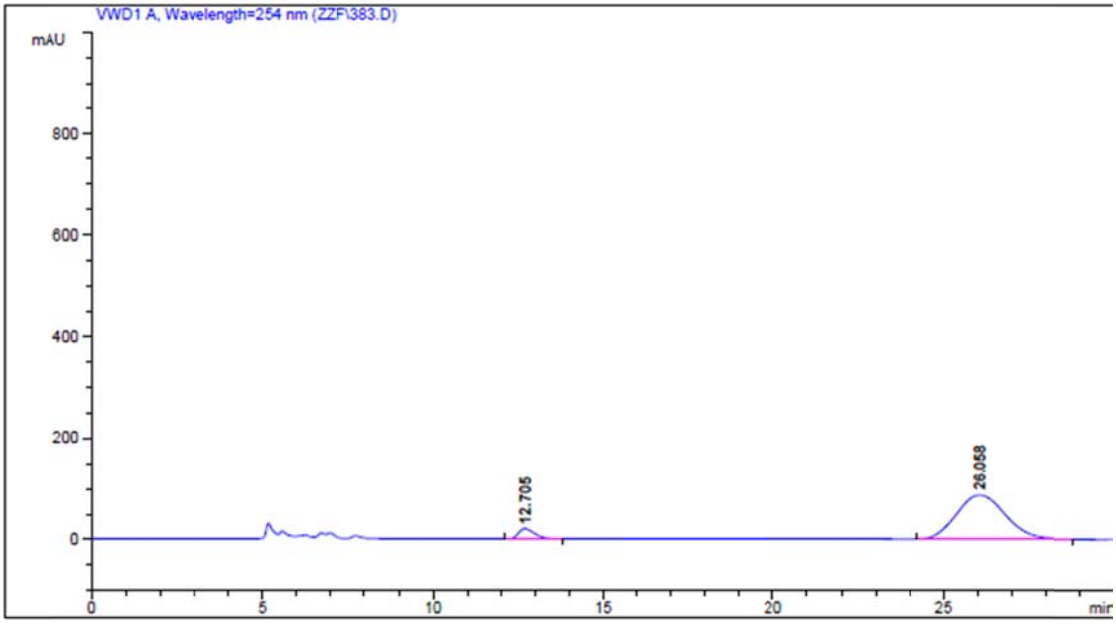


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.882	VB	0.6322	3559.90210	87.44145	6.9590
2	31.266	VB	2.2220	4.75955e4	335.10425	93.0410
Totals :				5.11554e4	422.54570	

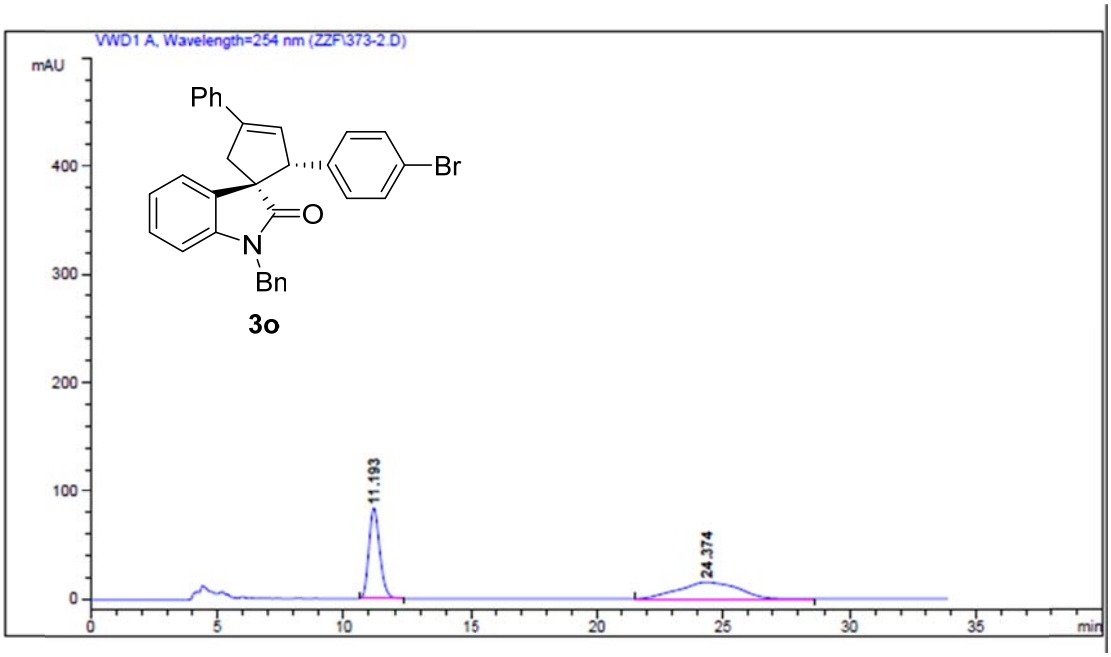


3n

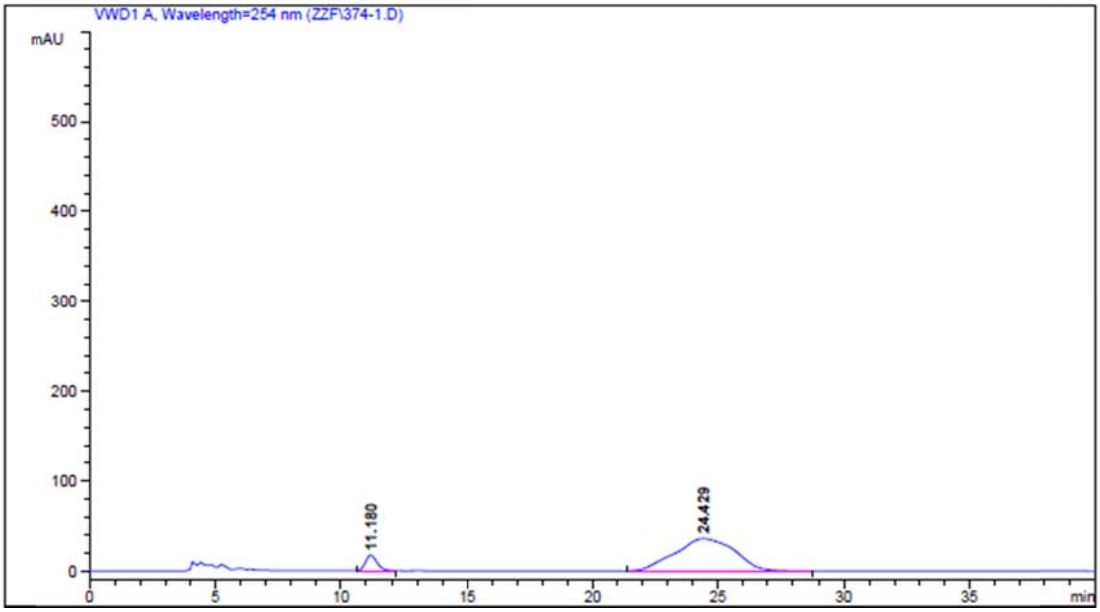
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.724	BB	0.5032	4517.72559	138.37834	49.8569
2	26.170	VB	1.5370	4543.65820	45.75814	50.1431



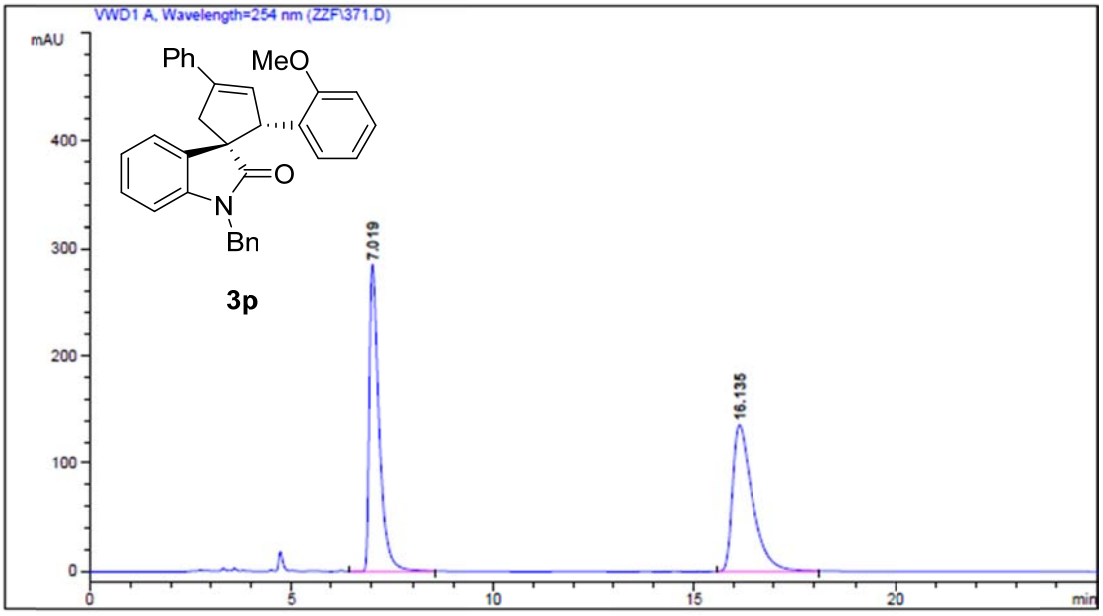
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.705	BB	0.5043	661.10522	20.18926	7.2056
2	26.058	BB	1.5337	8513.82324	85.77375	92.7944
Totals :				9174.92847	105.96301	



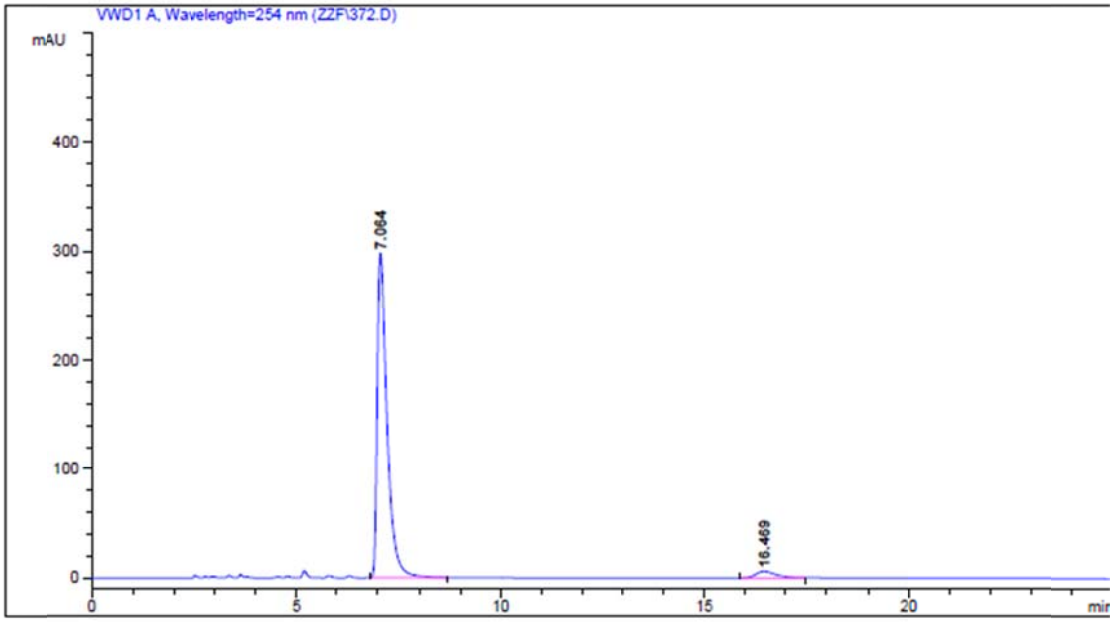
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.193	BB	0.4531	2473.27979	83.54762	49.7964
2	24.374	BB	2.1161	2493.50220	15.32984	50.2036
Totals :				4966.78198	98.87746	



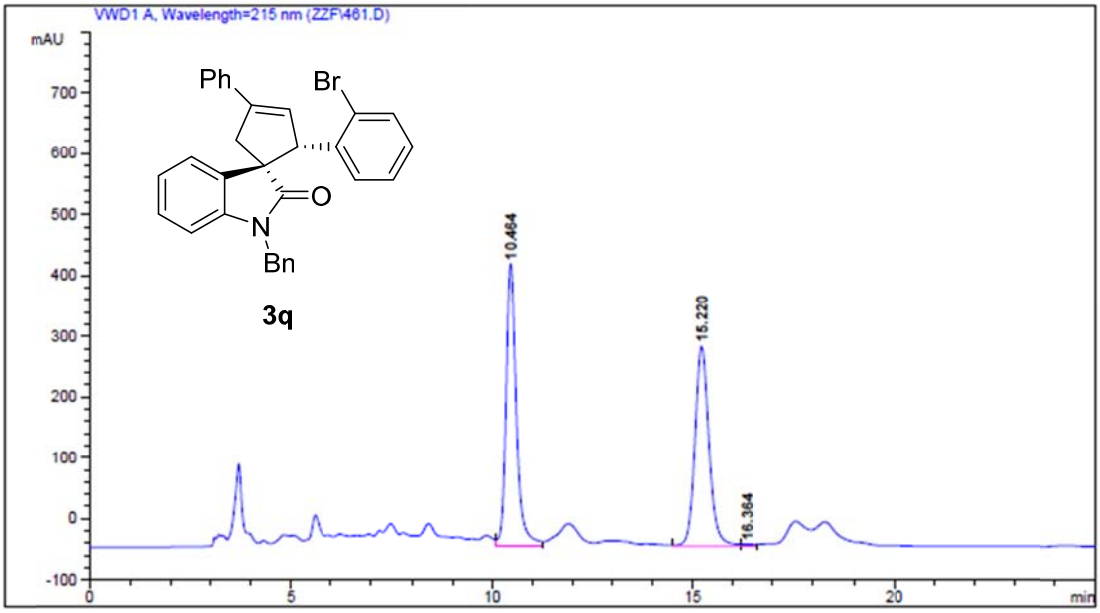
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.180	BB	0.4795	544.23975	17.35164	8.4572
2	24.429	BB	2.1904	5890.94873	35.91155	91.5428
Totals :				6435.18848	53.26319	



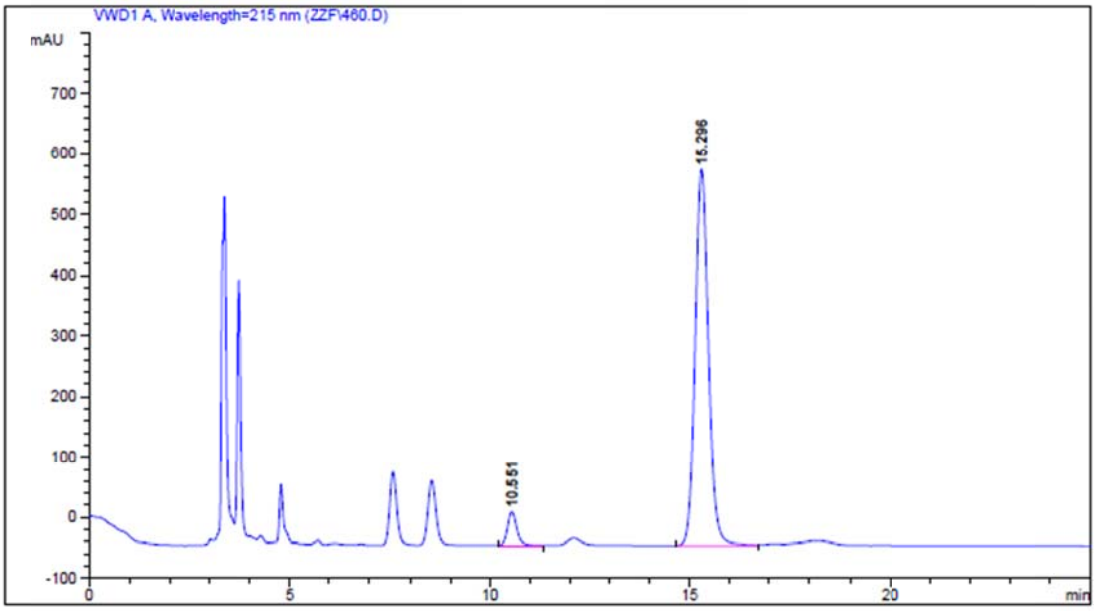
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.019	VB	0.2505	4765.27588	284.81226	50.2043
2	16.135	BB	0.5219	4726.49170	136.00298	49.7957
Totals :				9491.76758	420.81523	



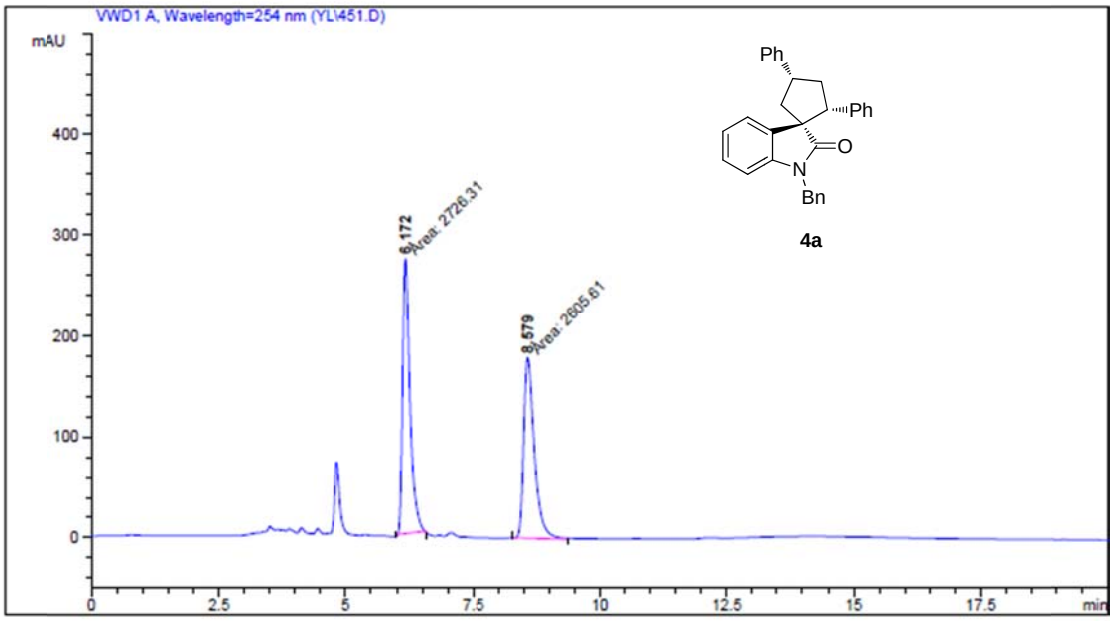
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.064	BB	0.2490	4945.31055	297.92294	95.9517
2	16.469	BB	0.5263	208.64653	6.02485	4.0483
Totals :				5153.95708	303.94779	



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.464	VV	0.2663	8293.77637	465.03253	50.6272
2	15.220	VV	0.3718	8013.20410	328.71484	48.9145
3	16.364	VV	0.3031	75.08259	3.45462	0.4583
Totals :				1.63821e4	797.20200	

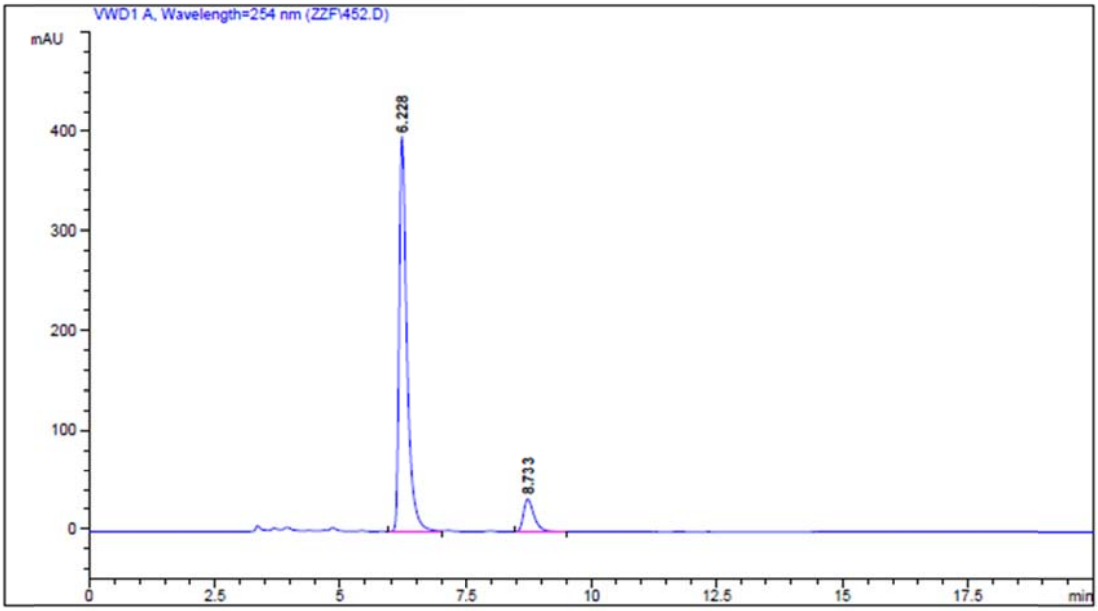


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.551	BB	0.2445	885.03351	55.42867	5.8072
2	15.296	BV	0.3571	1.43552e4	620.88031	94.1928
Totals :				1.52402e4	676.30898	



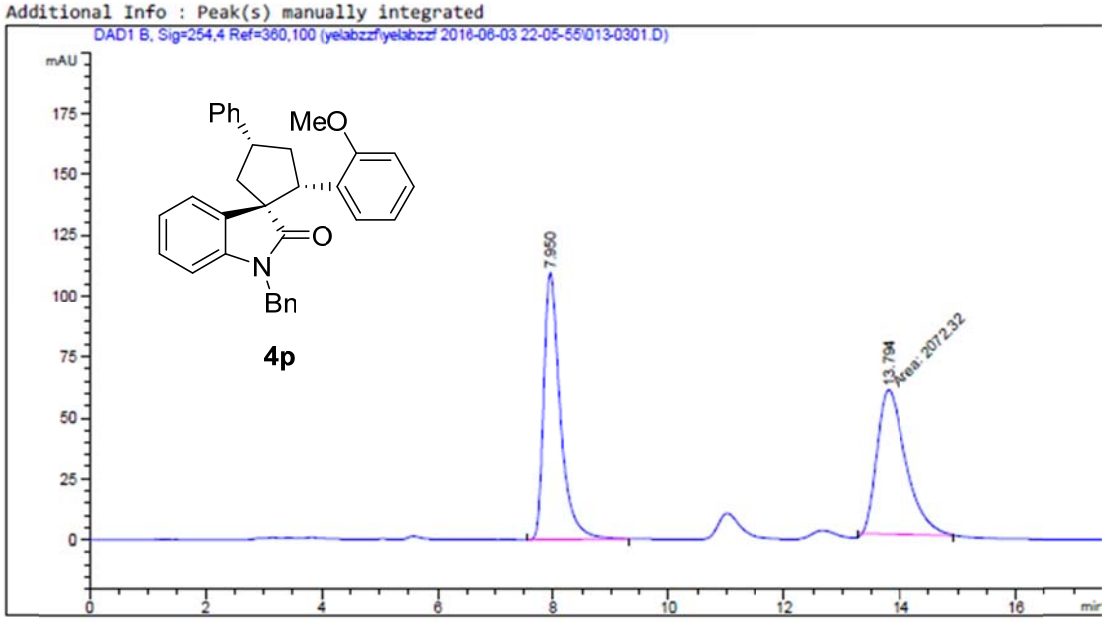
4a

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.172	MM	0.1667	2726.30859	272.63736	51.1319
2	8.579	MM	0.2430	2605.60962	178.68306	48.8681
Totals :				5331.91821	451.32042	



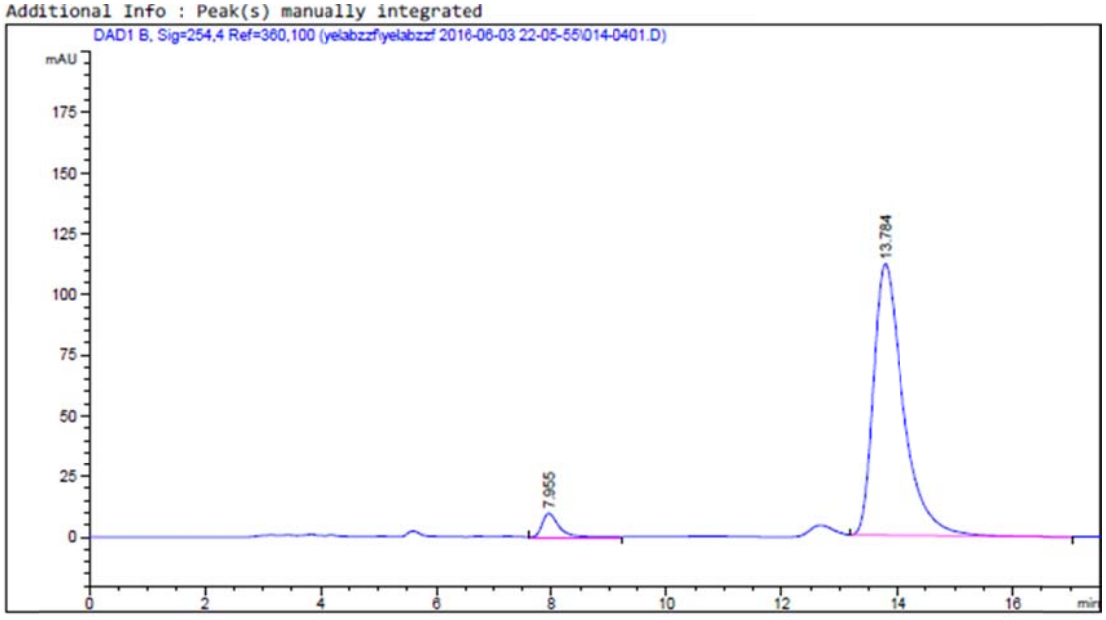
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.228	VV	0.1599	4256.80322	397.31143	89.7798
2	8.733	BB	0.2206	484.57822	33.03501	10.2202
Totals :				4741.38144	430.34644	

Additional Info : Peak(s) manually integrated



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.950	BB	0.2979	2152.47363	109.23408	50.9486
2	13.794	MM	0.5830	2072.31812	59.24289	49.0514

Additional Info : Peak(s) manually integrated



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.955	BB	0.2977	190.48271	9.67557	4.5229
2	13.784	BBA	0.5436	4021.04102	111.79024	95.4771
Totals :				4211.52373	121.46580	