

Supporting Information

Highly Diastereo and Enantioselective NHC-Catalyzed [3+2] Annulation of Enals and Isatins

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Table of Contents

Part I Experimental part	2
General Information.....	2
1.1 Enantioselective [3 + 2] Annulation of Enals and Isatins catalyzed by NHC ..	3
1.2 X-ray crystal structure of (-)- 3d	11
References.....	12
Part II NMR Spectra	13
Part III HPLC Spectra	26

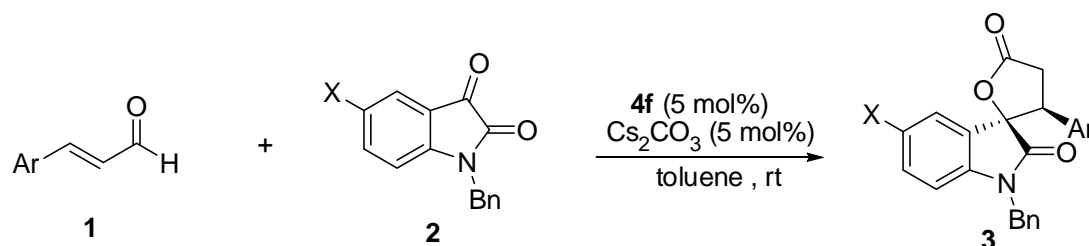
Part I Experimental part

General Information

Unless otherwise indicated, all reactions were carried out under an N₂ atmosphere in oven-dried glassware with magnetic stirring. Anhydrous THF and toluene were distilled from sodium and benzophenone, CH₂Cl₂ was distilled from CaH₂. Chiral triazolium salts **4a-4g**,^{1a-1d} were synthesized according to our previous reports and the literatures. Isatins and aldehydes were synthesized according to literatures^{2,3}. Column chromatograph was performed on silica gel 200~300 mesh. All ¹H NMR (300 MHz), ¹³C NMR (75 MHz) spectra were recorded on a Bruker-DMX 300 spectrometer in CDCl₃, 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). 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: [α]_D^T (concentration (g/100 mL), solvent).

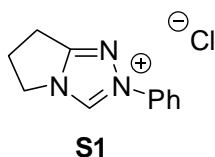
1.1 Enantioselective [3 + 2] Annulation of Enals and Isatins catalyzed by NHC

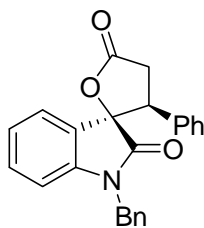
4f



Typical procedure. To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with trazolium salt **4f** (10.8 mg, 0.015 mmol), anhydrous Cs₂CO₃ (4.9mg, 0.015 mmol). This tube was closed with a septum, evacuated, and back-filled with nitrogen. To this mixture was added freshly distilled toluene (3 mL) and stirred for 30 minutes at room temperature. Then isatin **2a** (70.8 mg, 0.3 mmol) and aldehyde **1a** (56.6 ul, 1.5 mmol) were added. After stirring for 12 h, the reaction mixture was diluted with diethyl ether and passed through a short silica pad. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (ethyl acetate/petroleum ether, typically 1/5) to give the desired product.

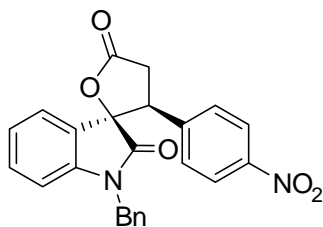
Racemic samples for the standard of chiral HPLC spectra were prepared using 10 mol% of achiral triazolium salt **S1** as the precatalyst.





3a

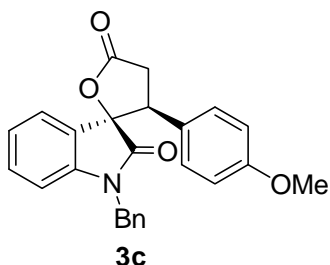
(2R,3S)-1'-benzoyl-3-phenyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 111.5 mg (97%), white solid, mp 145-147°C, $R_f = 0.48$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -108.5$ (c 1.0, CH_2Cl_2), HPLC analysis: 98% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 90:10, 1.0 mL/min, 254 nm, 44.4 min (major), 61.7 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.56 (*d*, $J = 7.0$ Hz, 1H), 7.33-7.28 (*m*, 1H), 7.24-7.12 (*m*, 5H), 7.09-6.99 (*m*, 4H), 6.44-6.40 (*m*, 3H), 4.98 (*d*, $J = 16.0$ Hz, 1H), 4.18 (*d*, $J = 16.1$ Hz, 1H), 4.19-4.14 (*m*, 1H), 3.95-3.85 (*m*, 1H), 2.94 (*dd*, $J = 7.9$ Hz, $J = 16.7$ Hz, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.8, 172.7, 143.6, 134.3, 132.0, 131.3, 128.8, 128.6, 128.4, 128.0, 127.3, 126.3, 124.6, 124.3, 123.5, 109.9, 86.3, 50.7, 43.6, 32.2. IR (KBr) ν 1793, 1731, 1616, 1373, 697. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{15}\text{NO}_3$, 369.1365, Found 369.1370.



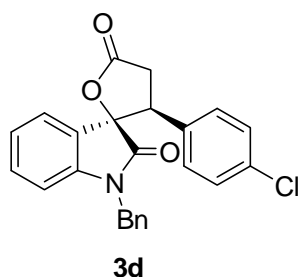
3b

(2R,3S)-1'-benzyl-3-(4-nitrophenyl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 106.6 mg (86%), white solid, mp 91-93°C, $R_f = 0.29$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -132.0$ (c 1.0, CH_2Cl_2), HPLC analysis: 93% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 39.0 min (major), 45.0 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.94 (*d*, $J = 8.7$ Hz, 2H), 7.59 (*d*, $J = 7.3$ Hz, 1H), 7.36-7.21 (*m*, 2H), 7.16-7.01 (*m*, 5H), 6.65-6.60 (*m*, 3H), 4.87 (*d*, $J = 15.6$ Hz, 1H), 4.26 (*d*, $J = 15.6$ Hz, 1H), 4.25-4.21 (*m*, 1H), 3.88 (*dd*, $J = 13.6$ Hz, $J = 16.7$ Hz, 1H), 3.02 (*dd*, $J = 8.0$ Hz, $J = 16.7$ Hz, 1H).

^{13}C NMR (300 MHz, CDCl_3) δ 173.7, 172.2, 147.9, 143.6, 139.5, 134.4, 131.8, 128.9, 128.5, 127.9, 126.8, 124.4, 123.9, 123.7, 109.9, 85.7, 50.3, 43.8, 32.1. IR (KBr) ν 1802, 1725, 1260, 1082, 800. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{18}\text{N}_2\text{O}_5$, 414.1216, Found 414.1222.

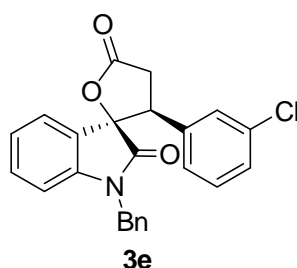


(2R,3S)-1'-benzyl-3-(4-methoxyphenyl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 114.1 mg (95%), white solid, mp 192-194°C, $R_f = 0.37$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -151.8$ (c 1.0, CH_2Cl_2), HPLC analysis: 85% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 70:30, 1.0 mL/min, 254 nm, 20.8 min (minor), 24.7 min (major)]. ^1H NMR (300 MHz, CDCl_3) δ 7.56-7.53 (*m*, 1H), 7.26-7.06 (*m*, 5H), 6.92 (*d*, $J = 8.5$ Hz, 2H), 6.75-6.71 (*m*, 2H), 6.46-6.44 (*m*, 3H), 5.05 (*d*, $J = 16.0$ Hz, 1H), 4.20 (*d*, $J = 16.1$ Hz, 1H), 4.12 (*dd*, $J = 8.1$ Hz, $J = 14.1$ Hz, 1H), 3.86 (*dd*, $J = 14.1$ Hz, $J = 16.5$ Hz, 1H), 3.77 (*s*, 3H), 2.93 (*dd*, $J = 7.8$ Hz, $J = 16.7$ Hz, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.9, 172.9, 159.7, 143.7, 134.4, 131.3, 129.2, 128.5, 127.4, 126.4, 124.8, 124.3, 123.8, 123.5, 114.1, 109.9, 86.5, 55.2, 50.2, 43.7, 32.4; IR (KBr) ν 1799, 1724, 1616, 1182, 490. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{25}\text{H}_{21}\text{NO}_4$, 399.1471, Found 399.1476.

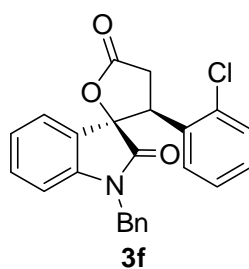


(2R,3S)-1'-benzyl-3-(4-chlorophenyl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 97.2 mg (80%), white solid, mp 204-206°C, $R_f = 0.47$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -160.6$ (c 1.0, CH_2Cl_2), HPLC analysis: 95% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0

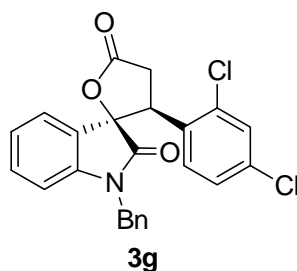
mL/min, 254 nm, 18.0 min (major), 24.4 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.57-7.54 (*m*, 1H), 7.29-7.26 (*m*, 1H), 7.24-7.15 (*m*, 6H), 6.93 (*d*, $J = 8.5\text{ Hz}$, 2H), 6.51-6.47 (*m*, 3H), 5.03 (*d*, $J = 15.9\text{ Hz}$, 1H), 4.21 (*d*, $J = 15.9\text{ Hz}$, 1H), 4.13 (*dd*, $J = 8.1\text{ Hz}$, $J = 13.9\text{ Hz}$, 1H), 3.85 (*dd*, $J = 13.8\text{ Hz}$, $J = 16.7\text{ Hz}$, 1H), 2.95 (*dd*, $J = 8.0\text{ Hz}$, $J = 16.7\text{ Hz}$, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.4, 172.6, 143.7, 134.5, 134.3, 131.5, 130.7, 129.4, 129.0, 128.7, 127.7, 126.34, 124.4, 124.3, 123.7, 110.0, 86.1, 50.2, 43.8, 32.2. IR (KBr) ν 1801, 1725, 1617, 1493, 1468, 697. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{18}\text{NO}_3\text{Cl}$, 403.0975, Found 403.0981.



(2R,3S)-1'-benzyl-3-(3-chlorophenyl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione
e. Yield: 113.8 mg (94%), white solid, mp 73-75°C, $R_f = 0.47$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -106.1$ (c 1.0, CH_2Cl_2), HPLC analysis: 89% ee [Daicel CHIRALPAK AS-H column, 20 °C, 254 nm, hexane/*i*-PrOH = 70:30, 1.0 mL/min, 254 nm, 23.1 min (minor), 33.6 min (major)]. ^1H NMR (300 MHz, CDCl_3) δ 7.56-7.54 (*m*, 1H), 7.25-7.05 (*m*, 7H), 6.98-5.84 (*m*, 2H), 6.56-6.50 (*m*, 3H), 4.96 (*d*, $J = 15.7\text{ Hz}$, 1H), 4.22 (*d*, $J = 15.8\text{ Hz}$, 1H), 4.15-4.10 (*m*, 1H), 3.87-3.78 (*m*, 1H), 2.96-2.92 (*m*, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.2, 172.4, 143.5, 134.6, 134.3, 134.2, 131.5, 130.0, 128.7, 128.6, 128.0, 127.5, 126.4, 126.1, 124.3, 124.2, 123.7, 109.9, 85.9, 50.1, 43.7, 32.1. IR (KBr) ν 1798, 1724, 1616, 1180, 688. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{18}\text{NO}_3\text{Cl}$, 403.0975, Found 403.0980.

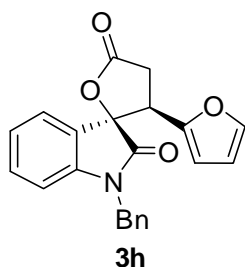


(2R,3S)-1'-benzyl-3-(2-chlorophenyl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 115.3 mg (95%), white solid, mp 93-95°C, $R_f = 0.51$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -72.6$ (c 1.0, CH_2Cl_2), HPLC analysis: 87% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 18.1 min (major), 21.5 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.70-7.69 (*m*, 1H), 7.59 (*d*, $J = 7.4$ Hz, 1H), 7.25-7.06 (*m*, 8H), 6.55 (*d*, $J = 7.3$ Hz, 2H), 6.44 (*d*, $J = 7.7$ Hz, 1H), 5.03 (*d*, $J = 15.9$ Hz, 1H), 4.90 (*dd*, $J = 8.4$ Hz, $J = 13.5$ Hz, 1H), 4.28 (*d*, $J = 16.0$ Hz, 1H), 3.74 (*dd*, $J = 13.5$ Hz, $J = 17.0$ Hz, 1H), 3.01 (*dd*, $J = 8.4$ Hz, $J = 17.0$ Hz, 1H); ^{13}C NMR (300 MHz, CDCl_3) δ 174.3, 173.1, 143.3, 135.1, 134.5, 131.3, 130.2, 130.0, 129.6, 129.5, 128.7, 127.5, 127.4, 126.4, 125.9, 123.9, 123.3, 109.7, 86.4, 45.3, 43.6, 34.2. IR (KBr) ν 1798, 1724, 1617, 1182, 751. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{18}\text{NO}_3\text{Cl}$, 403.0975, Found 403.0981,



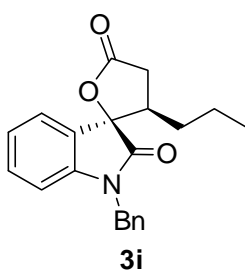
(2R,3S)-1'-benzyl-3-(2,4-dichlorophenyl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 113.2mg (86%), white solid, mp 162-165°C, $R_f = 0.57$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -120.7$ (c 1.0, CH_2Cl_2), HPLC analysis: 93% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 85:15, 1.0 mL/min, 254 nm, 21.5 min (major), 24.3 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ : 7.61-7.57 (*m*, 2H), 7.25-7.08 (*m*, 7H), 6.60 (*d*, $J = 6.9$ Hz, 2H), 6.51 (*d*, $J = 7.8$ Hz, 1H), 5.04 (*d*, $J = 15.9$ Hz, 1H), 4.83 (*dd*, $J = 8.4$ Hz, $J = 13.5$ Hz, 1H), 4.29(*d*, $J = 15.9$ Hz, 1H), 3.68(*dd*, $J = 13.6$ Hz, $J = 17.0$ Hz, 1H), 3.00 (*dd*, $J = 8.5$ Hz, $J = 17.0$ Hz, 1H); ^{13}C NMR (300 MHz, CDCl_3) δ 174.0, 172.8, 143.2, 135.8, 134.8, 134.4, 131.4, 130.5, 129.7, 128.8, 128.7, 127.7, 126.4, 125.9, 123.6, 123.4, 109.7, 86.1, 44.8, 43.7, 34.1; IR (KBr) ν 1803, 1724, 1479, 1182, 669. HRMS (EI) m/z : M^+ Calc. for

C₂₄H₁₇NO₃Cl₂, 437.0585, Found 437.0590.



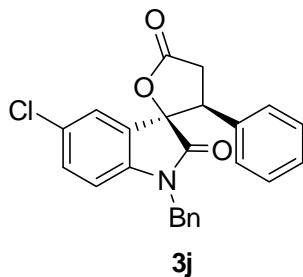
(2R,3S)-1'-benzyl-3-(furan-2-yl)-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione.

Yield: 104.5 mg (97%), white solid, mp 175-179 °C, $R_f = 0.43$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -79.1$ (c 1.0, CH₂Cl₂), HPLC analysis: 86% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 27.6 min (major), 42.4 min (minor)]. ¹H NMR (300 MHz, CDCl₃) δ 7.48 (*d*, $J = 7.2$ Hz, 1H), 7.29-7.11 (*m*, 6H), 6.89-6.87 (*m*, 2H), 6.61 (*d*, $J = 7.8$ Hz, 1H), 6.27-6.26 (*m*, 1H), 6.11-6.10 (*m*, 1H), 5.02 (*d*, $J = 15.8$ Hz, 1H), 4.40 (*d*, $J = 15.9$ Hz, 1H), 4.21 (*dd*, $J = 8.2$ Hz, $J = 13.2$ Hz, 1H), 3.78 (*dd*, $J = 13.6$ Hz, $J = 16.8$ Hz, 1H), 3.01 (*dd*, $J = 8.1$ Hz, $J = 16.9$ Hz, 1H). ¹³C NMR (300 MHz, CDCl₃) δ 174.1, 172.6, 147.5, 143.7, 142.7, 134.6, 131.4, 128.8, 127.6, 127.0, 124.7, 124.4, 123.6, 110.6, 109.9, 108.5, 84.7, 44.5, 43.8, 32.3; IR (KBr) ν 1799, 1726, 1617, 1187, 750. HRMS (EI) m/z : M⁺ Calc. for C₂₂H₁₇NO₄, 359.1158, Found 359,1163.

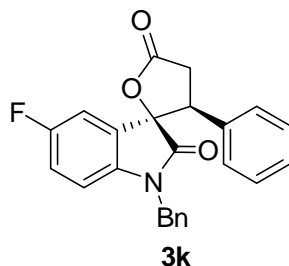


(2R,3R)-1'-benzyl-3-propyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 38.6 mg (38%), white solid, mp 106-108 °C, $R_f = 0.50$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} 25.7$ (c 1.0, CH₂Cl₂), HPLC analysis: 97% ee [Daicel CHIRALPAK IA-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 13.6 min (minor), 15.8 min (major)]. ¹H NMR (300 MHz, CDCl₃) δ 7.37-7.25 (*m*, 7H), 7.12-7.07 (*m*, 1H), 6.75 (*d*, $J = 7.8$ Hz, 1H), 5.01 (*d*, $J = 15.6$ Hz, 1H), 4.73 (*d*, $J =$

15.6 Hz, 1H), 3.09-2.94 (*m*, 1H), 2.89-2.72 (*m*, 2H), 1.51-1.42 (*m*, 1H), 1.29-1.17 (*m*, 2H), 1.13-1.04 (*m*, 1H), 0.78 (*t*, $J = 14.0$ Hz, 3H). ^{13}C NMR (300 MHz, CDCl_3) δ 175.5, 173.4, 143.6, 135.1, 131.1, 128.9, 127.9, 127.4, 125.4, 124.3, 123.5, 109.7, 85.5, 45.6, 43.9, 33.8, 30.8, 21.3, 13.8. IR (KBr) ν 1796, 1725, 1613, 1181, 754. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{21}\text{H}_{21}\text{NO}_3$, 335.1521, Found 335.1525.

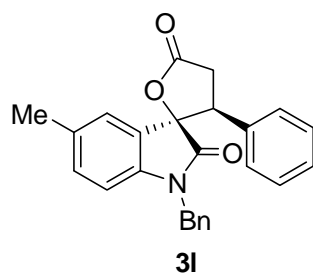


(2R,3S)-1'-benzyl-5'-chloro-3-phenyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 103.2 mg (85%), white solid, mp 221-223°C, $R_f = 0.56$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -140.4$ (c 1.0, CH_2Cl_2), HPLC analysis: 91% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 17.5 min (major), 29.8 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.56 (*s*, 1H), 7.36-7.31 (*m*, 1H), 7.25-7.03 (*m*, 8H), 6.42-6.34 (*m*, 3H), 4.98 (*d*, $J = 16.0$ Hz, 1H), 4.21-4.13 (*m*, 2H), 3.91 (*dd*, $J = 13.8$ Hz, $J = 16.7$ Hz, 1H), 2.94 (*dd*, $J = 7.9$ Hz, $J = 16.7$ Hz, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.3, 172.4, 142.1, 133.9, 131.7, 131.3, 129.0, 128.9, 128.8, 128.7, 128.1, 127.5, 126.4, 126.3, 124.8, 111.0, 86.0, 50.8, 43.8, 32.1; IR (KBr) ν 1805, 1726, 1612, 1178, 698. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{18}\text{NO}_3\text{Cl}$, 403.0975, Found 409.0980.

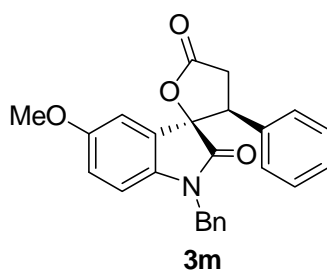


(2R,3S)-1'-benzyl-5'-fluoro-3-phenyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 107.4 mg (93%), white solid, mp 147-149°C, $R_f = 0.53$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -96.5$ (c 1.0, CH_2Cl_2), HPLC analysis: 80% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min,

254 nm, 19.5 min (major), 26.4 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.36-7.31 (*m*, 2H), 7.25-7.21 (*m*, 2H), 7.18-7.03 (*m*, 5H), 6.97-6.90 (*m*, 1H), 6.42-6.34 (*m*, 3H), 4.99 (*d*, $J=16.0$ Hz, 1H), 4.18 (*d*, $J=16.0$ Hz, 1H), 4.13 (*dd*, $J=7.7$ Hz, $J=13.6$ Hz, 1H), 3.90 (*dd*, $J=13.8$ Hz, $J=16.7$ Hz, 1H), 2.96 (*dd*, $J=7.9$ Hz, $J=16.7$ Hz, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.4, 172.6, 159.5 (*d*, $J=242.0$ Hz), 139.6, 143.0, 131.7, 128.9, 128.6, 128.0, 127.5, 126.3, 126.2, 117.8 (*d*, $J=23.4$ Hz), 112.4 (*d*, $J=25.0$ Hz), 110.8 (*d*, $J=7.8$ Hz), 86.2, 51.0, 43.8, 32.1. IR (KBr) ν 1804, 1726, 1585, 1493, 1175, 697. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{24}\text{H}_{18}\text{NO}_3\text{F}$, 387.1271, Found 387.1276.



(2R,3S)-1'-benzyl-5'-methyl-3-phenyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-dione. Yield: 105.1 mg (91%), white solid, mp 179-181°C, $R_f = 0.49$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -149.8$ (c 1.0, CH_2Cl_2), HPLC analysis: 86% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 80:20, 1.0 mL/min, 254 nm, 14.5 min (major), 26.4 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.38 (*s*, 1H), 7.33-7.28 (*m*, 1H), 7.24-7.11 (*m*, 3H), 7.08-6.99 (*m*, 5H), 6.42-6.39 (*m*, 2H), 6.31 (*d*, $J=8.0$ Hz, 1H), 4.95 (*d*, $J=16.0$ Hz, 1H), 4.16 (*d*, $J=16.0$ Hz, 1H), 4.20-4.16 (*m*, 1H), 3.89 (*dd*, $J=13.8$ Hz, $J=16.7$ Hz, 1H), 2.93 (*dd*, $J=8.0$ Hz, $J=16.7$ Hz, 1H), 2.35(*s*, 3H), ^{13}C NMR (300MHz, CDCl_3) δ 174.8, 172.7, 141.2, 134.4, 133.2, 132.1, 131.6, 128.7, 128.6, 128.3, 128.0, 127.2, 126.3, 124.9, 124.6, 109.6, 86.5, 50.6, 43.6, 32.2, 21.1. IR (KBr) ν 1796, 1724, 1603, 1172, 697. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{25}\text{H}_{21}\text{NO}_3$, 383.1521, Found 383.1526.



(2R,3S)-1'-benzyl-5'-methoxy-3-phenyl-3H-spiro[furan-2,3'-indoline]-2',5(4H)-di one. Yield: 107.7mg (90%), white solid , mp 205-207°C , $R_f = 0.37$ (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{20} -144.6$ (c 0.8, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 70:30, 1.0 mL/min, 254 nm, 15.6 min (major), 34.4 min (minor)]. ^1H NMR (300 MHz, CDCl_3) δ 7.34-7.02 (*m*, 9H), 6.75-6.72 (*m*, 1H), 6.42-6.39 (*m*, 2H), 6.33 (*d*, $J = 8.6$ Hz, 1H), 4.95 (*d*, $J = 16.0$ Hz, 1H), 4.18-4.12 (*m*, 2H), 3.90 (*dd*, $J = 13.8$ Hz, $J = 16.7$ Hz, 1H), 3.80 (*s*, 3H), 2.93 (*dd*, $J = 7.9$ Hz, $J = 16.7$ Hz, 1H). ^{13}C NMR (300 MHz, CDCl_3) δ 174.8, 172.5, 156.5, 136.8, 134.4, 132.0, 128.8, 128.6, 128.4, 128.0, 127.3, 126.3, 125.8, 115.8, 111.1, 110.5, 86.6, 55.8, 50.8, 43.7, 32.2. IR (KBr) ν 1796, 1723, 1603, 1182, 697. HRMS (EI) m/z : M^+ Calc. for $\text{C}_{25}\text{H}_{21}\text{NO}_4$, 399.1471, Found 399.1475.

1.2 X-ray crystal structure of (-)-3d

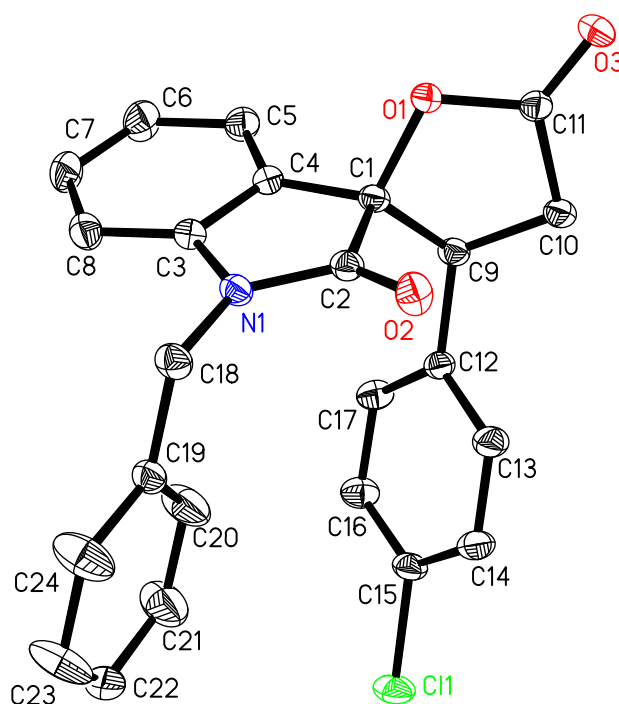


Figure S1 X-ray crystal structure of (-)-3d

The crystal was prepared from the solution of (-)-3d in ethyl acetate/petroleum

ether (5/1) with trace of petroleum ether.

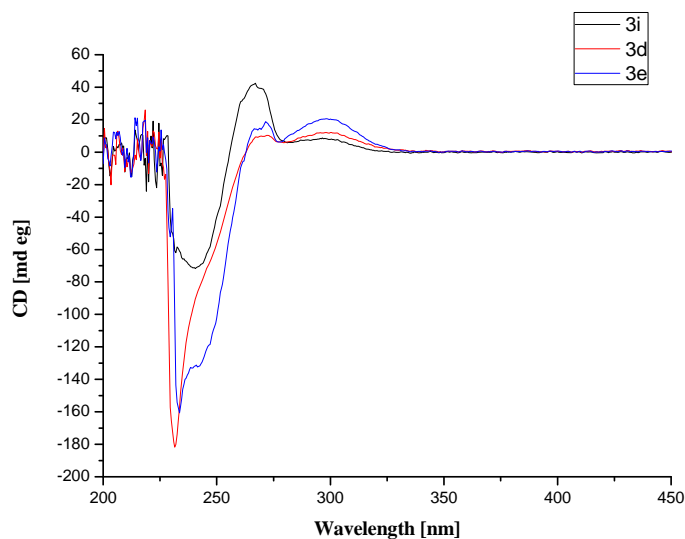


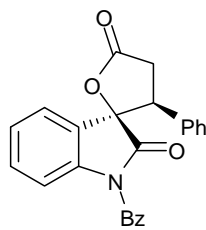
Figure S2 CD spectrum of (-)-**3d**, **3e** and **3i**

The stereochemistry of other products **3a-3m** were assigned by their specific rotation and CD spectrum.

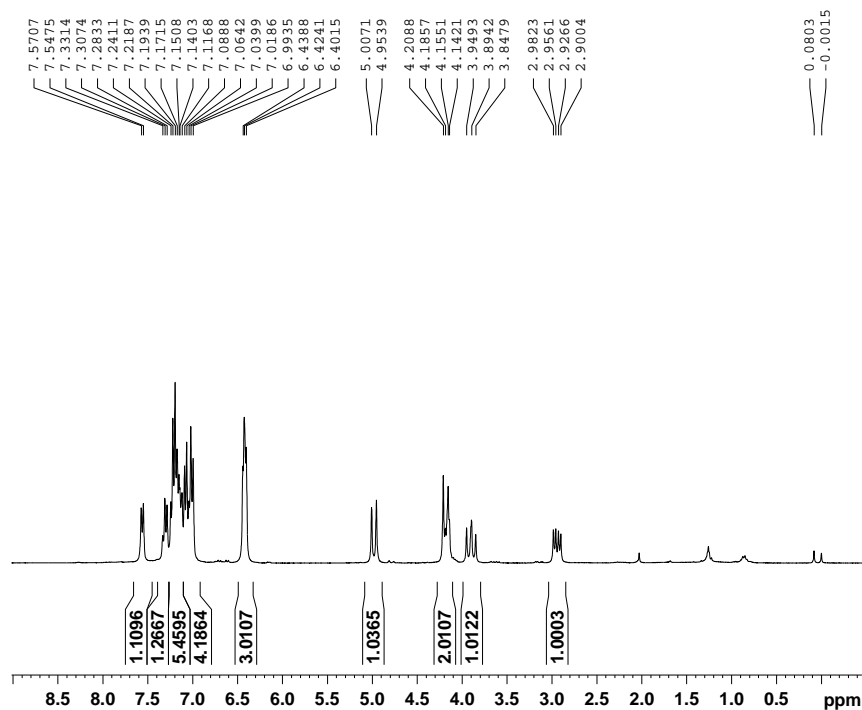
References

- (1) (a) Y. R. Zhang, L. He, X. Wu, P.L. Shao and S. Ye, *Org. Lett.* **2008**, *10*, 277. (b) H. Lv, Y. R. Zhang, X. L. Huang and S. Ye, *Adv. Synth. Catal.* **2008**, *350*, 2715. (c) L. He, Y. R. Zhang, X. L. Huang and S. Ye, *Synthesis*. **2008**, *17*, 2825. (d) X. L. Huang, L. He, P. L. Shao and S. Ye. *Angew. Chem. Int. Ed.* **2009**, *48*, 192.
- (2) Larry E. Overman and Emily A. Peterson. *Tetrahedron*. **2003**, *59*, 6905–6919.
- (3) Rosenmund Zetsche. *Chem. Ber.* **1923**, *56*, 1483.

Part II NMR Spectra



3aa

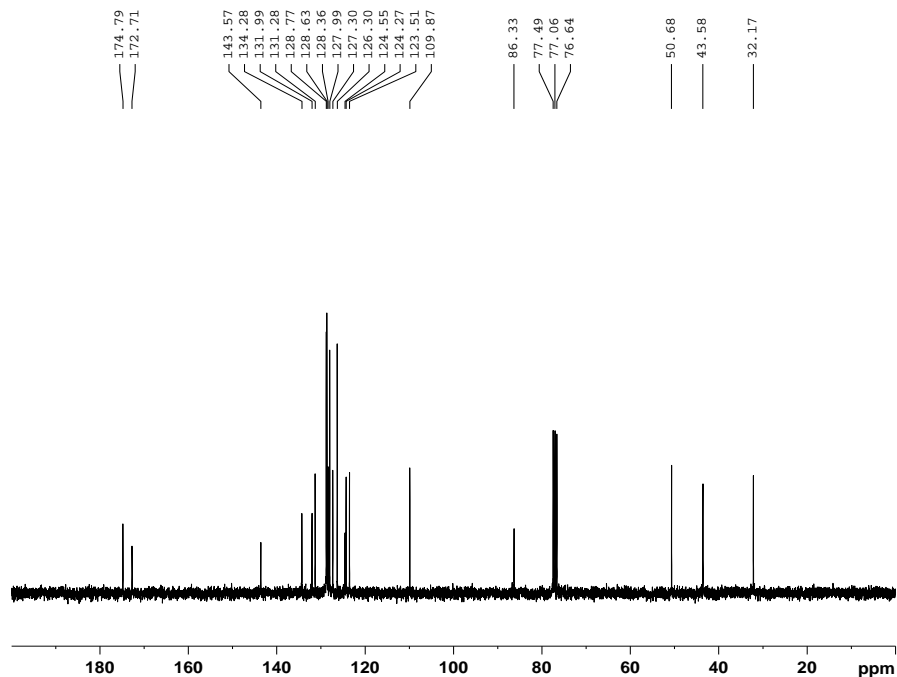


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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 128
DW 55.600 usec
DE 8.00 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz
```

```
F2 - Processing parameters
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SF 300.1300106 MHz
WDW EM
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GB 0
PC 1.00
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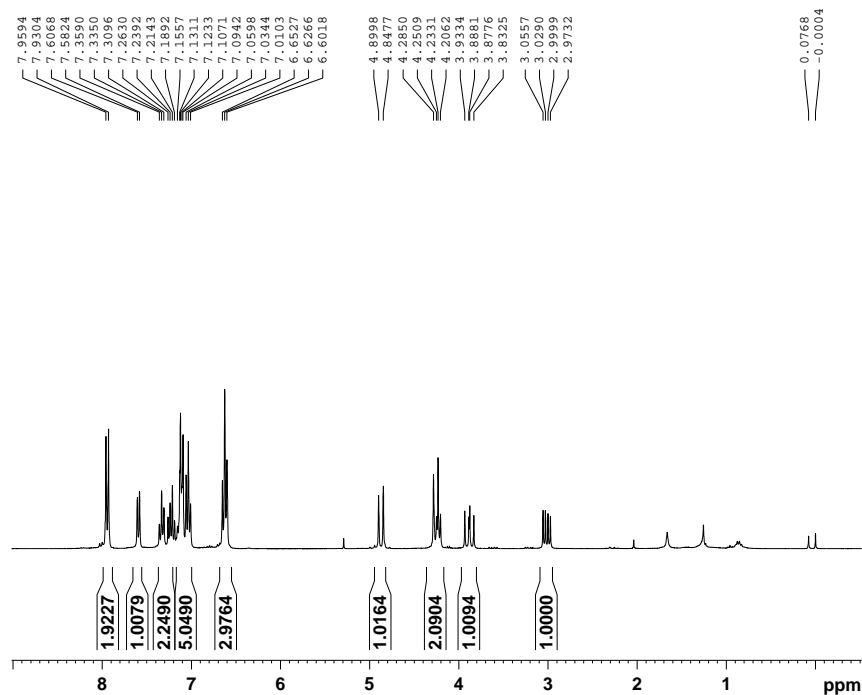
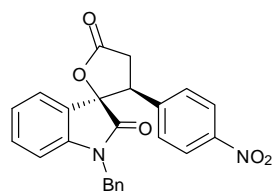
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EXPNO 21
PROCNO 1
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F2 - Acquisition Parameters
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 31
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 16384
DW 27.800 usec
DE 8.00 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz
```

```
F2 - Processing parameters
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WDW EM
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LB 1.00 Hz
GB 0
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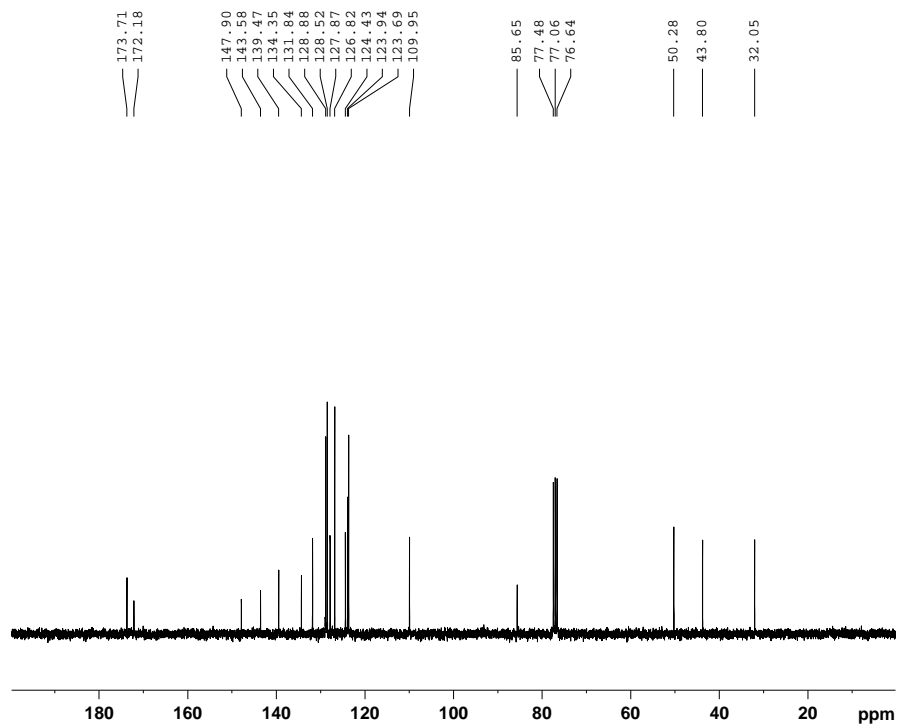


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 PROCNO 1

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 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 128
 DW 55.600 usec
 DE 8.00 usec
 TE 299.5 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.80 usec
 PL1 3.00 dB
 SFO1 300.1324010 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300050 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



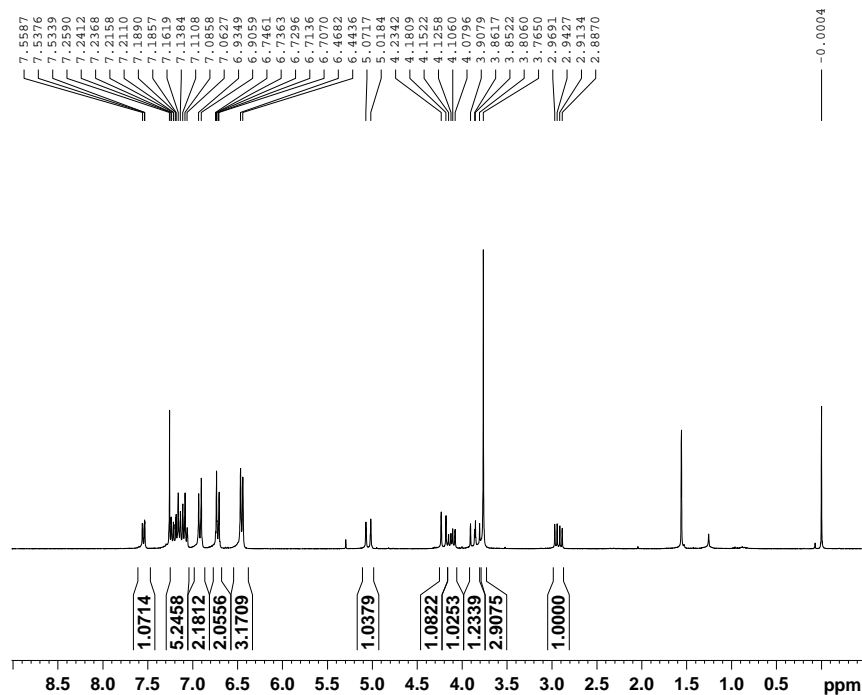
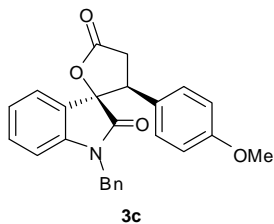
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 EXPNO 10
 PROCNO 1

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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 85
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 1625.5
 DW 27.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.50 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 3.00 dB
 PL12 22.33 dB
 PL13 23.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
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 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



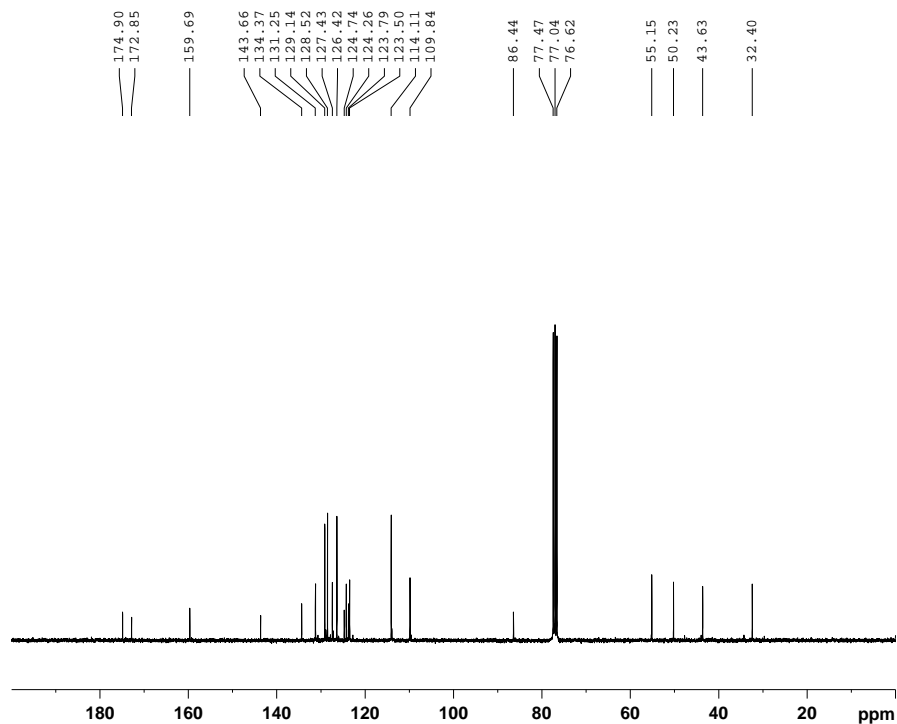
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EXPNO 10
PROCNO 1

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Time 8.18
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 456.1
DW 55.600 usec
DE 8.00 usec
TE 299.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
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SF 300.1300064 MHz
WDW EM
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LB 0.30 Hz
GB 0
PC 1.00
    
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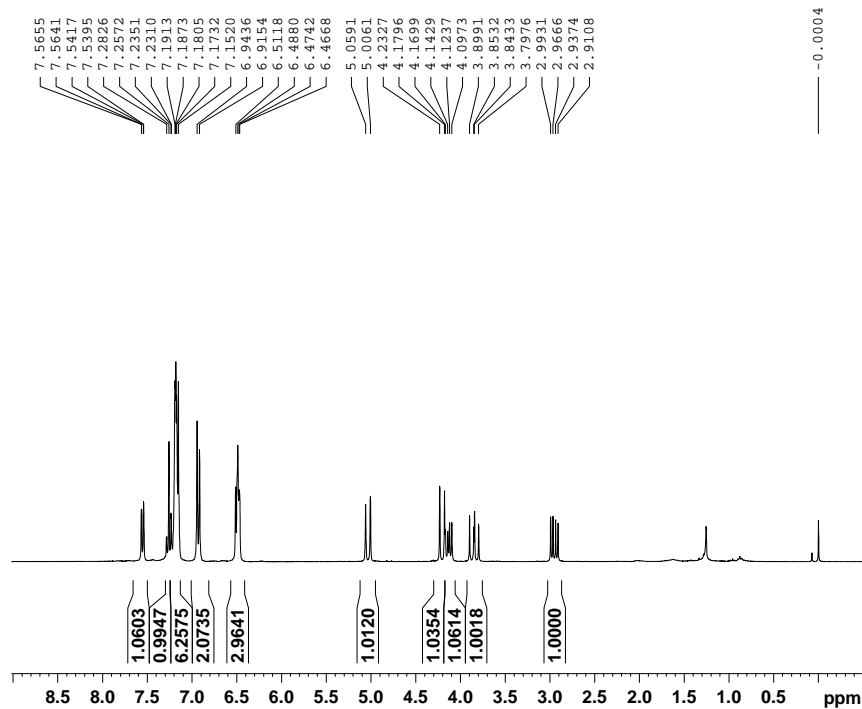
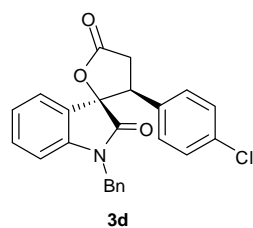
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EXPNO 20
PROCNO 1

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Time 21.54
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 5792.6
DW 27.800 usec
DE 8.00 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

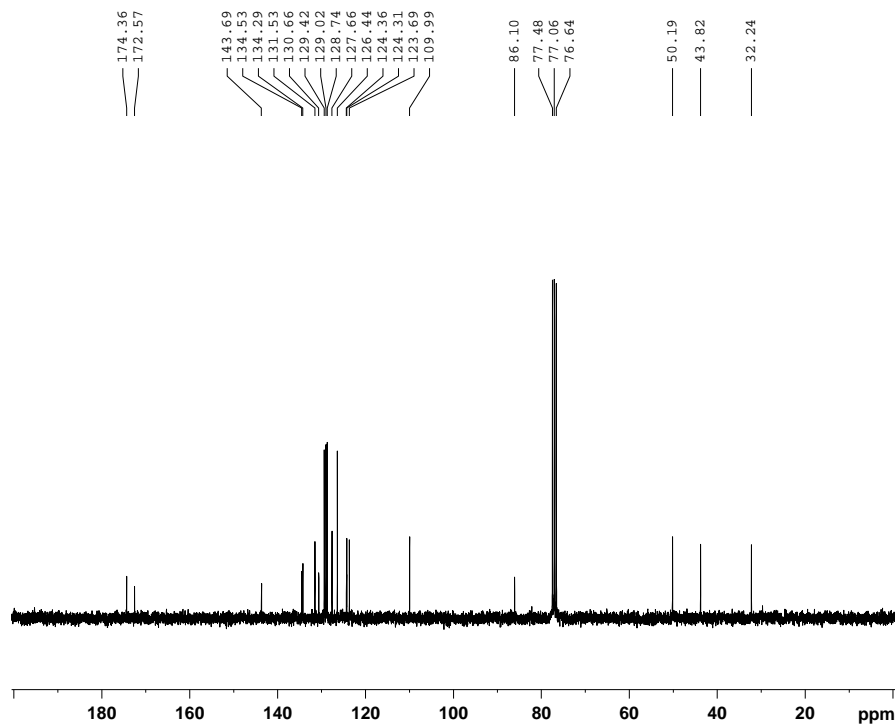


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PROCNO   1

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Time     20.50
INSTRUM  spect
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PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       0
SWH      8992.806 Hz
FIDRES   0.137219 Hz
AQ       3.6438515 sec
RG       256
DW       55.600 usec
DE       8.00 usec
TE       295.4 K
D1       1.00000000 sec
TD0      1
```

```
===== CHANNEL f1 =====
NUC1     1H
P1       10.80 usec
PL1      3.00 dB
SFO1     300.1324010 MHz
```

```
F2 - Processing parameters
SI       32768
SF       300.1300067 MHz
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LB       0.30 Hz
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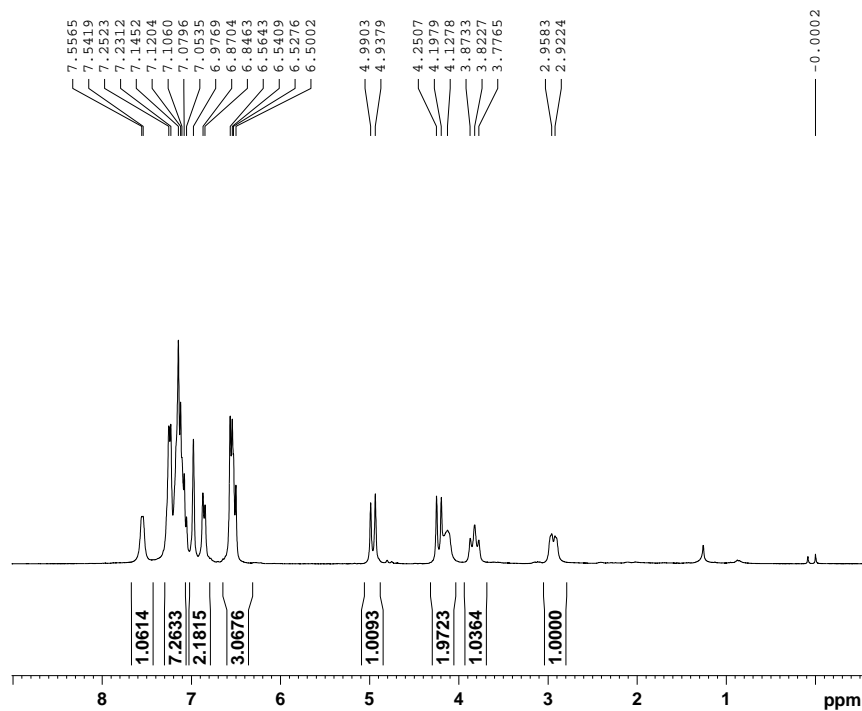
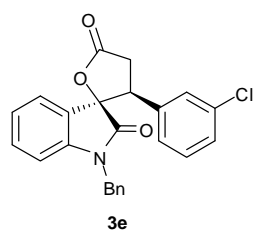
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PROCNO   1

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INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       111
DS       4
SWH      17985.611 Hz
FIDRES   0.274439 Hz
AQ       1.8219508 sec
RG       645.1
DW       27.800 usec
DE       6.50 usec
TE       299.7 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1
```

```
===== CHANNEL f1 =====
NUC1     13C
P1       12.50 usec
PL1      2.00 dB
SFO1     75.4752953 MHz
```

```
===== CHANNEL f2 =====
PCPDPRG2 waltz16
NUC2     1H
PCPD2    100.00 usec
PL2      3.00 dB
PL12     22.33 dB
PL13     23.00 dB
SFO2     300.1312005 MHz
```

```
F2 - Processing parameters
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SF       75.4675441 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

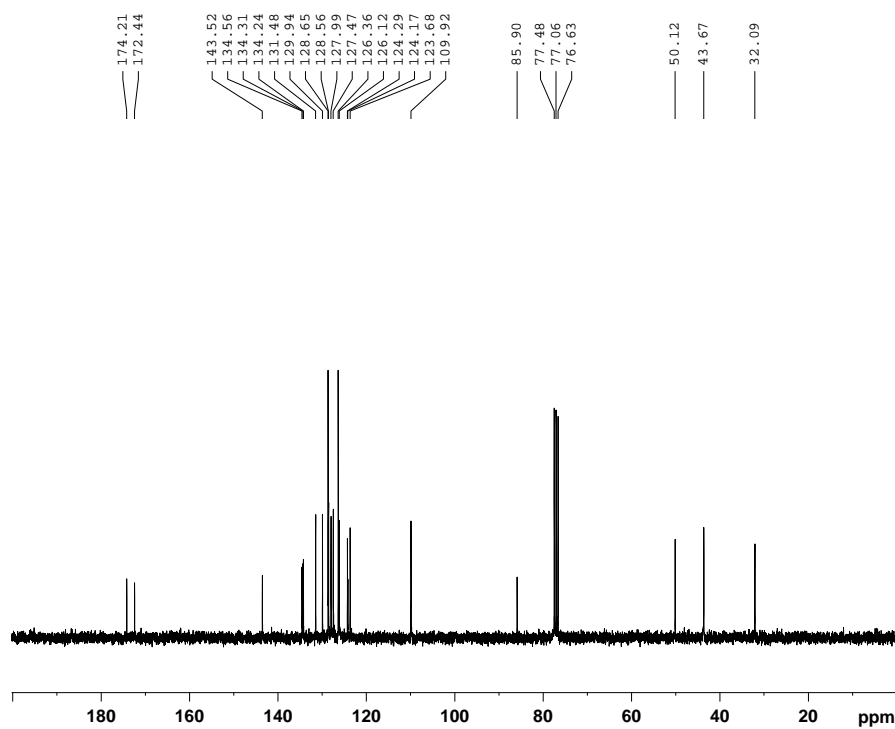



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EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
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Time 11.37
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 128
DW 55.600 usec
DE 8.00 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
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SF 300.1292086 MHz
WDW EM
SBB 0
LB 0.30 Hz
GB 0
PC 1.00
```



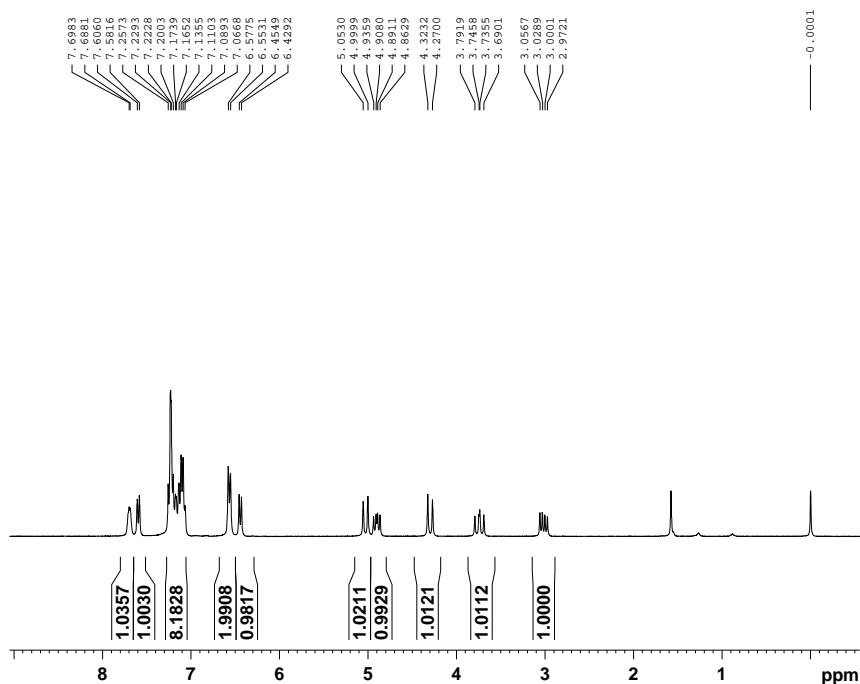
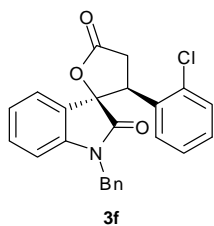
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EXPNO 11
PROCNO 1

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Time 11.40
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 24
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2896.3
DW 27.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz
```

```
F2 - Processing parameters
SI 32768
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LB 1.00 Hz
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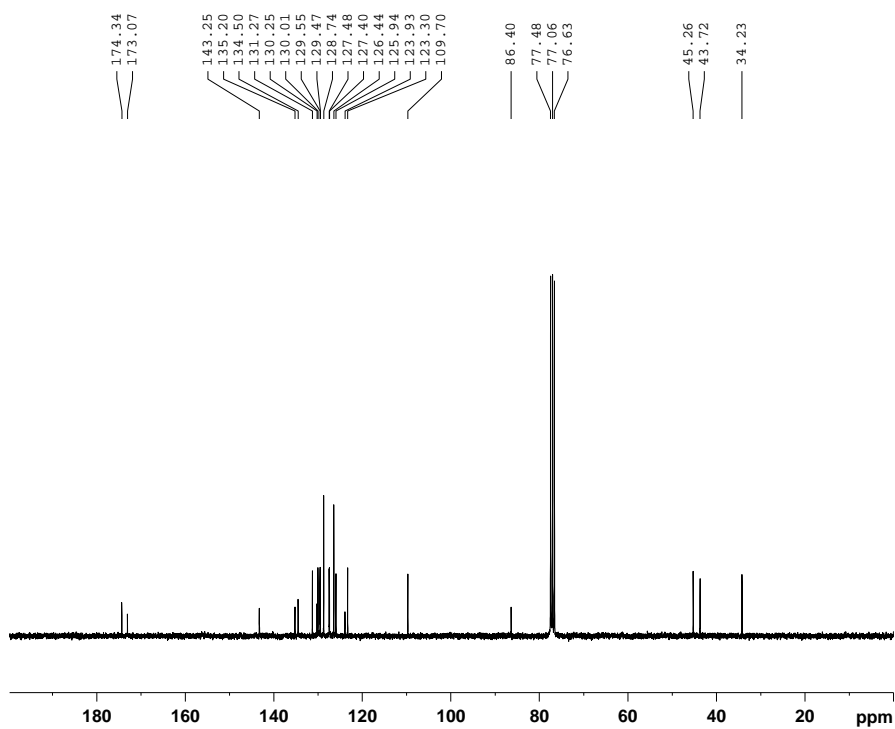
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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 456.1
DW 55.600 usec
DE 8.00 usec
TE 299.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
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SF 300.1292317 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
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```

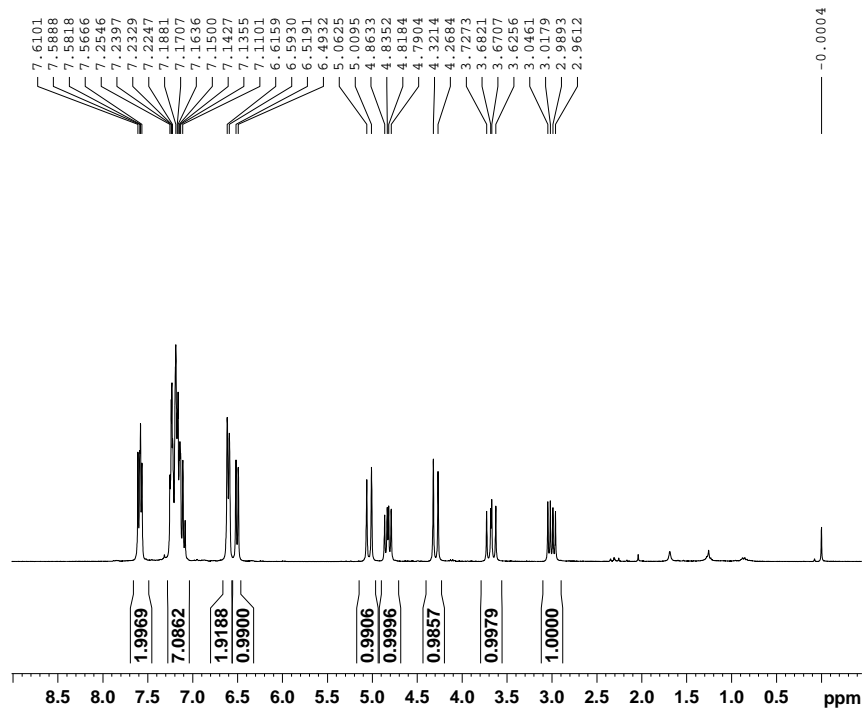
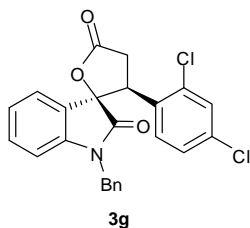
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EXPNO 10
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 823
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 456.1
DW 27.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
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SF 75.4675513 MHz
WDW EM
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LB 1.00 Hz
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PC 1.40
    
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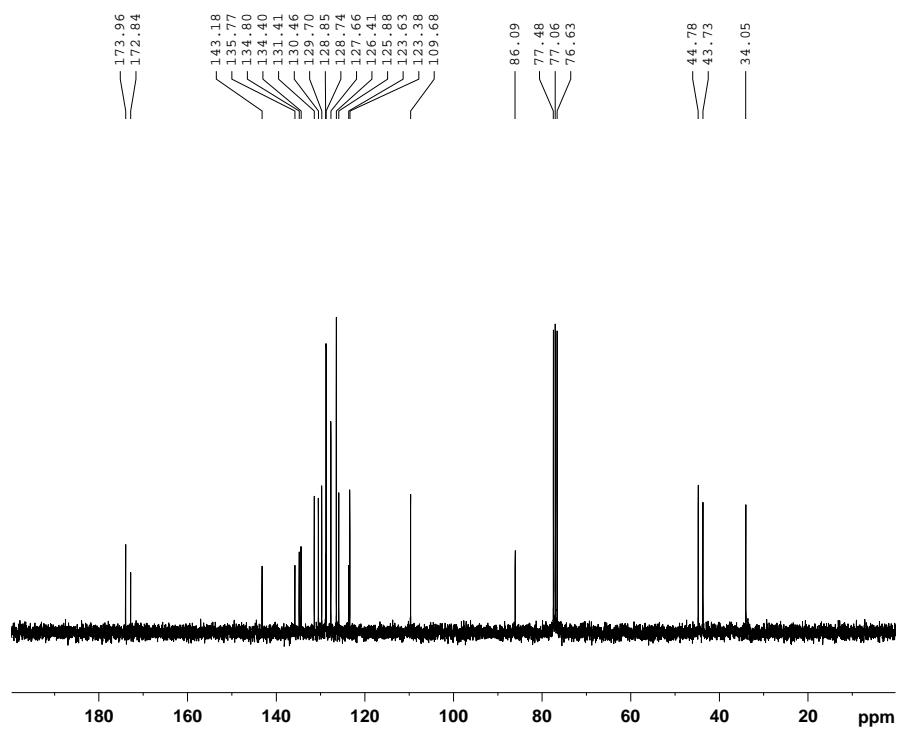


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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 128
DW 55.600 usec
DE 8.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
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LB 0.30 Hz
GB 0
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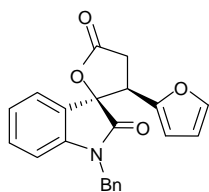
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PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 456.1
DW 27.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
```

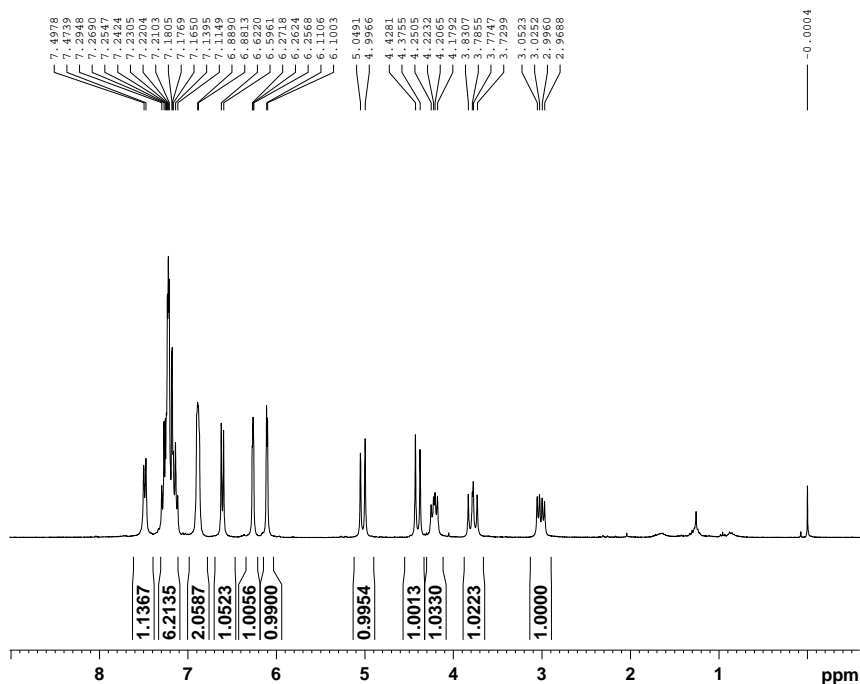
```
===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz
```

```
F2 - Processing parameters
SI 32768
SF 75.4677505 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```



3h

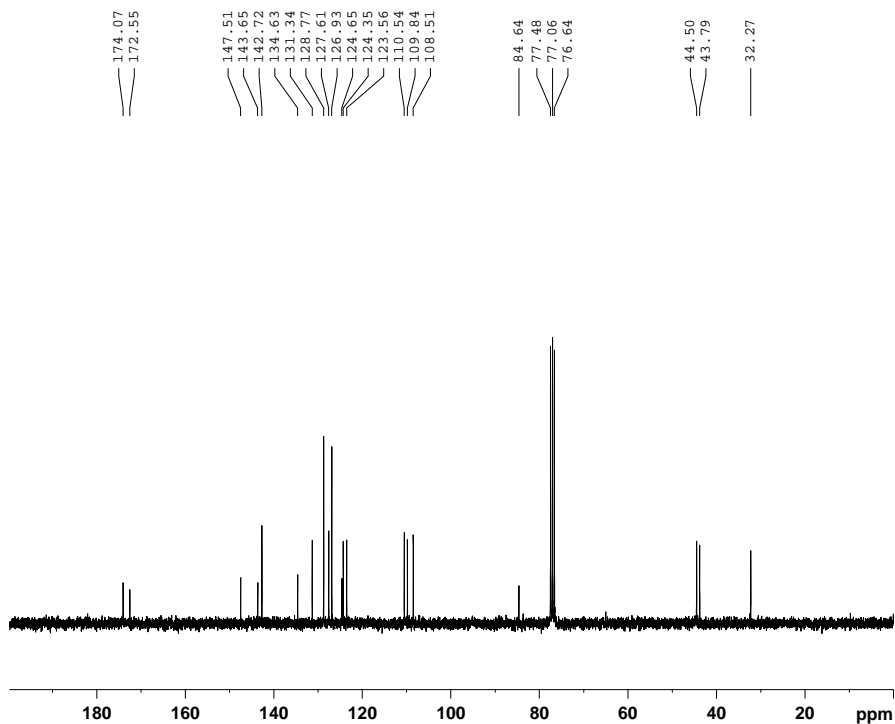


```
Current Data Parameters
NAME sunlh-443A
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110323
Time 8.23
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 181
DW 55.600 usec
DE 8.00 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300074 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```



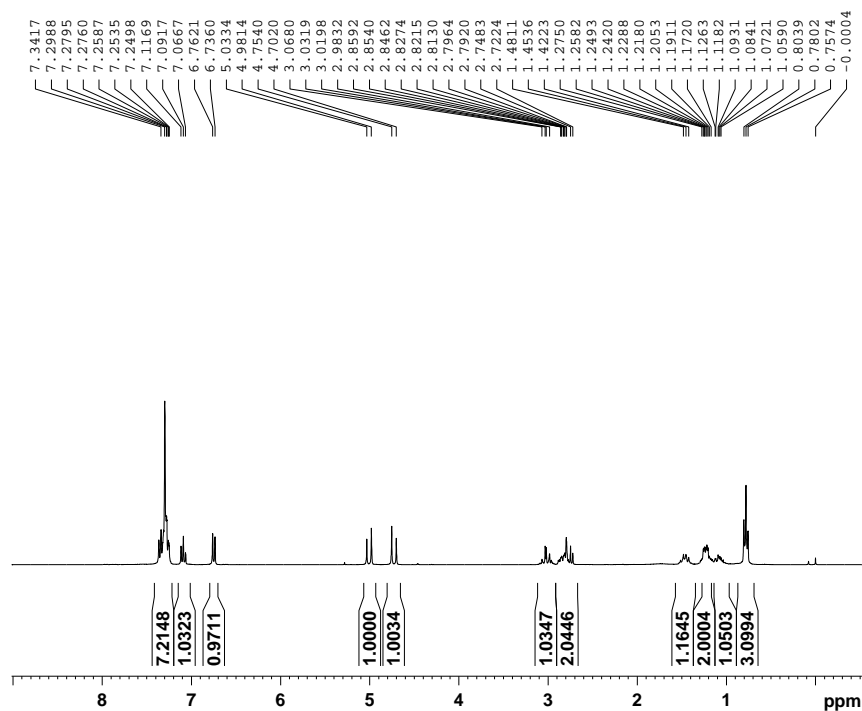
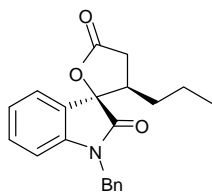
```
Current Data Parameters
NAME sunlh-443A
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110323
Time 8.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 49
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 8.00 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677511 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```

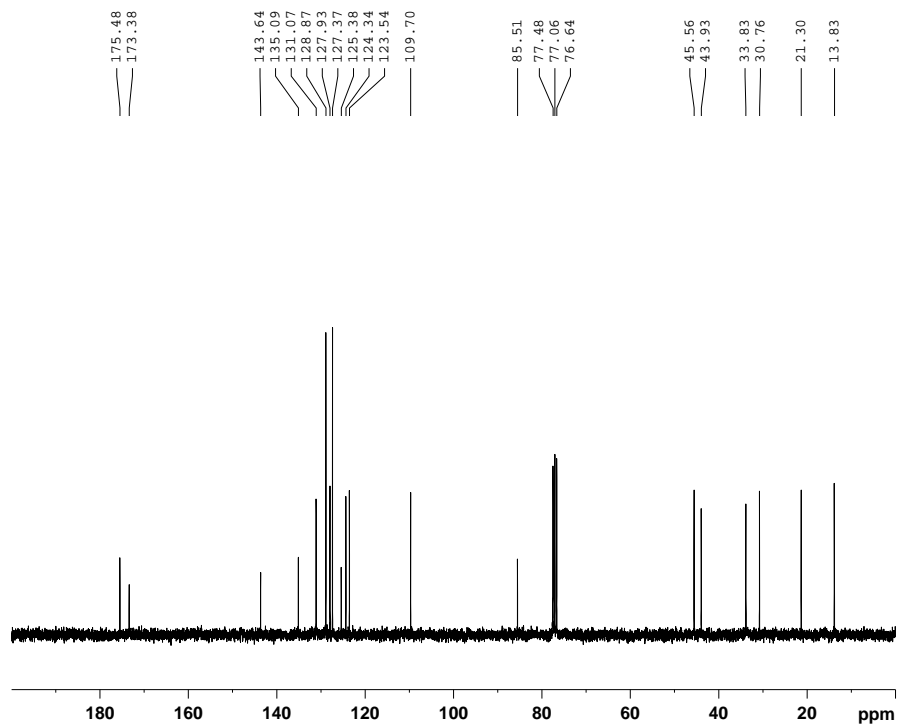


```
Current Data Parameters
NAME sunh-554H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110623
Time 20.53
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 128
DW 55.600 usec
DE 8.00 usec
TE 299.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SF01 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```



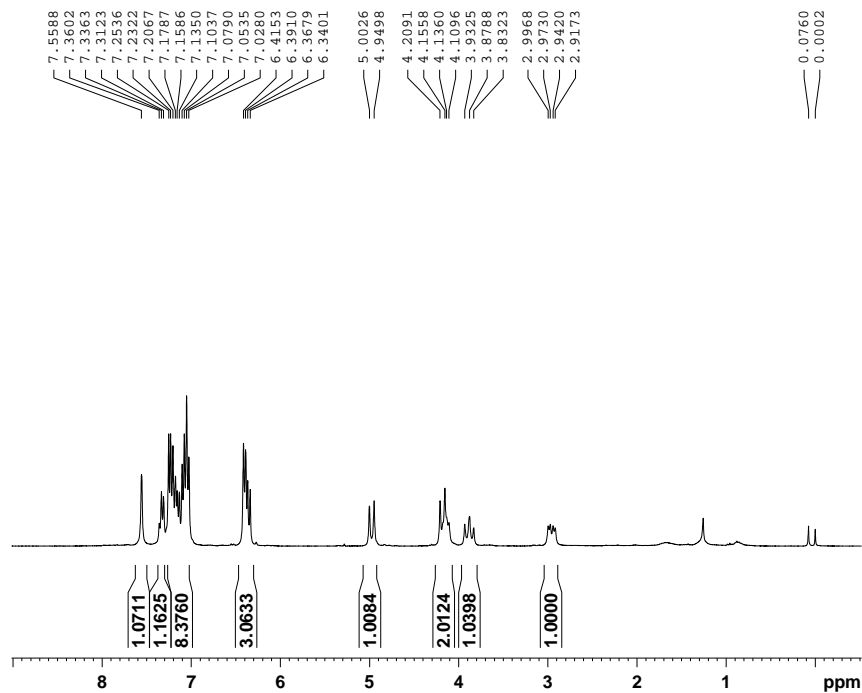
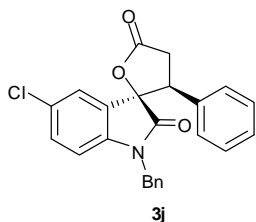
```
Current Data Parameters
NAME sunh-554C
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110623
Time 20.57
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 57
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 456.1
DW 27.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SF01 75.4752953 MHz

===== CHANNEL f2 =====
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```

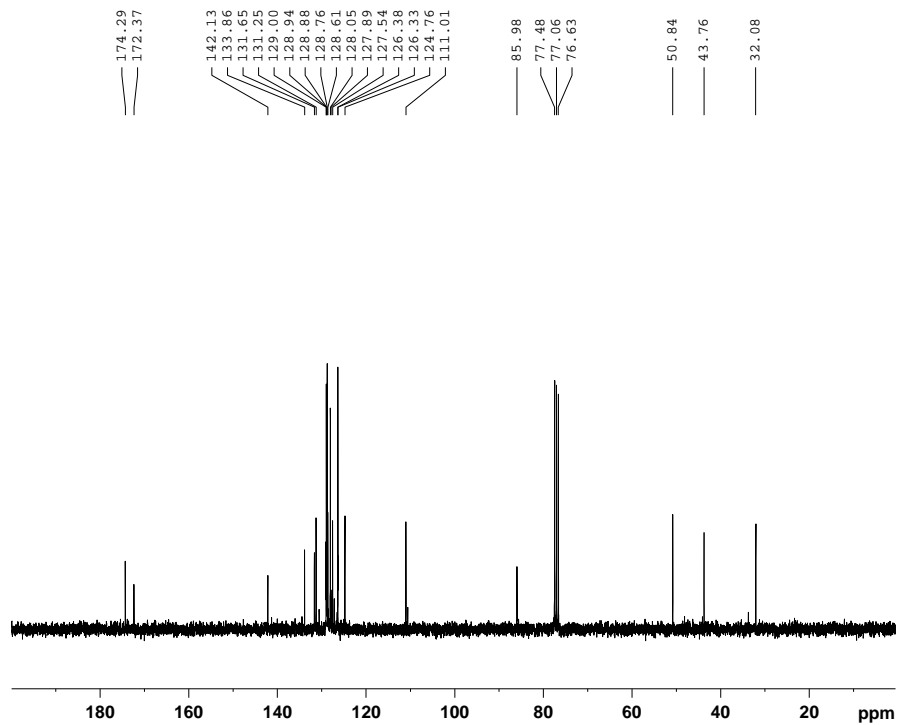


```
Current Data Parameters
NAME sunlh-0624-405
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110624
Time 16.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 11
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 181
DW 55.600 usec
DE 8.00 usec
TE 299.6 K
D1 1.00000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
PL 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1292073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```



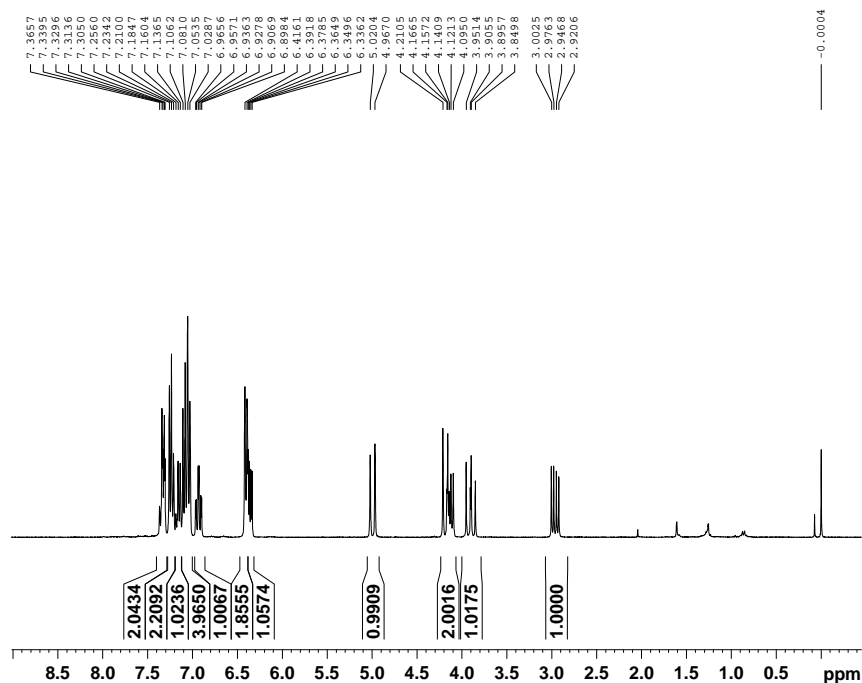
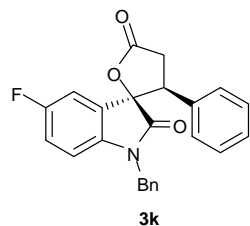
```
Current Data Parameters
NAME sunlh-424A
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110120
Time 17.33
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 56
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 6502
DW 27.800 usec
DE 8.00 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 13C
PL 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```

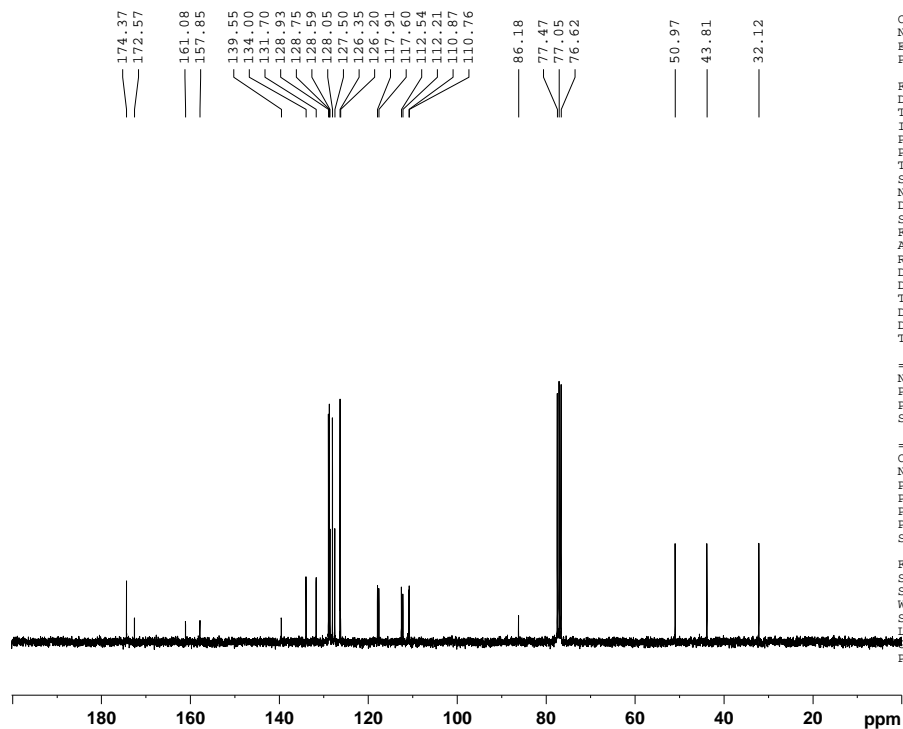


```
Current Data Parameters
NAME sunlh-407A
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101227
Time 18.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 228.1
DW 55.600 usec
DE 8.00 usec
TE 296.8 K
D1 1.0000000 sec
TD0 1
```

```
===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300072 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
```



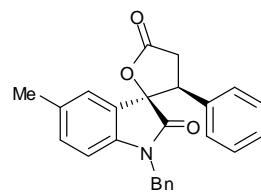
```
Current Data Parameters
NAME sunlh-407A
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101227
Time 18.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 153
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 6502
DW 27.800 usec
DE 8.00 usec
TE 297.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
```

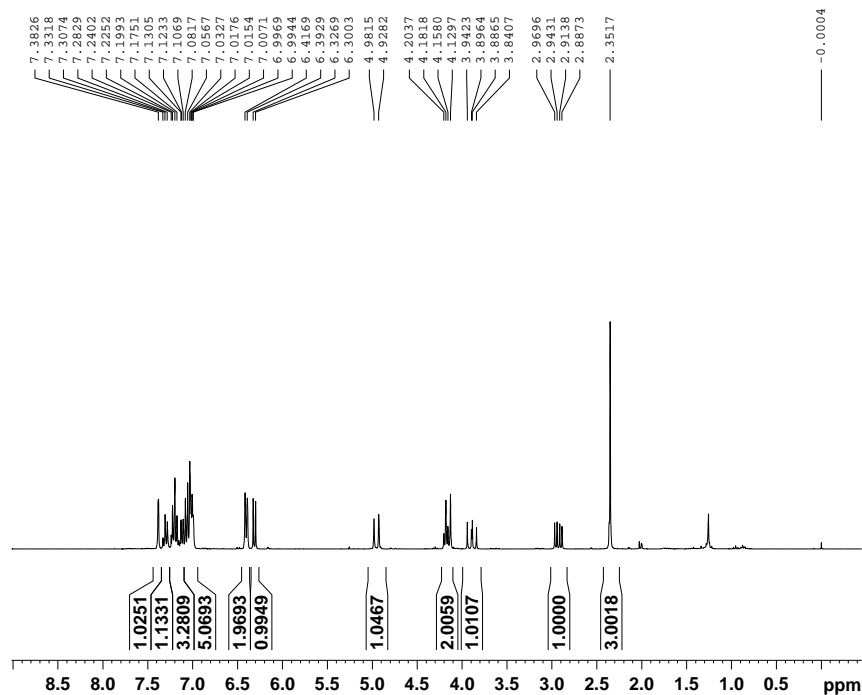
```
===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
```



3i



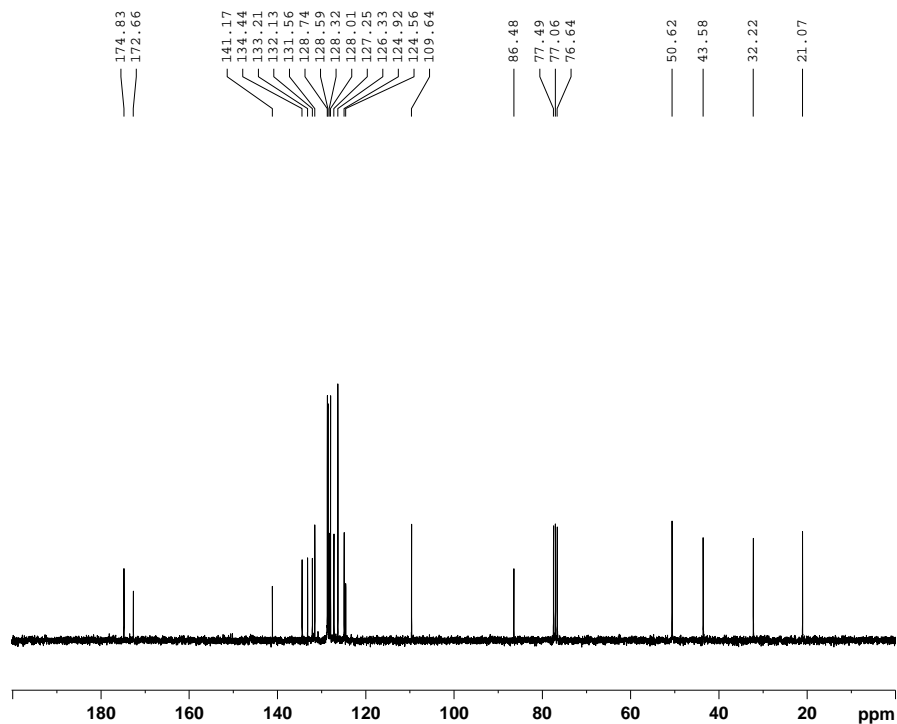
-0.0004

Current Data Parameters
 NAME sunlh-424B
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110125
 Time 8.55
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 11
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 57
 DW 55.600 usec
 DE 8.00 usec
 TE 295.1 K
 D1 1.00000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.80 usec
 PL1 3.00 dB
 SFO1 300.1324010 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300117 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



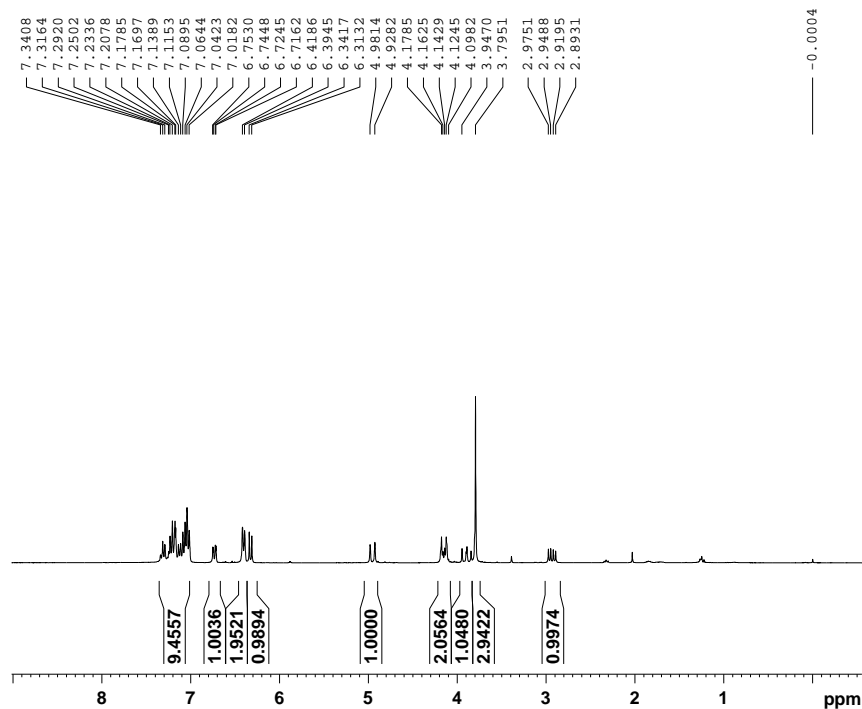
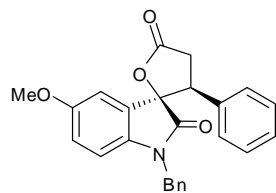
Current Data Parameters
 NAME sunlh-424B
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20110120
 Time 17.44
 INSTRUM spect
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 45
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 5792.6
 DW 27.800 usec
 DE 8.00 usec
 TE 296.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 12.50 usec
 PL1 2.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 100.00 usec
 PL2 3.00 dB
 PL12 22.33 dB
 PL13 23.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677538 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



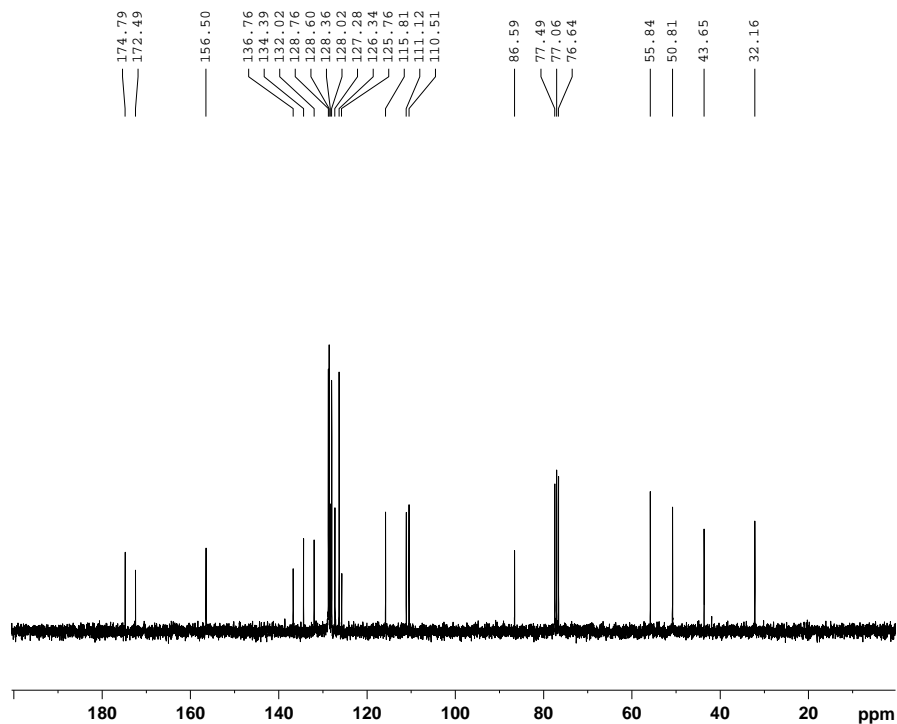
```

Current Data Parameters
NAME sunlh-425B
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110121
Time 16.35
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 128
DW 55.600 usec
DE 8.00 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
    
```



```

Current Data Parameters
NAME sunlh-425B
EXPNO 11
PROCNO 1

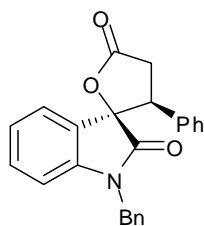
F2 - Acquisition Parameters
Date_ 20110121
Time 16.38
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 22
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 8.00 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.50 usec
PL1 2.00 dB
SFO1 75.4752953 MHz

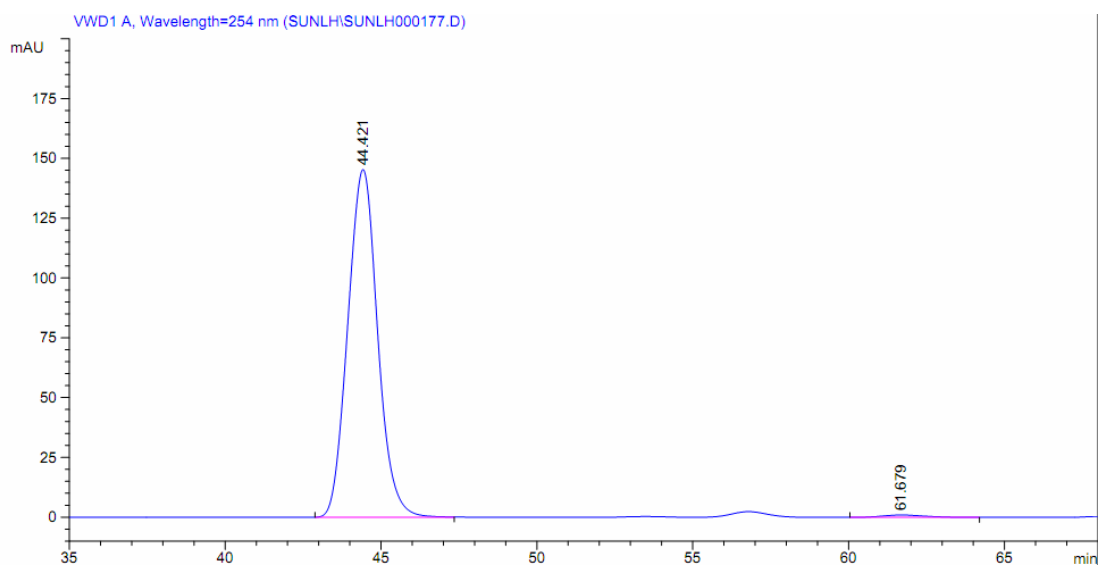
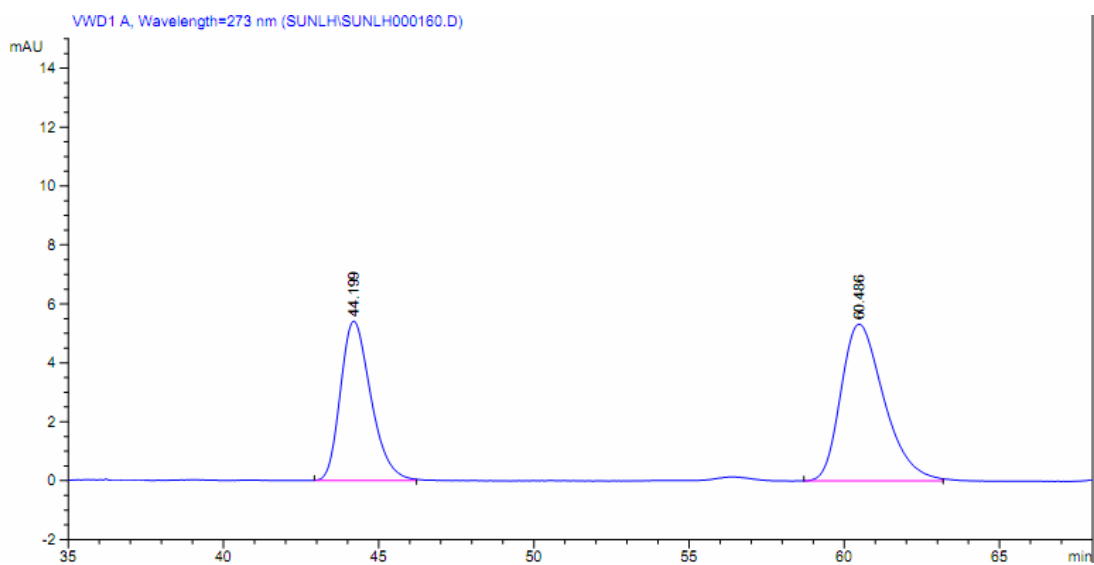
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 3.00 dB
PL12 22.33 dB
PL13 23.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677537 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```

Part III HPLC Spectra

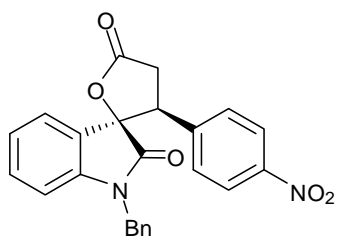


3aa

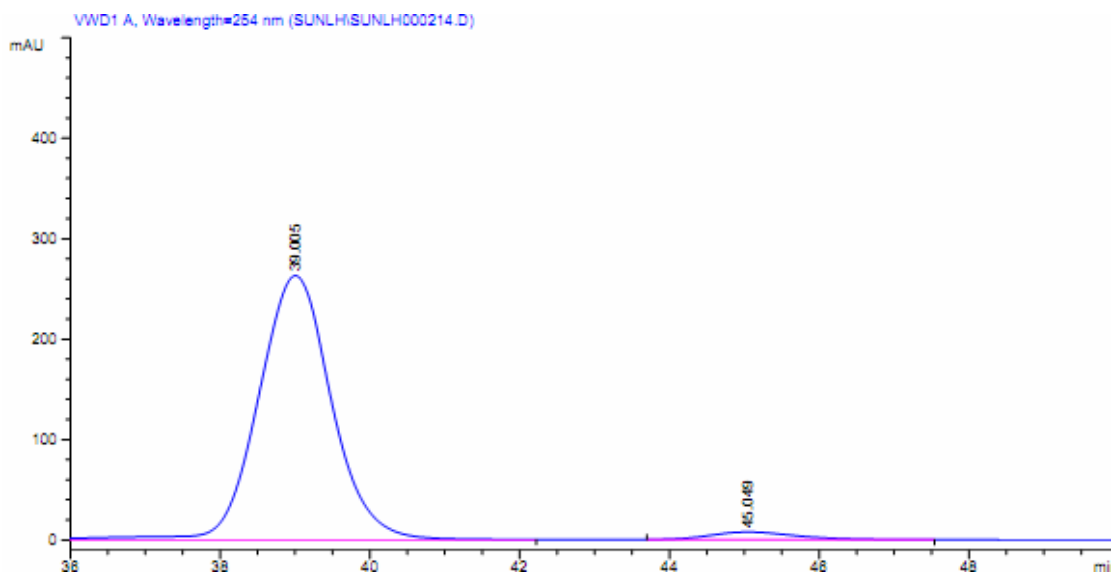
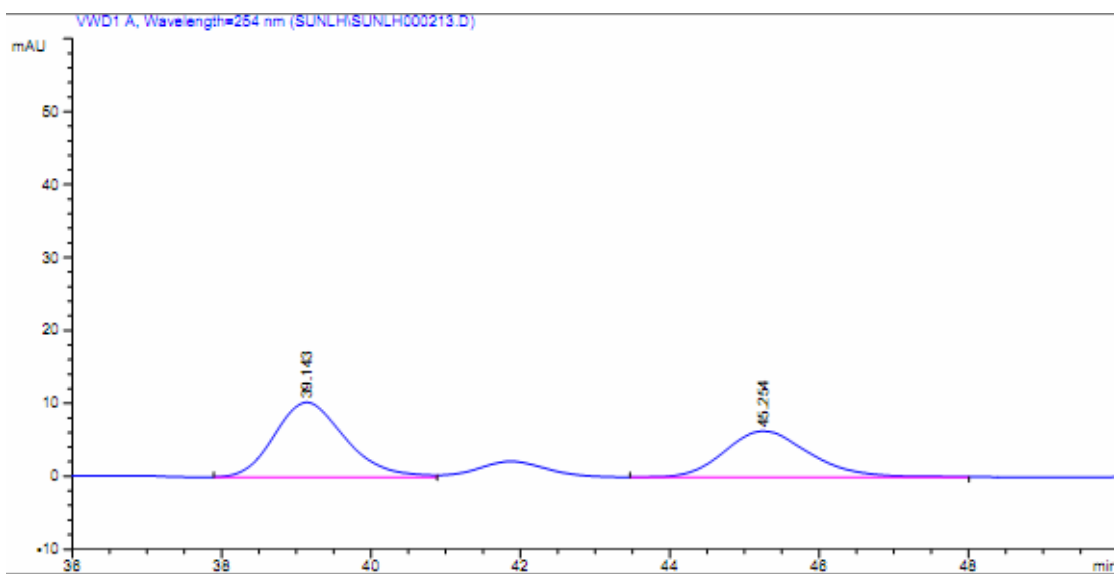


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	44.421	BB	1.0332	9677.83789	145.32806	99.0280
2	61.679	BB	1.4734	94.98950	9.75888e-1	0.9720

Totals : 9772.82739 146.30395



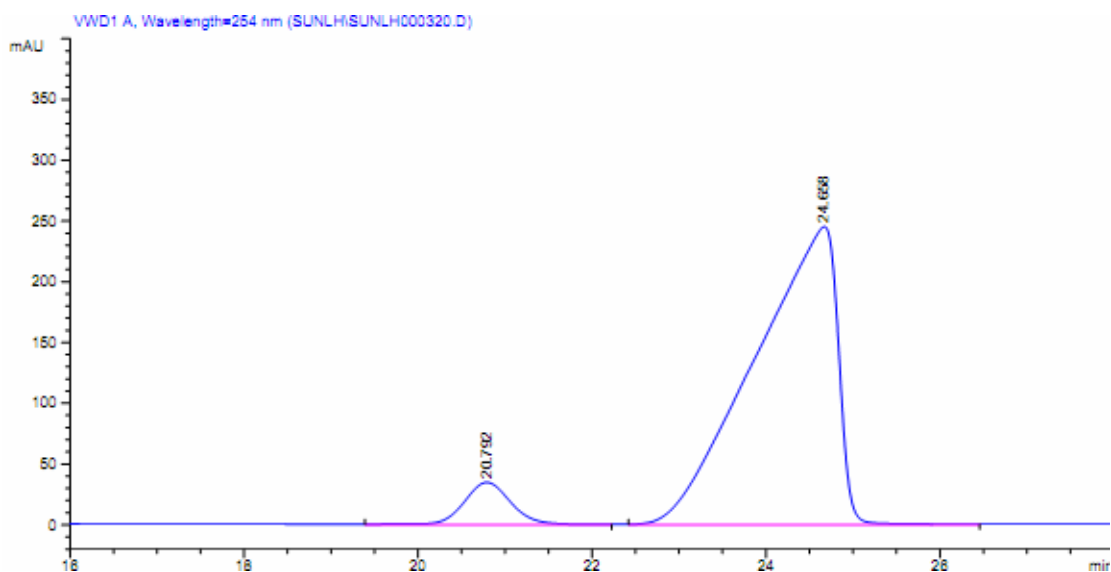
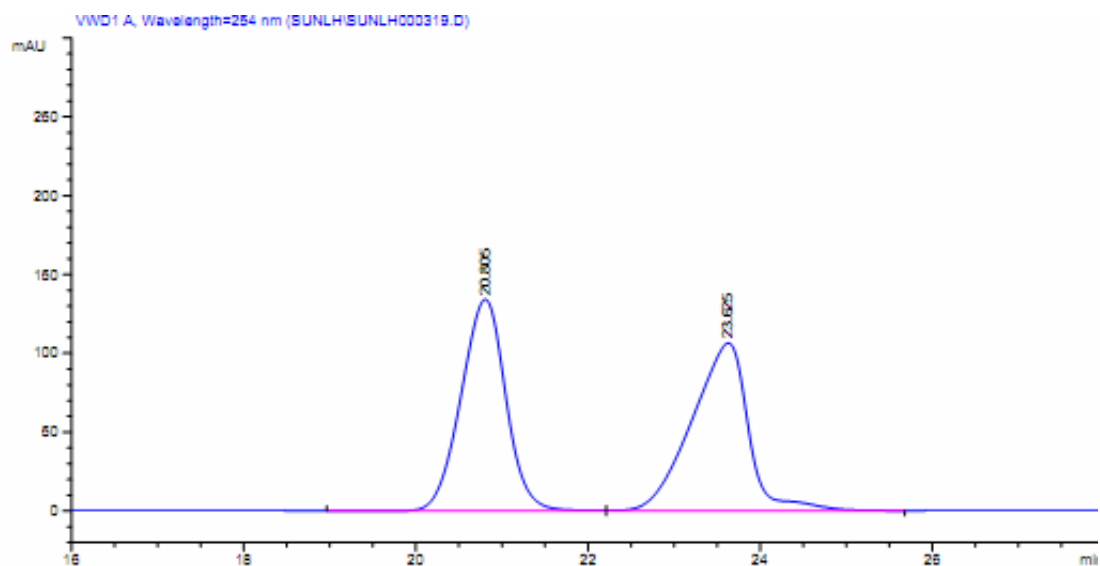
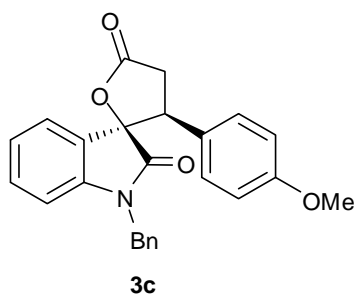
3b



Signal 1: VWD1 A, Wavelength=254 nm

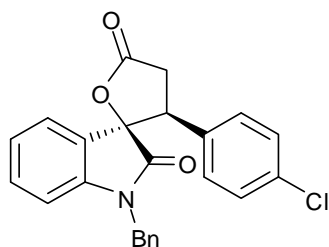
Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	39.005	BB	1.0513	1.79186e4	262.94363	96.5980
2	45.049	BB	1.2232	631.06580	7.70679	3.4020

Totals : 1.85497e4 270.65043

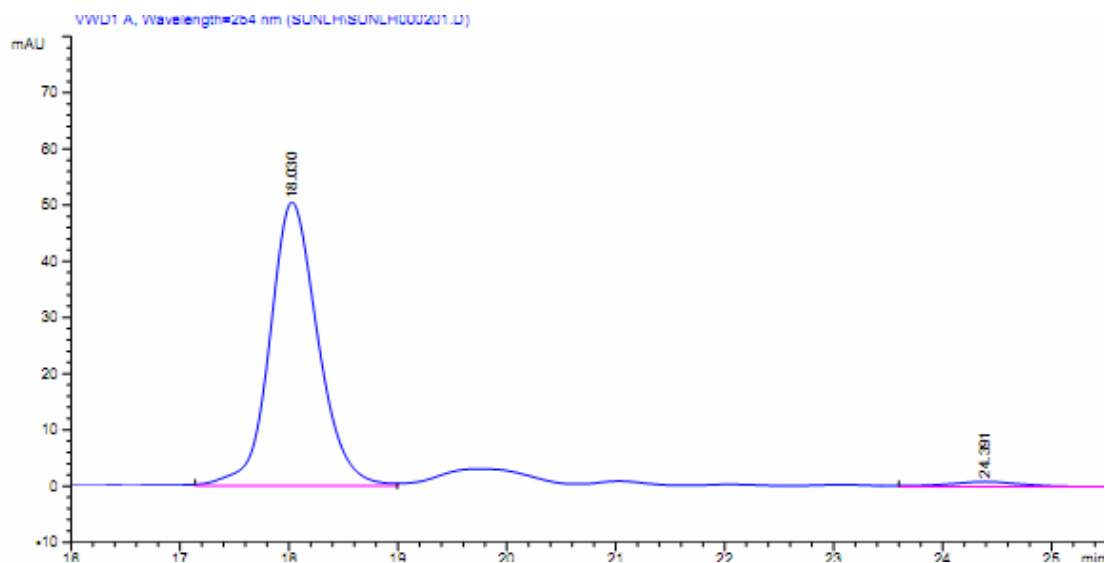
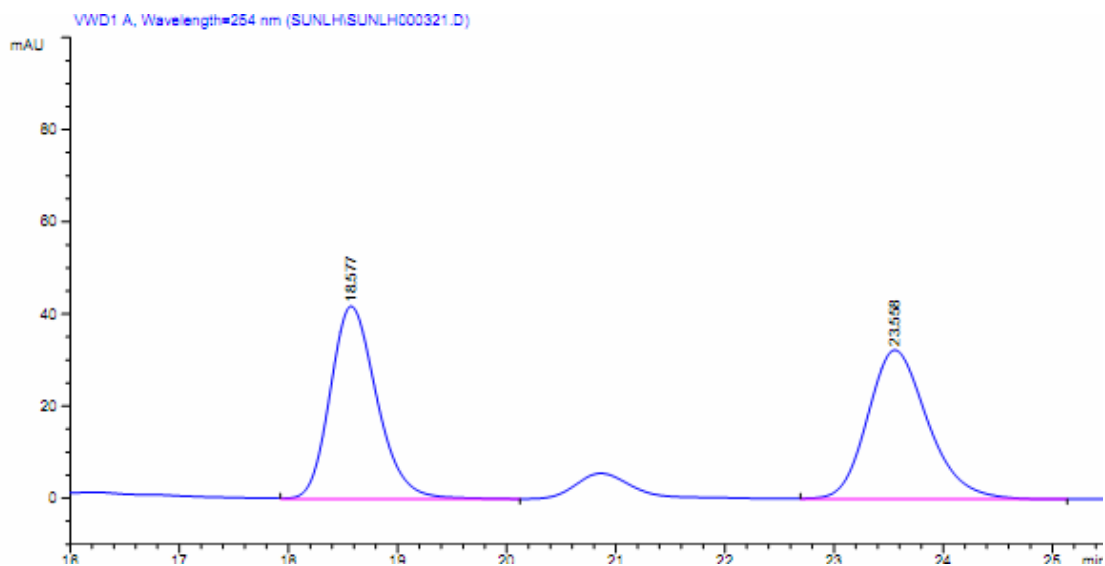


Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.792	BB	0.5803	1311.55823	34.73857	7.4535
2	24.658	BB	0.9491	1.62849e4	245.34042	92.5465
Totals :				1.75965e4	280.07899	



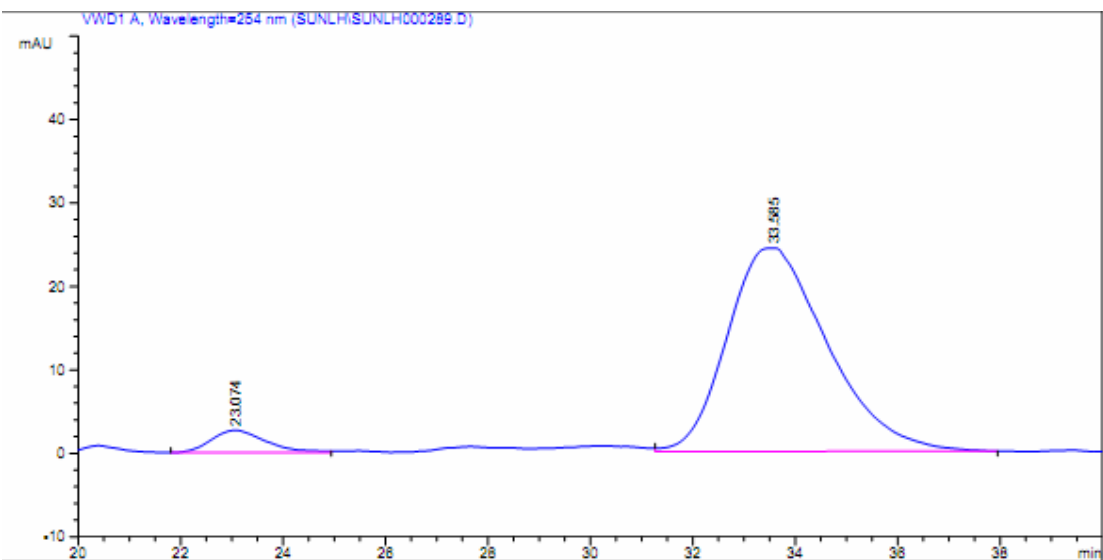
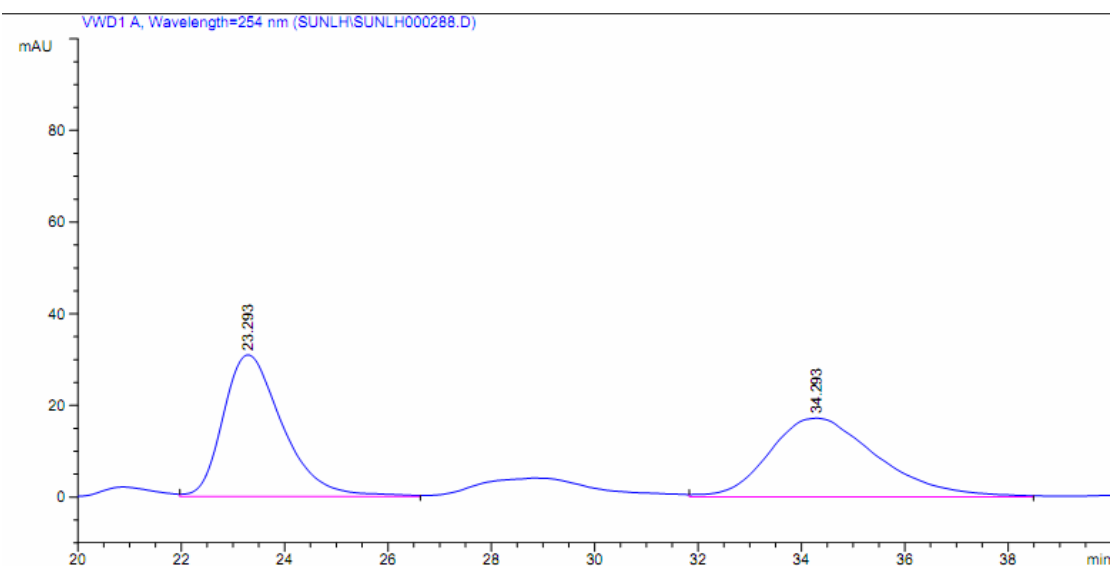
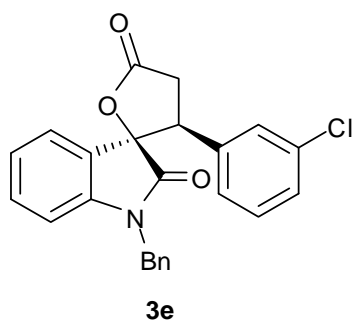
3d



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	18.030	BV	0.4632	1548.25806	50.39798	50.39798	97.5152
2	24.391	VB	0.6828	39.45204	8.66138e-1	8.66138e-1	2.4848

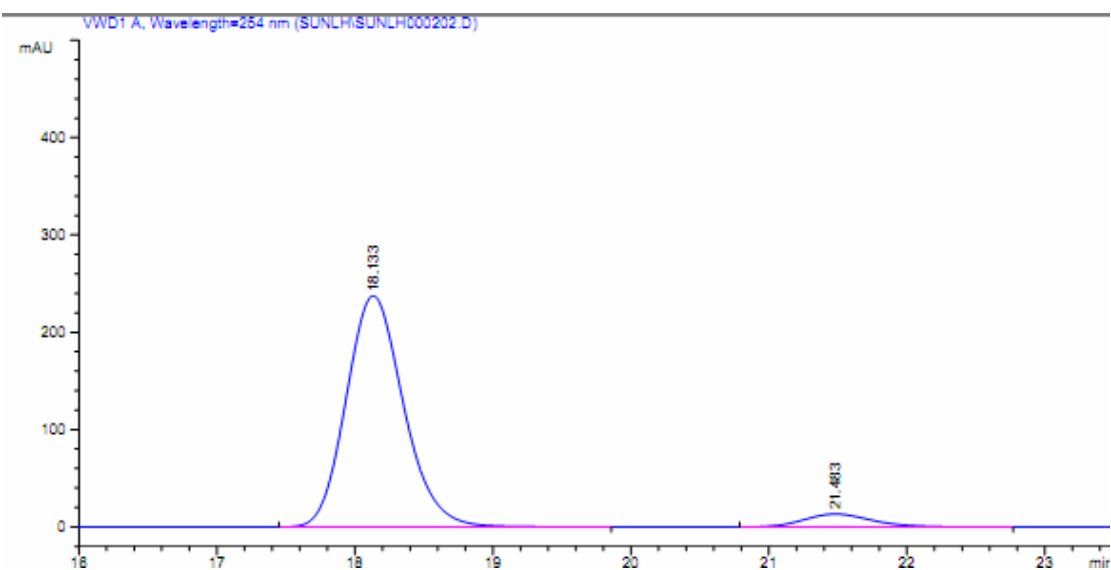
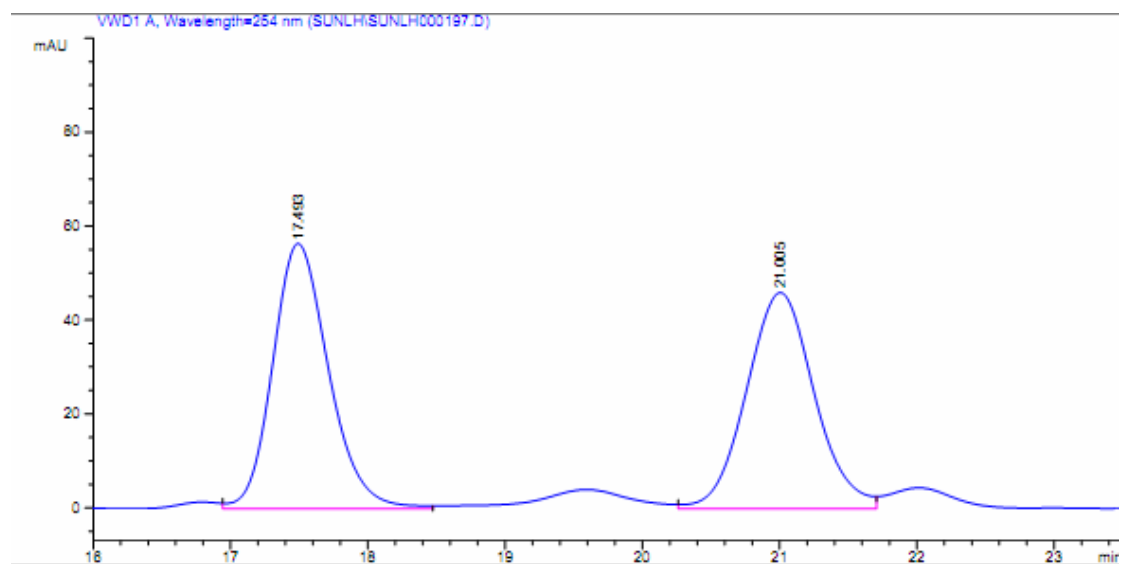
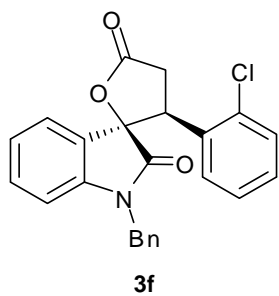
Totals : 1587.71010 51.26412



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.074	BB	1.1445	197.60316	2.66536	5.6110
2	33.585	VB	1.6616	3324.12988	24.36519	94.3890

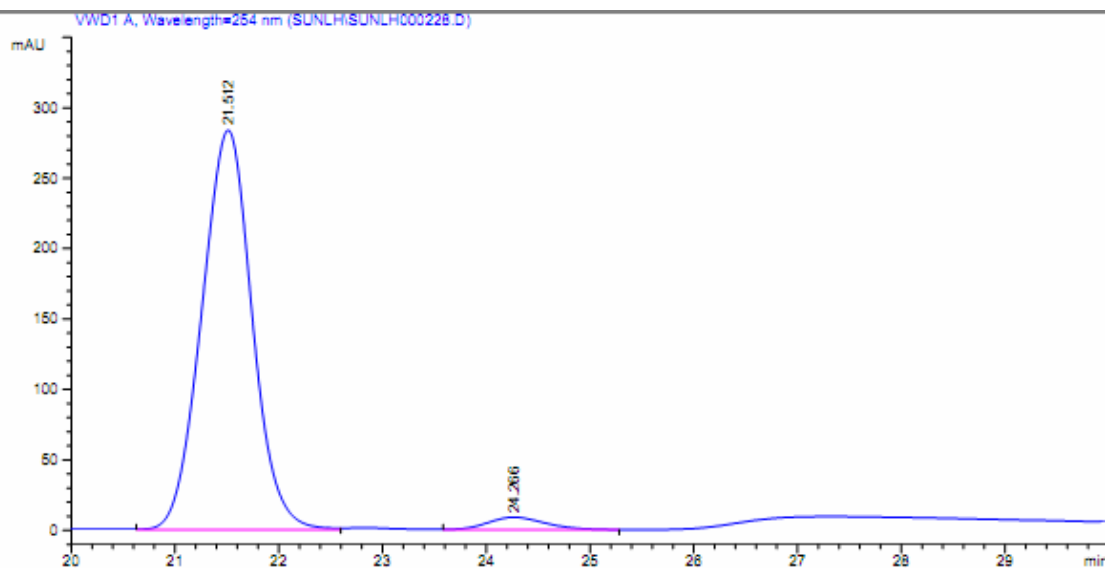
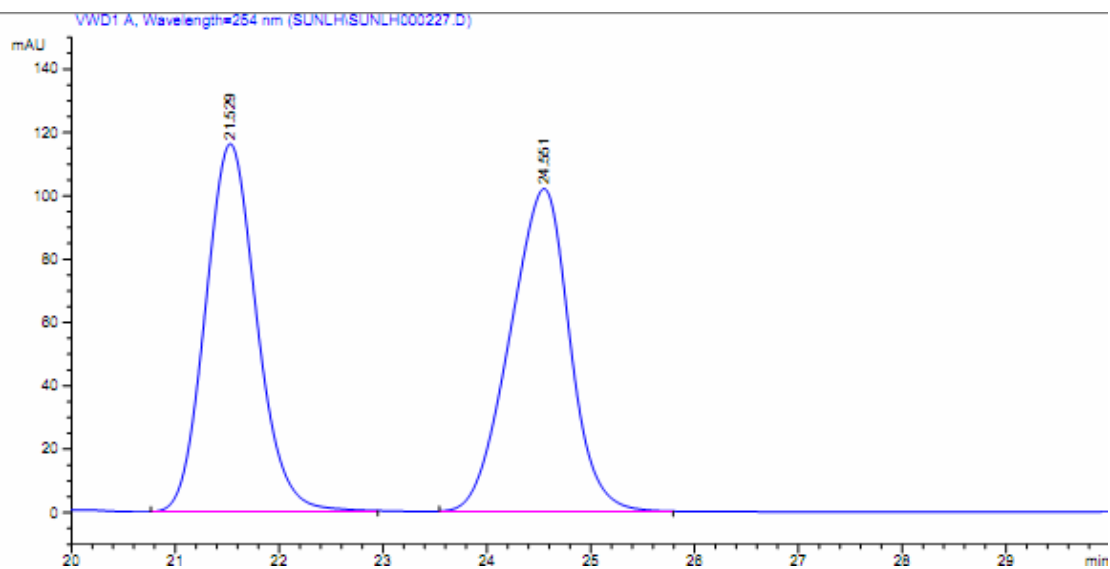
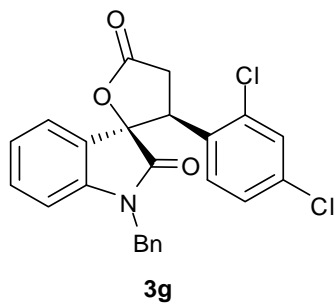
Totals : 3521.73305 27.03056



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.133	BB	0.4547	6880.00293	237.30621	93.6845
2	21.483	BB	0.5348	463.79675	13.30462	6.3155

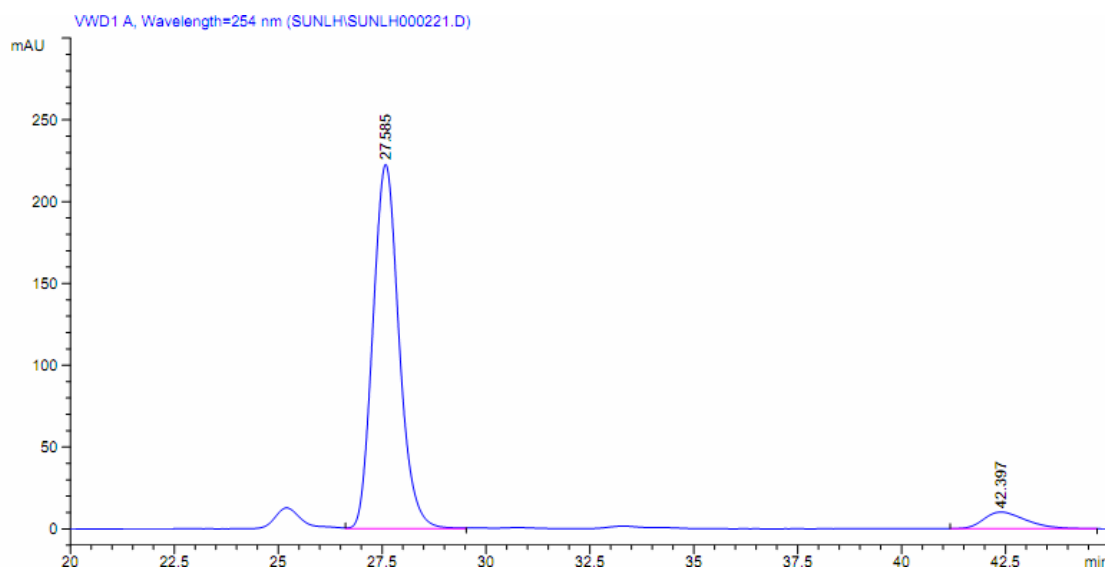
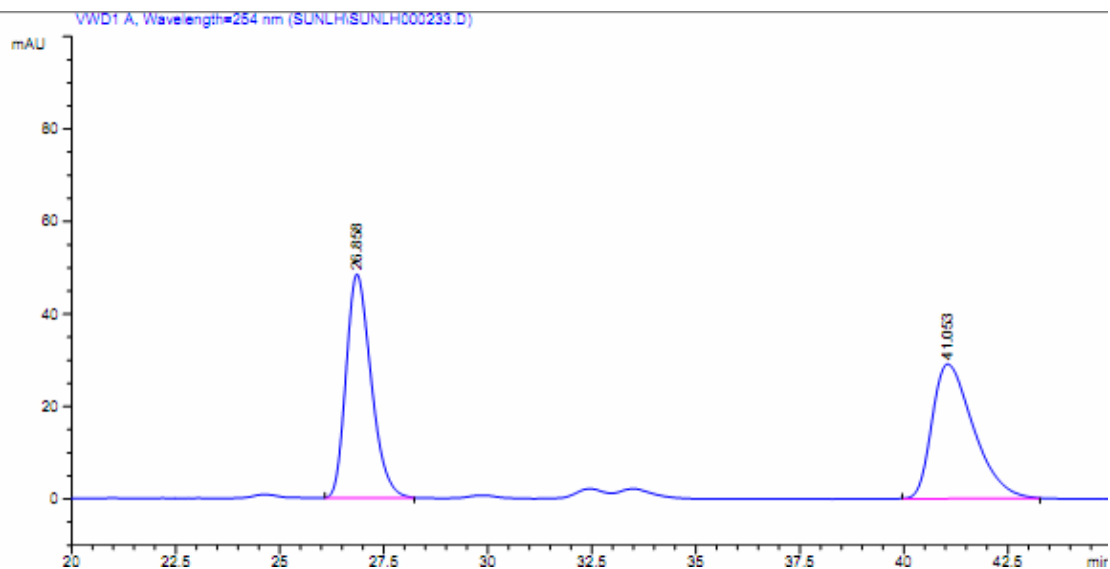
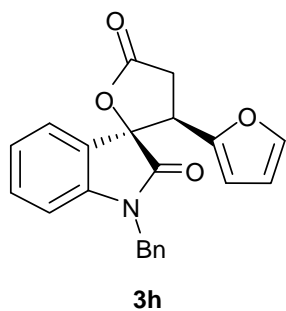
Totals : 7343.79968 250.61084



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.512	BB	0.5224	9549.57324	283.61209	96.5407
2	24.266	BB	0.5934	342.18488	8.82822	3.4593

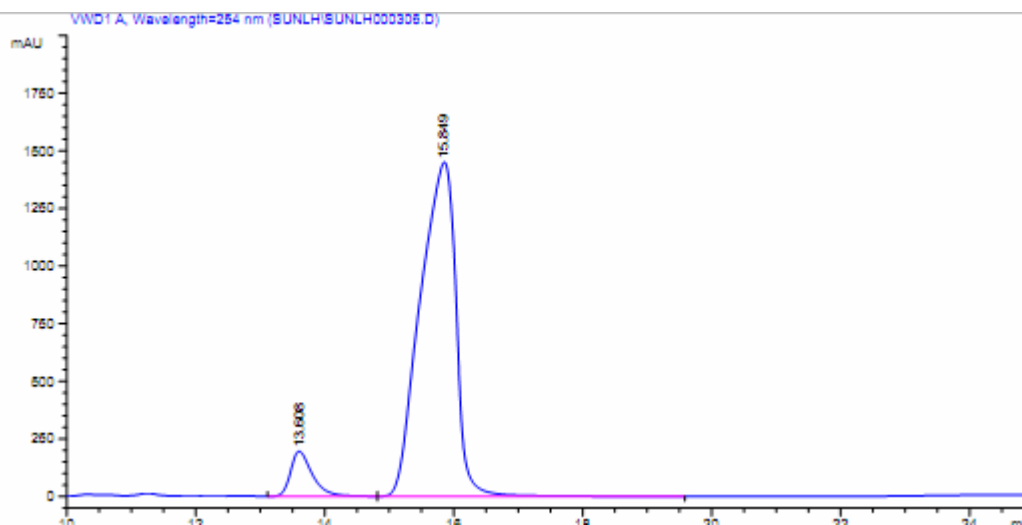
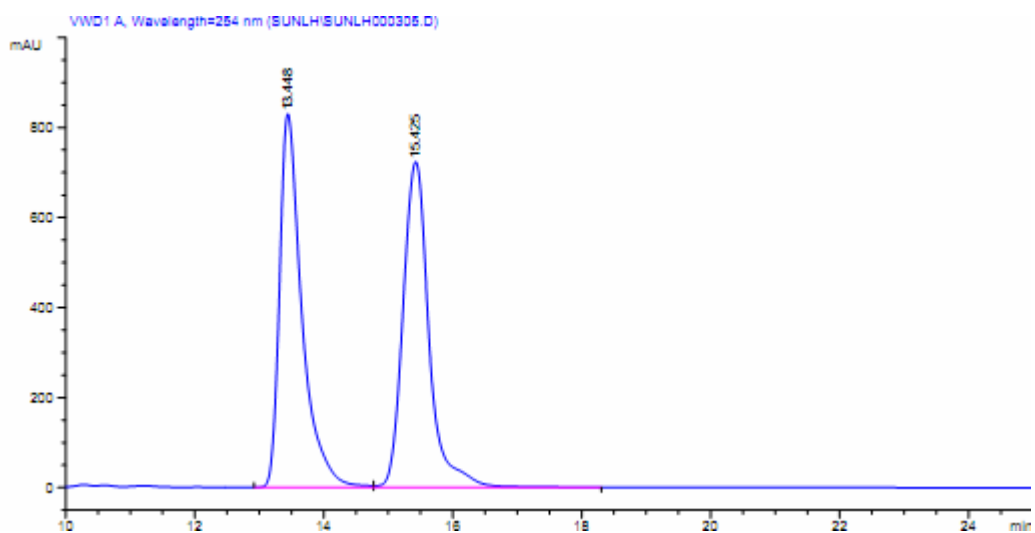
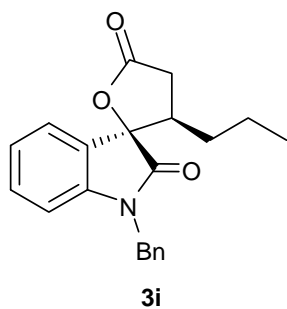
Totals : 9891.75812 292.44031



Signal 1: VWD1 A, Wavelength=254 nm

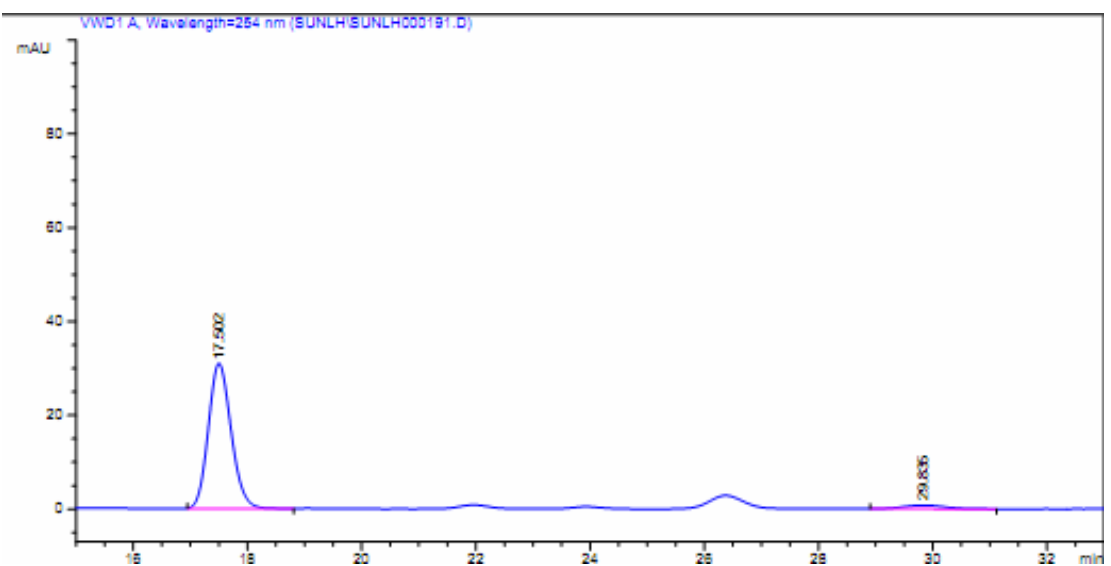
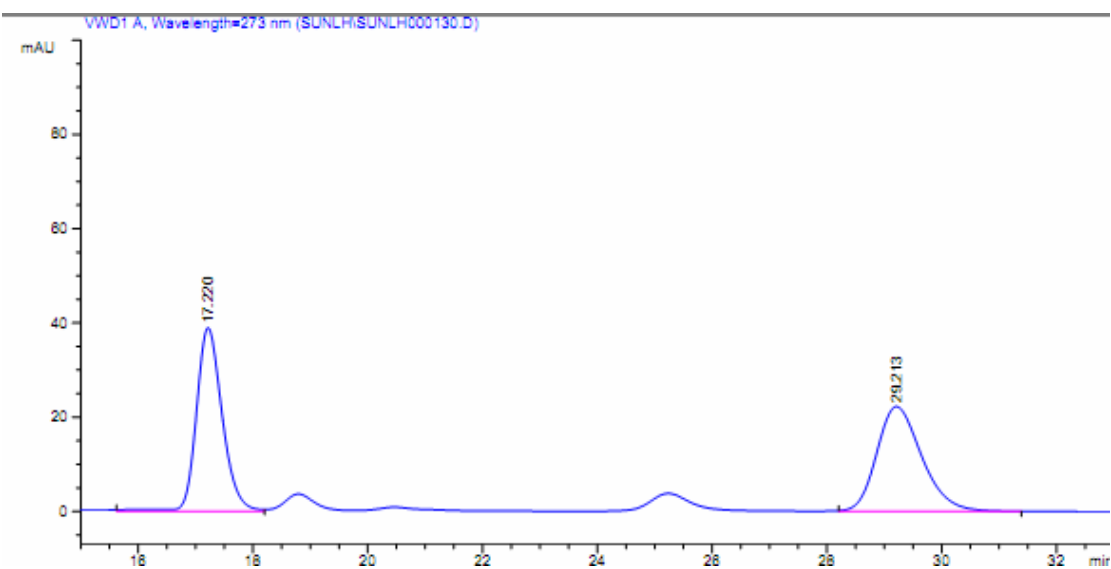
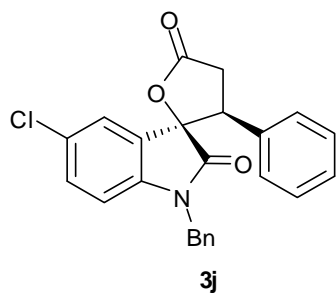
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.585	VB	0.6735	9736.73145	222.53751	93.1374
2	42.397	BB	1.1013	717.43085	10.18578	6.8626

Totals : 1.04542e4 232.72328



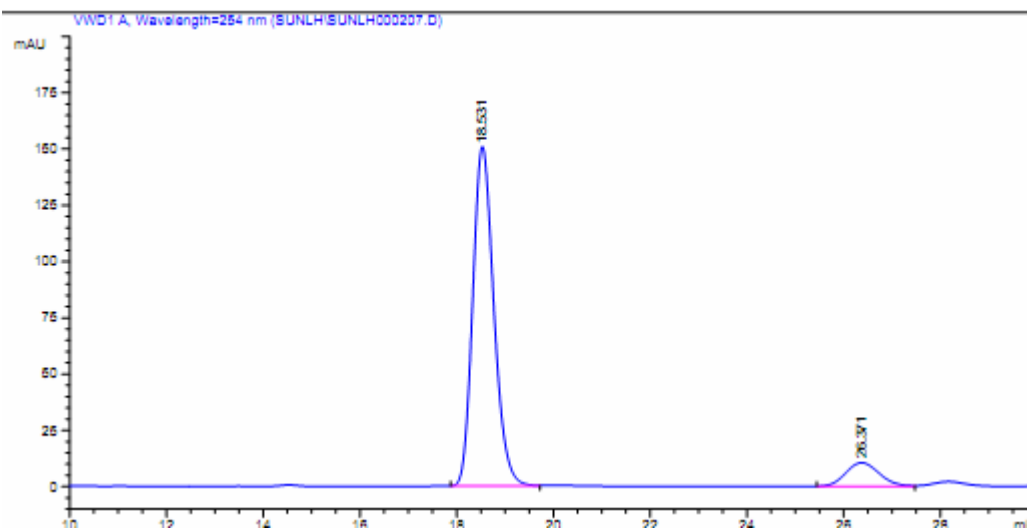
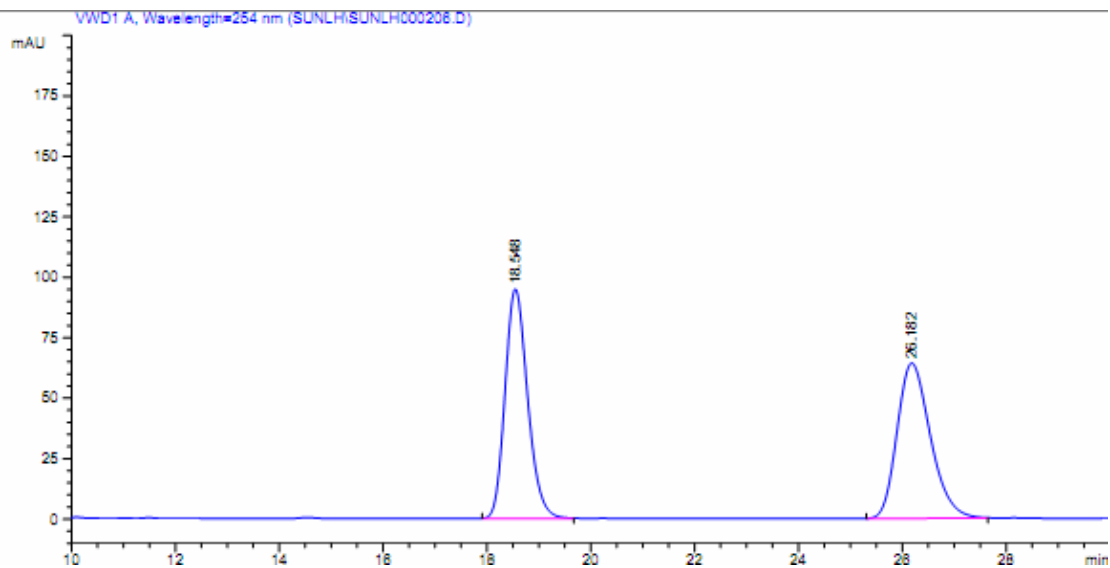
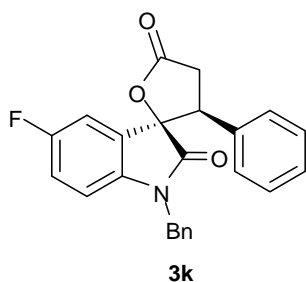
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.608	VV	0.3588	4648.39307	195.62587	7.6922
2	15.849	VB	0.6703	5.57815e4	1449.51282	92.3078
Totals :				6.04299e4	1645.13869	



Signal 1: VWD1 A, Wavelength=254 nm

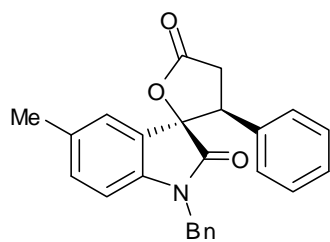
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]	Area %
1	17.502	BB	0.4290	959.75031	30.90669	95.6451	
2	29.835	BB	0.7529	39.10046	7.89934e-1	4.3549	
Totals :				997.85076		31.69562	



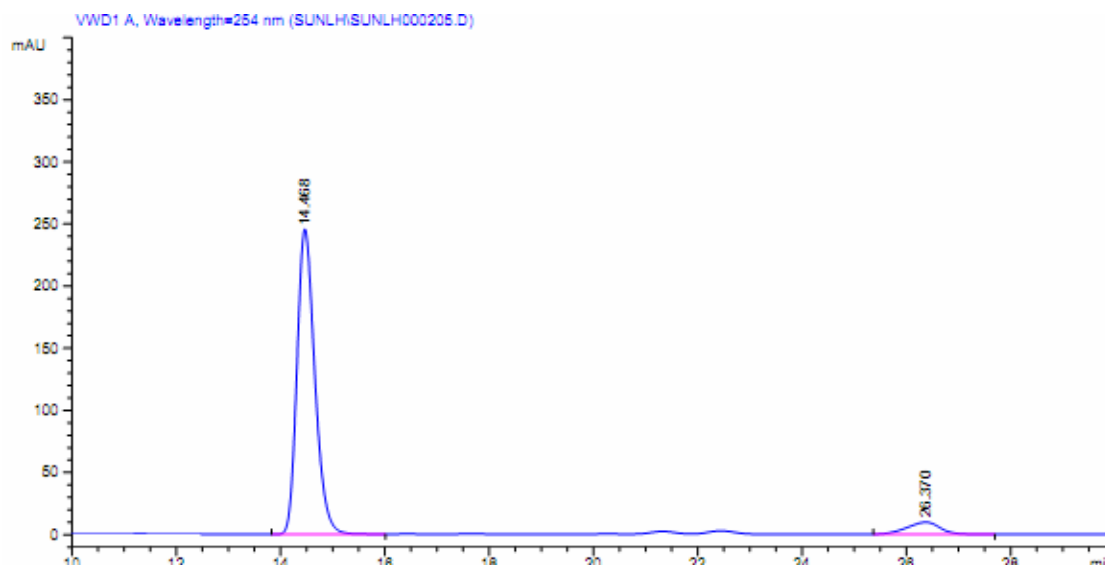
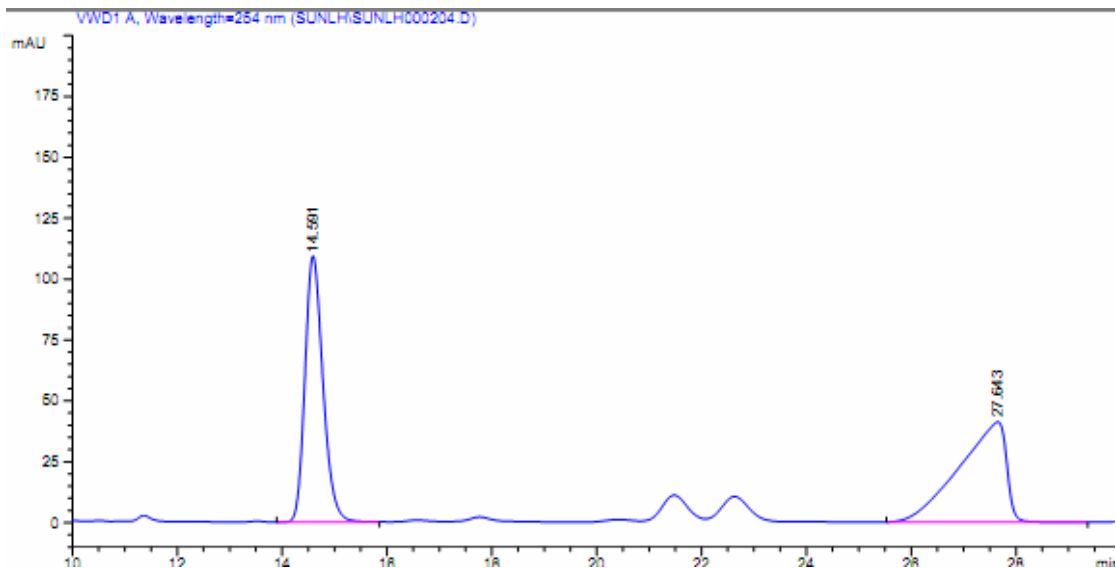
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]	Area %
1	18.531	BB	0.4660	4555.57910	90.0141	150.79636	90.0141
2	26.371	BV	0.7553	305.38345	9.9859	10.47154	9.9859

Totals : 5060.96255 161.26790



3I



Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU * s	Height [mAU]	Area %
1	14.468	VB	0.3714	5844.22803	245.06833	93.0980
2	26.370	BB	0.6938	433.26981	9.73868	6.9020

Totals : 6277.49783 254.80700

