Acetylphosphonate as a Surrogate of Acetate or Acetamide in Organocatalyzed Enantioselective Aldol Reactions

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Supporting Information

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General information

Unless otherwise noted, all reactions were carried out under nitrogen atmosphere. TLC was performed with silica gel GF_{254} precoated on plastic plates and spots were visualized with UV. ^{1}H and ^{13}C NMR spectra were recorded on a 300 MHz spectrometer (75 MHz for ^{13}C). The following abbreviations were used to designate chemical shift mutiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). Melting points were recorded in open capillaries and uncorrected. Flash column chromatography was performed on silica gel. HPLC analysis was performed on an HPLC instrument equipped with a UV-Vis detector.

Solvents used in this study were freshly distilled from an appropriate drying agent before use. Dialkyl alkanoylphosphonates were prepared according to literature.[24] *N*-Methyl,[25] *N*-benzyl,[26] and *N*-trityl[27] protected isatin derivatives were prepared as described previously. Catalysts were synthesized by following the published procedures.[18]

Experimental Procedures

General Experimental Procedure for the Aldol Reaction of Isatins and Phenylglyoxal Hydrate: A solution of catalyst **4c** or **4j** (2.3 mg, 5.0 μmol, 5.0 mol %) and *N*-tritylisatin **2a** (38.9 mg, 0.10 mmol) in THF (2.0 ml) were stirred at -15 ℃ for 15 min. Then diisopropyl acetylphosphonate **1c** (104.1 mg, 0.50 mmol) was added in one portion to the above mixture. The reaction mixture was further stirred at the above temperature for 6 h (monitored by TLC). Upon the completion of the reaction, the reaction mixture was allowed to warm to room temperature and then methanol (1.0 mL) and DBU (15.2 mg, 0.10 mmol) were added sequentially. After stirring for an additional 15 min, the volatile components were removed under reduced pressure. The residue was purified by column chromatography on silica gel (EtOAc/hexane=1:2) to afford the desired aldol product.

Experimental Procedure for the Synthesis of the Acetamide Aldol Product 7 (Scheme 1)

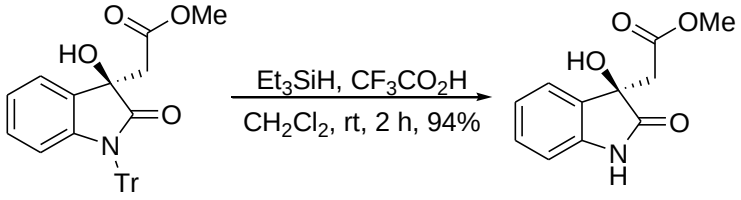
A solution of catalyst **4j** (2.3 mg, 5.0 μmol, 5.0 mol %) and *N*-tritylisatin **2a** (38.9 mg, 0.10 mmol) in THF (2.0 ml) were stirred at -15 ℃ for 15 min. Then diisopropyl acetylphosphonate **1c** (104.1 mg, 0.50 mmol) was added in one portion to the above mixture. The reaction mixture was further stirred at the above temperature for 6 h (monitored by TLC). Upon the completion of the reaction, the reaction mixture was allowed to warm to room temperature and then $MeNH_2$ solution in THF (2.0 M, 1.0 mL, 2.0 mmol) was added. After stirring for an additional 15 min, the volatile

components were removed under reduced pressure. The residue was purified by column chromatography on silica gel (EtOAc/hexane=1:2) to afford acetamide aldol product **7** (41.6 mg, 90%, 96% ee).

Experimental Procedure for the Synthesis of Pyrrolidine-2,5-dione 9 (Scheme 1)

A solution of catalyst **4j** (4.5 mg, 10.0 μmol, 10.0 mol %) and ethyl 2-oxo-4-phenylbut-3-ynoate **8** (20.2 mg, 0.10 mmol) in THF (1.0 ml) were stirred at -15 ℃ for 15 min. Then diisopropyl acetylphosphonate **1c** (104.1 mg, 0.50 mmol) was added in one portion to the above mixture. The reaction mixture was further stirred at the above temperature for 24 h (monitored by TLC). Then the reaction mixture was allowed to warm to room temperature and $MeNH_2$ solution in THF (2.0 M, 1.0 mL, 2.0 mmol) was added. After stirring for an additional 15 min, the volatile components were removed under reduced pressure. The residue was purified by column chromatography on silica gel (EtOAc/hexane=1:2) to afford the desired aldol product **9** (17.4 mg, 76% yield, 76% ee**)**.

Removing the Trityl Protecting Group: Converting the Aldol Product 3d to Compound 3a[22] (Scheme 2)

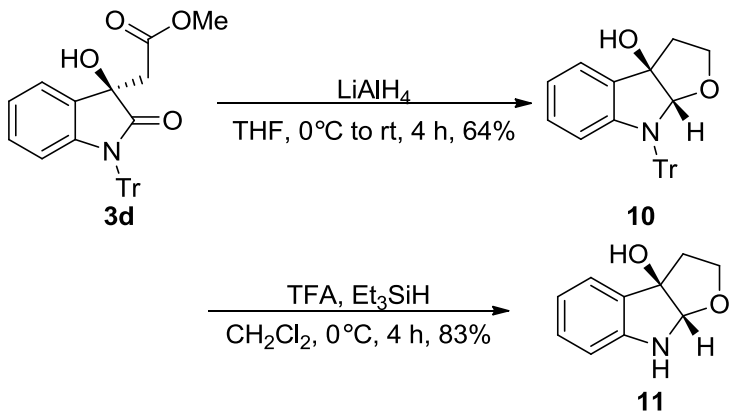


3d **3a**

Triethylsilane (64.0 μl, 0.4 mmol) and trifluoroacetic acid (1.0 mL) were added to a solution of **3d** (46.5 mg, 0.10 mmol; 95% ee) in CH_2Cl_2 (3.0 mL), and the mixture was stirred for 2 h at room temperature. The reaction was quenched with saturated aq. $NaHCO_3$ (6 mL) and extracted with CH_2Cl_2 (6 mL × 3 times). The organic layer was dried over $MgSO_4$, filtered, and concentrated under vacuum. The residue was purified by silica gel preparative TLC (EtOAc/hexane = 1/1) to afford compound **3a** as a white solid (20.5 mg, 94% yield, 96% ee).

Synthesis of Half Fragment of Madindoline A and B (compound 11, Scheme 3)

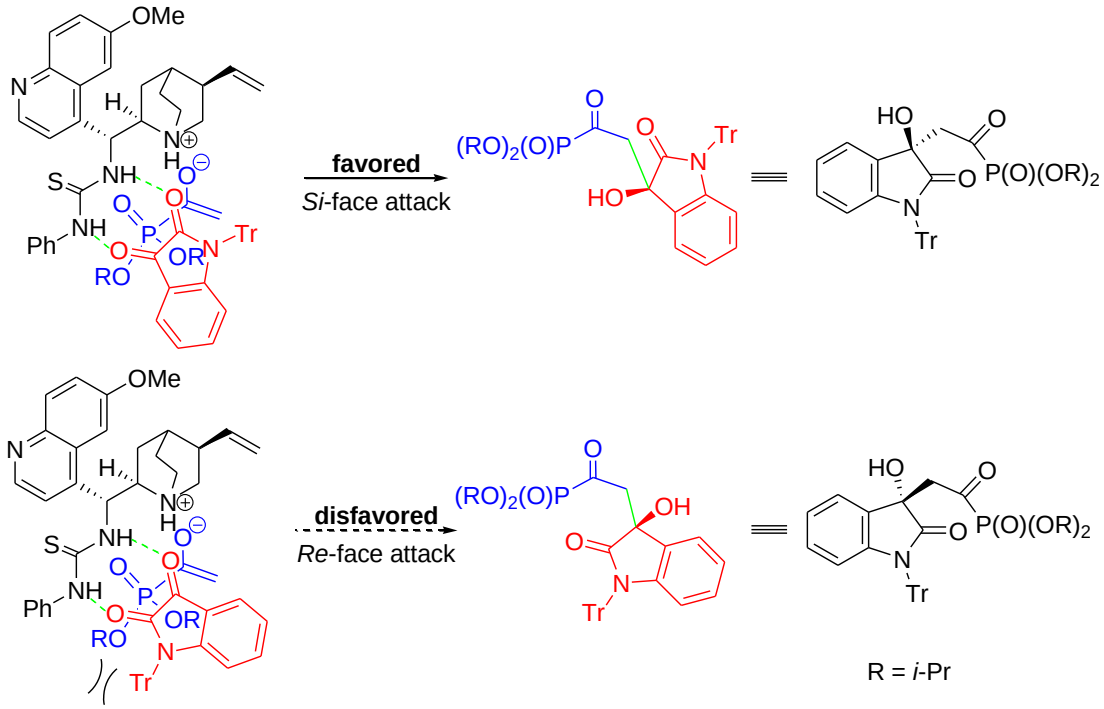
A solution of (*R*)-methyl 2-(3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (**3d**, 139.0 mg, 0.30 mmol, 95% ee) in THF (7.0 ml) was cooled to 0 ℃. To the mixture was added $LiAlH_4$-THF solution (1.0 M, 3.0 mL, 3.0 mmol) while stirring. The reation was further stirred for 1 h at 0 ℃ and then 3 h at room temperature. The reaction mixuter was quenched by adding water. The suspension so obtained was filtered through Celite. The Celite layer was washed with CH_2Cl_2. The solvent was evaporated and the crude product obtained was purified by flash column chromatography, eluting with 1:3 EtOAc/hexane, to give compound **10** as pale yellow solid (80.5 mg, 64%).



To a solution of **10** (80.5 mg, 0.19 mmol) in CH_2Cl_2 (4.0 mL) at 0 ℃ were added triethylsilane (64.0 µl, 0.40 mmol) and trifluoroacetic acid (3.0 µl, 0.040 mmol) consecutively within 30 min. The reaction mixture was stirred for 3.5 h at 0 ℃ and then quenched with $NaHCO_3$ (42.0 mg, 0.50 mmol). The solvent was removed under reduced pressure and the crude product was purified by flash column chromatography, eluting with 1:2 EtOAc/hexane, to give compound **11** as white solid (30.0 mg, 83%).

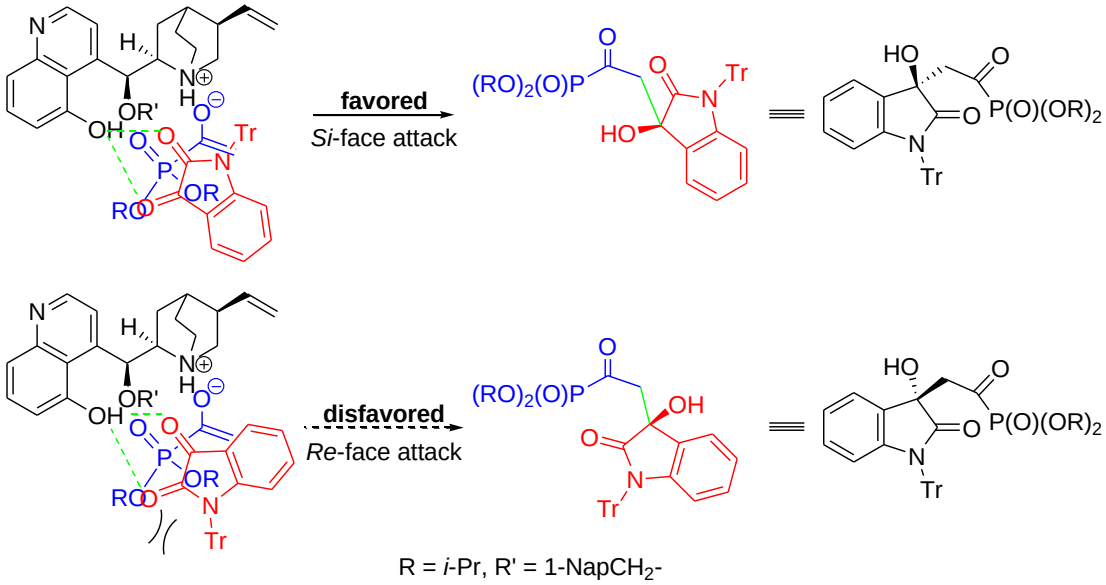
Proposed Transition State Models

The proposed transition state models for catalysts **4b** and **4j** are shown below in Scheme S-1 and Scheme S-2.



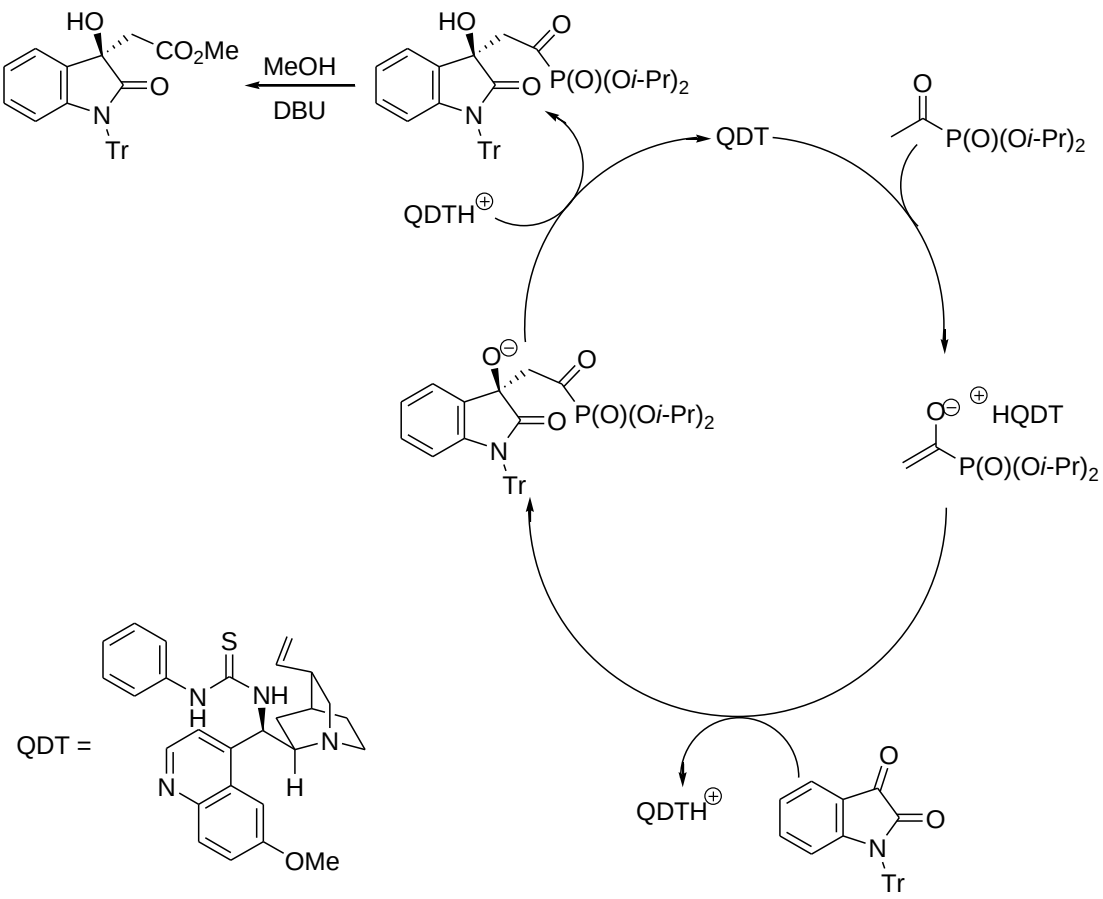
Scheme S-1: Proposed Transition State Models for Catalyst 4b

Based on these models, increasing the steric hindrance at 4-position will make the favored TS become less favored. This might be the reason why the stereoselectivity is reversed for 4-substituted isatins (Table 2 of the main text, entries 1-2).



Scheme S-2: Proposed Transition State Models for Catalyst 4j

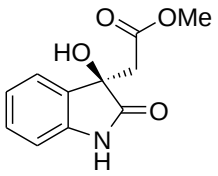
These models can explain the formation of the (*R*,*S*)-diastereomer **3r** as the major product with this catalyst (Scheme 1, equation A of the main text).



Scheme S-3: Proposed Mechanism for the Quinidine Thiourea-Catalyzed Aldol Reaction and *in-situ* Conversion of the Aldol Product to an Acetate Derivative.

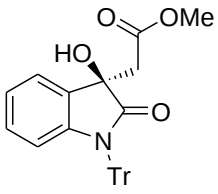
Compound Characterization Data

(*R*)-Methyl 2-(3-hydroxy-2-oxoindolin-3-yl)acetate (3a)[28]



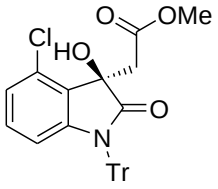
white solid, 94% yiled, m.p. 101-103 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.98 (s, 2H), 3.57 (s, 3H), 3.69 (s, 1H), 4.60 (br, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 7.07(t, *J* = 7.5 Hz, 1H), 7.27(t, *J* = 9.0 Hz, 1H), 7.38 (d, *J* = 7.8 Hz, 1H), 8.40(s, 1H); ^{13}C (75 MHz, $CDCl_3$): δ 41.1, 52.4, 74.0, 110.7, 123.3, 124.3, 129.7, 130.3, 140.6, 171.0, 178.3; ν_{max}: 1045, 1174, 1205, 1356, 1441, 1468, 1619, 1711, 3379 cm^{-1}. $[\alpha]_D^{25}$ = +120.6 (*c* 0.05, MeOH, 96% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using a ChiralCel OJ-H column (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 31.9 min, minor enantiomer: t_R = 40.7 min.

(*R*)-Methyl 2-(3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3d)



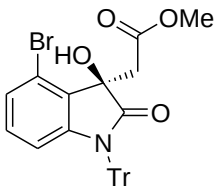
White solid; 90% yield; m.p. 172-174 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.91 (dd, *J* = 22.2, 15.3 Hz, 2H), 3.57 (s, 3H), 3.68 (s, 1H), 6.22 (d, *J* = 9 Hz, 1H), 6.81-6.89 (m, 2H), 7.11-7.26 (m, 10H), 7.36-7.39 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 42.2, 53.3, 73.4, 74.6, 116.4, 122.7, 123.2, 127.0, 127.7, 128.6, 129.3, 141.8, 143.2, 170.3, 177.7; ν_{max}: 1058, 1119, 1158, 1338, 1448, 1605, 1715, 3367 cm^{-1}. $[\alpha]_D^{25}$ = -33.2 (*c* 0.05, CH_2Cl_2, 95% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 10.7 min, minor enantiomer: t_R = 14.4 min. Anal. calcd. for $C_{30}H_{25}NO_4$: C, 77.74; H, 5.44; N, 3.02. Found: C, 77.52; H, 5.38; N, 3.01.

(*R*)-Methyl 2-(4-chloro-3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3e)



White solid; 87% yield; m.p. 200-202 °C; ^{1}H (300 MHz, $CDCl_3$): δ 3.29-3.46 (m, 3H), 3.65 (s, 3H), 6.26 (d, J = 7.5 Hz, 1H), 6.82-6.91 (m, 2H), 7.18-7.29 (m, 9H), 7.45-7.47 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 40.3, 52.2, 74.2, 75.0, 115.0, 123.8, 125.4, 127.1, 127.8, 129.4, 129.5, 130.9, 141.5, 145.6, 169.8, 177.2; ν_{max}: 1149, 1207, 1349, 1446, 1490, 1600, 1725 cm^{-1}. $[\alpha]_D^{25}$ = -45.8 (*c* 0.10, CH_2Cl_2, 96% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 9.2 min, minor enantiomer: t_R = 11.1 min. Anal. calcd. for $C_{30}H_{24}ClNO_4$: C, 72.36; H, 4.86; N, 2.81. Found: C, 71.94; H, 4.95; N, 2.79.

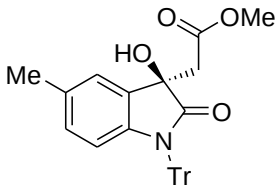
(*R*)-Methyl 2-(4-bromo-3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3f)



White solid; 92% yield; m.p. 147-149 °C; ^{1}H (300 MHz, $CDCl_3$): δ 3.32 (d, *J* = 15.0 Hz, 1H), 3.49 (d, *J* = 7.5 Hz, 1H), 3.65 (s, 3H), 6.31 (d, *J* = 8.1 Hz, 1H), 6.78 (t, *J* = 8.1 Hz, 1H), 7.06 (d, *J* = 8.1 Hz, 1H), 7.18-7.29 (m, 10H), 7.44-7.47 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 40.2, 52.2, 74.7, 75.0, 115.0, 118.7, 126.9, 127.1, 127.8,

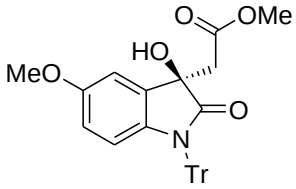
129.4, 129.5, 129.7, 141.5, 145.8, 169.7, 177.2; ν_{max}: 1139, 1206, 1259, 1443, 1490, 1582, 1728 cm^{-1}. $[\alpha]_D^{25}$ = -47.4 (*c* 0.10, CH_2Cl_2, 97% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 9.6 min, minor enantiomer: t_R = 12.3 min. Anal. calcd. for $C_{30}H_{24}BrNO_4$: C, 66.43; H, 4.46; N, 2.58. Found: C, 66.20; H, 4.58; N, 2.57.

(*R*)-Methyl 2-(3-hydroxy-5-methyl-2-oxo-1-tritylindolin-3-yl)acetate (3g)



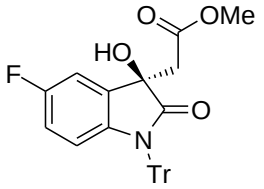
White solid; 92% yield; m.p. 156-159 °C; ^{1}H (300 MHz, $CDCl_3$): 2.18 (s, 3H), δ 2.94 (dd, *J* = 24.3, 15.6 Hz, 2H), 3.62 (s, 3H), 3.67 (s, 1H), 6.13 (d, *J* = 8.4 Hz, 1H), 6.67 (d, *J* = 8.4 Hz, 1H), 7.10-7.22 (m, 10H), 7.40-7.42 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 21.0, 42.3, 52.2, 73.5, 74.5, 116.1, 123.9, 126.9, 127.7, 129.1, 129.3, 132.3, 140.8, 141.9, 170.2, 177.8; ν_{max}: 1060, 1196, 1327, 1436, 1486, 1597, 1711, 3368 cm^{-1}. $[\alpha]_D^{25}$ = -59.7 (*c* 0.11, CH_2Cl_2, 94% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 9.4 min, minor enantiomer: t_R = 12.3 min. Anal. calcd. for $C_{31}H_{27}NO_4$: C, 77.97; H, 5.70; N, 2.93. Found: C, 77.71; H, 5.70; N, 3.04.

(*R*)-Methyl 2-(3-hydroxy-5-methoxy-2-oxo-1-tritylindolin-3-yl)acetate (3h)



White solid; 90% yield; m.p. 176-178 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.97 (dd, *J* = 21.9, 15.3 Hz, 2H), 3.69 (d, *J* = 9.3 Hz, 6H), 6.20 (d, *J* = 9.0 Hz, 1H), 6.46 (dd, *J* = 8.7 Hz, 2.7 Hz, 1H), 6.93 (d, *J* = 2.7 Hz, 1H), 7.17-7.28 (m, 8H), 7.44-7.47 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 42.4, 52.3, 55.7, 73.7, 74.5, 109.6, 113.7, 117.0, 127.0, 127.7, 129.3, 130.5, 136.3, 141.8, 155.6, 170.2, 177.6; ν_{max}: 1029, 1160, 1273, 1315, 1433, 1486, 1599, 1716, 3060 cm^{-1}. $[\alpha]_D^{25}$ = -53.1 (*c* 0.10, CH_2Cl_2, 94% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 12.6 min, minor enantiomer: t_R = 16.6 min. Anal. calcd. for $C_{31}H_{27}NO_5$: C, 75.44; H, 5.51; N, 2.84. Found: C, 75.19; H, 5.49; N, 2.86.

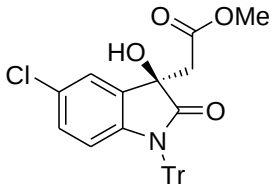
(*R*)-Methyl 2-(5-fluoro-3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3i)



White solid; 88% yield; m.p. 194-196 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.89 (dd, *J* = 18.0, 15.6 Hz, 2H), 3.59 (s, 3H), 3.67 (s, 1H), 6.15 (dd, *J* = 9.0, 4.2 Hz, 1H), 6.54 (td, *J* = 11.7, 8.7, 2.7 Hz, 1H), 6.98 (dd, *J* = 7.2, 2.7 Hz, 1H), 7.09-7.20 (m, 9H), 7.33-7.36 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 42.3, 52.5, 73.6, 74.9, 111.2, 111.5, 115.1, 115.4, 117.3, 117.4, 127.3, 128.0, 129.4, 131.0, 131.1, 139.2, 141.7, 157.3, 160.5, 170.2, 177.7; ν_{max}: 1064, 1163, 1262, 1448, 1476, 1611, 1726, 3059 cm^{-1}. $[\alpha]_D^{25}$ = -35.8 (*c* 0.11, CH_2Cl_2, 95% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel

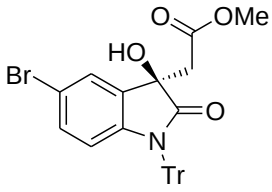
OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 9.4 min, minor enantiomer: t_R = 13.8 min. Anal. calcd. for $C_{30}H_{24}FNO_4$: C, 74.83; H, 5.02; N, 2.91. Found: C, 74.32; H, 5.55; N, 2.72.

(*R*)-Methyl 2-(5-chloro-3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3j)



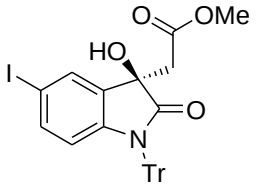
White solid; 84% yield; m.p. 184-185 °C; ^{1}H (300 MHz, $CDCl_3$): δ 3.12 (dd, *J* = 21.6, 15.6 Hz, 2H), 3.78 (s, 3H), 6.35 (d, *J* = 8.7 Hz, 1H), 7.00 (d, *J* = 8.7 Hz, 1H), 7.31-7.42 (m, 10H), 7.54-7.56 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 40.3, 52.2, 74.2, 75.0, 115.0, 123.8, 125.4, 127.1, 127.8, 129.4, 129.5, 130.9, 141.5, 145.6, 169.8, 177.2; ν_{max}: 1033, 1066, 1184, 1300, 1471, 1594, 1726, 3062, 3380 cm^{-1}. $[\alpha]_D^{25}$ = -26.9 (*c* 0.06, MeOH, 95% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 8.8 min, minor enantiomer: t_R = 13.0 min. Anal. calcd. for $C_{30}H_{24}ClNO_4$: C, 72.36; H, 4.86; N, 2.81. Found: C, 72.37; H, 5.01; N, 2.78.

(*R*)-Methyl 2-(5-bromo-3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3k)



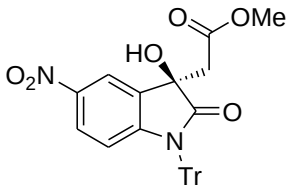
White solid; 89% yield; m.p. 201-203 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.99 (dd, *J* = 20.1, 15.6 Hz, 2H), 3.69 (s, 3H), 3.76 (s, 1H), 6.18 (d, *J* = 8.7 Hz, 1H), 7.01 (d, *J* = 8.7 Hz, 1H), 7.19-7.28 (m, 10H), 7.42-7.45 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 42.1, 52.4, 73.3, 74.8, 115.8, 117.8, 126.6, 127.2, 127.8, 129.2, 131.3, 131.5, 141.4, 142.4, 170.0, 177.2; ν_{max}: 1035, 1076, 1182, 1266, 1300, 1444, 1471, 1606, 1719, 3386 cm^{-1}. $[\alpha]_D^{25}$ = -35.8 (*c* 0.10, CH_2Cl_2, 93% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 8.9 min, minor enantiomer: t_R = 13.5 min. Anal. calcd. for $C_{30}H_{24}BrNO_4$: C, 66.43; H, 4.43; N, 2.58. Found: C, 66.21; H, 4.62; N, 2.43.

(*R*)-Methyl 2-(3-hydroxy-5-iodo-2-oxo-1-tritylindolin-3-yl)acetate (3l)



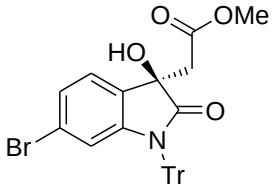
White solid; 91% yield; m.p. 127-129 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.97 (dd, *J* = 21.6, 15.6 Hz, 2H), 3.68 (s, 3H), 3.74 (s, 1H), 6.06 (d, *J* = 8.7 Hz, 1H), 7.17-7.27 (m, 11H), 7.40-7.43 (m, 6H), 7.60 (s, 1H); ^{13}C (75 MHz, $CDCl_3$): δ 42.2, 52.5, 73.3, 74.9, 86.1, 118.4, 127.3, 128.0, 129.4, 131.7, 132.3, 137.6, 141.6, 143.4, 170.1, 177.2; ν_{max}: 1032, 1071, 1129, 1182, 1263, 1313, 1470, 1602, 1718, 3386 cm^{-1}. $[\alpha]_D^{25}$ = -65.4 (*c* 0.10, MeOH, 94% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 8.2 min, minor enantiomer: t_R = 12.4 min. Anal. calcd. for $C_{30}H_{24}INO_4$: C, 61.13; H, 4.10; N, 2.38. Found: C, 61.26; H, 4.29; N, 2.33.

(*R*)-Methyl 2-(3-hydroxy-5-nitro-2-oxo-1-tritylindolin-3-yl)acetate (3m)



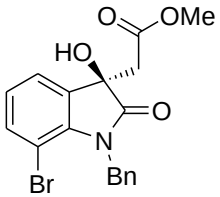
White solid; 88% yield; m.p. 228-229 °C; ^{1}H (300 MHz, $CDCl_3$): δ 3.00 (s, 2H), 3.60 (s, 4H), 3.74 (s, 1H), 6.35 (d, *J* = 9.0 Hz, 1H), 7.12-7.23 (m, 9H), 7.33-7.37 (m, 6H), 7.78 (dd, *J* = 9.0, 6.3 Hz, 1H), 8.11 (d, *J* = 9.0, 2.4 Hz, 1H); ^{13}C (75 MHz, $CDCl_3$): δ 41.8, 52.5, 72.8, 75.4, 115.0, 119.1, 125.1, 127.4, 128.0, 129.2, 130.2, 141.0, 143.1, 149.4, 169.8, 177.7; ν_{max}: 1036, 1078, 1111, 1163, 1183, 1217, 1271, 1332, 1419, 1445, 1475, 1513, 1613, 1727, 3402 cm^{-1}. $[\alpha]_D^{25}$ = -56.6 (*c* 0.09, CH_2Cl_2, 84% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralCel OD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 12.8 min, minor enantiomer: t_R = 20.2 min. Anal. calcd. for $C_{30}H_{24}N_2O_6$: C, 70.86; H, 4.76; N, 5.51. Found: C, 70.61; H, 4.96; N, 5.55.

(*R*)-Methyl 2-(6-bromo-3-hydroxy-2-oxo-1-tritylindolin-3-yl)acetate (3n)



White solid; 92% yield; m.p. 220-223 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.97 (dd, *J* = 20.7, 15.3 Hz, 2H), 3.66 (s, 3H), 3.72 (s, 1H), 7.09 (d, *J* = 7.5 Hz, 2H), 7.20-7.30 (m, 10H), 7.42-7.44 (m, 7H); ^{13}C (75 MHz, $CDCl_3$): δ 42.0, 52.4, 73.0, 74.9, 119.4, 122.4, 124.5, 125.6, 127.2, 127.9, 128.2, 129.2, 144.3, 144.6, 170.0, 177.5; ν_{max}: 1045, 1122, 1181, 1325, 1443, 1474, 1599, 1708, 3425 cm^{-1}. $[\alpha]_D^{25}$ = -68.3 (*c* 0.11, CH_2Cl_2, 93% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralPak AD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 12.0 min, minor enantiomer: t_R = 16.0 min. Anal. calcd. for $C_{30}H_{24}BrNO_4$: C, 66.43; H, 4.46; N, 2.58. Found: C, 66.28; H, 4.56; N, 2.66.

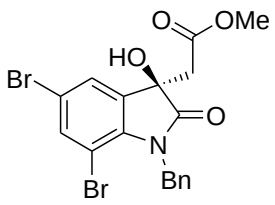
(*R*)-Methyl 2-(1-benzyl-7-bromo-3-hydroxy-2-oxoindolin-3-yl)acetate (3o)



White solid; 91% yield; m.p. 153-155 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.86 (dd, *J* = 26.4, 15.9 Hz, 2H), 3.56 (s, 3H), 4.37 (s, 1H), 5.23 (s, 2H), 6.80 (t, *J* = 7.8 Hz, 1H), 7.08-7.26 (m, 7H); ^{13}C (75 MHz, $CDCl_3$): δ 41.2, 44.8, 52.5, 72.8, 103.1, 123.1, 124.7, 126.3, 127.2, 128.6, 132.4, 136.1, 136.9, 140.3, 170.6, 177.1; ν_{max}: 1006, 1070, 1133, 1169, 1202, 1337, 1435, 1582, 1608, 1706, 1730, 3309 cm^{-1}. $[\alpha]_D^{25}$ = +28.3 (*c* 0.11, CH_2Cl_2, 93% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralPak AD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 23.4 min, minor enantiomer: t_R = 31.8 min. Anal. calcd. for $C_{18}H_{16}BrNO_4$: C, 55.40; H, 4.13; N, 3.59. Found: C, 55.42; H, 4.09; N, 3.60.

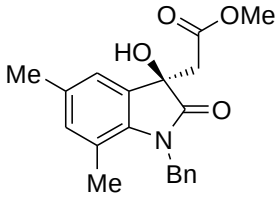
(*R*)-Methyl 2-(1-benzyl-5,7-dibromo-3-hydroxy-2-oxoindolin-3-yl)acetate (3p)

White solid; 90% yield; m.p. 121-123 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.91 (dd, *J* = 21.9, 16.2 Hz, 2H), 3.65 (s, 3H), 4.42 (s, 1H), 5.26 (s, 2H), 7.11-7.25 (m, 5H), 7.40 (d, *J* = 2.1 Hz, 1H), 7.48 (d, *J* = 1.8 Hz, 1H); ^{13}C (75 MHz, $CDCl_3$): δ 41.0, 44.9, 52.7, 72.8,



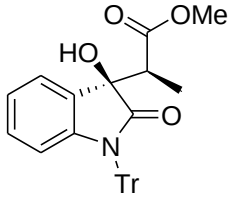
103.6, 116.4, 126.2, 126.5, 127.4, 128.7, 133.9, 136.6, 137.9, 139.6, 170.4, 176.6; ν_{max}: 1020, 1075, 1146, 1177, 1338, 1449, 1572, 1601, 1706, 1744, 3359 cm^{-1}. $[\alpha]_D^{25}$ = +18.9 (*c* 0.11, CH_2Cl_2, 93% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralPak AD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 16.2 min, minor enantiomer: t_R = 21.5 min. Anal. calcd. for $C_{18}H_{15}Br_2NO_4$: C, 46.08; H, 3.22; N, 2.83. Found: C, 46.21; H, 3.32; N, 2.83.

(*R*)-Methyl 2-(1-benzyl-3-hydroxy-5,7-dimethyl-2-oxoindolin-3-yl)acetate (3q)



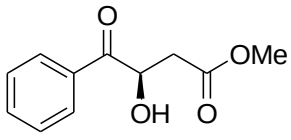
White solid; 82% yield; m.p. 149-150 °C; ^1H (300 MHz, $CDCl_3$): δ 2.15 (d, *J* = 22.2 Hz), 2.90 (dd, *J* = 34.5, 15.6 Hz, 2H), 3.63 (s, 3H), 4.32 (s, 1H), 5.05 (s, 2H), 6.71 (s, 1H), 7.00 (s, 1H,) 7.08-7.24 (m, 5H),; ^{13}C (75 MHz, $CDCl_3$): δ 18.9, 21.0, 41.4, 45.3, 52.4, 73.0, 120.3, 122.5, 125.8, 127.3, 129.0, 130.0, 133.1, 134.6, 137.3, 138.2, 171.0, 177.4; ν_{max}: 1046, 1161, 1205, 1318, 1358, 1438, 1485, 1604, 1670, 1736, 3271 cm^{-1}. $[\alpha]_D^{25}$ = +12.2 (*c* 0.11, CH_2Cl_2, 94% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using ChiralPak AD-H (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 21.8 min, minor enantiomer: t_R = 31.3 min. Anal. calcd. for $C_{20}H_{21}NO_4$: C, 70.78; H, 6.24; N, 4.13. Found: C, 70.64; H, 6.32; N, 4.26.

(*S*)-Methyl 2-[(*R*)-3-hydroxy-2-oxo-1-tritylindolin-3-yl]propanoate (3r)



m.p. 92-95 °C; ^1H (300 MHz, $CDCl_3$): δ 0.92 (d, *J* = 7.2 Hz, 3H), 2.96 (dd, *J* = 14.4, 7.2 Hz, 1H), 3.61 (s, 3H), 4.58 (s, 1H), 6.19 (d, *J* = 6.9 Hz, 1H), 6.80-6.84 (m, 2H,) 7.05-7.20 (m, 9H), 7.32-7.35 (m, 6H); ^{13}C (75 MHz, $CDCl_3$): δ 11.4, 45.9, 52.6, 74.7, 116.2, 122.4, 122.8, 123.4, 126.9, 127.7, 128.4, 128.9, 129.2, 141.9, 143.7, 173.7, 177.6; ν_{max}: 1002, 1183, 1310, 1448, 1607, 1727, 2950 cm^{-1}. $[\alpha]_D^{25}$ = -20.7 (*c* 0.16, CH_2Cl_2, dr: 80:20, 94% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using a ChiralPak AD-H column (hexanes/*i*-PrOH 81:19 at 0.7 mL/min), major diastereomer: t_R = 21.8 min (major enantiomer), t_R = 25.7 min (minor enantiomer); minor diastereomer: t_R = 18.8 min (major enantiomer), t_R = 15.1 min (minor enantiomer). Anal. calcd. for $C_{31}H_{27}NO_4$: C, 77.97; H, 5.70; N, 2.93. Found: C, 78.02; H, 5.68; N, 2.95.

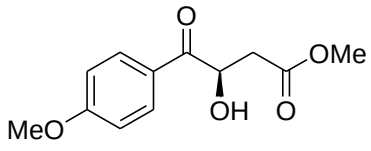
(*R*)-methyl 3-hydroxy-4-oxo-4-phenylbutanoate (6a)



Yellow oil; 61% yield; ^1H (300 MHz, $CDCl_3$): δ 2.42 (dd, *J* = 15.9, 7.8 Hz, 1H), 2.69 (dd, *J* = 15.9, 3.3 Hz, 1H), 3.52 (s, 3H), 3.80 (br, 1H), 5.53 (br, 1H), 7.30 (t, *J* = 7.8 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.74 (d, *J* = 6.9 Hz, 1H); ^{13}C (75 MHz, $CDCl_3$): δ 40.5, 52.3, 70.4, 128.7, 129.0, 133.3, 134.2, 170.8, 199.8; ν_{max}: 1099, 1165, 1264, 1438, 1597, 1683, 1733, 3461 cm^{-1}. $[\alpha]_D^{25}$ = -10.6 (*c* 0.10, CH_2Cl_2, 87% ee). Enantiomeric excess of the product was determined by chiral stationary phase

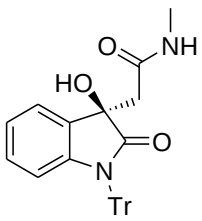
Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using a ChiralCel OD-H column (hexanes/*i*-PrOH 92:8 at 1.0 mL/min), major enantiomer: t_R = 18.9 min, minor enantiomer: t_R = 22.7 min. Anal. calcd. for $C_{11}H_{12}O_4$: C, 63.45; H, 5.81. Found: C, 63.35; H, 5.83.

(*R*)-methyl 3-hydroxy-4-oxo-4-(4-methoxyphenyl)butanoate (6b)



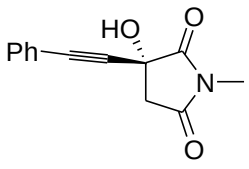
Brown oil; 66% yield; ^{1}H (300 MHz, $CDCl_3$): δ 2.50 (dd, *J* = 15.9, 8.4 Hz, 1H), 2.79 (dd, *J* = 15.9, 3.3 Hz, 1H), 3.66 (s, 3H), 3.81 (s, 3H), 5.33 (dd, *J* = 8.4, 3.3 Hz, 1H), 6.90 (d, *J* = 9.0 Hz, 2H), 7.86 (d, *J* = 9.0 Hz, 1H); ^{13}C (75 MHz, $CDCl_3$): δ 41.2, 52.6, 56.0, 70.1, 114.5, 126.0, 131.3, 164.5, 171.2, 198.2; ν_{max}: 784, 985, 1107, 1168, 1244, 1376, 1600, 1738, 2981 cm^{-1}. $[\alpha]_D^{25}$ = 42.5 (*c* 0.04, CH_2Cl_2, 84% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using a ChiralPak ID column (hexanes/*i*-PrOH 85:15 at 1.0 mL/min), major enantiomer: t_R = 79.9 min, minor enantiomer: t_R = 60.9 min.

(*R*)-2-(3-hydroxy-2-oxo-1-tritylindolin-3-yl)-*N*-methylacetamide (7)



White solid; 90% yield; m.p. 218-220 °C; ^{1}H (300 MHz, $CDCl_3$): δ 2.43 (d, *J* = 14.7 Hz, 1H), 2.59-2.64 (m, 4H), 5.09 (s, 1H), 6.18 (d, *J* = 6.6 Hz, 1H), 6.82 (d, *J* = 3.6 Hz, 1H), 7.01-7.33 (m, 15H); ^{13}C (75 MHz, $CDCl_3$): δ 26.4, 42.9, 74.2, 74.4, 116.1, 122.8, 123.5, 126.9, 127.7, 128.4, 129.1, 130.1, 141.7, 142.5, 170.2, 178.1; ν_{max}: 1002, 1033, 1154, 1314, 1449, 1534, 1597, 1649, 1670, 1720, 3418, 3639 cm^{-1}. $[\alpha]_D^{25}$ = -0.94 (*c* 0.10, CH_2Cl_2, 96% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using a ChiralPak AD-H column (hexanes/*i*-PrOH 85:15 at 1.0 mL/min), major enantiomer: t_R = 10.3 min, minor enantiomer: t_R = 9.0 min. Anal. calcd. for $C_{30}H_{26}N_2O_3$: C, 77.90; H, 5.67; N, 6.06. Found: C, 77.70; H, 6.27; N, 5.58.

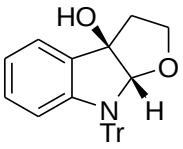
(*R*)- 3-Hydroxy-1-methyl-3-(phenylethynyl)pyrrolidine-2,5-dione (9)



Yellow oil; 76% yield; ^{1}H (500 MHz, $CDCl_3$): δ 3.03 (d, *J* = 21.5 Hz, 4H), 3.23 (d, *J* = 18.5 Hz, 1H), 3.81 (br, 1H), 5.53 (br, 1H), 7.23-7.38 (m, 5H); ^{13}C (125 MHz, $CDCl_3$): δ 25.7, 44.8, 68.7, 85.4, 87.6, 121.1 128.6, 129.7, 132.2, 173.3, 175.6; ν_{max}: 686, 755, 993, 1109, 1273, 1380, 1437, 1689, 1786, 2228, 2202 cm^{-1}. $[\alpha]_D^{25}$ = 20.0 (*c* 0.06, CH_2Cl_2, 76% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using a ChiralPak IB column (hexanes/*i*-PrOH 90:10 at 1.0 mL/min), major enantiomer: t_R = 11.8 min, minor enantiomer: t_R = 14.5 min.

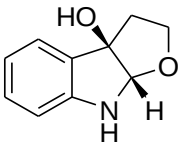
(3a*R*,8a*S*)-8-Trityl-3,3a,8,8a-tetrahydro-2*H*-furo[2,3-*b*]indol-3a-ol (10)

Pale yellow solid; 64% yield; ^{1}H (300 MHz, $CDCl_3$): δ 2.43-2.49 (m, 2H), 3.71 (ddd, *J* = 15.3, 9.6, 4.8 Hz, 1H), 4.00 (ddd, *J* = 8.7, 6.6, 2.1 Hz, 1H), 5.30 (s, 1H), 5.82 (s, *J*



= 8.1 Hz, 1H), 6.68 (t, *J* = 6.6 Hz, 1H), 6.78 (t, *J* = 7.8 Hz, 1H), 7.22-7.41 (m, 16H); ^{13}C (75 MHz, $CDCl_3$): δ 40.1, 67.4, 75.8, 86.4, 101.7, 114.5, 118.8, 123.2, 127.0, 129.0, 130.6, 131.3, 143.2, 150.1; ν_{max}: 1012, 1107, 1177, 1370, 1444, 1478, 1602, 1711 cm^{-1}. $[\alpha]_D^{25}$ = -48.9 (*c* 0.11, CH_2Cl_2). Anal. calcd. for $C_{20}H_{21}NO_4$: C, 83.03; H, 6.01; N, 3.34. Found: C, 82.81; H, 6.42; N, 3.31.

(3a*R*,8a*S*)- 3,3a,8,8a-Tetrahydro-2*H*-furo[2,3-*b*]indol-3a-ol (11)[23a,29]



White solid; 83% yield; ^{1}H (300 MHz, $CDCl_3$): δ 2.22-2.28 (m, 1H), 2.33-2.43 (m, 1H), 3.54-3.62 (m, 3H), 3.94-4.00 (m, 1H), 5.31 (s, 1H), 6.53 (d *J* = 7.8 Hz, 1H), 6.73 (t, *J* = 7.8 Hz, 1H), 7.08 (t, *J* = 7.8 Hz, 1H) , 6.53 (d *J* = 7.2 Hz, 1H),; ^{13}C (75 MHz, $CDCl_3$): δ 41.1, 67.5, 89.5, 99.5, 109.6, 119.6, 124.2, 130.1, 130.4, 149.5; $[\alpha]_D^{25}$ = -111.0 (*c* 0.10, CH_2Cl_2, 94% ee). Enantiomeric excess of the product was determined by chiral stationary phase HPLC analysis using an AD-H (hexanes: *i*-PrOH 81:19 at 0.7 mL/min), major enantiomer: t_R = 26.1 min, minor enantiomer: t_R = 21.6 min.

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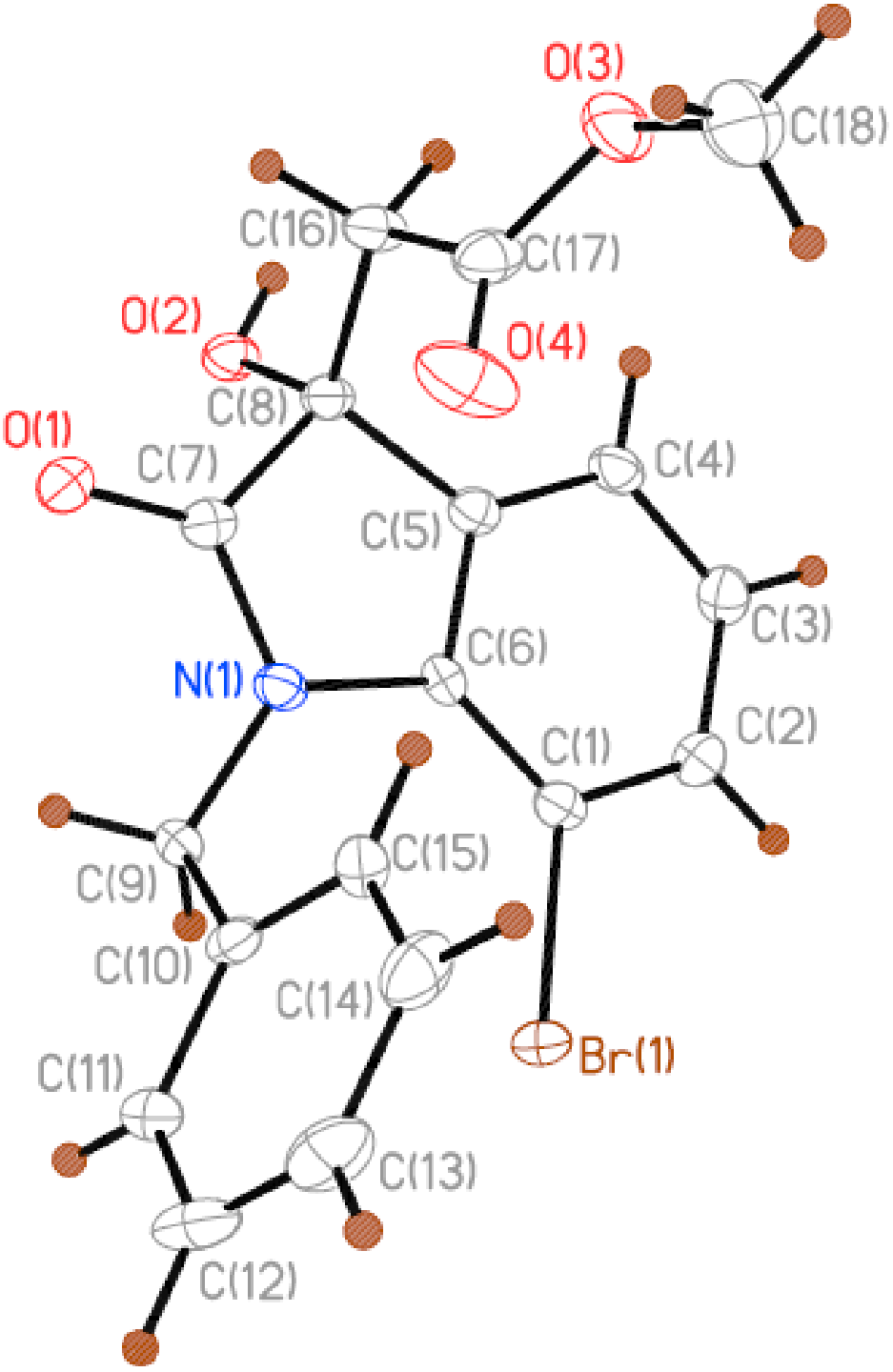


Figure S-1. ORTEP Drawing of Compound 3o.

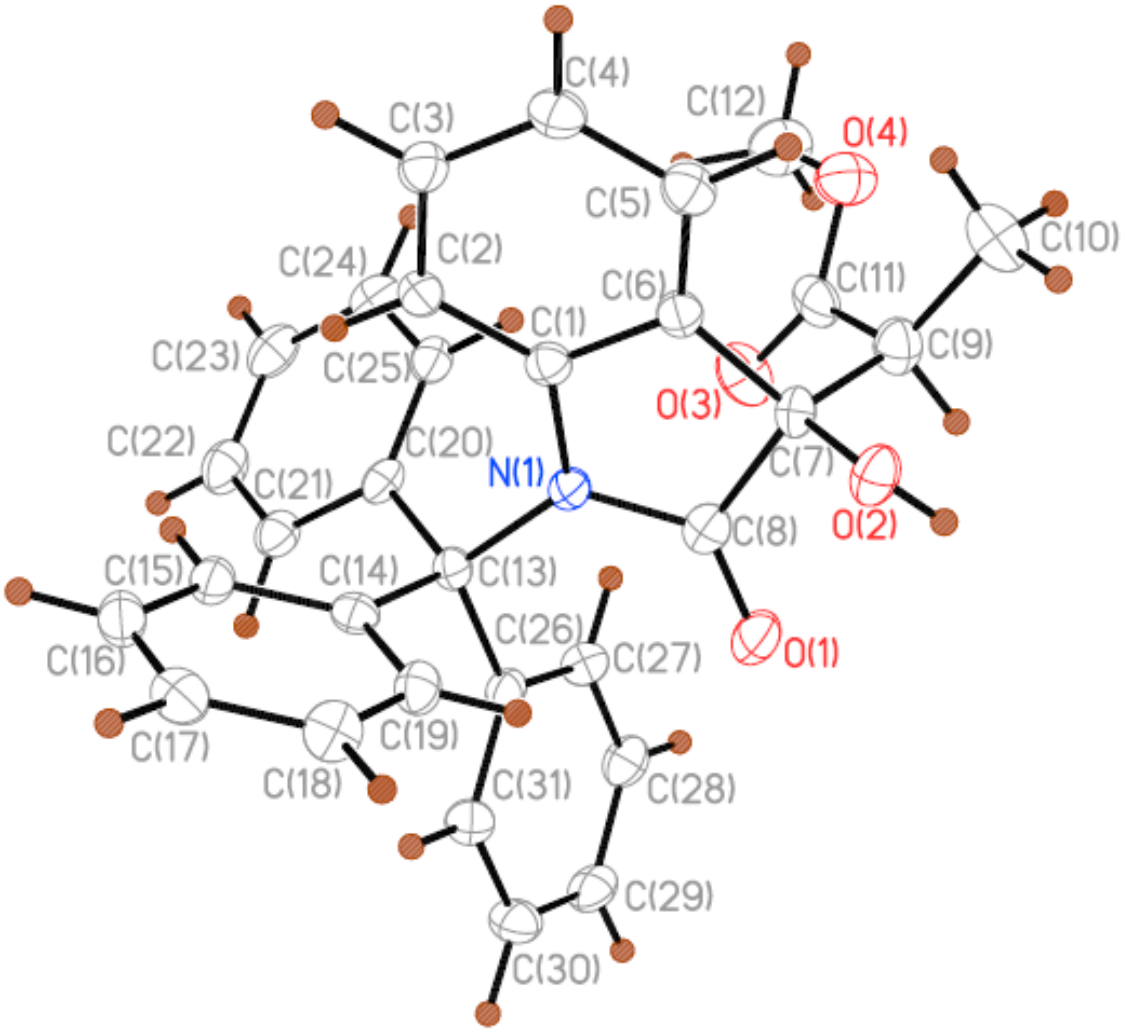
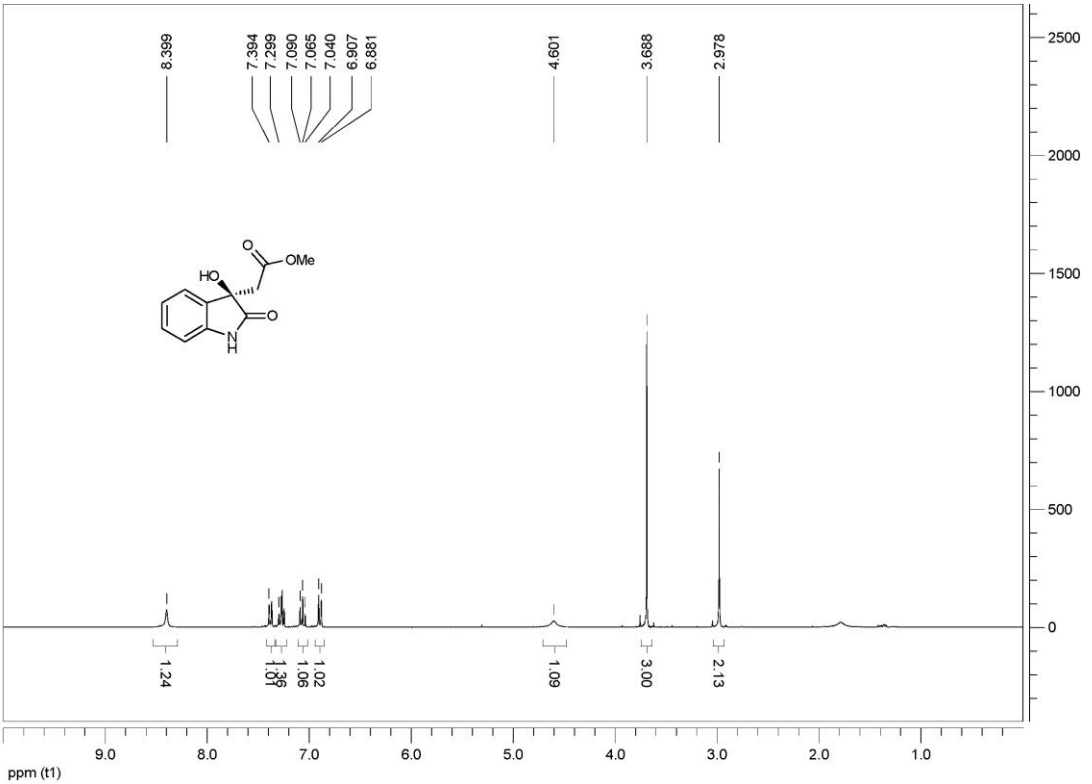
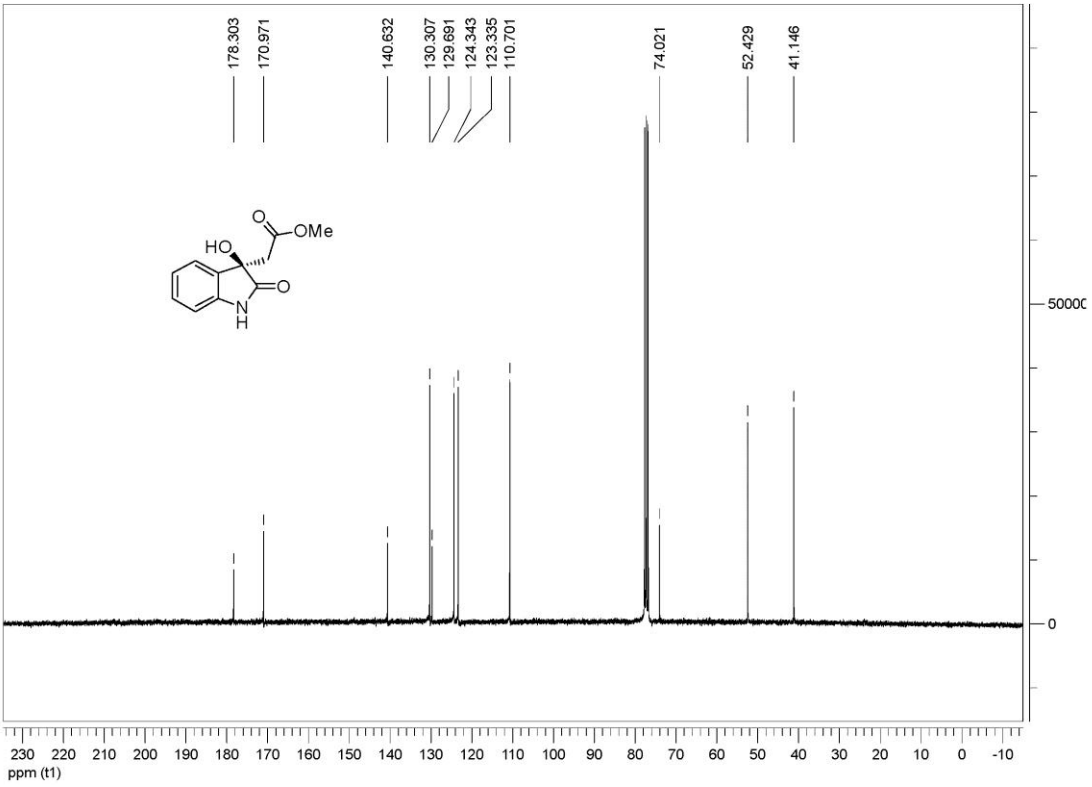


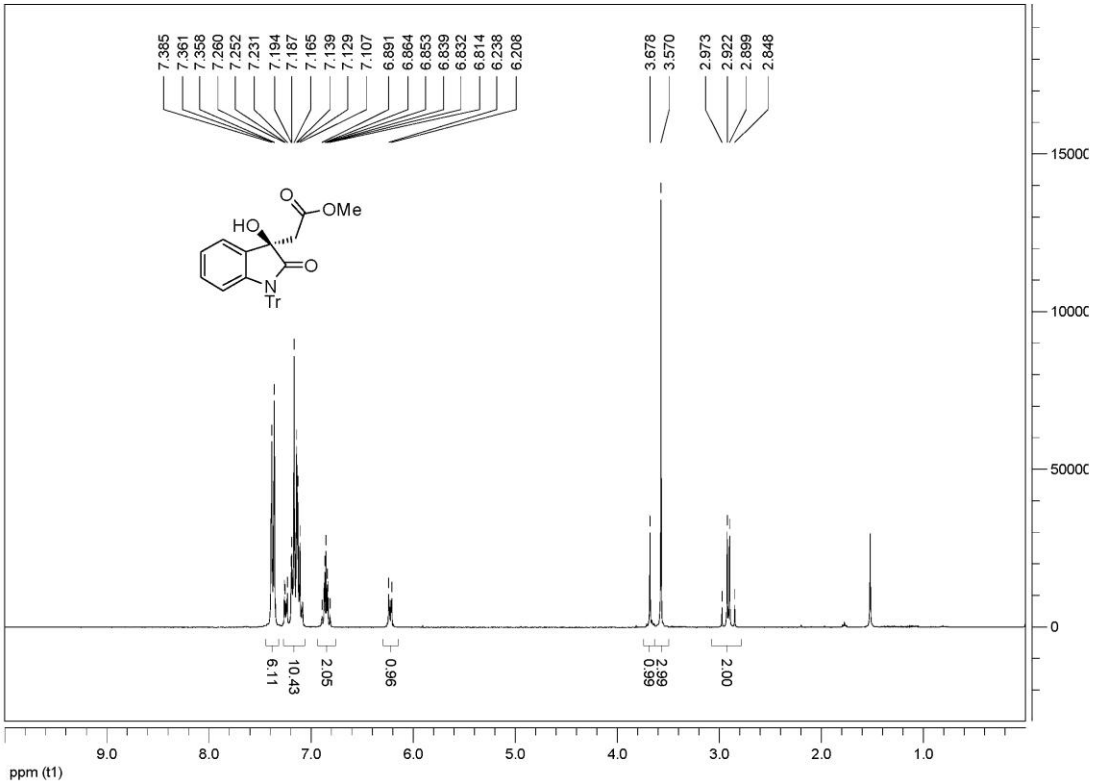
Figure S-2. ORTEP Drawing of Compound 3r.



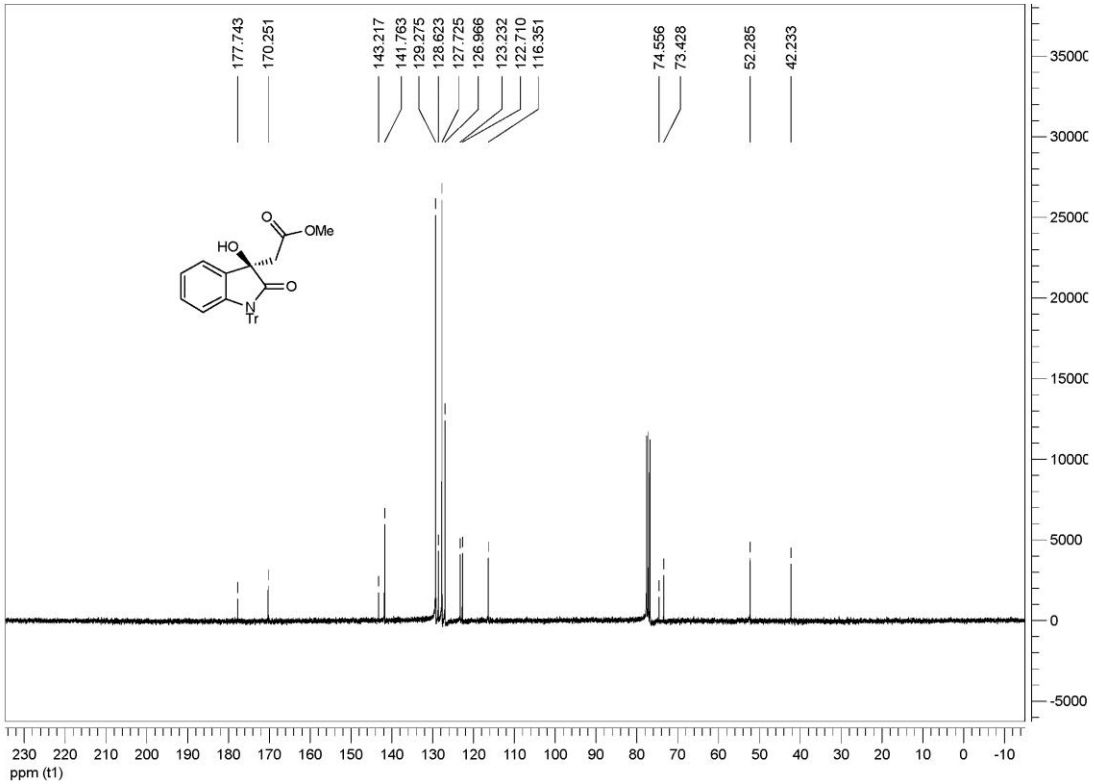
^{1}H NMR of **3a**



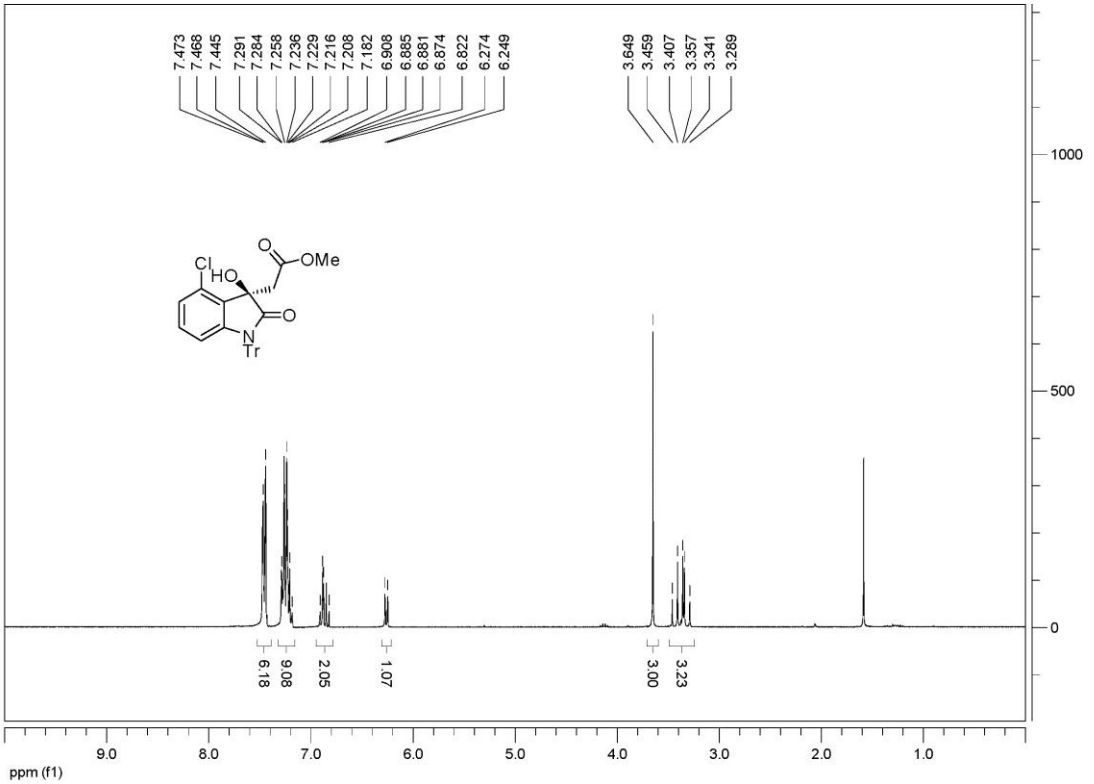
^{13}C NMR of **3a**



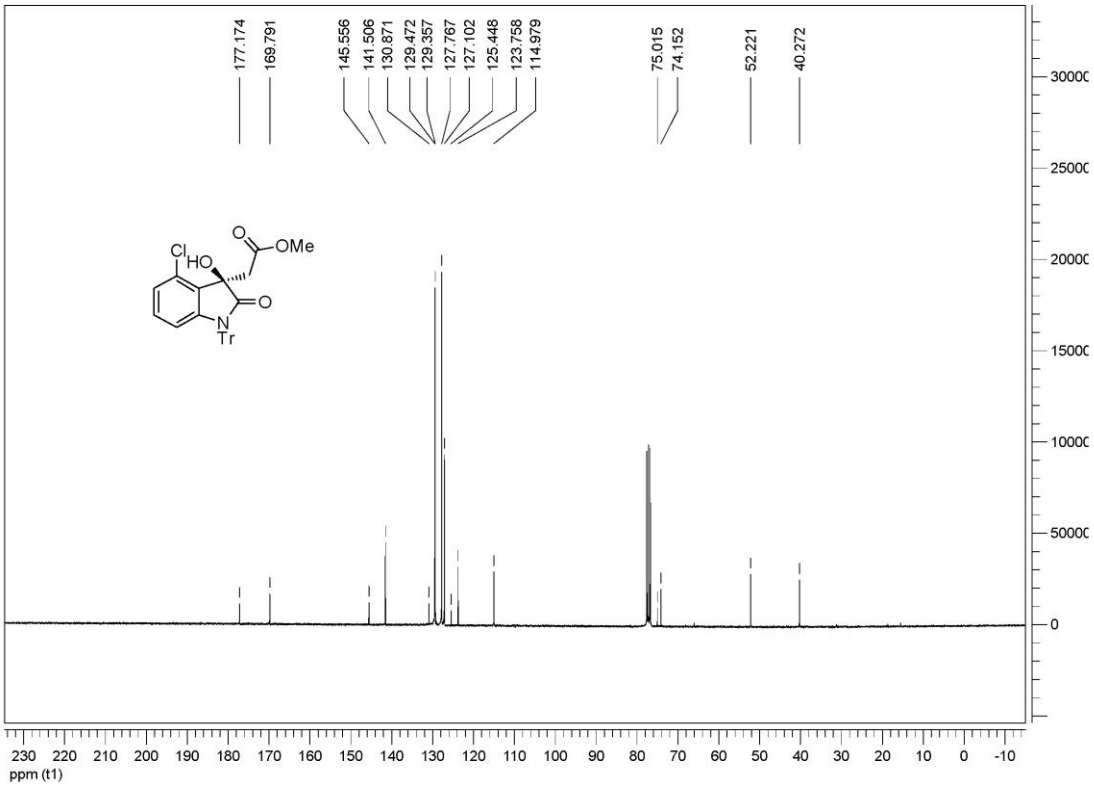
^{1}H NMR of **3d**



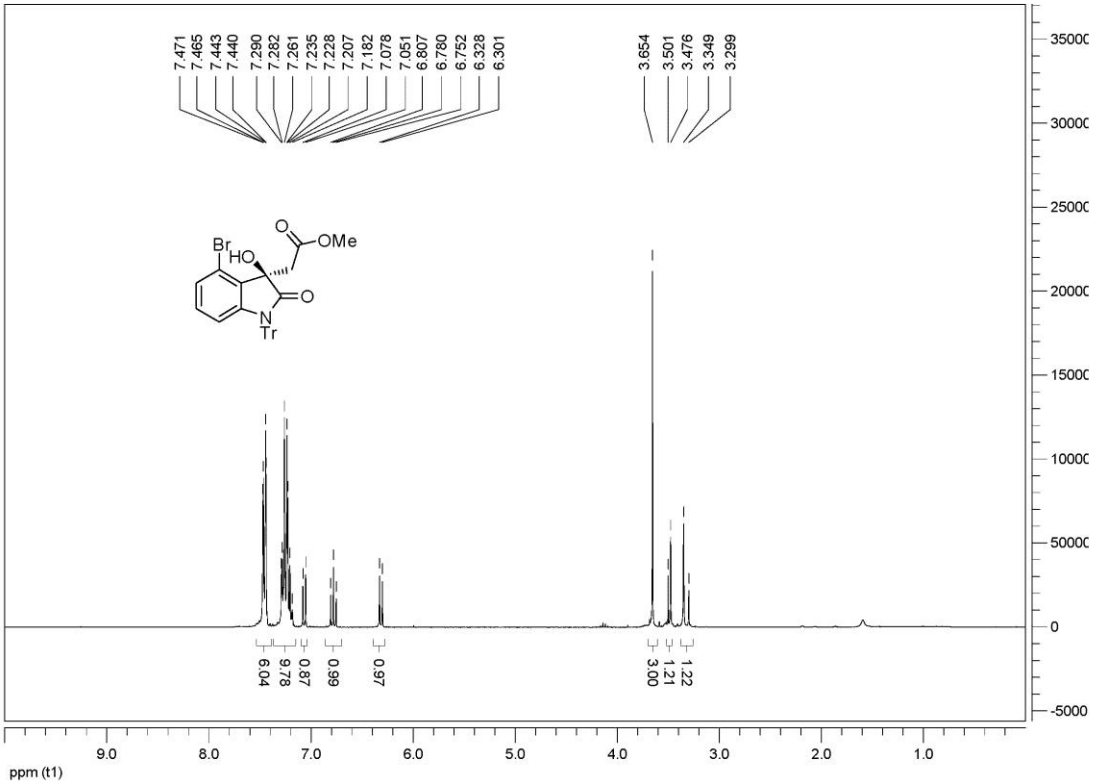
^{13}C NMR of **3d**



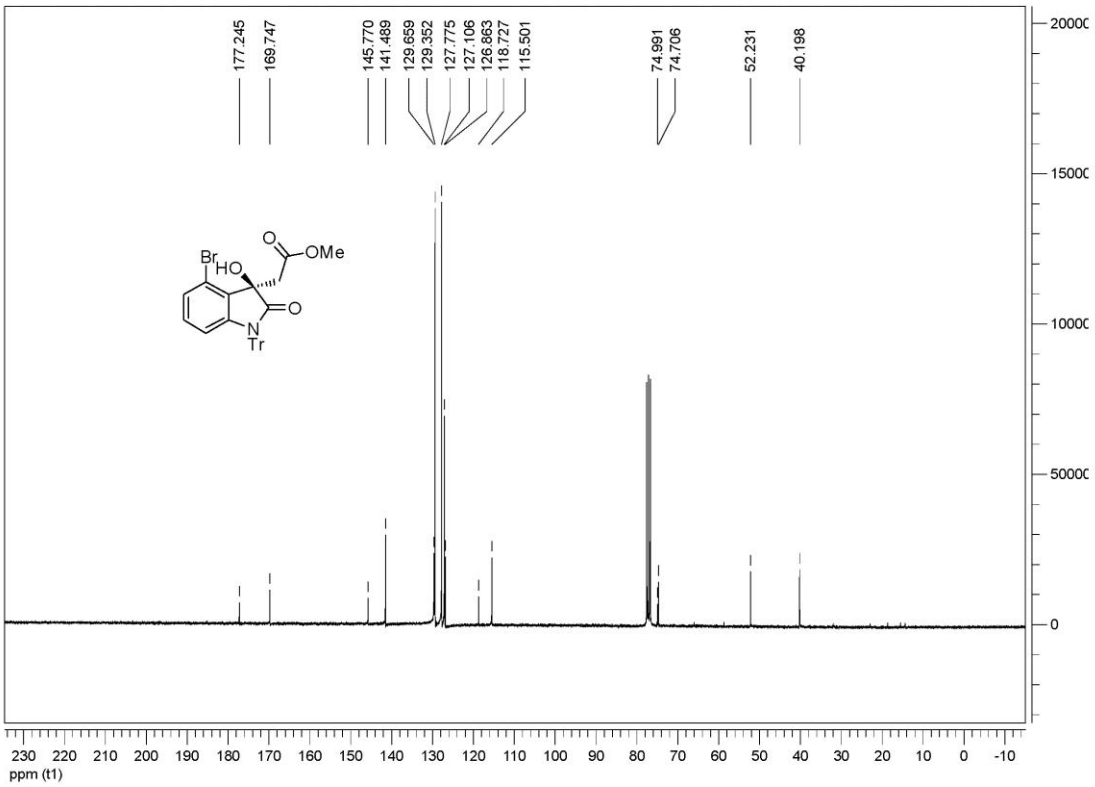
^{1}H NMR of **3e**



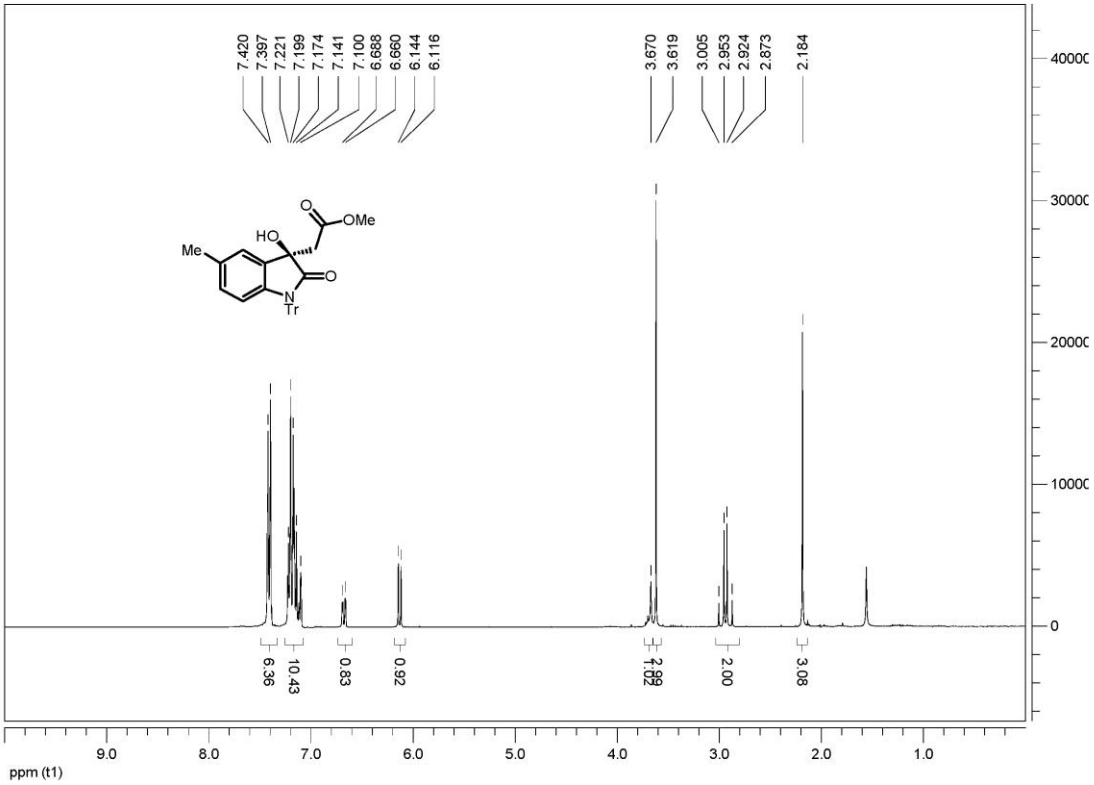
^{13}C NMR of **3e**



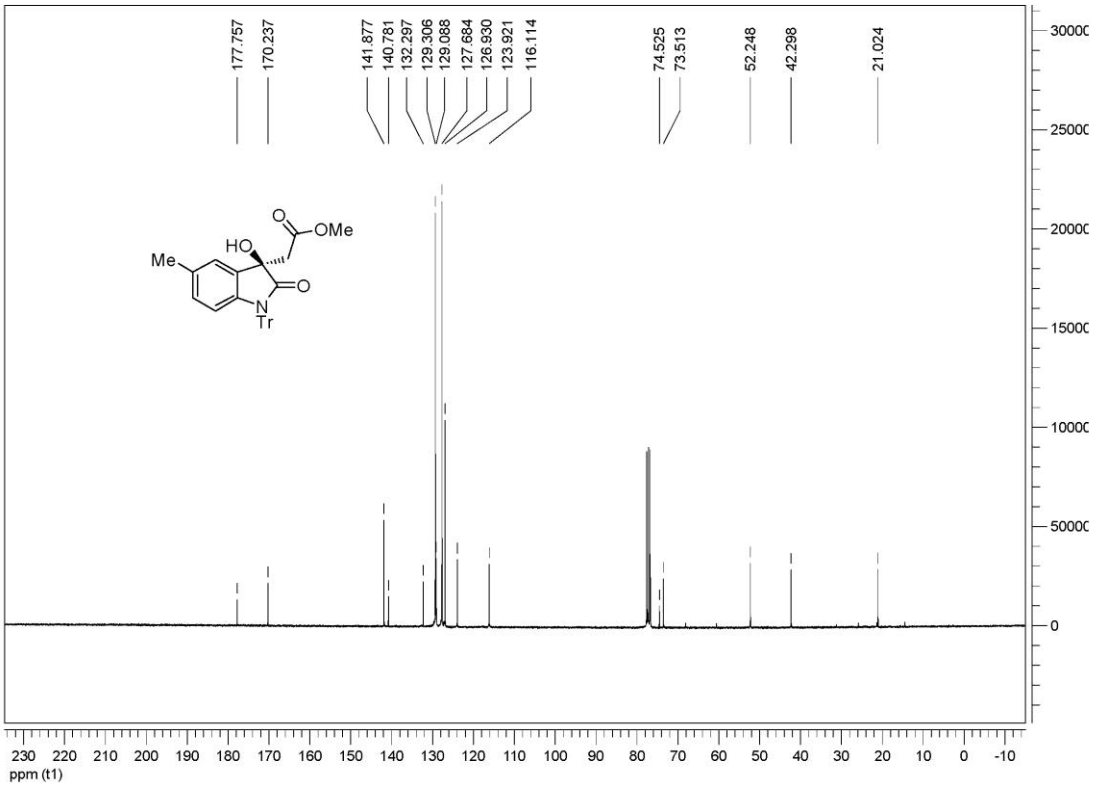
^{1}H NMR of **3f**



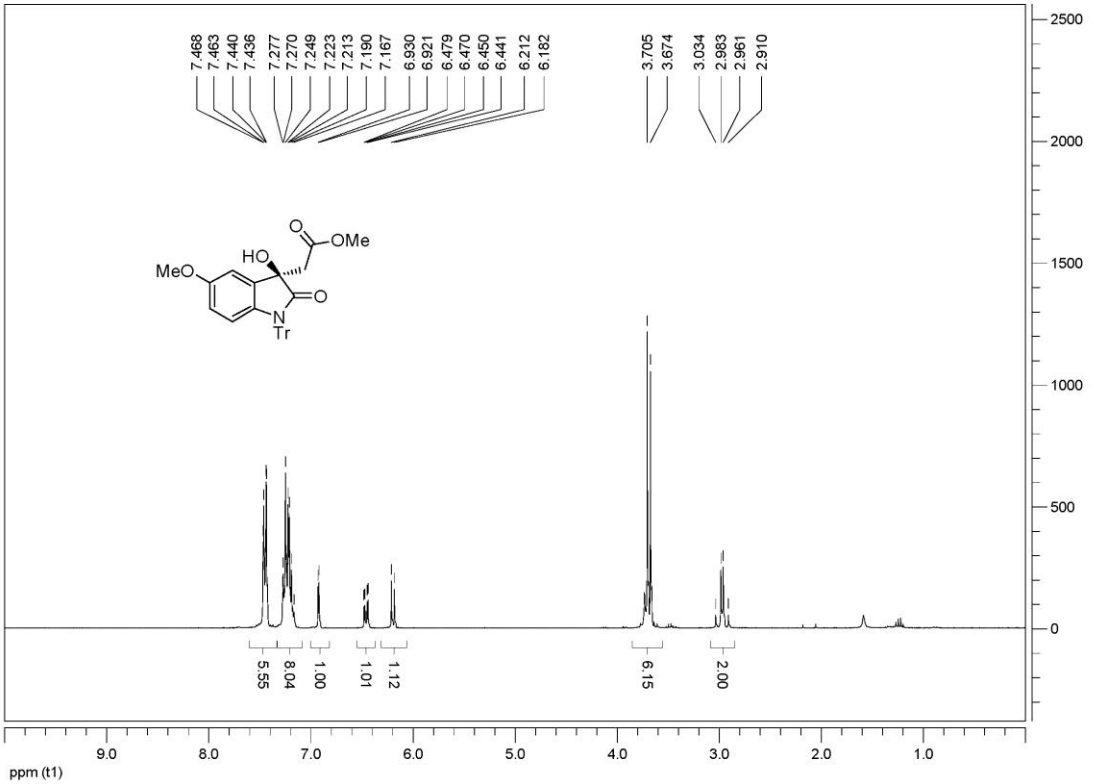
^{13}C NMR of **3f**



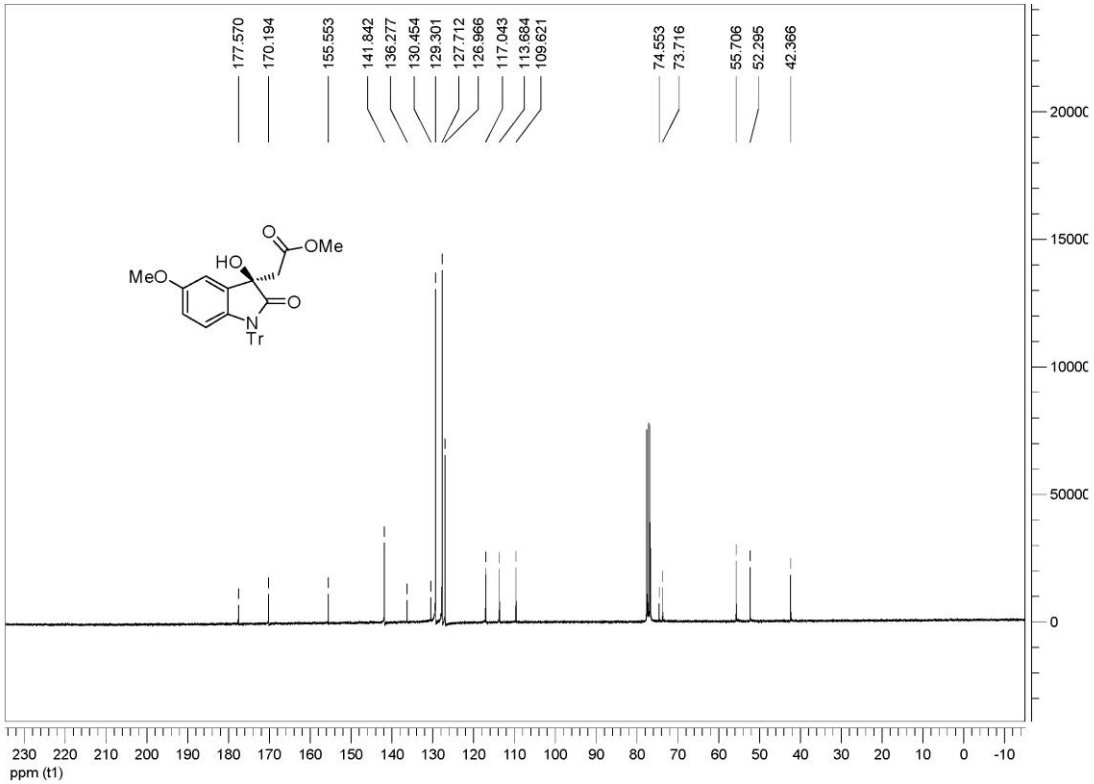
^{1}H NMR of **3g**



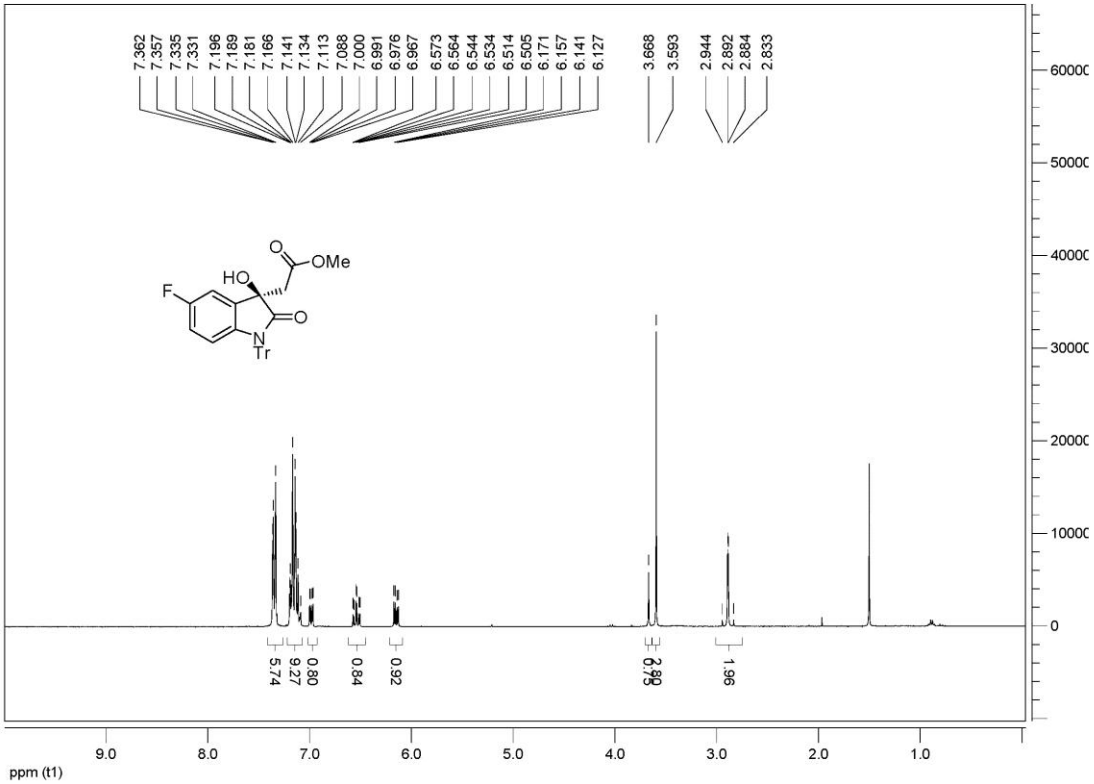
^{13}C NMR of **3g**



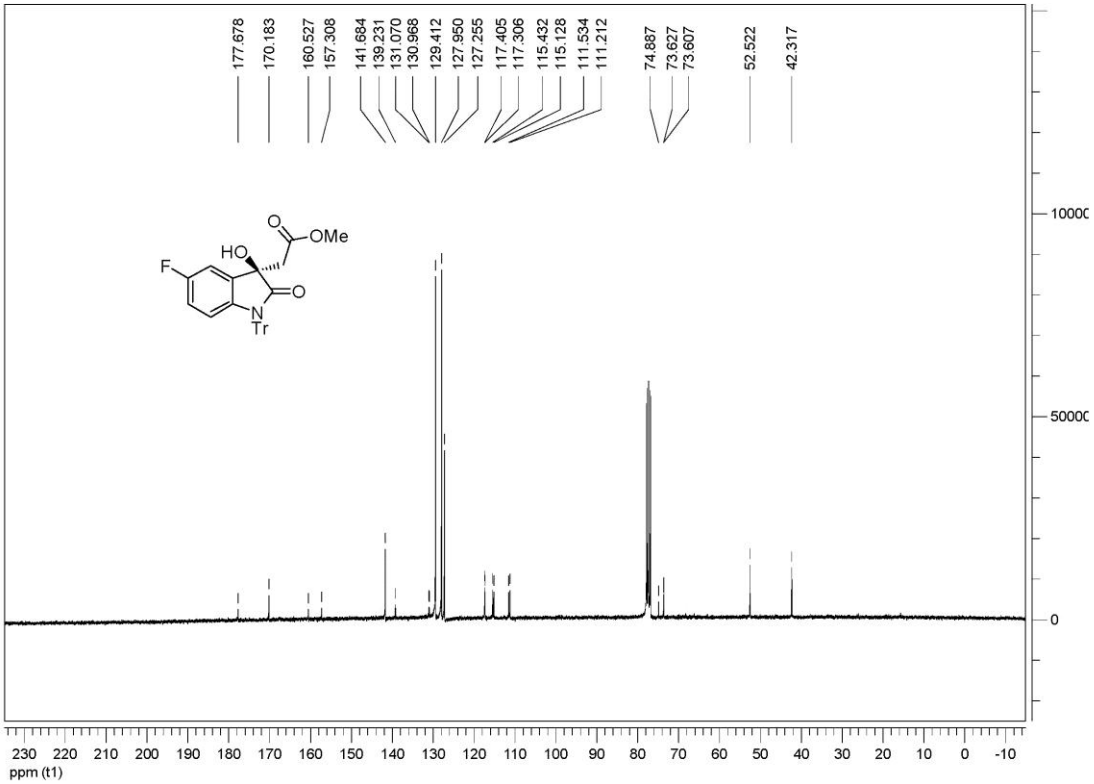
^{1}H NMR of **3h**



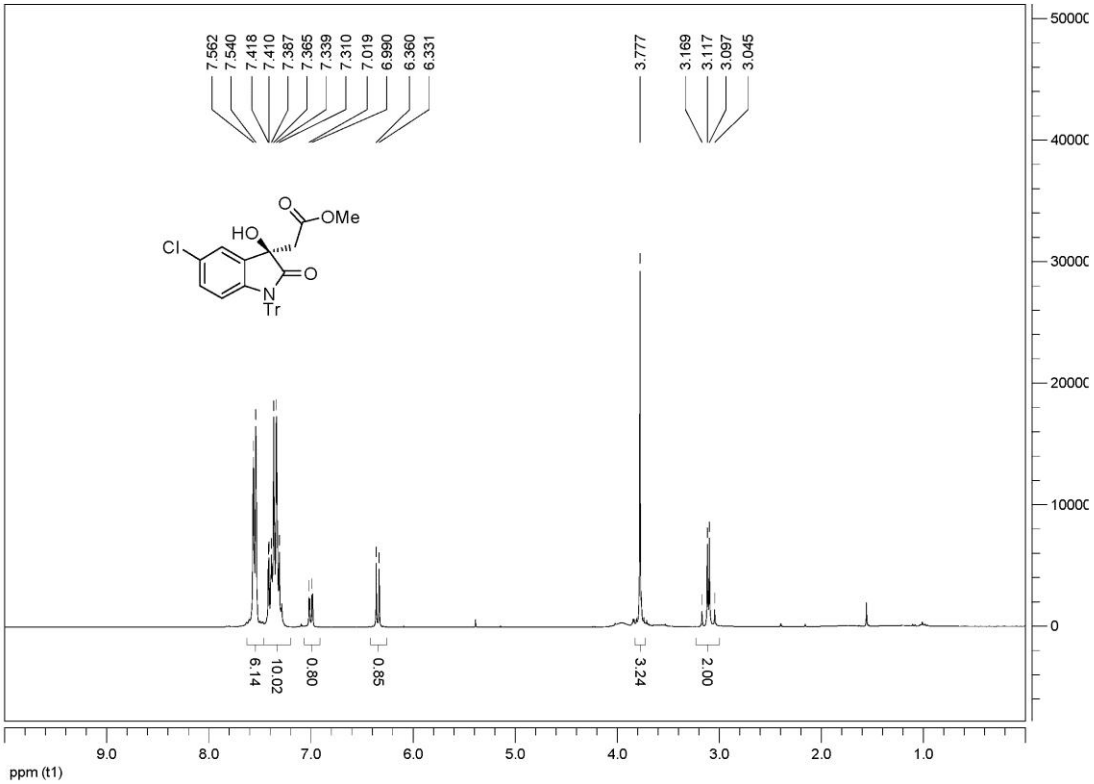
^{13}C NMR of **3h**



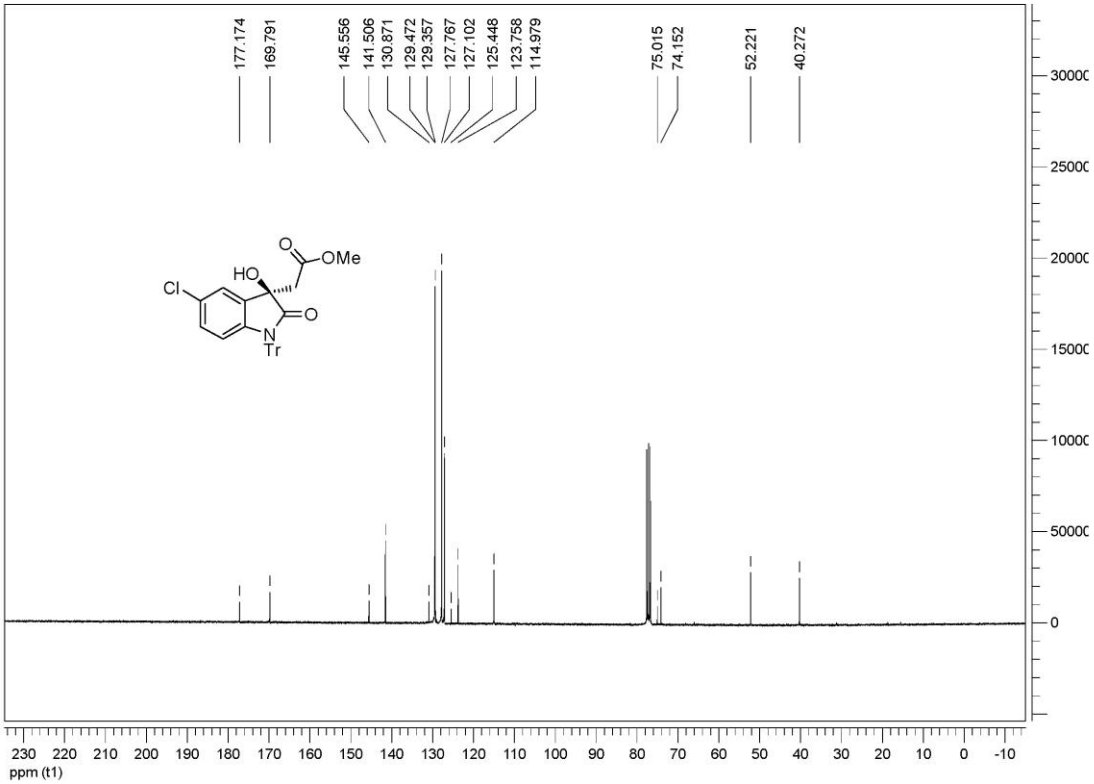
^{1}H NMR of **3i**



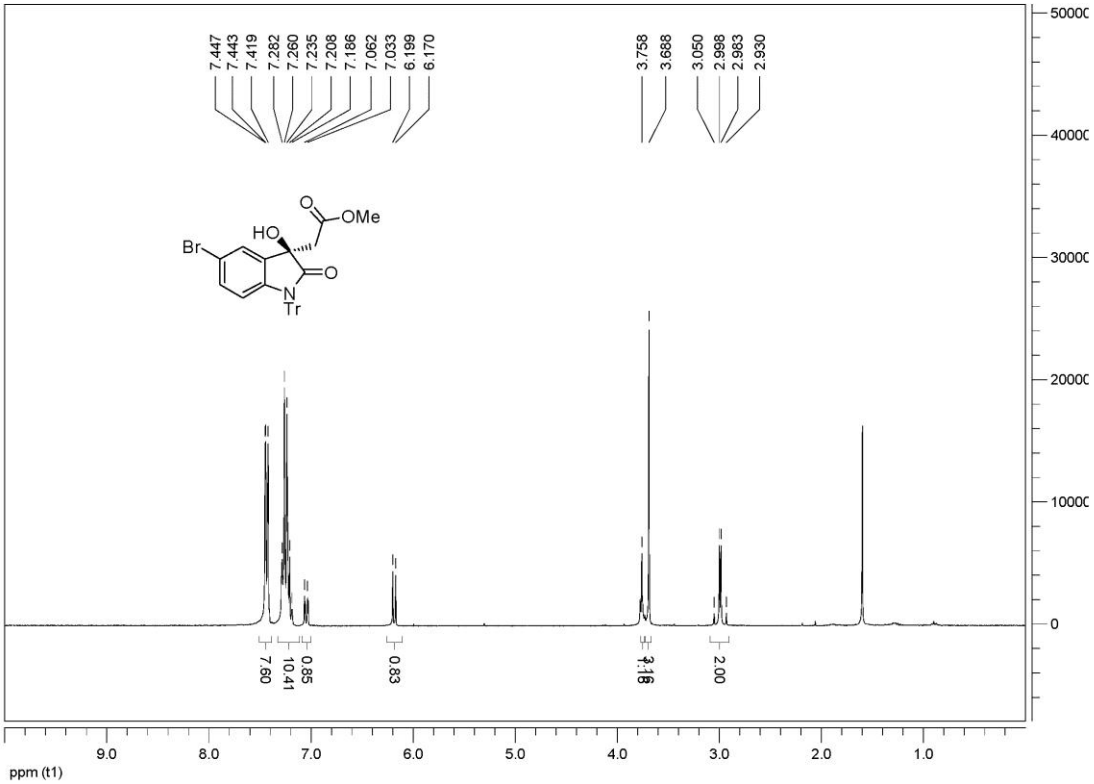
^{13}C NMR of **3i**



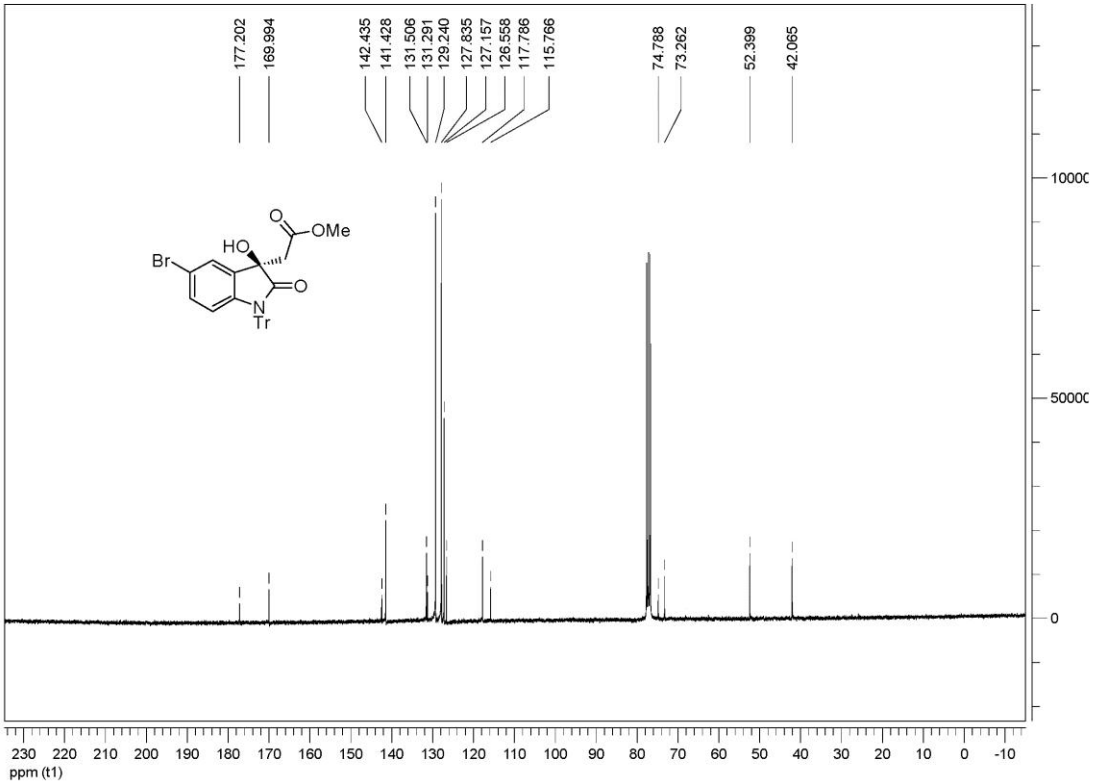
^{1}H NMR of **3j**



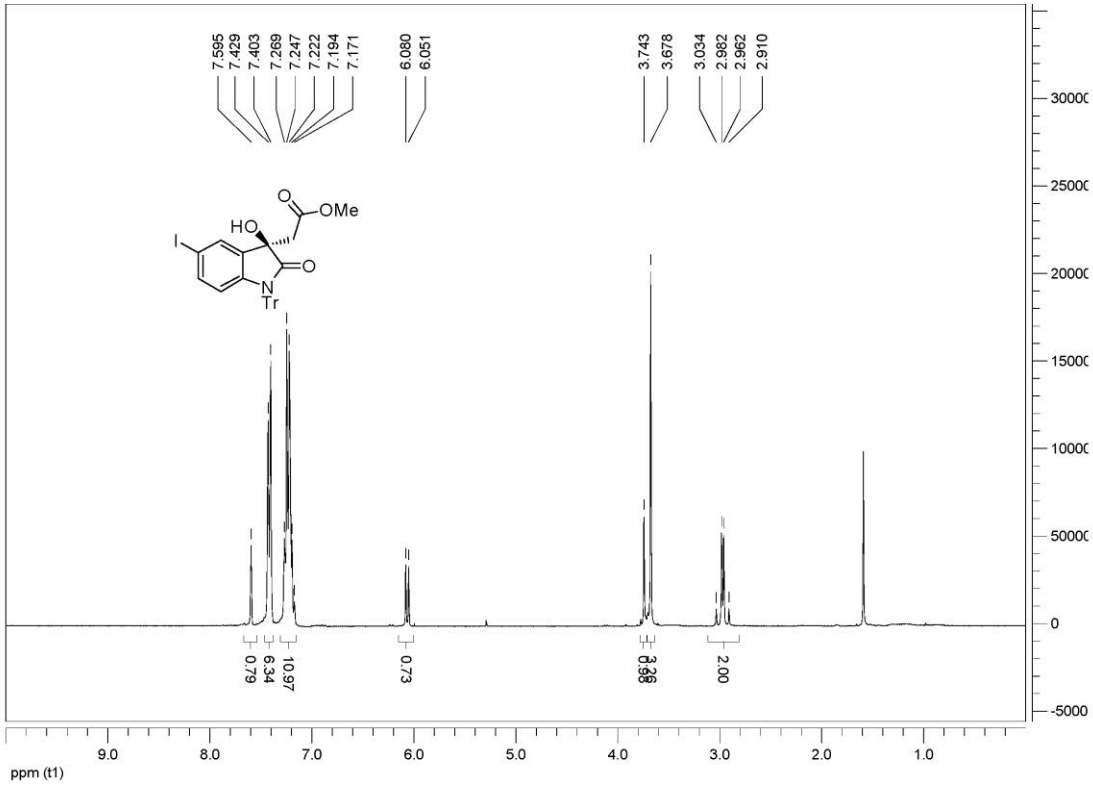
^{13}C NMR of **3j**



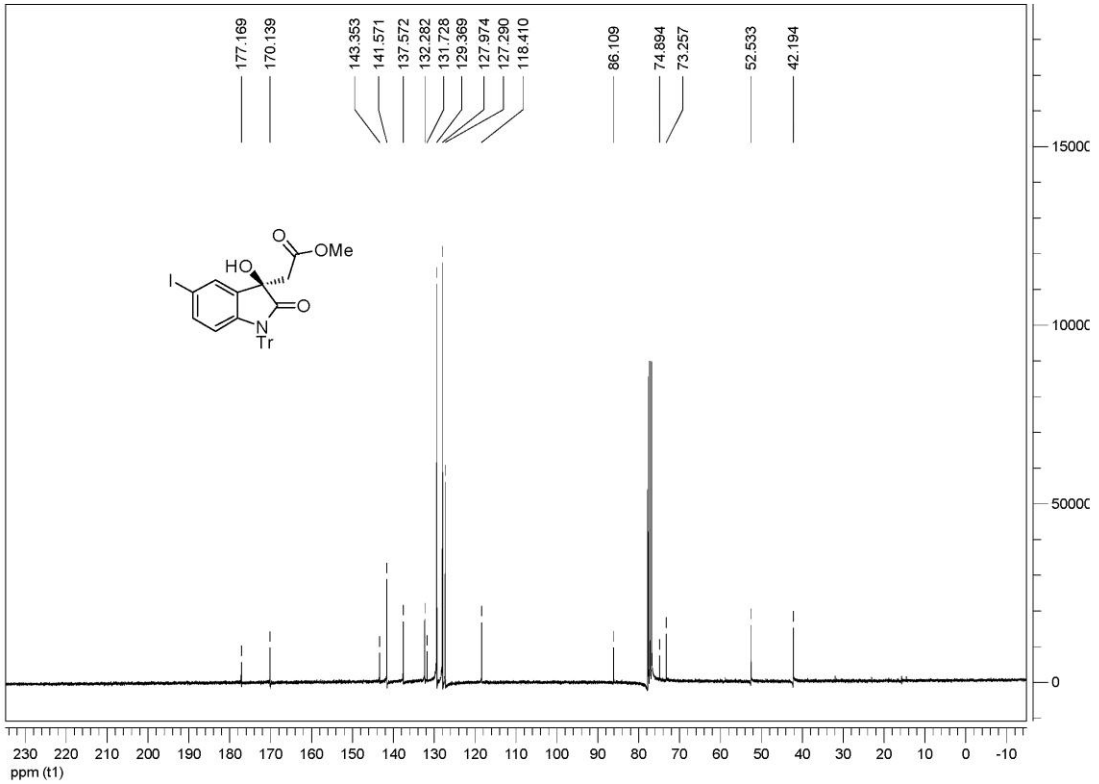
^{1}H NMR of **3k**



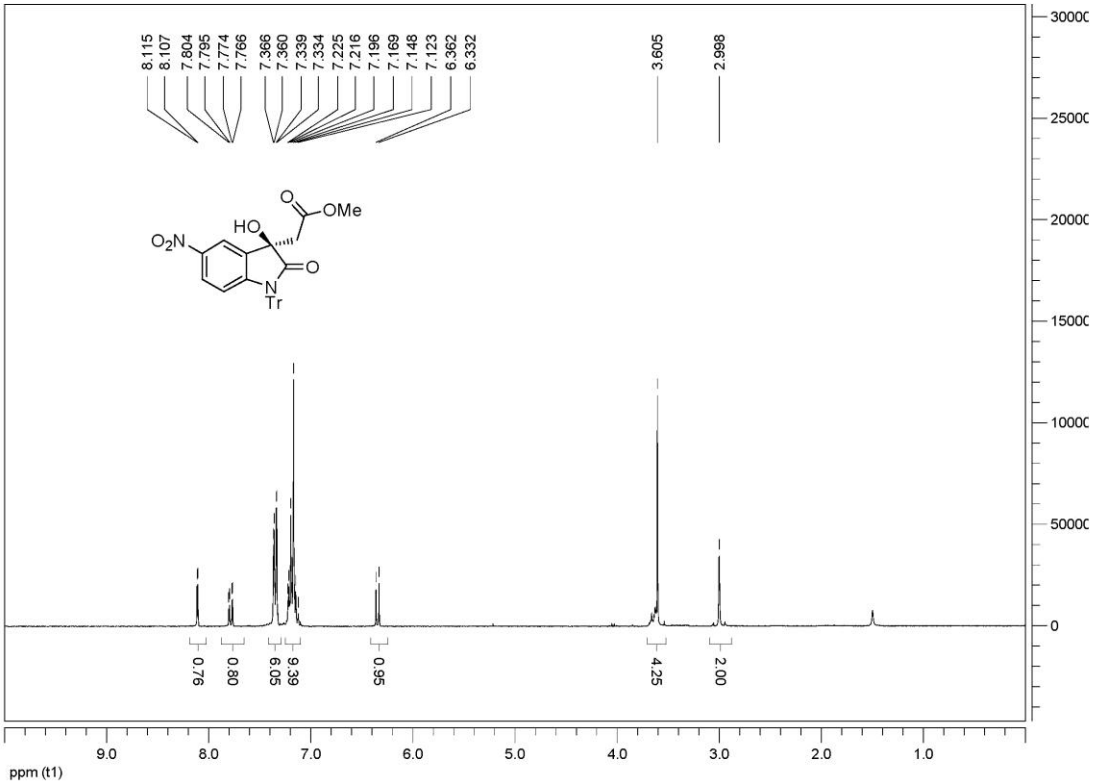
^{13}C NMR of **3k**



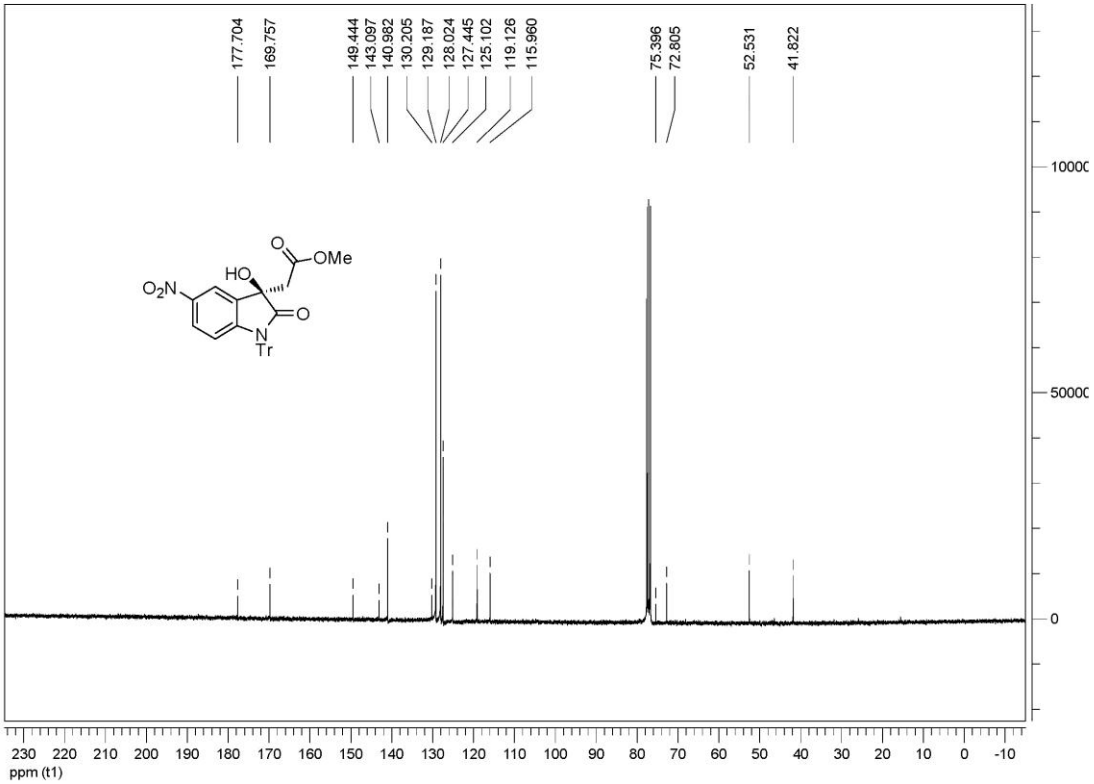
^{1}H NMR of **3l**



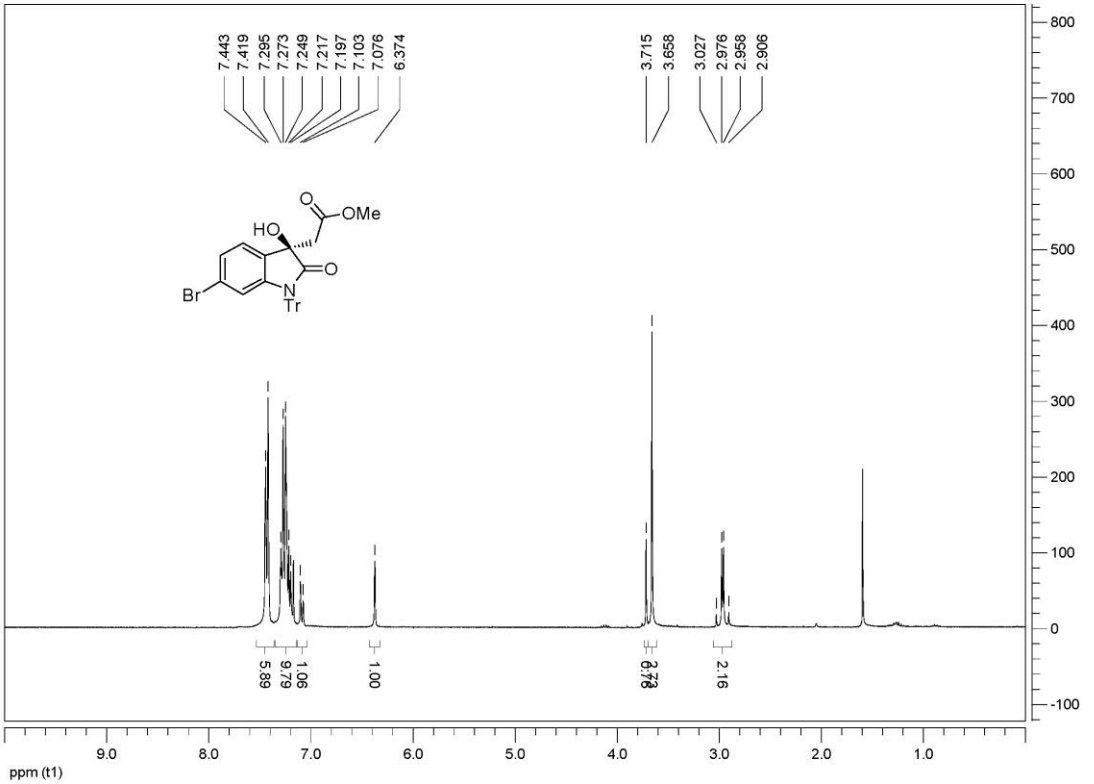
^{13}C NMR of **3l**



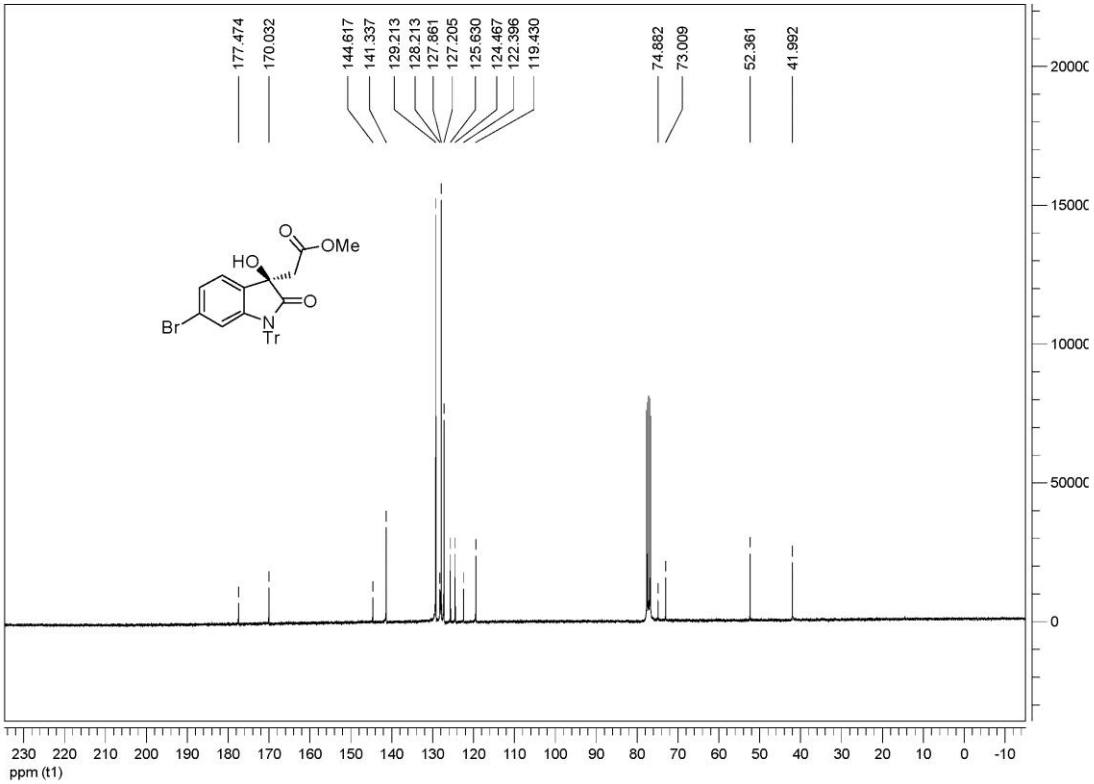
^{1}H NMR of **3m**



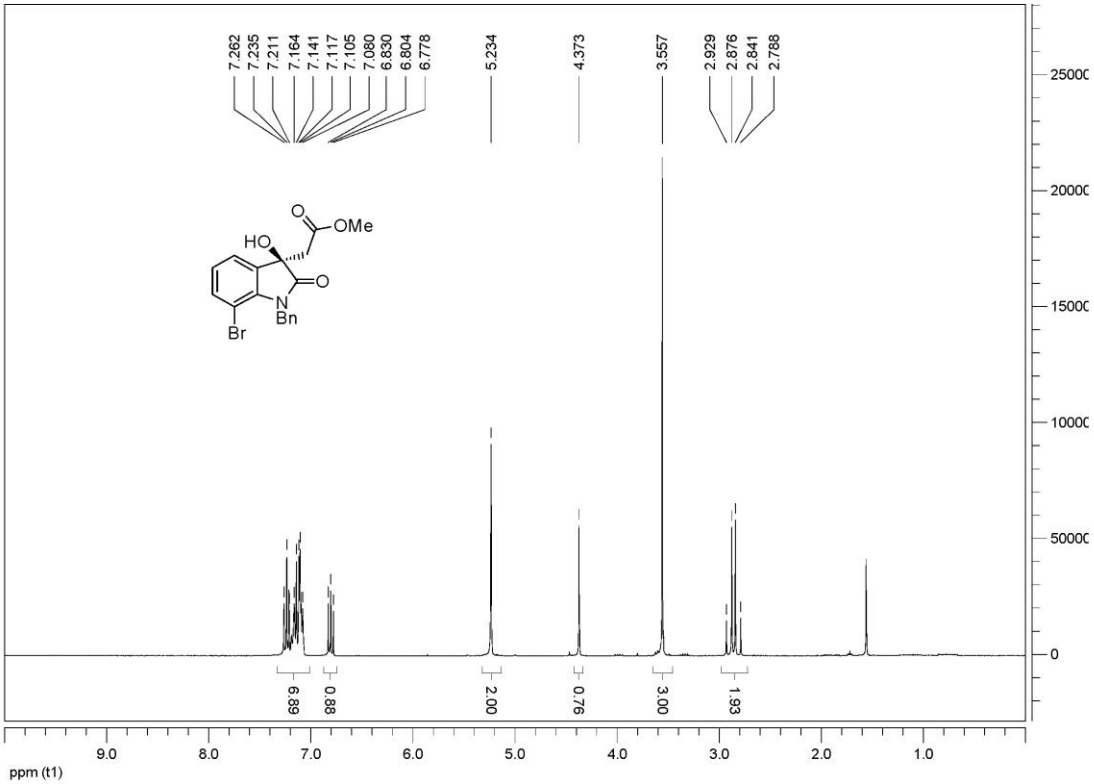
^{13}C NMR of **3m**



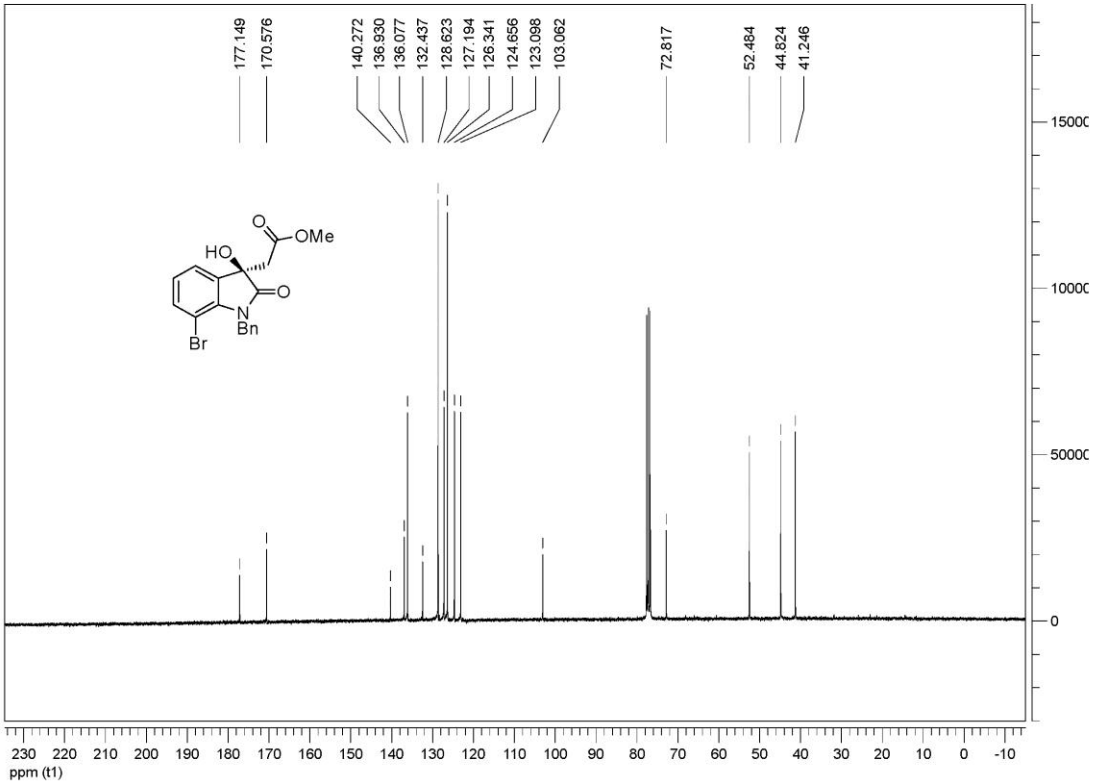
^{1}H NMR of **3n**



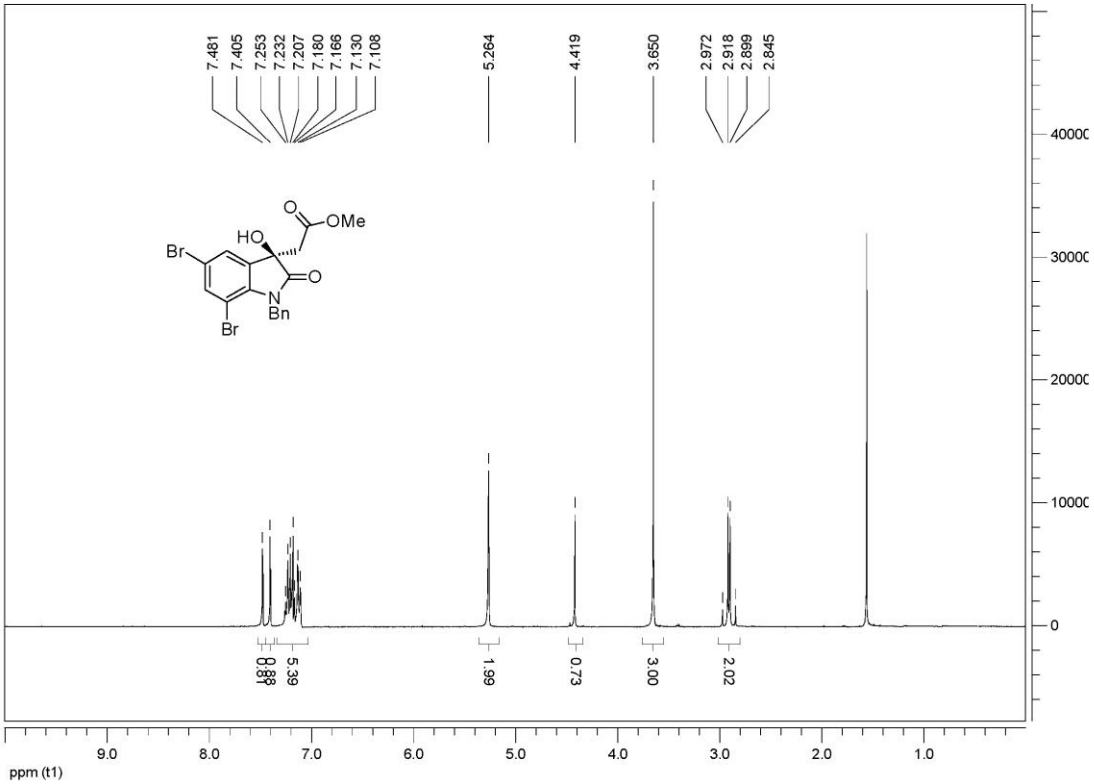
^{13}C NMR of **3n**



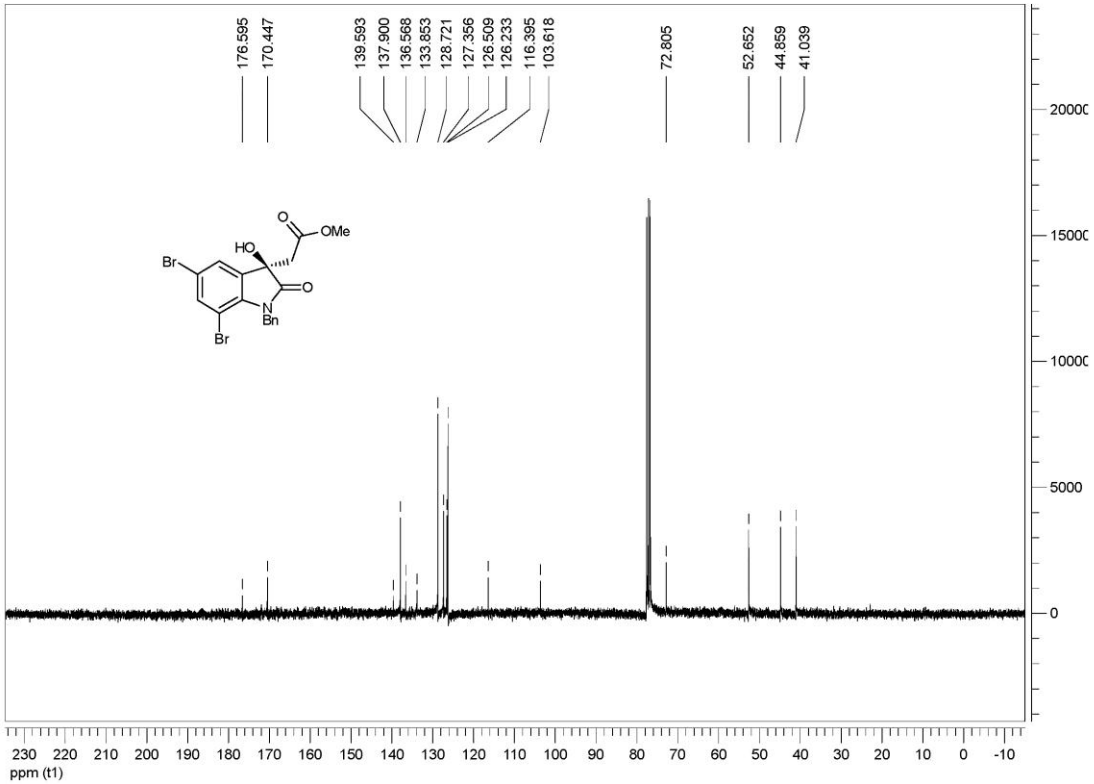
^{1}H NMR of **3o**



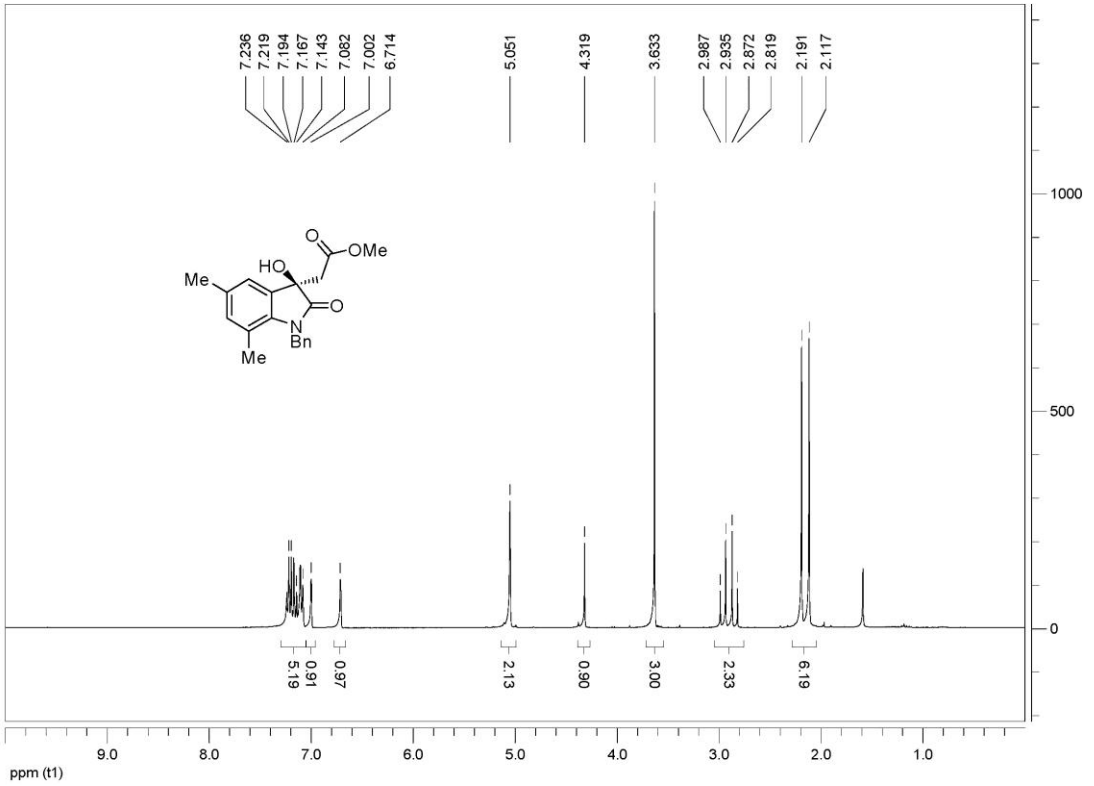
^{13}C NMR of **3o**



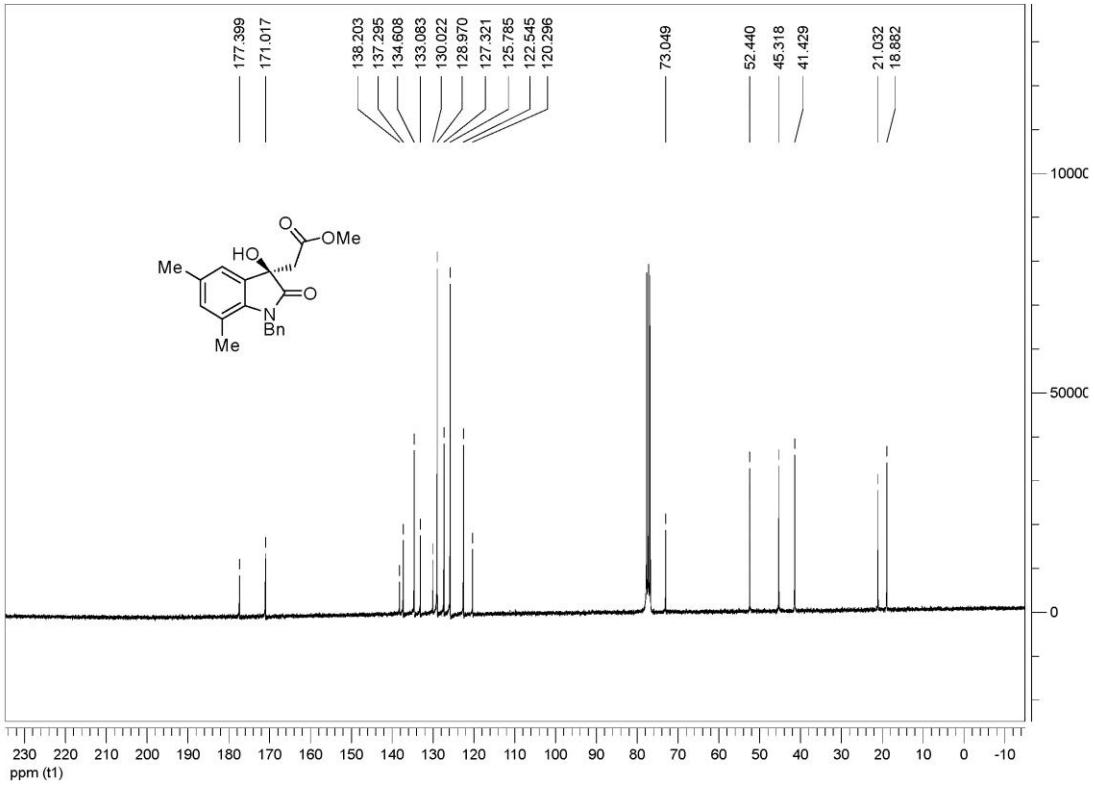
^{1}H NMR of **3p**



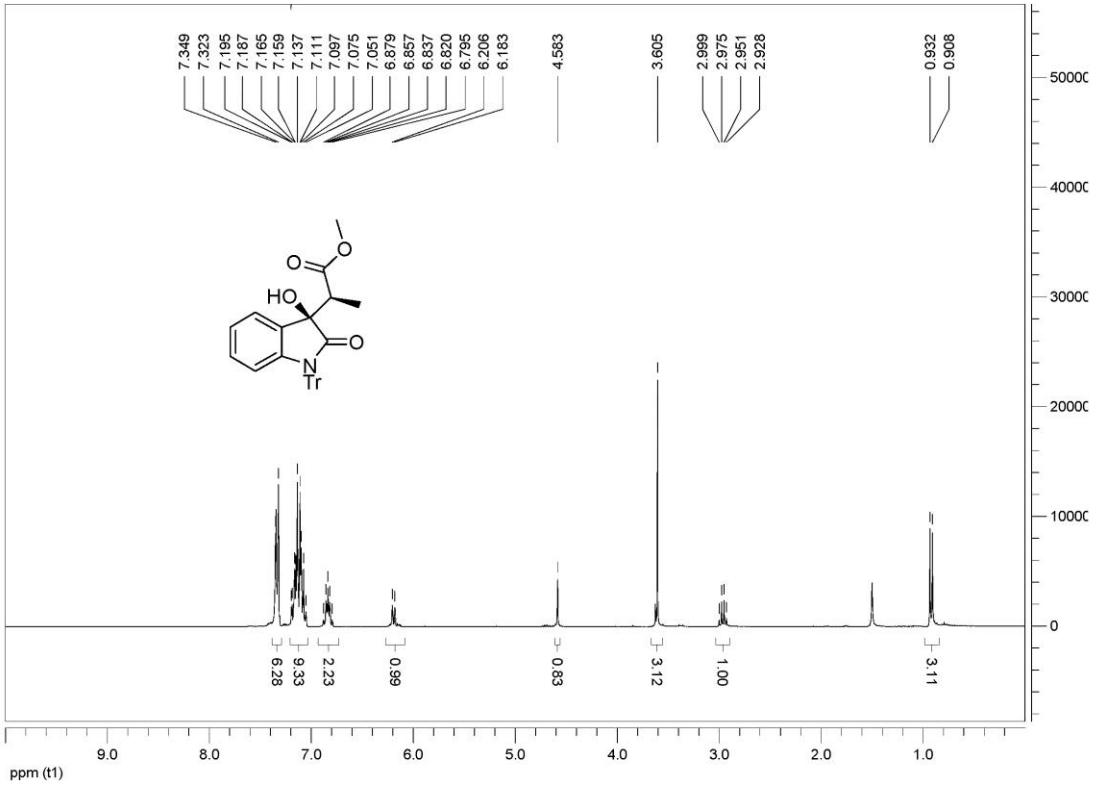
^{13}C NMR of **3p**



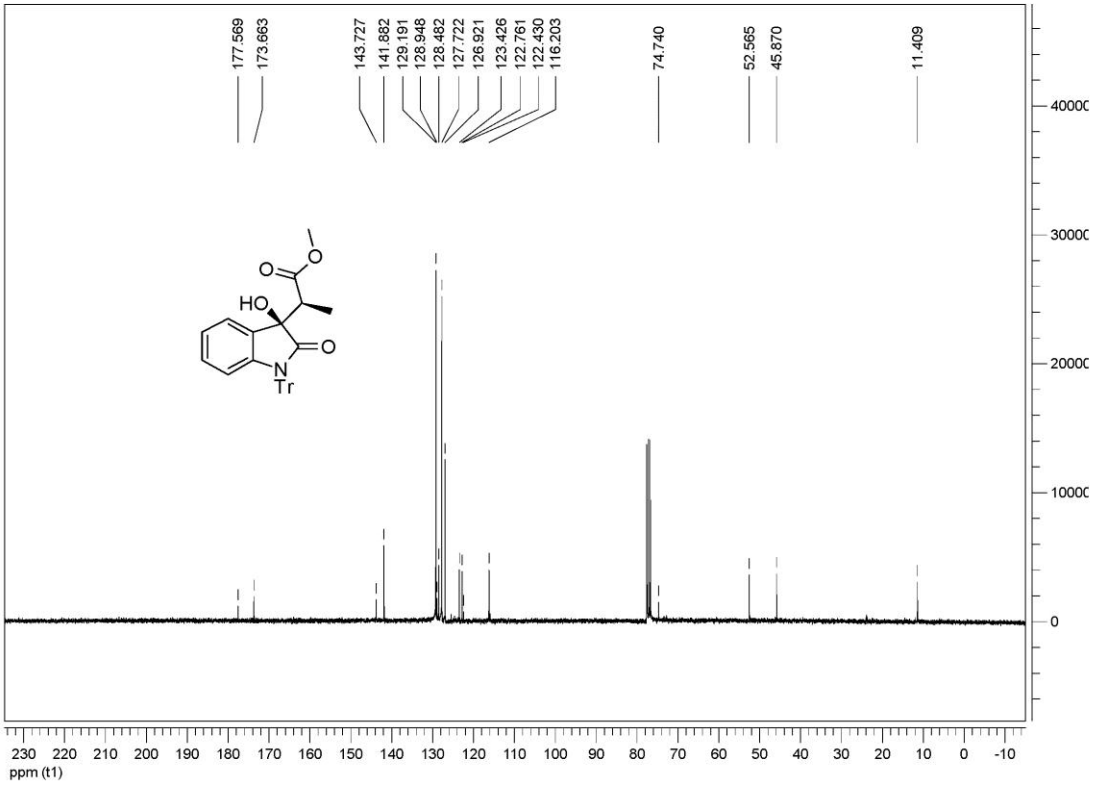
^{1}H NMR of **3q**



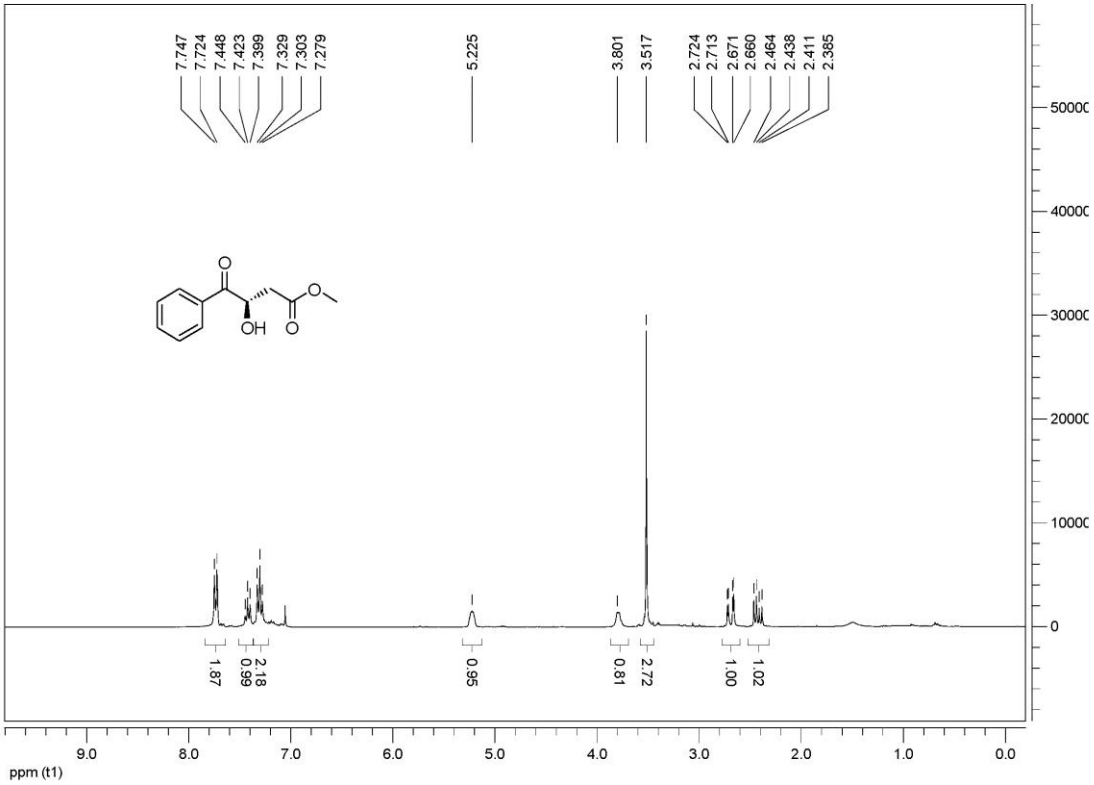
^{13}C NMR of **3q**



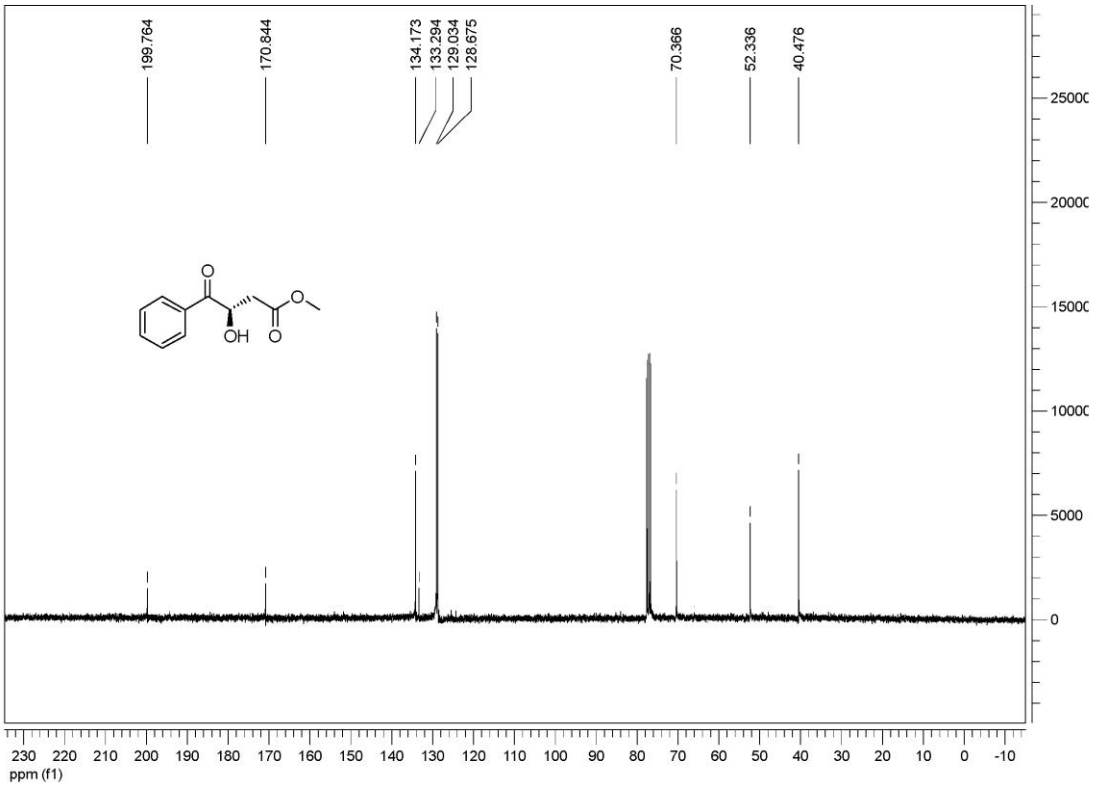
^{1}H NMR of **3r**



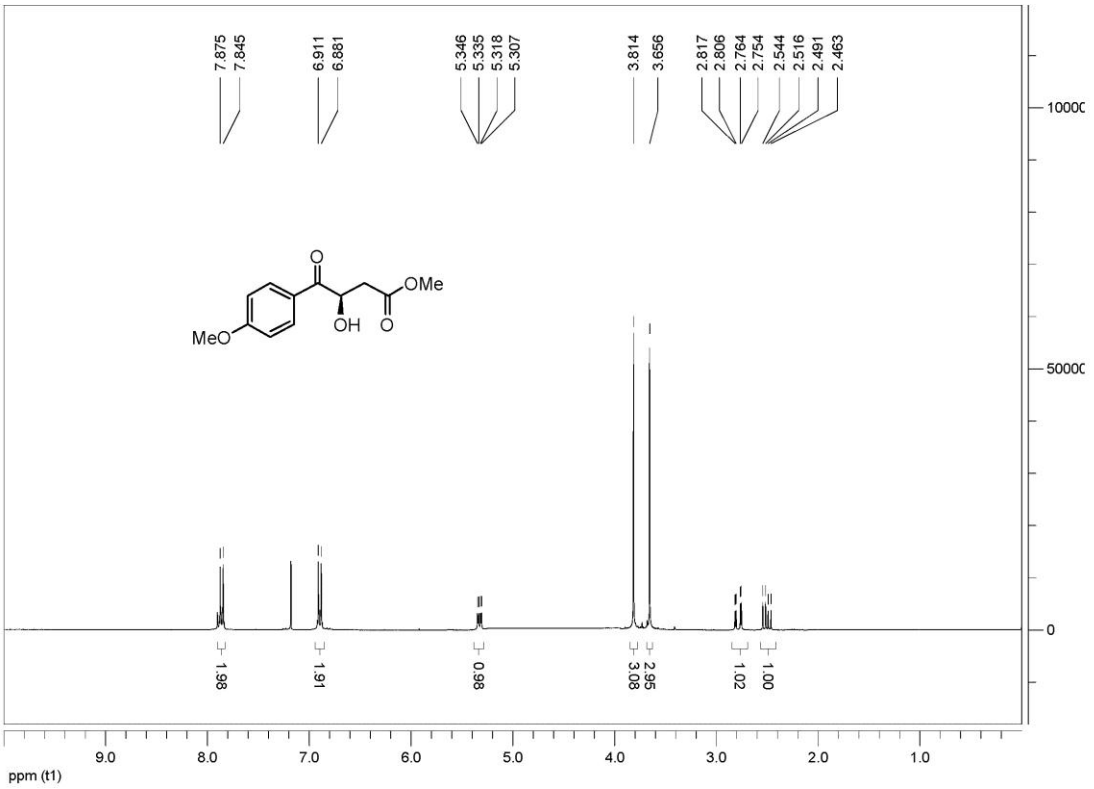
^{13}C NMR of **3r**



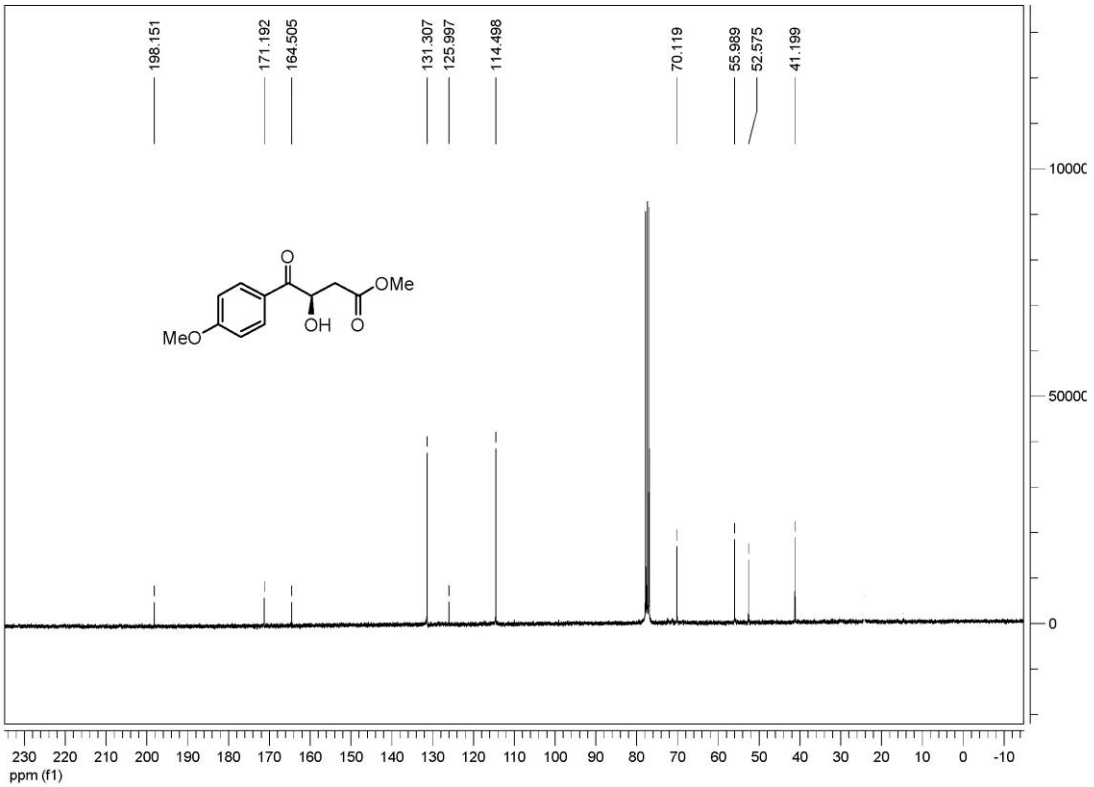
^{1}H NMR of **6a**



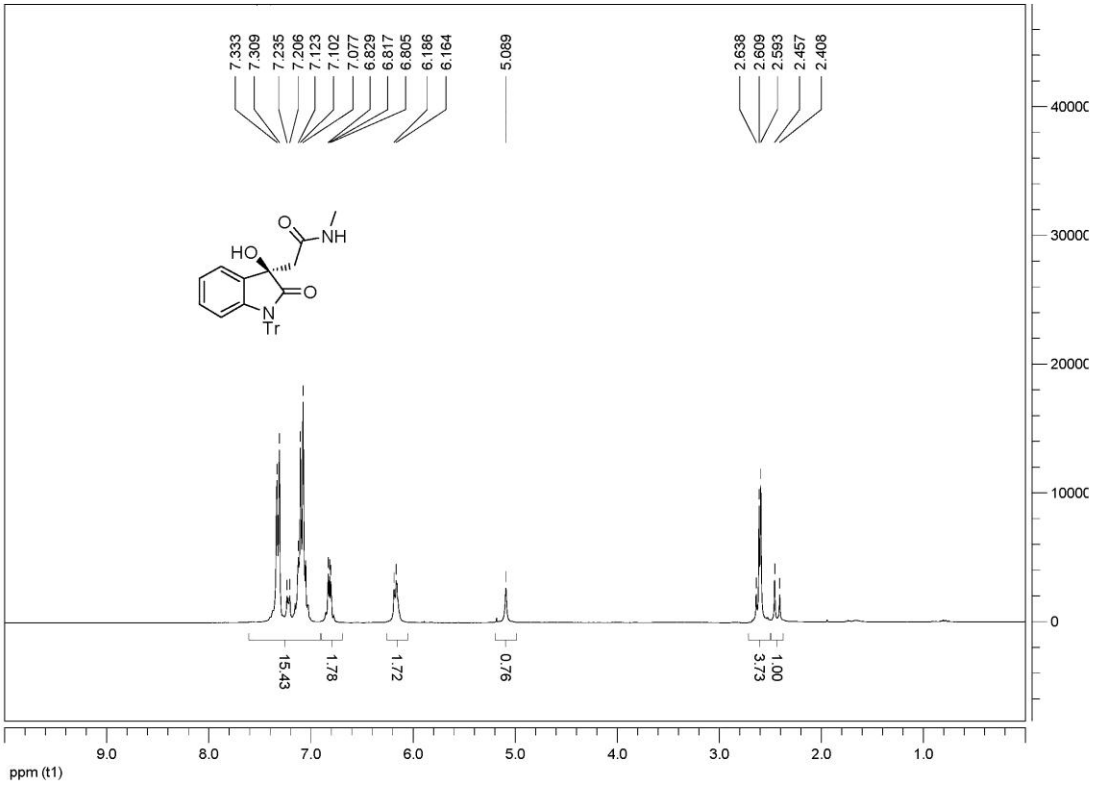
^{13}C NMR of **6a**



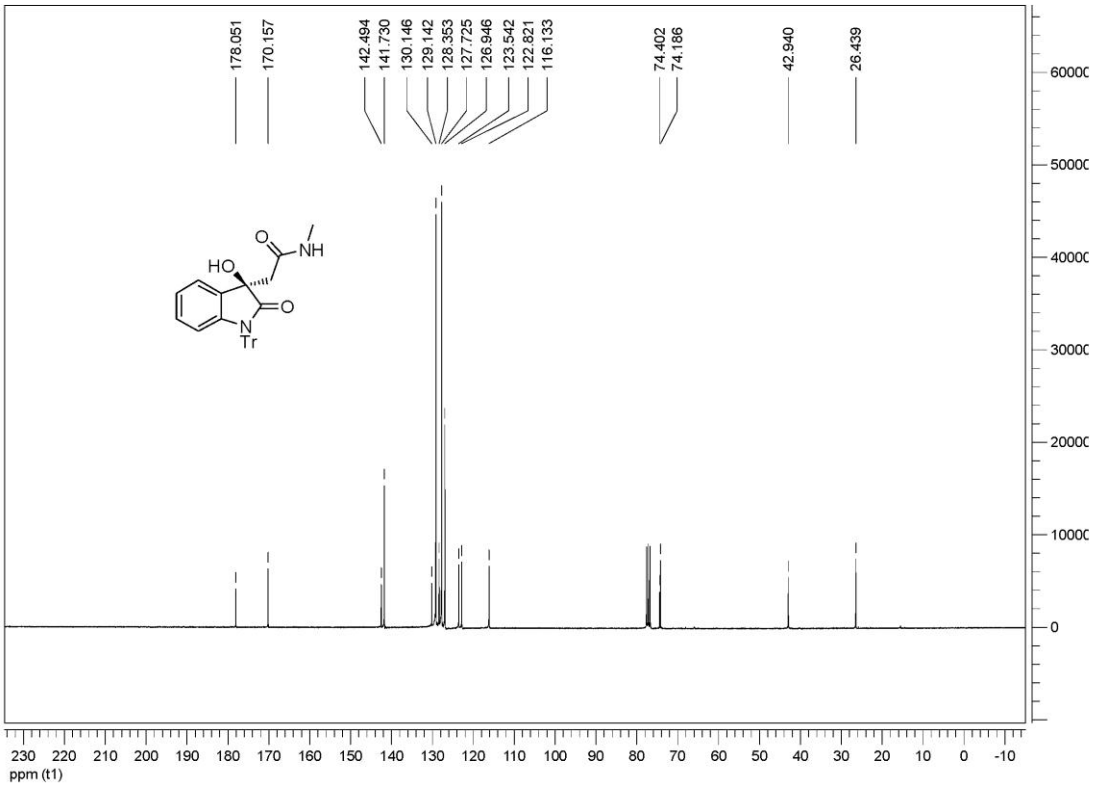
^{1}H NMR of **6b**



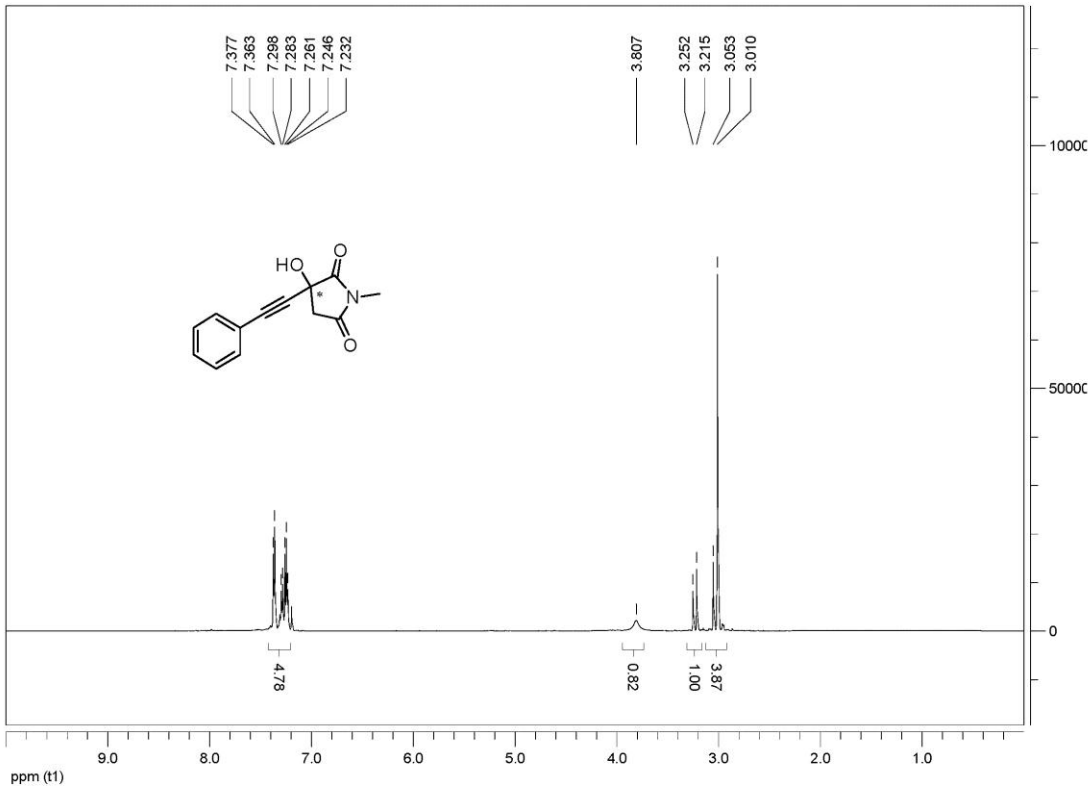
^{13}C NMR of **6b**



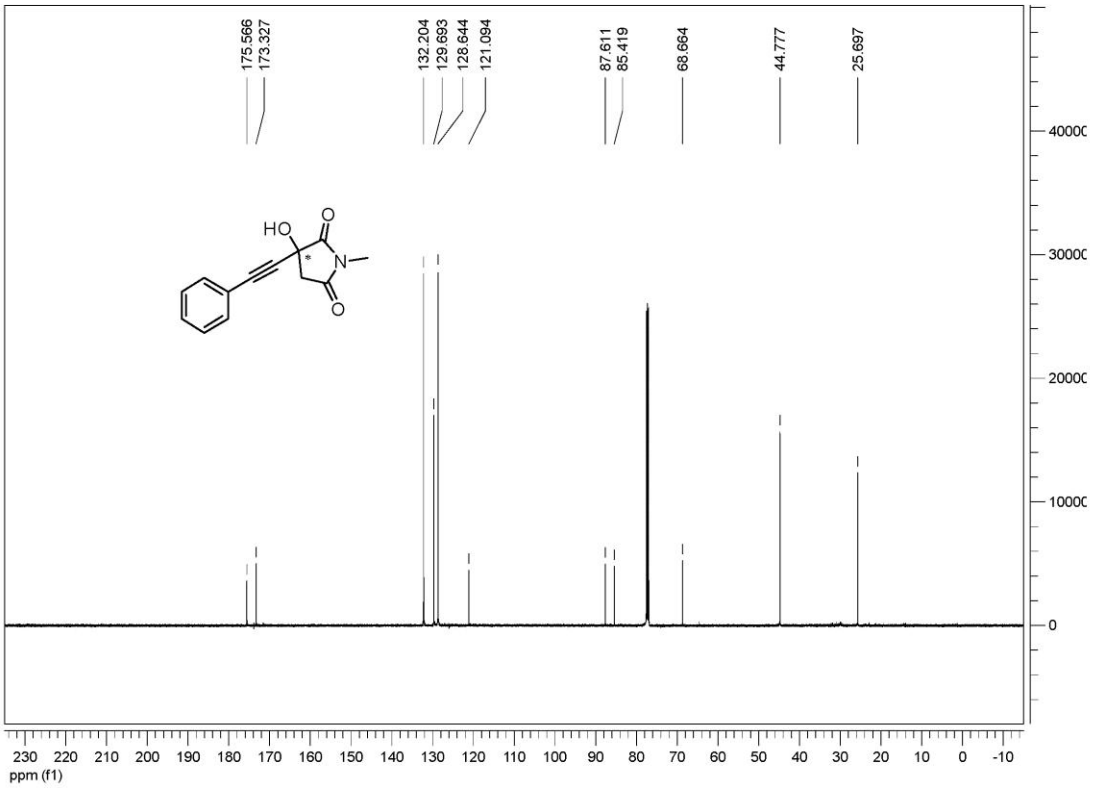
^{1}H NMR of **7**



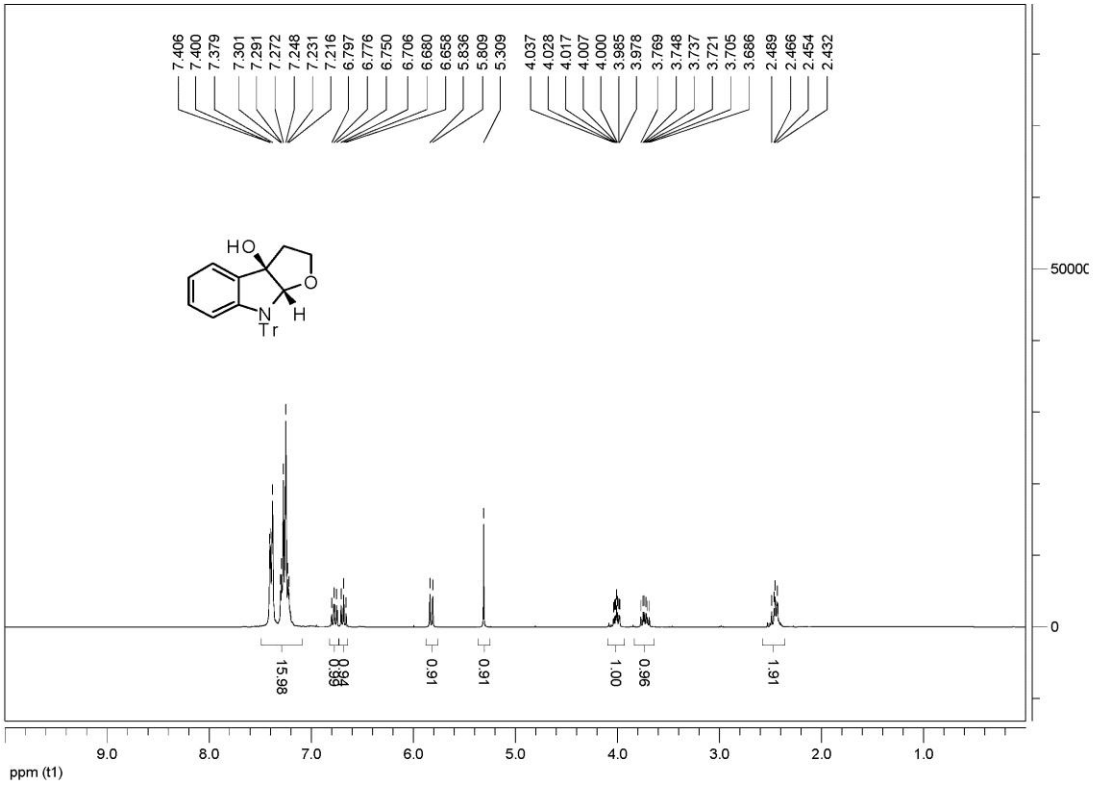
^{13}C NMR of **7**



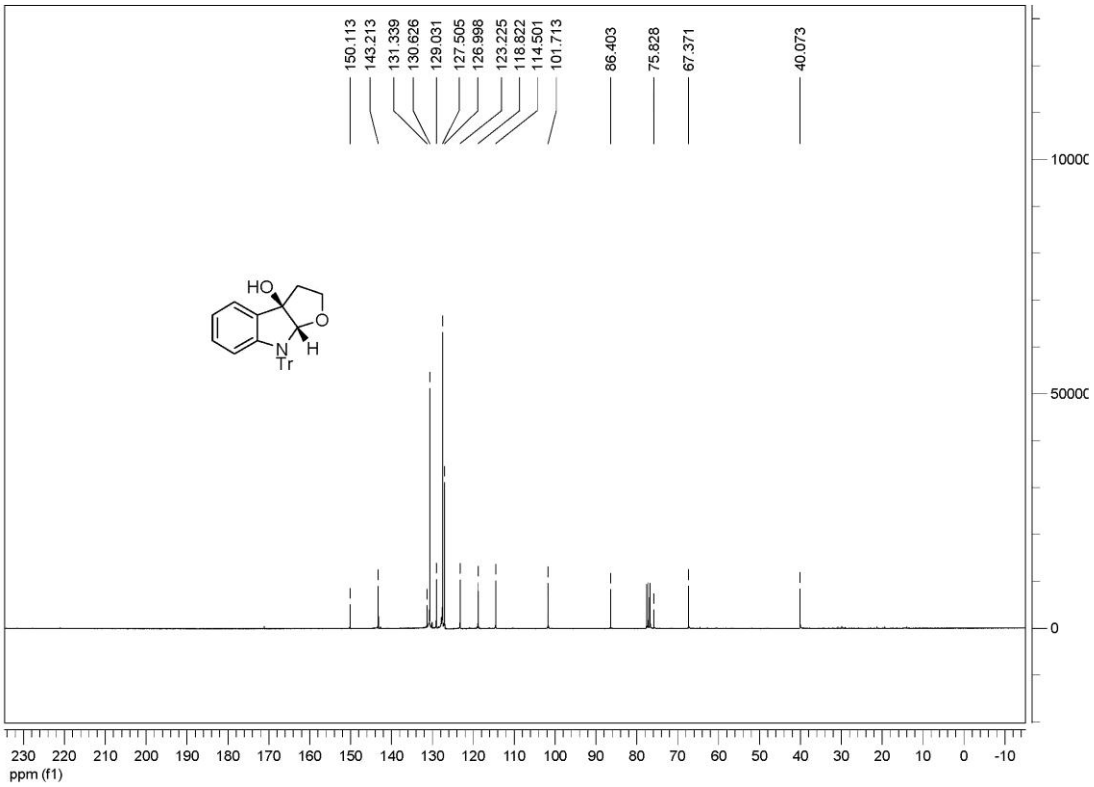
^{1}H NMR of **9**



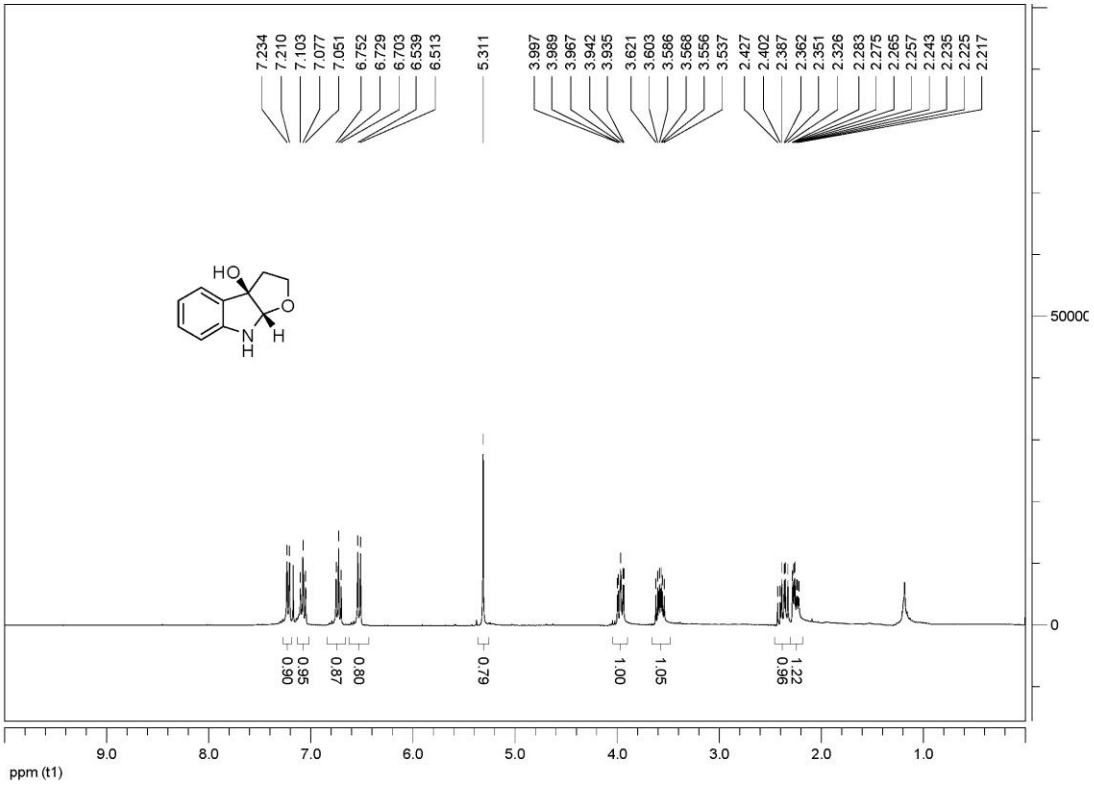
^{13}C NMR of **9**



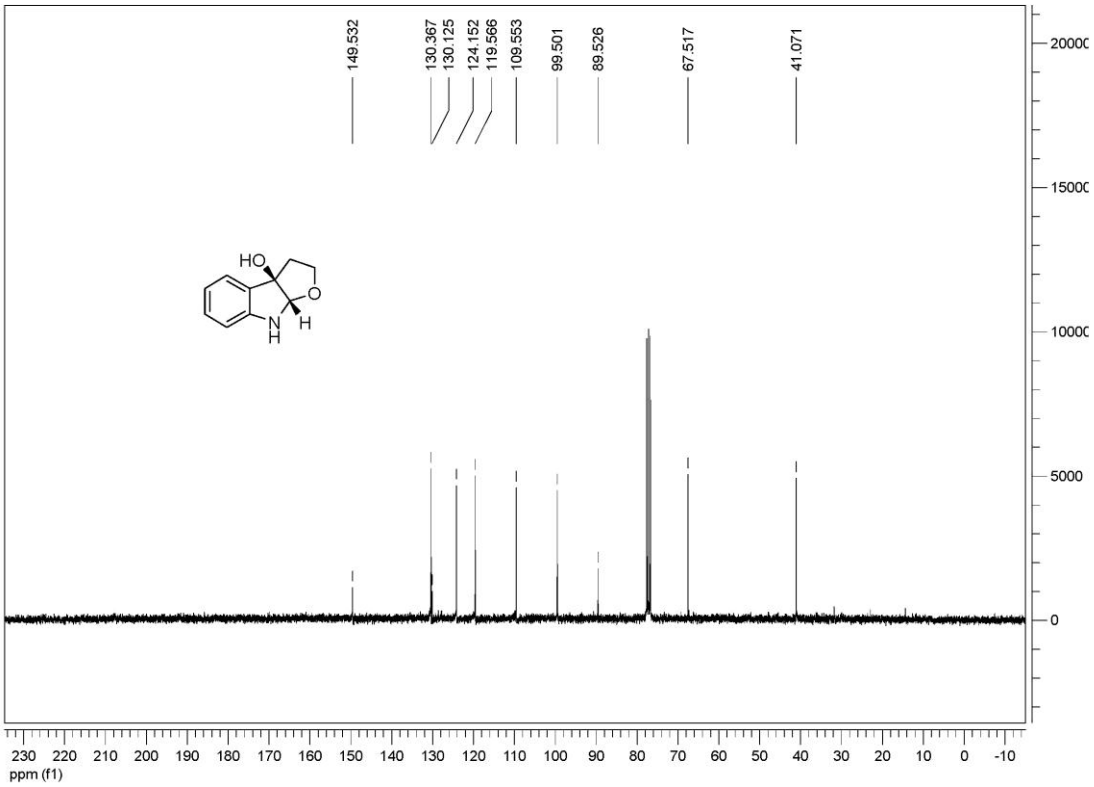
^{1}H NMR of **10**



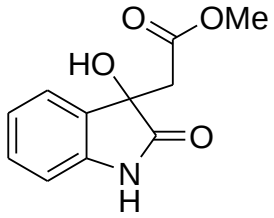
^{13}C NMR of **10**

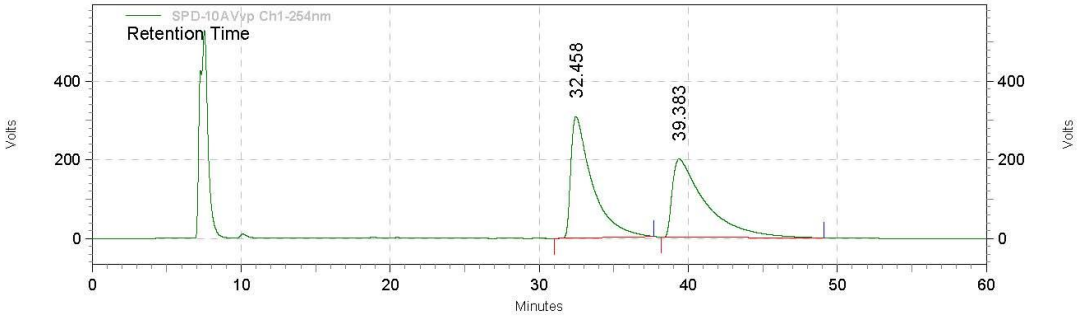


^{1}H NMR of **11**



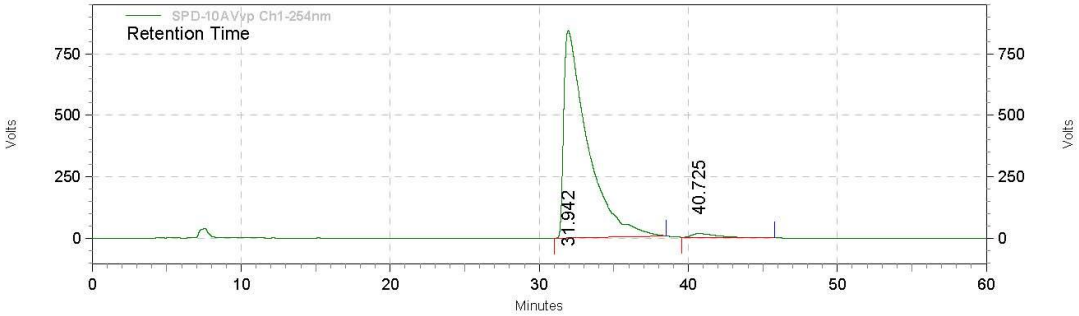
^{13}C NMR of **11**





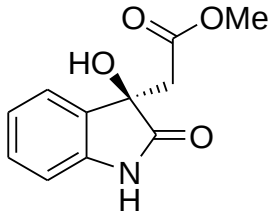
SPD-10AVvp
Ch1-254nm
Results

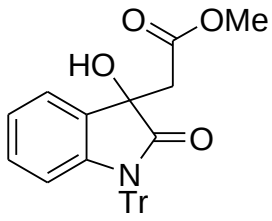
Retention Time	Area	Area %	Height	Height %
32.458	30708247	50.10	309008	60.87
39.383	30590716	49.90	198621	39.13
Totals	61298963	100.00	507629	100.00

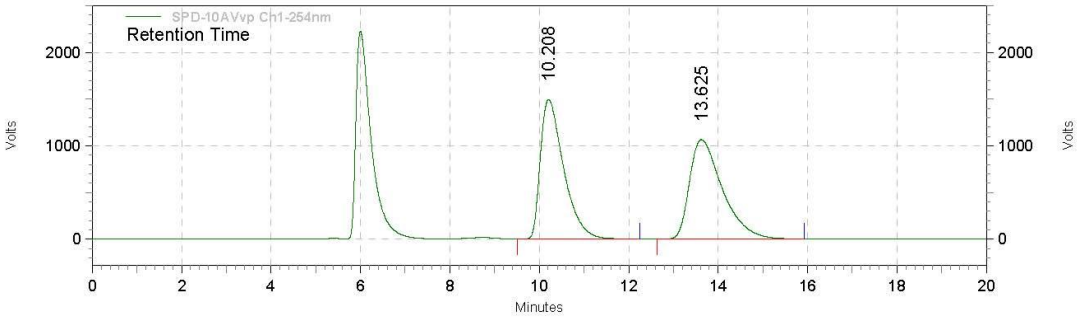


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
31.942	93472424	98.16	844661	98.22
40.725	1752787	1.84	15274	1.78
Totals	95225211	100.00	859935	100.00

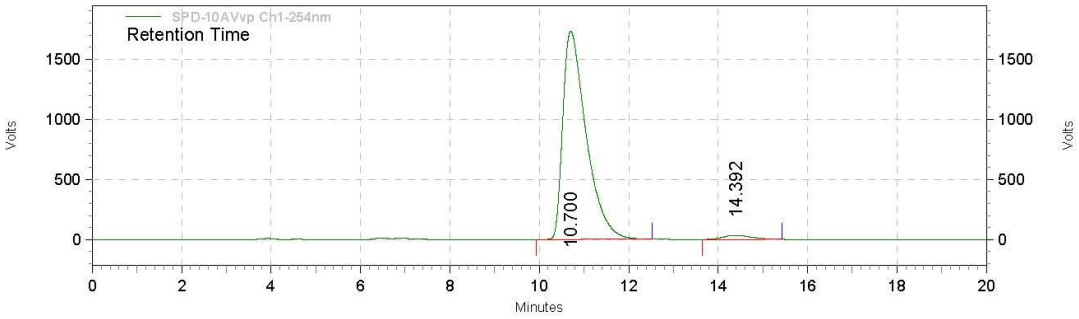






SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
10.208	53854485	49.85	1491566	58.36
13.625	54168814	50.15	1064324	41.64
Totals	108023299	100.00	2555890	100.00



SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
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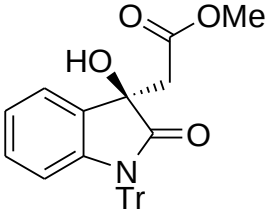
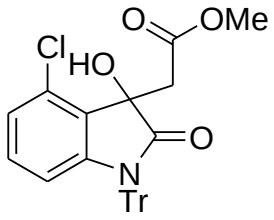
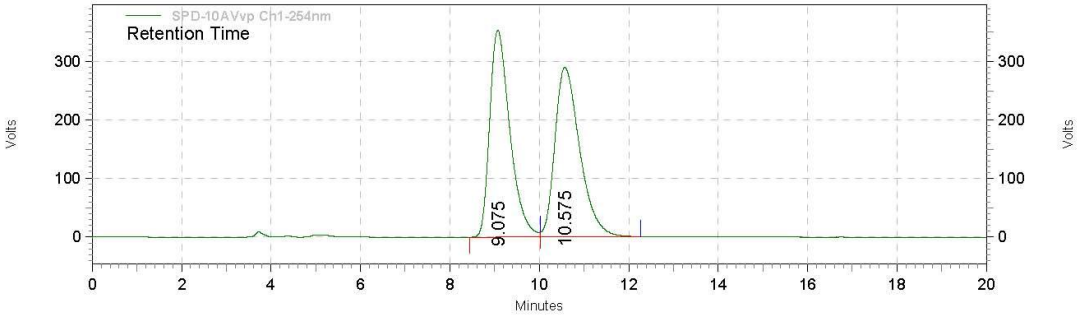
14.392	1493375	2.33	33704	1.91
Totals	64188487	100.00	1761929	100.00







SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
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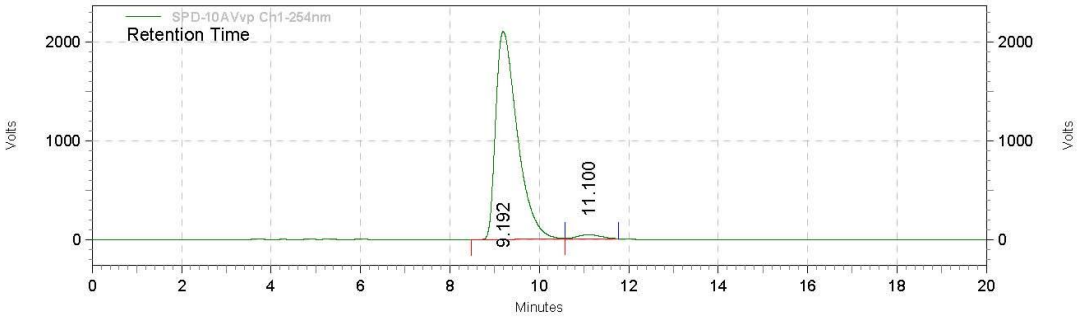
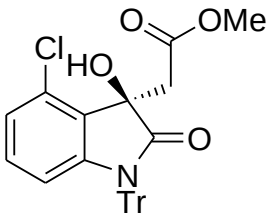
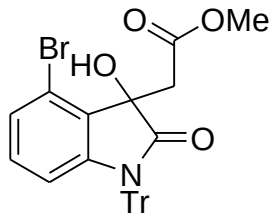
10.575	11192615	50.24	289671	45.06
Totals	22278301	100.00	642882	100.00

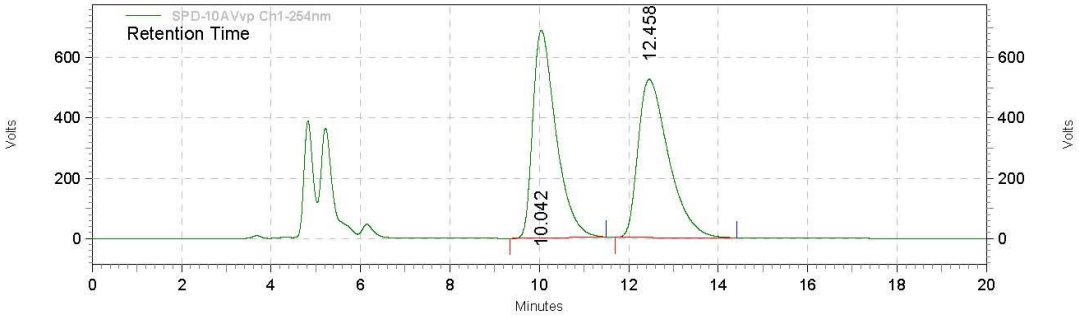


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
9.192	70555063	97.77	2103151	97.99
11.100	1612300	2.23	43171	2.01
Totals	72167363	100.00	2146322	100.00

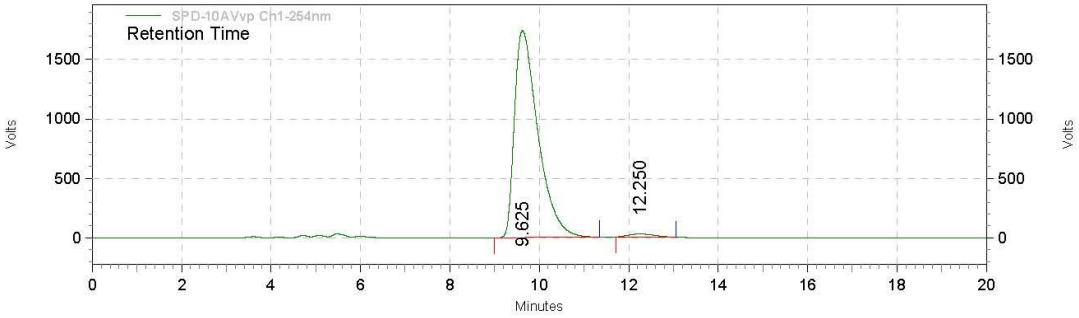






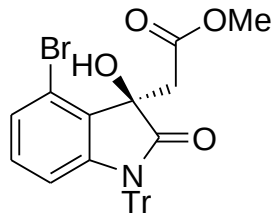
SPD-10AVvp
Ch1-254nm
Results

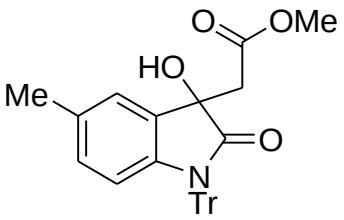
Retention Time	Area	Area %	Height	Height %
10.042	24842400	50.01	688034	56.72
12.458	24834375	49.99	525041	43.28
Totals	49676775	100.00	1213075	100.00

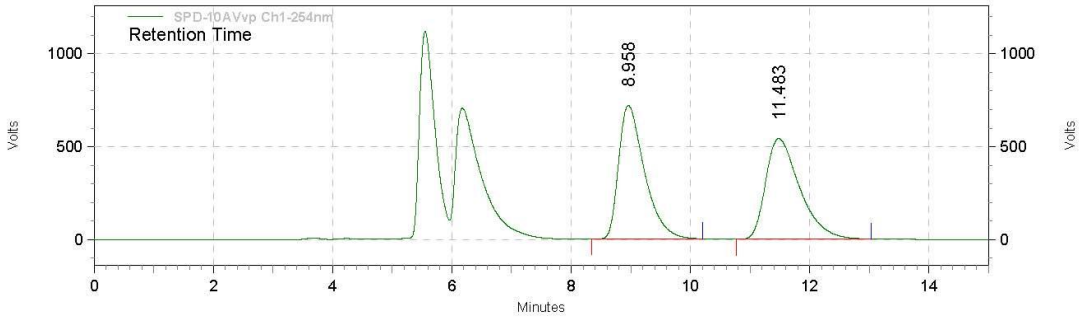


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
9.625	63499241	98.41	1742295	98.54
12.250	1028100	1.59	25731	1.46
Totals	64527341	100.00	1768026	100.00

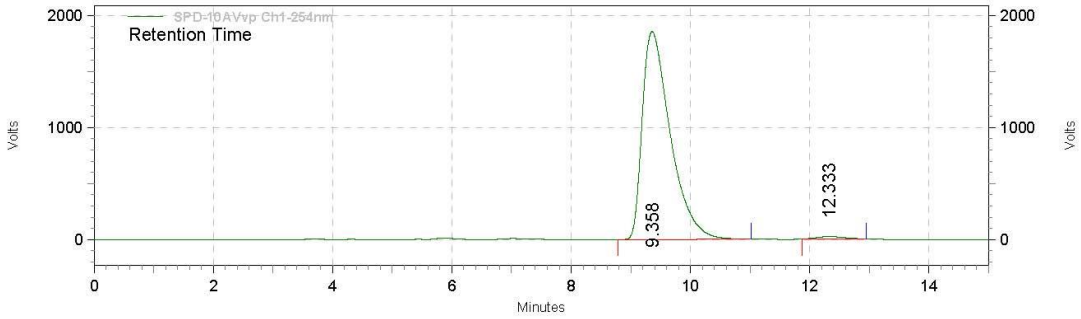






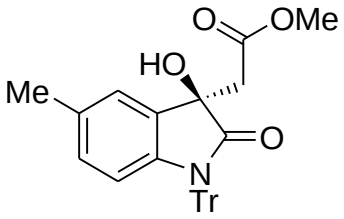
SPD-10AVvp
Ch1-254nm
Results

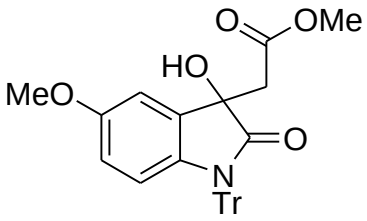
Retention Time	Area	Area %	Height	Height %
8.958	21568065	50.03	718351	56.98
11.483	21541515	49.97	542249	43.02
Totals	43109580	100.00	1260600	100.00

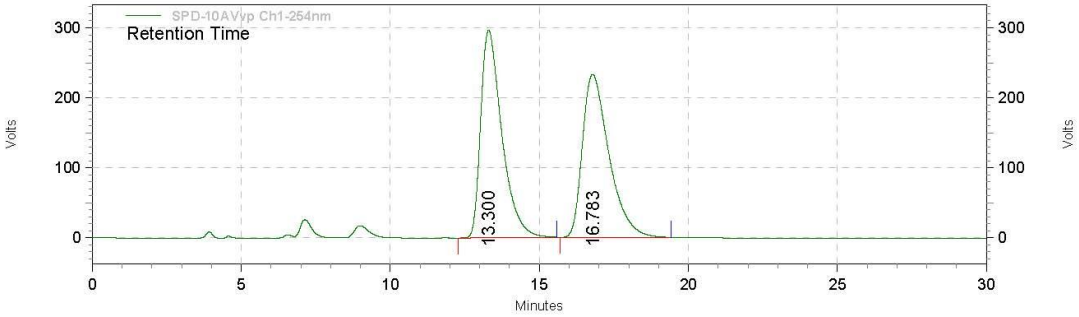


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
9.358	60674602	98.81	1855875	98.86
12.333	727665	1.19	21408	1.14
Totals	61402267	100.00	1877283	100.00

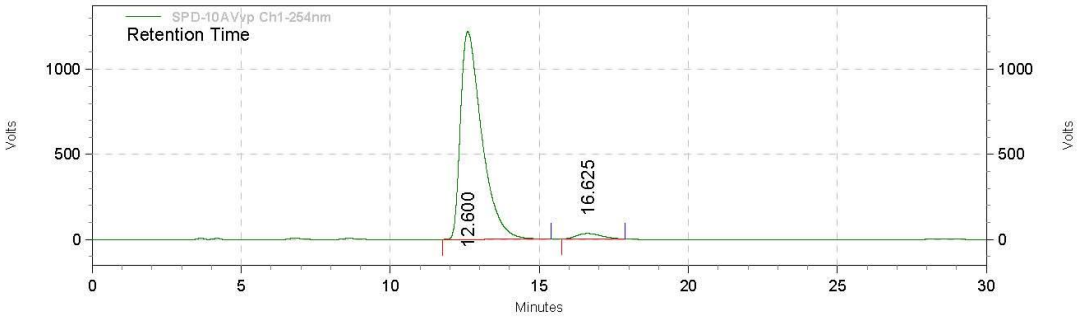






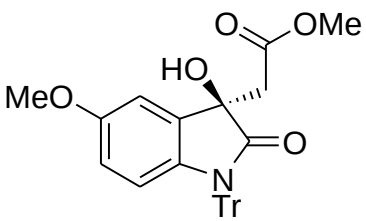
SPD-10AVvp
Ch1-254nm
Results

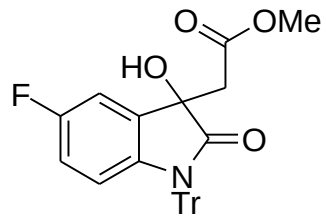
Retention Time	Area	Area %	Height	Height %
13.300	14632028	50.36	296318	55.99
16.783	14421834	49.64	232920	44.01
Totals	29053862	100.00	529238	100.00

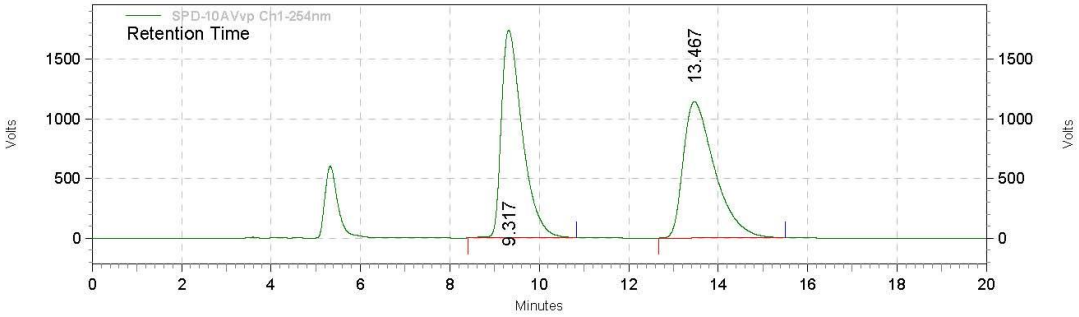


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
12.600	60603245	96.94	1221430	97.42
16.625	1913704	3.06	32332	2.58
Totals	62516949	100.00	1253762	100.00

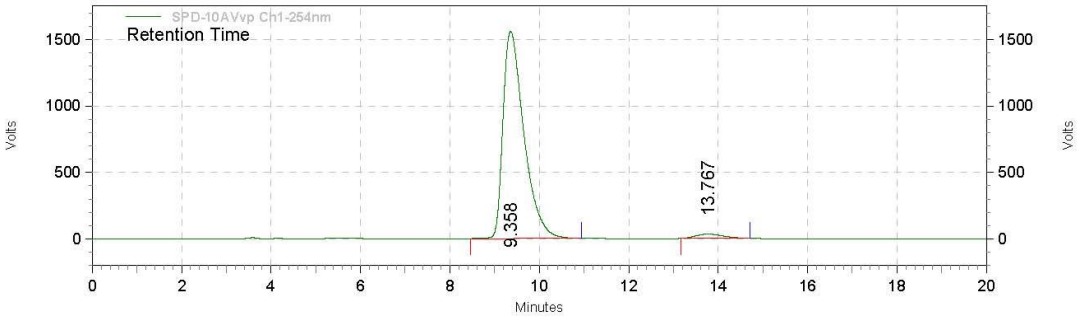






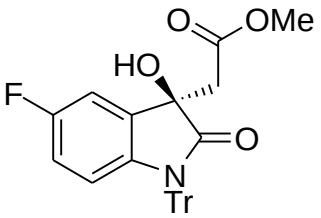
SPD-10AVvp
Ch1-254nm
Results

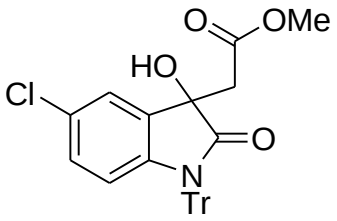
Retention Time	Area	Area %	Height	Height %
9.317	56569833	50.42	1736617	60.38
13.467	55636275	49.58	1139353	39.62
Totals	112206108	100.00	2875970	100.00

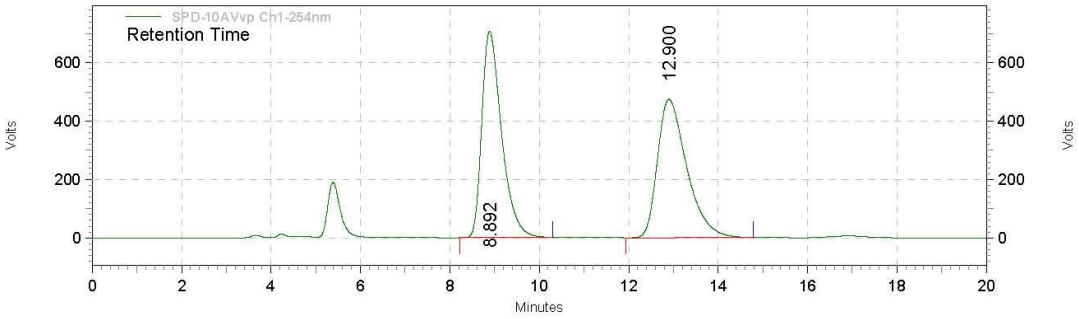


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
9.358	50576134	97.30	1557013	97.95
13.767	1404781	2.70	32564	2.05
Totals	51980915	100.00	1589577	100.00

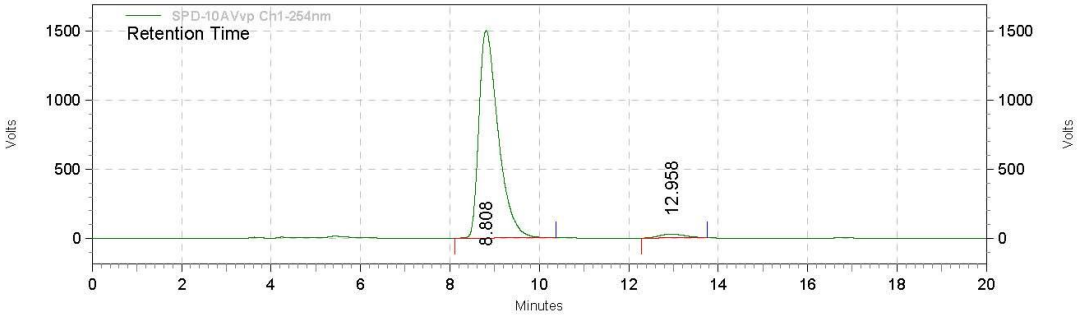






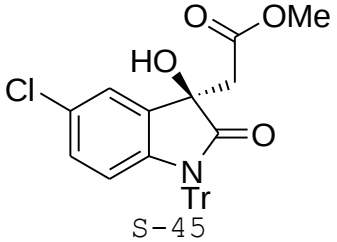
SPD-10AVvp
Ch1-254nm
Results

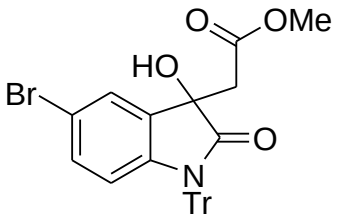
Retention Time	Area	Area %	Height	Height %
8.892	21615768	50.08	706123	59.83
12.900	21550640	49.92	474053	40.17
Totals	43166408	100.00	1180176	100.00

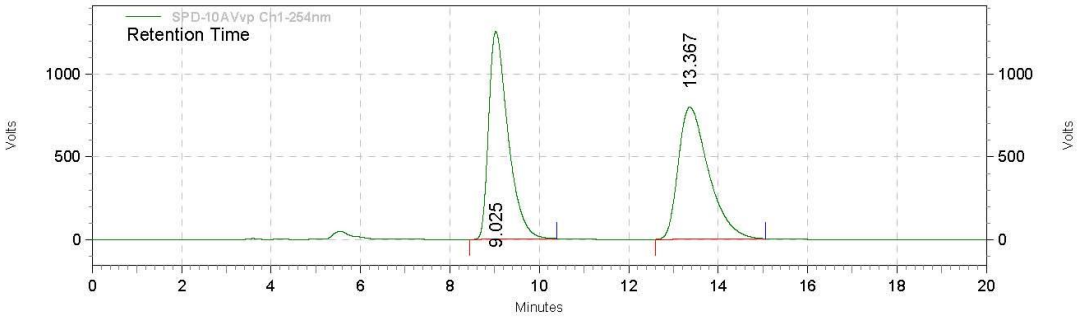


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
8.808	44988300	97.57	1505994	98.17
12.958	1119924	2.43	28006	1.83
Totals	46108224	100.00	1534000	100.00

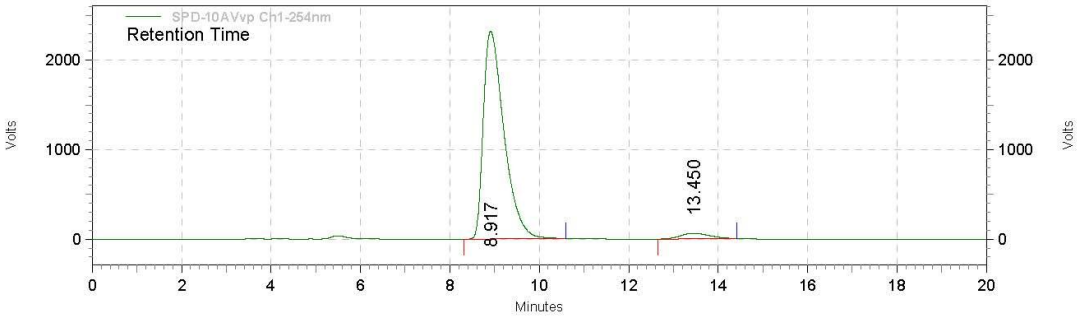






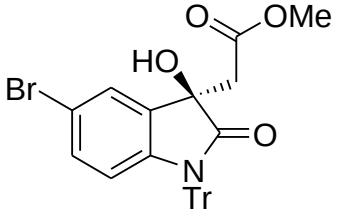
SPD-10AVvp
Ch1-254nm
Results

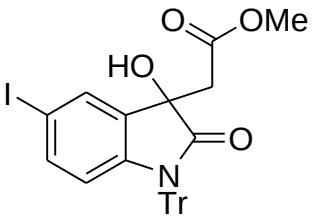
Retention Time	Area	Area %	Height	Height %
9.025	37750398	50.03	1256751	61.09
13.367	37698967	49.97	800601	38.91
Totals	75449365	100.00	2057352	100.00

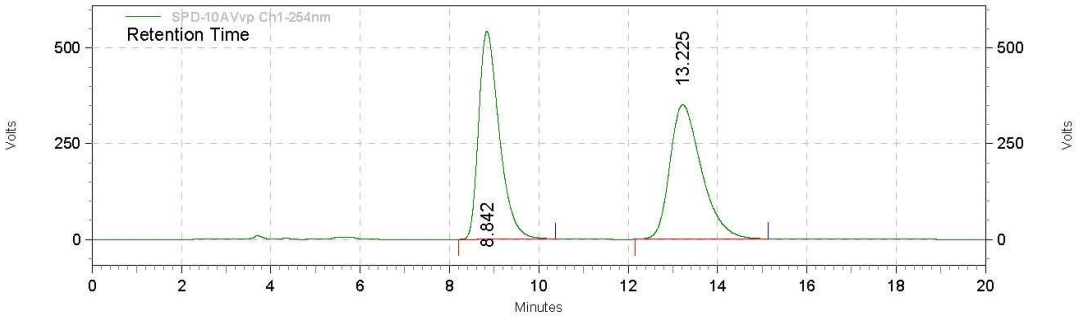


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
8.917	74562993	96.37	2320387	97.39
13.450	2809969	3.63	62096	2.61
Totals	77372962	100.00	2382483	100.00

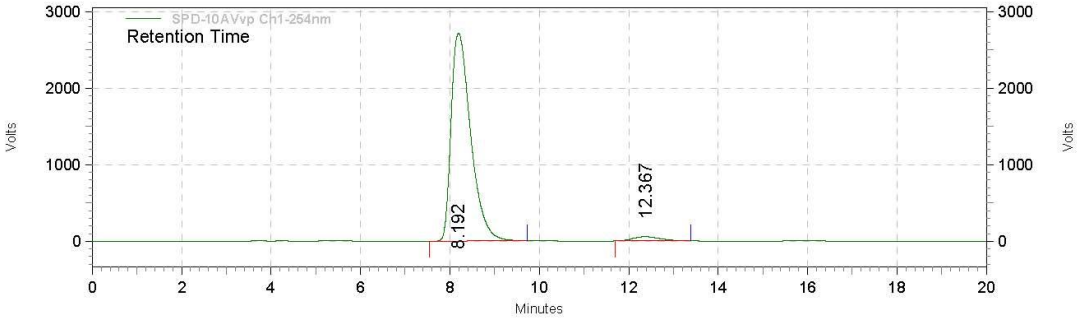






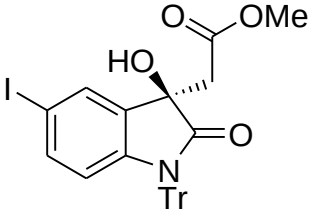
SPD-10AVvp
Ch1-254nm
Results

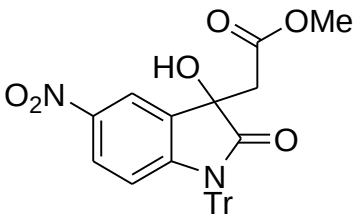
Retention Time	Area	Area %	Height	Height %
8.842	17385204	50.12	542624	60.71
13.225	17298523	49.88	351157	39.29
Totals	34683727	100.00	893781	100.00

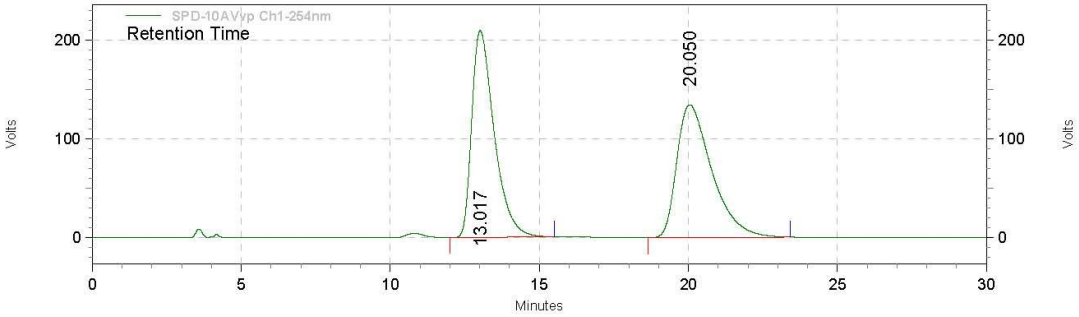


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
8.192	82426461	97.18	2711745	97.97
12.367	2389971	2.82	56097	2.03
Totals	84816432	100.00	2767842	100.00

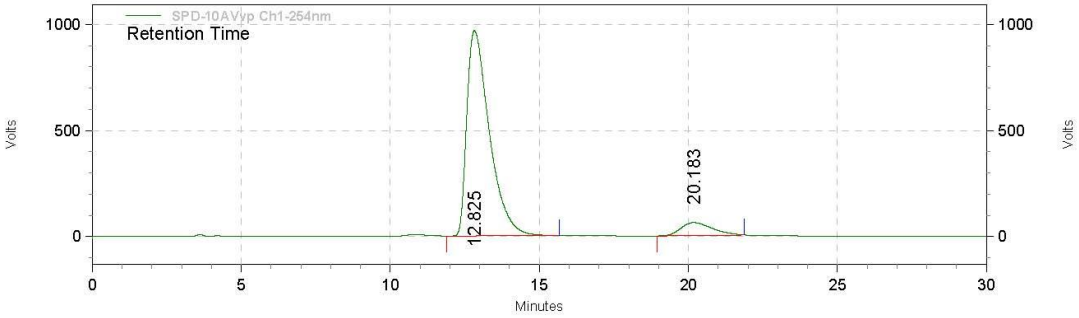






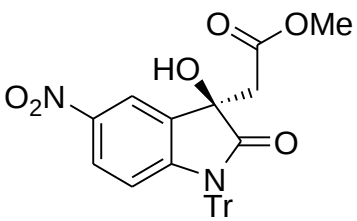
SPD-10AVvp
Ch1-254nm
Results

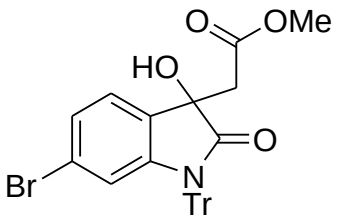
Retention Time	Area	Area %	Height	Height %
13.017	10959181	49.96	209649	60.90
20.050	10978224	50.04	134611	39.10
Totals	21937405	100.00	344260	100.00

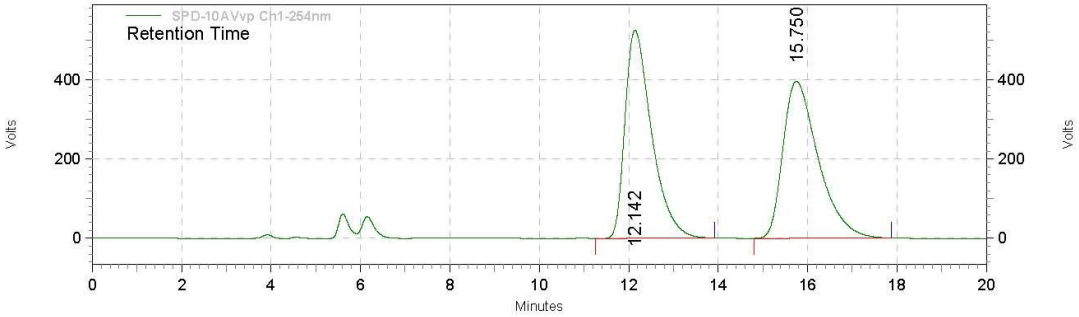


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
12.825	50921746	91.96	969074	94.07
20.183	4453589	8.04	61110	5.93
Totals	55375335	100.00	1030184	100.00



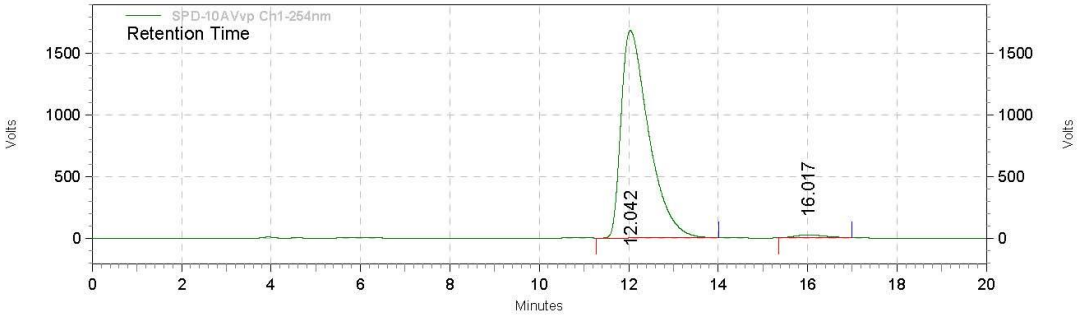




SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
12.142	21664266	49.97	524088	56.94
15.750	21688044	50.03	396261	43.06

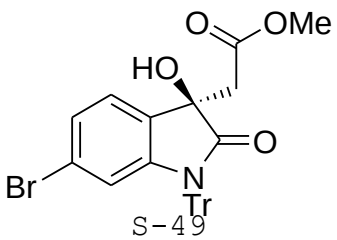
Totals	43352310	100.00	920349	100.00

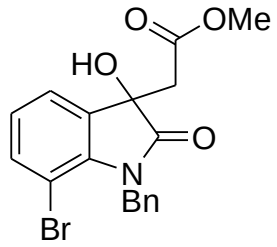


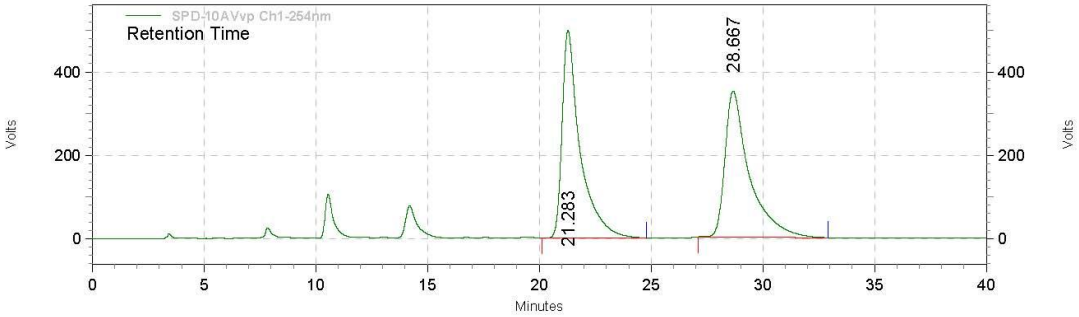
SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
12.042	72208595	98.50	1688011	98.66
16.017	1096059	1.50	22955	1.34

Totals	73304654	100.00	1710966	100.00

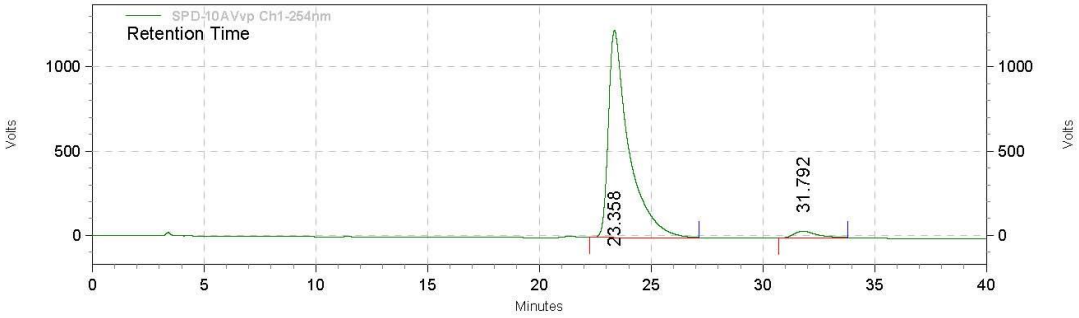






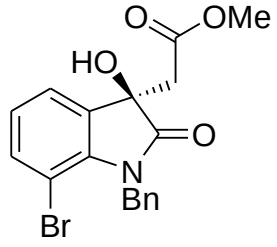
SPD-10AVvp
Ch1-254nm
Results

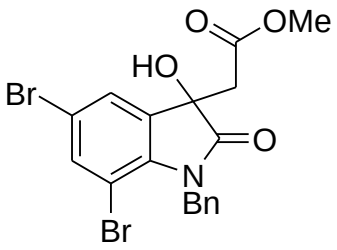
Retention Time	Area	Area %	Height	Height %
21.283	27696650	51.06	498155	58.68
28.667	26548120	48.94	350722	41.32
Totals	54244770	100.00	848877	100.00

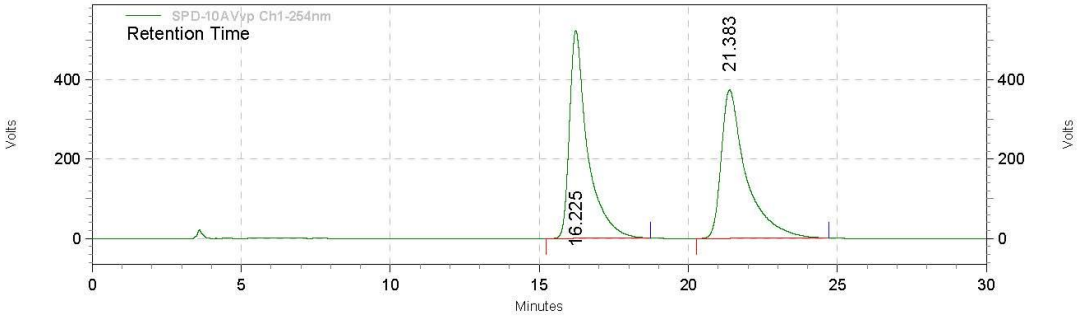


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
23.358	77574547	96.43	1228466	96.77
31.792	2871566	3.57	40986	3.23
Totals	80446113	100.00	1269452	100.00

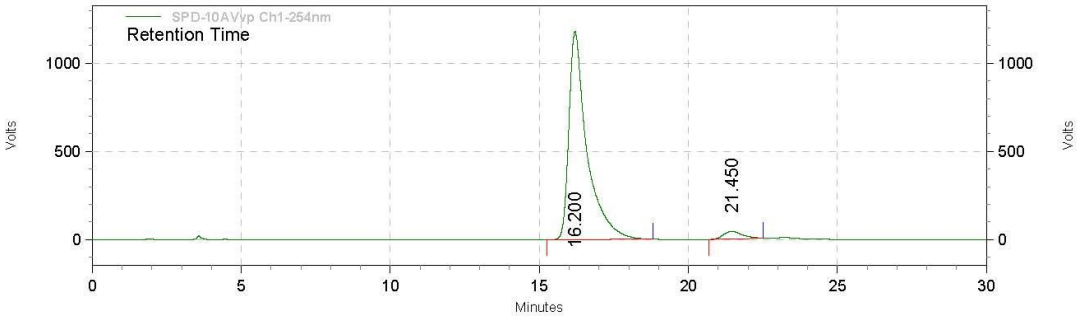






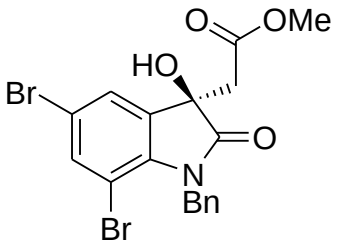
SPD-10AVvp
Ch1-254nm
Results

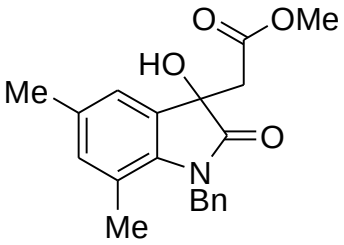
Retention Time	Area	Area %	Height	Height %
16.225	22044345	50.41	523551	58.34
21.383	21688563	49.59	373895	41.66
Totals	43732908	100.00	897446	100.00

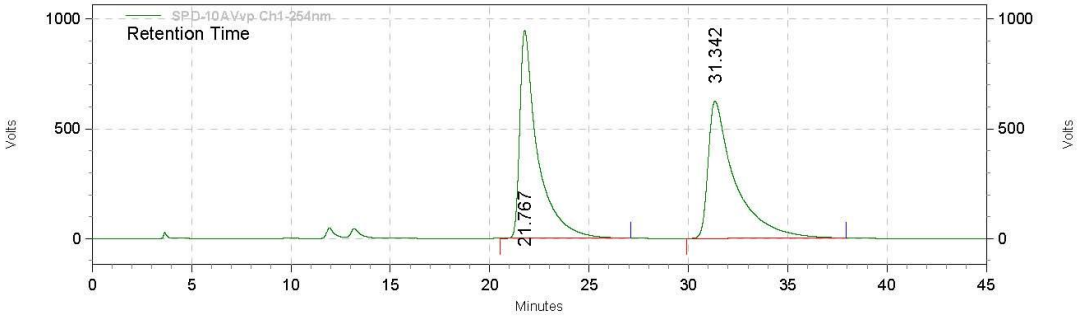


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
16.200	49696575	96.32	1180557	96.47
21.450	1900619	3.68	43215	3.53
Totals	51597194	100.00	1223772	100.00

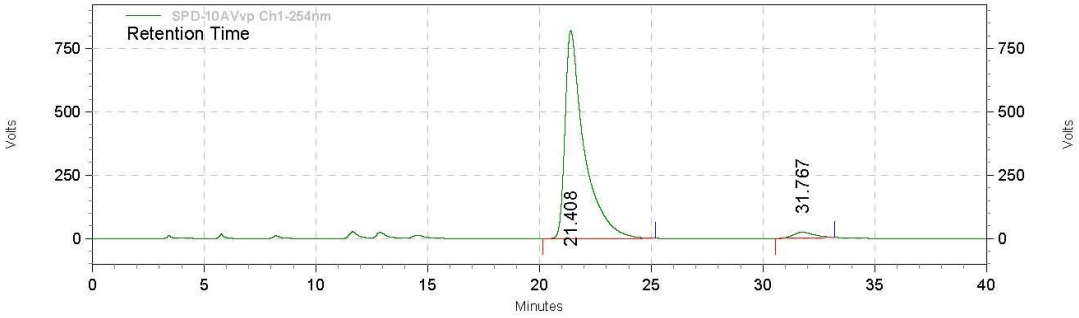






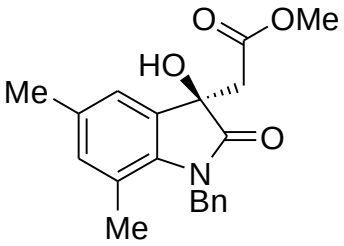
SPD-10AVvp
Ch1-254nm
Results

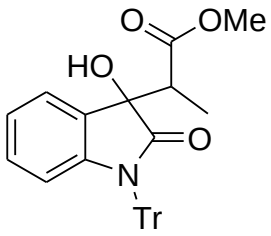
Retention Time	Area	Area %	Height	Height %
21.767	58689326	50.04	946878	60.29
31.342	58592579	49.96	623682	39.71
Totals	117281905	100.00	1570560	100.00

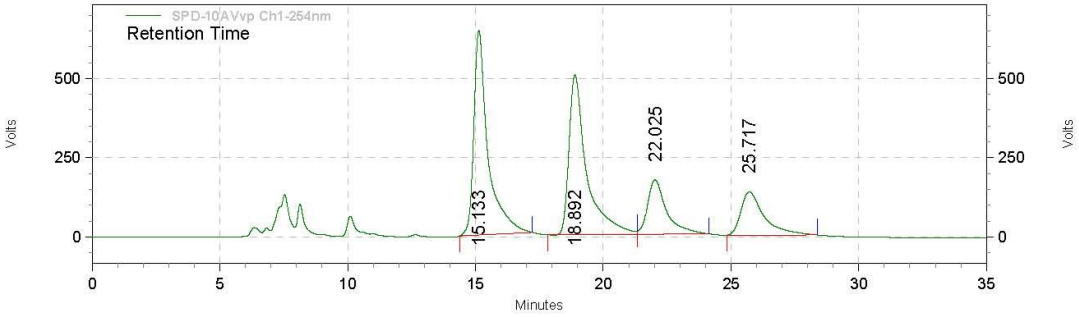


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
21.408	48645441	97.10	820902	97.34
31.767	1453690	2.90	22440	2.66
Totals	50099131	100.00	843342	100.00



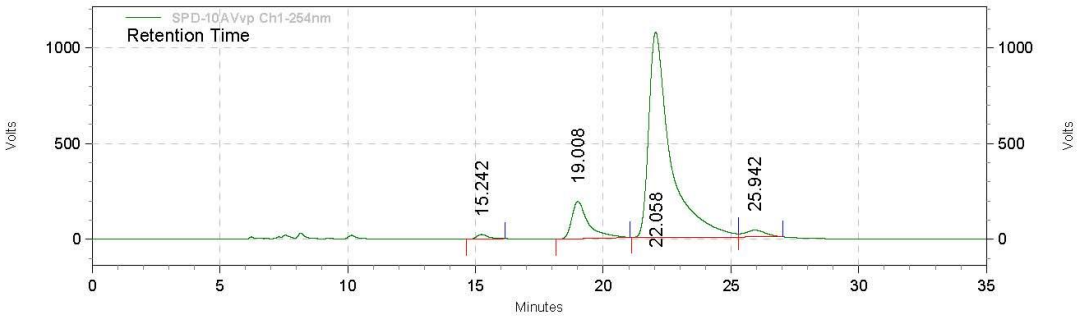




SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
15.133	24909407	37.40	645488	44.24
18.892	24182057	36.31	504753	34.59
22.025	8866769	13.31	171928	11.78
25.717	8647666	12.98	136928	9.38

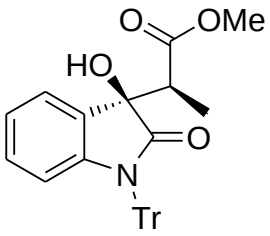
Totals				
	66605899	100.00	1459097	100.00

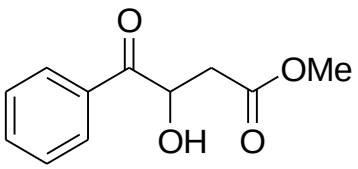


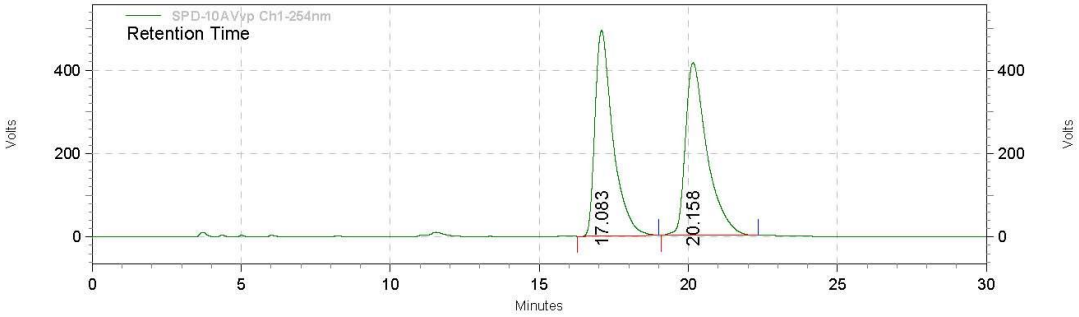
SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
15.242	719788	0.96	23126	1.75
19.008	8804862	11.74	194452	14.69
22.058	63437002	84.61	1070816	80.89
25.942	2012941	2.68	35473	2.68

Totals				
	74974593	100.00	1323867	100.00

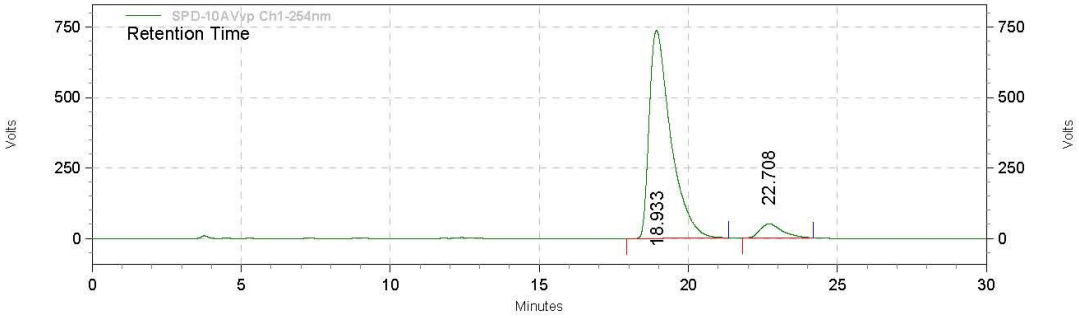






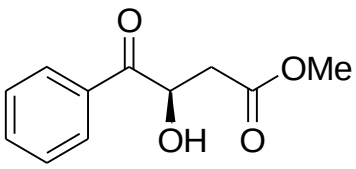
SPD-10AVvp
Ch1-254nm
Results

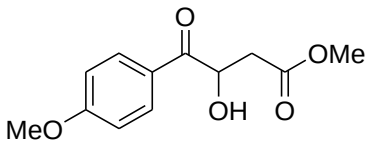
Retention Time	Area	Area %	Height	Height %
17.083	20548192	49.46	493422	54.36
20.158	20993883	50.54	414201	45.64
Totals	41542075	100.00	907623	100.00

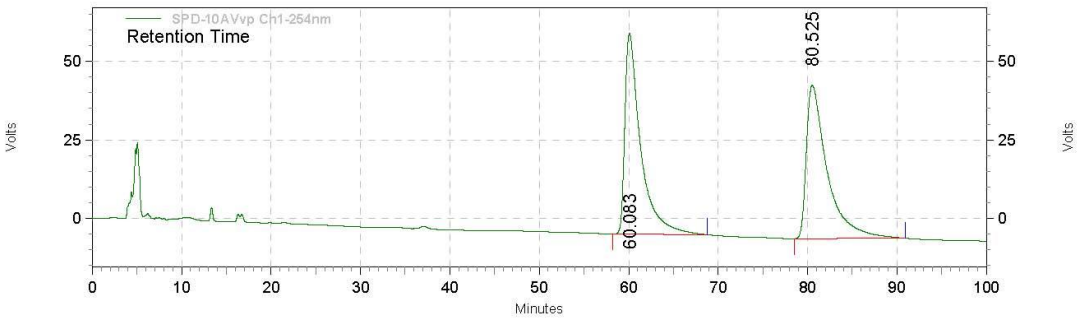


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
18.933	36905620	93.45	737755	93.64
22.708	2588255	6.55	50067	6.36
Totals	39493875	100.00	787822	100.00



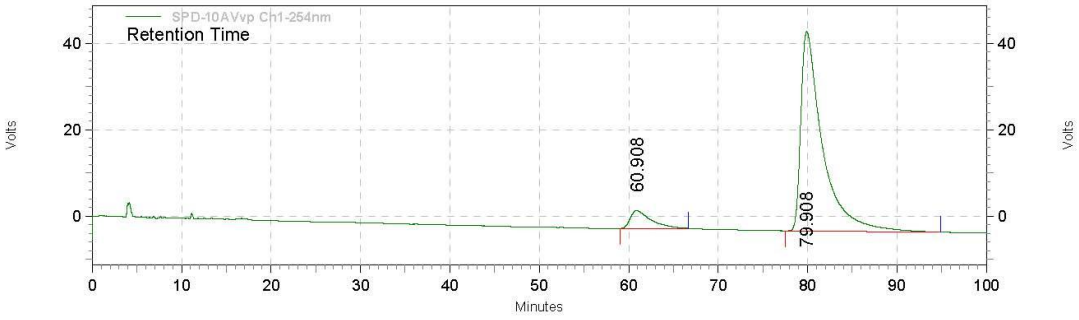




SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
60.083	7835175	49.69	63867	56.66
80.525	7934106	50.31	48855	43.34

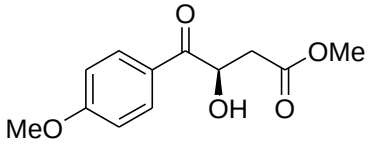
Totals				
	15769281	100.00	112722	100.00

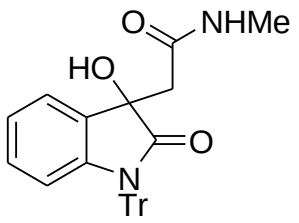


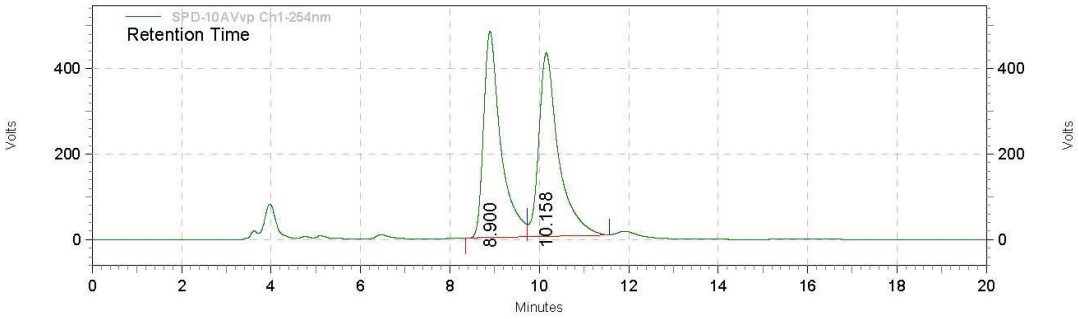
SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
60.908	664673	7.85	4123	8.18
79.908	7805207	92.15	46268	91.82

Totals				
	8469880	100.00	50391	100.00

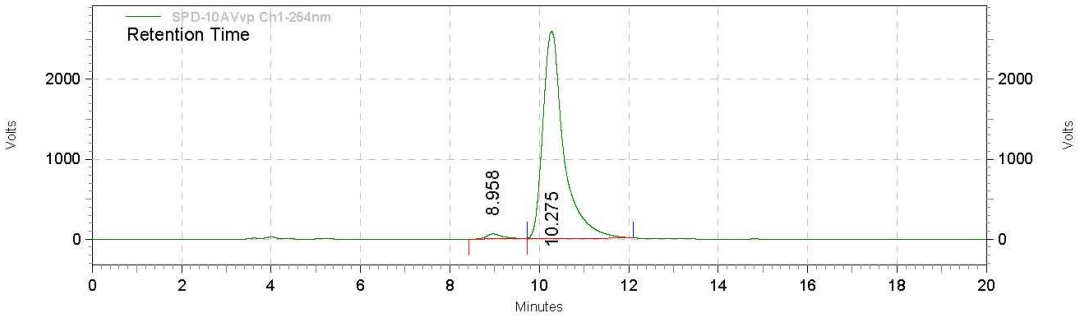






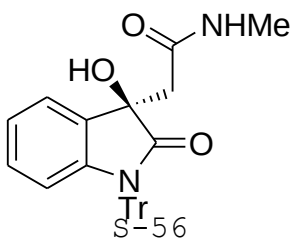
SPD-10AVvp
Ch1-254nm
Results

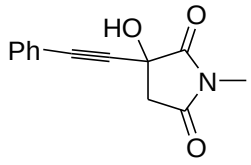
Retention Time	Area	Area %	Height	Height %
8.900	12825943	49.43	481591	52.90
10.158	13123457	50.57	428776	47.10
Totals	25949400	100.00	910367	100.00

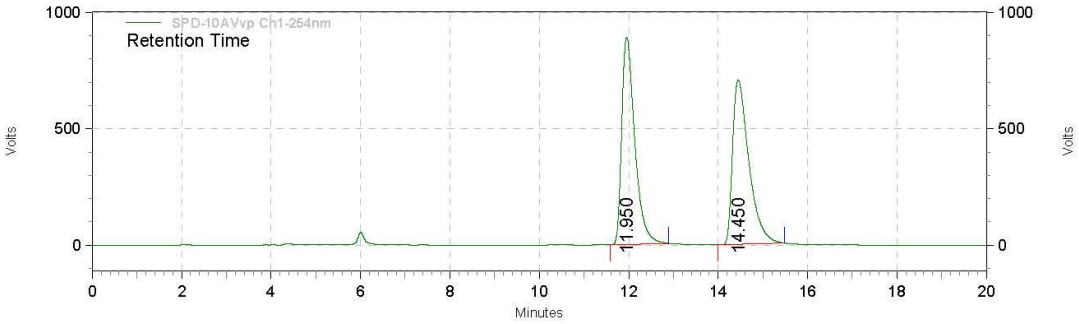


SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
8.958	1547190	1.82	61681	2.33
10.275	83547418	98.18	2589906	97.67
Totals	85094608	100.00	2651587	100.00

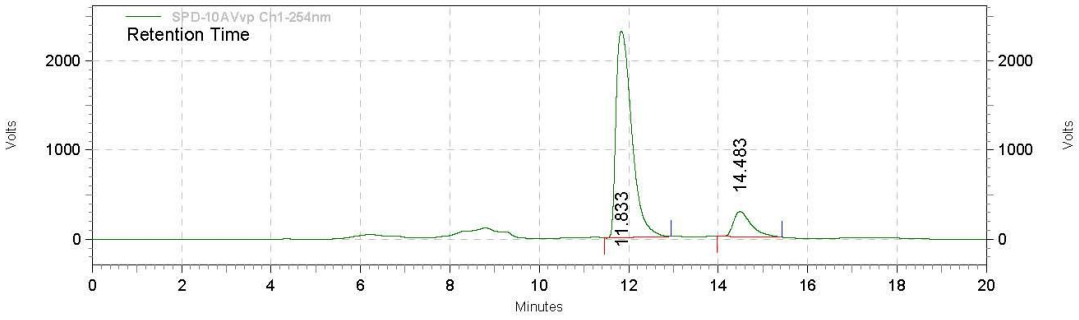






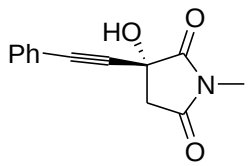
SPD-10AVvp
Ch1-254nm
Results

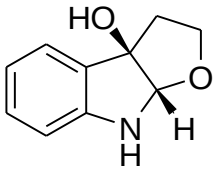
Retention Time	Area	Area %	Height	Height %
11.950	17980478	50.14	890060	55.77
14.450	17879392	49.86	705947	44.23
Totals	35859870	100.00	1596007	100.00



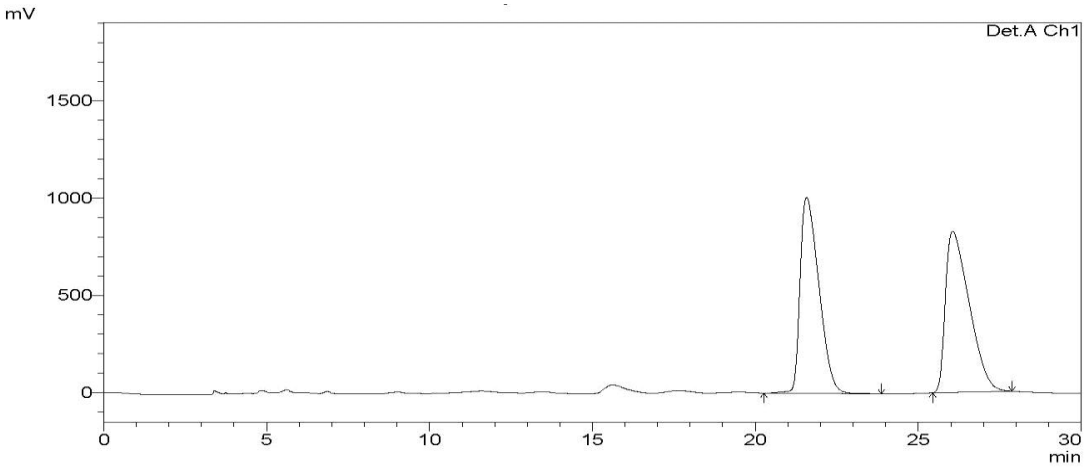
SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
11.833	56403955	87.79	2313695	89.12
14.483	7845276	12.21	282320	10.88
Totals	64249231	100.00	2596015	100.00





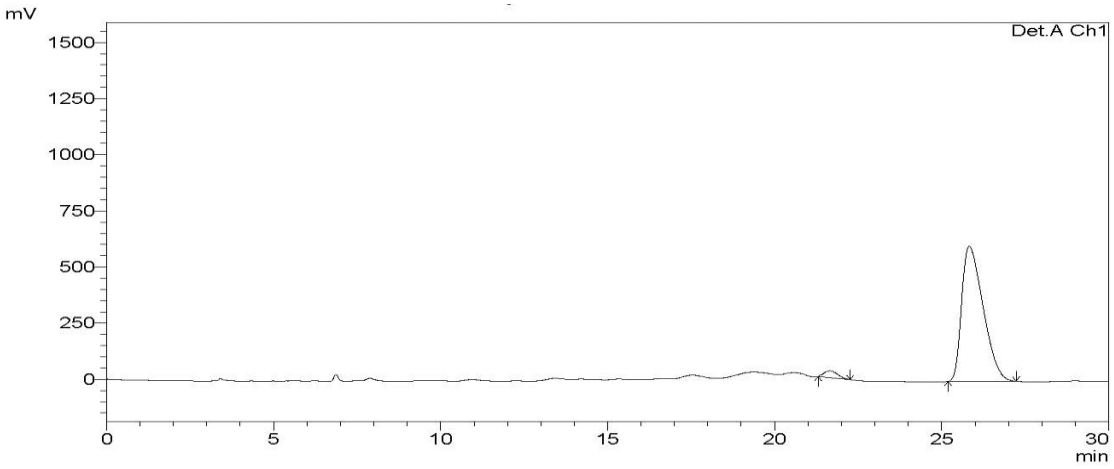
Racemic



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.570	40305219	1007243	49.112	54.899
2	26.053	41762628	827470	50.888	45.101
Total		82067847	1834713	100.000	100.000



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.645	844432	29931	3.056	4.743
2	25.824	26785377	601146	96.944	95.257
Total		27629809	631077	100.000	100.000