Supplementary Material (ESI) for Chemical Communications

Enantioselective Heterocyclic Synthesis of Spiro Chromanone-Thiochroman Complexes Catalyzed by a Bifunctional Indene Catalyst

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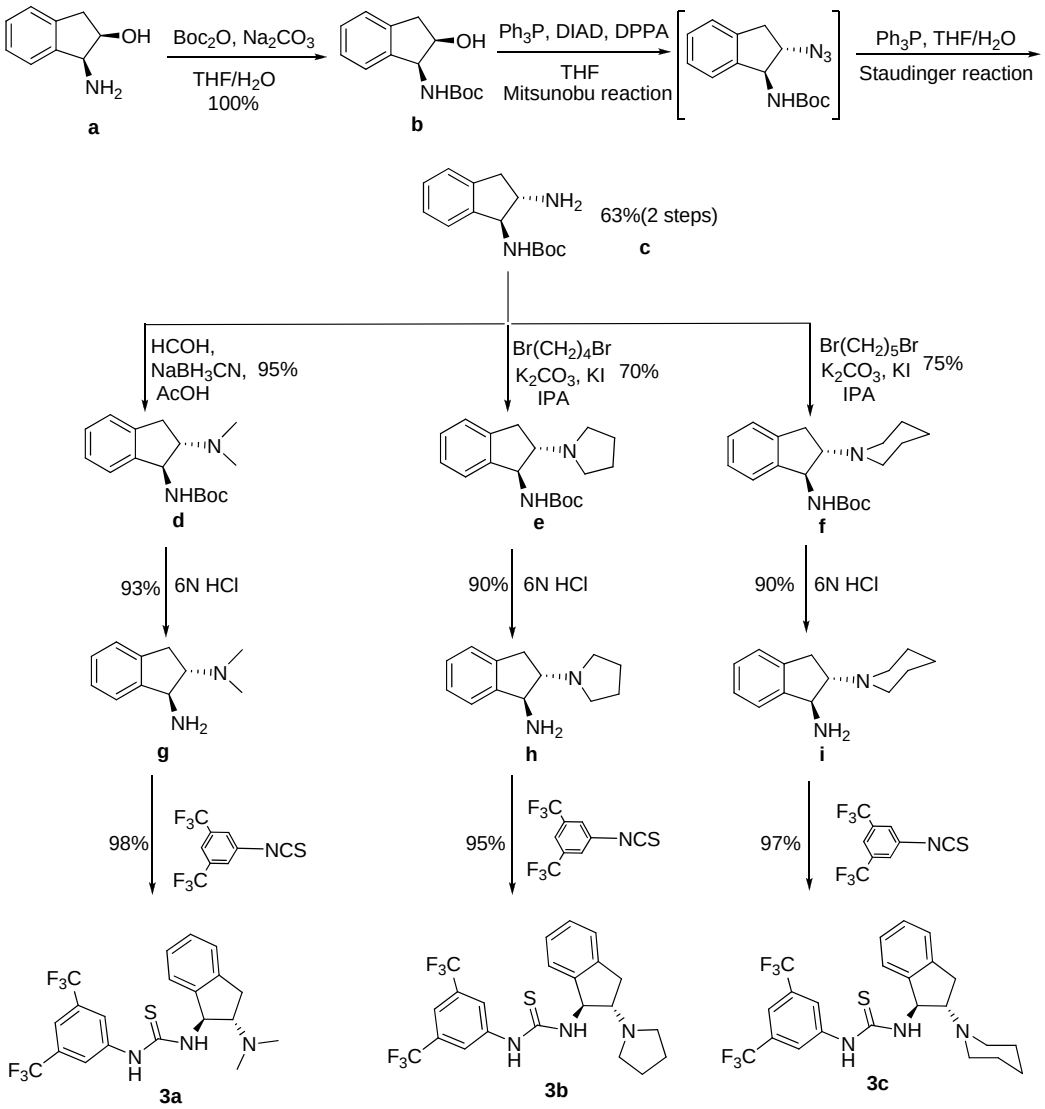
Supporting Information

Contents:

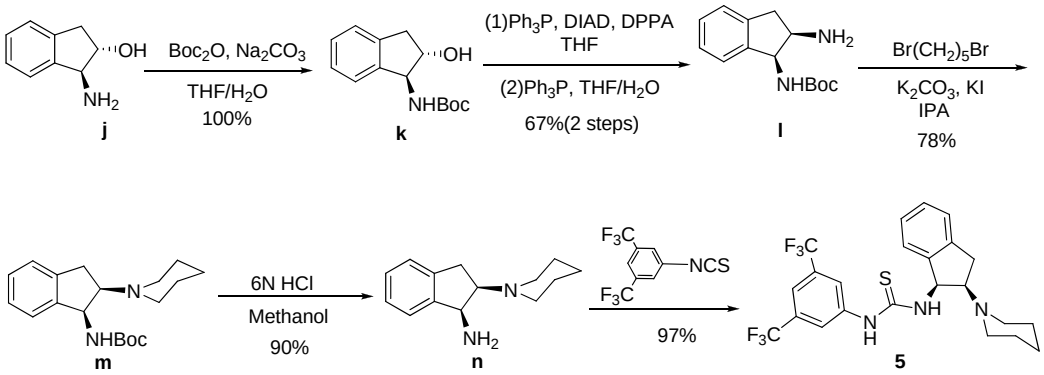
1. General Information

Chemicals and solvents were purchased from commercial suppliers and used as received. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker ACF300 (300 MHz) or AMX500 (500 MHz) spectrometer. Chemical shifts were reported in parts per million (ppm), and the residual solvent peak was used as an internal reference: proton (chloroform δ 7.26), carbon (chloroform δ 77.0) or tetramethylsilane (TMS δ 0.00) was used as a reference. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), bs (broad singlet). Coupling constants were reported in Hertz (Hz). Low resolution mass spectra were obtained on a Finnigan/MAT LCQ spectrometer in ESI mode, and a Finnigan/MAT 95XL-T mass spectrometer in EI mode. All high resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. For thin layer chromatography (TLC), Merck pre-coated TLC plates (Merck 60 F254) were used, and compounds were visualized with a UV light at 254 nm. Further visualization was achieved by staining with iodine, or ninhydrin followed by heating using a heat gun. Flash chromatography separations were performed on Merck 60 (0.040-0.063 mm) mesh silica gel. The enantiomeric excesses of products were determined by chiral phase HPLC analysis. Optical rotations were recorded on Jasco DIP-1000 polarimeter.

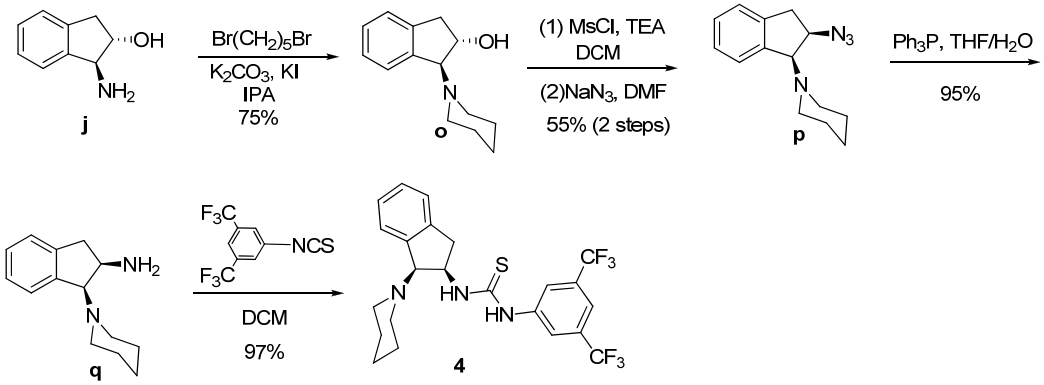
2. Preparation of the Catalysts



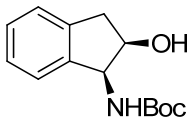
Scheme 1, Synthetic route for catalyst 3a, 3b, and 3c



Scheme 2, Synthetic route for catalyst 5

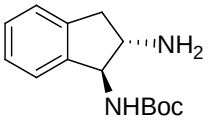


Scheme 3, Synthetic route for catalyst 4



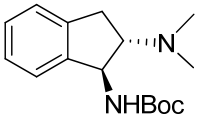
***tert*-butyl (1*S*,2*R*)-2-hydroxy-2,3-dihydro-1*H*-inden-1-ylcarbamate (b)**[1]**.** A solution of $(Boc)_2O$ (2.4 g, 11 mmol) in THF (5 ml) was added to the mixture of the amino alcohol **a** (1.5 g, 10 mmol) and sodium carbonate (2.12 g, 20 mmol) in THF/H_2O (1:1, 60 ml) at 0°C. The mixture was stirred at 0°C for 1h and then at room temperature for another two 2h (TLC was used to monitor the reaction). Water

(30 ml) was added to the mixture upon completion. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (3 x 50 ml). The combined organic layers was washed with brine (60 ml) and dried with anhydrous $MgSO_4$ for 1h. It was then filtered and the solvent was removed under vacuum to give the product (2.5 g) with quantitative yield. It was sufficiently pure for the next step. The pure product was obtained by purification with silica gel chromatography. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.3-7.29 (m, 1H), 7.24-7.22 (m, 3H), 5.11 (d, *J* = 28.7 Hz, 2H), 4.60 (s, 1H), 3.13 (dd, *J* = 16.6, 5.2 Hz, 1H), 2.93 (dd, *J* = 16.6, 1.7 Hz, 1H), 1.96 (s, 1H), 1.51 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 156.30, 140.82, 139.82, 128.18, 127.12, 125.33, 124.47, 79.88, 73.65, 58.89, 39.41, 28.39; HRMS (ESI) calcd for $C_{14}H_{19}NO_3$ ($M + H^+$) 249.1365, found 249.1367.

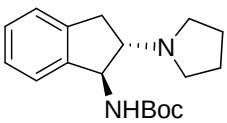


***tert*-butyl (1*S*,2*S*)-2-amino-2,3-dihydro-1*H*-inden-1-ylcarbamate (c)[2].** Diisopropyl azodicarboxylate (3.0 g, 15 mmol) was added to a stirred solution of compound **b** (2.5 g, 10 mmol) and triphenylphosphane (3.15 g, 12 mmol) in THF (50 mL) at 0 °C *via* syringe under nitrogen atmosphere. After 10 min, diphenylphosphoryl azide (DPPA) (4.1 g, 15 mmol) was added dropwise by syringe. The solution was stirred overnight at room temperature. After that, triphenylphosphane (5.3 g, 20 mmol) was added in one portion, and the solution was stirred at room temperature for 2 hours. Water (5 mL) was then added and the solution was heated at 50°C for 6 h. The reaction mixture was concentrated and the residue was purified by silica gel chromatography (eluting with 1:1 EtOAc-DCM then 1:10 methanol-DCM) to obtain the white solid product (1.57g, 63% yield, two steps). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.20-7.19 (d, *J* = 5.9 Hz, 4H), 4.85-4.63 (m, 2H), 3.42 (dd, *J* = 14.8, 7.5 Hz, 1H), 3.19 (dd,

J = 15.6, 7.4 Hz, 1H), 2.63 (dd, *J* = 15.6, 8.2 Hz, 1H), 1.75 (s, 2H), 1.49 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.29, 141.55, 140.60, 128.07, 126.94, 124.78, 123.80, 79.65, 64.52, 62.39, 39.25, 28.37; HRMS (ESI) calcd for $C_{14}H_{21}N_2O_2$ ($M + H^+$) 249.1603, found 249.1601.

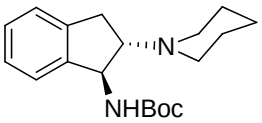


***tert*-butyl (1*S*,2*S*)-2-(dimethylamino)-2,3-dihydro-1*H*-inden-1-ylcarbamate (d)[3].** To a solution of compound **c** (0.75 g, 3 mmol) in 15 mL CH_3CN was added aqueous formaldehyde (37% w/w, 1.2 mL, 15 mmol), the solution was stirred at room temperature for 15 minutes. After that, $NaBH_3CN$ (0.38 g, 6 mmol) was added, followed 15 minutes stirring later by AcOH (1 mL). After 1 hour, the reaction mixture was dilute with 2% methanol-DCM (40 mL), washed with 1.0 M NaOH (3 x 30), dried by $MgSO_4$, and concentrated. The resulting residue was purified by silica gel chromatography (eluting with 1: 20 methanol-DCM) to afford the pure product (0.79 g, 95% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.26-7.15 (m, 4H), 5.20 (t, *J* = 8.4 Hz, 1H), 4.77 (d, *J* = 8.8 Hz, 1H), 3.03 (m, 2H), 2.87 (dd, *J* = 14.0, 7.4 Hz, 1H), 2.40 (s, 6H), 1.49 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.49, 142.61, 139.71, 127.90, 126.96, 124.59, 124.13, 79.49, 74.59, 57.25, 42.94, 33.68, 28.43; HRMS (ESI) calcd for $C_{16}H_{25}N_2O_2$ ($M + H^+$) 277.1916, found 277.1918.

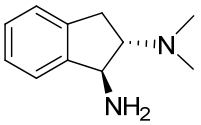


***tert*-butyl (1*S*,2*S*)-2-(pyrrolidin-1-yl)-2,3-dihydro-1*H*-inden-1-ylcarbamate (e)[4].** Compound **3c** (0.75 g, 3 mmol), 1,4-dibromobutane (0.78 g, 3.6 mmol), potassium carbonate (1.08 g, 7.8 mmol),

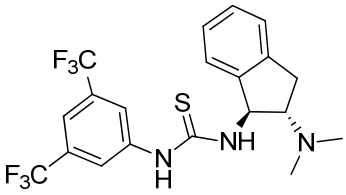
potassium iodide (0.1 g, 0.6 mmol) and 10 mL iso-propanol were added into a sealed tube. The mixture was heated at 80 °C for 48hrs and then allowed to cool to room temperature. The mixture was filtered and washing with DCM, the filtrate was concentrated and the resulting residue was purified by silica gel chromatography (eluting with 1:5 EtOAc- hexane then 1:10 methanol-DCM) to obtain the product **5** (0.64 g, 70% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.26-7.16 (m, 4H), 5.25-5.22 (m, 1H), 4.81 (d, *J* = 9.1 Hz, 1H), 3.17 (dd, *J* = 18.9, 11.0 Hz, 1H), 3.01-2.98 (m, 2H), 2.80-2.75 (m, 4H), 1.85 (s, 4H), 1.49 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 155.44, 142.25, 139.68, 128.06, 127.05, 124.56, 124.08, 79.61, 72.61, 59.61, 52.34, 36.24, 28.41, 23.36; HRMS (ESI) calcd for $C_{18}H_{27}N_2O_2$ ($M + H^+$) 303.2073, found 303.2062.



***tert*-butyl (1*S*,2*S*)-2-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-1-ylcarbamate (f)** [4]**.** Compound **c** (0.75 g, 3 mmol), 1,5-dibromopentane(0.83 g, 3.6 mmol), potassium carbonate (1.08 g, 7.8 mmol), potassium iodide (0.1 g, 0.6 mmol) and 10 mL iso-propanol were added into a sealed tube. The mixture was heated at 80 °C for 48hrs and then allowed to cool to room temperature. The mixture was filtered and washing with DCM, the filtrate was concentrated and the resulting residue was purified by silica gel chromatography (eluting with 1:10 EtOAc- hexane then 1:10 methanol-DCM) to obtain the product **5** (0.71g, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.28-7.12(m, 4H), 5.24 (dd, *J* = 13.7, 5.2 Hz, 1H), 4.89-4.87 (m, 1H), 3.05 (s, 2H), 2.87 (dd, *J* = 18.6, 11.7 Hz, 1H), 2.60 (dd, *J* = 23.2, 4.9 Hz, 4H), 1.61-1.59 (m, 4H), 1.48 (s, 11H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 155.41, 142.52, 139.97, 127.85, 126.88, 124.50, 124.14, 79.44, 74.64, 56.53, 51.60, 33.81, 28.41, 26.13, 24.45; HRMS (ESI) calcd for $C_{19}H_{29}N_2O_2$ ($M + H^+$) 317.2229, found 317.2227.

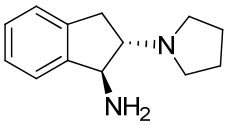


(1*S*,2*S*)-*N*^{2},*N*^{2}-dimethyl-2,3-dihydro-1*H*-indene-1,2-diamine (g). To a solution of compound **d** (0.7 g, 2.5 mmol) in 3 mL methanol was added 3 mL concentrated HCl solution. The mixture was stirred at room temperature for 5hrs, then 40% NaOH solution was added until pH of mixture was 14. After extraction by DCM (5 x 10 mL), the organic layer was combined and dried by $MgSO_4$, then concentrated. The pure product (0.41 g, 93% yield) was obtained by silica gel chromatography (very short column, eluting with 1:10 to 1:5 methanol-DCM). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.30 (d, *J* = 7.3 Hz, 1H), 7.23-7.16 (m, 3H), 4.23 (d, *J* = 7.3 Hz, 1H), 2.95 (dd, *J* = 13.4, 6.1 Hz, 1H), 2.89-2.80 (m, 2H), 2.40 (s, 6H), 1.81 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 145.30, 139.71, 127.30, 126.68, 124.59, 123.42, 77.76, 59.07, 43.02, 31.17; HRMS (ESI) calcd for $C_{11}H_{17}N_2$ ($M + H^+$) 177.1392, found 177.1391.

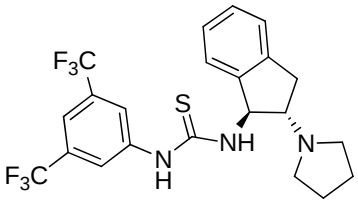


1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*S*,2*S*)-2-(dimethylamino)-2,3-dihydro-1*H*-inden-1-yl)thiourea (3a). To a solution of compound **g** (0.4 g, 2.27 mmol) in 10 mL DCM was added 1-isothiocyanato-3,5-bis(trifluoromethyl)benzene (0.59 g, 2.16 mmol) dropwise. The mixture was stirred at room temperature for 30min, reaction completed. The solvent was removed by rotary evaporation and pure product **3a** (1.0g, 98% yield) was obtained by silica gel chromatography (eluting with 1:5 EtOAc- hexane then 1: 10 methanol-DCM). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 12.84 (s, 1H), 8.10 (s, 2H), 7.61 (s, 1H), 7.42-7.28 (m, 4H), 6.86 (d, *J* = 3.2 Hz, 1H), 5.22 (m, 1H), 3.75 (q, *J* = 8.5

Hz, 1H), 3.08 (ddd, *J* = 44.1, 15.9, 9.0 Hz, 2H), 2.52 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 182.25, 141.95, 139.83, 137.38, 133.15, 132.06, 131.80, 131.53, 131.26, 129.35, 127.86, 125.57, 124.18, 123.46, 122.73, 122.01, 117.60, 74.45, 62.57, 40.75, 25.39; HRMS (ESI) calcd for $C_{20}H_{20}F_6N_3S$ (M + H^+) 448.1282, found 448.1277.

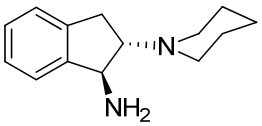


(1*S*,2*S*)-2-(pyrrolidin-1-yl)-2,3-dihydro-1*H*-inden-1-amine (h). To a solution of compound **e** (0.6 g, 2.0 mmol) in 3 mL methanol was added 3 mL concentrated HCl solution. The mixture was stirred at room temperature for 5hrs, then 40% NaOH solution was added until pH of mixture was 14. After extraction by DCM (5 x 10 mL), the organic layer was combined and dried by $MgSO_4$, then concentrated. The pure product (0.36 g, 90% yield) was obtained by silica gel chromatography (very short column, eluting with 1:10 to 1: 5 methanol-DCM). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.31-7.16 (m, 4H), 4.35 (d, *J* = 7.3 Hz, 1H), 3.10 (dd, *J* = 15.3, 7.7 Hz, 1H), 2.94 (dd, *J* = 15.3, 9.0 Hz, 1H), 2.86-2.81 (m, 5H), 2.41 (s, 2H), 1.86 (dd, *J* = 9.0, 3.9 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 145.37, 139.57, 127.40, 126.78, 124.37, 123.45, 75.99, 61.21, 52.62, 35.34, 23.23; HRMS (ESI) calcd for $C_{13}H_{19}N_2$ (M + H^+) 203.1548, found 203.1543.



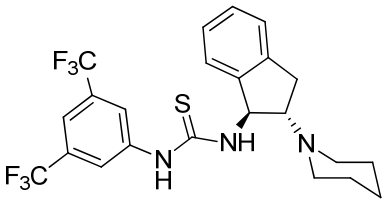
1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*S*,2*S*)-2-(pyrrolidin-1-yl)-2,3-dihydro-1*H*-inden-1-yl)thiou

rea (3b). To a solution of compound **e** (0.36 g, 1.78 mmol) in 10 mL DCM was added 1-isothiocyanato-3,5-bis(trifluoromethyl)benzene (0.51 g, 1.87 mmol) dropwise. The mixture was stirred at room temperature for 30min, reaction completed. The solvent was removed by rotary evaporation and pure product **3b** (0.84 g, 95% yield) was obtained by silica gel chromatography (eluting with 1:5 EtOAc- hexane then 1: 10 methanol-DCM). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 12.82 (s, 1H), 8.05 (s, 2H), 7.63 (s, 1H), 7.34 (ddd, *J* = 37.5, 21.3, 6.6 Hz, 4H), 6.68 (s, 1H), 5.25-5.23 (m, 1H), 4.03 (q, *J* = 8.2 Hz, 1H), 3.11 (dd, *J* = 8.7, 3.6 Hz, 2H), 2.94-2.84 (m, 4H), 1.90 (s, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 182.77, 141.97, 140.04, 137.91, 132.10, 131.83, 131.31, 129.36, 127.95, 125.62, 124.29, 123.49, 123.42, 122.12, 117.94, 77.25, 77.00, 76.75, 70.55, 63.20, 48.55, 26.86, 23.93; HRMS (ESI) calcd for $C_{22}H_{22}F_6N_3S$ $(M + H^+)$ 474.1439, found 474.1443.

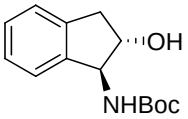


(1*S*,2*S*)-2-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-1-amine (i). To a solution of compound **f** (0.7 g, 2.2 mmol) in 3 mL methanol was added 3 mL concentrated HCl solution. The mixture was stirred at room temperature for 5hrs, then 40% NaOH solution was added until pH of mixture was 14. After extraction by DCM (5 x 10 mL), the organic layer was combined and dried by $MgSO_4$, then concentrated. The pure product (0.43 g, 90% yield) was obtained by silica gel chromatography (very short column, eluting with 1:10 to 1: 5 methanol-DCM). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.22 (d, *J* = 7.3 Hz, 1H), 7.14-7.07 (m, 3H), 4.25 (d, *J* = 6.9 Hz, 1H), 2.92 (dd, *J* = 13.9, 6.6 Hz, 1H), 2.81 (ddd, *J* = 21.8, 14.7, 8.2 Hz, 2H), 2.53 (s, 4H), 2.15 (s, 2H), 1.58-1.53 (m, 4H), 1.40 (dt, *J* = 11.3, 5.8 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 145.16, 139.87, 127.20, 126.56, 124.42, 123.38, 77.71, 58.33, 51.96, 31.95,

25.96, 24.40; HRMS (ESI) calcd for $C_{14}H_{21}N_2$ (M + H^+) 217.1705, found 217.1708.

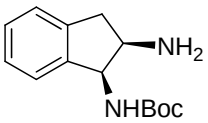


1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*S*,2*S*)-2-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-1-yl)thiourea (3c). To a solution of compound **i** (0.4 g, 1.85 mmol) in 10 mL DCM was added 1-isothiocyanato-3,5-bis(trifluoromethyl)benzene (0.53 g, 1.95 mmol) dropwise. The mixture was stirred at room temperature for 30min, reaction completed. The solvent was removed by rotary evaporation and pure product **3c** (0.87 g, 97% yield) was obtained by silica gel chromatography (eluting with 1:10 EtOAc- hexane then 1: 10 methanol-DCM). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 12.20 (s, 1H), 8.06 (s, 2H), 7.70 (s, 1H), 7.43-7.26 (m, 4H), 6.72 (s, 1H), 5.31 (m, 1H), 3.72 (q, *J* = 8.2 Hz, 1H), 3.21 (dd, *J* = 16.1, 8.5 Hz, 1H), 3.01 (dd, *J* = 16.1, 8.8 Hz, 1H), 2.77-2.67 (m, 4H), 1.56 (dd, *J* = 44.8, 22.4 Hz, 7H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 183.44, 141.53, 140.37, 137.76, 132.13, 131.85, 131.58, 131.31, 129.41, 127.85, 125.78, 125.58, 124.25, 123.61, 122.07, 118.82, 75.27, 62.04, 50.65, 26.33, 25.94, 23.84; HRMS (ESI) calcd for $C_{23}H_{24}F_6N_3S$ (M + H^+) 488.1595, found 488.1598.

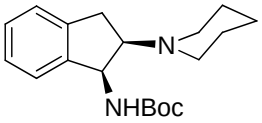


***tert*-butyl (1*S*,2*S*)-2-hydroxy-2,3-dihydro-1*H*-inden-1-ylcarbamate (k)[1].** It was prepared by using the same procedure for synthesis of compound **b**. Quantitative yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.18-7.11 (m, 4H), 5.00 (s, 1H), 4.82 (t, *J* = 5.9 Hz, 1H), 4.35-4.28 (m, 1H), 3.19 (dd, *J* = 15.8, 7.7 Hz, 1H), 2.81 (dd, *J* = 15.8, 8.1 Hz, 1H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 157.35, 140.12, 139.34, 128.40, 127.09, 125.09, 123.05, 81.82, 80.40, 63.98, 38.31, 28.31; HRMS (ESI) calcd for

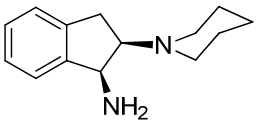
$C_{14}H_{19}NO_3$ (M + H^+) 249.1365, found 249.1366.



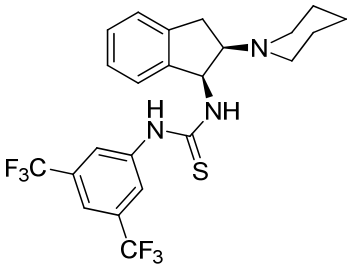
***tert*-butyl (1*S*,2*R*)-2-amino-2,3-dihydro-1*H*-inden-1-ylcarbamate (l)**[2]. It was prepared by using the same procedure for synthesis of compound **c** (67% yield, two steps). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.29 (m, 1H), 7.21 (d, *J* = 2.2 Hz, 3H), 5.23 (s, 1H), 5.03 (s, 1H), 3.82 (s, 1H), 3.12 (dd, *J* = 15.9, 6.1 Hz, 1H), 2.69 (dd, *J* = 15.9, 3.3 Hz, 1H), 1.73 (s, 2H), 1.49 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 156.05, 141.43, 140.60, 128.00, 126.94, 125.20, 124.71, 79.53, 58.56, 54.70, 39.70, 28.38; HRMS (ESI) calcd for $C_{14}H_{21}N_2O_2$ (M + H^+) 249.1603, found 249.1606.



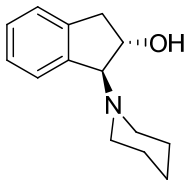
***tert*-butyl (1*S*,2*R*)-2-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-1-ylcarbamate (m)**[4]**.** It was prepared by using the same procedure for synthesis of compound **f** (75% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.58 (s, 1H), 7.21-7.14 (m, 3H), 5.81 (d, *J* = 5.3 Hz, 1H), 4.90 (s, 1H), 3.02 (p, *J* = 7.4 Hz, 1H), 2.92-2.89 (m, 2H), 2.42 (dd, *J* = 11.7, 6.2 Hz, 4H), 1.58 (dt, *J* = 12.0, 6.1 Hz, 6H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 156.03, 143.35, 140.42, 127.83, 126.62, 125.95, 124.17, 78.75, 67.58, 54.81, 52.40, 34.51, 28.34, 25.76, 24.26; HRMS (ESI) calcd for $C_{19}H_{29}N_2O_2$ (M + H^+) 317.2229, found 317.2225.



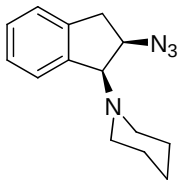
(1*S*,2*R*)-2-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-1-amine (n). It was prepared by using the same procedure for synthesis of compound **i** (90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.30 (d, *J* = 6.3 Hz, 1H), 7.14-7.08 (m, 3H), 4.15 (d, *J* = 5.4 Hz, 1H), 2.80 (qd, *J* = 14.8, 8.5 Hz, 2H), 2.72-2.68 (m, 1H), 2.48 (s, 2H), 2.38 (d, *J* = 3.8 Hz, 2H), 2.30 (s, 2H), 1.56 (dt, *J* = 11.0, 5.7 Hz, 4H), 1.42-1.40 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 144.85, 140.48, 127.51, 126.39, 124.44, 124.35, 69.87, 55.40, 52.58, 33.51, 25.53, 24.04; HRMS (ESI) calcd for $C_{14}H_{21}N_2$ ($M + H^+$) 217.1705, found 217.17010.



1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*S*,2*R*)-2-(piperidin-1-yl)-2,3-dihydro-1H-inden-1-yl)thiourea (5). It was prepared by using the same procedure for synthesis of catalyst **3c** (97% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.38 (s, 1H), 7.78 (d, *J* = 51.1 Hz, 3H), 7.60 (s, 1H), 7.15-7.05 (m, 3H), 5.53 (s, 1H), 3.06 (d, *J* = 6.6 Hz, 1H), 2.89 (dd, *J* = 15.4 Hz, 6.9, 1H), 2.79-2.74 (m, 1H), 2.31 (s, 4H), 1.25 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 180.13, 141.42, 140.74, 139.08, 133.36, 133.08, 132.81, 132.55, 128.71, 126.97, 126.83, 126.05, 124.47, 123.88, 123.71, 121.71, 119.54, 118.85, 68.02, 58.90, 52.26, 34.34, 25.63, 23.88; HRMS (ESI) calcd for $C_{23}H_{24}F_6N_3S$ ($M + H^+$) 488.1595, found 488.1590.

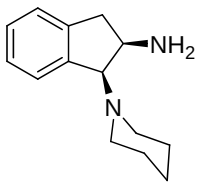


(1*S*,2*S*)-1-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-2-ol (o)[4]. Compound **j** (0.75 g, 5 mmol), 1,5-dibromopentane (1.38 g, 6.0 mmol), potassium carbonate (1.80 g, 13 mmol), potassium iodide (0.17 g, 1.0 mmol) and 10 mL iso-propanol were added into a sealed tube. The mixture was heated at 80 °C for 48hrs and then allowed to cool to room temperature. The mixture was filtered and washing with DCM, the filtrate was concentrated and the resulting residue was purified by silica gel chromatography (eluting with 1:5 EtOAc- hexane then 1:10 methanol-DCM) to obtain the product **o** (0.82 g, 75% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.36-7.33 (m, 1H), 7.12-7.16 (m, 3H), 4.66 (dd, *J* = 12.3, 5.3 Hz, 1H), 4.08 (d, *J* = 4.8 Hz, 1H), 3.25 (dd, *J* = 16.2, 7.1 Hz, 1H), 2.80 (dd, *J* = 16.2, 5.5 Hz, 1H), 2.62-2.61 (m, 4H), 1.58-1.54 (m, 4H), 1.46 (d, *J* = 5.3 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 140.62, 140.33, 127.66, 126.41, 125.82, 124.90, 78.49, 73.54, 50.79, 40.06, 26.51, 24.66; HRMS (ESI) calcd for $C_{14}H_{20}NO$ ($M + H^+$) 218.1545, found 218.1545.



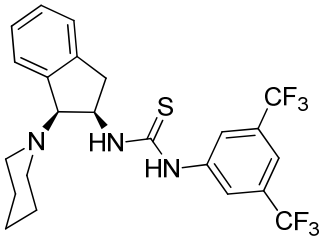
1-((1*S*,2*R*)-2-azido-2,3-dihydro-1*H*-inden-1-yl)piperidine (p)[5]**.** To a stirred solution of compound **o** (0.78 g, 3.6 mmol) and triethylamine (1.1 g, 10.8 mmol) in dry DCM (10 mL) at 0°C under nitrogen was added dropwise methanesulfonyl chloride (0.62 g, 5.4 mmol). The mixture was stirred for another 20 min at room temperature, and the solvent was evaporated under reduced pressure. The residue was extracted with DCM, washed successively with water, and brine, and dried over $MgSO_4$. The organic

layer was concentrated to afford crude mesylate intermediate. Then the crude mesylate intermediate was redissolve d in DMF (10 mL), followed by adding NaN_3 (1.87 g, 28.8 mmol). The mixture was heated under nitrogen at 70°C for 6 hrs. After the mixture was cooled, the solvent was evaporated under reduced pressure and the residue was extracted with EtOAc (25 mL x 3) and dried over $MgSO_4$. The organic layer was removed under reduced pressure, and the crude product was purified by silica gel chromatography (eluting with 1:10 EtOAc- hexane) to afford product **p** (0.48 g, 55% yield, two step). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.26-7.10 (m, 4H), 4.63 (d, *J* = 6.9 Hz, 1H), 3.17 (td, *J* = 8.0 Hz, 6.8, 1H), 3.03 (dd, *J* = 15.8, 7.9 Hz, 1H), 2.81 (dd, *J* = 15.8, 7.9 Hz, 1H), 2.48 (ddt, *J* = 38.5, 10.7, 5.2 Hz, 4H), 1.53 (dt, *J* = 11.3, 5.7 Hz, 4H), 1.39 (dd, *J* = 11.7, 6.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 140.43, 139.14, 128.56, 126.94, 124.71, 124.15, 72.82, 66.85, 51.91, 33.83, 25.94, 24.30; HRMS (ESI) calcd for $C_{14}H_{19}N_4$ ($M + H^+$) 243.1610, found 243.1612.



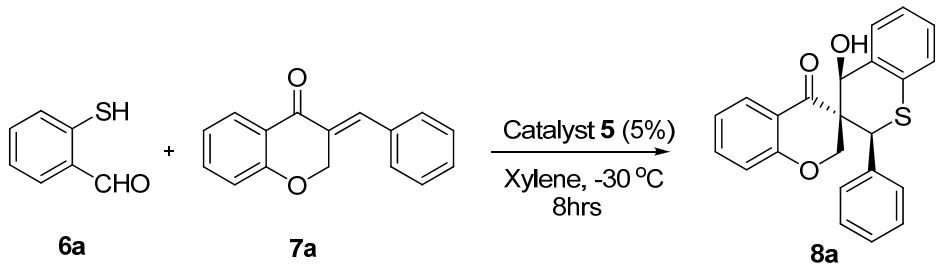
(1*S*,2*R*)-1-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-2-amine (q)[5]. To a solution of compound **p** (0.46 g, 1.9 mmol) in 10 mL THF was added triphenylphosphane (1.5 g, 5.7 mmol). The mixture was stirred at room temperature for 3h, then added 3 mL water, heated at 60°C for 4 hrs. The solvent was removed by reduced pressure, and the resulting residue was purified by a very short silica gel column (eluting with 1:10 to 1: 5 methanol-DCM) to afford compound **q** (0.39g, 95% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.27 (t, *J* = 5.8 Hz, 1H), 7.19-7.11 (m, 3H), 4.31 (d, *J* = 6.9 Hz, 1H), 2.99-2.83 (m, 3H), 2.60-2.58(m, 4H), 2.45 (s, 2H), 1.64-1.59 (m, 4H), 1.45 (dt, *J* = 11.7, 5.8 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 144.92, 139.73, 127.18, 126.54, 124.36, 123.34, 77.45, 58.16, 51.84, 31.67, 25.82, 24.29; HRMS (ESI)

calcd for $C_{14}H_{21}N_2$ $(M + H^+)$ 217.1705, found 217.1708.



1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*S*,2*R*)-1-(piperidin-1-yl)-2,3-dihydro-1*H*-inden-2-yl)thiourea (4). To a solution of compound **q** (0.39 g, 1.81 mmol) in 10 mL DCM was added 1-isothiocyanato-3,5-bis(trifluoromethyl)benzene (0.52 g, 1.90 mmol) dropwise. The mixture was stirred at room temperature for 30min, reaction completed. The solvent was removed by rotary evaporation and pure product **4** (0.90 g, 97% yield) was obtained by silica gel chromatography (eluting with 1:10 EtOAc- hexane then 1: 10 methanol-DCM). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 12.14 (s, 1H), 7.98 (s, 2H), 7.61 (s, 1H), 7.27 (ddd, *J* = 20.9, 12.6, 4.3 Hz, 4H), 6.68 (s, 1H), 5.22 (m, 1H), 3.63 (dd, *J* = 6.2, 8.3 Hz, 1H), 3.13 (dd, *J* = 16.1, 8.6 Hz, 1H), 2.92 (dd, *J* = 16.2, 8.9 Hz, 1H), 2.68-2.60 (m, 4H), 1.56-1.44 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 183.34, 141.50, 140.33, 137.67, 132.31, 131.87, 131.42, 130.98, 129.36, 127.78, 125.76, 125.53, 124.93, 123.58, 121.31, 118.75, 75.21, 62.01, 50.56, 26.29, 25.90, 23.78; HRMS (ESI) calcd for $C_{23}H_{24}F_6N_3S$ $(M + H^+)$ 488.1595, found 488.1587.

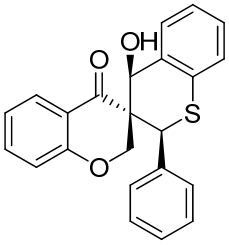
3. Representative Procedure



6a **7a** **8a**

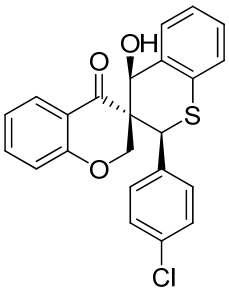
To a solution of 2-mercaptobenzaldehyde **6a** (13.8 mg, 0.1 mmol, 1 equiv.) in 0.95 mL xylene was added (E)-3-benzylidenechroman-4-one **7a** (20.4 mg, 0.1 mmol, 1 equiv.) at -30□, followed by adding of 50 μL of pre-cooled catalyst **5** solution (2.6 mg in 50 μL xylene, 0.005 mmol, 0.05 equiv.). The mixture was stirred at -30□ for 8 h. The crude product was purified by column chromatography on silica gel, eluted by hexane/EtOAc= 10:1 then 6: 1 to afford 36 mg (96% yield) of the desired product **8a** as white solid.

4. Analytical Data

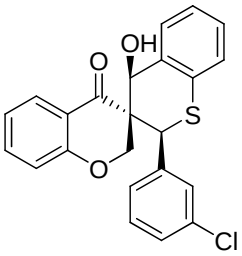


(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-phenylspiro[chroman-3,3'-thiochroman]-4-one (8a) (Table 2 , entry 1). ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.64-7.59 (m, 2H), 7.34-7.31 (m, 2H), 7.18-7.07 (m, 4H), 6.98-6.96 (m, 3H), 6.73 (t, *J* = 7.5 Hz, 1H), 6.50 (d, *J* = 8.3 Hz, 1H), 5.50 (d, *J* = 6.7 Hz, 1H), 5.00 (s, 1H), 4.77 (d, *J* = 12.6 Hz, 1H), 4.55 (d, *J* = 12.4 Hz, 1H), 2.78 (d, *J* = 6.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 195.32, 160.94, 136.09, 134.82, 133.23, 132.61, 129.50, 128.40, 127.82, 127.69, 127.17, 126.71, 125.19, 124.92, 121.58, 120.94, 117.55, 72.58, 66.86, 51.48, 51.37; HRMS (ESI) calcd for

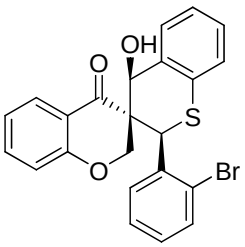
$C_{23}H_{18}O_3SNa$ (M + Na^+) 397.0874, found 397.0872; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 6.7min, t_{minor} = 9.6 min, *ee* = 97%, dr = 8.0:1; $[\alpha]^{25}_D$ (major) = +162.8 (*c* = 1.07 in $CHCl_3$).



(2'*S*,3*S*,4'*R*)-2'-(4-chlorophenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8b) (Table 2, entry 2). The title compound was prepared according the typical procedure, as described above in 98% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.70-7.67 (m, 2H), 7.35-7.15 (m, 6H), 7.02- 6.99 (m, 2H), 6.86-6.80 (m, 1H), 6.58 (d, *J* = 8.1 Hz, 1H), 5.56 (d, *J* = 6.4 Hz, 1H), 5.03 (s, 1H), 4.80 (d, *J* = 12.5 Hz, 1H), 4.61 (d, *J* = 12.5 Hz, 1H), 2.93 (d, *J* = 6.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.18, 160.85, 136.35, 134.19, 133.46, 133.06, 132.17, 130.85, 127.90, 127.78, 127.14, 126.68, 125.13, 125.07, 121.55, 121.22, 117.78, 72.85, 66.70, 51.40, 50.78; HRMS (EI) calcd for $C_{23}H_{17}ClO_3S$ 408.0587, found 408.0573; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 6.1 min, t_{minor} = 8.2 min, *ee* = 95%, dr = 8.1:1; $[\alpha]^{25}_D$ (major) = +138.6 (*c* = 1.25 in $CHCl_3$).

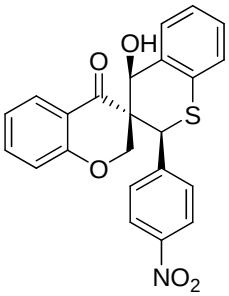


(2'*S*,3*S*,4'*R*)-2'-(3-chlorophenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8c) (Table 2, entry 3). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.72-7.67 (m, 2H), 7.48 (s, 1H), 7.29-7.19 (m, 5H), 7.02-6.92 (m, 2H), 6.84 (t, *J* = 7.5 Hz, 1H), 6.61 (d, *J* = 8.2 Hz, 1H), 5.58 (d, *J* = 6.4 Hz, 1H), 5.01 (s, 1H), 4.81 (d, *J* = 12.6 Hz, 1H), 4.62 (d, *J* = 12.6 Hz, 1H), 2.78 (dd, *J* = 6.4, 2.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 195.04, 160.85, 136.96, 136.38, 133.64, 133.04, 132.02, 129.54, 128.79, 128.53, 127.90, 127.82, 127.17, 126.68, 125.17, 125.10, 121.54, 121.10, 117.66, 72.66, 66.59, 51.28, 50.95; HRMS (EI) calcd for $C_{23}H_{17}O_4S$ 408.0587, found 408.0568; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.1 min, t_{minor} = 8.6 min, *ee* = 97%, dr = 7.6:1; $[\alpha]^{25}_D$ (major) = +140.4 (*c* = 1.15 in $CHCl_3$).

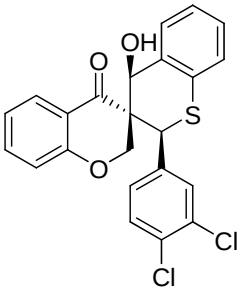


(2'*R*,3*S*,4'*R*)-2'-(2-bromophenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8d) (Table 2 , entry 4). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.00 (d, *J* = 7.9 Hz, 1H), 7.75 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.64 (d, *J* = 7.3 Hz, 1H), 7.23-7.09 (m, 7H), 6.90-6.79 (m, 2H), 6.57 (d, *J* = 8.3 Hz, 1H), 5.68 (d, *J* =

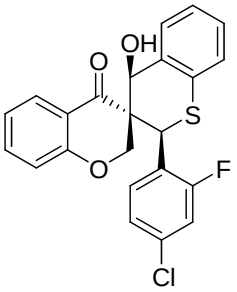
7.2 Hz, 2H), 4.81 (d, *J* = 12.6 Hz, 1H), 4.63 (d, *J* = 12.6 Hz, 1H), 3.09 (d, *J* = 5.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 193.88, 160.46, 136.17, 134.89, 133.36, 132.38, 132.23, 131.58, 129.74, 127.92, 127.38, 126.82, 125.43, 125.31, 124.94, 121.31, 120.93, 120.85, 117.42, 72.06, 66.95, 50.71, 48.35; HRMS (ESI) calcd for $C_{23}H_{17}BrO_4SNa$ (M + Na^+) 474.9979, found 474.9978; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 6.5 min, t_{minor} = 10.2 min, *ee* = 95%, dr = 19.0:1; $[\alpha]^{25}_D$ (major) = +405.3 (*c* = 1.50 in $CHCl_3$).



(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-(4-nitrophenyl)spiro[chroman-3,3'-thiochroman]-4-one (8e) (Table 2, entry 5). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.85-7.67 (m, 3H), 7.31-7.17 (m, 4H), 6.94-6.84 (m, 2H), 6.65 (dd, *J* = 9.9, 1.9 Hz, 1H), 6.53 (d, *J* = 8.5 Hz, 1H), 5.65 (d, *J* = 5.8 Hz, 1H), 5.35 (s, 1H), 4.77 (d, *J* = 12.6 Hz, 1H), 4.60 (d, *J* = 12.7 Hz, 1H), 2.98 (d, *J* = 5.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 194.37, 160.58, 136.40, 134.88, 132.90, 131.89, 131.76, 127.86, 127.15, 126.68, 125.25, 125.20, 123.86, 121.16, 117.54, 115.25, 114.90, 71.91, 66.60, 50.61, 42.29; HRMS (EI) calcd for $C_{23}H_{17}NO_5S$ 419.0827, found 419.0825; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 11.4 min, t_{minor} = 14.0 min, *ee* = 97%, dr = 7.7:1; $[\alpha]^{25}_D$ (major) = +92.2 (*c* = 0.67 in $CHCl_3$).

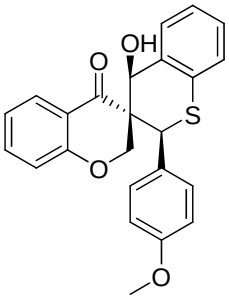


(2'*S*,3*S*,4'*R*)-2'-(3,4-dichlorophenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one(8f) (Table 2, entry 6). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.73-7.67 (m, 2H), 7.57 (d, *J* = 2.2 Hz, 1H), 7.32-7.29 (m, 1H), 7.26-7.16 (m, 4H), 7.08 (d, *J* = 8.5 Hz, 1H), 6.88-6.85 (m, 1H), 6.61 (d, *J* = 8.5 Hz, 1H), 5.56 (s, 1H), 4.97 (s, 1H), 4.78 (d, *J* = 12.6 Hz, 1H), 4.61 (d, *J* = 12.3 Hz, 1H), 2.81 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 194.94, 160.75, 136.54, 135.26, 132.92, 132.36, 131.83, 131.68, 131.37, 129.36, 128.91, 127.98, 127.17, 126.68, 125.23, 125.14, 121.51, 121.35, 117.68, 72.56, 66.51, 51.22, 50.40; HRMS (EI) calcd for $C_{23}H_{17}Cl_2O_3S$ 442.0197, found 442.0117; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 5.8 min, t_{minor} = 6.9 min, *ee* = 95%, dr = 8.2:1; $[\alpha]^{25}_D$ (major) = +125.5 (*c* = 1.40 in $CHCl_3$).



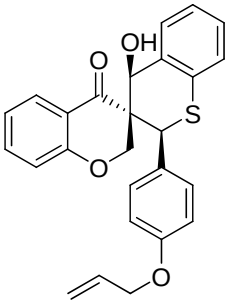
(2'*R*,3*S*,4'*R*)-2'-(4-chloro-2-fluorophenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8g) (Table 2, entry 7). The title compound was prepared according the typical procedure, as described above in 95% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.88 (t, *J* = 7.1 Hz, 2H), 7.78 (d, *J* = 8.0 Hz,

1H), 7.73-7.69 (m, 1H), 7.59 (d, *J* = 8.8 Hz, 0H), 7.49 (t, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.4 Hz, 1H), 7.24 (t, *J* = 5.3 Hz, 1H), 6.84 (t, *J* = 7.6 Hz, 1H), 6.54 (d, *J* = 8.3 Hz, 0H), 5.61 (d, *J* = 6.0 Hz, 1H), 5.12 (s, 1H), 4.80 (d, *J* = 12.7 Hz, 1H), 4.62 (d, *J* = 12.7 Hz, 1H), 2.78 (d, *J* = 6.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 191.78, 160.70, 142.76, 140.05, 136.73, 134.61, 134.25, 133.84, 132.91, 130.57, 128.05, 127.18, 126.73, 126.31, 125.38, 125.19, 122.63, 121.48, 117.71, 72.50, 66.57, 51.62, 50.97; HRMS (EI) calcd for $C_{23}H_{16}ClFO_3S$ 426.0493, found 426.0475; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.1 min, t_{minor} = 8.6 min, *ee* = 97%, dr = 20.0:1; $[\alpha]^{25}_D$ (major) = +150.6 (*c* = 1.23 in $CHCl_3$).

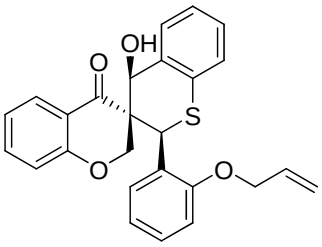


(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-(4-methoxyphenyl)spiro[chroman-3,3'-thiochroman]-4-one (8h) (Table 2, entry 8). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.69-7.65 (m, 2H), 7.33-7.30 (m, 2H), 7.24-7.14 (m, 4H), 6.81-6.79 (m, 1H), 6.61-6.56 (m, 3H), 5.54 (d, *J* = 6.6 Hz, 1H), 5.05 (s, 1H), 4.83 (d, *J* = 12.6 Hz, 1H), 4.62 (d, *J* = 12.3 Hz, 1H), 3.64 (s, 3H), 2.88 (d, *J* = 6.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.48, 160.93, 159.47, 136.02, 133.23, 132.80, 130.67, 127.77, 127.14, 126.68, 126.61, 125.07, 124.82, 121.65, 120.95, 117.74, 113.07, 73.05, 66.85, 55.18, 51.47, 50.74; HRMS (ESI) calcd for $C_{24}H_{20}O_4SNa$ ($M + Na^+$) 427.0980, found 427.0991; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 9.6 min, t_{minor} = 14.1 min, *ee* = 96%, dr = 8.0:1; $[\alpha]^{25}_D$

(major) = +116.4 (*c* = 1.17 in $CHCl_3$).

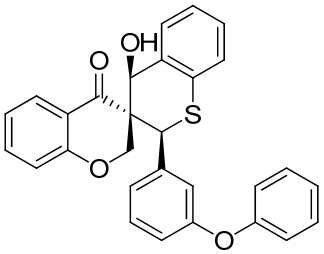


(2'*S*,3*S*,4'*R*)-2'-(4-(allyloxy)phenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8i) (Table 2, entry 9). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.71-7.65 (m, 2H), 7.32-7.17 (m, 6H), 6.83 (t, *J* = 7.5 Hz, 1H), 6.62-6.57 (m, 3H), 5.93 (ddd, *J* = 22.8, 10.6, 5.5 Hz, 1H), 5.55 (d, *J* = 6.6 Hz, 1H), 5.25 (m, 2H), 5.04 (s, 1H), 4.84 (d, *J* = 12.4 Hz, 1H), 4.62 (d, *J* = 12.4 Hz, 1H), 4.38 (d, *J* = 5.3 Hz, 2H), 2.70 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.45, 160.92, 158.45, 136.04, 133.16, 132.99, 132.78, 130.64, 127.79, 127.13, 126.75, 126.68, 125.08, 124.83, 121.63, 120.96, 117.77, 117.54, 113.91, 73.01, 68.64, 66.83, 51.44, 50.75; HRMS (ESI) calcd for $C_{26}H_{22}O_4SNa$ (M + Na^+) 453.1136, found 453.1119; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 8.6 min, t_{minor} = 12.7 min, *ee* = 96%, dr = 8.0:1; $[\alpha]^{25}_D$ (major) = +227.3 (*c* = 1.17 in $CHCl_3$).



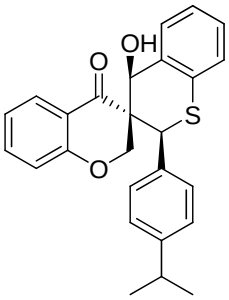
(2'*S*,3*S*,4'*R*)-2'-(2-(allyloxy)phenyl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8j) (Table

2, entry 10). The title compound was prepared according the typical procedure, as described above in 98% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.86 (dd, *J* = 7.9 Hz, 1.6, 1H), 7.68-7.64 (m, 2H), 7.24-7.15 (m, 4H), 6.96-6.93 (m, 1H), 6.79-6.75 (m, 2H), 6.46 (d, *J* = 8.2 Hz, 1H), 6.38 (d, *J* = 8.2 Hz, 1H), 6.02 (dddd, *J* = 17.3, 10.4, 5.7, 5.0 Hz, 1H), 5.65 (s, 2H), 5.36 (ddd, *J* = 17.0, 3.0, 1.6 Hz, 1H), 5.27 (m, 1H), 4.69 (d, *J* = 12.6 Hz, 1H), 4.55 (m, 1H), 4.26 (m, 1H), 4.17 (m, 1H), 3.22 (d, *J* = 5.4 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 194.48, 160.64, 155.65, 135.71, 133.47, 133.05, 132.96, 130.55, 129.21, 127.62, 127.02, 126.26, 125.51, 124.95, 124.15, 121.10, 120.36, 119.95, 117.52, 117.33, 110.28, 71.95, 68.87, 66.99, 50.82, 42.65; HRMS (ESI) calcd for $C_{26}H_{22}O_4SNa$ (M + Na^+) 453.1136, found 453.1141; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 6.4 min, t_{minor} = 7.3 min, *ee* = 99%, dr = 11.0:1; $[\alpha]^{25}_D$ (major) = +465.9 (*c* = 1.33 in $CHCl_3$).

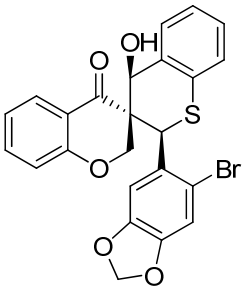


(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-(3-phenoxyphenyl)spiro[chroman-3,3'-thiochroman]-4-one (8k) (Table 2, entry 11). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.73-7.65 (m, 2H), 7.31-7.98 (m, 10H), 6.87-6.84 (m, 1H), 6.77-6.75 (m, 2H), 6.69-7.63 (m, 2H), 5.55 (s, 1H), 5.02 (s, 1H), 4.81 (d, *J* = 12.6 Hz, 1H), 4.61 (d, *J* = 12.3 Hz, 1H), 3.09 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.28, 161.03, 157.06, 156.24, 136.88, 136.19, 133.09, 132.30, 129.59, 128.93, 127.78, 127.14, 126.79, 125.11, 124.91, 124.63, 123.08, 121.56, 120.98, 120.35, 119.11, 118.42, 117.79, 72.75, 66.65, 51.54, 51.29; HRMS (ESI) calcd for $C_{29}H_{22}O_4SNa$ (M + Na^+) 489.1136, found 489.1143; HPLC (Chiralpak IC, *i*-propanol/hexane =

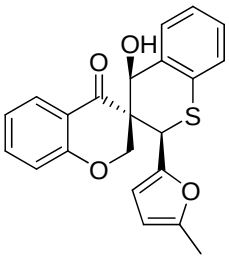
10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 6.5 min, t_{minor} = 7.2 min, *ee* = 98%, dr = 11.0:1; $[\alpha]^{25}_{D}$ (major) = +164.9 (*c* = 1.50 in $CHCl_3$).



(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-(4-isopropylphenyl)spiro[chroman-3,3'-thiochroman]-4-one (8l) (Table 2, entry 12). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.68-7.65 (m, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 7.21-7.14 (m, 4H), 6.86 (d, *J* = 8.2 Hz, 2H), 6.78-6.75 (m, 1H), 6.52 (d, *J* = 8.2 Hz, 1H), 5.58 (d, *J* = 6.3 Hz, 1H), 5.03 (s, 1H), 4.85 (d, *J* = 12.3 Hz, 1H), 4.61 (d, *J* = 12.3 Hz, 1H), 2.93 (d, *J* = 6.6 Hz, 1H), 2.67 (dt, *J* = 13.9, 6.9 Hz, 1H), 1.04 (dd, *J* = 6.9, 0.9 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.44, 160.84, 149.17, 135.88, 133.17, 132.71, 131.87, 129.35, 127.74, 127.19, 126.67, 125.56, 125.15, 124.81, 121.53, 120.66, 117.64, 72.67, 66.70, 51.32, 51.14, 33.67, 23.68, 23.66; HRMS (ESI) calcd for $C_{26}H_{24}O_3SNa$ ($M + Na^+$) 439.1344, found 439.1321; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} =7.0 min, t_{minor} = 10.9 min, *ee* = 97%, dr = 8.0:1; $[\alpha]^{25}_{D}$ (major) = +150.5 (*c* = 1.00 in $CHCl_3$).

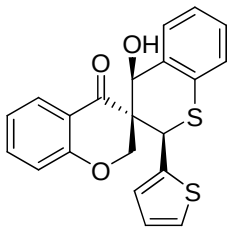


(2'*R*,3*S*,4'*R*)-2'-(5-bromobenzo[d][1,3]dioxol-4-yl)-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8m) (Table 2, entry 13). The title compound was prepared according the typical procedure, as described above in 98% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.76-7.74 (m, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.47 (s, 1H), 7.31-7.17 (m, 4H), 6.86-6.83 (m, 1H), 6.68 (d, *J* = 8.2 Hz, 1H), 6.63 (s, 1H), 5.84 (dd, *J* = 38.0, 1.4 Hz, 2H), 5.63 (d, *J* = 24.0 Hz, 2H), 4.79 (d, *J* = 12.6 Hz, 1H), 4.64 (d, *J* = 12.6 Hz, 1H), 3.12 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 193.85, 160.42, 148.04, 146.88, 136.10, 133.29, 132.31, 127.92, 127.59, 127.43, 126.82, 125.39, 125.27, 121.03, 120.93, 117.34, 115.76, 111.72, 110.92, 101.80, 71.95, 66.75, 50.66, 48.47; HRMS (ESI) calcd for $C_{24}H_{17}BrO_5SNa$ ($M + Na^+$) 518.9878, found 518.9897; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 8.6 min, t_{minor} = 13.0 min, *ee* = 98%, dr = 17.0:1; $[\alpha]^{25}_D$ (major) = +147.3 (*c* = 1.23 in $CHCl_3$).

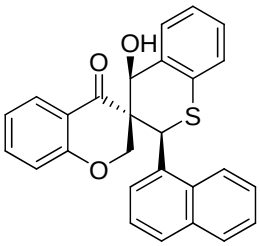


(2'*R*,3*S*,4'*R*)-4'-hydroxy-2'-(5-methylfuran-2-yl)spiro[chroman-3,3'-thiochroman]-4-one (8n) (Table 2, entry 14). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.82 (dd, *J* = 7.9 Hz, 1.6, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.37 (ddd, *J* = 8.8, 7.3, 1.9 Hz, 1H), 7.20-7.15 (m, 3H), 6.94-6.90 (m, 1H), 6.78 (m,

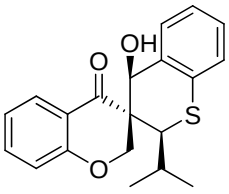
1H), 6.20 (d, *J* = 3.2 Hz, 1H), 5.67 (m, 1H), 5.41 (d, *J* = 6.3 Hz, 1H), 5.09 (s, 1H), 4.70 (d, *J* = 12.6 Hz, 1H), 4.59 (d, *J* = 12.3 Hz, 1H), 3.01 (d, *J* = 7.3 Hz, 1H), 1.98 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$): δ = 194.92, 161.35, 152.20, 146.68, 136.00, 133.47, 131.97, 127.82, 127.40, 127.03, 125.24, 125.06, 121.36, 121.14, 117.74, 111.00, 106.21, 72.50, 67.37, 51.10, 43.26, 13.19; HRMS (ESI) calcd for $C_{22}H_{18}O_4SNa$ (M + Na^+) 401.0823, found 401.0825; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.8 min, t_{minor} = 9.1 min, *ee* = 96%, dr = 8.4:1; $[\alpha]^{25}_D$ (major) = +80.1 (*c* = 1.33 in $CHCl_3$).



(2'*R*,3*S*,4'*R*)-4'-hydroxy-2'-(thiophen-2-yl)spiro[chroman-3,3'-thiochroman]-4-one (8o) (Table 2, entry 15). The title compound was prepared according the typical procedure, as described above in 98% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.78-7.76 (m, 2H), 7.34-7.31 (m, 1H), 7.25-7.16 (m, 3H), 7.04 (dd, *J* = 8.0 Hz, 4.3, 2H), 6.88 (t, *J* = 7.1 Hz, 1H), 6.72 (dd, *J* = 5.0, 3.5 Hz, 2H), 5.46-5.43 (m, 2H), 4.80 (d, *J* = 12.3 Hz, 1H), 4.63 (d, *J* = 12.3 Hz, 1H), 2.99 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.23, 161.28, 137.75, 136.18, 133.41, 132.33, 128.52, 127.91, 127.17, 126.96, 126.31, 125.57, 125.15, 124.87, 122.00, 121.34, 117.88, 73.68, 67.16, 51.67, 45.56; HRMS (ESI) calcd for $C_{21}H_{16}O_4S_2Na$ (M + Na^+) 403.0439, found 403.0420; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.6 min, t_{minor} = 9.3 min, *ee* = 97%, dr = 8.3:1; $[\alpha]^{25}_D$ (major) = +129.2 (*c* = 1.40 in $CHCl_3$).

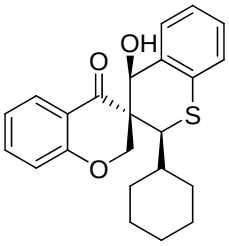


(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-(naphthalen-1-yl)spiro[chroman-3,3'-thiochroman]-4-one (8p) (Table 2, entry 16). The title compound was prepared according the typical procedure, as described above in 95% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.18 (d, *J* = 6.7 Hz, 1H), 7.91 (d, *J* = 8.5 Hz, 1H), 7.62 (dd, *J* = 22.6 Hz, 7.9, 2H), 7.52 (d, *J* = 8.2 Hz, 1H), 7.45-7.41 (m, 1H), 7.34-7.16 (m, 7H), 7.07-7.04 (m, 1H), 6.48-6.45 (m, 2H), 6.07 (s, 1H), 5.77 (d, *J* = 6.4 Hz, 1H), 4.87 (d, *J* = 12.5 Hz, 1H), 4.68 (s, 1H), 3.40 (d, *J* = 6.4 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 194.93, 160.49, 135.95, 133.53, 133.10, 133.07, 131.29, 131.01, 129.08, 128.49, 127.93, 127.90, 127.51, 126.29, 126.11, 125.63, 125.31, 125.23, 124.53, 122.70, 120.85, 120.37, 117.25, 72.62, 67.10, 51.15, 44.50; HRMS (ESI) calcd for $C_{27}H_{20}O_3SNa$ (M + Na^+) 447.1031, found 447.1021; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.7 min, t_{minor} = 10.6 min, *ee* = 95%, dr = 4.5:1; $[\alpha]^{25}_D$ (major) = +545.0 (*c* = 1.00 in $CHCl_3$).

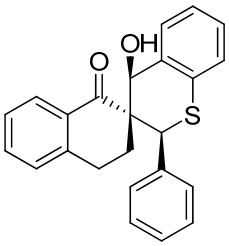


(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-isopropylspiro[chroman-3,3'-thiochroman]-4-one (8q) (Table 2, entry 17). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.93-7.90 (m, 1H), 7.61-7.45 (m, 2H), 7.19-6.92 (m, 5H), 5.24

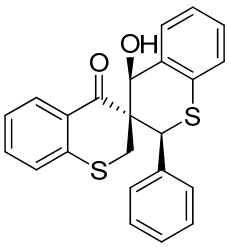
(d, *J* = 7.3 Hz, 1H), 4.50 (q, *J* = 12.4 Hz, 2H), 4.03 (d, *J* = 4.7 Hz, 1H), 2.84 (d, *J* = 7.3 Hz, 1H), 2.02-1.91 (m, 1H), 1.07 (d, *J* = 6.6 Hz, 3H), 0.97 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 196.70, 161.62, 136.41, 133.86, 132.66, 127.64, 127.09, 126.34, 125.49, 124.56, 122.70, 121.81, 118.25, 75.26, 66.93, 54.81, 51.77, 30.03, 24.24, 19.27; HRMS (ESI) calcd for $C_{20}H_{20}O_3SNa$ ($M + Na^+$) 363.1031, found 363.1038; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.3 min, t_{minor} = 8.6 min, *ee* = 95%, dr = 57.0:1; $[\alpha]^{25}_D$ (major) = +48.4 (*c* = 1.03 in $CHCl_3$).



(2'*S*,3*S*,4'*R*)-2'-cyclohexyl-4'-hydroxyspiro[chroman-3,3'-thiochroman]-4-one (8r) (Table 2, entry 18). The title compound was prepared according the typical procedure, as described above in 98% yield. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.92 (dd, *J* = 7.9 Hz, 1.6, 1H), 7.60-7.46 (m, 2H), 7.17-6.93 (m, 5H), 5.20 (d, *J* = 7.2 Hz, 1H), 4.50 (q, *J* = 12.3 Hz, 2H), 4.00 (d, *J* = 4.9 Hz, 1H), 2.77 (d, *J* = 7.6 Hz, 1H), 2.04 (d, *J* = 12.7 Hz, 1H), 1.70-1.43 (m, 4H), 1.26-0.83 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 196.45, 161.53, 136.29, 134.04, 132.84, 127.64, 127.10, 126.27, 125.42, 124.58, 122.81, 121.83, 118.13, 110.18, 75.48, 67.20, 53.86, 51.31, 40.16, 34.34, 30.27, 26.63, 26.32, 25.85; HRMS (ESI) calcd for $C_{23}H_{24}O_3SNa$ ($M + Na^+$) 403.1344, found 403.1345; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 5.7 min, t_{minor} = 6.3 min, *ee* = 96%, dr = 24.0:1; $[\alpha]^{25}_D$ (major) = +103.7 (*c* = 1.05 in $CHCl_3$).

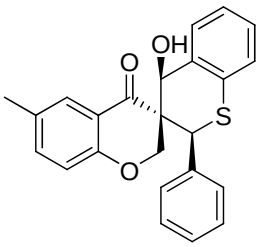


(2*S*,2'*S*,4'*R*)-4'-hydroxy-2'-phenyl-3,4-dihydro-1*H*-spiro[naphthalene-2,3'-thiochroman]-1-one (8s) (Table 2, entry 19). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.79-7.77 (m, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.44-7.42 (m, 2H), 7.29-7.10 (m, 8H), 6.94 (d, *J* = 7.6 Hz, 1H), 5.47 (s, 1H), 5.38 (d, *J* = 6.3 Hz, 1H), 2.98 (d, *J* = 6.6 Hz, 1H), 2.93-2.87 (m, 1H), 2.39-2.33 (m, 1H), 2.18-2.03 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 202.37, 144.23, 136.57, 135.20, 133.71, 133.68, 133.50, 129.88, 128.17, 128.07, 128.05, 127.33, 127.12, 126.28, 126.14, 125.12, 124.52, 77.38, 52.58, 52.57, 25.94, 22.02; HRMS (ESI) calcd for $C_{24}H_{20}O_2SNa$ (M + Na^+) 395.1082, found 395.1088; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.1 min, t_{minor} = 8.2 min, *ee* = 95%, dr = 10.0:1; $[\alpha]^{25}_D$ (major) = + 49.0 (*c* = 1.20 in $CHCl_3$).

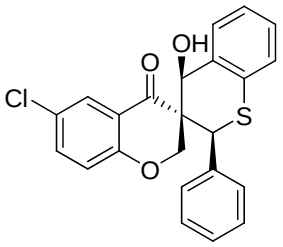


(2'*S*,3*S*,4'*R*)-4'-hydroxy-2'-phenyl-3,3'-spirobi[thiochroman]-4-one (8t) (Table 2, entry 20). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.97-7.96 (m, 1H), 7.69-7.68 (m, 1H), 7.50-7.48 (m, 2H), 7.29-7.09 (m, 9H), 5.52 (s, 1H), 5.30 (d, *J* = 11.0 Hz, 1H), 3.54 (dd, *J* = 14.2, 1.3 Hz, 1H), 3.48 (d, *J* = 11.0 Hz, 1H),

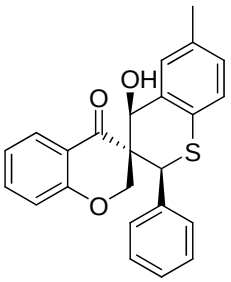
3.27 (d, *J* = 14.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 196.36, 139.71, 135.69, 135.22, 133.21, 133.03, 132.88, 130.24, 130.12, 128.41, 128.18, 127.91, 127.66, 126.39, 125.79, 125.30, 125.03, 78.40, 51.67, 51.36, 28.02; HRMS (EI) calcd for $C_{23}H_{18}O_2S_2$ 390.0748, found 390.0730; HPLC (Chiralpak IC, *i*-propanol/hexane = 5/95, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 14.3 min, t_{minor} = 15.6 min, *ee* = 96%, dr = 1.2:1; $[\alpha]^{25}_D$ (major) = -55.4 (*c* = 0.93 in $CHCl_3$).



(2'*S*,3*S*,4'*R*)-4'-hydroxy-6-methyl-2'-phenylspiro[chroman-3,3'-thiochroman]-4-one (8u) (Table 2, entry 21). The title compound was prepared according the typical procedure, as described above in 98% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.69-7.67 (m, 1H), 7.44-7.39 (m, 3H), 7.21 (t, *J* = 3.8 Hz, 2H), 7.18-7.14 (m, 1H), 7.06-7.01 (m, 4H), 6.47 (d, *J* = 8.5 Hz, 1H), 5.54 (d, *J* = 6.9 Hz, 1H), 5.09 (s, 1H), 4.79 (d, *J* = 12.6 Hz, 1H), 4.60 (d, *J* = 12.3 Hz, 1H), 3.15 (d, *J* = 6.6 Hz, 1H), 2.13 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.51, 159.12, 137.28, 134.96, 133.44, 132.67, 130.33, 129.54, 128.32, 127.74, 127.68, 127.14, 126.19, 125.14, 124.86, 121.23, 117.43, 73.19, 66.86, 51.55, 51.29, 20.24; HRMS (ESI) calcd for $C_{24}H_{20}O_3SNa$ (M + Na^+) 411.1031, found 411.1018; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 7.3 min, t_{minor} = 13.2 min, *ee* = 97%, dr = 16.0:1; $[\alpha]^{25}_D$ (major) = +109.5 (*c* = 1.57 in $CHCl_3$).

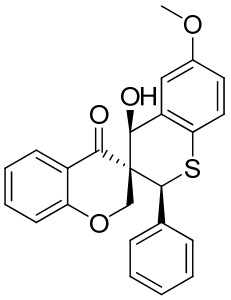


(2'*S*,3*S*,4'*R*)-6-chloro-4'-hydroxy-2'-phenylspiro[chroman-3,3'-thiochroman]-4-one (8v) (Table 2, entry 22). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.65 (dd, *J*=9.6, 5.2, 2H), 7.38 (dd, *J*=6.6, 2.8, 2H), 7.23-7.15 (m, 4H), 7.08-7.06 (m, 3H), 6.53 (d, *J*=9.1, 1H), 5.57 (d, *J*=6.0, 1H), 5.05 (s, 1H), 4.86 (d, *J*=12.6, 1H), 4.61 (d, *J*=12.6, 1H), 2.81 (d, *J*=6.3, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 194.71, 159.42, 135.89, 134.42, 132.93, 132.41, 129.42, 128.58, 127.86, 127.75, 127.17, 126.37, 125.75, 125.13, 124.94, 122.02, 119.41, 72.74, 66.88, 51.51, 51.46; HRMS (EI) calcd for $C_{23}H_{17}ClO_3S$ 431.0485, found 431.0466; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 5.6 min, t_{minor} = 7.1 min, *ee* = 98%, dr = 11.0:1; $[\alpha]^{25}_{D}$ (major) = +205.2 (*c* = 1.33 in $CHCl_3$).

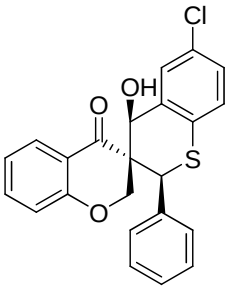


(2'*S*,3*S*,4'*R*)-4'-hydroxy-6'-methyl-2'-phenylspiro[chroman-3,3'-thiochroman]-4-one (8w) (Table 2, entry 23). The title compound was prepared according the typical procedure, as described above in 96% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.71-7.69 (m, 1H), 7.48 (s, 1H), 7.40-7.38 (m, 2H), 7.26-7.22 (m, 2H), 7.11 (d, *J* = 8.2 Hz, 1H), 7.05 (d, *J* = 6.6 Hz, 4H), 6.81 (t, *J* = 7.6 Hz, 1H), 6.58 (d, *J*

= 8.5 Hz, 1H), 5.54 (d, *J* = 6.6 Hz, 1H), 5.05 (s, 1H), 4.83 (d, *J* = 12.3 Hz, 1H), 4.62 (d, *J* = 12.6 Hz, 1H), 2.72 (d, *J* = 6.9 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.32, 160.96, 136.04, 135.04, 134.73, 133.07, 129.48, 128.96, 128.75, 128.34, 127.75, 127.69, 126.75, 125.21, 121.59, 120.93, 117.71, 73.02, 67.04, 51.72, 51.23, 21.12; HRMS (ESI) calcd for $C_{24}H_{20}O_3SNa$ (M + Na^+) 411.1031, found 411.1011; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 6.7 min, t_{minor} = 11.4 min, *ee* = 96%, dr = 9.0:1; $[\alpha]^{25}_D$ (major) = +85.5 (*c* = 1.33 in $CHCl_3$).

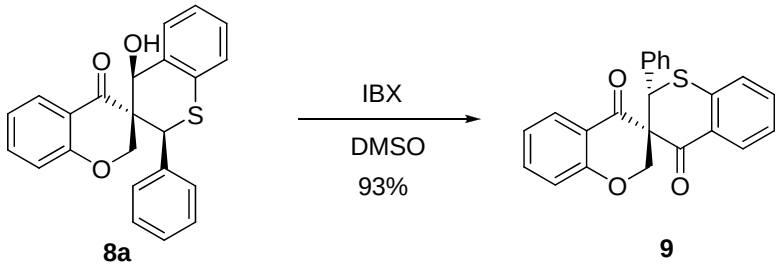


(2'*S*,3*S*,4'*R*)-4'-hydroxy-6'-methoxy-2'-phenylspiro[chroman-3,3'-thiochroman]-4-one (8x) (Table 2, entry 24). The title compound was prepared according the typical procedure, as described above in 97% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.70-7.68 (m, 1H), 7.39-7.37 (m, 2H), 7.26-7.22 (m, 2H), 7.13 (d, *J* = 8.5 Hz, 1H), 7.06-7.03 (m, 3H), 6.82 (m, 2H), 6.58 (d, *J* = 8.2 Hz, 1H), 5.53 (d, *J* = 6.6 Hz, 1H), 5.05 (s, 1H), 4.80 (d, *J* = 12.6 Hz, 1H), 4.61 (d, *J* = 12.3 Hz, 1H), 3.81 (s, 3H), 2.82 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.33, 160.97, 157.65, 136.08, 135.15, 134.78, 129.42, 128.32, 127.69, 126.75, 126.45, 123.17, 121.53, 120.94, 117.70, 114.59, 112.53, 73.11, 67.02, 55.44, 51.89, 51.28; HRMS (ESI) calcd for $C_{24}H_{20}O_4SNa$ (M + Na^+) 427.0980, found 427.0989; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 9.9 min, t_{minor} = 18.2 min, *ee* = 95%, dr = 15.0:1; $[\alpha]^{25}_D$ (major) = +59.5 (*c* = 1.10 in $CHCl_3$).



(2'*S*,3*S*,4'*R*)-6'-chloro-4'-hydroxy-2'-phenylspiro[chroman-3,3'-thiochroman]-4-one (8y) (Table 2, entry 25). The title compound was prepared according the typical procedure, as described above in 93% yield. ^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.70-7.68 (m, 2H), 7.39-7.37 (m, 2H), 7.21 (dddd, *J*=29.0, 27.4, 17.0, 5.0, 3H), 7.06-7.04 (m, 3H), 6.83-6.80 (m, 1H), 6.56 (d, *J*=8.5, 1H), 5.54 (d, *J*=6.6, 1H), 5.05 (s, 1H), 4.81 (d, *J*=12.3, 1H), 4.60 (d, *J*=12.6, 1H), 2.78 (d, *J*=6.6, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.03, 160.90, 136.25, 134.88, 134.41, 131.12, 130.83, 129.46, 128.56, 128.01, 127.72, 127.35, 126.68, 126.34, 121.48, 121.02, 117.74, 72.61, 66.56, 51.54, 51.14; HRMS (EI) calcd for $C_{23}H_{17}ClO_3S$ 408.0587, found 408.0569; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 5.9 min, t_{minor} = 22.2 min, *ee* = 92%, dr = 8.0:1; $[\alpha]^{25}_{D}$ (major) = +37.9 (*c* = 0.93 in $CHCl_3$).

5. Oxidation of Compound 8a

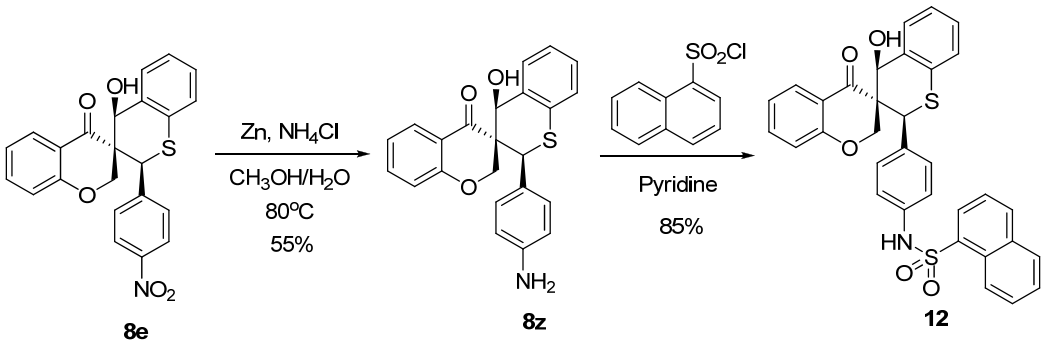


8a **9**

To a solution of **8a** (37.5 mg, 0.1 mmol) in 2 mL DMSO was added 2-Iodoxybenzoic acid (IBX, 84 mg, 0.3 mmol). The mixture was stirred at room temperature for 4h, reaction completed. The mixture was diluted with 10 mL water, extracted with EtOAc (3x 20 mL), the organic layers were combined, dried

and concentrated. The crude product was purified by silica gel chromatography (eluting with 1:6 EtOAc-hexane) to give product **9a** (34.6 mg, 93% yield).[6] ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.11 (dd, *J* = 7.9 Hz, 1.3, 1H), 7.90 (dd, *J* = 7.9 Hz, 1.9, 1H), 7.52-7.44 (m, 2H), 7.37 (dd, *J* = 7.1, 2.4 Hz, 2H), 7.27-7.24 (m, 5H), 7.04 (m, 2H), 5.00-4.97 (m, 2H), 4.44 (d, *J* = 12.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 192.54, 189.85, 160.74, 140.52, 136.33, 136.18, 133.89, 130.87, 129.24, 128.79, 128.74, 128.59, 128.01, 126.88, 125.30, 122.06, 120.43, 117.62, 69.20, 57.88, 47.57; HRMS (ESI) calcd for $C_{23}H_{16}O_3SNa$ (M + Na^+) 395.0718, found 395.0689; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 10.8 min, t_{minor} = 17.7 min, *ee* = 97%, dr = 8.0:1; $[\alpha]^{25}_D$ (major) = –300.2 (*c* = 1.07 in $CHCl_3$).

6. Preparation of Compound 12 for X-ray Crystallographic analysis



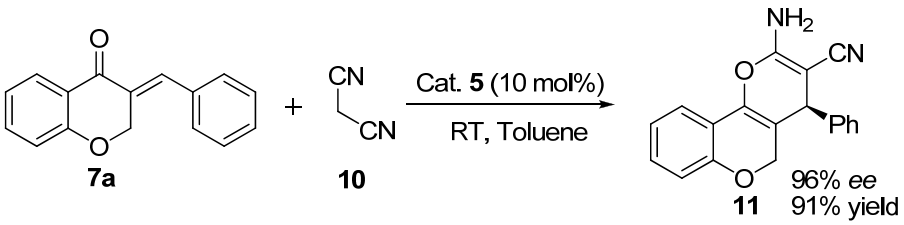
8e **8z** **12**

To a stirred mixture of compound **8e** (210 mg, 0.5 mmol) and water (6 mL), NH_4Cl (214 mg, 4 mmol) and zinc metal powder (490 mg, 7.5 mmol) were added at room temperature. After the reaction mixture was stirred for 1 hour at 80 °C, the mixture was extracted with EtOAc (3 x 20 mL). The combined organic layer was dried by $MgSO_4$ and concentrated, the resulting residue was purified by silica gel chromatography (eluting with EtOAc/hexane = 1/2) to give intermediate product **8z** (107 mg, 55% yield).[7]

The intermediate product **8z** (98 mg, 0.25 mmol) was dissolved in 2 mL dried pyridine. Then

naphthalene-1-sulfonyl chloride (170 mg, 0.75 mmol) was added into the mixture. The reaction mixture was stirred at room temperature overnight. After that, 10 mL 1.0 M HCl solution was added and the resulting mixture was extracted with EtOAc (3 x 20 mL). The combined organic layer was dried by $MgSO_4$ and concentrated, the resulting residue was purified by silica gel chromatography (eluting with EtOAc/Hexane = 1/3) to give product **12** (123 mg, 85% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.57 (d, *J* = 8.5 Hz, 1H), 8.08 (dd, *J* = 7.6, 0.9 Hz, 1H), 8.03 (d, *J* = 8.2 Hz, 1H), 7.93 (d, *J* = 7.6 Hz, 1H), 7.65-7.58 (m, 4H), 7.42 (t, *J* = 7.7 Hz, 1H), 7.18-7.13 (m, 5H), 7.05-7.01 (m, 1H), 6.72-6.68 (m, 2H), 6.62 (d, *J* = 8.5 Hz, 2H), 6.40 (d, *J* = 8.5 Hz, 1H), 5.47 (d, *J* = 6.6 Hz, 1H), 4.93 (s, 1H), 4.67 (d, *J* = 12.6 Hz, 1H), 4.52 (d, *J* = 12.6 Hz, 1H), 2.67 (d, *J* = 6.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 195.12, 160.74, 136.29, 136.10, 134.68, 134.16, 133.88, 133.07, 132.25, 131.68, 130.43, 130.34, 129.23, 128.56, 128.04, 127.85, 127.08, 126.97, 126.55, 125.10, 125.01, 124.07, 124.01, 121.45, 120.98, 119.93, 117.58, 72.86, 66.69, 51.34, 50.59; HRMS (ESI) calcd for $C_{33}H_{24}NO_5S_2$ (M $-H^+$) 578.1096, found 578.1110.

7. Preparation of the compound 11



To a solution of malononitrile **10** (10mg, 0.15mmol, 1.5equiv.) in 0.5mL toluene was added (E)-3-benzylidenechroman-4-one **7a** (24mg, 0.1mmol, 1equiv.) at room temperature, followed by adding catalyst IV (4.88mg, 0.01mmol, 0.1equiv.). The mixture was stirred at room temperature. Upon completion, the crude product was purified by column chromatography on silica gel, eluted by

hexane/EtOAc=8:1 then 4:1 to afford the desired product **11** (27.4 mg, 91% yield) as primrose yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.39 – 7.32 (m, 3H), 7.27 (q, *J* = 6.0 Hz, 3H), 7.23 – 7.15 (m, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.79 (d, *J* = 8.1 Hz, 1H), 4.64 (s, 2H), 4.63 (d, *J* = 13.2 Hz, 1H), 4.43 (d, *J* = 13.8 Hz, 1H), 4.03 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 158.77, 154.12, 140.87, 138.01, 130.30, 129.02, 127.92, 127.88, 121.26, 121.04, 119.26, 116.66, 115.94, 105.05, 66.48, 61.16, 39.71; HRMS (EI) calcd for $C_{19}H_{14}O_2N_2$ 302.1055, found 302.1048; HPLC (Chiralpak IC, *i*-propanol/hexane = 10/90, flow rate 1.0 mL/min, λ = 254 nm): t_{major} = 15.7 min, t_{minor} = 27.0 min, *ee* = 96%, $[\alpha]^{30}_{D}$ (major) = -64.1 (*c* = 0.98 in $CHCl_3$).

Reference:

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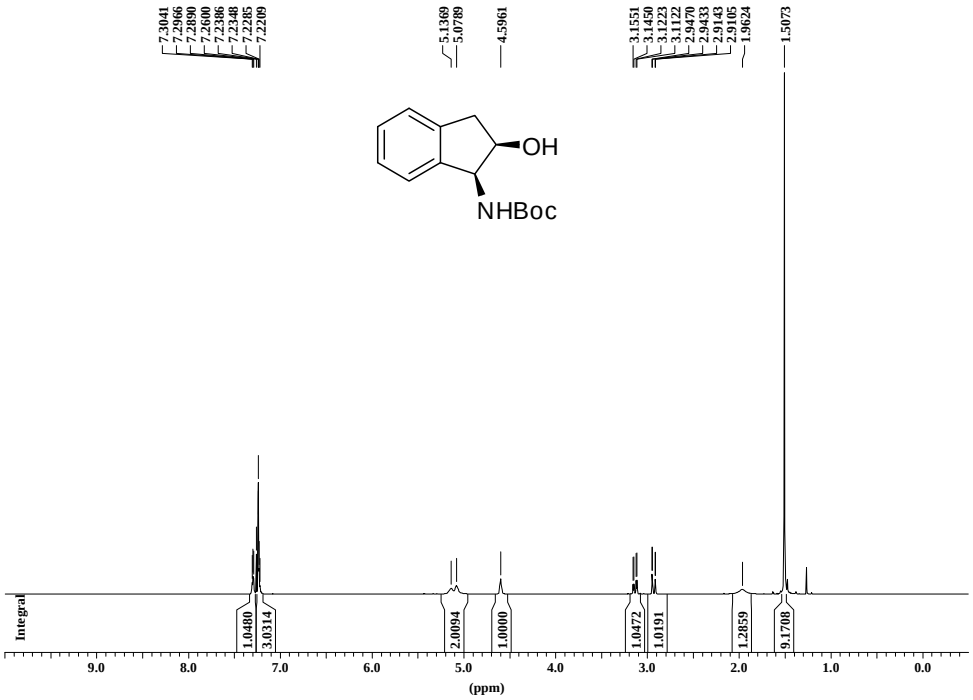
Compound **b**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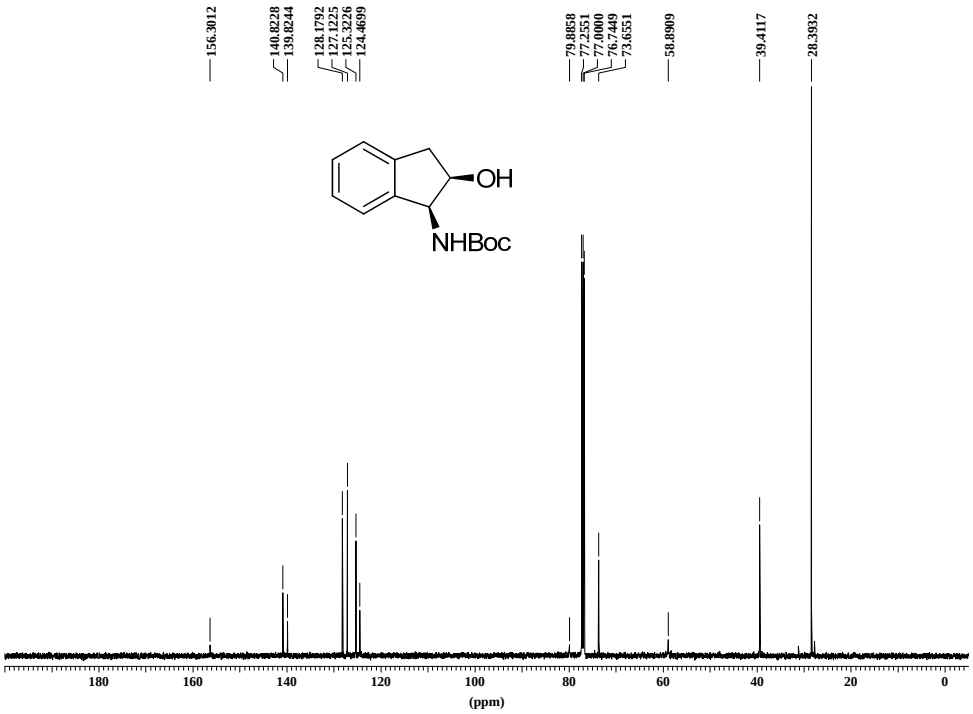
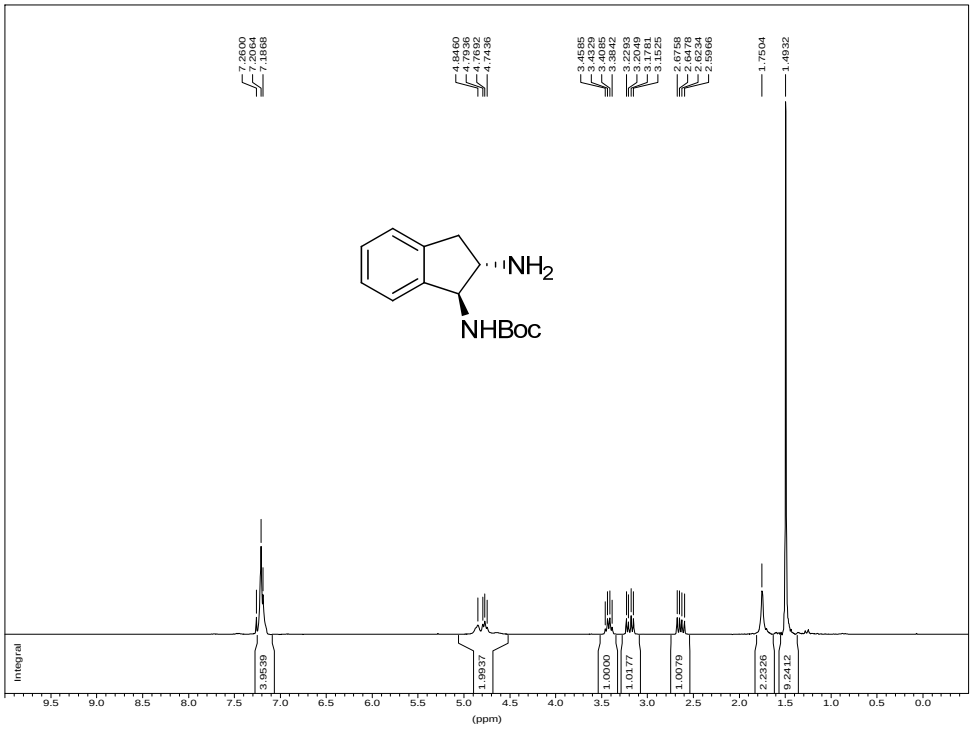
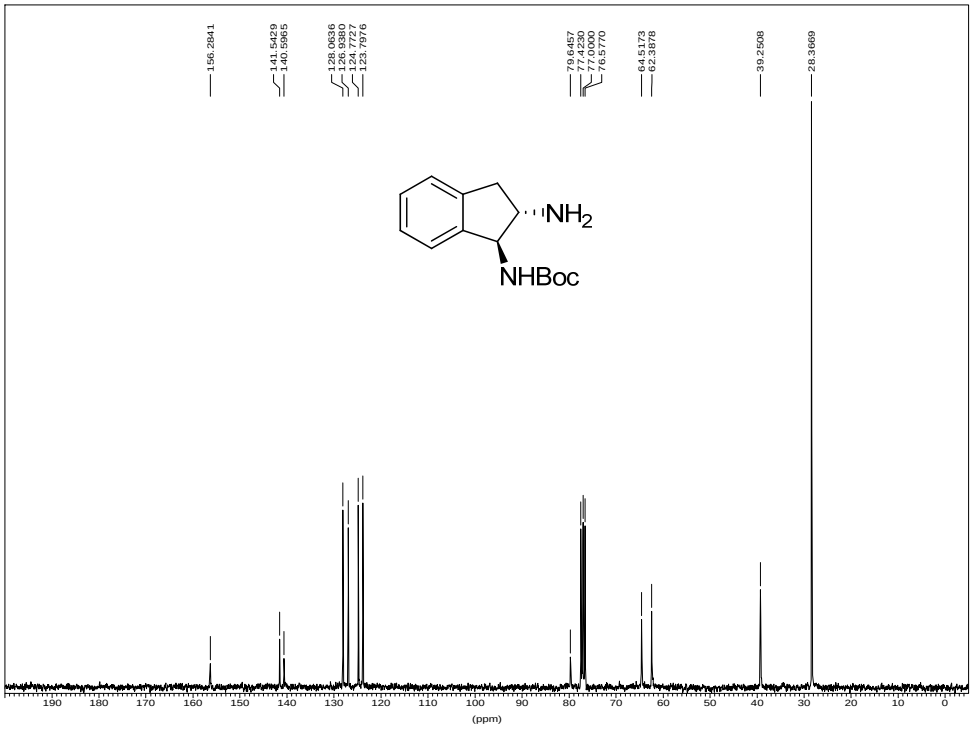
7.2966
7.2890
7.2600
7.2386
7.2348
7.2285
7.2209
5.1369
5.0789
4.5961
3.1551
3.1450
3.1223
3.1172
2.9470
2.9433
2.9143
2.9105
1.9624
1.5073
OH
NHBoc
Integral
1.0480
3.0314
2.0094
1.0000
1.0472
1.0191
1.2859
9.1708
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

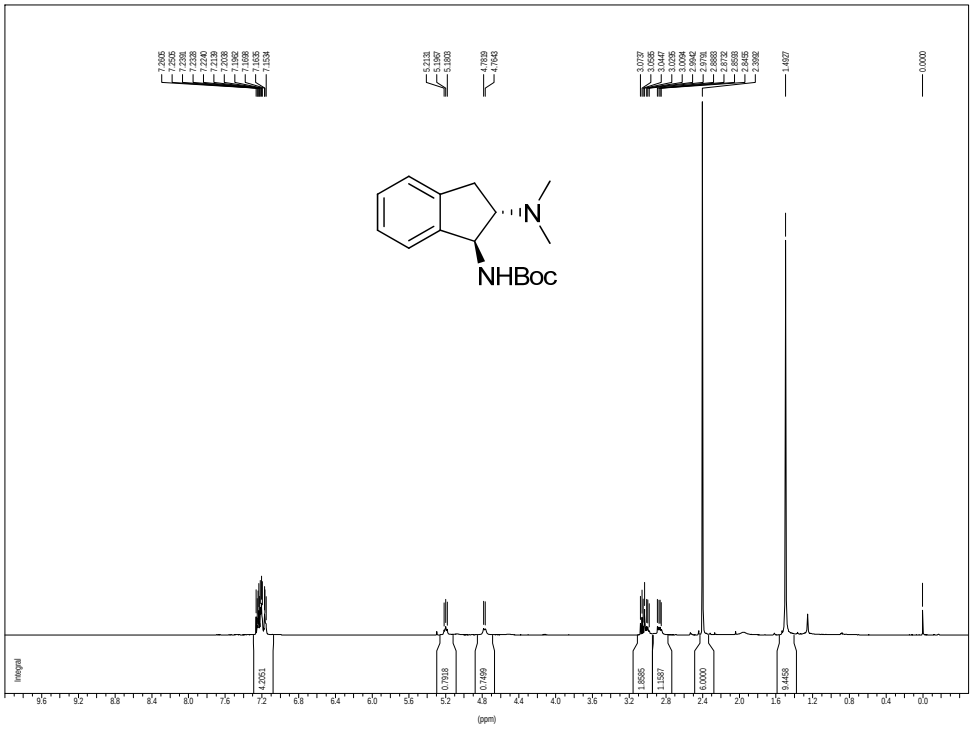

156.3012
140.8228
139.8244
128.1792
127.1225
125.3226
124.4699
79.8858
77.2551
77.0000
76.7449
73.6551
58.8909
39.4117
28.3932
OH
NHBoc
180
160
140
120
100
80
60
40
20
0
(ppm)

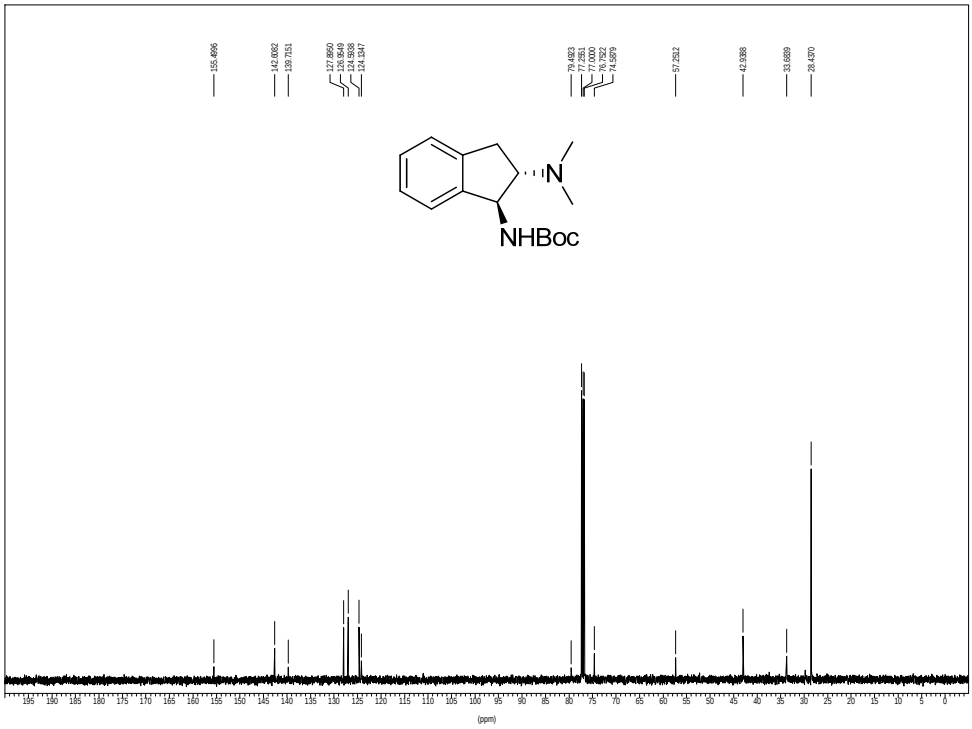
Compound **c**


7.2600
7.2064
7.1868
4.9460
4.7936
4.7692
4.7436
3.4595
3.4329
3.4085
3.3842
3.2293
3.2049
3.1781
3.1525
2.6758
2.6478
2.6234
2.5966
1.7504
1.4932
NH2
NHBoc
Integral
3.9539
1.9937
1.0000
1.0177
1.0079
2.2326
9.2412
(ppm)

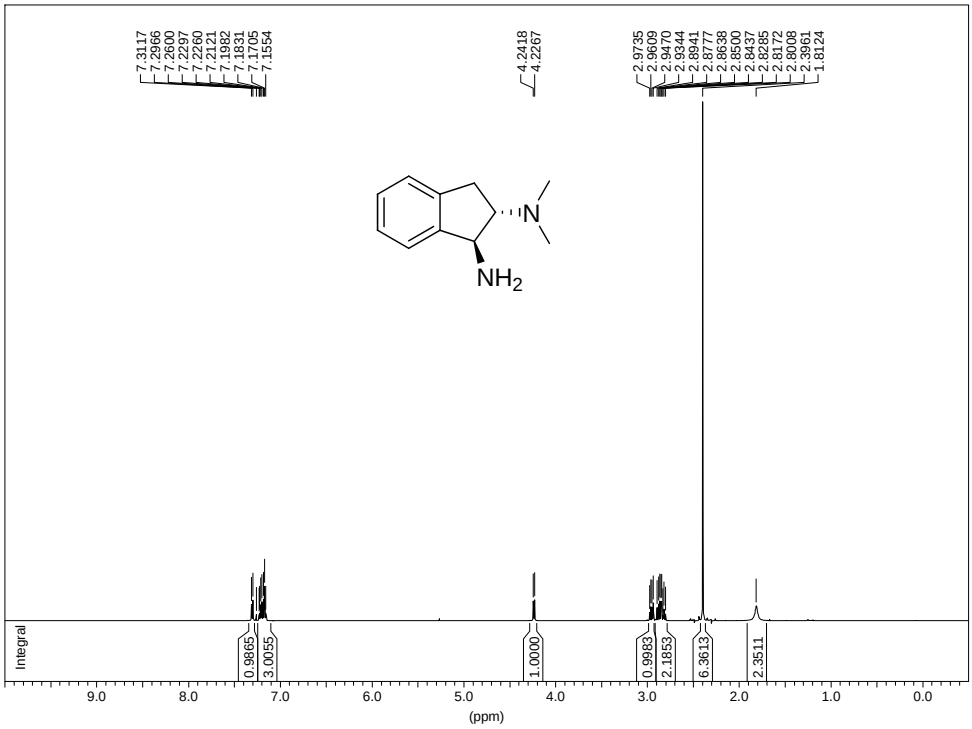

156.2841
141.5429
140.5965
128.0636
126.5980
124.7727
123.7976
79.6457
77.4230
77.0000
76.5770
64.5173
62.3878
39.2508
28.3669
NH2
NHBoc
(ppm)

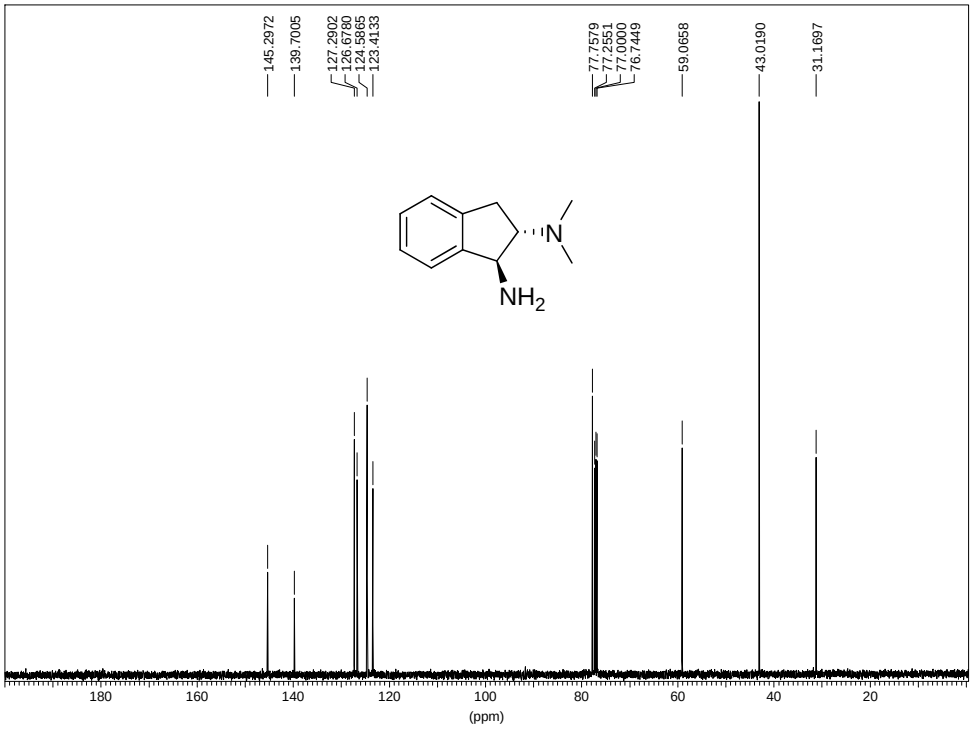
Compound **d**


7.3605
7.2505
7.2328
7.2328
7.2210
7.2210
7.2038
7.1962
7.1962
7.1638
7.1638
7.1534
6.2133
5.1967
5.1803
4.7929
4.7644
3.0737
3.0595
3.0447
3.0295
3.0094
3.0047
2.9791
2.8883
2.8712
2.8568
2.8495
2.3992
1.4927
0.0000
N
NHBoc
Integral
4.2051
0.7918
0.7499
1.8585
1.1587
6.0000
9.4458
(ppm)

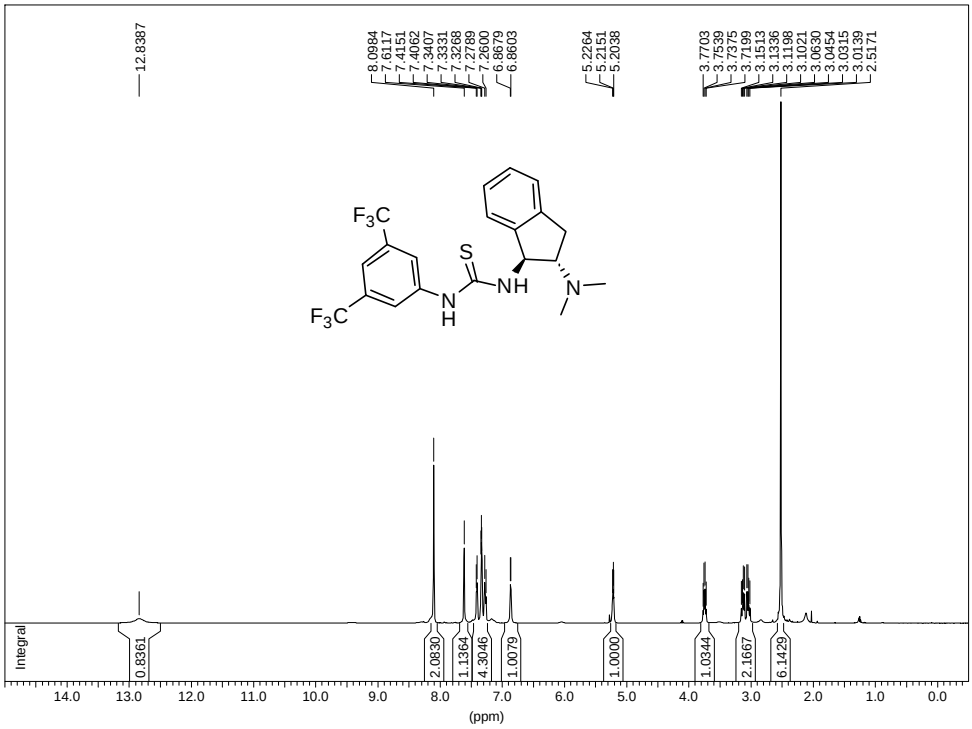

155.4066
142.0082
139.7151
127.8950
125.9549
124.5838
124.1347
79.4823
77.3201
77.0000
76.7922
74.5879
57.2512
42.9398
33.6839
28.4370
N
NHBoc
(ppm)

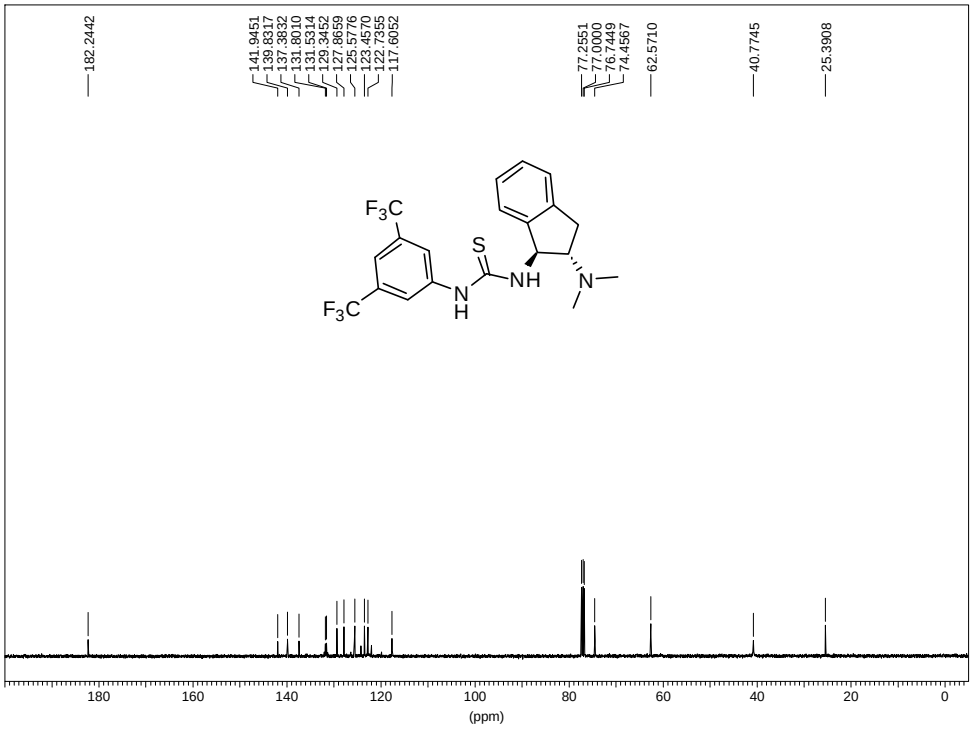
Compound **g**


7.3117
7.2966
7.2600
7.2297
7.2260
7.2121
7.1982
7.1831
7.1705
7.1554
4.2418
4.2267
2.9735
2.9609
2.9470
2.9344
2.8941
2.8777
2.8638
2.8500
2.8437
2.8285
2.8172
2.8008
2.3961
1.6124
N
NH2
Integral
0.9865
3.0055
1.0000
0.9983
2.1853
6.3613
2.3511
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

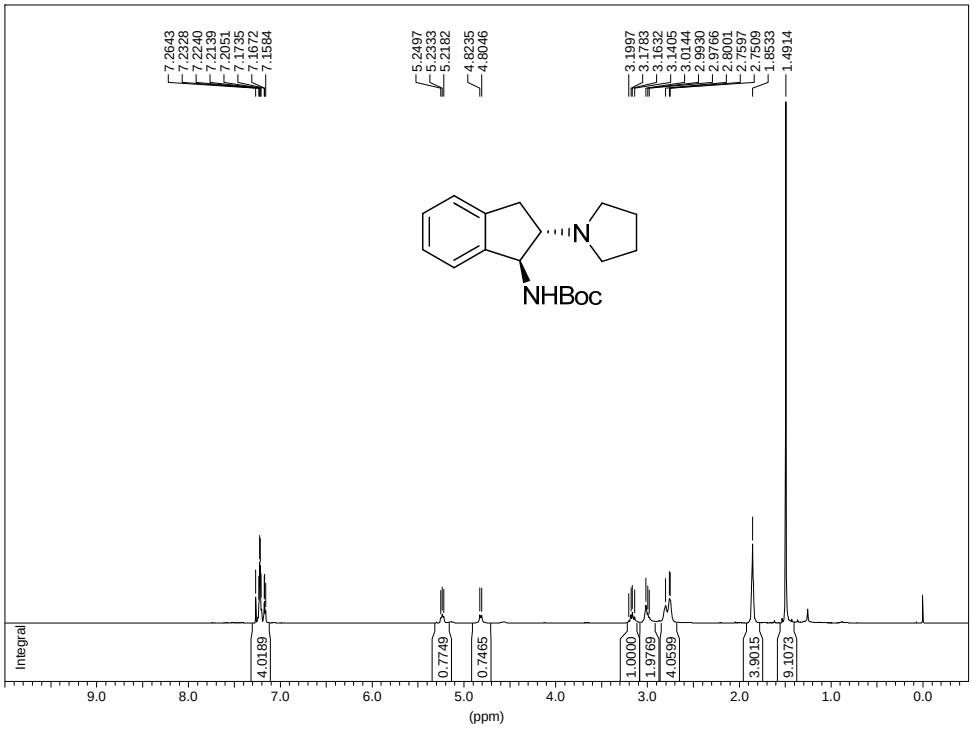

145.2972
139.7005
127.2902
126.6780
124.5865
123.4133
77.7579
77.2551
77.0000
76.7449
59.0658
43.0190
31.1697
N
NH2
180
160
140
120
100
80
60
40
20
(ppm)

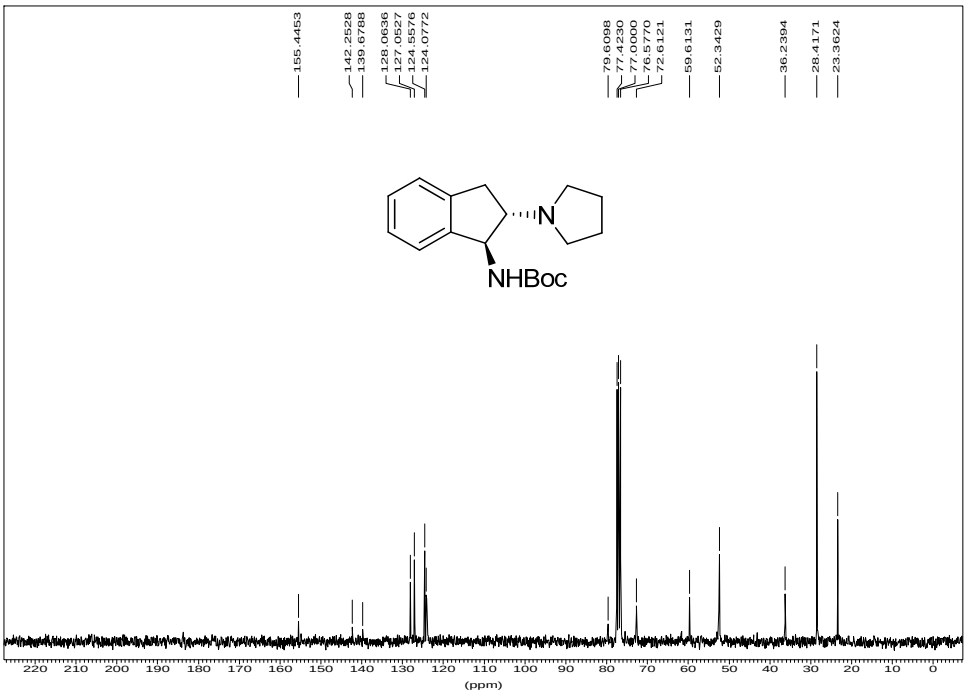
Compound **3a**


12.8387
8.0984
7.6117
7.4151
7.4062
7.3407
7.3331
7.3288
7.2789
7.2600
6.8679
6.8603
5.2264
5.2151
5.2038
3.7703
3.7539
3.7375
3.7199
3.1513
3.1336
3.1198
3.1021
3.0630
3.0454
3.0315
3.0139
2.5171
F3C
S
NH
N
H
F3C
N
Integral
0.8361
2.0830
1.1364
4.3046
1.0079
1.0000
1.0344
2.1667
6.1429
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

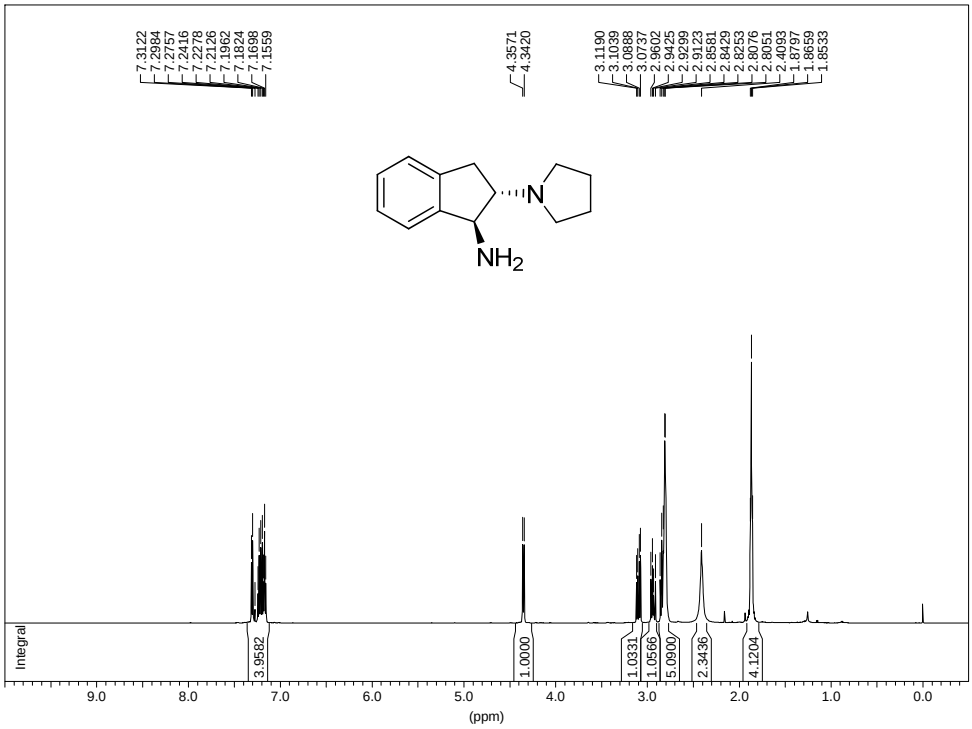

182.2442
141.9451
139.8317
137.3832
131.8010
131.5314
129.3452
127.8659
125.5776
123.4570
122.7355
117.6052
77.2551
77.0000
76.7449
74.4567
62.5710
40.7745
25.3908
F3C
S
NH
N
H
F3C
N
180
160
140
120
100
80
60
40
20
0
(ppm)

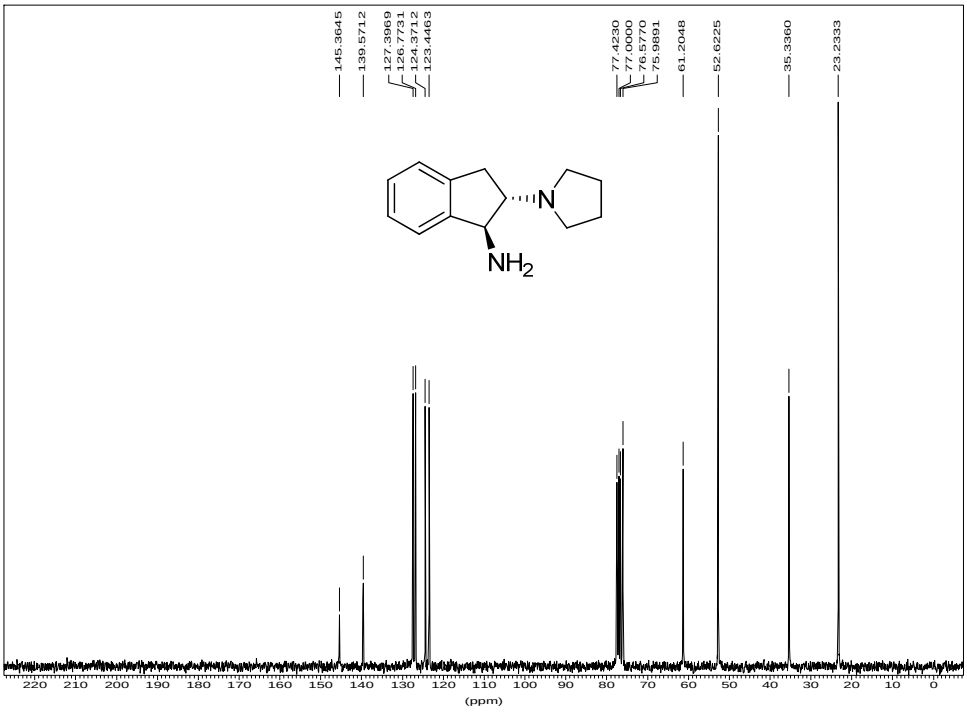
Compound **e**


7.2643
7.2328
7.2240
7.2139
7.2051
7.1735
7.1672
7.1584
5.2497
5.2333
5.2182
4.8235
4.6046
3.1997
3.1783
3.1632
3.1405
3.0144
2.9930
2.9766
2.8001
2.7597
2.7509
1.8533
1.4914
N
NHBoc
Integral
4.0189
0.7749
0.7465
1.0000
1.9769
4.0599
3.9015
9.1073
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

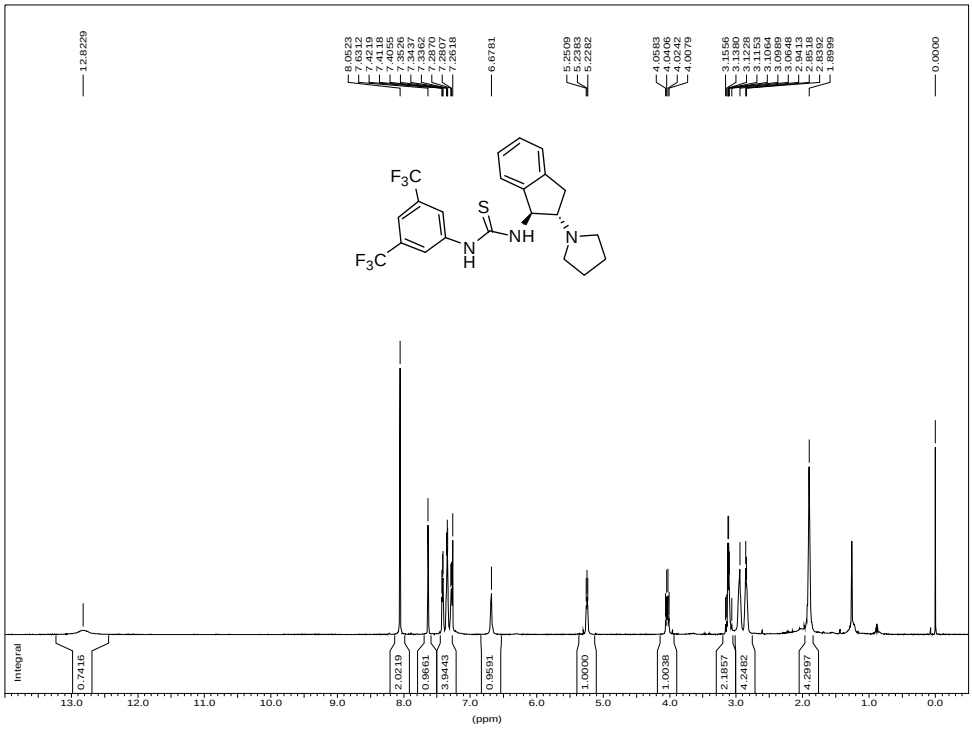

155.4453
142.2528
139.6788
128.0636
127.0527
124.5576
124.0772
79.6098
77.4230
77.0000
76.5770
72.6121
59.6131
52.3429
36.2394
28.4171
23.3624
N
NHBoc
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

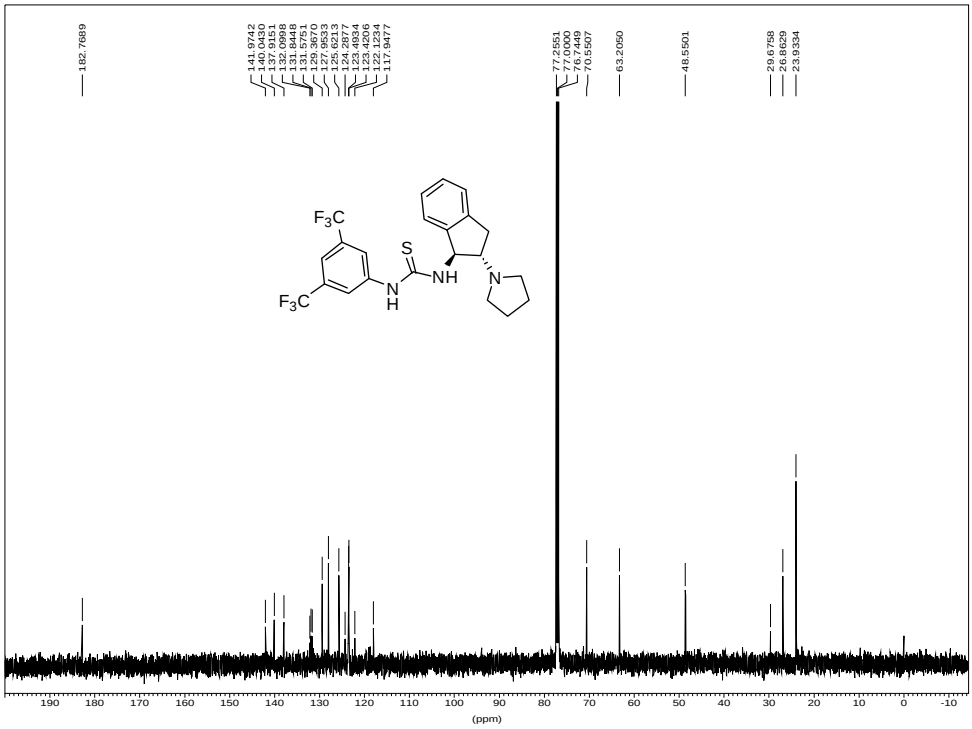
Compound **h**


7.3122
7.2984
7.2757
7.2416
7.2278
7.2126
7.1962
7.1824
7.1698
7.1559
4.3571
4.3420
3.1190
3.1039
3.0888
3.0737
2.9602
2.9425
2.9299
2.9123
2.8581
2.8429
2.8253
2.8076
2.8051
2.4093
1.8797
1.8659
1.8533
N
NH2
Integral
3.9582
1.0000
1.0331
1.0566
5.0900
2.3436
4.1204
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

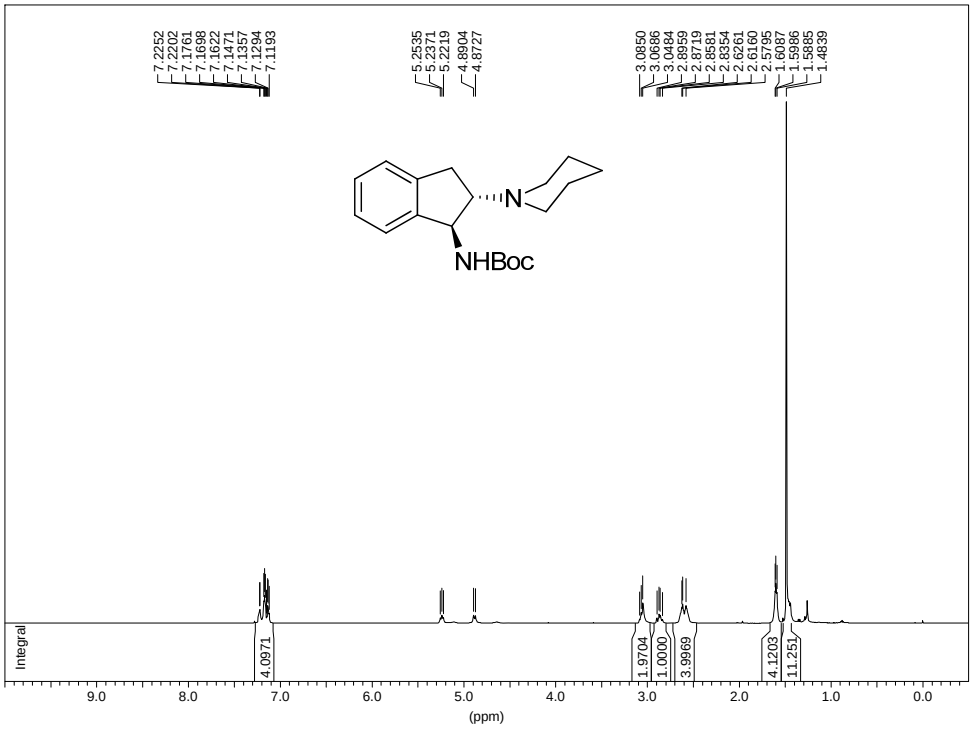

145.3645
139.5712
127.3969
126.7731
124.3712
123.4463
77.4230
77.0000
76.5770
75.9891
61.2048
52.6225
35.3360
23.2333
N
NH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

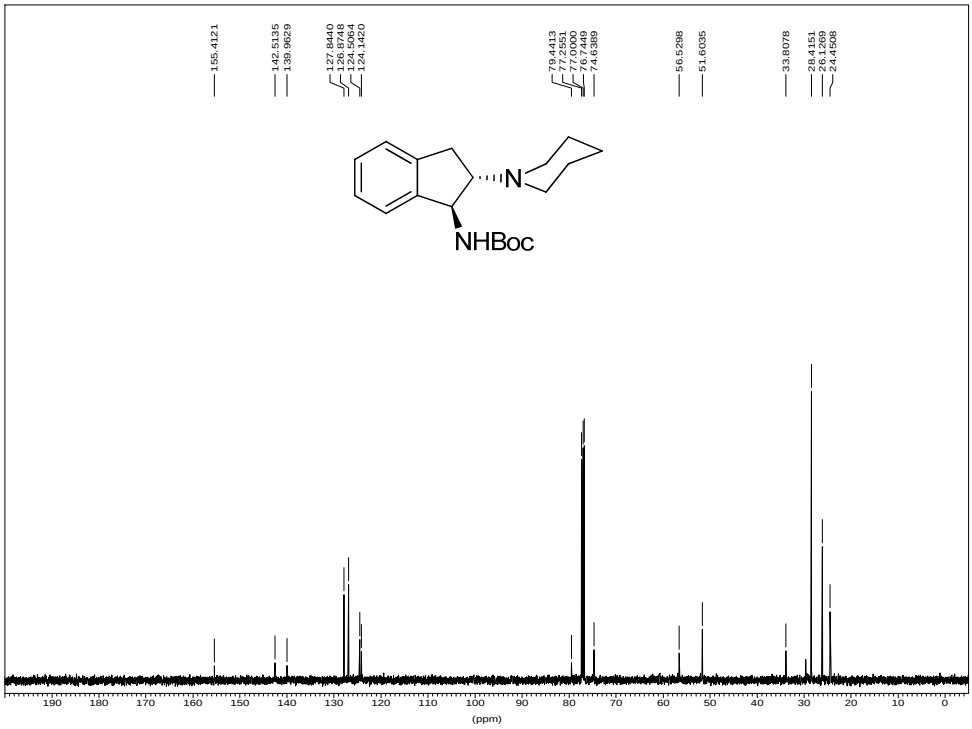
Compound **3b**


12.8229
8.0523
7.4312
7.4118
7.4055
7.3826
7.3447
7.3362
7.2870
7.2807
7.2618
6.6781
5.2509
5.2383
5.2282
4.0583
4.0406
4.0242
4.0079
3.1556
3.1390
3.1228
3.1153
3.1064
3.0949
3.0845
2.9413
2.8518
2.8092
1.8998
0.0000
F3C
F3C
S
NH
N
H
N
Integral
0.7416
2.0219
0.9661
3.9443
0.9591
1.0000
1.0038
2.1857
4.2482
4.2997
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

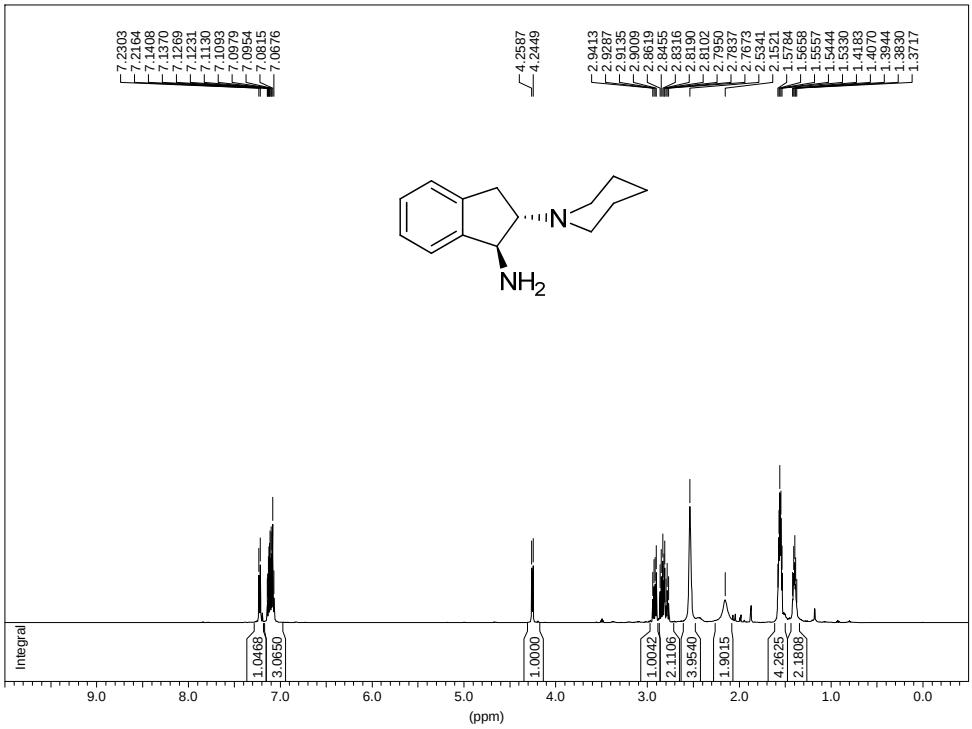

182.7689
141.9742
140.0430
137.1511
132.0998
131.8448
131.5751
129.2870
127.9553
125.6213
124.2877
123.4654
123.4206
122.1234
117.9477
77.2551
77.0000
76.7449
70.5507
63.2050
48.5501
29.6758
26.8629
23.9334
F3C
F3C
S
NH
N
H
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
(ppm)

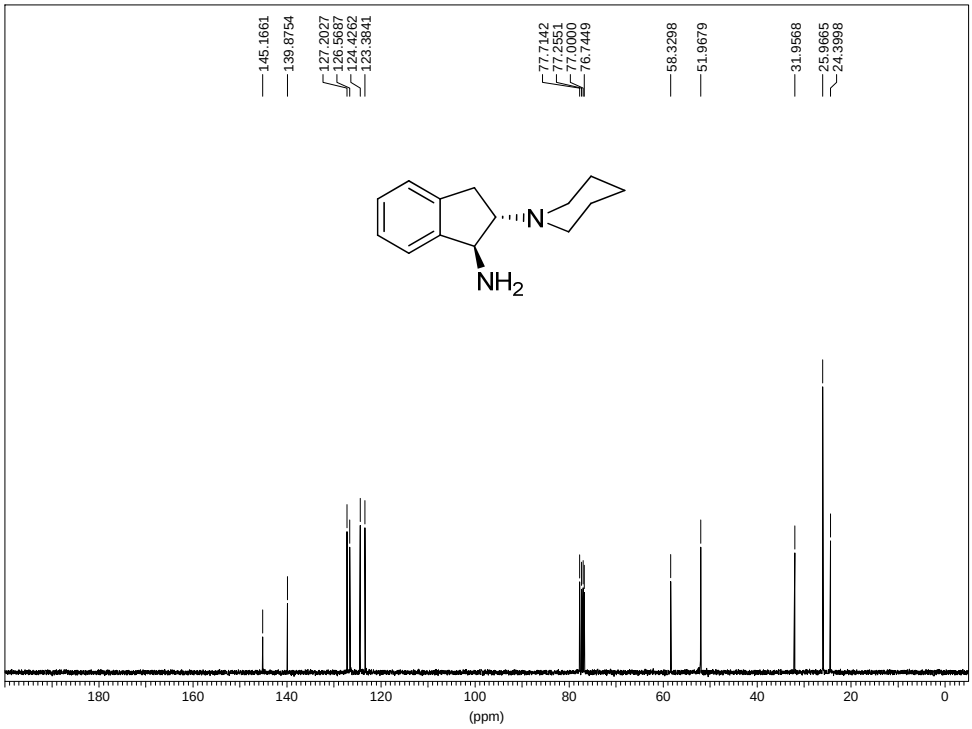
Compound **f**


7.2252
7.2202
7.1761
7.1698
7.1622
7.1471
7.1357
7.1294
7.1193
5.2535
5.2371
5.2219
4.8904
4.8727
3.0850
3.0686
3.0484
2.8959
2.8719
2.8581
2.8354
2.6261
2.6160
2.5795
1.6087
1.5986
1.5885
1.4839
N
NHBoc
Integral
4.0971
1.9704
1.0000
3.9969
4.1203
11.251
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

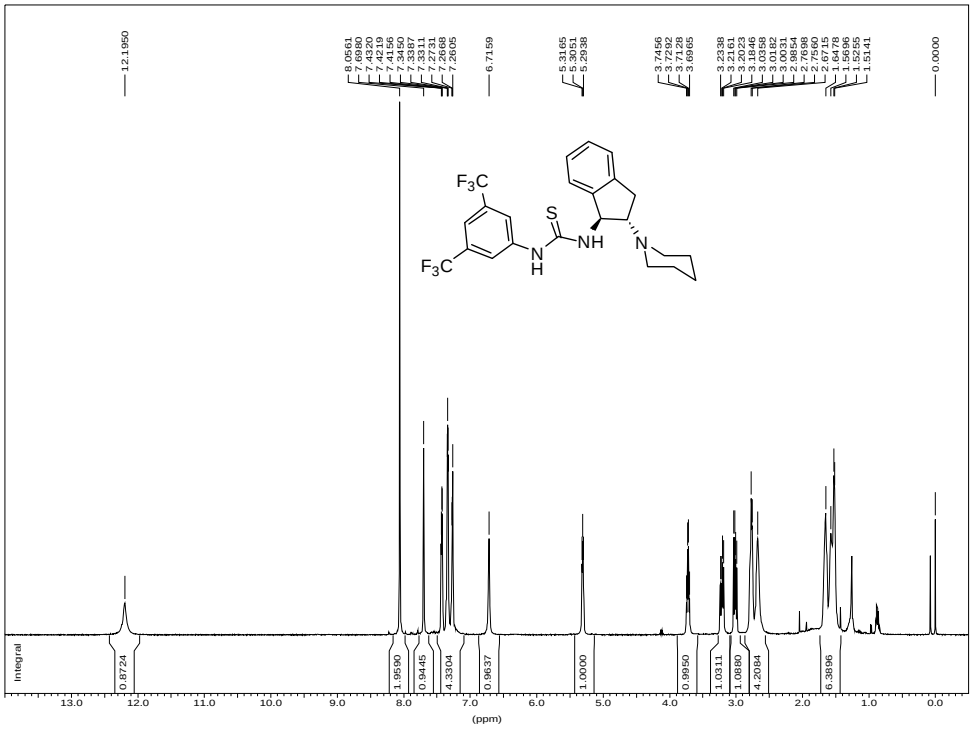

155.4121
142.5135
139.9629
127.9440
126.8748
124.5064
124.1420
79.4413
77.2551
77.0000
76.7449
74.6389
56.5298
51.6035
33.8078
28.4151
26.1269
24.4508
N
NHBoc
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

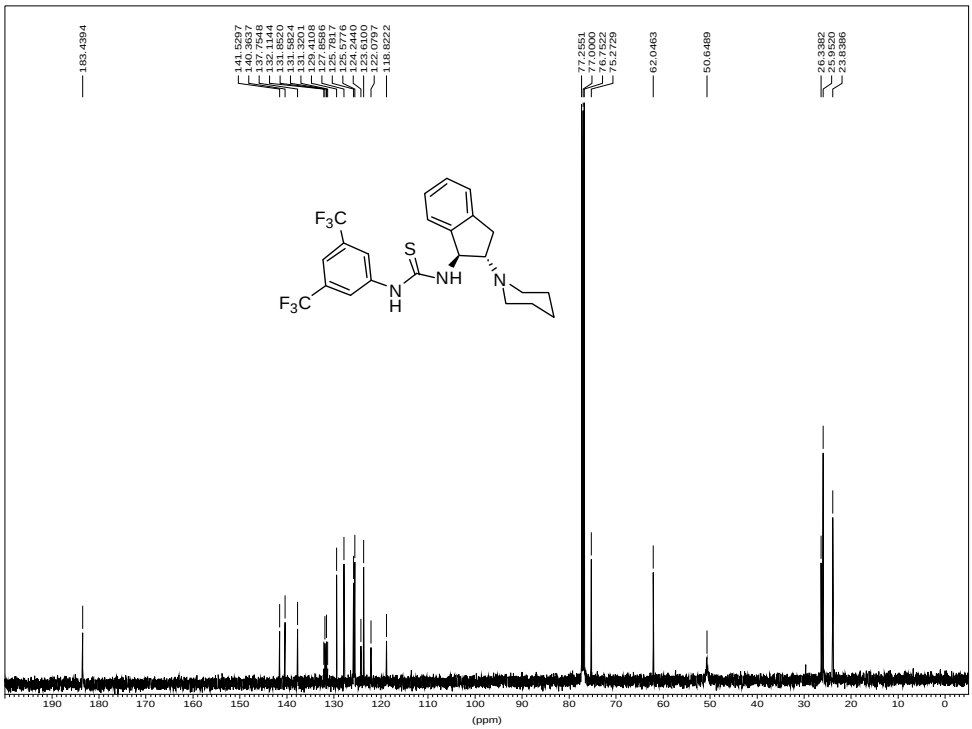
Compound **i**


7.2303
7.2164
7.1408
7.1370
7.1269
7.1231
7.1130
7.1093
7.0979
7.0954
7.0815
7.0676
4.2587
4.2449
2.9413
2.9287
2.9135
2.9009
2.8619
2.8455
2.8316
2.8190
2.8102
2.7950
2.7837
2.7673
2.5341
2.1521
1.5784
1.5658
1.5557
1.5444
1.5330
1.4183
1.4070
1.3944
1.3830
1.3717
N
NH2
Integral
1.0468
3.0650
1.0000
1.0042
2.1106
3.9540
1.9015
4.2625
2.1808
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

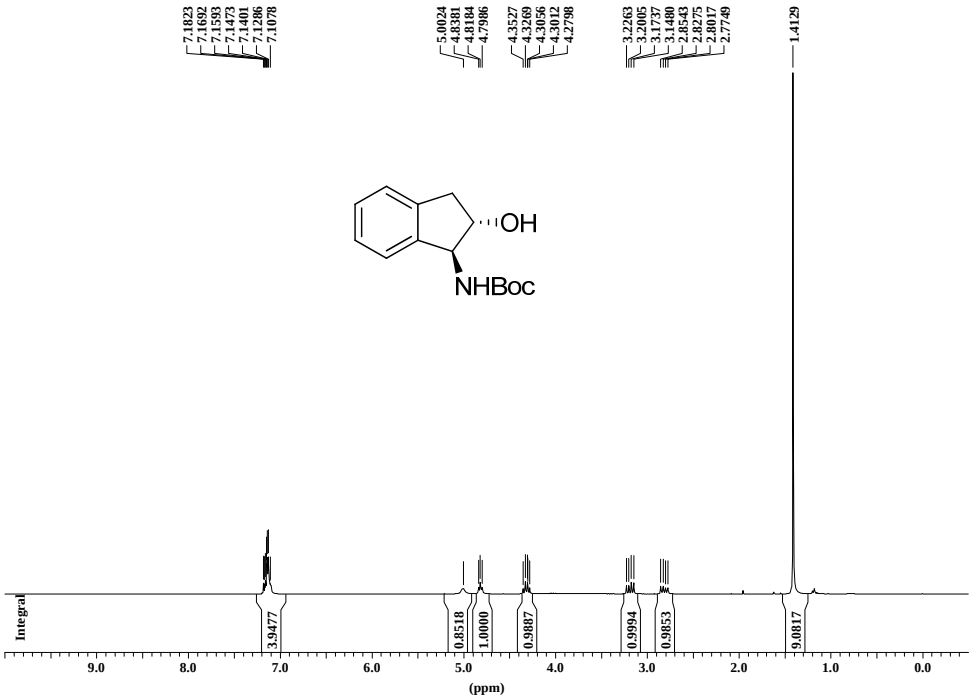

145.1661
139.8754
127.2027
126.5687
124.4262
123.3841
77.7142
77.2551
77.0000
76.7449
58.3298
51.9679
31.9568
25.9665
24.3998
N
NH2
180
160
140
120
100
80
60
40
20
0
(ppm)

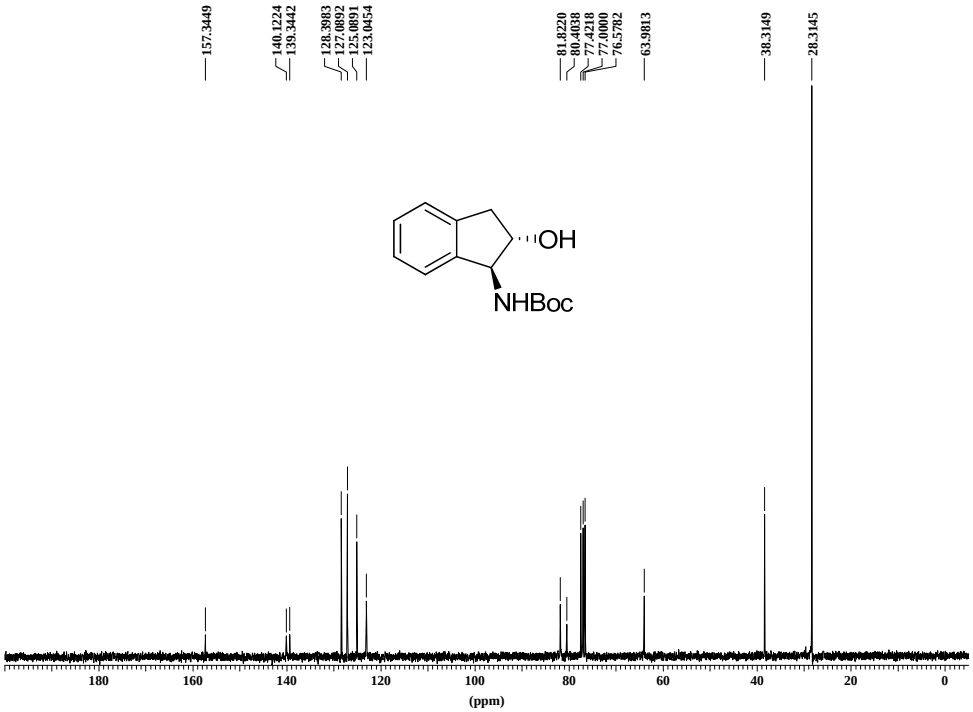
Compound **3c**


12.1950
8.0561
7.6980
7.4530
7.4219
7.4156
7.3490
7.3367
7.3311
7.2731
7.2668
7.2605
6.7159
5.3165
5.3051
5.2938
3.7456
3.7292
3.7128
3.6965
3.2338
3.2161
3.2023
3.1846
3.0939
3.0182
3.0031
2.9854
2.7668
2.7560
2.6715
1.6478
1.5966
1.5255
1.5141
0.0000
F3C
F3C
S
NH
N
H
N
Integral
0.8724
1.9590
0.9445
4.3304
0.9637
1.0000
0.9950
1.0311
1.0880
4.2084
6.3896
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

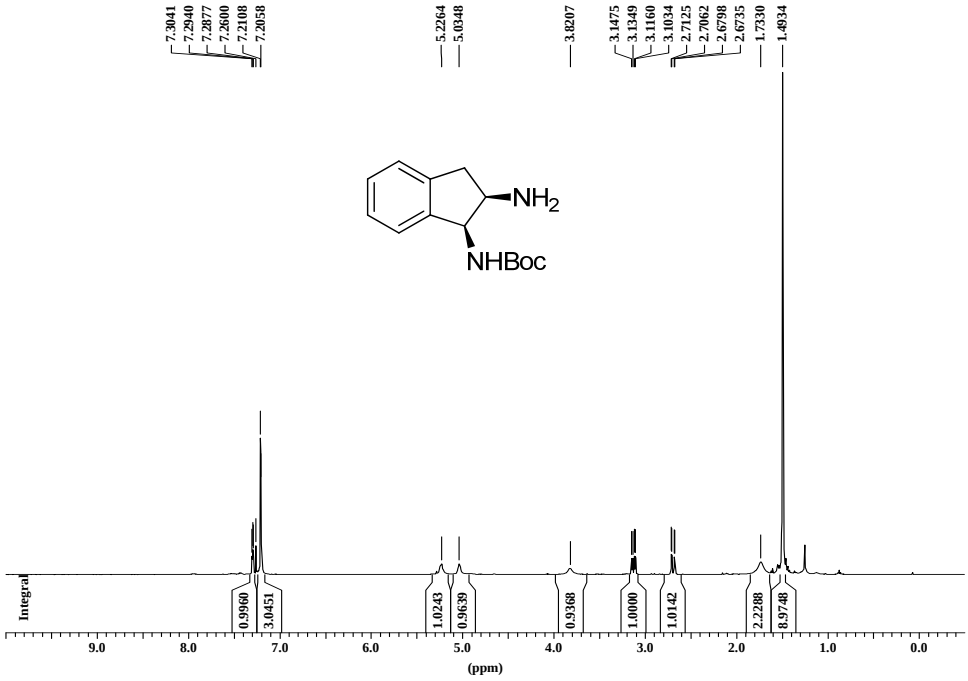

183.4394
141.5297
140.3637
137.7548
132.1144
131.8520
131.5824
131.3201
129.4108
127.8586
125.7817
125.5776
124.2440
123.6100
122.0797
118.8222
77.2551
77.0000
76.7522
75.2729
62.0463
50.6489
26.3382
25.8520
23.8386
F3C
F3C
S
NH
N
H
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

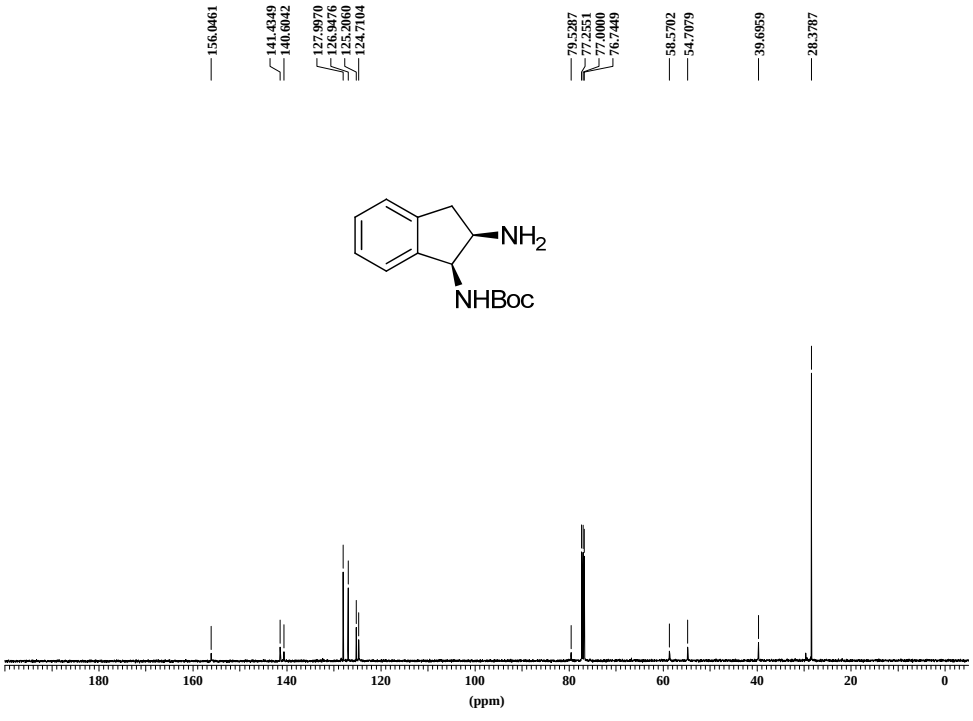
Compound **k**


7.1823
7.1692
7.1593
7.1473
7.1401
7.1286
7.1078
5.0024
4.8381
4.8184
4.7986
4.3527
4.3269
4.3056
4.3012
4.2798
3.2263
3.2005
3.1737
3.1480
2.8543
2.8275
2.8017
2.7749
1.4129
OH
NHBoc
Integral
3.9477
0.8518
1.0000
0.9887
0.9994
0.9853
9.0817
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

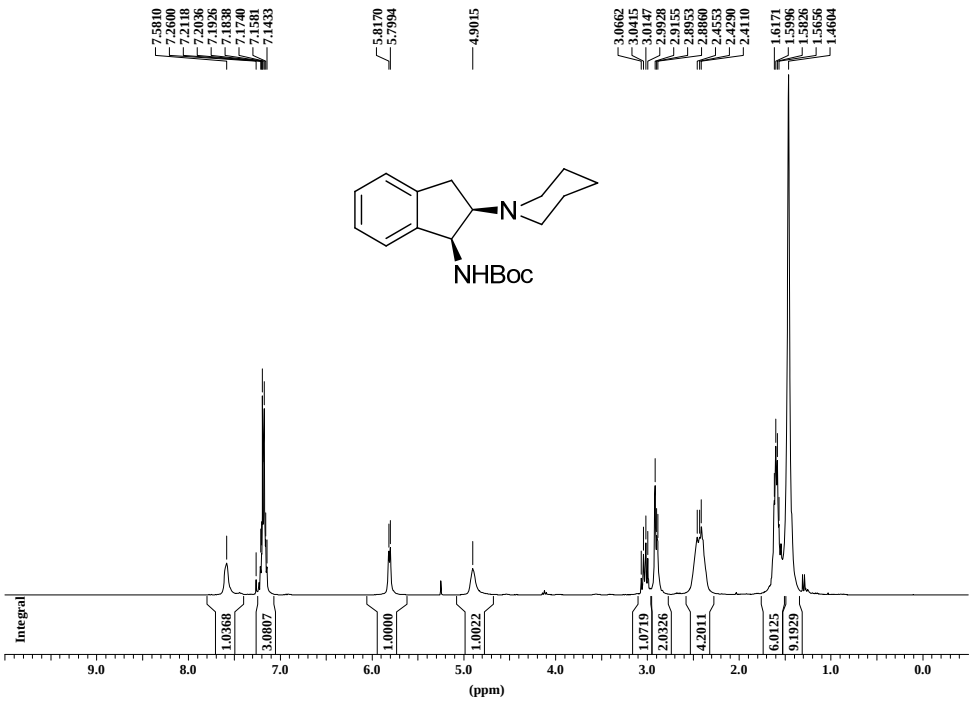

157.3449
140.1224
139.3442
128.3983
127.0892
125.0891
123.0454
81.8220
80.4038
77.4218
77.0000
76.5782
63.9813
38.3149
28.3145
OH
NHBoc
180
160
140
120
100
80
60
40
20
0
(ppm)

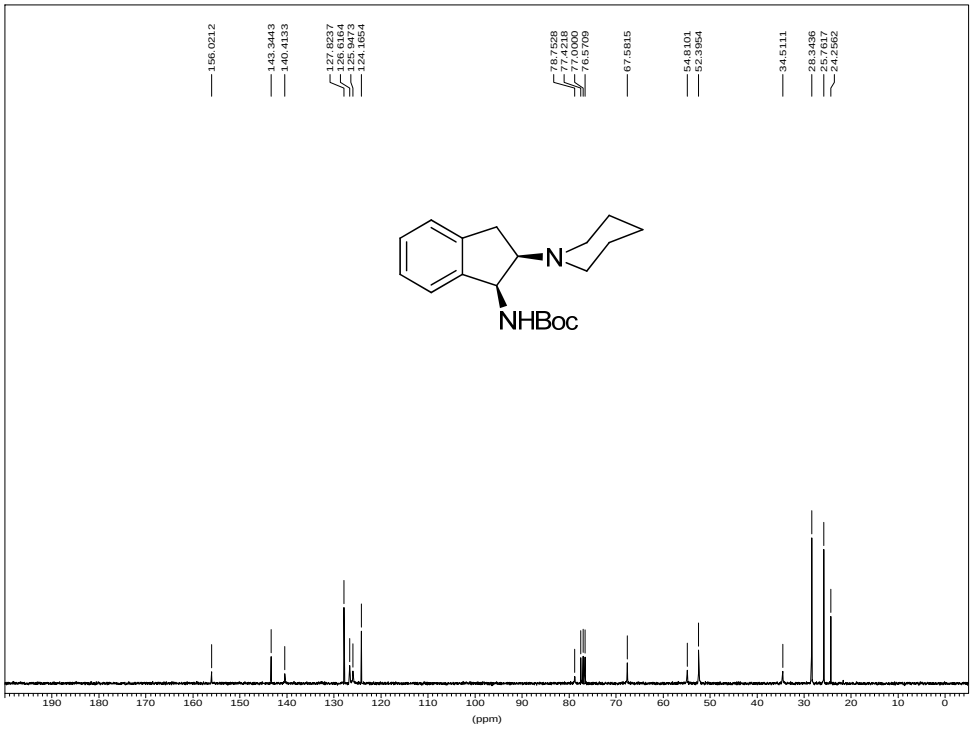
Compound **l**


7.3041
7.2940
7.2877
7.2600
7.2108
7.2058
5.2264
5.0348
3.8207
3.1475
3.1349
3.1160
3.1034
2.7125
2.7062
2.6798
2.6735
1.7330
1.4934
NH2
NHBoc
Integral
0.9960
3.0451
1.0243
0.9639
0.9368
1.0000
1.0142
2.2288
8.9748
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

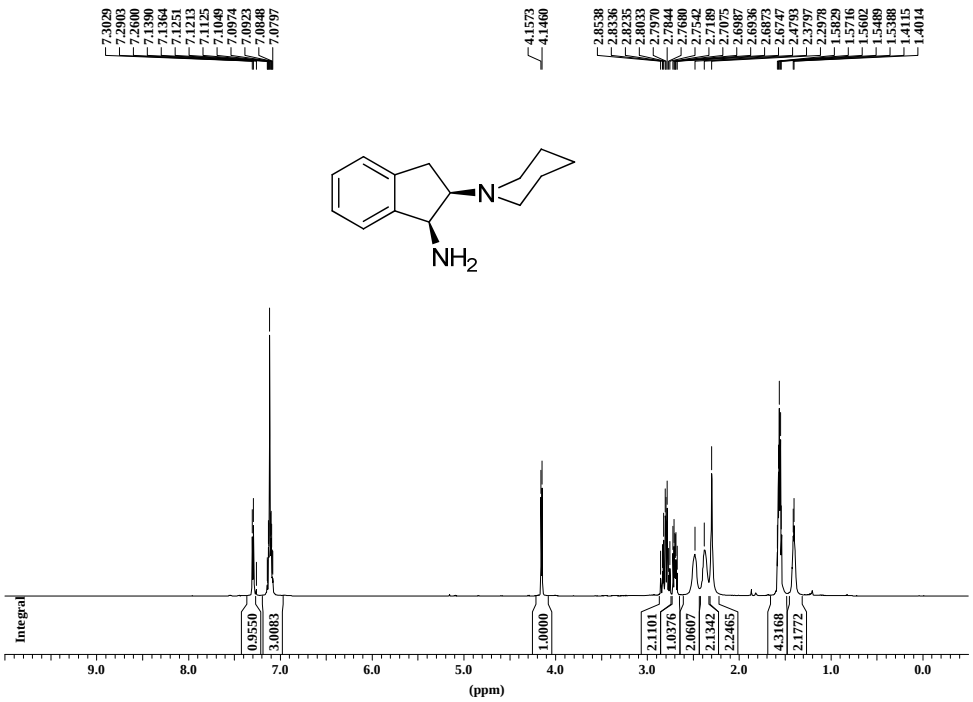

156.0461
141.4349
140.6042
127.9970
126.9476
125.2060
124.7104
79.5287
77.2551
77.0000
76.7449
58.5702
54.7079
39.6959
28.3787
NH2
NHBoc
180
160
140
120
100
80
60
40
20
0
(ppm)

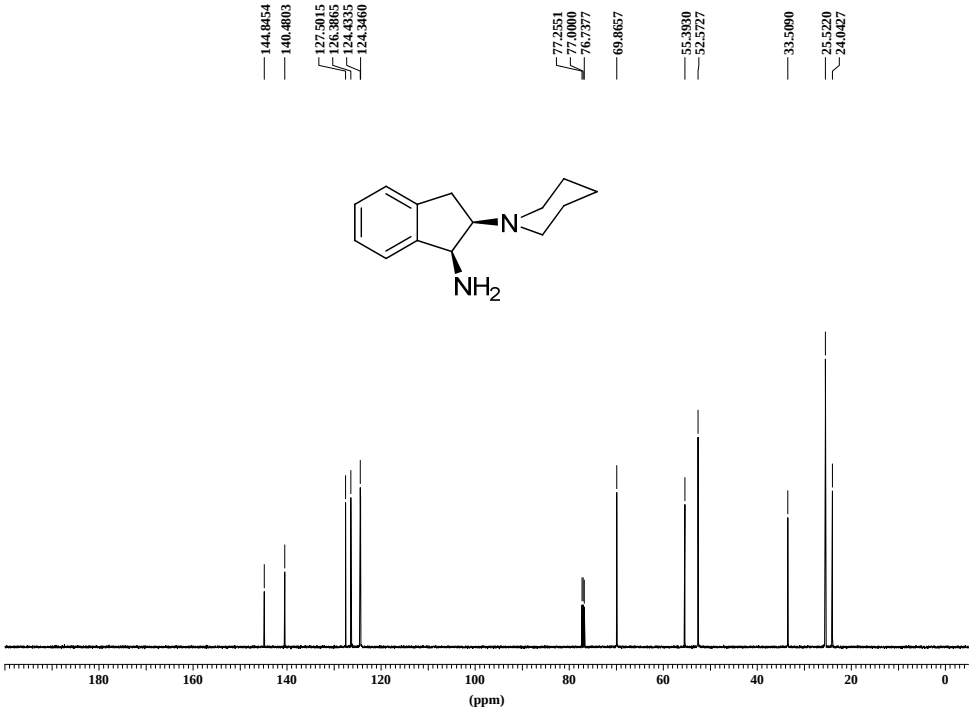
Compound **m**


7.5810
7.2600
7.2118
7.2036
7.1926
7.1838
7.1740
7.1581
7.1433
5.8170
5.7994
4.9015
3.0662
3.0415
3.0147
2.9928
2.9155
2.8953
2.8860
2.4553
2.4290
2.4110
1.6171
1.5996
1.5826
1.5656
1.4604
N
NHBoc
Integral
1.0368
3.0807
1.0000
1.0022
1.0719
2.0326
4.2011
6.0125
9.1929
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

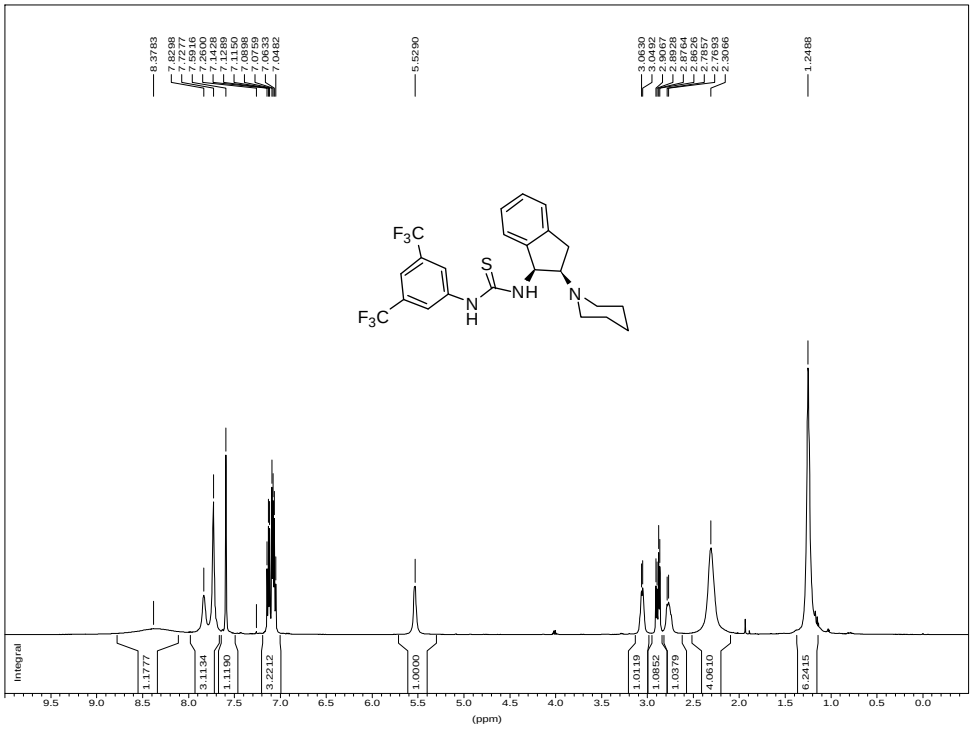

156.0212
143.3443
140.4133
127.8237
126.6164
125.9473
124.1654
79.7528
77.4218
77.0000
76.5709
67.5815
54.8101
52.3954
34.5111
28.3436
25.7617
24.2562
N
NHBoc
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)

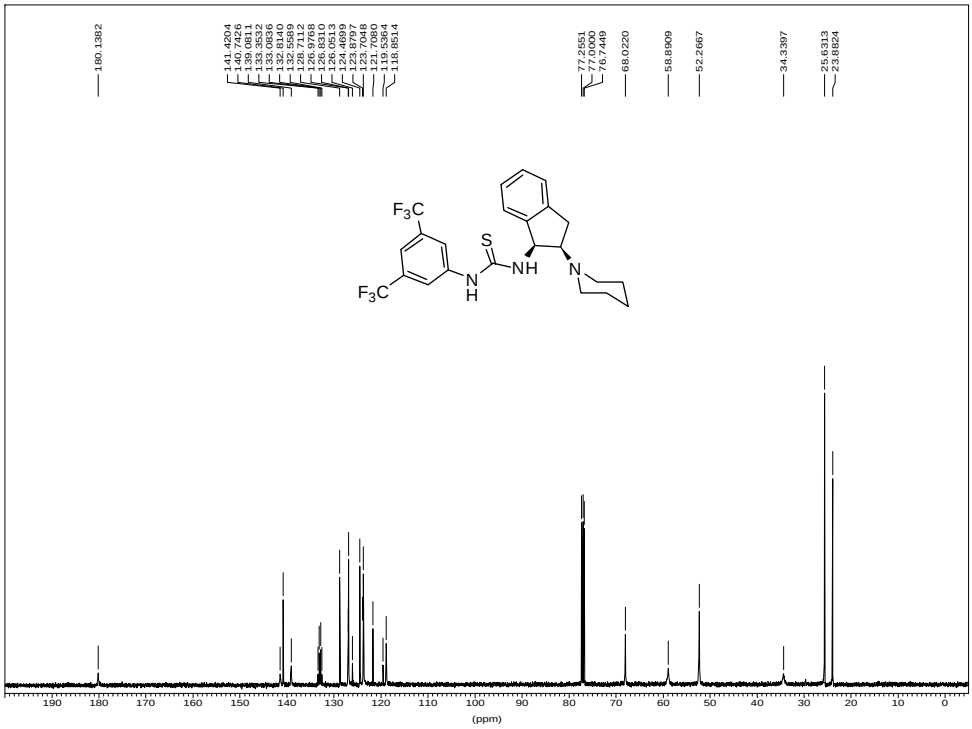
Compound **n**


7.3029
7.2903
7.2600
7.1390
7.1364
7.1251
7.1213
7.1125
7.1049
7.0974
7.0923
7.0848
7.0797
4.1573
4.1460
2.8538
2.8336
2.8235
2.8033
2.7970
2.7844
2.7680
2.7542
2.7189
2.7075
2.6987
2.6936
2.6873
2.6747
2.4793
2.3797
2.2978
1.5829
1.5716
1.5602
1.5489
1.5388
1.4115
1.4014
N
NH2
Integral
0.9550
3.0083
1.0000
2.1101
1.0376
2.0607
2.1342
2.2465
4.3168
2.1772
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

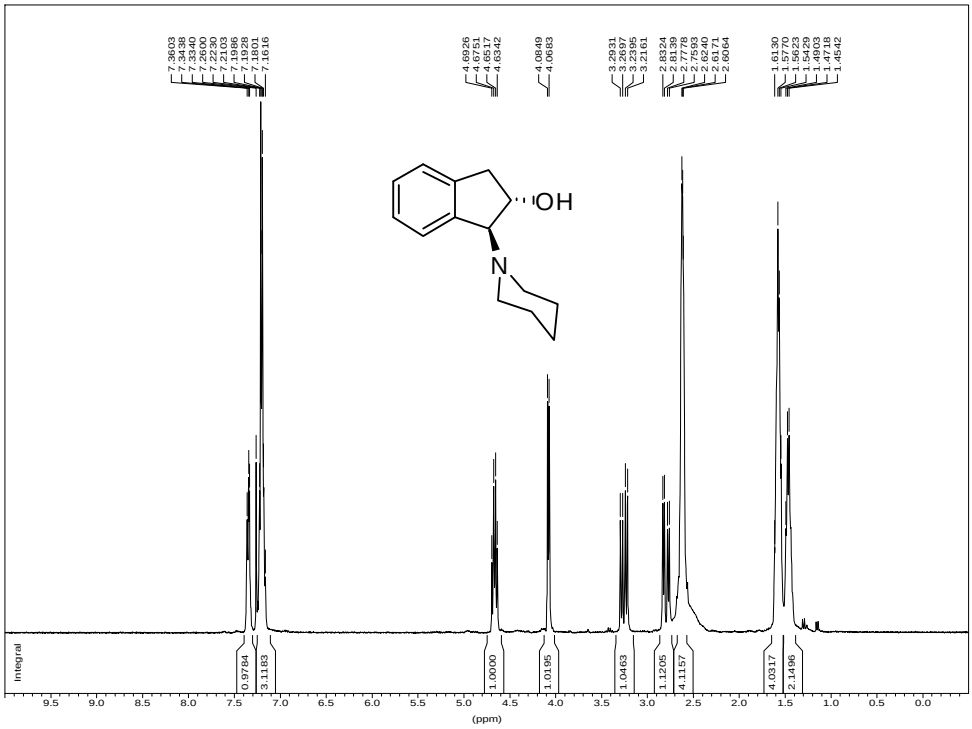

144.8454
140.4803
127.5015
126.3865
124.4335
124.3460
77.2551
77.0000
76.7377
69.8657
55.3930
52.5727
33.5090
25.5220
24.0427
N
NH2
180
160
140
120
100
80
60
40
20
0
(ppm)

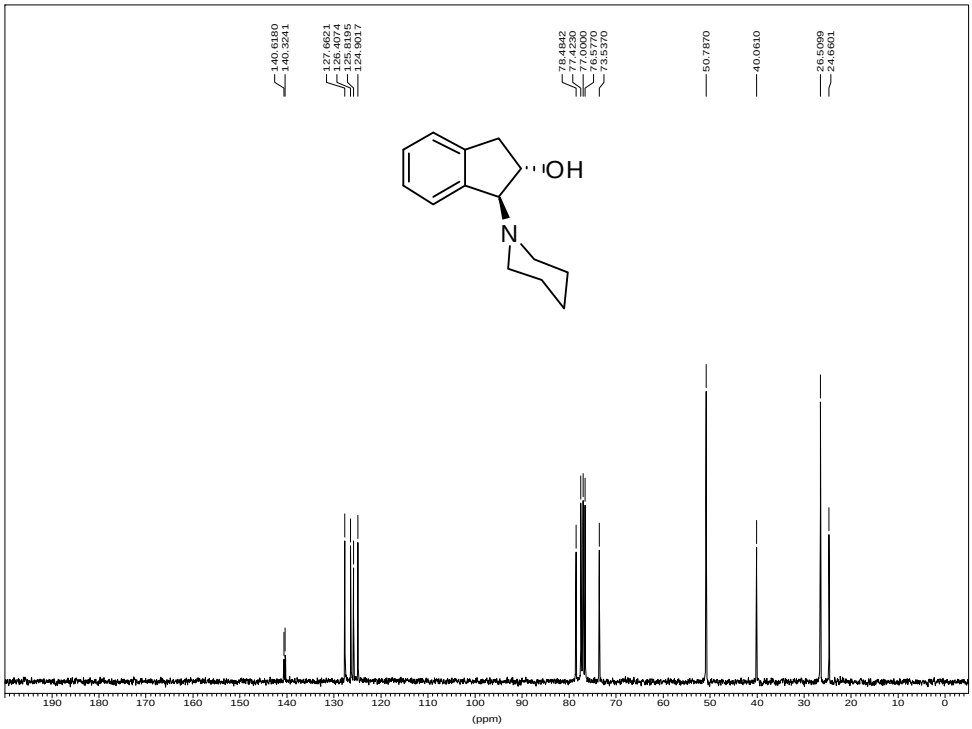
Compound **5**


8.3783
7.8298
7.7277
7.5916
7.5690
7.1428
7.1289
7.1190
7.0988
7.0759
7.0633
7.0482
5.5290
3.0630
3.0492
3.0367
2.8928
2.8764
2.8626
2.8457
2.7693
2.3066
1.2488
F3C
S
NH
N
H
F3C
Integral
1.1777
3.1134
1.1190
3.2212
1.0000
1.0119
1.0852
1.0379
4.0610
6.2415
(ppm)


180.1382
141.4204
140.7426
139.0811
138.3532
133.0836
132.8140
132.5589
128.7712
126.9768
126.8310
126.0513
124.4699
123.8797
123.7048
121.7080
119.5564
118.8514
77.2551
77.0000
76.7449
68.0220
58.8909
52.2667
34.3397
25.6313
23.8824
F3C
S
NH
N
H
F3C
(ppm)

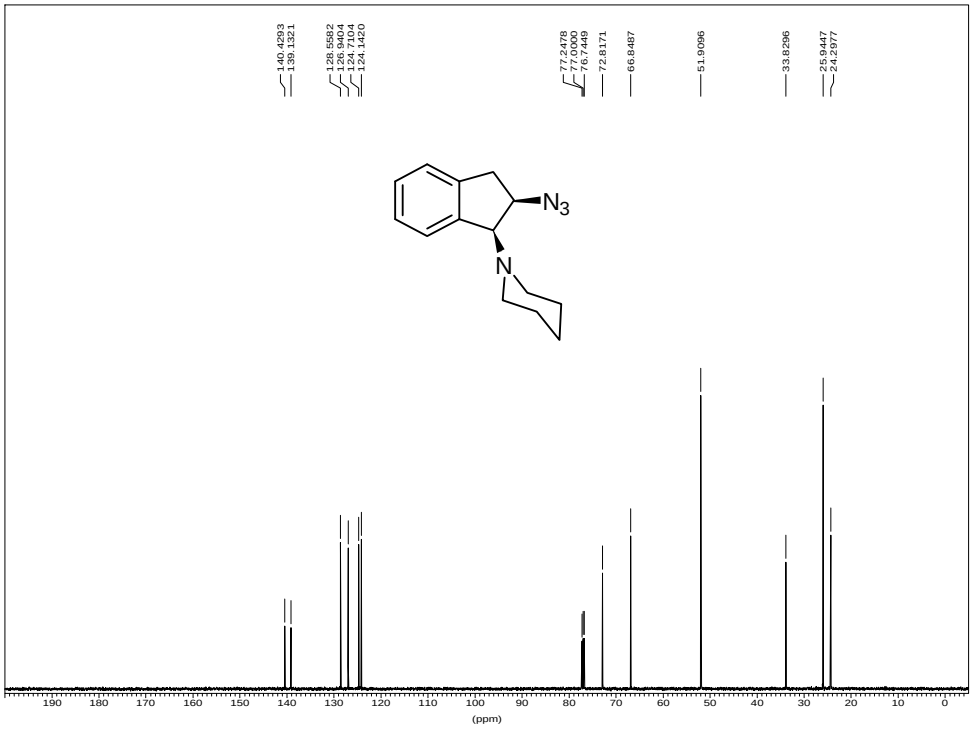
Compound **o**


7.3603
7.3438
7.3340
7.2660
7.2230
7.2103
7.1986
7.1938
7.1801
7.1616
4.6926
4.6751
4.6517
4.6342
4.0849
4.0683
3.2931
3.2697
3.2395
3.2161
2.8324
2.8139
2.7778
2.7593
2.6240
2.6171
2.6064
1.6130
1.5770
1.5623
1.5429
1.4963
1.4718
1.4542
OH
N
Integral
0.9784
3.1183
1.0000
1.0195
1.0463
1.1205
4.1157
4.0317
2.1496
(ppm)

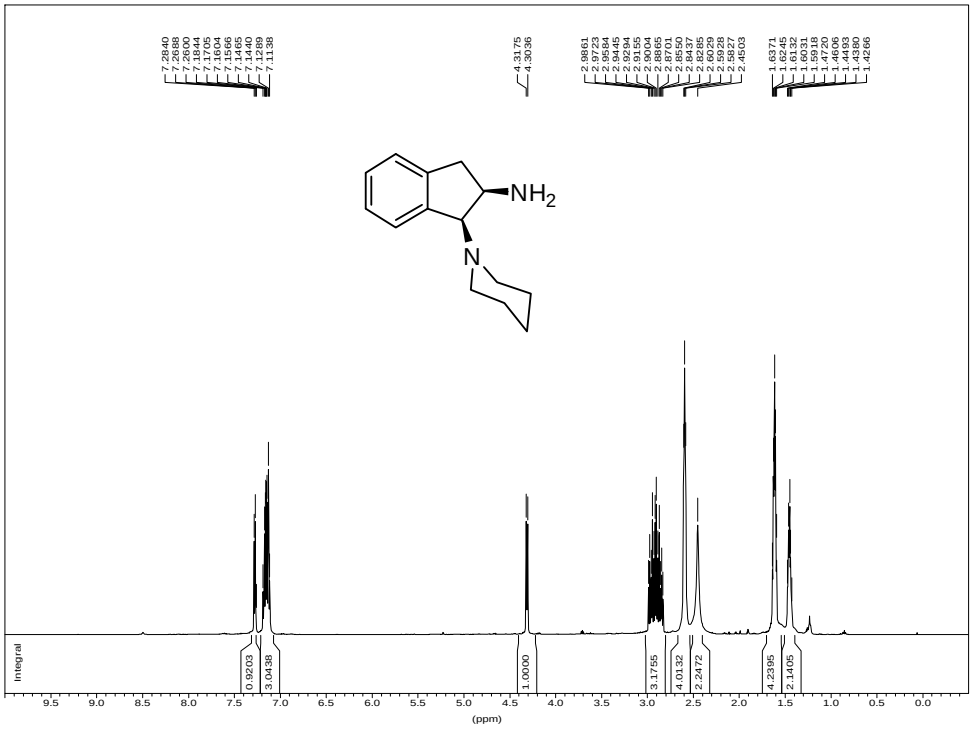

140.6180
140.3241
127.6621
126.4074
125.8195
124.9017
78.4842
77.4230
77.0000
75.5770
73.5370
50.7870
40.0610
26.5099
24.6601
OH
N
(ppm)

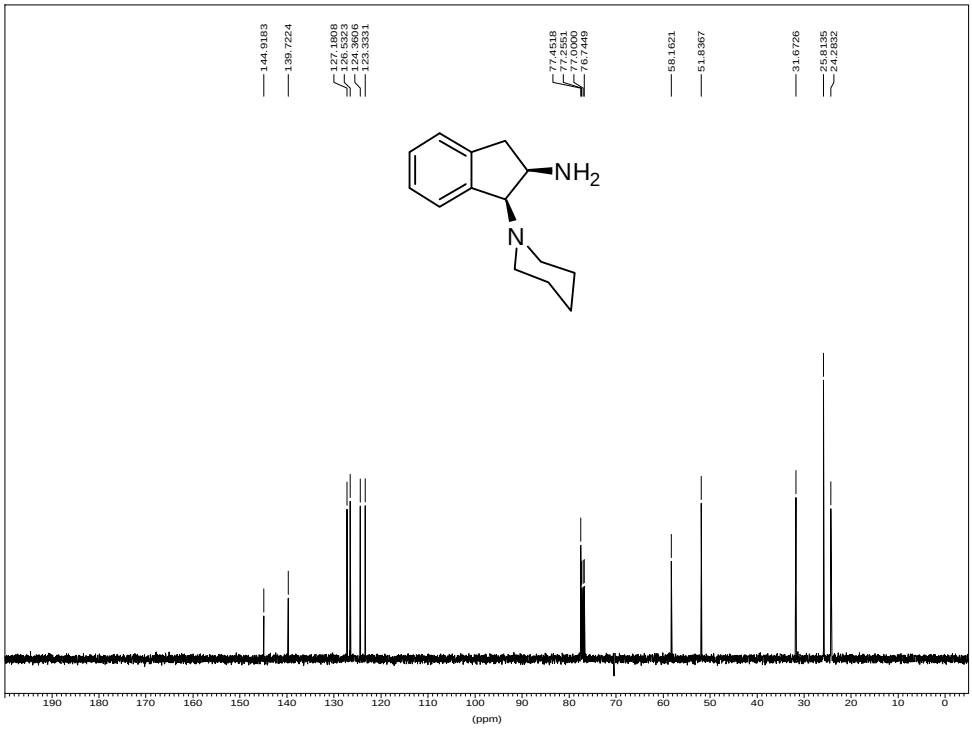
Compound **p**


7.2568
7.2454
7.2404
7.1660
7.1622
7.1159
7.1065
7.1446
7.1118
7.1067
7.0954
4.6395
4.6269
3.1934
3.1770
3.1632
3.1480
3.0585
3.0441
3.0257
3.0106
2.8341
2.8177
2.8026
2.7862
2.5328
2.5215
2.5114
2.5013
2.4672
2.4552
2.4458
2.4345
2.4244
2.4150
1.5457
1.5230
1.5116
1.4000
1.3944
1.3830
1.3717
1.3603
N3
N
Integral
3.8837
0.9410
1.0000
1.0378
1.0261
4.1220
4.1275
2.1250
(ppm)

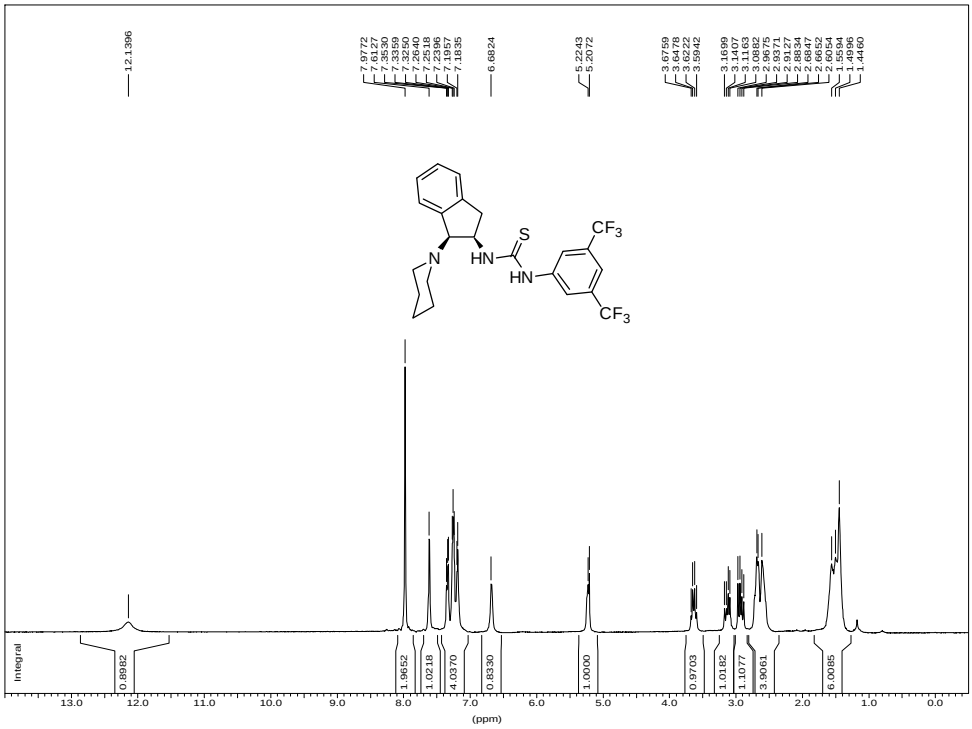

140.4293
139.1321
128.5582
126.9404
124.7104
124.1420
77.2478
77.0000
76.7449
72.8171
66.8487
51.9096
33.8296
25.9447
24.2977
N3
N
(ppm)

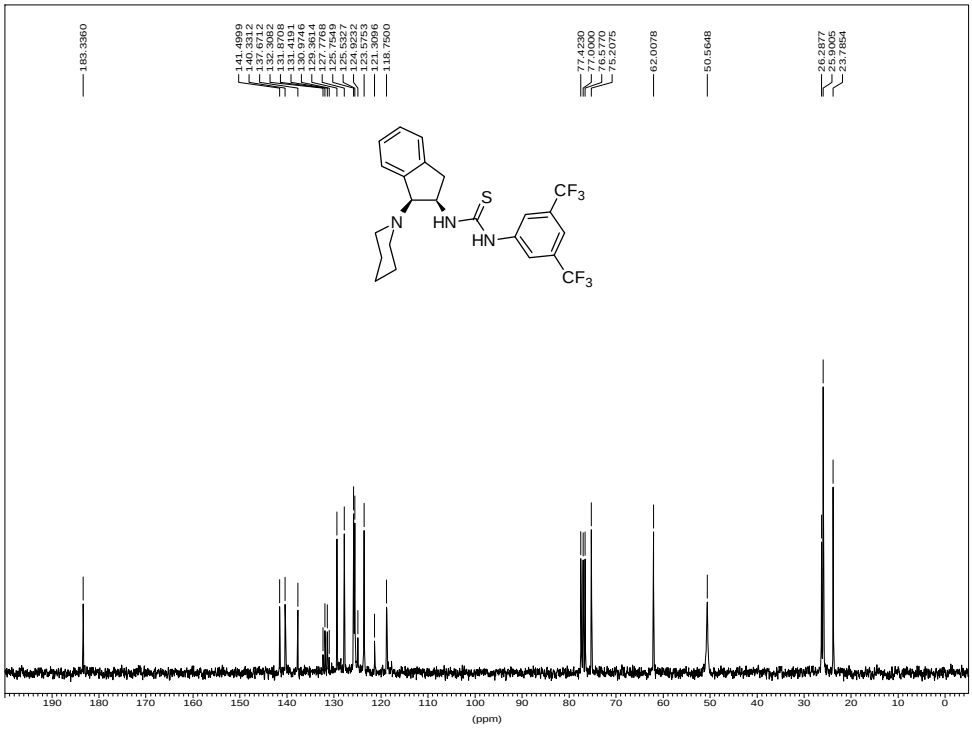
Compound **q**


NH2
N
Integral
0.9203
3.0438
1.0000
3.1755
4.0132
2.2472
4.2395
2.1405
(ppm)

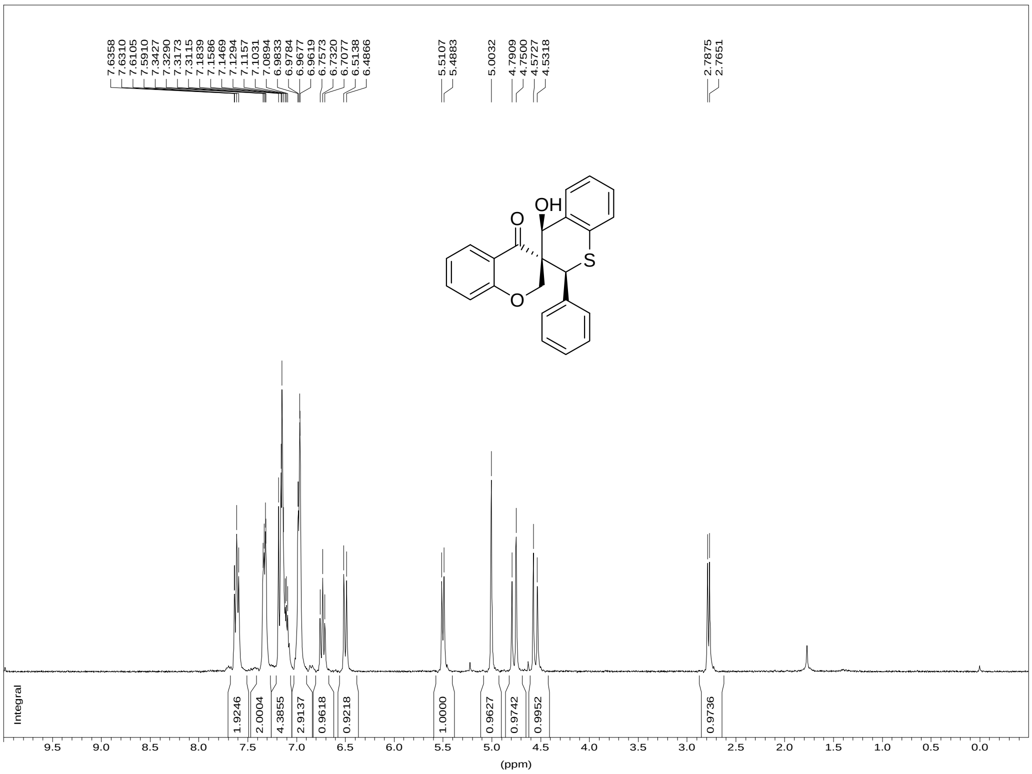

NH2
N
144.9183
139.7224
127.1808
126.3223
124.3636
123.3331
77.4518
77.2551
77.0000
76.7449
58.1621
51.8367
31.6726
25.8135
24.2832
(ppm)

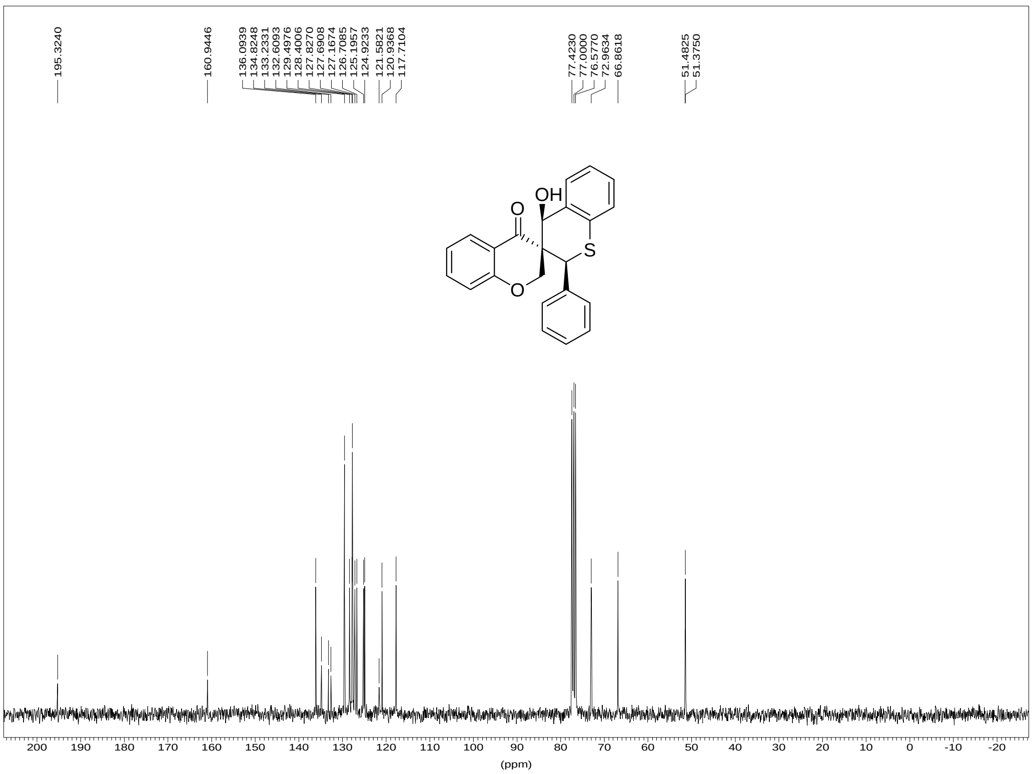
Compound **4**


12.1396
7.9772
7.6127
7.5569
7.3359
7.3250
7.2640
7.2518
7.2396
7.1957
7.1835
6.6824
5.2243
5.2072
3.6759
3.6478
3.6222
3.5942
3.1699
3.1497
3.1163
3.0882
2.9675
2.9371
2.9127
2.8834
2.6847
2.6652
2.6054
1.5594
1.4996
1.4460
S
HN
N
HN
CF3
CF3
Integral
0.8982
1.9652
1.0218
4.0370
0.8330
1.0000
0.9703
1.0182
1.1077
3.9061
6.0085
(ppm)

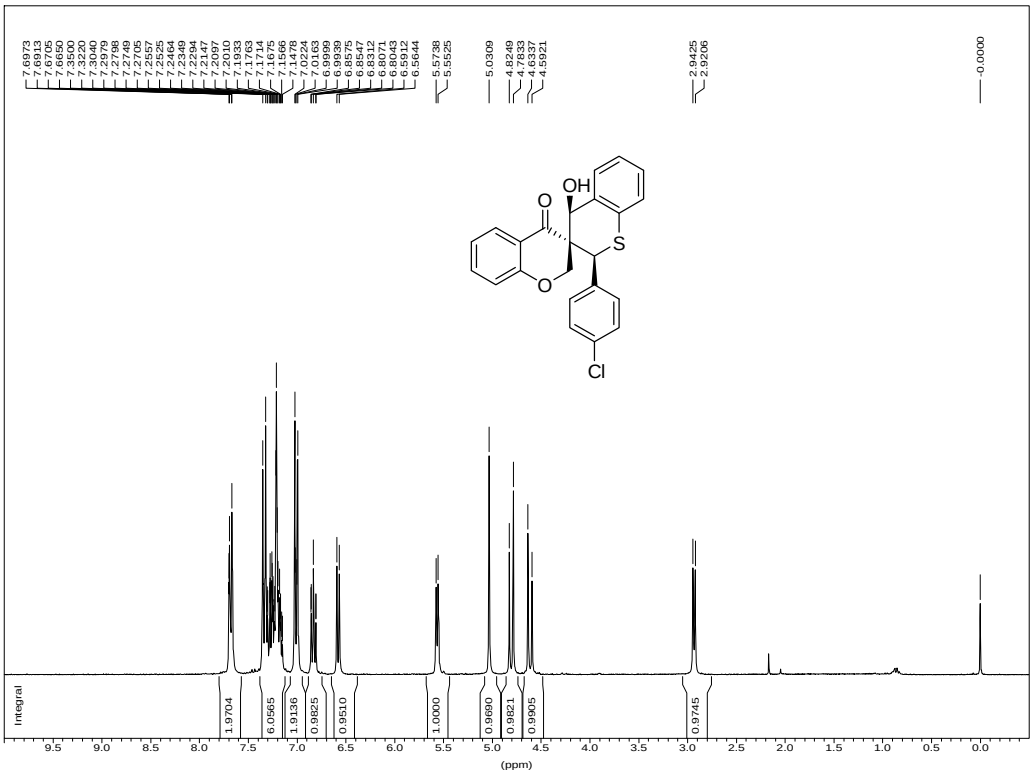

183.3360
141.4999
140.3312
137.6812
132.3062
131.8708
131.4191
130.0746
129.3614
127.7768
125.7549
125.5577
124.9232
123.5753
121.3096
118.7900
77.4230
77.0000
76.5770
75.2075
62.0078
50.5648
26.2877
25.9055
23.7854
S
HN
N
HN
CF3
CF3
(ppm)

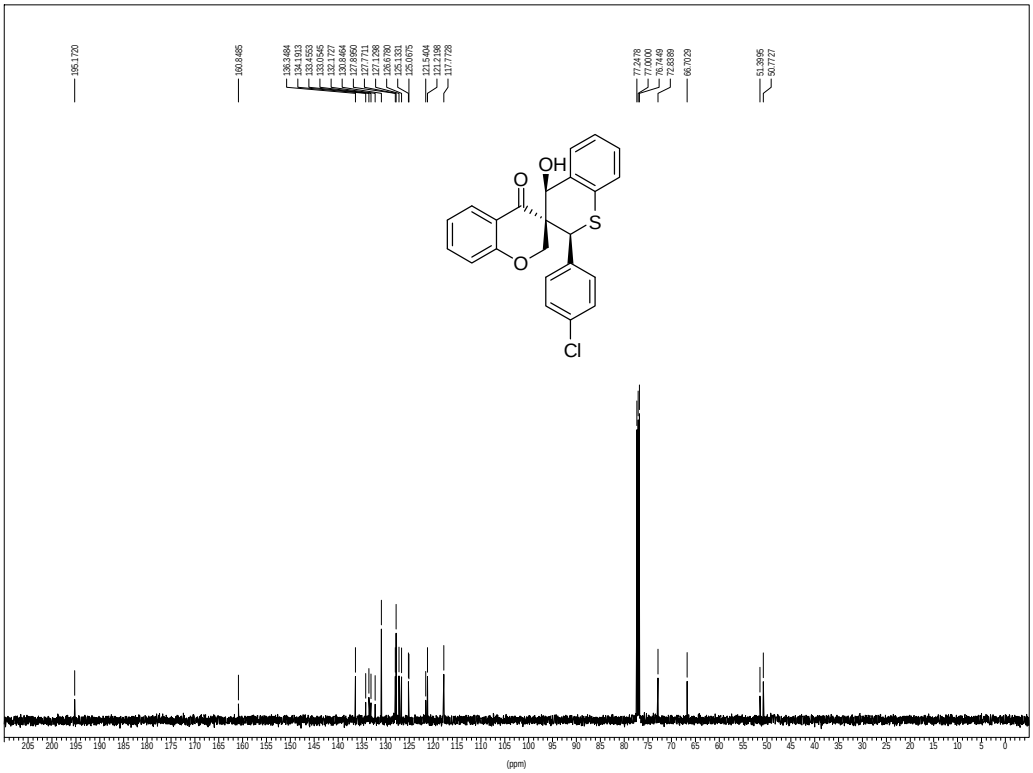
Compound **8a**


7.6358
7.6310
7.6105
7.5910
7.3427
7.3290
7.3173
7.3115
7.1839
7.1586
7.1469
7.1294
7.1157
7.1031
7.0894
6.9833
6.9784
6.9677
6.9619
6.7573
6.7320
6.7077
6.5138
6.4866
5.5107
5.4883
5.0032
4.7909
4.7500
4.5727
4.5318
2.7875
2.7651
OH
O
S
O
Integral
1.9246
2.0004
4.3855
2.9137
0.9618
0.9218
1.0000
0.9627
0.9742
0.9952
0.9736
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
(ppm)

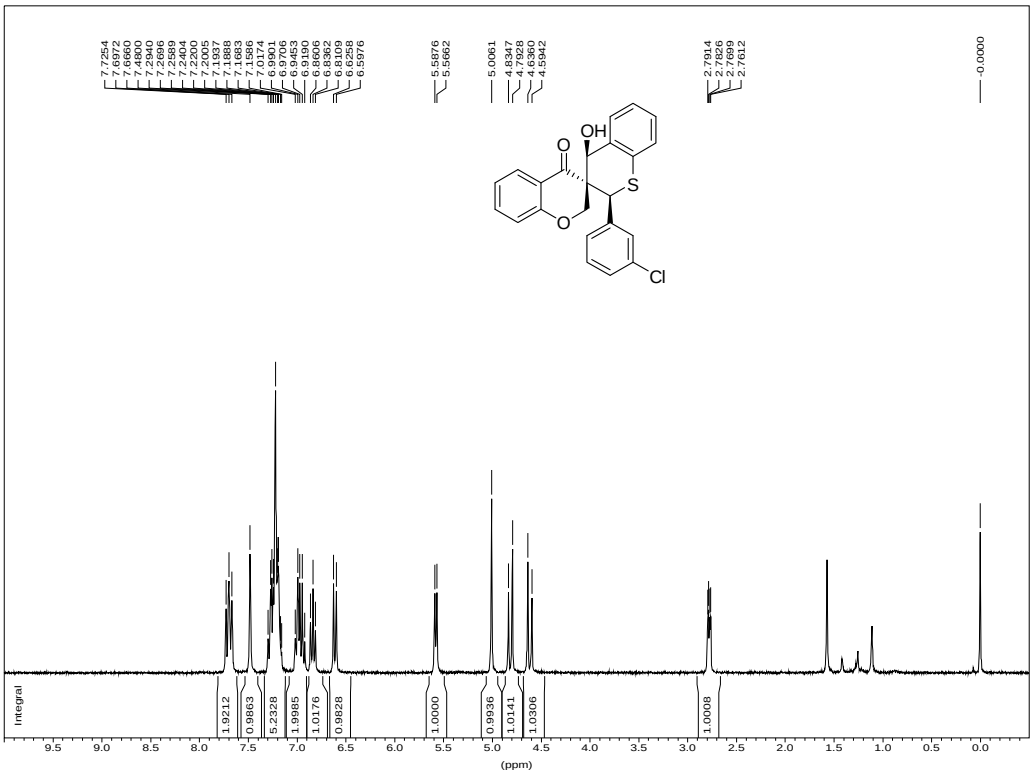

195.3240
160.9446
136.0939
134.8248
133.2331
132.6093
129.4976
128.4006
127.8270
127.6908
127.1674
126.7085
125.1957
124.9233
121.5821
120.9368
117.7104
77.4230
77.0000
76.5770
72.9634
66.8618
51.4825
51.3750
OH
O
S
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
(ppm)

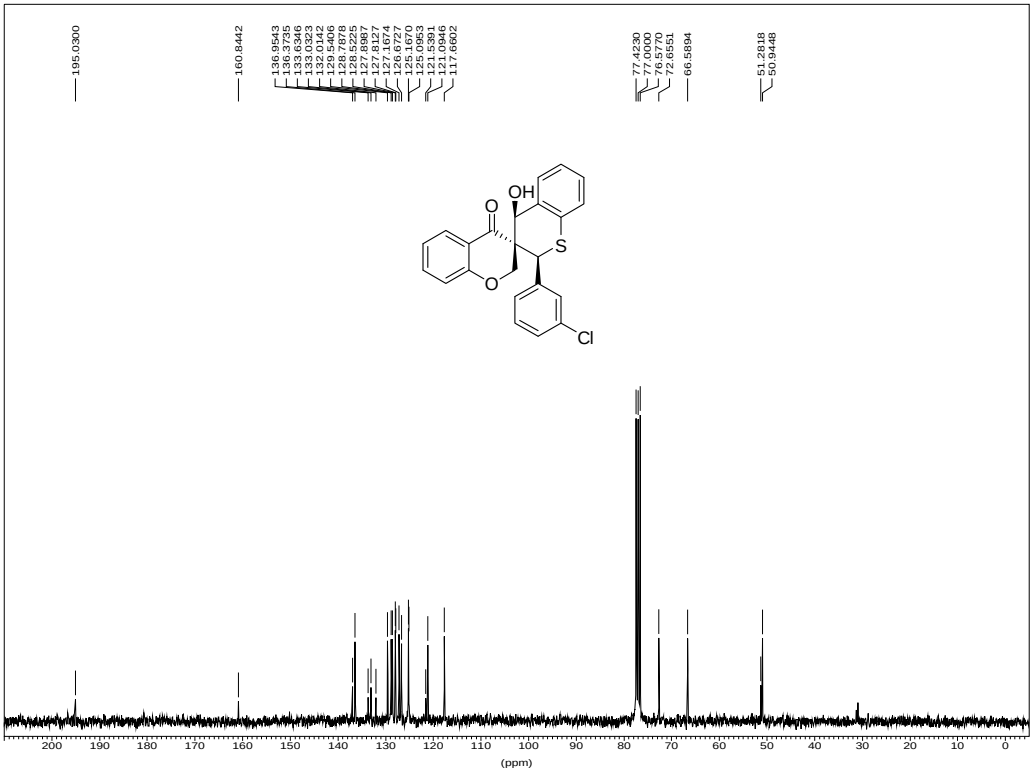
Compound **8b**


7.6973
7.6913
7.6705
7.6650
7.3560
7.3220
7.3040
7.2979
7.2798
7.2749
7.2705
7.2557
7.2525
7.2464
7.2349
7.2294
7.2147
7.2097
7.2010
7.1933
7.1763
7.1714
7.1675
7.1566
7.1478
7.0224
7.0163
6.9999
6.9938
6.8575
6.8547
6.8312
6.8071
6.8045
6.5912
6.5644
5.5738
5.5525
5.0309
4.8249
4.6253
4.6337
4.5921
2.9425
2.9206
-0.0000
O
OH
S
O
Cl
Integral
1.9704
6.0565
1.9136
0.9825
0.9510
1.0000
0.9690
0.9821
0.9905
0.9745
(ppm)

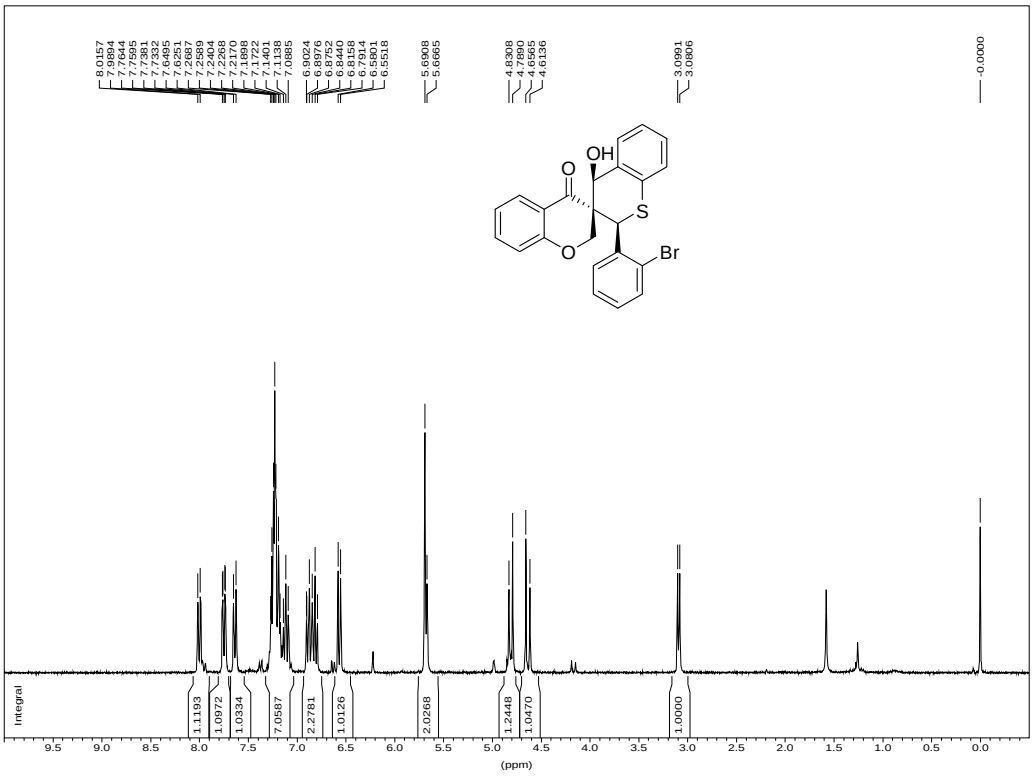

195.1720
160.8465
136.3484
134.1913
134.1455
133.0545
132.1727
130.8464
129.0900
127.7711
127.1288
126.6780
125.1331
125.0675
121.5404
121.2398
117.7769
77.2478
77.0000
76.7446
72.8339
66.7029
51.3905
50.7727
O
OH
S
O
Cl
(ppm)

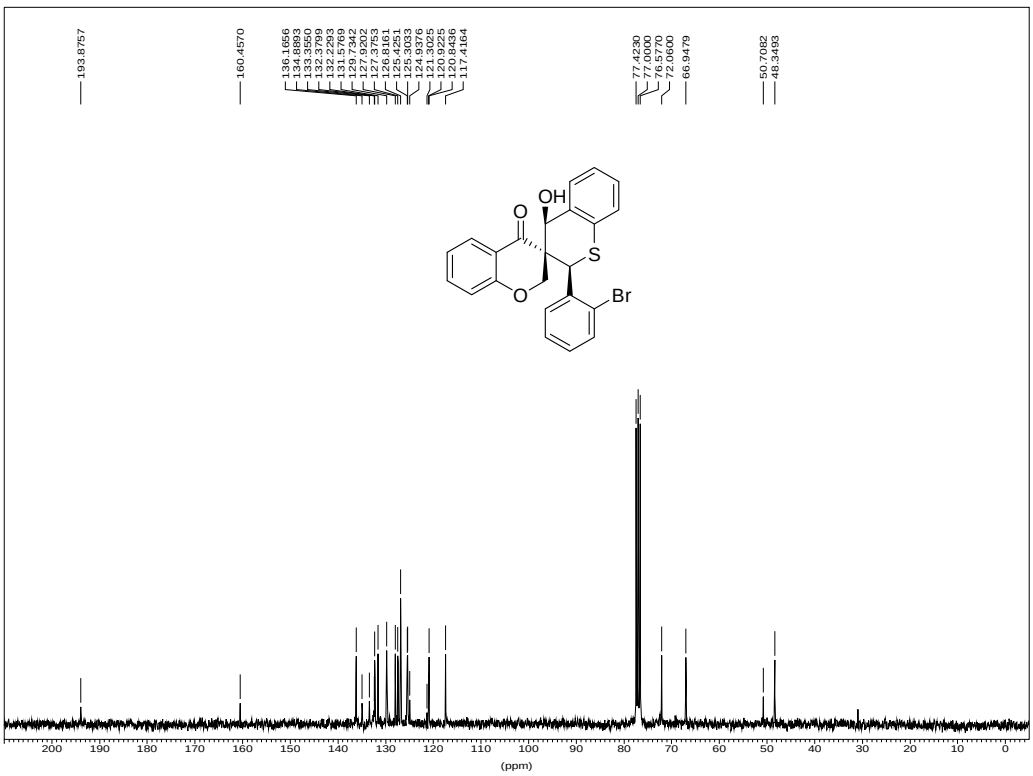
Compound **8**c


7.7254
7.6972
7.6660
7.4800
7.2940
7.2696
7.2369
7.2044
7.2200
7.2005
7.1937
7.1888
7.1683
7.1586
7.0174
6.9901
6.9705
6.9463
6.9190
6.8606
6.8362
6.8150
6.6258
6.5976
5.5876
5.5662
5.0061
4.8347
4.7098
4.6360
4.5942
2.7914
2.7826
2.7699
2.7612
-0.0000
OH
O
S
O
Cl
Integral
1.9212
0.9863
5.2328
1.9985
1.0176
0.9828
1.0000
0.9936
1.0141
1.0306
1.0008
(ppm)

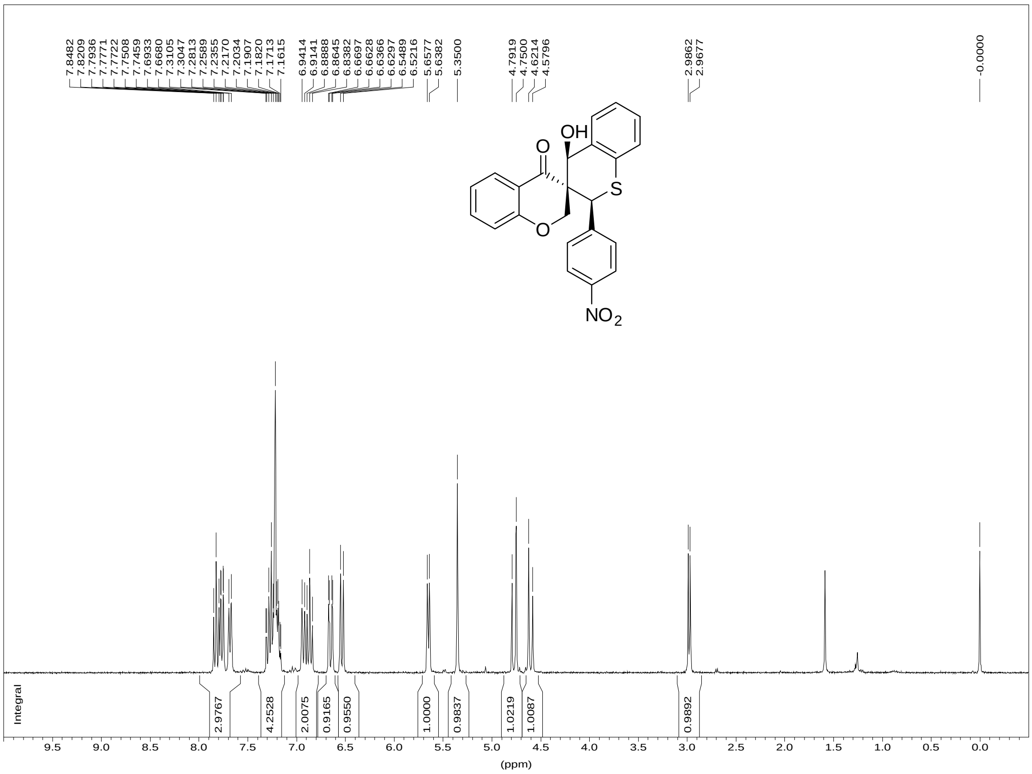

195.0300
160.8442
136.9543
136.3735
133.9346
133.0323
132.0142
129.5406
128.7878
128.7255
127.8987
127.8127
127.1674
126.6727
125.6570
125.0953
121.5391
121.0946
117.6602
77.4230
77.0000
76.5770
72.6551
66.5894
51.2818
50.9448
OH
O
S
O
Cl
(ppm)

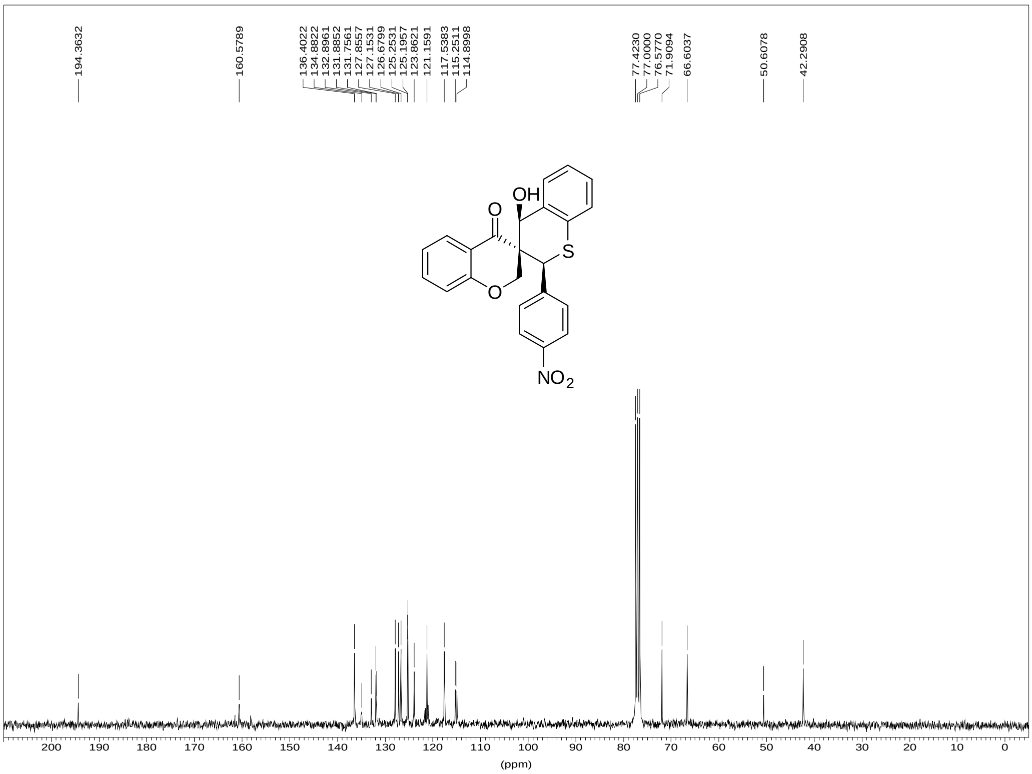
Compound **8d**


8.0157
7.9894
7.7644
7.7595
7.7381
7.7232
7.6495
7.6251
7.2687
7.2469
7.2404
7.2268
7.2170
7.1898
7.1722
7.1401
7.1138
7.0885
6.9024
6.8976
6.8752
6.8440
6.8158
6.7914
6.5801
6.5518
5.6908
5.6665
4.8308
4.7990
4.6565
4.6136
3.0991
3.0806
-0.0000
OH
O
S
O
Br
Integral
1.1193
1.0972
1.0334
7.0587
2.2781
1.0126
2.0268
1.2448
1.0470
1.0000
(ppm)


193.8757
160.4570
136.1666
134.9983
133.3550
132.3799
132.2293
131.5769
129.7412
127.9202
127.3753
126.8161
125.4251
125.1053
124.9376
121.3025
120.9225
120.8436
117.4164
77.4230
77.0000
76.5770
72.0600
66.9479
50.7082
48.3493
OH
O
S
O
Br
(ppm)

Compound **8e**


7.8482
7.8209
7.7936
7.7771
7.7722
7.7508
7.7459
7.6933
7.6680
7.3105
7.3047
7.2813
7.2589
7.2355
7.2170
7.2034
7.1907
7.1820
7.1713
7.1615
6.9414
6.9141
6.8888
6.8645
6.8382
6.6697
6.6628
6.6366
6.6297
6.5489
6.5216
5.6577
5.6382
5.3500
4.7919
4.7500
4.6214
4.5796
2.9862
2.9677
-0.0000
OH
O
S
O
NO2
Integral
2.9767
4.2528
2.0075
0.9165
0.9550
1.0000
0.9837
1.0219
1.0087
0.9892
(ppm)


194.3632
160.5789
136.4022
134.8822
132.8961
131.8852
131.7561
127.8557
127.1531
126.6799
125.2531
125.1957
123.8621
121.1591
117.5383
115.2511
114.8998
77.4230
77.0000
76.5770
71.9094
66.6037
50.6078
42.2908
OH
O
S
O
NO2
(ppm)

Supplementary Material (ESI) for Chemical Communications
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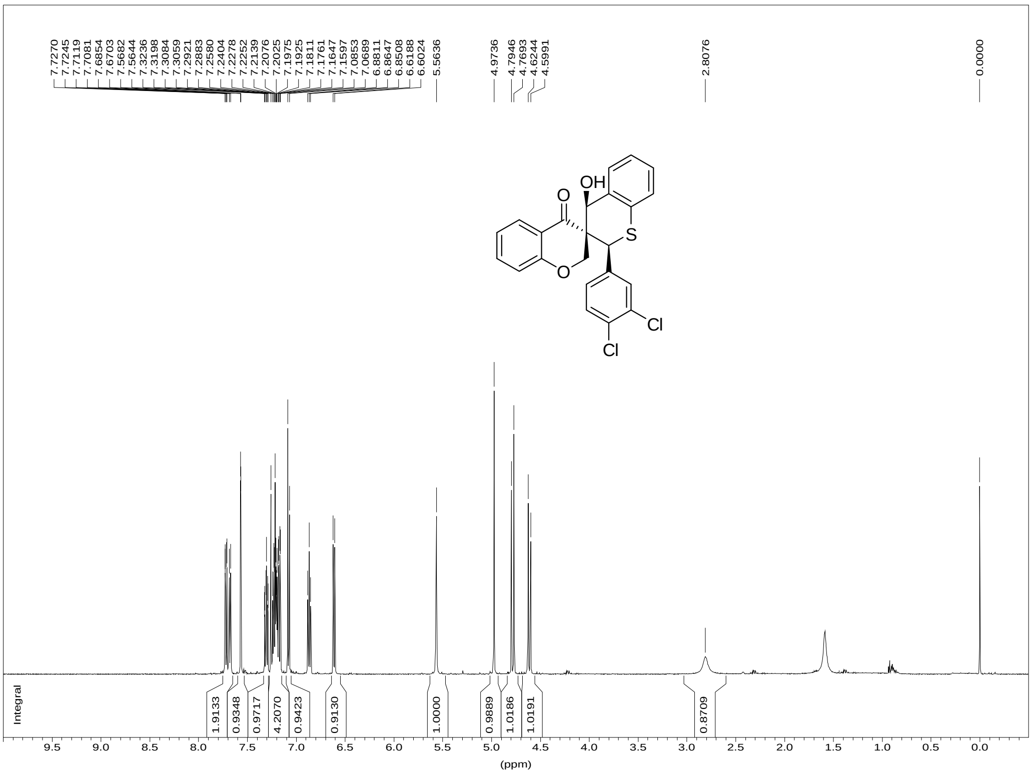
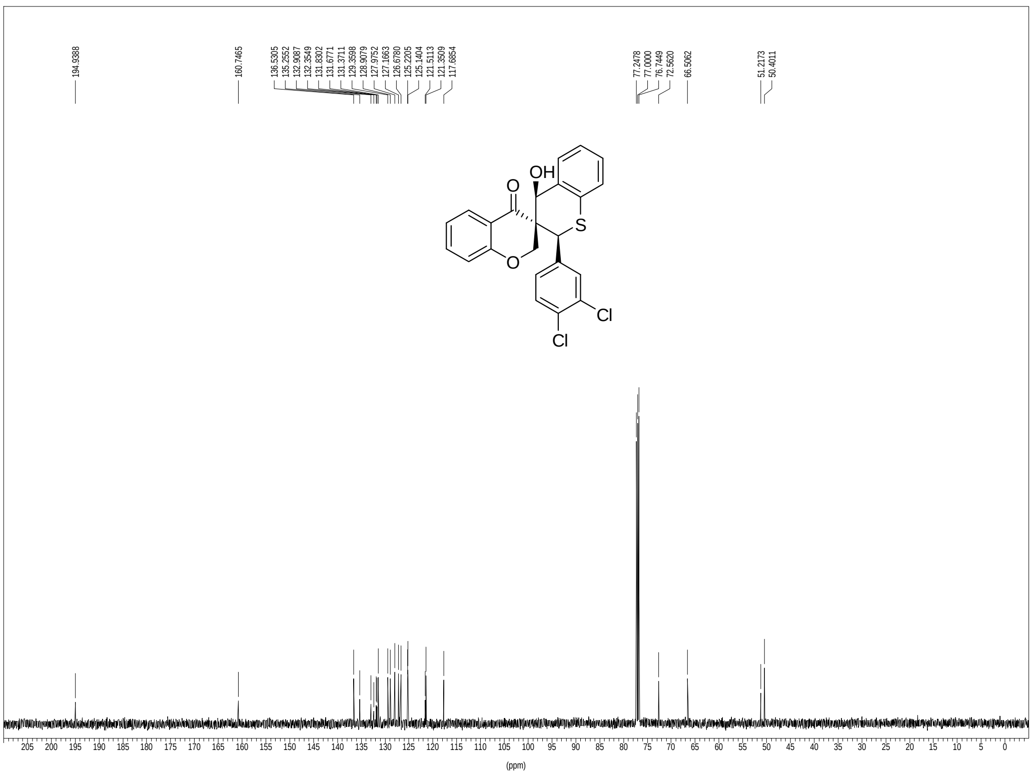
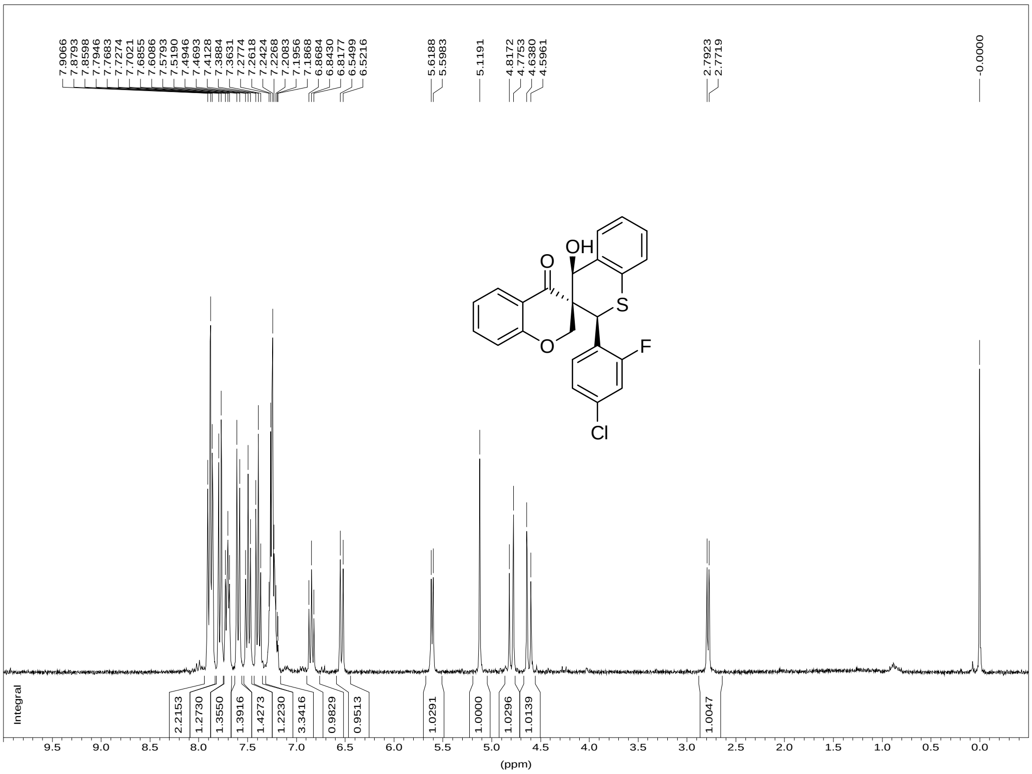
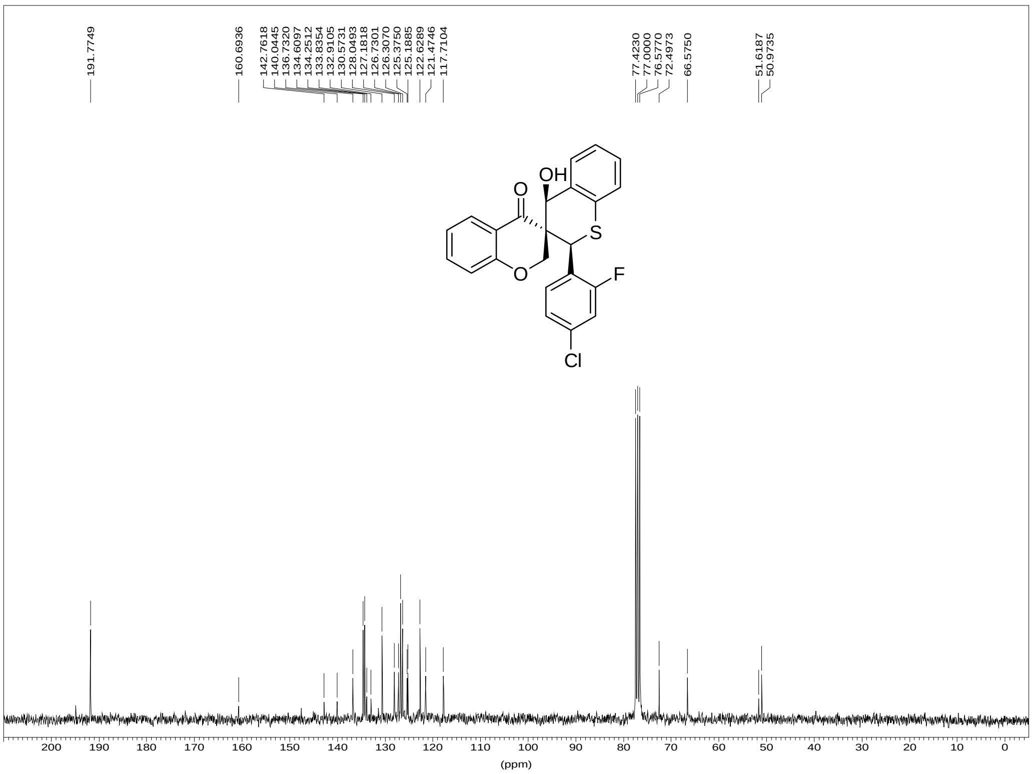
Compound **8f**



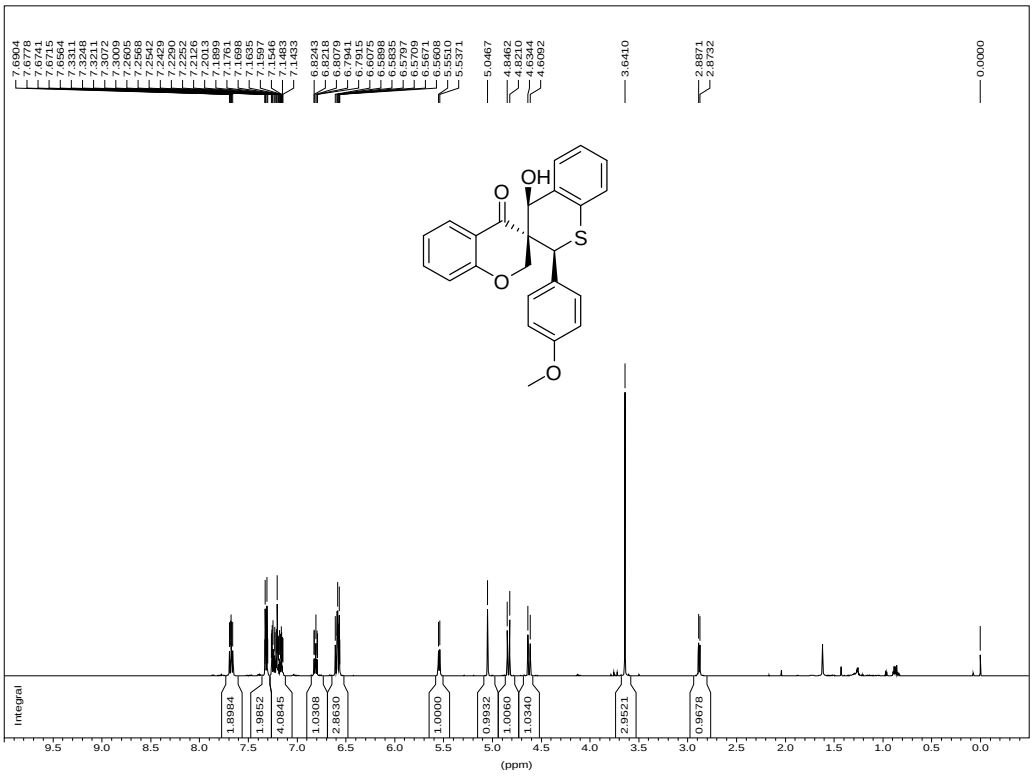


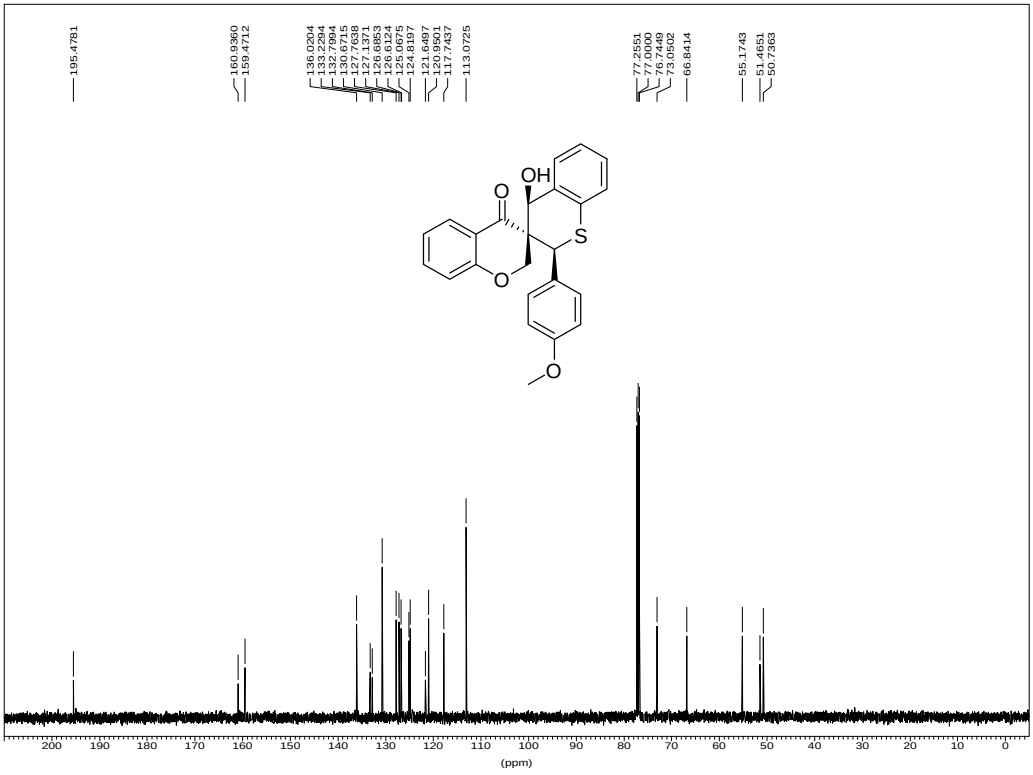
Compound **8g**


7.9066
7.8793
7.8598
7.7946
7.7683
7.7274
7.7021
7.6855
7.6086
7.5793
7.5190
7.4946
7.4693
7.4128
7.3884
7.3631
7.2774
7.2618
7.2424
7.2268
7.2083
7.1956
7.1868
6.8684
6.8430
6.8177
6.5499
6.5216
5.6188
5.5983
5.1191
4.8172
4.7753
4.6380
4.5961
2.7923
2.7719
-0.0000
OH
O
S
O
F
Cl
Integral
2.2153
1.2730
1.3550
1.3916
1.4273
1.2230
3.3416
0.9829
0.9513
1.0291
1.0000
1.0296
1.0139
1.0047
(ppm)

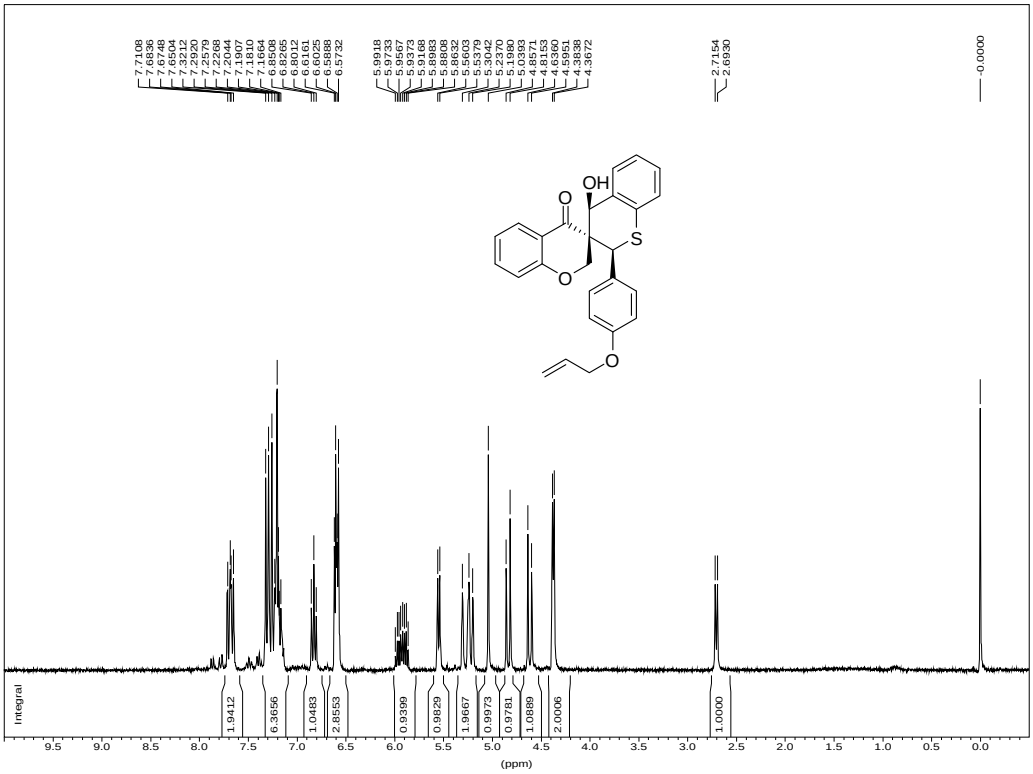

191.7749
160.6936
142.7618
140.0445
136.7320
134.6097
134.2512
133.8354
132.9105
130.5731
128.0493
127.1818
126.7301
126.3070
125.3750
125.1885
122.6289
121.4746
117.7104
77.4230
77.0000
76.5770
72.4973
66.5750
51.6187
50.9735
OH
O
S
O
F
Cl
(ppm)

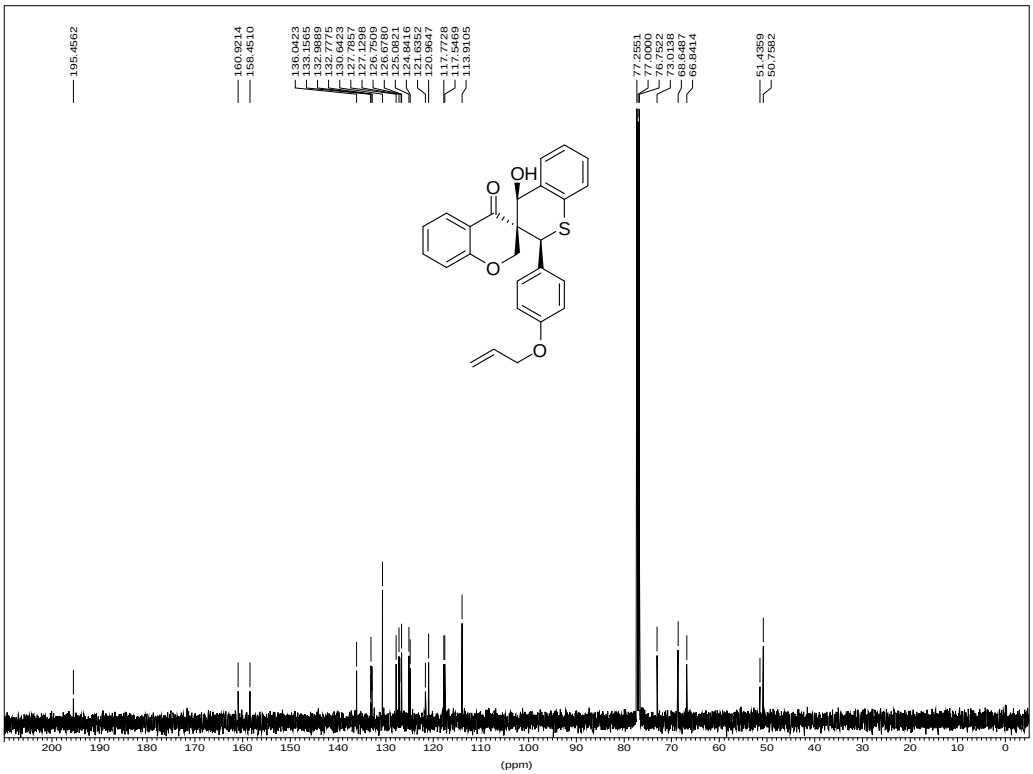
Compound **8h**


7.6904
7.6778
7.6741
7.6715
7.6564
7.6311
7.3248
7.3211
7.3072
7.3009
7.2955
7.2568
7.2542
7.2429
7.2290
7.2257
7.2126
7.2013
7.1899
7.1781
7.1698
7.1635
7.1597
7.1546
7.1493
7.1433
6.8243
6.8218
6.8070
6.7941
6.7915
6.6075
6.5898
6.5835
6.5797
6.5709
6.5671
6.5600
5.5510
5.5371
5.0467
4.8462
4.8210
4.6344
4.6092
3.6410
2.8871
2.8732
0.0000
OH
O
S
O
O
Integral
1.8984
1.9852
4.0845
1.0308
2.8630
1.0000
0.9932
1.0060
1.0340
2.9521
0.9678
(ppm)

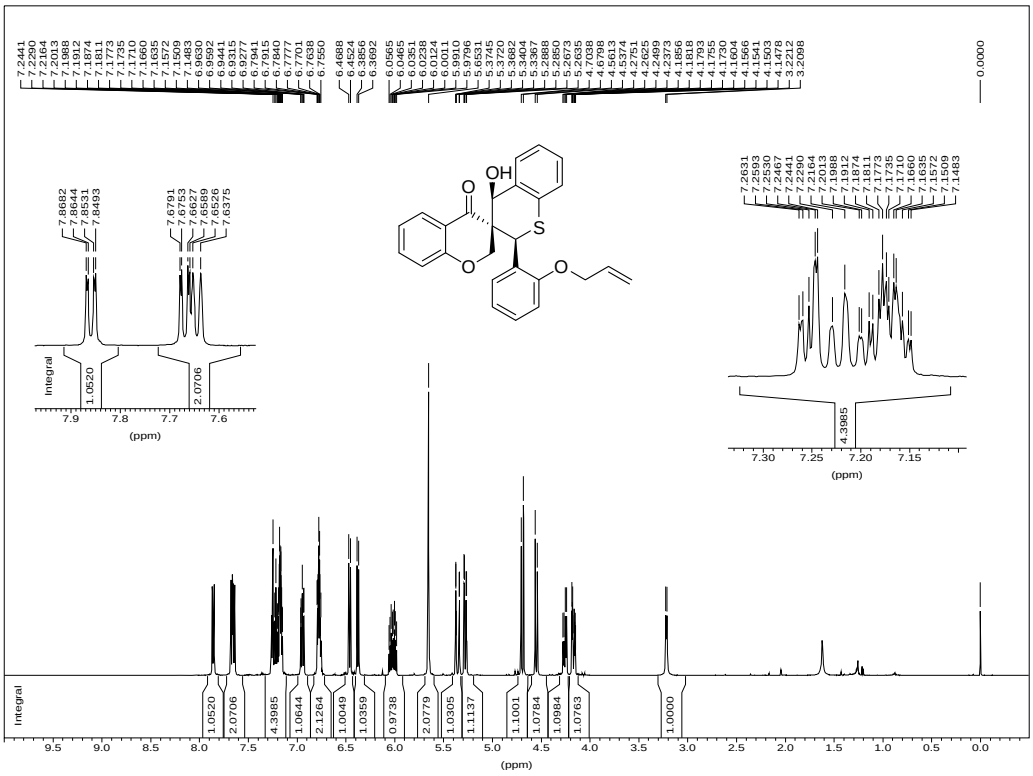

195.4781
160.9360
159.4712
136.0204
133.2294
132.7994
130.6715
127.7638
127.2331
126.6853
126.6124
125.0675
124.8197
121.6497
120.9651
117.7437
113.0725
77.2551
77.0000
76.7449
73.0502
66.8414
55.1743
51.4651
50.7583
OH
O
S
O
O
(ppm)

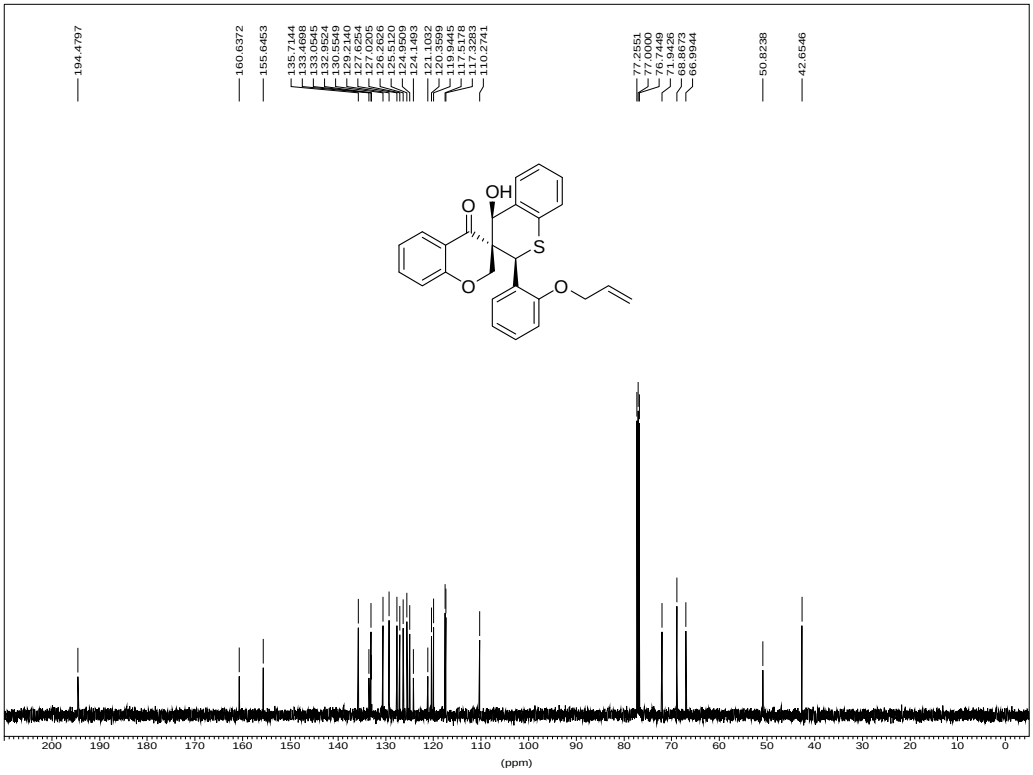
Compound **8i**


7.7108
7.6836
7.6748
7.6504
7.3212
7.2920
7.2579
7.2068
7.2044
7.1907
7.1810
7.1664
6.8558
6.8266
6.8012
6.6161
6.6025
6.5888
6.5732
5.9918
5.9733
5.9567
5.9373
5.9168
5.8983
5.8808
5.8652
5.5603
5.5379
5.3042
5.2370
5.1980
5.0393
4.8571
4.8153
4.6360
4.5951
4.3838
4.3672
2.7154
2.6930
0.0000
OH
O
S
O
O
Integral
1.9412
6.3656
1.0483
2.8553
0.9399
0.9829
1.9667
0.9973
0.9781
1.0889
2.0006
1.0000
(ppm)

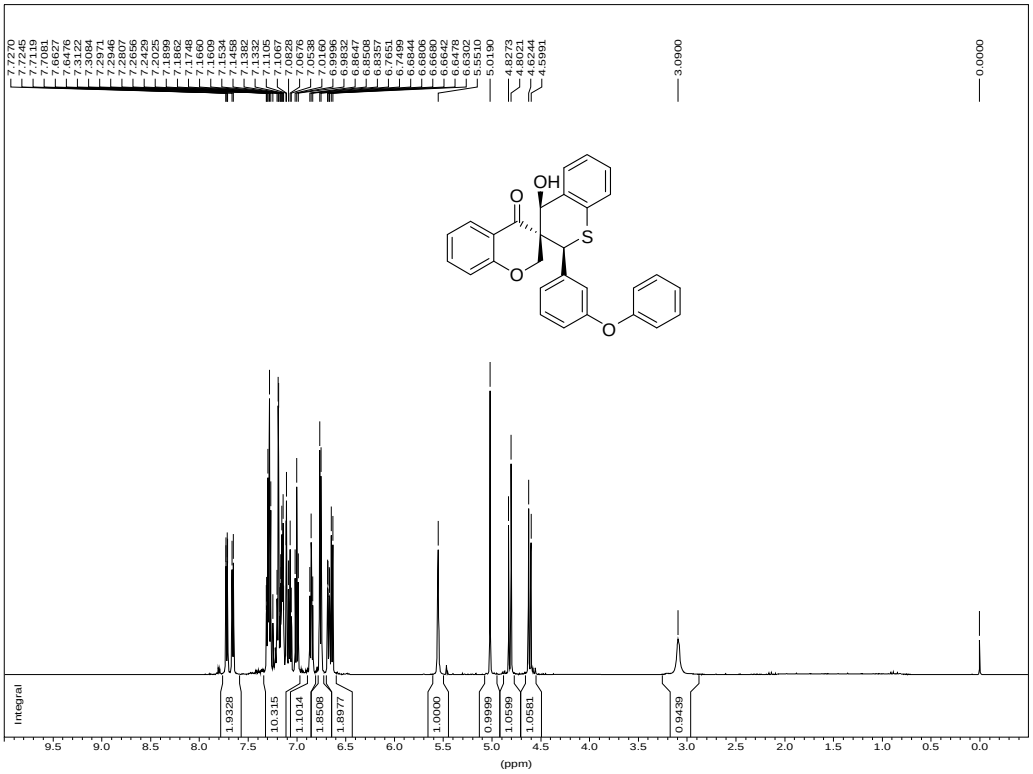

195.4562
160.9214
158.4510
136.0423
133.1565
132.9889
132.7775
130.7423
130.2457
127.1298
126.7509
126.6780
125.8921
124.9415
121.6352
120.9647
117.7728
117.5469
113.9105
77.2551
77.0000
76.7322
73.0138
68.6487
66.8414
51.4359
50.7582
OH
O
S
O
O
(ppm)

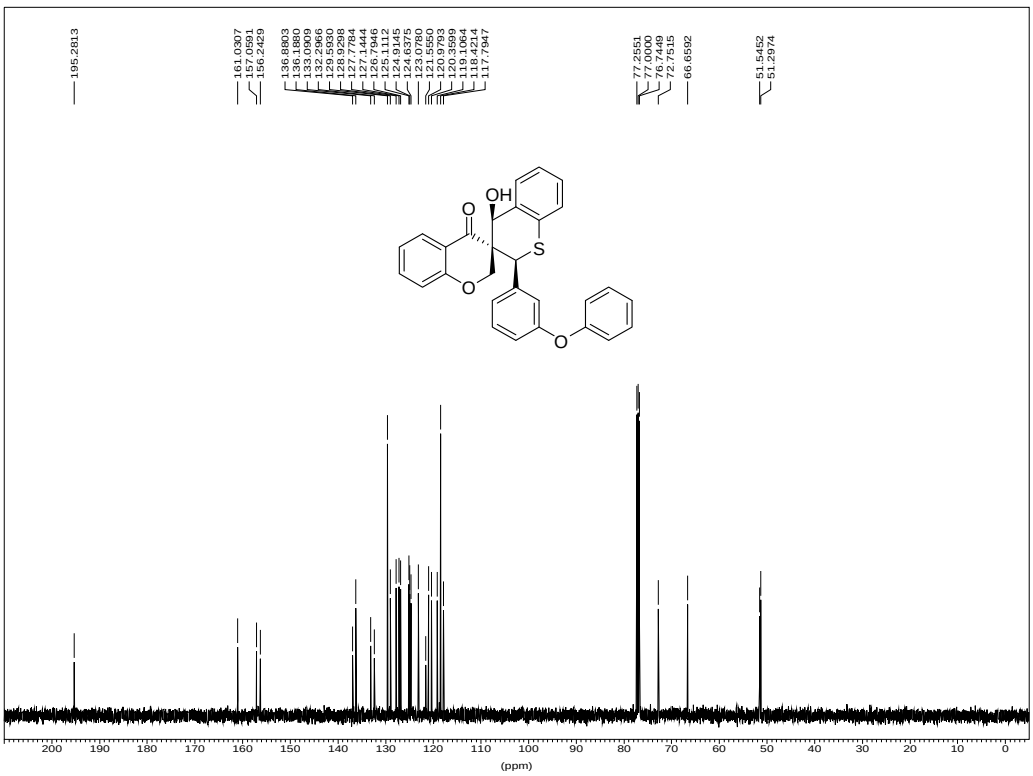
Compound **8j**



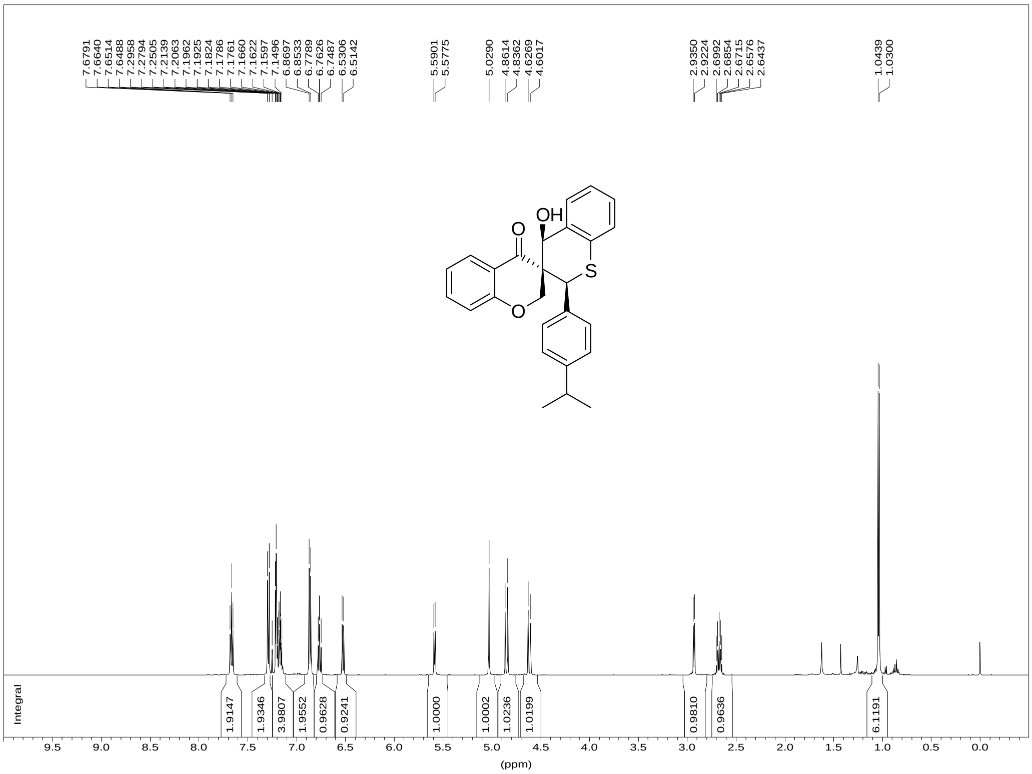


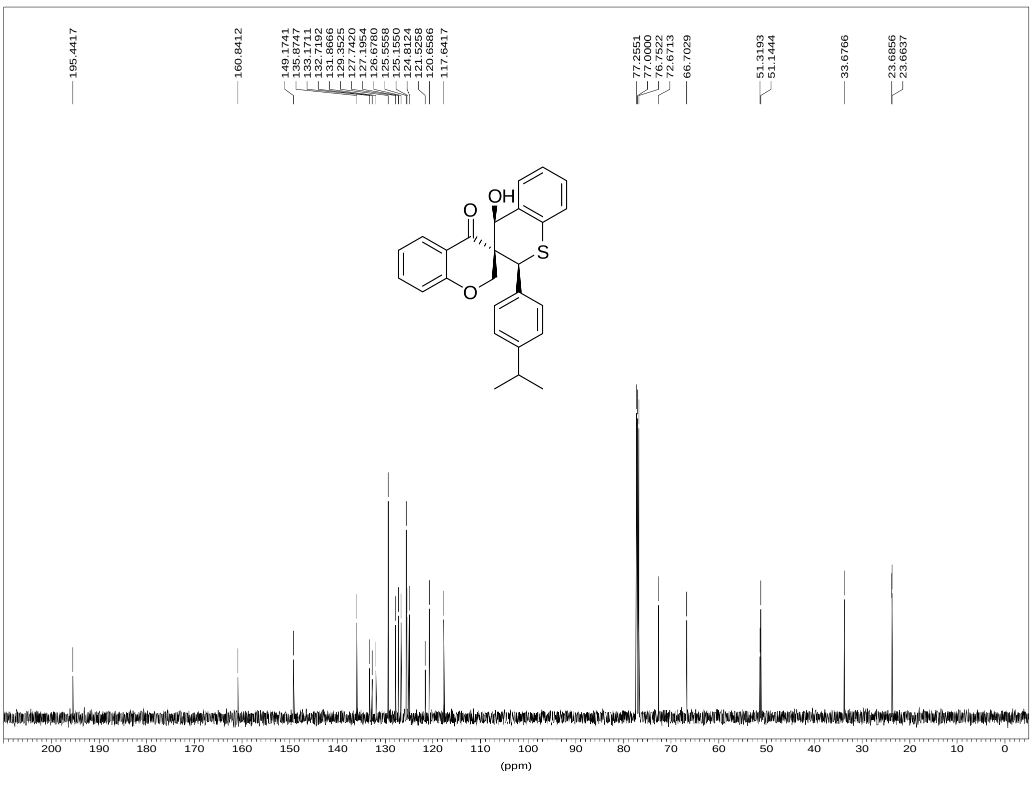
Compound **8k**


7.7270
7.7245
7.7115
7.7081
7.6627
7.6476
7.3312
7.3084
7.2971
7.2946
7.2807
7.2566
7.2425
7.2025
7.1899
7.1962
7.1748
7.1669
7.1609
7.1534
7.1458
7.1332
7.1105
7.1067
7.0828
7.0766
7.0538
7.0160
6.9996
6.9832
6.8647
6.8508
6.8357
6.7651
6.7499
6.6844
6.6806
6.6680
6.6642
6.6478
6.6301
5.5510
5.0190
4.8273
4.8021
4.6244
4.5991
3.0900
0.0000
OH
O
S
O
O
Integral
1.9328
10.315
1.1014
1.8508
1.8977
1.0000
0.9999
1.0599
1.0581
0.9439
(ppm)

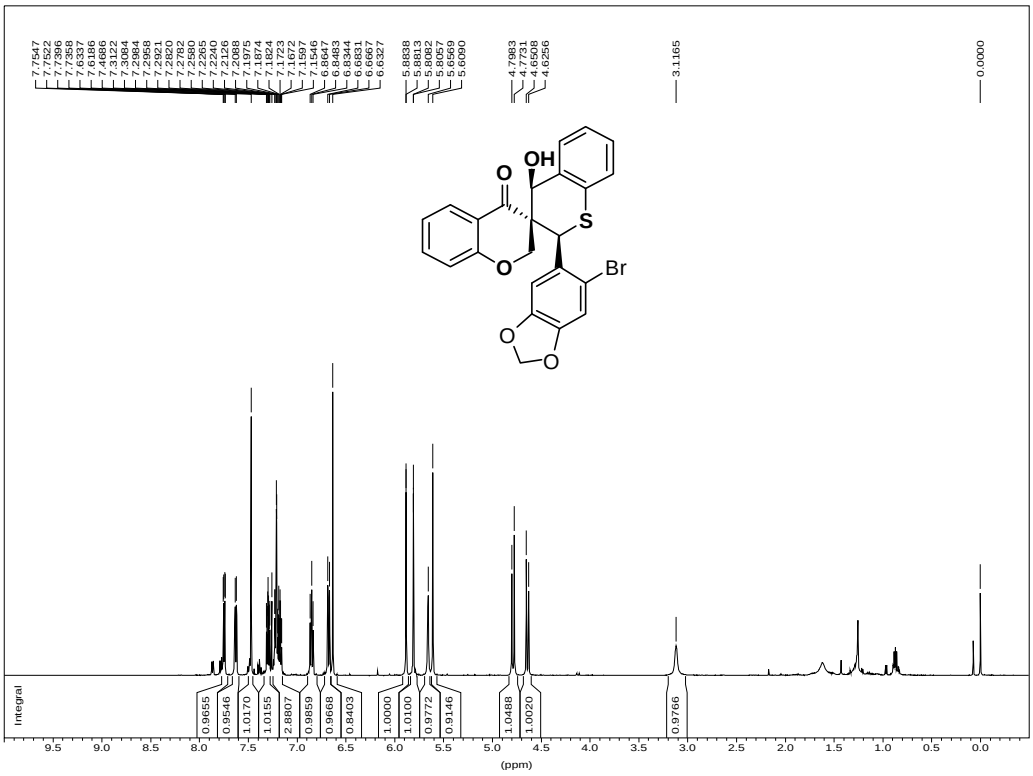

195.2813
161.0307
157.0591
156.2429
136.8803
136.3980
133.0909
132.2966
129.5930
128.9298
128.7704
127.1444
126.7946
125.1112
124.8145
124.4555
123.0780
121.5550
120.9793
120.3699
119.1564
118.4214
117.7947
77.2551
77.0000
76.7449
72.7515
66.6592
51.5452
51.2974
OH
O
S
O
O
(ppm)

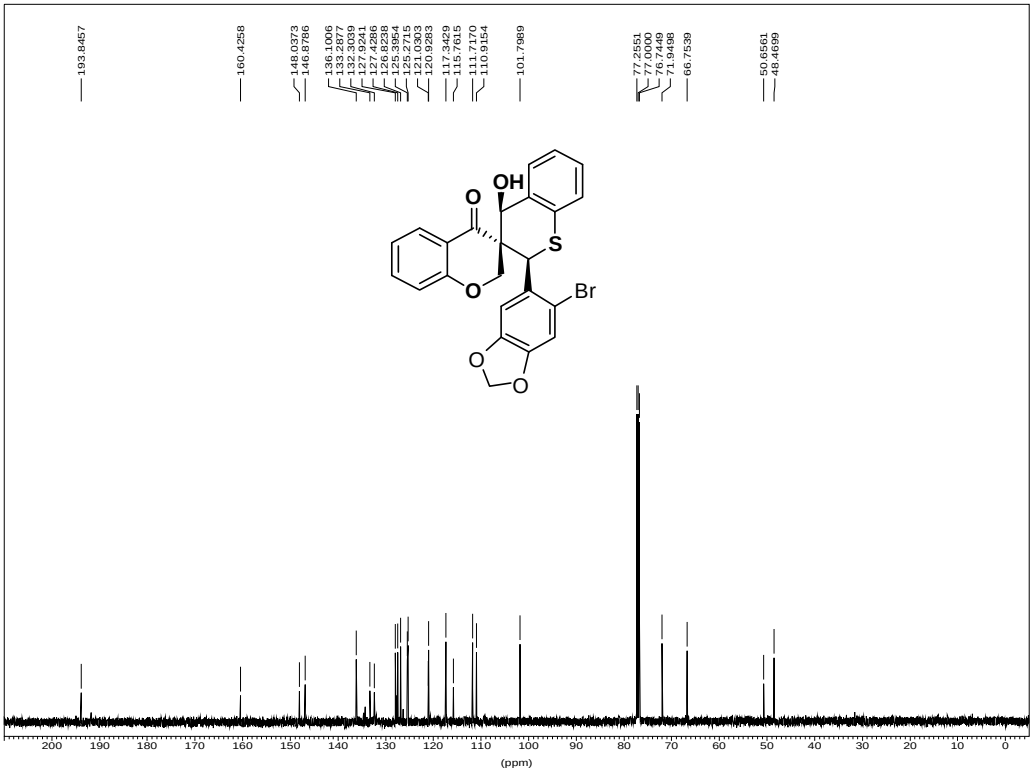
Compound **8l**



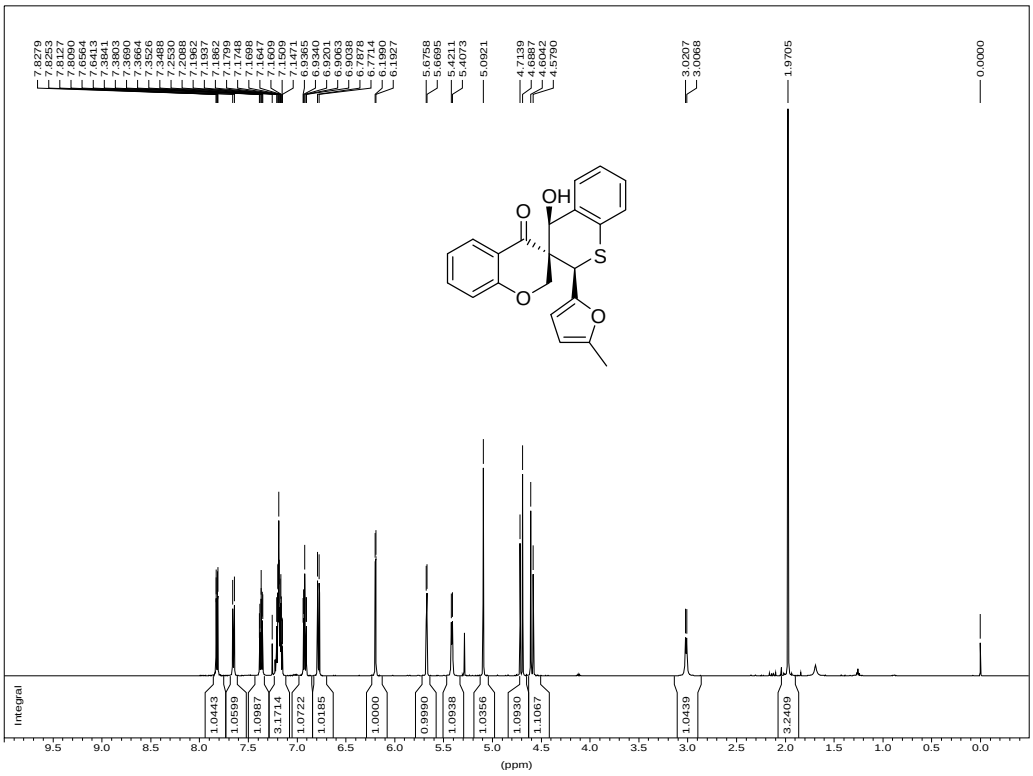


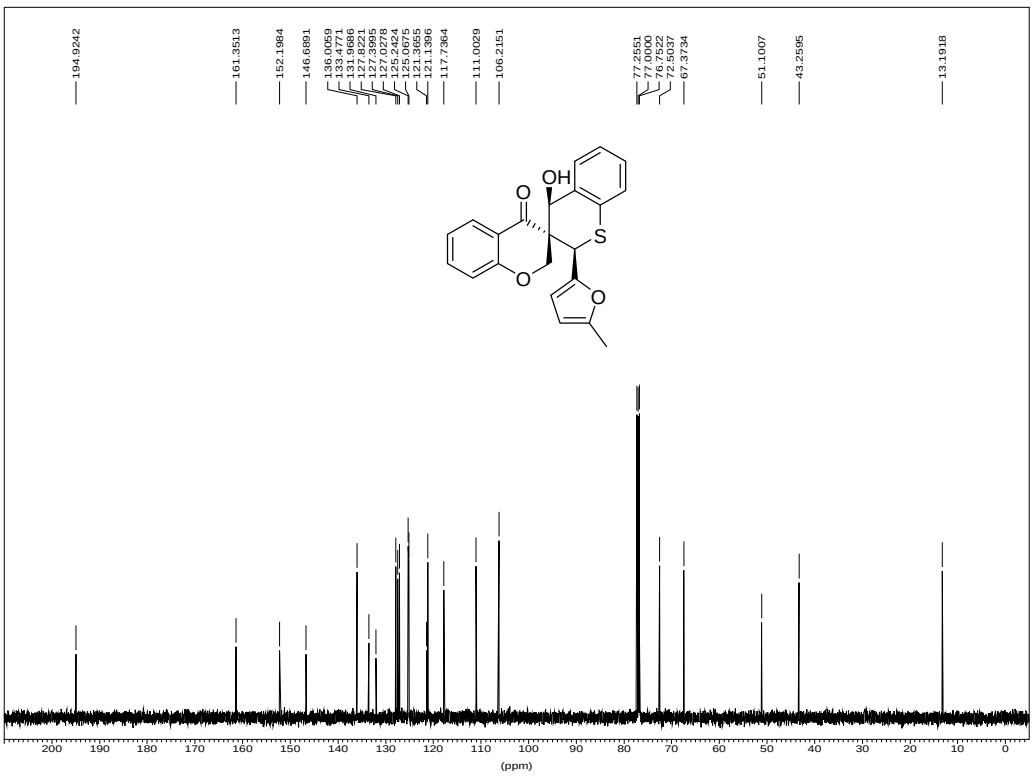
Compound **8m**


7.7547
7.7522
7.7386
7.7358
7.6337
7.6186
7.4686
7.4322
7.3084
7.2984
7.2958
7.2921
7.2800
7.2782
7.2580
7.2265
7.2210
7.2126
7.2088
7.1975
7.1874
7.1824
7.1723
7.1672
7.1597
7.1546
6.8647
6.8483
6.8344
6.6831
6.6667
6.6627
5.8838
5.8813
5.8082
5.8057
5.6690
5.6090
4.7983
4.7731
4.6598
4.6256
3.1165
0.0000
OH
O
S
O
Br
O
O
Integral
0.9655
0.9546
1.0170
1.0155
2.8807
0.9859
0.9668
0.8403
1.0000
1.0100
0.9772
0.9146
1.0488
1.0020
0.9766
(ppm)

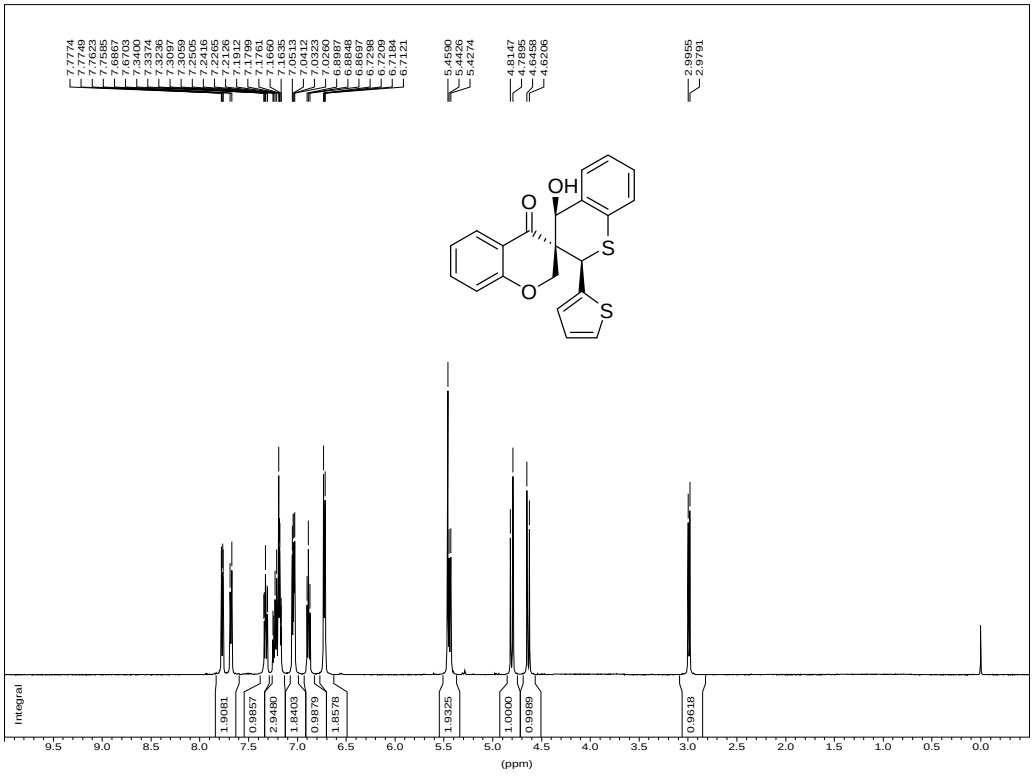

193.8457
160.4258
148.0373
146.8786
136.1006
133.2377
132.3809
127.9241
127.4286
126.8238
126.2864
125.2715
121.0303
120.9283
117.3429
115.7615
111.7170
110.9154
101.7989
77.2551
77.0000
76.7449
71.9498
66.7539
50.6561
48.4699
OH
O
S
O
Br
O
O
(ppm)

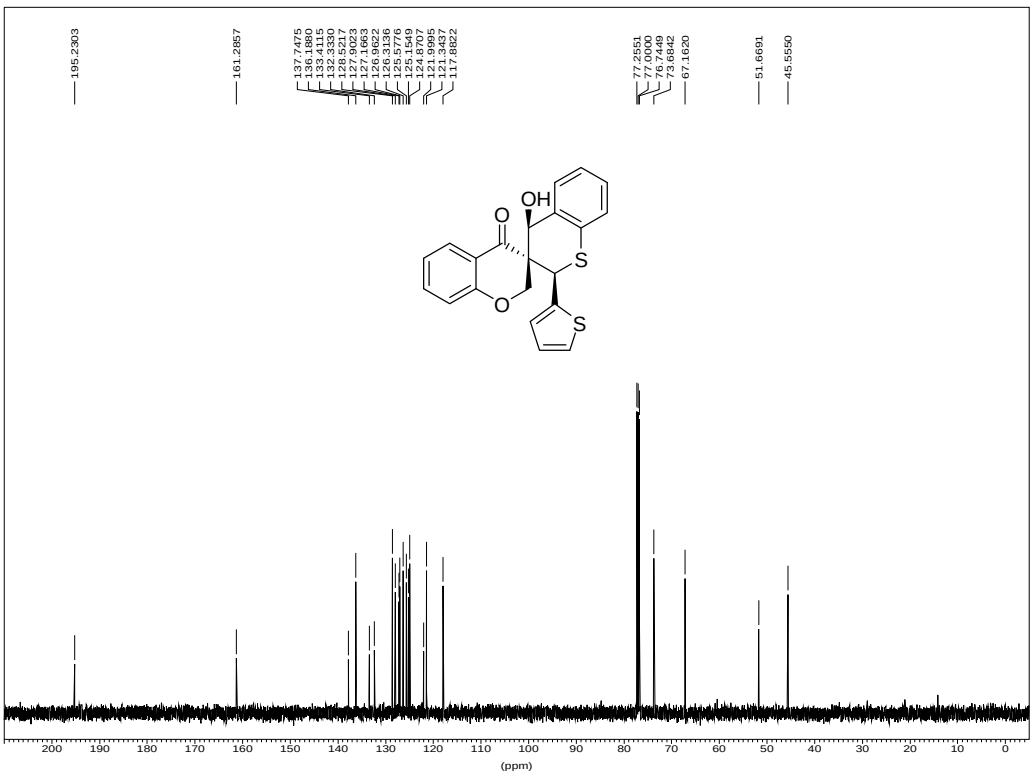
Compound **8n**


7.8279
7.8253
7.8127
7.6690
7.6564
7.6413
7.3941
7.3803
7.3680
7.3664
7.3526
7.3488
7.2650
7.2088
7.1962
7.1937
7.1862
7.1799
7.1748
7.1698
7.1647
7.1609
7.1569
7.1471
6.9365
6.9340
6.9201
6.9065
6.9038
6.7878
6.7714
6.1980
6.1927
5.6758
5.6695
5.4711
5.4073
5.0921
4.7139
4.6887
4.6042
4.5790
3.0207
3.0068
1.9705
0.0000
OH
O
S
O
O
Integral
1.0443
1.0599
1.0987
3.1714
1.0722
1.0185
1.0000
0.9990
1.0938
1.0356
1.0930
1.1067
1.0439
3.2409
(ppm)

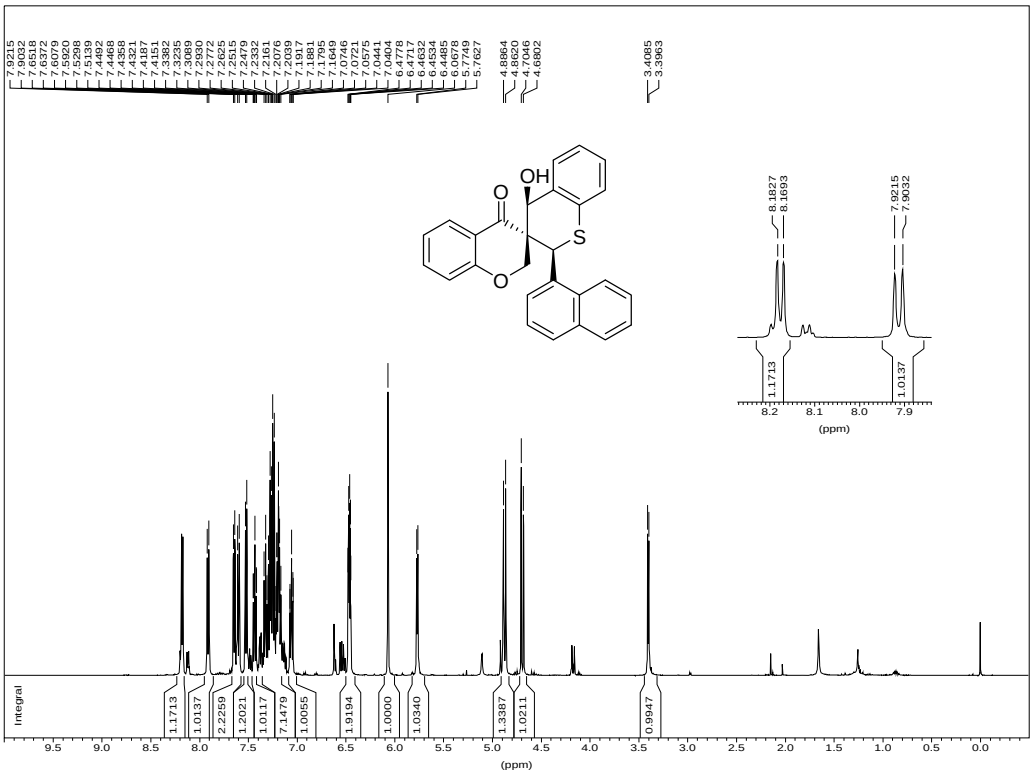

194.9242
161.3513
152.1984
146.6891
136.0059
133.4771
131.9666
127.8221
127.3995
127.0278
126.7454
125.0675
121.3655
121.1396
117.7364
111.0029
106.2151
77.2551
77.0000
76.7422
72.5037
67.3734
51.1007
43.2595
13.1918
OH
O
S
O
O
(ppm)

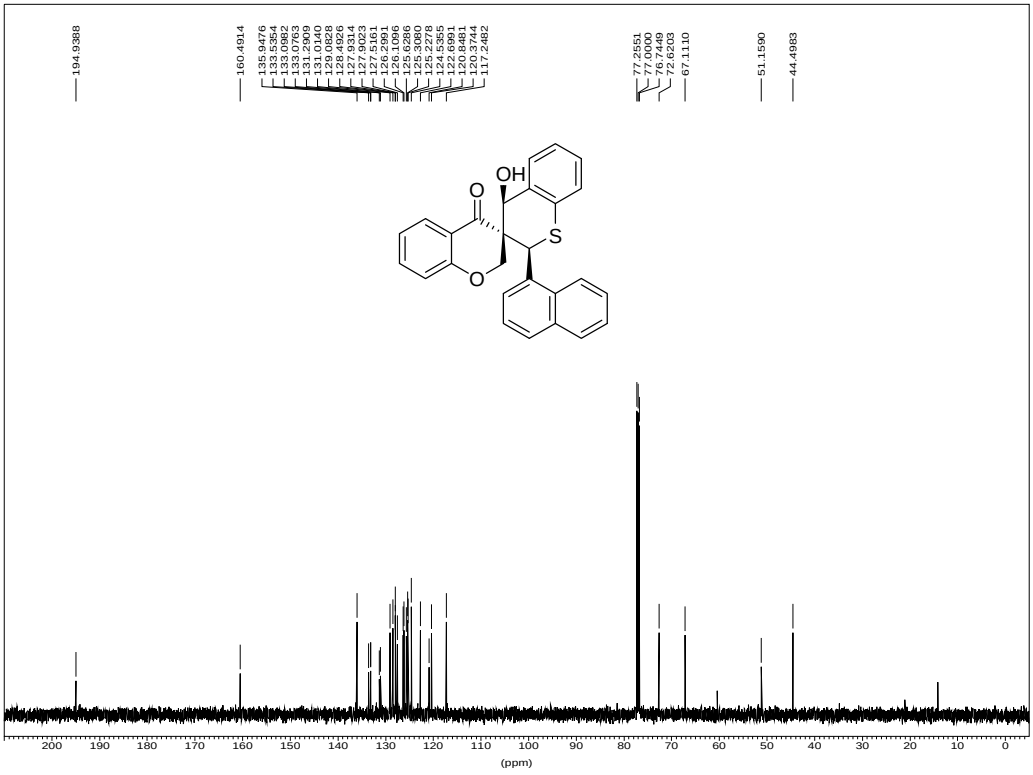
Compound **8o**


7.7774
7.7749
7.7623
7.7595
7.6867
7.6703
7.3400
7.3374
7.3296
7.3097
7.3059
7.2505
7.2416
7.2365
7.2126
7.1912
7.1799
7.1691
7.1660
7.1635
7.0513
7.0412
7.0323
7.0260
6.8987
6.8848
6.8697
6.8598
6.7298
6.7184
6.7121
5.4590
5.4426
5.4274
4.8147
4.7897
4.6458
4.6206
2.9955
2.9791
OH
O
S
O
S
Integral
1.9081
0.9857
2.9480
1.8403
0.9879
1.8578
1.9325
1.0000
0.9989
0.9618
(ppm)

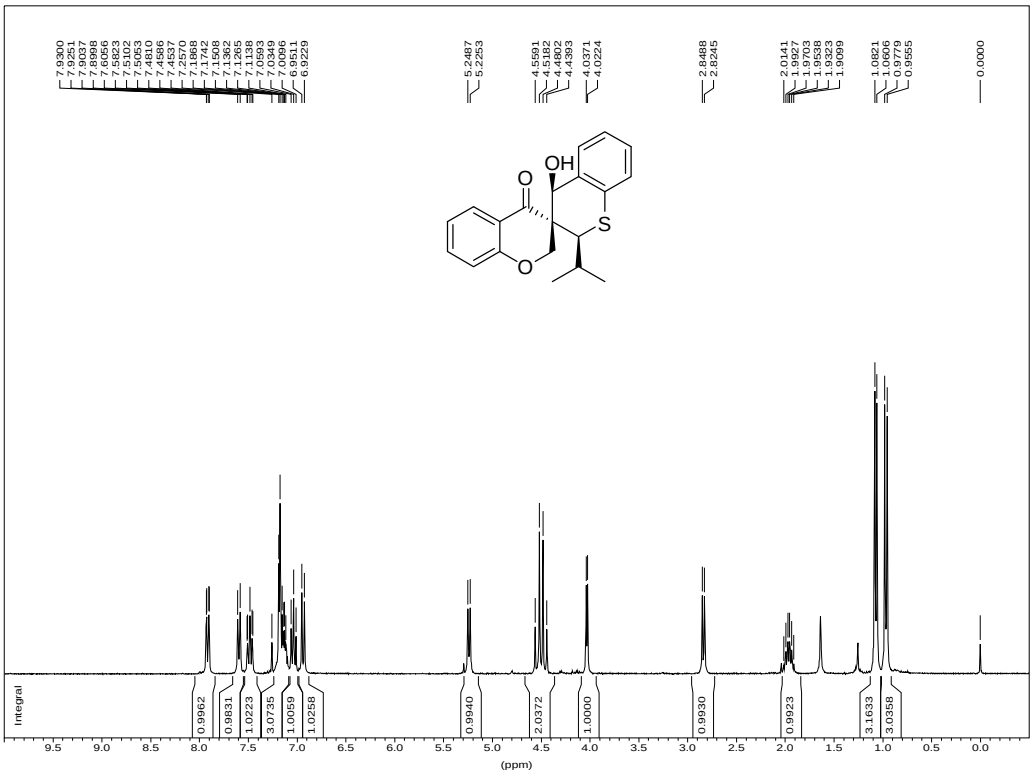

195.2303
161.2857
137.7475
136.1880
133.4115
132.3330
128.8217
128.0023
127.1663
126.9622
126.3136
125.5776
125.1569
124.8707
121.9995
121.3437
117.8822
77.2551
77.0000
76.7449
73.6842
67.1620
51.6691
45.5550
OH
O
S
O
S
(ppm)

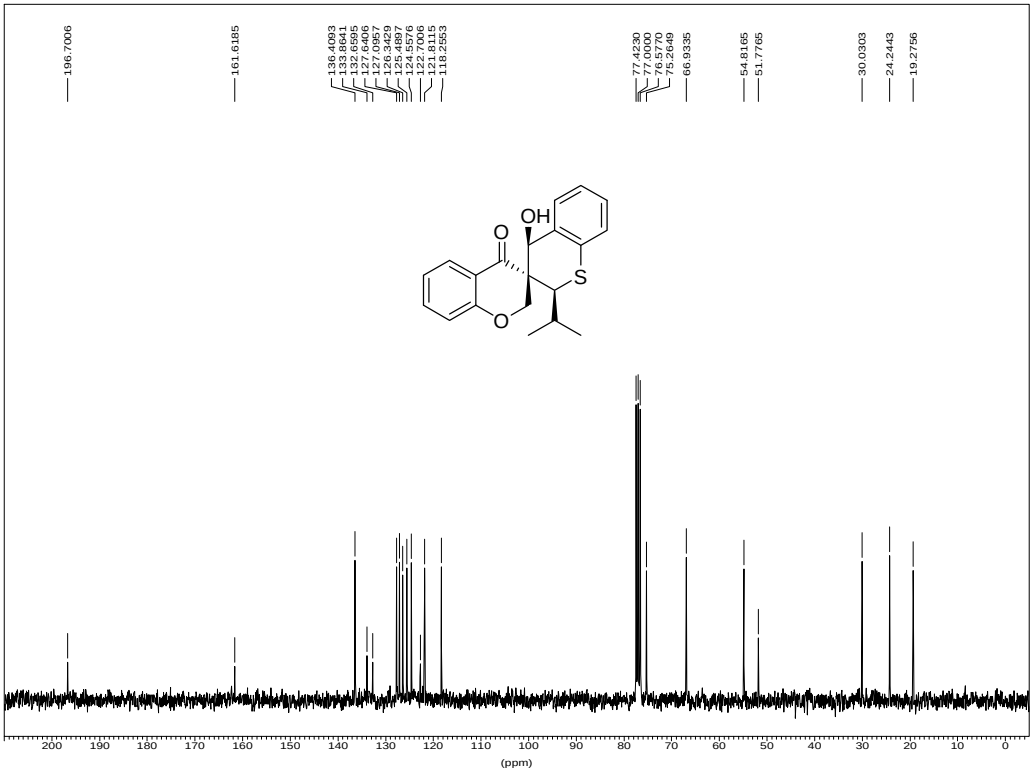
Compound **8p**



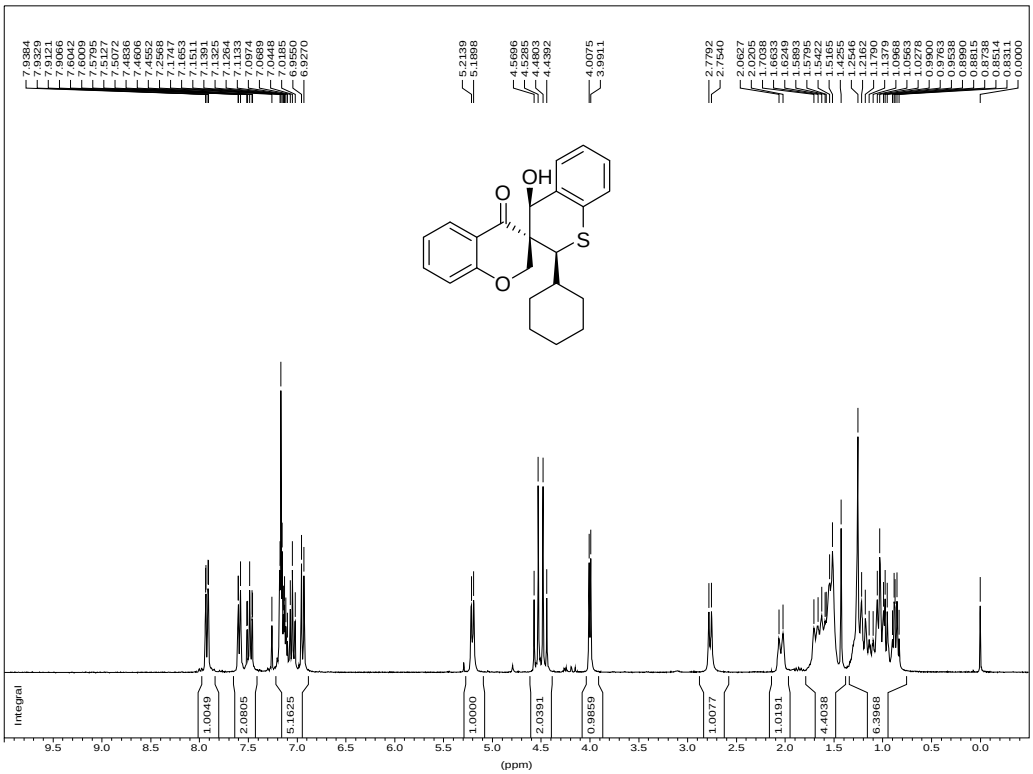


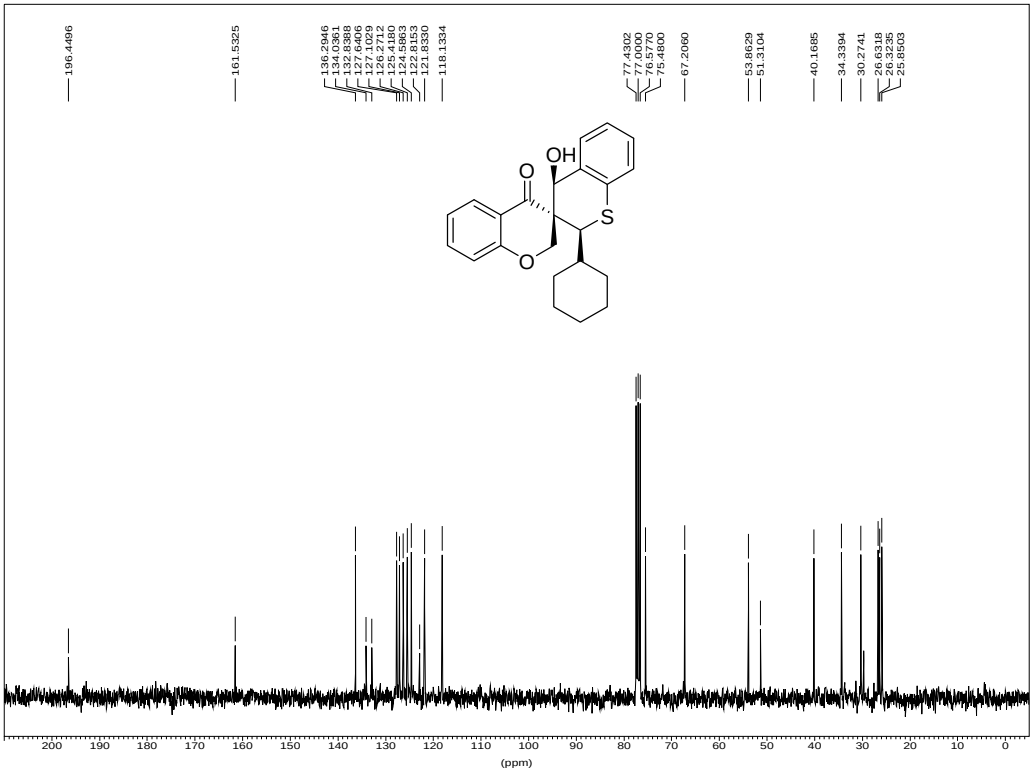
Compound **8q**


7.9300
7.9251
7.9037
7.8998
7.6056
7.5693
7.5100
7.5053
7.4810
7.4586
7.4537
7.2570
7.1868
7.1742
7.1508
7.1382
7.1265
7.1138
7.0593
7.0349
7.0096
6.9511
6.9229
5.2487
5.2253
4.5591
4.5182
4.4802
4.4393
4.0371
4.0224
2.8488
2.8245
2.0141
1.9927
1.9703
1.9538
1.9323
1.9099
1.0821
1.0606
0.9770
0.9555
0.0000
OH
O
S
O
Integral
0.9962
0.9831
1.0223
3.0735
1.0059
1.0258
0.9940
2.0372
1.0000
0.9930
0.9923
3.1633
3.0358
(ppm)

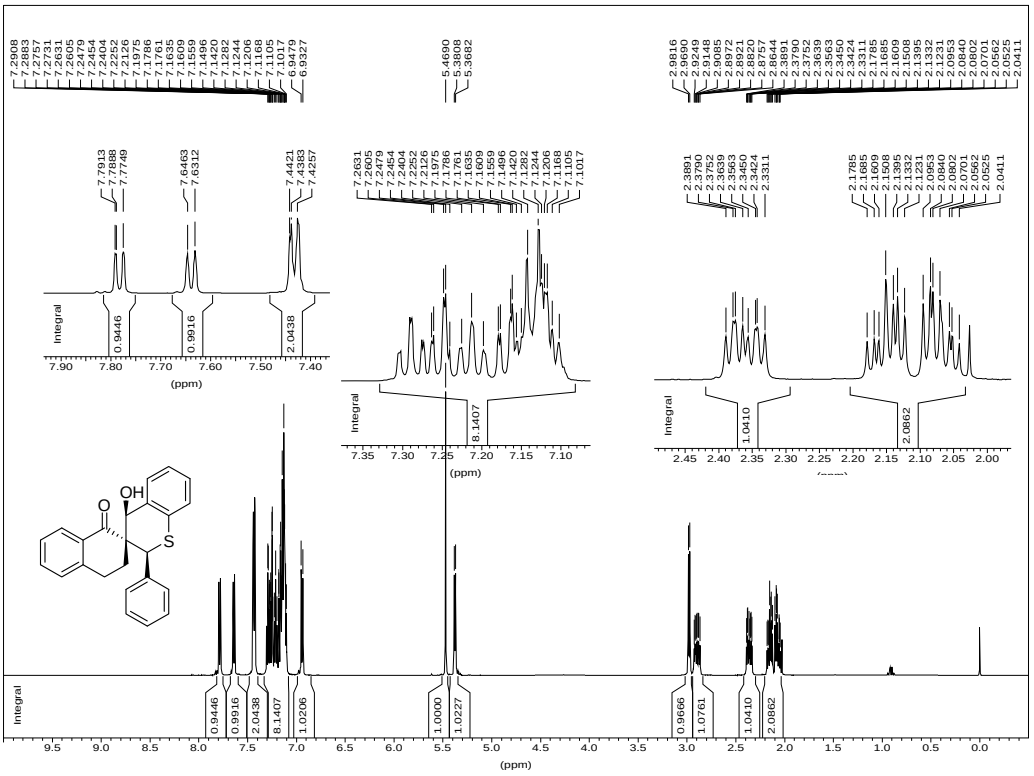

196.7006
161.6185
136.4093
133.8641
132.6895
127.6645
127.0957
126.3429
125.4897
124.4576
122.7006
121.8115
118.2553
77.4230
77.0000
76.5770
75.2649
66.9335
54.8165
51.7765
30.0303
24.2443
19.2756
OH
O
S
O
(ppm)

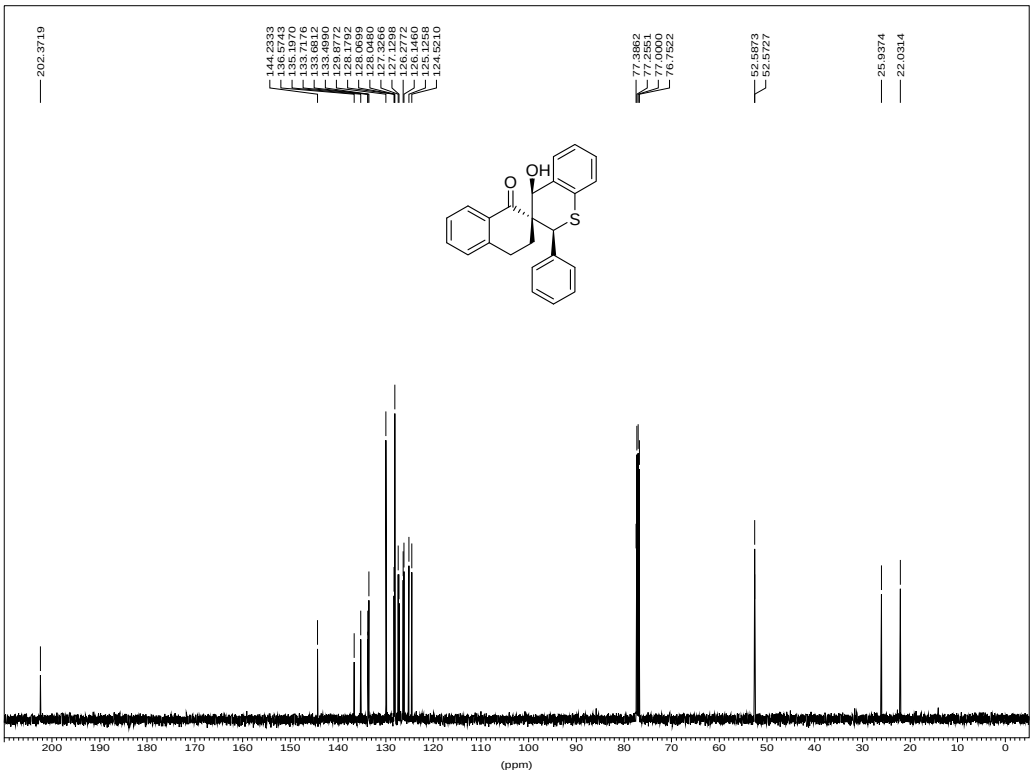
Compound **8r**


7.9384
7.9329
7.9121
7.9066
7.6042
7.6009
7.5795
7.5127
7.5072
7.4836
7.4606
7.4552
7.2568
7.1747
7.1663
7.1511
7.1391
7.1325
7.1264
7.1133
7.0974
7.0689
7.0448
7.0155
6.9550
6.9270
5.2139
5.1898
4.5696
4.5285
4.4803
4.4592
4.0075
3.9911
2.7792
2.7540
2.0627
2.0205
1.7038
1.6633
1.6249
1.5893
1.5795
1.5422
1.5155
1.4255
1.2546
1.2162
1.1790
1.1379
1.0968
1.0563
1.0278
0.9900
0.9655
0.9538
0.8990
0.8815
0.8738
0.8514
0.8311
0.0000
OH
O
S
O
Integral
1.0049
2.0805
5.1625
1.0000
2.0391
0.9859
1.0077
1.0191
4.4038
6.3968
(ppm)

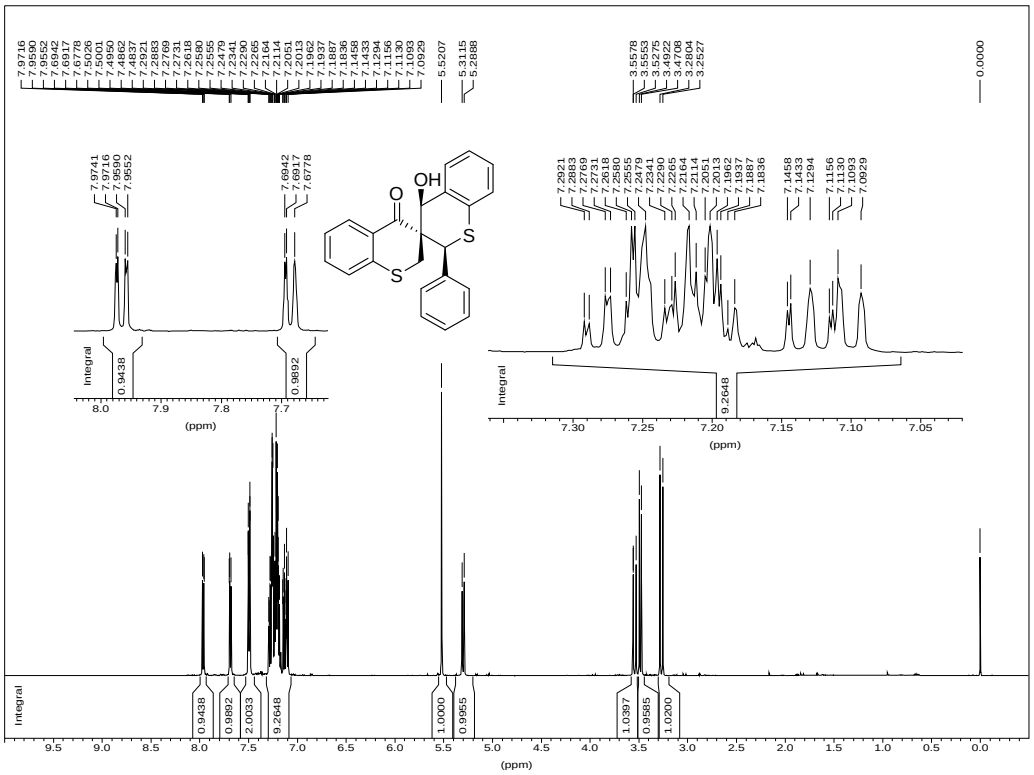

196.4496
161.5325
136.2946
134.0361
132.8388
127.6406
127.1029
126.2772
125.4180
124.5863
122.8153
121.8330
118.1334
77.4302
77.0000
76.5770
75.4600
67.2060
53.8629
51.3104
40.1685
34.3394
30.2741
26.6318
26.3235
25.8503
OH
O
S
O
(ppm)

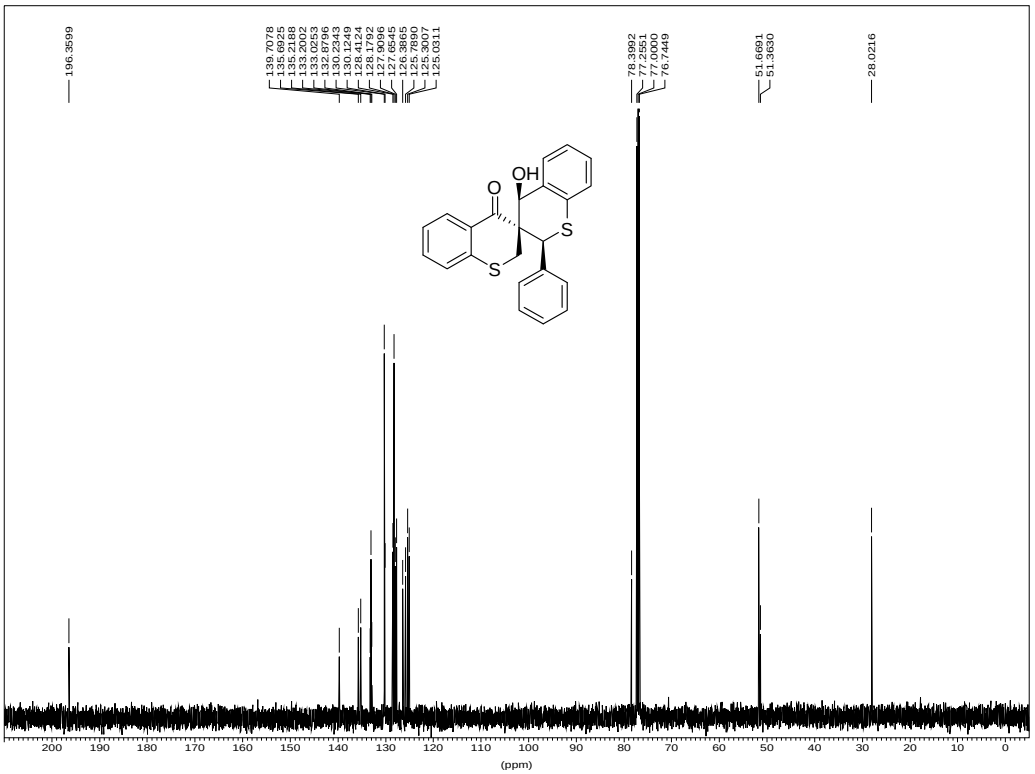
Compound **8s**



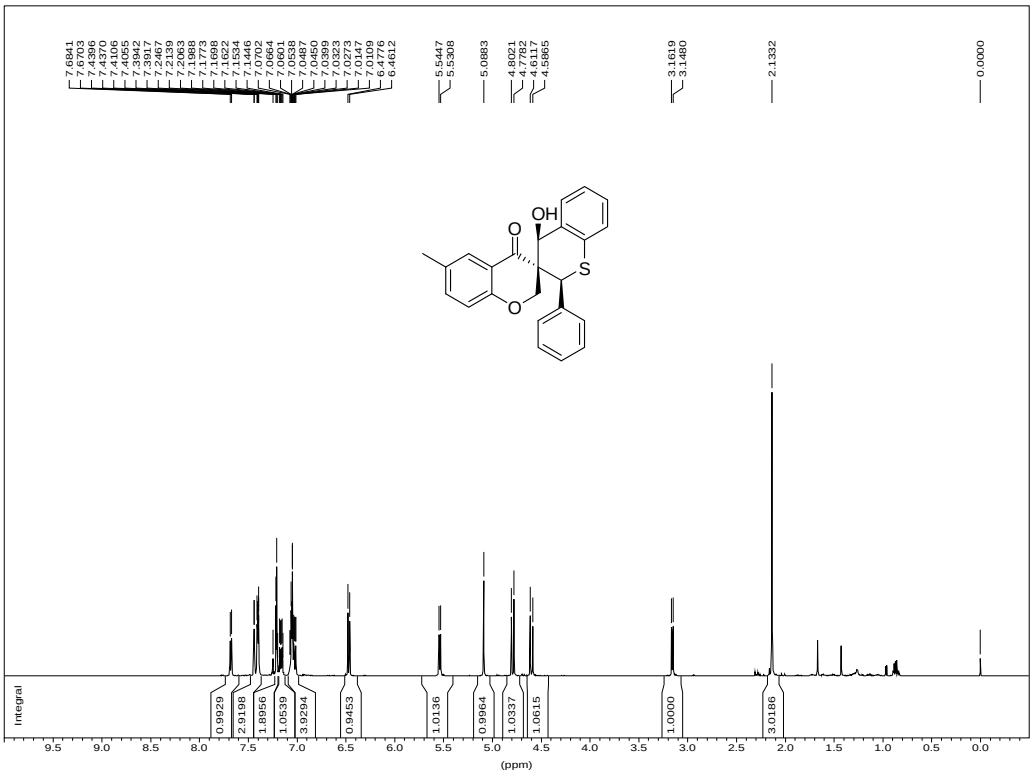


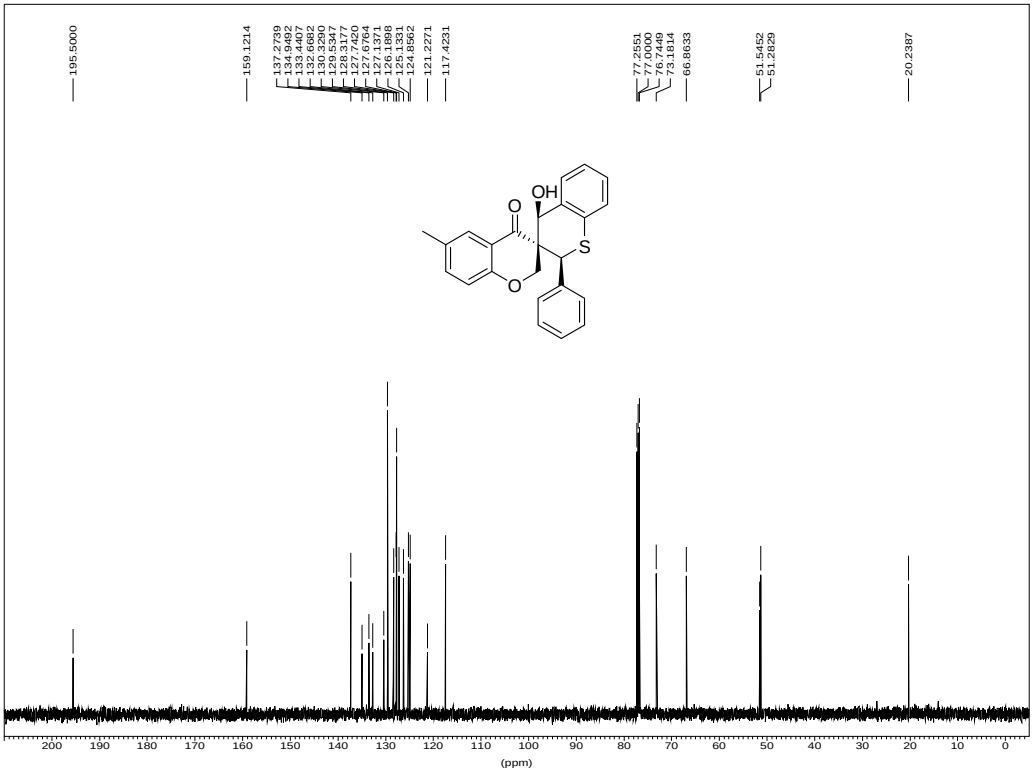
Compound **8t**


OH
O
S
S
Integral
0.9438
0.9892
2.0033
9.2648
1.0000
0.9965
1.0397
0.9585
1.0200
(ppm)

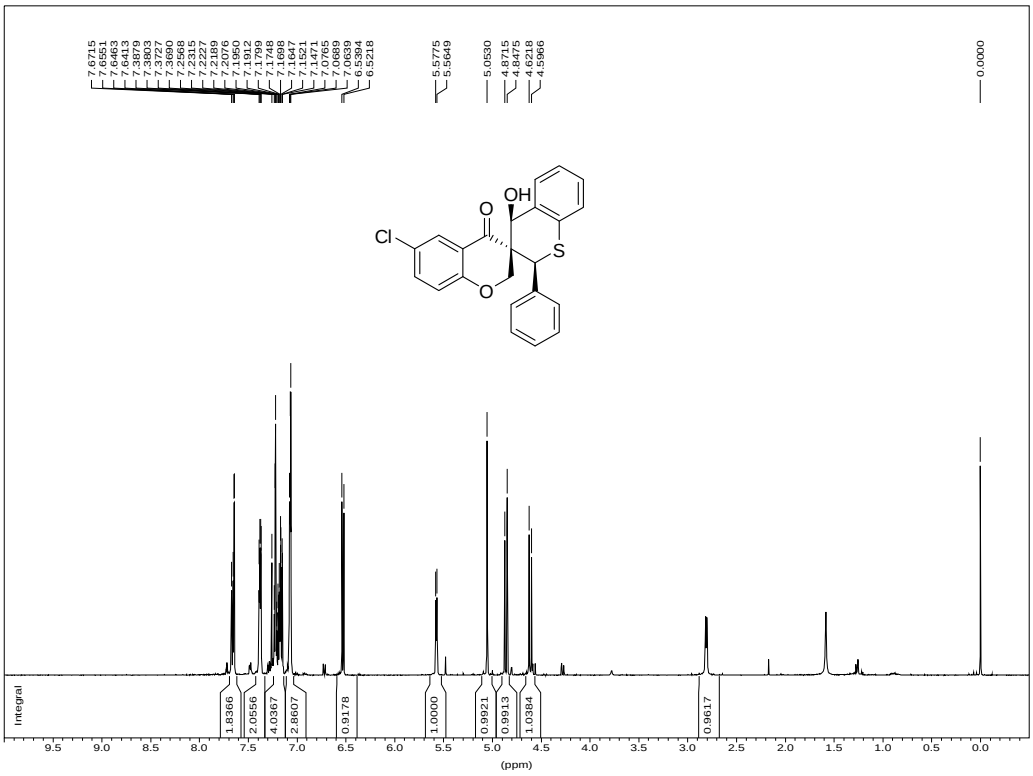

196.3599
139.7078
135.6925
135.2188
133.2002
133.0253
132.9746
130.2343
130.1249
128.4124
128.1792
127.9046
127.6545
126.3865
125.7890
125.3007
125.0311
78.3992
77.2551
77.0000
76.7449
51.6691
51.3630
28.0216
OH
O
S
S
(ppm)

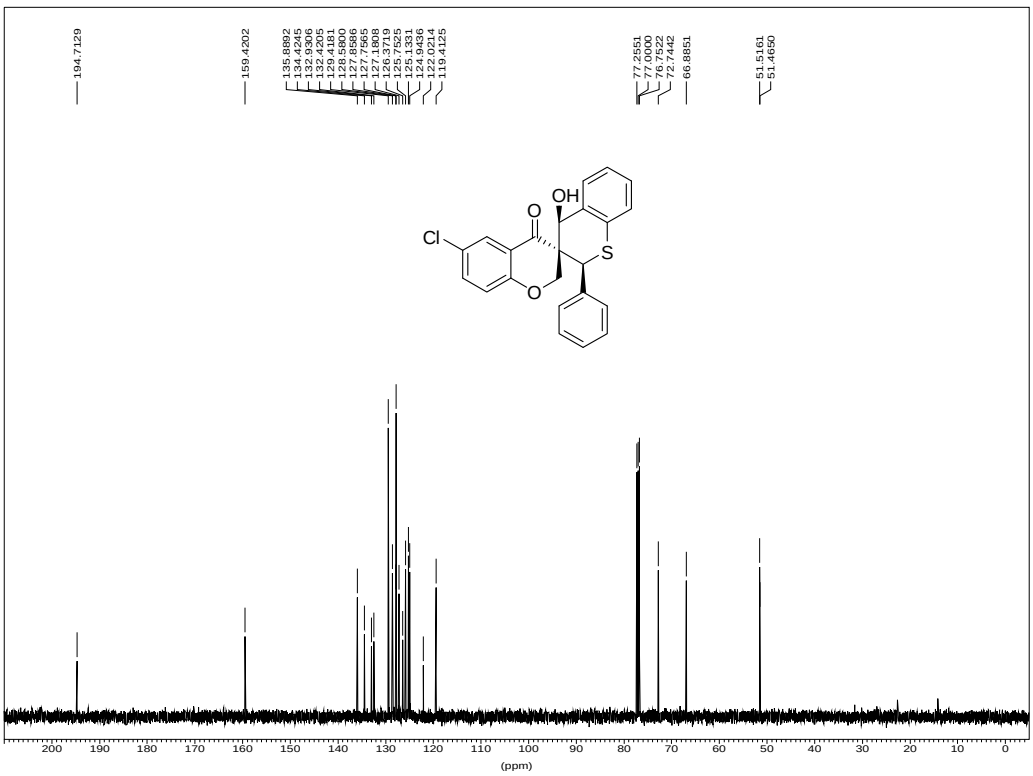
Compound **8u**


7.6841
7.6703
7.4396
7.4370
7.4156
7.4056
7.3942
7.3917
7.2467
7.2159
7.2063
7.1988
7.1773
7.1698
7.1622
7.1534
7.1446
7.0702
7.0664
7.0601
7.0538
7.0487
7.0450
7.0399
7.0323
7.0273
7.0147
7.0109
6.4778
6.4612
5.5447
5.5308
5.0883
4.8021
4.7782
4.6617
4.5865
3.1619
3.1480
2.1332
0.0000
OH
O
S
O
Integral
0.9929
2.9198
1.8956
1.0539
3.9294
0.9453
1.0136
0.9964
1.0337
1.0615
1.0000
3.0186
(ppm)

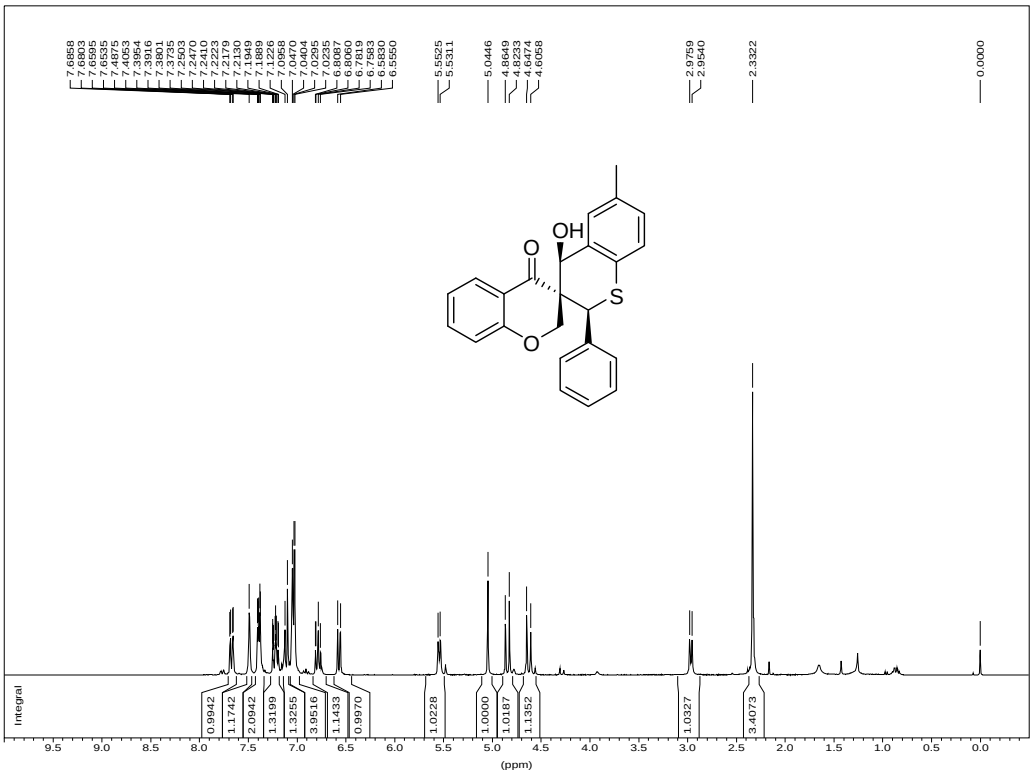

195.5000
159.1214
137.2739
134.9482
133.4407
132.9682
130.3290
129.5347
128.3177
127.7420
127.3764
127.1371
126.1898
125.1331
124.8562
121.2271
117.4231
77.2551
77.0000
76.7449
73.1814
66.8633
51.5452
51.2829
20.2387
OH
O
S
O
(ppm)

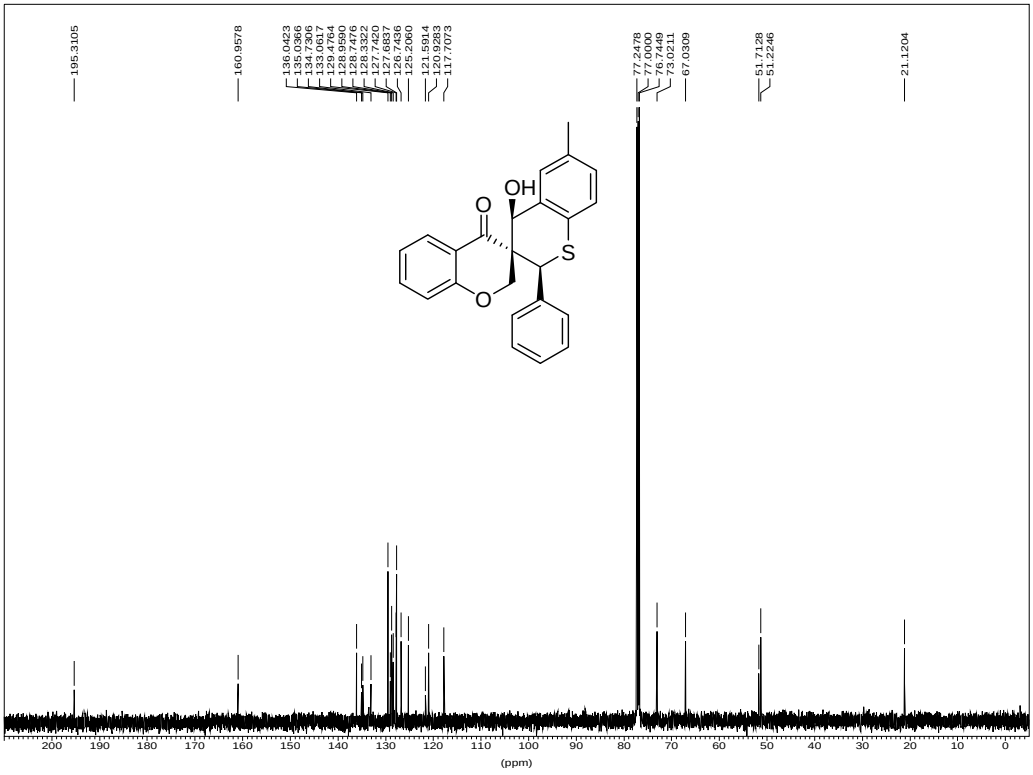
Compound **8v**


7.6715
7.6651
7.6465
7.6413
7.3879
7.3803
7.3727
7.3660
7.2568
7.2315
7.2227
7.2109
7.2070
7.1950
7.1912
7.1799
7.1748
7.1698
7.1647
7.1521
7.1471
7.1065
7.0689
7.0639
6.5394
6.5218
5.5775
5.5649
5.0530
4.8715
4.8475
4.6218
4.5966
0.0000
OH
O
Cl
S
O
Integral
1.8366
2.0556
4.0367
2.8607
0.9178
1.0000
0.9921
0.9913
1.0384
0.9617
(ppm)

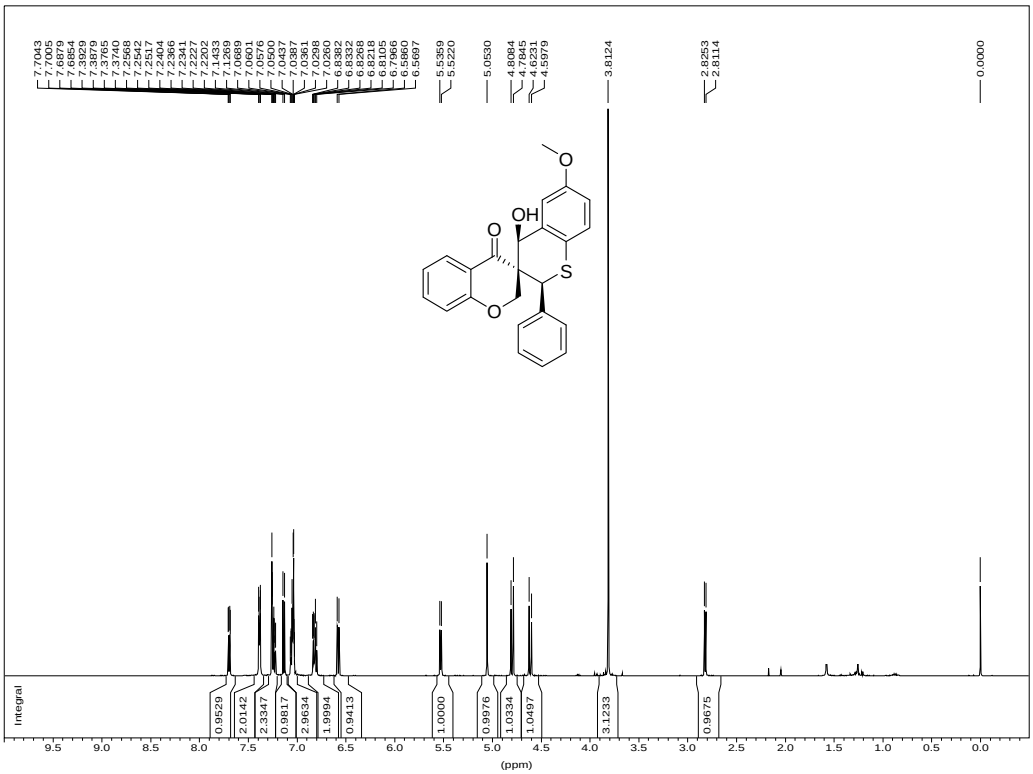

194.7129
159.4202
135.8882
134.4745
132.9436
132.4205
129.4181
128.5800
128.0966
127.7565
127.1808
126.3719
125.7525
125.5331
124.9436
122.0214
119.4125
77.2551
77.0000
76.7449
72.7442
66.8851
51.5161
51.4650
OH
O
Cl
S
O
(ppm)

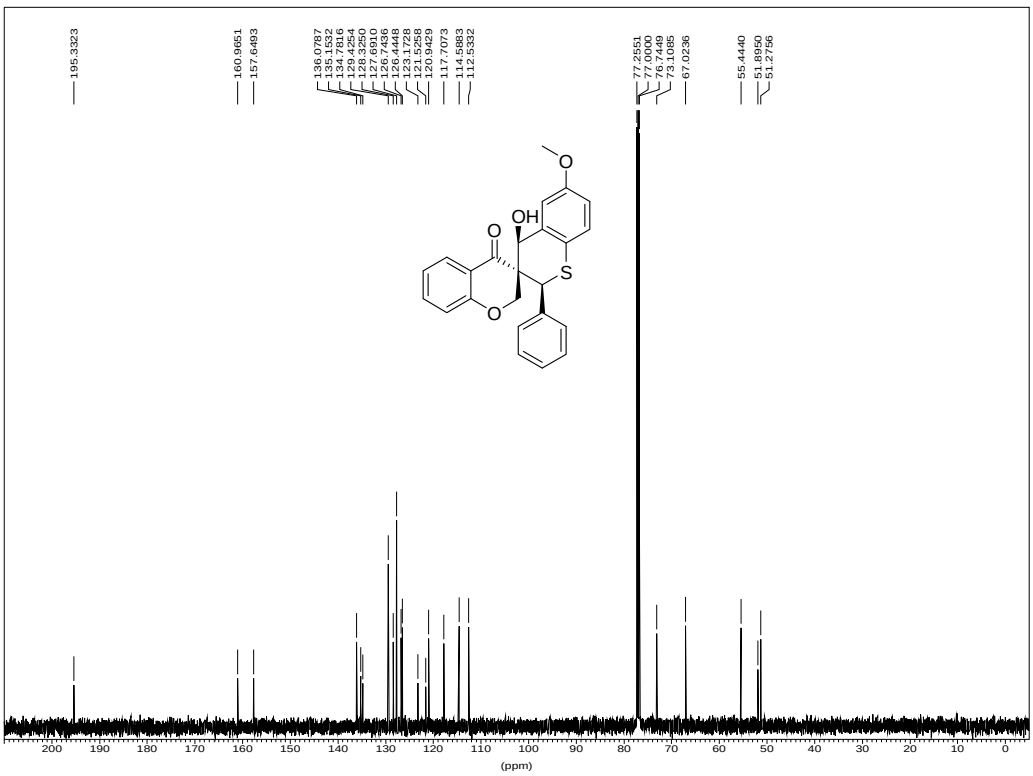
Compound **8w**


7.6858
7.6803
7.6595
7.6535
7.4675
7.4063
7.3954
7.3916
7.3801
7.3735
7.2503
7.2470
7.2410
7.2223
7.2170
7.2130
7.1949
7.1889
7.1226
7.0966
7.0470
7.0404
7.0295
7.0235
6.8067
6.8060
6.7819
6.7583
6.5630
6.5560
5.5525
5.5311
5.0446
4.8649
4.8233
4.6474
4.6058
2.9759
2.9540
2.3322
0.0000
OH
O
S
O
Integral
0.9942
1.1742
2.0942
1.3199
1.3255
3.9516
1.1433
0.9970
1.0228
1.0000
1.0187
1.1352
1.0327
3.4073
(ppm)

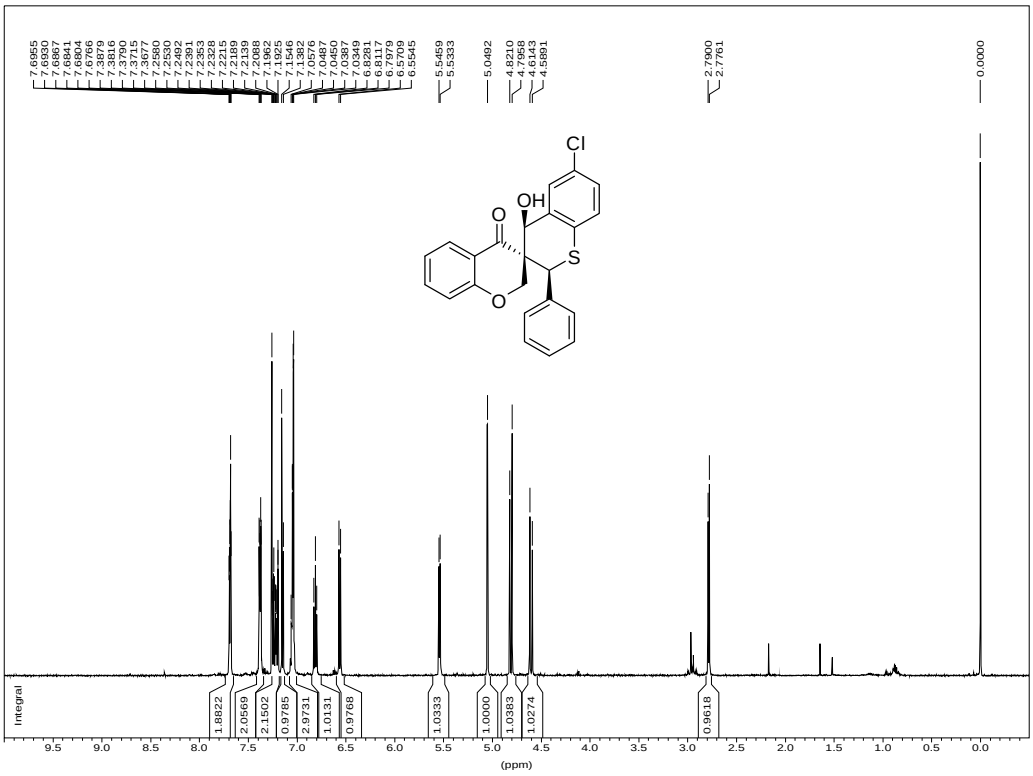

195.3105
160.9578
136.0423
135.7386
134.7436
133.0617
129.4764
128.9590
128.4476
128.3322
127.7420
127.6837
126.7436
125.2060
121.5914
120.2983
117.7073
77.2478
77.0000
76.7461
73.0211
67.0309
51.7128
51.2246
21.1204
OH
O
S
O
(ppm)

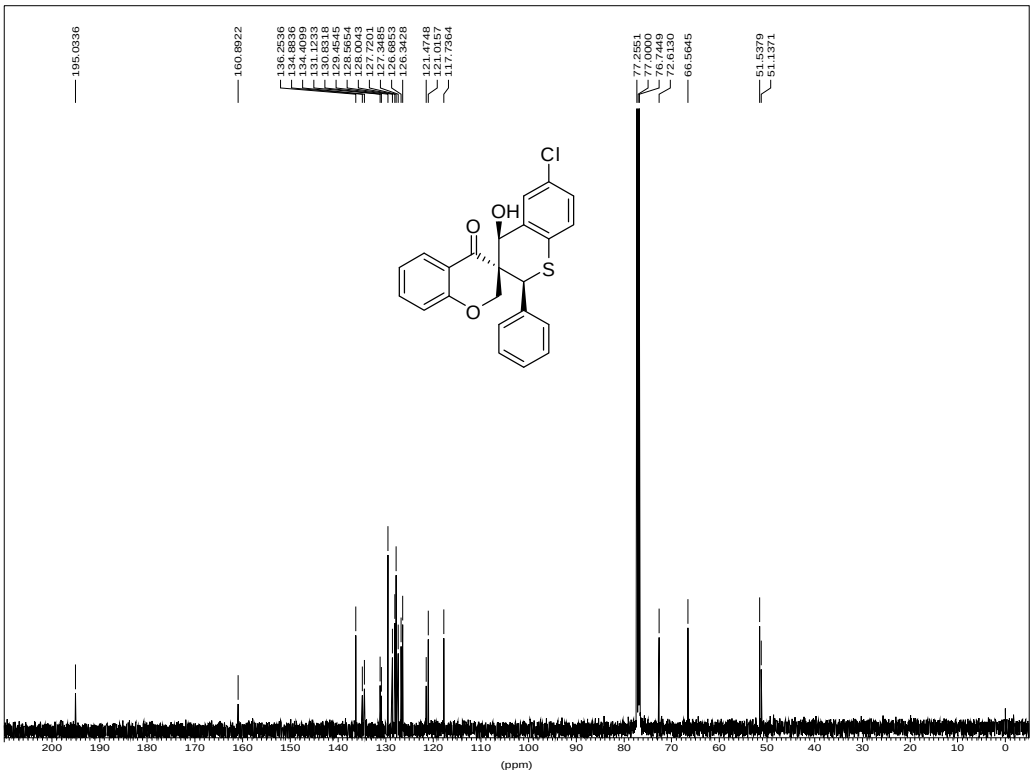
Compound **8x**


7.7043
7.7005
7.6873
7.6854
7.3929
7.3879
7.3765
7.3740
7.2566
7.2542
7.2517
7.2404
7.2386
7.2341
7.2227
7.2202
7.1433
7.1289
7.1089
7.0601
7.0576
7.0500
7.0437
7.0387
7.0361
7.0298
7.0260
6.8882
6.8332
6.8268
6.8218
6.8105
6.7966
6.5860
6.5697
5.5359
5.5220
5.0530
4.8084
4.7845
4.6251
4.5979
3.8124
2.8253
2.8114
0.0000
O
OH
O
S
O
Integral
0.9529
2.0142
2.3347
0.9817
2.9634
1.9994
0.9413
1.0000
0.9976
1.0334
1.0497
3.1233
0.9675
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

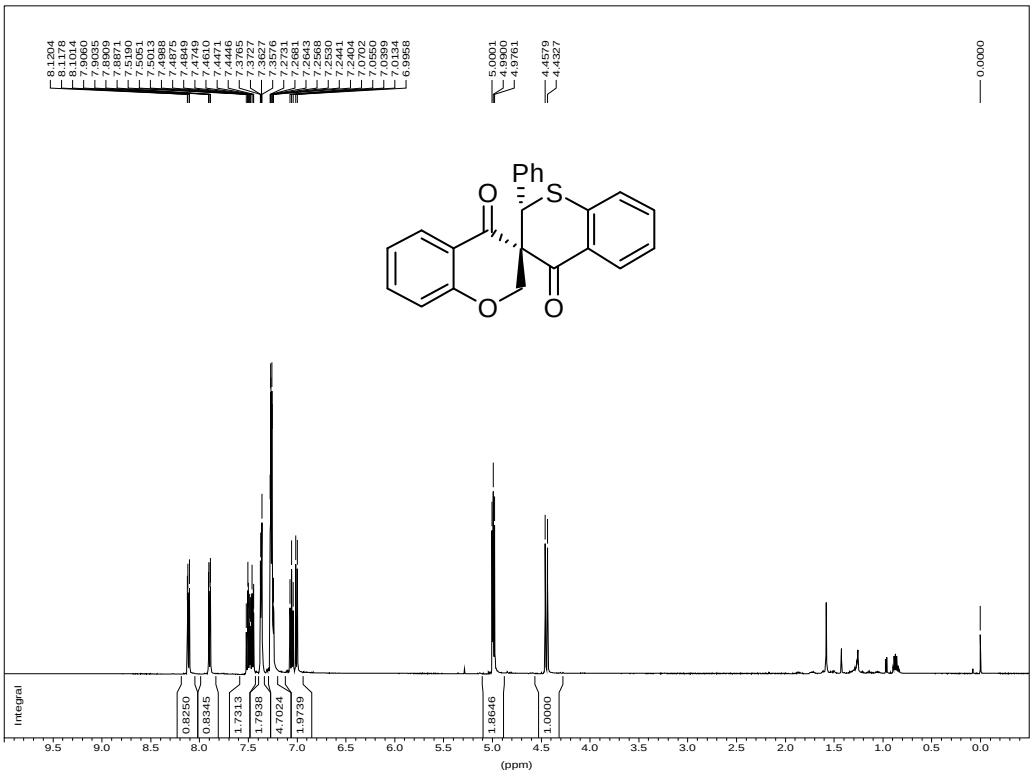

195.3323
160.9651
157.6493
136.0787
135.1532
134.7316
134.2254
128.3250
127.6910
126.7436
126.7448
123.1728
121.5258
120.9429
117.7073
114.5883
112.5332
77.2551
77.0000
76.7449
73.1085
67.0236
55.4440
51.8950
51.2756
O
OH
O
S
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

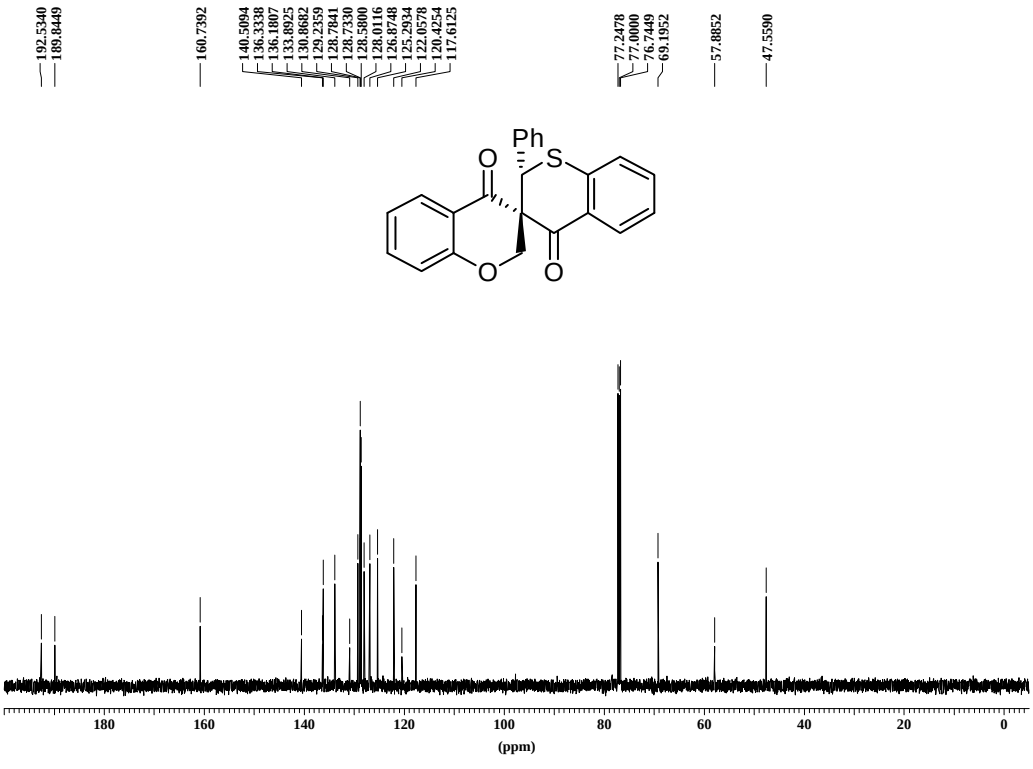
Compound **8y**



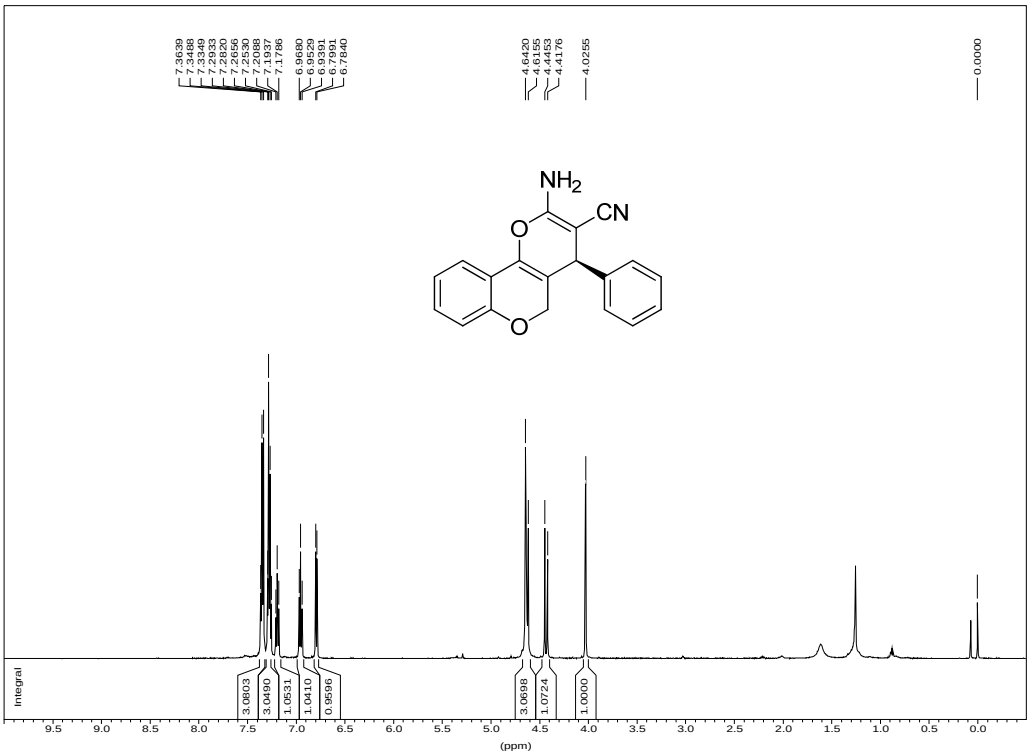


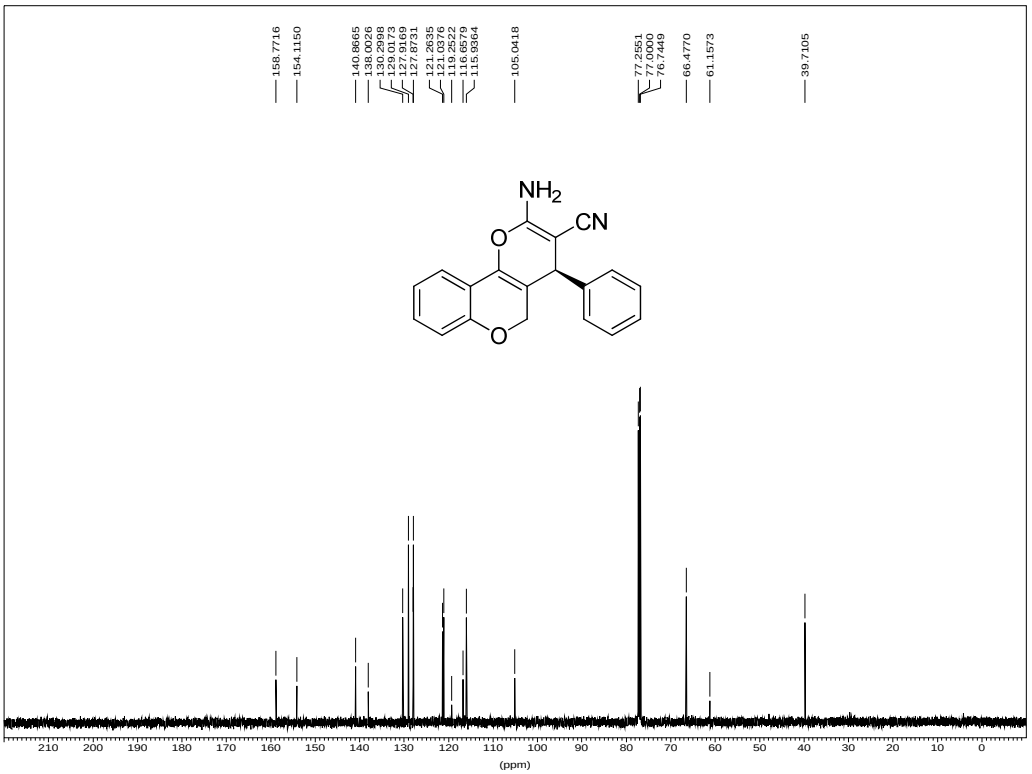
Compound **9**


8.1204
8.1178
8.1041
8.1014
7.9060
7.9035
7.8909
7.8871
7.5100
7.5061
7.5013
7.4988
7.4875
7.4648
7.4749
7.4610
7.4471
7.4446
7.3655
7.3727
7.3627
7.3576
7.2731
7.2681
7.2643
7.2568
7.2530
7.2441
7.2404
7.0702
7.0550
7.0399
7.0134
6.9958
5.0001
4.9900
4.9761
4.4579
4.4327
0.0000
Ph
O
S
O
O
Integral
0.8250
0.8345
1.7313
1.7938
4.7024
1.9739
1.8646
1.0000
(ppm)

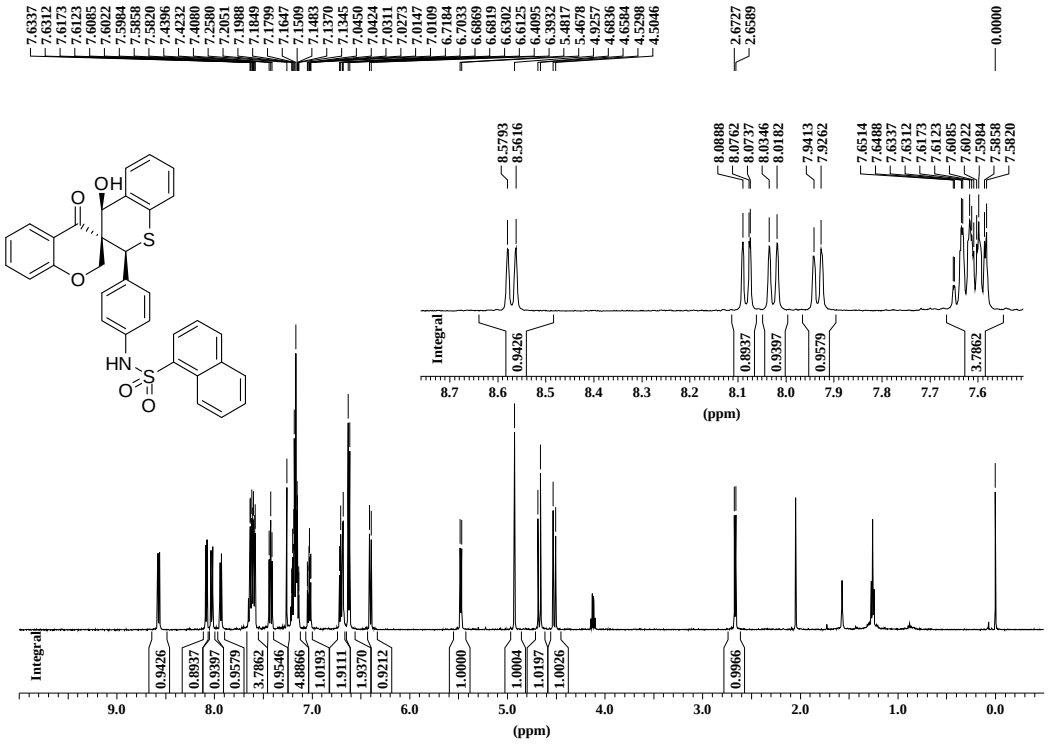

192.5340
189.8449
160.7392
140.5094
136.3338
136.1807
133.8925
130.8682
129.2359
128.7841
128.7330
128.5800
128.0116
126.8748
125.2934
122.0578
120.4254
117.6125
77.2478
77.0000
76.7449
69.1952
57.8852
47.5590
Ph
O
S
O
O
(ppm)

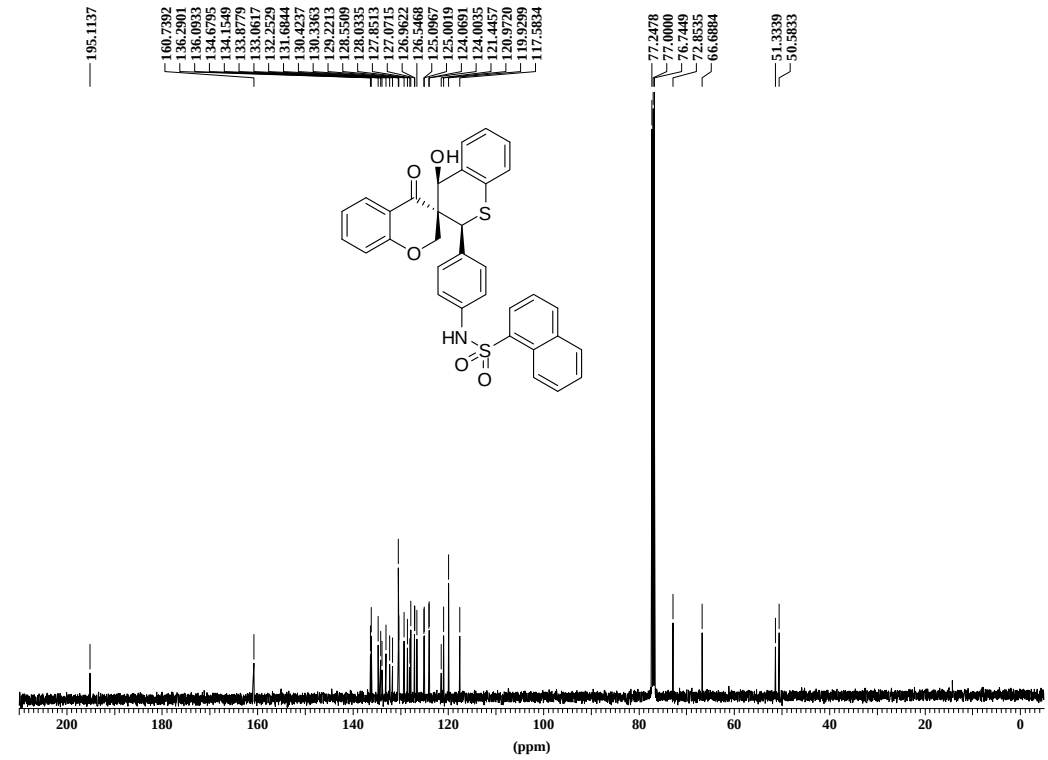
Compound **11**


7.3639
7.3488
7.3349
7.2953
7.2820
7.2656
7.2530
7.2088
7.1937
7.1786
6.9680
6.9539
6.9391
6.7991
6.7840
4.6420
4.6155
4.4455
4.4176
4.0255
0.0000
NH2
CN
O
O
Integral
3.0803
3.0490
1.0531
1.0410
0.9596
3.0698
1.0724
1.0000
(ppm)


158.7716
154.1150
140.8665
138.0026
130.2998
129.0173
127.9169
127.8731
121.2635
121.0376
119.2572
116.6579
115.9364
105.0418
77.2551
77.0000
76.7449
66.4770
61.1573
39.7105
NH2
CN
O
O
(ppm)

Compound **12**


7.6337
7.6312
7.6173
7.6123
7.6085
7.6022
7.5984
7.5858
7.5820
7.4396
7.4232
7.4080
7.2580
7.2051
7.1988
7.1849
7.1799
7.1647
7.1509
7.1483
7.1370
7.1345
7.0450
7.0424
7.0311
7.0273
7.0147
7.0109
6.7184
6.7033
6.6869
6.6819
6.6302
6.6125
6.4095
6.3932
5.4817
5.4678
4.9257
4.6836
4.6584
4.5298
4.5046
2.6727
2.6589
0.0000
8.5793
8.5616
8.0888
8.0762
8.0737
8.0346
8.0182
7.9413
7.9262
7.6514
7.6488
7.6337
7.6312
7.6173
7.6123
7.6085
7.6022
7.5984
7.5858
7.5820
Integral
0.9426
0.8937
0.9397
0.9579
3.7862
(ppm)
Integral
0.9426
0.8937
0.9397
0.9579
3.7862
0.9546
4.8866
1.0193
1.9111
1.9370
0.9212
1.0000
1.0004
1.0197
1.0026
0.9966
(ppm)

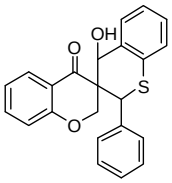

195.1137
160.7392
136.2901
136.0933
134.6795
134.1549
133.8779
133.0617
132.2529
131.6844
130.4237
130.3363
129.2213
128.5509
128.0335
127.8513
127.0715
126.9622
126.5468
125.0967
125.0019
124.0691
124.0035
121.4457
120.9720
119.9299
117.5834
77.2478
77.0000
76.7449
72.8535
66.6884
51.3339
50.5833
(ppm)

Racemic **8a**

==== Shimadzu LCsolution Analysis Report ====

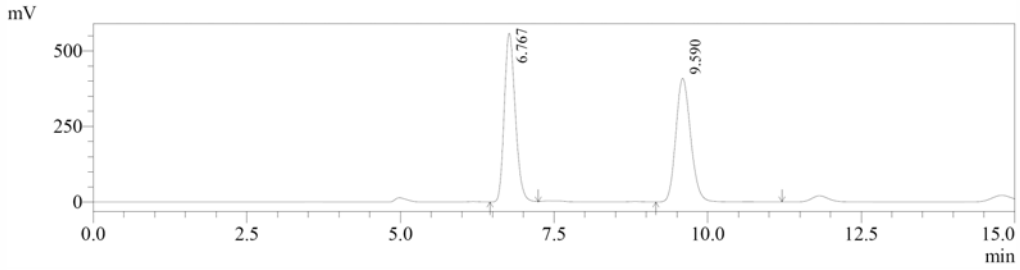
C:\Users\User\Desktop\LC data\Gao Yaojun\G089.lcd

Acquired by : Admin
Sample Name : GB036P1
Sample ID : GYJ
Data File Name : G089.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB036P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G089.lcd



PeakTable

SPD-20A Ch2 254nm

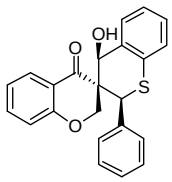
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.767	6961214	558294	50.379	57.705
2	9.590	6856543	409198	49.621	42.295
Total		13817757	967492	100.000	100.000

Enantiomeric enriched **8a**

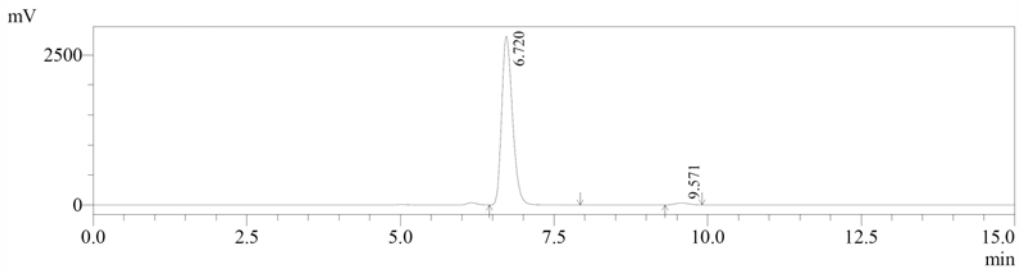
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G096.lcd

Acquired by : Admin
Sample Name : GB044P1- xylene
Sample ID : GYJ
Data File Name : G096.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA,



Chromatogram
GB044P1- xylene C:\Users\User\Desktop\LC data\Gao Yaojun\G096.lcd



PeakTable

SPD-20A Ch2 254nm

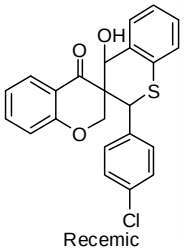
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.720	34413950	2811297	98.532	98.875
2	9.571	512696	31976	1.468	1.125
Total		34926645	2843273	100.000	100.000

Racemic **8b**

==== Shimadzu LCsolution Analysis Report ====

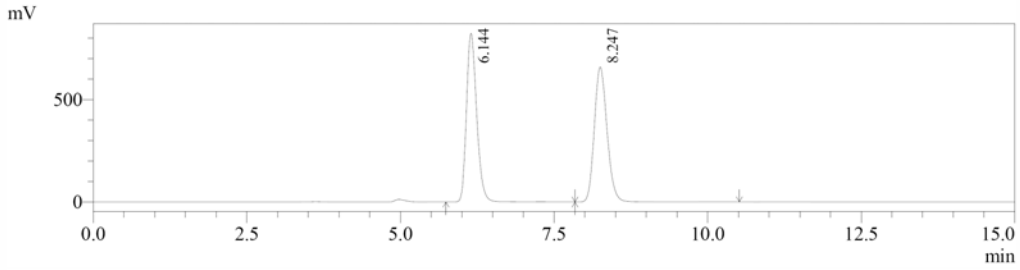
C:\Users\User\Desktop\LC data\Gao Yaojun\G115.lcd

Acquired by : Admin
Sample Name : GB068P1 recemic
Sample ID : GYJ
Data File Name : G115.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA,



Recemic

Chromatogram
GB068P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G115.lcd



PeakTable

SPD-20A Ch2 254nm

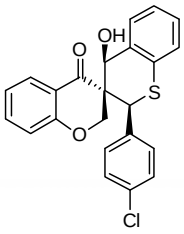
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.144	9529727	824123	50.049	55.553
2	8.247	9510944	659376	49.951	44.447
Total		19040671	1483500	100.000	100.000

Enantiomeric enriched **8b**

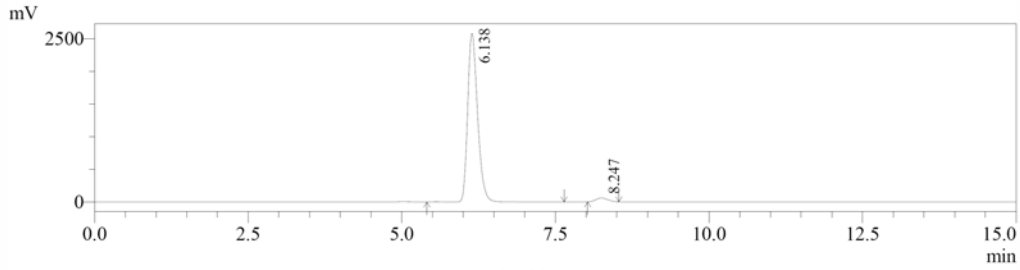
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G122.lcd

Acquired by : Admin
Sample Name : GB069P1
Sample ID : GYJ
Data File Name : G122.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Chromatogram
GB069P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G122.lcd



PeakTable

SPD-20A Ch2 254nm

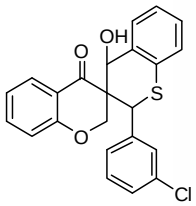
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.138	29104088	2586617	97.276	97.719
2	8.247	814947	60377	2.724	2.281
Total		29919035	2646994	100.000	100.000

Racemic **8c**

==== Shimadzu LCsolution Analysis Report ====

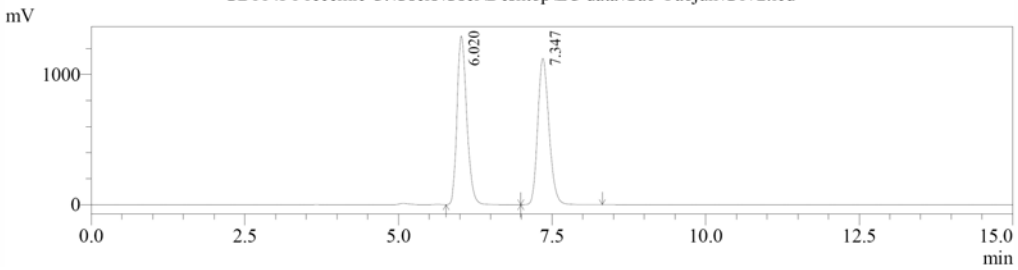
C:\Users\User\Desktop\LC data\Gao Yaojun\G172.lcd

Acquired by : Admin
Sample Name : GB114P1 recemic
Sample ID : GYJ
Data File Name : G172.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB114P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G172.lcd



PeakTable

SPD-20A Ch1 254nm

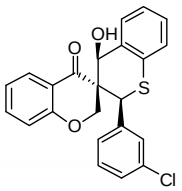
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.020	14301311	1296519	50.004	53.564
2	7.347	14299160	1123981	49.996	46.436
Total		28600472	2420501	100.000	100.000

Enantiomeric enriched **8c**

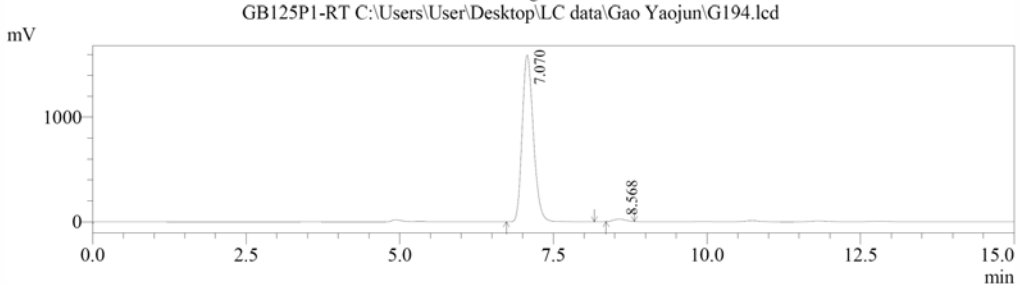
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G194.lcd

Acquired by : Admin
Sample Name : GB125P1-RT
Sample ID : GYJ
Data File Name : G194.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :Ic column with guard column,10%IPA



Chromatogram
GB125P1-RT C:\Users\User\Desktop\LC data\Gao Yaojun\G194.lcd



PeakTable

SPD-20A Ch1 254nm

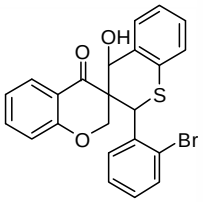
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.070	20216730	1596344	98.444	98.528
2	8.568	319454	23842	1.556	1.472
Total		20536184	1620186	100.000	100.000

Racemic **8d**

==== Shimadzu LCsolution Analysis Report ====

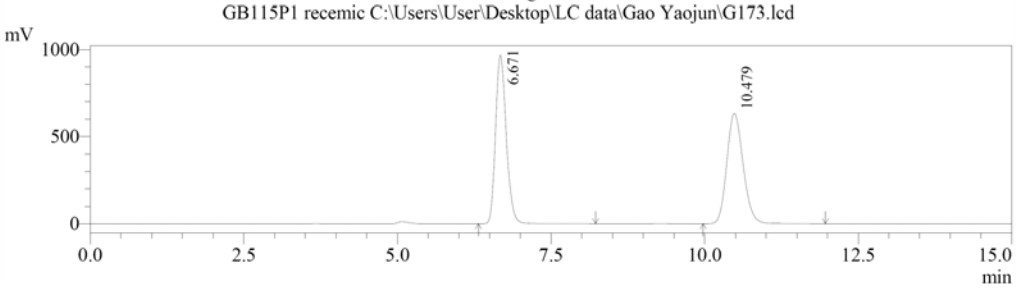
C:\Users\User\Desktop\LC data\Gao Yaojun\G173.lcd

Acquired by : Admin
Sample Name : GB115P1 recemic
Sample ID : GYJ
Data File Name : G173.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB115P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G173.lcd



PeakTable
SPD-20A Ch1 254nm

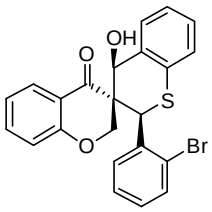
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.671	11708774	968306	50.028	60.445
2	10.479	11695767	633659	49.972	39.555
Total		23404541	1601965	100.000	100.000

Enantiomeric enriched **8d**

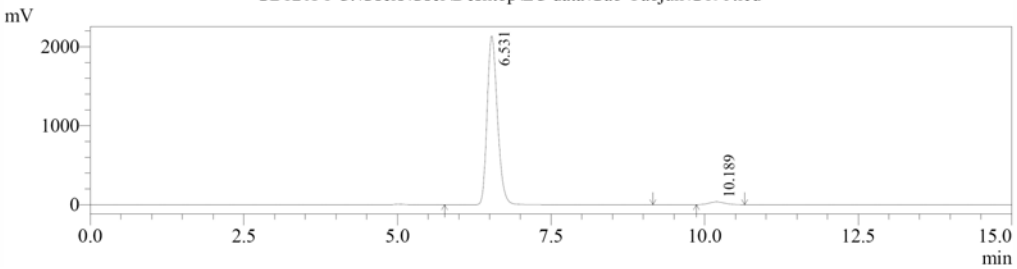
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G195.lcd

Acquired by : Admin
Sample Name : GB126P1
Sample ID : GYJ
Data File Name : G195.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB126P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G195.lcd



PeakTable
SPD-20A Ch1 254nm

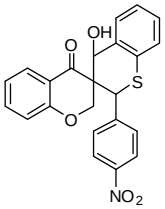
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.531	25026873	2138087	97.547	98.358
2	10.189	629339	35703	2.453	1.642
Total		25656212	2173790	100.000	100.000

Racemic **8e**

==== Shimadzu LCsolution Analysis Report ====

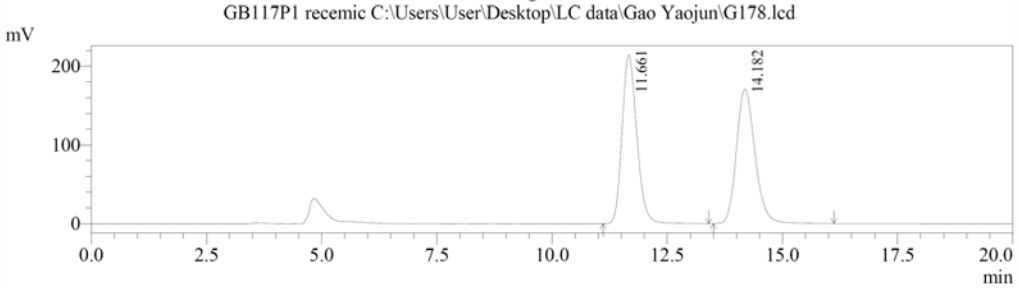
C:\Users\User\Desktop\LC data\Gao Yaojun\G178.lcd

Acquired by : Admin
Sample Name : GB117P1 recemic
Sample ID : GYJ
Data File Name : G178.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column



Recemic

Chromatogram
GB117P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G178.lcd



PeakTable

SPD-20A Ch1 254nm

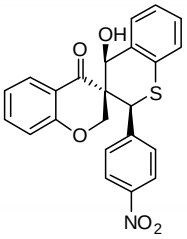
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.661	4781995	214291	50.040	55.650
2	14.182	4774387	170777	49.960	44.350
Total		9556382	385068	100.000	100.000

Enantiomeric enriched **8e**

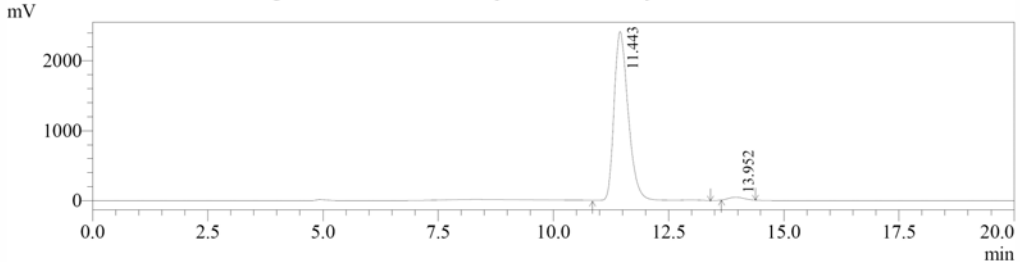
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G274.lcd

Acquired by : Admin
Sample Name : gc027
Sample ID : gyj
Data File Name : G274.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
gc027 C:\Users\User\Desktop\LC data\Gao Yaojun\G274.lcd



PeakTable

SPD-20A Ch1 254nm

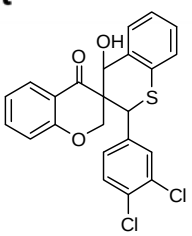
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.443	53167072	2428571	98.274	98.335
2	13.952	933708	41120	1.726	1.665
Total		54100780	2469690	100.000	100.000

Racemic **8f**

==== Shimadzu LCsolution Analysis Report ====

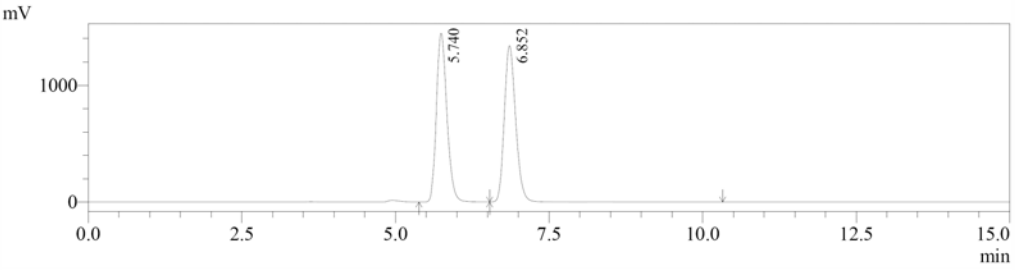
C:\Users\User\Desktop\LC data\Gao Yaojun\G118.lcd

Acquired by : Admin
Sample Name : GB070P1 recemic
Sample ID : GYJ
Data File Name : G118.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB070P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G118.lcd



PeakTable

SPD-20A Ch2 254nm

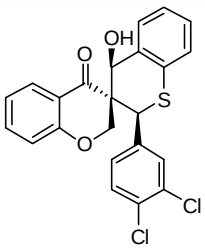
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.740	17217802	1445920	49.955	51.905
2	6.852	17248592	1339810	50.045	48.095
Total		34466394	2785730	100.000	100.000

Enantiomeric enriched **8f**

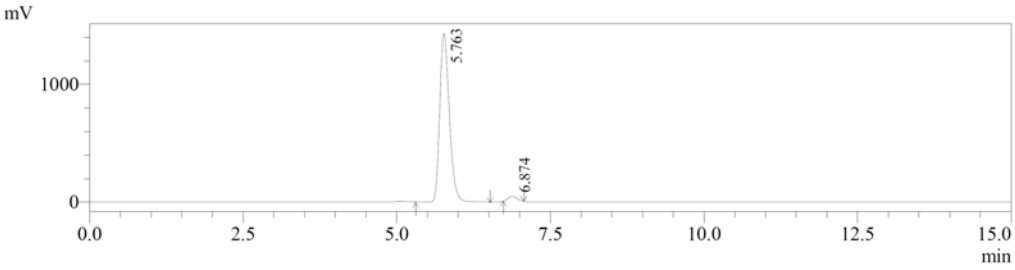
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G123.lcd

Acquired by : Admin
Sample Name : GB071P1
Sample ID : GYJ
Data File Name : G123.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Chromatogram
GB071P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G123.lcd



PeakTable

SPD-20A Ch2 254nm

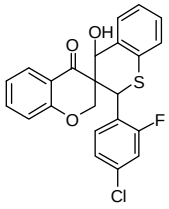
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.763	15951856	1441942	97.429	97.268
2	6.874	421002	40497	2.571	2.732
Total		16372858	1482438	100.000	100.000

Racemic **8g**

==== Shimadzu LCsolution Analysis Report ====

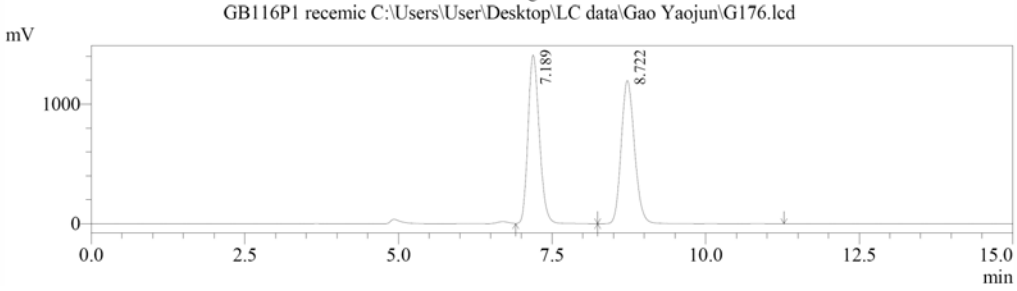
C:\Users\User\Desktop\LC data\Gao Yaojun\G176.lcd

Acquired by : Admin
Sample Name : GB116P1 recemic
Sample ID : GYJ
Data File Name : G176.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB116P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G176.lcd



PeakTable
SPD-20A Ch1 254nm

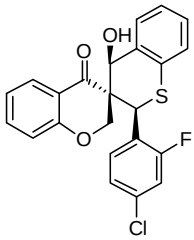
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.189	18097089	1410995	50.021	54.108
2	8.722	18081734	1196730	49.979	45.892
Total		36178824	2607725	100.000	100.000

Enantiomeric enriched **8g**

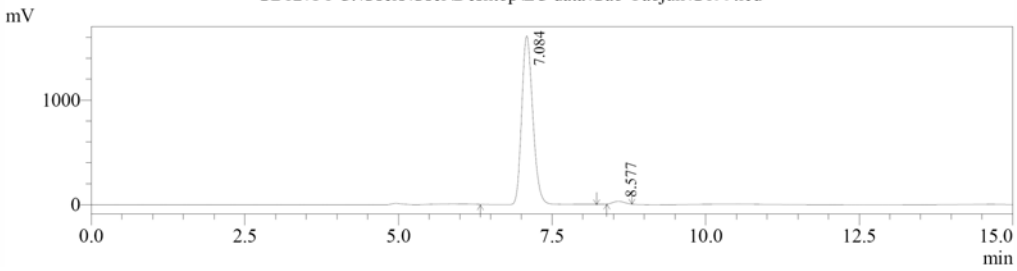
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G199.lcd

Acquired by : Admin
Sample Name : GB127P1
Sample ID : GYJ
Data File Name : G199.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB127P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G199.lcd



PeakTable
SPD-20A Ch1 254nm

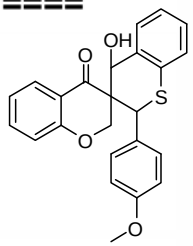
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.084	20546205	1613192	98.345	98.343
2	8.577	345845	27177	1.655	1.657
Total		20892051	1640370	100.000	100.000

Racemic **8h**

==== Shimadzu LCsolution Analysis Report ====

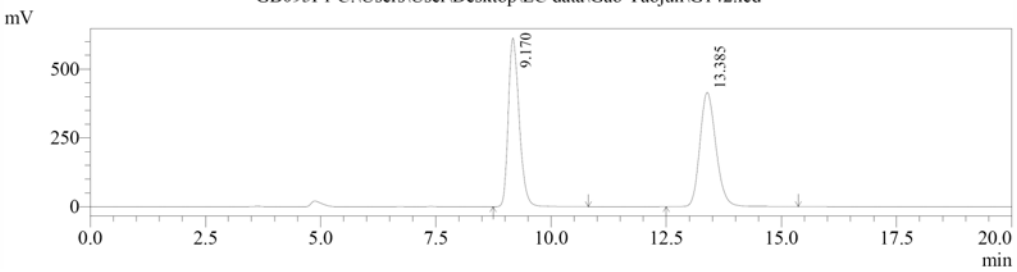
C:\Users\User\Desktop\LC data\Gao Yaojun\G142.lcd

Acquired by : Admin
Sample Name : GB093P1
Sample ID : GYJ
Data File Name : G142.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB093P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G142.lcd



PeakTable

SPD-20A Ch1 254nm

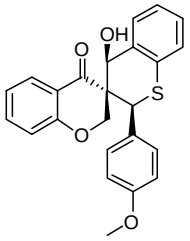
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.170	10066165	613019	50.017	59.659
2	13.385	10059176	414517	49.983	40.341
Total		20125341	1027537	100.000	100.000

Enantiomeric enriched **8h**

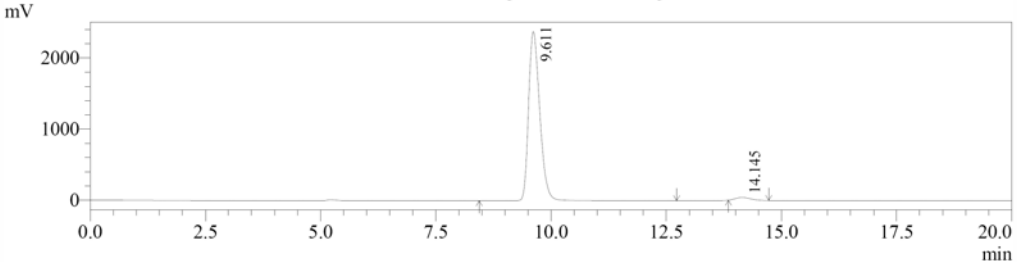
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G158.lcd

Acquired by : Admin
Sample Name : GB108P1
Sample ID : GYJ
Data File Name : G158.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Chromatogram
GB108P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G158.lcd



PeakTable

SPD-20A Ch1 254nm

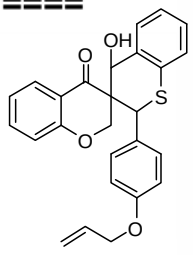
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.611	40437273	2378991	97.854	98.360
2	14.145	886668	39673	2.146	1.640
Total		41323940	2418665	100.000	100.000

Racemic **8i**

==== Shimadzu LCsolution Analysis Report ====

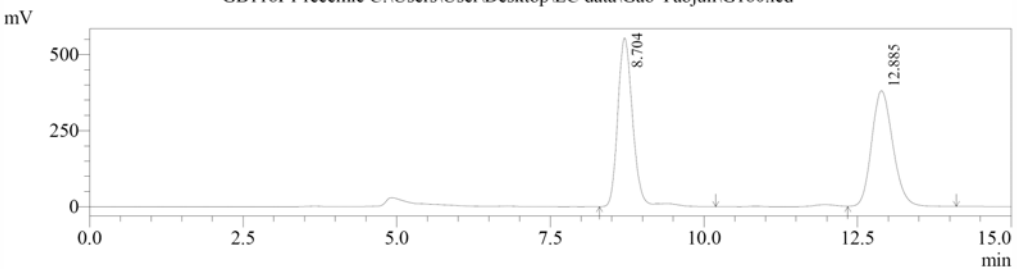
C:\Users\User\Desktop\LC data\Gao Yaojun\G180.lcd

Acquired by : Admin
Sample Name : GB118P1 recemic
Sample ID : GYJ
Data File Name : G180.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB118P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G180.lcd



PeakTable

SPD-20A Ch1 254nm

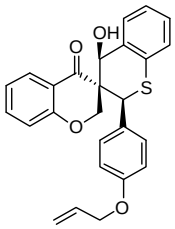
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.704	9379188	554469	50.550	59.275
2	12.885	9175259	380952	49.450	40.725
Total		18554447	935421	100.000	100.000

Enantiomeric enriched **8i**

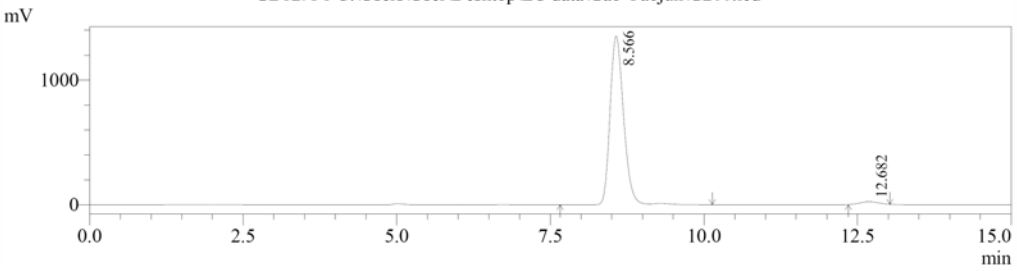
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G200.lcd

Acquired by : Admin
Sample Name : GB129P1
Sample ID : GYJ
Data File Name : G200.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB129P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G200.lcd



PeakTable

SPD-20A Ch1 254nm

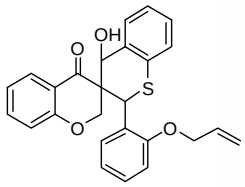
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.566	20854995	1355444	97.956	98.444
2	12.682	435269	21422	2.044	1.556
Total		21290265	1376866	100.000	100.000

Racemic **8j**

==== Shimadzu LCsolution Analysis Report ====

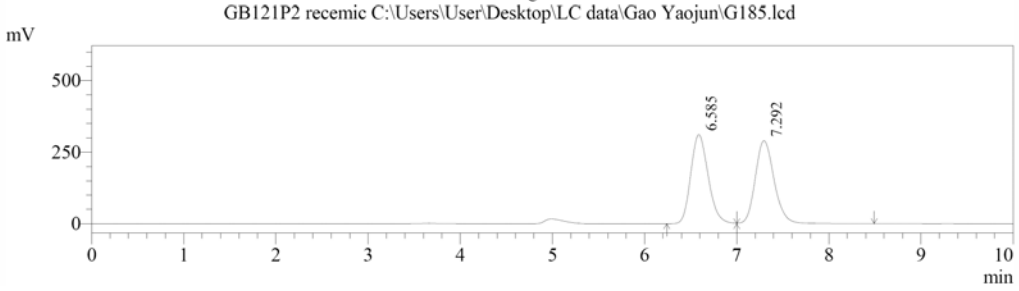
C:\Users\User\Desktop\LC data\Gao Yaojun\G185.lcd

Acquired by : Admin
Sample Name : GB121P2 recemic
Sample ID : GYJ
Data File Name : G185.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB121P2 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G185.lcd



PeakTable

SPD-20A Ch1 254nm

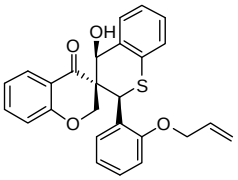
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.585	4089327	311322	49.779	51.792
2	7.292	4125693	289782	50.221	48.208
Total		8215020	601105	100.000	100.000

Enantiomeric enriched **8j**

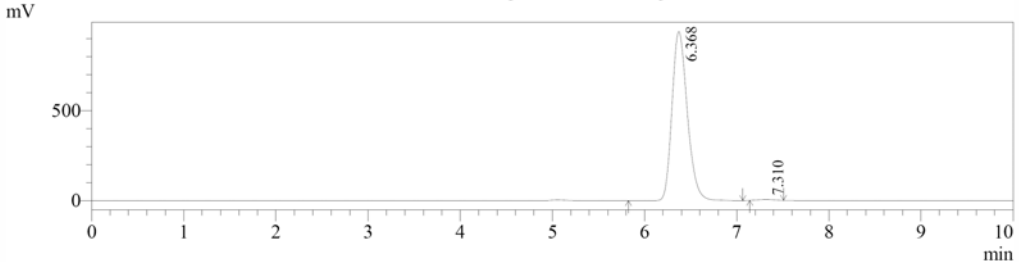
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G205.lcd

Acquired by : Admin
Sample Name : GB130P1
Sample ID : GYJ
Data File Name : G205.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB130P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G205.lcd



PeakTable

SPD-20A Ch1 254nm

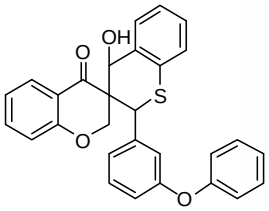
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.368	10872308	940744	99.462	99.447
2	7.310	58783	5227	0.538	0.553
Total		10931092	945971	100.000	100.000

Racemic **8k**

==== Shimadzu LCsolution Analysis Report ====

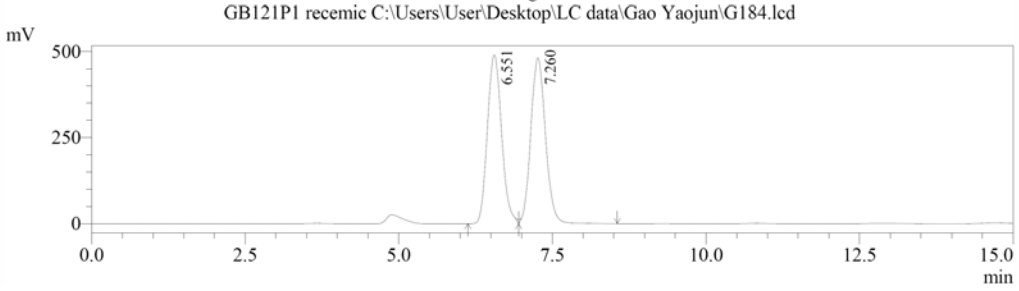
C:\Users\User\Desktop\LC data\Gao Yaojun\G184.lcd

Acquired by : Admin
Sample Name : GB121P1 recemic
Sample ID : GYJ
Data File Name : G184.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB121P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G184.lcd



PeakTable

SPD-20A Ch1 254nm

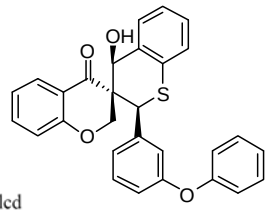
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.551	7897129	489936	49.591	50.444
2	7.260	8027488	481315	50.409	49.556
Total		15924618	971251	100.000	100.000

Enantiomeric enriched **8k**

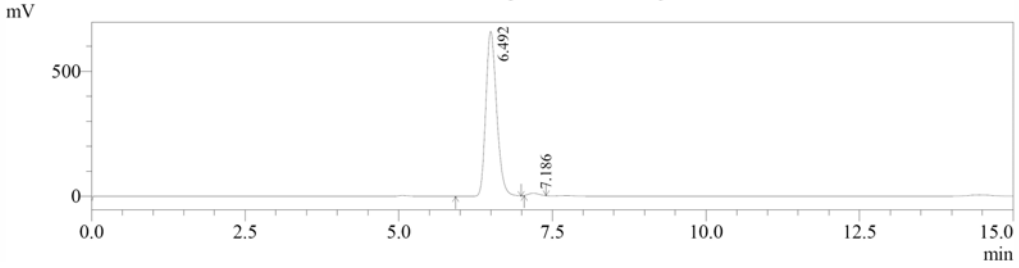
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G207.lcd

Acquired by : Admin
Sample Name : GB132P1
Sample ID : GYJ
Data File Name : G207.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB132P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G207.lcd



PeakTable

SPD-20A Ch1 254nm

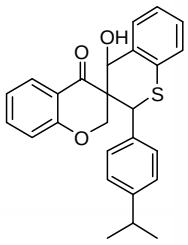
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.492	8175488	660598	98.723	98.602
2	7.186	105725	9368	1.277	1.398
Total		8281213	669965	100.000	100.000

Racemic **8l**

==== Shimadzu LCsolution Analysis Report ====

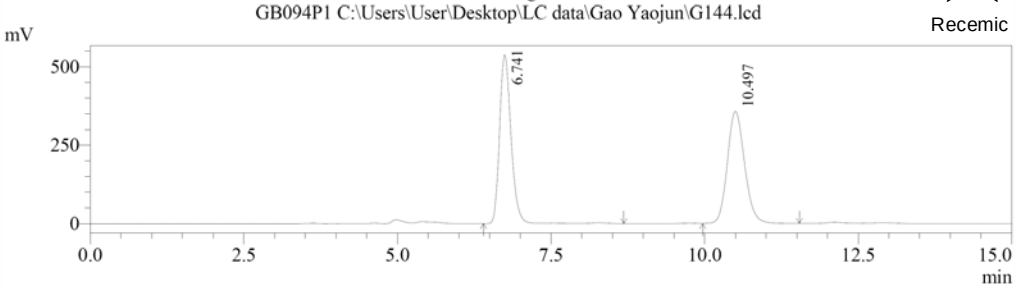
C:\Users\User\Desktop\LC data\Gao Yaojun\G144.lcd

Acquired by : Admin
Sample Name : GB094P1
Sample ID : GYJ
Data File Name : G144.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB094P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G144.lcd



PeakTable

SPD-20A Ch1 254nm

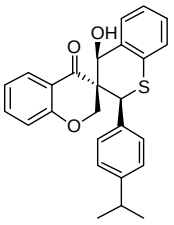
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.741	6959883	537213	49.954	60.040
2	10.497	6972665	357548	50.046	39.960
Total		13932547	894761	100.000	100.000

Enantiomeric enriched **8l**

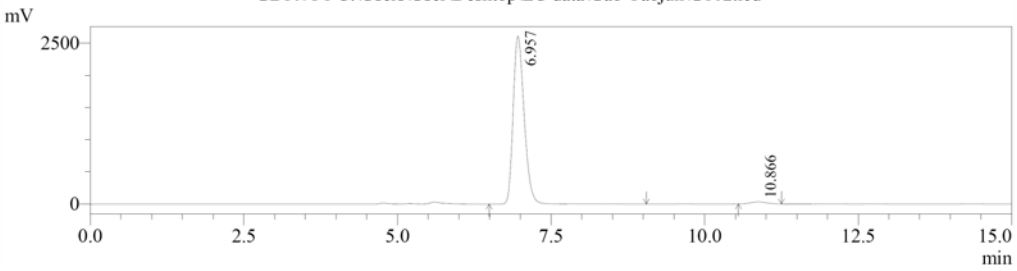
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G162.lcd

Acquired by : Admin
Sample Name : GB109P1
Sample ID : GYJ
Data File Name : G162.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Chromatogram
GB109P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G162.lcd



PeakTable

SPD-20A Ch1 254nm

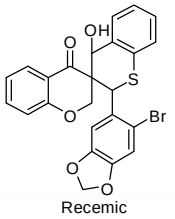
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.957	32658116	2612274	98.298	98.817
2	10.866	565553	31285	1.702	1.183
Total		33223668	2643559	100.000	100.000

Racemic **8m**

==== Shimadzu LCsolution Analysis Report ====

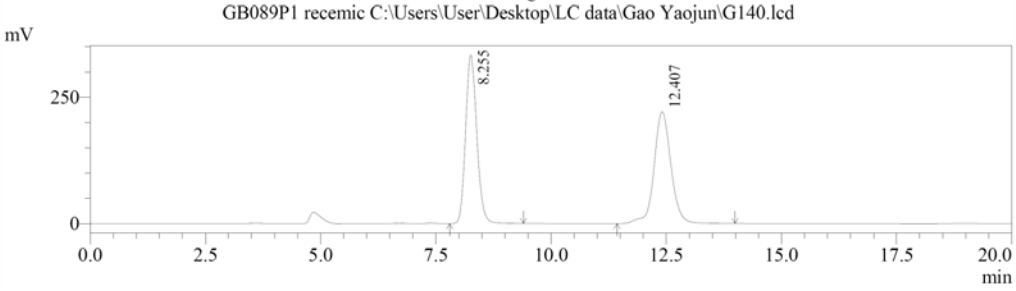
C:\Users\User\Desktop\LC data\Gao Yaojun\G140.lcd

Acquired by : Admin
Sample Name : GB089P1 recemic
Sample ID : GYJ
Data File Name : G140.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB089P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G140.lcd



PeakTable

SPD-20A Ch1 254nm

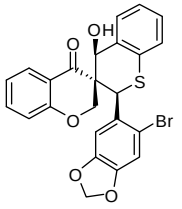
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.255	5633316	333763	50.020	60.128
2	12.407	5628906	221323	49.980	39.872
Total		11262222	555086	100.000	100.000

Enantiomeric enriched **8m**

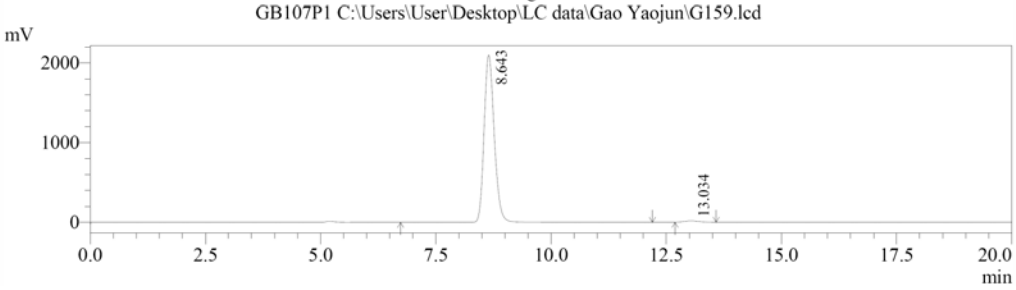
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G159.lcd

Acquired by : Admin
Sample Name : GB107P1
Sample ID : GYJ
Data File Name : G159.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB107P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G159.lcd



PeakTable

SPD-20A Ch1 254nm

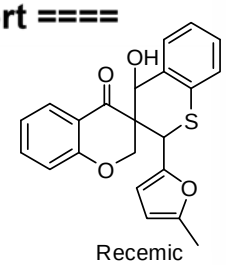
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.643	32412379	2101082	98.849	99.215
2	13.034	377511	16616	1.151	0.785
Total		32789890	2117697	100.000	100.000

Racemic **8n**

==== Shimadzu LCsolution Analysis Report ====

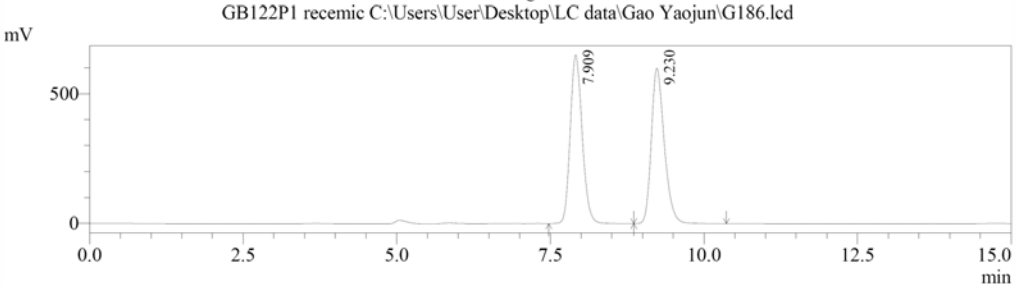
C:\Users\User\Desktop\LC data\Gao Yaojun\G186.lcd

Acquired by : Admin
Sample Name : GB122P1 recemic
Sample ID : GYJ
Data File Name : G186.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB122P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G186.lcd



PeakTable

SPD-20A Ch1 254nm

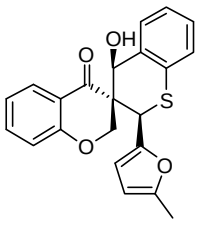
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.909	8813322	649707	50.097	52.047
2	9.230	8779030	598596	49.903	47.953
Total		17592353	1248303	100.000	100.000

Enantiomeric enriched **8n**

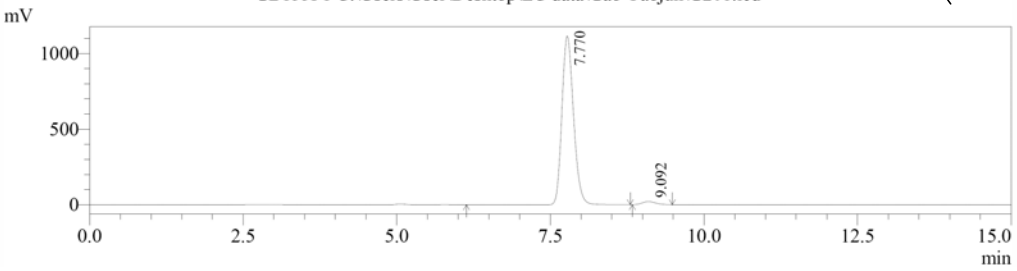
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G208.lcd

Acquired by : Admin
Sample Name : GB133P1
Sample ID : GYJ
Data File Name : G208.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB133P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G208.lcd



PeakTable

SPD-20A Ch1 254nm

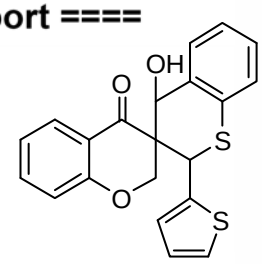
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.770	14711567	1116520	97.967	98.184
2	9.092	305284	20656	2.033	1.816
Total		15016851	1137176	100.000	100.000

Racemic **8o**

==== Shimadzu LCsolution Analysis Report ====

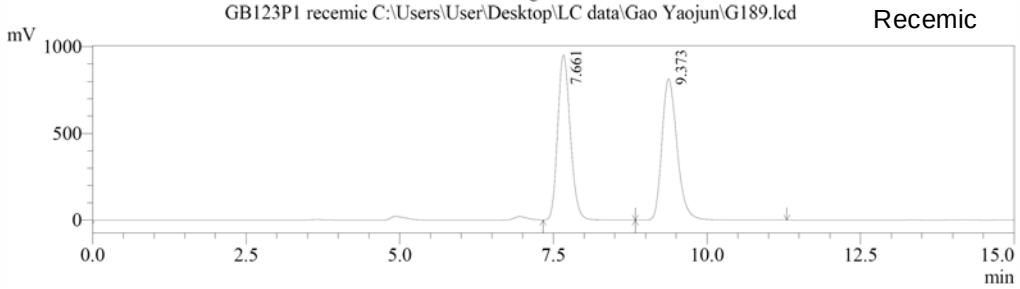
C:\Users\User\Desktop\LC data\Gao Yaojun\G189.lcd

Acquired by : Admin
Sample Name : GB123P1 recemic
Sample ID : GYJ
Data File Name : G189.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB123P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G189.lcd



PeakTable

SPD-20A Ch1 254nm

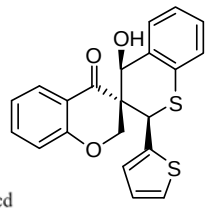
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.661	13628899	952684	49.351	53.895
2	9.373	13987432	814969	50.649	46.105
Total		27616330	1767653	100.000	100.000

Enantiomeric enriched **8o**

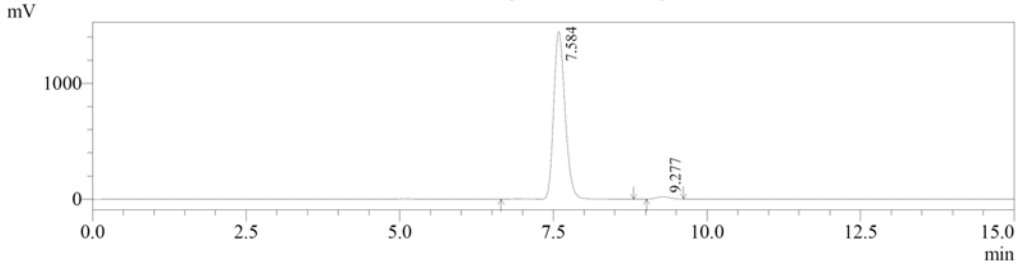
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G209.lcd

Acquired by : Admin
Sample Name : GB134P1
Sample ID : GYJ
Data File Name : G209.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB134P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G209.lcd



PeakTable

SPD-20A Ch1 254nm

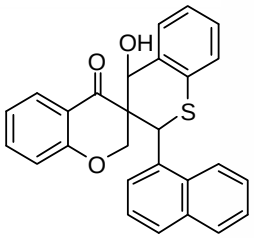
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.584	18817095	1448897	98.423	98.657
2	9.277	301475	19718	1.577	1.343
Total		19118570	1468615	100.000	100.000

Racemic **8p**

==== Shimadzu LCsolution Analysis Report ====

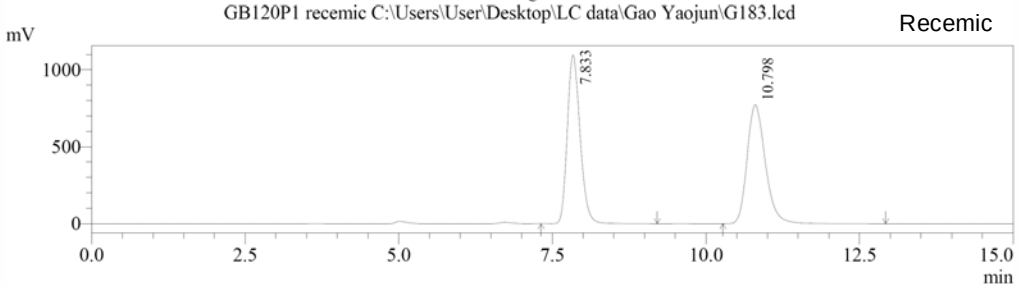
C:\Users\User\Desktop\LC data\Gao Yaojun\G183.lcd

Acquired by : Admin
Sample Name : GB120P1 recemic
Sample ID : GYJ
Data File Name : G183.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB120P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G183.lcd



PeakTable

SPD-20A Ch1 254nm

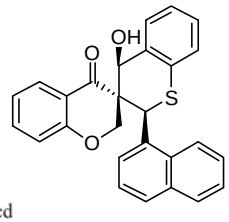
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.833	15880134	1096682	49.859	58.645
2	10.798	15969791	773361	50.141	41.355
Total		31849926	1870043	100.000	100.000

Enantiomeric enriched **8p**

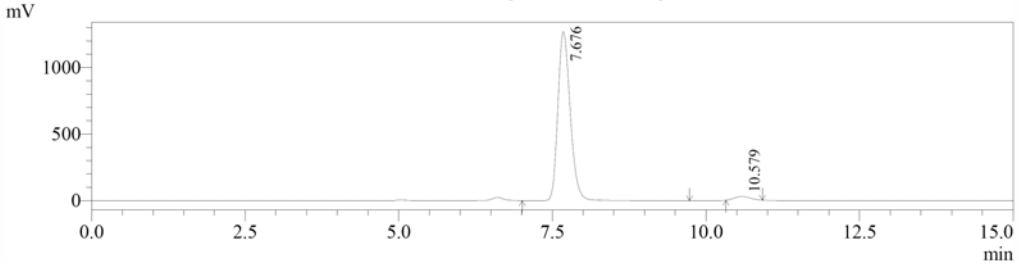
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G206.lcd

Acquired by : Admin
Sample Name : GB131P1
Sample ID : GYJ
Data File Name : G206.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB131P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G206.lcd



PeakTable

SPD-20A Ch1 254nm

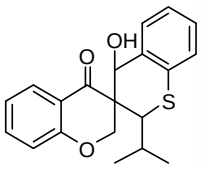
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.676	17875952	1271406	97.327	97.866
2	10.579	490942	27722	2.673	2.134
Total		18366893	1299128	100.000	100.000

Racemic **8q**

==== Shimadzu LCsolution Analysis Report ====

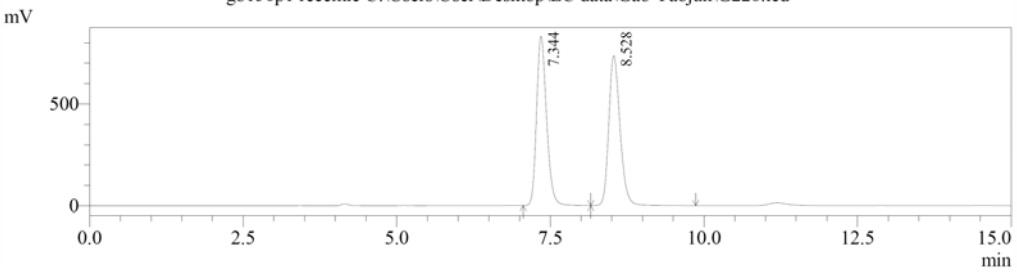
C:\Users\User\Desktop\LC data\Gao Yaojun\G228.lcd

Acquired by : Admin
Sample Name : gb156p1 recemic
Sample ID : gyj
Data File Name : G228.lcd
Method File Name : 5%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :Ib column with guard column,5%IPA



Recemic

Chromatogram
gb156p1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G228.lcd



PeakTable

SPD-20A Ch1 254nm

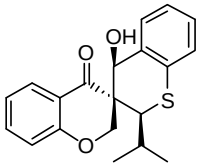
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.344	9403875	831047	50.045	52.978
2	8.528	9386815	737619	49.955	47.022
Total		18790690	1568666	100.000	100.000

Enantiomeric enriched **8q**

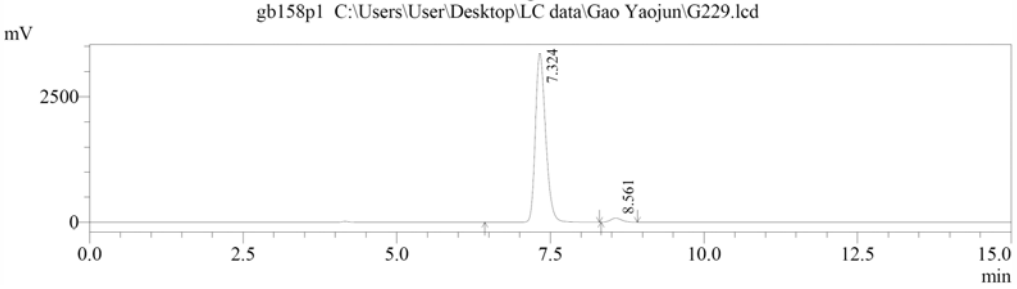
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G229.lcd

Acquired by : Admin
Sample Name : gb158p1
Sample ID : gyj
Data File Name : G229.lcd
Method File Name : 5%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :Ib column with guard column,5%IPA



Chromatogram
gb158p1 C:\Users\User\Desktop\LC data\Gao Yaojun\G229.lcd



PeakTable

SPD-20A Ch1 254nm

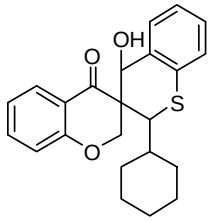
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.324	39151754	3354159	97.523	97.655
2	8.561	994452	80527	2.477	2.345
Total		40146207	3434686	100.000	100.000

Racemic **8r**

==== Shimadzu LCsolution Analysis Report ====

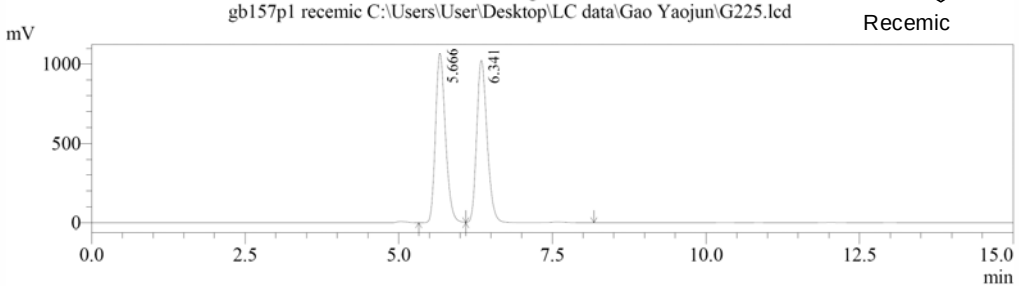
C:\Users\User\Desktop\LC data\Gao Yaojun\G225.lcd

Acquired by : Admin
Sample Name : gb157p1 recemic
Sample ID : gyj
Data File Name : G225.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :lc column with guard column,10%IPA



Recemic

Chromatogram
gb157p1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G225.lcd



PeakTable

SPD-20A Ch1 254nm

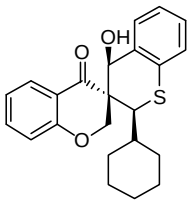
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.666	12015031	1065903	49.875	51.048
2	6.341	12075262	1022142	50.125	48.952
Total		24090293	2088045	100.000	100.000

Enantiomeric enriched **8r**

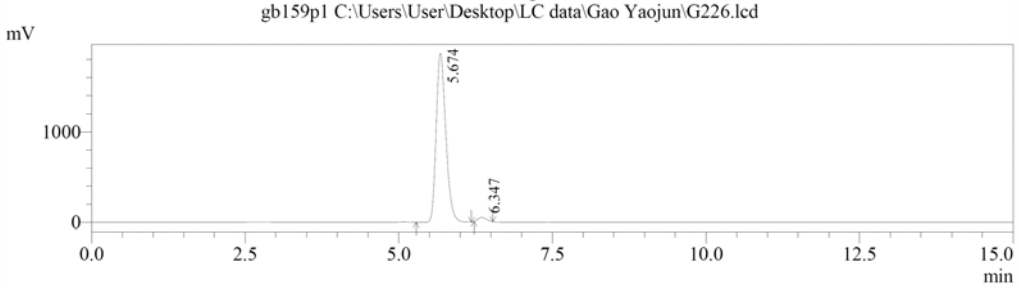
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G226.lcd

Acquired by : Admin
Sample Name : gb159p1
Sample ID : gyj
Data File Name : G226.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :lc column with guard column,10%IPA



Chromatogram
gb159p1 C:\Users\User\Desktop\LC data\Gao Yaojun\G226.lcd



PeakTable

SPD-20A Ch1 254nm

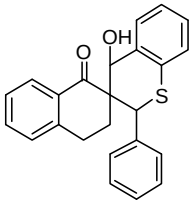
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.674	20394000	1865298	97.988	97.725
2	6.347	418738	43430	2.012	2.275
Total		20812738	1908728	100.000	100.000

Racemic **8s**

==== Shimadzu LCsolution Analysis Report ====

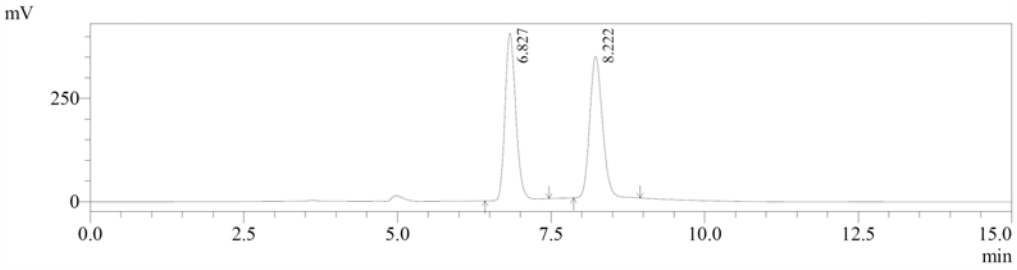
C:\Users\User\Desktop\LC data\Gao Yaojun\G125.lcd

Acquired by : Admin
Sample Name : GB072P1 recemic
Sample ID : GYJ
Data File Name : G125.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB072P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G125.lcd



PeakTable

SPD-20A Ch2 254nm

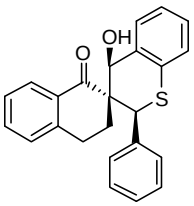
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.827	4861199	403598	49.522	54.102
2	8.222	4955041	342403	50.478	45.898
Total		9816240	746001	100.000	100.000

Enantiomeric enriched **8s**

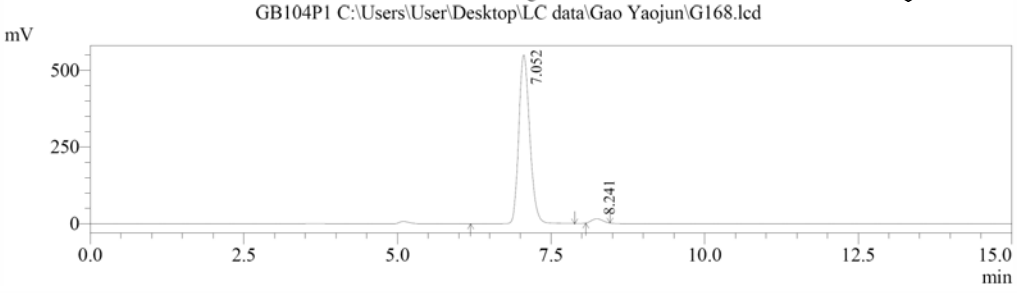
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G168.lcd

Acquired by : Admin
Sample Name : GB104P1
Sample ID : GYJ
Data File Name : G168.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB104P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G168.lcd



PeakTable

SPD-20A Ch1 254nm

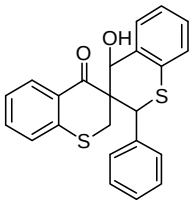
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.052	6903262	550548	97.600	97.537
2	8.241	169733	13904	2.400	2.463
Total		7072995	564452	100.000	100.000

Racemic **8t**

==== Shimadzu LCsolution Analysis Report ====

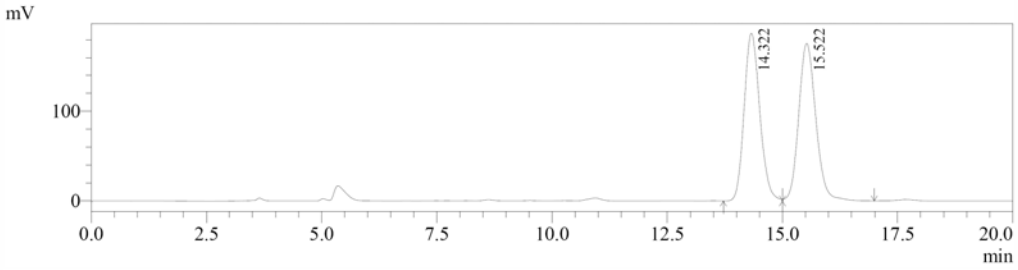
C:\Users\User\Desktop\LC data\Gao Yaojun\G129.lcd

Acquired by : Admin
Sample Name : GB073P1 recemic
Sample ID : GYJ
Data File Name : G129.lcd
Method File Name : 5%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 5%IPA



Recemic

Chromatogram
GB073P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G129.lcd



PeakTable

SPD-20A Ch2 254nm

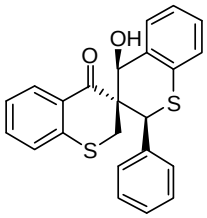
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.322	4425369	187294	49.270	51.568
2	15.522	4556462	175907	50.730	48.432
Total		8981830	363201	100.000	100.000

Enantiomeric enriched **8t**

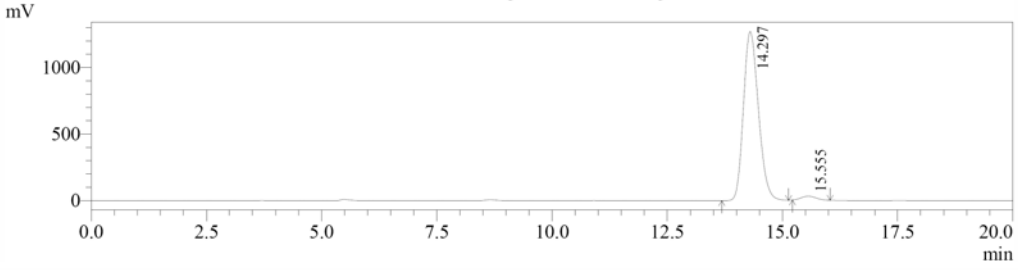
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G171.lcd

Acquired by : Admin
Sample Name : GB105P1
Sample ID : GYJ
Data File Name : G171.lcd
Method File Name : 5%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 5%IPA



Chromatogram
GB105P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G171.lcd



PeakTable

SPD-20A Ch1 254nm

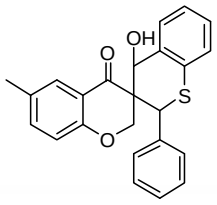
Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.297	28733699	1269317	97.769	97.728
2	15.555	655565	29510	2.231	2.272
Total		29389265	1298827	100.000	100.000

Racemic **8u**

==== Shimadzu LCsolution Analysis Report ====

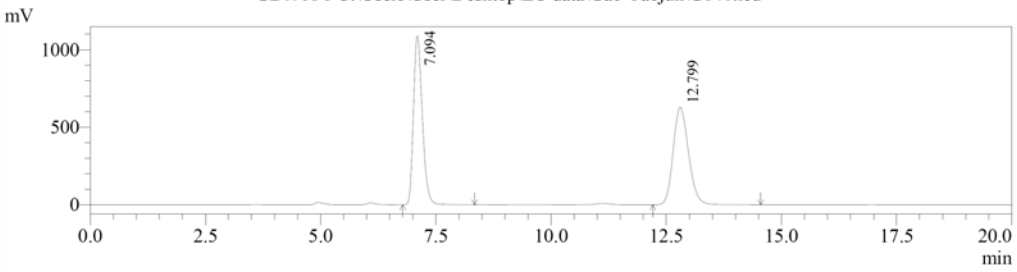
C:\Users\User\Desktop\LC data\Gao Yaojun\G146.lcd

Acquired by : Admin
Sample Name : GB095P1
Sample ID : GYJ
Data File Name : G146.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB095P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G146.lcd



PeakTable

SPD-20A Ch1 254nm

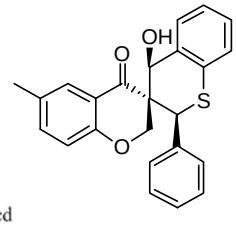
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.094	14505685	1088819	49.920	63.390
2	12.799	14552406	628834	50.080	36.610
Total		29058090	1717653	100.000	100.000

Enantiomeric enriched **8u**

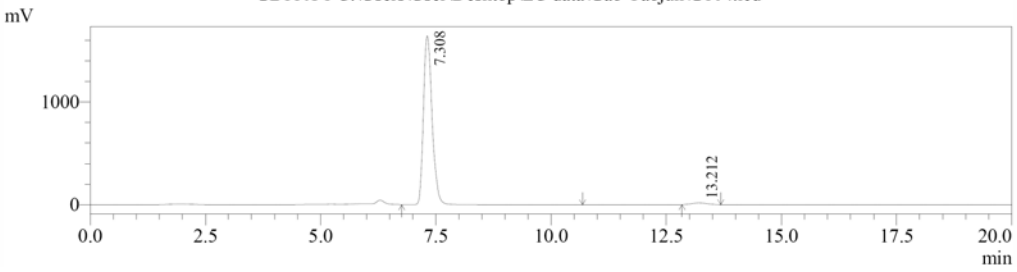
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G164.lcd

Acquired by : Admin
Sample Name : GB110P1
Sample ID : GYJ
Data File Name : G164.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Chromatogram
GB110P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G164.lcd



PeakTable

SPD-20A Ch1 254nm

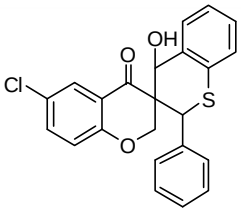
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.308	21835017	1642769	98.276	98.959
2	13.212	382941	17287	1.724	1.041
Total		22217958	1660056	100.000	100.000

Racemic **8v**

==== Shimadzu LCsolution Analysis Report ====

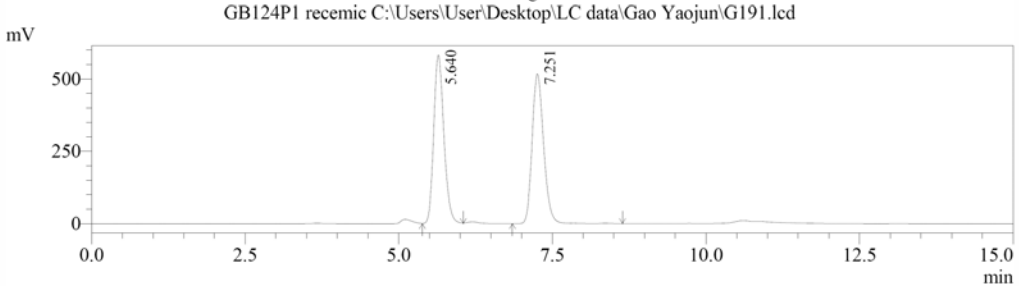
C:\Users\User\Desktop\LC data\Gao Yaojun\G191.lcd

Acquired by : Admin
Sample Name : GB124P1 recemic
Sample ID : GYJ
Data File Name : G191.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB124P1 recemic C:\Users\User\Desktop\LC data\Gao Yaojun\G191.lcd



PeakTable

SPD-20A Ch1 254nm

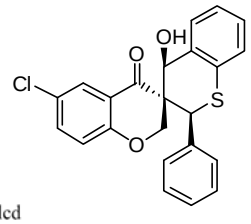
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.640	6728007	583183	49.941	52.967
2	7.251	6743981	517853	50.059	47.033
Total		13471988	1101036	100.000	100.000

Enantiomeric enriched **8v**

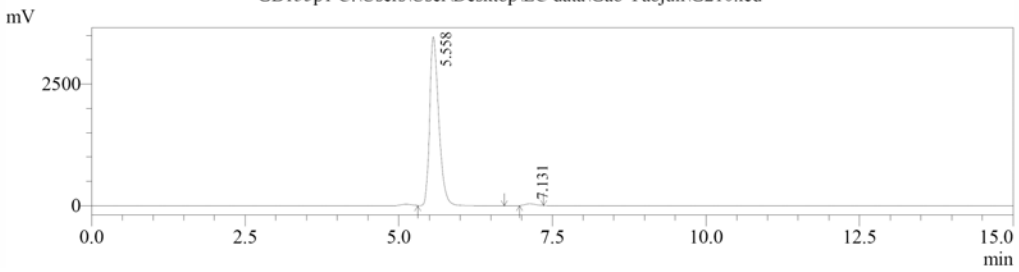
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G210.lcd

Acquired by : Admin
Sample Name : GB135p1
Sample ID : GYJ
Data File Name : G210.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB135p1 C:\Users\User\Desktop\LC data\Gao Yaojun\G210.lcd



PeakTable

SPD-20A Ch1 254nm

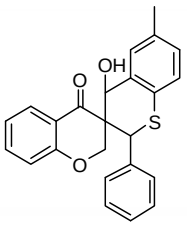
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.558	35795841	3468158	98.811	98.926
2	7.131	430688	37660	1.189	1.074
Total		36226528	3505818	100.000	100.000

Racemic **8w**

==== Shimadzu LCsolution Analysis Report ====

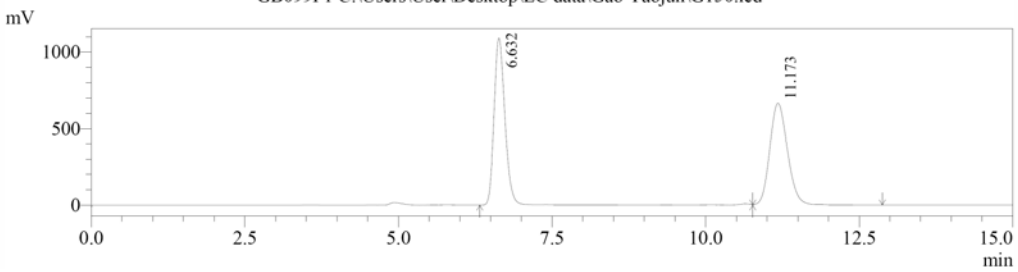
C:\Users\User\Desktop\LC data\Gao Yaojun\G150.lcd

Acquired by : Admin
Sample Name : GB099P1
Sample ID : GYJ
Data File Name : G150.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB099P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G150.lcd



PeakTable

SPD-20A Ch1 254nm

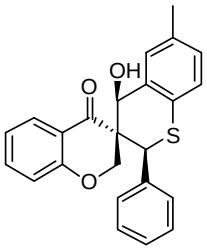
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.632	13555178	1092283	50.389	62.128
2	11.173	13345963	665834	49.611	37.872
Total		26901141	1758117	100.000	100.000

Enantiomeric enriched **8w**

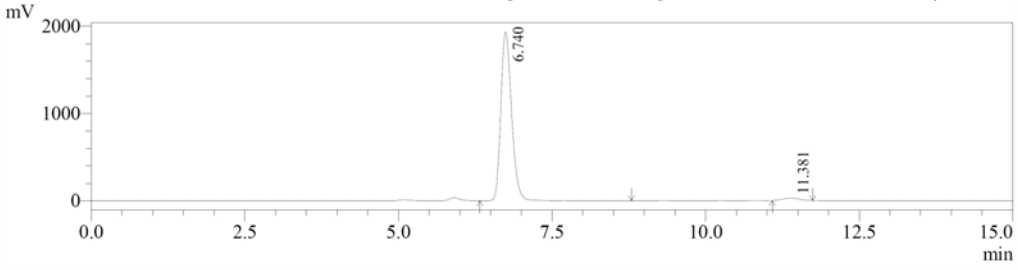
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G165.lcd

Acquired by : Admin
Sample Name : GB111P1
Sample ID : GYJ
Data File Name : G165.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB111P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G165.lcd



PeakTable

SPD-20A Ch1 254nm

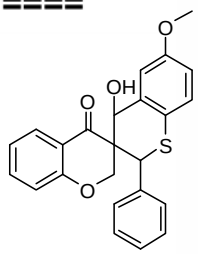
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.740	23571464	1935039	97.805	98.547
2	11.381	528952	28522	2.195	1.453
Total		24100416	1963561	100.000	100.000

Racemic **8x**

==== Shimadzu LCsolution Analysis Report ====

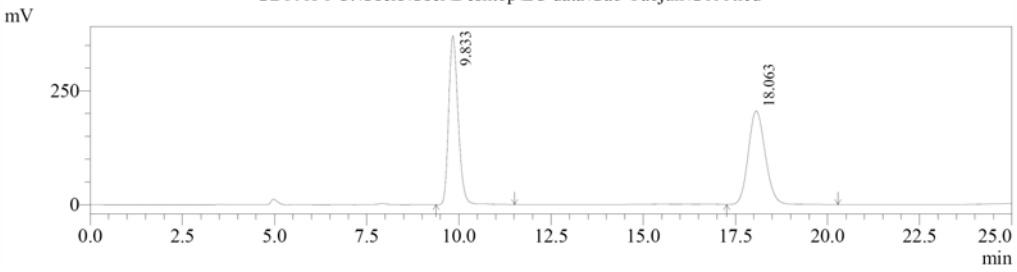
C:\Users\User\Desktop\LC data\Gao Yaojun\G155.lcd

Acquired by : Admin
Sample Name : GB101P1
Sample ID : GYJ
Data File Name : G155.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
GB101P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G155.lcd



PeakTable

SPD-20A Ch1 254nm

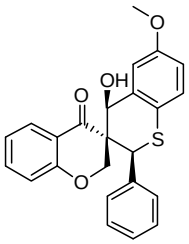
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.833	6617261	370747	50.089	64.308
2	18.063	6593621	205770	49.911	35.692
Total		13210882	576516	100.000	100.000

Enantiomeric enriched **8x**

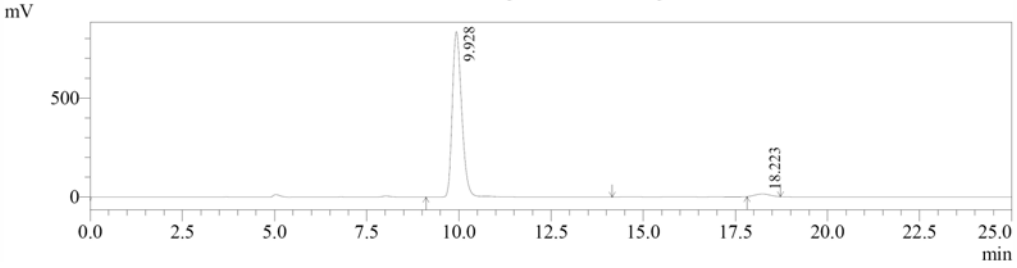
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G167.lcd

Acquired by : Admin
Sample Name : GB113P1
Sample ID : GYJ
Data File Name : G167.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB113P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G167.lcd



PeakTable

SPD-20A Ch1 254nm

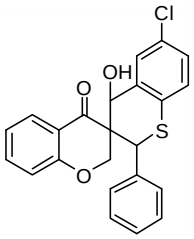
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.928	15019469	835523	97.514	98.376
2	18.223	382969	13792	2.486	1.624
Total		15402438	849316	100.000	100.000

Racemic **8y**

==== Shimadzu LCsolution Analysis Report ====

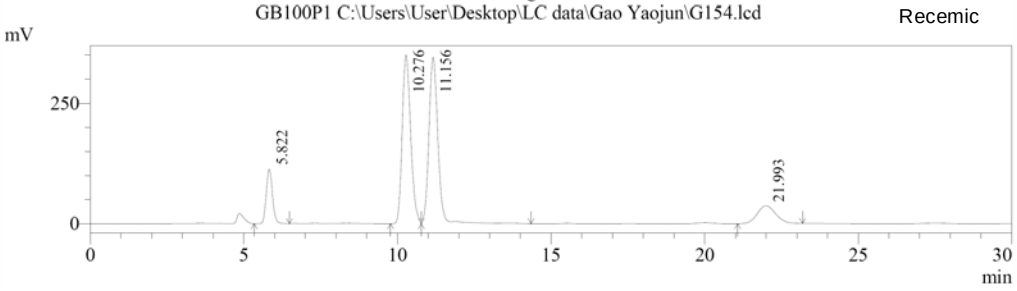
C:\Users\User\Desktop\LC data\Gao Yaojun\G154.lcd

Acquired by : Admin
Sample Name : GB100P1
Sample ID : GYJ
Data File Name : G154.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column, 10%IPA



Recemic

Chromatogram
GB100P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G154.lcd



PeakTable

SPD-20A Ch1 254nm

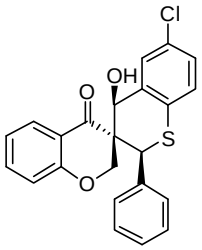
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.822	1585649	113212	9.385	13.389
2	10.276	6713879	350116	39.738	41.407
3	11.156	7054892	345344	41.756	40.843
4	21.993	1541037	36866	9.121	4.360
Total		16895457	845538	100.000	100.000

Enantiomeric enriched **8y**

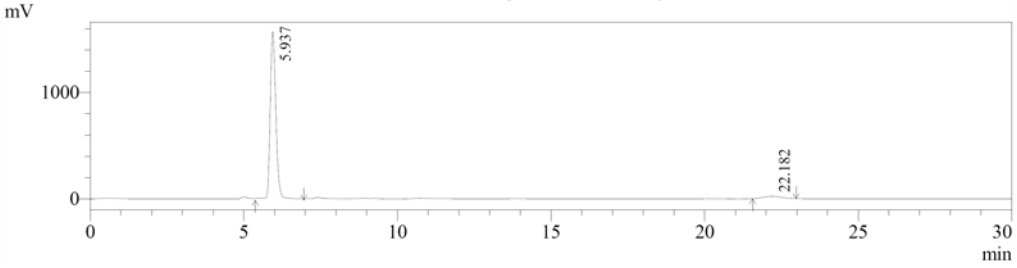
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G166.lcd

Acquired by : Admin
Sample Name : GB112P1
Sample ID : GYJ
Data File Name : G166.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GB112P1 C:\Users\User\Desktop\LC data\Gao Yaojun\G166.lcd



PeakTable

SPD-20A Ch1 254nm

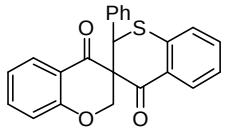
Peak#	Ret. Time	Area	Height	Area %	Height %
1	5.937	21158716	1587342	96.036	98.612
2	22.182	873298	22344	3.964	1.388
Total		22032014	1609686	100.000	100.000

Racemic **9**

==== Shimadzu LCsolution Analysis Report ====

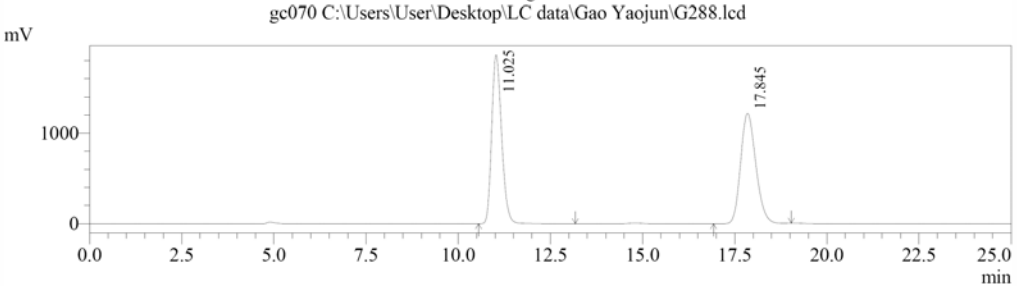
C:\Users\User\Desktop\LC data\Gao Yaojun\G288.lcd

Acquired by : Admin
Sample Name : gc070
Sample ID : gyj
Data File Name : G288.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Recemic

Chromatogram
gc070 C:\Users\User\Desktop\LC data\Gao Yaojun\G288.lcd



PeakTable

SPD-20A Ch1 254nm

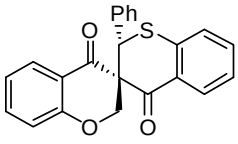
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.025	34847416	1864119	50.031	60.496
2	17.845	34803705	1217258	49.969	39.504
Total		69651121	3081377	100.000	100.000

Enantiomeric enriched **9**

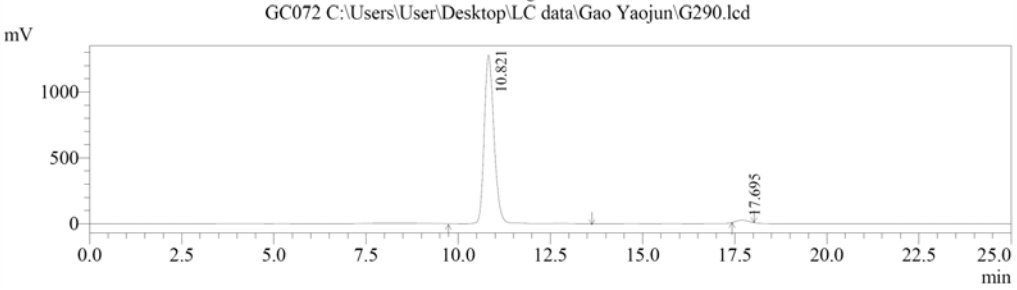
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Gao Yaojun\G290.lcd

Acquired by : Admin
Sample Name : GC072
Sample ID : gyj
Data File Name : G290.lcd
Method File Name : 10%IPA, 1ml-min, 60min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
GC072 C:\Users\User\Desktop\LC data\Gao Yaojun\G290.lcd



PeakTable

SPD-20A Ch1 254nm

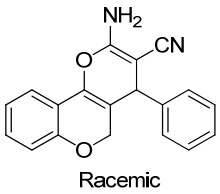
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.821	25242449	1286772	98.507	98.594
2	17.695	382594	18349	1.493	1.406
Total		25625043	1305121	100.000	100.000

Racemic **11**

==== Shimadzu LCsolution Analysis Report ====

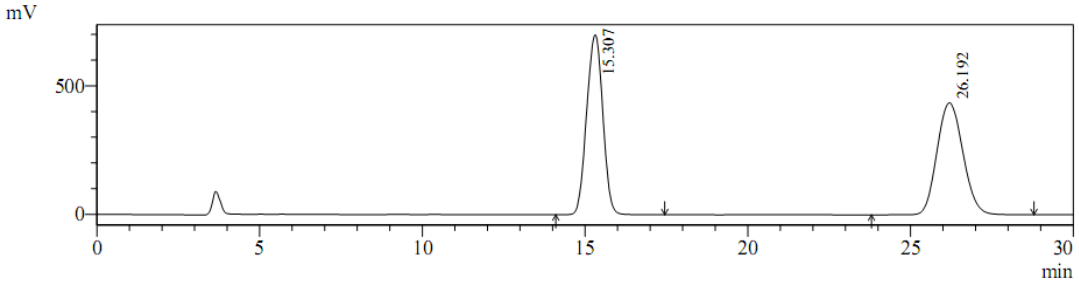
C:\Users\User\Desktop\LC data\Gao Yaojun\G063.lcd

Acquired by : Admin
Sample Name : GB025 P2
Sample ID : GYJ
Data File Name : G063.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column



Racemic

Chromatogram
GB025 P2 C:\Users\User\Desktop\LC data\Gao Yaojun\G063.lcd



PeakTable

SPD-20A Ch2 254nm

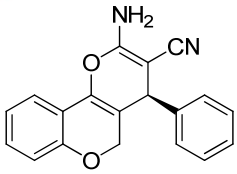
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.307	24124559	700094	49.993	61.630
2	26.192	24131021	435860	50.007	38.370
Total		48255580	1135954	100.000	100.000

Enantiomeric enriched **11**

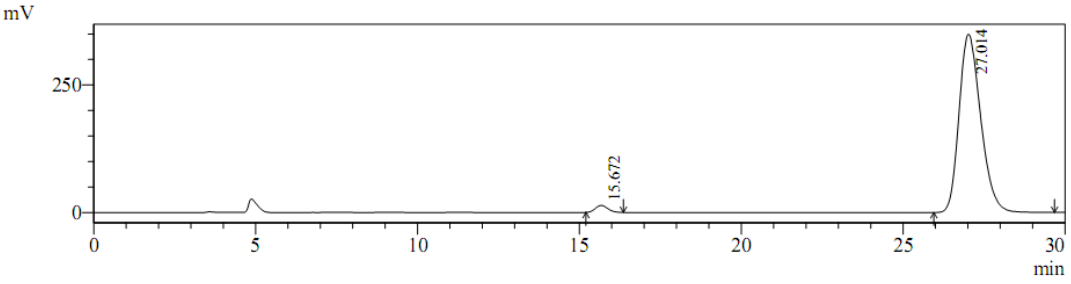
==== Shimadzu LCsolution Analysis Report ====

C:\Users\User\Desktop\LC data\Ren Qiao\rq052.lcd

Acquired by : Admin
Sample Name : R154-p2
Sample ID : rq
Data File Name : rq052.lcd
Method File Name : 10%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :IC column with guard column,10%IPA



Chromatogram
R154-p2 C:\Users\User\Desktop\LC data\Ren Qiao\rq052.lcd



PeakTable

SPD-20A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.672	370035	13809	2.222	3.800
2	27.014	16279568	349530	97.778	96.200
Total		16649603	363339	100.000	100.000