

**Expeditious assembly of fused dihydropyranones *via N-*
heterocyclic carbene-catalyzed tandem Michael
addition/lactonization**

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Supporting Information

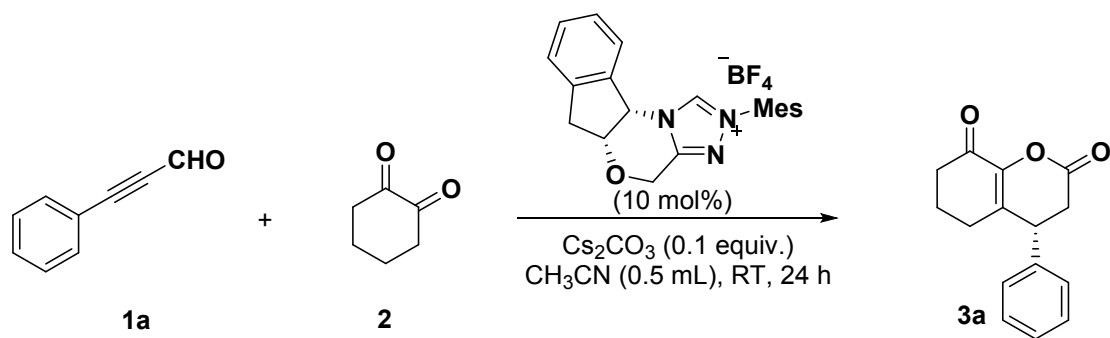
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1. General Information

Chemicals and solvents were purchased from commercial suppliers and used as received. ^1H and ^{13}C NMR spectra were recorded on a Bruker ACF300 (300 MHz), AVANCE III (400 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. 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. 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. Melting points were measured with a Tianjin automatic melting point ZRD-1 apparatus.

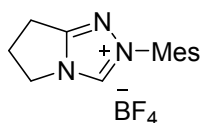
2. Representative Procedure for NHC-Catalyzed Reaction of Ynals with 1,2-Dione



Typical procedure for this reaction:

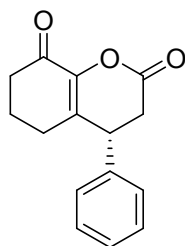
To a solution of 3-phenylpropionaldehyde **1a** (13.0 mg, 0.1 mmol) and 1,2-cyclohexanedione **2** (16.8 mg, 0.15 mmol) in CH₃CN (0.5 mL) was added catalyst **VI** (4.2 mg, 0.01 mmol) at room temperature, followed by adding the cesium carbonate (3.3 mg, 0.01 mmol). The resulting reaction mixture was kept stirring at room temperature for 24 h. The crude product was purified by silica gel flash chromatography, eluted by hexane/EtOAc = 1.5:1 to afford the desired product **3a** as colorless oil (20.8 mg, 86% yield).

Note: Racemic samples for the standard of chiral HPLC chromatograms were prepared with achiral NHC precursor **VIII** as catalyst.

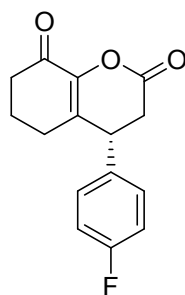


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3. Analytical Data of Fused Dihydropyranones



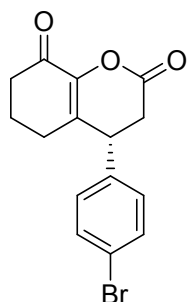
(R)-4-phenyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3a) 20.8 mg, 86% yield; Colorless oil; $^1\text{H NMR}$ (500 MHz, CDCl_3) δ (ppm) = 7.34 – 7.26 (m, 3H), 7.13 (d, $J = 7.1$ Hz, 2H), 3.75 (dd, $J = 7.5, 4.0$ Hz, 1H), 3.00 (dd, $J = 16.2, 7.6$ Hz, 1H), 2.82 (dd, $J = 16.2, 4.0$ Hz, 1H), 2.54 (t, $J = 6.7$ Hz, 2H), 2.47 – 2.26 (m, 2H), 2.08 – 1.86 (m, 2H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3) $\delta = 190.06, 165.48, 143.40, 138.37, 136.78, 129.35, 128.11, 127.06, 42.61, 37.56, 35.88, 27.61, 21.86$; HRMS (ESI): Exact mass calculated for $\text{C}_{15}\text{H}_{15}\text{O}_3$ $[\text{M}+\text{H}]^+$ 243.1016, found 243.1015; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, $\lambda = 254$ nm): t_{R} (minor) = 26.1 min, t_{R} (major) = 36.1 min, $ee = 98\%$; $[\alpha]_{\text{D}}^{25} = -157.3$ ($c = 0.83$ in DCM).



(R)-4-(4-fluorophenyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3b) 20.9 mg, 80% yield; White solid; mp 104–107 °C; $^1\text{H NMR}$ (300 MHz, CDCl_3) $\delta = 7.18 - 6.97$ (m, 4H), 3.73 (dd, $J = 7.4, 3.9$ Hz, 1H), 3.01 (dd, $J = 16.1, 7.5$ Hz, 1H), 2.82 (dd, $J = 16.1, 4.0$ Hz, 1H), 2.58 (t, $J = 6.7$ Hz, 2H), 2.49 – 2.24 (m, 2H), 2.13 – 1.88 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 189.91, 165.15, 162.46$ (d, $^1J_{\text{C-F}} = 246.3$ Hz), 143.57, 136.20, 134.17, 128.82 (d, $^3J_{\text{C-F}} = 8.1$ Hz), 116.46 (d, $^2J_{\text{C-F}} = 21.5$ Hz), 42.09, 37.65, 36.10, 27.69, 21.98; HRMS (ESI): Exact mass calculated for $\text{C}_{15}\text{H}_{14}\text{O}_3\text{F}$ $[\text{M}+\text{H}]^+$ 261.0921, found

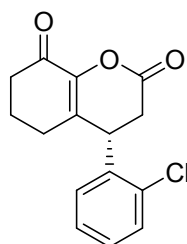
261.0929; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): t_R

(minor) = 29.9 min, t_R (major) = 34.2 min, ee = 96%; $[\alpha]_D^{25}$ = -174.4 (c = 0.78 in DCM).



(R)-4-(4-bromophenyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3c) 24.4 mg, 76% yield;

White solid; mp 169–172 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.48 (d, J = 8.2 Hz, 2H), 7.03 (d, J = 8.4 Hz, 2H), 3.71 (dd, J = 7.4, 3.9 Hz, 1H), 3.01 (dd, J = 16.1, 7.6 Hz, 1H), 2.81 (dd, J = 16.2, 4.0 Hz, 1H), 2.57 (t, J = 6.7 Hz, 2H), 2.44 – 2.27 (m, 2H), 2.06 – 1.94 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 189.85, 164.98, 143.69, 137.45, 135.69, 132.65, 128.85, 122.30, 42.29, 37.63, 35.82, 27.69, 21.96; HRMS (ESI): Exact mass calculated for $\text{C}_{15}\text{H}_{13}\text{O}_3\text{BrNa}$ $[\text{M}+\text{Na}]^+$ 242.9940, found 242.9927; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): t_R (minor) = 30.6 min, t_R (major) = 36.6 min, ee = 92%; $[\alpha]_D^{25}$ = -144.8 (c = 0.84 in DCM).



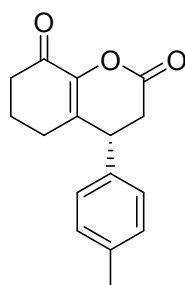
(S)-4-(2-chlorophenyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3d) 22.9 mg, 83% yield; Colorless oil; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm) = 7.47 – 7.32 (m, 1H), 7.29 – 7.13 (m, 2H), 7.00 (dd, J = 5.9, 3.4 Hz, 1H), 4.25 (dd, J = 7.2, 4.3 Hz, 1H), 2.89 (qd, J = 16.3, 5.9 Hz, 2H), 2.69 – 2.50 (m, 2H), 2.48 – 2.15 (m, 2H), 2.13 – 1.85 (m, 2H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ = 189.88, 165.05,

144.47, 135.56, 135.26, 133.71, 130.54, 129.60, 128.00, 127.76, 39.21, 37.70, 34.42, 27.63, 21.98;

HRMS (ESI): Exact mass calculated for C₁₅H₁₄O₃Cl [M+K]⁺ 277.0626, found 277.0615; HPLC

(Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): *t*_R (minor) = 30.5 min,

*t*_R (major) = 52.6 min, *ee* = 98%; [α]²⁵_D = +19.5 (*c* = 1.12 in DCM).



(R)-4-*p*-tolyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3e) 20.3 mg, 79% yield; Colorless

oil; ¹H NMR (500 MHz, CDCl₃) δ (ppm) = 7.15 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 8.0 Hz, 2H), 3.70 (dd,

J = 7.5, 4.1 Hz, 1H), 2.99 (dd, *J* = 16.1, 7.6 Hz, 1H), 2.82 (dd, *J* = 16.1, 4.1 Hz, 1H), 2.56 (t, *J* = 6.8 Hz,

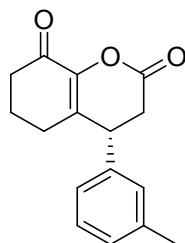
2H), 2.46 – 2.29 (m, 5H), 2.08 – 1.91 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ (ppm) = 190.09, 165.48,

143.44, 138.10, 136.91, 135.37, 130.10, 127.01, 42.44, 37.67, 36.07, 27.71, 21.98, 21.01; HRMS (ESI):

Exact mass calculated for C₁₆H₁₇O₃ [M+H]⁺ 257.1172, found 257.1171; HPLC (Chiralpak AS-H, *i*-

propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): *t*_R (minor) = 23.6 min, *t*_R (major) = 34.5

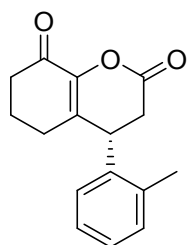
min, *ee* = 97%; [α]²⁵_D = -221.7 (*c* = 1.08 in DCM).



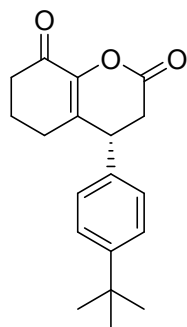
(R)-4-*m*-tolyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3f) 20.7 mg, 81% yield; Colorless

oil; ¹H NMR (500 MHz, CDCl₃) δ (ppm) = 7.23 (t, *J* = 7.9 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 1H), 6.93 (br,

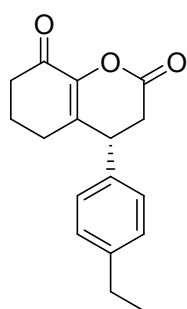
2H), 3.69 (dd, $J = 7.5, 4.0$ Hz, 1H), 2.99 (dd, $J = 16.2, 7.7$ Hz, 1H), 2.84 (dd, $J = 16.1, 4.1$ Hz, 1H), 2.61 – 2.53 (m, 2H), 2.47 – 2.35 (m, 2H), 2.33 (s, 3H), 2.08 – 1.90 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 190.05, 165.43, 143.49, 139.30, 138.43, 136.70, 129.31, 129.01, 127.75, 124.19, 42.80, 37.68, 35.98, 27.74, 21.99, 21.40; HRMS (ESI): Exact mass calculated for $\text{C}_{16}\text{H}_{17}\text{O}_3$ $[\text{M}+\text{H}]^+$ 257.1172, found 257.1179; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, $\lambda = 254$ nm): t_{R} (minor) = 26.5 min, t_{R} (major) = 33.4 min, $ee = 98\%$; $[\alpha]_{\text{D}}^{25} = -119.8$ ($c = 1.11$ in DCM).



(*R*)-4-*o*-tolyl-3,4,6,7-tetrahydro-2*H*-chromene-2,8(5*H*)-dione (3g) 18.3 mg, 71% yield; Colorless oil; ^1H NMR (500 MHz, CDCl_3) δ (ppm) = 7.25 – 7.15 (m, 3H), 6.97 (d, $J = 7.4$ Hz, 1H), 4.02 (dd, $J = 7.5$ Hz, 4.2, 1H), 3.04 – 2.94 (m, 1H), 2.81 – 2.71 (m, 1H), 2.65 – 2.53 (m, 2H), 2.43 – 2.25 (m, 5H), 2.10 – 1.94 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 189.91, 165.28, 144.27, 136.91, 136.17, 135.52, 131.36, 128.08, 127.23, 126.01, 38.63, 37.73, 35.10, 27.78, 22.03, 19.49; HRMS (ESI): Exact mass calculated for $\text{C}_{16}\text{H}_{17}\text{O}_3$ $[\text{M}+\text{H}]^+$ 257.1172, found 257.1178; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, $\lambda = 254$ nm): t_{R} (minor) = 33.1 min, t_{R} (major) = 47.4 min, $ee = 99\%$; $[\alpha]_{\text{D}}^{25} = -154.5$ ($c = 0.76$ in DCM).

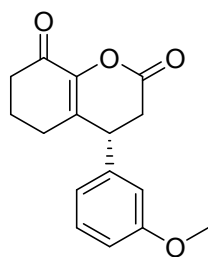


(R)-4-(4-*tert*-butylphenyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3h) 22.7 mg, 76% yield; White solid; mp 143–145 °C; ¹H NMR (300 MHz, CDCl₃) δ (ppm) = 7.39 – 7.30 (m, 2H), 7.06 (d, *J* = 8.3 Hz, 2H), 3.71 (dd, *J* = 7.3, 4.0 Hz, 1H), 2.99 (dd, *J* = 16.1, 7.5 Hz, 1H), 2.83 (dd, *J* = 16.1, 4.0 Hz, 1H), 2.57 (t, *J* = 6.7 Hz, 2H), 2.39 (dd, *J* = 9.0, 5.4 Hz, 2H), 2.12 – 1.87 (m, 2H), 1.30 (s, 9H); ¹³C NMR (75 MHz, CDCl₃) (ppm) δ = 190.10, 165.56, 151.25, 143.46, 136.97, 135.24, 126.78, 126.35, 42.34, 37.67, 36.05, 34.53, 31.21, 27.76, 21.98; HRMS (ESI): Exact mass calculated for C₁₉H₂₃O₃ [M+H]⁺ 299.1642, found 299.1649; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): *t*_R (minor) = 17.5 min, *t*_R (major) = 23.8 min, *ee* = 98%; [α]_D²⁵ = -218.7 (*c* = 1.39 in DCM).

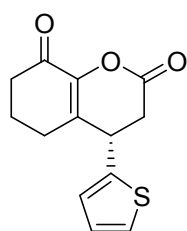


(R)-4-(4-ethylphenyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3i) 23.8 mg, 88% yield; Colorless oil; ¹H NMR (300 MHz, CDCl₃) δ (ppm) = 7.17 (d, *J* = 8.1 Hz, 2H), 7.12 – 7.01 (m, 2H), 3.70 (dd, *J* = 7.3, 4.1 Hz, 1H), 2.99 (dd, *J* = 16.1, 7.5 Hz, 1H), 2.83 (dd, *J* = 16.1, 4.2 Hz, 1H), 2.74 – 2.50 (m, 4H), 2.38 (dd, *J* = 11.0, 5.2 Hz, 2H), 2.14 – 1.89 (m, 2H), 1.22 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz,

CDCl₃) δ (ppm) = 190.04, 165.51, 144.35, 143.44, 136.93, 135.59, 128.89, 127.07, 42.46, 37.67, 36.07, 28.39, 27.73, 21.98, 15.33; HRMS (ESI): Exact mass calculated for C₁₇H₁₉O₃ [M+H]⁺ 271.1329, found 271.1333; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): t_R (minor) = 23.7 min, t_R (major) = 33.8 min, *ee* = 98%; [α]_D²⁵ = -164.8 (*c* = 1.01 in DCM).

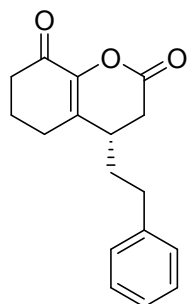


(R)-4-(3-methoxyphenyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3j) 21.8 mg, 80% yield; Yellow solid; mp 133–136 °C; ¹H NMR (300 MHz, CDCl₃) δ (ppm) = 7.27 (dd, *J* = 9.5, 6.4 Hz, 1H), 6.84 (dd, *J* = 8.1, 2.2 Hz, 1H), 6.72 (d, *J* = 7.6 Hz, 1H), 6.69 – 6.63 (m, 1H), 3.79 (s, 3H), 3.70 (dd, *J* = 7.4, 4.3 Hz, 1H), 3.00 (dd, *J* = 16.2, 7.5 Hz, 1H), 2.84 (dd, *J* = 16.1, 4.3 Hz, 1H), 2.57 (t, *J* = 6.7 Hz, 2H), 2.45 – 2.33 (m, 2H), 2.12 – 1.87 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ (ppm) = 190.04, 165.35, 160.28, 143.56, 139.93, 136.41, 130.57, 119.26, 113.34, 113.10, 55.27, 42.78, 37.67, 35.91, 27.70, 21.97; HRMS (ESI): Exact mass calculated for C₁₆H₁₇O₄ [M+H]⁺ 273.1121, found 273.1126; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): t_R (minor) = 48.7 min, t_R (major) = 55.9 min, *ee* = 94%; [α]_D²⁵ = -145.4 (*c* = 1.36 in DCM).

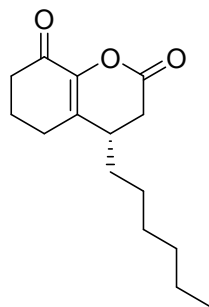


(S)-4-(thiophen-2-yl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3k) 18.3 mg, 74% yield;

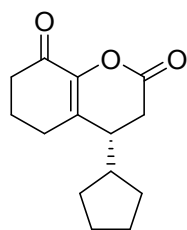
White solid; mp 156–157 °C; ¹H NMR (300 MHz, CDCl₃) δ (ppm) = 7.26 – 7.22 (m, 1H), 6.97 (dd, *J* = 5.1, 3.5 Hz, 1H), 6.92 – 6.85 (m, 1H), 4.11 – 3.87 (m, 1H), 3.14 – 2.89 (m, 2H), 2.66 – 2.44 (m, 4H), 2.17 – 1.93 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ (ppm) = 190.01, 165.00, 143.12, 140.52, 136.10, 127.50, 125.36, 125.33, 37.59, 36.47, 27.62, 21.99; Exact mass calculated for C₁₃H₁₃O₃S [M+H]⁺ 249.0580, found 249.0589; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): *t*_R (minor) = 32.4 min, *t*_R (major) = 49.2 min, *ee* = 92%; [α]_D²⁵ = -168.1 (*c* = 1.18 in DCM).



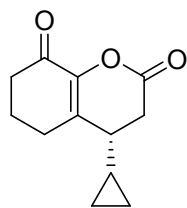
(S)-4-phenethyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (31) 22.3 mg, 83% yield; Colorless oil; ¹H NMR (500 MHz, CDCl₃) δ (ppm) = 7.30 (t, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.15 (d, *J* = 7.4 Hz, 2H), 2.83 – 2.66 (m, 3H), 2.61 (ddd, *J* = 13.9, 9.2, 7.4 Hz, 1H), 2.55 – 2.37 (m, 5H), 2.09 – 1.89 (m, 3H), 1.79 – 1.68 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 189.69, 166.16, 143.19, 140.14, 138.68, 128.66, 128.21, 126.42, 37.58, 35.79, 33.15, 32.64, 32.54, 27.82, 22.07; Exact mass calculated for C₁₇H₁₉O₃ [M+H]⁺ 271.1329, found 271.1328; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): *t*_R (minor) = 24.9 min, *t*_R (major) = 34.1 min, *ee* = 97%; [α]_D²⁵ = -113.0 (*c* = 0.74 in DCM).



(S)-4-hexyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3m) 20.7 mg, 83% yield; Yellow oil; ^1H NMR (300 MHz, CDCl_3) δ (ppm) = 2.66 (d, J = 4.7 Hz, 2H), 2.61 – 2.37 (m, 4H), 2.14 – 1.96 (m, 2H), 1.82 – 1.51 (m, 2H), 1.49 – 1.14 (m, 9H), 0.87 (t, J = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ (ppm) = 189.83, 166.43, 143.02, 139.29, 37.59, 36.51, 32.89, 31.91, 31.51, 29.09, 27.99, 26.53, 22.51, 22.10, 13.98; Exact mass calculated for $\text{C}_{15}\text{H}_{23}\text{O}_3$ $[\text{M}+\text{H}]^+$ 251.1642, found 251.1640; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): t_{R} (minor) = 12.5 min, t_{R} (major) = 14.4 min, ee = 98%; $[\alpha]_{\text{D}}^{25}$ = -37.8 (c = 0.55 in DCM).

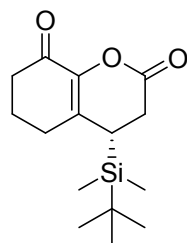


(R)-4-cyclopentyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3n) 15.9 mg, 70% yield; Colorless oil; ^1H NMR (500 MHz, CDCl_3) δ (ppm) = 2.74 (dd, J = 16.0, 2.0 Hz, 1H), 2.62 (dt, J = 10.8, 5.6 Hz, 2H), 2.57 – 2.43 (m, 3H), 2.28 (t, J = 6.4 Hz, 1H), 2.11 – 1.98 (m, 2H), 1.96 – 1.81 (m, 2H), 1.80 – 1.45 (m, 7H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 189.83, 166.83, 143.36, 139.95, 43.04, 41.57, 37.70, 32.83, 31.24, 30.31, 29.38, 25.07, 24.10, 22.32; Exact mass calculated for $\text{C}_{14}\text{H}_{19}\text{O}_3$ $[\text{M}+\text{H}]^+$ 235.1329, found 235.1335; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 254 nm): t_{R} (minor) = 18.3 min, t_{R} (major) = 26.6 min, ee = 98%; $[\alpha]_{\text{D}}^{25}$ = -56.3 (c = 1.14 in DCM).



(R)-4-cyclopropyl-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3o) 14.7 mg, 71% yield;

Colorless oil; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm) = 2.82 – 2.64 (m, 3H), 2.56 – 2.44 (m, 3H), 2.15 – 1.99 (m, 2H), 1.85 – 1.69 (m, 1H), 0.93 – 0.49 (m, 3H), 0.38 (td, $J = 9.6, 4.6$ Hz, 1H), 0.15 (td, $J = 9.9, 5.0$ Hz, 1H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ (ppm) = 189.99, 166.36, 142.73, 138.53, 41.33, 37.59, 34.17, 27.80, 21.98, 14.02, 5.03, 2.68; Exact mass calculated for $\text{C}_{12}\text{H}_{15}\text{O}_3$ $[\text{M}+\text{H}]^+$ 207.1016, found 207.1017; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, $\lambda = 254$ nm): t_{R} (minor) = 21.7 min, t_{R} (major) = 35.3 min, $ee = 98\%$; $[\alpha]_{\text{D}}^{25} = -115.7$ ($c = 0.42$ in DCM).



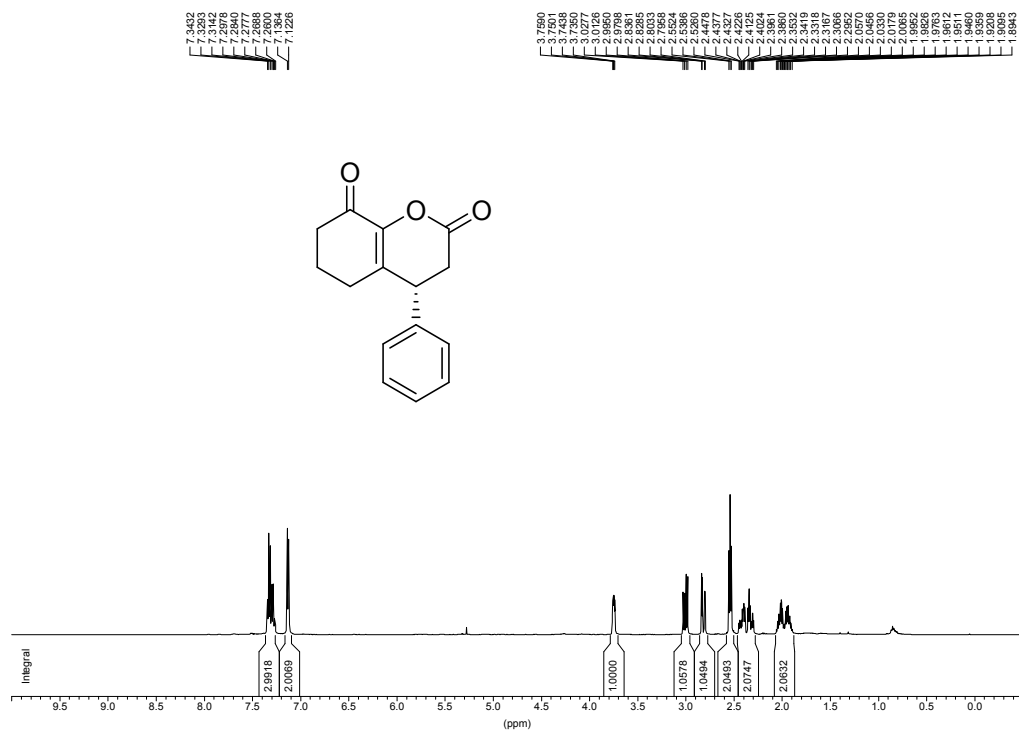
(S)-4-(tert-butyldimethylsilyl)-3,4,6,7-tetrahydro-2H-chromene-2,8(5H)-dione (3p) 20.4 mg, 73%

yield; White solid; mp 111–113 °C; $^1\text{H NMR}$ (300 MHz, CDCl_3) δ (ppm) = 2.77 (d, $J = 4.6$ Hz, 2H), 2.63 – 2.32 (m, 4H), 2.10 (t, $J = 4.6$ Hz, 1H), 2.07 – 1.92 (m, 1H), 0.95 (s, 9H), 0.11 (s, 3H), 0.03 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 188.72, 167.30, 142.90, 142.55, 37.59, 30.87, 30.21, 26.59, 25.39, 22.03, 17.62, -5.85, -6.93; Exact mass calculated for $\text{C}_{15}\text{H}_{25}\text{O}_3\text{Si}$ $[\text{M}+\text{H}]^+$ 281.1567, found 281.1574; HPLC (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, $\lambda = 254$ nm): t_{R} (minor) = 16.4 min, t_{R} (major) = 20.5 min, $ee = 93\%$; $[\alpha]_{\text{D}}^{25} = -167.7$ ($c = 0.91$ in DCM).

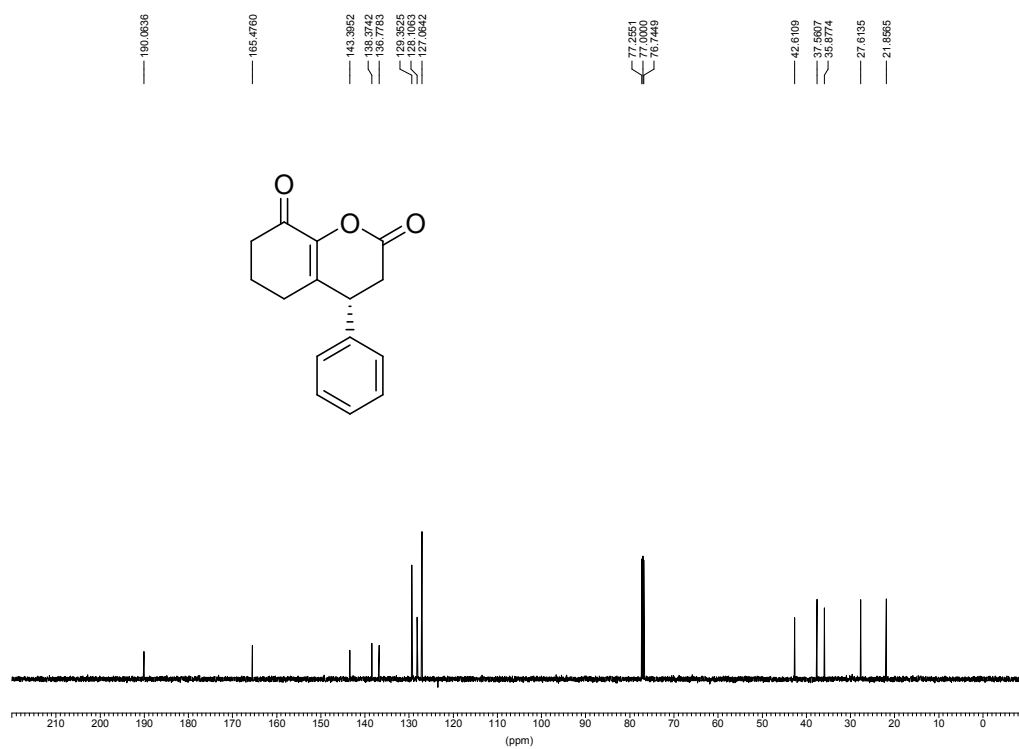
4. NMR Spectra of the Products

Compound 3a

^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (125 MHz, CDCl_3)

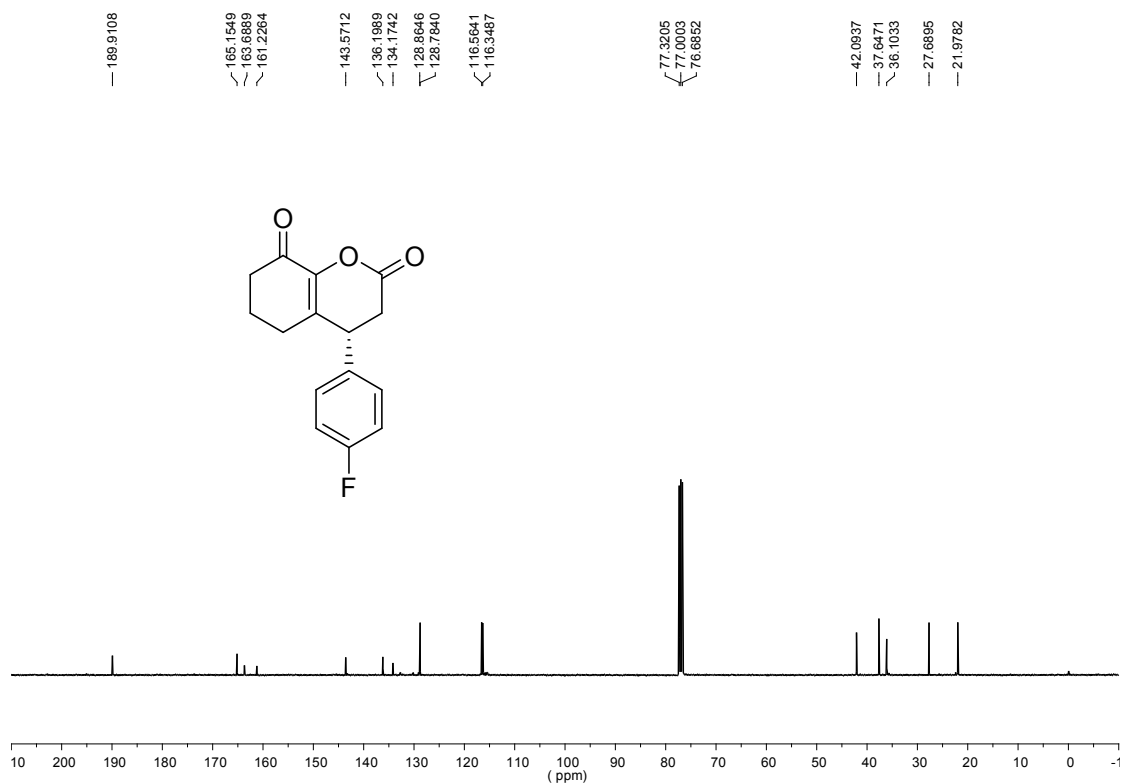


Compound 3b

^1H NMR (300 MHz, CDCl_3)

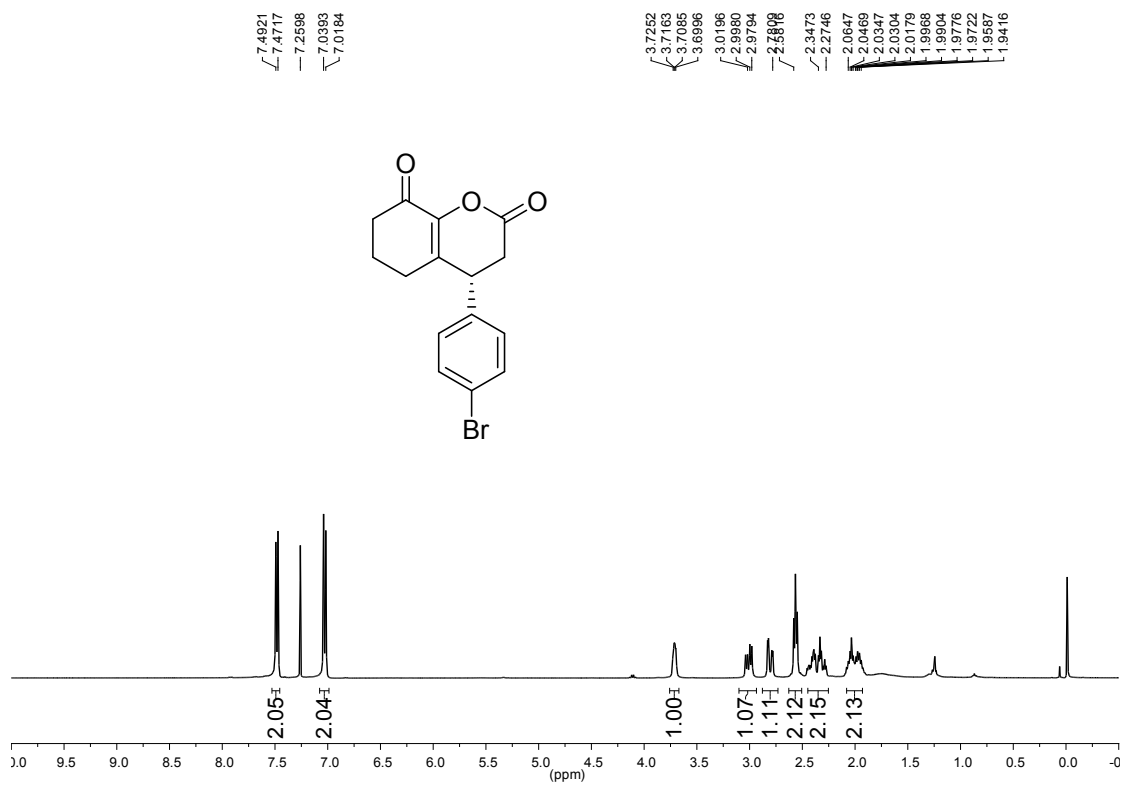


^{13}C NMR (100 MHz, CDCl_3)

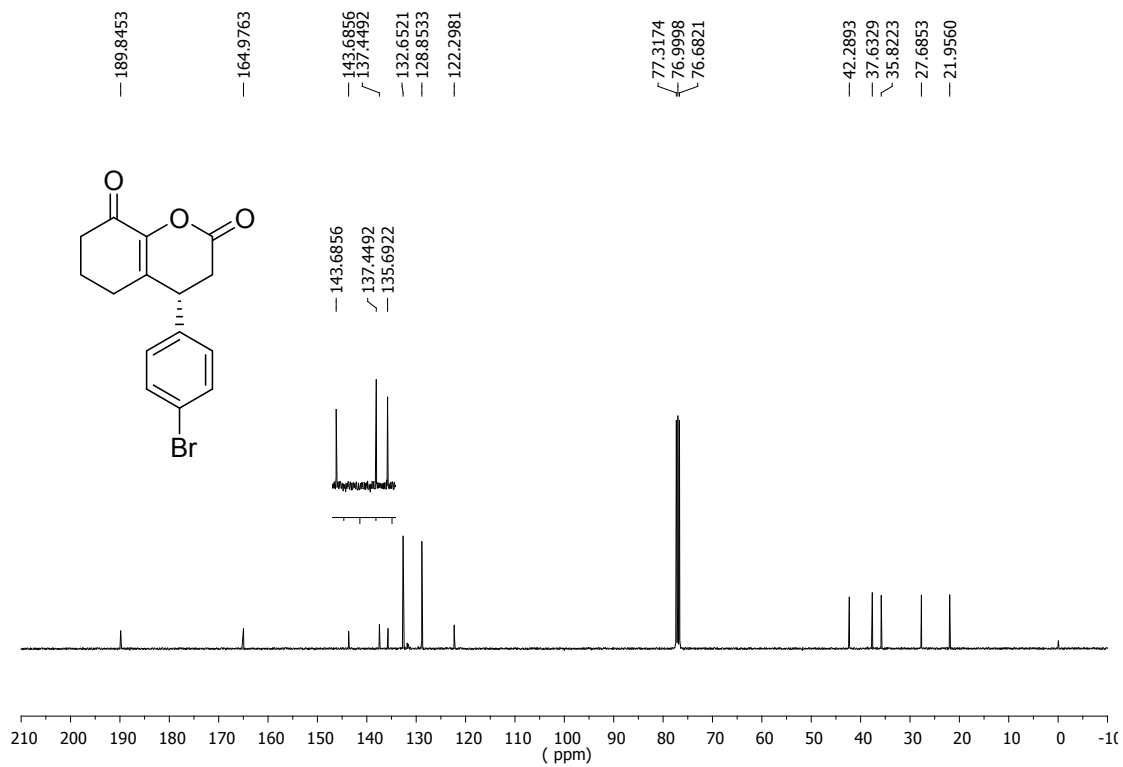


Compound 3c

^1H NMR (400 MHz, CDCl_3)

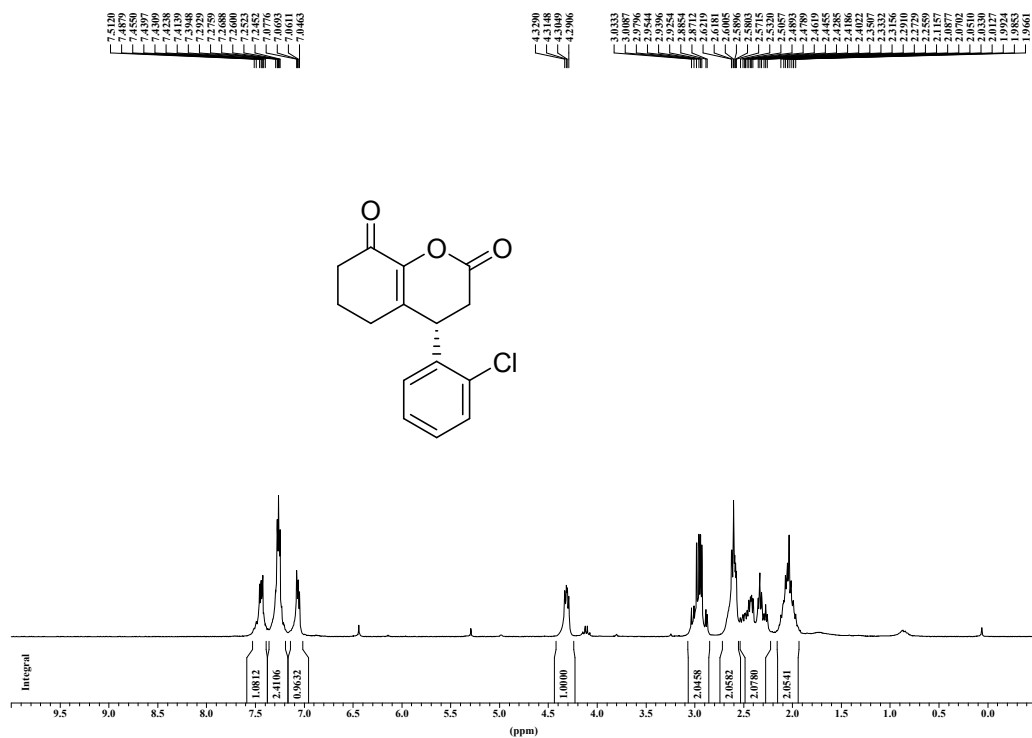


^{13}C NMR (100 MHz, CDCl_3)

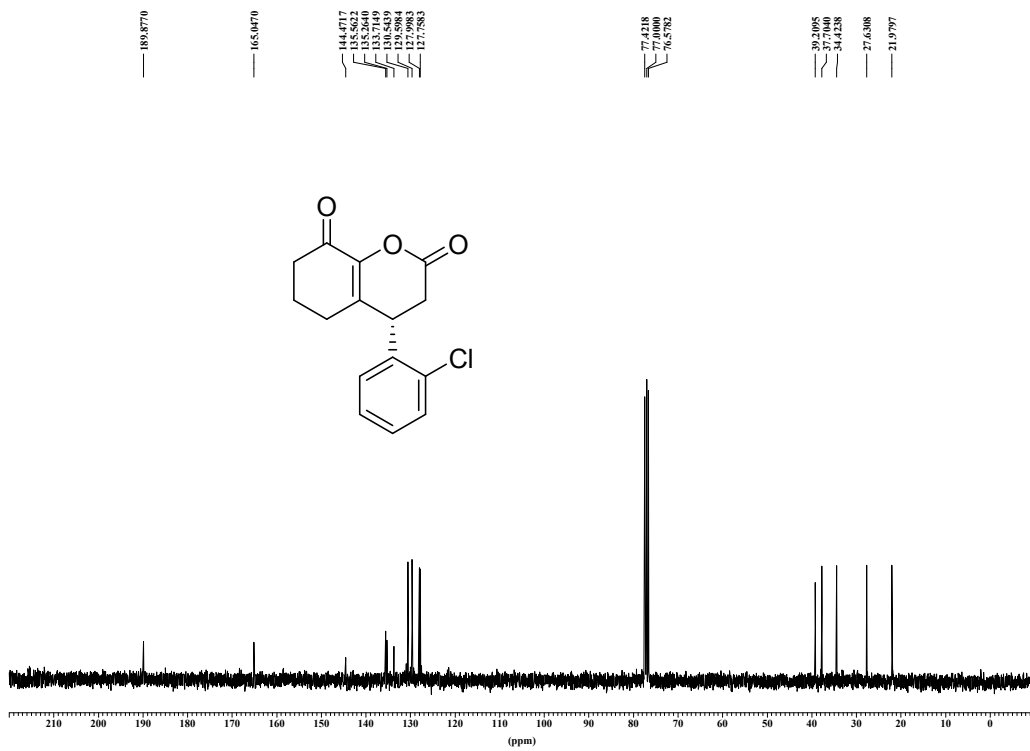


Compound 3d

^1H NMR (300 MHz, CDCl_3)

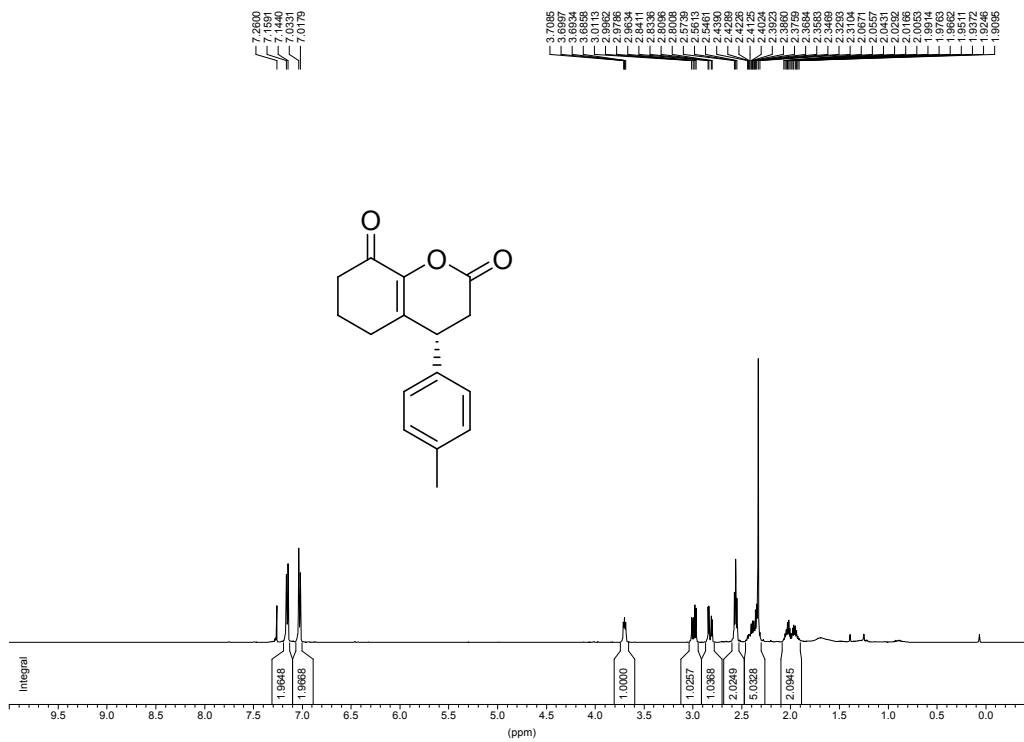


^{13}C NMR (75 MHz, CDCl_3)

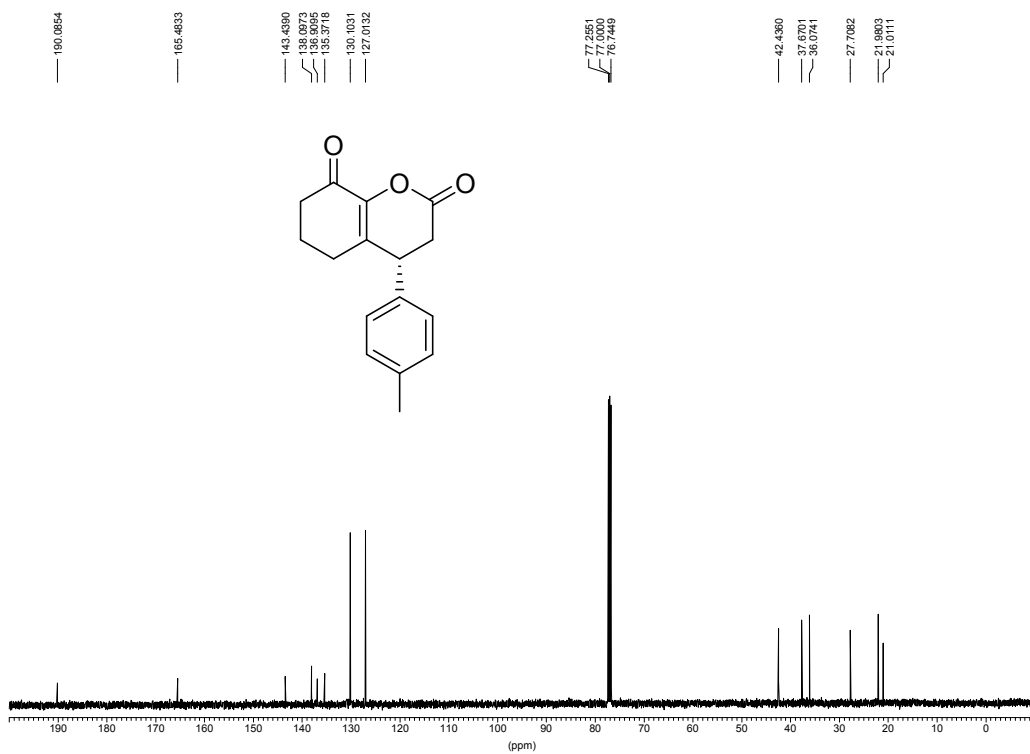


Compound 3e

¹H NMR (500 MHz, CDCl₃)

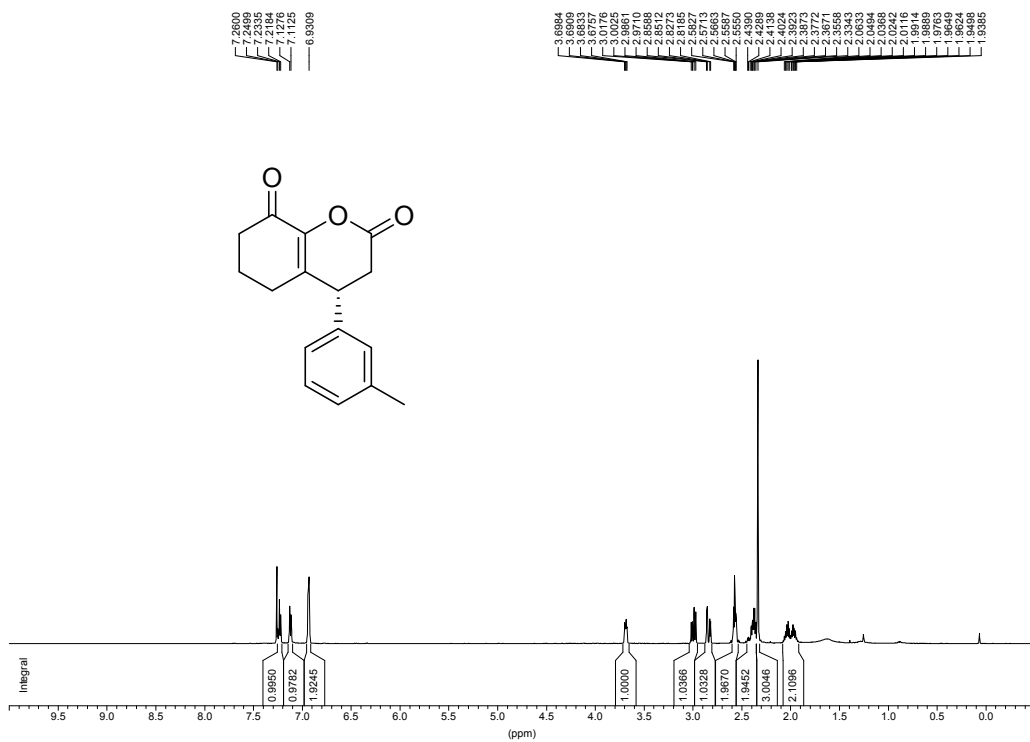


¹³C NMR (125 MHz, CDCl₃)

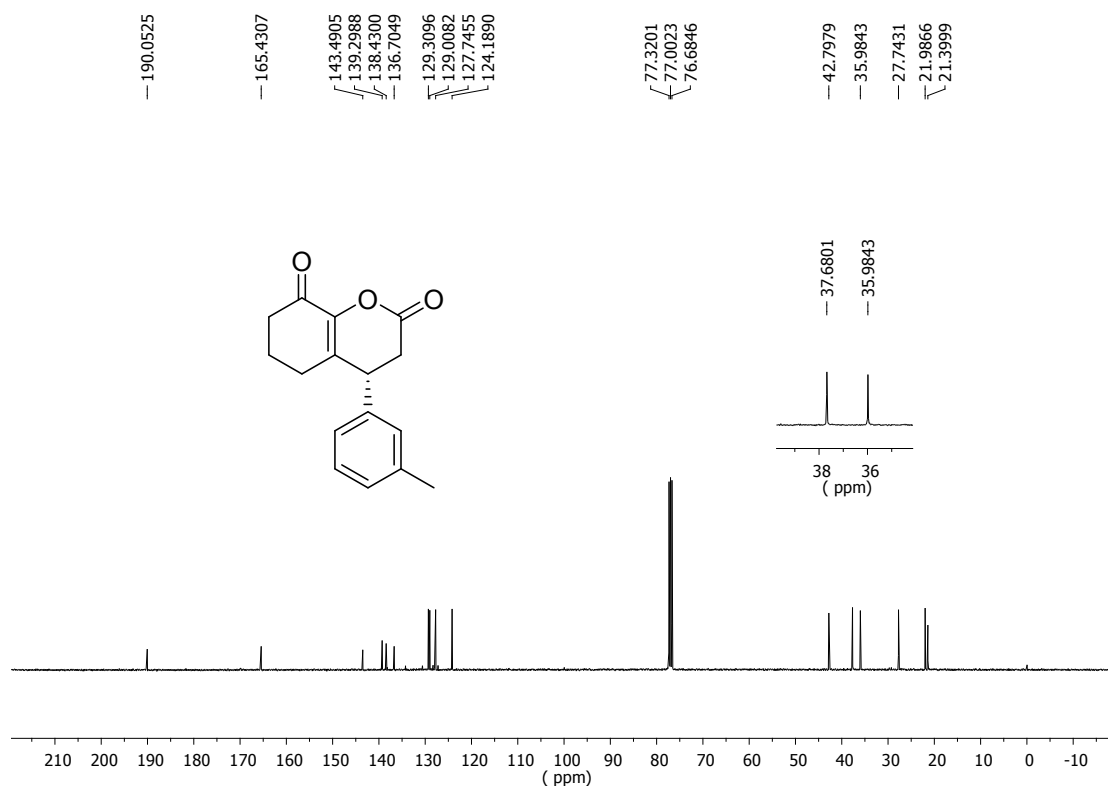


Compound 3f

¹H NMR (500 MHz, CDCl₃)

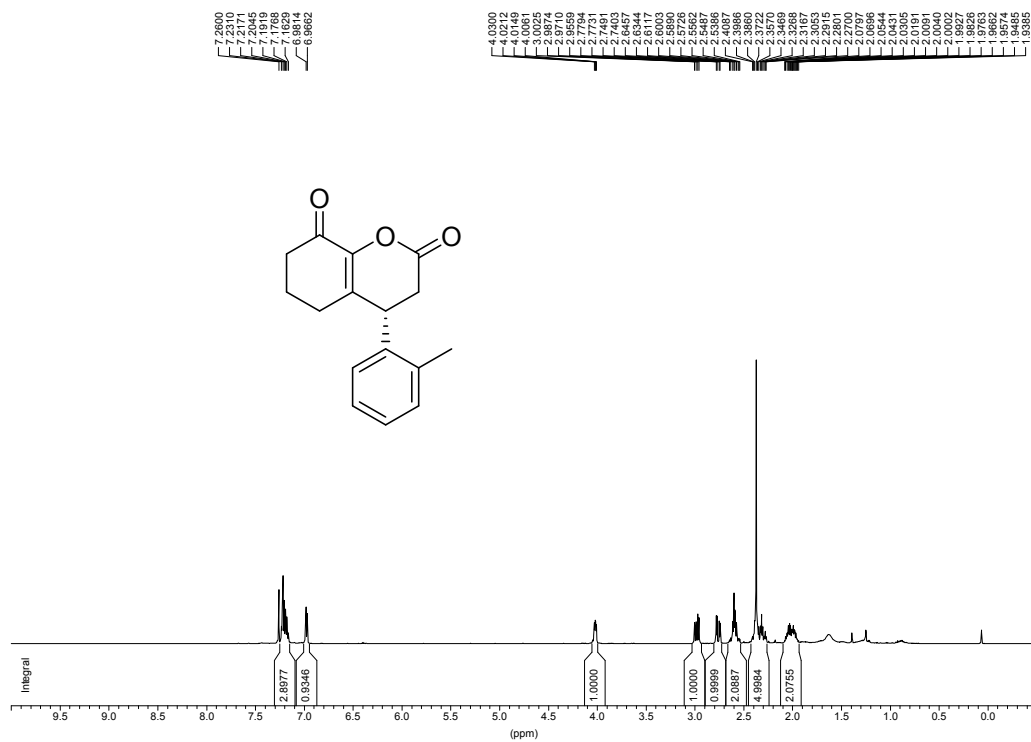


^{13}C NMR (100 MHz, CDCl_3)

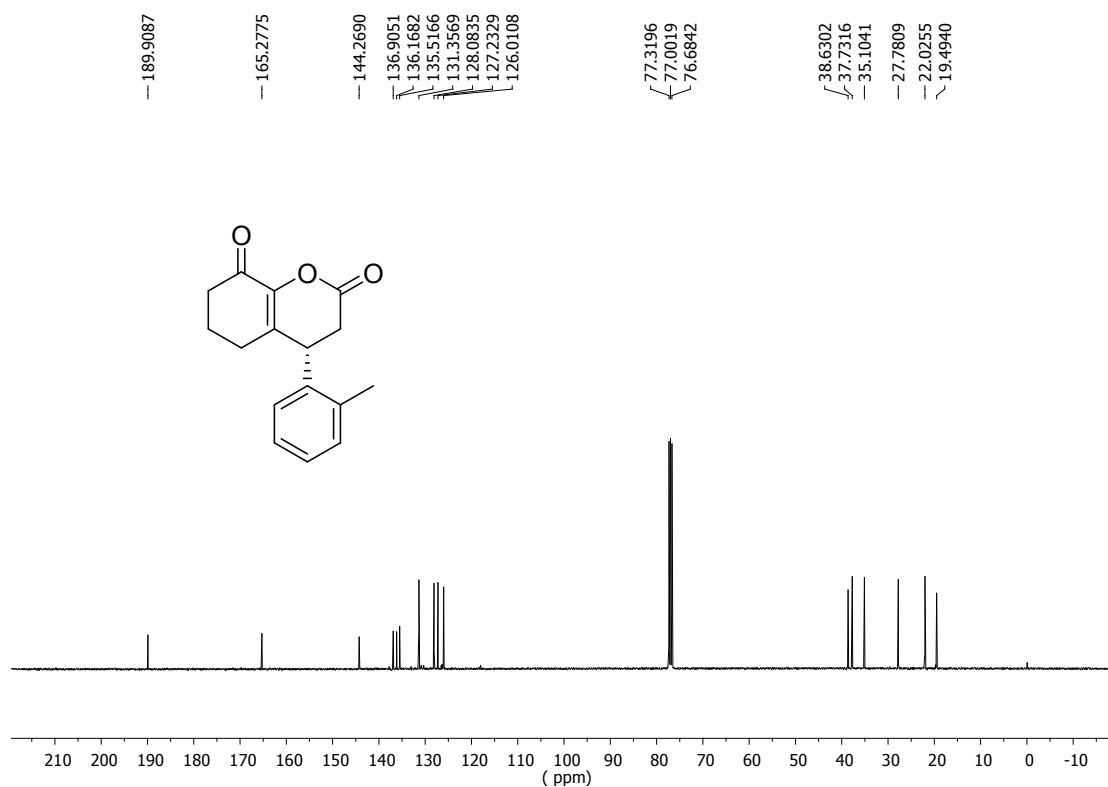


Compound 3g

^1H NMR (500 MHz, CDCl_3)



^{13}C NMR (100 MHz, CDCl_3)

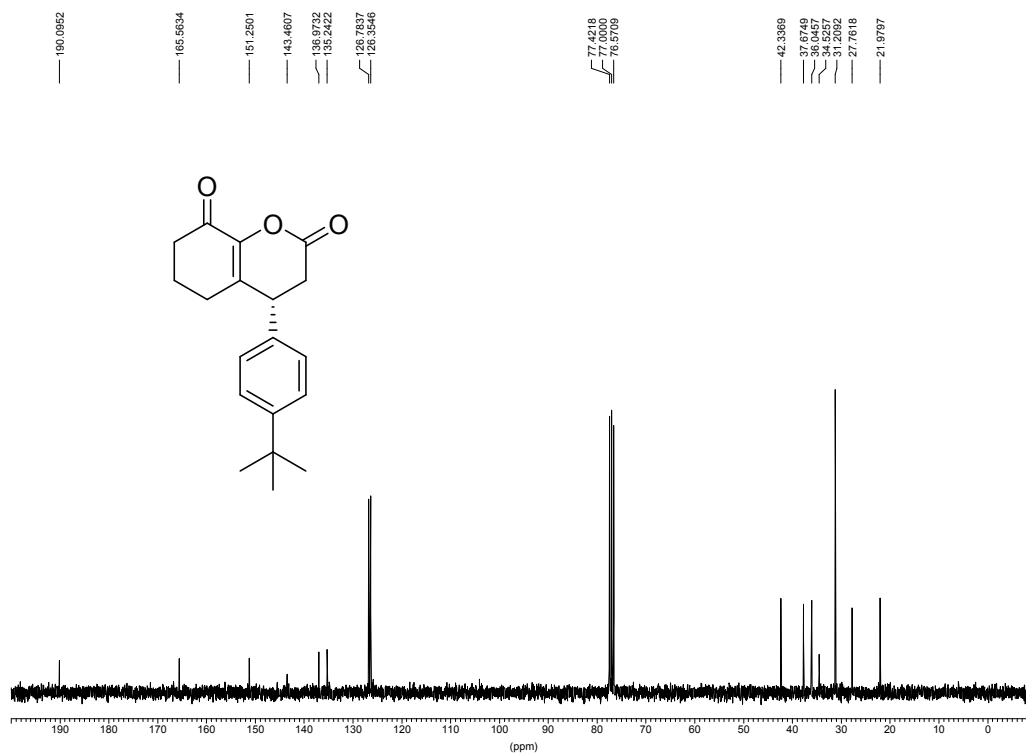


Compound 3h

^1H NMR (300 MHz, CDCl_3)

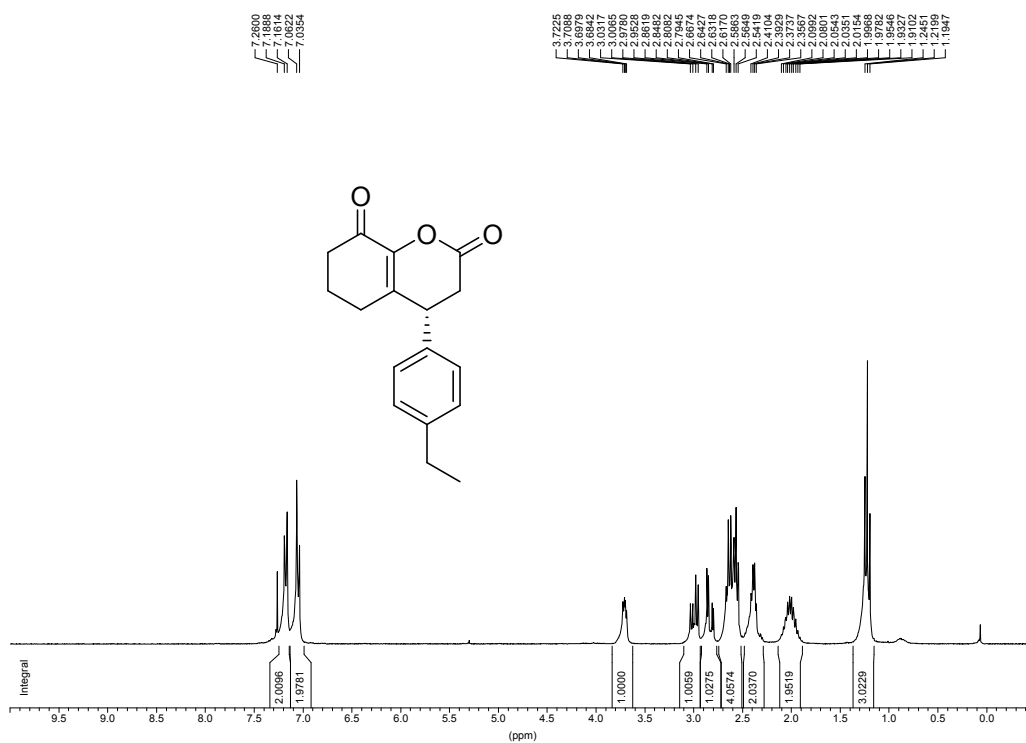


^{13}C NMR (75 MHz, CDCl_3)

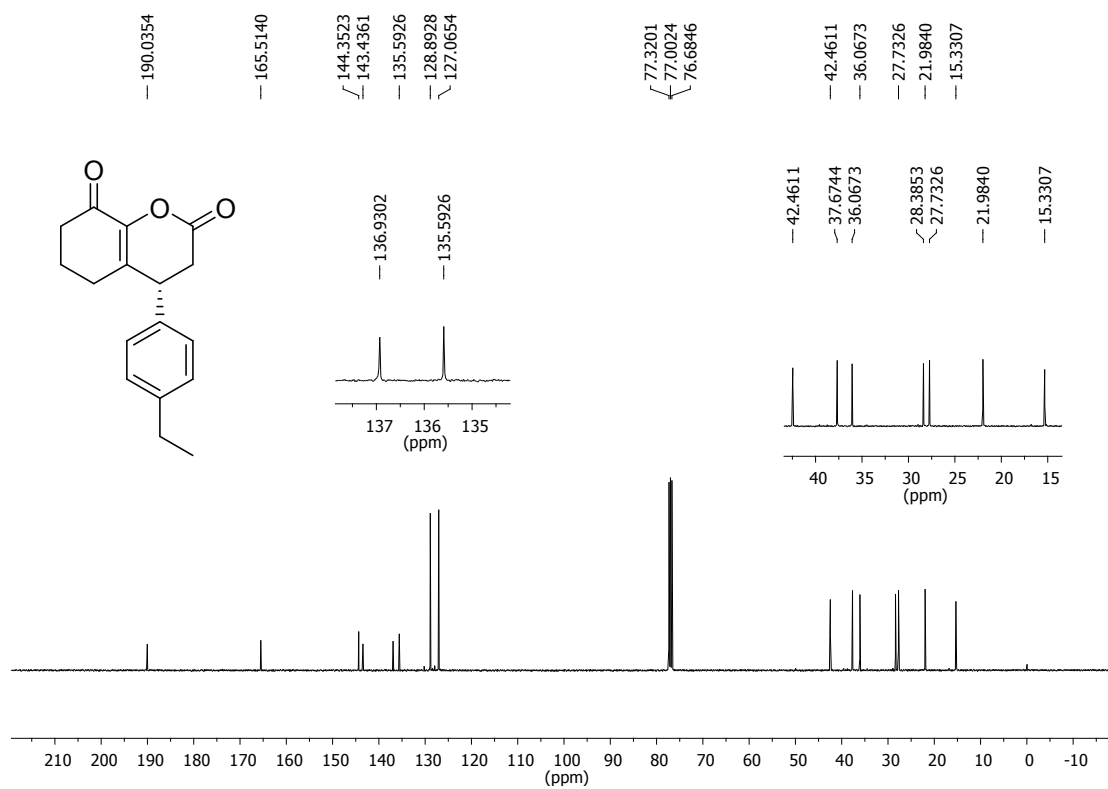


Compound 3i

^1H NMR (300 MHz, CDCl_3)



^{13}C NMR (100 MHz, CDCl_3)

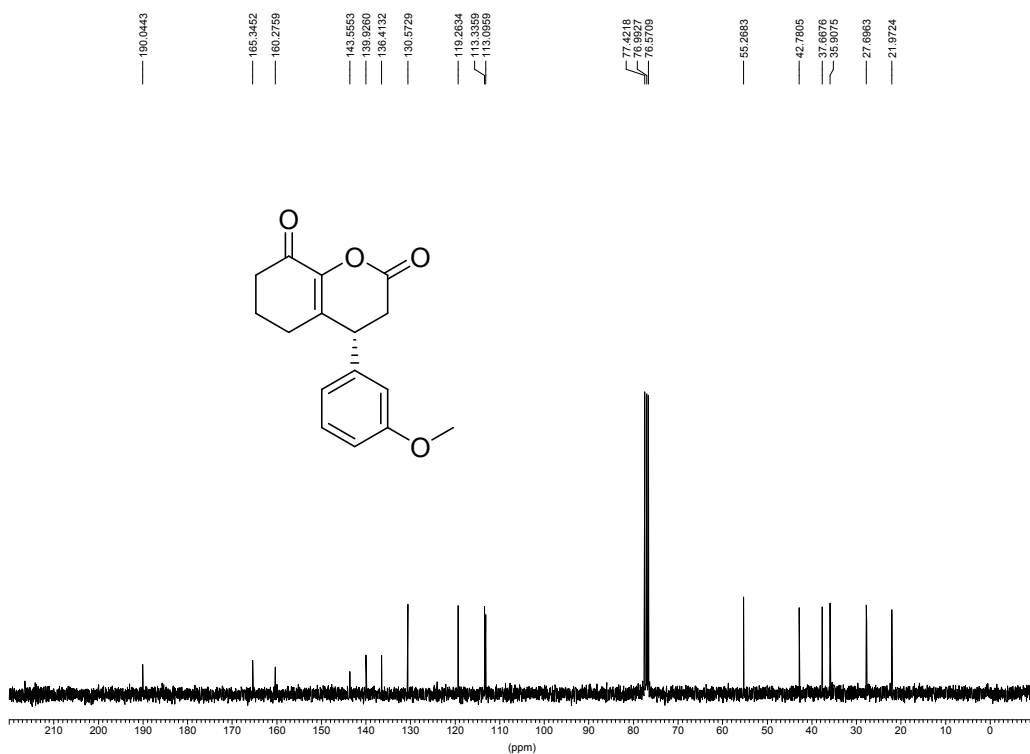


Compound 3j

^1H NMR (300 MHz, CDCl_3)

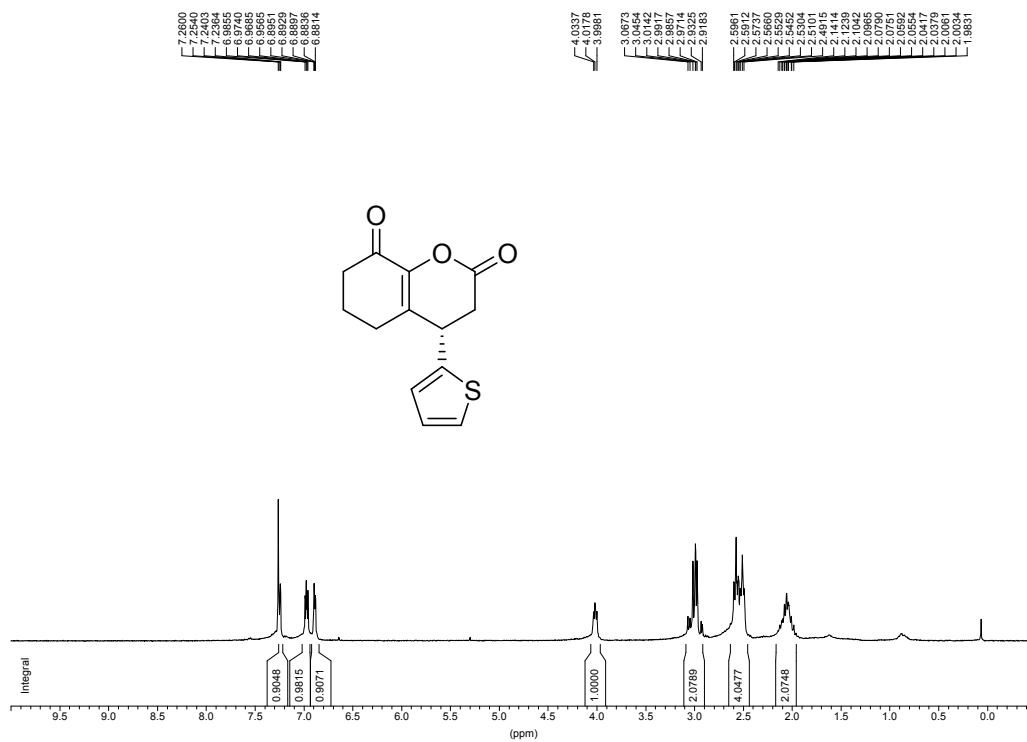


^{13}C NMR (75 MHz, CDCl_3)

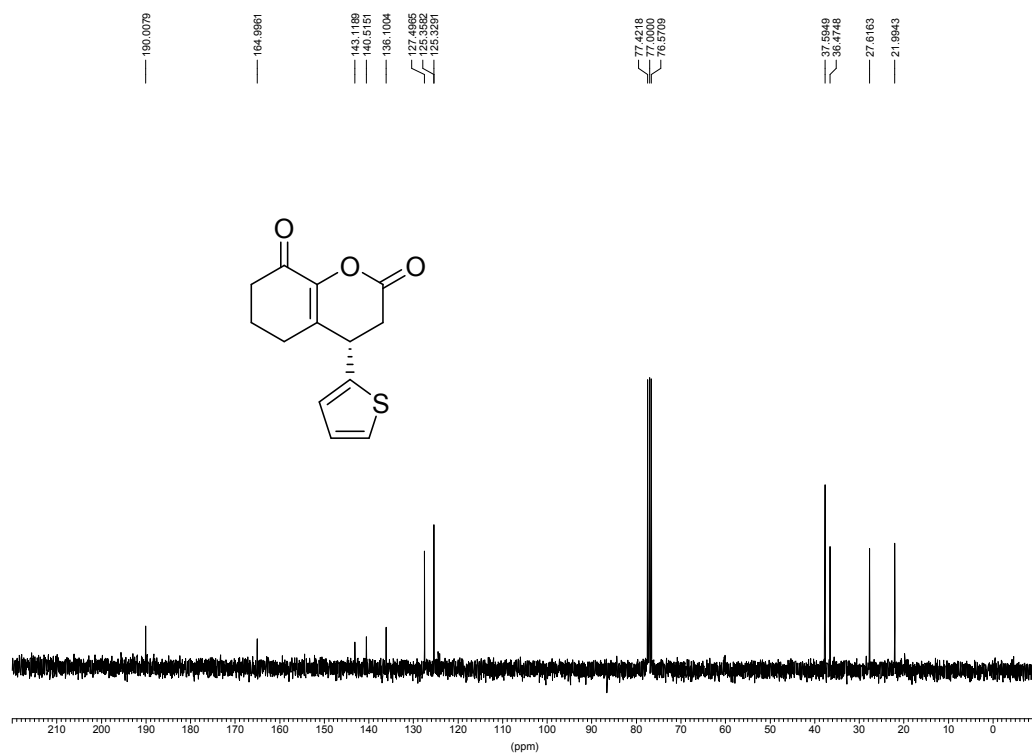


Compound 3k

¹H NMR (300 MHz, CDCl₃)

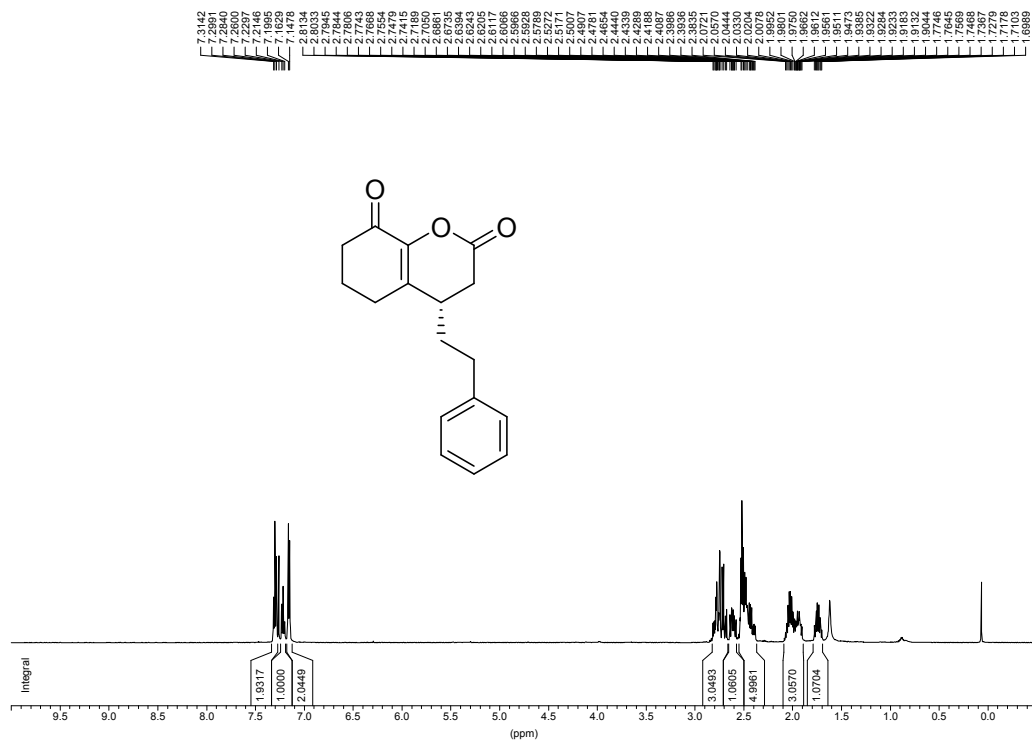


^{13}C NMR (75 MHz, CDCl_3)

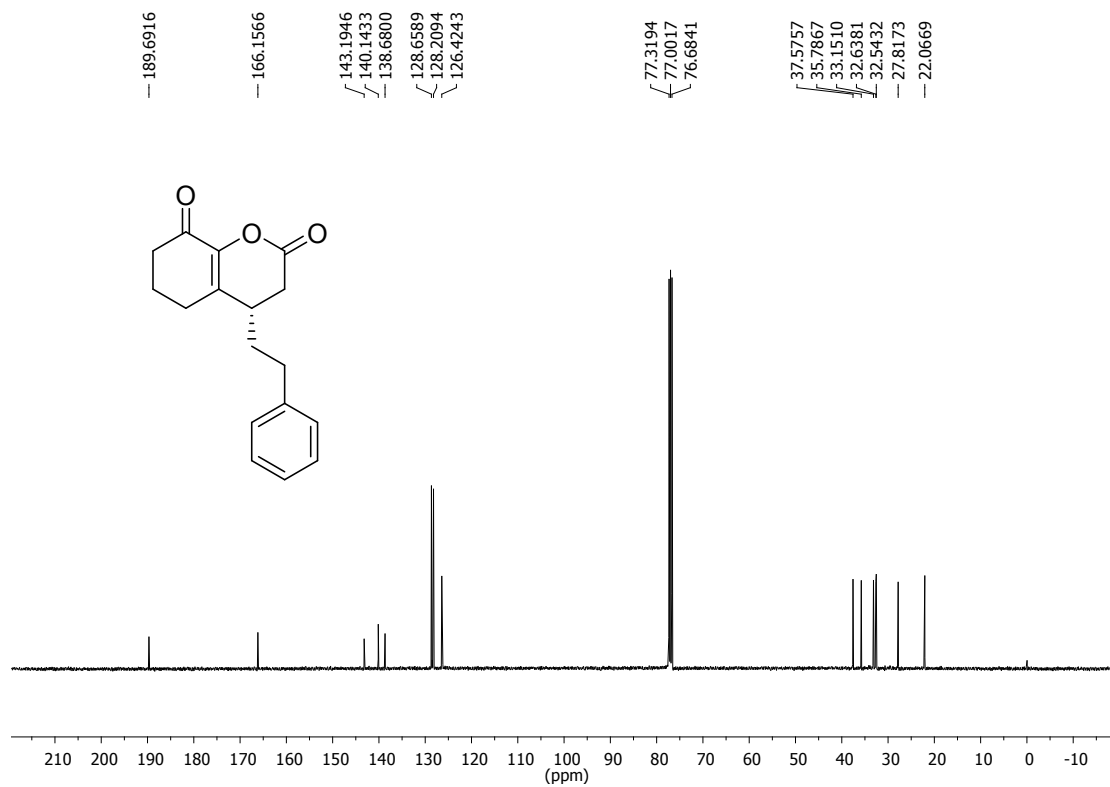


Compound 31

^1H NMR (500 MHz, CDCl_3)

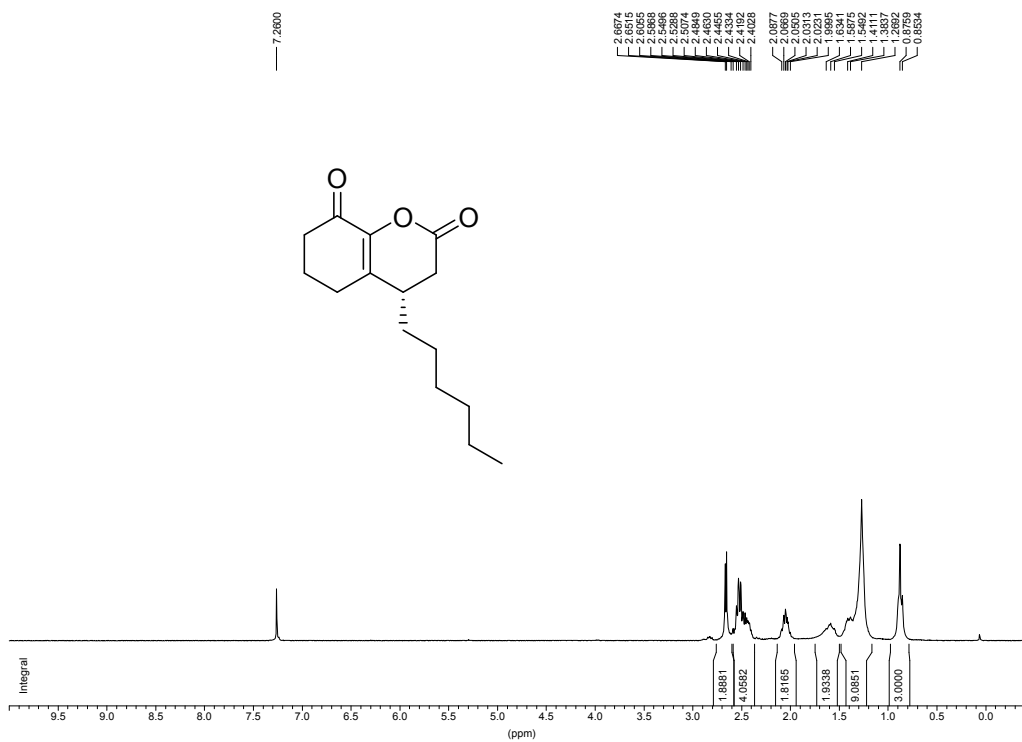


¹³C NMR (100 MHz, CDCl₃)

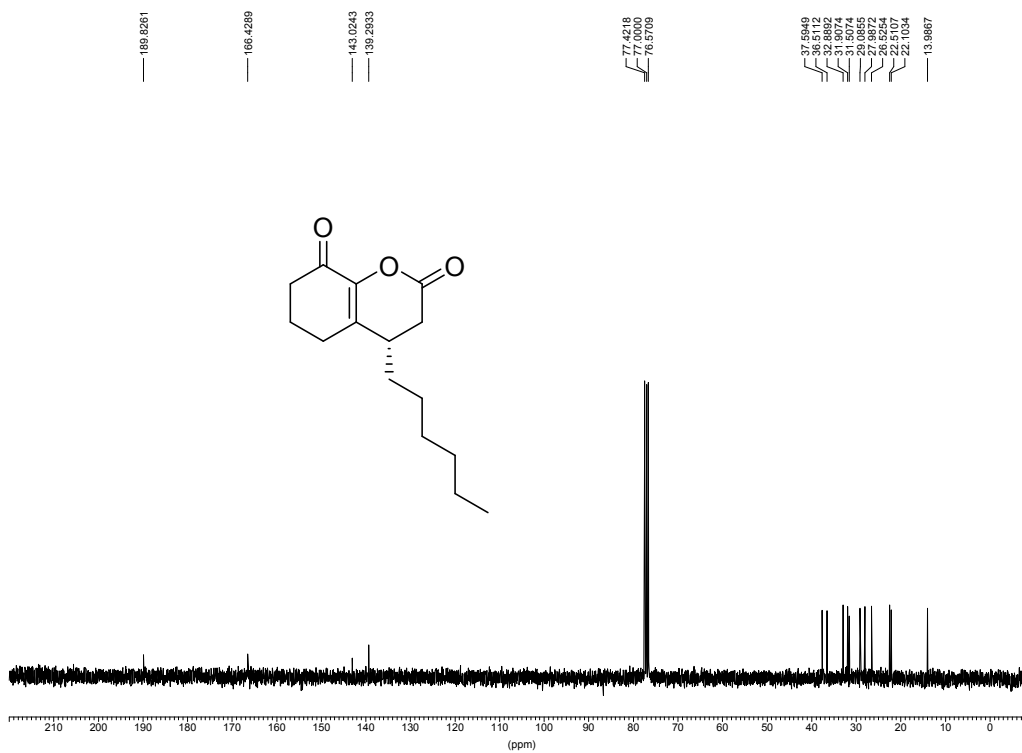


Compound 3m

¹H NMR (300 MHz, CDCl₃)

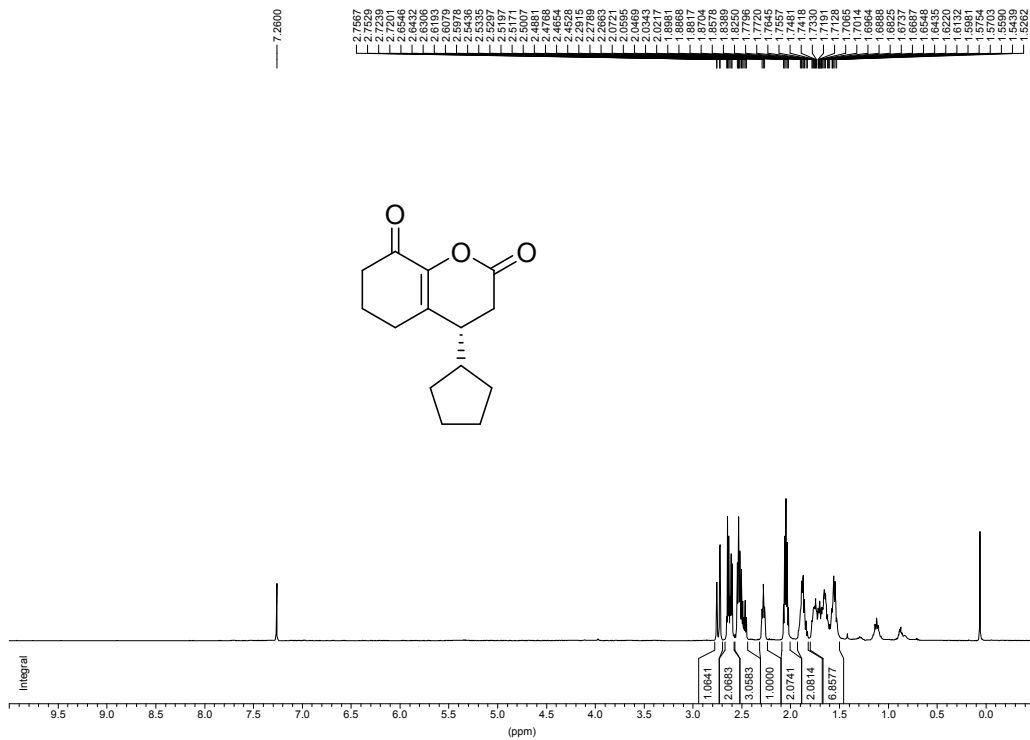


¹³C NMR (75 MHz, CDCl₃)

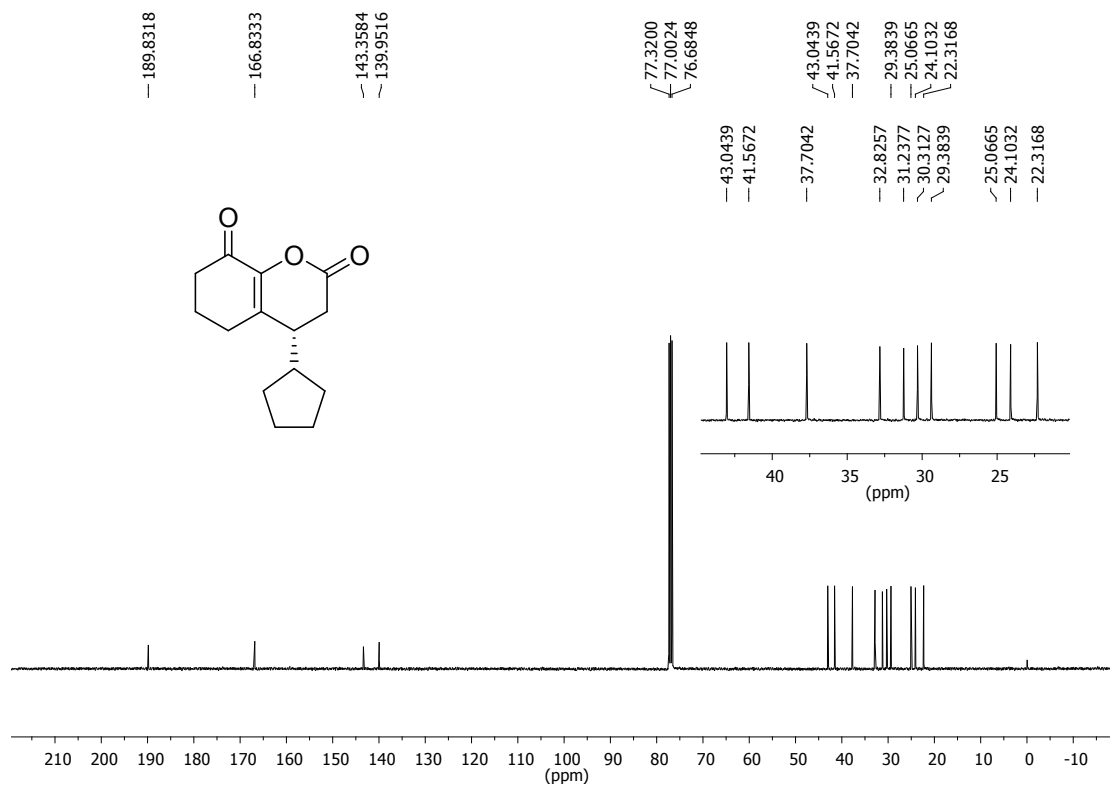


Compound 3n

¹H NMR (500 MHz, CDCl₃)

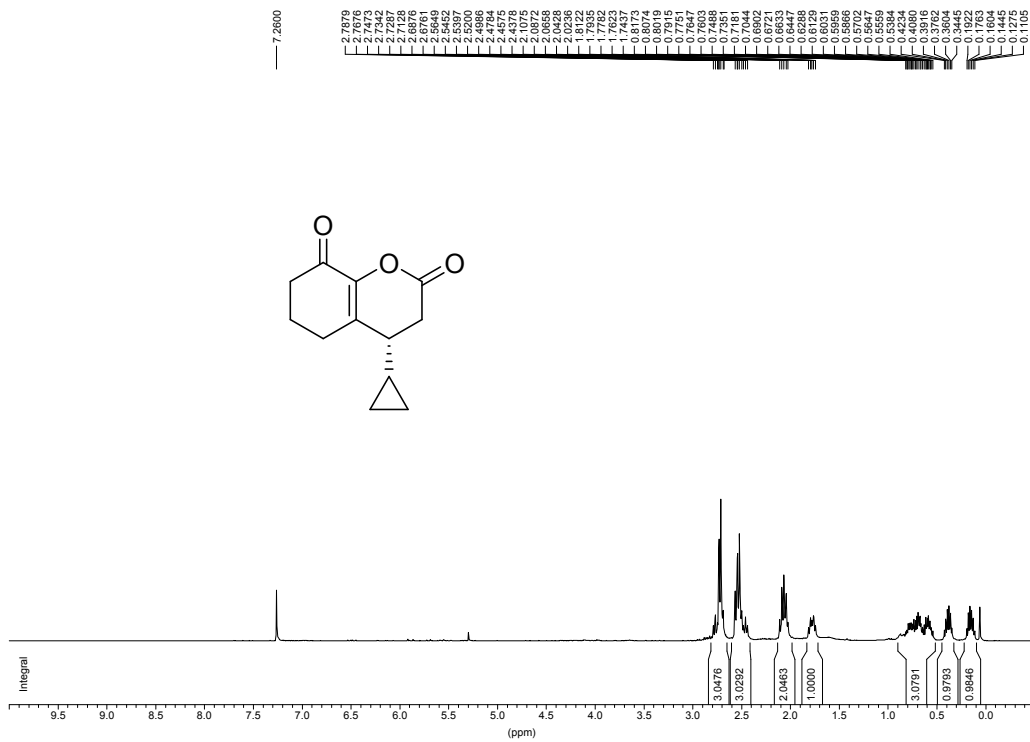


¹³C NMR (100 MHz, CDCl₃)

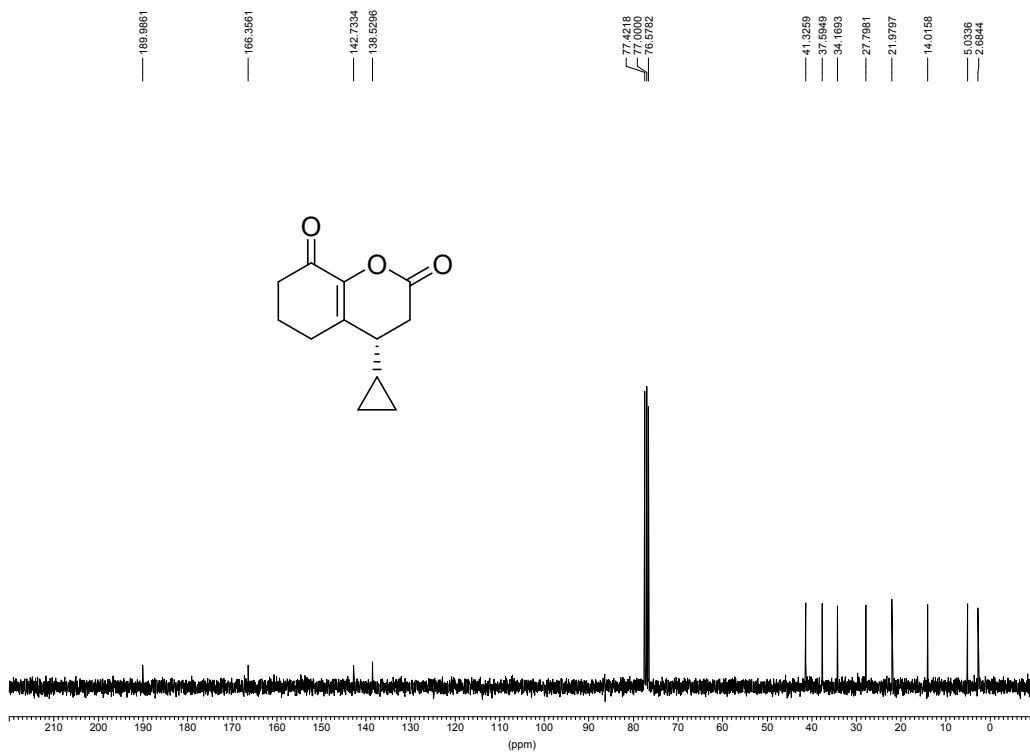


Compound 3o

¹H NMR (300 MHz, CDCl₃)

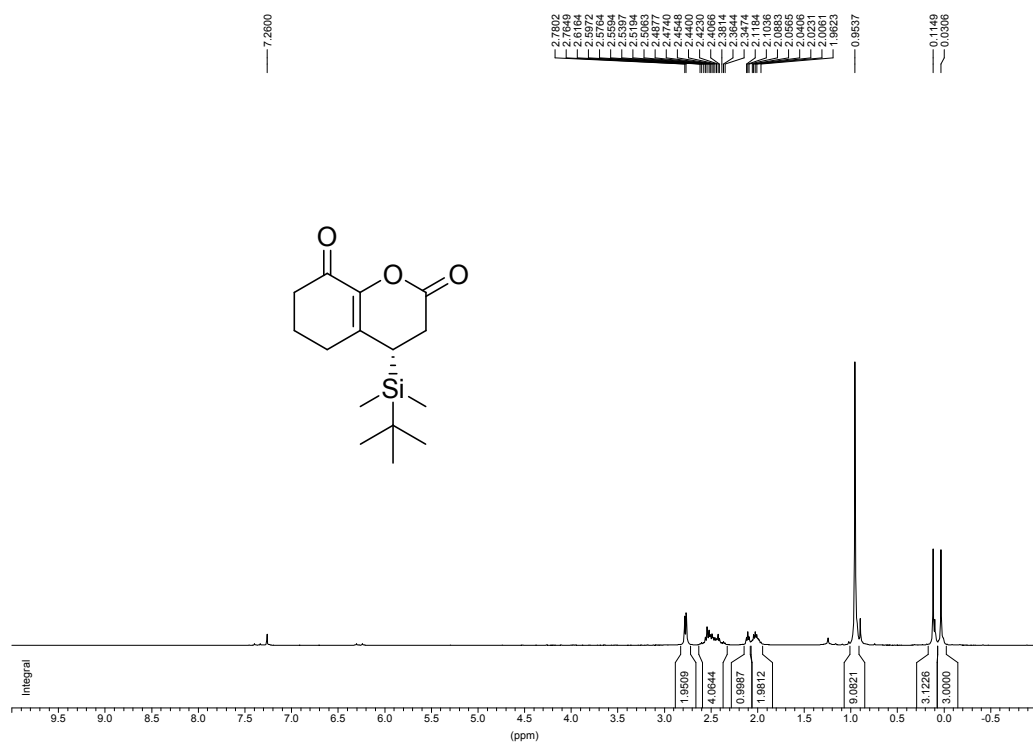


¹³C NMR (75 MHz, CDCl₃)

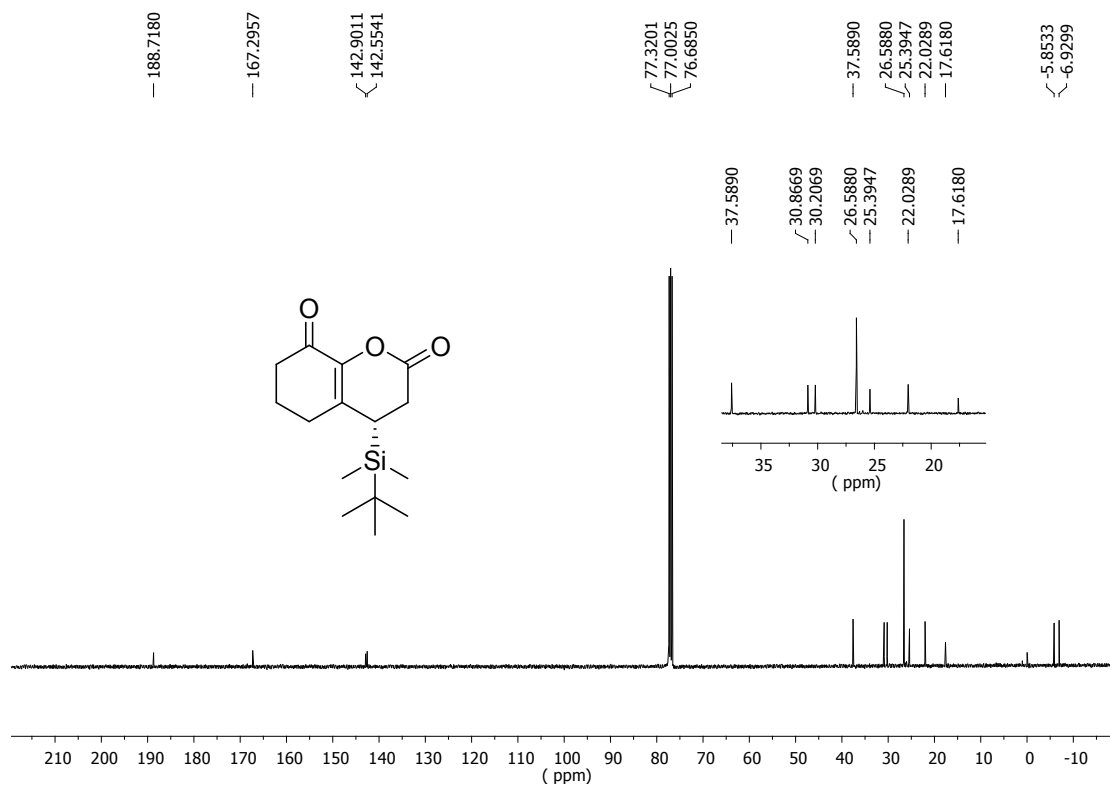


Compound 3p

¹H NMR (300 MHz, CDCl₃)



¹³C NMR (100 MHz, CDCl₃)



5. HPLC Chromatogram Profile of the Products

Racemic 3a

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq845.lcd

Acquired by : Admin

Sample Name : RQF045-3

Sample ID : RQ

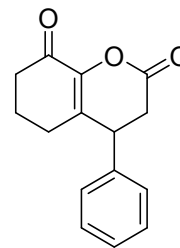
Data File Name : rq845.lcd

Method File Name : 40%IPA, 1ml-min, 40min.lcm

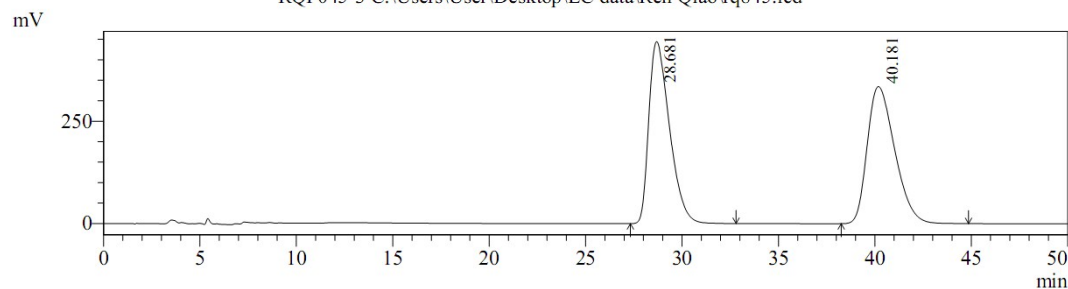
Batch File Name :

Report File Name : Default.lcr

Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram
RQF045-3 C:\Users\User\Desktop\LC data\Ren Qiao\rq845.lcd



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.681	33085665	444541	49.892	57.032
2	40.181	33228396	334922	50.108	42.968
Total		66314061	779463	100.000	100.000

Enantiomeric enriched 3a

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq857.lcd

Acquired by : Admin

Sample Name : RQF056-3

Sample ID : RQ

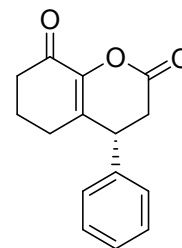
Data File Name : rq857.lcd

Method File Name : 40%IPA, 1ml-min, 40min.lcm

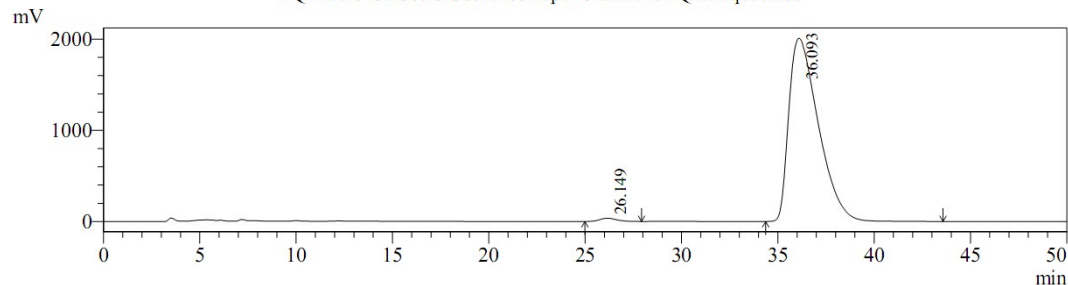
Batch File Name :

Report File Name : Default.lcr

Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram
RQF056-3 C:\Users\User\Desktop\LC data\Ren Qiao\rq857.lcd



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.149	2125304	36056	0.984	1.762
2	36.093	213953460	2009794	99.016	98.238
Total		216078764	2045850	100.000	100.000

Racemic 3b

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq874.lcd

Acquired by : Admin

Sample Name : RQF069

Sample ID : RQ

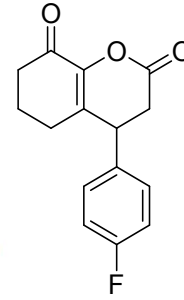
Data File Name : rq874.lcd

Method File Name : 40%IPA, 1ml-min, 40min.lcm

Batch File Name :

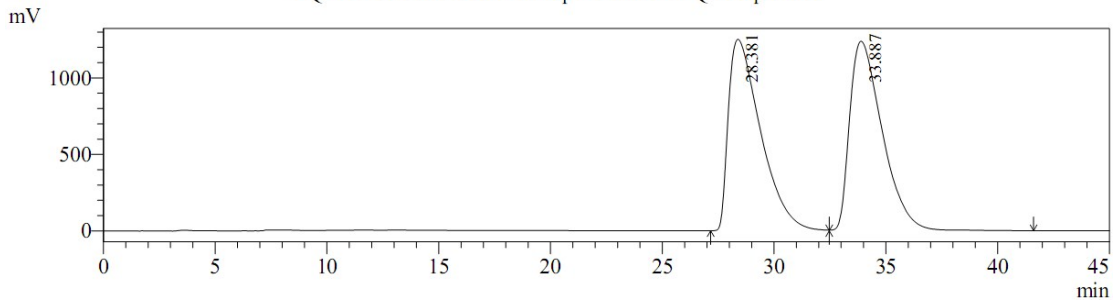
Report File Name : Default.lcr

Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram

RQF069 C:\Users\User\Desktop\LC data\Ren Qiao\rq874.lcd



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.381	125176838	1251224	49.921	50.257
2	33.887	125575227	1238434	50.079	49.743
Total		250752066	2489658	100.000	100.000

Enantiomeric enriched 3b

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq875.lcd

Acquired by : Admin

Sample Name : RQF070

Sample ID : RQ

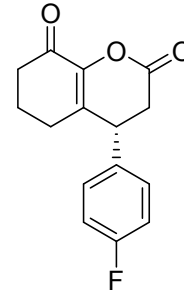
Data File Name : rq875.lcd

Method File Name : 40%IPA, 1ml-min, 40min.lcm

Batch File Name :

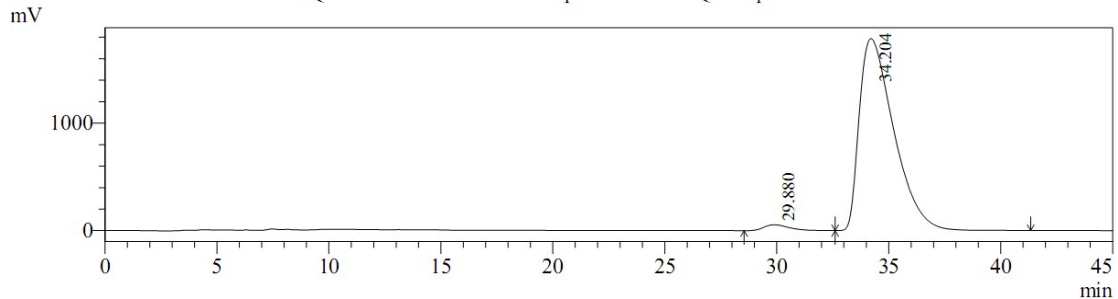
Report File Name : Default.lcr

Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram

RQF070 C:\Users\User\Desktop\LC data\Ren Qiao\rq875.lcd



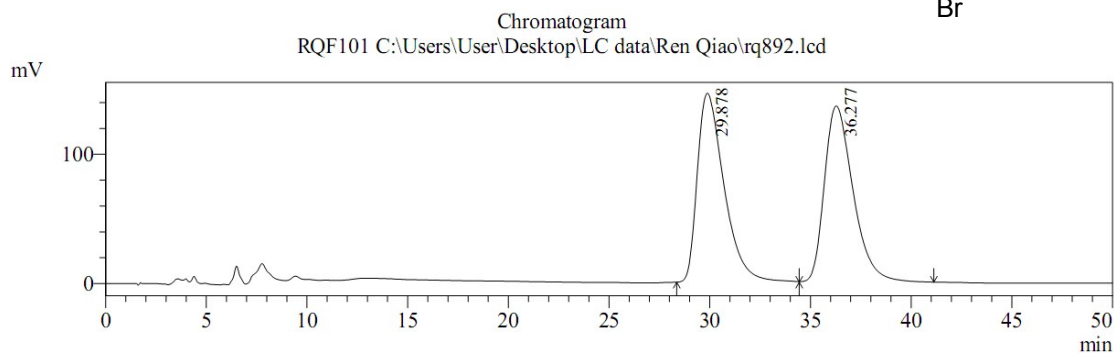
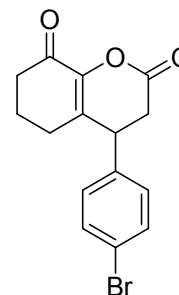
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.880	4336622	54784	2.216	2.974
2	34.204	191341258	1787298	97.784	97.026
Total		195677880	1842082	100.000	100.000

Racemic 3c

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq892.lcd
 Acquired by : Admin
 Sample Name : RQF101
 Sample ID : RQ
 Data File Name : rq892.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



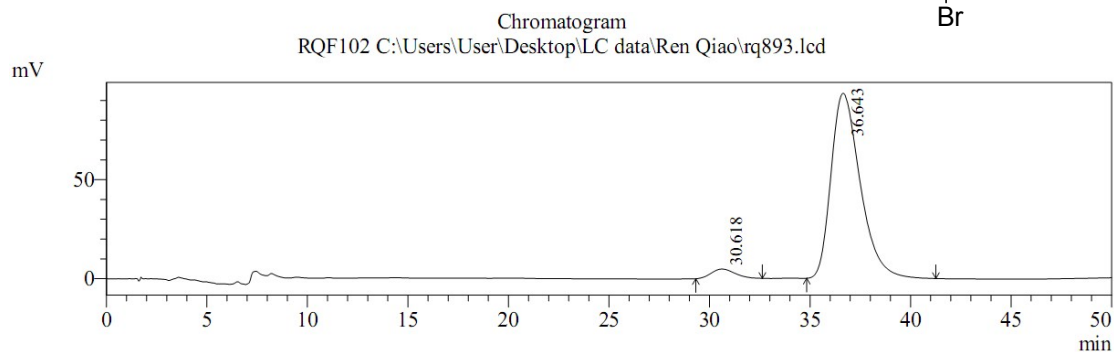
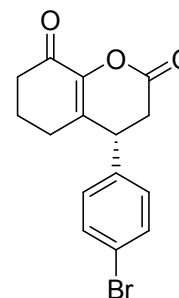
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.878	13530407	146140	49.990	51.737
2	36.277	13535831	136326	50.010	48.263
Total		27066238	282467	100.000	100.000

Enantiomeric enriched 3c

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq893.lcd
 Acquired by : Admin
 Sample Name : RQF102
 Sample ID : RQ
 Data File Name : rq893.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



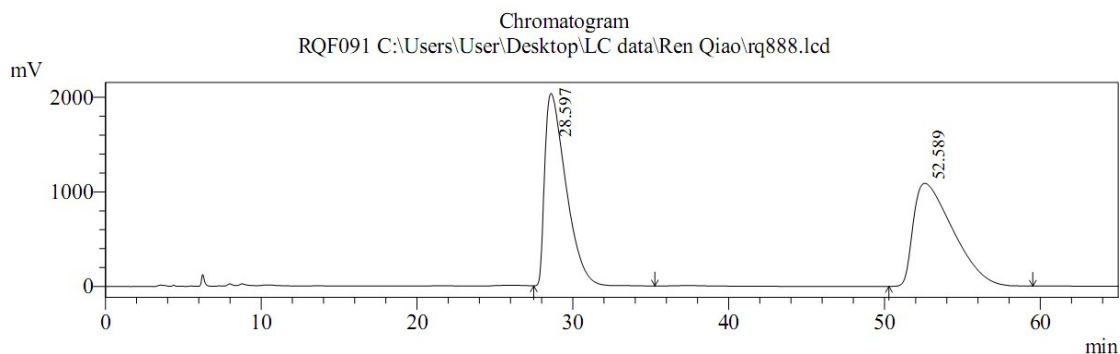
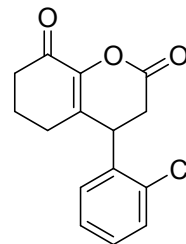
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.618	394865	4819	4.020	4.904
2	36.643	9428101	93460	95.980	95.096
Total		9822966	98279	100.000	100.000

Racemic 3d

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq888.lcd
 Acquired by : Admin
 Sample Name : RQF091
 Sample ID : RQ
 Data File Name : rq888.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



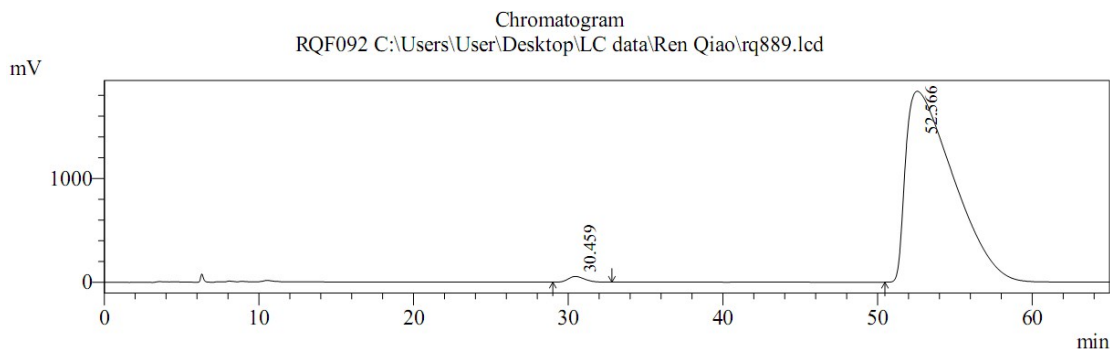
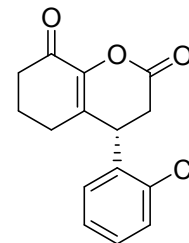
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.597	192240044	2035204	49.947	65.147
2	52.589	192647224	1088818	50.053	34.853
Total		384887268	3124022	100.000	100.000

Enantiomeric enriched 3d

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq889.lcd
 Acquired by : Admin
 Sample Name : RQF092
 Sample ID : RQ
 Data File Name : rq889.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



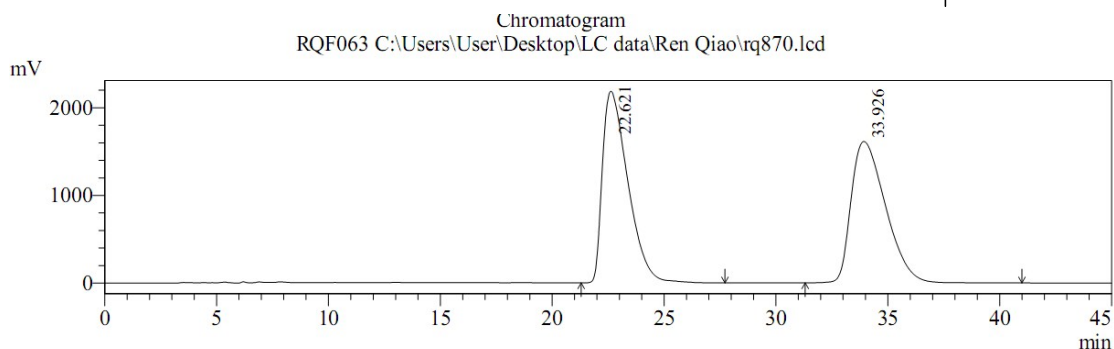
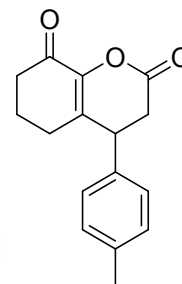
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.459	4089470	55077	1.023	2.908
2	52.566	395723213	1838900	98.977	97.092
Total		399812683	1893978	100.000	100.000

Racemic 3e

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq870.lcd
 Acquired by : Admin
 Sample Name : RQF063
 Sample ID : RQ
 Data File Name : rq870.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



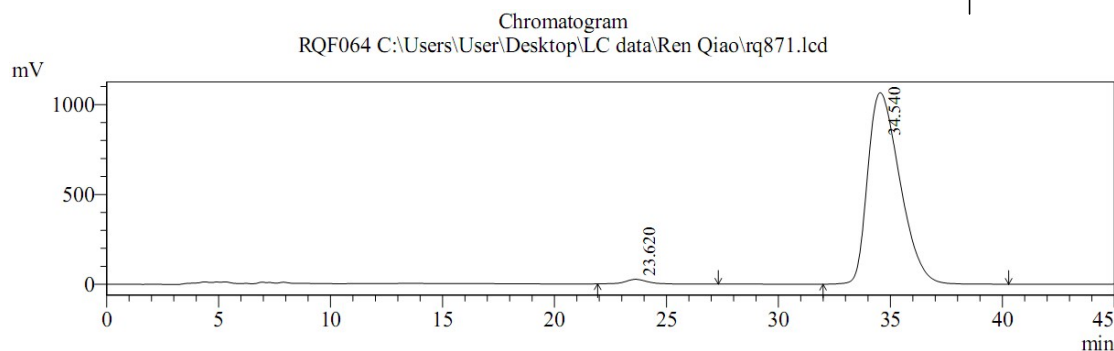
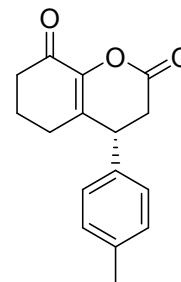
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.621	173006355	2185867	50.045	57.526
2	33.926	172697787	1613918	49.955	42.474
Total		345704142	3799785	100.000	100.000

Enantiomeric enriched 3e

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq871.lcd
 Acquired by : Admin
 Sample Name : RQF064
 Sample ID : RQ
 Data File Name : rq871.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



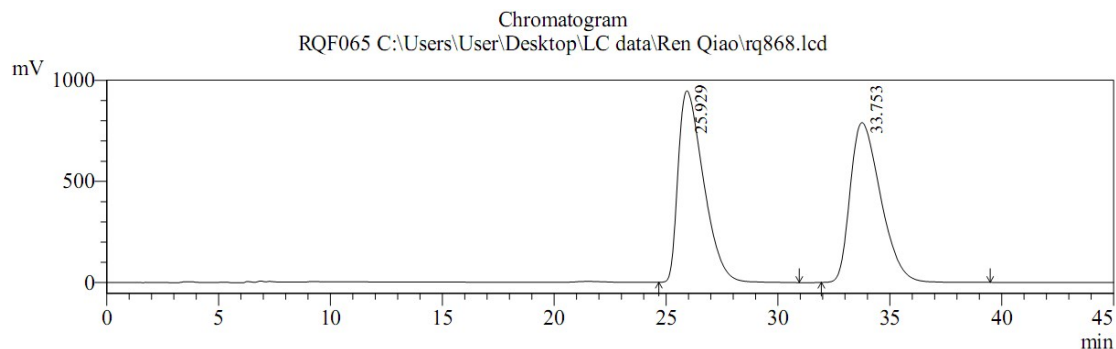
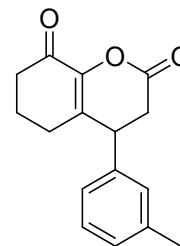
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.620	1796780	25905	1.658	2.373
2	34.540	106594140	1065802	98.342	97.627
Total		108390920	1091707	100.000	100.000

Racemic 3f

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq868.lcd
 Acquired by : Admin
 Sample Name : RQF065
 Sample ID : RQ
 Data File Name : rq868.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



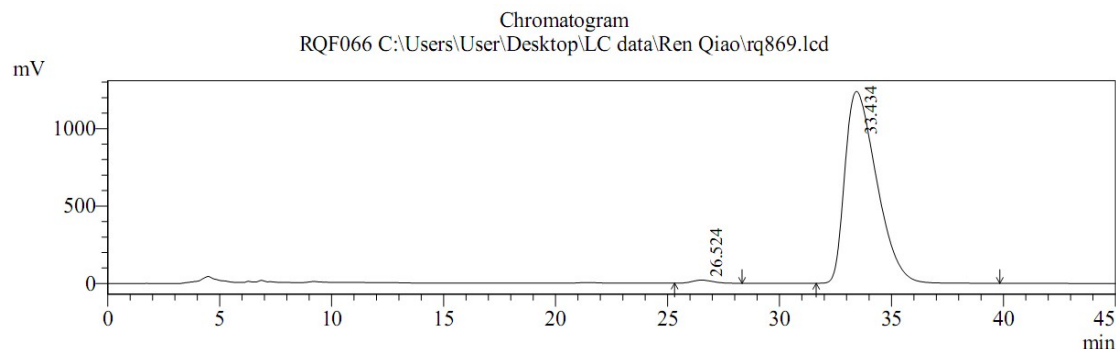
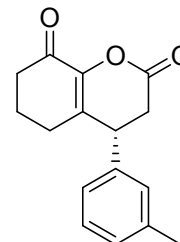
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.929	74490492	946731	49.932	54.499
2	33.753	74692394	790410	50.068	45.501
Total		149182886	1737141	100.000	100.000

Enantiomeric enriched 3f

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq869.lcd
 Acquired by : Admin
 Sample Name : RQF066
 Sample ID : RQ
 Data File Name : rq869.lcd
 Method File Name : 40%IPA, 1ml-min, 40min.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



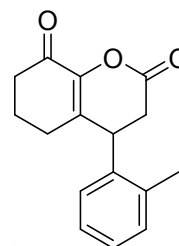
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.524	1327168	21028	1.075	1.668
2	33.434	122171204	1239507	98.925	98.332
Total		123498371	1260536	100.000	100.000

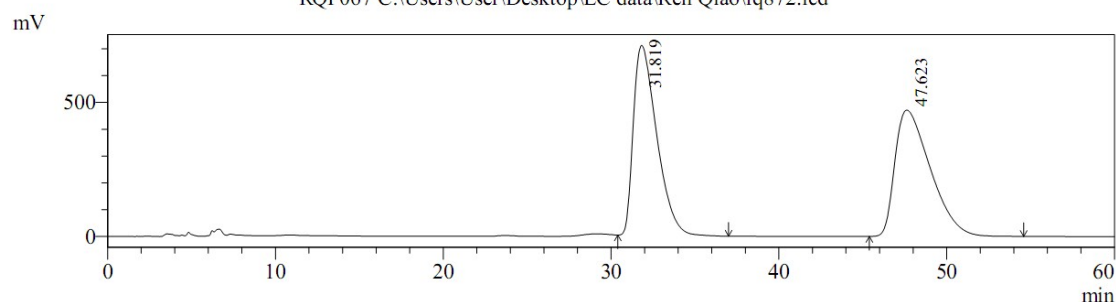
Racemic 3g

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq872.lcd
Acquired by : Admin
Sample Name : RQF067
Sample ID : RQ
Data File Name : rq872.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram
RQF067 C:\Users\User\Desktop\LC data\Ren Qiao\rq872.lcd



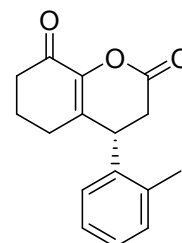
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.819	68701310	708777	49.820	60.061
2	47.623	69196720	471327	50.180	39.939
Total		137898029	1180104	100.000	100.000

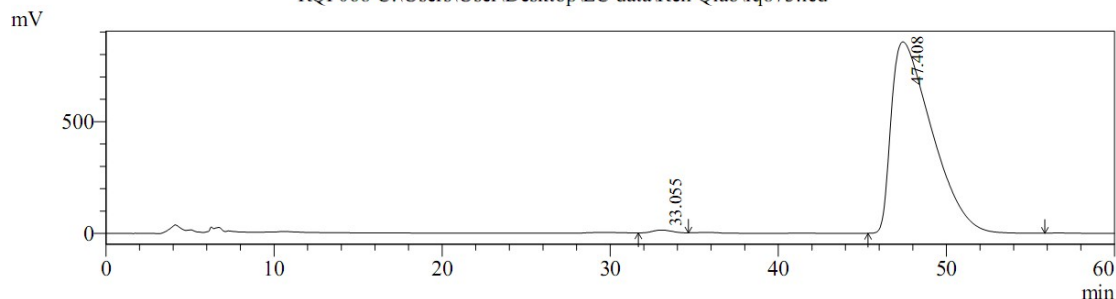
Enantiomeric enriched 3g

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq873.lcd
Acquired by : Admin
Sample Name : RQF068
Sample ID : RQ
Data File Name : rq873.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram
RQF068 C:\Users\User\Desktop\LC data\Ren Qiao\rq873.lcd



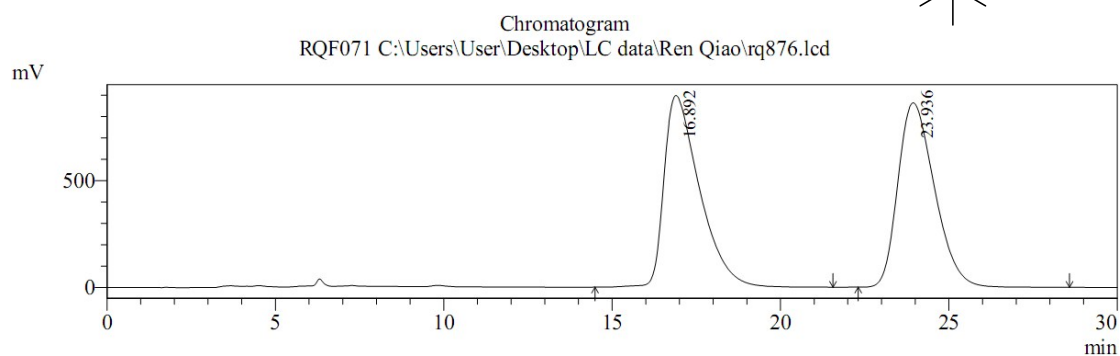
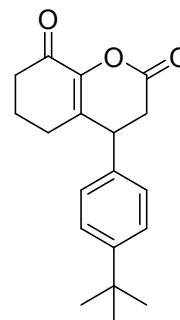
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.055	1038714	13464	0.716	1.548
2	47.408	144016561	856071	99.284	98.452
Total		145055275	869535	100.000	100.000

Racemic 3h

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq876.lcd
Acquired by : Admin
Sample Name : RQF071
Sample ID : RQ
Data File Name : rq876.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :AS-H column with IC guard column ;40%IPA ;1 ml/min



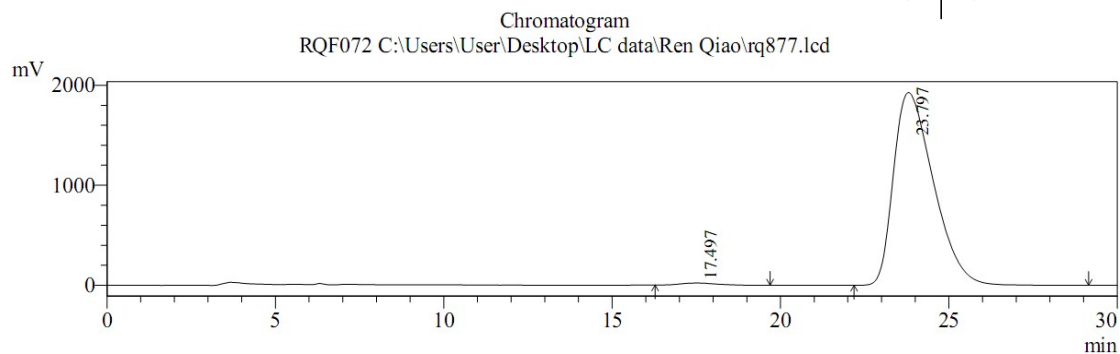
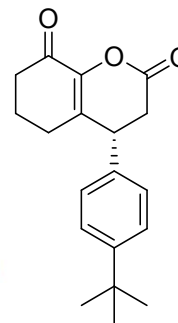
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.892	65795068	897646	50.230	50.974
2	23.936	65193182	863350	49.770	49.026
Total		130988250	1760996	100.000	100.000

Enantiomeric enriched 3h

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq877.lcd
Acquired by : Admin
Sample Name : RQF072
Sample ID : RQ
Data File Name : rq877.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :AS-H column with IC guard column ;40%IPA ;1 ml/min



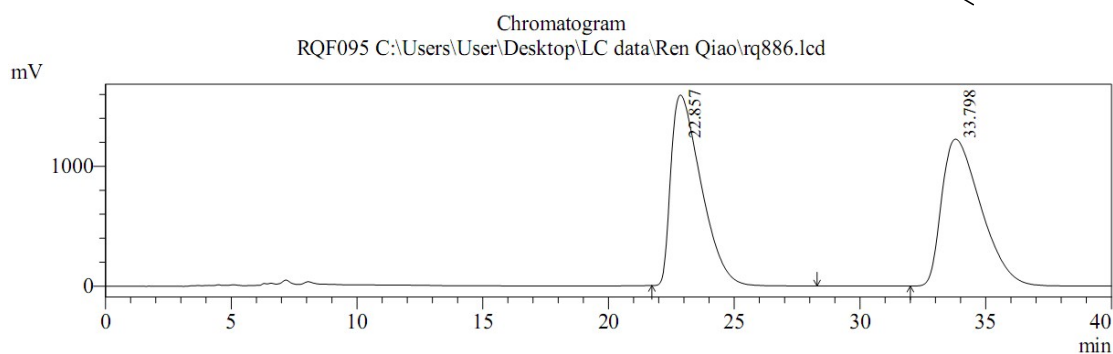
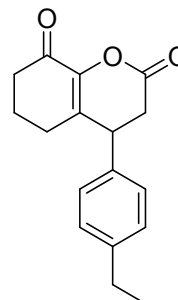
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.497	1544234	21760	0.964	1.117
2	23.797	158573619	1926962	99.036	98.883
Total		160117854	1948722	100.000	100.000

Racemic 3i

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq886.lcd
Acquired by : Admin
Sample Name : RQF095
Sample ID : RQ
Data File Name : rq886.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



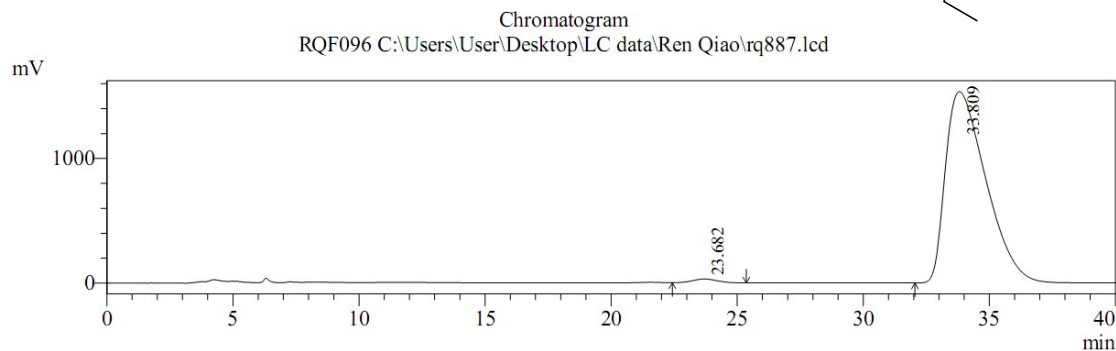
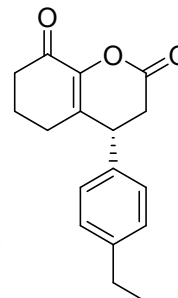
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.857	132528842	1589696	49.901	56.469
2	33.798	133054598	1225492	50.099	43.531
Total		265583441	2815188	100.000	100.000

Enantiomeric enriched 3i

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq887.lcd
Acquired by : Admin
Sample Name : RQF096
Sample ID : RQ
Data File Name : rq887.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



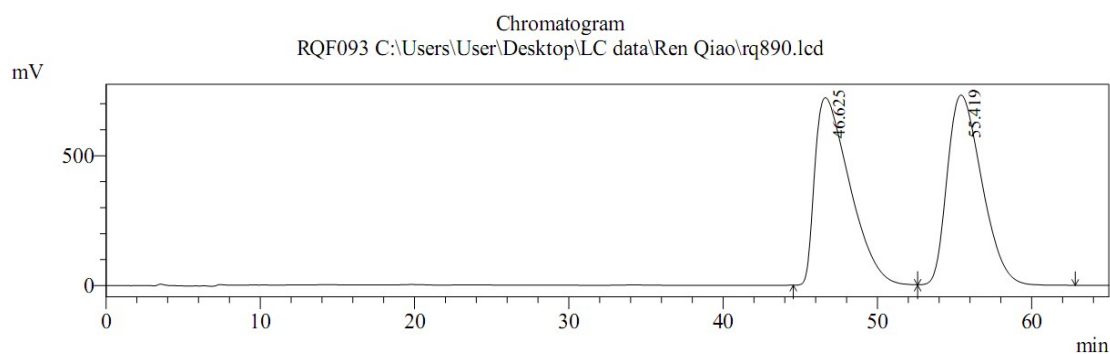
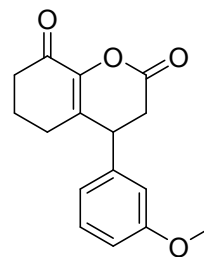
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.682	2012876	30193	1.160	1.928
2	33.809	171543355	1536233	98.840	98.072
Total		173556231	1566426	100.000	100.000

Racemic 3j

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq890.lcd
Acquired by : Admin
Sample Name : RQF093
Sample ID : RQ
Data File Name : rq890.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :AS-H column with IC guard column ;40%IPA ;1 ml/min



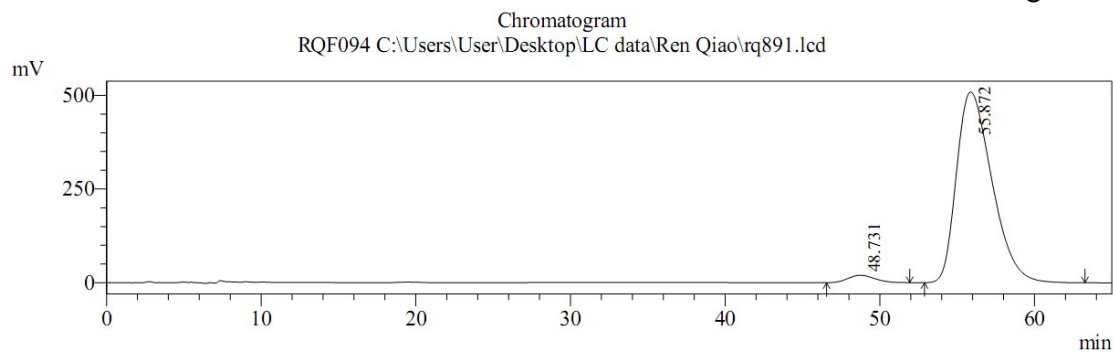
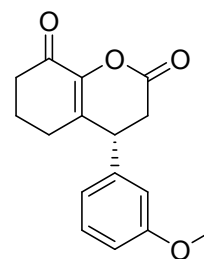
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	46.625	113953899	722193	49.906	49.655
2	55.419	114384305	732242	50.094	50.345
Total		228338204	1454435	100.000	100.000

Enantiomeric enriched 3j

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq891.lcd
Acquired by : Admin
Sample Name : RQF094
Sample ID : RQ
Data File Name : rq891.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description :AS-H column with IC guard column ;40%IPA ;1 ml/min



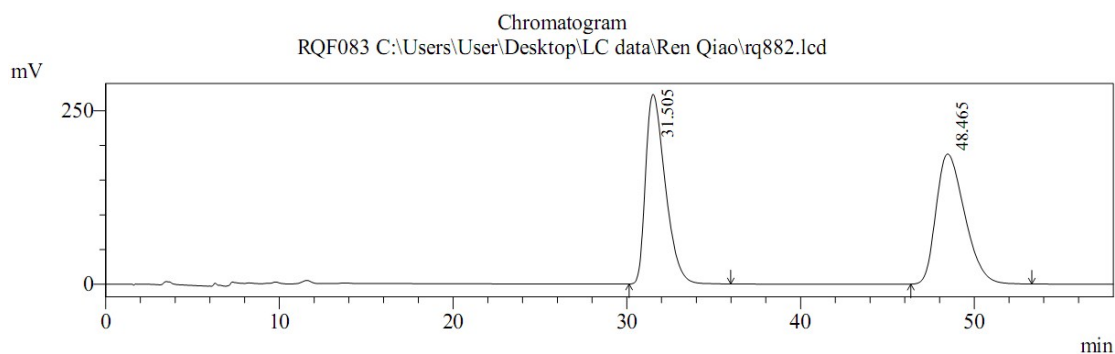
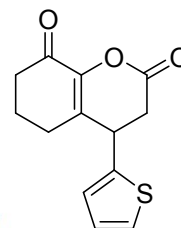
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	48.731	2395578	19828	2.872	3.754
2	55.872	81029889	508418	97.128	96.246
Total		83425467	528247	100.000	100.000

Racemic 3k

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq882.lcd
Acquired by : Admin
Sample Name : RQF083
Sample ID : RQ
Data File Name : rq882.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



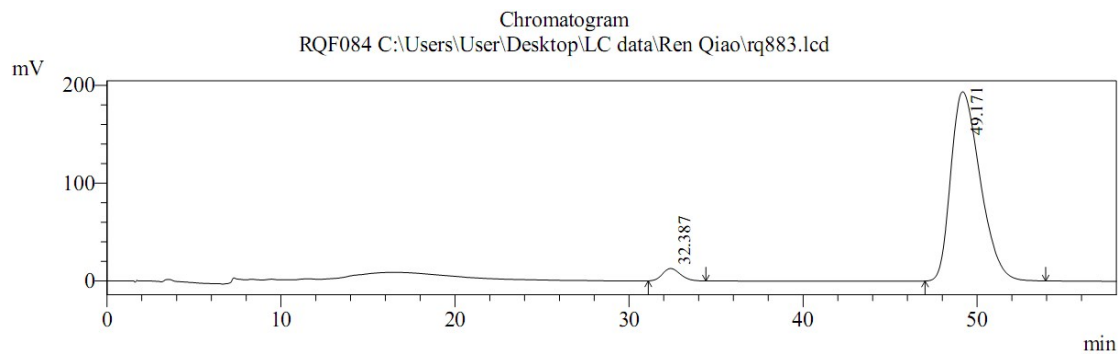
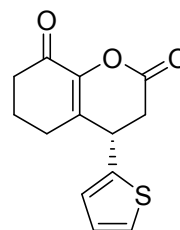
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	31.505	21481805	273059	50.043	59.320
2	48.465	21444927	187255	49.957	40.680
Total		42926731	460314	100.000	100.000

Enantiomeric enriched 3k

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq883.lcd
Acquired by : Admin
Sample Name : RQF084
Sample ID : RQ
Data File Name : rq883.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



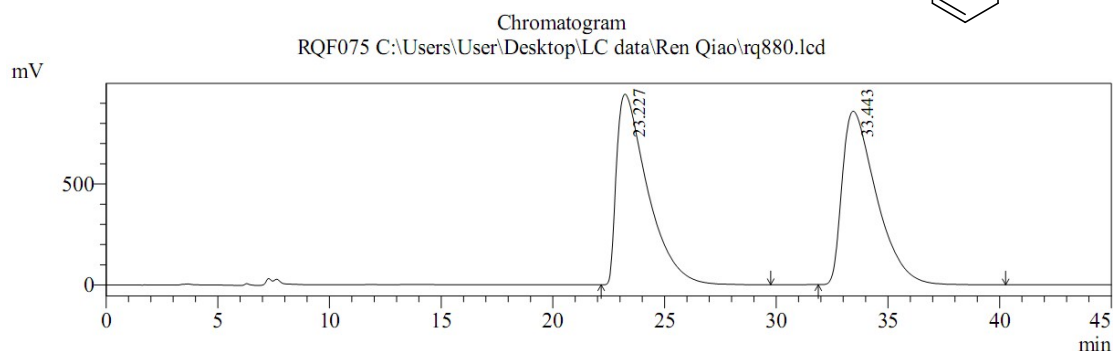
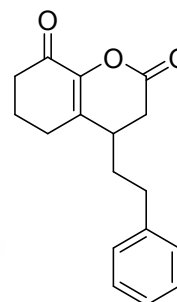
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.387	903406	12670	3.837	6.148
2	49.171	22642725	193424	96.163	93.852
Total		23546132	206094	100.000	100.000

Racemic 3I

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq880.lcd
Acquired by : Admin
Sample Name : RQF075
Sample ID : RQ
Data File Name : rq880.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



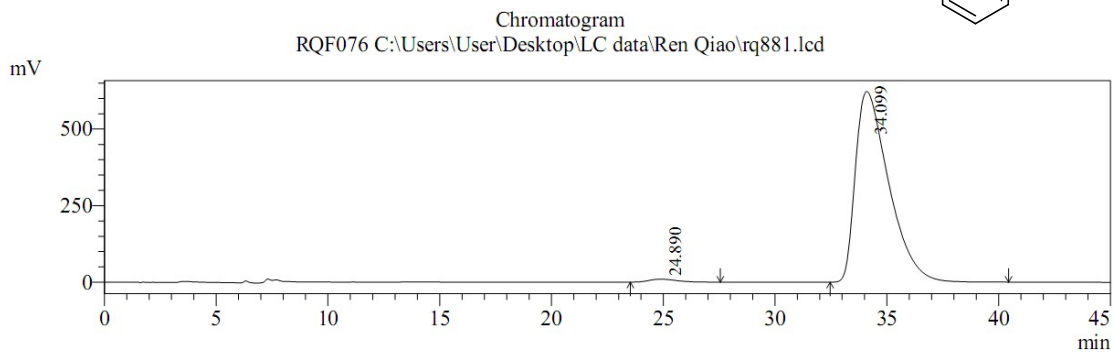
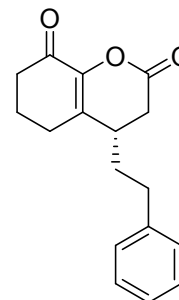
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.227	91443455	943659	49.986	52.343
2	33.443	91493288	859161	50.014	47.657
Total		182936743	1802820	100.000	100.000

Enantiomeric enriched 3I

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq881.lcd
Acquired by : Admin
Sample Name : RQF076
Sample ID : RQ
Data File Name : rq881.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



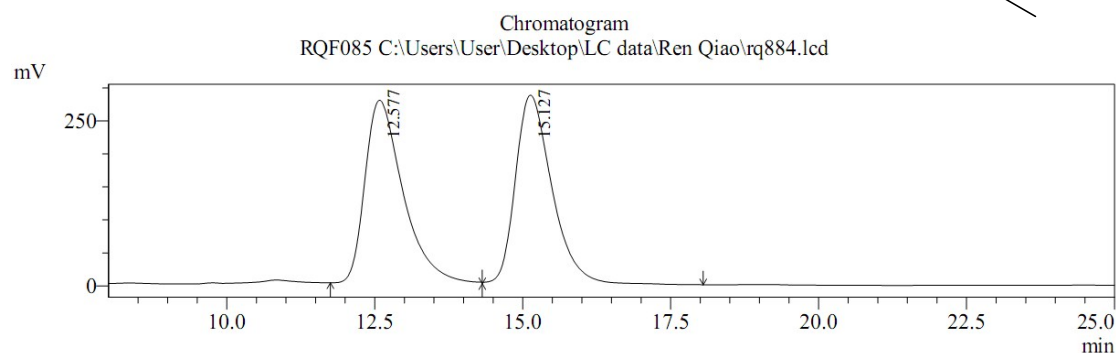
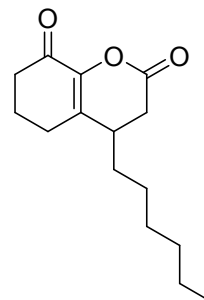
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.890	857472	10138	1.309	1.602
2	34.099	64647886	622841	98.691	98.398
Total		65505358	632979	100.000	100.000

Racemic 3m

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq884.lcd
Acquired by : Admin
Sample Name : RQF085
Sample ID : RQ
Data File Name : rq884.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



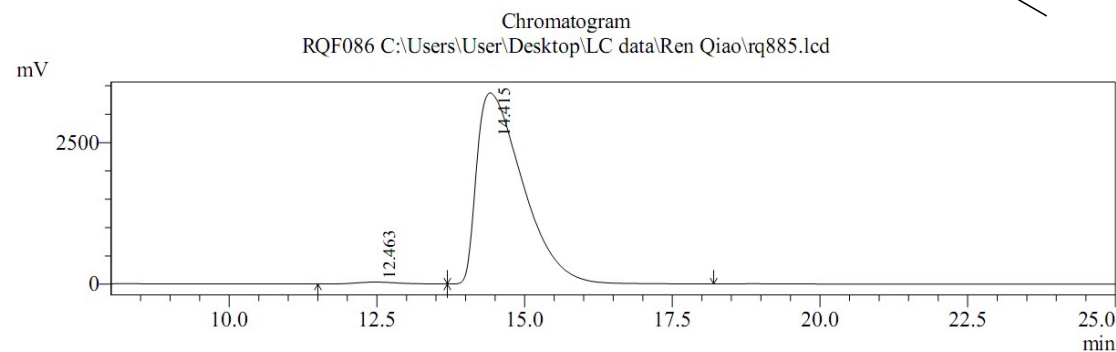
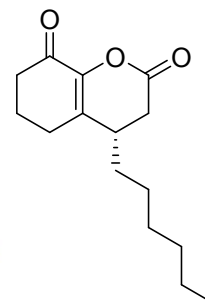
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.577	12423577	277306	50.289	49.206
2	15.127	12280659	286250	49.711	50.794
Total		24704236	563556	100.000	100.000

Enantiomeric enriched 3m

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq885.lcd
Acquired by : Admin
Sample Name : RQF086
Sample ID : RQ
Data File Name : rq885.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.463	1683380	34710	0.911	1.019
2	14.415	183016281	3373117	99.089	98.981
Total		184699661	3407827	100.000	100.000

Racemic 3n

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq878.lcd

Acquired by : Admin

Sample Name : RQF077

Sample ID : RQ

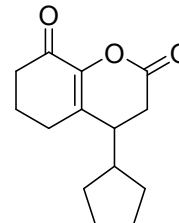
Data File Name : rq878.lcd

Method File Name : 40%IPA, 1ml-min, 40min.lcm

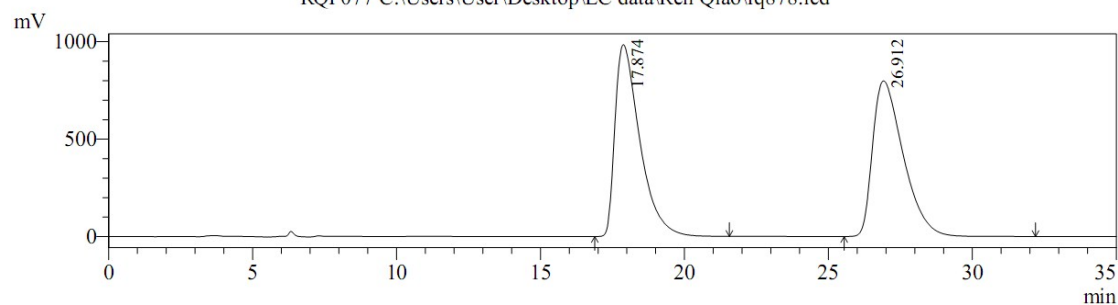
Batch File Name :

Report File Name : Default.lcr

Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram
RQF077 C:\Users\User\Desktop\LC data\Ren Qiao\rq878.lcd



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.874	58891572	983999	49.928	55.196
2	26.912	59060933	798745	50.072	44.804
Total		117952505	1782744	100.000	100.000

Enantiomeric enriched 3n

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq879.lcd

Acquired by : Admin

Sample Name : RQF078

Sample ID : RQ

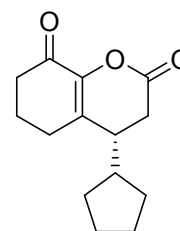
Data File Name : rq879.lcd

Method File Name : 40%IPA, 1ml-min, 40min.lcm

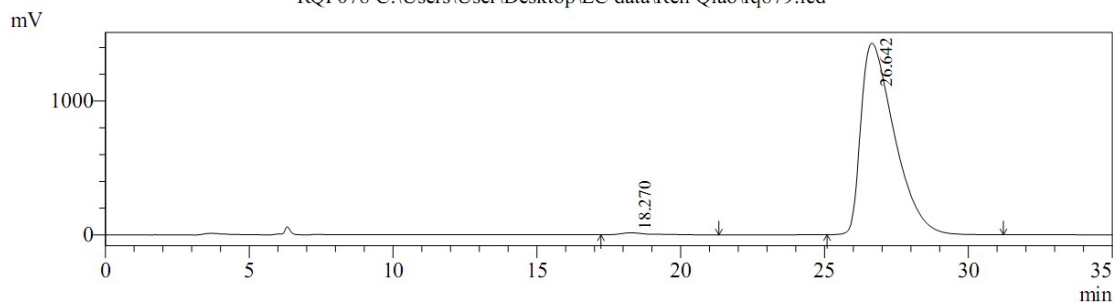
Batch File Name :

Report File Name : Default.lcr

Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Chromatogram
RQF078 C:\Users\User\Desktop\LC data\Ren Qiao\rq879.lcd



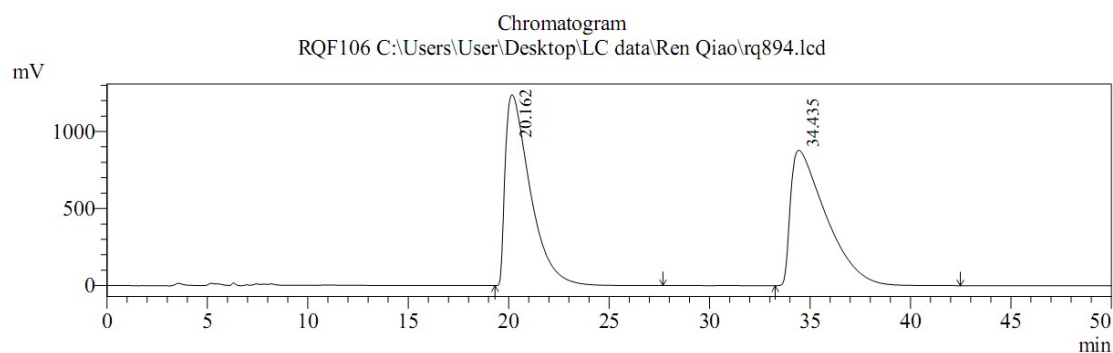
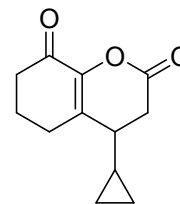
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.270	1040264	15260	0.891	1.056
2	26.642	115689646	1429696	99.109	98.944
Total		116729910	1444956	100.000	100.000

Racemic 3o

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq894.lcd
Acquired by : Admin
Sample Name : RQF106
Sample ID : RQ
Data File Name : rq894.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



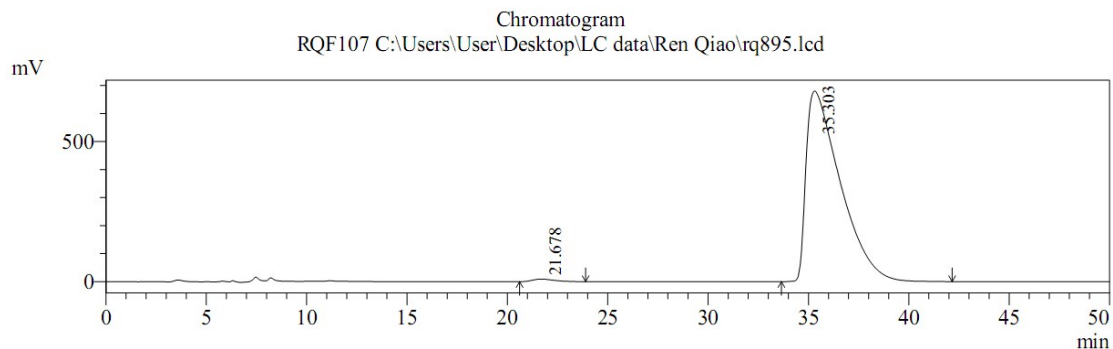
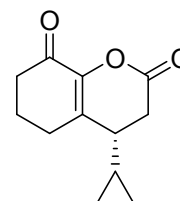
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.162	106967403	1238392	49.724	58.493
2	34.435	108152783	878765	50.276	41.507
Total		215120187	2117157	100.000	100.000

Enantiomeric enriched 3o

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq895.lcd
Acquired by : Admin
Sample Name : RQF107
Sample ID : RQ
Data File Name : rq895.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



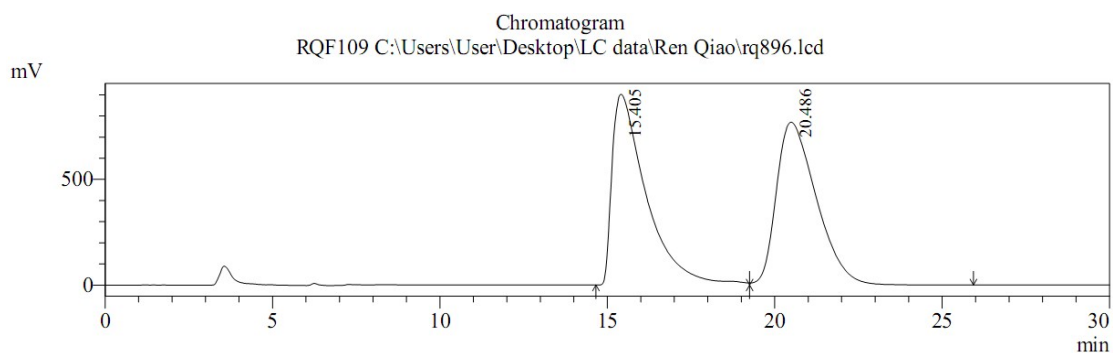
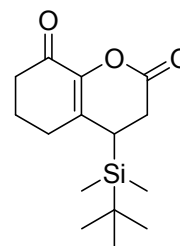
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.678	658965	9120	0.812	1.323
2	35.303	80518143	680075	99.188	98.677
Total		81177109	689195	100.000	100.000

Racemic 3p

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq896.lcd
Acquired by : Admin
Sample Name : RQF109
Sample ID : RQ
Data File Name : rq896.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



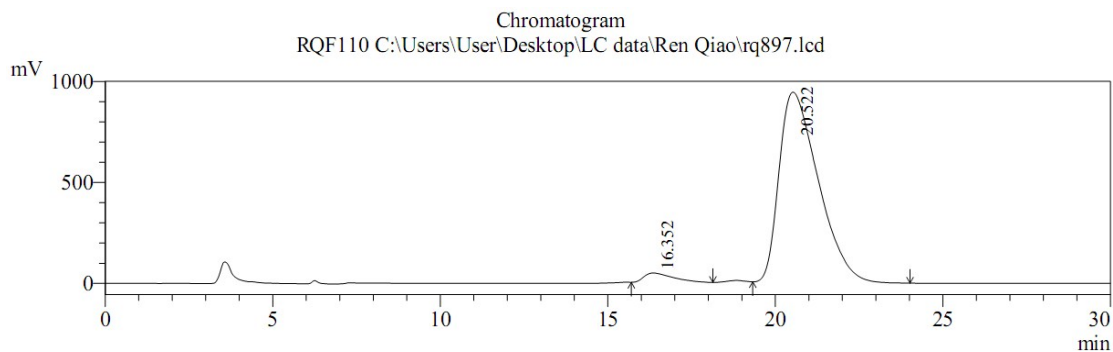
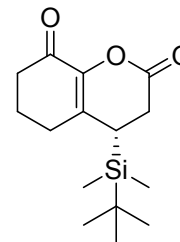
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.405	64302756	903012	50.103	53.956
2	20.486	64038072	770592	49.897	46.044
Total		128340827	1673604	100.000	100.000

Enantiomeric enriched 3p

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

C:\Users\User\Desktop\LC data\Ren Qiao\rq897.lcd
Acquired by : Admin
Sample Name : RQF110
Sample ID : RQ
Data File Name : rq897.lcd
Method File Name : 40%IPA, 1ml-min, 40min.lcm
Batch File Name :
Report File Name : Default.lcr
Description : AS-H column with IC guard column ;40%IPA ;1 ml/min



Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.352	2772977	46206	3.499	4.680
2	20.522	76482027	941014	96.501	95.320
Total		79255004	987220	100.000	100.000