

Supporting Information

A Novel Access for Indole-3-substituted Dihydrocoumarins in Artificial Sweetener Saccharin based Functional Ionic Liquid

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General: All the reagents and solvents were purchased from Sigma-Aldrich or Merck chemical Co. and were used directly without any further purification. Organic solvents were concentrated under reduced pressure on a Büchi rotary evaporator. The progress of reaction was checked by thin-layer chromatography. The plates were visualized first with UV illumination followed by iodine. ¹H NMR spectra were recorded at 200 or 300 MHz using Bruker DRX-200 or 300 spectrometer and are reported in parts per million (ppm) on the δ scale relative to TMS as an internal standard. Coupling constants (*J*) reported in Hz. ¹³C NMR spectra were recorded at 50 or 75 MHz. Mass spectra were obtained using JEOL SX-102 (ESI) instrument. Elemental analysis was performed using a Perkin-Elmer autosystem XL analyzer. All melting points are uncorrected.

Representative experimental procedure for the functional ionic liquid [bmim][Sac]

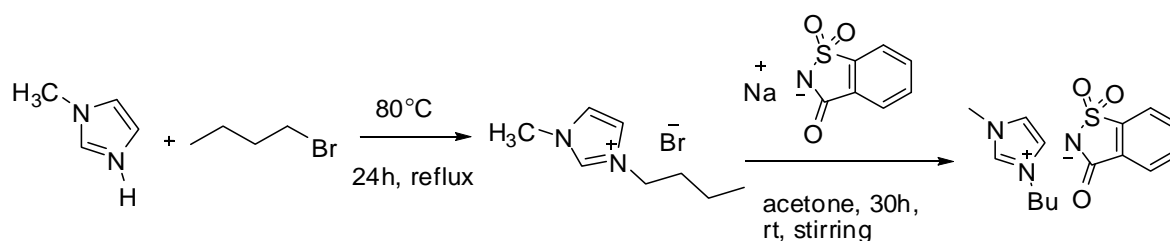


Figure 1 Representative procedure for synthesis of [bmim][Sac].

Representative experimental procedure for the sodium salt of saccharinate.

A three-neck 100 ml round bottom flask was fitted with overhead stirrer, condenser and inlet/outlet for nitrogen atmosphere. The flask was charged with 7.32g saccharin (0.04 mol) and 2.16g (0.04 mol) anhydrous sodium methoxide in 50 ml anhydrous methanol. The mixture was stirred and heated to reflux for about 10-20 minutes under nitrogen. Most of the solids went into solution. The system was then set-up for distillation. Methanol was removed under reduced pressure. Colorless solids of sodium saccharin (yield 90%) remained in the flask.

Preparation of the 1-n-butyl-3- methylimidazolium saccharinate [bmim][Sac]:

The sodium saccharinate (27.0 g, 0.112 mol) was added into a solution of 1-n-butyl-3-methylimidazolium bromide (BMImBr) (24.6 g, 0.112 mol) in 100 mL acetone at room temperature. After stirring for 24 h, the reaction mixture was filtered through a plug of celite. The volatiles were removed under reduced pressure overnight. Viscous oil was obtained. ¹H NMR (DMSO-d₆, 300 MHz) δ_H: 0.89 (t, *J* = 7.6 Hz, 3H), 1.28-1.21(m, 2H), 1.80-1.73 (m, 2H), 3.86 (s, 3H), 4.17 (t, *J* = 7.1 Hz, 2H), 7.71–7.78 (m, 4H), 7.75 (s, 1H), 7.82 (s, 1H), 9.18 (s, 1H). ¹³C NMR (DMSO-d₆, 50 MHz) δ_C: 13.20, 18.70, 31.30, 35.71, 48.43, 119.98, 122.20, 122.45, 123.51, 128.25, 130.99, 131.46, 133.00, 136.47, 167.88.

General procedure for the synthesis of indole-3-dihydrocoumarin derivatives via one pot multicomponent reaction of Indole, Salicylaldehyde and Meldrum acid:

A mixture of salicylaldehyde or 4-OMe-salicylaldehyde (1 mmol), meldrum acid (1 mmol), indole derivatives (1 mmol) and [bmim]Sac (2 ml) in a 50mL flask, and was vigorously stirred at room temperature (25–29 °C) for 8-10h, the progress of the reaction was monitored by TLC. Upon completion, the reaction mixture was extracted with ethyl acetate (3×20 ml). Then the combined organic part was dried over Na₂SO₄ and the solvent was evaporated to yield a crude residue. The crude products thus obtained were purified by column chromatography (silica gel, 60-120 mesh; ethyl acetate/petroleum ether). All desired products were characterized by ¹H NMR, ¹³C NMR and mass spectra.

Reusability of the [C₄MIM][Sac]: After completion of the reaction, the reaction mixture was diluted with EtOAc (20 mL) followed by addition of a 5 mL of water. The EtOAc layer was separated. The EtOAc extract was dried (Na₂SO₄) and concentrated under vacuo to obtain the crude product. The aqueous extract/layer containing the ionic liquid was concentrated under vacuum at 80° C for 60 min to recover the IL which was found to be identical (spectral data) with an authentic sample of [bmim][Sac] (unused ionic liquid). The recovered IL was reused for 4 successive batches of reactions at rt to afford crude product after usual work-up (Table 1).

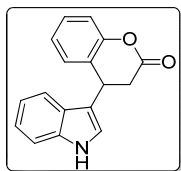
Table 1 Reusability of 1-methyl-3-(n-butyl) imidazolium Saccharinate [(C₄MIM)Sac] ionic liquid in indole -3 dihydricoumarin synthesis

Run	1	2	3	4
Yield (%)	93	91	87	85

^aReaction conditions: Indole (1.0 mmol), meldrum acid (1.0 mmol), salicylaldehyde (1.0 mmol), ionic liquid (2.0 mL), rt, 8 h. ^b Isolated yield.

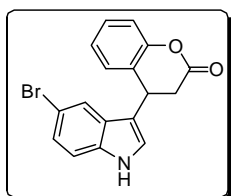
Characterization data for synthesized compounds:

4-(1H-indol-3-yl)chroman-2-one (a)



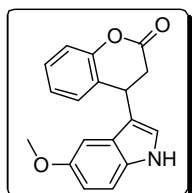
Physical state: oily. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.20 (s, 1H), 7.84 (d, $J = 7.7$ Hz, 1H), 7.37 (d, $J = 8.1$ Hz, 1H), 7.31-7.18 (m, 1H), 7.13-6.79 (m, 5H) 6.79 (s, 1H), 4.63 (t, $J = 6.18$ Hz, 1H), 3.26 (dd, $J = 7.6, 15.8$ Hz, 1H), 3.09 (dd, $J = 5.6, 15.8$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 168.6, 151.8, 136.9, 128.7, 128.4, 126.2, 125.9, 124.8, 122.7, 122.5, 120.0, 119.0, 117.2, 115.2, 111.8, 36.5, 32.8. Molecular formula: $\text{C}_{17}\text{H}_{13}\text{NO}_2$. ESI MS (m/z) = 264 (M+H). Analysis calculated for $\text{C}_{17}\text{H}_{13}\text{NO}_2$: C 77.55, H 4.98, N 5.32. Found: C 77.53, H 4.97, N 5.29.

4-(5-bromo-1H-indol-3-yl)chroman-2-one (b)



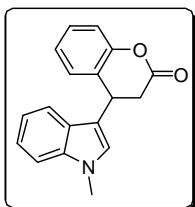
Physical state: oily. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.29 (s, 1H), 7.54 (s, 1H), 7.25-7.12 (m, 3H), 7.05-6.95 (m, 3H), 6.67 (d, $J = 2.1$ Hz, 1H), 4.49 (t, $J = 6.1$ Hz, 1H), 3.11 (dd, $J = 6.9, 15.7$ Hz, 1H), 2.97 (dd, $J = 5.6, 15.7$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 168.4, 151.7, 135.5, 129.0, 128.2, 127.6, 125.7, 125.6, 125.0, 123.7, 121.4, 117.3, 114.9, 113.3, 113.2, 36.5, 32.6. Molecular formula: $\text{C}_{17}\text{H}_{12}\text{BrNO}_2$, ESI MS (m/z): 342 (M+H). Analysis calculated for $\text{C}_{17}\text{H}_{12}\text{BrNO}_2$: C 59.67, H 3.53, N 4.09. Found, C 59.68, H 3.50, N 4.10

4-(5-methoxy-1H-indol-3-yl)chroman-2-one (c)



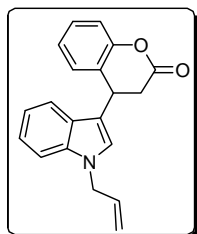
Physical state: White solid, mp: 155-157°C. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.01 (s, 1H), 7.32-7.21 (m, 2H), 7.15 (t, $J = 5.6$ Hz, 3H), 7.16-7.05 (m, 2H), 6.91 (s, 1H), 4.61 (t, $J = 6.7$ Hz, 1H), 3.81 (s, 3H), 3.23 (dd, $J = 7.6, 15.7$ Hz, 1H), 3.08 (dd, $J = 5.6, 15.7$ Hz, 1H). ^{13}C NMR (50 MHz, CDCl_3) δ_{C} : 168.4, 154.2, 151.7, 131.9, 128.6, 128.2, 126.3, 126.0, 124.7, 123.0, 117.1, 114.7, 112.6, 112.4, 100.9, 56.0, 36.3, 32.6. Chemical Formula: $\text{C}_{18}\text{H}_{15}\text{NO}_3$. ESI MS (m/z): 294 (M+H). Analysis calculated for $\text{C}_{18}\text{H}_{15}\text{NO}_3$: C 73.71, H 5.15, N 4.78. Found; C 73.73, H 5.11, N 4.80

4-(1-methyl-1H-indol-3-yl)chroman-2-one (d)



Physical state: oily: ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.41 (d, $J = 7.9$ Hz, 1H), 7.26-7.15 (m, 3H), 7.10-6.97 (m, 4H), 6.59 (s, 1H), 4.56 (t, $J = 6.1$ Hz, 1H), 3.63 (s, 3H), 3.15 (dd, $J = 7.2, 15.7$ Hz, 1H), 2.98 (dd, $J = 5.6, 15.7$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ_{C} : 29.7, 32.6, 36.5, 109.7, 113.7, 117.1, 118.9, 119.4, 122.1, 124.6, 126.9, 126.2, 128.2, 128.5, 12.5, 137.5, 151.7, 168.2. Molecular formula: $\text{C}_{18}\text{H}_{15}\text{NO}_2$. ESI MS (m/z): 278 (M+H). Analysis calculated for $\text{C}_{18}\text{H}_{15}\text{NO}_2$: C 77.96, H 5.45, N 5.05. Found; C 77.92, H 5.46, N 5.08.

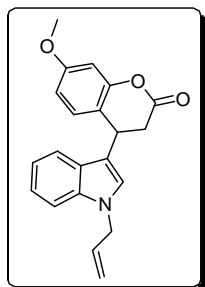
4-(1-allyl-1H-indol-3-yl)chroman-2-one (e)



Physical state: oily. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.52 (d, $J = 7.7$ Hz, 1H), 7.36 (d, $J = 7.6$ Hz, 3H), 7.29-7.10 (m, 4H), 6.77 (s, 1H), 6.02-5.93 (m, 1H), 5.21 (d, $J = 10.1$

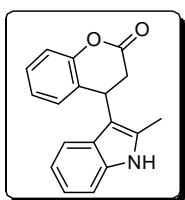
Hz, 1H), 5.09 (d, $J = 17.1$ Hz, 1H), 4.68 (d, $J = 9.9$ Hz, 2H), 4.31 (t, $J = 6.8$ Hz, 1H), 3.27 (dd, $J = 7.7, 15.7$ Hz, 1H), 3.11 (dd, $J = 5.4, 15.7$ Hz, 1H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 168.2, 150.1, 136.9, 135.2, 133.1, 128.5, 128.2, 126.4, 125.8, 124.6, 122.2, 119.5, 119.0, 117.5, 117.1, 114.0, 110.1, 48.8, 36.4, 29.7. Molecular formula: $\text{C}_{20}\text{H}_{17}\text{NO}_2$. ESI MS (m/z):= 304 (M+H). Analysis calculated for $\text{C}_{20}\text{H}_{17}\text{NO}_2$: C 79.19, H 5.65, N 4.62. Found; C 79.15, H 5.66, N 4.65.

4-(1-allyl-1H-indol-3-yl)-7-methoxychroman-2-one (f)



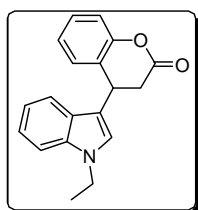
Physical state: oily: ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.40 (d, $J = 7.9$ Hz, 1H), 7.26-7.15 (m, 2H), 7.03 (t, $J = 6.41$, 1H), 6.95 (d, $J = 11.4$ Hz, 1H), 6.65 (s, 1H), 6.62 (d, $J = 2.4$ Hz, 1H), 6.57-6.53 (m, 1H), 5.91-5.80 (m, 1H), 5.21 (d, $J = 9.3$, 1H), 5.10 (d, $J = 9.3$ Hz, 1H), 4.41 (d, $J = 4.05$ Hz, 2H), 4.23 (t, $J = 6.6$ Hz, 1H), 3.73 (s, 3H), 3.14 (dd, $J = 7.6, 15.7$ Hz, 1H), 2.98 (dd, $J = 5.6, 15.7$ Hz, 1H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 168.4, 160.0, 152.5, 137.1, 133.4, 128.9, 126.0, 122.3, 119.6, 119.2, 118.0, 117.6, 115.5, 114.6, 110.8, 110.3, 102.7, 55.7, 49.0, 36.9, 32.2. Molecular formula: $\text{C}_{21}\text{H}_{19}\text{NO}_3$, ESI MS (m/z):= 334 (M+H). Analysis calculated for $\text{C}_{21}\text{H}_{19}\text{NO}_3$: C 75.66, H 5.74, N 4.20. Found: C 75.62, H 5.70, N 4.21.

4-(2-methyl-1H-indol-3-yl)chroman-2-one (g)



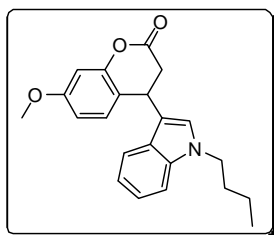
Physical state: White solid, mp: 108-110 °C. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.99 (s, 1H), 7.36 (d, $J = 7.2$ Hz, 1H), 7.29 (d, $J = 6.74$, 2H), 7.19-7.07 (m, 2H), 7.04-6.94 (m, 3H), 4.68 (t, $J = 5.9$ Hz, 1H), 3.29 (dd, $J = 13.2, 16.0$ Hz, 1H), 2.99 (dd, $J = 5.2, 16.0$ Hz, 1H), 2.39 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ_{c} : 168.7, 151.9, 135.7, 132.8, 128.5, 128.1, 126.4, 125.6, 124.5, 121.4, 119.5, 119.0, 116.9, 110.7, 108.6, 35.9, 31.9, 12.0. Chemical Formula: $\text{C}_{18}\text{H}_{15}\text{NO}_2$, ESI MS (m/z):= 278 (M+H). Analysis calculated for $\text{C}_{18}\text{H}_{15}\text{NO}_2$: C, 77.96; H, 5.45; N, 5.05. Found C, 77.99; H, 5.41; N, 5.07

4-(1-ethyl-1H-indol-3-yl)chroman-2-one (h)



Physical state: oily: ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.55 (d, $J = 7.8$ Hz, 1H), 7.42-7.27 (m, 4H), 7.21-7.10 (m, 3H), 6.81 (s, 1H), 4.66 (t, $J = 6.1$ Hz, 1H), 4.17-4.10 (m, 2H), 3.13 (dd, $J = 5.6, 15.8$ Hz, 1H), 3.29 (dd, $J = 7.5, 15.8$ Hz, 1H), 1.46 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ_{c} : 168.3, 151.7, 136.6, 128.5, 128.2, 126.4, 126.3, 125.1, 124.6, 122.0, 119.3, 119.1, 117.1, 113.7, 109.9, 41.0, 36.5, 32.7, 15.4. Molecular formula: $\text{C}_{19}\text{H}_{17}\text{NO}_2$. ESI MS (m/z):= 292 (M+H). Analysis calculated for $\text{C}_{19}\text{H}_{17}\text{NO}_2$: C 78.33, H 5.88, N 4.81. Found C 78.30, H 5.90, N 4.80.

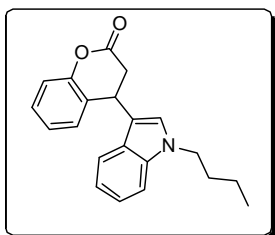
4-(1-butyl-1H-indol-3-yl)-7-methoxychroman-2-one (i)



Physical state: oily. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.50 (d, $J = 7.9$ Hz, 1H), 7.37 (d, $J = 7.2$ Hz, 1H), 7.24 (d, $J = 7.5$ Hz, 1H), 7.14-7.05 (m, 2H), 6.76 (s, 1H), 6.73 (d, $J = 2.4$ Hz, 1H), 6.67-6.63 (m, 1H), 4.61 (t, $J = 8.9$ Hz, 1H), 4.07 (t, $J = 7.1$ Hz, 2H), 3.84 (s, 3H), 3.24 (dd, $J = 7.7, 15.7$ Hz, 1H), 3.08 (dd, $J = 5.6, 15.7$ Hz, 1H), 1.85-1.75 (m, 2H), 1.38-1.30 (m, 2H), .95 (t, $J = 4.4$ Hz,

3H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 168.3, 159.8, 152.4, 136.8, 128.9, 126.3, 125.8, 121.9, 119.2, 119.1, 118.1, 113.9, 110.6, 110.0, 102.5, 55.6, 46.2, 36.8, 32.3, 32.0, 20.2, 13.8. Chemical Formula: $\text{C}_{22}\text{H}_{23}\text{NO}_3$. ESI MS (m/z):= 350 (M+H). Analysis calculated for $\text{C}_{22}\text{H}_{23}\text{NO}_3$: C, 75.62; H, 6.63; N, 4.01, Found, C, 75.64, H, 6.62, N, 4.05.

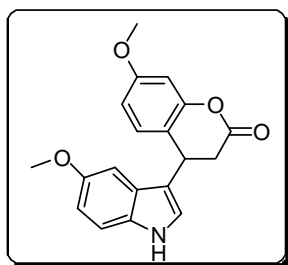
4-(1-butyl-1H-indol-3-yl)chroman-2-one (j)



Physical state: oily: ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.52 (d, $J = 7.9$ Hz, 1H), 7.39-7.24 (m, 2H), 7.18 (d, $J = 4.7$ Hz, 2H), 7.10 (t, $J = 8.9$ Hz, 3H), 6.77 (s, 1H), 4.68 (t, $J = 5.9$ Hz, 1H), 4.07 (t, $J = 7.1$ Hz, 2H), 3.27 (dd, $J = 7.7, 15.7$ Hz, 1H), 3.11 (dd, $J = 5.6, 15.7$ Hz, 1H), 1.85-1.75 (m, 2H), 1.38-1.30 (m, 2H), 94 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 168.3, 151.6, 137.9, 128.5, 128.2, 126.3, 126.2, 125.8, 124.6, 121.9, 119.2, 119.0, 117.0, 113.4, 109.9, 46.2, 36.5, 32.6, 32.3, 20.2, 13.7. Chemical Formula:

$\text{C}_{21}\text{H}_{21}\text{NO}_2$. ESI MS (m/z):= 320 (M+H). Analysis calculated for $\text{C}_{21}\text{H}_{21}\text{NO}_2$: C, 78.97; H, 6.63; N, 4.39; Found; C, 78.99; H, 6.62; N, 4.43.

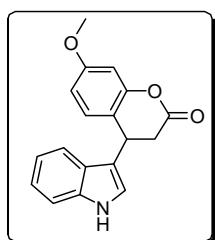
7-methoxy-4-(5-methoxy-1H-indol-3-yl)chroman-2-one (k)



Physical state: White solid, mp: 136-138°C. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.05 (s, 1H), 7.25 (s, 1H), 7.03 (d, $J = 7.43$ Hz, 1H), 6.88 (d, $J = 9.6$ Hz, 2H), 6.78 (d, $J = 2.0$ Hz, 1H), 6.63 (d, $J = 2.16$, 1H), 6.61 (d, $J = 2.16$ Hz, 1H), 4.55 (t, $J = 6.3$ Hz, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 3.19 (dd, $J = 7.6$ Hz, 15.7 Hz, 1H), 3.05 (dd, $J = 5.6, 15.7$ Hz, 1H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 168.3, 159.8, 154.1, 152.3, 131.9, 128.2, 126.2, 122.9, 117.8, 115.2, 112.5, 112.3, 110.6, 102.5, 100.9, 56.0, 55.6, 36.5, 32.0. Chemical Formula:

$\text{C}_{19}\text{H}_{17}\text{NO}_4$. ESI MS (m/z):= 324 (M+H). Analysis calculated for $\text{C}_{19}\text{H}_{17}\text{NO}_4$: C 70.58, H 5.30, N 4.33, Found; C 70.57, H 5.344, N 4.35.

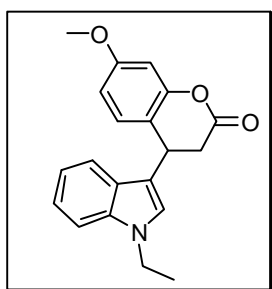
4-(1H-indol-3-yl)-7-methoxychroman-2-one (l)



Physical state: oily: ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.08 (s, 1H), 7.40 (d, $J = 7.8$ Hz, 1H), 7.29 (d, $J = 8.1$ Hz, 1H), 7.17-7.11 (m, 1H), 7.02 (t, $J = 7.1$ Hz, 1H), 6.94 (d, $J = 11.4$, 1H), 6.73 (d, $J = 2.2$ Hz, 1H), 6.61 (d, $J = 2.4$ Hz, 1H), 6.55-6.52 (m, 1H), 4.59 (t, $J = 3.5$ Hz, 1H), 3.72 (s, 3H), 3.41 (dd, $J = 7.6, 15.7$ Hz, 1H), 2.97 (dd, $J = 5.6, 15.7$ Hz, 1H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 166.5, 160.0, 152.5, 137.0, 128.9, 125.9, 122.7, 122.4, 120.0, 119.0, 118.0, 115.7, 111.8, 10.8, 102.7, 55.7, 36.8, 32.2. Molecular formula- $\text{C}_{18}\text{H}_{15}\text{NO}_3$, ESI MS (m/z):= 294 (M+H). Analysis

calculated for $\text{C}_{18}\text{H}_{15}\text{NO}_3$: C 73.71, H 5.15, N 4.78. Found, C 73.67, H 5.16, N 4.75

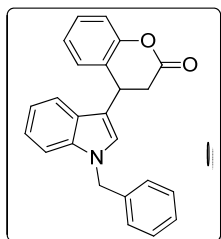
4-(1-ethyl-1H-indol-3-yl)-7-methoxychroman-2-one (m)



Physical state: White solid, mp: 96-98°C: ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.51 (d, $J = 7.8$ Hz, 1H), 7.38 (d, $J = 8.2$ Hz, 1H), 7.34-7.27 (m, 1H), 7.16-7.06 (m, 2H), 6.78 (s, 1H), 6.73 (d, $J = 2.2$ Hz, 1H), 6.68-6.64 (m, 1H), 4.62

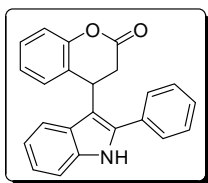
(t, $J = 6.1$ Hz, 1H), 4.17-4.09 (m, 2H), 3.84 (s, 3H), 3.25 (dd, $J = 7.5, 15.7$ Hz, 1H), 3.09 (dd, $J = 5.64, 15.7$ Hz, 1H), 1.45 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ_{c} : 168.3, 159.8, 152.3, 136.5, 128.8, 126.3, 125.1, 121.9, 119.2, 119.1, 118.0, 114.1, 110.6, 109.8, 102.5, 55.6, 41.0, 36.7, 32.0, 15.4. Molecular formula: $\text{C}_{20}\text{H}_{19}\text{NO}_3$. ESI MS (m/z):= 322 (M+H). Analysis calculated for $\text{C}_{20}\text{H}_{19}\text{NO}_3$: C 74.75, H 5.96, N 4.36. Found C 74.78, H 5.92, N 4.33.

4-(1-benzyl-1H-indol-3-yl)chroman-2-one (n)



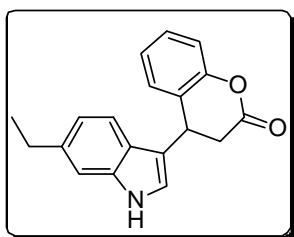
Physical state: oily. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 7.48 (d, $J = 7.7$ Hz, 1H), 7.26-7.19 (m, 6H), 7.16 (d, $J = 7.2$ Hz, 1H), 7.11 (t, $J = 5.2$ Hz, 1H), 7.09 (d, $J = 3.3$ Hz, 1H), 7.05 (t, $J = 5.19$ Hz, 3H), 6.78 (s, 1H), 5.21 (s, 2H), 4.43 (t, $J = 6.1$ Hz, 1H), 3.72 (dd, $J = 7.9, 15.8$ Hz, 1H), 3.05 (dd, $J = 5.5, 15.8$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ_{c} : 168.2, 151.7, 137.2, 137.1, 128.7, 128.6, 128.3, 128.2, 127.7, 126.5, 126.4, 126.1, 124.6, 122.4, 119.7, 119.2, 117.0, 114.2, 110.4, 50.1, 36.4, 32.6. Molecular formula $\text{C}_{24}\text{H}_{19}\text{NO}_2$. ESI MS (m/z):= 354 (M+H). Analysis calculated for $\text{C}_{24}\text{H}_{19}\text{NO}_2$: C 81.56, H 5.42, N 3.96 Found: C 81.59, H 5.40, N 3.98.

4-(2-phenyl-1H-indol-3-yl)chroman-2-one (o)



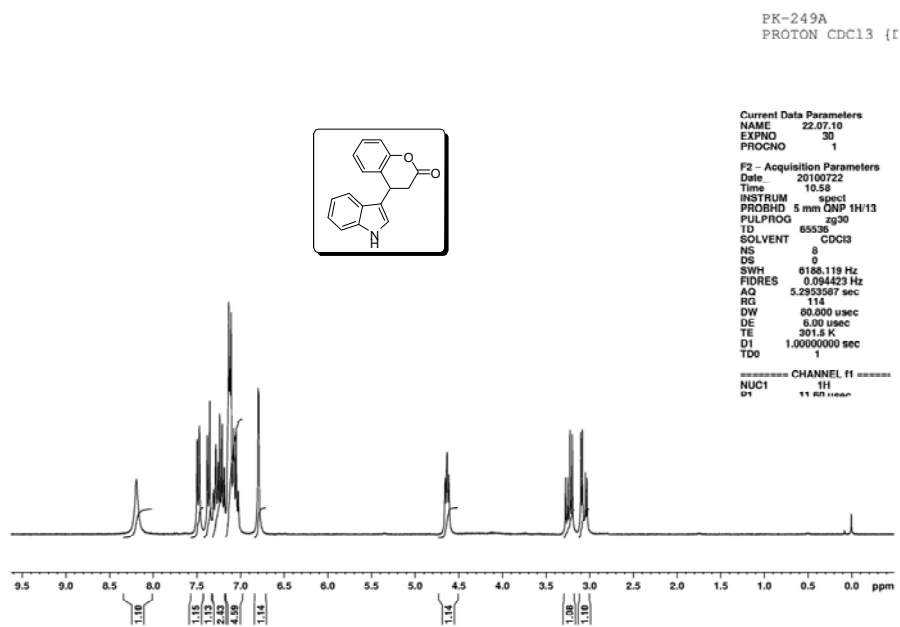
Physical state: White solid, mp: $>250^\circ\text{C}$. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.30 (s, 1H), 7.48 (d, $J = 6.4$ Hz, 5H), 7.32-7.22 (m, 3H), 7.16 (t, $J = 8.9$ Hz, 2H), 7.37 (d, $J = 2.9$ Hz, 3H), 4.84 (dd, $J = 4.6, 14.1$ Hz, 1H), 3.41 (t, $J = 15.9$ Hz, 1H), 2.98 (dd, $J = 4.8, 15.9$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ_{c} : 167.8, 151.3, 137.0, 136.6, 132.2, 128.8, 128.5, 128.3, 128.0, 127.6, 126.2, 125.8, 124.2, 121.5, 119.9, 118.8, 116.4, 111.7, 108.4, 35.0, 31.4. Molecular formula: $\text{C}_{23}\text{H}_{17}\text{NO}_2$. ESI MS (m/z):= 340 (M+H). Analysis calculated for $\text{C}_{23}\text{H}_{17}\text{NO}_2$: C 81.40, H 5.05, N 4.13. Found; C 81.41, H 5.09, N 4.16.

4-(6-ethyl-1H-indol-3-yl)chroman-2-one (p)

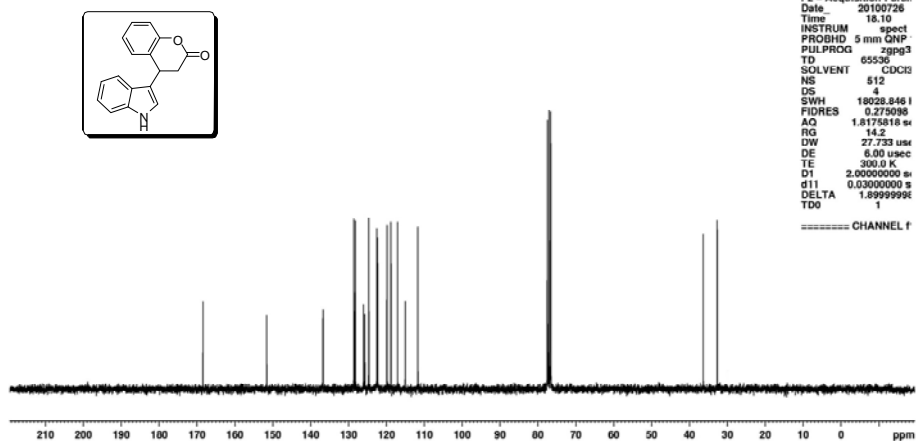


Physical state: oily. ^1H NMR (300 MHz, CDCl_3) δ_{H} : 8.27 (s, 1H), 7.42-7.34 (m, 2H), 7.18 (s, 1H), 7.16-7.10 (m, 4H), 6.81 (s, 1H), 4.68 (t, $J = 6.2$ Hz, 1H), 3.30 (dd, $J = 7.6$ Hz, 15.8 Hz, 1H), 3.12 (dd, $J = 11.2, 15.8$ Hz, 1H), 2.92-2.84 (m, 2H), 1.39 (t, $J = 7.5$ Hz, 3H). ^{13}C NMR (50 MHz, CDCl_3) δ_{c} : 168.6, 151.6, 135.7, 128.6, 128.3, 127.3, 126.2, 125.5, 124.7, 122.1, 121.1, 120.2, 117.0, 116.6, 115.4, 36.4, 32.8, 24.0, 13.8. Chemical Formula: $\text{C}_{19}\text{H}_{17}\text{NO}_2$. ESI MS (m/z):= 292 (M+H). Analysis calculated for $\text{C}_{19}\text{H}_{17}\text{NO}_2$: C 78.33, H 5.88, N 4.81. Found; C 78.35, H 5.85, N 4.80.

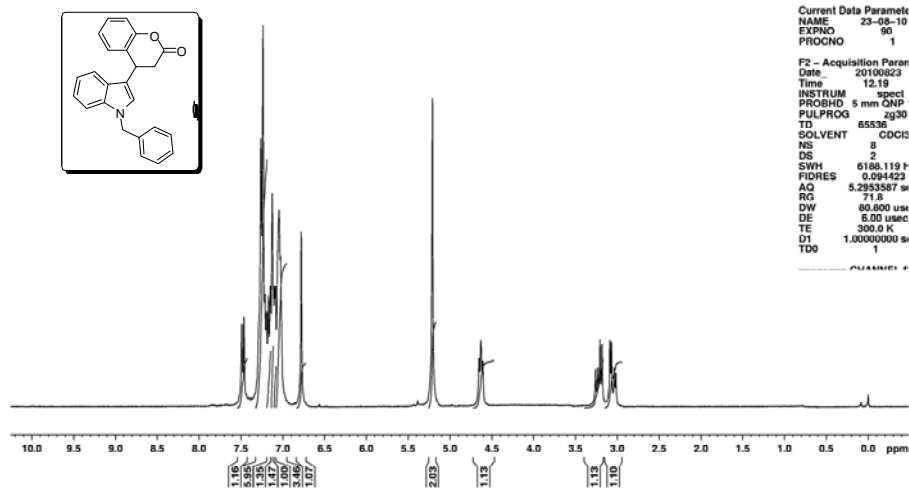
Spectral data for synthesized compounds (a-p).



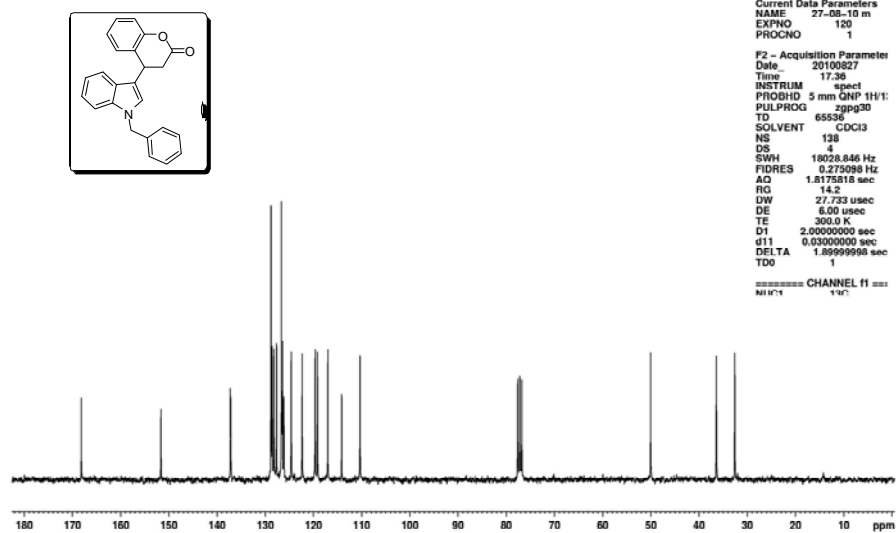
PK-249 A
C13CPD CDCl3 {D:\cdri



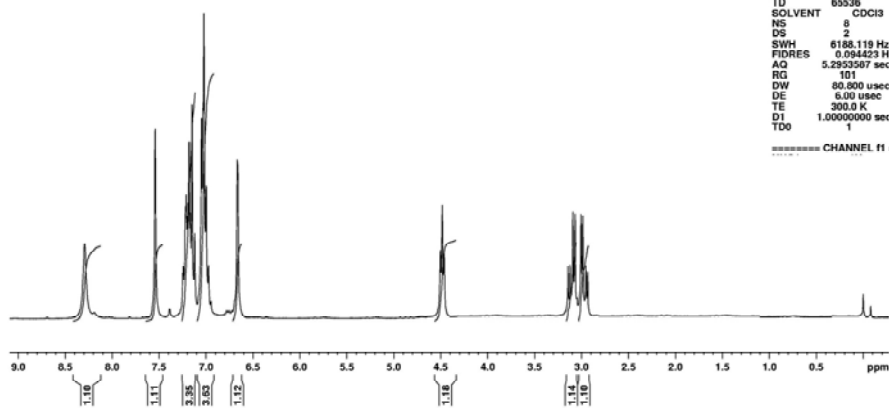
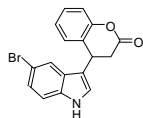
PK-275A
PROTON CDC13 {D:\cdr\}



PK-275A
C13CPD CDC1

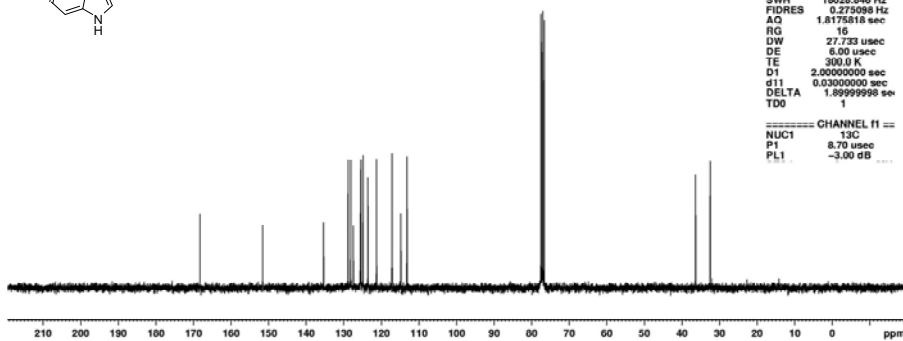
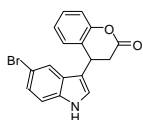


PK-266B
 PROTON CDC13 {D



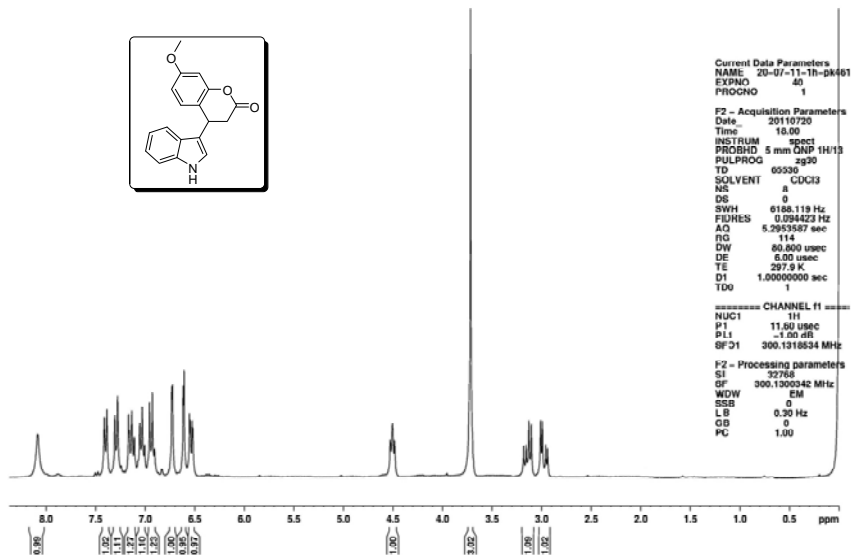
Current Data Parameter
 NAME 17-06_10 evn
 EXPNO 30
 PROCNO 1
 F2 - Acquisition Param
 Date_ 20100818
 Time 10.34
 INSTRUM spect
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 8
 DS 2
 SWH 6188.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.293507 sec
 RG 101
 DW 80.800 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec
 TDO 1
 ===== CHANNEL f1 =====

PK-266B
 C13CPD CDC13 {D:\cdri} user 12

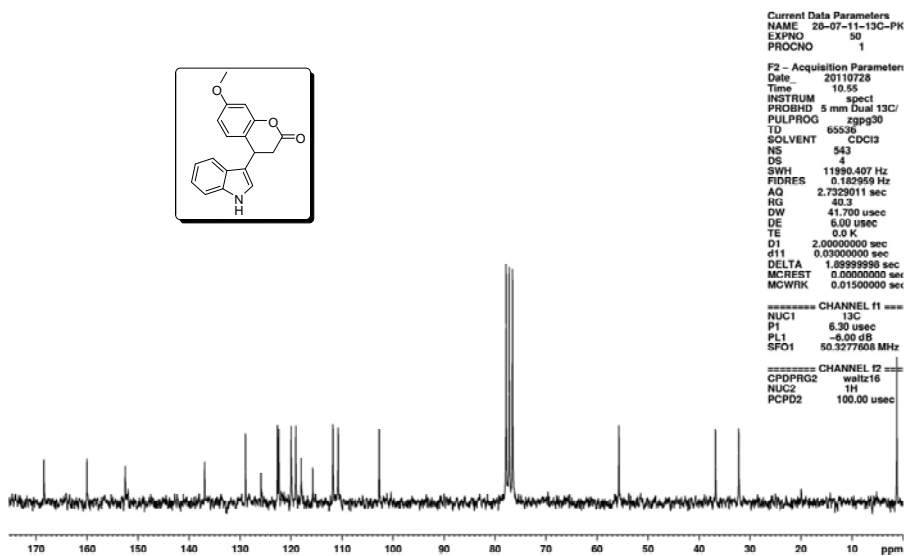


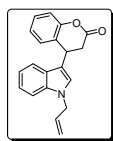
Current Data Parameters
 NAME 20-08-10
 EXPNO 120
 PROCNO 1
 F2 - Acquisition Paramete
 Date_ 20100820
 Time 14.20
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 4
 DS 4
 SWH 18028.846 Hz
 FIDRES 0.275088 Hz
 AQ 1.8175816 sec
 RG 16
 DW 27.733 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 8.70 usec
 PL1 -3.00 dB

PK-461A



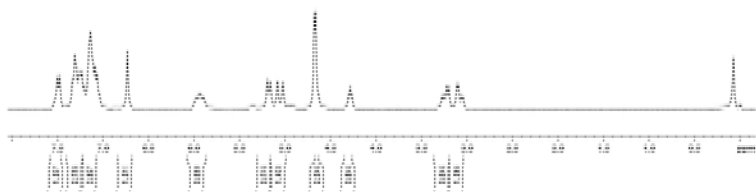
PK-461A



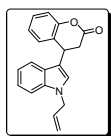


SI-005

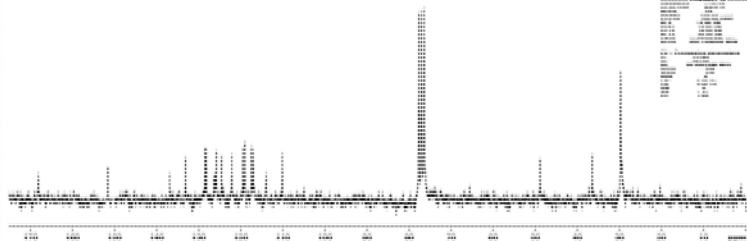
1H NMR
Chemical shift (ppm): 7.25 (d, 1H), 7.15 (d, 1H), 7.05 (d, 1H), 6.95 (d, 1H), 6.85 (d, 1H), 6.75 (d, 1H), 6.65 (d, 1H), 6.55 (d, 1H), 6.45 (d, 1H), 6.35 (d, 1H), 6.25 (d, 1H), 6.15 (d, 1H), 6.05 (d, 1H), 5.95 (d, 1H), 5.85 (d, 1H), 5.75 (d, 1H), 5.65 (d, 1H), 5.55 (d, 1H), 5.45 (d, 1H), 5.35 (d, 1H), 5.25 (d, 1H), 5.15 (d, 1H), 5.05 (d, 1H), 4.95 (d, 1H), 4.85 (d, 1H), 4.75 (d, 1H), 4.65 (d, 1H), 4.55 (d, 1H), 4.45 (d, 1H), 4.35 (d, 1H), 4.25 (d, 1H), 4.15 (d, 1H), 4.05 (d, 1H), 3.95 (d, 1H), 3.85 (d, 1H), 3.75 (d, 1H), 3.65 (d, 1H), 3.55 (d, 1H), 3.45 (d, 1H), 3.35 (d, 1H), 3.25 (d, 1H), 3.15 (d, 1H), 3.05 (d, 1H), 2.95 (d, 1H), 2.85 (d, 1H), 2.75 (d, 1H), 2.65 (d, 1H), 2.55 (d, 1H), 2.45 (d, 1H), 2.35 (d, 1H), 2.25 (d, 1H), 2.15 (d, 1H), 2.05 (d, 1H), 1.95 (d, 1H), 1.85 (d, 1H), 1.75 (d, 1H), 1.65 (d, 1H), 1.55 (d, 1H), 1.45 (d, 1H), 1.35 (d, 1H), 1.25 (d, 1H), 1.15 (d, 1H), 1.05 (d, 1H), 1.00 (s, 3H), 0.95 (d, 1H), 0.90 (d, 1H), 0.85 (d, 1H), 0.80 (d, 1H), 0.75 (d, 1H), 0.70 (d, 1H), 0.65 (d, 1H), 0.60 (d, 1H), 0.55 (d, 1H), 0.50 (d, 1H), 0.45 (d, 1H), 0.40 (d, 1H), 0.35 (d, 1H), 0.30 (d, 1H), 0.25 (d, 1H), 0.20 (d, 1H), 0.15 (d, 1H), 0.10 (d, 1H), 0.05 (d, 1H), 0.00 (s, 3H).



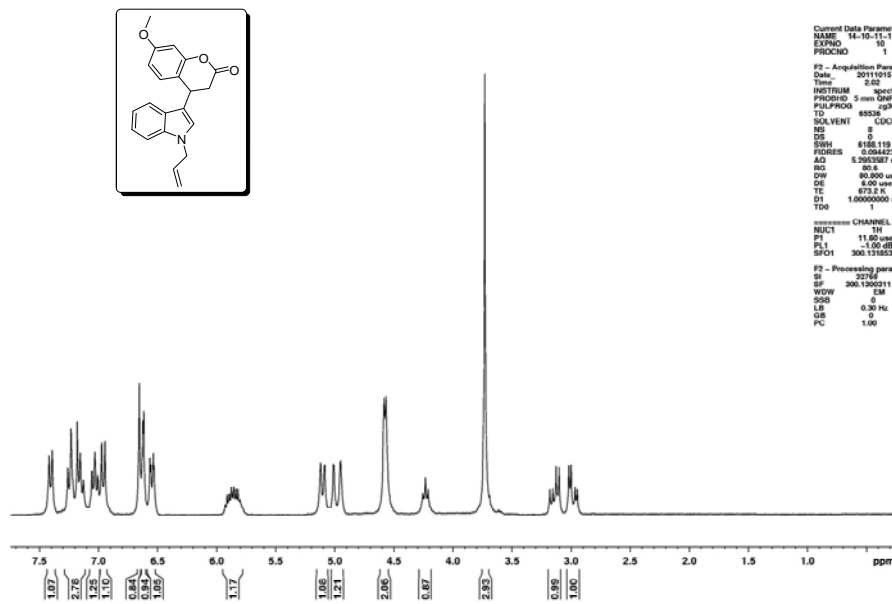
SI-006



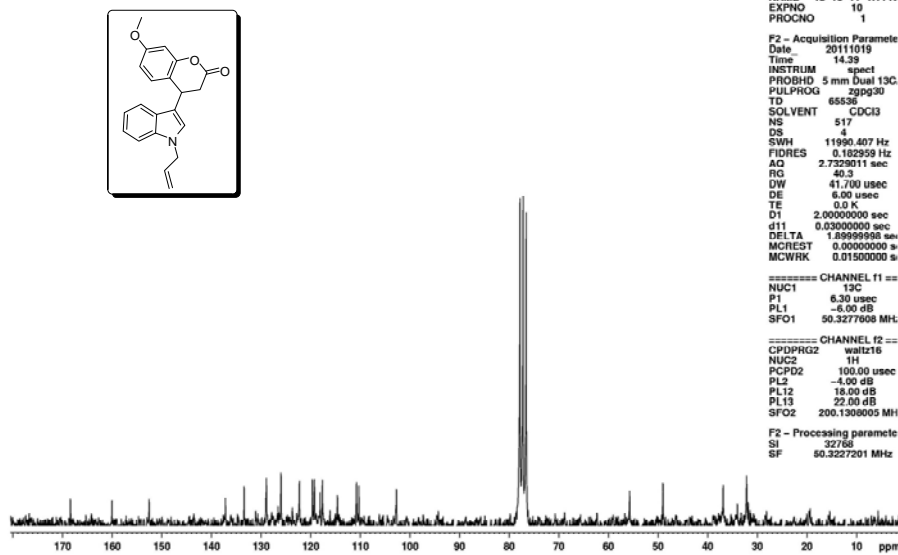
1H NMR
Chemical shift (ppm): 7.25 (d, 1H), 7.15 (d, 1H), 7.05 (d, 1H), 6.95 (d, 1H), 6.85 (d, 1H), 6.75 (d, 1H), 6.65 (d, 1H), 6.55 (d, 1H), 6.45 (d, 1H), 6.35 (d, 1H), 6.25 (d, 1H), 6.15 (d, 1H), 6.05 (d, 1H), 5.95 (d, 1H), 5.85 (d, 1H), 5.75 (d, 1H), 5.65 (d, 1H), 5.55 (d, 1H), 5.45 (d, 1H), 5.35 (d, 1H), 5.25 (d, 1H), 5.15 (d, 1H), 5.05 (d, 1H), 4.95 (d, 1H), 4.85 (d, 1H), 4.75 (d, 1H), 4.65 (d, 1H), 4.55 (d, 1H), 4.45 (d, 1H), 4.35 (d, 1H), 4.25 (d, 1H), 4.15 (d, 1H), 4.05 (d, 1H), 3.95 (d, 1H), 3.85 (d, 1H), 3.75 (d, 1H), 3.65 (d, 1H), 3.55 (d, 1H), 3.45 (d, 1H), 3.35 (d, 1H), 3.25 (d, 1H), 3.15 (d, 1H), 3.05 (d, 1H), 2.95 (d, 1H), 2.85 (d, 1H), 2.75 (d, 1H), 2.65 (d, 1H), 2.55 (d, 1H), 2.45 (d, 1H), 2.35 (d, 1H), 2.25 (d, 1H), 2.15 (d, 1H), 2.05 (d, 1H), 1.95 (d, 1H), 1.85 (d, 1H), 1.75 (d, 1H), 1.65 (d, 1H), 1.55 (d, 1H), 1.45 (d, 1H), 1.35 (d, 1H), 1.25 (d, 1H), 1.15 (d, 1H), 1.00 (s, 3H), 0.95 (d, 1H), 0.90 (d, 1H), 0.85 (d, 1H), 0.80 (d, 1H), 0.75 (d, 1H), 0.70 (d, 1H), 0.65 (d, 1H), 0.60 (d, 1H), 0.55 (d, 1H), 0.50 (d, 1H), 0.45 (d, 1H), 0.40 (d, 1H), 0.35 (d, 1H), 0.30 (d, 1H), 0.25 (d, 1H), 0.20 (d, 1H), 0.15 (d, 1H), 0.10 (d, 1H), 0.05 (d, 1H), 0.00 (s, 3H).



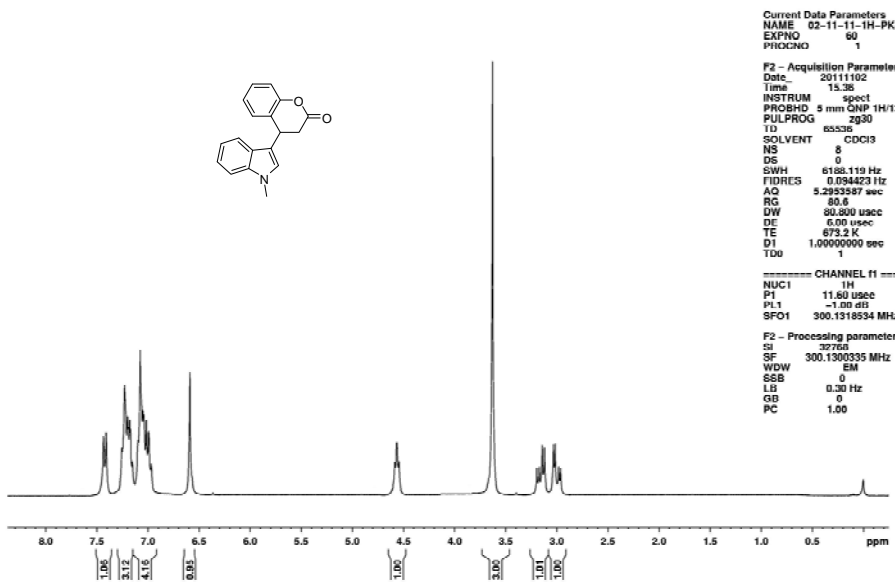
PK-509



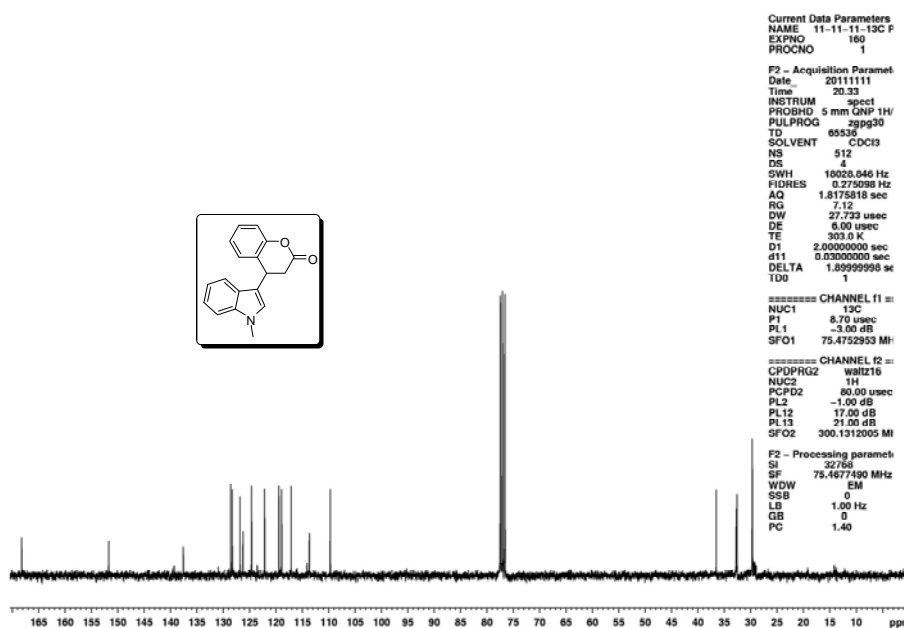
PK-509



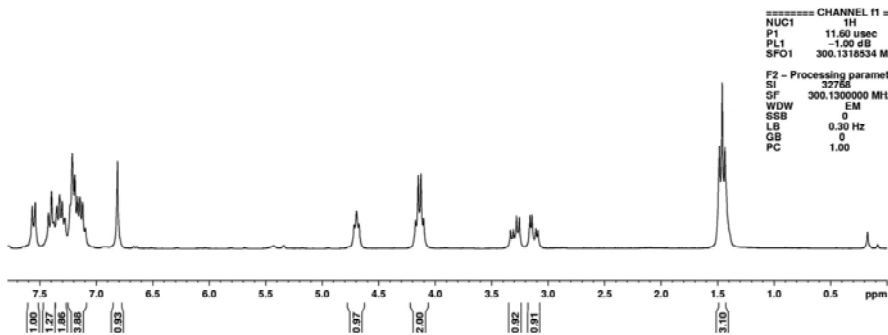
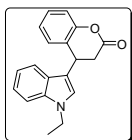
PK-513



PK-513



PK-494



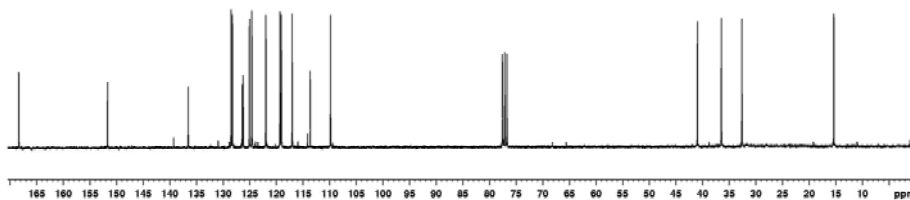
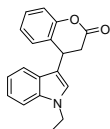
Current Data Parameters
 NAME 27-09-11-1H-PK
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameter
 Date 20110927
 Time 13.26
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 6166.119 Hz
 FIDRES 0.094423 Hz
 AQ 5.263367 sec
 RG 4
 DW 86.000 usec
 DE 6.00 usec
 TE 673.2 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 11.50 usec
 PL1 -1.00 dB
 SFO1 300.131534 MHz

F2 - Processing parameter
 SI 32768
 SF 300.130000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

PK-494



Current Data Parameters
 NAME 30-09-11-13C-PK-494
 EXPNO 209
 PROCNO 1

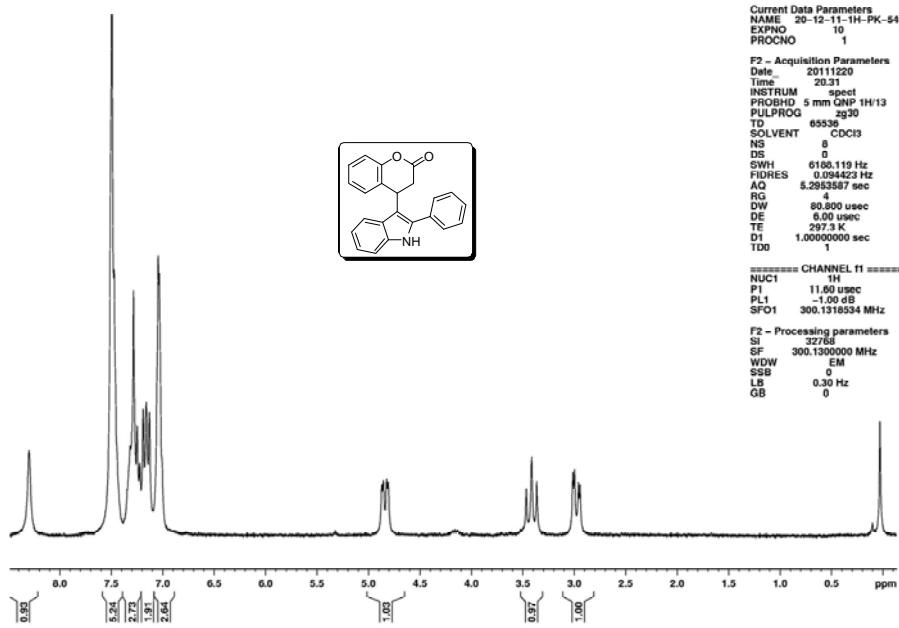
F2 - Acquisition Parameter
 Date 20110930
 Time 5.10
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 912
 DS 0
 SWH 10262.816 Hz
 FIDRES 0.272999 Hz
 AQ 1.6175216 sec
 RG 32768
 DW 27.723 usec
 DE 8.20 usec
 TE 673.2 K
 D1 3.0000000 sec
 d11 0.0000000 sec
 DELTA 1.3200000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.70 usec
 PL1 -2.00 dB
 SFO1 76.4702943 MHz

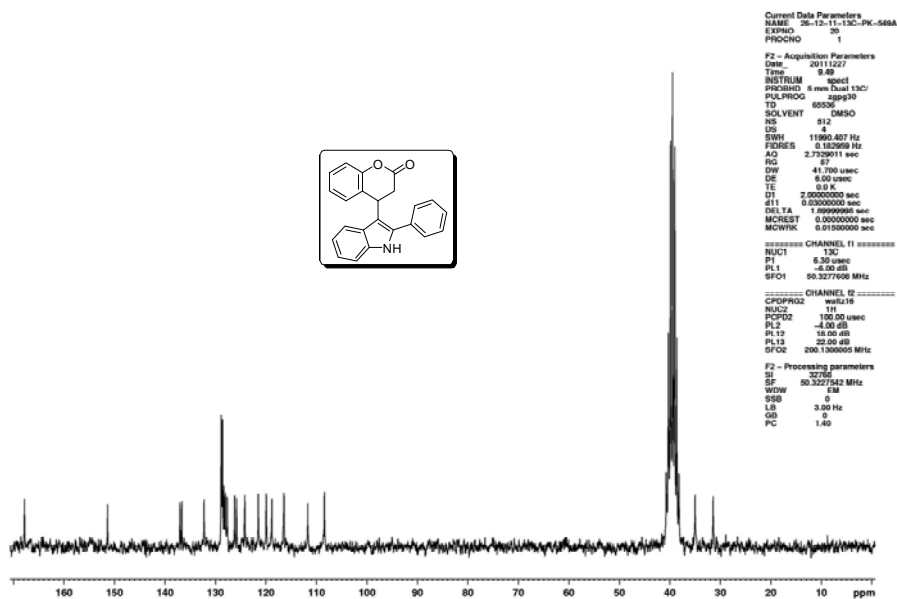
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 30.00 usec
 PL2 1.00 dB
 PL12 17.00 dB
 PL13 21.00 dB
 SFO2 300.1315000 MHz

F2 - Processing parameter
 SI 32768
 SF 76.4677029 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

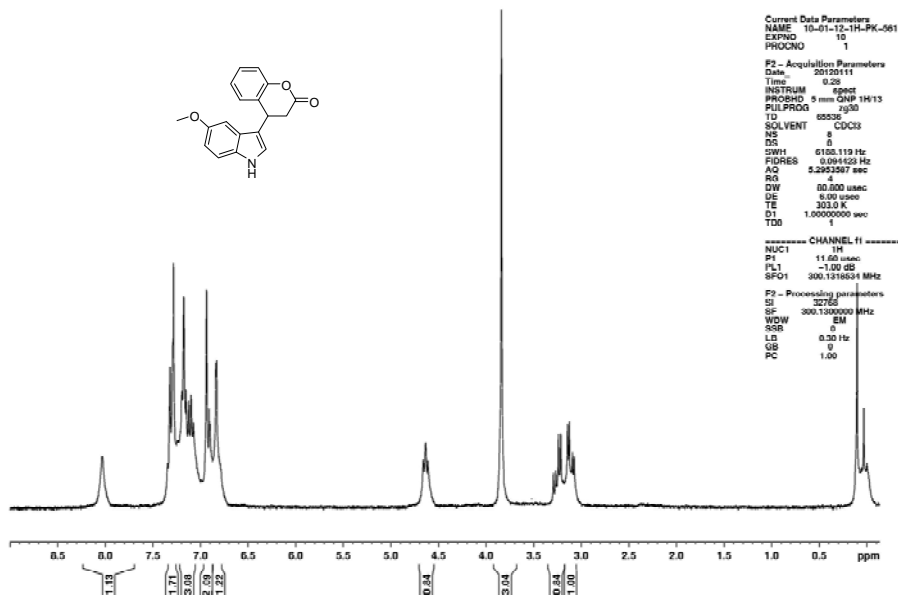
PK-549A



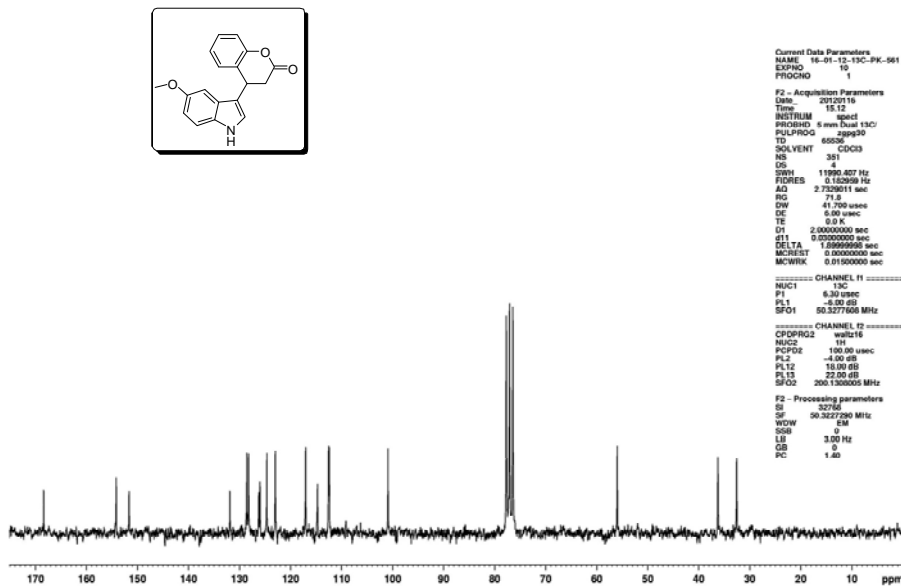
PK-549A



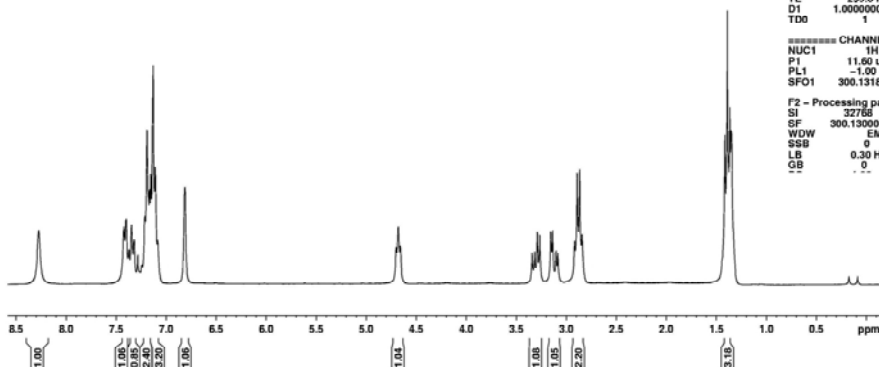
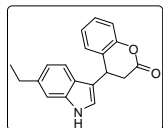
PK-561



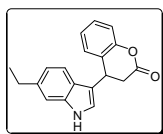
PK-561



PK-542A

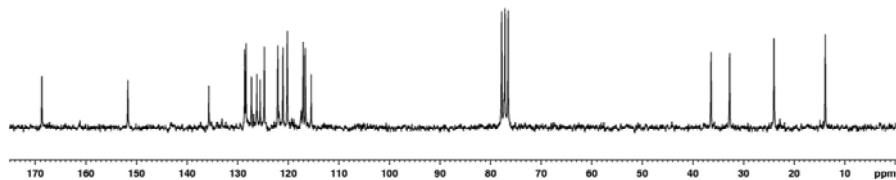


Current Data Parameters
 NAME 16-12-11-1H-
 EXPNO 30
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20111217
 Time 4.32
 INSTRUM spect
 PROBHD 5 mm QNP 1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 8
 DS 0
 SWH 0160.119 Hz
 FIDRES 0.0944221
 AQ 5.295387 se
 RG 8
 DW 80.800 usec
 DE 0.00 usec
 TE 295.2 K
 D1 1.0000000 se
 TDD 1
 ===== CHANNEL f1
 NUC1 1H
 P1 11.60 usec
 PL1 -1.00 dB
 SFO1 300.1318534
 F2 - Processing parameters
 SI 32768
 SF 300.1300000 M
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
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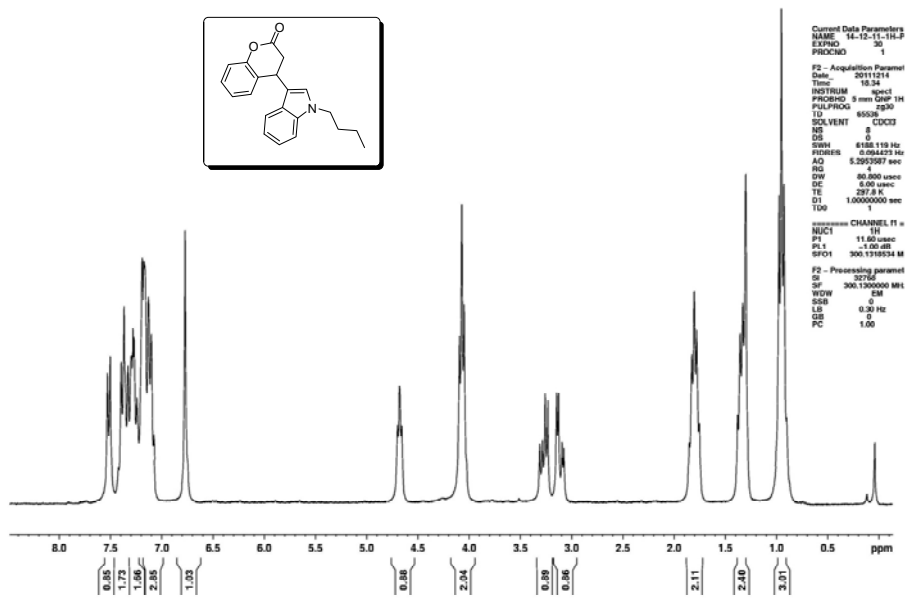


PK-542A

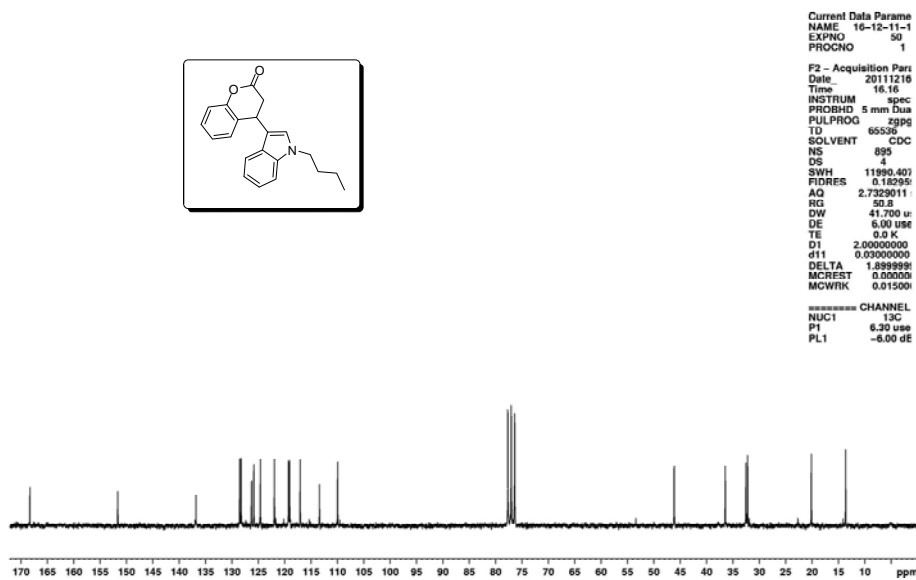
Current Data Parameters
 NAME 16-01-12-13C-PK-542
 EXPNO 30
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20110118
 Time 15.57
 INSTRUM spect
 PROBHD 5 mm Dual 13C/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 912
 DS 4
 SWH 11980.807 Hz
 FIDRES 0.32209 Hz
 AQ 2.722011 sec
 RG 26.6
 DW 41.100 usec
 DE 8.00 usec
 TE 303.2 K
 D1 2.0000000 sec
 d11 0.0000000 sec
 DELTA 1.8999999 sec
 MCHST 0.0000000 sec
 MCWRSK 0.0150000 sec
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 6.20 usec
 PL -6.00 dB
 SFO1 50.3277500 MHz
 ===== CHANNEL f2 =====
 CPROG2 spect16
 NUC2 1H
 PPR2 180.00 usec
 PL2 -4.00 dB
 PL12 18.00 dB
 PL13 22.00 dB
 SFO2 300.1318534 MHz
 F2 - Processing parameters
 SI 32768
 SF 50.3277298 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40



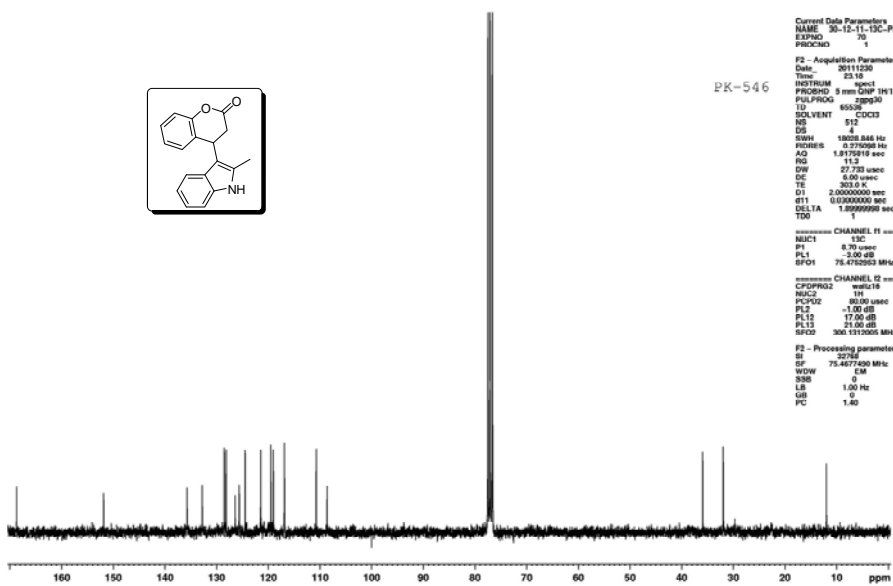
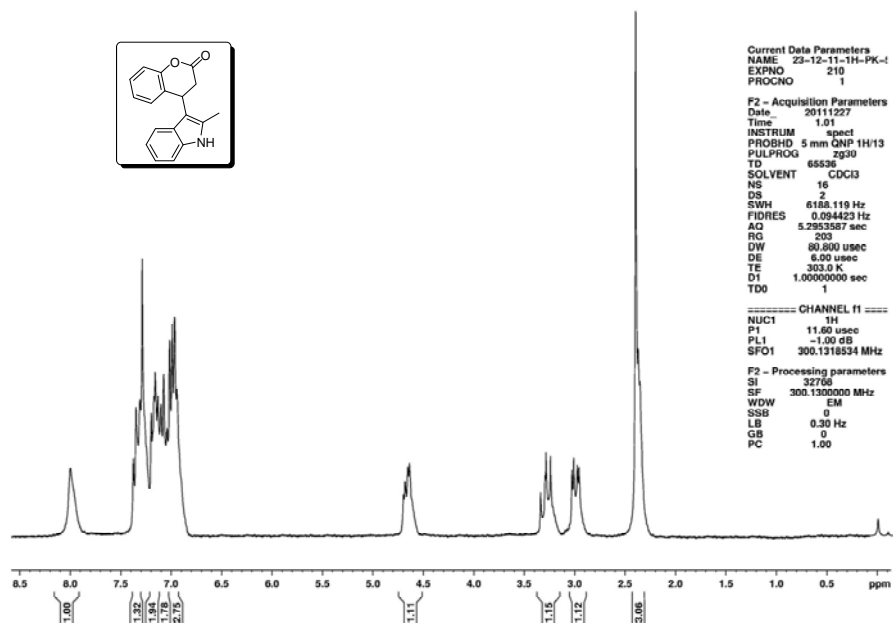
PK-539



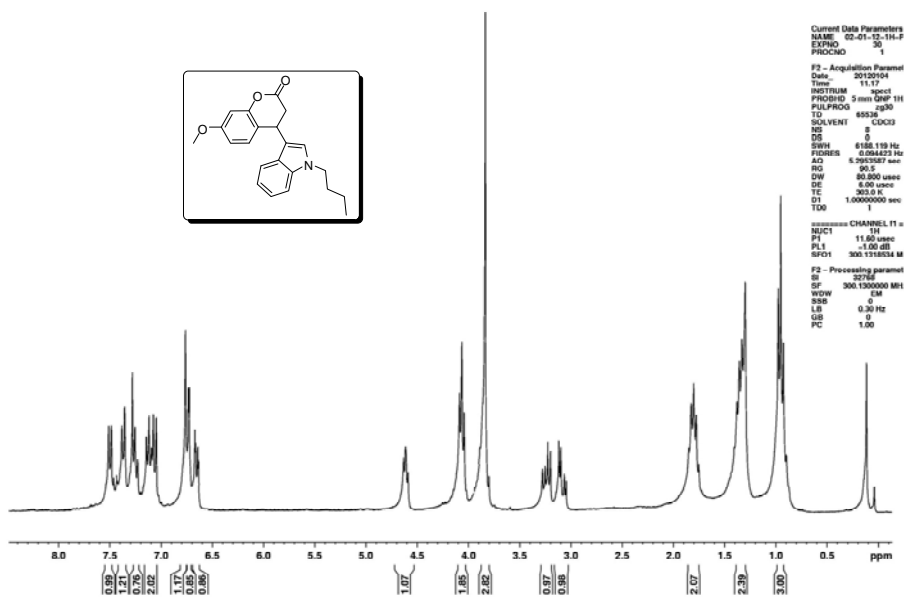
PK-539



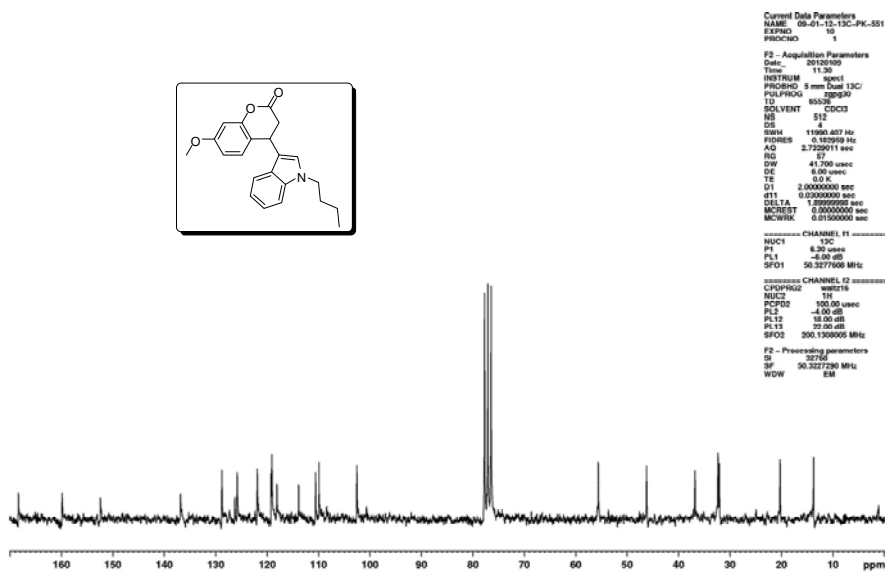
PK-546

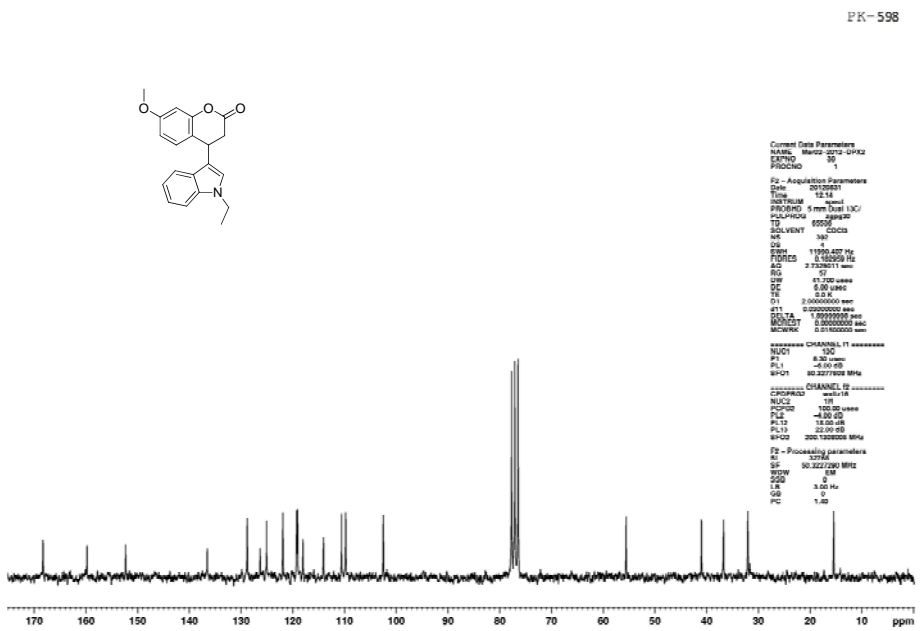
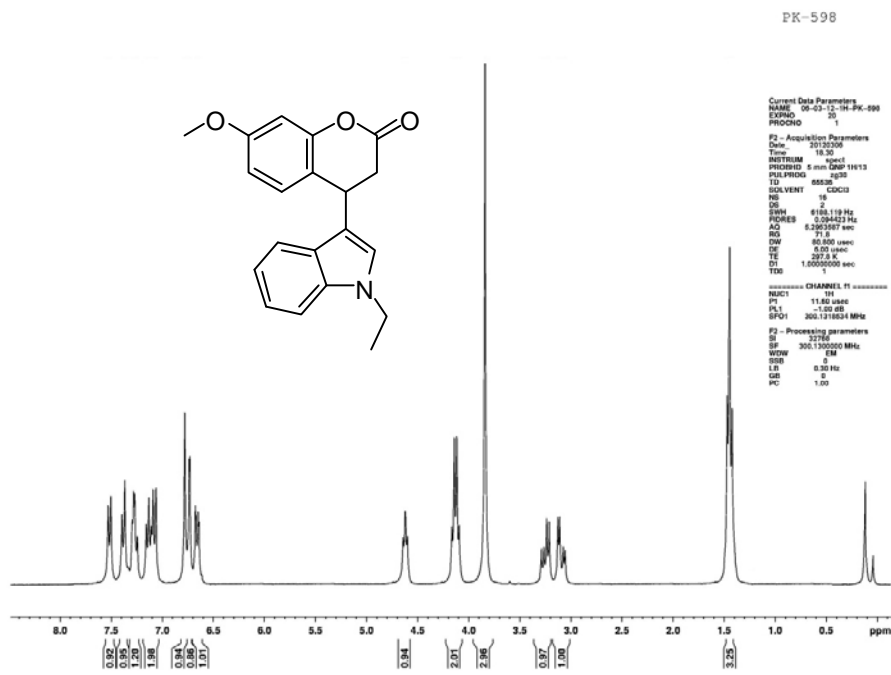


PK-551

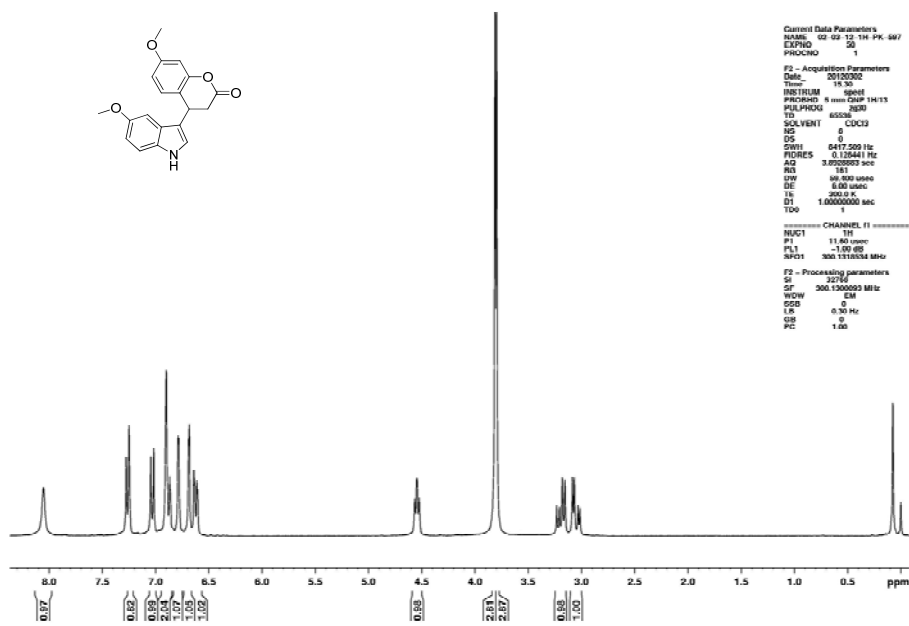


PK-551





PK-597



PK-597

