Supporting information

Bi(OTf)₃-Catalyzed Addition of Isocyanides to 2H-Chromene Acetals: An Efficient Pathway for Accessing 2-Carboxamide-2H-Chromenes

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General information

General experimental methods and instruments.

NMR Spectra were recorded on a Bruker DPX-400 or DPX-500 spectrometer at 400 MHz or 500 MHz for $^1$H NMR and 100 MHz or 125 MHz for $^{13}$C NMR in CDCl$_3$. Chemical shifts are reported in $\delta$ (ppm) referenced to an internal tetramethylsilane (TMS) standard or the residual deuterated solvent peaks and coupling constants ($J$) were expressed in Hz. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Flash column chromatograph was carried out using 200-300 mesh silica gel at medium pressure or ODS-A-HG C18 reversed silica gel. High resolution mass spectra (HRMS) were recorded on a LC-TOF spectrometer. ESI-HRMS data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI source. All chemicals were purchased from Acros, Alfa Aesar and TCI, and used as received.

General procedure for synthesis of 2-carboxamide-2H-chromenes in Table 2 and 3.

To a solution of Bi(OTf)$_3$ (0.025 mmol, 0.10 equiv) in indicated solvents (1,4-dioxane:H$_2$O = 10:1) (1.0 mL) were added chromene acetals 1 (0.25 mmol, 1.0 equiv) and isocyanides 2 (0.375 mmol, 1.5 equiv) in sequence. Then the result mixtures were stirred at 80 °C for 20 h. After the reactions were completed, the solvent was removed under reduced pressure. The crude products were purified by silica gel chromatography (6-50% EtOAc/PE with 0.1% Et$_3$N) to give the desired products 3 in 66-95% yields (Table 2 and 3).
General experimental procedure for synthesis of chromene acetals.

To a solution of 2-hydroxybenzaldehydes (244.2 mg, 2.0 mmol) in THF (5 mL) was added (formylmethylene)triphenylphosphorane (669.5 mg, 2.2 mmol) at room temperature. The resulting reaction mixture was refluxed at 100 °C for 20 h. The reaction mixture was cooled to room temperature, and extracted with H₂O (3 mL) and EtOAc (3 x 4 mL). The combined organic phase was washed with H₂O (4 mL), brine (4 mL), dried over Na₂SO₄ and concentrated under vacuo to give the desired crude product. The crude product was purified by column chromatography (20% EtOAc/PE) to afford 2-hydroxycinnamaldehyde in 87% yields.

The 2-hydroxycinnamaldehyde was added into a solution (Dioxane:MeOH = 1:1) with 1 mol% In(OTf)₃ and stirred at 80 °C for 12 h. After the reactions were completed, the solvent was removed under reduced pressure. The crude products were purified by silica gel chromatography (4-10% EtOAc/PE with 5% Et₃N) to afford the chromene acetal substrates in 91% yield.
Analytic data for the products in Table 2 and 3.

![Chemical Structure]

*N-cyclohexyl-2H-chromene-2-carboxamide (3aa)* was obtained in 95% yield. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.17-7.13 (m, 2H), 7.01-6.99 (m, 1H), 6.94-6.90 (m, 1H), 6.86 (d, $J$ = 8.4 Hz, 1H), 6.51 (d, $J$ = 7.2 Hz, 1H), 6.45 (dd, $J$ = 10.0, 2.8 Hz, 1H), 6.00 (dd, $J$ = 10.0, 2.8 Hz, 1H), 5.26 (t, $J$ = 2.8 Hz, 1H), 3.87-3.78 (m, 1H), 2.04-1.96 (m, 1H), 1.90-1.86 (m, 1H), 1.77-1.59 (m, 3H), 1.45-1.30 (m, 2H), 1.28-1.12 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.36, 151.96, 129.47, 127.02, 124.43, 122.46, 122.39, 121.58, 115.68, 75.24, 48.07, 33.02, 32.99, 25.51, 24.86, 24.83. HRMS (ESI) calcd for C$_{16}$H$_{19}$NO$_2$ (M+H)$^+$ 258.1489, found 258.1484.

![Chemical Structure]

*N-tert-butyl-2H-chromene-2-carboxamide (3ab)* was obtained in 91% yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.16-7.13 (m, 1H), 7.02-6.98 (m, 1H), 6.95-6.91 (m, 1H), 6.86 (d, $J$ = 8.0 Hz, 1H), 6.46 (dd, $J$ = 10.0, 2.5 Hz, 2H), 6.00 (dd, $J$ = 10.0, 3.0 Hz, 1H), 5.18 (d, $J$ = 3.0 Hz, 1H), 1.40 (s, 9H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 168.52, 152.02, 129.50, 127.06, 124.42, 122.71, 122.44, 121.67, 115.73, 75.50, 51.36, 28.85. HRMS (ESI) calcd for C$_{14}$H$_{17}$NO$_2$ (M+H)$^+$ 232.1332, found 232.1327.

![Chemical Structure]

*N-butyl-2H-chromene-2-carboxamide (3ac)* was obtained in 84% yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.17-7.13 (m, 1H), 7.02-6.99 (m, 1H), 6.94-6.91 (m, 1H), 6.86 (d, $J$ = 8.0 Hz, 1H), 6.62 (s, 1H), 6.46 (dd, $J$ = 9.5, 2.5 Hz, 1H), 6.00 (dd, $J$ = 10.0, 3.0 Hz, 1H), 5.29 (t, $J$ = 2.5 Hz, 1H), 3.37-3.29 (m, 2H), 1.56-1.50 (m, 2H), 1.39-1.32 (m, 2H), 0.93 (t, $J$ = 7.5 Hz, 3H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 169.43, 152.01, 129.58,

HRMS (ESI) calcd for C_{14}H_{17}NO_{2} (M+H)^+ 232.1332, found 232.1328.

![Image of chemical structure]

**Methyl (2H-chromene-2-carbonyl)glycinate (3ad)** was obtained in 83% yield. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.22-7.12 (m, 2H), 7.05-6.98 (m, 1H), 6.94-6.87 (m, 2H), 6.47 (dd, \(J = 10.0, 2.4\) Hz, 1H), 5.96 (dd, \(J = 10.0, 2.8\) Hz, 1H), 5.35 (t, \(J = 2.4\) Hz, 1H), 4.15 (dd, \(J = 18.0, 6.4\) Hz, 1H), 4.04 (dd, \(J = 18.4, 5.2\) Hz, 1H), 3.75 (s, 3H). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 169.87, 169.85, 151.77, 129.61, 127.01, 124.71, 122.39, 121.45, 121.22, 115.73, 75.08, 52.43, 40.89. HRMS (ESI) calcd for C_{13}H_{13}NO_{4} (M+H)^+ 248.0917, found 248.0912.

![Image of chemical structure]

**Ethyl (2H-chromene-2-carbonyl)glycinate (3ae)** was obtained in 92% yield. \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 7.18-7.14 (m, 2H), 7.01-6.99 (m, 1H), 6.94-6.91 (m, 1H), 6.90 (d, \(J = 8.0\) Hz, 1H), 6.48 (dd, \(J = 9.5, 2.5\) Hz, 1H), 5.98 (dd, \(J = 10.0, 3.0\) Hz, 1H), 5.36 (t, \(J = 3.0\) Hz, 1H), 4.23 (q, \(J = 7.5\) Hz, 2H), 4.15 (dd, \(J = 18.0, 5.5\) Hz, 1H), 4.04 (dd, \(J = 18.5, 5.5\) Hz, 1H), 1.29 (t, \(J = 7.0\) Hz, 3H). \(^{13}\)C NMR (125 MHz, CDCl\textsubscript{3}) \(\delta\) 169.91, 169.51, 151.91, 129.74, 127.14, 124.83, 122.52, 121.61, 121.37, 115.87, 75.22, 61.78, 41.21, 14.25. HRMS (ESI) calcd for C_{14}H_{13}NO_{4} (M+H)^+ 262.1074, found 262.1069.

![Image of chemical structure]

**N-benzyl-2H-chromene-2-carboxamide (3af)** was obtained in 80% yield. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.34-7.23 (m, 5H), 7.13-7.09 (m, 1H), 7.03-6.95 (m, 2H), 6.93-6.88 (m, 1H), 6.81 (d, \(J = 8.4\) Hz, 1H), 6.47 (dd, \(J = 9.6, 2.4\) Hz, 1H), 6.02 (dd, \(J = 2.9\) Hz, 1H).
= 9.6, 2.8 Hz, 1H), 5.35 (t, J = 2.8 Hz, 1H), 4.57-4.44 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.44, 151.86, 137.78, 129.56, 128.80, 127.73, 127.65, 127.06, 124.67, 122.44, 122.03, 115.73, 75.30, 43.17. HRMS (ESI) calcd for C$_{17}$H$_{15}$NO$_2$ (M+H)$^+$ 266.1176, found 266.1174.

Diethyl (2H-chromene-2-carboxamido)methylphosphonate (3ag) was obtained in 71% yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.18-7.15 (m, 1H), 7.02-6.99 (m, 1H), 6.98-6.91 (m, 2H), 6.88 (d, J = 8.0 Hz, 1H), 6.49 (dd, J = 9.5, 2.0 Hz, 1H), 5.97 (dd, J = 10.0, 3.0 Hz, 1H), 5.38-5.32 (m, 1H), 4.17-4.06 (m, 4H), 3.77 (dd, J = 12.0, 7.5 Hz, 2H), 1.34-1.28 (m, 6H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 169.61, 169.56, 151.75, 129.75, 127.11, 124.86, 122.57, 121.45, 121.28, 115.85, 75.13, 62.85, 62.80, 62.75, 35.26, 34.01, 16.49, 16.46. HRMS (ESI) calcd for C$_{15}$H$_{20}$NO$_5$P (M+H)$^+$ 326.1152, found 326.1142.

N-(tosylmethyl)-2H-chromene-2-carboxamide (3ah) was obtained in 85% yield. $^1$H NMR (400 MHz, CDCl$_3$) δ 7.70-7.65 (m, 2H), 7.45-7.36 (m, 1H), 7.28-7.25 (m, 2H), 7.23-7.18 (m, 1H), 7.04-6.95 (m, 2H), 6.91 (d, J = 8.0 Hz, 1H), 6.44 (dd, J = 9.6, 2.4 Hz, 1H), 5.65 (dd, J = 10.0, 3.2 Hz, 1H), 5.17 (t, J = 2.8 Hz, 1H), 4.80 (dd, J = 14.0, 7.6 Hz, 1H), 4.62 (dd, J = 14.4, 6.4 Hz, 1H), 2.42 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 169.33, 151.49, 145.56, 133.44, 130.06, 130.02, 129.06, 127.19, 124.98, 122.83, 121.10, 120.76, 116.00, 74.82, 59.80, 21.86. HRMS (ESI) calcd for C$_{18}$H$_{17}$NNaO$_4$S (M+Na)$^+$ 366.0770 found 366.0768.
N-(2,6-dimethylphenyl)-2H-chromene-2-carboxamide (3ai) was obtained in 73% yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.88 (s, 1H), 7.20-7.17 (m, 2H), 7.14-7.05 (m, 4H), 6.98-6.95 (m, 1H), 6.93 (d, J = 8.0 Hz, 1H), 6.55 (dd, J = 9.5, 2.5 Hz, 1H), 6.08 (dd, J = 10.0, 3.5 Hz, 1H), 5.50 (t, J = 3.0 Hz, 1H), 2.20 (s, 6H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 168.17, 151.92, 135.55, 132.89, 129.80, 128.40, 127.73, 127.30, 124.82, 122.72, 122.10, 121.50, 115.82, 75.72, 18.42. HRMS (ESI) calcd for C$_{18}$H$_{17}$NO$_2$ (M+H)$^+$ 280.1332, found 280.1325.

N-(4-methoxyphenyl)-2H-chromene-2-carboxamide (3aj) was obtained in 76% yield. $^1$H NMR (500 MHz, CDCl$_3$) δ 8.25 (s, 1H), 7.51-7.48 (m, 2H), 7.21-7.17 (m, 1H), 7.05-7.03 (m, 1H), 6.98-6.93 (m, 2H), 6.90-6.87 (m, 2H), 6.52 (dd, J = 10.0, 3.0 Hz, 1H), 6.06 (dd, J = 10.0, 3.0 Hz, 1H), 5.42 (t, J = 3.0 Hz, 1H), 3.80 (s, 3H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 167.32, 156.92, 151.81, 130.14, 129.75, 127.26, 124.89, 122.78, 122.05, 121.94, 121.58, 115.85, 114.37, 75.48, 55.63. HRMS (ESI) calcd for C$_{17}$H$_{15}$NO$_3$ (M+H)$^+$ 282.1125, found 282.1125.

N-cyclohexyl-6-methyl-2H-chromene-2-carboxamide (3ba) was obtained in 89% yield. $^1$H NMR (400 MHz, CDCl$_3$) δ 6.98-6.92 (m, 1H), 6.82 (d, J = 2.0 Hz, 1H), 6.77 (d, J = 8.0 Hz, 1H), 6.51 (d, J = 7.2 Hz, 1H), 6.42 (dd, J = 10.0, 2.8 Hz, 1H), 5.98 (dd, J = 9.6, 2.8 Hz, 1H), 5.22 (t, J = 2.4 Hz, 1H), 3.87-3.77 (m, 1H), 2.26 (s, 3H), 1.99-1.95 (m, 1H), 1.89-1.86 (m, 1H), 1.76-1.60 (m, 3H), 1.45-1.30 (m, 2H), 1.28-1.12 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.60, 149.86, 131.76, 129.89,
127.55, 124.63, 122.59, 121.44, 115.43, 75.28, 48.12, 33.10, 33.07, 25.59, 24.89, 20.65. HRMS (ESI) calcd for C\textsubscript{17}H\textsubscript{21}NO\textsubscript{2} (M+H)\textsuperscript{+} 272.1645, found 272.1638.

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\text{N-cyclohexyl-8-methyl-2H-chromene-2-carboxamide (3ca) was obtained in 75\% yield.}^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta 7.03-7.01 (m, 1H), 6.87-6.81 (m, 2H), 6.52 (d, J = 7.2 Hz, 1H), 6.44 (dd, J = 10.0, 2.8 Hz, 1H), 5.98 (dd, J = 10.0, 2.8 Hz, 1H), 5.28 (t, J = 2.8 Hz, 1H), 3.88-3.79 (m, 1H), 2.23 (s, 3H), 2.01-1.94 (m, 1H), 1.92-1.84 (m, 1H), 1.77-1.56 (m, 3H), 1.47-1.32 (m, 2H), 1.28-1.12 (m, 4H).}^{13}\text{C NMR (100 MHz, CDCl}_3\text{) }\delta 168.68, 149.91, 131.22, 124.87, 124.78, 124.73, 122.01, 121.83, 121.08, 75.40, 47.92, 33.04, 32.99, 25.59, 24.76, 24.72, 15.60. HRMS (ESI) calcd for C\textsubscript{17}H\textsubscript{21}NO\textsubscript{2} (M+H)\textsuperscript{+} 272.1645, found 272.1639.

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\text{N-cyclohexyl-7-methoxy-2H-chromene-2-carboxamide (3da) was obtained in 86\% yield.}^1\text{H NMR (400 MHz, CDCl}_3\text{) }\delta 6.92 (d, J = 8.0 Hz, 1H), 6.51-6.45 (m, 3H), 6.41 (dd, J = 10.0, 2.4 Hz, 1H), 5.85 (dd, J = 10.0, 4.0 Hz, 1H), 5.23 (t, J = 2.8 Hz, 1H), 3.80 (s, 3H), 2.03-1.94 (m, 1H), 1.93-1.84 (m, 1H), 1.79-1.58 (m, 3H), 1.45-1.31 (m, 2H), 1.26-1.12 (m, 3H).}^{13}\text{C NMR (100 MHz, CDCl}_3\text{) }\delta 168.57, 160.92, 153.23, 127.74, 124.15, 119.46, 114.90, 107.80, 102.04, 75.42, 55.57, 48.14, 33.12, 33.08, 25.60, 24.93, 24.90. HRMS (ESI) calcd for C\textsubscript{17}H\textsubscript{21}NO\textsubscript{3} (M+H)\textsuperscript{+} 288.1594, found 288.1586.
**N-cyclohexyl-5-methoxy-2H-chromene-2-carboxamide (3ea)** was obtained in 73% yield. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.09 (t, $J = 8.4$ Hz, 1H), 6.80 (dd, $J = 10.0$, 2.8 Hz, 1H), 6.55-6.45 (m, 3H), 5.92 (dd, $J = 10.0$, 3.2 Hz, 1H), 5.19 (t, $J = 2.8$ Hz, 1H), 3.82 (s, 3H), 2.02-1.93 (m, 1H), 1.92-1.83 (m, 1H), 1.78-1.57 (m, 3H), 1.45-1.31 (m, 2H), 1.29-1.11 (m, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 168.52, 155.56, 152.75, 129.45, 120.48, 119.26, 111.14, 108.62, 104.61, 74.85, 55.79, 48.06, 33.05, 33.01, 25.55, 24.88, 24.85. HRMS (ESI) calcd for C$_{17}$H$_{21}$NO$_3$ (M+H)$^+$ 288.1594, found 288.1586.

![Structure of N-cyclohexyl-5-methoxy-2H-chromene-2-carboxamide (3ea)](image)

**7-bromo-N-cyclohexyl-2H-chromene-2-carboxamide (3fa)** was obtained in 86% yield. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.24 (dd, $J = 7.5$, 2.0 Hz, 1H), 7.13 (d, $J = 2.0$ Hz, 1H), 6.76 (d, $J = 7.5$ Hz, 1H), 6.44-6.38 (m, 2H), 6.05 (dd, $J = 10.0$, 3.0 Hz, 1H), 5.26 (t, $J = 2.5$ Hz, 1H), 3.86-3.78 (m, 1H), 2.02-1.94 (m, 1H), 1.93-1.84 (m, 1H), 1.80-1.57 (m, 3H), 1.45-1.31 (m, 2H), 1.29-1.12 (m, 3H). $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 167.89, 151.06, 132.03, 129.55, 123.86, 123.49, 117.50, 114.53, 75.38, 48.25, 33.07, 25.55, 24.89. HRMS (ESI) calcd for C$_{16}$H$_{18}$BrNO$_2$ (M+H)$^+$ 336.0594, found 336.0592.

![Structure of 7-bromo-N-cyclohexyl-2H-chromene-2-carboxamide (3fa)](image)

**N-cyclohexyl-6-(trifluoromethyl)-2H-chromene-2-carboxamide (3ga)** was obtained in 23% yield. $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.41 (d, $J = 8.4$ Hz, 1H), 6.95 (d, $J = 8.4$ Hz, 1H), 6.48 (d, $J = 10.0$ Hz, 1H), 6.42 (d, $J = 6.5$ Hz, 1H), 6.10 (d, $J =$
10.0 Hz, 1H), 5.33 (s, 1H), 3.88-3.78 (m, 1H), 1.99 (d, \( J = 11.4 \) Hz, 1H), 1.89 (d, \( J = 11.6 \) Hz, 1H), 1.79-1.59 (m, 3H), 1.46-1.32 (m, 2H), 1.28-1.13 (m, 3H). \(^{3}\)C NMR (125 MHz, CDCl\(_3\)) \( \delta \) 167.65, 154.51, 126.67 (q, \( J = 3.75 \) Hz), 124.82 (q, \( J = 32.5 \) Hz), 124.19 (q, \( J = 3.75 \) Hz), 124.11 (q, \( J = 270.0 \) Hz), 123.88, 123.50, 116.08, 75.70, 48.33, 33.11, 33.09, 25.55, 24.93, 24.90. HRMS (ESI) calcd for C\(_{17}\)H\(_{19}\)F\(_3\)NO\(_2\) (M+H)+ 326.1362, found 326.1355.

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\( N \)-cyclohexyl-6-fluoro-2\( H \)-chromene-2-carboxamide (3ha) was obtained in 66% yield. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 6.99-6.92 (m, 1H), 6.67-6.95 (m, 2H), 6.43 (dd, \( J = 10.0 \), 2.8 Hz, 2H), 5.95 (dd, \( J = 10.0 \), 2.8 Hz, 1H), 5.26 (t, \( J = 2.8 \) Hz, 1H), 3.87-3.78 (m, 1H), 2.02-1.94 (m, 1H), 1.93-1.84 (m, 1H), 1.80-1.57 (m, 3H), 1.45-1.34 (m, 2H), 1.28-1.13 (m, 3H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 167.97, 164.36, 161.89, 153.12 (d, \( J_{CF} = 12.0 \) Hz), 127.88 (d, \( J_{CF} = 9.0 \) Hz), 123.64, 121.21 (d, \( J_{CF} = 3.0 \) Hz), 117.90 (d, \( J_{CF} = 4.0 \) Hz), 109.15 (d, \( J_{CF} = 21.0 \) Hz), 103.86 (d, \( J_{CF} = 26.0 \) Hz), 75.34, 48.19, 33.04, 33.02, 25.52, 24.88, 24.86. HRMS (ESI) calcd for C\(_{16}\)H\(_{18}\)FNO\(_2\) (M+H)+ 276.1394, found 276.1396.
Copies of $^1$H, $^{13}$C spectra in Table 2 and 3.

3aa (400 Hz, CDCl$_3$)
3ab (500 Hz, CDCl$_3$)

3ab (500 Hz, CDCl$_3$)
3ad (400 Hz, CDCl₃)
3ae (500 Hz, CDCl₃)
3da (400 Hz, CDCl$_3$)
26