Toward C2-nitrogenated chromones by copper-catalyzed β-C(sp²)-H N-heteroarylation of enaminones

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Contents

General experimental information .............................................................. S1

Table 1 Full data of condition optimization .................................................. S2

Characterization data for all products ..................................................... S3-S11

References ......................................................................................... S11

1H and 13C NMR spectra of all products .............................................. S12-S36

General experimental information

All experiments were carried out under air atmosphere. All enaminones 1 were synthesized following literature method, and other chemicals and solvents used in the experiments were obtained from commercial sources and used directly without further treatment. The 1H NMR and 13C NMR were recorded in 400 MHz apparatus using CDCl3 or DMSO-d₆ as solvent, the frequency for 1H NMR and 13C NMR test are 400 MHz and 100MHz, respectively. The chemical shifts were reported in ppm using TMS as internal standard. HRMS results were tested under ESI model in a spectrometer equipped with TOF analyzer. The melting points for solid samples were recorded in an X-4A apparatus.
Table 1 Optimization on the reaction conditions for 2-imidazolyl chromone

![Diagram of the reaction](image)

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*aGeneral conditions: 1a (0.2 mmol), 2a (0.24 mmol), catalyst (20 mol%), oxidant (2 equiv), base (2 equiv), I₂ (20 mol%) in 2 mL solvent, heated at 120 °C for 12 h. ¹Yield of isolated product based on 1a. ²Without I₂. ³With 10 mol% I₂. ⁴With 40 mol% I₂. ⁵With 4 equiv PhI(OAc)₂. ⁶With 5 equiv PhI(OAc)₂. ⁷With 10 mol% CuI. ⁸Reaction at 110 °C. ⁹Reaction at 130 °C. ¹⁰With 0.3 mmol 2a. ¹¹With 0.4 mmol 2a. ¹²With 0.5 mmol 2a.

S2
Characterization data for products 3

2-(1H-Imidazol-1-yl)-4H-chromen-4-one (3a).\(^2\) White solid; mp: 191-192 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.22 (d, \(J = 10.8\) Hz, 2 H), 7.77–7.72 (m, 1 H), 7.55 (d, \(J = 8.4\) Hz, 1 H), 7.48 (d, \(J = 6.2\) Hz, 2 H), 7.27 (s, 1 H), 6.41 (s, 1 H); \(^1\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.7, 154.2, 153.4, 134.8, 134.3, 131.8, 126.2, 126.0, 123.4, 117.6, 115.9, 97.1.

2-(1H-Imidazol-1-yl)-6-methyl-4H-chromen-4-one (3b). White solid; mp: 125 -126 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.22 (s, 1 H), 7.99 (s, 1 H), 7.55 (d, \(J = 8.4\) Hz, 1 H), 7.45 (d, \(J = 10.6\) Hz, 2 H), 7.26 (s, 1 H), 6.38 (s, 1 H), 2.48 (s, 3 H); \(^1\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.9, 153.3, 152.5, 136.4, 135.4, 134.8, 131.7, 125.4, 123.0, 117.3, 115.9, 97.0, 20.92; HRMS (ESI) m/z calcd for C\(_{13}\)H\(_{10}\)N\(_2\)NaO\(_2\)\(^+\) (M+Na)\(^+\) 249.0634, found 249.0640.

2-(1H-Imidazol-1-yl)-7-methyl-4H-chromen-4-one (3c). White solid; mp: 218-219 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.21 (s, 1 H), 8.10 (d, \(J = 8.1\) Hz, 1 H), 7.46 (s, 1 H), 7.35 (s, 1 H), 7.31–7.26 (m, 2 H), 6.35 (s, 1 H), 2.53 (s, 3 H); \(^1\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.7, 154.4, 153.2, 145.9, 134.8, 131.8, 127.6, 125.8, 121.1, 117.4, 115.9, 97.1, 21.8; HRMS (ESI) m/z calcd for C\(_{13}\)H\(_{10}\)N\(_2\)NaO\(_2\)\(^+\) (M+Na)\(^+\) 249.0634, found 249.0638.
2-(1H-Imidazol-1-yl)-6,7-dimethyl-4H-chromen-4-one (3d). White solid; mp: 212-213 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.19 (s, 1 H), 7.94 (s, 1 H), 7.45 (s, 1 H), 7.32 (s, 1 H), 7.26 (s, 1 H), 6.33 (s, 1 H), 2.42 (s, 3 H), 2.38 (s, 3 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.9, 153.1, 152.8, 144.9, 135.6, 134.8, 131.7, 125.7, 121.2, 117.8, 115.9, 97.0, 20.5, 19.3; HRMS (ESI) m/z calcd for C\(_{14}\)H\(_{12}\)N\(_2\)O\(_2\)Na\(^{+}\)(M+Na)\(^+\) 263.0791 found 263.0795.

2-(1H-Imidazol-1-yl)-6-methoxy-4H-chromen-4-one (3e). White solid; mp: 184-185 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.21 (s, 1 H), 7.58 (d, \(J = 3.0\) Hz, 1 H), 7.47 (t, \(J = 4.4\) Hz, 2 H), 7.34–7.29 (m, 1 H), 7.26 (s, 1 H), 6.38 (s, 1 H), 3.91 (s, 3 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.6, 157.7, 153.2, 148.8, 134.8, 131.8, 124.1, 123.9, 118.9, 115.9, 105.6, 96.5, 56.0; HRMS (ESI) m/z calcd for C\(_{13}\)H\(_{10}\)N\(_2\)O\(_3\)Na\(^{+}\)(M+Na)\(^+\) 265.0584, found 265.0587.

2-(1H-Imidazol-1-yl)-7-methoxy-4H-chromen-4-one (3f). White solid; mp: 129-130 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.21 (s, 1 H), 8.10 (d, \(J = 8.9\) Hz, 1 H), 7.47 (s, 1 H), 7.26 (s, 1 H), 7.05–6.99 (m, 1 H), 6.94 (d, \(J = 2.1\) Hz, 1 H), 6.33 (s, 1 H), 3.94 (s, 3 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.2, 164.6, 155.9, 153.1, 134.8, 131.7, 127.3, 117.0, 115.9, 114.9, 100.5, 97.0, 56.0; HRMS (ESI) m/z calcd for C\(_{13}\)H\(_{10}\)N\(_2\)O\(_3\)Na\(^{+}\)(M+Na)\(^+\) 265.0584, found 265.0593.
2-(1H-Imidazol-1-yl)-4H-benzo[h]chromen-4-one (3g). Yellow solid; mp: 250-251 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.45–8.39 (m, 1 H), 8.33 (s, 1 H), 8.15 (d, J = 8.7 Hz, 1 H), 8.00–7.95 (m, 1 H), 7.85 (d, J = 8.7 Hz, 1 H), 7.75 (t, J = 3.8 Hz, 2 H), 7.57 (s, 1 H), 7.34 (s, 1 H), 6.52 (s, 1 H); ¹³C NMR (100 MHz, CDCl₃) δ 177.6, 153.0, 151.6, 136.2, 134.8, 132.0, 129.7, 128.4, 127.7, 126.3, 123.2, 121.6, 120.6, 119.9, 116.1, 98.5; HRMS (ESI) m/z calcd for C₁₆H₁₀N₂O₂⁺ (M+Na)⁺ 285.0634, found 285.0646.

2-(4-Bromo-1H-imidazol-1-yl)-4H-chromen-4-one (3h). Yellow solid; mp: 179-180 °C; ¹H NMR (400 MHz, DMSO): δ 8.63 (s, 1 H), 8.23 (s, 1 H), 8.04 (d, J = 7.8 Hz, 1 H), 7.86 (t, J = 7.7 Hz, 1 H), 7.76 (d, J = 8.4 Hz, 1 H), 7.54 (t, J = 7.5 Hz, 1 H), 6.86 (s, 1 H); ¹³C NMR (100 MHz, DMSO) δ 177.6, 154.3, 152.8, 136.5, 135.1, 126.7, 125.4, 123.3, 118.7, 117.8, 116.6, 97.1; HRMS (ESI) m/z calcd for C₁₂H₈BrN₂O₂⁺ (M+H)⁺ 290.9764, found 290.9775.

2-(4-Iodo-1H-imidazol-1-yl)-4H-chromen-4-one (3i). Yellow solid; mp: 193-194 °C; ¹H NMR (400 MHz, DMSO): δ 8.58 (d, J = 1.3 Hz, 1 H), 8.25 (d, J = 1.4 Hz, 1 H), 8.05–8.00 (m, 1 H), 7.88–7.82 (m, 1 H), 7.75 (d, J = 7.9 Hz, 1 H), 7.55–7.50 (m, 1 H), 6.85 (s, 1 H); ¹³C NMR (100 MHz, DMSO) δ 177.6, 154.3, 152.7, 138.0, 135.0, 126.6, 125.3, 123.3, 122.3, 118.7, 96.9, 87.0; HRMS (ESI) m/z calcd for C₁₂H₈IN₂O₂⁺ (M+H)⁺ 338.9625, found 338.9639.
2-(4-Methyl-1H-imidazol-1-yl)-4H-chromen-4-one (3j). Yellow solid; mp: 150-151 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.22 (d, $J = 7.9$ Hz, 1 H), 8.14 (s, 1 H), 7.76–7.71 (m, 1 H), 7.53 (d, $J = 8.4$ Hz, 1 H), 7.48 (t, $J = 7.6$ Hz, 1 H), 7.16 (s, 1 H), 6.32 (s, 1 H), 2.31 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 177.8, 154.2, 153.3, 141.4, 134.1, 126.1, 126.0, 123.4, 117.5, 111.9, 96.4, 13.7; HRMS (ESI) m/z calcd for C$_{13}$H$_{10}$N$_2$NaO$_2^+$ (M+Na)$^+$ 249.0634, found 249.0642.

2-(2-Methyl-1H-imidazol-1-yl)-4H-chromen-4-one (3k). Yellow solid; mp: 128-129 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.25 (d, $J = 7.4$ Hz, 1 H), 7.76 (t, $J = 6.7$ Hz, 1 H), 7.51 (t, $J = 7.7$ Hz, 2 H), 7.28 (s, 1 H), 7.08 (s, 1 H), 6.33 (s, 1 H), 2.67 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 178.0, 167.8, 154.7, 154.3, 134.4, 129.3, 126.2, 126.1, 123.3, 118.4, 117.6, 102.3, 15.6; HRMS (ESI) m/z calcd for C$_{13}$H$_{11}$N$_2$O$_2^+$ (M+H)$^+$ 227.0815, found 227.0825.

2-(1H-Benzo[d]imidazol-1-yl)-4H-chromen-4-one (3l). Yellow solid; mp: 224-225 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.49 (s, 1 H), 8.27 (d, $J = 7.7$ Hz, 1 H), 7.90 (s, 2 H), 7.77 (t, $J = 7.3$ Hz, 1 H), 7.61 (d, $J = 8.0$ Hz, 1 H), 7.53–7.44 (m, 3 H), 6.59 (s, 1 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 177.8, 154.5, 153.9, 144.4, 139.9, 134.3, 131.0, 126.3, 126.1, 125.5, 124.7, 123.5, 121.4, 117.6, 112.5, 98.9.
2-(1H-Pyrazol-1-yl)-4H-chromen-4-one (3m). White solid; mp: 131-132 °C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 8.24 (d, \( J = 7.9 \) Hz, 2 H), 7.84 (s, 1 H), 7.71 (t, \( J = 7.7 \) Hz, 1 H), 7.53 (d, \( J = 8.4 \) Hz, 1 H), 7.46 (t, \( J = 7.5 \) Hz, 1 H), 6.90 (s, 1 H), 6.58 (s, 1 H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 178.0, 155.3, 154.1, 144.3, 133.8, 127.7, 126.1, 125.9, 123.8, 117.4, 109.6, 96.4.

6-Methyl-2-(1H-pyrazol-1-yl)-4H-chromen-4-one (3n). White solid; mp: 153-154 °C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 8.20 (d, \( J = 2.5 \) Hz, 1 H), 7.99 (s, 1 H), 7.82 (s, 1 H), 7.49 (d, \( J = 8.5 \) Hz, 1 H), 7.40 (d, \( J = 8.5 \) Hz, 1 H), 6.85 (s, 1 H), 6.57 (s, 1 H), 2.45 (s, 3 H). \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 178.1, 155.2, 152.3, 144.2, 135.9, 134.9, 127.7, 125.5, 123.4, 117.2, 109.5, 96.2, 20.9; HRMS (ESI) m/z calcd for C\(_{13}\)H\(_{10}\)N\(_2\)NaO\(_2\)\(^{2+}\) (M+Na)\(^+\) 249.0634, found 249.0640.

7-Methyl-2-(1H-pyrazol-1-yl)-4H-chromen-4-one (3o). White solid; mp: 154-155 °C; \( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 8.19 (s, 1 H), 8.09 (d, \( J = 8.0 \) Hz, 1 H), 7.82 (s, 1 H), 7.31 (s, 1 H), 7.24 (d, \( J = 7.9 \) Hz, 1 H), 6.84 (s, 1 H), 6.56 (s, 1 H), 2.50 (s, 3 H); \( ^{13}C \) NMR (100 MHz, CDCl\(_3\)) \( \delta \) 177.9, 155.1, 154.2, 145.3, 144.2, 127.7, 127.3, 125.8, 121.5, 117.3, 109.5, 96.2, 21.8; HRMS (ESI) m/z calcd for C\(_{13}\)H\(_{10}\)N\(_2\)NaO\(_2\)\(^{2+}\) (M+Na)\(^+\) 249.0634, found 249.0647.
**7-Methoxy-2-(1H-pyrazol-1-yl)-4H-chromen-4-one (3p).** White solid; mp: 155-156 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.18 (d, J = 2.7 Hz, 1 H), 8.11 (d, J = 8.8 Hz, 1 H), 7.82 (s, 1 H), 7.00–6.97 (m, 1 H), 6.92 (d, J = 2.3 Hz, 1 H), 6.78 (s, 1 H), 6.59–6.53 (m, 1 H), 3.92 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 177.4, 164.2, 155.7, 155.1, 144.1, 127.6, 127.3, 117.4, 114.5, 109.4, 100.5, 96.1, 55.9; HRMS (ESI) m/z calcd for C₁₃H₁₀N₂NaO₃⁺ (M+Na)⁺ 265.0584, found 265.0591.

**2-(1H-Benzimidazol-1-yl)-6-methyl-4H-chromen-4-one (3q).** White solid; mp: 236-237 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.47 (s, 1 H), 8.04 (s, 1 H), 7.88 (d, J = 8.0 Hz, 2 H), 7.56 (d, J = 8.0 Hz, 1 H), 7.51–7.42 (m, 3 H), 6.55 (s, 1 H), 2.49 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 177.9, 153.8, 152.8, 144.5, 139.9, 136.4, 135.4, 125.5, 125.5, 124.7, 123.1, 121.4, 117.3, 112.5, 98.8, 21.0; HRMS (ESI) m/z calcd for C₁₇H₁₂N₂NaO₂⁺ (M+Na)⁺ 299.0791, found 299.0796.

**2-(1H-Benzimidazol-1-yl)-7-methoxy-4H-chromen-4-one (3r).** White solid; mp: 184-185 °C; ¹H NMR (400 MHz, CDCl₃): δ 8.45 (s, 1 H), 8.17 (d, J = 8.9 Hz, 1 H), 7.90 (d, J = 7.6 Hz, 1 H), 7.85 (d, J = 7.9 Hz, 1 H), 7.50–7.42 (m, 2 H), 7.06 (d, J = 11.0 Hz, 1 H), 6.99 (s, 1 H), 6.52 (s, 1 H), 3.97 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 201.9, 177.3, 164.6, 156.3, 153.5, 139.9, 127.5, 125.4, 124.6, 121.4, 117.2, 114.9, 112.3, 100.5, 99.1, 56.1; HRMS (ESI) m/z calcd for C₁₇H₁₂N₂NaO₃⁺ (M+Na)⁺ 315.0740, found 315.0751.
2-(1H-Benzimidazol-1-yl)-4H-benzo[h]chromen-4-one (3s). Brown solid; mp: 182-183 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.56 (d, \(J = 7.2\) Hz, 1 H), 8.53 (s, 1 H), 8.48 (d, \(J = 8.0\) Hz, 1 H), 8.19 (d, \(J = 8.0\) Hz, 1 H), 7.99-7.93 (m, 3 H), 7.86 (d, \(J = 8.0\) Hz, 1 H), 7.76-7.74 (m, 2 H), 7.55-7.45 (m, 2 H), 6.70 (s, 1 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.6, 153.2, 152.1, 144.5, 140.2, 136.3, 131.2, 129.6, 128.5, 128.0, 126.2, 125.6, 124.8, 123.3, 121.5, 120.8, 119.9, 111.8, 100.9; HRMS (ESI) m/z calcd for C\(_{20}\)H\(_{12}\)N\(_2\)O\(_2\)Na\(^+\) (M+Na\(^+\)) 335.0791, found 335.804.

![Image of 2-(1H-Benzimidazol-1-yl)-4H-benzo[h]chromen-4-one](image)

6-Methyl-2-(4-methyl-1H-imidazol-1-yl)-4H-chromen-4-one (3t). Yellow solid; mp: 193-194 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.12 (s, 1 H), 7.99 (s, 1 H), 7.53 (d, \(J = 8.5\) Hz, 1 H), 7.41 (d, \(J = 8.5\) Hz, 1 H), 7.15 (s, 1 H), 6.28 (s, 1 H), 2.47 (s, 3 H), 2.31 (s, 3 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.9, 153.2, 152.5, 141.3, 136.2, 134.1, 134.1, 125.4, 123.1, 117.3, 111.9, 96.3, 20.9, 13.7; HRMS (ESI) m/z calcd for C\(_{14}\)H\(_{12}\)N\(_2\)O\(_2\)Na\(^+\) (M+Na\(^+\)) 263.0791, found 263.0802.

![Image of 6-Methyl-2-(4-methyl-1H-imidazol-1-yl)-4H-chromen-4-one](image)

7-Methoxy-2-(4-methyl-1H-imidazol-1-yl)-4H-chromen-4-one (3u). Yellow solid; mp: 184-185 °C; \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) 8.11 (d, \(J = 8.0\) Hz, 2 H), 7.14 (s, 1 H), 7.01 (d, \(J = 8.8\) Hz, 1 H), 6.93 (s, 1 H), 6.25 (s, 1 H), 3.94 (s, 3 H), 2.31 (s, 3 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 177.2, 164.5, 155.9, 153.0, 141.2, 134.0, 127.2, 117.0, 114.8, 111.9, 100.4, 96.3, 56.0, 13.6; HRMS (ESI) m/z calcd for C\(_{14}\)H\(_{12}\)N\(_2\)O\(_3\)Na\(^+\) (M+Na\(^+\)) 279.0740, found 279.0752.

![Image of 7-Methoxy-2-(4-methyl-1H-imidazol-1-yl)-4H-chromen-4-one](image)
2-(4-Bromo-1H-imidazol-1-yl)-7-methoxy-4H-chromen-4-one (3v). Yellow solid; mp: 135-136 °C; \textsuperscript{1}H NMR (400 MHz, DMSO-\textit{d}_6): \(\delta\) 8.56 (s, 1 H), 8.17 (s, 1 H), 7.89 (d, \(J = 8.8\) Hz, 1 H), 7.26 (s, 1 H), 7.06 (d, \(J = 8.5\) Hz, 1 H), 6.74 (s, 1 H), 3.89 (s, 3 H); \textsuperscript{13}C NMR (100 MHz, DMSO-\textit{d}_6) \(\delta\) 176.8, 164.4, 156.0, 152.5, 136.3, 126.7, 117.7, 116.9, 116.5, 115.3, 101.5, 96.7, 56.6; HRMS (ESI) m/z calcd for C\textsubscript{13}H\textsubscript{10}BrN\textsubscript{2}NaO\textsubscript{3}\textsuperscript{+} (M+H)\textsuperscript{+} 320.9883, found 320.9869.

2-(4-Bromo-1H-imidazol-1-yl)-6-chloro-4H-chromen-4-one (3w). White solid; mp: 188-189 °C; \textsuperscript{1}H NMR (400 MHz, DMSO-\textit{d}_6): \(\delta\) 8.64 (d, \(J = 1.3\) Hz, 2 H), 8.24 (d, \(J = 1.3\) Hz, 2 H), 7.95 (d, \(J = 2.5\) Hz, 1 H), 7.93–7.89 (m, 1 H), 7.82 (d, \(J = 8.9\) Hz, 1 H), 6.91 (s, 1 H); \textsuperscript{13}C NMR (100 MHz, DMSO-\textit{d}_6) \(\delta\) 176.5, 153.0, 152.9, 136.6, 134.9, 131.1, 124.5, 124.4, 121.1, 118.0, 116.6, 97.0; HRMS (ESI) m/z calcd for C\textsubscript{12}H\textsubscript{6}BrClN\textsubscript{2}NaO\textsubscript{2}\textsuperscript{+} (M+Na)\textsuperscript{+} 346.9193, found 346.9212.

2-(2H-1,2,3-Triazol-2-yl)-4H-chromen-4-one (3x). White solid; mp: 131-132 °C; \textsuperscript{1}H NMR (400 MHz, CDCl\textsubscript{3}): \(\delta\) 8.26 (d, \(J = 9.2\) Hz, 1 H), 8.01 (s, 2 H), 7.76 (t, \(J = 8.4\) Hz, 1 H), 7.68 (d, \(J = 8.3\) Hz, 1 H), 7.50 (t, \(J = 7.5\) Hz, 1 H), 7.05 (s, 1 H); \textsuperscript{13}C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 178.0, 154.5, 154.3, 138.3, 134.3, 126.2, 126.0, 123.8, 118.1, 98.6; HRMS (ESI) m/z calcd for C\textsubscript{11}H\textsubscript{7}N\textsubscript{3}NaO\textsubscript{2}\textsuperscript{+} (M+Na)\textsuperscript{+} 236.0430, found 236.0437.
2-(Pyrrolidin-1-yl)-4H-chromen-4-one (3y). White solid; mp: 198-199 °C; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.18–8.13 (m, 1 H), 7.54–7.49 (m, 1 H), 7.34–7.26 (m, 2 H), 5.32 (s, 1 H), 3.50 (s, 4 H), 2.07–2.01 (m, 4 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 176.0, 161.3, 153.8, 131.8, 125.6, 124.5, 123.1, 116.2, 86.3, 46.7, 25.1; HRMS (ESI) m/z calcd for C$_{13}$H$_{13}$NNaO$_2$ $(\text{M+Na})^+$ 238.0838, found 238.0849.

References


$^1$H and $^{13}$C NMR spectra of all products

$^1$H and $^{13}$C NMR spectra of 3a

S12
$^1$H and $^{13}$C NMR spectra of 3b
$^1$H and $^{13}$C NMR spectra of 3e
$^{1}$$H$ and $^{13}$$C$ NMR spectra of 3d
$^1$H and $^{13}$C NMR spectra of 3e
$^1$H and $^{13}$C NMR spectra of 3f

![NMR spectra](image)

H and C NMR spectra of 3f
\(^1\)H and \(^{13}\)C NMR spectra of 3g
$^1$H and $^{13}$C NMR spectra of 3h
$^1$H and $^{13}$C NMR spectra of 3i
$^1$H and $^{13}$C NMR spectra of 3j
$^1$H and $^{13}$C NMR spectra of 3k
$^1$H and $^{13}$C NMR spectra of 3l

![NMR spectra](image)

S23
\(^1\)H and \(^{13}\)C NMR spectra of 3m
$^1$H and $^{13}$C NMR spectra of 3n
$^1$H and $^{13}$C NMR spectra of 3o
$^1$H and $^{13}$C NMR spectra of 3p
$^1$H and $^{13}$C NMR spectra of 3q
$^1$H and $^{13}$C NMR spectra of 3r

![NMR spectra image](image-url)
$^1$H and $^{13}$C NMR spectra of 3s
$^1$H and $^{13}$C NMR spectra of 3t
$^1$H and $^{13}$C NMR spectra of 3u
$^1$H and $^{13}$C NMR spectra of 3v
$^{1}H$ and $^{13}C$ NMR spectra of 3w
$^1$H and $^{13}$C NMR spectra of 3x
$^1$H and $^{13}$C NMR spectra of 3y