Radical decarboxylative annulations of alkynoates with 2-oxoacetic acids: Synthesis of 3-acylcoumarins via 5-exo cyclization and ester Migration

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Supporting information

2. Characterization data of compound 3.
3. $^1$H and $^{13}$C NMR spectra of compound 3.
**General Materials and Methods:** All reactions were performed in reaction tubes under nitrogen atmosphere. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 µm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr (house vacuum) at 25–35 °C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale.

**General procedure for reactions of alkynoates 1 with 2-oxoacetic acids 2:**

Alkynoates (1 equiv), 2-oxoacetic acid (1.2 equiv), Ag$_2$CO$_3$ (20 mol %), K$_2$S$_2$O$_8$ (2.0 equiv), and NaOAc (2.5 equiv) were added into the test tube, and then co-solvent MeCN/H$_2$O (2 mL) was added. The mixture was stirred at 80°C overnight. After completion of reaction as indicated by TLC (6-12 hrs), the mixture was filtrated, and the filtrate was extracted with EtOAc, and dried by anhydrous Na$_2$SO$_4$. Evaporation of the solvent followed by purification on silica gel provided the product 3.

3-benzoyl-7-methyl-4-phenyl-2H-chromen-2-one (3a): a yellow solid, mp: 180-182 °C; reaction time: 8 hrs; $^1$H NMR (400 MHz, CDCl$_3$) δ 2.49 (s, 3H), 7.07 (d, $J$ = 8.2 Hz, 1H), 7.18 (d, $J$ = 8.1 Hz, 1H), 7.25-7.38 (m, 8H), 7.50 (t, $J$ = 7.4 Hz, 1H), 7.80 (d, $J$ = 8.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 21.7, 116.9, 117.3, 124.8, 125.9, 127.7, 128.5, 128.7, 129.2, 129.4, 132.5, 133.7, 136.3, 144.3, 153.1, 153.8,
159.0 192.3; HRMS (ESI): m/z [M + K]⁺ calcd for C₂₃H₁₆KO⁺: 379.0731; found: 379.0726.

3-benzoyl-4-phenyl-2H-chromen-2-one (3b): a yellow solid, mp: 158-159 °C; reaction time: 8 hrs; ¹H NMR (400 MHz, CDCl₃) δ 7.25-7.39 (m, 9H), 7.49 (t, J = 8.4 Hz, 2H), 7.61-7.65 (m, 1H), 7.81 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 117.2, 119.4, 124.6, 125.8, 127.9, 128.5, 128.7, 129.2, 129.5, 132.3, 132.7, 133.8, 136.2, 152.9, 153.7, 158.7, 192.1; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₂H₁₄NaO⁺: 349.0835; found: 349.0849.

3-benzoyl-7-tert-butyl-4-phenyl-2H-chromen-2-one (3c): a yellow solid, mp: 165-167 °C; reaction time: 8 hrs; ¹H NMR (400 MHz, CDCl₃) δ 1.39 (s, 9H), 7.23-7.38 (m, 9H), 7.49-7.75 (m, 2H), 7.80 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 31.0, 113.9, 116.9, 122.2, 125.0, 127.5, 128.5, 128.6, 129.2, 129.4, 132.5, 133.7, 136.4, 152.9, 153.8, 157.4, 159.1, 192.3; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₅H₂₂NaO⁺: 405.1461; found: 405.1467.

3-benzoyl-7-benzyl-4-phenyl-2H-chromen-2-one (3d): a yellow solid, mp: 132-135 °C; reaction time: 10 hrs; ¹H NMR (400 MHz, CDCl₃) δ 4.11 (m, 2H), 7.10 (d, J = 8.2 Hz, 1H), 7.20-7.39 (m, 14H), 7.51 (t, J = 7.4 Hz, 1H), 7.80 (d, J = 8.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 42.0, 117.2, 117.5, 125.1, 125.5, 126.7, 127.9, 128.5, 128.6, 128.8, 128.9, 129.2, 129.4, 130.1, 132.4, 133.7, 136.3, 139.3, 147.3, 152.9, 153.9, 158.8, 192.2; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₉H₂₀NaO⁺: 439.1305; found: 439.1315.
3-benzoyl-7-methoxy-4-phenyl-2H-chromen-2-one (3e): a yellow solid, mp: 142-145 °C; reaction time: 10 hrs; ¹H NMR (400 MHz, CDCl₃) δ 3.90 (s, 3H), 6.81 (d, J = 8.9, 1H), 6.94 (d, J = 2.4 Hz, 1H), 7.18 (d, J = 8.9 Hz, 1H), 7.23-7.26 (m, 2H), 7.31-7.37 (m, 5H), 7.49 (t, J = 7.2 Hz, 1H), 7.79 (d, J = 7.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 55.9, 100.9, 112.9, 122.6, 128.5, 128.6, 129.0, 129.2 129.4, 132.7, 133.6, 136.5, 153.5, 155.6 159.2, 163.5, 192.5; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₃H₁₆NaO₄⁺: 379.0941; found: 379.0937.

3-benzoyl-7-fluoro-4-phenyl-2H-chromen-2-one (3f): a yellow solid, mp: 168-170 °C; reaction time: 6 hrs; ¹H NMR (400 MHz, CDCl₃) δ 6.97–7.04 (m, 1H), 7.20 (d, J = 8.8, 1H), 7.18-7.39 (m, 8H), 7.52 (t, J = 7.4 Hz, 1H), 7.80 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 104.6 (d, 2J₉F = 22.4 Hz), 112.8 (d, 2J₉F = 22.4 Hz), 116.2, 128.6, 128.7, 129.2, 129.6, 129.8, 129.9, 132.1, 133.9, 136.1, 152.6, 154.8 (d, 3J₉F = 12.9 Hz), 158.4, 164.9 (d, 1J₉F = 254.9 Hz), 191.8; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₂H₁₃FNaO₃⁺: 367.0741; found: 367.0740.

3-benzoyl-7-chloro-4-phenyl-2H-chromen-2-one (3g): a yellow solid, mp: 186-187 °C; reaction time: 8 hrs; ¹H NMR (400 MHz, CDCl₃) δ 7.24-7.28 (m, 4H), 7.33-7.39 (m, 5H), 7.49-7.54 (m, 2H) 7.79 (d, J = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 117.4, 118.1, 125.2, 125.8, 128.6, 128.7, 128.9, 129.2, 129.7, 131.9, 133.9, 136.0, 138.7, 152.3, 153.9, 158.1, 191.7; HRMS (ESI): m/z [M + Na]⁺ calcd for C₂₂H₁₃ClNaO₃⁺: 383.0445; found: 383.0437.
3-benzoyl-7-bromo-4-phenyl-2H-chromen-2-one (3h): a yellow solid, mp: 196-197 °C; reaction time: 8 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.16 (d, \(J = 8.6\) Hz, 1H), 7.24-7.28 (m, 2H), 7.33-7.40 (m, 6H), 7.51 (t, \(J = 7.4\) Hz, 1H), 7.64 (d, \(J = 1.8\) Hz, 1H), 7.79 (d, \(J = 8.4\) Hz, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 118.4, 120.3, 126.0, 126.8, 128.1, 128.6, 128.7, 128.9, 129.2, 129.7, 131.8, 133.9, 136.0, 152.4, 153.8, 158.1, 191.7; HRMS (ESI): m/z [M + Na]\(^+\) calcd for C\(_{22}\)H\(_{13}\)BrNaO\(_3\): 426.9940; found: 426.9946.

3-benzoyl-7-iodo-4-phenyl-2H-chromen-2-one (3i): a yellow solid, mp: 188-189 °C; reaction time: 8 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 6.97 (d, \(J = 8.4\) Hz, 1H), 7.22-7.26 (m, 2H), 7.30-7.38 (m, 5H), 7.50 (t, \(J = 7.5\) Hz, 1H), 7.57 (dd, \(J = 8.4, 1.6\) Hz, 1H), 7.77 (d, \(J = 8.3\) Hz, 2H), 7.85 (d, \(J = 1.5\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 98.6, 119.0, 126.2, 126.8, 128.6, 128.7, 128.8, 129.2, 129.7, 131.8, 133.9, 136.0, 152.4, 153.4, 157.9, 191.7; HRMS (ESI): m/z [M + Na]\(^+\) calcd for C\(_{22}\)H\(_{13}\)INaO\(_3\): 474.9802; found: 474.9800.

7-acetyl-3-benzoyl-4-phenyl-2H-chromen-2-one (3j): a yellow solid, mp: 228-230 °C; reaction time: 7 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 2.68 (s, 3H), 7.26-7.28 (m, 2H), 7.34-7.41 (m, 6H), 7.52 (t, \(J = 7.4\) Hz, 1H), 7.78-7.82 (m, 3H), 7.98 (d, \(J = 1.5\) Hz, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 26.8, 115.3, 116.9, 122.8, 123.7, 127.8, 128.3, 128.6, 128.7, 129.2, 129.8, 130.7, 131.7, 134.0, 135.8, 139.8, 151.8, 153.5, 158.3, 191.6, 196.4; HRMS (ESI): m/z [M + Na]\(^+\) calcd for C\(_{24}\)H\(_{16}\)NaO\(_4\): 391.0941; found: 391.0932.
ethyl 3-benzoyl-2-oxo-4-phenyl-2H-chromene-7-carboxylate (3k): a yellow solid, mp: 230-231 °C; reaction time: 8 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 1.45 (t, \(J = 7.1\) Hz, 3H), 4.46 (q, \(J = 7.1\) Hz, 2H), 7.26-7.38 (m, 8H), 7.53 (t, \(J = 7.3\) Hz, 1H), 7.80 (d, \(J = 7.5\) Hz, 2H), 7.90 (dd, \(J = 8.2, 1.4\) Hz, 1H), 8.12 (s, 1H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 14.2, 61.8, 118.2, 122.7, 125.1, 127.7, 128.6, 128.7, 129.2, 129.7 131.8, 134.0, 134.0, 135.9, 151.9 153.3, 158.3, 164.9, 191.6; HRMS (ESI): m/z \([\text{M} + \text{Na}]^+\) calcd for C\(_{25}\)H\(_{18}\)NaO\(_5^+\): 421.1046; found: 421.1030.

3-benzoyl-4-p-tolyl-2H-chromen-2-one (3l): a yellow solid, mp: 167-170 °C; reaction time: 8 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 2.32 (s, 3H), 7.14-7.19 (m, 4H), 7.27 (d, \(J = 8.1\) Hz, 1H), 7.34-7.40 (m, 3H), 7.46-7.54 (m, 2H), 7.60-7.64 (m, 1H), 7.82 (d, \(J = 8.4\) Hz, 2H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 21.3, 117.1, 119.5, 124.6, 125.8, 128.0, 128.4, 128.6, 129.3, 130.1, 132.6, 133.6, 133.8, 136.2, 129.6, 153.2, 153.7, 158.8, 192.3; HRMS (ESI): m/z \([\text{M} + \text{Na}]^+\) calcd for C\(_{23}\)H\(_{16}\)NaO\(_3^+\): 363.0992; found: 363.0997.

3-benzoyl-4-(4-fluorophenyl)-2H-chromen-2-one (3m): a yellow solid, mp: 168-171 °C; reaction time: 8 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.05 (t, \(J = 8.6\) Hz, 2H), 7.24 – 7.33 (m, 4H), 7.40 (t, \(J = 7.7\) Hz, 2H),7.49 (d, \(J = 8.3\) Hz, 1H),7.54 (t, \(J = 7.4\) Hz, 1H),7.63-7.67 (m,1H), 7.77 – 7.83 (m, 2H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 115.8 (d, \(^2\)J\(_{CF}\) = 22.0 Hz), 117.3, 119.3, 127.7, 127.6, 128.2 (d, \(^2\)J\(_{CF}\) = 3.6 Hz), 128.5 (d, \(^2\)J\(_{CF}\) = 23.0 Hz), 128.7, 129.2, 130.1, 130.7 (d, \(^3\)J\(_{CF}\) = 8.4 Hz), 132.8, 133.7, 133.9, 136.1,
3-benzoyl-4-(naphthalen-1-yl)-2H-chromen-2-one (3n): a yellow solid, mp: 118-120 °C; reaction time: 6 hrs; $^1$H NMR (400 MHz, CDCl$_3$) δ 6.91 (dd, $J$ = 8.0, 1.4 Hz, 1H), 7.12 (t, $J$ = 7.2 Hz, 1H), 7.25 (t, $J$ = 7.6 Hz, 2H), 7.35-7.61 (m, 8H), 7.74 (d, $J$ = 7.2 Hz, 2H), 7.82-7.85 (m, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 117.1, 119.8, 124.7, 125.0, 125.7, 126.4, 126.8, 127.2, 127.5, 128.2, 128.3, 128.4, 129.0, 129.7, 130.0, 130.1, 130.7, 132.7, 133.1, 133.6, 135.9, 152.6, 153.3, 158.8, 191.7; HRMS (ESI): m/z [M + H$^+$] calcd for C$_{26}$H$_{17}$O$_3$: 377.1172; found: 377.1178.

3-benzoyl-4-(4-methoxyphenyl)-7-methyl-2H-chromen-2-one (3o): a yellow solid, mp: 142-144 °C; reaction time: 8 hrs; $^1$H NMR (400 MHz, CDCl$_3$) δ 2.50 (s, 3H), 3.78 (s, 3H), 6.85 (d, $J$ = 8.8 Hz, 2H), 7.08 (dd, $J$ = 8.2, 1.0 Hz, 1H), 7.20-7.28 (m, 4H), 7.37 (t, $J$ = 7.9 Hz, 2H), 7.51 (t, $J$ = 7.4 Hz, 1H), 7.82 (d, $J$ = 8.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 21.7, 55.2, 114.0, 117.2, 117.3, 124.6, 124.6, 127.6, 128.4, 128.5, 129.2, 130.1, 130.3, 133.7, 136.3, 144.1, 152.9, 153.8, 159.1, 160.3, 192.6; HRMS (ESI): m/z [M + H$^+$] calcd for C$_{24}$H$_{18}$NaO$_4$: 393.1097; found: 393.1096.

3-benzoyl-7-chloro-4-(4-methoxyphenyl)-2H-chromen-2-one (3p): a yellow solid, mp: 128-133 °C; reaction time: 8 hrs; $^1$H NMR (400 MHz, CDCl$_3$) δ 3.78 (s, 3H), 6.86 (dd, $J$ = 8.8, 2.1 Hz, 2H), 7.19-7.33 (m, 4H), 7.38 (td, $J$ = 8.0, 3.2 Hz 2H), 7.47-7.54 (m, 2H), 7.80 (d, $J$ = 7.8 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) δ 55.2, 144.2, 117.4,
3-benzoyl-6,7-difluoro-4-phenyl-2H-chromen-2-one (3q): a yellow solid, mp: 199-201 °C; reaction time: 6 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.09-7.14 (m, 1H), 7.22 - 7.24 (m, 8H), 7.53 (t, \(J = 7.4\) Hz, 1H), 7.74 - 7.81 (m, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 106.6 (d, \(^2\)J\(_{CF}\) = 21.3 Hz), 115.4 (d, \(^2\)J\(_{CF}\) = 20.5 Hz), 128.4, 128.6, 128.9, 129.2, 129.9, 131.7, 134.0, 135.9, 146.2 (d, \(^3\)J\(_{CF}\) = 13.4 Hz), 148.6 (d, \(^3\)J\(_{CF}\) = 13.2 Hz), 150.0, 151.7, 152.7 (d, \(^1\)J\(_{CF}\) = 257.5 Hz), 152.8 (d, \(^1\)J\(_{CF}\) = 257.4 Hz), 158.1, 191.4; HRMS (ESI): m/z [M + Na]\(^+\) calcd for C\(_{22}\)H\(_{15}\)ClNaO\(_4\)^+: 413.0551; found: 413.0558.

3-benzoyl-4-phenyl-2H-benzo[g]chromen-2-one (3r): a yellow solid, mp: 200-202 °C; reaction time: 12 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.27 (d, \(J = 8.8\) Hz, 1H), 7.31-7.40 (m, 7H), 7.52 (t, \(J = 7.4\) Hz, 1H), 7.64 (d, \(J = 8.8\) Hz, 1H), 7.69-7.74 (m, 2H), 7.85 (dd, \(J = 8.5, 1.3\) Hz, 2H), 7.89-7.92 (m, 1H), 8.65-8.68 (m, 1H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 106.5, 106.7, 114.6, 115.3, 115.5, 122.7, 122.7, 123.0, 124.4, 125.3, 127.8, 128.6, 128.7, 129.3, 129.3, 129.4, 132.7, 133.8, 134.0, 135.1, 136.3, 151.2, 154.0, 158.8, 192.3; HRMS (ESI): m/z [M + Na]\(^+\) calcd for C\(_{26}\)H\(_{16}\)NaO\(_3\)^+: 399.0992; found: 399.0996.

3-acetyl-7-methyl-4-phenyl-2H-chromen-2-one (3s): a yellow solid, mp: 70-72 °C; reaction time: 12 hrs; \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 2.26 (s, 3H), 2.47 (s, 3H), 7.03 (d, \(J = 8.2\) Hz, 1H), 7.11 (d, \(J = 8.2\) Hz, 1H), 7.22 (s, 1H), 7.30-7.32 (m, 2H), 7.51 (t, \(J = 3.0\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 21.7, 31.2, 116.9, 117.1, 125.8, 126.6,
127.9, 128.4, 128.7, 129.5, 132.8, 144.3, 152.1, 153.5, 158.7, 199.3; HRMS (ESI): m/z [M + Na]$^+$ calcd for C$_{18}$H$_{14}$NaO$_3^+$: 301.0835; found: 301.0836.

3-benzoyl-6-methyl-4-phenyl-2H-chromen-2-one (3t): a yellow solid, mp: 183-185 °C; reaction time: 8 hrs; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.56 (s, 3H), 7.11-7.16 (m, 2H), 7.25-7.39 (m, 7H), 7.47-7.53 (m, 2H), 7.81 (d, $J = 8.5$ Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 15.7, 112.9, 119.2, 124.1, 125.7, 126.6, 128.4, 128.5, 128.7, 129.2, 129.3, 132.6, 133.7, 134.0, 136.3, 152.0, 153.3, 158.8, 192.3; HRMS (ESI): m/z [M + K]$^+$ calcd for C$_{23}$H$_{16}$KO$_3^+$: 379.0731; found: 379.0734.

3-benzoyl-8-methyl-4-phenyl-2H-chromen-2-one (3t),: a yellow solid, mp: 180-181 °C; reaction time: 8 hrs; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 2.34 (s, 3H), 7.05 (d, $J = 1.2$ Hz, 1H), 7.26-7.29 (m, 2H), 7.34-7.39 (m, 6H), 7.44 (dd, $J = 8.4$, 1.2 Hz, 1H), 7.50 (t, $J = 7.5$ Hz, 1H), 7.80 (dd, $J = 8.5$, 1.4 Hz, 2H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 20.9, 116.9, 119.1, 125.9, 127.5, 128.5, 129.2, 129.4, 132.4, 133.7, 134.4, 136.2, 151.9, 152.9, 158.9, 192.2; HRMS (ESI): m/z [M + K]$^+$ calcd for C$_{23}$H$_{16}$KO$_3^+$: 379.0731; found: 379.0740.
$^1$HNMR spectrum of compound 3a

$^{13}$CNMR spectrum of compound 3a
$^1$HNMR spectrum of compound 3b

$^{13}$CNMR spectrum of compound 3b
$^1$HNMR spectrum of compound 3c

$^{13}$CNMR spectrum of compound 3c
$^{1}$HNMR spectrum of compound 3d

$^{13}$CNMR spectrum of compound 3d
$^{1} \text{HNMR spectrum of compound 3e}$

$^{13} \text{CNMR spectrum of compound 3e}$
HNMR spectrum of compound 3f

13CNMR spectrum of compound 3f
1H NMR spectrum of compound 3g

13C NMR spectrum of compound 3g
$^{1}$HNMR spectrum of compound 3h

$^{13}$CNMR spectrum of compound 3h
HNMR spectrum of compound 3i

CNMR spectrum of compound 3i
$^1$HNMR spectrum of compound 3j

$^{13}$CNMR spectrum of compound 3j
HNMR spectrum of compound 3k

CNMR spectrum of compound 3k
$^1$HNMR spectrum of compound 31

$^{13}$CNMR spectrum of compound 31
$\text{HNMR spectrum of compound } 3\text{m}$

$\text{CNMR spectrum of compound } 3\text{m}$
$^1$HNMR spectrum of compound 3n

$^{13}$CNMR spectrum of compound 3n
$^{1}$HNMR spectrum of compound 3o

$^{13}$CNMR spectrum of compound 3o
$^{1}\text{HNMR}$ spectrum of compound 3p

$^{13}\text{CNMR}$ spectrum of compound 3p
$^1$H NMR spectrum of compound 3q

$^{13}$C NMR spectrum of compound 3q
$^1$H NMR spectrum of compound 3r

$^{13}$C NMR spectrum of compound 3r
$^1$HNMR spectrum of compound 3s

$^{13}$CNMR spectrum of compound 3s
$^{1}$H NMR spectrum of compound 3t

$^{13}$C NMR spectrum of compound 3t
$^1$H NMR spectrum of compound 3t

$^{13}$C NMR spectrum of compound 3t