

Reactions of carbamoylated amino enones with 3-substituted chromones for the preparation of 2-pyridones and chromeno[4,3-*b*]pyridine-2,5-diones

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General experimental details

¹H and ¹³C NMR spectra were recorded on 500 and 125 MHz instruments, respectively, in DMSO-*d*₆. Chemical shifts are reported relative to TMS, DMSO-*d*₆ (δ = 2.50 ppm, ¹H NMR), and DMSO-*d*₆ (δ = 39.52 ppm, ¹³C NMR) as internal standards. IR spectra were recorded on a FTIR spectrometer with ATR accessory. Melting points were determined on Stuart SMP40 apparatus. All solvents used were dried and distilled by standard procedures.

General method for the preparation of pyridones **3 and chromeno[4,3-*b*]pyridine-2,5-diones **5**.** A mixture of carbamoylated amino enone **1** (0.34 mmol) and chromone **2** or **4** (0.41 mmol) was stirred with DMAP (50 mg, 0.41 mmol) in dry MeCN (2 ml) for 24 h. The reaction mixture was left at 20 °C for 24 h, the product was filtered off and washed with cold toluene.

(*Z*)-5-(2-Hydroxybenzoyl)-1-phenyl-3-(3-phenylamino-1-oxobut-2-en-1-yl)pyridin-2(1*H*)-one (3a**).** Yield 43% (67 mg), yellow powder, mp 246–248 °C. IR (ATR): 3192, 1652, 1565, 1531, 1486, 718 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.13 (s, 3H, Me), 6.52 (s, 1H, =CH), 6.69 (td, *J* = 7.5 Hz, *J* = 0.7 Hz, 1H, Ar), 6.72 (d, *J* = 8.7 Hz, 1H, Ar), 7.12 (td, *J* = 8.0 Hz, *J* = 1.6 Hz, 1H, Ar), 7.16–7.31 (m, 7H, Ph, Ar), 7.32 (d, *J* = 8.0 Hz, 2H, H-2,6 Ph), 7.44 (dd, *J* = 8.2 Hz, *J* = 7.6 Hz, 2H, H-3,5 Ph), 8.63 (s, 1H, pyridone), 9.18 (s, 1H, pyridone), 10.13 (s, 1H, OH), 13.29 (s, 1H, NH); ¹³C NMR (DMSO-*d*₆, ppm) δ 20.0, 97.7, 115.3, 115.6, 117.1, 118.5, 124.1, 125.7, 127.0, 128.2, 128.4, 129.2, 129.3, 130.9, 131.4, 137.4, 137.9, 138.0, 154.3, 158.6, 160.9, 162.9, 182.5, 187.6. Anal. Calcd for C₂₈H₂₂N₂O₄·0.25H₂O: C, 73.91; H, 4.98; N, 6.16. Found: C, 73.68; H, 4.73; N, 6.56.

(Z)-5-(5-Chloro-2-hydroxybenzoyl)-1-phenyl-3-(3-phenylamino-1-oxobut-2-en-1-yl)pyridin-2(1H)-one (3b). Yield 36% (59 mg), yellow powder, mp 253–255 °C. IR (ATR): 3181, 1653, 1530, 1484, 1087, 693 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.14 (s, 3H, Me), 6.52 (s, 1H, =CH), 6.72 (d, *J* = 8.8 Hz, 1H, H-6 Ar), 7.13–7.19 (m, 2H), 7.23–7.38 (m, 8H), 7.44 (dd, *J* = 8.2 Hz, *J* = 7.7 Hz, 2H, H-3,5 Ph), 8.63 (s, 1H, pyridone), 9.23 (s, 1H, pyridone), 10.46 (s, 1H, OH), 13.30 (s, 1H, NH). Anal. Calcd for C₂₈H₂₁ClN₂O₄: C, 69.35; H, 4.36; N, 5.78. Found: C, 69.09; H, 4.58; N, 5.91.

(Z)-5-(2-Hydroxy-5-methylbenzoyl)-1-phenyl-3-(3-phenylamino-1-oxobut-2-en-1-yl)pyridin-2(1H)-one (3c). Yield 37% (58 mg), yellow powder, mp 238–240 °C. IR (ATR): 3193, 1724, 1535, 1188, 829 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.05 (s, 3H, Me), 2.13 (s, 3H, Me), 6.53 (s, 1H, =CH), 6.61 (d, *J* = 8.3 Hz, 1H, H-6 Ar), 6.92 (dd, *J* = 8.5 Hz, *J* = 1.5 Hz, 1H, H-5 Ar), 6.98 (d, *J* = 1.5 Hz, 1H, H-3 Ar), 7.14–7.18 (m, 1H, H-4 Ph), 7.23–7.35 (m, 7H, Ph), 7.44 (dd, *J* = 8.2 Hz, *J* = 7.6 Hz, 2H, H-3,5 Ph), 8.63 (s, 1H, pyridone), 9.19 (s, 1H, pyridone), 9.87 (s, 1H, OH), 13.29 (s, 1H, NH); ¹³C NMR (DMSO-*d*₆, ppm) δ 19.7, 20.0, 97.7, 115.2, 115.5, 116.7, 124.0, 125.7, 127.0, 128.1, 128.3, 128.4, 129.2, 129.3, 130.8, 131.9, 137.3, 137.9, 138.0, 152.1, 158.6, 160.8, 162.8, 182.5, 187.7. Anal. Calcd for C₂₉H₂₄N₂O₄: C, 74.98; H, 5.21; N, 6.03. Found: C, 74.99; H, 5.34; N, 6.00.

(Z)-5-(2-Hydroxybenzoyl)-1-(4-methoxyphenyl)-3-[3-(4-methoxyphenylamino)-1-oxobut-2-en-1-yl]pyridin-2(1H)-one (3d). Yield 34% (59 mg), orange powder, mp 225–226 °C. IR (ATR): 3190, 1652, 1608, 1568, 1500, 1244, 812 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.05 (s, 3H, Me), 3.69 (s, 3H, MeO), 3.78 (s, 3H, MeO), 6.49 (s, 1H, =CH), 6.69–6.77 (m, 2H, H-5,6 Ar), 6.77–6.86 (m, 2H, Ar'), 6.99 (d, *J* = 8.4 Hz, 2H, Ar'), 7.09 (d, *J* = 8.4 Hz, 1H, Ar), 7.15 (t, *J* = 7.4 Hz, 1H, Ar), 7.32 (d, *J* = 8.0 Hz, 2H, H-2,6 Ph), 7.18–7.28 (m, 4H, 2Ar'), 8.63 (s, 1H, pyridone), 9.18 (s, 1H, pyridone), 10.13 (s, 1H, OH), 13.29 (s, 1H, NH); ¹³C NMR (DMSO-*d*₆, ppm) δ 19.8, 55.2, 55.3, 97.0, 113.5, 114.4, 115.3, 115.5, 117.3, 118.5, 125.9, 127.3, 128.1, 130.2, 130.5, 130.8, 131.3, 137.0, 154.3, 157.3, 158.7, 158.8, 161.1, 163.5, 182.1, 187.6. Anal. Calcd for C₃₀H₂₆N₂O₆: C, 70.58; H, 5.13; N, 5.49. Found: C, 70.54; H, 5.00; N, 5.53.

(Z)-1-Phenyl-3-(3-phenylamino-1-oxobut-2-en-1-yl)-1H-chromeno[4,3-*b*]pyridine-2,5-dione (5a). Yield 65% (99 mg), yellow powder, mp 274–275 °C. IR (ATR): 3051, 1738, 1673, 1526, 1279, 756 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.14 (s, 3H, Me), 6.27 (dd, *J* = 8.6 Hz, *J* = 1.3 Hz, 1H, H-10), 6.55 (s, 1H, =CH), 6.89 (ddd, *J* = 8.6 Hz, *J* = 8.5 Hz, *J* = 1.3 Hz, 1H, H-9), 7.28 (tt, *J* = 7.4 Hz, *J* = 1.0 Hz, 1H, H-4 Ph), 7.32 (d, *J* = 7.6 Hz, 2H, H-2,6 Ph), 7.44 (dd, *J* = 8.2 Hz, *J* = 7.6 Hz, 2H, H-3,5 Ph), 7.47 (dd, *J* = 8.6 Hz, *J* = 1.3 Hz, 1H, H-7), 7.50–7.54 (m, 2H, Ph), 7.56 (ddd, *J* = 8.6 Hz, *J* = 8.5 Hz, *J* = 1.3 Hz, 1H, H-8), 7.64–7.68 (m, 3H, Ph), 8.87 (s, 1H, H-4), 13.35 (s, 1H, NH); ¹³C NMR (DMSO-*d*₆, ppm) δ 20.1, 97.7, 103.8, 113.1, 118.2, 123.7,

124.1, 125.8, 126.5, 128.7, 129.3, 129.7, 130.0, 133.3, 137.9, 139.2, 139.9, 148.0, 153.4, 159.0, 160.9, 163.4, 181.3. Anal. Calcd for C₂₈H₂₀N₂O₄: C, 74.99; H, 4.50; N, 6.25. Found: C, 74.92; H, 4.48; N, 6.05.

(Z)-1-(4-Methoxyphenyl)-3-[3-(4-methoxyphenylamino)-1-oxobut-2-en-1-yl]-1H-chromeno[4,3-*b*]pyridine-2,5-dione (5b). Yield 73% (126 mg), orange powder, mp 224–225 °C. IR (ATR): 1717, 1699, 1498, 1277, 1052, 749 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.05 (s, 3H, Me), 3.78 (s, 3H, MeO), 3.88 (s, 3H, MeO), 6.44 (dd, *J* = 8.6 Hz, *J* = 1.1 Hz, 1H, H-10), 6.51 (s, 1H, =CH), 6.95–6.99 (m, 1H, H-9), 6.99 (d, *J* = 9.0 Hz, 2H, Ar), 7.18 (d, *J* = 9.0 Hz, 2H, Ar), 7.25 (d, *J* = 9.0 Hz, 2H, Ar), 7.40 (d, *J* = 9.0 Hz, 2H, Ar), 7.46 (dd, *J* = 8.3 Hz, *J* = 1.1 Hz, 1H, H-7), 7.56 (ddd, *J* = 8.3 Hz, *J* = 8.1 Hz, *J* = 1.3 Hz, 1H, H-8), 8.84 (s, 1H, H-4), 13.22 (s, 1H, NH). Anal. Calcd for C₃₀H₂₄N₂O₆: C, 70.86; H, 4.76; N, 5.51. Found: C, 70.46; H, 4.41; N, 5.26.

(Z)-1-(2-Methoxyphenyl)-3-[3-(2-methoxyphenylamino)-1-oxobut-2-en-1-yl]-1H-chromeno[4,3-*b*]pyridine-2,5-dione (5c). Yield 54% (94 mg), red powder, mp 229–230 °C. IR (ATR): 2930, 1727, 1677, 1564, 1247, 737 cm⁻¹; ¹H NMR (DMSO-*d*₆, ppm) δ 2.11 (s, 3H, Me), 3.70 (s, 3H, MeO), 3.88 (s, 3H, MeO), 6.50 (dd, *J* = 8.6 Hz, *J* = 1.3 Hz, 1H, H-10), 6.54 (s, 1H, =CH), 6.94 (ddd, *J* = 8.6 Hz, *J* = 8.5 Hz, *J* = 1.3 Hz, 1H, H-9), 6.99 (td, *J* = 7.7 Hz, *J* = 1.0 Hz, 1H, Ar), 7.15 (dd, *J* = 8.3 Hz, *J* = 1.0 Hz, 1H, Ar), 7.21 (td, *J* = 7.6 Hz, *J* = 0.9 Hz, 1H, Ar), 7.25 (td, *J* = 8.3 Hz, *J* = 1.5 Hz, 1H, Ar), 7.34–7.38 (m, 2H, Ar), 7.42 (dd, *J* = 7.7 Hz, *J* = 1.6 Hz, 1H, Ar), 7.48 (dd, *J* = 8.3 Hz, *J* = 1.2 Hz, 1H, H-7), 7.60 (ddd, *J* = 8.5 Hz, *J* = 8.3 Hz, *J* = 1.3 Hz, 1H, H-8), 7.67 (td, *J* = 8.0 Hz, *J* = 1.6 Hz, 1H, Ar), 8.87 (s, 1H, H-4), 13.12 (s, 1H, NH). Anal. Calcd for C₃₀H₂₄N₂O₆·0.25H₂O: C, 70.23; H, 4.81; N, 5.46. Found: C, 70.08; H, 4.69; N, 5.47.





















