

‘On water’ Knoevenagel condensation of isatins with malononitrile

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2-(2-Oxoindolin-3-ylidene)malononitrile 2a. Black-violet solid; mp 232–233 °C subl (lit. mp¹ 228–229 °C). ¹H NMR (300 MHz, DMSO-*d*₆): δ 6.93 (1H, d, *J*=7.3 Hz), 7.12 (1H, t, *J*=7.3 Hz), 7.56 (1H, t, *J*=7.7 Hz), 7.87 (1H, d, *J*=8.0 Hz), 11.19 (1H, s).

2-(1-Methyl-2-oxoindolin-3-ylidene)malononitrile 2b. Black-violet solid; mp 230.5–231.5 °C (lit. mp² 233 °C). ¹H NMR (300 MHz, DMSO-*d*₆): δ 3.15 (3H, s), 7.15 (1H, d, *J*=8.1 Hz), 7.20 (1H, t, *J*=8.4 Hz), 7.66 (1H, t, *J*=7.6 Hz), 7.91 (1H, d, *J*=8.1 Hz).

2-(1-Ethyl-2-oxoindolin-3-ylidene)malononitrile 2c. Black-violet solid; mp 207.5–208.5 °C. ¹H NMR (300 MHz, DMSO-*d*₆): δ 1.17 (3H, t, *J*=7.3 Hz), 3.73 (2H, q, *J*=7.3 Hz), 7.20 (2H, m), 7.66 (1H, t, *J*=7.6 Hz), 7.93 (1H, d, *J*=8.1 Hz). ¹³C NMR (75 MHz, DMSO-*d*₆): δ 12.20 (CH₃) 34.54 (CH₂N) 81.16 (=C(CN)₂) 110.50 (C-7) 111.39 (CN) 112.90 (CN) 118.11 (C-3a) 123.27 (C-5) 125.67 (C-4) 137.65 (C-6) 146.18 (C-7a) 149.77 (C-3) 162.00 (C-2). HRESI: C₁₃H₁₉N₃O Calc. for (M+Na) 246.0638. Found (M+Na) 246.0661. Calc. for (M+H) 224.0818. Found (M+H) 224.0811. IR (KBr, ν/cm⁻¹): 1716, 1612, 1596, 1476.

2-(1-Benzyl-2-oxoindolin-3-ylidene)malononitrile 2d. Black-violet solid; mp 189–190 °C (lit. mp³ 190–192 °C). ¹H NMR (300 MHz, DMSO-*d*₆): δ 4.94 (1H, s), 7.05 (1H, d, *J*=8.1 Hz), 7.19 (1H, t, *J*=7.7 Hz), 7.25–7.45 (5H, m), 7.58 (1H, t, *J*=8.1 Hz), 7.96 (1H, d, *J*=7.3 Hz).

2-(5-Chloro-2-oxoindolin-3-ylidene)malononitrile 2e. Brown-violet solid; mp 218–220 °C (lit. mp⁴ 220 °C). ¹H NMR (300 MHz, DMSO-*d*₆): δ 6.96 (1H, d, *J*=8.1 Hz), 7.63 (1H, dd, *J*=8.4 Hz, *J*=1.8 Hz), 7.76 (1H, d, *J*=2.2 Hz), 11.35 (1H, s).

2-(5-Bromo-2-oxoindolin-3-ylidene)malononitrile 2f. Brown-violet solid; mp 220–221 °C. ¹H NMR (300 MHz, DMSO-*d*₆): δ 6.90 (1H, d, *J*=8.4 Hz), 7.73 (1H, dd, *J*=8.4 Hz, *J*=1.8 Hz), 7.88 (1H, d, *J*=2.2 Hz), 11.35 (1H, s). ¹³C NMR (75 MHz, DMSO-*d*₆): δ 82.19 (C(CN)₂) 111.10

(CN) 112.66 (CN) 113.64 (C-7) 113.94 (C-5) 120.41 (C-3a) 127.56 (C-4) 139.65 (C-6) 145.49 (C-7a) 149.39 (C-3) 163.28 (C-2). HRESI: C₁₁H₄BrNO₃ Calc. for (M+Na) 295.943. Found (M+Na) 295.943. Calc. for (M-H) 271.946. Found (M-H) 271.946. IR (KBr, ν/cm^{-1}): 1728, 1612, 1596, 1464.

References

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