SUPPLEMENTARY MATERIAL

Cytotoxic substituted indolizines as new colchicine site tubulin polymerization inhibitors

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Spectral data for compounds 8a-l and 13a-d

\[ \begin{array}{ll}
\text{S2} & \text{1}^H \text{NMR and } \text{13}^C \text{NMR spectra of compound 11a} \\
\text{S8} & \text{1}^H \text{NMR and } \text{13}^C \text{NMR spectra of compound 11b} \\
\text{S9} & \text{1}^H \text{NMR and } \text{13}^C \text{NMR spectra of compound 11k} \\
\text{S10} & \text{1}^H \text{NMR and } \text{13}^C \text{NMR spectra of compound 14a} \\
\text{S11} & \text{1}^H \text{NMR and } \text{13}^C \text{NMR spectra of compound 15a} \\
\text{S12} & \text{1}^H \text{NMR and } \text{13}^C \text{NMR spectra of compound 15j} \\
\end{array} \]
Spectral data for compounds 8a-l and 13a-d

1-(2-oxo-2-(3,4,5-trimethoxyphenyl)ethyl)pyridin-1-ium bromide (8a). Yield 65%. All spectral data are in agreement with the literature.

1-(2-oxo-2-(3,5-dimethoxyphenyl)ethyl)pyridin-1-ium bromide (8b). Cream solid, yield 60%, mp 240-244 °C. IR ν(cm⁻¹): 1688, 1632, 1593, 1493, 1356, 1296, 1182, 1153, 1024. ¹H NMR (500 MHz, DMSO-d6): δ 3.85 (s, 6H, 2 x OMe), 6.50 (s, 2H, H₇), 6.93 (s, 1H, H₁₂), 7.18 (ad, J = 1.5 Hz, 2H, H₁₀, H₁₄), 8.27 (t, J = 7.5 Hz, 2H, H₃, H₅), 8.96 (t, J = 7.0 Hz, 1H, H₄), 9.00 (d, J = 6.0 Hz, 2H, H₂, H₆). ¹³C NMR (125 MHz, DMSO-d6): δ 55.8 2 x OMe, 66.4 C₇, 106.1 C₁₀, C₁₄, 106.2 C₁₂, 127.9 C₃, C₅, 135.4 C₉, 146.3 C₂, C₆, 146.5 C₄, 160.9 C₁₁, C₁₃, 190.5 C₈. Anal. Calcd. for C₁₅H₁₆BrNO₃: C, 53.27; H, 4.77; N, 4.14. Found: C, 53.25; H, 4.78; N, 4.18.

1-(2-(3,4-dimethoxyphenyl)-2-oxoethyl)pyridin-1-ium bromide (8c). Yellow solid, yield 30%, mp 194-196 °C. IR ν(cm⁻¹): 3038, 2953, 1591, 1580, 1524, 1493, 1348, 1279, 1180, 1138, 1013. ¹H NMR (500 MHz, DMSO-d6): δ 3.85 (3H, s, OMe), 3.90 (3H, s, OMe), 6.49 (s, 2H, H₇), 7.23 (d, J = 8.5 Hz, 1H, H₁₃), 7.52 (s, 1H, H₁₀), 7.77 (d, J = 8.5 Hz, 1H, H₁₄), 8.28 (t, J = 6.5 Hz, 2H, H₃, H₅), 8.73 (t, J = 7.5 Hz, 1H, H₄), 9.02 (d, J = 5.5 Hz, 2H, H₂, H₆). ¹³C NMR (125 MHz, DMSO-d6): δ 55.7 OMe, 56.0 OMe, 65.9 C₇, 110.2 C₁₀, 111.2 C₁₃, 123.3 C₁₄, 126.2 C₉, 127.8 C₃, C₅, 148.8 C₁₁, 146.2 C₂, C₆, 146.3 C₄, 154.3 C₁₂, 189.0 C₈. Anal. Calcd. for C₁₅H₁₆BrNO₃: C, 53.27; H, 4.77; N, 4.14. Found: C, 53.25; H, 4.78; N, 4.18.

1-(2-(4-bromophenyl)-2-oxoethyl)pyridin-1-ium bromide (8d). White solid, yield 60%, mp 240-243 °C. IR ν(cm⁻¹): 3020, 2905, 1692, 1636, 1584, 1489, 1234, 1179, 1070. ¹H NMR (400 MHz, DMSO-d6): δ 6.46 (s, 2H, H₇), 7.90 (d, J = 8.8 Hz, 2H, H₁₁, H₁₃), 7.99 (d, J = 8.8 Hz, 2H, H₁₀, H₁₄), 8.27 (dd, J = 7.6; 6.8 Hz, 2H, H₃, H₅), 8.74 (t, J = 8.0 Hz, 1H, H₄), 8.98 (d, J = 5.6 Hz, 2H, H₂, H₆). ¹³C NMR (100 MHz, DMSO-d6): δ 66.1 C₇, 127.8 C₃, C₅, 128.8 C₁₂, 130.2 C₁₀, C₁₄, 132.2 C₁₁, C₁₃, 132.6 C₉, 146.2 C₂, C₆, 146.4 C₄, 190.1 C₈. Anal. Calcd. for C₁₃H₁₁Br₂NO: C, 43.73; H, 3.11; N, 3.92. Found: C, 43.70; H, 3.08; N, 3.91.
1-(2-oxo-2-(3,4,5-trimethoxyphenyl)ethyl)-[4,4′-bipyridin]-1-ium bromide (8e). Cream solid, yield 50%, mp 240-242 °C; IR ν(cm⁻¹): 3010, 2978, 2940, 1688, 1641, 1584, 1462, 1415, 1344, 1317, 1161, 1119; 1H NMR (400 MHz, DMSO-d₆) δ ppm: 3.81 (s, 3H, OMe), 3.91 (s, 6H, 2 x OMe), 6.58 (s, 2H, H7), 7.39 (s, 2H, H10, H14), 8.18 (bs, 2H, 2 x Hpy), 8.78 (bs, 2H, H3, H5), 8.97 (bs, 2H, 2 x Hpy), 9.36 (bs, 2H, H2, H6); 13C NMR (100 MHz, DMSO-d₆) δ ppm: 56.4 (2 x OMe), 60.4 (OMe), 65.9 (C7), 106.1 (C10, C14), 122.5 (2 x CHpy), 125.3 (C3, C5), 128.7 (C9), 141.3 (Cpy), 143.1 (C12), 146.7 (C2, C6), 150.5 (C11, C13), 152.9 (C4), 153.0 (CHpy), 189.6 (C8); Anal. Calcd. for C₂₁H₂₁BrN₂O₄ (C, H, N): C, 56.64; H, 4.75; N, 6.29; Found: C, 56.65; H, 4.78; N, 6.26.

1-(2-oxo-2-(3,5-dimethoxyphenyl)ethyl)-[4,4′-bipyridin]-1-ium bromide (8f). Cream solid, yield 30%, mp 220-223 °C. IR ν(cm⁻¹): 3030, 2940, 1690, 1640, 1595, 1545, 1458, 1204, 1157, 1061). 1H NMR (500 MHz, DMSO-d₆): δ 3.86 (s, 3H, OMe), 3.91 (s, 3H, OMe), 6.56 (s, 2H, H7), 7.20 (d, J = 1.5 Hz, 2H, H10, H14), 8.08 (d, J = 5.5 Hz, 2H, 2 x Hpy), 8.75 (d, J = 6.5 Hz, 2H, H3, H5), 8.90 (d, J = 5.0 Hz, 2H, 2 x Hpy), 9.13 (d, J = 6.5 Hz, 2H, H2, H6). 13C NMR (125 MHz, DMSO-d₆): δ 55.8 2 x OMe, 66.0 C7, 106.1 C10, C14, 106.2 C12, 122.2 2 x CHpy, 125.3 C3, C5, 135.4 C9, 141.2 Cpy, 146.8 C2, C6, 150.8 2 x CHpy, 153.0 C4, 160.9 C11, C13, 190.4 C8. Anal. Calcd. for C₂₀H₁₉BrN₂O₃: C, 57.84; H, 4.61; N, 6.75. Found: C, 57.83; H, 4.60; N, 6.78.

1-(2-(3,4-dimethoxyphenyl)-2-oxoethyl)-[4,4′-bipyridin]-1-ium bromide (8g). Cream solid, yield 30%, mp 258-260 °C. IR ν(cm⁻¹): 3026, 2945, 1674, 1638, 1586, 1462, 1269, 1217, 1171, 1132, 1013, 816. 1H NMR (500 MHz, DMSO-d₆): δ 3.86 (s, 3H, OMe), 3.91 (s, 3H, OMe), 6.56 (s, 2H, H7), 7.24 (d, J = 8.5 Hz, 1H, H13), 7.54 (s, 1H, H10), 7.79 (d, J = 8.5 Hz, 1H, H14), 8.24 (bs, 2H, 2 x Hpy), 8.78 (d, J = 6.0 Hz, 2H, H3, H5), 8.97 (bs, 2H, 2 x Hpy), 9.20 (d, J = 6.0 Hz, 2H, H2, H6). 13C NMR (125 MHz, DMSO-d₆): δ 55.8 OMe, 56.0 OMe, 65.6 C7, 110.3 C10, 111.3 C13, 123.4 C14, 124.4 2 x CHpy, 125.2 C3, C5, 126.5 C9, 141.2 Cpy, 146.8 C2, C6, 148.9 C11, 152.9 2 x CHpy, 154.4 C12, C4, 188.9 C8. Anal. Calcd. for C₂₀H₁₉BrN₂O₃: C, 57.84; H, 4.61; N, 6.75. Found: C, 57.83; H, 4.64; N, 6.78.
1-(2-(4-bromofenil)-2-oxoetil)-[4,4'-bipyridin]-1-ium bromide (8h). Yield 65%. All spectral data are in agreement with the literature.2-3

1'-2-(3,4,5-trimethoxyphenyl)-2-oxoethyl)-[2,2'-bipyridin]-1'-ium bromide (8i). White solid, yield 64% , mp 198–199 °C. IR ν(cm⁻¹): 3066, 3014, 2950, 2835, 1694, 1637, 1592, 1462, 1411, 1349, 1321, 1162, 1117, 992. ¹H NMR (500 MHz, DMSO-d₆): δ ppm 3.81 (s, 3H, OMe), 3.91 (s, 6H, 2 x OMe), 6.54 (s, 2H, H₇), 7.38 (s, 2H, H₁₀, H₁₄), 7.71 (dd, J = 7.5; 4.5 Hz, 1H, H₉py), 8.16 (dt, J = 8.0; 1.5 Hz, 1H, H₉py), 8.49 (d, J = 7.5 Hz, 1H, H₉py), 8.91 (dd, J = 5.0; 1.5 Hz, 1H, H₉py), 8.93 (d, J = 7.0 Hz, 2H, H₁₂, H₁₆). ¹³C NMR (125 MHz, DMSO-d₆): δ 56.4 2 x OMe, 60.4 OMe, 65.8 C₇, 106.0 C₁₀, C₁₄, 123.7 CH₉py, 124.2 C₃, C₅, 126.8 CH₉py, 128.8 C₉, 138.4 CH₉py, 143.2 C₁₂, 146.7 C₂, C₆, 149.8 C₇py, 150.8 CH₉py, 153.1 C₁₁, C₁₃, 153.6 C₄, 189.8 C₈. Anal. Calcd. for C₂₁H₂BrN₂O₄: C, 56.64; H, 4.75; N, 6.29. Found: C, 56.62; H, 4.70; N, 6.31.

1'-2-(3,5-dimethoxyphenyl)-2-oxoethyl)-[2,2'-bipyridin]-1'-ium bromide (8j). White solid, yield 92%, mp 245-246 °C. IR ν(cm⁻¹): 3012, 2937, 1694, 1647, 1594, 1451, 1351, 1294, 1209, 1157, 1023, 847. ¹H NMR (500 MHz, DMSO-d₆): δ 3.86 (s, 6H, 2 x OMe), 6.47 (s, 2H, H₇), 6.95 (bs, 1H, H₁₂), 7.19 (d, J = 2.0 Hz, 2H, H₁₀, H₁₄), 7.71 (dd, J = 7.0; 5.0 Hz, 1H, H₉py), 8.16 (dt, J = 7.5; 1.5 Hz, 1H, H₉py), 8.49 (d, J = 7.5 Hz, 1H, H₉py), 8.91 (dd, J = 5.0; 1.5 Hz, 1H, H₉py), 8.93 (d, J = 7.0 Hz, 2H, H₁₂, H₁₆). ¹³C NMR (125 MHz, DMSO-d₆): δ 55.8 2 x OMe, 65.9 C₇, 106.1 C₁₀, C₁₄, 106.2 C₁₂, 123.6 C₂₀, 124.2 C₃, C₅, 126.8 C₁₈, 135.5 C₉, 138.4 C₁₉, 146.7 C₂, C₆, 149.8 C₁₅, 150.8 C₁₇, 153.6 C₄, 160.9 C₁₁, C₁₃, 190.6 C₈. Anal. Calcd. for C₂₀H₁₉BrN₂O₃: C, 57.84; H, 4.61; N, 6.75. Found: C, 57.80; H, 4.57; N, 6.77.

1'-2-(3,4-dimethoxyphenyl)-2-oxoethyl)-[2,2'-bipyridin]-1'-ium bromide (8k). White solid, yield 76%, mp 263-264 °C. IR ν(cm⁻¹): 3041, 2950, 2891, 1680, 1641, 1587, 1523, 1474, 1346, 1273, 1141, 1018, 778. ¹H NMR (500MHz, DMSO-d₆): δ 3.86 (s, 3H, OMe), 3.91 (s, 3H, OMe), 6.49 (s, 2H, H₇), 7.24 (d, J = 8.5 Hz, 1H, H₁₀), 7.53 (d, J = 2.0 Hz, 1H, H₁₃), 7.53 (d, J = 8.5 Hz, 1H, H₁₃), 7.71 (dd, J = 7.5; 5.0 Hz, 1H, H₉py), 7.78 (dd, J = 8.5; 2.0 Hz, 1H, H₁₄), 8.16 (dt, J = 8.0; 2.0 Hz, 1H, H₉py), 8.49 (d, J = 8.0 Hz, 1H, H₉py), 8.91 (dd, J = 5.0; 2.0 Hz, 1H, H₉py), 8.92 (d, J = 7.0 Hz,
2H, H3, H5), 9.10 (d, J = 6.5 Hz, 2H, H2, H6). 13C-NMR (125 MHz, DMSO-d6): δ 55.8 OMe, 56.0 OMe, 65.5 C7, 110.3 C10, 111.3 C13, 123.4 CHpy, 123.6 C14, 124.1 C3, C5, 126.3 C9, 126.8 CHpy, 138.3 CHpy, 146.8 C2, C6, 148.9 C11, 149.8 Cpy, 150.8 CHpy, 154.3 C12, 153.5 C4, 189.1 Cs. Anal. Calcd. for C20H15BrN2O3: C, 57.84; H, 4.61; N, 6.75. Found: C, 57.82; H, 4.60; N, 6.79.

1'-([2,4'-bipyridin]-1'-ium) bromide (8i). Cream solid, yield 94%, mp 253-255 °C. IR ν(cm−1): 2987, 2936, 1693, 1636, 1578, 1341, 988, 775. 1H NMR (500MHz, DMSO-d6): δ 6.47 (s, 2H, H7), 7.71 (dd, J = 8.0; 5.5 Hz, 1H, Hpy), 7.92 (d, J = 8.5 Hz, 2H, H11, H13), 8.02 (d, J = 8.5 Hz, 2H, H10, H14), 8.16 (dt, J = 8.0; 1.5 Hz, 1H, Hpy), 8.48 (d, J = 8.0 Hz, 1H, Hpy), 8.91 (dd, J = 5.5; 1.5 Hz, 1H, Hpy), 8.93 (d, J = 7.0 Hz, 2H, H3, H5), 9.07 (d, J = 7.0 Hz, 2H, H2, H6). 13C-NMR (125 MHz, DMSO-d6): δ 65.7 C7, 123.6 CHpy, 124.2 C3, C5, 126.8 CHpy, 128.9 C12, 130.2 C10, C14, 132.3 C11, C13, 132.7 C9, 138.4 CHpy, 146.8 C2, C6, 149.8 Cpy, 150.8 CHpy, 153.6 C4, 190.2 C8. Anal. Calcd. for C18H14BrN2O: C, 49.80; H, 3.25; N, 6.45. Found: C, 49.82; H, 3.20; N, 6.49.

1-(2-oxo-2-(3,4,5-trimethoxyphenyl)ethyl)-[2,2'-bipyridin]-1'-ium bromide (13a). Cream solid, yield 65%, mp 197-199 °C. IR ν(cm−1): 3057, 3016, 2977, 2945, 1692, 1583, 1416, 1318, 1161, 1124, 991. 1H NMR (500MHz, DMSO-d6): δ 3.78 (s, 3H, OMe), 3.84 (s, 6H, 2 x OMe), 6.53 (s, 2H, H7), 7.26 (s, 2H, H10, H14), 7.62 (ddd, J = 7.5; 5.0; 1.0 Hz, 1H, H18), 8.10 (d, J = 7.5 Hz, 1H, H20), 8.15 (dt, J = 8.0; 1.5 Hz, 1H, H19), 8.39-8.42 (overlapped signals, 2H, H17, H3), 8.55 (dd, J = 8.0; 1.0 Hz, 1H, H5), 8.90 (dt, J = 7.5; 1.0 Hz, 1H, H4), 9.23 (dd, J = 6.0; 1.0 Hz, 1H, H6). 13C NMR (125 MHz, DMSO-d6): δ 56.3 2 x OMe, 60.3 OMe, 64.6 C7, 106.0 C10, C14, 126.1 C18, 126.7 C20, 127.5 C5, 128.6 C9, 130.1 C3, 138.6 C19, 142.8 C12, 147.2 C15, 148.9 C4, 149.0 C6, 149.1 C17, 151.3 C2, 153.0 C11, C13, 189.3 C8. Anal. Calcd. for C21H23BrN2O4: C, 56.64; H, 4.75; N, 6.29. Found: C, 56.61; H, 4.72; N, 6.33.

1-(2-(3,5-dimethoxyphenyl)-2-oxoethyl)-[2,2'-bipyridin]-1'-ium bromide (13b). Cream solid, yield 22%, mp 195-196 °C. IR ν(cm−1): 3057, 3027, 2988, 2973, 1687, 1592, 1443, 1294, 1200, 1150, 1021, 765. 1H NMR (500MHz, DMSO-d6): δ 3.82 (s, 6H, 2 x OMe), 6.40 (s, 2H, H7),
6.89 (t, J = 2.0 Hz, 1H, H12), 7.26 (d, J = 2.0 Hz, 2H, H10, H14), 7.61 (ddd, J = 7.5; 4.5; 1.0 Hz, 1H, H18), 8.09 (d, J = 8.0 Hz, 1H, H20), 8.16 (dt, J = 8.0; 1.5 Hz, 1H, H19), 8.38 (dd, J = 6.5; 1.5 Hz, 1H, H17), 8.39 (dt, J = 7.5; 1.5 Hz, 1H, H5), 8.54 (dd, J = 8.0; 1.0 Hz, 2H, H3), 8.89 (dt, J = 8.0; 1.5 Hz, 1H, H4), 9.16 (dd, J = 6.0; 1.0 Hz, 1H, H6). 13C NMR (125 MHz, DMSO-d6): δ 55.7 2 x OMe, 64.8 C7, 105.9 C10, C14, C12, 126.1 C18, 126.7 C20, 127.5 C5, 130.0 C3, 135.3 C9, 138.6 C19, 147.2 C4, 148.9 C6, 149.0 C17, C15, 151.2 C2, 160.8 C11, C13, 189.9 C8. Anal. Calcd. for C20H19BrN2O3: C, 57.84; H, 4.61; N, 6.75. Found: C, 57.81; H, 4.56; N, 6.78.

1-(2-(3,4-dimethoxyphenyl)-2-oxoethyl)-[2,2′-bipyridin]-1-ium bromide (13c). Cream solid, yield 33%, mp 199-201 °C. IR ν(cm−1): 3060, 3032, 2979, 1686, 1583, 1515, 1419, 1270, 1165, 1151, 1012, 770. 1H NMR (500MHz, DMSO-d6): δ 3.81 (s, 3H, OMe), 3.88 (s, 3H, OMe), 6.43 (s, 2H, H2), 7.14 (d, J = 8.5 Hz, 1H, H13), 7.43 (d, J = 1.5 Hz, 1H, H10), 7.60 (dd, J = 7.0; 5.0 Hz, 1H, H18), 7.64 (dd, J = 8.5; 1.5 Hz, 1H, H14), 8.06 (d, J = 8.0 Hz, 1H, H20), 8.14 (dt, J = 8.0; 1.0 Hz, 1H, H19), 8.37-8.40 (overlapped signals, 2H, H17, H5), 8.52 (d, J = 8.0 Hz, 2H, H3), 8.88 (t, J = 8.0 Hz, 1H, H4), 9.19 (d, J = 6.0 Hz, 1H, H6). 13C NMR (125 MHz, DMSO-d6): δ 55.7 OMe, 56.0 OMe, 66.3 C7, 110.2 C10, 111.2 C13, 123.1 C14, 126.0 C9, 126.1 C18, 126.7 C20, 127.4 C5, 130.0 C3, 138.6 C19, 147.1 C4, 148.9 C11, 149.0 C6, C17, 149.2 C15, 151.4 C2, 154.1 C12, 188.6 C8. Anal. Calcd. for C20H19BrN2O3: C, 57.84; H, 4.61; N, 6.75. Found: C, 57.80; H, 4.57; N, 6.79.

1-(2-(3-bromophenyl)-2-oxoethyl)-[2,2′-bipyridin]-1-ium bromide (13d). Cream solid, yield 20%, mp 207-210°C. IR ν(cm−1) 3055, 3018, 2979, 2919, 1704, 1583, 1442, 1225, 992, 774. 1H NMR (500MHz, DMSO-d6): δ 6.40 (s, 2H, H2), 7.59 (dd, J = 7.5; 5.5 Hz, 1H, H18), 7.85 (d, J = 8.5 Hz, 2H, H11, H13), 7.93 (d, J = 8.5 Hz, 2H, H10, H14), 8.10 (d, J = 8.0 Hz, 1H, H20), 8.16 (t, J = 8.0 Hz, 1H, H19), 8.26 (d, J = 4.5 Hz, 1H, H17), 8.40 (t, J = 6.5 Hz, 1H, H5), 8.54 (d, J = 7.5 Hz, 2H, H3), 8.89 (t, J = 8.0 Hz, 1H, H4), 9.17 (d, J = 6.0 Hz, 1H, H6). 13C NMR (125 MHz, DMSO-d6): δ 64.5 C7, 126.2 C18, 126.8 C20, 127.5 C5, 128.6 C12, 130.0 C3, 130.1 C10, C14, 132.3 C11, C13, 132.5 C9, 138.7 C19, 147.2 C4, 148.9 C17, 149.0 C15, 149.1 C6, 151.0 C2, 189.6 C8. Anal. Calcd. for C18H14Br2N2O: C, 49.80; H, 3.25; N, 6.45. Found: C, 49.83; H, 3.21; N, 6.48.
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$^1$H NMR and $^{13}$C NMR spectra of compound 11a
\(^1\)H NMR and \(^{13}\)C NMR spectra of compound 11b
$^1$H NMR and $^{13}$C NMR spectra of compound 11k
\(^1\)H NMR and \(^{13}\)C NMR spectra of compound 14a
$^1$H NMR and $^{13}$C NMR spectra of compound 15a
$^1$H NMR and $^{13}$C NMR spectra of compound 15j