

Synthesis of Spirocyclic Carbazole- and Acridine-Lactams

Martina Würdemann and Jens Christoffers*

Institut für Reine und Angewandte Chemie, Carl von Ossietzky-Universität
Oldenburg, D-26111 Oldenburg, Germany; Fax: +49 441 / 798 3873; Tel. +49 441 /
798 4744; E-mail: jens.christoffers@uni-oldenburg.de

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General methods. Preparative column chromatography was carried out using Merck SiO₂ (0.035–0.070 mm, type 60 A) with ethyl acetate (EA), *tert*-butyl methyl ether (MTBE), toluene or methanol (MeOH) as eluents. TLC was performed on Merck SiO₂ F₂₅₄ plates on aluminium sheets. ¹H- and ¹³C-NMR spectra were recorded on a Bruker Avance DRX 500 and Avance DPX 300 at 23°C. Multiplicities in ¹³C-NMR were determined with DEPT experiments. EI-MS, CI-MS and HR-MS spectra were obtained with a Finnigan MAT 95 spectrometer, GC-MS (EI) spectra with a Focus GC and a DSQ MS-detector (Thermo-Fisher). IR spectra were recorded on a Bruker Tensor 27 spectrometer equipped with a "GoldenGate" diamond-ATR unit. *ortho*-Aminobenzaldehyde was always freshly prepared by reduction of nitrobenzaldehyde with iron-powder as reported previously.¹⁷ All other starting materials were commercially available.

1-Allyl-2-piperidone (5b): Allyl bromide (54.0 ml, 75.0 g, 620 mmol) was added dropwise to a stirred suspension of 2-piperidone (41.0 g, 414 mmol) and KOH powder (34.8 g, 620 mmol) in DMF (120 ml). The mixture was further stirred at 60°C for 40 h, then diluted with water (150 ml) and extracted with CH₂Cl₂ (3 x 150 ml). The combined organic extracts were dried (MgSO₄). After filtration and evaporation of the solvent the residue was submitted to vacuum distillation through a 10-cm Vigreux column. The product **5b** was obtained as the main fraction (bp. 92°C at 4 mbar) and as a colorless liquid (28.0 g, 201 mmol, 49%). ¹H-NMR (CDCl₃, 500 MHz): δ = 1.76–1.84 (m, 4H), 2.40–2.45 (m, 2H), 3.20–3.28 (m, 2H), 4.01 (d, br, *J* = 6 Hz, 2H), 5.16 (d, br, *J* = 16 Hz, 1H), 5.18 (d, br, *J* = 11 Hz, 1H), 5.77 (ddt, *J* = 16 Hz, *J* = 11 Hz, *J* = 6 Hz, 1H) ppm. ¹³C{¹H}-NMR (CDCl₃, 125 MHz): δ = 21.25 (CH₂), 23.01 (CH₂), 32.15

(CH₂), 47.08 (CH₂), 49.14 (CH₂), 116.94 (CH₂), 132.70 (CH), 169.37 (C) ppm. MS (EI, 70 eV): *m/z* (%) = 139 (56) [M⁺], 124 (100), 96 (36), 70 (44). IR (ATR): 3082 (w), 2958 (s), 2870 (m), 1632 (vs), 1495 (s), 1467 (m), 1449 (m), 1416 (s), 1353 (s), 1334 (m), 1283 (m), 1270 (m), 1180 (m), 1167 (m), 923 (s), 668 (s) cm⁻¹. HRMS: calcd. 139.0997 (for C₈H₁₃NO), found 139.0994 [M⁺]. C₈H₁₃NO (139.20).

3-Acetyl-1-allyl-2-piperidone (4b): LDA (234 mmol, 130 ml of a 1.8 mol dm⁻³ solution in THF/heptane/ethylbenzene) was added dropwise over a period of 30 min to a stirred and cooled (dry ice-acetone bath) solution of lactam **5b** (27.5 g, 198 mmol) in abs. THF (200 ml). Subsequently, MeOAc (39.0 ml, 36.3 g, 491 mmol) was added in one portion and the mixture was stirred and warmed to ambient temperature (30 min). Half concentrated hydrochloric acid (100 ml) was added, the layers were separated and the aqueous layer was extracted with CH₂Cl₂ (3 x 150 ml). The combined organic layers were dried (MgSO₄), the solvent was removed and the residue submitted to chromatography (SiO₂, MTBE, R_f = 0.19–0.62) to give the title compound **4b** as a colorless oil (28.0 g, 154 mmol, 78%). Alternatively, the product can be purified by vacuum distillation (bp. 100–104°C at 1.1 mbar). NMR spectra showed a doubled set of signals due to keto-enol-tautomerism (ratio ca. 4 : 3). ¹H-NMR (CDCl₃, 500 MHz): δ = 1.68–1.74 (m, 1H), 1.75–1.81 (m, 2H), 1.81–1.86 (m, 2H), 1.88 (s, 3H), 2.06–2.11 (m, 1H), 2.30 (s, 3H), 2.33–2.35 (m, 2H), 3.17–3.26 (m, 4H), 3.47 (t, *J* = 6.5 Hz, 1H), 3.90–3.99 (m, 4H), 5.09–5.12 (m, 4H), 5.66 – 5.76 (m, 2H), 14.80 (s, br, 1H) ppm. ¹³C{¹H}-NMR (CDCl₃, 125 MHz): δ = 18.39 (CH₃), 20.77 (CH₂), 22.40 (CH₂), 23.50 (CH₂), 23.96 (CH₂), 30.00 (CH₃), 47.08 (2 x CH₂), 48.87 (CH₂), 49.47 (CH₂), 55.26 (CH), 95.51 (C), 116.75 (CH₂), 117.29 (CH₂), 132.09 (CH), 132.60 (CH), 166.11 (C), 168.69 (C), 169.98 (C), 205.67 (C) ppm. MS (EI, 70 eV): *m/z* (%) = 181 (79) [M⁺], 166 (56), 138 (100), 124 (25), 110 (24), 96 (23), 70 (31). IR (ATR): 3082 (w), 2945 (m), 2864 (m), 1717 (m), 1631 (vs), 1595 (s), 1491 (s), 1464 (m), 1445 (m), 1418 (m), 1386 (m), 1354 (m), 1334 (m), 1323 (m), 1262 (vs), 1205 (s), 1176 (m), 1160 (m), 996 (m), 948 (s), 927 (s), 765 (m), 695 (m), 671 (m) cm⁻¹. HRMS: calcd. 181.1103 (for C₁₀H₁₅NO₂), found 181.1105 [M⁺]. C₁₀H₁₅NO₂ (181.23).

3-Acetyl-1-allyl-3-(3-oxobutyl)-2-piperidone (6b): $\text{FeCl}_3 \cdot 6 \text{H}_2\text{O}$ (4.05 g, 15.0 mmol) and MVK (24.3 ml, 21.0 g, 300 mmol) were subsequently added to a stirred solution of lactam **4b** (27.2 g, 150 mmol) in CH_2Cl_2 (75 ml). After stirring the mixture for 16 h at 23°C, water (150 ml) was added, the layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 100 ml). The combined organic layers were dried (MgSO_4), the solvent was removed and the residue submitted to chromatography on SiO_2 (MTBE, $R_f = 0.26$) to yield the title compound **6b** (35.8 g, 142 mmol, 95%) as a colorless oil. ^1H -NMR (CDCl_3 , 500 MHz): $\delta = 1.56\text{--}1.60$ (m, 1H), 1.72–1.79 (m, 1H), 1.85–1.92 (m, 1H), 2.11–2.21 (m, 3H), 2.13 (s, 3H), 2.22 (s, 3H), 2.43–2.49 (m, 1H), 2.53–2.59 (m, 1H), 3.28 (t, $J = 6.1$ Hz, 2H), 3.91 (dd, $J = 14.9$ Hz, $J = 5.8$ Hz, 1H), 4.06 (dd, $J = 14.9$ Hz, $J = 5.7$ Hz, 1H), 5.16 (d, br, $J = 17$ Hz, 1H), 5.19 (d, br, $J = 10$ Hz, 1H), 5.76 (ddt, br, $J = 17$ Hz, $J = 10$ Hz, $J = 6$ Hz, 1H) ppm. $^{13}\text{C}\{^1\text{H}\}$ -NMR (CDCl_3 , 125 MHz): $\delta = 19.62$ (CH_2), 26.68 (CH_3), 29.11 (CH_2), 29.27 (CH_2), 29.90 (CH_3), 39.00 (CH_2), 47.32 (CH_2), 49.80 (CH_2), 58.91 (C), 117.47 (CH_2), 132.24 (CH), 169.198 (C), 206.94 (C), 207.92 (C) ppm. MS (EI, 70 eV): m/z (%) = 251 (5) [M^+], 209 (18), 208 (21), 181 (18), 166 (9), 152 (100). IR (ATR): 3079 (w), 2943 (m), 2874 (m), 1707 (vs), 1626 (vs), 1490 (m), 1444 (m), 1416 (m), 1353 (s), 1275 (m), 1167 (s), 995 (m), 927 (m) cm^{-1} . HRMS: calcd. 251.1521 (for $\text{C}_{14}\text{H}_{21}\text{NO}_3$), found 251.1524 [M^+]. $\text{C}_{14}\text{H}_{21}\text{NO}_3$ (251.32).

2-Allyl-7-methyl-2-aza-spiro[5.5]undec-7-ene-1,9-dione (9b): Pyrrolidine (11.6 ml, 9.89 g, 139 mmol) was added to a cooled (ice-water-bath) and stirred solution of lactam **6b** (35.0 g, 139 mmol) in EtOAc (40 ml). Subsequently, glacial acetic acid (8.0 ml, 8.3 g, 139 mmol) was added, the cooling bath was removed and the mixture stirred for 16 h at ambient temperature. After concentrating the reaction mixture to half of its volume, it was directly transferred on top of a SiO_2 -column and chromatographed (MTBE, $R_f = 0.14$) to give the title compound **9b** (28.3 g, 121 mmol, 87%) as a colorless oil. ^1H -NMR (CDCl_3 , 500 MHz): $\delta = 1.84\text{--}1.98$ (m, 6H), 2.00–2.05 (m, 1H), 2.13 (dt, $J = 13.4$ Hz, $J = 4.3$ Hz, 1H), 2.38–2.49 (m, 2H), 2.55 (td, $J = 13.1$ Hz, $J = 5.4$ Hz, 1H), 3.27–3.43 (m, 1H), 3.40 (td, $J = 10.6$ Hz, $J = 4.9$ Hz, 1H), 3.98 (dd, $J = 14.9$ Hz, $J = 6.0$ Hz, 1H), 4.07 (dd, $J = 14.9$ Hz, $J = 6.0$ Hz, 1H), 5.18 (d, br, $J = 17$ Hz, 1H), 5.20 (d, br, $J = 10$ Hz, 1H), 5.77 (ddt, br, $J = 17$ Hz, $J = 10$ Hz, $J = 6$ Hz, 1H), 5.92 (s, br, 1H) ppm. $^{13}\text{C}\{^1\text{H}\}$ -NMR (CDCl_3 , 125 MHz): $\delta = 19.73$ (CH_2), 20.57 (CH_3), 28.64 (CH_2), 31.13 (CH_2), 32.29 (CH_2), 47.45 (CH_2), 48.06 (C), 50.03 (CH_2), 117.54

(CH₂), 128.39 (CH), 132.30 (CH), 163.61 (C), 171.32 (C), 197.75 (C) ppm. MS (EI, 70 eV): m/z (%) = 233 (38) [M⁺], 205 (88), 177 (100). IR (ATR): 2948 (m), 1668 (s), 1622 (vs), 1492 (m), 1460 (m), 1444 (m), 1418 (m), 1354 (m), 1278 (s), 1234 (m), 1202 (s), 1186 (m), 928 (m), 734 (m) cm⁻¹. HRMS: calcd. 233.1416 (for C₁₄H₁₉NO₂), found 233.1413 [M⁺]. C₁₄H₁₉NO₂ (233.31).

7-Methyl-2-aza-spiro[5.5]undec-7-ene-1,9-dione (8b): Pd(OAc)₂ (360 mg, 1.6 mmol, 0.04 eq) was added to solution of lactam **9b** (9.36 g, 40.1 mmol) in H₂O (20 ml) and TFA (20 ml). The resulting mixture was stirred for 16 h at 80°C. All volatile materials were removed in vacuo, and the residue dissolved in MeOH (5 ml) and chromatographed on SiO₂ (MTBE/MeOH 5 : 1, R_f = 0.17) to give the title compound **8b** (5.69 g, 29.4 mmol, 73%) as a colorless oil. ¹H-NMR (CDCl₃, 500 MHz): δ = 1.84–1.95 (m, 3H), 1.98 (s, 3H), 2.02–2.05 (m, 1H), 2.17 (dt, J = 13.0 Hz, J = 4.2 Hz, 1H), 2.39–2.47 (m, 2H), 2.49–2.56 (m, 1H), 3.39–3.42 (m, 2H), 5.95 (s, 1H), 6.42 (s, 1H) ppm. ¹³C{¹H}-NMR (CDCl₃, 125 MHz): δ = 19.73 (CH₂), 20.51 (CH₃), 28.12 (CH₂), 30.92 (CH₂), 32.11 (CH₂), 42.22 (CH₂), 47.51 (C), 128.65 (CH), 162.83 (C), 174.41 (C), 197.63 (C) ppm. MS (EI, 70 eV): m/z (%) = 193 (8) [M⁺], 178 (5), 176 (3), 165 (88), 137 (100). IR (ATR): 3289 (w), 3175 (m), 3038 (m), 2947 (m), 2873 (m), 1651 (vs), 1488 (m), 1461 (m), 1424 (s), 1372 (m), 1355 (m), 1336 (m), 1318 (m), 1263 (m), 1235 (m), 1208 (m), 1191 (m), 1125 (m), 913 (m), 866 (m), 849 (s), 777 (m), 666 (m), 632 (m) cm⁻¹. HRMS: calcd. 193.1103 (for C₁₁H₁₅NO₂), found 193.1099 [M⁺]. C₁₁H₁₅NO₂ (193.24).

cis-7-Methyl-2-azaspiro[5.5]undecane-1,9-dione (7b): A mixture of lactam **8b** (4.32 g, 22.4 mmol), Pd/C (223 mg, 10% w/w Pd), and *i*PrOH (35 ml) was degassed (three cycles of freeze, pump, thaw) and stirred at 40°C for 16 h under an atmosphere of H₂ (balloon). The solvent was removed in vacuo and the residue chromatographed on SiO₂ (MTBE/MeOH 5 : 1, R_f = 0.38) to give the title compound **7b** (3.20 g, 16.4 mmol, 73%) as a colorless solid, m.p. 112–113°C. ¹H-NMR (CDCl₃, 500 MHz): δ = 1.05 (d, J = 6.7 Hz, 3H; 7-CH₃), 1.50 (dt, J = 13.6 Hz, J = 4.0 Hz, 1H; 5-H), 1.60 (ddd, J = 13.9 Hz, J = 12.3 Hz, J = 4.5 Hz, 1H; 11-H), 1.74–1.93 (m, 3H; 7-H, 2 x 4-H), 2.02 (td, J = 13.4 Hz, J = 3.9 Hz, 1H; 5-H), 2.21 (ddd, J = 15.0 Hz, J = 4.8 Hz, J = 1.3 Hz, 1H; 8-H), 2.23–2.27 (m, 1H; 10-H), 2.43 (dt, J = 14.2 Hz, J = 5.3 Hz, 1H; 11-H), 2.80 (ddd, J = 15.1 Hz, J = 12.3 Hz, J = 5.6 Hz, 1H; 10-H), 3.11 (dd, J = 15.0 Hz, J = 12.4

Hz, 1H; 8-H), 3.29–3.30 (m, 2H; 2 x 3-H), 6.38 (s, 1H; 2-H) ppm. $^{13}\text{C}\{^1\text{H}\}$ -NMR (CDCl_3 , 125 MHz): δ = 17.37 (CH_3), 19.74 (CH_2 ; C-4), 33.95 (CH_2 ; C-5), 35.84 (CH_2 ; C-11), 38.15 (CH_2 ; C-10), 40.72 (CH, C-7), 42.54 (CH_2 ; C-3), 43.15 (C; C-6), 47.25 (CH_2 ; C-8), 175.64 (C; C-1), 213.46 (C; C-9) ppm. MS (EI, 70 eV): m/z (%) = 195 (23) [M^+], 167 (17), 152 (27), 126 (21), 112 (100). IR (ATR): 3281 (w), 3189 (m), 3065 (m), 2945 (s), 2878 (m), 2854 (m), 1711 (vs), 1646 (vs), 1493 (m), 1415 (s), 1315 (s), 1199 (s), 847 (s), 630 (s) cm^{-1} . HRMS calcd. 195.1259 (for $\text{C}_{11}\text{H}_{17}\text{NO}_2$), found 195.1255 [M^+]. $\text{C}_{11}\text{H}_{17}\text{NO}_2$ (195.26).

Fischer-indolization of lactam 7b: A mixture of ketone **7b** (207 mg, 1.06 mmol), glacial AcOH (2.6 ml), TFA (0.9 ml) and PhNHNH₂ (126 mg, 1.17 mmol) was stirred for 16 h at 100°C in a tightly closed reaction vial. The mixture was poured onto ice-water (ca. 20 g) and the resulting emulsion extracted with CH_2Cl_2 (3 x 10 ml). The combined organic layers were dried (MgSO_4) and the solvent removed under reduced pressure. The residue was chromatographed on SiO_2 (EtOAc) to yield carbazole **10b** (79 mg, 0.29 mmol, 28%) in the first fraction (R_f = 0.31) and carbazole **11b** (143 mg, 0.53 mmol, 50%) in the second fraction (R_f = 0.20).

cis-1,2,3,4-Tetrahydro-2-methylspiro[carbazole-3,3'-piperidine]-2'-one (10b): Colorless solid, mp. 223–224°C. ^1H -NMR ($\text{CDCl}_3/\text{CD}_3\text{OD}$ 1 : 1, 500 MHz): δ = 1.12 (d, J = 7.0 Hz, 3H; 2-Me), 1.50–1.56 (m, 1H; 4'-H), 1.74–1.81 (m, 1H; 5'-H), 1.88–1.97 (m, 1H; 5'-H), 2.02 (ddd, J = 14.0 Hz, J = 6.2 Hz, J = 3.4 Hz, 1H; 4'-H), 2.26–2.32 (m, 1H; 2-H), 2.45 (d, J = 15.9 Hz, 1H; 4-H), 2.56 (dd, J = 16.7 Hz, J = 2.0 Hz, 1H; 1-H), 2.96 (dd, J = 16.7 Hz, J = 5.5 Hz, 1H; 1-H), 3.25–3.35 (m, 2H; 6'-H), 3.42 (d, J = 15.9 Hz, 1H; 4-H), 6.98–7.01 (m, 1H; 6-H), 7.04–7.07 (m, 1H; 7-H), 7.28 (d, J = 7.9 Hz, 1H; 8-H), 7.39 (d, J = 7.7 Hz, 1H; 5-H) ppm. $^{13}\text{C}\{^1\text{H}\}$ -NMR ($\text{CDCl}_3/\text{CD}_3\text{OD}$ 1 : 1, 125 MHz): δ = 17.11 (CH_3 ; 2-Me), 18.37 (CH_2 ; C-5'), 27.99 (CH_2 ; C-4), 28.31 (CH_2 ; C-1), 31.42 (CH_2 ; C-4'), 33.42 (CH; C-2), 42.50 (CH_2 ; C-6'), 44.32 (C; C-3), 106.43 (C; C-4a), 111.16 (CH; C-8), 117.78 (CH; C-5), 118.97 (CH; C-6), 121.02 (CH; C-7), 128.46 (C; C-4b), 131.75 (C; C-9a), 137.21 (C; C-8a), 178.50 (C; C-2') ppm. MS (EI, 70 eV): m/z (%) = 268 (48) [M^+], 168 (12), 157 (100), 143 (48), 130 (13), 89 (11), 45 (38). IR (ATR): 3402 (m), 3287 (m), 3055 (w), 2930 (m), 1644 (vs), 1454 (s), 1327 (s), 1200 (m), 1005 (m), 739 (vs) cm^{-1} . HRMS calcd. 268.1576 (for $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}$), found 268.1568 [M^+]. $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}$ (268.35).

cis-1,2,3,4-Tetrahydro-4-methylspiro[carbazole-3,3'-piperidine]-2'-one (11b): Colorless solid, mp. 169–170°C. $^1\text{H-NMR}$ ($\text{CDCl}_3/\text{CD}_3\text{OD}$ 1 : 1, 500 MHz): δ = 1.20 (d, J = 7.0 Hz, 3H; 4-Me), 1.41 (ddd, J = 13.7, J = 13.5 Hz, J = 3.8 Hz, 1H; 4'-H), 1.47–1.51 (m, 1H; 2-H), 1.55–1.60 (m, 1H; 5'-H), 1.85–1.93 (m, 2H; 4'-H, 5'-H), 2.48–2.60 (m, 2H; 1-H, 2-H), 2.65–2.70 (m, 1H; 1-H) 3.15–3.27 (m, 3H; 4-H, 2 x 6'-H), 6.89–6.93 (m, 1H; 6-H), 6.94–6.98 (m, 1H; 7-H), 7.19 (d, J = 8.0 Hz, 1H; 8-H), 7.34 (d, J = 7.3 Hz 1H; 5-H) ppm. $^{13}\text{C}\{^1\text{H}\}\text{-NMR}$ ($\text{CDCl}_3/\text{CD}_3\text{OD}$ 1 : 1, 125 MHz): δ = 17.59 (CH_2 ; C-5'), 18.14 (CH_3 ; 4-Me), 18.45 (CH_2 ; C-1), 26.66 (CH_2 ; C-2), 28.08 (CH_2 ; C-4'), 31.61 (CH ; C-4), 41.32 (CH_2 ; C-6'), 43.06 (C; C-3), 110.16 (CH ; C-8), 111.48 (C; C-4a), 116.62 (CH ; C-5), 117.76 (CH ; C-6), 119.91 (CH ; C-7), 126.78 (C; C-4b), 131.09 (C; C-9a), 136.05 (C; C-8a), 177.34 (C; C-2') ppm. MS (EI, 70 eV): m/z (%) = 268 (29) [M^+], 157 (100), 133 (10), 121 (5), 89 (30), 45 (58). IR (ATR): 3396 (w), 3288 (m), 3057 (w), 2951 (m), 1684 (s), 1634 (vs), 1490 (s), 1455 (s), 1329 (s), 1280 (m), 1198 (s), 1136 (s), 905 (s), 728 (vs) cm^{-1} . HRMS calcd. 268.1576 (for $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}$), found 268.1569 [M^+]. $\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}$ (268.35).

Friedländer-synthesis with lactam 7b: 2-Aminobenzaldehyde (199 mg, 1.64 mmol) was added to a solution of ketone **7b** (247 mg, 1.26 mmol) in glacial acetic acid (3.2 ml) and the mixture was stirred for 16 h at 100°C. MTBE (30 ml) and 10% aqueous NaOH (15 ml) were added and the layers separated. The aqueous layer was further extracted with MTBE (2 x 20 ml) and the combined organic layers were dried over MgSO_4 . After filtration and evaporation of the solvent the residue was chromatographed on SiO_2 (toluene/acetone/ NEt_3 50 : 50 : 1) to yield acridine **12b** (160 mg, 0.57 mmol, 45%) in the first fraction (R_f = 0.28) and acridine **13b** (92 mg, 0.33 mmol, 26%) as the second fraction (R_f 0.21), both as colorless solids.

1,2,3,4-Tetrahydro-3-methyl-spiro[acridine-2,3'-piperidine]-2'-one (12b): Colorless solid, mp. 165–166°C. $^1\text{H-NMR}$ ($\text{CDCl}_3/\text{CD}_3\text{OD}$ 1 : 1, 500 MHz): δ = 1.21 (d, J = 6.9 Hz, 1H; 3-Me), 1.60–1.67 (m, 1H; 4'-H), 1.86–1.96 (m, 3H; 4'-H, 2 x 5'-H), 2.02–2.09 (m, 1H; 3-H), 2.78 (d, J = 16.3 Hz, 1H; 1-H), 3.12–3.21 (m, 2H; 2 x 4-H), 3.34–3.36 (m, 2H; 2 x 6'-H), 3.61 (d, J = 16.3 Hz, 1H; 1-H), 7.48–7.51 (m, 1H; 7-H), 7.64–7.67 (m, 1H; 6-H), 7.78 (d, J = 8.2 Hz, 1H; 8-H), 7.94 (s, 1H; 9-H), 7.95 (d, J = 8.1 Hz, 1H; 5-H) ppm. $^{13}\text{C}\{^1\text{H}\}\text{-NMR}$ ($\text{CDCl}_3/\text{CD}_3\text{OD}$ 1 : 1, 125 MHz): δ = 16.42 (CH_3 ; 3-Me),

17.96 (CH₂; C-5'), 32.81 (CH₂; C-4'), 35.93 (CH; C-3), 37.01 (CH₂; C-1), 37.81 (CH₂, C-4), 41.48 (CH₂; C-6'), 42.89 (C; C-2), 125.34 (CH; C-7), 126.31 (CH; C-5 or C-8), 126.54 (CH; C-8 or C-5), 126.98 (C; C-8a), 128.46 (CH; C-6), 128.78 (C; C-9a); 134.92 (CH; C-9); 145.32 (C; C-10a), 158.81 (C; C-4a); 175.57 (C; C-2') ppm. MS (EI, 70 eV): m/z (%) = 280 (100) [M⁺], 265 (28), 252 (38), 237 (16), 222 (23), 194 (73), 181 (33). IR (ATR): 3286 (w), 3202 (w), 3065 (w), 2941 (m), 2874 (m), 1650 (vs), 1491 (s), 1406 (s), 1319 (s), 1282 (s), 1198 (m), 989 (m), 949 (m), 910 (m), 779 (s), 749 (vs) cm⁻¹. HRMS calcd. 280.1576 (for C₁₈H₂₀N₂O), found 280.1570 [M⁺]. C₁₈H₂₀N₂O (280.36).

1,2,3,4-Tetrahydro-1-methyl-spiro[acridine-2,3'-piperidine]-2'-one (13b): Colorless solid, mp. 221–222°C. ¹H-NMR (CDCl₃/CD₃OD 1 : 1, 500 MHz): δ = 1.45 (d, J = 7.0 Hz, 3H; 1-Me), 1.72–1.77 (m, 1H; 4'-H), 1.84–1.97 (m, 4H; 3-H, 2 x 5'-H, 4'-H), 2.46 (ddd, J = 14.2 Hz, J = 10.2 Hz, J = 5.8 Hz, 1H; 3-H), 2.97 (q, J = 7.0 Hz, 1H; 1-H), 3.00–3.07 (m, 1H; 4-H), 3.22 (td, J = 17.3 Hz, J = 5.6 Hz, 1H; 4-H), 3.27–3.36 (m, 2H; 2 x 6'-H), 7.46–7.50 (m, 1H; 7-H), 7.62–7.67 (m, 1H; 6-H), 7.80 (d, J = 8.2 Hz, 1H; 8-H), 7.94 (d, J = 8.5 Hz, 1H; 5-H), 8.03 (s, 1H; 9-H) ppm. ¹³C{¹H}-NMR (CDCl₃/CD₃OD 1 : 1, 125 MHz): δ = 16.35 (CH₃; 1-Me), 17.77 (CH₂; C-5'), 28.28 (CH₂; C-4), 29.56 (CH₂; C-3), 31.06 (CH₂; C-4'), 39.17 (CH; C-1); 41.33 (CH₂; C-6'), 42.89 (C; C-2), 125.15 (CH; C-7), 126.12 (CH; C-5), 126.67 (CH; C-8), 127.01 (C; C-8a), 128.38 (CH; C-6), 134.11 (CH; C-9), 134.73 (C; C-9a), 144.84 (C; C-10a), 158.05 (C; C-4a), 175.34 (C; C-2') ppm. MS (EI, 70 eV): m/z (%) = 280 (41) [M⁺], 265 (18), 251 (7), 237 (19), 222 (30), 209 (27), 194 (61), 180 (33), 169 (100). IR (ATR): 3276 (w), 3189 (m), 3030 (m), 2925 (m), 1640 (vs), 1415 (s), 1315 (s), 1293 (s), 1108 (m), 1021 (m), 896 (m), 831 (m), 767 (s), 757 (s) cm⁻¹. HRMS calcd. 280.1576 (for C₁₈H₂₀N₂O), found 280.1584 [M⁺]. C₁₈H₂₀N₂O (280.36).