Electronic Supplementary Information

Novel oxazepinedione derived symmetric dimers: Synthesis and mesophase characterization of seven member heterocyclic compounds

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Chemical structure analysis data:

2b: Yield 77 %. IR: (KBr, νmax, cm⁻¹): 3013, 2960, 2879, 1607, 1583, 1261. ¹H NMR δ (ppm, DMSO): 8.36 (s, 2H, -N=CH), 7.92 (d, 4H, J = 8.12 Hz, Ar-H), 7.47 (d, 4H, J = 8.66 Hz), 7.34 (d, 4H, J = 8.87 Hz), 7.12 (d, 4H, J = 8.93 Hz), 4.15 (t, 4H, OCH2), 2.58 (t, 4H, Ar-CH2), 1.29-1.94 (m, 56H, CH2), 0.86 (t, 6H, CH3). ¹³C NMR δ (ppm): 164.56 (C=N), 159.83 (Ar-C=O), 116.33-140.52 (Ar-C), 61.33 (C-O-C), 36.10 (N-CH2), 23.07 (CH2), 14.72 (CH3). Elemental analysis found for C60H88N2O2 (%): C 82.89, H 10.20, N 3.22. Calc (%), C 82.89, H 10.20, N 3.22.

2c: Yield 70 %. IR: (KBr, νmax, cm⁻¹): 3009, 2959, 2883, 1600, 1581, 1257. ¹H NMR δ (ppm, DMSO): 8.45 (s, 2H, -N=CH), 7.96 (d, 4H, J = 8.19 Hz, Ar-H), 7.44 (d, 4H, J = 8.61 Hz), 7.38 (d, 4H, J = 8.80 Hz), 7.15 (d, 4H, J = 8.99 Hz), 4.18 (t, 4H, OCH2), 2.54 (t, 4H, Ar-CH2), 1.24-1.98 (m, 60H, CH2), 0.82 (t, 6H, CH3). ¹³C NMR δ (ppm): 163.05 (C=N), 160.67 (Ar-C=O), 115.03-141.83 (Ar-C), 63.50 (C-O-C), 36.77 (N-CH2), 23.42 (CH2), 14.90 (CH3). Elemental analysis found for C62H92N2O2 (%): C 82.86, H 10.46, N 3.06. Calc (%), C 82.98, H 10.33, N 3.12.

2d: Yield 69 %. IR: (KBr, νmax, cm⁻¹): 3019, 2966, 2888, 1604, 1587, 1262. ¹H NMR δ (ppm, DMSO): 8.41 (s, 2H, -N=CH), 7.98 (d, 4H, J = 8.28 Hz, Ar-H), 7.40 (d, 4H, J = 8.74 Hz), 7.37 (d, 4H, J = 8.83 Hz), 7.19 (d, 4H, J = 8.91 Hz), 4.14 (t, 4H, OCH2), 2.58 (t, 4H, Ar-CH2), 1.21-1.97 (m, 64H, CH2), 0.85 (t, 6H, CH3). ¹³C NMR δ (ppm): 164.73 (C=N), 160.86 (Ar-C=O), 116.18-140.92 (Ar-C), 64.08 (C-O-C), 36.08 (N-CH2), 22.03 (CH2), 14.40 (CH3). Elemental analysis found for C64H96N2O2 (%): C 83.21, H 10.30, N 3.19. Calc (%), C 83.06, H 10.46, N 3.03.
2e: Yield 66 %. IR: (KBr, \( \nu_{\text{max}}, \text{cm}^{-1} \)): 3011, 2962, 2880, 1603, 1585, 1253. \(^1\)H NMR \( \delta \) (ppm, DMSO): 8.49 (s, 2H, -N=CH), 7.91 (d, 4H, \( J = 8.43 \) Hz, Ar-H), 7.43 (d, 4H, \( J = 8.94 \) Hz), 7.34 (d, 4H, \( J = 8.71 \) Hz), 7.12 (d, 4H, \( J = 8.10 \) Hz), 4.17 (t, 4H, OCH\(_2\)), 2.51 (t, 4H, Ar-CH\(_2\)), 1.20-1.94 (m, 68H, CH\(_2\)), 0.88 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 164.05 (C=N), 160.38 (Ar-C-O), 116.89-139.76 (Ar-C), 62.68 (C-O-C), 37.14 (N-CH\(_2\)), 23.27 (CH\(_2\)), 14.97 (CH\(_3\)). Elemental analysis found for C\(_{66}\)H\(_{100}\)N\(_2\)O\(_2\) (%): C 83.03, H 10.69, N 2.87. Calc (%), C 83.14, H 10.57, N 2.94.

3b: Yield 59 %. IR: (KBr, \( \nu_{\text{max}}, \text{cm}^{-1} \)): 3019, 2956, 2879, 1699, 1602, 1586, 1258. \(^1\)H NMR \( \delta \) (ppm, DMSO): 9.95 (s, 2H), 7.89 (d, 4H, \( J = 8.27 \) Hz, Ar-H), 7.62 (d, 4H, \( J = 8.46 \) Hz), 7.51 (d, 4H, \( J = 8.50 \) Hz), 7.39 (d, 4H, \( J = 8.84 \) Hz), 6.22 (dd, 4H, \( J = 12.40 \) Hz & 12.78 Hz), 4.18 (t, 4H, OCH\(_2\)), 2.52 (t, 4H, Ar-CH\(_2\)), 1.21-1.96 (m, 56H, CH\(_2\)), 0.88 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 169.00, 167.86 (C=O), 160.48 (Ar-C-O), 116.16-138.58 (Ar-C), 90.08 (C-N), 63.36 (C-O-C), 37.20 (N-CH\(_2\)), 22.38 (CH\(_2\)), 14.90 (CH\(_3\)). Elemental analysis found for C\(_{68}\)H\(_{92}\)N\(_2\)O\(_8\) (%): C 76.60, H 8.89, N 2.70. Calc (%), C 76.65, H 8.70, N 2.63.

3c: Yield 49 %. IR: (KBr, \( \nu_{\text{max}}, \text{cm}^{-1} \)): 3008, 2959, 2882, 1690, 1604, 1589, 1262. \(^1\)H NMR \( \delta \) (ppm, DMSO): 9.97 (s, 2H), 7.82 (d, 4H, \( J = 8.42 \) Hz, Ar-H), 7.60 (d, 4H, \( J = 8.77 \) Hz), 7.58 (d, 4H, \( J = 8.90 \) Hz), 7.31 (d, 4H, \( J = 8.52 \) Hz), 6.55 (dd, 4H, \( J = 12.85 \) Hz & 12.09 Hz), 4.10 (t, 4H, OCH\(_2\)), 2.55 (t, 4H, Ar-CH\(_2\)), 1.23-1.96 (m, 60H, CH\(_2\)), 0.83 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 169.27, 167.86 (C=O), 159.63 (Ar-C-O), 115.71-139.08 (Ar-C), 90.59 (C-N), 62.20 (C-O-C), 37.58 (N-CH\(_2\)), 23.00 (CH\(_2\)), 14.16 (CH\(_3\)). Elemental analysis found for C\(_{70}\)H\(_{96}\)N\(_2\)O\(_8\) (%): C 76.60, H 8.89, N 2.70. Calc (%), C 76.65, H 8.70, N 2.63.

3d: Yield 56 %. IR: (KBr, \( \nu_{\text{max}}, \text{cm}^{-1} \)): 3014, 2962, 2880, 1694, 1603, 1588, 1258. \(^1\)H NMR \( \delta \) (ppm, DMSO): 9.92 (s, 2H), 7.81 (d, 4H, \( J = 8.53 \) Hz, Ar-H), 7.65 (d, 4H, \( J = 8.42 \) Hz), 7.55 (d, 4H, \( J = 8.05 \) Hz), 7.33 (d, 4H, \( J = 8.94 \) Hz), 6.56 (dd, 4H, \( J = 12.33 \) Hz & 12.41 Hz), 4.14 (t, 4H, OCH\(_2\)), 2.58 (t, 4H, Ar-CH\(_2\)), 1.21-1.98 (m, 64H, CH\(_2\)), 0.87 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 168.58, 166.06 (C=O), 159.63 (Ar-C-O), 115.25-140.35 (Ar-C), 90.64 (C-N), 63.83 (C-O-C), 38.74 (N-CH\(_2\)), 22.68 (CH\(_2\)), 14.70 (CH\(_3\)). Elemental analysis found for C\(_{72}\)H\(_{100}\)N\(_2\)O\(_8\) (%): C 77.31, H 8.83, N 2.57. Calc (%), C 77.10, H 8.99, N 2.50.
3e: Yield 49 %. IR: (KBr, \( \nu_{\text{max}} \), cm\(^{-1}\)): 3010, 2958, 2874, 1690, 1600, 1582, 1251. \(^1\)HNMR \( \delta \) (ppm, DMSO): 9.90 (s, 2H), 7.84 (d, 4H, \( J = 8.15 \) Hz, Ar-H), 7.69 (d, 4H, \( J = 8.65 \) Hz), 7.50 (d, 4H, \( J = 8.36 \) Hz), 7.35 (d, 4H, \( J = 8.82 \) Hz), 6.57 (dd, 4H, \( J = 12.54 \) Hz & 12.24 Hz), 4.18 (t, 4H, OCH\(_2\)), 2.53 (t, 4H, Ar-CH\(_2\)), 1.22-1.95 (m, 68H, CH\(_2\)), 0.89 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 169.06, 167.64 (C=O), 160.47 (Ar-C-O), 115.42-140.04 (Ar-C), 90.82 (C-N), 64.20 (C-O-C), 37.08 (N-CH\(_2\)), 22.36 (CH\(_2\)), 14.86 (CH\(_3\)). Elemental analysis found for C\(_{74}\)H\(_{104}\)N\(_2\)O\(_8\): C 77.50, H 9.01, N 2.52. Calc (%), C 77.31, H 9.12, N 2.44.

4a: Yield 57 %. IR: (KBr, \( \nu_{\text{max}} \), cm\(^{-1}\)): 3014, 2953, 2881, 1697, 1601, 1589, 1251. \(^1\)HNMR \( \delta \) (ppm, DMSO): 9.87 (s, 2H), 7.88 (d, 4H, \( J = 8.92 \) Hz, Ar-H), 7.56 (d, 4H, \( J = 8.37 \) Hz), 7.25 (d, 4H, \( J = 8.61 \) Hz), 7.07 (d, 4H, \( J = 8.49 \) Hz), 2.58 (t, 4H, \( J = 7.66 \) Hz), 2.43 (t, 4H, \( J = 7.24 \) Hz), 4.24 (t, 4H, OCH\(_2\)), 2.27 (t, 4H, Ar-CH\(_2\)), 1.13-1.85 (m, 52H, CH\(_2\)), 0.85 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 168.40, 166.04 (C=O), 160.27 (Ar-C-O), 115.11-139.37 (Ar-C), 90.20 (C-N), 60.19 (C-O-C), 38.35 (N-CH\(_2\)), 21.14 (CH\(_2\)), 15.30 (CH\(_3\)). Elemental analysis found for C\(_{66}H_{92}N_2O_8\): C 76.24, H 8.77, N 2.60. Calc (%), C 76.12, H 8.90, N 2.69.

4c: Yield 45 %. IR: (KBr, \( \nu_{\text{max}} \), cm\(^{-1}\)): 3018, 2964, 2883, 1691, 1604, 1585, 1260. \(^1\)HNMR \( \delta \) (ppm, DMSO): 9.88 (s, 2H), 7.84 (d, 4H, \( J = 8.92 \) Hz, Ar-H), 7.51 (d, 4H, \( J = 8.82 \) Hz), 7.28 (d, 4H, \( J = 8.53 \) Hz), 7.05 (d, 4H, \( J = 8.85 \) Hz), 2.46 (t, 4H, \( J = 7.66 \) Hz), 2.59 (t, 4H, \( J = 7.90 \) Hz), 4.22 (t, 4H, OCH\(_2\)), 2.22 (t, 4H, Ar-CH\(_2\)), 1.11-1.88 (m, 60H, CH\(_2\)), 0.86 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 168.29, 166.02 (C=O), 160.20 (Ar-C-O), 115.48-138.00 (Ar-C), 89.68 (C-N), 62.32 (C-O-C), 37.63 (N-CH\(_2\)), 22.40 (CH\(_2\)), 15.57 (CH\(_3\)). Elemental analysis found for C\(_{70}H_{100}N_2O_8\): C 76.78, H 8.77, N 2.63. Calc (%), C 76.60, H 8.90, N 2.69.

4d: Yield 58 %. IR: (KBr, \( \nu_{\text{max}} \), cm\(^{-1}\)): 3014, 2960, 2877, 1695, 1607, 1590, 1263. \(^1\)HNMR \( \delta \) (ppm, DMSO): 9.82 (s, 2H), 7.82 (d, 4H, \( J = 8.19 \) Hz), 7.51 (d, 4H, \( J = 8.82 \) Hz), 7.28 (d, 4H, \( J = 8.53 \) Hz), 7.05 (d, 4H, \( J = 8.85 \) Hz), 2.46 (t, 4H, \( J = 7.90 \) Hz), 2.59 (t, 4H, \( J = 7.90 \) Hz), 4.23 (t, 4H, OCH\(_2\)), 2.28 (t, 4H, Ar-CH\(_2\)), 1.14-1.86 (m, 64H, CH\(_2\)), 0.86 (t, 6H, CH\(_3\)). \(^{13}\)C NMR \( \delta \) (ppm): 168.04, 166.63 (C=O), 160.19 (Ar-C-O), 115.04-138.30 (Ar-C), 90.38 (C-N), 62.07 (C-O-C), 37.88 (N-CH\(_2\)), 21.68.
Elemental analysis found for C$_{72}$H$_{104}$N$_2$O$_8$ (%): C 76.98, H 9.46, N 2.56. Calc (%), C 76.83, H 9.31, N 2.49.

4e: Yield 44 %. IR: (KBr, $\nu_{\text{max}}$, cm$^{-1}$): 3019, 2961, 2883, 1698, 1602, 1585, 1260. $^1$H NMR $\delta$ (ppm, DMSO): 9.81 (s, 2H), 7.86 (d, 4H, $J = 8.97$ Hz), 7.52 (d, 4H, $J = 8.60$ Hz), 7.28 (d, 4H, $J = 8.57$ Hz), 7.01 (d, 4H, $J = 8.38$ Hz), 2.40 (t, 4H, $J = 7.35$ Hz), 2.57 (t, 4H, $J = 7.84$ Hz), 4.23 (t, 4H, OCH$_2$), 2.22 (t, 4H, Ar-CH$_2$), 1.12-1.88 (m, 68H, CH$_2$), 0.84 (t, 6H, CH$_3$). $^{13}$C NMR $\delta$ (ppm): 169.36, 167.20 (C=O), 160.48 (Ar-C=O), 116.70-139.18 (Ar-C), 90.87 (C-N), 62.00 (C-O-C), 38.60 (N-CH$_2$), 14.33 (CH$_3$).

Calc (%), C 77.04, H 9.44, N 2.43.

5a: Yield 41 %. IR: (KBr, $\nu_{\text{max}}$, cm$^{-1}$): 3012, 2957, 2875, 1696, 1603, 1581, 1255. $^1$H NMR $\delta$ (ppm, DMSO): 9.89 (s, 2H), 7.93 (d, 4H, $J = 8.73$ Hz), 7.64 (d, 4H, $J = 8.52$ Hz), 7.64 (t, 2H, $J = 7.80$ Hz), 7.74 (t, 2H, $J = 8.91$ Hz), 7.34 (d, 2H, $J = 8.09$ Hz), 4.28 (t, 4H, OCH$_2$), 2.43 (t, 4H, Ar-CH$_2$), 1.10-1.93 (m, 52H, CH$_2$), 0.90 (t, 6H, CH$_3$). $^{13}$C NMR $\delta$ (ppm): 168.18, 166.46 (C=O), 159.20 (Ar-C=O), 115.11-140.30 (Ar-C), 90.36 (C-N), 62.78 (C-O-C), 36.98 (N-CH$_2$), 23.67 (CH$_2$), 14.88 (CH$_3$). Elemental analysis found for C$_{74}$H$_{92}$N$_2$O$_8$ (%): C 78.30, H 8.28, N 2.35. Calc (%), C 78.13, H 8.15, N 2.46.

5b: Yield 49 %. IR: (KBr, $\nu_{\text{max}}$, cm$^{-1}$): 3018, 2951, 2878, 1694, 1601, 1581, 1261. $^1$H NMR $\delta$ (ppm, DMSO): 9.89 (s, 2H), 7.98 (d, 4H, $J = 8.37$ Hz), 7.88 (d, 2H, $J = 8.52$ Hz), 7.64 (t, 2H, $J = 7.80$ Hz), 7.78 (t, 2H, $J = 8.91$ Hz), 7.34 (d, 2H, $J = 8.09$ Hz), 4.28 (t, 4H, OCH$_2$), 2.43 (t, 4H, Ar-CH$_2$), 1.13-1.97 (m, 56H, CH$_2$), 0.88 (t, 6H, CH$_3$). $^{13}$C NMR $\delta$ (ppm): 168.77, 166.94 (C=O), 159.20 (Ar-C=O), 115.11-140.30 (Ar-C), 90.36 (C-N), 63.74 (C-O-C), 38.06 (N-CH$_2$), 22.44 (CH$_2$), 14.09 (CH$_3$). Elemental analysis found for C$_{76}$H$_{96}$N$_2$O$_8$ (%): C 78.20, H 8.46, N 2.29. Calc (%), C 78.31, H 8.30, N 2.40.

5d: Yield 42 %. IR: (KBr, $\nu_{\text{max}}$, cm$^{-1}$): 3014, 2960, 2879, 1697, 1600, 1581, 1256. $^1$H NMR $\delta$ (ppm, DMSO): 9.89 (s, 2H), 7.91 (d, 4H, $J = 8.80$ Hz), 7.62 (d, 4H, $J = 8.78$ (CH$_2$), 14.22 (CH$_3$).
Hz), 7.40 (d, 4H, J = 8.94 Hz), 7.10 (t, 4H, J = 8.64 Hz), 7.82 (d, 2H, J = 8.57 Hz), 7.61 (t, 2H, J = 7.42 Hz), 7.72 (t, 2H, J = 8.37 Hz), 7.31 (d, 2H, J = 8.01 Hz), 4.26 (t, 4H, OCH₂), 2.42 (t, 4H, Ar-CH₂), 1.11-1.96 (m, 64H, CH₂), 0.89 (t, 6H, CH₃). ¹³C NMR δ (ppm): 168.87, 166.01 (C=O), 159.66 (Ar-C-O), 116.04-140.68 (Ar-C), 90.86 (C-N), 60.72 (C-O-C), 38.15 (N-CH₂), 22.07 (CH₂), 14.33 (CH₃). Elemental analysis found for C₈₀H₁₀₄N₂O₈ (%): C 78.42, H 8.71, N 2.21. Calc (%), C 78.65, H 8.58, N 2.29.

5e: Yield 46 %. IR: (KBr, v_max, cm⁻¹): 3010, 2958, 2872, 1692, 1603, 1585, 1251. ¹H NMR δ (ppm, DMSO): 9.87 (s, 2H), 7.93 (d, 4H, J = 8.74 Hz), 7.65 (d, 4H, J = 8.41 Hz), 7.42 (d, 4H, J = 8.79 Hz), 7.12 (t, 4H, J = 8.35 Hz), 7.81 (d, 2H, J = 8.86 Hz), 7.60 (t, 2H, J = 7.67 Hz), 7.71 (t, 2H, J = 8.92 Hz), 7.33 (d, 2H, J = 8.18 Hz), 4.28 (t, 4H, OCH₂), 2.44 (t, 4H, Ar-CH₂), 1.13-1.98 (m, 68H, CH₂), 0.84 (t, 6H, CH₃). ¹³C NMR δ (ppm): 169.19, 167.47 (C=O), 160.58 (Ar-C-O), 115.65-140.07 (Ar-C), 90.48 (C-N), 61.24 (C-O-C), 37.09 (N-CH₂), 22.74 (CH₂), 14.68 (CH₃). Elemental analysis found for C₈₂H₁₀₈N₂O₈ (%): C 78.81, H 8.71, N 2.24. Calc (%), C 78.81, H 8.71, N 2.24.

Figure 4. Molecular models of compound 2c, 3a, 5c, and 5e using HyperChem
Program
HyperChem illustration of 2c
HyperChem illustration of 3a
HyperChem illustration of 5c
HyperChem illustration of 5e