

## Supporting Information

### Development of furan-2,5-dicarboxylic acid (FDCA)-based organogelators

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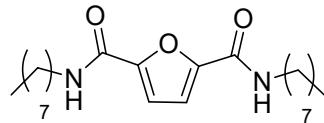
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## Experimental data.

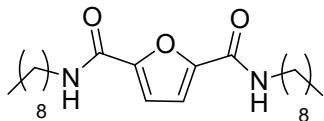
### Diamides.

#### *N<sup>2</sup>, N<sup>5</sup>-dioctylfuran-2,5-dicarboxamide (2a)*



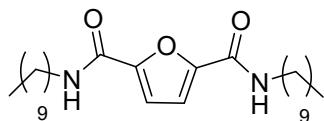
**61%**; white powder; **T<sub>f</sub>**: 75.2°C; **<sup>1</sup>H NMR** (300 MHz, DMSO-*d*<sub>6</sub>), δ (ppm): 8.42 (t, *J* = 5.9 Hz, 2H, NH), 7.09 (s, 2H, CH furan), 3.25 (q, *J* = 6.7 Hz, 4H, CH<sub>2</sub>-NH), 1.50 (m, *J* = 7.0 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.26 (m, 20H, (CH<sub>2</sub>)<sub>5</sub>), 0.84 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-*d*<sub>6</sub>, 340K), δ (ppm): 157.07 (C=O), 148.16, 114.17, 38.47, 38.35, 31.22, 29.26, 28.70, 28.61, 26.43, 22.06, 13.89.

#### *N<sup>2</sup>, N<sup>5</sup>-dinonylfuran-2,5-dicarboxamide (2b)*



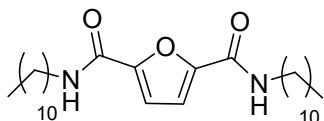
**86%**; white powder; **T<sub>f</sub>**: 77.3-83.7°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.21 (t, *J* = 5.9 Hz, 2H, NH), 7.01 (d, *J* = 4.1 Hz, 2H, CH furan), 3.29 (q, *J* = 6.6 Hz, 4H, CH<sub>2</sub>-NH), 1.55 (p, *J* = 6.7 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.26 (m, 24H, (CH<sub>2</sub>)<sub>6</sub>), 0.85 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 157.04 (C=O), 148.08, 113.71, 38.45, 31.22, 29.26, 28.89, 28.74, 28.61, 26.42, 22.02, 13.58.

#### *N<sup>2</sup>, N<sup>5</sup>-didecylfuran-2,5-dicarboxamide (2c)*



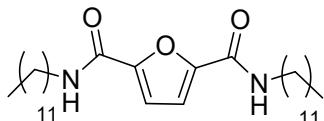
**43%**; white powder; **T<sub>f</sub>**: 88.8°C; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.41 (t, *J* = 5.9 Hz, 2H, NH), 7.09 (s, 2H, CH furan), 3.30-3.22 (q, 4H, CH<sub>2</sub>-NH), 1.50 (m, *J* = 7.0 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.26 (m, 28H, (CH<sub>2</sub>)<sub>7</sub>), 0.84 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (151 MHz, DMSO-d<sub>6</sub>, 340K), δ (ppm): 156.89 (C=O), 156.82 (C=O), 148.09, 113.72, 38.31, 38.19, 30.92, 28.90, 28.88, 28.58, 28.57, 28.38, 28.28, 26.11, 21.67, 13.47.

#### *N<sup>2</sup>, N<sup>5</sup>-diundecylfuran-2,5-dicarboxamide (2d)*



**73%**; white powder; **T<sub>f</sub>**: 91.7°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.13 (q, *J* = 5.6 Hz, 2H, NH), 7.00 (s, 2H, CH furan), 3.31 (q, *J* = 6.7 Hz, 4H, CH<sub>2</sub>-NH), 1.55 (q, *J* = 7.1 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.27 (m, 32H, (CH<sub>2</sub>)<sub>8</sub>), 0.86 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 157.02 (C=O), 148.08, 113.74, 38.43, 31.22, 29.24, 28.93, 28.71, 28.64, 26.40, 22.01, 13.62.

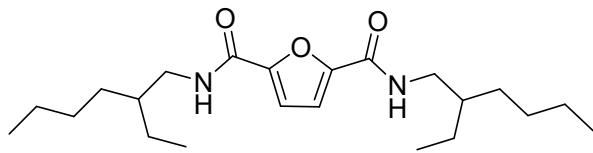
#### *N<sup>2</sup>, N<sup>5</sup>-didodecylfuran-2,5-dicarboxamide (2e)*



**25%**; white powder; **T<sub>f</sub>**: 96.3°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.15 (t, *J* = 5.9 Hz, 2H, NH), 7.01 (s, 2H, CH furan), 3.30 (q, *J* = 6.8 Hz, 4H, CH<sub>2</sub>-NH), 1.56 (m, *J* = 7.2 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.46-1.11 (m, 36H, (CH<sub>2</sub>)<sub>9</sub>), 0.86 (t, *J* = 6.7 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (151 MHz, DMSO-d<sub>6</sub>, 340K),

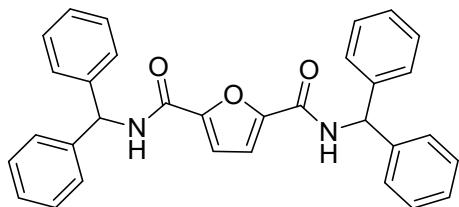
$\delta$  (ppm): 156.85 (**C=O**), 148.07, 113.68, 38.28, 30.90, 28.88, 28.63, 28.60, 28.58, 28.56, 28.35, 28.28, 26.09, 21.66, 13.46.

*N<sup>2</sup>, N<sup>5</sup>-bis(2-ethylhexyl)furan-2,5-dicarboxamide (2f)*



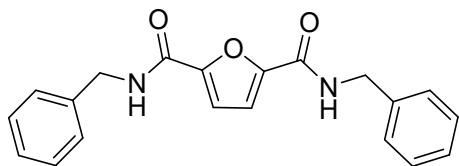
**86%**; white powder; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>),  $\delta$  (ppm): 8.35 (t,  $J$  = 6.0 Hz, 2H, NH), 7.11 (s, 2H, CH furan), 3.19 (t,  $J$  = 6.4 Hz, 4H, CH<sub>2</sub>-NH), 1.52 (p,  $J$  = 6.0 Hz, 2H, CH), 1.27 (m, 16H, CH<sub>2</sub>), 0.86 (m, 12H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 157.22, 148.20, 114.18, 41.81, 30.31, 28.28, 23.61, 22.43, 13.85, 10.60.

*N<sup>2</sup>, N<sup>5</sup>-dibenzhydrylfuran-2,5-dicarboxamide (2g)*



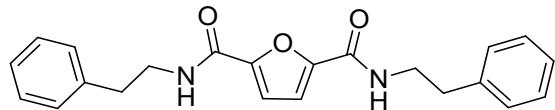
**30%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 9.26 (d,  $J$  = 8.7 Hz, 2H, NH), 7.33 (m, 22H, CH furan + Ar-H), 6.38 (d,  $J$  = 8.6 Hz, 2H, CH); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 156.78 (**C=O**), 148.12 (**C-C=O**), 141.52 (**C(i) Ph**), 128.40 (**C(m) Ph**), 127.66 (**C(o) Ph**), 127.21 (**C(p) Ph**), 115.11 (CH furan), 55.71 (NH-CH).

*N<sup>2</sup>, N<sup>5</sup>-dibenzylfuran-2,5-dicarboxamide (2h)*



**66%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 9.00 (t,  $J$  = 6.2 Hz, 2H, NH), 7.39-7.23 (m, 10H, Ar-H), 7.20 (s, 2H, CH furan), 4.51 (d,  $J$  = 6.1 Hz, 2H, CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 157.71 (**C=O**), 148.54 (**O-C-CO furan**), 139.46, 128.89, 127.71, 127.47, 115.24 (**CH furan**), 42.34 (**CH<sub>2</sub>-NH**).

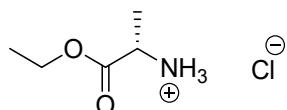
*N<sup>2</sup>, N<sup>5</sup>-diphenethylfuran-2,5-dicarboxamide (2i)*



**77%**; yellow oil; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.59 (t, *J* = 5.9 Hz, 2H, NH), 7.37-7.16 (m, 10H, Ar-H), 7.12 (s, 2H, CH furan), 3.50 (dt, *J* = 8.1, 6.2 Hz, 4H, CH<sub>2</sub>-NH), 2.85 (t, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 157.13 (C=O), 148.12, 139.15, 128.61, 128.37, 126.17, 114.29, 35.22.

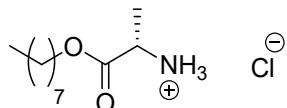
### Ester ammonium salts.

#### *ethyl L-alaninate ammonium chloride salt (3a)*



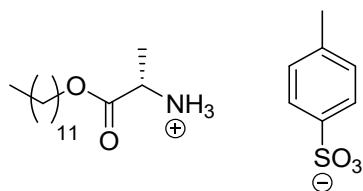
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.74 (s, 3H, NH<sub>3</sub>), 4.17 (m, 2H, CH<sub>2</sub>-O), 3.98 (q, *J* = 7.1 Hz, 1H, CH), 1.42 (d, *J* = 7.1 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.21 (t, *J* = 7.1 Hz, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.88 (C=O), 61.60 (CH<sub>2</sub>-O), 47.80 (CH), 15.64, 13.92.

#### *octyl L-alaninate ammonium chloride salt (3c)*



**37%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.64 (s, 3H, NH<sub>3</sub>), 4.21-3.97 (m, 3H, CH<sub>2</sub>-O + CH-NH<sub>3</sub>), 1.60 (p, *J* = 6.6 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.42 (d, *J* = 7.1 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.25 (m, 10H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>5</sub>), 0.84 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 170.00 (C=O), 65.51 (CH<sub>2</sub>-O), 47.83 (CH), 31.20, 28.57, 27.95, 25.17, 22.08, 15.71, 13.95.

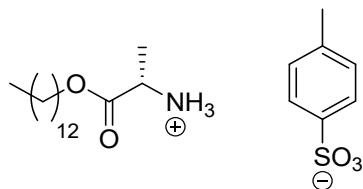
#### *dodecyl L-alaninate ammonium tosylate salt (3d)*



**90%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.15 (s, 3H, NH<sub>3</sub>), 7.49 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.9 Hz, 2H, Ar-H), 4.21-4.05 (m, 3H, CH<sub>2</sub>-O + CH-NH<sub>3</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.59 (m, *J* = 6.7 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.38 (d, *J* = 7.2 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.24 (m, 18H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.85

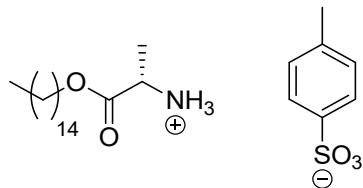
(t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.92 (C=O), 145.23, 138.00, 128.18, 125.56, 65.69 (CH<sub>2</sub>-O), 47.86 (CH), 31.37, 29.11, 29.08, 29.04, 28.99, 28.78, 28.68, 27.99, 25.23, 22.16, 20.82, 15.62, 15.59, 13.97.

*tridecyl L-alaninate ammonium tosylate salt (3e)*



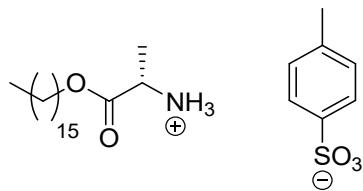
97%; white powder; <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.32 (s, 3H, NH<sub>3</sub>), 7.51 (d, *J*= 8.1 Hz, 2H, Ar-H), 7.13 (d, *J*= 8.0 Hz, 2H, Ar-H), 4.21-4.05 (m, 3H, CH<sub>2</sub>-O + CH-NH<sub>3</sub>), 2.30 (s, 3H, Ar-CH<sub>3</sub>), 1.60 (t, *J*= 7.0 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.40 (d, *J*= 7.2 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.25 (m, 20H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>10</sub>), 0.86 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.94 (C=O), 145.33, 137.79, 128.06, 125.48, 65.57 (CH<sub>2</sub>-O), 47.93 (CH), 31.29, 29.06, 29.03, 29.01, 28.97, 28.92, 28.71, 28.61, 27.92, 25.15, 22.08, 20.74, 15.67, 13.90.

*pentadecyl L-alaninate ammonium tosylate salt (3f)*



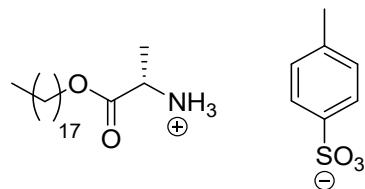
98%; white powder; <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.33 (s, 3H, NH<sub>3</sub>), 7.52 (d, *J*= 7.9 Hz, 2H, Ar-H), 7.13 (d, *J*= 7.8 Hz, 2H, Ar-H), 4.21-4.07 (m, 3H, CH<sub>2</sub>-O + CH-NH<sub>3</sub>), 2.30 (s, 3H, Ar-CH<sub>3</sub>), 1.59 (t, *J*= 6.7 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.40 (d, *J*= 7.2 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.25 (m, 24H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>12</sub>), 0.86 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); <sup>13</sup>C NMR (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.94 (C=O), 145.26, 137.83, 128.07, 125.49, 65.56, 47.94, 31.32, 29.08, 29.06, 29.04, 29.01, 28.95, 28.74, 28.65, 27.94, 25.18, 22.10, 20.75, 15.67, 13.89.

*hexadecyl L-alaninate ammonium tosylate salt (3g)*



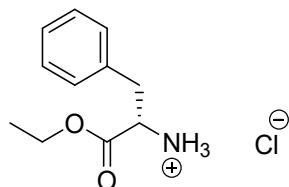
**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.31 (s, 3H, NH<sub>3</sub>), 7.50 (d, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H, Ar-H), 4.24-4.02 (m, 3H, CH<sub>2</sub>-O + CH-NH<sub>3</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.59 (t, *J* = 7.0 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.39 (d, *J* = 7.2 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.24 (m, 26H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>13</sub>), 0.86 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.93 (C=O), 145.35, 137.74, 128.03, 125.48, 65.55, 47.92, 31.31, 29.08, 29.04, 29.01, 28.95, 28.73, 28.64, 27.94, 25.17, 22.09, 20.73, 15.65, 13.86.

*octadecyl L-alaninate ammonium tosylate salt (3h)*



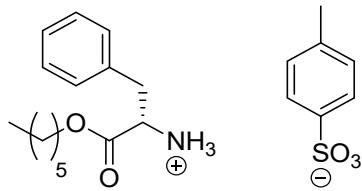
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.32 (s, 3H, NH<sub>3</sub>), 7.49 (d, *J* = 7.8 Hz, 2H, Ar-H), 7.11 (d, *J* = 7.8 Hz, 2H, Ar-H), 4.20-4.05 (m, 3H, CH<sub>2</sub>-O + CH-NH<sub>3</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.59 (m, *J* = 6.7 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.38 (d, *J* = 7.2 Hz, 3H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.23 (m, 30H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>15</sub>), 0.85 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>). **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.72 (C=O), 144.18, 138.23, 127.86, 125.29, 65.57, 48.06, 31.27, 29.03, 28.99, 28.95, 28.87, 28.69, 28.57, 27.82, 25.11, 22.05, 20.48, 15.43, 13.48.

*ethyl L-phenylalaninate ammonium chloride salt (4a)*



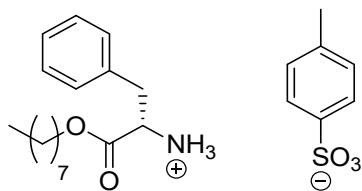
**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.84 (s, 3H, NH<sub>3</sub>), 7.40-7.18 (m, 5H, Ar-H), 4.18 (dd, *J* = 8.1, 5.4 Hz, 1H, NH<sub>3</sub>-CH), 4.08 (qd, *J* = 7.1, 2.1 Hz, 2H, CH<sub>2</sub>-O), 3.33-3.01 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 1.08 (t, *J* = 7.1 Hz, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 168.84 (C=O), 134.61 (C(i) Ph), 129.42 (CH(m) Ph), 128.47 (CH(o) Ph), 127.15 (CH(p) Ph), 61.45 (CH<sub>2</sub>-O), 53.23 (CH), 35.91 (CH<sub>2</sub>-Ph), 13.72 (CH<sub>3</sub>).

*hexyl L-phenylalaninate ammonium tosylate salt (4b)*



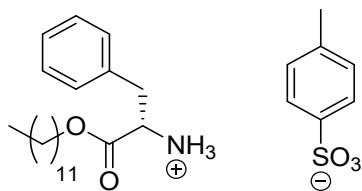
**90%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.45 (s, 3H, NH<sub>3</sub>), 7.51 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.39-7.18 (m, 5H, Ar-H), 7.13 (d, *J* = 7.9 Hz, 2H, Ar-H), 4.28 (t, *J* = 6.9 Hz, 1H, NH<sub>3</sub>-CH), 4.02 (t, *J* = 6.5 Hz, 2H, CH<sub>2</sub>-O), 3.24-2.94 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.42 (p, *J* = 6.7 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.31-1.06 (m, 6H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>3</sub>), 0.85 (t, *J* = 6.9 Hz, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.05 (C=O), 145.37, 137.81, 134.61, 129.29, 128.53, 128.09, 127.23, 125.49, 65.56 (CH<sub>2</sub>-O), 53.24 (CH), 36.15 (CH<sub>2</sub>-Ph), 30.77, 27.72, 24.75, 21.90, 20.76, 13.84.

*octyl L-phenylalaninate ammonium tosylate salt (4c)*



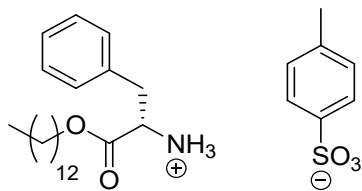
**92%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.44 (s, 3H, NH<sub>3</sub>), 7.51 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.39-7.18 (m, 5H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H, Ar-H), 4.28 (dd, *J* = 8.0, 5.8 Hz, 1H, NH<sub>3</sub>-CH), 4.02 (t, *J* = 6.5 Hz, 2H, CH<sub>2</sub>-O), 3.26-2.91 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.42 (p, *J* = 6.8 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.18 (m, 10H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>5</sub>), 0.86 (t, *J* = 6.6 Hz, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.05 (C=O), 145.43, 137.75, 134.60, 129.29, 128.52, 128.06, 127.21, 125.48, 65.56 (CH<sub>2</sub>-O), 53.22 (CH), 36.15 (CH<sub>2</sub>-Ph), 31.17, 28.52, 28.49, 27.75, 25.09, 22.05, 20.75, 13.91.

*dodecyl L-phenylalaninate ammonium tosylate salt (4d)*



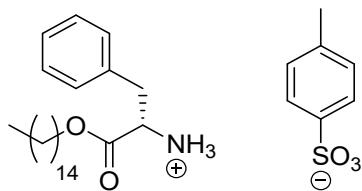
**96%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.49 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 8.2 Hz, 2H, Ar-H), 7.37-7.19 (m, 5H, Ar-H), 7.12 (d, *J* = 7.9 Hz, 2H, Ar-H), 4.29 (m, *J* = 7.9, 5.9 Hz, 1H, NH<sub>3</sub>-CH), 4.03 (t, *J* = 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.19-2.98 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.42 (m, *J* = 6.8 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.22 (m, 18H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.85 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.01 (C=O), 145.23, 137.94, 134.65, 129.32, 128.53, 128.13, 127.21, 125.53, 65.58, 53.19, 36.11, 31.34, 29.08, 29.05, 29.00, 28.90, 28.75, 28.62, 27.79, 25.14, 22.13, 20.78, 13.95.

*tridecyl L-phenylalaninate ammonium tosylate salt (4e)*



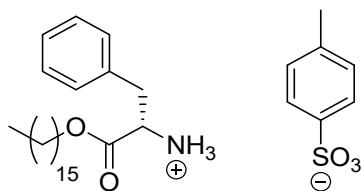
**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.46 (s, 3H, NH<sub>3</sub>), 7.52 (d, 2H, Ar-H), 7.42-7.18 (m, 5H, Ar-H), 7.13 (d, J = 7.9 Hz, 2H, Ar-H), 4.28 (m, J = 7.0 Hz, 1H, NH<sub>3</sub>-CH), 4.02 (t, J = 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.26-2.94 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.30 (s, 3H, Ar-CH<sub>3</sub>), 1.43 (m, J = 6.4 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 20H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>10</sub>), 0.86 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.03 (C=O), 145.36, 137.78, 134.62, 129.29, 128.49, 128.06, 127.17, 125.49, 65.54, 53.24, 36.15, 31.29, 29.05, 29.03, 29.01, 28.96, 28.86, 28.71, 28.58, 27.76, 25.10, 22.08, 20.75, 13.90.

*pentadecyl L-phenylalaninate ammonium tosylate salt (4f)*



**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.46 (s, 3H, NH<sub>3</sub>), 7.53 (d, J = 7.9 Hz, 2H Ar-H), 7.38 – 7.18 (m, 5H, Ar-H), 7.14 (d, J = 7.8 Hz, 2H Ar-H), 4.28 (m, 1H, NH<sub>3</sub>-CH), 4.02 (t, J = 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.26 – 2.94 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.30 (s, 3H, Ar-CH<sub>3</sub>), 1.42 (m, J = 6.6 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 24H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>12</sub>), 0.86 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.04 (C=O), 145.22, 137.90, 134.65, 129.30, 128.49, 128.11, 127.17, 125.51, 65.53, 53.26, 36.16, 31.31, 29.07, 29.03, 29.00, 28.89, 28.73, 28.62, 27.78, 25.13, 22.10, 20.76, 13.91.

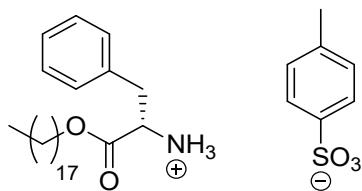
*hexadecyl L-phenylalaninate ammonium tosylate salt (4g)*



**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.45 (s, 3H, NH<sub>3</sub>), 7.52 (d, 2H Ar-H), 7.38-7.18 (m, 5H, Ar-H), 7.12 (d, J = 7.8 Hz, 2H Ar-H), 4.27 (m, 1H, NH<sub>3</sub>-CH), 4.01 (m, 2H, CH<sub>2</sub>-O), 3.26-2.92 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.40 (d, J = 6.9 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 26H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>13</sub>), 0.84 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.03 (C=O), 145.30,

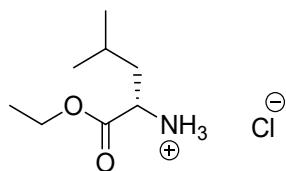
137.81, 134.64, 129.29, 128.47, 128.07, 127.15, 125.50, 65.51, 53.25, 36.15, 31.31, 29.07, 29.03, 29.00, 28.89, 28.73, 28.62, 27.77, 25.12, 22.09, 20.74, 13.88.

*octadecyl L-phenylalaninate ammonium tosylate salt (4h)*



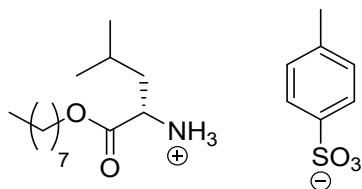
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.49 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 8.2 Hz, 2H, Ar-H), 7.37-7.19 (m, 5H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H, Ar-H), 4.29 (t, *J* = 7.0 Hz, 1H, NH<sub>3</sub>-CH), 4.03 (t, *J* = 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.19-2.98 (m, 2H, NH<sub>3</sub>-CH-CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.42 (m, *J* = 6.8 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.22 (m, 30H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>15</sub>), 0.85 (t, 3H, CH<sub>3</sub>-CH<sub>2</sub>). **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 168.77, (C=O), 144.31, 138.16, 134.25, 129.01, 128.18, 127.85, 126.91, 125.31, 65.53, 53.39, 36.01, 31.26, 29.01, 28.97, 28.93, 28.82, 28.67, 28.54, 27.67, 25.41, 25.06, 22.03, 13.50.

*ethyl L-leucinate ammonium chloride salt (5a)*



**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.75 (s, 3H, NH<sub>3</sub>), 4.18 (m, *J* = 7.1, 2.7 Hz, 2H, CH<sub>2</sub>-O), 3.84 (q, *J* = 6.9 Hz, 1H, NH<sub>3</sub>-CH), 1.87-1.56 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.22 (t, *J* = 7.1 Hz, 3H, CH<sub>3</sub>-CH<sub>2</sub>), 0.88 (d, *J* = 6.2 Hz, 6H, (CH<sub>3</sub>)<sub>2</sub>-CH); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.73 (C=O), 61.58 (CH<sub>2</sub>-O), 50.48 (CH-NH), 39.11 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 23.73, 22.19, 21.92, 13.90.

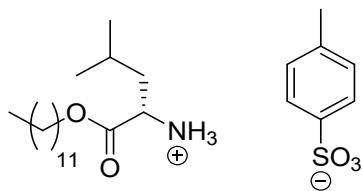
*octyl L-leucinate ammonium tosylate salt (5c)*



**40%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.33 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 8.0 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H Ar-H), 4.15 (m, 2H, CH<sub>2</sub>-O), 3.96 (q, *J* = 6.2 Hz, 1H, NH<sub>3</sub>-CH), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.83-1.50 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.26 (m, 10H, (CH<sub>2</sub>)<sub>5</sub>), 0.88 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.89 (C=O), 145.33 (**C(i)** Tosyl), 137.81 (**C(p)** Tosyl),

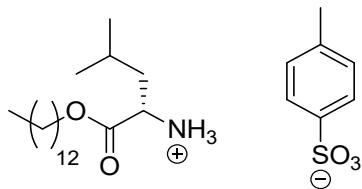
128.07 (**C(m)** Tosyl), 125.48 (**C(o)** Tosyl), 65.60 (CH<sub>2</sub>-O), 50.63 (CH-NH), 39.21 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 31.15, 28.54, 28.48, 27.86, 25.18, 23.79, 22.10, 22.05, 21.87, 20.75, 13.90.

*dodecyl L-leucinate ammonium tosylate salt (5d)*



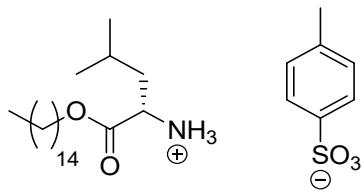
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.34 (s, 3H, NH<sub>3</sub>), 7.51 (d, *J* = 7.8 Hz, 2H, Ar-H), 7.13 (d, *J* = 7.8 Hz, 2H Ar-H), 4.14 (m, *J* = 10.8, 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.97 (q, *J* = 6.0 Hz, 1H, NH<sub>3</sub>-CH), 2.30 (s, 3H, Ar-CH<sub>3</sub>), 1.82-1.51 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 18H, (CH<sub>2</sub>)<sub>9</sub>), 0.88 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.88 (C=O), 145.34 (**C(i)** Tosyl), 137.78 (**C(p)** Tosyl), 128.05 (**C(m)** Tosyl), 125.48 (**C(o)** Tosyl), 65.59 (CH<sub>2</sub>-O), 50.62 (CH-NH), 39.21 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 31.29, 29.03, 29.00, 28.92, 28.90, 28.69, 28.54, 27.87, 25.19, 23.78, 22.10, 22.08, 21.86, 20.75, 13.90.

*tridecyl L-leucinate ammonium tosylate salt (5e)*



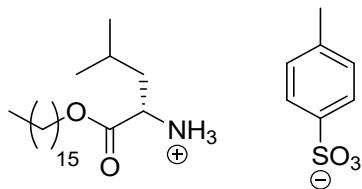
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.33 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 7.9 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H Ar-H), 4.13 (m, *J* = 10.8, 6.5 Hz, 2H, CH<sub>2</sub>-O), 3.96 (s, 1H, NH<sub>3</sub>-CH), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.82-1.47 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 20H, (CH<sub>2</sub>)<sub>10</sub>), 0.88 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.87 (C=O), 145.30 (**C(i)** Tosyl), 137.80 (**C(p)** Tosyl), 128.05 (**C(m)** Tosyl), 125.49 (**C(o)** Tosyl), 65.57 (CH<sub>2</sub>-O), 50.63 (CH-NH), 39.20 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 31.29, 29.05, 29.03, 29.01, 28.93, 28.91, 28.71, 28.55, 27.87, 25.20, 23.78, 22.09, 21.84, 20.74, 13.89.

*pentadecyl L-leucinate ammonium tosylate salt (5f)*



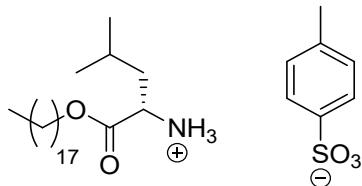
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.33 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 7.8 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H Ar-H), 4.12 (m, 2H, CH<sub>2</sub>-O), 3.96 (q, *J* = 6.0 Hz, 1H, NH<sub>3</sub>-CH), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.82-1.50 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 24H, (CH<sub>2</sub>)<sub>12</sub>), 0.87 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.87 (C=O), 145.33 (**C(i)** Tosyl), 137.78 (**C(p)** Tosyl), 128.04 (**C(m)** Tosyl), 125.49 (**C(o)** Tosyl), 65.57 (CH<sub>2</sub>-O), 50.62 (CH-NH), 39.20 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 31.30, 29.06, 29.04, 29.01, 28.93, 28.91, 28.71, 28.55, 27.87, 25.21, 23.78, 22.09, 21.84, 20.74, 13.88.

*hexadecyl L-leucinate ammonium tosylate salt (5g)*



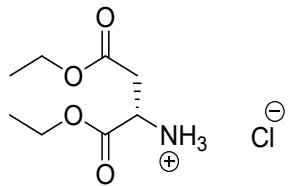
**93%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.33 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 7.9 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H Ar-H), 4.13 (m, *J* = 10.8, 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.96 (t, *J* = 7.0 Hz, 1H, NH<sub>3</sub>-CH), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.82-1.50 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 26H, (CH<sub>2</sub>)<sub>13</sub>), 0.88 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.87 (C=O), 145.37 (**C(i)** Tosyl), 137.74 (**C(p)** Tosyl), 128.03 (**C(m)** Tosyl), 125.48 (**C(o)** Tosyl), 65.57 (CH<sub>2</sub>-O), 50.61 (CH-NH), 39.20 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 31.30, 29.04, 29.01, 28.94, 28.92, 28.71, 28.55, 27.87, 25.21, 23.78, 22.09, 21.84, 20.73, 13.88.

*octadecyl L-leucinate ammonium tosylate salt (5h)*



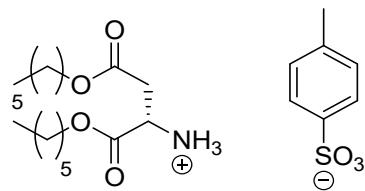
**99%**; white powder; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.33 (s, 3H, NH<sub>3</sub>), 7.51 (d, *J* = 7.8 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H Ar-H), 4.14 (m, *J* = 11.1, 6.4 Hz, 2H, CH<sub>2</sub>-O), 3.97 (t, *J* = 6.9 Hz, 1H, NH<sub>3</sub>-CH), 2.30 (s, 3H, Ar-CH<sub>3</sub>), 1.83-1.48 (m, 5H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 30H, (CH<sub>2</sub>)<sub>15</sub>), 0.87 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.87 (C=O), 145.41 (**C(i)** Tosyl), 137.71 (**C(p)** Tosyl), 128.02 (**C(m)** Tosyl), 125.48 (**C(o)** Tosyl), 65.56 (CH<sub>2</sub>-O), 50.60 (CH-NH), 39.20 (CH<sub>2</sub>-(CH<sub>3</sub>)<sub>2</sub>), 31.30, 29.05, 29.02, 28.95, 28.93, 28.72, 28.57, 27.87, 25.21, 23.77, 22.08, 21.84, 20.73, 13.86.

*diethyl L-aspartate ammonium chloride salt (6a)*



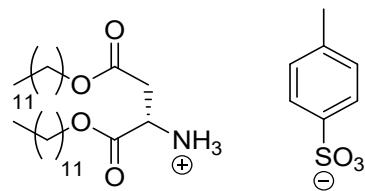
**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.85 (s, 3H, NH<sub>3</sub>), 4.37-3.98 (m, 5H, CH<sub>2</sub>-O + CH), 3.01 (m, 2H, CH<sub>2</sub>), 1.20 (td, *J* = 7.1, 1.8 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.02 (**C**=O), 168.14 (**C**=O), 61.91 (**CH<sub>2</sub>**-O), 60.84 (**CH<sub>2</sub>**-O), 48.44 (**CH**), 34.18 (**CH<sub>2</sub>**), 13.91 (**CH<sub>3</sub>**), 13.79 (**CH<sub>3</sub>**).

*dihexyl L-aspartate ammonium tosylate salt (6b)*



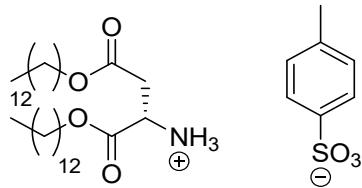
**63%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.41 (s, 3H, NH<sub>3</sub>), 7.50 (d, *J* = 8.1 Hz, 2H, Ar-H), 7.12 (d, *J* = 7.8 Hz, 2H Ar-H), 4.35 (t, *J* = 5.4 Hz, 1H, CH), 4.22-3.95 (m, 4H, CH<sub>2</sub>-O), 2.91 (m, 2H, CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.56 (m, *J* = 7.1 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.28 (m, 12H, (CH<sub>2</sub>)<sub>3</sub>), 0.87 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.13 (**C**=O), 168.31 (**C**=O), 145.31 (**C(i)** Tosyl), 137.83 (**C(p)** Tosyl), 128.07 (**C(m)** Tosyl), 125.49 (**C(o)** Tosyl), 65.89 (**CH<sub>2</sub>**-O), 64.88 (**CH<sub>2</sub>**-O), 48.58 (**CH-NH**), 34.26 (CH-CH<sub>2</sub>), 30.87, 30.82, 27.92, 27.82, 24.95, 24.81, 21.97, 20.75, 13.82.

*didodecyl L-aspartate ammonium tosylate salt (6d)*



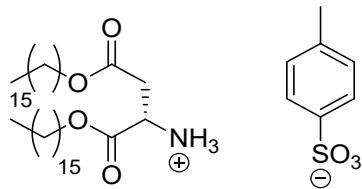
**98%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.44 (s, 3H, NH<sub>3</sub>), 7.50 (d, 2H, Ar-H), 7.11 (d, *J* = 7.9 Hz, 2H Ar-H), 4.36 (t, *J* = 5.4 Hz, 1H, CH), 4.23-3.98 (m, 4H, CH<sub>2</sub>-O), 2.92 (qd, *J* = 17.4, 5.4 Hz, 2H, CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.56 (m, *J* = 6.9 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 36H, (CH<sub>2</sub>)<sub>9</sub>), 0.85 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.04, 168.19, 145.35, 137.74, 128.03, 125.49, 65.87, 64.84, 48.50, 34.15, 31.33, 29.13, 29.10, 29.06, 29.00, 28.76, 28.70, 27.99, 27.87, 25.32, 25.21, 22.10, 20.73, 13.85.

*ditridecyl L-aspartate ammonium tosylate salt (6e)*



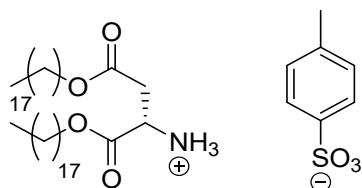
**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.44 (s, 3H, NH<sub>3</sub>), 7.50 (d, 2H, Ar-H), 7.11 (d, J = 7.9 Hz, 2H Ar-H), 4.36 (t, J = 5.4 Hz, 1H, CH), 4.24-3.94 (m, 4H, CH<sub>2</sub>-O), 2.92 (qd, J = 17.4, 5.4 Hz, 2H, CH<sub>2</sub>), 2.29 (s, 3H, Ar-CH<sub>3</sub>), 1.54 (m, J = 6.7 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 40H, (CH<sub>2</sub>)<sub>10</sub>), 0.84 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 169.02, 168.19, 145.35, 137.73, 128.02, 125.49, 65.86, 64.82, 48.49, 34.14, 31.34, 29.14, 29.11, 29.07, 29.02, 28.76, 28.71, 28.00, 27.87, 25.32, 25.22, 22.10, 20.73, 13.83.

*dihexadecyl L-aspartate ammonium tosylate salt (6g)*



**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.47 (s, 3H, NH<sub>3</sub>), 7.56 (d, 2H, Ar-H), 7.11 (d, J = 7.9 Hz, 2H Ar-H), 4.40-3.94 (m, 5H, CH + CH<sub>2</sub>-O), 2.94 (m, 2H, CH<sub>2</sub>), 2.31 (s, 3H, Ar-CH<sub>3</sub>), 1.58 (p, J = 6.9 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 52H, (CH<sub>2</sub>)<sub>13</sub>), 0.86 (t, 6H, CH<sub>3</sub>).

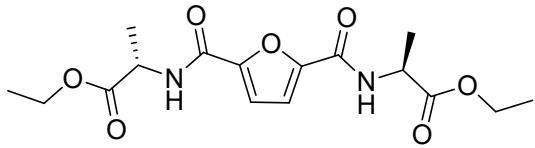
*dioctadecyl L-aspartate ammonium tosylate salt (6h)*



**99%**; white powder; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.49 (s, 3H, NH<sub>3</sub>), 7.59 (d, 2H, Ar-H), 7.11 (d, J = 7.8 Hz, 2H Ar-H), 4.32-3.99 (m, 5H, CH + CH<sub>2</sub>-O), 2.97 (dd, J = 5.4, 3.0 Hz, 2H, CH<sub>2</sub>), 2.32 (s, 3H, Ar-CH<sub>3</sub>), 1.59 (m, J = 6.5 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 60H, (CH<sub>2</sub>)<sub>15</sub>), 0.86 (t, 6H, CH<sub>3</sub>).

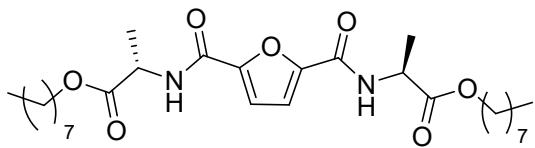
**Di(amido-esters).**

*N<sup>2</sup>, N<sup>5</sup>-di(ethyl L-alaninate)-furan-2,5-dicarboxamide (8a)*



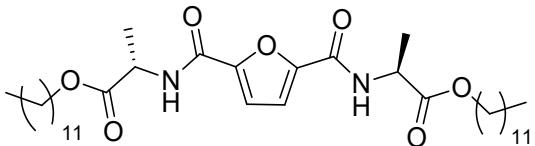
**84%**; yellow solid; **T<sub>f</sub>**: 109.6°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.49 (d, *J* = 7.9 Hz, 2H, NH), 7.13 (s, 2H, CH furan), 4.58 (m, 2H, CH-NH), 4.23-4.05 (m, 4H, CH<sub>2</sub>-O), 1.49 (d, *J* = 7.3 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.25 (t, *J* = 7.1 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.00 (C=O ester), 156.84 (C=O amide), 147.69 (C-C=O amide), 114.65 (CH furan), 60.54 (CH<sub>2</sub>-O), 47.42 (NH-CH), 16.77 (CH<sub>3</sub>-CH), 13.47 (CH<sub>3</sub>-CH<sub>2</sub>).

*N<sup>2</sup>, N<sup>5</sup>-di(octyl L-alaninate)-furan-2,5-dicarboxamide (8c)*



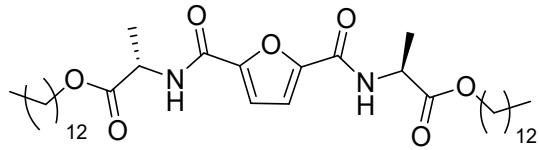
**84%**; yellow solid; **T<sub>f</sub>**: 51.3°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.53 (d, *J* = 7.6 Hz, 2H, NH), 7.13 (s, 2H, CH furan), 4.58 (p, *J* = 7.4 Hz, 2H, CH-NH), 4.22-3.94 (m, 4H, CH<sub>2</sub>-O), 1.61 (p, *J* = 6.7 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.48 (d, *J* = 7.3 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.26 (m, 20H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>5</sub>), 0.84 (t, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.09 (C=O ester), 156.90 (C=O amide), 147.72 (C-C=O amide), 114.68 (CH furan), 64.63 (CH<sub>2</sub>-O), 47.50 (NH-CH), 31.12, 28.52, 27.98, 25.20, 22.00, 16.78, 13.46.

*N<sup>2</sup>, N<sup>5</sup>-di(dodecyl L-alaninate)-furan-2,5-dicarboxamide (8d)*



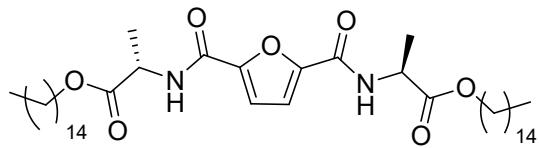
**75%**; yellow paste; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.44 (d, *J* = 7.8 Hz, 2H, NH), 7.11 (s, 2H, CH furan), 4.61 (p, *J* = 7.3 Hz, 2H, CH-NH), 4.18-4.04 (m, 4H, CH<sub>2</sub>-O), 1.62 (p, *J* = 6.9 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.49 (d, *J* = 7.3 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.25 (m, 36H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.86 (t, *J* = 6.5 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.07 (C=O ester), 156.84 (C=O amide), 147.71 (C-C=O amide), 114.63 (CH furan), 64.63 (CH<sub>2</sub>-O), 47.44 (NH-CH), 31.27, 28.99, 28.97, 28.91, 28.87, 28.69, 28.57, 27.97, 25.20, 22.04, 16.80, 13.43.

*N<sup>2</sup>, N<sup>5</sup>-di(tridecyl L-alaninate)-furan-2,5-dicarboxamide (8e)*



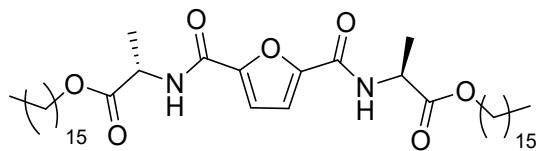
**80%**; yellow paste; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.52 (d, *J* = 7.7 Hz, 2H, NH), 7.12 (s, 2H, CH furan), 4.59 (p, *J* = 7.4 Hz, 2H, CH-NH), 4.09 (m, *J* = 10.8, 6.6 Hz, 4H, CH<sub>2</sub>-O), 1.61 (p, *J* = 6.7 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.48 (d, *J* = 7.3 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.24 (m, 40H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>10</sub>), 0.86 (t, *J* = 6.7 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.04 (C=O ester), 156.83 (C=O amide), 147.63 (C-C=O amide), 114.63 (CH furan), 64.58 (CH<sub>2</sub>-O), 47.45 (NH-CH), 31.25, 28.99, 28.96, 28.89, 28.85, 28.67, 28.55, 27.96, 25.18, 22.02, 16.77, 13.47.

***N*<sup>2</sup>, *N*<sup>5</sup>-di(pentadecyl L-alaninate)-furan-2,5-dicarboxamide (8f)**



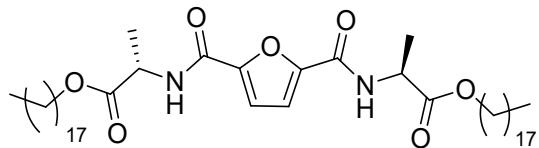
**63%**; yellow paste; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.50 (d, *J* = 7.7 Hz, 2H, NH), 7.12 (s, 2H, CH furan), 4.59 (p, *J* = 7.3 Hz, 2H, CH-NH), 4.09 (m, *J* = 10.8, 6.5 Hz, 4H, CH<sub>2</sub>-O), 1.61 (p, *J* = 6.8 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.48 (d, *J* = 7.3 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.24 (m, 48H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>12</sub>), 0.86 (t, *J* = 6.5 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.04 (C=O ester), 156.82 (C=O amide), 147.70 (C-C=O amide), 114.62 (CH furan), 64.58 (CH<sub>2</sub>-O), 47.43 (NH-CH), 31.25, 29.01, 28.99, 28.97, 28.89, 28.85, 28.67, 28.55, 27.96, 25.18, 22.02, 16.77, 13.46.

***N*<sup>2</sup>, *N*<sup>5</sup>-di(hexadecyl L-alaninate)-furan-2,5-dicarboxamide (8g)**



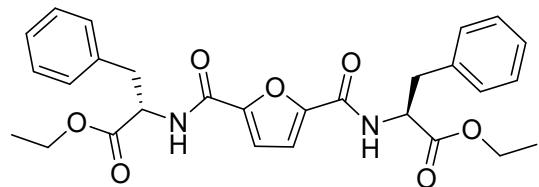
**71%**; yellow solid; **T<sub>f</sub>**: 32°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.51 (d, *J* = 7.6 Hz, 2H, NH), 7.12 (s, 2H, CH furan), 4.60 (p, *J* = 7.3 Hz, 2H, CH-NH), 4.09 (m, 4H, CH<sub>2</sub>-O), 1.61 (p, *J* = 6.9 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.48 (d, *J* = 7.3 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.24 (m, 52H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>13</sub>), 0.86 (t, *J* = 6.7 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.19 (C=O ester), 172.04 (C=O ester), 156.82 (C=O amide), 147.70 (C-C=O amide), 147.66 (C-C=O amide), 114.61 (CH furan), 64.58 (CH<sub>2</sub>-O), 47.43 (NH-CH), 47.36 (NH-CH), 31.25, 29.12 – 28.44 (m), 27.96, 25.18, 22.02, 16.77 (CH-CH<sub>3</sub>), 16.69 (CH-CH<sub>3</sub>), 13.45.

*N<sup>2</sup>, N<sup>5</sup>-di(octadecyl L-alaninate)-furan-2,5-dicarboxamide (8h)*



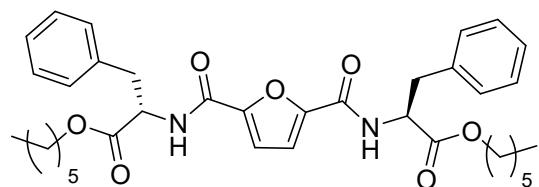
**88%**; white solid; **Tf:** 45.3°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.49 (dd, *J* = 13.4, 7.7 Hz, 2H, NH), 7.12 (d, *J* = 2.3 Hz, 2H, CH furan), 4.62 (pd, *J* = 7.4, 3.1 Hz, 2H, CH-NH), 4.18-4.04 (m, 4H, CH<sub>2</sub>-O), 1.62 (m, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.50 (dd, *J* = 7.4, 1.7 Hz, 6H, CH<sub>3</sub>-CH-NH<sub>3</sub>), 1.26 (m, 60H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>15</sub>), 0.87 (t, *J* = 6.6 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.03 (C=O ester), 156.80 (C=O amide), 147.67 (C-C=O amide), 114.59 (CH furan), 64.59, 47.40, 31.23, 28.99, 28.95, 28.88, 28.83, 28.65, 28.54, 27.94, 25.16, 22.01, 16.78, 16.69, 13.41.

*N<sup>2</sup>, N<sup>5</sup>-di(ethyl L-phenylalaninate)-furan-2,5-dicarboxamide (9a)*



**62%**; yellow solid; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.56 (d, *J* = 8.1 Hz, 2H, NH), 7.32-7.15 (m, 10H, Ar-H), 7.10 (s, 2H, CH furan), 4.77 (td, *J* = 8.5, 6.0 Hz, 2H, CH-NH), 4.14 (q, *J* = 7.1 Hz, 4H, CH<sub>2</sub>-O), 3.30-3.07 (m, 4H, CH<sub>2</sub>-Ph), 1.19 (t, *J* = 7.1 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.91 (C=O ester), 156.93 (C=O amide), 147.65 (C-C=O amide), 136.73 (C(i) Ph), 128.74 (CH(m) Ph), 127.88 (CH(o) Ph), 126.20 (CH(p) Ph), 114.67 (CH furan), 60.63 (CH<sub>2</sub>-O), 53.39 (NH-CH), 36.75 (CH<sub>2</sub>-Ph), 13.49 (CH<sub>3</sub>).

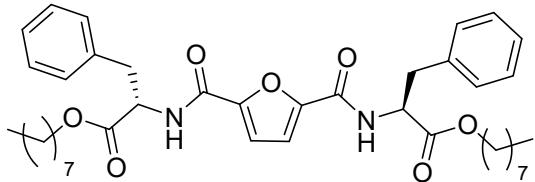
*N<sup>2</sup>, N<sup>5</sup>-di(hexyl L-phenylalaninate)-furan-2,5-dicarboxamide (9b)*



**81%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.61 (q, *J* = 7.5, 6.1 Hz, 2H, NH), 7.38-7.15 (m, 10H, Ar-H), 7.11 (s, 2H, CH furan), 4.76 (q, *J* = 7.3, 6.8 Hz, 2H CH-NH), 4.07 (t, *J* = 6.5 Hz, 4H, CH<sub>2</sub>-O), 3.38-3.03 (m, 4H, CH<sub>2</sub>-Ph), 1.54 (m, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.26 (m, 12H, (CH<sub>2</sub>)<sub>3</sub>), 0.85 (t, *J* = 6.1 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.01 (C=O ester), 156.95 (C=O amide), 147.66 (C-C=O amide), 136.80 (C(i) Ph), 128.72 (CH(m) Ph), 127.91 (CH(o) Ph), 126.22

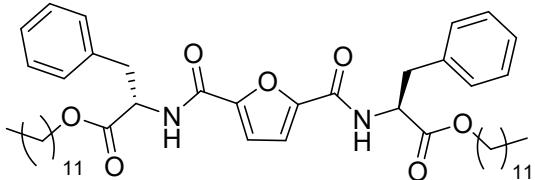
(CH(**p**) Ph), 114.67 (CH furan), 64.70 (CH<sub>2</sub>-O), 53.40 (NH-CH), 36.73 (CH<sub>2</sub>-Ph), 30.73, 27.85, 24.83, 21.86, 13.36.

*N<sup>2</sup>, N<sup>5</sup>-di(octyl L-phenylalaninate)-furan-2,5-dicarboxamide (**9c**)*



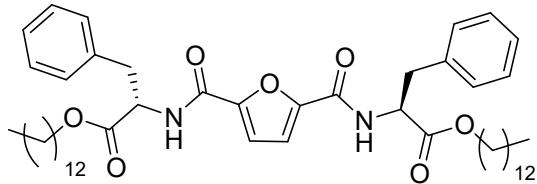
**80%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.65 (d, *J* = 8.3 Hz, NH (**R,S**)), 8.59 (d, *J* = 8.1 Hz, 2H, NH (**S,S**)), 7.41-7.14 (m, 10H, Ar-H), 7.10 (s, 2H, CH furan (**S,S**)), 7.05 (s, CH furan (**R,S**)), 4.76 (m, *J* = 6.1 Hz, 2H, CH-NH), 4.08 (m, *J* = 6.5 Hz, 4H, CH<sub>2</sub>-O), 3.39-2.96 (m, 4H, CH<sub>2</sub>-Ph), 1.55 (h, *J* = 6.5 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 20H, (CH<sub>2</sub>)<sub>5</sub>), 0.86 (t, *J* = 6.8 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.30 (C=O ester (**R,S**)), 170.99 (C=O ester (**S,S**)), 156.97 (C=O amide (**R,S**)), 156.92 (C=O amide (**S,S**)), 147.64 (C-C=O amide (**S,S**)), 147.49 (C-C=O amide (**R,S**)), 136.80 (C(i) Ph (**R,S**)), 136.77 (C(i) Ph (**S,S**)), 128.71 (CH(m) Ph), 127.92 (CH(o) Ph (**R,S**)), 127.89 (CH(o) Ph (**S,S**)), 126.24 (CH(p) Ph (**R,S**)), 126.20 (CH(p) Ph (**S,S**)), 114.65 (CH furan), 64.78 (CH<sub>2</sub>-O (**R,S**)), 64.70 (CH<sub>2</sub>-O (**S,S**)), 53.43 (NH-CH (**R,S**)), 53.38 (NH-CH (**S,S**)), 36.74 (CH<sub>2</sub>-Ph (**S,S**)), 36.69 (CH<sub>2</sub>-Ph (**R,S**)), 31.11, 28.49, 28.47, 27.91, 27.87, 25.16, 21.98, 13.46.

*N<sup>2</sup>, N<sup>5</sup>-di(dodecyl L-phenylalaninate)-furan-2,5-dicarboxamide (**9d**)*



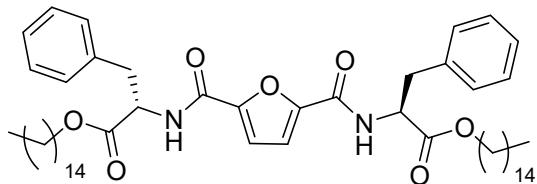
**77%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.60 (d, *J* = 8.3 Hz, NH (**R,S**)), 8.50 (d, *J* = 8.1 Hz, 2H, NH (**S,S**)), 7.37-7.14 (m, 10H, Ar-H), 7.09 (s, 2H, CH furan (**S,S**)), 7.04 (s, CH furan (**R,S**)), 4.78 (m, *J* = 6.0 Hz, 2H, CH-NH), 4.07 (t, *J* = 6.7 Hz, 4H, CH<sub>2</sub>-O), 3.31-3.08 (m, 4H, CH<sub>2</sub>-Ph), 1.55 (p, *J* = 6.4 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 36H, (CH<sub>2</sub>)<sub>9</sub>), 0.86 (t, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.30 (C=O ester (**R,S**)), 171.01 (C=O ester (**S,S**)), 156.98 (C=O amide (**R,S**)), 156.95 (C=O amide (**S,S**)), 147.68 (C-C=O amide (**S,S**)), 147.53 (C-C=O amide (**R,S**)), 136.84 (C(i) Ph), 128.74 (CH(m) Ph), 127.93 (CH(o) Ph), 126.23 (CH(p) Ph), 114.69 (CH furan), 64.77 (CH<sub>2</sub>-O (**R,S**)), 64.68 (CH<sub>2</sub>-O (**S,S**)), 53.44 (NH-CH), 36.70 (CH<sub>2</sub>-Ph), 31.26, 28.99, 28.97, 28.91, 28.84, 28.68, 28.56, 27.91, 25.18, 22.04, 13.54.

*N<sup>2</sup>, N<sup>5</sup>-di(tridecyl L-phenylalaninate)-furan-2,5-dicarboxamide (9e)*



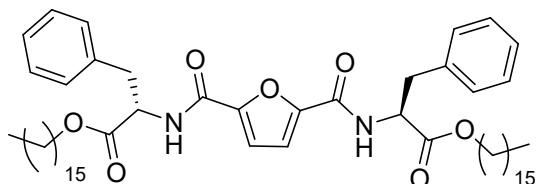
**78%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.59 (d, *J* = 8.1 Hz, 2H, NH), 7.31-7.16 (m, 10H, Ar-H), 7.10 (s, 2H, CH furan), 4.77 (td, *J* = 8.4, 6.0 Hz, 2H, CH-NH), 4.06 (t, *J* = 6.6 Hz, 4H, CH<sub>2</sub>-O), 3.28-3.10 (m, 4H, CH<sub>2</sub>-Ph), 1.55 (p, *J* = 6.6 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 m, 40H, (CH<sub>2</sub>)<sub>10</sub>), 0.87 (t, *J* = 6.6 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.99 (C=O ester), 156.92 (C=O amide), 147.66 (C-C=O amide), 136.77 (**C(i)** Ph), 128.71 (**CH(m)** Ph), 127.89 (**CH(o)** Ph), 126.20 (**CH(p)** Ph), 114.65 (CH furan), 64.69 (CH<sub>2</sub>-O), 53.39 (NH-CH), 36.76 (CH<sub>2</sub>-Ph), 31.25, 29.01, 28.97, 28.90, 28.83, 28.67, 28.55, 27.89, 25.17, 22.03, 13.49.

*N<sup>2</sup>, N<sup>5</sup>-di(pentadecyl L-phenylalaninate)-furan-2,5-dicarboxamide (9f)*



**65%**; yellow solid; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.59 (d, *J* = 8.1 Hz, 2H, NH), 7.32-7.15 (m, 10H, Ar-H), 7.10 (s, 2H, CH furan), 4.77 (td, *J* = 8.4, 6.0 Hz, 2H, CH-NH), 4.07 (t, *J* = 6.6 Hz, 4H, CH<sub>2</sub>-O), 3.30-3.09 (m, 4H, CH<sub>2</sub>-Ph), 1.55 (p, *J* = 6.6 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 m, 48H, (CH<sub>2</sub>)<sub>12</sub>), 0.87 (t, *J* = 6.7 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.02 (C=O ester), 156.97 (C=O amide), 147.71 (C-C=O amide), 136.89 (**C(i)** Ph), 128.77 (**CH(m)** Ph), 127.94 (**CH(o)** Ph), 126.25 (**CH(p)** Ph), 114.71 (CH furan), 64.68 (CH<sub>2</sub>-O), 53.47 (NH-CH), 36.72 (CH<sub>2</sub>-Ph), 31.29, 29.06, 29.02, 28.95, 28.88, 28.72, 28.60, 27.94, 25.21, 22.07, 13.57.

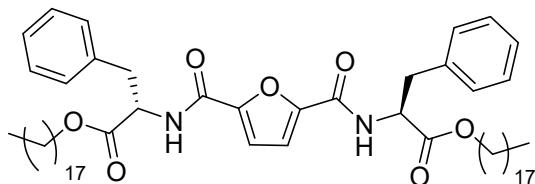
*N<sup>2</sup>, N<sup>5</sup>-di(hexadecyl L-phenylalaninate)-furan-2,5-dicarboxamide (9g)*



**70%**; yellow paste; **Tf:** 8°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.66 (d, *J* = 8.3 Hz, NH (**R,S**)), 8.60 (d, *J* = 8.1 Hz, 2H, NH (**S,S**))), 7.32 – 7.16 (m, 10H, Ar-H), 7.10 (s, 2H, CH furan (**S,S**))),

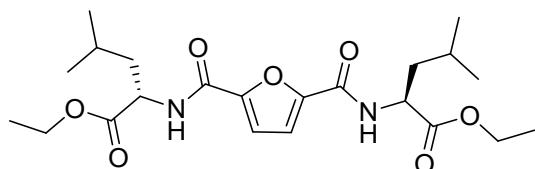
7.05 (s, CH furan (**R,S**)), 4.76 (td,  $J = 8.4, 6.0$  Hz, 2H, CH-NH), 4.06 (t,  $J = 6.7$  Hz, 4H, CH<sub>2</sub>-O), 3.31-3.07 (m, 4H, CH<sub>2</sub>-Ph), 1.55 (p,  $J = 6.5$  Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 52H, (CH<sub>2</sub>)<sub>13</sub>), 0.86 (t,  $J = 6.6$  Hz, 6H, CH<sub>3</sub>); <sup>13</sup>C NMR (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.29 (C=O ester (**R,S**)), 170.98 (C=O ester (**S,S**)), 156.96 (C=O amide (**R,S**)), 156.92 (C=O amide (**S,S**)), 147.67 (C-C=O amide (**S,S**)), 147.51 (C-C=O amide (**R,S**)), 136.80 (C(i) Ph), 128.72 (CH(m) Ph), 127.89 (CH(o) Ph), 126.25 (CH(p) Ph), 114.65 (CH furan), 64.77 (CH<sub>2</sub>-O (**R,S**)), 64.68 (CH<sub>2</sub>-O (**S,S**)), 53.40 (NH-CH), 36.75 (CH<sub>2</sub>-Ph), 31.26, 29.02, 28.98, 28.91, 28.84, 28.68, 28.56, 27.90, 25.17, 22.03, 13.50.

*N<sup>2</sup>, N<sup>5</sup>-di(octadecyl L-phenylalaninate)-furan-2,5-dicarboxamide (**9h**)*



**90%**; white solid; Tf: 32.7°C; <sup>1</sup>H NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.62 (d,  $J = 8.3$  Hz, NH (**R,S**)), 8.54 (d,  $J = 8.1$  Hz, 2H, NH (**S,S**)), 7.35-7.16 (m, 10H, Ar-H), 7.10 (s, 2H, CH furan (**S,S**)), 7.05 (s, CH furan (**R,S**)), 4.79 (td,  $J = 8.4, 6.1$  Hz, 2H, CH-NH), 4.10 (dt,  $J = 16.0, 6.7$  Hz, 4H, CH<sub>2</sub>-O (**S,S**)), 3.94 (m, CH<sub>2</sub>-O (**R,S**)), 3.44 (q,  $J = 6.3$  Hz, CH<sub>2</sub>-Ph (**R,S**)), 3.31-3.11 (m, 4H, CH<sub>2</sub>-Ph (**S,S**)), 1.58 (p,  $J = 6.6$  Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.26 (m, 60H, (CH<sub>2</sub>)<sub>15</sub>), 0.86 (t,  $J = 6.6$  Hz, 6H, CH<sub>3</sub>); <sup>13</sup>C NMR (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.27 (C=O ester (**R,S**)), 170.95 (C=O ester (**S,S**)), 156.93 (C=O amide (**R,S**)), 156.88 (C=O amide (**S,S**)), 147.63 (C-C=O amide (**S,S**)), 147.47 (C-C=O amide (**R,S**)), 136.73 (C(i) Ph (**R,S**)), 136.69 (C(i) Ph (**R,S**)), 128.67 (CH(m) Ph), 127.87 (CH(o) Ph (**R,S**)), 127.85 (CH(o) Ph (**S,S**)), 126.20 (CH(p) Ph (**R,S**)), 126.17 (CH(p) Ph (**S,S**)), 114.61 (CH furan), 64.77 (CH<sub>2</sub>-O (**R,S**)), 64.69 (CH<sub>2</sub>-O (**S,S**)), 53.39 (NH-CH (**R,S**)), 53.34 (NH-CH (**R,S**)), 36.79 (CH<sub>2</sub>-Ph), 31.23, 28.99, 28.96, 28.89, 28.82, 28.65, 28.54, 27.90, 27.86, 25.15, 22.00, 13.43.

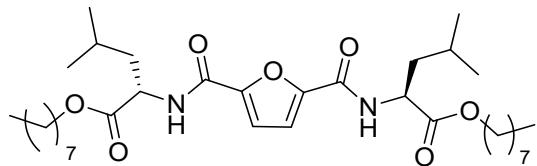
*N<sup>2</sup>, N<sup>5</sup>-di(ethyl L-leucinate)-furan-2,5-dicarboxamide (**10a**)*



**85%**; yellow oil; <sup>1</sup>H NMR (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.51 (dd,  $J = 8.4, 2.6$  Hz, 2H, NH), 7.17 (d, 2H, CH furan), 4.63 (m, 2H, CH-NH<sub>3</sub>), 4.16 (m, 4H, CH<sub>2</sub>-O), 1.72 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub>), 1.25 (m, 6H, CH<sub>3</sub>), 0.94 (m, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>); <sup>13</sup>C NMR (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm):

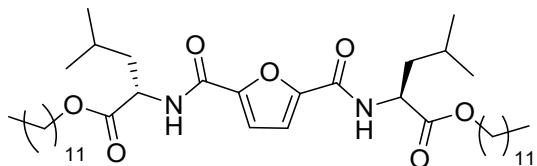
172.02 (**C=O** ester), 157.12 (**C=O** amide), 147.77 (**C-C=O** amide), 114.74 (**CH** furan), 60.49 (**CH<sub>2</sub>-O**), 50.07 (**NH-CH**), 39.81 (**CH-(CH<sub>3</sub>)<sub>2</sub>**), 24.27, 22.33, 20.93, 13.63.

*N<sup>2</sup>, N<sup>5</sup>-di(octyl L-leucinate)-furan-2,5-dicarboxamide (10c)*



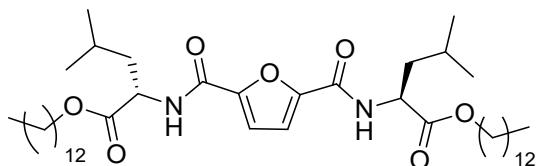
**89%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.47 (d, *J*= 8.3 Hz, 2H, NH), 7.15 (s, 2H, CH furan), 4.62 (m, 2H, CH-NH<sub>3</sub>), 4.09 (m, 4H, CH<sub>2</sub>-O), 1.83-1.55 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.26 (m, 20H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>5</sub>), 0.94 (dd, *J*= 11.9, 5.1 Hz, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>), 0.85 (t, *J*= 6.7 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.06 (**C=O** ester), 157.08 (**C=O** amide), 147.77 (**C-C=O** amide), 114.70 (**CH** furan), 64.52 (**CH<sub>2</sub>-O**), 50.09 (**NH-CH**), 39.87 (**CH-(CH<sub>3</sub>)<sub>2</sub>**), 31.08, 28.49, 28.46, 27.93, 25.19, 24.28, 22.26, 21.97, 20.97, 13.47.

*N<sup>2</sup>, N<sup>5</sup>-di(dodecyl L-leucinate)-furan-2,5-dicarboxamide (10d)*



**88%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.47 (d, *J*= 8.3 Hz, 2H, NH), 7.15 (s, 2H, CH furan), 4.62 (m, 2H, CH-NH<sub>3</sub>), 4.09 (m, 4H, CH<sub>2</sub>-O), 1.84-1.52 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 36H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.94 (dd, *J*= 11.7, 5.3 Hz, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>), 0.86 (t, *J*= 6.6 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.04 (**C=O** ester), 157.07 (**C=O** amide), 147.78 (**C-C=O** amide), 114.69 (**CH** furan), 64.50 (**CH<sub>2</sub>-O**), 50.10 (**NH-CH**), 39.88 (**CH-(CH<sub>3</sub>)<sub>2</sub>**), 31.24, 28.96, 28.94, 28.87, 28.84, 28.66, 28.52, 27.94, 25.20, 24.28, 22.27, 22.02, 20.99, 13.49.

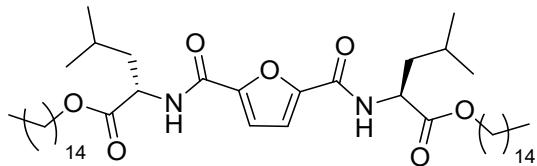
*N<sup>2</sup>, N<sup>5</sup>-di(tridecyl L-leucinate)-furan-2,5-dicarboxamide (10e)*



**73%**; yellow oil; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.47 (d, *J*= 8.3 Hz, 2H, NH), 7.15 (s, 2H, CH furan), 4.62 (m, 2H, CH-NH<sub>3</sub>), 4.08 (m, 4H, CH<sub>2</sub>-O), 1.85-1.52 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 40H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>10</sub>), 0.94 (dd, *J*= 8.7, 5.7 Hz, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>), 0.85 (t, *J*= 6.7 Hz,

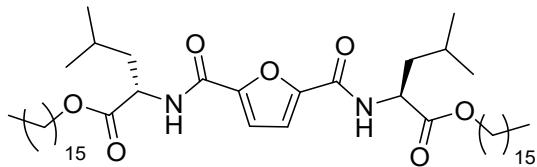
6H, **CH<sub>3</sub>-CH<sub>2</sub>**); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.02 (**C=O ester**), 157.06 (**C=O amide**), 147.77 (**C-C=O amide**), 114.67 (**CH furan**), 64.49 (**CH<sub>2</sub>-O**), 50.09 (**NH-CH**), 39.87 (**CH-(CH<sub>3</sub>)<sub>2</sub>**), 31.23, 28.97, 28.94, 28.85, 28.82, 28.64, 28.50, 27.93, 25.18, 24.27, 22.25, 22.00, 20.98, 13.48.

*N<sup>2</sup>, N<sup>5</sup>-di(pentadecyl L-leucinate)-furan-2,5-dicarboxamide (10f)*



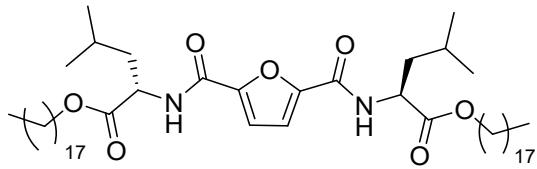
**65%**; yellow oil; **Tf:** -1°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.53 (d, **NH (R,S)**), 8.47 (d, *J* = 8.3 Hz, 2H, **NH (S,S)**), 7.15 (s, 2H, **CH furan (S,S)**), 7.12 (s, **CH furan (R,S)**), 4.62 (m, 2H, **CH-NH<sub>3</sub>**), 4.08 (m, 4H, **CH<sub>2</sub>-O**), 1.85-1.52 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 48H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>12</sub>), 0.94 (dd, *J* = 8.7, 5.8 Hz, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>), 0.85 (t, *J* = 6.6 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.02 (**C=O ester**), 157.06 (**C=O amide**), 147.77 (**C-C=O amide**), 114.68 (**CH furan**), 64.49 (**CH<sub>2</sub>-O**), 50.10 (**NH-CH**), 39.87 (**CH-(CH<sub>3</sub>)<sub>2</sub>**), 31.23, 28.98, 28.95, 28.86, 28.83, 28.65, 28.51, 27.93, 25.18, 24.28, 22.26, 22.01, 20.99, 13.49.

*N<sup>2</sup>, N<sup>5</sup>-di(hexadecyl L-leucinate)-furan-2,5-dicarboxamide (10g)*



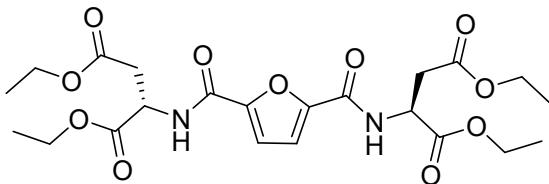
**88%**; yellow paste; **Tf:** 11,6°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.50 (d, *J* = 8.2 Hz, **NH (R,S)**), 8.43 (d, *J* = 8.3 Hz, 2H, **NH (S,S)**), 7.14 (s, 2H, **CH furan (S,S)**), 7.11 (s, **CH furan (R,S)**), 4.63 (m, 2H, **CH-NH**), 4.09 (m, 4H, **CH<sub>2</sub>-O**), 1.84-1.53 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 52H, (CH<sub>2</sub>)<sub>13</sub>), 0.95 (m, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>), 0.86 (t, *J* = 6.7 Hz, 6H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.30 (**C=O ester (R,S)**), 172.04 (**C=O ester (S,S)**), 157.10 (**C=O amide (R,S)**), 157.05 (**C=O amide (S,S)**), 147.76 (**C-C=O amide (S,S)**), 147.63 (**C-C=O amide (R,S)**), 114.66 (**CH furan (S,S)**), 114.54 (**CH furan (R,S)**), 64.51 (**CH<sub>2</sub>-O**), 50.20 (**NH-CH (R,S)**), 50.07 (**NH-CH (S,S)**), 39.63 (**CH-(CH<sub>3</sub>)<sub>2</sub>**), 31.24, 28.99, 28.96, 28.87, 28.83, 28.66, 28.51, 27.92, 25.18, 24.34, 24.27, 22.22, 22.01, 20.96, 20.84, 13.45.

*N<sup>2</sup>, N<sup>5</sup>-di(octadecyl L-leucinate)-furan-2,5-dicarboxamide (10h)*



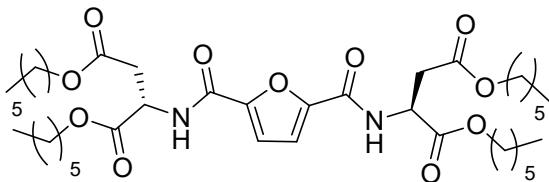
**77%**; yellow solid; **T<sub>f</sub>**: 26.9°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.52 (d, *J* = 7.9 Hz, NH (**R,S**)), 8.45 (d, *J* = 8.2 Hz, 2H, NH (**S,S**)), 7.14 (s, 2H, CH furan (**S,S**)), 7.11 (s, CH furan (**R,S**)), 4.62 (m, 2H, CH-NH), 4.09 (m, 4H, CH<sub>2</sub>-O), 1.83-1.52 (m, 10H, (CH<sub>3</sub>)<sub>2</sub>-CH-CH<sub>2</sub> + CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 60H, (CH<sub>2</sub>)<sub>15</sub>), 0.94 (dd, *J* = 11.5, 5.3 Hz, 12H, CH-(CH<sub>3</sub>)<sub>2</sub>), 0.85 (t, *J* = 6.5 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 172.30 (**C=O** ester (**R,S**)), 172.05 (**C=O** ester (**S,S**)), 157.11 (**C=O** amide (**R,S**)), 157.06 (**C=O** amide (**S,S**)), 147.75 (**C-C=O** amide (**S,S**)), 147.63 (**C-C=O** amide (**R,S**)), 114.68 (CH furan (**S,S**)), 114.57 (CH furan (**R,S**)), 64.51 (CH<sub>2</sub>-O), 50.20 (NH-CH (**R,S**)), 50.08 (NH-CH (**S,S**)), 39.90 (CH-(CH<sub>3</sub>)<sub>2</sub>), 31.25, 29.00, 28.96, 28.87, 28.84, 28.67, 28.52, 27.93, 25.19, 24.34, 24.27, 22.25, 22.02, 20.97, 20.85, 13.48.

*N<sup>2</sup>, N<sup>5</sup>-bis(diethyl L-aspartate)-furan-2,5-dicarboxamide (11a)*



**82%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.73 (d, *J* = 8.2 Hz, 2H, NH), 7.16 (s, 2H, CH furan), 4.88 (qd, *J* = 7.4, 6.6, 1.3 Hz, 2H, CH-NH), 4.25-4.01 (m, 8H, CH<sub>2</sub>-O), 3.08-2.72 (m, 4H, CH<sub>2</sub>-C=O), 1.21 (q, *J* = 7.2 Hz, 12H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.98 (**C=O** ester), 169.74 (**C=O** ester), 156.90 (**C=O** amide), 147.66 (**C-C=O** amide), 114.89 (CH furan), 60.97 (CH<sub>2</sub>-O), 60.20 (CH<sub>2</sub>-O), 48.54 (NH-CH), 35.56 (CH<sub>2</sub>), 13.55 (CH<sub>3</sub>), 13.53 (CH<sub>3</sub>).

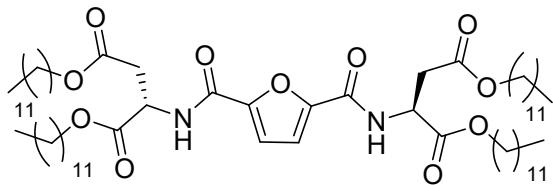
*N<sup>2</sup>, N<sup>5</sup>-bis(dihexyl L-aspartate)-furan-2,5-dicarboxamide (11b)*



**86%**; yellow oil; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.74 (d, *J* = 8.1 Hz, 2H, NH), 7.16 ((d, *J* = 8.1 Hz, 2H, CH furan), 4.89 (q, *J* = 7.4 Hz, 2H, CH-NH), 4.07 (m, 8H, CH<sub>2</sub>-O), 3.07-2.79 (m, 4H, CH<sub>2</sub>-C=O), 1.57 (m, 8H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.27 (m, 24H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>3</sub>), 0.84 (t, *J* = 7.0 Hz, 12H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.03 (d, *J* = 4.2 Hz, **C=O** ester), 169.79 (**C=O**

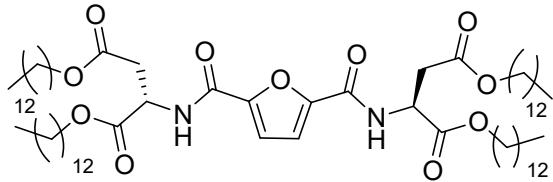
ester), 156.86 (**C**=O amide), 147.66 (**C**-**C**=O amide), 114.81 (CH furan), 64.99 (**CH**<sub>2</sub>-O), 64.29 (**CH**<sub>2</sub>-O), 48.53 (NH-CH), 35.52, 30.75, 30.70, 27.89, 27.83, 24.87, 24.80, 21.85, 13.37, 13.34.

*N*<sup>2</sup>, *N*<sup>5</sup>-bis(didodecyl L-aspartate)-furan-2,5-dicarboxamide (**11d**)



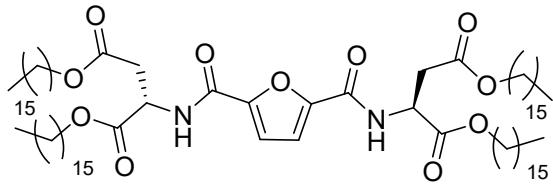
**75%**; yellow paste; **Tf:** -1,1°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.69 (d, *J* = 8.1 Hz, 2H, NH), 7.14 (s, 2H, CH furan), 4.89 (q, *J* = 7.0 Hz, 2H, CH-NH), 4.15-4.01 (m, 8H, CH<sub>2</sub>-O), 3.07-2.76 (m, 4H, CH<sub>2</sub>-C=O), 1.58 (m, 8H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 72H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.86 (t, *J* = 6.7 Hz, 12H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.95 (C=O ester), 169.75 (C=O ester), 156.82 (C=O amide), 147.66 (C-C=O amide), 114.79 (CH furan), 64.98 (CH<sub>2</sub>-O), 64.29 (CH<sub>2</sub>-O), 48.50 (NH-CH), 35.56, 31.24, 28.98, 28.95, 28.91, 28.85, 28.66, 28.59, 28.56, 27.94, 27.88, 25.22, 25.17, 22.01, 13.45.

*N*<sup>2</sup>, *N*<sup>5</sup>-bis(ditridecyl L-aspartate)-furan-2,5-dicarboxamide (**11e**)



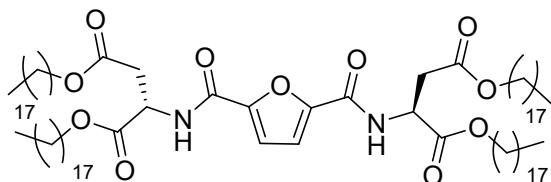
**74%**; yellow paste; **Tf:** 10,3°C; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.74 (d, *J* = 8.1 Hz, 2H, NH), 7.15 (s, 2H, CH furan), 4.88 (q, *J* = 7.0 Hz, 2H, CH-NH), 4.18-3.98 (m, 8H, CH<sub>2</sub>-O), 3.07-2.76 (m, 4H, CH<sub>2</sub>-C=O), 1.57 (m, 8H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.25 (m, 80H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>10</sub>), 0.86 (t, *J* = 6.7 Hz, 12H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.97 (C=O ester), 169.74 (C=O ester), 156.83 (C=O amide), 147.69 (C-C=O amide), 114.84 (CH furan), 64.94 (CH<sub>2</sub>-O), 64.26 (CH<sub>2</sub>-O), 48.54 (NH-CH), 35.56, 31.25, 29.01, 28.98, 28.93, 28.87, 28.68, 28.61, 28.57, 27.97, 27.90, 25.24, 25.19, 22.02, 13.53.

*N*<sup>2</sup>, *N*<sup>5</sup>-bis(dihexadecyl L-aspartate)-furan-2,5-dicarboxamide (**11g**)



**47%**; white solid; **T<sub>f</sub>**: 37.6°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.68 (d, *J* = 8.1 Hz, 2H, NH), 7.14 (s, 2H, CH furan), 4.89 (m, 2H, CH-NH), 4.19-3.95 (m, 8H, CH<sub>2</sub>-O), 3.07-2.76 (m, 4H, CH<sub>2</sub>-C=O), 1.59 (m, 8H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 104H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>13</sub>), 0.84 (t, 12H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.94 (C=O ester), 169.75 (C=O ester), 156.81 (C=O amide), 147.66 (C-C=O amide), 114.80 (CH furan), 64.97 (CH<sub>2</sub>-O), 64.28 (CH<sub>2</sub>-O), 48.49 (NH-CH), 35.56, 31.25, 29.03, 28.99, 28.93, 28.87, 28.68, 28.61, 28.57, 27.95, 27.89, 25.24, 25.18, 22.02, 13.45.

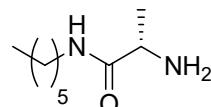
#### *N<sup>2</sup>, N<sup>5</sup>-bis(dioctadecyl L-aspartate)-furan-2,5-dicarboxamide (11h)*



**64%**; white solid; **T<sub>f</sub>**: 50.5°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.68 (d, *J* = 8.1 Hz, 2H, NH), 7.14 (s, 2H, CH furan), 4.88 (q, 2H, CH-NH), 4.19-3.98 (m, 8H, CH<sub>2</sub>-O), 3.07-2.76 (m, 4H, CH<sub>2</sub>-C=O), 1.58 (m, 8H, CH<sub>2</sub>-CH<sub>2</sub>-O), 1.24 (m, 120H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>15</sub>), 0.85 (t, 12H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.94 (C=O ester), 169.74 (C=O ester), 156.81 (C=O amide), 147.66 (C-C=O amide), 114.80 (CH furan), 64.97 (CH<sub>2</sub>-O), 64.28 (CH<sub>2</sub>-O), 48.49 (NH-CH), 35.56, 31.26, 29.03, 28.99, 28.94, 28.88, 28.68, 28.62, 28.58, 27.96, 27.89, 25.24, 25.18, 22.02, 13.46.

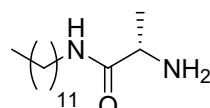
#### Amino-amides.

##### *N-hexyl-L-alaninamide (12a)*



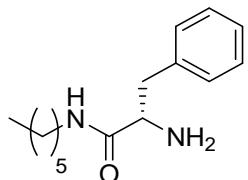
**95%**; white solid; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.75 (t, *J* = 5.9 Hz, 1H, NH), 3.21 (q, *J* = 6.8 Hz, 1H, CH), 3.04 (dt, *J* = 7.8, 6.0 Hz, 2H, CH<sub>2</sub>-NH), 2.01 (s, 2H, NH<sub>2</sub>), 1.39 (p, *J* = 7.3 Hz, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.25 (m, 6H, (CH<sub>2</sub>)<sub>3</sub>), 1.10 (d, *J* = 6.9 Hz, 3H, CH<sub>3</sub>-CH), 0.84 (t, 3H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 175.44 (C=O), 50.25 (CH), 38.21 (CH<sub>2</sub>-NH), 30.97, 29.12, 26.00, 22.02, 21.68, 13.81.

##### *N-dodecyl-L-alaninamide (12d)*



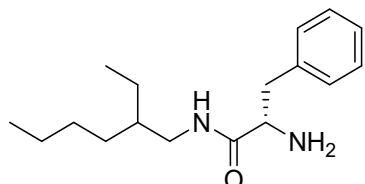
**60%**; white solid; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.77 (t, *J* = 5.9 Hz, 1H, NH), 3.25 (q, *J* = 6.9 Hz, 1H, CH), 3.03 (m 2H, CH<sub>2</sub>-NH), 2.63 (s, 2H, NH<sub>2</sub>), 1.38 (m, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.26 (m, 18H, (CH<sub>2</sub>)<sub>9</sub>), 1.11 (d, *J* = 6.9 Hz, 3H, CH<sub>3</sub>-CH), 0.84 (t, 3H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 174.90 (C=O), 50.07 (CH), 38.22 (CH<sub>2</sub>-NH), 31.26, 29.11, 29.01, 28.98, 28.96, 28.71, 28.67, 26.31, 22.05, 21.34, 13.90.

*N-hexyl-L-phenylalaninamide (13a)*



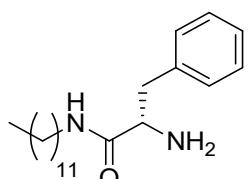
**98%**; white solid; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.75 (t, *J* = 5.7 Hz, 1H, NH), 7.34-7.12 (m, 5H, Ar-H), 3.36 (dd, *J* = 7.8, 5.5 Hz, 1H, CH), 3.02 (h, *J* = 6.4 Hz, 2H, CH<sub>2</sub>-NH), 2.93-2.56 (m, 2H, CH<sub>2</sub>-Ph), 1.85 (s, 2H, NH<sub>2</sub>), 1.33 (m, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.21 (m, 6H, (CH<sub>2</sub>)<sub>3</sub>), 0.84 (t, *J* = 6.8 Hz, 3H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 173.86 (C=O amide), 138.68 (C(i) Ph), 129.25 (CH(m) Ph), 128.00 (CH(o) Ph), 126.01 (CH(p) Ph), 56.23 (NH<sub>2</sub>-CH), 41.22 (CH<sub>2</sub>-NH), 38.29 (CH<sub>2</sub>-Ph), 30.97, 29.02, 25.99, 22.01, 13.89.

*N-2-ethylhexyl-L-phenylalaninamide (13b)*



**99%**; white solid; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.88 (t, *J* = 5.8 Hz, 1H, NH), 7.36-7.09 (m, 5H, Ar-H), 3.63-3.56 (m, 1H, CH), 3.13-2.68 (m, 4H, CH<sub>2</sub>-Ph + NH-CH<sub>2</sub>), 1.42-0.96 (m, 9H, CH-CH<sub>2</sub> + (CH<sub>2</sub>)<sub>3</sub>), 0.82 (m, 6H, CH<sub>3</sub>).

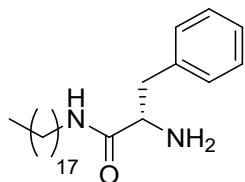
*N-dodecyl-L-phenylalaninamide (13d)*



**98%**; white solid; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.74 (t, *J* = 5.8 Hz, 1H, NH), 7.31-7.13 (m, 5H, Ar-H), 3.35 (dd, *J* = 7.9, 5.4 Hz, 1H, CH), 3.01 (m, 2H, CH<sub>2</sub>-NH), 2.95-2.54 (m, 2H, CH<sub>2</sub>-Ph), 1.90

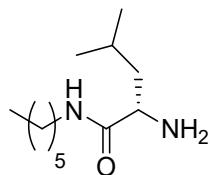
(s, 2H, NH<sub>2</sub>), 1.31 (m, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.24 (m, 18H, (CH<sub>2</sub>)<sub>9</sub>), 0.84 (t, 3H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 173.90 (C=O amide), 138.72 (**C(i)** Ph), 129.23 (**CH(m)** Ph), 127.97 (**CH(o)** Ph), 125.97 (**CH(p)** Ph), 56.25 (NH<sub>2</sub>-CH), 41.26 (CH<sub>2</sub>-NH), 38.26 (CH<sub>2</sub>-Ph), 31.27, 29.05, 29.02, 28.97, 28.94, 28.73, 28.68, 26.30, 22.06, 13.91.

*N-octadecyl-L-phenylalaninamide (13f)*



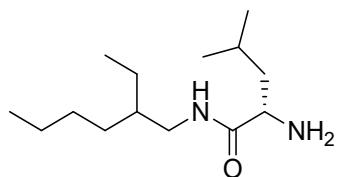
**91%**; white solid; <sup>1</sup>H NMR (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 7.57 (t, *J* = 5.2 Hz, 1H, NH), 7.33-7.09 (m, 5H, Ar-H), 3.43 (dd, *J* = 8.4, 4.9 Hz, 1H, CH), 3.09 (m, 2H, CH<sub>2</sub>-NH), 3.04-2.61 (m, 2H, CH<sub>2</sub>-Ph), 1.39 (m, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.24 (m, 30H, (CH<sub>2</sub>)<sub>15</sub>), 0.85 (t, 3H, CH<sub>3</sub>). <sup>13</sup>C NMR (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.15 (C=O amide), 136.28 (**C(i)** Ph), 129.50 (**CH(m)** Ph), 128.34 (**CH(o)** Ph), 126.78 (**CH(p)** Ph), 54.91 (NH<sub>2</sub>-CH), 39.00 (CH<sub>2</sub>-NH), 38.73 (CH<sub>2</sub>-Ph), 31.60, 29.37, 29.35, 29.31, 29.06, 29.01, 26.65, 22.39, 13.86.

*N-hexyl-L-leucinamide (14a)*



**77%**; white solid; <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.79 (t, *J* = 5.9 Hz, 1H, NH), 3.17-2.91 (m, 3H, CH + NH-CH<sub>2</sub>), 1.69 (m, 1H, CH), 1.37 (m, 2H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.33-1.12 (m, 8H, CH-CH<sub>2</sub> + (CH<sub>2</sub>)<sub>3</sub>), 0.86 (m, 9H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>), δ (ppm): 175.40 (C=O amide), 53.18 (NH<sub>2</sub>-CH), 44.59 (CH-CH<sub>2</sub>), 38.18 (NH-CH<sub>2</sub>), 30.95, 29.10, 25.99, 24.15, 23.13, 22.03, 21.92, 13.85.

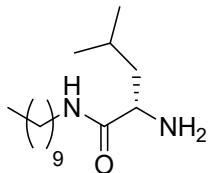
*N-2-ethylhexyl-L-leucinamide (14b)*



**88%**; white solid; <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 7.85 (t, *J* = 5.9 Hz, 1H, NH), 3.24 (dd, *J* = 8.3, 5.9 Hz, 1H, CH), 3.13-2.88 (m, 2H, NH-CH<sub>2</sub>), 1.67 (m, 1H, CH), 1.47-1.12 (m, 11H, CH-CH<sub>2</sub> +

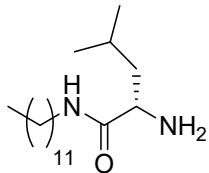
$(\text{CH}_2)_4$ , 0.85 (m, 12H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (75 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 174.24 ( $\text{C}=\text{O}$  amide), 52.77 ( $\text{NH}_2\text{-CH}$ ), 43.81 ( $\text{CH}\text{-CH}_2$ ), 41.16 + 41.13 ( $\text{NH}\text{-CH}_2$  R+S), 38.85, 38.82, 30.33, 28.36, 28.28, 24.09, 23.62, 22.95, 22.47, 21.99, 13.91, 10.72, 10.67.

*N-decyl-L-leucinamide (14c)*



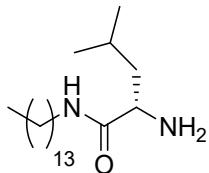
**98%**; white solid;  $^1\text{H}$  NMR (400 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 7.78 (t,  $J = 5.8$  Hz, 1H, NH), 3.15-2.92 (m, 3H,  $\text{CH-NH}_2 + \text{NH-CH}_2$ ), 1.67 (m, 1H, CH), 1.37 (m, 2H,  $\text{CH}_2\text{-CH}_2\text{-NH}$ ), 1.31-1.14 (m, 16H,  $\text{CH-CH}_2 + (\text{CH}_2)_7$ ), 0.85 (m, 9H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (101 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 174.91 ( $\text{C}=\text{O}$  amide), 53.03 ( $\text{NH}_2\text{-CH}$ ), 44.27 ( $\text{CH}\text{-CH}_2$ ), 38.23 ( $\text{NH}\text{-CH}_2$ ), 31.31, 29.10, 29.01, 28.94, 28.73, 28.72, 28.70, 26.33, 24.14, 23.07, 22.10, 21.97, 13.94.

*N-dodecyl-L-leucinamide (14d)*



**91%**; white solid;  $^1\text{H}$  NMR (400 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 7.81 (t,  $J = 5.8$  Hz, 1H, NH), 3.17-2.94 (m, 3H,  $\text{CH} + \text{NH-CH}_2$ ), 1.67 (m, 1H, CH), 1.38 (m, 2H,  $\text{CH}_2\text{-CH}_2\text{-NH}$ ), 1.32-1.16 (m, 20H,  $\text{CH-CH}_2 + (\text{CH}_2)_9$ ), 0.85 (m, 9H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (101 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 175.08 ( $\text{C}=\text{O}$  amide), 53.08 ( $\text{NH}_2\text{-CH}$ ), 44.40 ( $\text{CH}\text{-CH}_2$ ), 38.18 ( $\text{NH}\text{-CH}_2$ ), 31.29, 29.10, 29.05, 29.01, 28.99, 28.96, 28.72, 28.70, 26.32, 24.13, 23.08, 22.08, 21.93, 13.91.

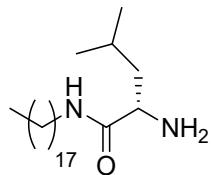
*N-tetradecyl-L-leucinamide (14e)*



**72%**; white solid;  $^1\text{H}$  NMR (300 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 8.49 (t,  $J = 5.6$  Hz, 1H, NH), 7.59 (s, 2H,  $\text{NH}_2$ ), 3.61 (t,  $J = 7.1$  Hz, 1H,  $\text{CH-NH}$ ), 3.08 (m, 2H,  $\text{NH-CH}_2$ ), 1.60 (m, 1H, CH), 1.50 (m, 2H,  $\text{CH}_2\text{-CH}_2\text{-NH}$ ), 1.41 (m, 2H,  $\text{CH-CH}_2$ ), 1.23 (m, 22H,  $(\text{CH}_2)_{11}$ ), 0.87 (m, 9H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (101 MHz, DMSO-d<sub>6</sub>),

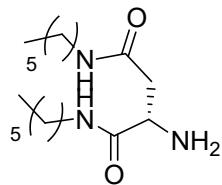
$\delta$  (ppm): 169.44 (C=O amide), 51.30 (NH<sub>2</sub>-CH), 40.82 (CH-CH<sub>2</sub>), 38.57 (NH-CH<sub>2</sub>), 31.30, 29.06, 29.02, 29.00, 28.98, 28.78, 28.71, 26.32, 23.80, 22.44, 22.30, 22.10, 13.95.

*N-octadecyl-L-leucinamide (14f)*



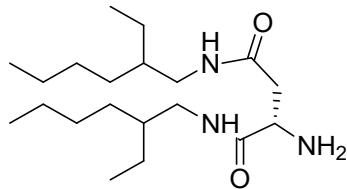
**80%**; white solid; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>),  $\delta$  (ppm): 7.58 (t,  $J$  = 7.0 Hz, 1H, NH), 3.24-3.02 (m, 3H, CH + NH-CH<sub>2</sub>), 1.70 (m, 1H, CH-(CH<sub>3</sub>)<sub>2</sub>), 1.61-1.35 (m, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH + CH-CH<sub>2</sub>), 1.24 (m, 30H, (CH<sub>2</sub>)<sub>15</sub>), 0.89 (m, 9H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>),  $\delta$  (ppm): 175.05 (C=O amide), 53.01 (NH<sub>2</sub>-CH), 44.08 (CH-CH<sub>2</sub>), 38.19 (NH-CH<sub>2</sub>), 31.22, 29.05, 28.98, 28.92, 28.68, 28.64, 26.30, 24.08, 22.72, 22.00, 21.18, 13.46.

*N-dihexyl-L-aspartamide (15a)*



**76%**; white solid; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 7.90 (q,  $J$  = 5.8 Hz, 2H, NH), 3.52 (dd,  $J$  = 8.8, 4.4 Hz, 1H, CH), 3.16-2.91 (m, 4H, NH-CH<sub>2</sub>), 2.46-2.12 (m, 2H, CH-CH<sub>2</sub>), 1.28 (m, 16H, (CH<sub>2</sub>)<sub>4</sub>), 0.85 (m, 6H, CH<sub>3</sub>).

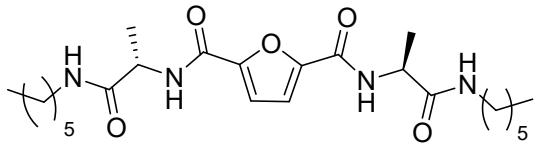
*N-bis(2-ethylhexyl)-L-aspartamide (15b)*



**49%**; white solid; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 7.87 (t,  $J$  = 5.8 Hz, 1H, NH), 7.79 (t,  $J$  = 6.0 Hz, 1H, NH), 3.49 (dd,  $J$  = 9.0, 4.2 Hz, 1H, CH), 3.11-2.87 (m, 4H, NH-CH<sub>2</sub>), 2.45-2.13 (m, 2H, NH-CH-CH<sub>2</sub>), 1.48-1.09 (m, 18H, CH-CH<sub>2</sub> + (CH<sub>2</sub>)<sub>3</sub>), 0.84 (m, 12H, CH<sub>3</sub>). **<sup>13</sup>C NMR** (75 MHz, DMSO-d<sub>6</sub>),  $\delta$  (ppm): 173.80 (C=O amide), 170.36 (C=O amide), 52.13 (NH<sub>2</sub>-CH), 41.25 (NH-CH<sub>2</sub>), 41.15 (NH-CH<sub>2</sub>), 40.73, 38.79, 30.35, 30.30, 28.34, 28.31, 23.59, 23.54, 22.47, 13.92, 10.67.

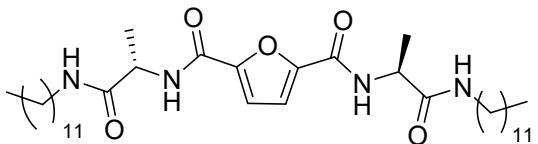
**Di(amido-amides).**

*N<sup>2</sup>, N<sup>5</sup>-bis(hexyl L-alaninamide)-furan-2,5-dicarboxamide (16a)*



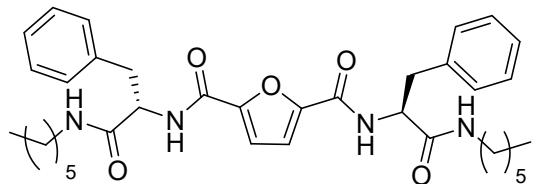
**80%**; white solid; **T<sub>f</sub>**: 224°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.38 (d, *J* = 8.0 Hz, 2H, NH), 7.77 (t, *J* = 5.8 Hz, 2H, NH), 7.10 (s, 2H, CH furan), 4.54 (p, *J* = 7.2 Hz, 2H, CH-NH), 3.09 (m, 4H, CH<sub>2</sub>-NH), 1.43 (m, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.38 (d, *J* = 7.0 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.25 (m, 12H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>3</sub>), 0.84 (t, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.38 (C=O amide), 156.70 (C=O amide furan), 147.98 (C-C=O amide), 114.52 (CH furan), 48.09 (NH-CH), 38.62 (CH<sub>2</sub>-NH), 30.88, 28.86, 25.94, 21.94, 17.98, 13.42; **HRMS (ESI, m/z)**: 487.289 [M + Na]<sup>+</sup>, 487.2891 calculated for C<sub>24</sub>H<sub>40</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(dodecyl L-alaninamide)-furan-2,5-dicarboxamide (16d)*



**17%**; white solid; **T<sub>f</sub>**: 202,3°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.43 (d, *J* = 8.0 Hz, 2H, NH), 7.87 (t, *J* = 5.8 Hz, 2H, NH), 7.13 (s, 2H, CH furan), 4.52 (p, *J* = 7.3 Hz, 2H, CH-NH), 3.08 (m, 4H, CH<sub>2</sub>-NH), 1.43 (m, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.37 (d, *J* = 7.1 Hz, 6H, CH<sub>3</sub>-CH-NH), 1.24 (m, 36H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.85 (t, *J* = 6.5 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.39 (C=O amide), 156.68 (C=O amide furan), 148.01 (C-C=O amide), 114.66 (CH furan), 48.10 (NH-CH), 38.55 (CH<sub>2</sub>-NH), 31.24, 28.99, 28.94, 28.68, 28.65, 26.28, 22.02, 18.16, 13.66; **HRMS (ESI, m/z)**: 655.477 [M + Na]<sup>+</sup>, 655.4769 calculated for C<sub>36</sub>H<sub>64</sub>N<sub>4</sub>O<sub>5</sub>Na.

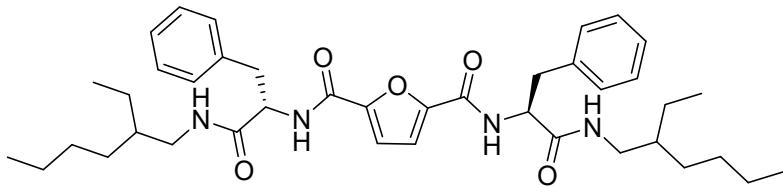
*N<sup>2</sup>, N<sup>5</sup>-bis(hexyl L-phenylalaninamide)-furan-2,5-dicarboxamide (17a)*



**37%**; white solid; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.80 (d, *J* = 8.8 Hz, 2H, NH), 8.08 (t, *J* = 5.7 Hz, 2H, NH), 7.35-7.10 (m, 10H, Ar-H), 7.08 (s, 2H, CH furan), 4.69 (td, *J* = 8.9, 5.8 Hz, 2H, CH-NH), 3.18-2.96 (m, 8H, CH<sub>2</sub>-NH + CH<sub>2</sub>-Ph), 1.38 (p, *J* = 6.8 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.22 (m, 12H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>3</sub>), 0.84 (t, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.39

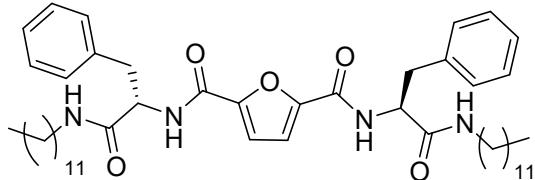
(C=O amide), 156.78 (C=O amide furan), 147.90 (C-C=O amide), 137.80 (**C(i)** Ph), 129.04 (**CH(m)** Ph), 127.88 (**CH(o)** Ph), 126.08 (**CH(p)** Ph), 114.64 (CH furan), 54.45 (NH-CH), 37.75 (CH<sub>2</sub>-NH), 30.94, 28.83, 25.99, 21.99, 13.68; **HRMS (ESI, m/z):** 639.3516 [M + Na]<sup>+</sup>, 639.3517 calculated for C<sub>36</sub>H<sub>48</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(2-ethylhexyl L-phenylalaninamide)-furan-2,5-dicarboxamide (17b)*



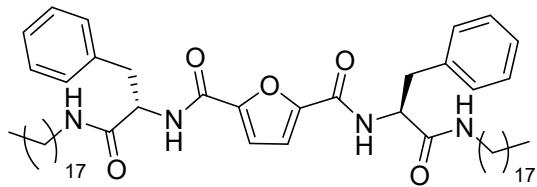
**21%**; white solid; **<sup>1</sup>H NMR** (400 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 9.09 (d, J = 8.9 Hz, 2H, NH), 8.02 (t, J = 5.8 Hz, 2H, NH), 7.38-7.09 (m, 10H, Ar-H), 7.03 (s, 2H, CH furan), 4.74 (td, J = 9.0, 5.9 Hz, 2H, CH-NH), 3.19-2.95 (m, 8H, CH<sub>2</sub>-NH + CH<sub>2</sub>-Ph), 1.42 (p, J = 5.9 Hz, 2H, CH-CH<sub>2</sub>-NH), 1.22 (m, 16H, CH<sub>2</sub>-CH<sub>3</sub> + (CH<sub>2</sub>)<sub>3</sub>), 0.82 (t, J = 7.0 Hz, 12H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.70 (C=O amide), 156.66 (C=O amide furan), 147.83 (C-C=O amide), 137.84 (**C(i)** Ph), 128.93 (**CH(m)** Ph), 127.72 (**CH(o)** Ph), 125.90 (**CH(p)** Ph), 114.38 (CH furan), 54.79 (NH-CH), 41.57, 38.64, 37.68, 30.27, 28.28, 28.22, 23.52, 22.39, 13.58, 10.39, 10.34; **HRMS (ESI, m/z):** 695.4143 [M + Na]<sup>+</sup>, 695.4143 calculated for C<sub>40</sub>H<sub>56</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(dodecyl L-phenylalaninamide)-furan-2,5-dicarboxamide (17d)*



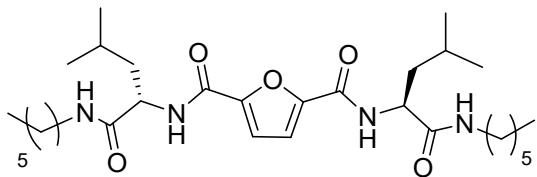
**39%**; white solid; **T<sub>f</sub>:** 181.3°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.57 (d, J = 8.6 Hz, 2H, NH), 8.01 (t, J = 5.7 Hz, 2H, NH), 7.35-7.11 (m, 10H, Ar-H), 7.09 (s, 2H, CH furan), 4.71 (td, J = 8.8, 5.7 Hz, 2H, CH-NH), 3.18-2.93 (m, 8H, CH<sub>2</sub>-NH + CH<sub>2</sub>-Ph), 1.36 (t, J = 6.8 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.22 (m, 36H, CH<sub>3</sub>-(CH<sub>2</sub>)<sub>9</sub>), 0.85 (t, J = 6.5 Hz, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.21 (C=O amide), 156.79 (C=O amide furan), 147.92 (C-C=O amide), 137.68 (**C(i)** Ph), 129.02 (**CH(m)** Ph), 127.87 (**CH(o)** Ph), 126.07 (**CH(p)** Ph), 114.64 (CH furan), 54.49 (NH-CH), 38.62 (**CH<sub>2</sub>**-Ph), 37.84 (**CH<sub>2</sub>-NH**), 31.27, 29.03, 28.99, 28.95, 28.88, 28.72, 28.69, 26.31, 22.06, 13.73; **HRMS (ESI, m/z):** 807.5397 [M + Na]<sup>+</sup>, 807.5395 calculated for C<sub>48</sub>H<sub>72</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(octadecyl L-phenylalaninamide)-furan-2,5-dicarboxamide (17f)*



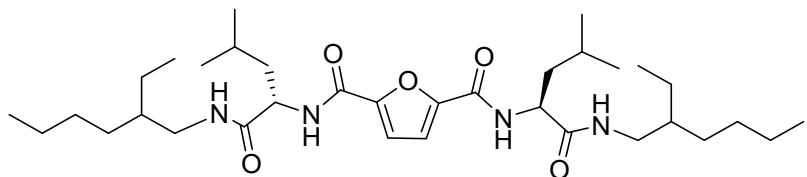
**61%**; white solid; **T<sub>f</sub>**: 167,9°C; **<sup>1</sup>H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.60 (d, *J* = 8.7 Hz, 2H, NH (**S,S**)), 8.53 (d, *J* = 9.4 Hz, NH (**R,S**)), 7.88 (t, *J* = 5.7 Hz, 2H, NH), 7.33-7.11 (m, 10H, Ar-H), 7.04 (s, 2H, CH furan (**S,S**)), 7.00 (s, CH furan (**R,S**)), 4.74 (td, *J* = 8.7, 6.0 Hz, 2H, CH-NH), 3.35-2.91 (m, 8H, CH<sub>2</sub>-NH + CH<sub>2</sub>-Ph), 1.36 (t, *J* = 6.8 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.42-1.21 (m, 64H, (CH<sub>2</sub>)<sub>16</sub>), 0.87 (t, 6H, CH<sub>3</sub>-CH<sub>2</sub>); **<sup>13</sup>C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 170.11 (C=O amide), 156.69 (C=O amide furan), 147.85 (C-C=O amide), 137.50 (**C(i)** Ph), 128.87 (**CH(m)** Ph), 127.66 (**CH(o)** Ph), 125.86 (**CH(p)** Ph), 114.38 (CH furan), 54.16 (NH-CH), 38.63 (CH<sub>2</sub>-Ph), 37.82 (CH<sub>2</sub>-NH), 31.23, 28.99, 28.95, 28.92, 28.81, 28.69, 28.65, 26.30, 22.01, 13.50; **HRMS (ESI, m/z)**: 975.7275 [M + Na]<sup>+</sup>, 975.7273 calculated for C<sub>60</sub>H<sub>96</sub>N<sub>4</sub>O<sub>5</sub>Na.

#### *N<sup>2</sup>, N<sup>5</sup>-bis(hexyl L-leucinamide)-furan-2,5-dicarboxamide (18a)*



**52%**; white solid; **T<sub>f</sub>**: 199,4°C; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.45 (d, *J* = 8.7 Hz, 2H, NH), 8.08 (t, *J* = 5.6 Hz, 2H, NH), 7.21 (s, 2H, CH furan), 4.53 (td, *J* = 13.9, 6.9 Hz, 2H, CH-NH), 3.05 (m, 4H, CH<sub>2</sub>-NH), 1.60 (m, 6H, CH<sub>2</sub>-CH-(CH<sub>3</sub>)<sub>2</sub>), 1.39 (p, *J* = 7.0 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.24 (m, 12H, (CH<sub>2</sub>)<sub>3</sub>), 0.87 (m, 18H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 171.20 (C=O amide), 156.92 (C=O amide furan), 148.06 (C-C=O amide), 115.07 (CH furan), 50.95 (NH-CH), 40.99 (NH-CH-CH<sub>2</sub>), 38.50 (CH<sub>2</sub>-NH), 30.90, 28.90, 25.93, 24.40, 22.88, 22.02, 21.64, 13.83; **HRMS (ESI, m/z)**: 571.3831 [M + Na]<sup>+</sup>, 571.3830 calculated for C<sub>30</sub>H<sub>52</sub>N<sub>4</sub>O<sub>5</sub>Na.

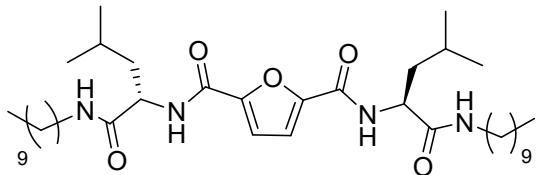
#### *N<sup>2</sup>, N<sup>5</sup>-bis(2-ethylhexyl L-leucinamide)-furan-2,5-dicarboxamide (18b)*



**46%**; white solid; **T<sub>f</sub>**: 182,3°C; **<sup>1</sup>H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.44 (d, *J* = 8.7 Hz, 2H, NH), 8.01 (t, *J* = 5.8 Hz, 2H, NH), 7.21 (s, 2H, CH furan), 4.57 (td, *J* = 9.0, 4.4 Hz, 2H, CH-NH), 3.16-2.85 (m,

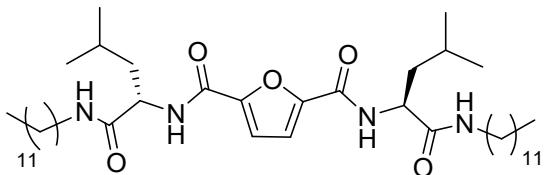
4H, **CH<sub>2</sub>-NH**), 1.61 (m, 6H, **CH<sub>2</sub>-CH-(CH<sub>3</sub>)<sub>2</sub>**), 1.38 (p, *J* = 6.1 Hz, 2H, **CH-CH<sub>2</sub>-NH**), 1.22 (m, 16H, **CH<sub>2</sub>-CH<sub>3</sub> + (CH<sub>2</sub>)<sub>3</sub>**), 0.86 (m, 24H, **CH<sub>3</sub>**); <sup>13</sup>**C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 171.40 (**C=O amide**), 156.88 (**C=O amide furan**), 148.08 (**C-C=O amide**), 115.06 (**CH furan**), 50.98 (**NH-CH**), 41.40 + 41.36 (**CH<sub>2</sub>-NH**), 41.05 (**NH-CH-CH<sub>2</sub>**), 38.79 + 38.76 (**CH-CH<sub>2</sub>-NH, R + S**), 30.28, 28.35 + 28.24 (**CH<sub>2</sub> ethyl, R + S**), 24.41, 23.65, 23.60, 22.85, 22.46, 21.70, 13.87, 10.77 + 10.64 (**CH<sub>3</sub> ethyl, R + S**); **HRMS (ESI, m/z)**: 627.4455 [M + Na]<sup>+</sup>, 627.4456 calculated for C<sub>34</sub>H<sub>60</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(decyl L-leucinamide)-furan-2,5-dicarboxamide (18c)*



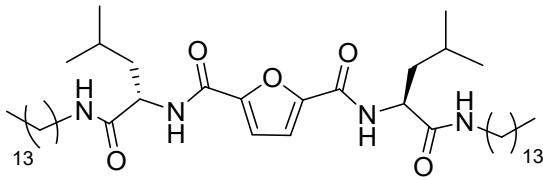
**36%**; white solid; **Tf**: 175,4°C; <sup>1</sup>**H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.44 (d, *J* = 8.7 Hz, 2H, **NH**), 8.08 (t, *J* = 5.6 Hz, 2H, **NH**), 7.20 (s, 2H, **CH furan**), 4.52 (m, 2H, **CH-NH**), 3.03 (m, 4H, **CH<sub>2</sub>-NH**), 1.61 (m, 6H, **CH<sub>2</sub>-CH-(CH<sub>3</sub>)<sub>2</sub>**), 1.39 (p, *J* = 6.8 Hz, 4H, **CH<sub>2</sub>-CH<sub>2</sub>-NH**), 1.22 (m, 28H, **(CH<sub>2</sub>)<sub>7</sub>**), 0.88 (m, 18H, **CH<sub>3</sub>**); <sup>13</sup>**C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 171.20 (**C=O amide**), 156.89 (**C=O amide furan**), 148.05 (**C-C=O amide**), 115.03 (**CH furan**), 51.01 (**NH-CH**), 41.00 (**NH-CH-CH<sub>2</sub>**), 38.46 (**CH<sub>2</sub>-NH**), 31.26, 28.96, 28.89, 28.67, 28.65, 26.23, 24.38, 22.85, 22.06, 21.64, 13.91; **HRMS (ESI, m/z)**: 683.5082 [M + Na]<sup>+</sup>, 683.5082 calculated for C<sub>38</sub>H<sub>68</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(dodecyl L-leucinamide)-furan-2,5-dicarboxamide (18d)*



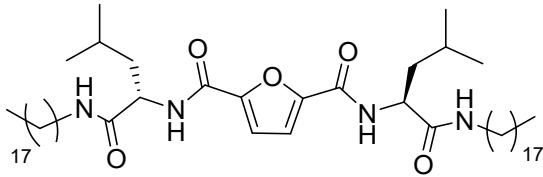
**57%**; white solid; **Tf**: 156,5°C; <sup>1</sup>**H NMR** (400 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.54 (d, *J* = 8.8 Hz, 2H, **NH**), 8.10 (t, *J* = 5.6 Hz, 2H, **NH**), 7.19 (s, 2H, **CH furan**), 4.51 (td, *J* = 9.2, 4.8 Hz, 2H, **CH-NH**), 3.03 (m, 4H, **CH<sub>2</sub>-NH**), 1.62 (m, 6H, **CH<sub>2</sub>-CH-(CH<sub>3</sub>)<sub>2</sub>**), 1.38 (p, *J* = 6.7 Hz, 4H, **CH<sub>2</sub>-CH<sub>2</sub>-NH**), 1.22 (m, 36H, **(CH<sub>2</sub>)<sub>9</sub>**), 0.87 (m, 18H, **CH<sub>3</sub>**); <sup>13</sup>**C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 171.24 (**C=O amide**), 156.87 (**C=O amide furan**), 148.05 (**C-C=O amide**), 115.01 (**CH furan**), 51.13 (**NH-CH**), 40.99 (**NH-CH-CH<sub>2</sub>**), 38.46 (**CH<sub>2</sub>-NH**), 31.28, 29.03, 28.99, 28.96, 28.94, 28.88, 28.69, 28.66, 26.24, 24.39, 22.85, 22.08, 21.62, 13.92; **HRMS (ESI, m/z)**: 739.571 [M + Na]<sup>+</sup>, 739.5708 calculated for C<sub>42</sub>H<sub>76</sub>N<sub>4</sub>O<sub>5</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(tetradecyl L-leucinamide)-furan-2,5-dicarboxamide (18e)*



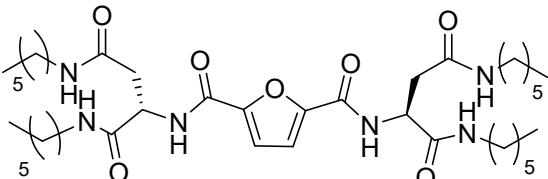
**46%**; white solid; Tf: 168,8°C; **1H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.72 (d, *J* = 8.9 Hz, 2H, NH), 8.04 (t, *J* = 5.6 Hz, 2H, NH), 7.12 (s, 2H, CH furan), 4.55 (td, *J* = 9.3, 4.8 Hz, 2H, CH-NH), 3.07 (m, 4H, CH<sub>2</sub>-NH), 1.66 (m, 6H, CH<sub>2</sub>-CH-(CH<sub>3</sub>)<sub>2</sub>), 1.42 (p, *J* = 6.8 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.22 (m, 44H, (CH<sub>2</sub>)<sub>11</sub>), 0.88 (m, 18H, CH<sub>3</sub>); **13C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.31 (C=O amide), 156.76 (C=O amide furan), 148.00 (C-C=O amide), 114.59 (CH furan), 51.35 (NH-CH), 41.00 (NH-CH-CH<sub>2</sub>), 38.52 (CH<sub>2</sub>-NH), 31.23, 28.99, 28.95, 28.92, 28.81, 28.65, 26.28, 24.28, 22.57, 22.02, 21.41, 13.63; **HRMS (ESI, m/z)**: 795.6335 [M + Na]<sup>+</sup>, 795.6334 calculated for C<sub>46</sub>H<sub>84</sub>N<sub>4</sub>O<sub>5</sub>Na.

#### *N<sup>2</sup>, N<sup>5</sup>-bis(octadecyl L-leucinamide)-furan-2,5-dicarboxamide (18f)*



**67%**; white solid; Tf: 163,6°C; **1H NMR** (300 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 8.30 (d, *J* = 8.8 Hz, 2H, NH), 7.72 (t, *J* = 5.7 Hz, 2H, NH), 7.10 (s, 2H, CH furan), 4.60 (q, *J* = 7.5 Hz, 2H, CH-NH), 3.12 (m, 4H, CH<sub>2</sub>-NH), 1.65 (m, 6H, CH<sub>2</sub>-CH-(CH<sub>3</sub>)<sub>2</sub>), 1.44 (p, *J* = 6.5 Hz, 4H, CH<sub>2</sub>-CH<sub>2</sub>-NH), 1.24 (m, 64H, (CH<sub>2</sub>)<sub>16</sub>), 0.89 (m, 18H, CH<sub>3</sub>); **13C NMR** (75 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 171.11 (C=O amide), 156.83 (C=O amide furan), 147.97 (C-C=O amide), 114.51 (CH furan), 50.91 (NH-CH), 40.96 (NH-CH-CH<sub>2</sub>), 38.59 (CH<sub>2</sub>-NH), 31.24, 29.00, 28.95, 28.93, 28.84, 28.66, 26.29, 24.23, 22.37, 22.01, 21.35, 13.44; **HRMS (ESI, m/z)**: 907.7589 [M + Na]<sup>+</sup>, 907.7586 calculated for C<sub>54</sub>H<sub>100</sub>N<sub>4</sub>O<sub>5</sub>Na.

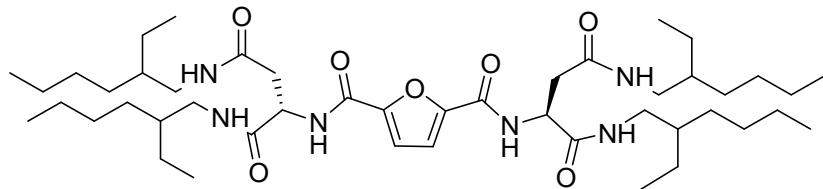
#### *N<sup>2</sup>, N<sup>5</sup>-bis(dihexyl L-aspartamide)-furan-2,5-dicarboxamide (19a)*



**27%**; white solid; **1H NMR** (300 MHz, DMSO-d<sub>6</sub>, 373K), δ (ppm): 8.46 (d, *J* = 8.2 Hz, 2H, NH), 7.86 (t, *J* = 5.7 Hz, 2H, NH), 7.76 (t, *J* = 5.6 Hz, 2H, NH), 7.18 (s, 2H, CH furan), 4.72 (td, *J* = 8.0, 5.9 Hz, 2H, CH-NH), 3.02 (m, 8H, CH<sub>2</sub>-NH), 2.59 (m, 2H, CH<sub>2</sub>-C=O), 1.46-1.10 (m, 32H, (CH<sub>2</sub>)<sub>4</sub>), 0.83 (m, 12H, CH<sub>3</sub>); **13C NMR** (101 MHz, CD<sub>2</sub>Cl<sub>2</sub>-d<sub>2</sub> / DMSO-d<sub>6</sub>), δ (ppm): 169.84 (C=O amide), 169.26 (C=O amide),

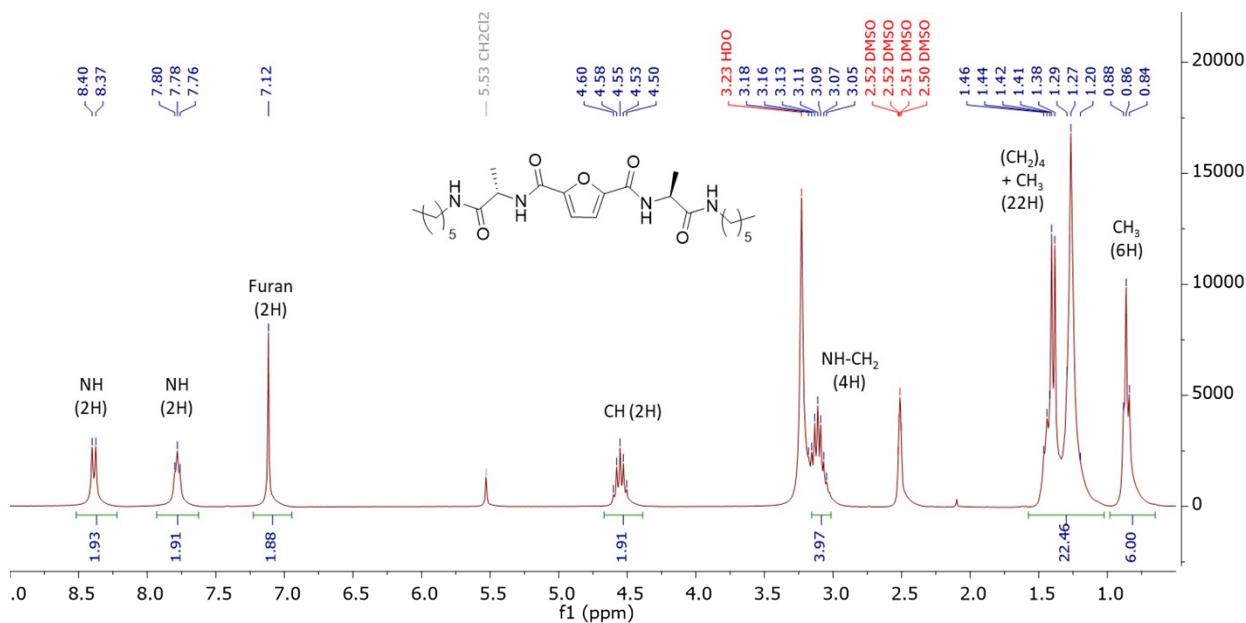
156.76 (**C=O** amide furan), 147.97 (**C-C=O** amide), 114.47 (**CH** furan), 49.86 (**NH-CH**), 38.77 (**CH<sub>2</sub>-NH**), 38.65 (**CH<sub>2</sub>-NH**), 37.69, 30.91, 30.88, 28.90, 28.83, 26.00, 25.92, 21.93, 13.40; **HRMS (ESI, m/z)**: 741.4886 [M + Na]<sup>+</sup>, 741.4885 calculated for C<sub>38</sub>H<sub>66</sub>N<sub>6</sub>O<sub>7</sub>Na.

*N<sup>2</sup>, N<sup>5</sup>-bis(bis(2-ethylhexyl) L-aspartamide)-furan-2,5-dicarboxamide (19b)*

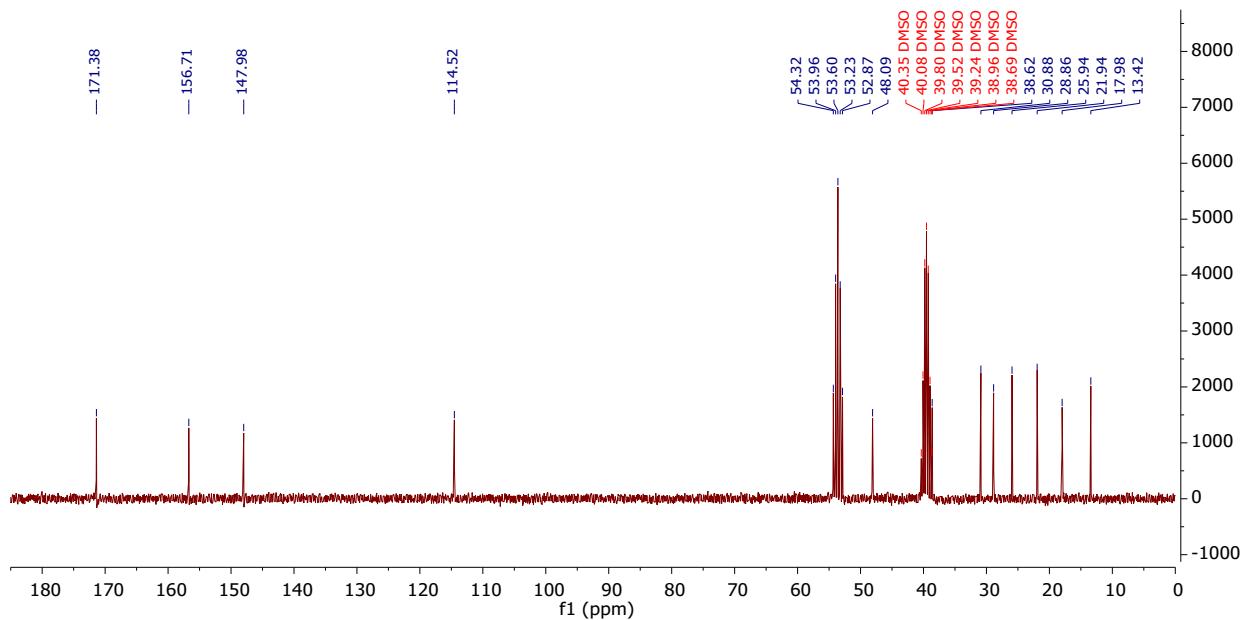


**18%**; white solid; **T<sub>f</sub>**: 172.8-200.6°C; **<sup>1</sup>H NMR** (300 MHz, DMSO-d<sub>6</sub>), δ (ppm): 8.50 (d, *J* = 8.2 Hz, 2H, NH), 7.87 (t, *J* = 5.2 Hz, 2H, NH), 7.73 (t, *J* = 4.7 Hz, 2H, NH), 7.19 (s, 2H, CH furan), 4.75 (q, *J* = 7.3 Hz, 2H, CH-NH), 2.97 (m, 8H, CH<sub>2</sub>-NH), 2.59 (d, *J* = 6.9 Hz, 4H, CH<sub>2</sub>-C=O), 1.46-1.01 (m, 36H, CH<sub>2</sub>-CH-(CH<sub>2</sub>)<sub>3</sub>), 0.81 (m, 24H, CH<sub>3</sub>); **<sup>13</sup>C NMR** (101 MHz, DMSO-d<sub>6</sub>), δ (ppm): 170.32 (**C=O** amide), 169.14 (**C=O** amide), 157.85 (**C=O** amide furan), 148.17 (**C-C=O** amide), 114.80 (**CH** furan), 50.11 (**NH-CH**), 41.56 (**CH<sub>2</sub>-NH**), 41.42 (**CH<sub>2</sub>-NH**), 38.76 + 38.74 (**CH-CH<sub>2</sub>-NH, R + S**), 38.70, 37.73, 28.32 + 28.27 (**CH<sub>2</sub>** ethyl, **R + S**), 23.50, 23.48, 23.43, 22.47, 22.44, 13.92, 10.68, 10.62 + 10.60 (**CH<sub>3</sub>** ethyl, **R + S**), 10.55; **HRMS (ESI, m/z)**: 853.6168 [M + Na]<sup>+</sup>, 853.6137 calculated for C<sub>46</sub>H<sub>82</sub>N<sub>6</sub>O<sub>7</sub>Na.

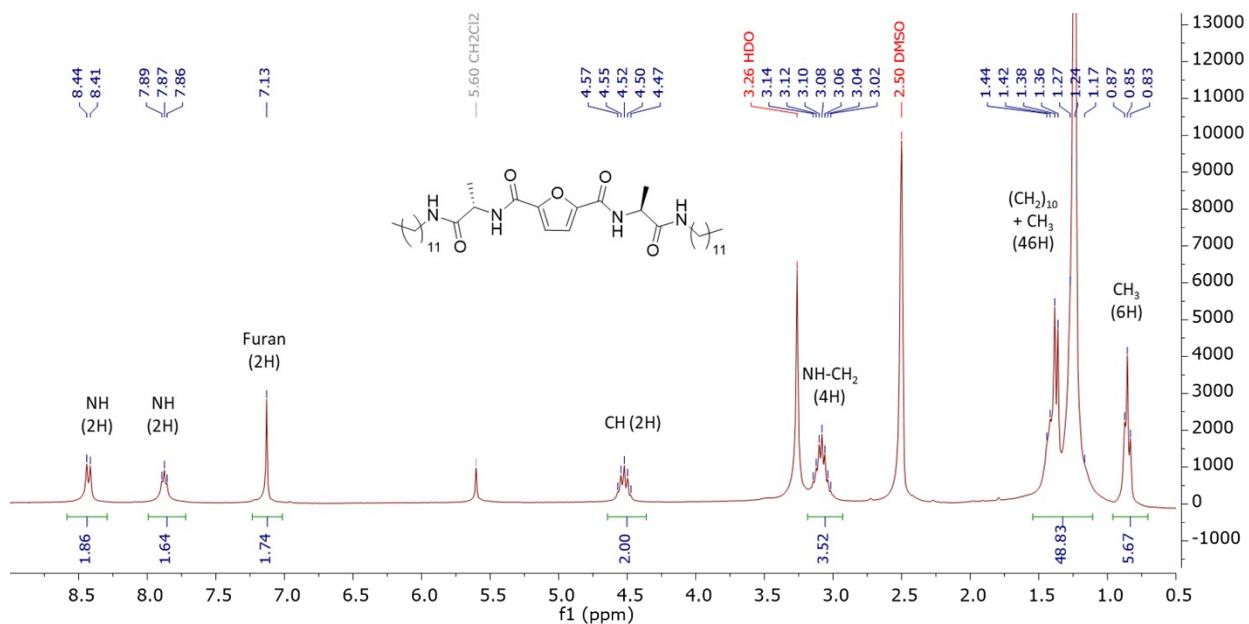
## Analyses.



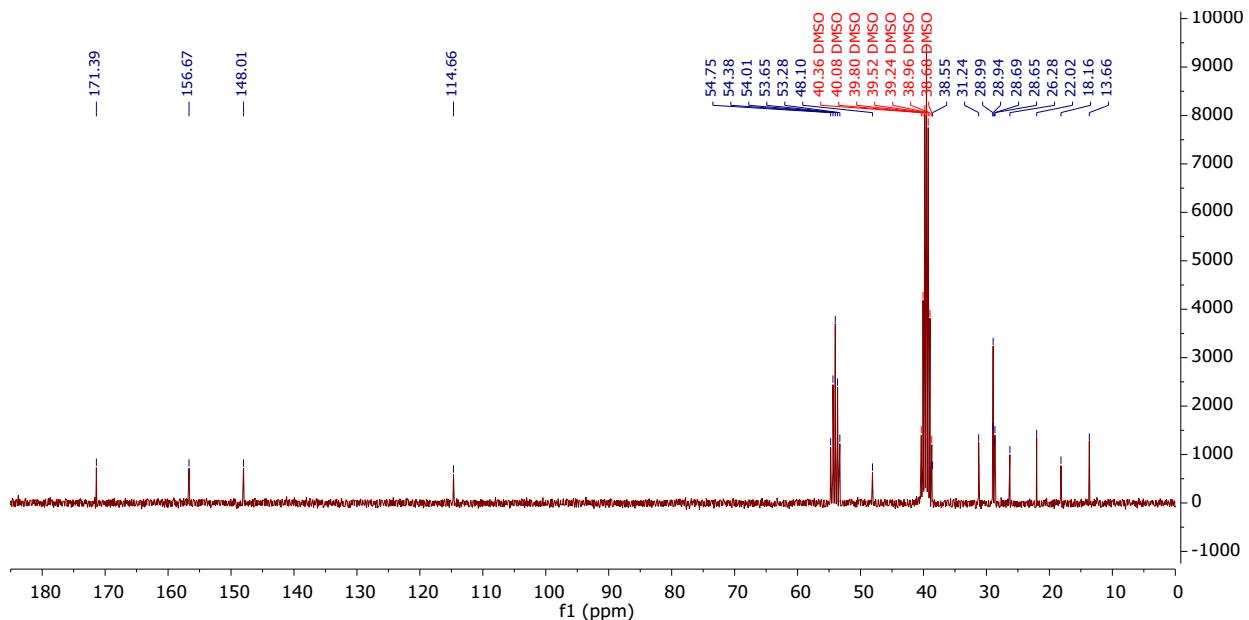
**Figure S1.** <sup>1</sup>H NMR spectrum of compound 16a in CD<sub>2</sub>Cl<sub>2</sub>/DMSO-D<sub>6</sub>



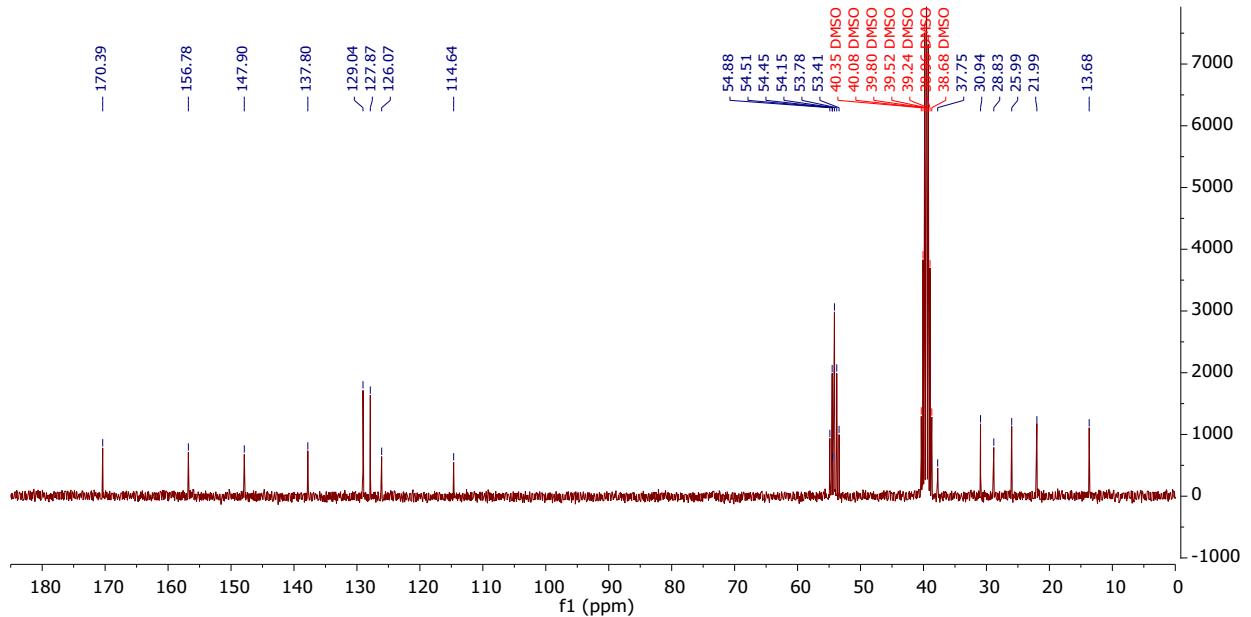
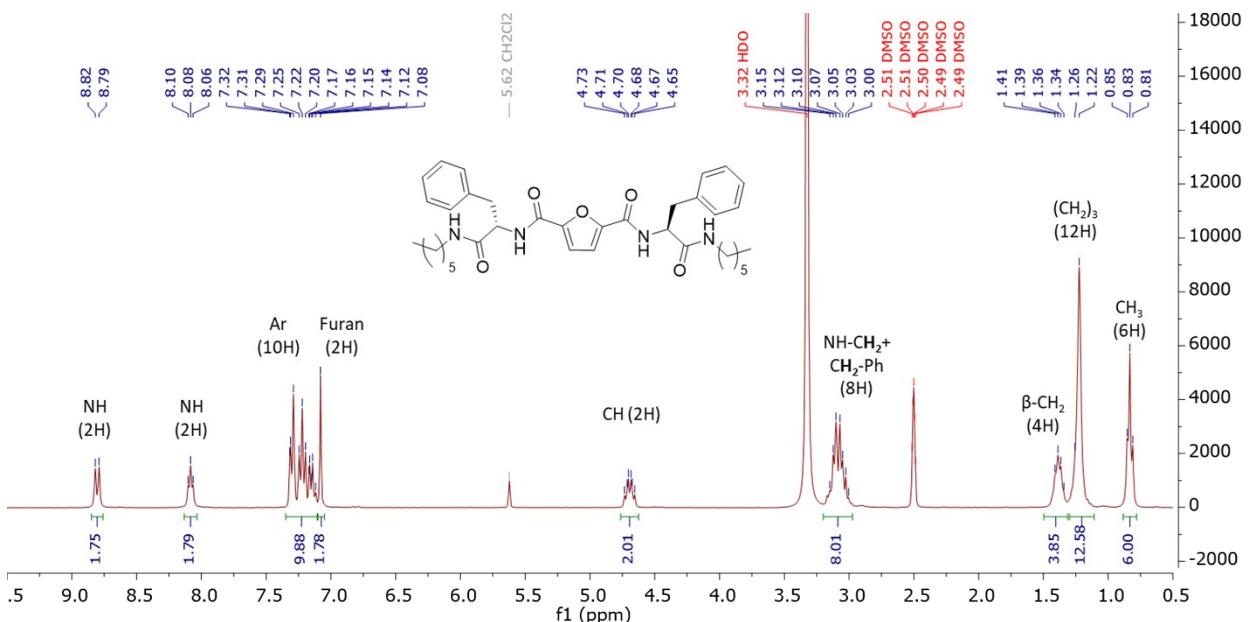
**Figure S2.** <sup>13</sup>C NMR spectrum of compound 16a in CD<sub>2</sub>Cl<sub>2</sub>/DMSO-D<sub>6</sub>



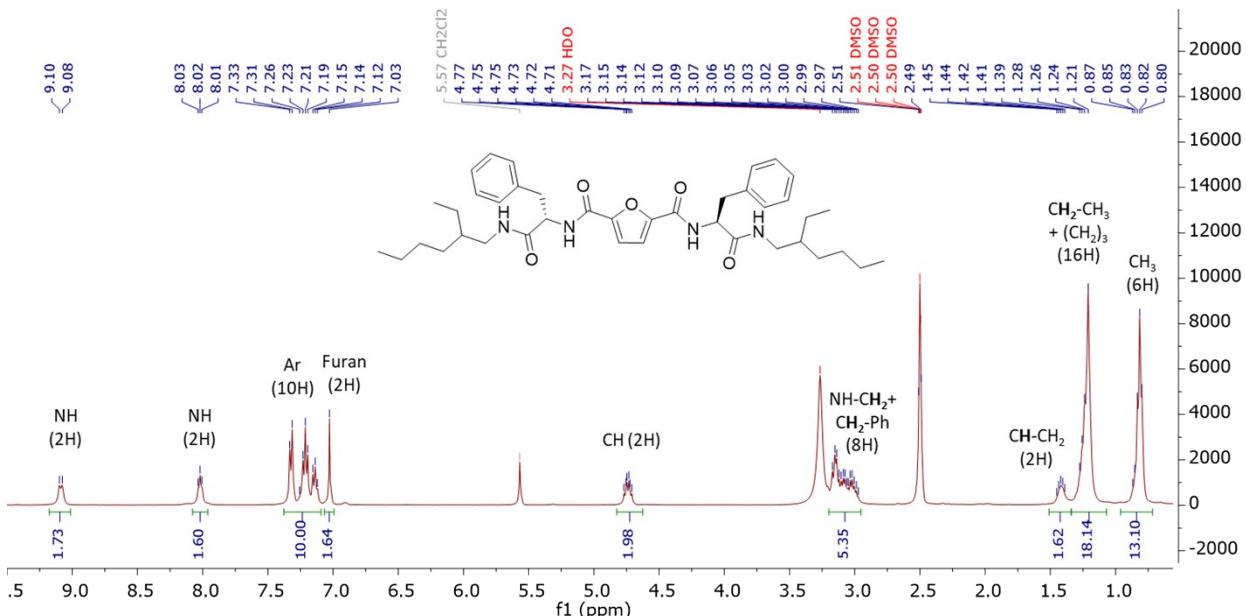
**Figure S3.**  $^1\text{H}$  NMR spectrum of compound 16d in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$



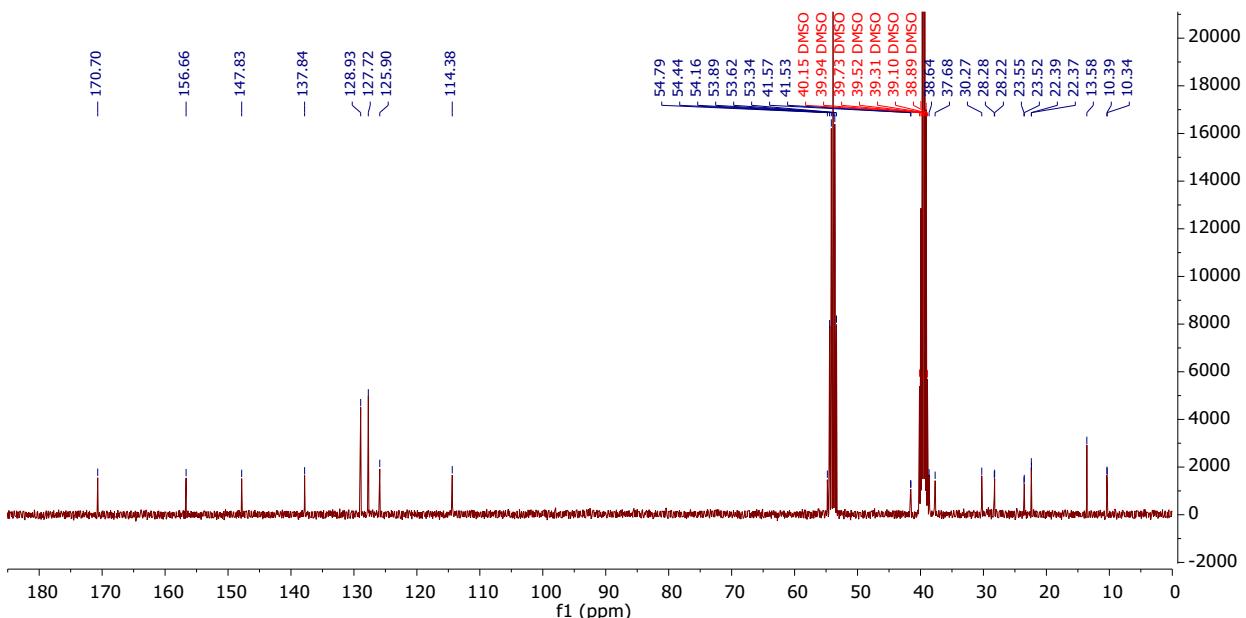
**Figure S4.**  $^{13}\text{C}$  NMR spectrum of compound 16d in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$



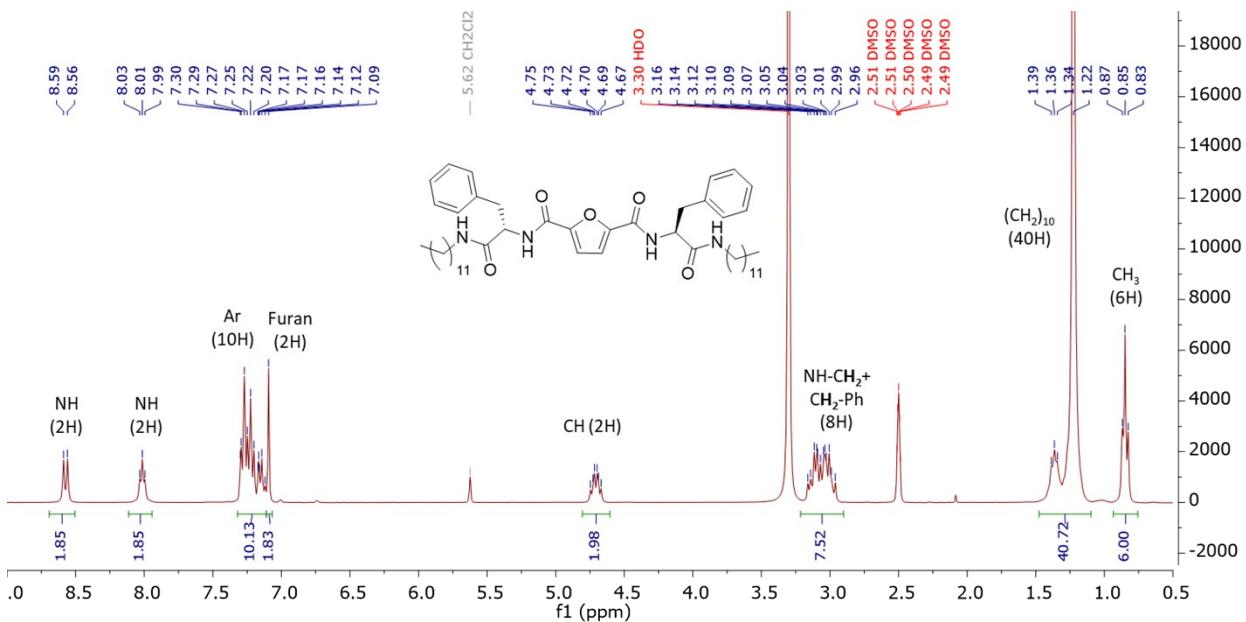
**Figure S6.**  $^{13}\text{C}$  NMR spectrum of compound 17a in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$



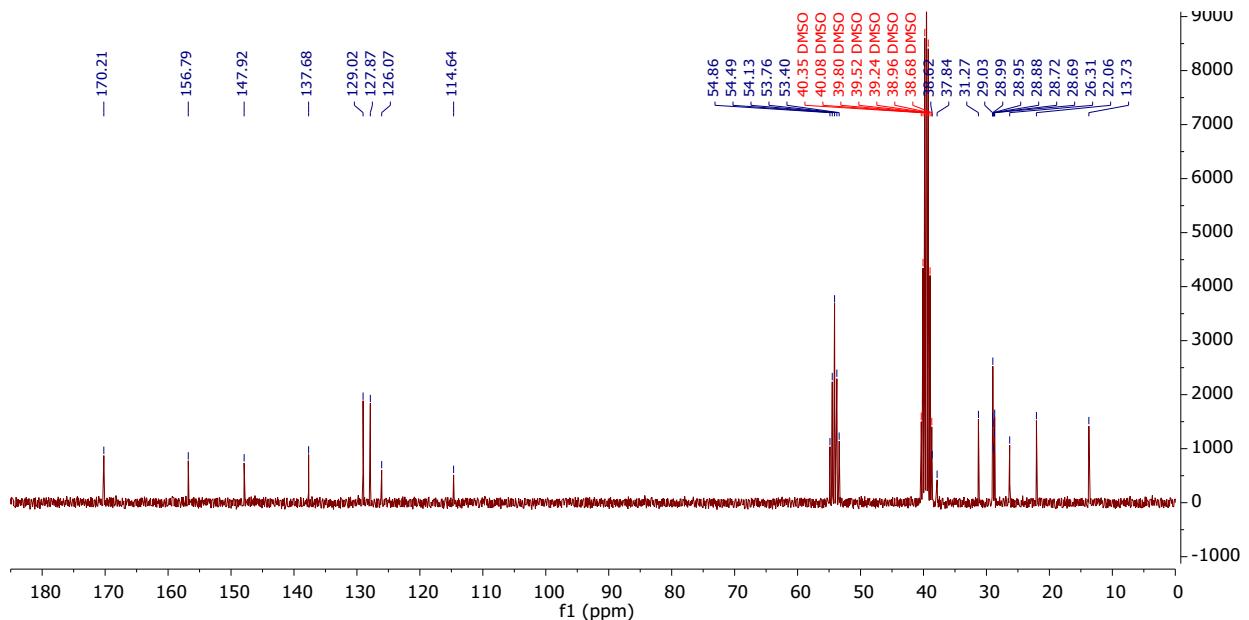
**Figure S7.**  $^1\text{H}$  NMR spectrum of compound 17b in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$



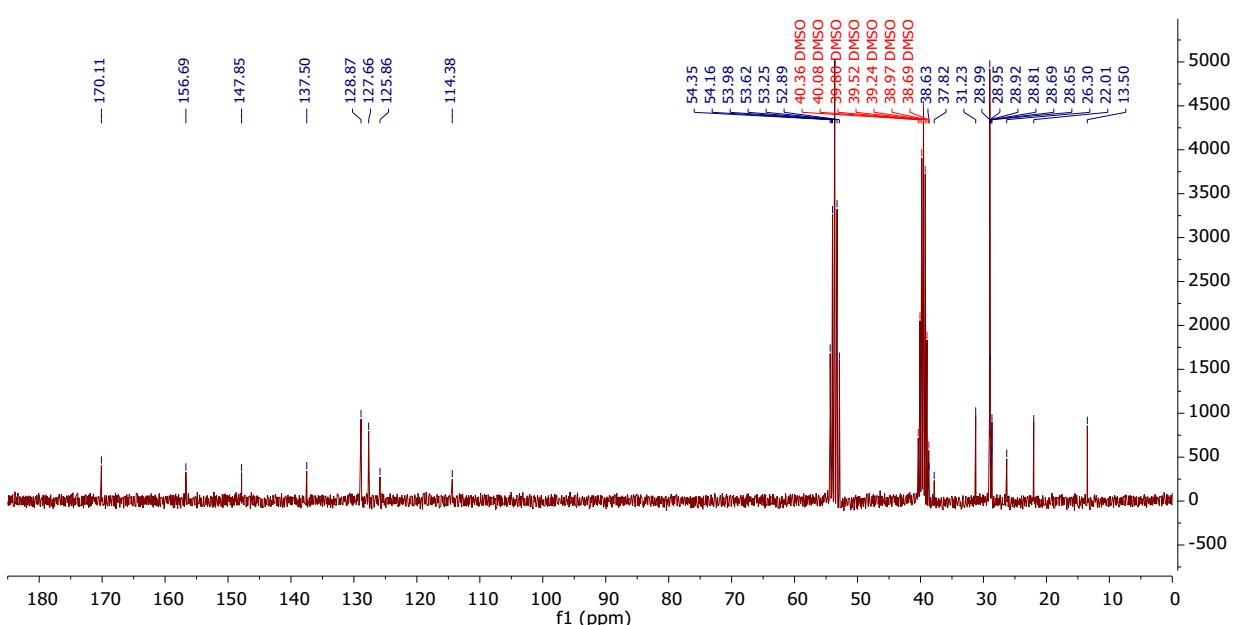
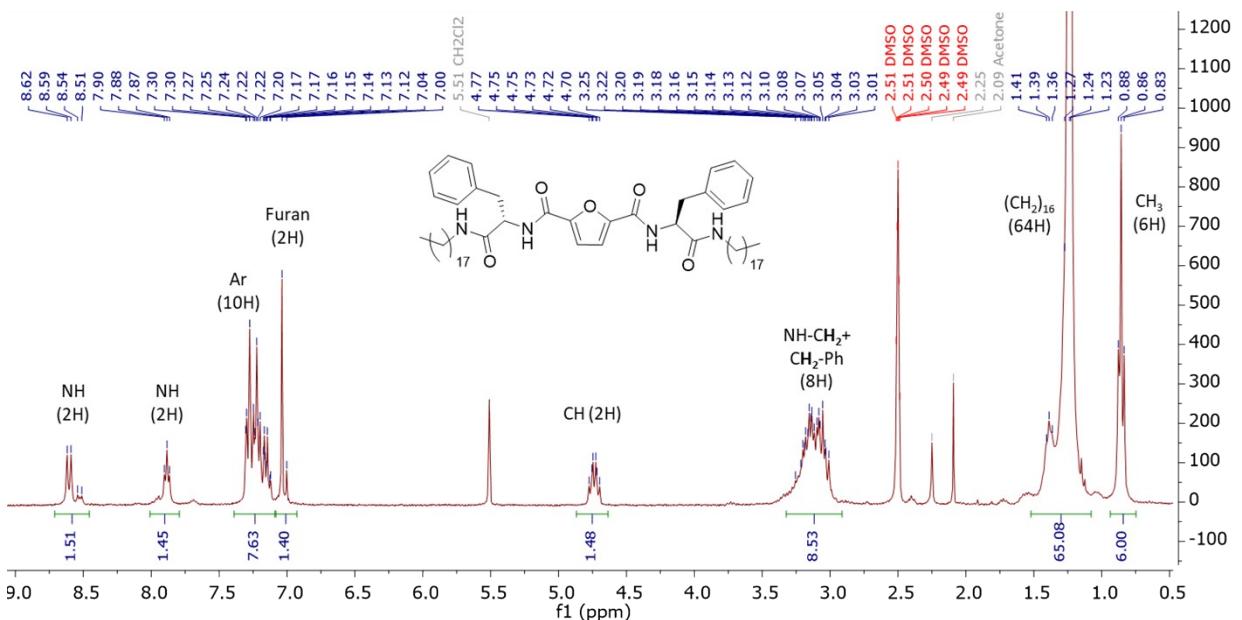
**Figure S8.**  $^{13}\text{C}$  NMR spectrum of compound 17b in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$

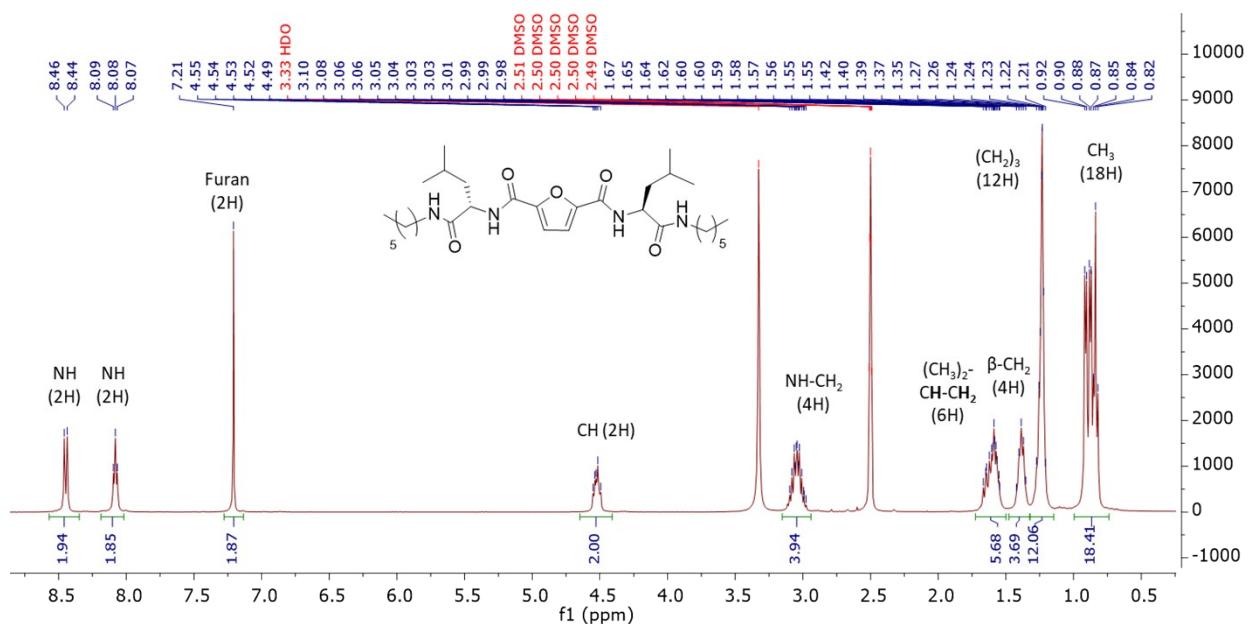


**Figure S9.** <sup>1</sup>H NMR spectrum of compound 17d in CD<sub>2</sub>Cl<sub>2</sub>/DMSO-D<sub>6</sub>

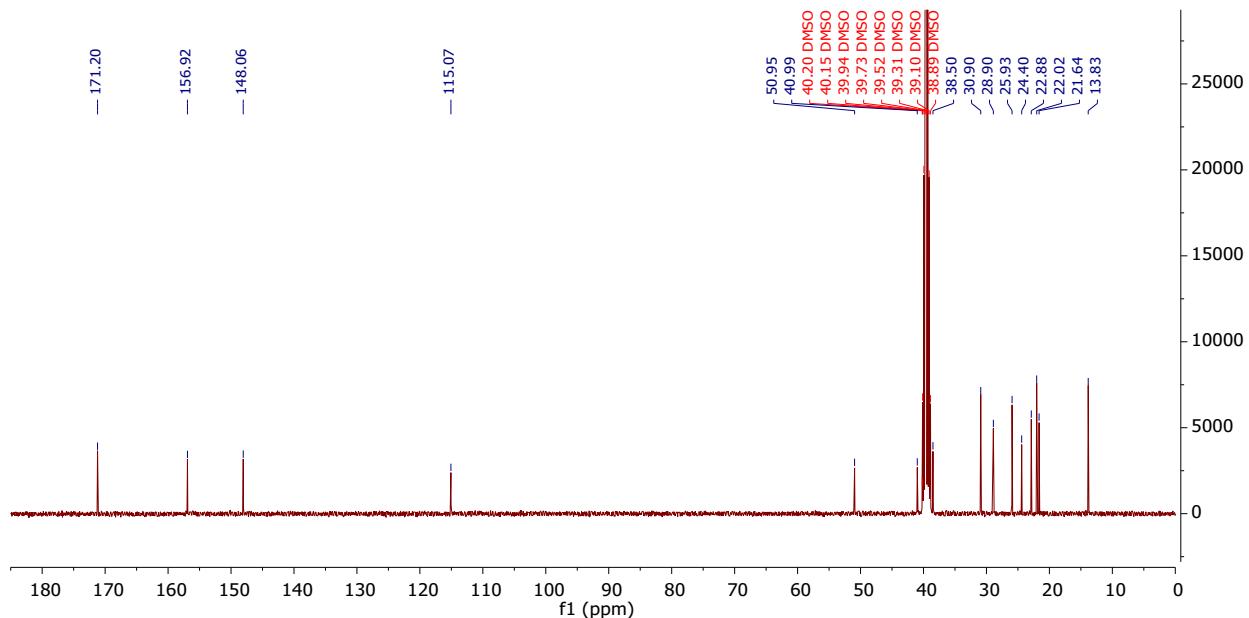


**Figure S10.** <sup>13</sup>C NMR spectrum of compound 17d in CD<sub>2</sub>Cl<sub>2</sub>/DMSO-D<sub>6</sub>

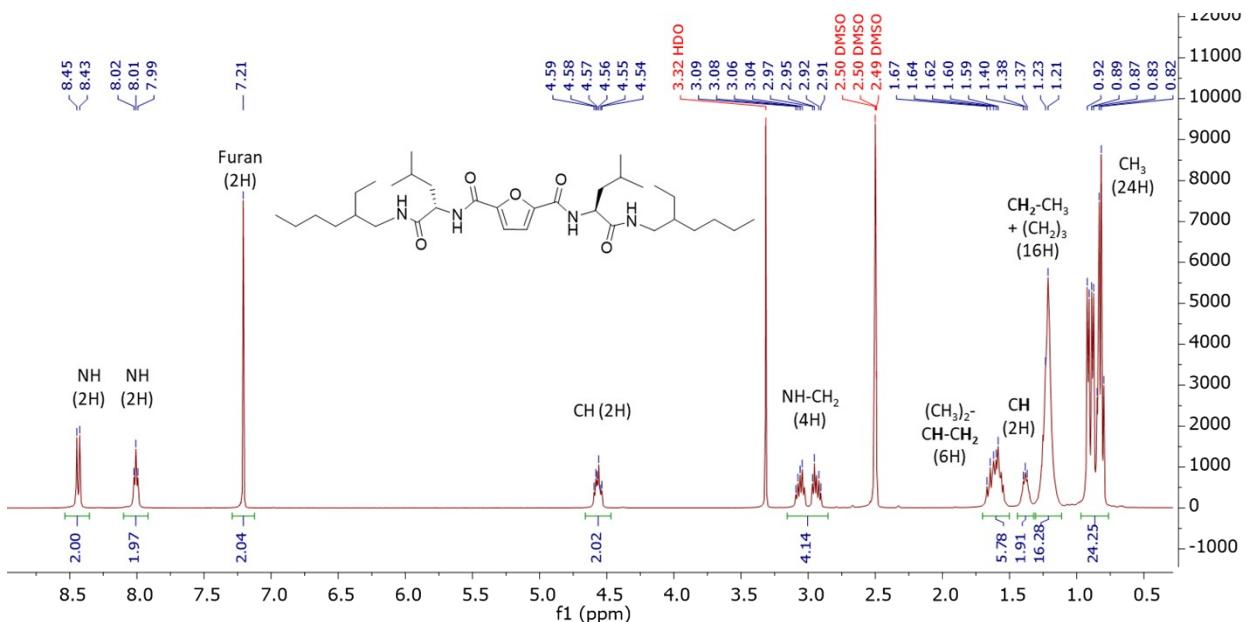




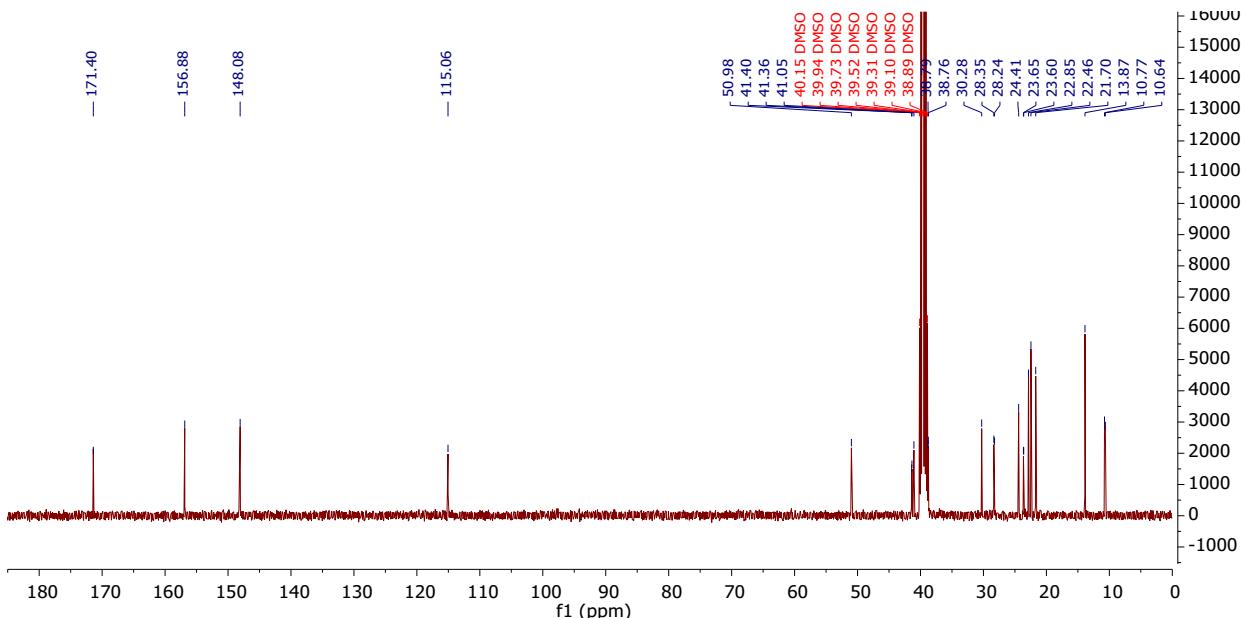
**Figure S13.**  $^1\text{H}$  NMR spectrum of compound 18a in DMSO- $\text{D}_6$



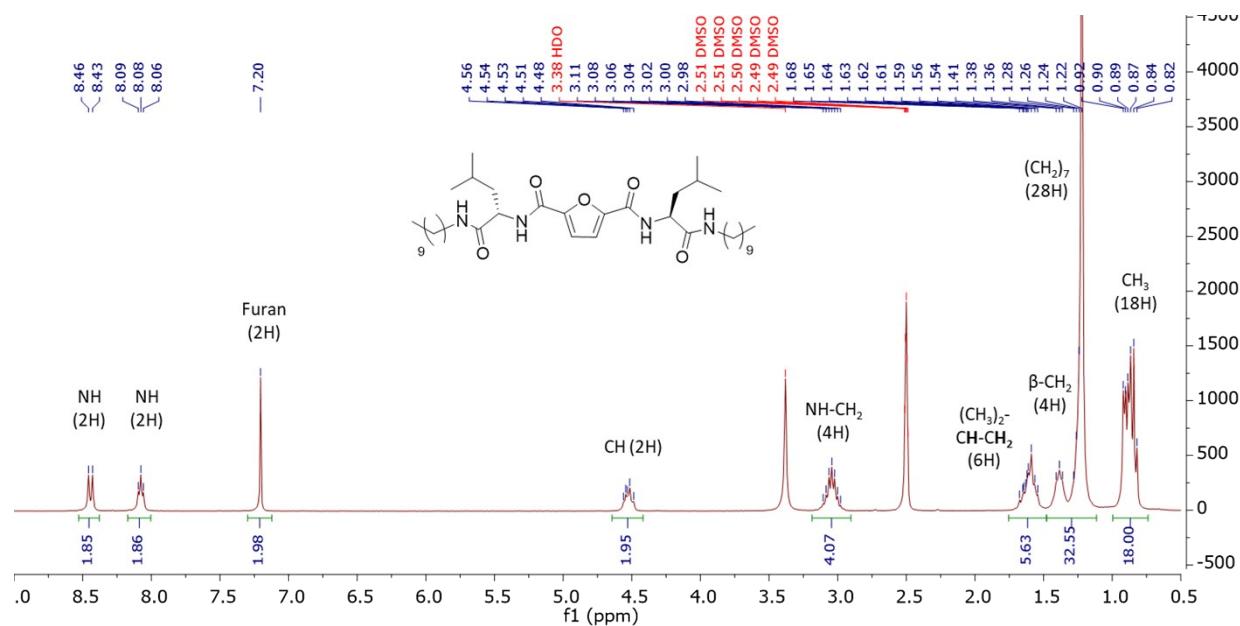
**Figure S14.**  $^{13}\text{C}$  NMR spectrum of compound 18a in  $\text{DMSO-D}_6$



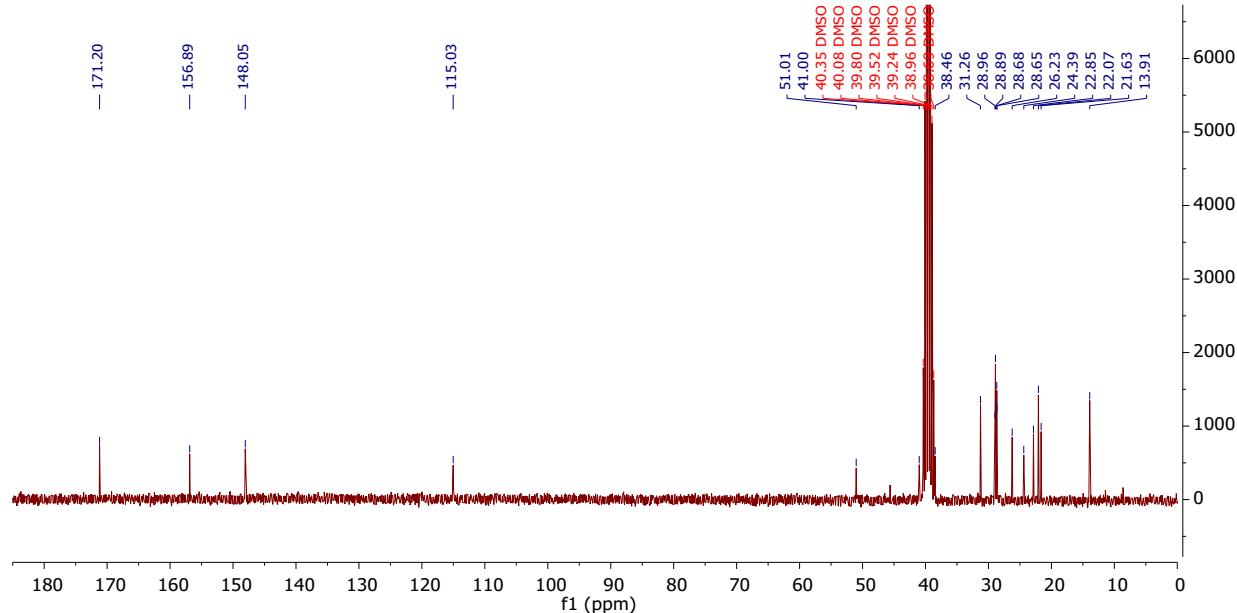
**Figure S15.**  $^1\text{H}$  NMR spectrum of compound 18b in DMSO-D<sub>6</sub>



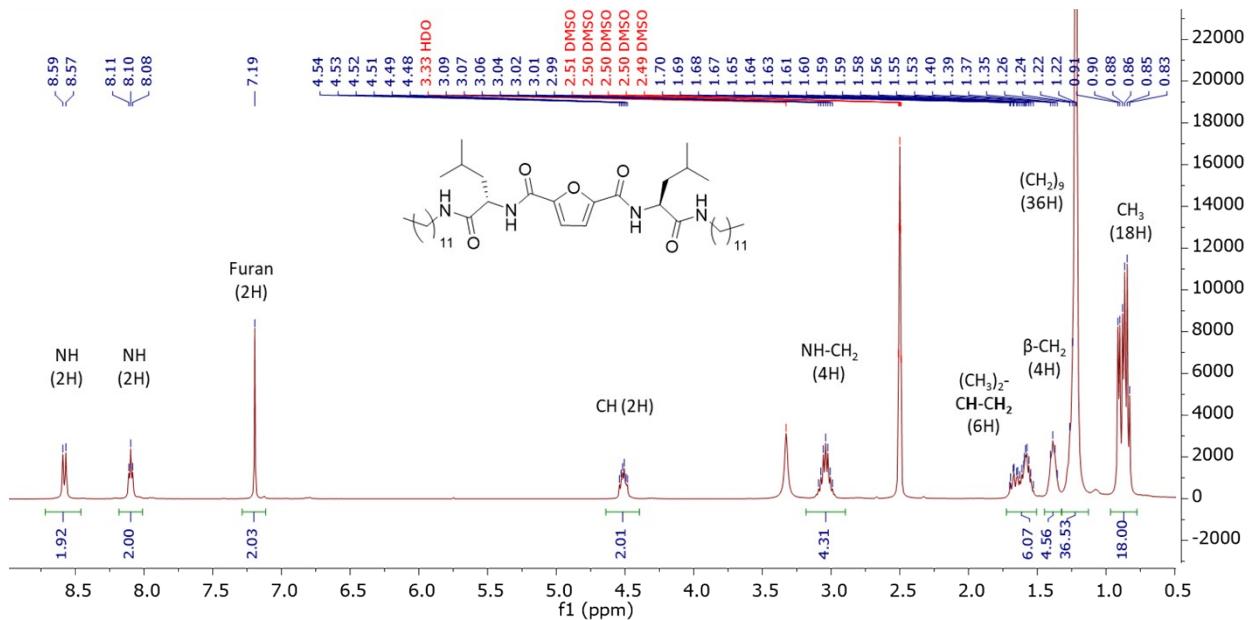
**Figure S16.**  $^{13}\text{C}$  NMR spectrum of compound 18b in  $\text{DMSO-D}_6$



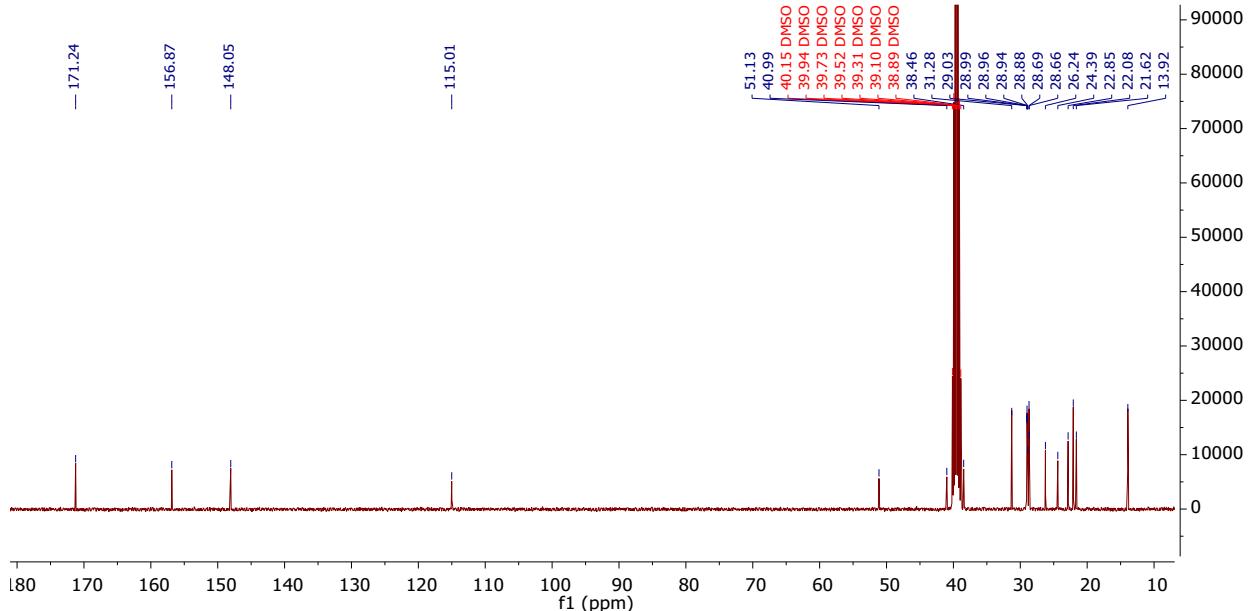
**Figure S17.** <sup>1</sup>H NMR spectrum of compound 18c in DMSO-D<sub>6</sub>



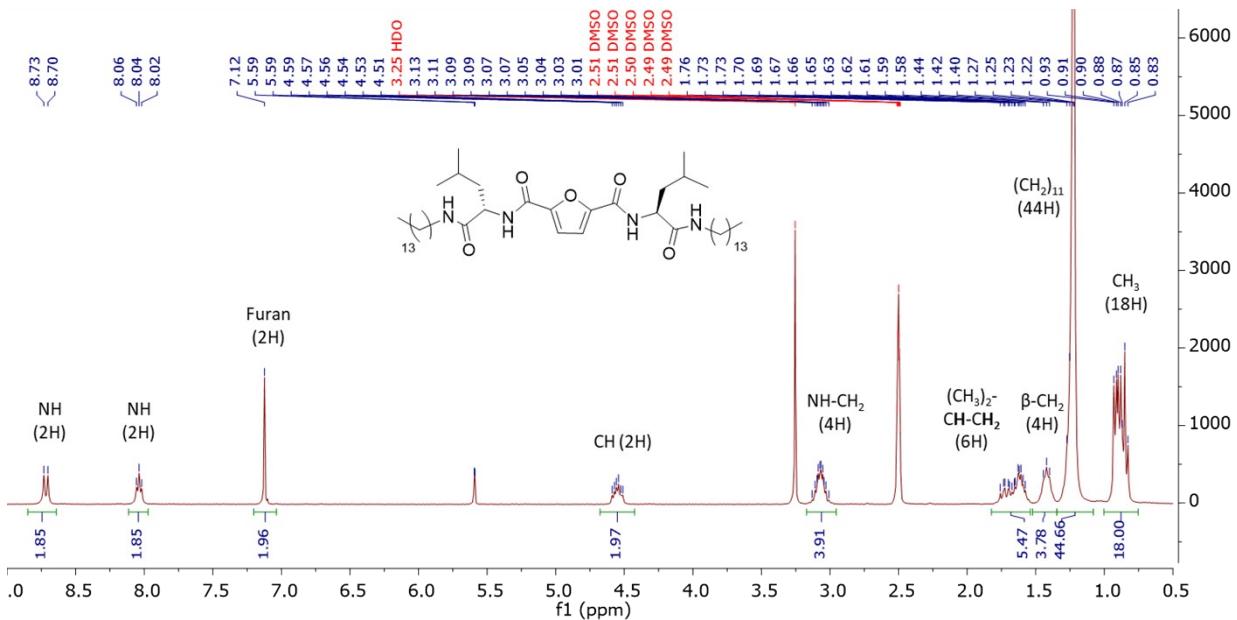
**Figure S18.** <sup>13</sup>C NMR spectrum of compound 18c in DMSO-D<sub>6</sub>



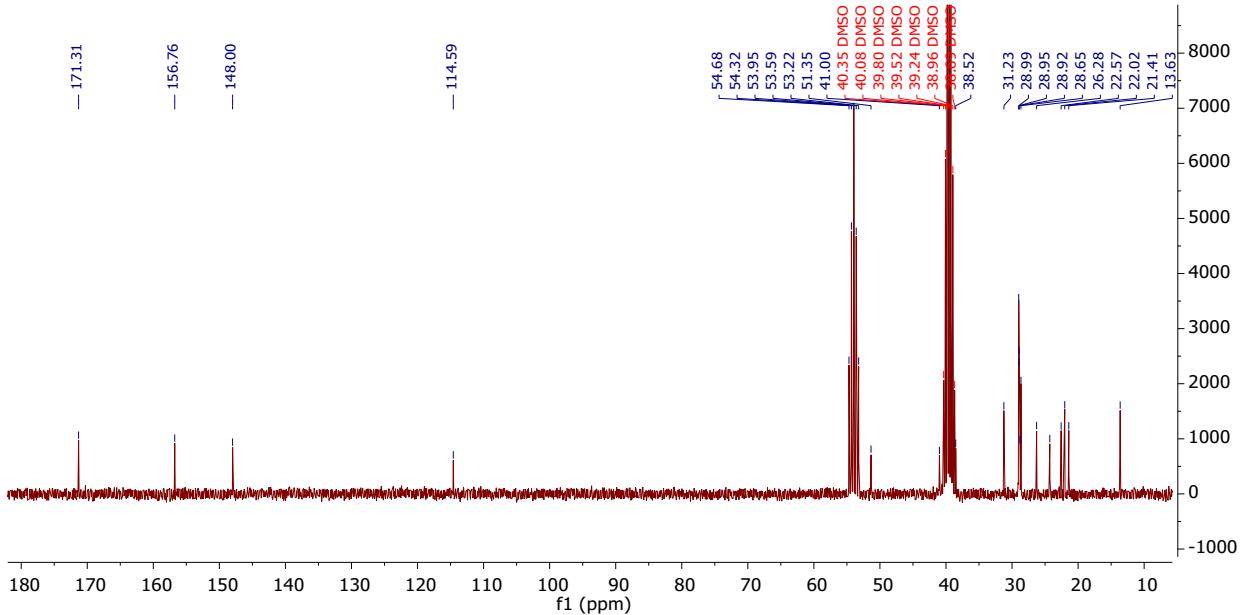
**Figure S19.**  $^1\text{H}$  NMR spectrum of compound 18d in DMSO- $\text{D}_6$



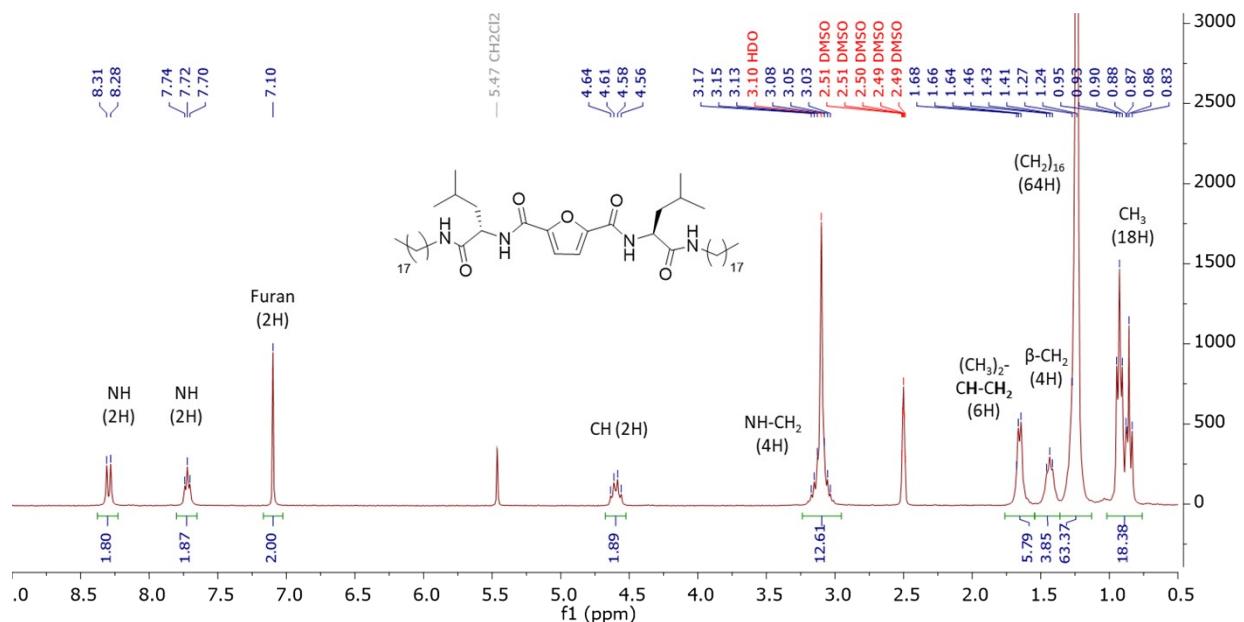
**Figure S20.**  $^{13}\text{C}$  NMR spectrum of compound 18d in DMSO- $\text{D}_6$

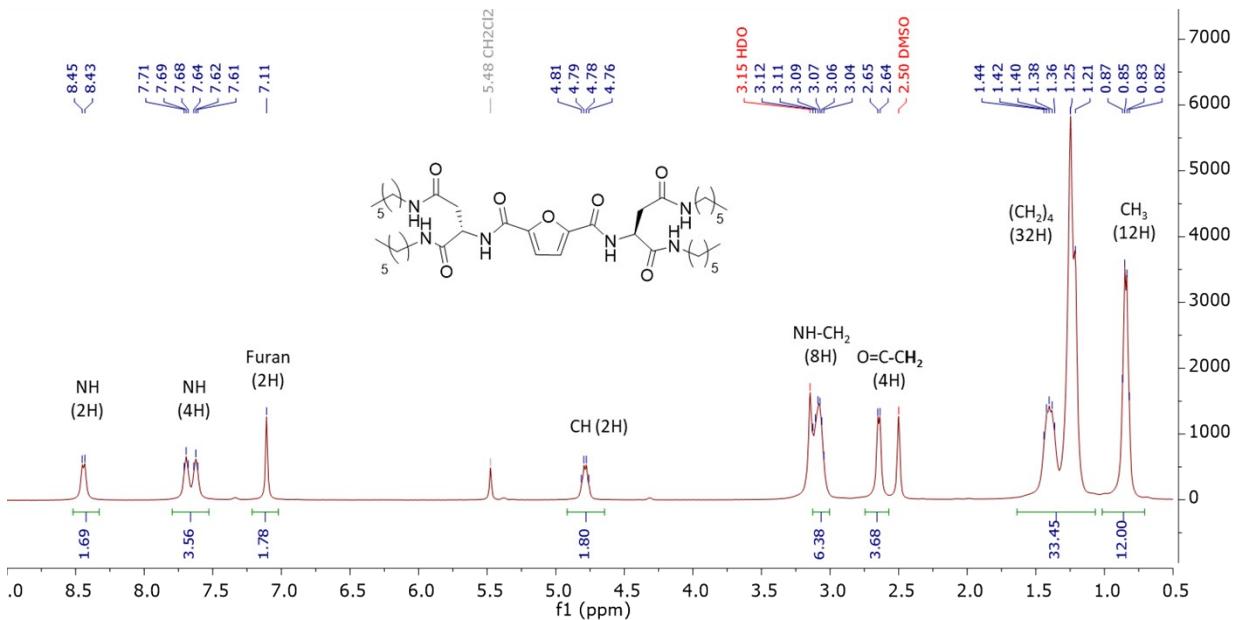


**Figure S21.**  $^1\text{H}$  NMR spectrum of compound 18e in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$

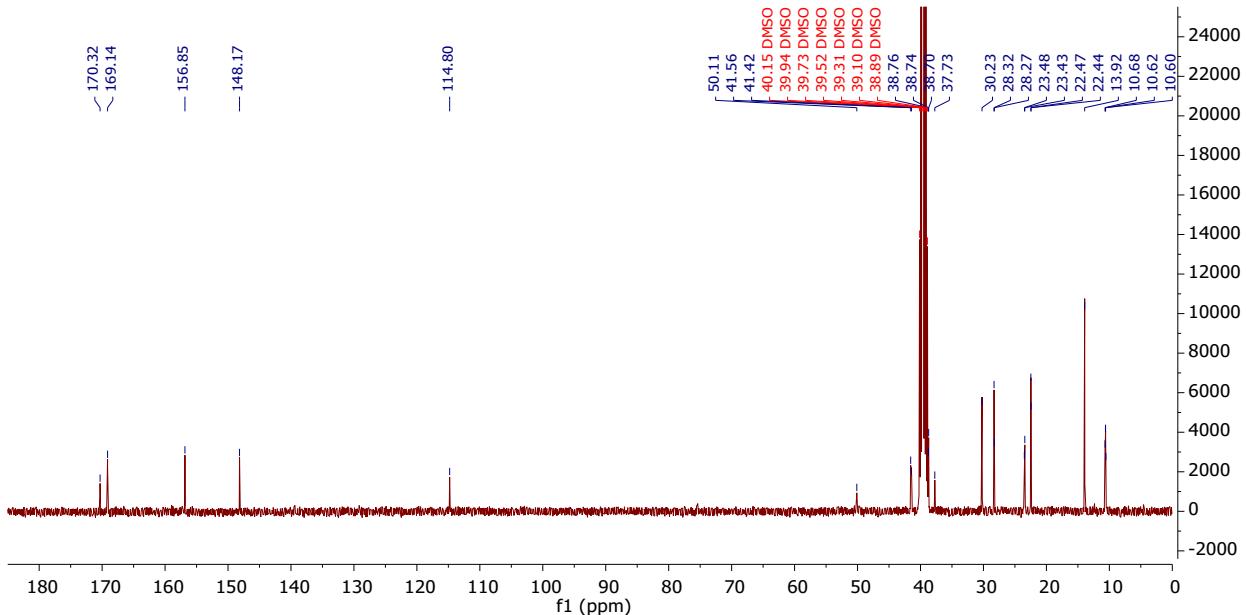


**Figure S22.**  $^{13}\text{C}$  NMR spectrum of compound 18e in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$

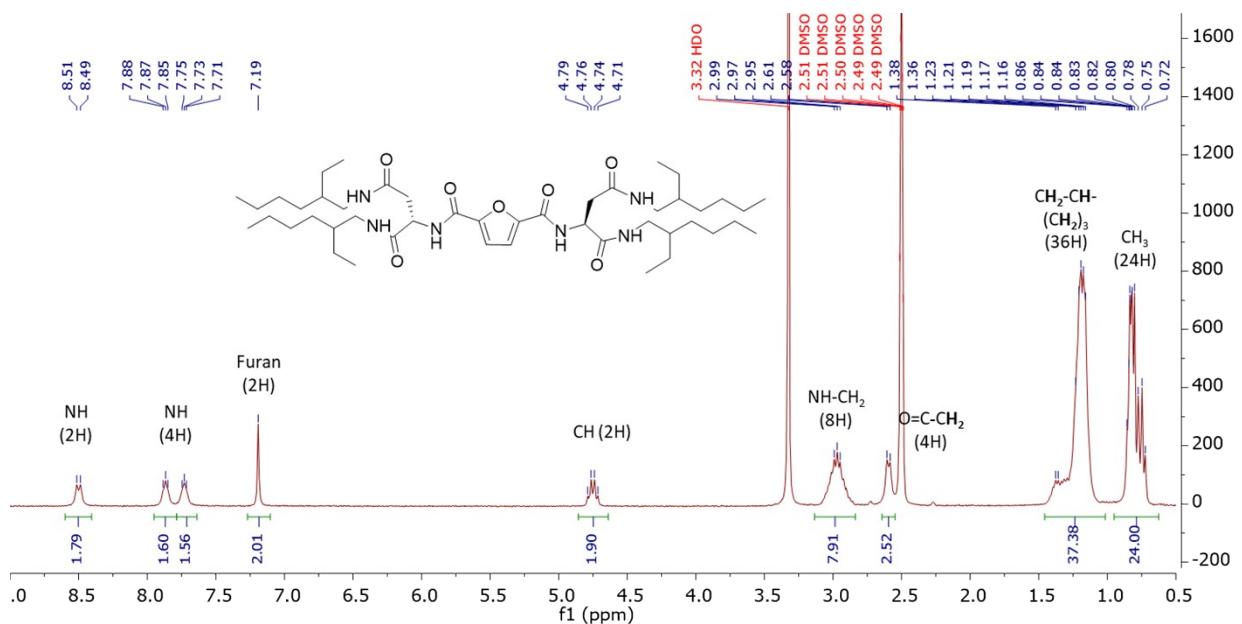




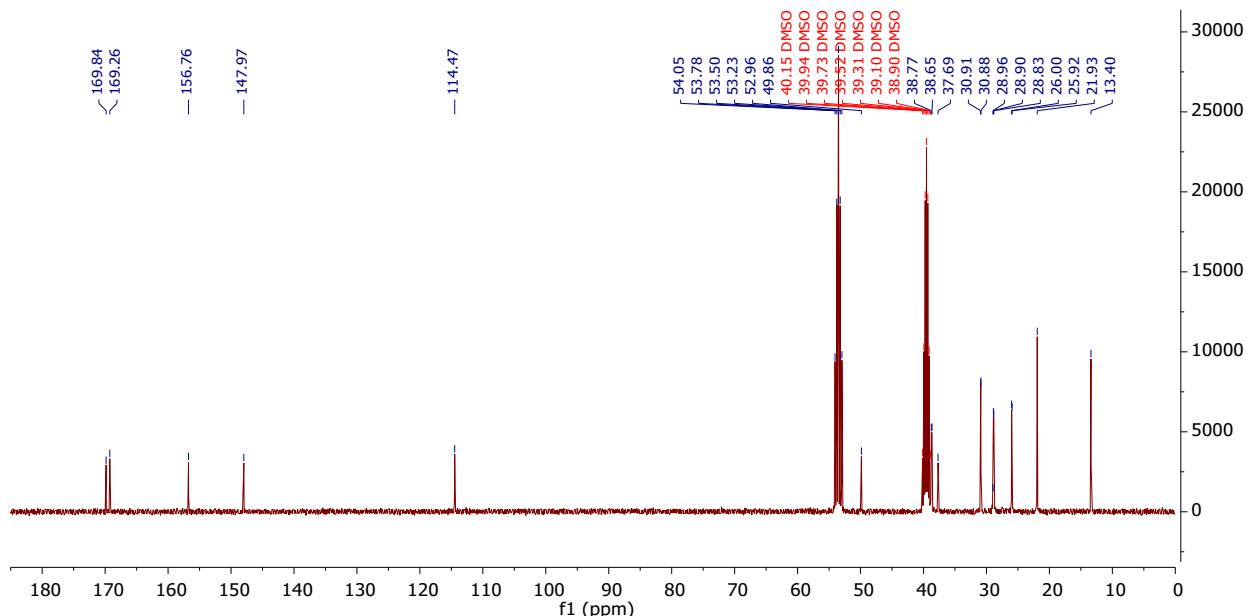
**Figure S25.**  $^1\text{H}$  NMR spectrum of compound 19a in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$



**Figure S26.**  $^{13}\text{C}$  NMR spectrum of compound 19a in  $\text{CD}_2\text{Cl}_2/\text{DMSO-D}_6$



**Figure S27.**  $^1\text{H}$  NMR spectrum of compound 19b in DMSO- $\text{D}_6$



**Figure S28.**  $^{13}\text{C}$  NMR spectrum of compound 19b in  $\text{DMSO-D}_6$

**Table S1. Gelation tests of alkyl and aromatic diamides at 1wt%<sup>a</sup>**

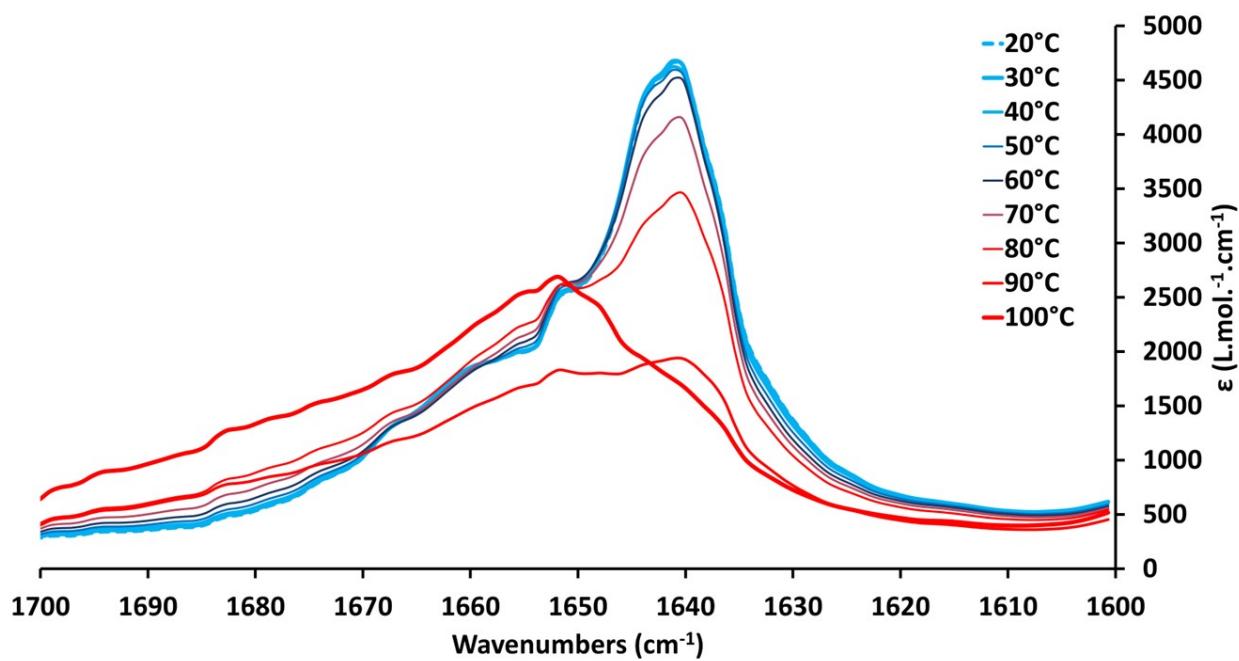
Compound	propylene carbonate	diacetone alcohol	benzyl acetate	1,4-dioxane	<i>m</i> -xylene	<i>R</i> (+)-limonene	rapeseed oil	n-hexadecane
<b>2a</b>	P	S	S	S	S	P	I	I
<b>2b</b>	I	S	P	S	P	P	I	I
<b>2c</b>	I	S	P	S	P	P	I	I
<b>2d</b>	I	P	P	S	P	P	I	I
<b>2e</b>	I	P	P	P	P	P	I	I
<b>2f</b>	S	S	S	S	S	S	S	I
<b>2g</b>	I	I	I	S	I	I	I	I
<b>2h</b>	S	S	S	S	I	I	I	I
<b>2i</b>	S	S	S	S	P	I	I	I

<sup>a</sup> P: precipitate; S: soluble; I: insoluble.

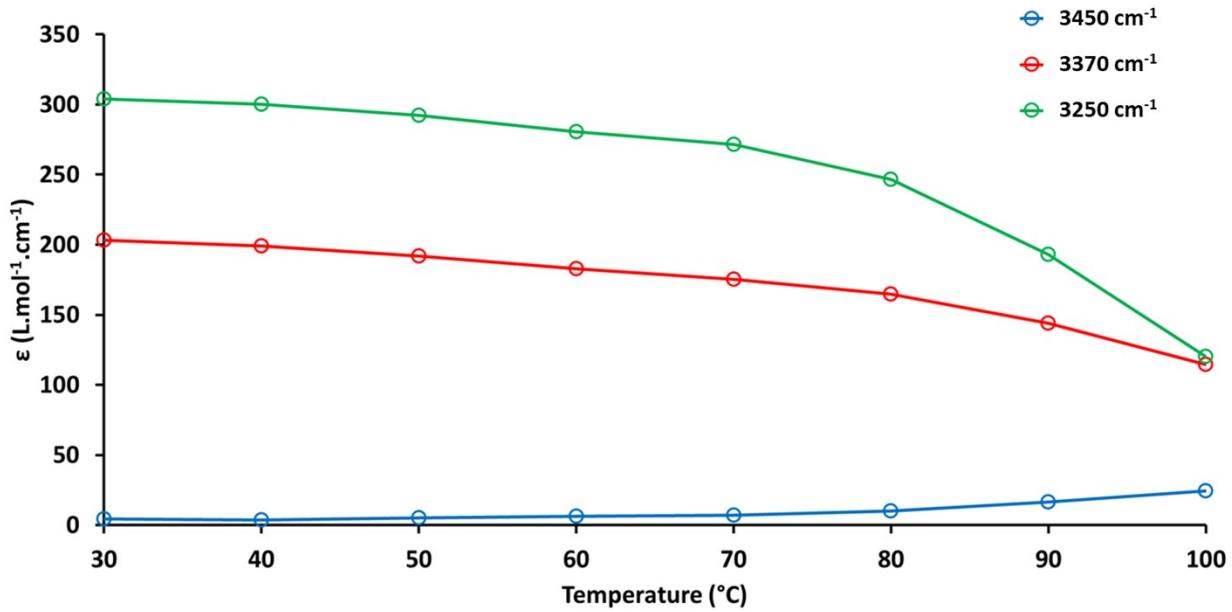
**Table S2. Gelation tests of di(amido-esters) at 1wt%**<sup>a</sup>

Compound	propylene carbonate	diacetone alcohol	benzyl acetate	1,4-dioxane	<i>m</i> -xylene	<i>R</i> (+)-limonene	rapeseed oil	n-hexadecane
<b>8a</b>	S	S	S	S	S	S	I	I
<b>8c</b>	S	S	S	S	S	S	S	S
<b>8d</b>	S	S	S	S	S	S	S	S
<b>8e</b>	S	S	S	S	S	S	S	S
<b>8f</b>	P	S	S	S	S	S	S	S
<b>8g</b>	P	S	S	S	S	S	S	S
<b>8h</b>	P	S	S	S	S	S	S	P
<b>9a</b>	S	S	S	S	S	S	S	I
<b>9b</b>	S	S	S	S	S	S	S	P
<b>9c</b>	S	S	S	S	S	S	S	S
<b>9d</b>	S	S	S	S	S	S	S	S
<b>9e</b>	S	S	S	S	S	S	S	S
<b>9f</b>	S	S	S	S	S	S	S	S
<b>9g</b>	P	S	S	S	S	S	S	S
<b>9h</b>	P	S	S	S	S	S	S	S
<b>10a</b>	S	S	S	S	S	S	S	P
<b>10c</b>	S	S	S	S	S	S	S	S
<b>10d</b>	S	S	S	S	S	S	S	S
<b>10e</b>	S	S	S	S	S	S	S	S
<b>10f</b>	P	S	S	S	S	S	S	S
<b>10g</b>	P	S	S	S	S	S	S	S
<b>10h</b>	P	S	S	S	S	S	S	S
<b>11a</b>	S	S	S	S	S	S	S	I
<b>11b</b>	S	S	S	S	S	S	S	S
<b>11d</b>	P	S	S	S	S	S	S	S
<b>11e</b>	P	S	S	S	S	S	S	S
<b>11g</b>	P	P	S	S	S	S	P	P
<b>11h</b>	P	P	P	S	P	P	P	P

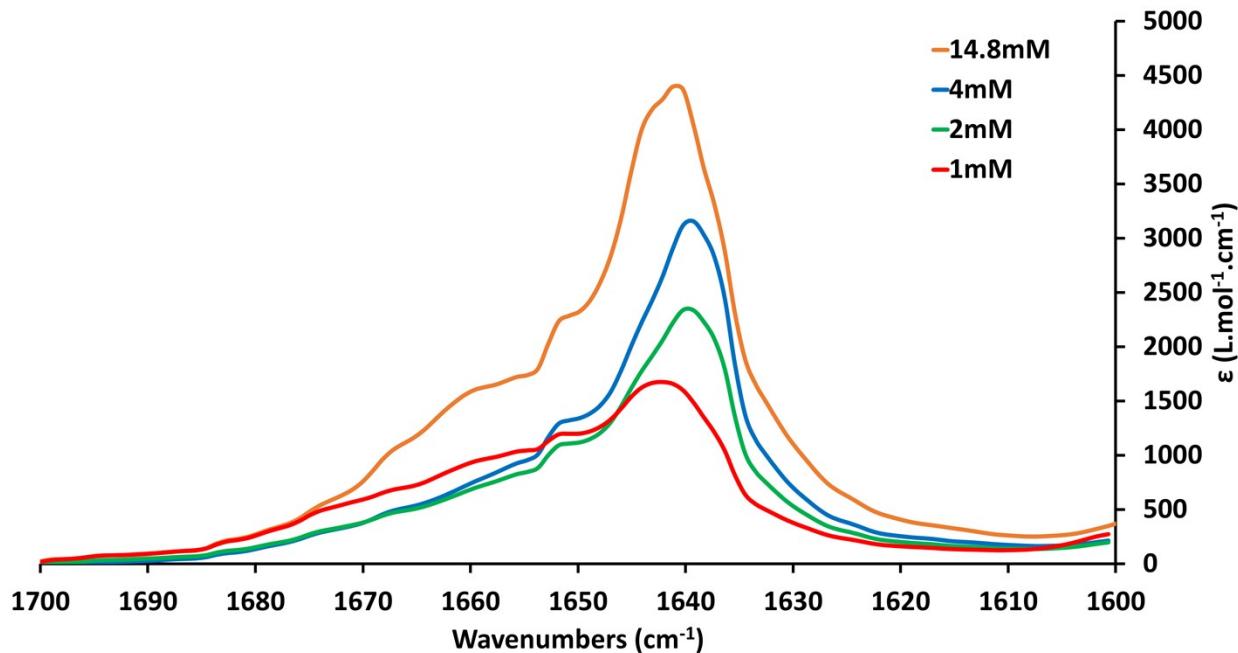
<sup>a</sup> P: precipitate; S: soluble; I: insoluble.



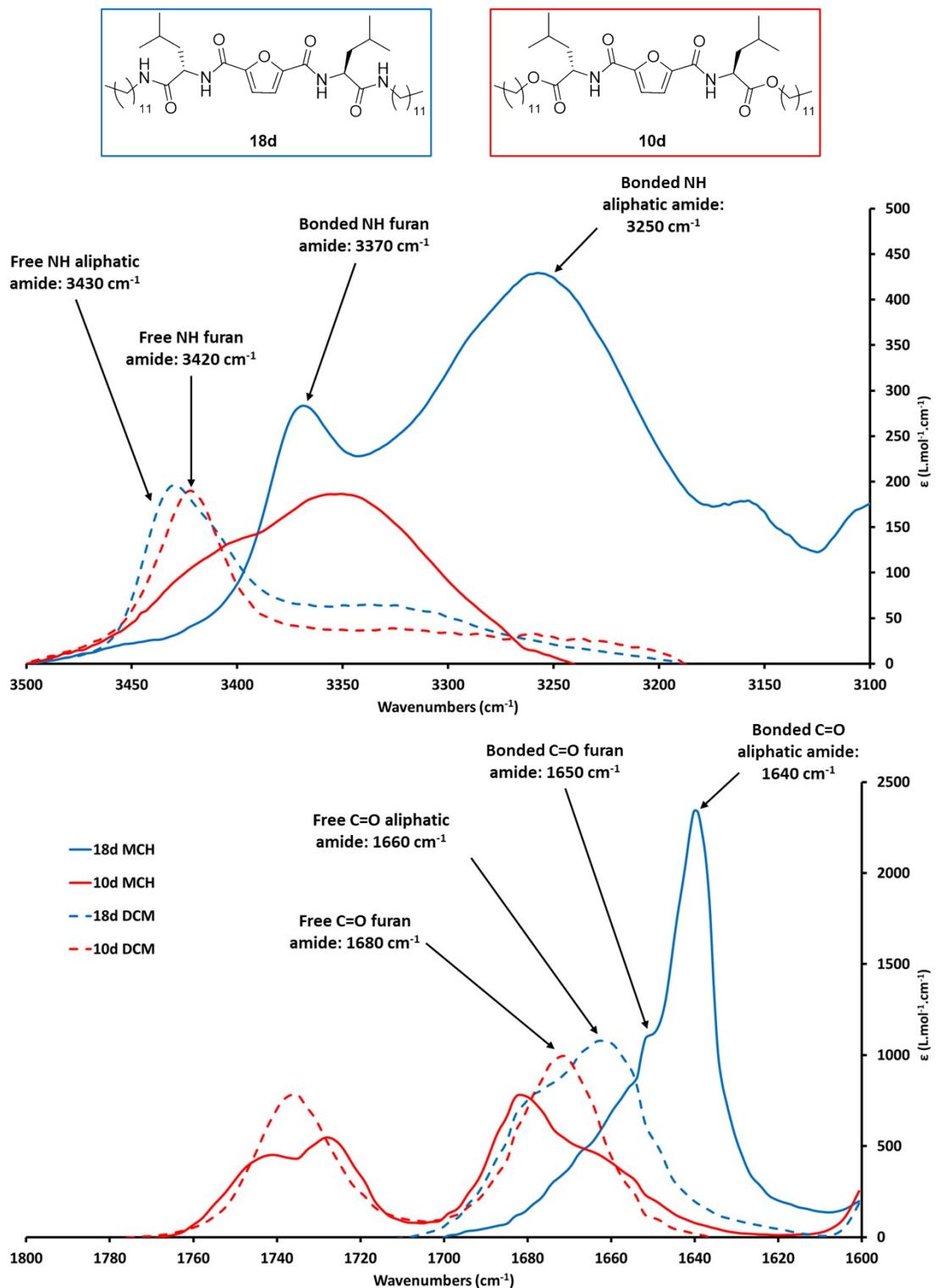
**Figure S29.** VT-FTIR spectra of gelator 18d in methylcyclohexane; 1wt%, heating process, 1°C/min ramp; C=O bands.



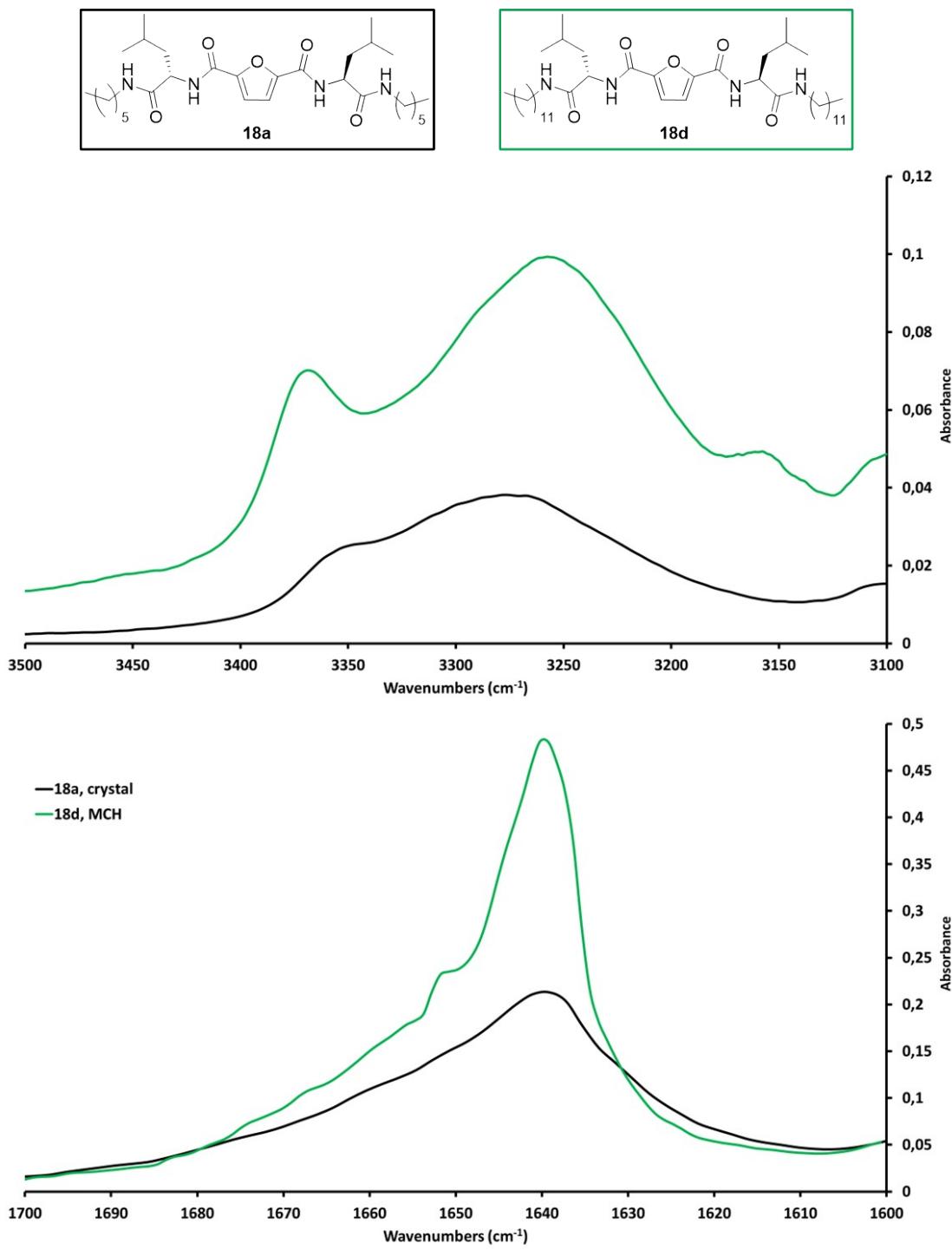
**Figure S30.** Intensity of the NH bands depending on the temperature of gelator 18d in methylcyclohexane; 1wt%, heating process, 1°C/min ramp; free NH 3450 cm<sup>-1</sup> (blue line), bonded NH 3370 cm<sup>-1</sup> (red line) and 3250 cm<sup>-1</sup> (green line).



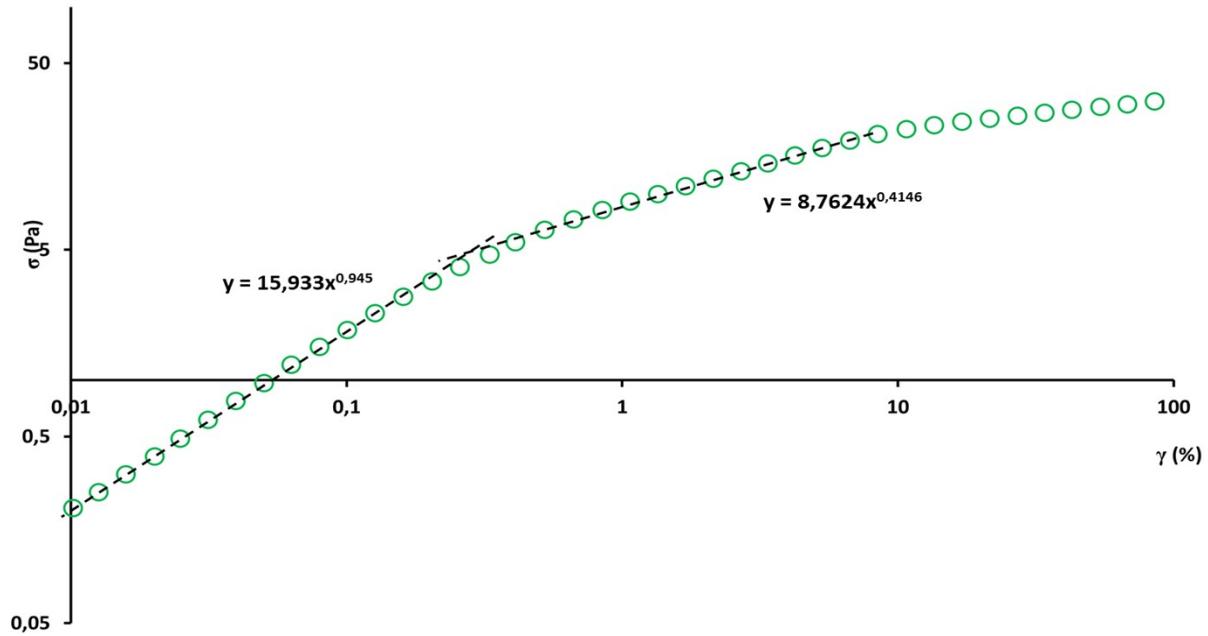
**Figure S31.** FTIR spectra of gelator 18d solutions at 20°C in methylcyclohexane at various concentrations: 14.8mM (orange line), 4mM (blue line), 2mM (red line) and 1mM (green line); C=O bands



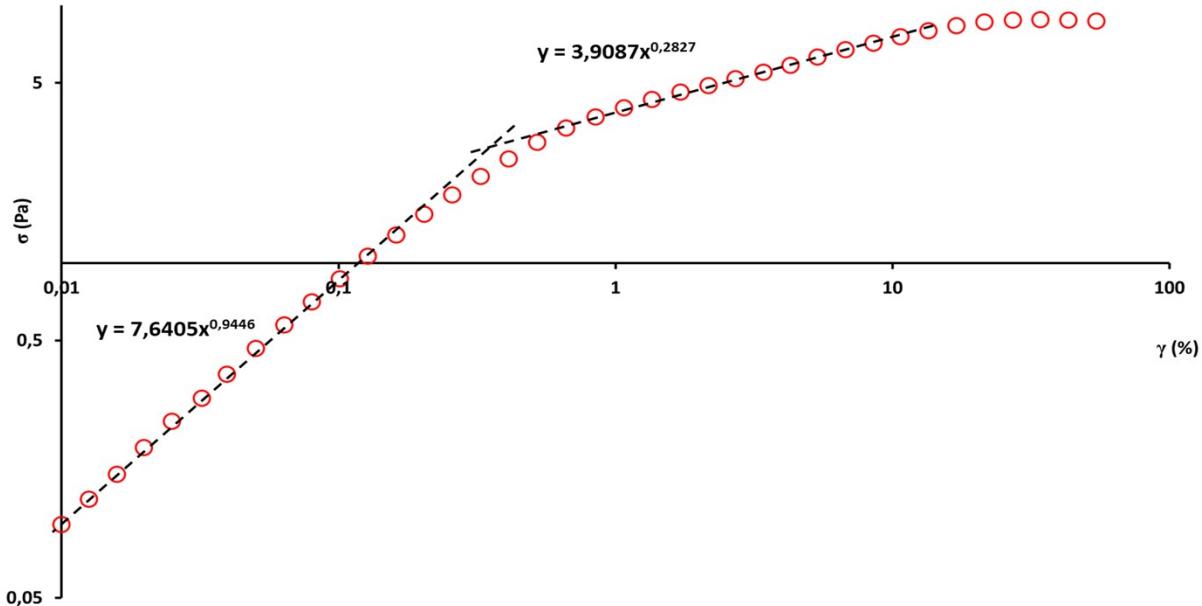
**Figure S32.** FTIR analyses of compounds **18d** (red) and **10d** (blue) in MCH (bold lines) and DCM (dotted lines), absorbance mode. Top) NH region (3500-3100 cm<sup>-1</sup>); Bottom) C=O region (1800-1600 cm<sup>-1</sup>)



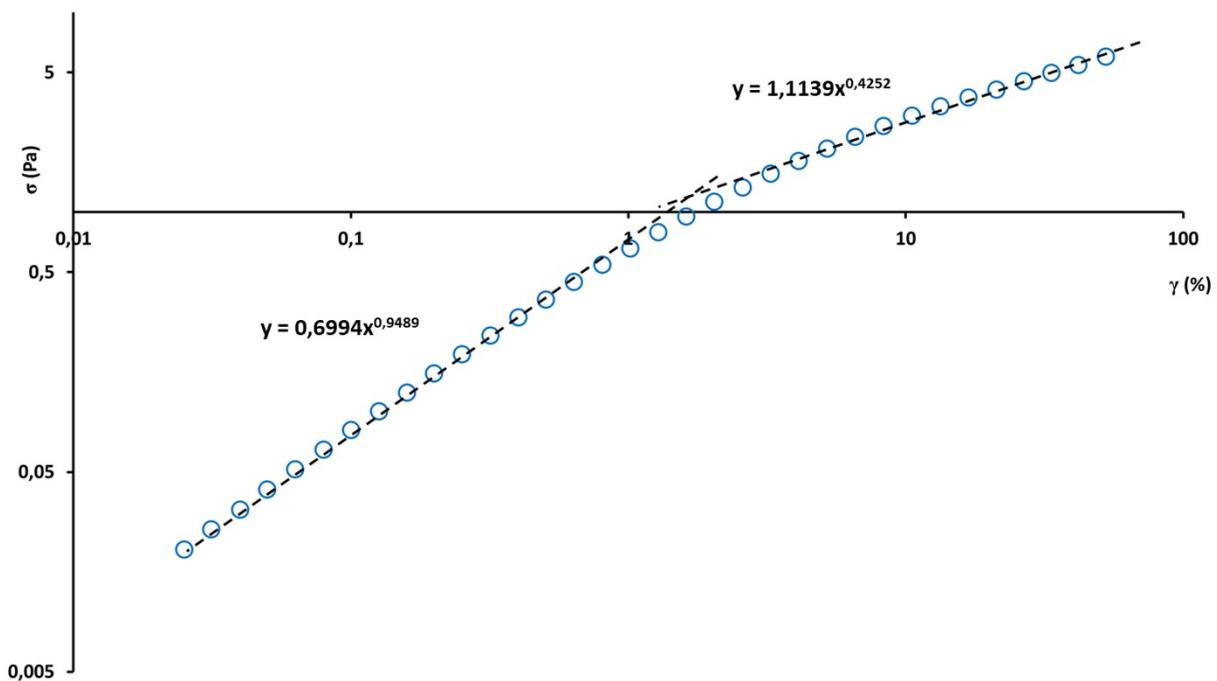
**Figure S33.** FTIR comparison of compounds **18a** (black) crystal and **18d** (green) gel in MCH, absorbance mode. Top) NH region ( $3500\text{-}3100\text{ cm}^{-1}$ ); Bottom) C=O region ( $1800\text{-}1600\text{ cm}^{-1}$ )



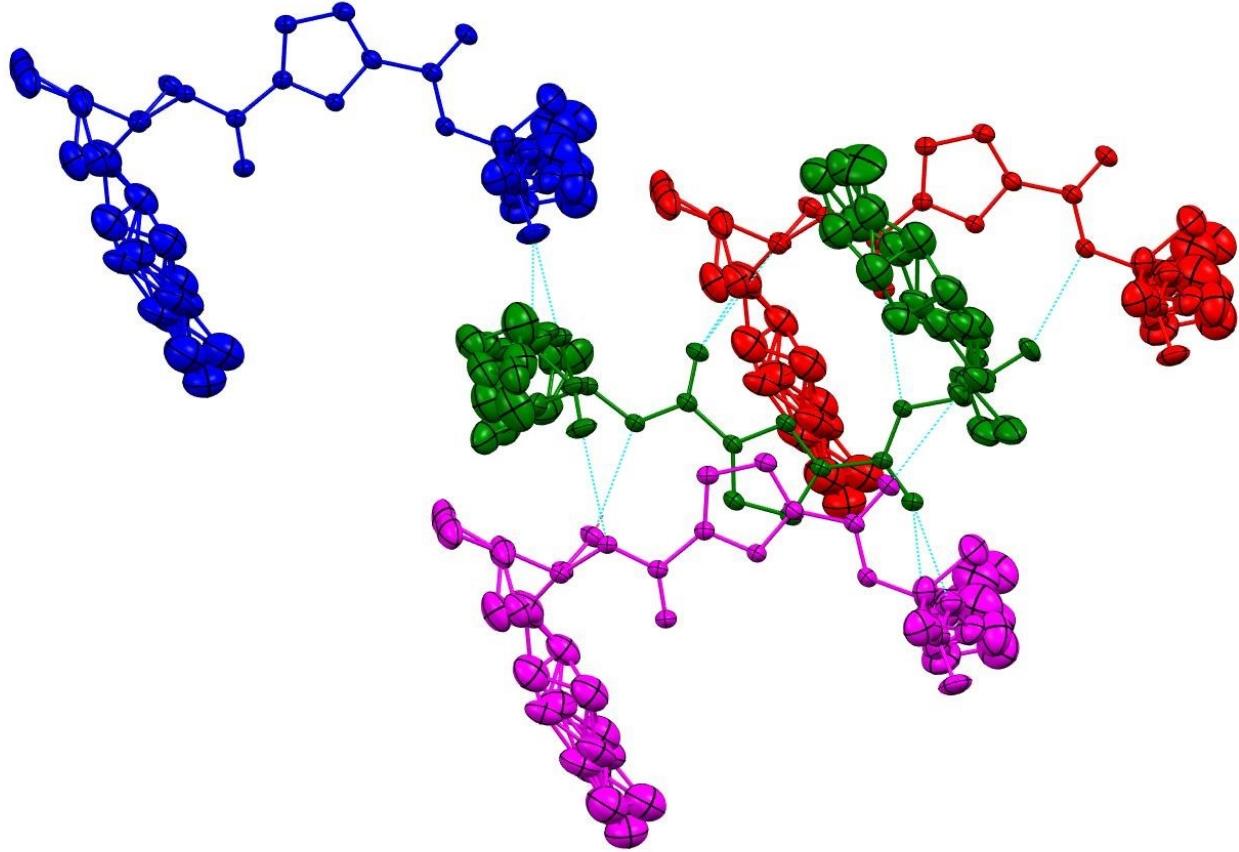
**Figure S34.** Evolution of the stress ( $\sigma$ ) as a function of strain ( $\gamma$ ) for the 2wt% gel in rapeseed oil. The dotted lines correspond to the linear regression lines used to determine the yield stress



**Figure S35.** Evolution of the stress ( $\sigma$ ) as a function of strain ( $\gamma$ ) for the 1.5wt% gel in rapeseed oil. The dotted lines correspond to the linear regression lines used to determine the yield stress



**Figure S36.** Evolution of the stress ( $\sigma$ ) as a function of strain ( $\gamma$ ) for the 1wt% gel in rapeseed oil. The dotted lines correspond to the linear regression lines used to determine the yield stress



**Figure S37. Crystal packing of 18a.** Each neighboring molecule of 18a is depicted in a different color. Green molecule is bounded to three neighboring molecules by eight H-bonds (3 with the red one, 4 with the pink one and 1 with the blue one). These H-bonds are shown in light blue. All hydrogen atoms are not shown for sake of clarity. Only S,S enantiomer is present. Ellipsoids are drawn with 30% probability.

**Table S3. Crystallographic data for 18a**

18a	
<b>CCDC deposit number</b>	2221526
<b>Empirical formula<sup>a</sup></b>	C <sub>30</sub> H <sub>52</sub> N <sub>4</sub> O <sub>5</sub>
<b>Moiety Formula</b>	C <sub>30</sub> H <sub>52</sub> N <sub>4</sub> O <sub>5</sub>
<b>Formula weight (g/mol)</b>	548.75
<b>Temperature (K)</b>	200
<b>Crystal system</b>	Triclinic
<b>Space group</b>	P1
<b>a (Å)</b>	9.5490(4)
<b>b (Å)</b>	13.3560(6)
<b>c (Å)</b>	14.7285(6)
<b>α (°)</b>	108.404(2)
<b>β (°)</b>	103.569(2)
<b>γ (°)</b>	95.911(2)
<b>Volume (Å<sup>3</sup>)</b>	1700.38(13)
<b>Z</b>	2
<b>ρ<sub>calc</sub> (g/cm<sup>3</sup>)</b>	1.072
<b>Absorption coefficient μ (mm<sup>-1</sup>)</b>	0.581 (CuKα)
<b>F(000)</b>	600
<b>Crystal size (mm<sup>2</sup>)</b>	0.50 × 0.31 × 0.13
<b>Wavelength λ (Å)</b>	1.54178
<b>2Θ range (°)</b>	6.594 - 133.104
<b>Miller indexes ranges</b>	-11 ≤ h ≤ 11, -15 ≤ k ≤ 15, -17 ≤ l ≤ 17
<b>Measured reflections</b>	57746
<b>Unique reflections</b>	11639
<b>R<sub>int</sub> / R<sub>sigma</sub></b>	0.0285 / 0.0175
<b>Reflections [I ≥ 2σ(I)]</b>	11272
<b>Restraints</b>	1142
<b>Parameters</b>	1102
<b>Goodness-of-fit F<sup>2</sup></b>	1.027
<b>Final R indexes<sup>b c</sup> [all data]</b>	R1 = 0.0509, wR2 = 0.1421
<b>Final R indexes<sup>b c</sup> [I ≥ 2σ(I)]</b>	R1 = 0.0498, wR2 = 0.1400
<b>Largest diff. peak/hole (e/Å<sup>3</sup>)</b>	0.25 / -0.24
<b>Flack parameter</b>	0.01(12)

<sup>a</sup> Including solvent molecules (if presence)

$$_{\text{b}} \quad R1 = \sum ||F_o| - |F_c|| / \sum |F_o|$$

$$_{\text{c}} \quad wR2 = \sqrt{\sum (w(F_o^2 - F_c^2)) / \sum (w(F_o^2)^2)}$$

**Table S4. Hydrogen Bond geometric parameters (lengths and angle) for 18a.***Calculated by software PLATON.*

H-bonds <b>D-H···A</b>	Lengths (Å)			Angle (°) [D-H···A]
	d(D-H)	d(H···A)	d(D···A)	
N1-H1···O8 <sup>#</sup>	0.94(5)	1.99(5)	2.903(3)	162(4)
(i) N2A-H2A···O10 <sup>##</sup>	0.98(6)	1.91(6)	2.803(7)	150(6)
(i) N2B-H2B···O10 <sup>##</sup>	0.99(3)	2.13(10)	2.839(11)	127(10)
N3-H3···O7	0.97(3)	2.14(4)	2.921(3)	137(3)
N4-H4···O9 <sup>#</sup>	0.99(3)	1.92(3)	2.833(4)	153(4)
N5-H5···O3 <sup>###</sup>	0.95(4)	1.92(4)	2.840(4)	164(4)
(ii) N6A-H6A···O2	1.00(11)	1.94(11)	2.93(2)	168(11)
(ii) N6B-H6B···O2	1.01(15)	1.79(13)	2.74(3)	156(15)
N7-H7···O5	0.95(3)	1.87(3)	2.762(3)	155(4)
(iii) N8A-H8AA···O4 <sup>###</sup>	0.97(6)	1.95(6)	2.870(6)	159(6)
(iii) N8B-H8BA···O4 <sup>###</sup>	0.984(17)	1.961(2)	2.835(15)	146(10)

Symmetry codes:  
 (#) - $I+x, y, z$   
 (##) - $I+x, y, -I+z$   
 (###)  $I+x, y, z$

**D** (donor), **A** (acceptor), (i), (ii) and (iii) correspond to atoms with a partial occupancy.