Induction of Ionic Smectic C Phases: A Systematic Study of Alkyl-linked Guanidinium-based Liquid Crystals – Electronic Supporting Information

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Experimental Section

Materials and Equipment

All chemicals were, unless stated otherwise, provided by the supplier and used without further purification. Dry solvents were dried by conventional laboratory methods. The eluents for chromatography petroleum ether (low-boiling) and ethyl acetate were distilled prior to use.

For thinlayer chromatography silica gel 60 F254 glass plates with a layer thickness of 0.25 mm on aluminium (pore size 60 Å) from the company Merck were used. Column chromatography was performed using silica gel with a particle diameter of 40 – 60 µm from the company Fluka.

¹H NMR spectra were measured using the Bruker Avance 300 and Bruker Avance 500 spectrometers at 300 MHz and 500 MHz, respectively and ¹³C-NMR spectra at 75 MHz and 126 MHz, respectively. Deuterated chloroform (CDCl₃) and deuterated dimethyl sulfoxide (DMSO-d₆) were used as solvents. The chemical shift δ in ppm (parts per million) refers to the standard tetramethylsilane. The numbering of atoms can be different from the IUPAC nomenclature in order to ensure better comparison of various compounds. In order to assign the signals of the ¹H and ¹³C NMR spectra, COSY, HSQC and HMBC measurements were carried out.

FT-IR spectra were measured on a Bruker Vektor 22, with a MKII Golden Gate Single Reflection Diamond ATR. Mass spectra (MS) and high resolution mass spectra (HRMS) were measured by electrospray ionisation (ESI) with a Bruker MicrOTOF-Q spectrometer, as well as electron ionisation (EI) with a Bruker Varian MAT 711 spectrometer.

For elemental analyses, an Elemental Analyzer Model 1106 from the company Carlo Erba Strumentazione was used.

Melting points as well as mesophase textures were, unless otherwise stated, determined using a polarising optical microscope Olympus BX 50, which was heated via a hot plate Linkam LTS. The temperature regulation was carried out with the control units TP93 and LNP from the company Linkam ($\Delta T = \pm 1$ K).

For differential scanning calorimetry, a DSC822e from the company Mettler Toledo was used. The compounds were analysed in 40 μ l sealed aluminium crucibles with heating and cooling rates of 5 K min⁻¹ and the phase transition temperatures of the extrapolated onset values were determined using the software STARe 7.01.

Temperature-dependant X-ray diffraction measurements were performed using Bruker AXS Nanostar C with a ceramic tube generator (1500 W) with cross-coupled Goebel mirrors providing monochromatic Cu K $_{\alpha 1}$ radiation (1.5405 Å). For all measurements, the compounds were melted in a glass capillary from the company Hilgenberg GmbH (external diameter of 0.7 mm). Diffraction patterns were measured using a Bruker HI-STAR detector. Calibration before each measurement was carried out using the diffraction pattern of silver behenate at room temperature. The analysis of measured values was performed using the software SAXS from the company Bruker. The obtained diffraction patterns were analysed using the software Datasqueeze and QtiPlot.

TGA measurements were carried out on a Netzsch STA 449 C Jupiter using aluminium oxide crucibles and a synthetic air atmosphere with a heating rate of 5 K min⁻¹.

Synthesis of the *Ortho*-substituted Derivative ArGua(10,10)Cl

Tert-butyl (2-hydroxyphenyl)carbamate **4**

Following a procedure by Dumez,[1] 2-aminophenol **3** (5.00 g, 45.8 mmol) was suspended in dry THF (50 mL). Di-*tert*-butyl dicarbonate (10.5 g, 48.1 mmol) was added dropwise and the reaction was stirred for 50 h at room temperature. The solvent was removed under reduced pressure and the solid was dissolved in Et₂O and washed with citric acid. The combined organic phases were washed with brine and dried with MgSO₄. The solvent was removed under reduced pressure to yield **4** as a colourless solid (8.35 g, 39.9 mmol, 87 %).

¹H-NMR (300 MHz, CDCl₃): δ = 1.51 (s, 9H, C(CH₃)₃), 6.77–7.03 (m, 4H, 3-H, 4-H, 5-H, 6-H), 7.23 (s, 1H, NH), 8.27 (s, 1H, OH) ppm; ¹³C-NMR (75 MHz, CDCl₃) δ = 27.6, 28.3, 82.0 (C(CH₃)₃), 118.4, 120.8, 121.2, 125.3, 125.7, 147.1, 155.0 (C=O) ppm.

The spectroscopic data are in agreement with the literature values.[2]

Tert-butyl (2-((10-bromodecyl)oxy)phenyl)carbamate **5**

Following a procedure by Haenle,[3] *tert*-butyl (2-hydroxyphenyl)carbamate **4** (3.00 g, 14.3 mmol) and K₂CO₃ (5.94 g, 43.0 mmol) were suspended in MeCN (50 mL), and 1,10-dibromodecane (12.9 g, 43.0 mmol) was added dropwise to the suspension. The

reaction mixture was heated under reflux for 49 h, then cooled to room temperature and the precipitate was removed by filtration. The MeCN phase was discarded and the precipitate was washed with a (1 : 1) mixture of CH₂Cl₂ / EtOAc (500 mL). The organic phase was concentrated under reduced pressure and purified by column chromatography (eluent: PE / EtOAc = (30 : 1) → (1 : 0)) to yield **5** as a colourless liquid (4.05 g, 9.44 mmol, 66 %).

¹H-NMR (500 MHz, CDCl₃): δ = 1.25–1.48 (m, 12H, 6 × CH₂), 1.53 (s, 9H, C(CH₃)₃), 1.76–1.89 (m, 4H, OCH₂CH₂, BrCH₂CH₂), 3.39 (t, *J* = 6.8 Hz, 2H, BrCH₂), 3.99 (t, *J* = 6.6 Hz, 2H, OCH₂), 6.78–6.86 (m, 1H, 3-H), 6.88–6.97 (m, 2H, 4-H, 5-H), 7.06 (s, 1H, NH), 8.01–8.12 (m, 1H, 6-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 26.0, 28.1, 28.4, 28.7, 29.1, 29.3, 29.3, 29.4, 29.4, 32.8 (8 × CH₂, 3 × CH₃), 34.0 (BrCH₂), 68.6 (OCH₂), 80.2 (C(CH₃)₃), 110.9 (C-3), 118.0 (C-6), 120.9 (C-5), 122.2 (C-4), 128.2 (C-1), 146.8 (C-2), 152.7 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 3436 (w), 2929 (w), 2855 (w), 2251 (w), 1992 (w), 1725 (m), 1601 (w), 1519 (s), 1482 (w), 1448 (m), 1392 (w), 1367 (w), 1334 (w), 1291 (w), 1235 (m), 1206 (w), 1154 (s), 1119 (w), 1047 (m), 1024 (m), 907 (s), 834 (w), 728 (s), 647 (w), 595 (w) cm⁻¹; MS (ESI): *m/z* = 450 [M⁺ + Na⁺]; HRMS (ESI): *m/z* for C₂₁H₃₄BrNO₃Na⁺ calc.: 450.1625 [M + Na⁺], found: 450.1605.

Tert-butyl (2-((10-((4'-(decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)oxy)phenyl)carbamate

7

Following a procedure by Haenle,[3] 4'-(dodecyloxy)-[1,1'-biphenyl]-4-ol **6a** (526 mg, 1.61 mmol), *tert*-butyl (2-((10-bromodecyl)oxy)phenyl)carbamate **5** (840 mg, 1.96 mmol) and K₂CO₃ (774 mg, 5.60 mmol) were heated under reflux in MeCN (45 mL) for 25 h. After cooling to 0 °C, the residual solid was removed by filtration, washed with cold MeCN (2 × 50 mL) and taken up in Water (50 mL) and CH₂Cl₂ (50 mL). The layers were separated and the aqueous layer was extracted with CH₂Cl₂ (3 × 30 mL) and dried with MgSO₄. The solvent was removed under reduced pressure to yield **7** as a colourless solid (999 mg, 1.45 mmol, 90 %).

¹H-NMR (500 MHz, CDCl₃): δ = 0.88 (t, *J* = 6.6 Hz, 3H, CH₃), 1.16–1.70 (m, 35H, 13 × CH₂, C(CH₃)₃), 1.72–1.94 (m, 6H, 3 × OCH₂CH₂), 3.80–4.19 (m, 6H, 3 × OCH₂), 6.80–6.87 (m, 1H, 3-H), 6.89–7.01 (m, 6H, 3-H', 3-H'', 4-H, 5-H), 7.07 (s, 1H, NH), 7.40–7.53 (m, 4H, 2'-H, 2''-H), 8.02–8.12 (m, 1H, 6-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 26.0, 26.1, 28.4, 29.1, 29.3, 29.3, 29.3, 29.4, 29.4, 29.5, 29.6, 29.6, 31.9 (16 × CH₂), 68.0, 68.1, 68.7 (3 × OCH₂), 80.2 (C(CH₃)₃), 111.0 (C-3), 114.7, 114.7 (C-3', C-3''), 118.0 (C-6), 120.9 (C-5), 122.2 (C-4), 127.6 (C-2', C-2''), 128.2 (C-1), 133.3, 133.3 (C-1', C-1''), 146.9 (C-2), 152.8 (C=O), 158.2, 158.2 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3441 (w), 2919 (m), 2850 (w), 1734 (w), 1708 (w), 1604 (w), 1569 (w), 1519 (w), 1500 (m), 1475 (w), 1448 (m), 1392 (w), 1367 (w), 1329 (m), 1273 (w), 1244 (s), 1154 (s), 1120 (w), 1034 (w), 996 (w), 909 (w), 824 (s), 809 (w), 767 (w), 740 (m), 646 (w), 595 (w), 519 (w) cm⁻¹; MS (ESI): *m/z* = 696 [M⁺ + Na⁺]; HRMS (ESI): *m/z* for C₄₃H₆₃NO₅Na⁺ calc.: 696.4598 [M + Na⁺], found: 696.4583; CHN for C₄₃H₆₃NO₅: calc. C 76.63 H 9.42 N 2.08, found C 76.60 H 9.76 N 2.03.

2-((10-((4'-(Decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)oxy)aniline **8**

Following a procedure by Haenle,[3] *tert*-butyl (2-((10-((4'-(decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)oxy)phenyl)carbamate **7** (250 mg, 371 μ mol) was stirred in CH_2Cl_2 (10 mL) and methanesulphonic acid (144 μ L, 214 mg, 2.22 mmol) was added dropwise to the reaction mixture. After 1 h, a solution of K_2CO_3 (615 mg, 4.45 mmol) in water (20 mL) was added to the reaction mixture. The reaction mixture was extracted with CH_2Cl_2 (4×20 mL) and dried with MgSO_4 . The solvent was removed under reduced pressure. Purification via column chromatography (eluent: PE / EtOAc = (1 : 10) $\rightarrow$ (2 : 1)) and removal of the solvent under reduced pressure yielded **8** as a colourless solid (1.84 g, 321 μ mol, 86 %).

^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 7.1 Hz, 3H, CH_3), 1.19–1.53 (m, 26H, $13 \times \text{CH}_2$), 1.74–1.85 (m, 6H, $3 \times \text{OCH}_2\text{CH}_2$), 3.98 (t, J = 6.5 Hz, 6H, $3 \times \text{OCH}_2$), 6.67–6.74 (m, 2H, 4-H, 6-H), 6.74–6.81 (m, 2H, 3-H, 5-H), 6.90–6.98 (m, 4H, 3'-H, 3''-H), 7.41–7.51 (m, 4H, 2'-H, 2''H) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14. (CH_3), 22.7, 26.1, 26.1, 26.1, 29.3, 29.3, 29.4, 29.4, 29.4, 29.5, 29.6, 29.6, 31.9 ($16 \times \text{CH}_2$), 68.1, 68.1, 68.2 ($3 \times \text{OCH}_2$), 111.4 (C-3), 114.7 (C-3', C-3''), 115.0 (C-6), 118.5 (C-4), 120.9 (C-5), 127.6 (C-2', C-2''), 133.3, 133.3 (C-1', C-1''), 136.2 (C-1), 146.8 (C-2), 158.2, 158.2 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3454 (w), 3365 (w), 3040 (w), 2954 (w), 2933 (w), 2920 (s), 2873 (w), 2851 (m), 1607 (w), 1569 (w), 1500 (m), 1475 (w), 1463 (w), 1394 (w), 1342 (w), 1273 (s), 1249 (m), 1220 (m), 1178 (w), 1138 (w), 1079 (w), 1044 (w), 1033 (w), 1011 (w), 995 (w), 980 (w), 913 (w), 824 (s), 809 (w), 781 (w), 742 (w), 731 (m), 646 (w), 595 (w), 555 (w), 517 (w) cm^{-1} ; MS (ESI): m/z = 574 [$\text{M}^+ + \text{H}^+$]; HRMS (ESI): m/z for

$\text{C}_{38}\text{H}_{56}\text{NO}_3^+$ calc.: 574.4255 $[\text{M} + \text{H}^+]$, found: 574.4254; CHN for $\text{C}_{38}\text{H}_{55}\text{NO}_3$: calc. C 79.53 H 9.66 N 2.44, found C 79.54 H 9.71 N 2.39.

N-(bis(dimethylamino)methylene)-2-((10-((4'-(decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)oxy)benzenaminium chloride **ArGua(10,10)Cl**

Following a procedure by Butschies,[4] 2-((10-((4'-(decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)oxy)aniline **8** (170 mg, 296 μmol) was stirred with chloroformamidinium chloride (3.91 mL, 504 μmol) in dry CH_2Cl_2 (6 mL) for 4 h. Water (two droplets) was added and the reaction mixture was stirred intensely (10 min). The reaction mixture was filtered and the solvent was removed under reduced pressure. Purification via column chromatography was performed using silica gel treated with hydrochloric acid.¹ The column was flushed with pure EtOAc, then CH_2Cl_2 / MeOH = (10 : 1) $\rightarrow$ (20 : 1). The solvent was removed under reduced pressure to yield **ArGua(10,10)Cl** as a colourless solid (199 mg, 281 μmol , 95 %).

^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.7 Hz, 3H, CH_3), 1.18–1.53 (m, 38H, $13 \times \text{CH}_2$, $2 \times \text{N}(\text{CH}_3)_2$), 1.62–1.86 (m, 6H, $3 \times \text{OCH}_2\text{CH}_2$), 3.89–4.04 (m, 6H, $3 \times \text{OCH}_2$), 6.84–7.03 (m, 6H, 3-H, 3'-H, 3''-H, 6-H), 7.11–7.18 (m, 1H, 5-H), 7.41–7.51 (m, 4H, 2'-H, 2''H), 7.56–7.64 (m, 1H, 4-H), 11.75 (s, 1H, NH) ppm;
 ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1. (CH_3), 22.7, 25.8, 26.1, 29.3, 29.3, 29.3, 29.4,

¹ For further details, see GP 5.

29.5, 29.5, 29.6, 29.6, 31.9 ($16 \times \text{CH}_2$), 68.0, 68.1, 69.0 ($3 \times \text{OCH}_2$), 100.0, 112.3 (C-3), 114.7, 114.7 (C-3', C-3''), 122.1 (C-6), 125.7 (C-5), 126.7, 127.0 (C-4), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1, C-1', C-1''), 151.4 (C-2), 158.2, 158.3 (C-4', C-4''), 160.5 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu} = 3393$ (w), 2919 (m), 2851 (w), 2187 (w), 1890 (w), 1607 (m), 1565 (w), 1499 (s), 1473 (w), 1402 (w), 1321 (w), 1272 (m), 1245 (s), 1176 (w), 1119 (w), 1036 (w), 1013 (w), 996 (w), 826 (m), 807 (w), 727 (s), 640 (w), 621 (w), 596 (w), 516 (w) cm^{-1} ; MS (ESI): $m/z = 673$ [M^+]; HRMS (ESI): m/z for $\text{C}_{43}\text{H}_{66}\text{N}_3\text{O}_3^+$ calc.: 672.5099 [M^+], found: 672.5106; CHN for $\text{C}_{43}\text{H}_{66}\text{ClN}_3\text{O}_3$: calc. C 72.90 H 9.39 N 5.93, found C 72.77 H 9.39 N 5.80.

Synthesis of the Alkyl-linked Gudnidinium Salts Gua(10,10)X

1,14-Tetradecanediol **14**

Following a procedure by Girlanda-Junges,[5] a solution of 1,14-tetradecanedioic acid (7.88 g, 30.5 mmol) in dry THF (250 mL) was added slowly to a suspension of LiAlH_4 (3.47 g, 91.6 mmol) in dry THF (80 mL) at 0 °C. Once the addition had finished, the suspension was stirred at room temperature for 3 h. After cooling to 0 °C, a solution of potassium sodium tartrate (20.00 g, 70.9 mmol) in water was added. The reaction mixture was extracted with diethylether, the combined organic phases were washed with brine and dried with MgSO_4 . The solvent was removed under reduced pressure and the product was dried under vacuum to yield **14** as a colourless solid (5.65 g, 24.5 mmol, 80 %).

^1H -NMR (300 MHz, CDCl_3): $\delta = 1.21\text{--}1.44$ (m, 22H, $10 \times \text{CH}_2$), 1.49–1.65 (m, 4H, $2 \times \text{OCH}_2\text{CH}_2$), 3.64 (t, $J = 6.6$ Hz, 4H, $2 \times \text{OCH}_2$) ppm; ^{13}C -NMR (75 MHz, CDCl_3) $\delta = 25.7, 29.4, 29.6, 29.6, 29.6, 32.8$ ($12 \times \text{CH}_2$), 63.1 ($2 \times \text{OCH}_2$) ppm. The spectroscopic data are in agreement with the literature values.[6]

1,14-Dibromotetradecane **15**

Following a procedure by Skabara,[7] 1,14-tetradecanediol (318 mg, 1.38 mmol) and PPh_3 (905 mg, 3.45 mmol) were dissolved in dry CH_2Cl_2 (10 mL). A solution of CBr_4 (946 mg, 3.04 mmol) in dry CH_2Cl_2 (10 mL) was then added slowly, and the reaction was left to stir at room temperature for 3 d. The solvent was removed under reduced pressure and the crude was purified by column chromatography with PE. The solvent was removed under reduced pressure and dried under vacuum to yield **15** as a colourless solid (415 mg, 1.16 mmol, 84 %).

^1H -NMR (300 MHz, CDCl_3): δ = 1.17–1.51 (m, 20H, $10 \times \text{CH}_2$), 1.79–1.93 (m, 4H, $2 \times \text{BrCH}_2\text{CH}_2$), 3.41 (t, J = 6.9 Hz, 4H, $2 \times \text{BrCH}_2$) ppm; ^{13}C -NMR (75 MHz, CDCl_3) δ = 28.2, 28.8, 29.4, 29.5, 29.6, 32.8 ($14 \times \text{CH}_2$), 34.1 ($2 \times \text{BrCH}_2$) ppm. The spectroscopic data are in agreement with the literature values.[8]

General Procedure for the Formation of the Alkoxy-biphenyl Derivatives 6a–e

Following a procedure by Benbayer,[9] 4,4'-dihydroxybiphenyl **9** (53.7 mmol) and KOH (162 mmol) were stirred in DMSO (50 mL) at room temperature for 30 min. Slow addition of the corresponding alkyl bromide (59.1 mmol) followed, after which the reaction mixture was left to stir for 63 h. After checking for completion using TLC, the reaction mixture was acidified with HCl to pH = 1, whereby a solid precipitated. This solid was filtered, washed with water, PE and recrystallized from EtOAc, EtOH or purified via column chromatography.

4'-(Hexyloxy)-[1,1'-biphenyl]-4-ol **6b**

Setup: 4,4'-Dihydroxybiphenol **9** (7.35 g, 39.5 mmol), 1-bromohexane (7.17 g, 43.4 mmol), KOH (6.65 g, 118 mmol)

Column: PE : CH_2Cl_2 = (1 : 10) $\rightarrow$ (2 : 3)

Colourless solid (31 %, 3.27 g, 12.1 mmol); ^1H -NMR (300 MHz, DMSO): δ = 0.88 (t, J = 6.9 Hz, 3H, CH_3), 1.20–1.51 (m, 6H, $3 \times CH_2$), 1.59–1.84 (m, 2H, OCH_2CH_2), 3.94 (t, J = 6.5 Hz, 2H, OCH_2), 6.69–7.05 (m, 4H, 3'-H, 3''-H), 7.29–7.57 (m, 4H, 2'-H, 2''-H), 9.47 (s, 1H, OH) ppm; ^{13}C -NMR (75 MHz, DMSO) δ = 13.9 (CH_3), 22.1, 25.2, 28.7, 31.0 ($4 \times CH_2$), 67.4 (OCH_2), 114.6, 115.6 (C-3', C-3''), 126.9, 127.1 (C-2', C-2''), 130.7, 132.5 (C-1', C-1''), 156.4, 157.5 (C-4', C-4'') ppm. The spectroscopic data are in agreement with the literature values.[10]

4'-(Octyloxy)-[1,1'-biphenyl]-4-ol **6c**

Setup: 4,4'-Dihydroxybiphenol **9** (10.0 g, 53.7 mmol), 1-bromooctane (11.4 g, 59.1 mmol), KOH (9.06 g, 161 mmol)

Workup: Hot filtration in methanol.

Colourless solid (24 %, 3.88 g, 13.0 mmol); ^1H -NMR (300 MHz, DMSO): δ = 0.86 (t, J = 6.8 Hz, 3H, CH_3), 1.18–1.49 (m, 10H, $5 \times CH_2$), 1.71 (tt, J = 6.5 Hz, 6.5 Hz, 2H, OCH_2CH_2), 3.96 (t, J = 6.5 Hz, 2H, OCH_2), 6.75–7.00 (m, 4H, 3'-H, 3''-H), 7.34–7.54 (m,

4H, 2'-H, 2''-H), 9.43 (s, 1H, OH) ppm; ^{13}C -NMR (75 MHz, DMSO) δ = 13.9 (CH_3), 22.1, 25.5, 28.6, 28.7, 28.7, 31.2 ($6 \times \text{CH}_2$), 67.4 (OCH_2), 114.7, 115.6 (C-3', C-3''), 126.9, 127.1 (C-2', C-2''), 130.7, 132.6 (C-1', C-1''), 156.4, 157.5 (C-4', C-4'') ppm.

The spectroscopic data are in agreement with the literature values.[10]

4'-(Decyloxy)-[1,1'-biphenyl]-4-ol 6a

Setup: 4,4'-Dihydroxybiphenol **9** (7.14 g, 38.3 mmol), 1-bromodecane (9.34 g, 42.2 mmol), KOH (6.53 g, 115 mmol)

Column: PE : CH_2Cl_2 = (0 : 1) $\rightarrow$ (1 : 3)

Colourless solid (19 %, 2.36 g, 7.23 mmol); ^1H -NMR (500 MHz, DMSO, 373 K): δ = 0.86 (t, J = 6.9 Hz, 3H, CH_3), 1.19–1.48 (m, 14H, $7 \times \text{CH}_2$), 1.67–1.75 (m, 2H, OCH_2CH_2), 3.97 (t, J = 6.5 Hz, 2H, OCH_2), 6.79–6.95 (m, 4H, 3'-H, 3''-H), 7.34–7.47 (m, 4H, 2'-H, 2''-H), 9.05 (s, 1H, OH) ppm; ^{13}C -NMR (126 MHz, DMSO, 373 K) δ = 13.6 (CH_3), 21.9, 25.5, 28.5, 28.7, 28.8, 28.8, 28.9, 31.2 ($8 \times \text{CH}_2$), 67.9 (OCH_2), 115.0, 115.7 (C-3', C-3''), 126.9, 127.1 (C-2', C-2''), 131.1, 133.0 (C-1', C-1''), 156.5, 157.7 (C-4', C-4'') ppm. The spectroscopic data are in agreement with the literature values.[10]

4'-(Dodecyloxy)-[1,1'-biphenyl]-4-ol 6d

Setup: 4,4'-Dihydroxybiphenol **9** (10.0 g, 53.7 mmol), 1-bromododecane (14.7 g, 59.1 mmol), KOH (9.12 g, 162 mmol)

Workup: Recrystallized from EtOAc.

Colourless solid (77 %, 14.6 g, 41.2 mmol); $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ = 0.87 (t, J = 6.5 Hz, 3H, CH_3), 1.15–1.53 (m, 18H, $9 \times \text{CH}_2$), 1.78 (tt, J = 6.5 Hz, 6.5 Hz, 2H, OCH_2CH_2), 3.98 (t, J = 6.5 Hz, 2H, OCH_2), 6.76–6.97 (m, 4H, 3'-H, 3''-H), 7.30–7.48 (m, 4H, 2'-H, 2''-H) ppm; $^{13}\text{C-NMR}$ (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.1, 29.4, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 32.0 ($10 \times \text{CH}_2$), 68.3 (OCH_2), 115.0, 115.0, 115.7 (C-3', C-3''), 127.7, 127.7, 128.0 (C-2', C-2''), 133.4, 134.0 (C-1', C-1''), 154.7, 158.5 (C-4', C-4'') ppm. The spectroscopic data are in agreement with the literature values.[10]

4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-ol 6e

Setup: 4,4'-Dihydroxybiphenol **9** (7.58 g, 40.7 mmol), 1-bromotetradecane (12.4 g, 44.8 mmol), KOH (6.85 g, 112 mmol)

Column: PE : CH_2Cl_2 = (0 : 1) $\rightarrow$ (1 : 5)

Colourless solid (60 %, 9.41 g, 2.46 mmol); $^1\text{H-NMR}$ (500 MHz, DMSO, 373 K): δ = 0.74–0.84 (m, 3H, CH_3), 1.10–1.43 (m, 22H, $11 \times \text{CH}_2$), 1.60–1.71 (m, 2H, OCH_2CH_2), 3.92 (t, J = 6.5 Hz, 2H, OCH_2), 6.67–7.03 (m, 4H, 3'-H, 3''-H), 7.18–7.60 (m, 4H, 2'-H, 2''-H), 8.99 (s, 1H, OH) ppm; $^{13}\text{C-NMR}$ (126 MHz, DMSO) δ = 14.1 (CH_3), 22.4, 26.0, 29.0, 29.1, 29.3, 29.3, 29.4, 30.9, 31.7 ($12 \times \text{CH}_2$), 68.4 (OCH_2), 115.5, 116.2 (C-3', C-3''), 127.4, 127.6 (C-2', C-2''), 131.5, 133.5 (C-1', C-1''), 157.0, 158.2 (C-4', C-4'') ppm.

The spectroscopic data are in agreement with the literature values.[10]

General Procedure for the Spacer Formation by Bromoalkylation of Potassium Phthalamide 11a–e

Following a procedure by Kong,[11] potassium phthalamide **10** (4.27 mmol) was heated with dibromoalkane (19.2 mmol) in DMF (25 mL) at 100 °C for 30 h. The reaction mixture was co-evaporated with toluene (2 × 25 mL) under reduced pressure followed by purification via column chromatography.

2-(6-Bromohexyl)isoindoline-1,3-dione 11a

Setup: Potassium phthalamide **10** (6.47 g, 34.5 mmol), 1,6-dibromohexane (51.7 g, 162.8 mmol)

Column: PE : EtOAc = (0 : 1) → (1 : 0), followed by recrystallization from EtOH.

Colourless solid (27 %, 2.97 g, 9.57 mmol); ^1H -NMR (300 MHz, $CDCl_3$): δ = 1.31–1.56 (m, 4H, 2 × CH_2), 1.63–1.77 (m, 2H, NCH_2CH_2), 1.79–1.94 (m, 2H, $BrCH_2CH_2$), 3.40 (t, J = 6.8 Hz, 2H, $BrCH_2$), 3.69 (t, J = 7.2 Hz, 2H, NCH_2), 7.67–7.77 (m, 2H, 3-H), 7.80–7.90 (m, 2H, 2-H) ppm; ^{13}C -NMR (75 MHz, $CDCl_3$) δ = 26.0, 27.7, 28.4, 32.6 (4 × CH_2), 33.7 ($BrCH_2$), 37.8 (NCH_2), 123.2 (C-2), 132.1 (C-1), 133.9 (C-3), 168.4 (C=O) ppm.

The spectroscopic data are in agreement with the literature values.[12]

2-(8-Bromooctyl)isoindoline-1,3-dione 11b

Setup: Potassium phthalamide **10** (5.94 g, 32.1 mmol), 1,8-dibromooctane (39.3 g, 144 mmol)

Column: PE : EtOAc = (5 : 1) $\rightarrow$ (1 : 0)

Colourless solid (67 %, 7.21 g, 21.3 mmol); $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ = 1.22–1.49 (m, 8H, $4 \times \text{CH}_2$), 1.59–1.75 (m, 2H, NCH_2CH_2), 1.84 (tt, J = 6.8 Hz, 6.8 Hz, 2H, BrCH_2CH_2), 3.39 (t, J = 6.8 Hz, 2H, BrCH_2), 3.68 (t, J = 7.5 Hz, 2H, NCH_2), 7.67–7.76 (m, 2H, 3-H), 7.80–7.89 (m, 2H, 2-H) ppm; $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ = 26.7, 28.1, 28.5, 28.6, 28.9 ($5 \times \text{CH}_2$), 32.7 (NCH_2CH_2), 34.0 (BrCH_2), 38.0 (NCH_2), 123.1 (C-2), 132.2 (C-1), 133.8 (C-3), 168.4 (C=O) ppm. The spectroscopic data are in agreement with the literature values.[12]

*2-(10-Bromodecyl)isoindoline-1,3-dione **IIc***

Setup: Potassium phthalimide **10** (6.32 g, 34.1 mmol), 1,10-dibromodecane (48.9 g, 163 mmol)

Column: PE : EtOAc = (0 : 1) $\rightarrow$ (1 : 0), followed by recrystallization from EtOH.

Colourless solid (64 %, 8.02 g, 21.9 mmol); $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ = 1.17–1.51 (m, 12H, $6 \times \text{CH}_2$), 1.59–1.74 (m, 2H, NCH_2CH_2), 1.77–1.93 (m, 2H, BrCH_2CH_2), 3.40 (t, J = 6.9 Hz, 2H, BrCH_2), 3.68 (t, J = 6.6 Hz, 2H, NCH_2), 7.67–7.76 (m, 2H, 3-H), 7.79–7.89 (m, 2H, 2-H) ppm; $^{13}\text{C-NMR}$ (75 MHz, CDCl_3) δ = 26.8, 28.1, 28.6, 28.7, 29.1, 29.3, 29.3, 29.3, 32.8 ($8 \times \text{CH}_2$), 34.0 (BrCH_2), 38.0 (NCH_2), 123.1 (C-2), 132.1 (C-1), 133.8 (C-3), 168.4 (C=O) ppm. The spectroscopic data are in agreement with the literature values.[12]

2-(12-Bromododecyl)isoindoline-1,3-dione **11d**

Setup: Potassium phthalamide **10** (3.03 g, 16.3 mmol), 1,12-dibromododecane (24.1 g, 73.5 mmol)

Column: PE : EtOAc = (8 : 1) → (1 : 0)

Colourless solid (67 %, 4.31 g, 10.9 mmol); ¹H-NMR (300 MHz, CDCl₃): δ = 1.17–1.49 (m, 16H, 8 × CH₂), 1.60–1.74 (m, 2H, NCH₂CH₂), 1.85 (tt, *J* = 6.8 Hz, 6.8 Hz, 2H, BrCH₂CH₂), 3.40 (t, *J* = 6.8 Hz, 2H, BrCH₂), 3.67 (t, *J* = 7.4 Hz, 2H, NCH₂), 7.66–7.75 (m, 2H, 3-H), 7.80–7.88 (m, 2H, 2-H) ppm; ¹³C-NMR (75 MHz, CDCl₃) δ = 26.8, 28.2, 28.6, 28.7, 29.2, 29.4, 29.4, 29.5, 29.5, 32.8 (NCH₂CH₂), 34.1 (BrCH₂), 38.1 (NCH₂), 123.1 (C-2), 132.2 (C-1), 133.8 (C-3), 168.5 (C=O) ppm. The spectroscopic data are in agreement with the literature values.[12]

2-(14-Bromotetradecyl)isoindoline-1,3-dione **11e**

Setup: Potassium phthalamide **10** (790 mg, 4.27 mmol), 1,10-dibromotetradecane (6.84 g, 19.2 mmol)

Column: PE : EtOAc = (1 : 5) → (80 : 1)

Colourless solid (73 %, 1.32 g, 3.12 mmol); ¹H-NMR (300 MHz, CDCl₃): δ = 1.15–1.49 (m, 20H, 10 × CH₂), 1.61–1.74 (m, 2H, NCH₂CH₂), 1.79–1.93 (m, 2H, BrCH₂CH₂), 3.41 (t, *J* = 6.9 Hz, 2H, BrCH₂), 3.67 (t, *J* = 7.2 Hz, 2H, NCH₂), 7.67–7.75 (m, 2H, 3-H), 7.80–7.88 (m, 2H, 2-H) ppm; ¹³C-NMR (75 MHz, CDCl₃) δ = 26.9, 28.2, 28.6, 28.8, 29.2, 29.4, 29.5, 29.5, 29.5, 29.6, 32.9 (12 × CH₂), 34.1 (BrCH₂), 38.1 (NCH₂), 123.1 (C-2),

132.2 (C-1), 133.8 (C-3), 168.5 (C=O) ppm. The spectroscopic data are in agreement with the literature values.[13]

General Procedure for Attachment of the Alkoxy-biphenyl Core 6 to the Spacer 11

Following a procedure by Haenle,[3] biphenyl **6** (5.65 mmol) was heated under reflux in MeCN (110 mL) with the spacer **11** (7.05 mmol) and K₂CO₃ (22.6 mmol) for 18 h. The reaction mixture was cooled down, the precipitate was filtered and washed with cold MeCN, and then with warm CH₂Cl₂. The solvent from the CH₂Cl₂ phase was removed under reduced pressure and the solid was dried under vacuum to yield **12a-i**.

2-(6-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexyl)isoindoline-1,3-dione 12a

Setup: 4'-(Hexyloxy)-[1,1'-biphenyl]-4-ol **6b** (505 mg, 1.87 mmol), 2-(6-bromohexyl)isoindoline-1,3-dione **11a** (722 mg, 2.33 mmol), K₂CO₃ (1.03 g, 7.47 mmol)

Colourless solid (69 %, 648 mg, 1.36 mmol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.91 (t, *J* = 7.0 Hz, 3H, CH₃), 1.29–1.67 (m, 10H, 5 × CH₂), 1.72 (tt, *J* = 7.3 Hz, 7.3 Hz, 2H, NCH₂CH₂), 1.74–1.84 (m, 4H, 2 × OCH₂CH₂), 3.70 (t, *J* = 7.3 Hz, 2H, NCH₂), 3.93–4.02 (m, 4H, 2 × biphenyl-OCH₂), 6.88–6.97 (m, 4H, 3'-H, 3''-H), 7.40–7.49 (m, 4H, 2'-H, 2''-H), 7.64–7.75 (m, 2H, 3-H), 7.79–7.89 (m, 2H, 2-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.6, 25.7, 25.7, 26.6, 28.5, 29.1, 29.3, 31.6 (8 × CH₂), 37.9 (NCH₂), 67.8, 68.1 (2 × OCH₂), 114.7 (C-3', C-3''), 123.2 (C-2), 127.6 (C-2', C-2''), 132.1 (C-1), 133.3, 133.3 (C-1', C-1''), 133.8 (C-3), 158.1, 158.2 (C-4', C-4''), 168.4 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2930 (w), 2858 (w), 1774 (w), 1699 (s), 1606 (w),

1568 (w), 1500 (m), 1465 (w), 1435 (w), 1398 (m), 1372 (w), 1338 (w), 1271 (w), 1243 (s), 1178 (w), 1136 (w), 1049 (m), 1015 (w), 997 (w), 927 (w), 879 (w), 823 (m), 806 (w), 716 (s), 645 (w), 622 (w), 599 (w), 530 (w), 517 (w) cm^{-1} ; MS (ESI): $m/z = 522$ [$\text{M} + \text{Na}^+$]; HRMS (ESI): m/z for $\text{C}_{32}\text{H}_{38}\text{NO}_4^+$ calc.: 500.2795 [$\text{M} + \text{H}^+$], found: 500.2790; CHN for $\text{C}_{32}\text{H}_{37}\text{NO}_4$: calc. C 72.92 H 7.46 N 2.80, found C 72.97 H 7.10 N 2.58.

2-(8-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)octyl)isoindoline-1,3-dione **12b**

Setup: 4'-(Octyloxy)-[1,1'-biphenyl]-4-ol **6c** (2.00 g, 6.70 mol), 2-(8-bromooctyl)isoindoline-1,3-dione **11b** (2.83 mg, 8.38 mmol), K_2CO_3 (3.70 mg, 26.8 mmol)

Colourless solid (84 %, 3.14 g, 5.64 mol); ^1H -NMR (500 MHz, CDCl_3): $\delta = 0.89$ (t, $J = 7.1$ Hz, 3H, CH_3), 1.22–1.50 (m, 18H, $9 \times \text{CH}_2$), 1.64–1.73 (m, 2H, NCH_2CH_2), 1.74–1.83 (m, 4H, $2 \times \text{OCH}_2\text{CH}_2$), 3.65–3.71 (m, 2H, $2 \times \text{OCH}_2\text{CH}_2$), 3.93–4.01 (m, 4H, $2 \times \text{biphenyl-OCH}_2$), 6.90–6.96 (m, 4H, 3'-H, 3''-H), 7.42–7.47 (m, 4H, 2'-H, 2''-H), 7.68–7.72 (m, 2H, 3-H), 7.81–7.86 (m, 2H, 2-H) ppm; ^{13}C -NMR (126 MHz, CDCl_3) $\delta = 14.1$ (CH_3), 22.7, 26.0, 26.1, 26.8, 28.6, 29.1, 29.2, 29.3, 29.3, 29.4, 31.8, 32.7, 34.0, 38.0 (NCH_2), 68.0, 68.1 ($2 \times \text{OCH}_2$), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.3 (C-1', C-1''), 133.8, 133.8 (C-3), 158.2, 158.2 (C-4', C-4''), 168.5 (C=O) ppm; FT-IR: $\tilde{\nu} = 3041$ (w), 2923 (w), 2852 (w), 1774 (w), 1705 (s), 1606 (w), 1568 (w), 1499 (m), 1467 (w), 1436 (w), 1396 (m), 1367 (w), 1330 (w), 1273 (m), 1246 (s), 1179 (w), 1138 (w), 1058 (w), 1036 (w), 995 (w), 955 (w), 884 (w), 824 (s), 808 (w), 715 (s), 646 (w), 621 (w), 599 (w), 529 (w), 517 (w) cm^{-1} ; MS (ESI): m/z

= 578 [M + Na⁺]; HRMS (ESI): m/z for C₃₆H₄₅NO₄Na⁺ calc.: 578.3241 [M + Na⁺], found: 578.3261; CHN for C₃₆H₄₅NO₄: calc. C 78.80 H 8.16 N 2.52, found C 75.86 H 7.92 N 2.67.

2-(10-((4'-(Decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)isoindoline-1,3-dione **12c**

Setup: 4'-(Dodecyloxy)-[1,1'-biphenyl]-4-ol **6a** (500 mg, 1.53 mmol), 2-(10-bromodecyl)isoindoline-1,3-dione **11c** (701 mg, 1.91 mmol), K₂CO₃ (847 mg, 6.13 mmol)

Colourless solid (48 %, 453 mg, 740 μmol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.88 (t, J = 6.9 Hz, 3H, CH₃), 1.21–1.51 (m, 26H, 13 × CH₂), 1.62–1.73 (m, 2H, NCH₂CH₂), 1.73–1.84 (m, 4H, 2 × OCH₂CH₂), 3.67 (t, J = 7.2 Hz, 2H, NCH₂), 3.92–4.01 (m, 4H, 2 × biphenyl-OCH₂), 6.89–6.97 (m, 4H, 3'-H, 3''-H), 7.41–7.49 (m, 4H, 2'-H, 2''-H), 7.65–7.71 (m, 2H, 3-H), 7.80–7.85 (m, 2H, 2-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 26.0, 26.1, 26.8, 28.6, 29.1, 29.3, 29.3, 29.4, 29.4, 29.4, 29.6, 29.6, 31.9 (16 × CH₂), 38.0 (NCH₂), 68.0, 68.0 (2 × OCH₂), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.2, 133.3 (C-1', C-1''), 133.8 (C-3), 158.2 (C-4', C-4''), 168.4 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2919 (m), 2850 (w), 2253 (w), 1773 (w), 1707 (s), 1606 (w), 1569 (w), 1499 (m), 1467 (w), 1437 (w), 1396 (m), 1369 (w), 1329 (w), 1272 (w), 1246 (w), 1178 (w), 1137 (w), 1085 (w), 1060 (w), 1034 (w), 1011 (w), 995 (w), 908 (w), 875 (w), 823 (m), 808 (m), 714 (s), 647 (w), 621 (w), 595 (w), 553 (w), 530 (w), 518 (w) cm⁻¹; MS (EI): m/z (%) = 611 (100) [M⁺], 186 (27), 160 (9); HRMS (EI): m/z for

$C_{40}H_{53}NO_4$ calc.: 611.3975 [M^+], found: 611.3961; CHN for $C_{40}H_{53}NO_4$: calc. C 78.52 H 8.73 N 2.29, found C 78.24 H 8.74 N 2.28.

2-(12-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecyl)isoindoline-1,3-dione 12d

Setup: 4'-(Dodecyloxy)-[1,1'-biphenyl]-4-ol **6d** (1.25 g, 3.53 mol), 2-(12-bromododecyl)isoindoline-1,3-dione **11d** (1.74 g, 4.41 mmol), K_2CO_3 (1.95 g, 14.1 mmol)

Colourless solid (72 %, 1.70 g, 2.54 mol); ^1H -NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.21–1.51 (m, 34H, $17 \times CH_2$), 1.63–1.71 (m, 2H, NCH_2CH_2), 1.75–1.83 (m, 4H, $2 \times OCH_2CH_2$), 3.67 (t, J = 7.3 Hz, 2H, NCH_2), 3.98 (t, J = 6.5 Hz, 4H, $2 \times$ biphenyl- OCH_2), 6.91–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.48 (m, 4H, 2'-H, 2''-H), 7.68–7.73 (m, 2H, 3-H), 7.81–7.87 (m, 2H, 2-H) ppm; ^{13}C -NMR (126 MHz, $CDCl_3$) δ = 14.1 (CH_3), 22.7, 26.1, 26.1, 26.9, 28.6, 29.2, 29.3, 29.4, 29.4, 29.4, 29.5, 29.5, 29.5, 29.6, 29.6, 29.6, 29.7, 31.9 ($20 \times CH_2$), 38.1(NCH_2), 68.1 ($2 \times OCH_2$), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.3, 133.3 (C-1', C-1''), 133.8 (C-3), 158.2 (C-4', C-4''), 168.5 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2918 (s), 2850 (m), 1775 (w), 1711 (s), 1608 (w), 1569 (w), 1501 (m), 1474 (w), 1437 (w), 1397 (w), 1370 (w), 1329 (w), 1274 (m), 1249 (s), 1178 (w), 1137 (w), 1035 (w), 1013 (w), 894 (w), 824 (w), 809 (w), 718 (w), 623 (w), 595 (w), 531 (w) cm^{-1} ; MS (ESI): m/z = 668 [$M + H^+$]; HRMS (ESI): m/z for $C_{44}H_{62}NO_4^+$ calc.: 668.4673 [$M + H^+$], found: 668.4671; CHN for $C_{44}H_{61}NO_4$: calc. C 79.12 H 9.20 N 2.10, found C 79.20 H 9.14 N 1.89.

2-(14-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecyl)isoindoline-1,3-dione
12e

Setup: 4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-ol **6e** (362 mg, 947 μ mol), 2-(14-bromotetradecyl)isoindoline-1,3-dione **11e** (500 mg, 1.18 mmol), K_2CO_3 (534 mg, 3.86 mmol)

Colourless solid (89 %, 611 mg, 844 μ mol); ^1H -NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.20–1.51 (m, 42H, $21 \times CH_2$), 1.67 (tt, J = 7.3 Hz, 7.3 Hz, 2H, NCH_2CH_2), 1.79 (tt, J = 6.6 Hz, 6.6 Hz, 4H, $2 \times OCH_2CH_2$), 3.67 (t, J = 7.3 Hz, 2H, NCH_2), 3.98 (t, J = 6.6 Hz, 4H, $2 \times$ biphenyl- OCH_2), 6.91–6.97 (m, 4H, 3'-H, 3''-H), 7.40–7.49 (m, 4H, 2'-H, 2''-H), 7.67–7.73 (m, 2H, 3-H), 7.80–7.87 (m, 2H, 2-H) ppm; ^{13}C -NMR (126 MHz, $CDCl_3$) δ = 14.1 (CH_3), 22.7, 26.1, 26.9, 28.6, 29.2, 29.3, 29.4, 29.4, 29.5, 29.6, 29.6, 29.6, 29.7, 29.7, 29.7, 31.9 ($24 \times CH_2$), 38.1 (NCH_2), 68.1 ($2 \times OCH_2$), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.3, 133.3 (C-1', C-1''), 133.8 (C-3), 158.2 (C-4', C-4''), 168.5 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2917 (s), 2849 (s), 1775 (w), 1710 (s), 1607 (w), 1569 (w), 1501 (m), 1474 (w), 1465 (w), 1438 (w), 1398 (w), 1368 (w), 1329 (w), 1273 (m), 1249 (m), 1178 (w), 1138 (w), 1059 (w), 1035 (w), 1006 (w), 922 (w), 884 (w), 824 (m), 808 (w), 717 (w), 595 (w), 530 (w) cm^{-1} ; MS (ESI): m/z = 747 [$M + Na^+$]; CHN for $C_{48}H_{69}NO_4$: calc. C 79.62 H 9.61 N 1.93, found C 79.12 H 9.72 N 1.86.

2-(14-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecyl)isoindoline-1,3-dione **12f**

Setup: 4'-(Hexyloxy)-[1,1'-biphenyl]-4-ol **6b** (256 mg, 947 μ mol), 2-(14-bromotetradecyl)isoindoline-1,3-dione **11e** (500 mg, 1.18 mmol), K_2CO_3 (545 mg, 3.94 mmol)

Colourless solid (89 %, 518 mg, 847 μ mol); ^1H -NMR (500 MHz, $CDCl_3$): δ = 0.91 (t, J = 7.0 Hz, 3H, CH_3), 1.17–1.53 (m, 26H, $13 \times CH_2$), 1.67 (tt, J = 7.4 Hz, 7.4 Hz, 2H, NCH_2CH_2), 1.73–1.85 (m, 4H, $2 \times OCH_2CH_2$), 3.67 (t, J = 7.4 Hz, 2H, NCH_2), 3.93–4.03 (m, 4H, $2 \times$ biphenyl- OCH_2), 6.90–6.97 (m, 4H, 3'-H, 3''-H), 7.39–7.51 (m, 4H, 2'-H, 2''-H), 7.67–7.74 (m, 2H, 3-H), 7.79–7.88 (m, 2H, 2-H) ppm; ^{13}C -NMR (126 MHz, $CDCl_3$) δ = 14.1 (CH_3), 22.6, 25.7, 26.1, 26.9, 28.6, 29.2, 29.3, 29.3, 29.4, 29.5, 29.6, 29.6, 29.6, 29.6, 31.6 ($16 \times CH_2$), 38.1 (NCH_2), 68.1 ($2 \times OCH_2$), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.3, 133.3 (C-1', C-1''), 133.8 (C-3), 158.2 (C-4', C-4''), 168.5 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2917 (m), 2850 (w), 1774 (w), 1713 (s), 1606 (w), 1568 (w), 1499 (m), 1466 (w), 1435 (w), 1397 (m), 1364 (w), 1334 (w), 1271 (w), 1242 (s), 1177 (w), 1107 (w), 1089 (w), 1057 (w), 1035 (w), 996 (w), 953 (w), 935 (w), 919 (w), 883 (w), 861 (w), 823 (m), 808 (w), 720 (s), 625 (w), 597 (w), 555 (w), 531 (w) cm^{-1} ; MS (ESI): m/z = 612 [$M + H^+$]; HRMS (ESI): m/z for $C_{40}H_{54}NO_4^+$ calc.: 612.4047 [$M + H^+$], found: 612.4053; CHN for $C_{40}H_{53}NO_4$: calc. C 78.52 H 8.73 N 2.29, found C 78.22 H 8.79 N 2.23.

2-(12-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecyl)isoindoline-1,3-dione **12g**

Setup: 4'-(Octyloxy)-[1,1'-biphenyl]-4-ol **6c** (1.30 g, 4.37 mol), 2-(12-bromododecyl)isoindoline-1,3-dione **11d** (2.15 g, 5.45 mmol), K₂CO₃ (2.41 g, 17.4 mmol)

Colourless solid (96 %, 2.57 g, 4.20 mol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.89 (t, *J* = 7.2 Hz, 3H, CH₃), 1.22–1.51 (m, 26H, 13 × CH₂), 1.62–1.72 (m, 2H, NCH₂CH₂), 1.74–1.84 (m, 4H, 2 × OCH₂CH₂), 3.67 (t, *J* = 7.2 Hz, 2H, NCH₂), 3.98 (t, *J* = 6.7 Hz, 4H, 2 × biphenyl-OCH₂), 6.91–6.96 (m, 4H, 3'-H, 3''-H), 7.42–7.48 (m, 4H, 2'-H, 2''-H), 7.67–7.72 (m, 2H, 3-H), 7.81–7.86 (m, 2H, 2-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 26.1, 26.1, 26.9, 28.6, 29.2, 29.3, 29.3, 29.4, 29.5, 29.5, 29.5, 31.8 (16 × CH₂), 38.1 (NCH₂), 68.1 (2 × OCH₂), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.3, 133.3 (C-1', C-1''), 133.8 (C-3), 158.2 (C-4', C-4''), 168.5 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 3042 (w), 2920 (s), 2852 (w), 2023 (w), 1772 (w), 1710 (s), 1608 (w), 1570 (w), 1501 (m), 1474 (w), 1466 (w), 1438 (w), 1395 (w), 1367 (w), 1331 (w), 1275 (m), 1251 (m), 1179 (w), 1138 (w), 1034 (w), 1015 (w), 995 (w), 908 (w), 825 (m), 809 (w), 720 (w), 647 (w), 595 (w), 530 (w), 519 (w) cm⁻¹; MS (ESI): *m/z* = 612 [M + H⁺]; HRMS (ESI): *m/z* for C₄₀H₅₄NO₄⁺ calc.: 612.4047 [M + H⁺], found: 612.4043; CHN for C₄₀H₅₃NO₄: calc. C 78.52 H 8.73 N 2.29, found C 78.44 H 8.58 N 2.20.

2-(8-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)octyl)isoindoline-1,3-dione **12h**

Setup: 4'-(Dodecyloxy)-[1,1'-biphenyl]-4-ol **6d** (2.00 g, 5.65 mol), 2-(8-bromooctyl)isoindoline-1,3-dione **11b** (2.37 g, 7.05 mmol), K₂CO₃ (3.12 g, 22.6 mmol)

Colourless solid (96 %, 3.33 g, 5.44 mol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.88 (t, *J* = 6.9 Hz, 3H, CH₃), 1.21–1.50 (m, 26H, 13 × CH₂), 1.63–1.72 (m, 2H, NCH₂CH₂), 1.74–1.83 (m, 4H, 2 × OCH₂CH₂), 3.68 (t, *J* = 7.3 Hz, 2H, NCH₂), 3.94–4.01 (m, 4H, 2 × biphenyl-OCH₂), 6.90–6.96 (m, 4H, 3'-H, 3''-H), 7.42–7.48 (m, 4H, 2'-H, 2''-H), 7.67–7.73 (m, 2H, 3-H), 7.81–7.86 (m, 2H, 2-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 26.0, 26.1, 26.8, 28.6, 29.1, 29.2, 29.2, 29.3, 29.4, 29.4, 29.6, 29.6, 29.6, 29.7, 31.9 (16 × CH₂), 38.0 (NCH₂), 68.0, 68.1 (2 × OCH₂), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.2 (C-1), 133.3 (C-1', C-1''), 133.8 (C-3), 158.2, 158.2 2 (C-4', C-4''), 168.5 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2918 (m), 2850 (w), 1774 (w), 1702 (s), 1606 (w), 1568 (w), 1499 (s), 1466 (w), 1436 (w), 1397 (m), 1366 (w), 1329 (w), 1272 (m), 1244 (s), 1178 (w), 1137 (w), 1056 (w), 1037 (w), 994 (w), 956 (w), 881 (w), 824 (m), 808 (w), 712 (s), 623 (w), 598 (w), 531 (w), 518 (w) cm⁻¹; MS (ESI): *m/z* = 612 [M + H⁺]; HRMS (ESI): *m/z* for C₄₀H₅₄NO₄⁺ calc.: 612.4047 [M + H⁺], found: 612.4043; CHN for C₄₀H₅₃NO₄: calc. C 78.52 H 8.73 N 2.29, found C 77.39 H 8.67 N 2.22.

2-(6-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexyl)isoindoline-1,3-dione **12i**

Setup: 4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-ol **6e** (3.50 g, 9.16 mmol), 2-(6-bromohexyl)isoindoline-1,3-dione **11a** (3.55 g, 11.4 mmol), K₂CO₃ (5.54 g, 40.1 mmol)

Colourless solid (94 %, 5.25 g, 8.57 mmol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.88 (t, *J* = 6.9 Hz, 3H, CH₃), 1.21–1.57 (m, 26H, 13 × CH₂), 1.72 (tt, *J* = 7.3 Hz, 7.3 Hz, 2H, NCH₂CH₂), 1.78 (tt, *J* = 7.3 Hz, 7.3 Hz, 4H, 2 × OCH₂CH₂), 3.70 (t, *J* = 7.3 Hz, 2H, NCH₂), 3.93–4.01 (m, 4H, 2 × biphenyl-OCH₂), 6.88–6.96 (m, 4H, 3'-H, 3''-H), 7.40–7.48 (m, 4H, 2'-H, 2''-H), 7.66–7.72 (m, 2H, 3-H), 7.79–7.86 (m, 2H, 2-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 25.7, 26.1, 26.6, 28.5, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 29.7, 31.9 (16 × CH₂), 37.9 (NCH₂), 67.8, 68.1 (2 × OCH₂), 114.7 (C-3', C-3''), 123.1 (C-2), 127.6 (C-2', C-2''), 132.1 (C-1), 133.3, 133.3 (C-1', C-1''), 133.8 (C-3), 158.1, 158.2 (C-4', C-4''), 168.4 (C=O) ppm; FT-IR: $\tilde{\nu}$ = 2927 (w), 2855 (w), 2254 (w), 1772 (w), 1710 (m), 1608 (w), 1499 (w), 1467 (w), 1397 (w), 1371 (w), 1270 (w), 1240 (m), 1173 (w), 1057 (w), 904 (s), 823 (w), 724 (s), 649 (m), 600 (w), 530 (w) cm⁻¹; MS (ESI): *m/z* = 612 [M + H⁺]; HRMS (ESI): *m/z* for C₄₀H₅₄NO₄⁺ calc.: 612.4047 [M + H⁺], found: 612.4029; CHN for C₄₀H₅₃NO₄: calc. C 78.52 H 8.73 N 2.29, found C 78.37 H 8.77 N 2.20.

General Procedure for the Conversion of 18 to the Amines 13

Following an adapted procedure by Li,[14] 2-(12-((4'-(dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecyl)isoindoline-1,3 dione **12** (749 μmol) was suspended in ethanol (10 mL) and heated with N₂H₄·H₂O (74.9 mmol) in the microwave at 100 °C for 1 h. The reaction

mixture was cooled to room temperature, the precipitate was filtered and washed with cold ethanol (50 mL), then dried under vacuum to yield **13a-i**.

6-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-amine 13a

Setup: 2-(6-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexyl)isoindoline-1,3-dione, **12a** (150 mg, 300 μmol), $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (1.5 mL, 1.50 g, 30.0 mmol)

Colourless solid (60 %, 67.0 mg, 181 μmol); ^1H -NMR (500 MHz, DMSO, 373 K): δ = 0.85–0.92 (m, 3H, CH_3), 1.27–1.49 (m, 12H, $6 \times \text{CH}_2$), 1.65–1.78 (m, 4H, $2 \times \text{OCH}_2\text{CH}_2$), 2.59 (t, J = 6.5 Hz, 2H, NCH_2), 4.00 (t, J = 6.5 Hz, 4H, $2 \times \text{biphenyl-OCH}_2$), 6.78–7.07 (m, 4H, 3'-H, 3''-H), 7.29–7.59 (m, 4H, 2'-H, 2''-H) ppm. Due to the poor solubility, a high temperature ^{13}C NMR could not be obtained in adequate quality; FT-IR: $\tilde{\nu}$ = 3451 (w), 2250 (w), 2124 (w), 1657 (w), 1053 (s), 1024 (s), 1005 (s), 821 (m), 758 (m), 622 (w) cm^{-1} ; MS (ESI): m/z = 370 [$\text{M} + \text{H}^+$]; HRMS (ESI): m/z for $\text{C}_{24}\text{H}_{36}\text{NO}_2^+$ calc.: 370.2741 [$\text{M} + \text{H}^+$], found: 370.2743; CHN for $\text{C}_{24}\text{H}_{35}\text{NO}_2$: calc. C 78.00 H 9.55 N 3.79, found C 74.14 H 8.91 N 3.40.

8-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)octan-1-amine 13b

Setup: 2-(8-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)octyl)isoindoline-1,3-dione **12b** (500 mg, 900 μmol), $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (4.4 mL, 4.50 g, 90.0 mmol)

Colourless solid (76 %, 291 mg, 684 μmol); $^1\text{H-NMR}$ (500 MHz, DMSO, 373 K): δ = 0.87 (t, J = 7.1 Hz, 3H, CH_3), 1.24–1.48 (m, 20H, $10 \times \text{CH}_2$), 1.72 (tt, J = 6.5 Hz, 6.5 Hz, 4H, $2 \times \text{OCH}_2\text{CH}_2$), 2.55 (t, J = 6.5 Hz, 2H, NCH_2), 4.00 (t, J = 6.5 Hz, 4H, $2 \times \text{biphenyl-OCH}_2$), 6.90–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.49 (m, 4H, 2'-H, 2''-H) ppm; $^{13}\text{C-NMR}$ (126 MHz, DMSO, 373 K) δ = 13.5 (CH_3), 21.8, 25.4, 25.5, 26.3, 28.4, 28.6, 28.7, 28.8, 28.8, 31.1, 33.4 ($12 \times \text{CH}_2$), 41.6 (NCH_2), 68.1 ($2 \times \text{OCH}_2$), 115.2 (C-3', C-3''), 127.2 (C-2', C-2''), 132.7 (C-1', C-1''), 158.1 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3042 (w), 2953 (w), 2920 (m), 2852 (m), 1606 (w), 1569 (w), 1499 (m), 1473 (m), 1393 (w), 1329 (w), 1273 (m), 1248 (s), 1178 (w), 1138 (w), 1063 (w), 1034 (m), 1013 (w), 995 (w), 952 (w), 824 (s), 808 (s), 757 (w), 721 (w), 646 (w), 594 (w), 518 (w), 484 (w), 417 (w) cm^{-1} ; MS (ESI): m/z = 426 [$\text{M} + \text{H}^+$]; HRMS (ESI): m/z for $\text{C}_{28}\text{H}_{44}\text{NO}_2^+$ calc.: 426.3367 [$\text{M} + \text{H}^+$], found: 426.3373; CHN for $\text{C}_{28}\text{H}_{43}\text{NO}_4$: calc. C 79.01 H 10.18 N 3.29, found C 78.60 H 10.00 N 3.23.

10-((4'-(Decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decan-1-amine 13c

Setup: 2-(10-((4'-(Decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decyl)isoindoline-1,3-dione
12c (235 mg, 384 μmol), $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (1.9 mL, 1.92 g, 38.4 mmol)

Colourless solid (94 %, 270 mg, 455 μmol); $^1\text{H-NMR}$ (500 MHz, DMSO, 373 K): δ = 0.86 (t, J = 6.7 Hz, 3H, CH_3), 1.17–1.48 (m, 28H, $14 \times \text{CH}_2$), 1.72 (tt, J = 6.6 Hz, 6.6 Hz, 4H, $2 \times \text{OCH}_2\text{CH}_2$), 2.51–2.55 (m, 2H, NCH_2), 4.00 (t, J = 6.6 Hz, 4H, $2 \times \text{biphenyl-OCH}_2$), 6.89–6.97 (m, 4H, 3'-H, 3''-H), 7.41–7.51 (m, 4H, 2'-H, 2''-H) ppm; $^{13}\text{C-NMR}$ (126 MHz, DMSO, 373 K) δ = 13.6 (CH_3), 21.9, 25.4, 25.4, 28.6, 28.6, 28.8, 28.8, 28.9, 28.9, 31.1, 33.4 ($16 \times \text{CH}_2$), 41.7 (NCH_2), 68.0 ($2 \times \text{OCH}_2$), 115.2 (C-3', C-3''), 127.2 (C-2', C-2''), 132.7 (C-1', C-1''), 158.1 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3042 (w), 2953 (w), 2920 (m), 2852 (m), 1606 (w), 1569 (w), 1499 (m), 1473 (m), 1393 (w), 1329 (w), 1273 (m), 1248 (s), 1178 (w), 1138 (w), 1063 (w), 1034 (m), 1013 (w), 995 (w), 952 (w), 824 (s), 808 (s), 757 (w), 721 (w), 646 (w), 594 (w), 518 (w), 484 (w), 417 (w) cm^{-1} ; MS (ESI): m/z = 481 [$\text{M} + \text{H}^+$]; HRMS (ESI): m/z for $\text{C}_{32}\text{H}_{51}\text{NO}_2^+$ calc.: 481.75, found: 481.75.

C-3''), 127.2 (C-2', C-2''), 132.6 (C-1', C-1''), 158.0 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 2250 (w), 2124 (w), 1619 (w), 1053 (s), 1025 (s), 1006 (s), 820 (m), 757 (m), 622 (w) cm^{-1} ; MS (ESI): m/z = 482 [$\text{M} + \text{H}^+$]; HRMS (ESI): m/z for $\text{C}_{32}\text{H}_{52}\text{NO}_2^+$ calc.: 482.3993 [$\text{M} + \text{H}^+$], found: 482.3995; CHN for $\text{C}_{32}\text{H}_{51}\text{NO}_2$: calc. C 79.78 H 10.67 N 2.91, found C 78.67 H 10.56 N 2.75.

12-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecan-1-amine 13d

Setup: 2-(12-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecyl)isoindoline-1,3-dione **12d** (500 mg, 749 μmol), $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (3.6 mL, 3.75 g, 74.9 mmol)

Colourless solid (95 %, 384 mg, 714 μmol); ^1H -NMR (500 MHz, DMSO, 373 K): δ = 0.86 (t, J = 6.8 Hz, 3H, CH_3), 1.23–1.48 (m, 36H, $18 \times \text{CH}_2$), 1.72 (tt, J = 6.5 Hz, 6.5 Hz, 4H, $2 \times \text{OCH}_2\text{CH}_2$), 2.54 (t, J = 6.6 Hz, 2H, NCH_2), 4.00 (t, J = 6.5 Hz, 4H, $2 \times \text{biphenyl-OCH}_2$), 6.91–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.50 (m, 4H, 2'-H, 2''-H) ppm; ^{13}C -NMR (126 MHz, DMSO, 373 K) δ = 13.5 (CH_3), 21.8, 25.4, 26.4, 28.5, 28.6, 28.8, 28.8, 28.9, 28.9, 31.1, 33.5 ($20 \times \text{CH}_2$), 41.7 (NCH_2), 68.1 ($2 \times \text{OCH}_2$), 115.2 (C-3', C-3''), 127.2 (C-2', C-2''), 132.7 (C-1', C-1''), 158.1 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3043 (w), 2954 (w), 2916 (s), 2872 (w), 2848 (s), 1607 (w), 1570 (w), 1500 (m), 1473 (m), 1464 (m), 1393 (w), 1329 (w), 1274 (s), 1248 (s), 1178 (w), 1138 (w), 1063 (w), 1036 (m), 1005 (w), 997 (w), 953 (w), 825 (s), 808 (s), 765 (w), 729 (w), 719 (w), 646 (w), 594 (w), 519 (w), 466 (w), 417 (w) cm^{-1} ; MS (ESI): m/z = 538 [$\text{M} + \text{H}^+$]; HRMS (ESI): m/z for $\text{C}_{36}\text{H}_{60}\text{NO}_2^+$ calc.: 538.4619 [$\text{M} + \text{H}^+$], found: 538.4622; CHN for $\text{C}_{36}\text{H}_{59}\text{NO}_2$: calc. C 80.39 H 11.06 N 2.60, found C 80.03 H 10.84 N 2.42.

14-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecan-1-amine 13e

Setup: 6-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-amine **12e**

(56.0 mg, 116 μ mol), $N_2H_4 \cdot H_2O$ (2.4 mL, 2.42 g, 48.3 mmol)

Colourless solid (94 %, 132 mg, 274 μ mol); ^1H -NMR (500 MHz, DMSO, 373 K): δ = 0.85 (t, J = 7.0 Hz, 3H, CH_3), 1.17–1.47 (m, 44H, $22 \times CH_2$), 1.72 (tt, J = 6.5 Hz, 6.5 Hz, 4H, $2 \times OCH_2CH_2$), 2.53 (t, J = 6.7 Hz, 2H, NCH_2), 3.99 (t, J = 6.5 Hz, 4H, $2 \times$ biphenyl- OCH_2), 6.92–6.97 (m, 4H, 3'-H, 3''-H), 7.44–7.49 (m, 4H, 2'-H, 2''-H) ppm. Due to the poor solubility, a high temperature ^{13}C NMR could not be obtained in adequate quality; FT-IR: $\tilde{\nu}$ = 3447 (w), 2250 (w), 2125 (w), 1659 (w), 1053 (s), 1024 (s), 1005 (s), 821 (m), 758 (m), 623 (w), 514 (w) cm^{-1} ; MS (ESI): m/z = 595 [$M + H^+$]; HRMS (ESI): m/z for $C_{40}H_{68}NO_2^+$ calc.: 594.5245 [$M + H^+$], found: 594.5248; CHN for $C_{40}H_{67}NO_2$: calc. C 80.88 H 11.37 N 2.36, found C 80.39 H 11.26 N 2.33.

14-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecan-1-amine 13f

Setup: 2-(14-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecyl)isoindoline-1,3-

dione **12f** (250 mg, 409 μ mol), $N_2H_4 \cdot H_2O$ (2.0 mL, 2.05 g, 40.9 mmol)

Colourless solid (67 %, 132 mg, 274 μ mol); ^1H -NMR (500 MHz, DMSO, 373 K): δ = 0.88 (t, J = 7.1 Hz, 3H, CH_3), 1.23–1.48 (m, 28H, $14 \times CH_2$), 1.72 (tt, J = 6.4 Hz, 6.4 Hz, 4H, $2 \times OCH_2CH_2$), 2.53 (t, J = 6.7 Hz, 2H, NCH_2), 4.00 (t, J = 6.4 Hz, 4H, $2 \times$ biphenyl-

OCH₂), 6.74–7.06 (m, 4H, 3'-H, 3''-H), 7.44–7.50 (m, 4H, 2'-H, 2''-H) ppm. Due to the poor solubility, a high temperature ¹³C NMR could not be obtained in adequate quality; FT-IR: $\tilde{\nu}$ = 3406 (w), 2254 (w), 2127 (w), 1657 (w), 1049 (m), 1023 (s), 999 (s), 823 (m), 761 (m), 524 (m) cm⁻¹; MS (ESI): m/z = 482 [M + H⁺]; HRMS (ESI): m/z for C₃₂H₅₂NO₂⁺ calc.: 482.3993 [M + H⁺], found: 482.3991; CHN for C₃₂H₅₁NO₂: calc. C 79.78 H 10.67 N 2.91, found C 78.88 H 10.63 N 2.80.

12-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecan-1-amine 13g

Setup: 2-(12-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecyl)isoindoline-1,3-dione **12g** (500 mg, 817 μ mol), N₂H₄·H₂O (4.0 mL, 4.09 g, 81.7 mmol)

Colourless solid (88 %, 345 mg, 716 μ mol); ¹H-NMR (500 MHz, DMSO, 373 K): δ = 0.87 (t, J = 6.5 Hz, 3H), 1.21–1.47 (m, 28H, 14 $\times$ CH₂), 1.72 (tt, J = 6.5 Hz, 6.5 Hz, 4H, 2 $\times$ OCH₂CH₂), 2.54 (t, J = 6.7 Hz, 2H, NCH₂), 4.00 (t, J = 6.5 Hz, 4H, 2 $\times$ biphenyl-OCH₂), 6.91–6.97 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H) ppm; ¹³C-NMR (126 MHz, DMSO, 373 K) δ = 13.5 (CH₃), 21.8, 25.4, 25.5, 26.3, 28.4, 28.6, 28.6, 28.8, 28.8, 28.8, 28.9, 31.1, 33.4 (16 $\times$ CH₂), 41.6 (NCH₂), 68.1 (2 $\times$ OCH₂), 115.2 (C-3', C-3''), 127.2 (C-2', C-2''), 132.7 (C-1', C-1''), 158.1 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3278 (w), 3042 (w), 2954 (w), 2917 (m), 2849 (m), 1606 (w), 1568 (w), 1499 (s), 1473 (m), 1393 (w), 1330 (w), 1274 (s), 1248 (s), 1178 (w), 1159 (w), 1138 (w), 1034 (w), 1011 (w), 996 (w), 952 (w), 886 (w), 824 (s), 809 (m), 767 (w), 720 (w), 690 (w), 646 (w), 631 (w), 594 (w), 518 (w), 466 (w), 412 (w) cm⁻¹; MS (ESI): m/z = 482 [M + H⁺]; HRMS (ESI): m/z for C₃₂H₅₂NO₂⁺ calc.: 482.3993 [M + H⁺], found:

482.3990; CHN for C₃₂H₅₁NO₂: calc. C 79.78 H 10.67 N 2.91, found C 77.57 H 10.37 N 3.03.

8-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)octan-1-amine **13h**

Setup: 2-(8-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)octyl)isoindoline-1,3-dione **12h** (500 mg, 817 μ mol), N₂H₄·H₂O (4.0 mL, 4.09 g, 81.7 mmol)

Colourless solid (94 %, 369 mg, 766 μ mol); ¹H-NMR (500 MHz, DMSO, 373 K): δ = 0.81–1.00 (m, 3H, CH₃), 1.22–1.63 (m, 28H, 14 $\times$ CH₂), 1.69–1.85 (m, 4H, 2 $\times$ OCH₂CH₂), 2.54–2.62 (m, 2H, NCH₂), 3.96–4.18 (m, 4H, 2 $\times$ biphenyl-OCH₂), 6.91–7.10 (m, 4H, 3'-H, 3''-H), 7.43–7.63 (m, 4H, 2'-H, 2''-H) ppm; ¹³C-NMR (126 MHz, DMSO, 373 K) δ = 13.5 (CH₃), 21.8, 25.4, 26.3, 28.5, 28.6, 28.7, 28.8, 28.8, 28.8, 28.9, 31.1 (16 $\times$ CH₂), 41.6 (NCH₂), 68.1 (2 $\times$ OCH₂), 115.2 (C-3', C-3''), 127.2 (C-2', C-2''), 132.7 (C-1', C-1''), 158.1 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3042 (w), 2955 (w), 2917 (m), 2873 (w), 2849 (w), 1606 (w), 1569 (w), 1499 (m), 1473 (w), 1464 (w), 1393 (w), 1329 (w), 1273 (m), 1248 (s), 1178 (w), 1138 (w), 1095 (w), 1035 (w), 1005 (w), 996 (w), 952 (w), 824 (s), 808 (s), 765 (w), 720 (w), 646 (w), 594 (w), 518 (w) cm⁻¹; MS (ESI): m/z = 482 [M + H⁺]; HRMS (ESI): m/z for C₃₂H₅₂NO₂⁺ calc.: 482.3993 [M + H⁺], found: 482.4007; CHN for C₃₂H₅₁NO₂: calc. C 79.78 H 10.67 N 2.91, found C 78.92 H 10.49 N 2.52.

6-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-amine 13i

Setup: 2-(6-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexyl)isoindoline-1,3-dione **12i** (577 mg, 943 μ mol), $N_2H_4 \cdot H_2O$ (4.6 mL, 4.72 g, 94.3 mmol)

Recrystallisation with DMSO (15 mL) followed. The precipitate was filtered and the solvent was removed under reduced pressure.

Colourless solid (89 %, 402 mg, 834 μ mol); ^1H -NMR (500 MHz, DMSO, 373 K): δ = 0.85 (t, J = 6.5 Hz, 3H, CH_3), 1.18–1.55 (m, 28H, $14 \times CH_2$), 1.72 (tt, J = 6.5 Hz, 6.5 Hz, 4H, $2 \times OCH_2CH_2$), 2.54–2.58 (m, 2H, NCH_2), 3.99 (t, J = 6.5 Hz, 4H, $2 \times$ biphenyl- OCH_2), 6.91–6.99 (m, 4H, 3'-H, 3''-H), 7.44–7.54 (m, 4H, 2'-H, 2''-H) ppm; ^{13}C -NMR (126 MHz, DMSO, 373 K) δ = 13.6 (CH_3), 21.9, 25.4, 26.1, 28.5, 28.6, 28.7, 28.8, 28.9, 28.9, 31.2, 33.3 ($16 \times CH_2$), 41.6 (NCH_2), 67.9 ($2 \times OCH_2$), 115.1 (C-3', C-3''), 127.1 (C-2', C-2''), 132.6 (C-1', C-1''), 158.0 (C-4', C-4'') ppm; FT-IR: $\tilde{\nu}$ = 3438 (w), 2251 (w), 2125 (w), 1660 (w), 1052 (s), 1024 (s), 1004 (s), 821 (m), 758 (m), 623 (w), 515 (w) cm^{-1} ; MS (ESI): m/z = 482 [$M + H^+$]; HRMS (ESI): m/z for $C_{32}H_{52}NO_2^+$ calc.: 482.3993 [$M + H^+$], found: 482.3956; CHN for $C_{32}H_{51}NO_2$: calc. C 79.78 H 10.67 N 2.91, found C 78.73 H 10.51 N 2.79.

Cchloroformamidinium chloride 16

According to a procedure by Butschies,[15] 1,1,3,3-tetramethylurea (8.2 mL, 7.95 g, 68.5 mmol) was dissolved in dry THF (70 mL) and cooled to 0 °C. Oxalyl chloride

(18.8 g, 144 mmol) was added dropwise and the reaction mixture was left to stir for 5 d. The solid was filtered under nitrogen atmosphere and washed with cold, dry THF and dried under vacuum, yielding **16** as a colourless solid (931 mg, 58.0 mmol, 85 %). The product was dissolved in dry CH₂Cl₂ (450 mL, 129 mM) and stored at 4 °C.

General Procedure for the Formation of the Guanidinium Chlorides Gua(n,m)Cl

According to a procedure by Butschies,[15] the corresponding amine **13** (126 µmol), chlorformamidinium chloride **16** (3.0 mL, 391 µmol) and dry NEt₃ (504 µmol) were heated under reflux with dry MeCN (6 mL) for 6 h. The reaction mixture was cooled to room temperature, deionized water (50 µL) was added and the reaction mixture was stirred intensely for 20 min. Purification via column chromatography followed, using silica gel treated with hydrochloric acid.² The column was flushed with pure EtOAc, then CH₂Cl₂, followed by a CH₂Cl₂ / MeOH = (20 : 1) → (5 : 1) mixture. The clean product was dissolved in MeOH, stirred with a solution of NaOH in MeOH for 20 min. The solvent was removed under reduced pressure. After 2 d under vacuum, the solid was suspended in dry CH₂Cl₂ (10 mL) and filtered. HCl in diethylether was added to the organic phase to achieve pH = 1, and the suspension was stirred for 10 min. The solvent was removed under reduced pressure and the solid was dried under vacuum to yield **Gua(n,m)Cl**.

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² The silica gel was stirred in 6 M HCl for 30 min, then filtered and washed with water and acetone and, finally, dried under vacuum.

N-(Bis(dimethylamino)methylene)-6-((4'-(hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(6,6)Cl**

Setup: 6-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-amine **13a** (63.0 mg, 126 μmol), chloroformamidinium chloride **16** (3.0 mL, 391 μmol), dry NEt_3 (70 μL , 51.0 mg, 504 μmol)

Colourless solid (85 %, 54.0 mg, 107 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.89–0.95 (m, 3H, CH_3), 1.23–1.56 (m, 10H, $5 \times \text{CH}_2$), 1.75–1.88 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.87–3.26 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.94–4.03 (m, 4H, $2 \times \text{OCH}_2$), 6.90–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.49 (m, 4H, 2'-H, 2''-H), 9.90 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.6, 25.6, 25.7, 26.6, 29.1, 29.3, 29.7, 31.6 ($8 \times \text{CH}_2$), 39.9, 40.6 ($2 \times \text{N}(\text{CH}_3)_2$), 45.3 (NCH_2), 67.7, 68.1 (OCH_2), 114.7, 114.8 (C 3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3036 (w), 2930 (w), 2859 (w), 2189 (w), 1606 (m), 1498 (s), 1469 (w), 1402 (w), 1288 (w), 1271 (w), 1240 (s), 1174 (w), 1114 (w), 1066 (w), 1037 (w), 1014 (w), 997 (w), 925 (m), 909 (w), 822 (m), 727 (s), 640 (w), 599 (w), 520 (w) cm^{-1} ; MS (ESI): m/z = 468 [M^+]; HRMS (ESI): m/z for $\text{C}_{29}\text{H}_{46}\text{N}_3\text{O}_2^+$ calc.: 468.3585 [M^+]; found: 468.3597; CHN for $\text{C}_{29}\text{H}_{46}\text{ClN}_3\text{O}_2$: calc. C 69.09 H 9.20 N 8.33, found C 68.91 H 9.06 N 8.21; DSC: Cr₁ 71 °C [2.5 kJ mol⁻¹] Cr₂ 75 °C [1.9 kJ mol⁻¹] SmA 133 °C [3.0 kJ mol⁻¹] I.

N-(Bis(dimethylamino)methylene)-8-((4'-(octyloxy)-[1,1'-biphenyl]-4-yl)oxy)octan-1-aminium chloride **Gua(8,8)Cl**

Setup: 8-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)octan-1-amine **13b** (250 mg, 587 μmol), chloroformamidinium chloride **16** (14.1 mL, 1.82 mmol), dry NEt_3 (330 μL , 238 mg, 2.35 mmol)

Colourless solid (77 %, 252 mg, 500 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.89 (t, J = 7.4 Hz, 3H, CH_3), 1.23–1.50 (m, 18H, $9 \times \text{CH}_2$), 1.74–1.84 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.89–3.23 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.5 Hz, 4H, $2 \times \text{OCH}_2$), 6.90–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.49 (m, 4H, 2'-H, 2''-H), 9.82 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.0, 26.1, 26.9, 29.1, 29.2, 29.3, 29.3, 29.4, 29.8, 31.8 ($12 \times \text{CH}_2$), 39.9, 40.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.5, 45.8 (NCH_2), 68.0, 68.1 (OCH_2), 114.7, 114.7 (C-3', C-3''), 127.6, 127.6 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.2, 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3401 (w), 2931 (m), 2854 (w), 1607 (s), 1501 (s), 1474 (w), 1403 (w), 1274 (m), 1248 (s), 1178 (w), 1138 (w), 1036 (w), 1016 (w), 996 (w), 899 (w), 825 (m), 808 (w), 727 (w), 595 (w), 517 (w) cm^{-1} ; MS (ESI): m/z = 524 [M^+]; HRMS (ESI): m/z for $\text{C}_{33}\text{H}_{54}\text{N}_3\text{O}_2^+$ calc.: 524.4211 [M^+], found: 524.4221; CHN for $\text{C}_{33}\text{H}_{54}\text{ClN}_3\text{O}_2$: calc. C 70.75 H 9.72 N 7.50, found C 70.39 H 9.40 N 7.27; DSC: Cr_1 56 $^\circ\text{C}$ [6.4 kJ mol^{-1}] Cr_2 67 $^\circ\text{C}$ [5.1 kJ mol^{-1}] Cr_3 101 $^\circ\text{C}$ [6.4 kJ mol^{-1}] SmA 129 $^\circ\text{C}$ [3.5 kJ mol^{-1}] I.

N-(Bis(dimethylamino)methylene)-10-((4'-(decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decan-1-aminium chloride **Gua(10,10)Cl**

Setup: 10-((4'-(Decyloxy)-[1,1'-biphenyl]-4-yl)oxy)decan-1-amine **13c** (140 mg, 291 μ mol), chloroformamidinium chloride **16** (7.0 mL, 901 μ mol), dry NEt_3 (162 μ L, 118 mg, 1.16 mmol)

Colourless solid (74 %, 133 mg, 216 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.19–1.52 (m, 26H, $13 \times \text{CH}_2$), 1.70–1.87 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.84–3.25 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.6 Hz, 4H, $2 \times \text{OCH}_2$), 6.88–7.00 (m, 4H, 3'-H, 3''-H), 7.40–7.52 (m, 4H, 2'-H, 2''-H), 9.87 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.0, 26.1, 26.9, 29.1, 29.3, 29.3, 29.4, 29.5, 29.6, 29.6, 29.7, 29.9, 31.9 ($16 \times \text{CH}_2$), 39.9, 40.6 ($2 \times \text{N}(\text{CH}_3)_2$), 45.4 (NCH_2), 68.1, 68.1 ($2 \times \text{OCH}_2$), 114.7 (C-3', C-3''), 127.6 (C-2', C-2''), 133.3, 133.3 (C-1', C-1''), 158.2, 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3387 (w), 3042 (w), 2918 (m), 2850 (w), 2186 (w), 1607 (m), 1500 (s), 1474 (w), 1403 (w), 1376 (w), 1327 (w), 1273 (m), 1247 (s), 1177 (w), 1139 (w), 1035 (w), 1012 (w), 996 (w), 927 (w), 898 (w), 826 (m), 808 (w), 726 (w), 641 (w), 595 (w), 516 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4825; CHN for $\text{C}_{37}\text{H}_{62}\text{ClN}_3\text{O}_2$: calc. C 72.10 H 10.14 N 6.82, found C 72.03 H 10.09 N 6.69; DSC: Cr₁ 77 °C [25.4 kJ mol⁻¹] Cr₂ 117 °C [4.9 kJ mol⁻¹] SmA 143 °C [5.7 kJ mol⁻¹] I.

N-(Bis(dimethylamino)methylene)-12-((4'-(dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecan-1-aminium chloride **Gua(12,12)Cl**

Setup: 12-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecan-1-amine **13d**
(340 mg, 632 μmol), chloroformamidinium chloride **16** (15.2 mL,
1.95 mmol), dry NEt_3 (350 μL , 256 mg, 2.53 mmol)

Colourless solid (71 %, 301 mg, 448 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.21–1.51 (m, 34H, $17 \times \text{CH}_2$), 1.73–1.83 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.87–3.23 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.5 Hz, 4H, $2 \times \text{OCH}_2$), 6.91–6.96 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H), 9.91 (s, 1H, NH) ppm;
 ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.1, 26.9, 29.1, 29.3, 29.3, 29.4, 29.4, 29.5, 29.5, 29.6, 29.6, 29.6, 29.6, 29.7, 29.9, 31.9 ($20 \times \text{CH}_2$), 39.9, 40.6 ($2 \times \text{N}(\text{CH}_3)_2$), 45.4, 45.8 (NCH_2), 68.1, 68.1 (OCH_2), 114.7 (C-3', C-3''), 127.6 (C-2', C-2''), 133.3, 133.3 (C-1', C-1''), 158.2, 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm;
FT-IR: $\tilde{\nu}$ = 3396 (w), 2917 (s), 2849 (m), 2176 (w), 1607 (m), 1500 (m), 1474 (w), 1402 (w), 1328 (w), 1274 (m), 1247 (s), 1177 (w), 1139 (w), 1066 (w), 1036 (w), 1014 (w), 996 (w), 927 (w), 899 (w), 826 (m), 808 (w), 725 (w), 640 (w), 595 (w), 516 (w) cm^{-1} ;
MS (ESI): m/z = 637 [M^+]; HRMS (ESI): m/z for $\text{C}_{41}\text{H}_{70}\text{N}_3\text{O}_2^+$ calc.: 636.5463 [M^+], found: 636.5480; CHN for $\text{C}_{41}\text{H}_{70}\text{ClN}_3\text{O}_2$: calc. C 73.23 H 10.49 N 6.25, found C 72.98 H 10.37 N 6.10; DSC:Cr 90 $^\circ\text{C}$ [20.1 kJ mol^{-1}] SmC 125 $^\circ\text{C}$ [12.1 kJ mol^{-1}] SmA 134 $^\circ\text{C}$ [9.4 kJ mol^{-1}] I.

N-(Bis(dimethylamino)methylene)-14-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecan-1-aminium chloride **Gua(14,14)Cl**

Setup: 14-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecan-1-amine **13e**
 (230 mg, 387 μmol), chloroformamidinium chloride **16** (9.3 mL,
 1.20 mmol), dry NEt_3 (216 μL , 157 mg, 1.55 mmol)

Colourless solid (49 %, 98.0 mg, 135 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.9 Hz, 3H, CH_3), 1.21–1.51 (m, 42H, $21 \times \text{CH}_2$), 1.71–1.84 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.88–3.23 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.6 Hz, 4H, $2 \times \text{OCH}_2$), 6.91–6.97 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H), 9.90 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.1, 27.0, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.6, 29.6, 29.7, 29.7, 29.9, 31.9 ($24 \times \text{CH}_2$), 39.8, 40.6 ($2 \times \text{N}(\text{CH}_3)_2$), 45.4 (NCH_2), 68.1 ($2 \times \text{OCH}_2$), 114.7 (C-3', C-3''), 127.6 (C-2', C-2''), 133.3, 133.3 (C-1', C-1''), 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3389 (w), 2917 (s), 2849 (s), 2186 (w), 1728 (w), 1607 (m), 1501 (m), 1473 (w), 1465 (w), 1402 (w), 1377 (w), 1274 (m), 1248 (s), 1178 (w), 1139 (w), 1067 (w), 1039 (w), 1009 (w), 997 (w), 926 (w), 905 (w), 824 (m), 808 (w), 727 (w), 640 (w), 595 (w), 518 (w) cm^{-1} ; MS (ESI): m/z = 693 [M^+]; HRMS (ESI): m/z for $\text{C}_{45}\text{H}_{78}\text{N}_3\text{O}_2^+$ calc.: 692.6089 [M^+], found: 692.6100; CHN for $\text{C}_{45}\text{H}_{78}\text{ClN}_3\text{O}_2$: calc. C 74.18 H 10.79 N 5.77, found C 74.96 H 10.63 N 5.60; DSC: Cr₁ 97 °C [23.3 kJ mol⁻¹] Cr₂ 128 °C [15.1 kJ mol⁻¹] SmA 145 °C [15.1 kJ mol⁻¹]
 I.

N-(Bis(dimethylamino)methylene)-14-((4'-(hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecan-1-aminium chloride **Gua(6,14)Cl**

Setup: 14-((4'-(Hexyloxy)-[1,1'-biphenyl]-4-yl)oxy)tetradecan-1-amine **13f**
(100 mg, 208 μmol), chloroformamidinium chloride **16** (5.0 mL,
643 μmol), dry NEt_3 (116 μL , 84.0 mg, 830 μmol)

Colourless solid (54 %, 69.0 mg, 112 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.91 (t, J = 7.0 Hz, 3H, CH_3), 1.20–1.52 (m, 26H, $13 \times \text{CH}_2$), 1.71–1.84 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.85–3.27 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.3 Hz, 4H, $2 \times \text{OCH}_2$), 6.91–6.96 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 4H, 2'-H, 2''-H), 9.87 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.0 (CH_3), 22.6, 25.7, 26.1, 27.0, 29.2, 29.3, 29.3, 29.4, 29.6, 29.6, 29.9, 31.6 ($16 \times \text{CH}_2$), 39.9, 40.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.4 (NCH_2), 68.1 ($2 \times \text{OCH}_2$), 114.7 (C-3', C-3''), 127.6 (C-2', C-2''), 133.3 (C-1', C-1''), 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3395 (w), 3041 (w), 2921 (s), 2852 (m), 2179 (w), 1726 (w), 1607 (s), 1501 (m), 1475 (w), 1403 (w), 1378 (w), 1274 (m), 1250 (s), 1179 (w), 1139 (w), 1067 (w), 1036 (w), 1013 (w), 994 (w), 927 (w), 899 (w), 825 (m), 809 (w), 729 (w), 641 (w), 594 (w), 518 (w), 450 (w), 427 (w), 408 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4831; CHN for $\text{C}_{37}\text{H}_{62}\text{ClN}_3\text{O}_2$: calc. C 72.10 H 10.14 N 6.82, found C 71.79 H 9.87 N 6.53; DSC: Cr₁ 53 °C [1.0 kJ mol⁻¹] Cr₂ 78 °C [3.5 kJ mol⁻¹] Cr₃ 131 °C [7.0 kJ mol⁻¹] SmA 151 °C [6.5 kJ mol⁻¹] I.

N-(Bis(dimethylamino)methylene)-12-((4'-(octyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecan-1-aminium chloride **Gua(8,12)Cl**

Setup: 12-((4'-(Octyloxy)-[1,1'-biphenyl]-4-yl)oxy)dodecan-1-amine **13g** (300 mg, 623 μmol), chloroformamidinium chloride **16** (15.0 mL, 1.93 mmol), dry NEt_3 (350 μL , 252 mg, 2.49 mmol)

Colourless solid (32 %, 122 mg, 198 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.89 (t, J = 7.2 Hz, 3H, CH_3), 1.22–1.51 (m, 26H, $13 \times \text{CH}_2$), 1.72–1.84 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.86–3.26 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.6 Hz, 4H, $2 \times \text{OCH}_2$), 6.91–6.96 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H), 9.75 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.1, 26.1, 27.0, 29.2, 29.3, 29.3, 29.4, 29.5, 29.5, 29.9, 31.8 ($16 \times \text{CH}_2$), 40.0, 40.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.5 (NCH_2), 68.1, 68.1 (OCH_2), 114.7 (C-3', C-3''), 127.6 (C-2', C-2''), 133.3, 133.3 (C-1', C-1''), 158.2, 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3387 (w), 2919 (s), 2851 (m), 1607 (m), 1501 (m), 1474 (w), 1403 (w), 1377 (w), 1328 (w), 1274 (s), 1250 (s), 1178 (w), 1139 (w), 1066 (w), 1035 (w), 996 (w), 900 (w), 825 (m), 809 (w), 722 (w), 595 (w), 519 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4847; CHN for $\text{C}_{37}\text{H}_{62}\text{ClN}_3\text{O}_2$: calc. C 72.10 H 10.14 N 6.82, found C 71.92 H 10.03 N 6.61; DSC: Cr₁ 95 °C [8.8 kJ mol⁻¹] Cr₂ 143 °C [24.3 kJ mol⁻¹] I.

N-(Bis(dimethylamino)methylene)-8-((4'-(dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)octan-1-aminium chloride **Gua(12,8)Cl**

Setup: 8-((4'-(Dodecyloxy)-[1,1'-biphenyl]-4-yl)oxy)octan-1-amine **13h** (300 mg, 623 μmol), chloroformamidinium chloride **16** (15.0 mL, 1.93 mmol), dry NEt_3 (350 μL , 252 mg, 2.49 mmol)

Colourless solid (74 %, 285 mg, 462 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.9 Hz, 3H, CH_3), 1.22–1.50 (m, 26H, $13 \times \text{CH}_2$), 1.74–1.83 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.88–3.24 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.7 Hz, 4H, $2 \times \text{OCH}_2$), 6.91–6.97 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H), 9.90 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 26.0, 26.1, 26.9, 29.1, 29.2, 29.3, 29.4, 29.4, 29.6, 29.6, 29.6, 29.7, 29.8, 31.9 ($16 \times \text{CH}_2$), 39.9, 40.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.5, 45.8 (NCH_2), 68.0, 68.1 (OCH_2), 114.7, 114.7 (C-3', C-3''), 127.6, 127.6 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.2, 158.2 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3401 (w), 2917 (s), 2849 (w), 2178 (w), 1607 (m), 1499 (s), 1472 (w), 1403 (w), 1273 (m), 1246 (s), 1177 (w), 1066 (w), 1036 (w), 1016 (w), 926 (w), 906 (w), 825 (m), 808 (w), 727 (w), 640 (w), 595 (w), 515 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4847; CHN for $\text{C}_{37}\text{H}_{62}\text{ClN}_3\text{O}_2$: calc. C 72.10 H 10.14 N 6.82, found C 72.01 H 10.08 N 6.68; DSC: Cr 77 $^\circ\text{C}$ [22.0 kJ mol^{-1}] SmC 101 $^\circ\text{C}$ [2.6 kJ mol^{-1}] SmA 156 $^\circ\text{C}$ [1.6 kJ mol^{-1}] I.

N-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl**

Setup: 6-((4'-(Tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-amine **13i**
(200 mg, 415 μmol), chloroformamidinium chloride **16** (10.0 mL,
1.28 mmol), dry NEt_3 (232 μL , 168 mg, 1.66 mmol)

Colourless solid (57 %, 147 mg, 238 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.9 Hz, 3H, CH_3), 1.21–1.56 (m, 26H, $13 \times \text{CH}_2$), 1.75–1.88 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.87–3.27 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.5 Hz, 4H, $2 \times \text{OCH}_2$), 6.89–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.50 (m, 4H, 2'-H, 2''-H), 9.93 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 25.6, 26.1, 26.6, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 31.9 ($16 \times \text{CH}_2$), 39.9, 40.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.3 (NCH_2), 67.7, 68.1 ($2 \times \text{OCH}_2$), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3394 (w), 3041 (w), 2918 (s), 2849 (m), 2182 (w), 1607 (m), 1500 (s), 1472 (w), 1403 (w), 1273 (m), 1247 (s), 1177 (w), 1136 (w), 1069 (w), 1039 (w), 1010 (w), 997 (w), 927 (w), 901 (w), 825 (w), 809 (w), 729 (w), 642 (w), 595 (w), 515 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4859; CHN for $\text{C}_{37}\text{H}_{62}\text{ClN}_3\text{O}_2$: calc. C 72.10 H 10.14 N 6.82, found C 71.92 H 10.04 N 6.74; DSC: Cr 66 $^\circ\text{C}$ [8.0 kJ mol^{-1}] SmC 93 $^\circ\text{C}$ [3.5 kJ mol^{-1}] SmA 194 $^\circ\text{C}$ [0.9 kJ mol^{-1}] I.

General Procedure for the Anion Exchange of Guanidinium Chloride Gua(14,6)Cl

According to a procedure by Butschies,[15] *N*-(bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (81.1 μmol) was suspended in dry MeCN (15 mL) and heated with the respective salt under reflux for 10 min. The solvent was removed under reduced pressure, suspended in dry CH₂Cl₂ (5 mL) and filtered. The solvent was removed under reduced pressure and the product **Gua(14,6)X** was dried under vacuum.

N-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium bromide **Gua(14,6)Br**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl] 4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (50.0 mg, 81.1 μmol), potassium bromide (42.0 mg, 353 μmol)

Colourless solid (82 %, 44.0 mg, 66.6 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.20–1.56 (m, 26H, $13 \times \text{CH}_2$), 1.75–1.87 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2\text{NCH}_2\text{CH}_2$), 2.88–3.25 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.4 Hz, 4H, $2 \times \text{OCH}_2$), 6.90–6.96 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H), 9.87 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 25.6, 26.1, 26.6, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 31.9 ($16 \times \text{CH}_2$), 39.9, 40.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.3, 45.8 (NCH_2), 67.7, 68.1 ($2 \times \text{OCH}_2$), 114.7, 114.8 c, 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.4 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3395 (w), 2917 (s), 2848 (m), 2183 (w), 1607 (m), 1499 (s), 1472 (w), 1403 (w), 1273 (m), 1246 (s), 1177 (w), 1136 (w), 1039 (w), 1010 (w), 997 (w), 927 (w), 903 (w), 825 (w), 809 (w),

728 (w), 641 (w), 596 (w), 515 (w) cm^{-1} ; MS (ESI): $m/z = 580 [\text{M}^+]$; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{65}\text{N}_3\text{O}_2^+$ calc.: 580.4837 $[\text{M}^+]$, found: 580.4853; CHN for $\text{C}_{37}\text{H}_{62}\text{BrN}_3\text{O}_2$: calc. C 67.25 H 9.46 N 6.36, found C 67.04 H 9.31 N 6.22; DSC: Cr₁ 72 °C [18.4 kJ mol⁻¹] Cr₂ 80 °C [3.0 kJ mol⁻¹] SmA 187 °C [1.2 kJ mol⁻¹] I.

N-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium iodide **Gua(14,6)I**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (50.0 mg, 81.1 μmol), potassium iodide (23.0 mg, 139 μmol)

Colourless solid (94 %, 54.0 mg, 76.3 μmol); ^1H -NMR (500 MHz, CDCl_3): $\delta = 0.88$ (t, $J = 6.8$ Hz, 3H, CH_3), 1.20–1.60 (m, 26H, $13 \times \text{CH}_2$), 1.75–1.87 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.91–3.29 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.95–4.02 (m, 4H, $2 \times \text{OCH}_2$), 6.90–6.96 (m, 4H, 3'-H, 3''-H), 7.42–7.49 (m, 4H, 2'-H, 2''-H), 8.06 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) $\delta = 14.1$ (CH_3), 22.7, 25.6, 26.1, 26.5, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 29.9, 31.9 ($16 \times \text{CH}_2$), 40.1 ($2 \times \text{N}(\text{CH}_3)_2$), 45.0 (NCH_2), 67.7, 68.1 ($2 \times \text{OCH}_2$), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.5 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.2 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu} = 3208$ (w), 2918 (m), 2849 (w), 2198 (w), 1607 (m), 1585 (m), 1499 (s), 1472 (w), 1401 (w), 1272 (w), 1245 (s), 1177 (w), 1136 (w), 1039 (w), 1011 (w), 996 (w), 908 (w), 825 (w), 809 (w), 727 (s), 642 (w), 598 (w), 515 (w) cm^{-1} ; MS (ESI): $m/z = 580 [\text{M}^+]$; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 $[\text{M}^+]$, found: 580.4838; CHN for $\text{C}_{37}\text{H}_{62}\text{IN}_3\text{O}_2$: calc.

C 62.78 H 8.83 N 5.94, found C 62.67 H 8.60 N 5.77; DSC: Cr 80 °C [30.6 kJ mol⁻¹]
SmA 193 °C [0.9 kJ mol⁻¹] I.

N-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium trifluoromethanesulfonate **Gua(14,6)OTf**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (50.0 mg, 81.1 μmol),
sodium trifluoromethylsulfonate (28.0 mg, 163 μmol)

Colourless solid (90 %, 53.0 mg, 72.6 μmol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.88 (t, *J* = 6.8 Hz, 3H, CH₃), 1.20–1.56 (m, 26H, 13 × CH₂), 1.66–1.84 (m, 6H, 2 × OCH₂CH₂, NCH₂CH₂), 2.91–3.23 (m, 14H, 2 × N(CH₃)₂, NCH₂), 3.94–4.03 (m, 4H, 2 × OCH₂), 6.88–6.97 (m, 4H, 3'-H, 3''-H), 7.42–7.49 (m, 4H, 2'-H, 2''-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 25.6, 26.1, 26.4, 29.0, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 31.9 (16 × CH₂), 40.0 (2 × N(CH₃)₂), 45.4 (NCH₂), 67.6, 68.1 (2 × OCH₂), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.7 (CN(CH₃)₂) ppm; FT-IR: $\tilde{\nu}$ = 3290 (w), 2920 (w), 2851 (w), 2252 (w), 1608 (w), 1499 (w), 1499 (w), 1470 (w), 1404 (w), 1272 (w), 1242 (m), 1161 (w), 1030 (m), 906 (m), 824 (w), 727 (s), 636 (m), 599 (w), 573 (w), 517 (w) cm⁻¹; MS (ESI): *m/z* for C₃₇H₆₂N₃O₂⁺ = 580 [M⁺]; HRMS (ESI): *m/z* for C₃₇H₆₂N₃O₂⁺ calc.: 580.4837 [M⁺], found: 580.4852; MS (ESI): *m/z* for CF₃O₃S⁻ = 149 [M⁺]; HRMS (ESI): *m/z* for CF₃O₃S⁻ calc.: 148.9526 [M⁻], found: 148.9534; CHN for C₃₈H₆₂F₃N₃O₅S:

calc. C 62.52 H 8.56 N 5.76, found C 62.49 H 8.41 N 5.66; DSC: Cr 60 °C [40.1 kJ mol⁻¹] SmA 127 °C [0.8 kJ mol⁻¹] I.

N-Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium tetrafluorobromate **Gua(14,6)BF₄**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (50.0 mg, 81.1 μmol), potassium tetrafluorobromate (32.0 mg, 254 μmol)

Colourless solid (quant., 54.0 mg, 81 μmol); ¹H-NMR (500 MHz, CDCl₃): δ = 0.88 (t, *J* = 6.8 Hz, 3H, CH₃), 1.21–1.56 (m, 26H, 13 × CH₂), 1.69–1.84 (m, 6H, 2 × OCH₂CH₂, NCH₂CH₂), 2.90–3.22 (m, 14H, 2 × N(CH₃)₂, NCH₂), 3.98 (t, *J* = 6.4 Hz, 4H, 2 × OCH₂), 6.89–6.96 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H) ppm; ¹³C-NMR (126 MHz, CDCl₃) δ = 14.1 (CH₃), 22.7, 25.6, 26.1, 26.4, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 31.9 (16 × CH₂), 40.0 (2 × N(CH₃)₂), 45.4 (NCH₂), 67.7, 68.1 (2 × OCH₂), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2 (C-1', C-1''), 158.3 (C-4', C-4''), 161.7 (CN(CH₃)₂) ppm; FT-IR: $\tilde{\nu}$ = 3356 (w), 3042 (w), 2917 (s), 2848 (w), 1607 (m), 1500 (s), 1472 (w), 1405 (w), 1273 (w), 1246 (s), 1177 (w), 1054 (m), 1038 (m), 899 (w), 825 (w), 809 (w), 729 (w), 646 (w), 598 (w), 517 (w) cm⁻¹; MS (ESI): *m/z* = 580 [M⁺]; HRMS (ESI): *m/z* for C₃₇H₆₂N₃O₂⁺ calc.: 580.4837 [M⁺], found: 580.4850; CHN for C₃₇H₆₂BF₄N₃O₂: calc. C 66.56 H 9.36 N 6.29, found C 66.43 H 9.30 N 6.18; DSC: Cr 62 °C [10.3 kJ mol⁻¹] SmC 89 °C [2.0 kJ mol⁻¹] SmA 183 °C [1.0 kJ mol⁻¹] I.

N-Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium hexafluorophosphate **Gua(14,6)PF₆**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (50.0 mg, 81.1 μmol), potassium hexafluorophosphate (26.0 mg, 141 μmol)

Colourless solid (92 %, 54.0 mg, 74.4 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.22–1.55 (m, 26H, $13 \times \text{CH}_2$), 1.64–1.84 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.92–3.22 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.95–4.00 (m, 4H, $2 \times \text{OCH}_2$), 6.90–6.95 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 25.6, 26.1, 26.3, 29.0, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 29.7, 31.9 ($16 \times \text{CH}_2$), 39.7 ($2 \times \text{N}(\text{CH}_3)_2$), 45.5 (NCH_2), 67.7, 68.1 ($2 \times \text{OCH}_2$), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.6 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3398 (w), 3039 (w), 2918 (w), 2850 (w), 1608 (w), 1589 (w), 1499 (m), 1472 (w), 1404 (w), 1272 (w), 1244 (m), 1176 (w), 1067 (w), 1039 (w), 1011 (w), 998 (w), 908 (m), 836 (s), 730 (s), 649 (w), 599 (w), 557 (m), 515 (w) cm^{-1} ; MS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{62}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4840; CHN for $\text{C}_{37}\text{H}_{62}\text{F}_6\text{N}_3\text{O}_2\text{P}$: calc. C 61.22 H 8.61 N 5.79, found C 61.03 H 8.49 N 5.61; DSC: Cr 51 $^\circ\text{C}$ [28.0 kJ mol^{-1}] SmA 178 $^\circ\text{C}$ [0.7 kJ mol^{-1}] I.

N-Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium acetate **Gua(14,6)OAc**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (25.0 mg, 41.0 μmol), potassium acetate (8.00 mg, 81.5 μmol)

Colourless solid (quant., 26.0 mg, 40.6 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.21–1.55 (m, 26H, $13 \times \text{CH}_2$), 1.74–1.83 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.01 (s, 3H, CH_3COO^-), 2.87–3.23 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.98 (t, J = 6.4 Hz, 4H, $2 \times \text{OCH}_2$), 6.90–6.96 (m, 4H, 3'-H, 3''-H), 7.43–7.48 (m, 4H, 2'-H, 2''-H) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 25.6, 26.1, 26.6, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 29.7, 31.9 ($16 \times \text{CH}_2$), 39.8, 40.3 ($2 \times \text{N}(\text{CH}_3)_2$), 45.2 (NCH_2), 67.7, 68.1 ($2 \times \text{OCH}_2$), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.7 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3395 (w), 3042 (w), 2918 (s), 2849 (m), 1608 (m), 1500 (s), 1472 (w), 1403 (w), 1326 (w), 1273 (m), 1248 (s), 1178 (w), 1137 (w), 1069 (w), 1039 (w), 1010 (w), 997 (w), 900 (w), 826 (w), 809 (w), 729 (w), 646 (w), 596 (w), 516 (w), 467 (w), 420 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{65}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4832; CHN for $\text{C}_{39}\text{H}_{65}\text{N}_3\text{O}_4$: calc. C 73.20 H 10.24 N 6.57, found C 73.08 H 10.18 N 6.46; DSC: Cr 62 $^\circ\text{C}$ [22.9 kJ mol^{-1}] SmC 110 $^\circ\text{C}$ [0.3 kJ mol^{-1}] SmA 142 $^\circ\text{C}$ [1.7 kJ mol^{-1}] I.

N-Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium thiocyanate **Gua(14,6)SCN**

Setup: *N*-(Bis(dimethylamino)methylene)-6-((4'-(tetradecyloxy)-[1,1'-biphenyl]-4-yl)oxy)hexan-1-aminium chloride **Gua(14,6)Cl** (25.0 mg, 41.0 μmol), potassium thiocyanate (14.0 mg, 114 μmol)

Colourless solid (93 %, 48.0 mg, 75.1 μmol); ^1H -NMR (500 MHz, CDCl_3): δ = 0.88 (t, J = 6.8 Hz, 3H, CH_3), 1.21–1.57 (m, 26H, $13 \times \text{CH}_2$), 1.70–1.85 (m, 6H, $2 \times \text{OCH}_2\text{CH}_2$, NCH_2CH_2), 2.91–3.22 (m, 14H, $2 \times \text{N}(\text{CH}_3)_2$, NCH_2), 3.95–4.02 (m, 4H, $2 \times \text{OCH}_2$), 6.91–6.96 (m, 4H, 3'-H, 3''-H), 7.42–7.48 (m, 4H, 2'-H, 2''-H), 8.25 (s, 1H, NH) ppm; ^{13}C -NMR (126 MHz, CDCl_3) δ = 14.1 (CH_3), 22.7, 25.6, 26.1, 26.5, 29.1, 29.3, 29.4, 29.4, 29.6, 29.6, 29.7, 29.7, 29.7, 29.7, 31.9 ($16 \times \text{CH}_2$), 40.1, 40.3 ($2 \times \text{N}(\text{CH}_3)_2$), 45.5 (NCH_2), 67.7, 68.1 ($2 \times \text{OCH}_2$), 114.7, 114.8 (C-3', C-3''), 127.6, 127.7 (C-2', C-2''), 133.2, 133.4 (C-1', C-1''), 158.1, 158.3 (C-4', C-4''), 161.5 ($\text{CN}(\text{CH}_3)_2$) ppm; FT-IR: $\tilde{\nu}$ = 3246 (w), 3042 (w), 2917 (s), 2848 (m), 2052 (m), 1607 (s), 1588 (m), 1499 (s), 1472 (m), 1402 (w), 1273 (m), 1245 (s), 1177 (w), 1136 (w), 1068 (w), 1039 (w), 1010 (w), 997 (w), 899 (w), 825 (w), 809 (w), 729 (w), 645 (w), 597 (w), 515 (w) cm^{-1} ; MS (ESI): m/z = 580 [M^+]; HRMS (ESI): m/z for $\text{C}_{37}\text{H}_{65}\text{N}_3\text{O}_2^+$ calc.: 580.4837 [M^+], found: 580.4840; CHN for $\text{C}_{38}\text{H}_{62}\text{N}_4\text{O}_2\text{S}$: calc. C 71.43 H 9.78 N 8.77, found C 71.21 H 9.65 N 8.49; DSC: Cr 57 $^\circ\text{C}$ [23.4 kJ mol^{-1}] SmA 175 $^\circ\text{C}$ [0.9 kJ mol^{-1}] I.

References

- [1] Dumez E, Snaith JS, Jackson RFW, McElroy AB, Overington J, Wythes MJ, Withka JM, McLellan TJ. Synthesis of Macrocyclic, Potential Protease Inhibitors Using a Generic Scaffold. *J. Org. Chem.* 2002;67:4882–4892.
- [2] Porzelle A, Woodrow MD, Tomkinson NCO. Rearrangement of Differentially Protected *N*-Arylhydroxylamines. *Eur. J. Org. Chem.* 2008;30:5135–5143.
- [3] Haenle JC, Neidhardt MM, Beardsworth S, Kirres J, Baro A, Laschat S. Cyanobiphenyl versus Alkoxybiphenyl: Which Mesogenic Unit Governs the Mesomorphic Properties of Guanidinium Ionic Liquid Crystals? *Aust. J. Chem.* 2014;67:1088–1099.
- [4] Butschies M, Sauer S, Kessler E, Siehl H-U, Claasen B, Fischer P, Frey W, Laschat S. Influence of *N*-Alkyl Substituents and Counterions on the Structural and Mesomorphic Properties of Guanidinium Salts: Experiment and Quantum Chemical Calculations. *ChemPhysChem.* 2010;11:3752–3765.
- [5] Girlanda-Junges C, Keyling-Bilger F, Schmitt G, Luu B. Effect of Cyclohexenonic Long Chain Fatty Alcohols on Neurite Outgrowth. Study on Structure-Activity Relationship. *Tetrahedron.* 1998;54:7735–7748.
- [6] Jaschinski T, Hiersemann M. {1,6}-Transannular Catalytic Asymmetric Gosteli–Claisen Rearrangement. *Org. Lett.* 2012;14:4114–4117.
- [7] Skabara PJ, Müllen K, Bryce MR, Howard JAK, Batsanov AS. The Synthesis, Redox Properties and X-ray Crystal Structures of Two New Tetrathiafulvalene-thiophene Donors. *J. Mater. Chem.* 1998;8:1719–1724.
- [8] Matsubara H, Tsukida M, Ishihara D, Kuniyoshi K, Ryu I. A Photoirradiative Phase-Vanishing Method: Efficient Generation of HBr from Alkanes and Molecular Bromine and its Use for Subsequent Radical Addition to Terminal Alkenes. *Synlett.* 2010;13:2014–2018.
- [9] Benbayer C, Kheddam N, Saïdi-Besbes S, Givenchy ET de, Guittard F, Grelet E, Safer AM, Derdour A. Synthesis and Mesomorphic Properties of Novel [1,2,3]-Triazole Mesogenic Based Compounds. *J. Mol. Struct.* 2013;1034:22–28.

- [10] Naito K. Rewritable Color Recording Media Consisting of Leuco Dye and Biphenyl Developer with a Long Alkyl Chain. *J. Mater. Chem.* 1998;8:1379–1384.
- [11] Kong X, He Z, Zhang Y, Mu L, Liang C, Chen B, Jing X, Cammidge AN. A Mesogenic Triphenylene–Perylene–Triphenylene Triad. *Org. Lett.* 2011;13:764–767.
- [12] Mary A, Renko DZ, Guillou C, Thal C. Potent Acetylcholinesterase Inhibitors: Design, Synthesis, and Structure–Activity Relationships of Bis-Interacting Ligands in the Galanthamine Series. *Bioorg. Med. Chem.* 1998;6:1835–1850.
- [13] Halby L, Champion C, Sénamaud-Beaufort C, Ajjan S, Drujon T, Rajavelu A, Ceccaldi A, Jurkowska R, Lequin O, Nelson WG, Guy A, Jeltsch A, Guianvarc’h D, Ferroud C, Arimondo PB. Rapid Synthesis of New DNMT Inhibitors Derivatives of Procainamide. *ChemBioChem.* 2012;13:157–165.
- [14] Li Y, Marks TJ. Organolanthanide-Catalyzed Intramolecular Hydroamination/Cyclization of Aminoalkynes. *J. Am. Chem. Soc.* 1996;118:9295–9306.
- [15] Butschies M, Haenle JC, Tussetschläger S, Laschat S. Liquid Crystalline Guanidinium Phenylalkoxybenzoates: Towards Room Temperature Liquid Crystals via Bending of the Mesogenic Core and the Use of Triflate Counter Ions. *Liq. Cryst.* 2013;40:52–71.

NMR Spectra: *Ortho*-substituted Aryl Guanidinium Salt ArGua(10,10)Cl

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Oct27-2014/ 120/ fid
Title	Oct27-2014.120.fid
Comment	2 Trbojev ic TRB06-dry raw product
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl ₃
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	81
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
Acquisition Date	2014-10-27T10:55:00
Modification Date	2014-10-27T10:55:33
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1252.5
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_500/ nmr/ Nov07-2014/ 60/ fid
Title	Nov07-2014.60.fid
Comment	2 Trbojevic TRB-08
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	28
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2014-11-08T10:37:00
Modification Date	2014-11-08T10:37:42
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2097.9
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

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Analysis Info

Analysis Name Trbojevic-TRB-13_14_01_26361.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-13
Comment

Acquisition Date 20.11.2014 15:43:05

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 200 m/z
Scan End 2500 m/z
Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 600.0 Vpp

Set Nebulizer 0.4 Bar
Set Dry Heater 200 °C
Set Dry Gas 4.0 l/min
Set Divert Valve Waste

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_500/ nmr/ Nov17-2014/ 110/ fid
Title	Nov17-2014.110.fid
Comment	2 Trbojevic TRB15
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	228
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2014-11-17T18:31:00
Modification Date	2014-11-17T18:31:13
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2096.1
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

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Analysis Info

Analysis Name Trbojevic-TRB-15_15_01_26362.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-15
Comment

Acquisition Date 20.11.2014 15:49:09

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	200 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_500/ nmr/ Nov18-2014/ 110/ fid
Title	Nov18-2014.110.fid
Comment	2 Trbojevic TRB16
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	64
Receiver Gain	362
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2014-11-18T20:31:00
Modification Date	2014-11-18T20:31:18
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2090.8
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-12_13_01_26487.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-12
Comment

Acquisition Date 27.11.2014 13:10:17

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	200 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

NMR Spectra: Alkyl Gudnidinium Salts Gua(n,m)X

Parameter	Value
Title	Nov21-2014.230.fid
Comment	2 Trbojevic TRB14
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	322
Relaxation Delay	1.0000
Pulse Width	11.0500
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1246.0
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Nov21-2014/ 231/ fid
Title	Nov21-2014.231.fid
Comment	2 Trbojevic TRB14
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	512
Receiver Gain	3250
Relaxation Delay	2.0000
Pulse Width	11.7500
Presaturation Frequency	
Acquisition Time	1.8176
Acquisition Date	2014-11-21T21:39:00
Modification Date	2014-11-21T21:39:41
Class	
Spectrometer Frequency	75.48
Spectral Width	18028.8
Lowest Frequency	-1468.1
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Dec02-2014/ 370/ fid
Title	Dec02-2014.370.fid
Comment	2 Trbojevic TRB18
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	287
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
Acquisition Date	2014-12-03T01:54:00
Modification Date	2014-12-03T01:54:44
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1246.5
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Dec02-2014/ 371/ fid
Title	Dec02-2014.371.fid
Comment	2 Trbojevic TRB18
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	512
Receiver Gain	2580
Relaxation Delay	2.0000
Pulse Width	11.7500
Presaturation Frequency	
Acquisition Time	1.8176
Acquisition Date	2014-12-03T02:29:00
Modification Date	2014-12-03T02:29:24
Class	
Spectrometer Frequency	75.48
Spectral Width	18028.8
Lowest Frequency	-1468.1
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Alkoxy-biphenyl Derivatives 6a–e

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Oct27-2014/ 110/ fid
Title	Oct27-2014.110.fid
Comment	2 Trbojev ic TRB03-product
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	101
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
Acquisition Date	2014-10-27T10:48:00
Modification Date	2014-10-27T10:48:29
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1239.3
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value

Title	Mar09-2015.20.fid
Comment	2 Trbojevic TRB46
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	296.4
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	322
Relaxation Delay	1.0000
Pulse Width	11.0500
Class	

Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1241.5
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_500/ nmr/ Oct21-2014/ 20/ fid
Title	Oct21-2014.20.fid
Comment	2 Trbojevic TRB01-RackII,III 100C
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	364.9
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	114
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2014-10-21T10:03:00
Modification Date	2014-10-21T10:03:38
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2093.1
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Title	Mar04-2015.21.fid
Comment	2 Trbojevic TRB42-1-318K
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	316.3
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	228
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2100.9
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_500/ nmr/ Nov03-2014/ 1000/ fid
Title	Nov03-2014/ 1000
Comment	02 Trbojevic TRB04-1-373K
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	364.9
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	181
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2014-11-03T13:40:05
Modification Date	2014-11-03T13:40:31
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2120.6
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Alkyl Phthalimides 11a–e

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Nov 03-2014/ 170/ fid
Title	Nov 03-2014.170.fid
Comment	2 Trbojevic TRB05-2
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl ₃
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	161
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
Acquisition Date	2014-11-03T12:43:00
Modification Date	2014-11-03T12:44:02
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1240.9
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Oct21-2014/ 90/ fid
Title	Oct21-2014.90.fid
Comment	2 Trbojevic TRB 02-3 filtered solid
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	81
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
Acquisition Date	2014-10-21T09:43:00
Modification Date	2014-10-21T09:44:06
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1235.0
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Title	Mar04-2015.340.fid
Comment	2 Trbojevic TRB45 clean
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.7
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	322
Relaxation Delay	1.0000
Pulse Width	11.0500
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1245.8
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_300/ nmr/ Dec02-2014/ 70/ fid
Title	Dec02-2014.70.fid
Comment	2 Trbojevic TRB21
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	296.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z862701/ 0034
Number of Scans	16
Receiver Gain	322
Relaxation Delay	1.0000
Pulse Width	11.0500
Presaturation Frequency	
Acquisition Time	2.6477
Acquisition Date	2014-12-02T09:36:00
Modification Date	2014-12-02T09:36:49
Class	
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1246.1
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Biphenyl Phthalimides 12a-i

Analysis Info

Acquisition Date 27.11.2014 11:07:05

Operator	wo/tri	
Instrument	microTOF-Q	43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Parameter	Value

Title	Mar11-2015.90.fid
Comment	2 Trbojevic TRB47
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	181
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-fl-TRB-47_5_01_27892.d
Method loop-tune-wide.m
Sample Name Trbojevic-fl-TRB-47
Comment

Acquisition Date 16.03.2015 08:29:11

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 250 m/z
Scan End 2500 m/z
Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 600.0 Vpp

Set Nebulizer 1.0 Bar
Set Dry Heater 200 °C
Set Dry Gas 8.0 l/min
Set Divert Valve Waste

D:\Xcalibur\data\trbojevic-trb-07
EI positiv-Ion, 70eV

Tquelle=215°C
Tprobe=310°C

11/7/2014 1:34:25 PM
C40H53NO4

TRB-07
Nina Trbojevic

trbojevic-trb-07 #134-140 RT: 9.80-10.23 AV: 7 NL: 6.93E7
T: + c EI Full ms [34.50-850.50]

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-50.d
Method loop-tune-low-apci.m
Sample Name TRB-50
Comment

Acquisition Date 20.03.2015 09:08:00

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type APCI
Focus Not active
Scan Begin 50 m/z
Scan End 2000 m/z

Ion Polarity Positive
Set Capillary 4000 V
Set End Plate Offset -500 V
Set Collision Cell RF 150.0 Vpp

Set Nebulizer 1.7 Bar
Set Dry Heater 300 °C
Set Dry Gas 5.0 l/min
Set Divert Valve Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-23_7_01_26697.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-23
Comment

Acquisition Date 11.12.2014 10:41:07

Operator wo/tri
Instrument microTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	200 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Data File Name	N:/ data/ TRBOJEVIC_500/ nmr/ Dec04-2014/ 90/ fid
Title	Dec04-2014.90.fid
Comment	2 Trbojevic TRB24
Origin	Bruker BioSpin GmbH
Owner	guest
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	203
Relaxation Delay	2.0000
Pulse Width	11.2300
Presaturation Frequency	
Acquisition Time	1.5860
Acquisition Date	2014-12-04T20:32:00
Modification Date	2014-12-04T20:32:33
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2093.5
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-24.d
Method loop-tune-wide.m
Sample Name TRB-24
Comment geloest in Methanol

Acquisition Date 05.12.2014 10:39:23

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type APCI
Focus Not active
Scan Begin 200 m/z
Scan End 2500 m/z

Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 600.0 Vpp

Set Nebulizer 1.7 Bar
Set Dry Heater 300 °C
Set Dry Gas 5.0 l/min
Set Divert Valve Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-48.d
Method loop-tune-low-apci.m
Sample Name TRB-48
Comment

Acquisition Date 20.03.2015 08:09:55

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type APCI
Focus Not active
Scan Begin 50 m/z
Scan End 2000 m/z

Ion Polarity Positive
Set Capillary 4000 V
Set End Plate Offset -500 V
Set Collision Cell RF 150.0 Vpp

Set Nebulizer 1.7 Bar
Set Dry Heater 300 °C
Set Dry Gas 5.0 l/min
Set Divert Valve Waste

Massenspektrometrie - Universität Stuttgart

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-49.d
Method loop-tune-low-apci.m
Sample Name TRB-49
Comment

Acquisition Date 20.03.2015 08:46:53

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type APCI
Focus Not active
Scan Begin 50 m/z
Scan End 2000 m/z

Ion Polarity Positive
Set Capillary 4000 V
Set End Plate Offset -500 V
Set Collision Cell RF 150.0 Vpp

Set Nebulizer 1.7 Bar
Set Dry Heater 300 °C
Set Dry Gas 5.0 l/min
Set Divert Valve Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojeviy-TRB-10_12_01_26281.d
Method loop-tune-low.m
Sample Name Trbojeviy-TRB-10
Comment

Acquisition Date 17.11.2014 14:29:24

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	24.7 psi
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	5.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Amines 13a-i

Parameter	Value
Title	Feb18-2015.60.fid
Comment	2 Trbojevic TRB38
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	364.9
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	575
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2094.3
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-38.d
Method loop-tune-low.m
Sample Name TRB-38
Comment

Acquisition Date 24.02.2015 11:08:49
Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	160.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-51_7_01_28043.d
Method loop-tune-low.m
Sample Name Trbojevic-TRB-51
Comment

Acquisition Date 07.04.2015 11:16:47

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 50 m/z
Scan End 1000 m/z
Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 160.0 Vpp

Set Nebulizer 1.0 Bar
Set Dry Heater 200 °C
Set Dry Gas 8.0 l/min
Set Divert Valve Waste

Parameter	Value
Title	Feb11-2015.150.fid
Comment	2 Trbojevic TRB-35
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	366.7
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	575
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2095.2
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-35.d
Method loop-tune-wide.m
Sample Name TRB-35
Comment

Acquisition Date 17.02.2015 10:54:02

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

— 158.08
 — 132.72
 — 127.15
 — 115.24
 — 68.09
 41.67
 33.47
 31.14
 28.92
 28.85
 28.81
 28.78
 28.61
 28.49
 26.36
 25.45
 21.84
 13.53

Parameter	Value
Title	Mar31-2015.31.fid
Comment	2 Trbojevic TRB-54-373K
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	364.8
Pulse Sequence	zgpg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	1024
Receiver Gain	2890
Relaxation Delay	2.0000
Pulse Width	10.2000
Class	
Spectrometer Frequency	125.78
Spectral Width	32894.7
Lowest Frequency	-3934.0
Nucleus	¹³ C
Acquired Size	32768
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-54_10_01_28052.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-54
Comment

Acquisition Date 07.04.2015 13:50:11

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Title	Feb13-2015.20.fid
Comment	2 Trbojevic TRB37
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	364.9
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	575
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2094.2
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name	Trbojevic-TRB-37_7_01_27553.d
Method	loop-tune-wide.m
Sample Name	Trbojevic-TRB-37
Comment	

Acquisition Date 17.02.2015 10:38:06

Operator	wo/tri	
Instrument	micrOTOF-Q	43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Title	Feb16-2015.100.fid
Comment	2 Trbojevic TRB33
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	364.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	645
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2094.6
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-33.d
Method loop-tune-low.m
Sample Name TRB-33
Comment

Acquisition Date 18.02.2015 11:18:13

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	160.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Title	Mar31-2015.40.fid
Comment	2 Trbojevic TRB-52-373K
Origin	Bruker BioSpin GmbH
Solvent	DMSO
Temperature	364.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	362
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2097.0
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-52_8_01_28044.d
Method loop-tune-low.m
Sample Name Trbojevic-TRB-52
Comment

Acquisition Date 07.04.2015 11:20:39

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 50 m/z
Scan End 1000 m/z
Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 160.0 Vpp

Set Nebulizer 1.0 Bar
Set Dry Heater 200 °C
Set Dry Gas 8.0 l/min
Set Divert Valve Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-17_31_01_26575.d
Method loop-tune-low.m
Sample Name Trbojevic-TRB-17
Comment

Acquisition Date 02.12.2014 14:03:08

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	150.0 Vpp	Set Divert Valve	Waste

Guanidinium Chlorides Gua(n,m)Cl

Parameter	Value
Title	Mar27-2015.140.fid
Comment	2 Trbojev ic TRB43
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	287
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2088.5
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-43_6_01_28036.d
Method loop-tune-low.m
Sample Name Trbojevic-TRB-43
Comment

Acquisition Date 07.04.2015 10:38:58

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	1000 m/z	Set Collision Cell RF	160.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Title	Apr20-2015.90.fid
Comment	2 Trbojevic TRB-55
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	228
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2087.6
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-55_18_01_28280.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-55
Comment

Acquisition Date 21.04.2015 14:53:30

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-58_22_01_28284.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-58
Comment

Acquisition Date 21.04.2015 15:08:45

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-39_5_01_28035.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-39
Comment

Acquisition Date 07.04.2015 10:35:12

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-34_3_01_28031.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-34
Comment

Acquisition Date 07.04.2015 08:35:27

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 250 m/z
Scan End 2500 m/z
Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 600.0 Vpp

Set Nebulizer 1.0 Bar
Set Dry Heater 200 °C
Set Dry Gas 8.0 l/min
Set Divert Valve Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-56_19_01_28281.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-56
Comment

Acquisition Date 21.04.2015 14:57:19

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-57_20_01_28283.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-57
Comment

Acquisition Date 21.04.2015 15:05:02

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-32_2_01_28029.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-32
Comment

Acquisition Date 07.04.2015 08:27:53

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Guanidinium Salts Gua(14,6)X

Parameter	Value
Data File Name	C:/Users/Nindza/Desktop/May 21-2015/ 260/ fid
Title	May 21-2015.260.fid
Comment	2 Trbojevic TRB77
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	256
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2088.6
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-77_7_01_28781.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-77
Comment

Acquisition Date 26.05.2015 09:24:41

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-78_8_01_28765.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-78
Comment

Acquisition Date 26.05.2015 08:26:18

Operator wo/tri
Instrument micrOTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 250 m/z
Scan End 2500 m/z

Ion Polarity Positive
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 600.0 Vpp

Set Nebulizer 1.0 Bar
Set Dry Heater 200 °C
Set Dry Gas 8.0 l/min
Set Divert Valve Waste

Parameter	Value
Title	May21-2015.280.fid
Comment	2 Trbojevic TRB79
Origin	Bruker BioSpin GmbH
Solvent	CDCl ₃
Temperature	300.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	362
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2092.2
Nucleus	¹ H
Acquired Size	16384
Spectral Size	65536

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Analysis Info

Analysis Name	Trbojevic-TRB-79_1_01_29334.d
Method	loop-tune-wide.m
Sample Name	Trbojevic-TRB-79
Comment	

Acquisition Date 12.06.2015 08:51:14

Operator	wo/tri	
Instrument	micrOTOF-Q	43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

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Analysis Info

Analysis Name Trbojevic-TRB-79_1_01_29279.d
Method loop-tune-low-negativ.m
Sample Name Trbojevic-TRB-79
Comment

Acquisition Date 11.06.2015 10:04:51

Operator wo/tri
Instrument microTOF-Q 43

Acquisition Parameter

Source Type ESI
Focus Not active
Scan Begin 50 m/z
Scan End 1000 m/z
Ion Polarity Negative
Set Capillary 4500 V
Set End Plate Offset -500 V
Set Collision Cell RF 160.0 Vpp

Set Nebulizer 1.0 Bar
Set Dry Heater 200 °C
Set Dry Gas 8.0 l/min
Set Divert Valve Waste

Parameter	Value
Title	May20-2015.260.fid
Comment	2 Trbojevic TRB-80
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	300.0
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	287
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	

Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2091.4
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

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Analysis Info

Analysis Name Trbojevic-TRB-80_9_01_28767.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-80
Comment

Acquisition Date 26.05.2015 08:33:36

Operator wo/tri
Instrument microTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Parameter	Value
Title	May20-2015.280.fid
Comment	2 Trbojevic TRB-82
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.8
Pulse Sequence	zg30
Experiment	1D
Probe	5 mm PABBO BB-1H/ D Z-GRD Z800701/ 0072
Number of Scans	32
Receiver Gain	322
Relaxation Delay	2.0000
Pulse Width	11.2300
Class	
Spectrometer Frequency	500.16
Spectral Width	10330.6
Lowest Frequency	-2090.2
Nucleus	1H
Acquired Size	16384
Spectral Size	65536

Massenspektrometrie - Universität Stuttgart

Analysis Info

Analysis Name Trbojevic-TRB-82_10_01_28768.d
Method loop-tune-wide.m
Sample Name Trbojevic-TRB-82
Comment

Acquisition Date 26.05.2015 08:37:18

Operator wo/tri
Instrument microTOF-Q 43

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.0 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	250 m/z	Set End Plate Offset	-500 V	Set Dry Gas	8.0 l/min
Scan End	2500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

DSC Curves of Gua(n,m)X

ArGua(10,10)Cl Gua(6,6)Cl

Gua(6,6)Cl

Gua(8,8)Cl

Gua(10,10)Cl

Gua(12,12)Cl

Gua(14,14)Cl

Gua(6,14)Cl

Gua(8,12)Cl

Gua(12,8)Cl

Gua(14,6)Cl

Gua(14,6)Br

Gua(14,6)I

Gua(14,6)OTf

Gua(14,6)BF₄

Gua(14,6)PF₆

Gua(14,6)OAc

Gua(14,6)SCN

Table 1. Phase transitions temperatures / °C (and corresponding enthalpies / kJ mol⁻¹) of guanidinium chlorides **Gua(n,m)Cl**.

	Cr ₁			Cr _x			SmC		SmA		I	
Gua(6,6)Cl	•	58	(-0.9)	•	73	(-4.8)	—		•	132	(-5.2)	• 2 nd heat
	•	71	(2.5)	•	75	(1.9)	—		•	133	(3.0)	• 2 nd cool
Gua(8,8)Cl	• ^a	67	(-5.1)	•	101	(-6.4)	—		•	129	(-3.5)	• 2 nd heat
	• ^a	69	(5.1)	•	101	(11.0)	—		•	130	(2.7)	• 2 nd cool
Gua(10,10)Cl	•	76	(-26.4)	•	113	(-4.8)	—		•	140	(-5.4)	• 2 nd heat
	•	77	(25.4)	•	117	(4.9)	• ^b 118	—	•	143	(5.7)	• 2 nd cool
Gua(12,12)Cl	•	94	(-15.4)	•	124	(-12.4)	—		•	132	(-10.1)	• 2 nd heat
	•	90	(20.1)	•	125	(12.1)	• ^b 126	—	•	134	(9.4)	• 2 nd cool
Gua(14,14)Cl	•	100	(-25.7)	•	124	(-15.6)	—		•	140	(-10.3)	• 2 nd heat
	•	97	(23.3)	•	128	(15.1)	• ^b 129	—	•	145	(9.0)	• 2 nd cool

^a additional Cr-Cr transition at 56 °C (2nd heat), 69 °C (2nd cool); ^b mesophase indicated by POM textures upon cooling; Cr crystalline; SmC smectic C; SmA smectic A; I isotropic; • observed; — not observed; cooling rate 5 K min⁻¹.

Table 2. Phase transitions temperatures / °C (and corresponding enthalpies / kJ mol⁻¹) of guanidinium chlorides **Gua(n,m)Cl**

	Cr ₁			Cr _x			SmC		SmA		I	
Gua(6,14)Cl	• ^a	77	(-3.1)	•	130	(-7.5)	—		•	142	(-6.6)	• 2 nd heat
	• ^a	78	(3.5)	•	131	(7.0)	—		•	151	(6.5)	• 2 nd cool
Gua(8,12)Cl	•	95	(-9.9)	•	142	(-27.4)	—		—			• 2 nd heat
	•	95	(8.8)	•	143	(24.3)	—		—			• 2 nd cool
Gua(10,10)Cl	•	76	(-26.4)	•	113	(-4.8)	—		•	140	(-5.4)	• 2 nd heat
	•	77	(25.4)	•	117	(4.9)	• ^b 118	—	•	143	(5.7)	• 2 nd cool
Gua(12,8)Cl	•	86	(-21.8)	—			• 103	(-1.1)	•	155	(-2.4)	• 2 nd heat
	•	87	(22.0)	—			• 101	(2.6)	•	156	(1.6)	• 2 nd cool
Gua(14,6)Cl^c	•	65	(-24.0)	—			• 84	(-2.8)	•	193	(-1.0)	• 2 nd heat
	•	66	(8.0)	—			• 93	(3.5)	•	194	(0.9)	• 2 nd cool

^a additional Cr-Cr transition at 57 °C (2nd heat), 53 °C (2nd cool); ^b mesophase indicated by POM textures upon cooling; ^c Clearing transitions decreased upon successive heating and cooling cycles; Cr crystalline; SmC smectic C; SmA smectic A; I isotropic; • observed; — not observed; cooling rate 5 K min⁻¹.

Table 3. Phase transitions temperatures / °C (and corresponding enthalpies / kJ mol⁻¹) of guanidinium salts **Gua(14,6)X** with different anions.

Gua(14,6)	Cr			Cr_x			SmC		SmA		I	
Br^a	•	74	(-21.6)	–			–		•	192	(-1.1)	• 2 nd heat
	•	72	(18.4)	•	80	(3.0)	–		•	187	(1.2)	• 2 nd cool
I^a	•	112	(-28.5)	–			–		•	193	(-0.9)	• 2 nd heat
	•	80	(30.6)	–			–		•	193	(0.9)	• 2 nd cool
OTf	•	71	(-34.1)	–			–		•	126	(-0.8)	• 2 nd heat
	•	60	(40.1)	–			–		•	127	(0.8)	• 2 nd cool
BF₄^a	•	87	(-39.6)	–			–		•	195	(-0.9)	• 2 nd heat
	•	62	(10.3)	–			•	89 (2.0)	•	183	(1.0)	• 2 nd cool
PF₆	•	67	(-43.9)	•	81	(-6.4)	–		•	175	(-1.0)	• 2 nd heat
	•	51	(28.0)	–			–		•	178	(0.7)	• 2 nd cool
OAc	•	72	(-21.9)	–			•	94 (-1.4)	•	139	(-1.3)	• 2 nd heat
	•	62	(22.9)	–			•	110 (0.3)	•	142	(1.7)	• 2 nd cool
SCN^a	•	67	(-26.0)	–			–		•	173	(-0.8)	• 2 nd heat
	•	57	(23.4)	–			–		•	175	(0.9)	• 2 nd cool

^a Clearing transitions decreased upon successive heating and cooling cycles; Cr crystalline; SmC smectic C; SmA smectic A; I isotropic; • observed; – not observed; cooling rate 5 K min⁻¹.

Table 4. Phase transitions temperatures / °C (and corresponding enthalpies / kJ mol⁻¹) of guanidinium chlorides **ArGua(n,m)Cl**.

	Cr₁		Cr₂		Cr₃		I	
ArGua(10,10)Cl	•					145 (-68.6)	•	2 nd heat
	•	85 (13.8)	•	91 (5.9)	•	97 (17.1)	•	2 nd cool

Cr crystalline; I isotropic; • observed; – not observed; cooling rate 5 K min⁻¹.

DSC and TG measurement

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XRD Layer Spacing d_{001} of Gua(n,m)X