

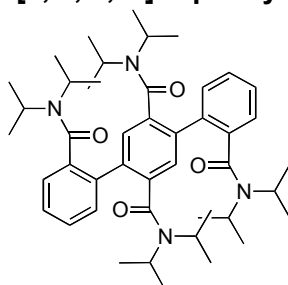
Supplementary information for

Transmitting information along oligoparaphenylenes: 1,12- stereochemical control in a terphenyl tetracarboxamide.

Jonathan Clayden, Lluís Vallverdú and Madeleine Helliwell

General experimental details have been reported previously.¹

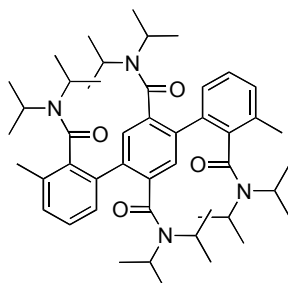
N,N,N',N',N'',N''',N''''-Octaisopropyl-[1,1';4',1'']terphenyl-2,2',5',2''-tetracarboxamide 3



2-(*N,N*-Diisopropylcarboxamido)phenylboronic acid² (300 mg, 1.20 mmol), Pd(PPh₃)₄ (76 mg, 0.066 mmol), and Na₂CO₃ 2M aqueous solution (15 mL) were added to a solution of 2,5-diiodo-*N,N,N',N'*-tetraisopropylterephthalamide³ (260 mg, 0.44 mmol) in toluene (20 mL). The mixture was stirred at reflux overnight. The mixture was partitioned between AcOEt and saturated aqueous NH₄Cl aq, and the aqueous phase was extracted with AcOEt. The organic extracts were dried, filtered and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography (Petrol to petrol:AcOEt 40%) to yield tetramide **3** (284 mg, 86%).

¹H-NMR (CDCl₃, 500 MHz) δ 0.38 (d, *J* = 6.5 Hz, 3H), 0.81 (d, *J* = 6.5 Hz, 3H), 0.97-1.10 (m, 9H), 1.13 (d, *J* = 6.5 Hz, 3H), 1.16 (d, *J* = 6.5 Hz, 3H), 1.22 (d, *J* = 6.5 Hz, 3H), 1.27 (d, *J* = 6.5 Hz, 3H), 1.30-1.30 (m, 3H), 1.35 (d, *J* = 6.5 Hz, 3H), 1.39 (d, *J* = 6.5 Hz, 3H), 1.40 (d, *J* = 6.5 Hz, 3H), 1.38-1.43 (m, 3H), 1.43 (d, *J* = 6.5 Hz, 3H), 1.53 (d, *J* = 6.5 Hz, 3H), 3.16 (septet, *J* = 6.5 Hz, 1H), 3.25-3.36 (m, 2H), 3.42 (septet, *J* = 6.5 Hz, 1H), 3.78 (septet, *J* = 6.5 Hz, 1H), 3.85-3.90 (m, 1H), 3.92 (septet, *J* = 6.5 Hz, 1H), 4.07-4.19 (m, 1H), 7.10 (br s, Hz, 1H), 7.13-7.18 (m, 3H), 7.20-7.25 (m, 5H), 7.40-7.72 (m, 1H). ¹³C-NMR (CDCl₃, 75.5 MHz) δ 19.5 (CH₃), 20.28 (CH₃), 20.36 (CH₃), 20.45 (CH₃), 20.5 (CH₃), 20.6 (CH₃), 20.8 (CH₃), 21.2 (CH₃), 45.6 (CH), 45.8 (CH), 50.3 (CH), 50.6 (CH), 125.7 (CH), 126.5 (CH), 127.0 (CH), 127.2 (CH), 127.4 (CH), 127.8 (CH), 129.8 (Cq), 132.2 (CH), 134.9 (Cq), 136.1 (Cq), 136.5 (Cq), 137.5 (Cq), 137.9 (Cq), 138.0 (Cq), 138.2 (Cq), 168.9 (Cq), 169.4 (Cq), 170.1 (Cq). IR (film) 1635 cm⁻¹. HRMS Calcd for C₄₆H₆₆N₄O₄Na: 761.4976. Found 761.4969. Anal. Calcd for C₄₆H₆₆N₄O₄·⁵/₄CH₂Cl₂: C, 67.16; H, 8.17; N, 6.63. Found: C, 67.16; H, 8.21; N, 6.47.

N,N,N',N',N'',N''',N''''-Octaisopropyl-3,3''-dimethyl-[1,1';4',1'']terphenyl-2,2',5',2''-tetracarboxamide 5



To a solution of **3** (100 mg, 0.13 mmol) in dry THF (10 mL) was added *N,N,N',N'*-tetramethylethylenediamine (204 μL, 1.35 mmol) and the solution was cooled to -78 °C under nitrogen and stirred for 5 min. *s*-Butyllithium (1.06 mL, 1.35 mmol, 1.28 M in hexanes) was added and the mixture was stirred for 1 h. Methyl iodide (168 μL, 2.70 mmol) was added and the mixture was stirred for 30 min. The solution was allowed to warm to room temperature and the mixture was

J. Clayden, L. Vallverdú, M. Helliwell:
Transmitting information along oligoparaphenylenes: 1,12- stereochemical control in a terphenyl tetracarboxamide

partitioned between AcOEt and H₂O. The aqueous phase was extracted with AcOEt, and the organic extracts were dried, filtered and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography (Petrol to petrol:AcOEt 30%) to yield **5** (22 mg, 21%):

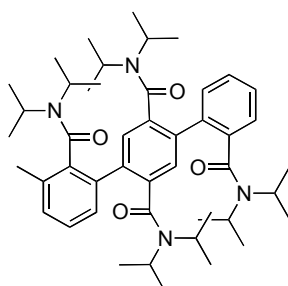
M.p. 278-280 °C. ¹H-NMR (CDCl₃, 300 MHz) δ 0.66 (d, *J* = 6.6Hz, 6H), 1.02 (d, *J* = 6.6Hz, 6H), 1.31 (d, *J* = 6.9Hz, 18H), 1.36 (d, *J* = 6.6Hz, 6H), 1.45 (d, *J* = 6.9Hz, 6H), 1.51 (d, *J* = 6.9 Hz, 6H), 2.36 (s, 6H), 3.27 (septet, *J* = 6.9 Hz, 2H), 3.49 (septet, *J* = 6.9 Hz, 2H), 3.90 (septet, *J* = 6.6 Hz, 2H), 4.30 (septet, *J* = 6.6 Hz, 2H), 7.04 (d, *J* = 6.6 Hz, 1H), 7.05 (d, *J* = 6.6 Hz, 1H), 7.12-7.16 (m, 4H), 8.03 (s, 2H).

¹³C-NMR (CDCl₃, 75.5 MHz) δ 19.3 (CH₃), 19.9 (CH₃), 20.1 (CH₃), 20.2 (CH₃), 20.4 (CH₃), 20.88 (CH₃), 20.92 (CH₃), 21.2 (CH₃), 22.7 (CH₃), 45.7 (CH), 45.9 (CH), 50.9 (CH), 51.2 (CH), 126.5 (CH), 128.2 (CH), 129.2 (CH), 129.6 (CH), 134.3 (Cq), 134.7 (Cq), 137.2 (Cq), 137.3 (Cq), 138.1 (Cq), 168.7 (Cq), 169.6 (Cq).

IR (film) 1634cm⁻¹

HRMS Calcd for C₄₈H₇₁N₄O₄: 767.5470. Found 767.5474.

N,N,N',N',N'',N''',N''''-Octaisopropyl-3-methyl-[1,1';4',1''']terphenyl-2,2',5',2''-tetracarboxamide 4

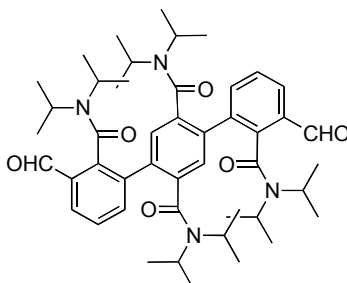


Also obtained from the reaction described above was **4** (45mg, 44%).

¹H-NMR (CDCl₃, 300 MHz) δ 0.64 (d, *J* = 6.6 Hz, 3H), 0.73 (d, *J* = 6.6 Hz, 3H), 0.97 (d, *J* = 6.6 Hz, 3H), 1.01 (d, *J* = 6.6 Hz, 3H), 1.18-1.38 (m, 24H), 1.44 (d, *J* = 6.9 Hz, 3H), 1.45 (d, *J* = 6.9 Hz, 3H), 1.52 (d, *J* = 6.9 Hz, 6H), 2.36 (s, 3H), 3.22-3.34 (m, 2H), 3.48 (septet, *J* = 6.6 Hz, 1H), 3.49 (septet, *J* = 6.9 Hz, 1H), 3.87 (septet, *J* = 6.6 Hz, 1H), 3.90 (septet, *J* = 6.9 Hz, 1H), 4.31 (septet, *J* = 6.6 Hz, 2H), 7.04-7.33 (m, 7H), 8.00 (d, *J* = 7.02 Hz, 2H).

¹³C-NMR (CDCl₃, 75.5 MHz) δ 19.4 (CH₃), 19.8 (CH₃), 19.89 (CH₃), 19.93 (CH₃), 20.1 (CH₃), 20.2 (CH₃), 20.4 (CH₃), 20.5 (CH₃), 20.9 (CH₃), 21.0 (CH₃), 21.2 (CH₃), 21.8 (CH₃), 22.6 (CH₃), 45.6 (CH₂), 45.77 (CH₂), 45.78 (CH₂), 45.9 (CH₂), 50.87 (CH₂), 50.93 (CH₂), 51.0 (CH₂), 51.1 (CH₂), 126.59 (CH), 126.64 (CH), 127.1 (CH), 127.6 (CH), 128.1 (CH), 129.1 (CH), 129.3 (CH), 129.6 (CH), 130.5 (CH), 134.3 (Cq), 134.7 (Cq), 136.8 (Cq), 137.2 (Cq), 137.3 (Cq), 138.0 (Cq), 138.1 (Cq), 168.5 (Cq), 168.6 (Cq), 169.5 (Cq), 170.3 (Cq). IR (film) 1633 cm⁻¹. HRMS Calcd for C₄₇H₆₉N₄O₄: 753.5313. Found: 753.5310.

3,3''-Diformyl-N,N,N',N',N'',N''',N''''-octaisopropyl-[1,1';4',1''']terphenyl-2,2',5',2''-tetracarboxamide 7



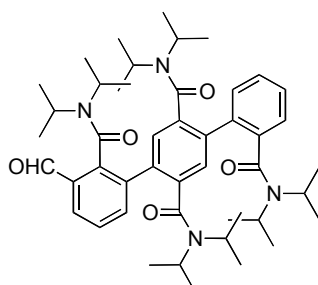
To a solution of **3** (332 mg, 0.45 mmol) in dry THF (60 mL) was added *N,N,N',N'*-tetramethylethylenediamine (0.7 mL, 4.64 mmol) and the solution was cooled at -78°C under nitrogen and stirred for 5 min. *s*-Butyllithium (3.80 mL, 4.56 mmol, 1.2 M in hexanes) was added and the mixture was stirred for 1 h. Dry DMF (1 mL) was added and the mixture was stirred for 1h. The solution was allowed to warm to room temperature and stirred for 1h. The mixture was partitioned between AcOEt and H₂O and the aqueous phase was extracted with AcOEt. The organic extracts were dried, filtered and the solvent was removed under reduced pressure. The crude product was

J. Clayden, L. Vallverdú, M. Helliwell:
Transmitting information along oligoparaphenylenes: 1,12- stereochemical control in a terphenyl tetracarboxamide

purified by flash chromatography (Petrol to petrol:AcOEt 50%) to yield recovered starting material (73mg, 22%), **6** (130 mg, 38%), and **7** (72 mg, 20%).

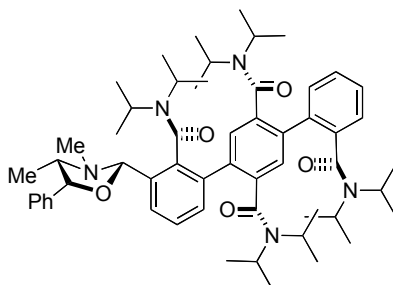
¹H-NMR (CDCl₃, 300 MHz) δ 0.71 (d, *J* = 6.6 Hz, 6H), 0.89 (d, *J* = 6.9 Hz, 6H), 0.90 (d, *J* = 6.9 Hz, 6H), 0.98 (d, *J* = 6.6 Hz, 6H), 1.30-1.36 (m, 12H), 1.44 (d, *J* = 6.9 Hz, 6H), 1.55 (d, *J* = 6.9 Hz, 6H), 3.35 (septet, *J* = 6.9 Hz, 2H), 3.51 (septet, *J* = 6.6 Hz, 2H), 3.89 (septet, *J* = 6.6 Hz, 2H), 4.33 (septet, *J* = 6.9 Hz, 2H), 7.42 (d, *J* = 7.2 Hz, 2H), 7.44 (d, *J* = 7.2 Hz, 2H), 7.95 (dd, *J* = 7.5 and 1.5 Hz, 2H), 8.10 (s, 2H), 10.16 (s, 2H). ¹³C-NMR (CDCl₃, 75.5 MHz) δ 20.17 (CH₃), 20.29 (CH₃), 20.34 (CH₃), 20.4 (CH₃), 20.8 (CH₃), 21.3 (CH₃), 21.4 (CH₃), 22.7 (CH₃), 46.0 (CH₂), 46.5 (CH₂), 51.1 (CH₂), 51.6 (CH₂), 127.4 (CH), 128.0 (CH), 129.2 (CH), 133.2 (CH), 135.2 (Cq), 136.4 (Cq), 136.6 (Cq), 138.6 (Cq), 140.2 (Cq), 167.0 (Cq), 167.9 (Cq), 190.8 (CH). IR (film) 1631 cm⁻¹. HRMS Calcd for C₄₈H₆₆N₄O₆Na: 817.4875. Found: 817.4868.

3-Formyl-*N,N,N',N',N'',N''',N''',N''''*-octaisopropyl-[1,1';4,1'']terphenyl-2,2',5',2''-tetracarboxamide **6**



¹H-NMR (CDCl₃, 300 MHz) δ 0.96 (d, *J* = 6.6 Hz, 3H), 0.99 (d, *J* = 6.6 Hz, 3H), 1.53-1.89 (m, 33H), 1.43 (d, *J* = 6.6 Hz, 3H), 1.49 (d, *J* = 6.3 Hz, 3H), 1.54 (d, *J* = 6.3 Hz, 3H), 3.30 (septet, *J* = 6.9 Hz, 1H), 3.32 (septet, *J* = 6.6 Hz, 1H), 3.49 (septet, *J* = 6.3 Hz, 2H), 3.81-3.97 (m, 2H), 4.24-4.38 (m, 2H), 7.21-7.36 (m, 5H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.94 (d, *J* = 7.5 Hz, 1H), 8.01-9.07 (m, 1H), 10.16 (s, 1H). ¹³C-NMR (CDCl₃, 75.5 MHz) δ 19.8 (CH₃), 19.9 (CH₃), 20.1 (CH₃), 20.2 (CH₃), 20.4 (CH₃), 20.5 (CH₃), 20.8 (CH₃), 21.0 (CH₃), 21.1 (CH₃), 21.8 (CH₃), 22.2 (CH₃), 23.8 (CH₃), 45.6 (CH), 45.9 (CH), 46.0 (CH), 46.5 (CH), 50.4 (CH), 50.98 (CH), 51.00 (CH), 51.5 (CH), 125.9 (CH), 126.7 (CH), 127.2 (CH), 127.3 (CH), 127.7 (CH), 127.9 (CH), 128.9 (CH), 129.5 (CH), 130.5 (CH), 133.2 (Cq), 134.1 (Cq), 135.5 (Cq), 135.8 (Cq), 136.4 (Cq), 137.7 (Cq), 138.0 (Cq), 138.3 (Cq), 140.3 (Cq), 167.0 (Cq), 168.1 (Cq), 168.2 (Cq), 170.2 (Cq), 190.0 (CH). IR (film) 1632 cm⁻¹. HRMS Calcd for C₄₇H₆₆N₄O₅Na: 789.4925. Found: 789.4928.

N,N,N',N',N'',N''',N''',N''''-Octaisopropyl-3-[(2*S*,4*S*,5*R*)-3,4-dimethyl-5-phenyloxazolidin-2-yl]-[1,1';4,1'']terphenyl-2,2',5',2''-tetracarboxamide **8**



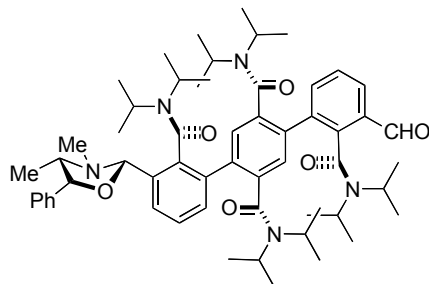
To a solution of **6** (90 mg, 0.12 mmol), in toluene (30 mL) was added (1*R*,2*S*)-(-)-ephedrine (75 mg, 0.45 mmol), and the solution was stirred at reflux under a Dean-Stark condenser for 48 h. The solvent was removed under reduced pressure and the crude product was purified by flash chromatography (Petrol to petrol:AcOEt 20% + 1% Et₃N) to yield **8** (84 mg, 78%).

¹H-NMR (C₆D₆, 300 MHz) δ 0.69 (d, *J* = 6.3 Hz, 6H), 0.73 (d, *J* = 6.3 Hz, 3H), 0.87 (d, *J* = 6.9 Hz, 3H), 0.92 (d, *J* = 6.6 Hz, 3H), 0.95 (d, *J* = 6.6 Hz, 3H), 1.10 (d, *J* = 6.6 Hz, 3H), 1.14 (d, *J* = 6.6 Hz, 3H), 1.29 (d, *J* = 6.6 Hz, 3H), 1.32 (d, *J* = 6.6 Hz, 3H), 1.54 (d, *J* = 6.9 Hz, 3H), 1.60 (d, *J* = 6.3 Hz, 3H), 1.55-1.61 (m, 3H), 1.64 (s, 3H), 1.65 (d, *J* = 6.3 Hz, 3H), 1.68 (d, *J* = 6.9 Hz, 3H), 1.76 (d, *J* = 6.6 Hz, 3H), 2.32 (s, 3H), 2.69 (dq, *J* = 8.4 and 6.6 Hz, 1H), 3.04 (septet, *J* = 6.6 Hz, 2H), 3.14 (septet, *J* = 6.6 Hz, 1H), 3.23 (septet, *J* = 6.9 Hz, 1H), 4.17 (septet, *J* = 6.3 Hz, 1H), 4.25-4.45 (m, 3H), 5.01 (d, *J* = 8.4 Hz, 1H), 5.27 (s, 1H), 7.09-7.20 (m, 6H), 7.27-7.30 (m, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 8.38 (d, *J* =

J. Clayden, L. Vallverdú, M. Helliwell:
Transmitting information along oligoparaphenylenes: 1,12- stereochemical control in a terphenyl tetracarboxamide

7.5 Hz, 1H), 8.58 (s, 1H), 8.65 (s, 1H). ¹³C-NMR (C₆D₆, 75,5 MHz) δ 15.8 (CH₃), 20.18 (CH₃), 20.28 (CH₃), 20.33 (CH₃), 20.36 (CH₃), 20.41 (CH₃), 20.49 (CH₃), 20.55 (CH₃), 20.69 (CH₃), 20.76 (CH₃), 20.86 (CH₃), 20.93 (CH₃), 22.3 (CH₃), 23.2 (CH₃), 36.5 (CH₃), 45.9 (CH), 46.0 (CH), 46.1 (CH), 46.4 (CH), 51.07 (CH), 51.13 (CH), 51.2 (CH), 51.8 (CH), 64.3 (CH), 82.6 (CH), 94.7 (CH), 127.2 (CH), 127.4 (CH), 127.5 (CH), 128.1 (CH), 128.9 (CH), 130.0 (CH), 130.3 (CH), 130.9 (CH), 131.7 (CH), 135.0 (Cq), 135.3 (Cq), 136.7 (Cq), 138.0 (Cq), 138.2 (Cq), 138.9 (Cq), 139.3 (Cq), 139.5 (Cq), 140.1 (Cq), 140.4 (Cq), 168.6 (Cq), 168.7 (Cq), 168.9 (Cq), 170.1 (Cq). IR (film) 1632 cm⁻¹. HRMS Calcd for C₅₇H₈₀N₅O₅: 914.6154. Found: 914.6152.
For *ent*-**8**, [α]_D²⁵ = +50 (c = 0.2).⁴

3-Formyl-*N,N,N',N',N'',N''',N''''*-Octaisopropyl-3'-[(4*S*,5*R*)-3,4-dimethyl-5-phenyl-oxazolidin-2-yl]-[1,1';4',1'']terphenyl-2,2',5',2''-tetracarboxamide **9**

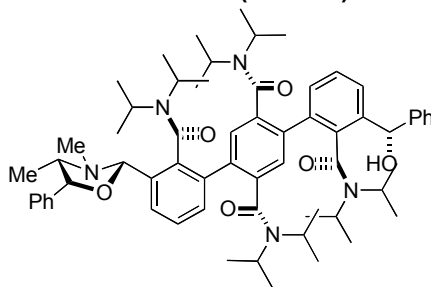


A solution of **8** (84 mg, 0.09 mmol) in dry THF (40 mL) was cooled to -78 °C under nitrogen and stirred for 5 min. *s*-Butyllithium (0.75 mL, 0.9 mmol, 1.2 M in hexanes) was added and the mixture was stirred for 1 h. Dry DMF (1 mL) was added and the mixture was stirred for 1h. The solution was allowed to warm to room temperature, partitioned between AcOEt and H₂O, and the aqueous phase was extracted with AcOEt. The organic extracts were dried, filtered and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography (Petrol to petrol:AcOEt 30%) to yield **9** (62 mg, 77%).

¹H-NMR (C₆D₆, 300 MHz) δ 0.61 (d, *J* = 6.6 Hz, 3H), 0.67 (d, *J* = 6.6 Hz, 3H), 0.71 (d, *J* = 6.6 Hz, 3H), 0.85 (d, *J* = 6.3 Hz, 3H), 0.93 (d, *J* = 6.6 Hz, 3H), 1.14 (d, *J* = 6.6 Hz, 3H), 1.16 (d, *J* = 6.6 Hz, 3H), 1.27 (d, *J* = 6.9 Hz, 3H), 1.29 (d, *J* = 6.9 Hz, 3H), 1.32-1.37 (m, 3H), 1.54 (s, 3H), 1.53-1.60 (m, 12H), 1.64 (d, *J* = 6.9 Hz, 3H), 1.76 (d, *J* = 6.6 Hz, 3H), 2.32 (s, 3H), 2.68 (dq, *J* = 8.4 and 6.6 Hz, 1H), 3.03 (septet, *J* = 6.9 Hz, 2H), 3.07 (septet, *J* = 6.9 Hz, 1H), 3.21 (septet, *J* = 6.9 Hz, 1H), 4.14 (septet, *J* = 6.3 Hz, 1H), 4.32 (septet, *J* = 6.3 Hz, 1H), 4.33 (septet, *J* = 6.3 Hz, 2H), 5.00 (d, *J* = 8.4 Hz, 1H), 5.26 (s, 1H), 6.95-7.12 (m, 3H), 7.19 (d, *J* = 7.8 Hz, 1H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.2 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.98 (dd, *J* = 7.5 and 1.2 Hz, 1H), 8.39 (dd, *J* = 7.5 and 1 Hz, 1H), 8.60 (s, 1H), 8.69 (s, 1H), 10.55 (s, 1H). ¹³C-NMR (C₆D₆, 75,5 MHz) δ 15.7 (CH₃), 20.14 (CH₃), 20.20 (CH₃), 20.26 (CH₃), 20.30 (CH₃), 20.38 (CH₃), 30.49 (CH₃), 20.52 (CH₃), 20.68 (CH₃), 20.70 (CH₃), 20.8 (CH₃), 22.7 (CH₃), 23.3 (CH₃), 36.5 (CH₃), 46.1 (CH), 46.2 (CH), 46.4 (CH), 46.7 (CH), 51.2 (CH), 51.3 (CH), 51.7 (CH), 51.8 (CH), 64.3 (CH), 82.5 (CH), 94.6 (CH), 127.4 (CH), 127.5 (CH), 127.8 (CH), 127.9 (CH), 128.6 (CH), 129.1 (CH), 130.1 (CH), 130.3 (CH), 131.8 (CH), 134.2 (Cq), 134.7 (Cq), 136.2 (Cq), 136.3 (CH), 136.8 (Cq), 137.0 (Cq), 138.9 (Cq), 139.1 (Cq), 139.4 (Cq), 139.9 (Cq), 140.3 (Cq), 141.4 (Cq), 167.1 (Cq), 168.5 (Cq), 168.6 (Cq), 170.0 (Cq), 190.3 (CH). IR (film) 1632 cm⁻¹. HRMS Calcd for C₅₈H₈₀N₅O₆: 942.6103. Found: 942.6125.
For *ent*-**9**, [α]_D²⁵ = +26 (c = 0.2).⁴

J. Clayden, L. Vallverdú, M. Helliwell:
Transmitting information along oligoparaphenylenes: 1,12- stereochemical control in a terphenyl tetracarboxamide

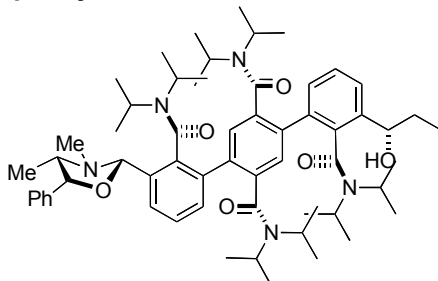
3-Hydroxybenzyl-*N,N,N',N',N'',N''',N''''*-octoisopropyl-3'-[(2*S*,4*S*,5*R*)-3,4-dimethyl-5-phenyloxazolidin-2-yl]-[1,1';4',1'']terphenyl-2,2',5',2''-tetracarboxamide **10 ($R^2 = \text{Ph}$)**



A solution of **9** (10 mg, 0.011 mmol) in THF (10 mL) was cooled to -78°C under nitrogen and stirred for 5 min. Phenylmagnesium bromide (0.2 mL, 0.18 mmol, 0.9M in THF) was slowly added dropwise. The solution was stirred at -78°C for 2 h, then allowed to gradually warm to room temperature, and quenched with saturated aqueous ammonium chloride solution (10 mL). The mixture was partitioned between AcOEt and H_2O , and the aqueous phase was extracted with AcOEt. The organic extracts were dried, filtered and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography (Petrol to petrol:AcOEt 20% + Et_3N 1%) to yield **10** (10 mg, 98%) as a single diastereoisomer (by NMR).

M.p. $82\text{--}84^\circ\text{C}$. $^1\text{H-NMR}$ (C_6D_6 , 300 MHz) δ 0.63 (d, $J = 6.6$ Hz, 3H), 0.67 (d, $J = 6.3$ Hz, 3H), 0.78 (d, $J = 6.6$ Hz, 3H), 0.77 (d, $J = 6.6$ Hz, 3H), 0.90 (d, $J = 6.3$ Hz, 3H), 0.93 (d, $J = 6.3$ Hz, 3H), 1.05 (d, $J = 6.6$ Hz, 3H), 1.14 (d, $J = 6.3$ Hz, 3H), 1.24 (d, $J = 6.9$ Hz, 3H), 1.28 (d, $J = 6.9$ Hz, 3H), 1.27-1.31 (m, 3H), 1.51 (d, $J = 6.6$ Hz, 3H), 1.56-1.58 (m, 3H), 1.58 (d, $J = 6.6$ Hz, 3H), 1.59 (d, $J = 6.6$ Hz, 3H), 1.68 (d, $J = 6.9$ Hz, 3H), 1.76 (d, $J = 6.6$ Hz, 3H), 2.31 (s, 3H), 2.67 (dq, $J = 8.4$ and 6.3 Hz, 1H), 2.98 (septet, $J = 6.9$ Hz, 1H), 3.02 (septet, $J = 6.9$ Hz, 1H), 3.10 (septet, $J = 6.9$ Hz, 1H), 3.23 (septet, $J = 6.9$ Hz, 1H), 4.26-4.32 (m, 1H), 4.29 (septet, $J = 6.3$ Hz, 1H), 4.30 (septet, $J = 6.6$ Hz, 1H), 4.45 (septet, $J = 6.9$ Hz, 1H), 4.99 (d, $J = 8.4$ Hz, 1H), 5.25 (s, 1H), 6.39 (s, 1H), 6.60 (dd, $J = 7.5$ and 1.2 Hz, 2H), 6.76 (ddd, $J = 7.5$, 1.2 and 0.9 Hz, 2H), 6.96-7.37 (m, 7H), 7.48 (d, $J = 7.2$ Hz, 1H), 7.50 (d, $J = 7.2$ Hz, 1H), 7.78 (d, $J = 7.2$ Hz, 2H), 8.41 (dd, $J = 7.8$ and 1.2 Hz, 1H), 8.46 (s, 1H), 8.69 (s, 1H). $^{13}\text{C-NMR}$ (C_6D_6 , 75.5 MHz) δ 15.8 (CH₃), 20.1 (CH₃), 20.2 (CH₃), 20.33 (CH₃), 20.38 (CH₃), 20.43 (CH₃), 20.48 (CH₃), 20.54 (CH₃), 20.6 (CH₃), 20.7 (CH₃), 20.8 (CH₃), 22.8 (CH₃), 23.2 (CH₃), 36.5 (CH₃), 46.1 (CH), 46.2 (CH), 46.4 (CH), 46.8 (CH), 51.2 (CH), 51.3 (CH), 51.9 (CH), 52.0 (CH), 64.3 (CH), 73.0 (CH), 82.6 (CH), 94.6 (CH), 115.5 (CH), 120.2 (CH), 129.1 (CH), 129.8 (CH), 130.0 (CH), 130.3 (CH), 130.6 (CH), 131.9 (CH), 136.8 (Cq), 137.8 (Cq), 137.9 (Cq), 138.6 (Cq), 139.4 (Cq), 139.8 (Cq), 140.4 (Cq), 142.0 (Cq), 144.2 (Cq), 144.8 (Cq), 168.6 (Cq), 168.7 (Cq), 169.0 (Cq), 171.4 (Cq). IR (film) 3330, 1632 cm^{-1} . HRMS Calcd for $\text{C}_{64}\text{H}_{86}\text{O}_6\text{N}_5$: 1020.6573. Found: 1020.6581. For *ent*-**10** ($R^2 = \text{Ph}$), $[\alpha]_{\text{D}}^{25} = +88$ ($c = 0.1$).⁴

3-(1-Hydroxypropyl)-*N,N,N',N',N'',N''',N''''*-octoisopropyl-3'-[(2*S*,4*S*,5*R*)-3,4-dimethyl-5-phenyloxazolidin-2-yl]-[1,1';4',1'']terphenyl-2,2',5',2''-tetracarboxamide **10 ($R^2 = \text{Et}$)**



A solution of **9** (93 mg, 0.099 mmol) in THF (10 mL) was cooled to -78°C under nitrogen and stirred for 5 min. Ethylmagnesium bromide (0.25 mL, 0.5 mmol, 2M in THF) was slowly added dropwise. The solution was stirred at -78°C for 2 h, then allowed to gradually warm to room temperature, and quenched with saturated aqueous ammonium chloride solution (10 mL). The mixture was partitioned between AcOEt and H_2O , and the aqueous phase was extracted with AcOEt. The organic extracts were dried, filtered and the solvent was removed under reduced pressure to yield **10** ($R^2 = \text{Et}$) (93 mg, 97%) as a single diastereoisomer (by NMR).

J. Clayden, L. Vallverdú, M. Helliwell:
Transmitting information along oligoparaphenylenes: 1,12- stereochemical control in a terphenyl tetracarboxamide

M.p. 162-164 °C. ¹H-NMR (C₆D₆, 300 MHz) δ 0.66 (d, *J* = 6.6 Hz, 3H), 0.69 (d, *J* = 6.6 Hz, 3H), 0.65-0.75 (m, 6H), 0.85-0.95 (m, 9H), 1.04-1.08 (m, 3H), 1.16 (d, *J* = 6.6 Hz, 3H), 1.16-1.19 (m, 3H), 1.30 (d, *J* = 6.9 Hz, 3H), 1.32 (d, *J* = 6.9 Hz, 3H), 1.34 (d, *J* = 6.9 Hz, 3H), 1.58 (d, *J* = 6.6 Hz, 3H), 1.59 (d, *J* = 6.6 Hz, 3H), 1.62 (d, *J* = 6.9 Hz, 3H), 1.67 (d, *J* = 6.6 Hz, 3H), 1.77 (d, *J* = 6.6 Hz, 3H), 1.90 (m, 1H), 2.21 (m, 1H), 2.32 (s, 3H), 2.69 (dq, *J* = 8.5 and 6.6 Hz, 1H), 3.03 (septet, *J* = 6.6 Hz, 2H), 3.13 (septet, *J* = 6.9 Hz, 1H), 3.24 (septet, *J* = 6.6 Hz, 1H), 4.22-4.37 (m, 4H), 4.92 (dd, *J* = 8.8 and 4.7 Hz, 1H), 5.01 (d, *J* = 8.5 Hz, 1H), 5.26 (s, 1H), 7.10-7.14 (m, 2H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.22 (d, *J* = 7.2 Hz, 1H), 7.33 (dd, *J* = 7.8 and 7.6 Hz, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.48-7.52 (m, 3H), 7.53 (d, *J* = 7.9 Hz, 1H), 8.39 (d, *J* = 7.9 Hz, 1H), 8.42 (s, 1H), 8.69 (s, 1H). IR (film) 3386, 1632 cm⁻¹. HRMS Calcd for C₆₀H₈₆O₆N₅: 972.6573. Found: 972.6568.
For *ent*-**10** (*R*² = Et), [α]_D²⁵ = +49.3 (*c* = 0.3).⁴

References:

1. J. Clayden, L. Vallverdú and M. Helliwell, *Org. Biomol. Chem.* 2006, **4**, 2106.
2. B. I. Alo, A. Kandil, P. A. Patil, M. J. Sharp, M. A. Siddiqui and V. Snieckus, *J. Org. Chem.* 1991, **56**, 3763
3. R. J. Perry, S. E. Tunney and B. D. Wilson, *Macromolecules* 1996, **29**, 1014
4. Optical rotation determined using a sample made from (1*S*,2*R*)-(+)-ephedrine. The compound as shown in the paper is *laevorotatory*.