Electronic Supplementary Material (ESI) for New Journal of Chemistry

Electronic supplementary information

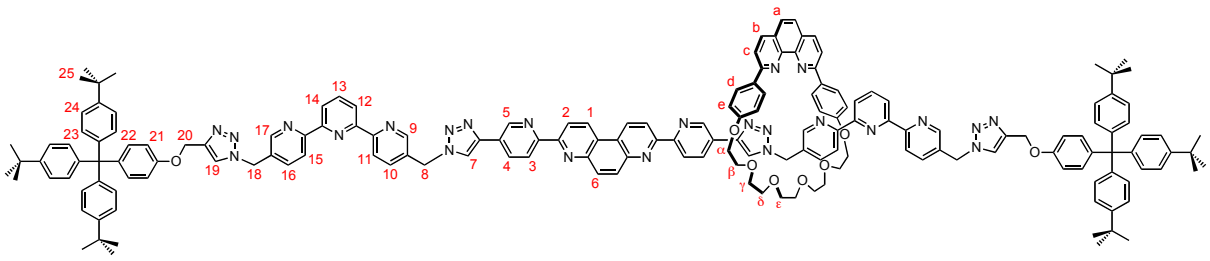
Synthesis of [2]-, [3]-, and [4]rotaxanes whose axis contains two bidentate and two tridentate chelates

Jean-Paul Collin,* Stéphanie Durot,* Jean-Pierre Sauvage,* and Yann Trolez

Laboratoire de Chimie Organo-Minérale, Institut de Chimie, LC3 UMR 7177 du CNRS, Université de Strasbourg, 4 rue Blaise Pascal, 67070 Strasbourg Cedex, France.

^{1}H NMR assignment of compounds 6, 7 and 8

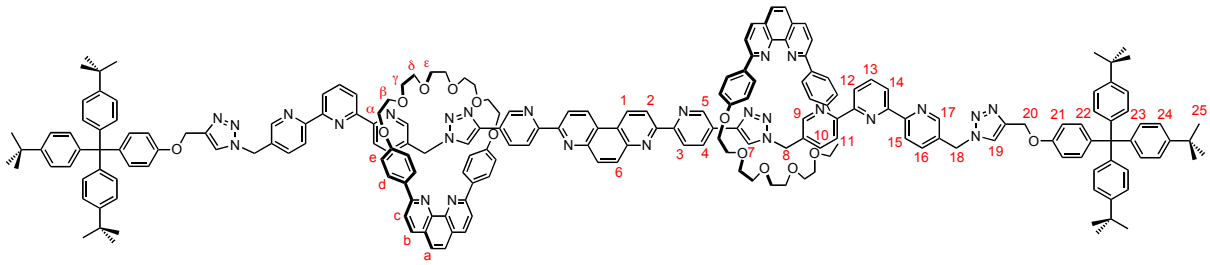
The spectrum was referenced to residual proton-solvent references. In the assignments, the chemical shift (in ppm) is given first, followed, in brackets, by the multiplicity of the signal (s: singlet, d: doublet, dd: doublet of doublet, t: triplet, m: multiplet), the value of the coupling constants in Hertz if applicable, the number of protons implied and finally the assignment.



6

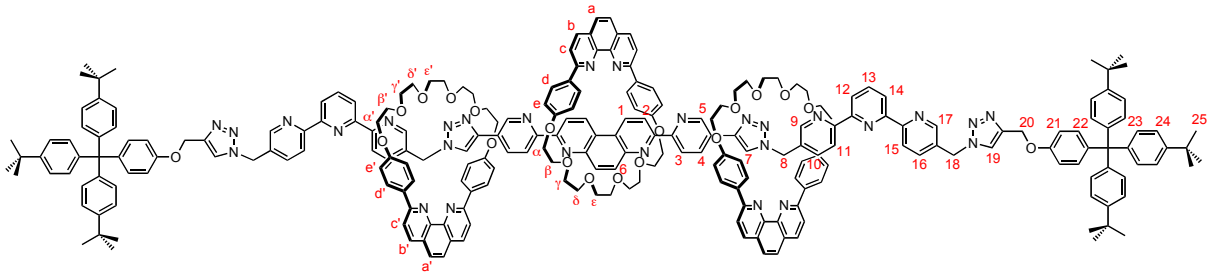
^{1}H-NMR (400 MHz, CD_2Cl_2, 25 °C) : δ = 9.20 (d, ^4J = 1.7 Hz, 2H, H-5), 9.06 (d, ^3J = 8.8 Hz, 2H, H-1), 8.79 (d, ^3J = 8.7 Hz, 2H, H-2), 8.75 (d, ^3J = 8.2 Hz, 2H, H-3), 8.66 (d, ^4J = 1.5 Hz, 2H, H-9), 8.56 (d, ^4J = 1.6 Hz, 2H, H-17), 8.46 (d, ^3J = 8.1 Hz, 2H, H-11), 8.41 (d, ^3J = 7.6 Hz, 2H, H-15), 8.40-8.37 (m, 6H, H-4, H-13, H-7), 8.29 (s, 2H, H-6), 8.17 (d, ^3J = 8.2 Hz, 2H, H-b), 7.95-7.90 (m, 8H, H-12, H-14, H-d), 7.85 (d, ^3J = 8.4 Hz, 2H, H-c), 7.84 (s, 2H, H-19), 7.72 (dd, ^3J = 8.2 Hz, ^4J = 2.0 Hz, 2H, H-10), 7.69 (s, 2H, H-a), 7.59 (dd, ^3J = 8.3 Hz, ^4J

= 2.0 Hz, H-16), 7.23 (d, ^3J = 8.6 Hz, 12H, H-24), 7.14 (d, ^3J = 9.4 Hz, 4H, H-22), 7.13 (d, ^3J = 8.7 Hz, 12H, H-23), 6.87 (d, ^3J = 9.0 Hz, 4H, H-21), 6.66 (d, ^3J = 8.6 Hz, 4H, H-e), 5.63 (s, 4H, H-8), 5.54 (s, 4H, H-18), 5.15 (s, 4H, H-20), 3.98 (t, ^3J = 5.8 Hz, 4H, H-α), 3.64 (s, 4H, H-ε), 3.63-3.55 (m, 12H, H-β, H-γ, H-δ), 1.27 (s, 54H, H-25) ppm.



7

^{1}H-NMR (300 MHz, $CDCl_3$, 25 °C) : δ = 9.22 (d, ^4J = 1.6 Hz, 2H, H-5), 9.05 (d, ^3J = 8.8 Hz, 2H, H-1), 8.74 (d, ^3J = 8.6 Hz, 2H, H-2), 8.70 (d, ^3J = 8.2 Hz, 2H, H-3), 8.70 (s, 2H, H-7), 8.60 (d, ^4J = 1.7 Hz, 2H, H-9), 8.44-8.39 (m, 4H, H-4, H-17), 8.39-8.28 (m, 8H, H-6, H-11, H-12, H-14), 8.22 (d, ^3J = 8.2 Hz, 2H, H-15), 8.10 (d, ^3J = 8.2 Hz, 4H, H-b), 8.02 (s, 2H, H-19), 7.91 (d, ^3J = 8.6 Hz, 4H, H-d), 7.86 (d, ^3J = 7.9 Hz, 2H, H-13), 7.79 (d, ^3J = 8.4 Hz, 4H, H-c), 7.66-7.62 (m, 6H, H-a, H-10), 7.33 (dd, ^3J = 8.2 Hz, ^4J = 2.0 Hz, 2H, H-16), 7.20 (d, ^3J = 8.6 Hz, 12H, H-24), 7.08 (d, ^3J = 8.8 Hz, 4H, H-22), 7.05 (d, ^3J = 8.6 Hz, 12H, H-23), 6.90 (d, ^3J = 8.9 Hz, 4H, H-21), 6.65 (d, ^3J = 8.8 Hz, 8H, H-e), 5.51 (s, 4H, H-8), 5.39 (s, 4H, H-18), 5.17 (s, 4H, H-20), 3.99 (m, 8H, H-α), 3.71-3.59 (m, 34H, H-β, H-γ, H-δ, H-ε), 1.27 (s, 54H, H-25) ppm.



8

^{1}H-NMR (400 MHz, CD_2Cl_2, 25 °C) : δ = 9.24 (s, 2H, H-5), 9.12 (d, ^3J = 8.7 Hz, 2H, H-1), 8.92 (s, 2H, H-7), 8.77 (d, ^3J = 8.7 Hz, 2H, H-2), 8.72 (d, ^3J = 8.4 Hz, 2H, H-3), 8.49 (s, 2H, H-9), 8.42 (m, 4H, H-4, H-13), 8.35-8.24 (m, 10H, H-6, H-12, H-14, H-17, H-11), 8.19 (d, ^3J = 8.0 Hz, 2H, H-15), 8.18 (s, 2H, H-19), 8.12 (d, ^3J = 8.4 Hz, 4H, H-b'), 8.07 (d, ^3J = 7.7 Hz,

2H, H-b), 7.94 (d, ^{3}J = 7.0 Hz, 8H, H-d'), 7-90-7.75 (m, 10H, H-d, H-c', H-c), 7.66 (s, 4H, H-a'), 7.57 (s, 2H, H-a), 7.53 (d, ^{3}J = 7.7 Hz, 2H, H-10), 7.33 (d, ^{3}J = 7.5 Hz, 2H, H-16), 7.22 (d, ^{3}J = 8.5 Hz, 12H, H-24), 7.12 (d, ^{3}J = 8.5 Hz, 12H, H-23), 7.10 (d, ^{3}J = 9.2 Hz, 4H, H-22), 6.91 (d, ^{3}J = 8.8 Hz, 4H, H-21), 6.64 (d, ^{3}J = 8.4 Hz, 8H, H-e'), 6.51 (d, ^{3}J = 7.8 Hz, 4H, H-e), 5.42 (s, 4H, H-8), 5.32 (s, 4H, H-18), 5.12 (s, 4H, H-20), 3.93 (m, 8H, H-α'), 3.85 (t, ^{3}J = 5.8 Hz, 4H, H-α), 3.80-3-50 (H-β, H-β', H-γ, H-γ', H-δ, H-δ', H-ε, H-ε'), 1.27 (s, 54H, H-25) ppm.