Supplementary Material (ESI) for New Journal of Chemistry

Electronic supplementary information

[3]rotaxanes and [3]pseudorotaxanes with a rigid two-bidentate chelate axles threaded through two coordinating rings

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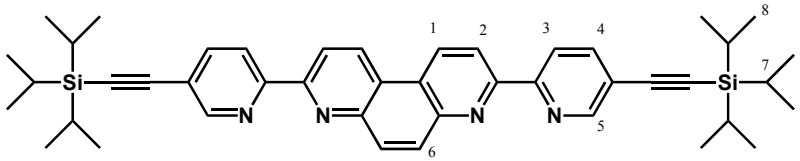
General methods

The spectra were 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, m: multiplet, q: quintuplet), the value of the coupling constants in Hertz if applicable, the number of protons implied and finally the assignment.
Mass spectra were obtained by using a Bruker MicroTOF spectrometer (ES-MS).

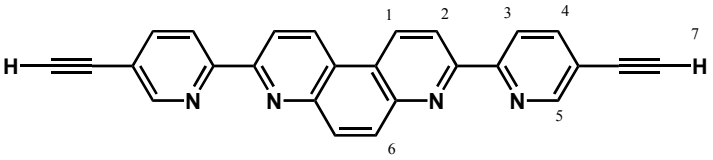
^{1}H NMR assignments of compounds 1^{2+}, 4, 5, 8^{2+}, 9^{2+}, 11, 12

TIPS axle (4) :



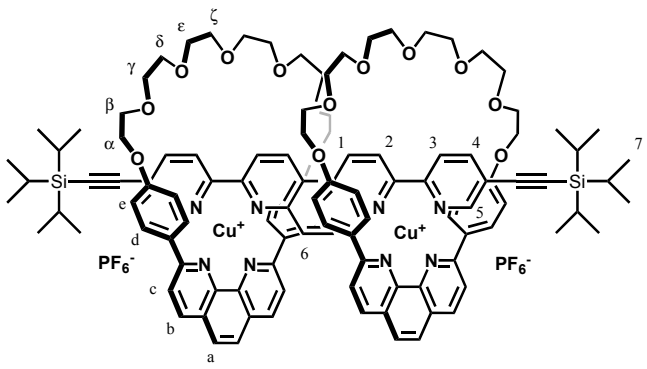
^{1}H-NMR (300 MHz, $CDCl_3$, 25°C) : δ = 9.05 (d, J^3 = 8.8 Hz, 2H, H-1), 8.82 (dd, J^4 = 1.3 Hz, J^5 = 0.7 Hz, 2H, H-5), 8.78 (d, J^3 = 8.8 Hz, 2H, H-2), 8.68 (dd, J^3 = 8.2 Hz, J^5 = 0.7, 2H, H-3), 8.33 (s, 2H, H-6), 7.95 (dd, J^3 = 8.2 Hz, J^4 = 2.2 Hz, 2H, H-4), 1.17 (s, 42H, H-7,8) ppm.

Acetylenic axle (5) :



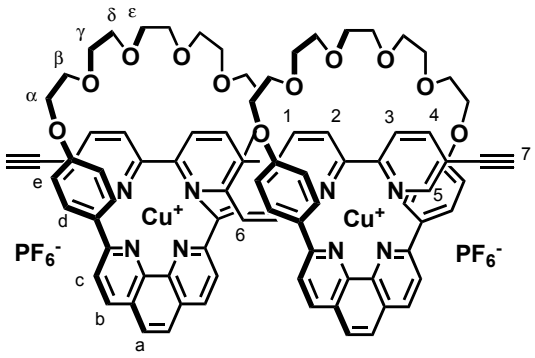
^{1}H-NMR (300 MHz, $CDCl_3$, 25°C) : δ = 9.07 (d, J^3 = 8.8 Hz, 2H, H-1), 8.85 (d, J^4 = 2.0 Hz, J^5 = 0.7 Hz, 2H, H-5), 8.78 (d, J^3 = 8.8 Hz, 2H, H-2), 8.72 (d, J^3 = 8.1 Hz, J^5 = 0.6 Hz, 2H, H-3), 8.33 (s, 2H, H-6), 7.99 (dd, J^3 = 8.1 Hz, J^4 = 2.1 Hz, 2H, H-4), 3.34 (s, 2H, H-7) ppm.

TIPS [3]pseudorotaxane ($[8^{2+}](PF_6^-)_2$) :



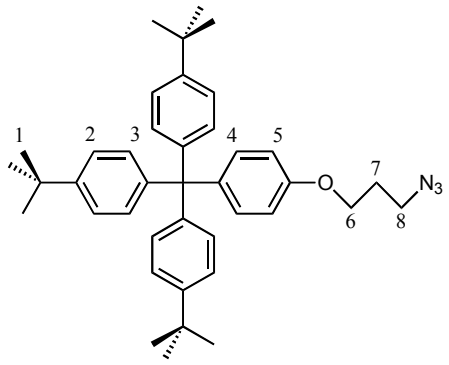
^{1}H-NMR (300 MHz, CD_2Cl_2, 25°C) : δ = 9.44 (d, J^3 = 8.8 Hz, 2H, H-1), 8.69 (d, J^3 = 8.8 Hz, 2H, H-2), 8.60 (d, J^3 = 8.3 Hz, 4H, H-b), 8.50 (d, J^3 = 8.3 Hz, 2H, H-3), 8.26 (dd, J^3 = 8.3 Hz, J^4 = 1.8 Hz, 2H, H-4), 8.08 (s, 4H, H-a), 7.98 (d, J^3 = 8.3 Hz, 4H, H-c), 7.94 (d, J^4 = 1.8 Hz, 2H, H-5), 7.30 (s, 2H, H-6), 7.24 (d, J^3 = 8.6 Hz, 8H, H-d), 6.11 (d, J^3 = 8.6 Hz, 8H, H-e), 4.00 – 3.70 (m, 48H, Hα, H-β, H-γ, H-δ, H-ε, H-ζ), 1.15 (s, 42H, H-7) ppm.

Acetylenic [3]pseudorotaxane ($[9^{2+}](PF_6^-)_2$) :



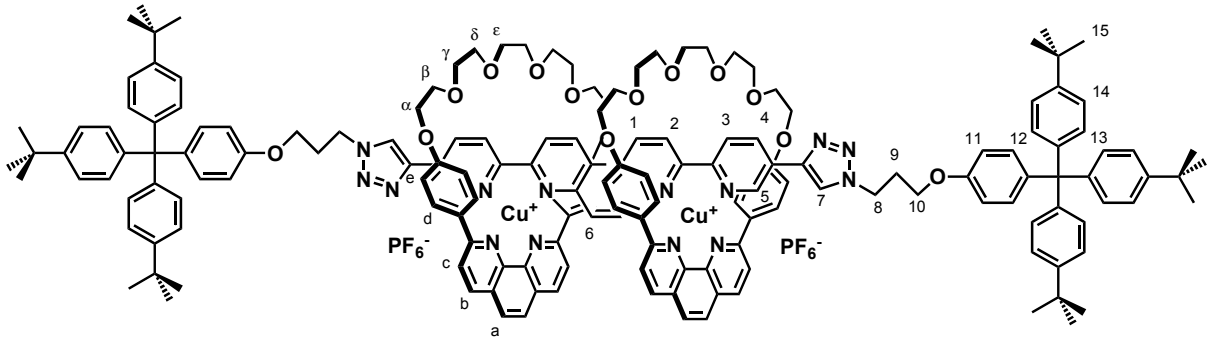
^{1}H-NMR (300 MHz, CD_2Cl_2, 25°C) : δ = 9.54 (d, J^3 = 8.8 Hz, 2H, H-1), 8.77 (d, J^3 = 8.8 Hz, 2H, H-2), 8.64 (d, J^3 = 8.4, 4H, H-b), 8.57 (d, J^3 = 8.3, 2H, H-3), 8.23 (dd, J^3 = 8.2 Hz, J^4 = 1.9 Hz, 2H, H-4), 8.13 (s, 4H, H-a), 8.06 (d, J^4 = 1.6 Hz, 2H, H-5), 8.03 (d, J^3 = 8.3 Hz, 4H, H-c), 7.38 (s, 2H, H-6), 7.22 (d, J^3 = 8.7 Hz, 8H, H-d), 6.12 (d, J^3 = 8.7 Hz, 8H, H-e), 4.00 – 3.75 (m, 40H, H-ạH-ḅH-gH-ḍH-e), 3.53 (s, 2H, H-7) ppm.

Azide stopper (11) :



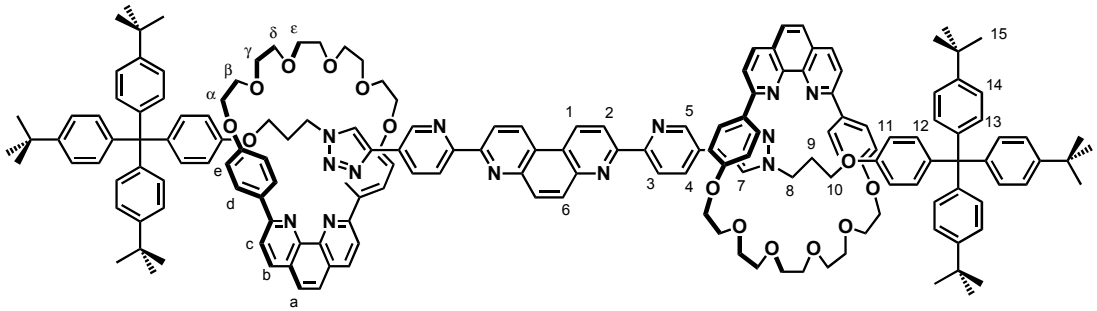
^{1}H-NMR (300 MHz, $CDCl_3$, 25°C) : δ = 7.27 (d, J^3 = 8.8 Hz, 6H, H-3), 7.16 (d, J^3 = 8.6 Hz, 6H, H-2), 7.16 (d, J^3 = 8.6 Hz, 2H, H-4), 6.79 (d, J^3 = 8.8 Hz, 2H, H-5), 4.02 (t, J^3 = 6.0 Hz, 2H, H-8), 3.50 (t, J^3 = 6.7 Hz, 2H, H-6), 2.03 (q, J^3 = 6.3 Hz, 2H, H-7), 1.31 (s, 27H, H-1) ppm.

[3]rotaxane ($1^{2+}(PF_6^-)_2$) :



^{1}H-NMR (300 MHz, CD_2Cl_2, 25°C) : δ = 9.49 (d, J^3 = 8.8 Hz, 2H, H-1), 8.72 (d, J^3 = 8.8 Hz, 2H, H-2), 8.58 (d, J^3 = 8.2 Hz, 4H, H-b), 8.57 (d, J^3 = 8.2 Hz, 2H, H-3), 8.50 (s, 2H, H-7), 8.47 (dd, J^3 = 8.3 Hz, J^4 = 1.9 Hz, 2H, H-4), 8.19 (s, 2H, H-5), 8.07 (s, 4H, H-a), 7.94 (d, J^3 = 8.1 Hz, 4H, H-c), 7.34 (s, 2H, H-6), 7.22 (d, J^3 = 8.6 Hz, 12H, H-14), 7.21 (d, J^3 = 8.9 Hz, 4H, H-12), 7.15 (d, J^3 = 7.9 Hz, 8H, H-d), 7.11 (d, J^3 = 8.8 Hz, 12H, H-13), 6.78 (d, J^3 = 8.9 Hz, 4H, H-11), 6.00 (d, J^3 = 7.9 Hz, 8H, H-e), 4.65 (t, J^3 = 6.8 Hz, 4H, H-8), 3.99 (t, J^3 = 5.4 Hz, 4H, H-10), 3.92 – 3.70 (m, 40H, H-a, H-b, H-g, H-d, H-e), 2.43 (q, J^3 = 6.1 Hz, 4H, H-9), 1.26 (s, 54H, H-15) ppm.

Demetalated [3]rotaxane (**12**) :



^{1}H-NMR (300 MHz, CD_2Cl_2, 25°C) : δ = 9.16 (d, J^3 = 8.9 Hz, 2H, H-1), 9.07 (d, J^4 = 1.4 Hz, 2H, H-5), 8.81 (s, 2H, H-7), 8.77 (d, J^3 = 8.6 Hz, 2H, H-2), 8.64 (d, J^3 = 8.2 Hz, 2H, H-3), 8.31 (s, 2H, H-6), 8.26 (d, J^3 = 8.4 Hz, 4H, H-b), 8.15 (d, J^3 = 8.2 Hz, 2H, H-4), 8.14 (d, J^3 = 8.7 Hz, 8H, H-d), 7.95 (d, J^3 = 8.4 Hz, 4H, H-c), 7.77 (s, 4H, H-a), 7.24 (d, J^3 = 8.6 Hz, 12H, H-14), 7.14 (d, J^3 = 8.7 Hz, 12H, H-13), 7.09 (d, J^3 = 8.8 Hz, 4H, H-12), 6.78 (d, J^3 = 9.0 Hz, 4H, H-11), 6.72 (d, J^3 = 8.7 Hz, 8H, H-e), 4.31 (t, J^3 = 7.1 Hz, 4H, H-8), 3.92-3.85 (m, 12H, H-10, H-a), 3.64 (s, 8H, H-e), 3.55-3.45 (m, 24H, H-b, H-g, H-d), 2.21 (q, J^3 = 6.4 Hz, 4H, H-9), 1.29 (s, 54H, H-15) ppm.