

Singular Supramolecular Self-assembly of Bis-Cyclodextrinyl-Bis-Lariat Hosts with Bis-Aryl-Sulfonates as Guests

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Supporting Information

1. General procedures

2. Characterization of the products

3. NMR Spectra

1. General procedures

Chemicals and solvents (reagent grade or better) were purchase from Sigma-Aldrich. Anhydrous solvents were dried by usual procedures and stored over 4Å molecular sieves under Ar before use. Chromatography was carried out using Grace silicagel. ¹H and ¹³C-NMR spectra were recorded on a Bruker DRX 400 FT-NMR spectrometer at 400MHz for ¹H and 100 MHz for ¹³C. Chemicals shifts δ were reported in parts per million (ppm), calibrated to the residual solvent peak set, with coupling constants reported in Hertz (Hz). High Resolution Electrospray (HR-ESIMS) and (MALDI-TOF) Mass spectra were recorded on a Bruker MicroQ-TOF and a Bruker Daltonics Reflex IV spectrometer respectively. FT-IR spectra were obtained from KBr pellets on a perkin Elmer Spectrum 1000 photometer.

Zn^{II}, Cu^{II} and Ca^{II} bis-tosylates, Zn^{II} and Cu^{II} bis-sulfanilate salts were obtained by a slightly modified method of the literature.⁸ Synthesis of the chiral bis-β-cyclodextrinyl-diazacrown receptor see.⁶ Synthesis of the achiral bis-β-cyclodextrinyl-diazacrown receptor see.⁷

General procedure for pseudo-rotaxane preparation

1 equiv. of metal bis-tosylate or bis-sulfanilate salt is added to 1 equiv. of dimer **1**, **2** or **3** in water (3mL) at r.t. The solution is stirred overnight at r. t., evaporated to dryness and then lyophilised.

2. Characterization of the products

[bis-triazolo-peracetylated-β-cyclodextrinyl]-diazacrown **6**

To a stirred solution of *N,N'*-bis-propargyl-diazacrown **5** (17 mg, 1,47 mmol, 338.44 g.mol⁻¹) in CH₂Cl₂ at r.t. are added mono-azido peracetylated β-cyclodextrin (100 mg, 2000.80 g.mol⁻¹, 2.1 equiv.), Na-ascorbate (4 mg, 0.4 equiv.) and CuSO₄·5 H₂O (2.5mg, 0.2 equiv.) under argon. The yellow pale mixture is stirred overnight and evaporated. The crude material is purified by chromatography on silicagel with CH₂Cl₂/MeOH gradient as eluent. The final product is obtained as a white powder in 65% yield. ¹H NMR (400MHz, CDCl₃, 298K): δ= 8.01 (s, 2H; Triazol H), 5.47 (s, 2H; H₁), 5.30-5.40 (m, 7H ; H₂,

H₃, H₄, H₅, H₆, H₆), 3.90-3.75 (m; CH₂ crown), 3.70-3.40 (m; H₄, CH₂ crown), 2.70 (bs, 2H; CH₂ triazol), 2.10-1.90 (m, 80H; CH₃-CO). ¹³C NMR (100MHz, CDCl₃, 298K): δ=170.7-170.6-170.5-169.3 (CH₃CO), 144.1 (CH₂-C=C-N triazol), 126.2 (CH₂ triazol) 97 (C1), 77 (C4), 70.3-70.6-69.6-69.5-69.3 (C2, C3, C5), 68 (CH₂ crown), 62.8-62.6-62.4-62.2 (C6), 53.3 (N-CH₂-C=C triazol), 50 (CH₂ crown), 48.9 (C6'), 29.7 (CH₂ crown), 20.7 (CH₃-CO).

[bis-triazolo-β-cyclodextrinyl]-diazacrown **3**

NH₃ is bubbling during 3 hours through a solution of **6** (0.120mg, 0.0278 mmol, 4339.9 g.mol⁻¹) in anhyd. MeOH. The solution is stirred into the MeOH /NH₃ saturated solution during 2 or 3 days. The mixture is then evaporated to dryness, lyophilised to give **3** in a quantitative yield as flocculent white solid. ¹H NMR (400MHz, D₂O, 298K): δ=7.93 (s, 2H, TriazolH), 5.10 (s, 2H; CH₂ crown), 5.00 (s, 14H; H₁), 4.98 (b s; CH₂ crown), 4.65-4.55 (m, 2H; CH₂ crown), 4.00-3.35 (m, 84H; H₂, H₃, H₄, H₅, H₆, H₆), 3.20-3.10 (m; CH₂ crown), 3.10-3.00 (m; CH₂ crown), 2.85-2.70 (m; CH₂ crown), 2.71 (s, 4H; CH₂ triazol). ¹³C NMR (100MHz, D₂O, 298K): δ=126.9 (CH₂ triazol), 102.1 (C1), 81.1 (C4), 73.1-72.1-71.9 (C2, C3, C5), 69.7 (C6), 60.1 (C6'), 52.3 (CH₂ triazol), 51 (CH₂ crown), 47.6 (CH₂ triazol), 44.7 (CH₂ crown), 25.1 (CH₂ crown). ESIMS: *m/z*: 1329.49 [M/2+1H⁺].

Pseudo-[2]rotaxane **7**

[bis-β-cyclodextrinyl]-diazacrown dimer **1** (50mg, 0,019 mmol, M = 2582,33 g.mol⁻¹). Zn^{II} *p*-tosylate (7,91 mg, 1,94.10⁻² mmol, M=407,78 g.mol⁻¹). **7** was obtained quantitatively as a pale white flocculent solid. IR (KBr): ν =3300 (OH), 2950 (CH-), 2500 (CH arom.), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ=7.60 (d, ³J (H,H)=8Hz, 4H; Har), 7.25 (d, ³J (H,H)=8Hz, 4H; Har), 5.30 (s, 4H; H₁), 5.14 (d, ³J (H,H) = 4Hz, 1H; CH₂ crown), 4.94 (bs, 7H; H₁), 4.57-4.52 (m, 1H; CH₂ crown), 4.05-3.91 (m, 4H; CH₂ crown), 3.90-3.81 (m, 6H; CH₂ crown), 3.78-3.41 (m, 84H; H₂, H₃, H₄, H₅, H₆, H₆), 3.41-3.10 (m, 8H; CH₂ crown). ¹³C NMR (100MHz, D₂O, 298K): δ=160.6 (C=O; urea), 142.0 (q), 140.0 (q), 129.1 (CH arom.), 125.3 (CH arom.), 101.9 (C1), 99.5 (C1), 95.8 (CH₂ crown), 92.0 (CH₂ crown), 83.3 (CH₂ crown), 76.6 (CH₂ crown), 75.9-73.2-71.8 (C2, C3, C5), 69.6 (C6) 65.0 (CH₂ crown), 62.5 (CH₂ crown), 60.4 (C6'), 48.6 (CH₂ crown), 44.6 (CH₂ crown), 34.5 (CH₂ crown), 20.5 (CH₃Ph). Maldi-TOF [C₁₁₂H₁₇₈N₄O₈₀S₂Zn: 2986.87u.m.a.], 2582.55 [M- Zn(OTs)₂]

Pseudo-[2]rotaxane 8

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **1** (50mg, 0,019 mmol, M = 2582,33 g.mol⁻¹). Cu^{II} *p*-tosylate (7,87mg, 1,93.10⁻² mmol, M=405,93 g.mol⁻¹). **8** was obtained quantitatively as a pale green flocculent solid. IR (KBr): ν = 3300 (OH) 2950 (CH-) 2500 (CH arom.), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ =7.60 (d, ³J(H,H)=8Hz, 4H; Har), 7.24 (d, ³J(H,H)=8Hz, 4H; Har), 5.30 (bs, 4H; H1), 5.13 (s, 1H; CH₂-crown), 4.94 (s, 7H; H₁), 4.57 (m, 2H; CH₂ crown), 4.05-3.38 (m, 92H; H₂, H₃, H₄, H₅, H₆, CH₂ crown), 3.38-3.09 (m, 8H; CH₂ crown), 2.26 (s, 6H; CH₃-Ph). ¹³C NMR (100MHz, D₂O, 298K): δ =160.7 (C=O urea), 142.0 (q), 140.12 (q), 129.1 (CH arom.), 125.3 (CH arom.), 101.9 (C1), 99.5 (C1), 95.8 (CH₂ crown), 95.7 (CH₂ crown), 91.8 (CH₂ crown), 83.3 (CH₂ crown), 80.9 (C4), 76.8 (C2), 76.1 (CH₂ crown), 74.5-74.1-73.9 (CH₂ crown), 73.3 (C3), 72.8-72.7 (CH₂ crown), 71.8 (C5), 69.6 (C6), 67.2 (CH₂ crown), 60.4 (C6'), 48.6-47.1, 44.7 (CH₂ crown), 20.5 (CH₃) Maldi-TOF [C₁₁₂H₁₇₈CuN₄O₈₀S₂: 2985.87 u.m.a.]: 2645.908 [M-Cu(OTs)₂+Cu], 2582.897 [M-Cu(OTs)₂].

Pseudo-[2]rotaxane 9

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **1** (50mg, 0,019 mmol, M = 2582,33 g.mol⁻¹). Ca^{II} *p*-tosylate (7,42 mg, 1,94.10⁻² mmol, M=382,47 g.mol⁻¹). **9** was obtained quantitatively as a pale white flocculent solid. ¹H NMR (400MHz, D₂O, 298K) δ =7.62 (d, ³J(H,H)=8Hz, 4H; Har), 7.29 (d, ³J(H,H)=8Hz, 4H; Har), 5.33 (s, 4H, CH₂ crown), 5.17 (d, ³J(H,H)=4Hz, 1H; CH₂ crown), 4.97 (bs, 7 H; H1), 4.58-4.55 (m, 1H; CH₂ crown), 4.18-3.92 (m, 4H; CH₂ crown), 3.84-3.45 (m, 84H; H₂, H₃, H₄, H₅, H₆, H₆), 3.44-3.13 (m, 8H; CH₂ crown). ¹³C NMR (100MHz, D₂O, 298K): δ =160.6 (C=O urea), 142.0 (q), 140.0 (q), 129.1(CH arom.), 125.3 (CH arom.), 101.9 (C1), 99.5 (CH₂ crown), 95.8 (CH₂ crown), 92.0 (CH₂ crown), 83.3 (CH₂ crown), 76.6 (CH₂ crown), 75.9-73.2-71.8 (C2, C3, C5), 69.6 (C6) 65.0 (CH₂ crown), 62.5 (CH₂ crown), 60.4 (C6'), 48.6 (CH₂ crown), 44.6 (CH₂ crown), 34.5 (CH₂ crown), 20.5 (CH₃). Maldi-TOF [C₁₁₂H₁₇₈CaN₄O₈₀S₂: 2962.91u.m.a.]: 2582.55 [M-Ca(OTs)₂].

Pseudo-rotaxane 10

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **2** (50mg, 0,019 mmol, M = 2670.44 g.mol⁻¹). Zn^{II} *p*-tosylate (7.62 mg, 1,86.10⁻² mmol, M=407.78 g.mol⁻¹). **10** was obtained quantitatively as a white flocculent solid. IR (KBr): ν = 3300 (OH), 2950 (CH-), 2500 (CHar), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ =7.61 (d, ³J(H,H)=8Hz, 4H; Har), 7.22 (d, ³J(H,H)=8Hz, 4H; Har), 4.95 (s, 14H; H1), 3.90-3.23 (m, 108H; H₂, H₃, H₄, H₅, H₆, H₆, CH₂crown), 2.28 (s, 6H, CH₃). ¹³C NMR (100MHz, D₂O, 298K): δ =160.7 (C=O urea), 141.8 (q), 140.2 (q), 128.9 (CH arom.), 125.4 (CH arom.), 101.9 (C1), 80.8 (C4), 73.0-72.1-72.0-71.9-71.8 (C2, C3, C4, C5) (C2), 70.3 (C6), 60.0 (C6'), 53.0 (CH₂ crown), 41.6 (CH₂ crown), 20.2 (CH₃). Maldi-TOF [C₁₁₆H₁₈₆N₄O₈₂S₂Zn: 3074.9 u.m.a.]: 2671.078 [M- Zn(OTs)₂+H⁺]

Pseudo-[2]rotaxane 11

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **2** (50mg, 0,019 mmol, M = 2670.44 g.mol⁻¹). Cu^{II} *p*-tosylate (7,59 mg, 1,87.10⁻² mmol, M=405,93 g.mol⁻¹). **11** was obtained quantitatively as a pale green flocculent solid. IR (KBr): ν =3300 (OH), 2950 (CH-), 2500 (CHar), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K) δ =7.61 (d, ³J(H,H) = 8Hz, 4H; Har), 7.22 (d, ³J(H,H) = 8Hz, 4H; Har), 4.95 (s, 14H; H1), 3.90-3.23 (m, 108H; H₂, H₃, H₄, H₅, H₆, H₆, CH₂ crown), 2.28 (s, 6H, CH₃). ¹³C NMR (100MHz, D₂O, 298K): δ =160.7 (C=O urea), 141.2 (q), 139.8 (q), 128.4 (CH arom.), 124.9 (CH arom.), 101.4 (C1), 80.4 (C4), 72.6, 72.4, 71.6, 71.5, (C2, C3, C4, C5), 69.8 (C6), 59.5 (C6'), 52.5 (CH₂ crown), 40.7 (CH₂ crown), 20.2 (CH₃). Maldi-TOF [C₁₁₆H₁₈₆CuN₄O₈₂S₂: 3073.92 u.m.a.]:

2670.868 [M-Cu(OTs)₂], 2733.836 [M-Cu(OTs)₂+Cu]; ESIMS: *m/z* : 1336.4961[M/2+2H⁺],1422.5129[M-Cu(OTs)₂+Cu+OTs], 1539.5081 [M+3H⁺]

Pseudo-[2]rotaxane 12

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **2** (25mg, 0,0093 mmol, M = 2670.44 g.mol⁻¹). Ca^{II}-*p*-tosylate (3.58 mg, 8,78.10⁻³ mmol, M=407.78 g.mol⁻¹). **12** was obtained quantitatively as an off-white flocculent solid. IR (KBr): ν = 3300 (OH), 2950 (CH-), 2500 (CHar), 1637 (C=O urea), cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ =7.69 (d, ³J(H,H) = 8Hz, 4H; Har), 7.26 (d, ³J(H,H) = 8Hz, 4H; Har), 5.00 (s, 14H; H1), 3.99-3.28 (m, 108H H₂, H₃, H₄, H₅, H₆, H₆, CH₂crown), 2.33 (s, 6H; CH₃). ¹³C NMR (100MHz, D₂O, 298K): δ = 160.7 (C=O urea), 141.8 (q), 140.2 (q), 128.9 (q), 125.4 (q), 101.9 (C1), 80.8 (C4), 73.0, 72.1, 72.0, 71.9, 71.8 (C2, C3, C4, C5) (C2), 70.3 (C6), 60.0 (C6'), 53.0 (CH₂ crown), 41.6 (CH₂ crown), 20.2 (CH₃). Maldi-TOF [C₁₁₆H₁₈₆CaN₄O₈₂S₂: 3050.96 u.m.a.]: 2670.40 [M- Ca(OTs)₂]

Pseudo-[2]rotaxane 13

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **1** (50mg, 0,019 mmol, M = 2582,33 g.mol⁻¹). Zn^{II} bis-sulfanilate (8.6 mg, 1,93.10⁻² mmol, M=445.8 g.mol⁻¹) **13** was obtained quantitatively as a white solid. IR (KBr): ν = 3300 (OH), 2950 (CH-), 2500 (CHar), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ =7.51 (d, ³J (H,H) = 8Hz, 4H; Har), 6.78 (d, ³J (H,H) = 8Hz, 4H; Har), 5.32 (s, 2H; H1), 5.15 (d, ³J (H,H) = 4Hz, 1H; CH₂ crown), 4.98 (bs, 12 H; H1), 4.57 (d, ³J (H,H) = 8Hz, 1H; CH₂ crown), 4.51-3.92 (m, 4H; CH₂ crown), 3.91-3.39 (m, 86H; H₂, H₃, H₄, H₅, H₆, H₆, CH₂ crown), 3.40-3.11 (m, 5H, CH₂ crown). ¹³C NMR (100MHz, D₂O, 298K): δ =160.6 (C=O urea), 142.0 (q), 140.0 (q), 129.1(CH arom.), 125.3 (CH arom.), 101.9 (C1), 99.5 (C1), 95.8 (CH₂ crown), 92.0 (CH₂ crown), 83.3 (CH₂ crown), 76.6 (CH₂ crown), 75.9, 73.2, 71.8 (C2, C3, C5), 69.6 (C6) 65.0 (CH₂ crown), 62.5 (CH₂ crown), 60.4 (C6'), 48.6 (CH₂ crown), 44.6 (CH₂ crown), 34.5 (CH₂ crown). Maldi-TOF [C₁₁₀H₁₇₆N₆O₈₀S₂Zn: 2988.86u.m.a.]: 2583.048 [M- Zn(NH₂PhSO₃)₂+H⁺]

Pseudo-[2]rotaxane 14

[bis- β -cyclodextrinyl]-diaz-[18-6]-crown dimer **1** (50mg, 0,019 mmol, M = 2582,33 g.mol⁻¹). Ca^{II} bis-sulfanilate (7.65 mg, 1,94.10⁻² mmol, M=395 g.mol⁻¹). **14** was obtained quantitatively as a white solid. IR (KBr): ν =3300 (OH), 2950 (CH-), 2500 (CH arom.), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ =7.51 (d, ³J (H,H) = 8Hz, 4H; CH arom.), 6.78 (d, ³J(H,H) = 8Hz, 4H; Har), 5.32 (s, 2H; H1), 5.15 (d, ³J(H,H) = 4Hz, 1H; CH₂ crown), 4.98 (bs, 12 H; H1), 4.57 (d, ³J (H,H) = 8Hz, 1H; CH₂ crown), 4.51-3.92 (m, 4H; CH₂ crown), 3.91-3.39 (m, 86H; H₂, H₃, H₄, H₅, H₆, H₆, CH₂ crown), 3.40-3.11 (m, 5H; CH₂ crown). ¹³C NMR (100MHz, D₂O, 298K) δ =160.6 (CO urea), 142.0 (q) 140.0 (q), 129.1(CH arom.), 125.3 (CH arom.), 101.9 (C1), 99.5 (C1), 95.8 (CH₂ crown), 92.0 (CH₂ crown), 83.3 (CH₂ crown), 76.6 (CH₂ crown), 75.9, 73.2, 71.8 (C2, C3, C5), 69.6 (C6) 65.0 (CH₂ crown), 62.5 (CH₂ crown), 60.4 (C6'), 48.6 (CH₂ crown), 44.6 (CH₂ crown), 34.5 (CH₂ crown). Maldi-TOF [C₁₁₀H₁₇₆CaN₆O₈₀S₂: 2964.90 u.m.a.]: 2583.119[M- Ca(NH₂PhSO₃)₂+H⁺]

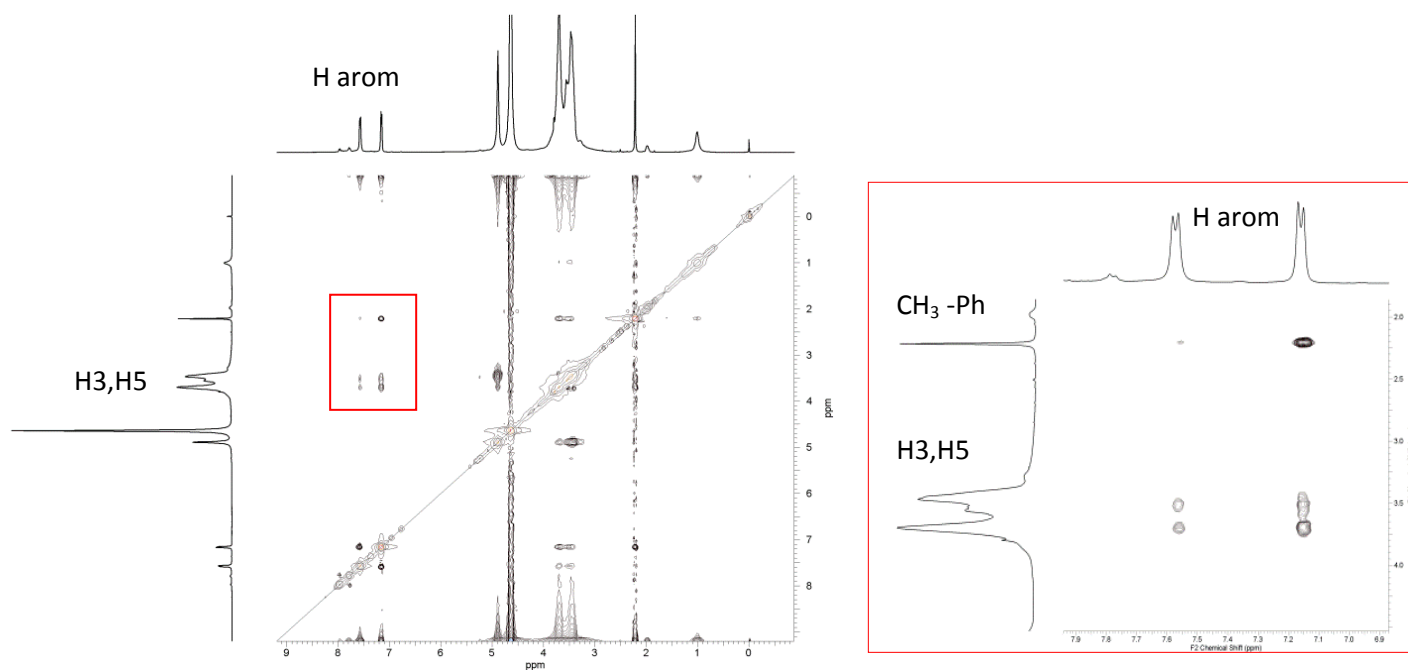
Pseudo-rotaxane 15

Bis-triazolo- β -cyclodextrinyl-diaz-[18-6]-crown dimer **3** (20mg, 0,0075 mmol, M = 2658.44 g.mol⁻¹). Zn^{II} bis-sulfanilate (3.35 mg, 7,51.10⁻³ mmol, M=445.8 g.mol⁻¹) in water, was obtained quantitatively as a white solid. IR (KBr): ν =3300 (OH) 2950 (CH-) 2500 (CH arom.), 1637 (C=O urea) cm⁻¹. ¹H NMR (400MHz, D₂O, 298K): δ =8.13 (s, 2H; TriazolH), 7.51 (d, ³J (H,H)=8Hz, 4H; Har), 6.77 (d, ³J (H,H)=8Hz, 4H; Har), 5.09 (s, 2H; CH₂ crown), 4.99 (s,14H; H₁), 4.92 (b s, 3; CH₂ crown), 4.61 (m, 2H; CH₂ crown), 4.10 (m; CH₂ crown), 3.88-3.38 (m, 84H;

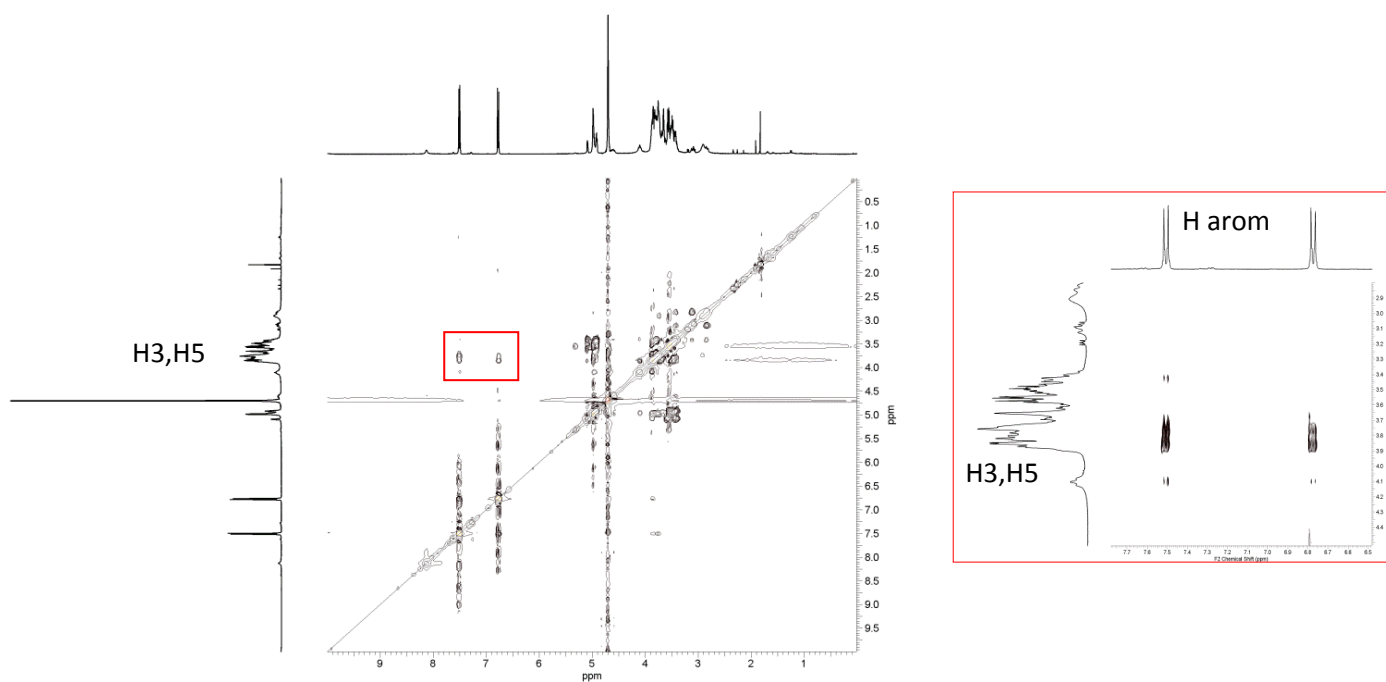
H₂, H₃, H₄, H₅, H₆, H₆'), 3.14-3.08 (m; CH₂ crown), 3.10-3.00 (m; CH₂ crown), 2.91-2.82 (m; CH₂ crown), 2.90 (s, 4H; CH₂ triazol). ¹³C NMR (100MHz, D₂O, 298K): δ=132.6 (CH arom.), 126.9 (CH₂ triazol), 124.8 (CH arom.), 101.8 (C1), 81.1 (C4), 73.1-72.1-71.8 (C2, C3, C5), 69.6 (C6), 60.2 (C6'), 52.4 (CH₂ triazol), 51.2 (CH₂ crown), 42.9 (CH₂ triazol), 44.7 (CH₂ crown), 22.1 (CH₂ crown). Maldi-TOF [C₁₁₄H₁₈₀N₁₀O₇₈S₂Zn: 3064.92 u.m.a.]: 1330.99 [M- Zn(NH₂PhSO₃)₂+2H⁺]

3. NMR Spectra

Pseudo-[2]rotaxane **11** 2D NMR (ROESY)

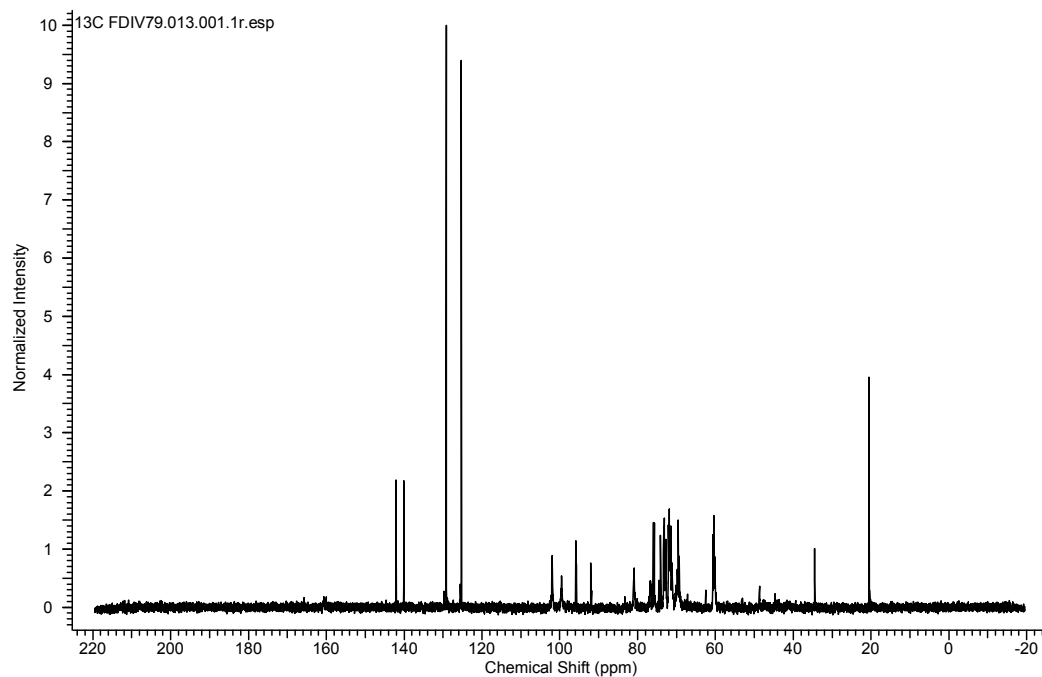


Pseudo-[2]rotaxane **15** 2D NMR (ROESY)

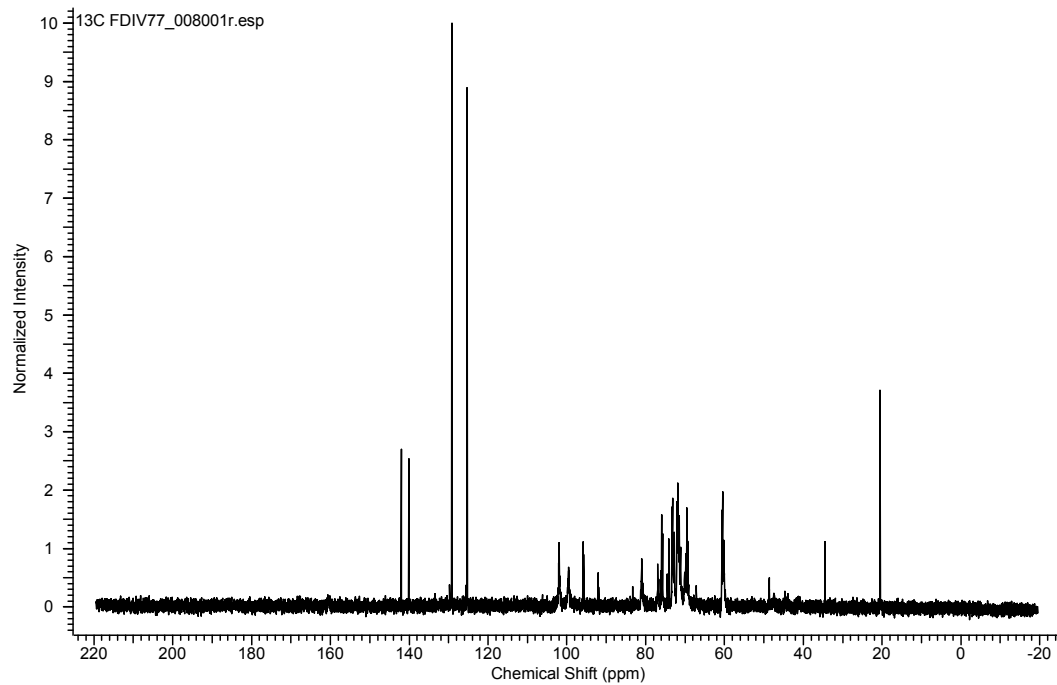


¹³C NMR SPECTRA

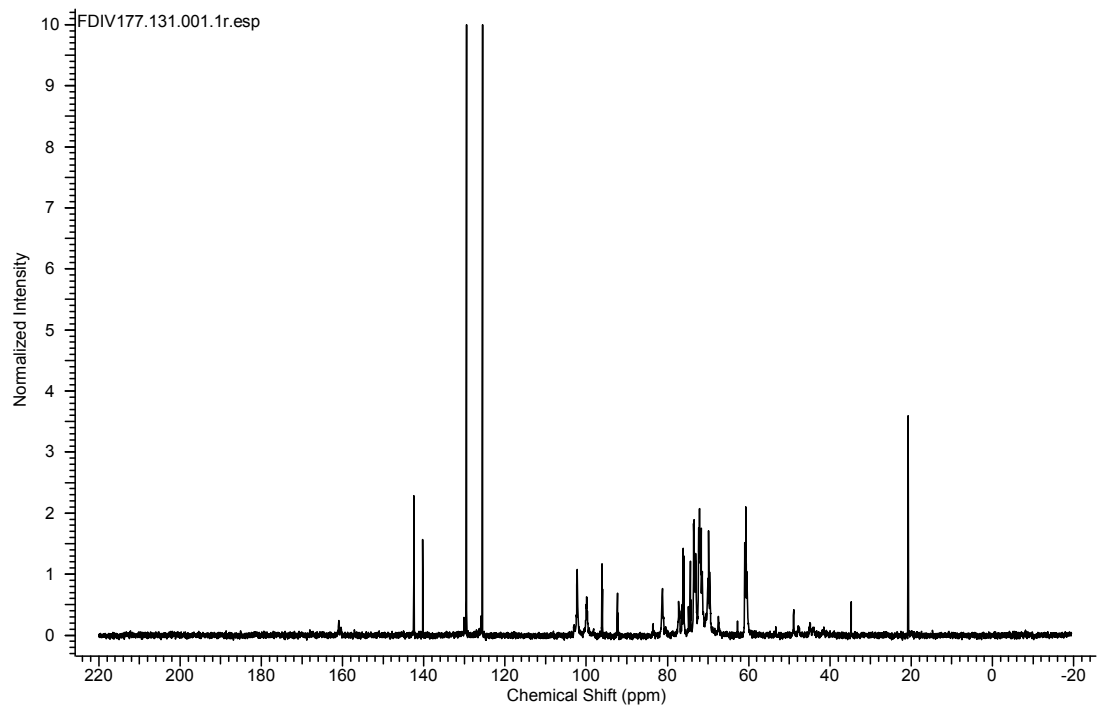
Pseudorotaxane 7 :



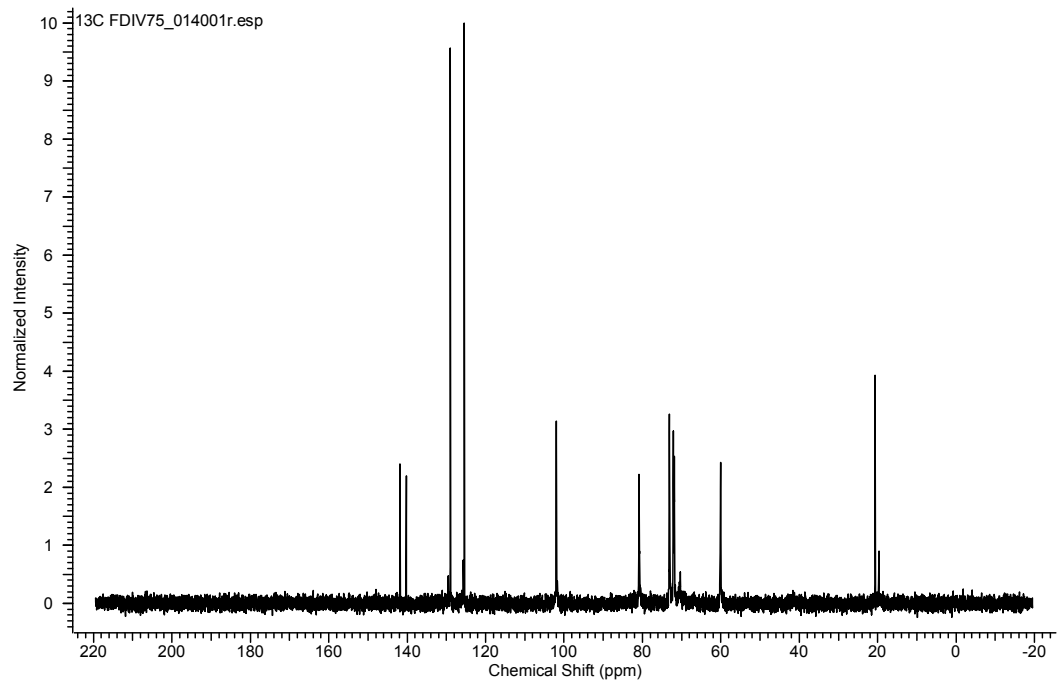
Pseudorotaxane 8



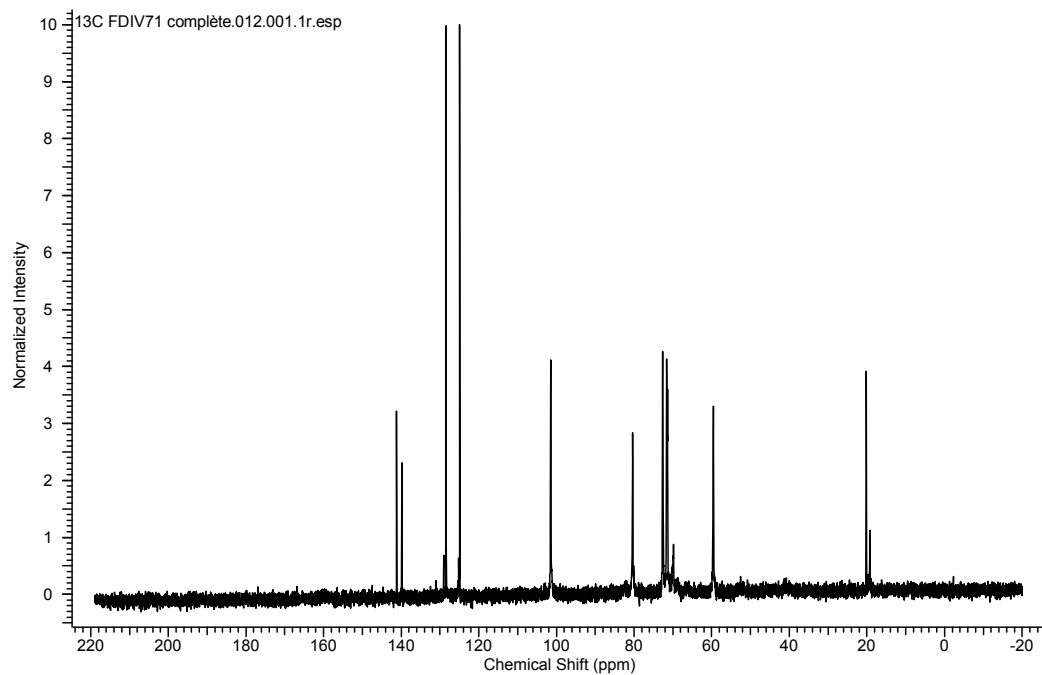
Pseudorotaxane 9 :



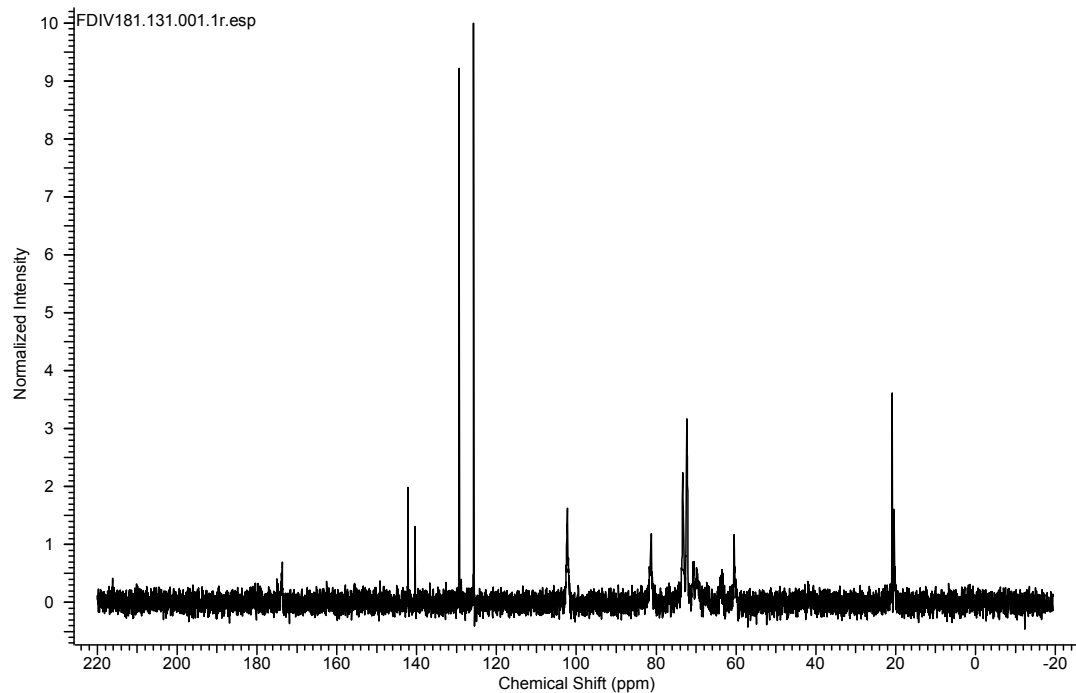
Pseudorotaxane 10:



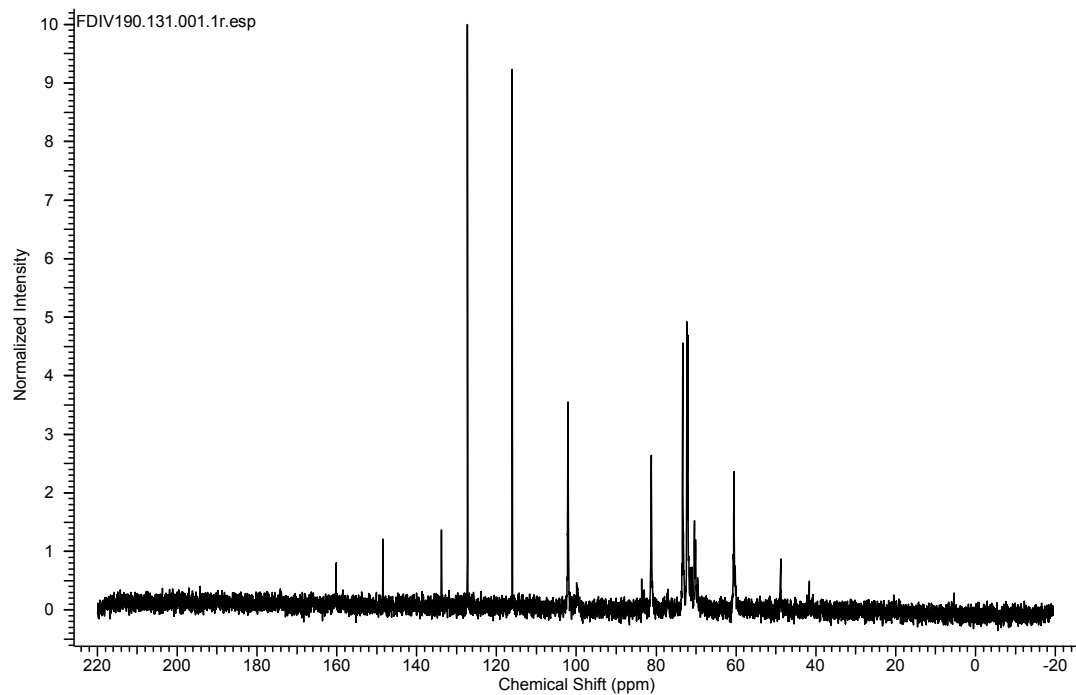
Pseudorotaxane 11 :



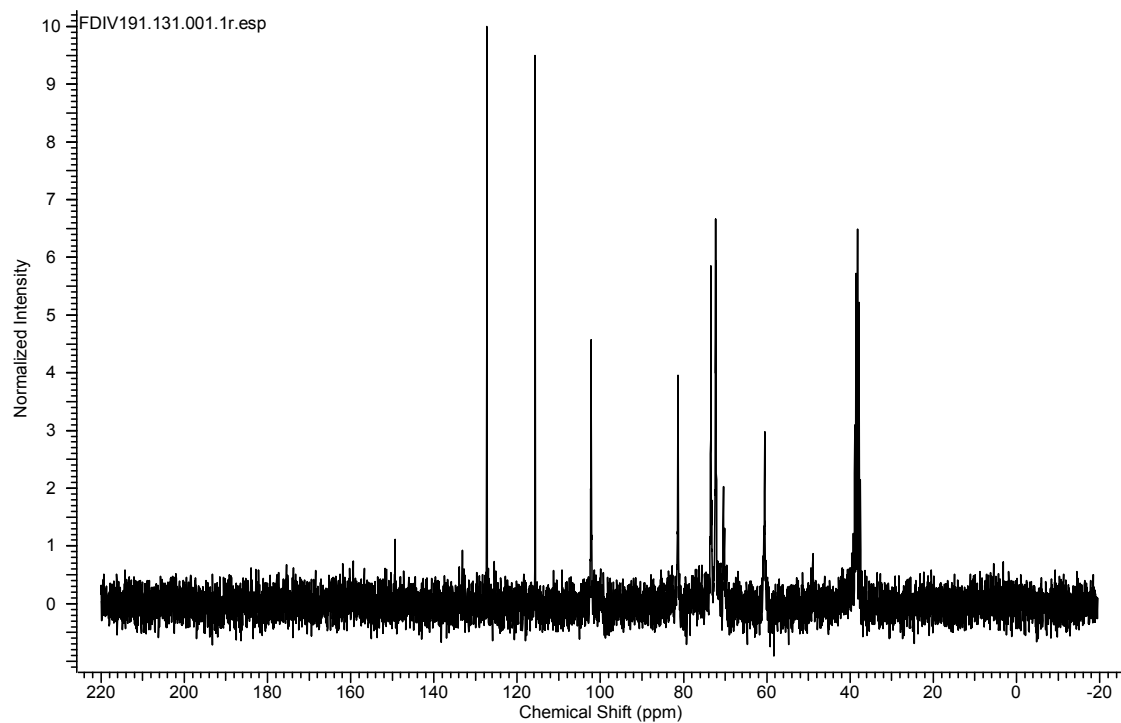
Pseudorotaxane 12:



Pseudorotaxane 13:



Pseudorotaxane 14:

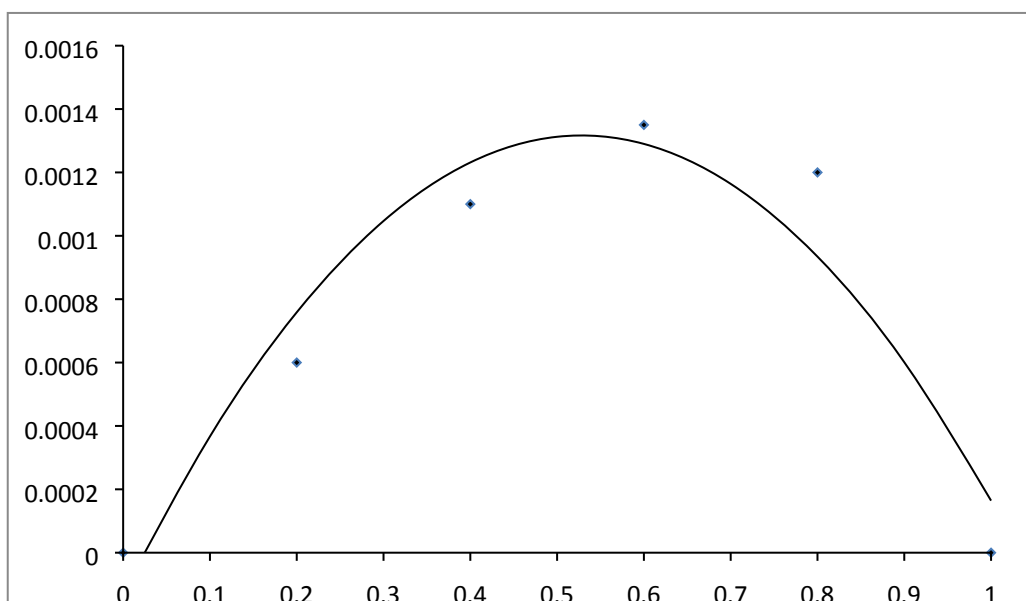


Job plots of pseudo[2]rotaxanes 7 to 15

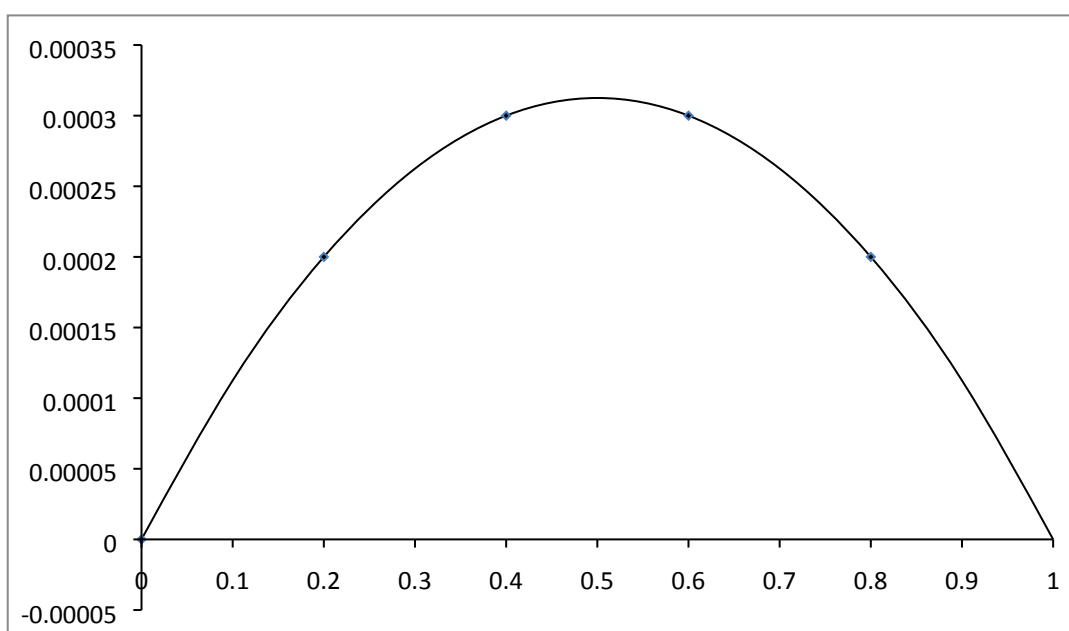
NB : job plots of 11 and 12 : are not given because of less amount of starting the chiral dimer 2 material.

Pseudo[2]rotaxane 7

Variation of CD H₃ protons

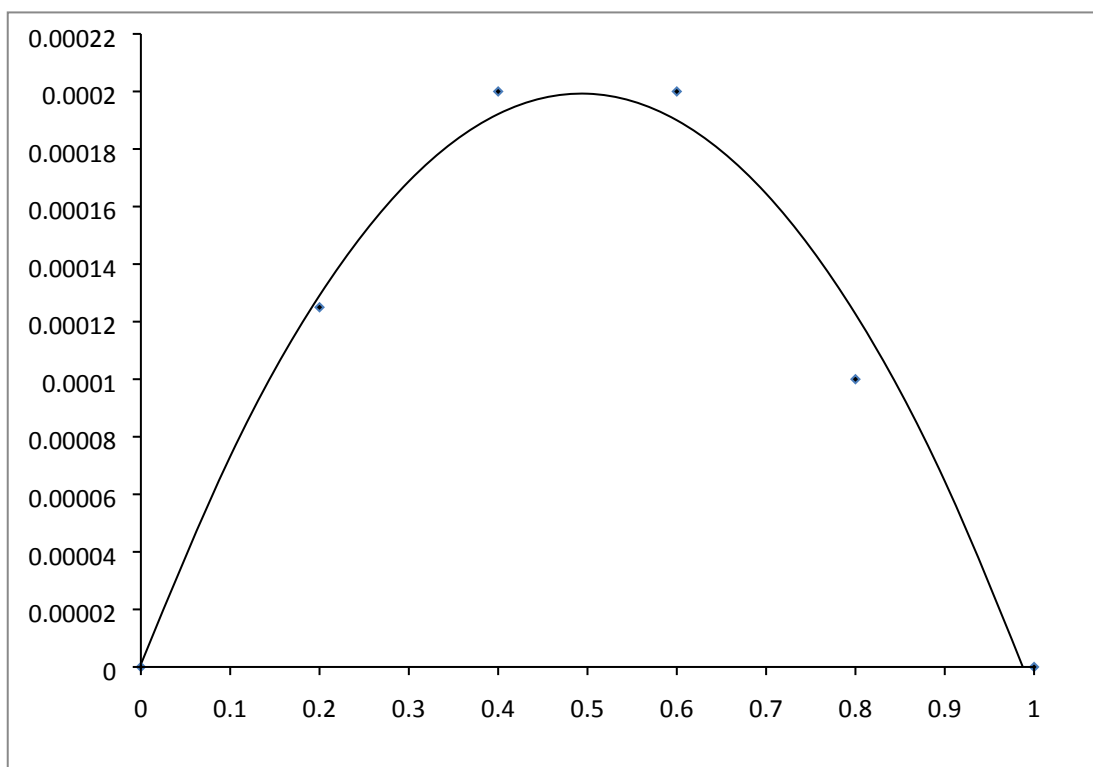


Variation of tosylate aromatic protons

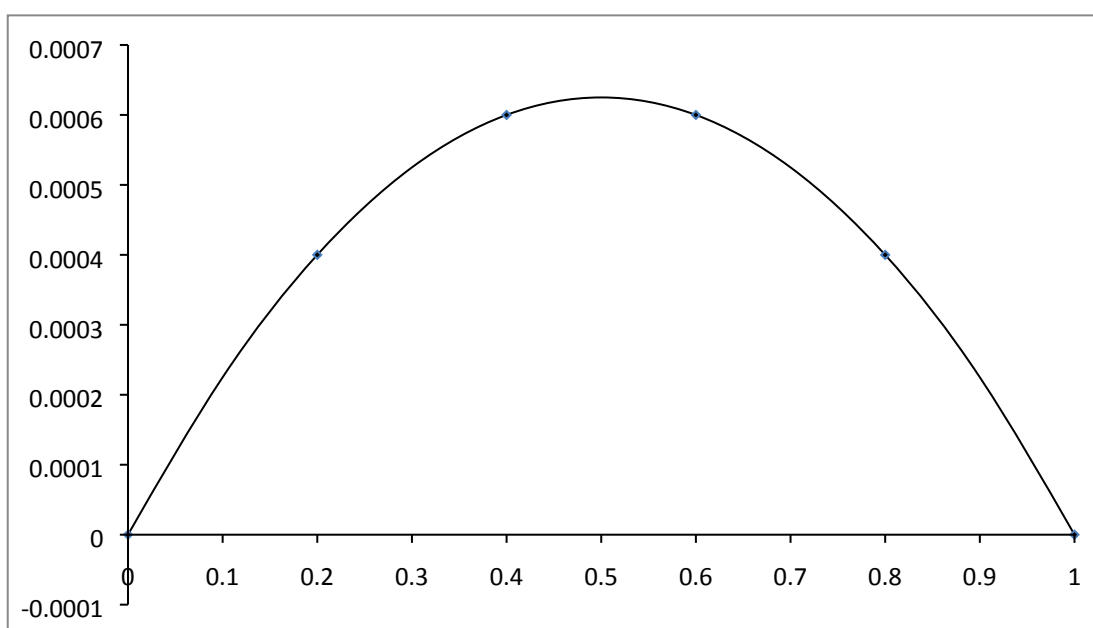


Pseudo[2]rotaxane 8 :

Variation of CD H₃ protons

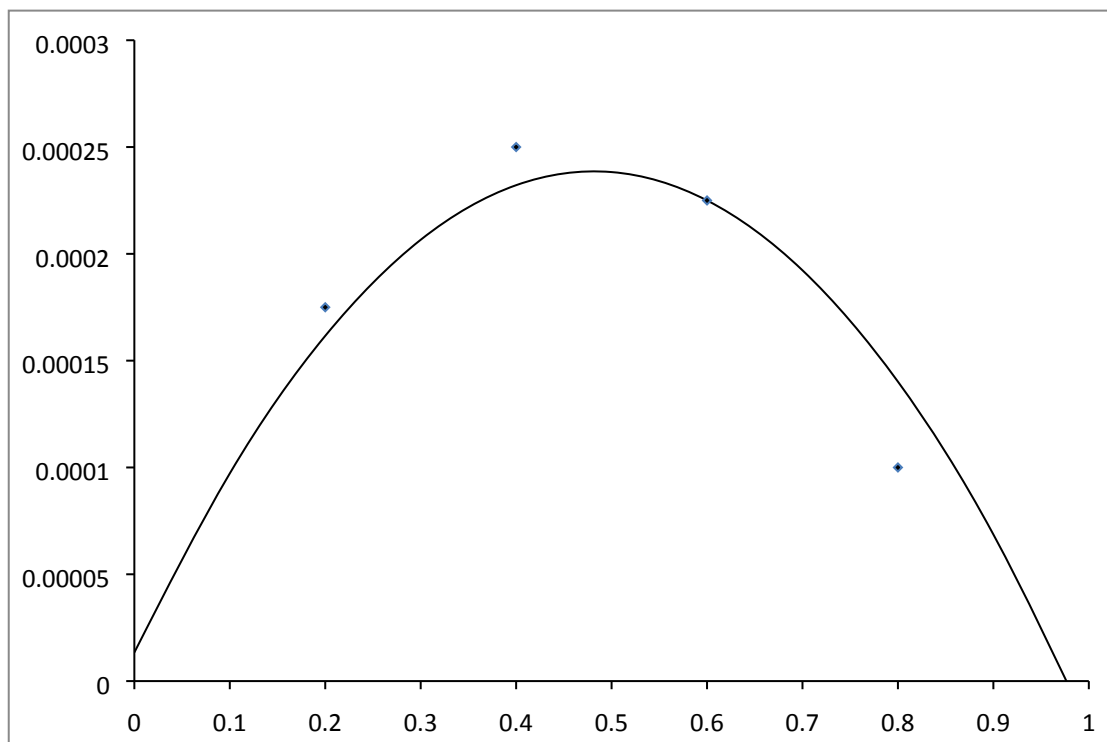


Variation of tosylate aromatic protons

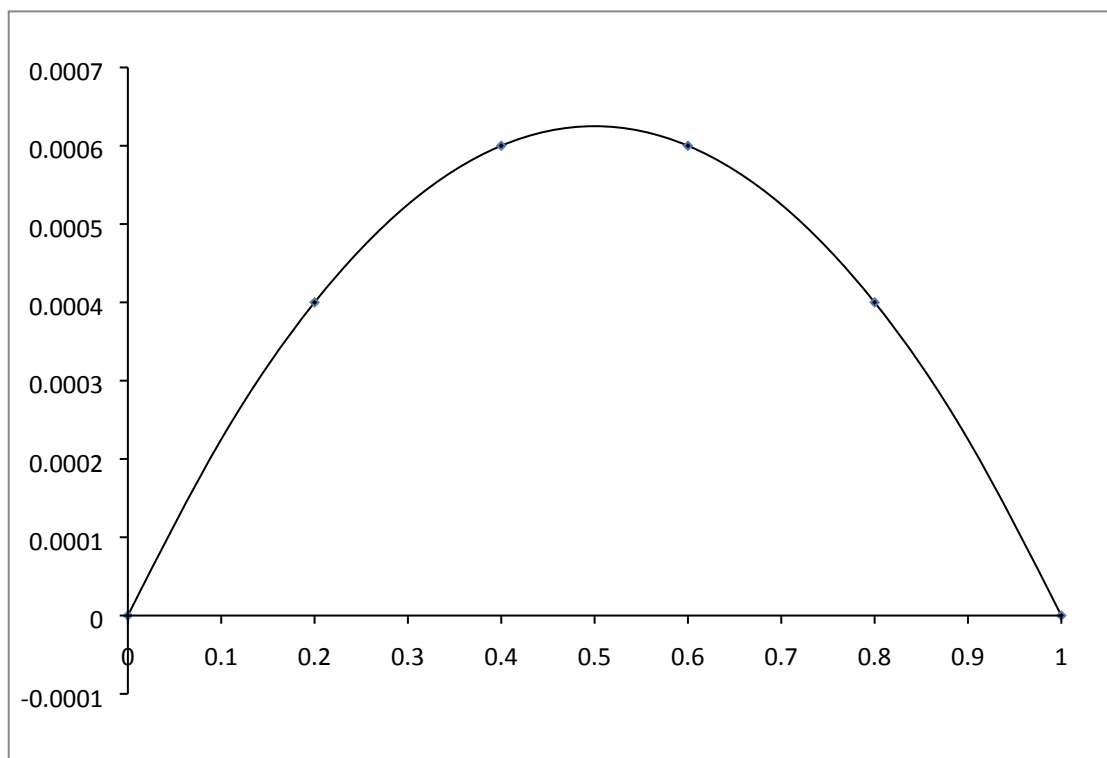


Pseudo[2]rotaxane 9 :

Variation of CD H₃ protons

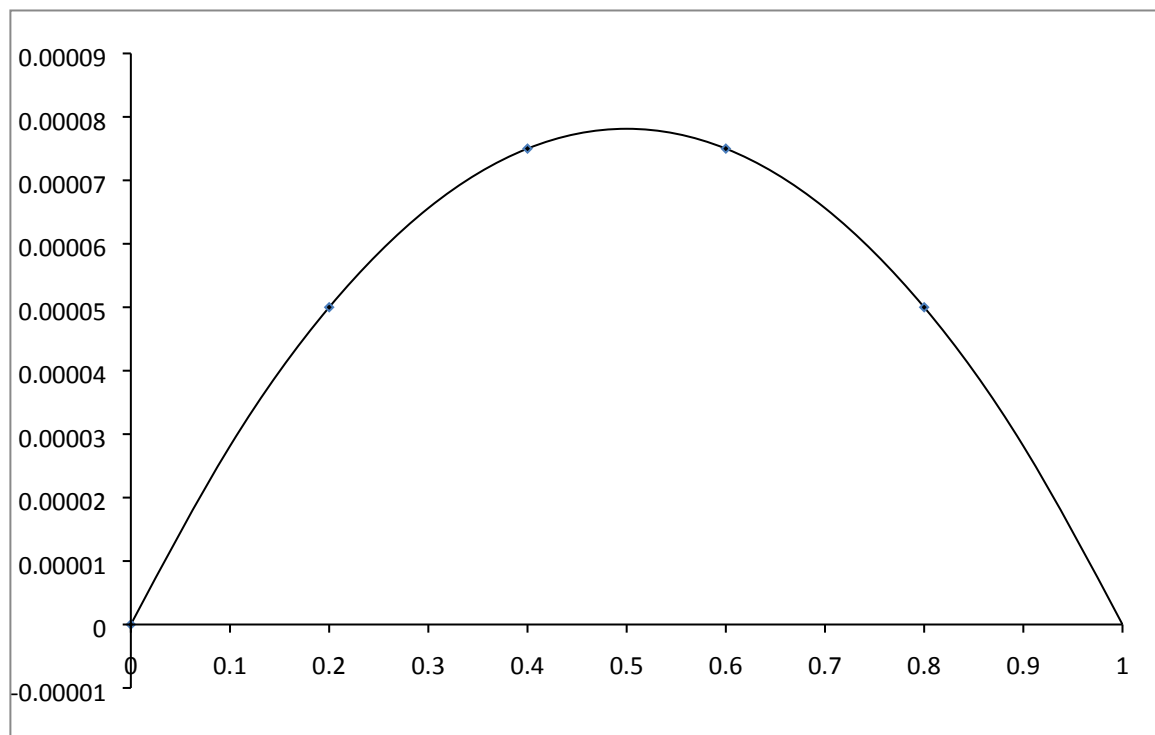


Variation of tosylate aromatic protons

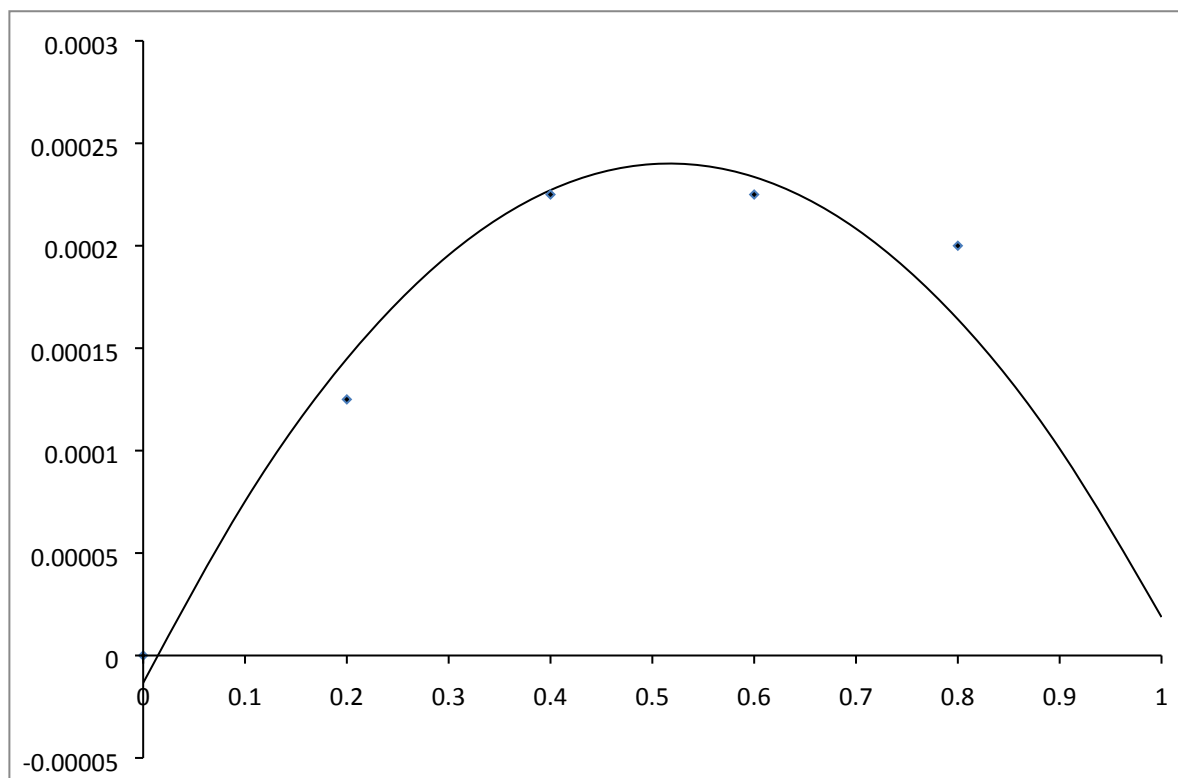


Pseudo[2]rotaxane 10 :

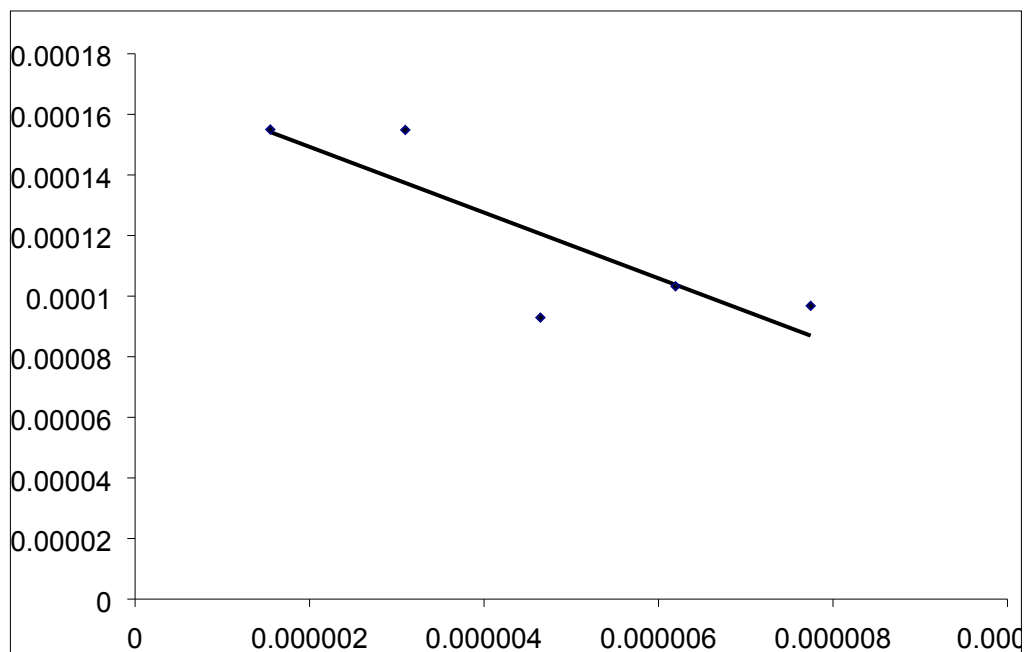
Variation of tosylate aromatic protons



Variation of CD H₃ protons



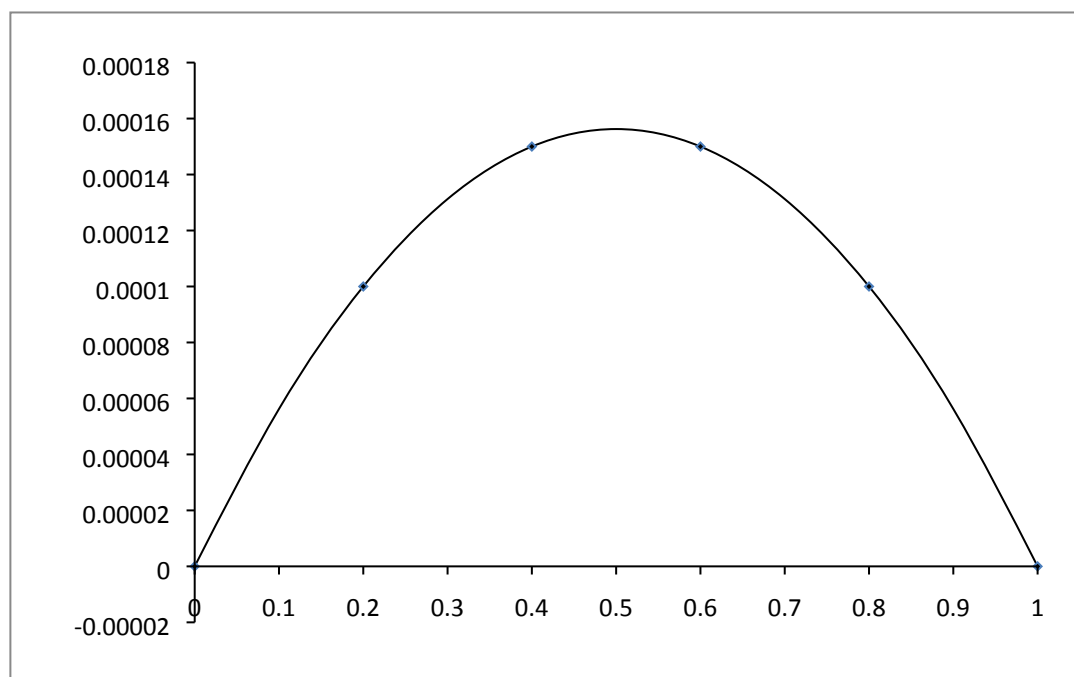
pseudorotaxane 13 Titration of **13** with Zn^{II} bisulfanilate



	1 sur	Ka
pente	10.848	0.092183
ord ori	2.00E-04	5000
		54240

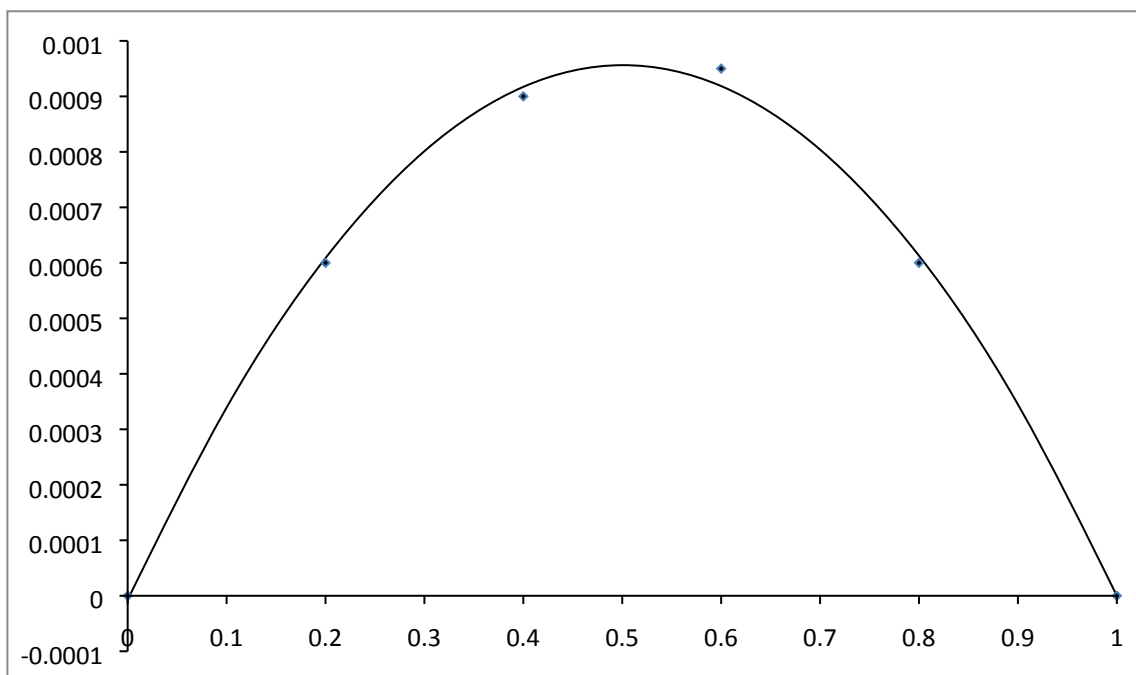
Ka = 54240 M⁻¹

Variation of sulfanilate aromatic protons

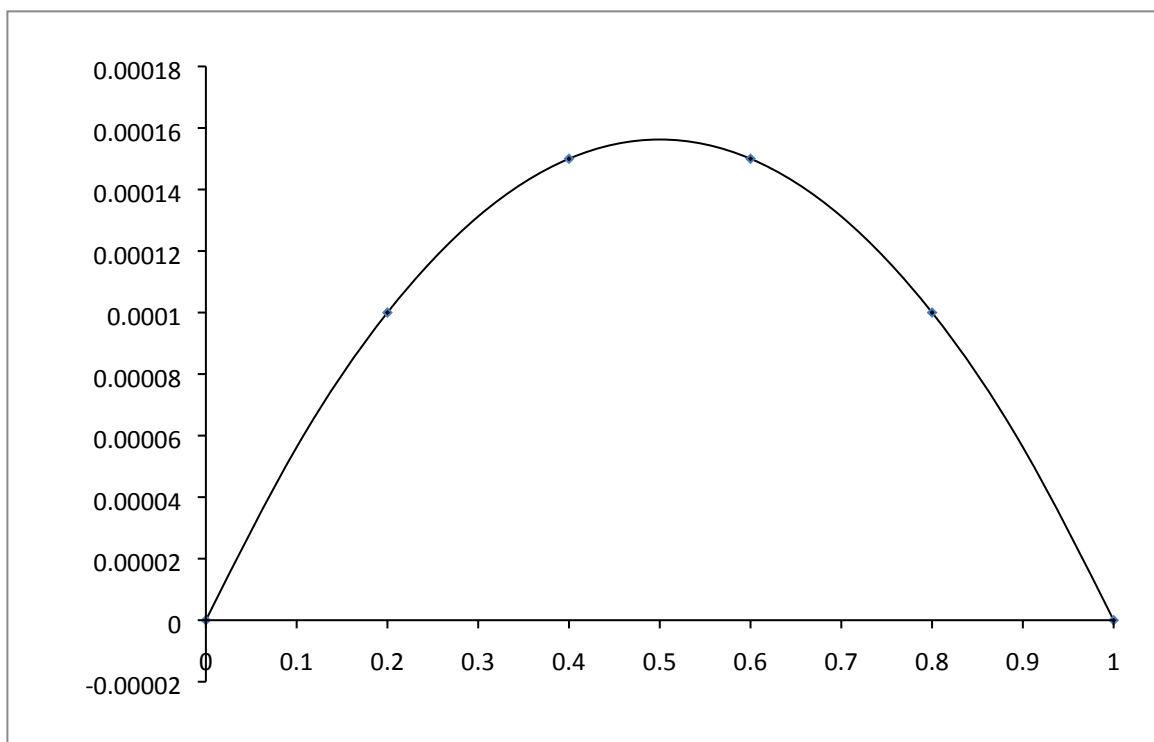


Pseudo[2]rotaxane 14 :

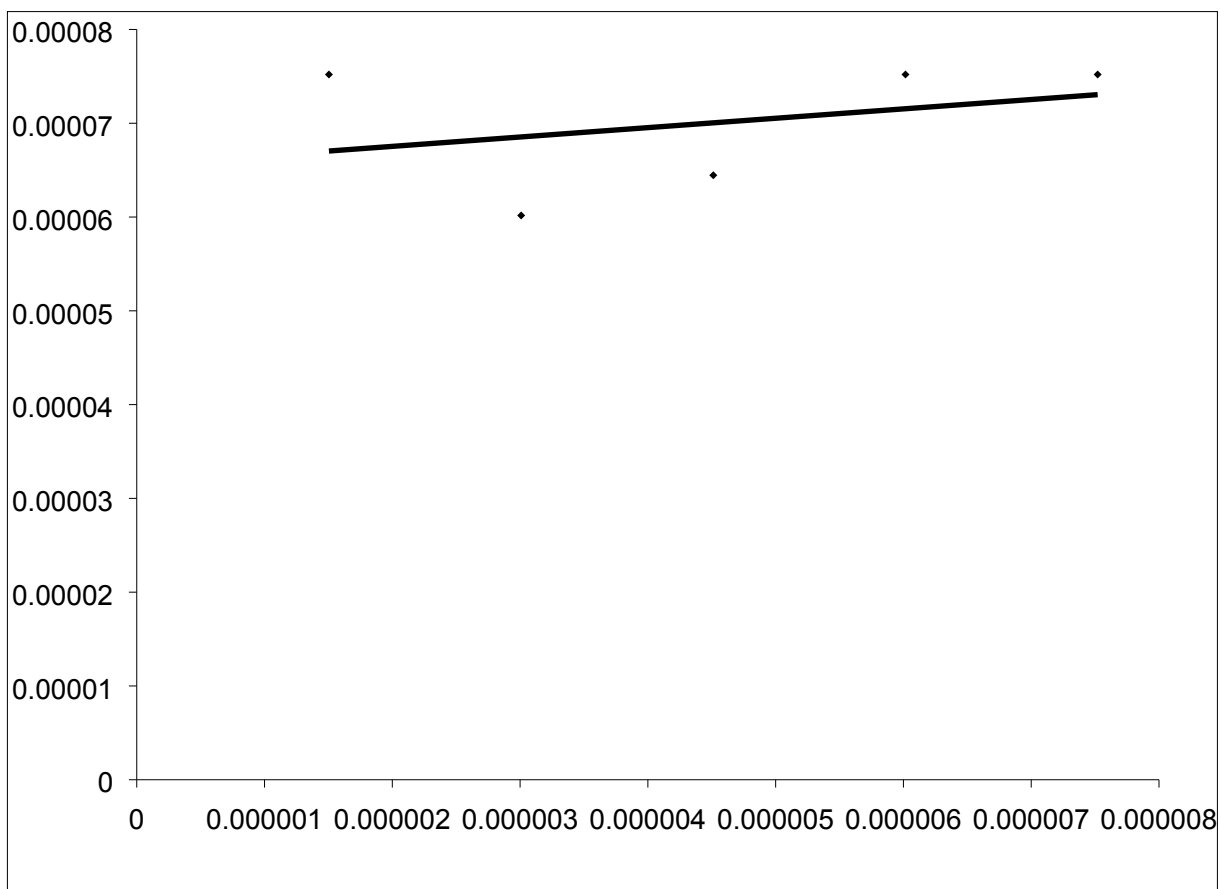
Variation of CD H₃ protons



Variation of sulfanilate aromatic protons



Pseudo[2]rotaxane 15 : Titration of 15 with Zn^{II} bisulfanilate



:

	1 sur	Ka
pende	0.9993	1.0007
ord ori	7.00E-05	14285.71 14275.71

Ka = 14276 M⁻¹

References

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