Tetracarboxylic acids on a thiacalixarene scaffold: synthesis and binding of dopamine hydrochloride

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Electronic Supplementary Information

Table S1. The NMR data for compound 3. The ^{1}H-NMR, ^{13}C-NMR, TOCSY, NOESY, HSQC and HMBC spectra (600 MHz, DMSO-d_6, 303 K)			
Position number	^{13}C chemical shifts (ppm); functional group	^{1}H chemical shifts (ppm); multiplicity; coupling constant (Hz)	Heteronuclear multiple bond correlation
1	156.76; C		H3,5; H7
2	127.64; C		
3	128.24; CH	7.36; s	C1; C4; C4a; C4b
4	145.24; C		H3,5
4a	33.72; C		H3,5; H4b
4b	30.72; CH_3	1.20; s	H3,5; C4a
5	128.24; CH	7.36; s	
6	127.64; C		
7	67.49; CH_2	3.83; t; 7.5	H8; H9; C1; C8; C9
8	28.48; CH_2	1.35; m	H7; H9; C7; C9
9	35.98; CH_2	3.07; m	H7; H8; C7; C8; C11, C12
10	NH	8.59; s	
11	164.91; CO		H12; H14
12	124.52; CH	6.20; s	H14; C11; C13; C14; C15
13	142.38; C		H12; H14
14	21.59; CH_3	1.95; s	H12; C11; C12; C13; C15
15	167.72; COOH		H12; H14

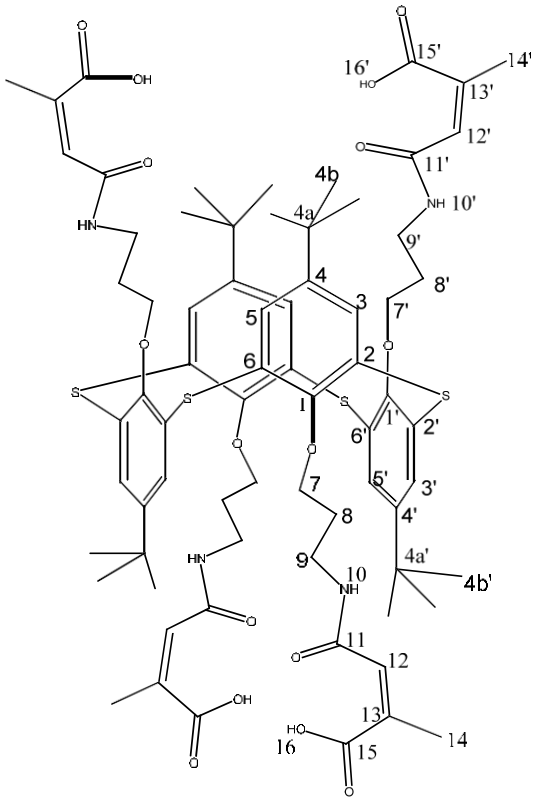


Fig. S1. ^{1}H NMR spectrum of *1,3-alternate*-2, DMSO-d$_6$, 298 K, 400 MHz

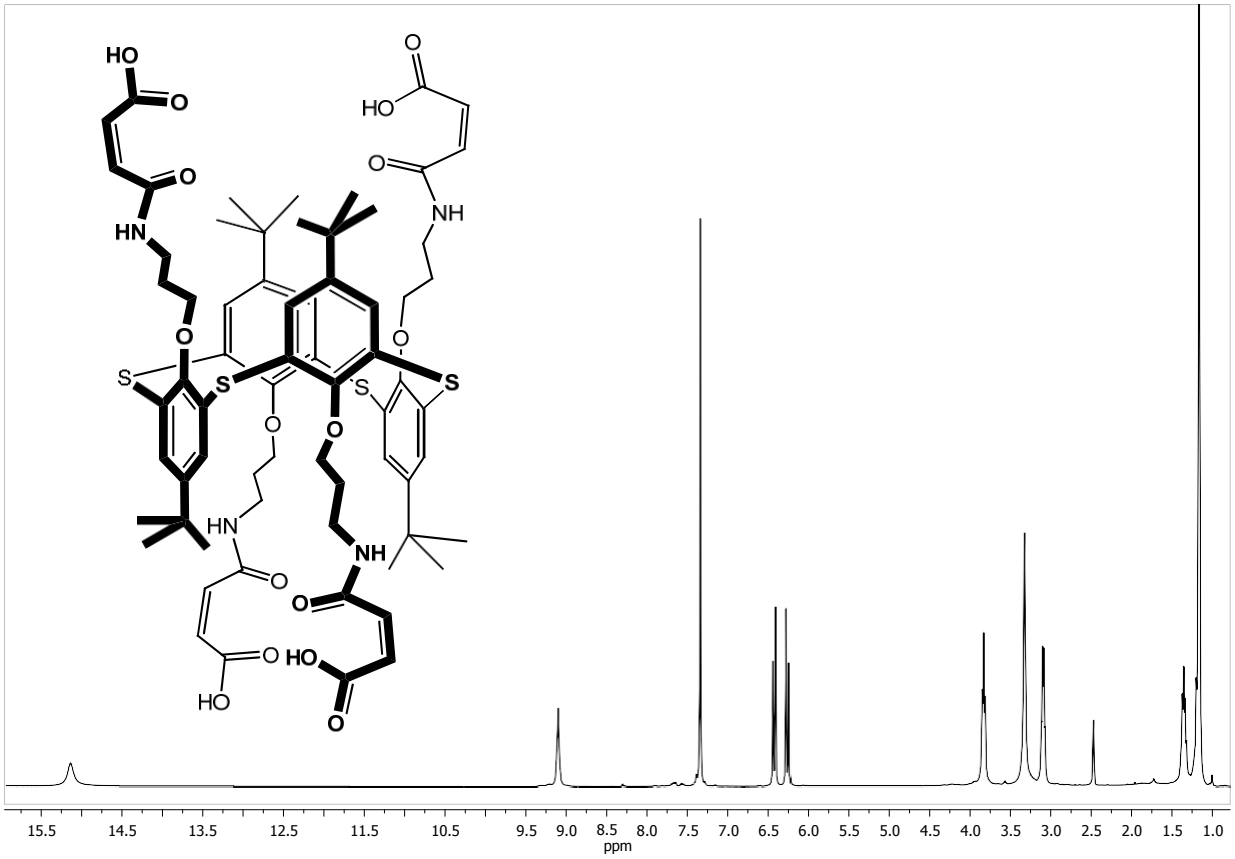


Fig. S2. ^{1}H NMR spectrum of *1,3-alternate*-3, DMSO-d$_6$, 298 K, 400 MHz

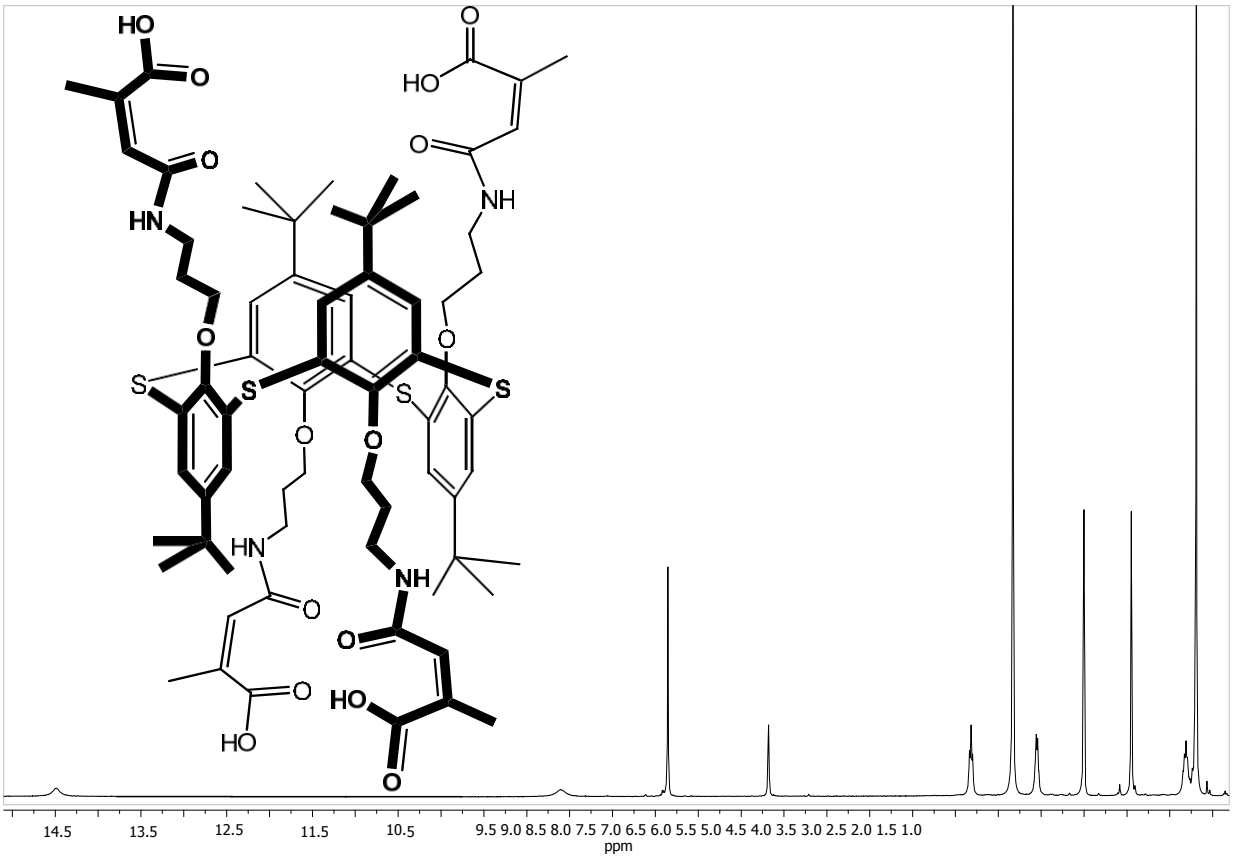


Fig. S3. ^{1}H NMR spectrum of *1,3-alternate*-4, DMSO-d_6, 298 K, 400 MHz

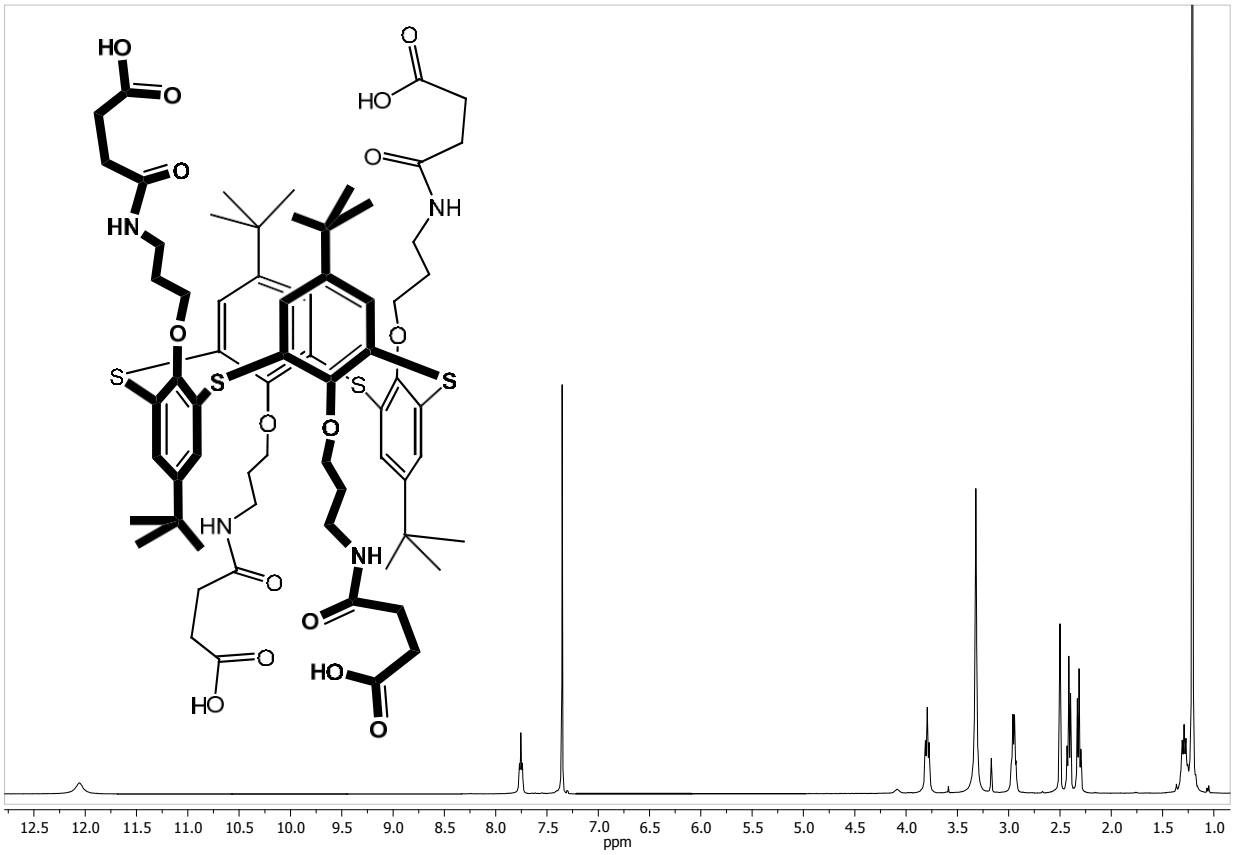


Fig. S4. ^{1}H NMR spectrum of *1,3-alternate*-5, DMSO-d_6, 298 K, 400 MHz

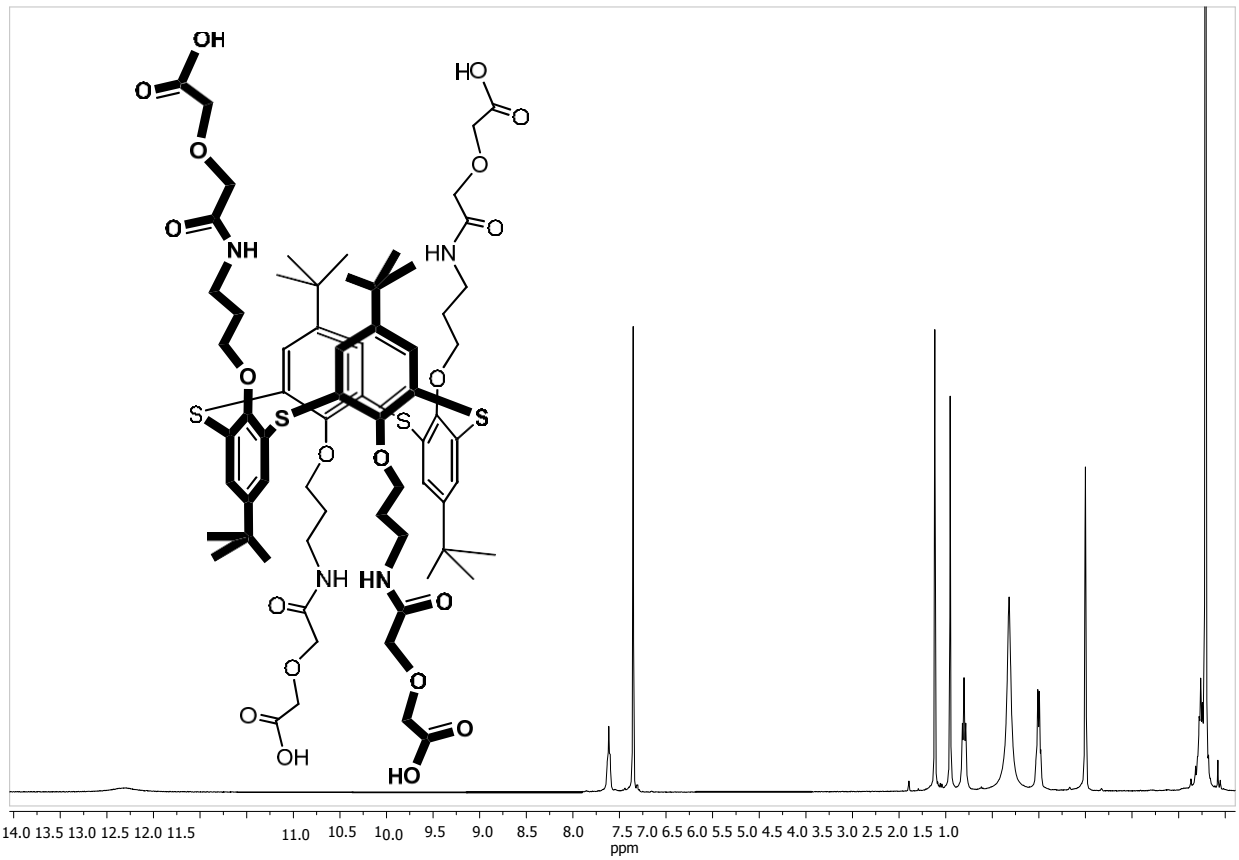


Fig. S5. ^{1}H NMR spectrum of *1,3-alternate*-6, $CDCl_3$, 298 K, 400 MHz

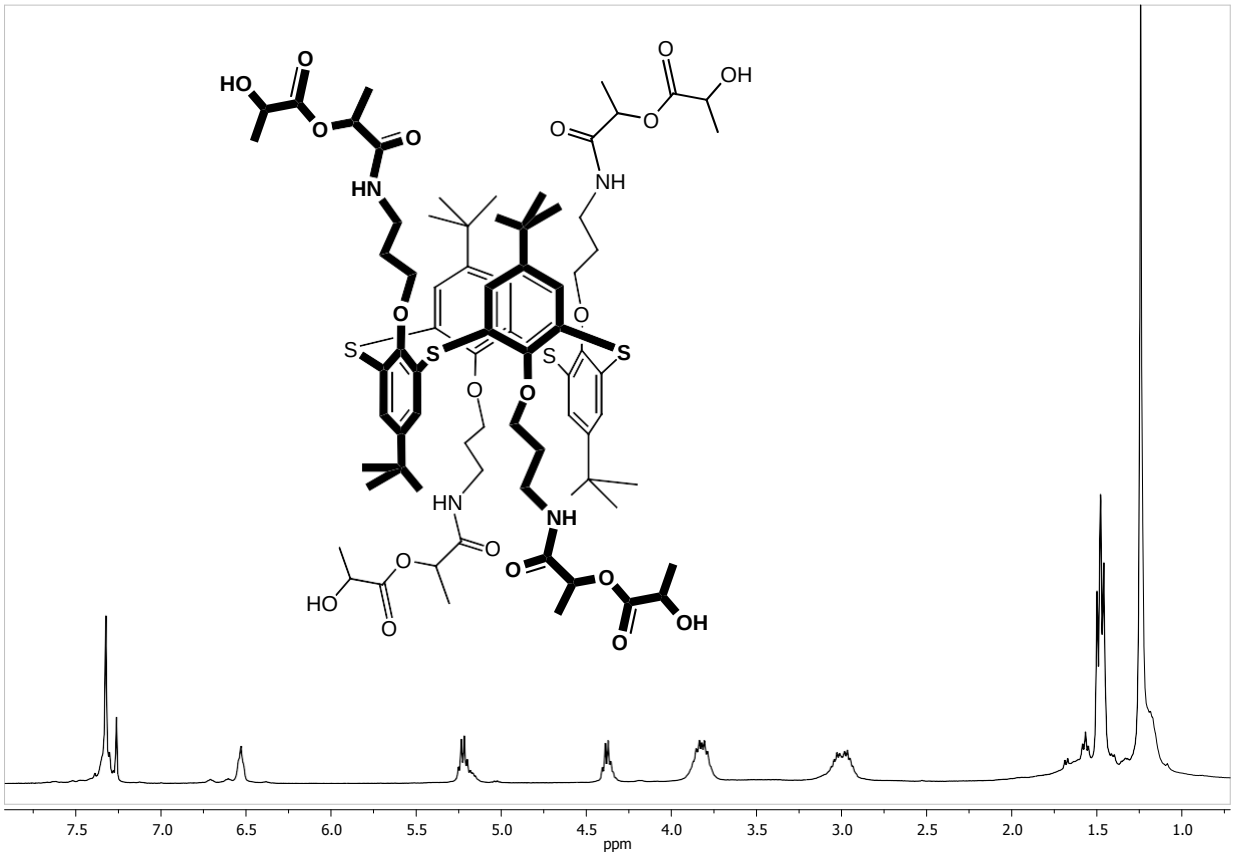


Fig. S6. ^{13}C NMR spectrum of *1,3-alternate*-2, DMSO-d_6, 298 K, 100 MHz

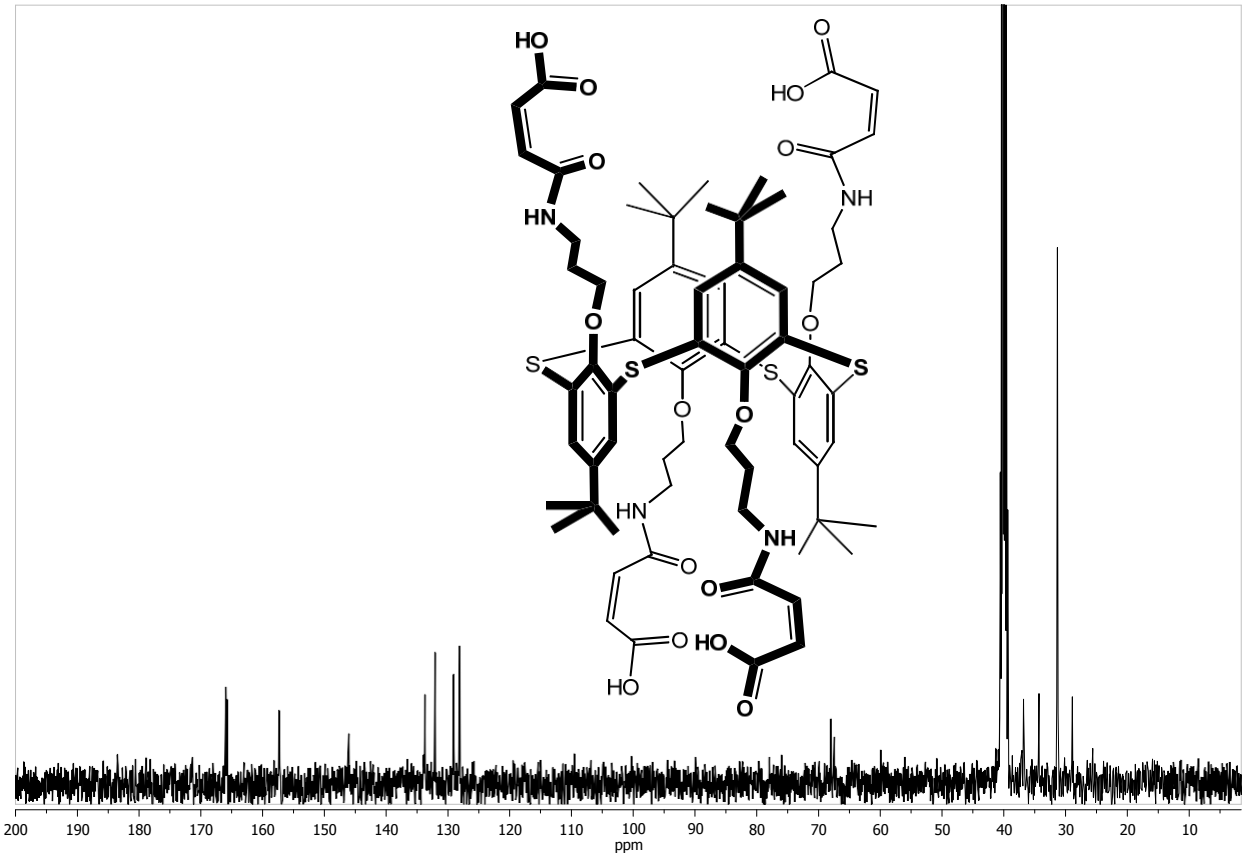


Fig. S7. ^{13}C NMR spectrum of *1,3-alternate*-3, DMSO-d$_6$, 298 K, 100 MHz

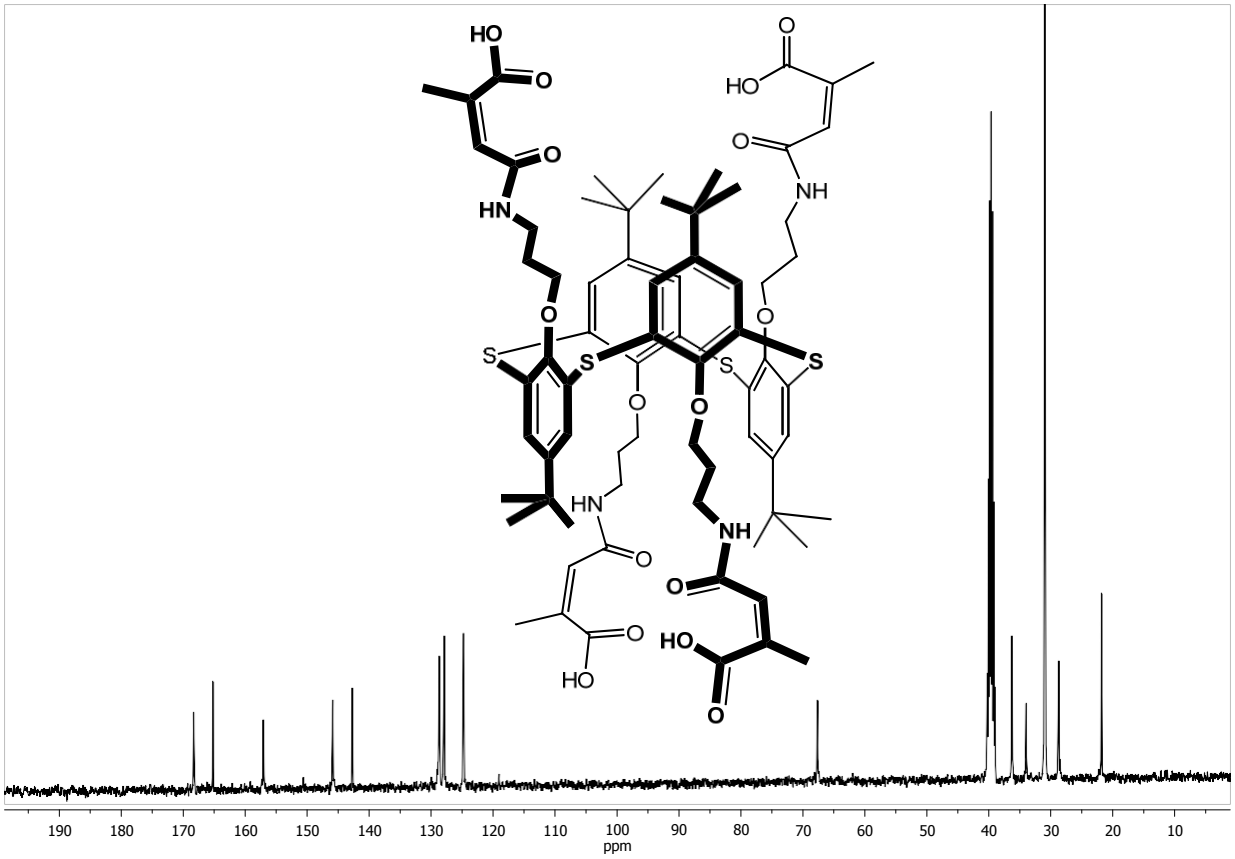


Fig. S8. ^{13}C NMR (DEPT) spectrum of *1,3-alternate*-4, DMSO-d$_6$, 298 K, 100 MHz

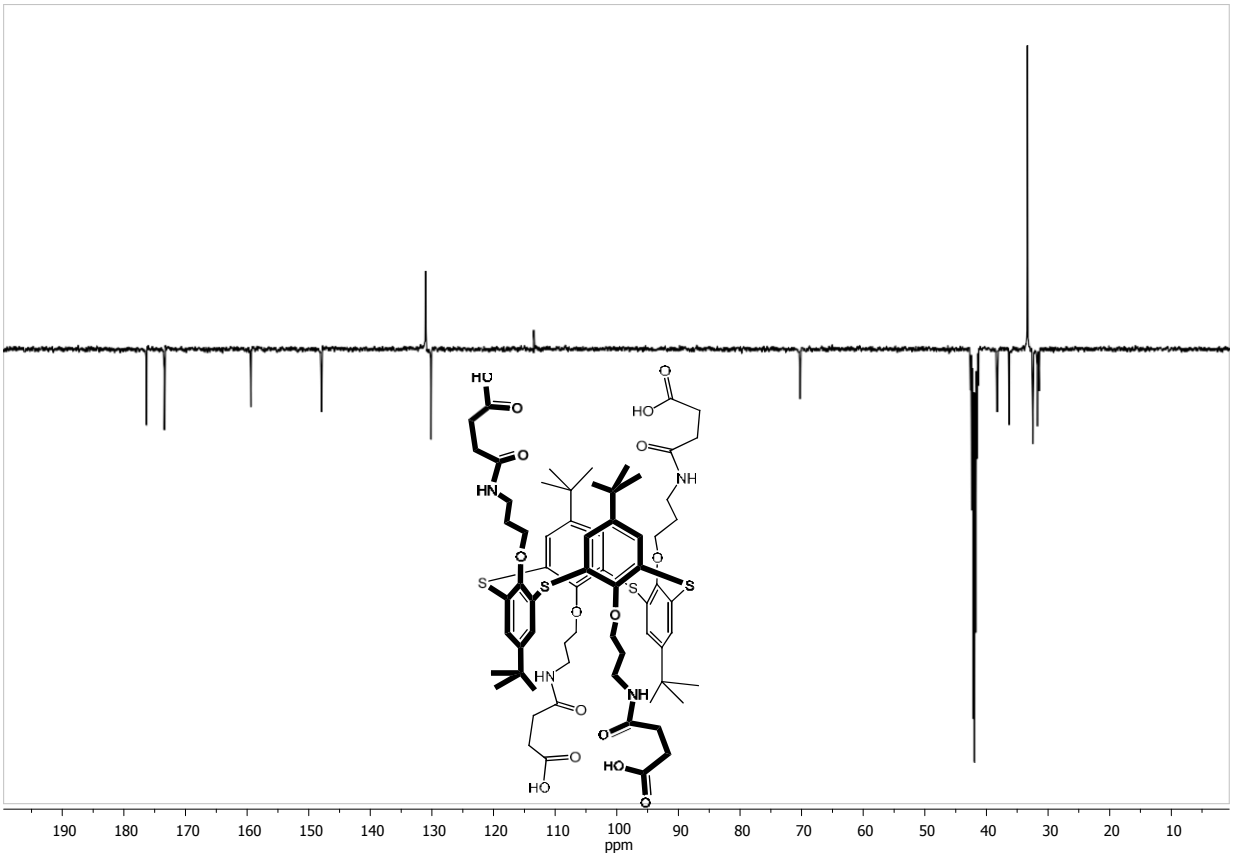


Fig. S9. ^{13}C NMR spectrum of *1,3-alternate*-5, DMSO-d_6, 298 K, 100 MHz

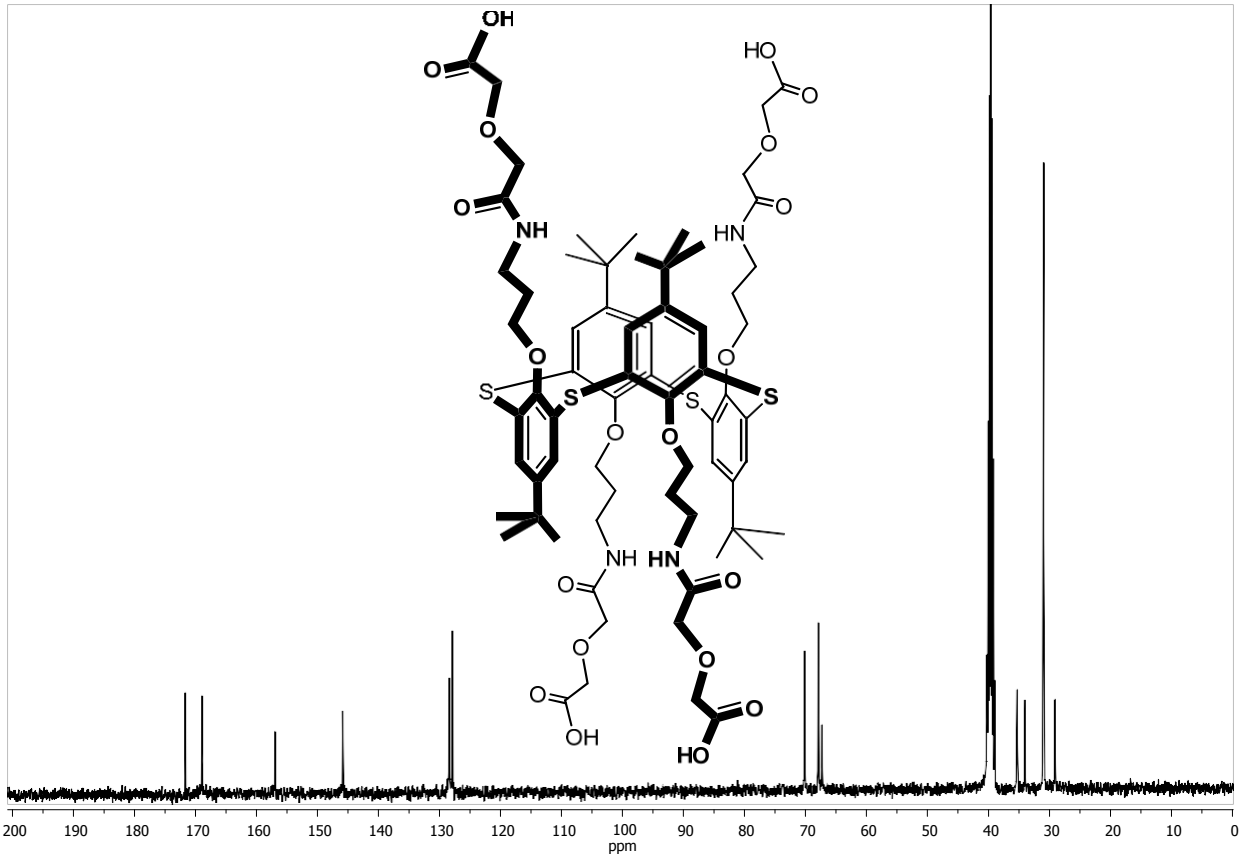


Fig. S10. ^{13}C NMR (DEPT) spectrum of *1,3-alternate*-6, $CDCl_3$, 298 K, 100 MHz

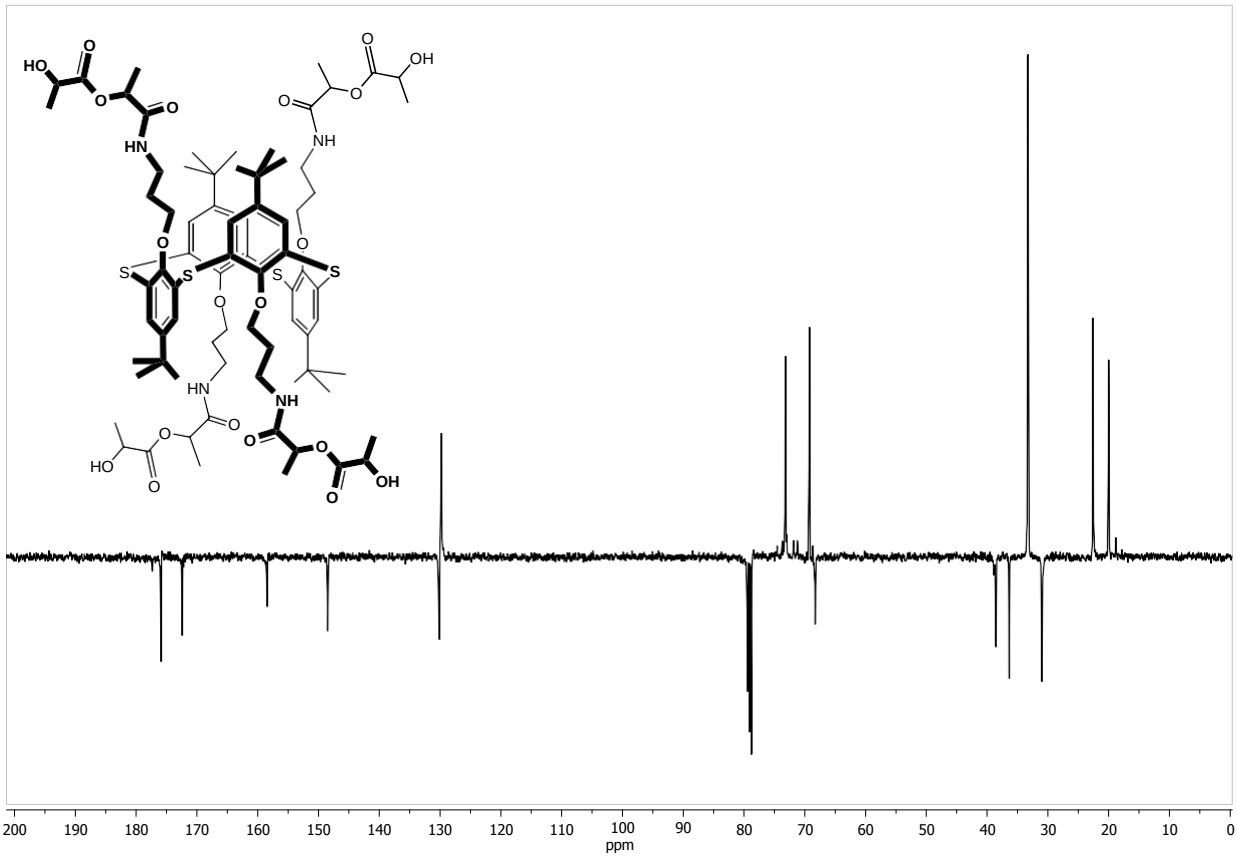


Fig. S11. NOESY ^{1}H-^{1}H spectrum of compound 2, DMSO-d_6, 298 K, 400 MHz

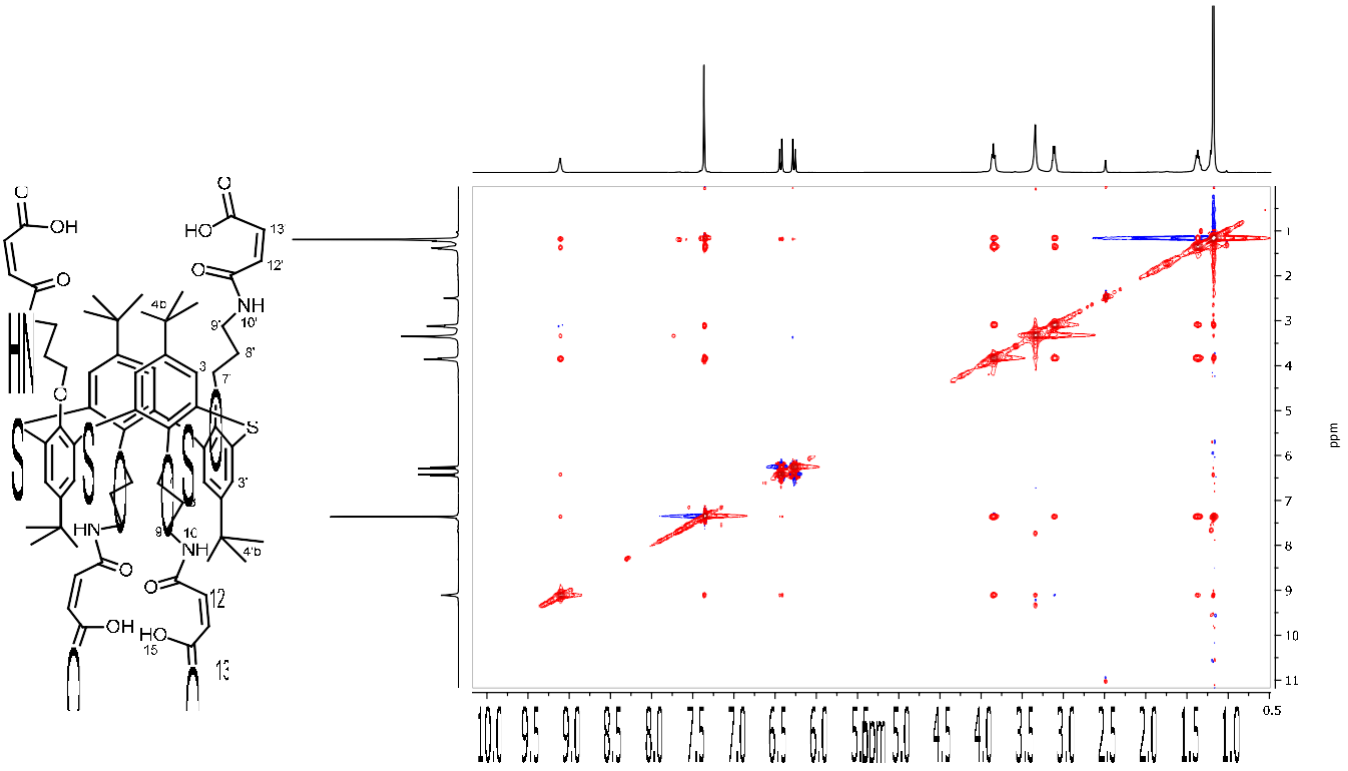


Fig. S12. NOESY ^{1}H-^{1}H spectrum of compound 3, DMSO-d_6, 298 K, 400 MHz

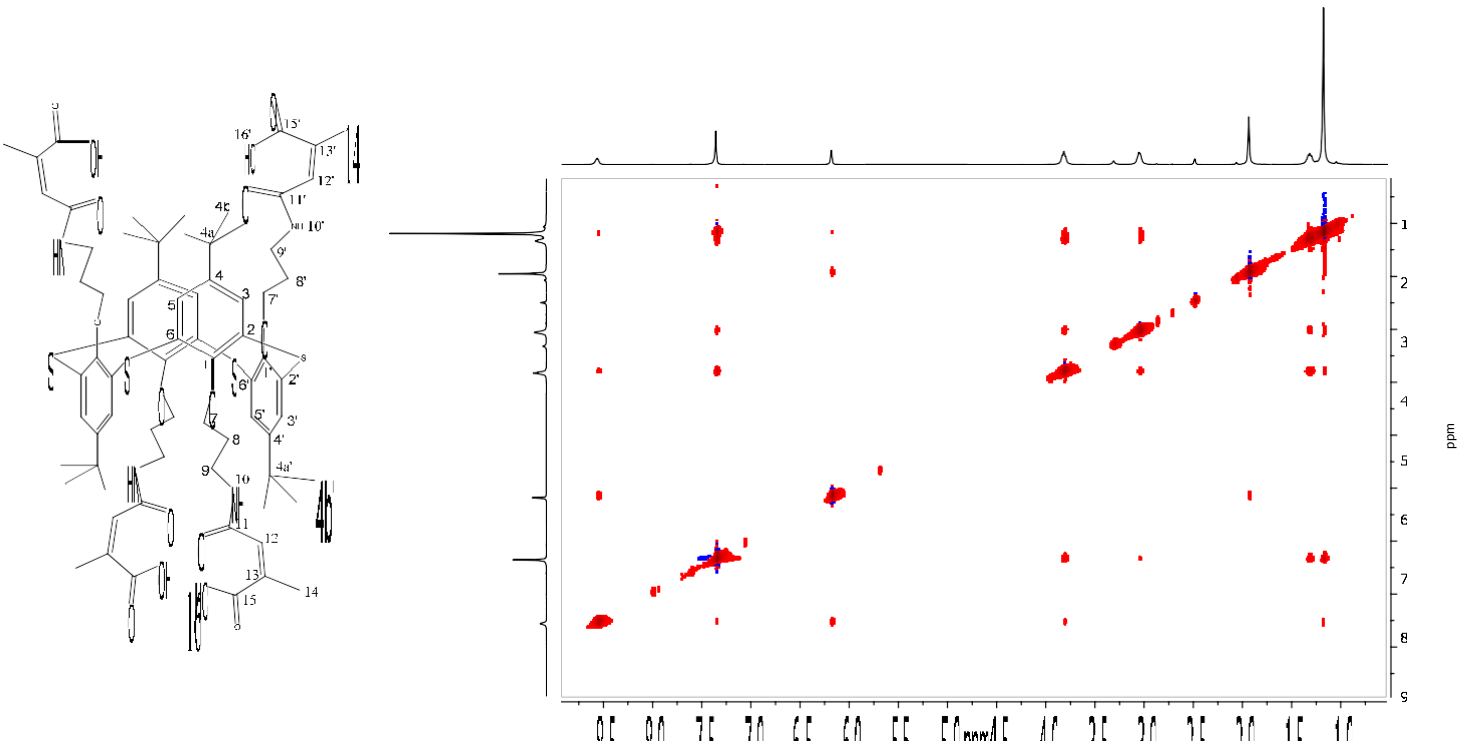


Fig. S13. NOESY ^{1}H-^{1}H spectrum of compound 4, DMSO-d_6, 298 K, 400 MHz

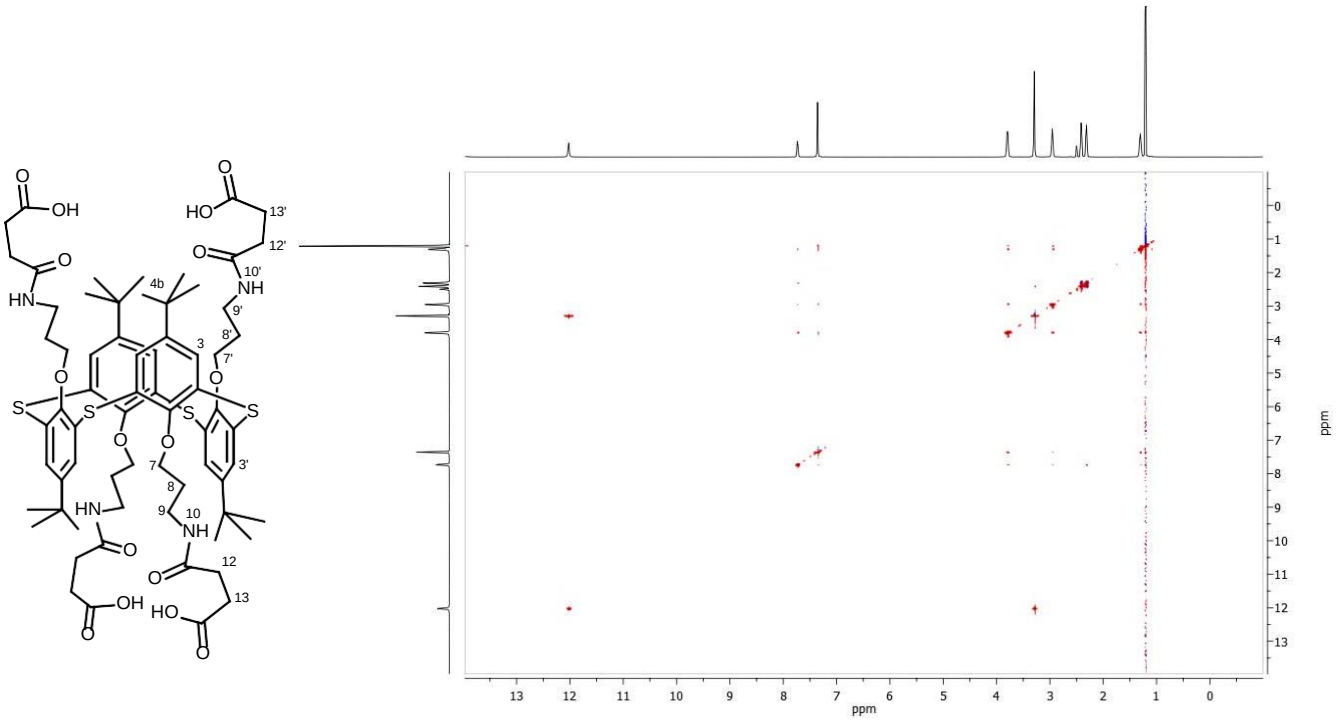


Fig. S14. NOESY ^{1}H-^{1}H spectrum of compound 5, DMSO-d_6, 298 K, 400 MHz

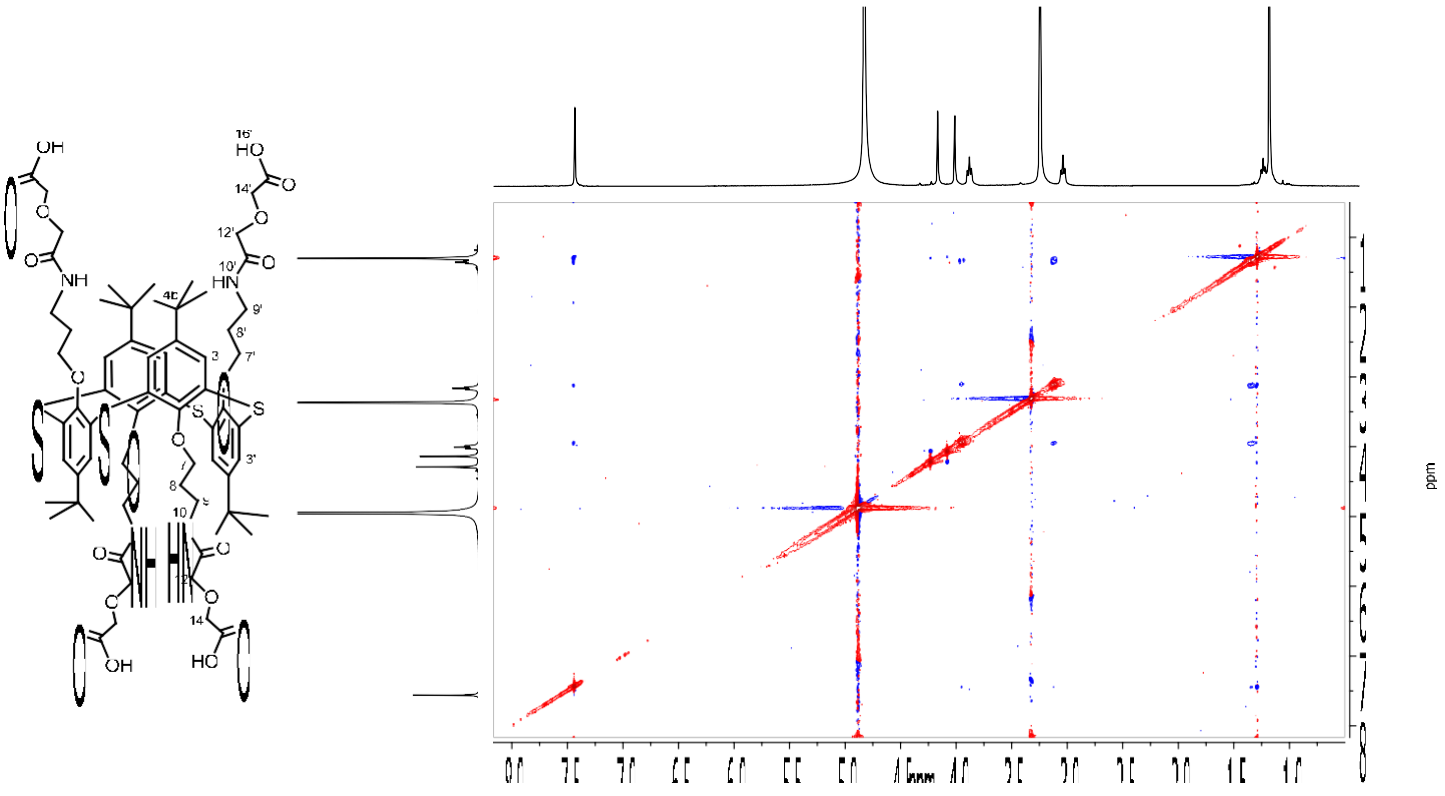


Fig. S15. NOESY ^{1}H-^{1}H spectrum of compound 6, $CDCl_3$, 298 K, 400 MHz

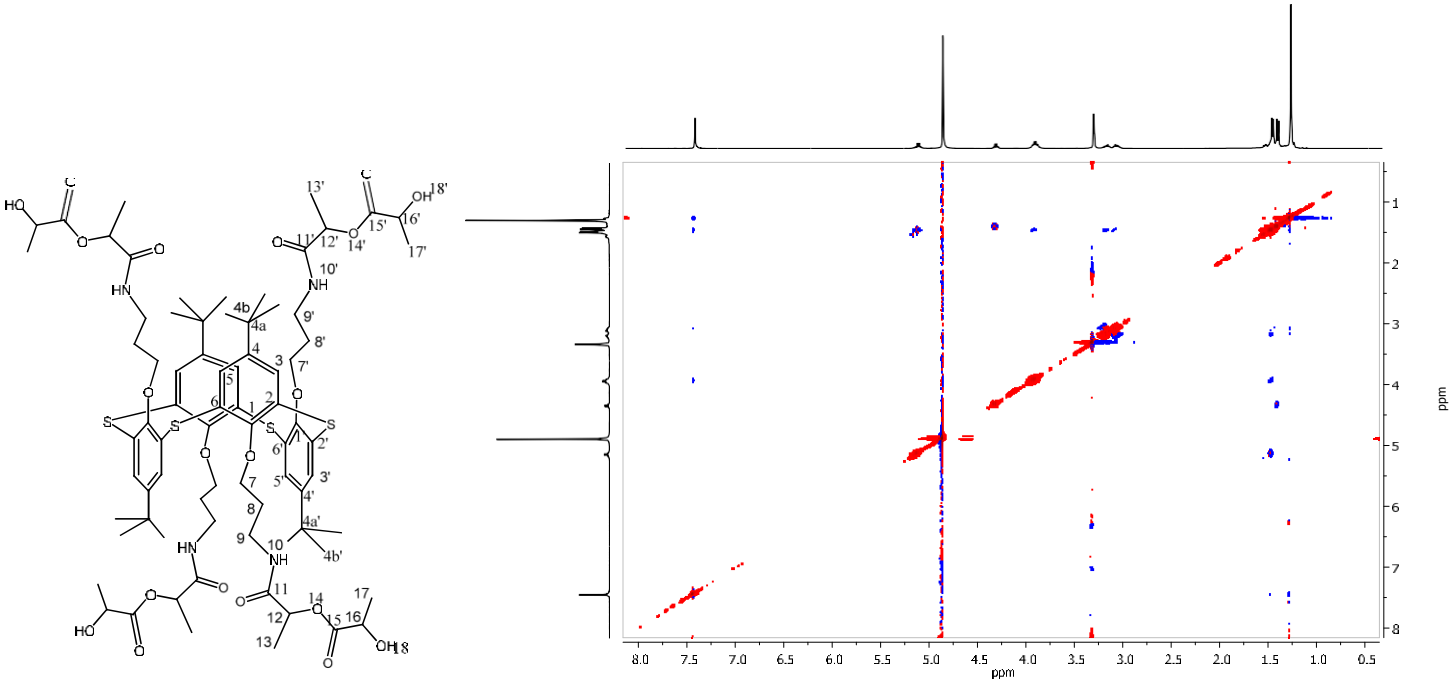


Fig. S16. ^{1}H-^{1}H TOCSY spectrum of compound 3, DMSO-d_6, 303 K, 600 MHz

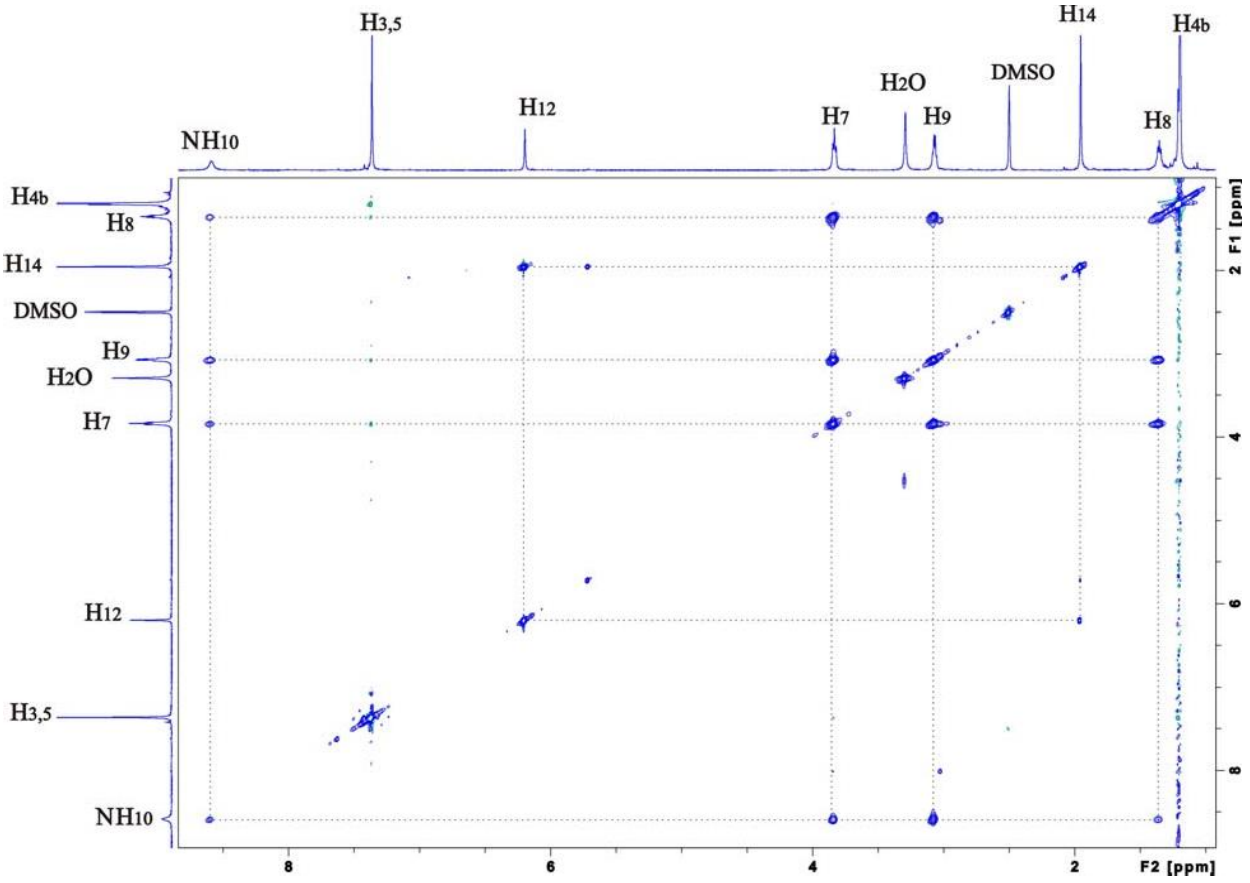


Fig. S17. ^{1}H-^{13}C HSQC spectrum of compound 3, DMSO-d_6, 303 K, 600 MHz

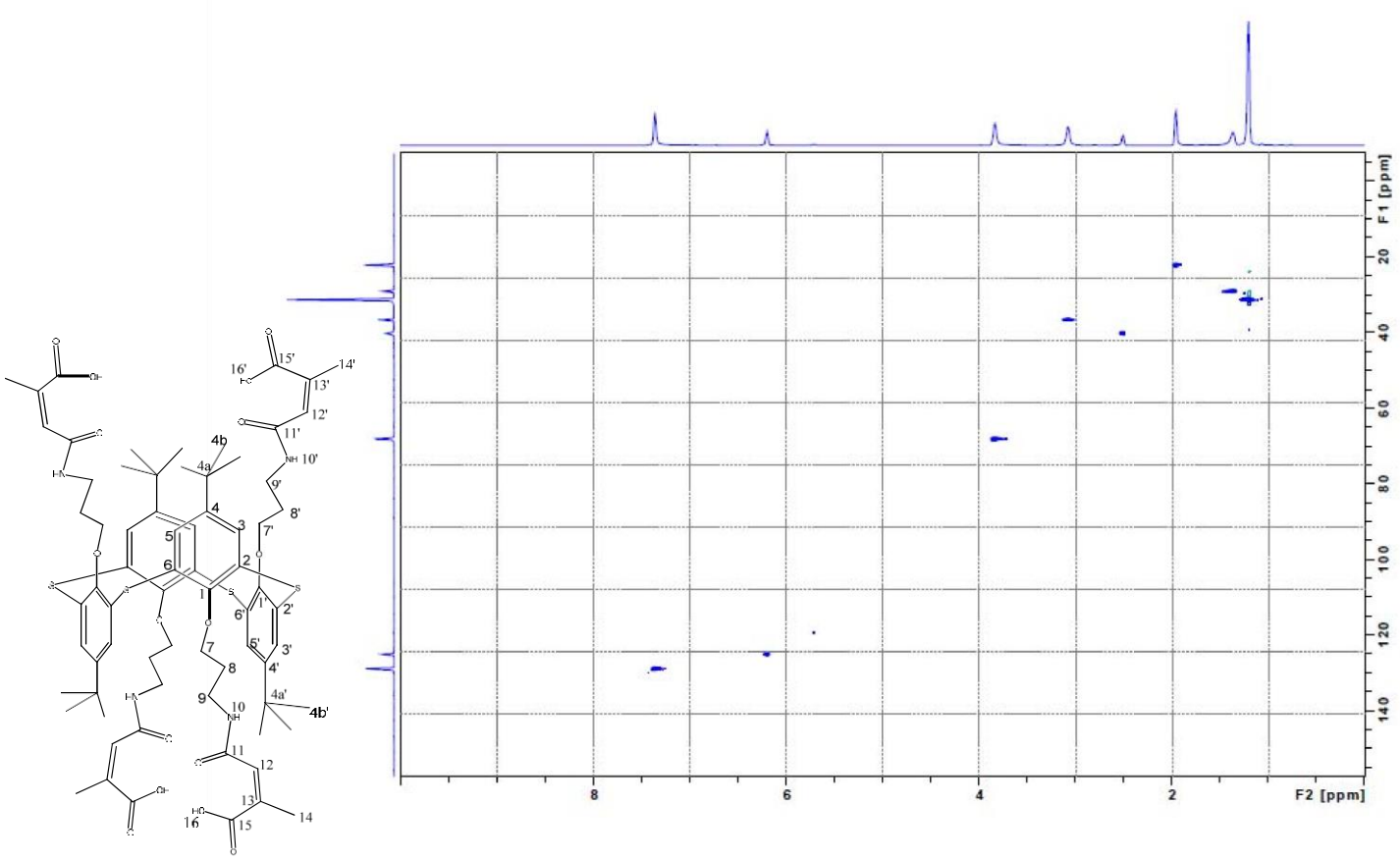


Fig. S18. ^{1}H-^{13}C HMBC spectrum of compound 3, DMSO-d_6, 303 K, 600 MHz

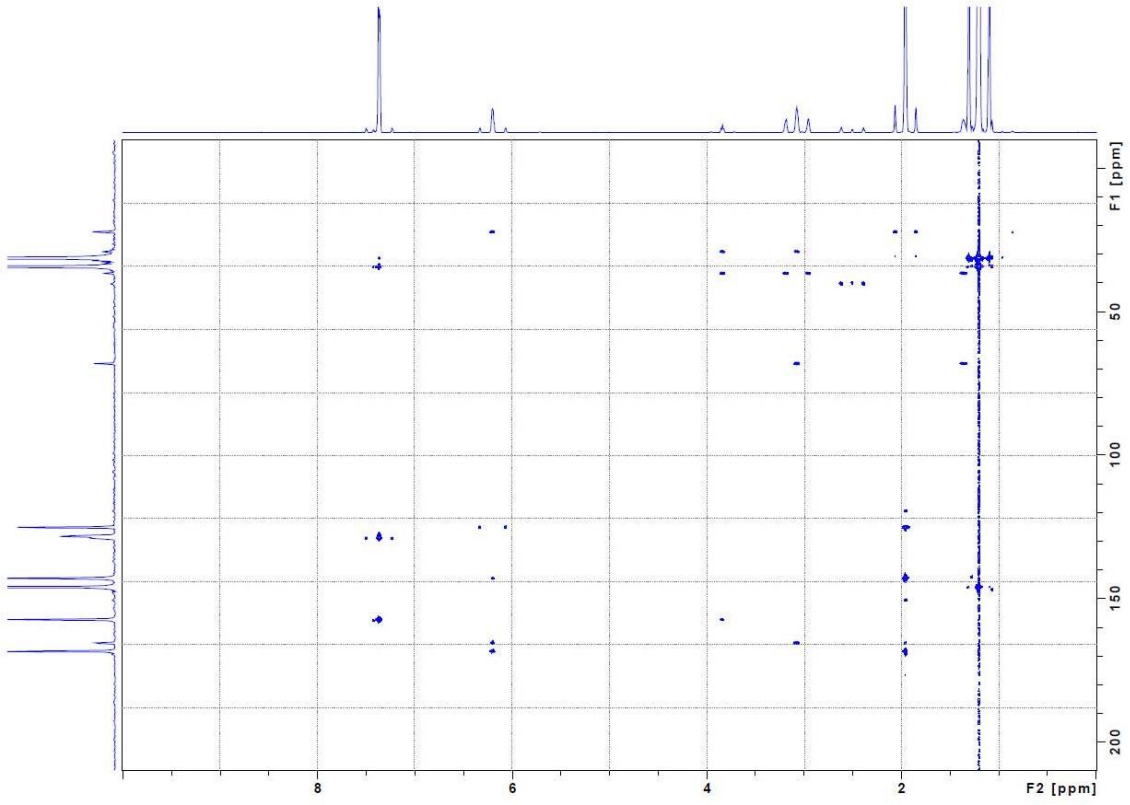


Fig. S19. Mass spectrum (MALDI TOF, 4-nitroaniline matrix) of *1,3-alternate*-2

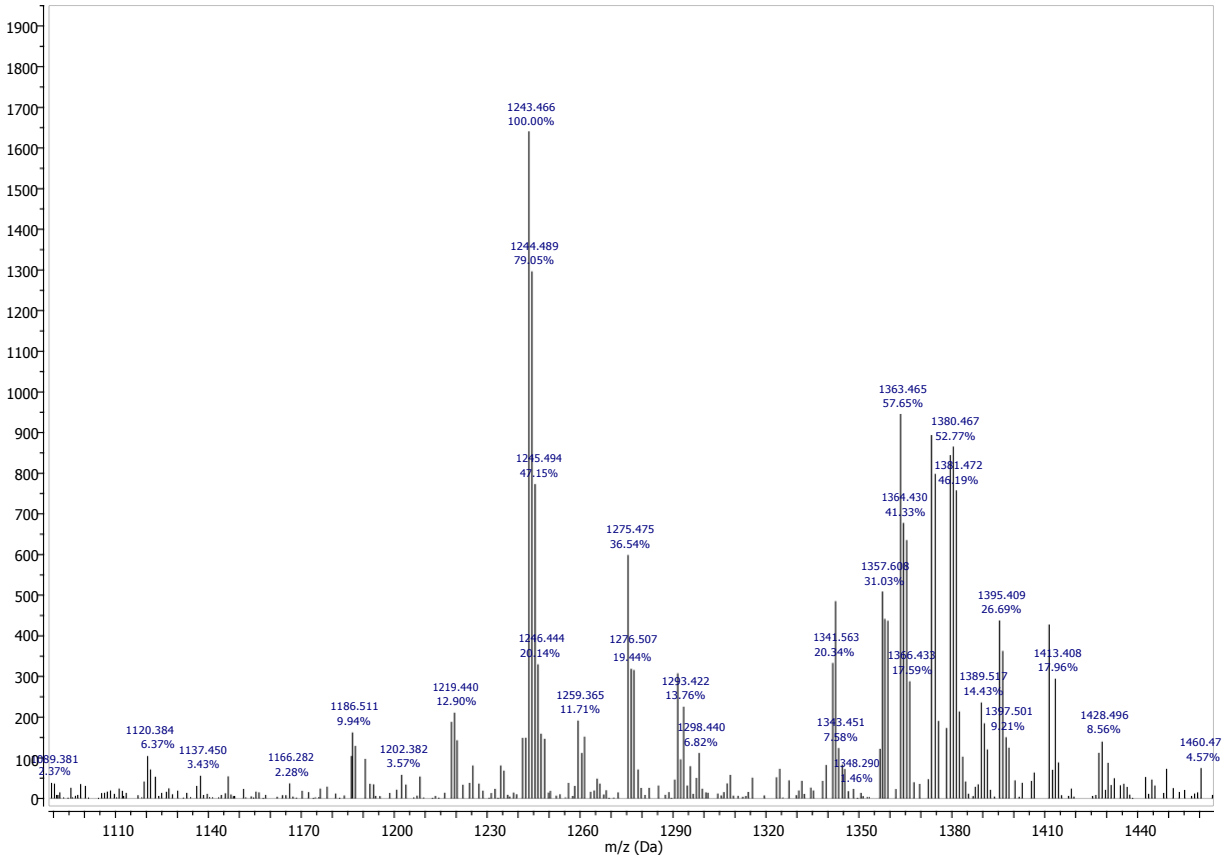


Fig. S20. Mass spectrum (MALDI TOF, 4-nitroaniline matrix) of *1,3-alternate*-3

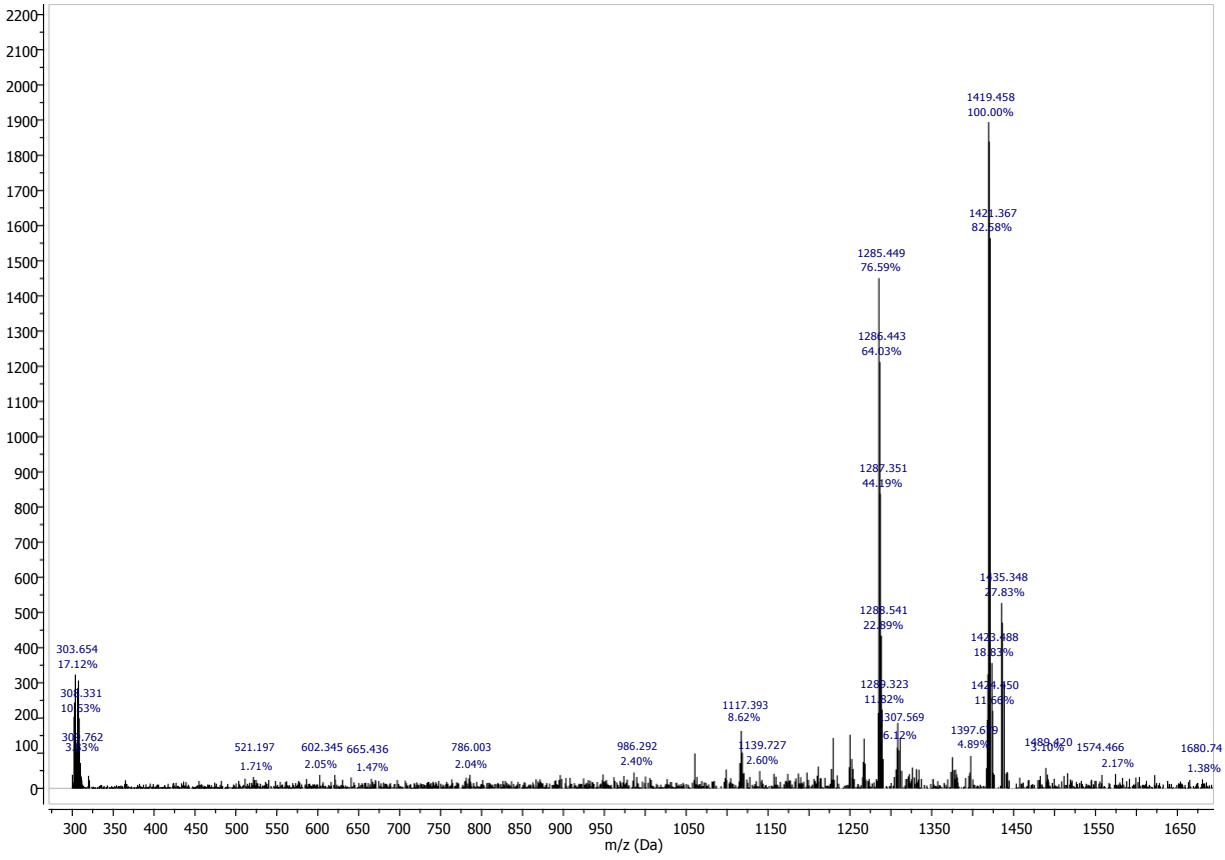


Fig. S21. Mass spectrum (MALDI TOF, 4-nitroaniline matrix) of *1,3-alternate*-4

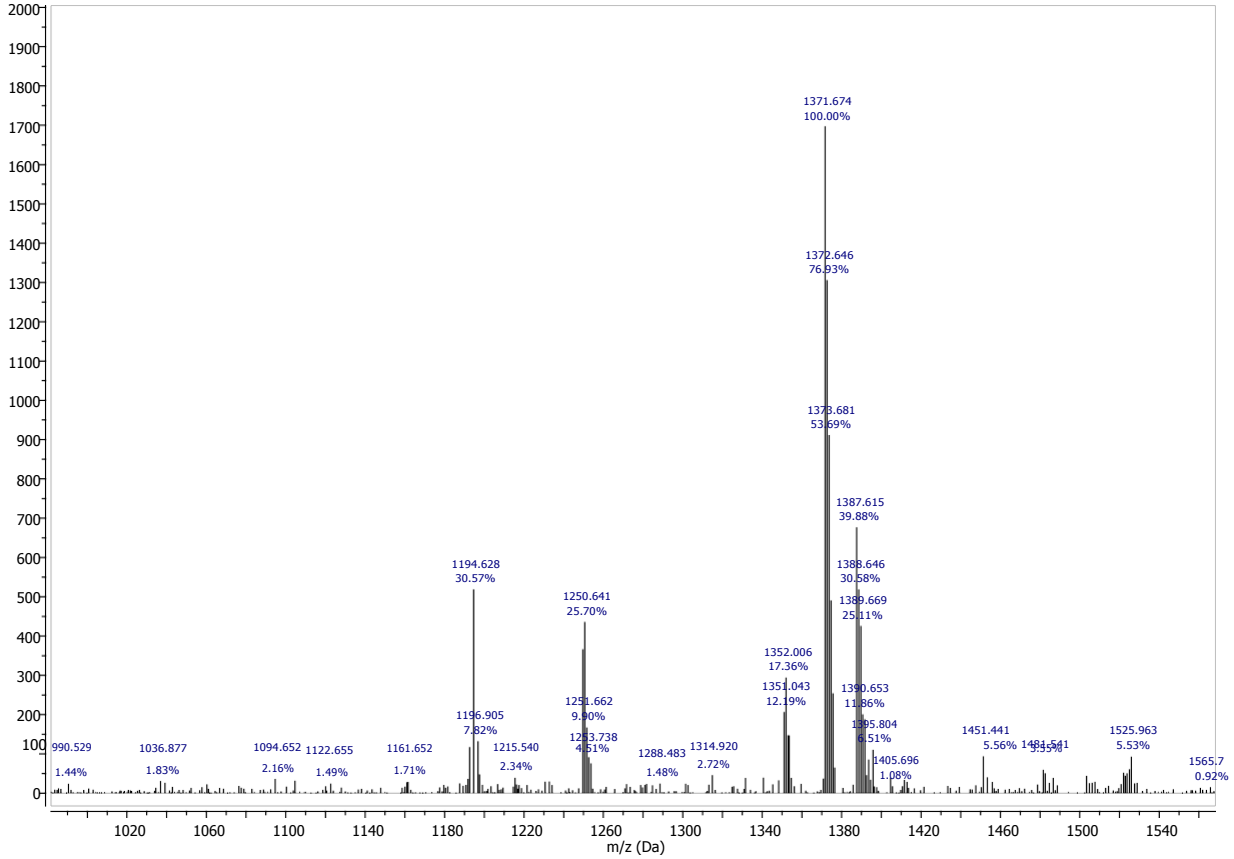


Fig. S22. Mass spectrum (MALDI TOF, 4-nitroaniline matrix) of *1,3-alternate*-5

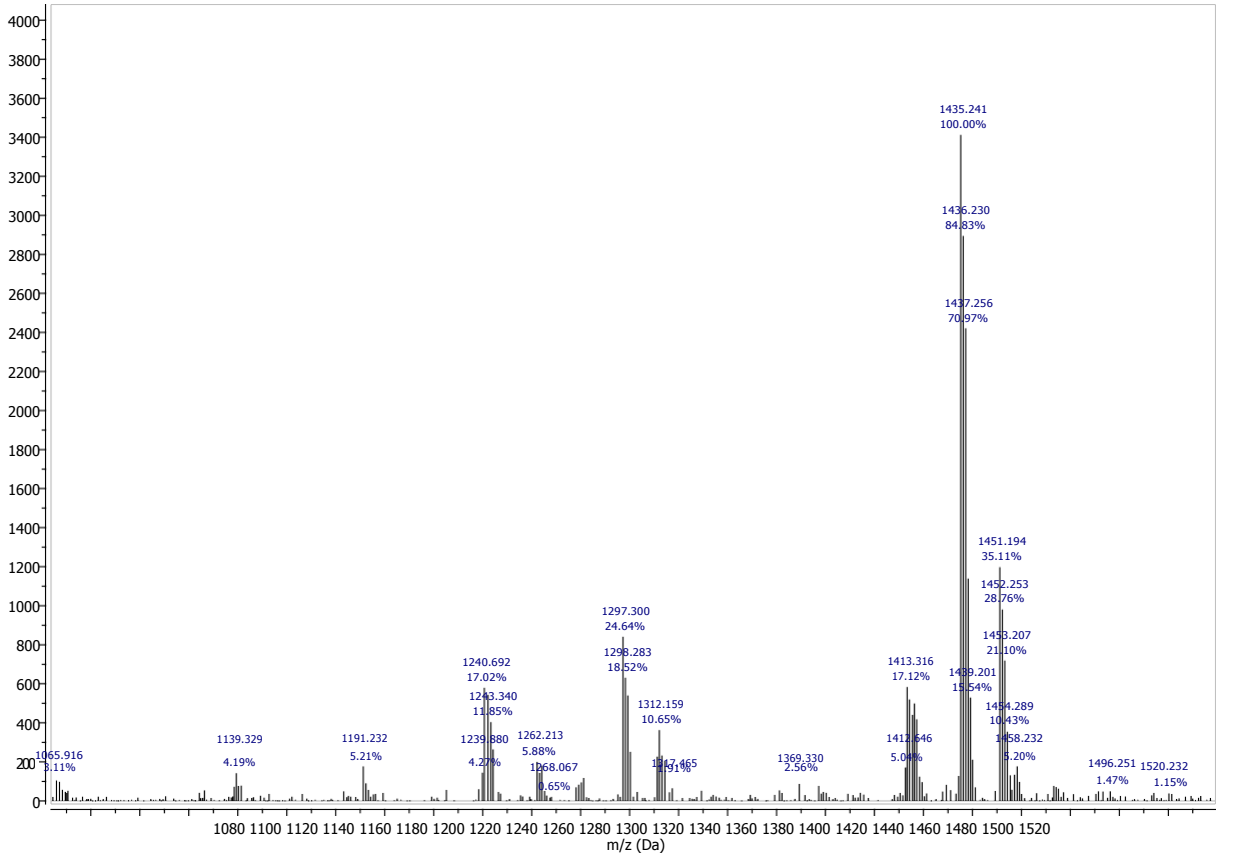


Fig. S23. Mass spectrum (ESI) of *1,3-alternate*-6

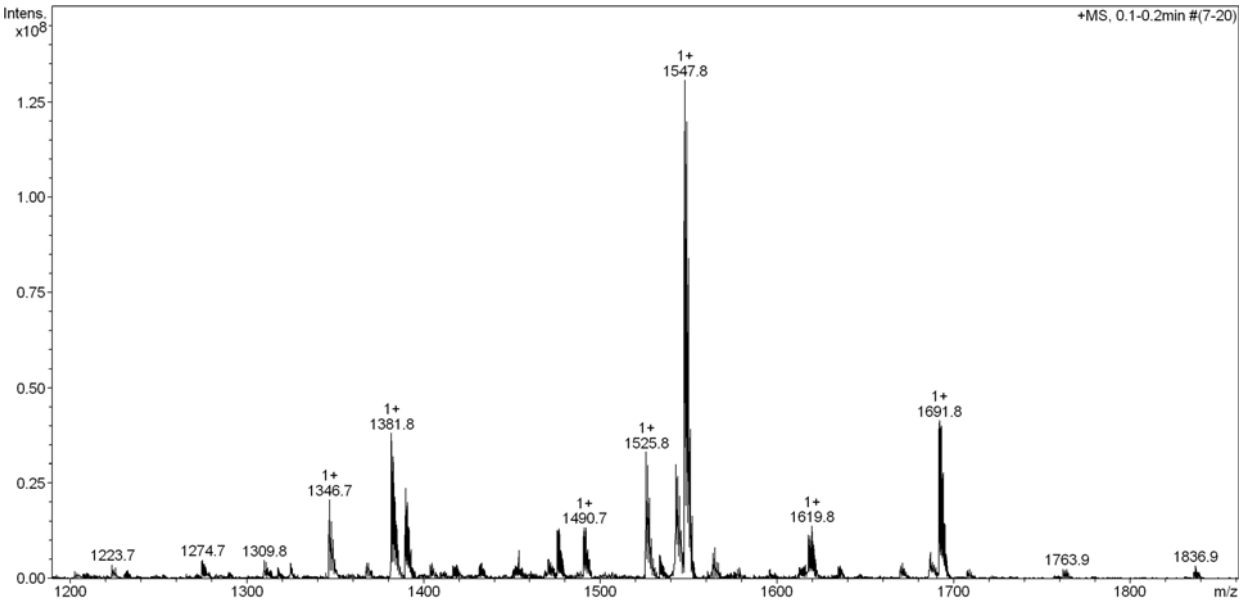


Fig. S24. IR spectrum of *1,3-alternate*-2

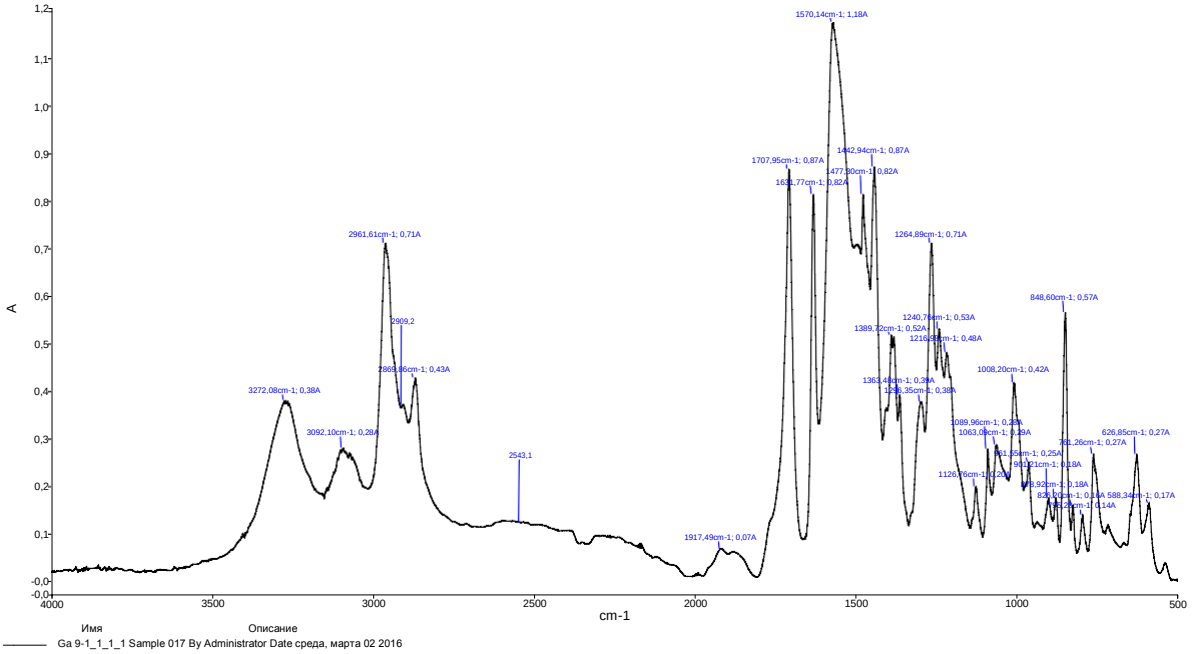


Fig. S25. IR spectrum of *1,3-alternate*-3

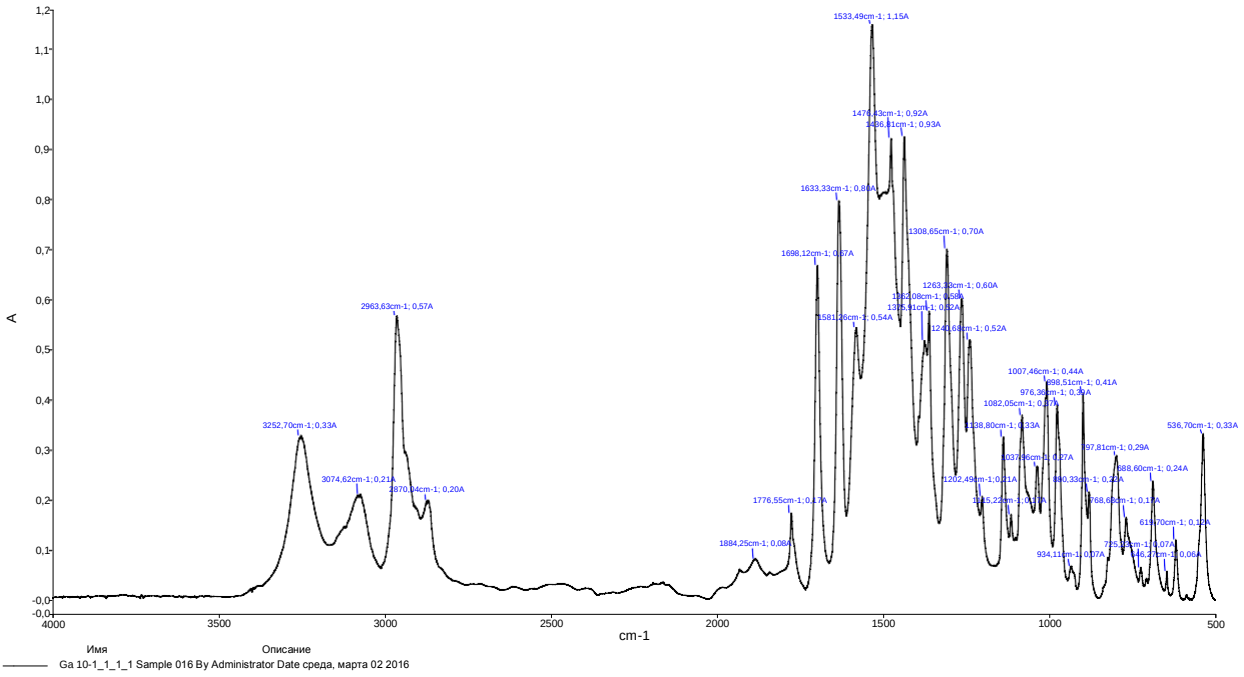


Fig. S26. IR spectrum of *1,3-alternate*-4

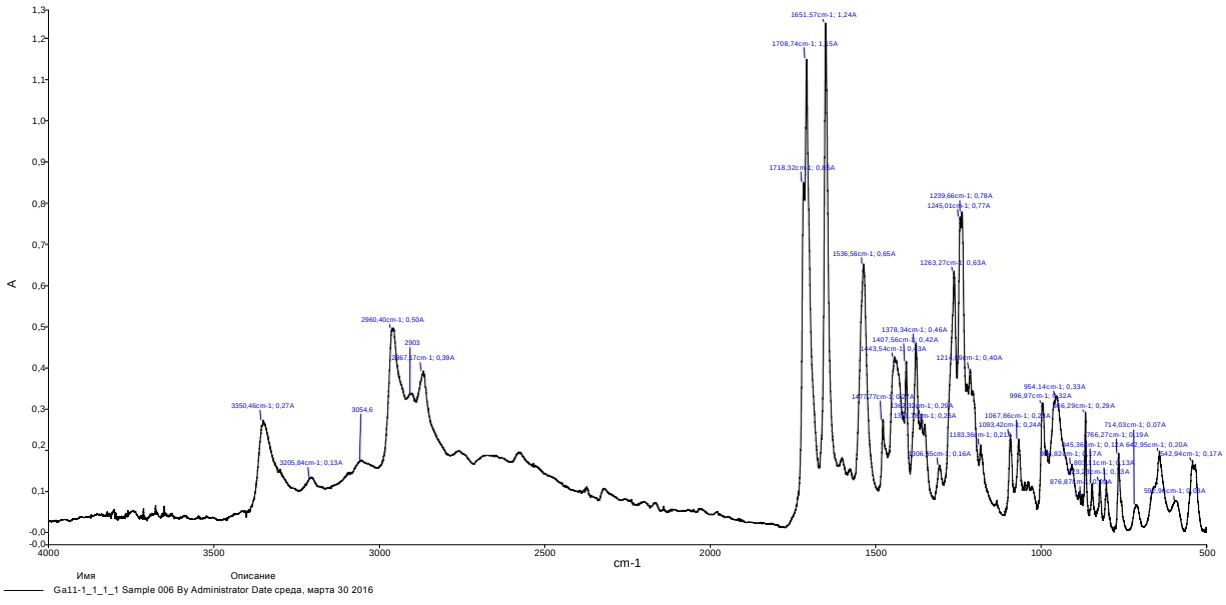


Fig. S27. IR spectrum of *1,3-alternate*-5

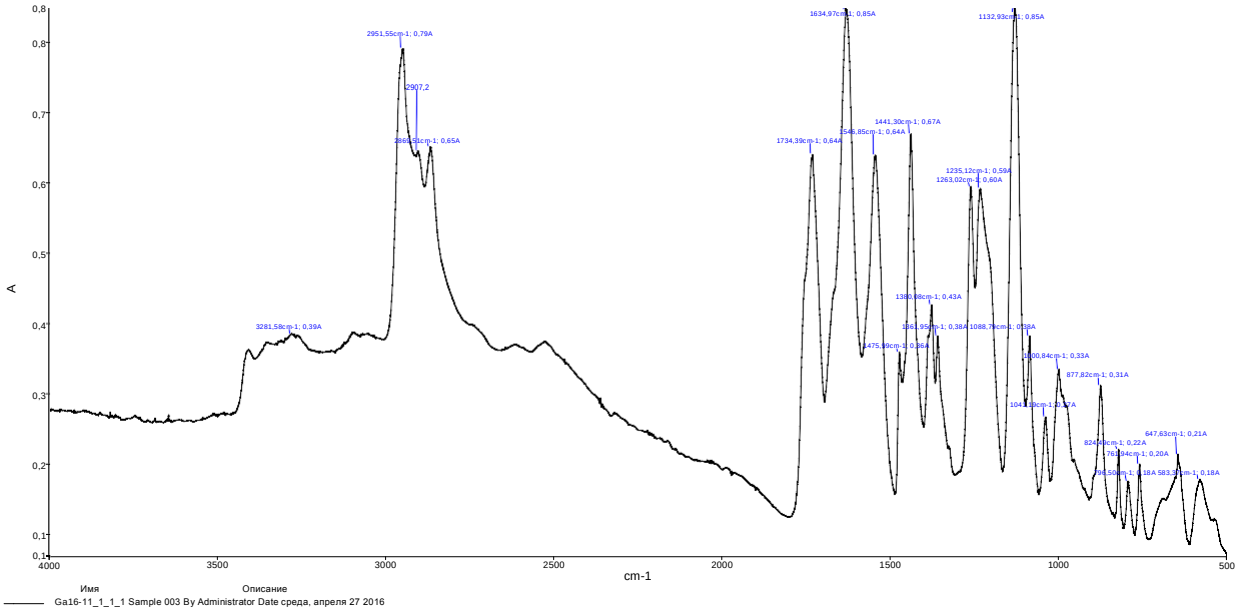


Fig. S28. IR spectrum of *1,3-alternate*-6

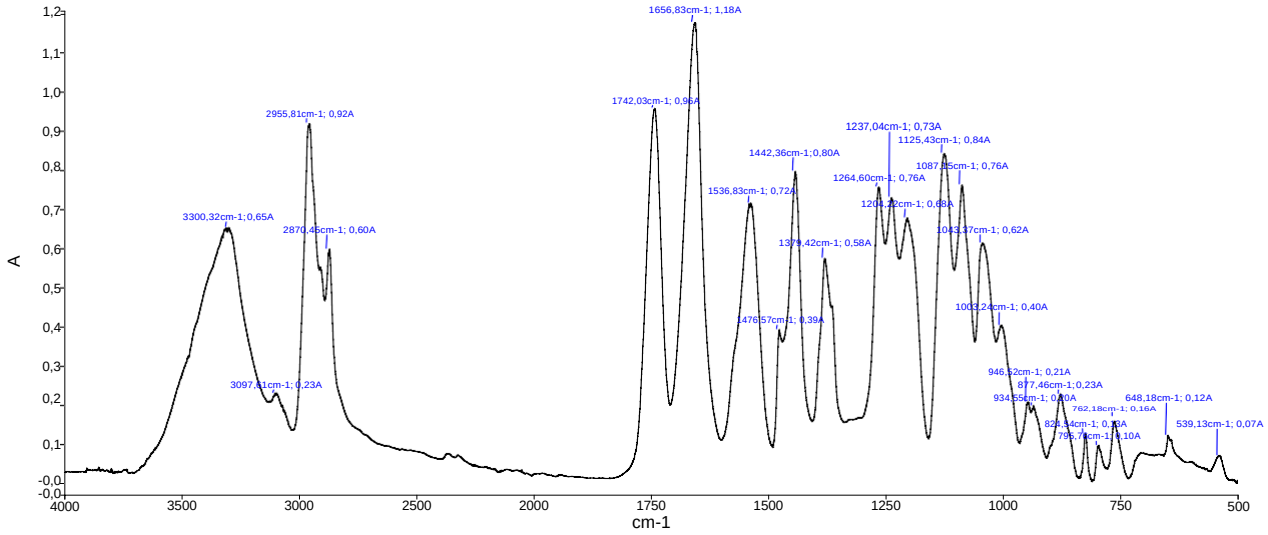


Fig. S29. CD spectra of the macrocycle 6 (2.5×10^{-4} M) and (3S)-*cis*-3,6-dimethyl-1,4-dioxane-2,5-dione (L-lactide) (10^{-3} M) in methanol at 293 K

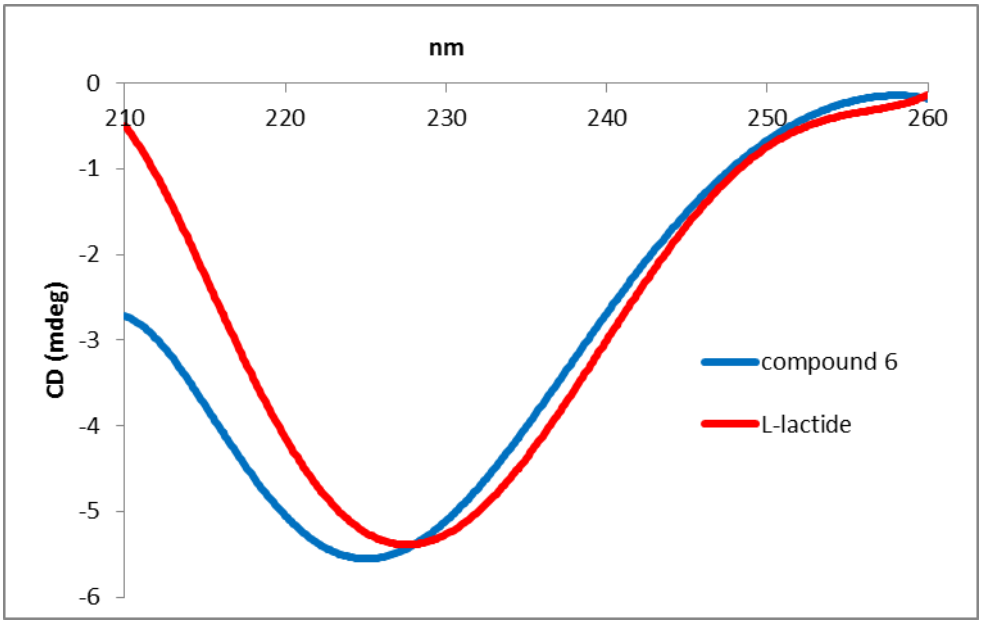
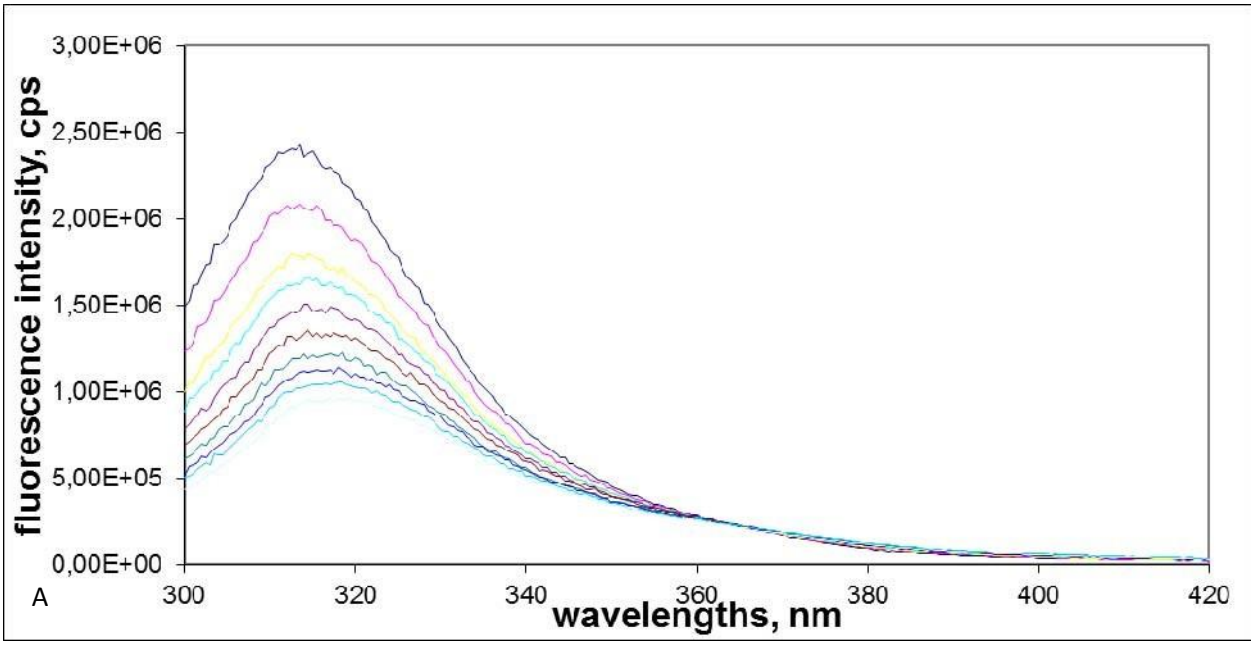
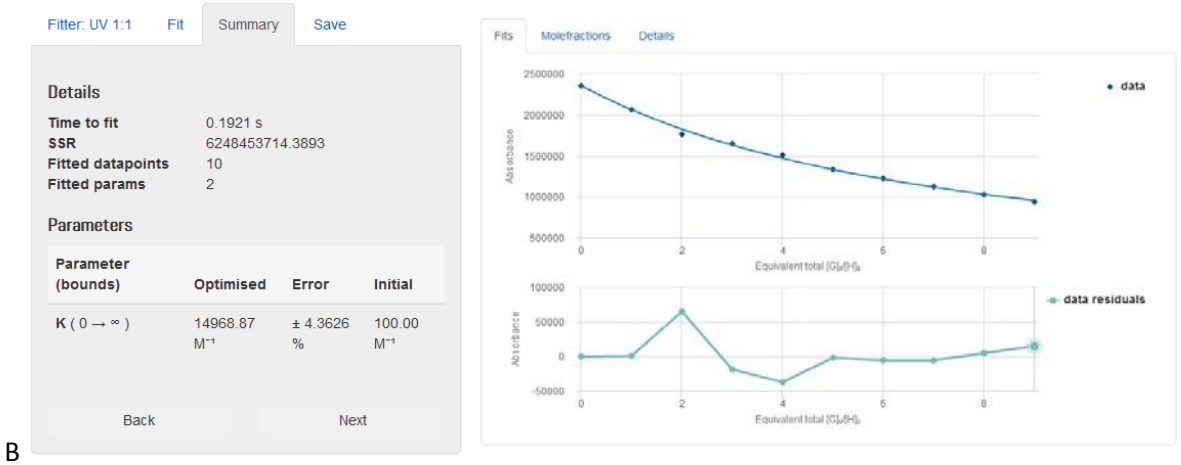


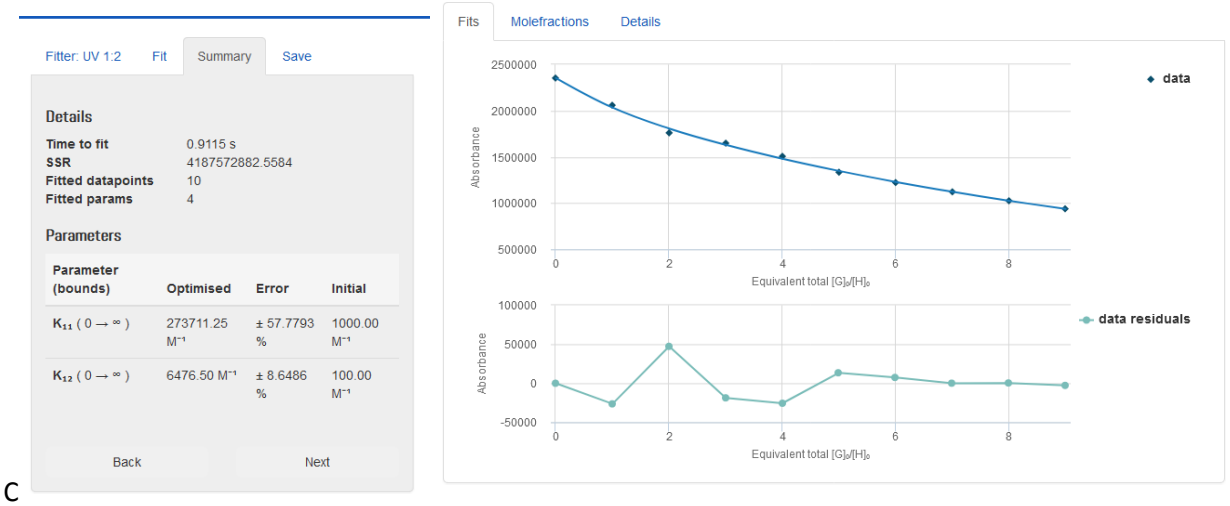
Fig. S30. A) Fluorescence spectra of dopamine-HCl (10 µM) at different concentrations of compound 5 (0-90 µM); B) and C) Screenshots taken from the summary window of the website supramolecular.org. This screenshots shows the raw data for titration of 5 with dopamine-HCl, the data fitted to 1:1 binding model (B) and 1:2 binding model (C).



A

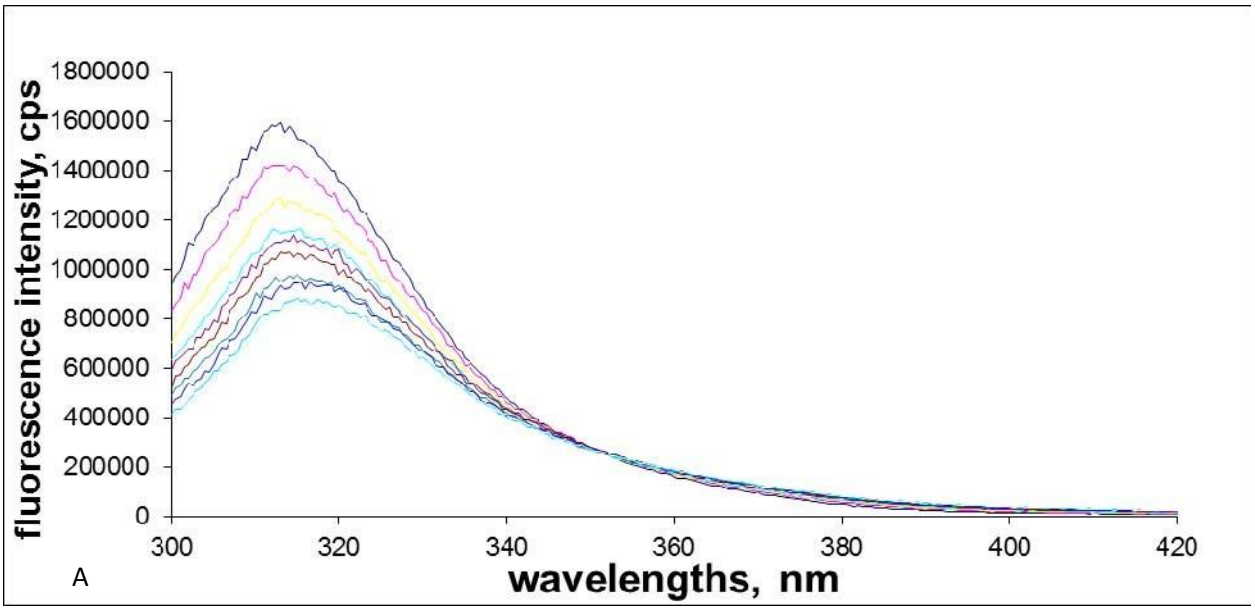


B

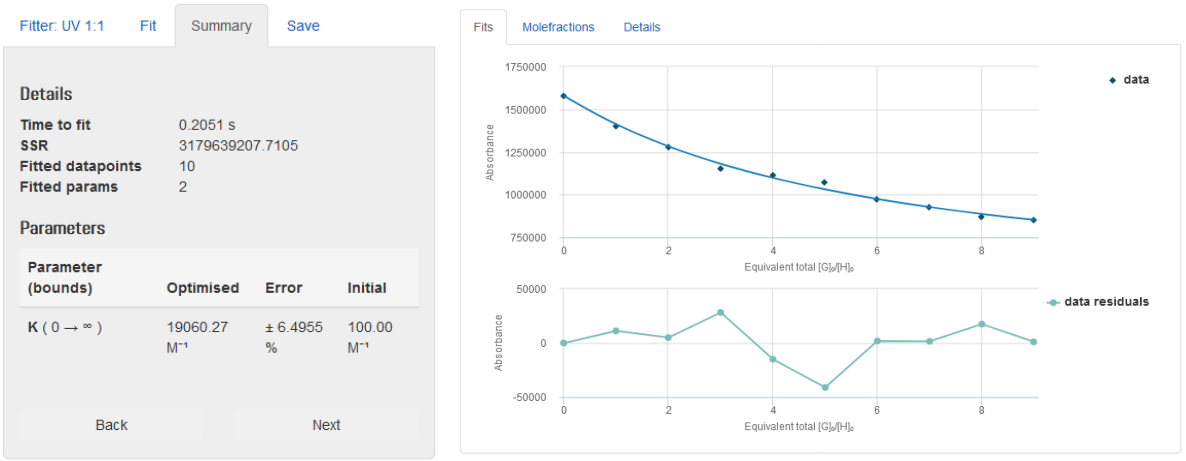


C

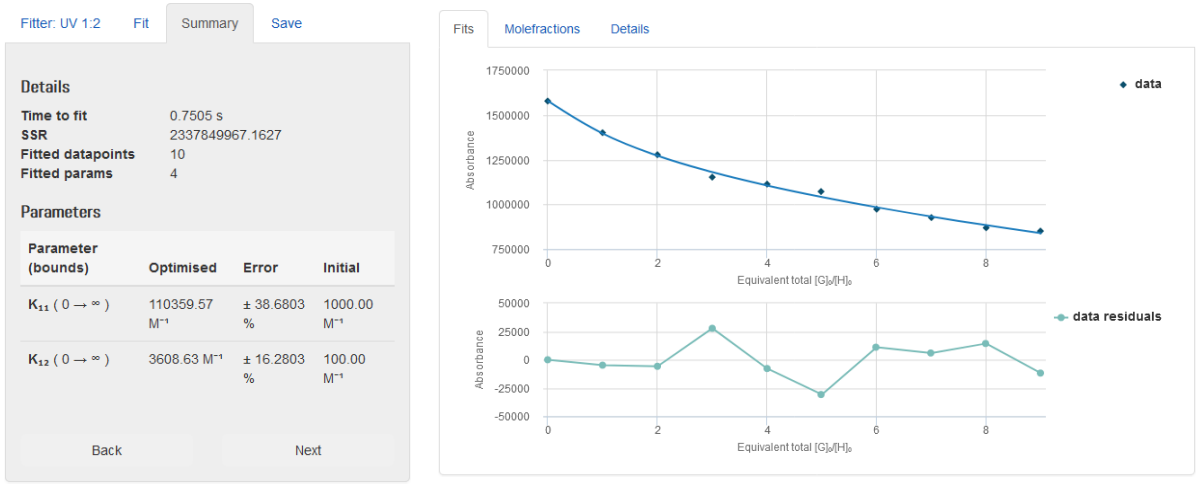
Fig. S31. A) Fluorescence spectra of dopamine-HCl (10 µM) at different concentrations of compound 6 (0-90 µM); B) and C) Screenshots taken from the summary window of the website supramolecular.org. This screenshots shows the raw data for titration of 6 with dopamine-HCl, the data fitted to 1:1 binding model (B) and 1:2 binding model (C).



A



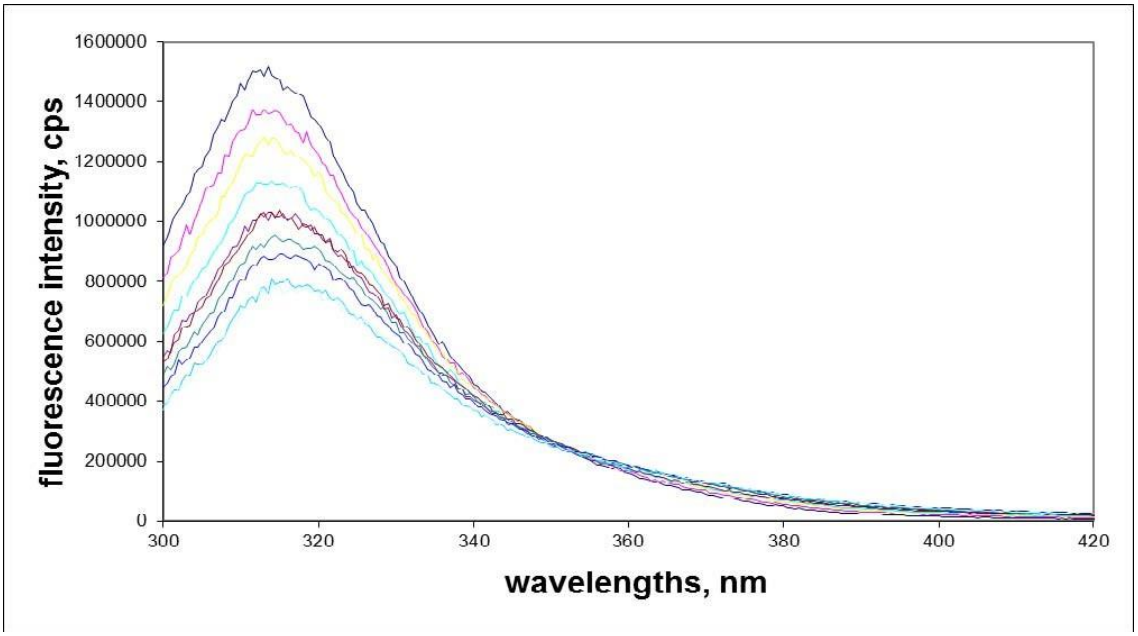
B



C

Fig. S32. A) Fluorescence spectra of dopamine-HCl (10 µM) at different concentrations of compound 4 (0-90 µM); B) and C) Screenshots taken from the summary window of the website supramolecular.org. This screenshots shows the raw data for titration of 4 with dopamine-HCl, the data fitted to 1:1 binding model (B) and 1:2 binding model (C).

A



B

Fitter: UV 1:1 | Fit | Summary | Save

Details

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SSR	4286816042.4627
Fitted datapoints	10
Fitted params	2

Parameters

Parameter (bounds)	Optimised	Error	Initial
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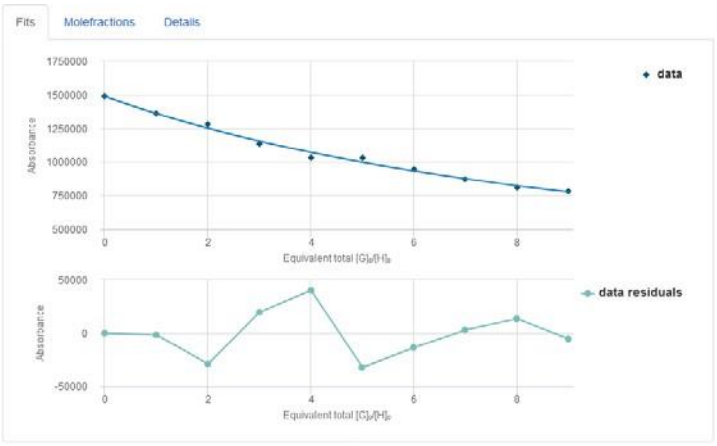
Back | Next



C

Fitter: UV 1:2 | Fit | Summary | Save

Details

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Fitted params	4

Parameters

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K_{12} (0 → ∞)	10483.88 M^{-1}	± 17.1923 %	100.00 M^{-1}

Back | Next

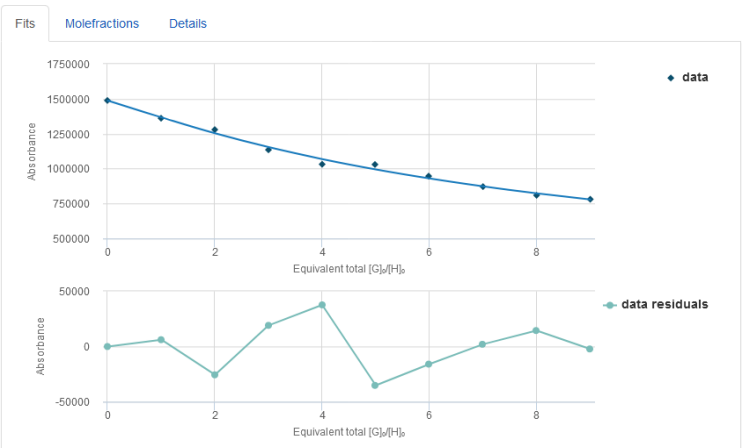
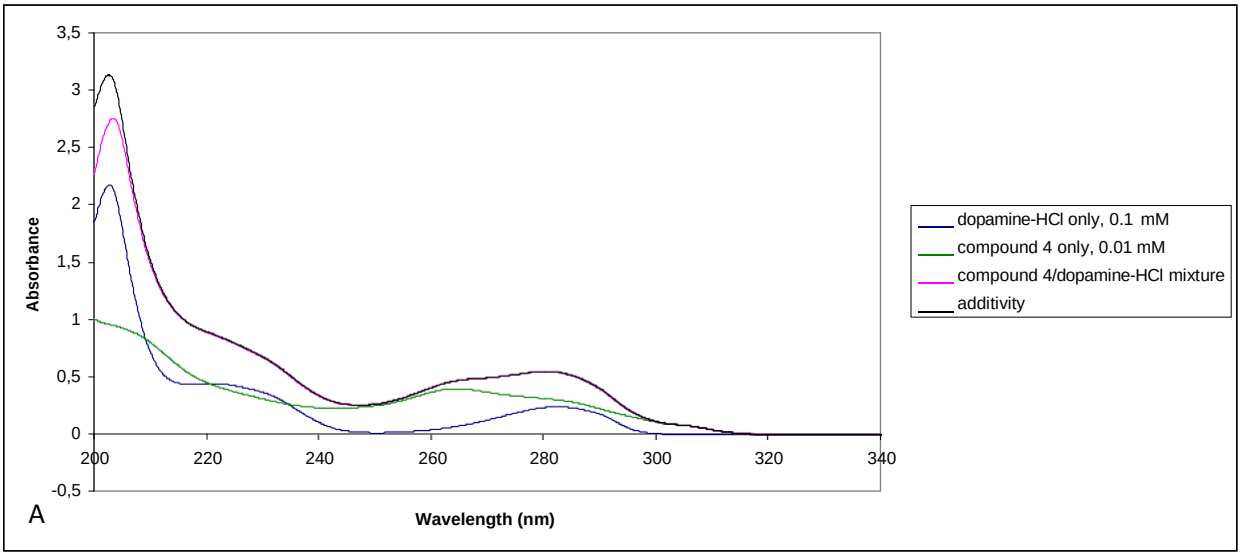


Fig. S33. UV/vis absorption spectra of the macrocycles 4 (A) and 6 (B) with dopamine-HCl mixtures (1:10 molar ratio) and additive spectra of individual components in the concentration studied (293 K)



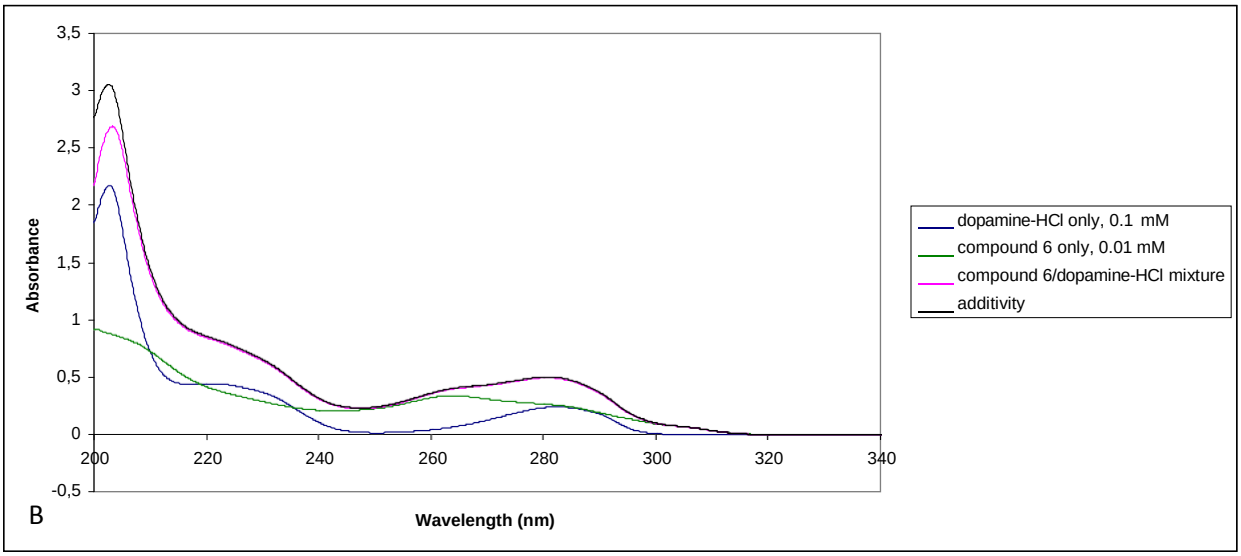
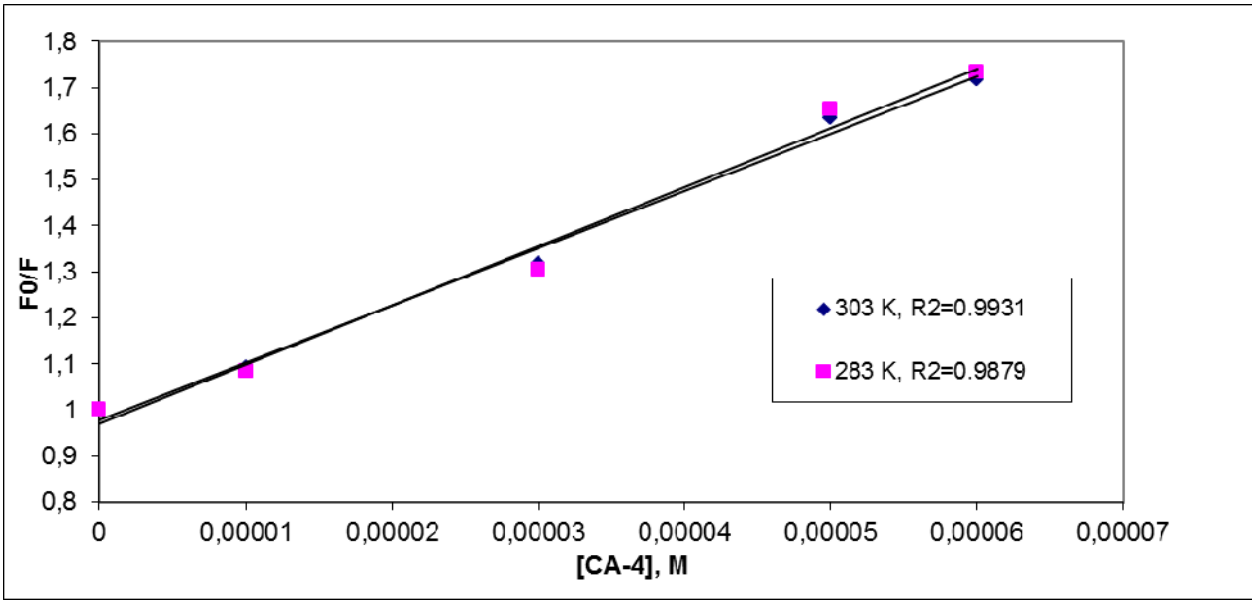
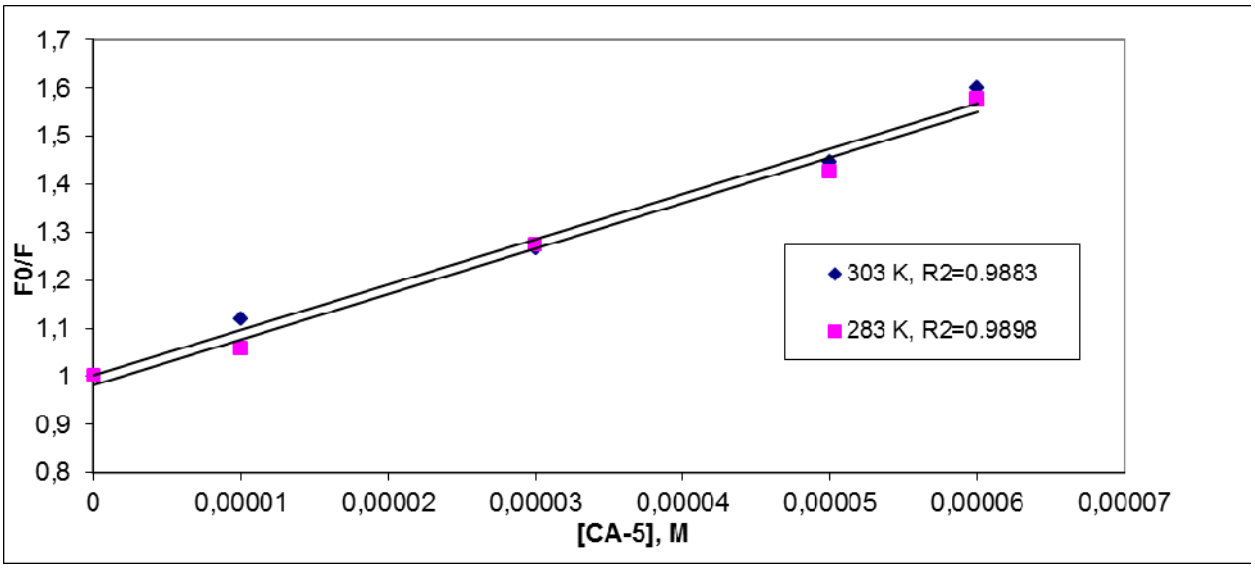


Fig. S34. The plots of F_0/F vs. [CA] at different temperatures (283 and 303 K). λ_{ex}=285 nm; λ_{em}=315 nm; [Dop-HCl]=10 μM





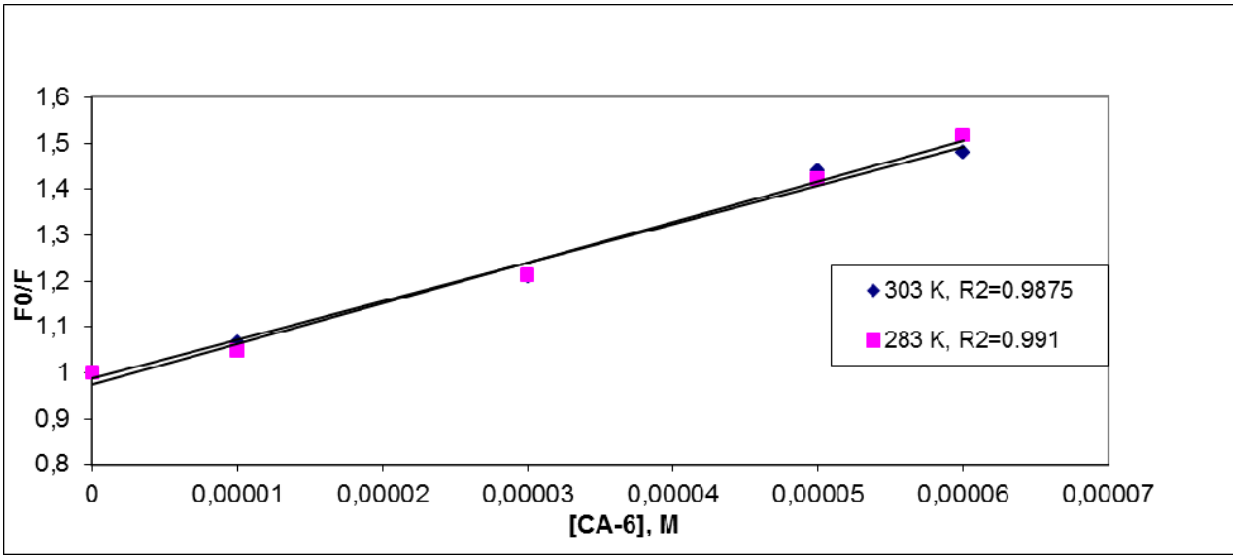
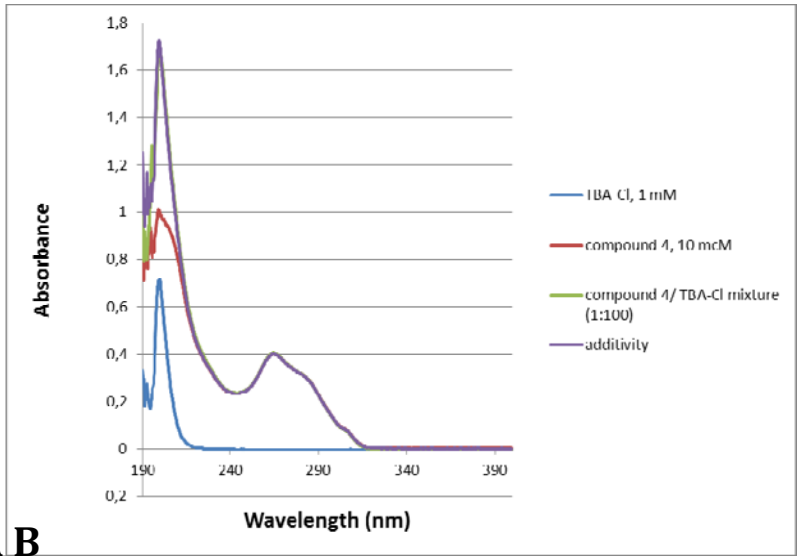
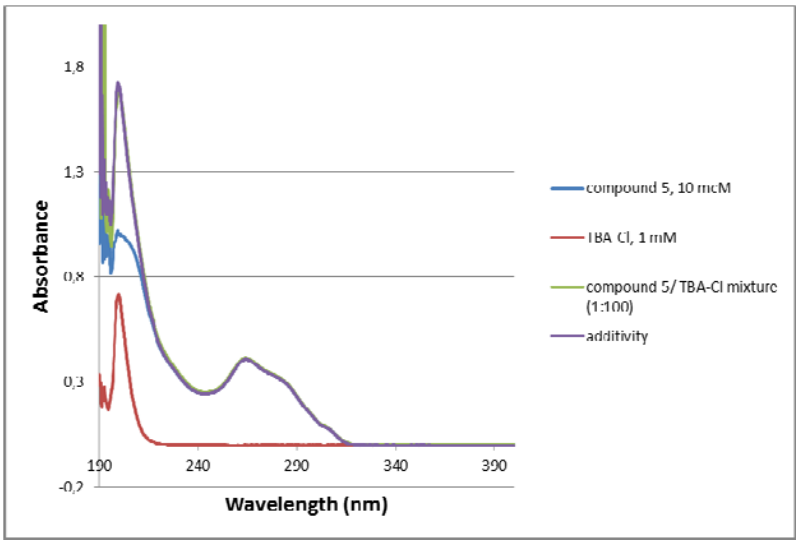


Fig. S35. UV/vis absorption spectra of the macrocycles 4 (A), 5 (B) and 6 (C) with tetrabutylammonium chloride mixtures (10:1 molar ratio) and additive spectra of individual components in the concentration studied (293 K)



A B



C

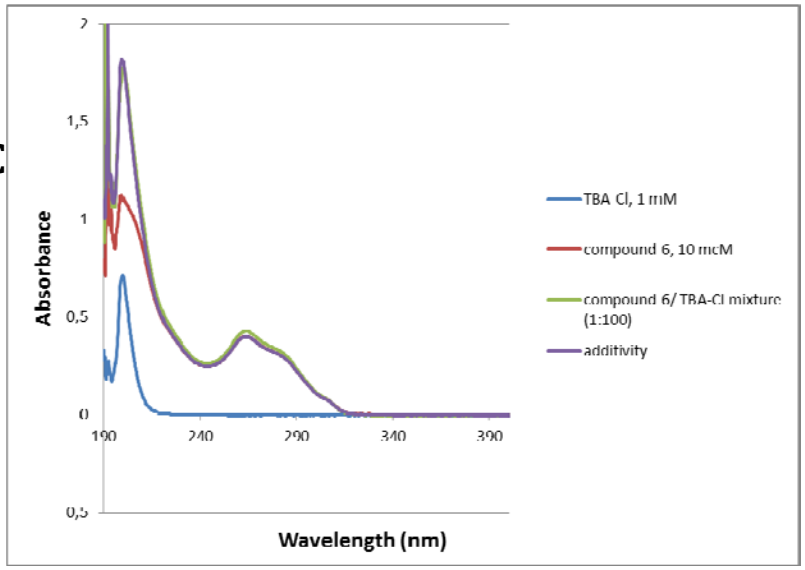


Fig. S36. ^{1}H NMR spectra for the Dop-HCl (1), the compound 6 (3), and system Dop-HCl-6 (2) (CD_3OD, 298 K, Bruker Avance-400, 400 MHz)

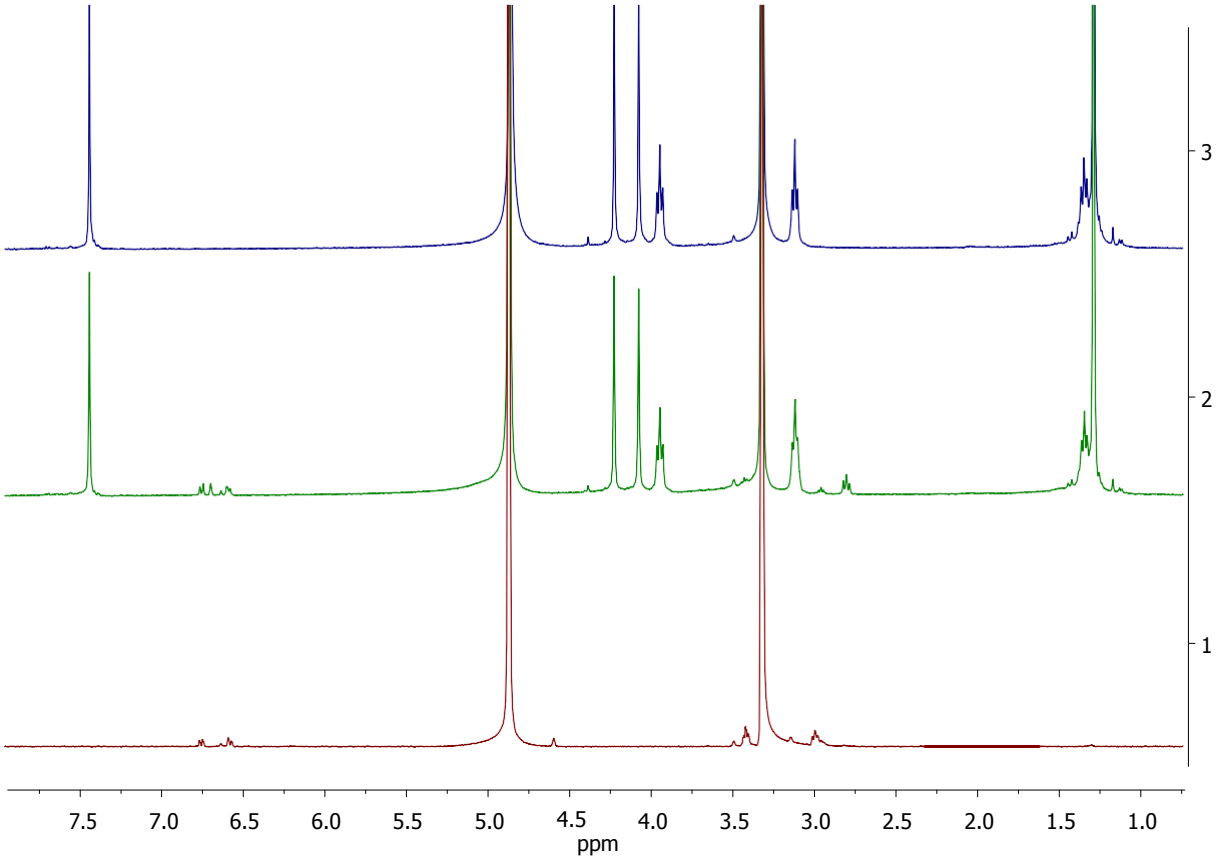


Fig. S37. ^{1}H NMR (DOSY) spectrum for the system Dop-HCl-6 (CD_3OD, 298 K, Bruker Avance-400, 400 MHz)