SUPPORTING INFORMATION

Homo and hetero-[3]rotaxanes with two π-systems clasped in a single macrocycle

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Section 1: Synthetic Procedures

Materials and Methods. All reagents were purchased from commercial sources. All solvents were used as purchased with the exception of DMSO, which was distilled from CaH_2 prior to use. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker DPX200, DPX400, DRX500 and AV500 with 13C{1H} cryoprobe. Low and high resolution ESI-MS were carried out on a Fisons platform and Micromass LCT platform, respectively. UV-vis spectra were performed on a Perkin Elmer Lamda-6. Fluorescence data were recorded with a Fluoromax-2 fluorimeter (HORIBA Jobin Yvon Ltd).

HPLC Methods. HPLC analyses were carried out on an HP 1100 series workstation using a Zorbax Eclipse XDB C8 analytical column (4.6 x 15 mm, 5 micron). The analytes were eluted using a timetabled two-solvent gradient system and monitored using a diode array detector using 'Method 1', as shown in Figure S1. [Solvent A = 0.25% w/v dicyclohexylammonium phosphate (CHAPS) in ultra-pure water, Solvent B = MeOH]. Semipreparative HPLC was carried out using a Zorbax Eclipse XDB C8 semi-preparative column (9.4 x 250 mm, 5 micron) using 'Method 2' (shown in Figure S1). [Solvent A = 0.25% w/v dicyclohexylammonium phosphate (CHAPS) in ultra-pure water, Solvent B = MeCN]

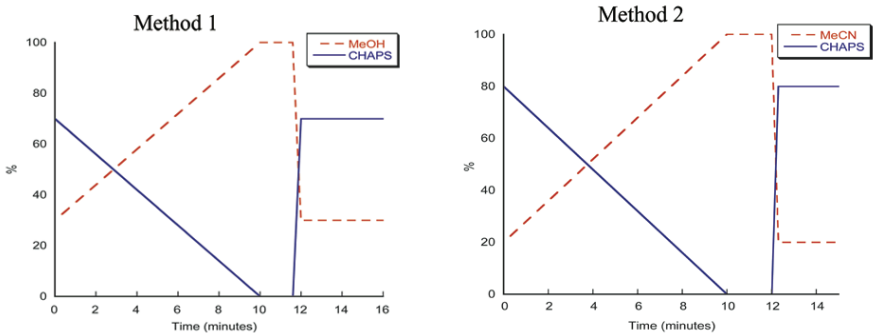
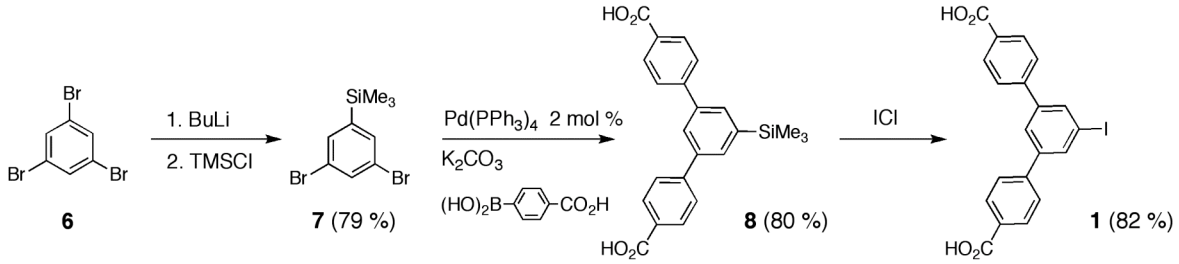


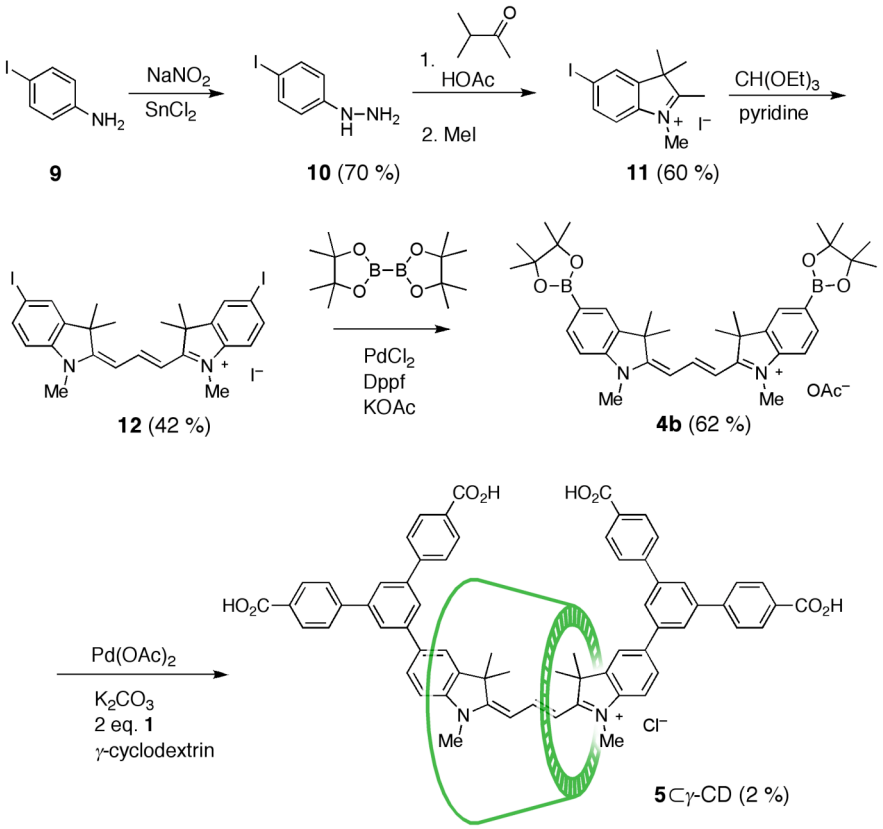
Figure S1. HPLC gradients for reaction analysis. 'Method 1' uses a flow rate of 1 ml min^{-1}, while 'Method 2' uses a flow rate of 4 ml min^{-1}.



Scheme S1. Synthetic route for aryl iodide stopper **1**.

Synthesis of 8. *N,N*-Dimethylformamide (250 cm^3) was added to a round-bottomed flask containing 3,5-dibromo-1-(trimethylsilyl)benzene[1] **7** (2.41 g, 7.81 mmol), tetrakis(triphenylphosphine)palladium(0) (0.86 g, 0.74 mmol), and 4-carboxyphenylboronic acid (3.14 g, 18.90 mmol), the mixture was deoxygenated and nitrogen saturated. Deoxygenated, nitrogen saturated potassium carbonate (aq., 3.2 M, 50 cm^3) was added and the solution stirred at 80 °C overnight. The reaction mixture was allowed to cool then acidified [CARE! EFFERVESCENCE!] with hydrochloric acid (2.0 M, 50 cm^3), to pH 1. The resultant precipitate was collected by centrifugation and dissolved in NaOH (2.0 M aq., 25 cm^3), then filtered. The solution was acidified with HCl (2.0 M aq., 25 cm^3) and the precipitate collected by centrifuge, washed with water and dried under vacuum to yield an off-white powder. Recrystallisation from toluene gave **8** (2.43 g, 80%) as a white powder; m.p. 298–299 °C; λ_{max} (MeOH) 273 nm (ε = 31,000); δ_H (400 MHz, d_6-DMSO) 0.36 (9 H, s, Si(C*H*$_3$)$_3$), 7.85 (s, 2H), 7.92 (d, *J* = 8.5 Hz, 4H) 7.99 (s, 1H) 8.05 (d, *J* = 8.5 Hz, 4H), 12.99 (2 H, br s, CO$_2$*H*); δ_C(400 MHz, d_6-DMSO) 1.1, 126.5, 127.3, 129.8, 129.9, 131.6, 139.3, 141.9, 144.4, 167.1; *m/z* (ESI-) 389.3 ([M-H]$^-$, 24%) 194.2 ([M-2H]$^{2-}$, 100%); HRMS (ESI-): calculated for $C_{23}H_{21}O_4Si_4$ 389.1208, observed 389.1204.

Synthesis of 1. Iodine monochloride (5.5 g, 34 mmol) was warmed to a liquid and transferred by pipette into a pear-shaped flask over ice. *N,N*-Dimethylformamide (5 cm^3) was added and the solution stirred for 5 min. 3,5-Bis(4-carboxyphenyl)-1-(trimethylsilyl)benzene **8** (2.4 g, 6.2 mmol) was added. The mixture was allowed to warm to room temperature and stirred for 64 h, after which time analysis by HPLC (Method 1) showed the reaction to be complete. The mixture was poured into CH_2Cl_2 (20 cm^3), and the white solid was collected by suction filtration and dried to yield **1** as a light yellow powder (2.2 g, 82%); m.p. >250 °C (dec.); λ_{max} (phosphate buffer pH 11.4) 273 nm (ε = 43,800); δ_H (400 MHz, d_6-DMSO) 7.92 (d, *J* = 8.5 Hz, 2H), 8.01–8.06 (5 H, m), 8.09 (s, 2H); δ_C (400 MHz, d_6-DMSO) 96.5, 125.3, 127.3, 129.9, 130.3, 135.1, 141.9, 142.5, 167.1; *m/z* (ESI-) 221 ([M-2H]$^{2-}$ 100%), 443 ([M-H]$^-$ 50%); HRMS (ESI-): calculated for $C_{20}H_{12}IO_4$ 442.9775, observed 442.9778.



Scheme S2. Synthesis of **4b** and **5**⊂γ-CD.

Synthesis of 11. This compound was prepared by adaptation of a published procedure.[2] A mixture of *para*-iodo-phenyl hydrazine (3.5 g, 15 mmol), acetic acid (5.1 cm^3, 90 mmol) and *iso*-propylmethyl ketone (1.6 cm^3, 15 mmol) was refluxed under N_2 for 2.5 hrs. The solution was then diluted with H_2O (20 cm^3) and $NaHCO_3$ was added to bring the pH to 7. The indole was then extracted with diethylether to yield a brown oil (3.69 g, 12.99 mmol, 86% crude). This oil was dissolved in methanol (10 cm^3) and iodomethane (1.77 cm^3, 28 mmol) and heated in a sealed tube at 110 °C for 6 hrs. The resulting precipitate was filtered, washed with chloroform and recrystallized using DMSO/$CHCl_3$ to yield **11** as a red solid (2.33 g, 60%); m.p. > 250 °C; ^{1}H NMR (200 MHz, d_6-DMSO) δ 1.54 (s, 6H), 2.75 (s, 3H), 3.95 (s, 3H), 7.74 (d, J = 8.5 Hz, 1H), 8.05 (d, J = 8.5 Hz, 1H), 8.10 (s, 1H); ^{13}C NMR (100 MHz, d_6-DMSO) δ 15.0, 22.3, 35.6, 54.9, 96.9, 118.0, 133.1, 138.3, 142.8, 144.6, 196.9; HRMS (ESI+): calculated for $C_{12}H_{15}IN$ 300.0249, observed 300.0243.

Synthesis of 12. This compound was prepared by adaptation of a published procedure.[3] To a solution of **11** (500 mg, 1.17 mmol) in pyridine (1.5 cm^3) was added triethylorthoformate (584 µl, 3.5 mmol). The mixture was refluxed under N_2 for 3 hrs and the resulting green precipitate was filtered and washed with diethyl ether (30 cm^3). The crude cyanine dye was then recrystallized using hot methanol to yield **12** as green crystals (720 mg, 42%); m.p. > 250 °C; λ_{max} (MeOH) 558 nm (ε = 690,000); ^{1}H NMR (400 MHz, d_6-DMSO) δ 1.67 (s, 12H), 3.60 (s, 6H), 6.45 (d, J = 13.5 Hz, 2H), 7.30 (d, J = 7.5 Hz, 2H), 7.80 (d, J = 7.5 Hz, 2H), 8.04 (s, 2H), 8.29 (t, J = 13.5 Hz, 1H); ^{13}C NMR (100 MHz, d_6-DMSO) δ 27.9, 32.4, 49.7, 90.5, 104.0, 114.6, 131.9, 138.0, 143.4, 143.8, 150.0, 174.6; HRMS (ESI+): calculated for $C_{25}H_{27}N_2I_2$ 609.0269, observed 609.0264.

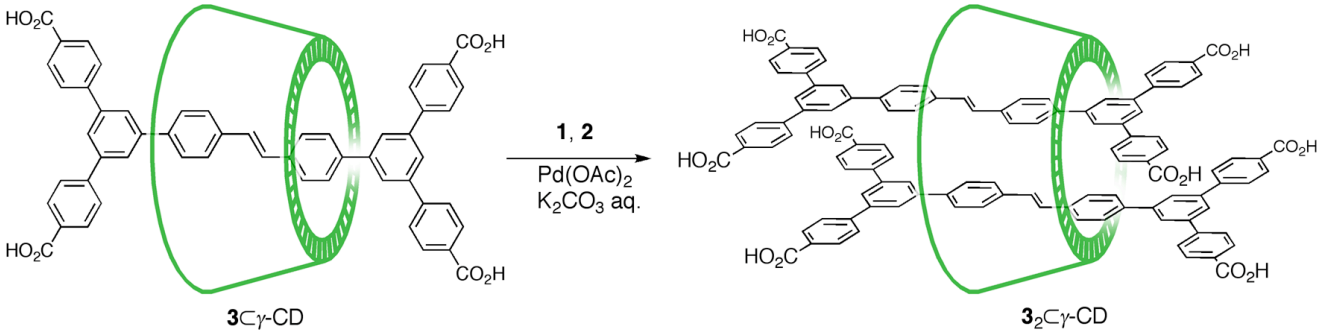
Synthesis of 4b. This was synthesised by adaptation of a reported procedure.[4] A flask was charged with **12** (200 mg, 0.272 mmol), bis(pinacolato)diboron (300 mg, 1.1 mmol), $PdCl_2$ (5 mg, 27 µmol), 1,1'-bis(diphenylphosphino)ferrocene (20 mg, 30 µmol) and anhydrous KOAc (160 mg, 1.6 mmol). To this was added dry, degassed DMF (6.0 cm^3). The solution was stirred under nitrogen at 80 °C for 3 hrs. The reaction mixture was poured into water and extracted with CH_2Cl_2. The solvent was removed *in vacuo* and the crude solid was purified by column chromatography (1-propanol:ethyl acetate:water:acetic acid, 40:31:28:1 volume ratio) to

yield **4b** as red solid (120 mg, 62%). m.p. > 250 °C; λ_{max} (MeOH) 556 nm (ε = 85,400); ^{1}H NMR (400 MHz, d_6-DMSO) δ 1.31 (s, 24H), 1.59 (s, 12H), 3.66 (s, 6H), 6.50 (d, 2H, J = 13.5 Hz), 7.49 (d, J = 8.0 Hz, 2H), 7.76 (d, J = 8.0 Hz, 2H), 7.85 (s, 2H), 8.37 (t, J = 13.0 Hz, 1H); ^{13}C NMR (100 MHz, d_6-DMSO) δ 24.8, 28.0, 32.8, 48.4, 84.1, 105.4, 110.1, 125.0, 127.8, 136.0, 139.7, 145.0, 151.0, 174.8; HRMS (ESI+): calculated for $C_{37}H_{51}B_2N_2O_4$ 609.4035, observed 609.4052.

Synthesis of stilbene [2]rotaxane 3⊂γ-CD. Degassed water (10 cm^3) was added to a mixture of **2** (36 mg, 0.11 mmol), iodoterphenylene dicarboxylic acid **1** (100 mg, 0.23 mmol), γ-cyclodextrin (870 mg, 0.67 mmol), potassium carbonate (300 mg, 2.2 mmol). The mixture was heated to 45 °C and palladium(II) acetate (25 µl, 1.1 µmol) was added from a stock solution (45 mM in DMSO). The mixture was stirred under nitrogen at 45 °C for 6 hrs. The mixture was then allowed to cool, diluted with deionized water (15 cm^3), and filtered through paper. HCl (25 cm^3, 1 M) was added and the resulting suspension was centrifuged. The supernatant was removed and the solid was washed twice with water (40 cm^3). The solid was subsequently taken up into DMSO (2 cm^3) and reprecipitated with MeCN (40 cm^3). This process was repeated four times. The solid was subsequently washed with 20 cm^3 MeCN, dried *in vacuo* to yield the rotaxane, **3**⊂γ-CD as a white solid (conversion: 22% by HPLC; yield: 40 mg, 19 µmol, 17%). λ_{max} (phosphate buffer pH 11.4) 355 nm (ε = 44,000), 270 nm (ε = 55,000); ^{1}H NMR (500 MHz, d_6-DMSO) δ 3.29–3.39 (m, 16H), 3.52–3.67 (m, 32H), 4.51 (t, J = 5.0 Hz, 8H), 4.87 (d, J = 3.0 Hz, 8H), 5.67 (d, J = 1.0 Hz, 8H), 5.74 (d, J = 7.0 Hz, 8H), 7.18 (d, J = 16.0 Hz, 1H), 7.23 (d, J = 16.0 Hz, 1H), 7.69 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.0 Hz, 2H), 7.93 (d, J = 8.0 Hz, 2H), 7.95 (d, J = 8.0 Hz, 2H), 8.05–8.14 (m, 22H); *m/z* (ESI-) 526 $[M]^{4-}$(100%).

Synthesis of cyanine [2]rotaxane 5⊂γ-CD. Degassed water (4 cm^3) was added to a mixture of **4b** (100 mg, 0.15 mmol), iodoterphenylene dicarboxylic acid **1** (133 mg, 0.30 mmol), γ-cyclodextrin (3.20 g, 2.5 mmol), potassium carbonate (240 mg, 1.70 mmol). The mixture was heated to 45 °C and palladium(II) acetate (36 µl, 1.6 µmol) was added from a stock solution (45 mM in DMSO). The mixture was stirred under nitrogen at 45 °C for 6 hrs. The mixture was then allowed to cool, diluted 15 cm^3 with deionized water, and filtered through paper. HCl (15 cm^3, 1.0 M) was added and the resulting suspension was centrifuged. The supernatant was removed and the solid was washed twice with water (20 cm^3). The solid was subsequently washed four times with acetone (20 cm^3) and dried. The crude material was dissolved in DMSO (1 cm^3) and purified by semipreparative HPLC using 'Method 2' (See Fig. S1). The fractions were collected, concentrated *in vacuo*. The solid was collected and washed three times with 20 cm^3 water and dried to yield the rotaxane **5**⊂γ-CD as a red solid (conversion: 11% by HPLC; yield: 6 mg, 2%). λ_{max} (phosphate buffer pH 11.4) 573 nm, 270 nm; ^{1}H NMR (500 MHz, d_6-DMSO) δ 1.78 (s, 3H), 1.80 (s, 3H), 1.90 (s, 3H), 1.92 (s, 3H), 3.30–3.70 (m, 51H), 3.82 (s, 3H), 4.90 (d, J = 2.5 Hz, 8H), 6.25 (d, J = 14.0 Hz, 1H), 6.43 (d, J = 14.0 Hz, 1H), 7.49 (d, J = 7.5 Hz, 1H), 7.69 (d, J = 8.5 Hz, 1H), 7.80 (s, 1H), 7.97 (d, J = 8.5 Hz, 1H), 8.03–8.14 (m, 24H), 8.48 (t, J = 14.0 Hz, 1H); *m/z* (ESI-) 571 $[M]^{4-}$(75%), 761 $[M]^{3-}$(100%), 1142 $[M]^{2-}$(40%).

Synthesis of stilbene-cyanine [3]rotaxane (3·5)⊂γ-CD. Degassed water (2 cm^3) was added to a mixture of **4b** (64 mg, 0.096 mmol), iodoterphenylene dicarboxylic acid (**1**) (85 mg, 0.19 mmol), [2]rotaxane **3**⊂γ-CD (83 mg, 39 µmol), potassium carbonate (317 mg, 2.3 mmol). The mixture was heated to 45 °C and palladium(II) acetate (22 µl, 0.96 µmol) was added from a stock solution (45 mM in DMSO). The mixture was stirred under nitrogen at 45 °C for 6 hrs. The mixture was then allowed to cool, diluted 15 cm^3 with deionized water and HCl (15 cm^3, 1.0 M) was added until effervescence ceased and the resulting suspension was centrifuged. The supernatant was removed and the solid was washed twice with 20 cm^3 water and dried *in vacuo*. The crude solid was dissolved in 1 cm^3 DMSO and purified by semipreparative HPLC using 'Method 2' (See Fig. S1). The fractions were collected, concentrated *in vacuo* and washed three times with 20 cm^3 water. The solid was dried in *vacuo* to yield the rotaxane, (**3·5**)⊂γ-CD as a blue solid (22 mg, 18%; the reaction proceeds quantitatively by HPLC, and this yield reflects losses during isolation). λ_{max} (phosphate buffer pH 11.4) 583 nm (ε = 79,000), 359 nm (ε = 48,0000), 269 nm (ε = 115,000); ^{1}H NMR (500 MHz, d_6-DMSO) δ 1.66 (s, 3H), 1.69 (s, 3H), 1.71 (s, 3H), 1.73 (s, 3H), 3.26 (s, 3H), 3.33–3.45 (m, 24H), 3.54–3.62 (m, 16H), 3.66–3.70 (m, 8H), 4.88 (d, J = 3.0 Hz, 8H), 5.81 (d, J = 13.5 Hz, 1H), 5.87 (d, J = 13.5 Hz, 1H), 6.68 (d, J = 16.0 Hz, 1H), 6.8 (d, J = 16.0 Hz, 1H), 7.25–8.13 (m, 62H); *m/z* (ESI-) 773 $[M]^{4-}$(100%), 1032 $[M]^{3-}$(50%).



Scheme S3. Synthesis of stilbene-stilbene homo-[3]rotaxane $\mathbf{3}_2$⊂γ-CD.

Synthesis of stilbene-stilbene [3]rotaxane $\mathbf{3}_2$⊂γ-CD. Degassed water (6 cm^3) was added to a mixture of **2** (20 mg, 62 μmol), iodoterphenylene dicarboxylic acid **1** (55 mg, 113 μmol), [2]rotaxane **3**⊂γ-CD (20 mg, 9.50 μmol) and potassium carbonate (188 mg, 1.4 mmol) under nitrogen. The mixture was heated to 45 °C and palladium(II) acetate (1.0 μl, 0.63 μmol) was added from a stock solution (0.63 M in DMSO). The mixture was stirred for 6 hrs at 45 °C. The volume was made up to 15 cm^3 with deionized water, and the cooled mixture was filtered through paper. HCl (20 cm^3, 1.0 M) was added and the resulting suspension was centrifuged. The supernatant was removed and the solid was washed twice with 20 cm^3 water. The solid was subsequently dried *in vacuo* and dissolved in DMSO (1 cm^3) and purified by semipreparative HPLC using 'Method 2' (See Fig. S1). The fractions were concentrated *in vacuo*; the solid was subsequently collected by centrifugation, washed with water (3 × 20 cm^3) to yield rotaxane $\mathbf{3}_2$⊂γ-CD as a white solid (conversion 97% by HPLC; yield: 24 mg, 8.2 μmol, 87%). λ_{max} (3:1 MeOH: aq. CHAPS) 344 nm, 276 nm; ^{1}H NMR (500 MHz, d_6-DMSO) δ 3.29–3.46 (m, 16H), 3.59–3.71 (m, 32H) 4.49 (t, J = 5 Hz, 8H), 4.84 (br s, 8H), 5.60 (br s, 8H), 5.79 (d, J = 5.7 Hz, 8H), 6.63 (d, J = 16.0 Hz, 2H), 6.75 (d, J = 16.0 Hz, 2H), 7.27 (d, J = 7.6 Hz, 4H), 7.43 (d, J = 7.9 Hz, 4H), 7.44 (d, J = 7.9 Hz, 4H), 7.54–7.57 (m, 10H), 7.62 (s, 2H), 7.65 (d, J = 8.2 Hz, 8H), 7.72 (s, 4H), 7.73 (d, J = 8.5 Hz, 8H), 7.94–7.97 (m, 16 Hz). *m/z* (TOF MS ES-) found 583 $[M-5H]^{5-}$ (10%), 730 $[M-4H]^{4-}$ (100%), 973 $[M-3H]^{3-}$ (50%), 1460 $[M-2H]^{2-}$ (10%).

Section 2: NMR Spectra, Mass Spectra and HPLC Traces

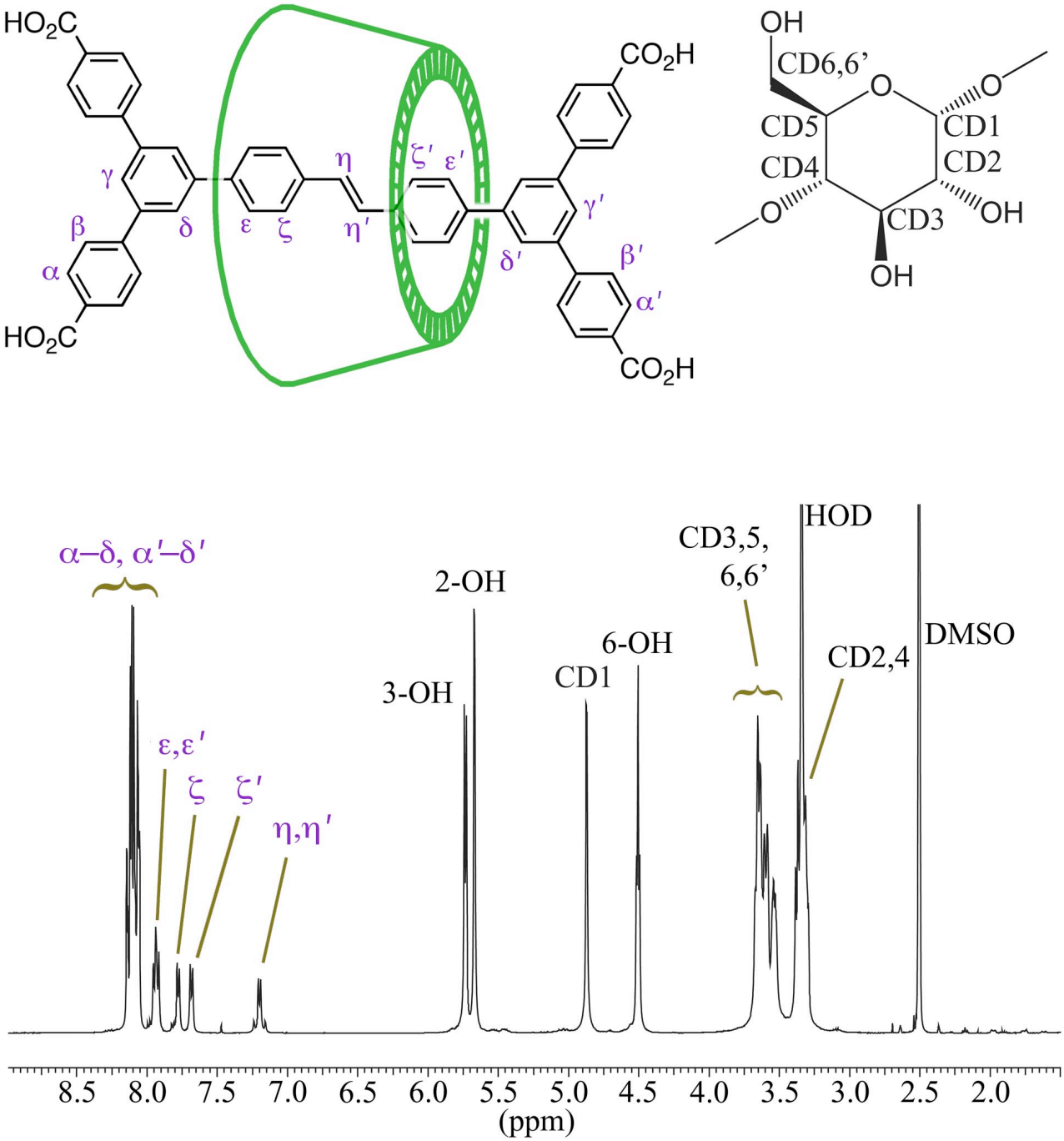


Figure S2. ^{1}H NMR of **3**⊂*γ*-CD (500 MHz, d_6-DMSO, 298 K). The spectrum was assigned using a combination of COSY, NOESY and HSQC experiments.

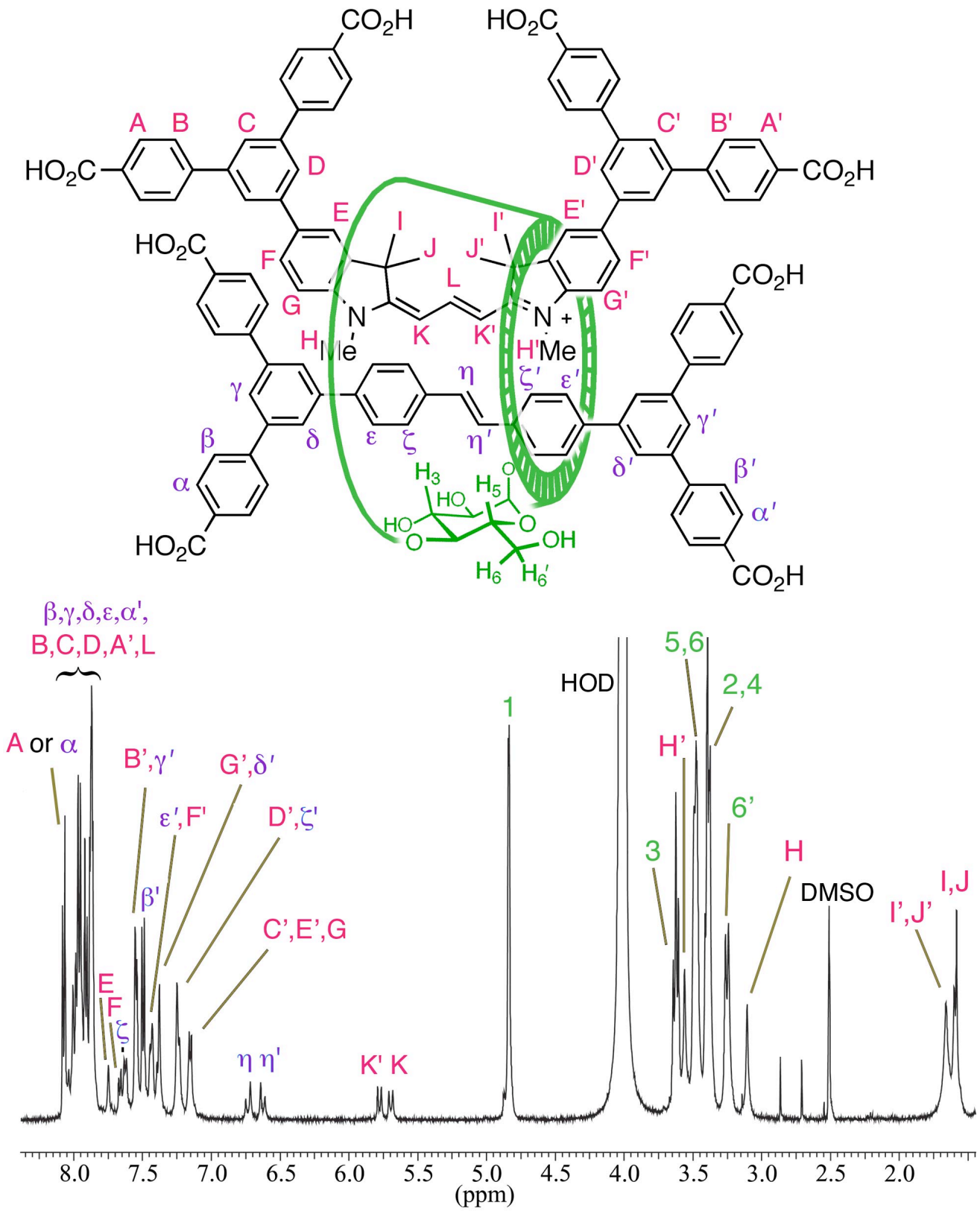
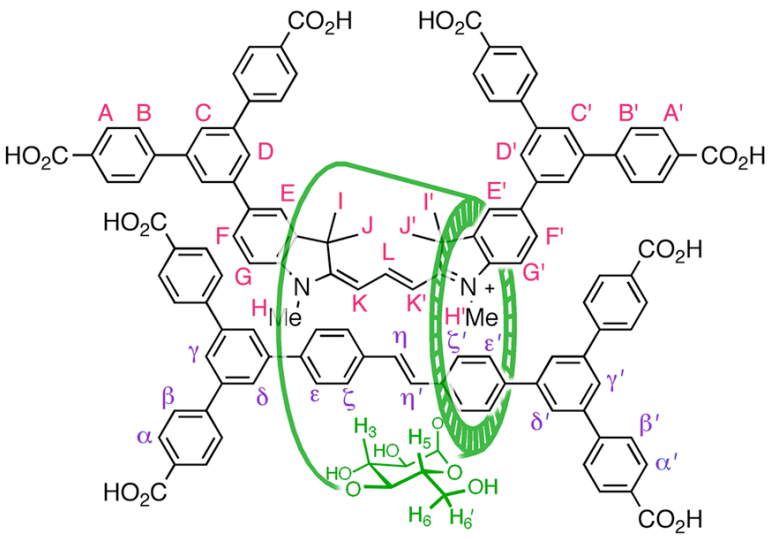


Figure S3. ^{1}H NMR spectrum of (**3·5**)⊂γ-CD (500 MHz, d_6-DMSO, 343 K). The spectrum was assigned using a combination of COSY, NOESY, ROESY and HSQC experiments. The spectrum was recorded at 343 K because severe broadening was observed at room temperature. On some occasions this compound gave broad spectra in d_6-DMSO even at 343 K, and sharp spectra were obtained by adding a trace of DCl (1 μl of 1.0 M DCl in D_2O).



		stilbene resonances (**3**)						γ-CD resonances		
		ε	**ζ**	**η**	**η'**	**ζ'**	**ε'***	**CD3**	**CD5,6**	**CD6'**
cyanine dye resonances (**5**)	**B**	–	–	–	–	–	–	–	–	–
	E	–	–	–	–	–	–	–	–	–
	H	–	–	–	–	–		–	–	–
	I,J	M	–	–	–	–	–	M	–	–
	K	–	–	–	–	–	–	–	–	–
	L	–	–	–	–	–	–	–	–	–
	K'	–	–	–	–	–	–	–	–	–
	I',J'	–	w	–	w	–	–	**S**	M	–
	H'	w	M	–	–	–	–	§	–	–
	G'	–	–	–	–	–	–	M	**S**	–
	F'*	–	–	–	–	–	–		**(S*)**	w*
	E'	–	–	–	–	–	–	w	**S**	–
	D'	–	–	–	–	–	–	–	**S**	w
	B'	–	–	–	–	–	–	–	M	M
	A'								w	M
γ-CD	**CD3**	M	**S**	M	w	–	–			
	CD5,6	–	M	w	M	**S**	**S***			
	CD6'	–	–	–	–	w	w*			

Table S1. NOE correlations between the three components of (**3**·**5**)⊂γ-CD from the ROESY spectrum in d_6-DMSO (500 MHz, 343 K, 1 mm microprobe sample tube, 200 ms mixing time using the transverse-ROESY sequence[5]). **S** = strong, M = medium, w = weak. Resonances are listed in order, with a red line corresponding to the centre of each dumbbell component, as a guide to the eye. Note that the stilbene-CD correlations are evenly spread across the center of the stilbene dumbbell, with a slight weighting towards the left-hand end of the stilbene (the end nearer the 2,3-rim of the γ-CD), whereas the cyanine-CD correlations are located at the right-hand end of the cyanine (the end nearer the 5,6-rim of the γ-CD). In keeping with this, the stilbene-cyanine correlations are mostly between the left-hand end of the stilbene and the right-hand end of the cyanine.

* F' and ε' overlap leading to ambiguity, but the strong cross-peak with CD5,6 appears to be a correlation with ε' based on its line shape.

§ It is impossible to see whether there is a H'-CD3 cross-peak because it would be too close to the diagonal.

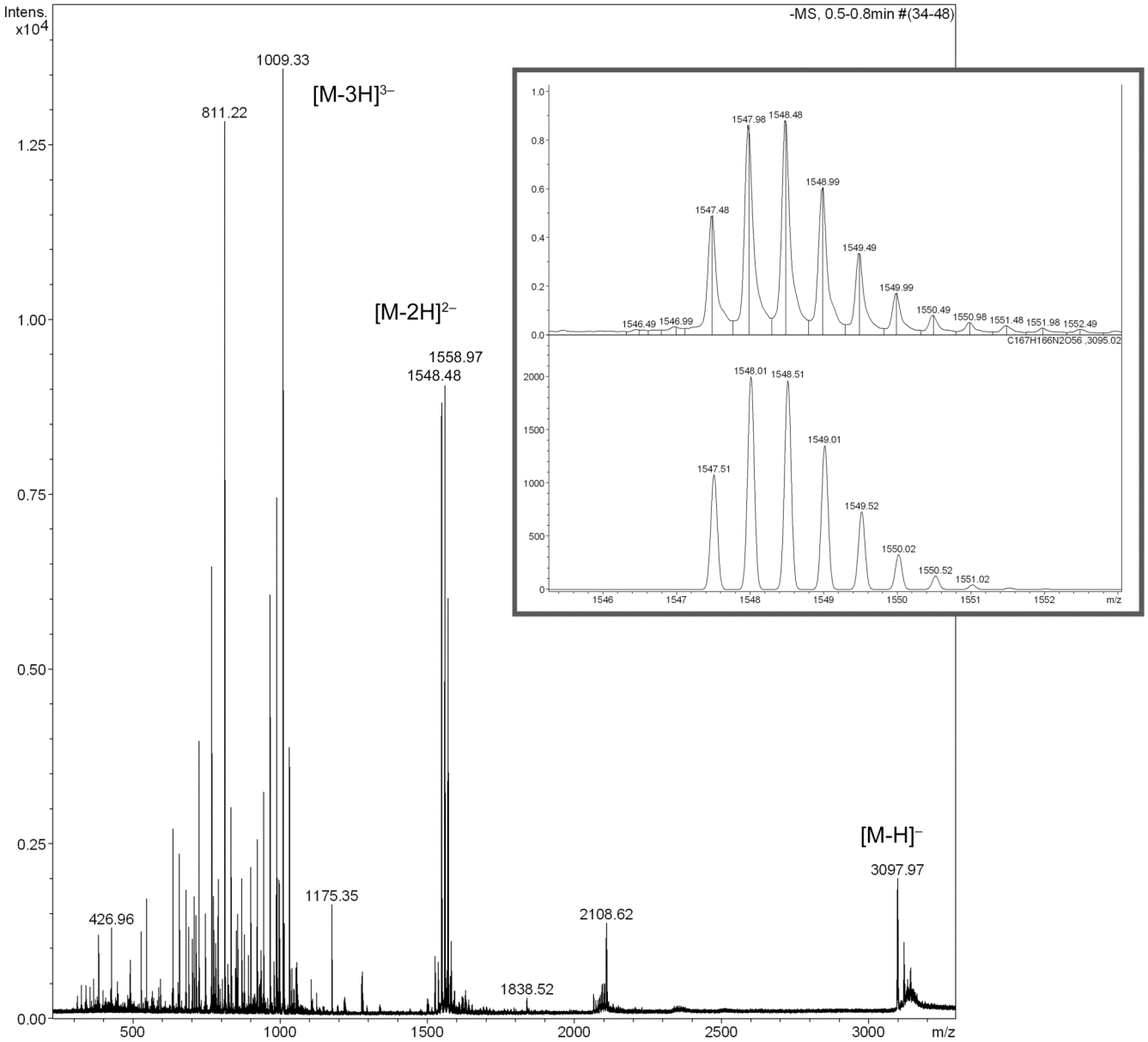


Figure S4. Mass spectrum (ESI –ive) of (**3·5**)⊂*γ*-CD showing peaks due to $[M\text{-}H]^-$ (3097.97), $[M\text{-}2H]^{2-}$ (1548.48) and $[M\text{-}3H]^{3-}$ (1009.33). Insert: High-resolution ESI MS of $[M\text{-}2H]^{2-}$ of (**3·5**)⊂*γ*-CD (Top: experimental spectrum; Bottom: calculated spectrum for $[C_{167}H_{166}N_2O_{56}]^{2-}$).

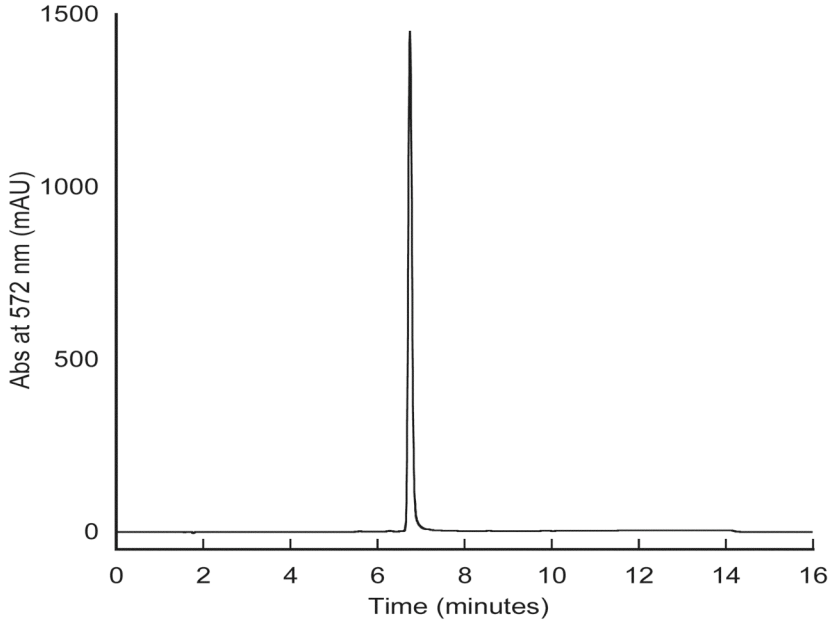


Figure S5. HPLC trace of the [3]rotaxane (**3·5**)⊂*γ*-CD (absorbance at 572 nm, 'Method 1' – see Fig. S1).

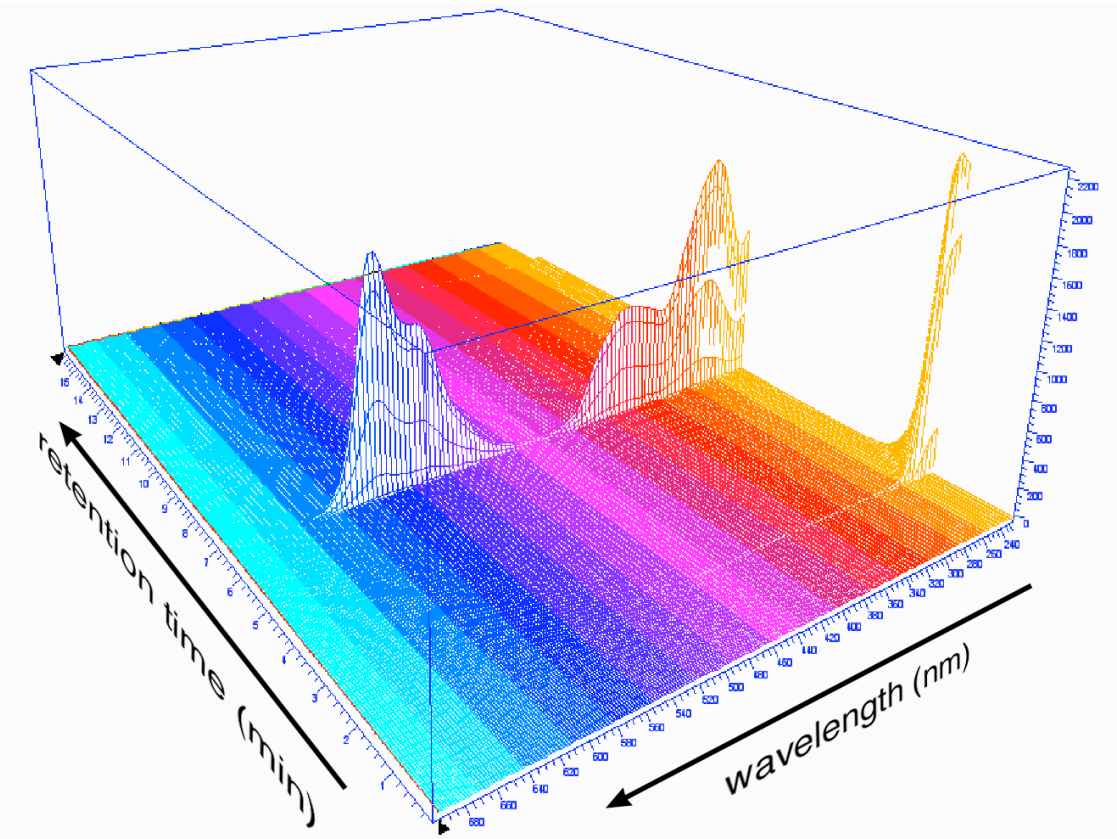


Figure S6. 3D HPLC trace of (**3·5**)⊂*γ*-CD (wavelength 250–800 nm and time 0–16 minutes; 'Method 1' – see Fig. S1).

Section 3: UV-vis Titrations

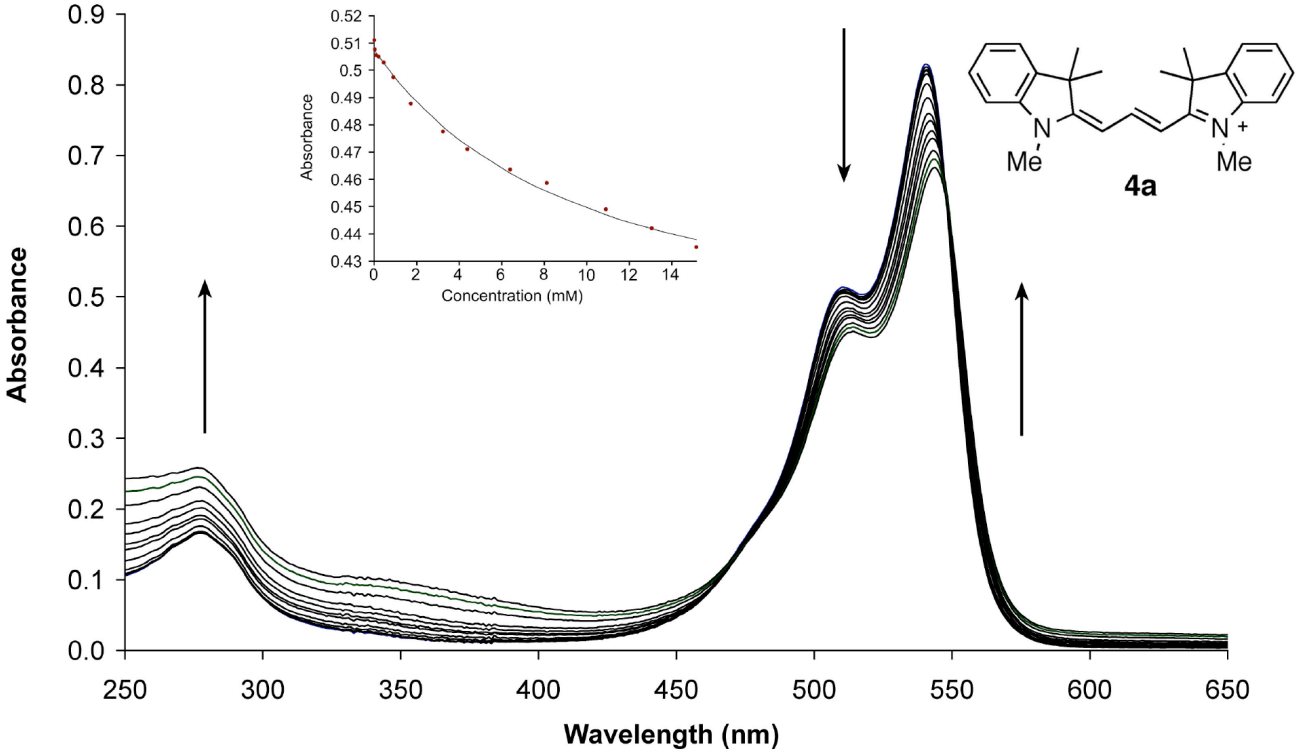


Figure S7. Binding titration of cyanine dye **4a** iodide salt and γ-CD in aqueous sodium phosphate buffer (pH = 11.4). The data at 509 nm were fitted to a 1:1 binding isotherm using Specfit-32 (inset) to give a binding constant of $K = 87 \pm 15$ M^{-1}. Arrows indicate areas of increasing and decreasing absorbance during the titration. The titration was carried out at a constant concentration of **4a** (5.9 μM), by monitoring absorption spectra with increasing concentration of γ-CD (0–15.2 mM).

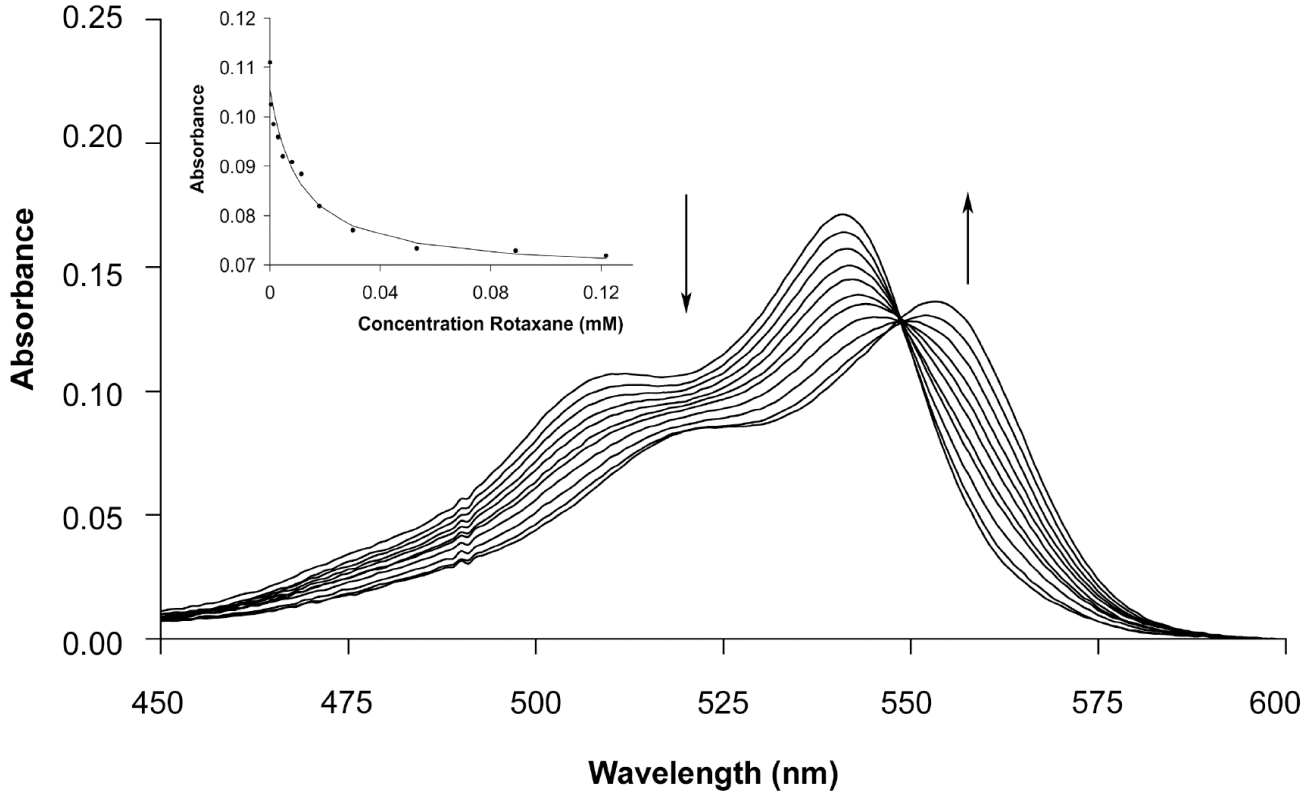


Figure S8. Binding titration of **3**⊂γ-CD and **4a** in aqueous sodium phosphate buffer (pH 11.4). The data at 509 nm were fitted to a 1:1 binding isotherm using Specfit-32 (inset) giving a binding constant of $K = 1.0 \pm 0.2 \times 10^5$ M^{-1}. Arrows indicate areas of increasing and decreasing absorbance during the titration. The titration was carried out at a constant concentration of **4a** (0.91 μM), by monitoring absorption spectra with increasing concentration of **3**⊂γ-CD (0–0.12 mM).

Section 4: Fluorescence Data

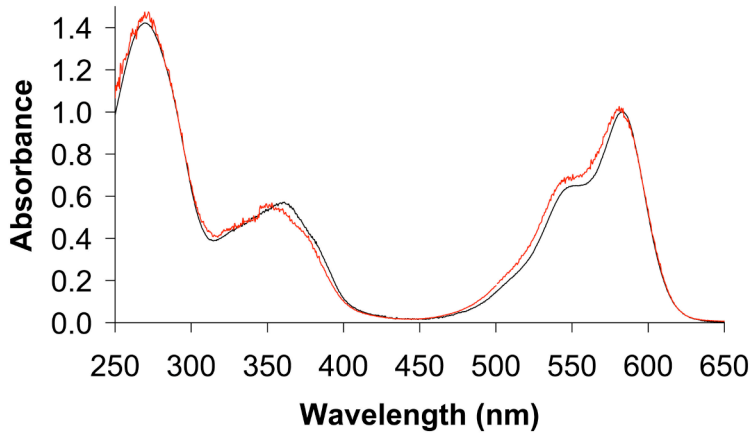


Figure S9. Fluorescence excitation spectrum (red) and UV-vis absorption spectrum (blue) of (**3·5**)⊂*γ*-CD in aqueous sodium phosphate buffer (pH 11.4). The excitation spectrum was corrected using two standards: DCM[‡] in MeCN (from 250–500 nm) and styryl 9M[¶] in ethanol (from 500–650 nm). The ratio (normalized UV-vis spectrum/ normalized fluorescence excitation spectrum) between the UV-vis and excitation spectra of these standards was used to correct the fluorescence excitation spectrum of (**3·5**)⊂*γ*-CD over the ranges specified.

Compound:	**5**⊂*γ*-CD	**3**⊂*γ*-CD	(**3·5**)⊂*γ*-CD
Quantum yield Φ_F:	0.12	0.54	0.56

Table S2. Fluorescence quantum yields for [2]rotaxanes **5**⊂*γ*-CD and **5**⊂*γ*-CD and [3]rotaxane (**3·5**)⊂*γ*-CD, all in aqueous sodium phosphate buffer (pH 11.4). The standard used for **3**⊂*γ*-CD quantum yield was quinine sulfate in 0.1 M H_2SO_4 (Φ_F = 0.54).[6] The standard used for **5**⊂*γ*-CD and (**3·5**)⊂*γ*-CD quantum yield was cresyl violet[#] perchlorate in EtOH (Φ_F = 0.52).[7] The excitation wavelength for **5**⊂*γ*-CD and (**3·5**)⊂*γ*-CD was 490 nm. The excitation wavelength for **3**⊂*γ*-CD was 350 nm. Fluorescence quantum yield of (**3·5**)⊂*γ*-CD, when excited at 350 nm compared to DCM[‡] in MeCN (Φ_F = 0.60),[8] was found to be almost identical to quantum yield when excited at 490 nm.

[‡] "DCM" is 4-dicyanomethylene-2-methyl-6-[*para*-(dimethylamino)styryl]-4H-pyran.
[¶] "Styryl 9M" is 2-(6-(4-dimethylaminophenyl)-2,4-neopentylene-1,3,5-hexatrienyl)-3-methylbenzothiazolium perchlorate.
[#] "Cresyl violet" is 5,9-diaminobenzo[*a*]phoxazonium perchlorate.

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