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Electronic Supplementary Information for

Sodium Cation-Templated Synthesis and Crystal Structure of a Ion-Pair Binding Heteroditopic [2]Catenane

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Contents

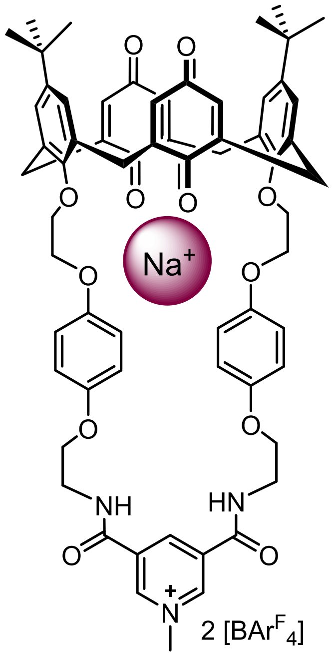
1. Experimental and ^{1}H, ^{13}C NMR and HRMS spectra

1.1 General Considerations

Commercial grade chemicals and solvents were used without further purification. Where anhydrous solvents were used, they were degassed with N_2 and passed through an MBraun MPSP-800 column. Triethylamine was distilled from and stored over potassium hydroxide. De-ionised water dispensed from a Millipore Milli-Q purification system was used in all cases. Tetrabutylammonium (TBA) salts were stored under vacuum in a desiccator. Microwave reactions were carried out using a Biotage Initiator 2.0 microwave. Routine 300 MHz NMR spectra were recorded on a Varian Mercury 300 spectrometer, ^{1}H NMR operating at 300 MHz, ^{13}C{^{1}H} at 76 MHz, ^{19}F at 283 MHz and ^{31}P at 121 MHz . Where the solubility of the compounds were too low, or not enough compound existed, a Bruker AVII500 with ^{13}C Cryoprobe spectrometer was used for obtaining ^{13}C{^{1}H} at 126 MHz, however in some cases a compete ^{13}C{^{1}H} spectrum could not be obtained. All 500 MHz ^{1}H Spectra and all ^{1}H NMR titrations were recorded on a Varian Unity Plus 500 spectrometer. All chemical shift (δ) values are given in parts per million and are referenced to the solvent. In cases where solvent mixtures are used, the solvent with the highest percentage of proto-solvent is taken as the reference. Where an apparent multiplet (*e.g.* app. t.) is quoted, J_{app} is given. Low resolution ESI mass spectra were recorded on a Micromass LCT Premier XE spectrometer. Accurate masses were determined to four decimal places using Bruker µTOF and Micromass GCT spectrometers.

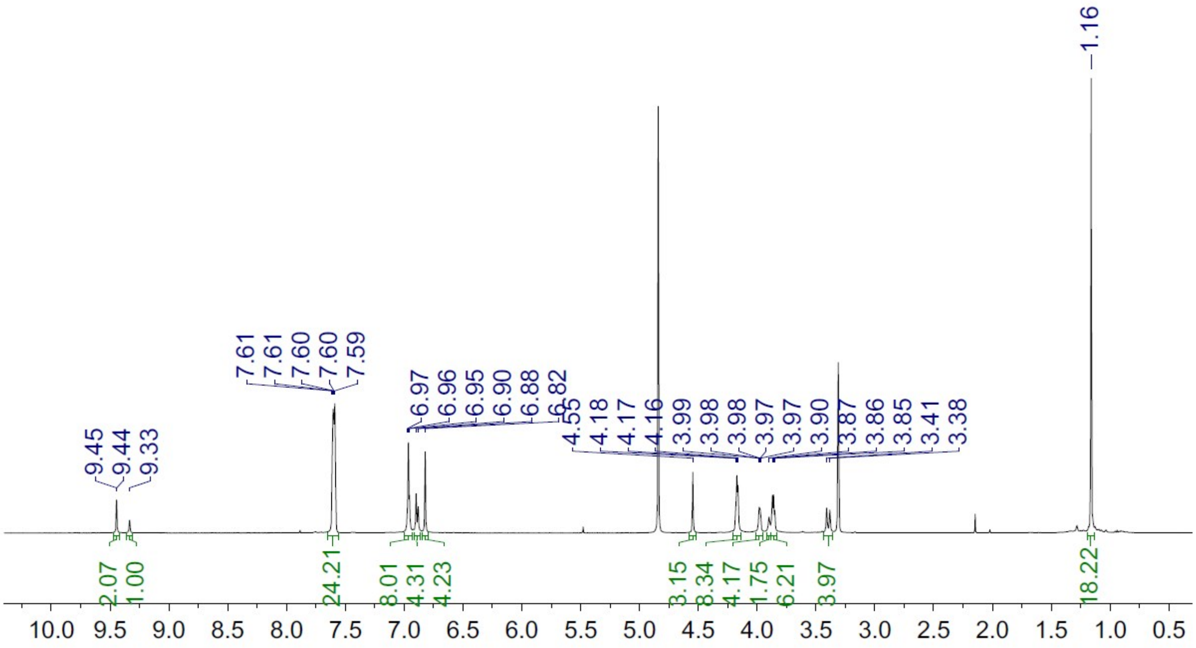
Calix[4]diquinone macrocycle **1**,[1] Na[BAr$^{F}{}_{4}$],[2] 3,5-pyridine-*N*-oxide dicarboxylic acid **3**,[3] diamine **5**,[4] and 3,5-dihexylamide-N-methyl pyridinium thread **6·Cl**[5] were synthesised as previously described.

1.2 Synthesis of calix[4]diquinone pyridinium macrocycle 2·Na·[BAr$^{F}{}_{4}$]$_2$

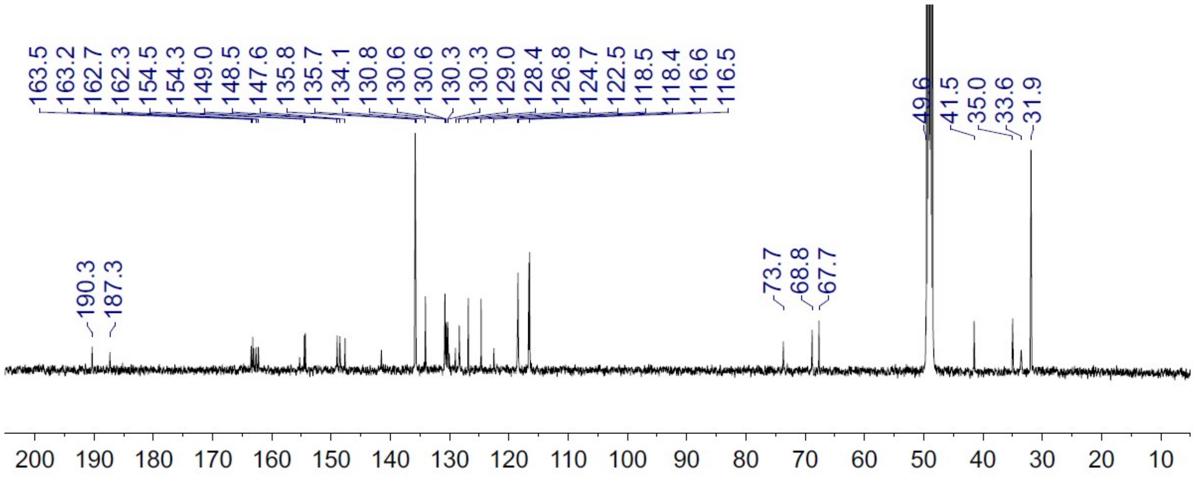


Calix[4]diquinone pyridine macrocycle **1** (100 mg, 0.095 mmol) was dissolved in $CHCl_3$ (10 mL). MeI (2 mL) was added and the solution was stirred at ambient temperature for three days after which time a precipitate had formed. The solvent was removed *in vacuo* and the resulting residue suspended in $CHCl_3$ (30 mL). Sodium tetrakis[(3,5-trifluoromethyl)phenyl]borate (Na[BAr$^{F}{}_{4}$]) (177 mg, 0.200 mmol) was added and the reaction stirred at ambient temperature for 30 minutes, after which time the precipitate had dissolved. The solution was washed with H_2O (3 × 30 mL), dried over anhydrous $MgSO_4$, filtered and the solvent removed *in vacuo* to give **2·Na·[BAr^F$_4$]$_2$** as an orange waxy solid (249 mg, 93%, 88.3 µmol). ^{1}H NMR (500 MHz, CD_3OD) δ_H 9.44 (2H, s, Py$\underline{\text{H}}$), 9.33 (s, 1H, Py$\underline{\text{H}}$), 7.58 – 7.62 (24H, m, Ar^F$\underline{\text{H}}$), 6.94 – 6.98 (8H, m, Ar$\underline{\text{H}}$ + HQ$\underline{\text{H}}$), 6.89 (4H, d, $^3J_{HH}$ = 9.0, HQ$\underline{\text{H}}$), 6.82 (4H, s, Quin$\underline{\text{H}}$), 4.55 (3H, s, NCH_3), 4.15 – 4.19 (8H, m , OC$\underline{\text{H}}_2$), 3.95 – 4.01 (4H, m, OC$\underline{\text{H}}_2$), 3.83 – 3.91 (8H, m, ArC$\underline{\text{H}}$H + NC$\underline{\text{H}}_2$), 3.40 (4H, d, $^2J_{HH}$ =13.3, ArCH$\underline{\text{H}}$), 1.16 (18H, s, $C(CH_3)_3$); ^{13}C NMR (126 MHz, MeOD) δ 190.3 (s, Quin), 187.3 (s, Quin), 163.2 (s, Ar), 162.9 (q, $^1J_{BC}$ = 50, Ar^F), 154.5 (s, Ar), 154.4 (s, Ar), 149.0 (s, Ar), 148.5 (s, Ar), 147.6 (s, Ar), 141.50 (s, Ar), 135.8 (s, Ar^F), 135.7 (s, Ar), 134.1 (s, Ar), 130.8 (s, Ar), 130.4 (qq, $^2J_{FC}$ = 32, $^3J_{BC}$ = 3, Ar^F), 129.0 (s, Ar), 125.8 (q, $^1J_{FC}$ = 271, Ar^F), 118.5 (sept., $^3J_{FC}$ = 41, Ar^F), 116.61 (s, Ar), 116.5 (s, Ar), 73.69 (s, $\underline{C}H_2$), 68.8 (s, $\underline{C}H_2$), 67.7 (s, $\underline{C}H_2$), 49.6 (s, $\underline{C}H_2$), 41.5 (s, $\underline{C}H_3$), 35.0 (s, $\underline{C}H_2$), 33.6 (s, $\underline{C}(CH_3)_3$), 31.9 (s, $C(\underline{C}H_3)_3$); ESI-HRMS m/z calcd. for $[C_{128}H_{90}B_2F_{28}N_3NaO_{12} - Na - 2[BAr^F{}_4]]^+$ 1068.4641, found 1068.4612.

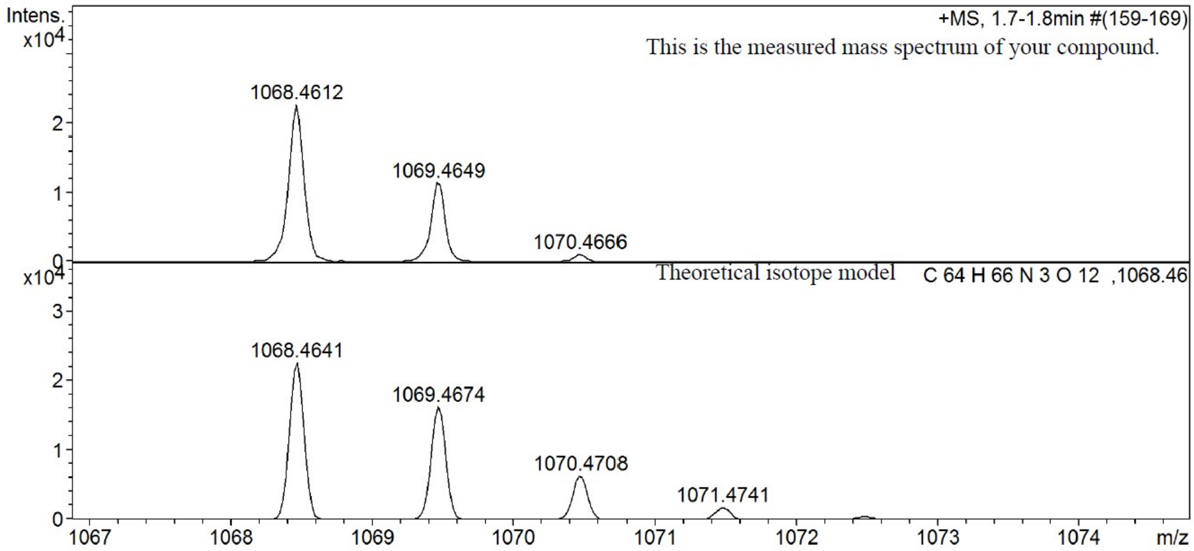
^{1}H NMR spectrum (400 MHz, CD_3OD, 298 K)



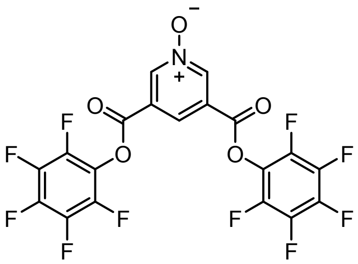
^{13}C NMR spectrum (126 MHz, CD_3OD, 298 K)



HRMS spectrum (ESI)

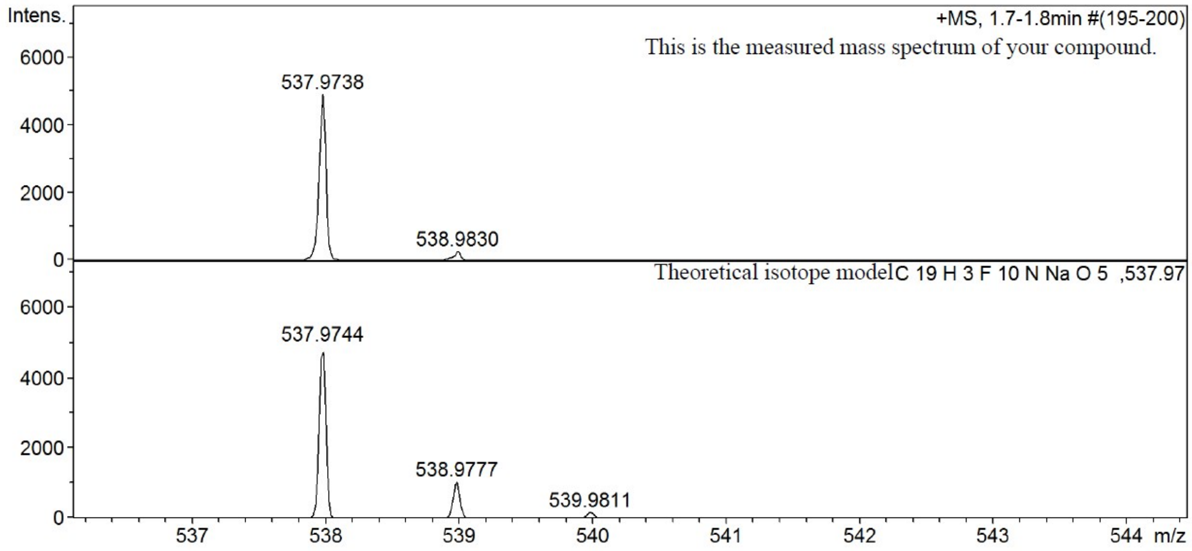


1.3 Synthesis of di-pentafluorophenyl-3,5-pyridine-*N*-oxide ester 4

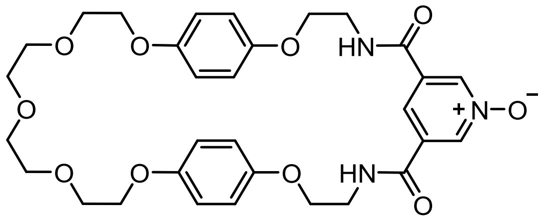


3,5-pyridine-*N*-oxide dicarboxylic acid **3** (50 mg, 0.27 mmol) was dissolved in dry DMF (5 mL). 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide (92.8 mg, 0.60 mmol) and pentafluorophenol (110 mg, 0.60 mmol) were added and the reaction stirred at ambient temperature for 18 hours. The reaction was poured onto H_2O (50 mL) and filtered immediately to give **4** (103 mg, 74%, 0.200 mmol). *This compound was used immediately without full characterisation due to its high reactivity*. ESI-HRMS m/z calcd. for $[C_{19}H_3F_{10}NO_5 + Na]^+$ 573.9744, found 573.9738.

HRMS spectrum (ESI)

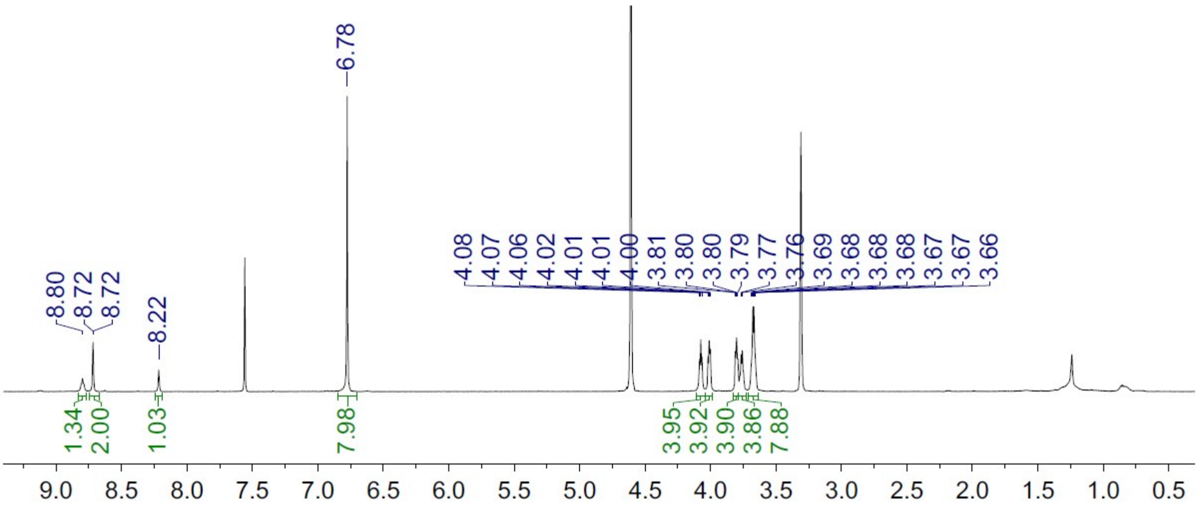


1.4 Synthesis of pyridine *N*-oxide macrocycle 7

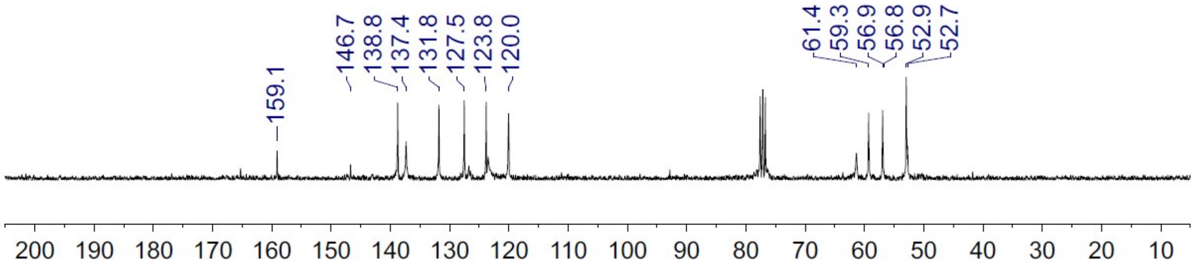


Di-pentafluorophenyl-3,5-pyridine-*N*-oxide ester **4** (100 mg, 0.19 mmol, 1.0 eq.), pyridinium bis-hexylamide **6·Cl** (74 mg, 0.19 mmol, 0.10 eq.) and Et_3N (81 µL, 0.58 mmol, 3.0 eq.) was dissolved in anhydrous CH_2Cl_2 (10 mL). Bis-amine **5** (90 mg , 0.19 mmol, 0.10 eq.) in anhydrous CH_2Cl_2 (10 mL) was added dropwise over one hour at ambient temperature. The solvent was removed *in vacuo* and the product purified by column chromatography (SiO_2, CH_2Cl_2/MeOH 97:3) to afford the compound **7** as a white waxy solid (27 mg, 21%, 44.1 µmol). ^1H NMR (500 MHz, $CDCl_3/CD_3OD$ 1:1) δ_H 8.80 (2H, t, $^3J_{HH}$ = 5.0, N$\underline{H}$), 8.72 (2H, s, Py$\underline{H}$), 8.22 (1H, s, Py$\underline{H}$), 6.78 (8H, s, Ar$\underline{H}$), 4.07 (4H, t, $^3J_{HH}$ = 4.9, C$\underline{H}_2$), 4.01 (4H, dd, $^3J_{HH}$ = 5.6, 3.6, C$\underline{H}_2$), 3.80 (4H, dd, $^3J_{HH}$ = 5.6, 3.5, C$\underline{H}_2$), 3.74 – 3.78 (4H, m, C$\underline{H}_2$), 3.64 – 3.70 (8H, *m*, C$\underline{H}_2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.1 (s, NH$\underline{C}$O), 146.7 (s, Ar), 138.8 (s, Ar), 137.4 (s, Ar), 131.8 (s, Ar), 127.5 (s, Ar), 123.8 (s, Ar), 120.0 (s, Ar), 61.4 (s, $\underline{C}H_2$), 59.3 (s, $\underline{C}H_2$), 56.9 (s, $\underline{C}H_2$), 56.8 (s, $\underline{C}H_2$), 52.9 (s, $\underline{C}H_2$), 52.7 (s, $\underline{C}H_2$); ESI-HRMS m/z calcd. for $[C_{31}H_{37}N_3O_{10} + Na]^+$ 634.2371, found 634.2370.

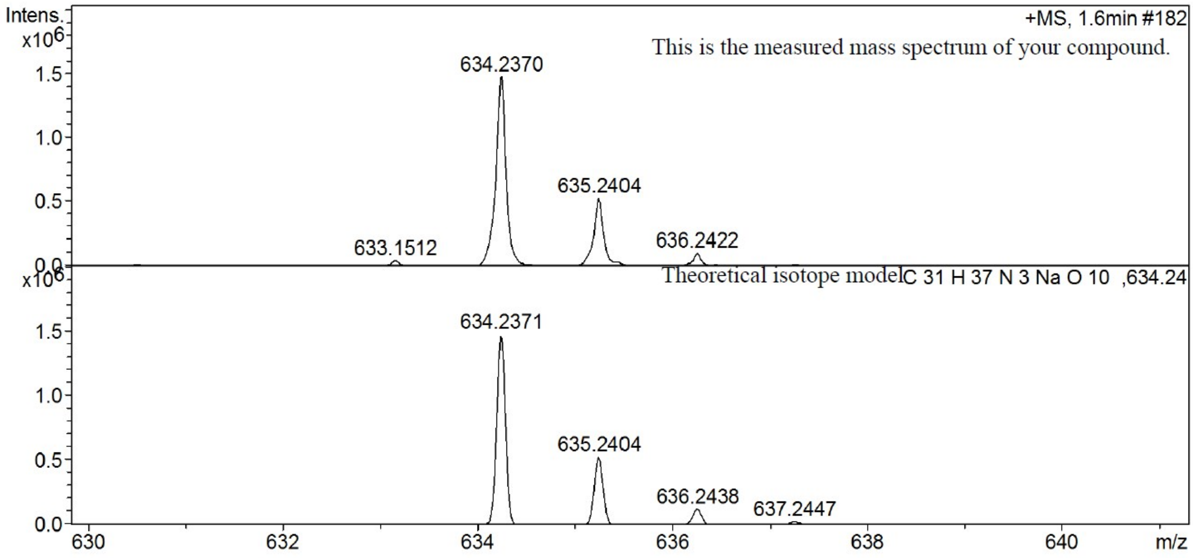
^{1}H NMR spectrum (500 MHz, $CDCl_3/CD_3OD$ 1:1, 298 K)



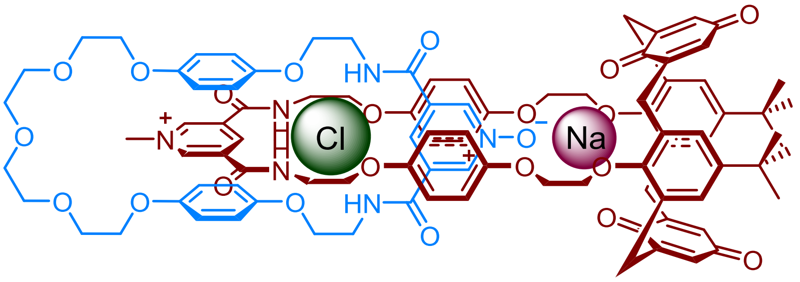
^{13}C NMR spectrum (76 MHz, $CDCl_3$, 298 K)



HRMS spectrum (ESI)

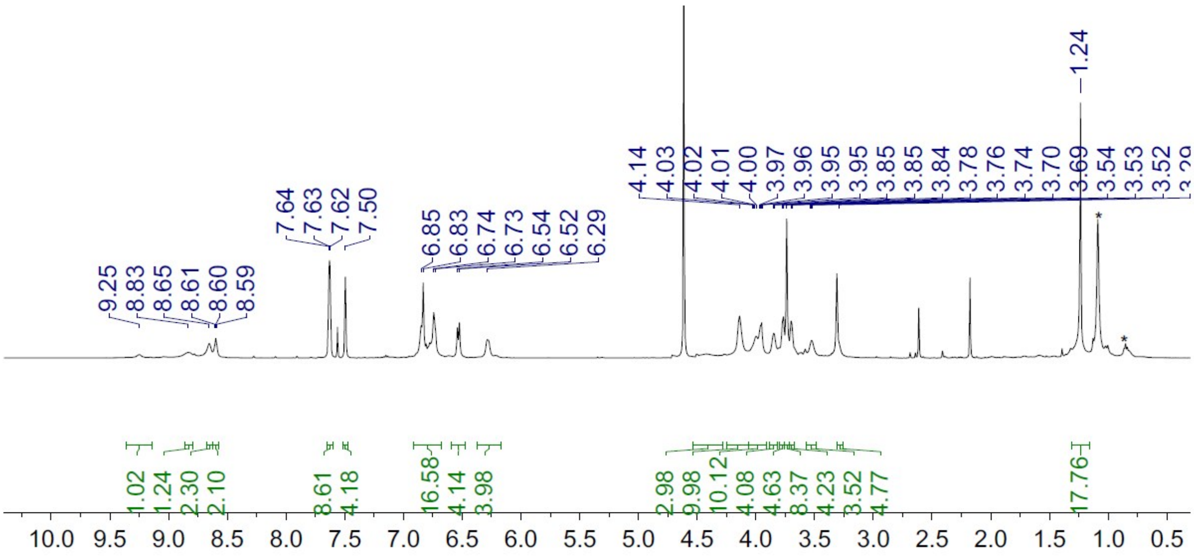


1.5 Synthesis of calix[4]diquinone pyridinium [2]catenane 8·NaCl·$[\mathrm{BAr^F}_4]$

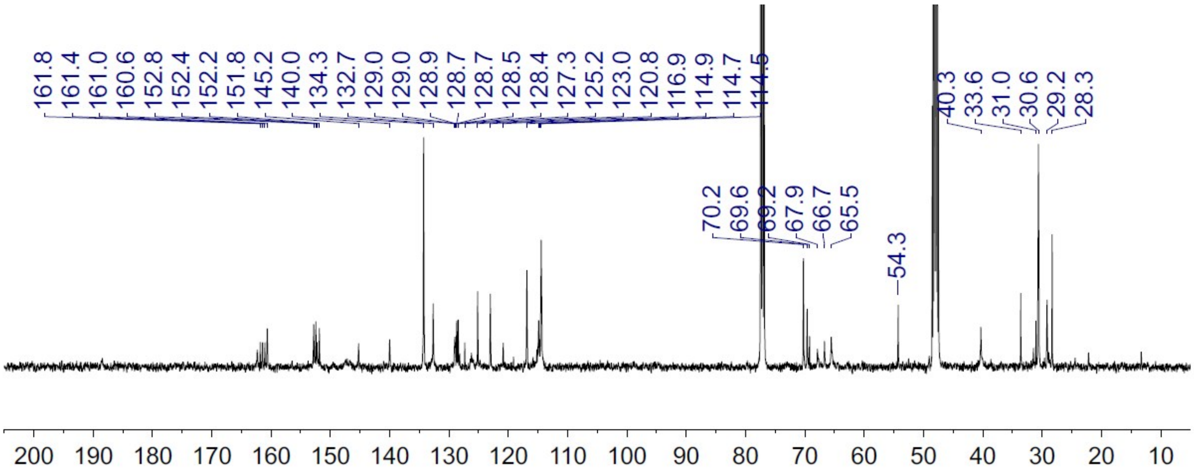


Calix[4]diquinone pyridinium macrocycle **2·Na·$[\mathrm{BAr^F}_4]_2$** (100 mg, 35.5 µmol, 1.0 eq.) and pyridine *N*-oxide bis-ester (18.3 mg, 35.5 µmol, 1.0 eq.) were dissolved in anhydrous CH_2Cl_2 (5 mL). Bis-amine (16.5 mg, 35.5 µmol, 1.0 eq.) and Et_3N (14.8 µL, 106.5 µmol, 3.0 eq.) dissolved in anhydrous CH_2Cl_2 (5 mL) was added dropwise *via* syringe pump over 18 hours and the reaction stirred at ambient temperature for a further 24 hours. The solvent was removed *in vacuo* and the crude material purified by size-exclusion chromatography (Bio-Beads SX-1; 3 cycles; $CHCl_3$) and preparative thin-layer chromatography (SiO_2; gradient elution CH_2Cl_2/MeOH; 100:0→95:5) to afford the compound **8·NaCl·$[\mathrm{BAr^F}_4]$** as a yellow oil (6.2 mg, 6.7%, 2.4 µmol), the source of chloride is assumed to be the extensive size-exclusion purification in chloroform and has been noted for other chloride binding receptors.[6] ^1H NMR (500 MHz, $CDCl_3/CD_3OD$ 1:1) δ_H 9.25 (1H, s, $Py^+\underline{H}$), 8.83 (1H, s, $Py^{NO}\underline{H}$), 8.65 (2H, s, $Py^+\underline{H}$), 8.59 (2H, s, $Py^{NO}\underline{H}$), 7.65 – 7.60 (8H, s, Ar^F), 7.50 (4H, s, Ar^F), 6.91 – 6.68 (16H, m, $Ar\underline{H}$ + $Quin\underline{H}$ + $HQ\underline{H}$), 6.53 (4H, d, $^3J_{HH}$ = 8.4, $HQ\underline{H}$), 6.28 (4H, d, $^3J_{HH}$ = 8.3, $HQ\underline{H}$), 4.41 (3H, s, $N\underline{H_3}$), 4.14 (10H, s, $C\underline{H_2}$), 4.06 – 3.91 (10H, m, $C\underline{H_2}$), 3.88 – 3.82 (4H, m, $C\underline{H_2}$), 3.77 (4H, s, $C\underline{H_2}$), 3.74 (8H, s, $C\underline{H_2}$), 3.70 (4H, s, $C\underline{H_2}$), 3.53 (4H, s, $C\underline{H_2}$), 3.29 (4H, app. s, $C\underline{H_2}$), 1.24 (18H, s, $C(CH_3)_3$); ^{13}C NMR (126 MHz, $CDCl_3/CD_3OD$ 1:1) δ 162.3 (s, Quin.), 162.26 (s, Quin.), 161.2 (q, $^1J_{BC}$ = 50, Ar^F), 152.8 (s, Ar), 152.4 (s, Ar), 152.2 (s, Ar), 151.9 (s, Ar), 145.2 (s, Ar), 140.0 (s, Ar), 134.3 (s, Ar^F), 132.7 (s, Ar), 129.0 (s, Ar), 129.0 (s, Ar), 128.6 (qq, $^2J_{FC}$ = 31, $^3J_{BC}$ = 3, Ar^F), 124.1 (q, $^1J_{FC}$ = 271, Ar^F), (sept., $^3J_{FC}$ = 41, Ar^F), 115.2 (s, Ar), 115.0 (s, Ar), 114.9 (s, Ar), 114.7 (s, Ar), 114.5 (s, Ar), 70.2 (s, $\underline{C}H_2$), 69.6 (s, $\underline{C}H_2$), 69.2 (s, $\underline{C}H_2$), 67.9 (s, $\underline{C}H_2$), 66.7 (s, $\underline{C}H_2$), 65.6 (s, $\underline{C}H_2$), 54.3 (s, $\underline{C}H_2$), 40.3 (s, $N\underline{C}H_3$), 33.6 (s, $N\underline{C}H_2$), 31.1 (s, $N\underline{C}H_3$), 30.6 (s, $Ar\underline{C}HH$), 29.2 (s, $\underline{C}(CH_3)_2$), 28.3 (s, $C(\underline{C}H_3)_2$), remaining quaternary carbons undetected; ESI-HRMS m/z calcd. for $[C_{127}H_{115}BClF_{24}N_6NaO_{22} - [\mathrm{BAr^F}_4] - Cl]^{2+}$ 1702.6972, found 1702.6930.

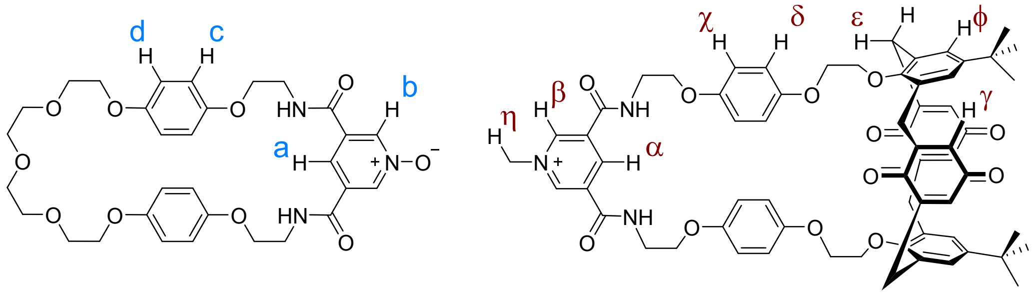
^{1}**H NMR** spectrum (500 MHz, $CDCl_3$/CD_3OD 1:1, 298 K)

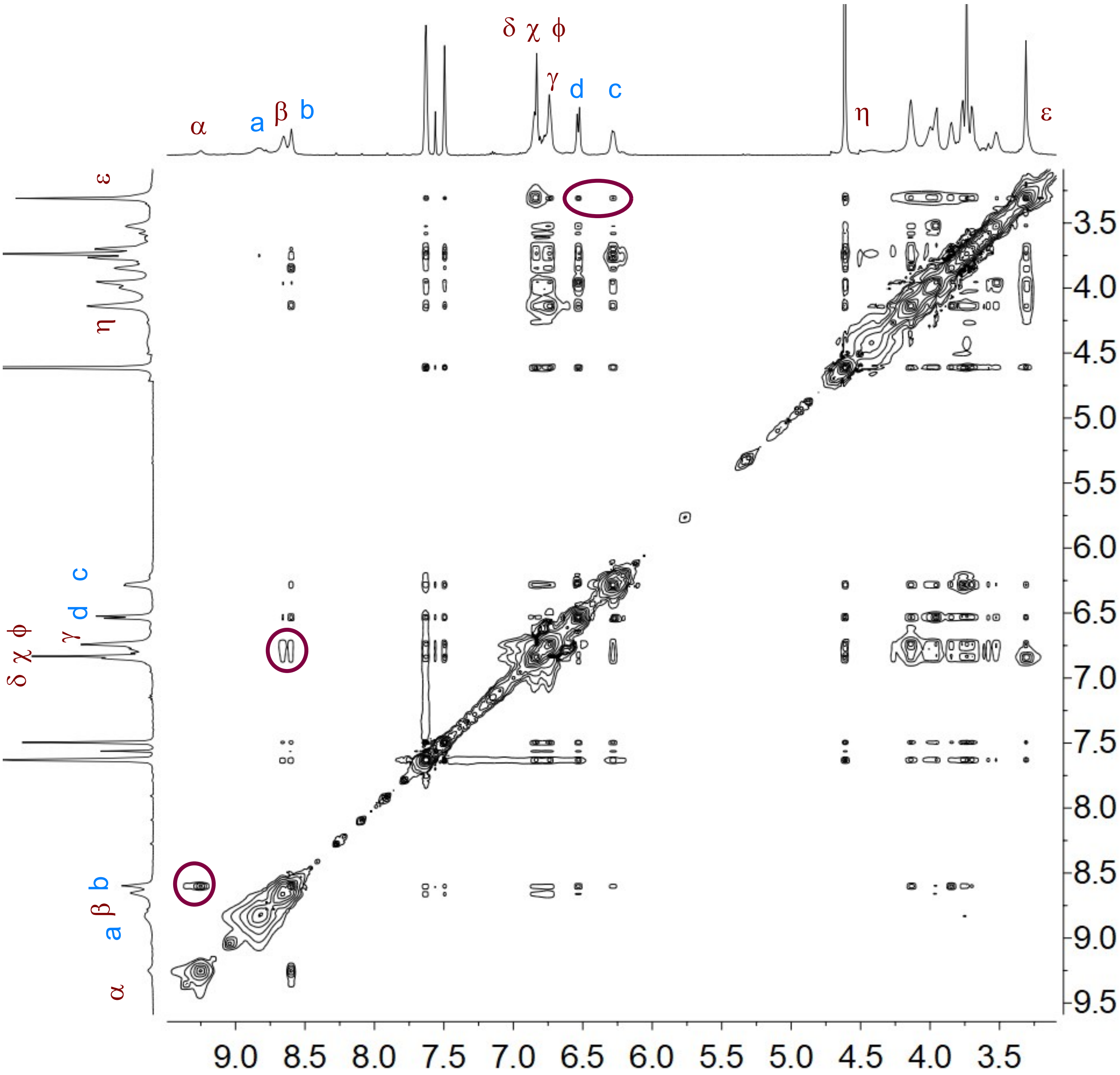


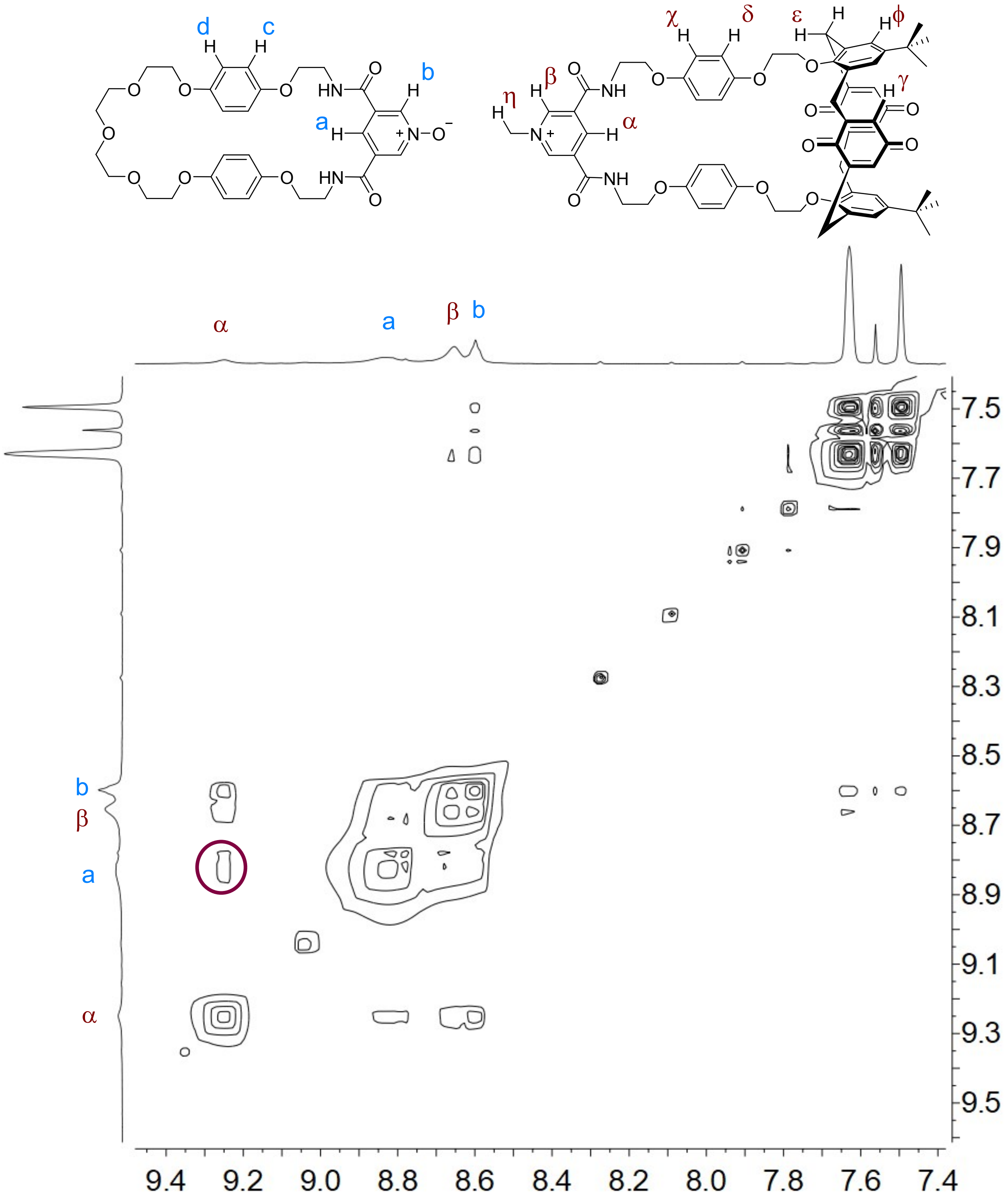
^{13}**C NMR** spectrum (126 MHz, $CDCl_3$/CD_3OD 1:1, 298 K)



^{1}H-^{1}H ROESY 2D Correlation NMR spectrum (500 MHz, $CDCl_3/CD_3OD$ 1:1, 298 K)



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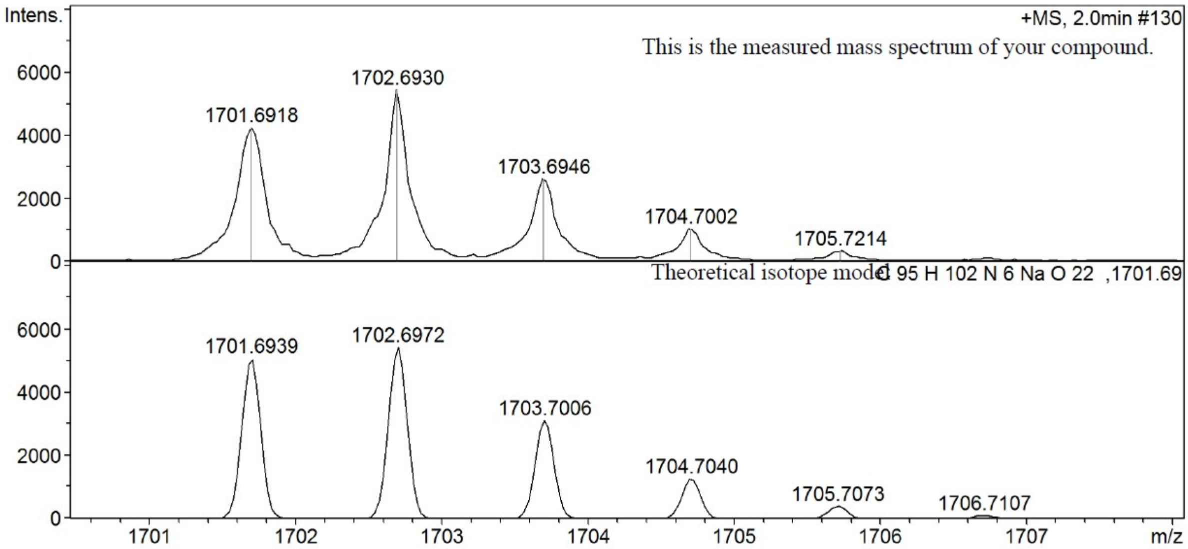


2. Single crystal X-ray crystallography

2.1 General Crystallographic Details

Data were collected using silicon double crystal monochromated synchrotron radiation (λ = 0.66890 Å) at Diamond Light Source, Beamline I19 on a custom built Rigaku diffractometer.

When using synchrotron radiation, ω-scans were performed such that a half-sphere of data was collected to a maximum resolution of 0.77 Å. Cell refinement, data reduction and scaling were performed using the CrystalClear package.[7]

The structures were solved by direct methods using the SIR92 software[8] or by charge flipping using Superflip.[9] Structures were refined using full-matrix least-squares on F^2 within the CRYSTALS suite.[10] All non-hydrogen atoms were refined with anisotropic displacement parameters unless specified otherwise. Disordered portions were modelled using refined partial occupancies. Geometric and vibrational restraints were applied where appropriate to ensure physically reasonable models.

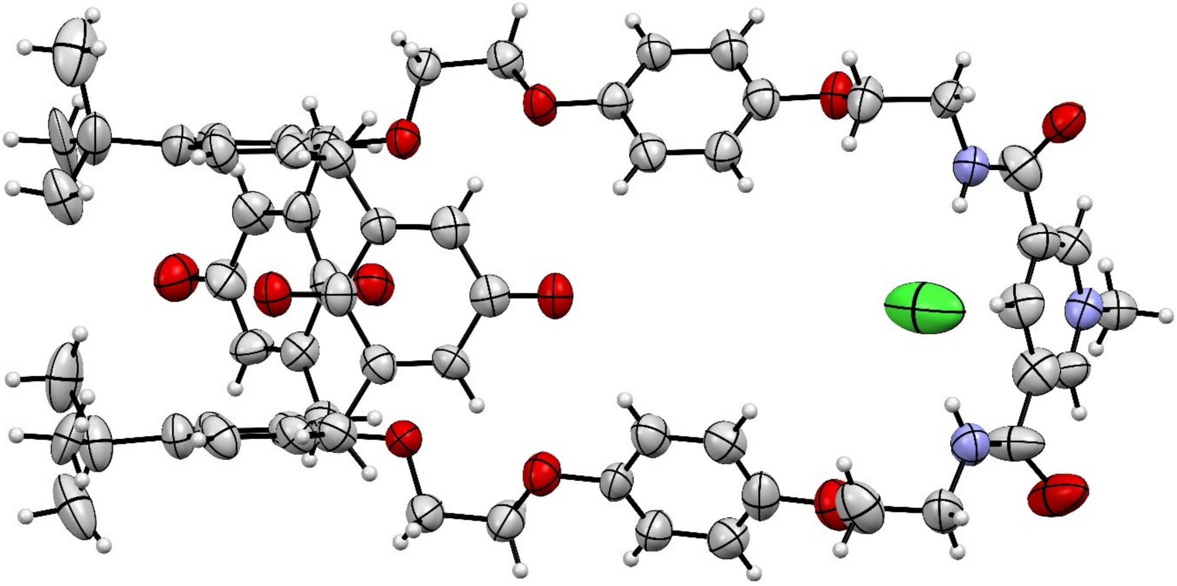
In some cases, the molecular structure within solvent voids could not be resolved in the difference map and PLATON SQUEEZE[11] was therefore used to account for the residual electron density in the refinement.

After the construction of a stable, physically reasonable and complete model, the weights were optimised,[10] analogous reflections were omitted and absent high-angle data (in the case of poorly diffracting samples) were pruned using the Wilson plot. This generally led to convergence of the refinement, giving the final structure. For the more challenging structures, in which the refinement did not converge immediately, initially half-shifts, then restraints and finally rigid body refinement were used to overcome the problem. IUCr

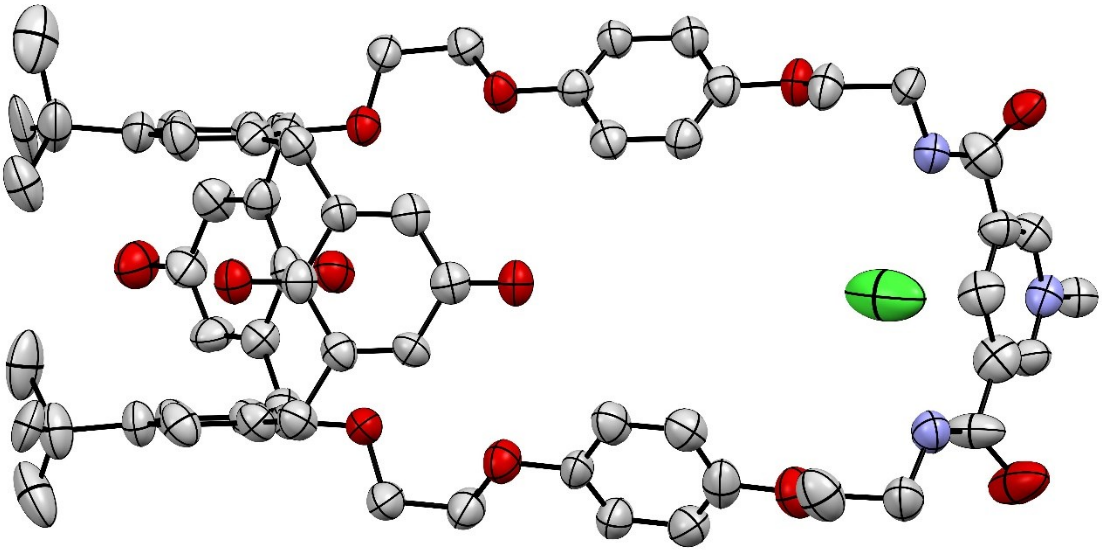
CheckCIF/PLATON[12] was used to validate the structures and warnings were dealt with as appropriate or justified using validation reply forms.

2.2 Single-crystal X-ray structure of Calix[4]diquinone pyridinium macrocycle 2·Cl (CCDC 1996546)

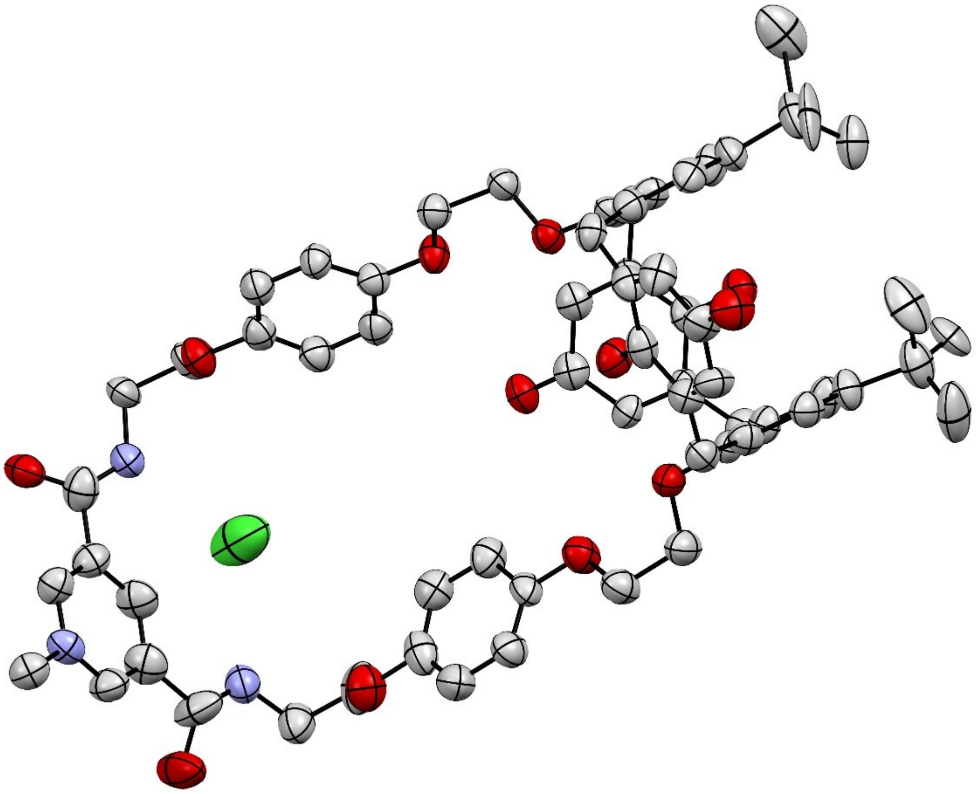
2.2.1 Single-crystal X-ray images of Calix[4]diquinone pyridinium macrocycle 2·Cl



Single crystal X-ray structure of calix[4]diquinone pyridinium macrocycle **2·Cl** (ellipsoids are plotted at the 50% probability level; disorder omitted for clarity)



Single crystal X-ray structure of calix[4]diquinone pyridinium macrocycle **2·Cl** (ellipsoids are plotted at the 50% probability level; disorder and hydrogen atoms omitted for clarity)



Single crystal X-ray structure of calix[4]diquinone pyridinium macrocycle **2·Cl** (ellipsoids are plotted at the 50% probability level; disorder and hydrogen atoms omitted for clarity)

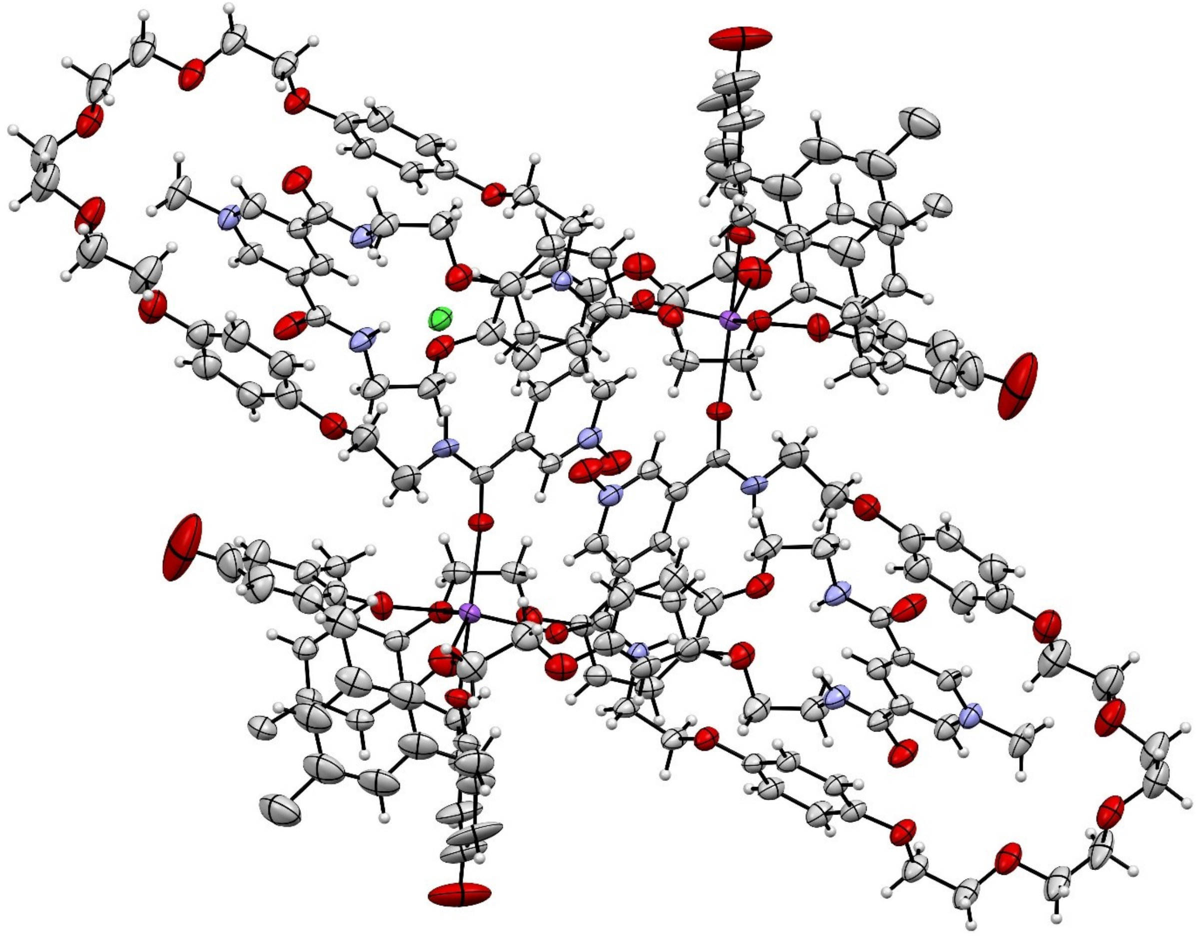
CCDC 1996546 contains the supplementary crystallographic data for this paper. These can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif. Single crystals of were grown from vapour diffusion of diisopropyl ether into a chloroform solution of the compound and excess TBA·Cl over several weeks.

2.2.2 Single-crystal X-ray data table of Calix[4]diquinone pyridinium macrocycle 2·Cl

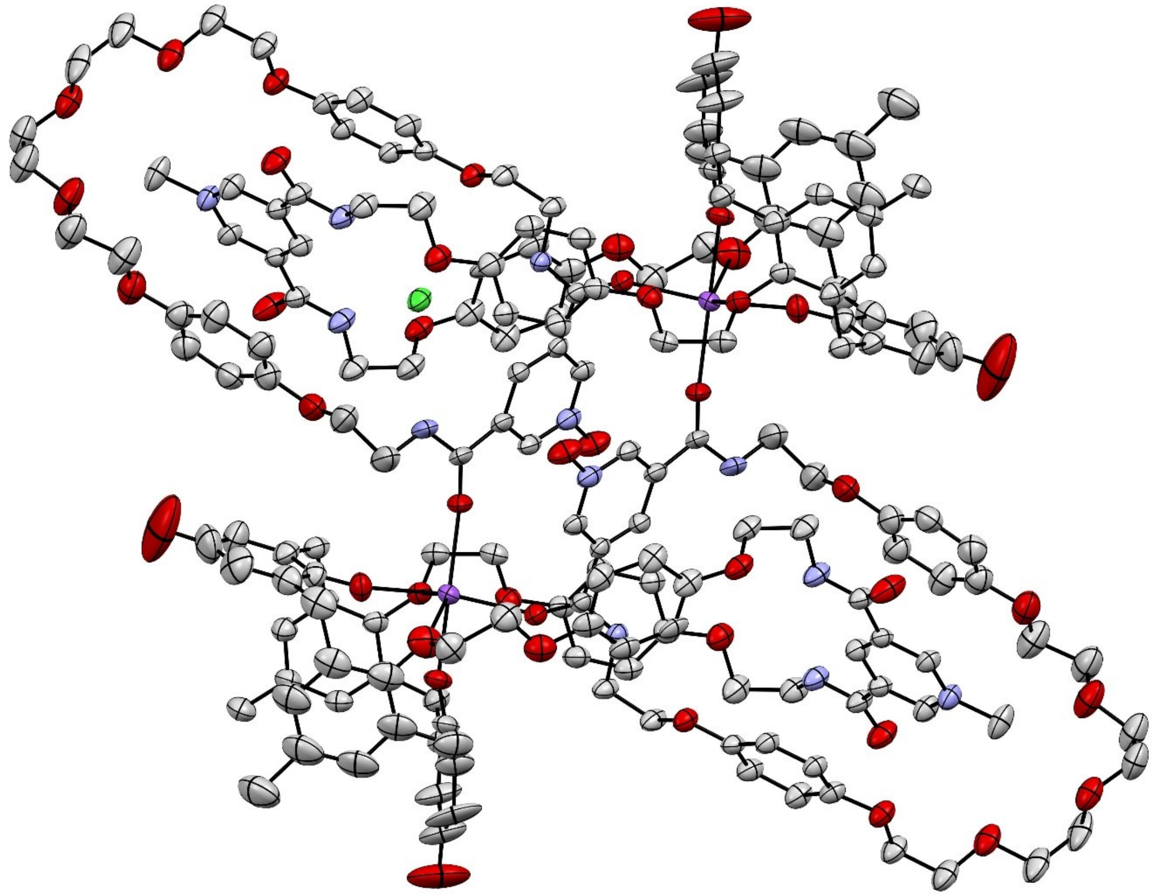
Compound reference	Calix[4]diquinone pyridinium macrocycle 2·Cl
Chemical formula	$C_{64}H_{65}N_3O_{12}\cdot Cl$
Formula Mass	1103.69
Crystal system	Orthorhombic
a/Å	11.2073(7)
b/Å	20.3291(15)
c/Å	25.6084(16)
α/°	90
β/°	90
γ/°	90
Unit cell volume/Å^3	5834.5(7)
Temperature/K	100
Space group	$P212121$
No. of formula units per unit cell, Z	4
No. of reflections measured	5144
No. of independent reflections	5059
R_{int}	0.135
Final R_1 values ($I > 2\sigma(I)$)	0.0635
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.0758
Final R_1 values (all data)	0.1071
Final $wR(F^2)$ values (all data)	0.0947

2.3 Single-crystal X-ray structure of Calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F_4$] (CCDC 1996545)

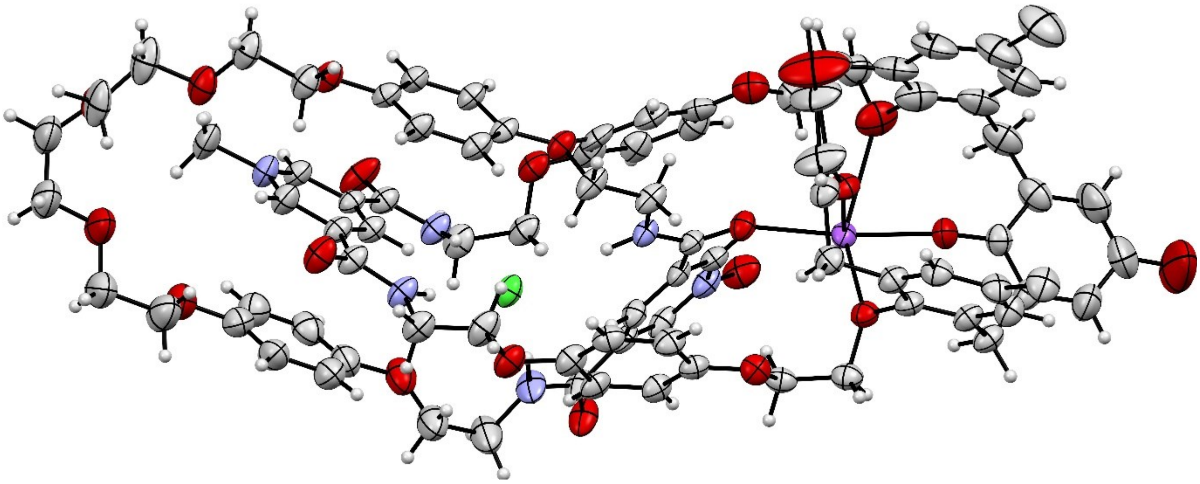
2.3.1 Single-crystal X-ray images of Calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F_4$]



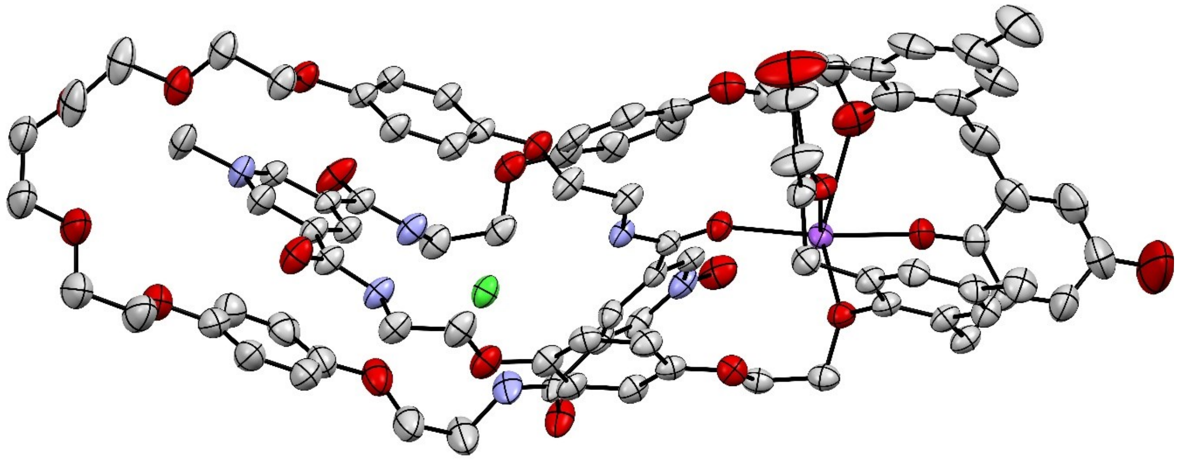
Single crystal X-ray structure of unit cell of calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F{}_4$] (ellipsoids are plotted at the 30% probability level; [BAr$^F{}_4$] counterion and disorder omitted for clarity)



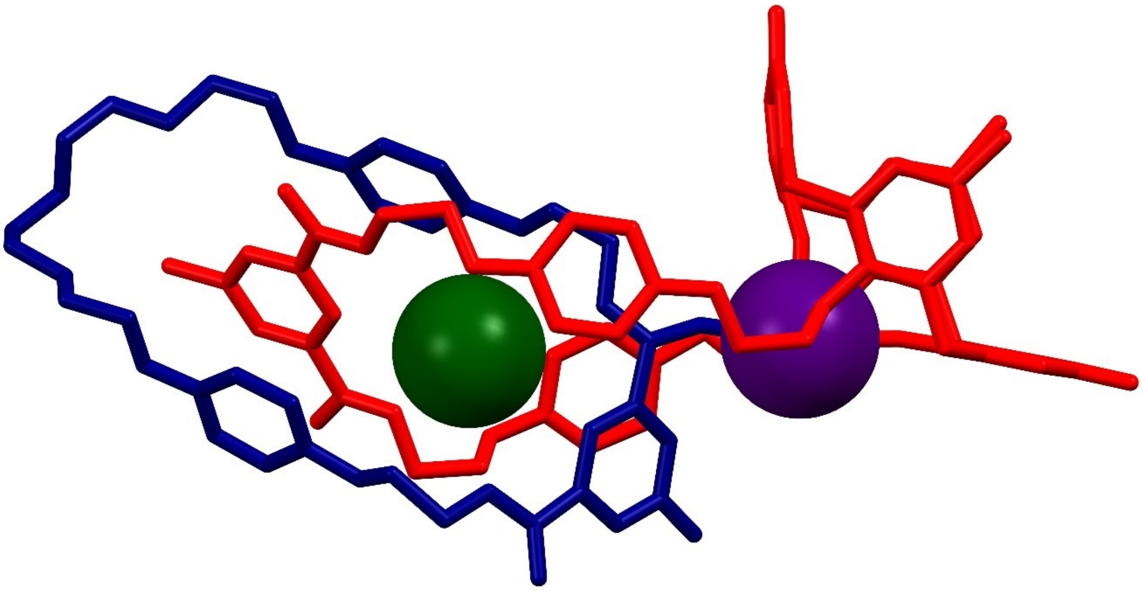
Single crystal X-ray structure of unit cell of calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F_4$] (ellipsoids are plotted at the 30% probability level; [BAr$^F_4$] counterion, disorder and hydrogen atoms omitted for clarity)



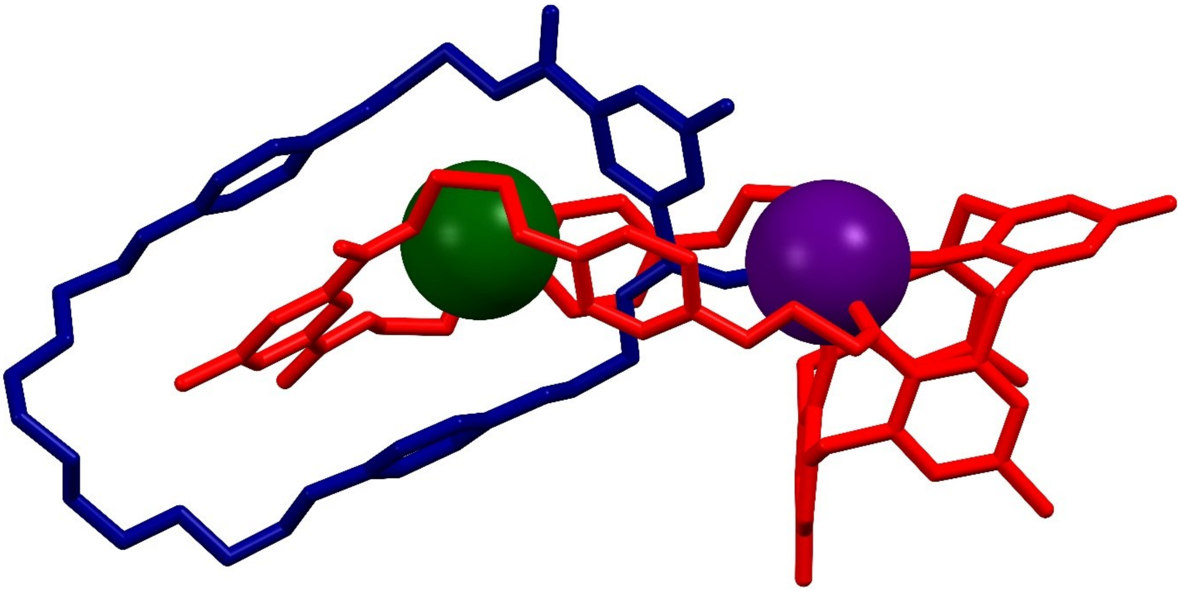
Single crystal X-ray structure of calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F_4$] (ellipsoids are plotted at the 30% probability level; [BAr$^F_4$] counterion and disorder omitted for clarity)



Single crystal X-ray structure of calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F{}_4$] (ellipsoids are plotted at the 30% probability level; [BAr$^F{}_4$] counterion, disorder and hydrogen atoms omitted for clarity)



Single crystal X-ray structure of calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F{}_4$] (stick representation; [BAr$^F{}_4$] counterion, disorder and hydrogen atoms omitted for clarity)



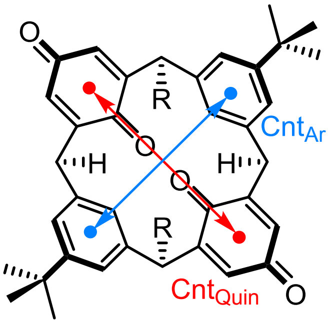
Single crystal X-ray structure of calix[4]diquinone pyridinium [2]catenane·NaCl·[BAr$^F_4$] (stick representation; tBu groups, [BAr$^F_4$] counterion, disorder and hydrogen atoms omitted for clarity)

CCDC 1996545 contains the supplementary crystallographic data for this paper. These can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif. Single crystals of were grown from vapour diffusion of diisopropyl ether into a chloroform solution of the compound over several months.

2.3.2 Single-crystal X-ray data table of Calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F{}_4$]

Compound reference	**Calix[4]diquinone pyridinium [2]catenane 8·NaCl·[BAr$^F{}_4$]**
Chemical formula	$C_{190}H_{204}N_{12}O_{44}\cdot\cdot 2Na2(C_{32}H_{12}BF_{24})\cdot 2(Cl)$
Formula Mass	2598.17
Crystal system	Triclinic
a/Å	13.0829(10)
b/Å	24.11(2)
c/Å	24.51(2)
α/°	109.487(15)
β/°	90.816(17)
γ/°	90.235(9)
Unit cell volume/Å^3	7287(9)
Temperature/K	100
Space group	*P***Error!**
No. of formula units per unit cell, *Z*	2
No. of reflections measured	52582
No. of independent reflections	52584
R_{int}	0.000
Final R_1 values ($I > 2\sigma(I)$)	0.1260
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.3149
Final R_1 values (all data)	0.1431
Final $wR(F^2)$ values (all data)	0.3454

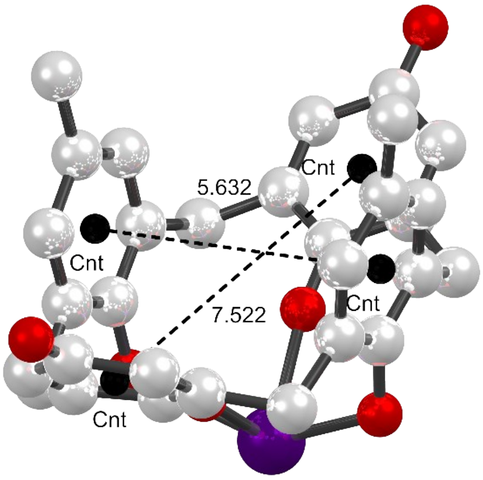
2.3.3 Calculation of Calix[4]diquinone Conformation



$$\mathrm{ROCD_{Cal}} = r(\mathrm{Cnt_{Ar}{-}{-}{-}Cnt_{Ar}})/r'(\mathrm{Cnt_{Quin}{-}{-}{-}Cnt_{Quin}})$$

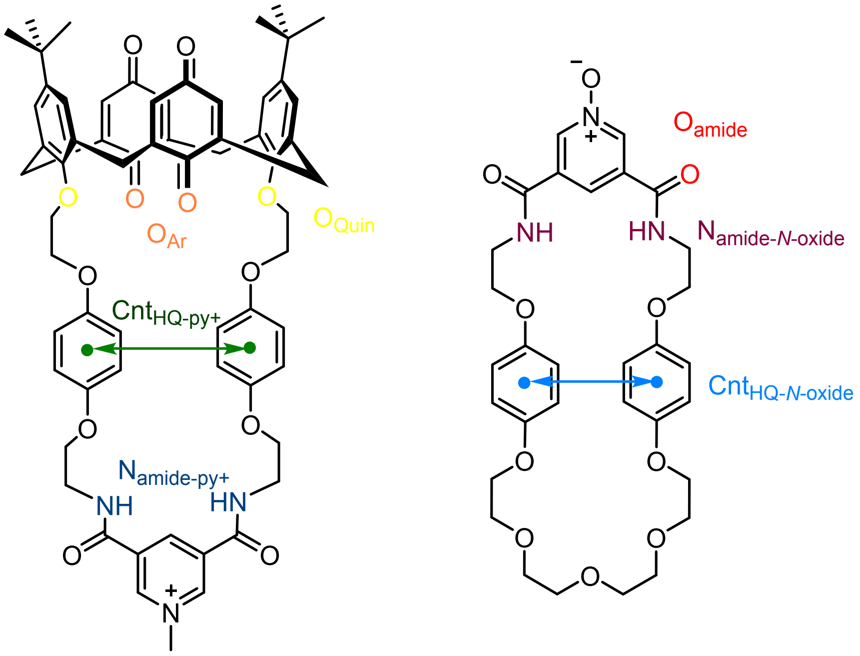
ROCD = ratio of opposing centroid distances

Metrics used to calculate the calix[4]diquinone conformation for [2]catenane **8·NaCl·[BAr$^F{}_4$]** in the solid-state (Ar = ^{t}butyl benzene, Quin = quinone)



Truncated single-crystal X-ray structure of [2]catenane **8·NaCl·[BAr$^F_4$]** with displayed r(Cnt---Cnt) distances for Cnt$_{Ar}$ and Cnt$_{Quin}$ (Ar = ^{t}butylphenol, Quin = quinone)[13]

2.4 Atom naming for Calix[4]diquinone pyridinium macrocycle 2·Cl and [2]catenane 8·NaCl·[BAr$^F_4$]



2.5 Selected Bond Lengths for Calix[4]diquinone pyridinium macrocycle 2·Cl and [2]catenane 8·NaCl·[BAr$^F_4$]

		2·Cl		8·NaCl·[BArF4]	
	Cl-N$_{amide-py+}$	3.307(11) Å	3.434(10) Å	3.209(7) Å	3.576(7) Å
Chloride	Cl-N$_{amide-Noxide}$	-		3.379(8) Å	3.309(8) Å
	Cl-C$_{Ar}$			3.329(7) Å	3.389(8) Å

Sodium	Na-O$_{\text{Ar}}$	-	2.570(5) Å	2.825(9) Å
	Na-O$_{\text{Quin}}$	-	2.274(6) Å	2.278(6) Å
	Na-O$_{\text{amide}}$	-	2.293(6) Å	2.334(6) Å
Centroids	Cnt$_{\text{HQ-}\textit{N-oxide}}$- Cnt$_{\text{HQ-}\textit{N-oxide}}$	7.220(5) Å	7.042(8) Å	
	Cnt$_{\text{HQ- py+}}$- Cnt$_{\text{HQ- py+}}$	-	7.712(8) Å	
	Cnt$_{\text{Ar}}$- Cnt$_{\text{Ar}}$	5.376(5) Å	5.632(7) Å	
	Cnt$_{\text{Quin}}$- Cnt$_{\text{Quin}}$	-	7.522(6) Å	
	ROCD$_{\text{Cal}}$	-	1.33(1) Å	

3. References

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