Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Electronic Supplementary Information

The Rapid Synthesis and Dynamic Behaviour of an Isophthalamide [2]Catenane

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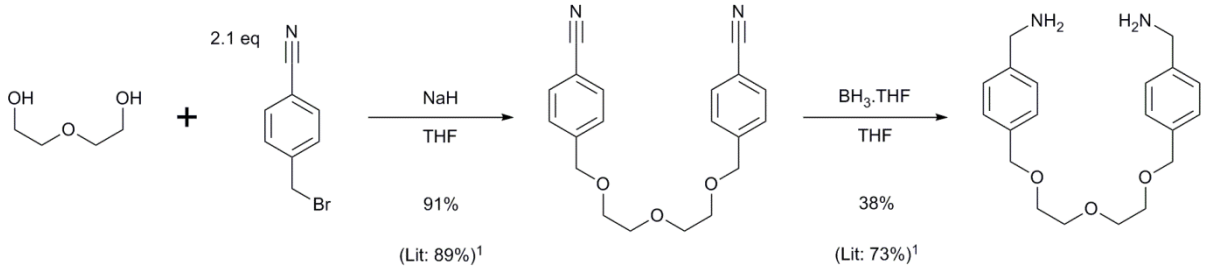
Contents

Part I: Synthesis

Additional Notes on Experimental Procedures

The dimethanamine used in the macrocyclisation was prepared following a literature procedure,[1] as summarized in the scheme below.

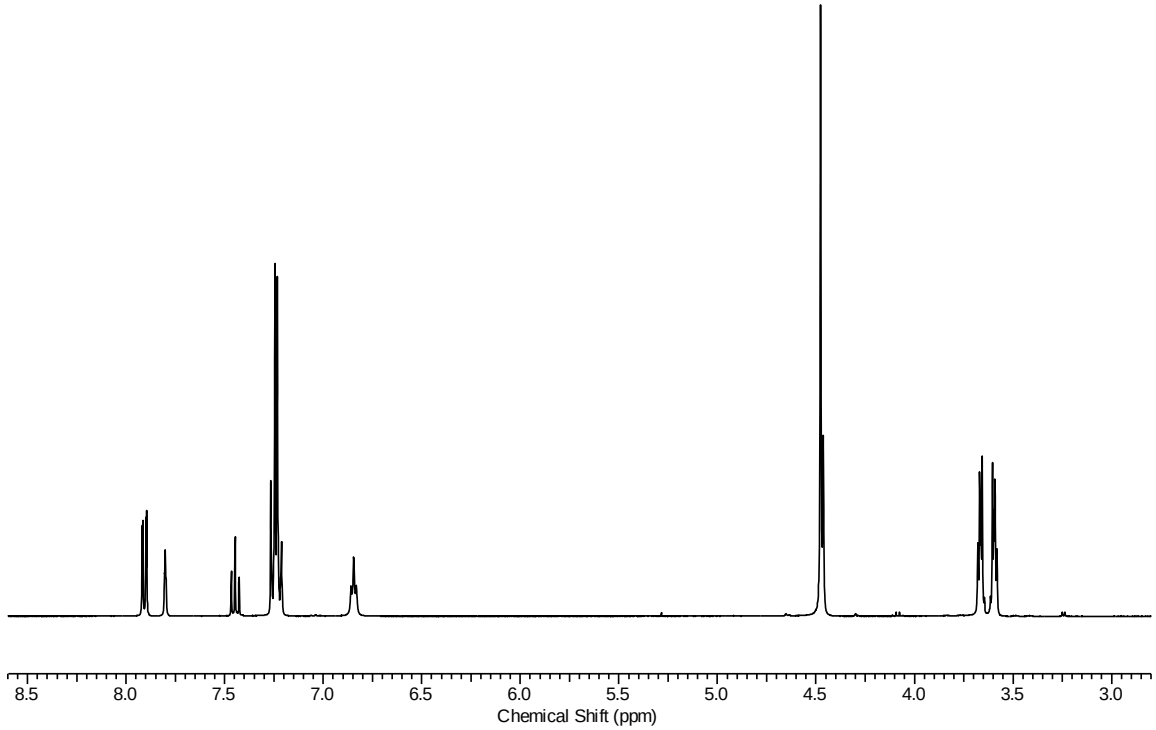


Our low yield of reduction (compared to the reported literature procedure) is attributed to retention of the bis-amine on the silica stationary phase used during column chromatographic purification.

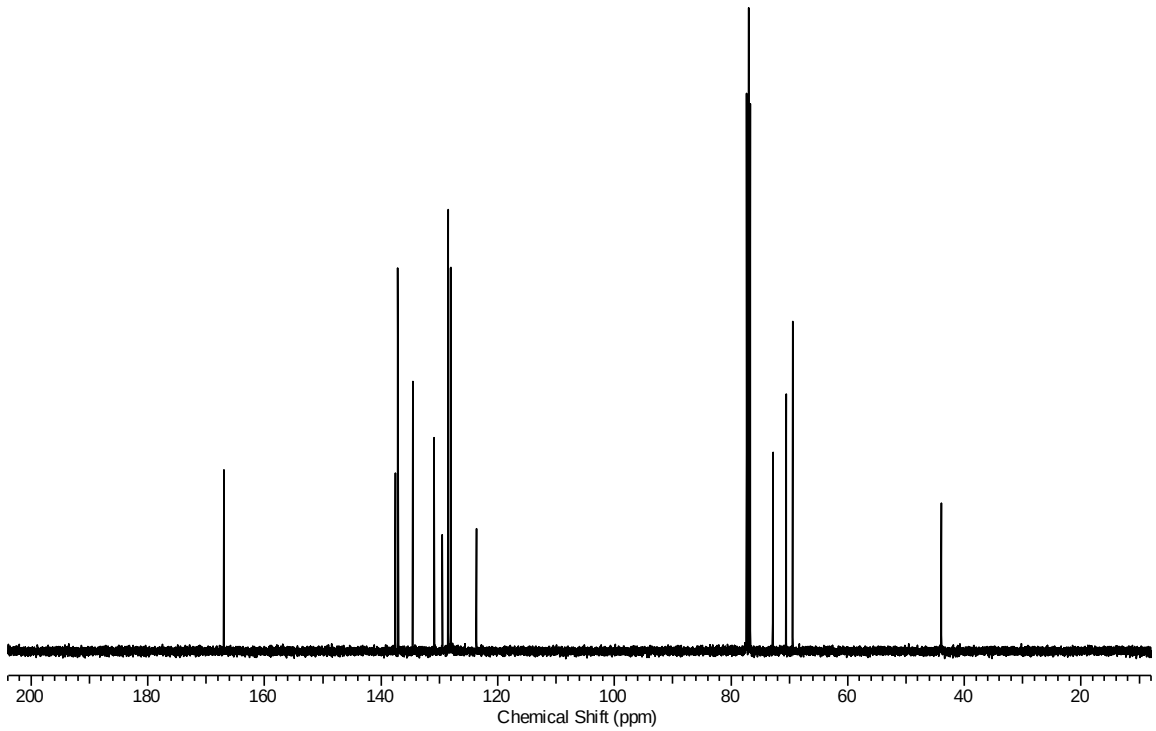
Part II: Spectral Characterisation

Macrocycle 1

^{1}H NMR ($CDCl_3$, 400 MHz)

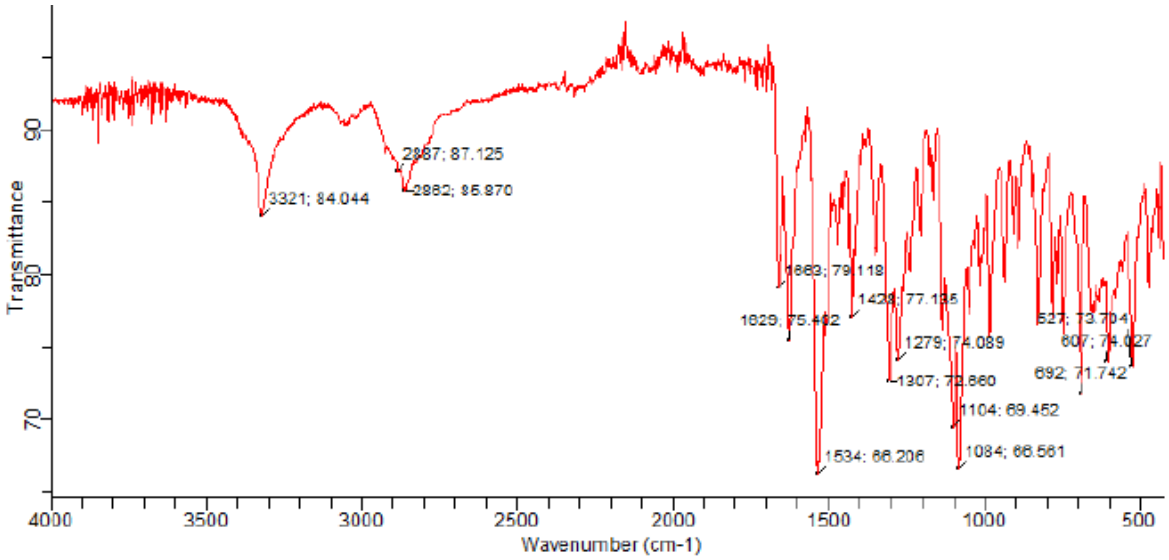


^{13}C NMR ($CDCl_3$, 100 MHz)

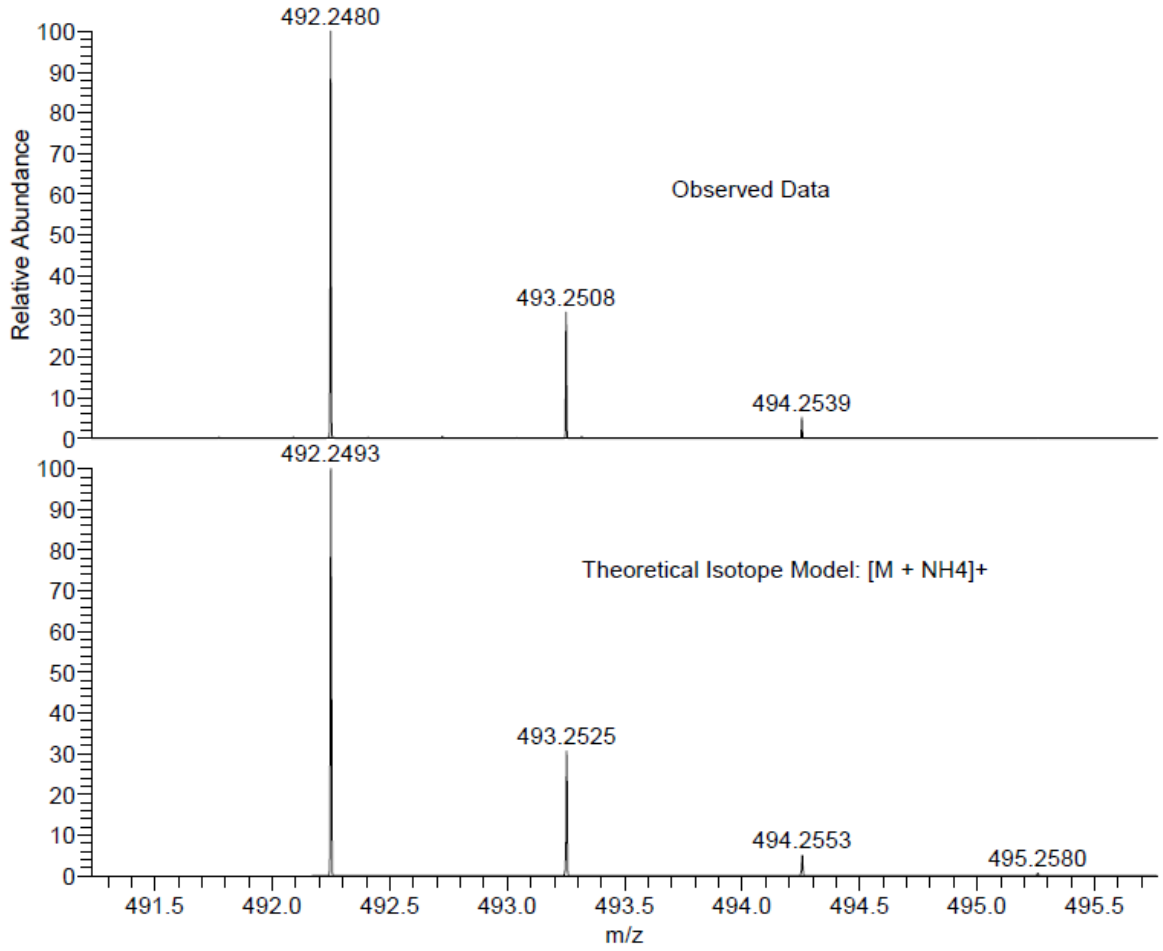


Macrocycle 1

IR Spectrum (neat)

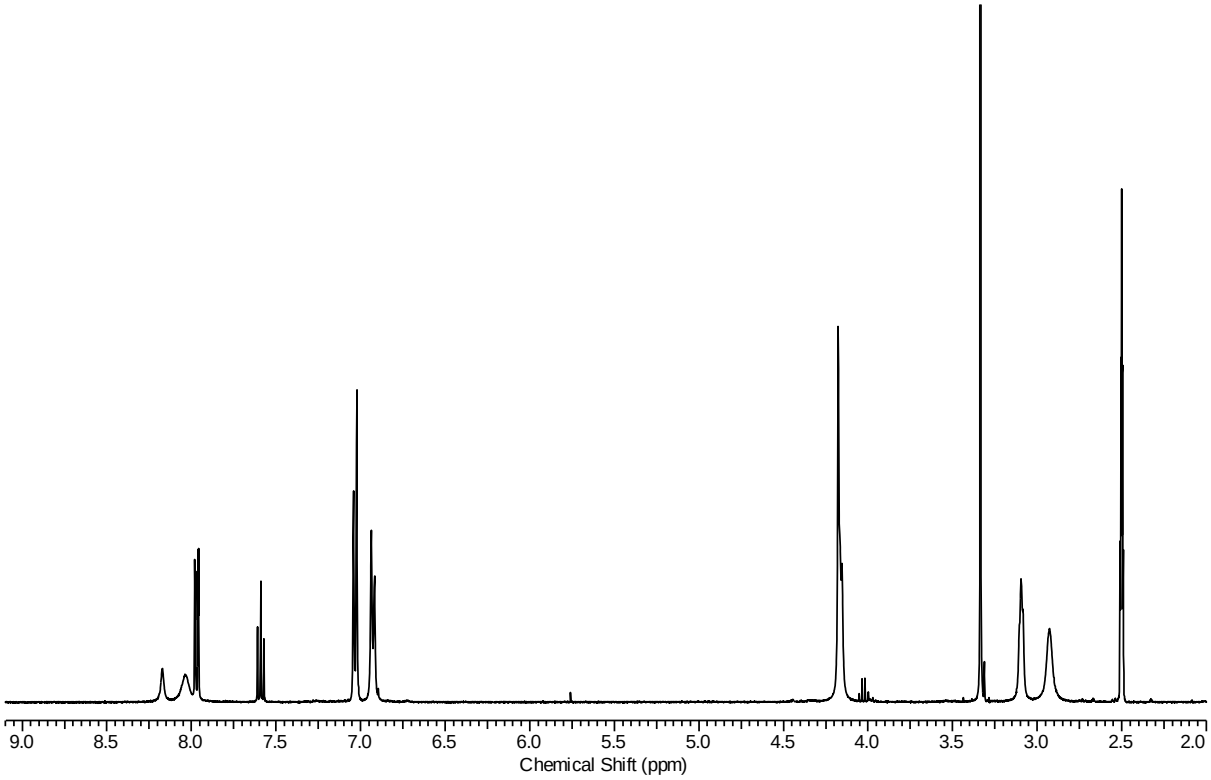


Mass Spectrum (ES +ve)

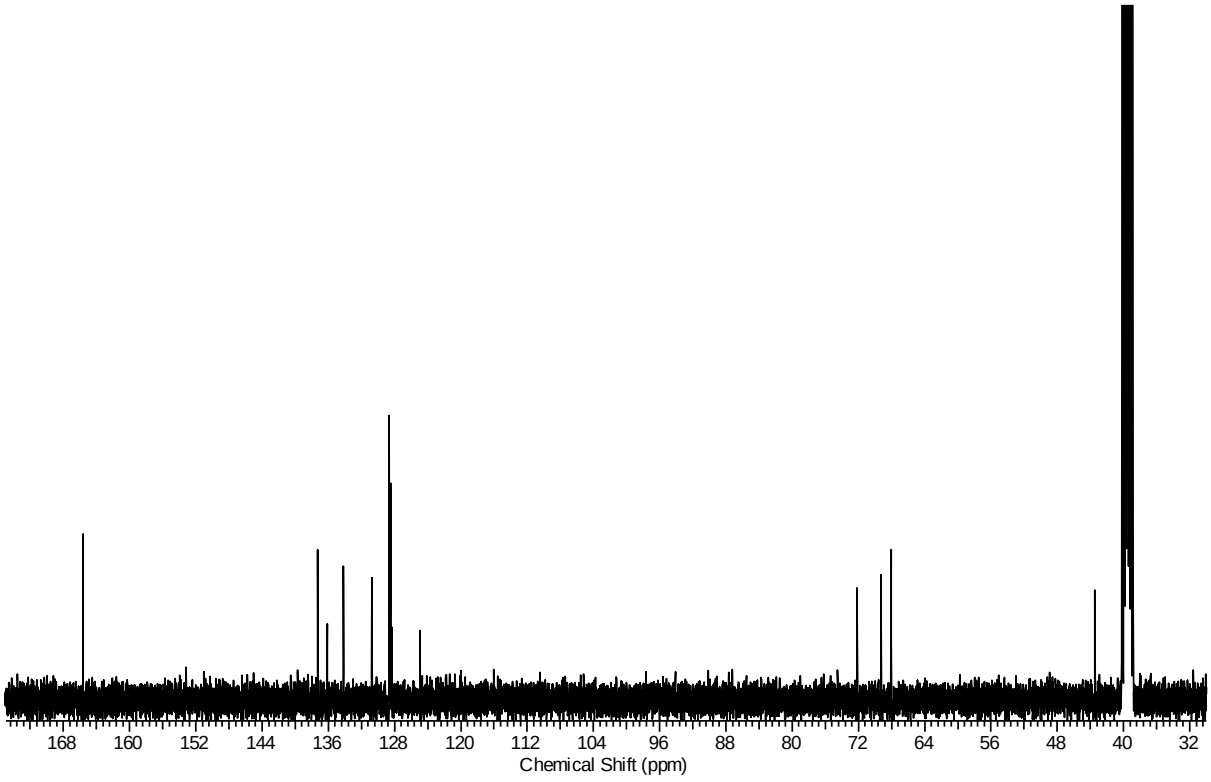


Catenane 2

^{1}H NMR (d_6-DMSO, 400 MHz)

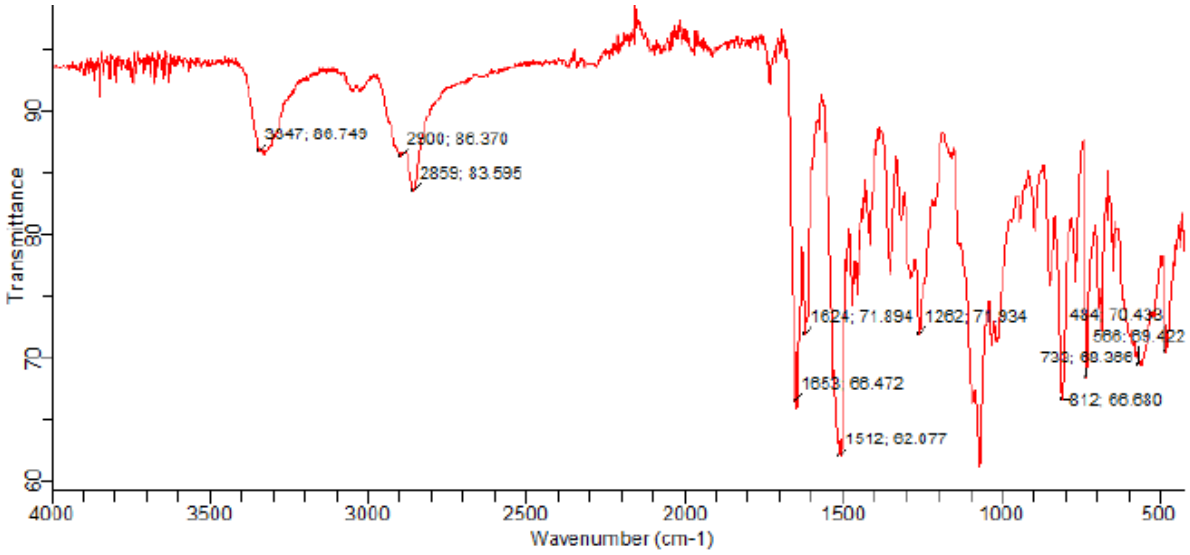


^{13}C NMR (d_6-DMSO, 100 MHz)

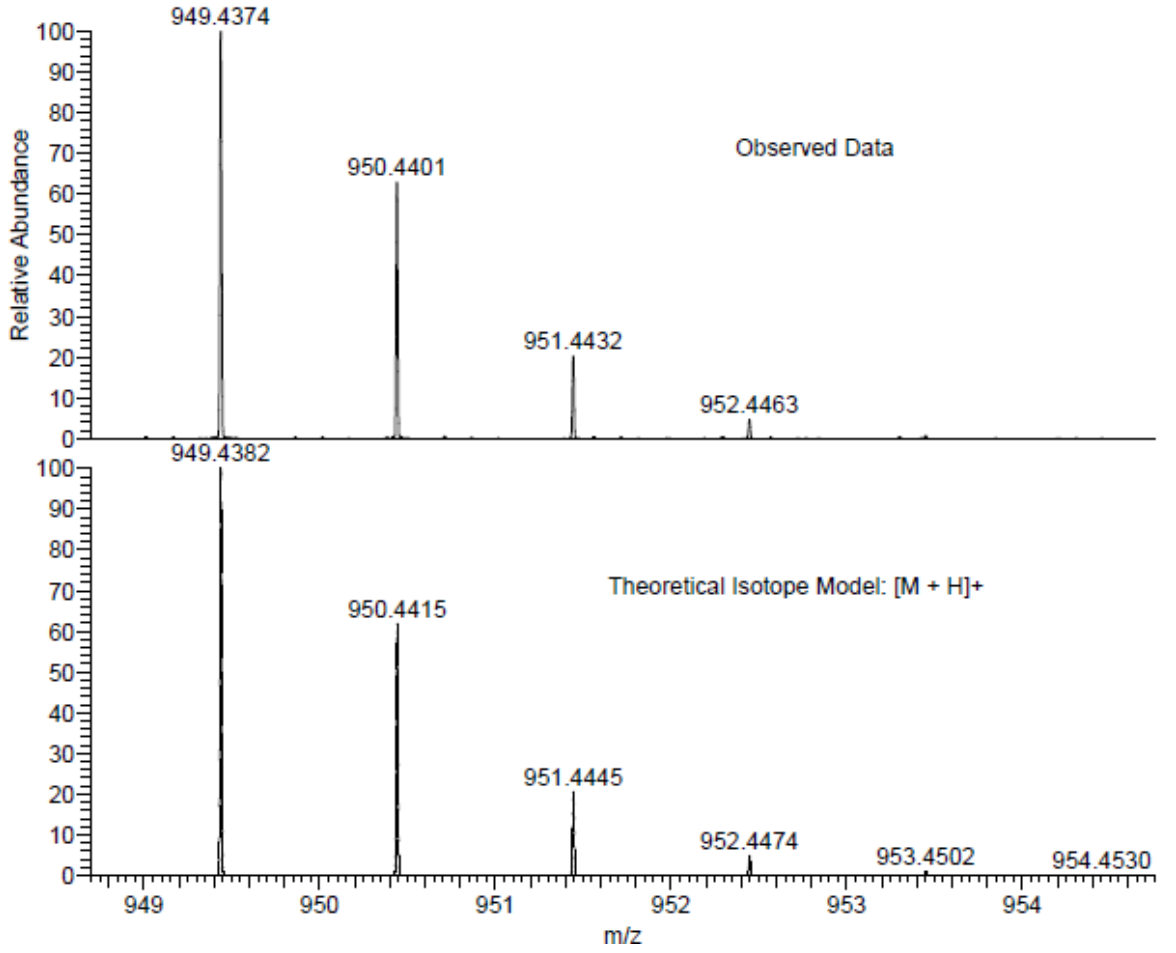


Catenane 2

IR Spectrum (neat)



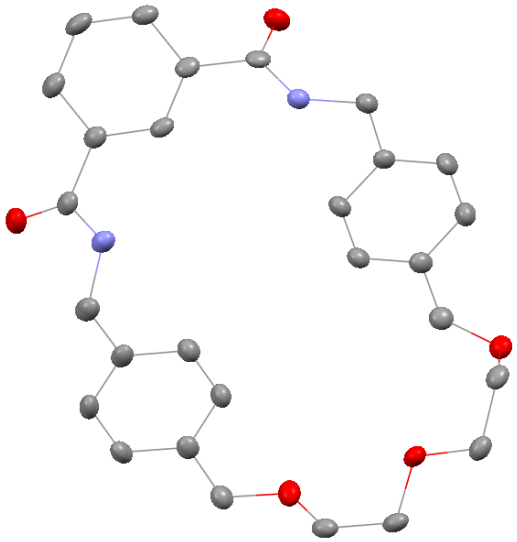
Mass Spectrum (ES +ve)



Part III: Crystallographic Data

Macrocycle 1

Single crystals of macrocycle **1** were grown by slow evaporation of a chloroform solution. A suitable crystal was selected and studied using an Agilent SuperNova AtlasS2 diffractometer. Using Olex2,[3] the structure was solved with the ShelXS[4] structure solution program using Direct Methods and refined with the ShelXL[4] refinement package using Least Squares minimisation.



*X-ray crystal structure of macrocycle **1**. Thermal ellipsoids are displayed at 50% probability.*

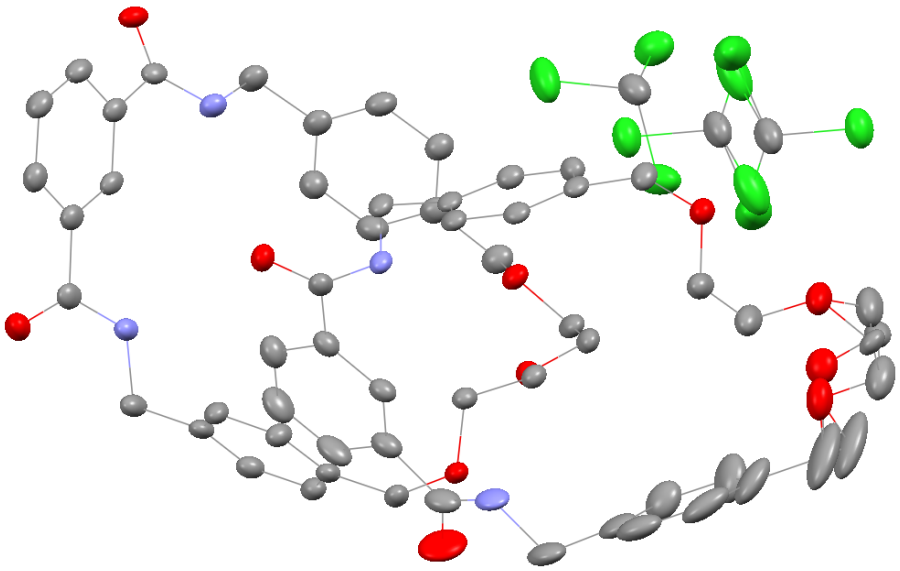
*Crystal data and structural refinement for macrocycle **1**:*

CCDC number	1416976
Empirical formula	$C_{28}H_{28}O_7N_{0.25}$
Formula weight	474.56
Temperature/K	99.97(10)
Crystal system	orthorhombic
Space group	$Pca2_1$
a/Å	10.6388(3)
b/Å	9.0437(3)
c/Å	24.8594(8)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2391.83(13)
Z	4

ρ_{calc}g/cm^3	1.3178
μ/mm^{-1}	0.736
F(000)	1011.3
Crystal size/mm^3	0.2 × 0.2 × 0.2
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	12.1 to 146.22
Index ranges	$-13 \leq h \leq 11$, $-10 \leq k \leq 7$, $-25 \leq l \leq 30$
Reflections collected	5121
Independent reflections	3287 [R_{int} = 0.0227, R_{sigma} = 0.0318]
Data/restraints/parameters	3287/0/323
Goodness-of-fit on F^2	0.959
Final R indexes [I>=2σ (I)]	R_1 = 0.0292, wR_2 = 0.0759
Final R indexes [all data]	R_1 = 0.0302, wR_2 = 0.0774
Largest diff. peak/hole / e Å^{-3}	0.20/-0.16
Flack parameter	0.11(16)

Catenane 2

Single crystals of catenane **2** were by slow evaporation of a chloroform solution. A suitable crystal was selected and studied on an Agilent SuperNova AtlasS2 diffractometer. Using Olex2,[3] the structure was solved with the ShelXS[4] structure solution program using Direct Methods and refined with the ShelXL[4] refinement package using Least Squares minimisation.



*X-ray crystal structure of catenane **2**. Thermal ellipsoids are displayed at 50% probability.*

*Crystal data and structural refinement for catenane **2**:*

CCDC number	1416977
Empirical formula	$C_{60}H_{69}N_3O_{13}Cl_{0.25}$
Formula weight	1049.04
Temperature/K	99.98(10)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	21.3403(9)
b/Å	10.9688(3)
c/Å	25.5657(10)
α/°	90.00
β/°	113.174(5)
γ/°	90.00
Volume/Å^3	5501.5(4)
Z	4
ρ_{calc}g/cm^3	1.267
μ/mm^{-1}	0.834

F(000)	2233.0
Crystal size/mm^3	0.2 × 0.1 × 0.1
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.9 to 145.96
Index ranges	$-14 \leq h \leq 26$, $-12 \leq k \leq 13$, $-31 \leq l \leq 31$
Reflections collected	18667
Independent reflections	10501 [R_{int} = 0.0472, R_{sigma} = 0.0623]
Data/restraints/parameters	10501/4/736
Goodness-of-fit on F^2	1.016
Final R indexes [I>=2σ (I)]	R_1 = 0.0511, wR_2 = 0.1080
Final R indexes [all data]	R_1 = 0.0845, wR_2 = 0.1244
Largest diff. peak/hole / e $Å^{-3}$	0.54/-0.46

Part IV: References

1) A. Vidonne and D. Philp, *Tetrahedron*, 2008, **64**, 8464-8475.
2) O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Cryst.*, 2009, **42**, 339-341.
3) G. M. Sheldrick, *Acta Cryst. A*, 2008, **64**, 112-122.