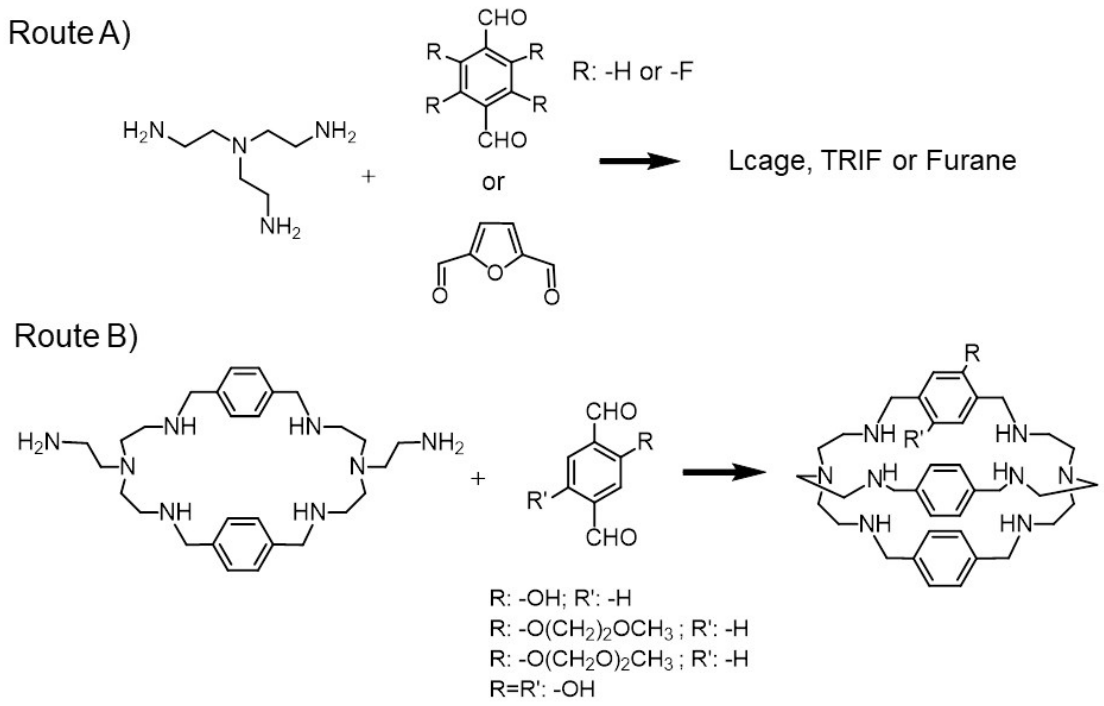
Electronic Supplementary Material (ESI) for Dalton Transactions.

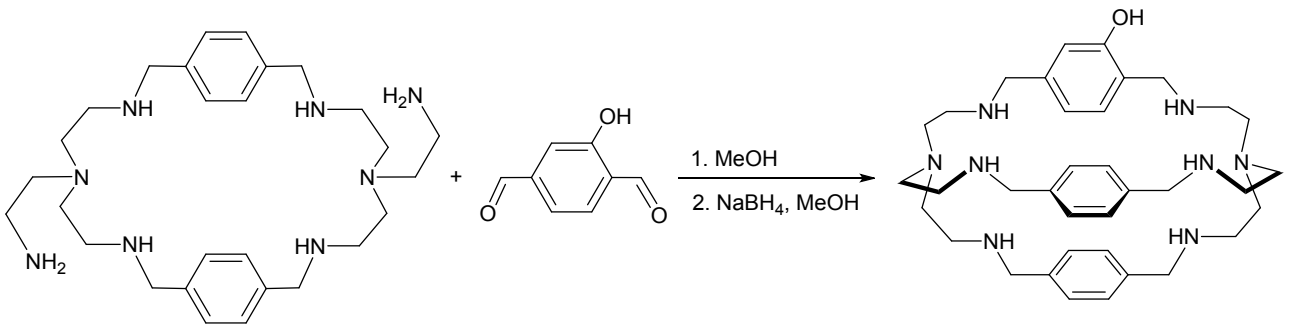
SUPPLEMENTARY INFORMATION

1. Syntheses of azacryptands

For the syntheses of azacryptands, two different approaches were followed (see Route A and B in the Scheme below).[1] Route A) consisted in the Schiff base condensation of *tren* with the chosen dialdehyde, mixed in a 2:3 molar ratio. The obtained polyimine intermediate product was then reduced to the corresponding polyamine with either $NaBH_4$ or $NaBH_3CN$. This method was employed in the syntheses of Lcage, Furane and TRIF. In the case of TRIF, Ag(I) was employed as templating agent in the Schiff condensation step. Route B) consisted in the reaction of a p-xylyl-based macrocyclic compound with the chosen dialdehyde. As for Route A), the polyimine intermediate was then reduced with $NaBH_4$ or $NaBH_3CN$, to obtain the final azacryptand. Route B) was followed for MOH, 2OH, 1-ARM and PEG cages.



1.1. Synthesis of MOH



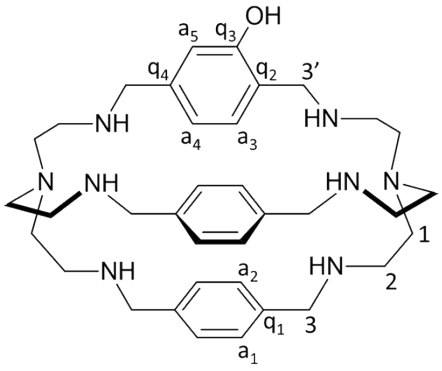
$C_{28}H_{48}N_8$
MW = 496.73

$C_8H_6O_3$
MW = 150.13

$C_{36}H_{54}N_8O$
MW = 614.87

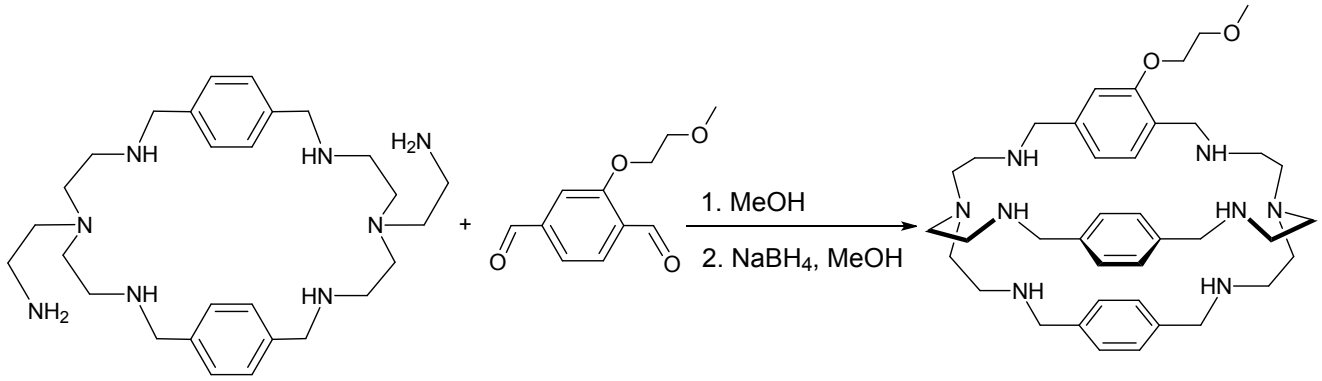
A solution of 2-hydroxy-1,4-benzenedicarboxaldehyde[1] (0.256 g, 1.71 mmol) in MeOH (100 mL) was added dropwise, under vigorous stirring, to a solution of the preformed macrocycle[2] (0.85 g, 1.71 mmol) in MeOH (200 mL). The mixture was then stirred at room temperature for 24 h. The reaction advancement was monitored through ESI-MS spectrometry.

The product of the Schiff condensation was then reduced by addition of $NaBH_4$ in excess (1.3 g, 34 mmol) to the reaction mixture, in small portions under reflux. After 4 h, an additional portion of $NaBH_4$ was added and the mixture was stirred overnight at 50°C. The solvent was finally removed under reduced pressure, 30 mL of brine was added and the aqueous phase was extracted with dichloromethane (7x30 mL). The collected organic phases were dried over anhydrous Na_2SO_4. The product is an orange oil, that becomes a yellowish filterable solid (0.66 g; yield: 63%) under treatment with diethyl ether.



ESI-MS in MeOH, m/z: 308.66 $[M + 2H]^{2+}$, 616.76 $[M + H]^{+}$. ^{1}H-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm: 7.39 (s, 8H, **a1-a2**), 7.24 (m, 1H, **a3**), 6.88 (d, 1H, **a4**), 6.86 (s, 1H, **a5**), 4.17 (m, 12H, **3**), 3.11 (m, 12H, **2**), 2.71 (m, 12H, **1**).^{13}C-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm: 155.47 (**q3**), 133.31 (**q4**), 132.48 (**a3**), 131.50 (**q1**), 130.65 (**a1-a2**), 124.31-114.85 (q, δ_m = 119.58 $^{1}J_{C\text{-}F}$ = 317.33 Hz, -CF_3 group of triflic acid), 121.57 (**a4**), 118.38 (**q2**), 113.98 (**a5**), 70.55 (**5**), 67.78 (**4**), 58 30 (**6**), 51.28 (**3**), 50.10 (**1**), 47.30 (**3'**), 44.62(**2**).

1.2. Synthesis of 1-ARM

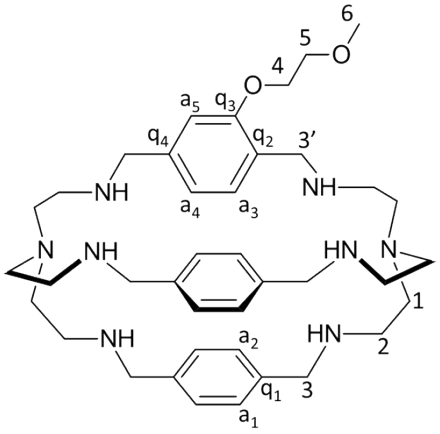


$C_{28}H_{48}N_8$
MW = 496.73

$C_{11}H_{12}O_4$
MW = 208.21

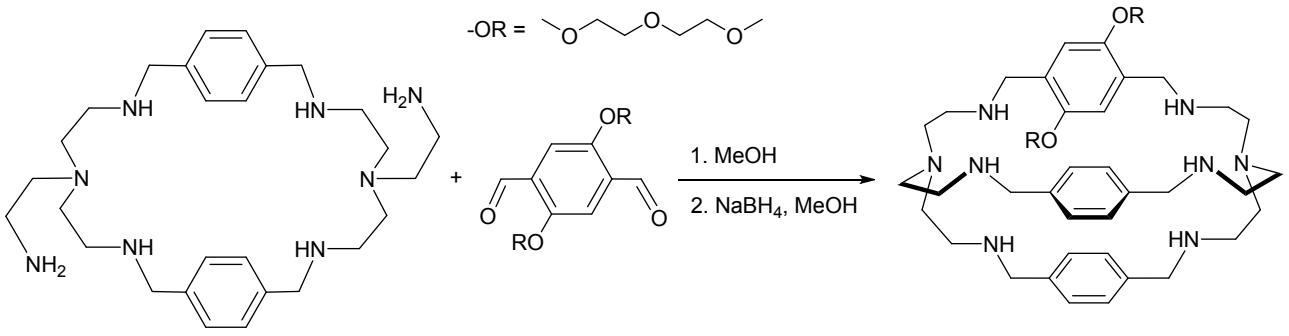
$C_{39}H_{60}N_8O_2$
MW = 672.95

A solution of 2-(2-methoxyethoxy)-1,4-benzenedicarboxaldehyde[3] (0.19 g, 0.9 mmol) in 100 mL MeOH was added dropwise, under vigorous stirring, to a solution of the preformed macrocycle[2] (0.453 g, 0.9 mmol) in 250 mL MeOH. The mixture was stirred at room temperature, under inert atmosphere for 24 h. The polyimine product was then reduced under reflux by addition of $NaBH_4$ in excess (0.7 g, 18 mmol). The reaction advancement was monitored through ESI-MS spectrometry. The reducing agent was added in small portions. After reacting under stirring overnight at 50°C, the solvent was removed by evaporation under reduced pressure and 20 mL of brine were added. The aqueous phase was extracted with DCM (7x30 mL) and the collected organic phases were dried over anhydrous Na_2SO_4. The polyamine product was purified by precipitation as polyammonium salt. This latter was obtained by treatment of a solution of the azacryptand in ethanol with nitric acid (0.55 g; yield 91%).



ESI-MS in MeOH, m/z: 337.55 $[M+2H]^{2+}$, 673.55 $[M+H]^{+}$. ^{1}H-NMR (400 MHz) in D_2O + CF_3SO_3H , ppm: 7.39 (s, 8H, **a1+a2**); 7.30 (d, 1H, **a3**); 7.04 (s, 1H, **a5**); 6.98 (d, 1H, **a4**); 4.20 (m, 12H + 2H, **3 + 4**), 3.84 (m, 2H, **5**), 3.39 (m, 3H, **6**); 3.12 (m, 12H, **2**); 2.71 (m, 12H, **1**).^{13}C-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm: 157.22 (**q3**), 133.64 (**q4**), 132.61 (**a3**), 131.49 (**q1**), 130.59 (**a1+a2**), 124.32-114.86 (q, δ_m = 119.60, $^1J_{C-F}$ = 317.42 Hz, -CF_3 group of triflic acid), 122.76 (**a4**), 121.17 (**q2**), 113.98 (**a5**), 70.55 (**5**), 67.78 (**4**), 58.33 (**6**), 51.28 (**3**), 50.10 (**1**), 47.30 (**3'**), 44.75(**2**).

1.3. Synthesis of PEG



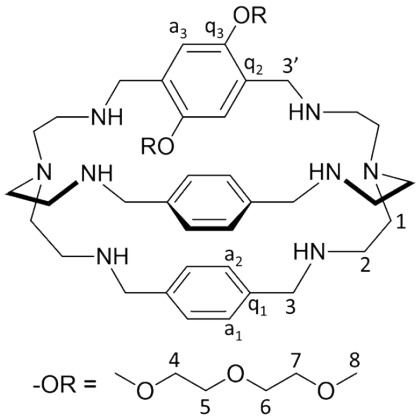
$C_{28}H_{48}N_8$
MW = 496.73

$C_{18}H_{26}O_8$
MW = 370.39

$C_{46}H_{74}N_8O_6$
MW = 835.13

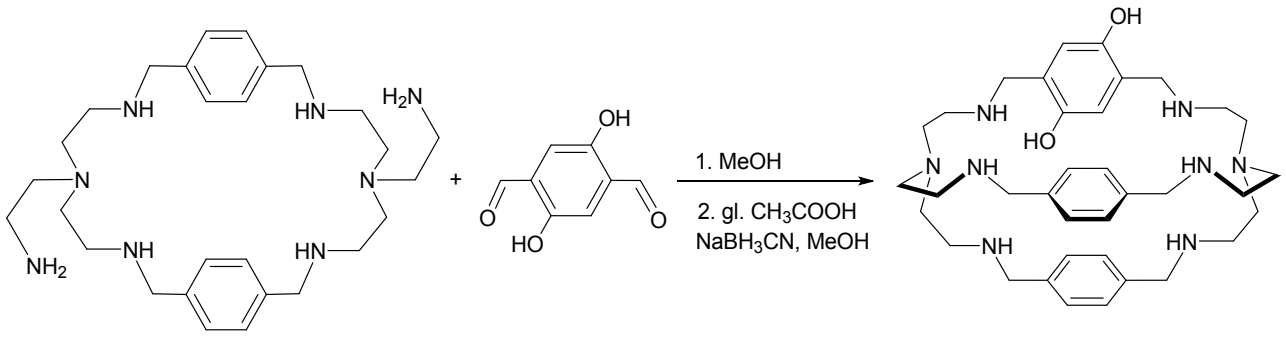
A solution of 2,5-bis(2-methoxyethoxy)-1,4-benzenedicarboxaldehyde[4] (0.3064 g, 0.827 mmol) in 100 mL MeOH was added dropwise, under vigorous stirring, to a solution of the preformed macrocycle[2](0.41 g, 0.825 mmol) in 250 mL MeOH. The mixture was stirred at room temperature, under inert atmosphere for 24 h. The reaction advancement was monitored through ESI-MS spectrometry.

The reduction of the polyimine intermediate product was obtained by addition of $NaBH_4$ in excess (0.7 g, 18.5 mmol) to the reaction mixture at 50°C. After stirring overnight, an additional portion of $NaBH_4$ was added to the solution. The solvent was then evaporated under reduced pressure and the residue was dissolved in brine. The aqueous phase was then extracted with DCM (7x30 mL) and the collected organic phases were then dried over anhydrous Na_2SO_4. The final product is a gluey yellow solid (0.66 g; yield: 95%).



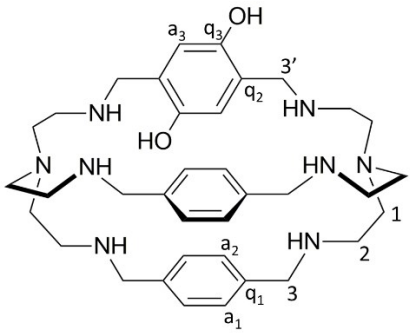
ESI-MS in MeOH, m/z: 419.27 $[M+2H]^{2+}$, 835.91 $[M+H]^{+}$. ^{1}H-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm: 7.36 (s, 8H, **a1+a2**); 7.00 (s, 2H, **a3**); 4.20 (m, 16H, **3 + 4**); 3.80 (m, 4H, **5**); 3.64 (m, 4H, **6**); 3.52 (m, 4H, **7**); 3.24 (s, 6H, **8**); 3.09 (m, 12H, **2**), 2.69 (m, 12H, **1**). ^{13}C-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm:151.3 (**q3**), 131.50 (**q1**), 130.54 (**a1+a2**), 124.33-114.87 (q, δ_m = 119.58, $^{1}J_{C-F}$ = 317.33 Hz, -CF_3 group of triflic acid), 121.89 (**q2**), 117.27(**a3**), 70.76 (**7**), 69.53 (**6**), 68.94 (**5**), 68.77 (**4**), 58.02 (**8**), 51.23 (**3**), 50.03 (**1**), 47.53 (**3'**), 45 (**2**).

1.4. Synthesis of 2-OH



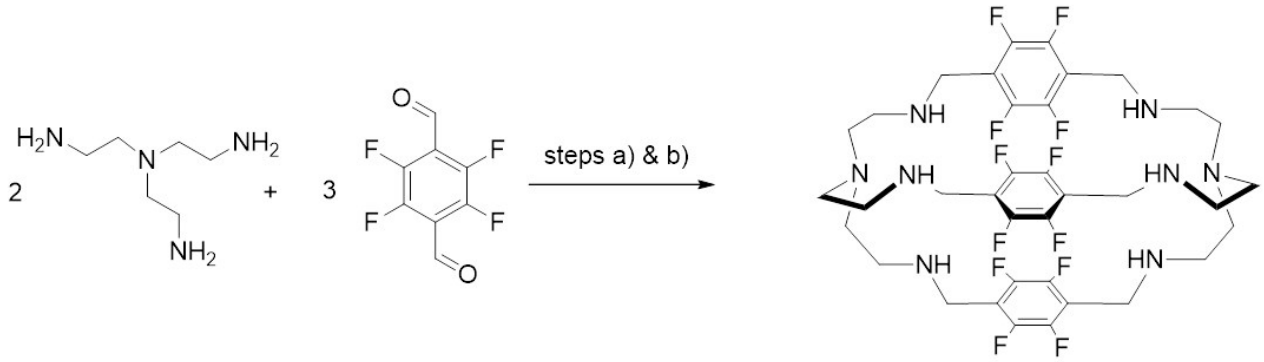
$C_{28}H_{48}N_8$ MW = 496.73 | $C_8H_6O_4$ MW = 166.13 | $C_{36}H_{54}N_8O_2$ MW = 630.87

A solution of 2,5-bishydroxy-1,4-benzenedicarboxaldehyde[4] (0.340 g, 2.0 mmol) in 250 mL MeOH was added dropwise, under vigorous stirring, to a solution of the preformed macrocycle[2](1.01 g, 2.0 mmol) in 150 mL MeOH. The mixture was stirred at room temperature, under inert atmosphere for three days. The reaction advancement was monitored through ESI-MS spectrometry.
The reaction mixture was concentrated under reduced pressure and 20 mL of glacial CH_3COOH were added; reduction of the imine bonds was obtained by addition of $NaBH_3CN$ in excess (2.52 g, 40 mmol). The reaction mixture was stirred overnight at 50°C. MeOH was finally evaporated under reduced pressure and the aqueous phase was basified with NaOH. The basic aqueous solution was then extracted with DCM (5x 50 mL). The collected organic phases were dried over anhydrous Na_2SO_4. The crude azacryptand (0.8 g; yield 63%) is a red oil that becomes a filterable solid by drying under vacuum. The product was purified by preparative HPLC on an Agilent system SERIES 1260 combining a preparative PUMP, a Diode array system and an automatic fraction Collector. The preparative column was XSelect CSH Prep Phenyl-Hexyl 5µm (150 x 30 mm, Waters). The solvent used for the HPLC purification was a mixture 0.1 % trifluoroacetic acid in acetonitrile/water (gradient elution: from 5% to 70% v/v acetonitrile/water). The purification yielded 0.19 g of 2OH (yield after purification: 15%).



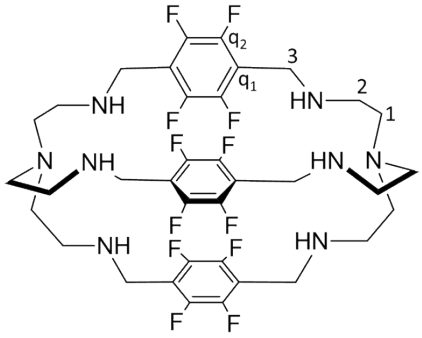
ESI-MS in MeOH, m/z: 316.39 $[M + 2H]^{2+}$, 631.42 $[M + H]^{+}$. ^{1}H-NMR (400 MHz) in CD_3OD, ppm: 7.55 (s, 8H, **a2+a1**), 6.87 (s, 2H, **a3**), 4.27 (m, 12H, **3**), 3.21 (m, 12H, **2**), 2.85 (m, 12H, **1**), 2.05 (residual CH_3CN). ^{13}C-NMR (100 MHz) in CD_3OD, ppm: 161.37 (-CF_3 group of TFA acid), 148.90 (**q3**), 132.15 (**q1**), 130.37 (**a1+a2**), 119.94 (**q2**), 118.16 (**a3**), 115.14 (-C=O group of TFA acid), 50.78 (**3**), 49.82 (**1**), 44.46 (**2**).

1.5. Synthesis of TRIF



Chemical Formula: $C_{36}H_{42}F_{12}N_8$
Molecular Weight: 814.75

To a solution of $AgNO_3$ (170 mg, 1.0 mmol) in 100 ml MeOH, 206 mg of dialdehyde (1.0 mmol) in 100 mL MeOH were added, under stirring and N_2 flux, at 50°C. A solution of tren (0.10 mL; 0.65 mmol) in 120 mL MeOH was then added over a period of 1 h. After the addition, the mixture was stirred overnight. The polyimine intermediate compound was reduced using an excess of $NaBH_4$ (1.5 g) and the reaction mixture was refluxed overnight. Ag(0) was filtered off and the solution was evaporated to dryness. The residue was dissolved in 50 ml 10% NaOH(aq.) and extracted in dichloromethane (5x50 mL). The collected organic phases were dried over anhydrous Na_2SO_4. The final product is a white powder (550 mg, yield: 40%).

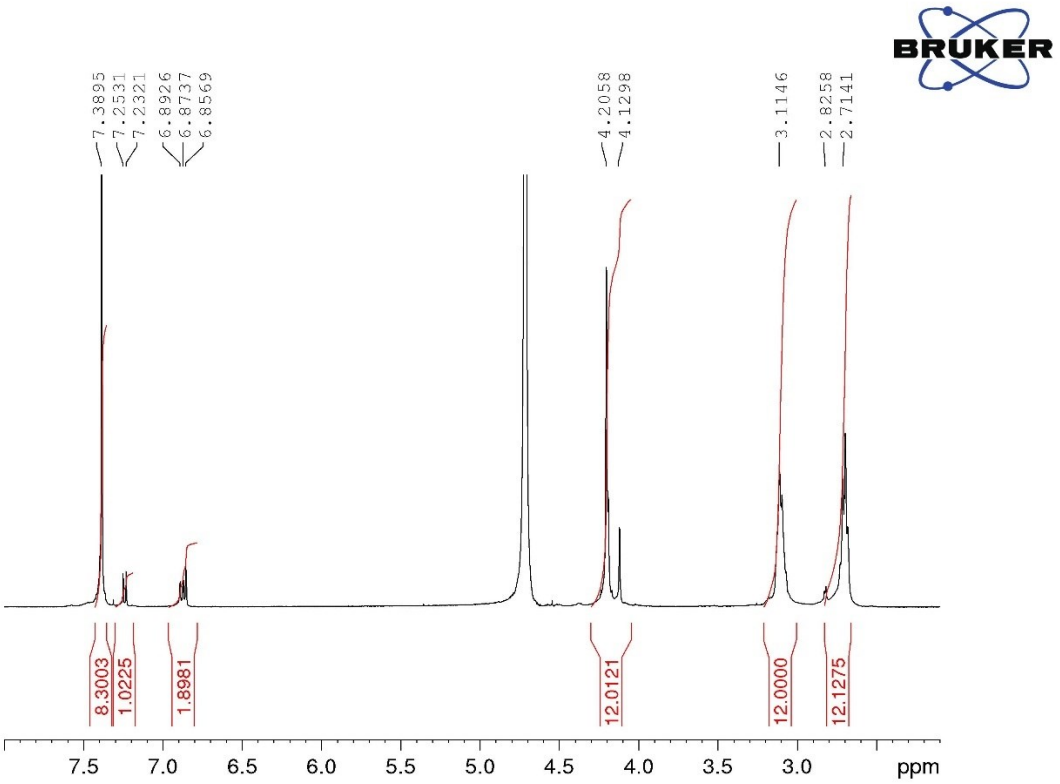


ESI-MS in MeOH, m/z: 408.22 $[M + 2H]^{2+}$. ^{1}H-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm: 4.48 (s, 12H, **3**); 3.19 (t, 12H, **2**); 2.81 (t, 12H, **1**).^{13}C-NMR (400 MHz) in D_2O + CF_3SO_3H, ppm: 146.77-144.29 (d, δ_m = 145.53, $^1J_{C\text{-}F}$ = 249.58 Hz, **q2**), 124.32-114.85 (q, δ_m = 119.59, $^1J_{C\text{-}F}$ = 317.47 Hz, -CF_3 group of triflic acid), 111.46 (**q1**), 50.98 (**1**), 45.68 (**2**), 38.75 (**3**). ^{19}F-NMR (400 MHz) in $CDCl_3$, ppm: -145.91

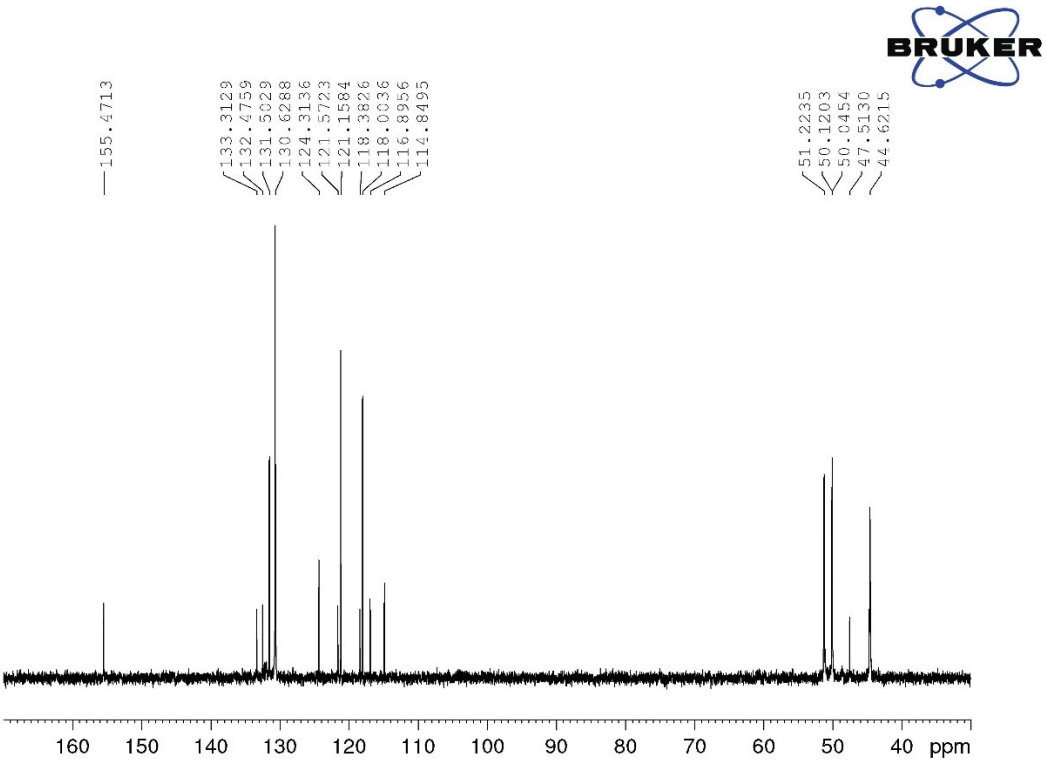
2. Characterization by NMR spectroscopy

- **NMR spectra of MOH cage**

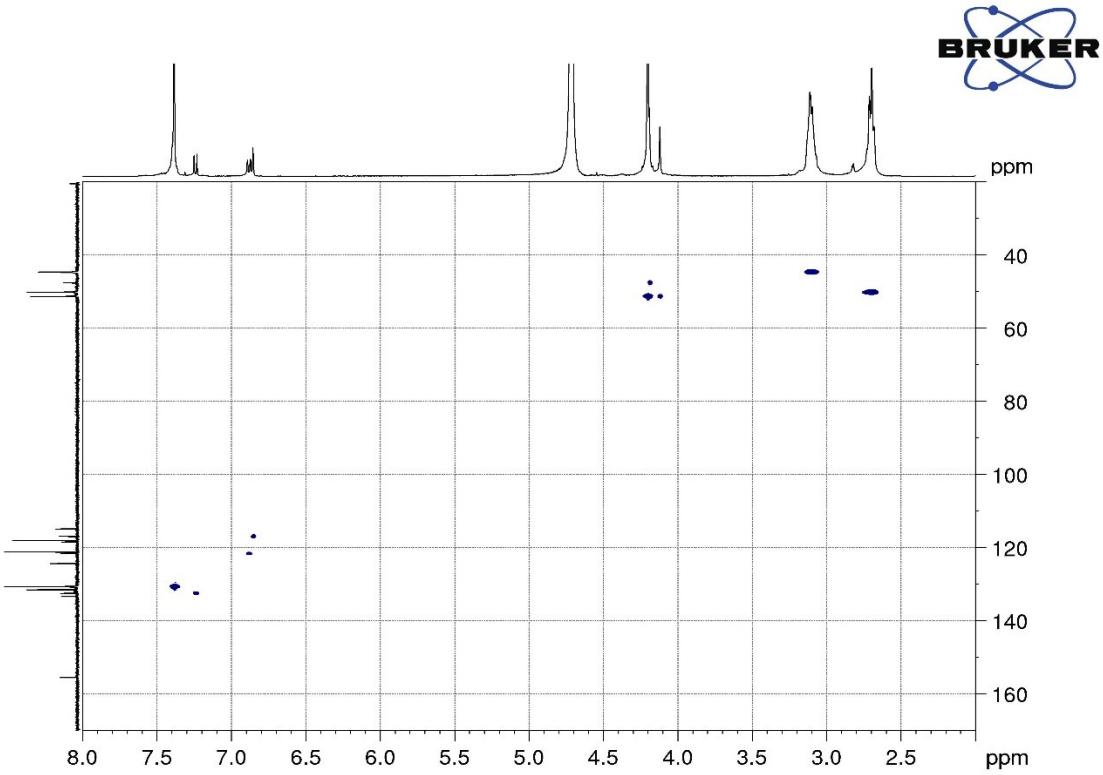
a)



b)



c)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120
140
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6.0
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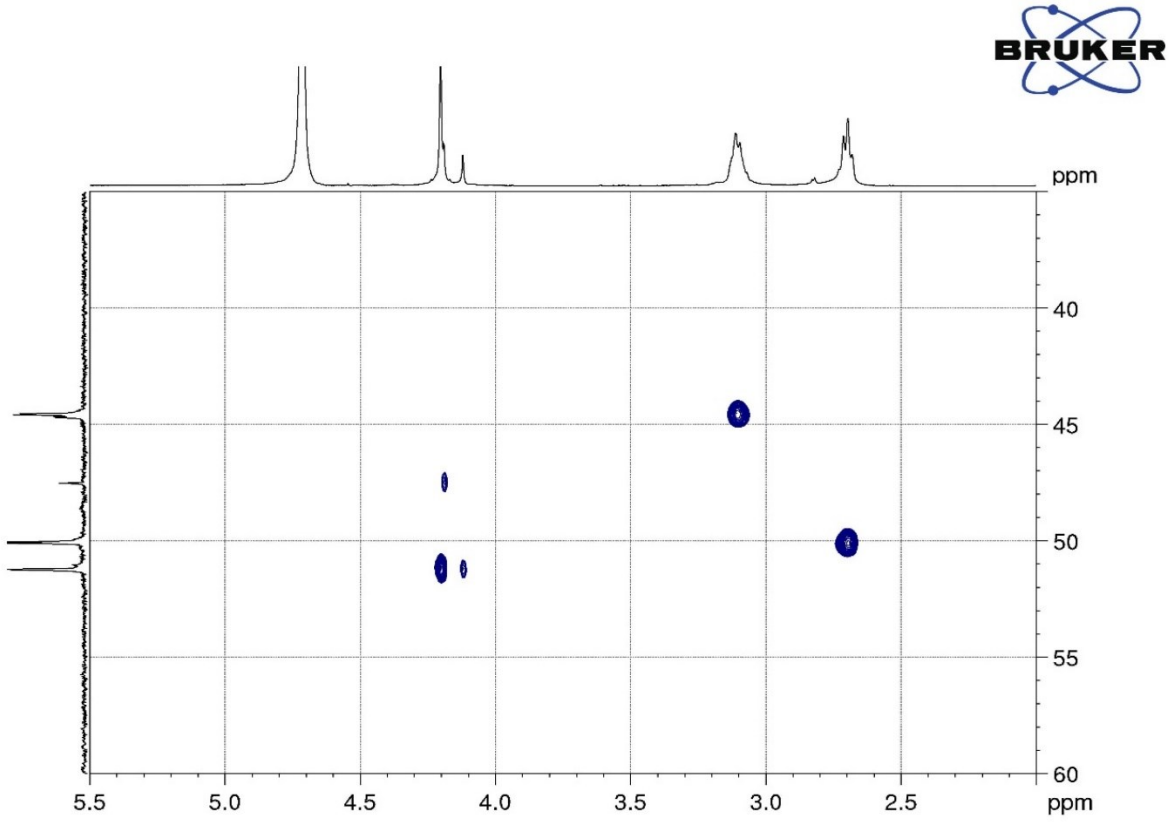
5.0
4.5
4.0
3.5
3.0
2.5
ppm

d)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55
60
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5.0
4.5
4.0
3.5
3.0
2.5
ppm

e)

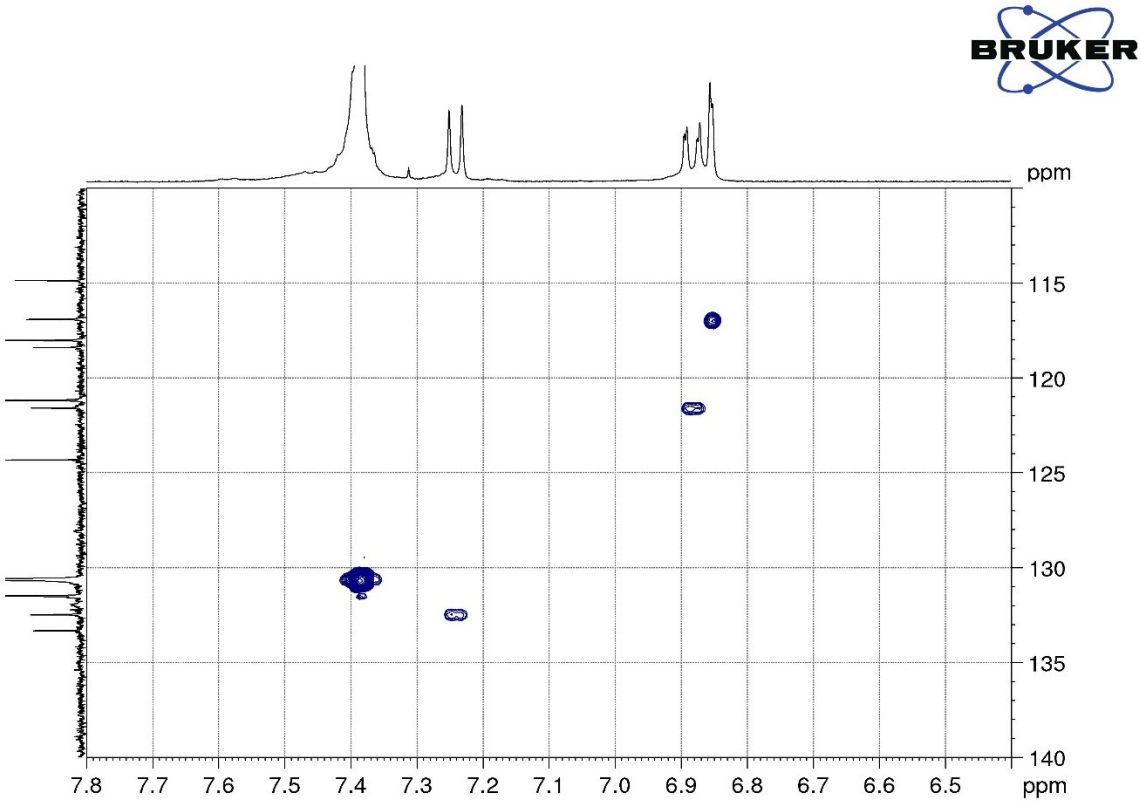
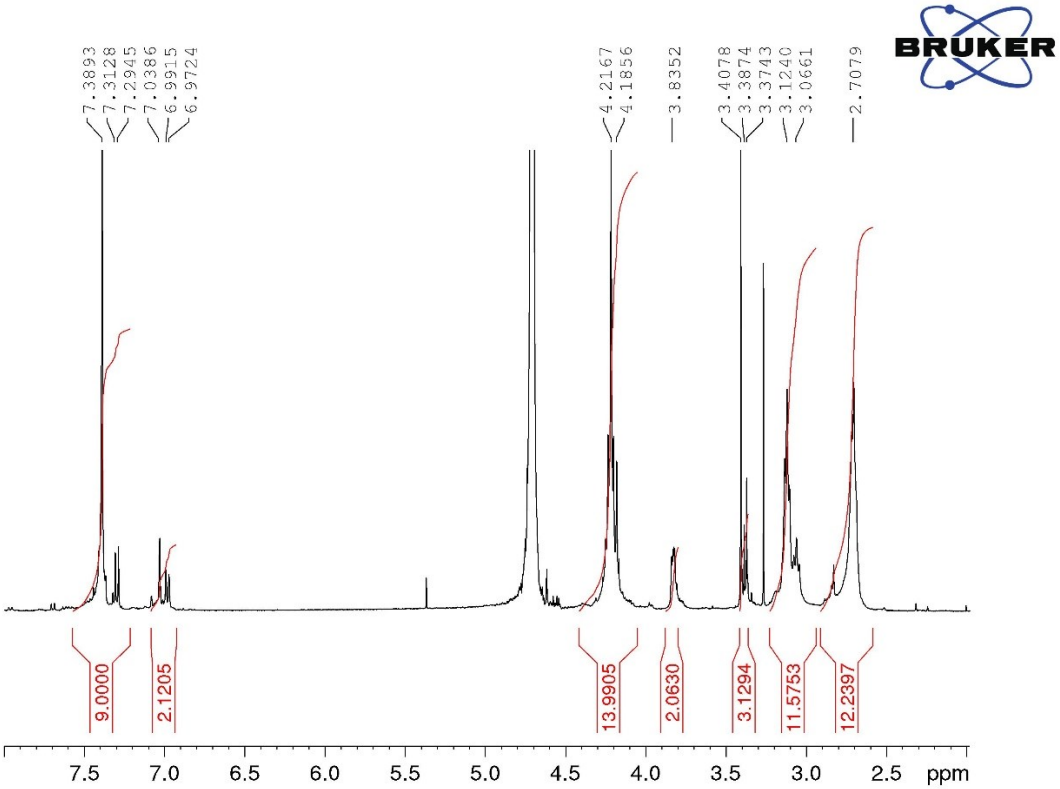


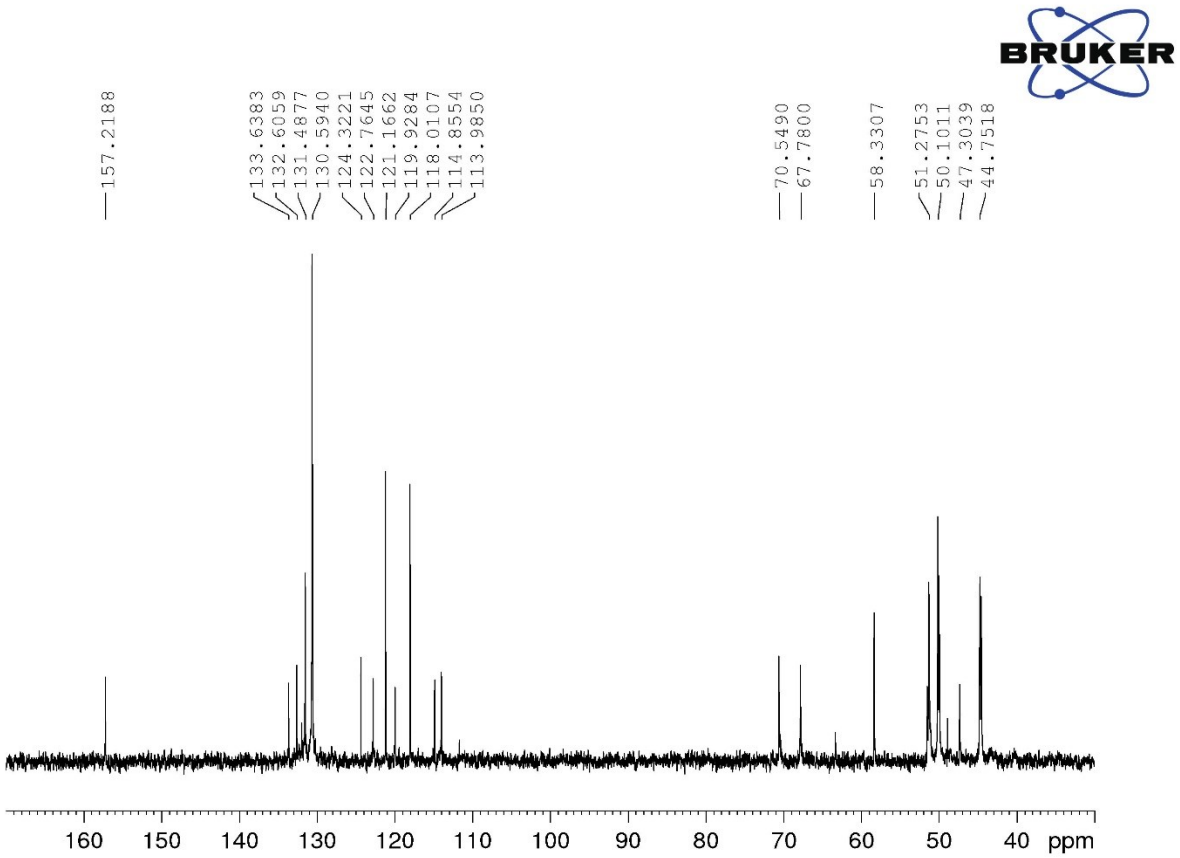
Figure S1: NMR characterization of MOH cage (400 MHz, 298K, D_2O + CF_3SO_3H): a) ^{1}H-NMR; b) ^{13}C-NMR; c) HSQC; d) HSQC zoomed in the aliphatic zone; e) HSQC zoomed in the aromatic zone.

- **NMR spectra of 1-ARM cage**

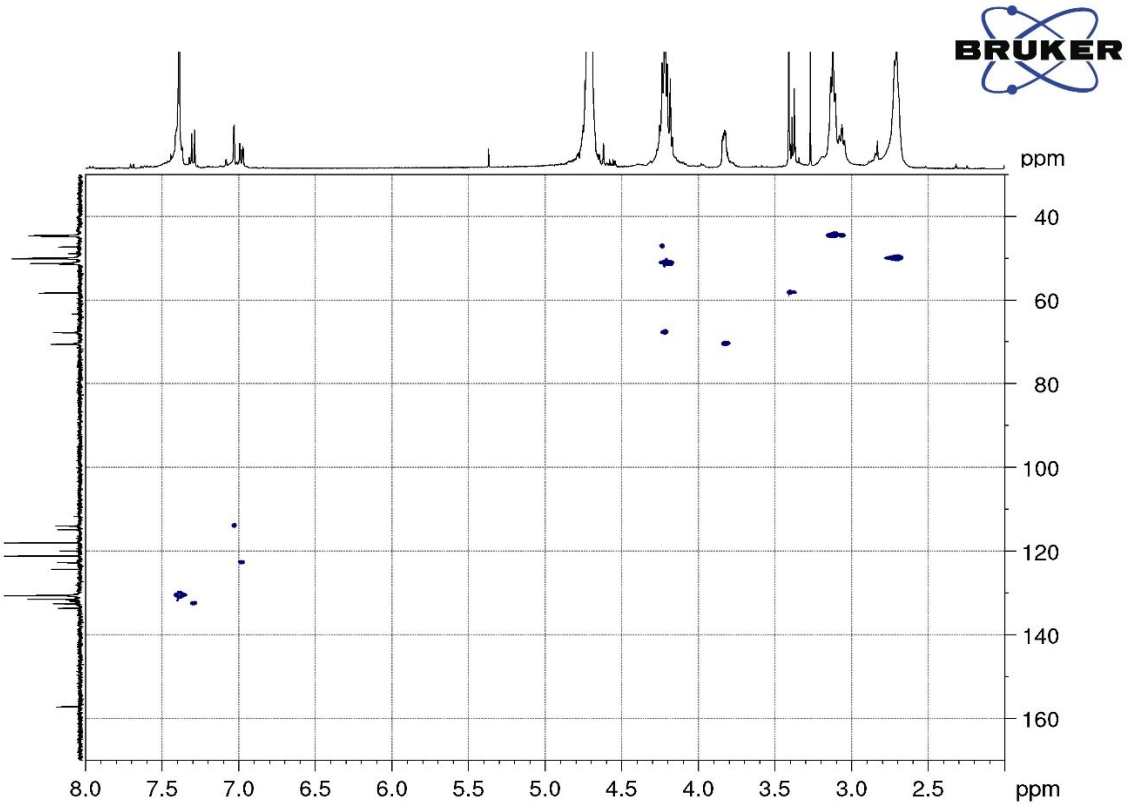
a)



b)



c)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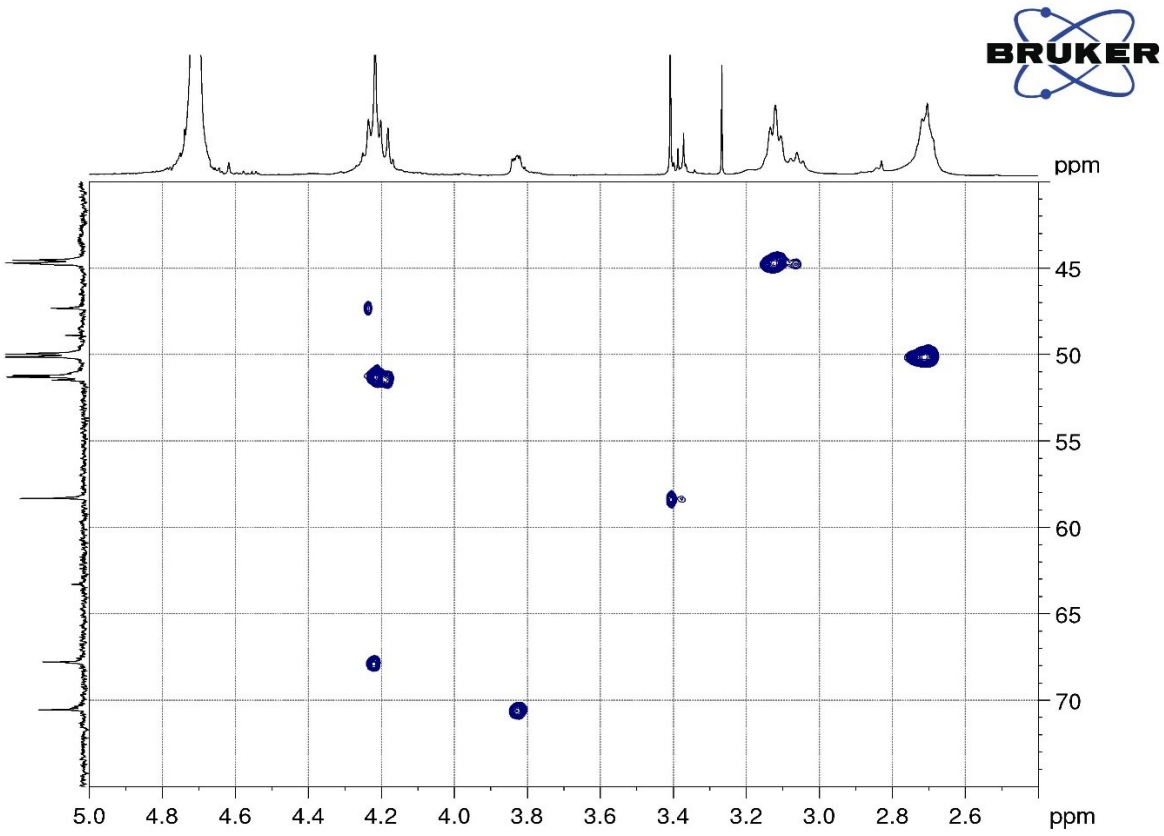
4.0
3.5
3.0
2.5
ppm

d)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3.2
3.0
2.8
2.6
ppm

e)

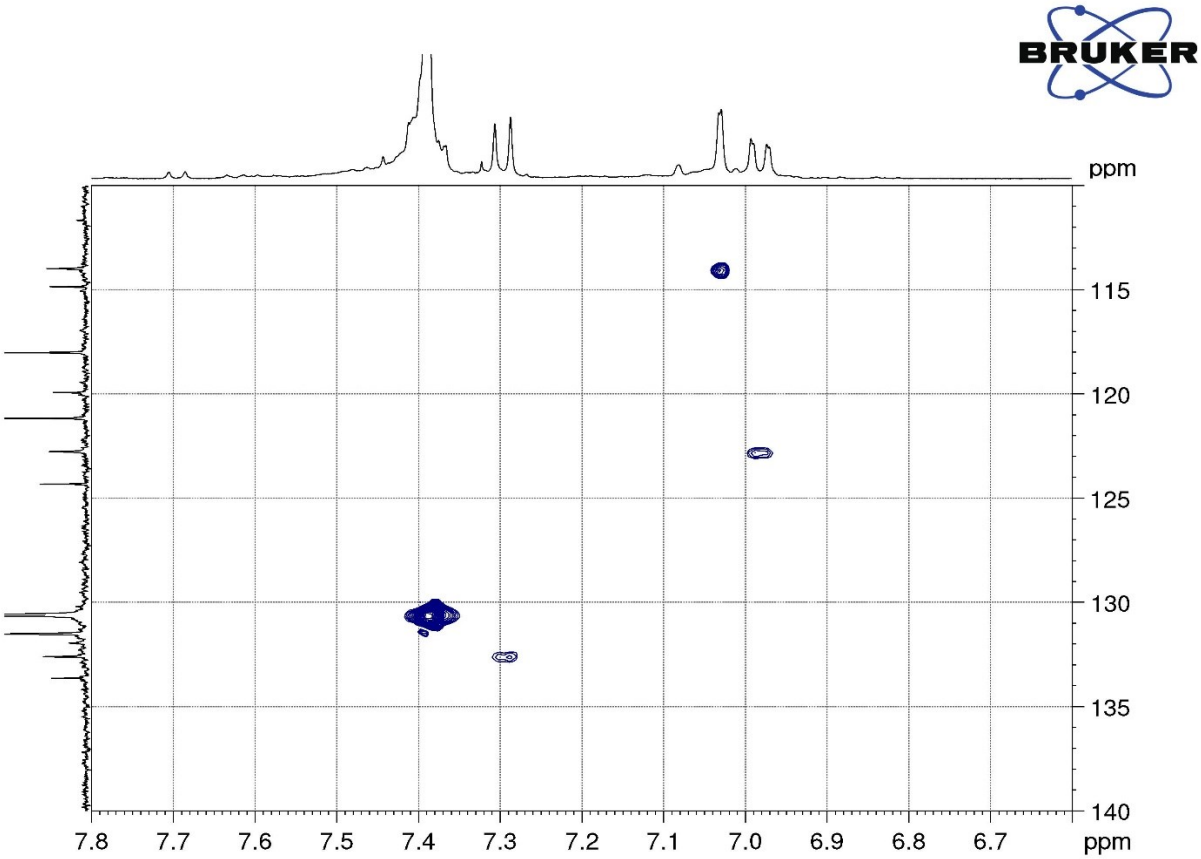
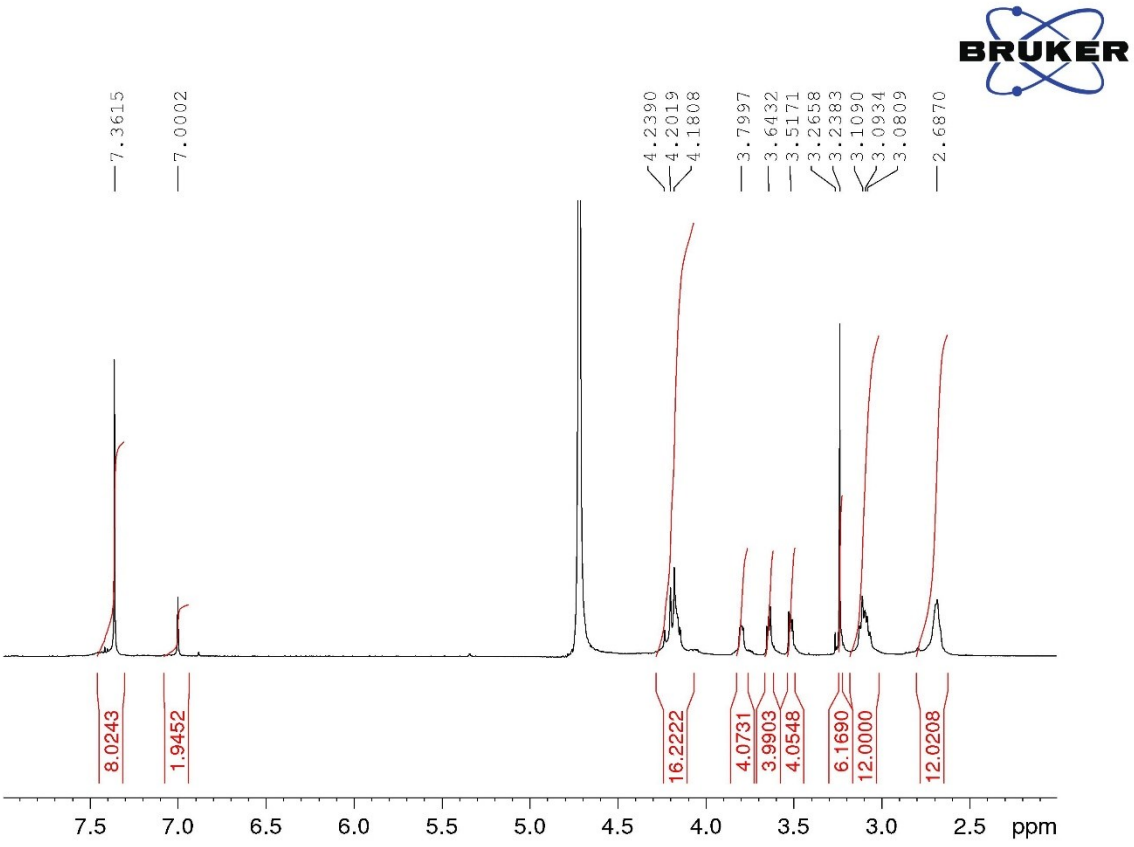


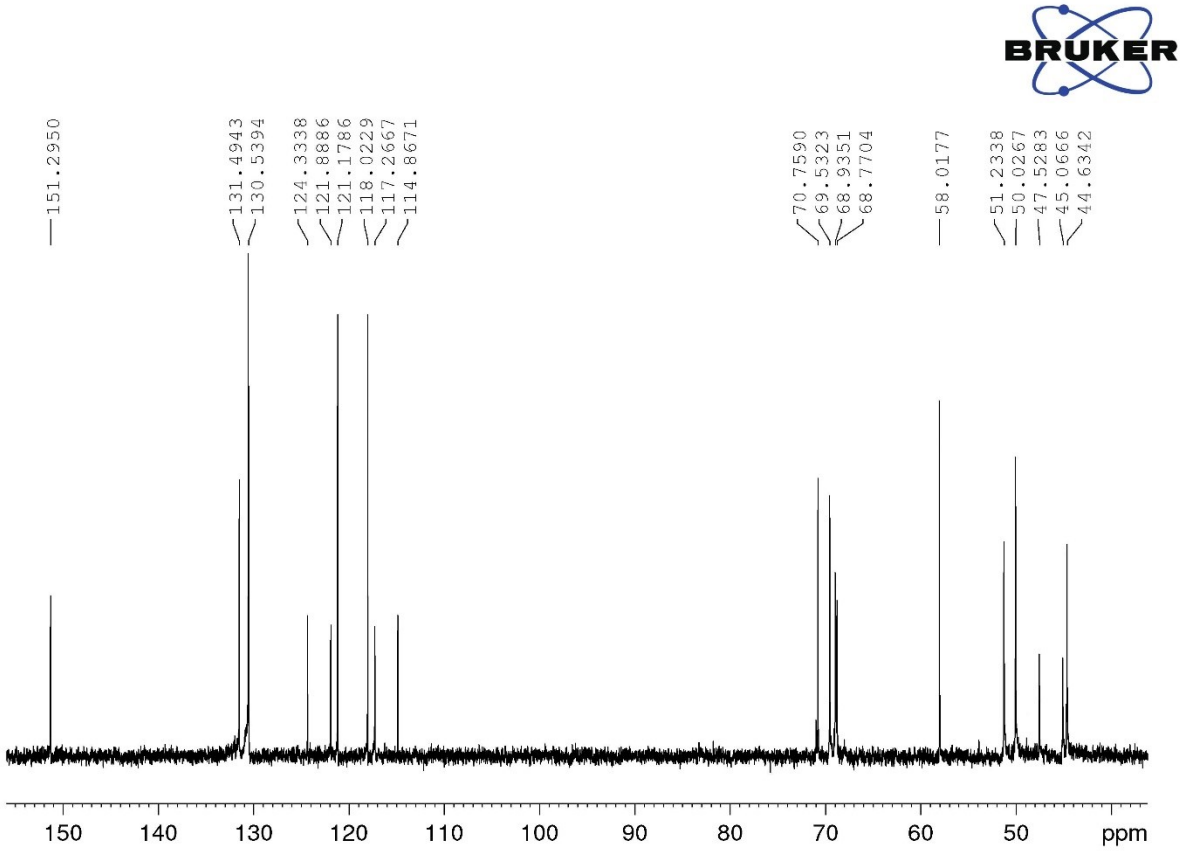
Figure S2: NMR characterization of 1-ARM cage (400 MHz, 298K, D_2O + CF_3SO_3H): a) ^{1}H-NMR; b) ^{13}C-NMR; c) HSQC; d) HSQC zoomed in the aliphatic zone; e) HSQC zoomed in the aromatic zone.

- **NMR spectra of PEG cage**

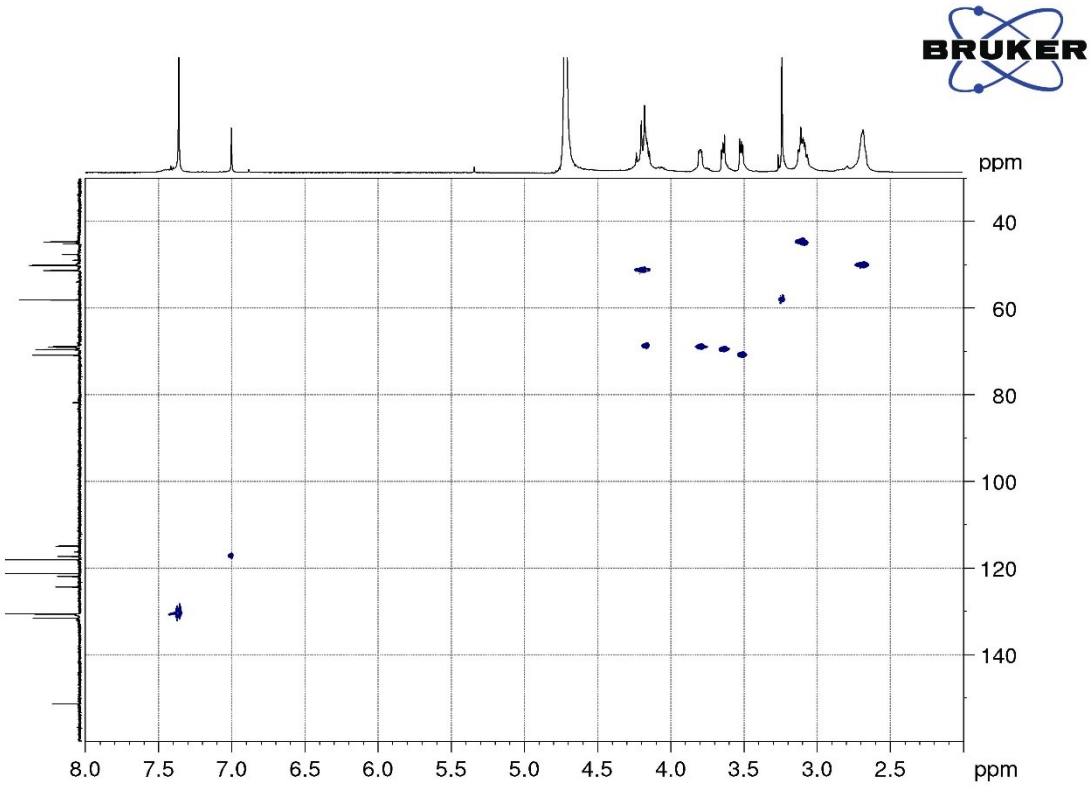
a)



b)



c)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6.0
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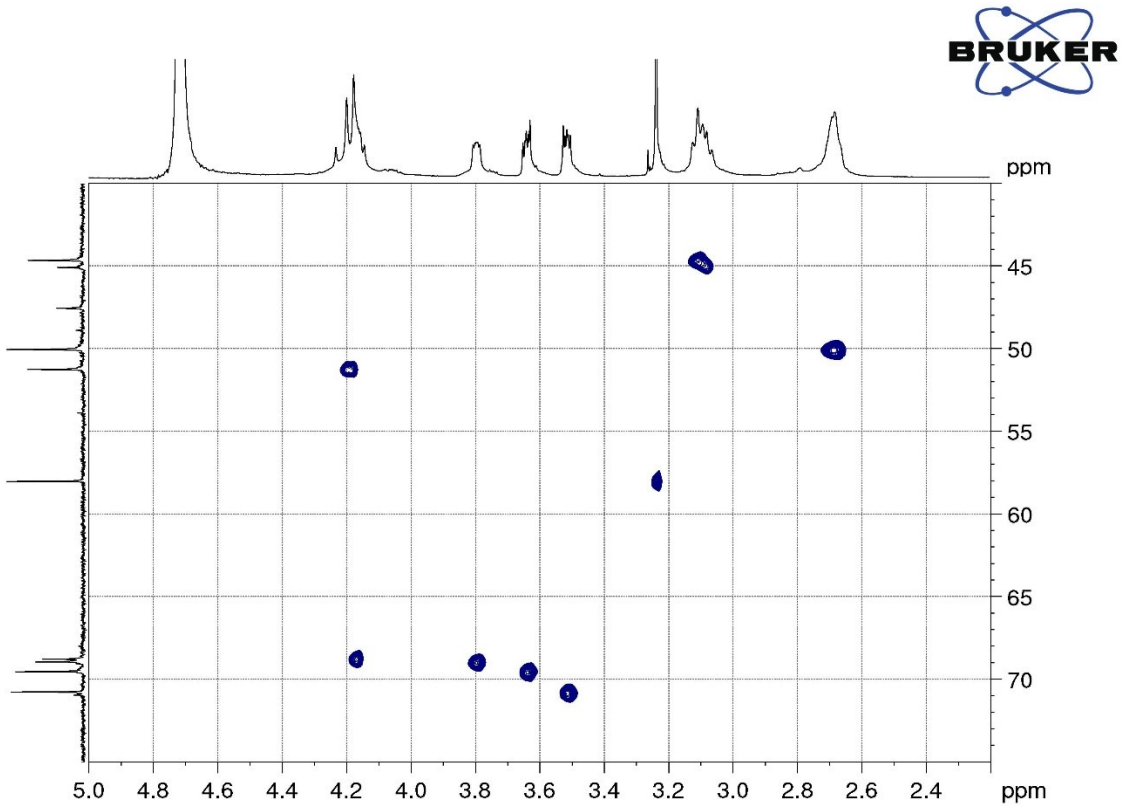
4.0
3.5
3.0
2.5
ppm

d)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2.8
2.6
2.4
ppm

e)

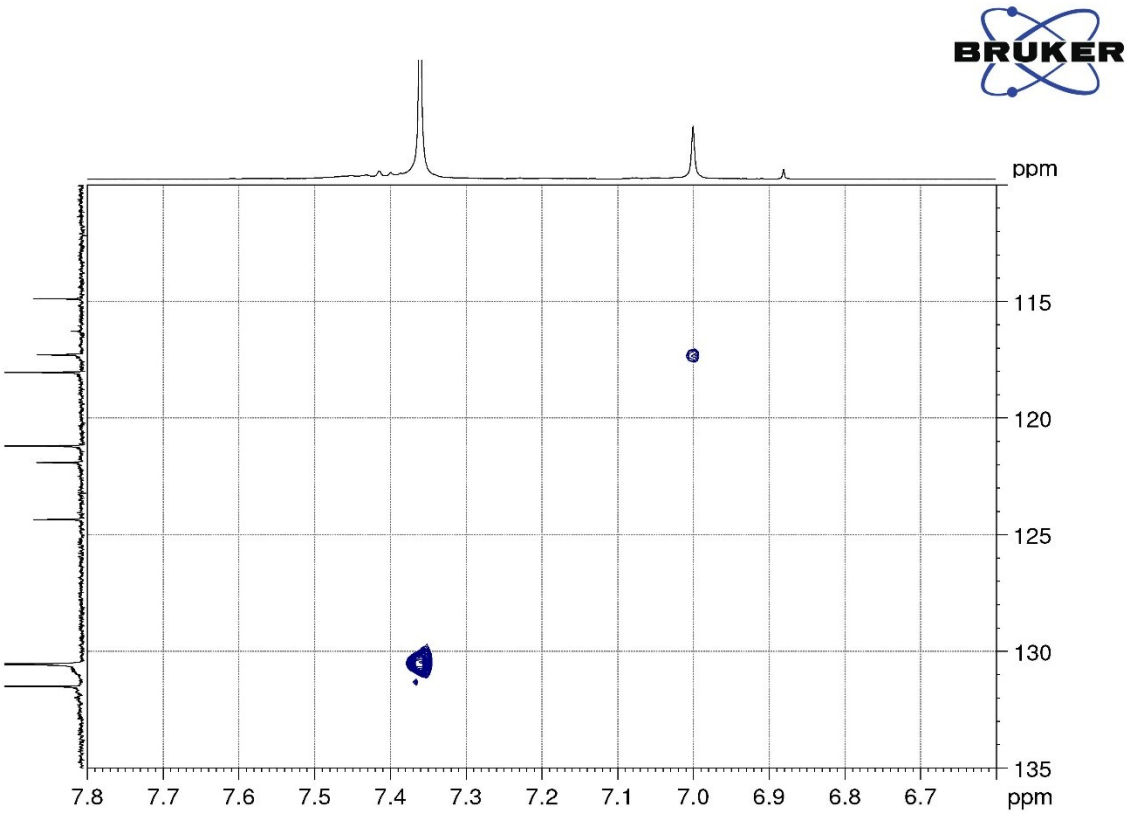
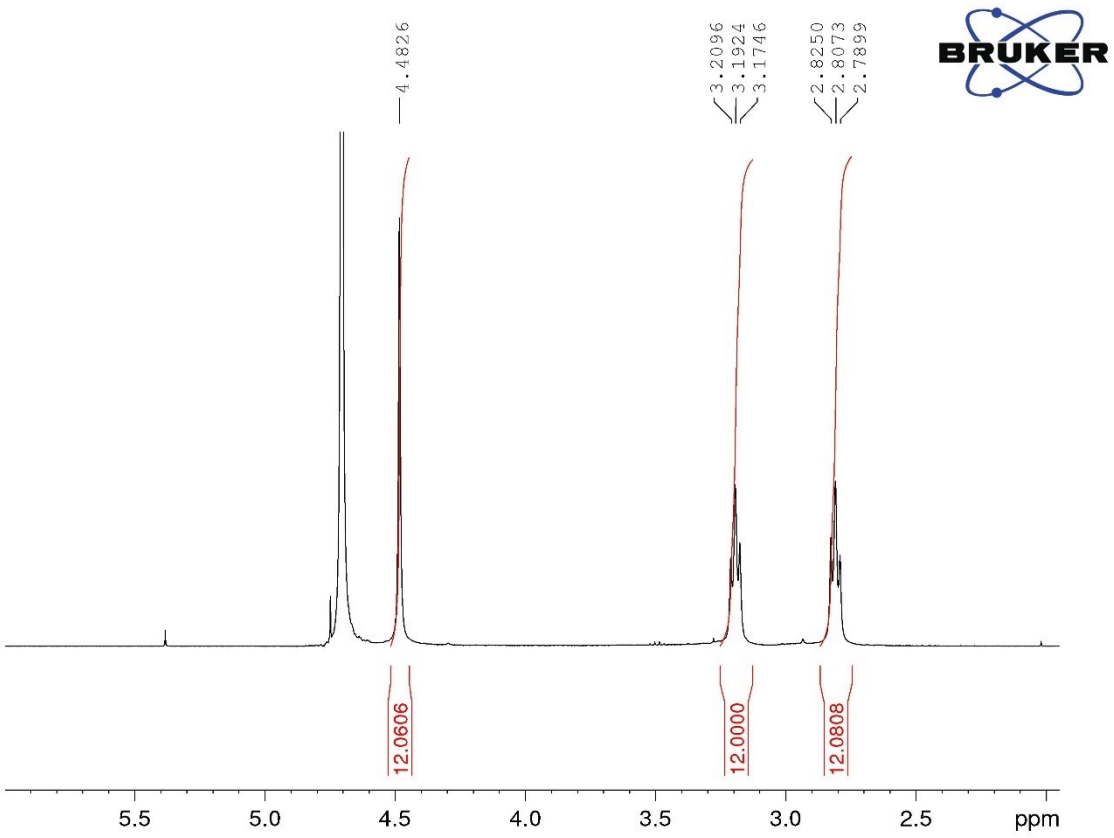


Figure S3: NMR characterization of PEG cage (400 MHz, 298K, D_2O + CF_3SO_3H): a) ^{1}H-NMR; b) ^{13}C-NMR; c) HSQC; d) HSQC zoomed in the aliphatic zone; e) HSQC zoomed in the aromatic zone.

- **NMR spectra of TRIF cage**

a)



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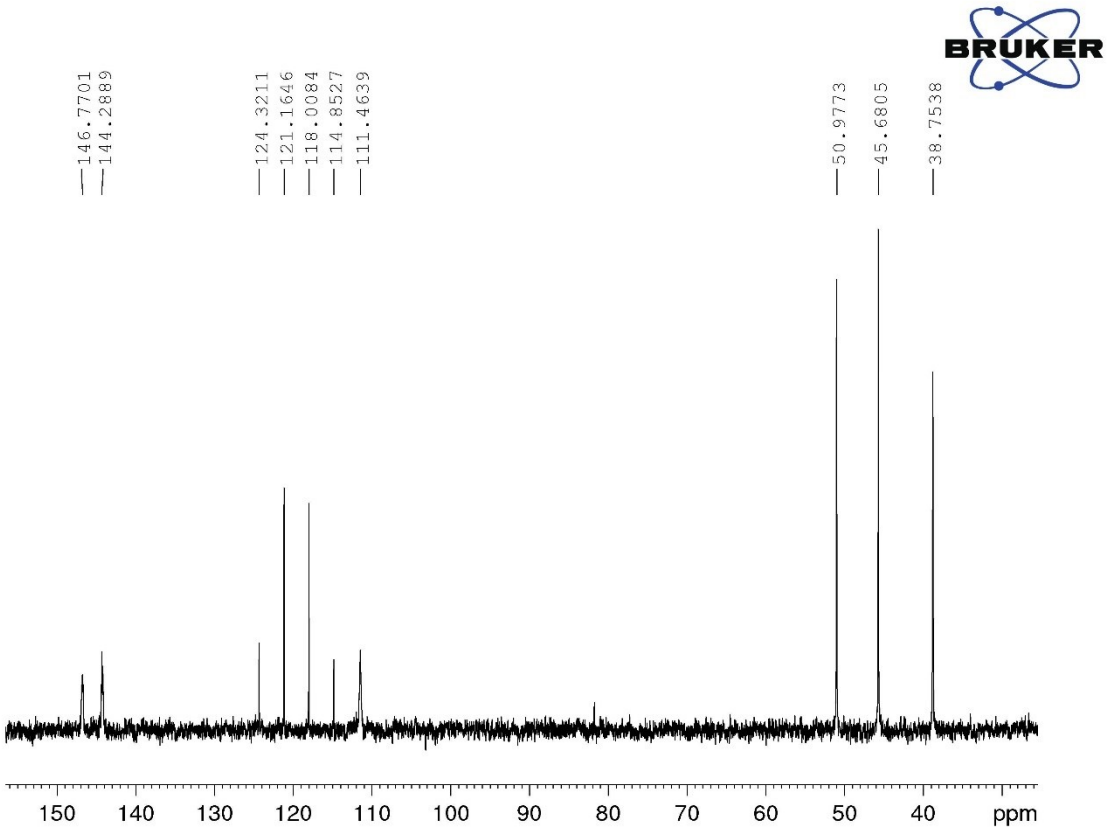
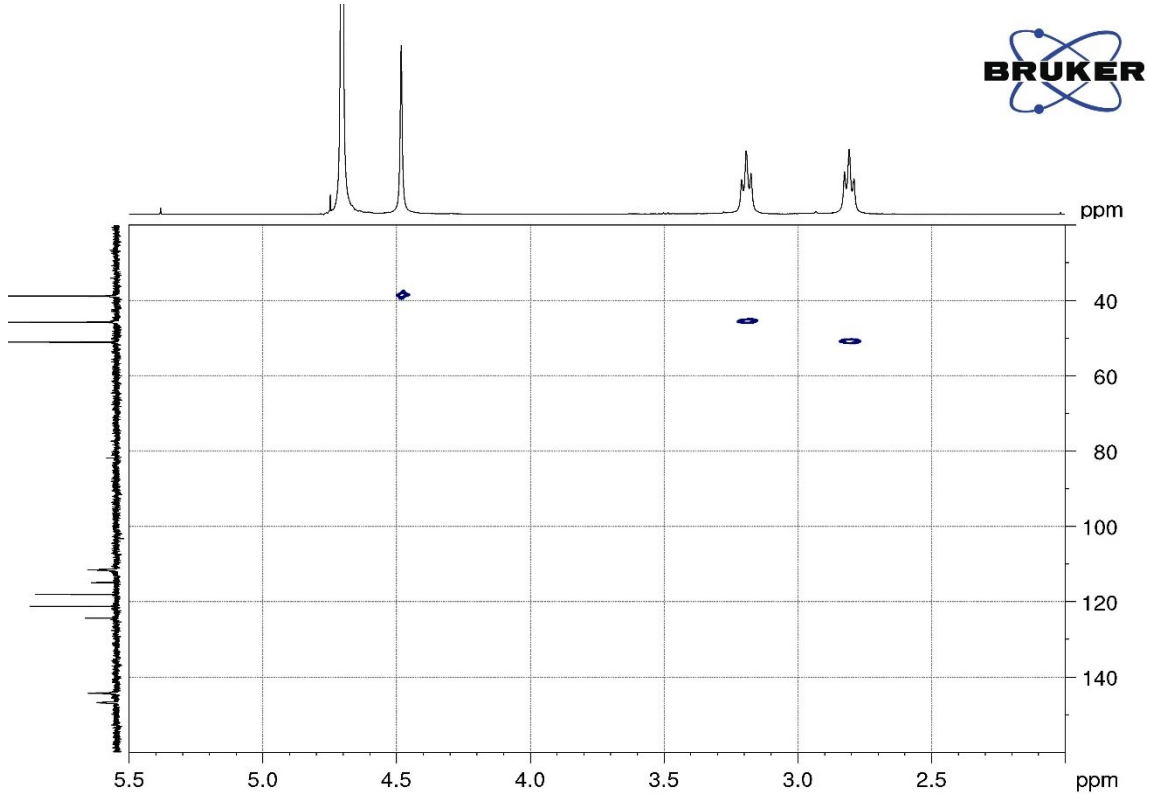


c)



d)

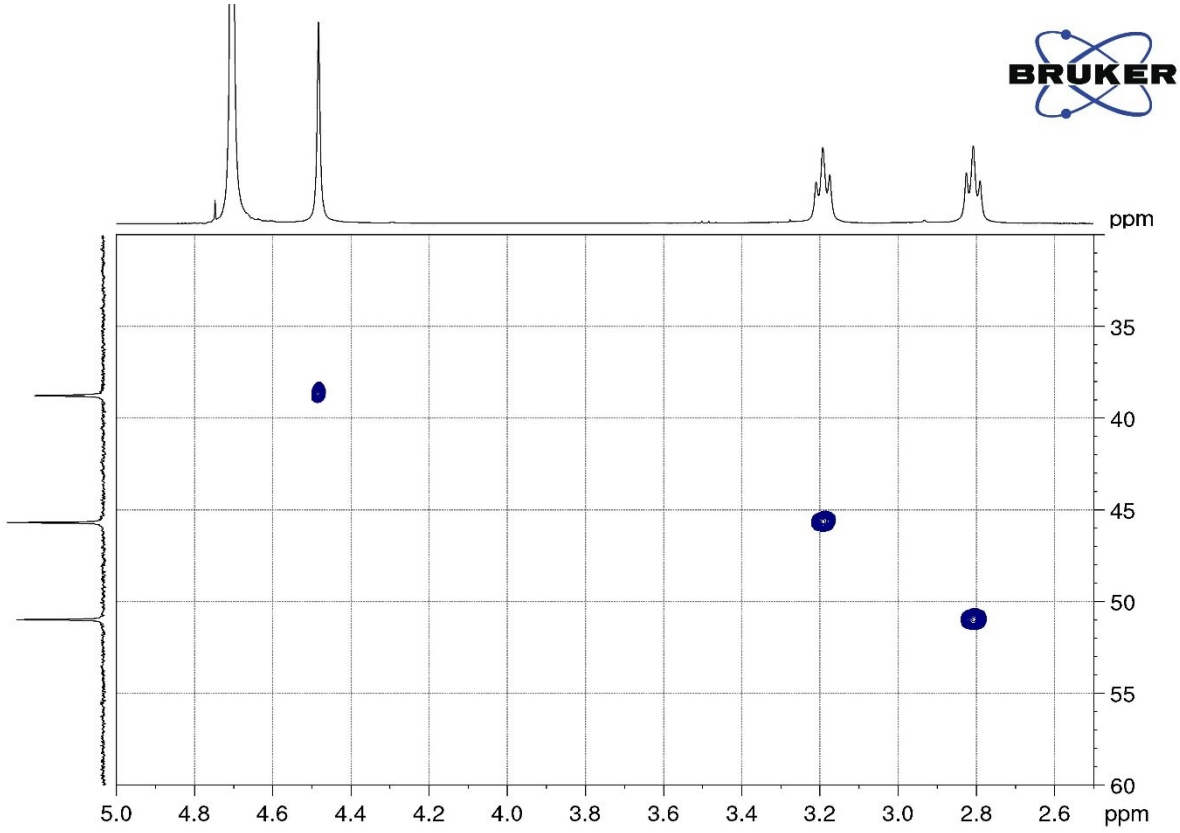
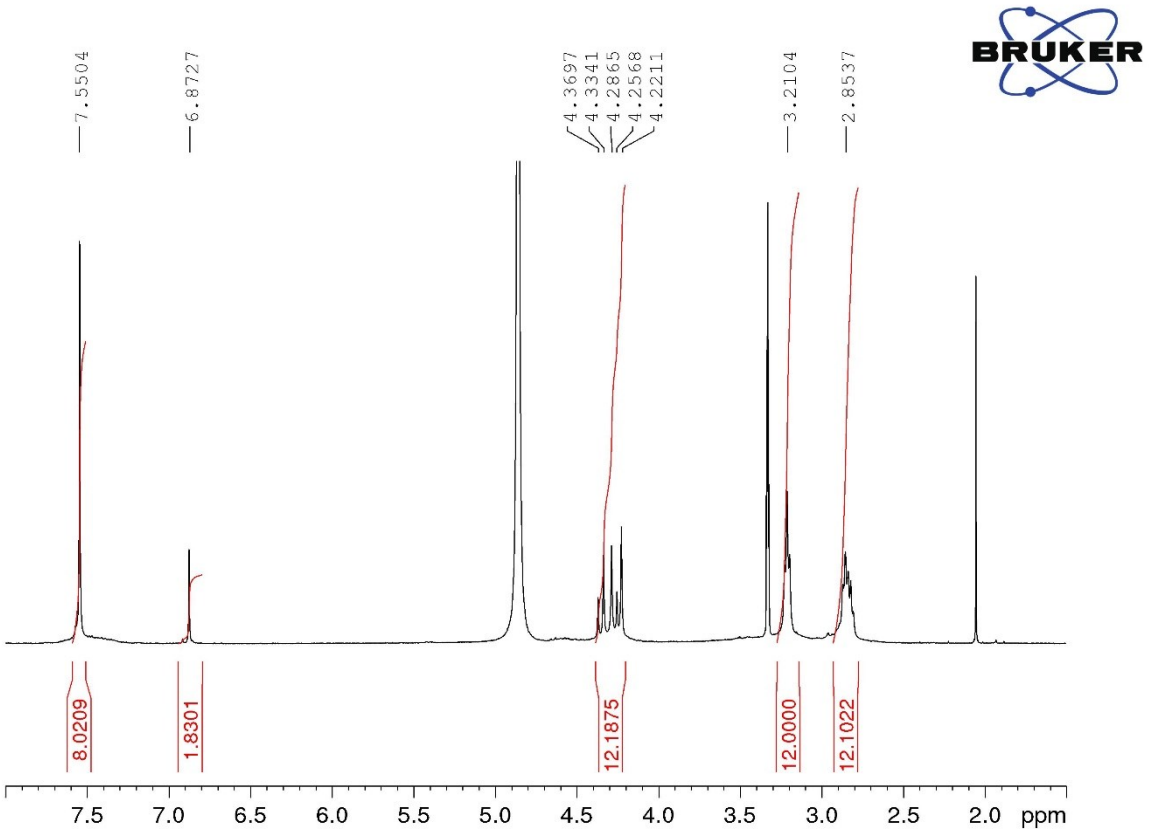


Figure S4: NMR characterization of TRIF cage (400 MHz, 298K, D_2O + CF_3SO_3H): a) ^{1}H-NMR; b) ^{13}C-NMR; c) HSQC; d) HSQC zoomed in the aliphatic zone.

- **NMR spectra of 2-OH cage**

a)



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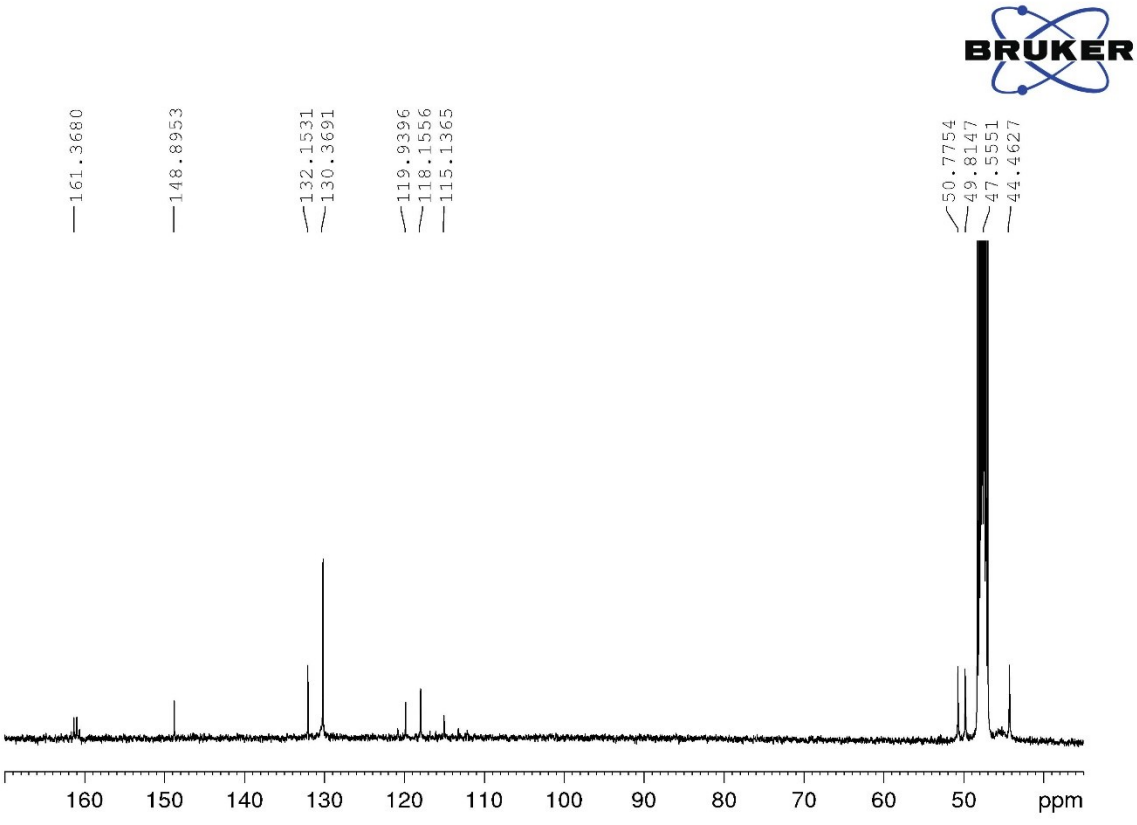
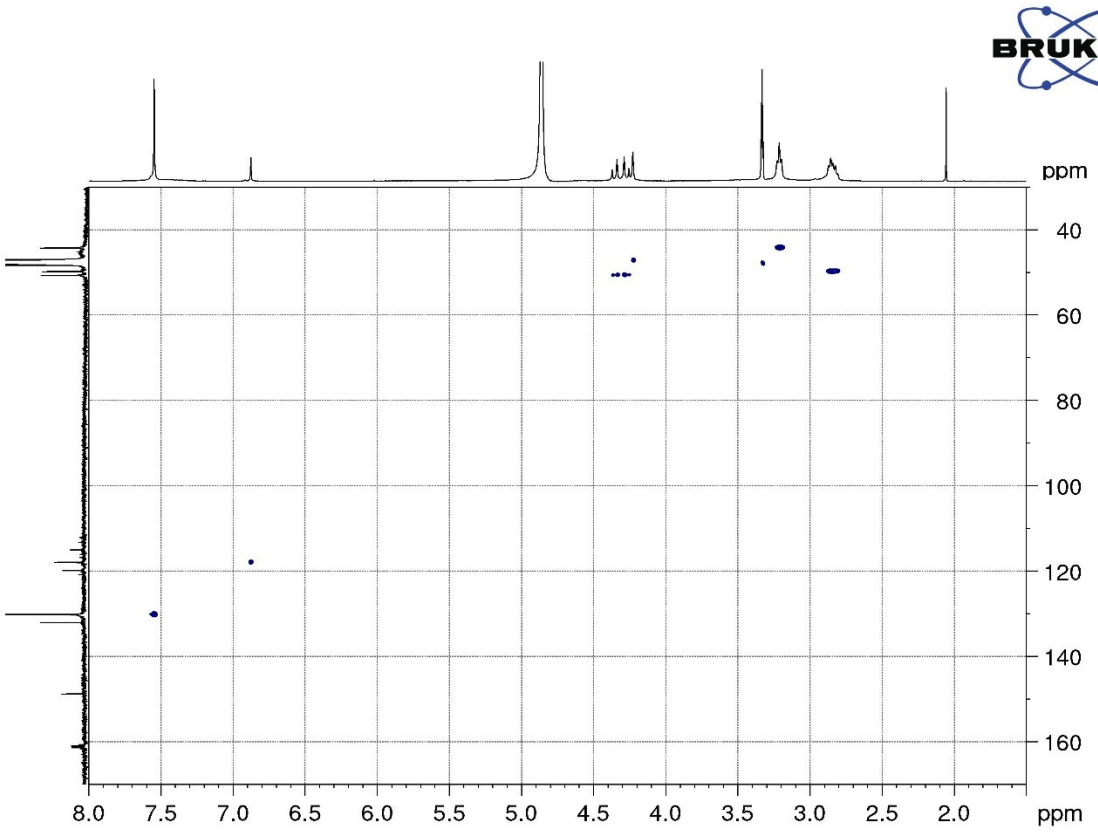


c)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60
80
100
120
140
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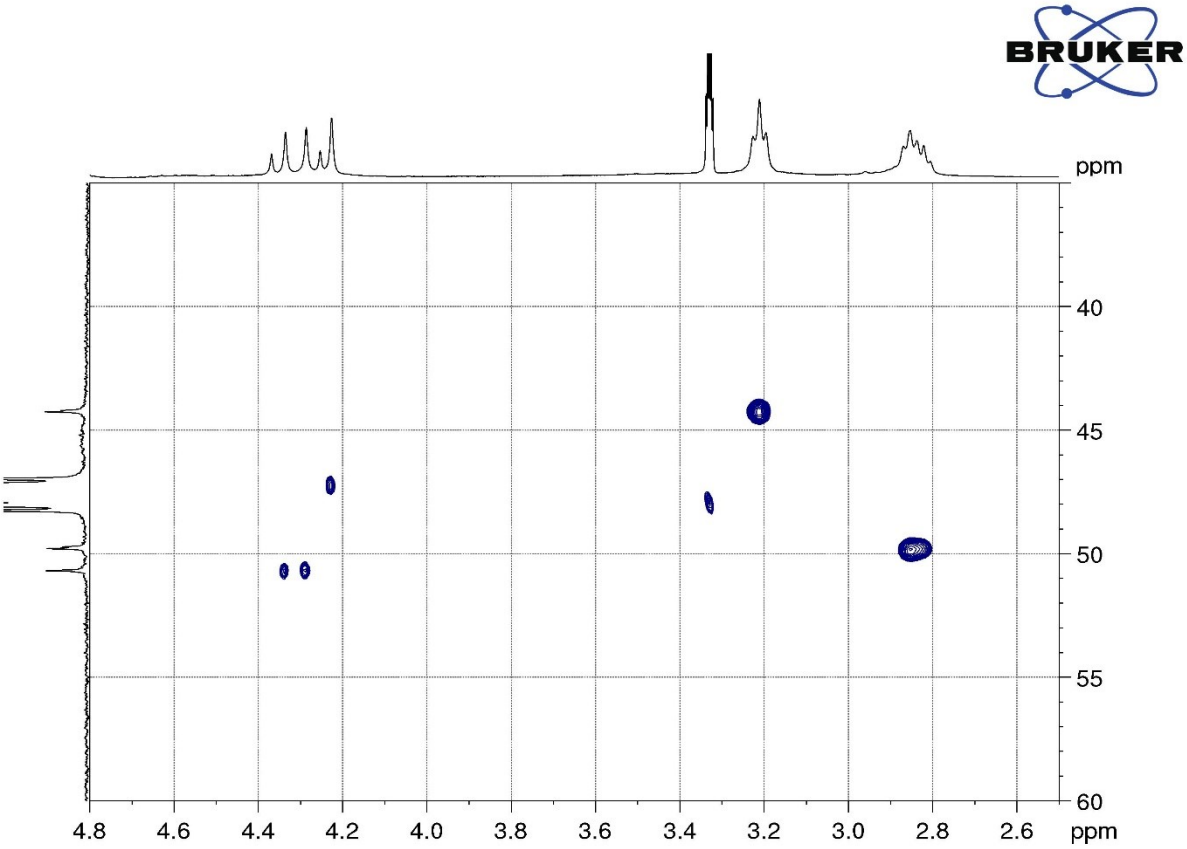
4.5
4.0
3.5
3.0
2.5
2.0
ppm

d)


BRUKER
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45
50
55
60
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4.4
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4.0
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3.2
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2.8
2.6
ppm

e)

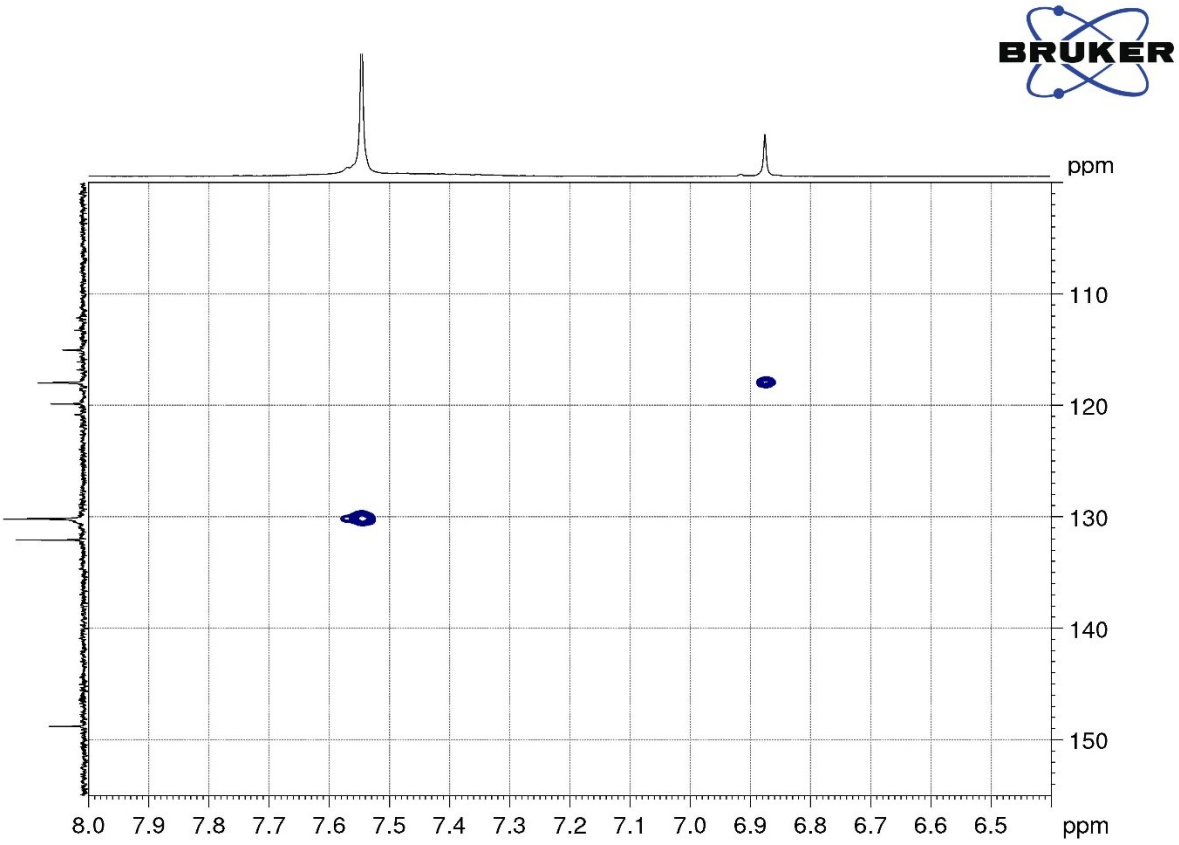


Figure S5: NMR characterization of 2-OH cage (400 MHz, 298K, CD_3OD): a) ^{1}H-NMR; b) ^{13}C-NMR; c) HSQC; d) HSQC zoomed in the aliphatic zone; e) HSQC zoomed in the aromatic zone.

3. Structural characterizations

- **Characterizations of Lcage precipitate obtained in solvent extraction**

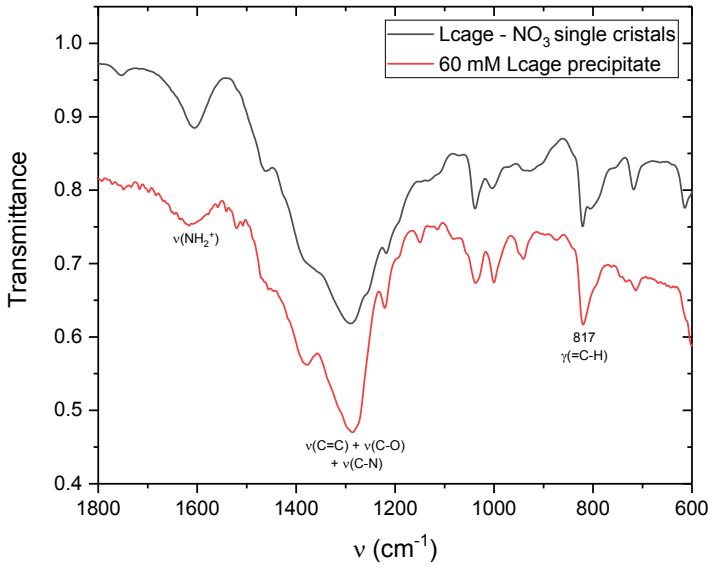


Figure S6: Infrared spectrum of the precipitate obtained with the Lcage derivative during liquid-liquid extraction tests (red spectrum, $C^{aq,ini}_{HNO_3}$= 0.5 M, $C^{aq,ini}_{Tc(VII)}$ = 0.3 g·L^{-1}, $C^{aq,ini}_{Lcage}$ = 100 mM, $A_{^{99m}Tc}$ = 10 kBq·mL^{-1}, $C^{aq,ini}_{U(VI)}$ = 30 g·L^{-1}, C_{MOEHA} = 1.4 M in isane, T = 25°C, V_{aq}/V_{org} = 1, agitation time = 15 min)[5] compared to a reference of a Lcage-nitrate single cristal (black spectrum).

- **Characterizations with rhenium**

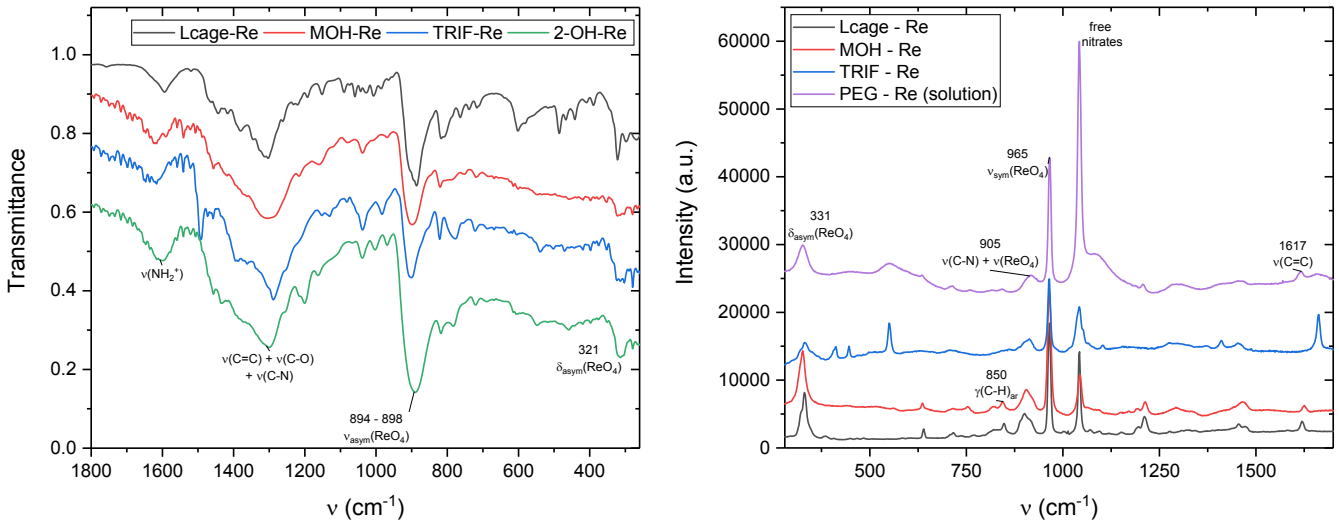


Figure S7: Infrared (left) and Raman (right) spectra of the complexes obtained for all the derivatives with the perrhenate anion (solid state except for PEG – Re in the Raman spectrum).

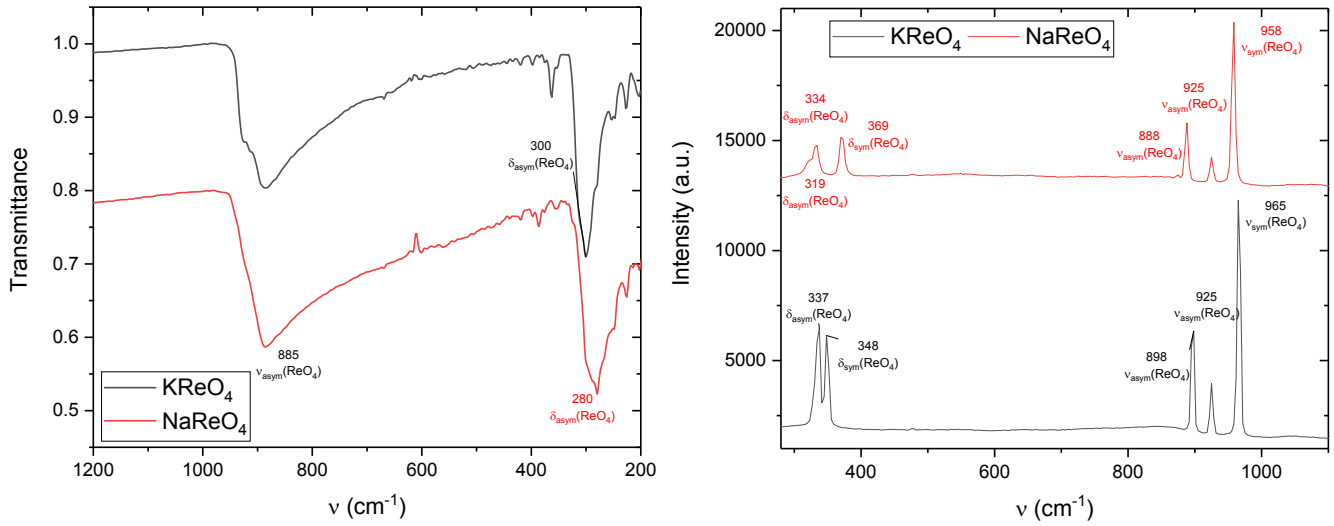


Figure S8: Infrared (left) and Raman (right) spectra of the perrhenate salts.

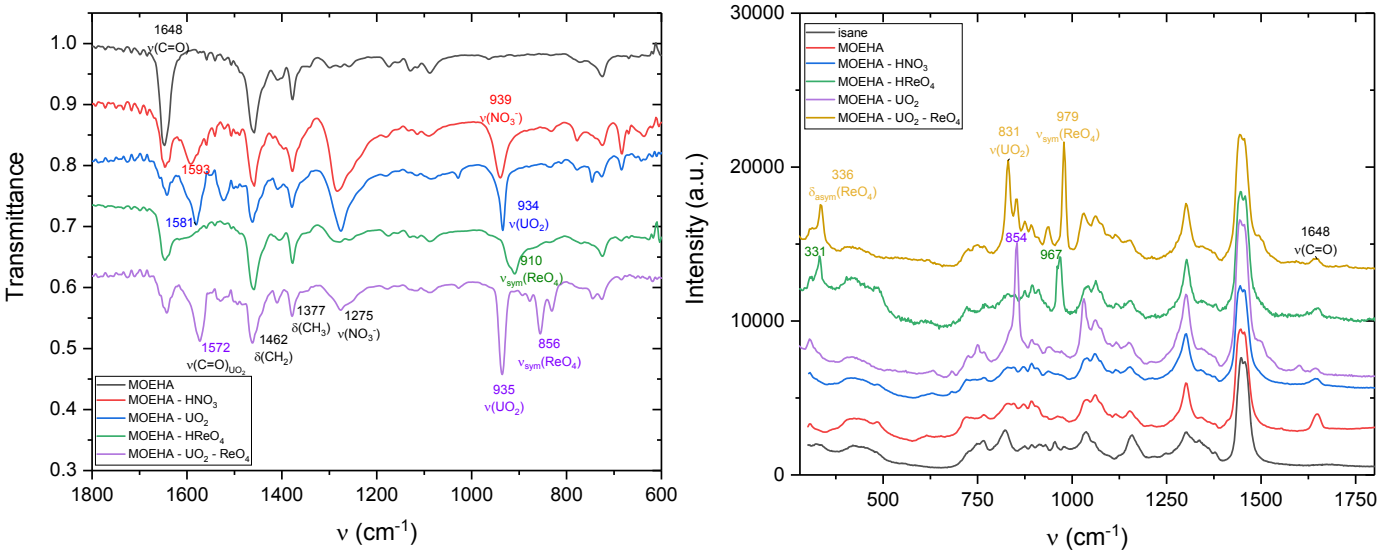


Figure S9: Infrared (left) and Raman (right) spectra of the mixed $UO_2(NO_3)(R\bar{e}O_4)(MOEHA)_2$ and $(HReO_4)\bar{(}MOEHA)_2$ complexes (solvent extraction conditions: $C^{aq,ini}_{Re(VII)}$ = 100 g·L^{-1}, $C^{aq,ini}_{U(VI)}$ = 60 g·L^{-1}, $C^{aq,ini}_{HNO_3}$ = 0.7 M, C_{MOEHA} = 1.4 M in isane, V_{aq}/V_{org} = 2, T = 25°C, and $C^{aq,ini}_{Re(VII)}$ = 200 g·L^{-1}, $C^{aq,ini}_{HNO_3}$ = 0.7 M, C_{MOEHA} = 1.4 M in isane, V_{aq}/V_{org} = 2, T = 25°C respectively).

- **Characterizations with technetium**

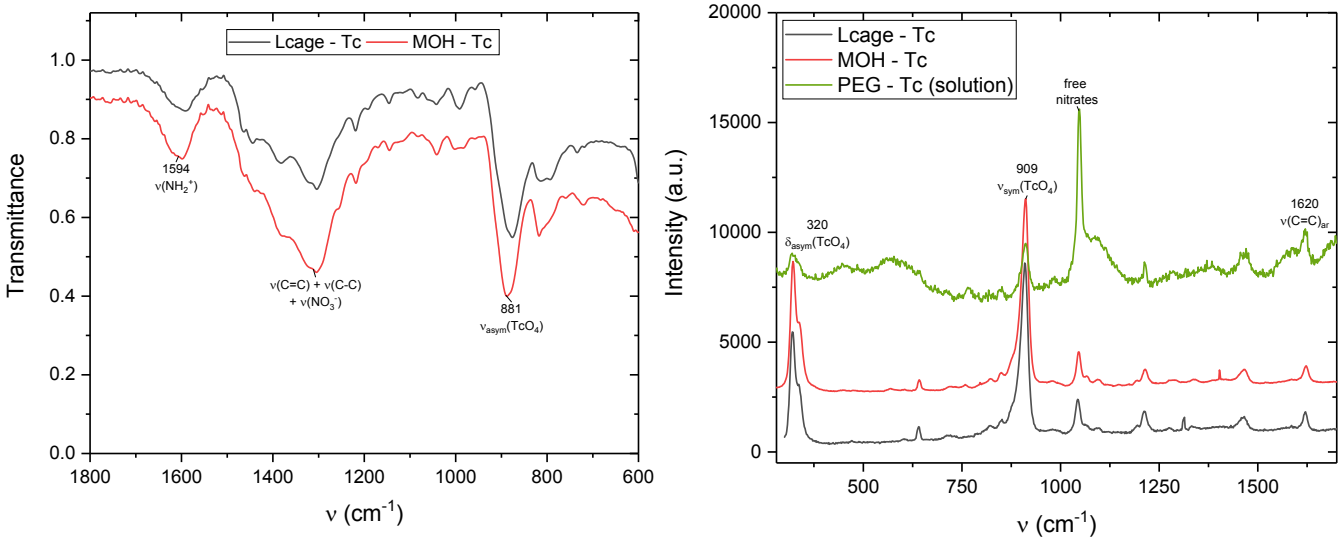


Figure S10: Infrared (left) and Raman (right) spectra of the complexes obtained for all the derivatives with the pertechnetate anion (solid state except for PEG – Re in the Raman spectrum).

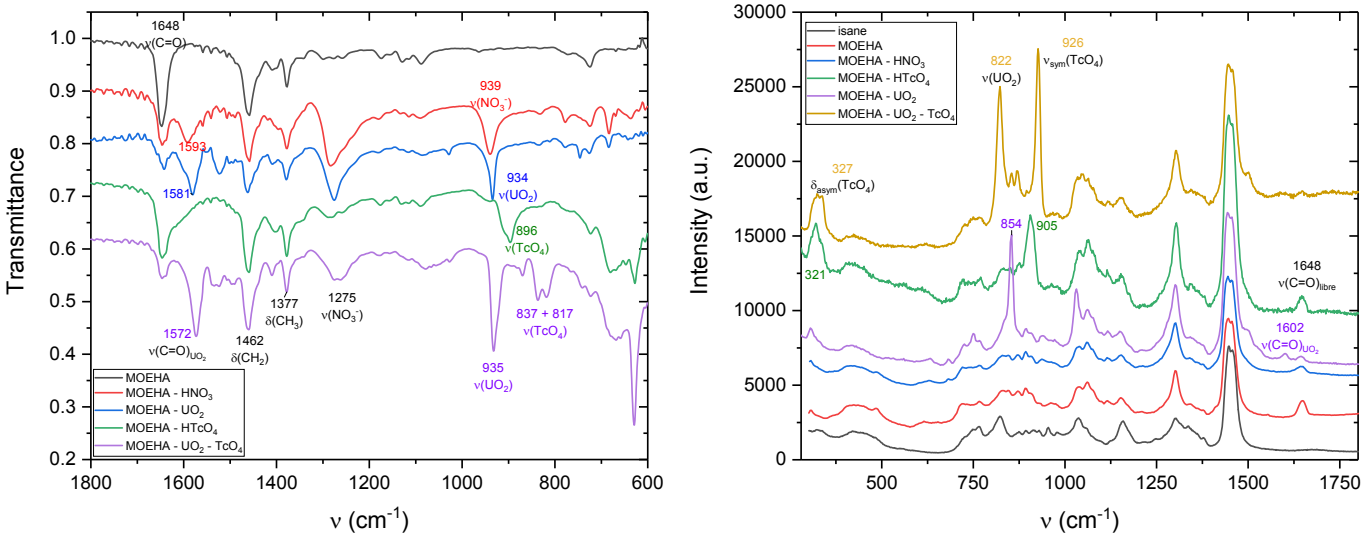
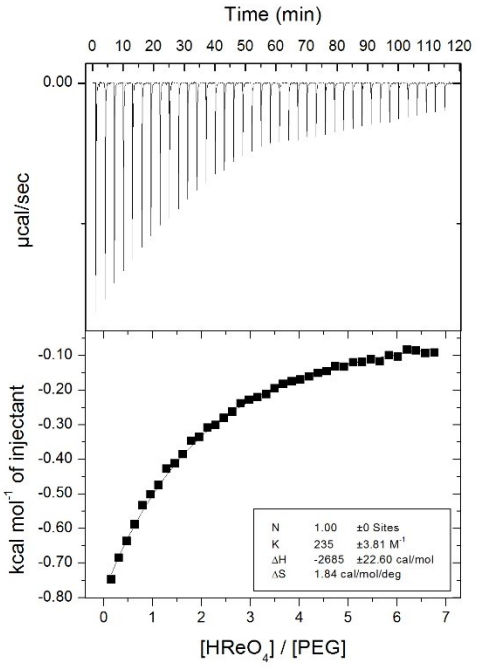
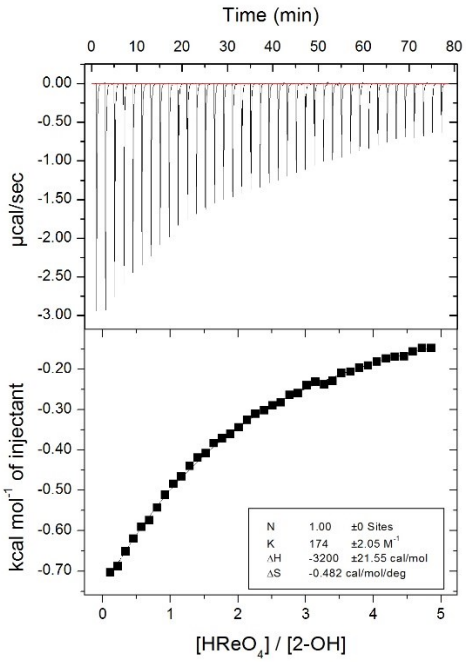


Figure S11: Infrared (left) and Raman (right) spectra of the mixed $UO_2(NO_3)(T\bar{c}O_4)(MOEHA)_2$ and $(HTcO_4)(\bar{M}OEHA)_2$ complexes (solvent extraction conditions: $C^{aq,ini}_{Tc(VII)}$ = 50 g·L^{-1}, $C^{aq,ini}_{U(VI)}$ = 60 g·L^{-1}, $C^{aq,ini}_{HNO_3}$ = 1.5 M, C_{MOEHA} = 1.4 M in isane, V_{aq}/V_{org} = 2, T = 25°C, and $C^{aq,ini}_{Tc(VII)}$ = 50 g·L^{-1}, $C^{aq,ini}_{HNO_3}$ = 1.5 M, C_{MOEHA} = 1.4 M in isane, V_{aq}/V_{org} = 2, T = 25°C respectively).

4. Thermodynamic studies

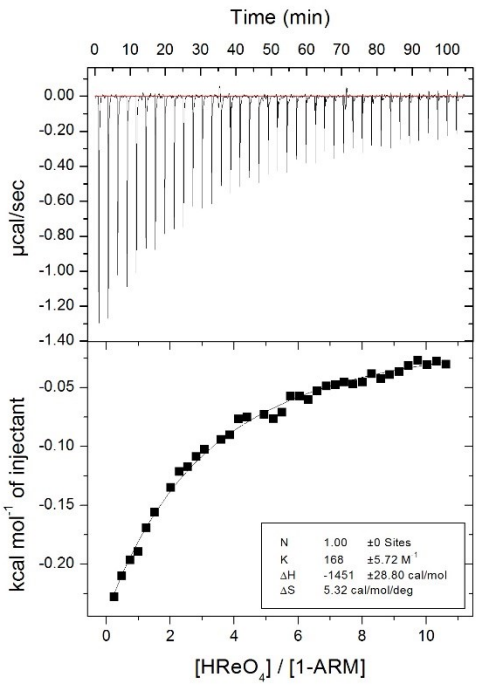


a)

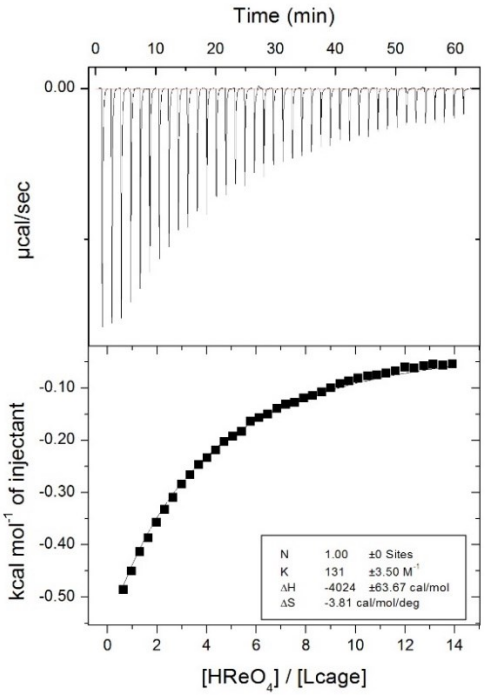


b)

Figure S12: Thermograms and fit of the titration curve obtained for 39 x 1µL injections of: (a) $HReO_4$ (54.8 mM) in 0.5 M HNO_3 in a PEG (1.7 mM) solution in 0.5 M HNO_3, and (b) $HReO_4$ (40.0 mM) in 0.5 M HNO_3 in 2-OH (1.7 mM) solution in 0.5 M HNO_3.

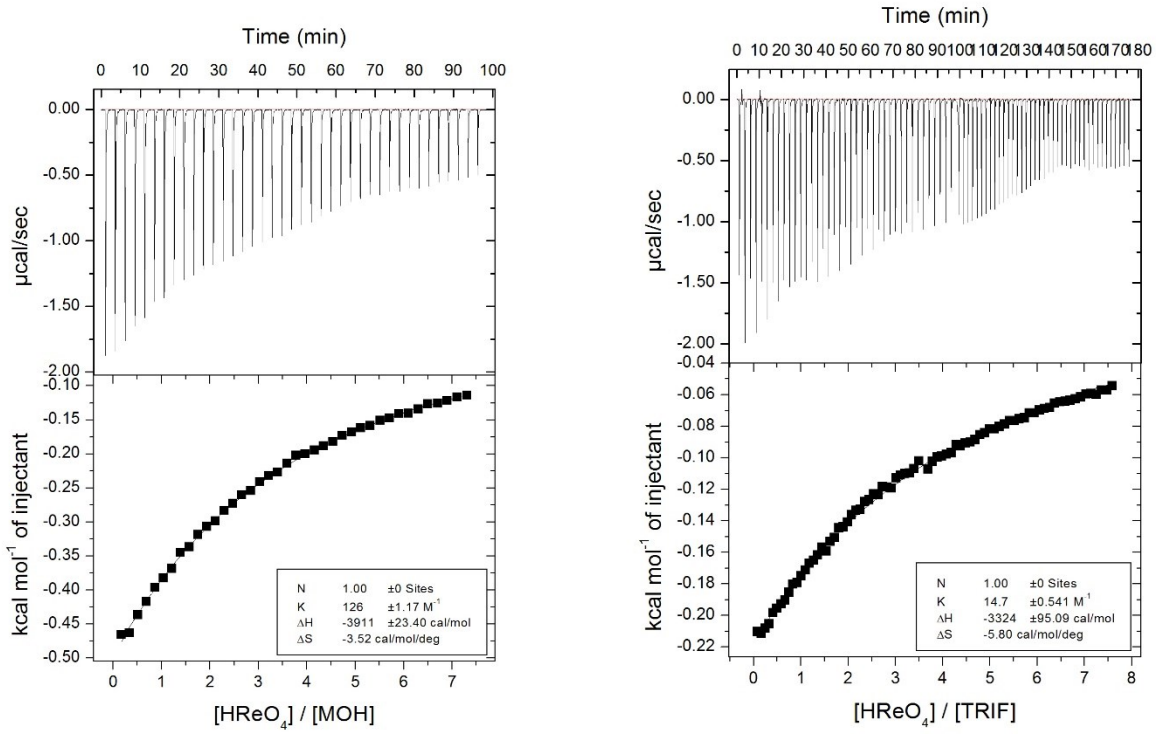


a)



b)

Figure S13: Thermograms and fit of the titration curve obtained for 39 x 1µL injections of: (a) $HReO_4$ (58.7 mM) in 0.5 M HNO_3 in a 1-ARM (1.1 mM) solution in 0.5 M HNO_3, and (b) $HReO_4$ (40.0 mM) in 0.5 M HNO_3 in Lcage (1.2 mM) solution in 0.5 M HNO_3.



a) b)

Figure S14: Thermograms and fit of the titration curve obtained for 39 x 1µL injections of: (a) $HReO_4$ (40.0 mM) in 0.5 M HNO_3 in a MOH (1.1 mM) solution in 0.5 M HNO_3, and (b) $HReO_4$ (79.6 mM) in 0.5 M HNO_3 in TRIF (4.7 mM) solution in 0.5 M HNO_3.

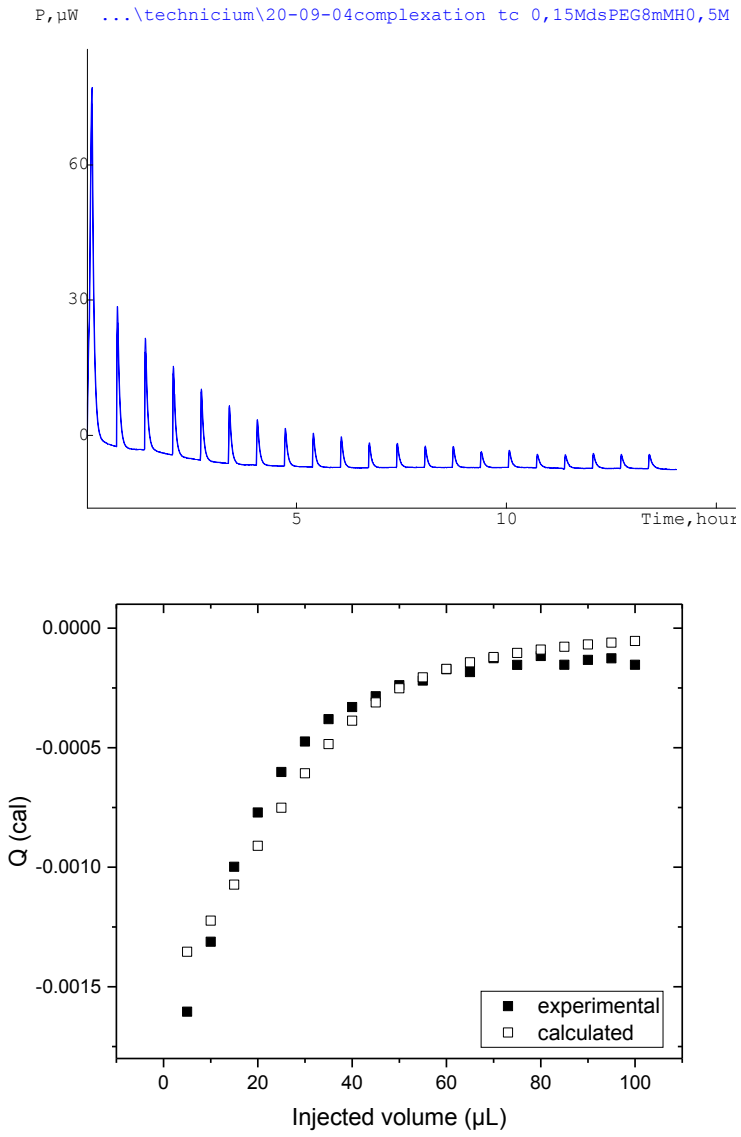


Figure S15: Thermogram ant fit obtained for 20 x 5µL injections of 0.15 M $HTcO_4$ in 0.5 M HNO_3 in a PEG azacryptand solution in 0.5 M HNO_3 (C_{PEG} = 7.7 mM).

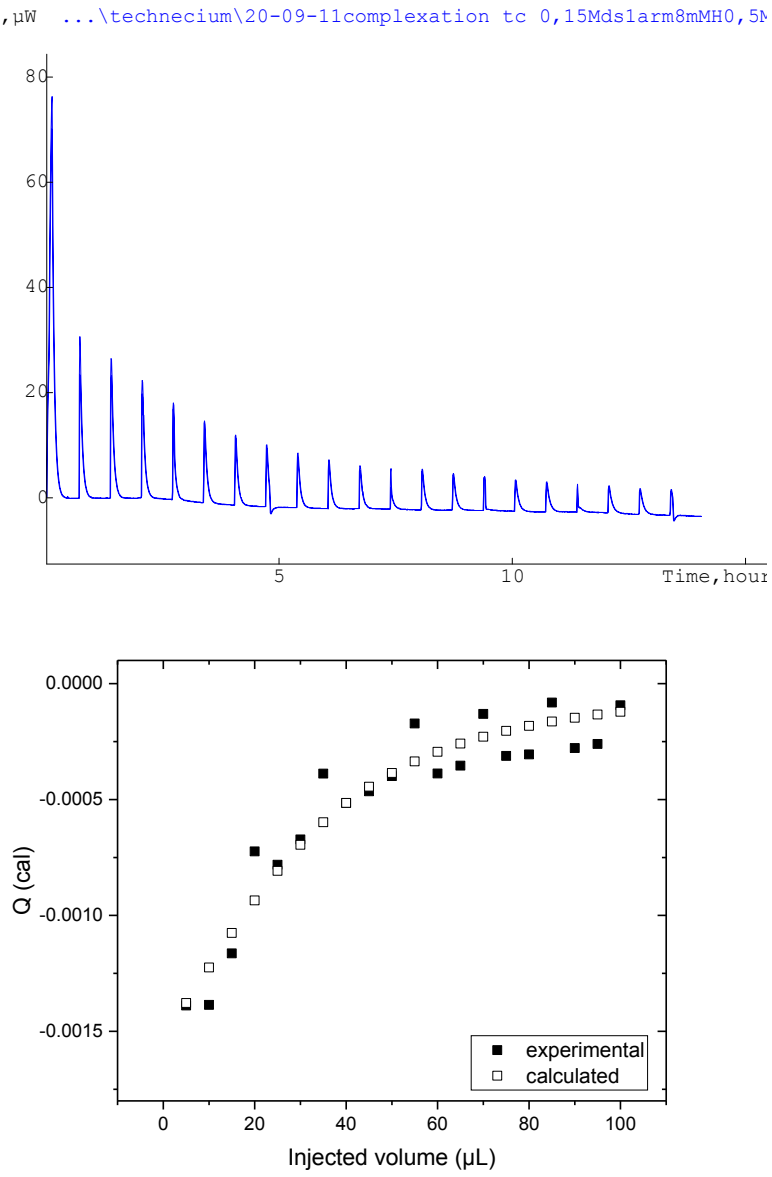


Figure S16: Thermogram and fit obtained for 20 x 5µL injections of 0.15 M $HTcO_4$ in 0.5 M HNO_3 in a 1-ARM azacryptand solution in 0.5 M HNO_3 ($C_{1\text{-ARM}}$ = 7.7 mM).

5. SC-XRD

Table S1: Features of hydrogen bond interactions involving the protonated amines and several acceptor O atom species in the $[H_6$**TRIF**$(ReO_4)](CF_3SO_3)_5 \cdot 5(H_2O)$ crystal.

Donor group	D···A (Å)	H···A (Å)	D-H···A (°)	Acceptor atom
N(2)-H(2A)	2.79(1)	1.92(1)	168.4(4)	$O(6)_{triflate}$
N(2)-H(2B)	2.82(1)	1.94(1)	170.4(5)	$O(13)_{triflate}$
N(3)-H(3A)	2.89(1)	2.05(1)	156.3(4)	$O(4w)_{water}$
N(3)-H(3B)	2.96(1)	2.08(1)	168.6(4)	$O(2w)_{water}$
N(4)-H(4A)	2.96(1)	2.14(1)	152.4(4)	$O(4)_{perrhenate}$
N(4)-H(4B)	2.74(1)	1.86(1)	171.7(4)	$O(1w)_{water}$
N(5)-H(5A)	3.08(1)	2.47(1)	126.2(4)	$O(3)_{perrhenate}$
N(5)-H(5A)	2.91(1)	2.15(1)	142.5(4)	$O(2w)_{water}$
N(5)-H(5B)	2.87(1)	2.03(1)	156.3(4)	$O(16)_{triflate}$
N(6)-H(6A)	2.98(1)	2.32(1)	131.5(4)	$O(4)_{perrhenate}$
N(6)-H(6A)	2.99(1)	2.46(1)	118.6(4)	$O(5)_{triflate}$
N(6)-H(6B)	2.98(1)	2.14(1)	157.0(4)	$O(8)_{triflate}$
N(7)-H(7A)	2.82(1)	1.99(1)	153.3(5)	$O(4w)_{water}$
N(7)-H(7B)	2.97(1)	2.17(1)	149.3(4)	$O(3)_{perrhenate}$
O(1w)	2.92(1)	n.d.	n.d.	$O(1)_{perrhenate}$
O(2w)	2.85(1)	n.d.	n.d.	$O(1)_{perrhenate}$

References

[1] a) V. Amendola, G. Alberti, G. Bergamaschi, R. Biesuz, M. Boiocchi, S. Ferrito and F.-P. Schmidtchen, Eur. J. Inorg. Chem., 2012, 3410 and references therein; b) G. Bergamaschi, M. Boiocchi, M. L. Perrone, A. Poggi, I. Viviani, V. Amendola, *Dalton Trans.*, 2014, **43**, 11352.

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[3] a) K. K. Tanabe, C. A. Allen, S. M. Cohen, *Angew. Chem. Int. Ed*., 2009, **48**, 4639; b) Y. Okada, M. Sugai, K. Chiba, *J.Org. Chem*., 2016, **81**, 10922.

[4] S. Jeon, S. Park, J. Nam, Y. Kang, J.-M. Kim, *ACS Appl. Mater. Interfaces*, 2016, **8**, 1813.

[5] A. Thevenet, C. Marie, C. Tamain, V. Amendola, A. Miljkovic, D. Guillaumont, N. Boubals and P. Guilbaud, *Dalton Trans*., 2020, **49**, 1446.