A Clipped [3]Rotaxane Derived From Bis-*nor*-*seco*-Cucurbit[10]uril

Supplementary Information

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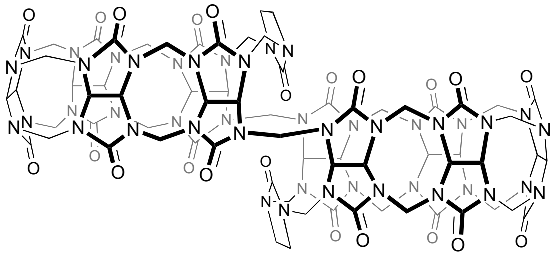
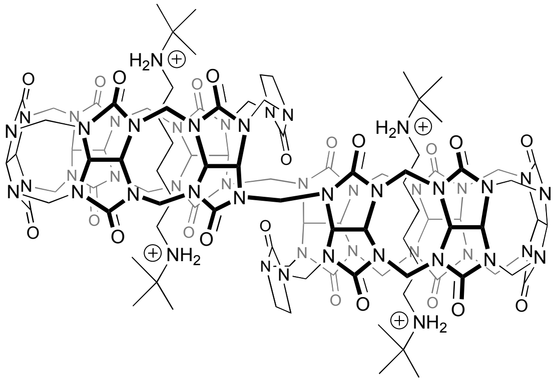
General Experimental. Starting materials were purchased from commercial suppliers and were used without further purification. Bis-*ns*-CB[10] is known in the literature.[1] Melting points were measured on a Meltemp apparatus in open capillary tubes and are uncorrected. TLC analysis was performed using pre-coated plastic plates from Merck. IR spectra were recorded on a JASCO FT/IR 4100 spectrometer and are reported in cm^{-1}. ^1H NMR spectra were measured on a Bruker DRX-400 instrument operating at 400 MHz. ^{13}C NMR spectra were measured on a Bruker DRX-500 instrument operating at 500 MHz (125 MHz for ^{13}C NMR). Mass spectrometry was performed using a JEOL AccuTOF electrospray instrument (ESI).

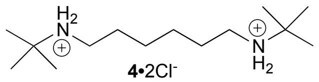
Synthetic Procedures and Characterization.



Compound **1**: To a solution of bis-*ns*-CB[10] (100.0 mg, 0.061 mmol) in HCl (8 M, 0.53 mL) in a 1 dram glass vial was added **2** (13.0 mg, 0.134 mmol) and then CH_2O (8.0 mg, 0.269 mmol). The vial was then sealed with a screw cap. The mixture was stirred and heated at 50 ºC for 1 h. The precipitate was collected by vacuum filtration. The precipitate was then washed with HCl (8 M, 0.5 mL) and H_2O (3 × 1.0 mL) and dried under high vacuum to yield **1** as a white solid (89.0 mg, 0.048 mmol, 78%). M.p. > 300 ºC. IR (cm^{-1}): 1734s, 1463m, 1378m, 1258s, 1219s, 966m, 796s, 758m. ^1H NMR (400 MHz, D_2O, as **1•3**$_2$, RT): 6.82 (d, *J* = 8.1, 4H), 6.66 (d, *J* = 8.1, 4H), 6.60 (s, 4H), 5.84 (d, *J* = 6.0, 4H), 5.80 (d, *J* = 6.0, 4H), 5.77 (s, 4H), 5.73 (d, *J* = 14.4, 4H), 5.56 (d, *J* = 16.4, 4H), 5.53 (d, *J* = 9.0, 4H), 5.47 (d, *J* = 15.6, 4H), 5.38 (d, *J* = 9.0, 4H), 5.30-5.20 (m, 8H), 4.55-4.35 (m, 16H), 4.33 (s, 8H), 4.08 (d, *J* = 15.6, 4H), 4.03 (d, *J* = 15.6, 4H), 3.76 (dd, *J* = 6.8, 8.4, 4H). ^{13}C NMR (125 MHz, D_2O, **3** as guest, RT, 1,4-dioxane as internal standard): δ 160.69, 160.20, 158.13, 157.52, 157.17, 157.03, 156.09, 133.21, 132.40, 126.57, 124.99, 71.63, 70.52, 70.28, 70.09, 66.98, 56.65, 51.50, 51.36, 51.31, 47.19, 44.46, 42.50, 41.94. MS (ESI, **3** as guest): *m/z* 711 ([M•**3**$_2$+3H]$^{3+}$).



Compound **1•4**$_2$: To a solution of bis-*ns*-CB[10] (100.0 mg, 0.061 mmol) in HCl (8 M, 0.53 mL) in a 1 dram glass vial was added **4** (37.0 mg, 0.122 mmol) and the reaction was stirred at RT until homogeneous. To the reaction mixture was added **2** (13.0 mg, 0.134 mmol) and CH_2O (8.0 mg, 0.269 mmol). The vial was then sealed with a screw cap. The mixture was stirred and heated at 50 ºC for 1 h. The precipitate was collected by vacuum filtration. The precipitate was stirred with MeOH (5.0 mL), collected by centrifugation, and dried under high vacuum to yield **1•4**$_2$ as a white solid (98.0 mg, 0.040 mmol, 69%). M.p. > 300 ºC. IR (cm^{-1}): 3459br, 1726s, 1471, 1322m, 1257m, 1223s, 1184s, 1142m, 966s, 848m, 797s, 760m. ^1H NMR (400 MHz, D_2O, RT): 6.31 (s, 4H), 5.85-5.75 (m, 12H), 5.72 (d, *J* = 15.6, 4H), 5.67 (s, 4H), 5.63 (s, 8H), 5.62 (d, *J* = 15.6, 4H) 5.51 (d, *J* = 9.4, 4H), 5.35 (d, *J* = 9.4, 4H), 4.43 (d, *J* = 15.6, 4H), 4.40-4.30 (m, 12H), 4.28 (d, *J* = 15.6, 4H), 4.17 (dd, *J* = 7.2, 8.6, 4H), 3.57 (dd, *J* = 7.2, 8.6, 4H), 3.0-2.80 (m, 8H), 1.55 (s, 18H), 1.46 (s, 18H), 0.75-0.60 (m, 12H), 0.60-0.50 (m, 4H). ^{13}C NMR (125 MHz, D_2O, RT, 1-4-dioxane as internal reference): δ 164.08, 160.30, 159.58, 158.45, 156.98, 156.85, 156.66, 156.39, 71.57, 70.60, 70.53, 70.37, 67.02, 57.21, 56.24, 51.56, 51.33, 47.39, 44.49, 42.06, 41.66, 26.96, 26.82, 25.93, 25.86, 25.54. MS (ESI): *m/z* 772.3 ($[M+3H]^{3+}$).



4•2Cl$^-$

Compound **4**•2Cl$^-$: To a stirring and refluxing solution of *t*-butylamine (10.0 g, 41.0 mmol) in THF (7.0 mL) in a 3-neck RB flask was added a solution of 1,6-dibromohexane (18.0 g, 246.1 mmol) in THF (8.0 mL) dropwise. The reaction mixture was stirred at reflux for 3 h at which point a solution of KOH (5.5 g, 98.4 mmol) in H_2O (3.3 mL) was added. The reaction mixture was stirred at reflux for 14 h then cooled to RT. The KBr salt produced from the reaction was filtered off, and the filtrate was concentrated by rotary evaporation to give a crude oil. The crude oil was dissolved in toluene (30.0 mL) and the mixture was concentrated by rotary evaporation and dried under high vacuum. The crude waxy solid was purified by column chromatography (4:1 $CHCl_3$/MeOH, 2% NH_4OH, $R_f = 0.25$) to yield **4** as a waxy solid (7.5 g, 32.9 mmol, 80%). The spectroscopic data (^{1}H NMR and ^{13}C NMR) matches that reported in the literature.[2] Compound **4**•2HCl was obtained by adding **4** (7.5 g, 32.9 mmol) to H_2O (100.0 mL) followed by the addition of conc. HCl until pH = 2. The homogeneous mixture was then concentrated by rotary evaporation and dried under high vacuum to yield **4**•2HCl as a white solid (9.8 g, 98%). M.p. > 300 °C. IR (cm^{-1}): 3514s, 3465s, 3069m, 2978s, 2870m, 2803s, 2482m, 2437m, 1641m, 1598s, 1482m, 1447s, 1409s, 1382s, 1257m, 1216s, 997s, 877m, 794m. ^{1}H NMR (400 MHz, D_2O, RT): 2.99 (t, 4H), 1.63 (p, 4H), 1.43 (p, 4H), 1.34 (s, 18H). ^{13}C NMR (125 MHz, D_2O, RT, 1,4-dioxane as internal reference): δ 56.94, 41.28, 26.08, 25.50, 24.96. MS (ESI): *m/z* 229.3 ($[M+H]^+$).

Sample ^{1}H NMR experiment for the formation of complex **1**•**3**$_2$: To a solution of **3** (2.1 mg, 0.01 mmol) in D_2O (1.0 mL) in a 1 dram glass vial was added an excess of solid **1** (11.0 mg, 0.006 mmol) and stirred at RT overnight. The heterogeneous mixture was filtered through a 0.2 μm PES (polyethersulfone) filtering device into an NMR tube. The ^{1}H NMR spectra were recorded at RT.

References:
1) Huang, W. -H.; Liu, S.; Zavalij, P. Y.; Isaacs, L. *J. Am. Chem. Soc.* **2006**, *128*, 14744-14745.
2) Nagel, M.; Hany, R.; Lippert, T.; Molberg, M.; Nuesch, F. A.; Rentsch, D. *Macromol. Chem. Phys.* **2007**, *208*, 277-286.

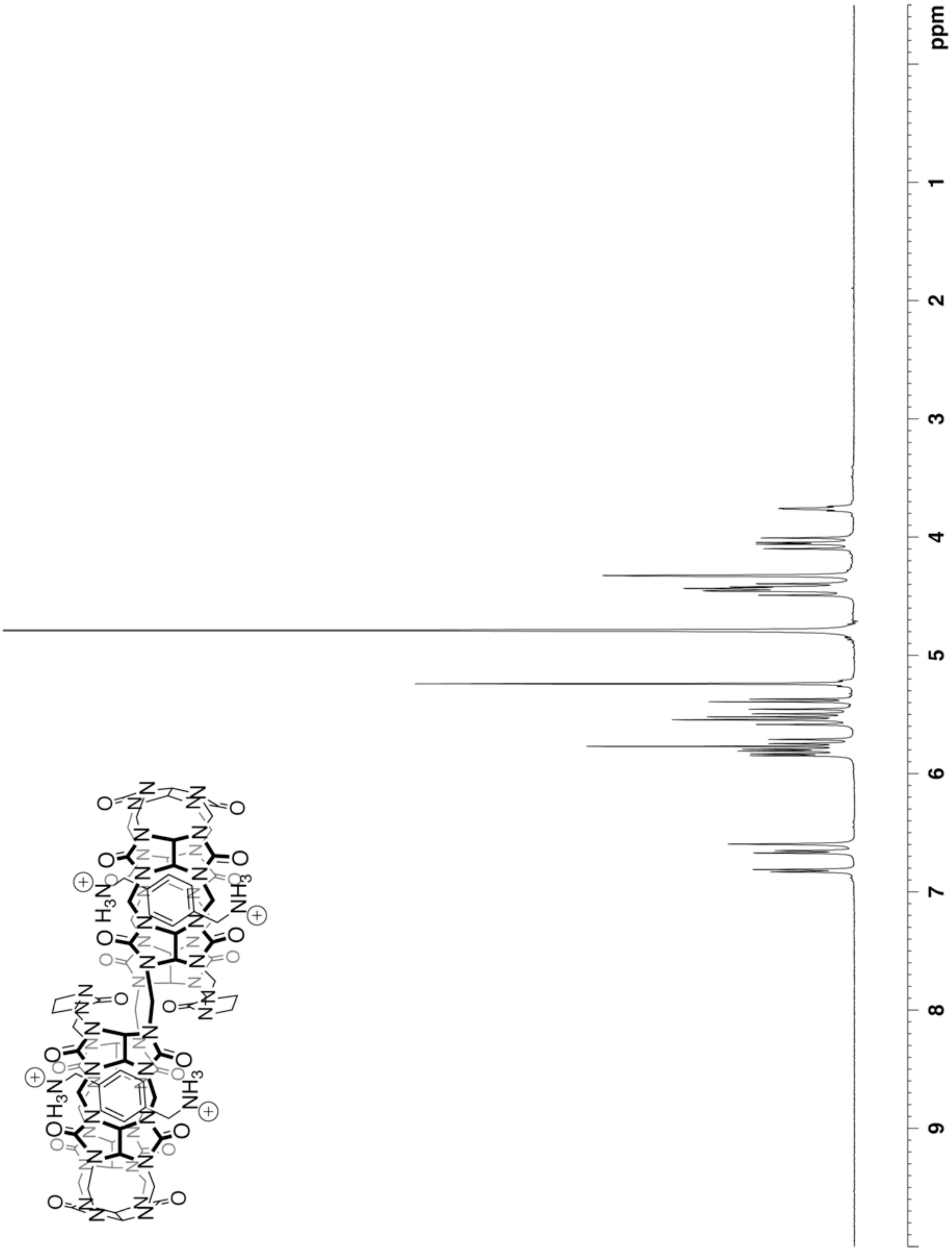


Figure 1. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for a mixture of *p*-xylenediammonium dihydrochloride and **1** (2:1 ratio).

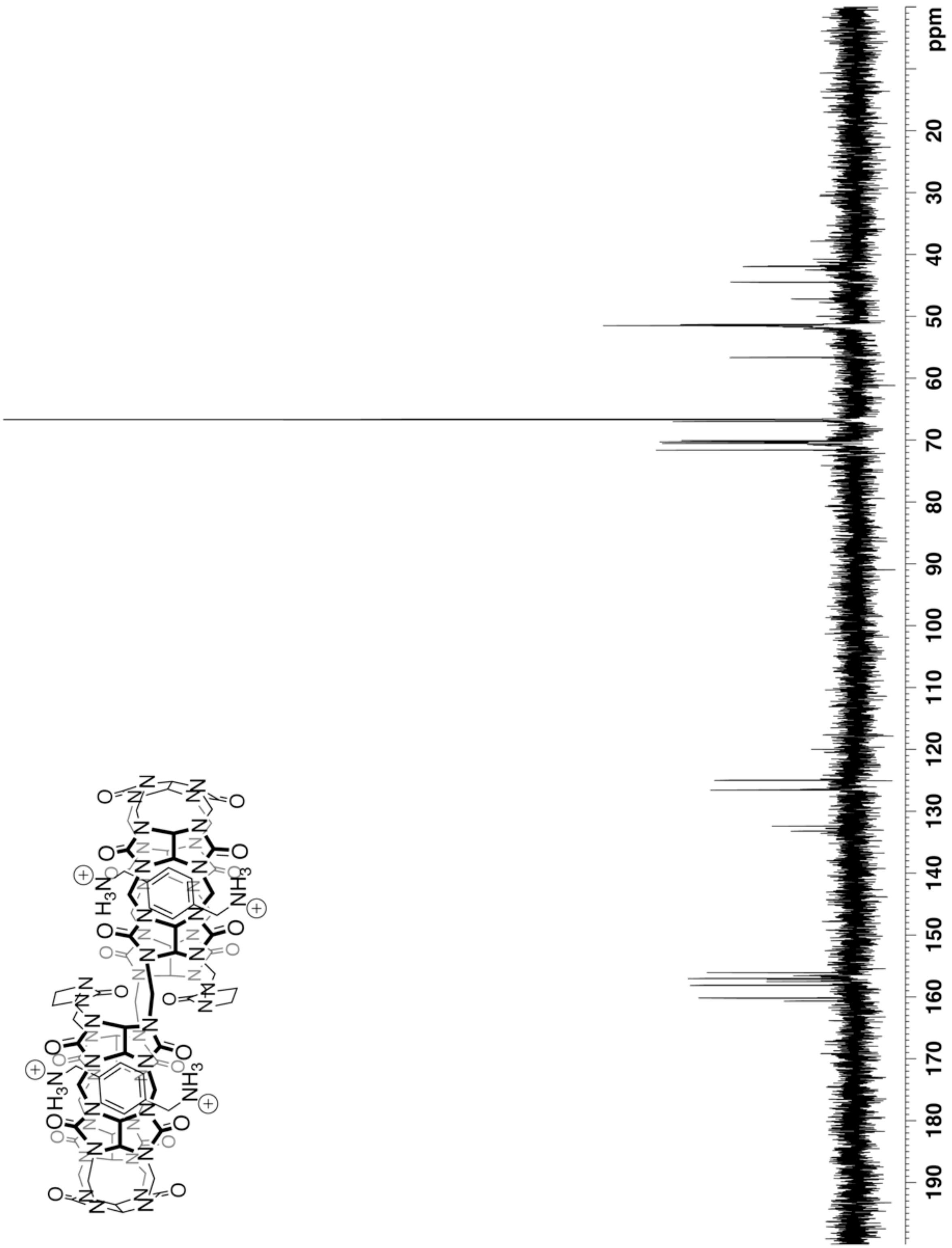


Figure 2. ^{13}C spectrum (125 MHz, D_2O, 1,4-dioxane as internal reference, RT) recorded for a mixture of *p*-xylenediammonium dihydrochloride and **1** (2:1 ratio).

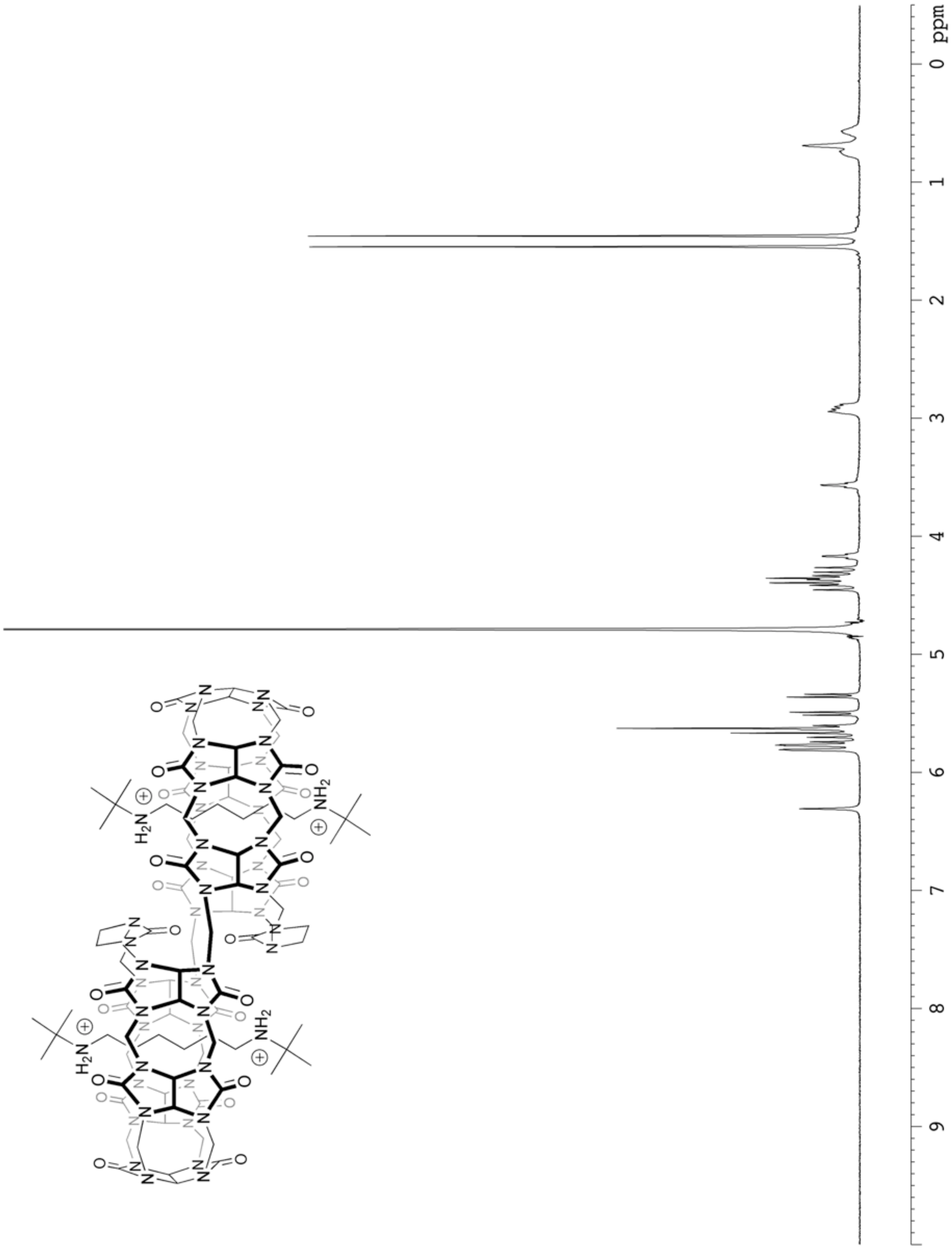


Figure 3. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for [3]rotaxane **1•4**$_2$.

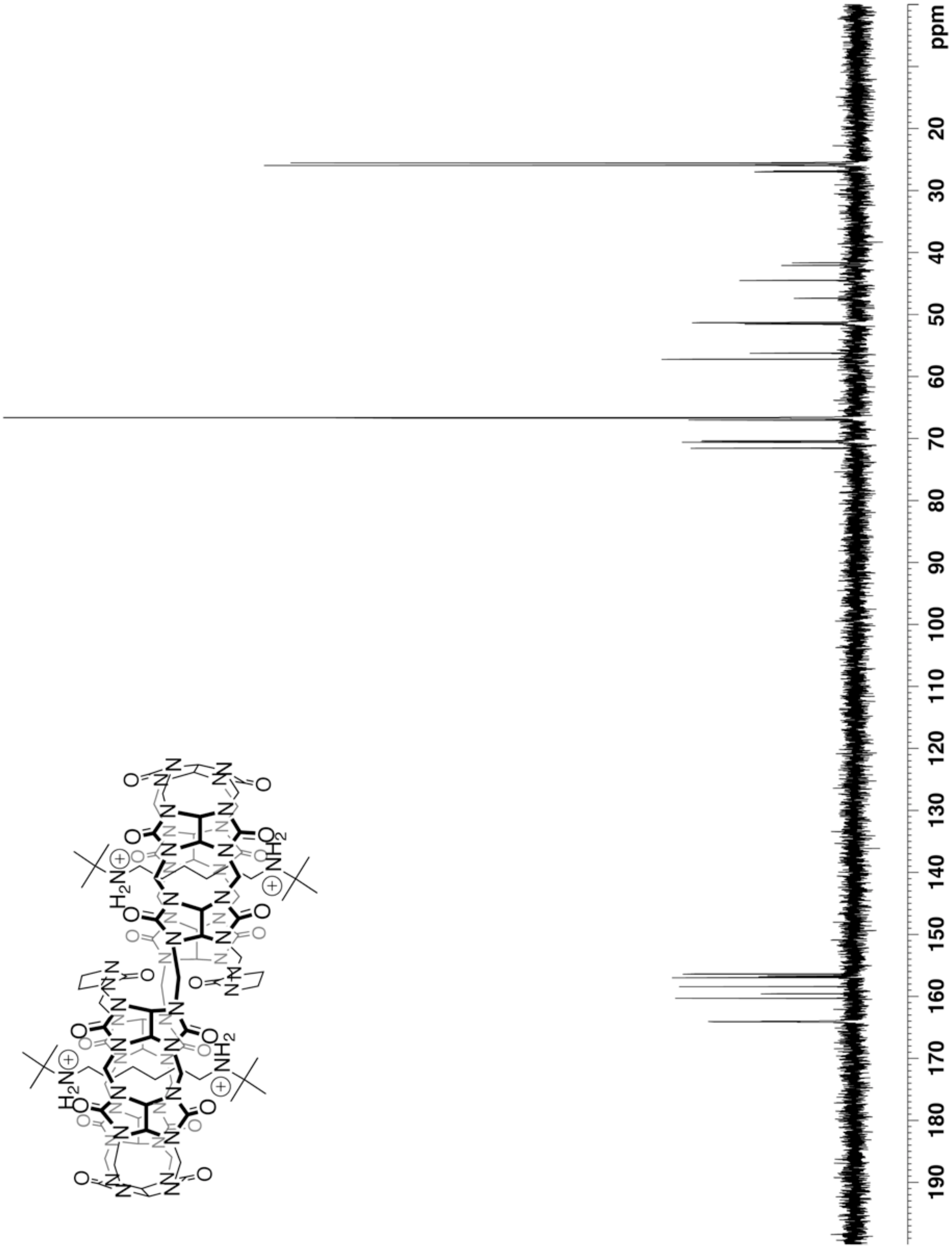


Figure 4. ^{13}C Spectrum (125 MHz, D_2O, 1,4-dioxane as internal reference, RT) recorded for [3]rotaxane **1•4**$_2$.

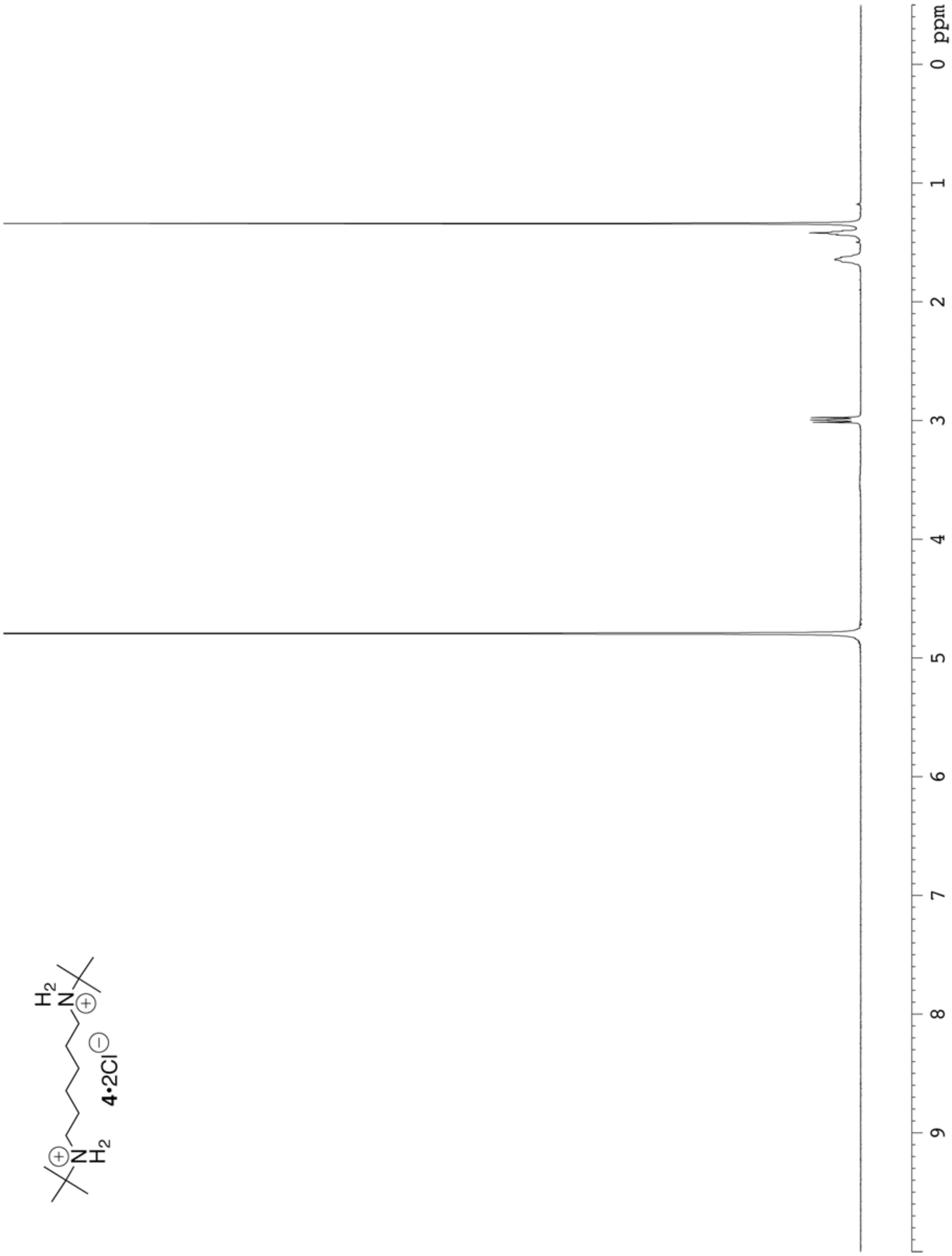


Figure 5. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for compound **4**•2Cl$^-$.

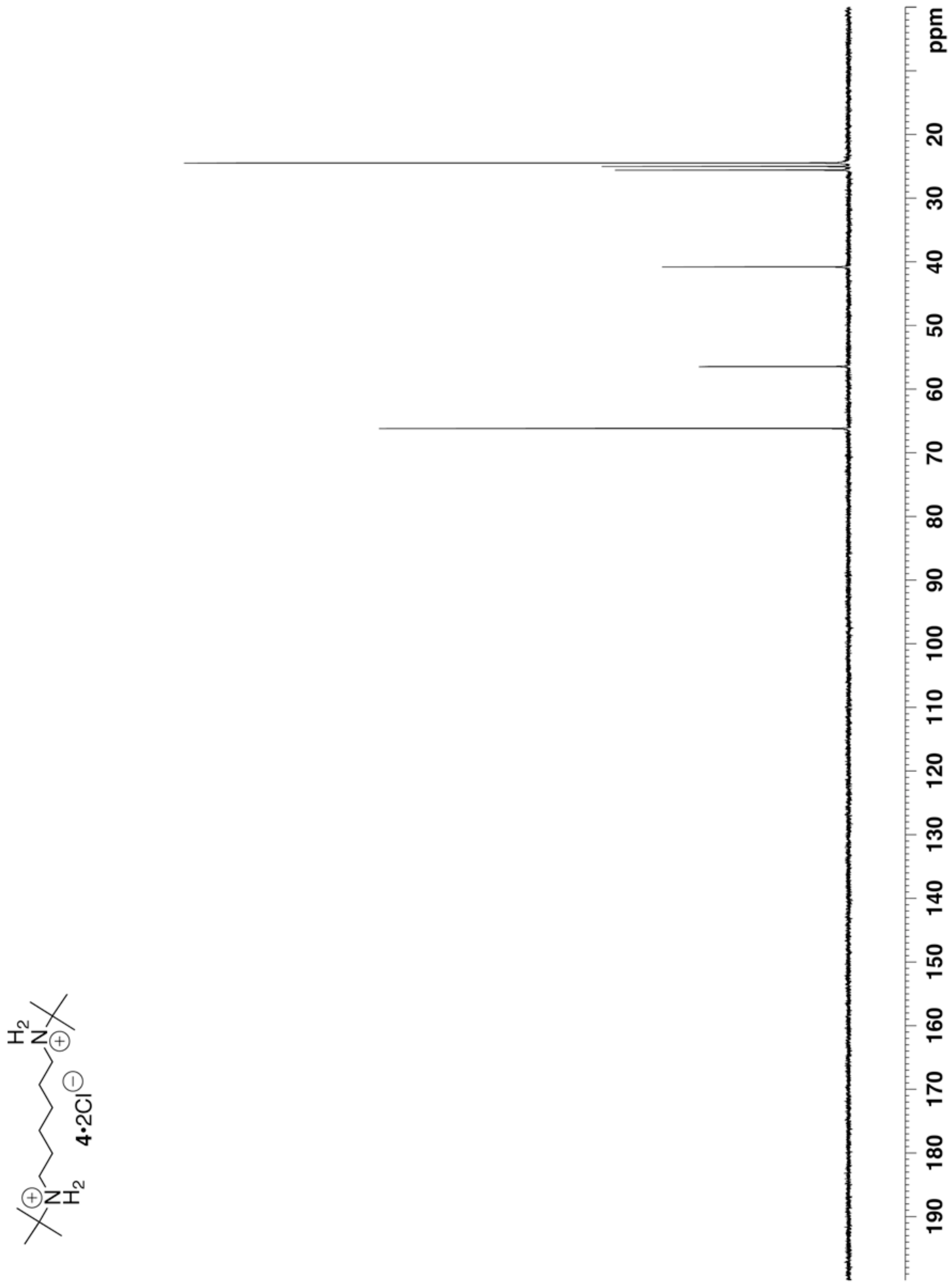


Figure 6. ^{13}C NMR spectrum (125 MHz, D_2O, 1,4-dioxane as internal reference, RT) recorded for compound **4**•$2Cl^-$.

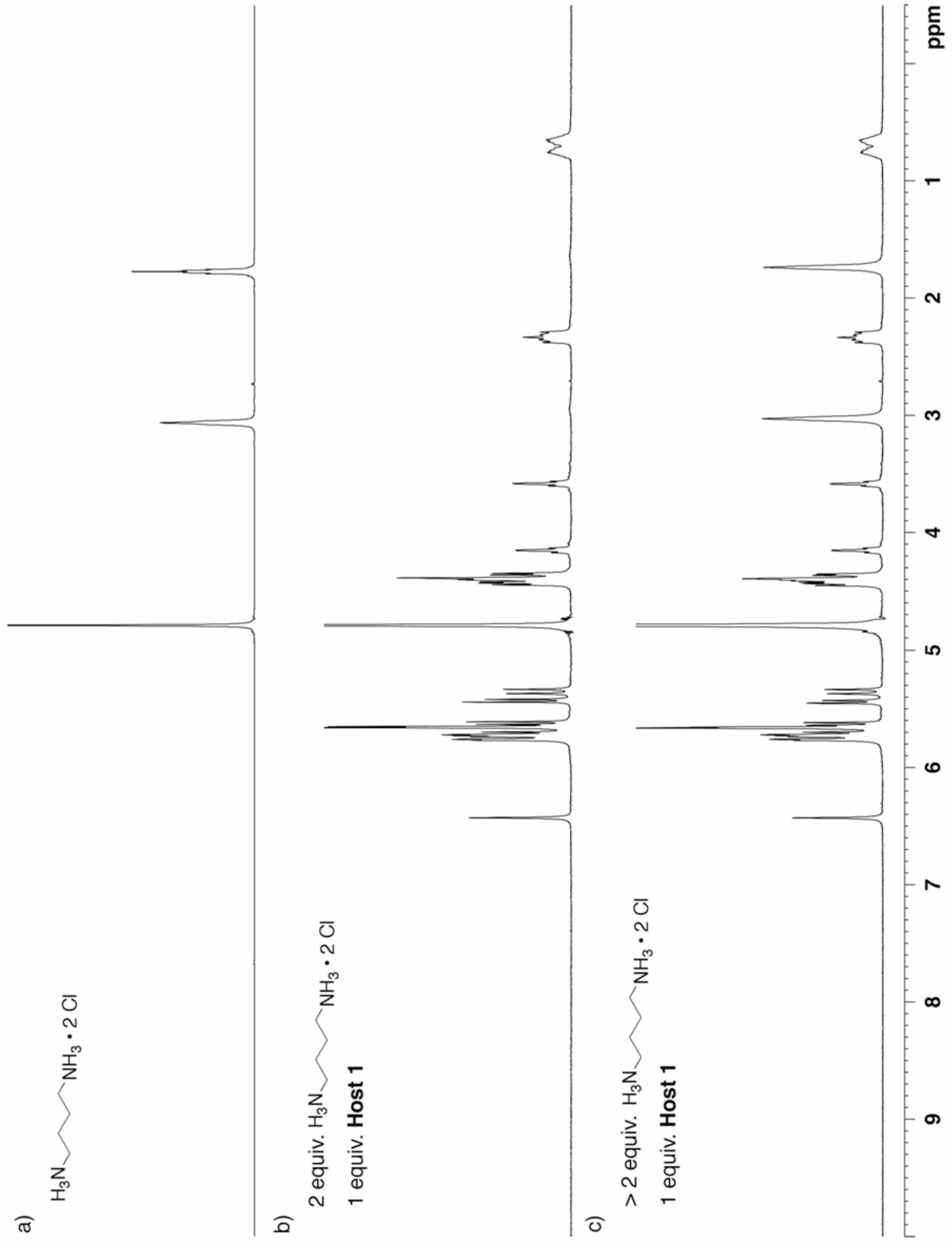


Figure 7. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,4-butanediammonium dihydrochloride, b) a mixture of 1,4-butanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,4-butanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

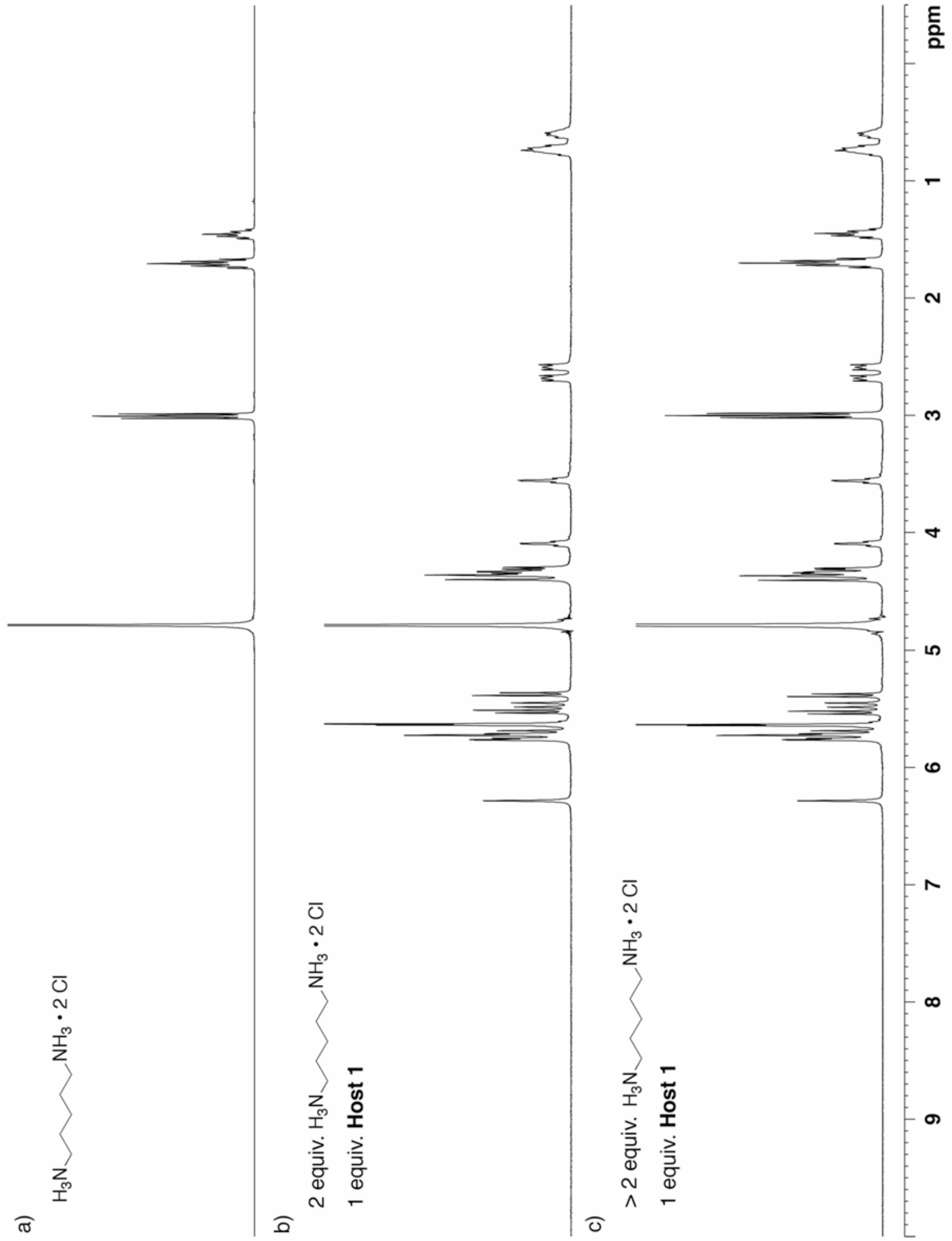


Figure 8. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,5-pentanediammonium dihydrochloride, b) a mixture of 1,5-pentanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,5-pentanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

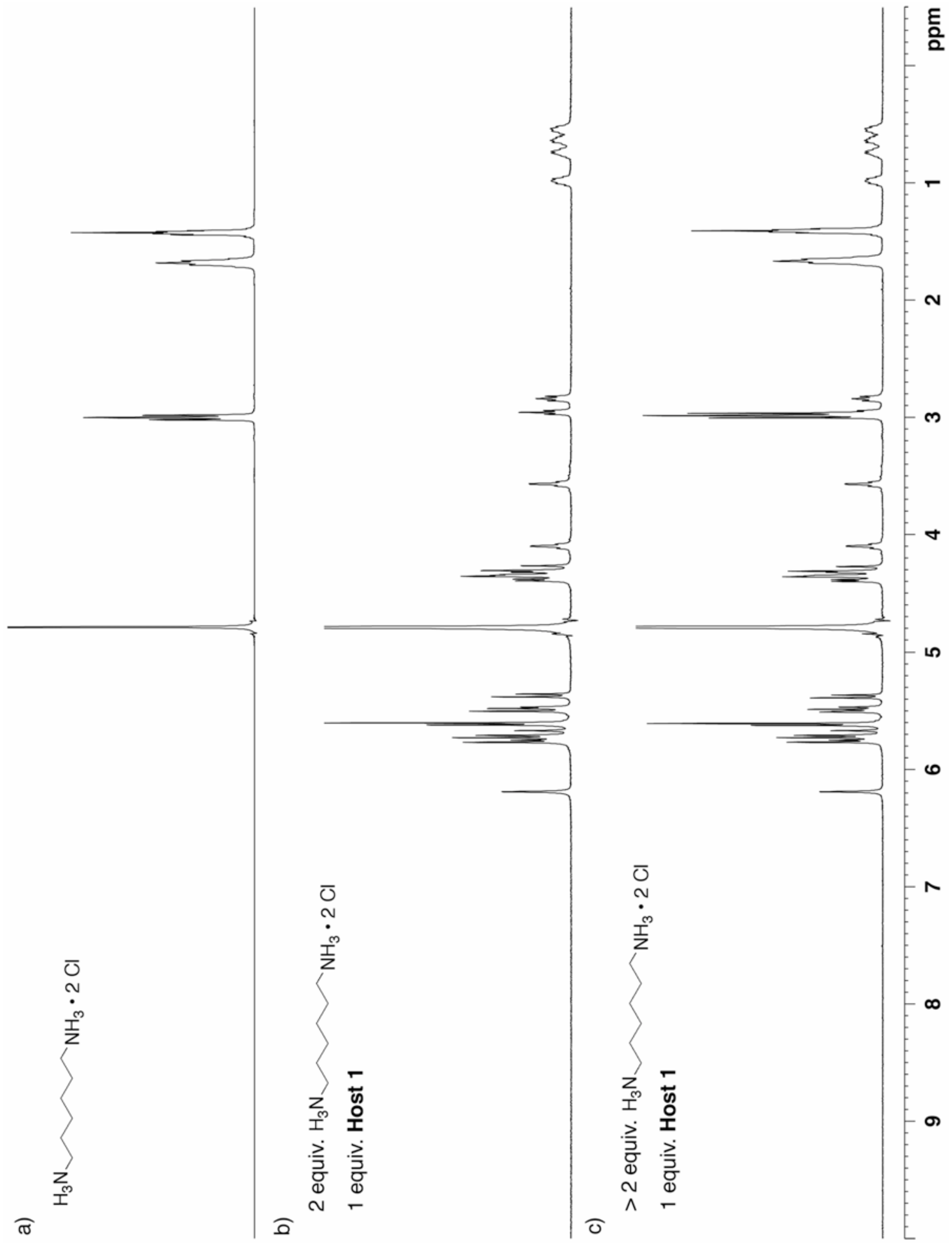


Figure 9. ^1H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,6-hexanediammonium dihydrochloride, b) a mixture of 1,6-hexanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,6-hexanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

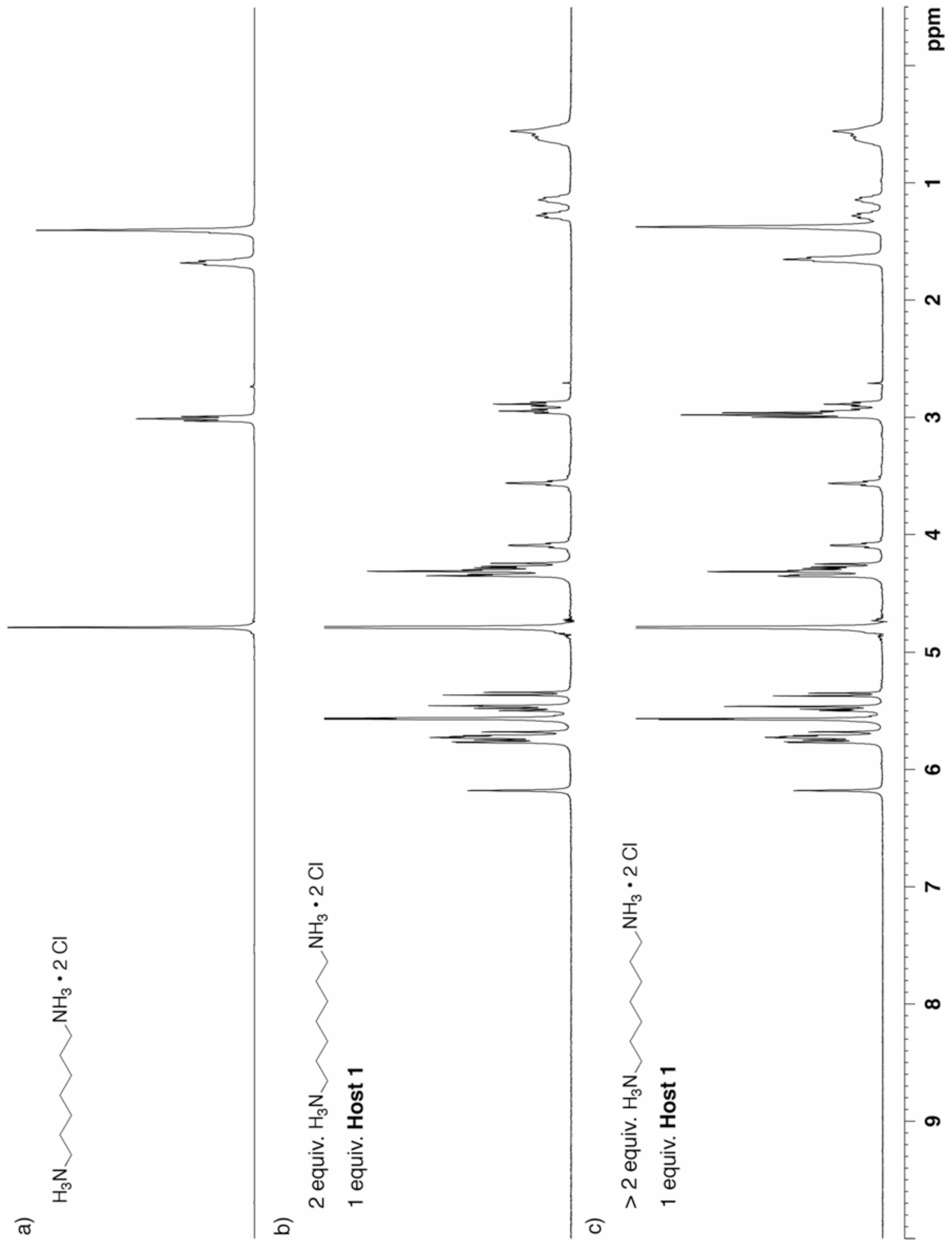


Figure 10. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,7-heptanediammonium dihydrochloride, b) a mixture of 1,7-heptanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,7-heptanediammonium dihydrochloride and **1** (>2:1 ratio).

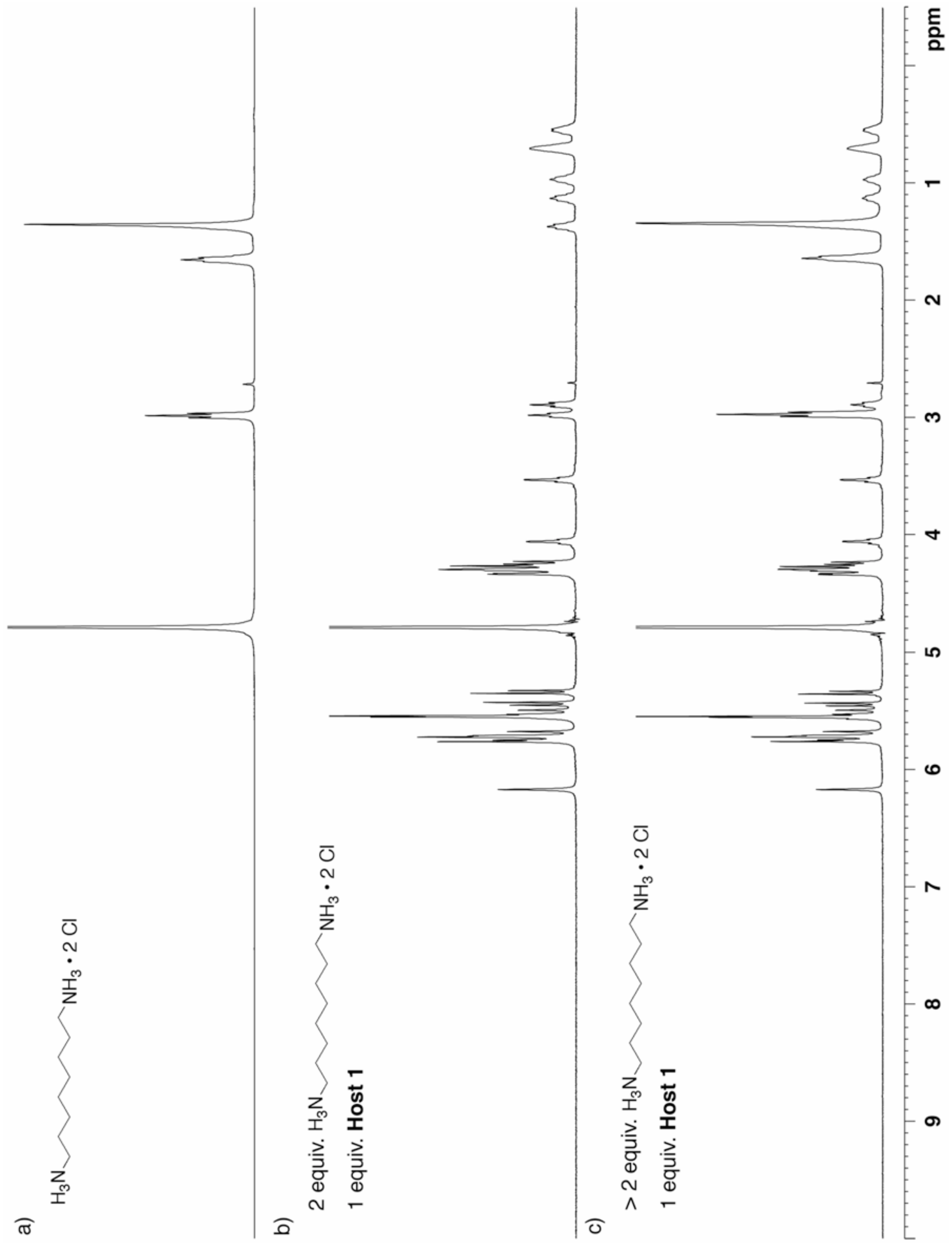


Figure 11. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,8-octanediammonium dihydrochloride, b) a mixture of 1,8-octanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,8-octanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

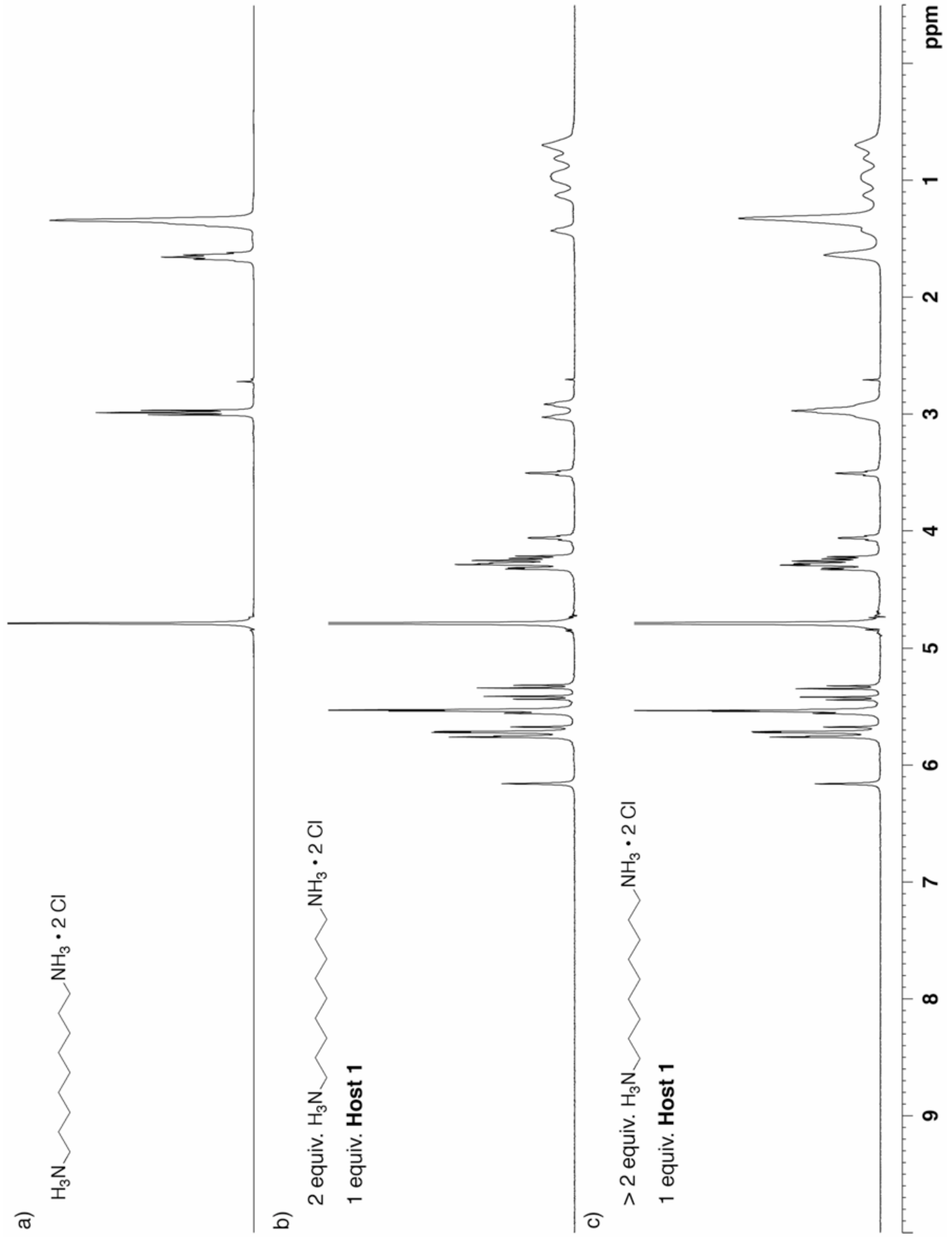


Figure 12. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,9-nonanediammonium dihydrochloride, b) a mixture of 1,9-nonanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,9-nonanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

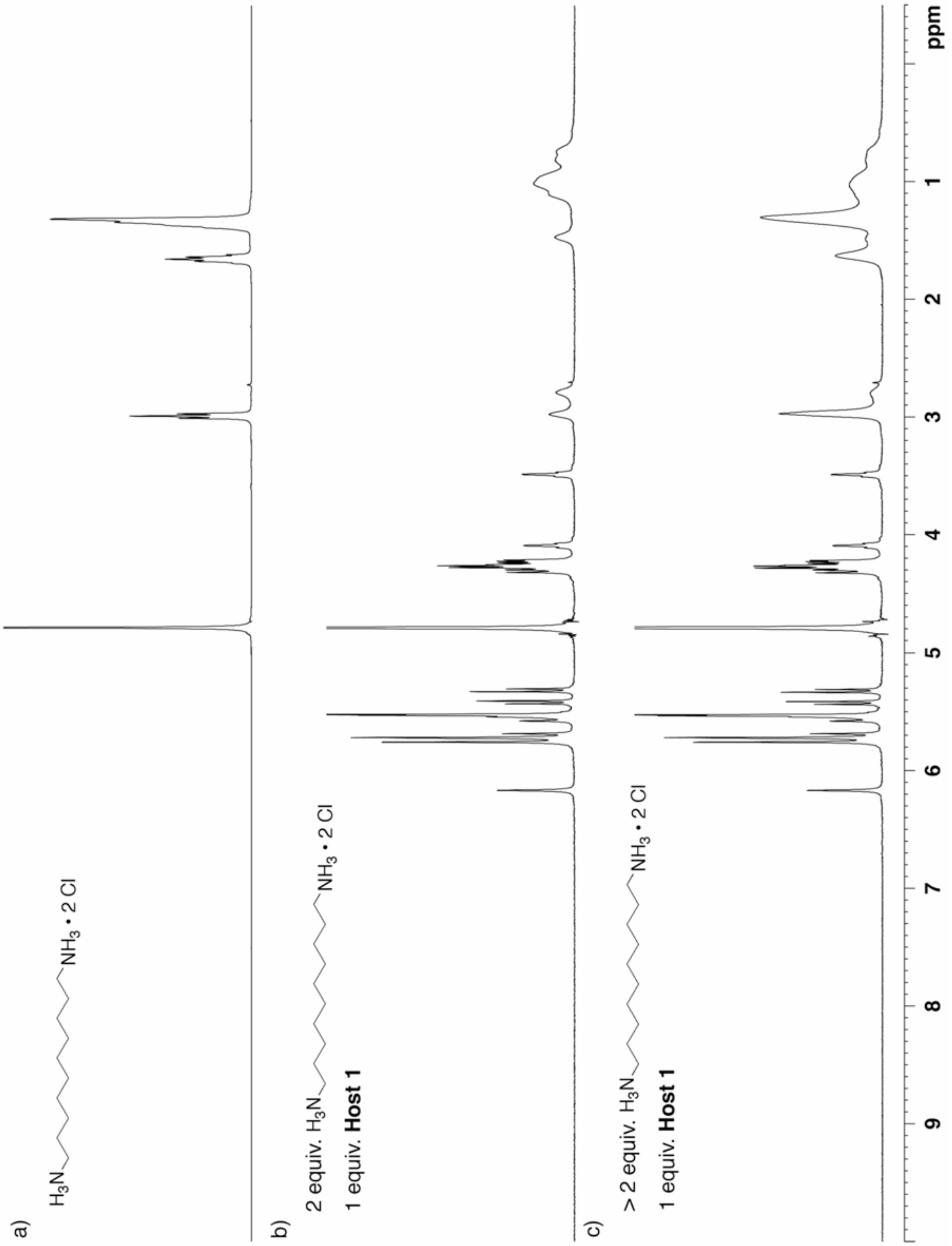


Figure 13. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,10-decanediammonium dihydrochloride, b) a mixture of 1,10-decanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,10-decanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

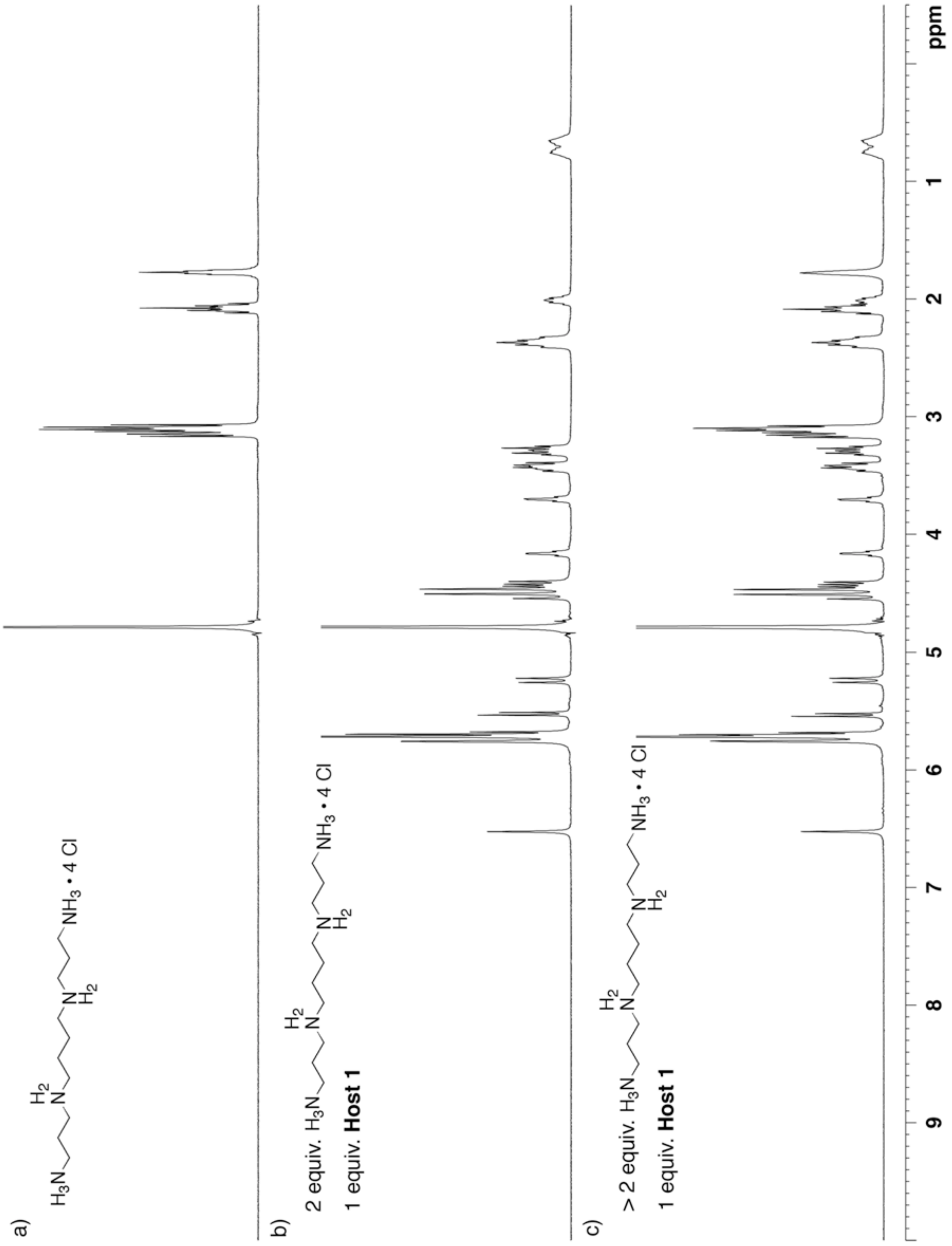


Figure 14. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) spermine tetrahydrochloride, b) a mixture of spermine tetrahydrochloride and **1** (2:1 ratio), and c) a mixture of spermine tetrahydrochloride and **1** (>2:1 ratio)**.**

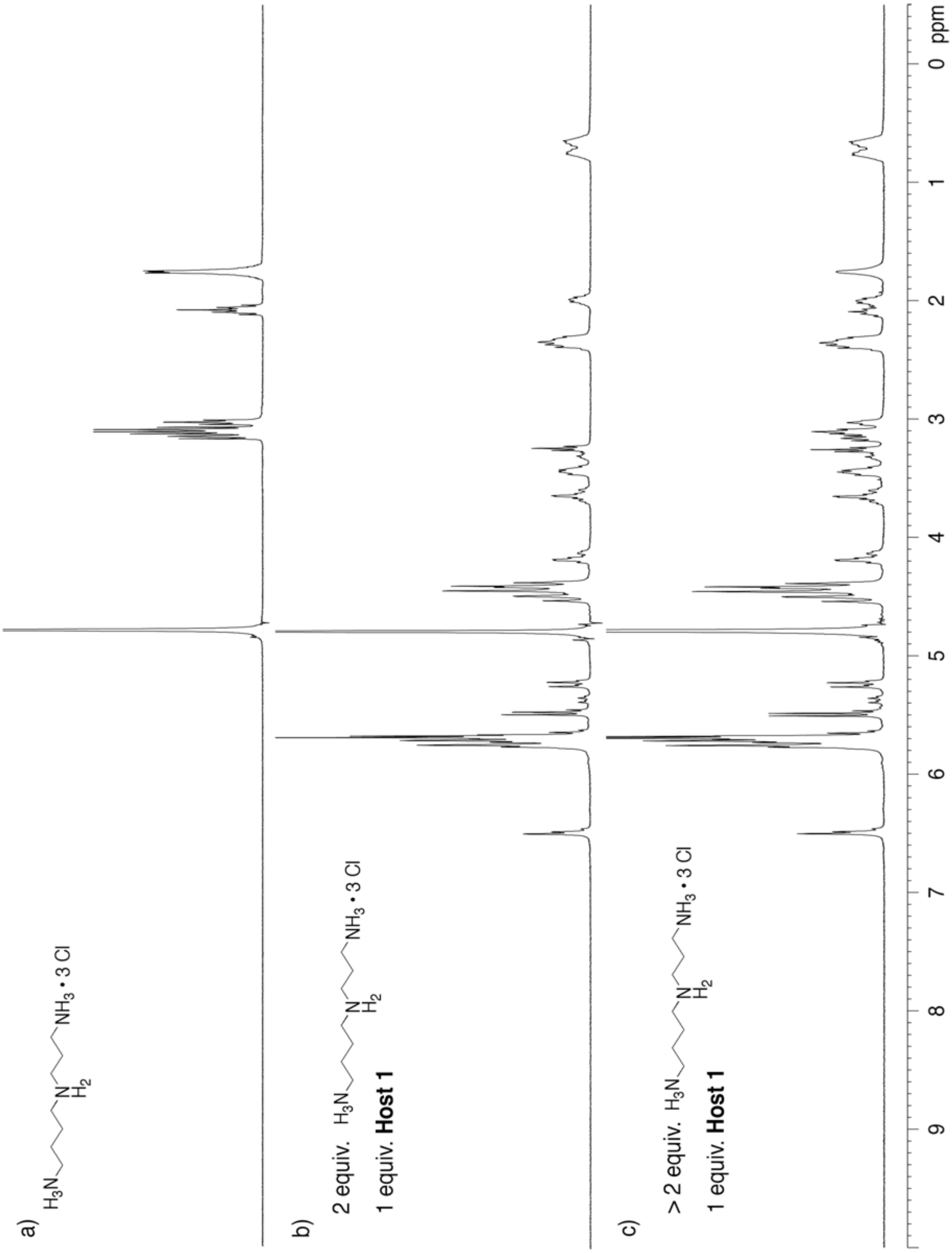


Figure 15. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) spermidine trihydrochloride, b) a mixture of spermidine trihydrochloride and **1** (2:1 ratio), and c) a mixture of spermidine trihydrochloride and **1** (>2:1 ratio)**.**

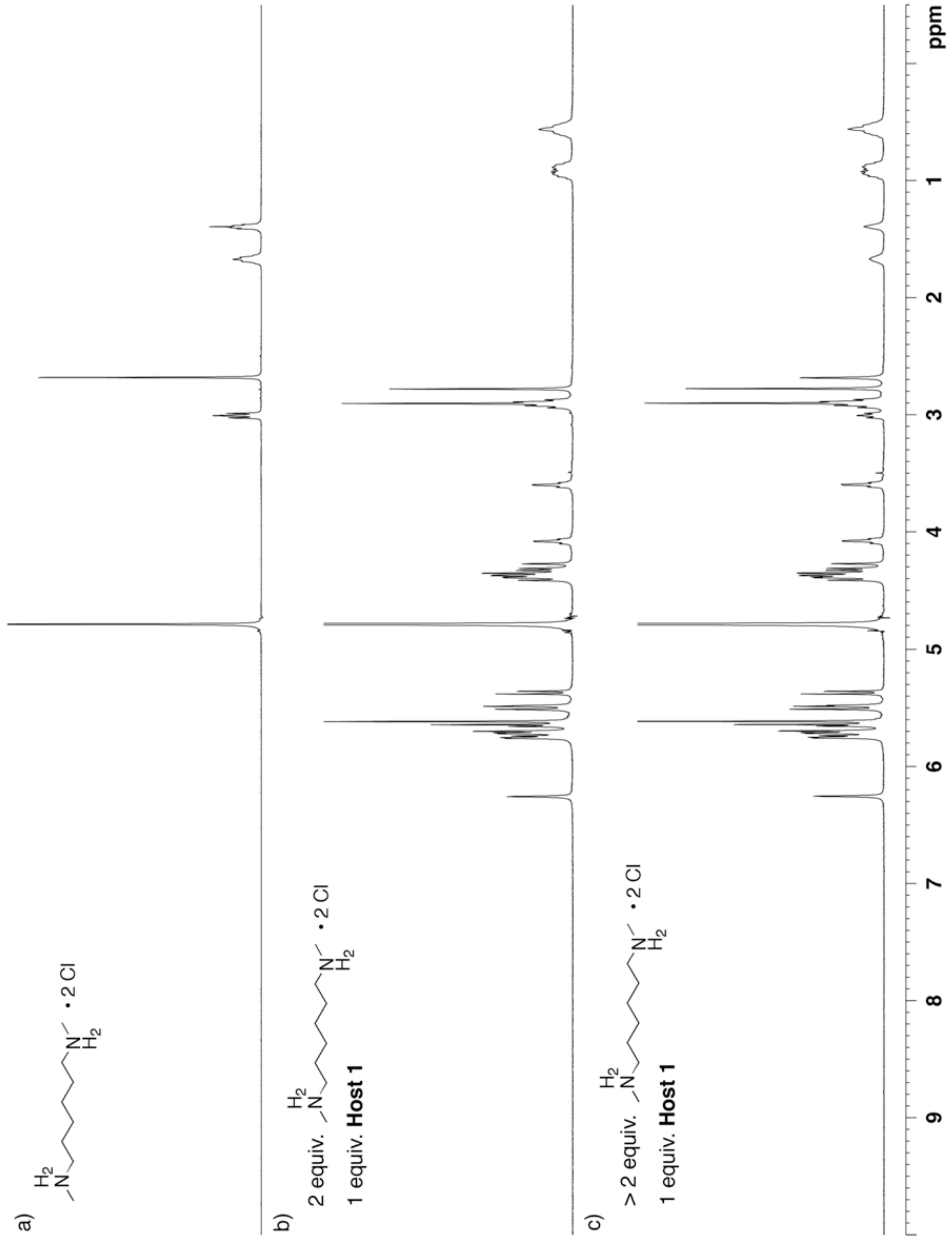


Figure 16. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) N,N'-dimethyldiammonium dihydrochloride, b) a mixture of N,N'-dimethyldiammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of N,N'-dimethyldiammonium dihydrochloride and **1** (>2:1 ratio).

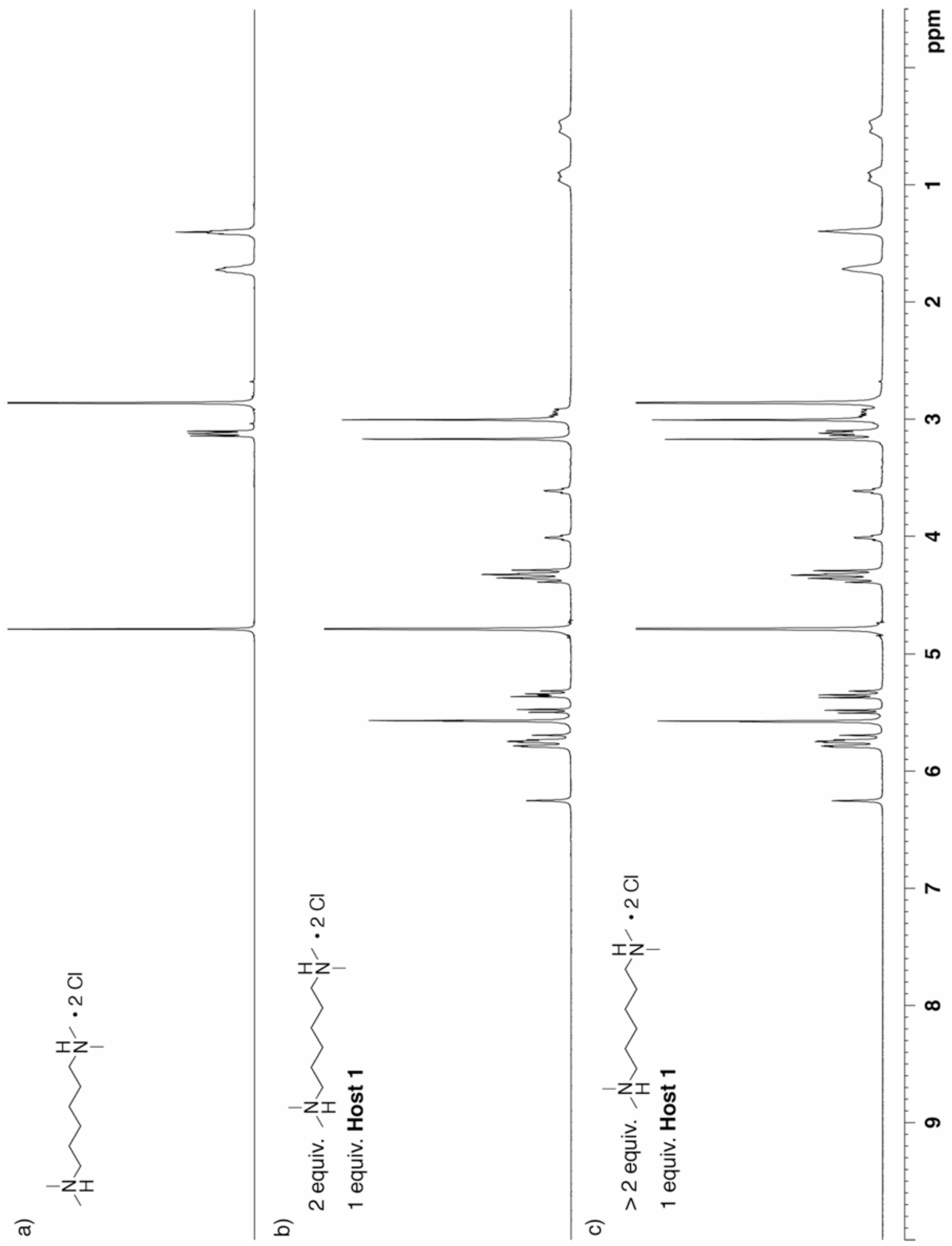


Figure 17. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) N,N,N’,N’-tetramethyldiammonium dihydrochloride, b) a mixture of N,N,N’,N’-tetramethyldiammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of N,N,N’,N’-tetramethyldiammonium dihydrochloride and **1** (>2:1 ratio)**.**

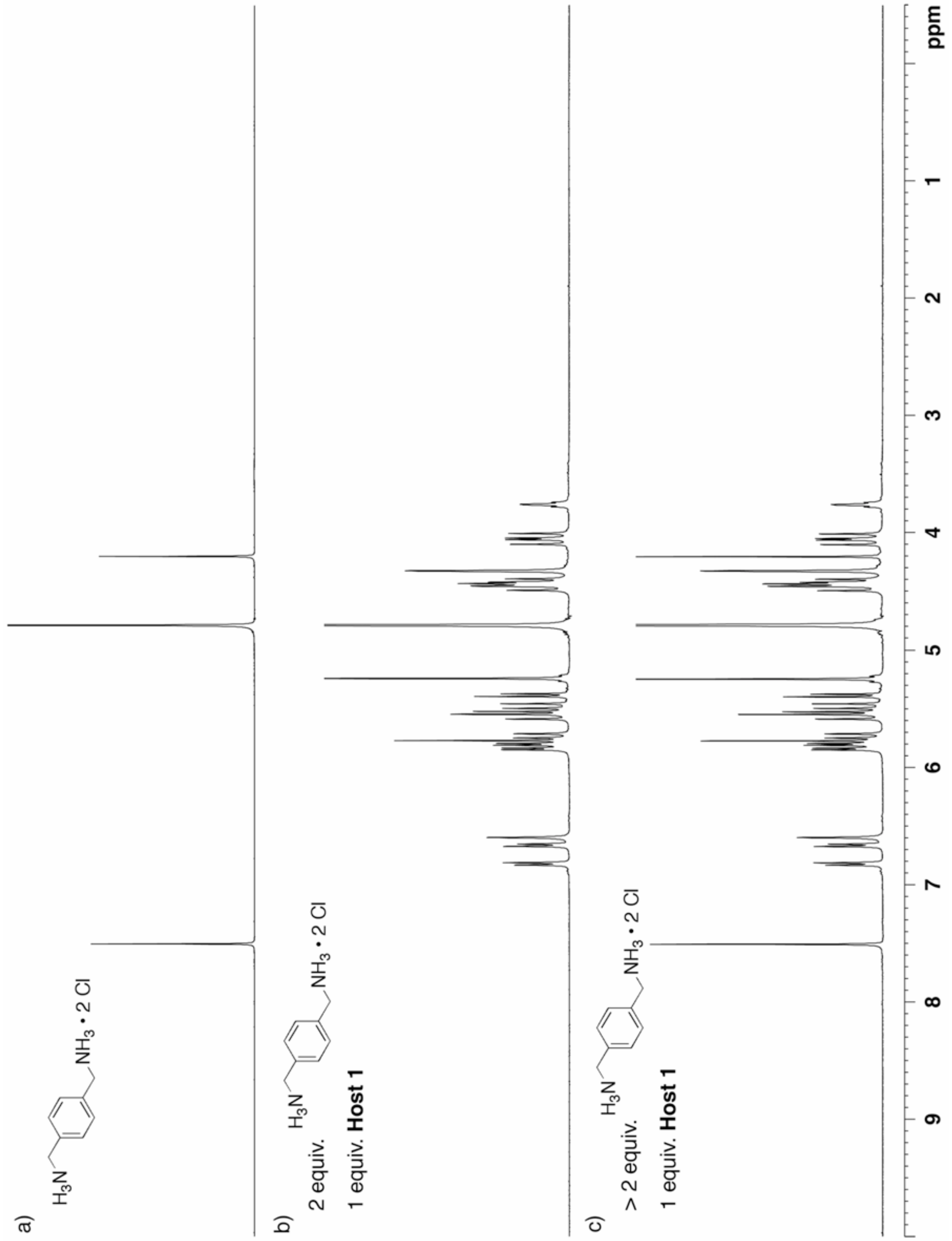


Figure 18. ^1H NMR spectra (400 MHz, D_2O, RT) recorded for: a) *p*-xylenediammonium dihydrochloride, b) a mixture of *p*-xylenediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of *p*-xylenediammonium dihydrochloride and **1** (>2:1 ratio)**.**

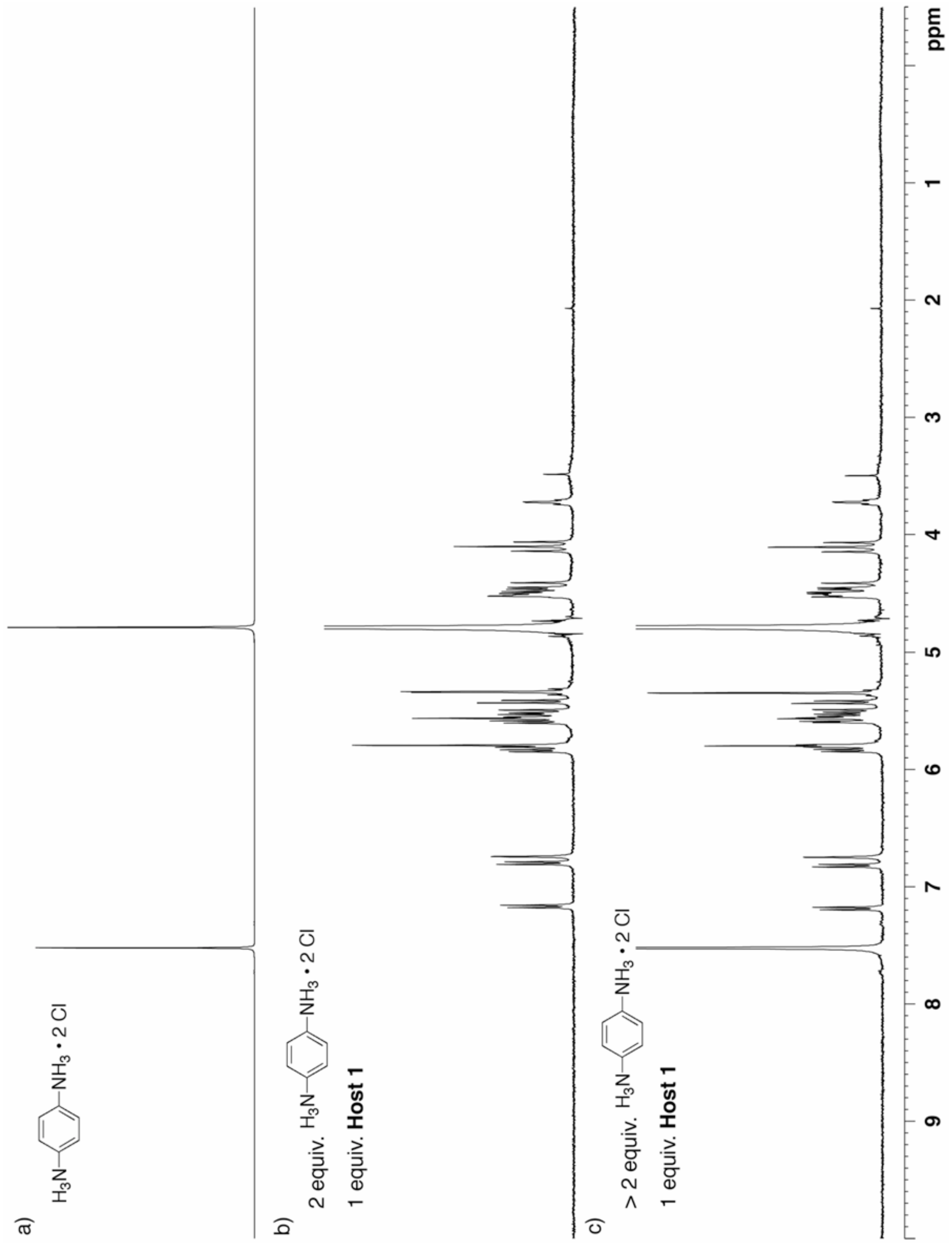


Figure 19. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) *p*-phenylenediammonium dihydrochloride, b) a mixture of *p*-phenylenediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of *p*-phenylenediammonium dihydrochloride and **1** (>2:1 ratio).

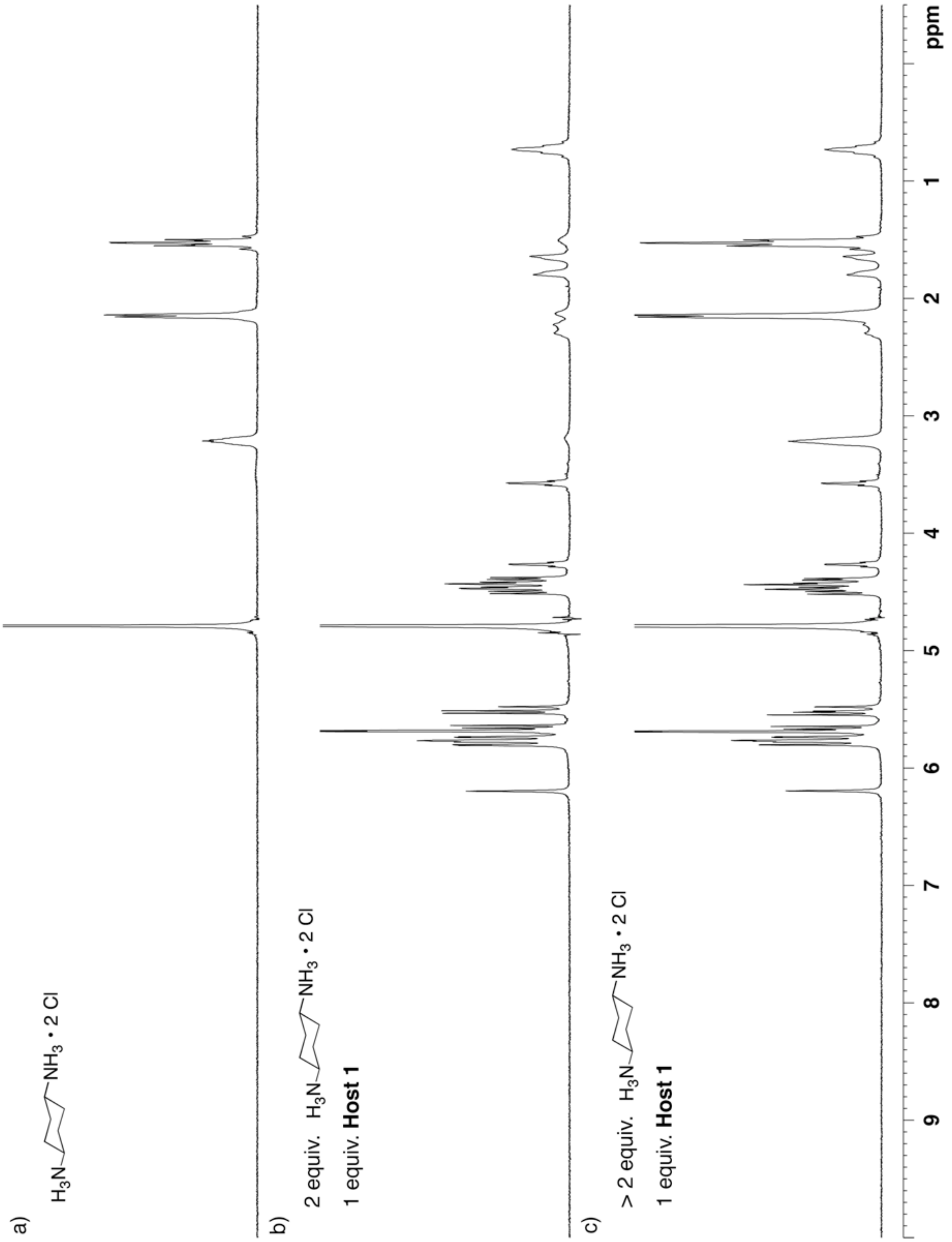


Figure 20. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) 1,4-cyclohexanediammonium dihydrochloride, b) a mixture of 1,4-cyclohexanediammonium dihydrochloride and **1** (2:1 ratio), and c) a mixture of 1,4-cyclohexanediammonium dihydrochloride and **1** (>2:1 ratio)**.**

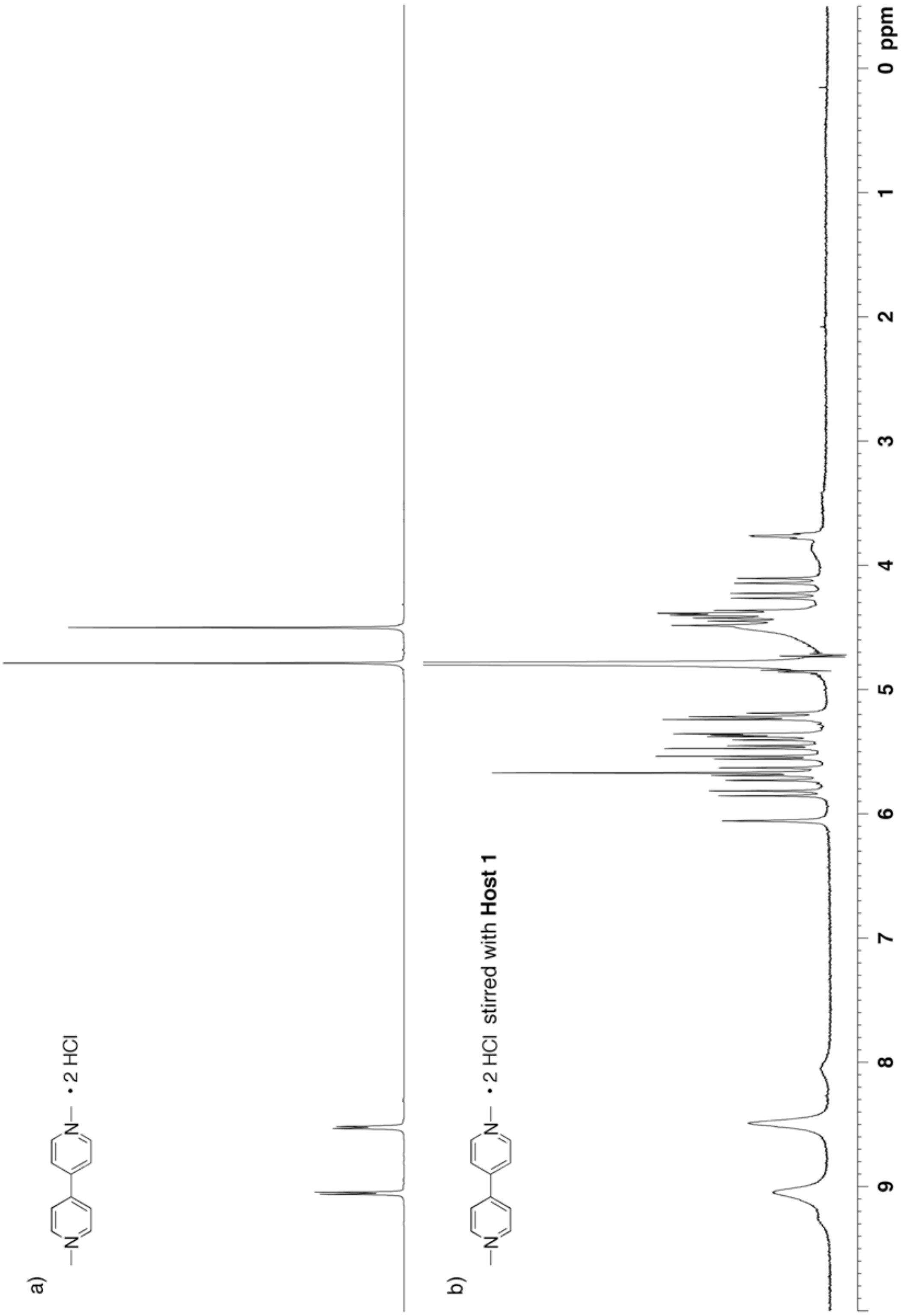


Figure 21. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) methyl viologen and b) a mixture of methyl viologen and **1** (2:1 ratio).

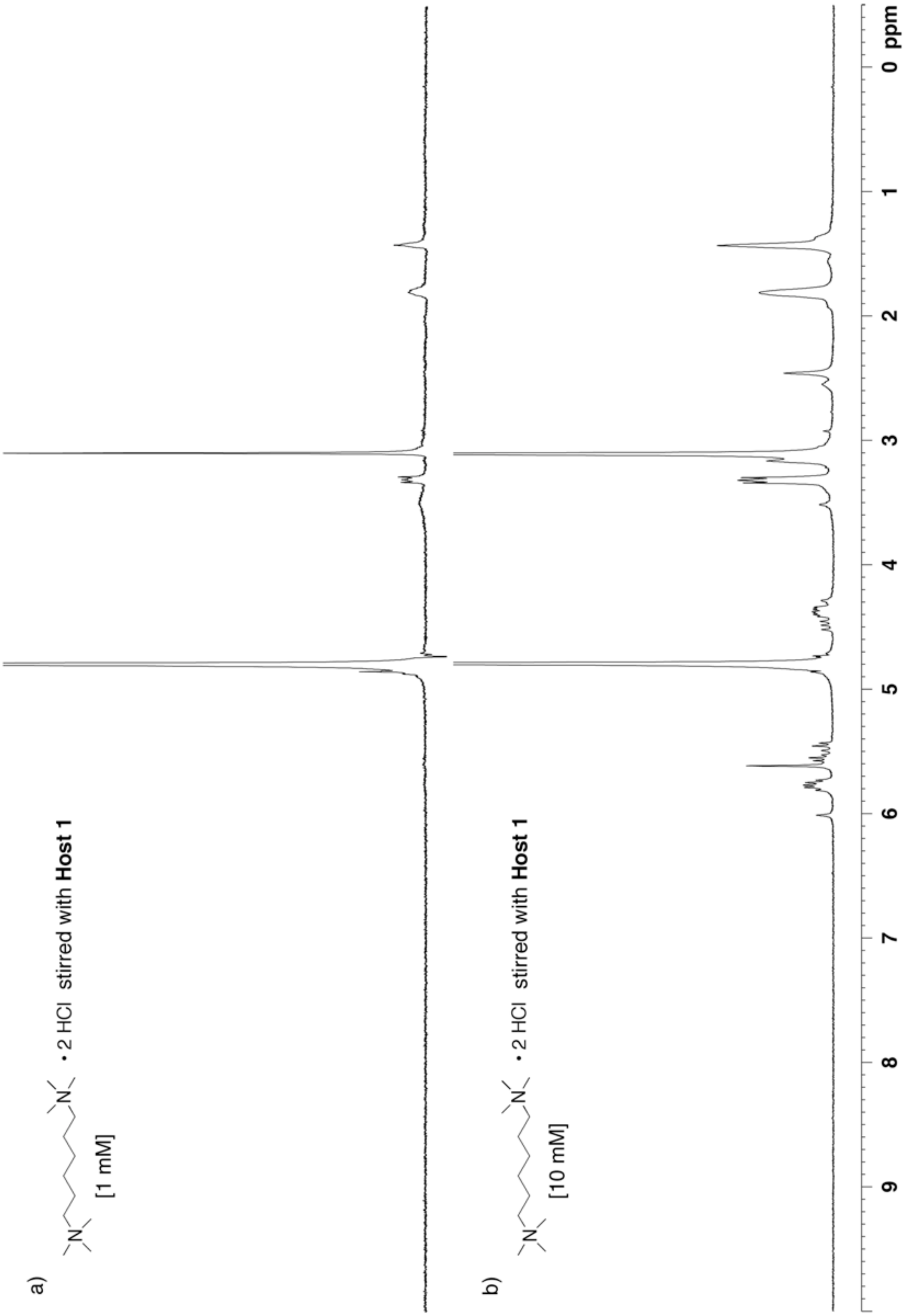


Figure 22. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) a solution obtained by stirring hexamethonium chloride (1 mM) with an excess of solid **1** followed by filtration and b) a solution obtained by stirring hexamethonium chloride (10 mM) with an excess of solid **1** followed by filtration. The fact that the resonances for **1** increase higher concentration of hexamethonium chloride indicate weak binding.

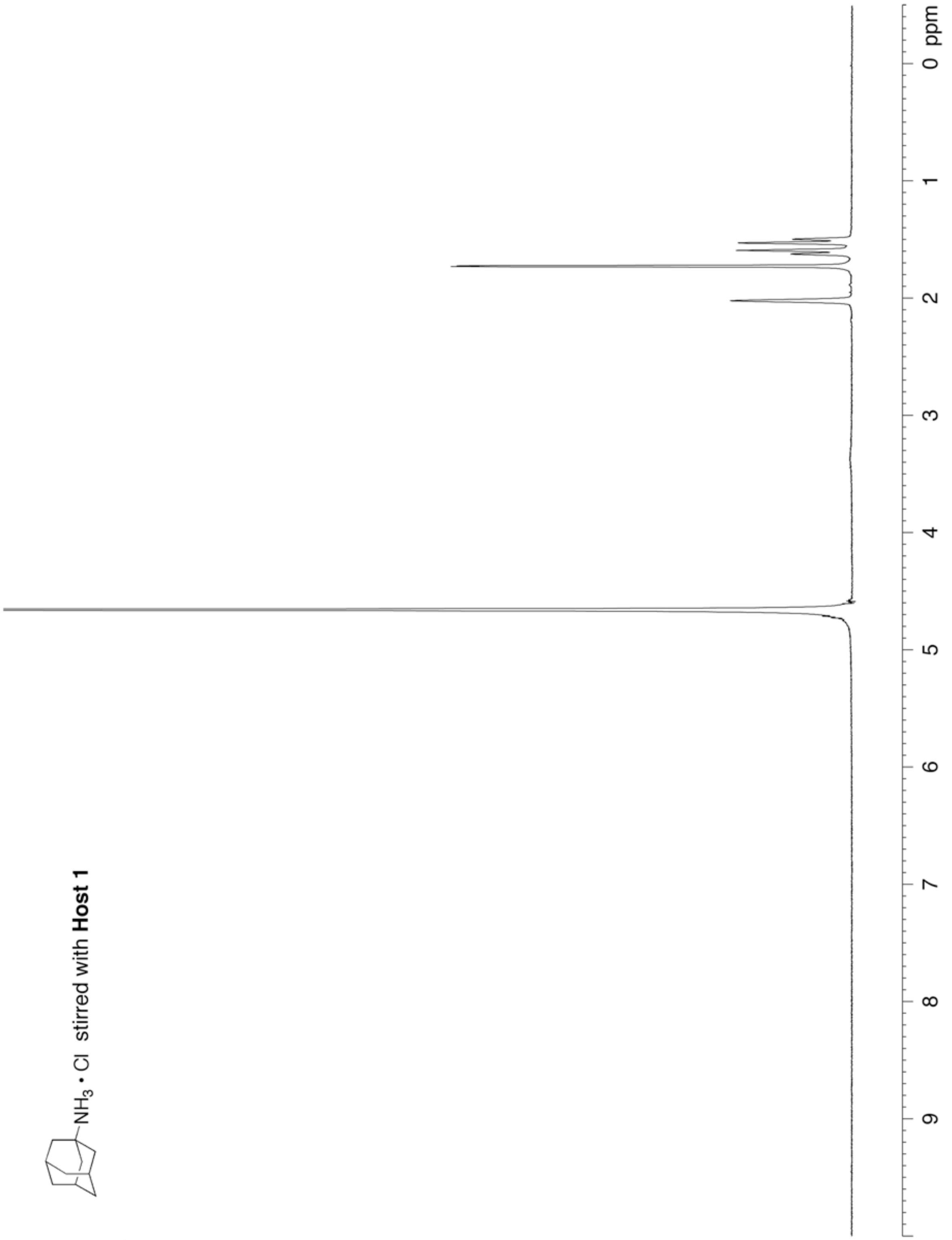


Figure 23. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for a solution obtained by stirring 1-adamantaneamine hydrochloride (10 mM) with an excess of solid **1** followed by filtration. No resonances are seen for **1** and no depletion of the resonances of 1-adamantaneamine hydrochloride is seen, which indicates no complexation has occurred.

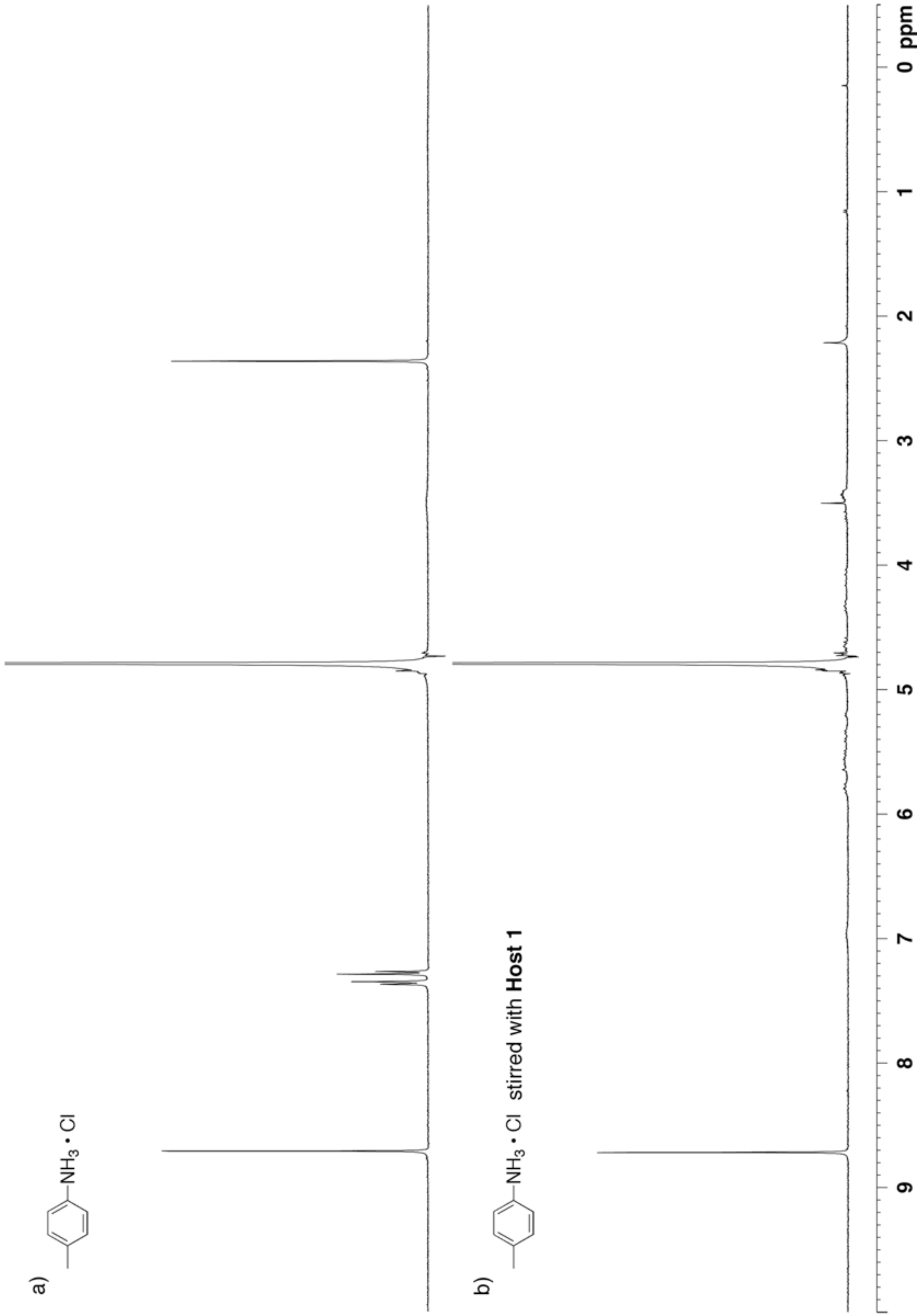


Figure 24. ^{1}H NMR spectrum (400 MHz, D_2O, RT, 1,3,5-tribenzenetricarboxylic acid as internal standard) recorded for: a) *p*-toluidine hydrochloride and b) a solution obtained by stirring *p*-toluidine hydrochloride (10 mM) with an excess of solid **1** followed by filtration. The depletion of the resonances for *p*-toluidine hydrochloride in spectrum b) indicates it forms an insoluble complex with **1**.

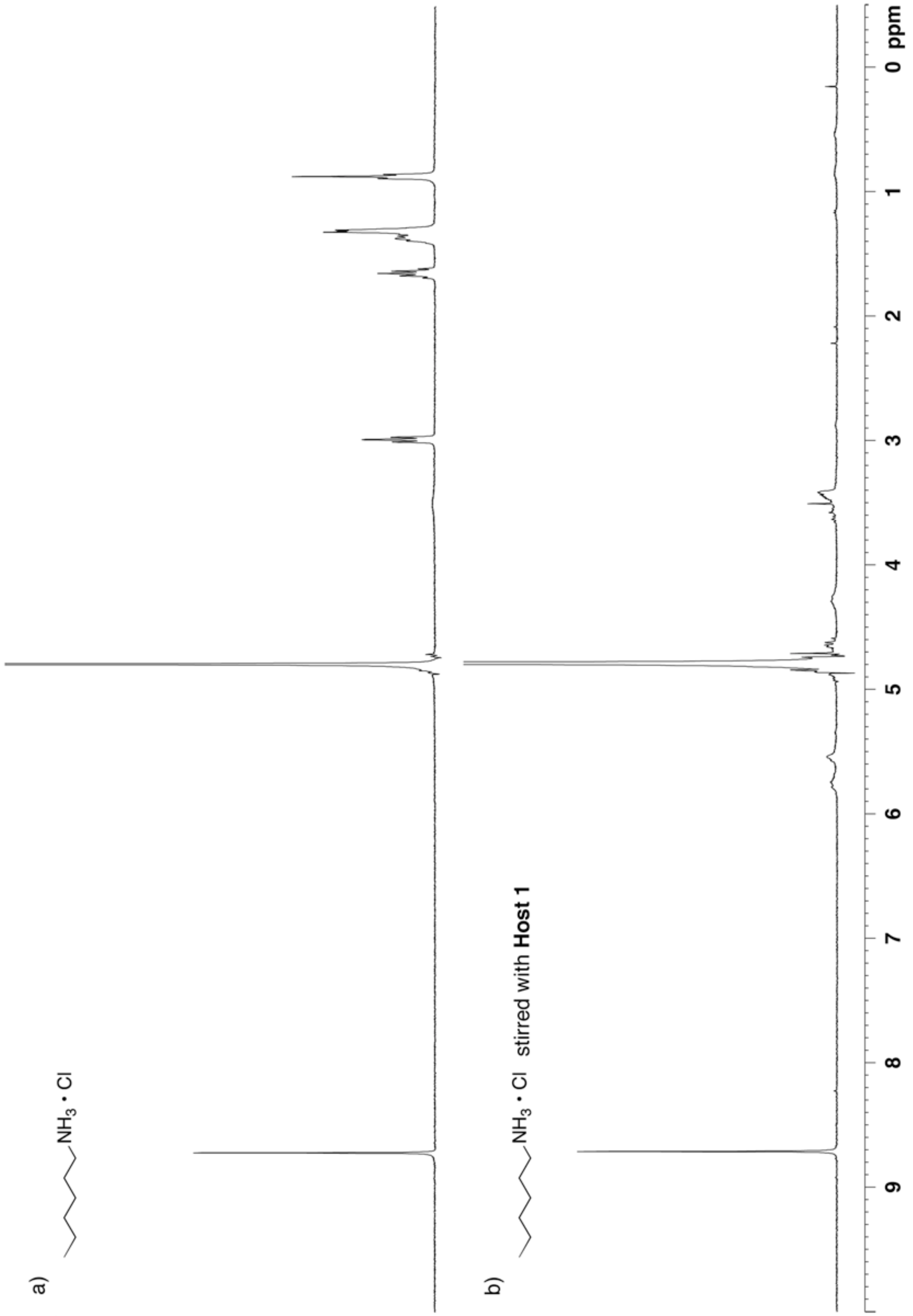


Figure 25. ^{1}H NMR spectrum (400 MHz, D$_2$O, RT, 1,3,5-tribenzenetricarboxylic acid as internal standard) recorded for: a) hexylammonium hydrochloride and b) a solution obtained by stirring hexylammonium hydrochloride (10 mM) with an excess of solid **1** followed by filtration. The depletion of the resonances for hexylammonium chloride in spectrum b) indicates it forms an insoluble complex with **1**.

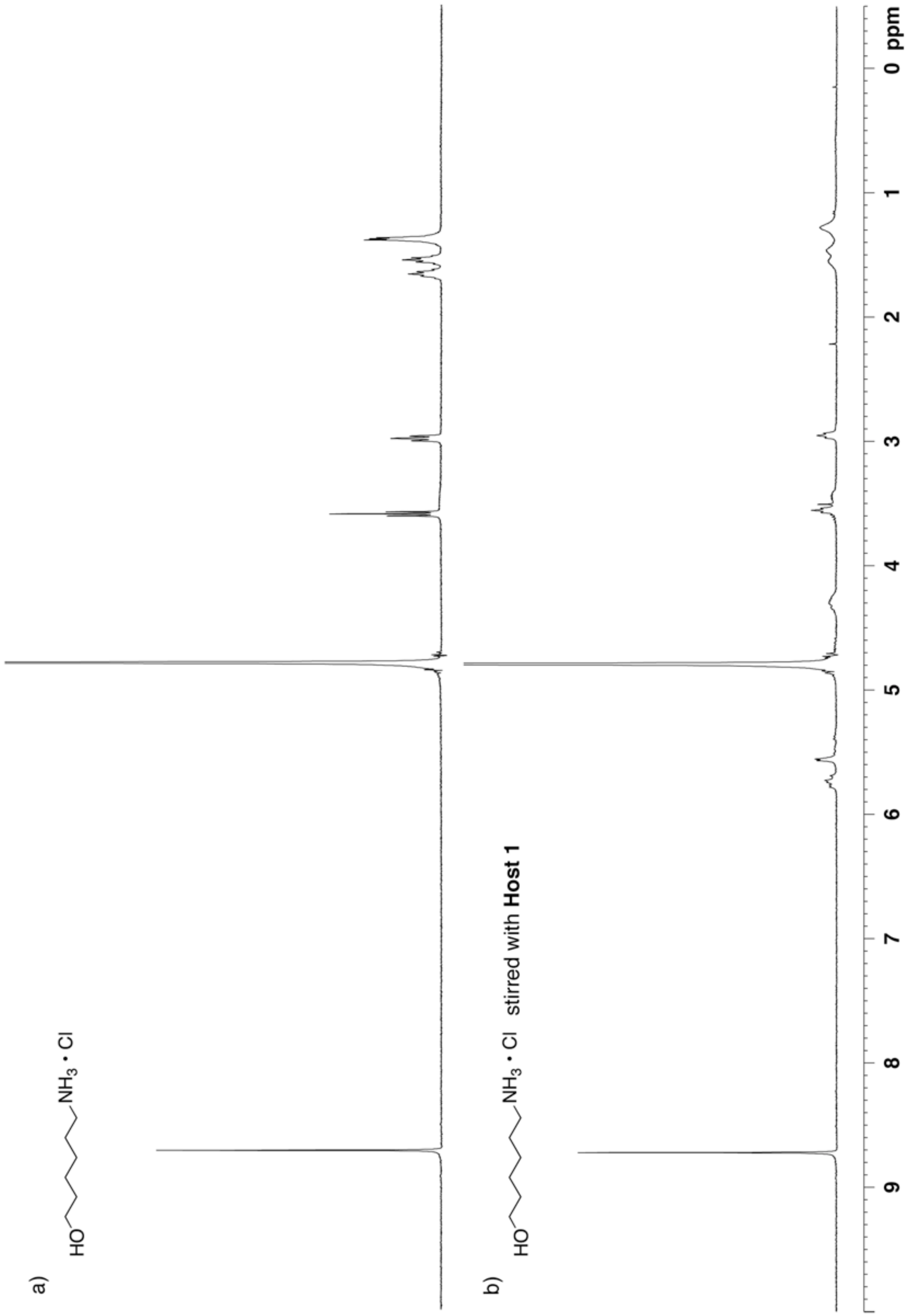


Figure 26. ^{1}H NMR spectrum (400 MHz, D_2O, RT, 1,3,5-tribenzenetricarboxylic acid as internal standard) recorded for: a) aminohexanol hydrochloride and b) a solution obtained by stirring aminohexanol hydrochloride (10 mM) with an excess of solid **1** followed by filtration.

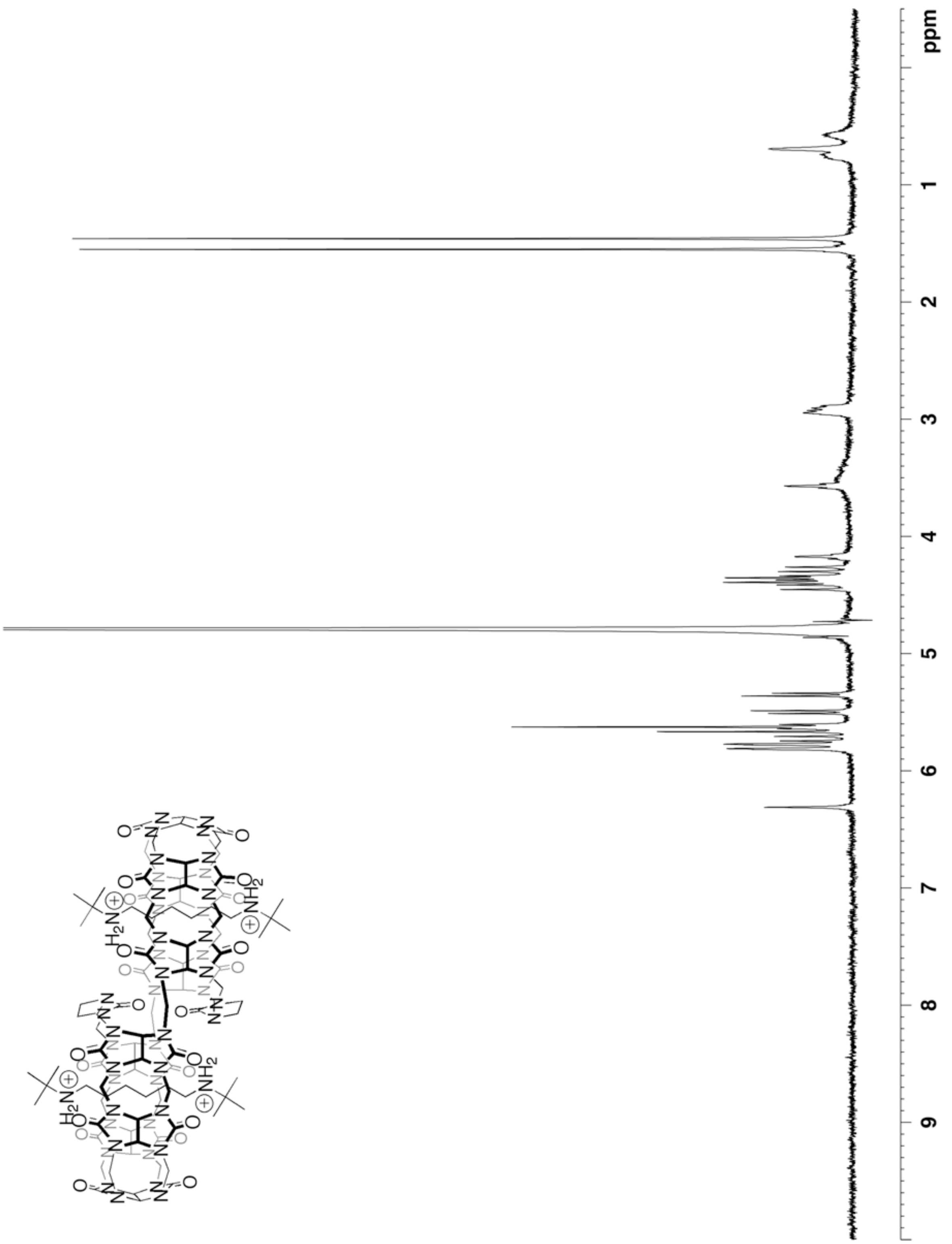


Figure 27. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for **1•4**$_2$ after washing with 0.1 M NaOH in MeOH in an attempt to remove of **4**. This experiment provides evidence that **1•4**$_2$ is a rotaxane because deprotonation does not result in destruction of the complex.

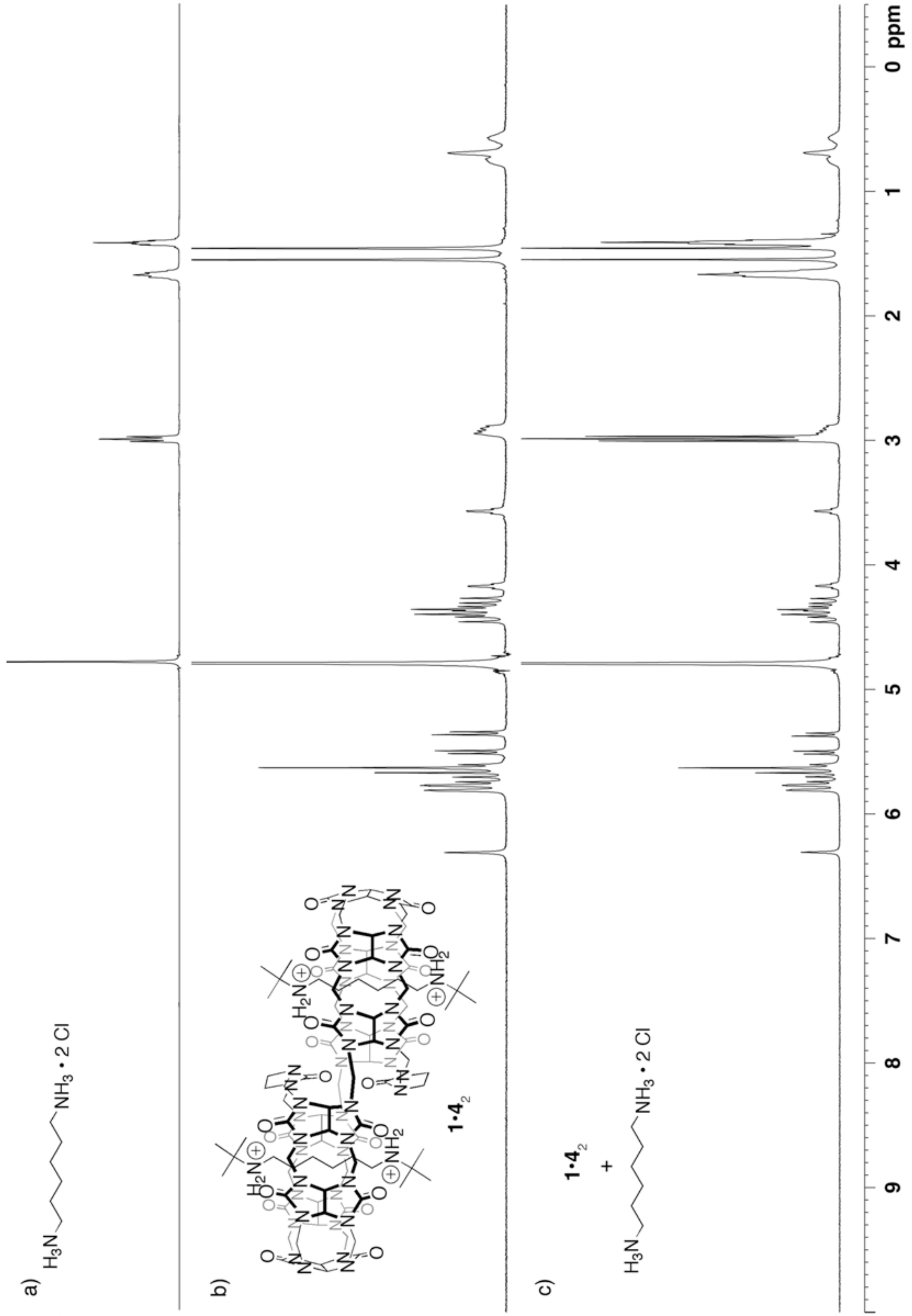


Figure 28. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for: a) 1,6-hexanediammonium dihydrochloride (**5**(n=6)), b) **1•4**$_2$, and c) a solution of **5**(n=6) (20 mM) and **1•4**$_2$ (2 mM). This experiment provides evidence that **1•4**$_2$ is a rotaxane because the addition of **5**(n=6) does not cause the dissociation of **4** from the **1•4**$_2$ complex.

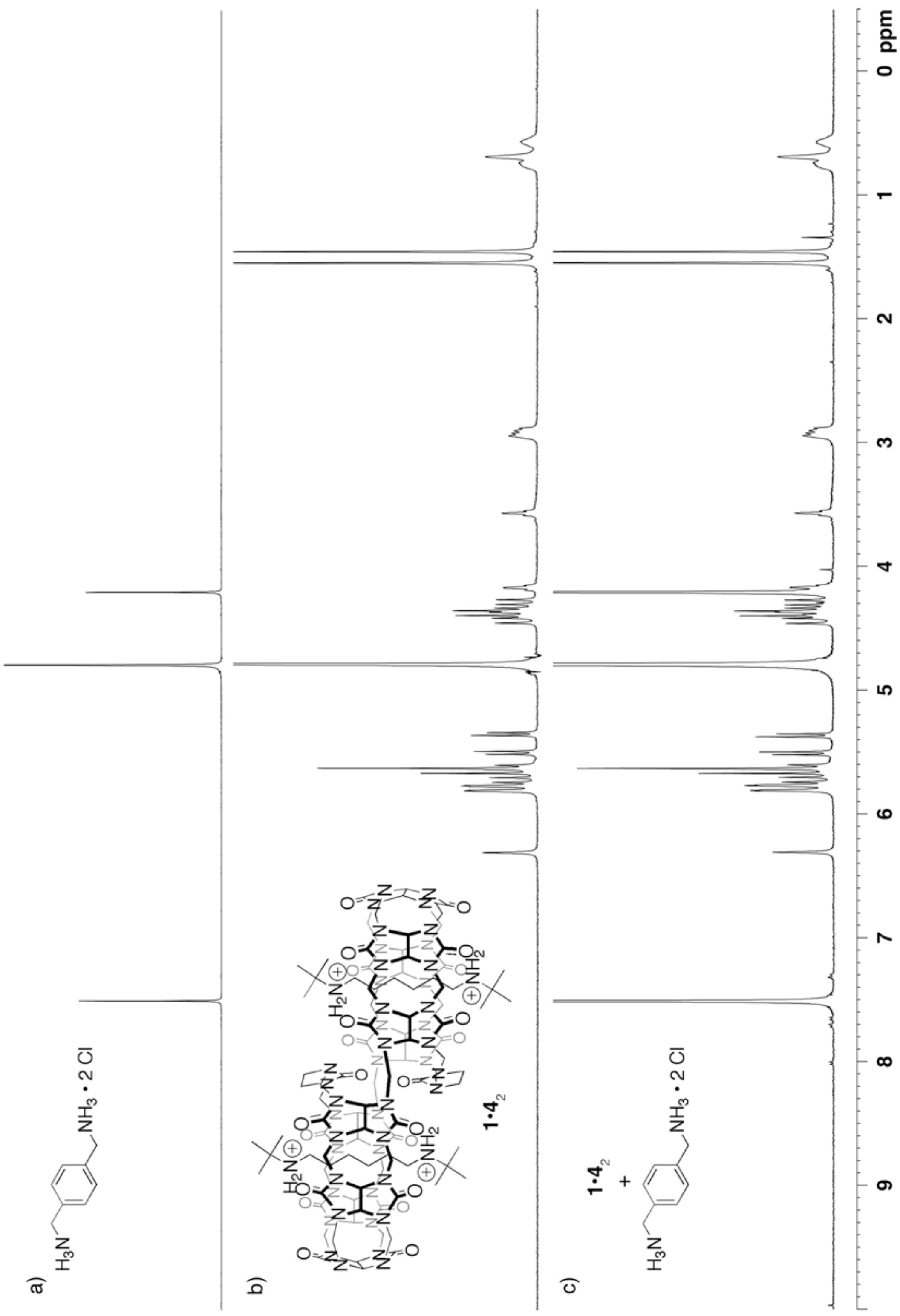


Figure 29. ^{1}H NMR spectrum (400 MHz, D_2O, RT) recorded for: a) *p*-xyxlenediammonium dihydrochloride, b) **1•4**$_2$, and c) a solution of **3** (20 mM) and **1•4**$_2$ (2 mM). This experiment provides evidence that **1•4**$_2$ is a true rotaxane because the addition of **3** does not cause the dissociation of **4** from the **1•4**$_2$ complex.

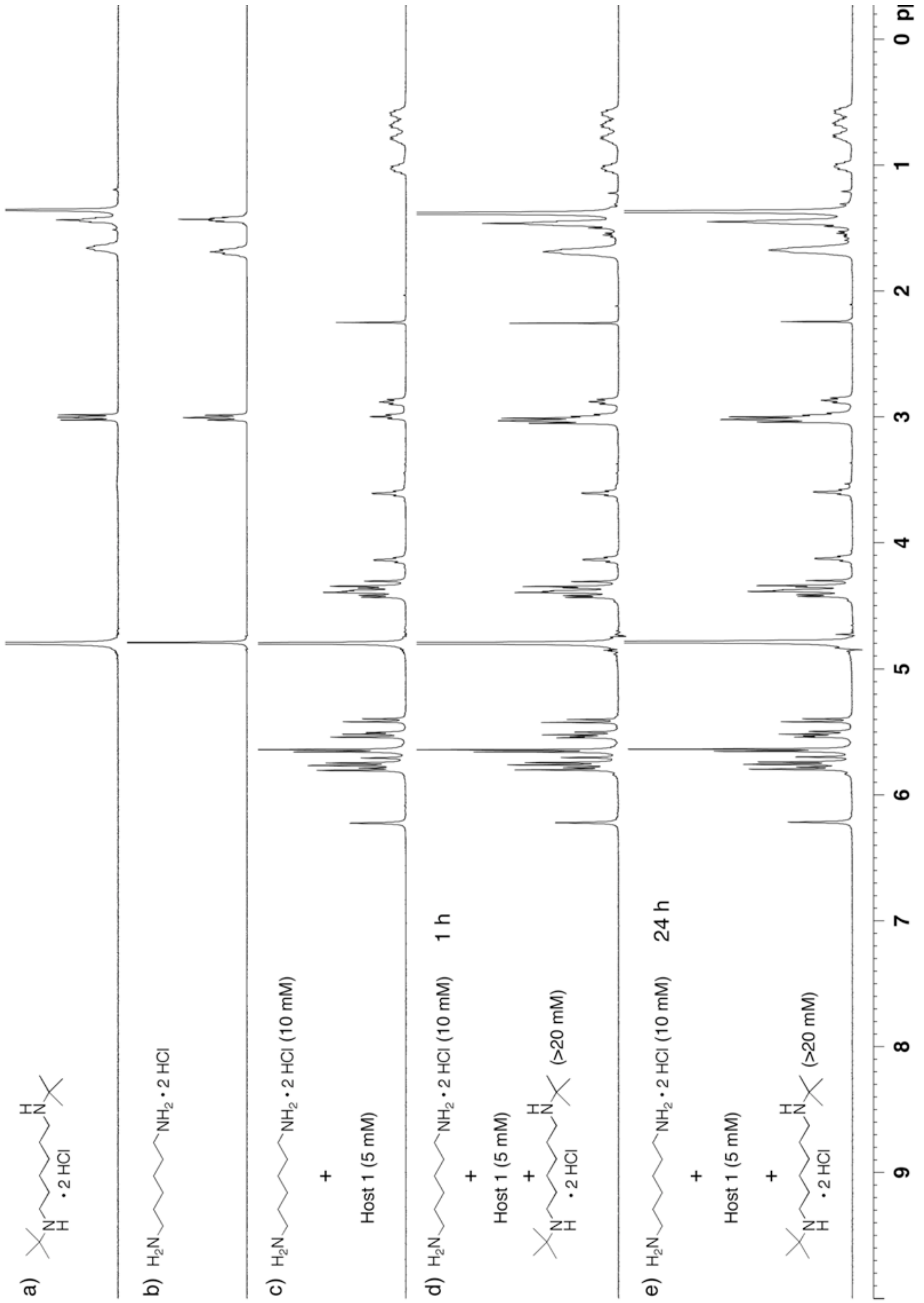


Figure 30. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) **4**, b) **5**(n=6), c) Host **1** (5 mM) stirred with compound **5**(n=6) (10 mM), d) Host **1** (5 mM) stirred with **5**(n=6) (10 mM) and **4** (>20 mM) for 1 h at 50 ºC, and e) Host **1** (5 mM) stirred with **5**(n=6) (10 mM) and **4** (>20 mM) for 24 h at 50 ºC. The observation that **1•4**$_2$ does not form in part e) (50 ˚C, D_2O; conditions where the imidazolidone rings of **1** do not open and close) provides strong evidence that under the reaction conditions used to synthesize rotaxane **1•4**$_2$ (50 ˚C, 8 M HCl, 1 h), that the reaction proceeds by a clipping reaction of the bis-ns-CB[10]•**4**$_2$ complex as shown in Scheme 1.

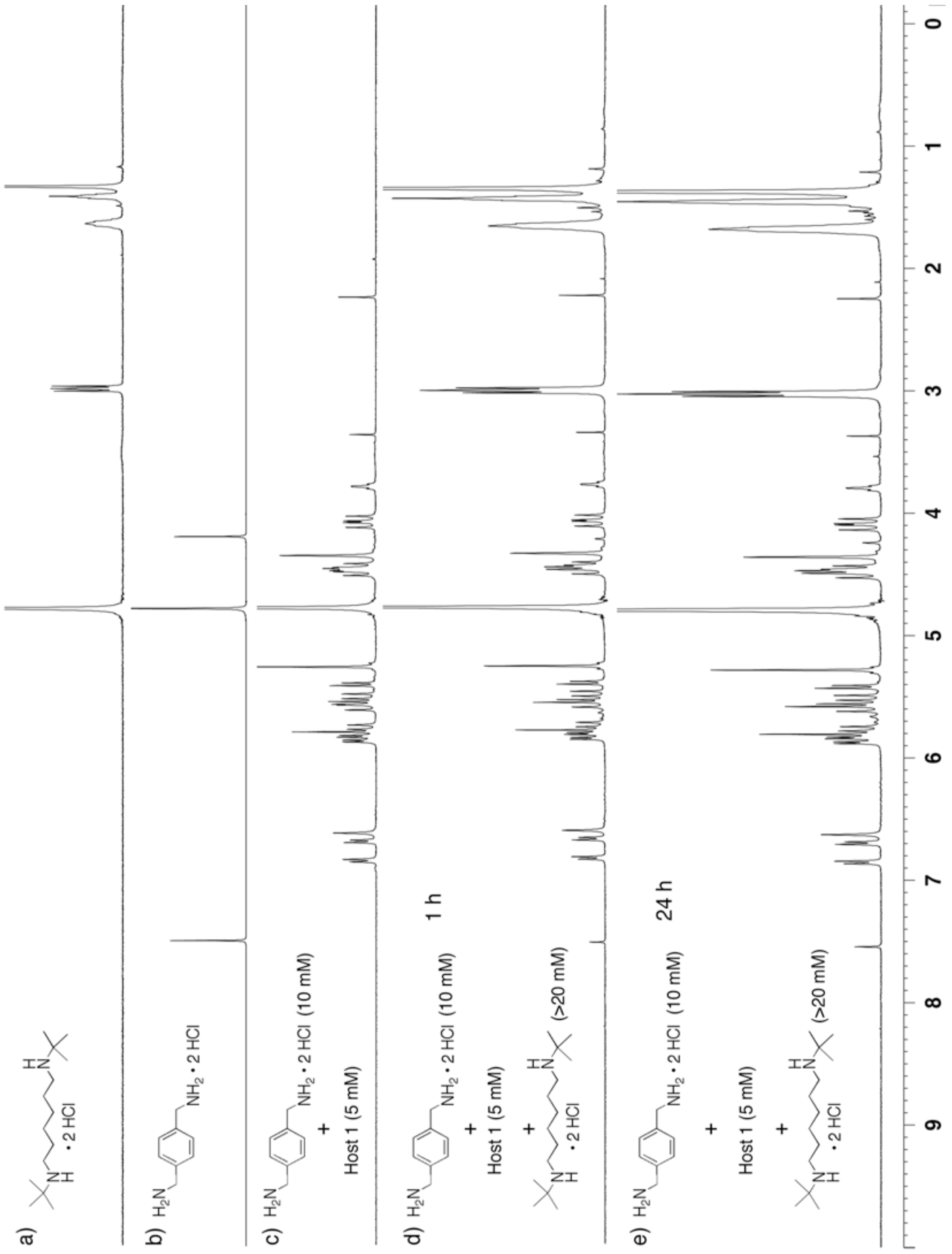
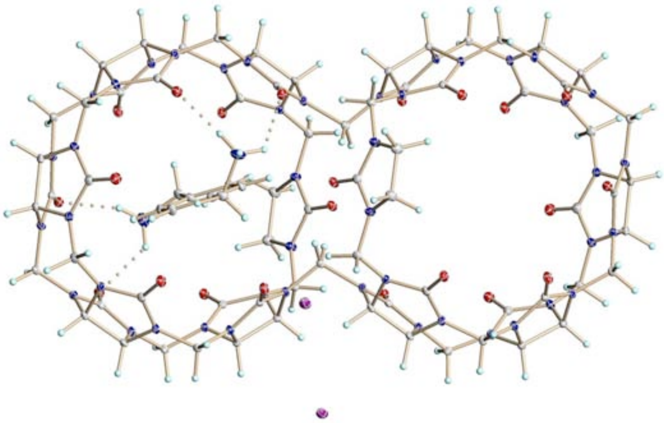


Figure 31. ^{1}H NMR spectra (400 MHz, D_2O, RT) recorded for: a) compound **4**, b) compound **3**, c) Preformed host **1** (5 mM) stirred with compound **3** (10 mM), d) preformed host **1** (5 mM) stirred with compound **3** (10 mM) and compound **4** (>20 mM) for 1 h at 50 ºC, and e) preformed host **1** (5 mM) stirred with compound **3** (10 mM) and compound **4** (>20 mM) for 24 h at 50 ºC. The observation that **1•4**$_2$ does not form in part e) (50 ˚C, D_2O; conditions where the imidazolidone rings of **1** do not open and close) provides strong evidence that under the reaction conditions used to synthesize rotaxane **1•4**$_2$ (50 ˚C, 8 M HCl, 1 h), that the reaction proceeds by a clipping reaction of the bis-ns-CB[10]•**4**$_2$ complex as shown in Scheme 1.



Details of the crystal structure of 1. A colorless prism of $[(C_{68}H_{72}N_{44}O_{22})<(C_8H_{14}N_2)_2]I_4\cdot 18H_2O$, approximate dimensions 0.21×0.24×0.37 mm^3, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 250(2) K on a three-circle diffractometer system equipped with Bruker Smart Apex II CCD area detector using a graphite monochromator and a MoKα fine-focus sealed tube (λ= 0.71073 Å) . The detector was placed at a distance of 6.000 cm from the crystal.

A total of 1330 frames were collected with a scan width of -0.30° an exposure time of 20 sec/frame using Apex2 (Bruker, 2005). The total data collection time was 10 hours. The frames were integrated with Apex2 software package using a narrow-frame integration algorithm. The integration of the data using a Monoclinic unit cell yielded a total of 40898 reflections to a maximum θ angle of 27.50°, of which 13130 were independent (completeness = 99.5%, R_{int} = 3.09%, R_{sig} = 3.53%) and 10224 were greater than 2σ(I). The final cell dimensions of a = 13.690(2) Å, b = 13.972(2) Å, c = 30.064(4) Å, α = 90°, β = 93.1108(19)°, γ = 90°, V = 5742.3(15) Å^3, are based upon the refinement of the XYZ-centroids of 15779 reflections with 2.2 < θ < 28.2° using Apex2 software. Analysis of the data showed 0 % decay during data collection. Data were corrected for absorption effects with the Semi-empirical from equivalents method using SADABS (Sheldrick, 1996). The minimum and maximum transmission coefficients were 0.674 and 0.779.

The structure was solved and refined using the SHELXS-97 (Sheldrick, 1990) and SHELXL-97 (Sheldrick, 1997) software in the space group $P2_1/n$ with Z = 2 for the formula unit $[(C_{68}H_{72}N_{44}O_{22})<(C_8H_{14}N_2)_2]I_4\cdot 18H_2O$. The final anisotropic full-matrix least-squares refinement on F^2 with 862 variables converged at R_1 = 4.60 % for the observed data and wR_2 = 9.94 % for all data. The goodness-of-fit was 1.000. The largest peak on the final difference map was 1.323 $\bar{e}$/Å^3 and the largest hole was -1.156 $\bar{e}$/Å^3. On the basis of the final model, the calculated density was 1.715 g/cm^3 and F(000), 3016 $\bar{e}$.

Comments:

- Data quality: very good
- Disorder: 1 or 9 water molecules is disordered in two positions (O9w and O10w); another molecule (O6w) has one H atom disordered in two alternative positions
- H-atoms: constrained geometry as riding on attached atom (A) for C-H and N-H and soft restrains on distances & angle to attached atom (A) for H2O; $U_{iso}(H)=1.5U_{iso}(A)$ for CH_3 and H2O and $1.2U_{iso}(A)$ for other groups
- Residual density: near heavy atoms

Disorder-Order phase transition observed during the experiment:

At first crystal structure was indexed in a monoclinic system, sp.gr. $P2_1/n$, a = 14.901(9), b = 14.231(8), c = 29.591(18) Å, β = 96.712(9)°, V = 6232(11) Å^3. The structure determination revealed the same structure as reported one. However all the water molecules except one and one of two iodine ions were disordered as well as one ammonium group in the guest molecule. Analyses of the frames showed that crystal was changing during the data collection. Therefore after experiment was finished a new data collection was undertaken. It revealed that even so symmetry is unchanged but cell dimensions are noticeable different a = 13.690(2), b = 13.972(2), c = 30.064(4), β = 93.1108(19)°, V = 5742.3(15) Å^3. The crystal structure determination showed the same composition and type of structure including guest-host system. However guest molecule, both iodine ions, and most of the water molecules are fully ordered.

Thus the title compound undergoes disorder to order phase transition at 250 K which takes about 10 hours for completion.

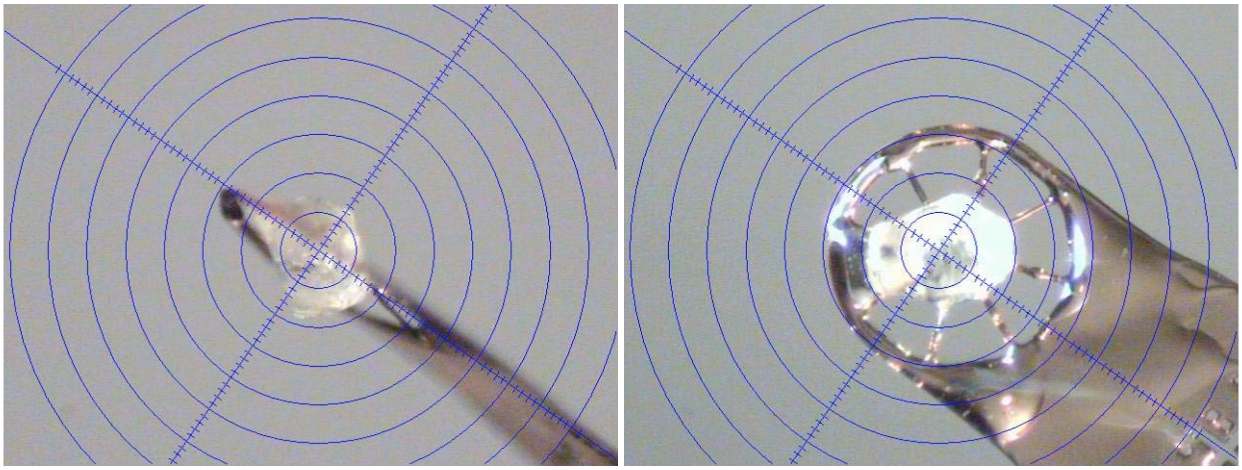


Table S1. Crystal data and structure refinement for UM#1946.

X-ray lab book No.	1946	
Crystal ID	Isaacs/Wittenberg JW-3-138 @250K	
Empirical formula	$[(C_{68}H_{72}N_{44}O_{22})<(C_8H_{14}N_2)_2]I_4 \cdot 18H_2O$	
Formula weight	2966.01	
Temperature	250(2) K	
Wavelength	0.71073 Å	
Crystal size	0.37×0.24×0.21 mm^3	
Crystal habit	colorless prism	
Crystal system	Monoclinic	
Space group	P21/n	
Unit cell dimensions	a = 13.690(2) Å	α = 90°
	b = 13.972(2) Å	β = 93.1108(19)°
	c = 30.064(4) Å	γ = 90°
Volume	5742.3(15) $Å^3$	
Z	2	
Density, ρ_{calc}	1.715 g/cm^3	
Absorption coefficient, μ	1.192 mm^{-1}	
F(000)	3016 ē	
Diffractometer	Bruker Smart Apex II CCD area detector	
Radiation source	fine-focus sealed tube, MoKα	
Detector distance	6.000 cm	
Data collection method	ω and φ scans	
Total frames	1330	
Frame size	512 pixels	
Frame width	-0.30°	
Exposure per frame	20 sec	
Total measurement time	10 hours	
θ range for data collection	2 to 27.50°	
Index ranges	$-17 \le h \le 17, -17 \le k \le 18, -39 \le l \le 38$	
Reflections collected	40898	
Independent reflections	13130	
Observed reflection, I>2σ(I)	10224	
Coverage of independent reflections	99.5 %	
Variation in check reflections	0 %	
Absorption correction	Semi-empirical from equivalents SADABS (Sheldrick, 1996)	
Max. and min. transmission	0.779 and 0.674	
Structure solution technique	direct	
Structure solution program	SHELXS-97 (Sheldrick, 1990)	
Refinement technique	Full-matrix least-squares on F^2	
Refinement program	SHELXL-97 (Sheldrick, 1997)	
Function minimized	$\Sigma w(F_o^2 - F_c^2)^2$	
Data / restraints / parameters	13130 / 48 / 862	
Goodness-of-fit on F^2	1.260	
Δ/σ_{max}	0.001	
Final R indices: R_1, I>2σ(I)	0.0460	
wR_2, all data	0.0994	
R_{int}	0.0309	
R_{sig}	0.0353	
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 24.22P]$, $P = [\max(F_o^2, 0) + 2F_o^2]/3$	
Largest diff. peak and hole	1.323 and -1.156 ē/$Å^3$	

$R_1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR_2 = [\Sigma w(F_o^2-F_c^2)^2/\Sigma w(F_o^2)^2]^{1/2}$