
Organic & Biomolecular Chemistry

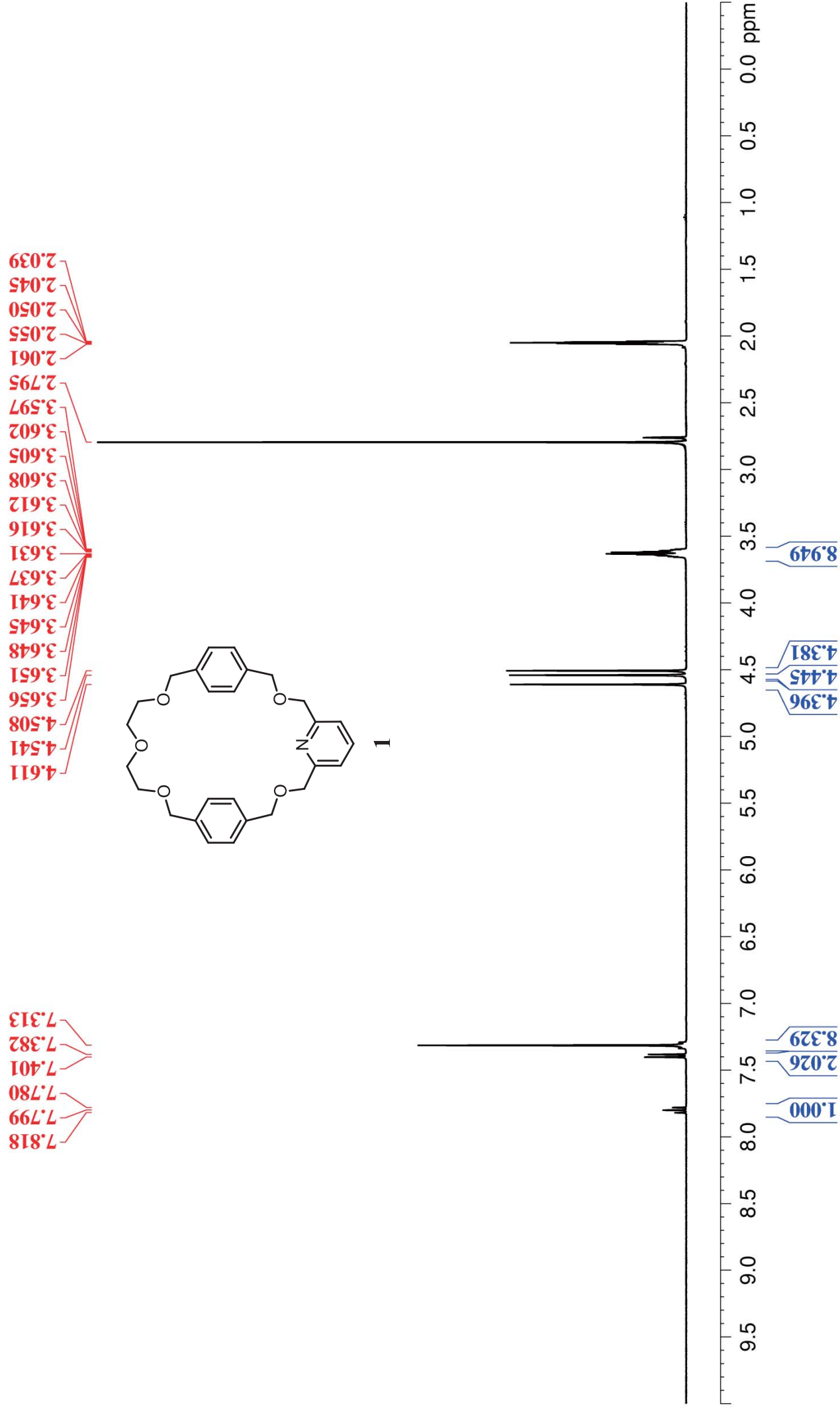
Five Additional Macrocycles that Allow the Na⁺ Ion-Templated Threading of Guest Units Featuring a Single Urea or Amide Functionality

You-Han Lin, Chien-Chen Lai, and Sheng-Hsien Chiu*

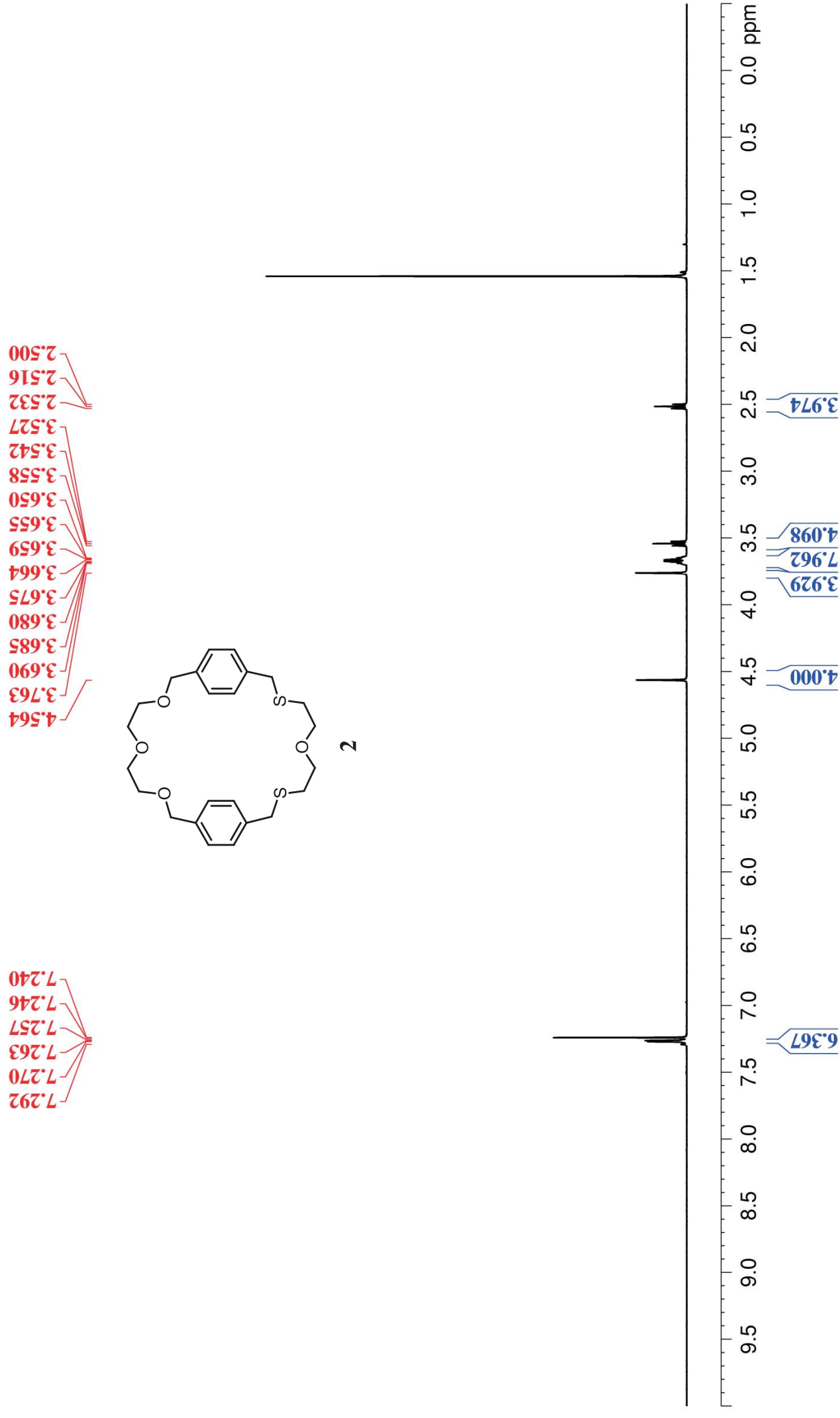
Supplementary Material

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¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **1**

^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **2**

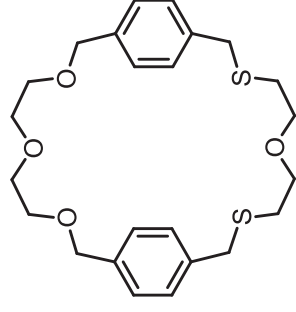


^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **2**

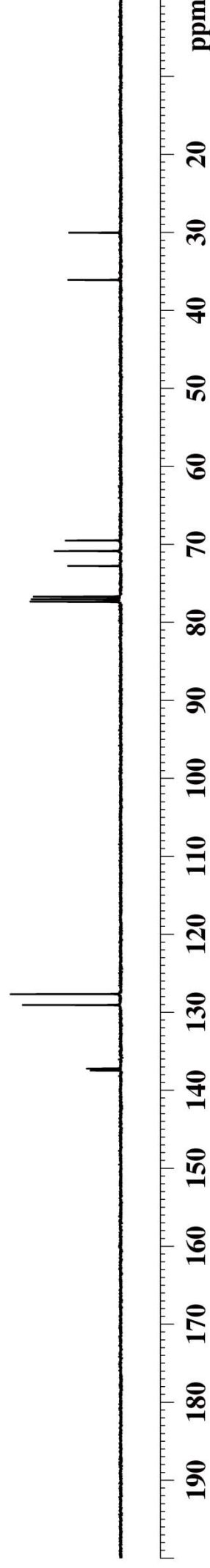
137.444
137.183
129.043
127.635

77.317
77.000
76.683
72.740
70.844
70.828
69.474

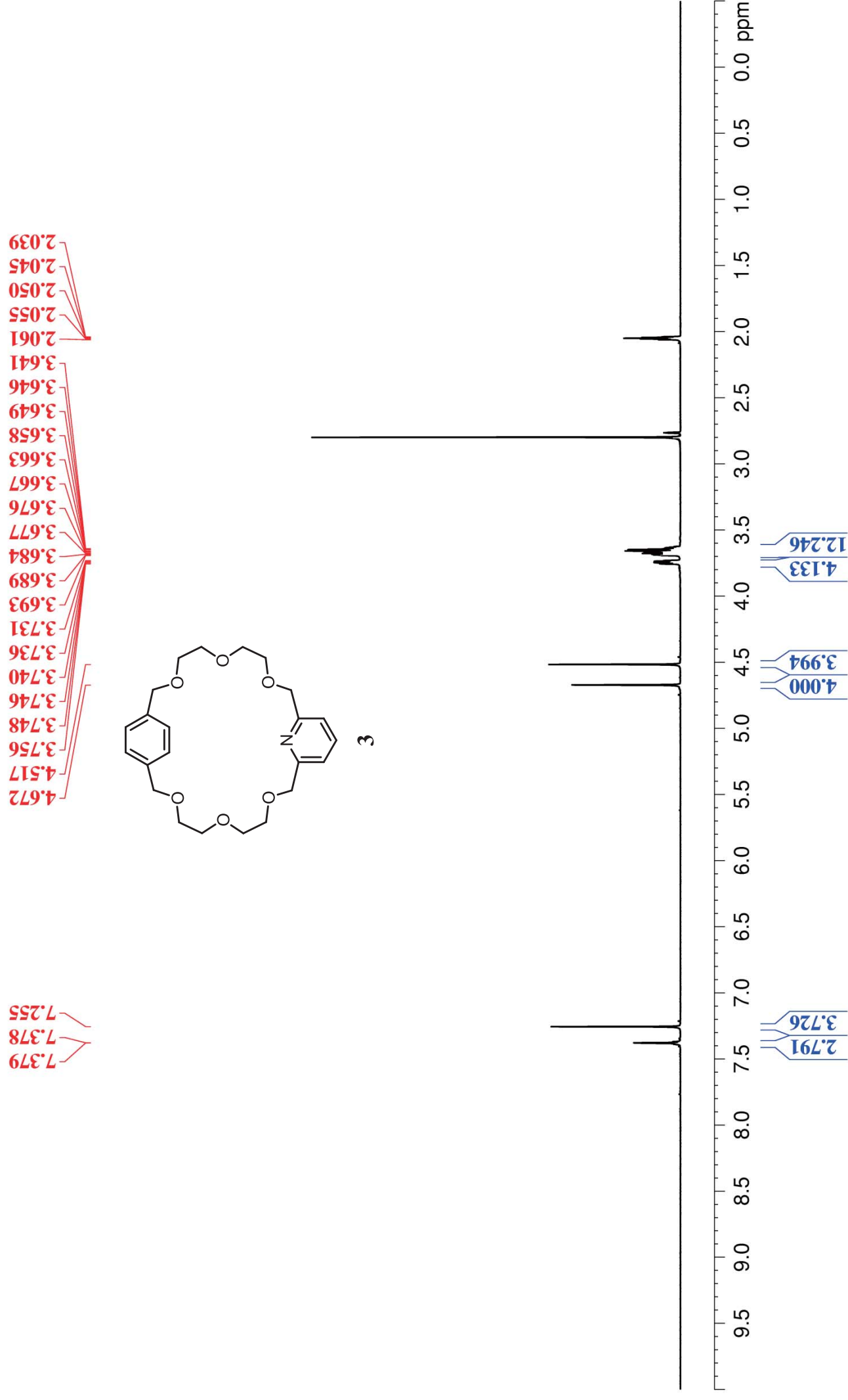
36.068
30.011



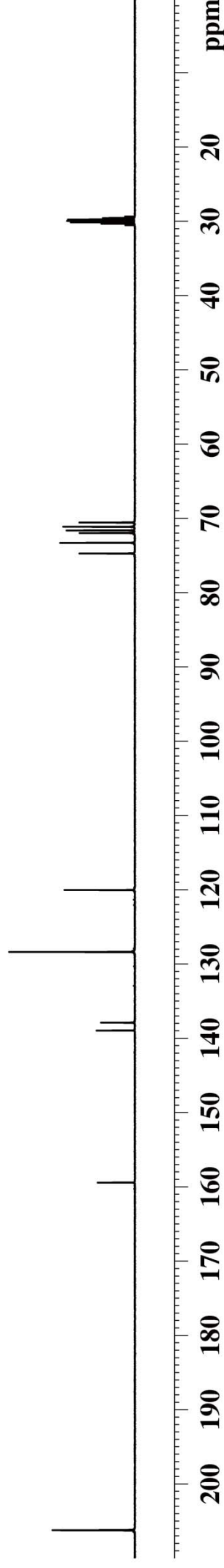
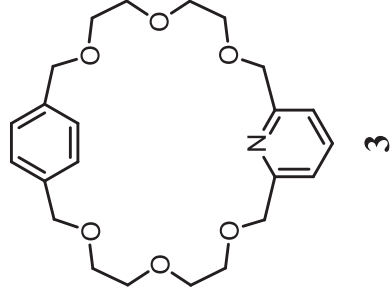
2



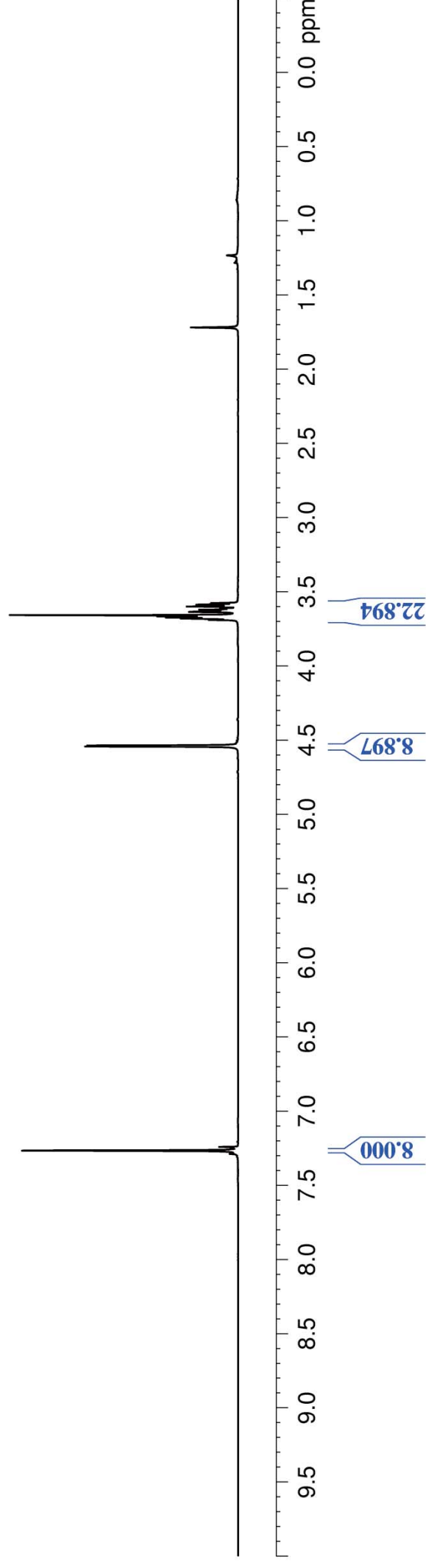
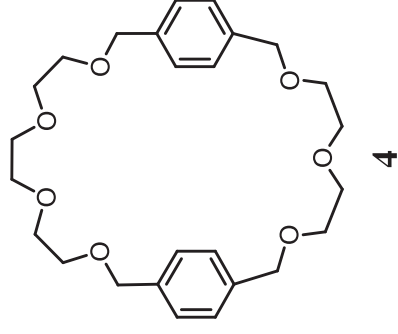
^1H NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **3**



^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **3**



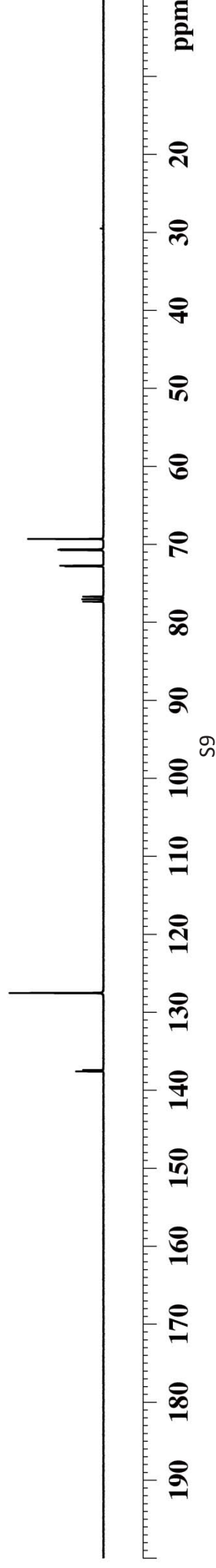
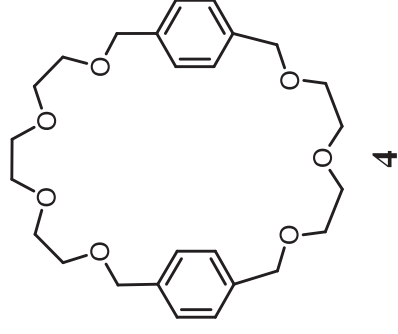
^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **4**



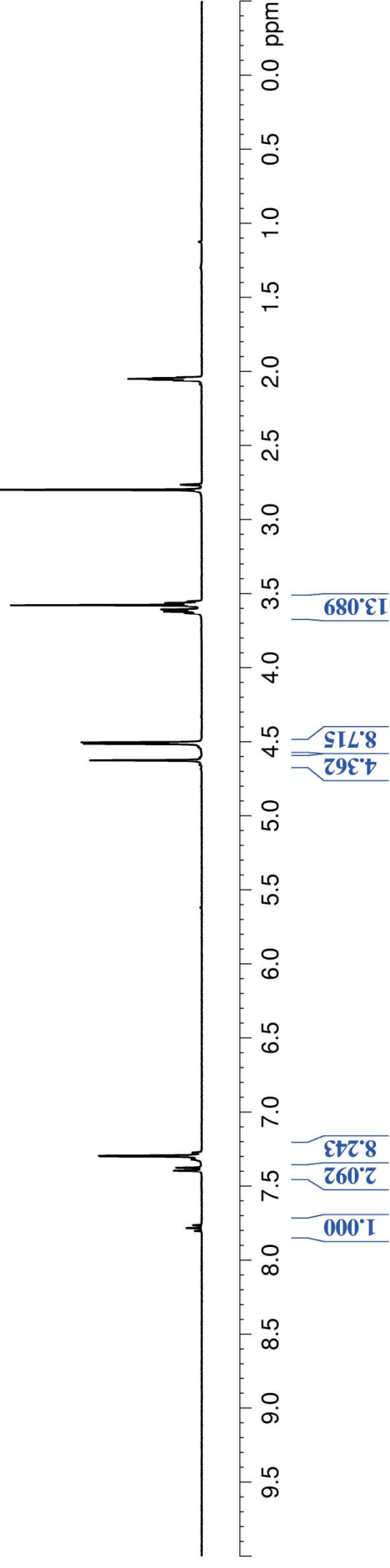
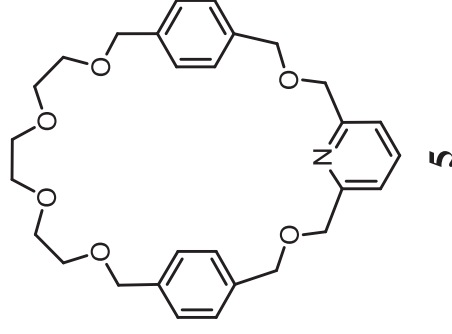
^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **4**

137.566
137.389
127.518
127.490

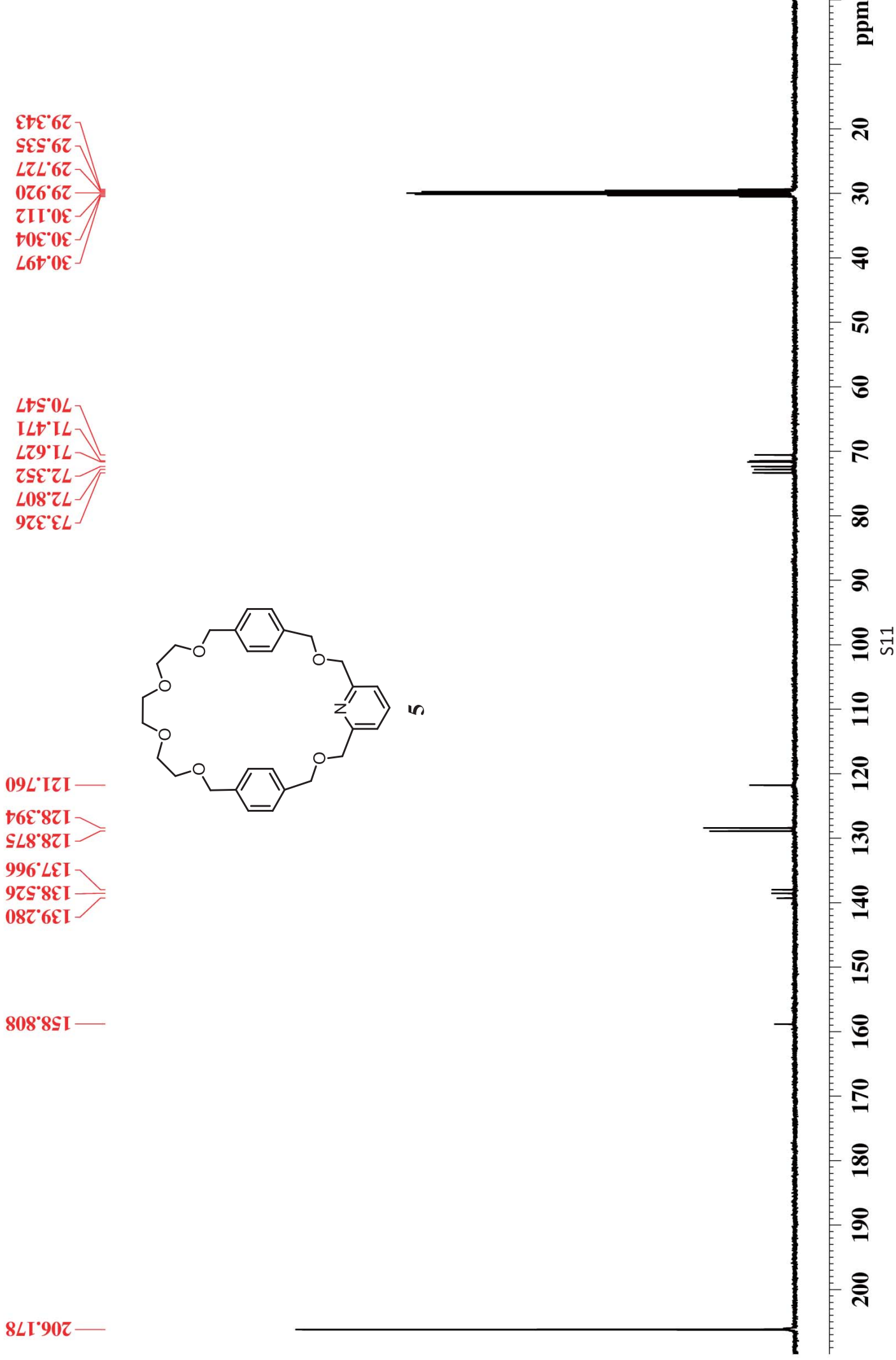
77.319
77.000
76.681
72.797
72.701
70.727
70.650
70.617
69.288



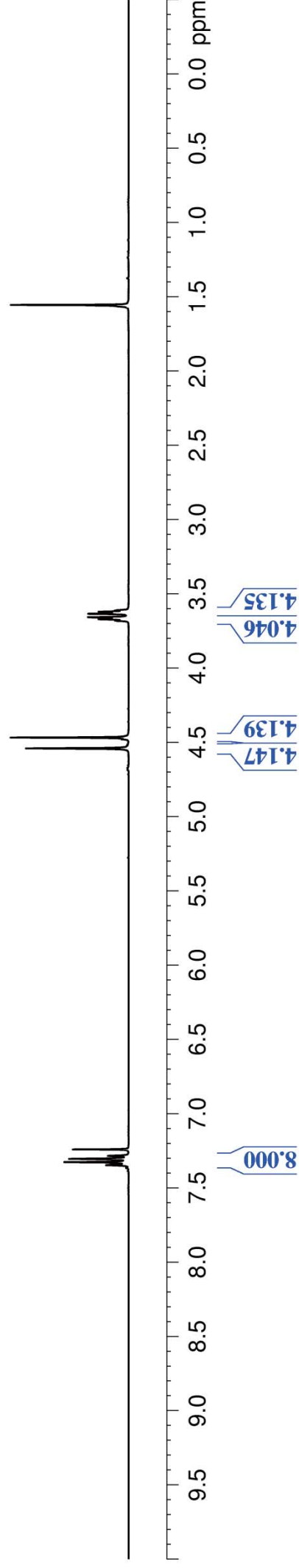
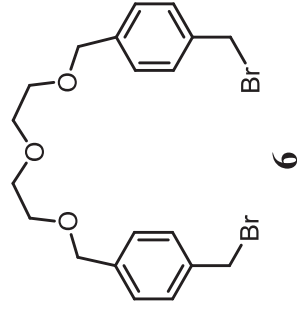
^1H NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **5**



^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **5**



^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **6**

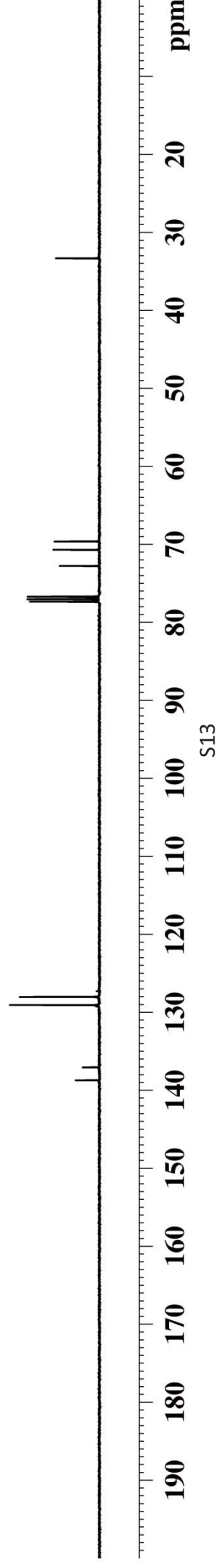
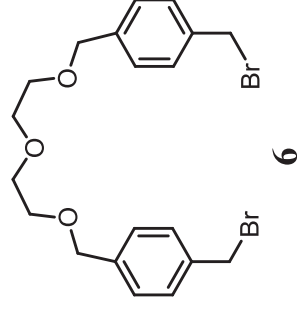


^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **6**

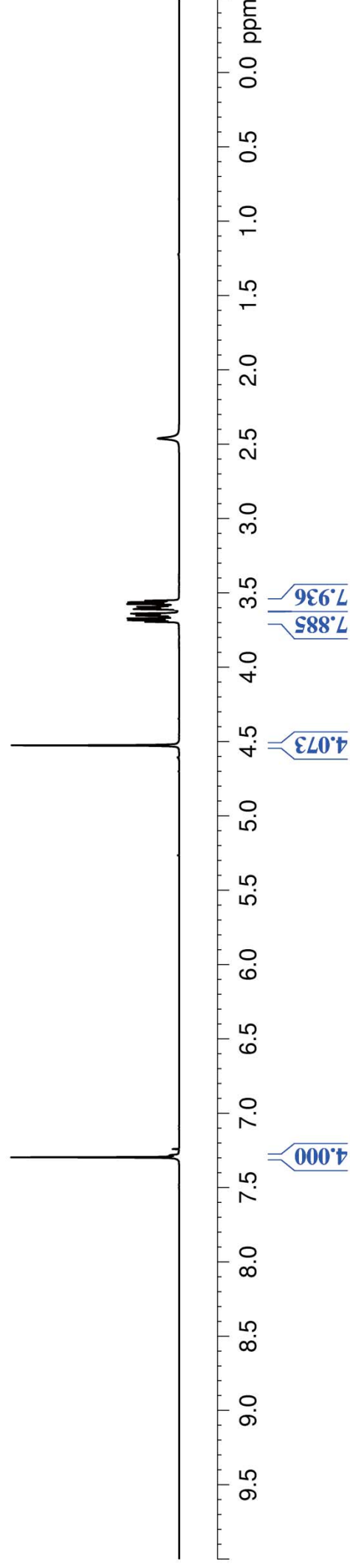
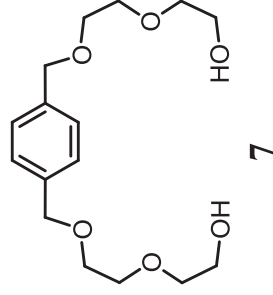
138.687
137.037
129.054
128.001

77.318
77.000
76.682
72.746
70.677
69.600

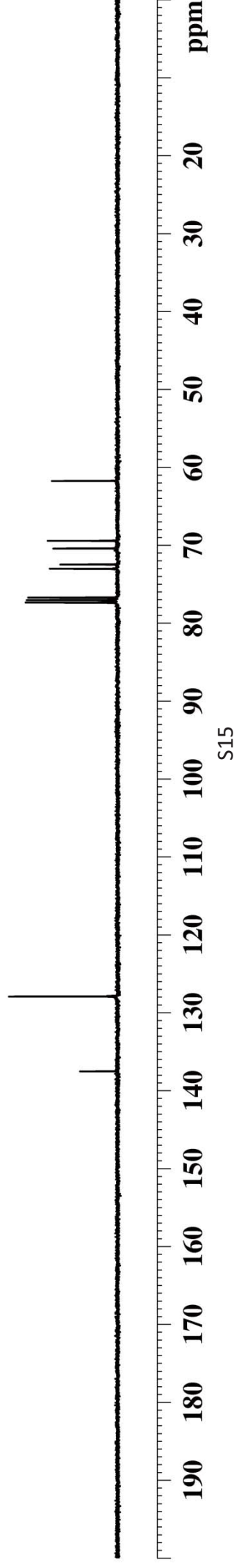
33.308

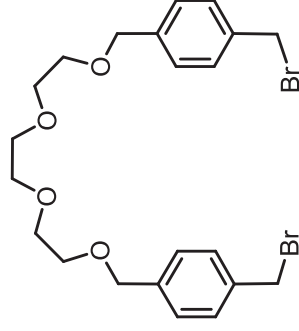
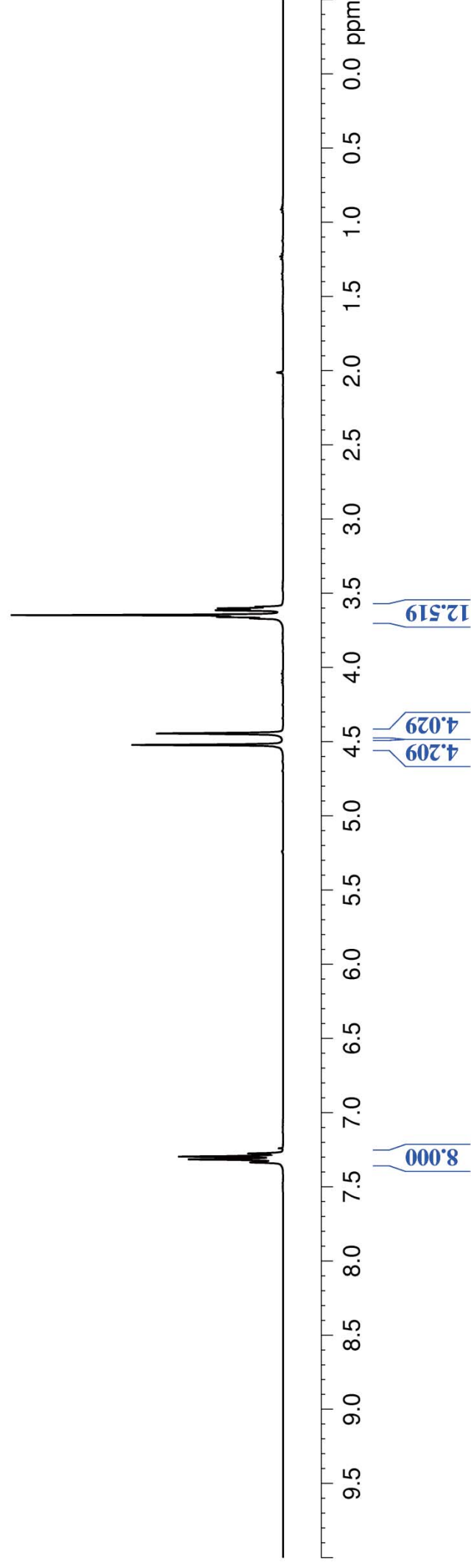


^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **7**

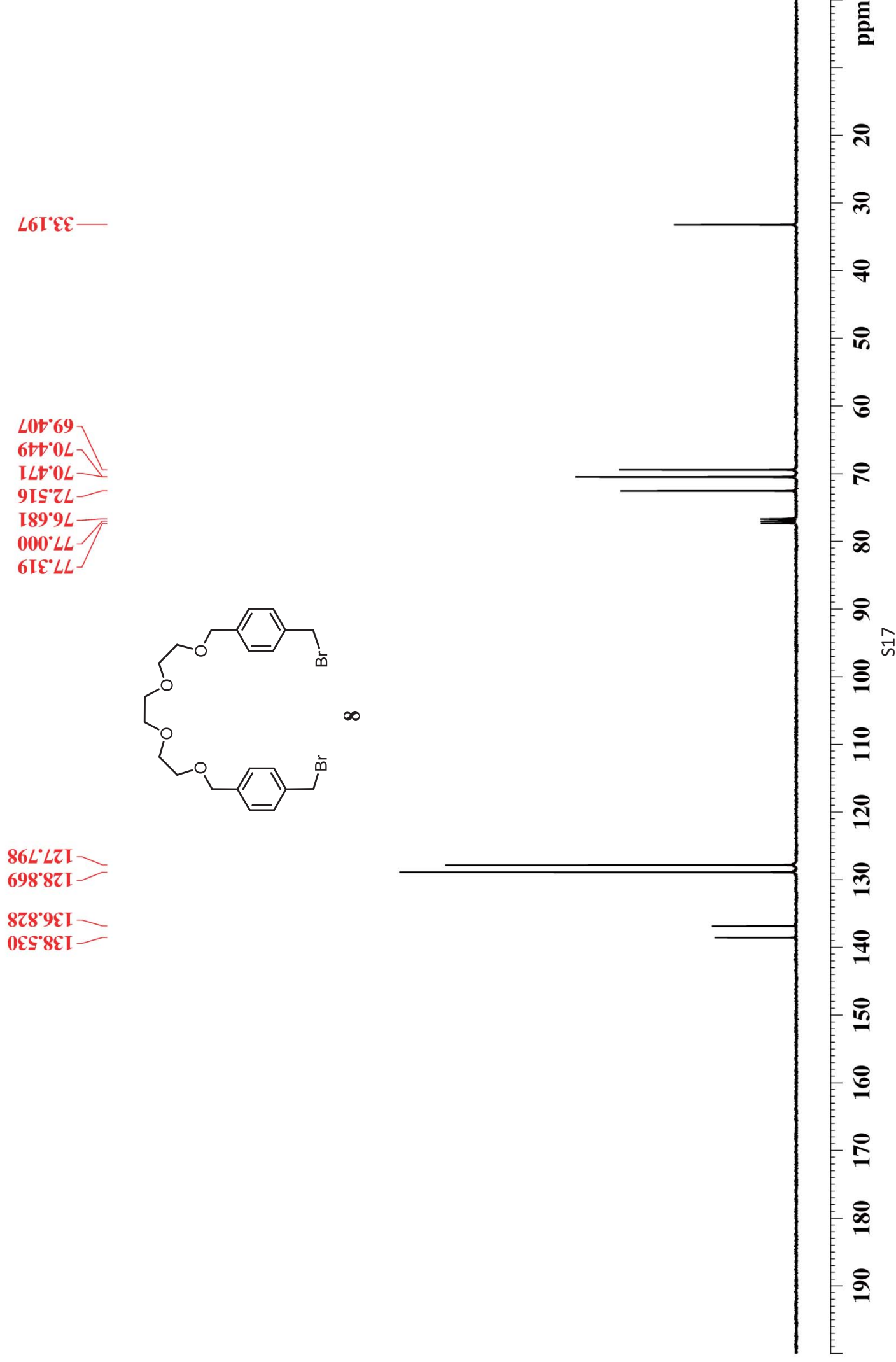
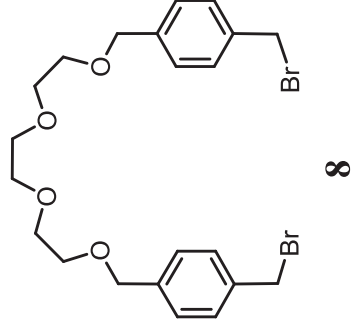


^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **7**

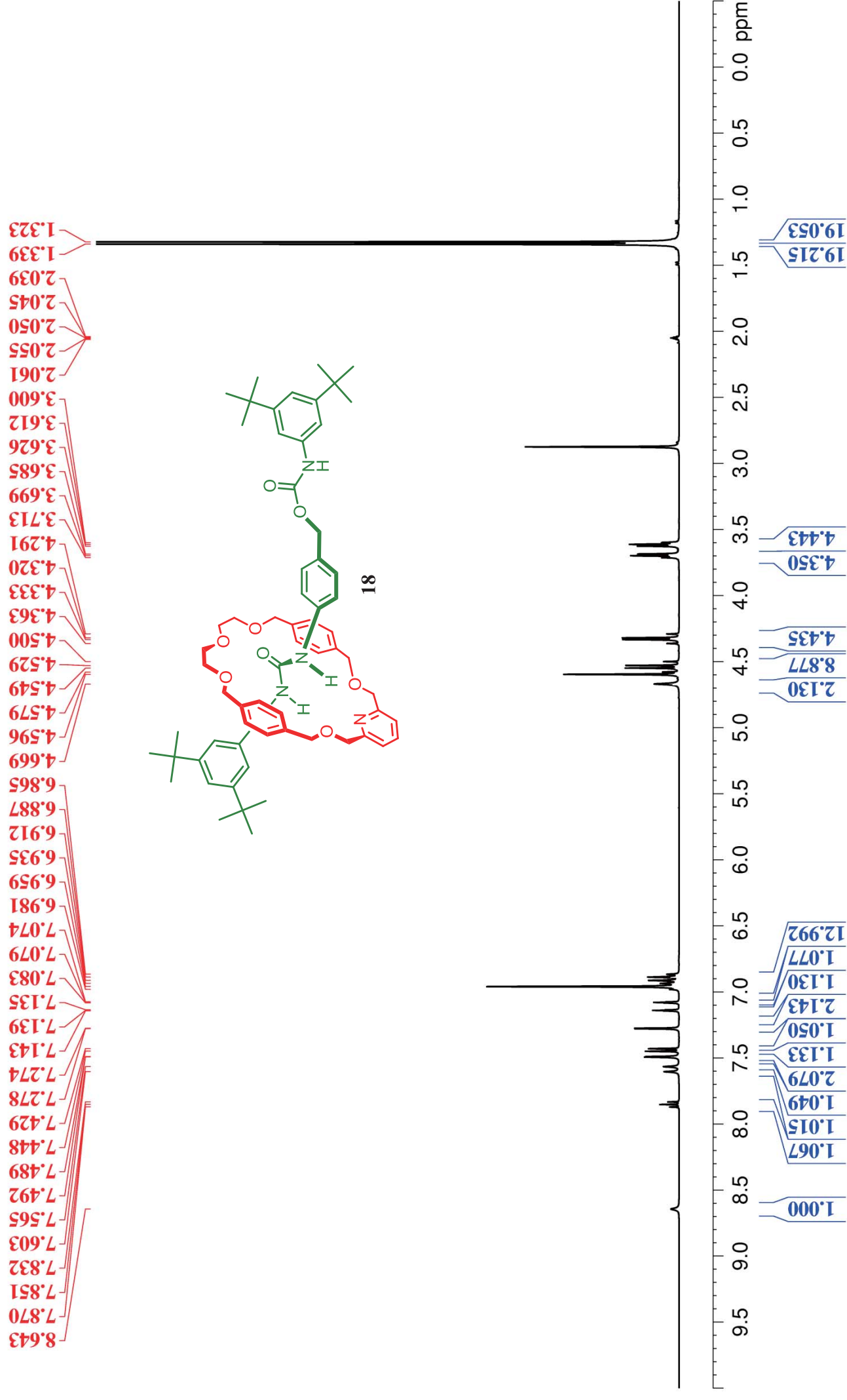


¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of 8 ∞ 

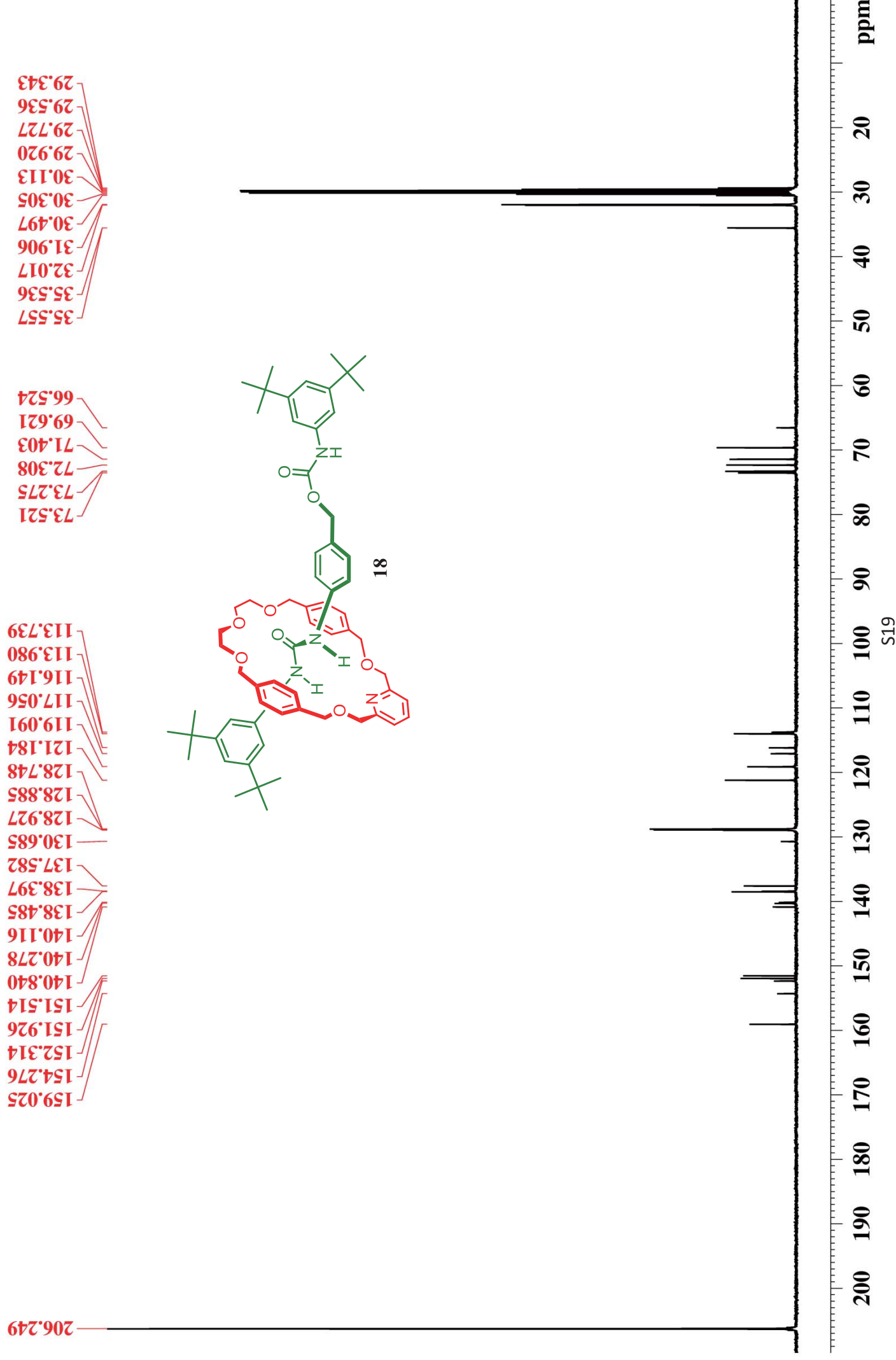
^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **8**



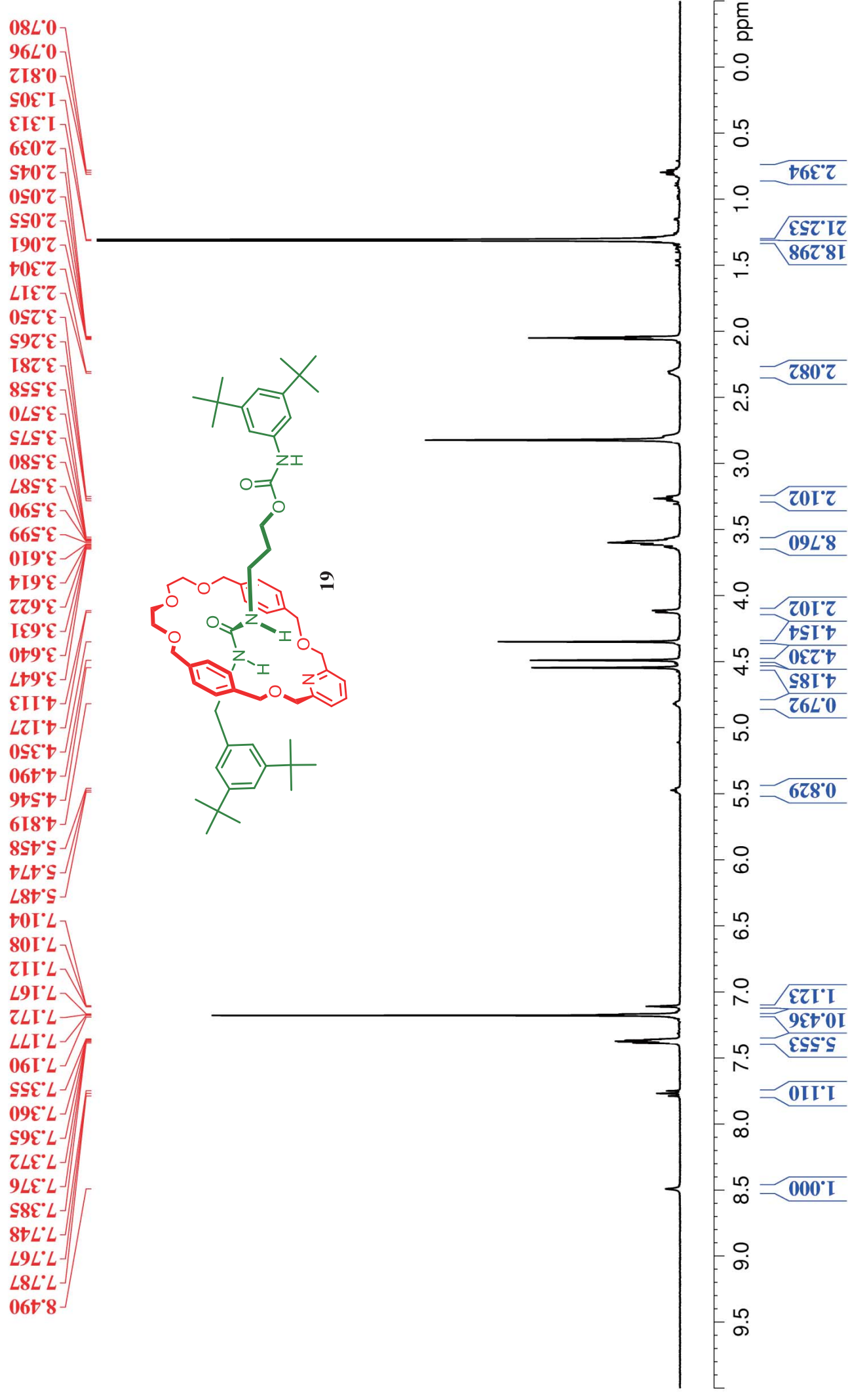
¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **18**



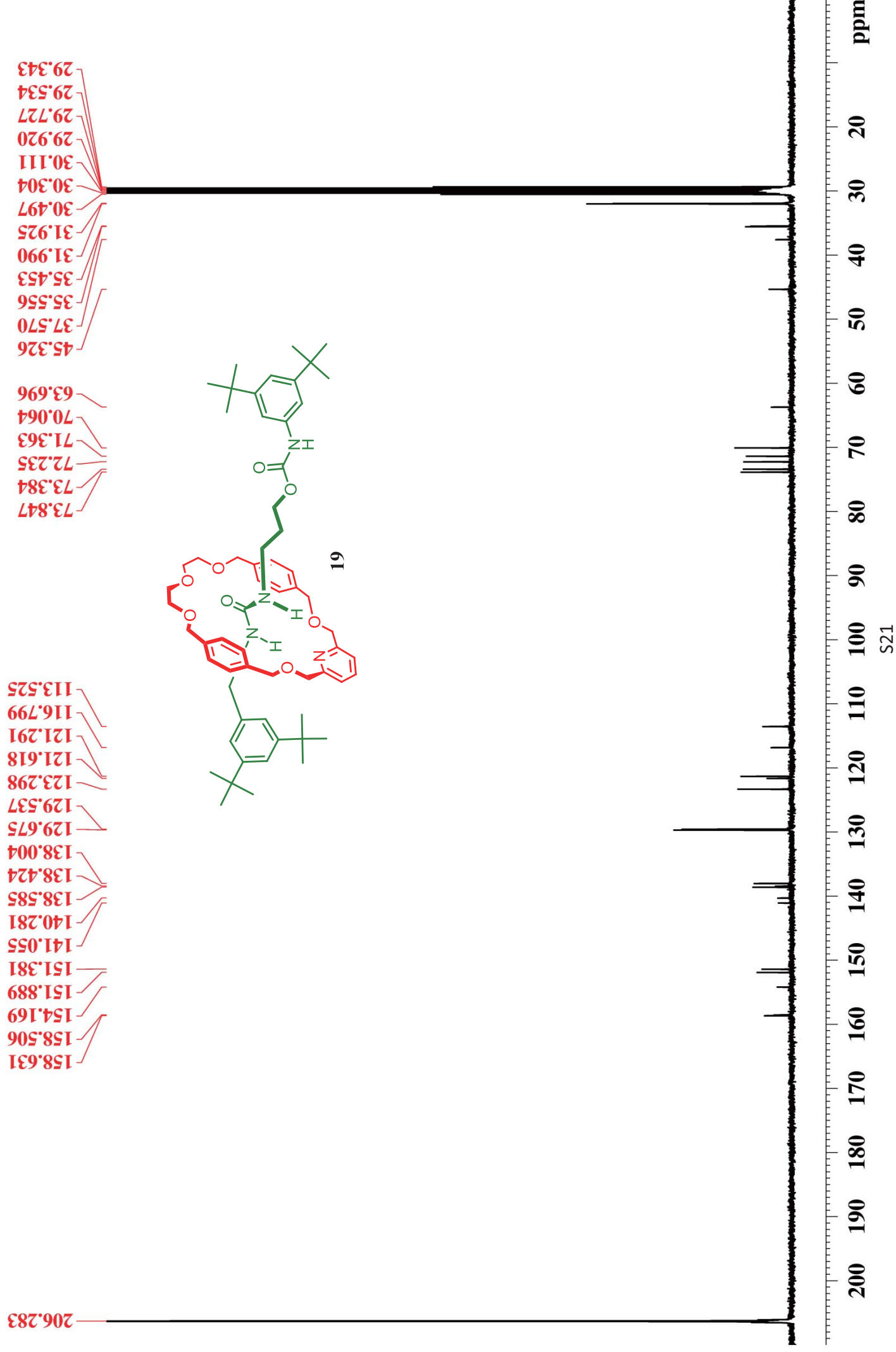
^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **18**



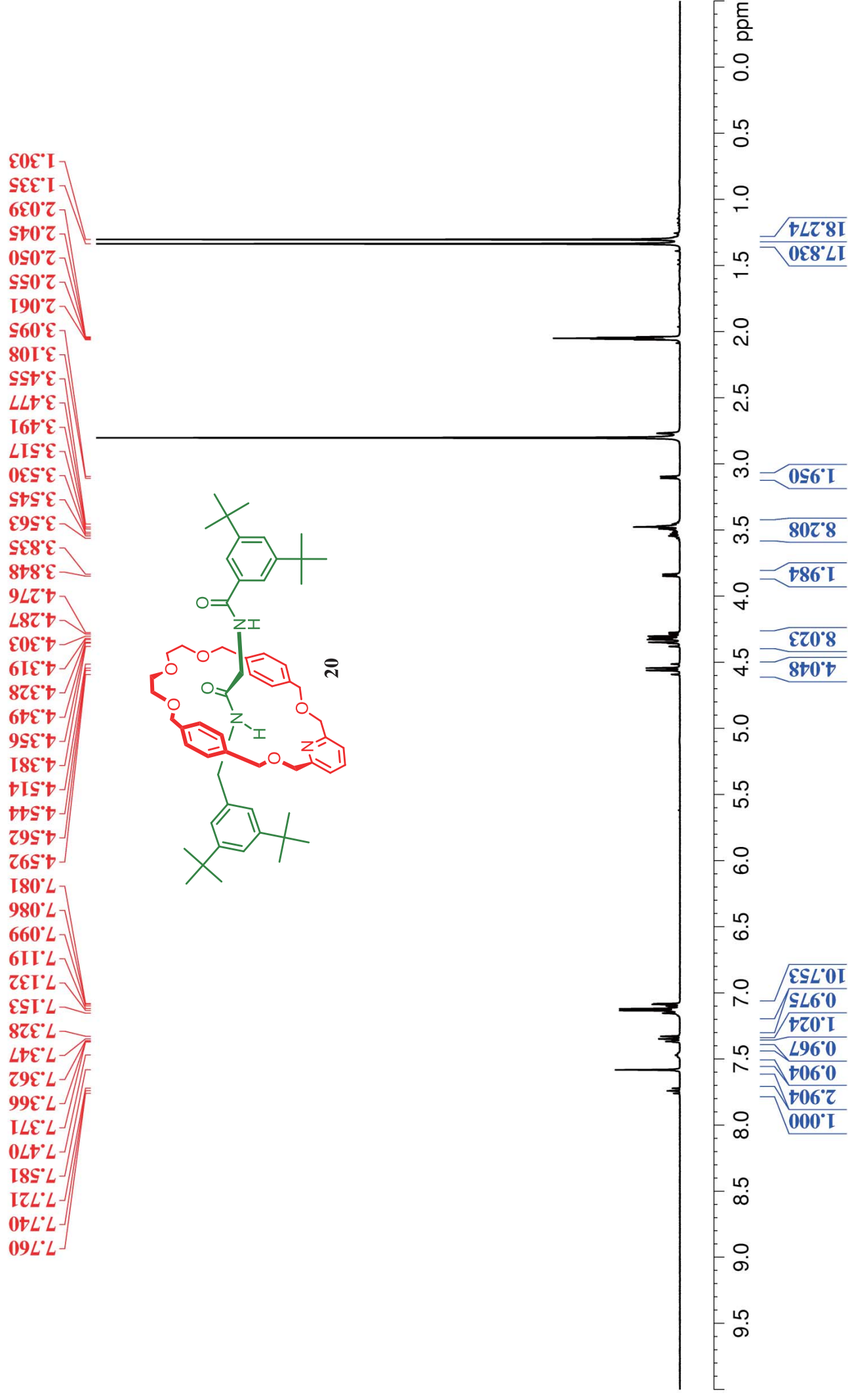
¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **19**



^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **19**



¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **20**



¹³C NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **20**

206.151

168.485

167.852

158.507

151.105

151.026

139.317

138.300

138.049

137.912

136.397

129.603

129.407

125.442

124.234

122.876

121.789

120.998

73.795

73.034

71.909

71.209

69.870

44.469

43.437

35.579

35.417

32.005

31.850

30.497

30.305

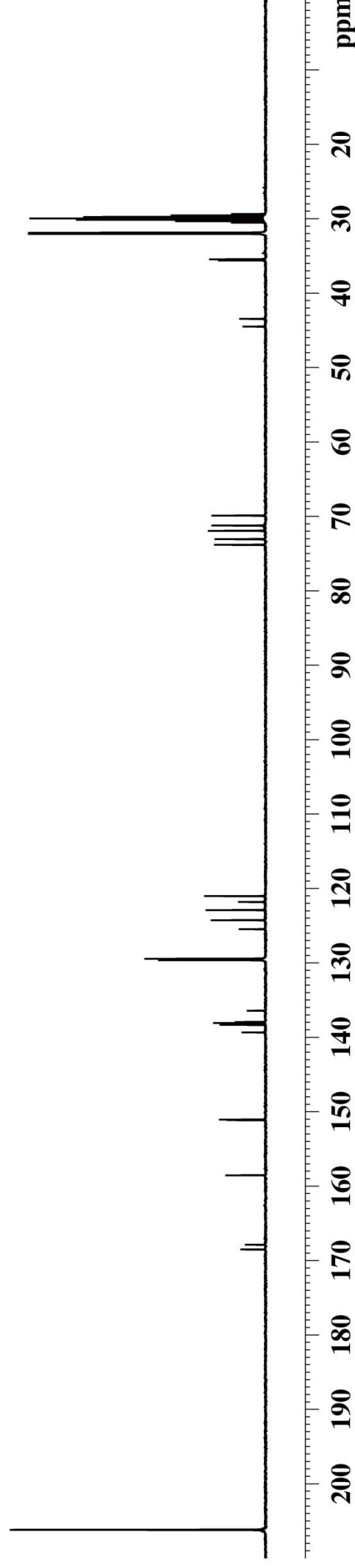
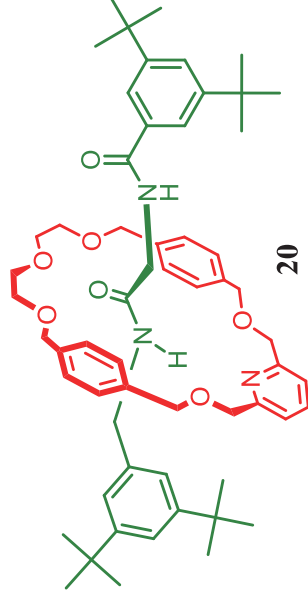
30.111

29.920

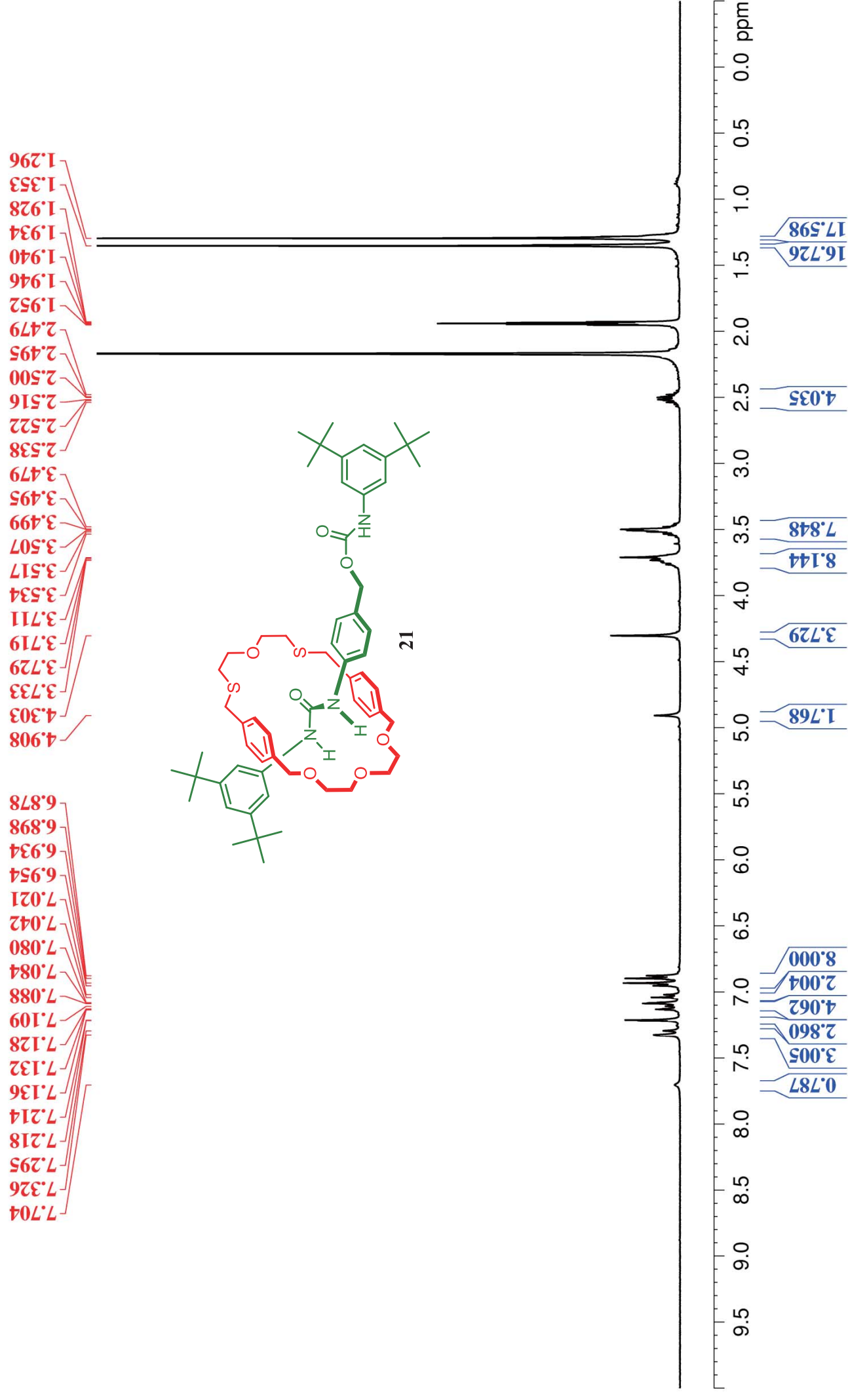
29.728

29.534

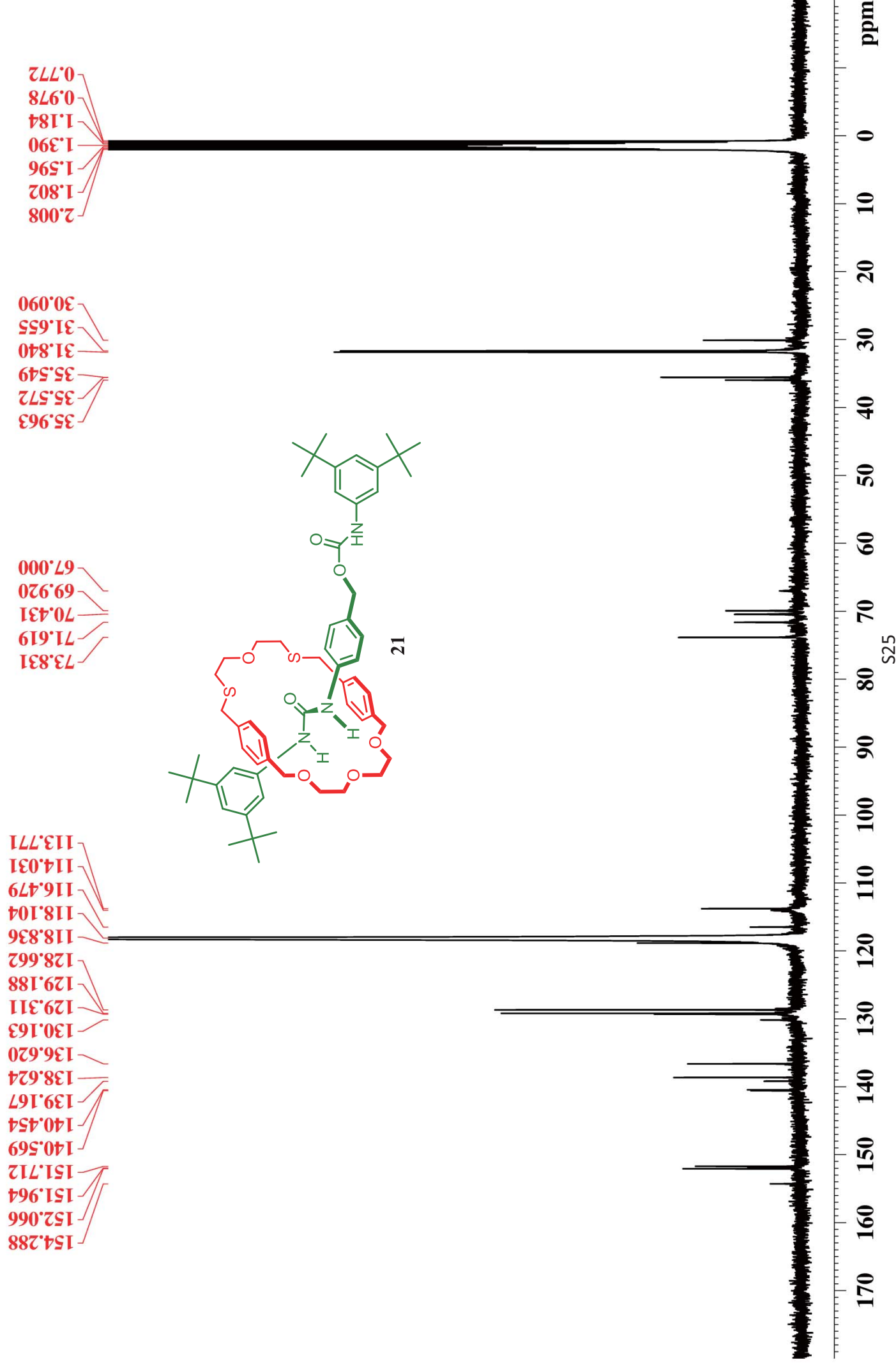
29.343



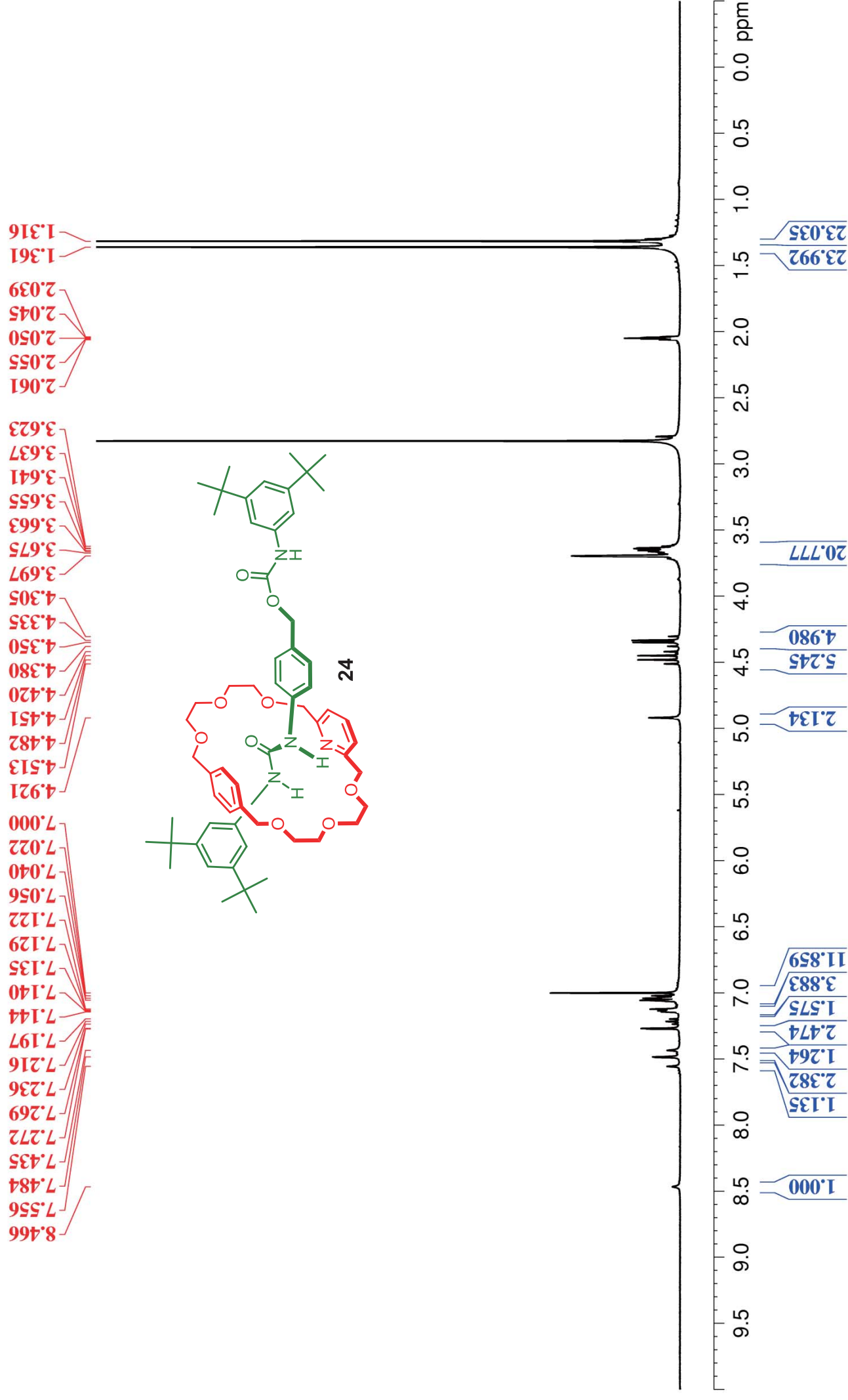
¹H NMR spectrum (400 MHz, CD₃CN, 298 K) of **21**



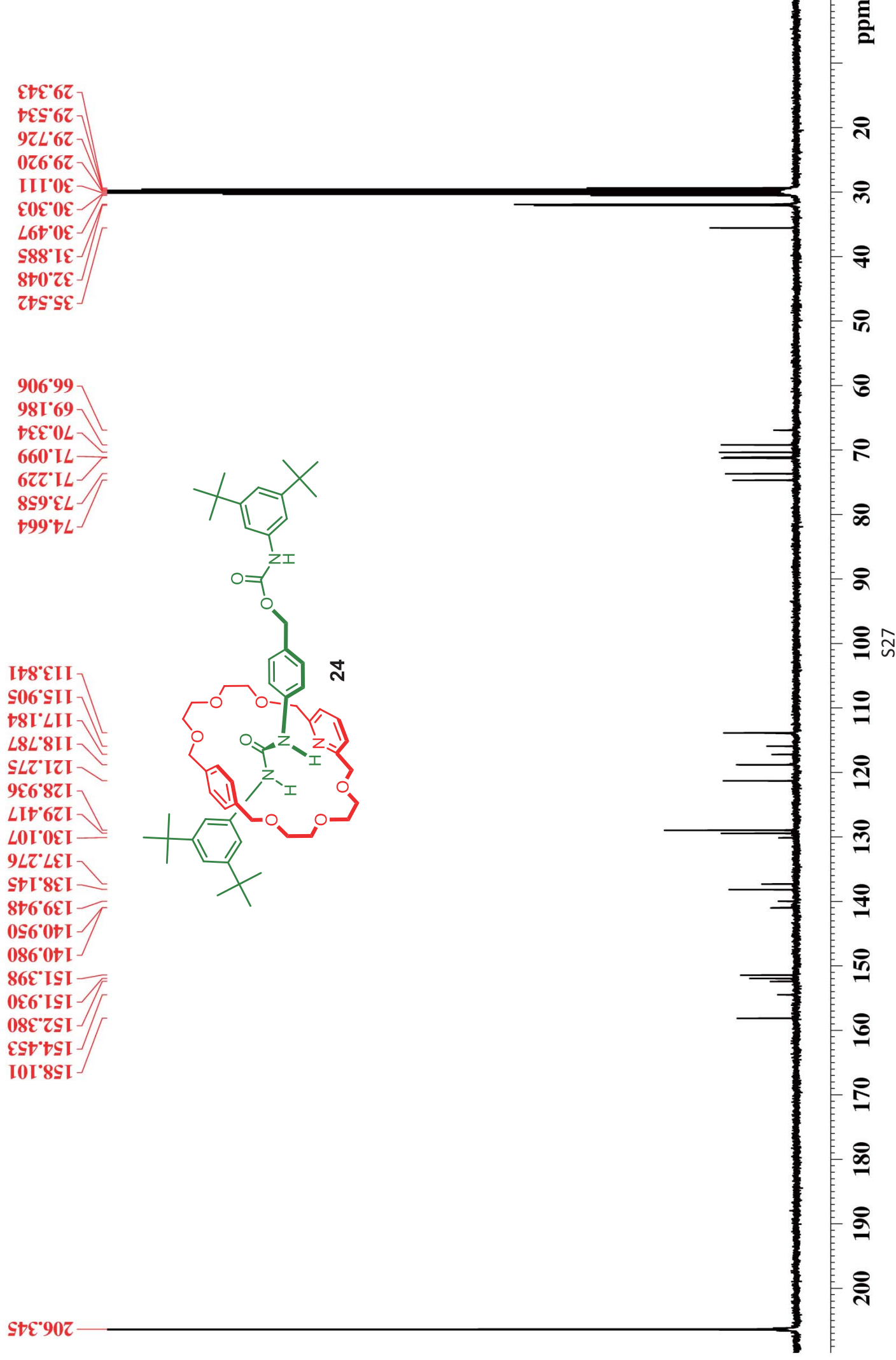
^{13}C NMR spectrum (400 MHz, CD_3CN , 298 K) of **21**



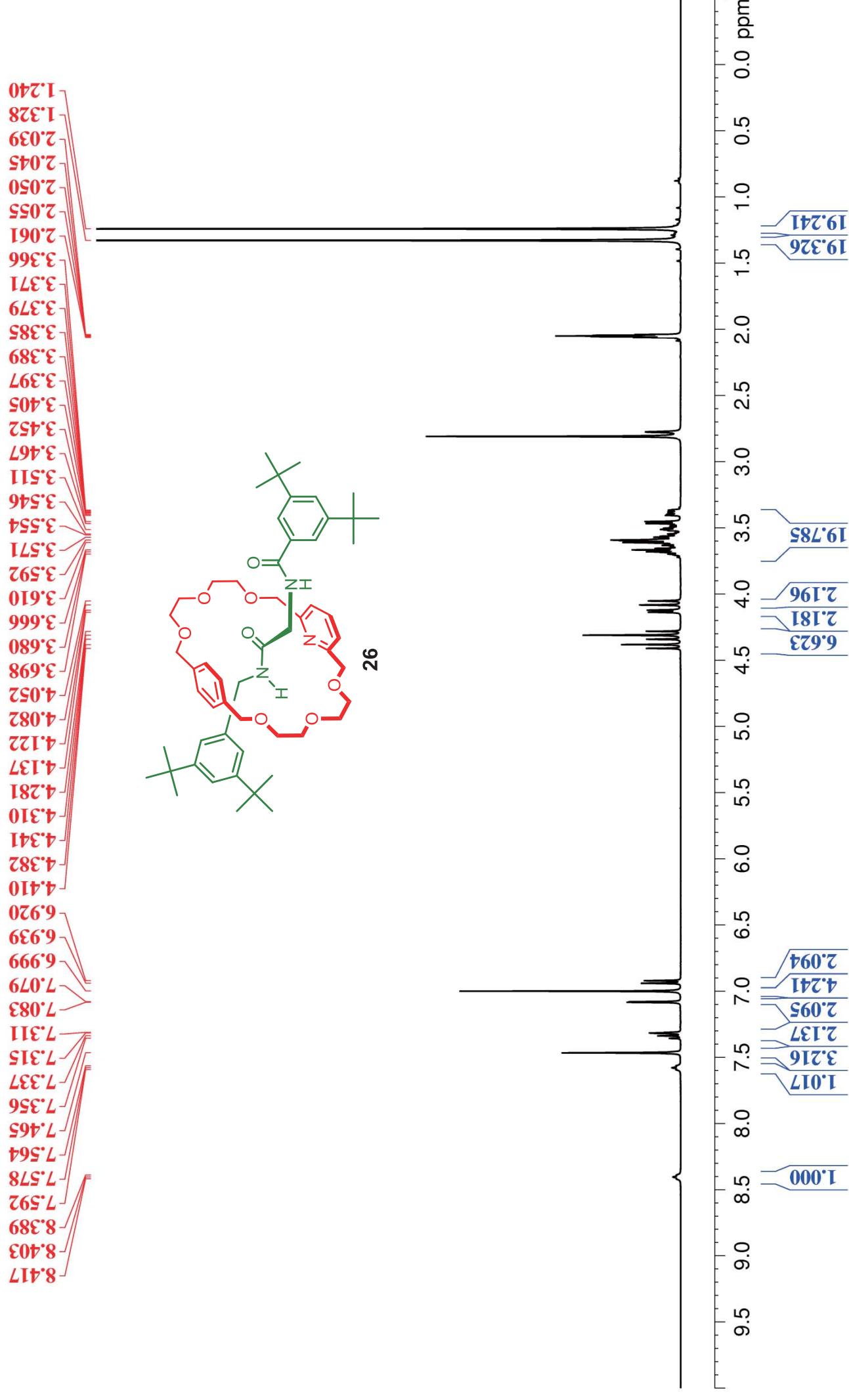
¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **24**



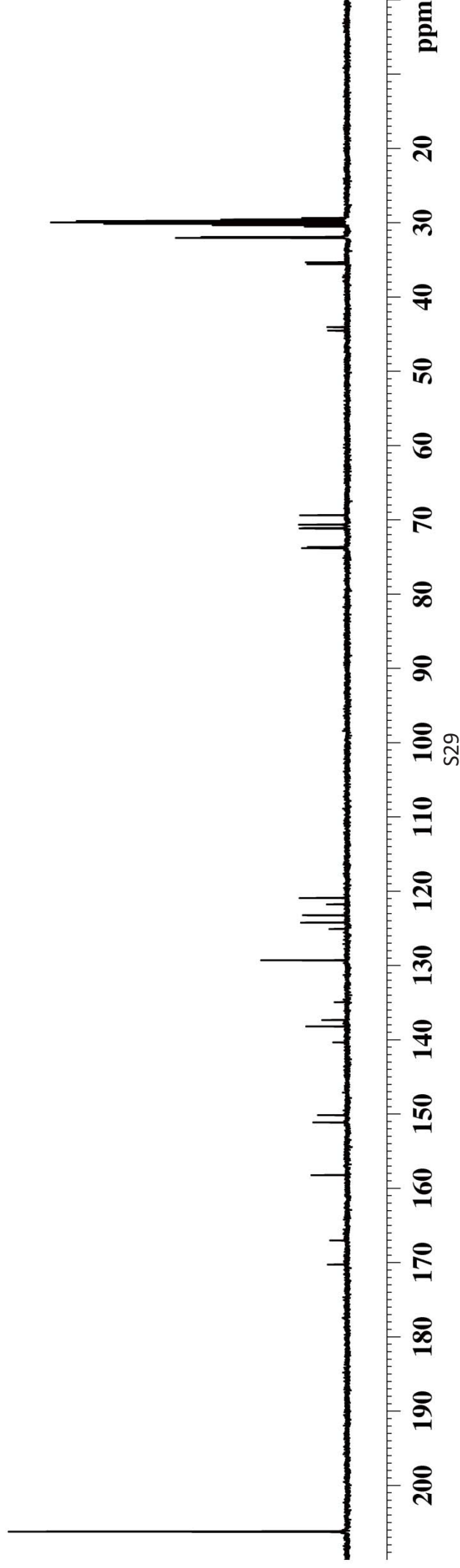
^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **24**



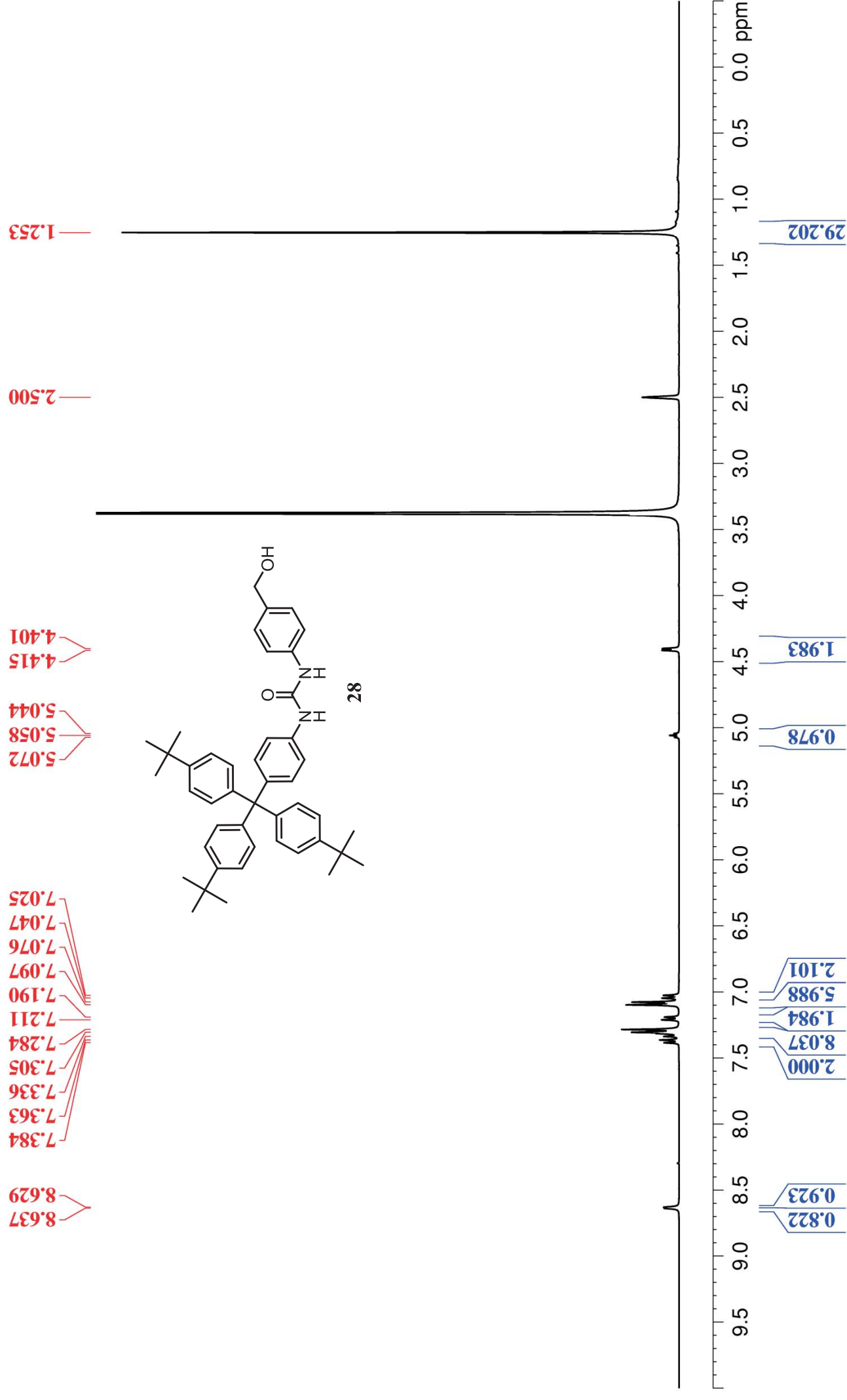
¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **26**



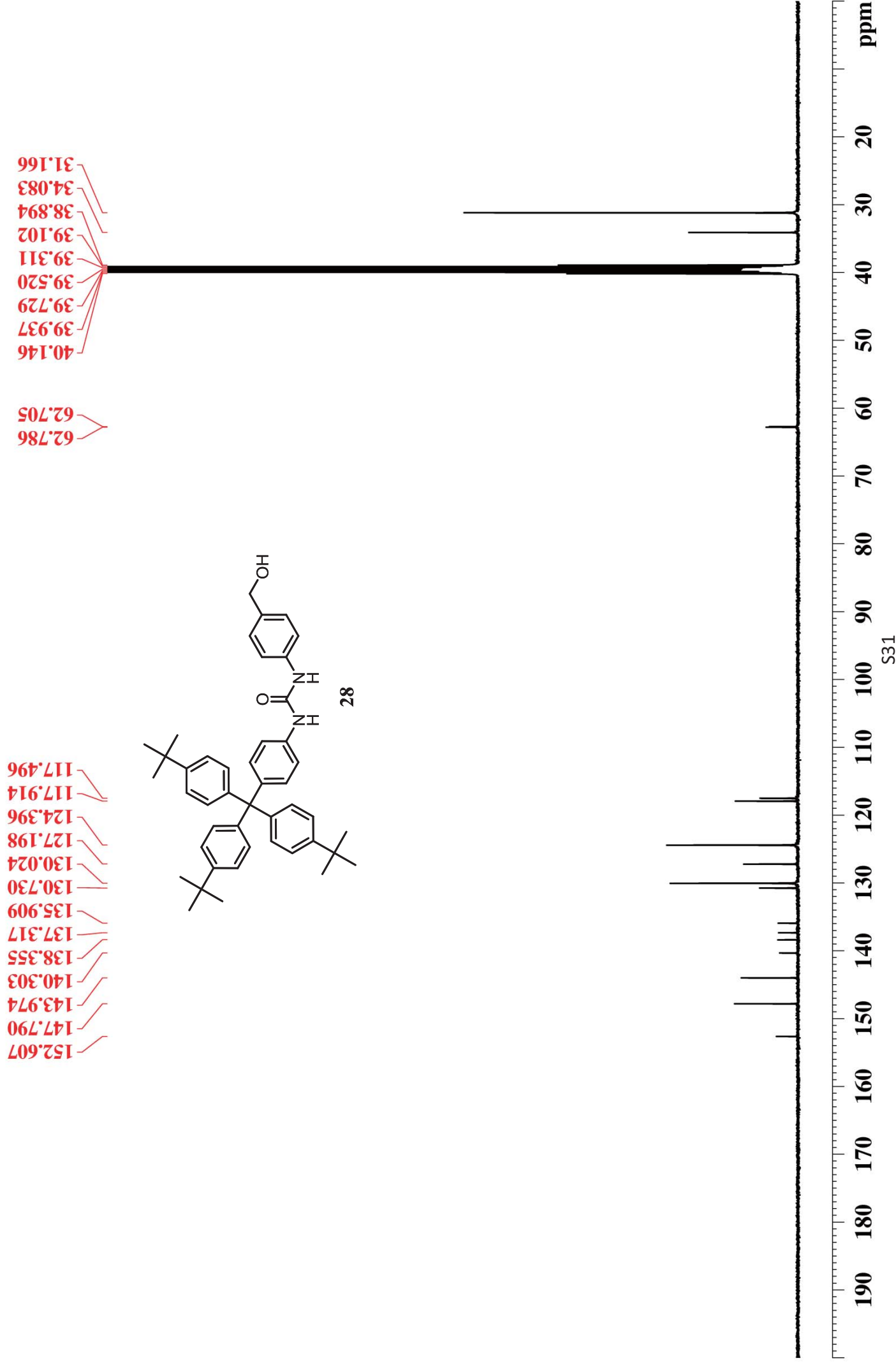
^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **26**



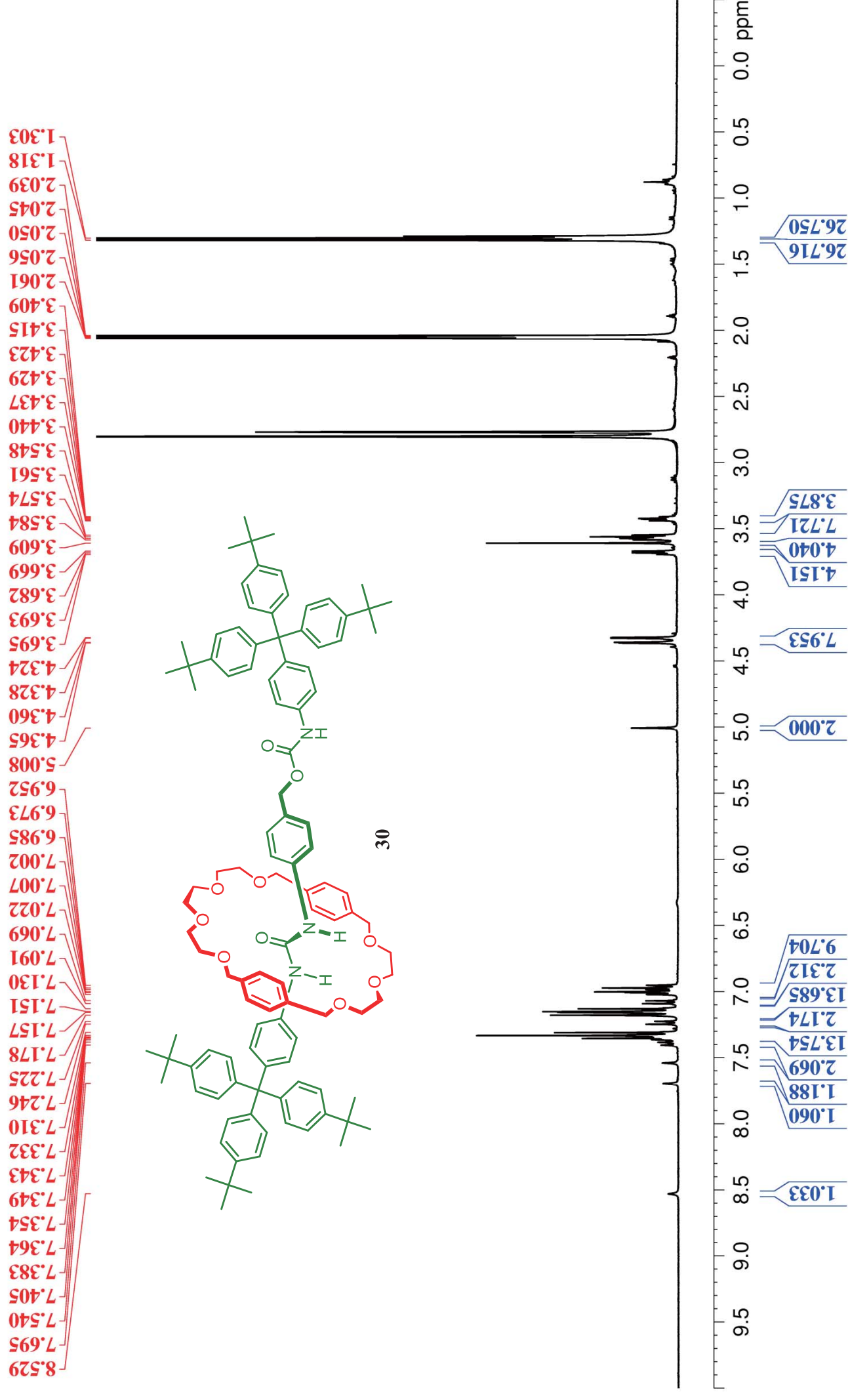
¹H NMR spectrum (400 MHz, CD₃SOCD₃, 298 K) of **28**



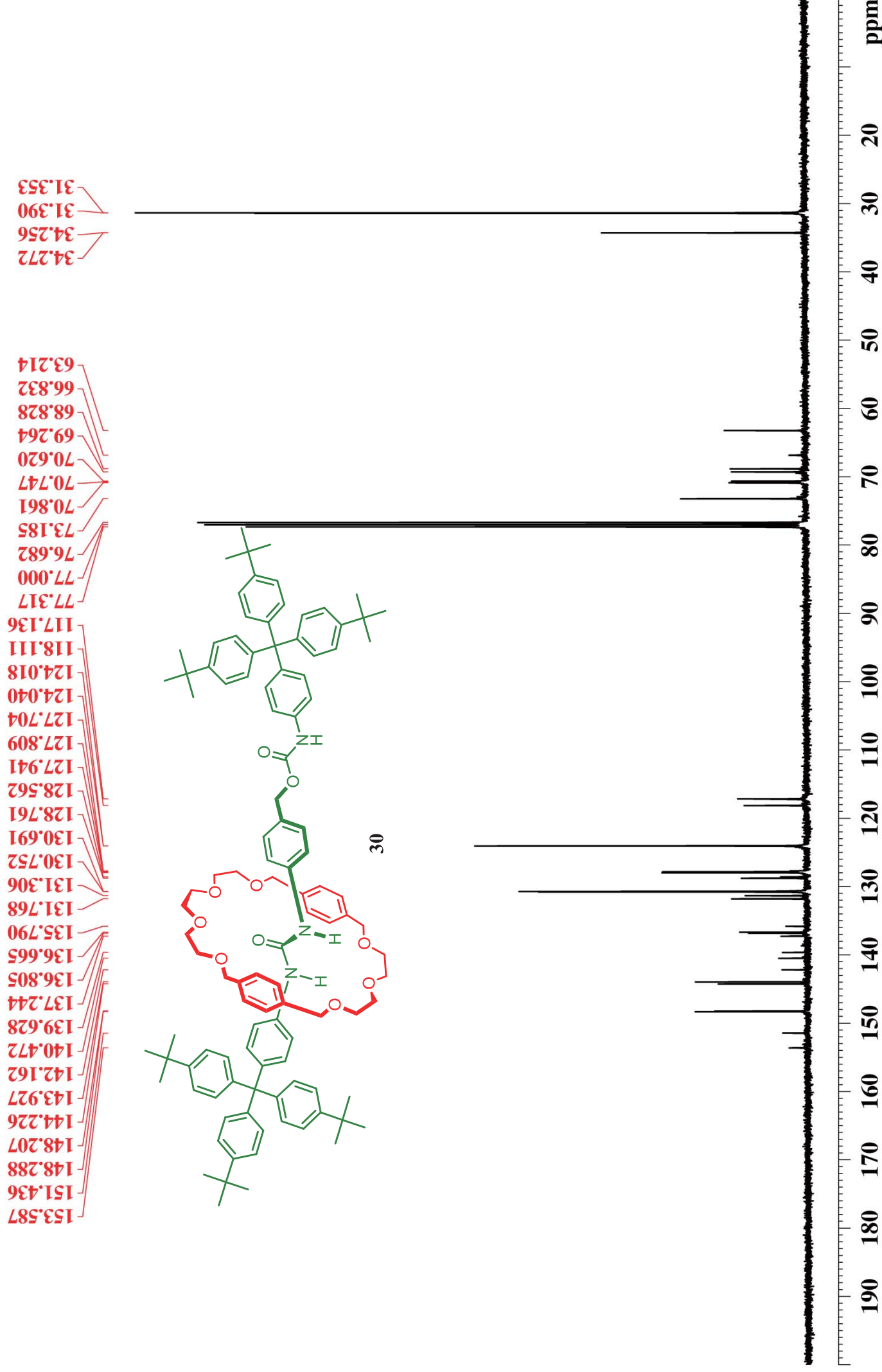
^{13}C NMR spectrum (400 MHz, CD_3SOCD_3 , 298 K) of **28**



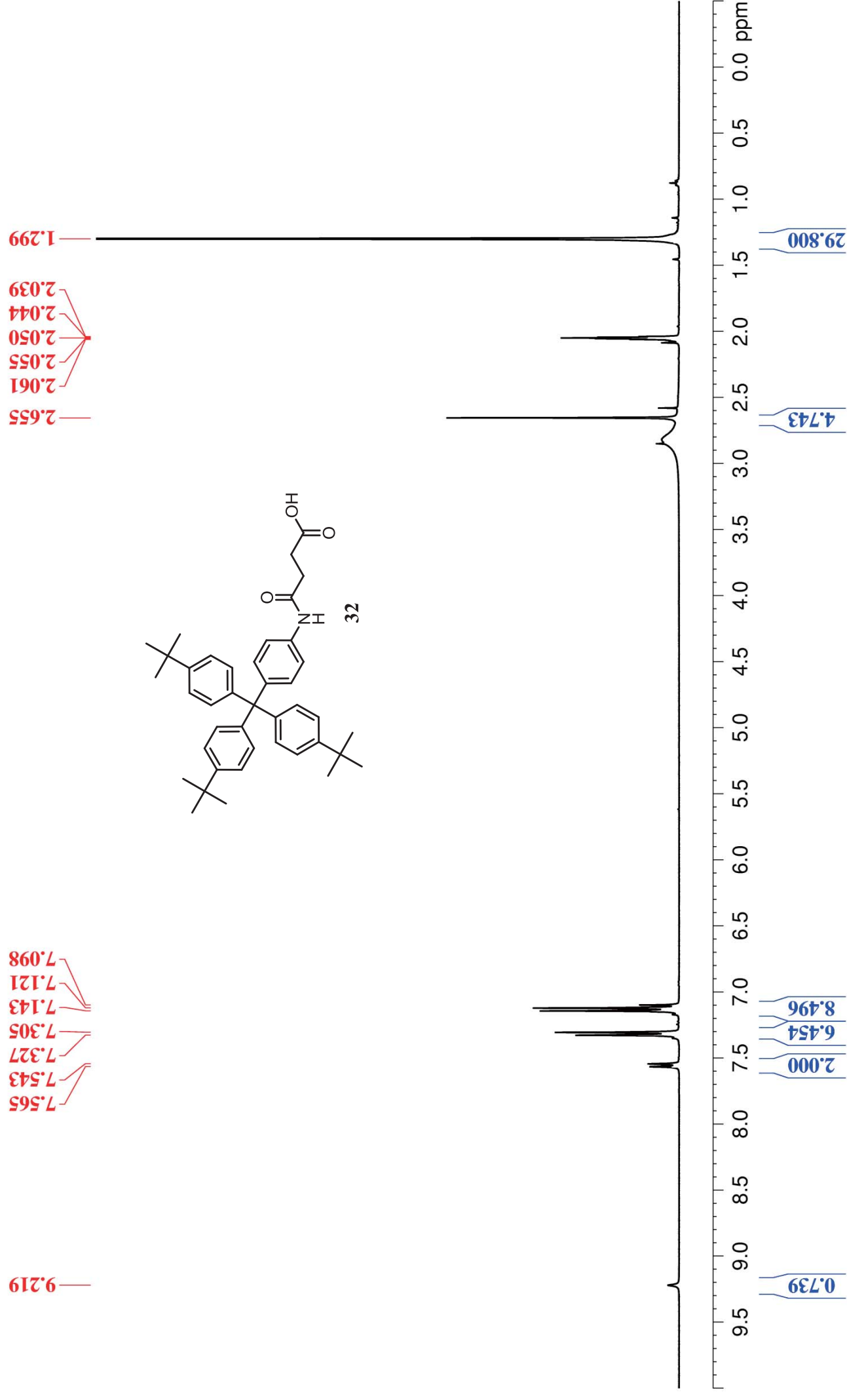
¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **30**



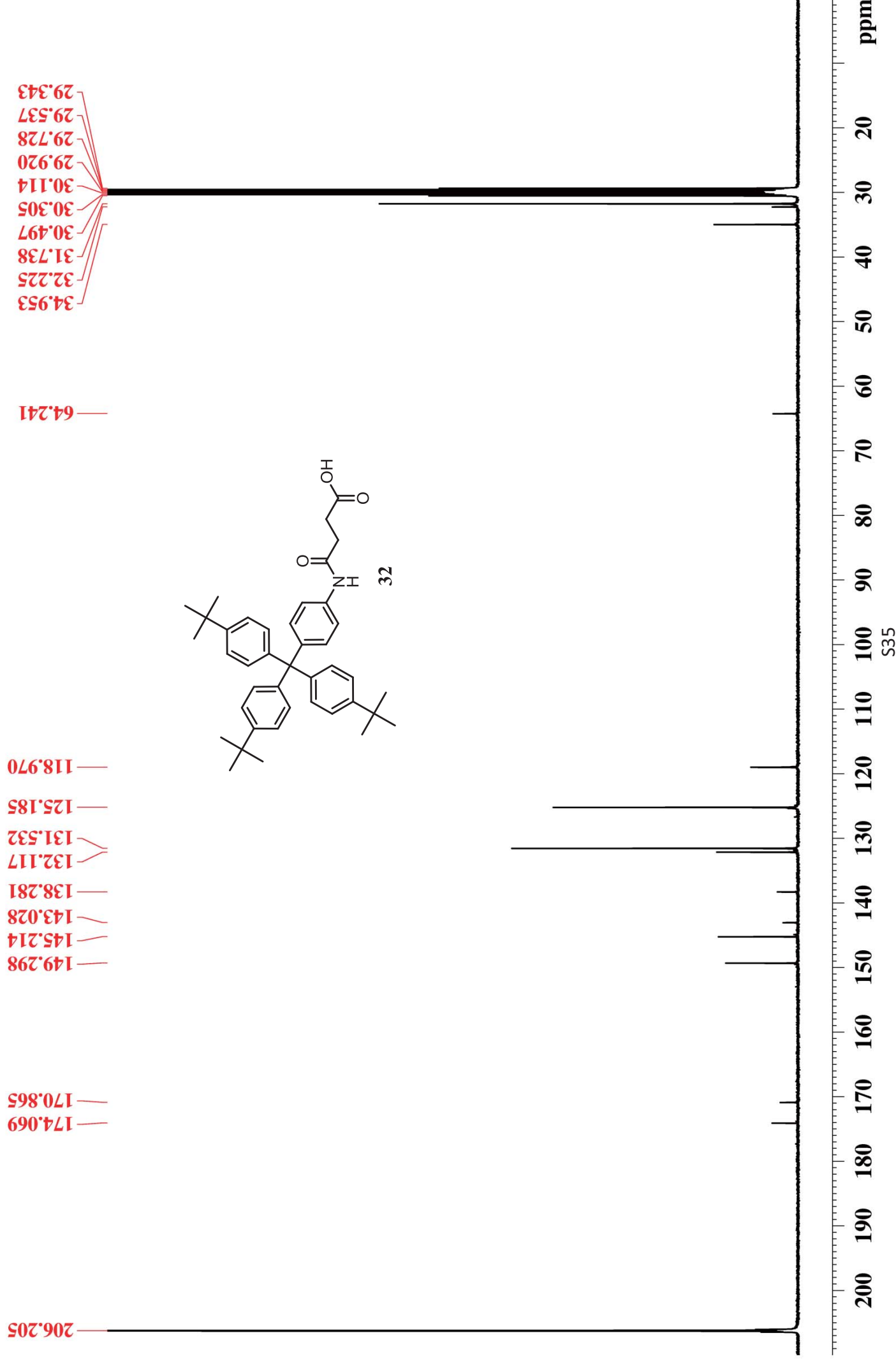
^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of 30



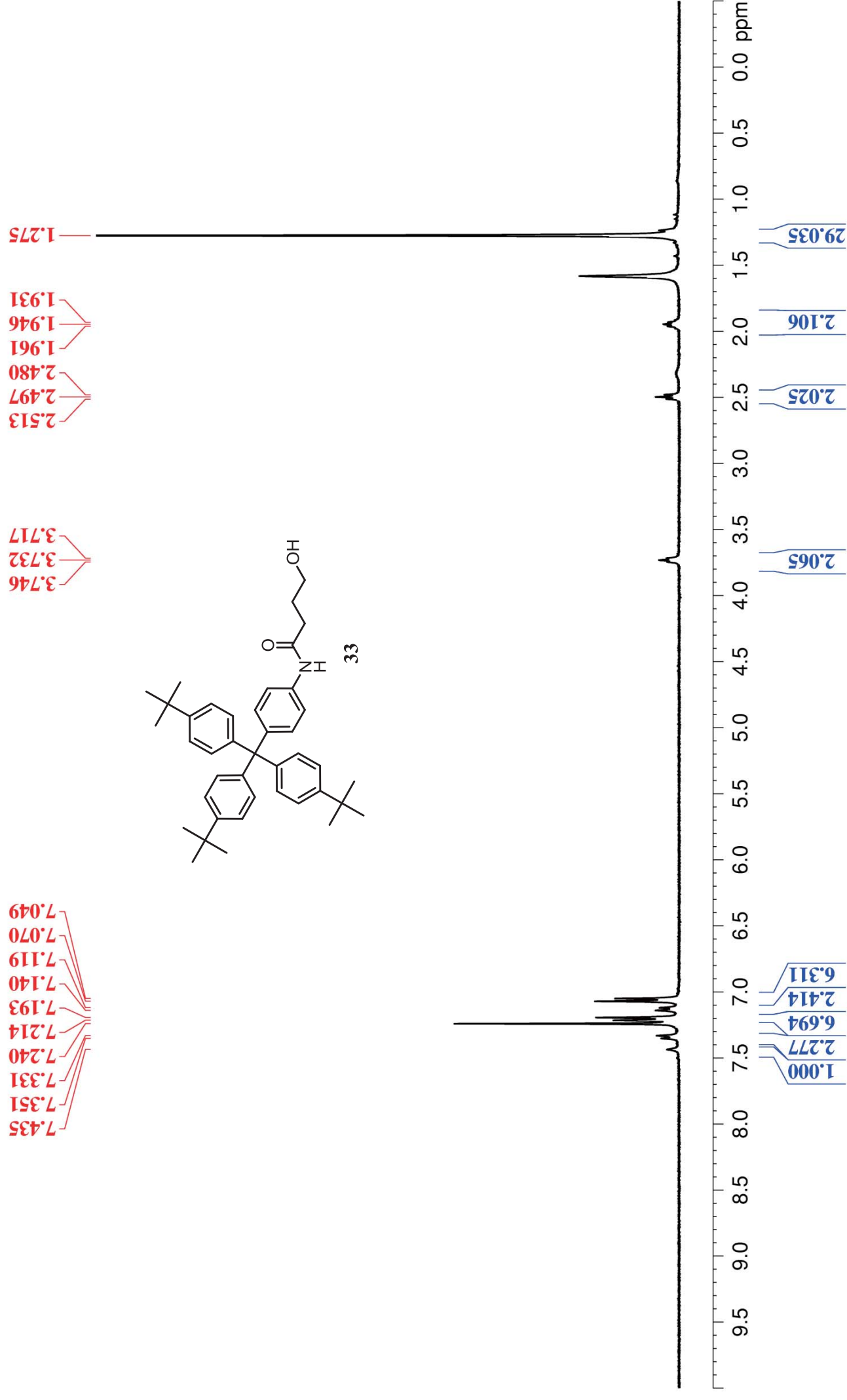
^1H NMR spectrum (400 MHz, CD_3SOCD_3 , 298 K) of **32**



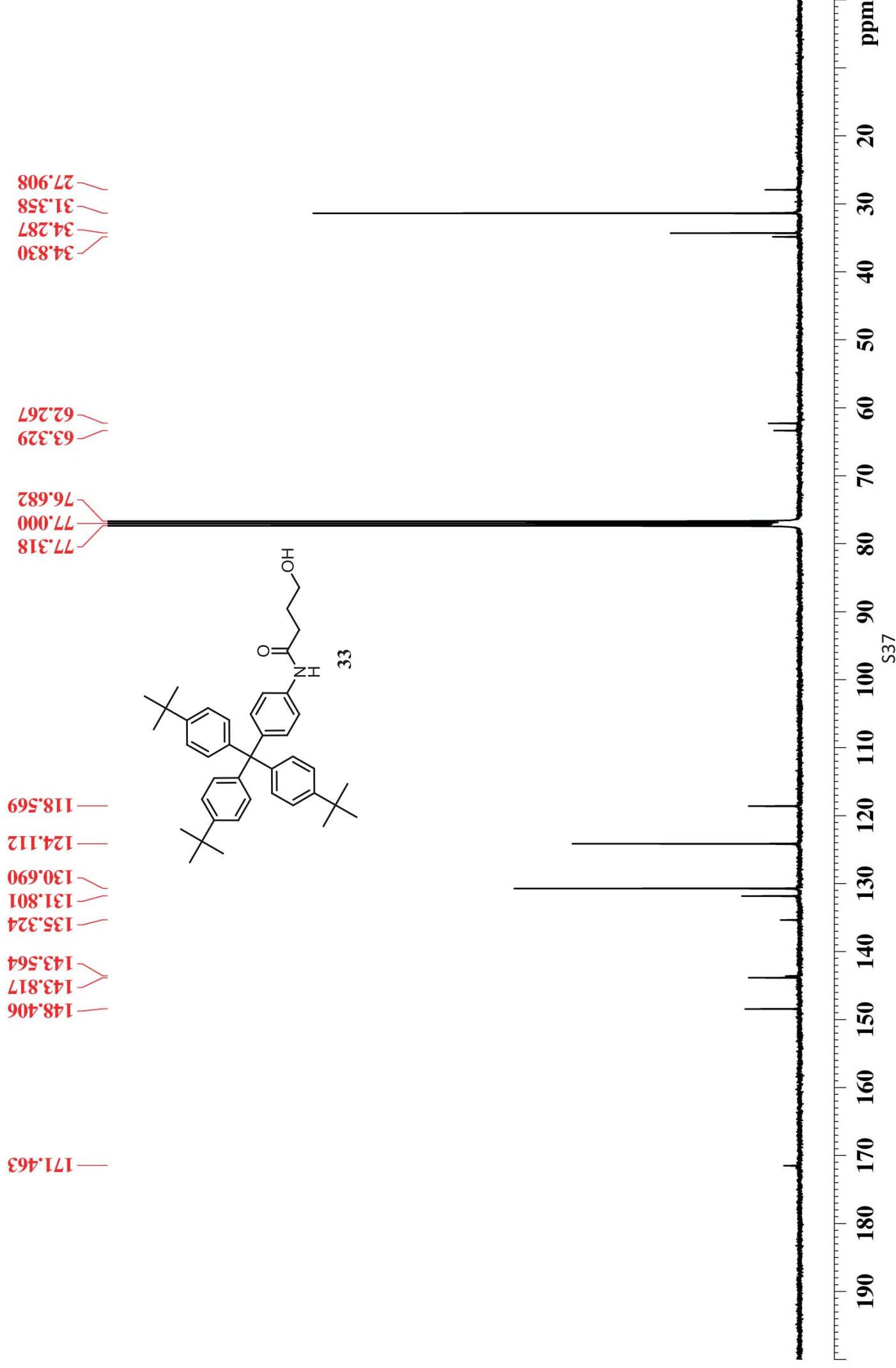
^{13}C NMR spectrum (400 MHz, CD_3SOCD_3 , 298 K) of 32



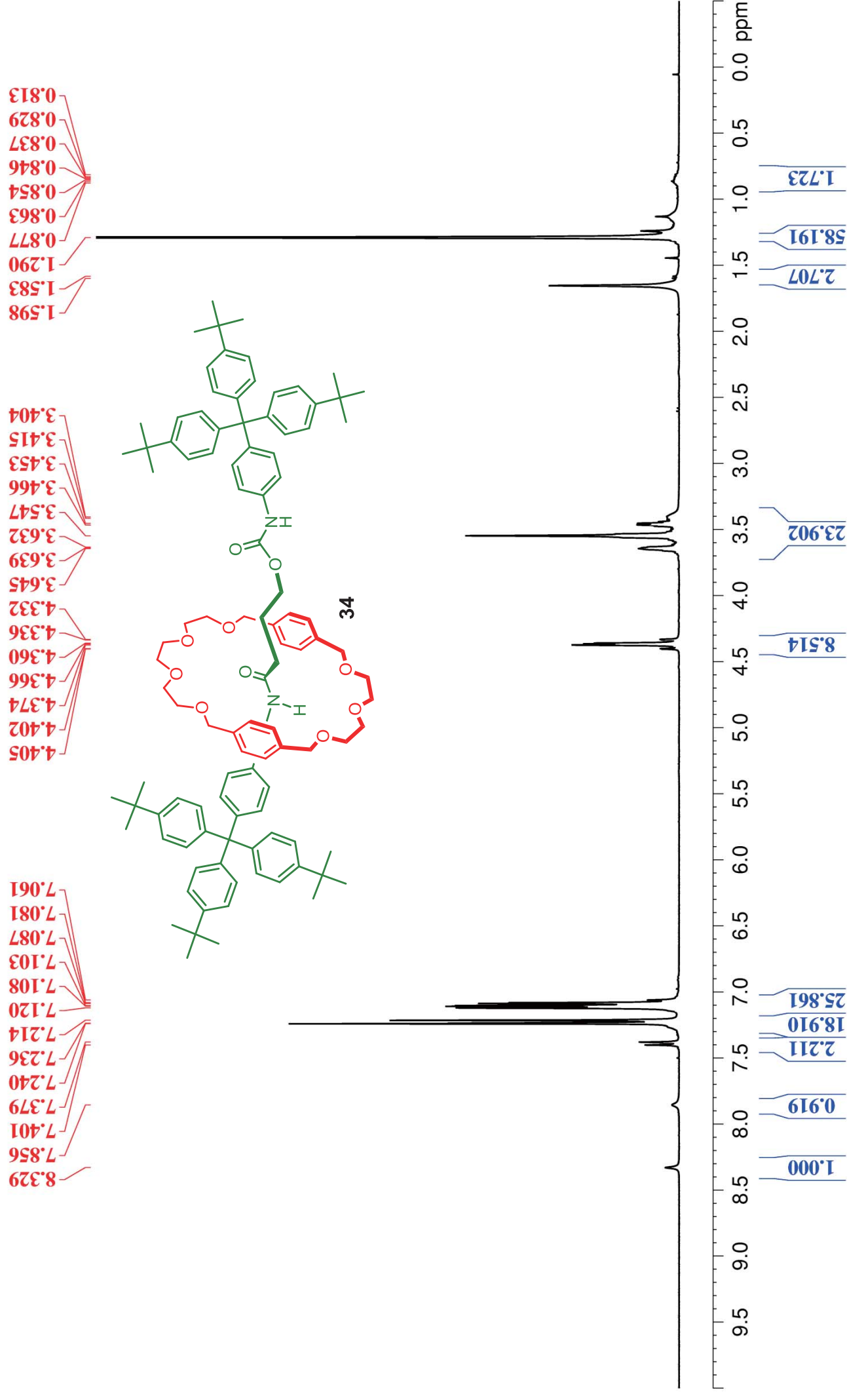
^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **33**



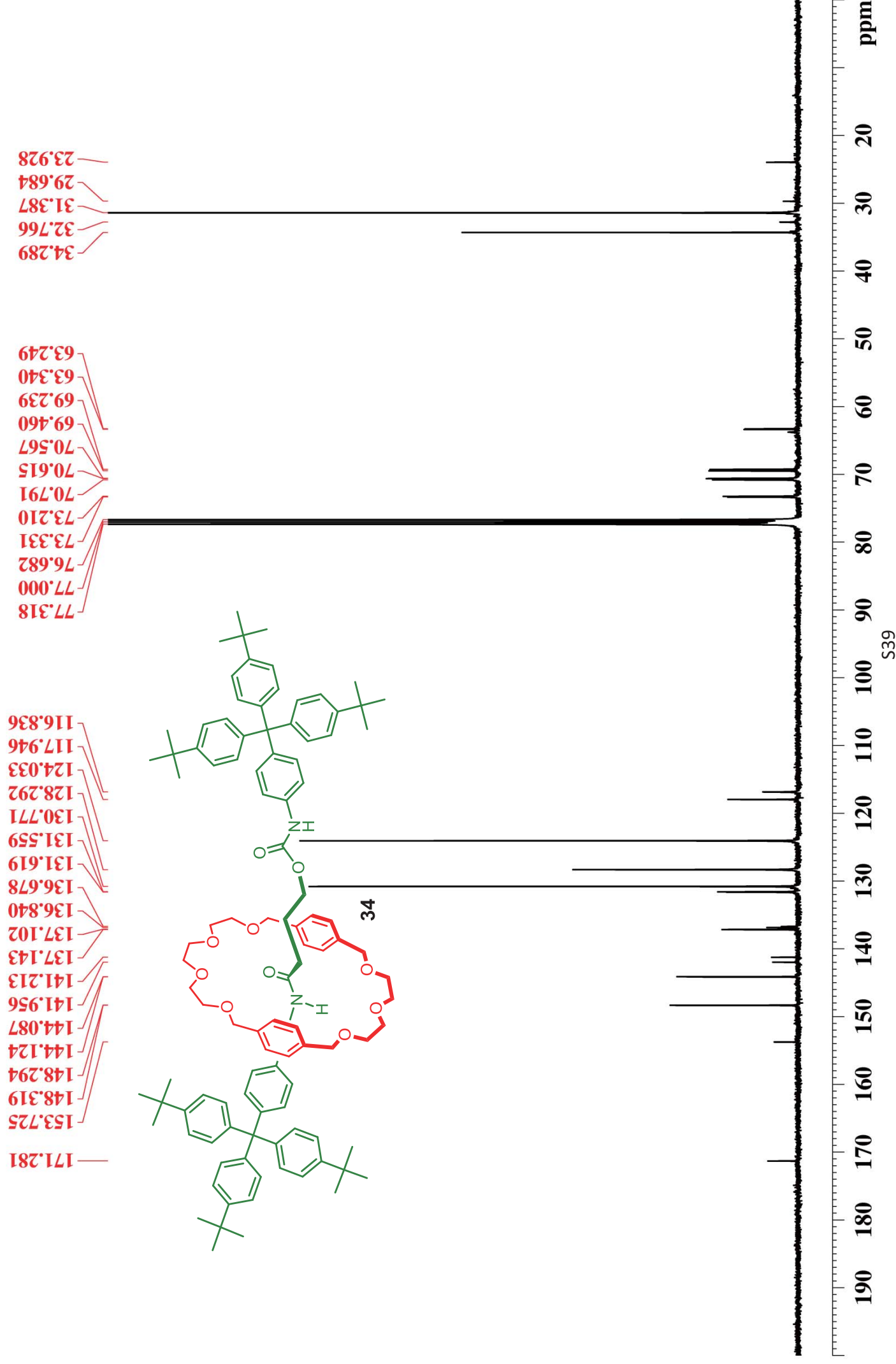
^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **33**



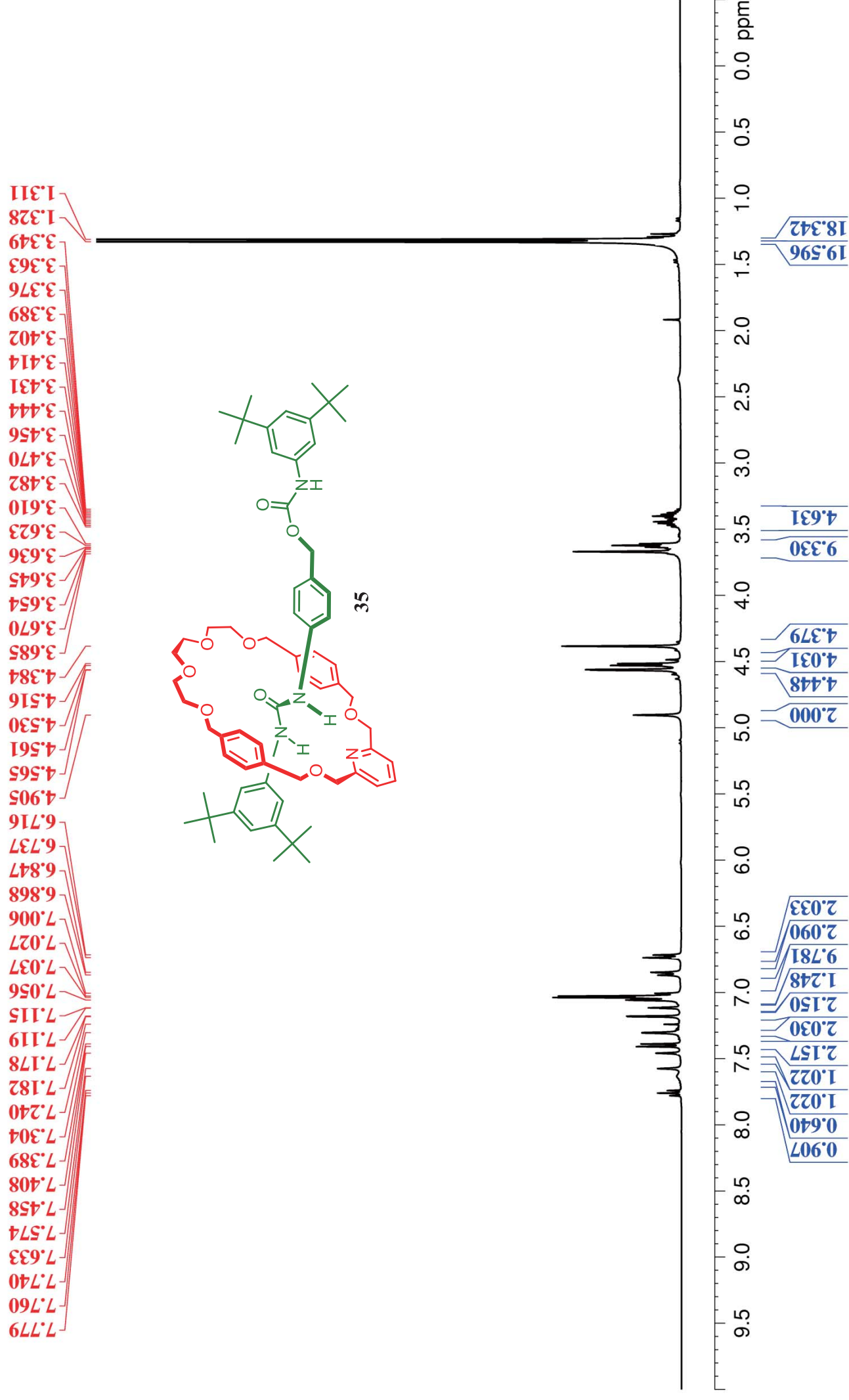
^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **34**



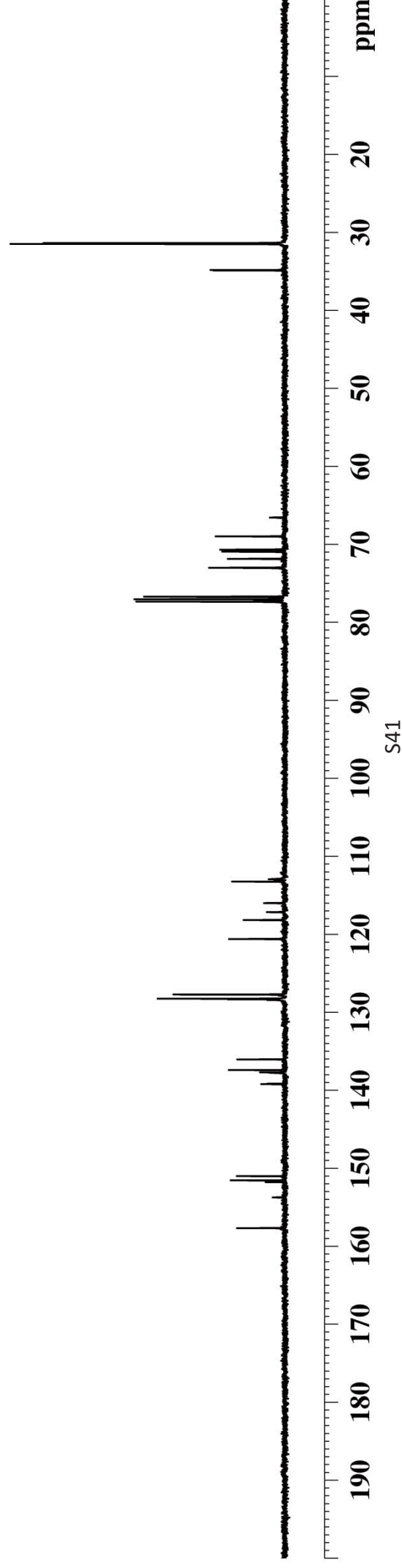
^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **34**



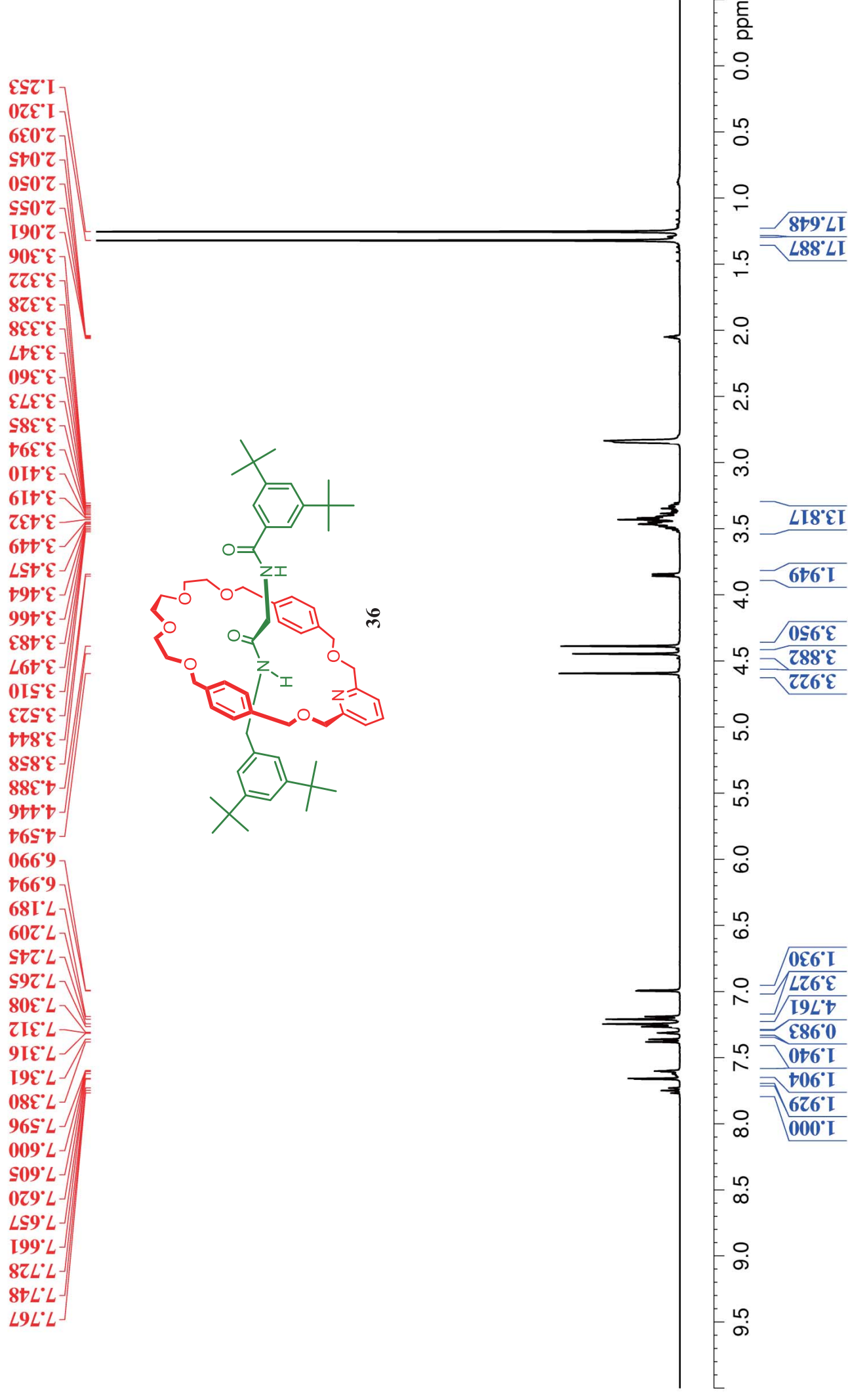
^1H NMR spectrum (400 MHz, CDCl_3 , 298 K) of **35**



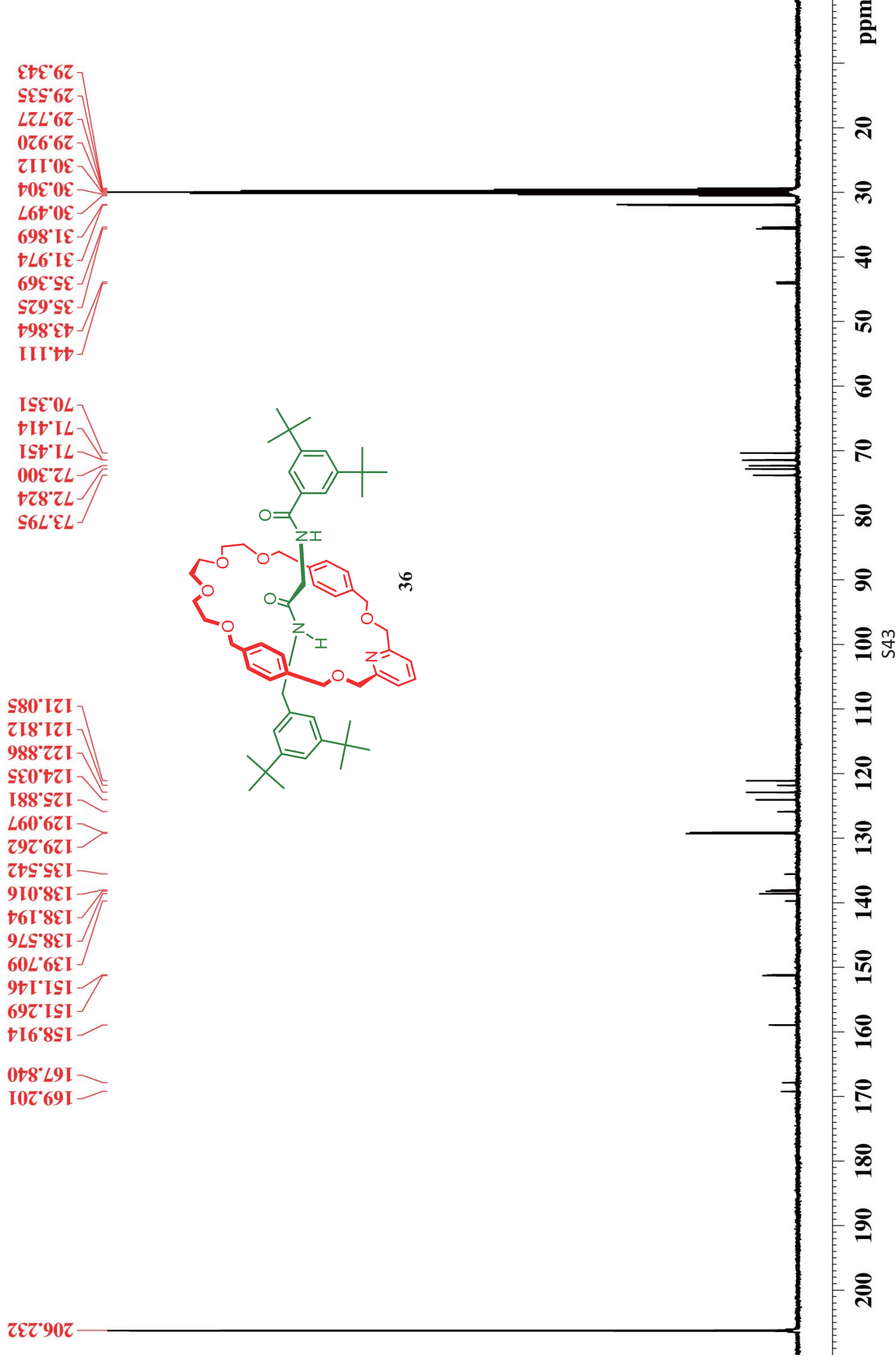
^{13}C NMR spectrum (400 MHz, CDCl_3 , 298 K) of **35**



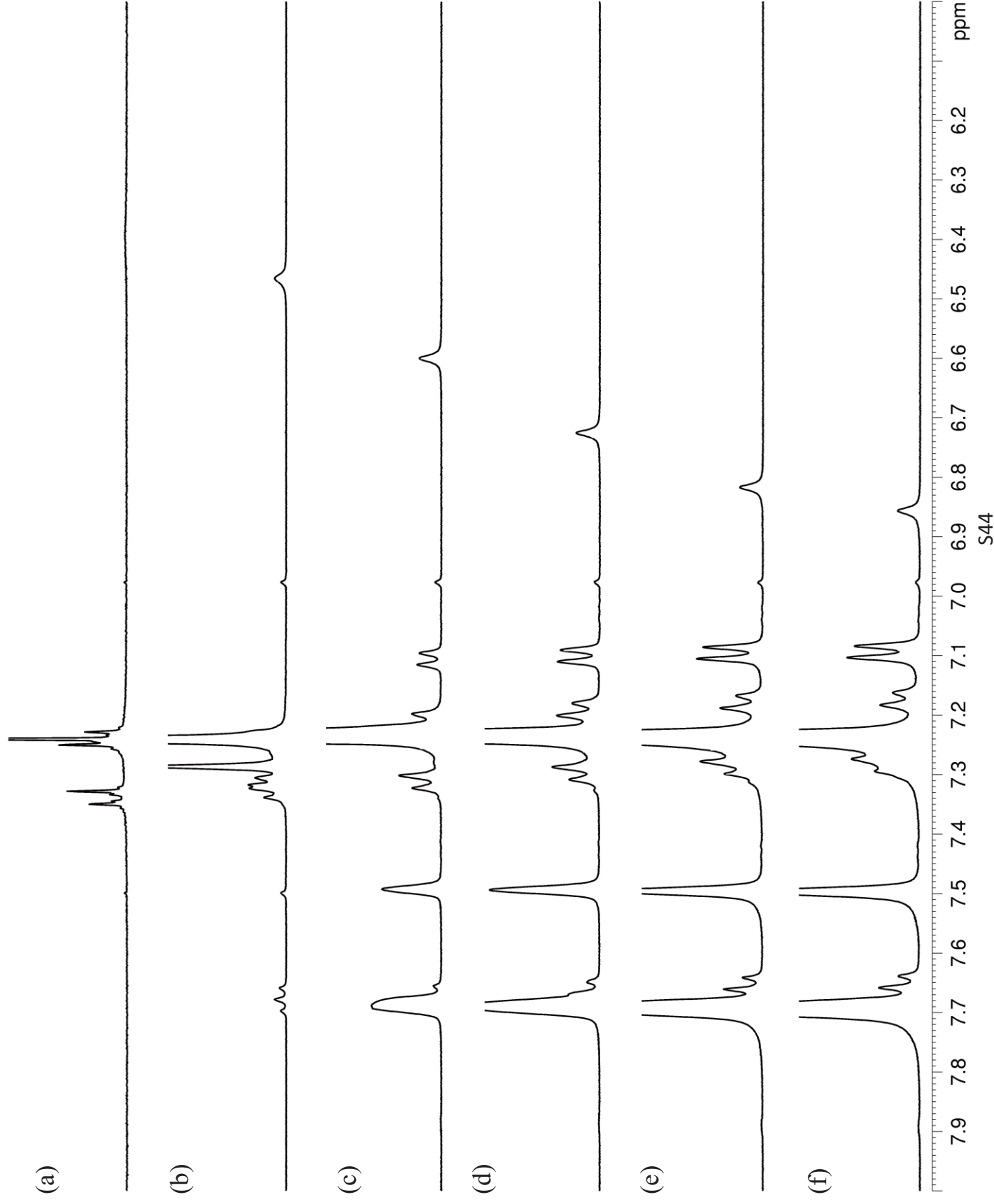
¹H NMR spectrum (400 MHz, CD₃COCD₃, 298 K) of **36**



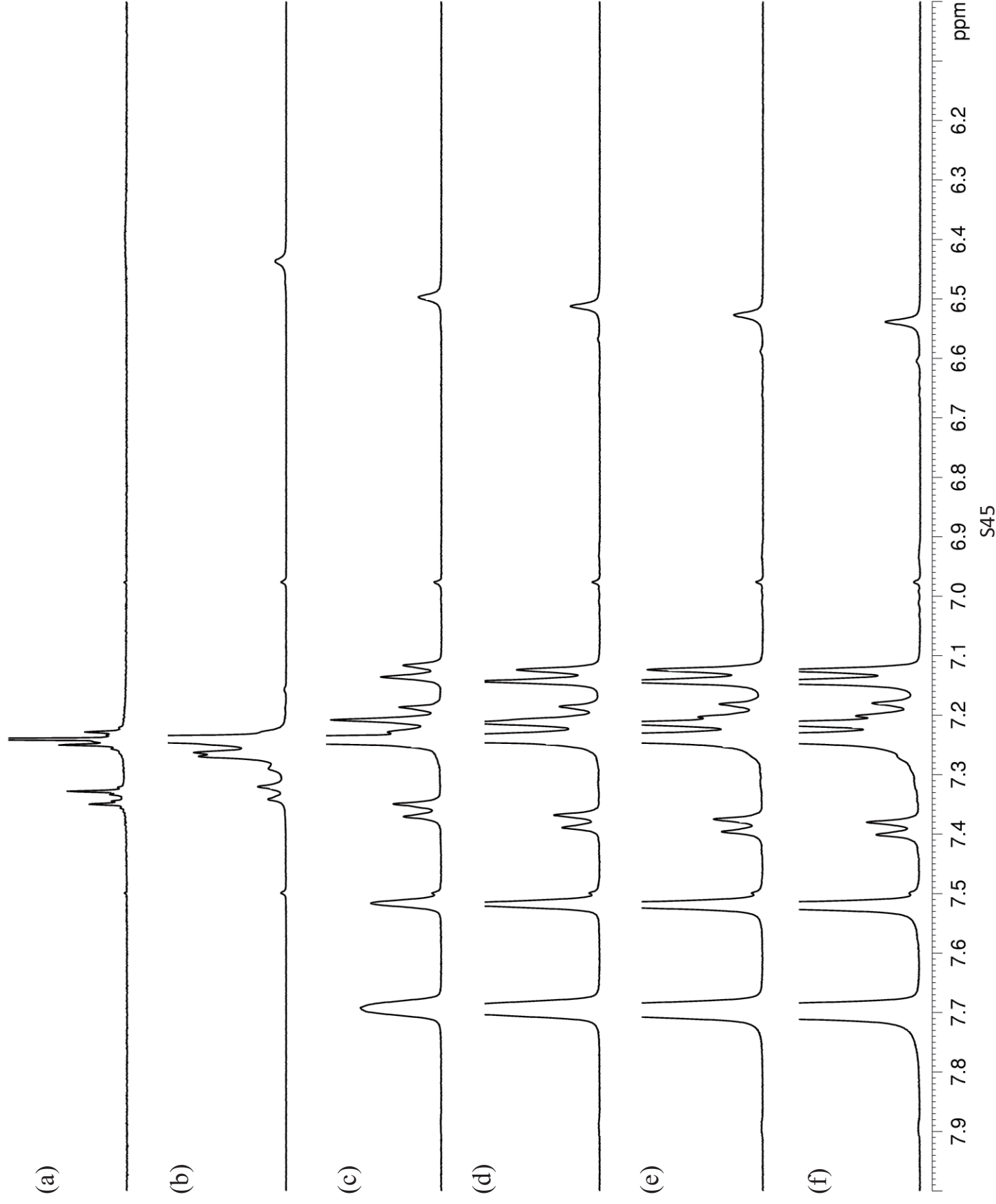
^{13}C NMR spectrum (400 MHz, CD_3COCD_3 , 298 K) of **36**



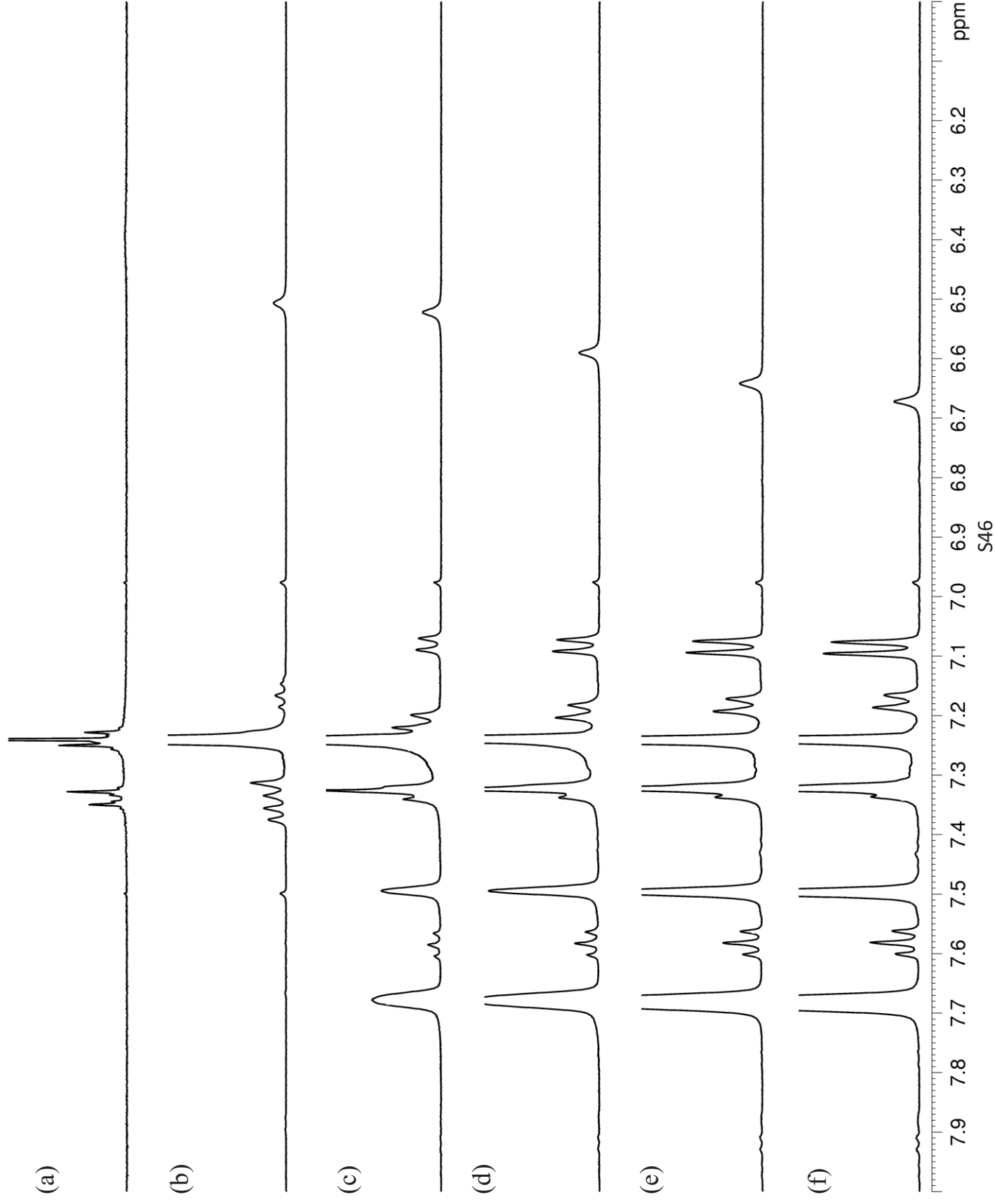
Partial ^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) urea **9**, (b) an equimolar mixture of macrocycle **1** and **9** (5 mM), and (c-f) mixtures of **1**, NaTFPB, and **9** at concentrations of (c) 5/5/5 mM, (d) 5/10/10 mM, (e) 5/15/15 mM, (f) 5/20/20 mM.



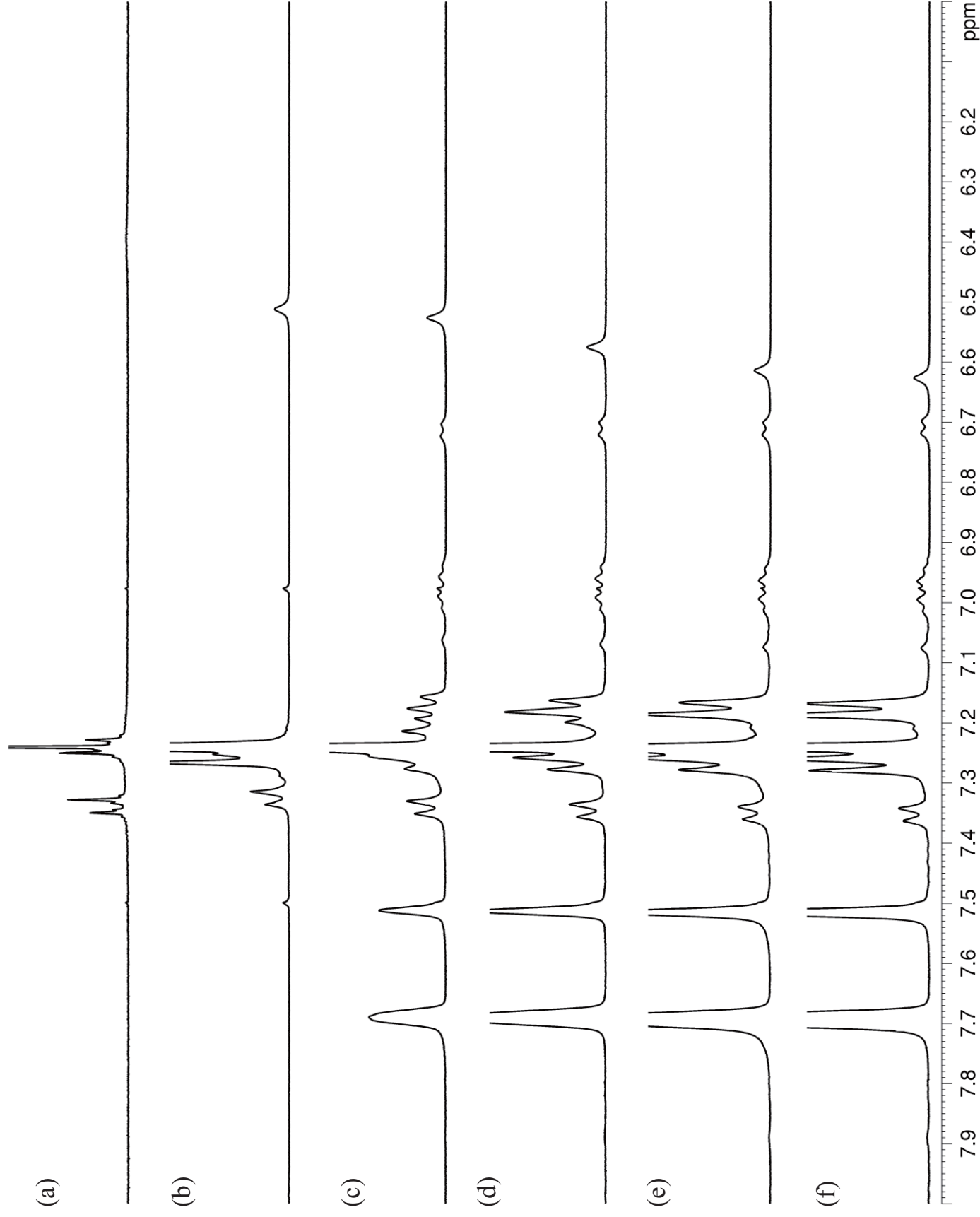
Partial ^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) urea **9**, (b) an equimolar mixture of macrocycle **2** and **9** (5 mM), and (c-f) mixtures of **2**, NaTFPB, and **9** at concentrations of (c) 5/5/5 mM, (d) 5/10/10 mM, (e) 5/15/15 mM, (f) 5/20/20 mM.



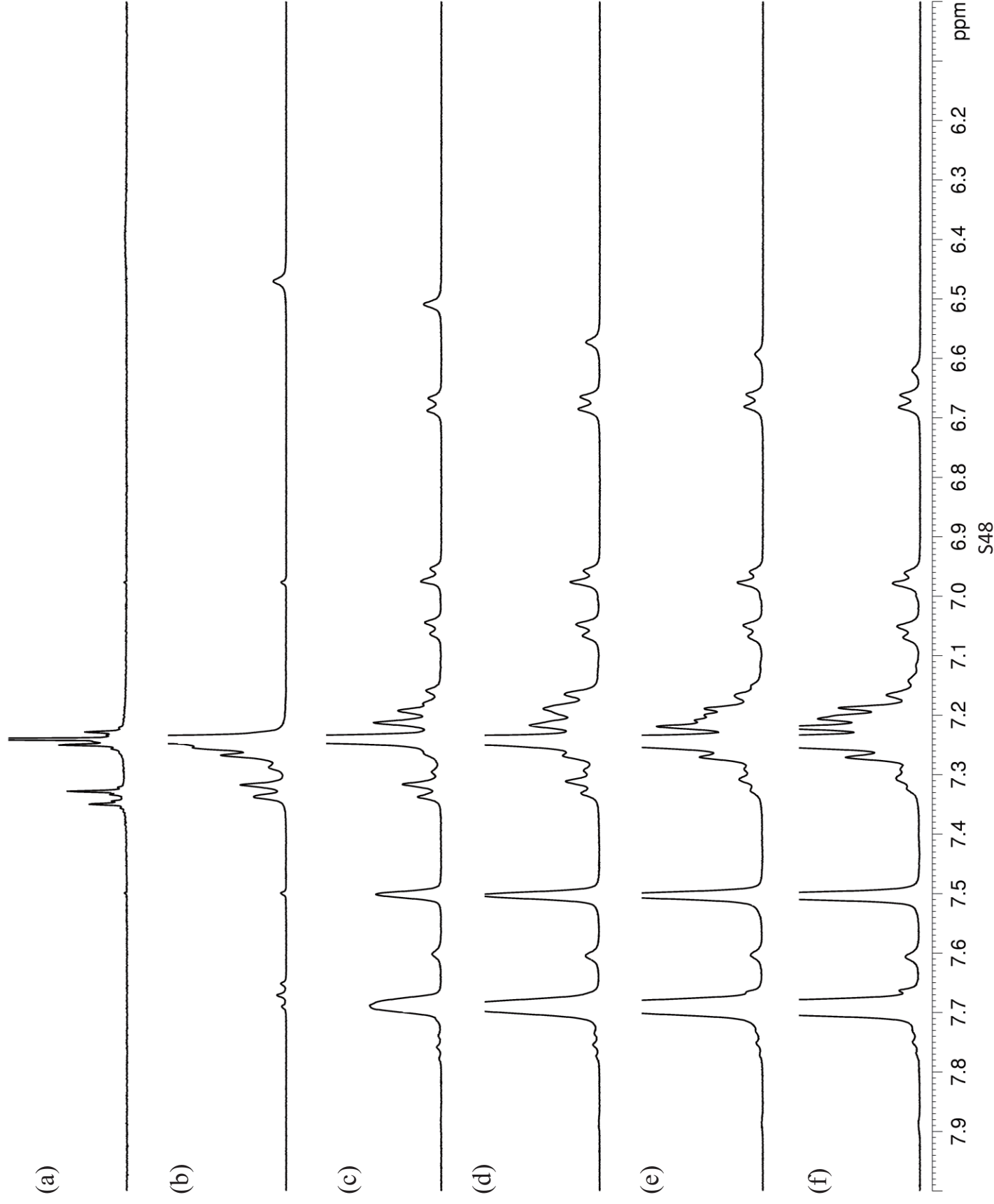
Partial ^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) urea **9**, (b) an equimolar mixture of macrocycle **3** and **9** (5 mM), and (c-f) mixtures of **3**, NaTFPB, and **9** at concentrations of (c) 5/5/5 mM, (d) 5/10/10 mM, (e) 5/15/15 mM, (f) 5/20/20 mM.



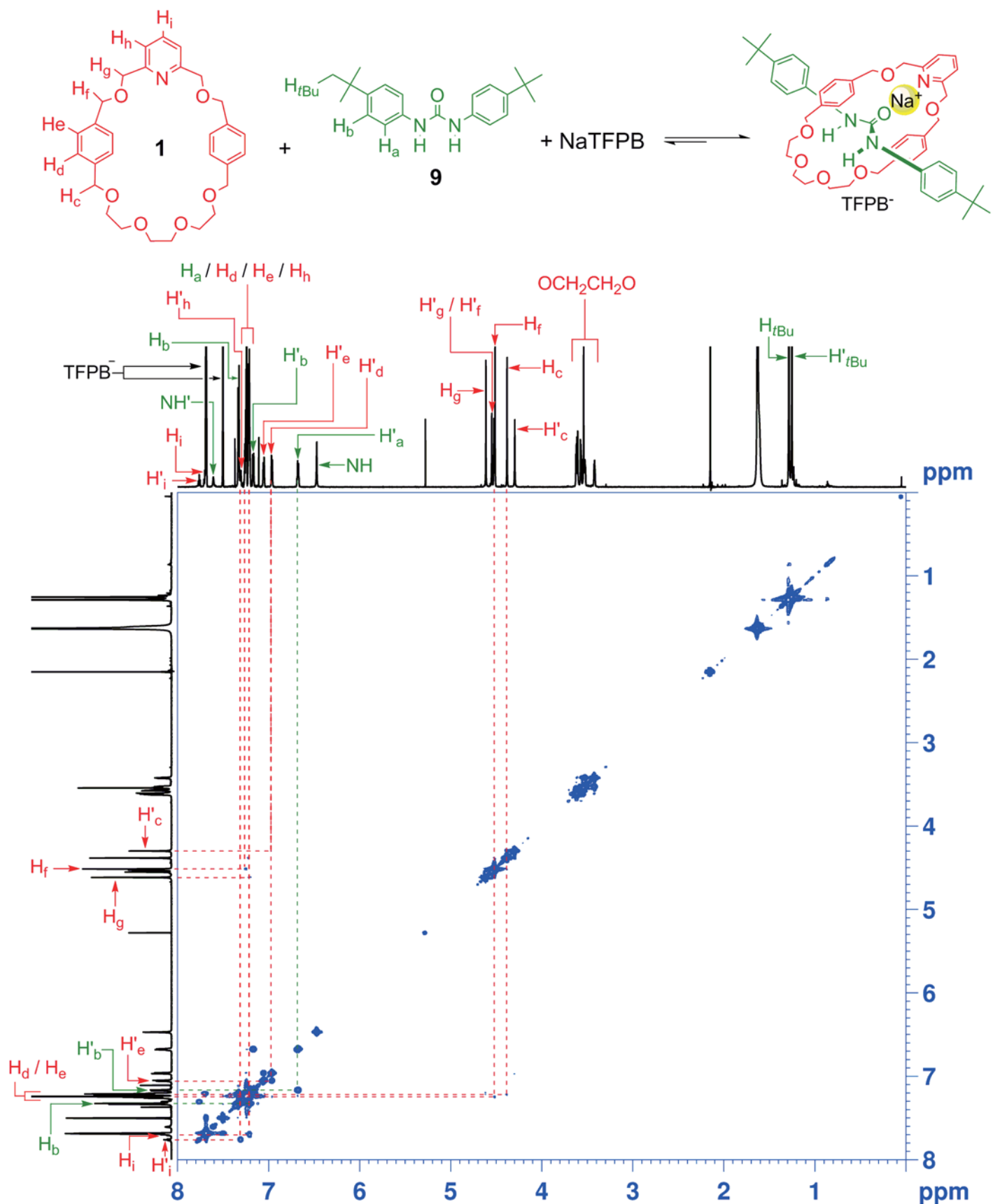
Partial ^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) urea **9**, (b) an equimolar mixture of macrocycle **4** and **9** (5 mM), and (c-f) mixtures of **4**, NaTFPB, and **9** at concentrations of (c) 5/5/5 mM, (d) 5/10/10 mM, (e) 5/15/15 mM, (f) 5/20/20 mM.



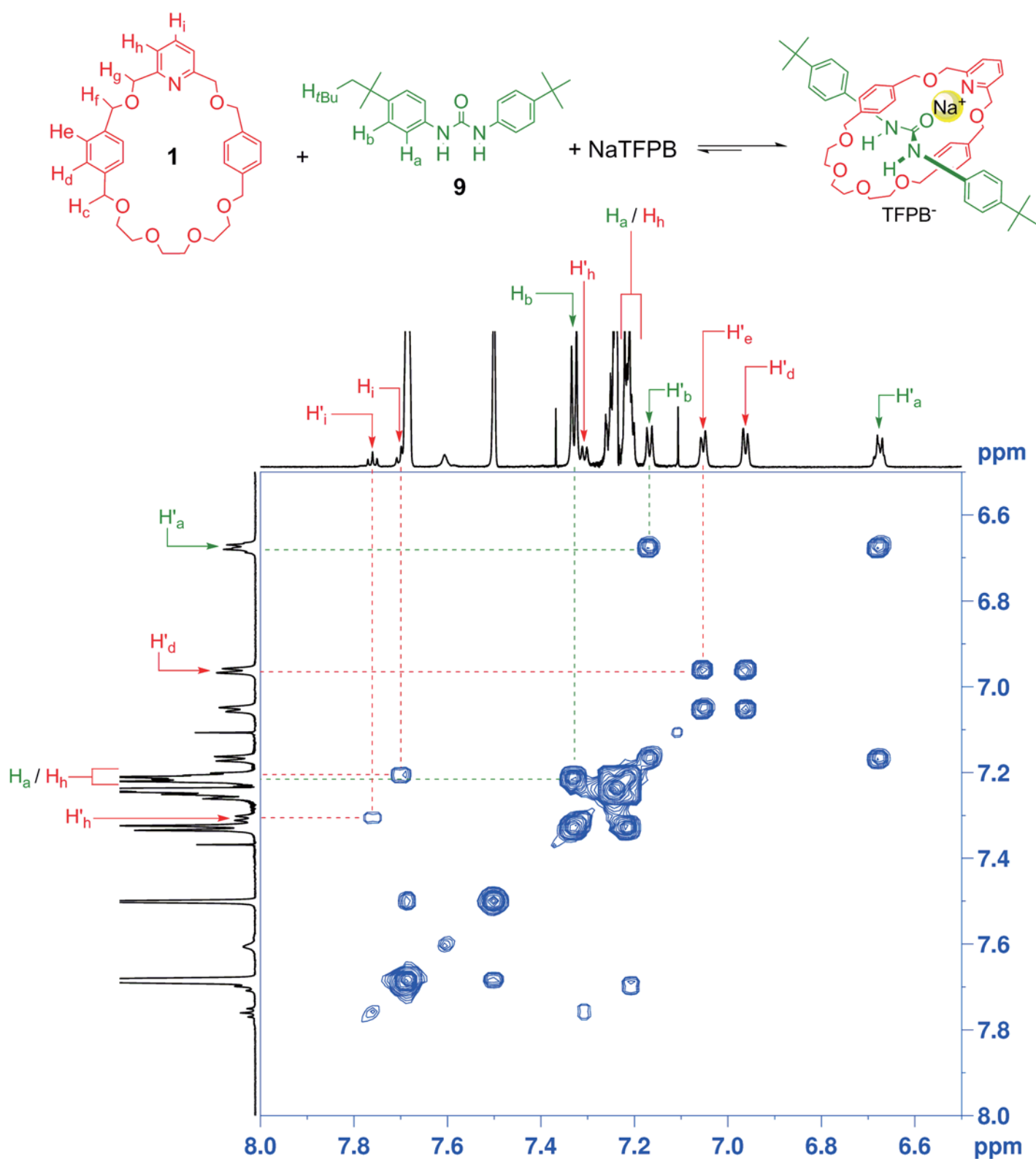
Partial ^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) urea **9**, (b) an equimolar mixture of macrocycle **5** and **9** (5 mM), and (c-f) mixtures of **5**, NaTFPB, and **9** at concentrations of (c) 5/5/5 mM, (d) 5/10/10 mM, (e) 5/15/15 mM, (f) 5/20/20 mM.



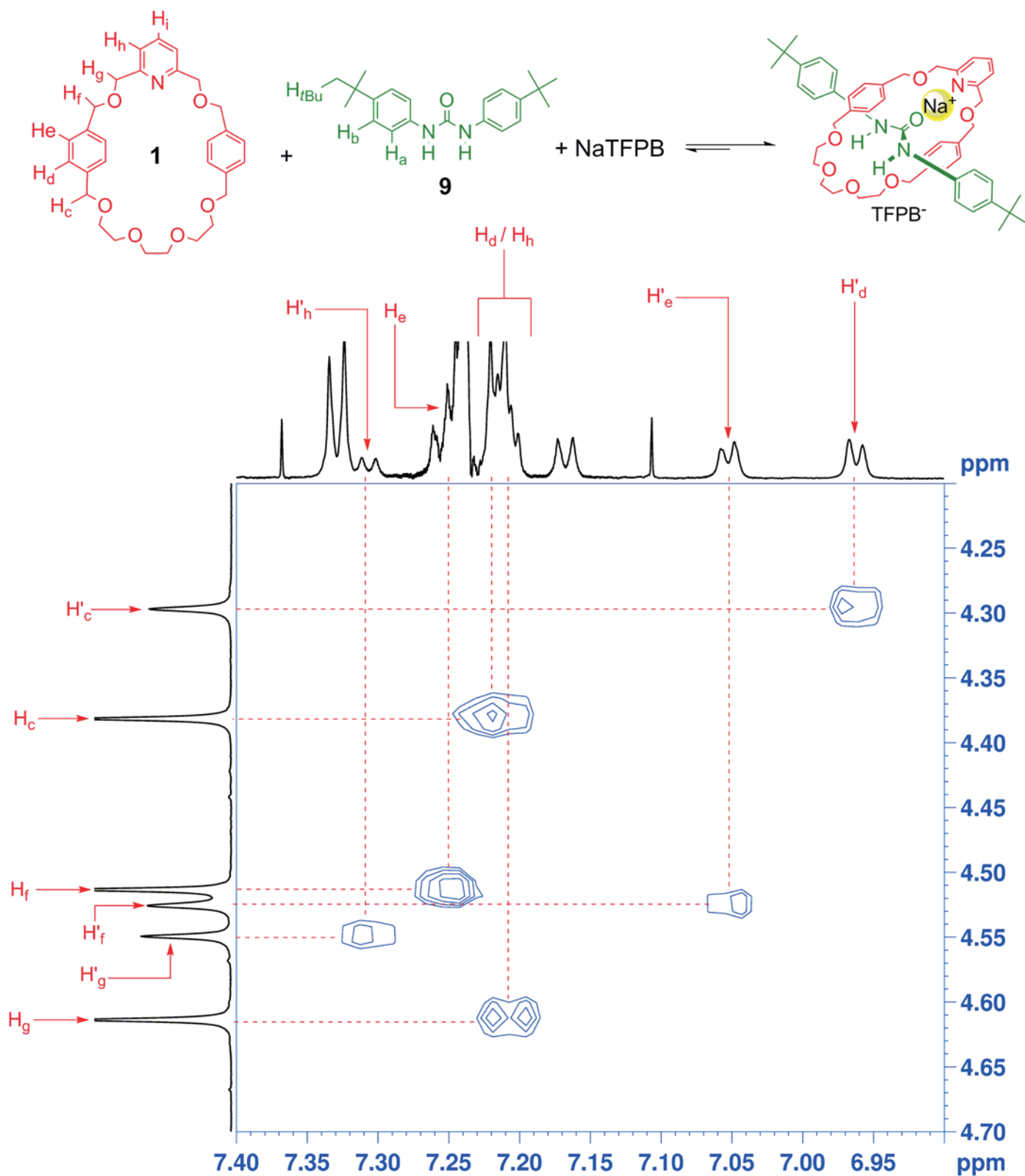
COSY Spectrum (800 MHz, CDCl₃, 298 K, 5mM)



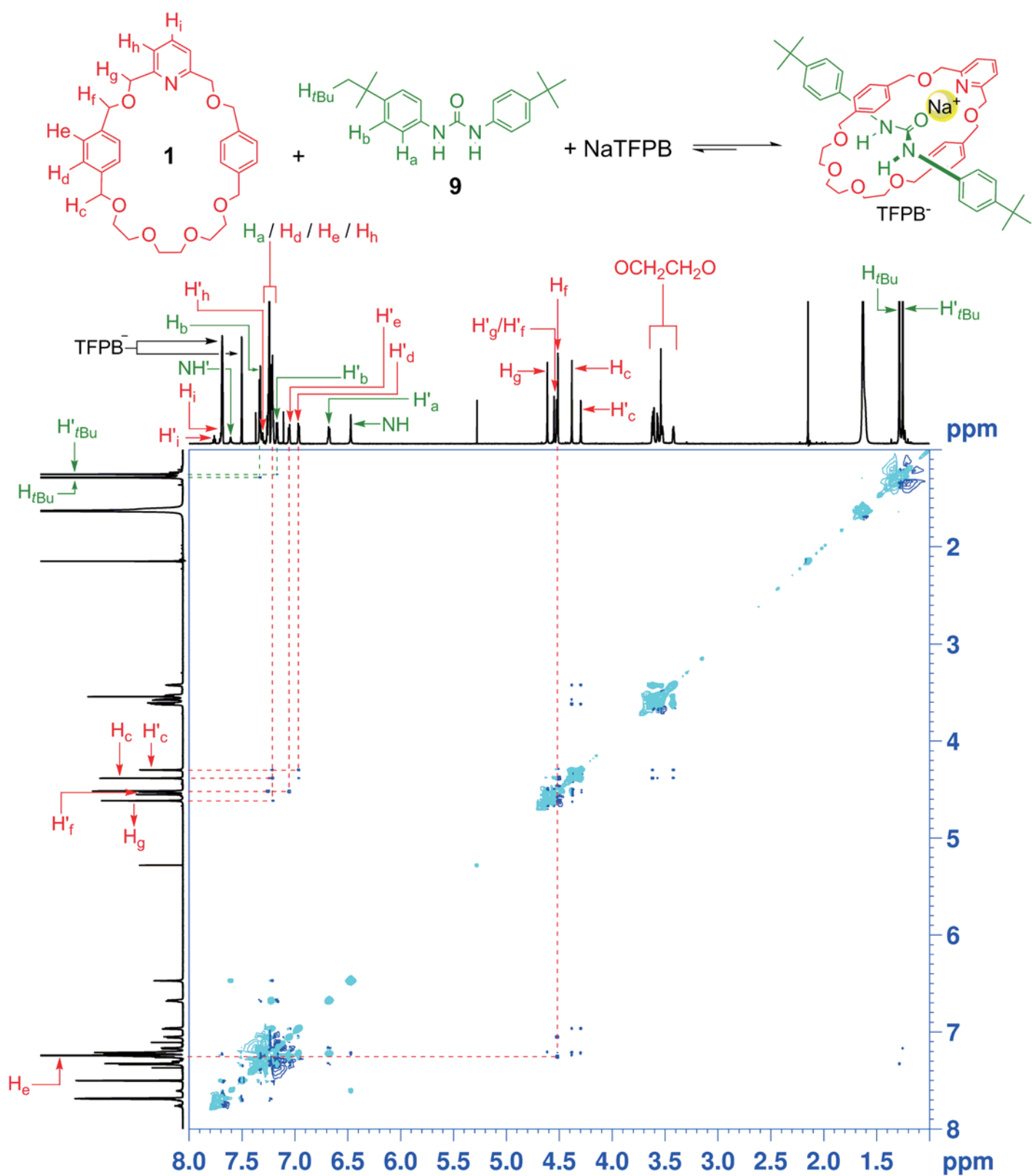
Partial COSY Spectrum (800 MHz, CDCl₃, 298 K, 5mM)



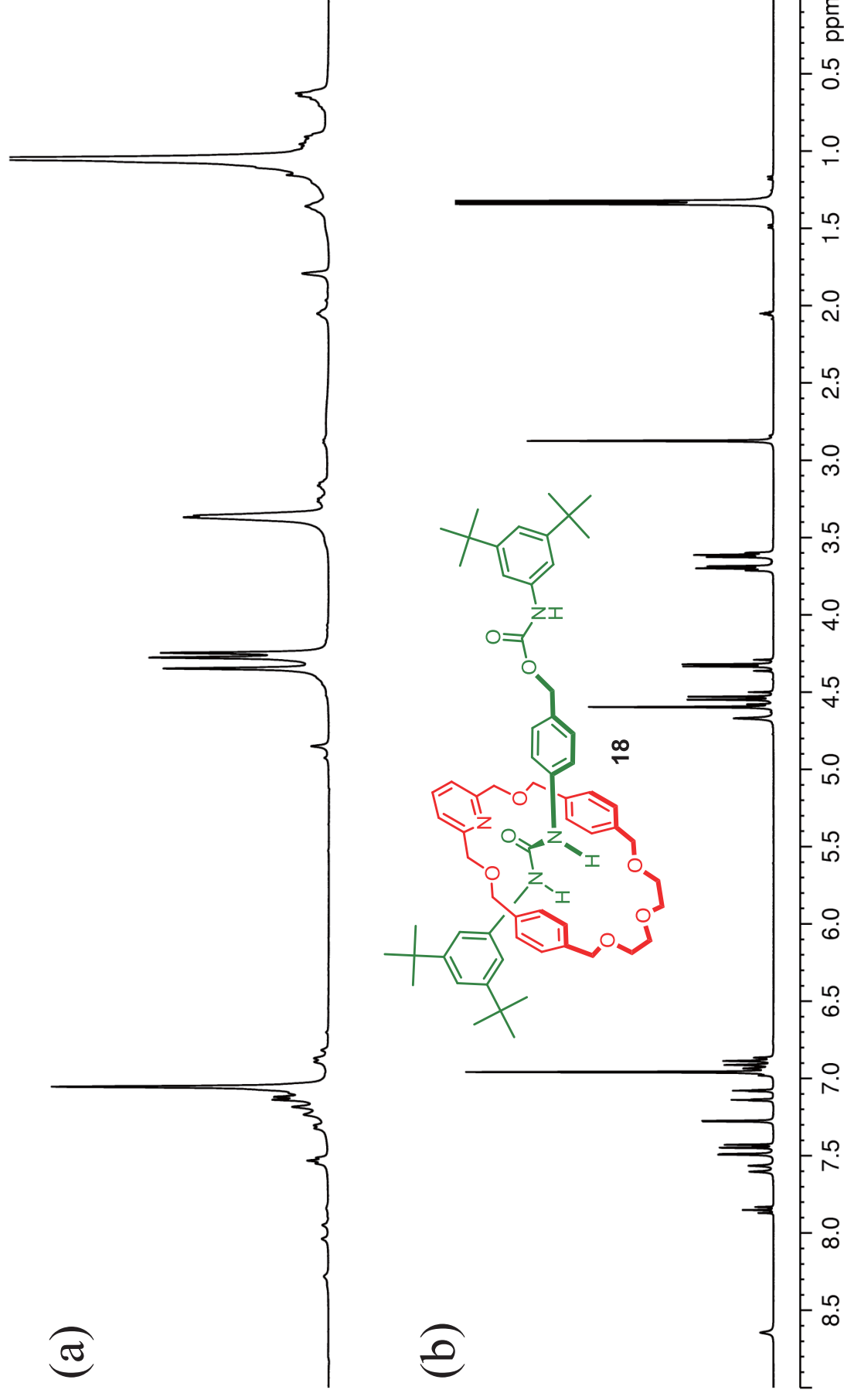
Partial COSY Spectrum (800 MHz, CDCl₃, 298 K, 5mM)



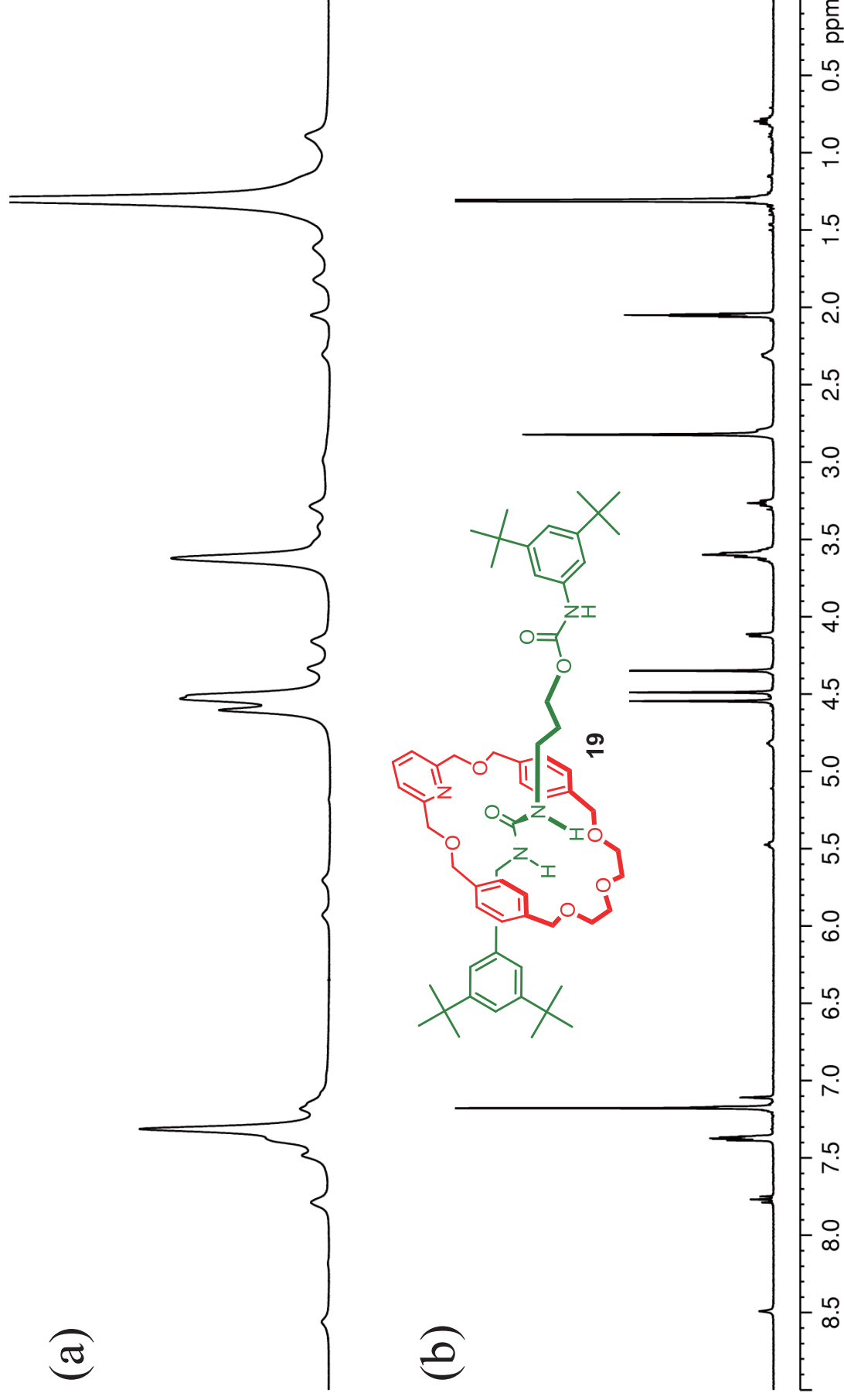
ROESY Spectrum (800 MHz, CDCl₃, 298 K, 5mM)



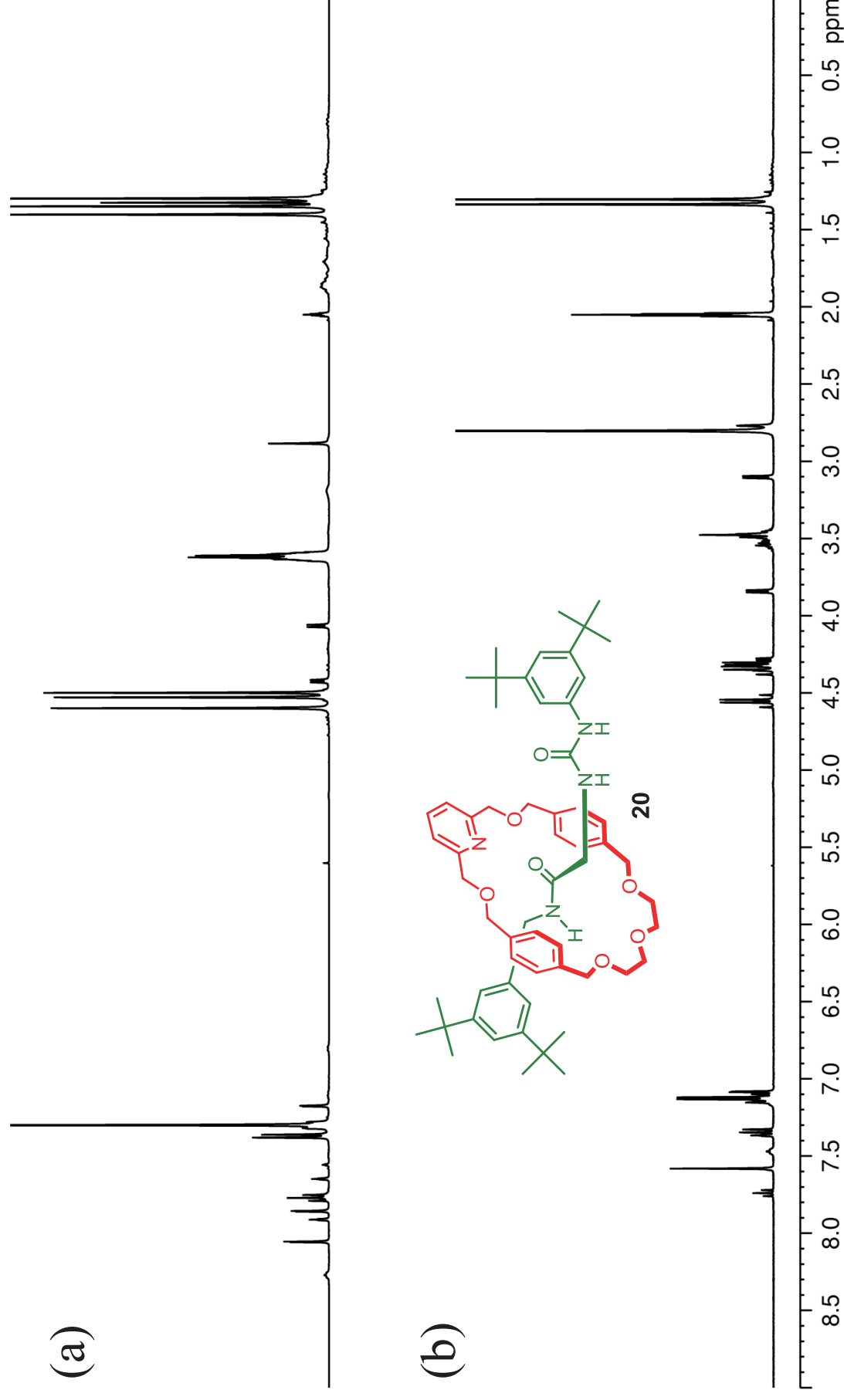
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **10**, macrocycle **1**, and stopper **13** and (b) purified [2]rotaxane **18**



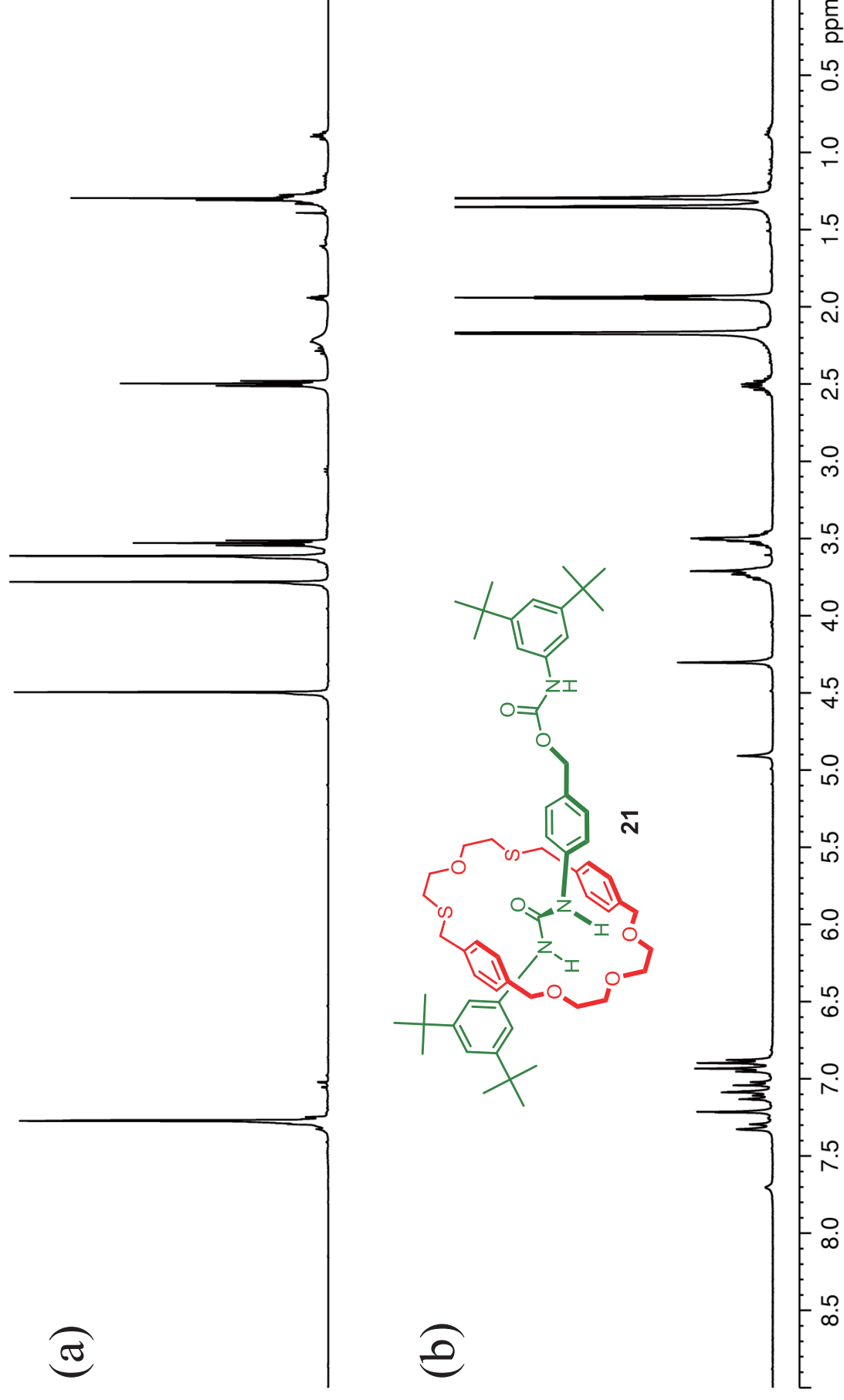
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **11**, macrocycle **1**, and stopper **13** and (b) purified [2]rotaxane **19**



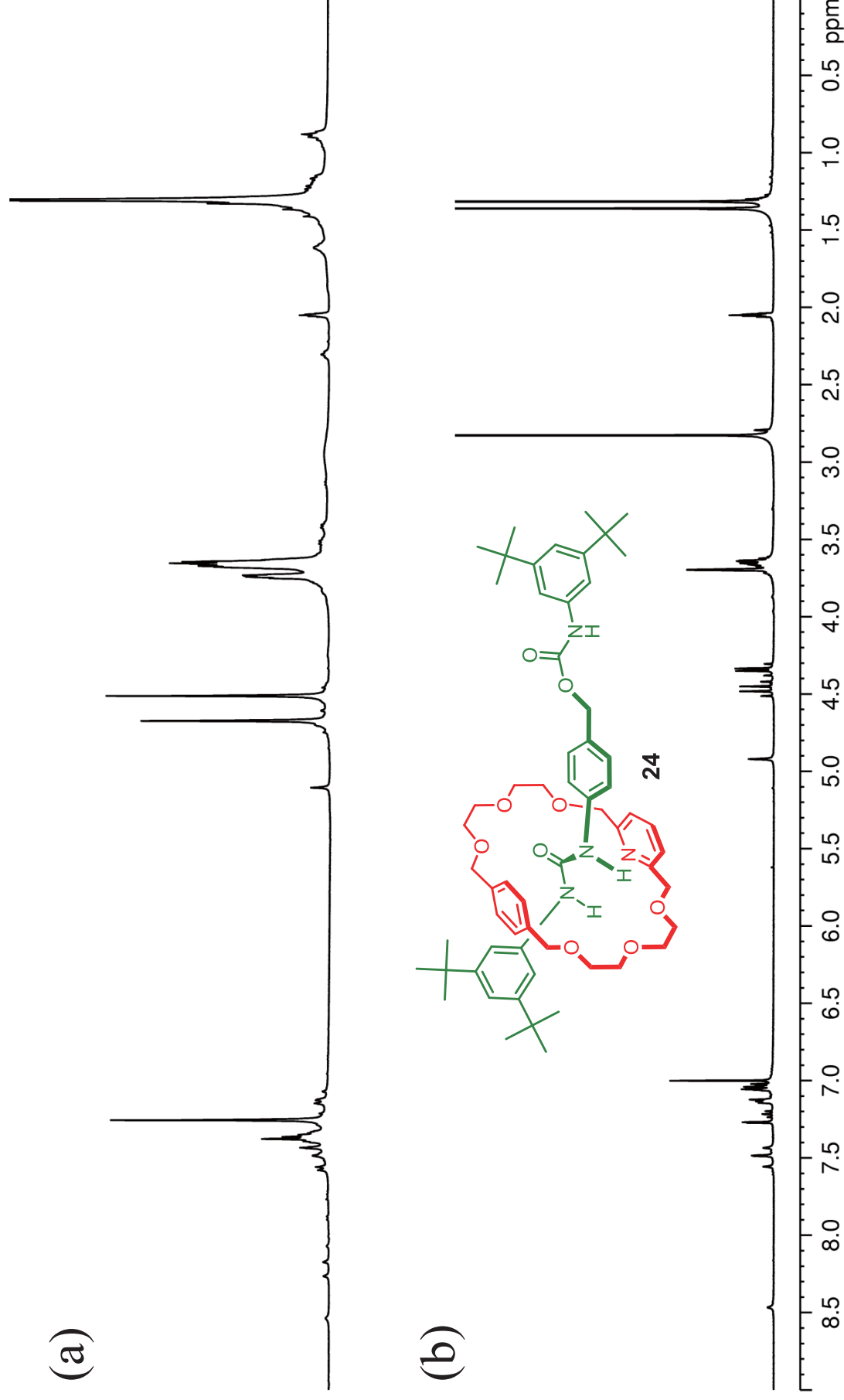
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **12**, macrocycle **1**, and stopper **14** and (b) purified [2]rotaxane **20**



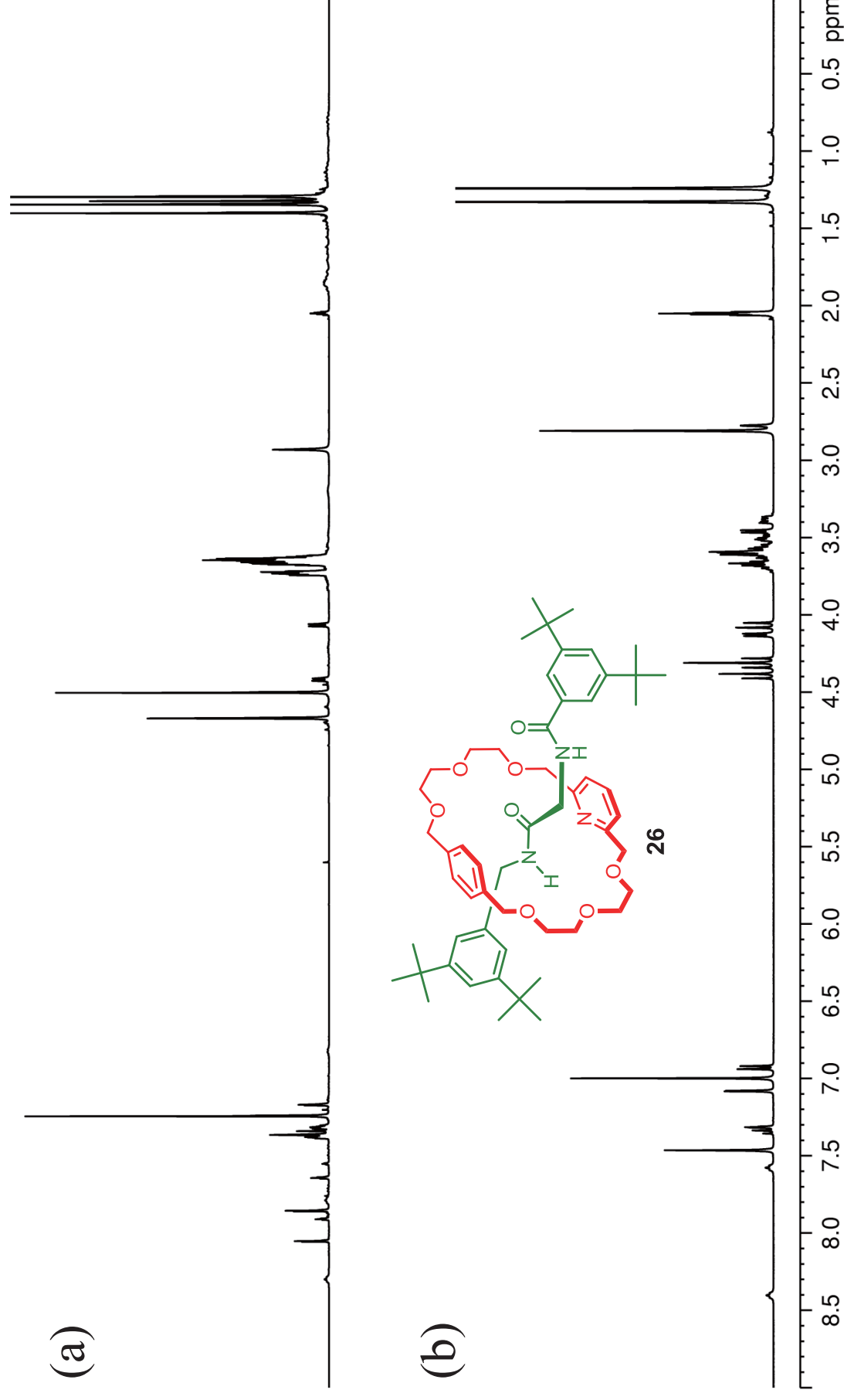
^1H NMR spectra (400 MHz, CD_3CN , 298 K) of (a) crude products obtained from the mixture of thread **10**, macrocycle **2**, and stopper **13** and (b) purified [2]rotaxane **21**



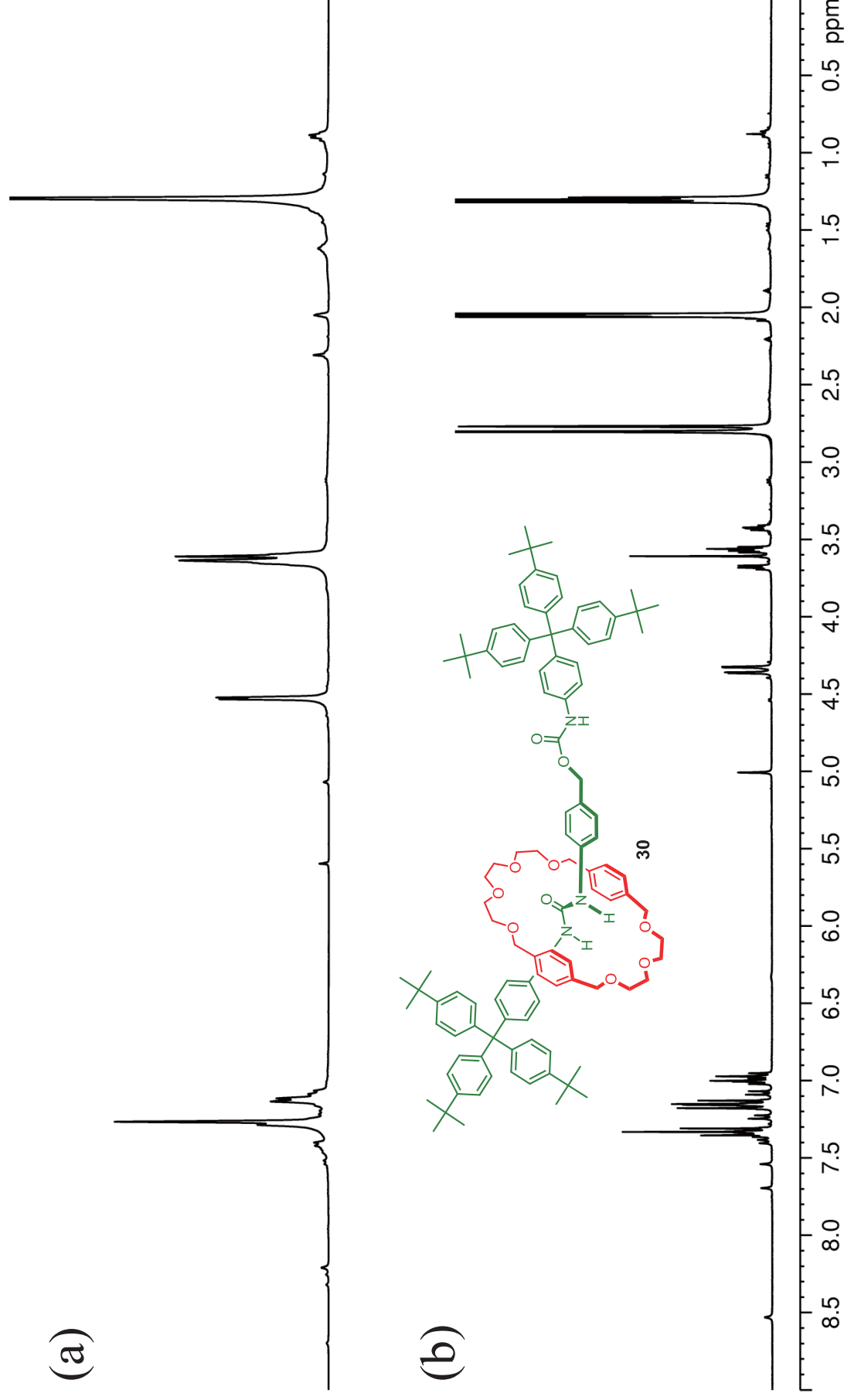
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **10**, macrocycle **3**, and stopper **13** and (b) purified [2]rotaxane **24**



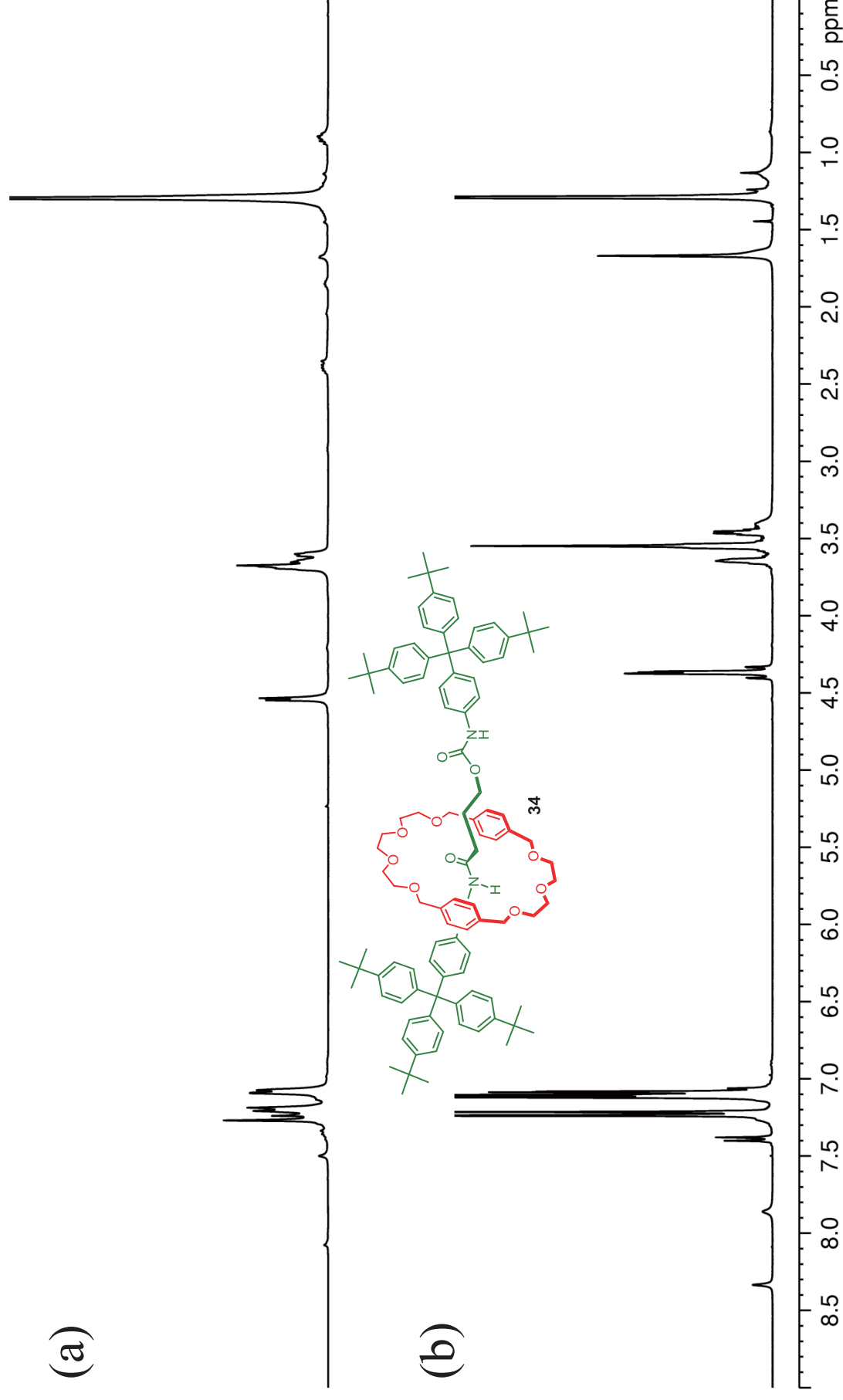
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **12**, macrocycle **3**, and stopper **14** and (b) purified [2]rotaxane **26**



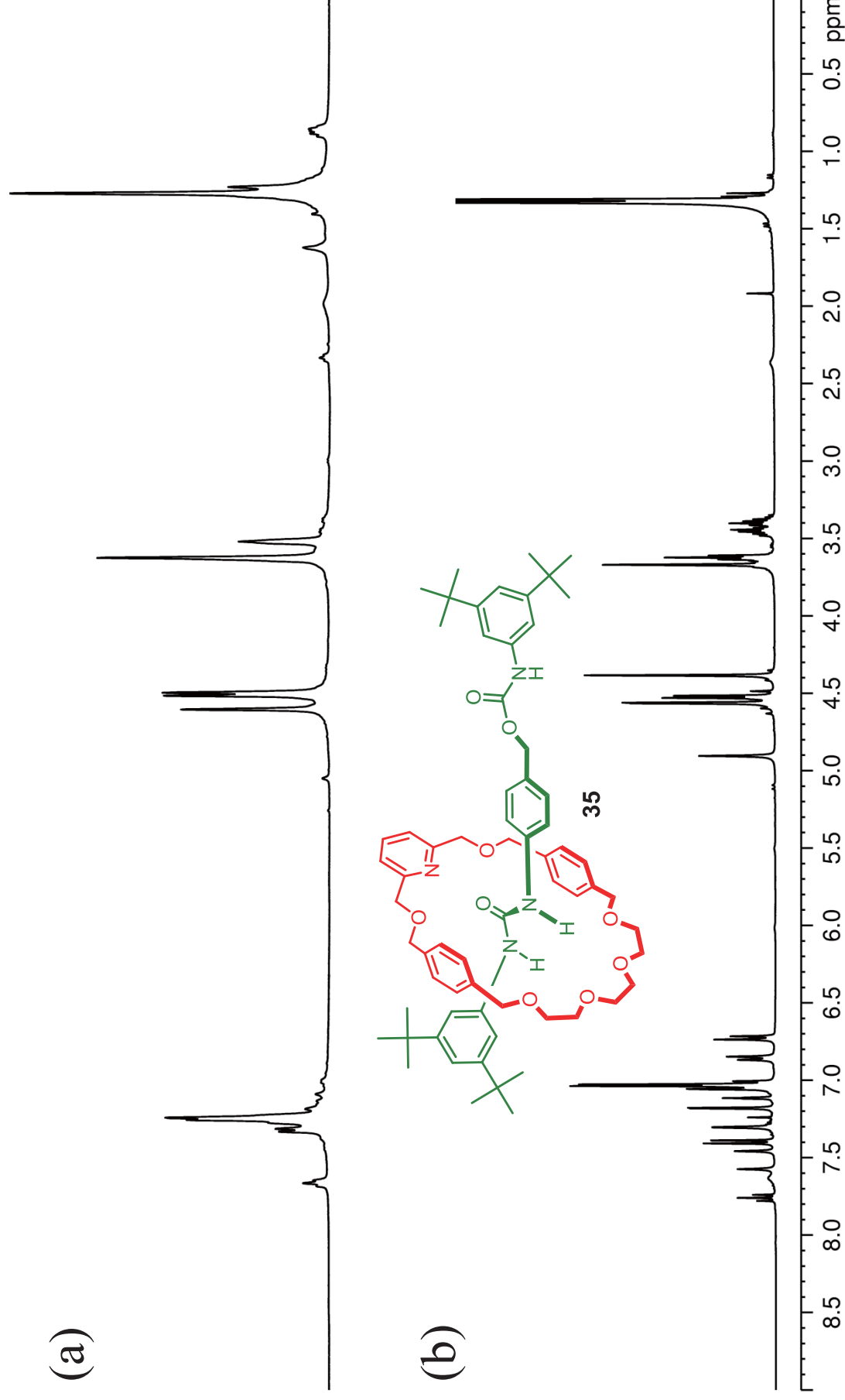
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **28**, macrocycle **4**, and stopper **29** and (b) purified [2]rotaxane **30**



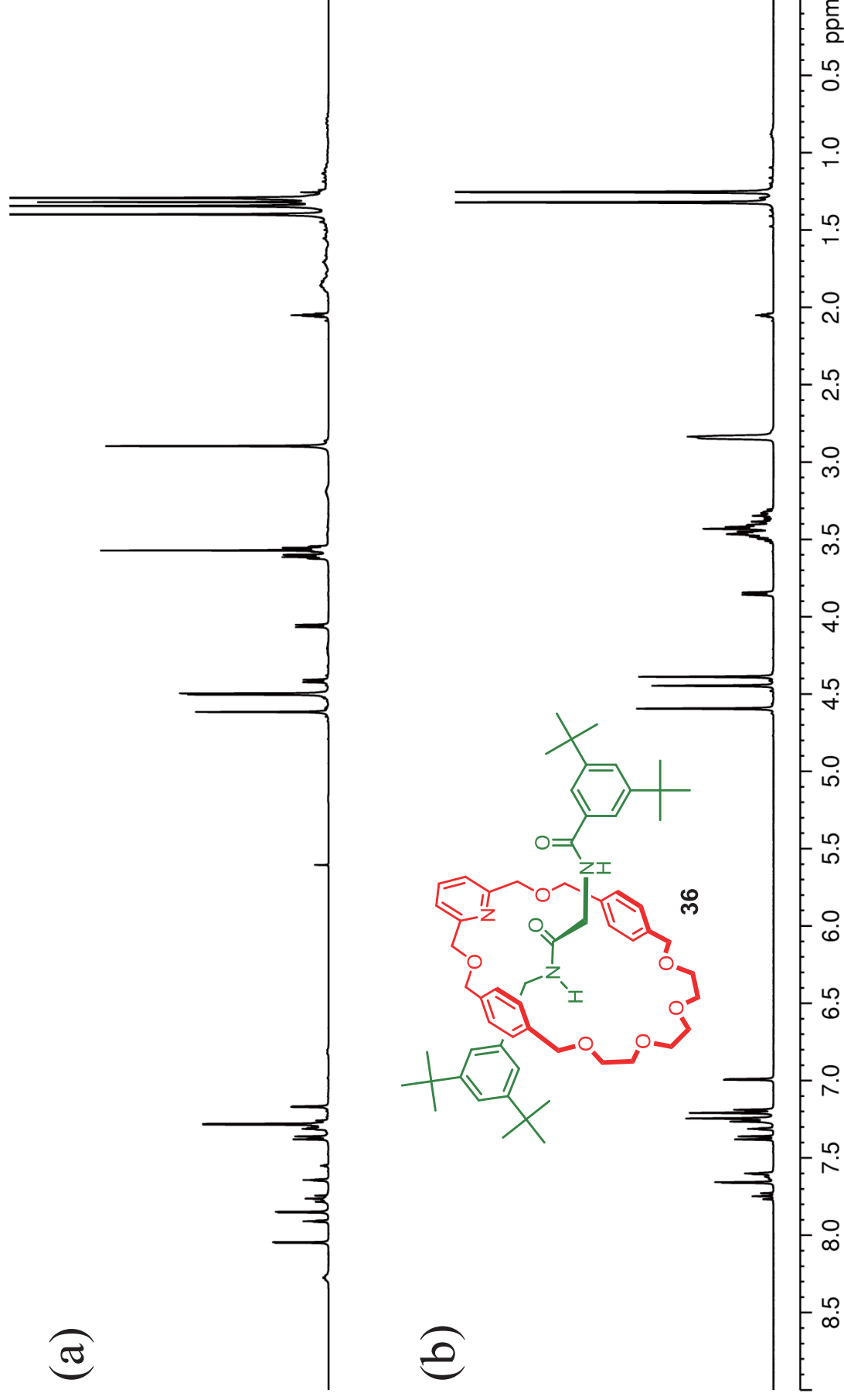
^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) crude products obtained from the mixture of thread **33**, macrocycle **4**, and stopper **29** and (b) purified [2]rotaxane **34**



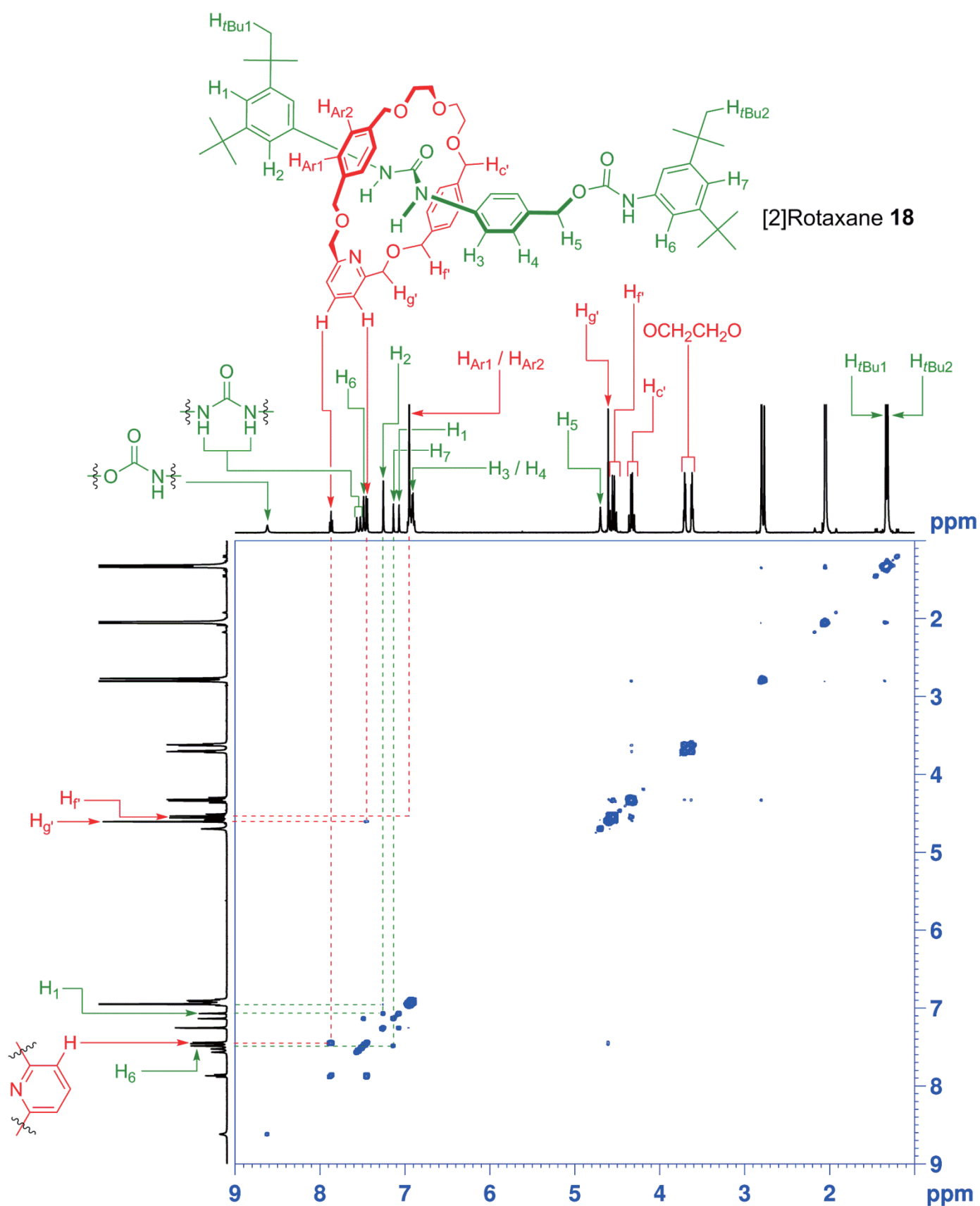
^1H NMR spectra (400 MHz, CDCl_3 , 298 K) of (a) crude products obtained from the mixture of thread **10**, macrocycle **5**, and stopper **13** and (b) purified [2]rotaxane **35**



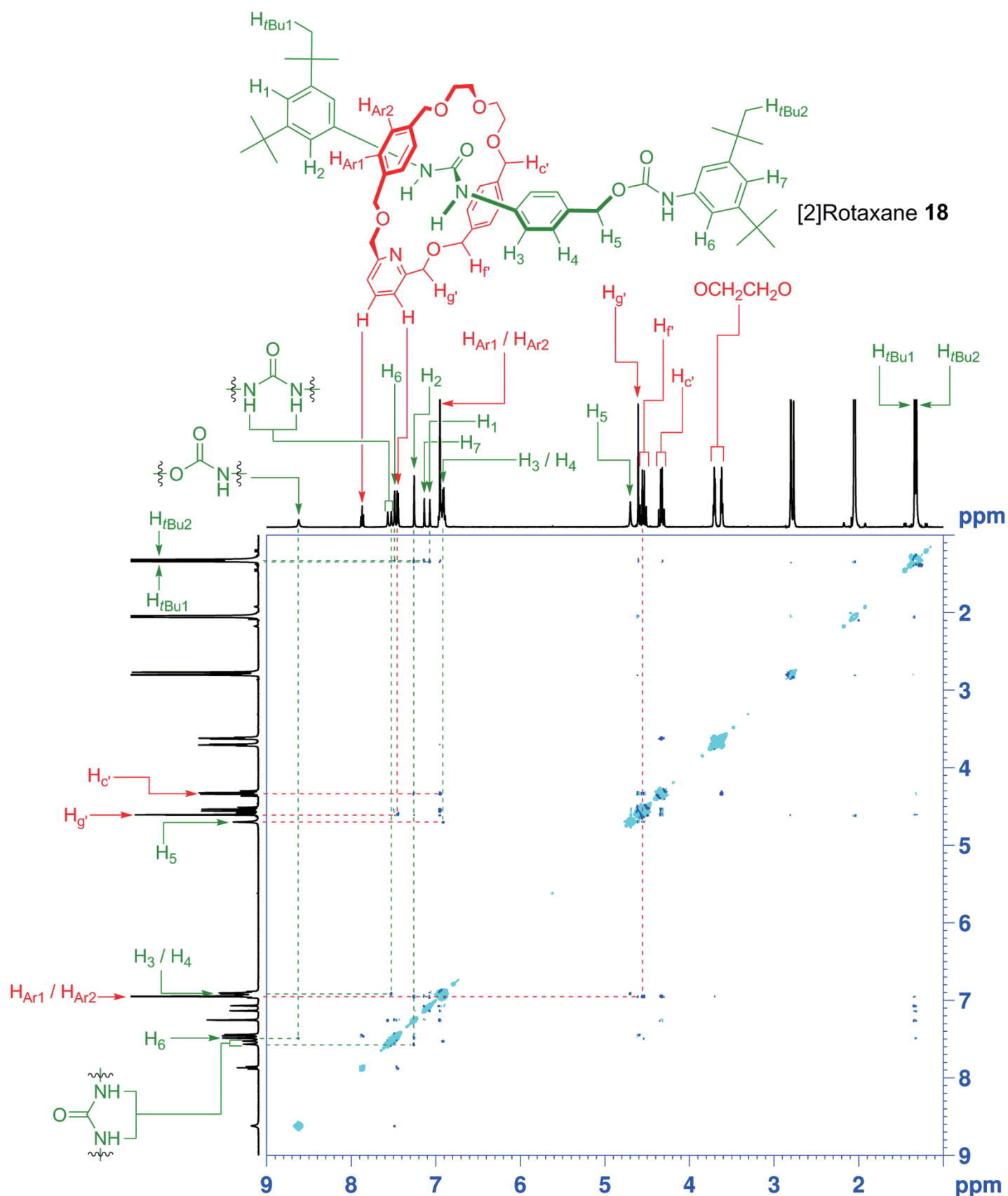
^1H NMR spectra (400 MHz, CD_3COCD_3 , 298 K) of (a) crude products obtained from the mixture of thread **12**, macrocycle **5**, and stopper **14** and (b) purified [2]rotaxane **36**



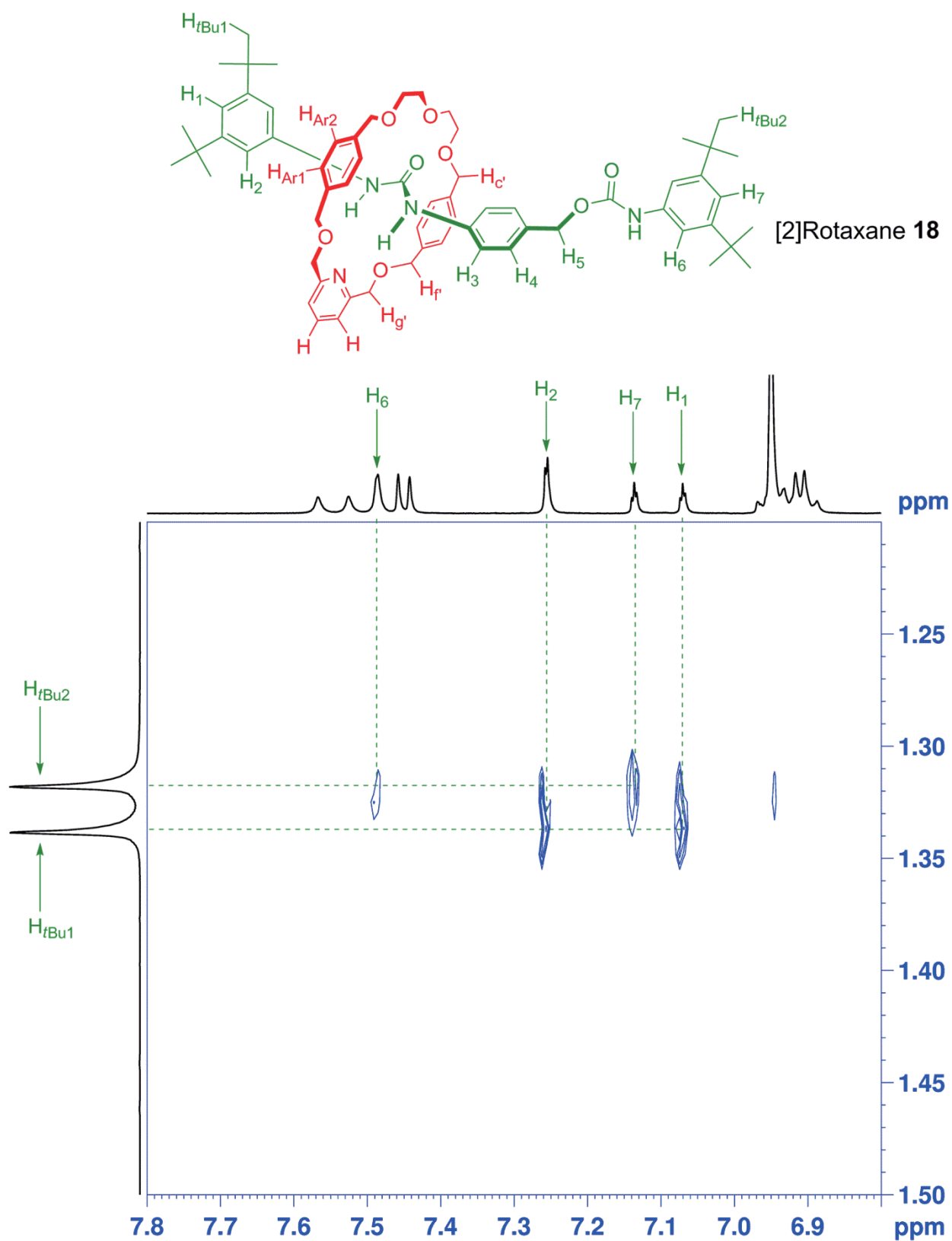
COSY Spectrum (500 MHz, CD₃COCD₃, 298K)



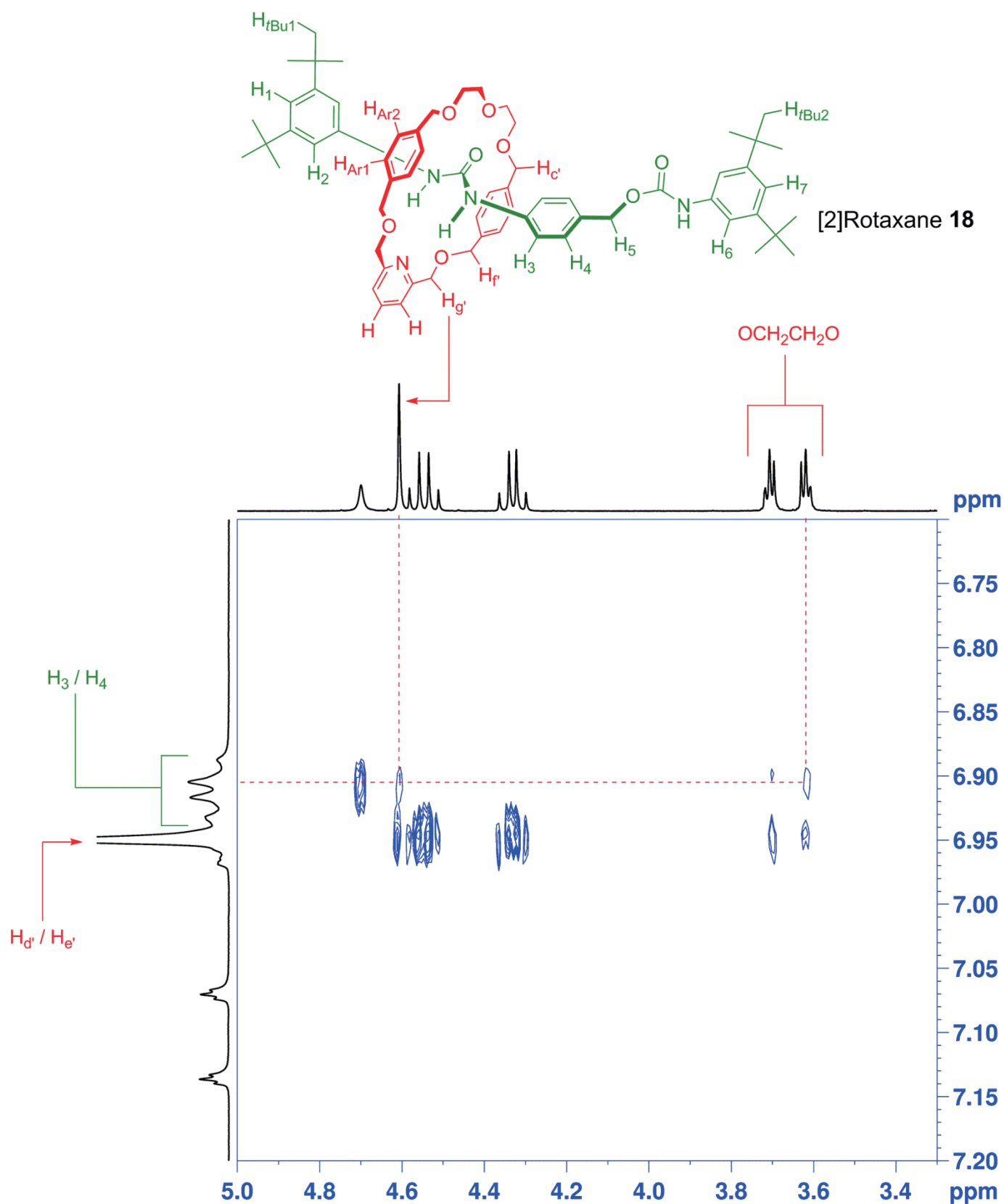
ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



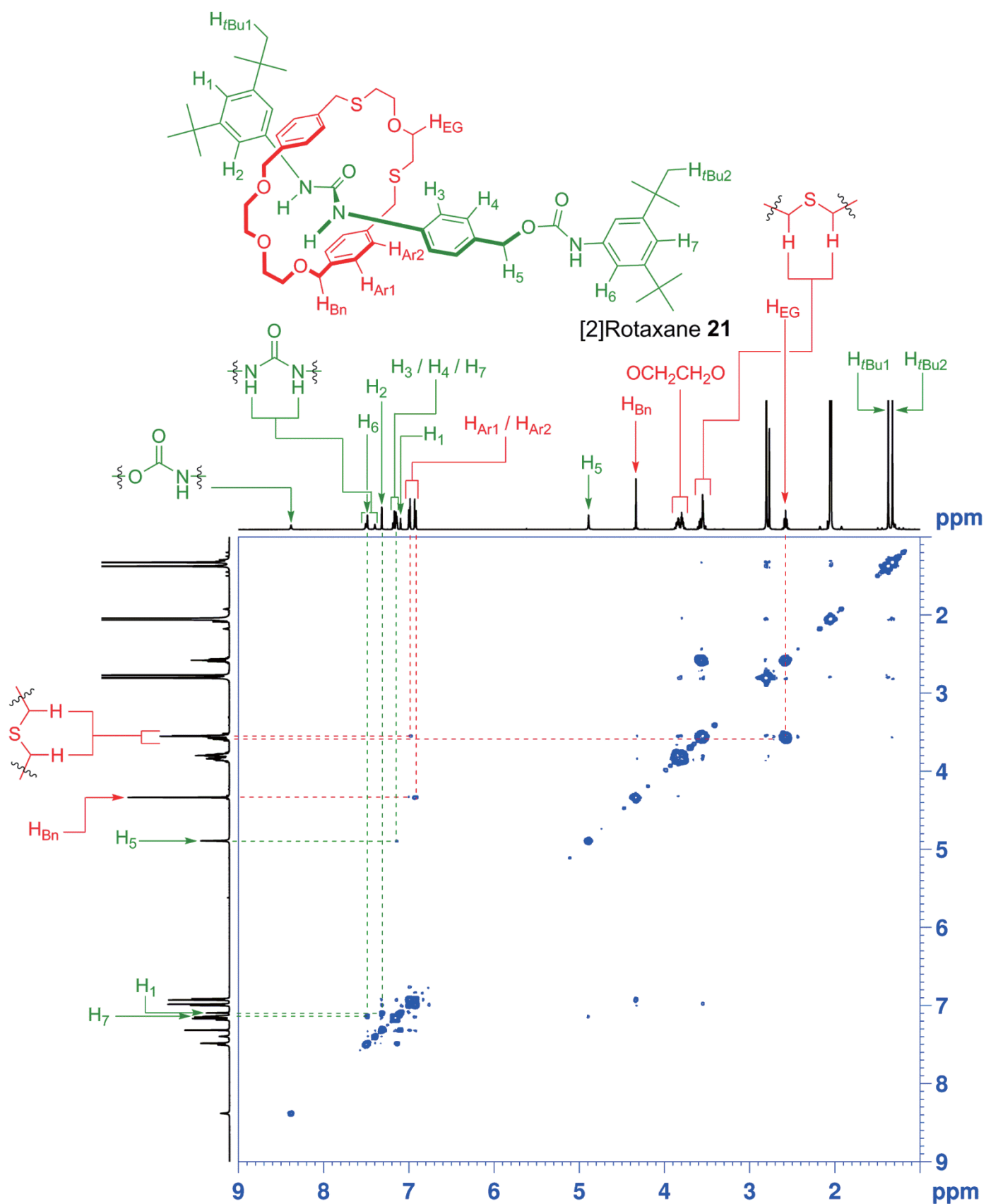
Partial ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



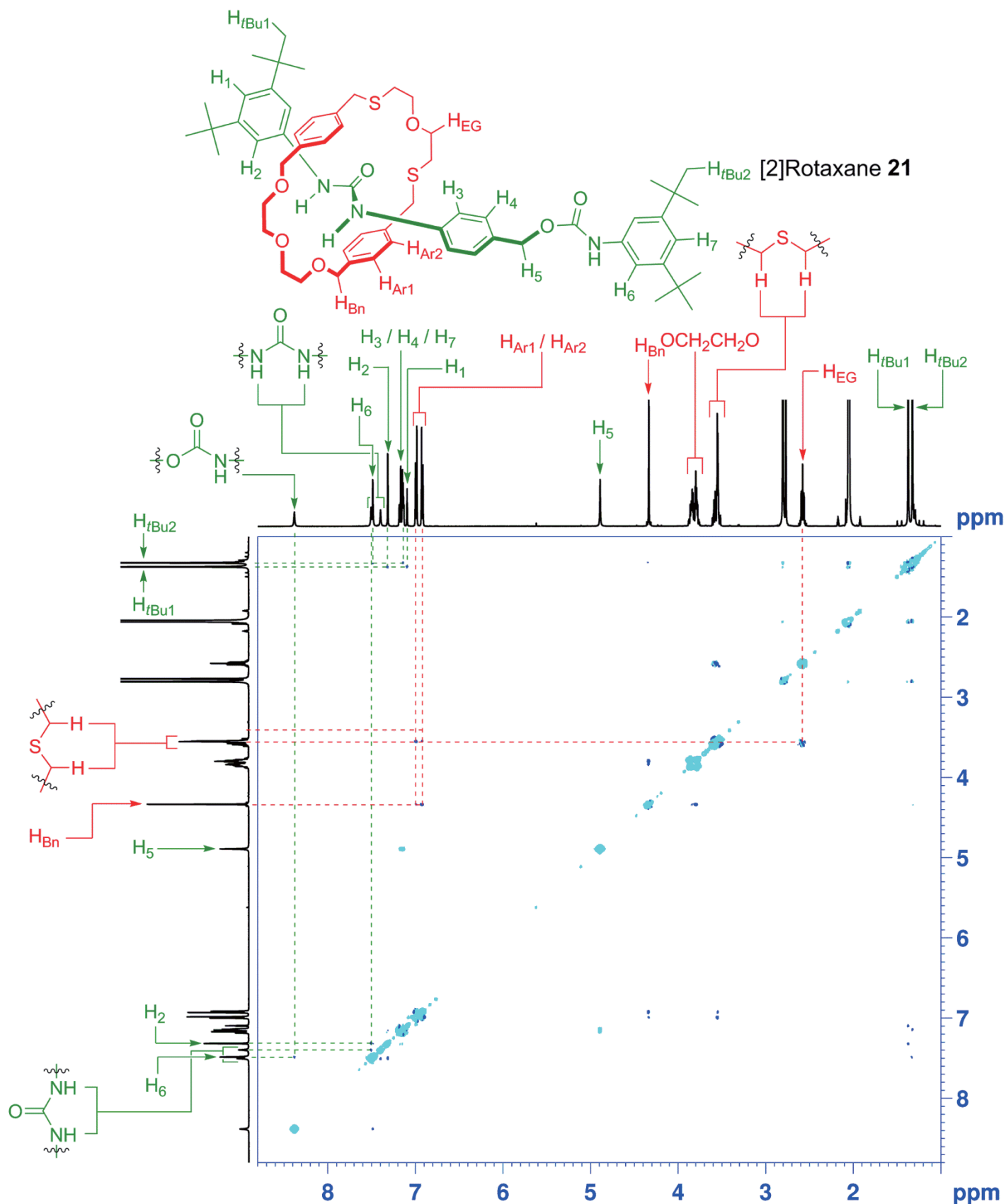
Partial ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



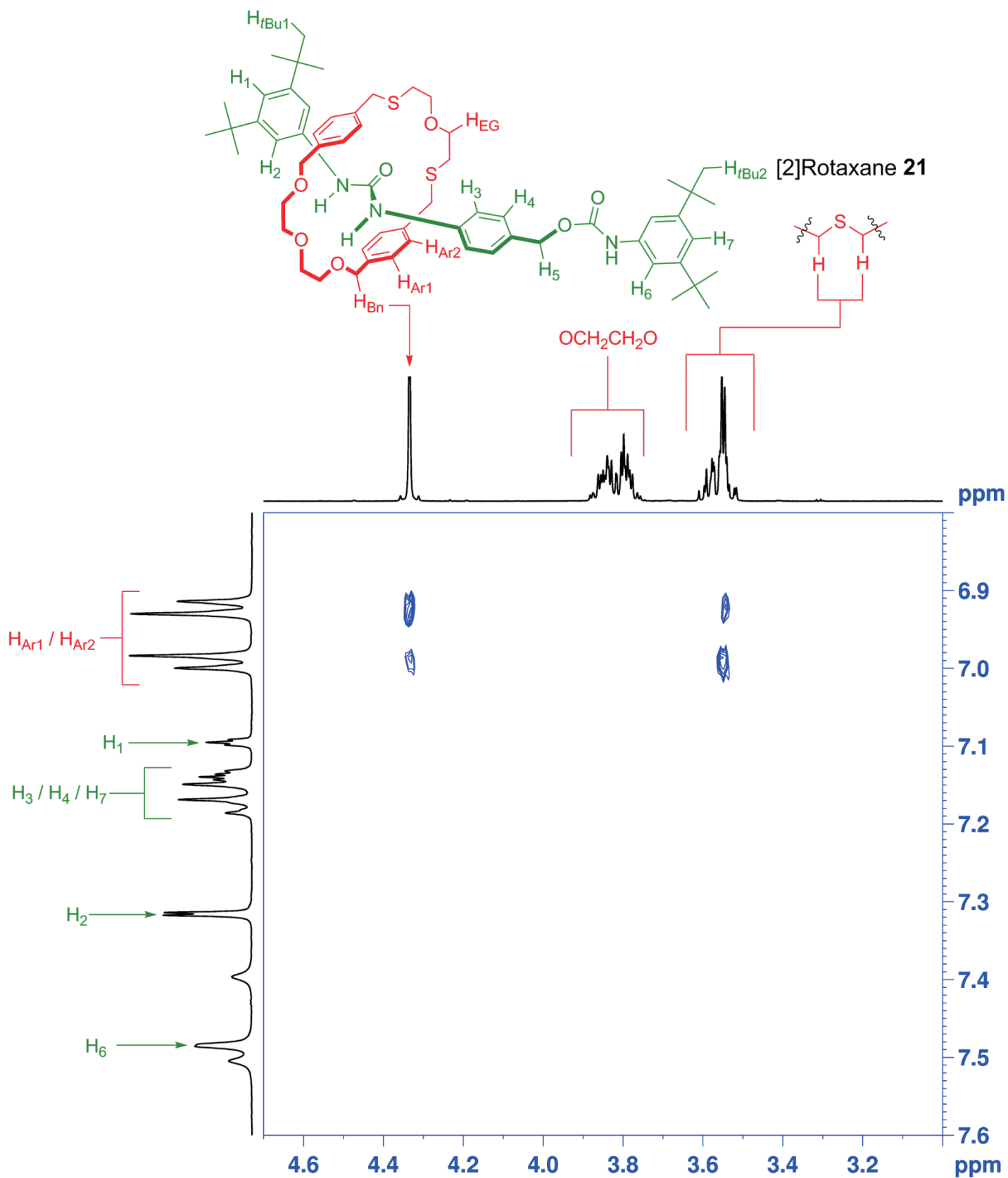
COSY Spectrum (500 MHz, CD₃COCD₃, 298K)



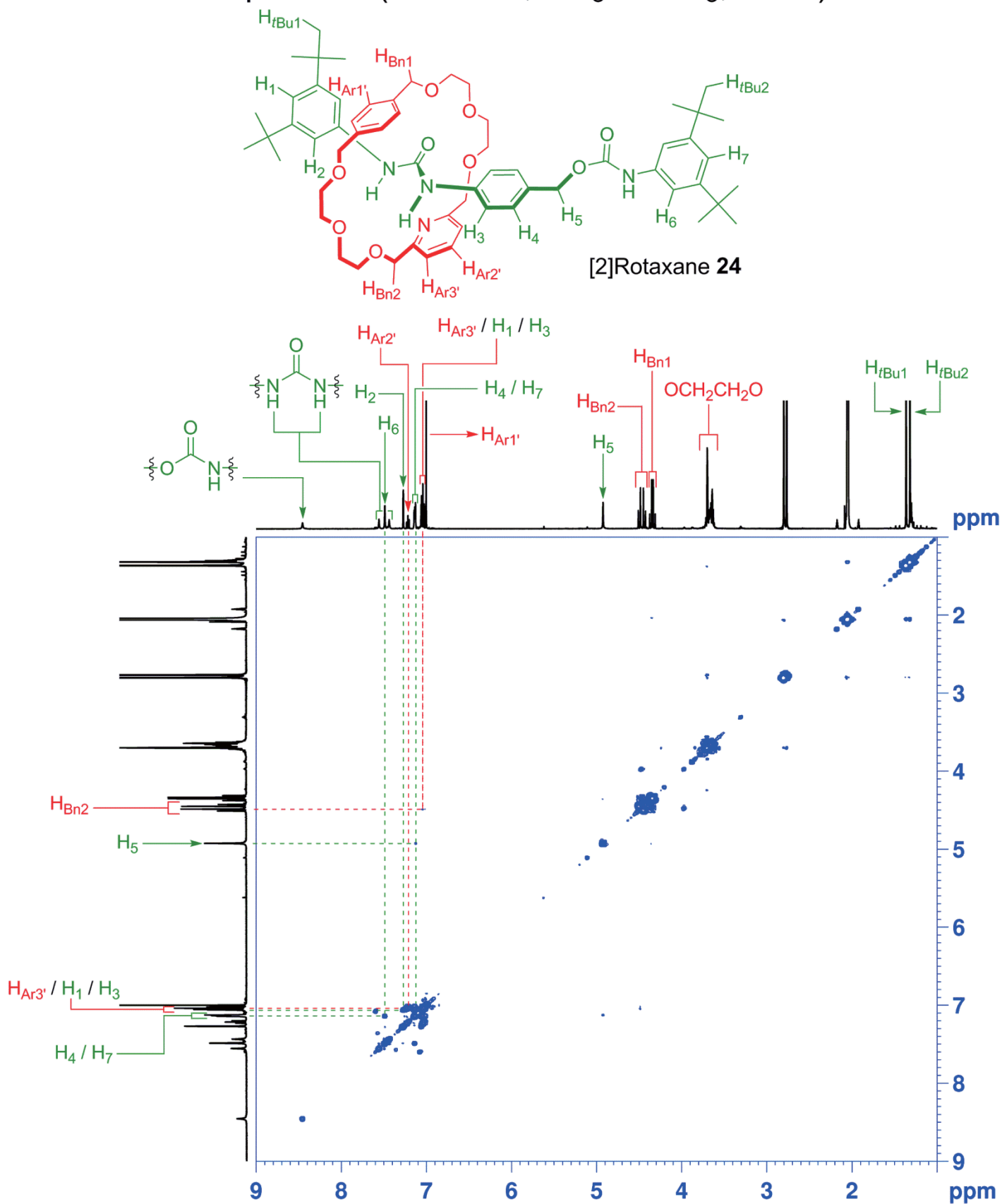
ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



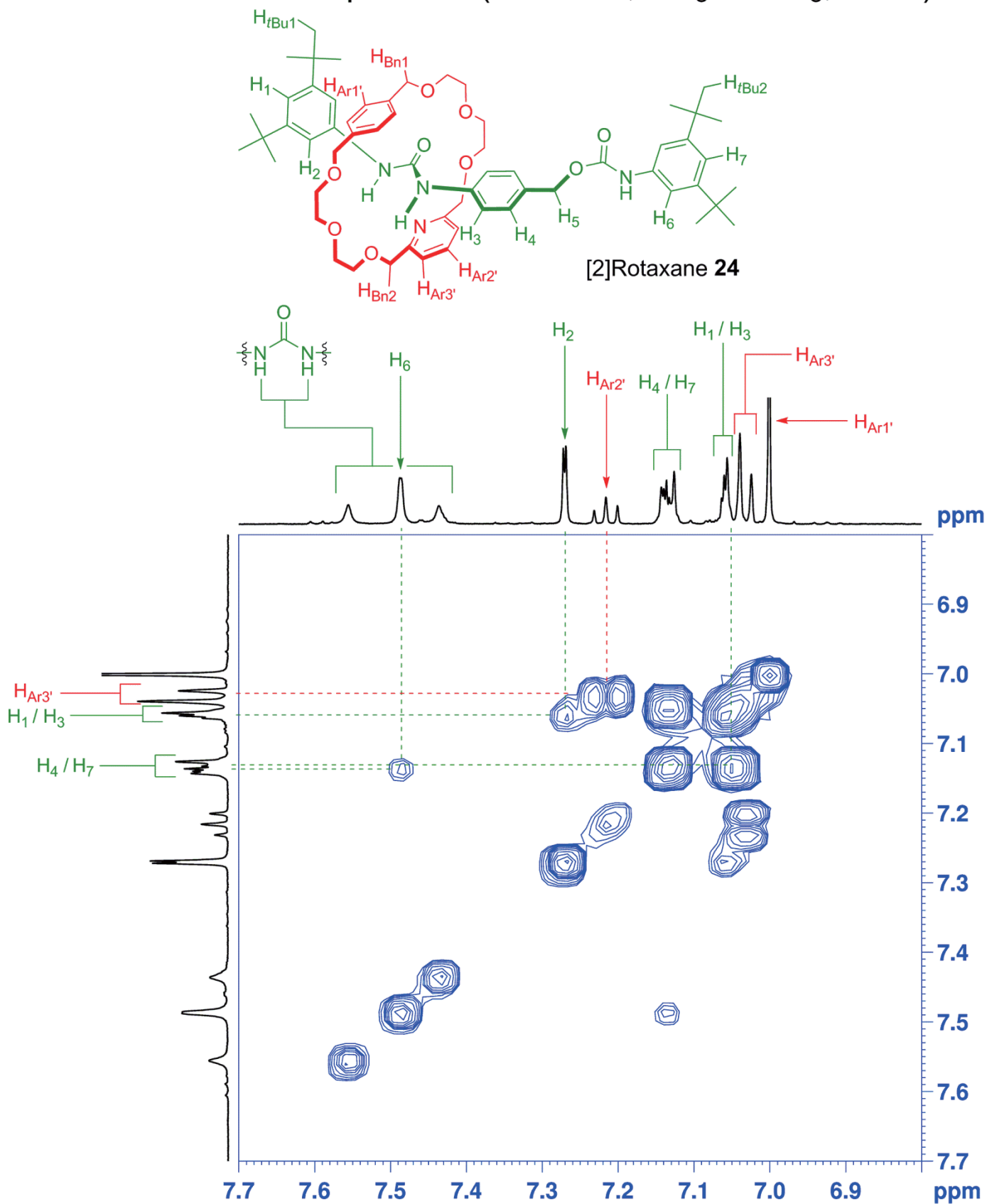
Partial ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



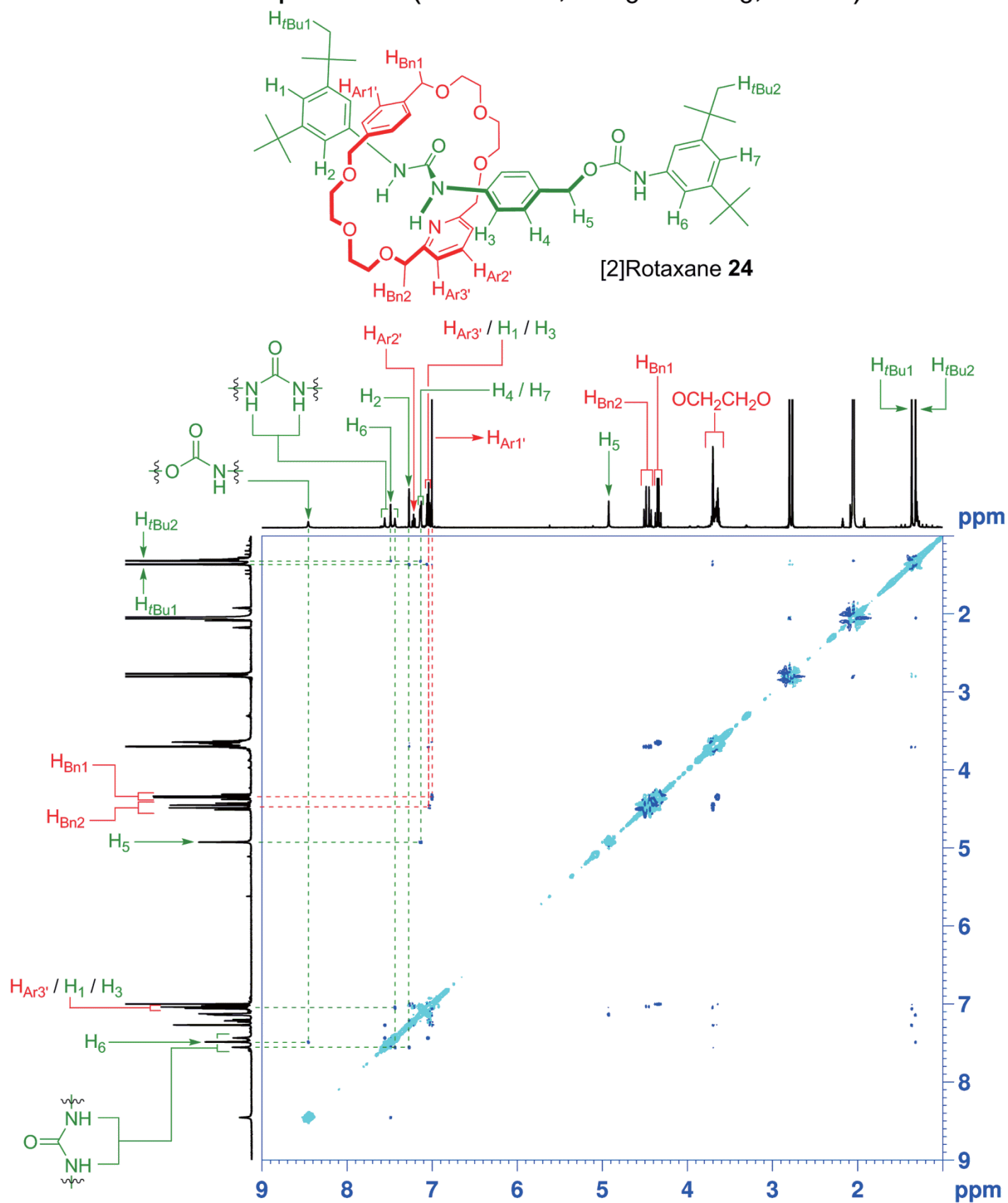
COSY Spectrum (500 MHz, CD₃COCD₃, 298K)



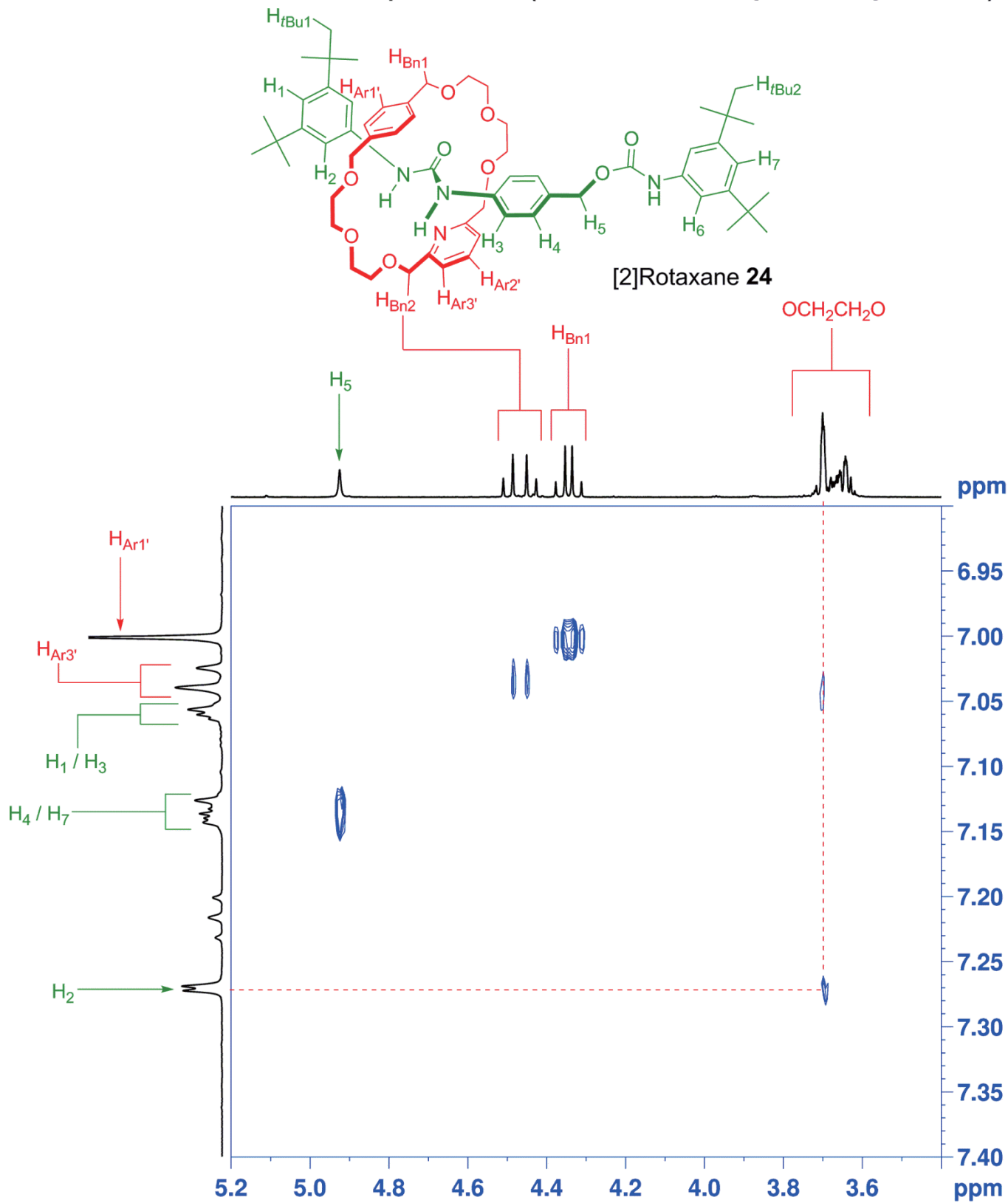
Partial COSY Spectrum (500 MHz, CD₃COCD₃, 298K)



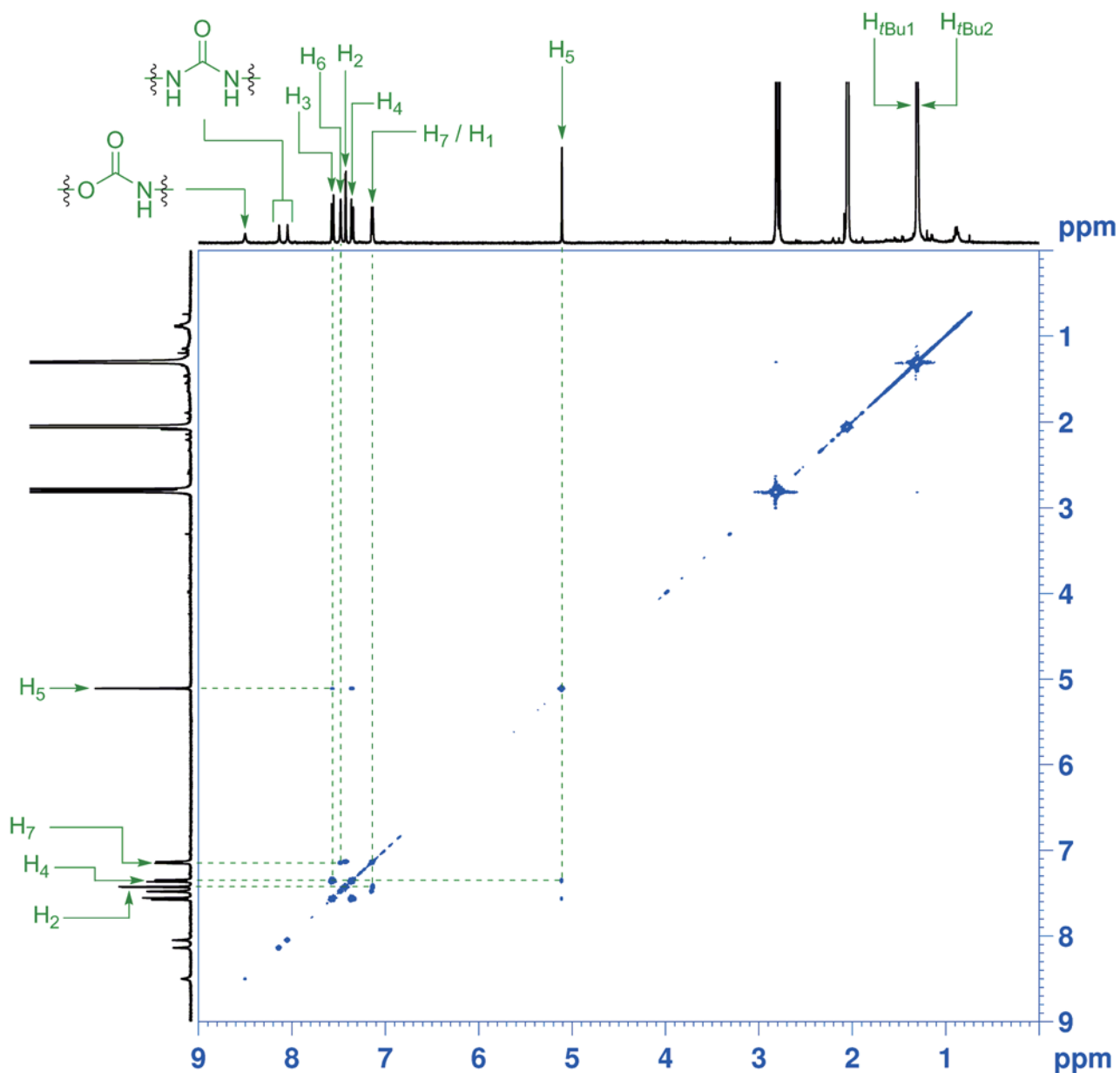
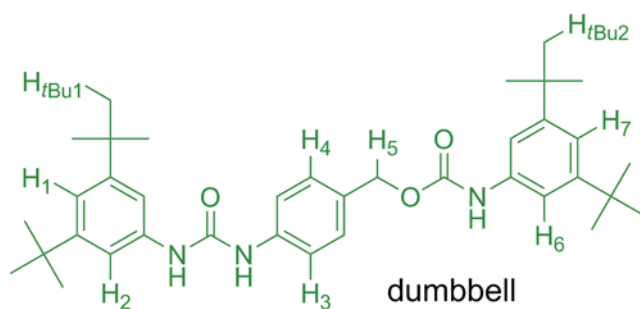
ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



Partial ROESY Spectrum (500 MHz, CD₃COCD₃, 298K)



COSY Spectrum (400 MHz, CD₃COCD₃, 298K)



NOESY Spectrum (400 MHz, CD₃COCD₃, 298K)

