Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting information for

Calixarenes with naphthalene units: calix[4]naphthalenes and hybrid[4]arenes

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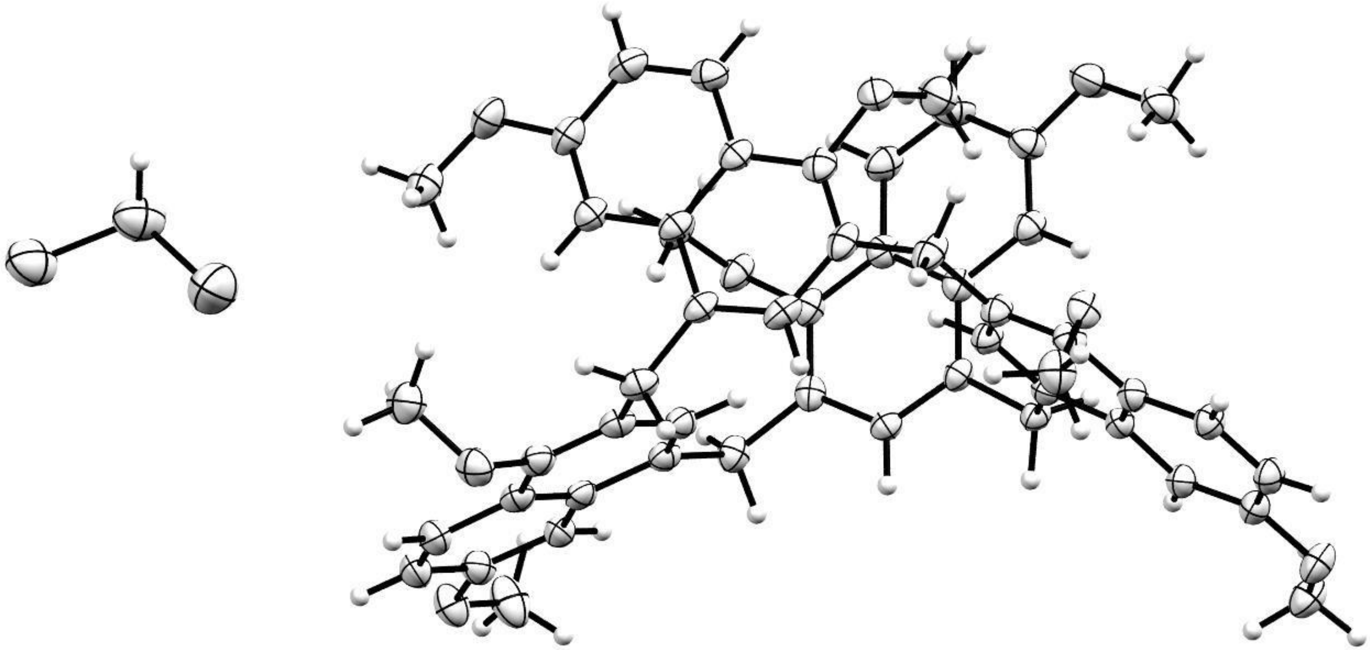


Figure S1: ORTEP view of X-ray structure of **4** (thermal ellipsoids are plotted at the level of 50% probability).

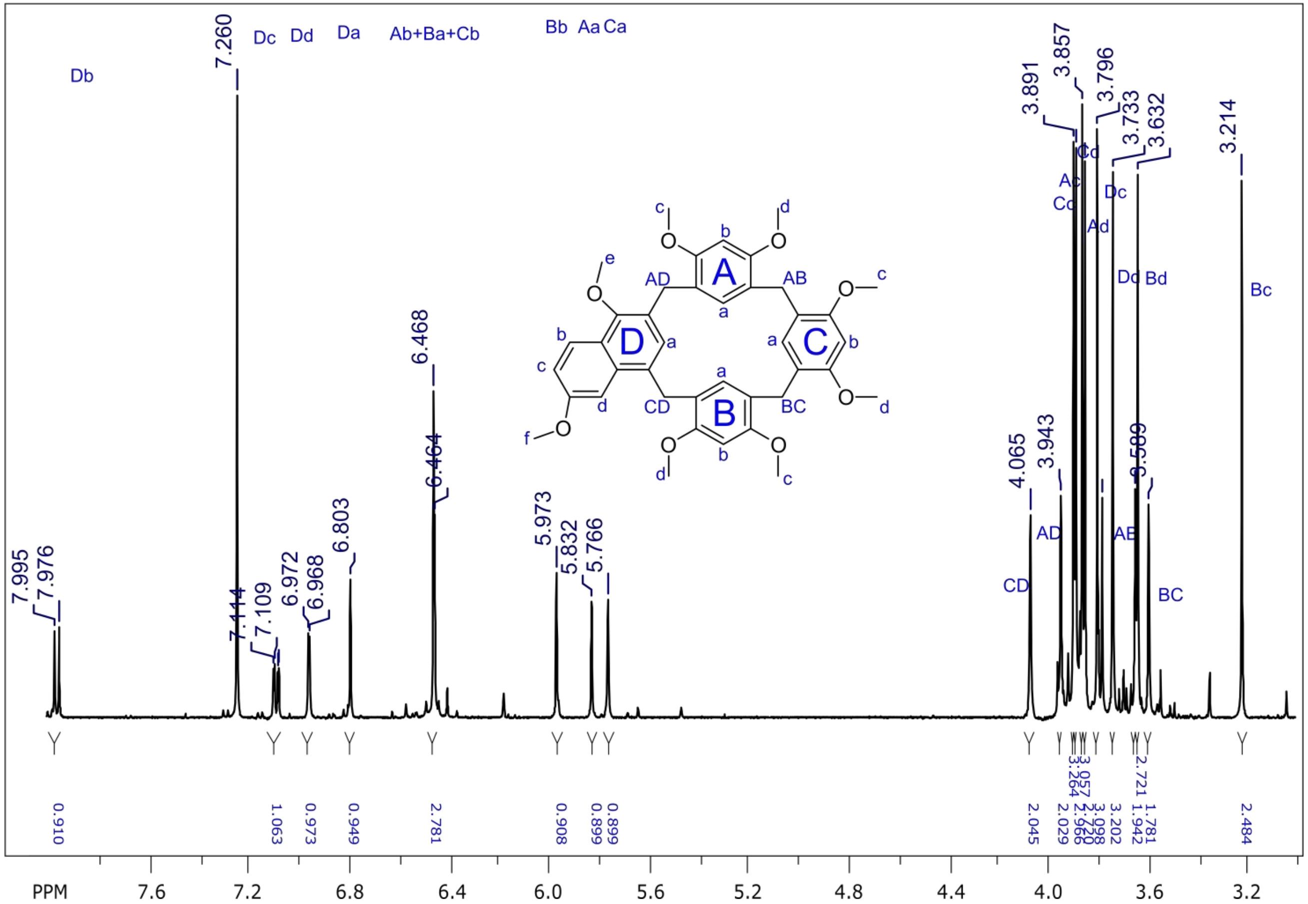


Figure S2: ^{1}H NMR spectrum of **2**

^{1}H NMR ($CDCl_3$, 500 MHz, 298K): 7.99 (d, 1H, J = 9Hz), 7.11 (dd, 1H, J = 9Hz, J = 2Hz), 6.98 (d, 1H, J = 2Hz); 6.81 (s, 1H), 6.47 (s, 3H), 5.98 (s, 1H), 5.84 (s, 1H), 5.77 (s, 1H), 4.07 (s, 2H), 3.95 (s, 2H), 3.90 (s, 3H), 3.89 (s, 3H), 3.86 (s, 3H), 3.85 (s, 3H), 3.80 (s, 3H), 3.78 (s, 3H), 3.74 (s, 3H), 3.65 (s, 2H), 3.64 (s, 3H), 3.60 (s, 2H), 3.22 (s, 3H)

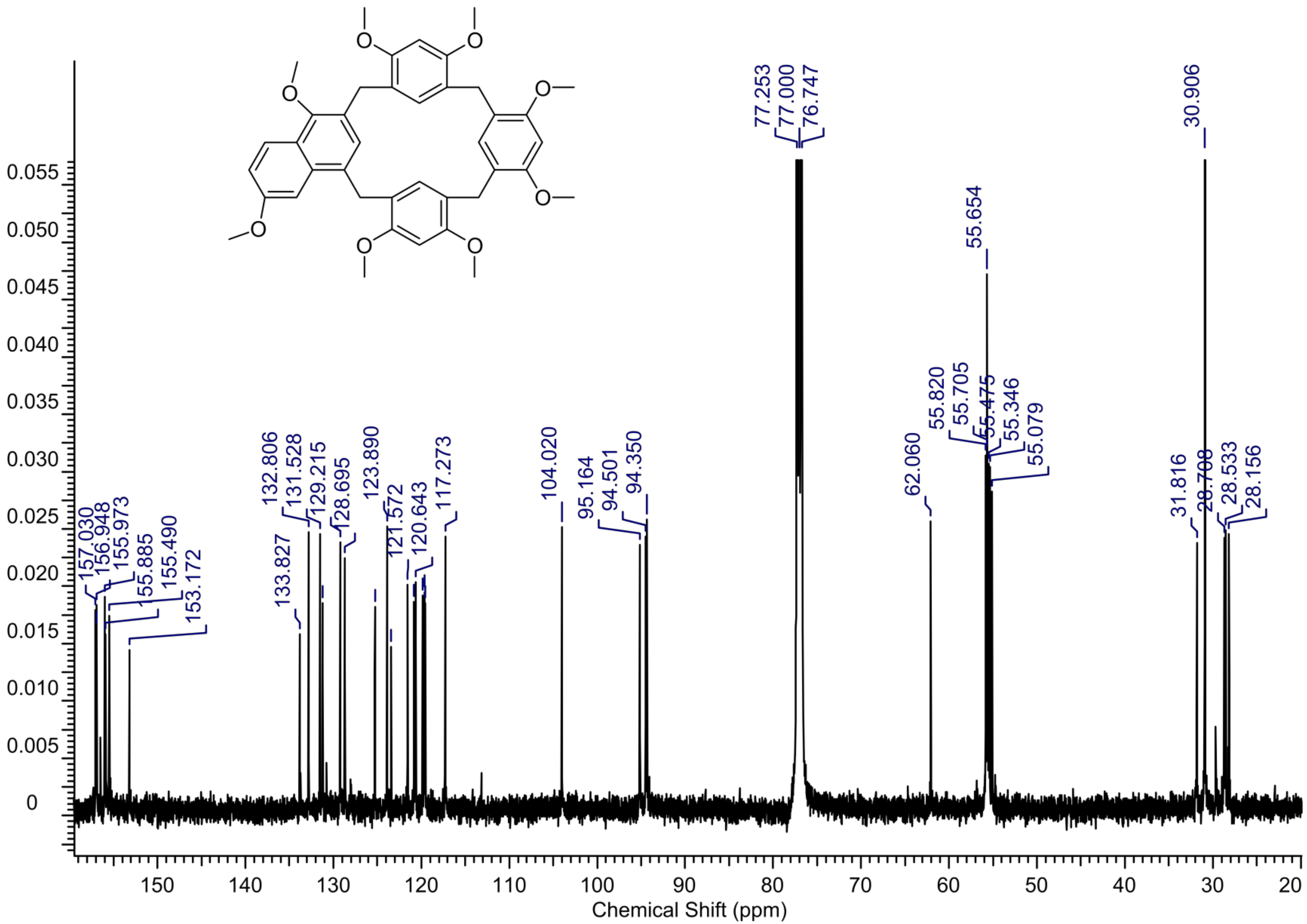


Figure S3: ^{13}C NMR spectrum of **2** ($CDCl_3$, 298K, 500MHz)

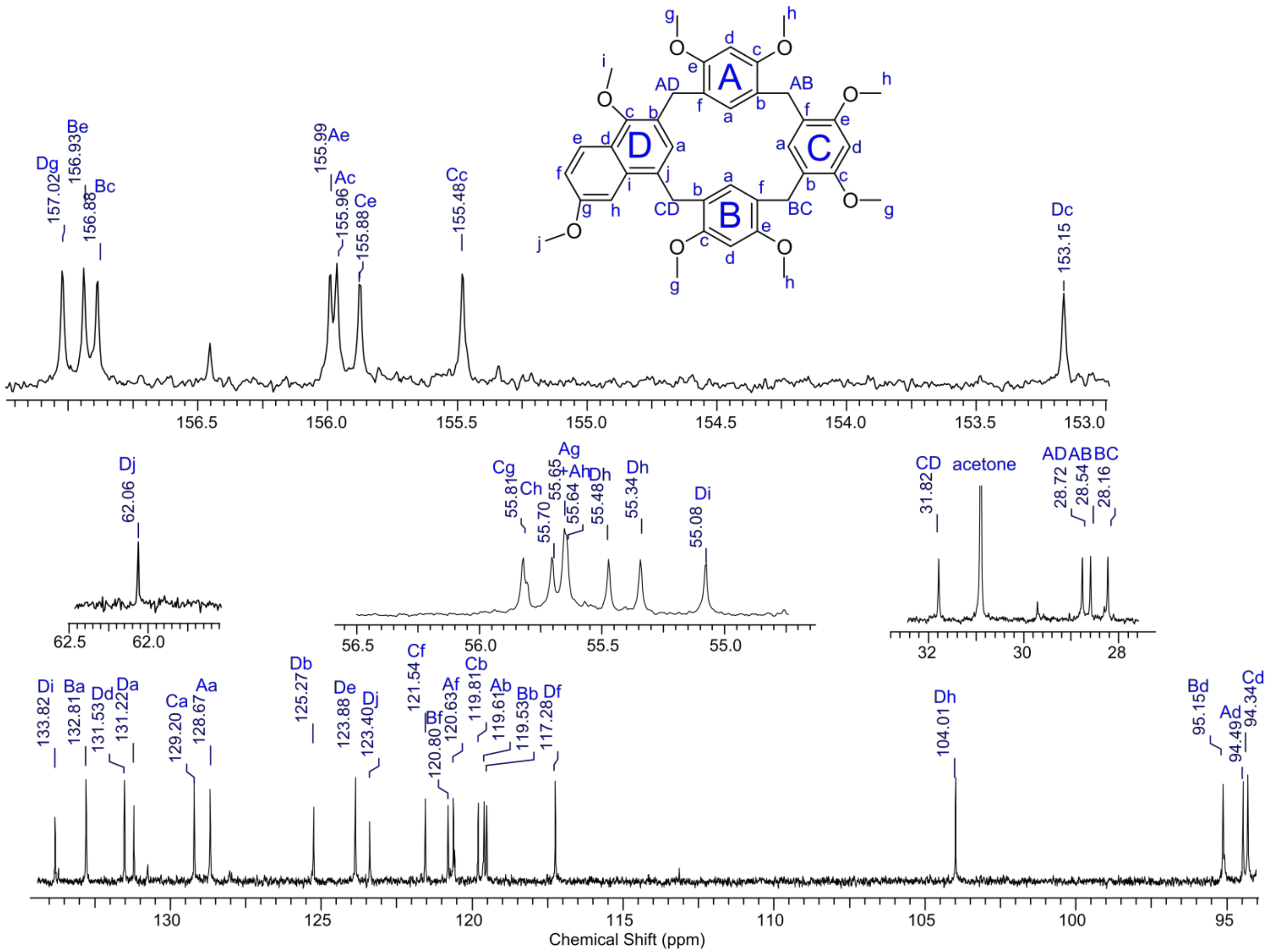


Figure S4: ^{13}C NMR spectrum of **2**

^{13}C NMR ($CDCl_3$, 125 MHz, 298K) : 157.02, 156.93, 156.88, 155.99, 155.96, 155.88, 155.48, 153.15, 133.82, 132.81, 131.53, 131.22, 129.20, 128.67, 125.27, 123.88, 123.40, 121.54, 120.80, 120.63, 119.81, 119.61, 119.53, 117.28, 104.01, 95.15, 94.49, 94.34, 62.06, 55.81, 55.70, 55.65, 55.64, 55.48, 55.34, 55.08, 31.82, 28.72, 28.54, 28.16

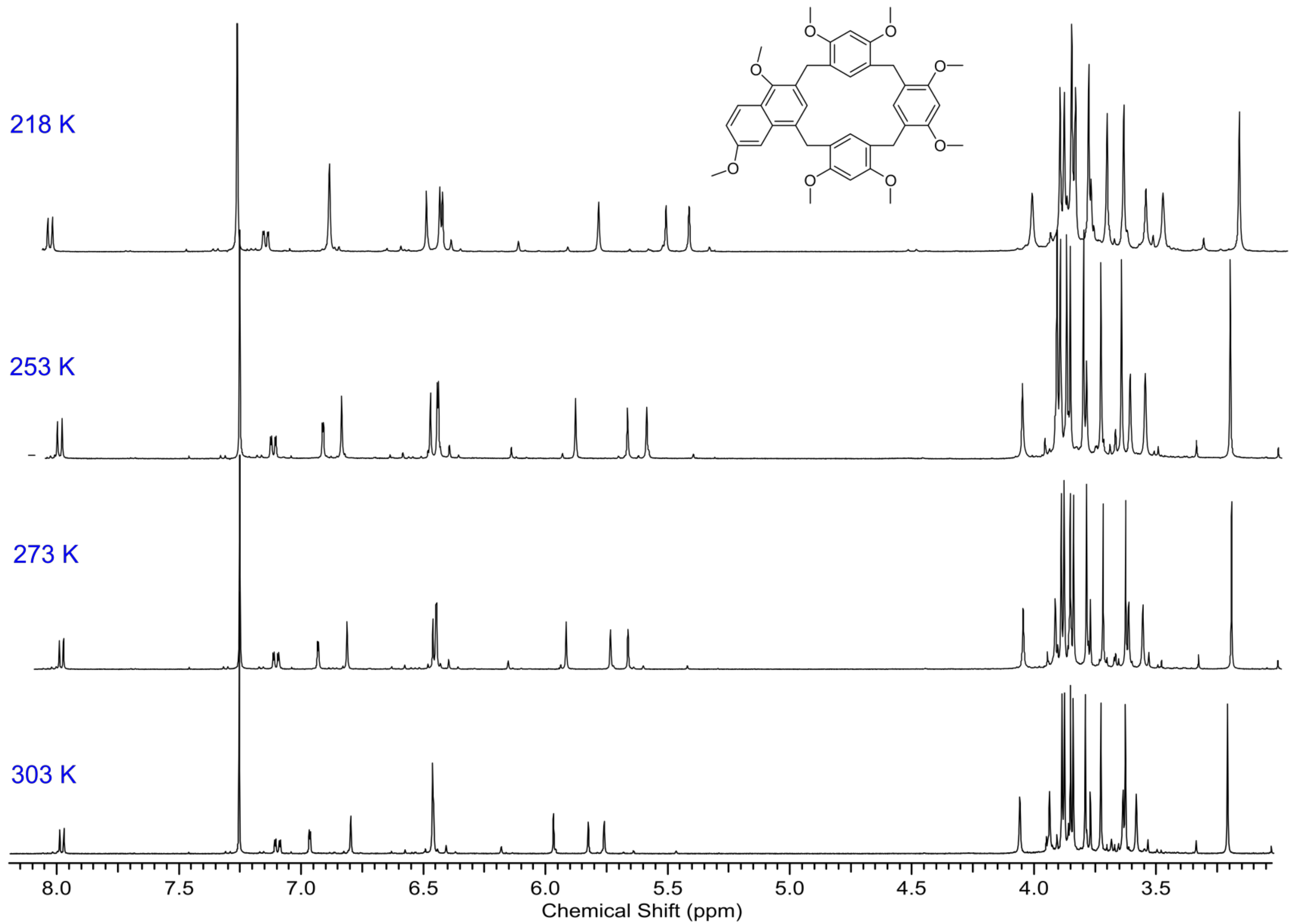


Figure S5: ^{1}H NMR spectra of **2** at various temperatures ($CDCl_3$, 500MHz)

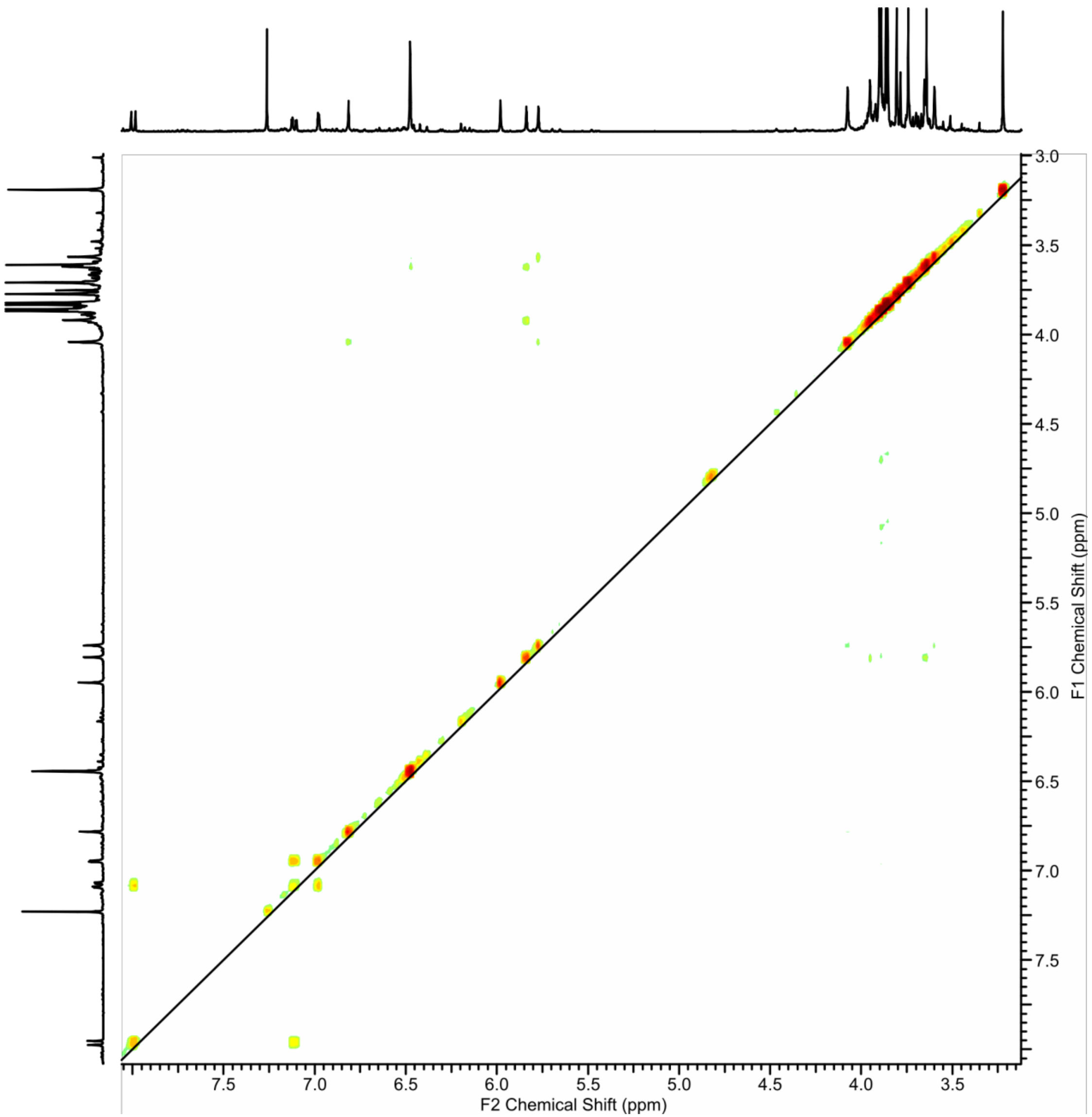


Figure S6: ^1H-^1H COSY spectrum of **2** ($CDCl_3$, 298K, 400MHz)

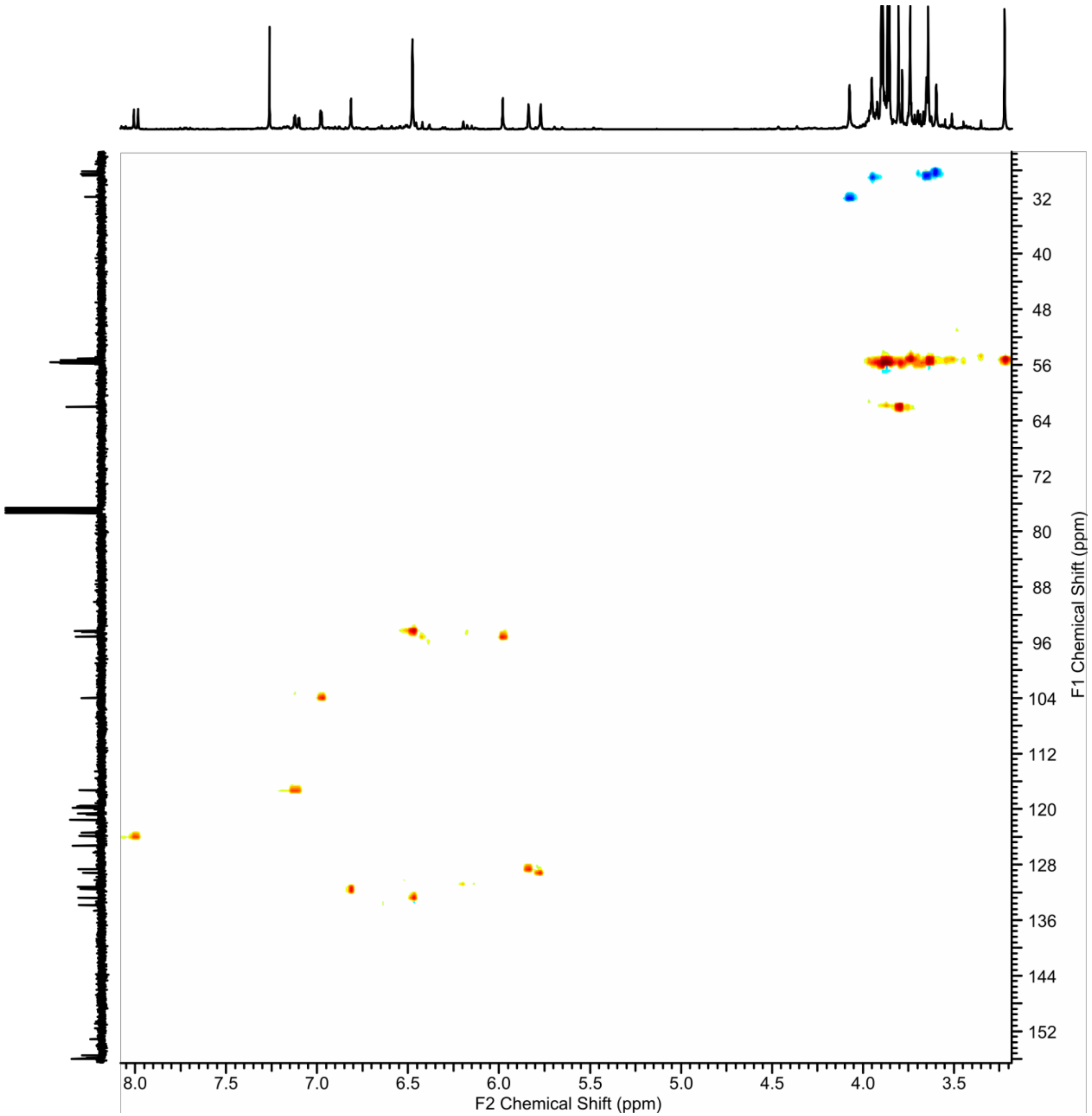


Figure S7: ^{1}H-^{13}C HSQC spectrum of **2** ($CDCl_3$, 298K, 400MHz)

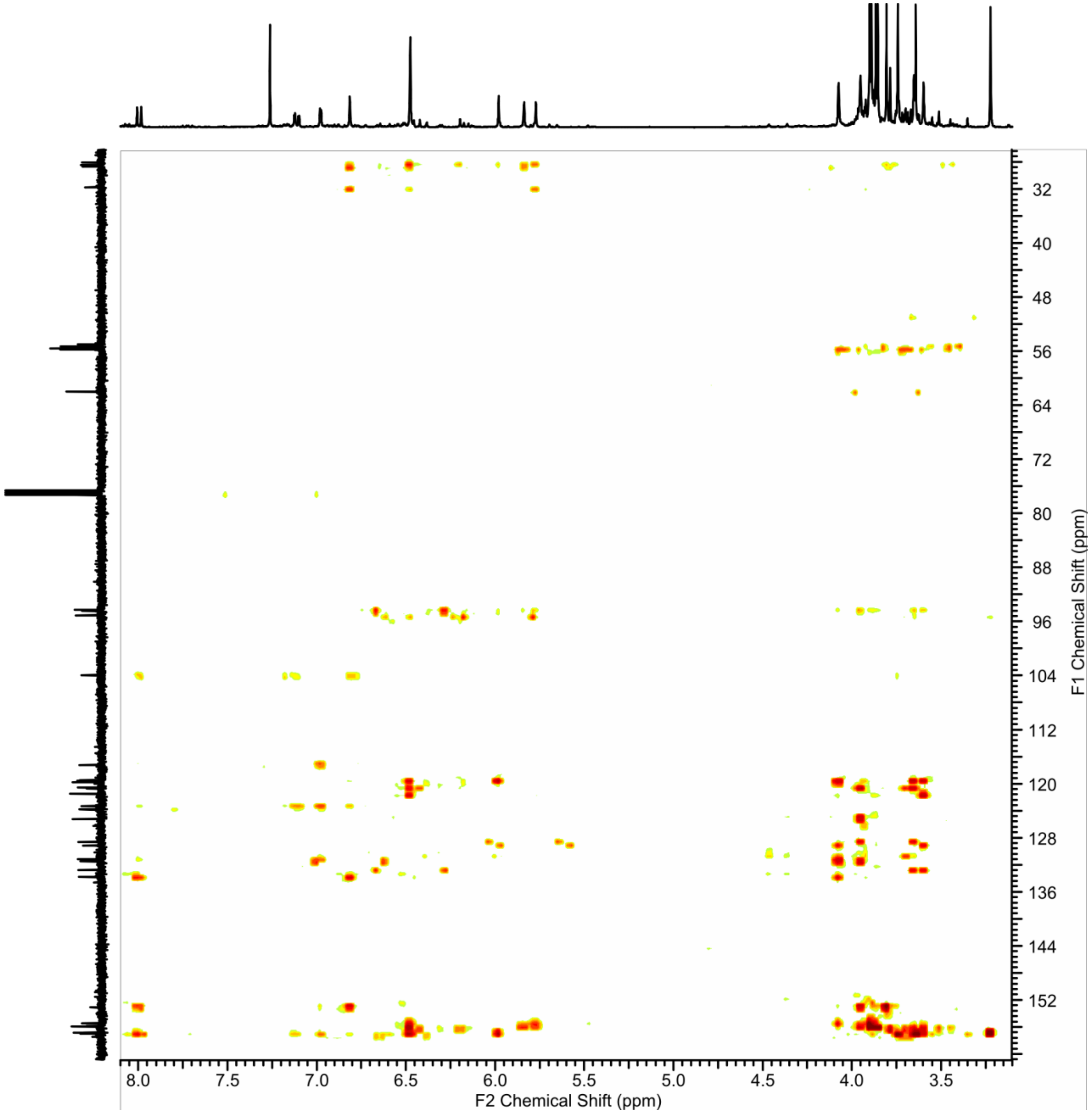


Figure S8: ^1H-^{13}C HMBC spectrum of **2** ($CDCl_3$, 298K, 400MHz)

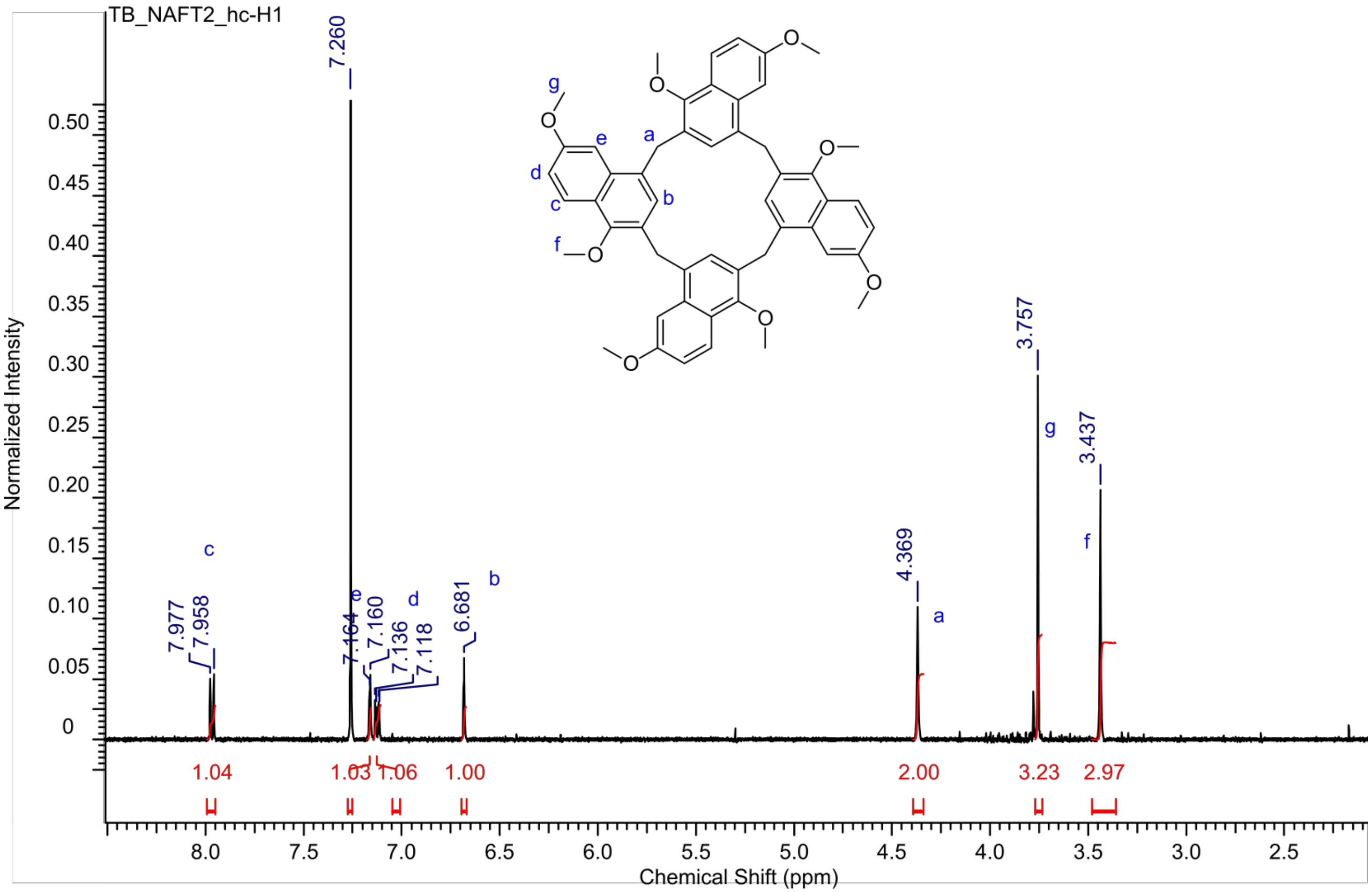


Figure S9: [1]H NMR spectrum of **3**

[1]H NMR ($CDCl_3$, 298K, 500MHz) : 7.97 (d, 4H, *J* = 9Hz), 7.16 (d, 4H J = 2Hz), 7.15 (dd, 4H, J = 9Hz, J = 2Hz), 6.69 (s, 4H), 4.37 (s, 8H), 3.76 (s, 12H), 3.44 (s, 12H)

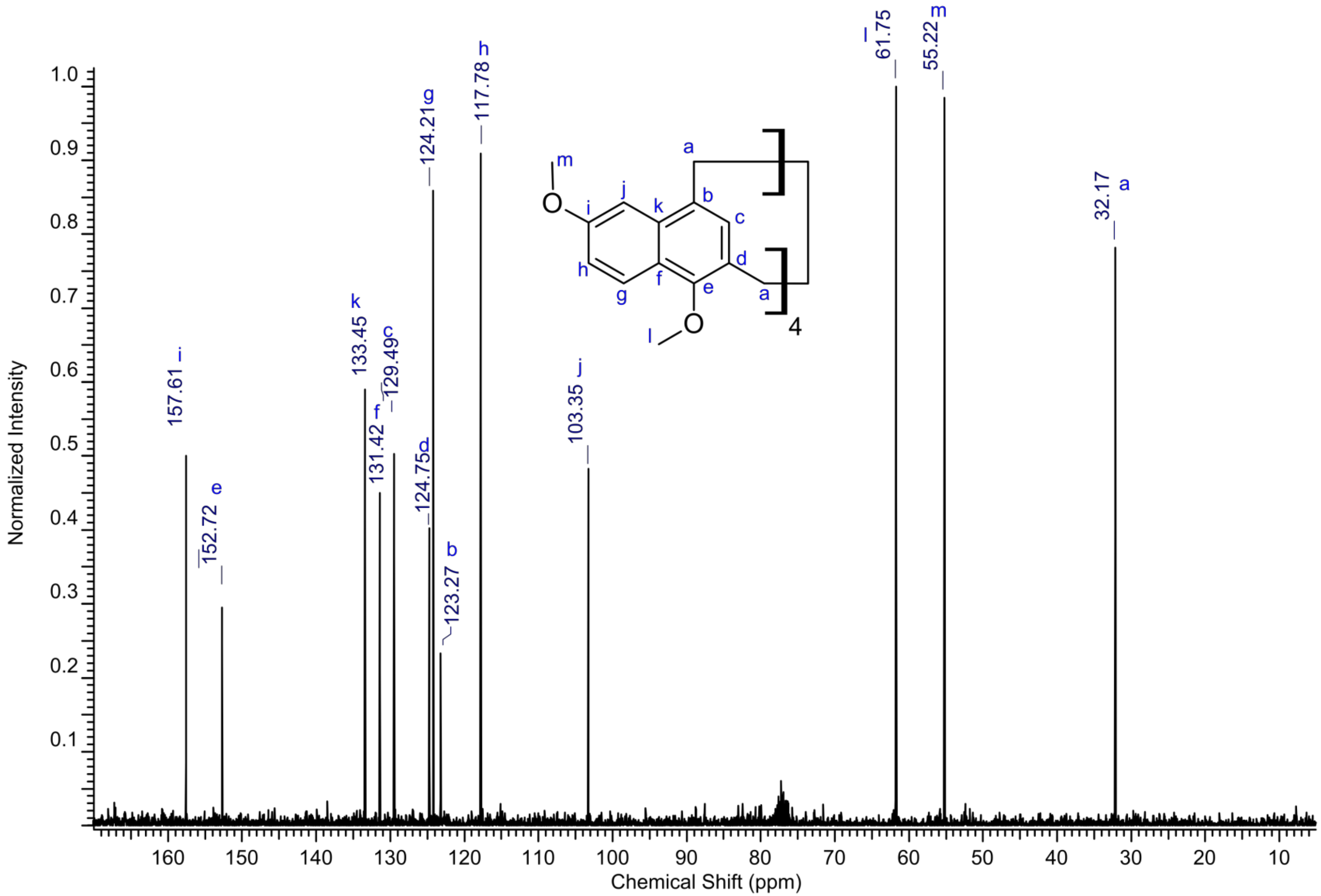


Figure S10: ^{13}C NMR spectrum of **3**

^{13}C NMR ($CDCl_3$, 298K, 125 MHz) : 157.61, 152.72, 133.45, 131.42, 129.49, 124.75, 124.21, 123.27, 117.78, 103.55, 61.76, 55.22, 32.16

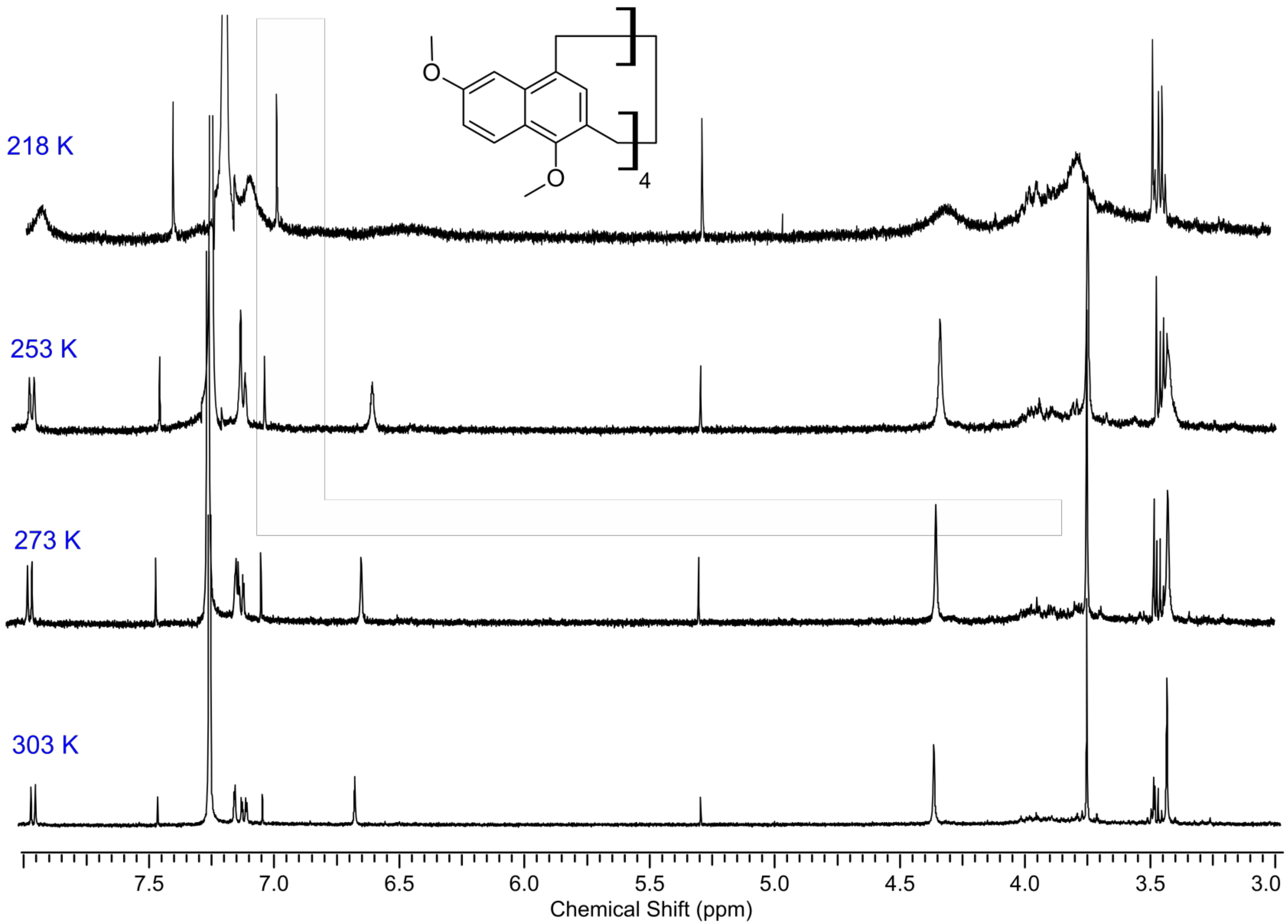


Figure S11: ^{1}H NMR spectra of **3** at various temperatures (CDCl$_3$, 500MHz)

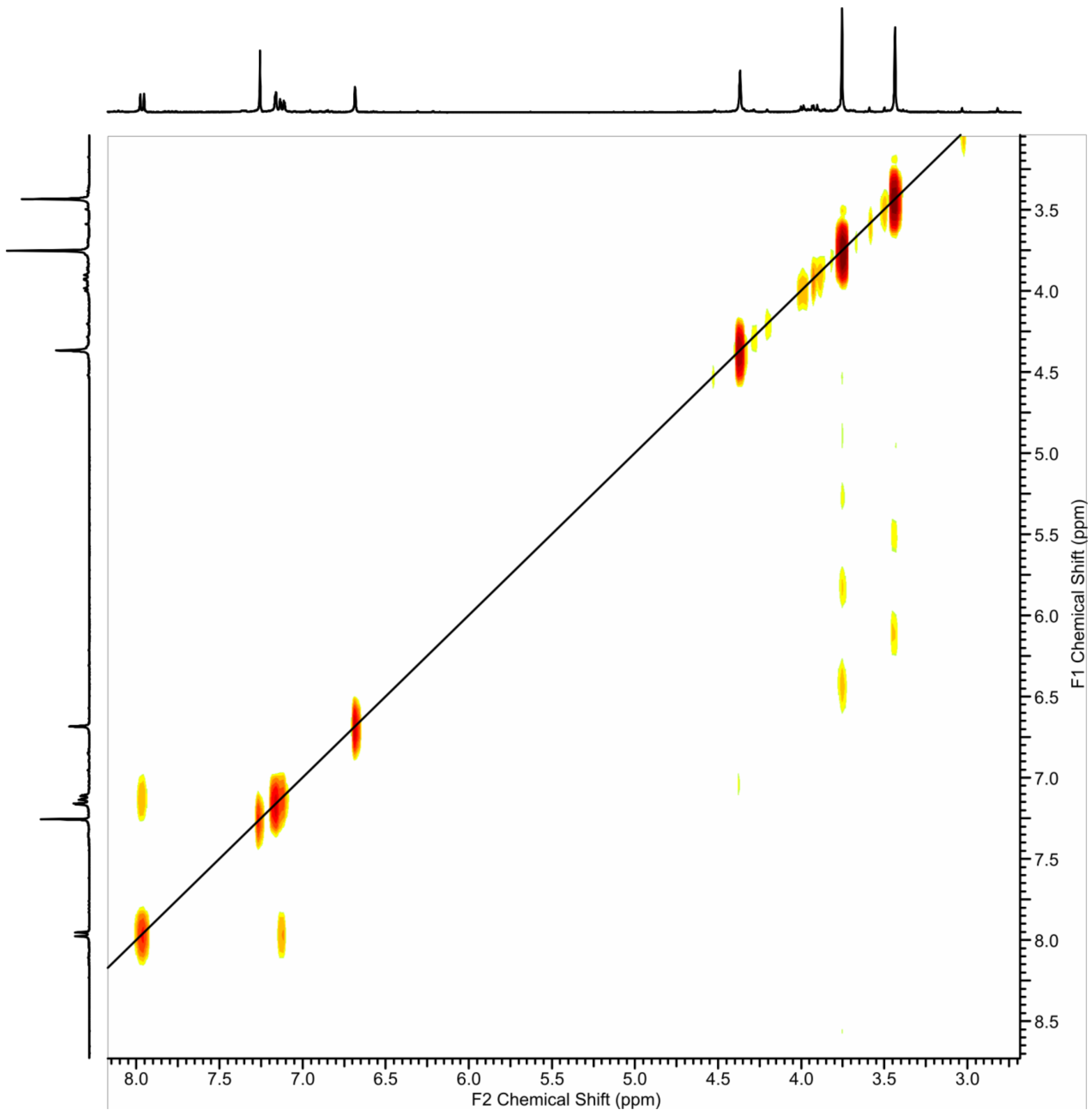


Figure S12: ^{1}H-^{1}H COSY spectrum of **3** ($CDCl_3$, 298K, 400MHz)

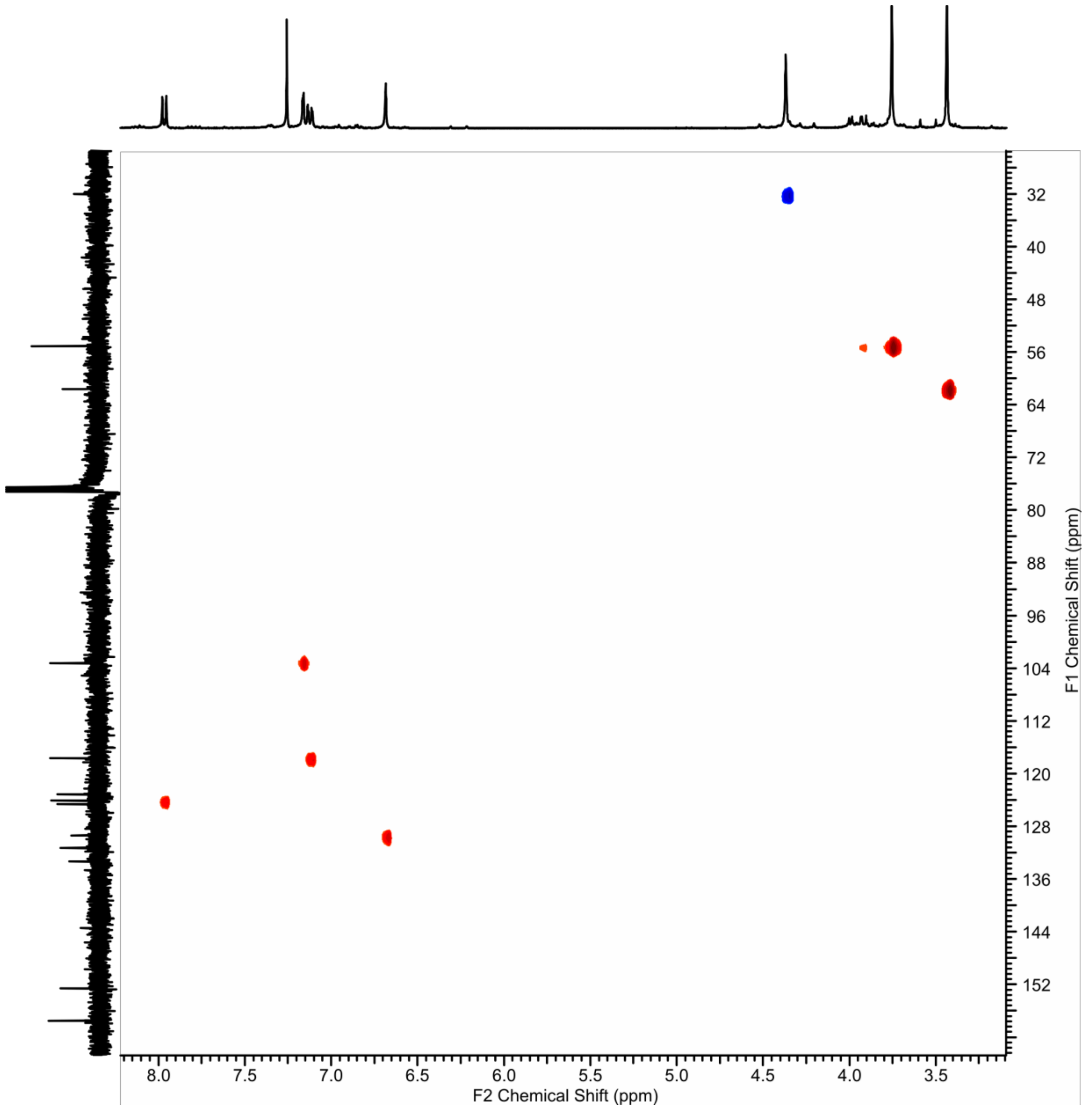


Figure S13: ^{1}H-^{13}C HSQC spectrum of **3** ($CDCl_3$, 298K, 400MHz)

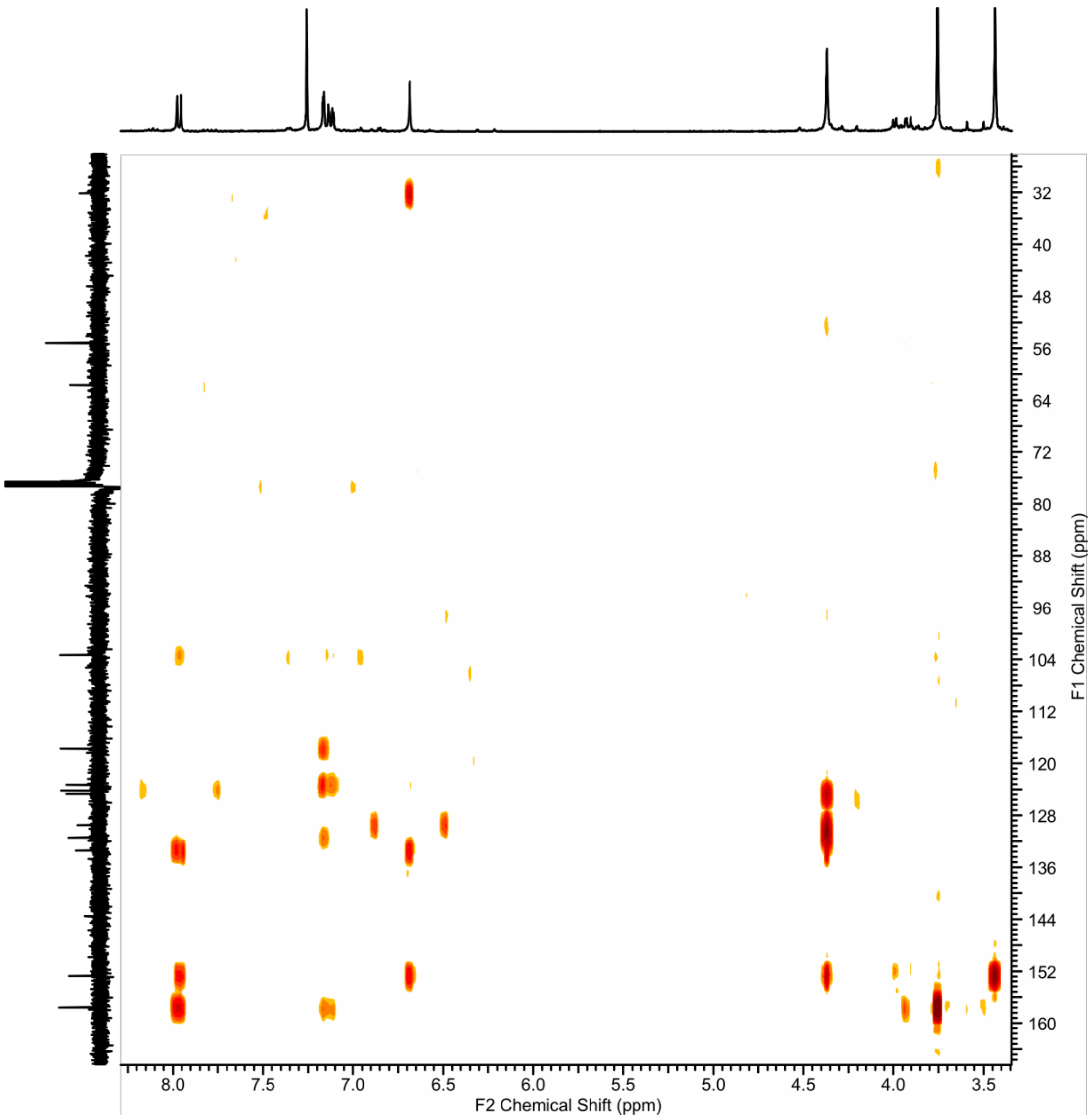


Figure S14: ^{1}H-^{13}C HMBC spectrum of **3** ($CDCl_3$, 298K, 400MHz)

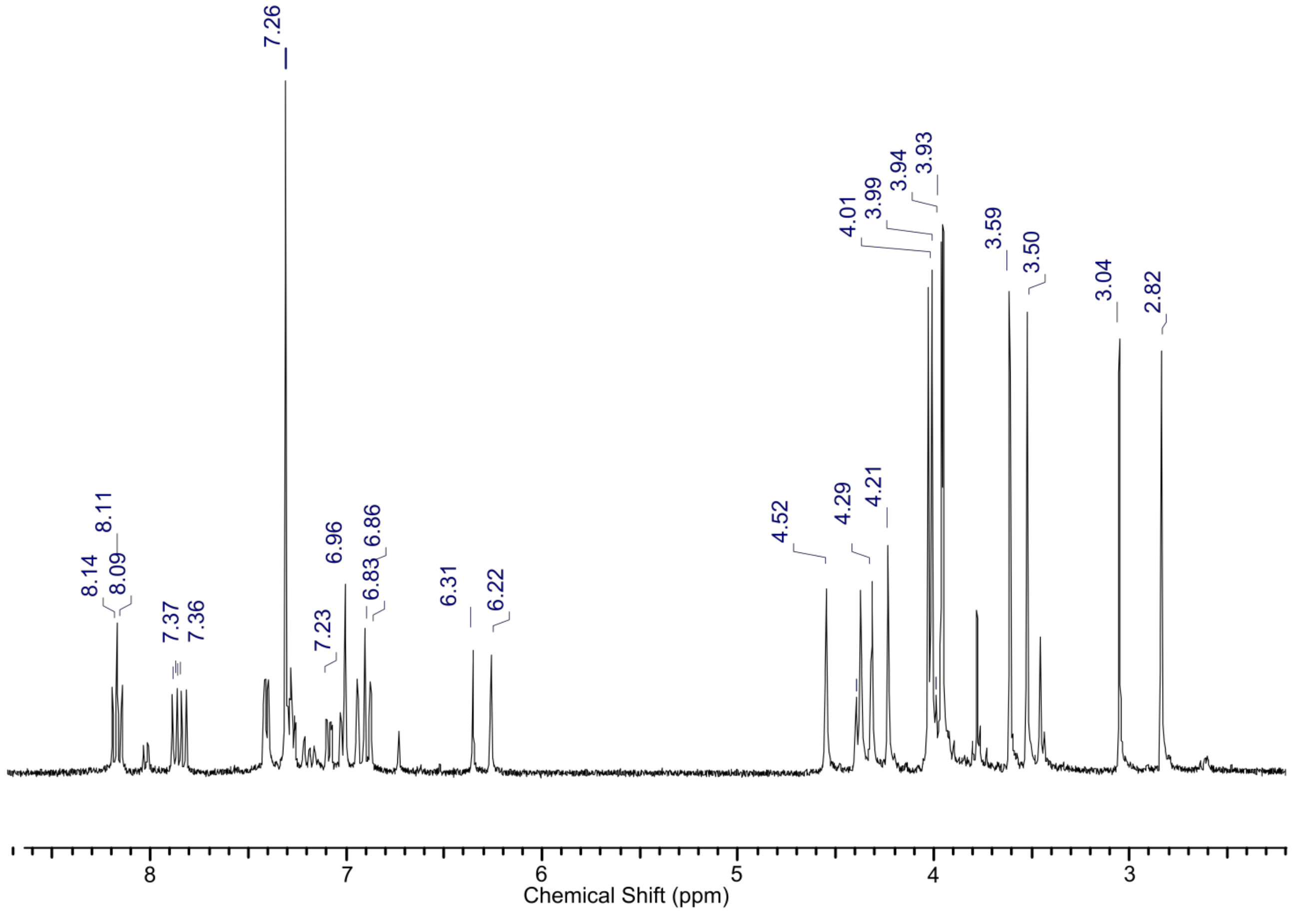


Figure S15: ^{1}H NMR spectrum of **4**

^{1}H NMR ($CDCl_3$, 298K, 400MHz): 8.13 (d, 1H, *J* = 9 Hz), 8.10 (d, 1H, *J* = 9 Hz), 7.82 (d, 1H, *J* = 9 Hz), 7.77 (d, 1H, *J* = 9 Hz), 7.36 (m, 2H), 7.23 (m, 2H), 6.97 (m, 1H), 6.96 (s, 1H), 6.90(d, 1H, *J* = 2Hz), 6.86 (s, 1H), 6.83 (d, 1H, *J* = 2Hz), 6.31 (s, 1H), 6.22 (s, 1H), 4.52 (s, 2H), 4.35 (s, 2H), 4.29 (s, 2H), 4.21 (s, 2H), 4.01 (s, 3H), 3.99 (s, 3H), 3.94 (s, 3H), 3.93 (s, 3H), 3.59 (s, 3H), 3.50 (s, 3H), 3.04 (s, 3H), 2.82 (s,3H)

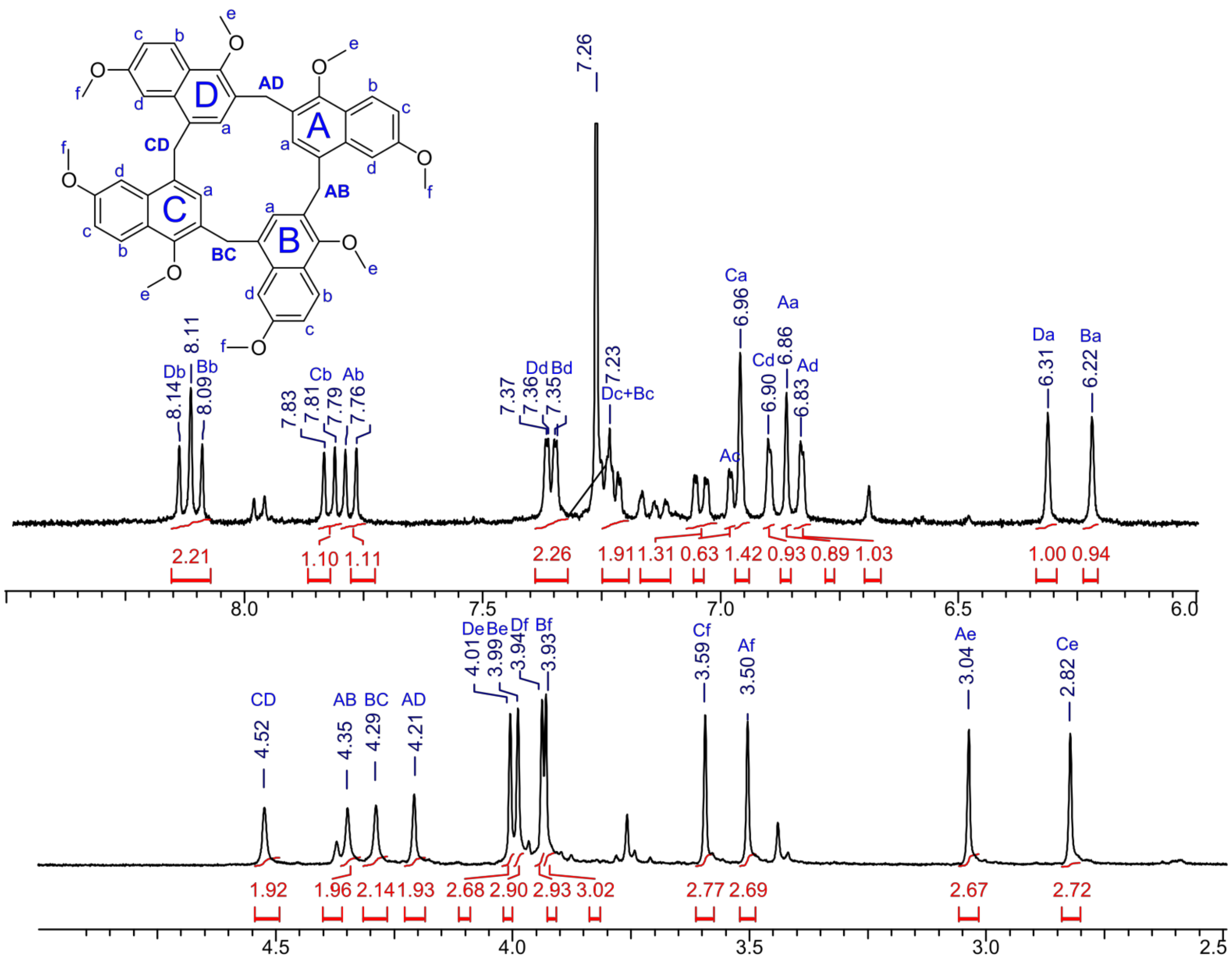


Figure S16: ^{1}H NMR spectrum of **4** - correlated signals ($CDCl_3$, 400MHz, 298K)

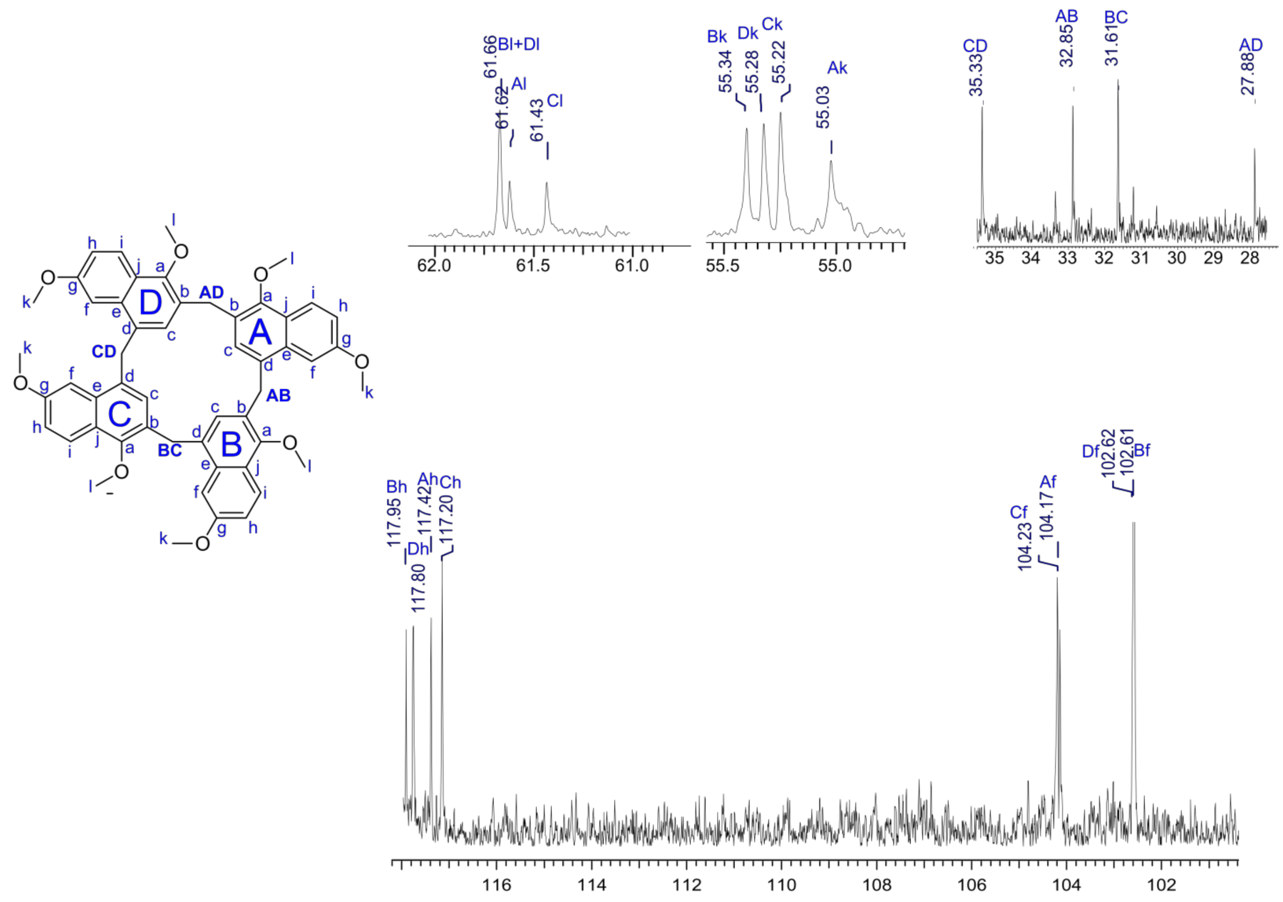


Figure S17: ^{13}C NMR spectrum of **4** ($CDCl_3$, 298K, 100MHz)

^{13}C NMR ($CDCl_3$, 100MHz, 298K): 157.74, 157.71, 157.58, 157.28, 153.52, 153.42, 152.60, 151.79, 133.87, 133.30, 133.24, 133.08, 131.98, 131.78, 131.23, 130.73, 130.23, 128.34, 127.16, 126.34, 125.79, 125.38, 124.78, 124.40, 124.38, 124.27, 124.25, 124.03, 123.41, 123.30, 123.14, 122.34, 117.95, 117.80, 117.42, 117.20, 104.23, 104.17, 102.62, 102.61, 99.99, 61.66, 61.62, 61.43, 55.34, 55.28, 55.22, 55.03, 35.33, 32.85, 31.61, 27.88

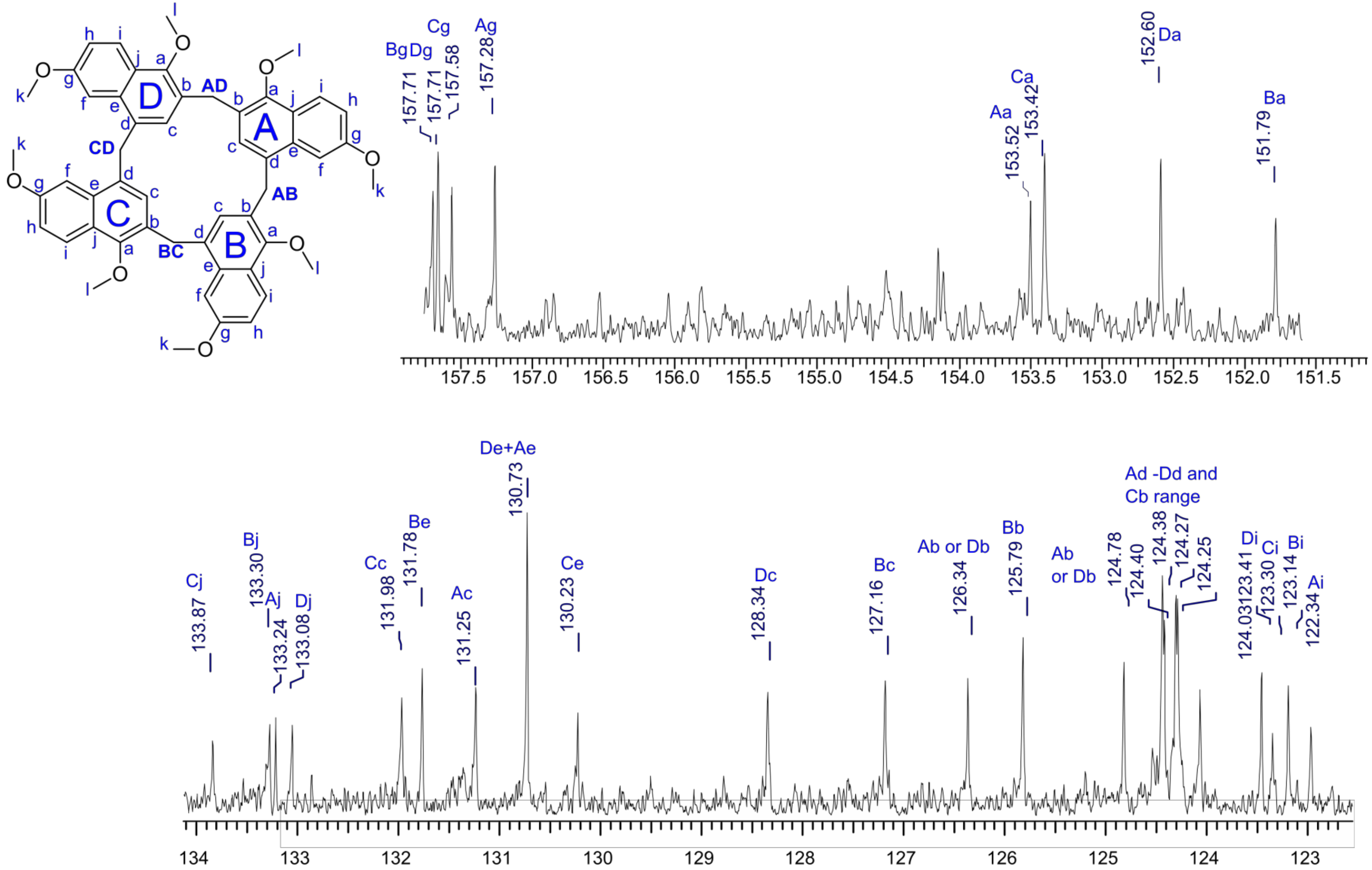


Figure S18: ^{13}C NMR spectrum of **4** ($CDCl_3$, 298K, 100MHz)

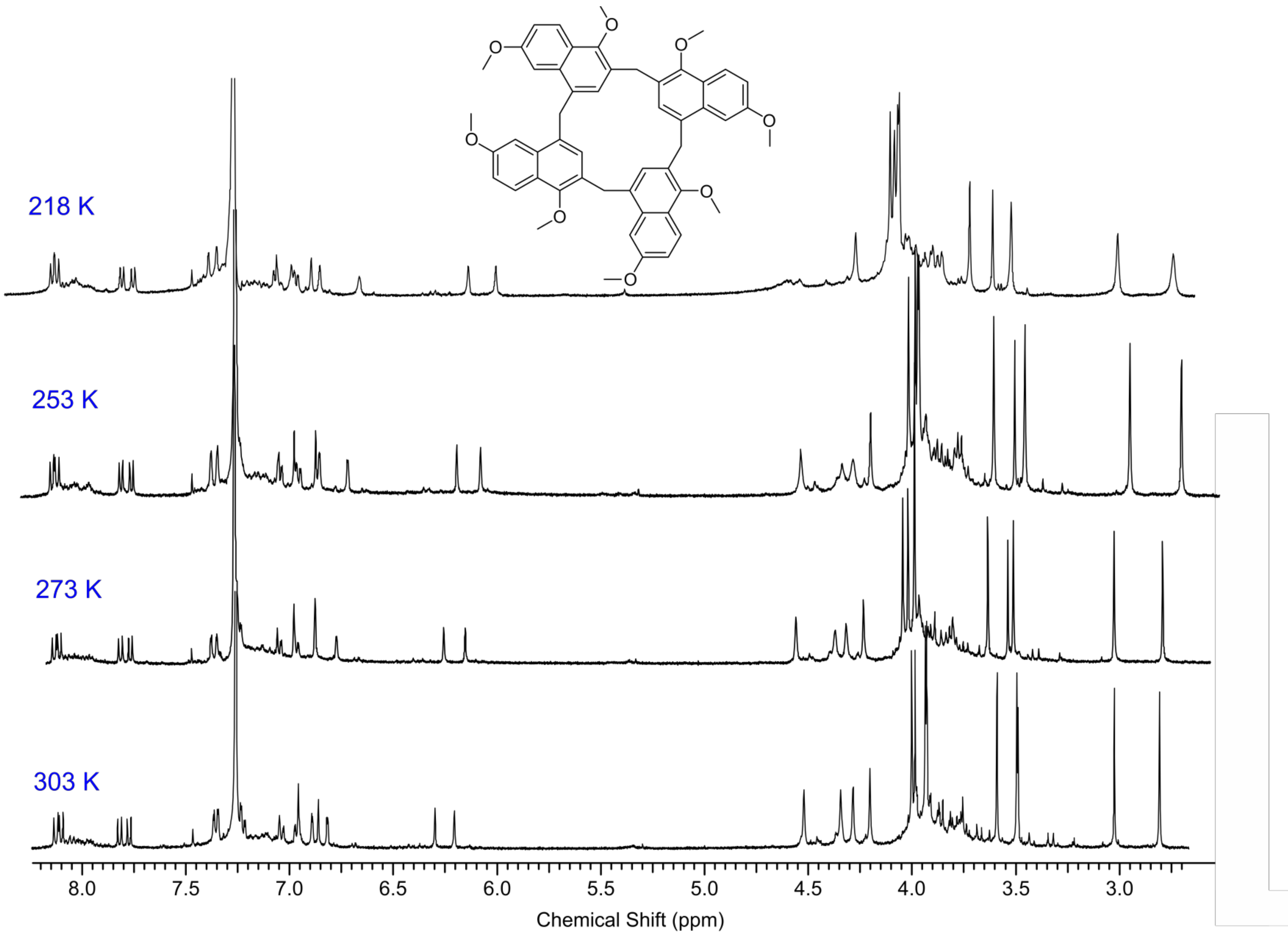


Figure S19: ^{1}H NMR spectra of **4** at various temperatures ($CDCl_3$, 500MHz)

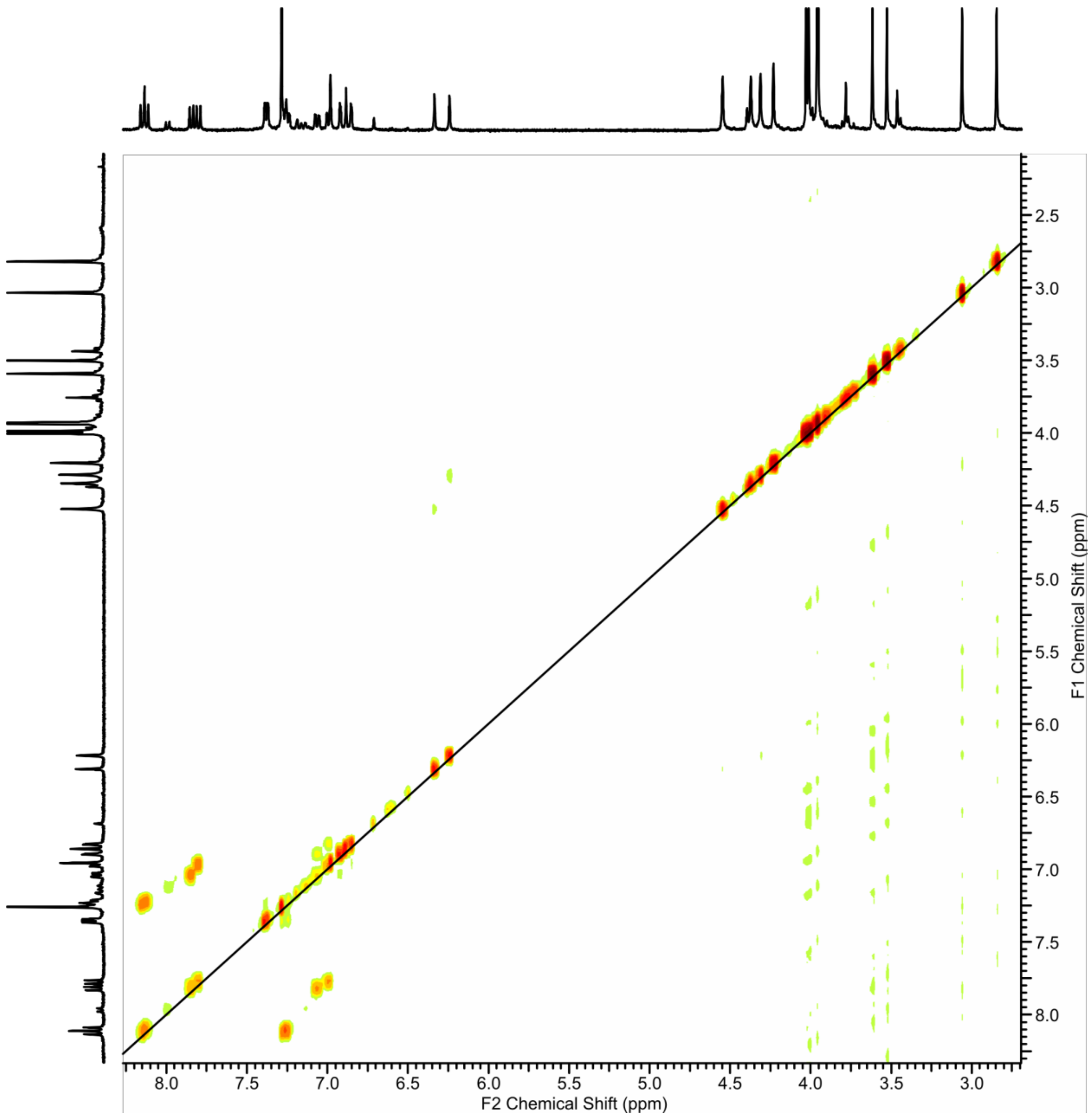


Figure S20: ^{1}H -^{1}H COSY spectrum of **4** ($CDCl_3$, 298K, 400MHz)

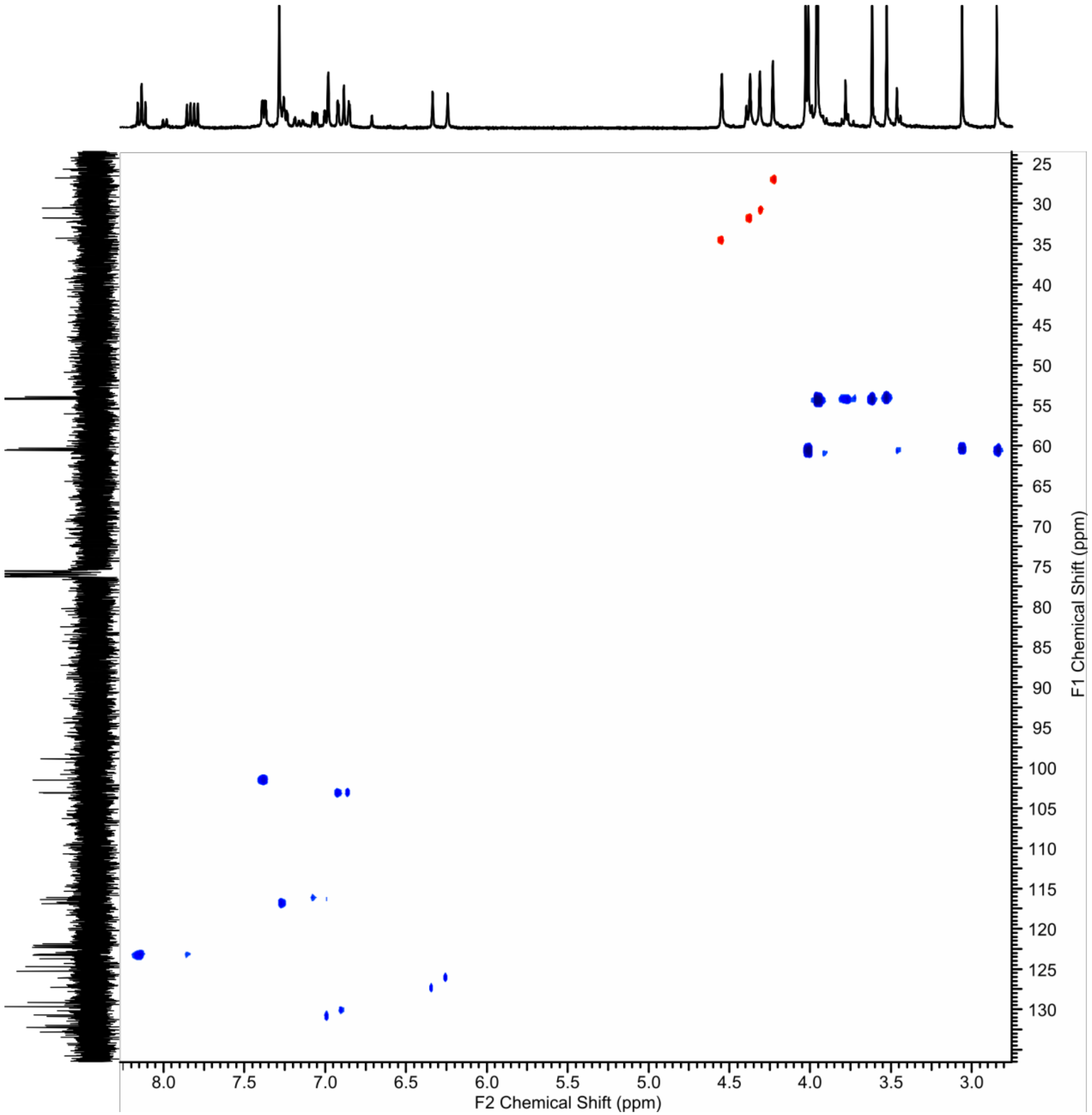


Figure S21: ^{1}H - ^{13}C HSQC NMR spectrum of **4** ($CDCl_3$, 298K, 400MHz)

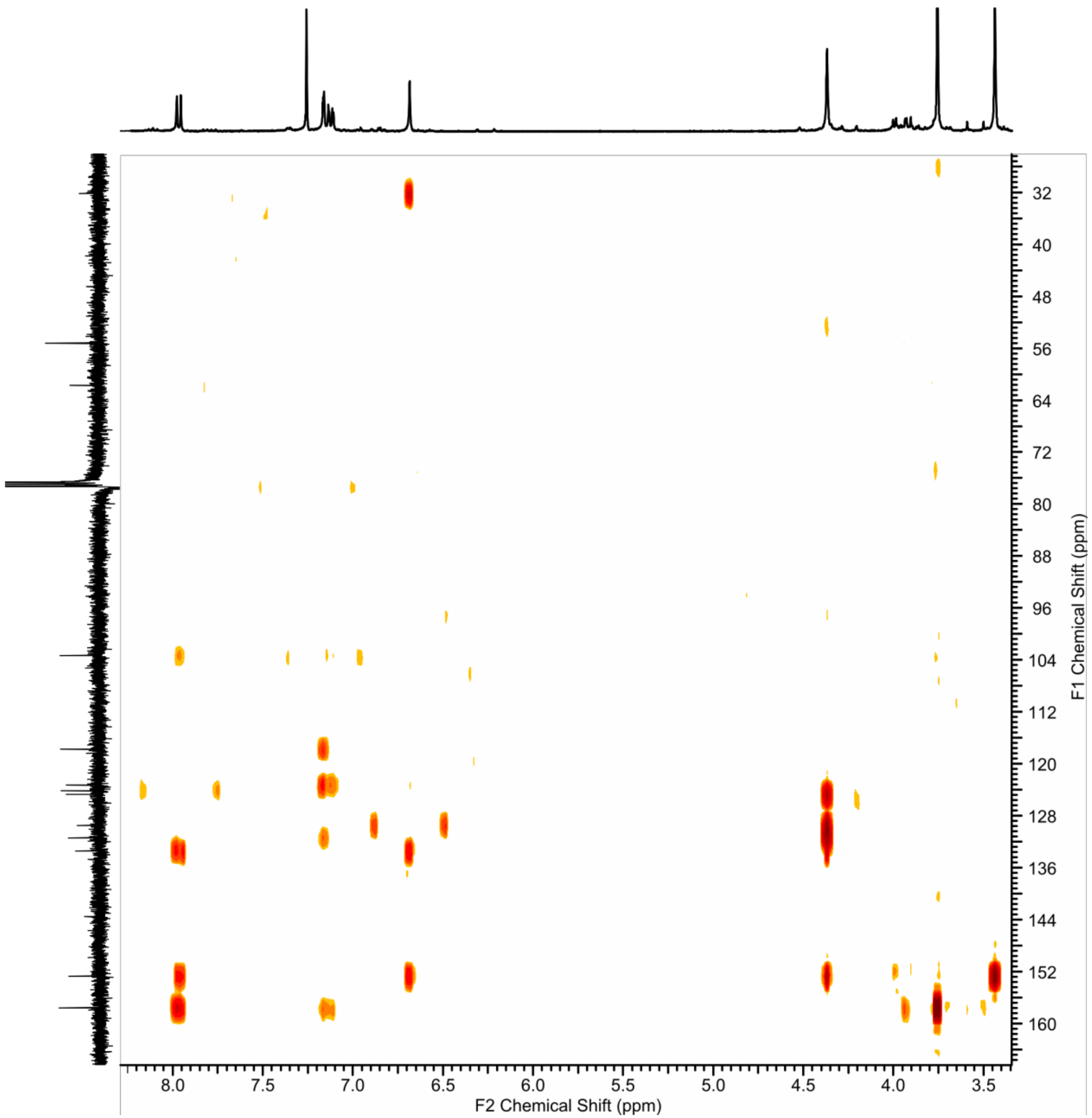


Figure S22: ^1H - ^{13}C HMBC NMR spectrum of **4** ($CDCl_3$, 298K, 400MHz)

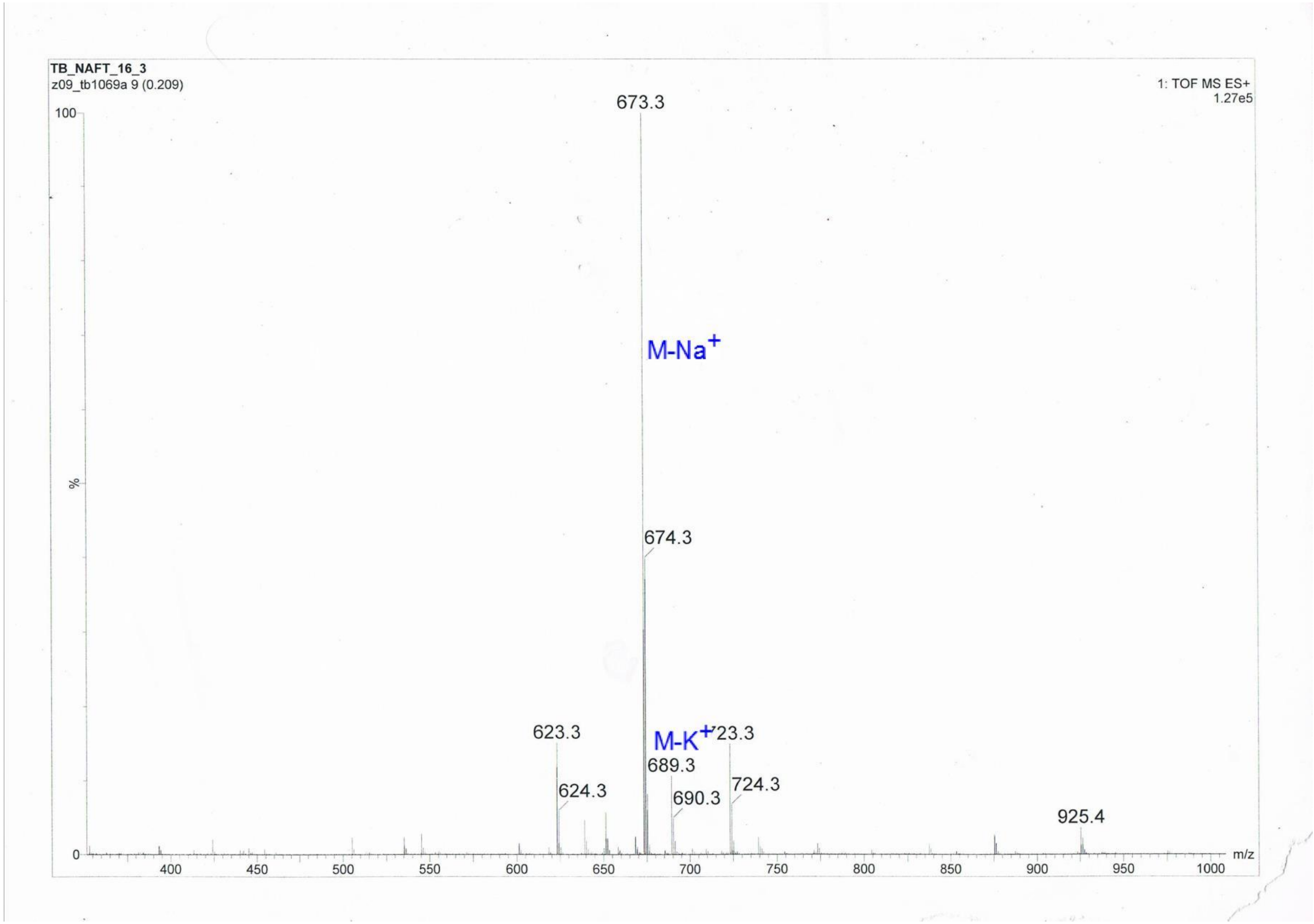


Figure S23: ESI MS of **2**

Elemental Composition Report

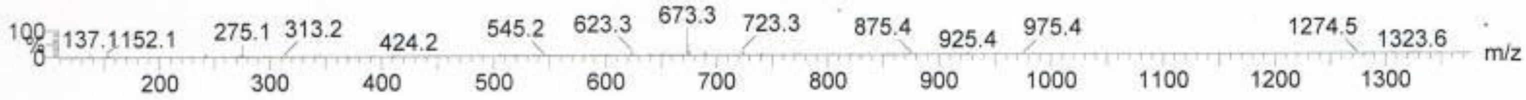
Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -1.5, max = 150.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
139 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-70 H: 0-150 O: 0-20 Na: 1-1
TB_NAFT_16_3
z09_tb1069a 9 (0.209)

1: TOF MS ES+
4.73e+005



	Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
Minimum:					-1.5				
Maximum:			3.0	10.0	150.0				
	673.2767	673.2777	-1.0	-1.5	19.5	79.8	n/a	n/a	C40 H42 O8 Na

Figure S24: HR ESI MS of **2**

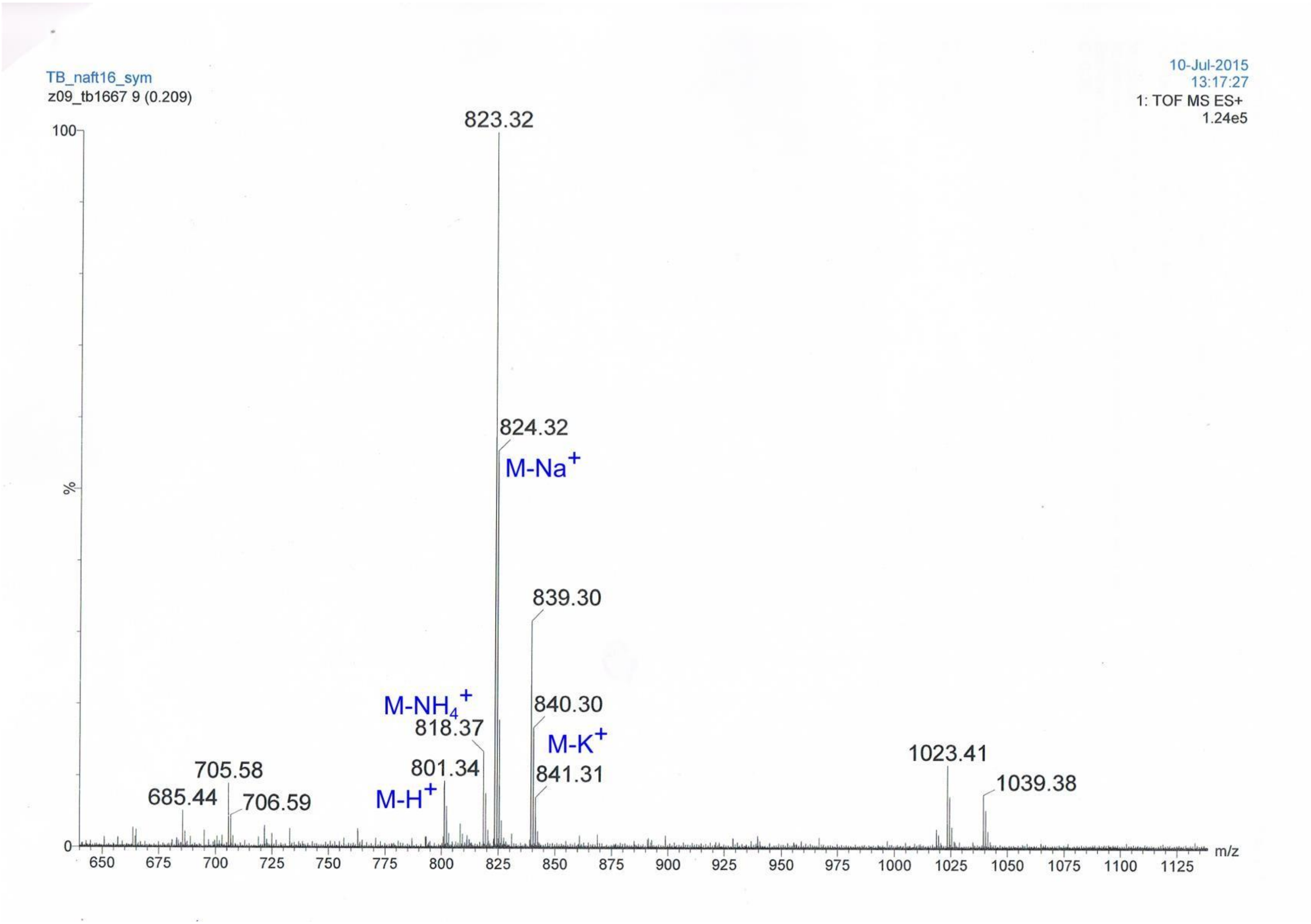


Figure S25: ESI MS of **3**

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -1.5, max = 300.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
369 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-80 H: 0-150 O: 0-20 Na: 0-1

Minimum:				-1.5				
Maximum:		3.0	10.0	300.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
823.3231	823.3247	-1.6	-1.9	28.5	310.3	0.000	99.99	C52 H48 O8 Na
	823.3212	1.9	2.3	40.5	320.0	9.725	0.01	C61 H43 O3

Figure S26: HR ESI MS of **3**

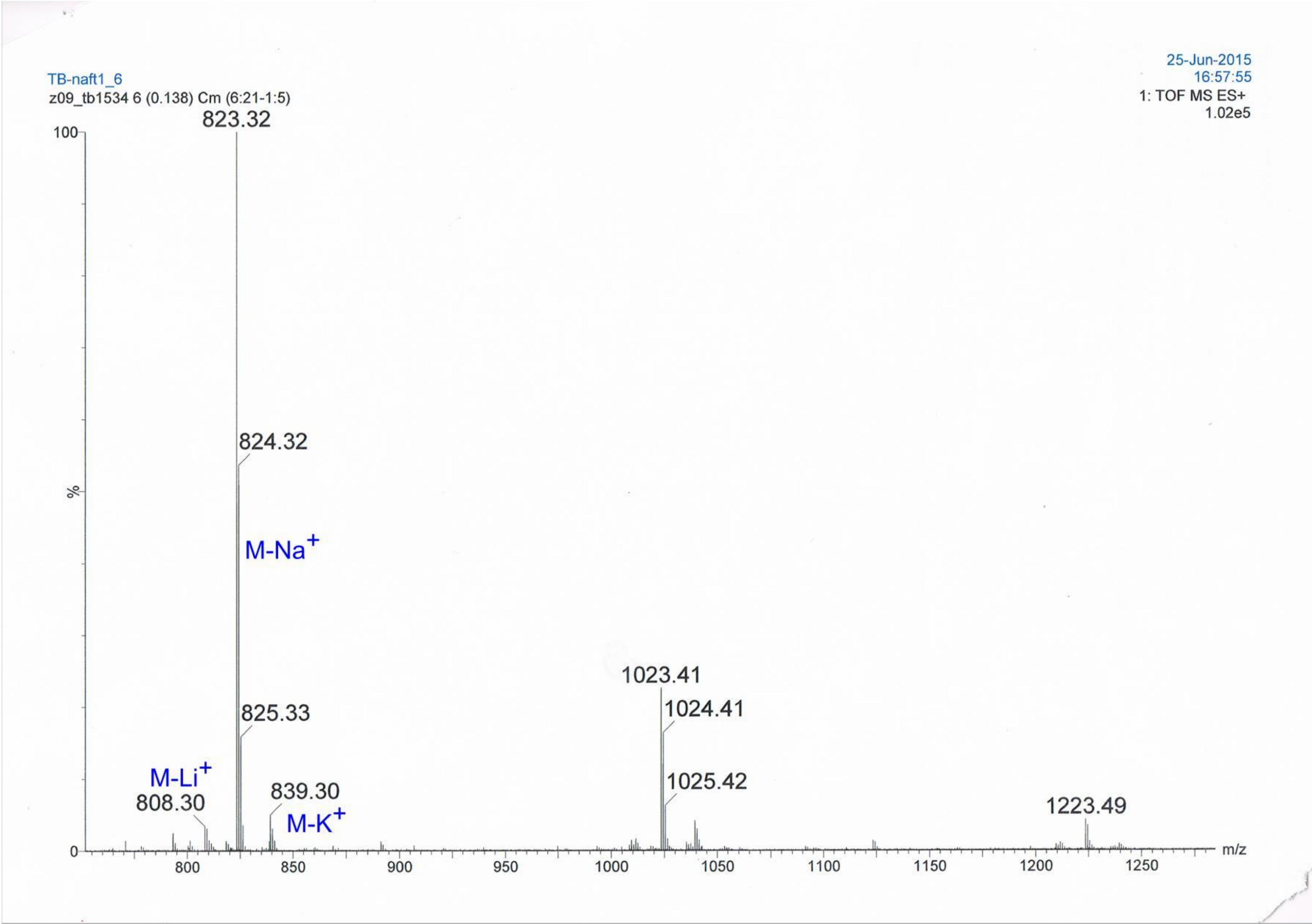


Figure S27: ESI MS of **4**

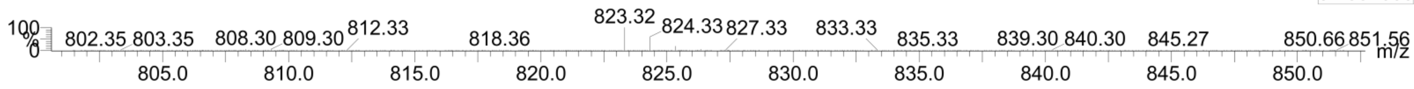
Elemental Composition Report — Page 1

Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -1.5, max = 100.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 9

Monoisotopic Mass, Even Electron Ions
91 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-100 H: 0-200 O: 6-10 Na: 0-1

TB-naft1_6 — 25-Jun-201516:57:55
z09_tb1534 16 (0.327) AM2 (Ar,30000.0,0.00,0.00); Cm (9:16-(2:7+41:49)) — 1: TOF MS ES+
3.46e+005



	mDa	PPM	DBE
Minimum:			-1.5
Maximum:	3.0	10.0	100.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
823.3228	823.3247	-1.9	-2.3	28.5	862.7	n/a	n/a	C52 H48 O8 Na

Figure S28: HR ESI MS of **4**

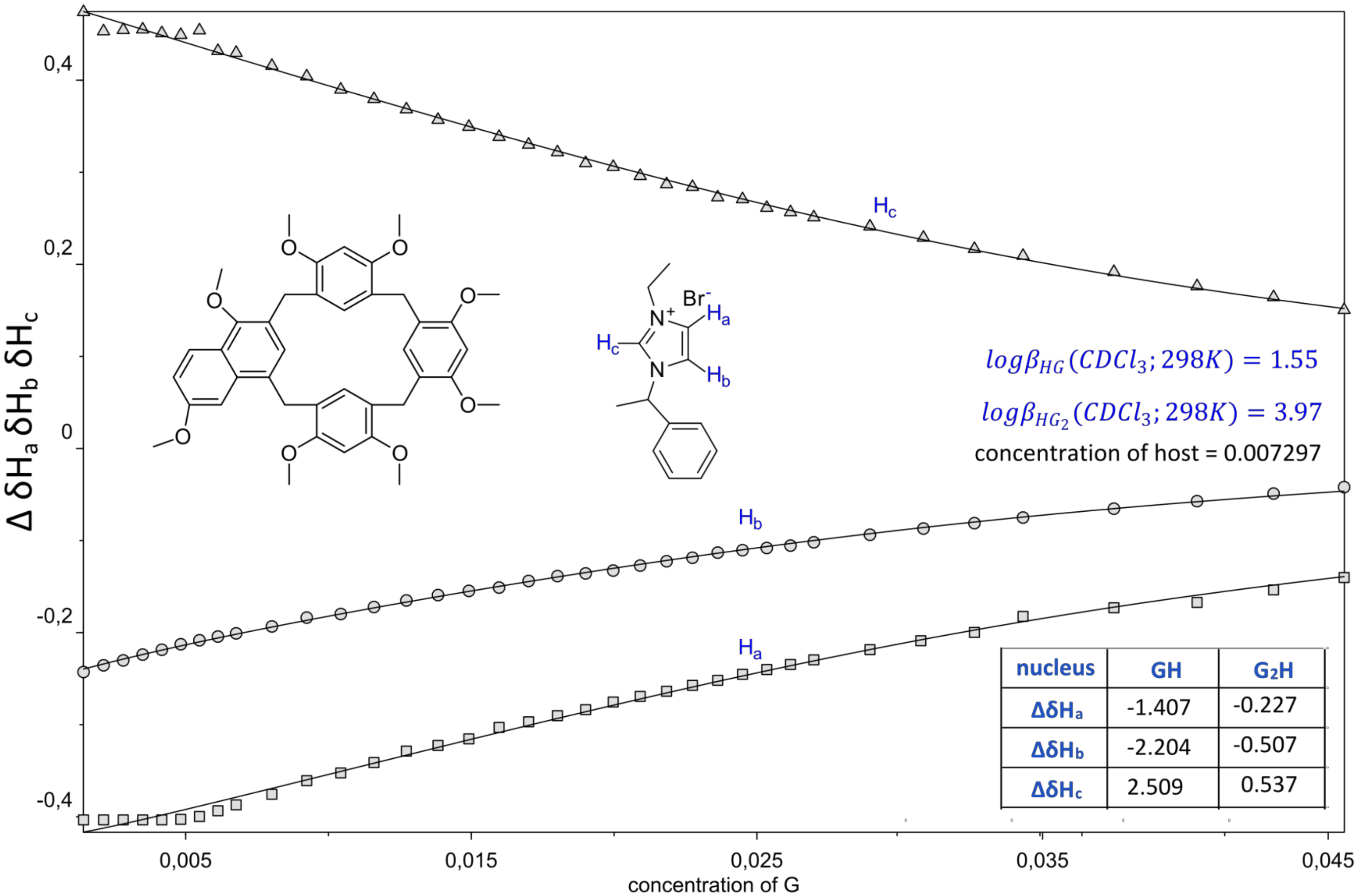
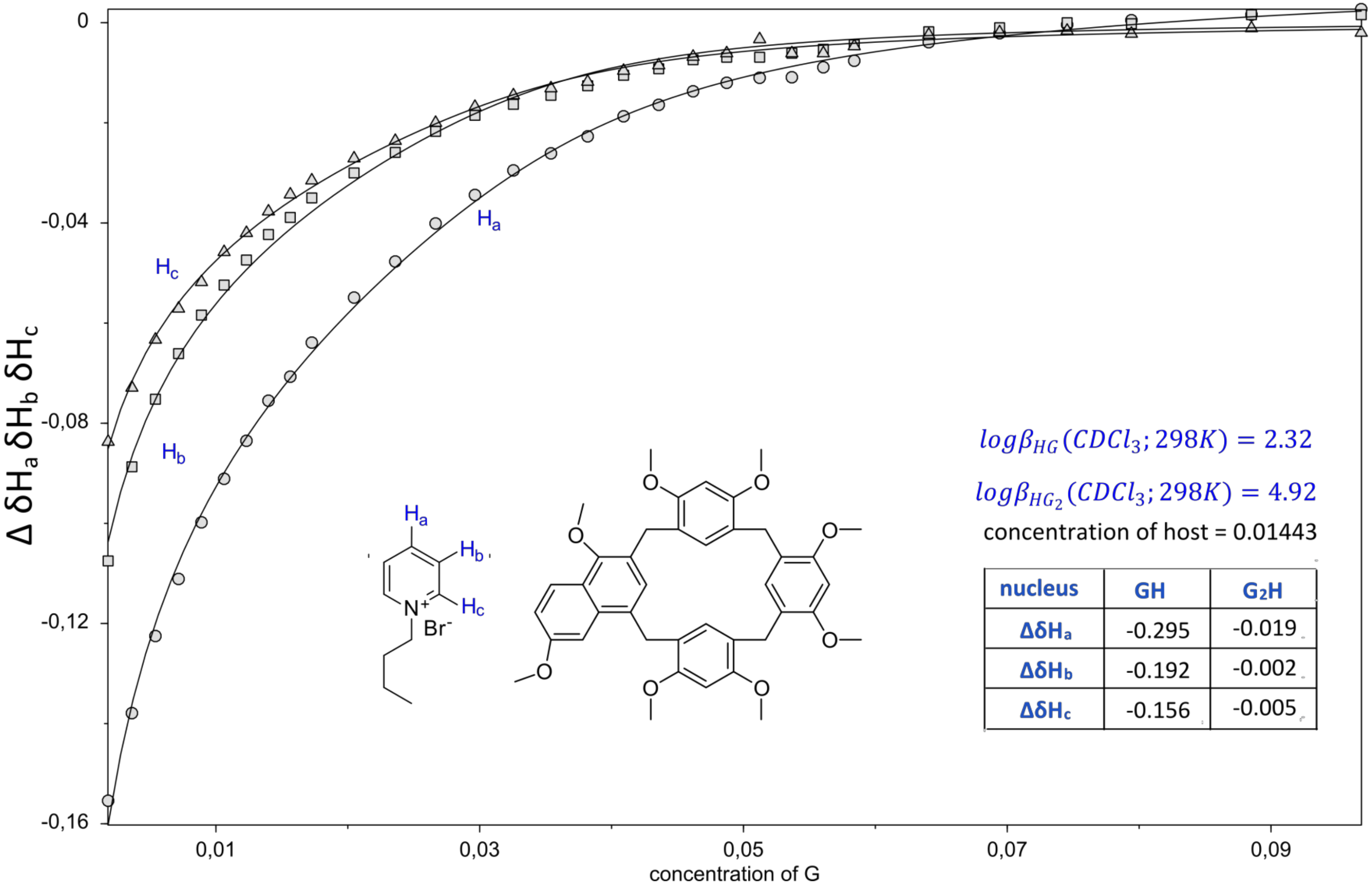


Figure S29: Titration of **5** by **2** ($CDCl_3$, 298K)



nucleus	GH	G_2H
$\Delta\delta H_a$	-0.295	-0.019
$\Delta\delta H_b$	-0.192	-0.002
$\Delta\delta H_c$	-0.156	-0.005

Figure S30: Titration of **6** by **2** ($CDCl_3$, 298K)

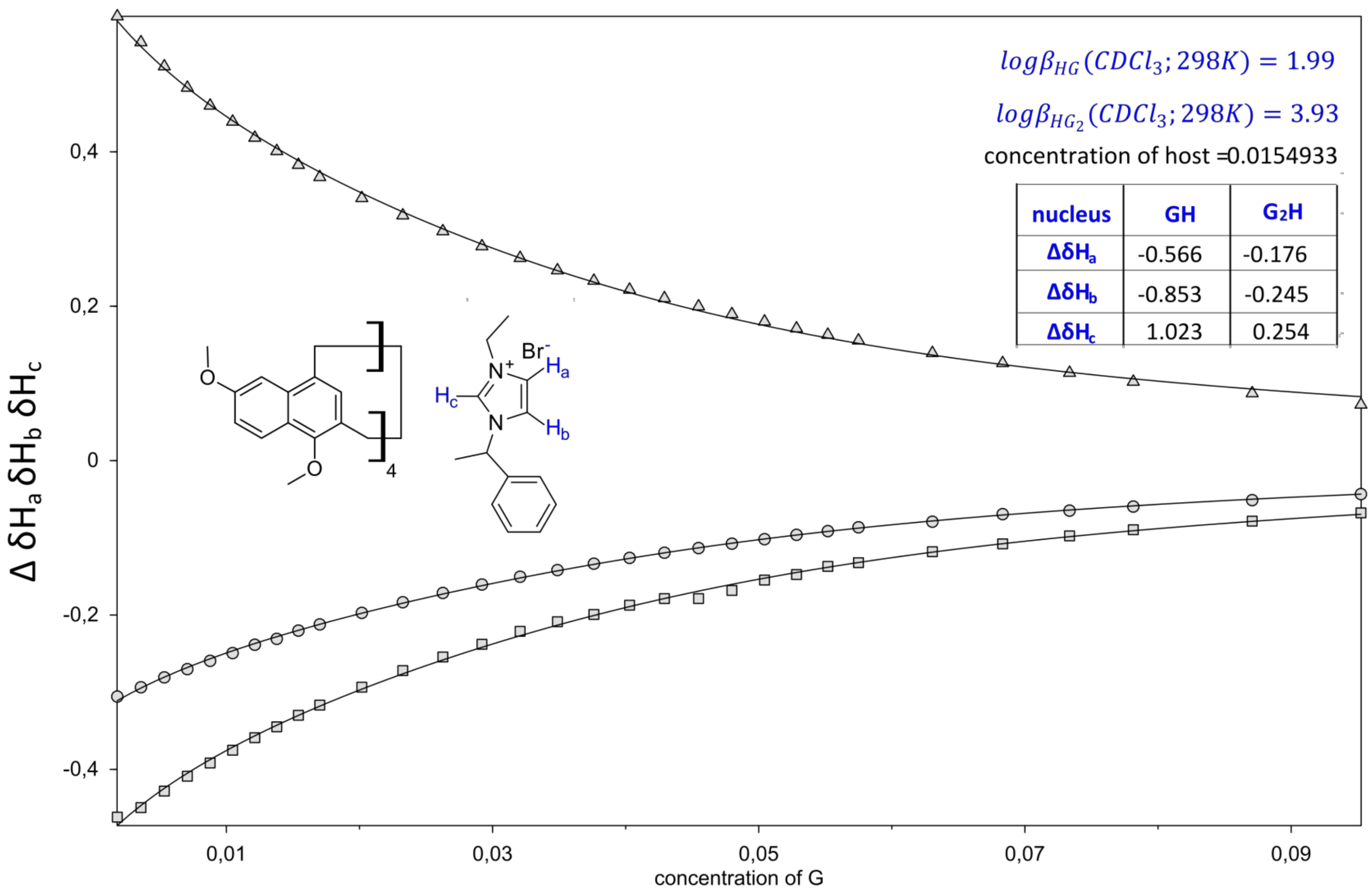
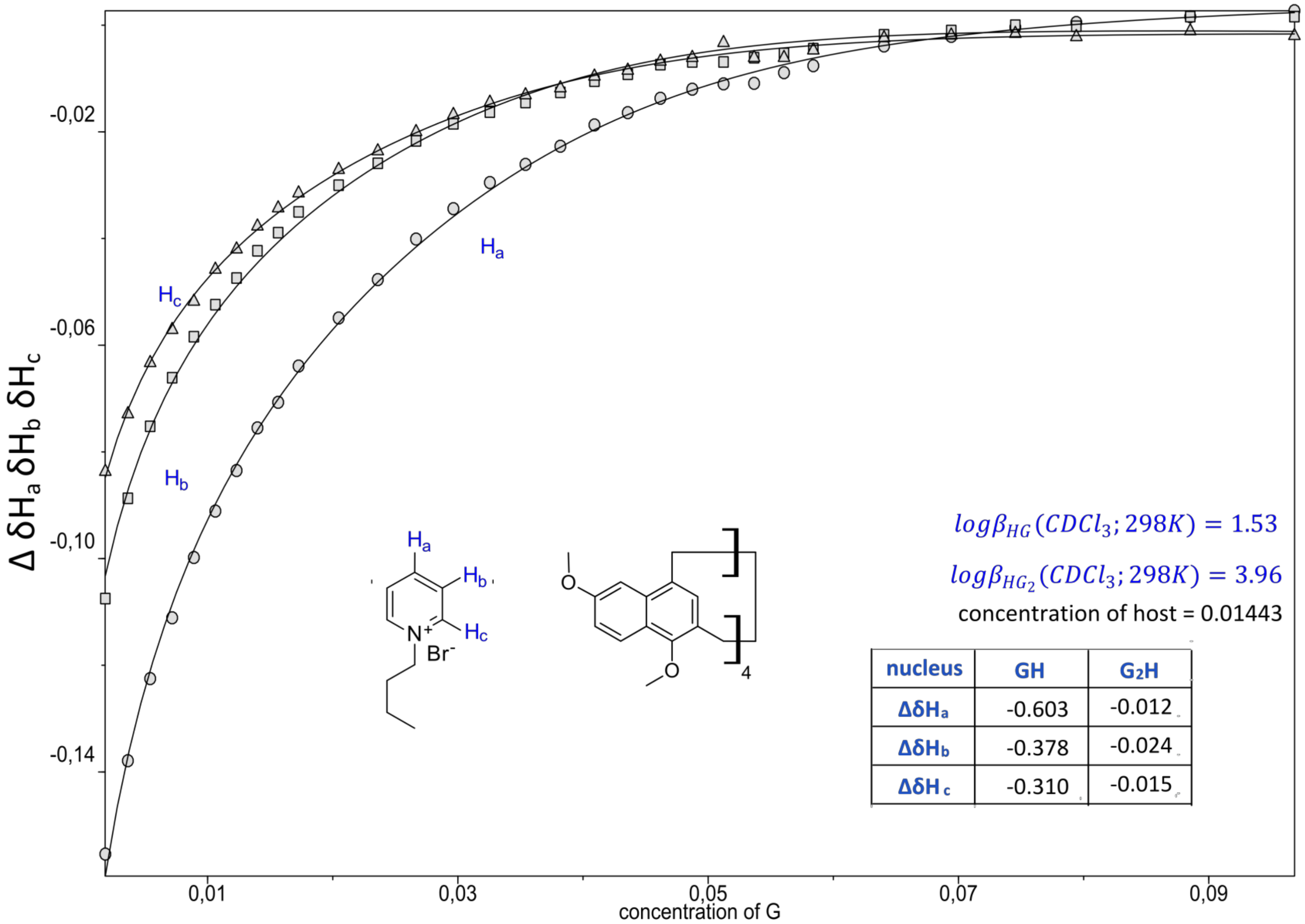
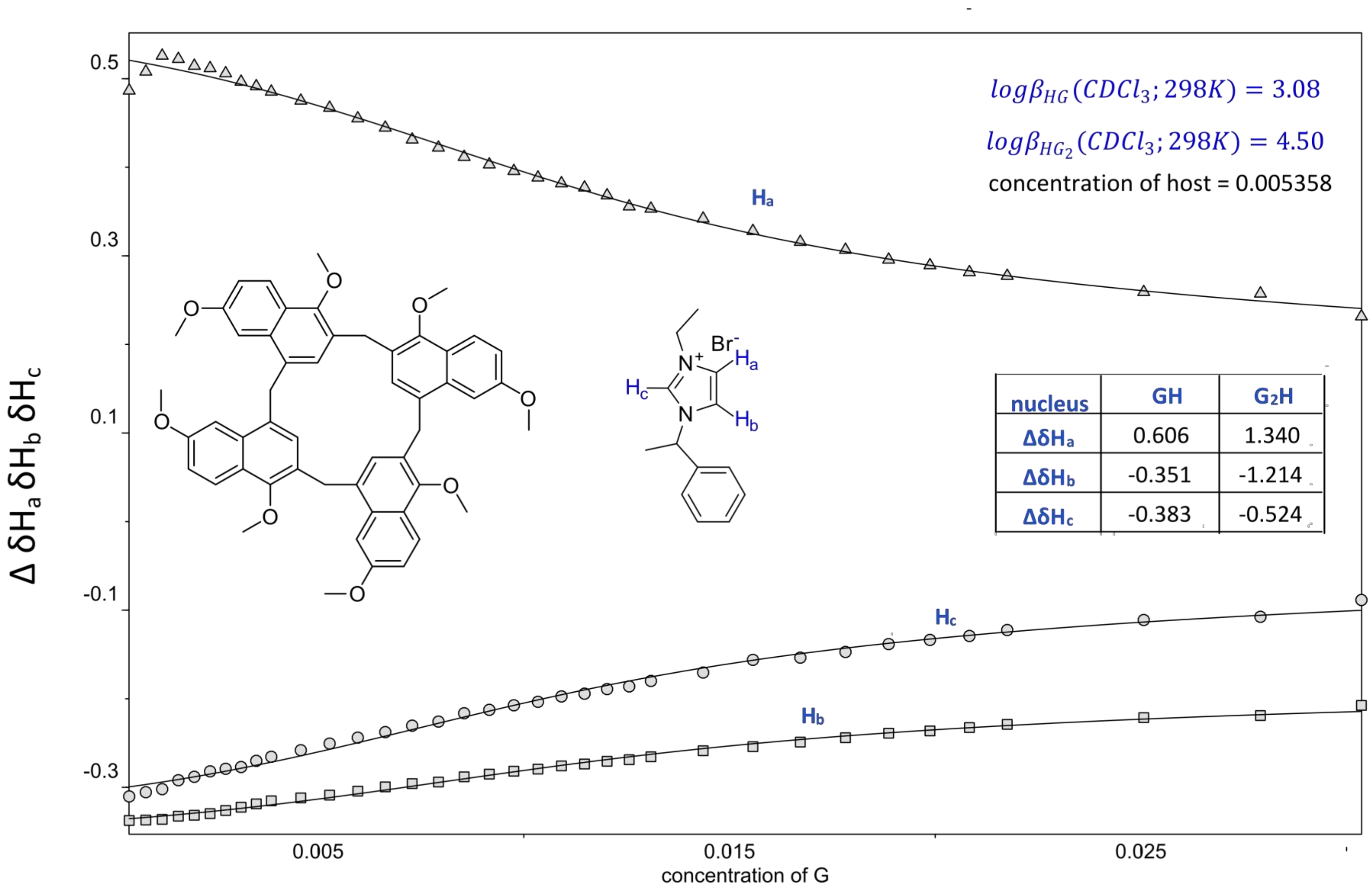


Figure S31: Titration of **5** by **3** ($CDCl_3$, 298K)



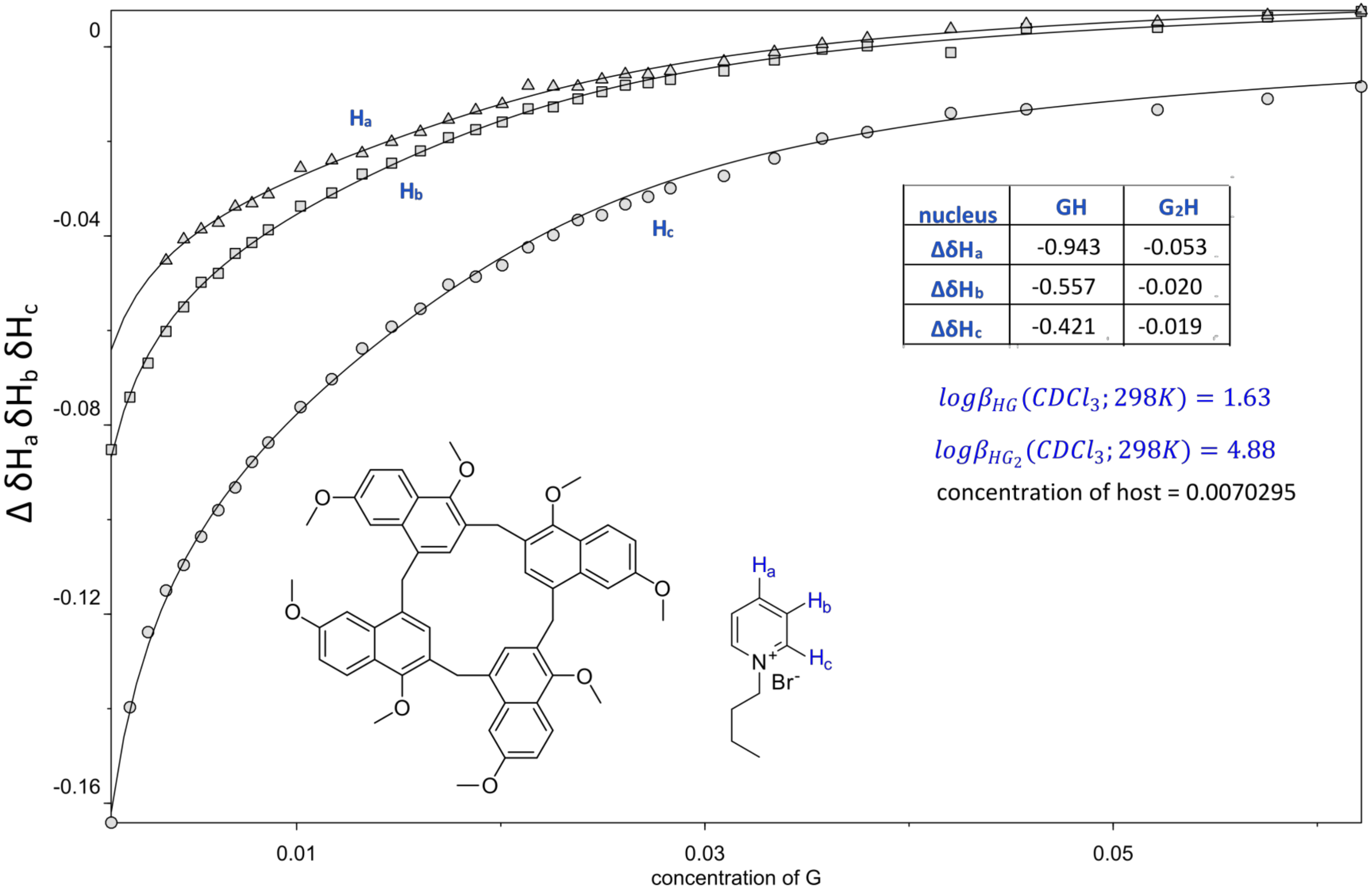
nucleus	GH	G_2H
$\Delta\delta H_a$	-0.603	-0.012
$\Delta\delta H_b$	-0.378	-0.024
$\Delta\delta H_c$	-0.310	-0.015

Figure S32: Titration of **6** by **3** ($CDCl_3$, 298K)



nucleus	GH	G_2H
$\Delta\delta H_a$	0.606	1.340
$\Delta\delta H_b$	-0.351	-1.214
$\Delta\delta H_c$	-0.383	-0.524

Figure S33: Titration of **5** by **4** ($CDCl_3$, 298K)



nucleus	GH	G_2H
$\Delta\delta H_a$	-0.943	-0.053
$\Delta\delta H_b$	-0.557	-0.020
$\Delta\delta H_c$	-0.421	-0.019

Figure S34: Titration of **6** by **4** ($CDCl_3$, 298K)

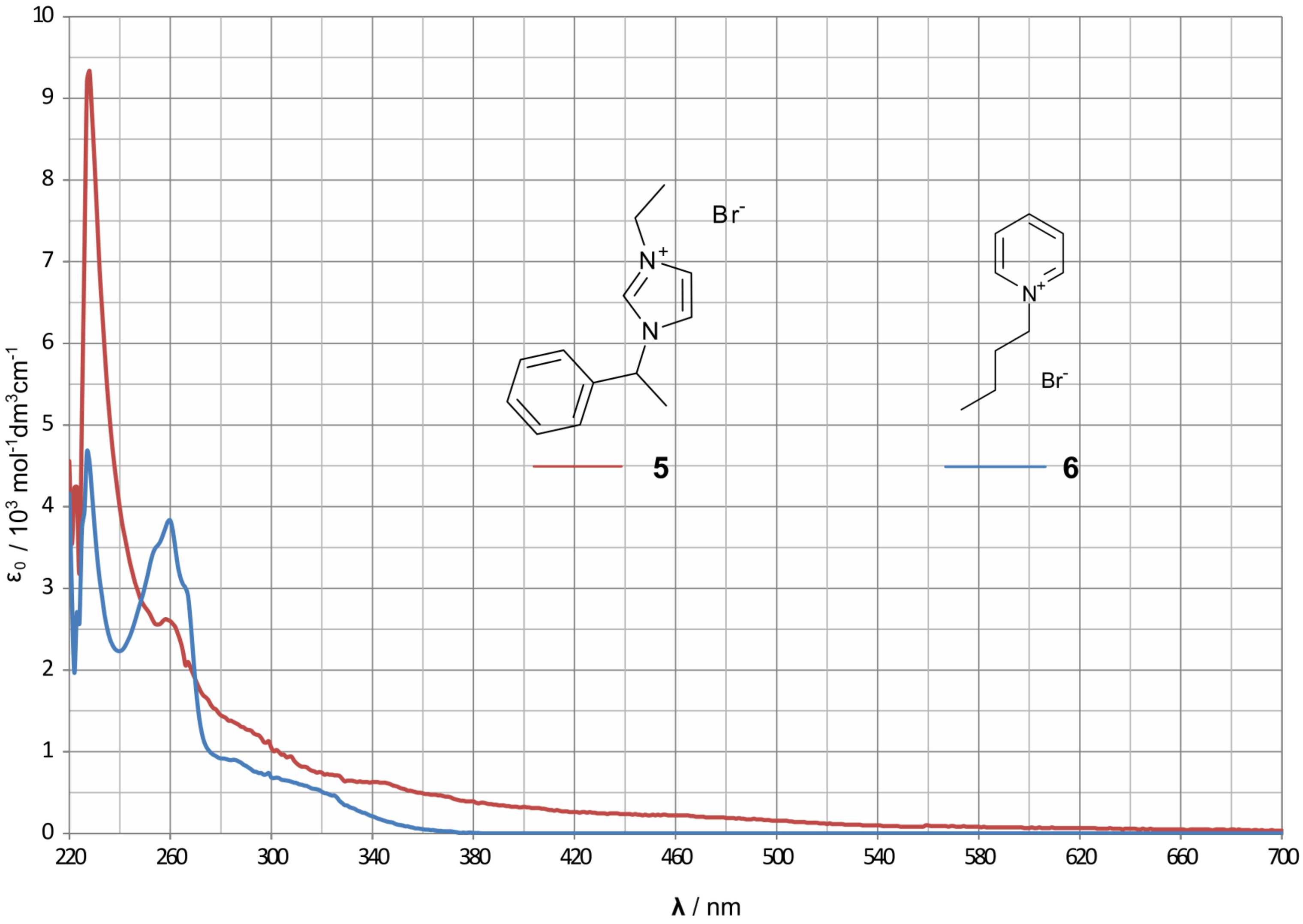


Figure S35: UV-Vis spectra of guests **5** and **6** (CH_2Cl_2)

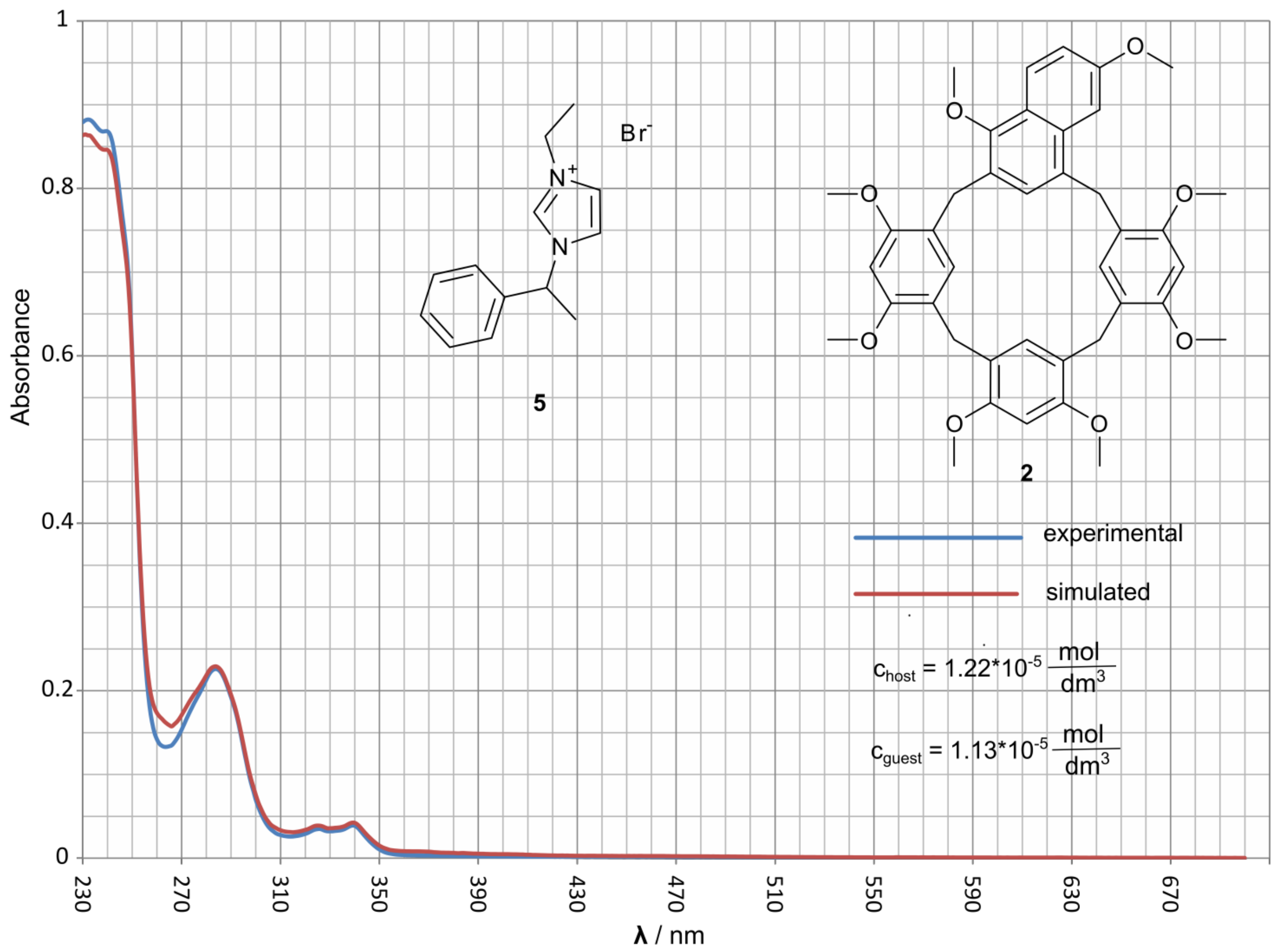


Figure S36: UV-Vis spectra for complexation of **5** by **2** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration).

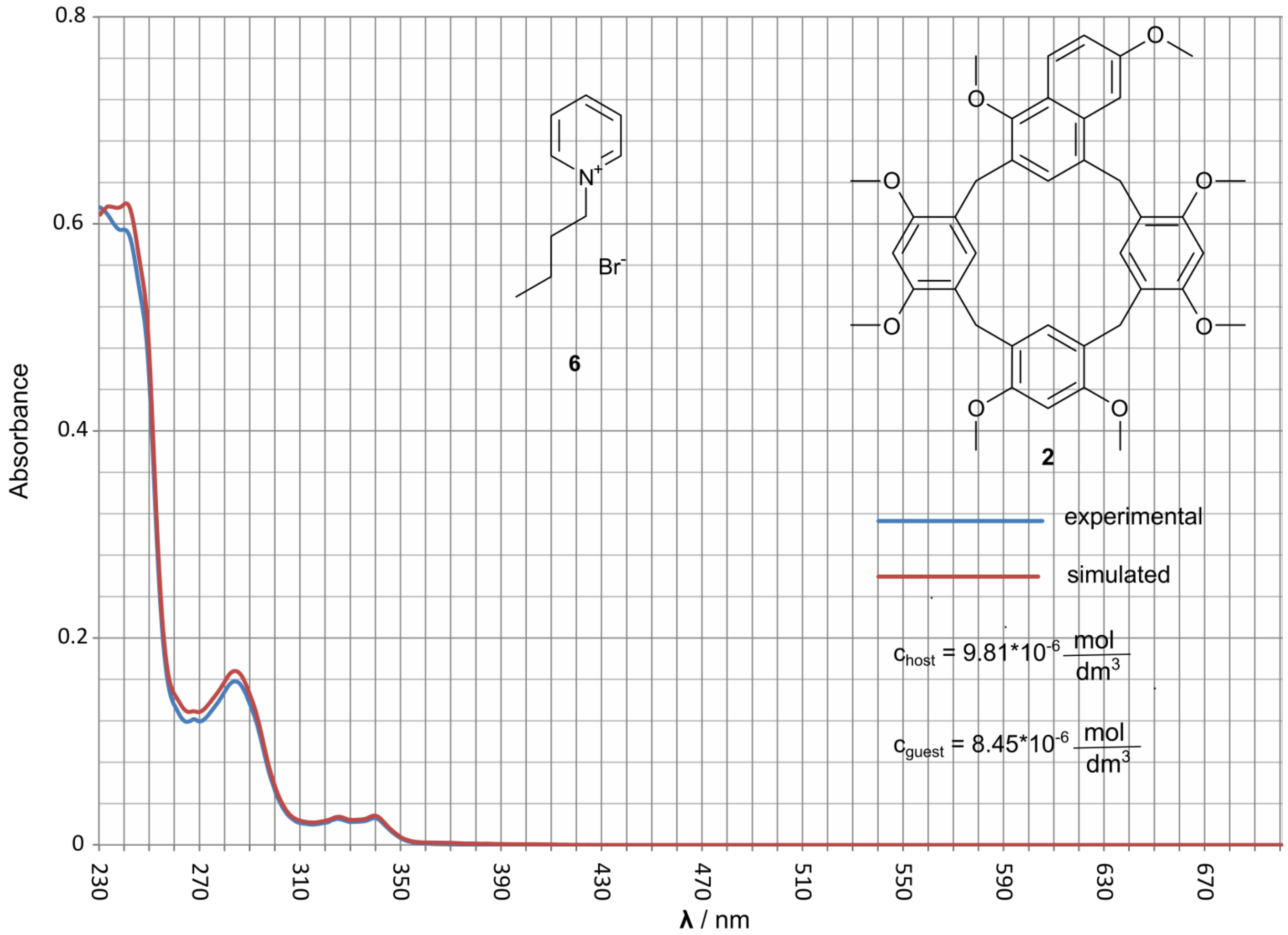


Figure 37: UV-Vis spectra for complexation of **6** by **2** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration)

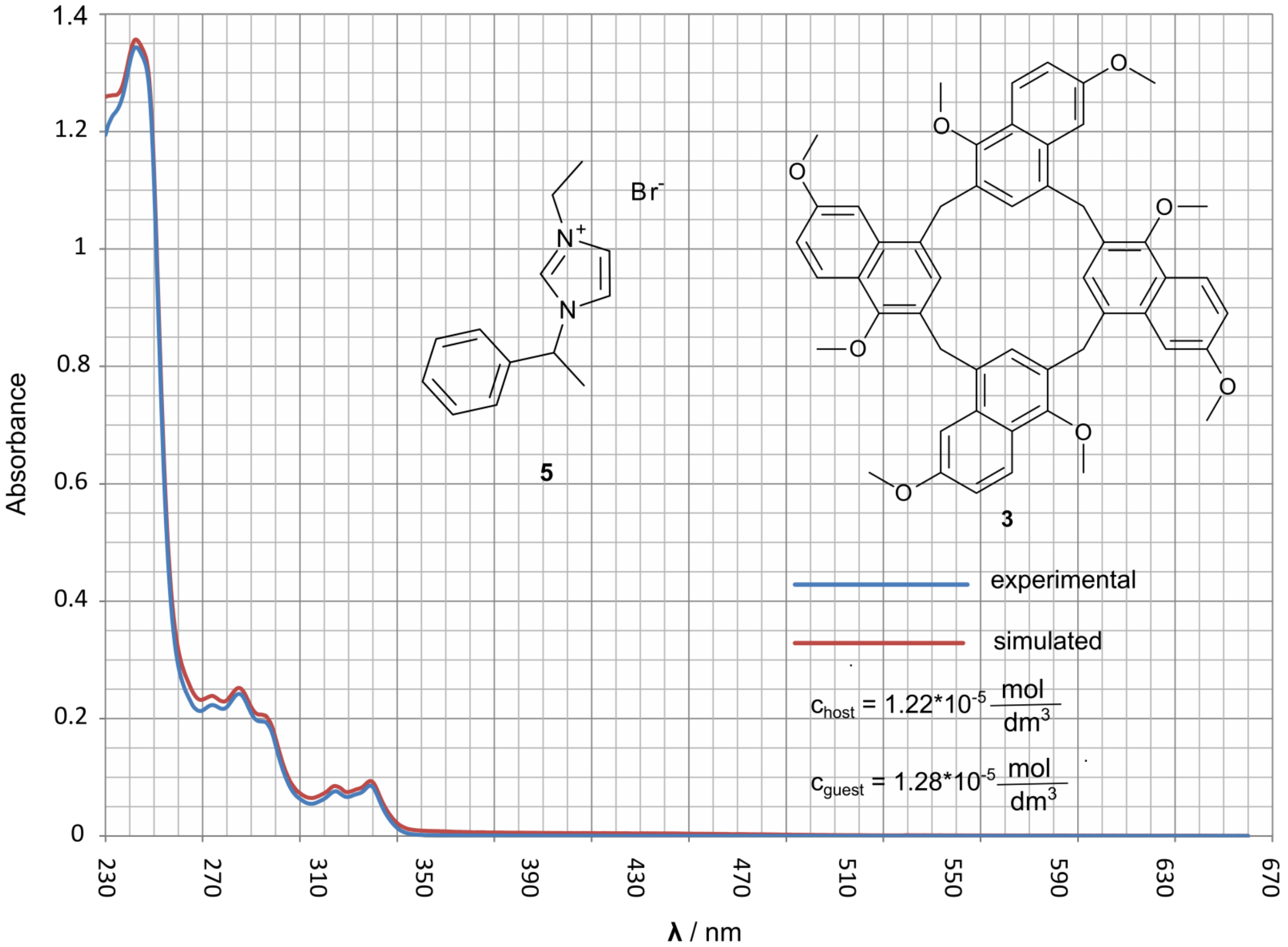


Figure S38: UV-Vis spectra for complexation of **5** by **3** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration)

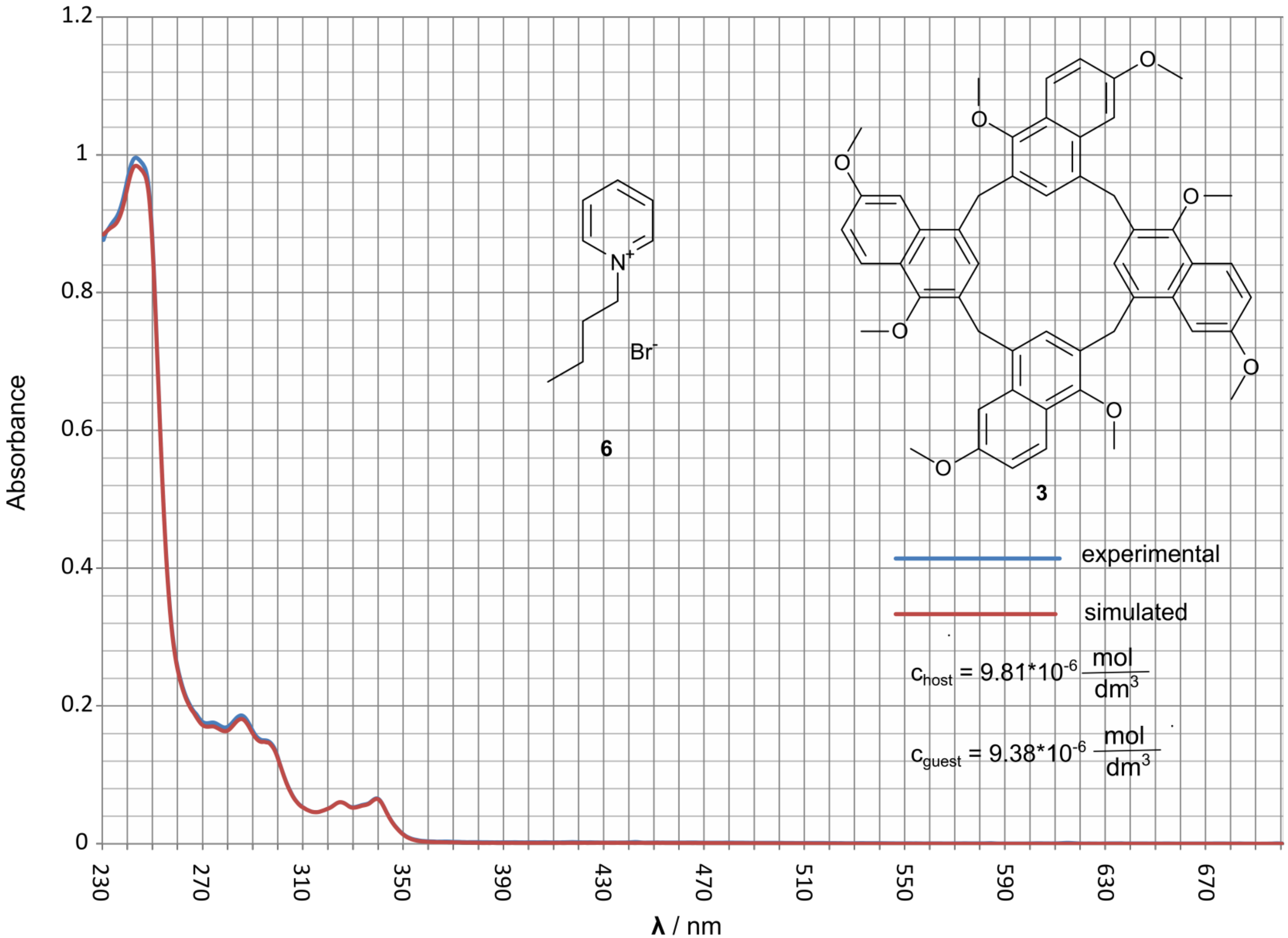


Figure S39: UV-Vis spectra for complexation of **6** by **3** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration)

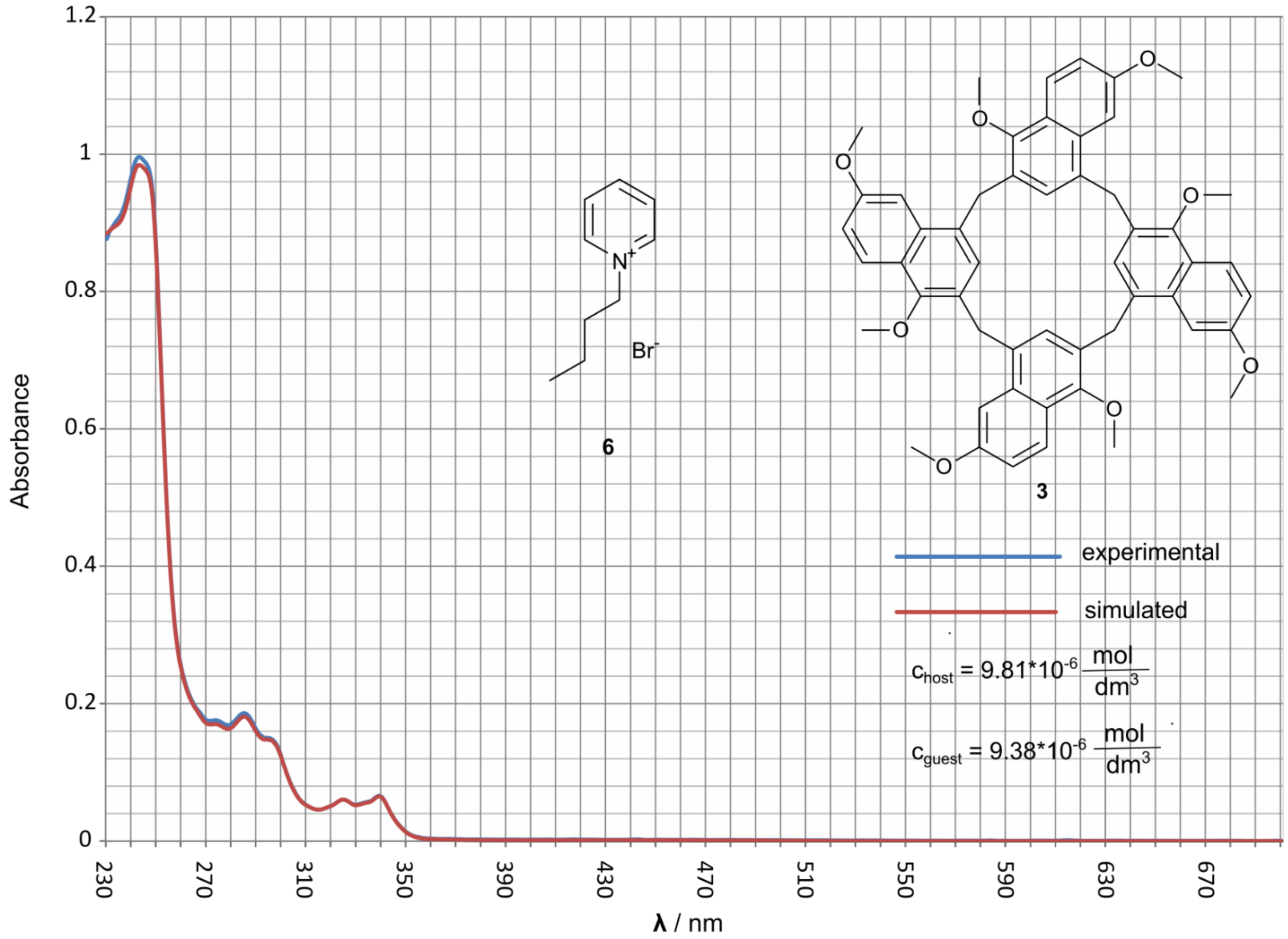


Figure 40: UV-Vis spectra for complexation of **6** by **3** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration)

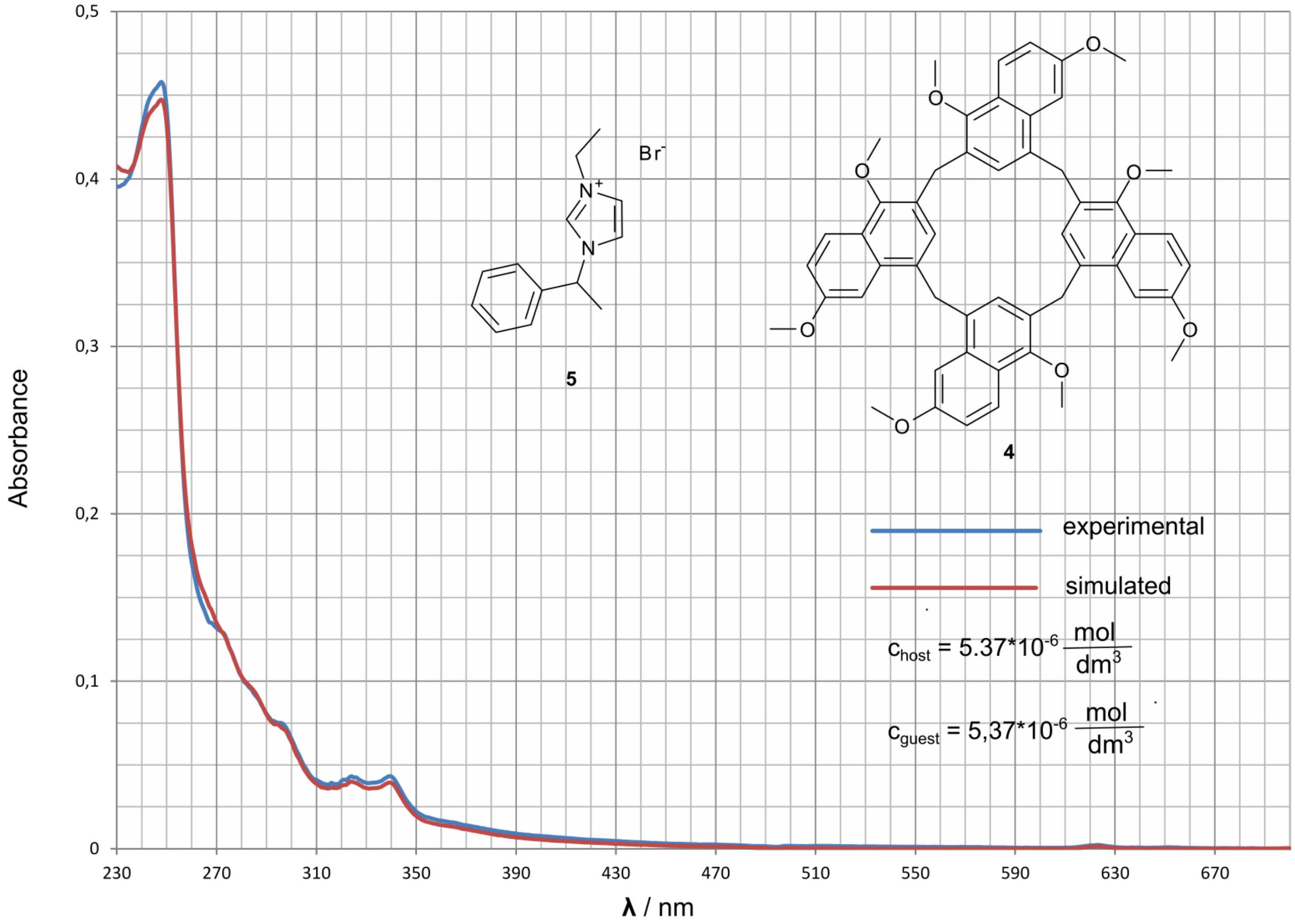


Figure 41: UV-Vis spectra for complexation of **5** by **4** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration)

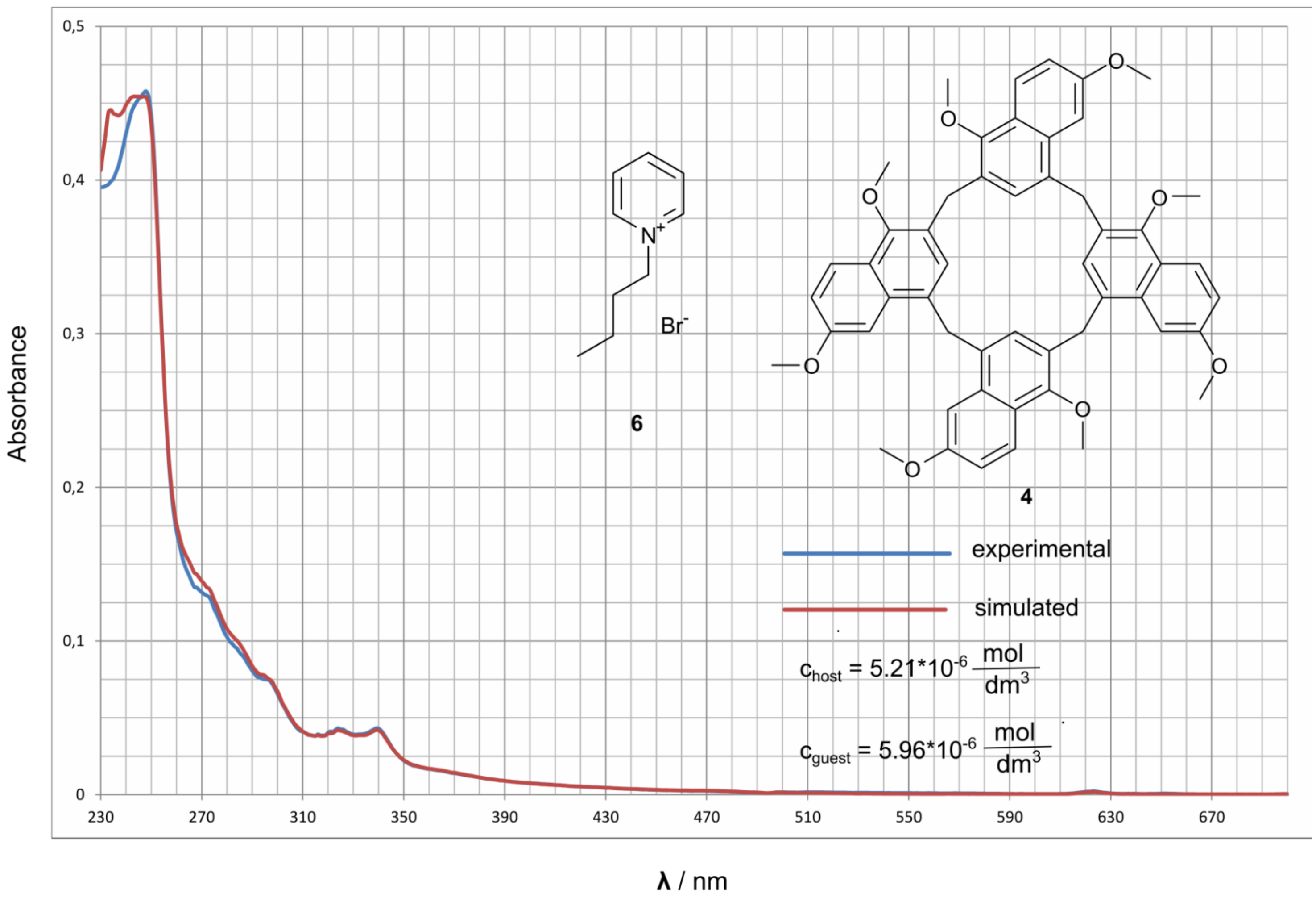


Figure 42: UV-Vis spectra for complexation of **6** by **4** in CH_2Cl_2. Simulated spectra are obtained by addition of UV-Vis a spectra of the host and the guest (corrected for the experimental concentration)

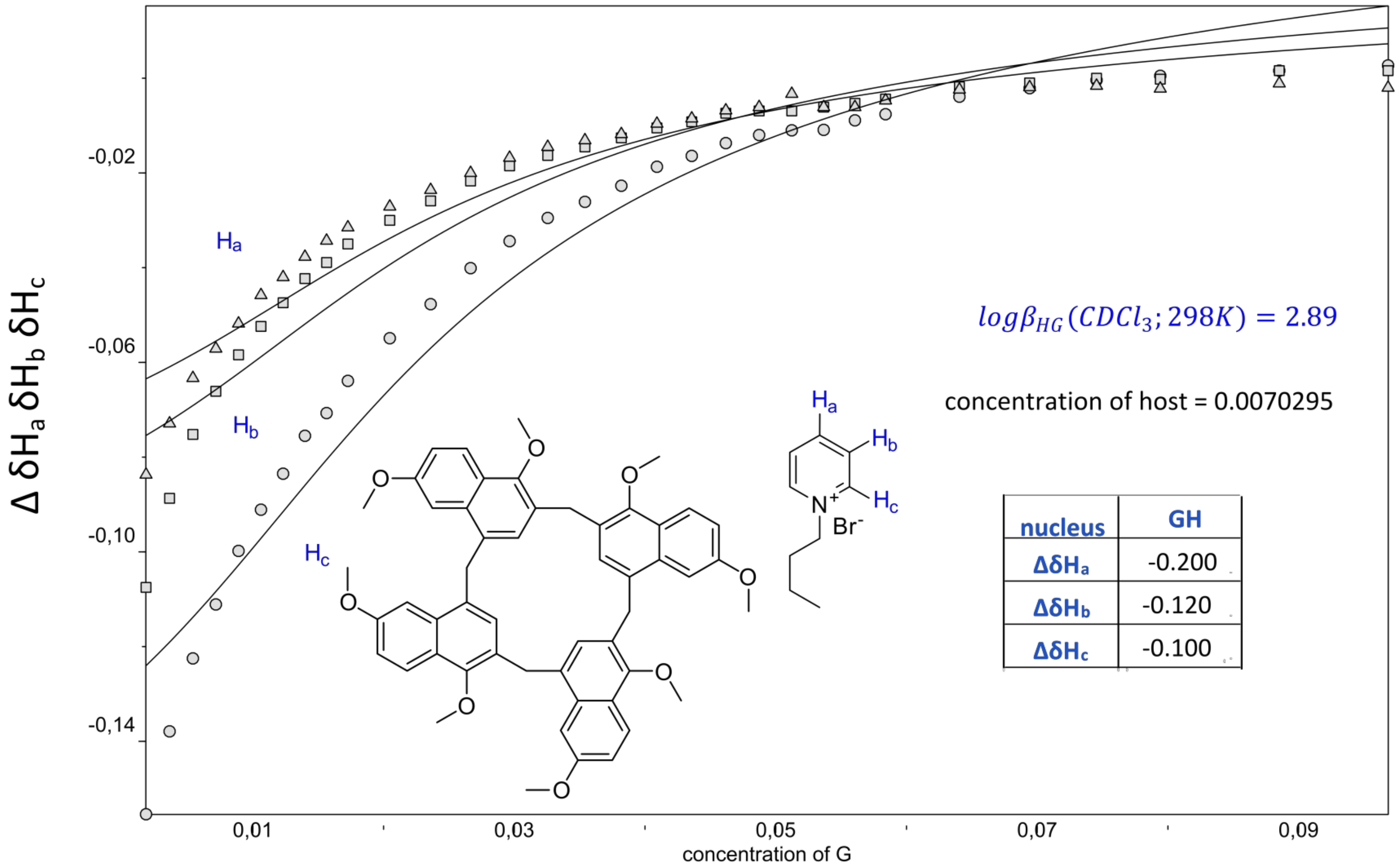


Figure 43 Exemplary fitting of titration data using incorrect 1:1 model (shows systematic errors).