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Supplementary data for

Calix[4]arenes Containing Ureido Functionality on the Lower Rim as Highly Efficient Receptors for Anion Recognition

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1. Spectra of compounds

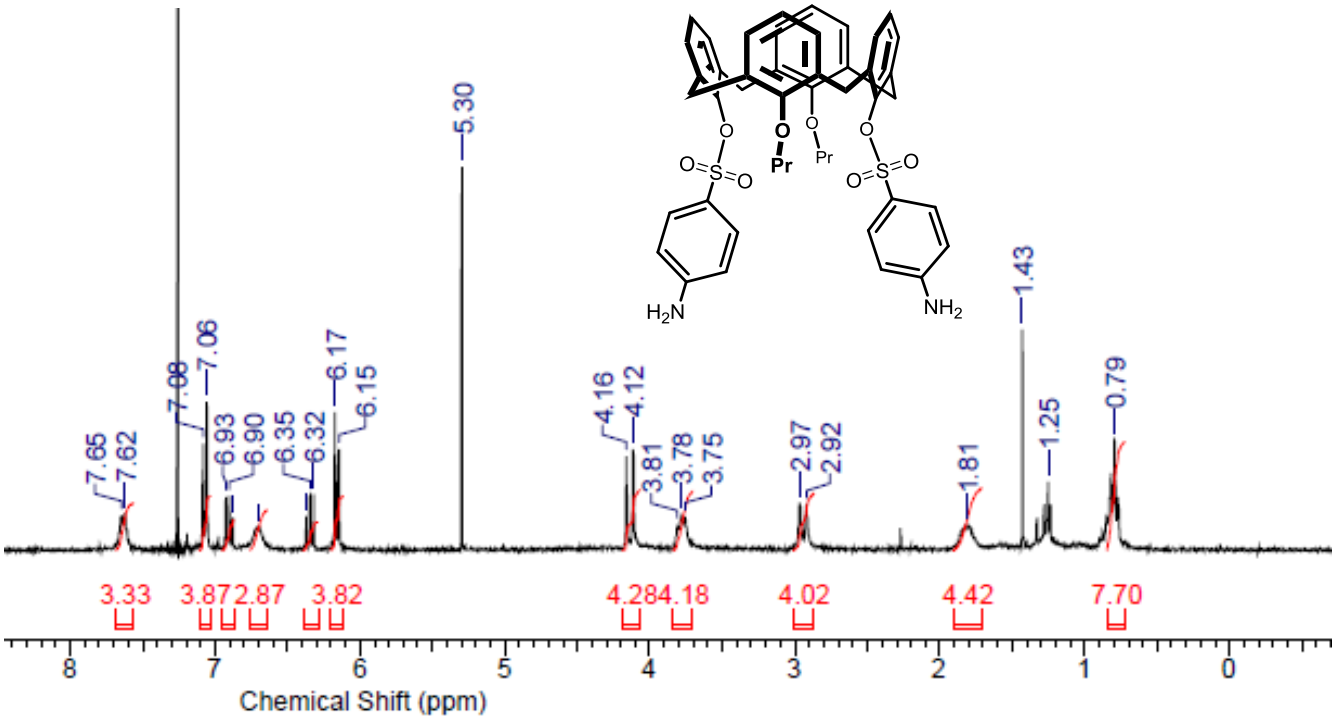


Figure 1: ^{1}H NMR spectrum of compound **3** (300 MHz, $CDCl_3$, 298 K).

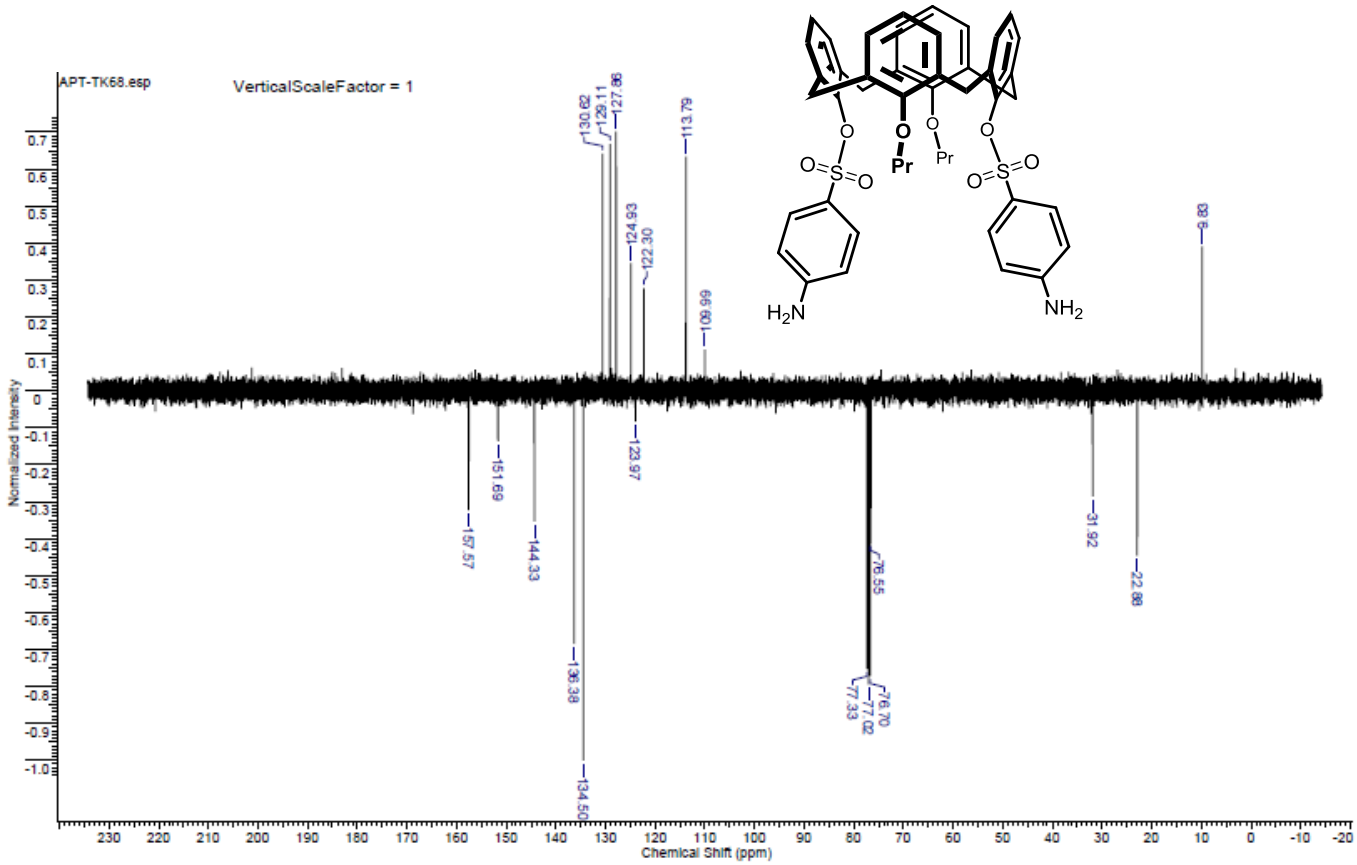


Figure 2: ^{13}C NMR (APT) spectrum of compound **3** (75 MHz, $CDCl_3$, 298 K).

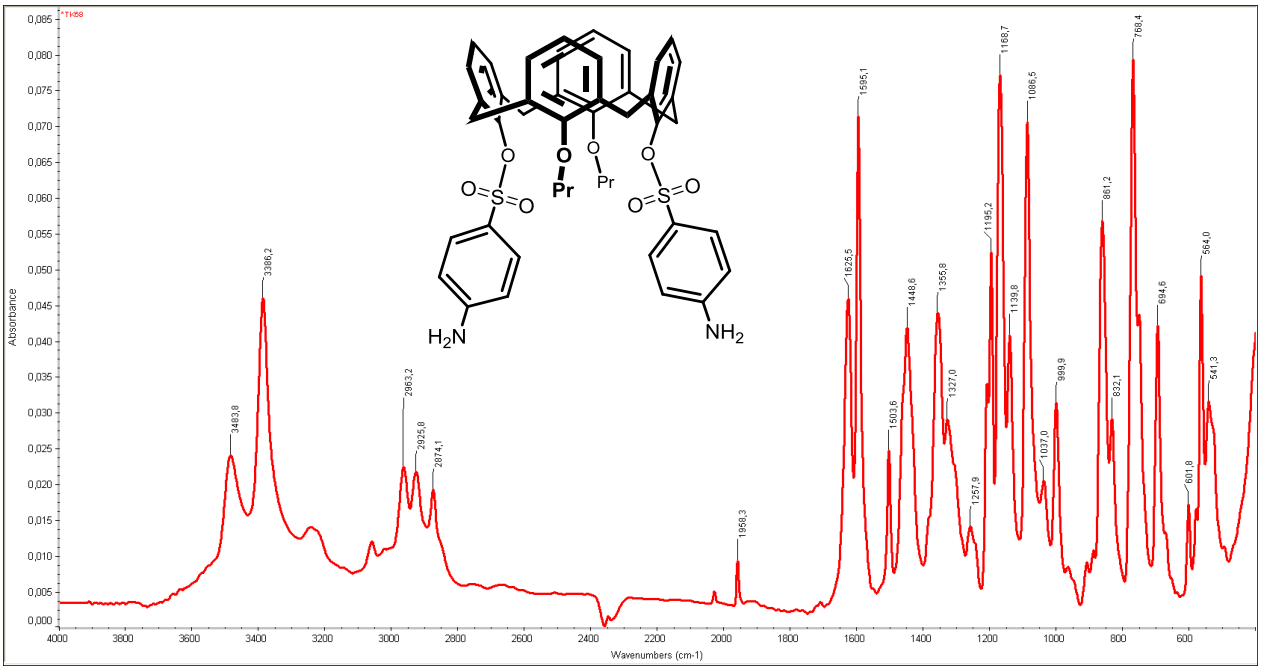


Figure 3: IR spectrum of compound **3** (KBr).

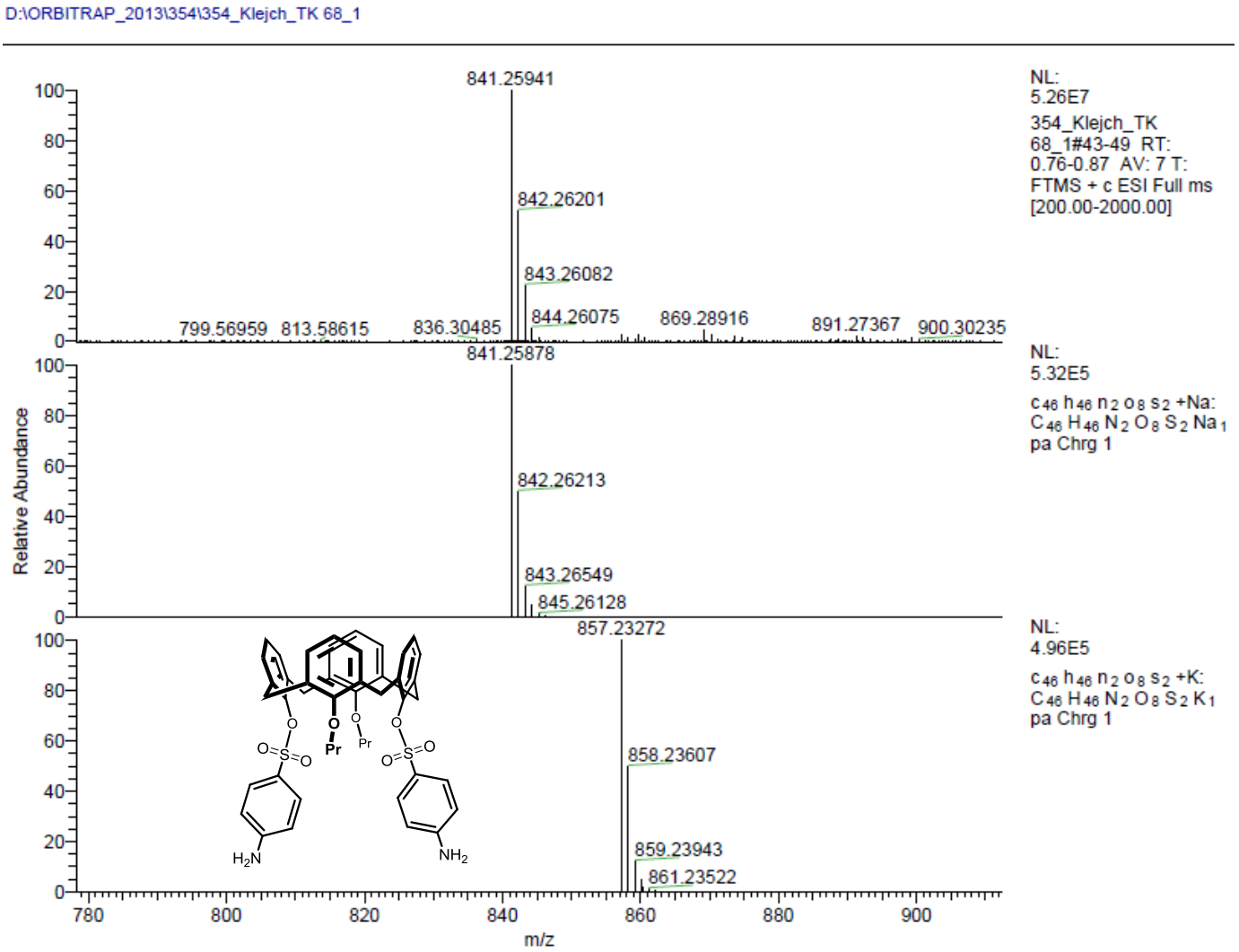


Figure 4: HRMS spectrum of compound **3** (orbitrap).

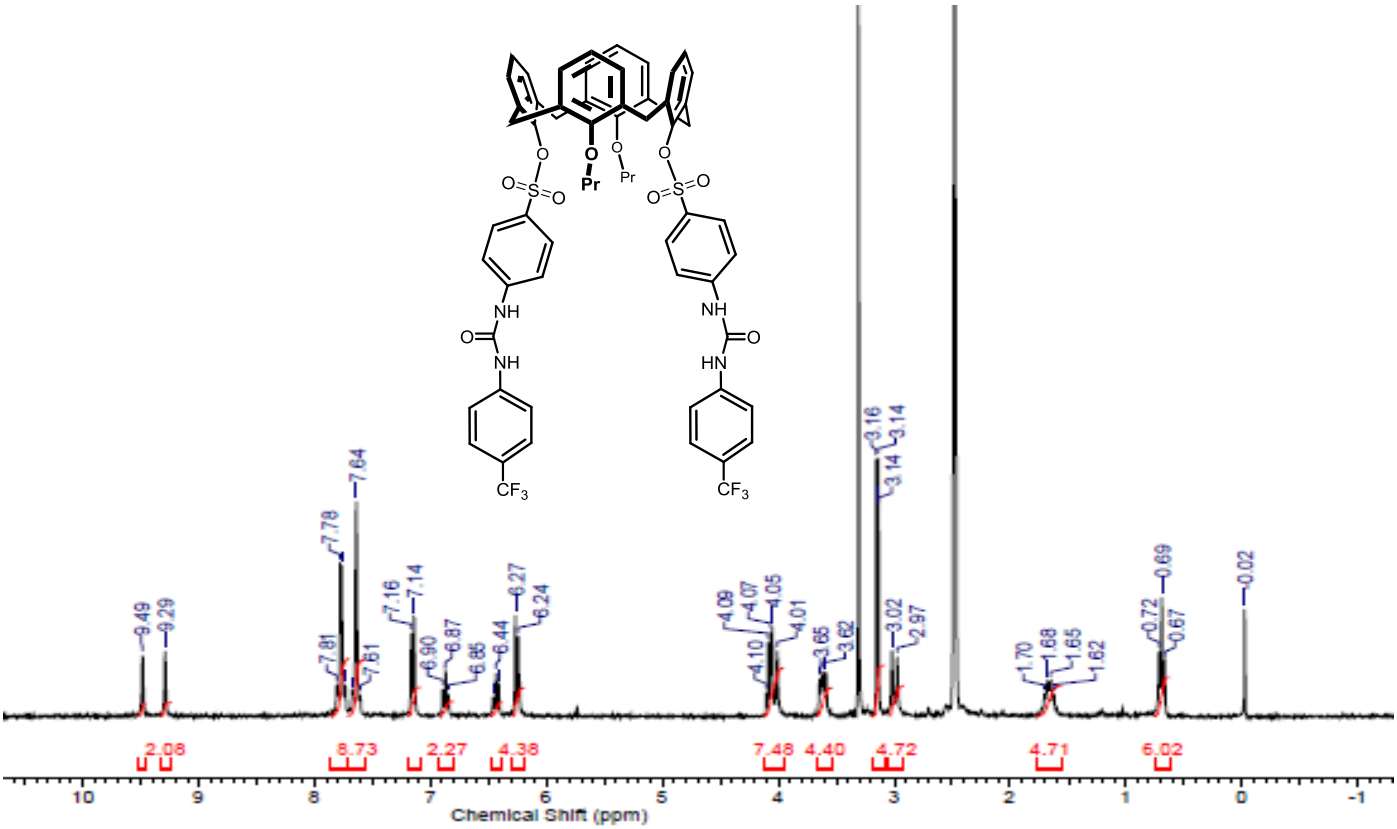


Figure 5: ^{1}H NMR spectrum of compound **4a** (400 MHz, DMSO-d_6, 298 K).

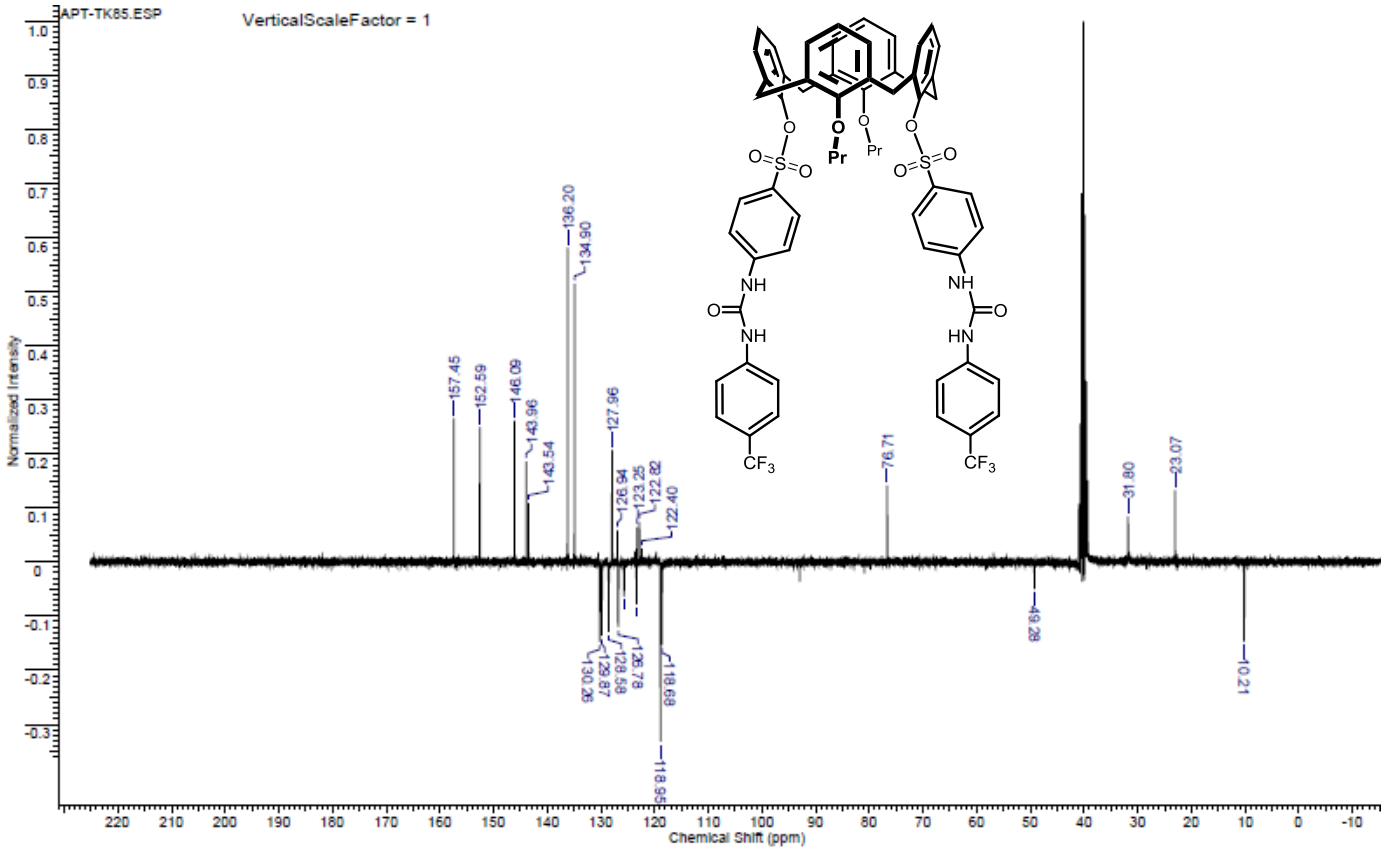


Figure 6: ^{13}C NMR (APT) spectrum of compound **4a** (100 MHz, DMSO-d_6, 298 K).

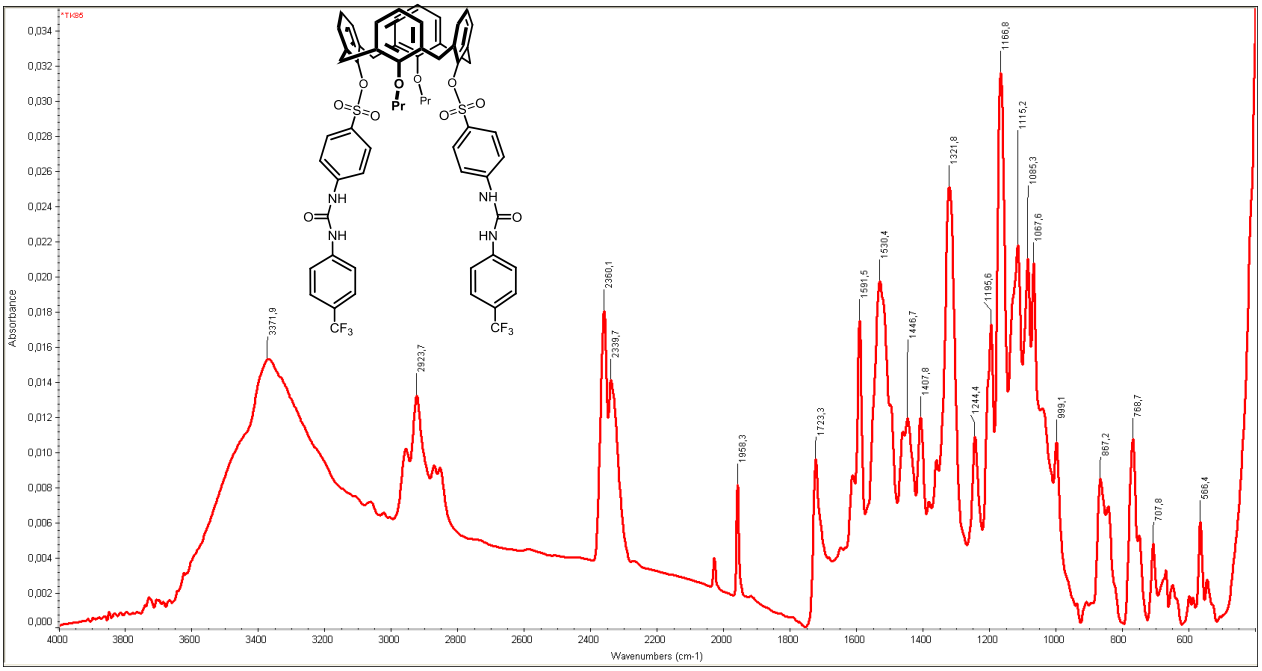


Figure 7: IR spectrum of compound **4a** (KBr).

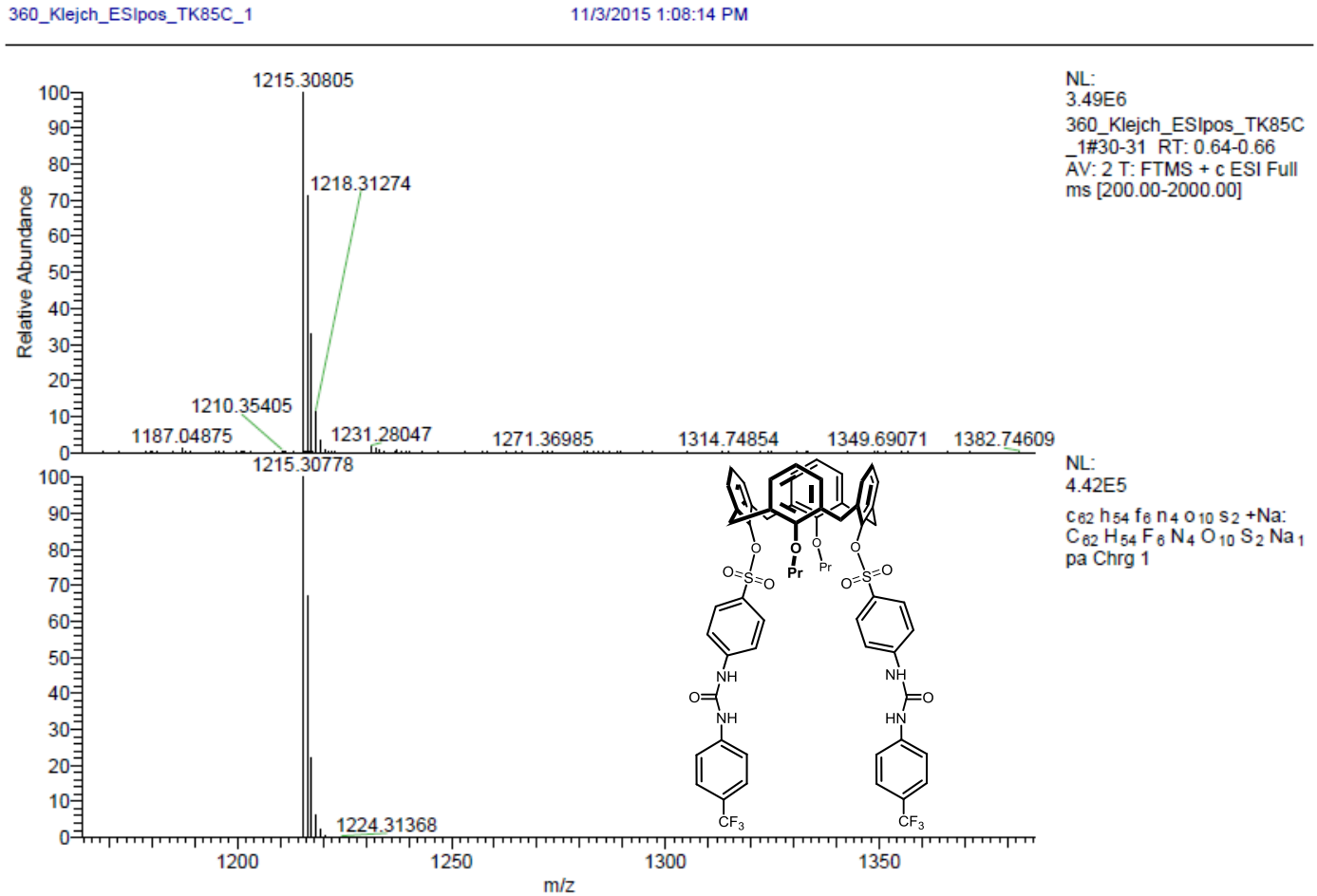


Figure 8: HRMS spectrum of compound **4a** (orbitrap).

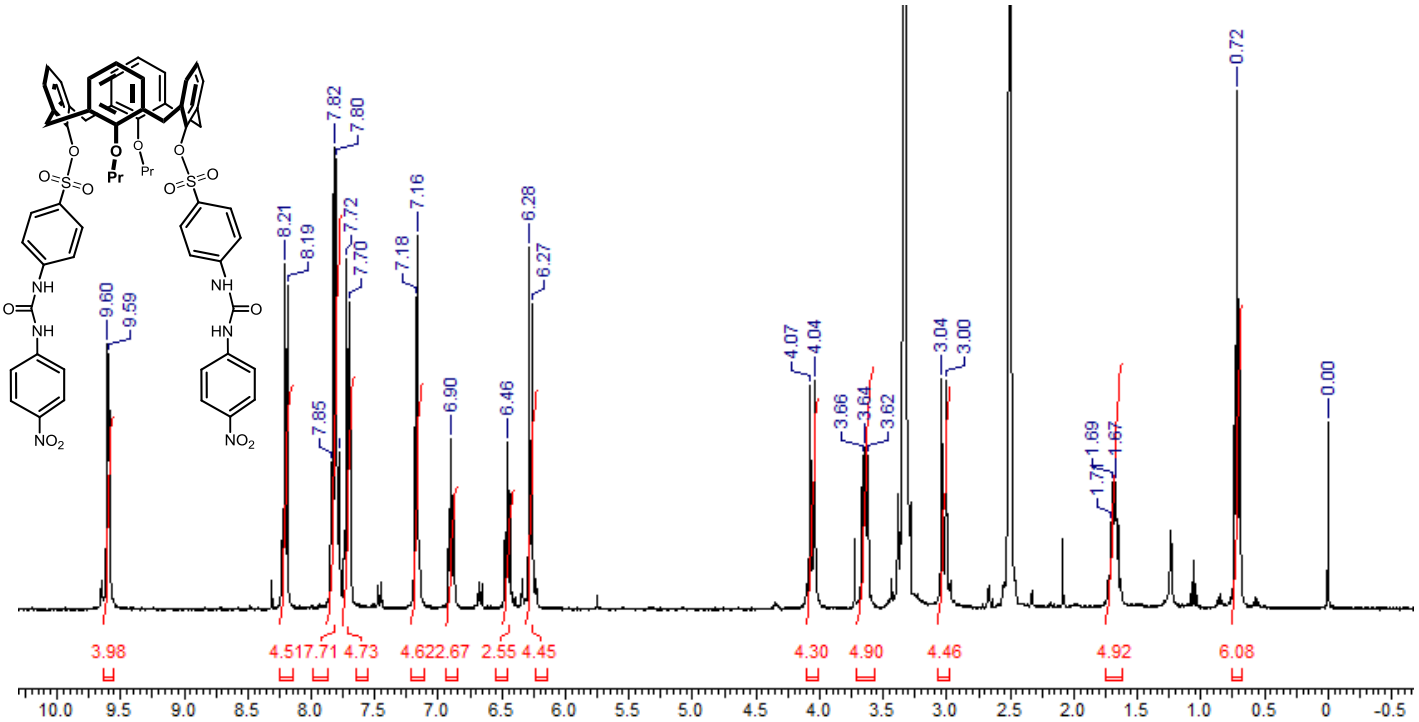


Figure 9: ^{1}H NMR spectrum of compound **4b** (400 MHz, DMSO-d_6, 298 K).

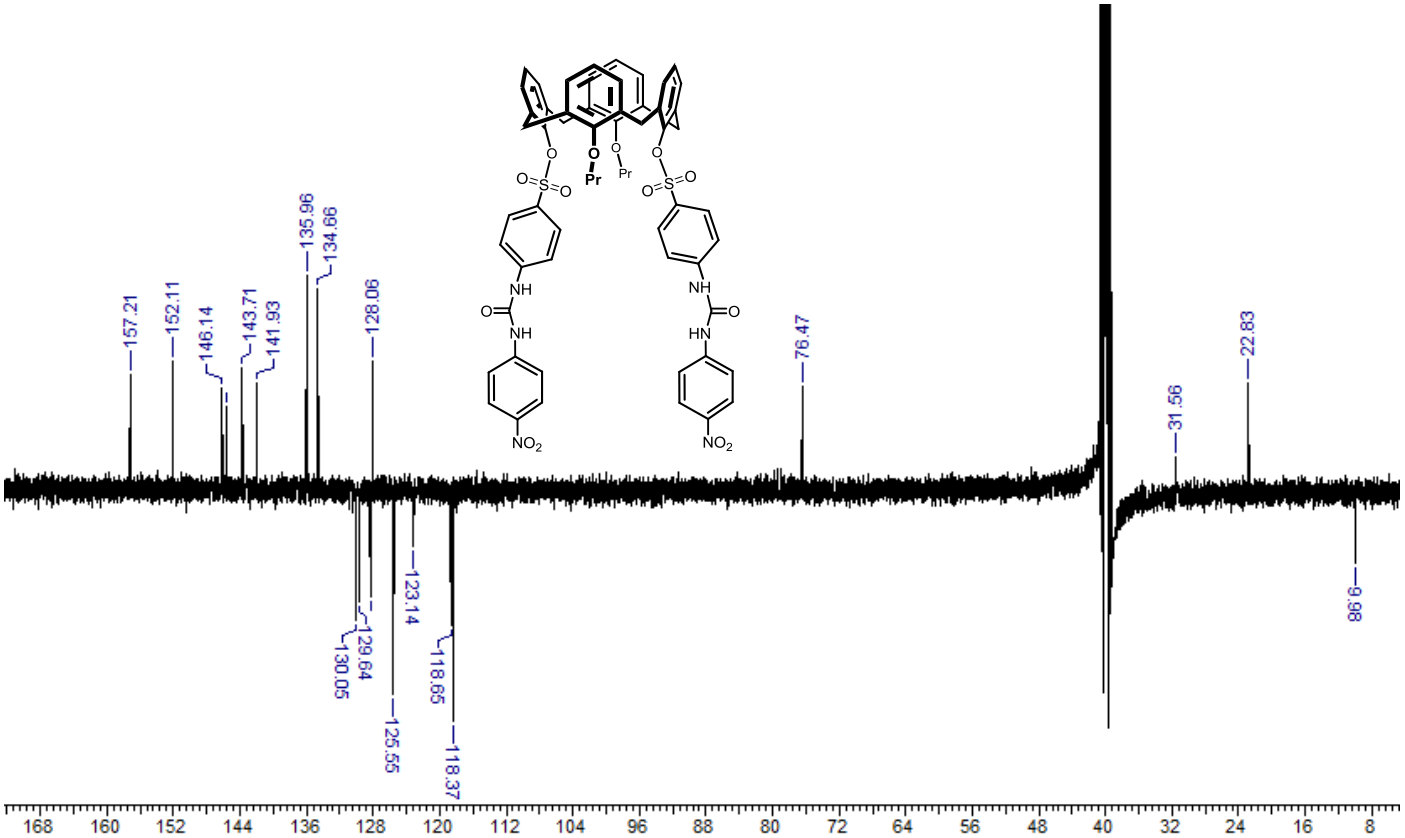


Figure 10: ^{13}C NMR (APT) spectrum of compound **4b** (100 MHz, DMSO-d_6, 298 K).

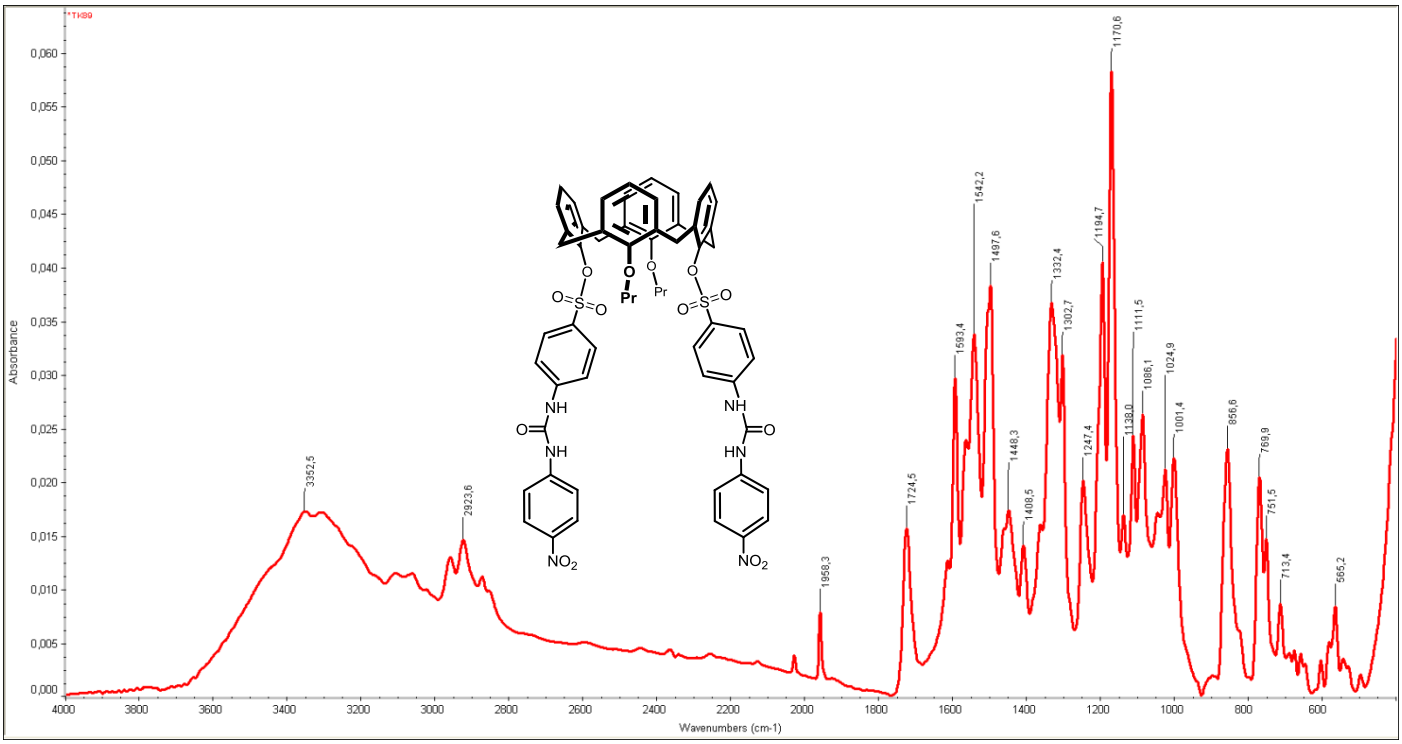


Figure 11: IR spectrum of compound **4b** (KBr).

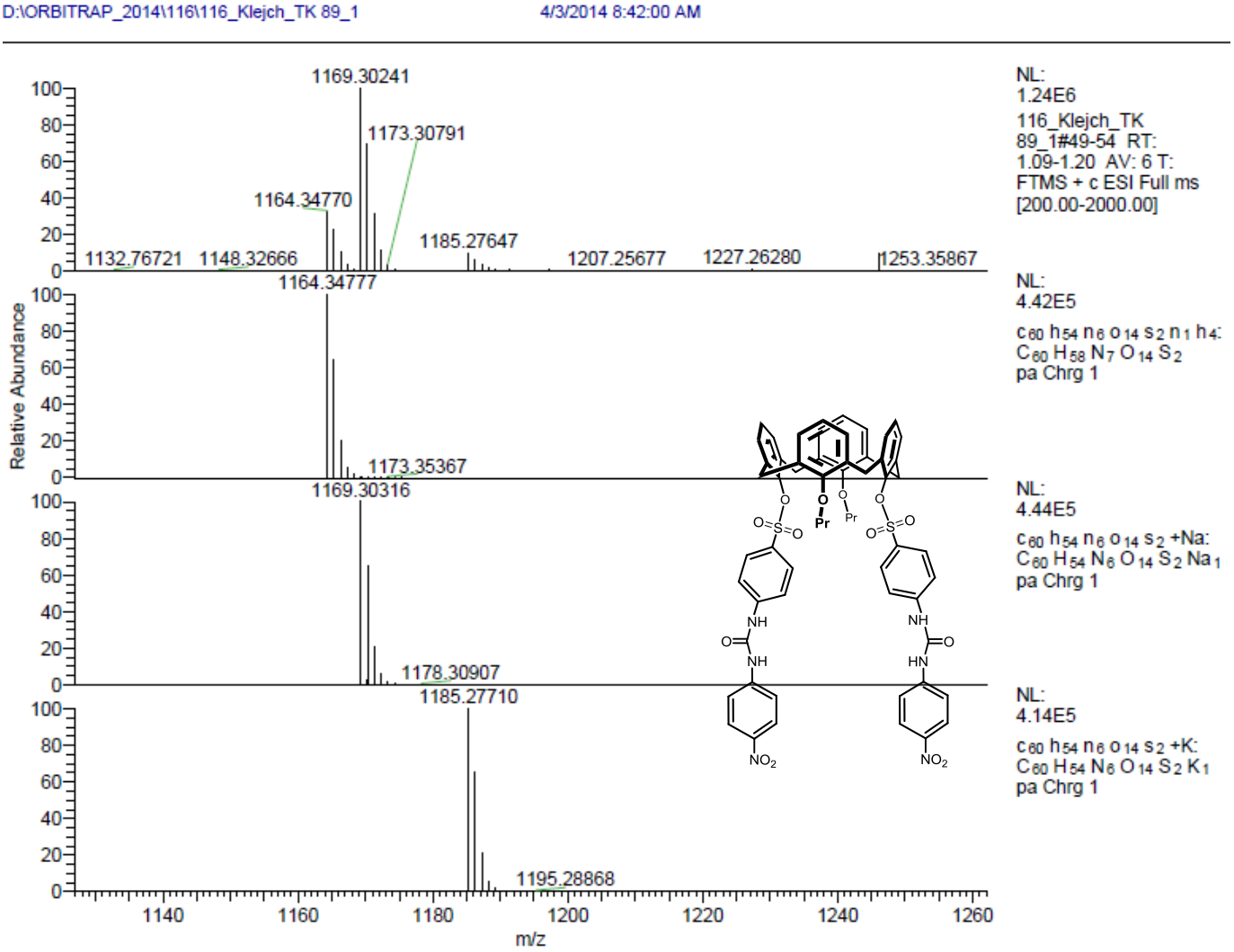


Figure 12: HRMS spectrum of compound **4b** (orbitrap).

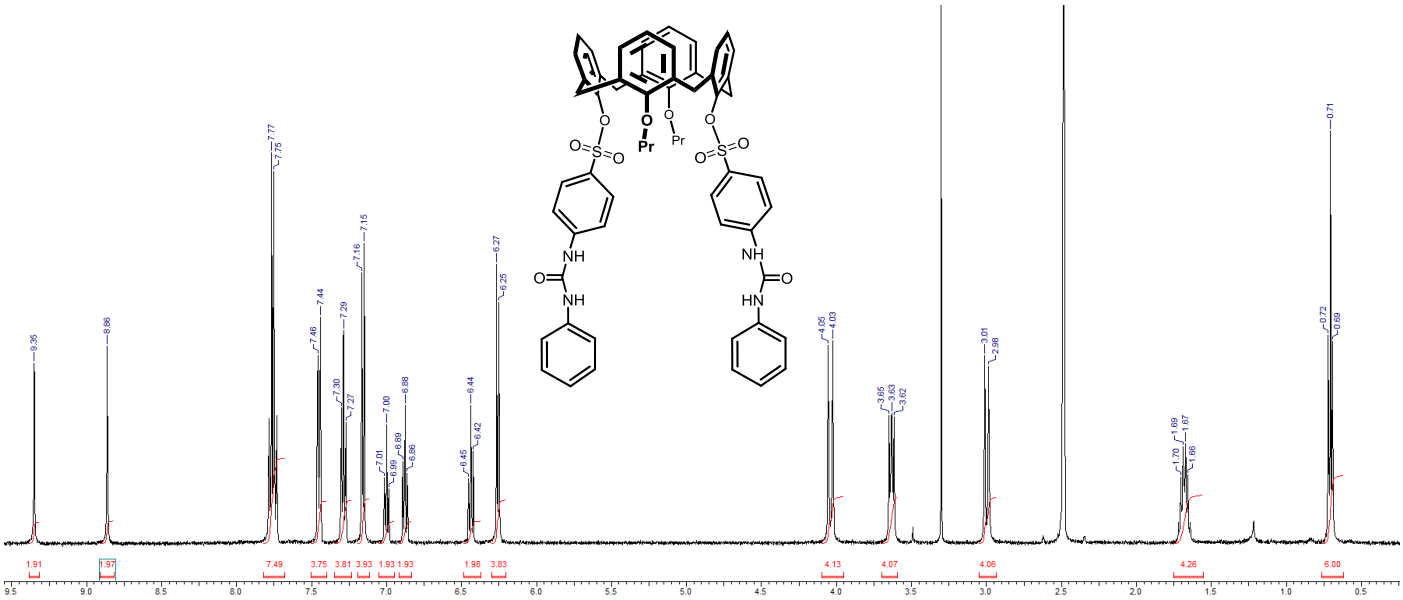


Figure 13: ^{1}H NMR spectrum of compound **4c** (300 MHz, DMSO-d_6, 298 K).

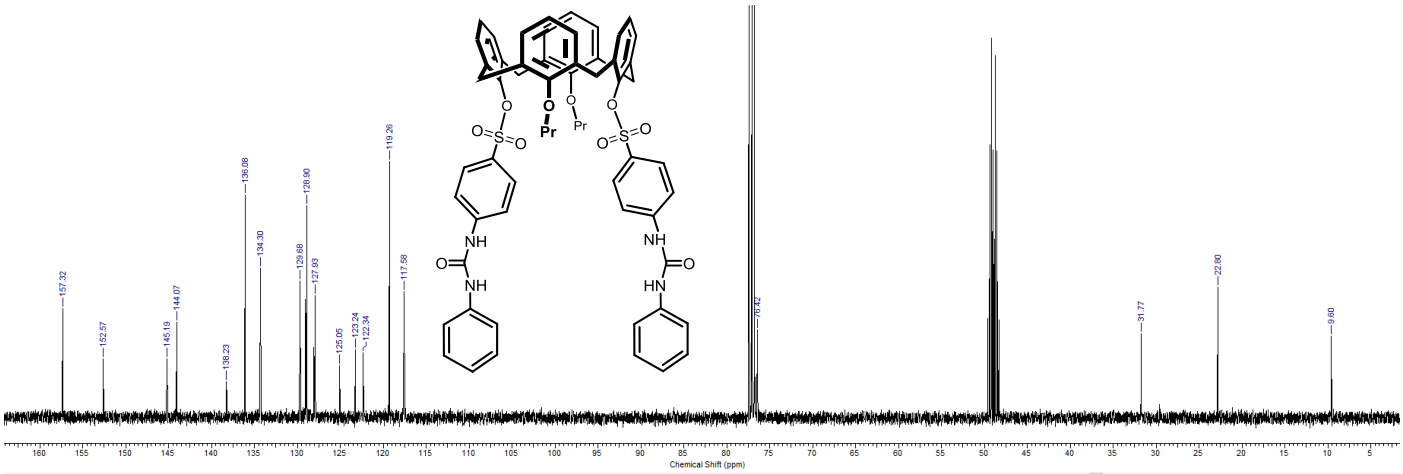


Figure 14: ^{13}C NMR spectrum of compound **4c** (100 MHz, $CDCl_3$/MeOD-d_4, 298 K).

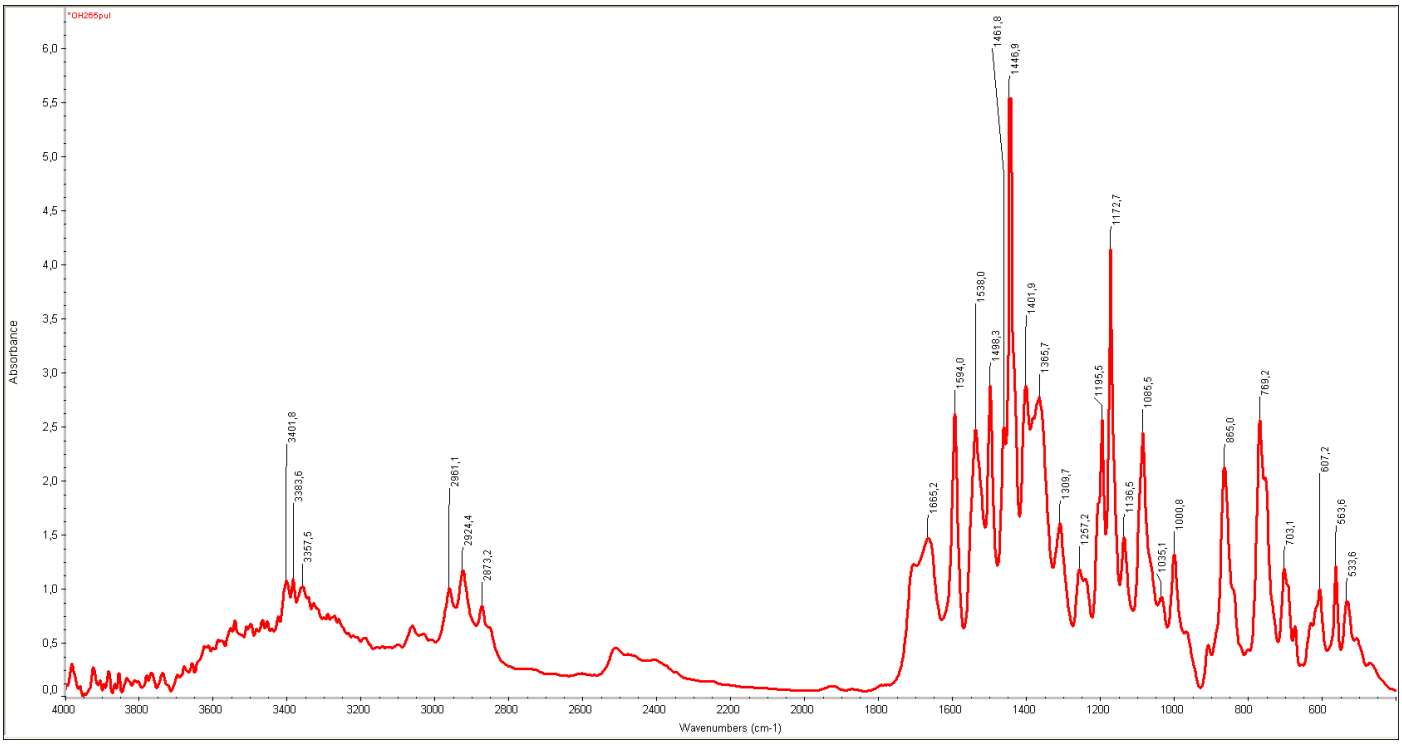


Figure 15: IR spectrum of compound **4c** (KBr).

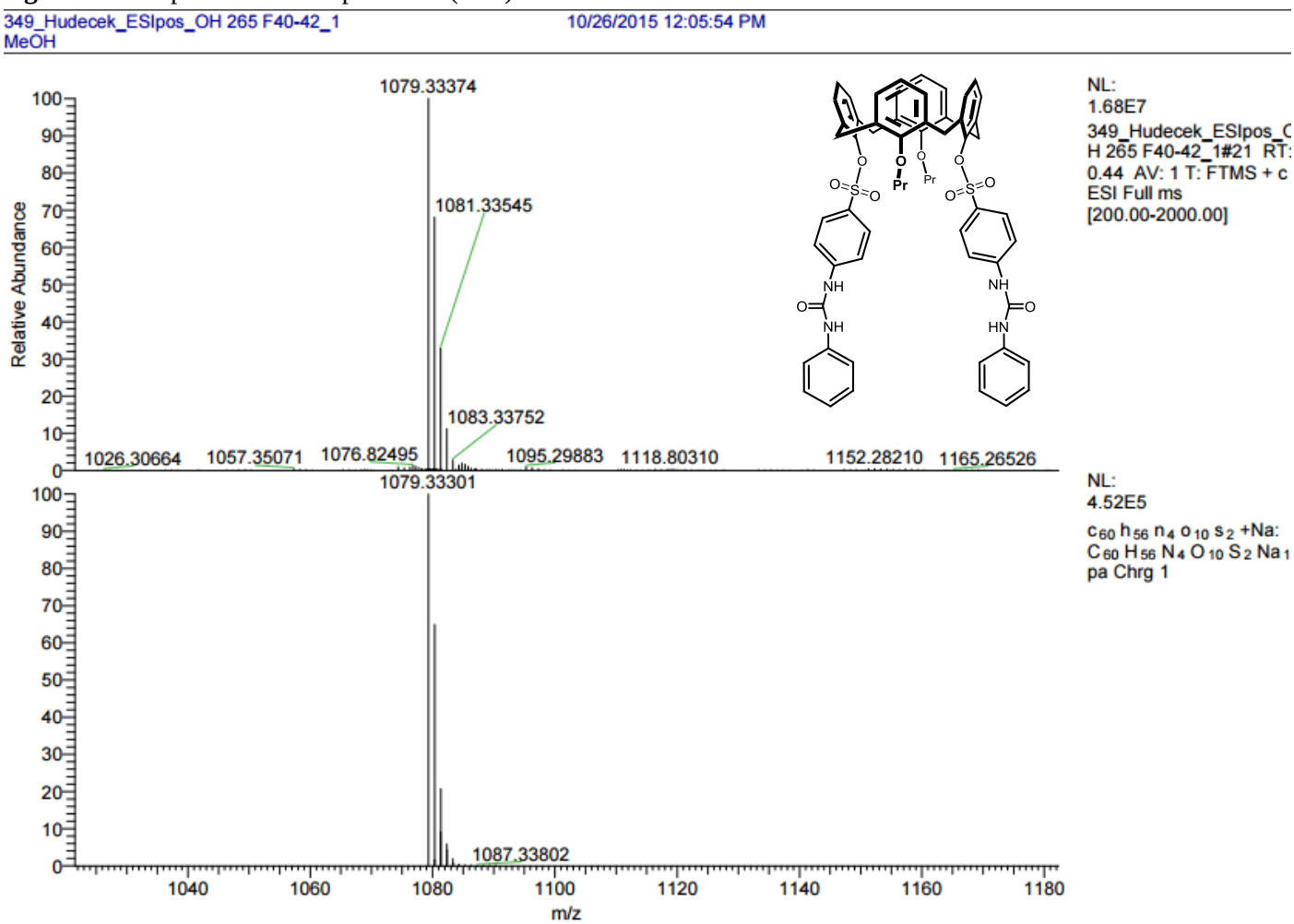


Figure 16: HRMS spectrum of compound **4c** (orbitrap).

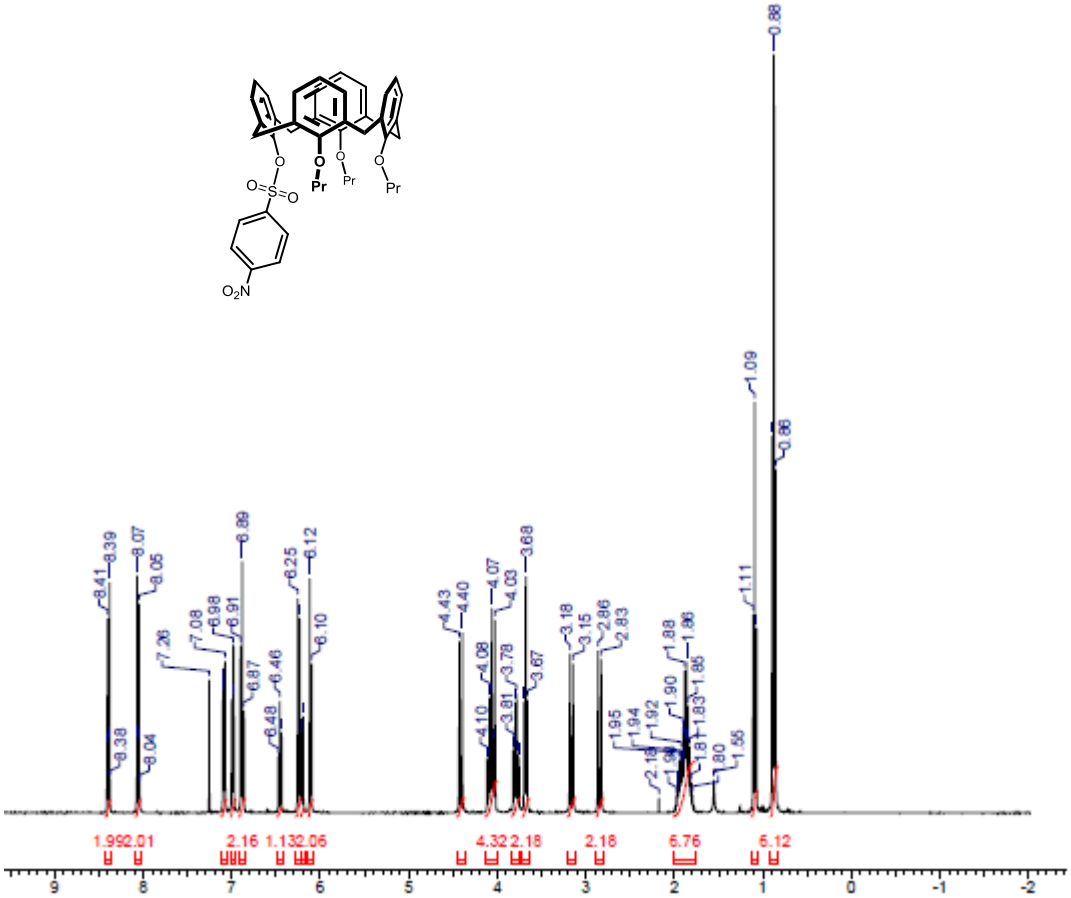


Figure 17: ^{1}H NMR spectrum of compound **6** (300 MHz, $CDCl_3$, 298 K).

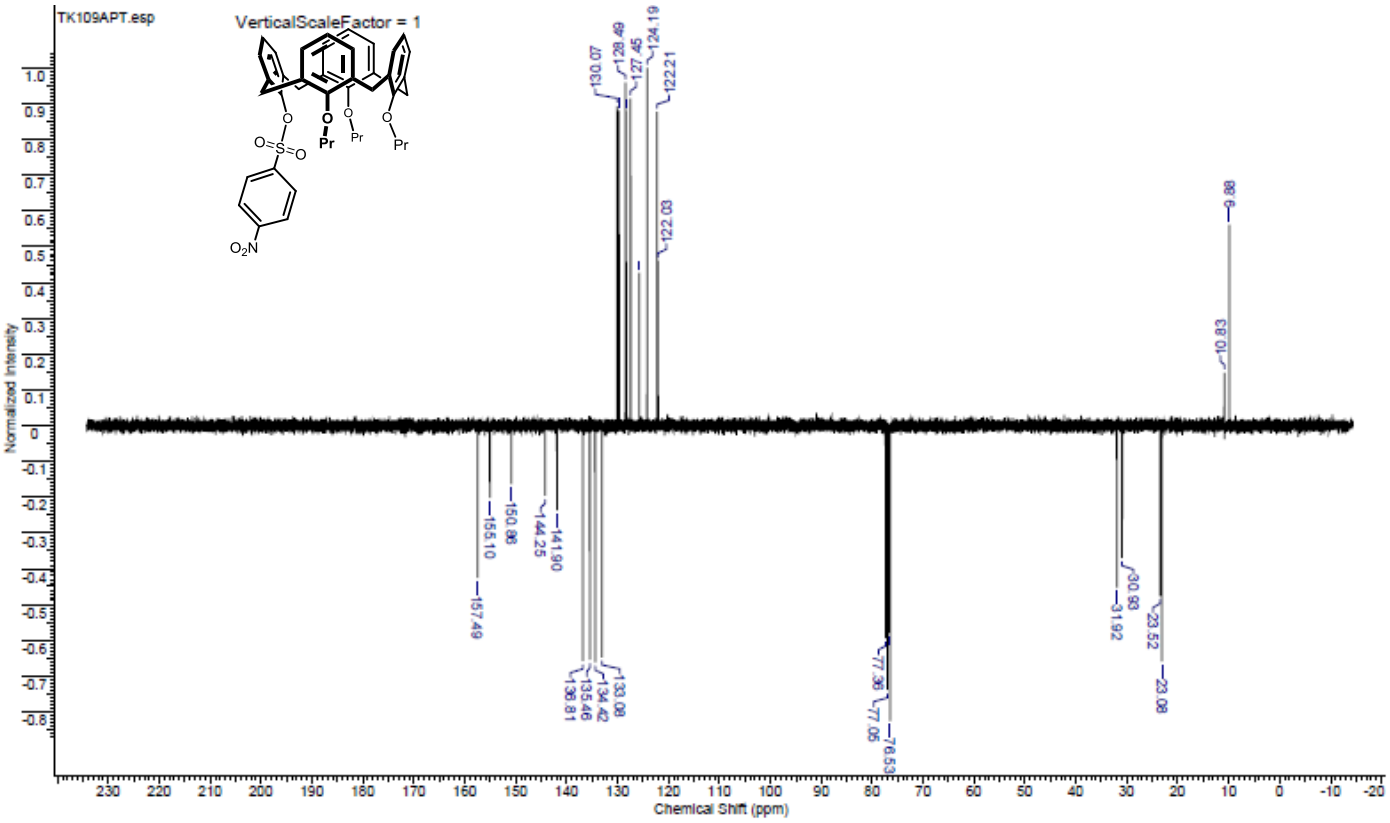


Figure 18: ^{13}C NMR (APT) spectrum of compound **6** (75 MHz, $CDCl_3$, 298 K).

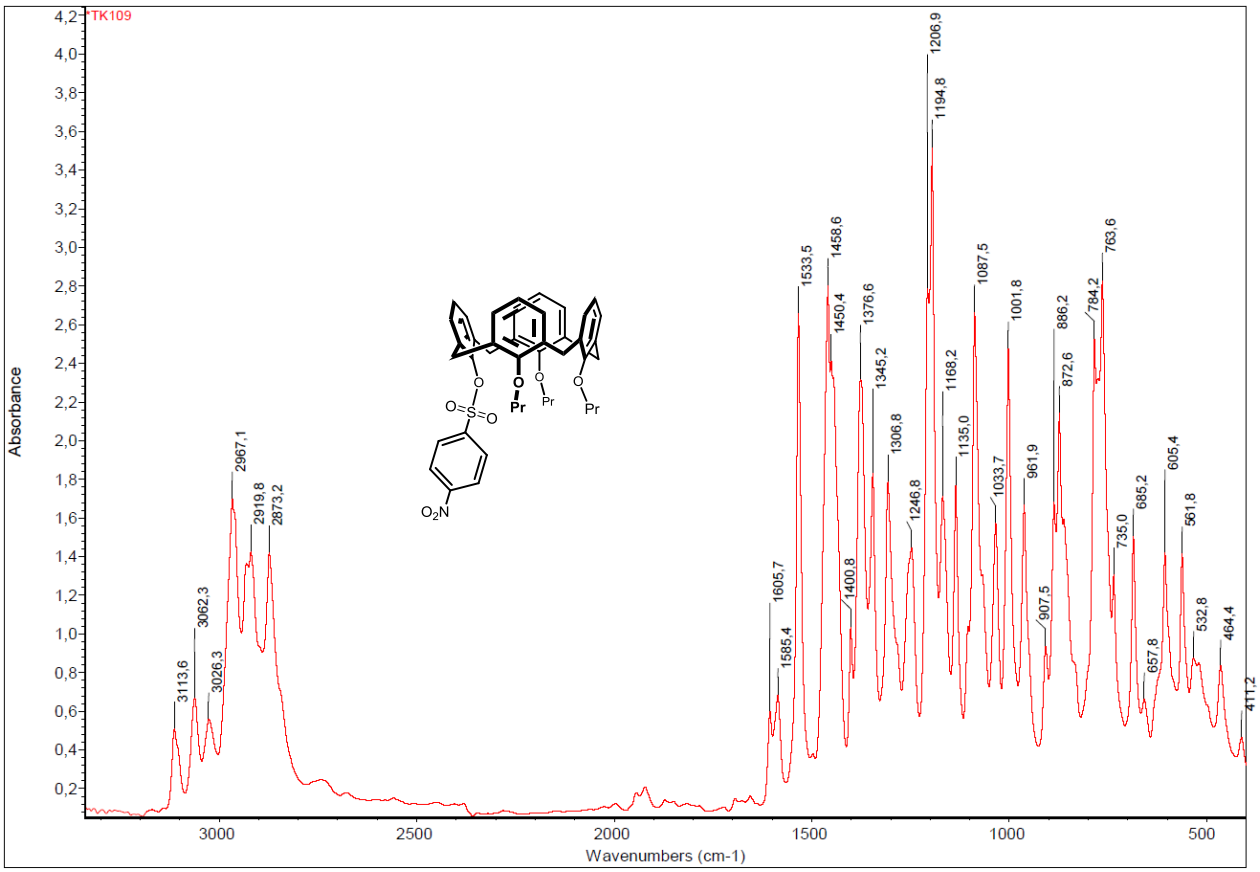


Figure 19: IR spectrum of compound **6** (KBr).

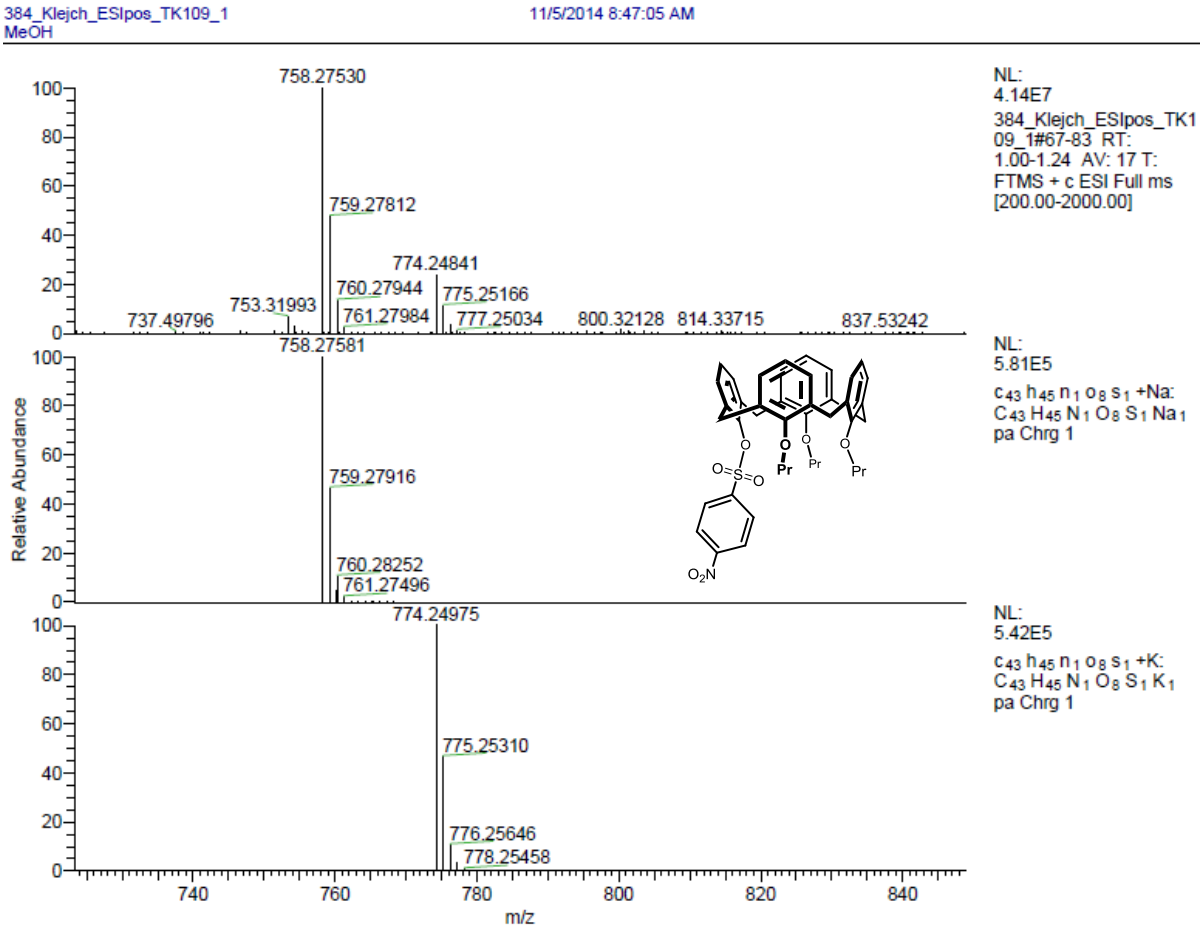


Figure 20: HRMS spectrum of compound **6** (orbitrap).

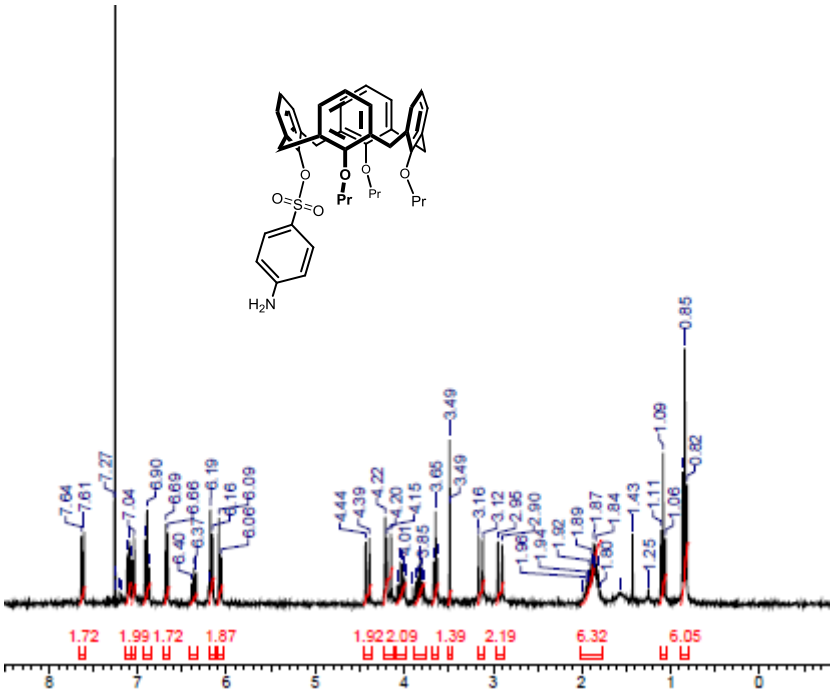


Figure 21: ^{1}H NMR spectrum of compound **7** (300 MHz, $CDCl_3$, 298 K).

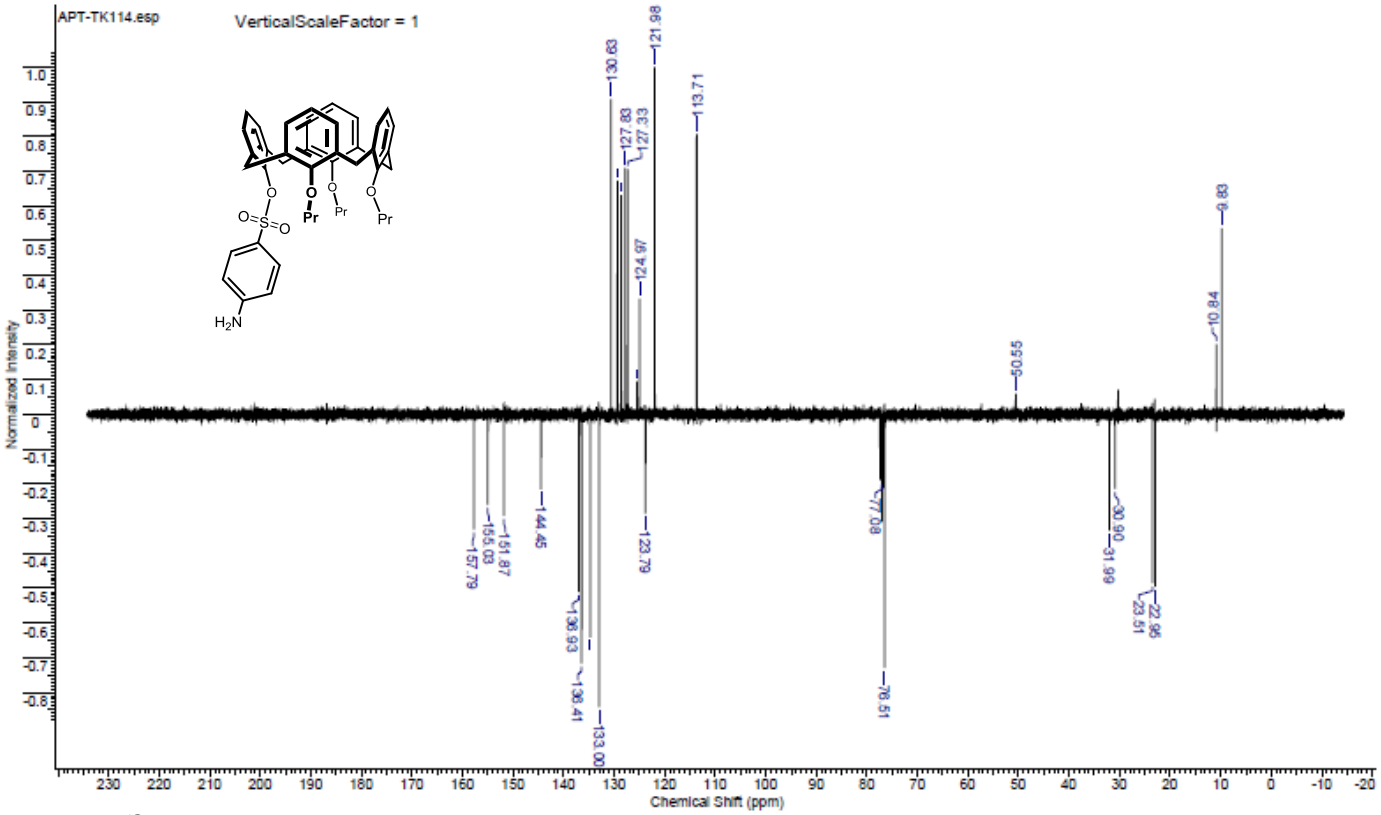


Figure 22: ^{13}C NMR (APT) spectrum of compound **7** (75 MHz, $CDCl_3$, 298 K).

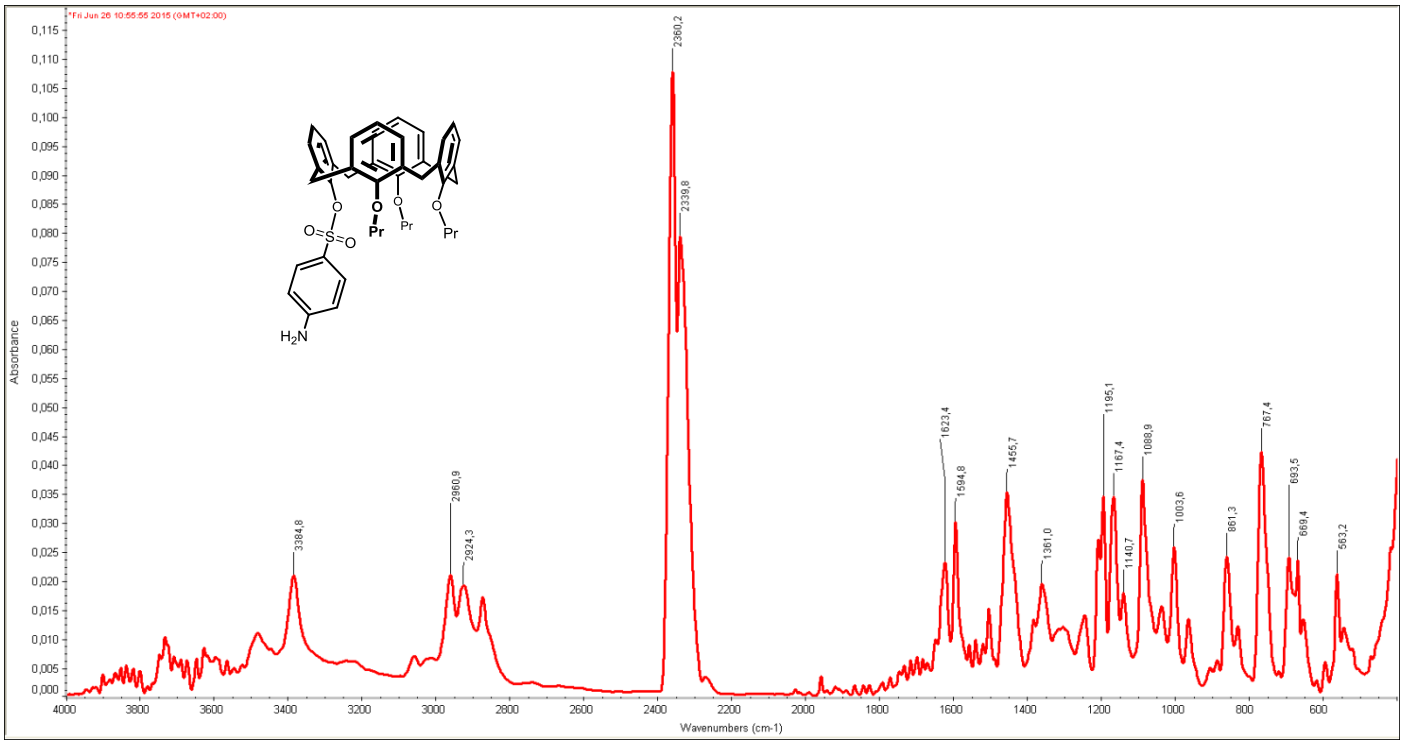


Figure 23: IR spectrum of compound **7** (KBr).

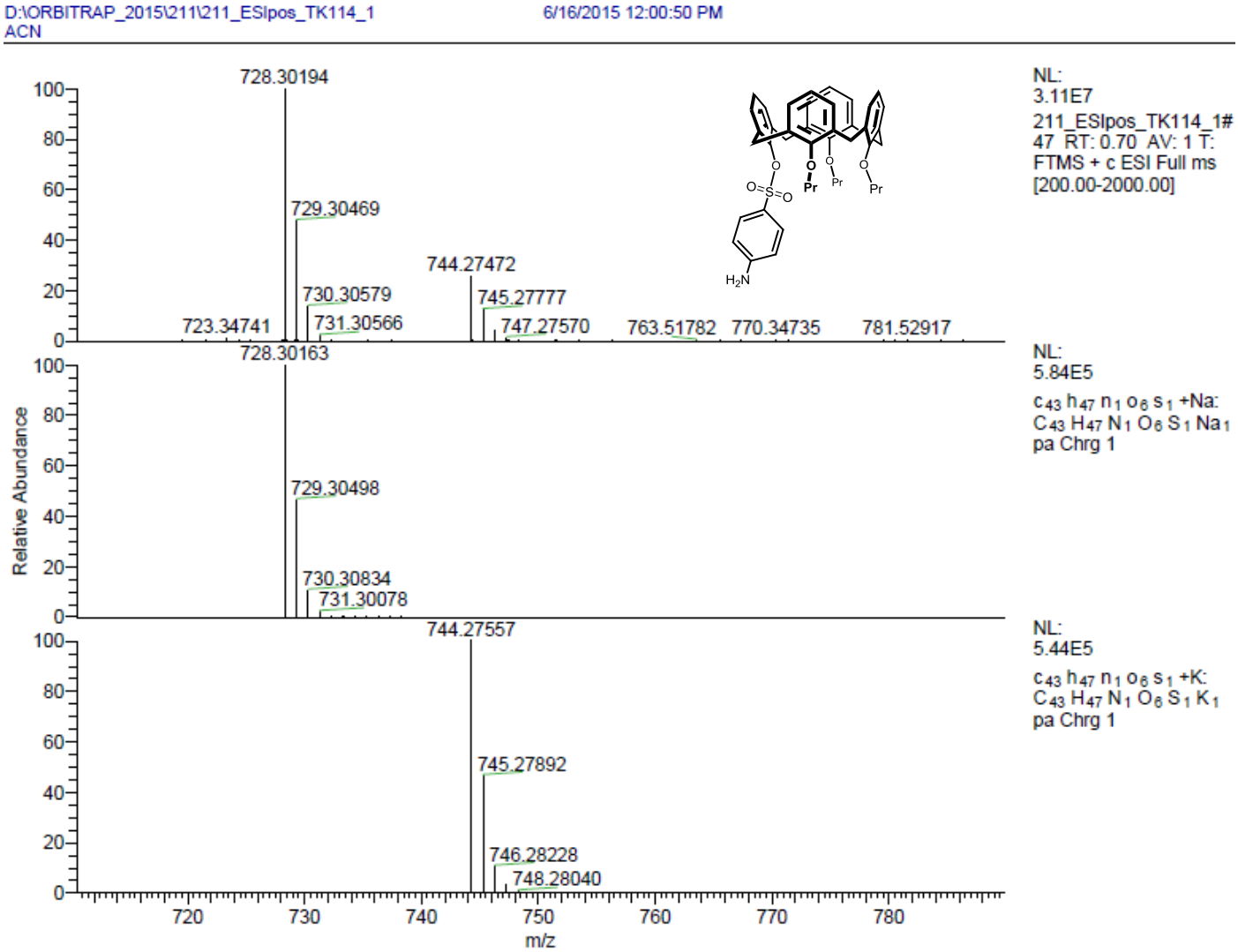


Figure 24: HRMS spectrum of compound **7** (orbitrap).

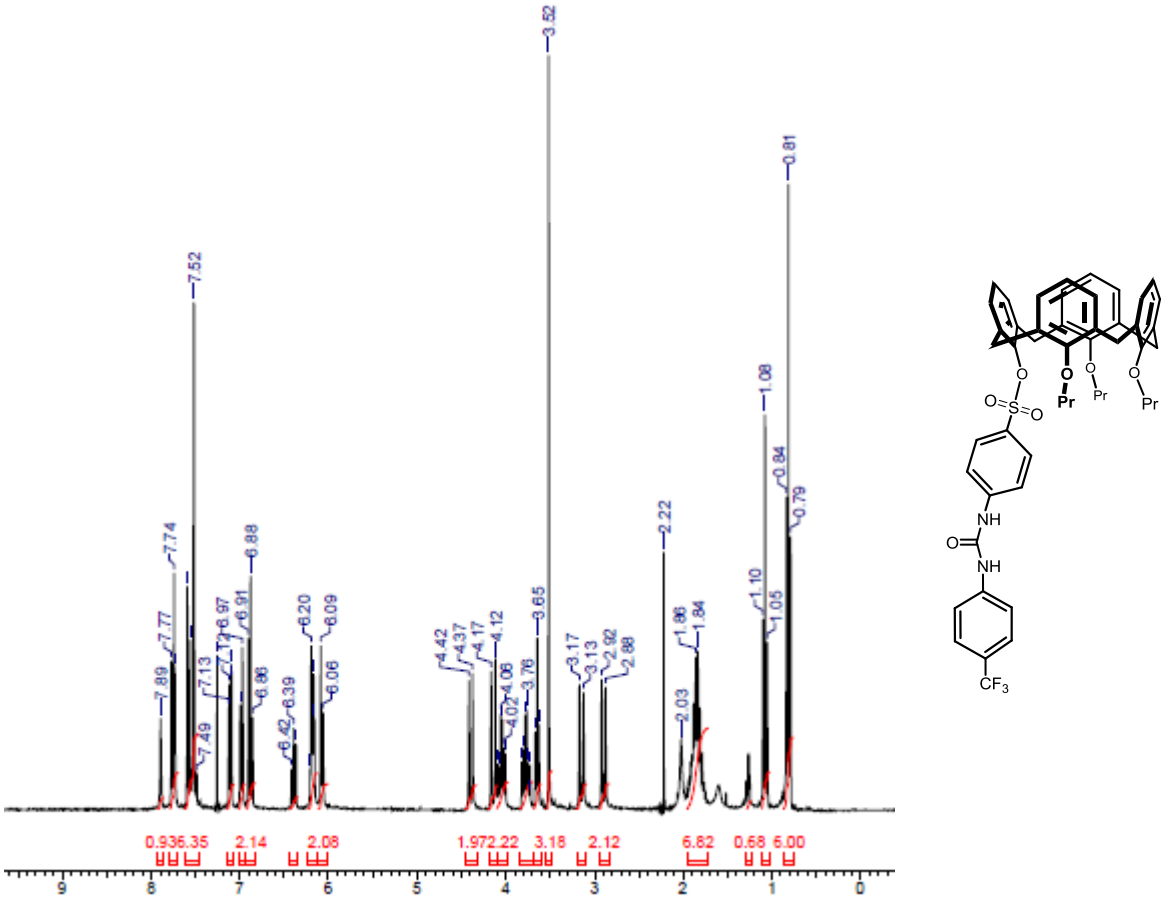


Figure 25: ^{1}H NMR spectrum of compound **8a** (300 MHz, DMSO-d$_6$, 298 K).

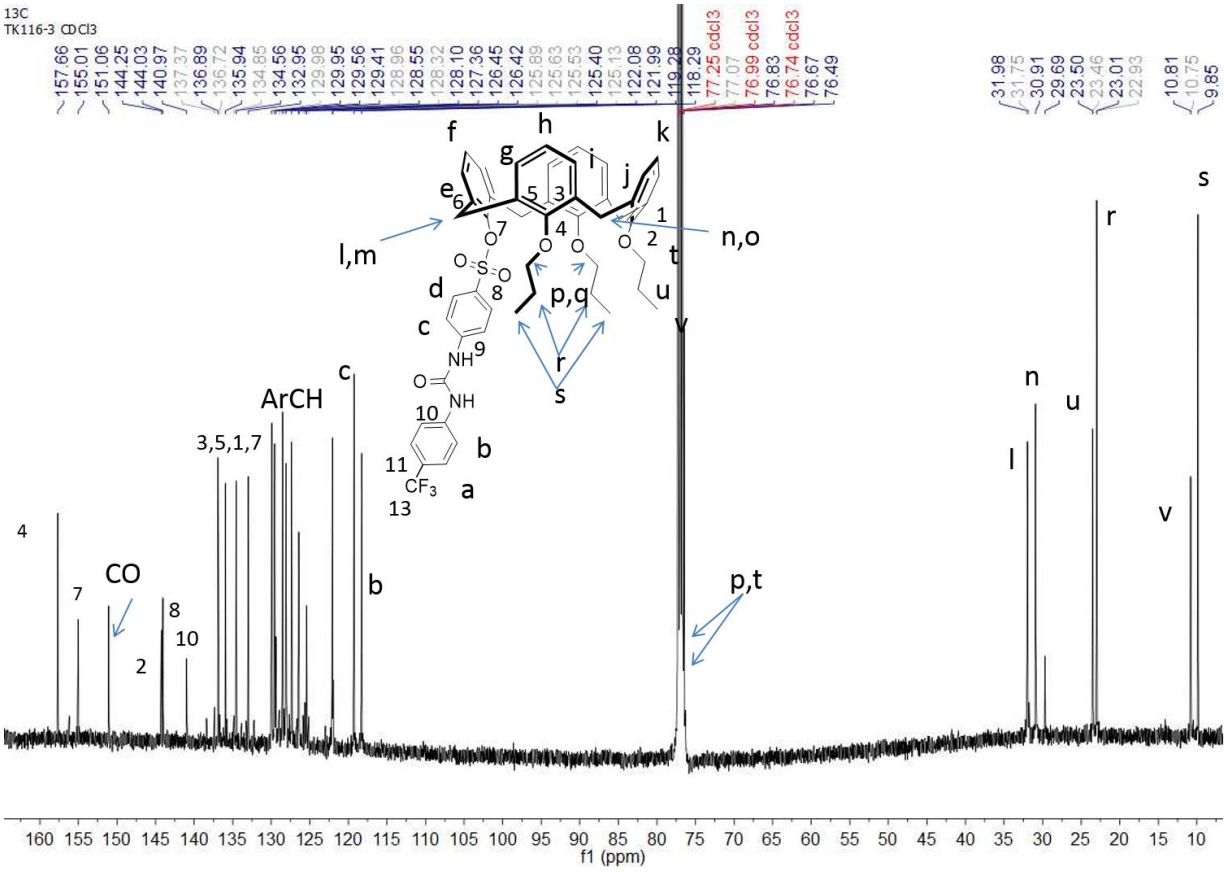


Figure 26: ^{13}C NMR (APT) spectrum of compound **8a** (75 MHz, CDCl$_3$, 298 K).

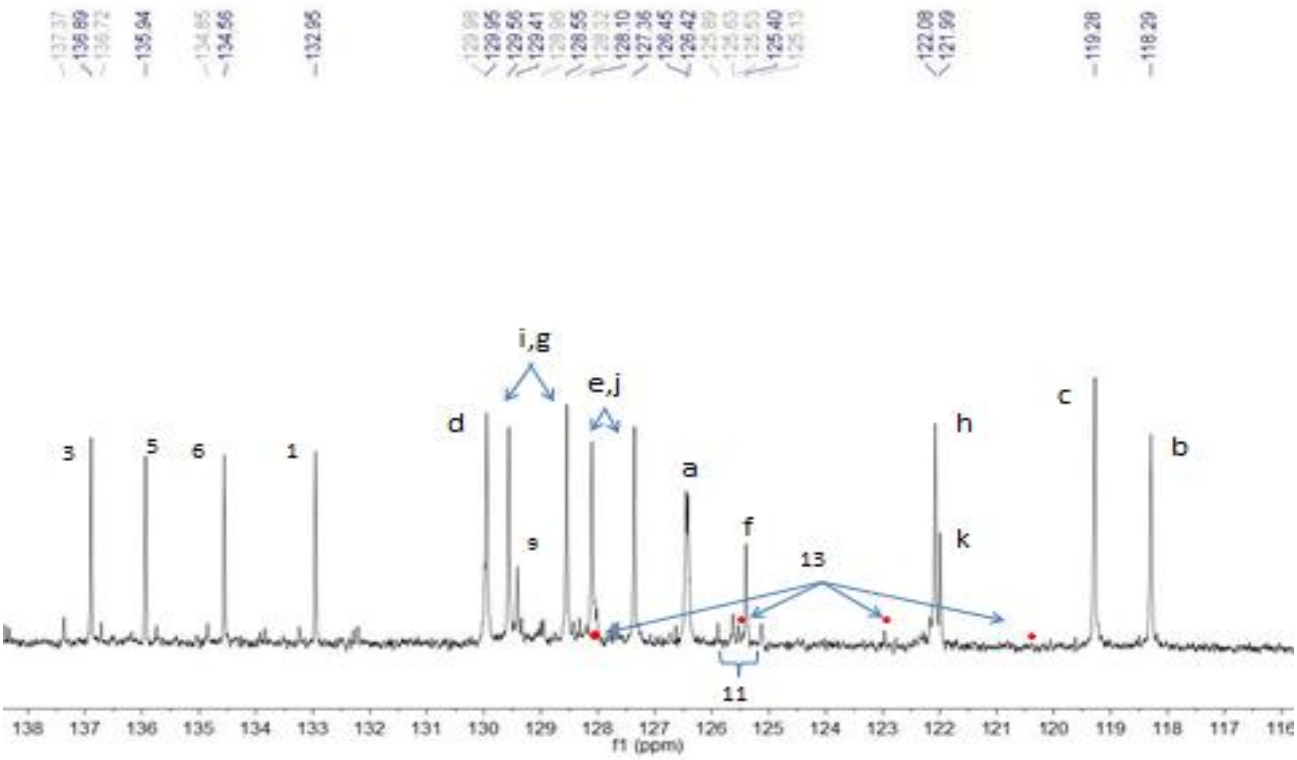


Figure 27: ^{13}C NMR (APT) spectrum of compound **8a** (75 MHz, $CDCl_3$, 298 K)- aromatic part of spectrum.

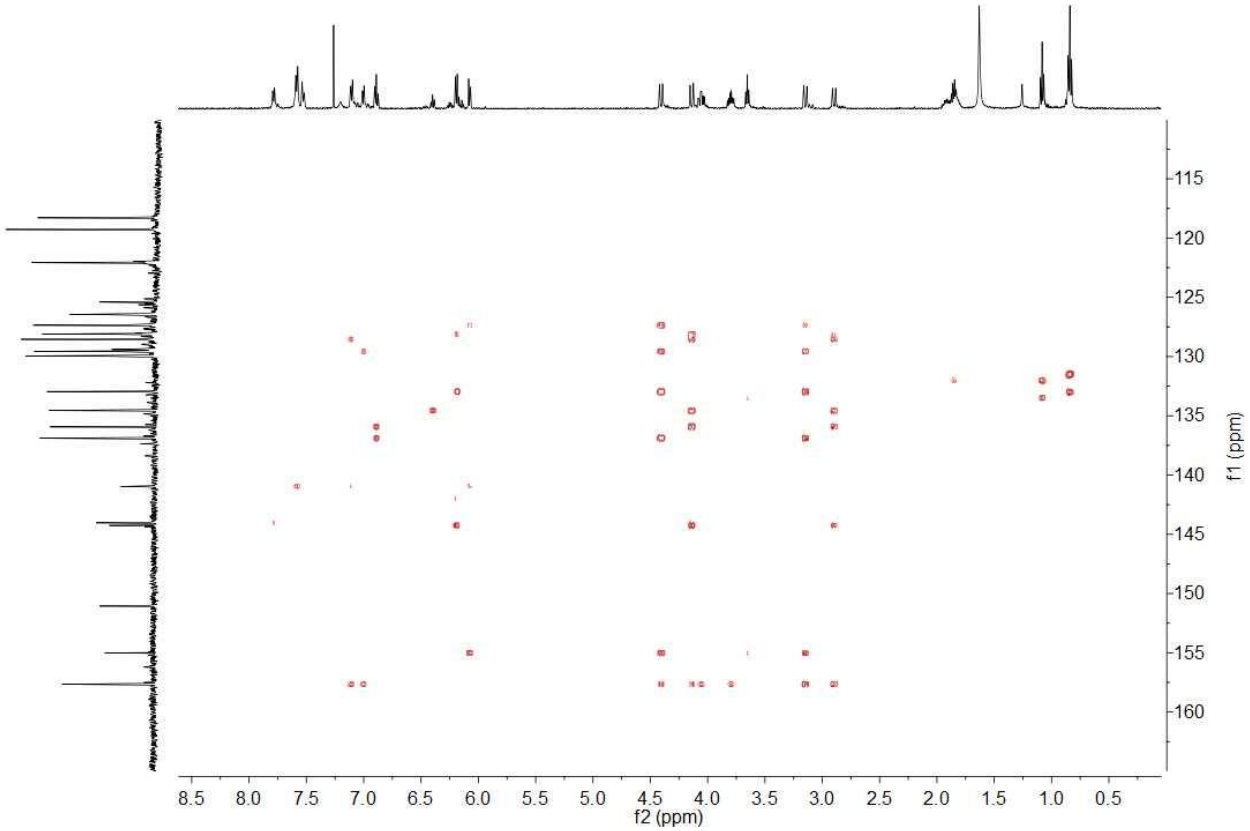


Figure 28: gHMBC spectrum of **8a** ($CDCl_3$, 298 K).

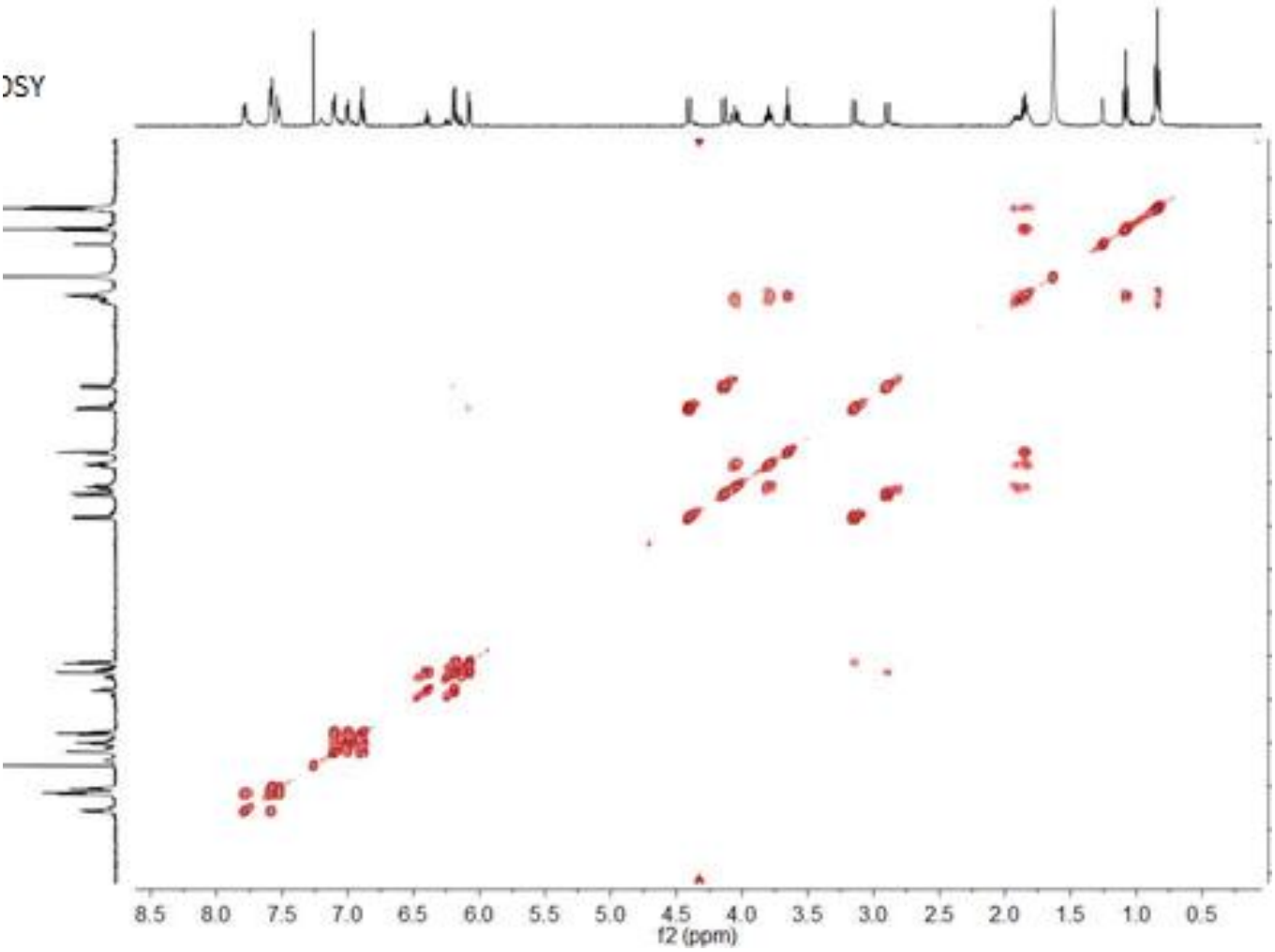


Figure 29: gCOSY spectrum of **8a** ($CDCl_3$, 298 K).

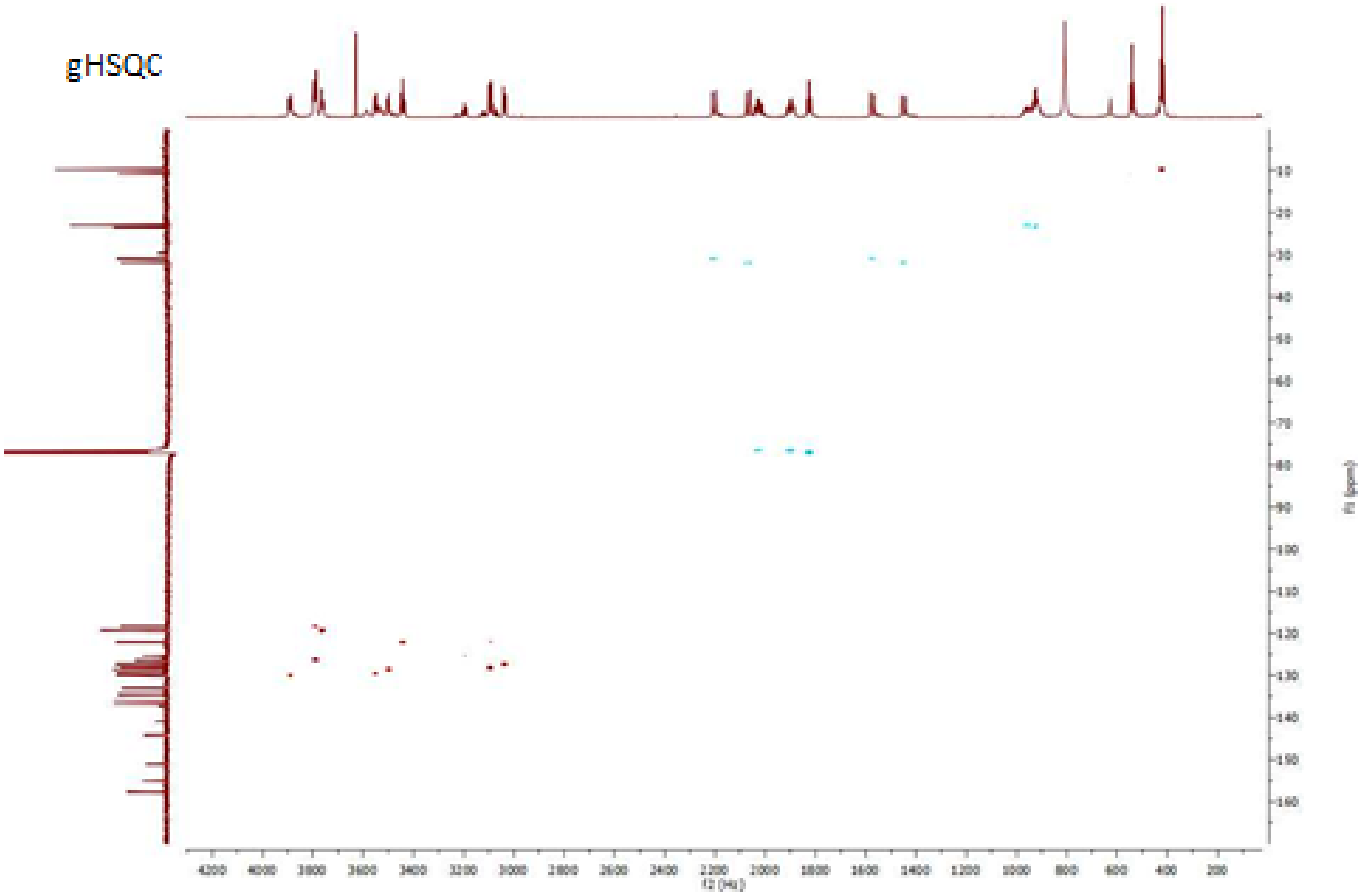


Figure 30: gHSQC spectrum of **8a** ($CDCl_3$, 298 K).

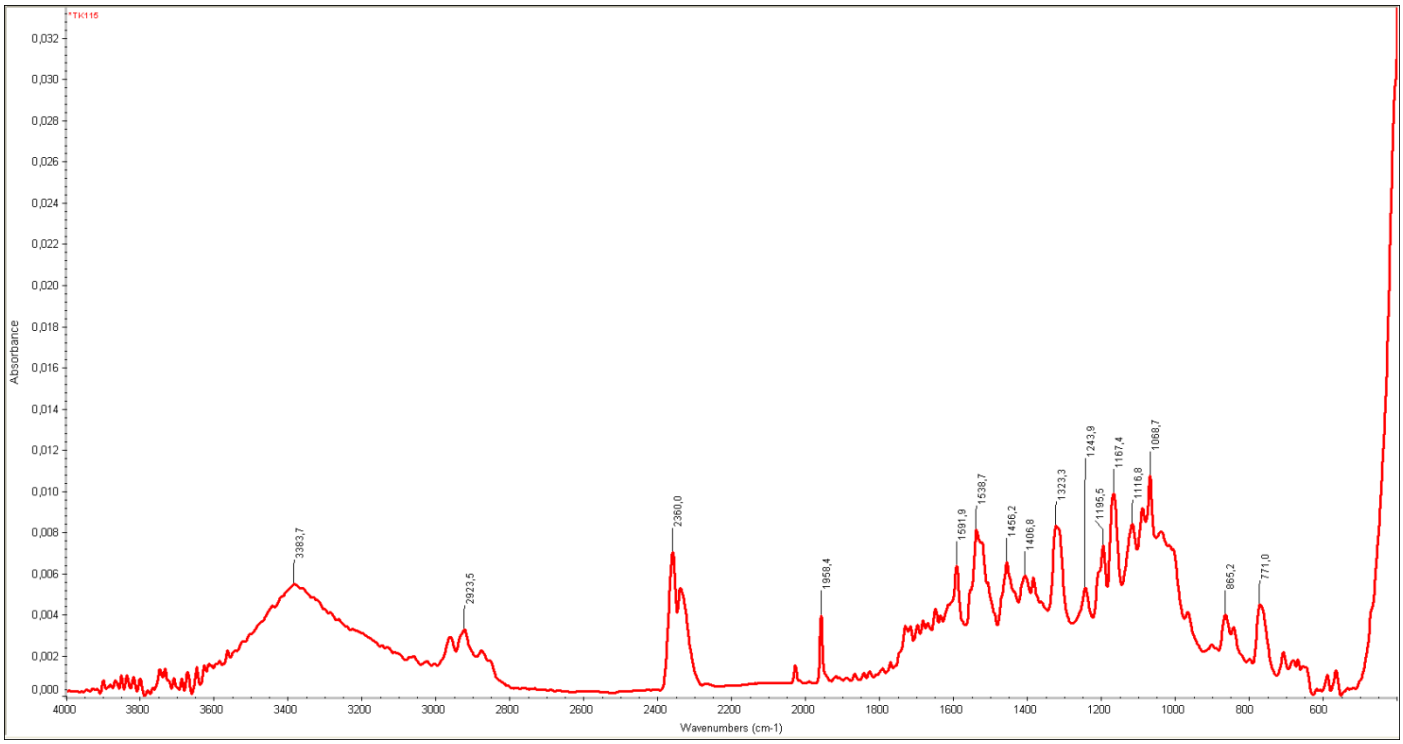


Figure 31: IR spectrum of compound **8a** (KBr).

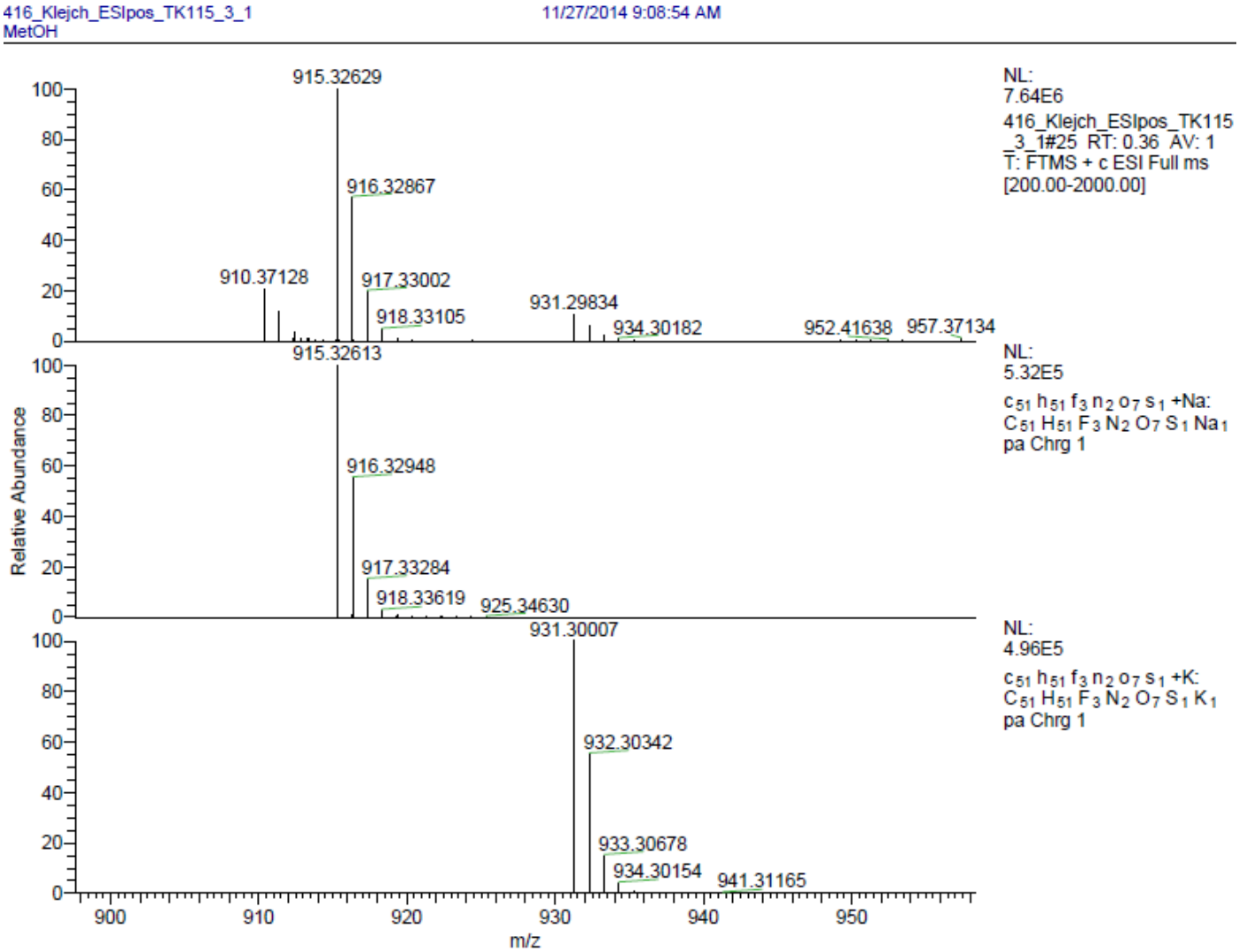


Figure 32: HRMS spectrum of compound **8a** (orbitrap).

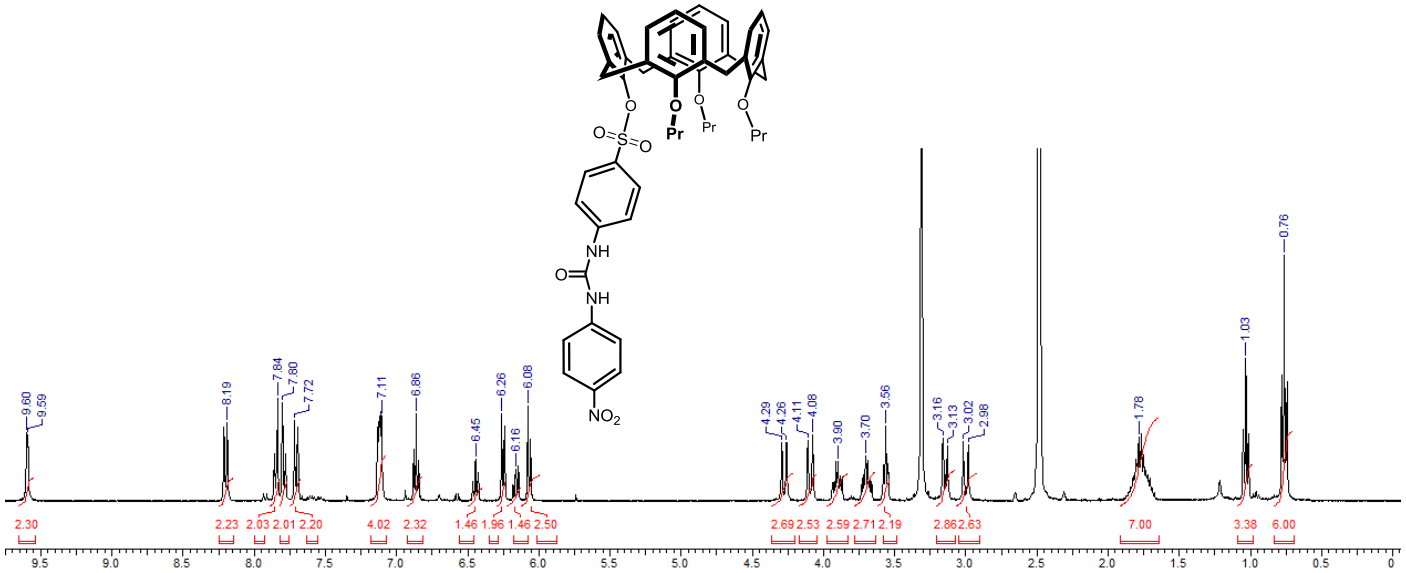


Figure 33: ^{1}H NMR spectrum of compound **8b** (300 MHz, DMSO-d_6, 298 K).

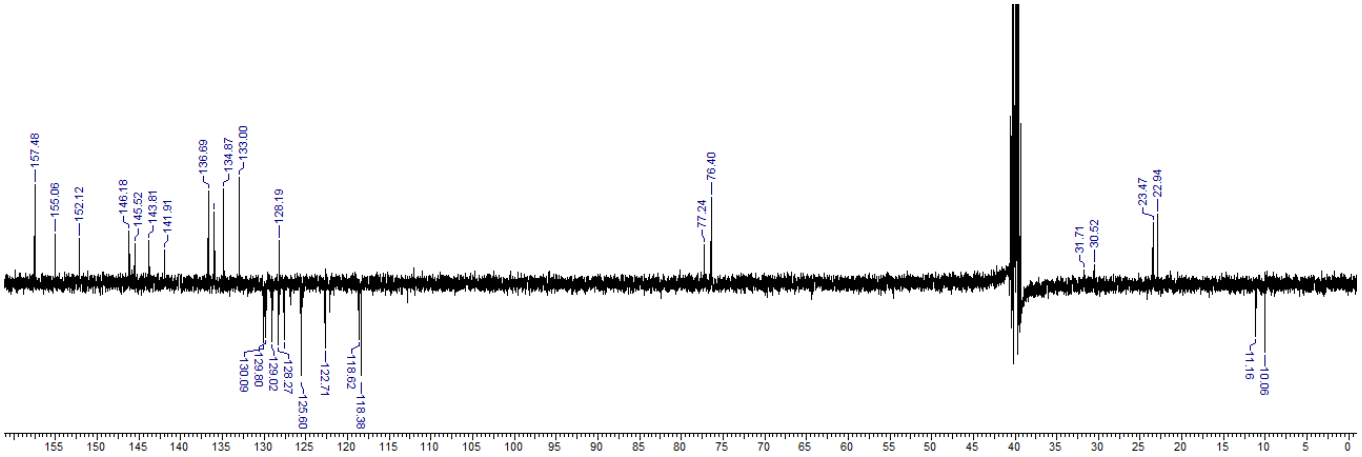


Figure 34: ^{13}C NMR (APT) spectrum of compound **8b** (75 MHz, DMSO-d_6, 298 K).

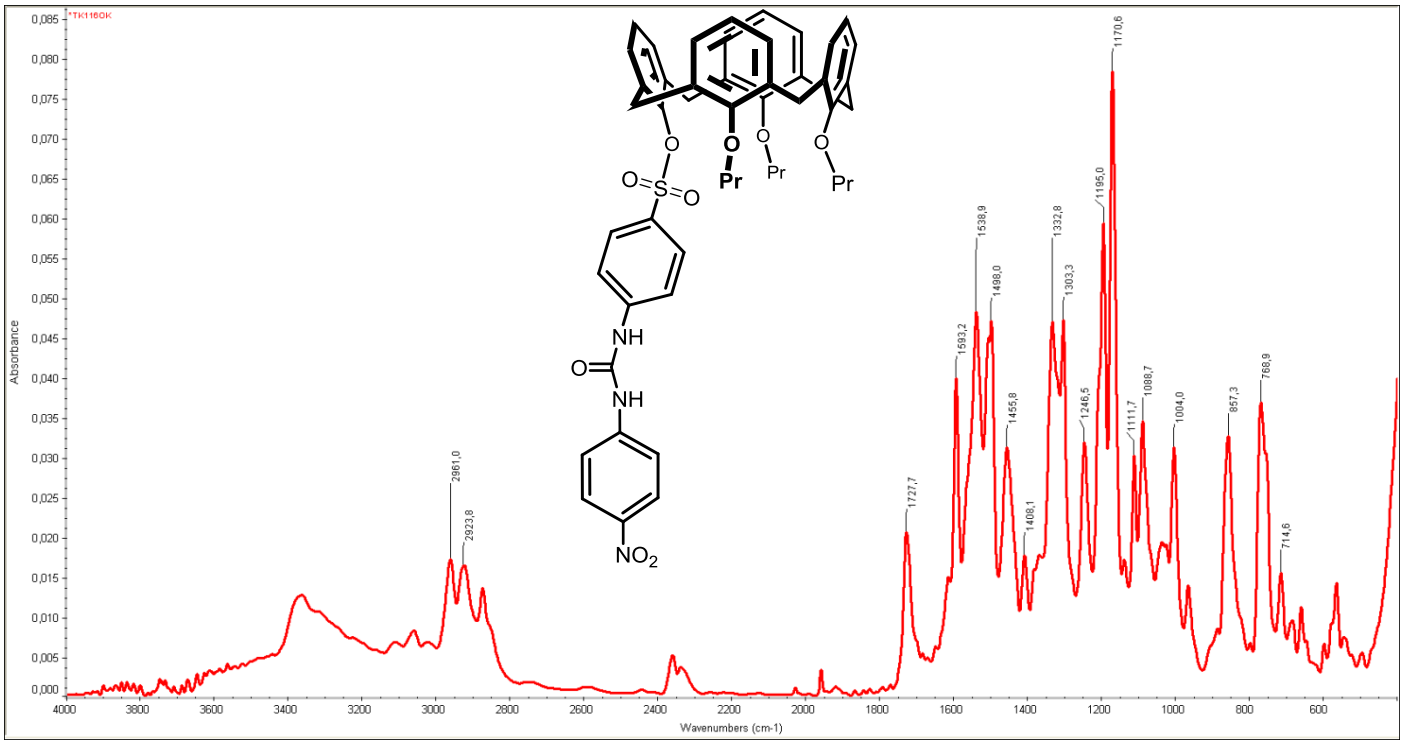


Figure 35: IR spectrum of compound **8b** (KBr).

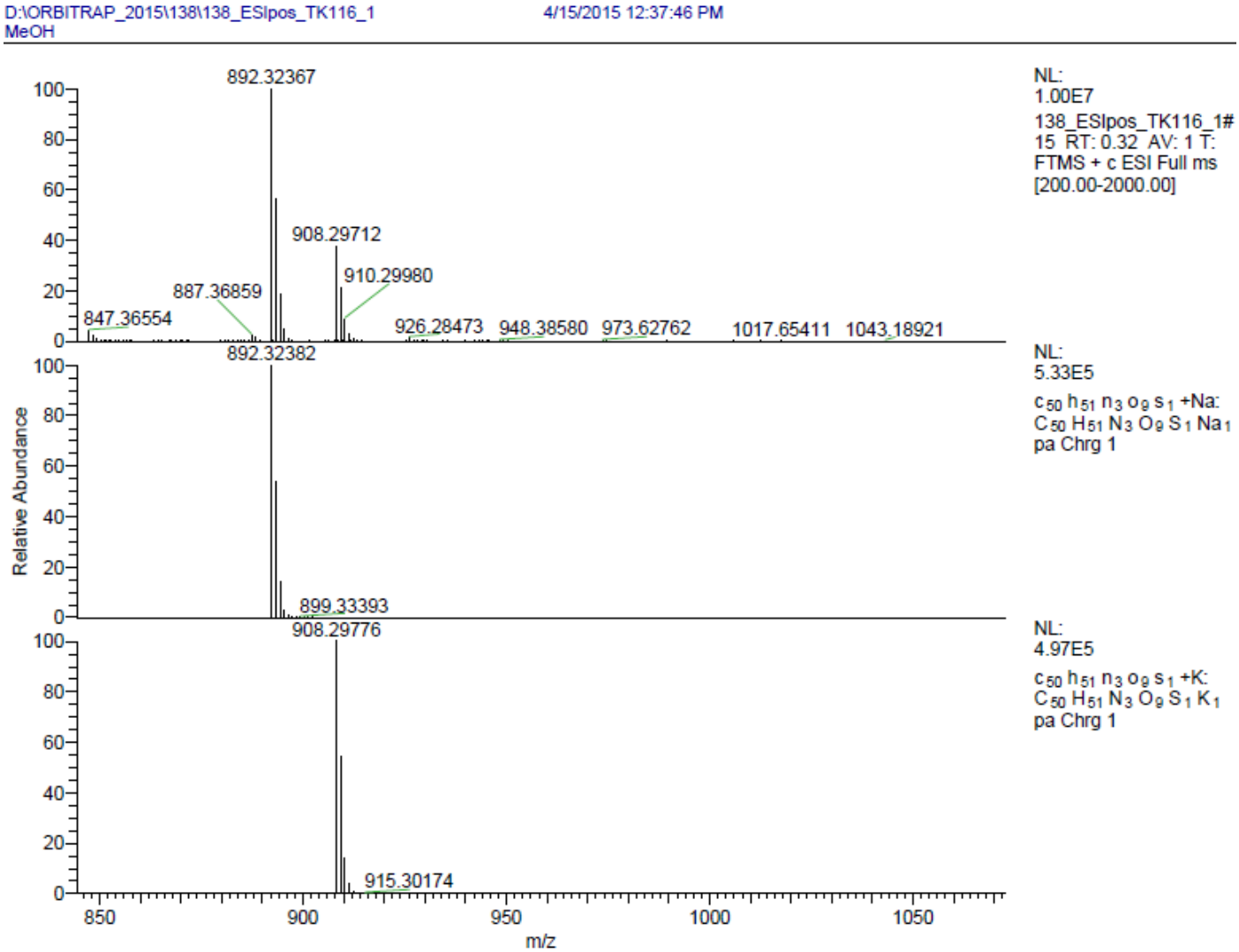
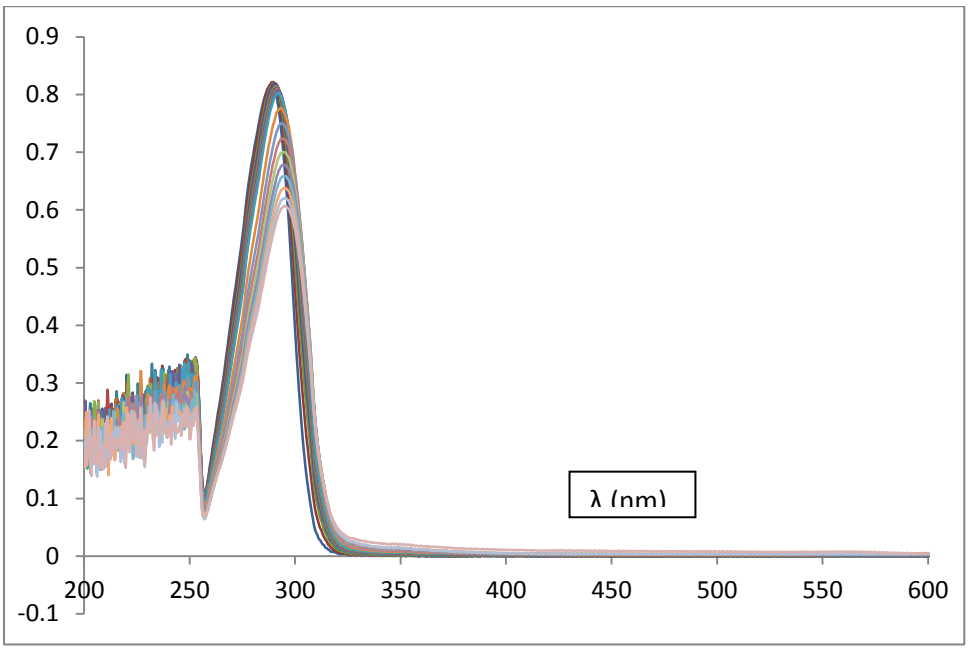


Figure 36: HRMS spectrum of compound **8b** (orbitrap).

Examples of UV-vis titrations

Figure 37: UV-vis titration of compound **4a** by $TBA^{+}H_2PO_4^{-}$, DMSO, 298 K,

Cintra 20 spectrometer (GBC Scientific Equipment Ltd.), λ 200-600nm, step: 0.48

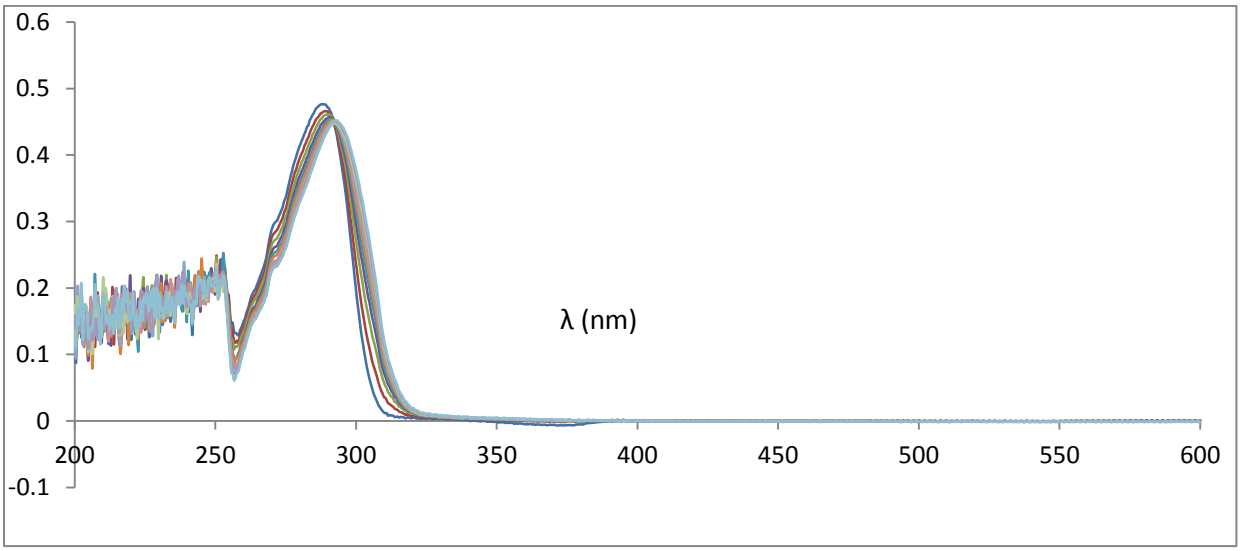


Starting concentration of calixarene 4a : 1.01E-05

Concentration ratio in each point of the titration:

$C_{H2PO4}/C_{calixarene}$
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
19.9
29.9
39.9
49.9
59.8
69.8
79.8
89.8
99.7

Figure 38: UV-vis titration of compound **8a** by $TBA^{+}H_2PO_4^{-}$.
DMSO, Cintra 20 spectrometer (GBC Scientific Equipment Ltd.), λ 200-600nm, step: 0.48



Starting concentration of calixarene 8a : 9.97E-06

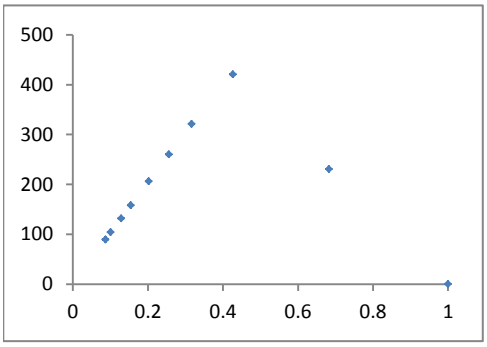
Concentration ratio in each point of the titration:

$C_{H2PO4}/C_{calixarene}$
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.1
9.1
10.1

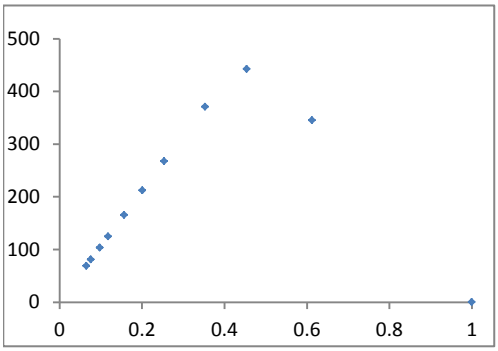
Examples of Job plot experiments:

Fig. 39
The JOB plots experiments for 8b with the corresponding anions: (^{1}H NMR, 500 MHz, 298 K, DMSO-d_6)

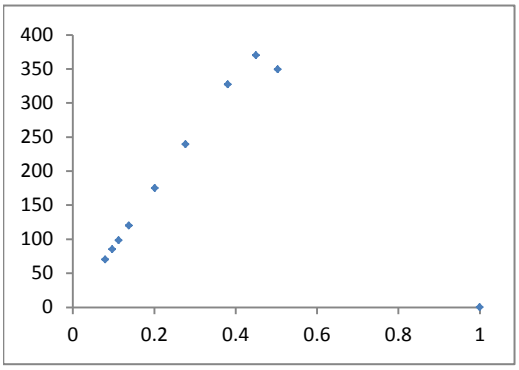
Benzoate



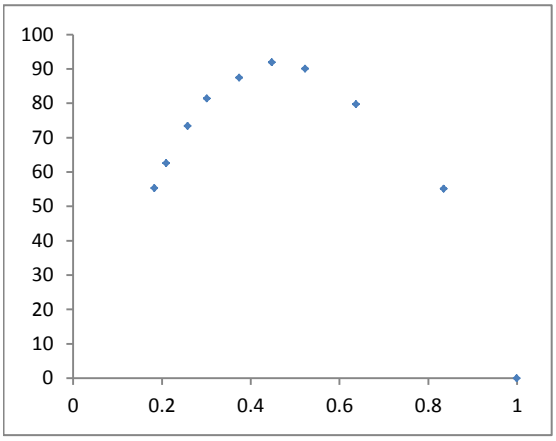
Acetate



Dihydrogenphosphate



Chloride



Hydrogensulfate

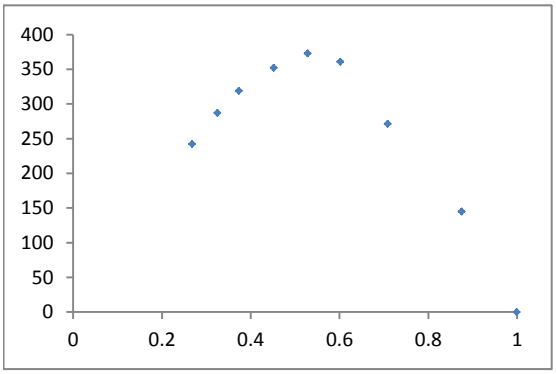
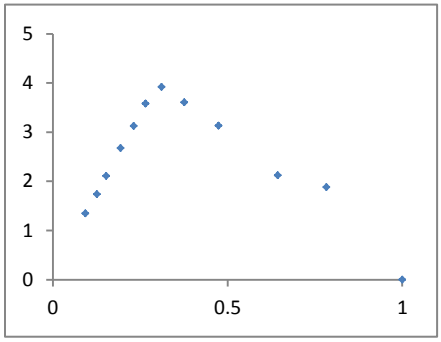


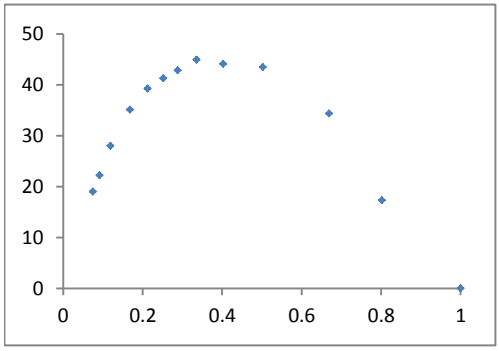
Fig. 40

The JOB plots experiments for 4b with the corresponding anions: (^{1}H NMR, 500 MHz, 298 K, DMSO-d_6)

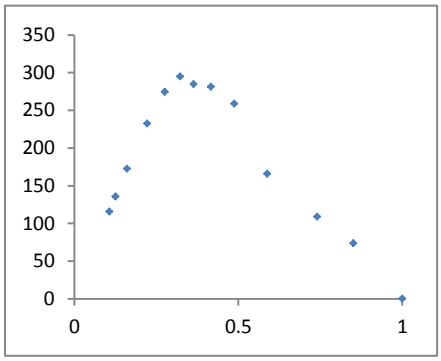
Acetate



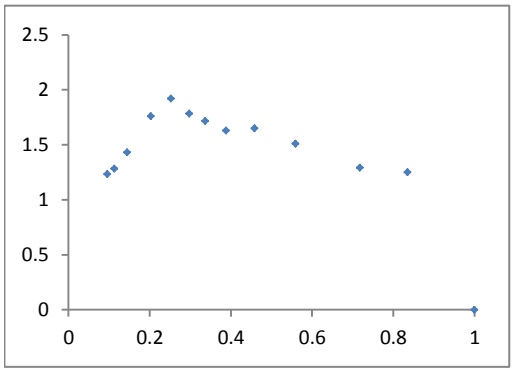
Chloride



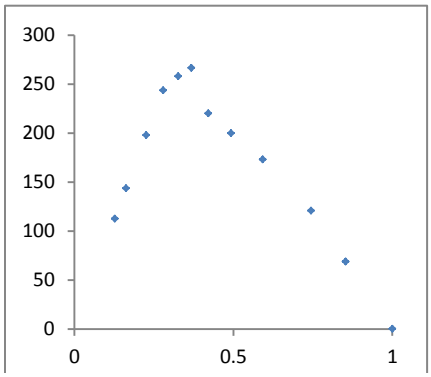
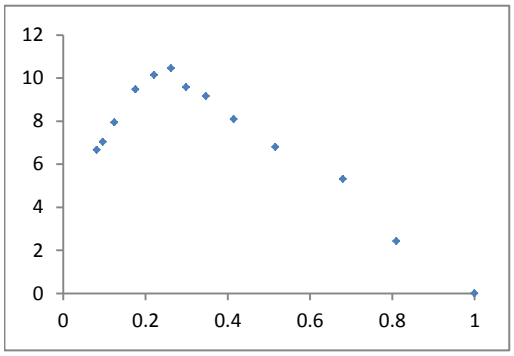
Benzoate



Nitrate



Phosphate



Sulfate