Supporting Information

Upper Rim-Bridged Calix[4]arenes via Cyclization of *meta*- Alkynyl Intermediates with Diphenyl Diselenide

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1. Spectral characterization of compounds

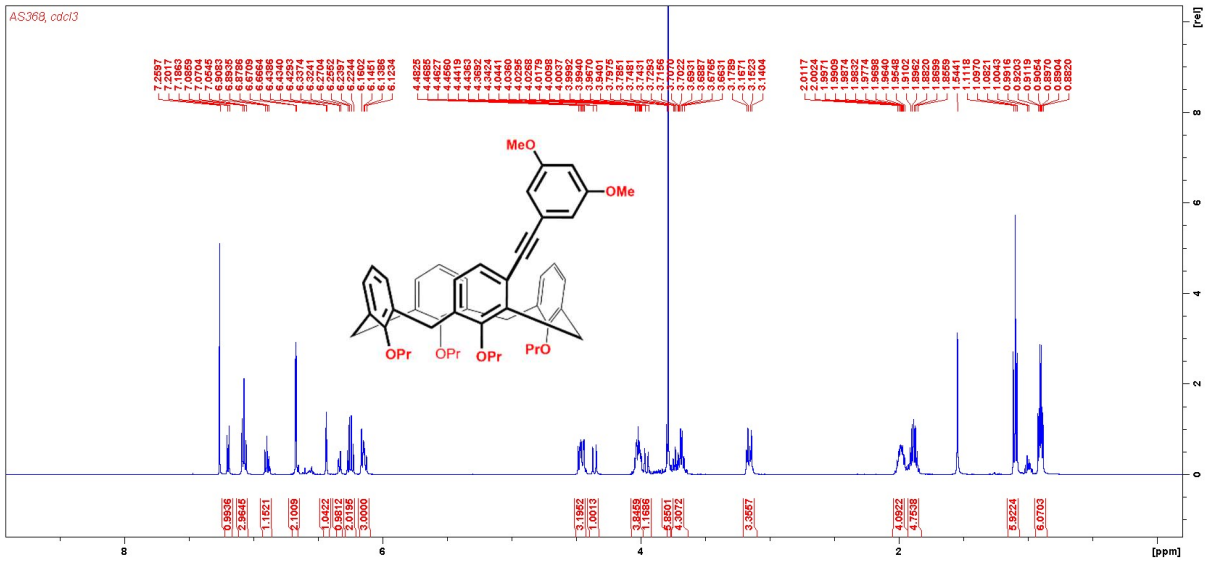


Figure S1: ^{1}H NMR of compound **5a** ($CDCl_3$, 500 MHz, 298 K)

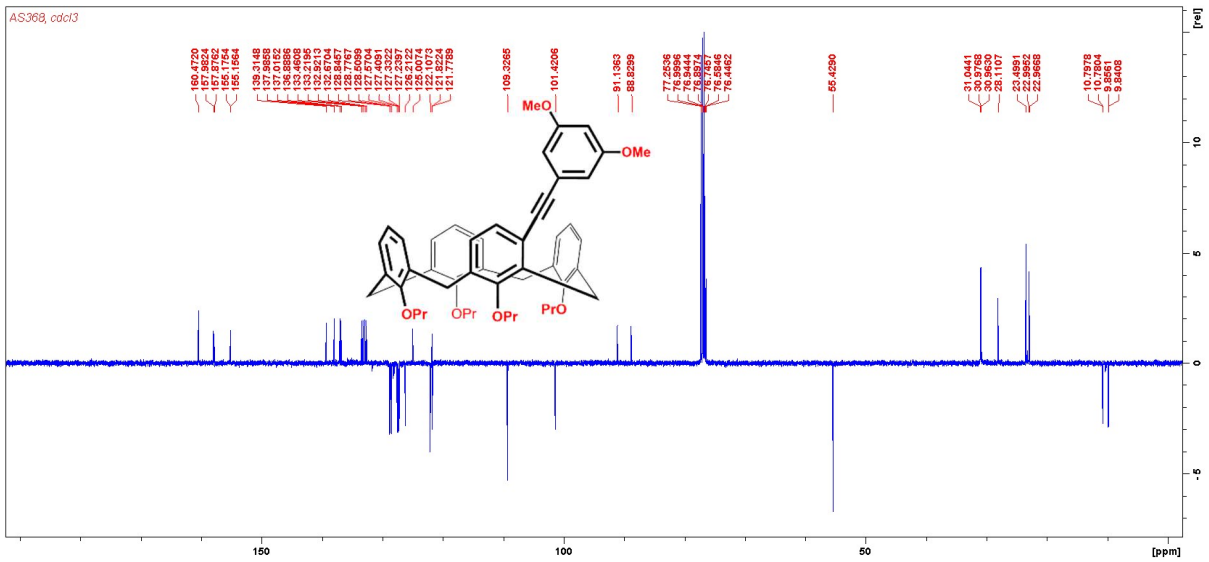


Figure S2: ^{13}C(APT) NMR of compound **5a** ($CDCl_3$, 125 MHz, 298 K)

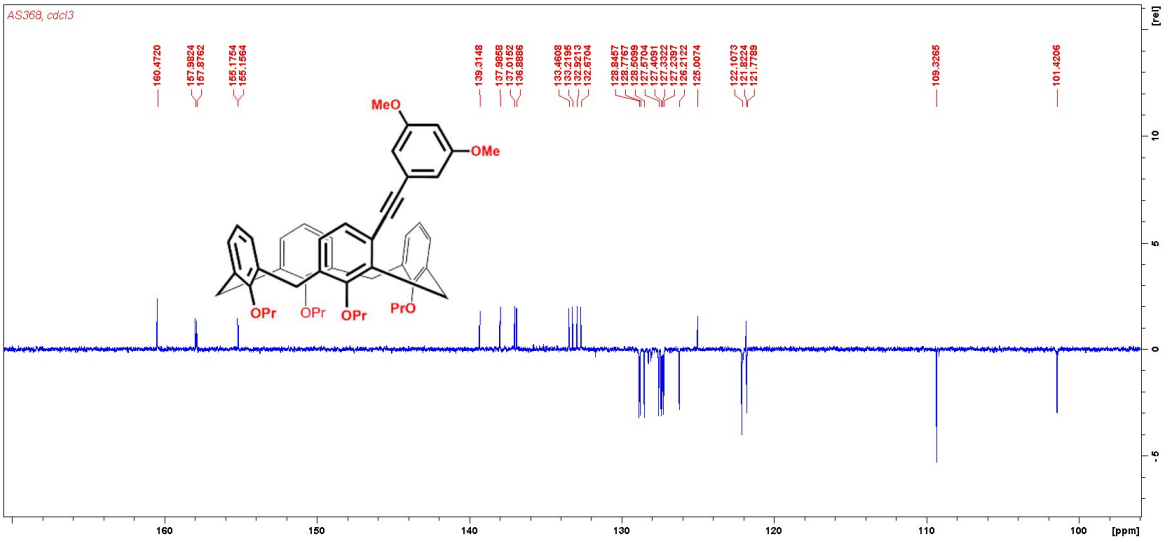


Figure S3: ^{13}C(APT) NMR of compound **5a,** aromatic region ($CDCl_3$, 125 MHz, 298 K)

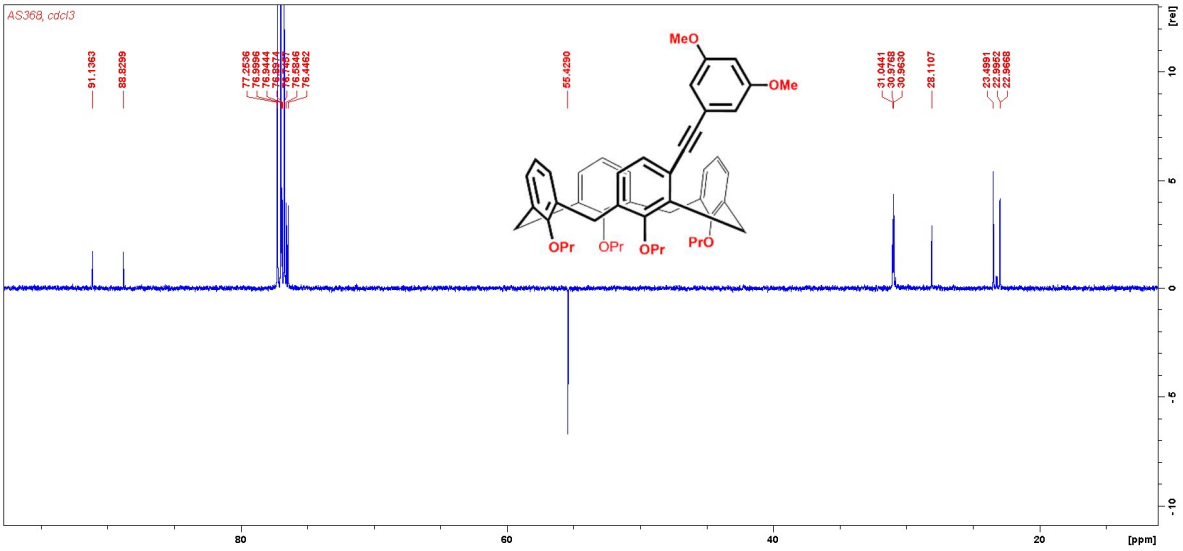


Figure S4: ^{13}C(APT) NMR of compound **5a,** aliphatic region ($CDCl_3$, 125 MHz, 298 K)

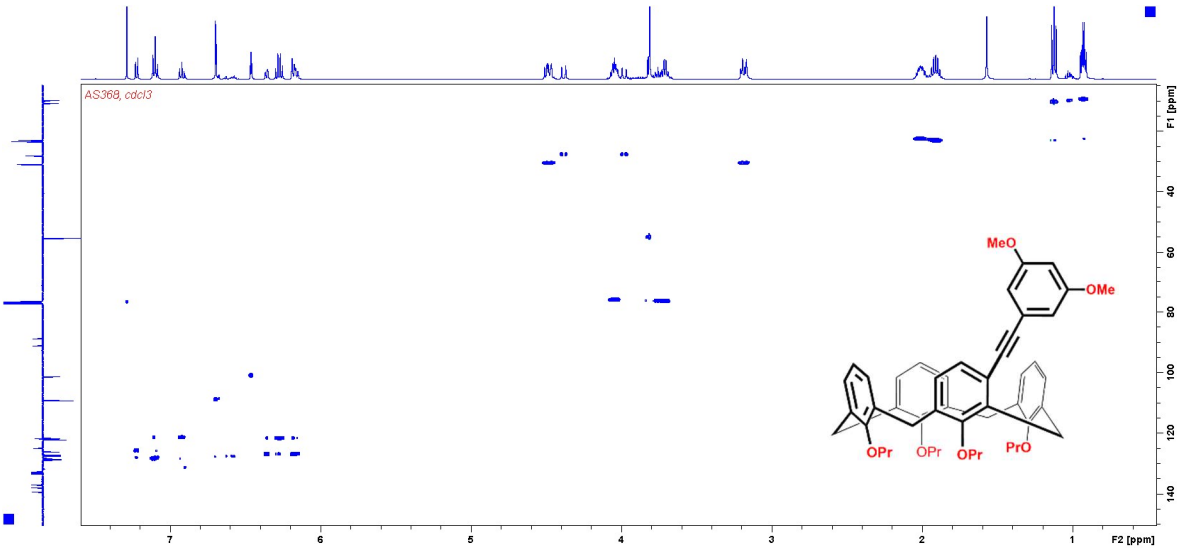


Figure S5: HSQC NMR of compound **5a** ($CDCl_3$, 125 MHz, 298 K)

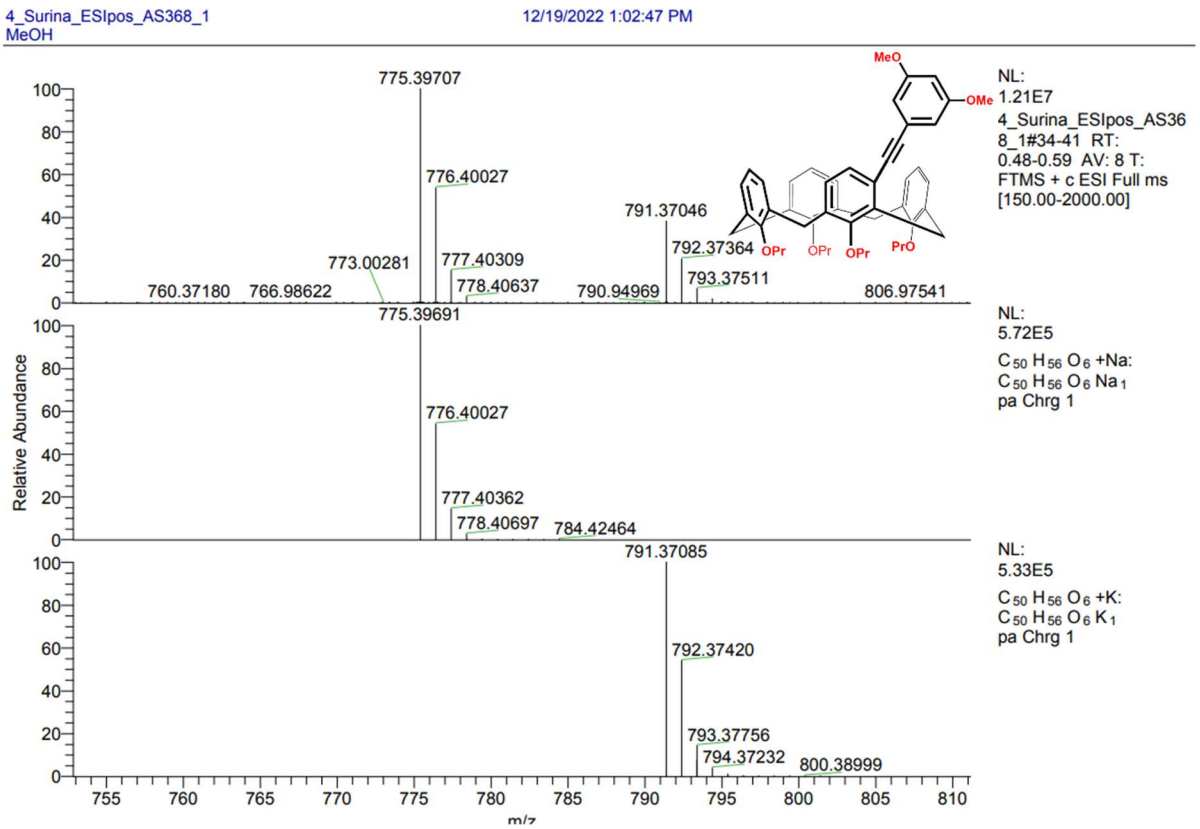


Figure S6: HRMS of compound **5a** (ESI^+)

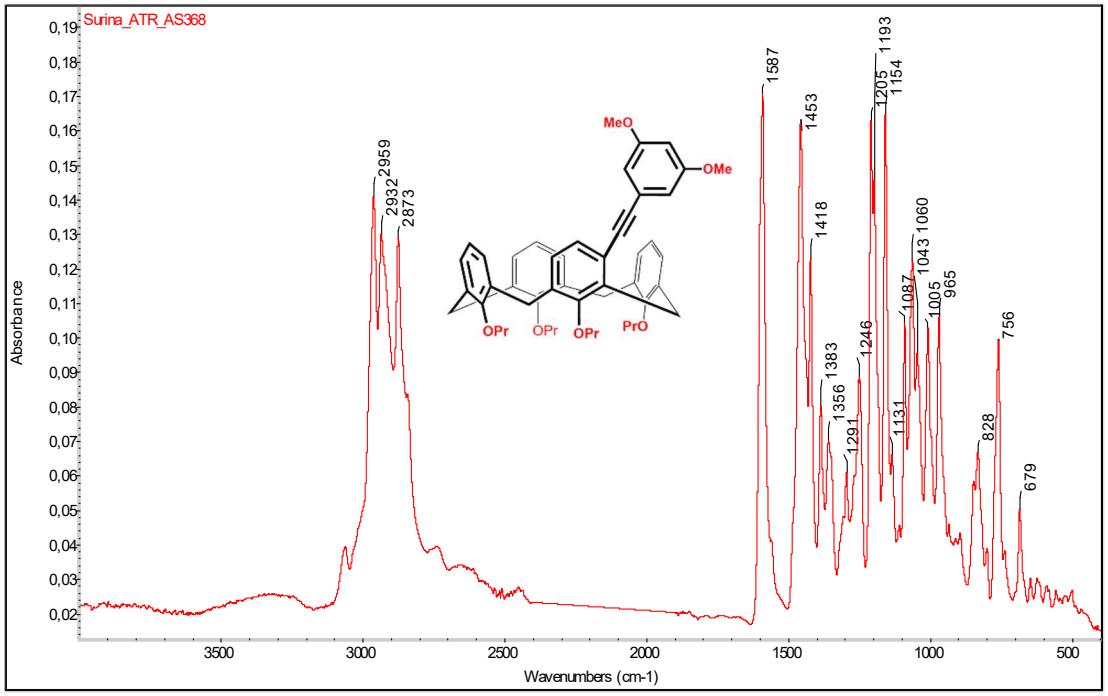


Figure S7: IR of compound **5a** (KBr)

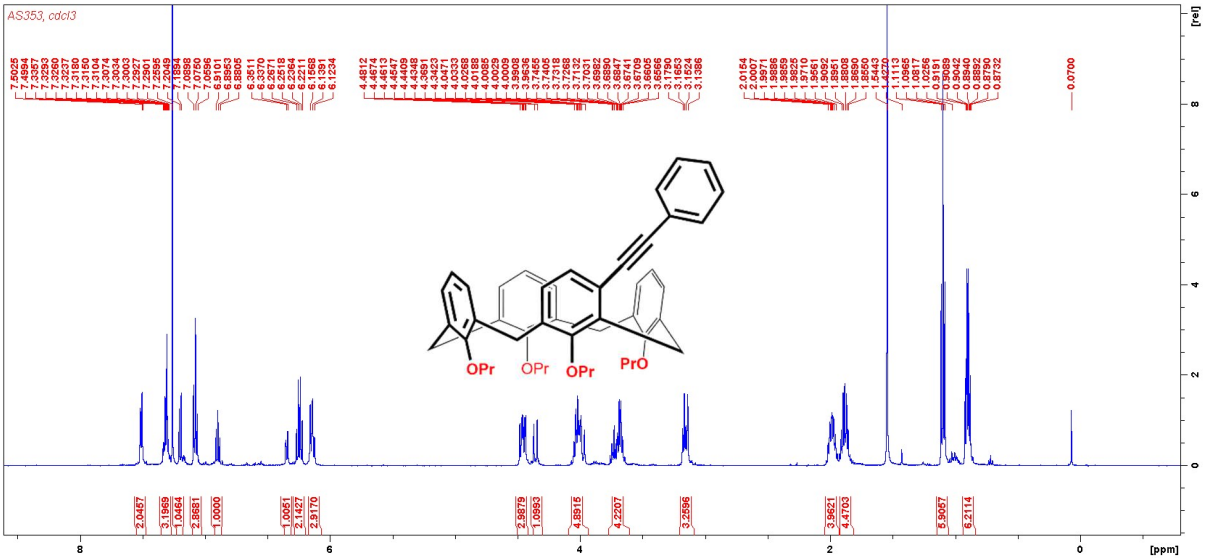


Figure S8: ^{1}H NMR of compound **5b** ($CDCl_3$, 500 MHz, 298 K)

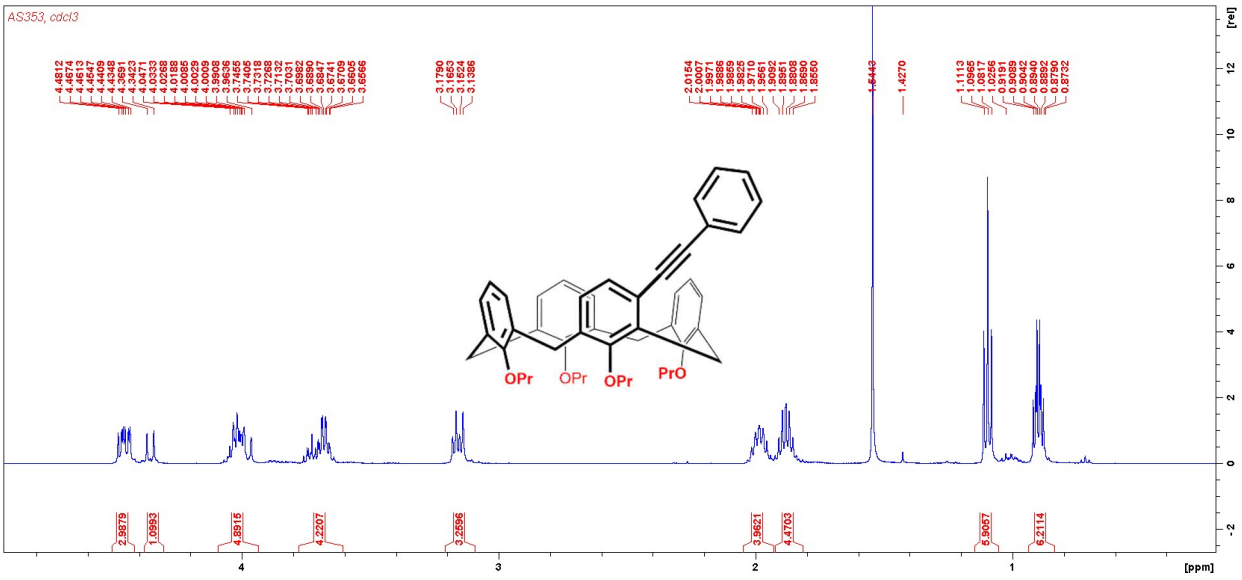


Figure S9: ^{1}H NMR of compound **5b,** aliphatic region ($CDCl_3$, 500 MHz, 298 K)

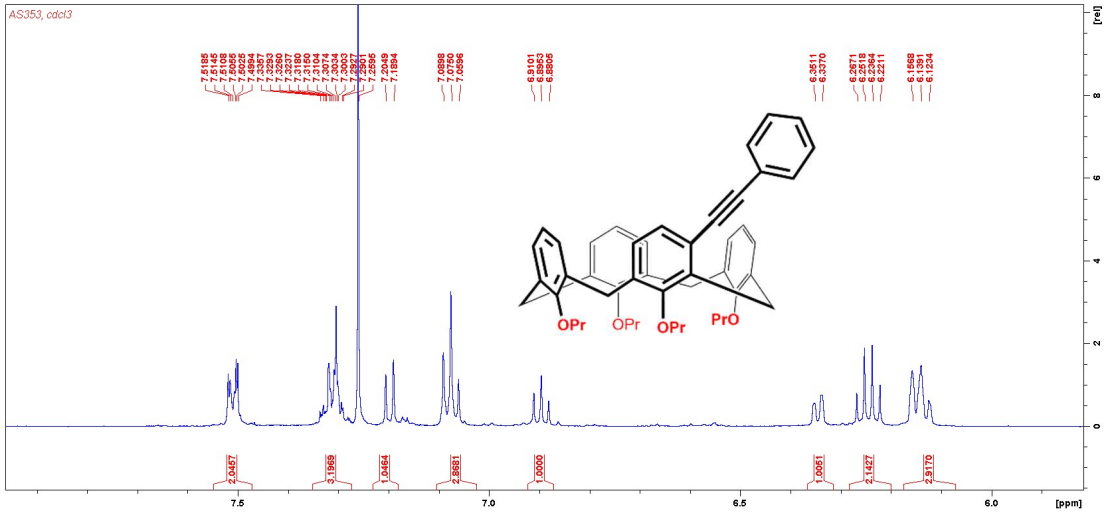


Figure S10: ^{1}H NMR of compound **5b,** aromatic region ($CDCl_3$, 500 MHz, 298 K)

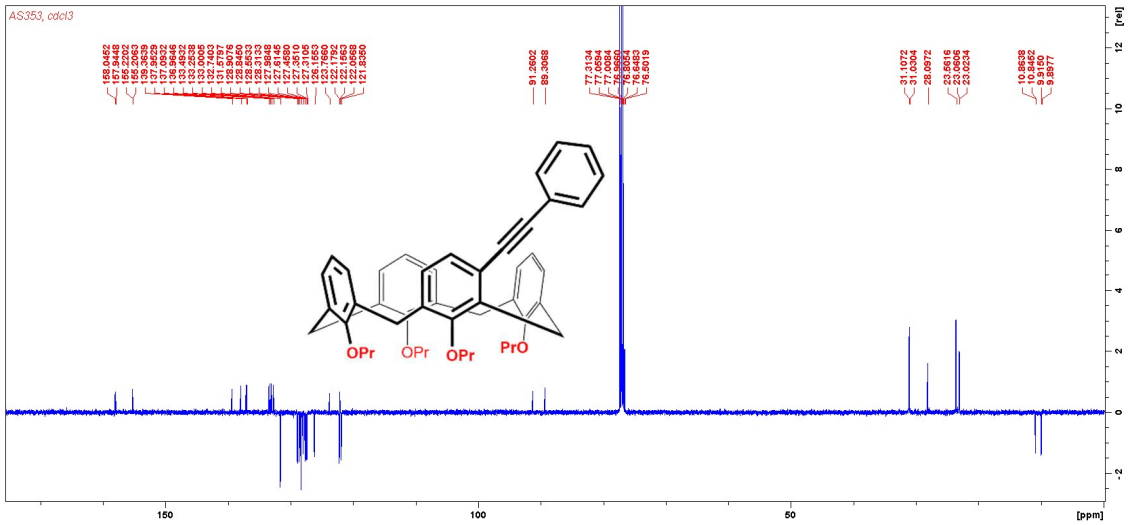


Figure S11: ^{13}C(APT) NMR of compound **5b** ($CDCl_3$, 125 MHz, 298 K)

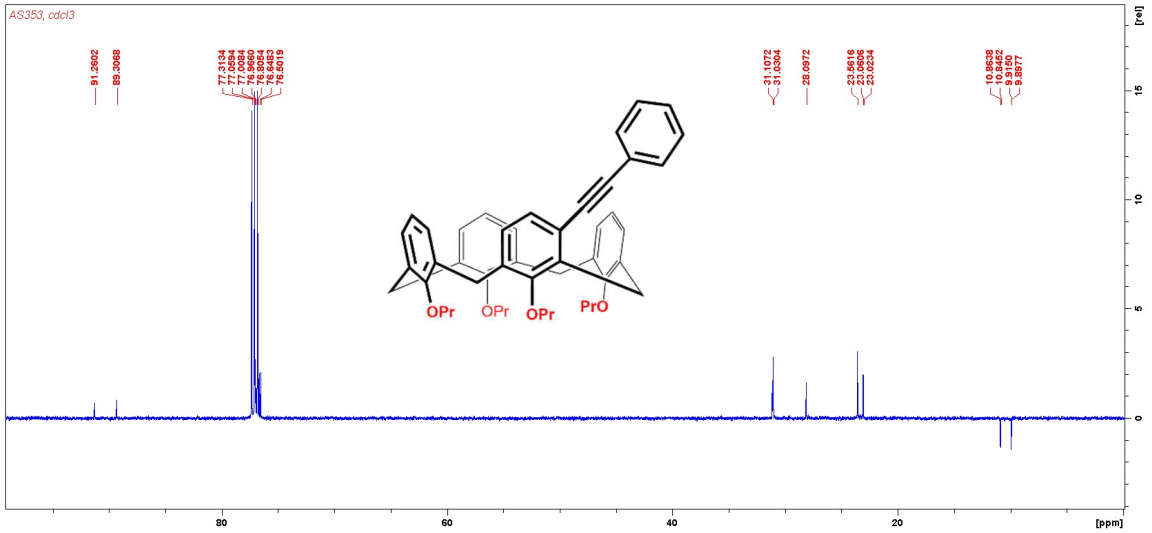


Figure S12: ^{13}C(APT) NMR of compound **5b,** aliphatic region ($CDCl_3$, 125 MHz, 298 K)

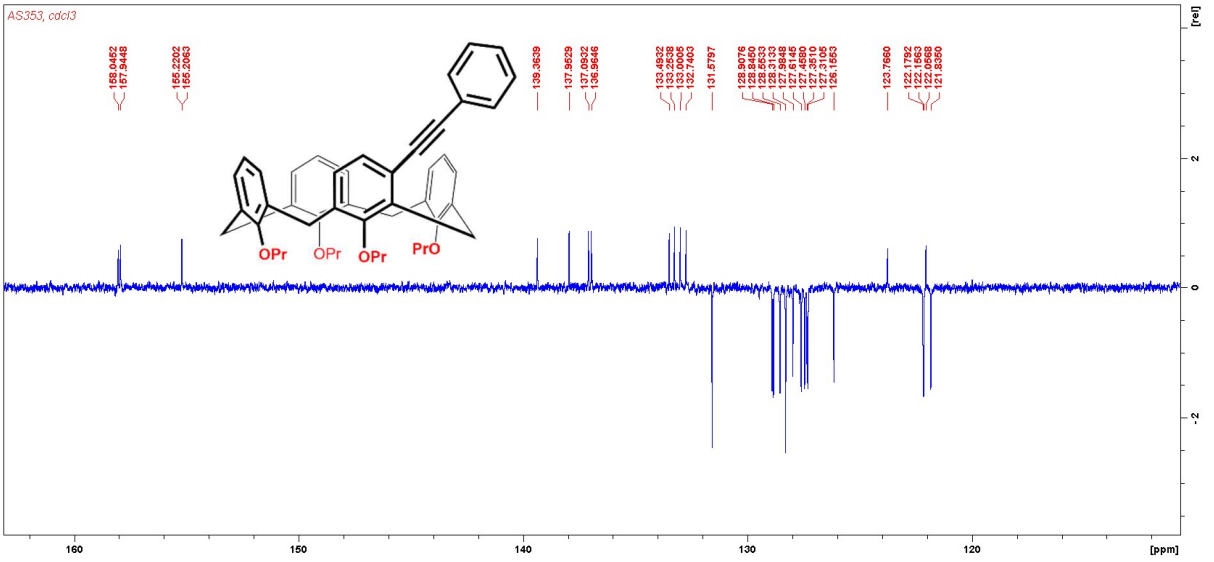


Figure S13: ^{13}C(APT) NMR of compound **5b,** aromatic region ($CDCl_3$, 125 MHz, 298 K)

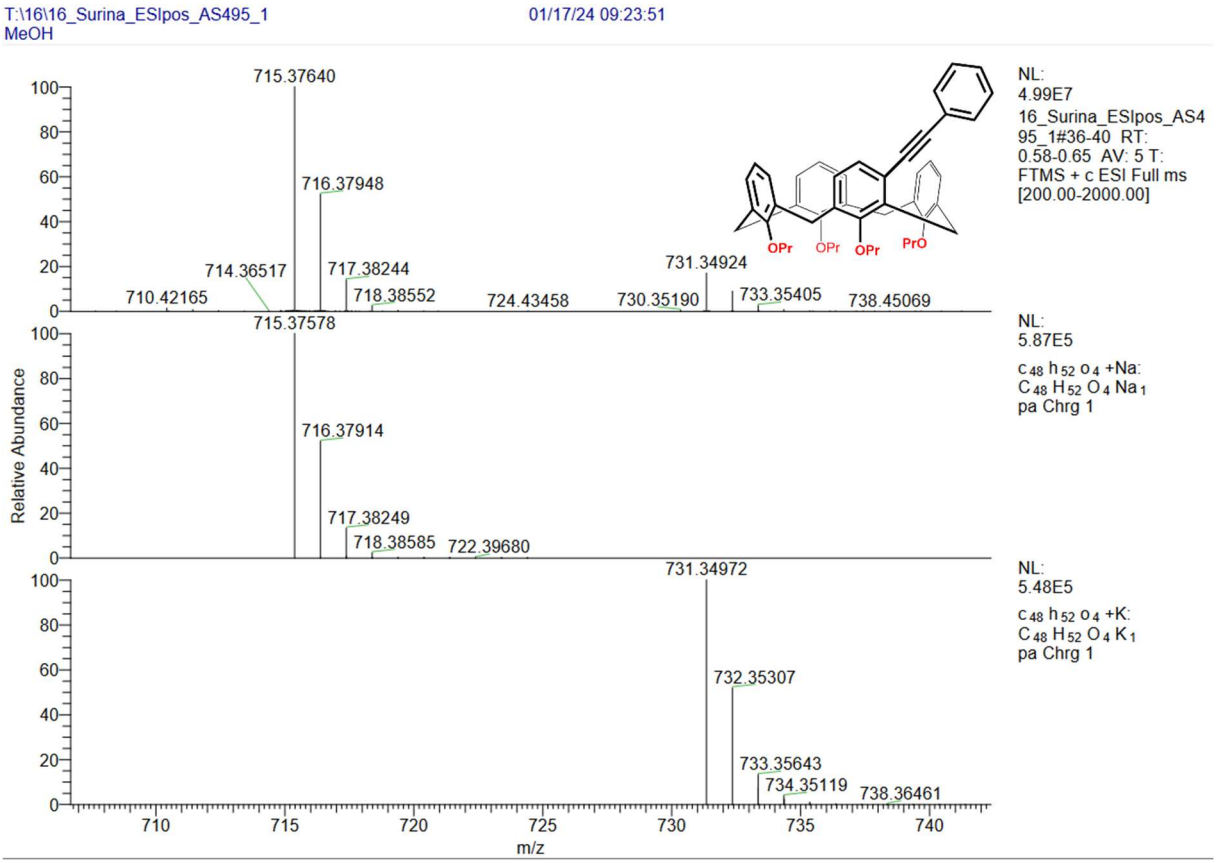


Figure 14: HRMS of compound **5b** (ESI^+)

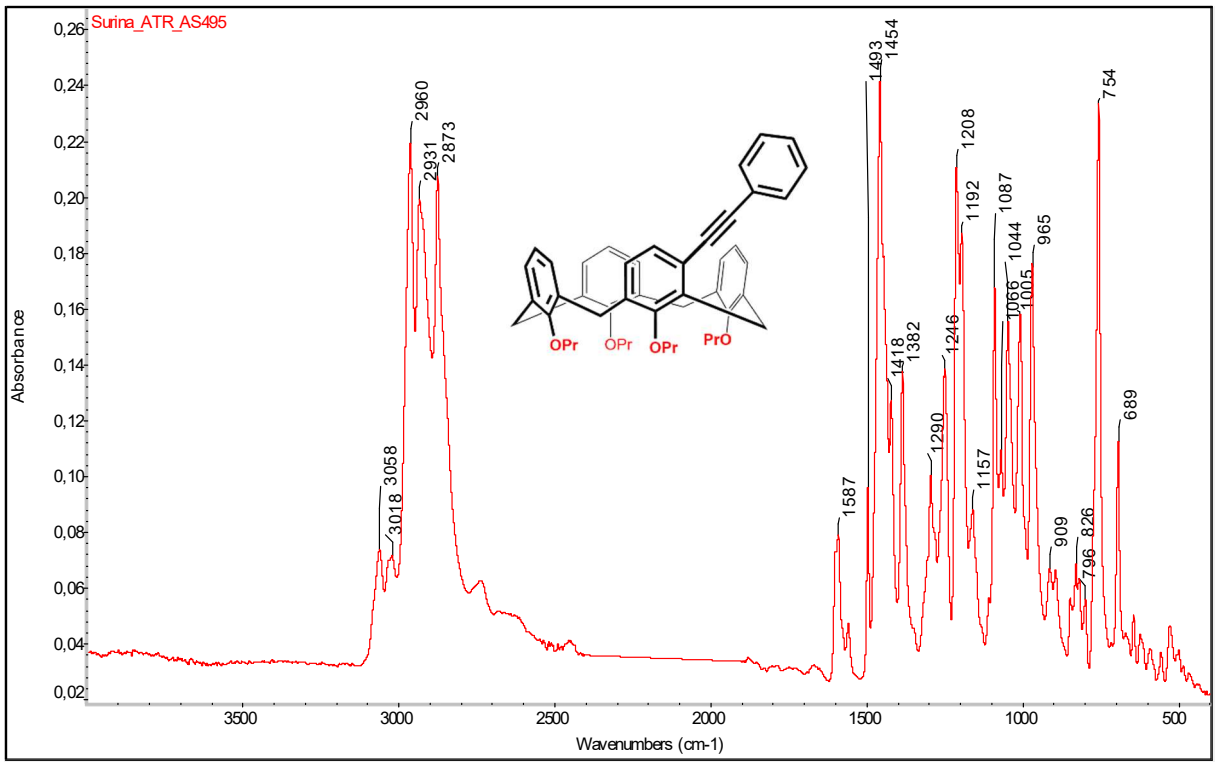


Figure S15: IR of compound **5b** (KBr)

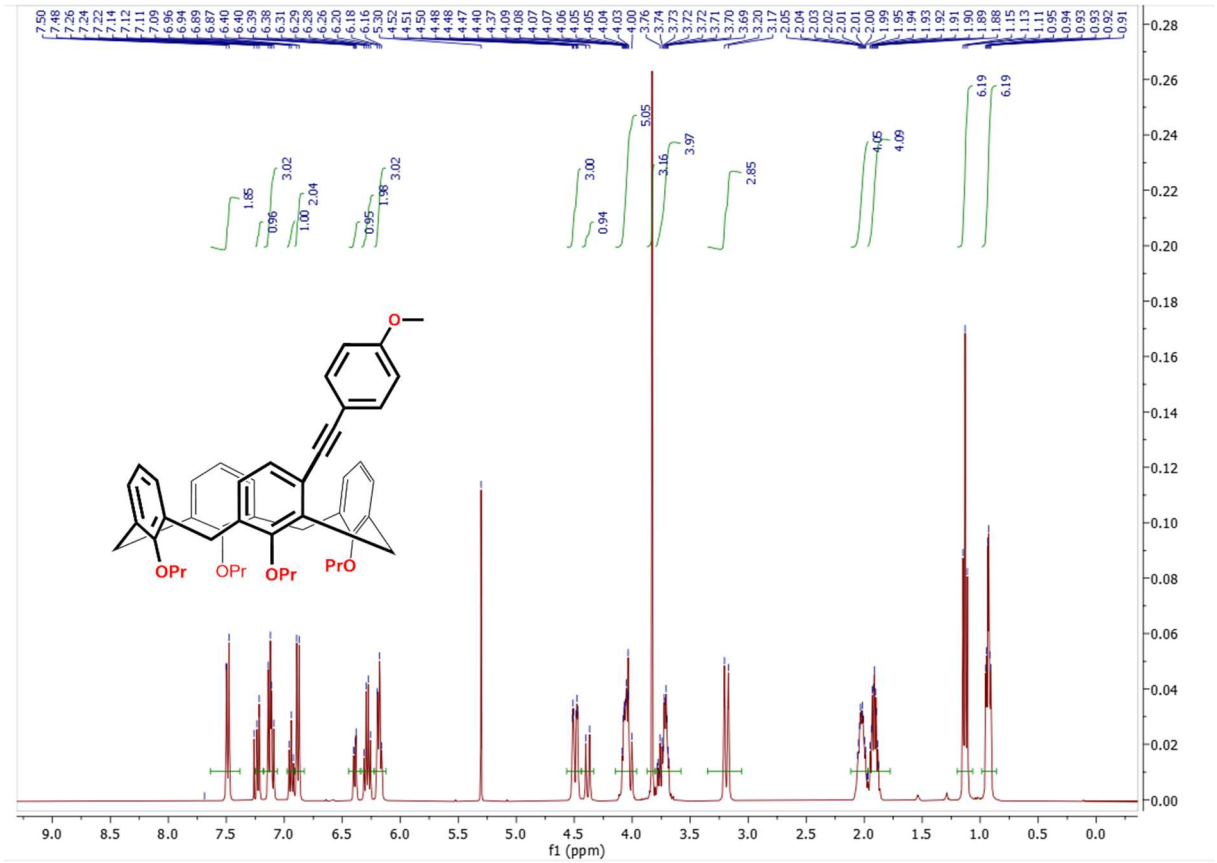


Figure S16: ^{1}H NMR of compound **5c** ($CDCl_3$, 400 MHz, 298 K)

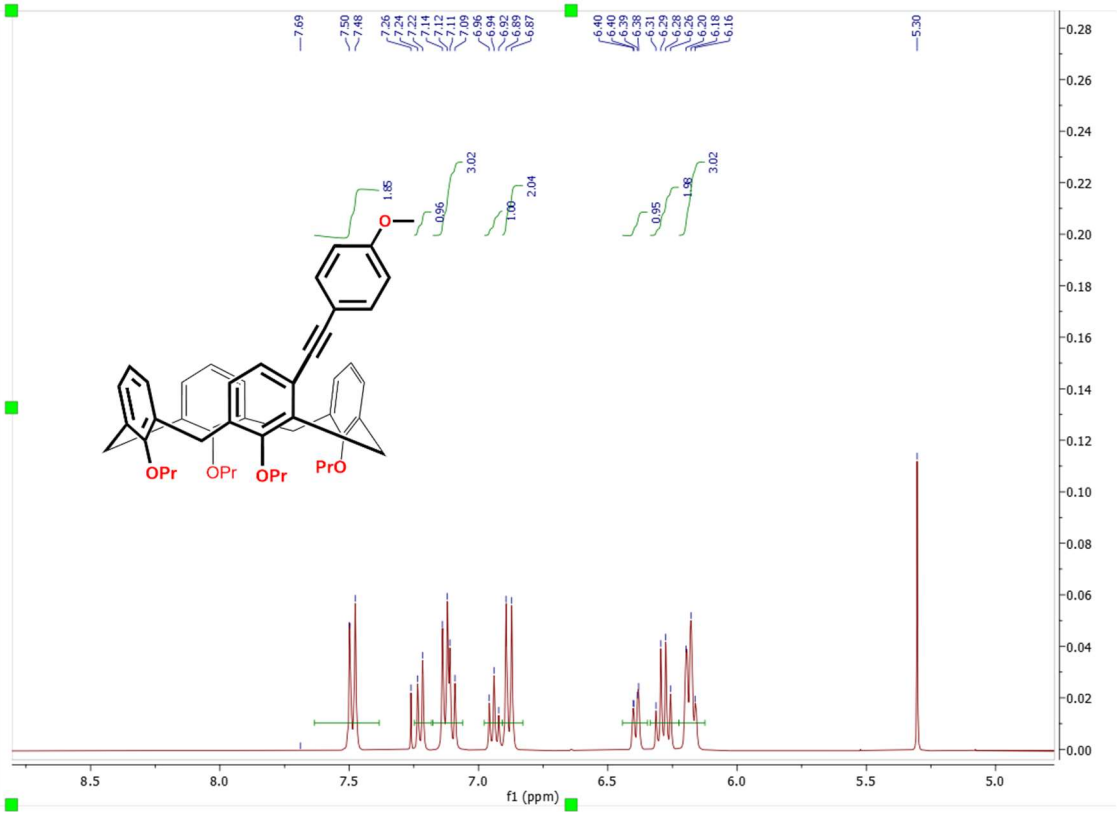


Figure S17: ^{1}H NMR of compound **5c**, aromatic region ($CDCl_3$, 400 MHz, 298 K)

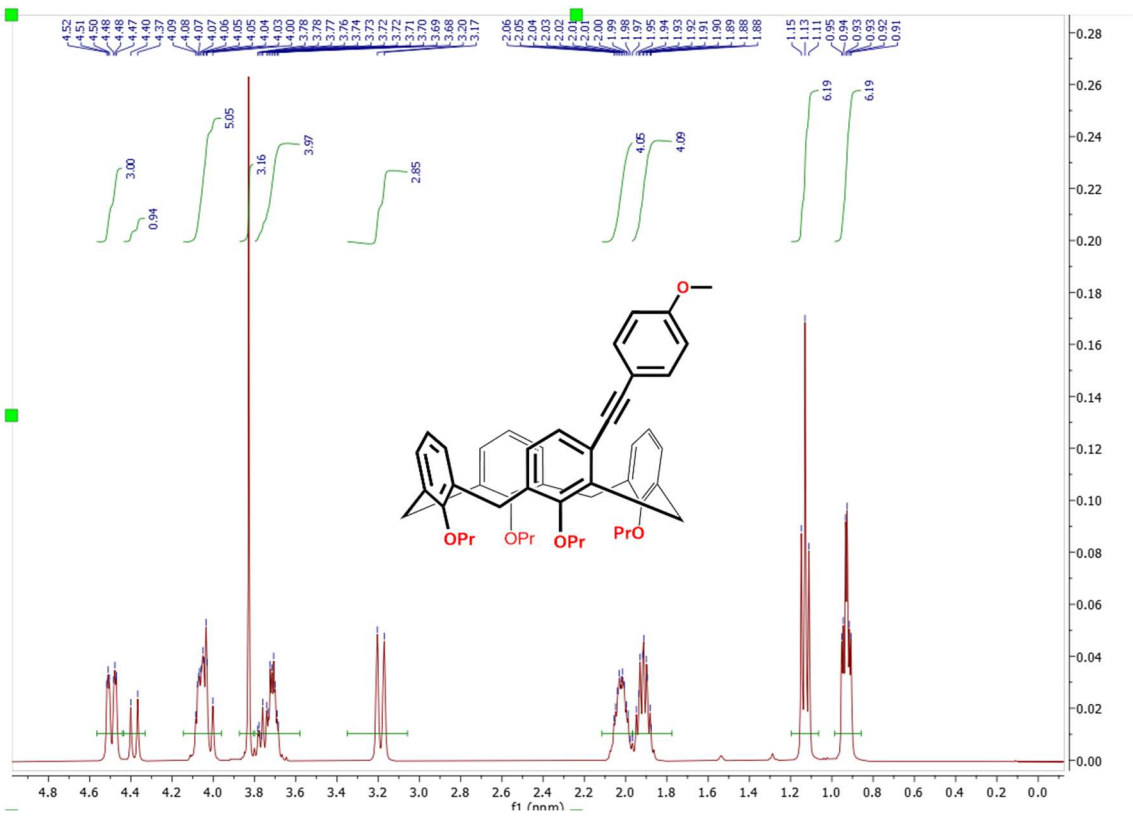


Figure S18: ^{1}H NMR of compound **5c**, aliphatic region ($CDCl_3$, 400 MHz, 298 K)

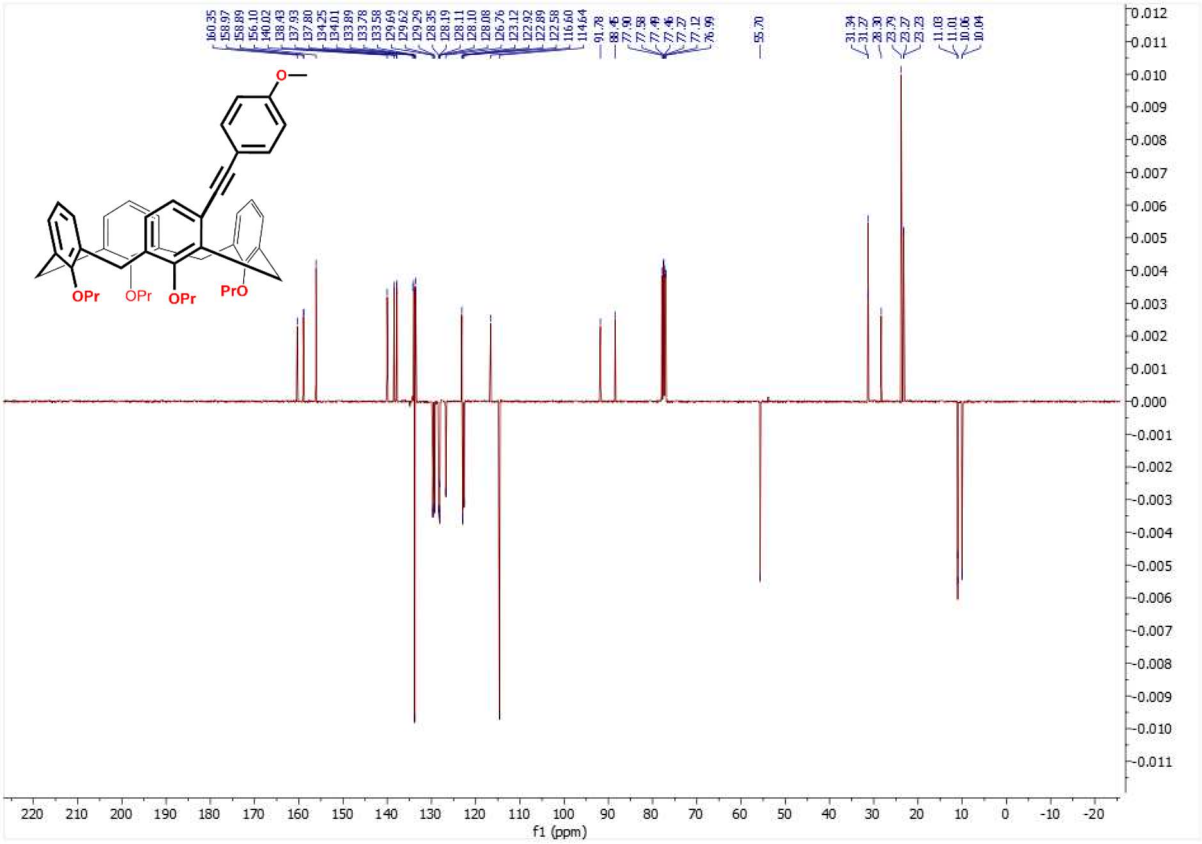


Figure S19: ^{13}C(APT) NMR of compound **5c** ($CDCl_3$, 100 MHz, 298 K)

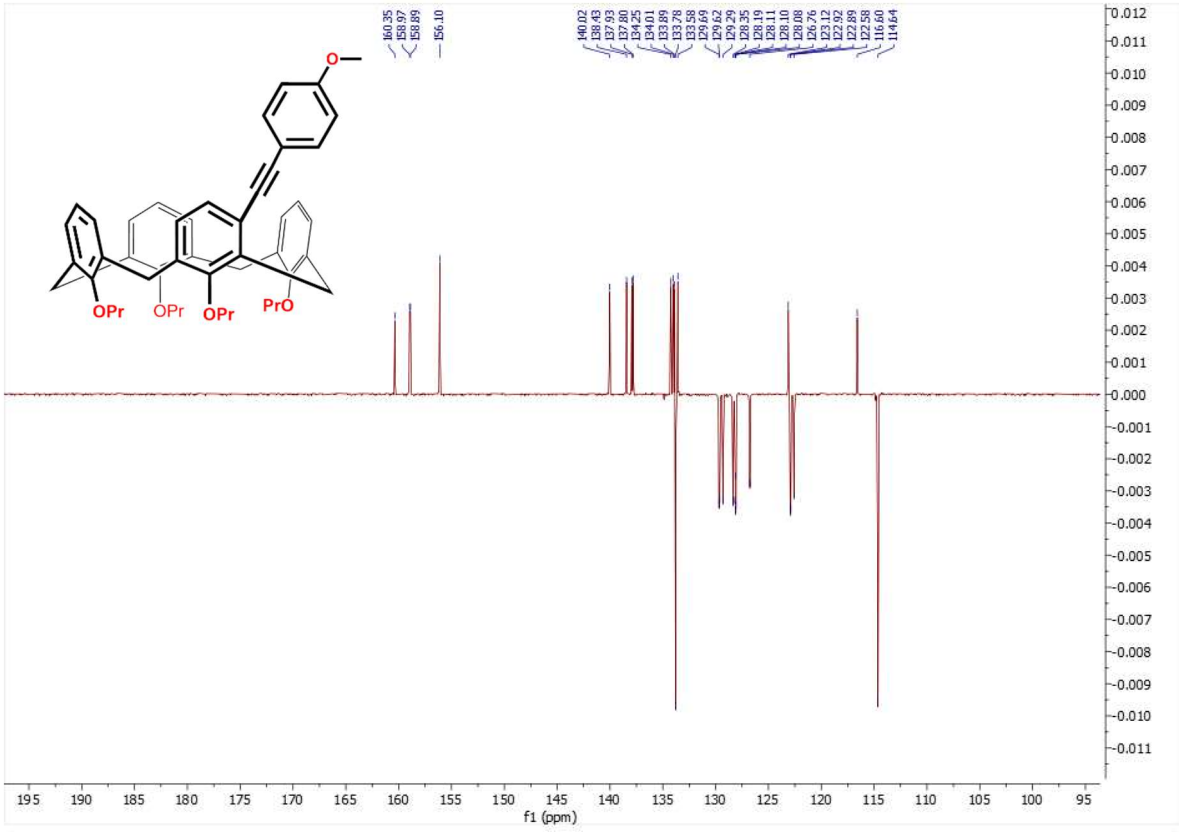


Figure S20: ^{13}C(APT) NMR of compound **5c,** aromatic region ($CDCl_3$, 100 MHz, 298 K)

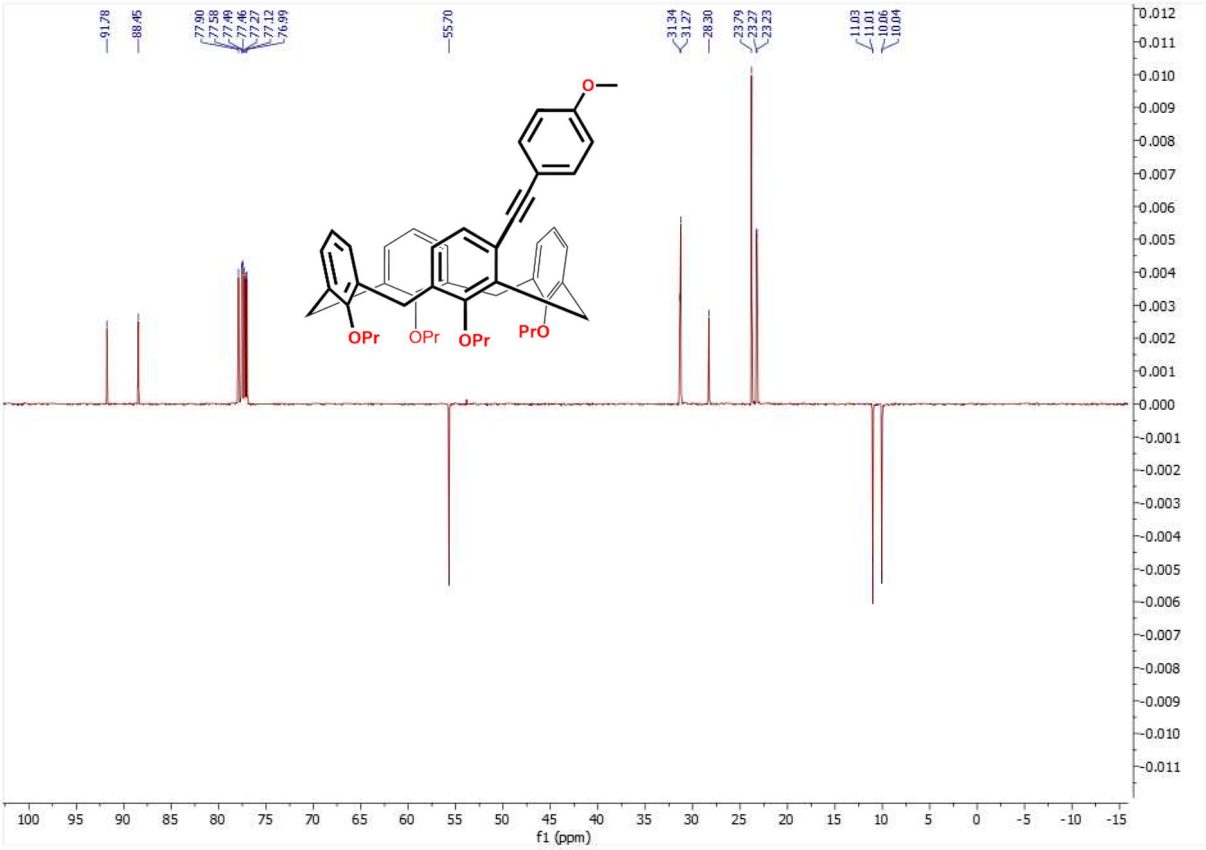


Figure S21: ^{13}C(APT) NMR of compound **5c,** aliphatic region ($CDCl_3$, 100 MHz, 298 K)

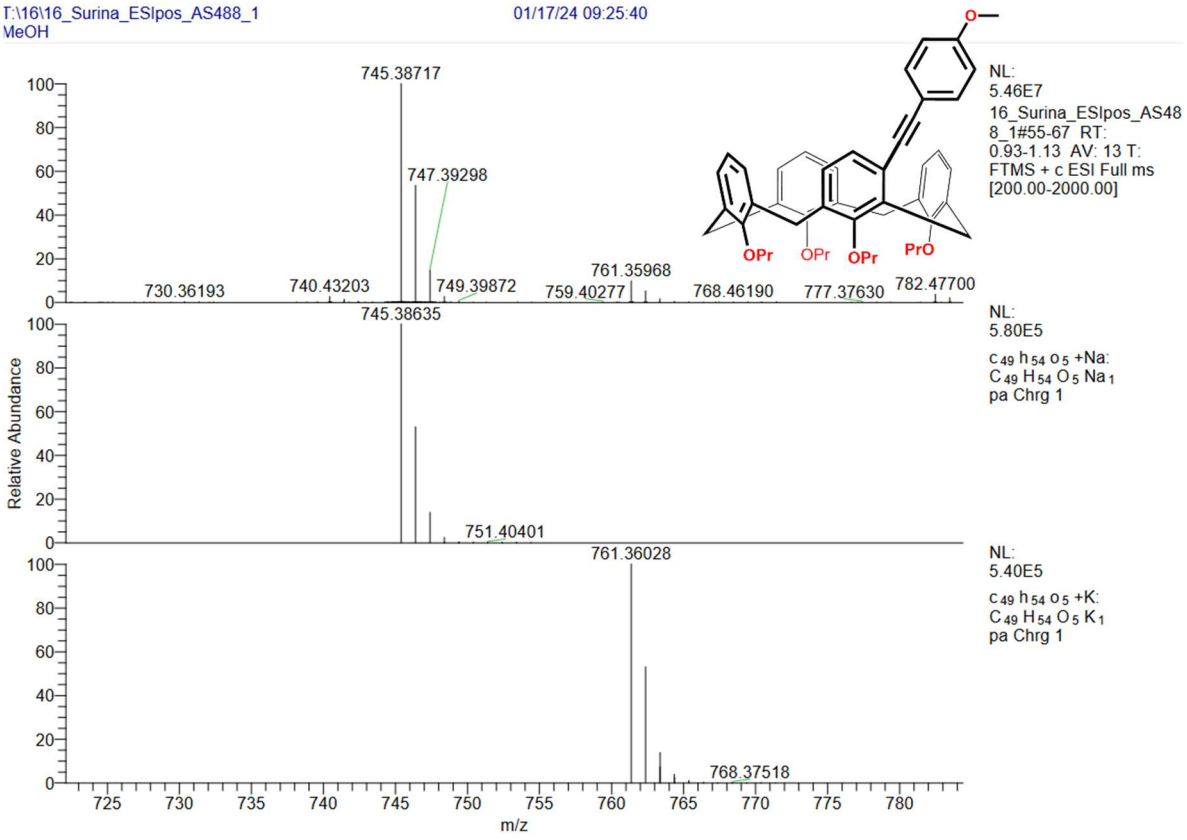


Figure S22: HRMS of compound **5c** (ESI^+)

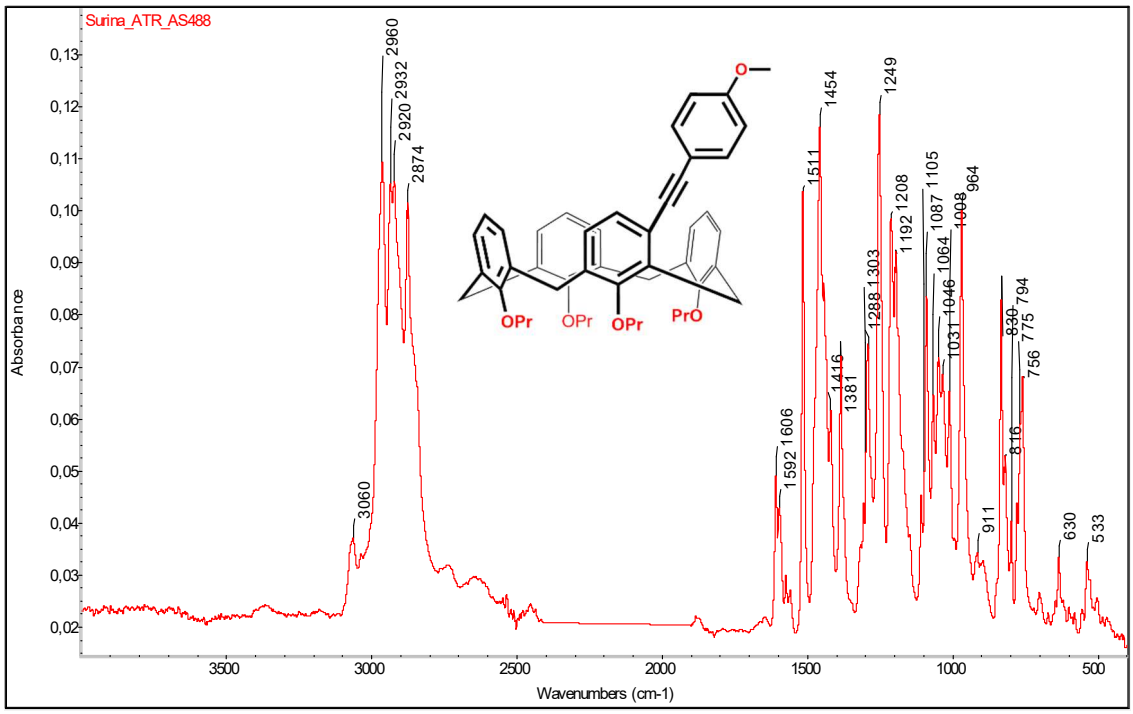


Figure S23: IR of compound **5c** (KBr)

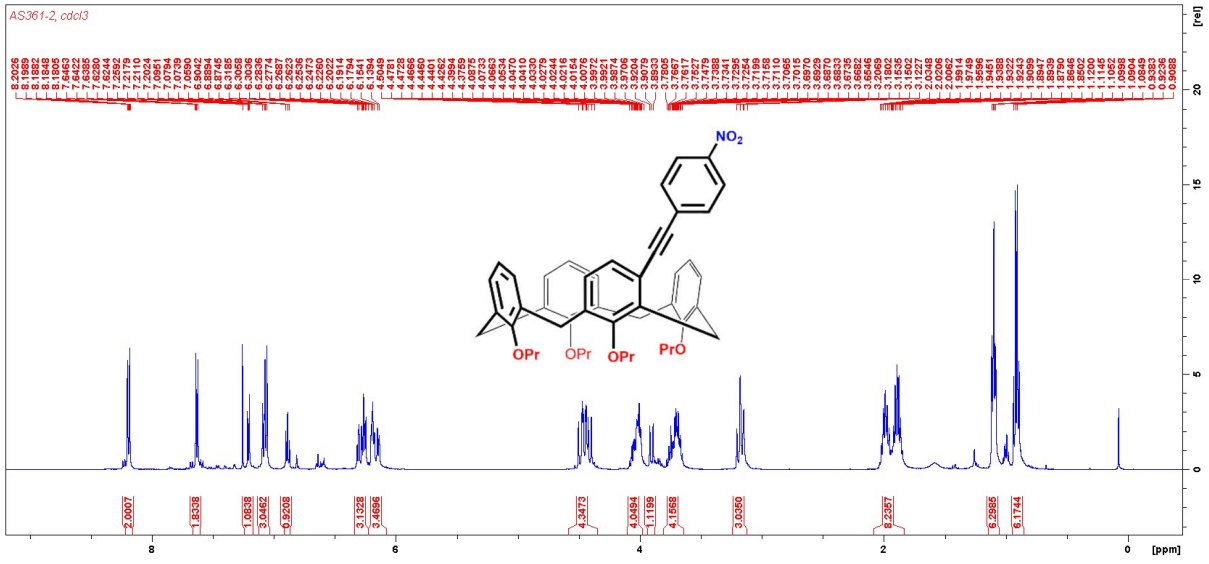


Figure S24: ^{1}H NMR of compound **5d** ($CDCl_3$, 500 MHz, 298 K)

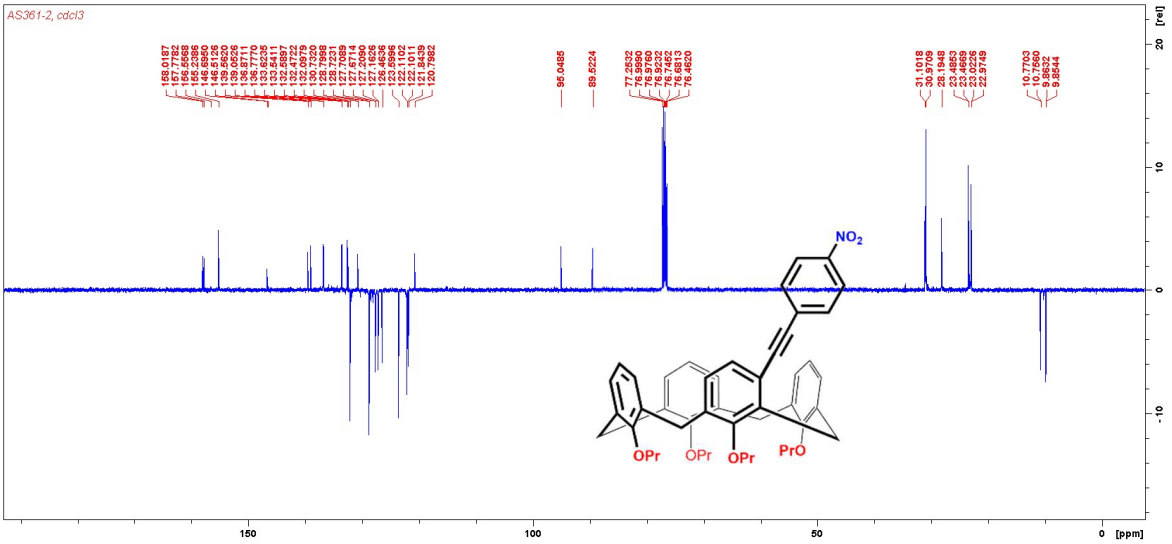


Figure S25: ^{13}C(APT) NMR of compound **5d** ($CDCl_3$, 125 MHz, 298 K)

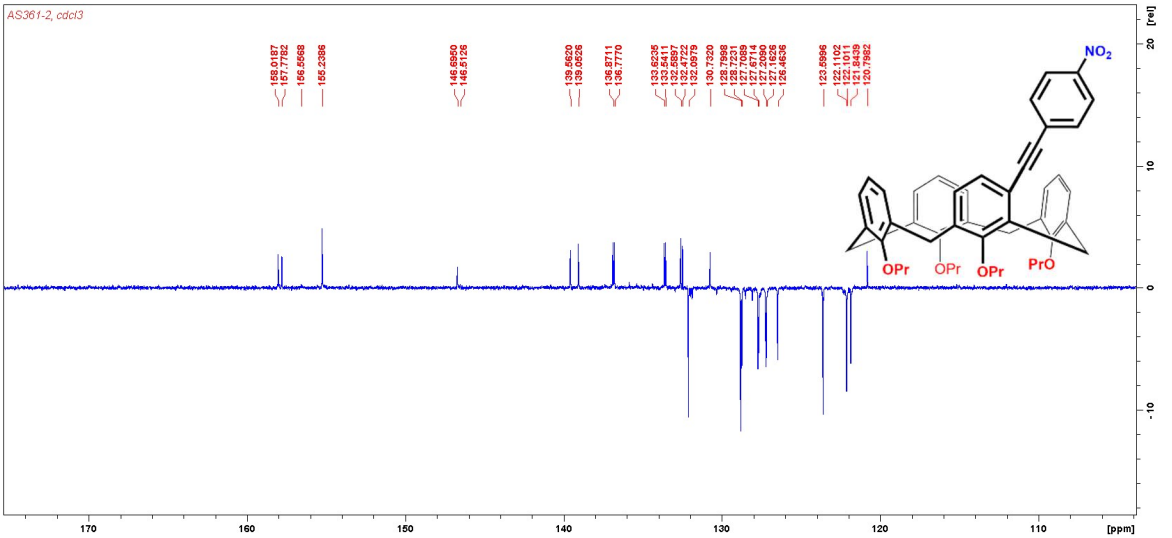


Figure S26: ^{13}C(APT) NMR of compound **5d,** aromatic region ($CDCl_3$, 125 MHz, 298 K)

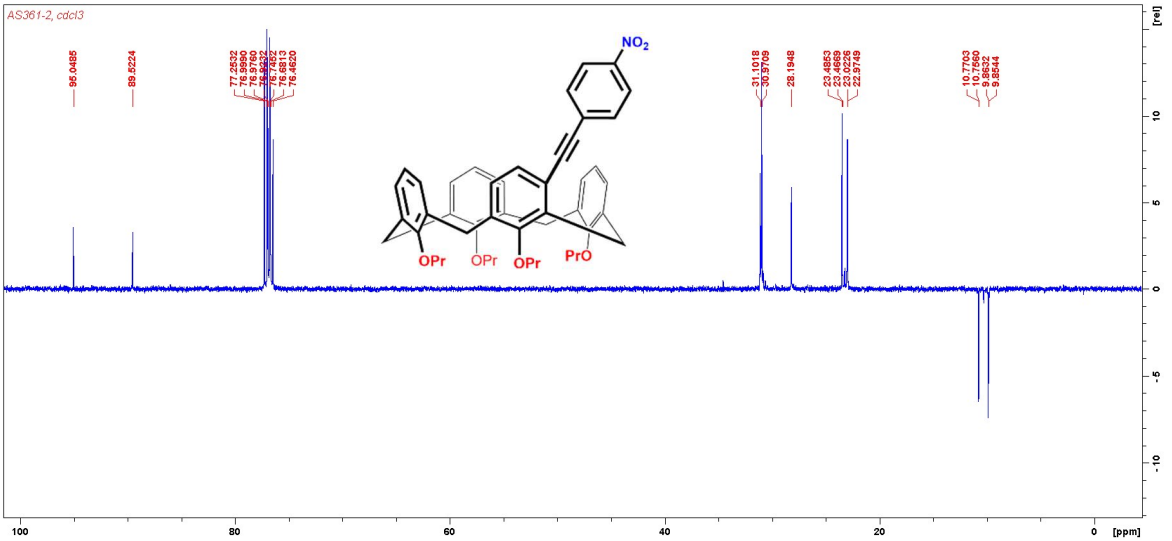


Figure S27: ^{13}C(APT) NMR of compound **5d,** aliphatic region ($CDCl_3$, 125 MHz, 298 K)

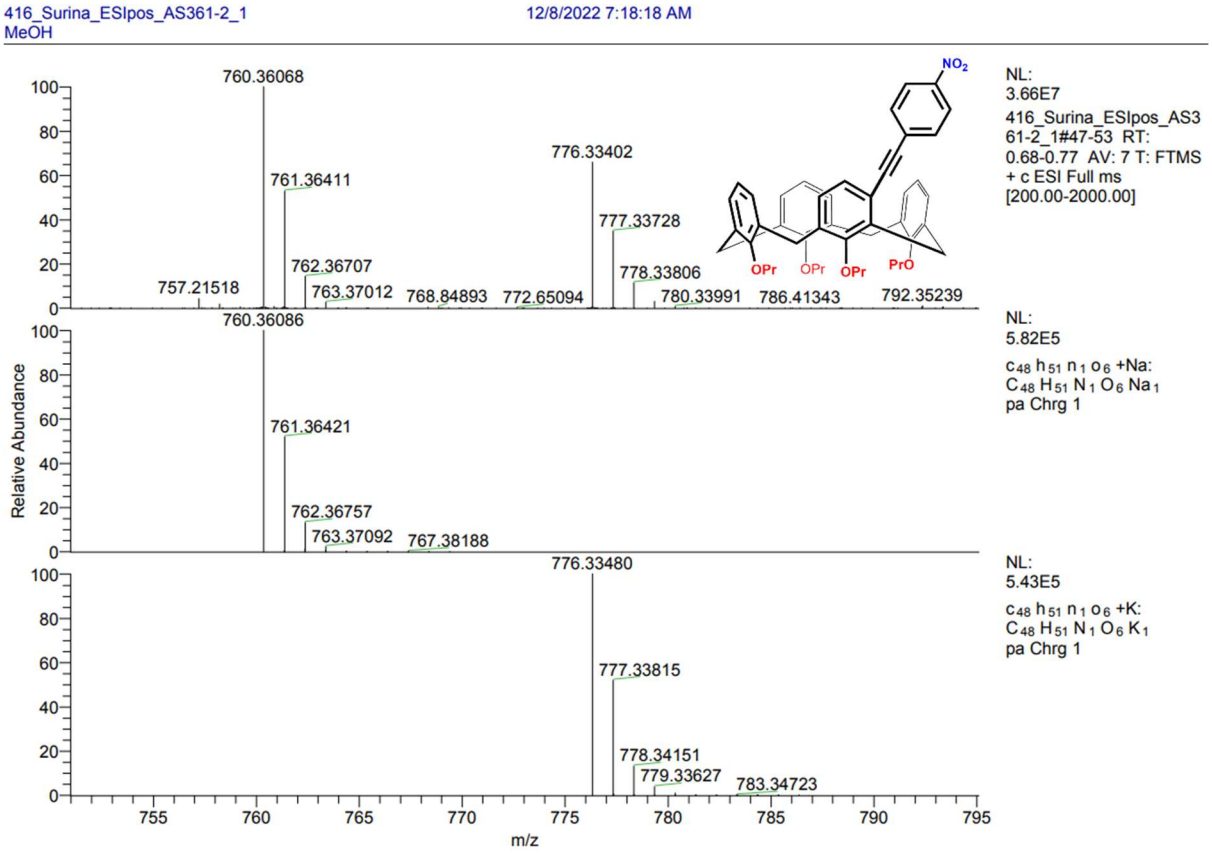


Figure S28: HRMS of compound **5d** (ESI^+)

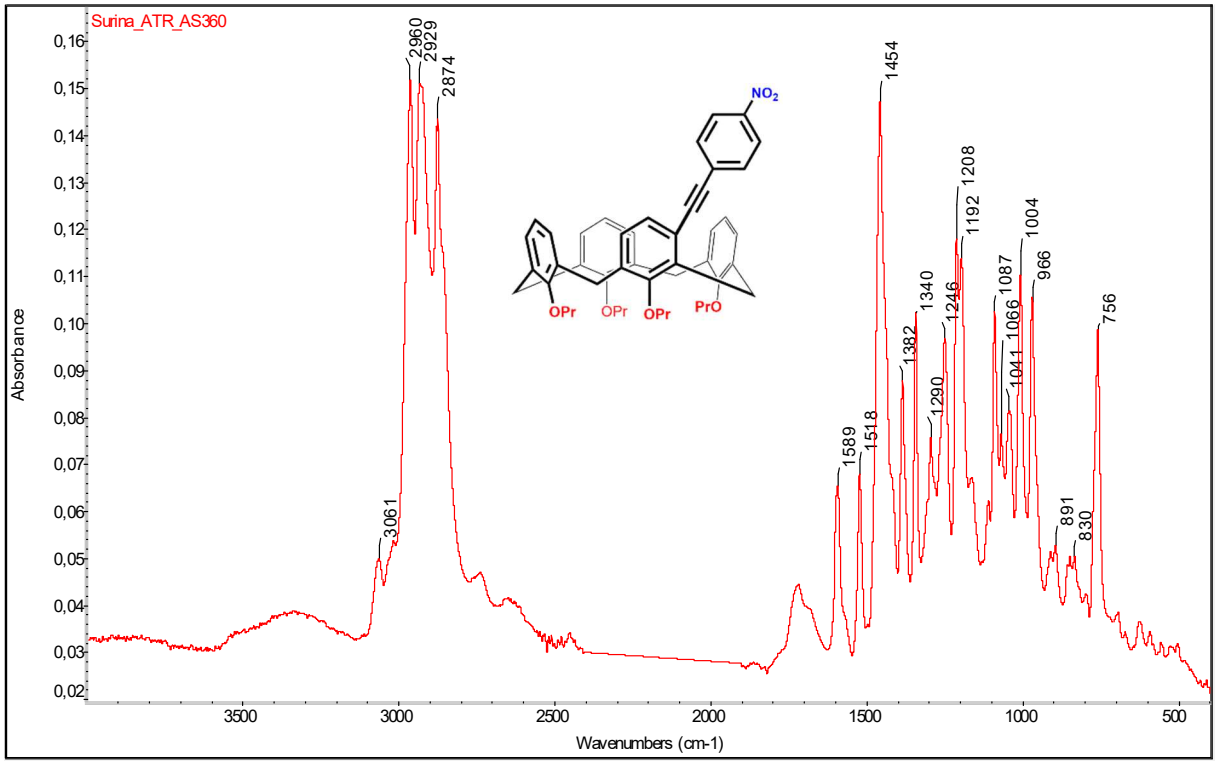


Figure S29: IR of compound **5d** (KBr)

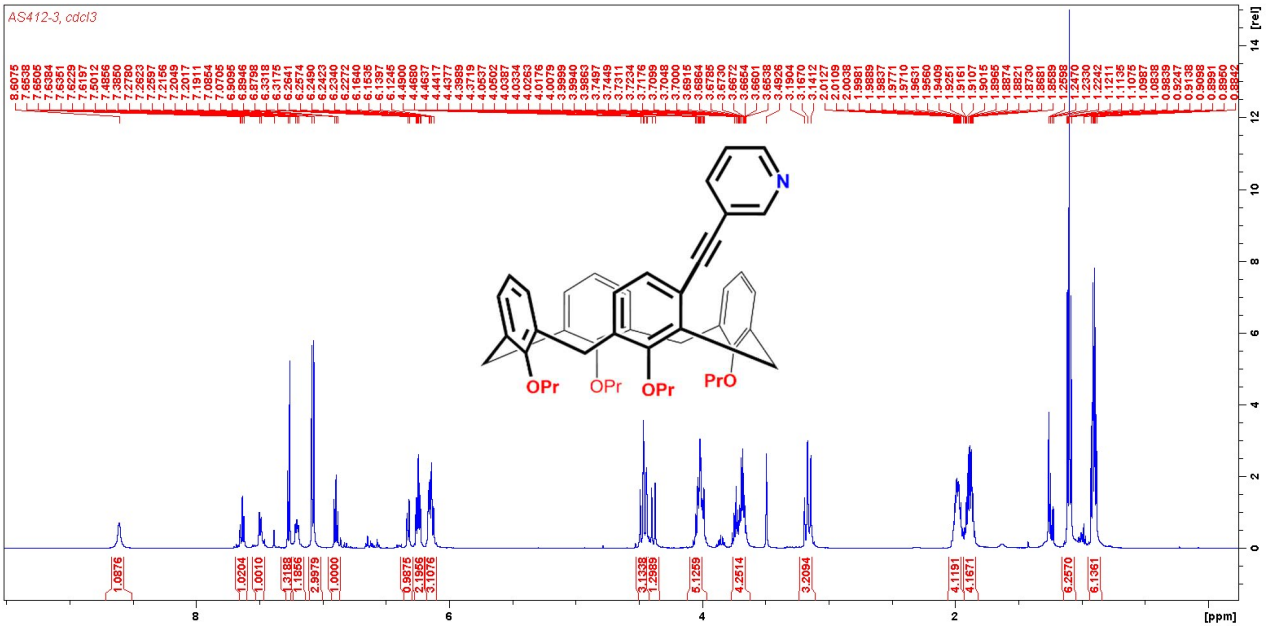


Figure S30: ^{1}H NMR of compound **5e** ($CDCl_3$, 500 MHz, 298 K)

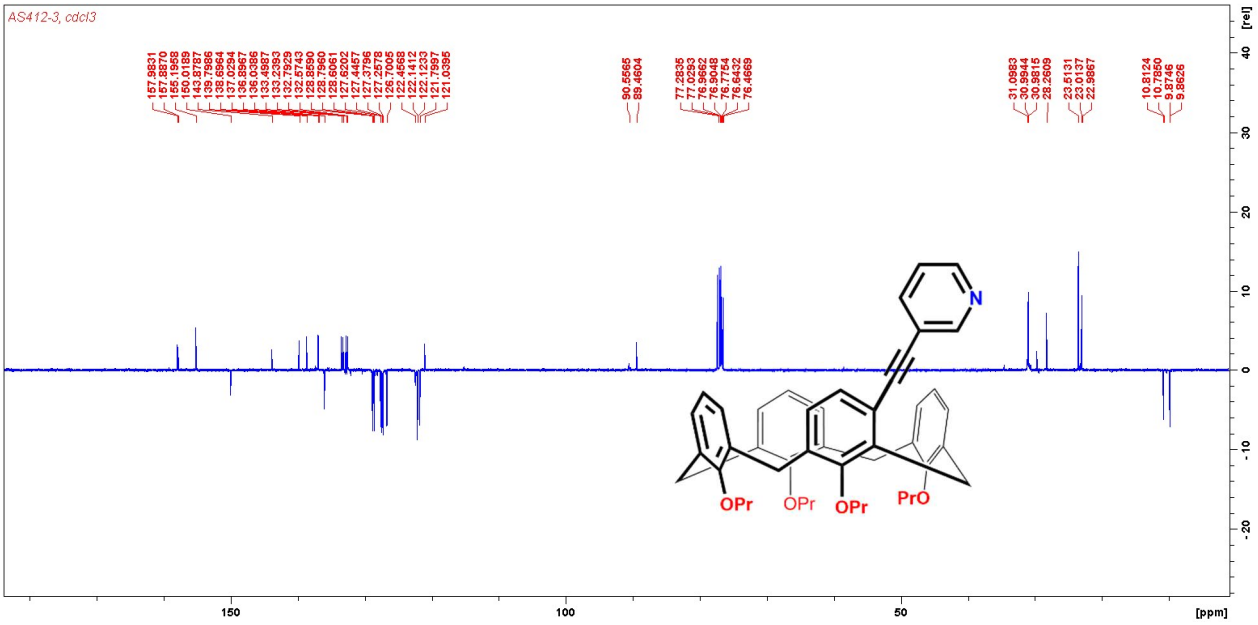


Figure S31: ^{13}C(APT) NMR of compound **5e** ($CDCl_3$, 125 MHz, 298 K)

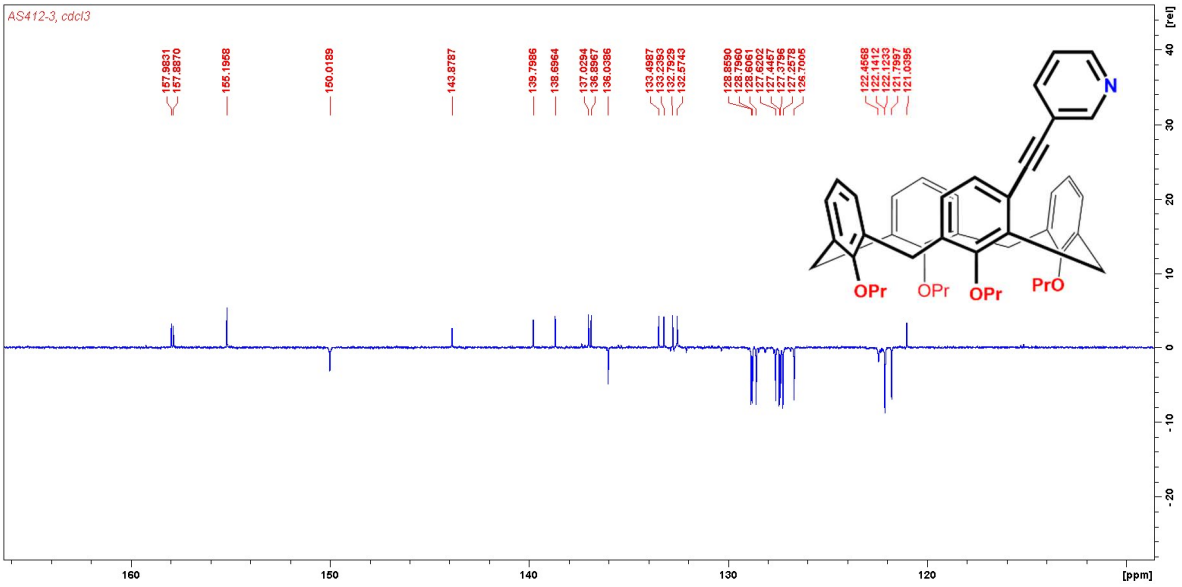


Figure S32: ^{13}C(APT) NMR of compound **5e,** aromatic region ($CDCl_3$, 125 MHz, 298 K)

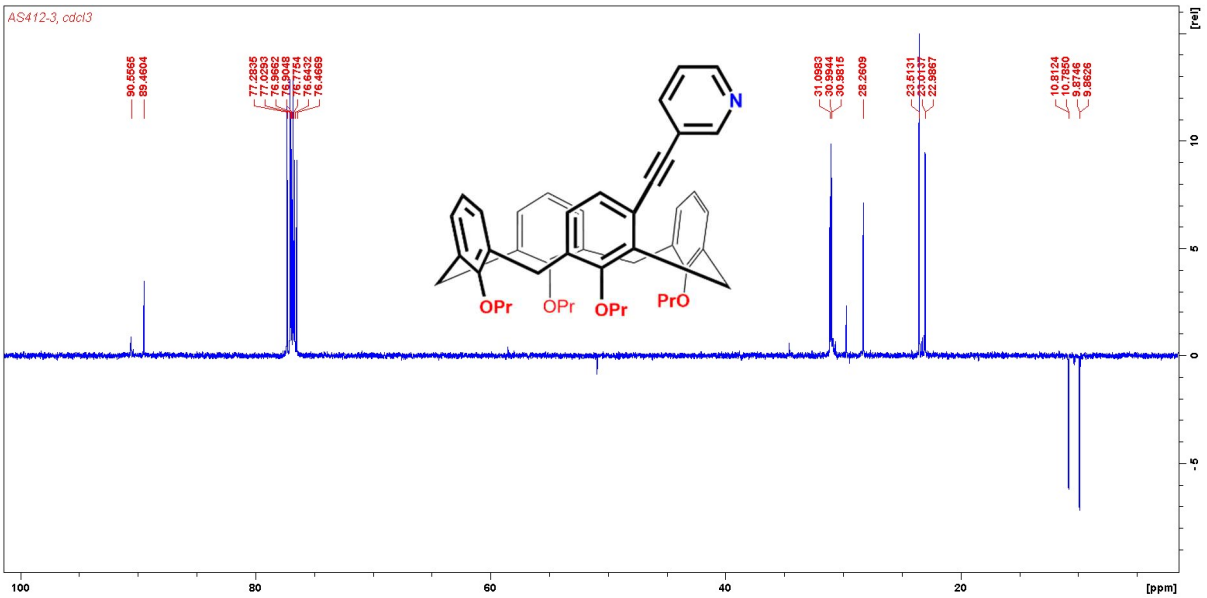


Figure S33: ^{13}C(APT) NMR of compound **5e,** aliphatic region ($CDCl_3$, 125 MHz, 298 K)

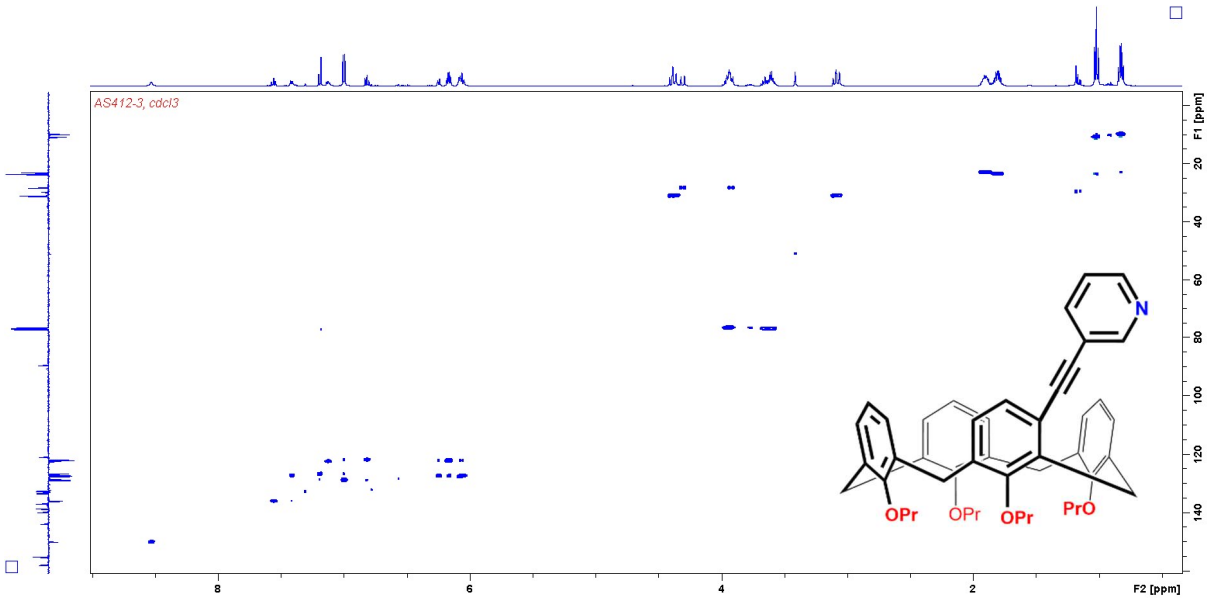


Figure S34: HSQC NMR of compound **5e** ($CDCl_3$, 125 MHz, 298 K)

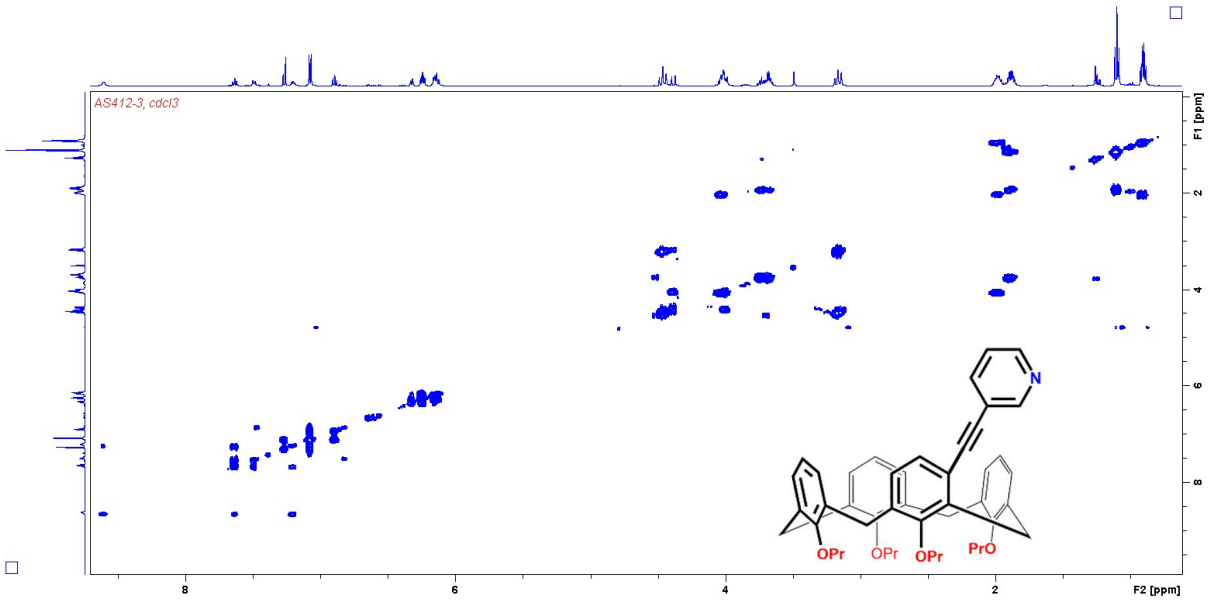


Figure S35: HMBC NMR of compound **5e** ($CDCl_3$, 125 MHz, 298 K)

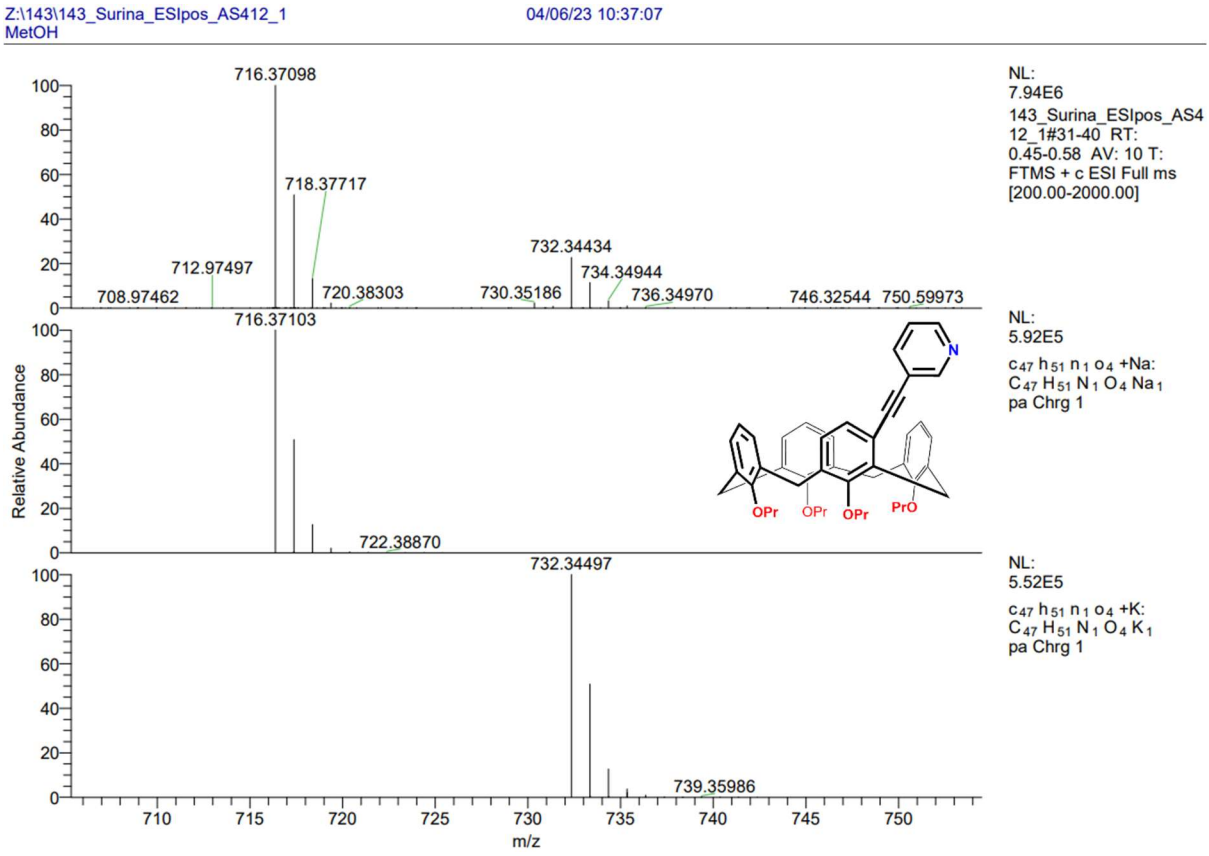


Figure S36: HRMS of compound **5e** (ESI^+)

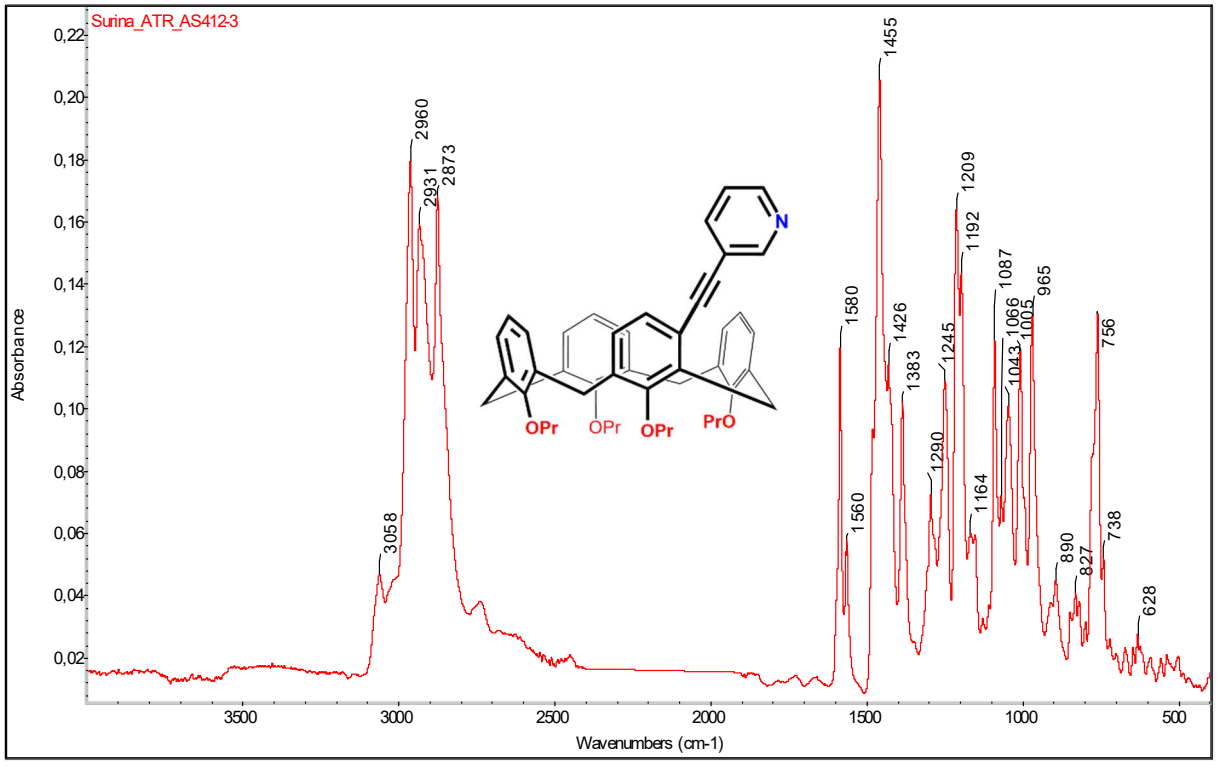


Figure S37: IR of compound **5e** (KBr)

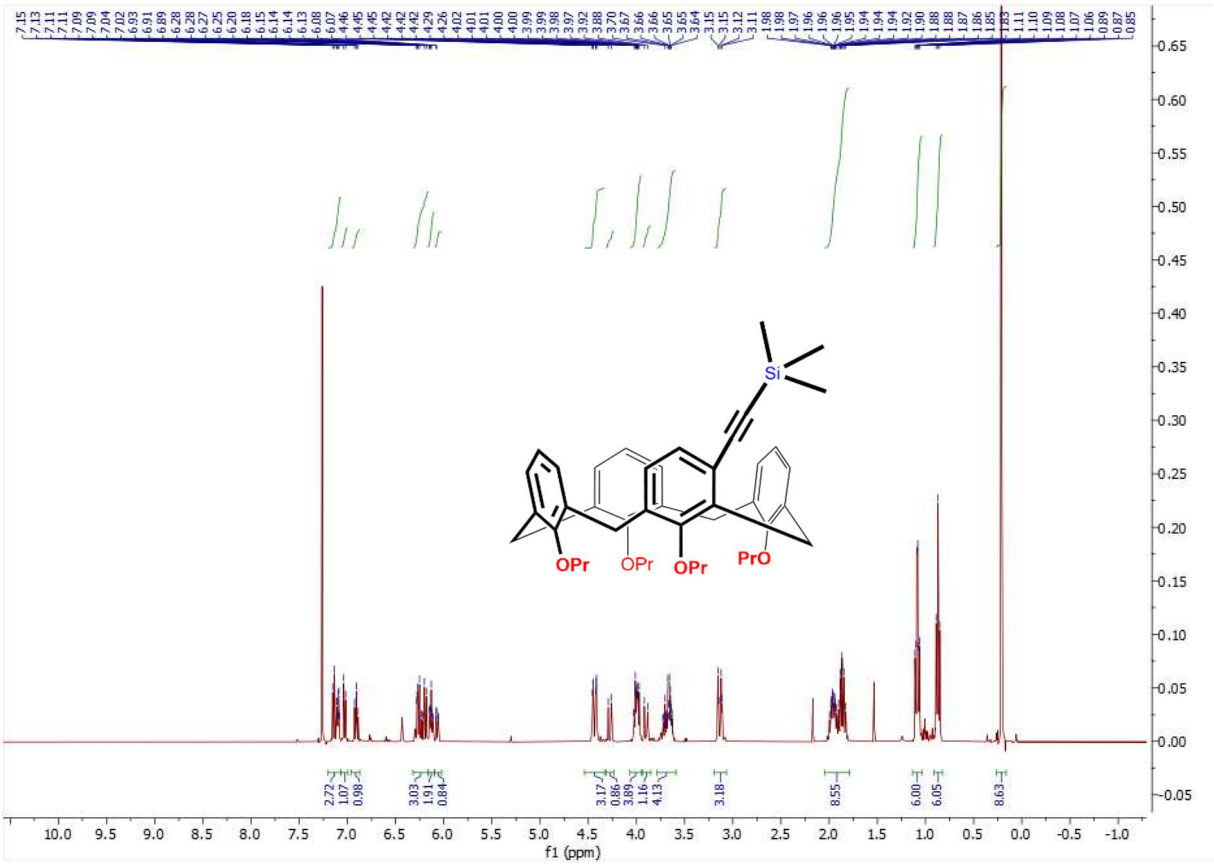


Figure S38: ^{1}H NMR of compound **5f** ($CDCl_3$, 500 MHz, 298 K)



Figure S39: ^{13}C(APT) NMR of compound **5f** ($CDCl_3$, 100 MHz, 298 K)

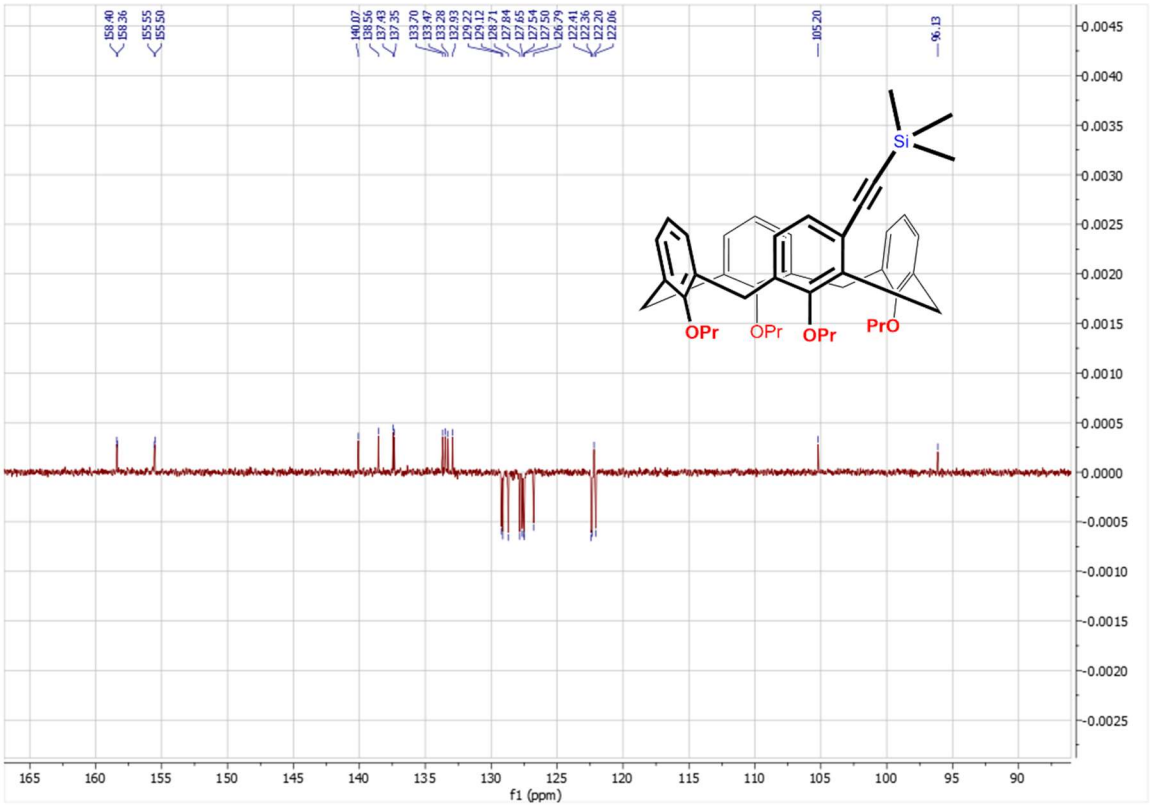


Figure S40: ^{13}C(APT) NMR of compound **5f,** aromatic region ($CDCl_3$, 100 MHz, 298 K)

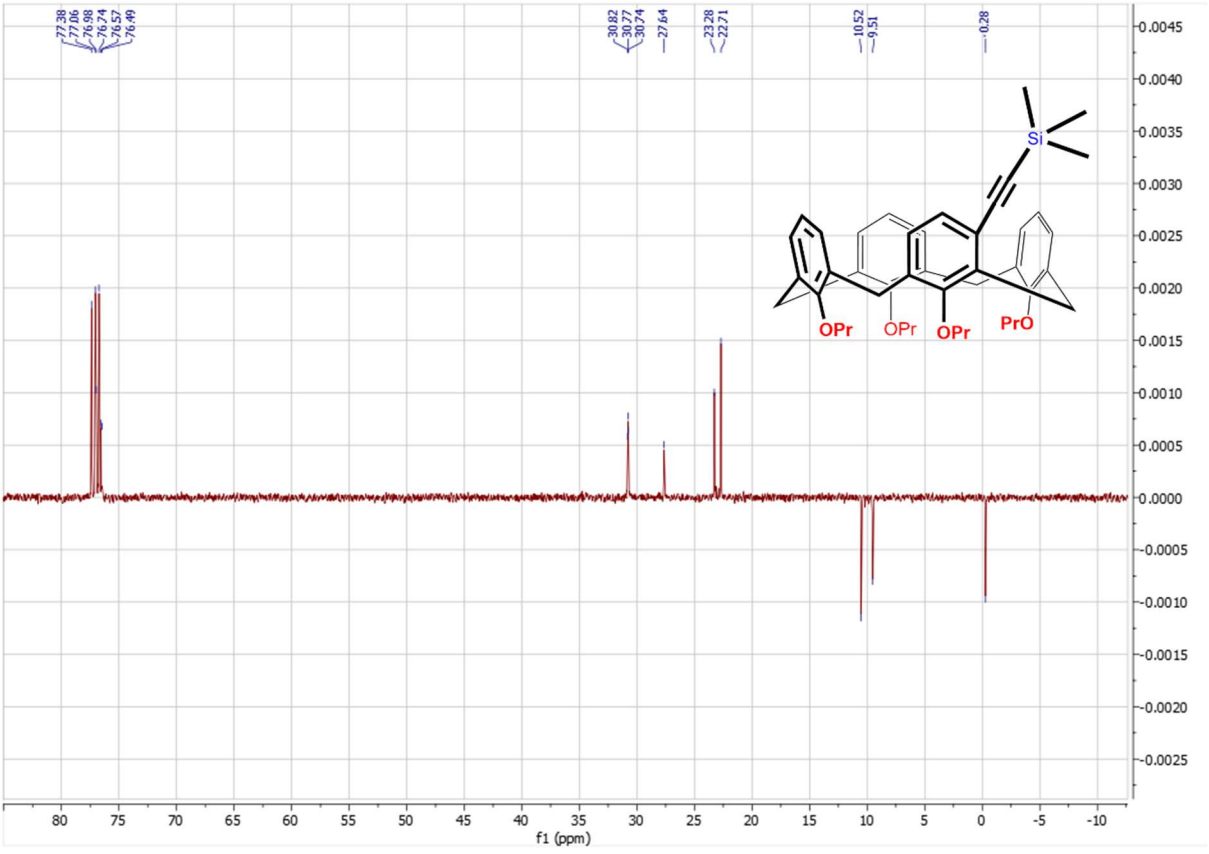


Figure S41: ^{13}C(APT) NMR of compound **5f,** aliphatic region ($CDCl_3$, 100 MHz, 298 K)

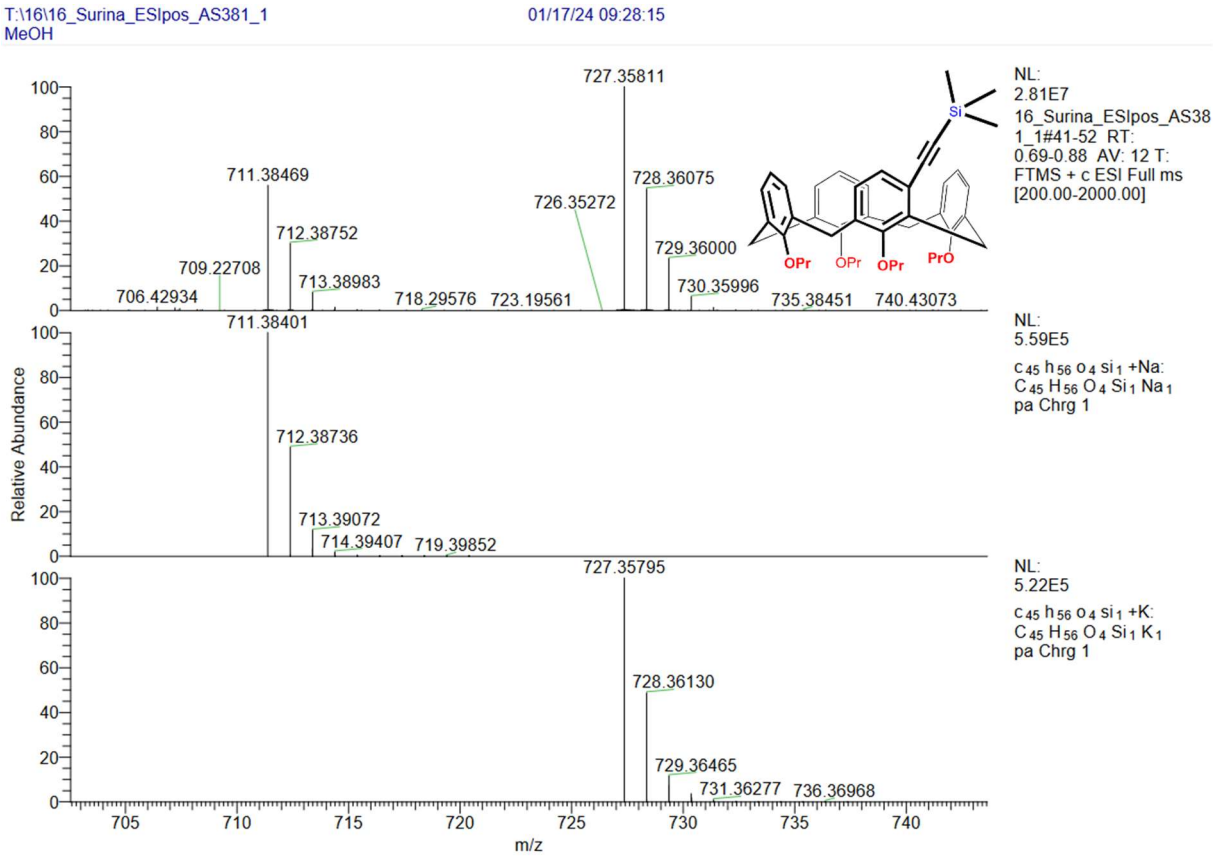


Figure S42: HRMS of compound **5f** (ESI^+)

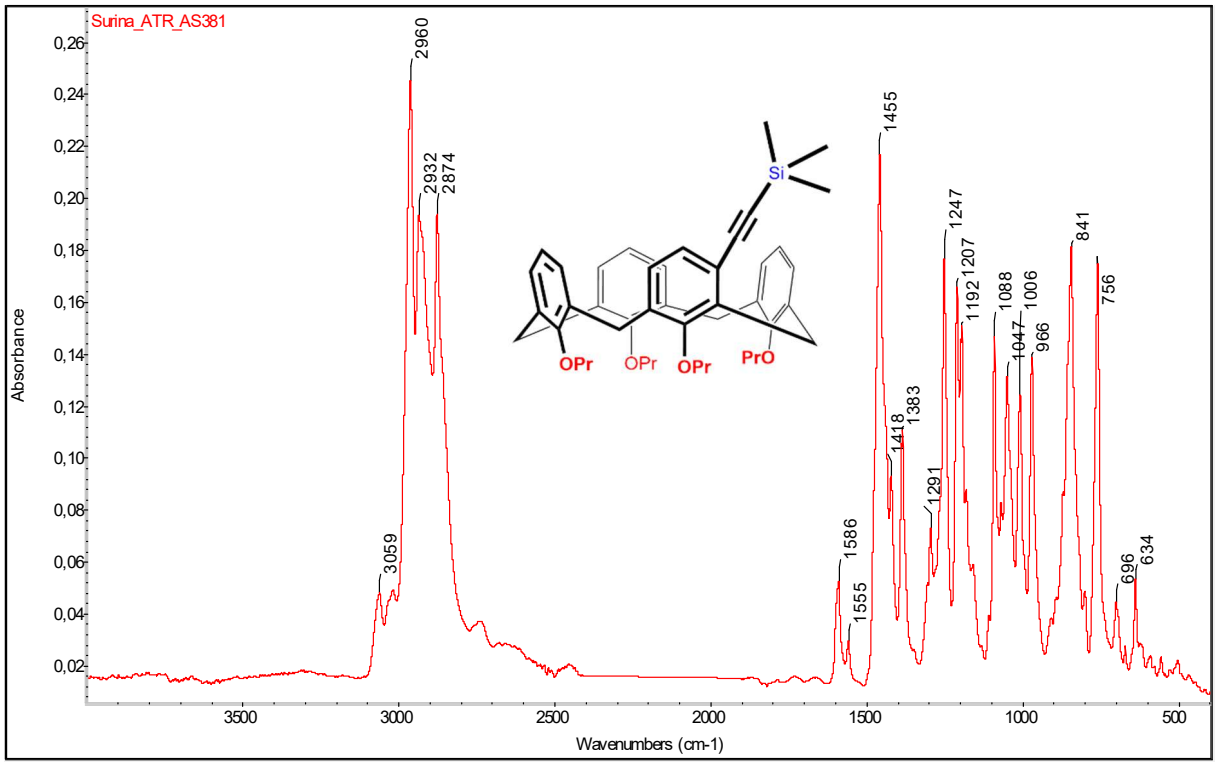


Figure S43: IR of compound **5f** (KBr)

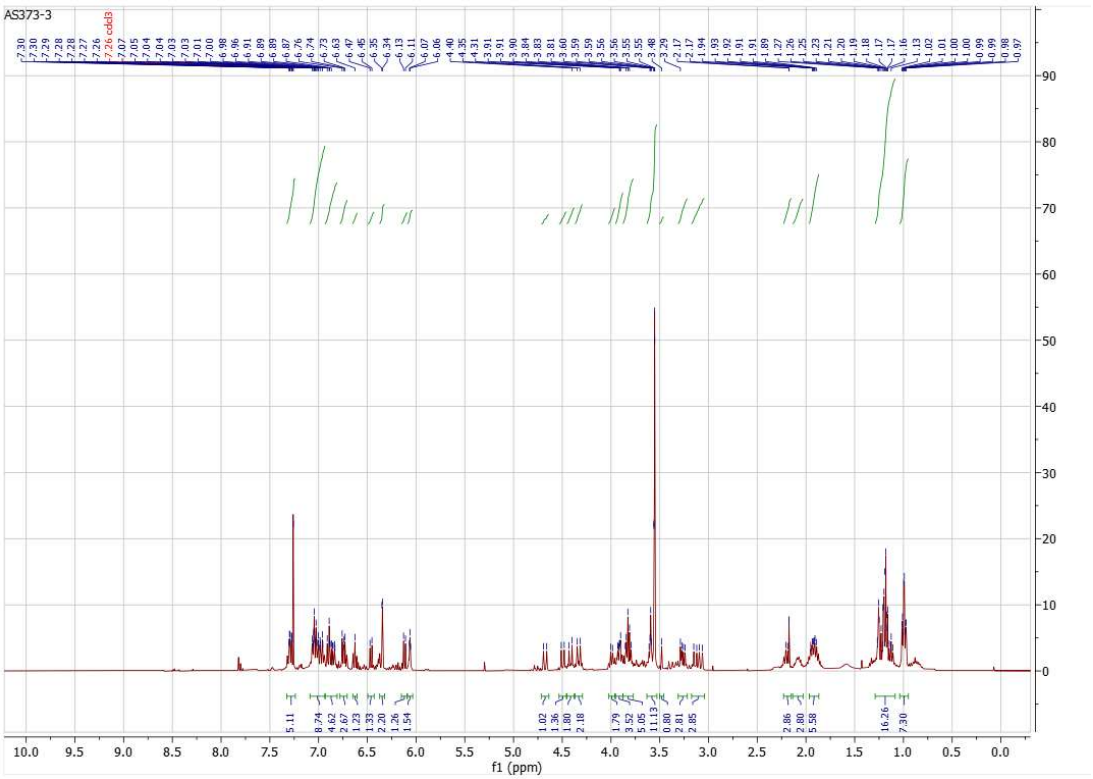


Figure S44: ^{1}H NMR of mixture **7a** with impurities. Integration cannot be used for determination of structure due to impurities. ($CDCl_3$, 400 MHz, 298 K)

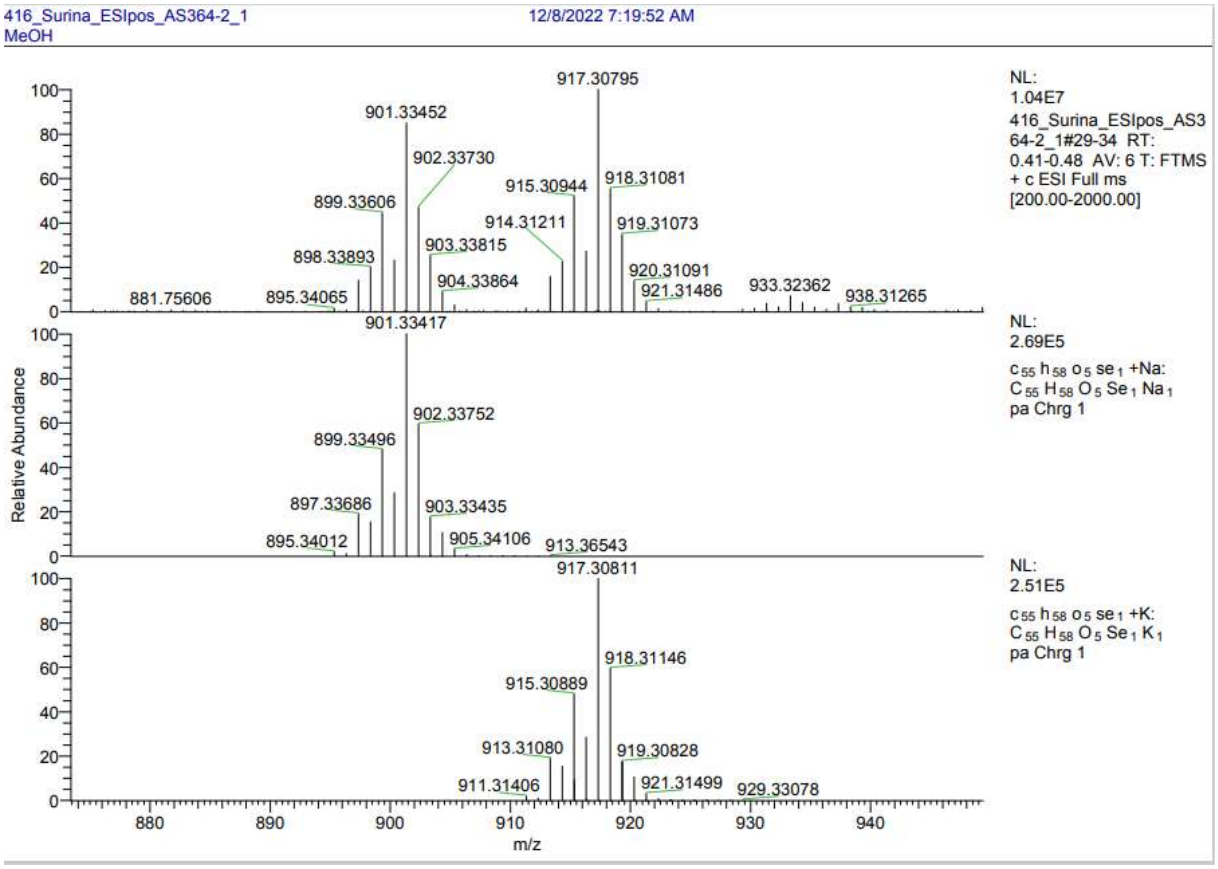


Figure S45: HRMS of compound **7a** (ESI^{+})

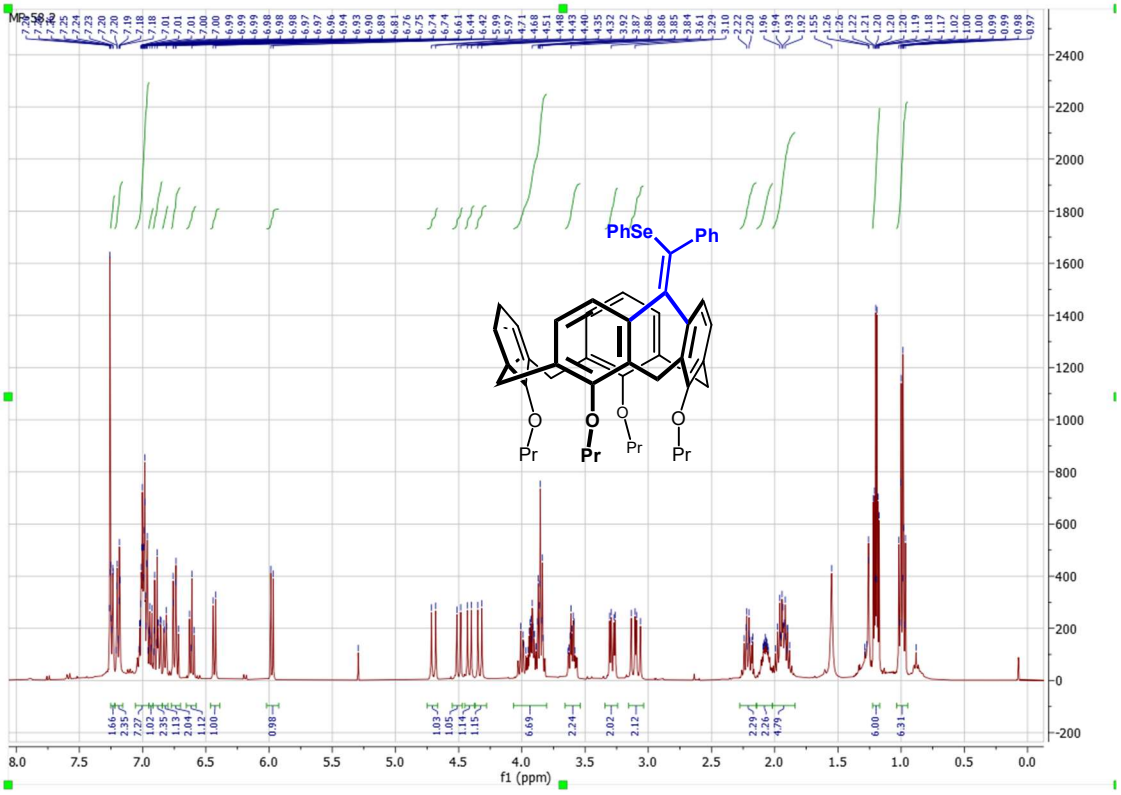


Figure S46: ^{1}H NMR of compound **7b** ($CDCl_3$, 400 MHz, 298 K)

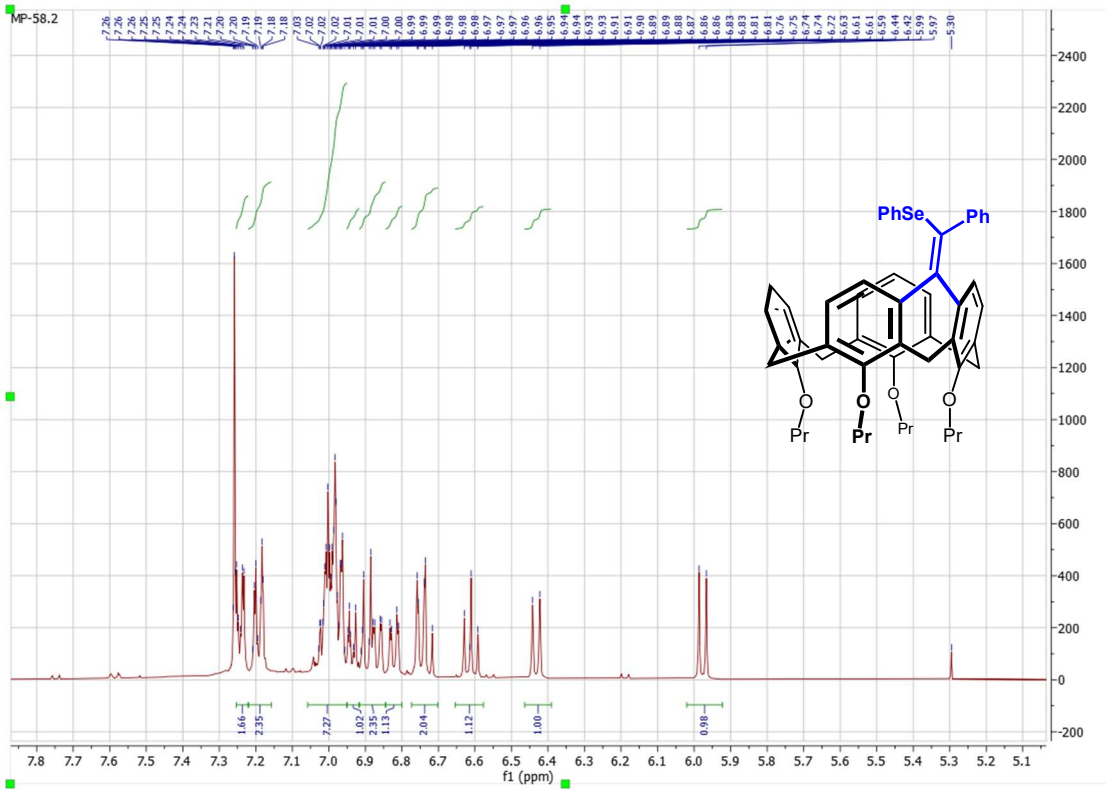


Figure S47: ^{1}H NMR of compound **7b,** aromatic region ($CDCl_3$, 400 MHz, 298 K)

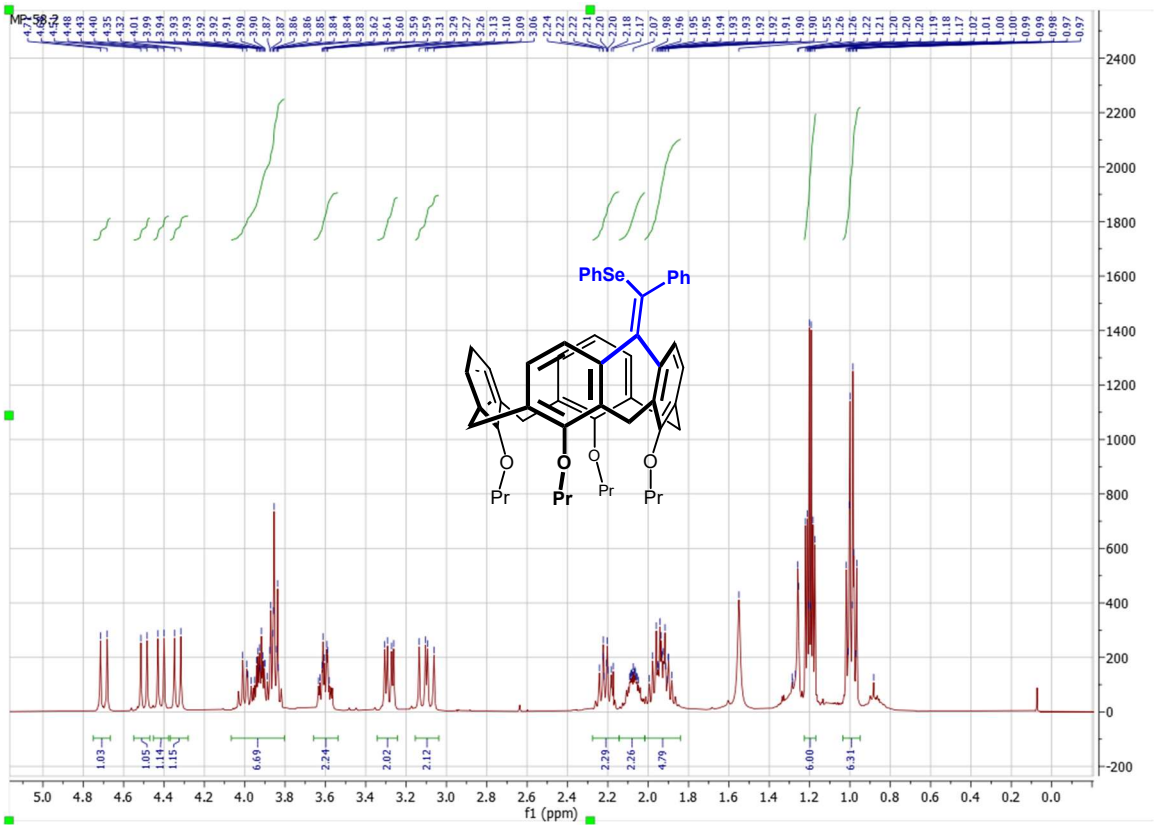


Figure S48: ^{1}H NMR of compound **7b,** aliphatic region ($CDCl_3$, 400 MHz, 298 K)

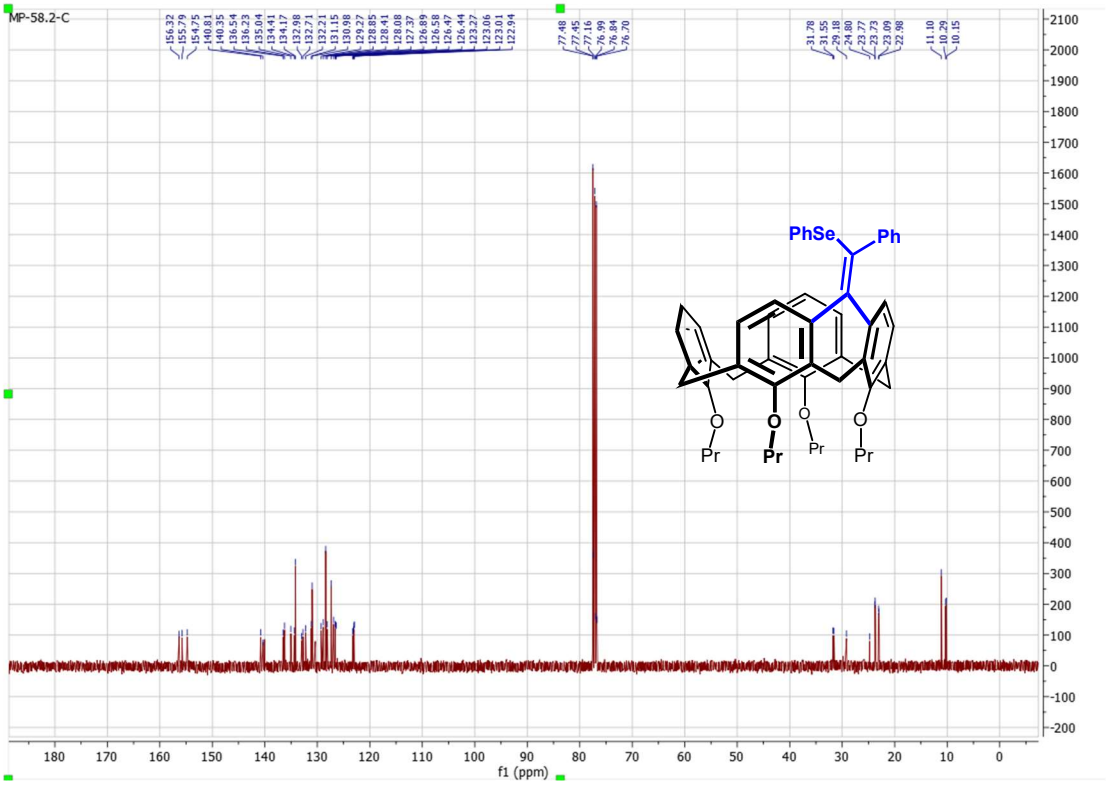


Figure S49: ^{13}C NMR of compound **7b** ($CDCl_3$, 100 MHz, 298 K)

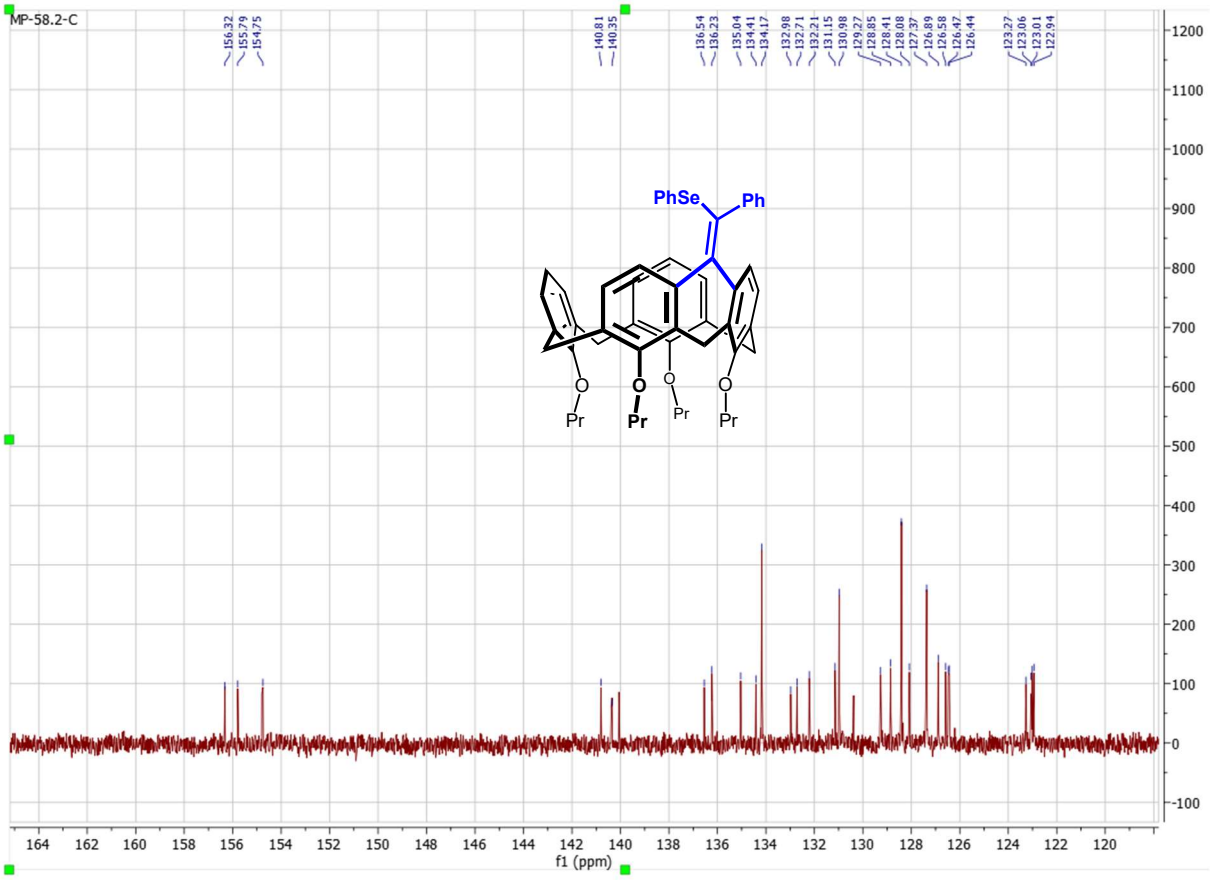


Figure S50: ^{13}C NMR of compound **7b,** aromatic region ($CDCl_3$, 100 MHz, 298 K)

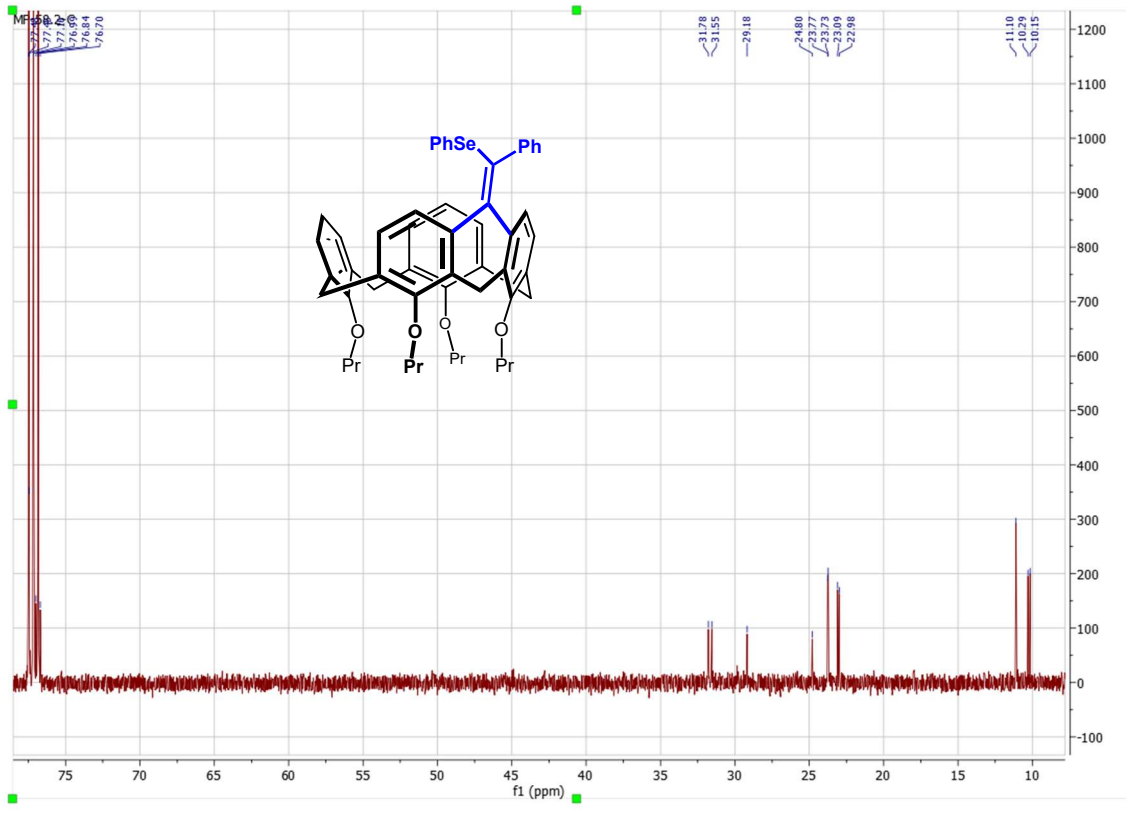


Figure S51: ^{13}C NMR of compound **7b,** aliphatic region ($CDCl_3$, 100 MHz, 298 K)

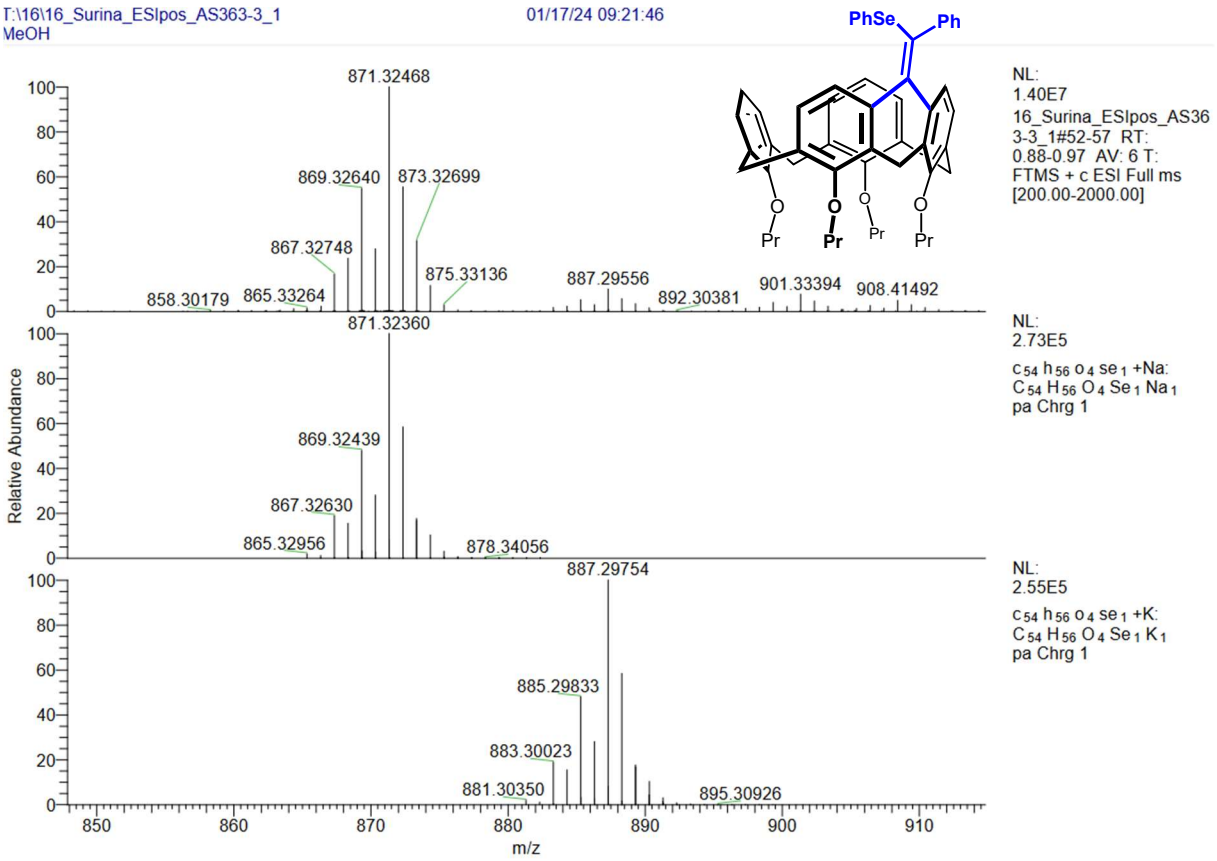


Figure S52: HRMS of compound **7b** (ESI^+)

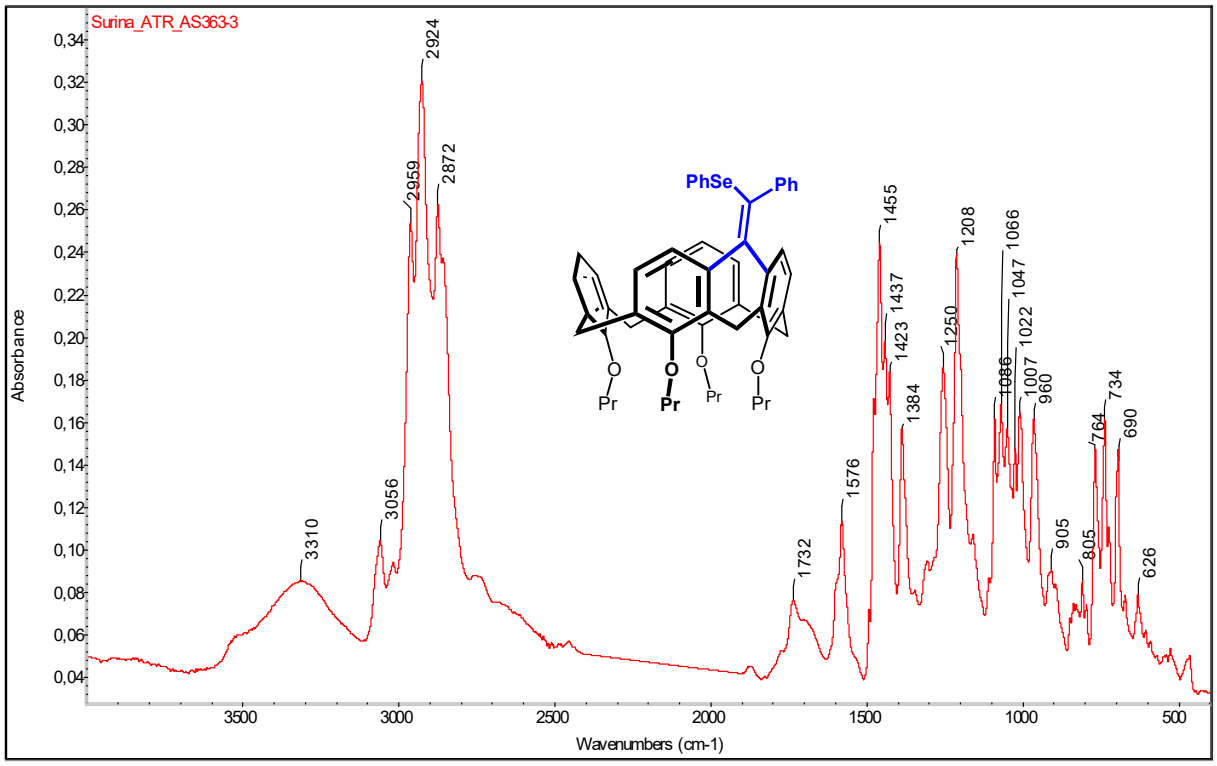


Figure S53: IR of compound **7b** (KBr)

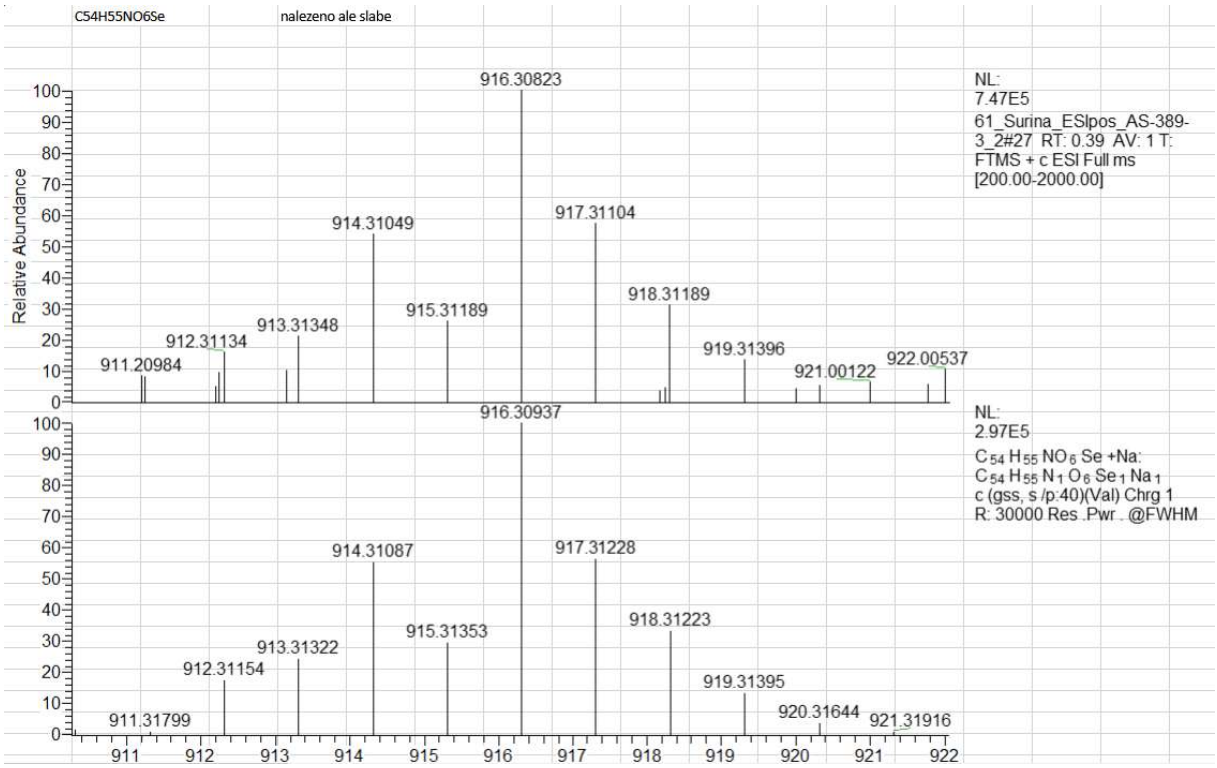


Figure S54: HRMS of compound **7d** (ESI^+)

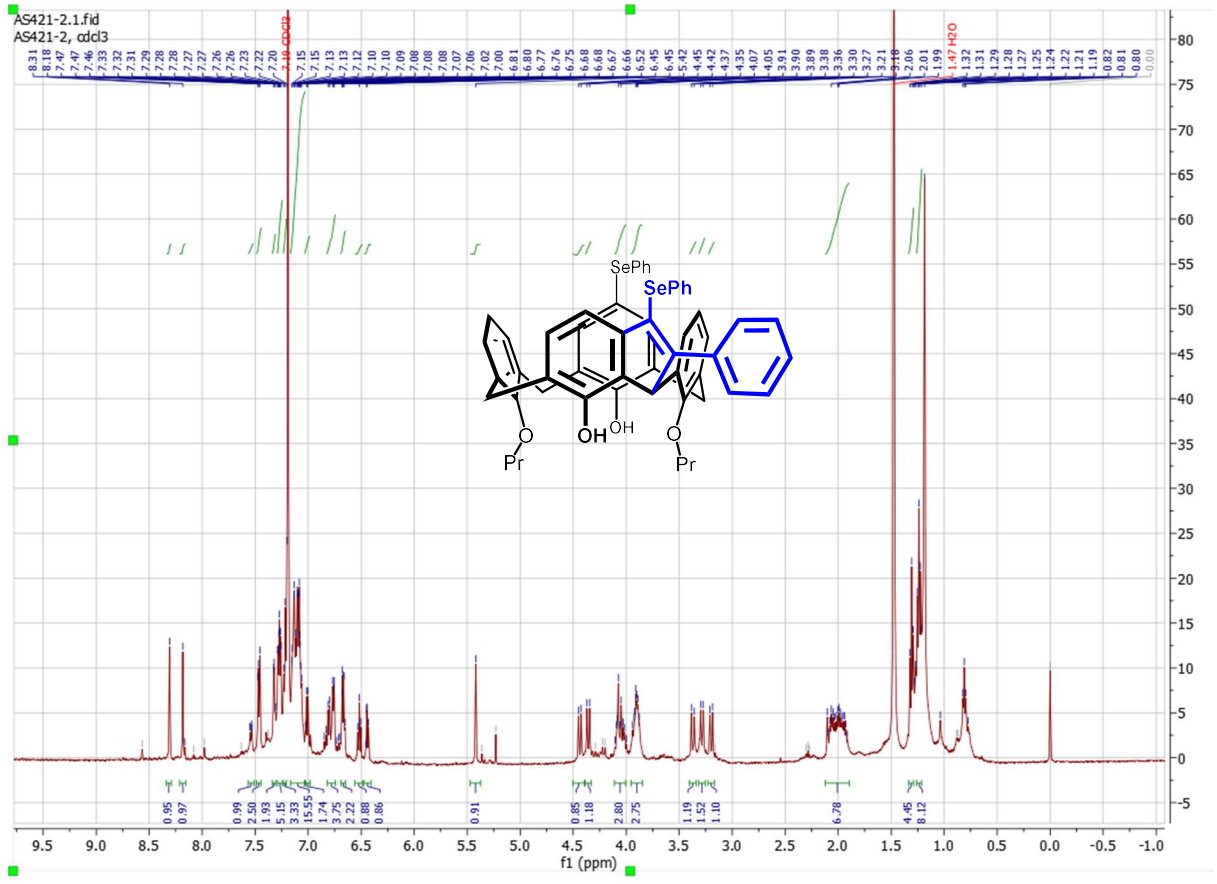


Figure S55: ^1H NMR of compound **8b** ($CDCl_3$, 500 MHz, 298 K)

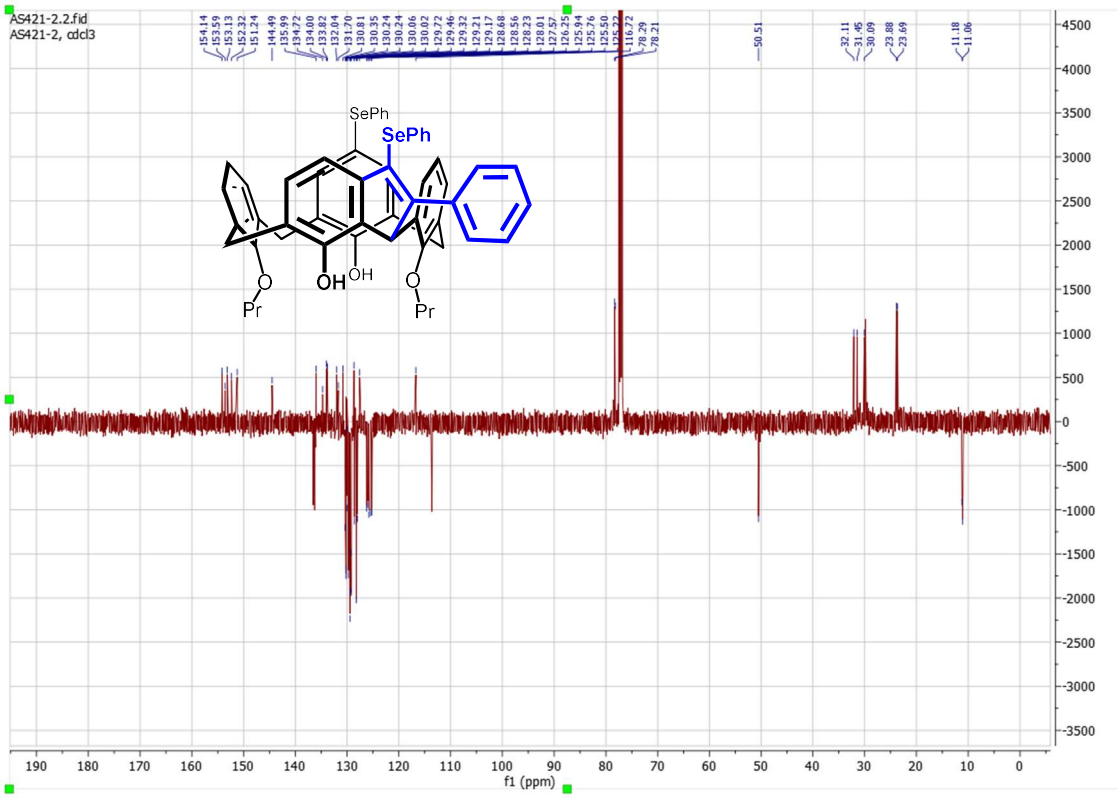


Figure S56: ^{13}C(APT) NMR of compound **8b** ($CDCl_3$, 125 MHz, 298 K)

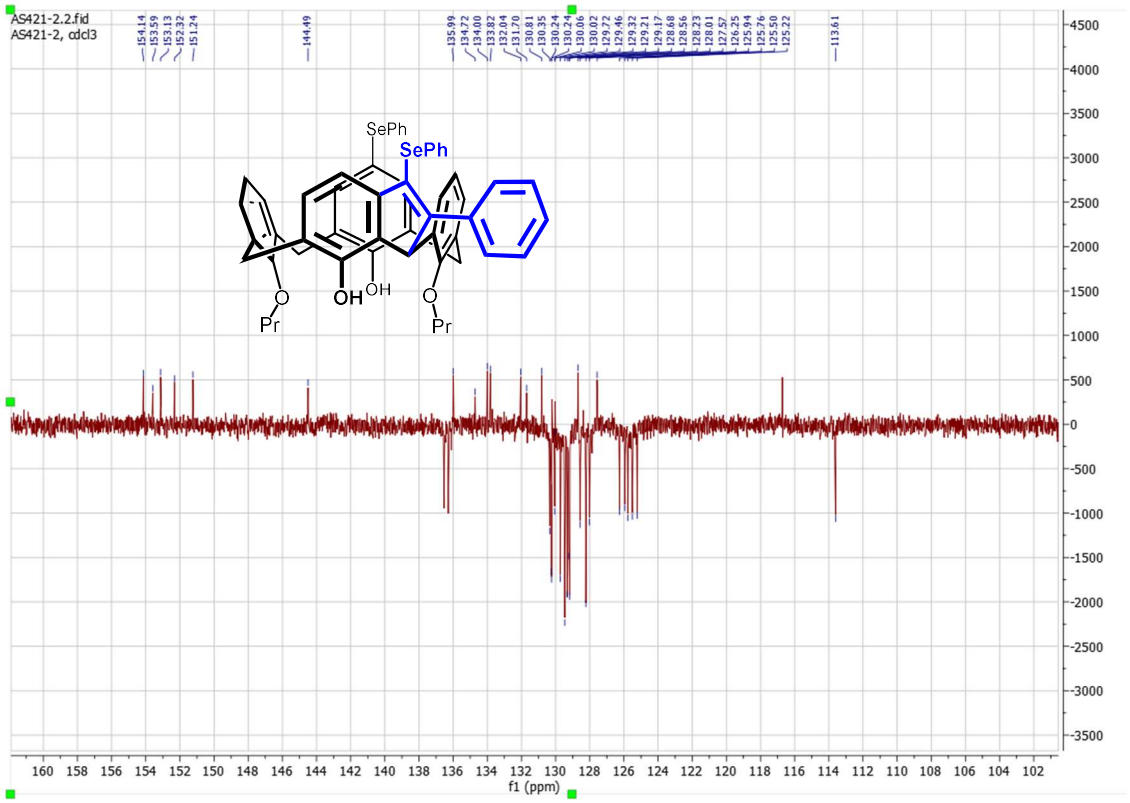


Figure S57: ^{13}C(APT) NMR of compound **8b,** aromatic region ($CDCl_3$, 125 MHz, 298 K)

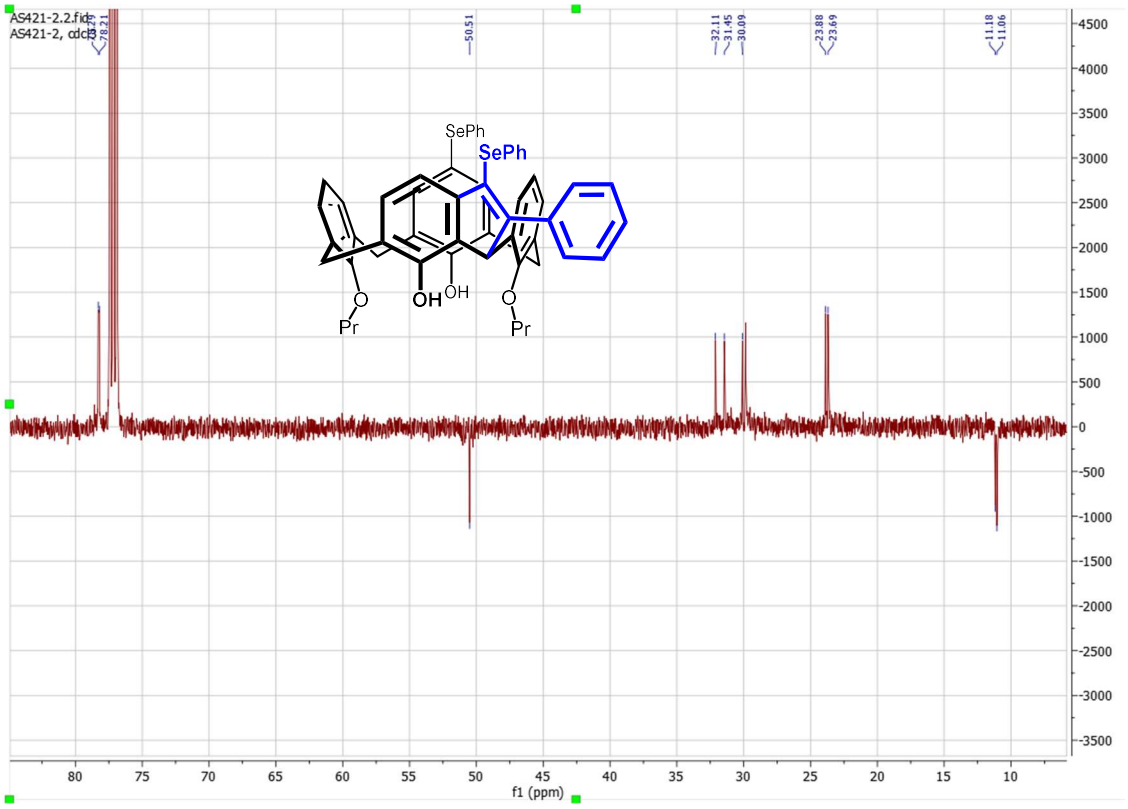


Figure S58: ^{13}C(APT) NMR of compound **8b,** aliphatic region ($CDCl_3$, 125 MHz, 298 K)

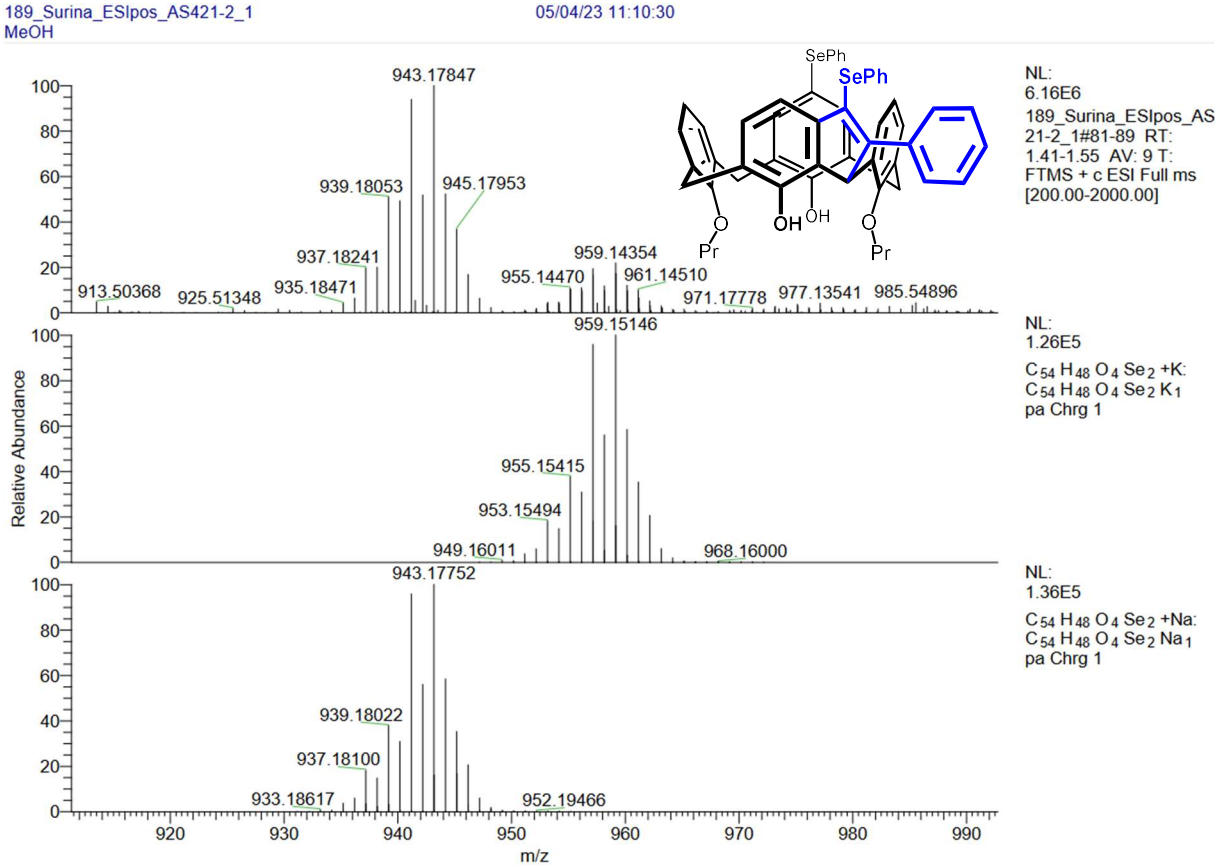


Figure S59: HRMS of compound **8b** (ESI^+)

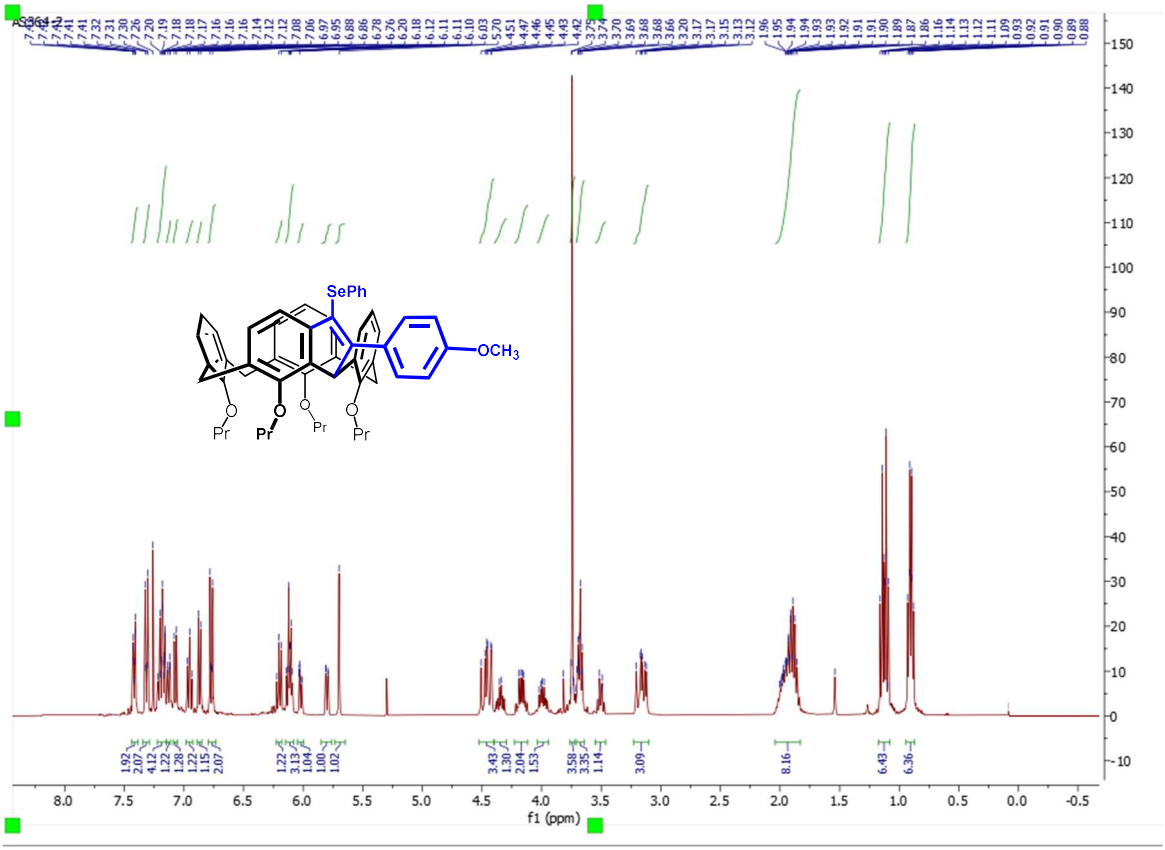


Figure S60: ^{1}H NMR of compound **9c** ($CDCl_3$, 400 MHz, 298 K)

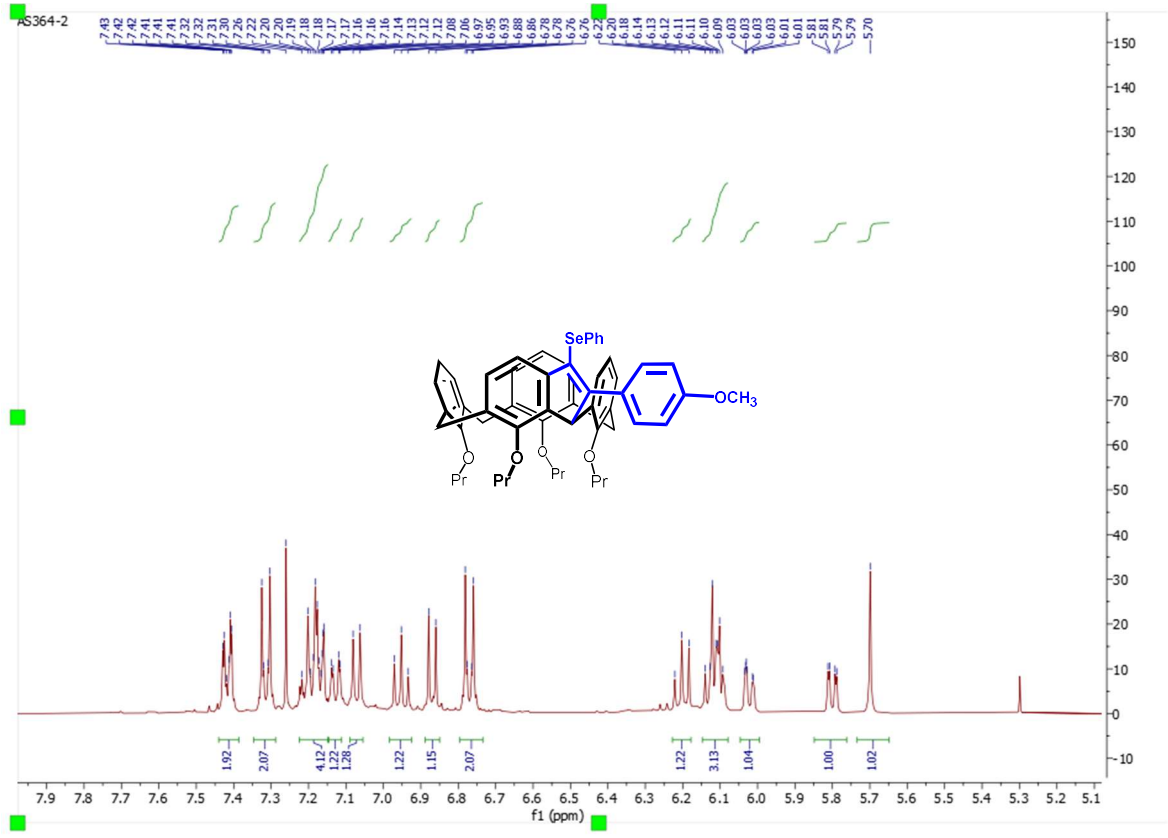


Figure S61: ^{1}H NMR of compound **9c,** aromatic region ($CDCl_3$, 400 MHz, 298 K)

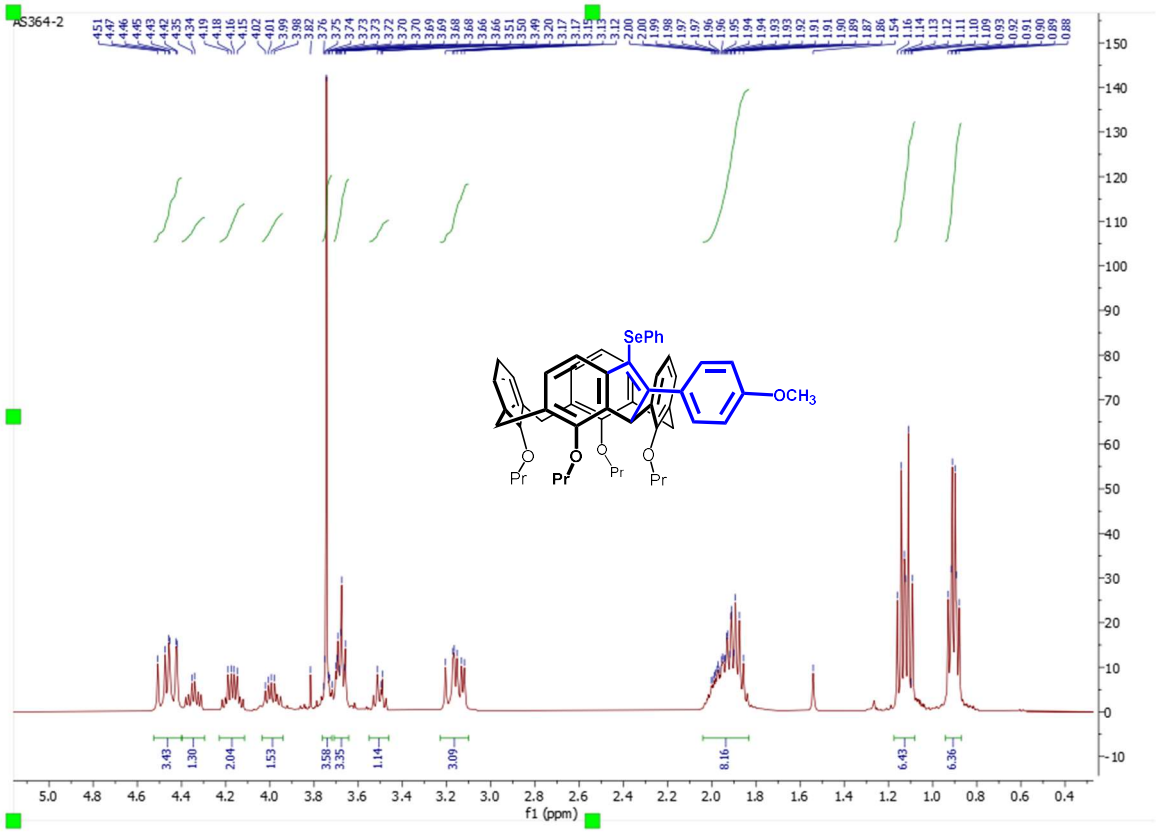


Figure S62: ^{1}H NMR of compound **9c,** aliphatic region ($CDCl_3$, 400 MHz, 298 K)

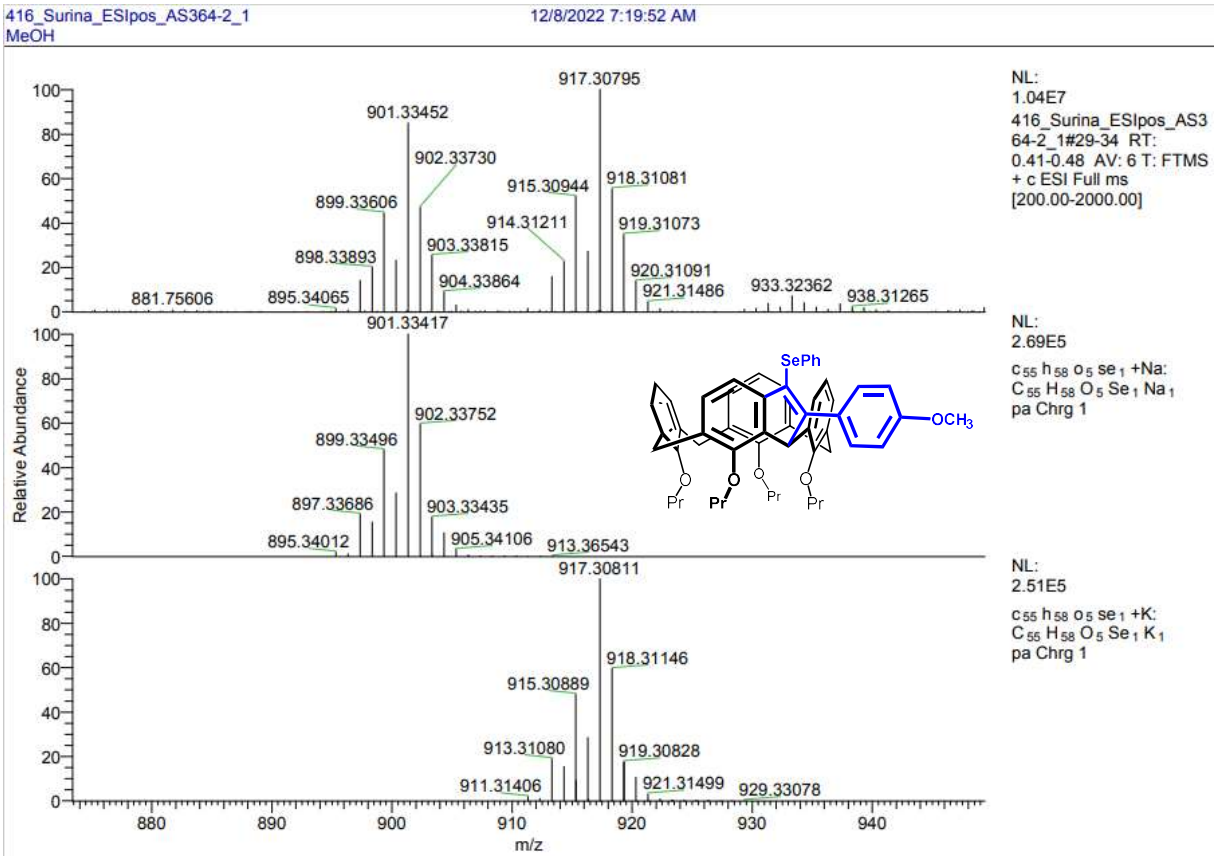


Figure S63: HRMS of compound **9c** (ESI^+)

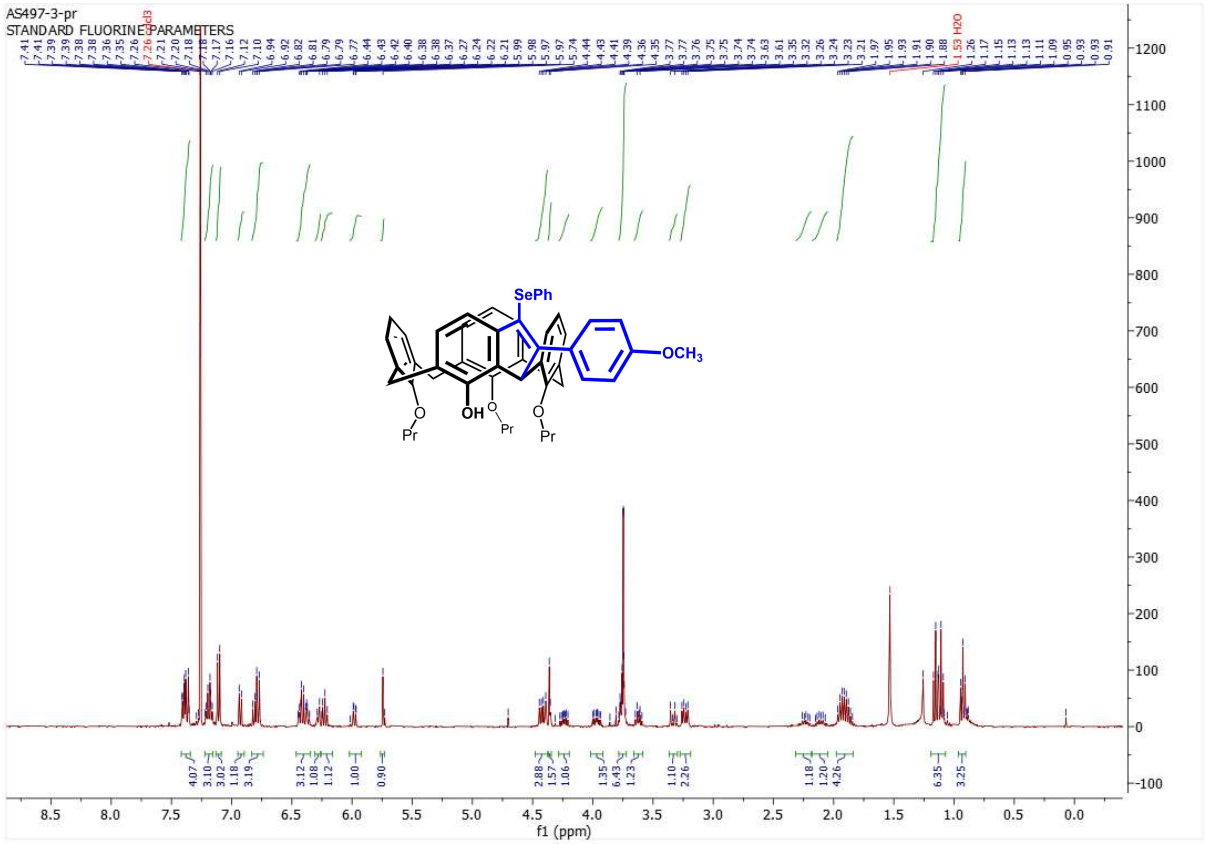


Figure S64: ^{1}H NMR of compound **10c** ($CDCl_3$, 400 MHz, 298 K)

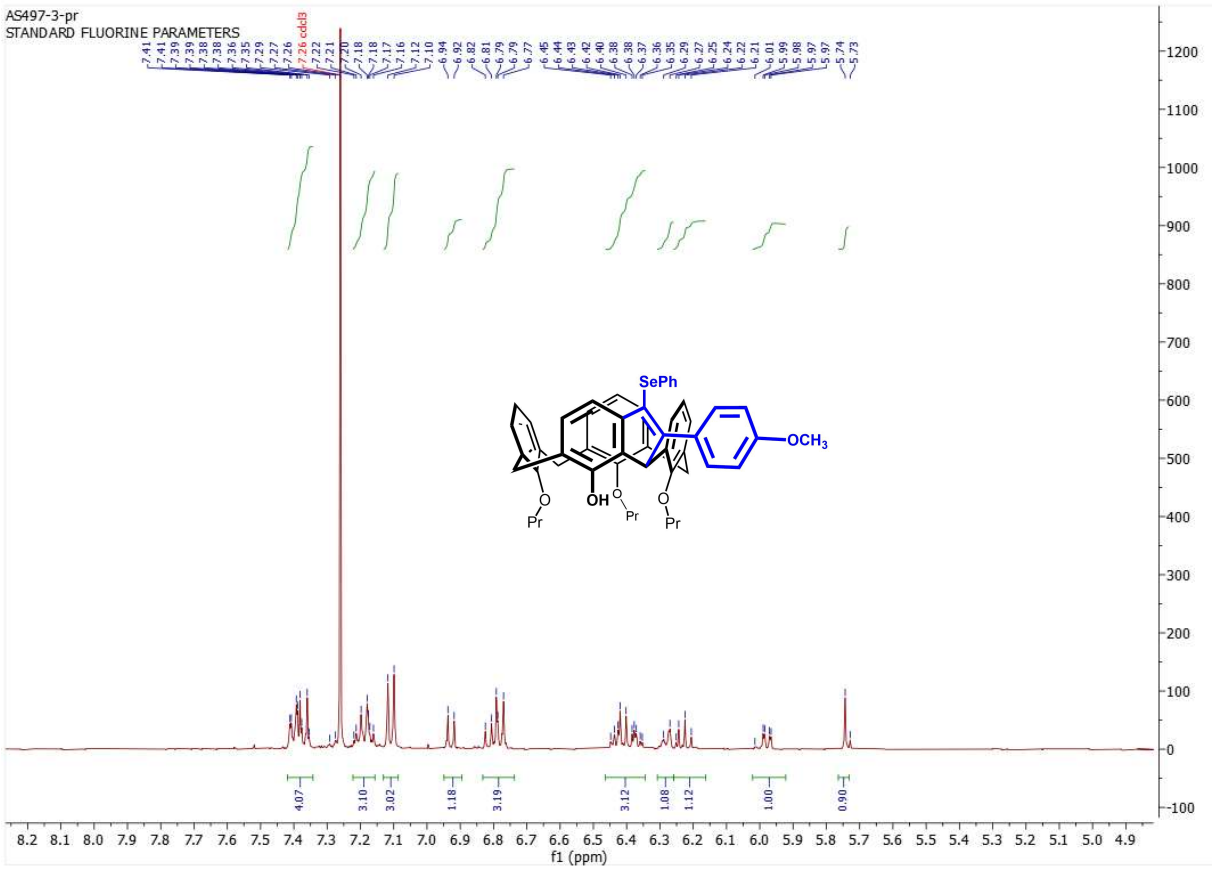


Figure S65: ^{1}H NMR of compound **10c,** aromatic region ($CDCl_3$, 400 MHz, 298 K)

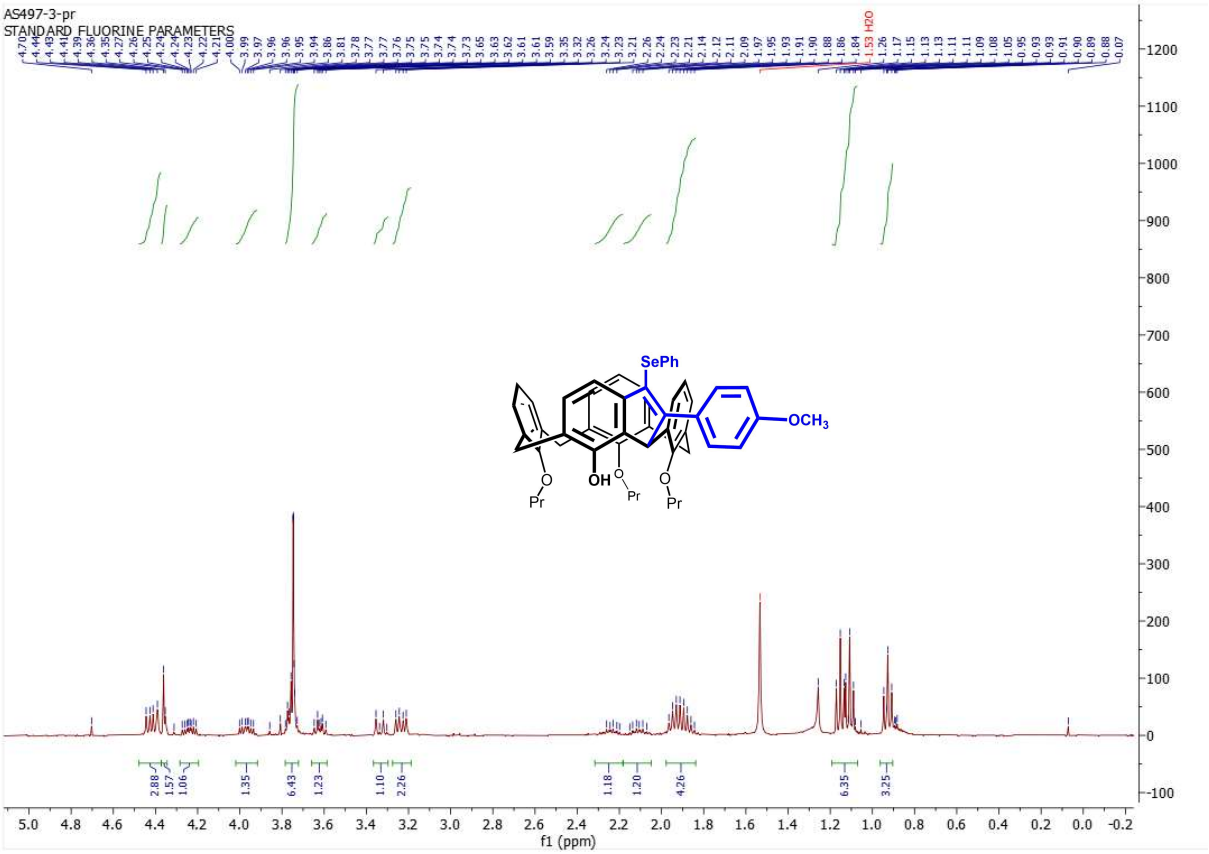


Figure S66: ^{1}H NMR of compound **10c,** aliphatic region ($CDCl_3$, 400 MHz, 298 K)

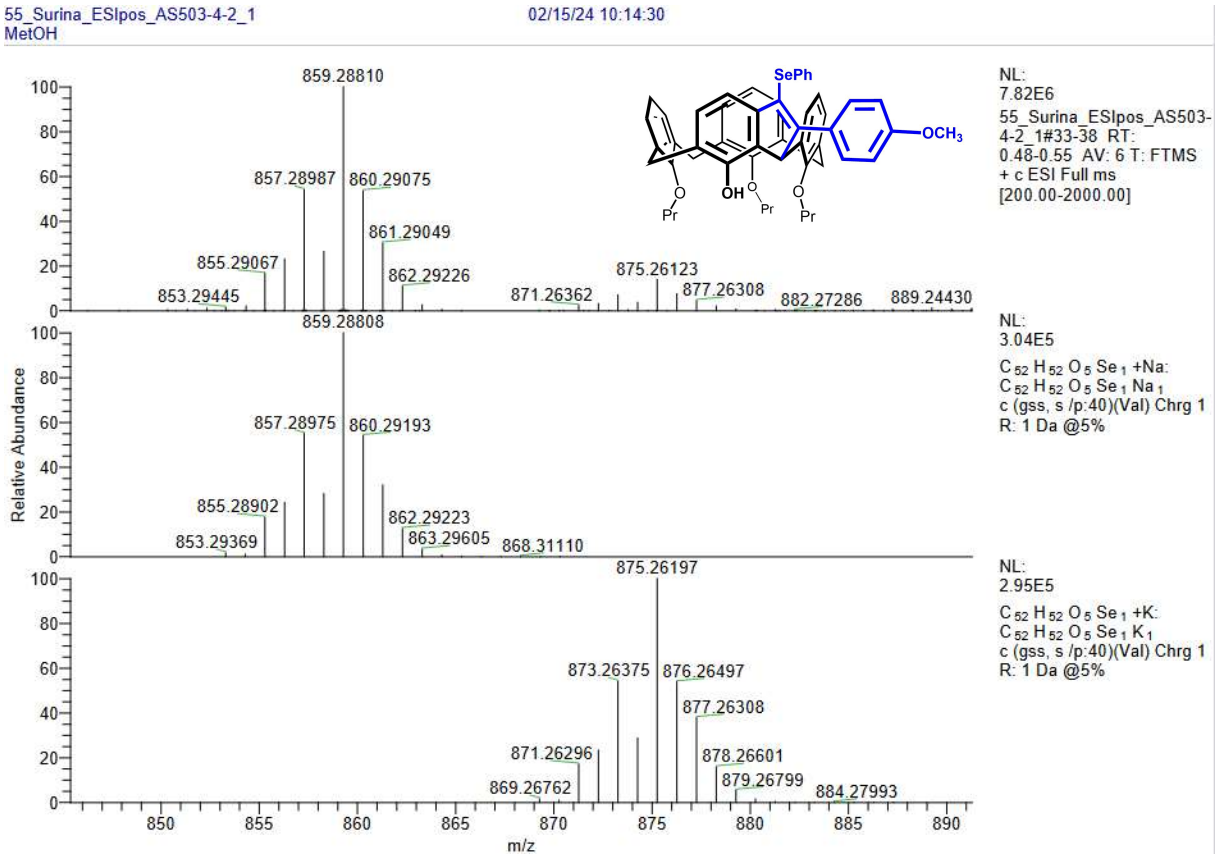


Figure S67: HRMS of compound **10c** (ESI^+)

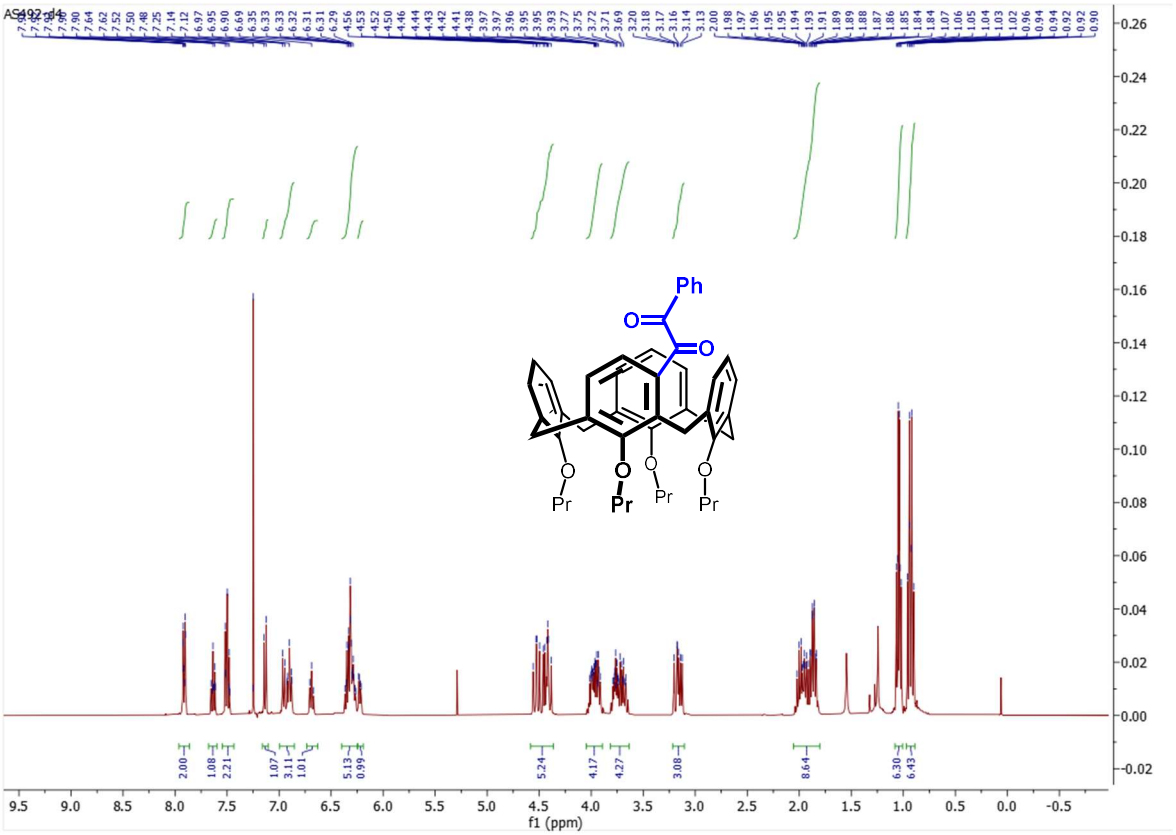


Figure S68: ^{1}H NMR of compound **11** ($CDCl_3$, 400 MHz, 298 K)

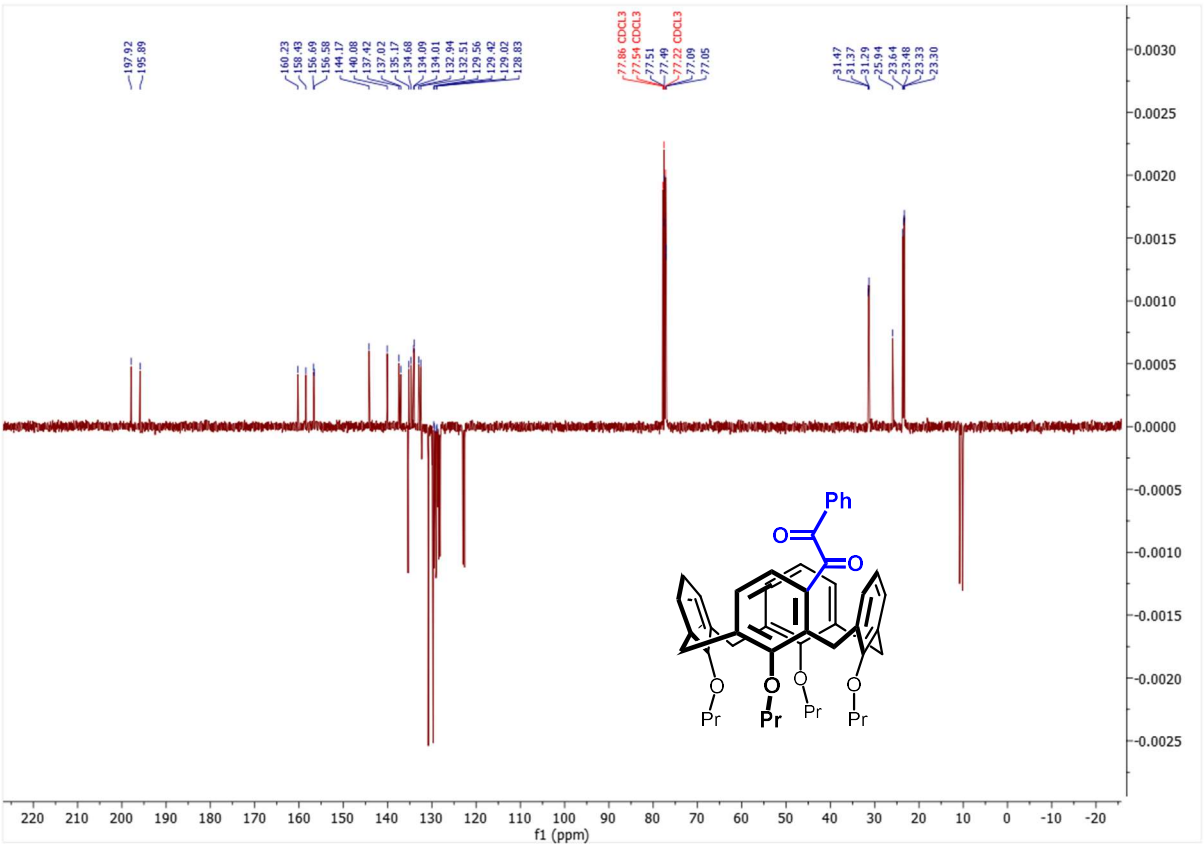


Figure S69: ^{13}C(APT) NMR of compound **11** ($CDCl_3$, 100 MHz, 298 K)

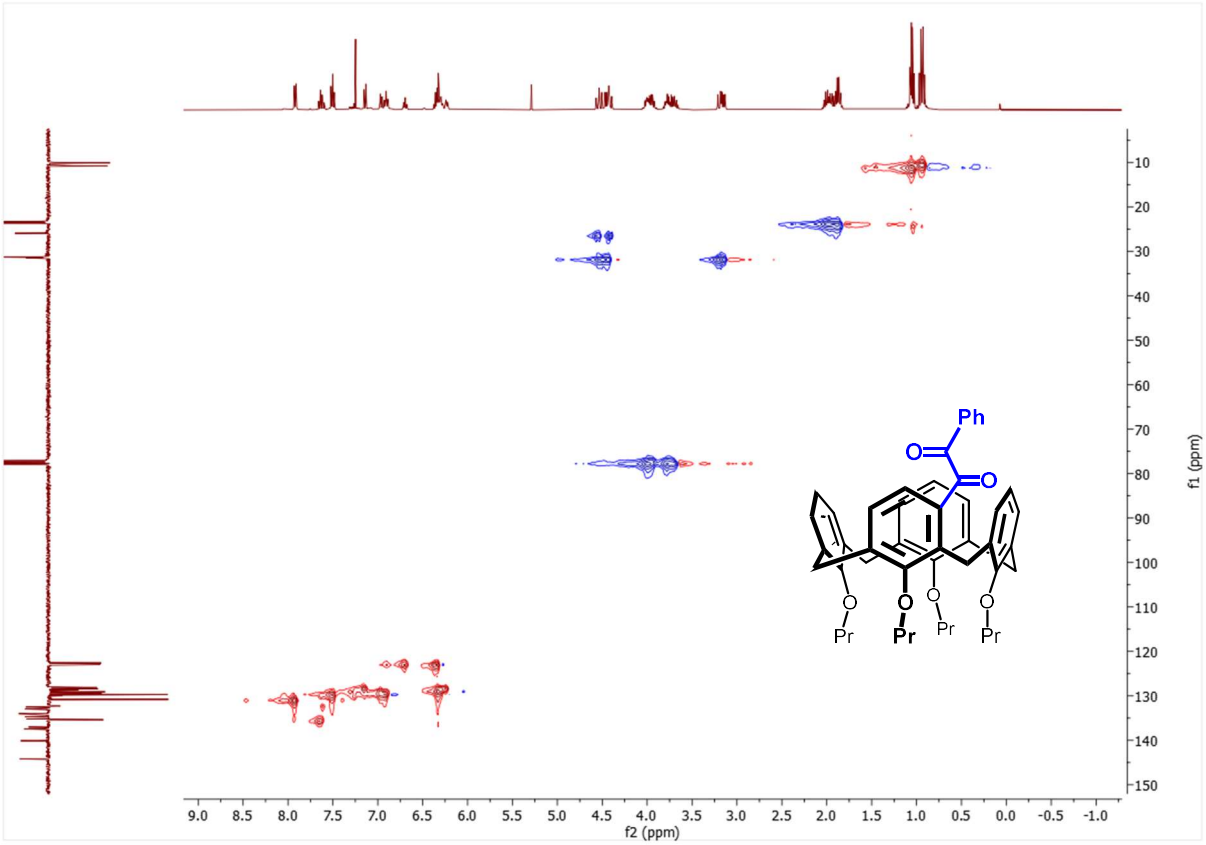


Figure S70: HSQC NMR of compound **11** ($CDCl_3$, 100 MHz, 298 K)

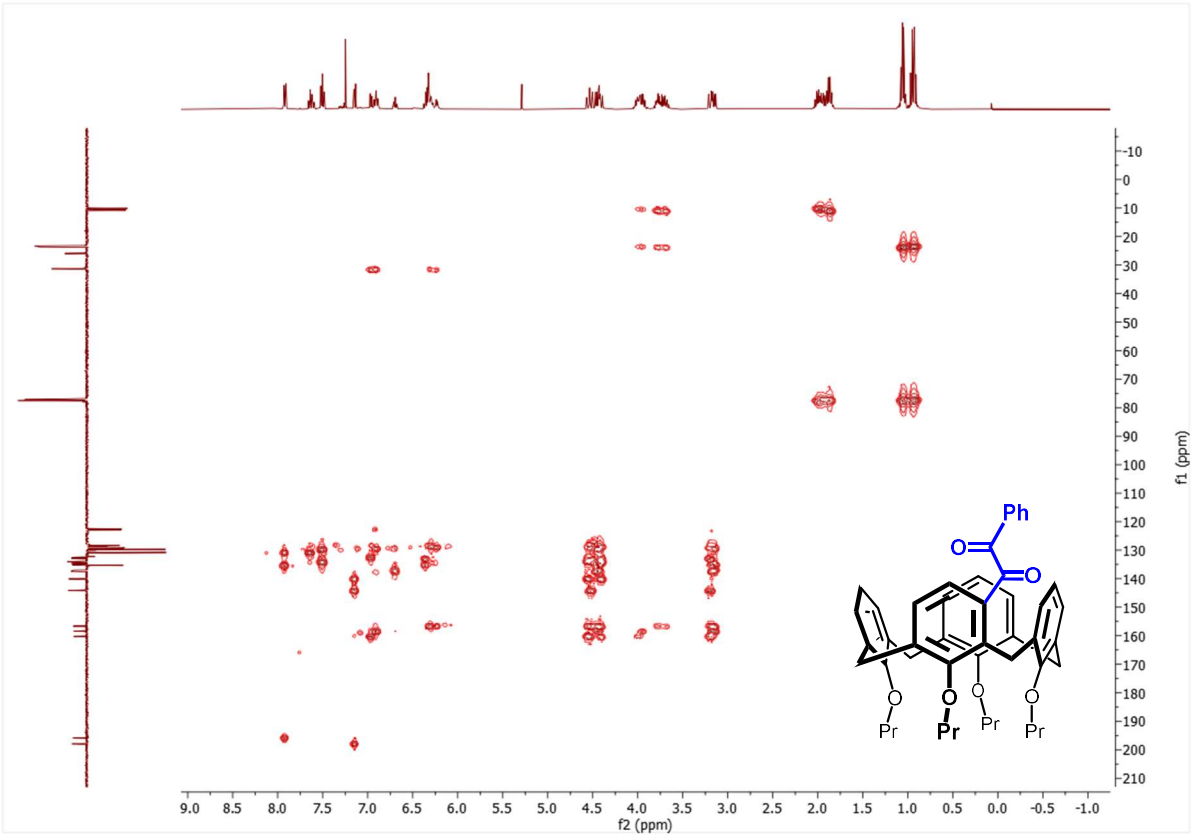


Figure S71: HMBC NMR of compound **11** ($CDCl_3$, 100 MHz, 298 K)

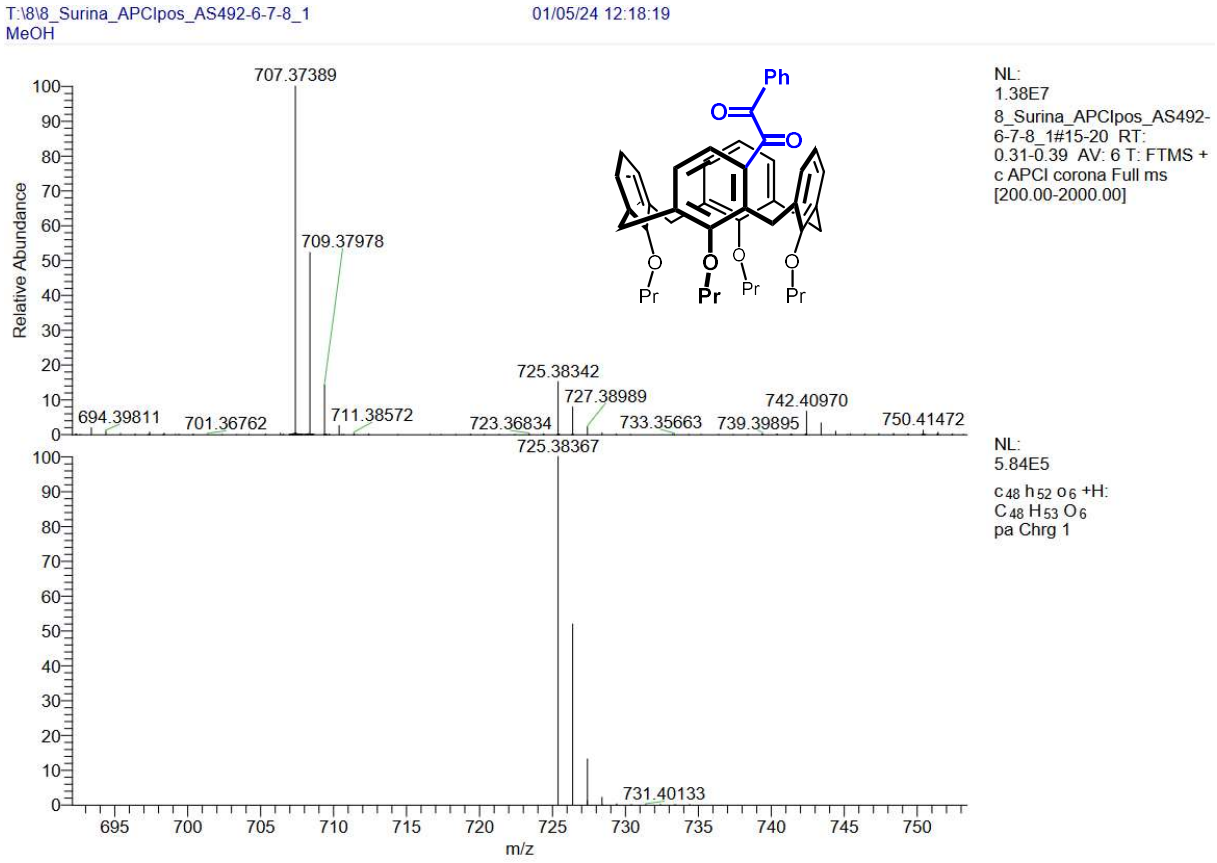


Figure S72: HRMS of compound **11** + H^+ (ESI^+)

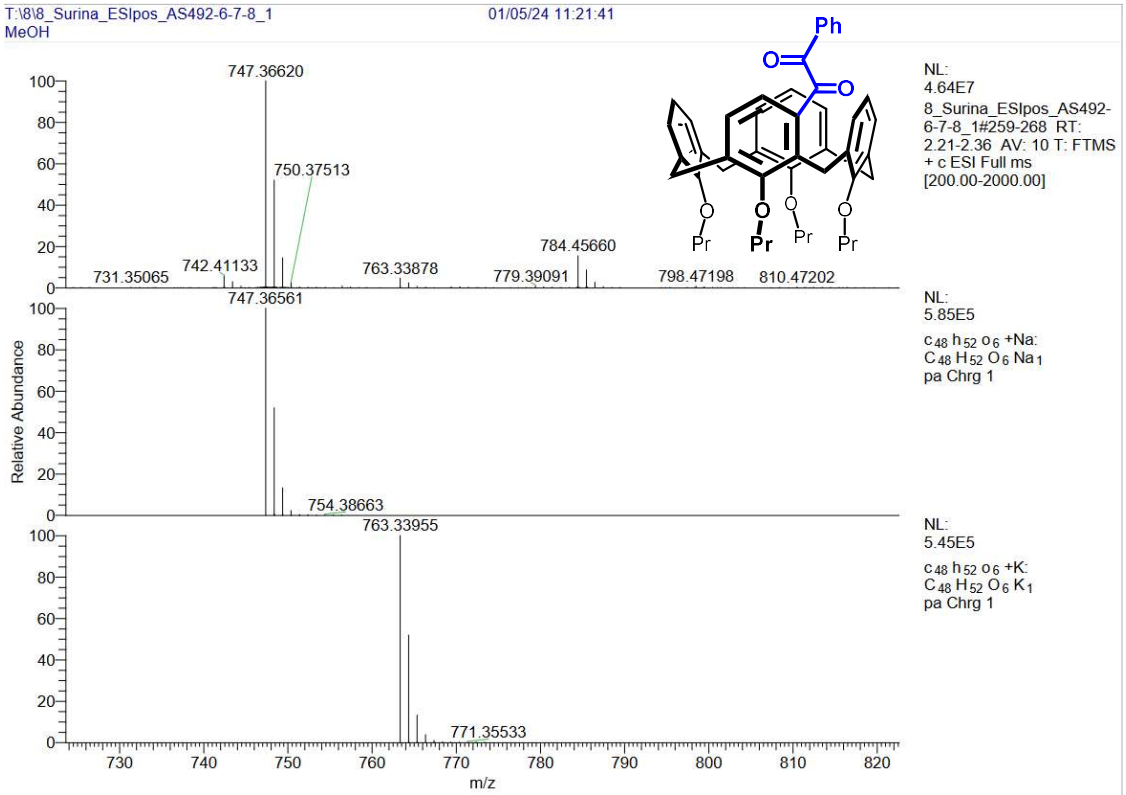


Figure S73: HRMS of compound **11** (ESI^+)

2. Crystallographic data

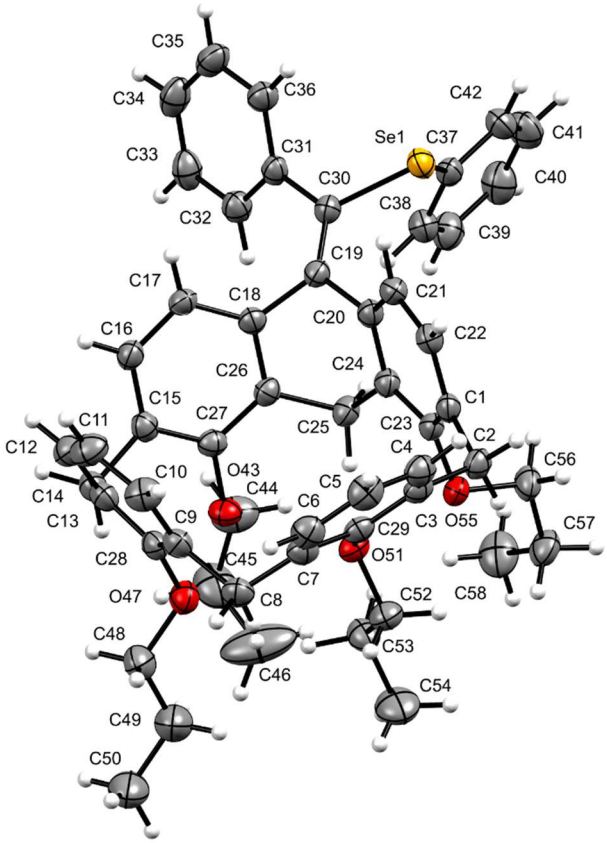


Figure S74: Numbering scheme of crystal structure of **7b**, the ORTEP diagram with 50% probability.

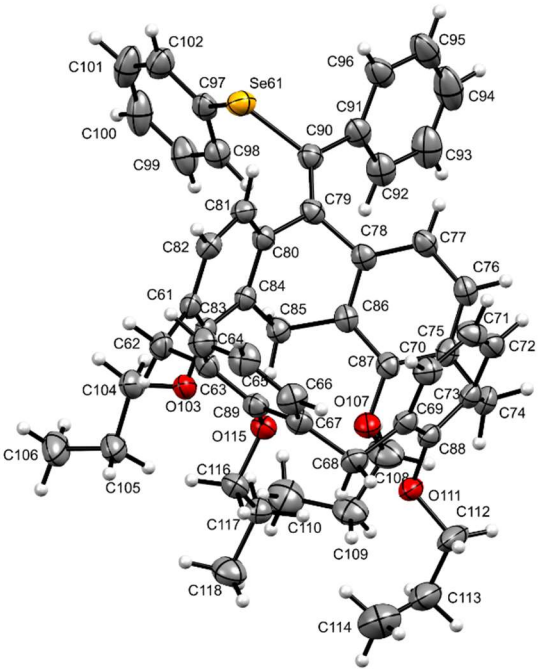
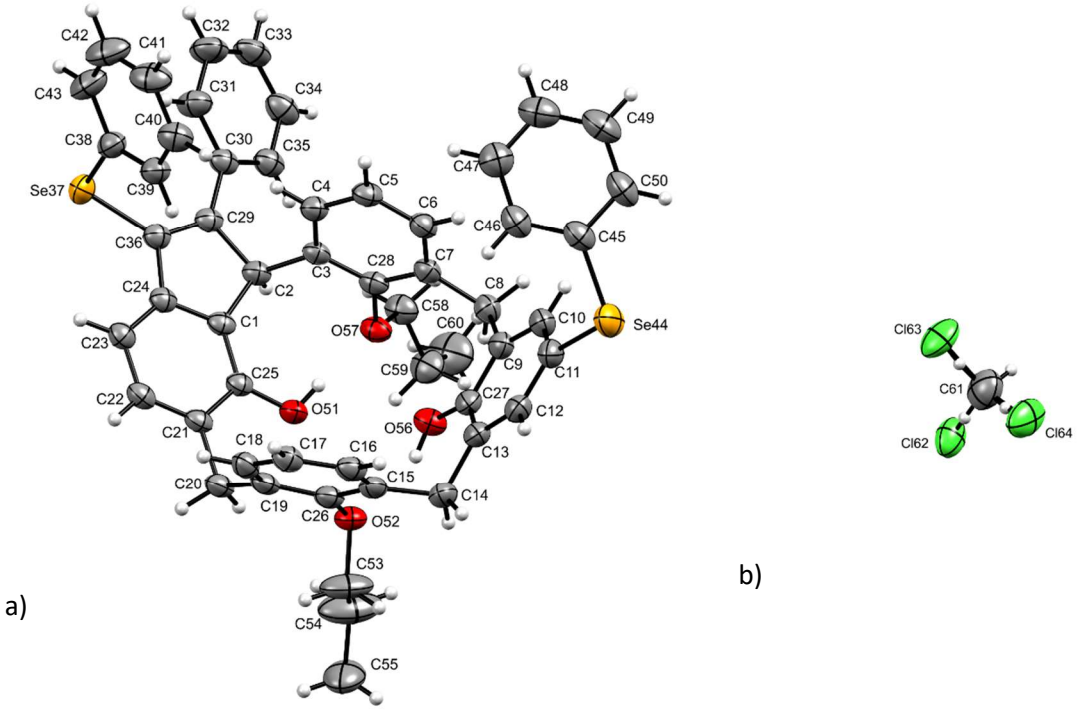


Figure S75: Numbering scheme of crystal structure of **7b** (different view), the ORTEP diagram with 50% probability.



a) b)

Figure S76: Numbering scheme of crystal structure of a) **8b** and b) solvate molecule, the ORTEP diagram with 50% probability.

3. Electrochemistry

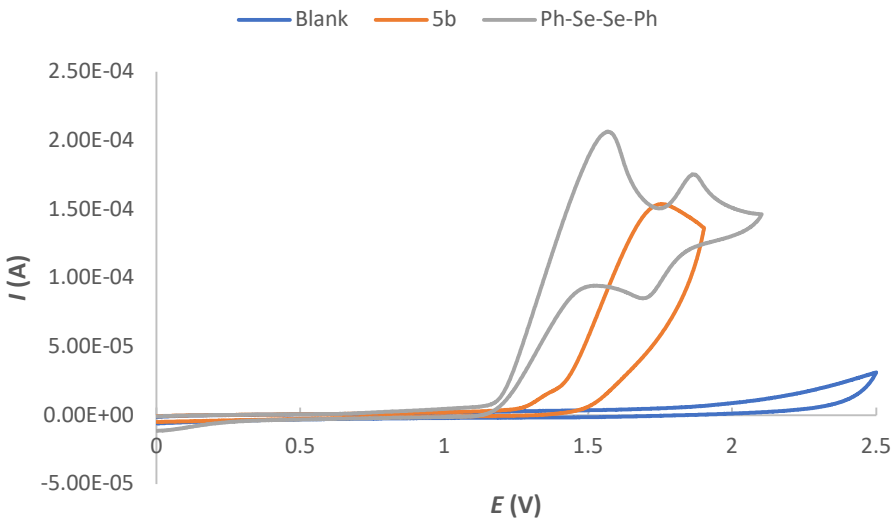


Figure S77: a) blue Cyclic voltammogram (W = GC, Ref = SCE, Aux = Pt) of blank solution (MeCN, 0.02 M $LiClO_4$) with scan rate 100 mV/s (IUPAC plotting convention). **b) orange** Cyclic voltammogram (W = GC, Ref = SCE, Aux = Pt) of compound **5b** (4.14 mM) in MeCN (0.02 M $LiClO_4$) with scan rate 100 mV/s (IUPAC plotting convention). **c) grey** Cyclic voltammogram (W = GC, Ref = SCE, Aux = Pt) of diphenyl diselenide (2.12 mM) in MeCN (0.02 M $LiClO_4$) with scan rate 100 mV/s (IUPAC plotting convention).

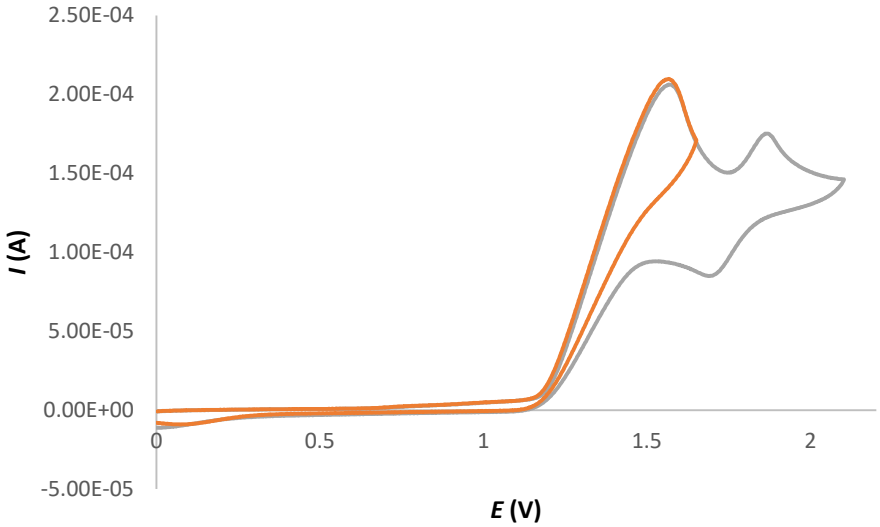


Fig. S78: Cyclic voltammograms (W = GC, Ref = SCE, Aux = Pt) of diphenyl diselenide (2.12 mM) in MeCN (0.02 M $LiClO_4$) with scan rate 100 mV/s (IUPAC plotting convention).

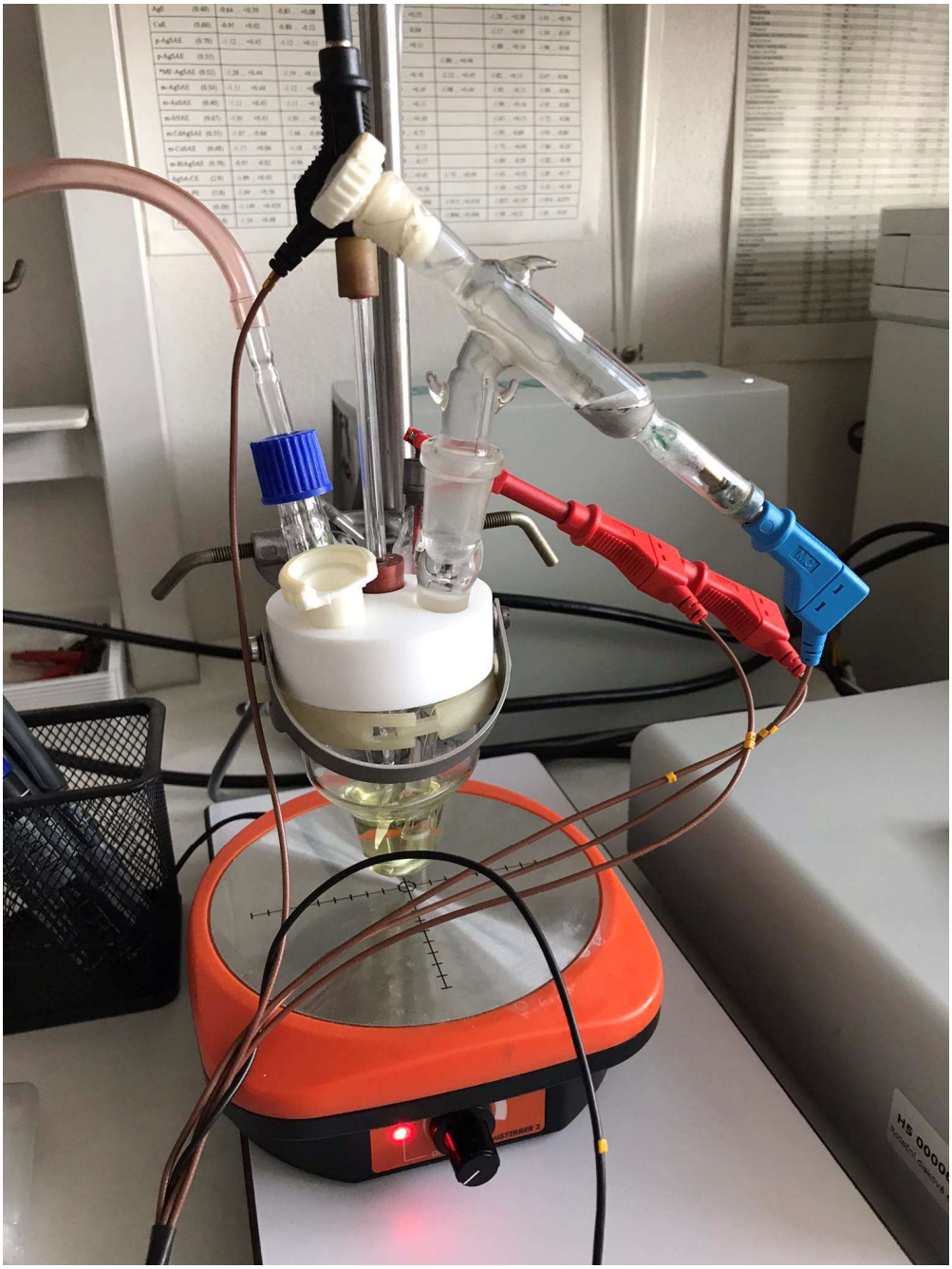


Figure S79: Illustrative picture for batch electrolysis of **5b** in undivided cell equipped with Pt plate used as a working electrode, and SCE utilized as reference electrode, and Pt plate counter electrode.