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*Supporting Information for:*

# Chiral Nanostructuring of Multivalent Macrocycles in Solution and on Surfaces

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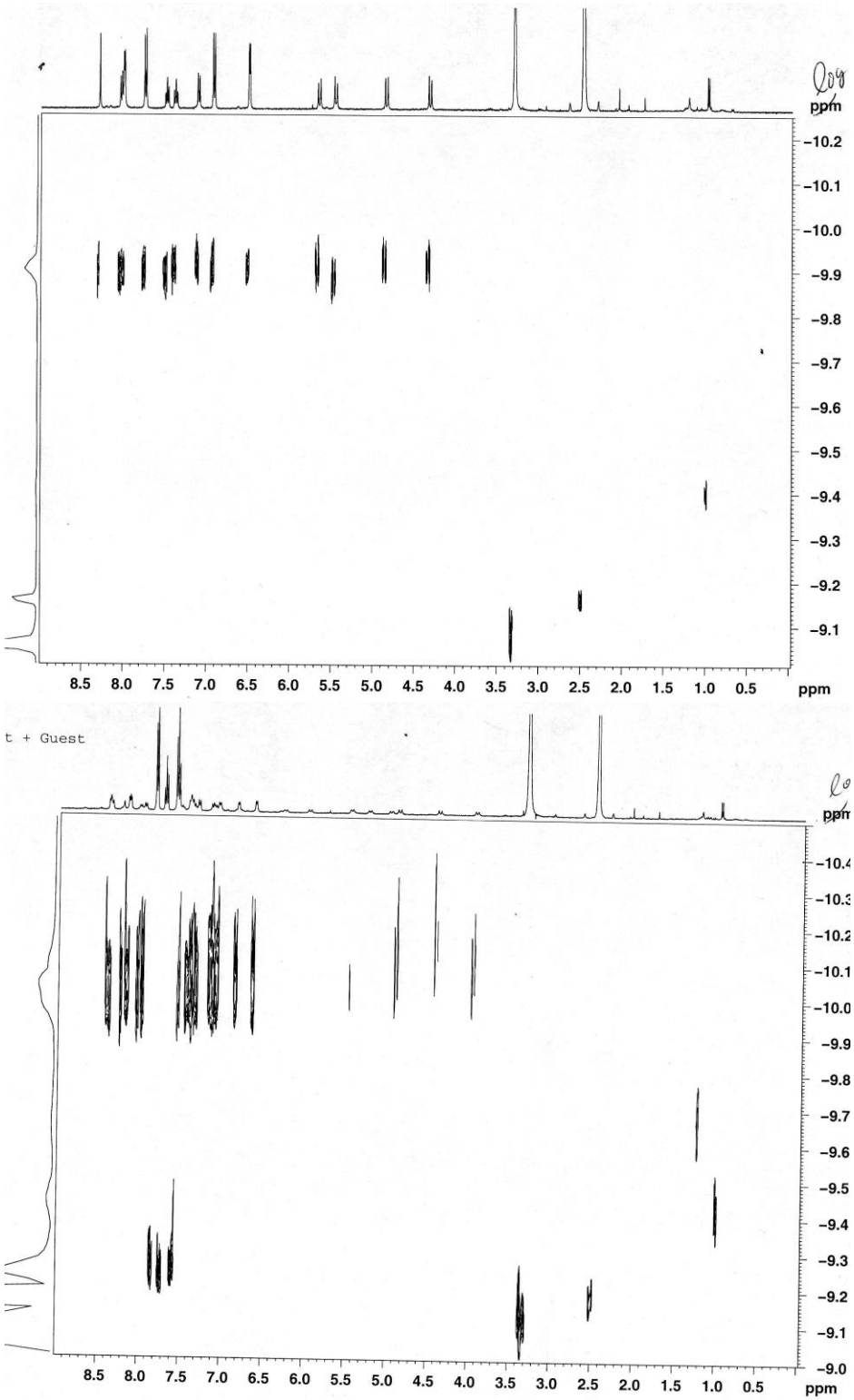



**Figure S1.** 2D $^{1}$H-DOSY NMR spectra of macrocyle (*RR*)-**8** (top, 2 mM), and the adduct obtained by the addition of 2 equivalents of $Pd(PhCN)_2Cl_2$.

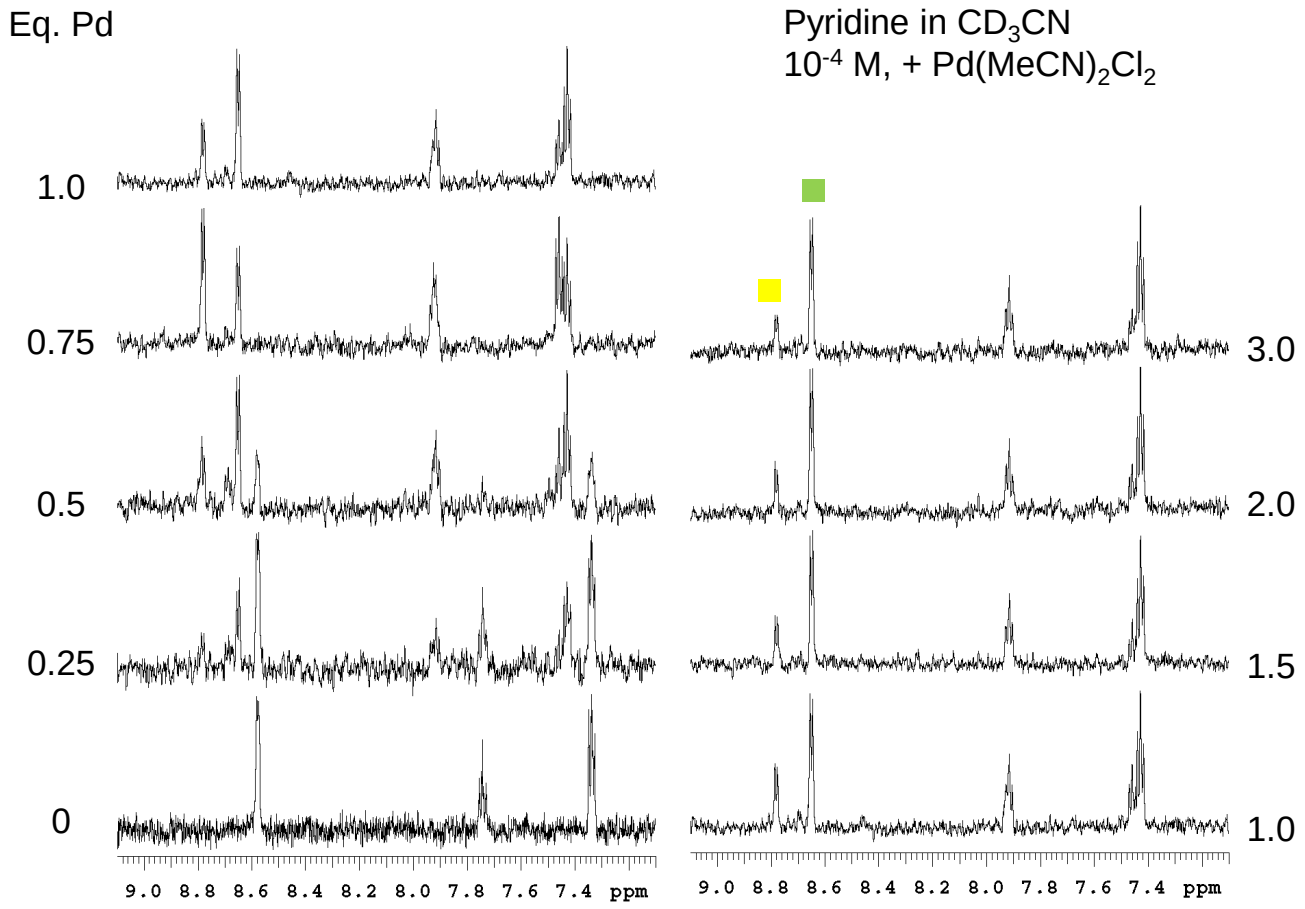



**Figure S2.** $^{1}$H NMR titration of pyridine (0.1 mM) with $Pd(MeCN)_2Cl_2$ in $d_3$-MeCN at 25 °C. The peaks marked with yellow and green squares, respectively, are attributed to the α-pyridyl proton resonances of the *cis* and *trans* square-planar $Pd(py)_2Cl_2$ complex.

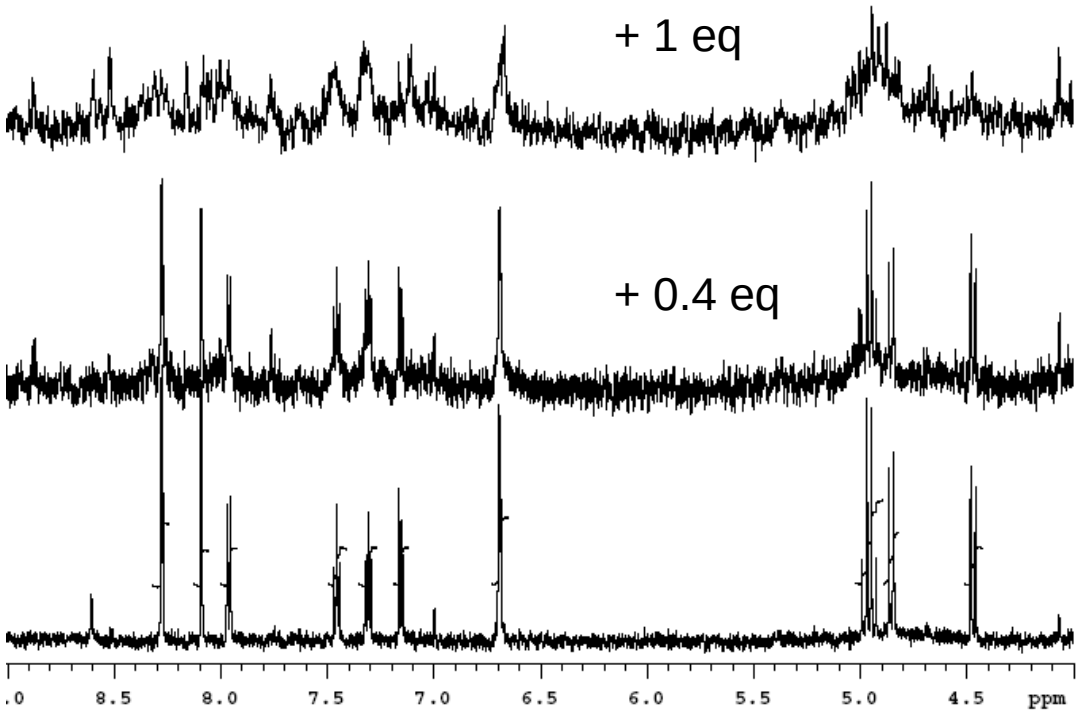



**Figure S3.** Titration of compound (*R*)-**6** (1.8 x $10^{-5}$ M) with $Pd(MeCN)_2Cl_2$ in $d_3$-MeCN at 25 °C (500 MHz).

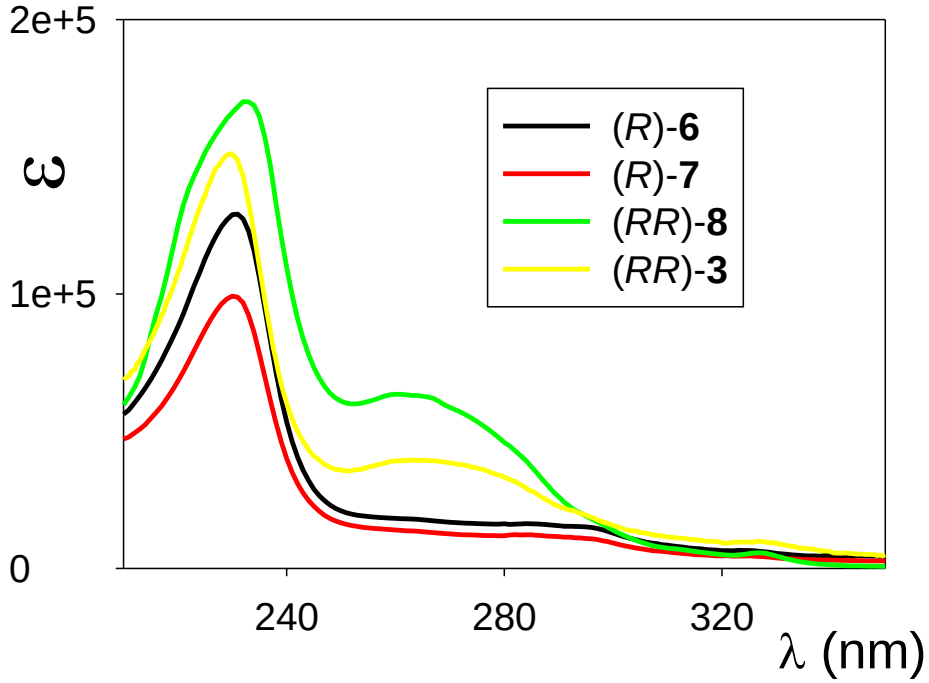



**Figure S4.** UV spectra of pyridine containing precursors (*R*)-**6** and (*R*)-**7**, and of macrocycles (*RR*)-**3** and (*RR*)-**8** (MeCN, c = 1-1.33 x $10^{-5}$ M).

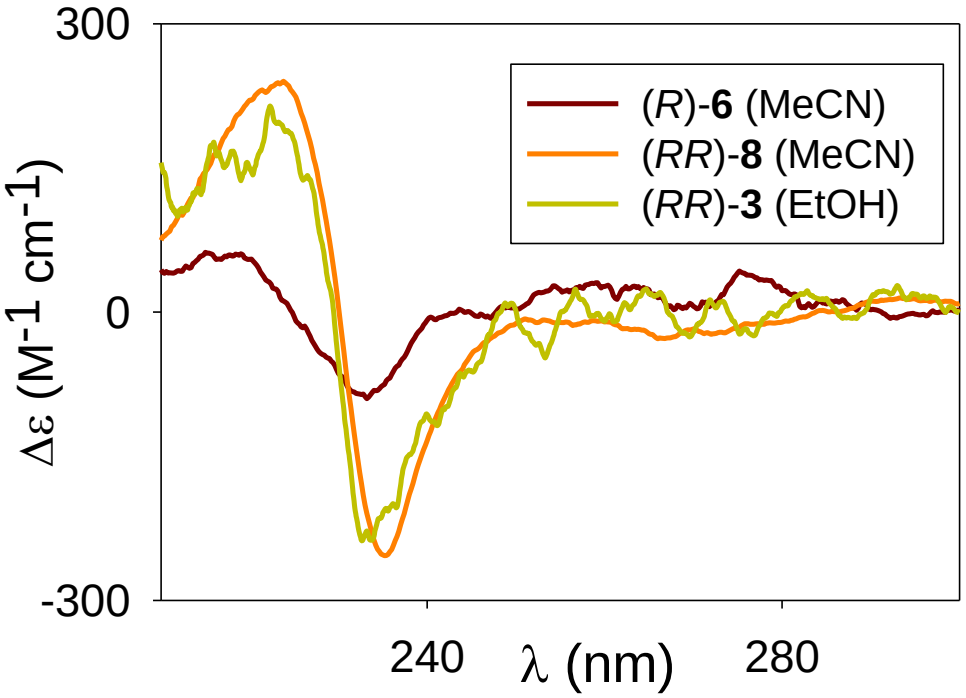



**Figure S5.** CD spectra of pyridine containing precursor (*R*)-**6**, and of macrocycles (*RR*)-**3** and (*RR*)-**8** (c = 1-5 x $10^{-6}$ M).

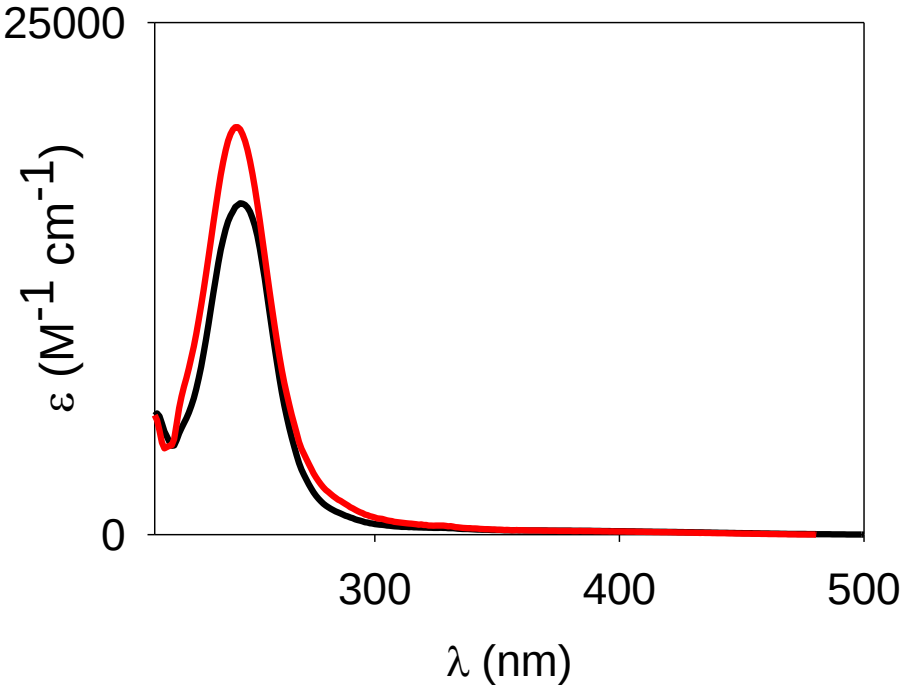



**Figure S6.** UV/Vis spectra of $Pd(MeCN)_2Cl_2$ (1.48 x $10^{-4}$-8.07 x $10^{-5}$ M) in MeCN.

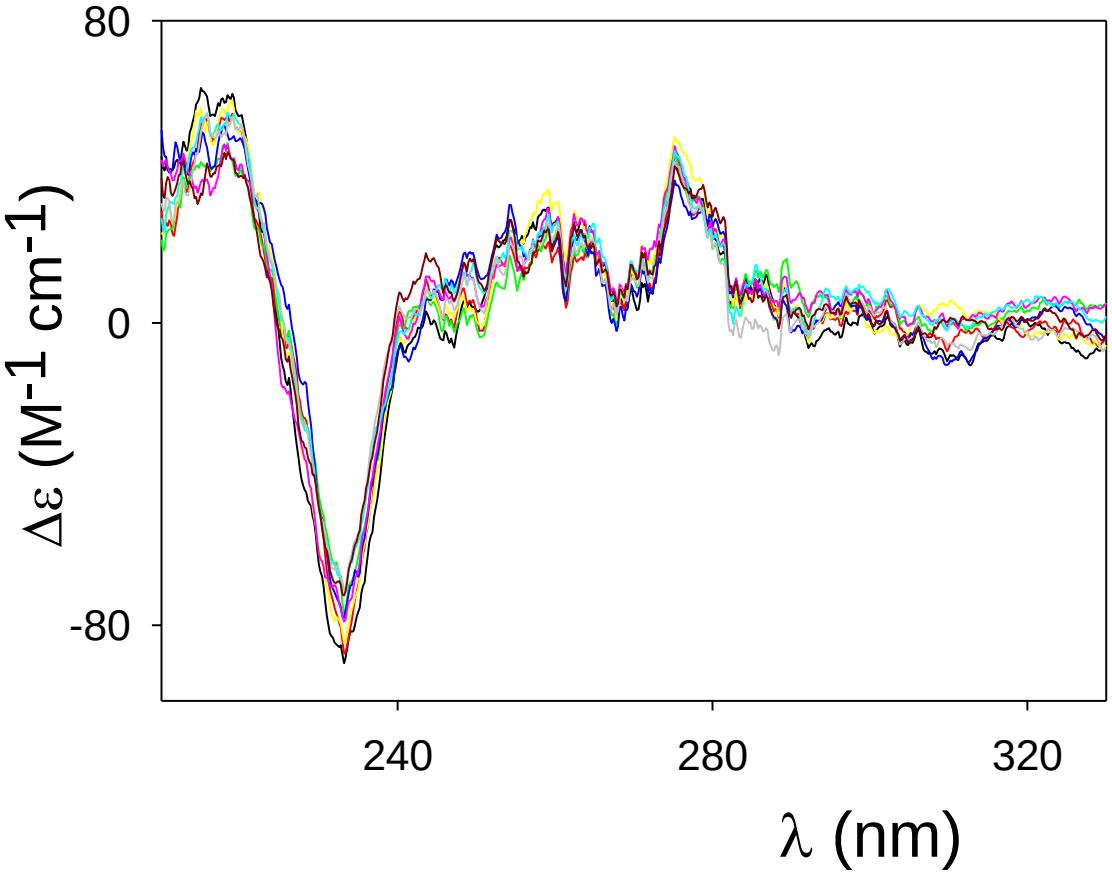
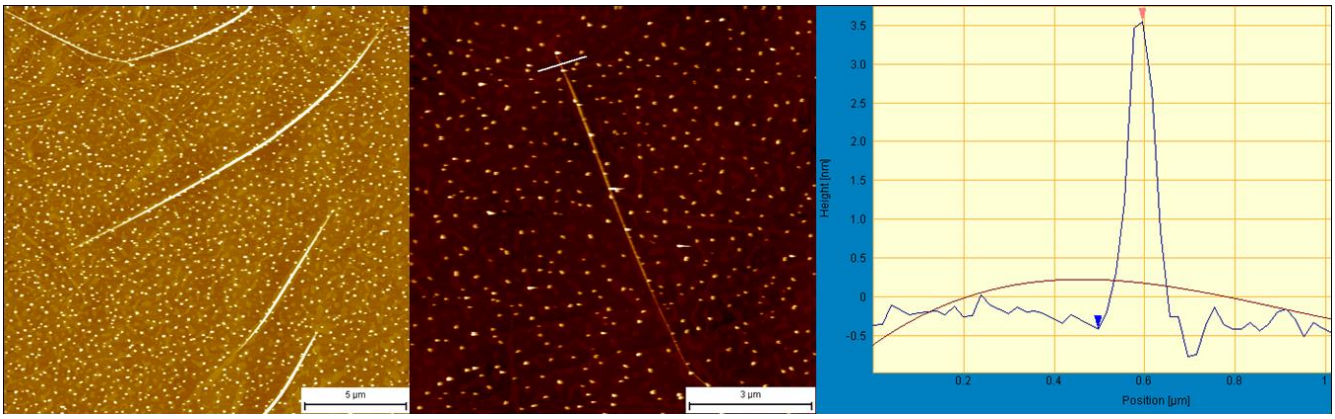



**Figure S7.** CD Titration of compound (*R*)-**6** (1.33 x $10^{-6}$ M) with $Pd(MeCN)_2Cl_2$ in MeCN at 25°C.



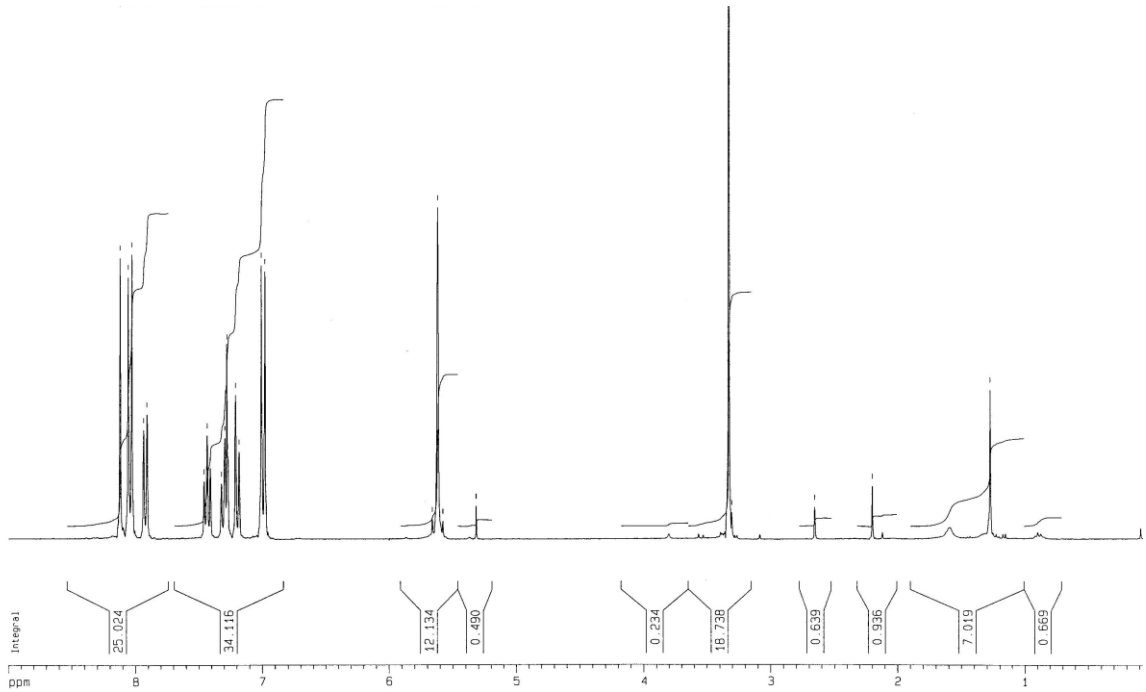

A B C

**Figure S8**. AFM images of (*RR*)-**8**, $C_{60}$ and $Pd(MeCN)_2Cl_2$ diluted in $CHCl_3$ at the graphite interface. (A and B) Example of elongated fibres. (C) A height profile measured along the white line shown (B).

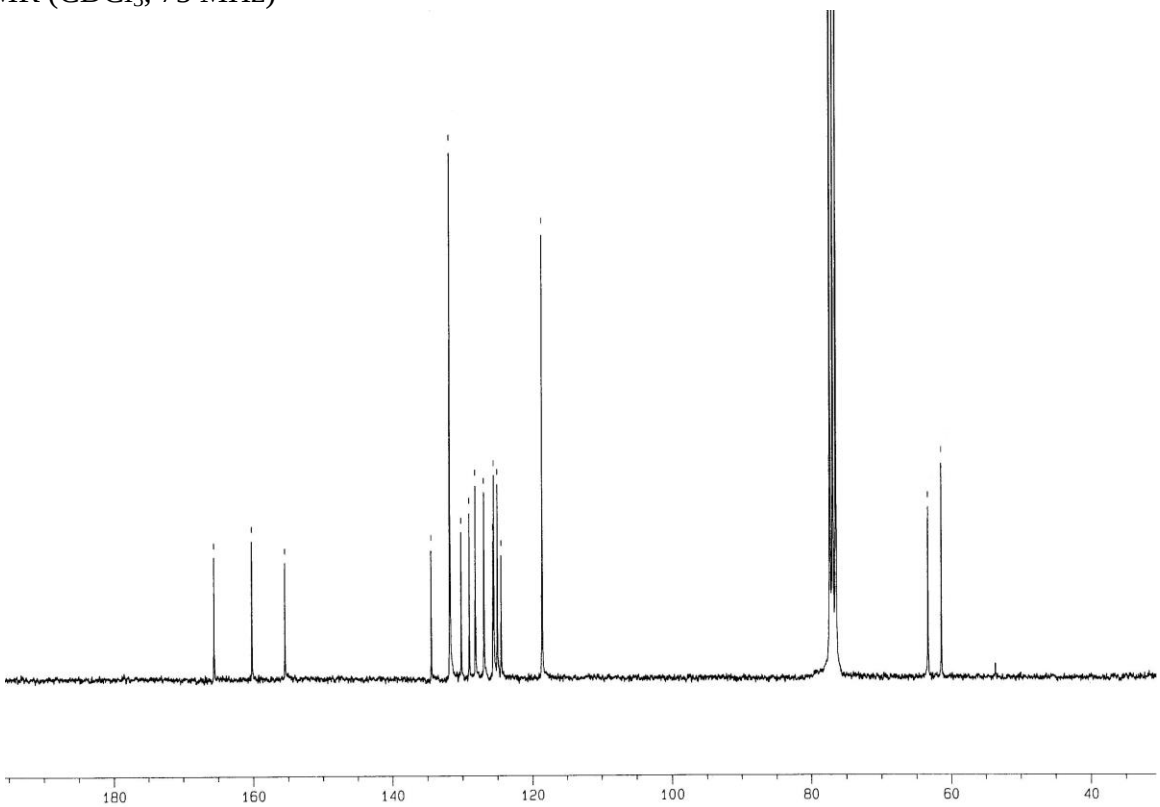
*Copies of NMR and Mass Spectra of Newly Synthesized Compounds*

*Macrocycle* (*RR*)-**3**.

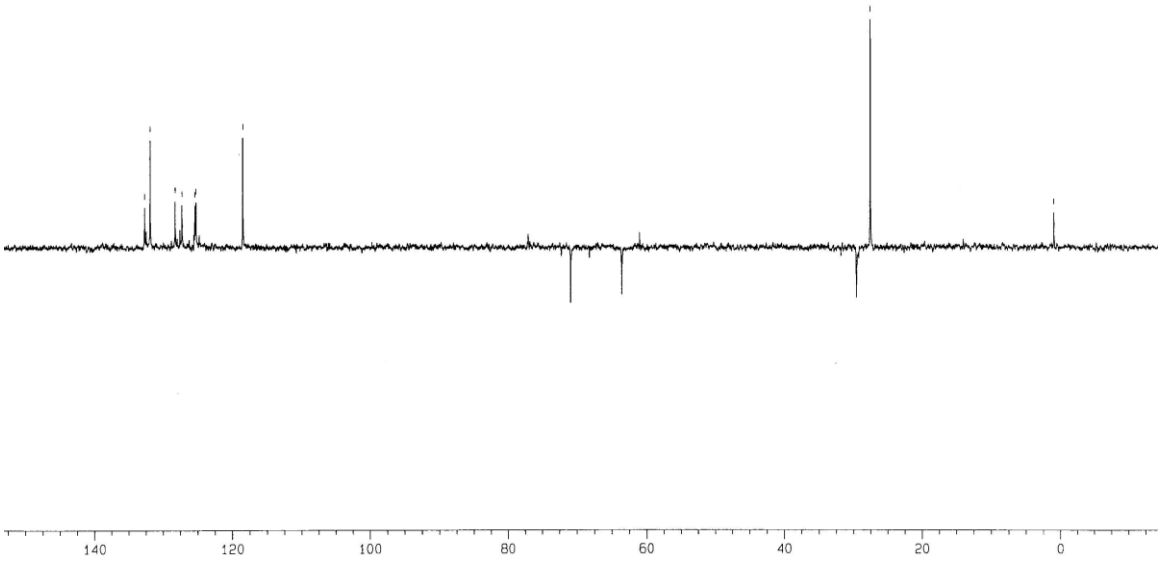
$^{1}$H NMR ($CDCl_3$, 300 MHz)



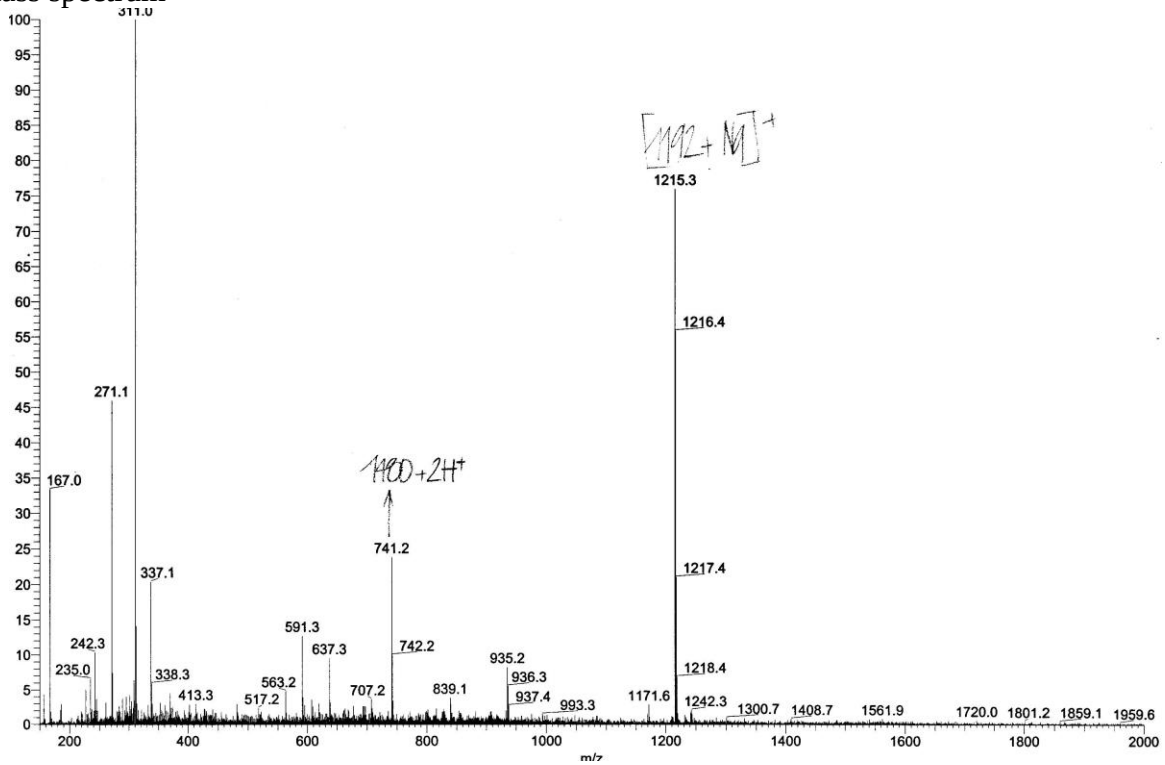

$^{13}$C NMR ($CDCl_3$, 75 MHz)



$^{13}C$ NMR DEPT ($CDCl_3$, 75 MHz)

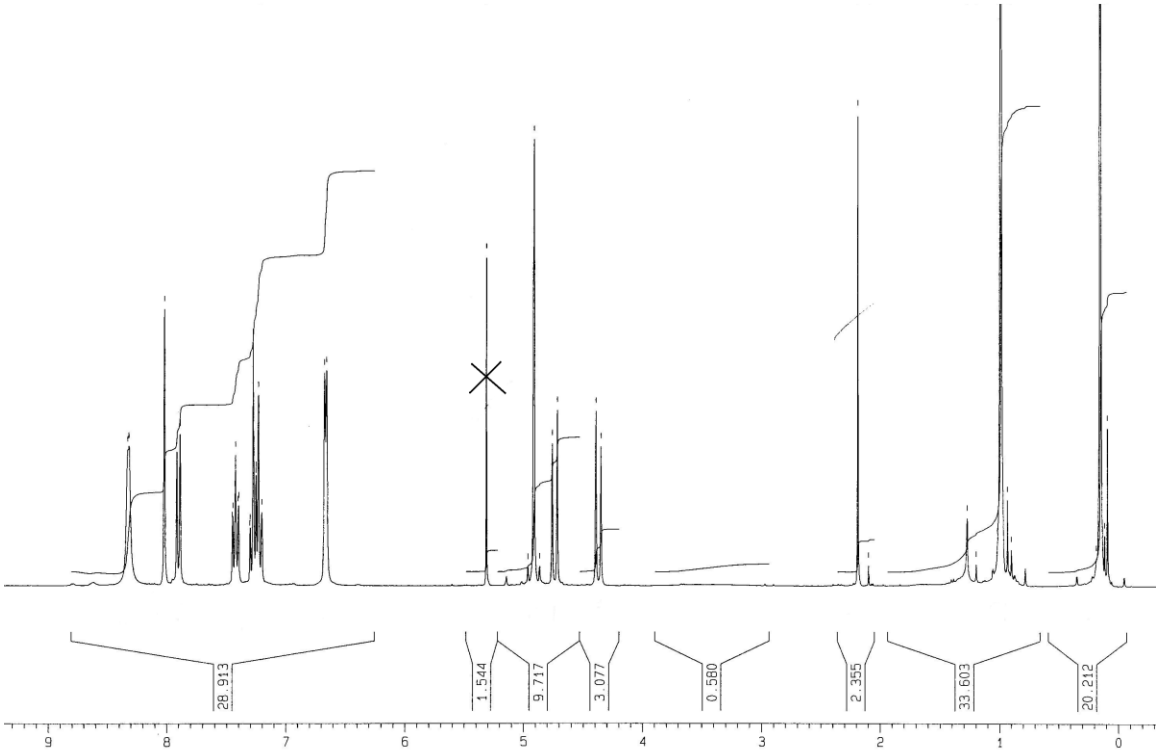



ESI mass spectrum

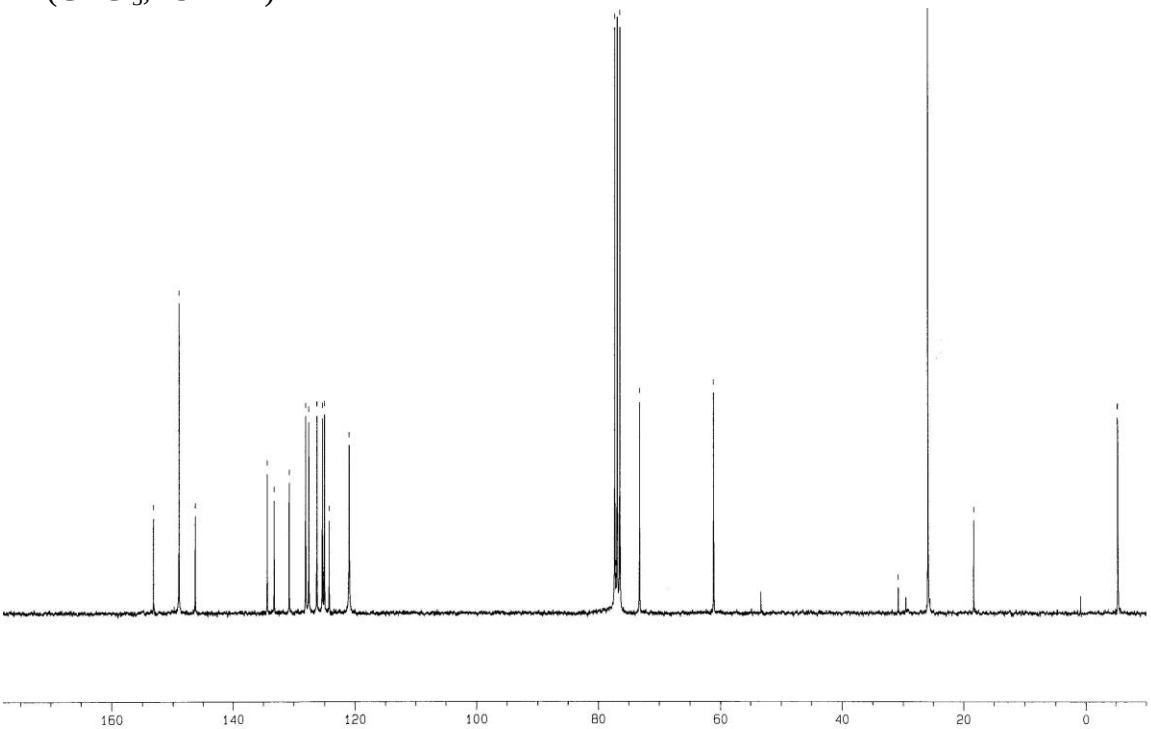


*Compound* (*R*)-**6**

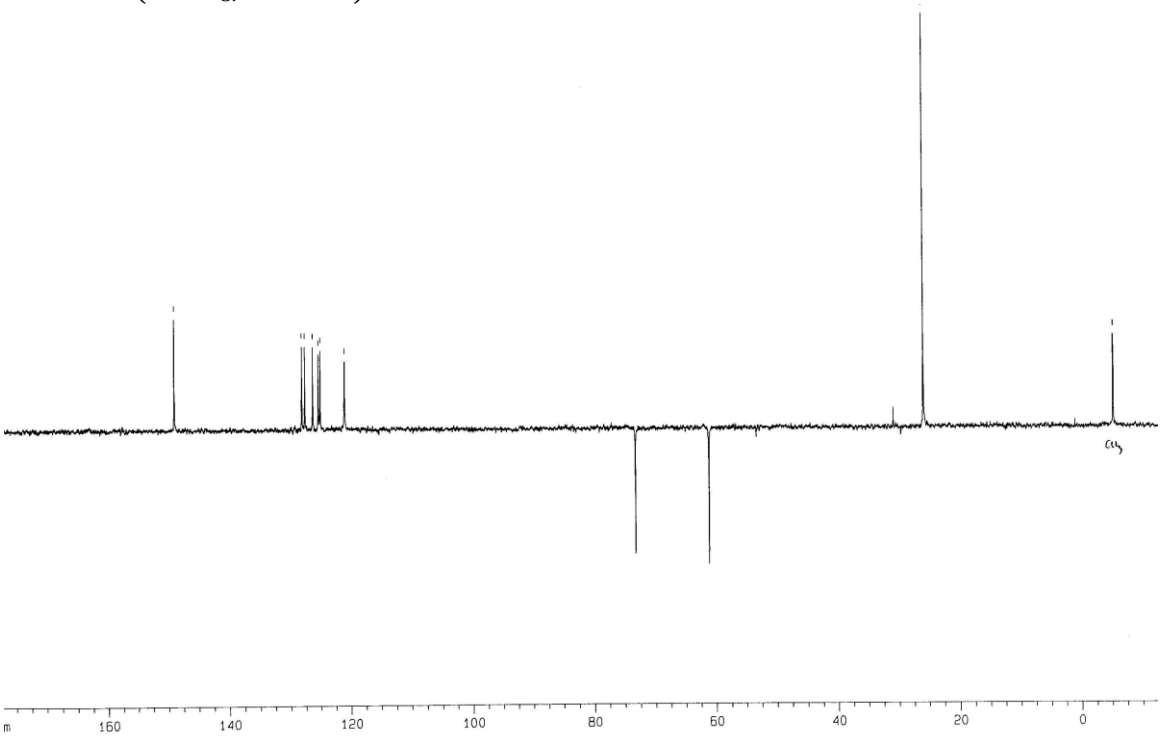
$^{1}$H NMR ($CDCl_3$, 300 MHz)



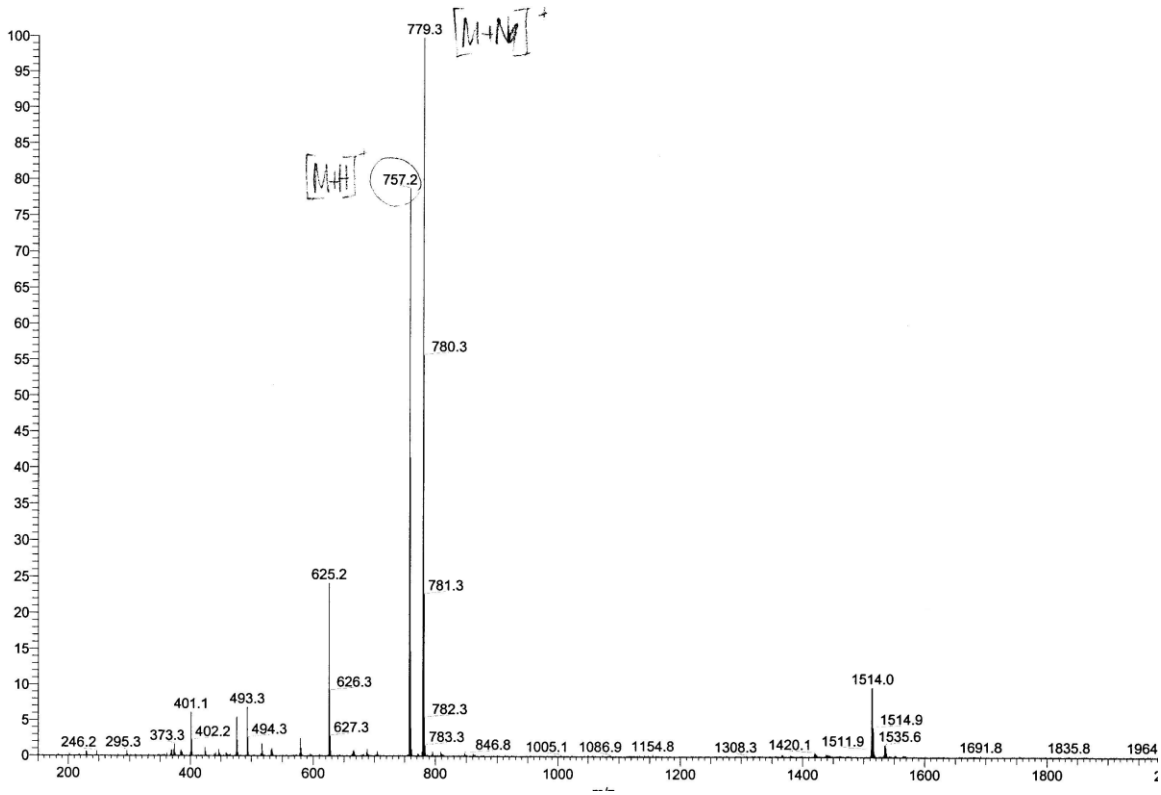

$^{13}$C NMR ($CDCl_3$, 75 MHz)



$^{13}C$ NMR DEPT ($CDCl_3$, 75 MHz)

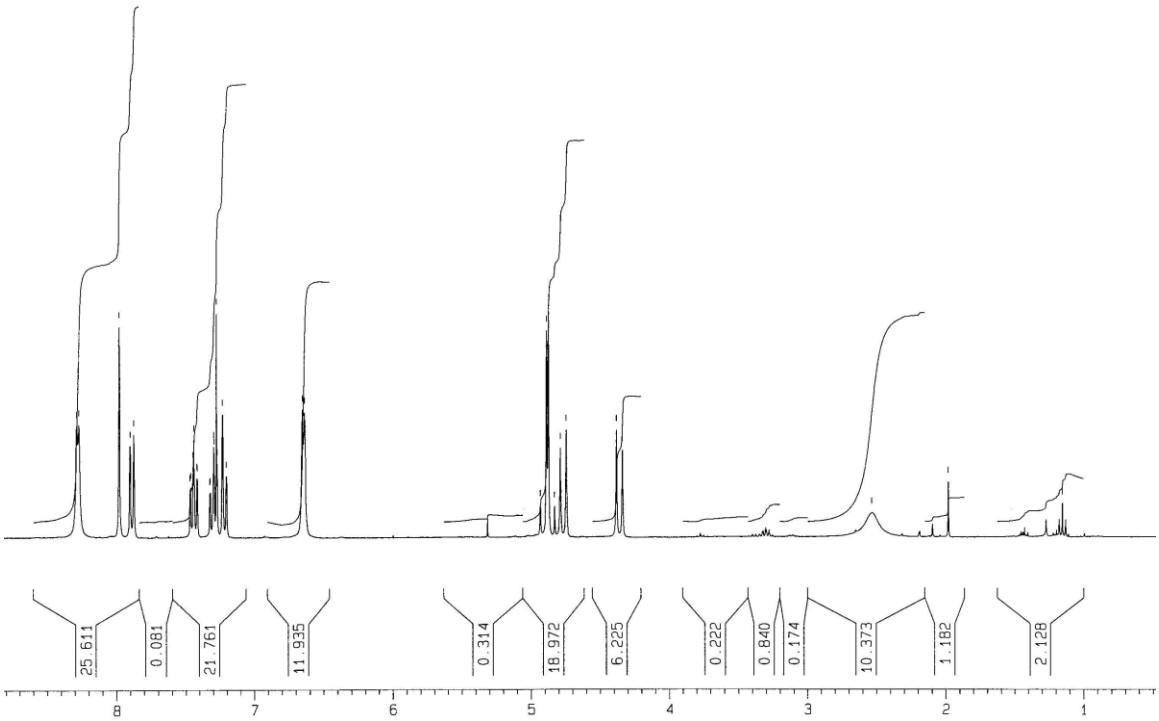



ESI mass spectrum

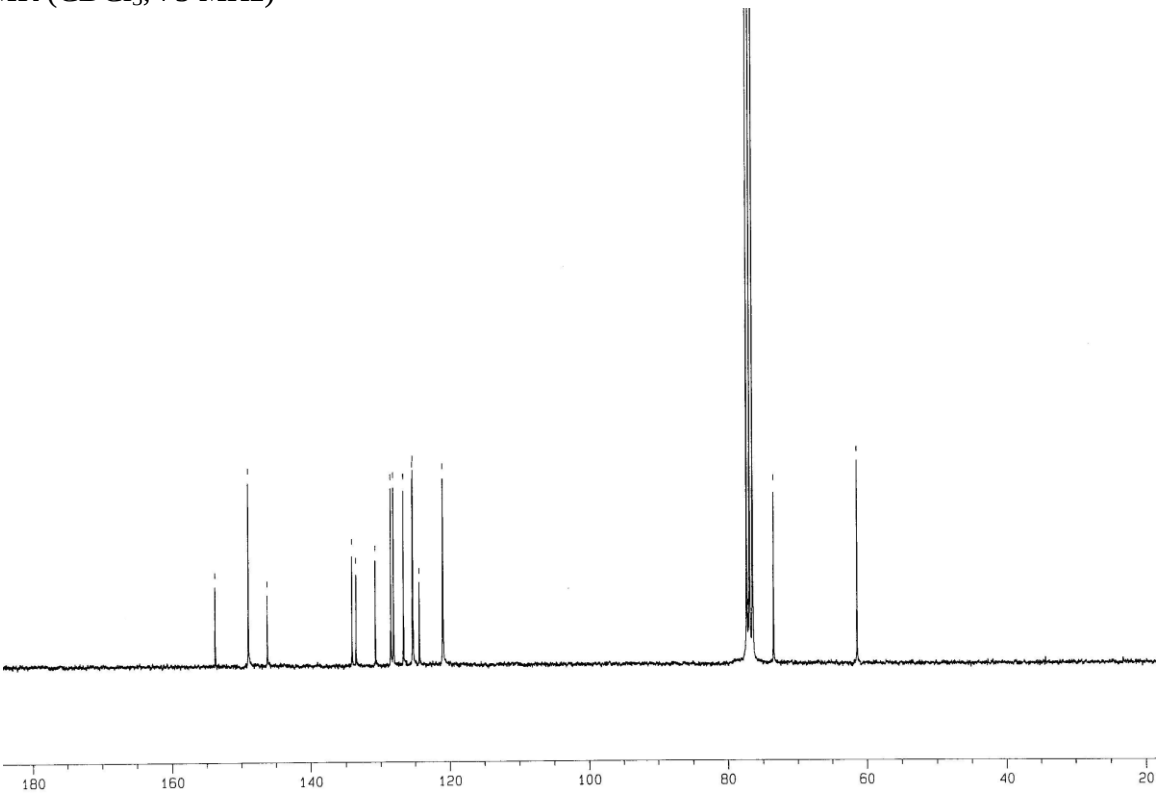


*Compound* (*R*)-**7**.

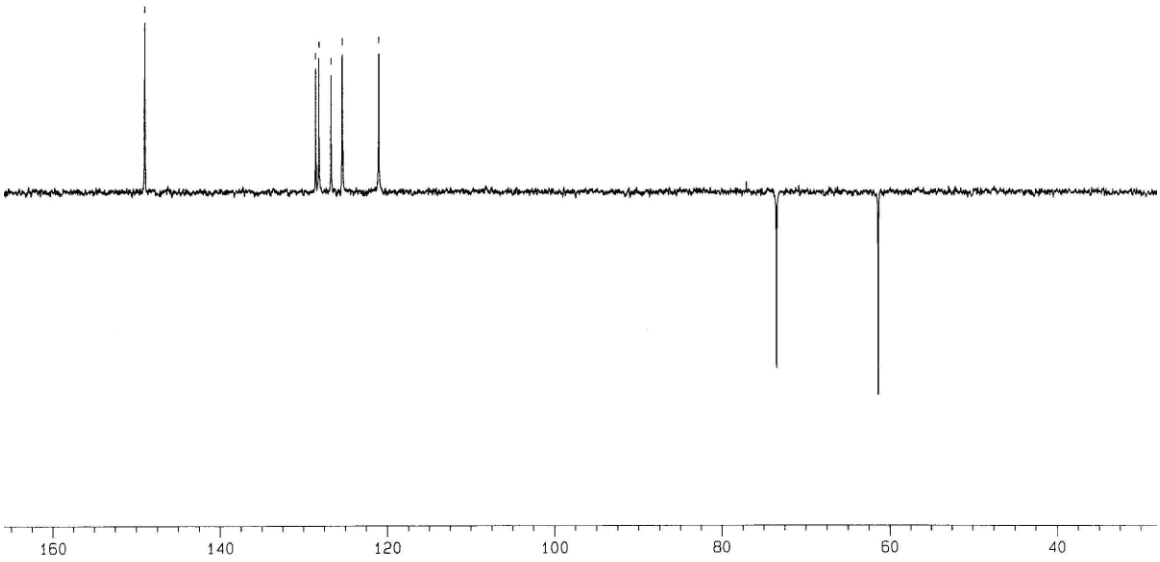
$^{1}$H NMR ($CDCl_3$, 300 MHz)



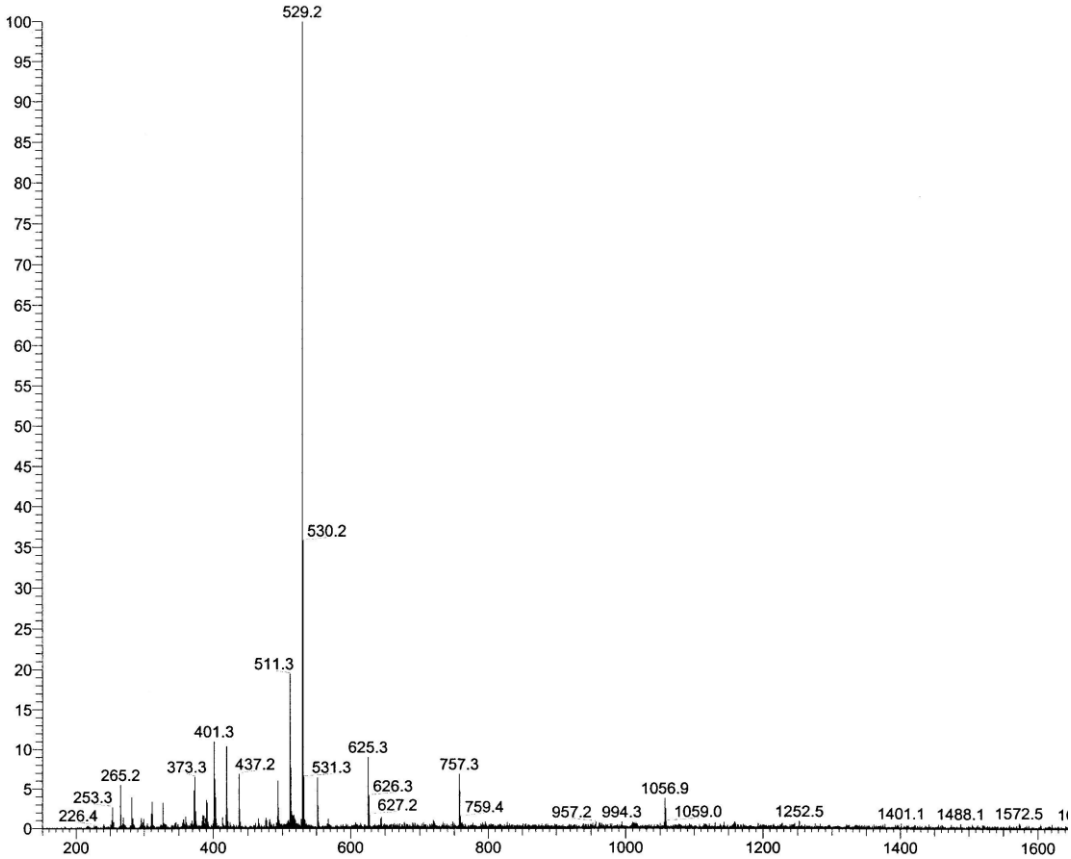

$^{13}$C NMR ($CDCl_3$, 75 MHz)



$^{13}C$ NMR DEPT ($CDCl_3$, 75 MHz)

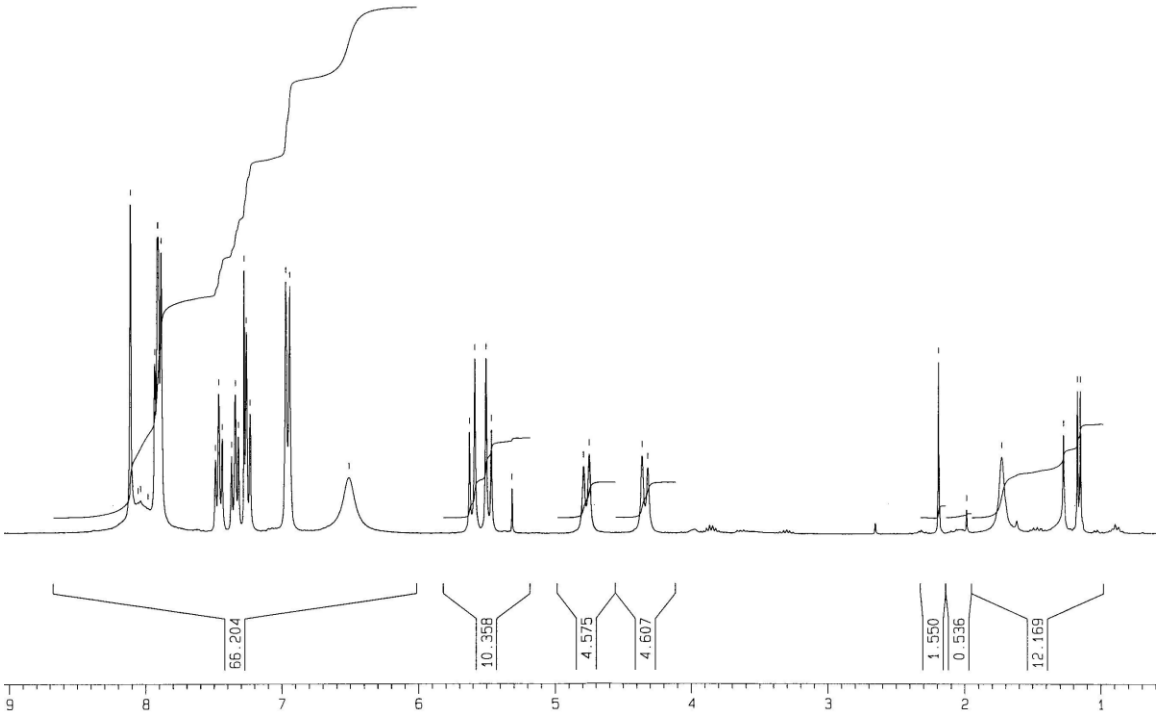



ESI mass spectrum

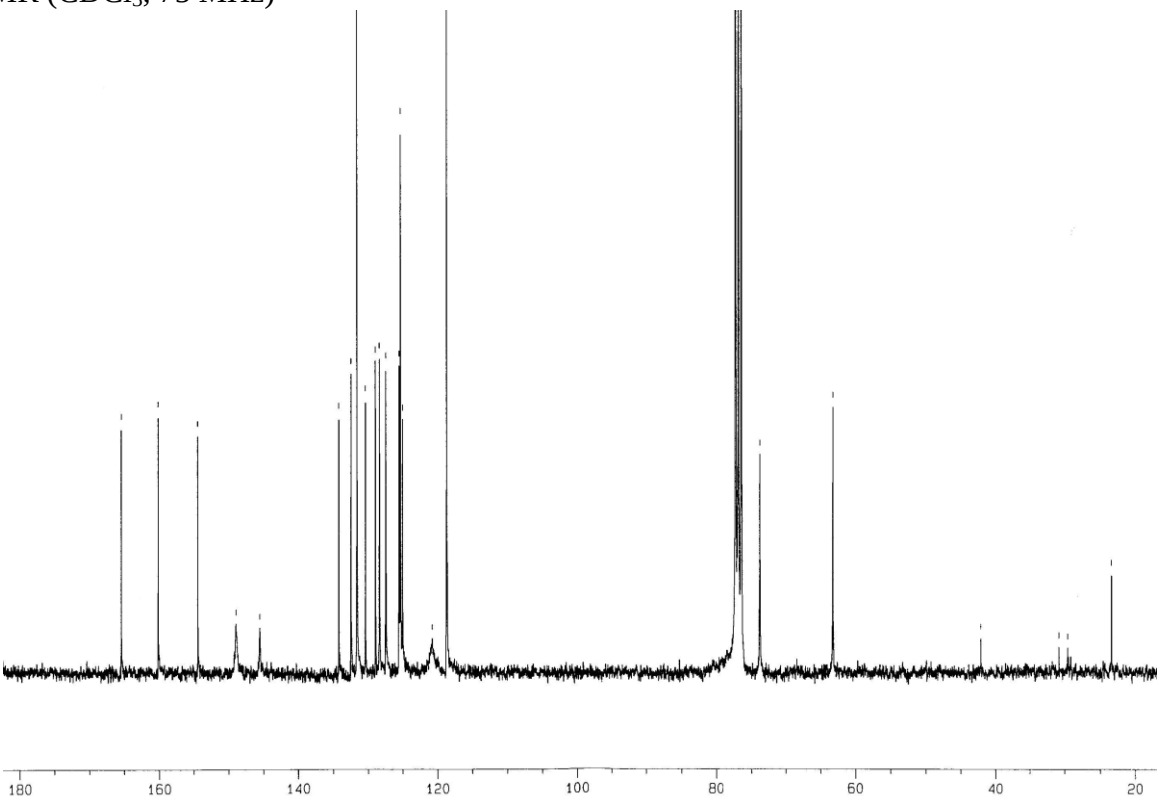


*Macrocycle* (*RR*)-**8**.

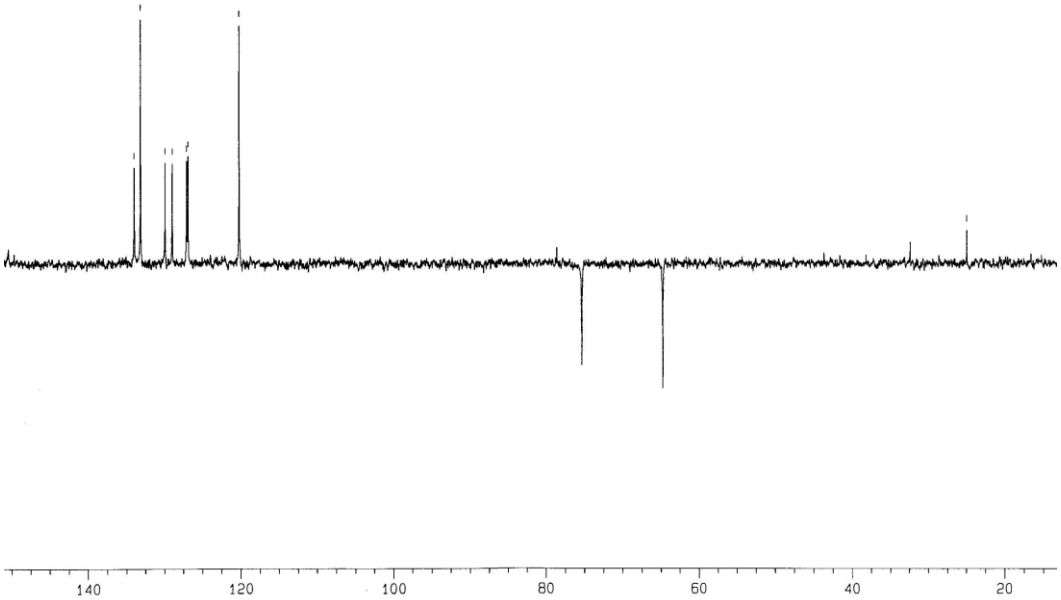
$^{1}$H NMR ($CDCl_3$, 300 MHz)




$^{13}$C NMR ($CDCl_3$, 75 MHz)



$^{13}C$ NMR DEPT ($CDCl_3$, 75 MHz)




ESI mass spectrum