

**Supporting Information**  
**for**  
**Self-assembly of metallosupramolecular rhombi**  
**from chiral concave 9,9'-spirobifluorene-derived**  
**bis(pyridine) ligands**

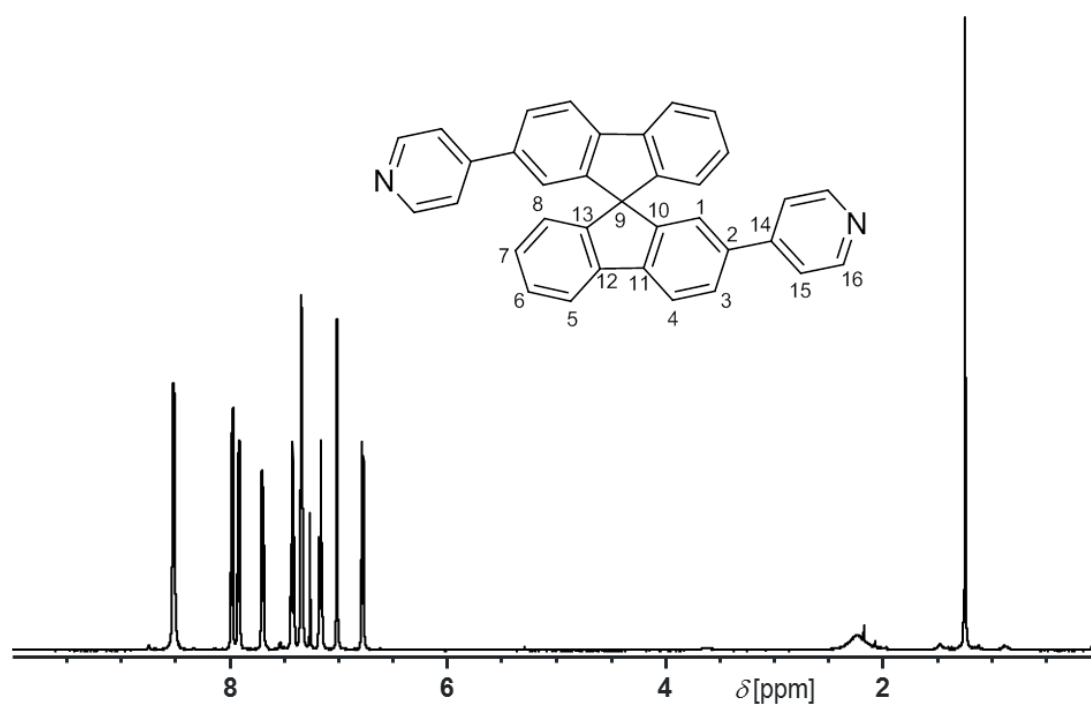
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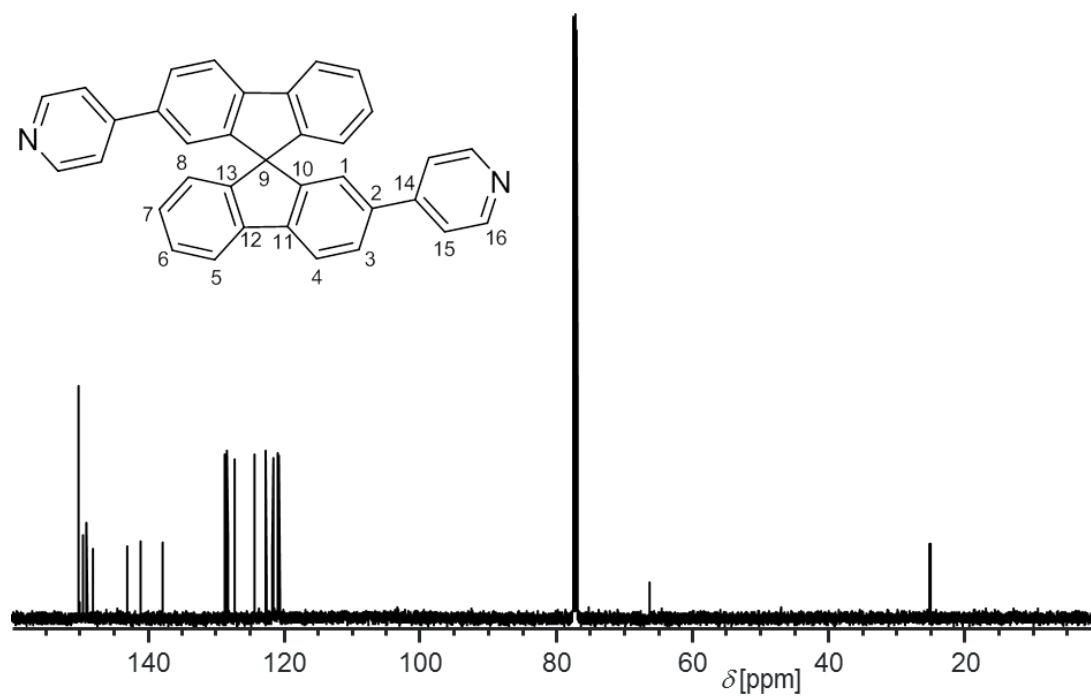
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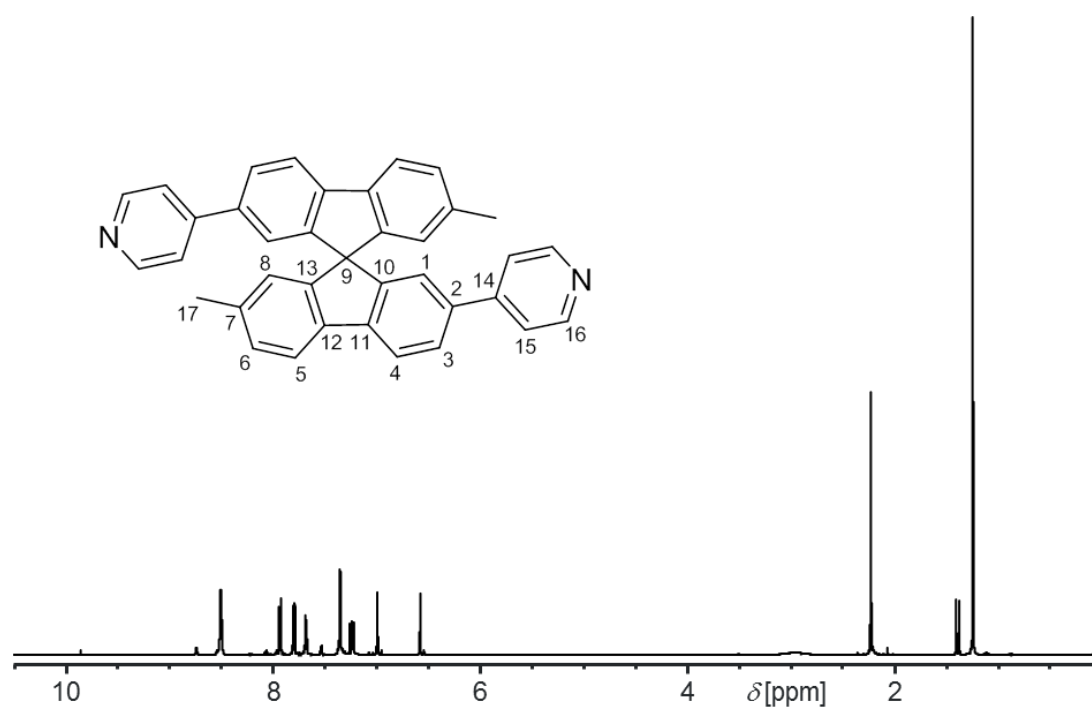
**NMR and ESI mass spectra of compounds 3 and 6 and their metal complexes**



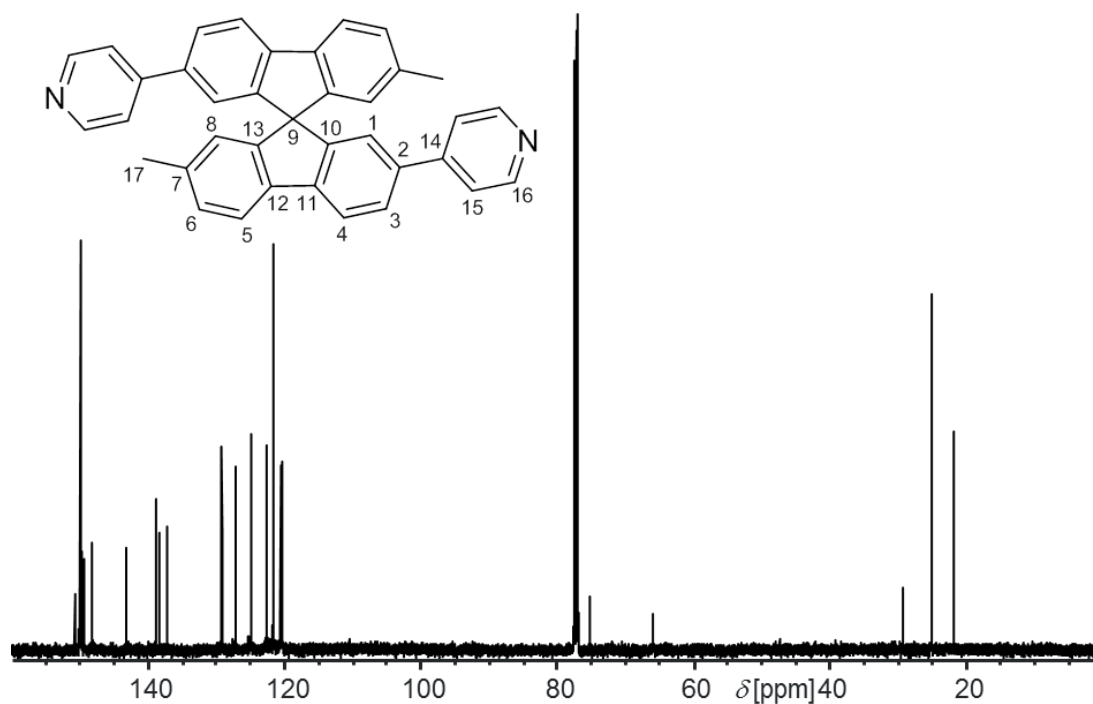
**Figure S1:**  $^1\text{H}$  NMR (400.1 MHz, in  $\text{CDCl}_3$  at 293 K) of **(rac)-3**.



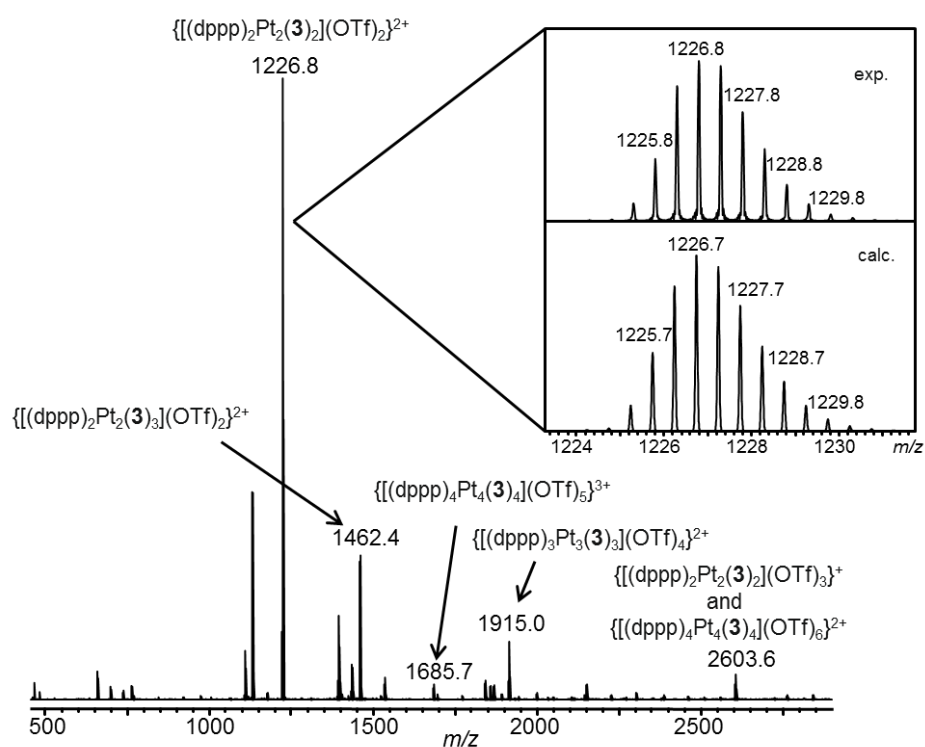
**Figure S2:**  $^{13}\text{C}$  NMR (100.6 MHz, in  $\text{CDCl}_3$  at 293 K) of **(rac)-3**.



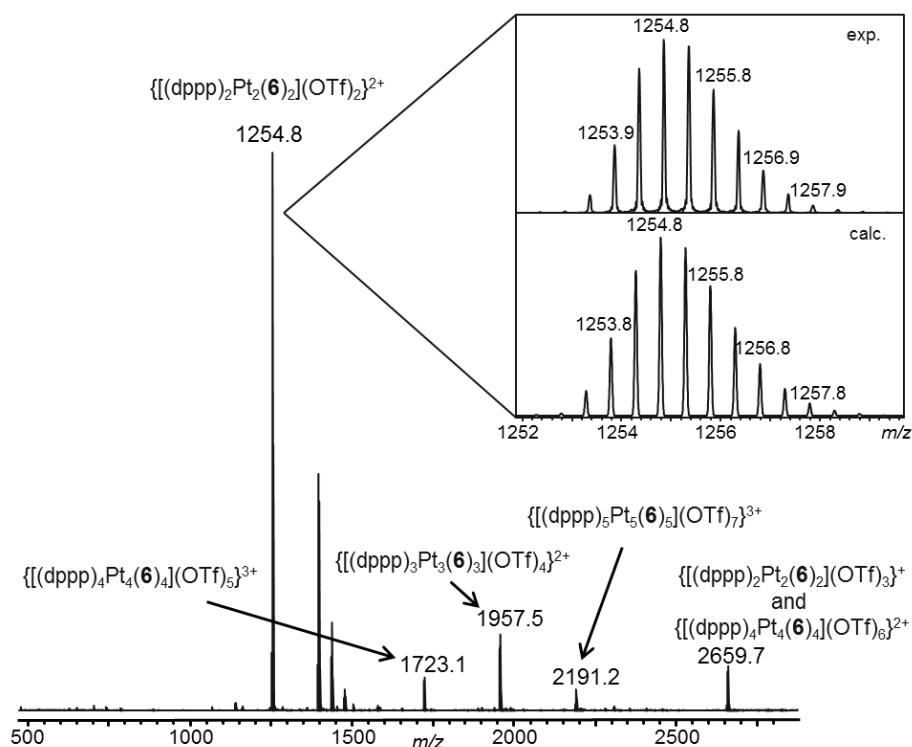
**Figure S3:**  $^1\text{H}$  NMR (400.1 MHz, in  $\text{CDCl}_3$  at 293 K) of (S)-6.



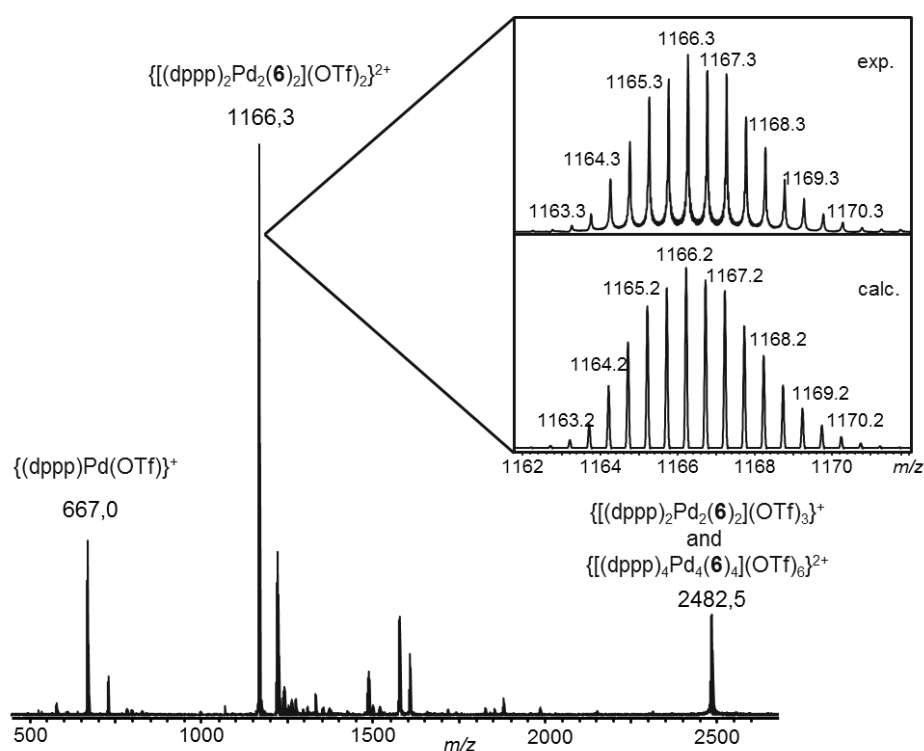
**Figure S4:**  $^{13}\text{C}$  NMR (100.6 MHz, in  $\text{CDCl}_3$  at 293 K) of (S)-6.



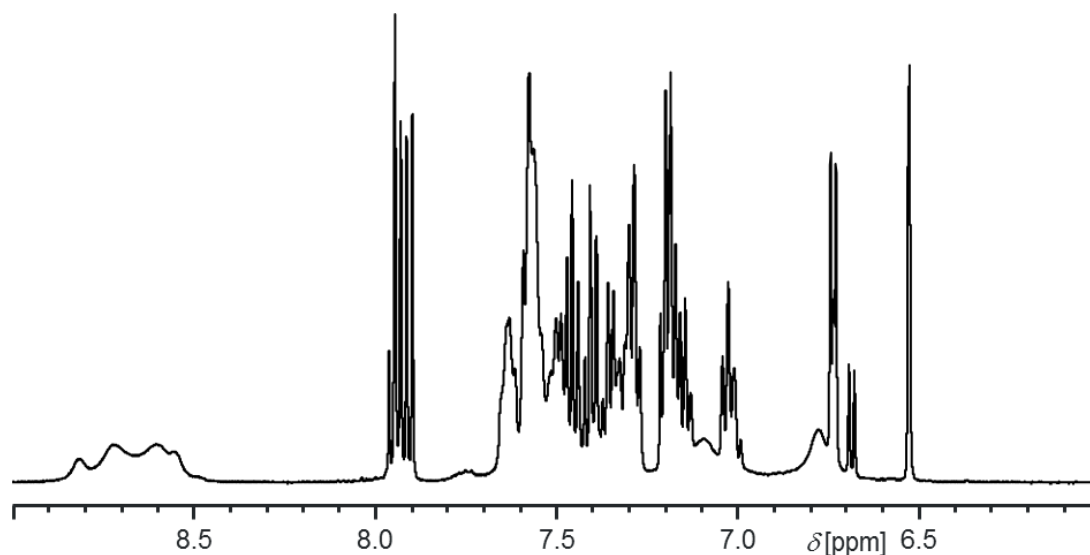
**Figure S5:** ESIMS (positive mode, sprayed from an acetone solution) of  $[(\text{dppp})_2\text{Pt}_2\{(R)\text{-}\mathbf{3}\}_2](\text{OTf})_4$ .



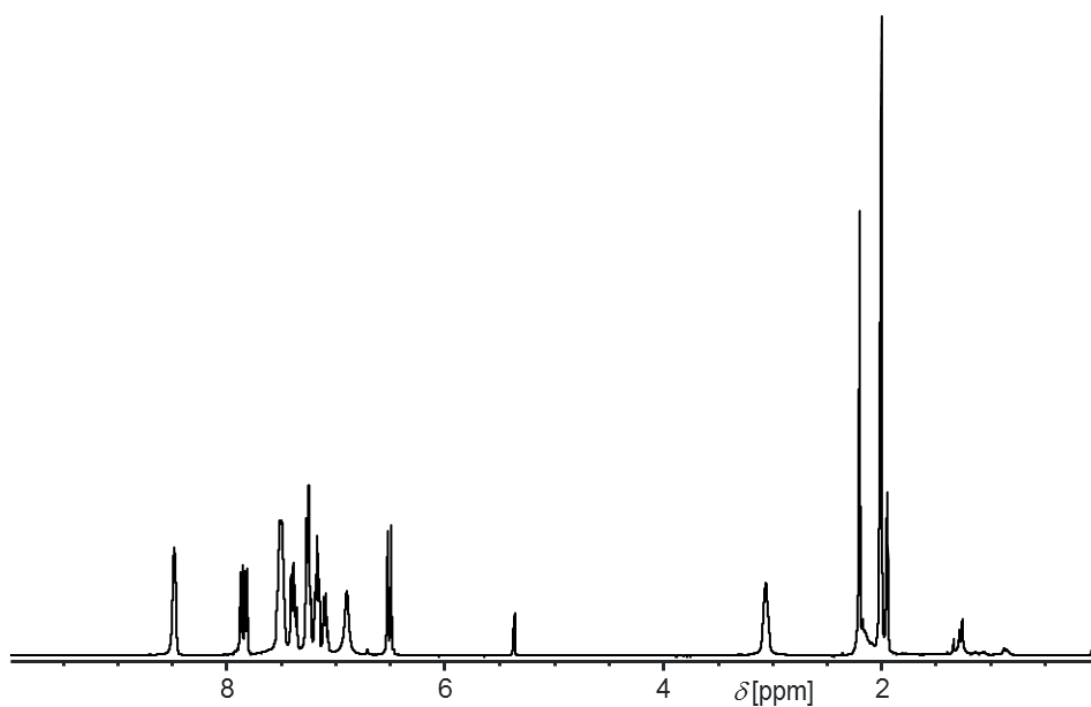
**Figure S6:** ESIMS (positive mode, sprayed from an acetone solution) of  $[(\text{dppp})_2\text{Pt}_2\{(S)\text{-}\mathbf{6}\}_2](\text{OTf})_4$ .



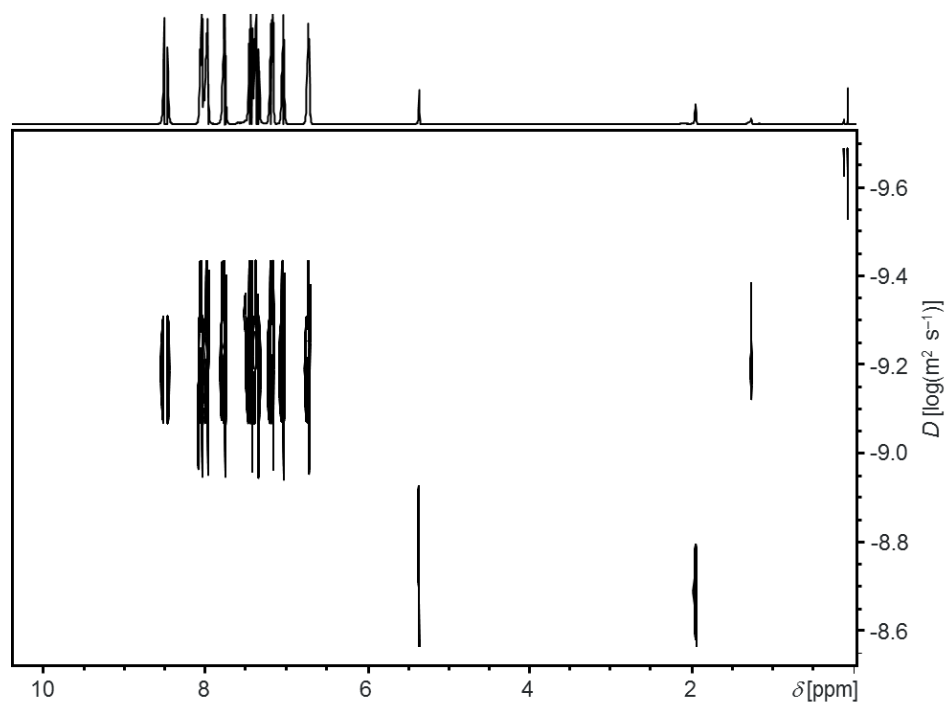
**Figure S7:** ESIMS (positive mode, sprayed from an acetone solution) of  $[(dppp)_2Pd_2\{(S)-\mathbf{6}\}_2](OTf)_4$ .



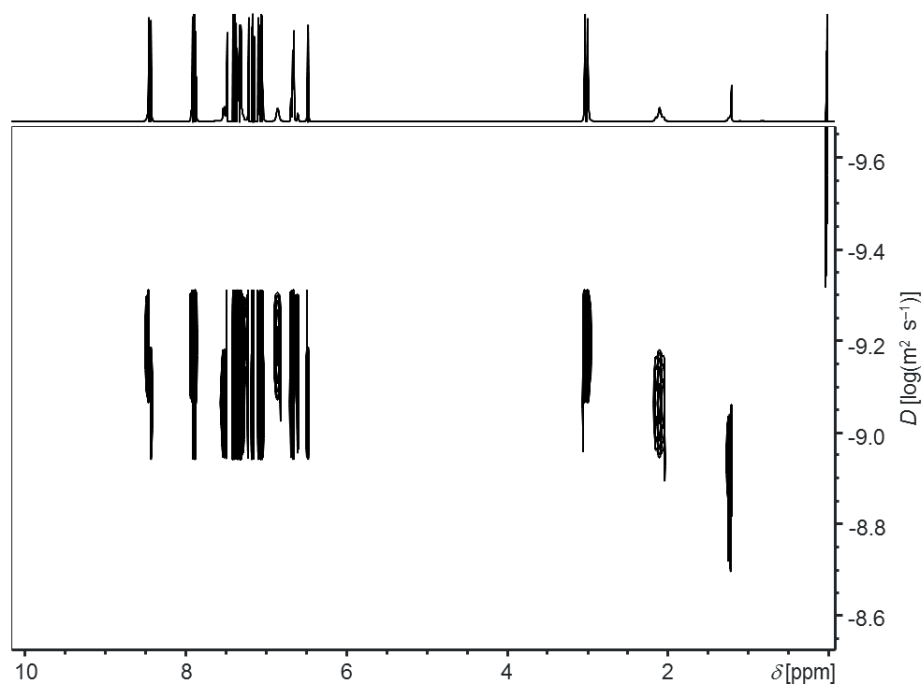
**Figure S8:**  $^1H$  NMR (500.1 MHz, in  $CD_2Cl_2$  at 298 K) of a 1:1 mixture of  $[Pt(dppp)]OTf_2$  and  $(rac)\text{-}\mathbf{3}$ .



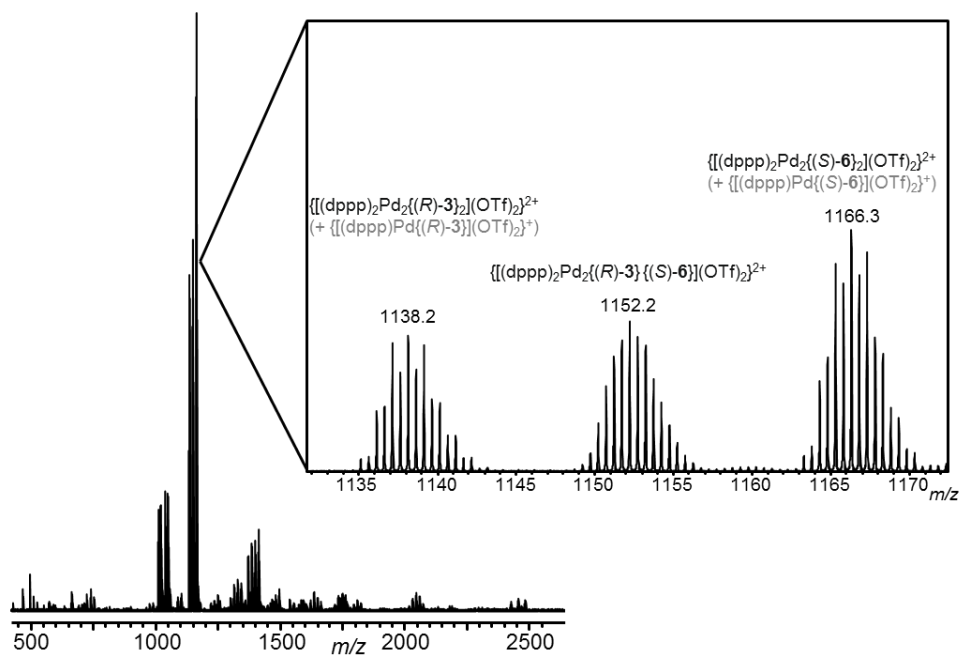
**Figure S9:**  $^1\text{H}$  NMR (500.1 MHz, in  $\text{CD}_2\text{Cl}_2/\text{CD}_3\text{CN}$  3:1 at 298 K) of a 1:1 mixture of  $[\text{Pd}(\text{dppp})]\text{OTf}_2$  and (S)-**6**.



**Figure S10:**  $^1\text{H}$  2D-DOSY-NMR (500.1 MHz, in  $\text{CD}_2\text{Cl}_2/\text{CD}_3\text{CN}$  3:1 at 298 K) of (rac)-**3**.



**Figure S11:**  $^1\text{H}$  2D-DOSY-NMR (500.1 MHz, in  $\text{CD}_2\text{Cl}_2/\text{CD}_3\text{CN}$  3:1 at 298 K) of a 1:1 mixture of  $[\text{Pd}(\text{dppp})]\text{OTf}_2$  and (*rac*)-**3**.



**Figure S12:** ESIMS (positive mode, sprayed from an acetonitrile solution) of a mixture of 2 equiv  $[(\text{dppp})\text{Pd}(\text{OTf})_2]$  + 1 equiv (*R*)-**3** + 1 equiv (*S*)-**6**.