

Supplemental Material. NMR Data and Figures.

Figure 1. Two diastereoisomers for octahedral *racemic* $[M([18]aneS_4N_2)]^{n+}$ complexes.

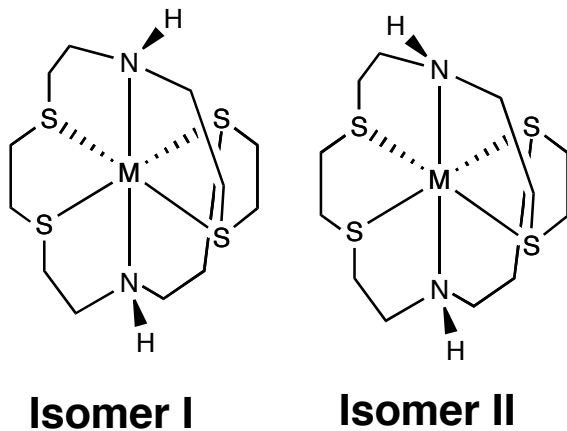


Figure 2. Representative $^{113}Cd\{^1H\}$ -NMR spectrum of $[Cd([10]aneS_3)_2](ClO_4)_2$ in CD_3NO_2 at $25^\circ C$.

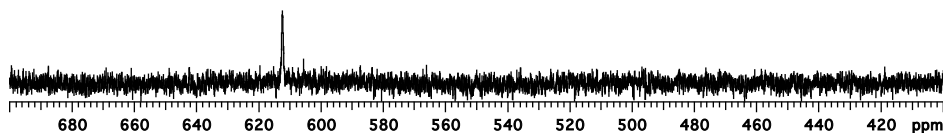


Figure 3. $^{113}Cd\{^1H\}$ -NMR spectrum of $[Cd([9]aneN_3)_2](ClO_4)_2$ in CD_3NO_2 at $25^\circ C$. Note single resonance consistent with the presence of one diastereoisomer.

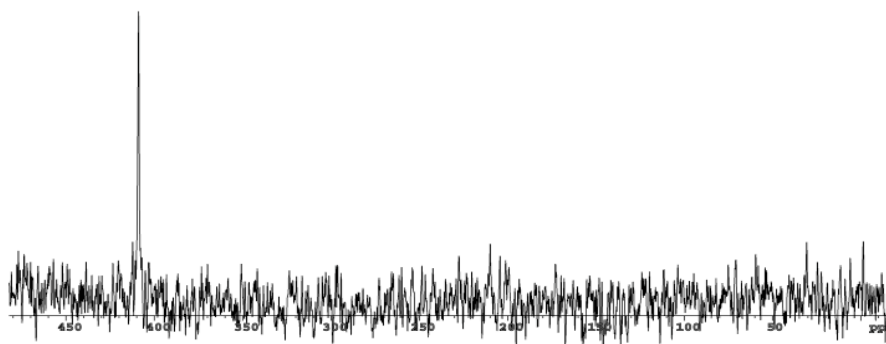


Figure 4. Variable temperature ^1H -NMR spectra of $[\text{Cd}([\text{12}]\text{aneS}_4)_x](\text{ClO}_4)_2$ in CD_3NO_2 .

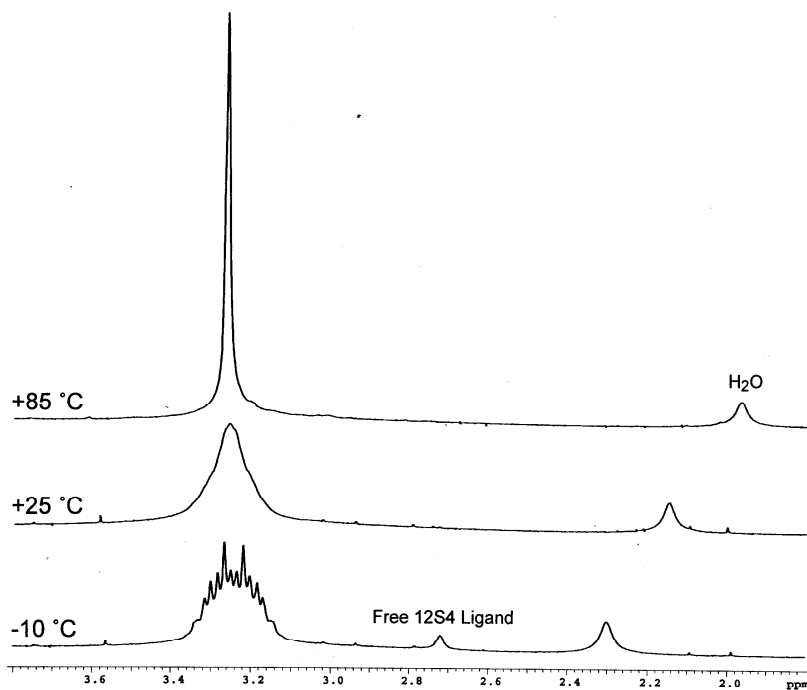


Figure 5. ^1H NMR spectrum of $[\text{Cd}([\text{18}]\text{aneS}_4\text{N}_2)](\text{PF}_6)_2$ in CD_3NO_2 at $25\text{ }^\circ\text{C}$.

