

Supplementary Information

Variable Temperature NMR (VT NMR) Spectra

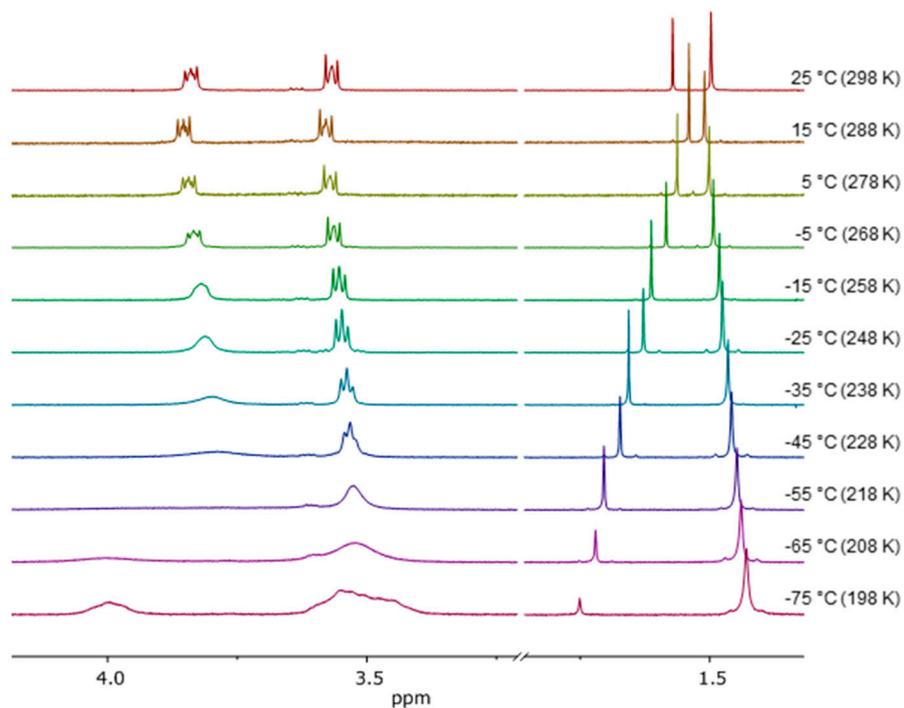


Figure S1. Partial ^1H NMR spectra (400 MHz, CD_2Cl_2) of $[\text{Na}^+\text{c-o-Me}_2\text{-1.1.1}]\text{BARF}^-$.

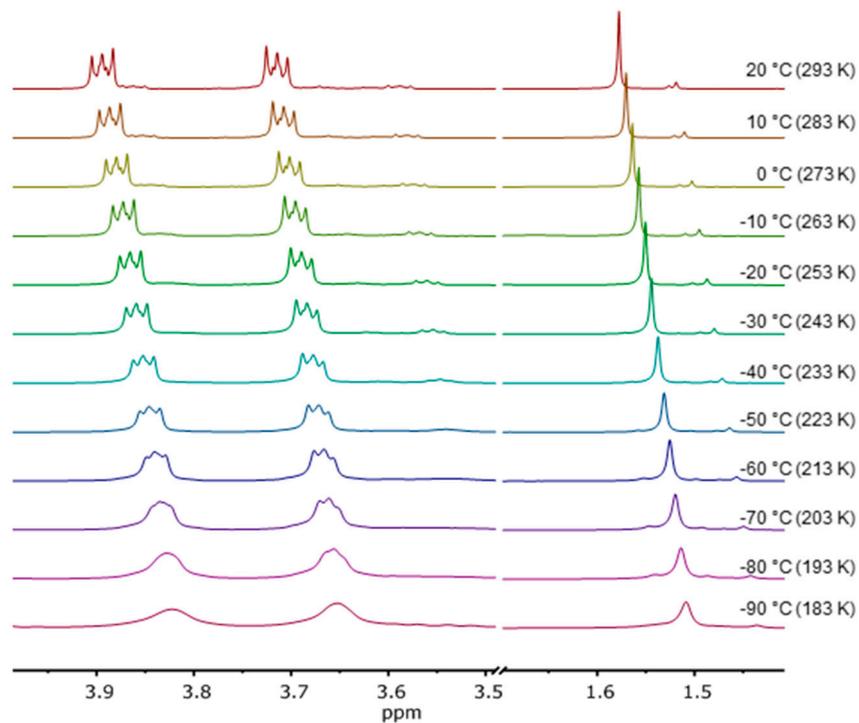


Figure S2. Partial ^1H NMR spectra (400 MHz, CD_2Cl_2) of $[\text{Li}^+\text{c-o-Me}_2\text{-1.1.1}]\text{TPFPB}^-$.

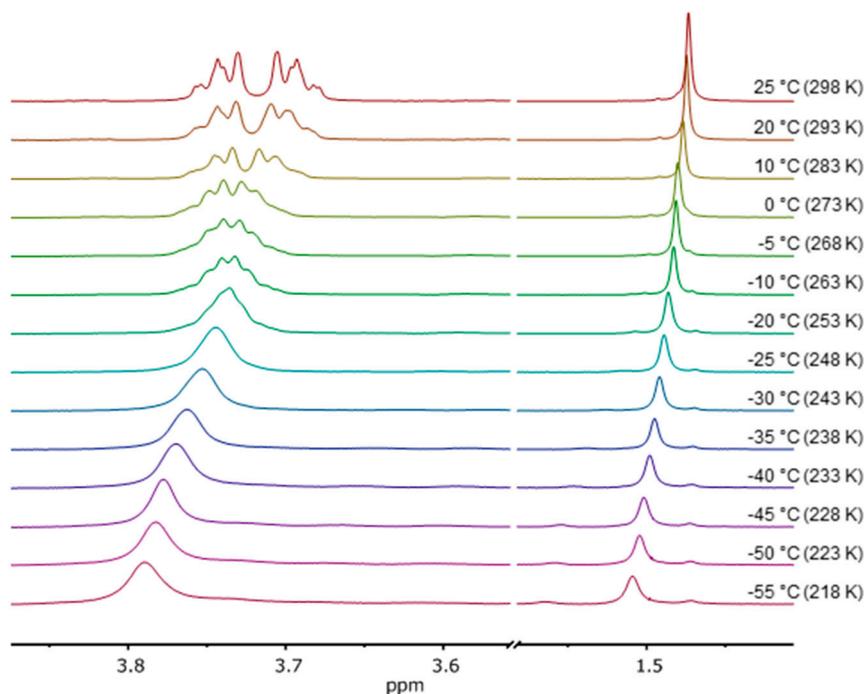


Figure S3. Partial ^1H NMR spectra (400 MHz, CDCl_3) of $[\text{K}^+\cdot o\text{-Me}_2\text{-1.1.1}]\text{BArF}^-$.

Titration Plots

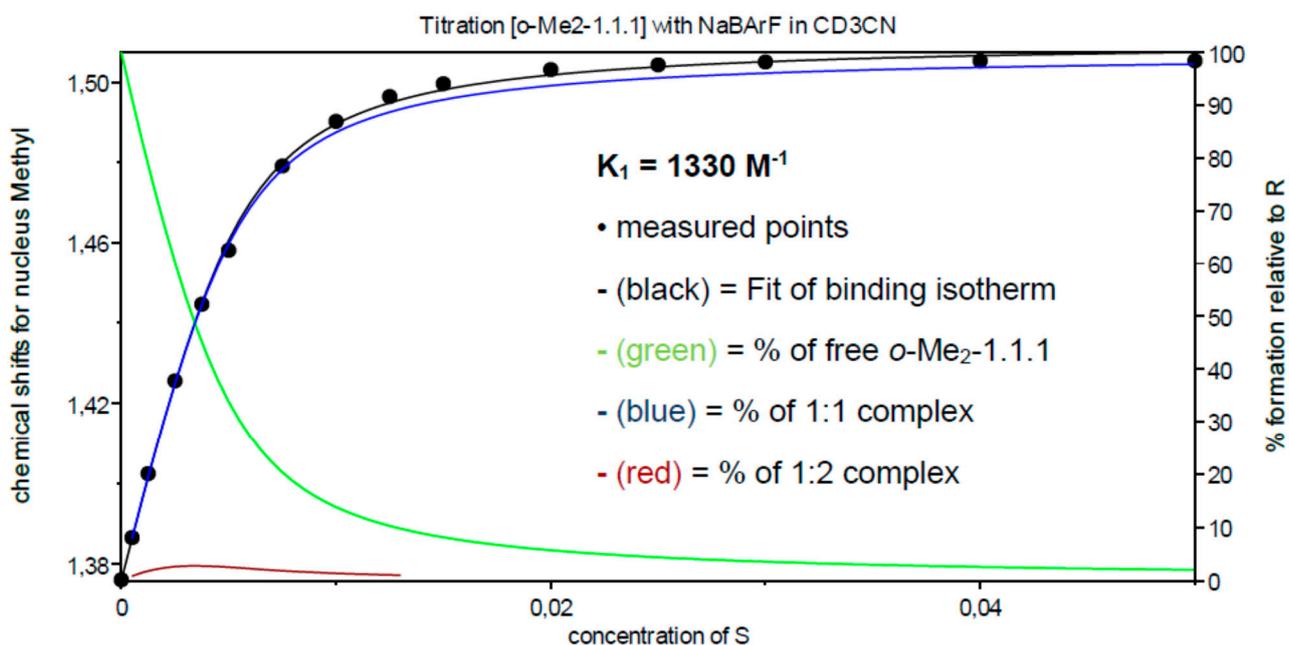


Figure S4. Binding isotherm with plot of different species for titration of $o\text{-Me}_2\text{-1.1.1}$ (5 mM) with NaBArF from 0 to 1000 mol % in CD_3CN (below 1% HypNMR does not plot species).

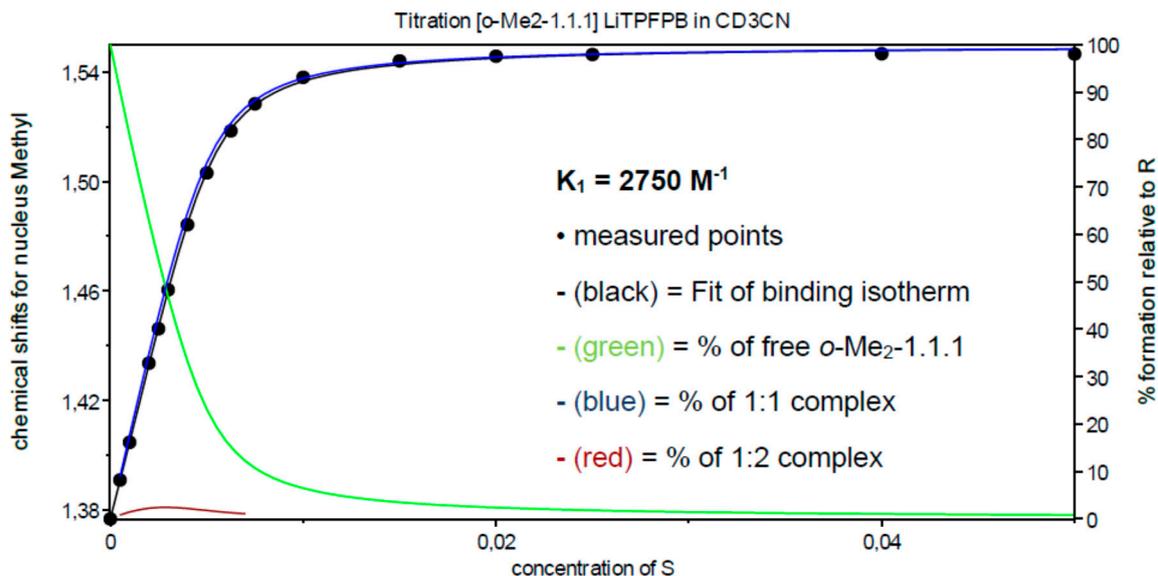


Figure S5. Binding isotherm with plot of different species for titration of *o*-Me₂-1.1.1 (5 mM) with LiTPFPB from 0 to 1000 mol % in CD₃CN (below 1% HypNMR does not plot species).

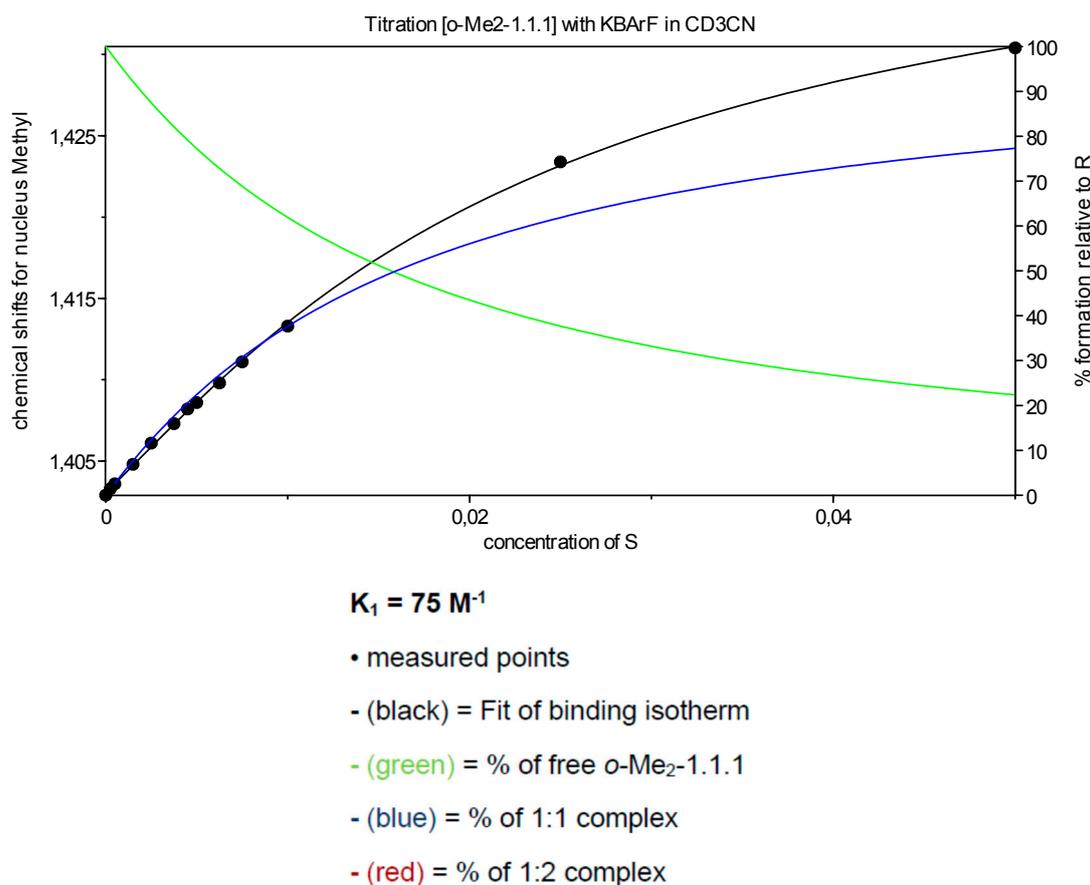


Figure S6. Binding isotherm with plot of different species for titration of *o*-Me₂-1.1.1 (5 mM) with KBArF from 0 to 1000 mol % in CD₃CN (below 1% HypNMR does not plot species, which is why there is no red line shown).

Titration of *o*-Me₂-1.1.1 (5mM) with NaBF₄

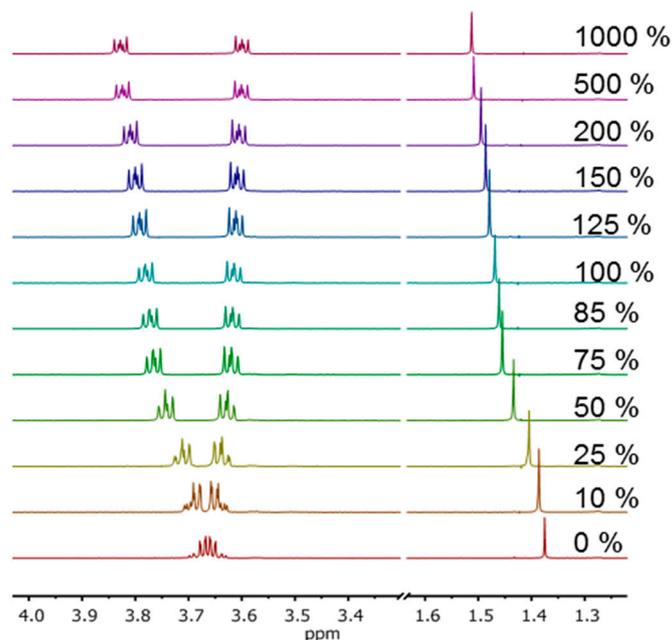


Figure S7. Partial ¹H NMR (400 MHz, 298 K, CD₃CN) stack plot of *o*-Me₂-1.1.1 (5 mM) titration with NaBF₄ from 0 to 1000 mol %.

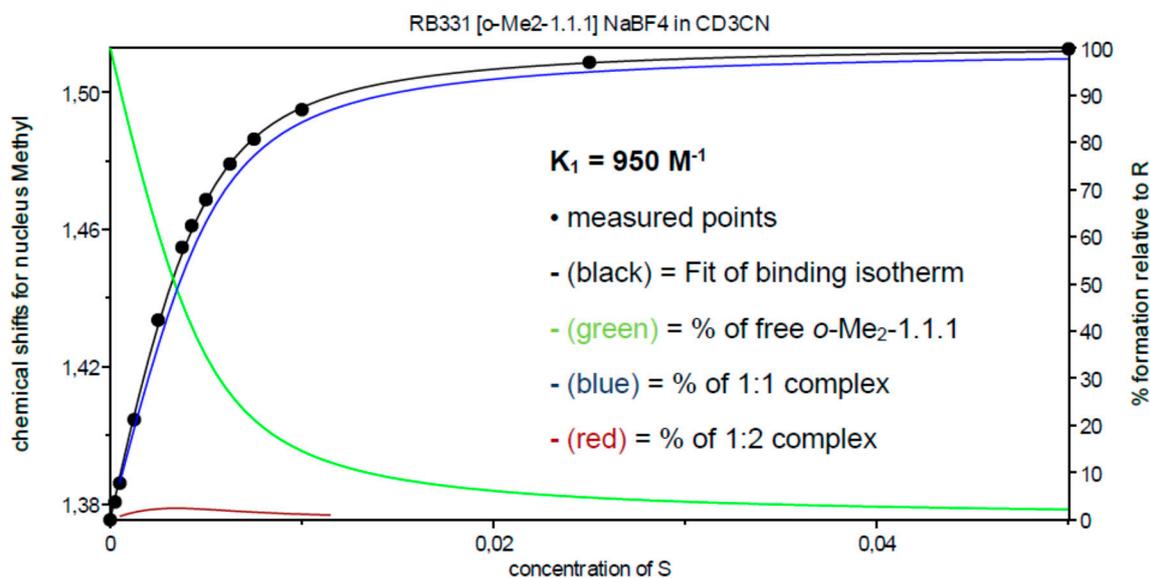


Figure S8. Binding isotherm with plot of different species for titration of *o*-Me₂-1.1.1 (5 mM) with NaBF₄ from 0 to 1000 mol % in CD₃CN (below 1% HypNMR does not plot species).

Titration of *o*-Me₂-1.1.1 (5mM) with LiBF₄

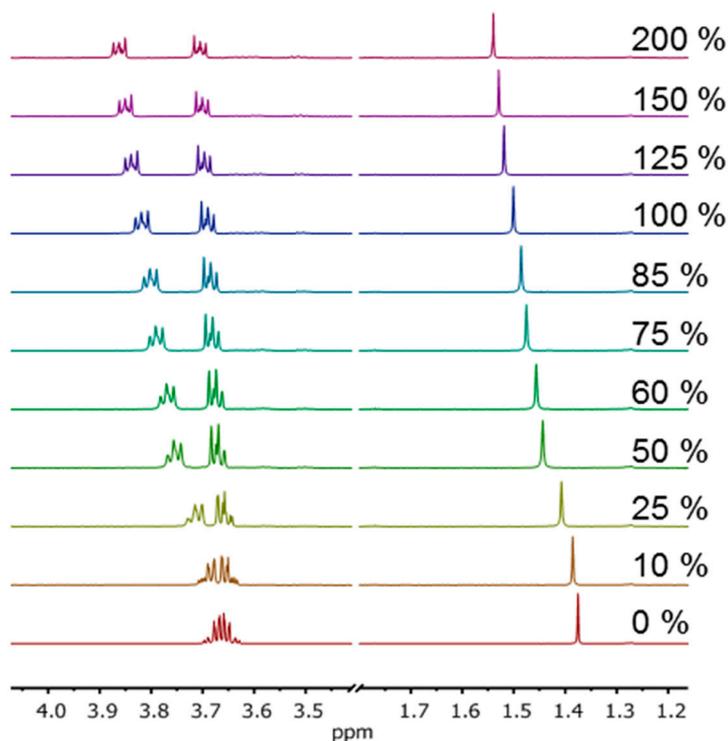


Figure S9. Partial ¹H NMR (400 MHz, 298 K, CD₃CN) stack plot of *o*-Me₂-1.1.1 (5 mM) titration with LiBF₄ from 0 to 200 mol %. (After addition of 200 mol % salt, noticeable hydrolysis of the cage occurred (most likely LiBF₄ is the most acidic of the salts used), which is why the fit of the binding isotherm was based only on these “early” titration points.)

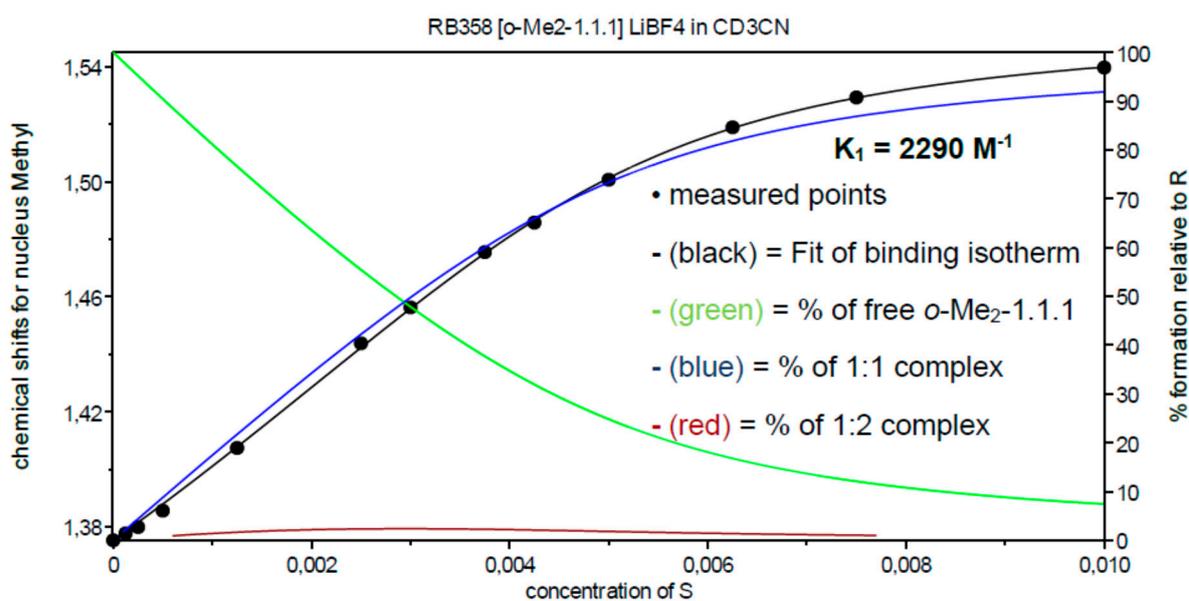


Figure S10. Binding isotherm with plot of different species for titration of *o*-Me₂-1.1.1 (5 mM) with LiBF₄ from 0 to 500 mol % in CD₃CN (below 1% HypNMR does not plot species).

Titration of *o*-Me₂-1.1.1 (5mM) with LiBArF

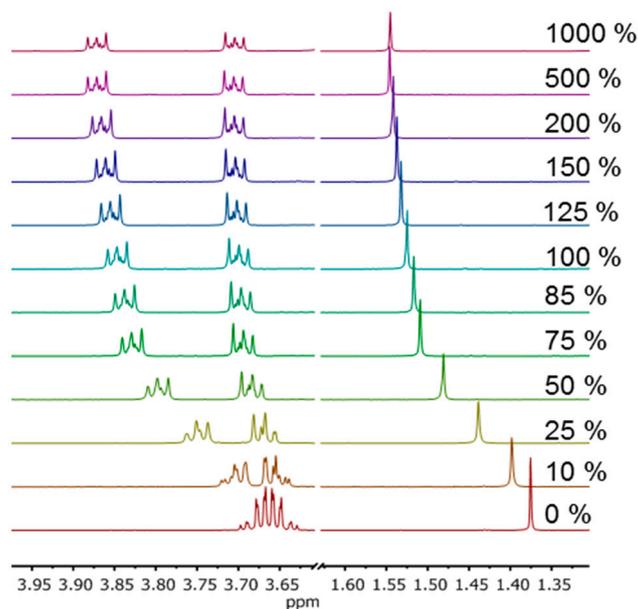


Figure S11. Partial ¹H NMR (400 MHz, 298 K, CD₃CN) stack plot of *o*-Me₂-1.1.1 (5 mM) titration with LiBArF from 0 to 1000 mol %.

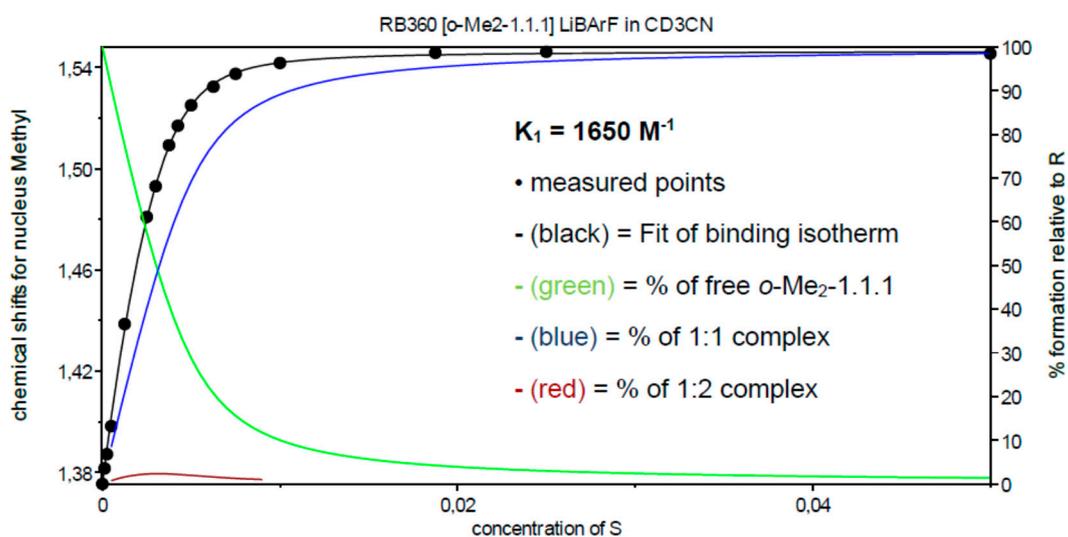


Figure S12. Binding isotherm with plot of different species for titration of *o*-Me₂-1.1.1 (5 mM) with LiBArF from 0 to 1000 mol % in CD₃CN (below 1% HypNMR does not plot species).