κ²- coordination of 18-crown-6 to Ce(III) cation: Solution dynamics and reactivity

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Experimental Procedures

**General Methods.** Unless otherwise indicated all reactions and manipulations were performed under an inert atmosphere (N\textsubscript{2}) using standard Schlenk techniques or in a Vacuum Atmospheres, Inc. Nexus II drybox equipped with a molecular sieves 13X / Q5 Cu-0226S catalyst purifier system. Glassware was oven-dried overnight at 150 °C prior to use. \textsuperscript{1}H, \textsuperscript{19}F, and \textsuperscript{13}C NMR spectra were obtained at room temperature on a Bruker DMX-300 Fourier transform NMR spectrometer operating at \textsuperscript{1}H frequency of 300 MHz. \textsuperscript{1}H and \textsuperscript{19}F variable temperature NMR measurements were carried out at 300 MHz and 282 MHz. \textsuperscript{1}H EXSY experiments were collected at room temperature on Bruker UNI-400 Fourier transform NMR spectrometer operating at \textsuperscript{1}H frequency of 400 MHz over a range of mix times (0, 10, 25, 50, 100, 200 ms). Chemical shifts were recorded in units of parts per million referenced against residual proteo solvent peaks (\textsuperscript{1}H) deteuro solvent peaks (\textsuperscript{13}C) or fluorobenzene (\textsuperscript{19}F, -113.15 ppm). Elemental analyses were performed at the University of California, Berkeley, Microanalytical Facility using a Perkin-Elmer Series II 2400 CHNS analyzer.

**Materials.** Tetrahydrofuran, diethyl ether, dichloromethane, fluorobenzene, hexanes, and \textit{n}-pentane were purchased from Fisher Scientific. The solvents were sparged for 20 min with dry N\textsubscript{2} and dried using a commercial two-column solvent purification system comprising columns packed with Q5 reactant and neutral alumina respectively (for hexanes and \textit{n}-pentane), or two columns of neutral alumina (for THF, Et\textsubscript{2}O and toluene). Deuterated solvents were purchased from Cambridge Isotope Laboratories, Inc.. Prior to use, pyridine-\textit{d}\textsubscript{5} and C\textsubscript{6}D\textsubscript{6} were stored overnight over molecular sieves and potassium mirror, respectively. 4,4\textquotesingle-di-\textit{t}ert-butyl-2,2\textquotesingle-dipyridyl was purchased from Sigma-Aldrich and used as received. Ce[N(SiMe\textsubscript{3})\textsubscript{2}]\textsubscript{3} and HN(SiMe\textsubscript{3})PhF\textsuperscript{2} were prepared according to reported procedures.
**X-Ray Crystallography.** X-ray reflection intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å) at a temperature of 143(1) K. In all cases, rotation frames were integrated using SAINT,\(^3\) producing a listing of unaveraged F\(^2\) and σ(F\(^2\)) values which were then passed to the SHELXTL\(^4\) program package for further processing and structure solution on a Dell Pentium 4 computer. The intensity data were corrected for Lorentz and polarization effects and for absorption using TWINABS\(^5\) or SADABS.\(^6\) The structures were solved by direct methods (SHELXS-97).\(^7\) Refinement was by full-matrix least squares based on F\(^2\) using SHELXL-97.\(^7\) All reflections were used during refinements. The weighting scheme used was \(w = 1/[σ^2(F_o^2) + (0.0907P)^2 + 0.3133P]\) where \(P = (F_o^2 + 2F_c^2)/3\). Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model.
Synthetic Details and Characterization

**Synthesis of Ce$^{III}$[N(SiMe$_3$)PhF]$_3$ (1).** To a vial containing Ce[N(SiMe$_3$)$_2$]$_3$ (1.24 g, 2.00 mmol, 1.00 equiv) dissolved in 10 mL pentane, a pentane solution containing HN(SiMe$_3$)PhF (1.690 g, 6.600 mmol, 3.30 equiv) was added. White solids gradually formed after stirring this mixture for 6 d. The precipitates were collected by filtration over a medium porosity fritted filter, washed with 3 × 3 mL pentane and dried under reduced pressure for 1 h. Yield: 1.49 g, 1.65 mmol, 83%.

$^1$H NMR (C$_6$D$_6$): δ –9.15 (s, 36H, –SiMe$_3$).

$^{19}$F NMR (C$_6$D$_6$): δ –163.47 (d, 6F, m-F, J = 23 Hz), –173.09 (br, 6F, o-F, FWHM 350 Hz), –173.42 (t, 3F, p-F, J = 23 Hz). Elemental analysis found (calculated) for C$_{27}$H$_{27}$F$_{15}$N$_3$Si$_3$Ce: C, 35.65 (35.92), H, 2.95 (3.01), N, 4.63 (4.65). Single crystals suitable for X-ray analysis were obtained by storing a saturated pentane solution at –21 °C overnight.

**Synthesis of Ce(k$^2$-18-crown-6)[N(SiMe$_3$)PhF]$_3$ (2).** To a vial containing Ce[N(SiMe$_3$)PhF]$_3$ (0.045 g, 0.050 mmol, 1.00 equiv) suspended in 5 mL pentane, a 1 mL pentane solution containing 18-crown-6 (0.013 g, 0.050 mmol, 1.00 equiv) was added. After stirring for 0.5 h, the mixture was filtered through Celite packed in a pipette, concentrated to 1 mL and stored at –21 °C overnight to yield colorless crystals. The crystals were collected by a filtration over a medium porosity fritted filter and dried under reduced pressure. Crystalline yield: 0.040 g, 0.034 mmol, 69 %.

$^1$H NMR (tol-$d_8$): –1.30 (br, 24H, 18-crown-6), –4.22 (s, 27H, -SiMe$_3$). $^{19}$F NMR (tol-$d_8$): –151.80 (br, 6F, o-F, FWHM 105 Hz), –163.76 (d, 6F, m-F, J = 17 Hz), –168.42 (t, 3F, p-F, J = 20 Hz). Elemental analysis found (calculated) for C$_{39}$H$_{51}$F$_{15}$N$_3$Si$_3$O$_6$Ce: C, 39.80 (40.13), H, 4.24 (4.40), N, 3.57 (3.60). Single crystals suitable for X-ray analysis were obtained by storing a pentane solution at –21°C overnight. The same reaction performed on NMR scale (i.e. 0.014 g 1)
in toluene or Et$_2$O with addition of 18-crown-6 in corresponding solvent dropwise (1 ml in ~30 s) gave near quantitative conversion to 2 by NMR spectroscopy after stirring for 0.5 h.

**Synthesis of {Ce($\kappa^6$-18-crown-6)[N(SiMe$_3$)PhF$_3$]$_2$}$^+${Ce[N(SiMe$_3$)PhF]$_4$}$^-$ (3).** To a vial containing Ce[N(SiMe$_3$)PhF]$_3$ (0.27 g, 0.30 mmol, 2.0 equiv) dissolved in 2 mL THF, a THF solution containing 18-crown-6 (0.040 g, 0.15 mmol, 1.0 equiv) was added. After stirring for 3 h, the conversion to 3 was near quantitative, as indicated by NMR spectroscopy (Figure S11-S12).

The volatiles were removed under reduced pressure. The solid residue was triturated with pentane and collected by filtration over a medium porosity fritted filter. The white solids were further washed with 3 × 3 mL pentane and dried under reduced pressure for 1 h. Yield: 0.30 g, 0.15 mmol, 97%. $^1$H NMR (thf-$d_8$): $\delta$ 5.92 (s, 18H, -SiMe$_3$cation), –3.54 (s, 24H, 18-crown-6), –5.32 (s, 36H, -SiMe$_3$anion). $^{19}$F NMR (thf): $\delta$ –141.08 (d, 4F, o-Fcation, $J$ = 2 Hz), –166.11 (t, 4F, m-Fcation, $J$ = 20 Hz), –167.06 (br, 8F, o-Fanion), –166.90 (d, 8F, m-Fanion, $J$ = 23 Hz), –167.77 (t, 2F, p-Fcation, $J$ = 23 Hz), –175.21 (t, 4F, p-Fanion, $J$ = 20 Hz). Elemental analysis found (calculated) for C$_{66}$H$_{78}$F$_{30}$N$_6$Si$_6$O$_6$Ce$_2$: C, 38.13 (38.29), H, 3.83 (3.80), N, 4.09 (4.06). Single crystals suitable for X-ray analysis were obtained by THF/pentane layering stored at –21 °C. The same reaction performed on NMR scale in dimethoxyethane, dichloromethane or pyridine similarly gave near quantitative conversion to 3.

**Transformation of 2 to 3**: Ce($\kappa^2$-18-crown-6)[N(SiMe$_3$)PhF$_3$]$_3$ was generated quantitatively *in situ* by adding 18-crown-6 (0.006 g, 0.022 mmol, 1.10 equiv) into an Et$_2$O solution containing Ce[N(SiMe$_3$)PhF]$_3$ (0.018 g, 0.020 mmol, 1.00 equiv) following stirring for 1 h. The solvent was removed under reduced pressure and CH$_2$Cl$_2$ was added into the mixture. $^{19}$F and $^1$H NMR measurements confirmed its near quantitative conversion to {Ce($\kappa^6$-18-crown-}
6)[N(SiMe₃)Ph₂]₂⁺ {Ce[N(SiMe₃)Ph]₄}⁻. The same reaction performed on NMR scale in tetrahydrofuran, dimethoxyethane, or pyridine also gave 3 in near quantitative yield. Representative NMR was shown in Figure S13-S14.

**Synthesis of Ce(′Bu₂bipy)[N(SiMe₃)Ph]₃ (4).** To a vial containing Ce[N(SiMe₃)Ph]₃ (0.045 g, 0.050 mmol, 1.00 equiv) dissolved in suspended in 2 mL pentane, a 1 mL pentane solution containing 4,4′-di-tert-butyl-2,2′-dipyridyl (0.013 g, 0.050 mmol, 1.00 equiv) was added resulting in an orange clear solution. After stirring for 0.5 h, the solution was concentrated to 1 mL and stored at −21 °C overnight to yield orange crystals. The orange crystals were collected by filtration over a medium porosity fritted filter and dried under reduced pressure. Crystalline yield: 0.050 g, 0.043 mmol, 86 %. ¹H NMR (C₆D₆): 19.50 (br, 2H, py-H), 7.83 (s, 2H, py-H), 3.68 (s, 2H, py-H), 0.20 (s, 18H, -t-Bu), −3.69 (s, 27H, -SiMe₃). ¹⁹F NMR (tol-d₈): −162.55 (br, 6F, o-F, FWHM 130 Hz), −166.04 (d, 6F, m-F, J = 23 Hz), −171.50 (t, 3F, p-F, J = 23 Hz). Elemental analysis found (calculated) for C₄₅H₅₁F₁₅N₅Si₃Ce: C, 46.07 (46.15), H, 4.21 (4.39), N, 5.77 (5.98). Single crystals suitable for X-ray analysis were obtained by storing a pentane solution of 4 at −21°C.

**Reaction of Ce(κ²-18-crown-6)[N(SiMe₃)Ph]₃ with 4,4′-di-tert-butyl-2,2′-dipyridyl:** Ce(κ²-18-crown-6)[N(SiMe₃)Ph]₃ was generated quantitatively in situ by adding 18-crown-6 (0.003 g, 0.011 mmol, 1.10 equiv) into a C₆D₆ solution containing Ce[N(SiMe₃)Ph]₃ (0.009 g, 0.010 mmol, 1.00 equiv). After stirring for 1 h, white solids of 4,4′-di-tert-butyl-2,2′-dipyridyl (0.003 g, 0.011 mmol, 1.10 equiv) were added to the mixture causing an immediate color change to orange, followed by stirring for 3 h. The quantitative conversion into Ce(′Bu₂bipy)[N(SiMe₃)Ph]₃ was confirmed by NMR (Figure S15-16).
Synthesis of KN(SiMe₃)PhF. To a 20 mL colorless ethyl ether solution containing HN(SiMe₃)PhF (2.530 g, 9.9 mmol, 1.00 equiv) in a 150 mL flask, a ethyl ether solution containing KN(SiMe₃)₂ (1.970 g, 9.9 mmol, 1.00 equiv) was added. The mixture turned slightly yellow and was stirred for 4 h and dried under vacuum. The resulting white solid were collected by filtration over medium porosity fritted filter and washed with 5 mL pentane three times. Drying for 3 h under reduced pressure yielded white powder identified by ¹H NMR as KN(SiMe₃)PhF without solvation. Yield: 2.750 g, 9.4 mmol, 95 %. ¹H NMR (pyr-d₅): δ 0.49 (s, 9H, -SiMe₃). ¹⁹F NMR (pyr-d₅): δ −164.67 (t, 2F, o-F, J = 17 Hz), −171.66 (t, 2F, m-F, J = 23 Hz), −195.08 (m, 2F, p-F). Elemental analysis found (calculated) for C₉H₉F₅NSiK: C, 36.82 (36.85), H, 3.12 (3.09), N, 5.00 (4.77). Single crystals of KN(SiMe₃)PhF-Et₂O suitable for X-ray analysis were obtained from Et₂O/pentane.

Reaction of Ce(κ²-18-crown-6)[N(SiMe₃)PhF]₃ with KN(SiMe₃)PhF: Ce(κ²-18-crown-6)[N(SiMe₃)PhF]₃ was generated quantitatively in situ by adding a Et₂O solution of 18-crown-6 (0.007 g, 0.026 mmol, 1.00 equiv) into a Et₂O solution containing Ce[N(SiMe₃)PhF]₃ (0.023 g, 0.026 mmol, 1.00 equiv). After stirring for 1 h, a Et₂O solution containing KN(SiMe₃)PhF (0.008 g, 0.026 mmol, 1.00 equiv) was added to the mixture and further stirred for 3 h. NMR measurement confirmed the nearly quantitative conversion into {Ce[N(SiMe₃)PhF]₄}⁻ (3-anion).
**Figure S1.** Thermal ellipsoid plot of Ce$^{III}$[N(SiMe$_3$)Ph]$^f_3$ (1) at 30% probability. Selected bond length (Å) and angles (deg): Ce(1)–N(1) 2.416(2), Ce(1)–N(2) 2.381(2), Ce(1)–N(3) 2.394(2), Ce(1)–F(1) 2.6140(16), Ce(1)–F(2) 2.6185(16), Ce(1)–F(3) 2.6418(16); N(1)–Ce(1)–N(2) 119.97(8), N(1)–Ce(1)–N(3) 128.92(8), N(2)–Ce(1)–N(3) 111.11(8).
Figure S2. Thermal ellipsoid plot of Ce$^{III}$($κ^2$-18-crown-6)[N(SiMe$_3$)Ph$^F$]$_3$ (2) at 30% probability.

Selected bond length (Å) and angles (deg): Ce(1)–N(1) 2.4595(17), Ce(1)–N(2) 2.3785(17), Ce(1)–N(3) 2.4057(17), Ce(1)–F(1) 2.7545(12), Ce(1)–O(1) 2.5201(14), Ce(1)–O(2) 2.6050(15); N(1)–Ce(1)–N(2) 92.15(6), N(1)–Ce(1)–N(3) 123.64(6), N(2)–Ce(1)–N(3) 113.57(6), O(1)–Ce(1)–O(2) 64.63(4), F(1)–Ce(1)–N(1) 62.84(5).
Figure S3. Thermal ellipsoid plot of \{Ce(κ^6-18-crown-6)[N(SiMe_3)PH]^2\} Ce[N(SiMe_3)PH]^2\}^- (3) at 30% probability (Left: Ce^{III}[N(SiMe_3)PH]^2, right: Ce(κ^6-18-crown-6)[N(SiMe_3)PH]^2; interstitial THF molecule is omitted for clarity). Selected bond length (Å) and angles (deg): Ce(1)–N(1) 2.409(3), Ce(1)–N(2) 2.489(3), Ce(1)–N(3) 2.527(3), Ce(1)–N(4) 2.402(3), Ce(2)–N(5) 2.472(3), Ce(2)–N(6) 2.454(3), Ce(1)–F(1) 2.678(2), Ce(1)–F(2) 2.681(2), Ce(2)–Oave 2.614(3); N(1)–Ce(1)–N(2) 86.51(10), N(1)–Ce(1)–N(3) 123.42(10), N(1)–Ce(1)–N(4) 116.99(10), N(2)–Ce(1)–N(4) 118.43(10), N(3)–Ce(1)–N(4) 89.89(10), N(3)–Ce(1)–F(1) 63.33(8), N(2)–Ce(1)–F(2) 63.87(8), N(5)–Ce(2)–N(6) 168.31(11).
Figure S4. Thermal ellipsoid plot of Ce$^{III}$(Bu$_2$bipy)[N(SiMe$_3$)Ph]$^-$$_3$ (4) at 30% probability. Selected bond length (Å) and angles (deg): Ce(1)–N(1) 2.491(6), Ce(1)–N(2) 2.407(7), Ce(1)–N(3) 2.402(6), Ce(1)–N(4) 2.662(6), Ce(1)–N(5) 2.613(7), Ce(1)–F(1) 2.673(4); N(1)–Ce(1)–N(2) 102.8(2), N(1)–Ce(1)–N(3) 130.5(2), N(2)–Ce(1)–N(3) 110.7(2).
Figure S5. Thermal ellipsoid plot of KN(SiMe$_3$)Ph$^+$·Et$_2$O at 30% probability.
VT NMR Spectra

Figure S6. $^1$H VT NMR data for $\text{Ce}^{\text{III}}[\text{N(SiMe}_3\text{)}\text{Ph}^\text{R}]_3$ (1) in toluene-$d_8$ between 200–300 K. Peaks downfield of -5 ppm were attributed solely to solvent impurities, so this region of the spectrum was omitted for clarity.
Figure S7. $^{19}$F VT NMR data for $\text{Ce}^{\text{III}}[\text{N(SiMe$_3$)}\text{Ph}^F]_3$ (1) in toluene-$d_8$ between 200–300 K. Minor impurity HN(SiMe$_3$)Ph$^F$ is visible as a minor impurity in some spectra, indicated by *.
Figure S8. $^1$H VT NMR data for Ce$^{	ext{III}}$(κ$^2$-18-crown-6)[N(SiMe$_3$)Ph]$^+$$_3$ (2) in toluene-$d_8$ between 200–300 K. Resonances downfield of 0 ppm can be solely attributed to solvent residue (pentane and toluene), a minor amount of HN(SiMe$_3$)Ph$^+$ and free 18-crown-6.
Figure S9. $^{19}$F VT NMR data for Ce$^{III}$(κ$^2$-18-crown-6)[N(SiMe$_3$)PhF]$_3$ (2) in toluene-$d_8$ between 200–300 K. Minor impurity HN(SiMe$_3$)PhF is visible in some spectra, indicated by *.
NMR Spectra

Figure S10. $^1$H NMR of reaction aliquots of Ce[N(SiMe$_3$)PhF$_3$]$_3$ (1) with 18-crown-6 in thf-h$_8$. Quantitative conversion to {$\text{Ce}^{\text{III}}(\kappa^6\text{-18-crown-6})\text{[N(SiMe$_3$)PhF]}_2$}$^+$ {$\text{Ce}^{\text{III}}\text{[N(SiMe$_3$)PhF]}_4$}$^-$ (3) was observed. Proteo-solvent (thf) resonances are indicated by *.

Figure S11. $^{19}$F NMR of reaction aliquots of Ce[N(SiMe$_3$)PhF$_3$]$_3$ (1) with 18-crown-6 in thf-h$_8$. Quantitative conversion to {$\text{Ce}^{\text{III}}(\kappa^6\text{-18-crown-6})\text{[N(SiMe$_3$)PhF]}_2$}$^+$ {$\text{Ce}^{\text{III}}\text{[N(SiMe$_3$)PhF]}_4$}$^-$ (3) was observed. Minor impurity of HN(SiMe$_3$)PhF$^-$ was also noted in the spectrum, indicated by *. 
Figure S12. $^{19}$F NMR of reaction aliquots of Ce($\kappa^2$-18-crown-6)[N(SiMe$_3$)Ph$^F$]$_3$ (2) in CH$_2$Cl$_2$. Near quantitative conversion to \{Ce$^{III}$(\(\kappa^6$-18-crown-6)[N(SiMe$_3$)Ph$^F$])$_2$\}$^+$ \{Ce$^{III}$[N(SiMe$_3$)Ph$^F$]$_4$\}$^-$ (3) was observed. A minor impurity of HN(SiMe$_3$)Ph$^F$ was also noted in the spectrum, indicated by *.

Figure S13. $^1$H NMR of reaction aliquots of Ce($\kappa^2$-18-crown-6)[N(SiMe$_3$)Ph$^F$]$_3$ (2) in CH$_2$Cl$_2$. Near quantitative conversion to \{Ce$^{III}$(\(\kappa^6$-18-crown-6)[N(SiMe$_3$)Ph$^F$])$_2$\}$^+$ \{Ce$^{III}$[N(SiMe$_3$)Ph$^F$]$_4$\}$^-$ (3) and free 18-crown-6 was observed. Minor amount of HN(SiMe$_3$)Ph$^F$ (*), residue solvent Et$_2$O (\(\Delta\)) were also noted in the spectrum.
Figure S14. $^1$H NMR of reaction aliquots of Ce($\kappa^2$-18-crown-6)[N(SiMe$_3$)PhF]$_3$ (2) with 4,4’-di-tert-butyl-2,2’-dipyridyl. NMR obtained in C$_6$D$_6$ shows near quantitative conversion to Ce$^{III}$(Bu$_2$bipy)[N(SiMe$_3$)PhF]$_3$ (4), excessive 4,4’-di-tert-butyl-2,2’-dipyridyl (indicated by ●), byproduct 18-crown-6 (indicated by ▲) and a minor impurity HN(SiMe$_3$)PhF (●) is also evident in the spectra.

Figure S15. $^{19}$F NMR of reaction aliquots of Ce($\kappa^2$-18-crown-6)[N(SiMe$_3$)PhF]$_3$ (2) with 4,4’-di-tert-butyl-2,2’-dipyridyl. NMR obtained in C$_6$D$_6$ shows near quantitative conversion to Ce$^{III}$(Bu$_2$bipy)[N(SiMe$_3$)PhF]$_3$ (4).
Figure S16. $^1$H NMR of KN(SiMe$_3$)Ph$^+$ in pyr-$d_5$. The lack of Et$_2$O resonances suggests the compound is obtained with no solvation.

Figure S17. $^{19}$F NMR of KN(SiMe$_3$)Ph$^+$ in pyr-$d_5$. 
$^1$H EXSY Spectra

Figure S18. $^1$H EXSY experiment of Ce($\kappa^2$-18-crown-6)[N(SiMe$_3$)Ph$_2$]$_3$ (2) with free 18-crown-6 in $C_6D_6$ (each with a concentration of $1.5 \times 10^{-2}$ mol•L$^{-1}$). The spectrum was collected with a mix time of 10 ms. The presence of off-diagonal peaks suggests a facile exchange between free and bound 18-crown-6 molecules. Note: Trace amounts of compound 3 are formed during the course of data collection (noted by ~).
Figure S19. $^1$H EXSY experiment of $\{\text{Ce}(\kappa^6-18\text{-crown-6})[\text{N(SiMe}_3\text{)Ph}^\text{F}]_2\}^+ \{\text{Ce}[\text{N(SiMe}_3\text{)Ph}^\text{F}]_4\}^-$ (3) with free 18-crown-6 in pyr-$d_5$ (each with a concentration of $1.5\times10^{-2}$ mol$\cdot$L$^{-1}$). The spectrum was collected with mix time of 10 ms. Resonances are assigned in 1D spectrum. No off-diagonal peaks were observed at any of the mix times measured (0, 10, 25, 50 100, 200 ms), indicating no exchange occurring between the rigidly bound $\kappa^6$-18-crown-6 and free 18-crown-6.
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