Redox Transmetallation Approaches to the Synthesis of Extremely Bulky Amido-Lanthanoid(II) and Calcium(II) Complexes

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SUPPORTING INFORMATION

(5 Pages)

Table S1. Summary of Crystallographic Data for **1-3**, **5**, **6**, **8**, **9**, $^{Ph}L^{\dagger}H$ **1S**, and [{Yb(thf)$_2$(μ-Br)$_2$}$_\infty$] **2S**.

	1·(benzene)$_{0.5}$	**2**·(benzene)$_{0.5}$	**3**·(thf)$_2$	**5**	**6**
empirical formula	$C_{64}H_{64}CaNO_2Si$	$C_{64}H_{64}EuNO_2Si$	$C_{65}H_{69}NO_3SiYb$	$C_{88}H_{100}B_2N_6Si_2Yb_2$	$C_{64}H_{64}N_2OYb$
formula weight	947.33	1059.21	1113.34	1665.62	834.03
crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
space group	$P2_1/n$	$P2_1/n$	$P2_1/n$	$P2_1/c$	$C2/c$
a (Å)	10.389(2)	10.421(2)	10.4277(4)	12.009(2)	16.6188(8)
b (Å)	21.553(4)	21.831(4)	21.9902(12)	14.686(3)	11.5082(6)
c (Å)	22.714(5)	22.608(5)	22.7904(7)	21.476(4)	21.4270(10)
α (deg.)	90	90	90	90	90
β (deg)	92.37(3)	93.42(3)	92.113(3)	95.80(3)	94.766(5)
γ (deg.)	90	90	90	90	90
vol (Å^3)	5081.6(18)	5134.2(18)	5222.4(4)	3768.2(13)	4083.8(3)
Z	4	4	4	2	4
ρ (calcd) (g.cm^{-3})	1.238	1.370	1.416	1.468	1.357
μ (mm^{-1})	0.194	1.291	1.863	2.549	2.325
F(000)	2020	2192	2296	1696	1728
T (K)	100(2)	123(2)	123(2)	100(2)	100(2)
reflections collected	45609	89878	36755	27642	20074
unique reflections	12086	12242	10281	7407	3884
R_{int}	0.0600	0.0791	0.0603	0.0552	0.0391
R1 indices [$I>2\sigma(I)$]	0.0515	0.0532	0.0442	0.0314	0.0245
wR2 indices (all data)	0.1354	0.1441	0.1098	0.0792	0.0554
Largest peak and hole (e/Å^3)	0.729, -0.386	1.545, -1.150	2.181, -1.215	0.833, -0.821	0.990, -0.448
CCDC No.	1845271	1845273	1845272	1845275	1845274

Table S1 (contd.). Summary of Crystallographic Data for **1-3**, **5**, **6**, **8**, **9**, $^{Ph}L^{\dagger}H$ **1S**, and $[\{Yb(thf)_2(\mu\text{-}Br)_2\}_\infty]$ **2S**.

	8	**9**	**1S**	**2S**
empirical formula	$C_{76}H_{80}N_2Si_2Yb$	$C_{84}H_{96}Eu_2I_2N_2O_2Si_2$	$C_{53}H_{47}NSi$	$C_8H_{16}Br_2O_2Yb$
formula weight	1250.64	1779.53	726.01	477.07
crystal system	Monoclinic	Triclinic	Triclinic	Orthorhombic
space group	$P2_1/n$	P-1	P-1	$Pnnm$
a (Å)	16.442(3)	9.7676(7)	11.0822(11)	7.6870(15)
b (Å)	21.321(4)	13.1133(10)	13.2591(12)	18.840(4)
c (Å)	19.126(4)	14.9720(12)	14.8802(14)	4.1890(8)
α (deg.)	90	90.368(4)	90.661(4)	90
β (deg)	113.47(3)	106.621(4)	106.424(4)	90
γ (deg.)	90	91.770(4)	104.870(4)	90
vol (Å^3)	6150(2)	1836.5(2)	2018.7(3)	606.7(2)
Z	4	1	2	2
ρ (calcd) (g.cm^{-3})	1.351	1.609	1.194	2.612
μ (mm^{-1})	1.605	2.614	0.096	14.264
F(000)	2592	890	772	440
T (K)	100(2)	123(2)	123(2)	100(2)
reflections collected	92209	59258	26419	4674
unique reflections	13249	7339	8045	732
R_{int}	0.1516	0.0829	0.0676	0.1546
R1 indices [$I>2\sigma(I)$]	0.0645	0.0321	0.0562	0.0388
wR2 indices (all data)	0.1581	0.0619	0.1417	0.0955
Largest peak and hole (e/Å^3)	2.712, -1.879	0.930, -0.790	0.385, -0.375	1.19, -1.65
CCDC No.	1845276	1845277	1845270	1845278

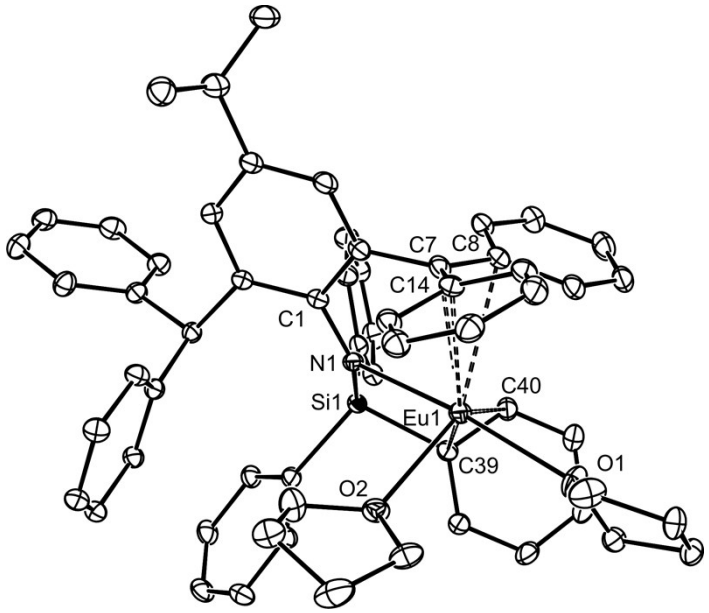

Figure S1. Thermal ellipsoid (25%) drawing of [Eu(^{Ph}L$^{\dagger-H}$)(thf)$_2$] **2**. Hydrogen atoms omitted. Selected bond distances (Å) and angles (°) can be found in Table 1 in the main text.

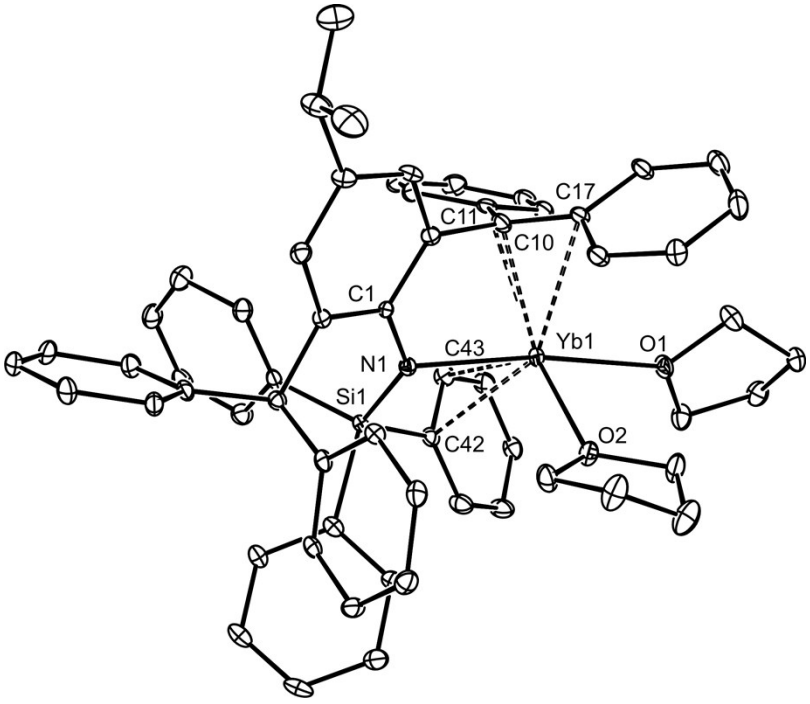

Figure S2. Thermal ellipsoid (25%) drawing of [Yb(^{Ph}L$^{\dagger-H}$)(thf)$_2$] **3**. Hydrogen atoms omitted. Selected bond distances (Å) and angles (°) can be found in Table 1 in the main text.

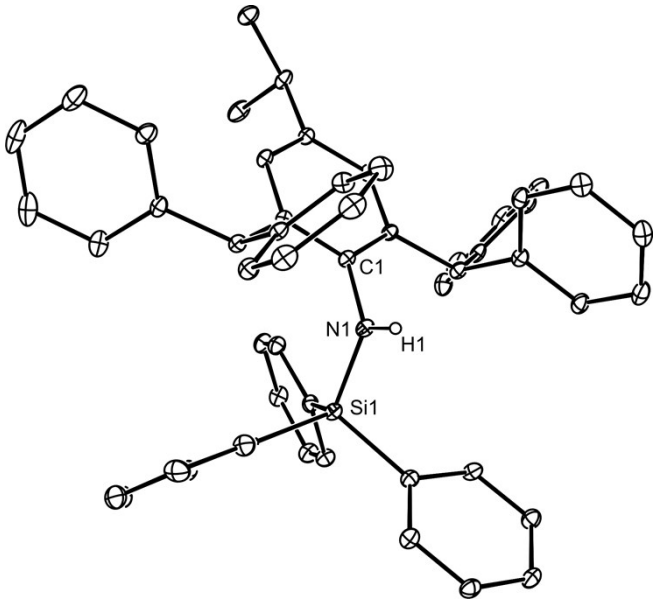

Figure S3. Thermal ellipsoid (25%) drawing of $^{Ph}L^{\dagger}H$ **1S**. Hydrogen atoms, except amine hydrogen, omitted. Selected bond distances (Å) and angles (°): Si(1)-N(1) 1.733(2), N(1)-C(1) 1.429(3), C(1)-N(1)-Si(1) 124.95(15).

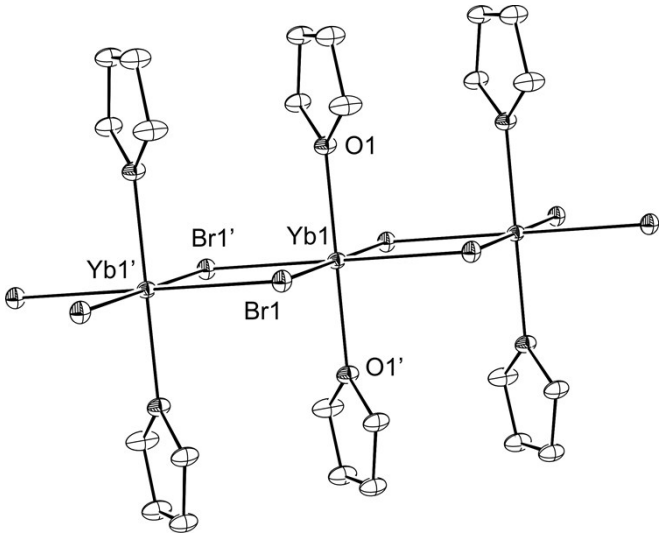

Figure S4. Thermal ellipsoid (25%) drawing of $[\{Yb(thf)_2(\mu\text{-}Br)_2\}_\infty]$ **2S**. Hydrogen atoms omitted. Selected bond distances (Å) and angles (°): Yb(1)-Br(1) 2.8875(7), Yb-O(1) 2.379(7), Yb(1)-Br(1)-Yb(1)' 93.00(3), Br(1)-Yb(1)-Br(1)' 87.00(3). Symmetry operation: ' x, y, -1+z.