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SUPPLEMENTARY MATERIAL

Extremely Bulky Amido and Amidinato Complexes of Boron and Aluminium Halides: Synthesis and Reduction Studies

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X-Ray Crystallography

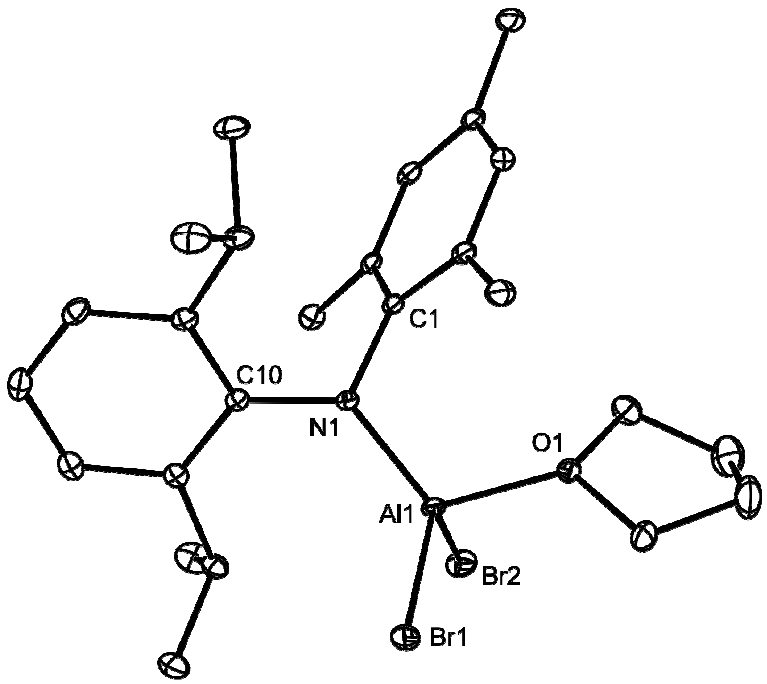

Fig. S1. ORTEP diagram of **7** (25% thermal ellipsoids; hydrogen atoms omitted). Selected bond

lengths (Å) and angles (º): Br(1)-Al(1) 2.2852(6), Br(2)-Al(1) 2.3262(6), Al(1)-N(1) 1.8199(16), Al(1)-O(1) 1.8648(15), N(1)-Al(1)-O(1) 111.30(7), Br(1)-Al(1)-Br(2) 114.55(3), C(10)-N(1)-C(1) 115.95(14).

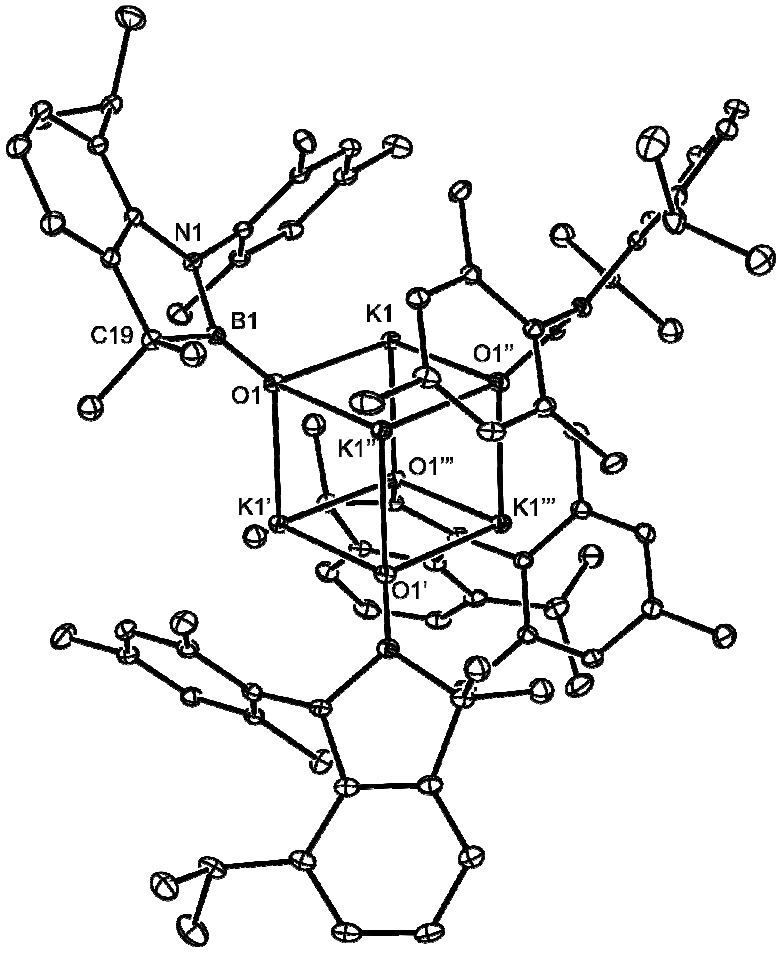

Fig. S2. ORTEP diagram of **9** (25% thermal ellipsoids; hydrogen atoms omitted). Selected bond lengths (Å) and angles (º): K(1)-O(1) 2.6778(18), O(1)-B(1) 1.316(3), K(1)'-O(1) 2.6273(16), K(1)"-O(1) 2.6828(18), N(1)-B(1) 1.493(3), B(1)-C(19) 1.614(4), N(1)-B(1)-C(19) 104.7(2). Symmetry operation: ' -y-1, x, -z-1, " -x-1, -y-1, z, ''' y, -x-1, -z-1.

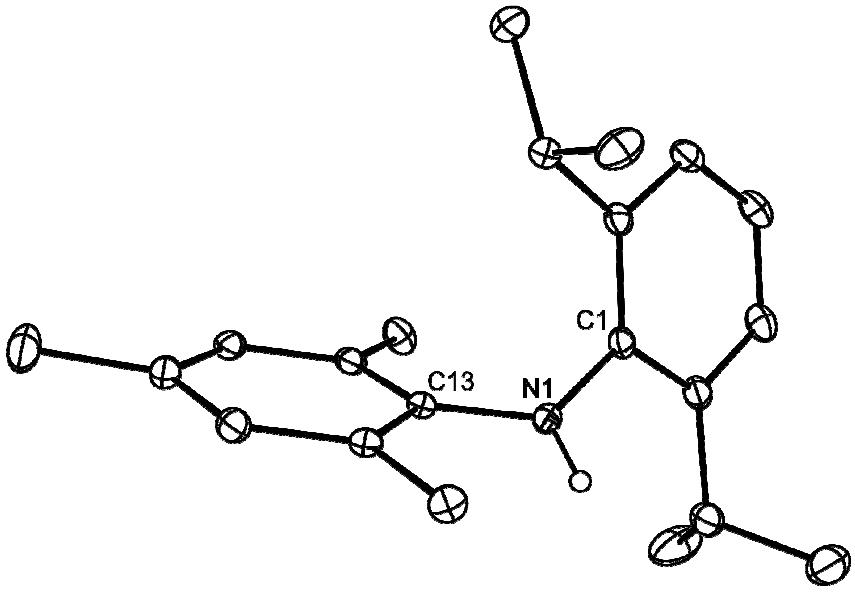

Fig. S3. ORTEP diagram of HL^{Mes} (25% thermal ellipsoids; hydrogen atoms, except amino proton, omitted). Selected bond lengths (Å) and angles (º): N(1)-C(13) 1.4089(15), N(1)-C(1) 1.4167(15), C(13)-N(1)-C(1) 127.41(10).

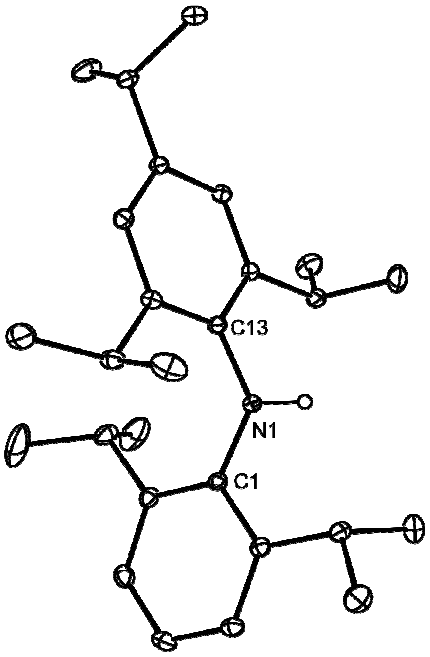

Fig. S4. ORTEP diagram of HL^{Trip} (25% thermal ellipsoids; hydrogen atoms, except amino proton, omitted). Selected bond lengths (Å) and angles (º): N(1)-C(1) 1.4072(14), N(1)-C(13) 1.4212(14), C(1)-N(1)-C(13) 131.72(9).

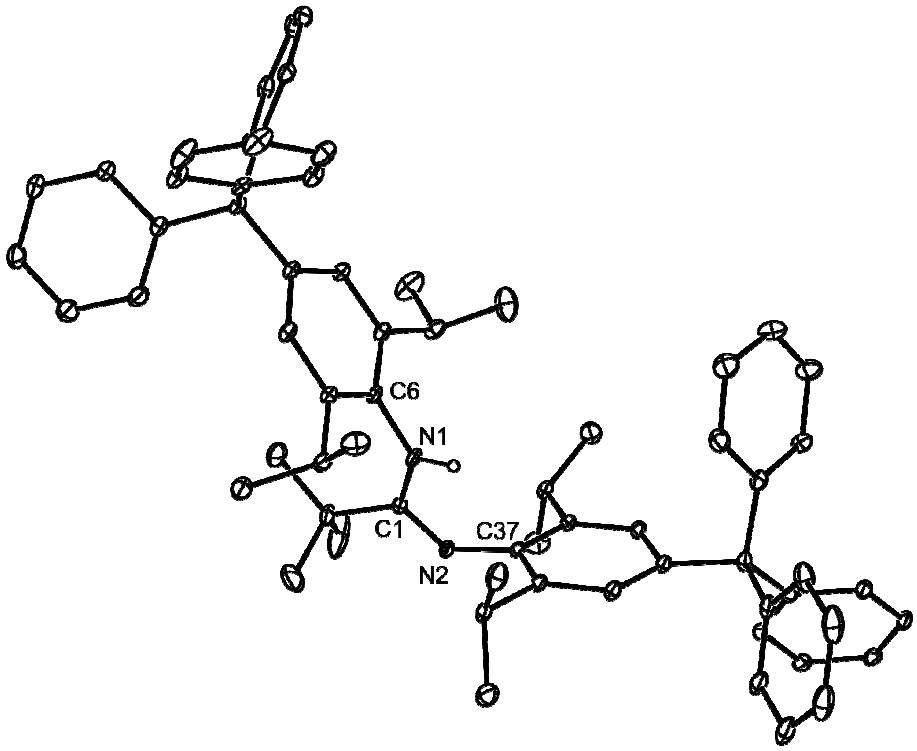

Fig. S5. ORTEP diagram of Piso"H (25% thermal ellipsoids; hydrogen atoms, except amino proton, omitted). Selected bond lengths (Å) and angles (º): N(1)-C(1) 1.363(2), N(2)-C(1) 1.295(2), N(2)-C(1)-N(1) 121.21(15), C(1)-N(1)-C(6) 129.73(15), C(1)-N(2)-C(37) 123.52(15).

Table S1. Summary of crystallographic data for **3-9**, **12**, HL^{Mes}, HL^{Trip}, Piso*H and Piso"H

	3	**4**	**5**	**6**	**7**	**8**	**9**	**12**.(hexane)
empirical formula	$C_{51}H_{60}KNSi$	$C_{44}H_{53}BBrNSi$	$C_{47.5}H_{56}AlBr_2NSi$	$C_{21}H_{28}BBr_2N$	$C_{25}H_{36}AlBr_2NO$	$C_{31}H_{48}AlBr_2NO$	$C_{84}H_{108}B_4K_4N_4O_4$	$C_{73}H_{85}AlI_2N_2$
formula weight	754.19	714.68	855.82	465.07	553.35	637.50	1437.38	1271.21
crystal system	monoclinic	triclinic	monoclinic	monoclinic	monoclinic	monoclinic	tetragonal	triclinic
space group	$P2_1/n$	P-1	$C2/c$	$P2_1/c$	$P2_1/n$	$P2_1/n$	$P\text{–}42_1c$	P-1
a (Å)	11.3213(4)	10.1023(4)	22.0631(10)	12.5739(12)	8.9492(2)	9.2127(4)	18.900(3)	12.967(3)
b (Å)	21.1256(7)	10.5999(4)	10.4993(3)	12.2680(7)	18.4716(5)	18.8947(9)	18.900(3)	16.068(3)
c (Å)	18.4141(6)	17.6169(7)	39.3147(18)	14.0054(8)	15.6478(4)	17.8733(8)	13.152(3)	16.695(3)
α (°)	90	91.561(3)	90	90	90	90	90	89.13(3)
β (°)	103.5510(10)	91.981(3)	107.445(5)	96.940(7)	99.0790(10)	91.2640(10)	90	84.59(3)
γ (°)	90	95.155(3)	90	90	90	90	90	70.51(3)
V (Å^3)	4281.5(2)	1876.85(13)	8688.3(6)	2144.6(3)	2554.27(11)	3110.5(2)	4698.3(13)	3264.0(11)
Z	4	2	8	4	4	4	2	2
T (K)	123(2)	123(2)	123(2)	123(2)	123(2)	123(2)	173(2)	100(2)
ρ_{calcd} (g·cm^3)	1.170	1.265	1.309	1.440	1.439	1.361	1.016	1.293
μ (mm^{-1})	0.187	1.159	1.947	3.783	3.225	2.658	0.233	1.018
F(000)	1624	756	3560	944	1136	1328	1536	1312
reflns collected	27934	12339	25593	8382	13889	17365	36083	27061
unique reflns	7531	7327	8522	4185	4675	6781	5223	10799
R_{int}	0.0242	0.0310	0.0380	0.0351	0.0182	0.0267	0.0876	0.0202
R1 indices [I > 2σ(I)]	0.0398	0.0449	0.0470	0.0425	0.0243	0.0298	0.0498	0.0475
wR2 indices (all data)	0.1025	0.1068	0.0999	0.0851	0.0604	0.0665	0.1293	0.1346
largest peak and hole (e·Å^{-3})	0.85, -0.33	0.66, -0.71	1.05, -0.48	1.02, –0.63	0.62, –0.30	0.49, –0.51	0.41, –0.34	1.30, -2.35 (near I(2))
CCDC no.	933402	933403	933404	933405	933406	933407	933408	933401

Table S1 (contd.). Summary of crystallographic data for **3-9**, **12**, HL^{Mes}, HL^{Trip}, Piso*H and Piso"H

	HL^{Mes}	HL^{Trip}	Piso*H.$(DME)_{0.5}$	Piso"H
empirical formula	$C_{21}H_{29}N$	$C_{27}H_{41}N$	$C_{73}H_{69}N_2O$	$C_{67}H_{72}N_2$
formula weight	295.45	379.61	990.30	905.27
crystal system	monoclinic	triclinic	tetragonal	triclinic
space group	$P2_1/n$	P-1	$P4_2/n$	P-1
a (Å)	9.6537(3)	9.1877(3)	28.7323(1)	10.4287(5)
b (Å)	12.7371(4)	9.3985(3)	28.7323(1)	12.6751(5)
c (Å)	14.4806(5)	14.1557(4)	13.5461(1)	21.2839(8)
α (°)	90	97.476(3)	90	100.067(2)
β (°)	93.123(3)	95.922(3)	90	98.285(2)
γ (°)	90	97.036(3)	90	98.844(2)
V ($Å^3$)	1777.89(10)	1193.99(6)	11182.92(10)	2690.7(2)
Z	4	2	8	2
T (K)	123(2)	123(2)	123(2)	123(2)
ρ_{calcd} ($g \cdot cm^3$)	1.104	1.056	1.176	1.117
μ (mm^{-1})	0.063	0.060	0.519	0.064
F(000)	648	420	4232	976
reflns collected	11826	18608	85483	35386
unique reflns	3893	5195	9907	12208
R_{int}	0.0217	0.0164	0.0294	0.0310
R1 indices [$I > 2\sigma(I)$]	0.0439	0.0453	0.0580	0.0596
wR2 indices (all data)	0.1193	0.1200	0.1559	0.1667
largest peak and hole ($e \cdot Å^{-3}$)	0.25, -0.25	0.33, -0.36	0.75, -0.53	0.70, -0.35
CCDC no.	933397	933398	933400	933399