Electronic Supplementary Material (ESI) for Dalton Transactions.

Alkali Metal Reduction of 1,3,2-Diazaborol and 1,3,2-Diazagermol Derivatives Based on 1,2-Bis[(2,6-diisopropylphenyl)imino]acenaphthene

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X-ray crystallography. The X-ray data for **3**, **4** and **8** were collected at 100(2) K on a Bruker AXS D8 Quest Photon and Agilent Xcalibur E (MoKa-radiation, ω-scan technique, λ = 0.71073 Å). The structures were solved by direct methods and were refined by full-matrix least squares on F^2 using SHELXTL [Sheldrick G.M. (2014). SHELXTL v. 2014/7, Structure Determination Software Suite, Bruker AXS, Madison, Wisconsin, USA]. All hydrogen atoms were placed in calculated positions and were refined in the riding model. SCALE3 ABSPACK [SCALE3 ABSPACK: Empirical absorption correction, CrysalisPro Software Package Agilent Technologies, 2012] for **3**, **8** and SADABS [Sheldrick G.M. (2012). SADABS v.2012/1, Bruker/Siemens Area Detector Absorption Correction Program, Bruker AXS, Madison, Wisconsin, USA] for **4** were used to perform area-detector scaling and absorption corrections. Crystallographic data and structural refinement details are given in Table 1. CCDC 1957529 (**3**), 1957531 (**4**), 1957530 (**8**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table 1. Crystal Data and Structure Refinement Details for Compounds **3**, **4** and **8**.

	3 · 0.875 $C_{10}H_8$	**4**	**8**
Formula	$C_{96.75}H_{127}B_2Br_2Li_4N_4O_4$	$C_{79}H_{88}B_2K_2N_4O_2$	$C_{48}H_{71}GeN_2NaO_6$
Formula weight	1619.22	1225.35	867.64
Temperature/K	100(2)	100(2)	100(2)
Crystal system	Triclinic	Tetragonal	Orthorhombic
Space group	P-1	I-4	P2(1)2(1)2(1)
a/Å *b*/Å *c*/Å *α*/deg *β*/deg γ/deg	13.2924(4) 14.4434(3) 24.8139(7) 101.192(2) 102.617(2) 91.829(2)	33.394(3) 33.394(3) 12.3370(9) 90 90 90	12.205(3) 19.4474(18) 20.653(6) 90 90 90
V/Å^3	4546.7(2)	13758(2)	4902.1(18)
Z	2	8	4
density/g/sm^3	1.183	1.183	1.176
μ/mm^{-1}	0.942	0.187	0.679
F(000)	1719	5232	1856
Crystal size /mm	0.700 × 0.435 × 0.330	0.350 × 0.250 × 0.200	0.510 × 0.270 × 0.110
θ-range/deg	3.038–26.000	0.862– 24.999	3.556– 25.999
Index ranges	–16 ≤ h ≤ 16 –17 ≤ k ≤ 17 –30 ≤ l ≤ 30	–39 ≤ h ≤ 38 –20 ≤ k ≤ 39 –14 ≤ l ≤ 14	–15 ≤ h ≤ 13 –22 ≤ k ≤ 23 –15 ≤ l ≤ 25
Reflections collected	73871	38965	17664
Independent reflections	17853 [R_{int} = 0.0719]	12049 [R(int) = 0.0594]	9511 [R_{int} = 0.0424]
Goodness-of-fit on F^2	1.004	1.018	1.011
R_1/wR_2 (I>2σ(I))	0.0551 / 0.1285	0.0658 / 0.1604	0.0478 / 0.0906
R_1/wR_2 (all parameters)	0.0997 / 0.1456	0.0959 / 0.1753	0.0733 / 0.0995
Largest diff peak/hole [e Å^{-3}]	0.880 / –0.848	0.808 / -0.293	0.306 / 0.303

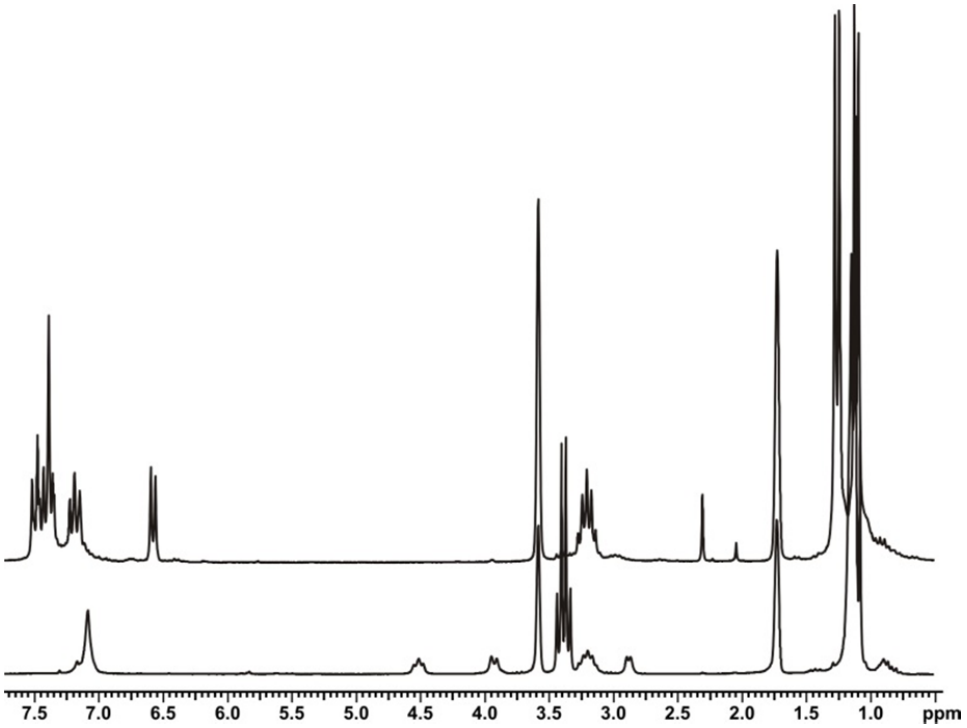

Figure 1S. ^{1}H NMR spectra of the compounds **1** (top) and **3** (bottom) (200 MHz, 300 K, thf-d_8).

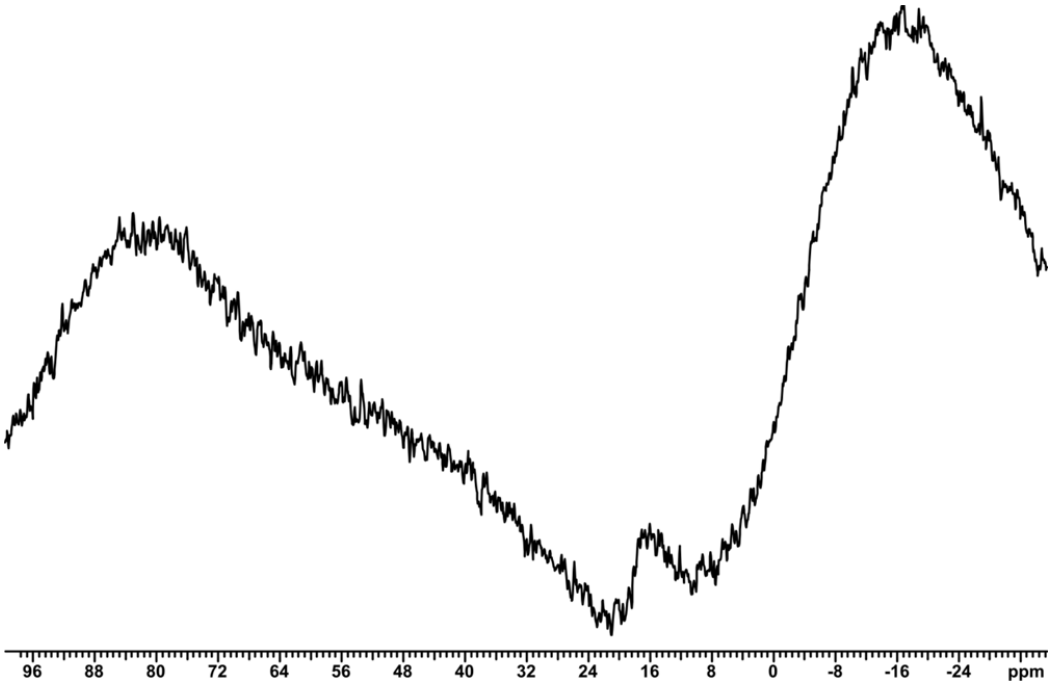

Figure 2S. ^{11}B NMR spectrum of the compound **3** (64.21 MHz, 300 K, thf-d_8).

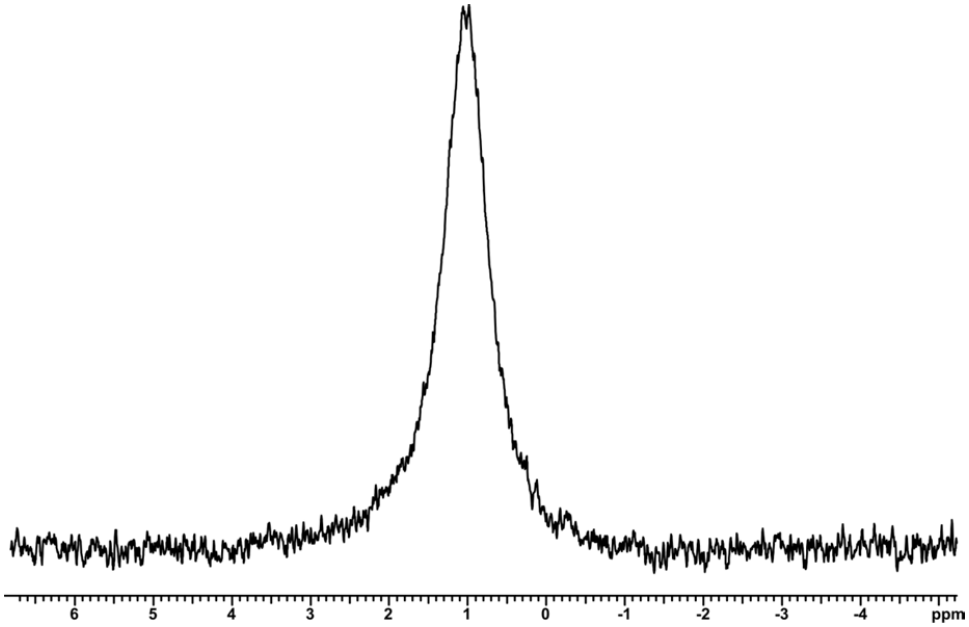

Figure 3S. ^{7}Li NMR spectrum of the compound **3** (77.78 MHz, 300 K, thf-d_8).

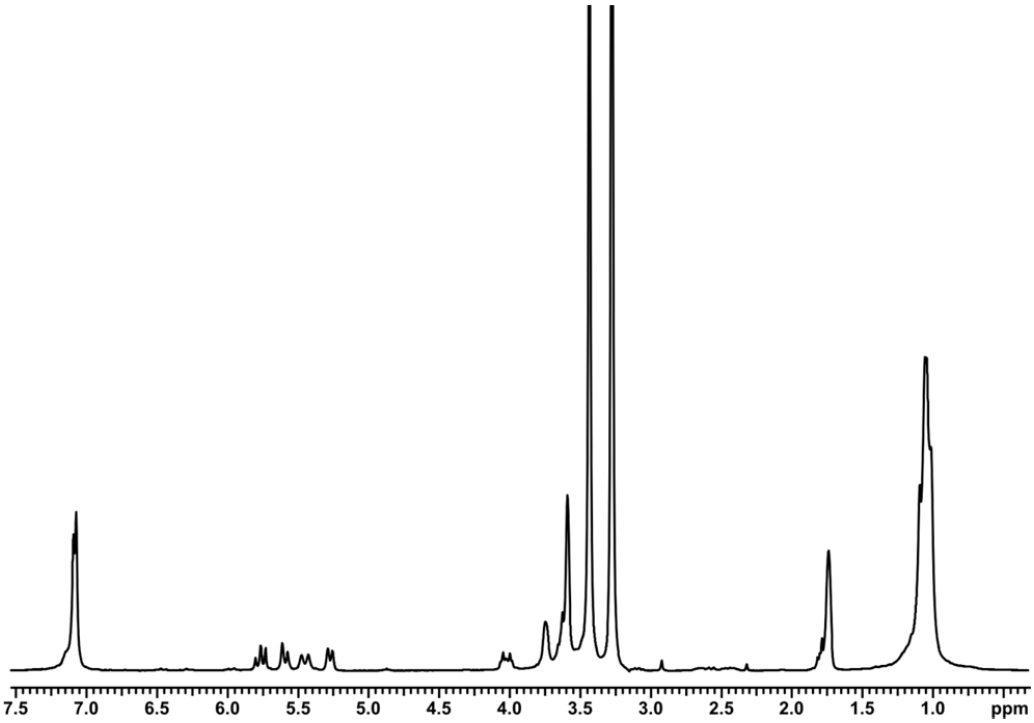

Figure 4S. ^{1}H NMR spectrum of the compound **6** (200 MHz, 298 K, thf-d_8)

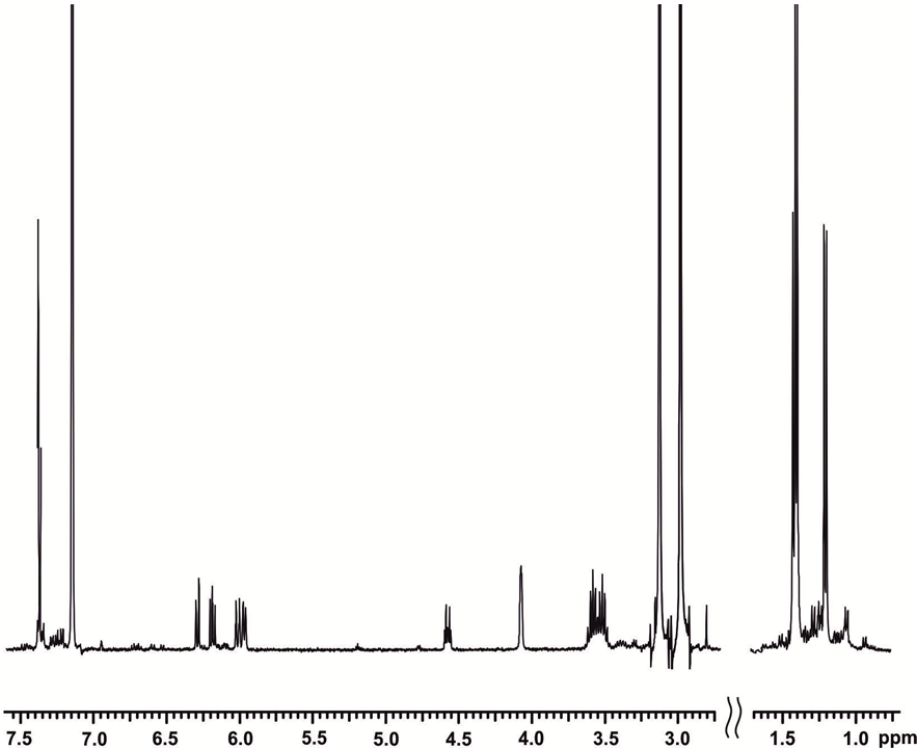

Figure 5S. ^{1}H NMR spectrum of the compound **8** (400 MHz, 296 K, C_6D_6)

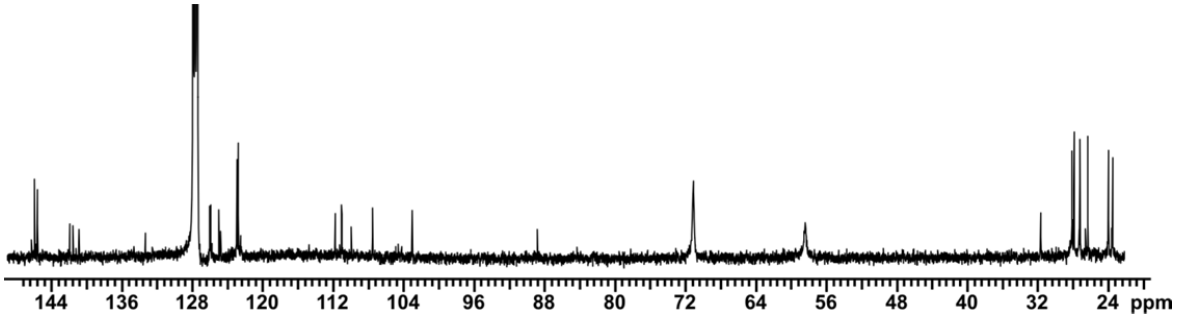

Figure 6S. ^{13}C NMR spectrum of the compound **8** (100 MHz, 296 K, C_6D_6)

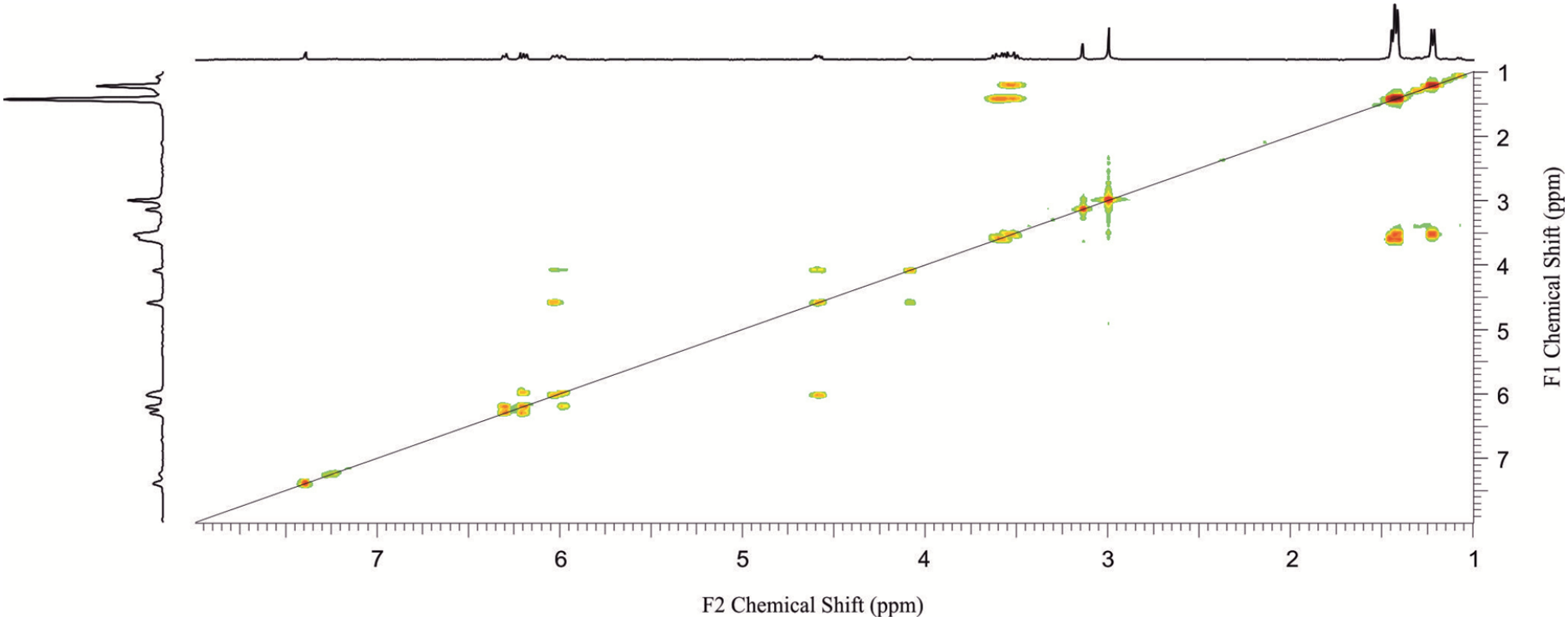

Figure 7S. ^{1}H-^{1}H COSY spectrum of **8** (400 MHz, 296 K, C_6D_6).