Electronic Supplementary Material (ESI) for Dalton Transactions.

Supporting Information

Synthesis of Heteroleptic Gallium-substituted Antimony Hydrides by Stepwise β-H Elimination

*Christoph Helling, Christoph Wölper, and Stephan Schulz**

Institute of Inorganic Chemistry and Center for Nanointegration Duisburg-Essen (CENIDE), University of Duisburg-Essen, 45117 Essen, Germany

Contents

I. Computational Studies

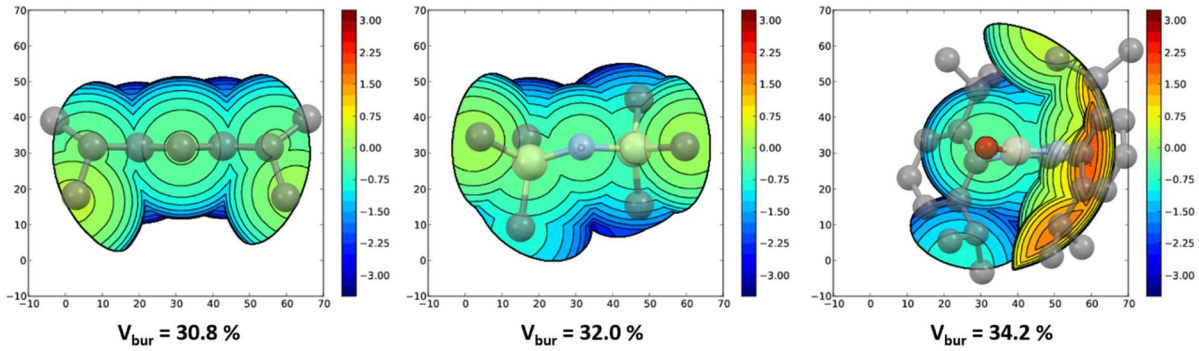

Figure S1. Steric maps and buried volumes V_{bur} of ligands Dip (left), $N(SiMe_3)_2$ (middle), and $OB(NDipCH)_2$ (right). The sphere radius was set at 3.5 Å.

II. Spectroscopic characterisation

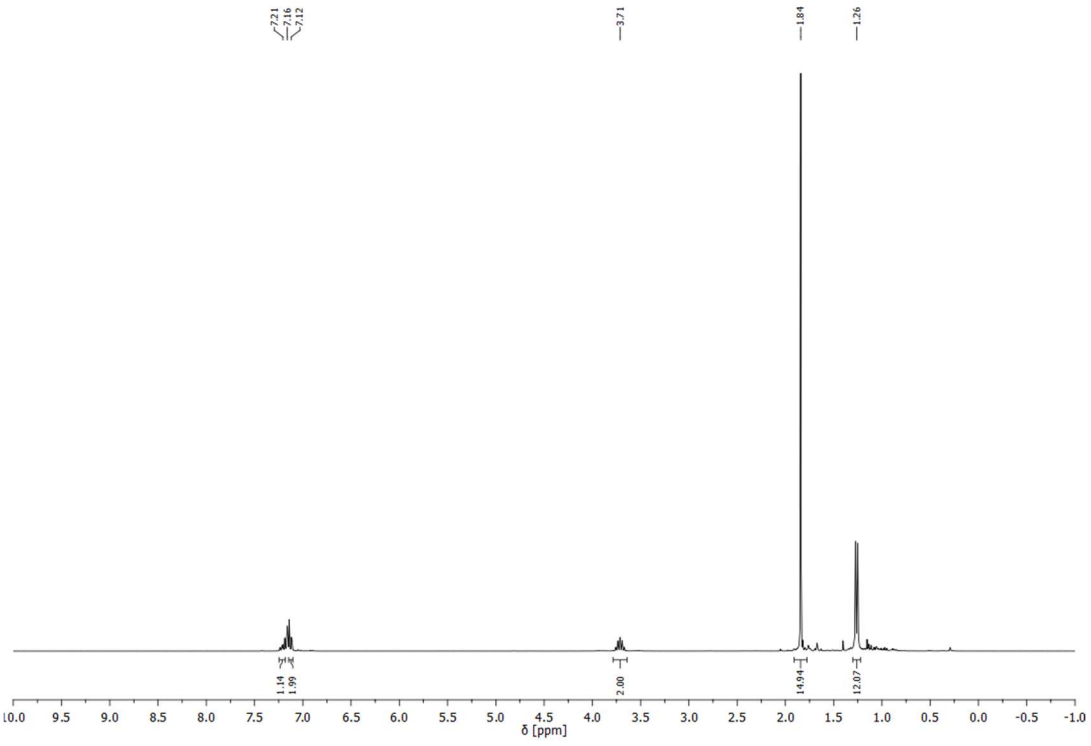

Figure S2. ^1H NMR spectrum of **1** in C_6D_6.

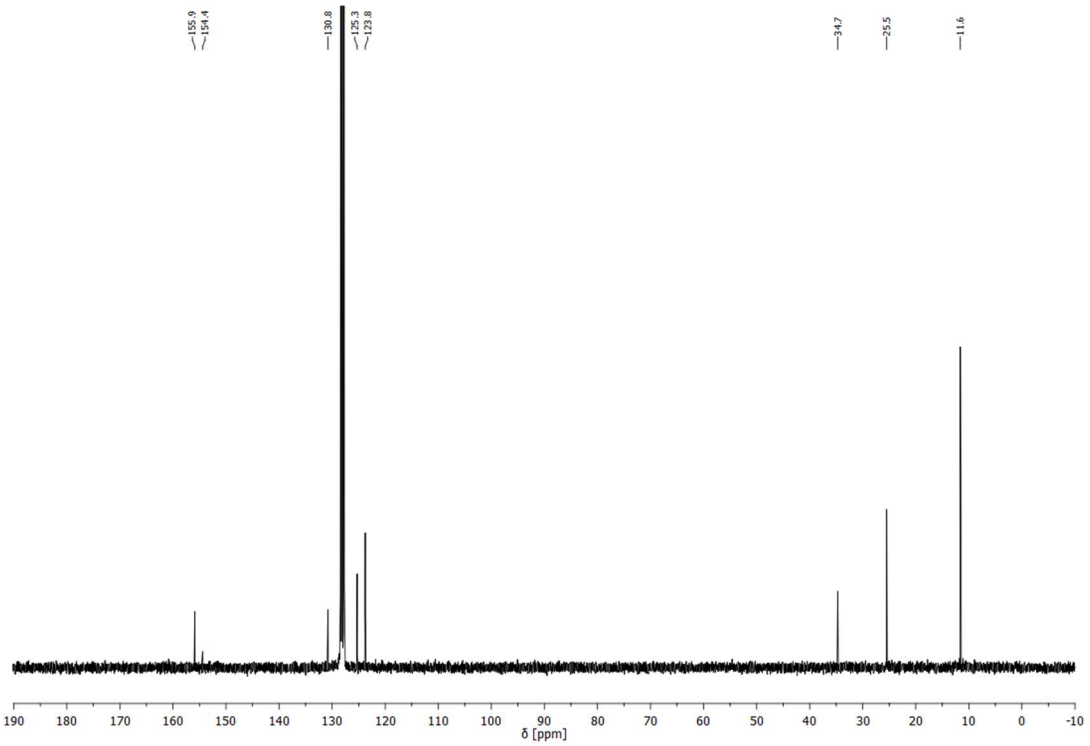

Figure S3. $^{13}C\{^{1}H\}$ NMR spectrum of **1** in C_6D_6.

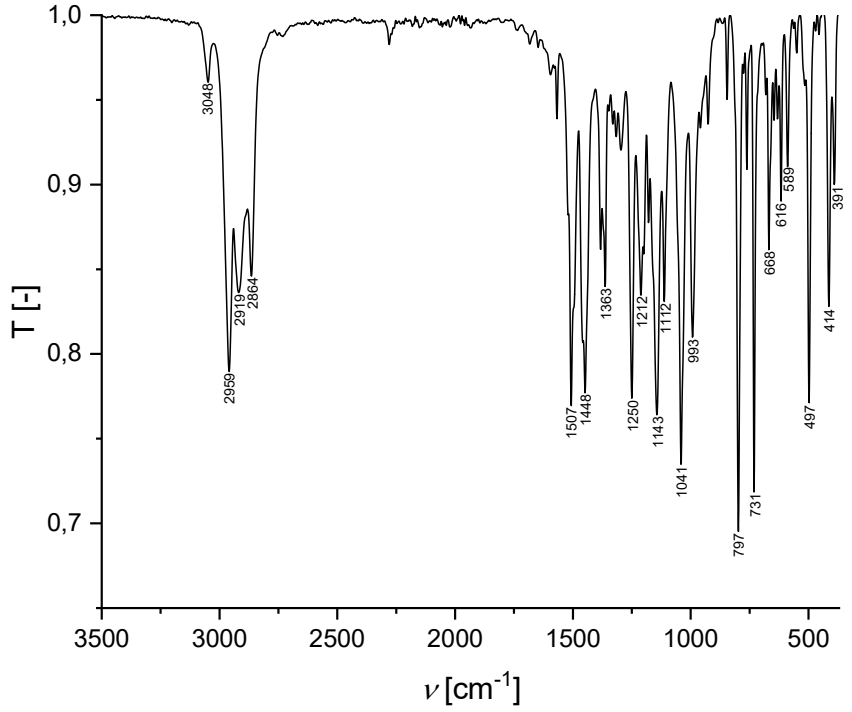

Figure S4. IR spectrum of **1**.

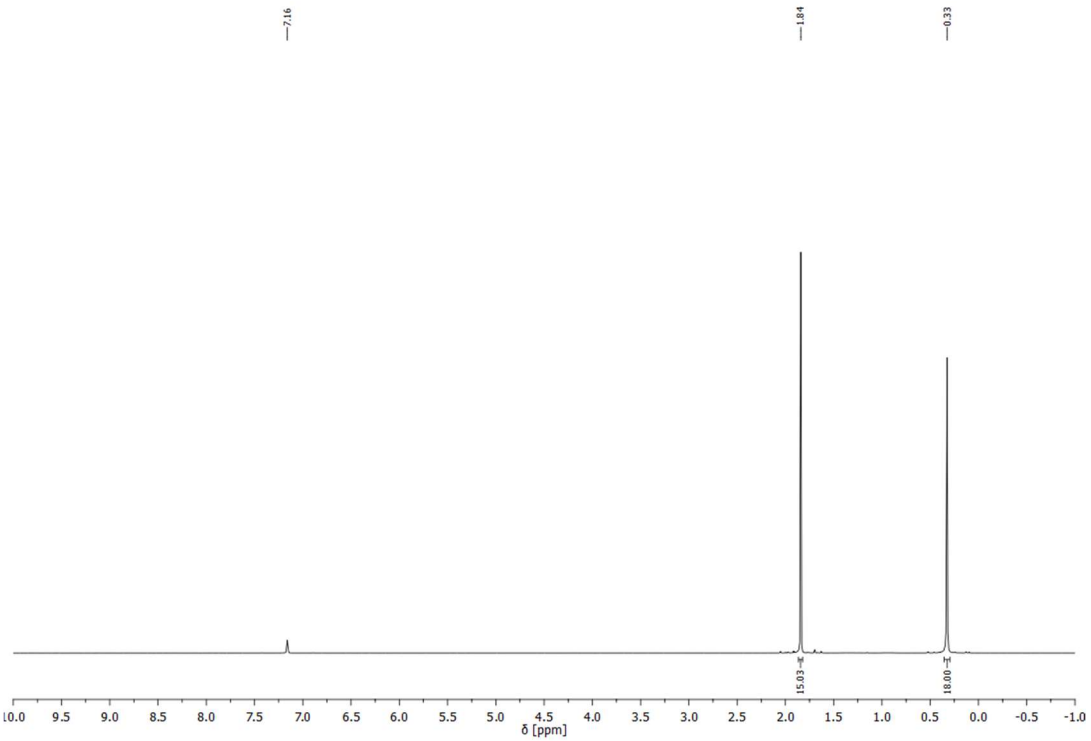

Figure S5. ^{1}H NMR spectrum of **2** in C_6D_6.

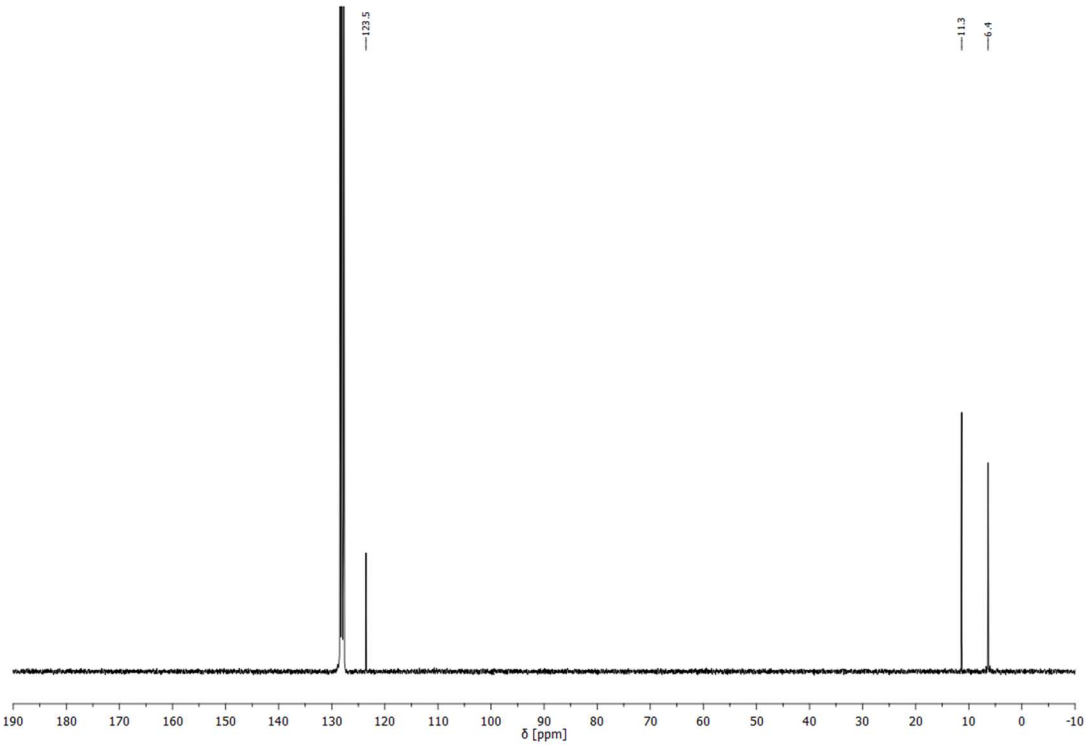

Figure S6. $^{13}C\{^{1}H\}$ NMR spectrum of **2** in C_6D_6.

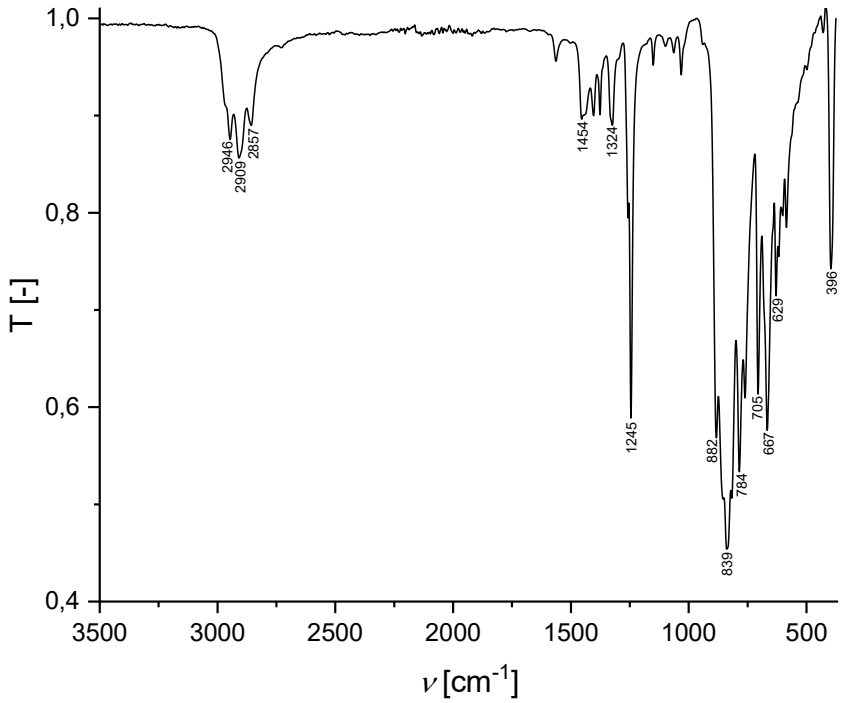

Figure S7. IR spectrum of **2**.

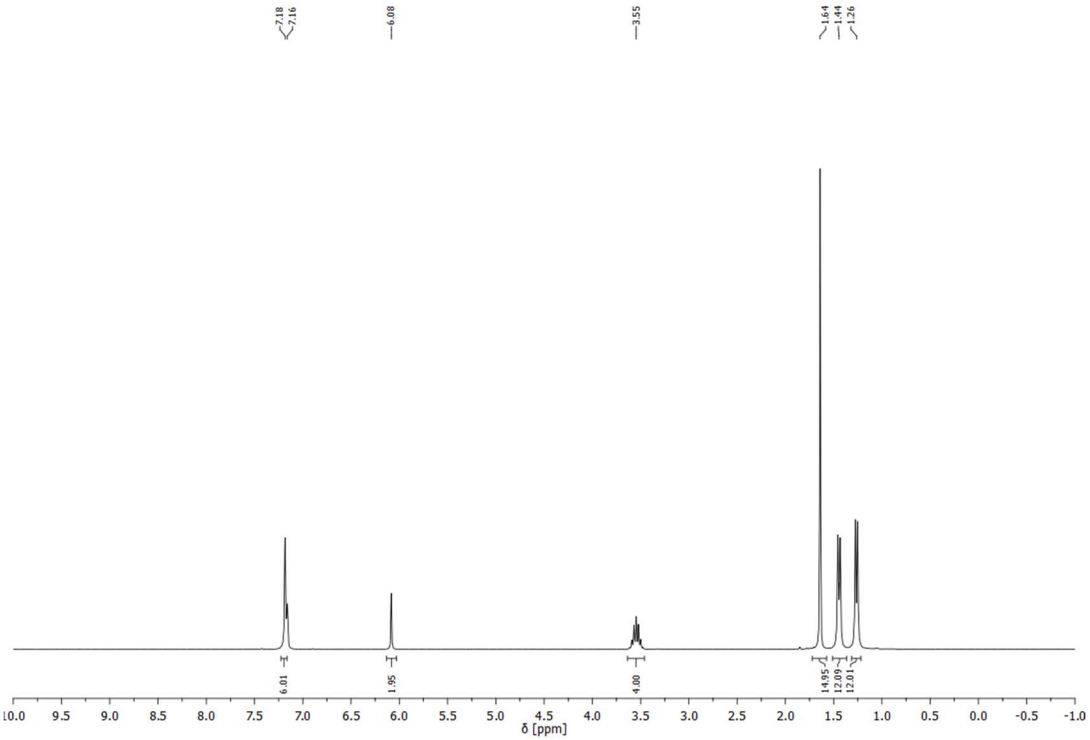

Figure S8. ^{1}H NMR spectrum of **3** in C_6D_6.

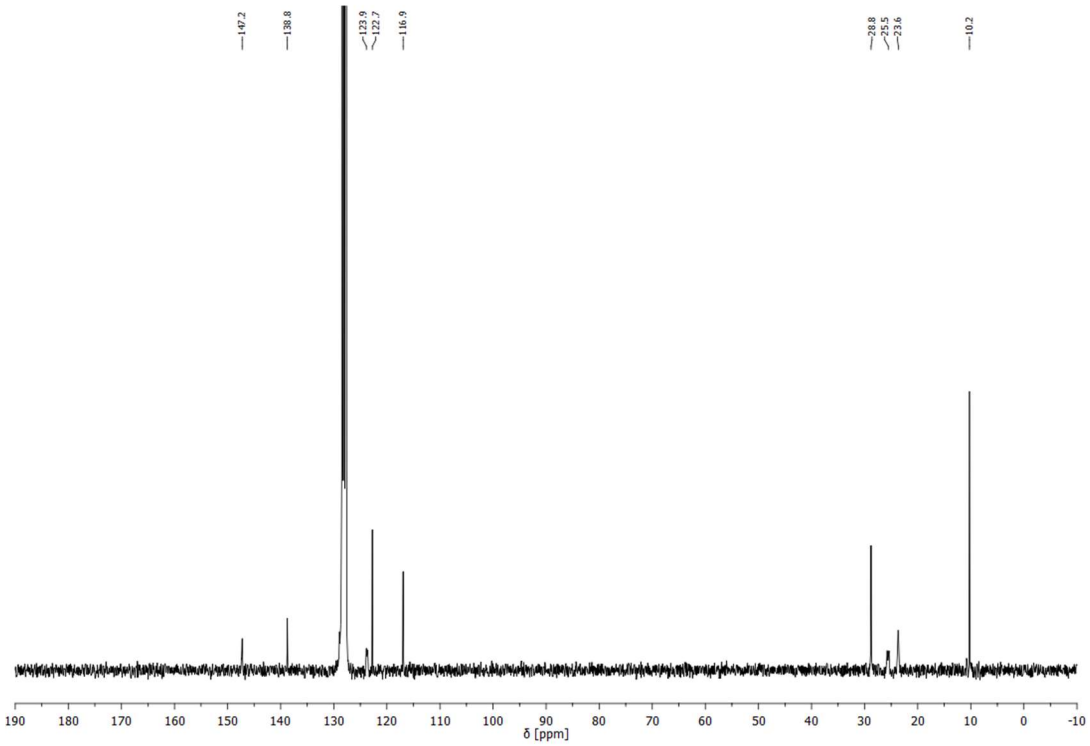

Figure S9. $^{13}C\{^{1}H\}$ NMR spectrum of **3** in C_6D_6.

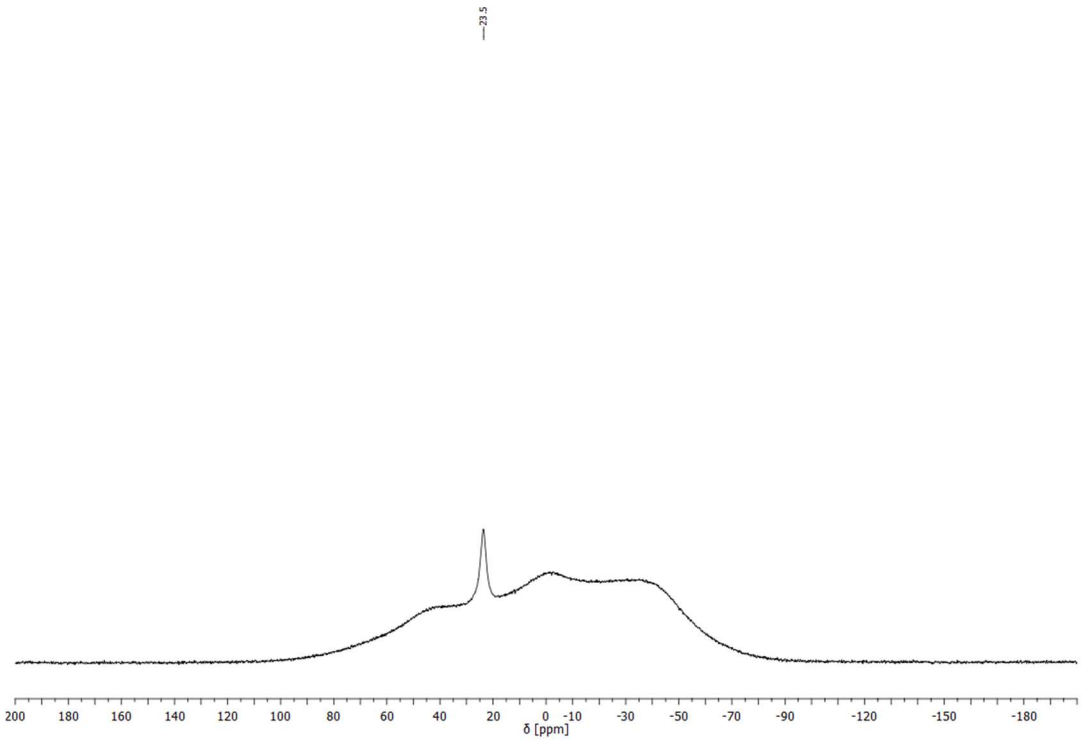

Figure S10. $^{11}B\{^{1}H\}$ NMR spectrum of **3** in C_6D_6.

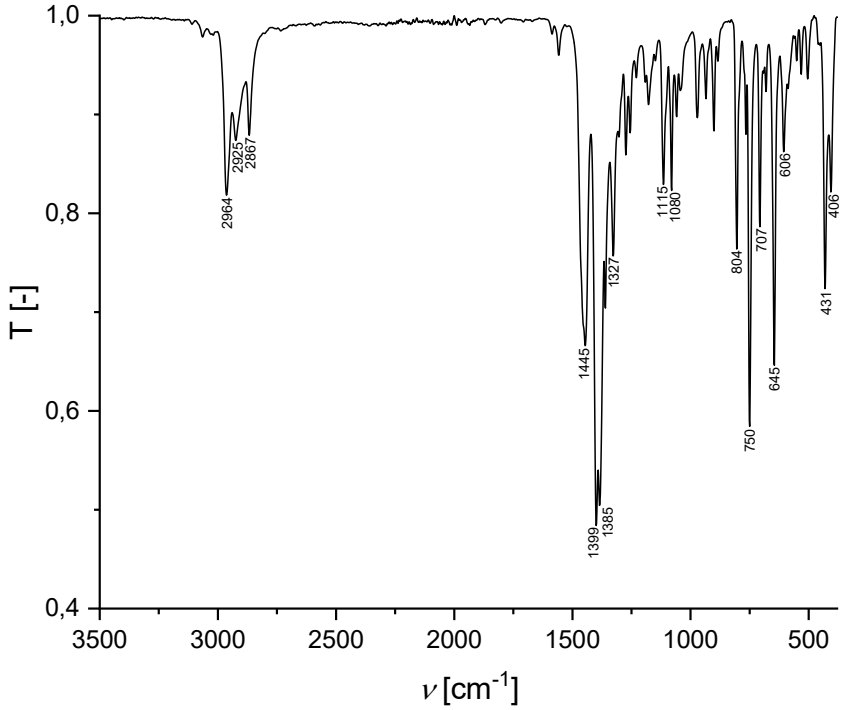

Figure S11. IR spectrum of **3**.

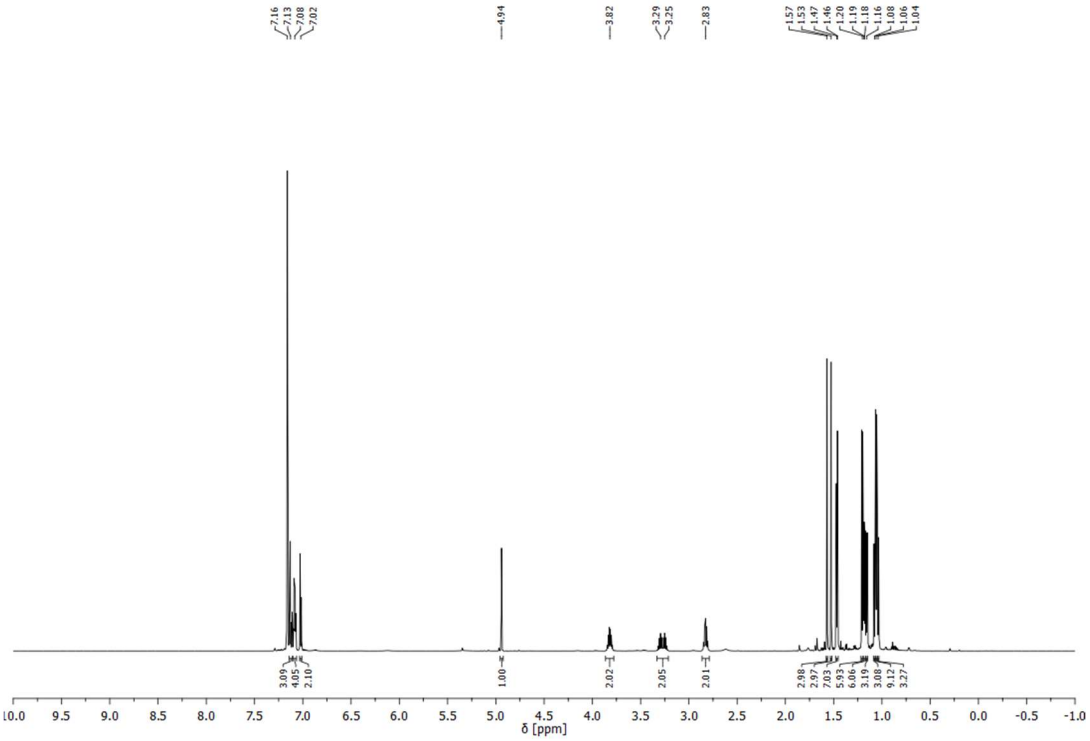

Figure S12. ^{1}H NMR spectrum of **4** in C_6D_6.

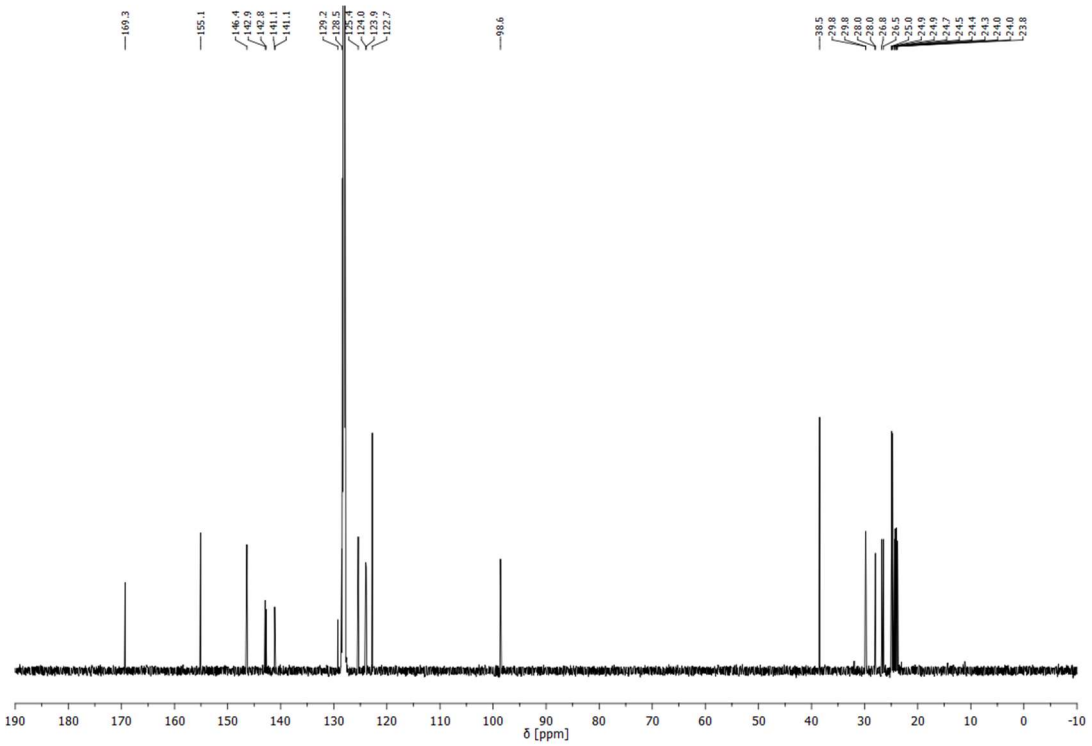

Figure S13. $^{13}C\{^{1}H\}$ NMR spectrum of **4** in C_6D_6.

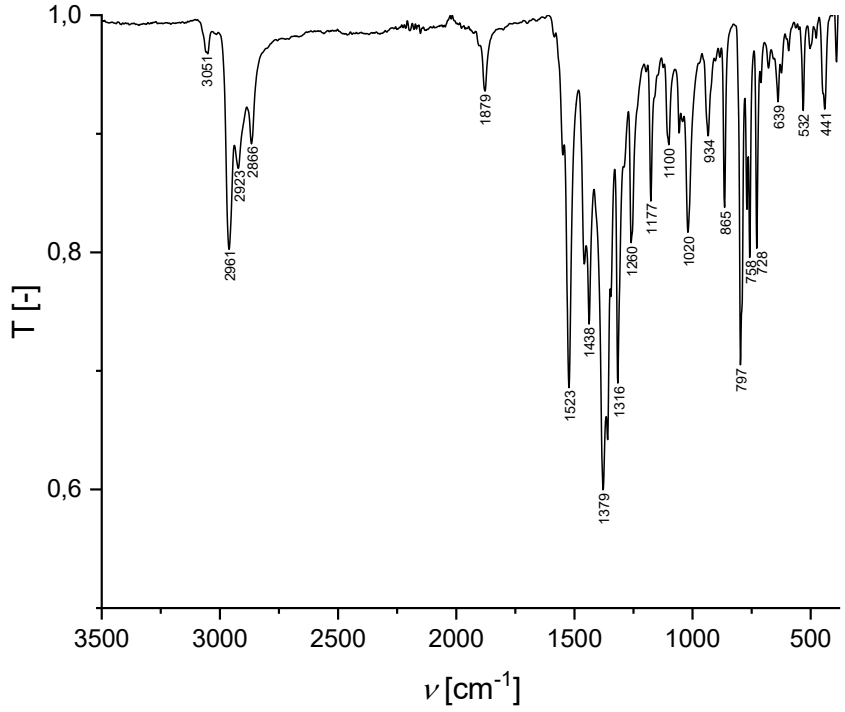

Figure S14. IR spectrum of **4**.

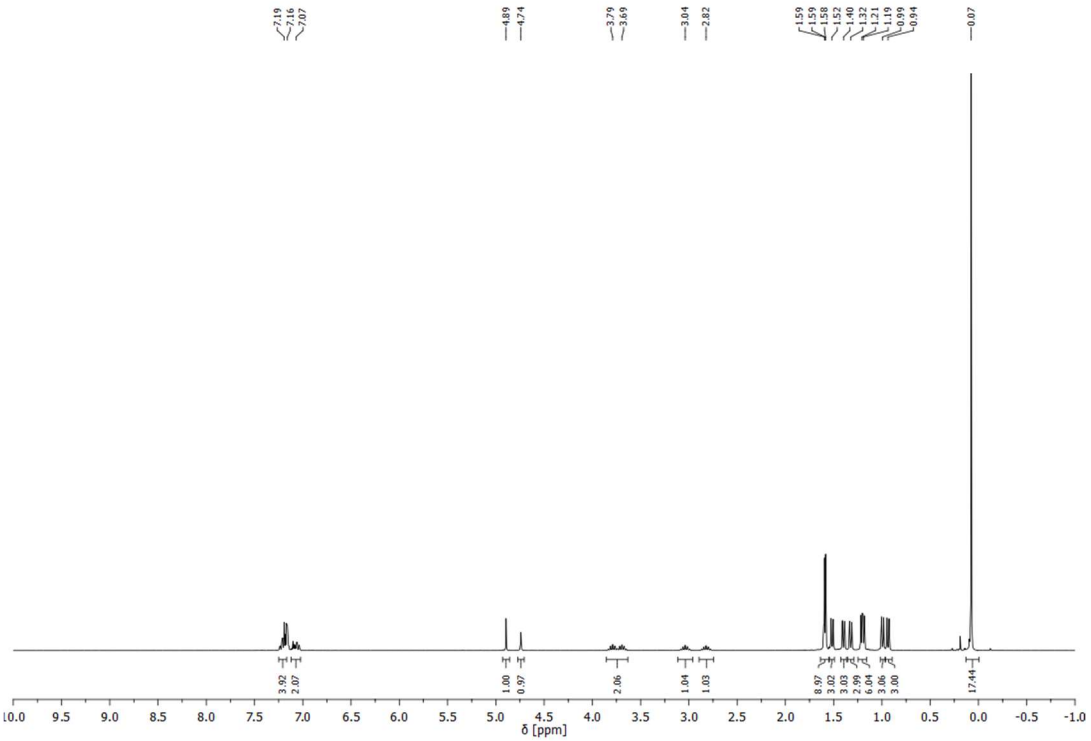

Figure S15. ^{1}H NMR spectrum of **5** in C_6D_6.

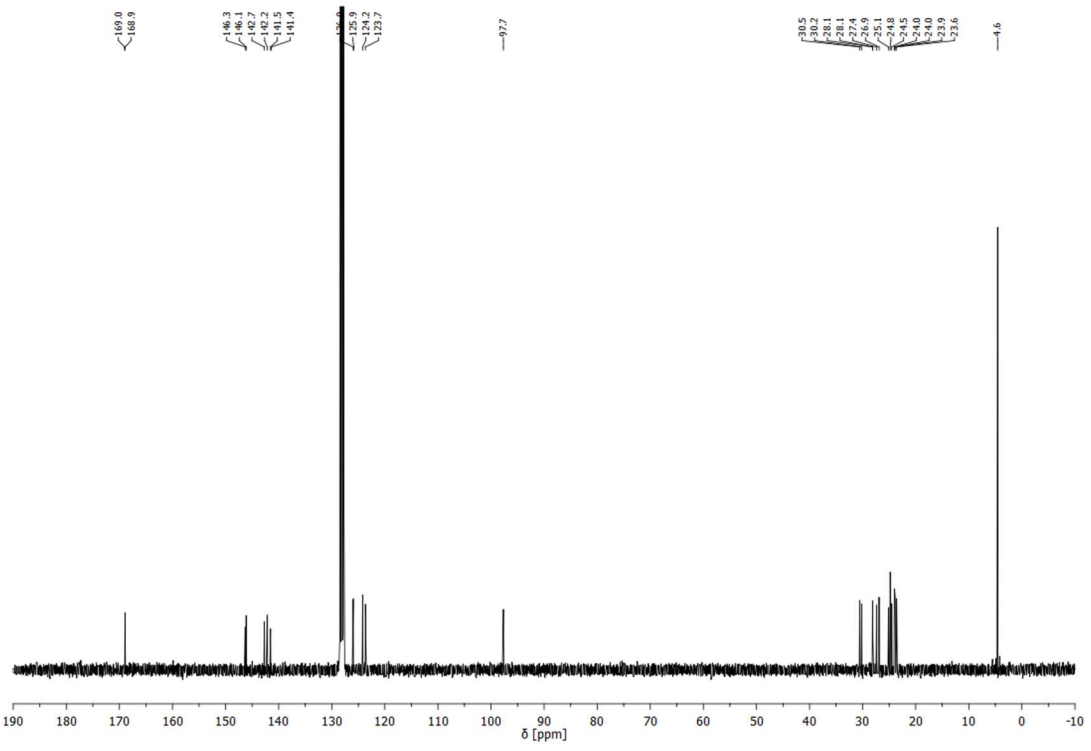

Figure S16. $^{13}C\{^{1}H\}$ NMR spectrum of **5** in C_6D_6.

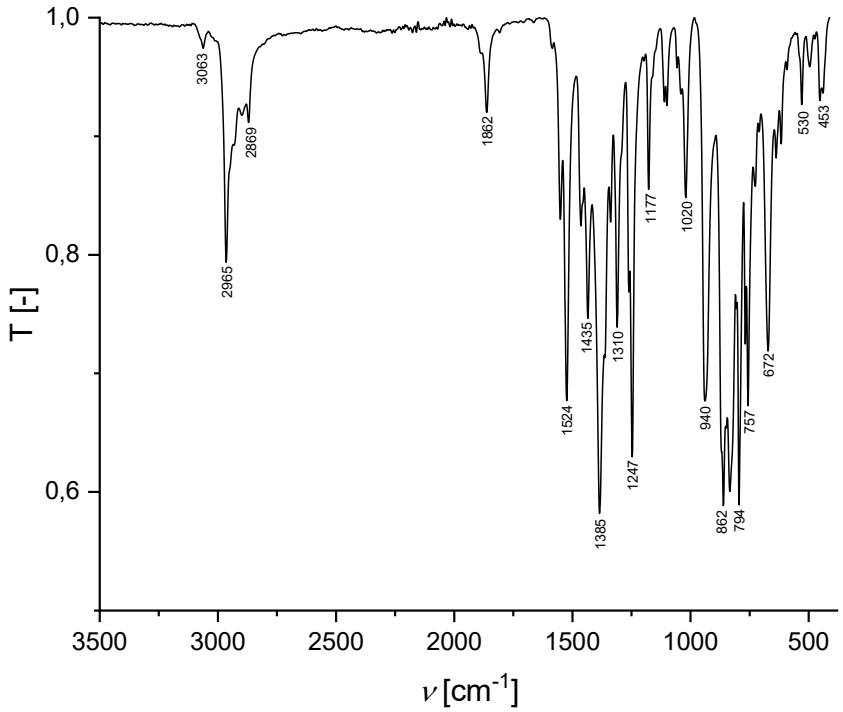

Figure S17. IR spectrum of **5**.

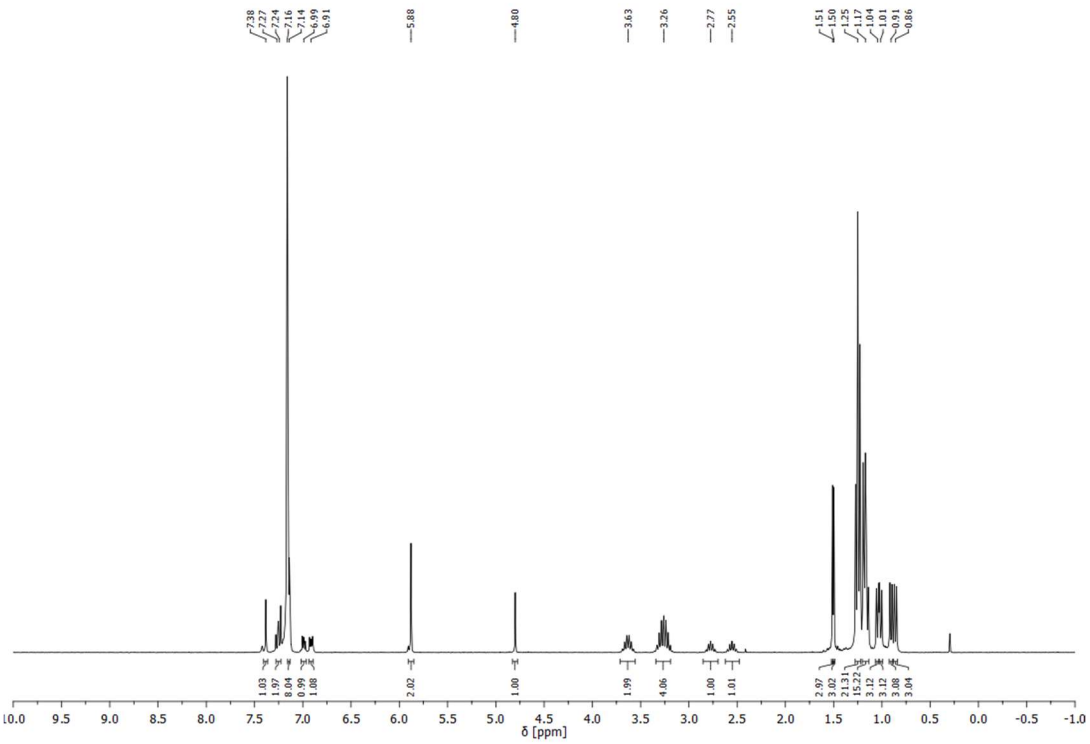

Figure S18. ^{1}H NMR spectrum of **6** in C_6D_6.

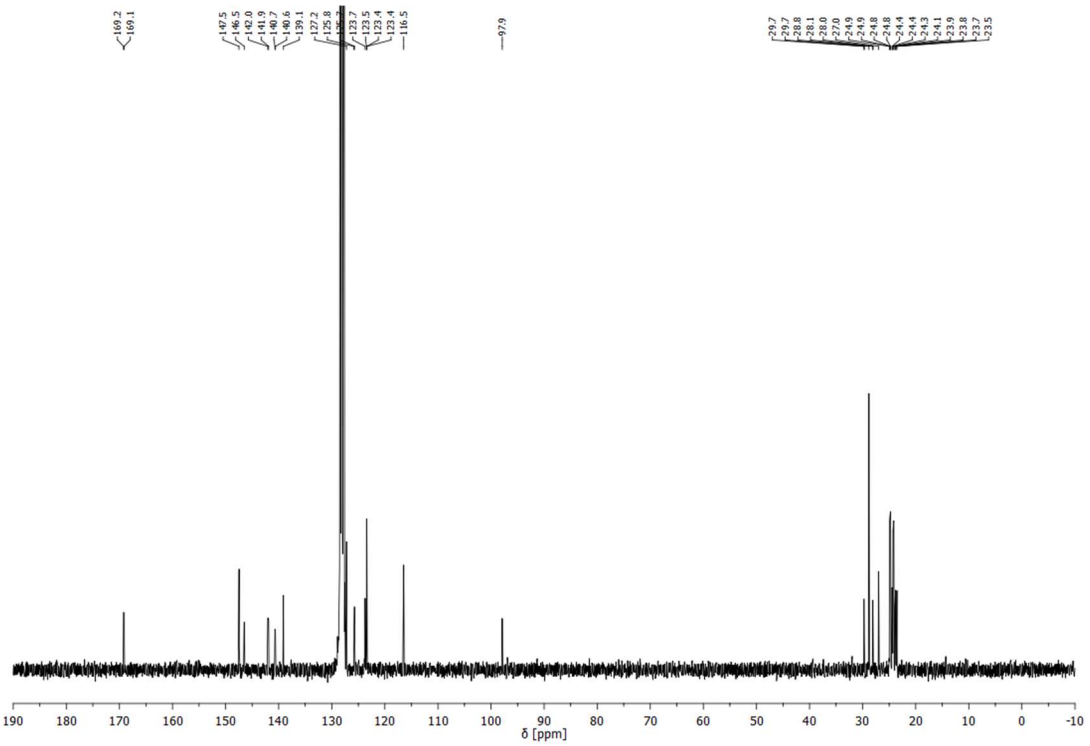

Figure S19. $^{13}C\{^{1}H\}$ NMR spectrum of **6** in C_6D_6.

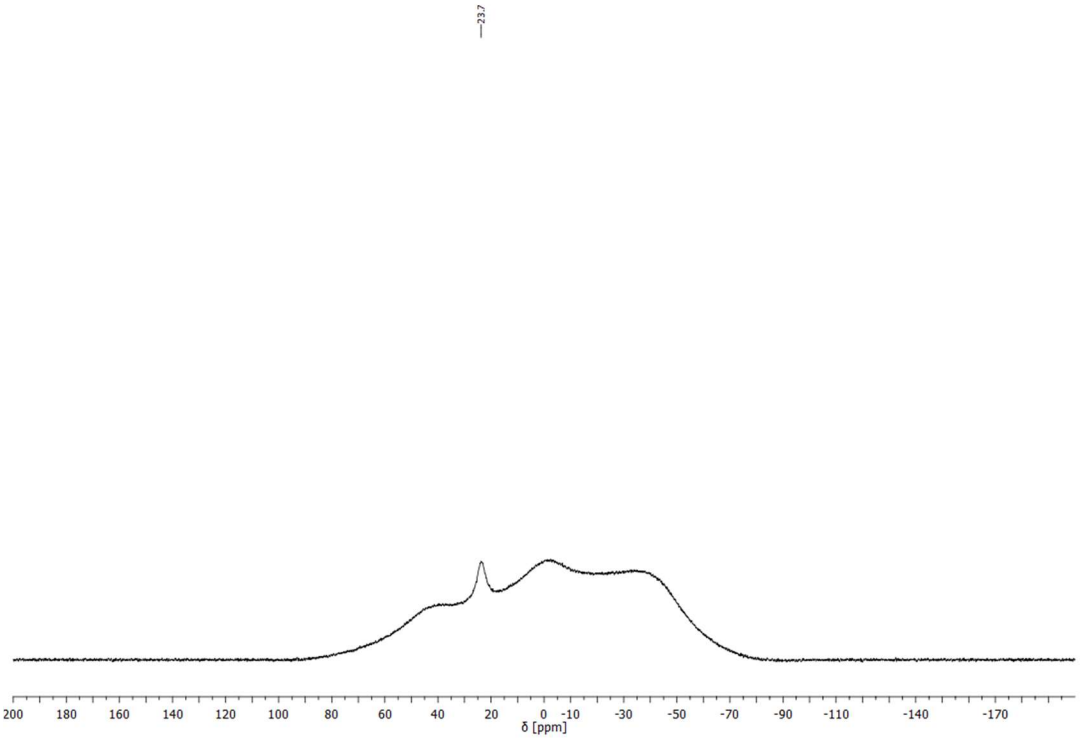

Figure S20. $^{11}B\{^{1}H\}$ NMR spectrum of **6** in C_6D_6.

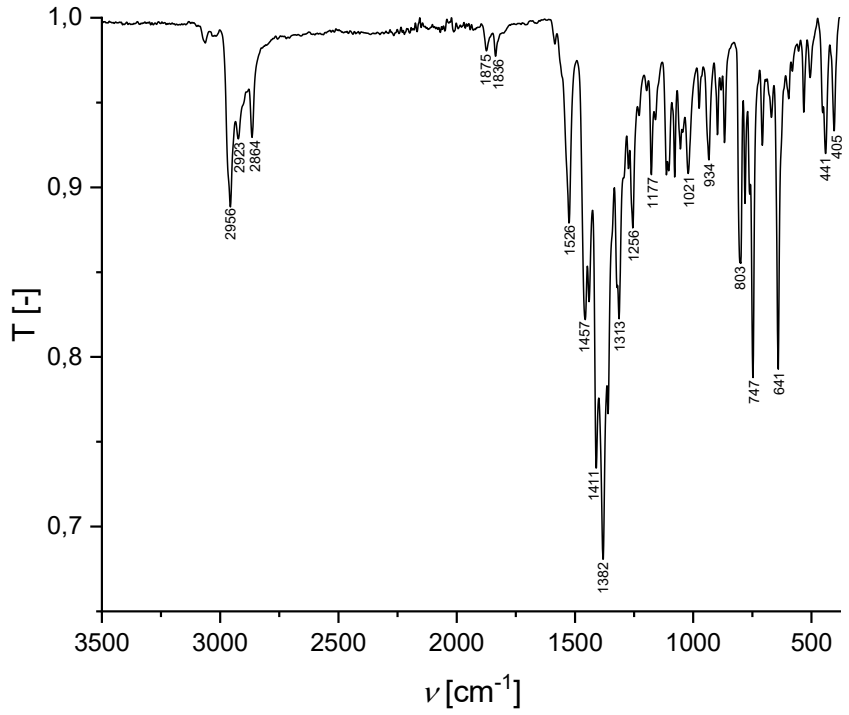

Figure S21. IR spectrum of **6**.

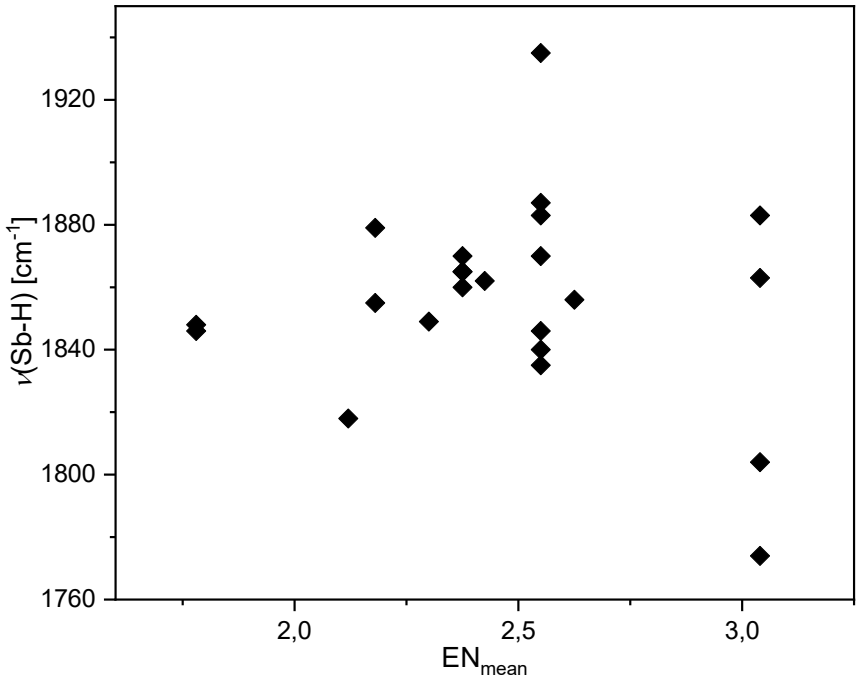

Figure S22. *ν*(Sb-H) vs. EN_{mean} plot for known Sb hydrides.

III. Crystallographic Details

Table S1. Crystallographic details of **4**–**6.**

	4	**5**	**6**
Empirical formula	$C_{41}H_{59}ClGaN_2Sb$	$C_{35}H_{60}ClGaN_3SbSi_2$	$C_{55}H_{78}BClGaN_4OSb$
M [g mol^{-1}]	806.82	805.96	1048.94
Crystal size [mm]	0.184 × 0.147 × 0.128	0.275 × 0.184 × 0.078	0.420 × 0.120 × 0.100
T [K]	100(2)	100(2)	100(2)
Crystal system	triclinic	monoclinic	monoclinic
Space group	P-1	$P2_1/c$	$P2_1/c$
a [Å]	12.5644(10)	9.861(3)	19.112(3)
b [Å]	16.2940(13)	19.979(6)	13.617(2)
c [Å]	20.5464(14)	20.390(6)	21.170(4)
α [°]	105.056(3)	90	90
β [°]	90.827(4)	103.101(14)	99.737(3)
γ [°]	97.901(4)	90	90
V [Å^3]	4017.9(5)	3913(2)	5430.1(16)
Z	4	4	4
D_{calcd} [g cm^{-3}]	1.334	1.368	1.283
μ(K_α [mm^{-1}])	1.438 (Mo)	1.535 (Mo)	1.083 (Mo)
Transmissions	0.75/0.67	0.75/0.59	0.75/0.65
F(000)	1627	1672	2192
Index ranges	-18 ≤ h ≤ 18	-15 ≤ h ≤ 15	-27 ≤ h ≤ 27
	-23 ≤ k ≤ 24	-30 ≤ k ≤ 30	-19 ≤ k ≤ 19
	-31 ≤ l ≤ 29	-30 ≤ l ≤ 31	-30 ≤ l ≤ 30
ϑ_{max} [°]	33.348	33.141	30.774
Reflections collected	143731	146299	170987
Independent reflections	27366	14916	16905
R_{int}	0.0307	0.0459	0.0773
Refined parameters	865	447	599
R_1 [$I > 2\sigma(I)$]	0.0349	0.0292	0.0367
wR_2 [all data]	0.0821	0.0825	0.0782
GooF	1.021	1.026	1.053
$\Delta\rho_{final}$ (max/min) [e·Å^{-3}]	2.201/-3.789	1.852/-0.607	0.982/-0.739