Supplementary Information

Synthesis and Reactivity of Fluorenyl-Tethered N-heterocyclic Stannylenes

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Supplementary Information

Additional experimental details

Synthesis of t-BuN(H)$C_2H_4NH_2$

Adapted from a literature procedure:[1] $BrCH_2CH_2NH_2$.HBr (21.1 g, 103 mmol), dissolved in H_2O (60 cm^3), and *t*-butyl amine (52 cm^3, 36 g, 492 mmol) were heated under reflux for 24 h. The volatiles were removed under vacuum leaving a white solid which was dissolved in MeOH (60 cm^3) and solid KOH (17.5 g) was slowly added (3-4 g at a time) with water bath cooling (slightly exothermic). After stirring for 50 mins, the MeOH was removed under vacuum (rotary vane oil pump) until the mixture was no longer cool to the touch, then the diamine was distilled directly from the flask (48°C and 48 mbar) yielding *t*-BuN(H)$C_2H_4NH_2$ as a colourless liquid (5.14 g, 44.2 mmol, 43%).

^{1}H-NMR (300 MHz, $CDCl_3$): δ 2.71 (m, 2H, CH2), 2.54 (m, 2H, CH2), 1.04 (br. s, 4 H, NH), 1.03 (s, 9H, t-Bu).
^{13}C-NMR (75.5 MHz, $CDCl_3$): δ 50.0 (*C*Me_3), 45.2 (CH_2), 42.8 (CH_2), 29.1 (*t*-Bu CH_3).

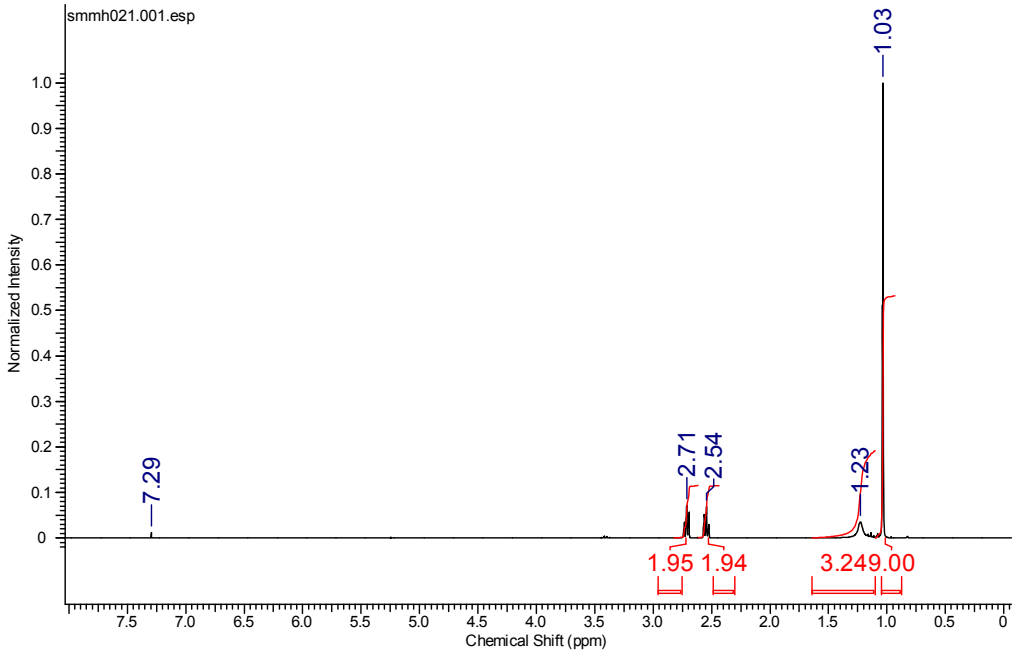


Figure S1. ^{1}H-NMR spectrum of ***t*-BuN(H)$C_2H_4NH_2$** in $CDCl_3$ at 25 °C

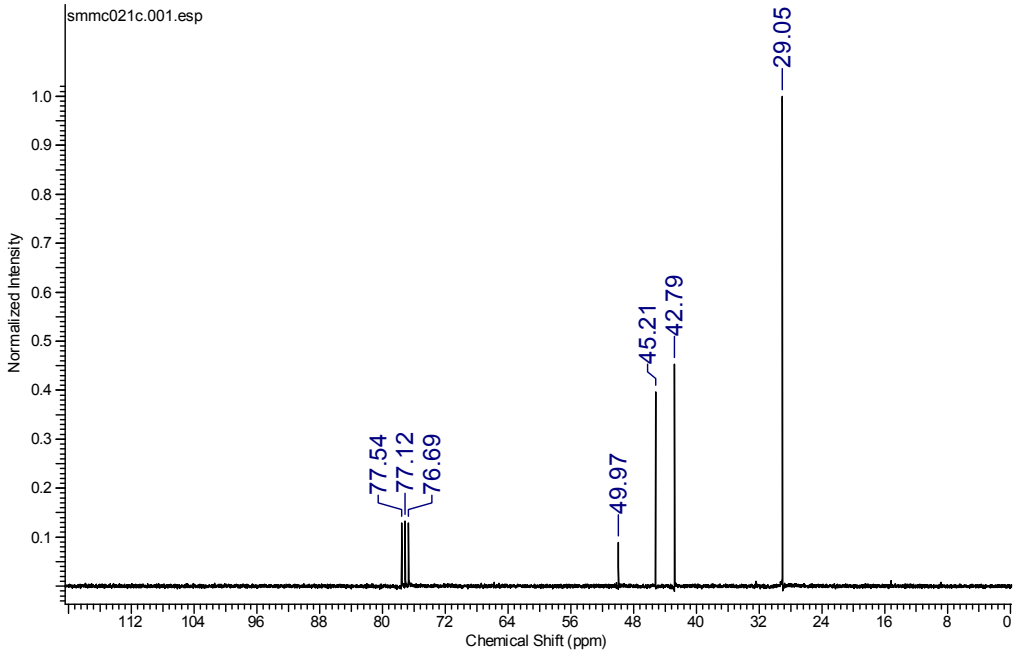
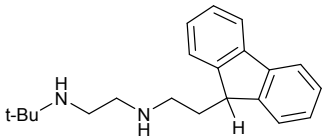


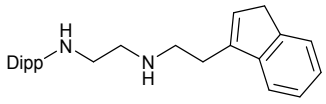
Figure S2. ^{13}C-NMR spectrum of ***t*-BuN(H)$C_2H_4NH_2$** in $CDCl_3$ at 25 °C.



1b

(9H-$C_{13}H_9$)$C_2H_4N(H)C_2H_4N(H)^tBu$ (1b)

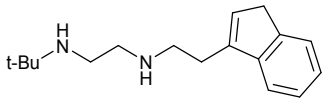
HCl (1.65 cm^3, 1M soln. in Et_2O, 1.5 eq.) was added to $^tBuN(H)C_2H_4NH_2$ (382 mg, 3.29 mmol, 3 eq.) and stirred for 5 mins at 25°C. The volatiles were then removed under reduced pressure, FlC_2H_4Br (300 mg, 1.10 mmol, 1 eq.) added and the mixture heated at 100 °C for 3 hours. The crude product was then dissolved in DCM (3 cm^3) and treated with KOH (155 mg, 2.75 mmol, 2.5 eq.) dissolved in water (2 cm^3). The organic fraction was separated and then purified by column chromatography first with DCM elution to separate the spirocyclopropane impurity. NEt_3 (0.5 cm^3) was then introduced followed by elution with acetone containing 5% NEt_3 to remove the disubstituted diamine impurity then elution with methanol containing 5% NEt_3 extracted the desired product which, after solvent removal under reduced pressure, yielded **1b** as a white solid (113 mg, 0.37 mmol, 33%). Spectroscopic yield before chromatography was found to be 68 - 55%. **^{1}H-NMR (300 MHz, 25°C, $CDCl_3$)**: δ = 7.75 (d, $^3J_{HH}$ = 7.3 Hz, 2H, Ar_{Fl} H), 7.52 (d, $^3J_{HH}$ = 7.3 Hz, 2H, Ar_{Fl} H), 7.36 (tdd, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 1.5 Hz, $^4J_{HH}$ = 0.7 Hz, 2H, Ar_{Fl} H), 7.30 (td, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 1.5 Hz, 2H, Ar_{Fl} H), 4.06 (t, $^3J_{HH}$ = 5.9 Hz, 1H, 9*H*-fluorene), 2.56 (m, 4H, CH_2), 2.48 (m, 2H, CH_2), 2.23 (m, 2H, CH_2), 1.37 (br, 2H, NH), 1.05 (s, 9H, ^{t}Bu); **^{13}C-NMR (100 MHz, 25°C, $CDCl_3$)**: δ 147.1 (Fl-C_q), 141.1 (Fl-C_q), 127.1, 127.0, 124.4, 120.0 (collection of Fl-CH), 50.4 (CH_2), 50.2 (CH_2), 46.3 (CH_2), 45.9 (Fl-9C), 42.1 (CH_2), 33.2 (^{t}Bu-C_q), 29.1 (^{t}Bu-CH_3); **elemental analysis** calcd (%) for $C_{21}H_{28}N_2$: C 81.77, H 9.15, N 9.08; found: C 81.70, H 9.21, N 8.97; **HRMS (ESI):** Calculated for M+1: 309.2325, Found: 309.2397.



1c

(2H-C_9H_7)$C_2H_4N(H)C_2H_4N(H)$Dipp (1c)

Synthesised as for **1a** using $IndC_2H_4Br$ [2] (0.5 g, 2.21 mmol) and $DippN(H)C_2H_4NH_2$ (1.5.g, 6.63 mmol, 3 eq.), heating at 100 °C for 3 hours and purifying using column chromatography (CH_2Cl_2 followed by ethyl acetate) yielding a pale yellow oil (563 mg, 1.55 mmol, 64% yield). **^{1}H-NMR (400 MHz, 25°C $CDCl_3$)**: δ = 7.49 (m, 1H, Ar_{Ind} H), 7.43 (dt, $^3J_{HH}$ = 7.6 Hz, $^4J_{HH}$ = 0.9 Hz, 1H, Ar_{Ind} H), 7.33 (m, 1H, Ar_{Ind} H), 7.24 (dt, $^3J_{HH}$ = 7.3 Hz, $^4J_{HH}$ = 0.9 Hz, 1H, Ar_{Ind} H), 7.12–7.04 (m, 3H, Dipp Ar H), 6.32 (q, $^3J_{HH}$ = 1.2 Hz, 1H, Ar_{Ind} H), 3.37 (m, 2H, Ar_{Ind} CH_2) 3.34 (sept, $^3J_{HH}$ = 6.8 Hz, 2H, Dipp CH), 3.04 (m, 2H, CH_2), 2.99 (m, 2H, CH_2), 2.92 (m, 2H, CH_2), 2.85 (m, 2H, CH_2), 1.25 (d, $^3J_{HH}$ = 6.7 Hz, 12H, Dipp CH_3); **^{13}C-NMR (100 MHz, 25°C $CDCl_3$)**: δ = 145.3 (Ar_{Ind}), 144.6 (Ar_{Ind}), 144.6 (Dipp *ipso*), 142.5 (Dipp *ortho*), 142.3 (Ar_{Ind}), 129.2 (Ar_{Ind} CH), 126.2 (Ar_{Ind} CH), 124.8 (Ar_{Ind} CH), 123.9 (Ar_{Ind} CH), 123.6 (Dipp CH), 123.5 (Dipp CH), 119.0(Ar_{Ind} CH), 51.4 (CH_2), 49.9 (CH_2), 48.1 (CH_2), 37.9 (Ind-CH_2), 28.6 (CH_2), 27.7 (CH ^{i}Pr), 24.4 (CH_3 ^{i}Pr); **elemental analysis** calcd (%) for $C_{25}H_{34}N_2$: C 82.82, H 9.45, N 7.73; found: C 82.59, H 9.63, N 7.78.



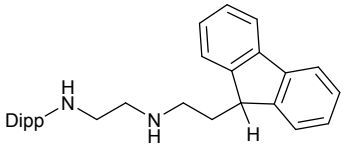
1d

(2H-C_9H_7)C_2H_4N(H)C_2H_4N(H)^{t}Bu (1d)

This was prepared and purified in an analogous manner to **1b** using IndC_2H_4Br [2] (300 mg, 1.34 mmol, 1.0 eq.), ^{t}BuN(H)$C_2H_4NH_2$ (192 mg, 1.65 mmol, 1.3 eq.) and HCl (1.3 cm^3, 1 M Et_2O soln., 1 eq.) after 2 hours at 100 °C. Spectroscopic yield was around 70% and after chromatography **1d** was obtained as a pale yellow oil (95 mg, 0.37 mmol, 42%). **^{1}H-NMR (400 MHz, 25°C, $CDCl_3$)**: δ 7.46 (d, $^3J_{HH}$ = 7.3 Hz, 1H, Ar_{Ind} H), 7.39 (d, $^3J_{HH}$ = 7.6 Hz, 1H, Ar_{Ind} H), 7.30 (t, $^3J_{HH}$ = 7.5 Hz, 1H, Ar_{Ind} H), 7.21 (t, $^3J_{HH}$ = 7.3 Hz, 1H, Ar_{Ind} H), 6.27 (m, 1H, Ar_{Ind} H), 3.34 (m, 2H, Ar_{Ind}, CH_2), 2.96 (m, CH_2, 2H), 2.77 (m, overlapping CH_2, 4H), 2.67 (m, CH_2, 2H), 1.25 (br, NH, 2H), 1.08 (s, ^{t}Bu, 9H); **^{13}C-NMR (100 MHz, 25°C, $CDCl_3$)**: δ 145.4 (Ind-C_q), 144.6 (Ind-C_q), 142.5 (Ind-C_q), 129.1, 126.2, 124.8, 124.0, 119.1 (collection of Ind-CH), 50.3 (CH_2), 50.3 (CH_2), 48.1 (CH_2), 42.1 (CH_2), 37.9 (Ind-CH_2), 29.1 (^{t}Bu-CH_3), 28.5 (^{t}Bu-C_q); **HRMS (ESI)**: Calculated for M+1: 259.2169, Found: 259.2193.

Additional spectroscopic details and NMR spectra

(9H-$C_{13}H_9$)C_2H_4N(H)C_2H_4N(H)Dipp (1a)



1a

^{1}H-NMR (400 MHz, d_8-THF): δ 7.76 (d, $^3J_{HH}$ = 7.1 Hz, 2H, Ar_{Fl} H), 7.56 (d, $^3J_{HH}$ = 7.3 Hz, 2H, Ar_{Fl} H), 7.33 – 7.25 (m, 4H, Ar_{Fl} H), 7.00 – 6.88 (m, 3H, Diip Ar H), 4.12 (t, $^3J_{HH}$ = 6.0 Hz, 1H, 9*H*-fluorene), 3.68 (t, $^3J_{HH}$ = 6.6 Hz, 1H, NH), 3.38 (sept, $^3J_{HH}$ = 6.8 Hz, 2H, Diip CH), 2.85 (m, 2H, CH_2), 2.76 (m, 2H, CH_2), 2.62 (m, 2H, CH_2), 2.19 (m, 2H, CH_2), 1.37 (m, 1H, NH), 1.16 (d, $^3J_{HH}$ = 7.1 Hz, 12H, Diip CH_3). **^{13}C-NMR (100 MHz, d_8-THF)**: δ 148.6, 145.1, 143.5, 142.1, 127.8, 125.3, 124.1, 121.0, 120.7, 52.6, 50.8, 47.7, 47.2, 46.7, 35.0, 28.4.

^{1}H-NMR (400 MHz, C_6D_6): δ 7.63 (d, $^3J_{HH}$ = 6.6 Hz, 2H, Ar_{Fl} H), 7.38 (m, 2H, Ar_{Fl} H), 7.26 – 7.18 (m, 4H, Ar_{Fl} H), 7.15 – 7.08 (m, 3H, Diip Ar H), 3.90 (t, $^3J_{HH}$ = 5.9 Hz, 1H, 9*H*-fluorene), 3.66 (br, 1H, NH), 3.45 (sept, $^3J_{HH}$ = 6.8 Hz, 2H, Diip CH), 2.78 (m, 2H, CH_2), 2.42 (m, 2H, CH_2), 2.37 (m, 2H, CH_2), 1.99 (m, 2H, CH_2), 1.24 (d, $^3J_{HH}$ = 6.7 Hz, 12H, Diip CH_3), 0.39 (br, 1H, NH). **^{13}C-NMR (100 MHz, C_6D_6)**: δ 148.1, 143.0, 141.9, 127.7, 127.6, 125.0, 124.4, 124.2, 120.6, 52.1, 50.2, 47.0, 46.4, 34.3, 28.2, 24.9.

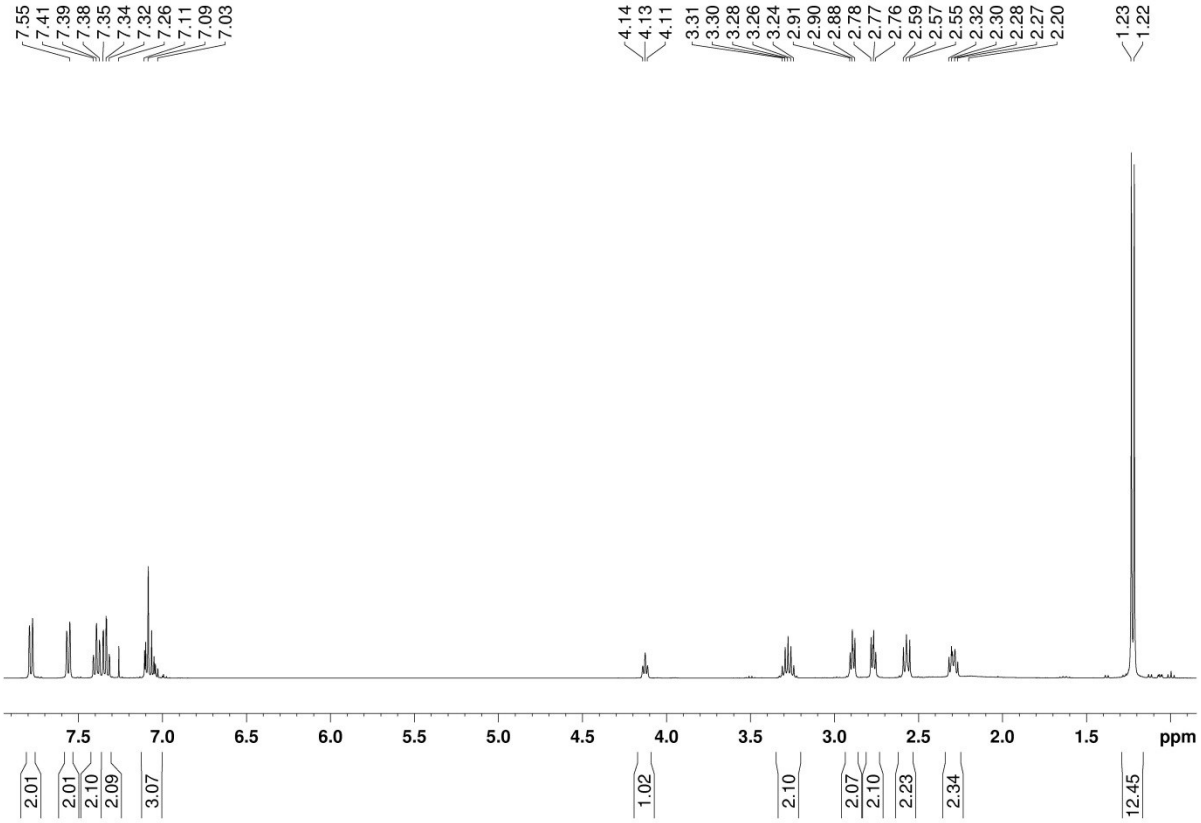


Figure S3. ^{1}H-NMR of **1a** in $CDCl_3$ at 25 °C

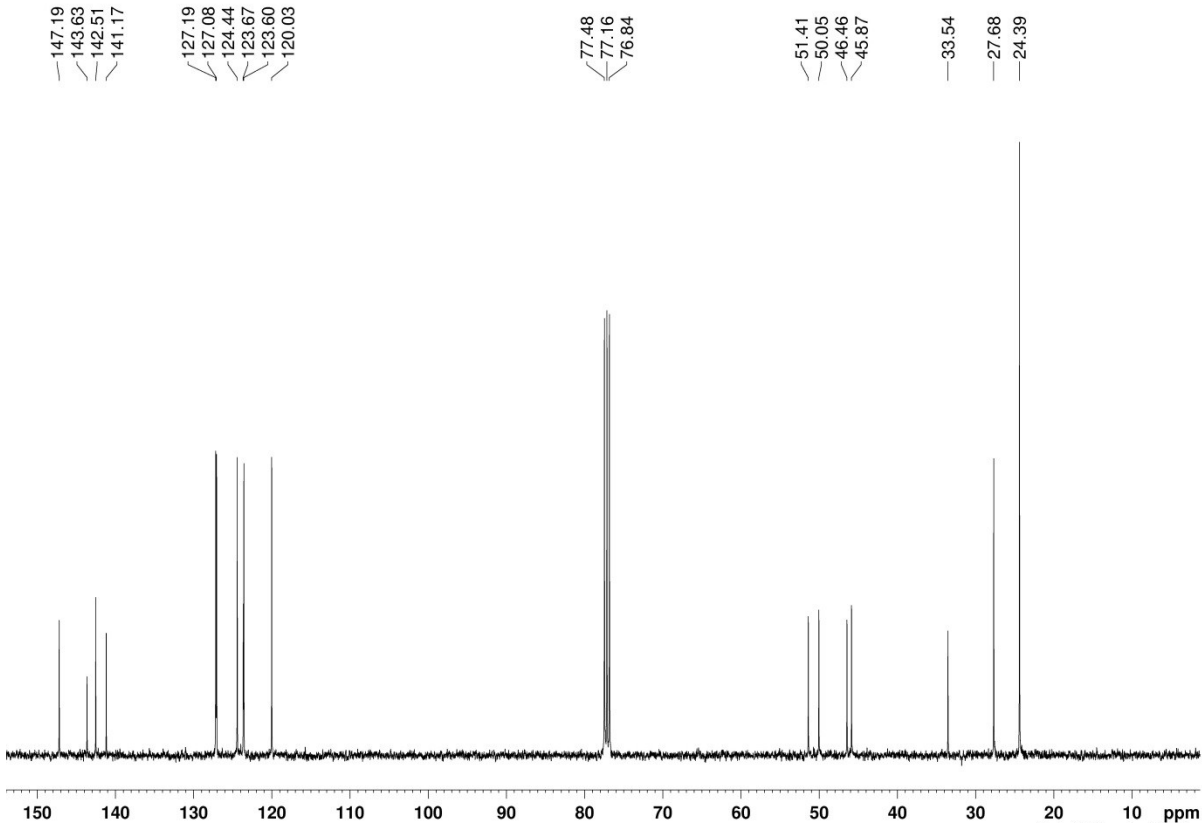


Figure S4. ^{13}C-NMR of **1a** in $CDCl_3$ at 25 °C

1b

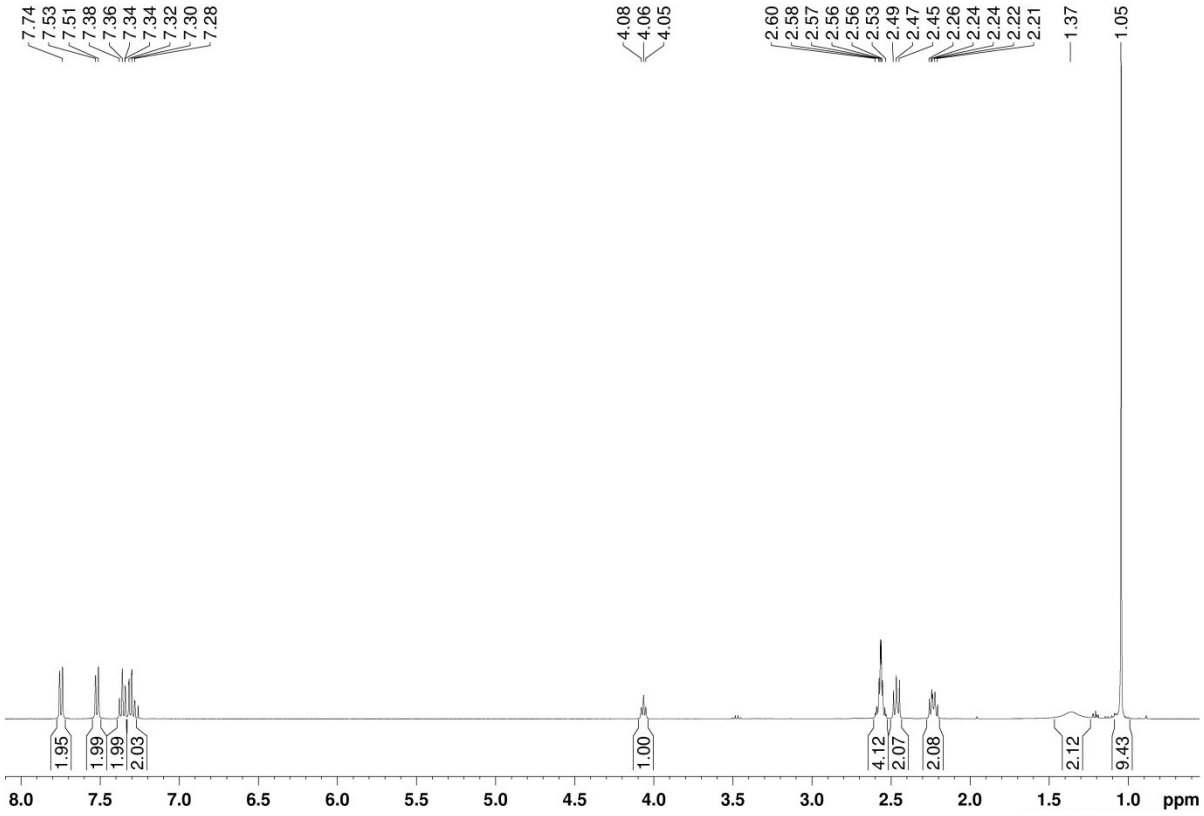


Figure S5. ^{1}H-NMR of **1b** in $CDCl_3$ at 25 °C

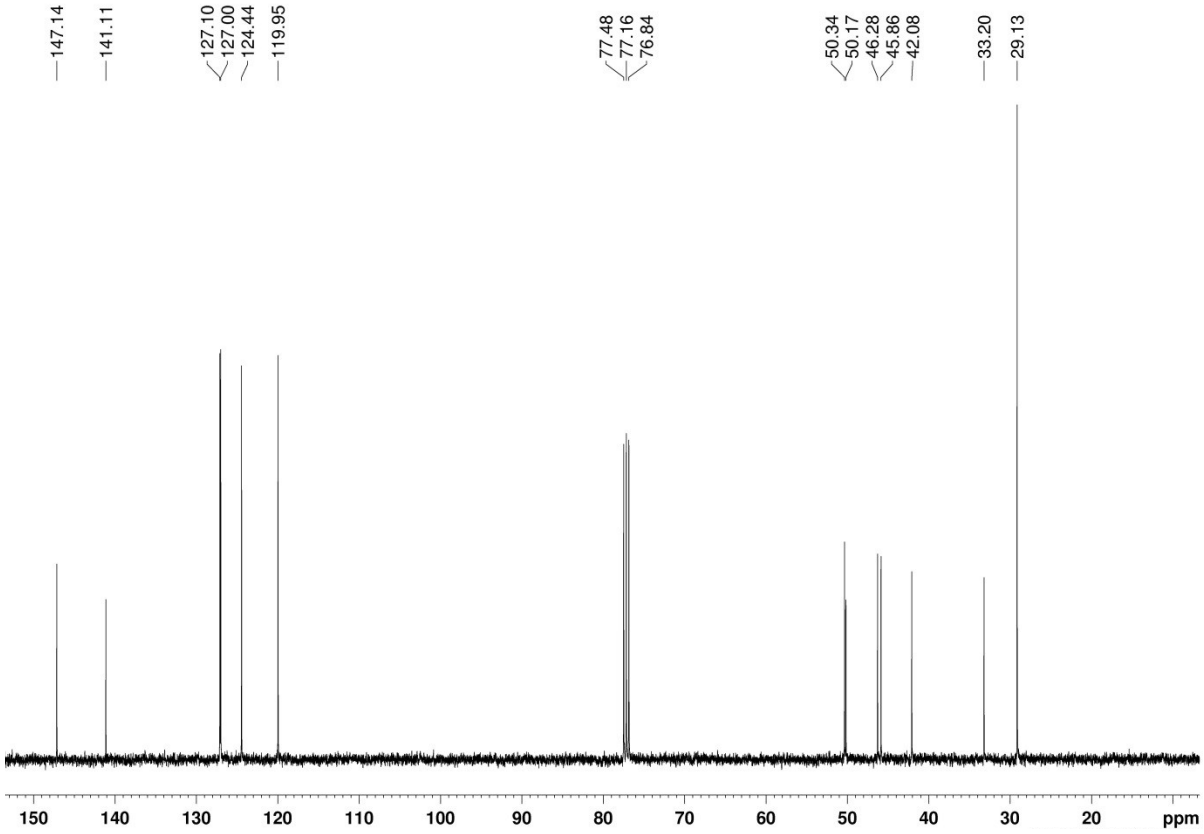


Figure S6. ^{13}C-NMR of **1b** in $CDCl_3$ at 25 °C

1c

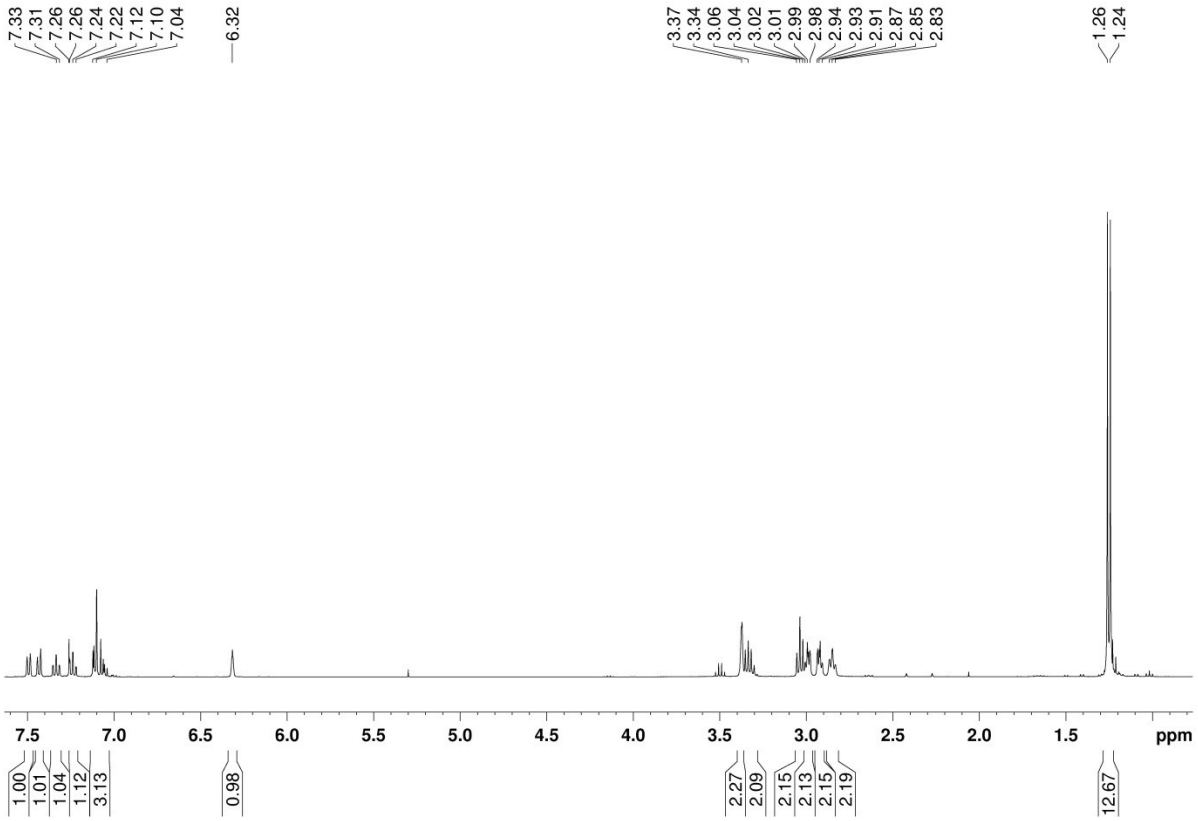


Figure S7. ^{1}H-NMR of **1c** in $CDCl_3$ at 25 °C

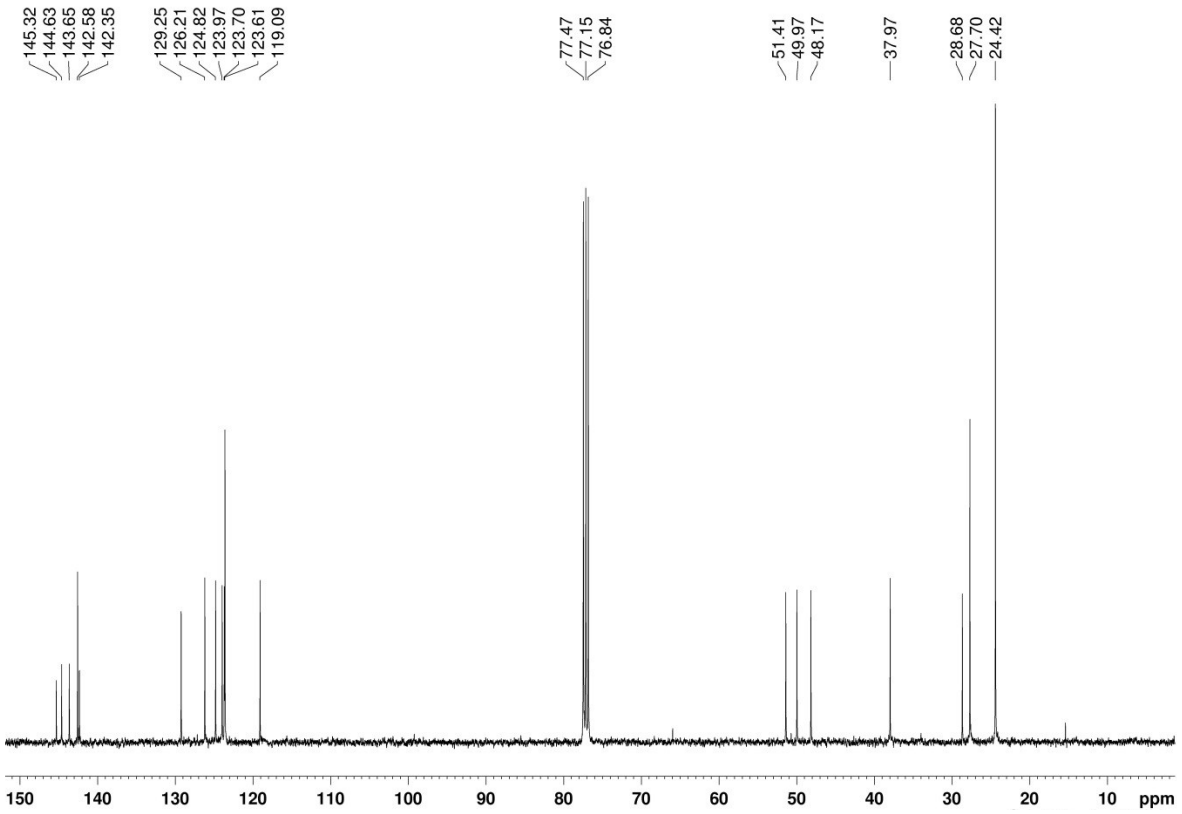


Figure S8. ^{13}C-NMR of **1c** in $CDCl_3$ at 25 °C

1d

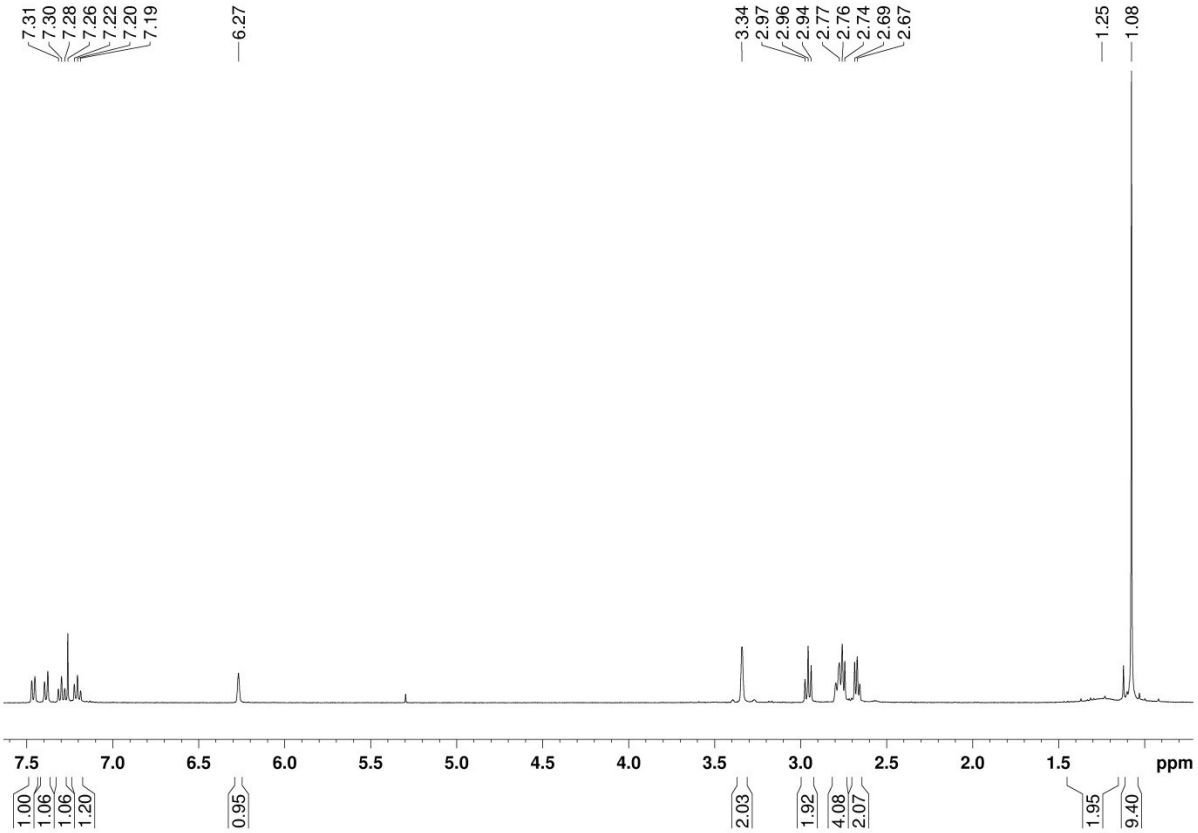


Figure S9. ^{1}H-NMR of **1d** in $CDCl_3$ at 25 °C

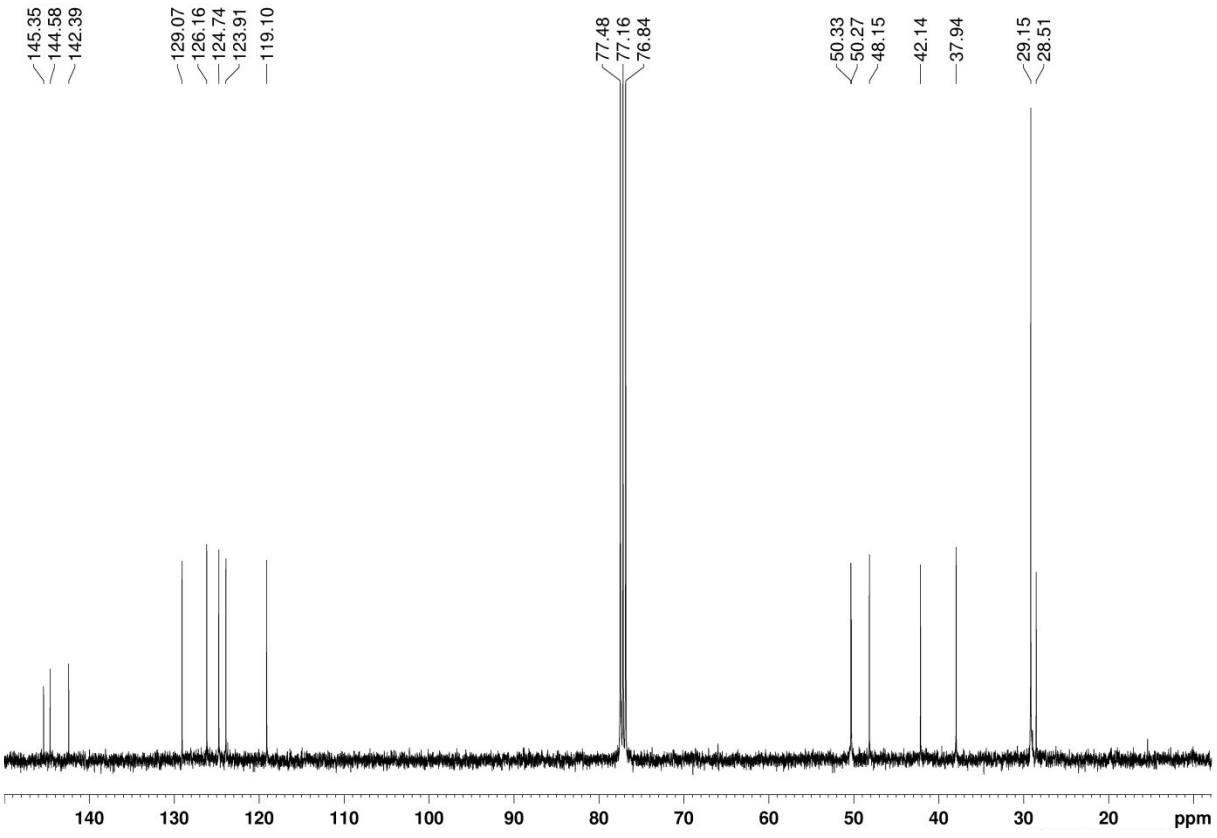


Figure S10. ^{13}C-NMR of **1d** in $CDCl_3$ at 25 °C

2

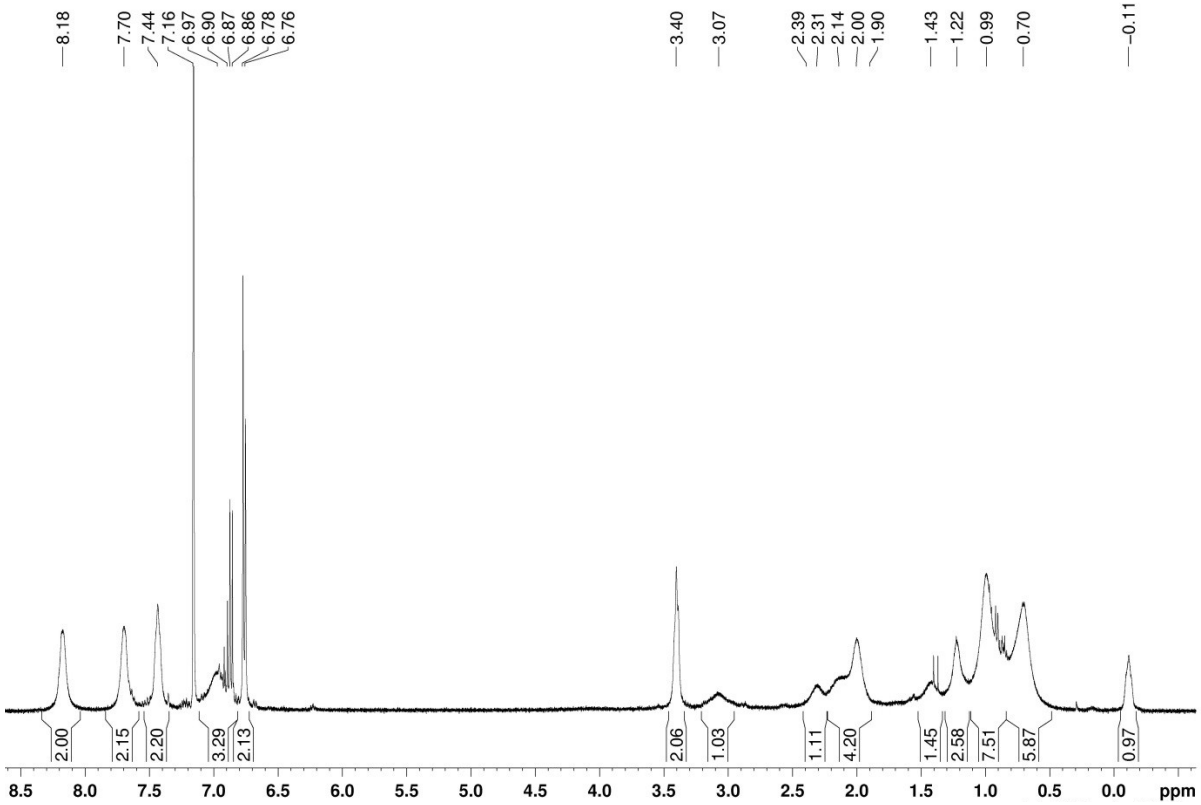


Figure S11. ^{1}H-NMR of **2** in C_6D_6 at 25 °C

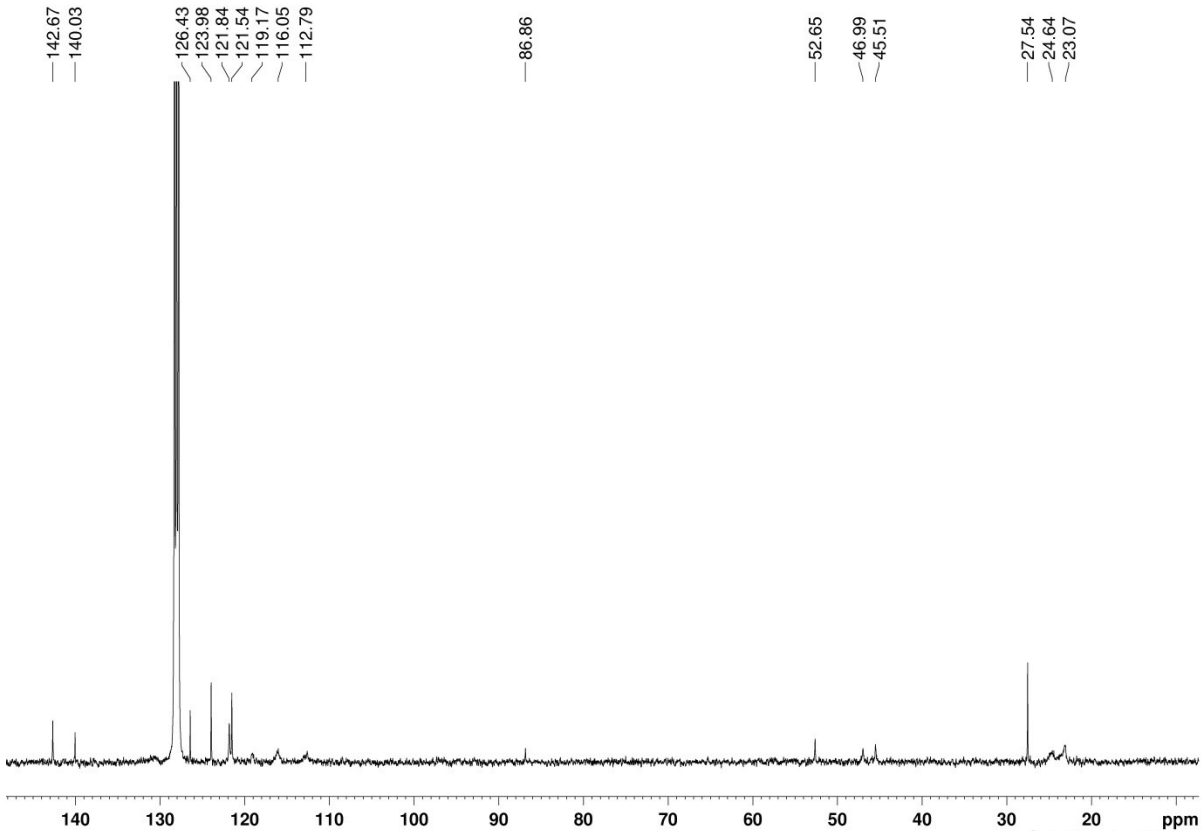


Figure S12. ^{13}C-NMR of **2** in C_6D_6 at 25 °C

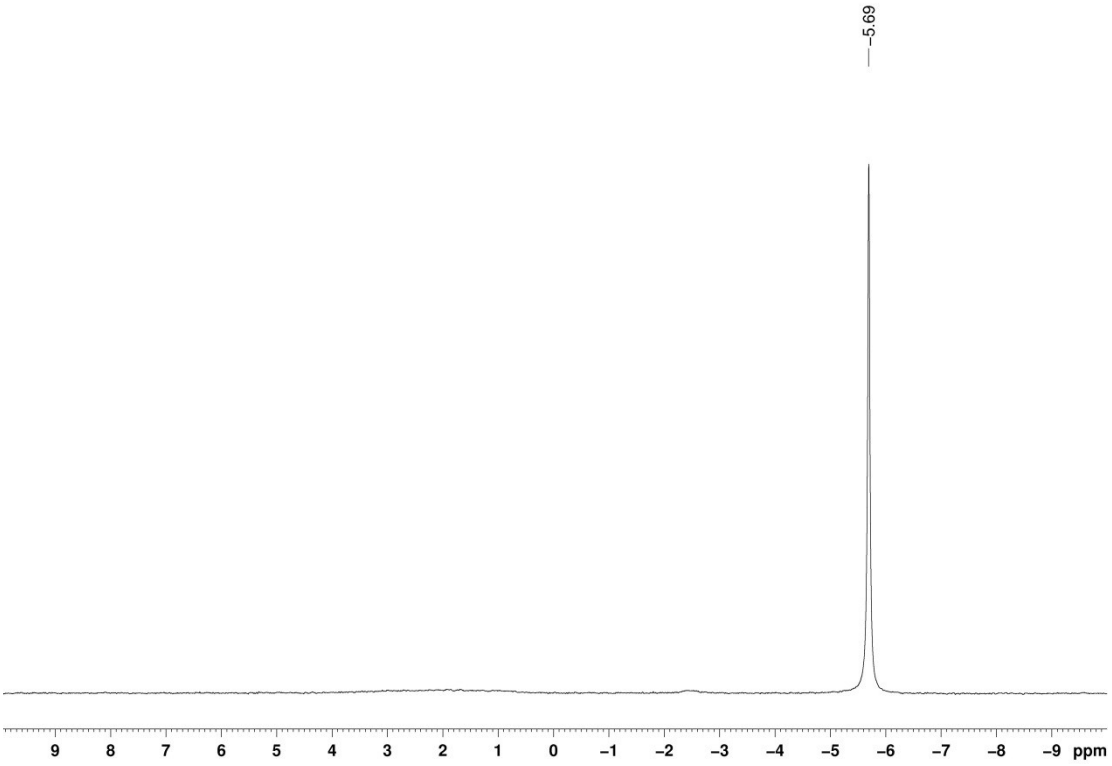


Figure S13. ^{7}Li-NMR of **2** in C_6D_6 at 25 °C

Li{κ^5-($C_{13}H_9$)C_2H_4N(H)C_2H_4N(H)Dipp}$(THF)_n$, [2(thf)$_n$]

A drop of dry THF was added to a sample of **2** in C_6D_6. A species with sharp signals at room temperature was observed by NMR spectroscopy. **^{1}H-NMR (400 MHz, C_6D_6)**: δ 8.40 (d, $^3J_{HH}$ = 7.6 Hz, 2H, Ar_{Fl} H), 7.69 (d, $^3J_{HH}$ = 8.2 Hz, 2H, Ar_{Fl} H), 7.44 (m, 2H, Ar_{Fl} H), 7.03 (m, 2H, Ar_{Fl} H), 6.98 (s, 3H, Diip Ar H), 3.45 (m, 2H, CH_2), 3.06 (sept, $^3J_{HH}$ = 6.8 Hz, 2H, Diip CH), 2.86 (m, 2H, CH_2), 2.60 (m, 1H, NH), 2.49 (m, 2H, CH_2), 2.13 (m, 2H, CH_2), 1.15 (d, $^3J_{HH}$ = 6.7 Hz, 12H, Diip CH_3), 0.71 (m, 1H, NH). **^{13}C-NMR (100 MHz, C_6D_6)**: δ 142.6, 141.7, 133.2, 127.6, 126.0, 124.4, 122.1, 121.2, 115.2, 112.1, 86.5, 52.4, 49.1, 46.8, 28.0, 24.8, 24.4. **^{7}Li-NMR (155.4 MHz, C_6D_6)**: δ -2.65.

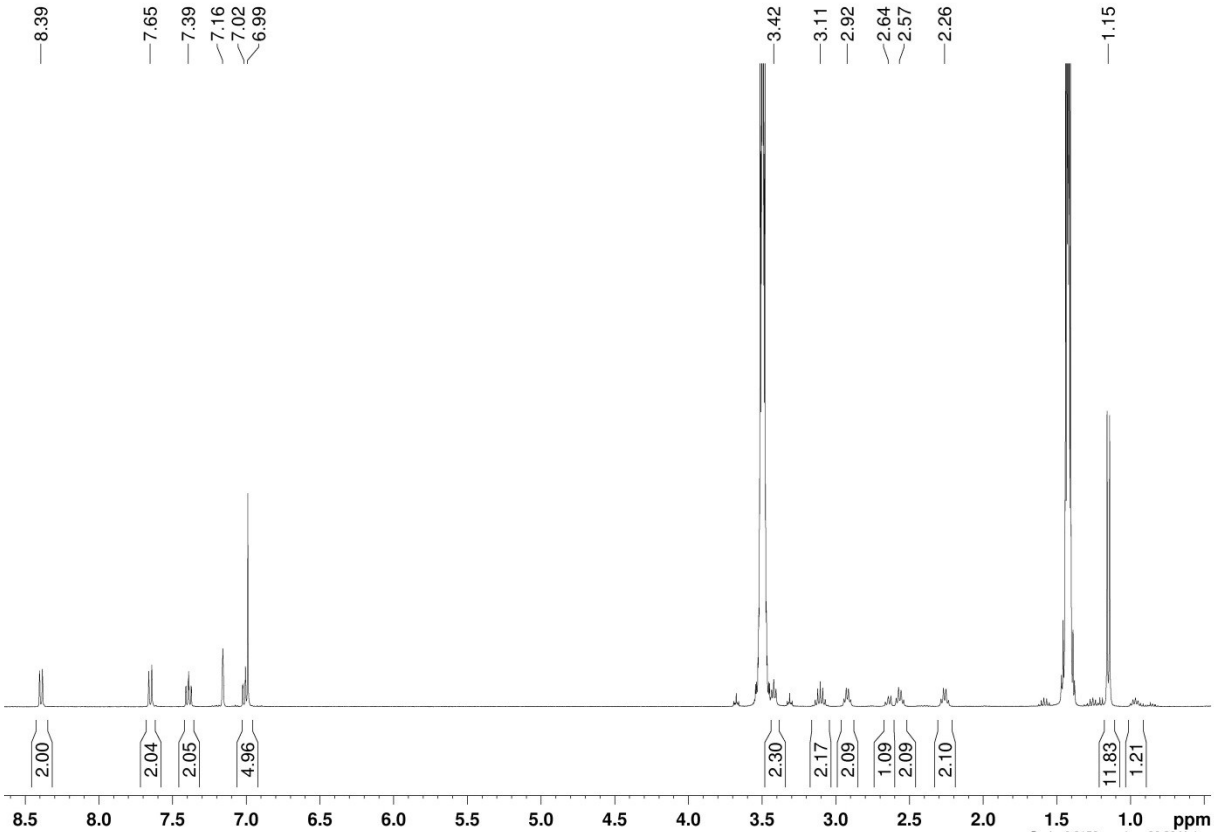


Figure S14. ^{1}H-NMR of **2·THF** in C_6D_6 at 25 °C

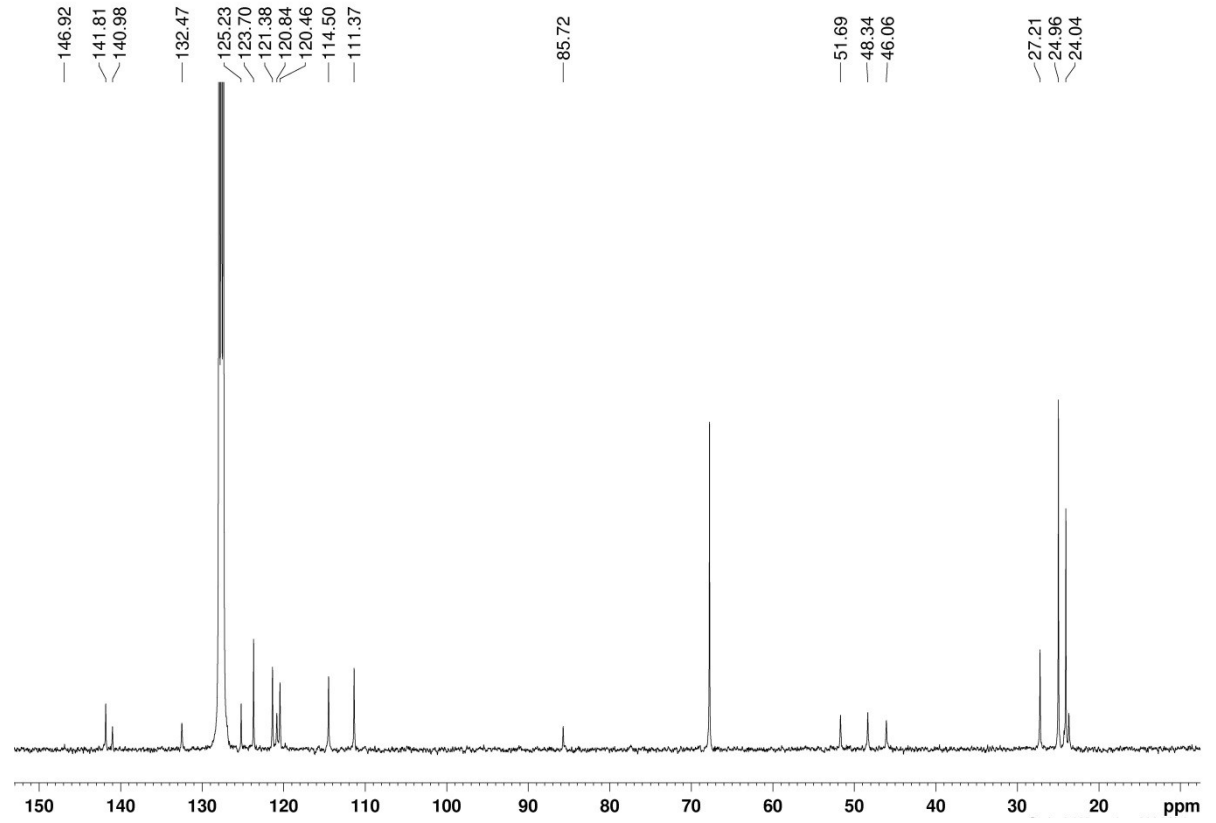


Figure S15. ^{13}C-NMR of **2·THF** in C_6D_6 at 25 °C

Variable temperature NMR study of compound 2

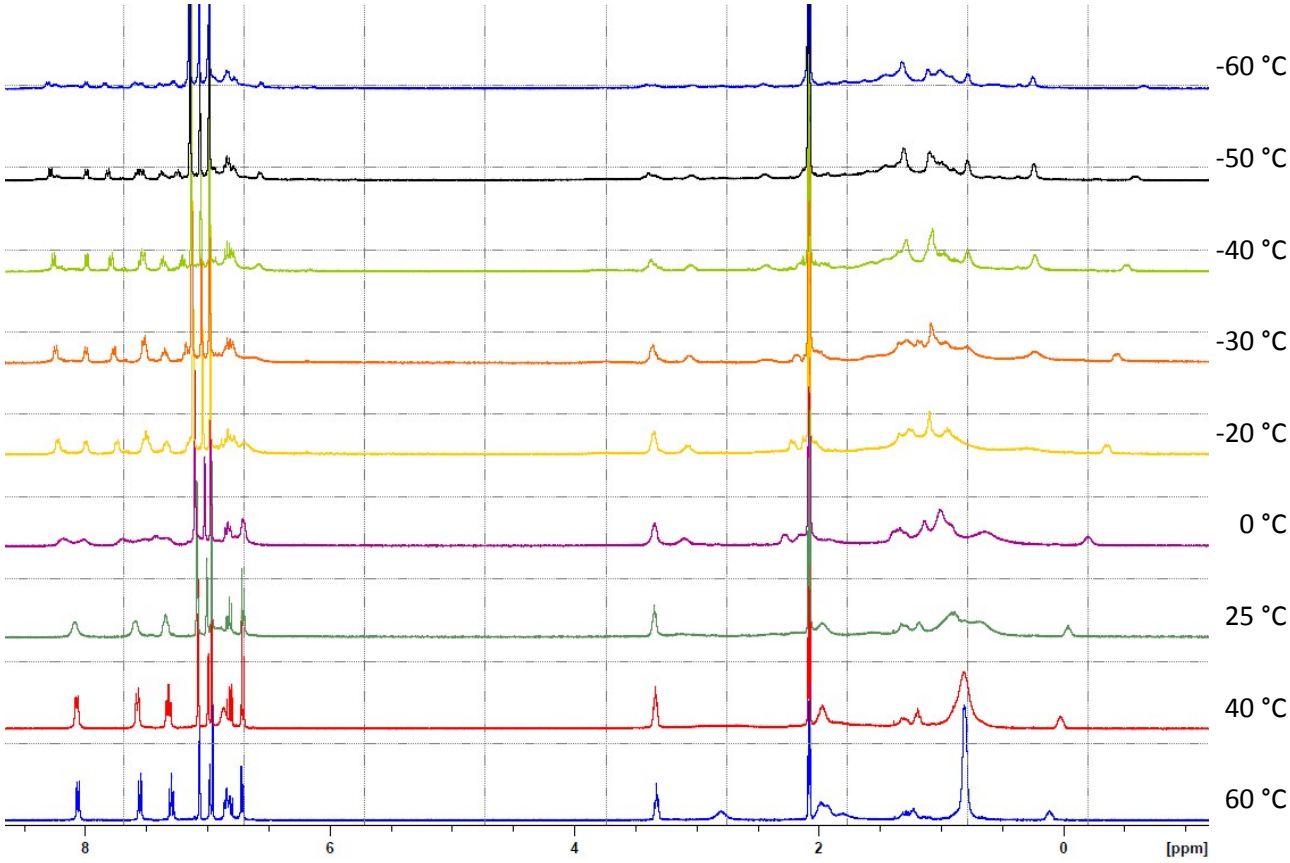


Figure S16. Variable temperature NMR study of Li{κ^5-($C_{13}H_9$)C_2H_4N(H)C_2H_4N(H)Dipp} (**2**) in d^8-toluene**.**

3

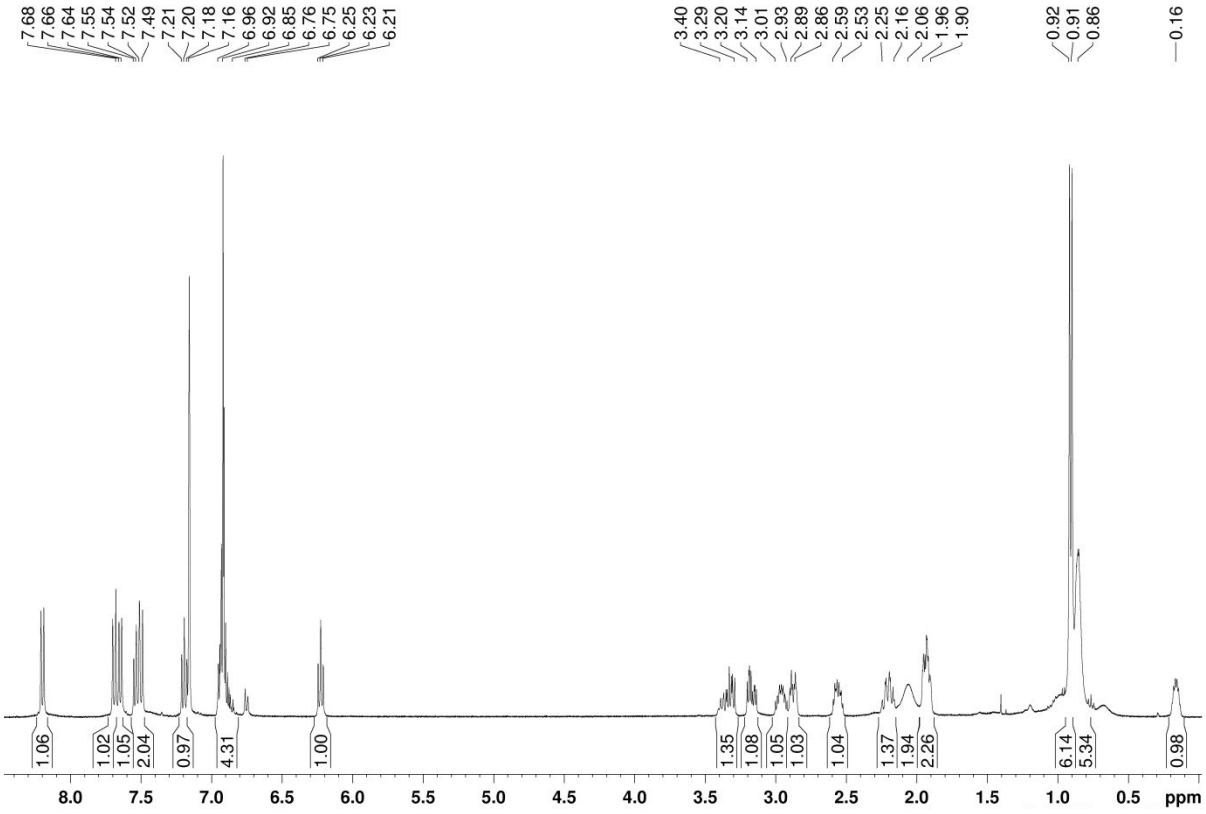


Figure S17. ^{1}H-NMR of **3** in C_6D_6 at 25 °C

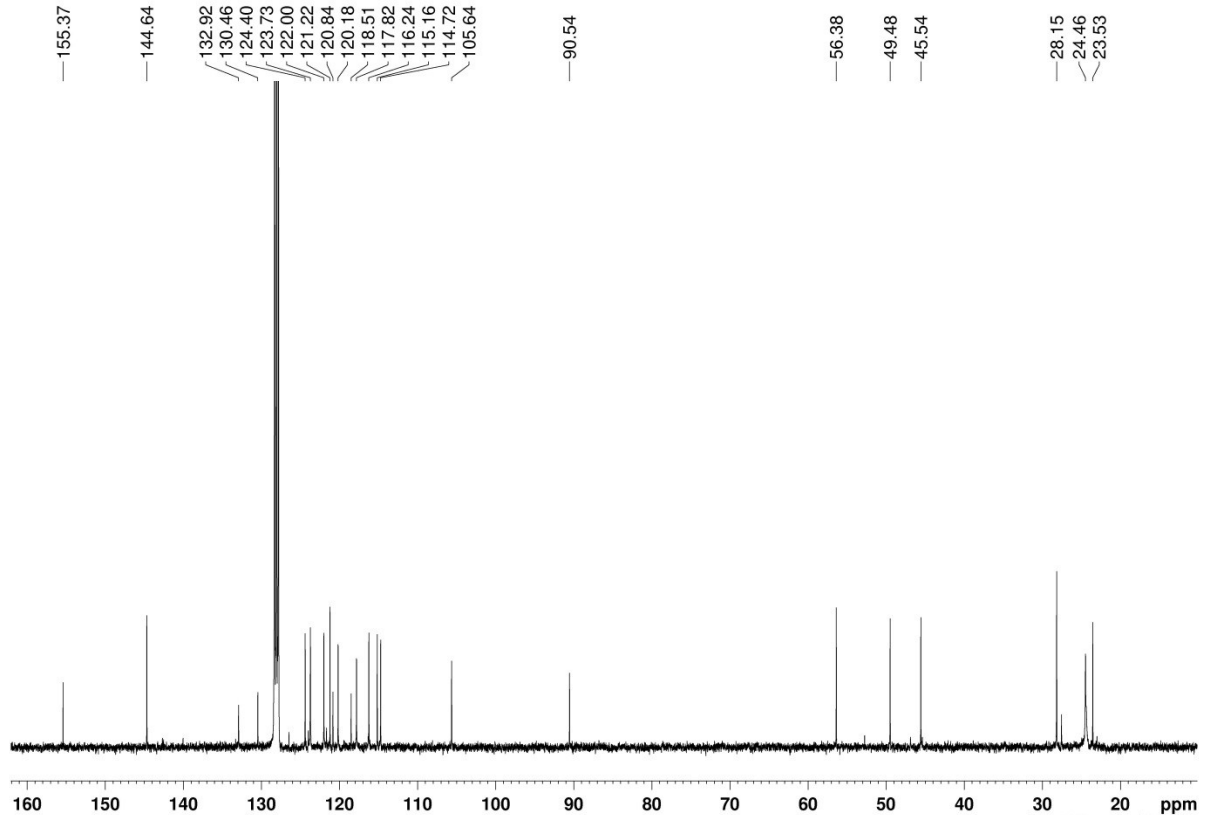


Figure S18. ^{13}C-NMR of **3** in C_6D_6 at 25 °C

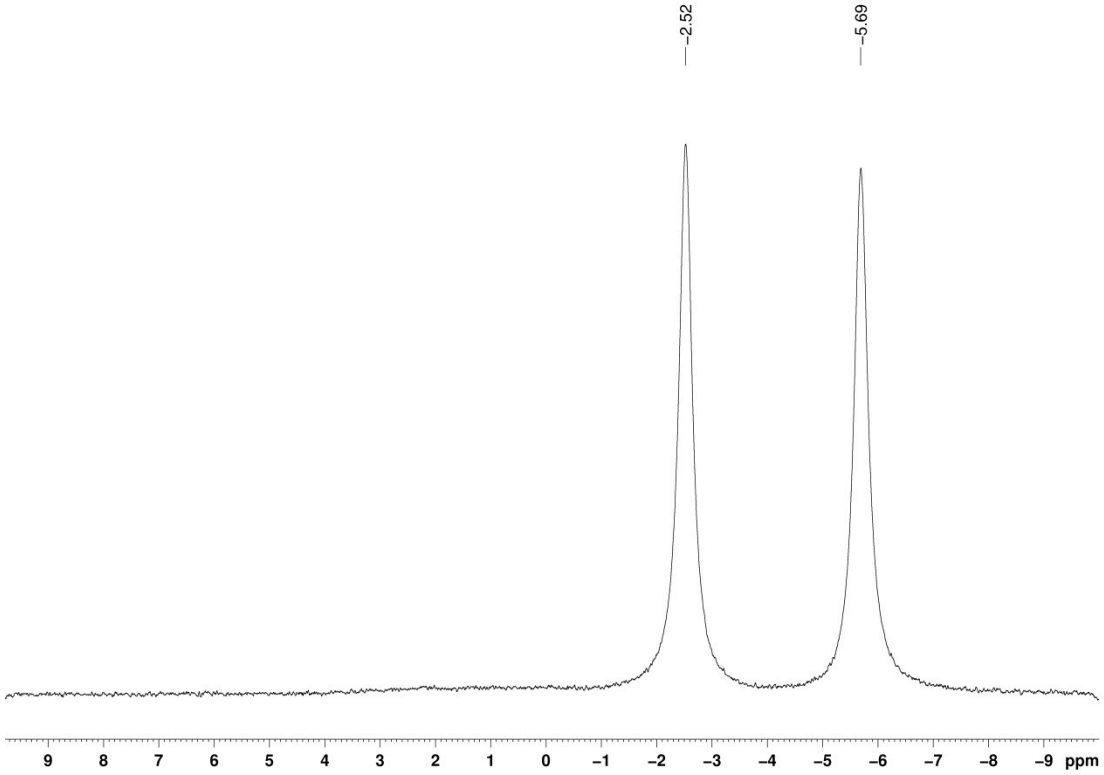


Figure S19. ^{7}Li-NMR of **3** in C_6D_6 at 25 °C

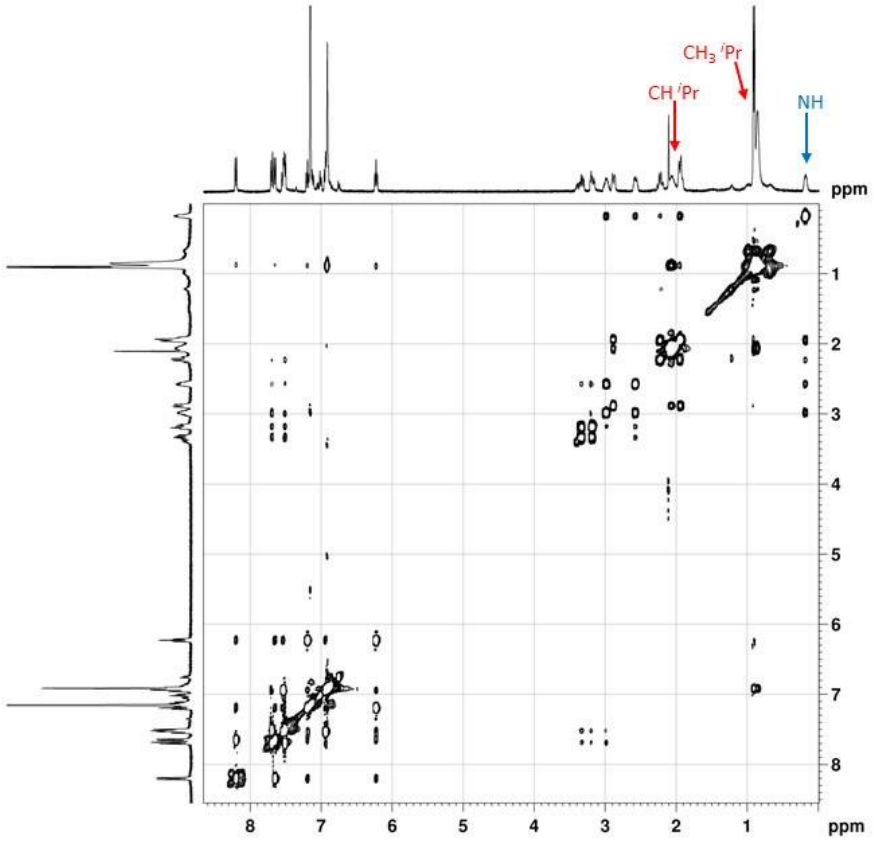


Figure S20. {^{1}H, ^{1}H} - NOESY of **3** in C_6D_6 at 25 °C. As indicated, the NH signal does not present crosspeaks with any of the isopropyl resonances from the Dipp fragment.

4

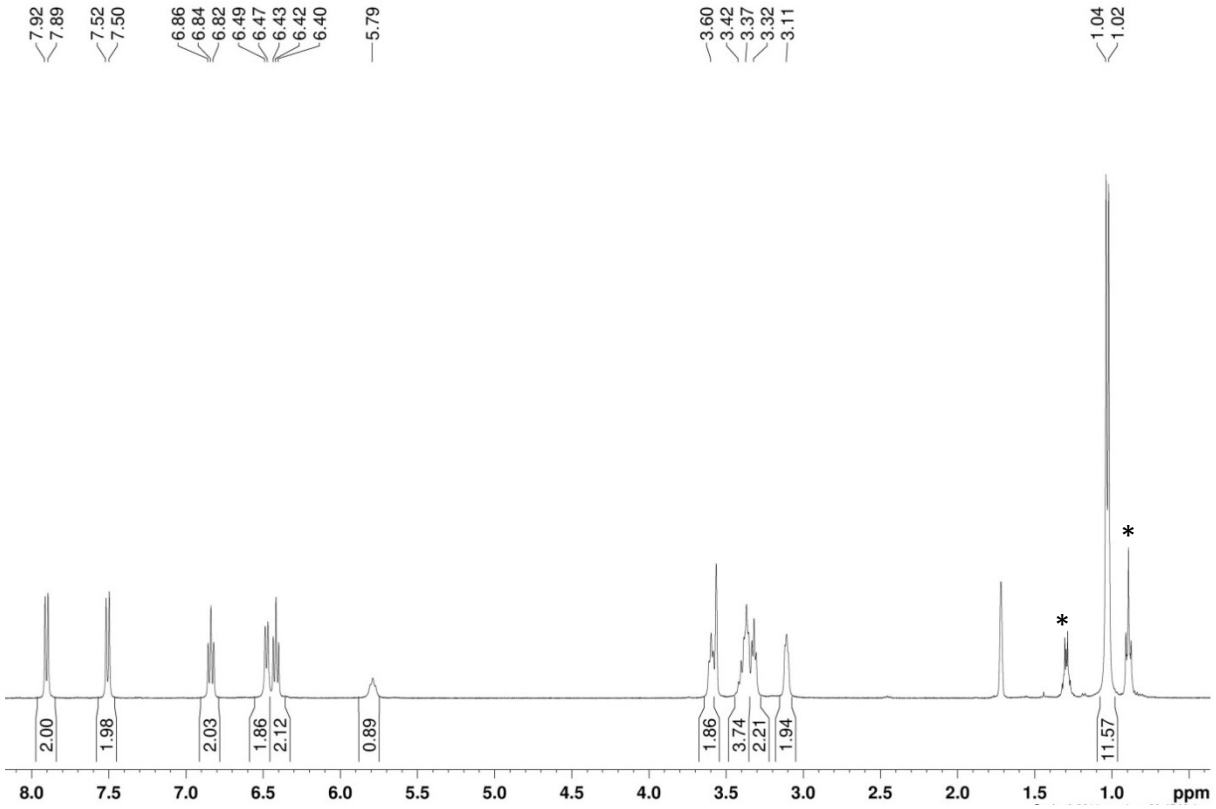


Figure S21. ^{1}H-NMR of **4** in d_8-THF at 25 °C. Signals marked with * arise from traces of hexane.

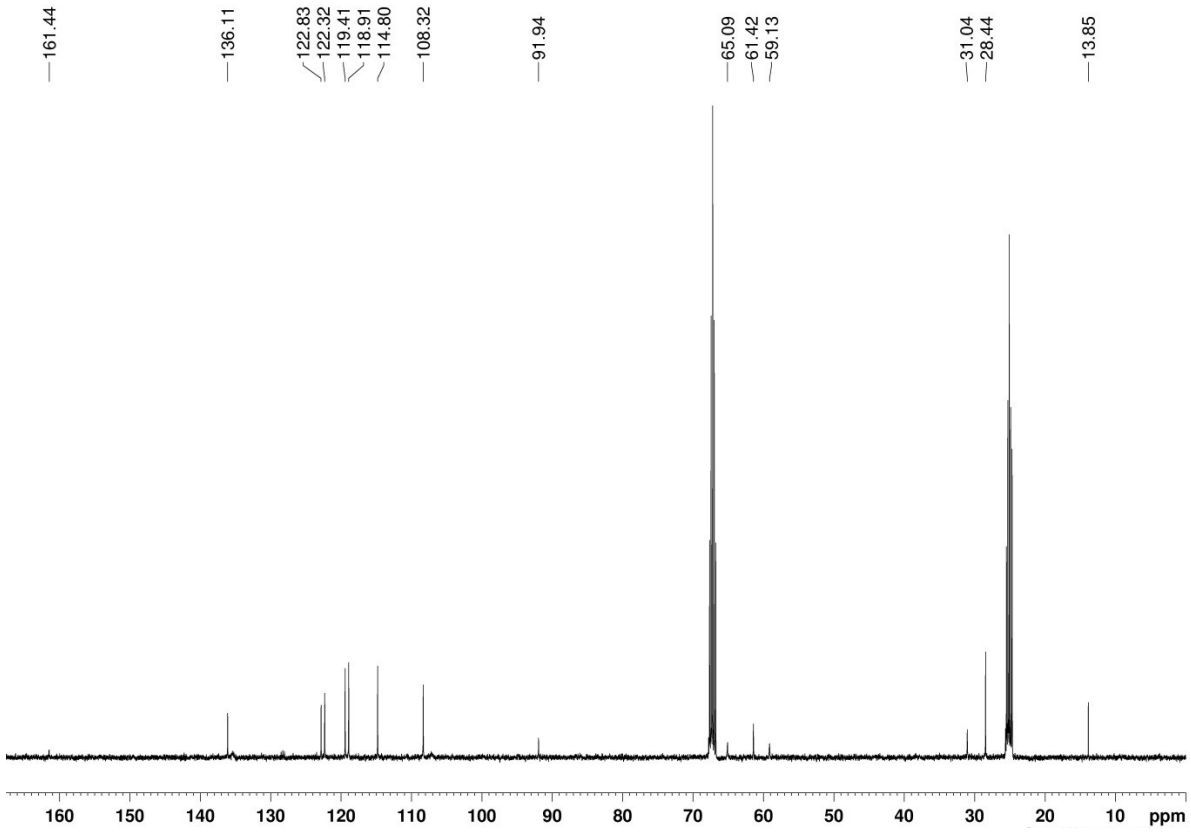


Figure S22. ^{13}C-NMR of **4** in d_8-THF at 25 °C

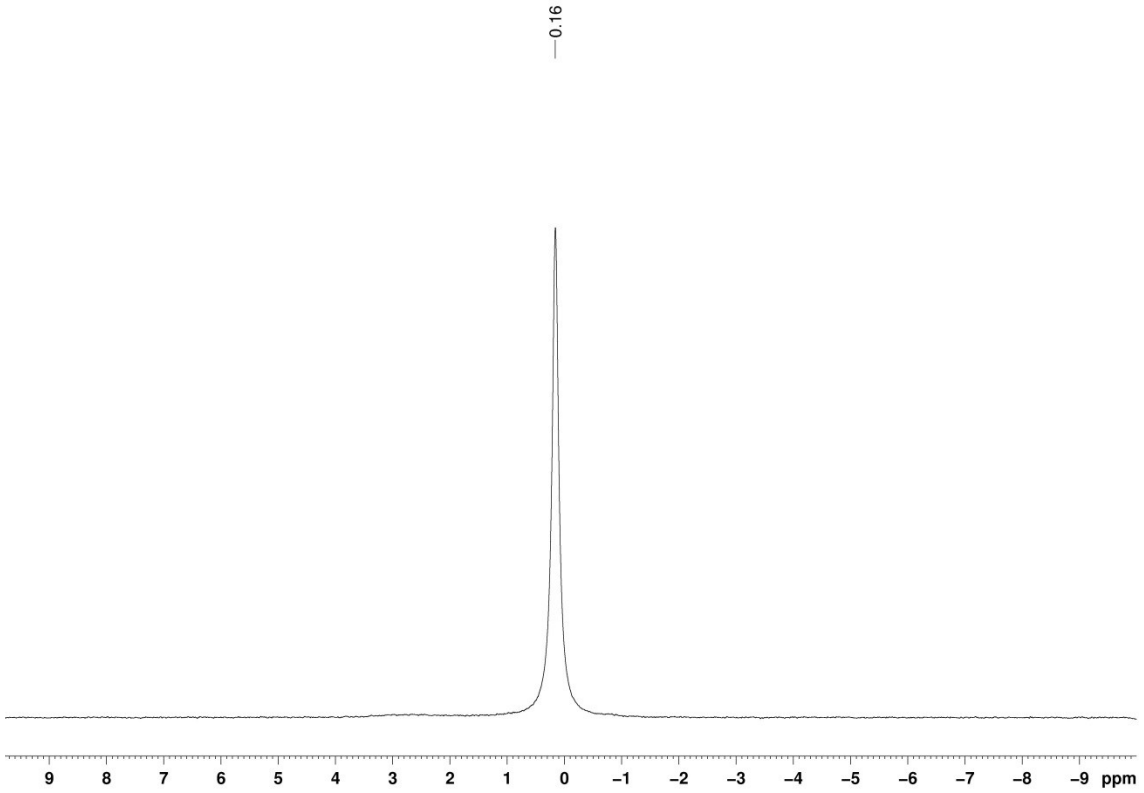


Figure S23. ^{7}Li-NMR of **4** in d_8-THF at 25 °C

5

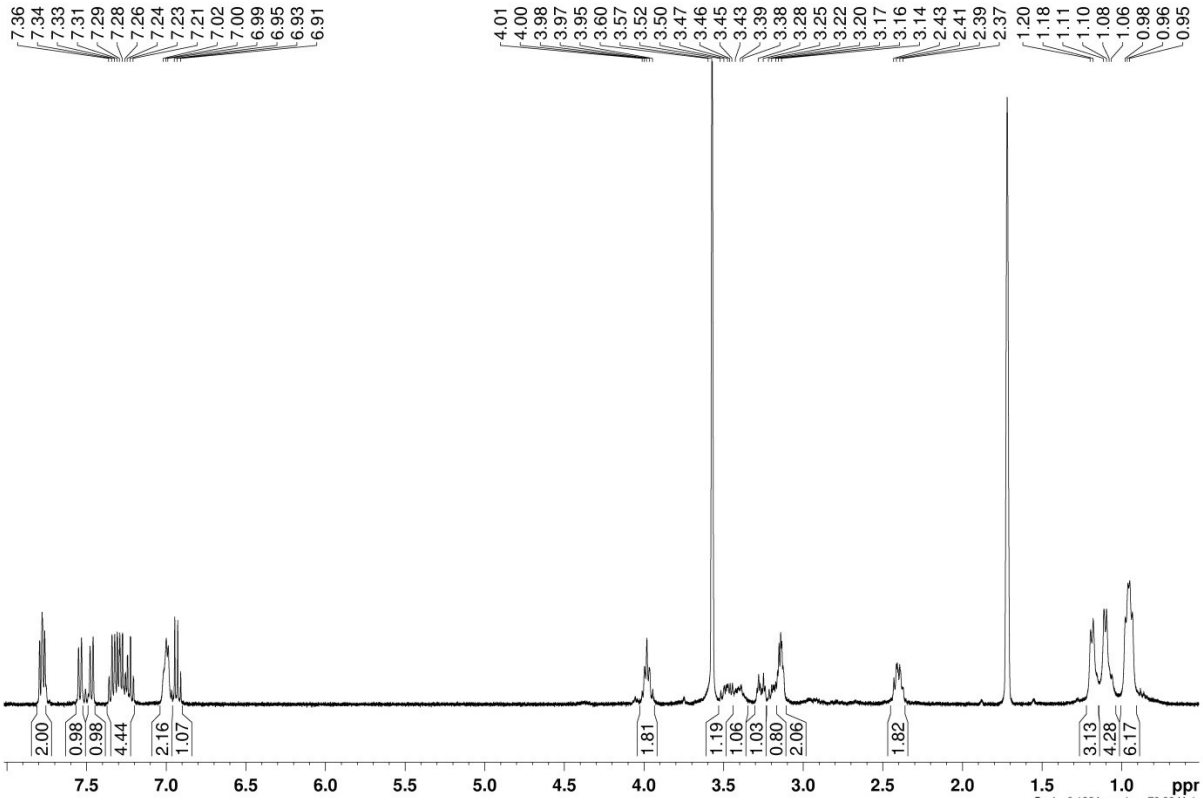


Figure S24. ^{1}H-NMR of **5** in d_8-THF at 25 °C

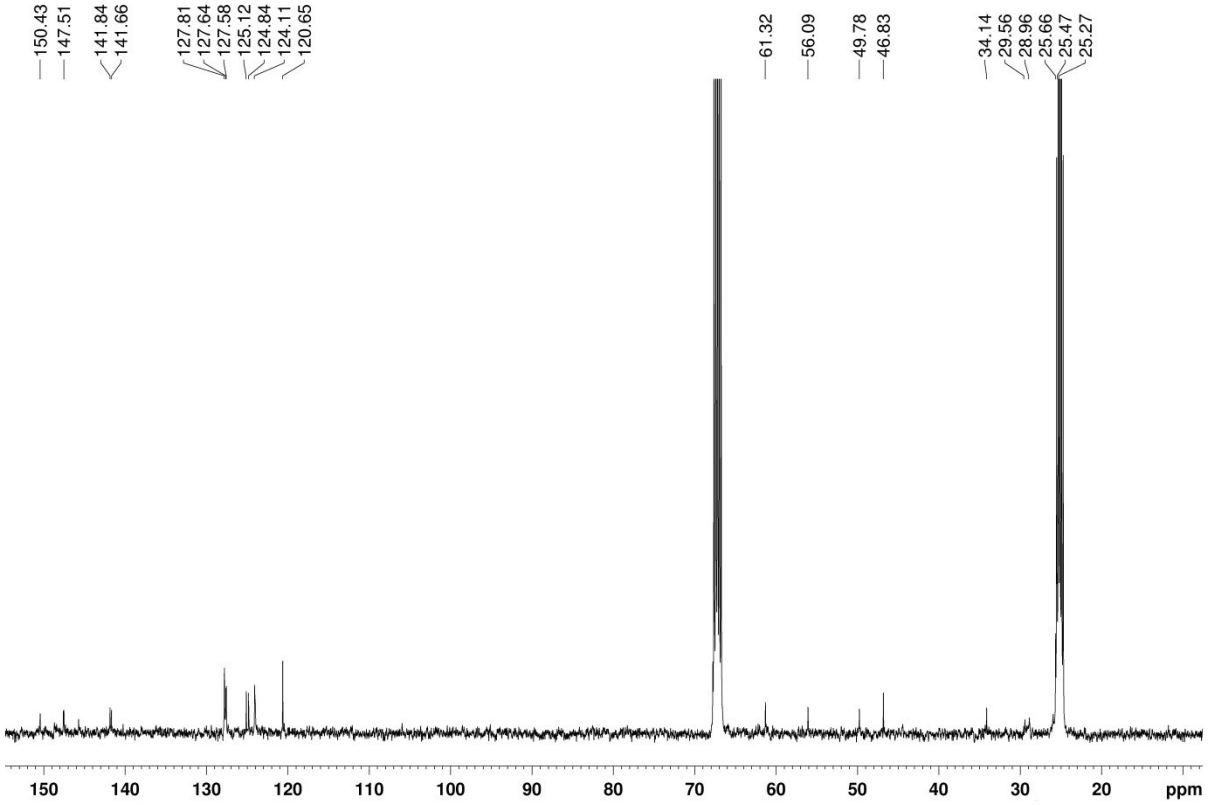


Figure S25. ^{13}C-NMR of **5** in d_8-THF at 25 °C

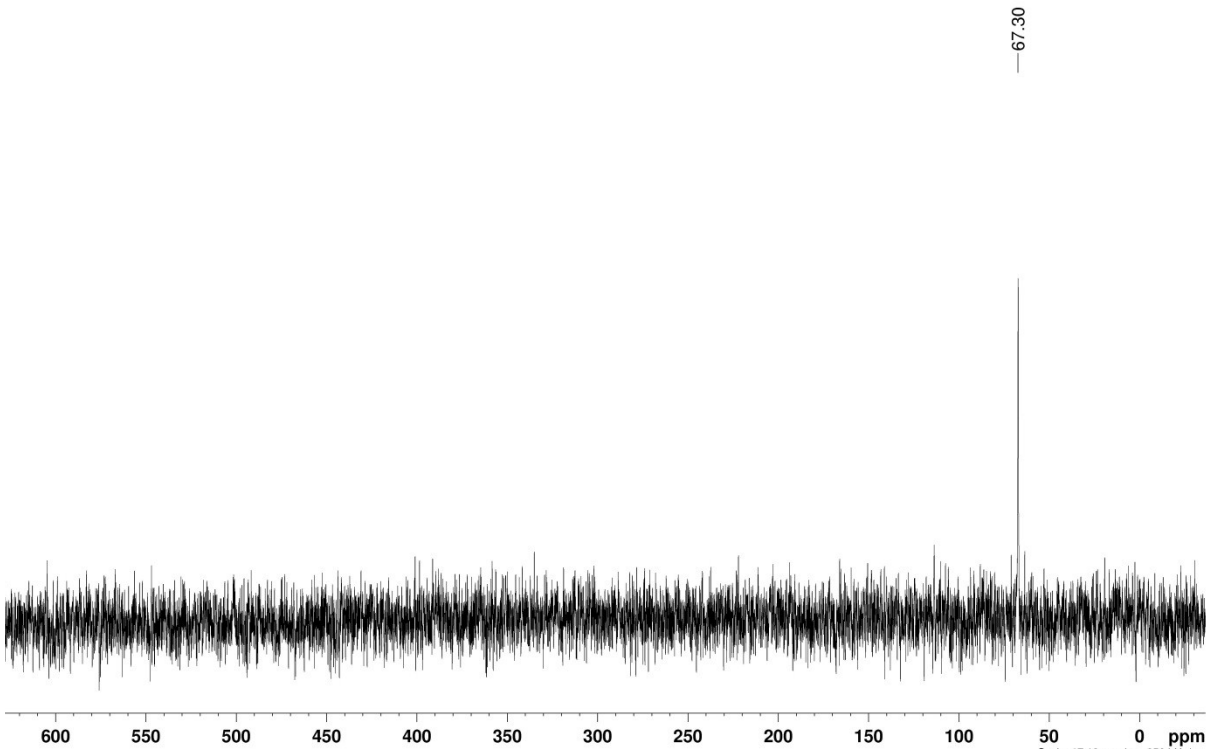


Figure S26. ^{119}Sn-NMR of **5** in d_8-THF at 25 °C

6

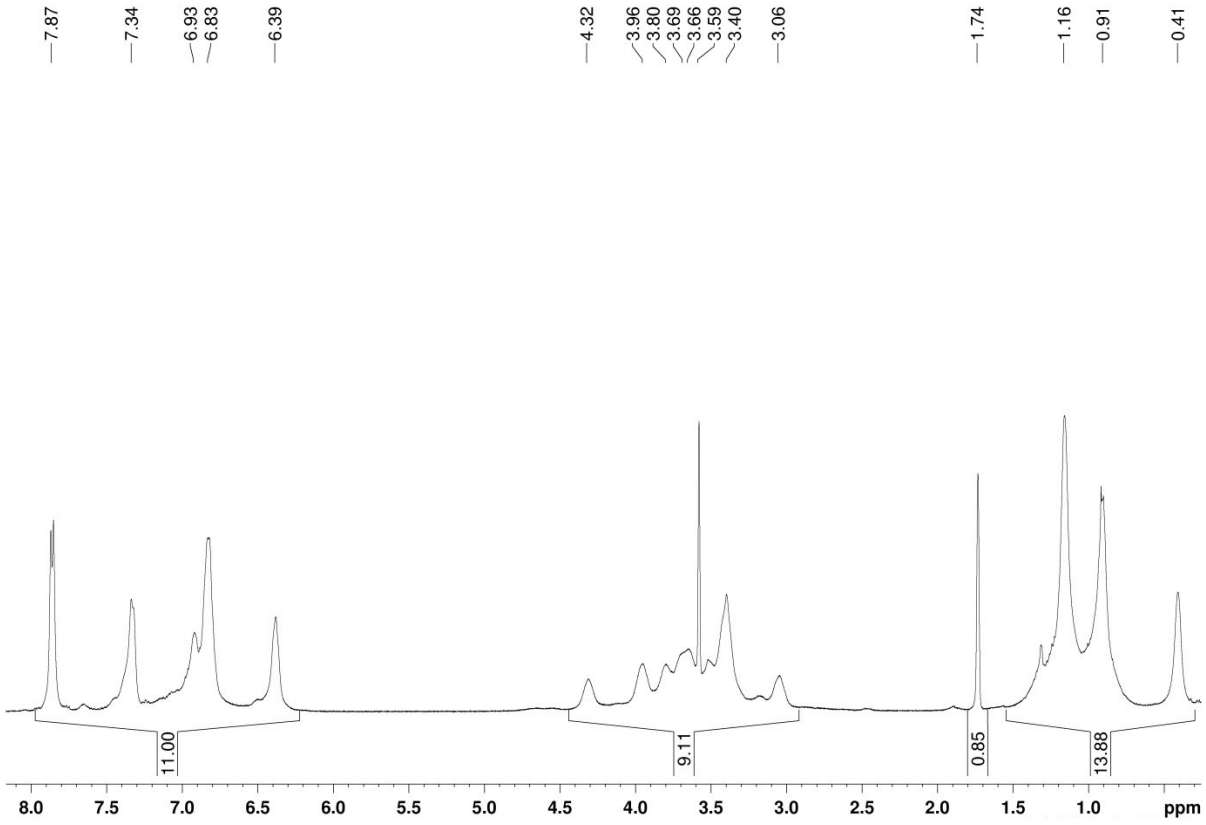


Figure S27. ^{1}H-NMR of **6** in d_8-THF at 25 °C (0.034 mmol ml^{-1})

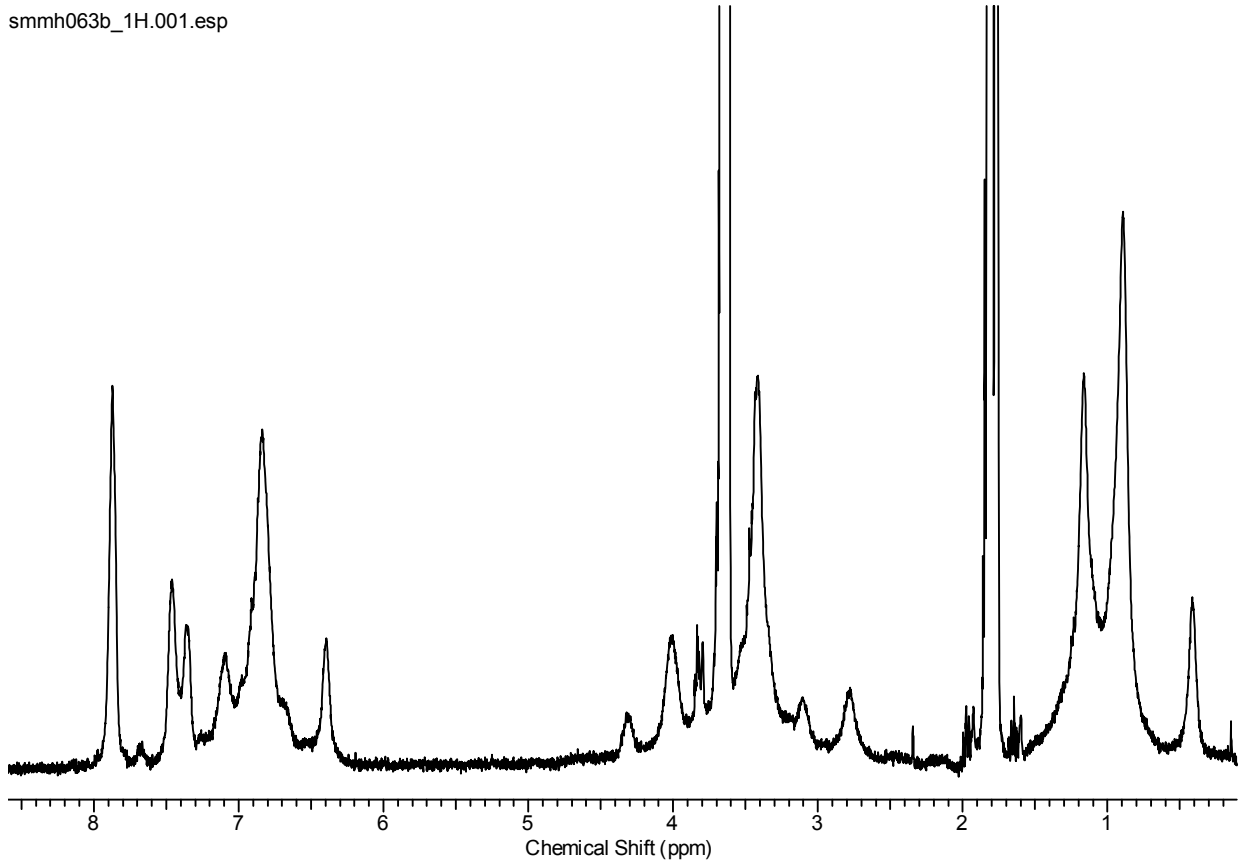


Figure S28. ^{1}H-NMR of **6** in d_8-THF at 25 °C (0.003 mmol ml^{-1})

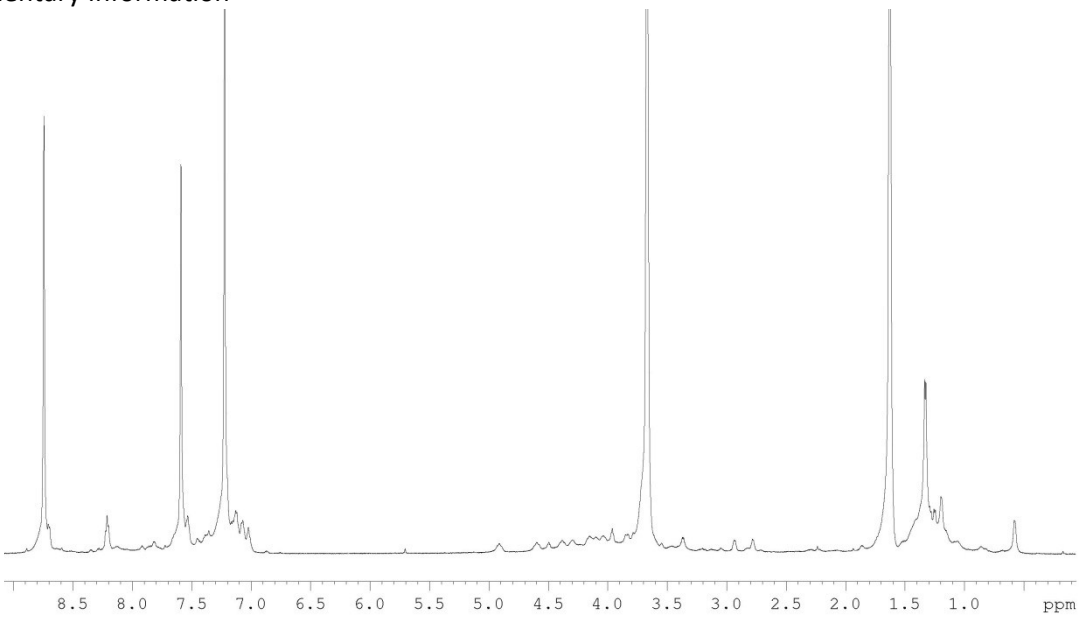


Figure S29. ^{1}H-NMR of **6** at 700MHz in d_5-pyridine at 25 °C. The large solvent resonances are from thf molecules displaced from the [Li(thf)$_4$] cations and residual protio impurities in the d^5-pyridine solvent.

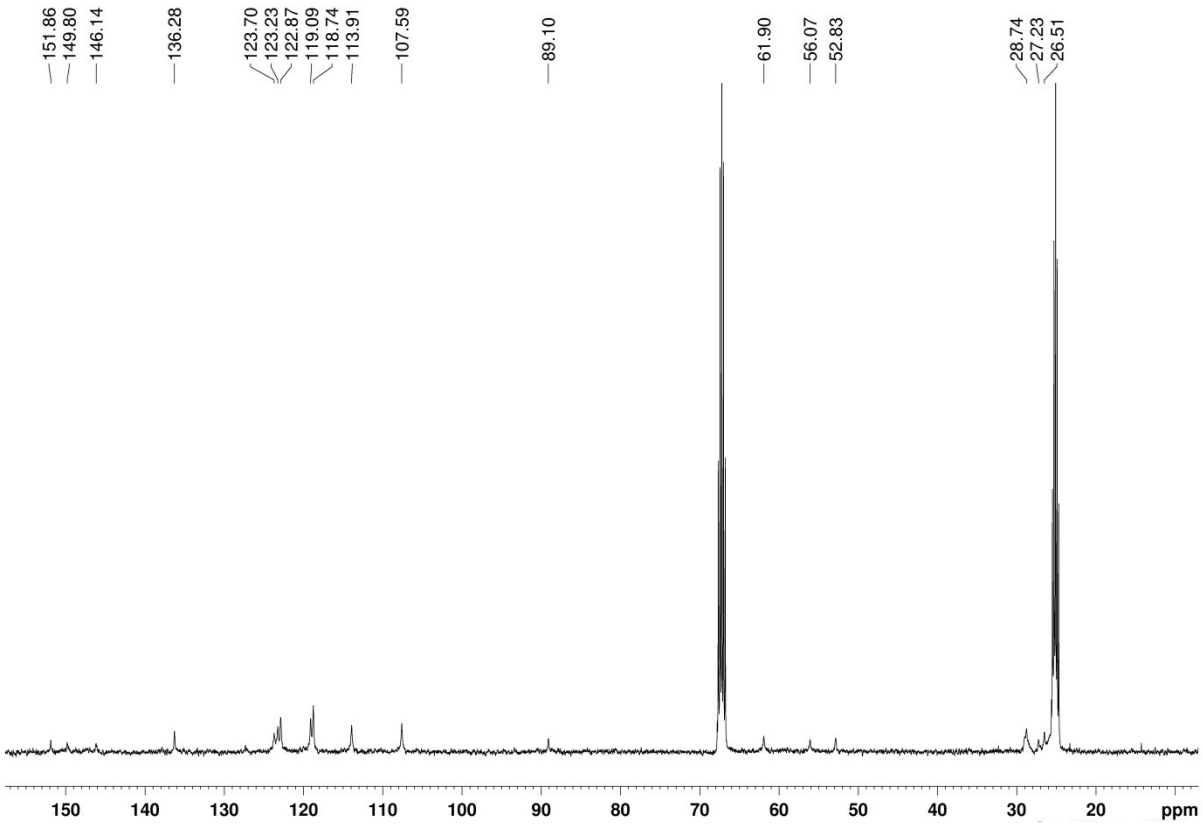


Figure S30. ^{13}C-NMR of **6** in d_8-THF at 25 °C

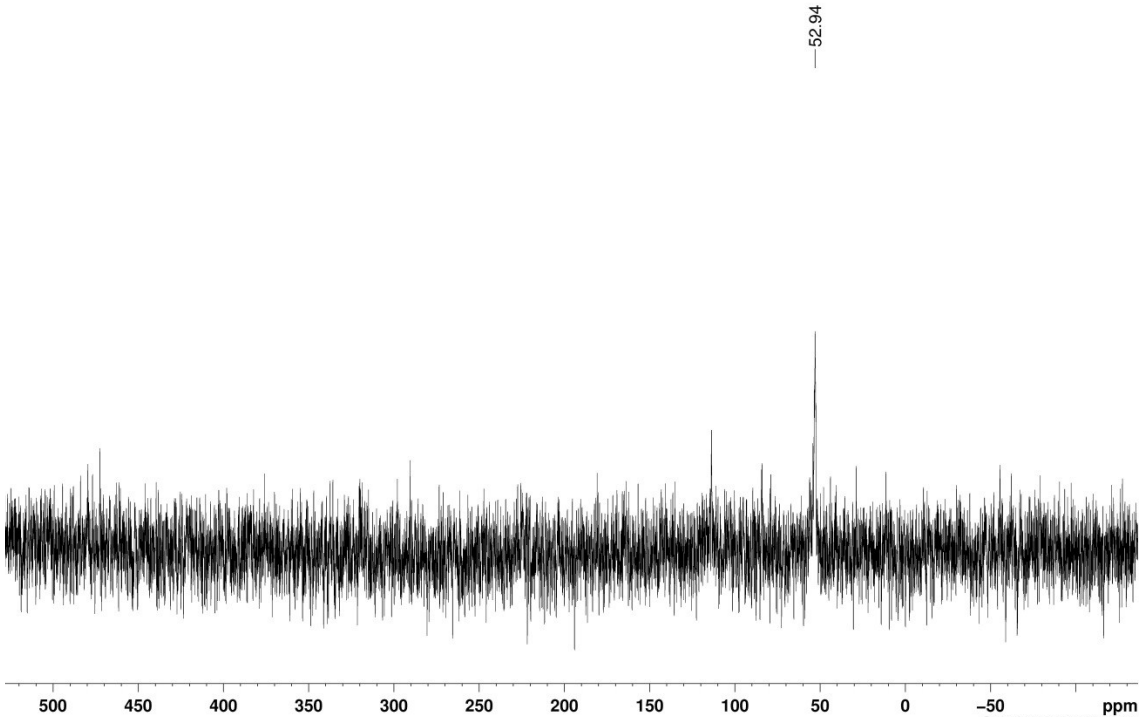


Figure S31. ^{119}Sn-NMR of **6** in d_8-THF at 25 °C (0.034 mmol ml^{-1}). The very weak resonance at 115 ppm is from the thermal decomposition product of **6**.

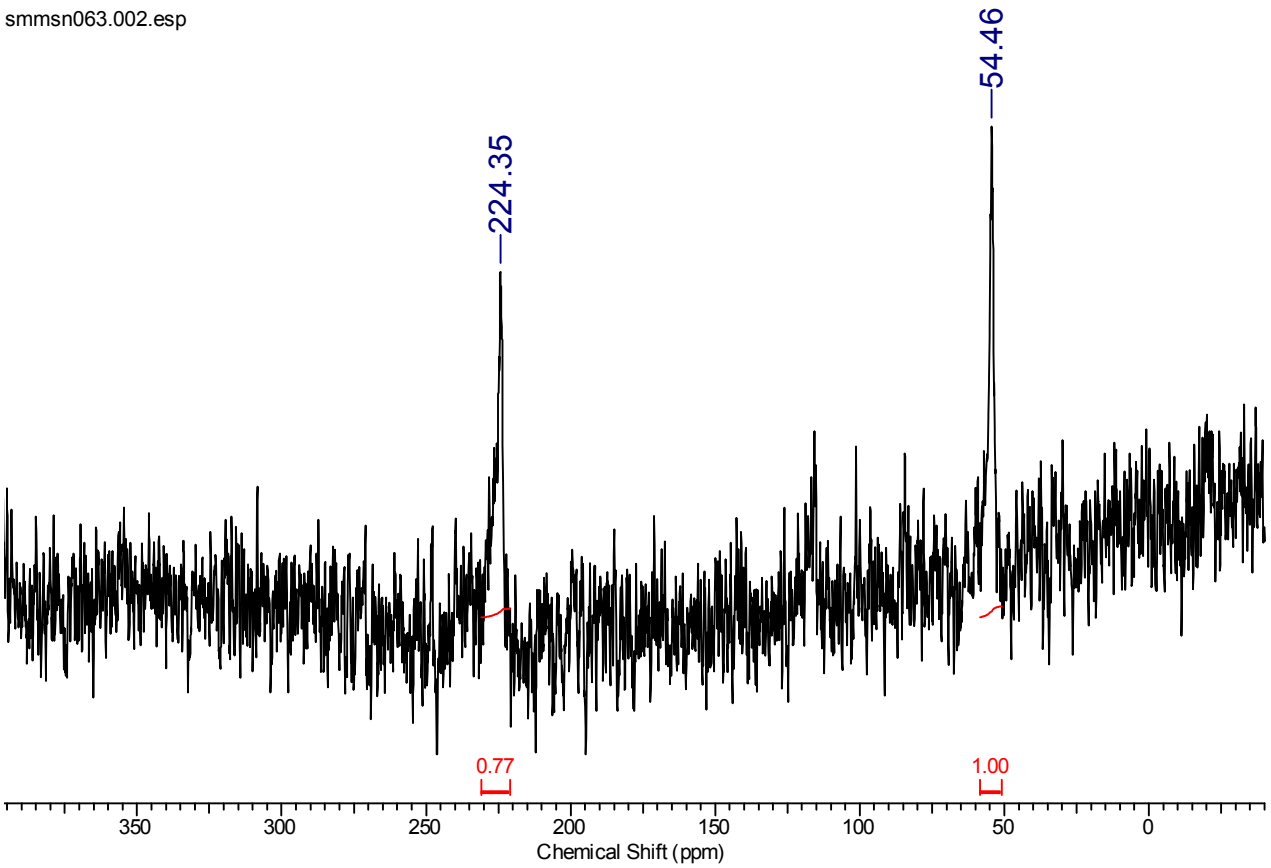


Figure S32. ^{119}Sn-NMR of **6** in d_8-THF at 25 °C (0.003 mmol ml^{-1}). The very weak resonance at 115 ppm is from the thermal thermal decomposition of **6**.

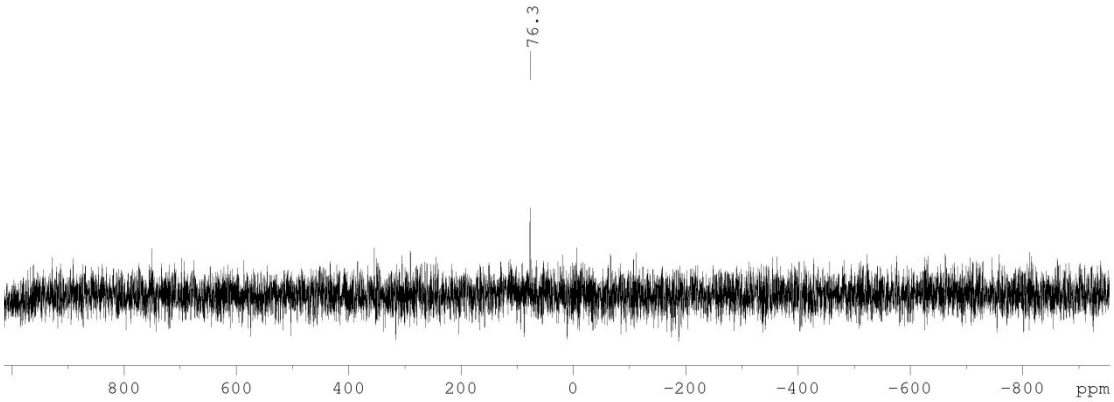


Figure S33. ^{119}Sn-NMR of **6** at 261 MHz (700MHz spectrometer) in d_5-pyridine at 25 °C (0.02 mmol cm^{-3})

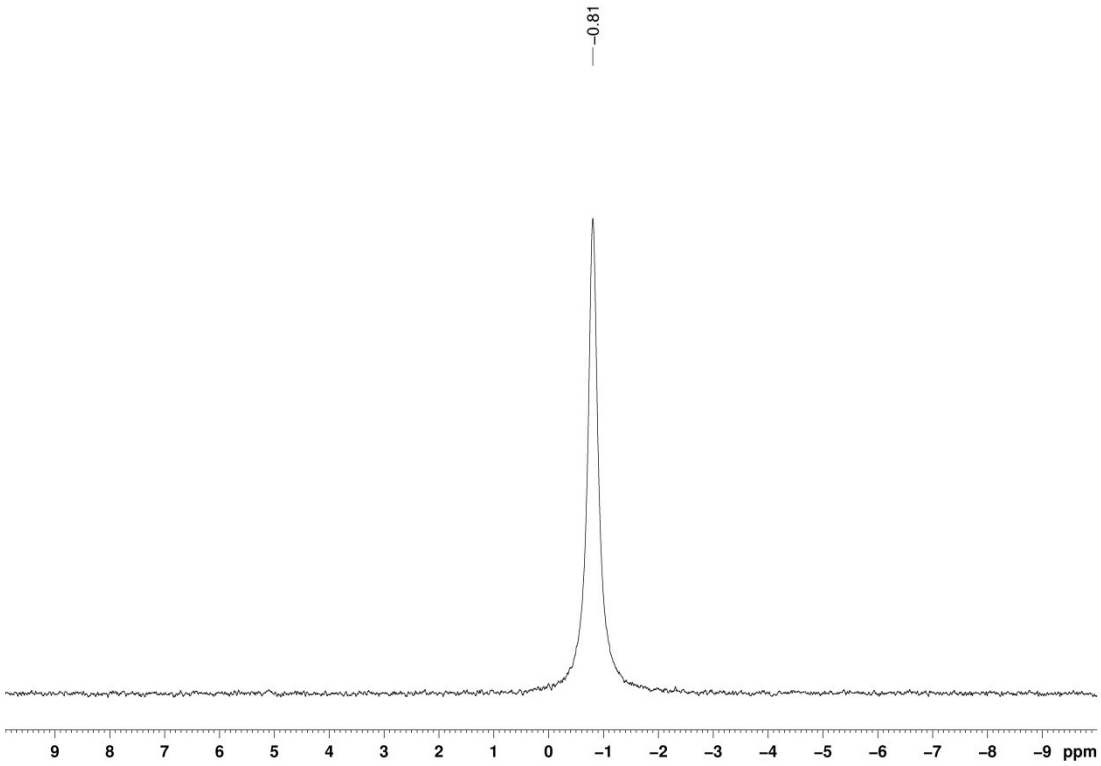


Figure S34. ^{7}Li-NMR of **6** in d_8-THF at 25 °C

Variable temperature NMR spectra for 6

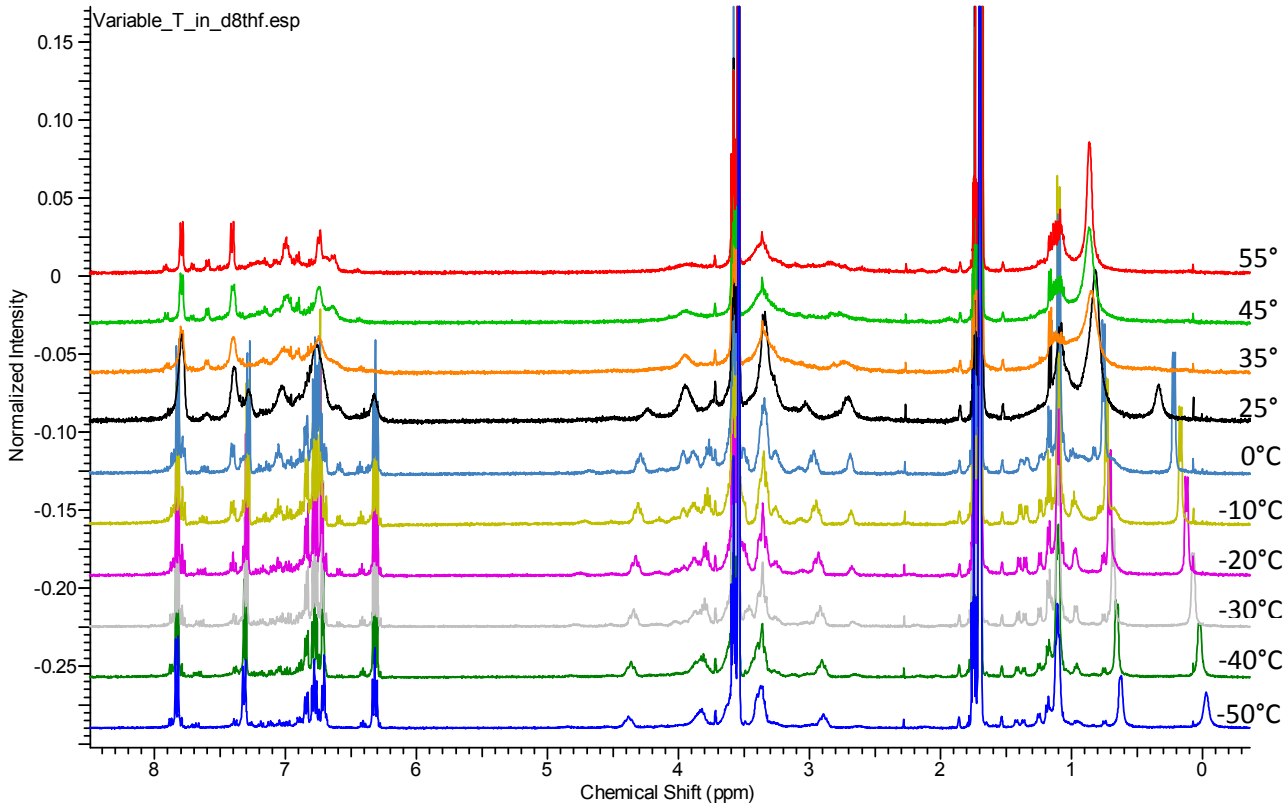


Figure S35. Variable temperature ^{1}H-NMR of **6** d_8-thf

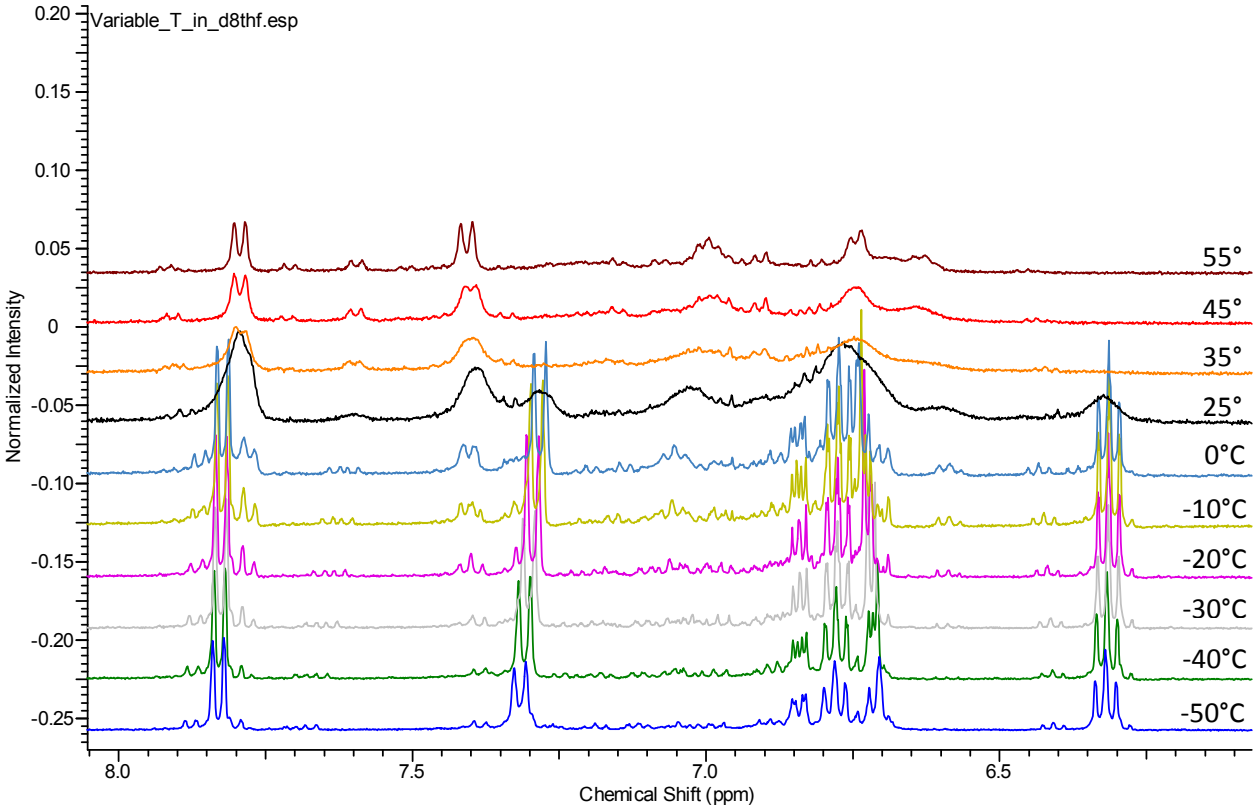


Figure S36. Variable temperature ^{1}H-NMR of **6** d_8-thf: high frequency section

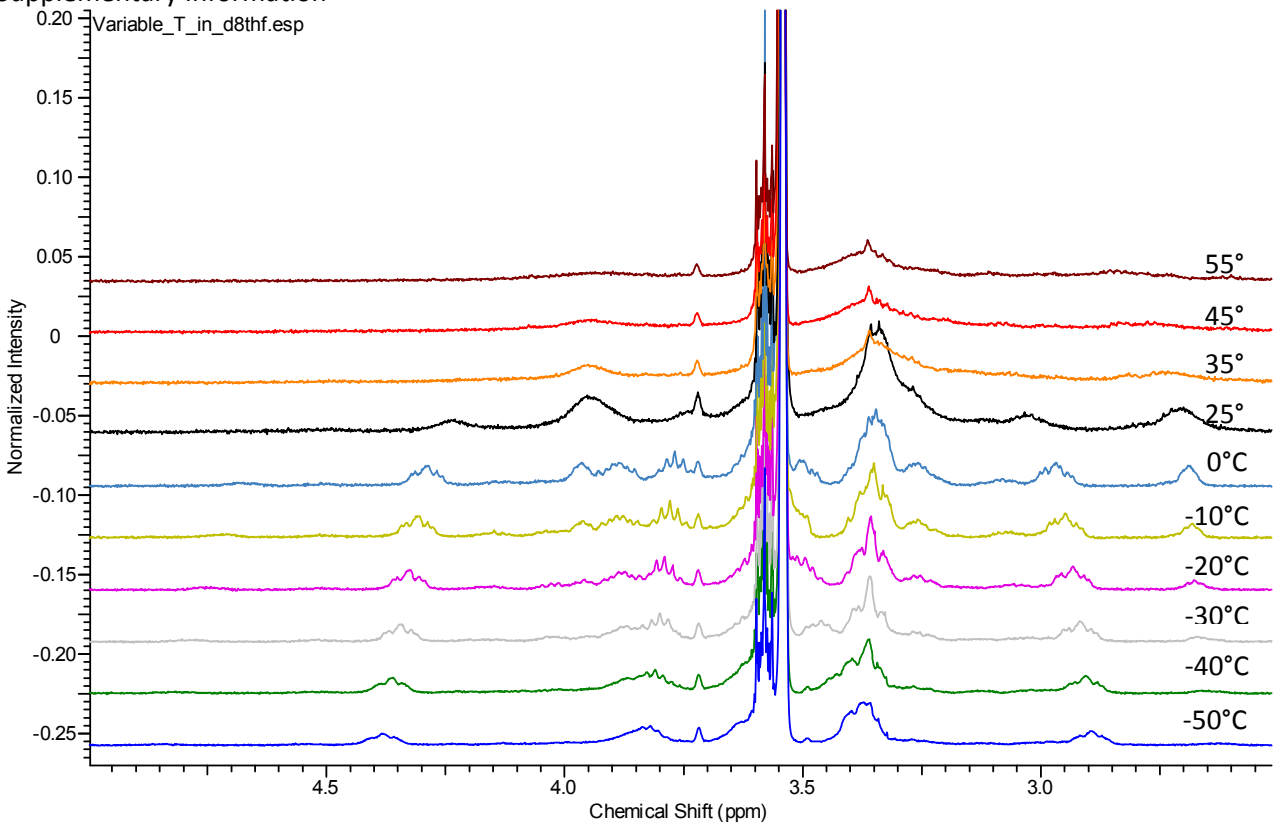


Figure S37. Variable temperature ^{1}H-NMR of **6** d_8-thf: medium frequency section

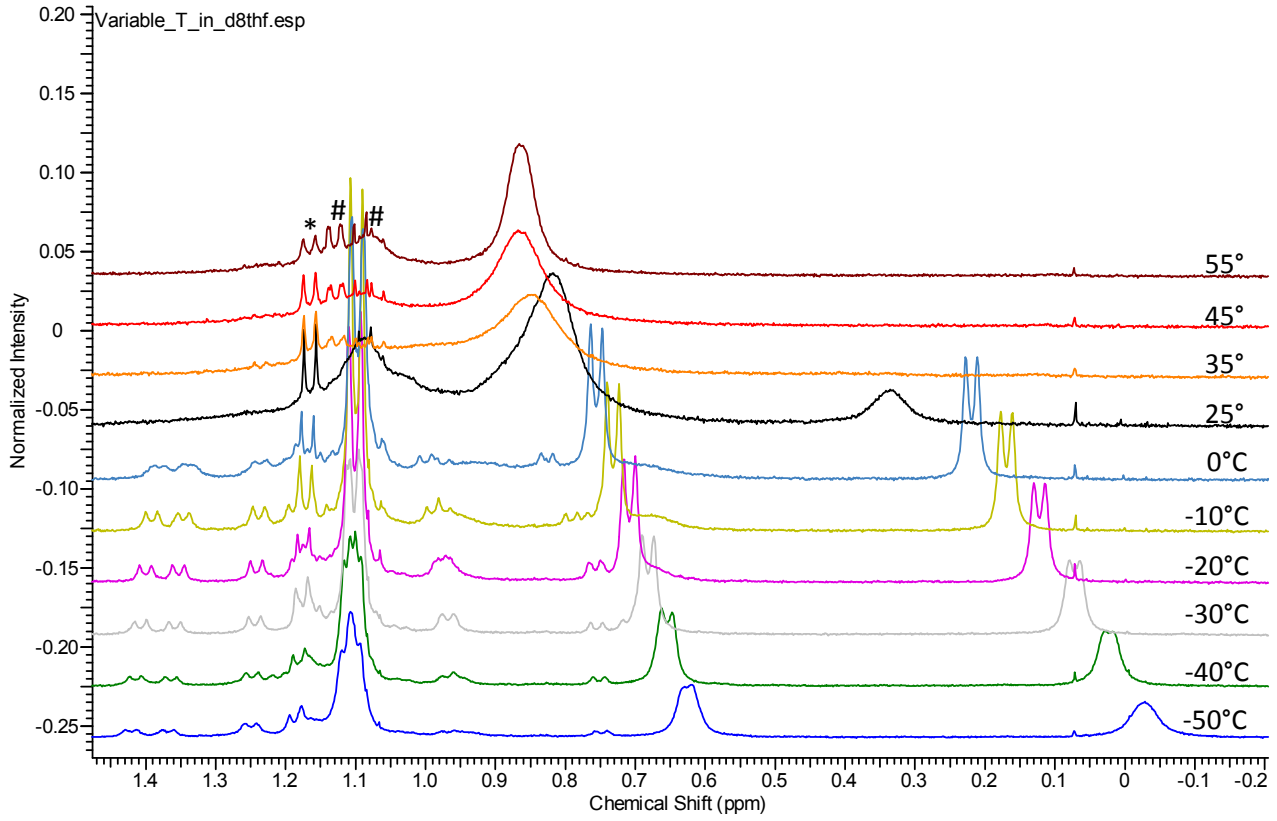


Figure S38. Variable temperature ^{1}H-NMR of **6** d_8-thf: low frequency section. The resonances under * are from trace hydrolysis of the complex. The resonances under # arise from a thermal decomposition process at higher temperatures.

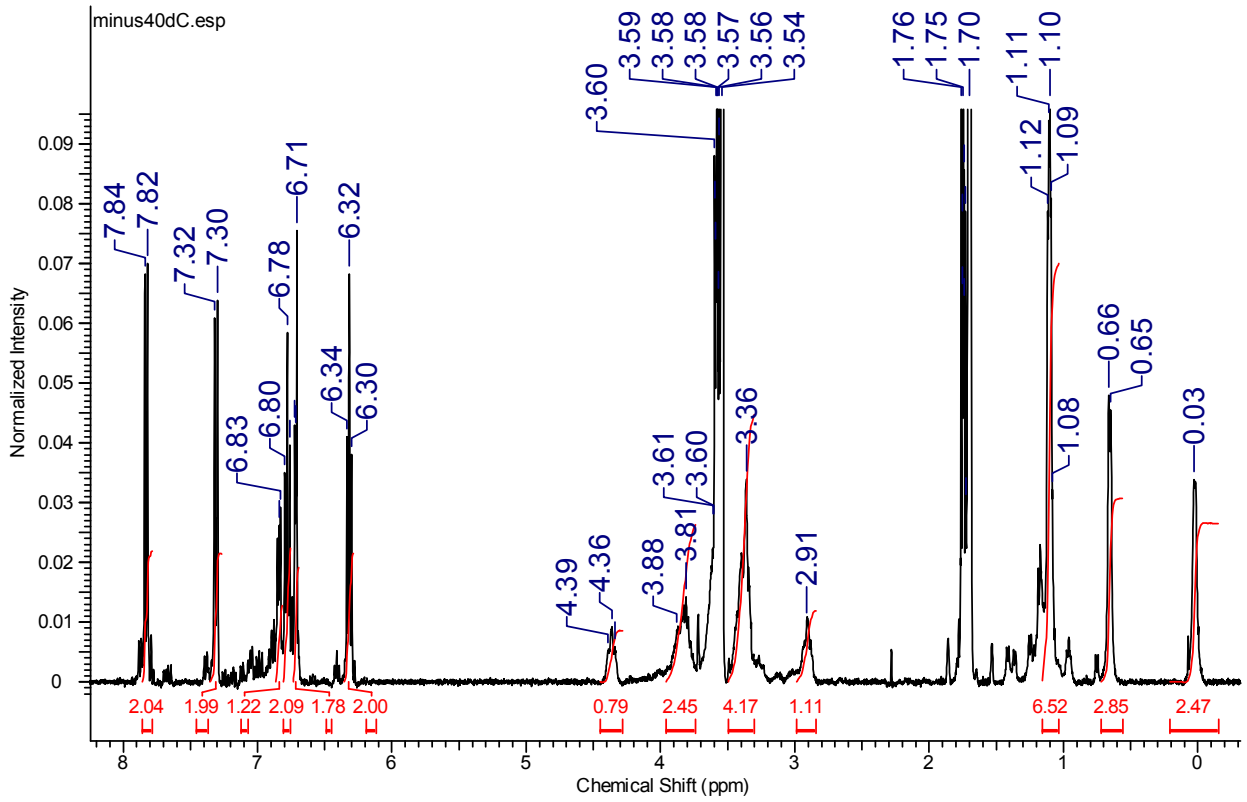


Figure S39. ^{1}H-NMR of **6** in d_8-THF at -40 °C

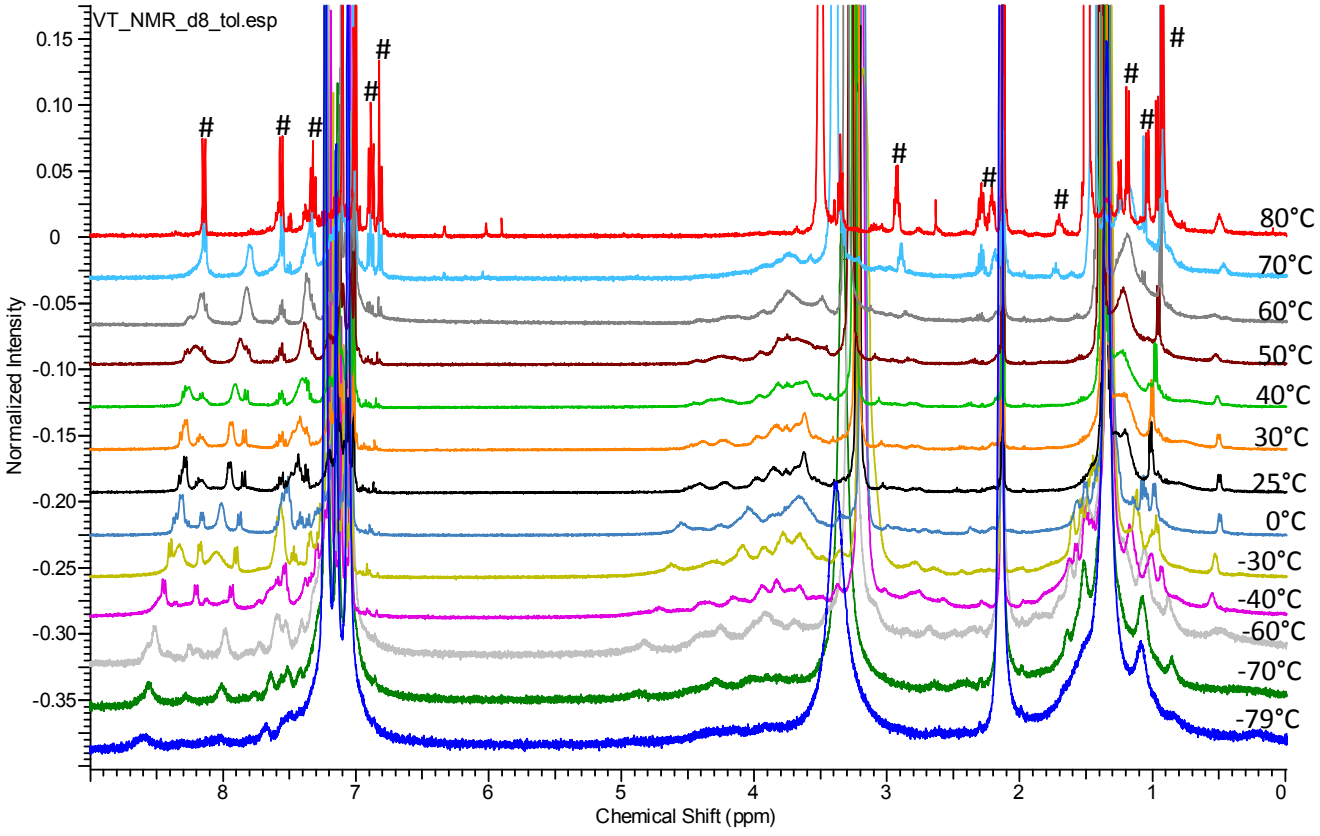


Figure S40. Variable temperature ^{1}H-NMR of **6** d_8-toluene. The resonances under # arise from a thermal decomposition process at higher temperatures.

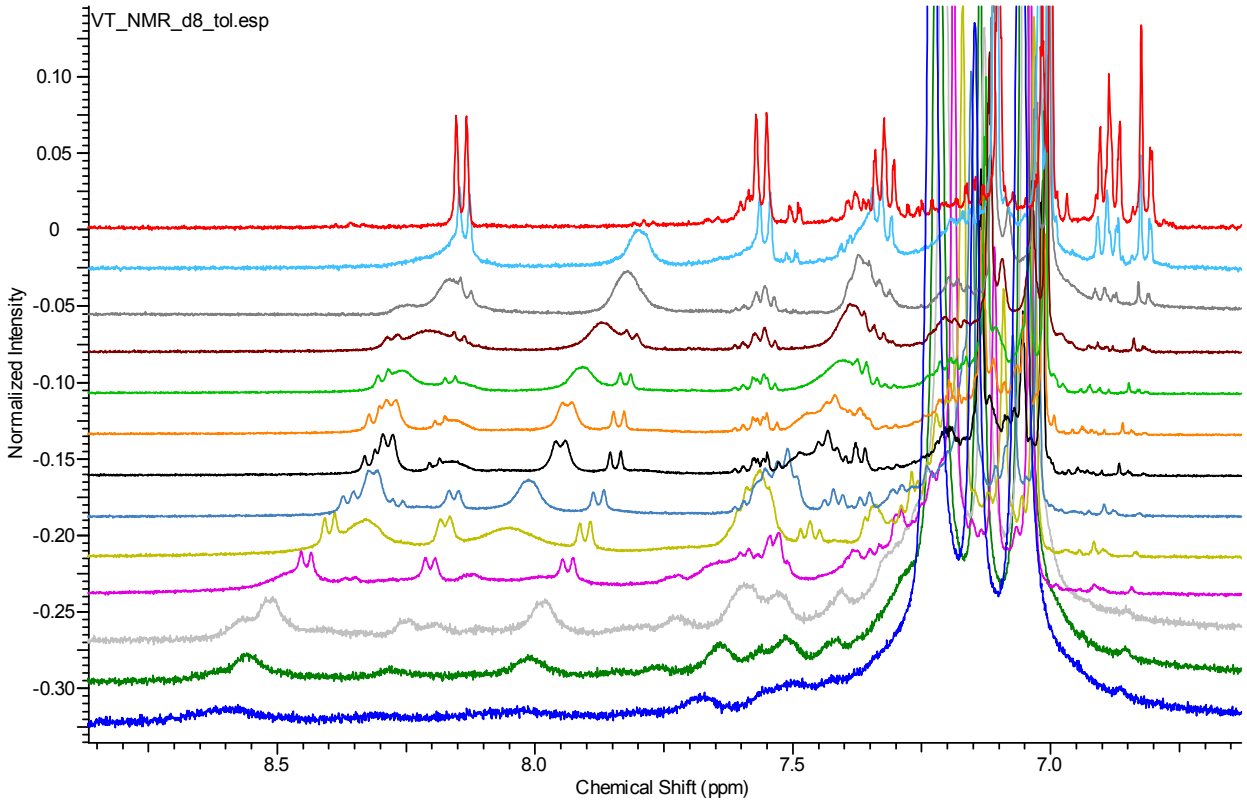


Figure S41. Variable temperature ^{1}H-NMR of **6** d_8-toluene: high frequency region.

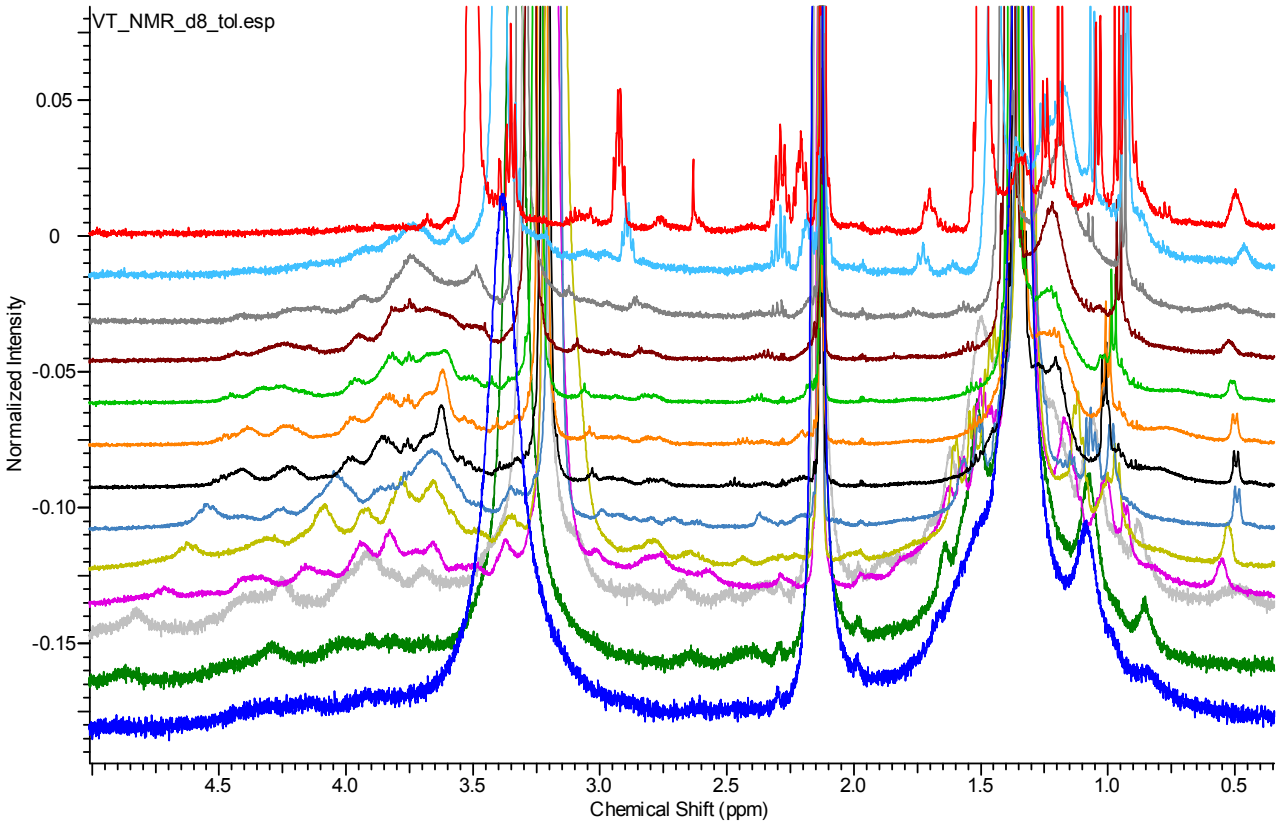


Figure S42. Variable temperature ^{1}H-NMR of **6** d_8-toluene: low frequency region.

7

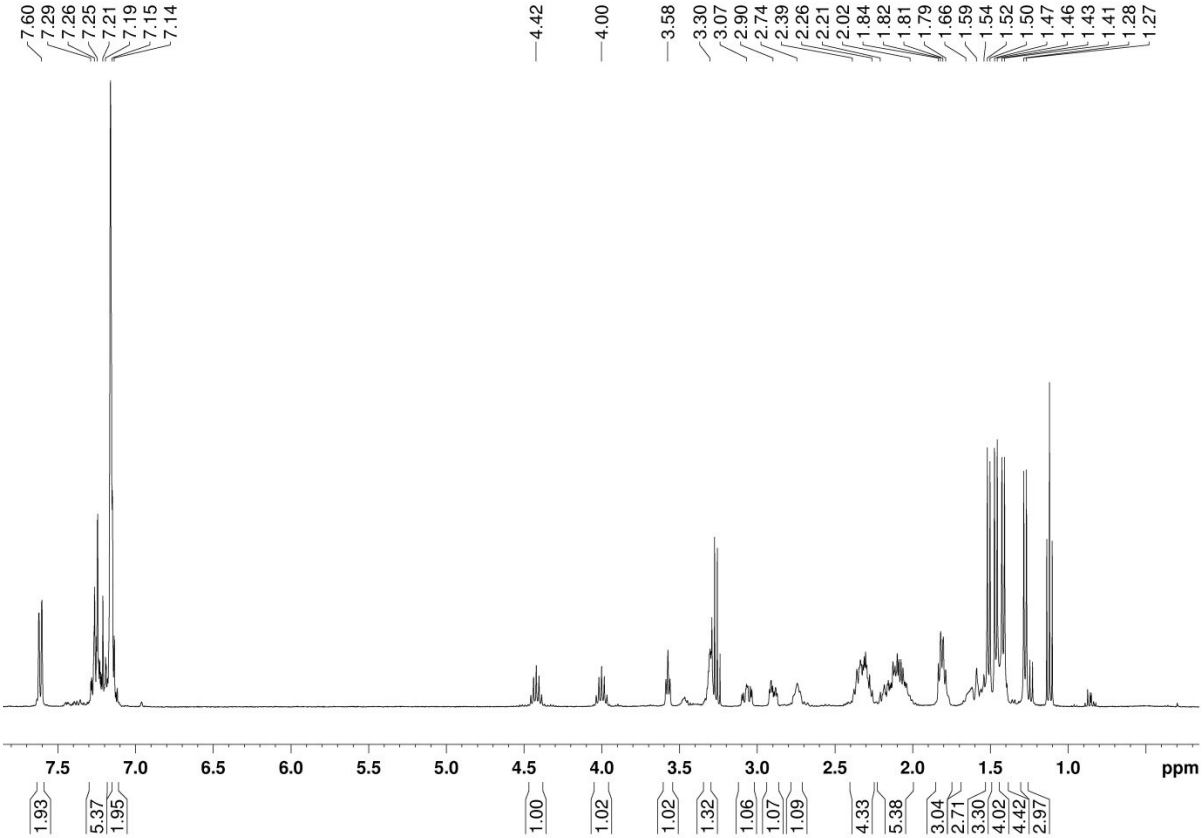


Figure S43. ^{1}H-NMR of **7** in C_6D_6 at 25 °C

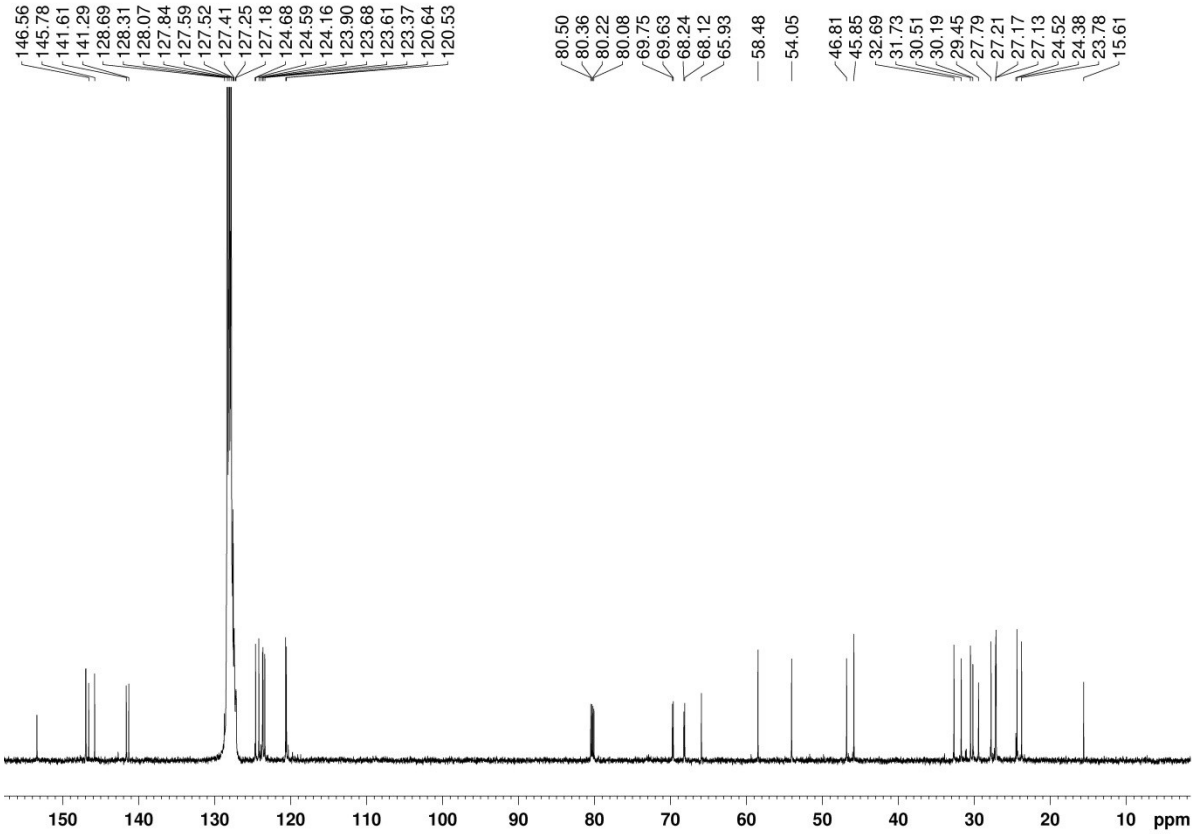


Figure S44. ^{13}C-NMR of **7** in C_6D_6 at 25 °C

8

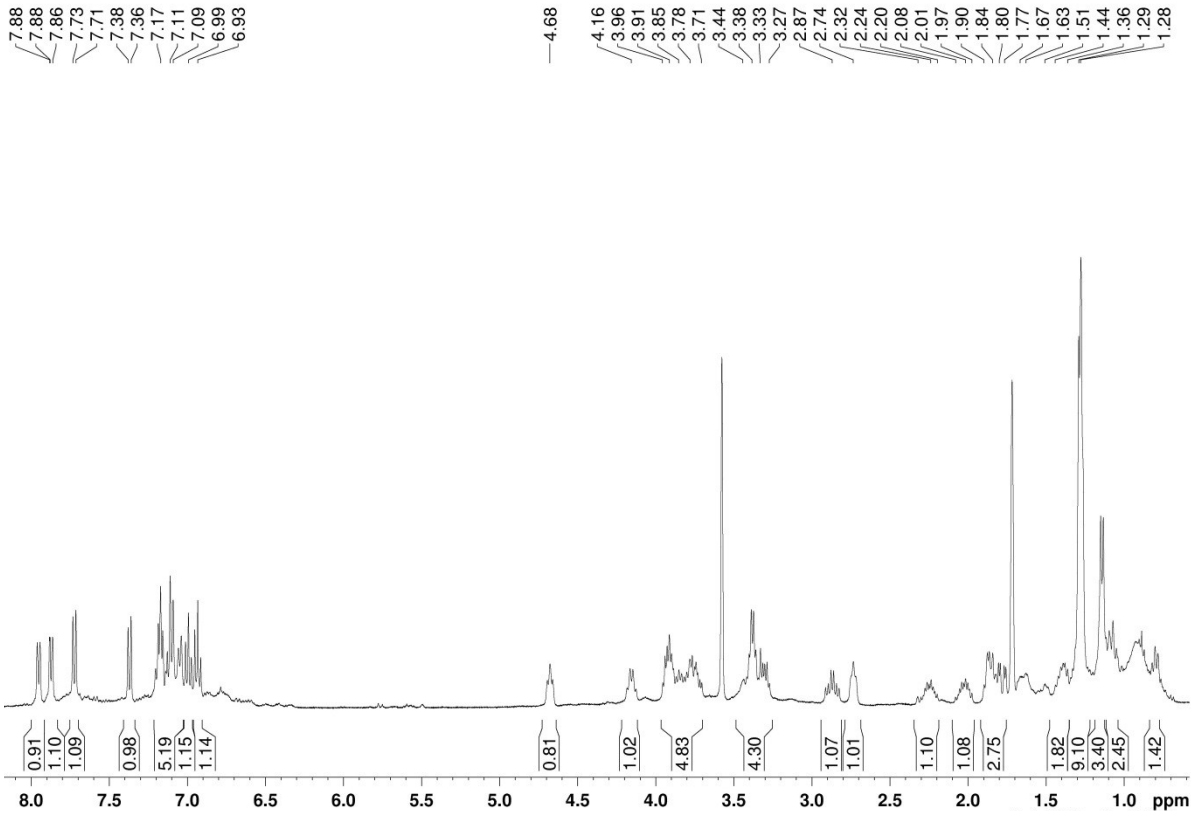


Figure S45. ^{1}H-NMR of **8** in d_8-thf at 25 °C

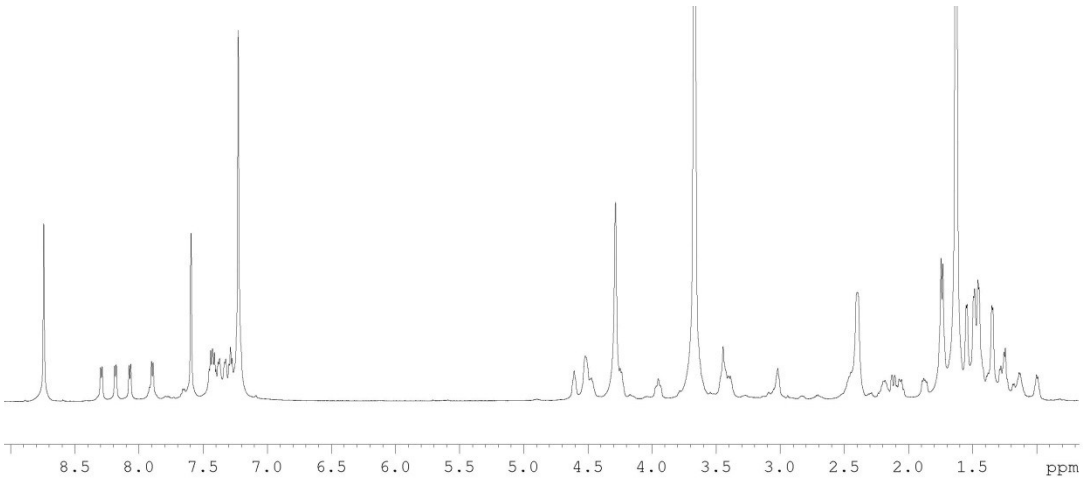


Figure S46. ^{1}H-NMR of **8** at 700 MHz in d_5-pyridine at 25 °C

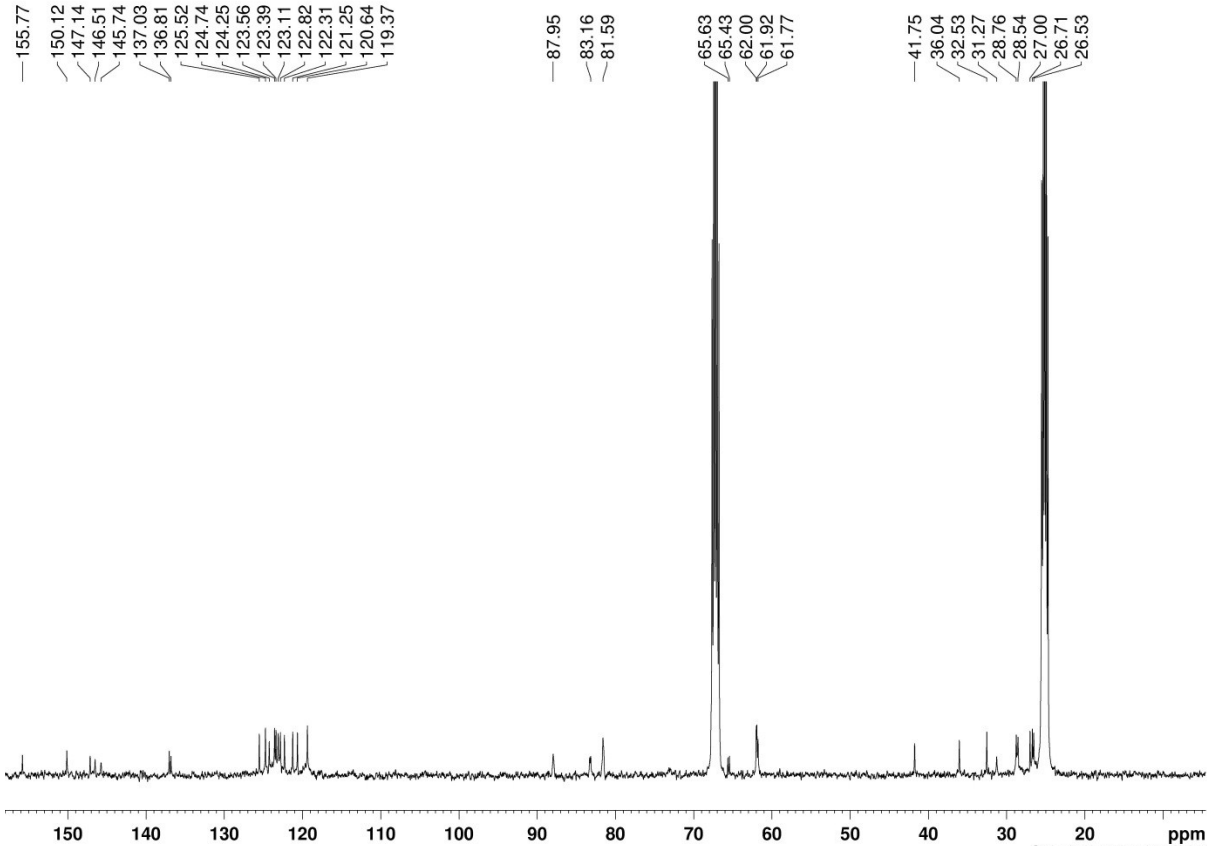


Figure S47. ^{13}C-NMR of **8** in d_8-thf at 25 °C

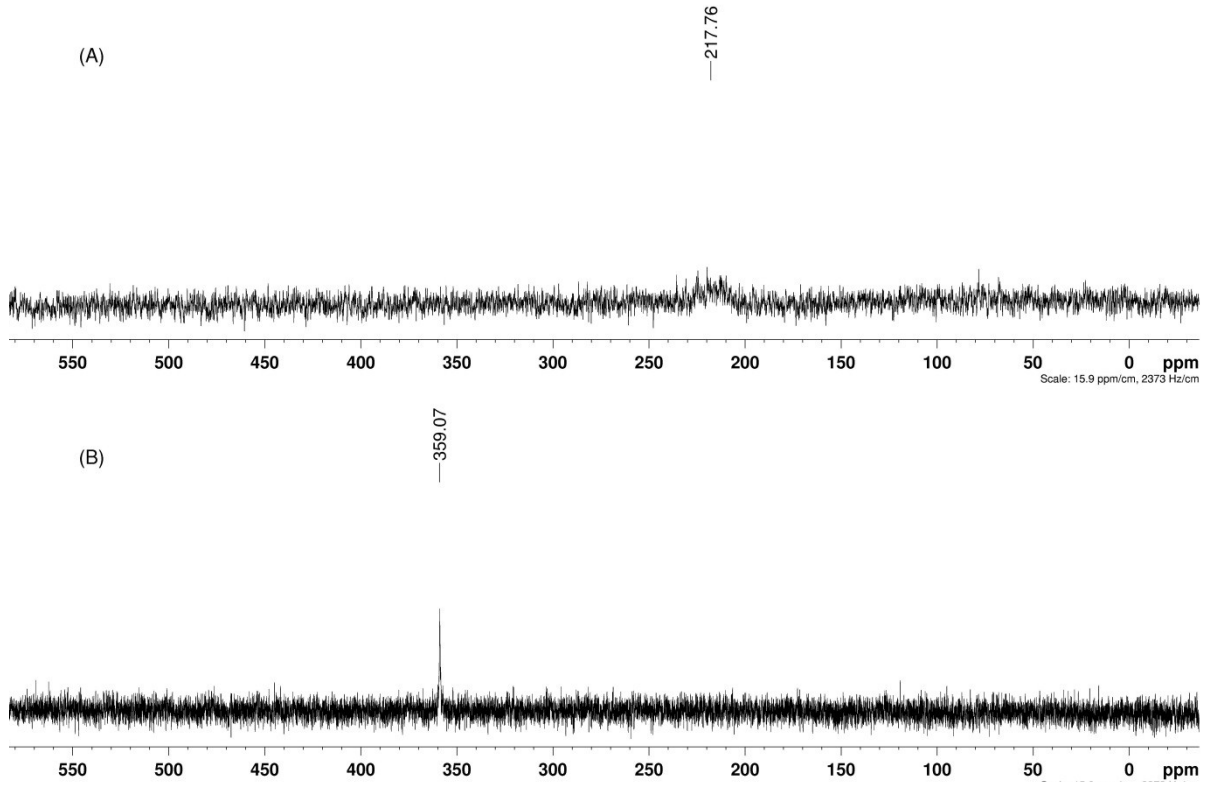


Figure S48. ^{119}Sn-NMR of **8** at in (A) d_8-thf at 25 °C, (B) C_6D_6 at 25 °C.

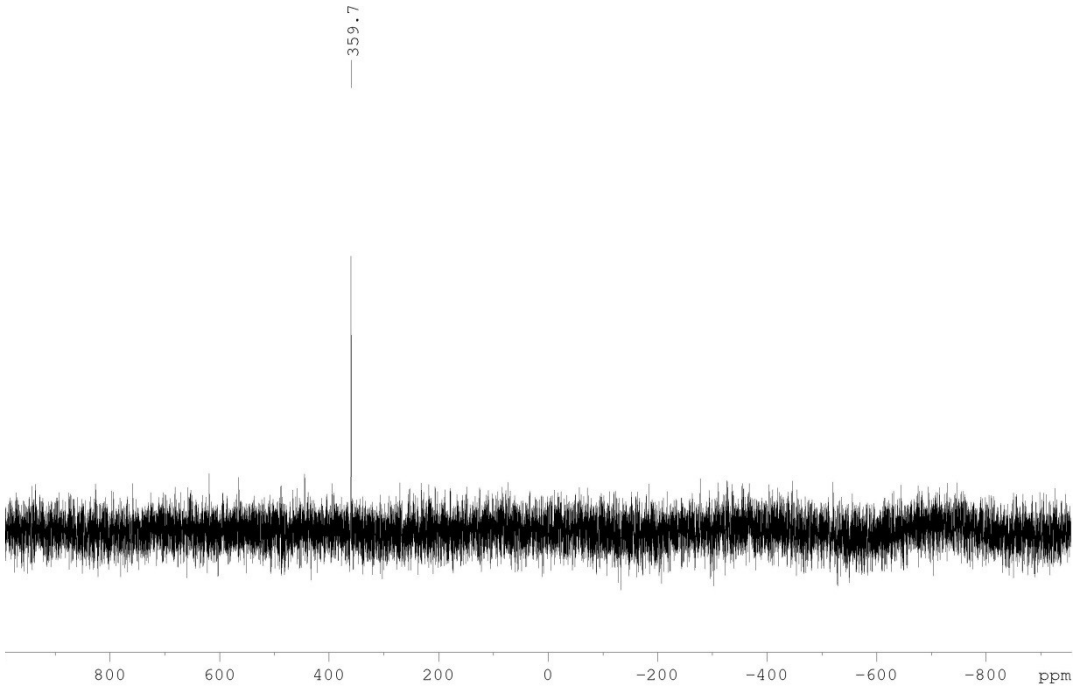


Figure S49. ^{119}Sn-NMR of **8** at 261 MHz (700MHz spectrometer) in C_6D_6 at 25 °C. No signal was observed in d_5-pyridine at 261 MHz after similar experiment time.

Supplementary Information

Additional crystallographic details

Crystals of **1b** were pseudo-merohedrally twinned and a twin law was successfully applied. Refinement as a mixture of 7 parts **1b** to 1 part **1b·HCl** gave the best model, with hydrogen bonding to H_2O molecules modelled with the correct occupancies, to give a final moiety formula of 7($C_{21}H_{28}N_2$), 1($C_{21}H_{29}N_2$)Cl, 4(H_2O), 6(H_2O).

Identification code	**1a**	**1b**	**1b·2HCl**
Empirical formula	$C_{29}H_{36}N_2$	$C_{168}H_{245}ClN_{16}O_{10}$	$C_{29}H_{36}N_2$
Formula weight	412.60	2684.24	412.60
Temperature/K	100.0(2)	100(2)	100.0
Crystal system	triclinic	monoclinic	monoclinic
Space group	*P***Error!**	*C*2/*c*	$P2_1/n$
a/Å	8.8412(7)	34.733(3)	11.0999(6)
b/Å	10.2994(7)	5.8657(5)	7.4994(4)
c/Å	14.6904(11)	19.4635(17)	28.9734(15)
α/°	90.460(3)	90	90
β/°	102.300(3)	106.437(4)	100.948(2)
γ/°	111.703(3)	90	90
Volume/Å^3	1208.77(16)	3803.3(6)	2367.9(2)
Z	2	1	4
ρ_{calc}g/cm^3	1.134	1.172	1.157
μ/mm^{-1}	0.065	0.089	0.067
F(000)	448.0	1462.0	896.0
Crystal size/mm^3	0.48 × 0.38 × 0.26	0.2 × 0.2 × 0.06	0.45 × 0.20 × 0.10
2Θ range for data collection/°	2.85 to 64.746	2.182 to 49.492	5.132 to 54.928
Index ranges	-13 ≤ h ≤ 13, -15 ≤ k ≤ 15, -22 ≤ l ≤ 20	-40 ≤ h ≤ 39, -6 ≤ k ≤ 6, -22 ≤ l ≤ 22	-14 ≤ h ≤ 14, -9 ≤ k ≤ 9, -36 ≤ l ≤ 37
Reflections collected	32628	11915	38959
Independent reflections	8568 [R_{int} = 0.0295, R_{sigma} = 0.0323]	3205 [R_{int} = 0.0371, R_{sigma} = 0.0451]	5392 [R_{int} = 0.0499, R_{sigma} = 0.0400]
Data/restraints/parameters	8568/0/290	3205/10/260	5392/0/290
GooF on F^2	1.025	1.049	1.060
Final R indexes [I>=2σ (I)]	R_1 = 0.0444, wR_2 = 0.1143	R_1 = 0.0493, wR_2 = 0.1077	R_1 = 0.0544, wR_2 = 0.1131
Final R indexes [all data]	R_1 = 0.0600, wR_2 = 0.1237	R_1 = 0.0733, wR_2 = 0.1171	R_1 = 0.0826, wR_2 = 0.1244
Largest diff. peak/hole / e Å^{-3}	0.43/-0.23	0.19/-0.22	0.26/-0.28

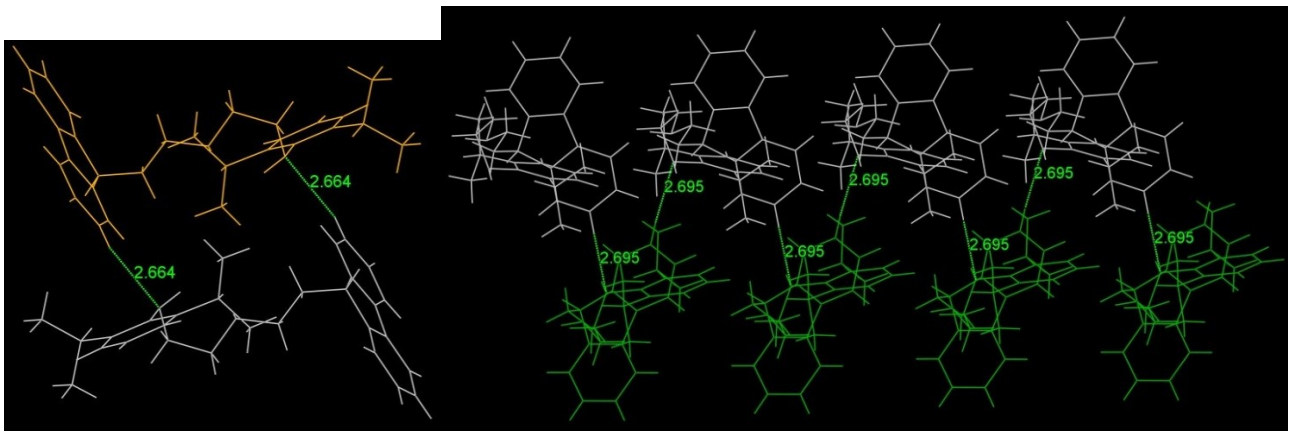


Figure S50. Diagrams showing intermolecular interactions between N2 and H4 in of **1a** (*P***Error!**, left) and in the $P2_1/n$ polymorph of **1a** (top) leading to pairs of molecules and ribbons respectively.

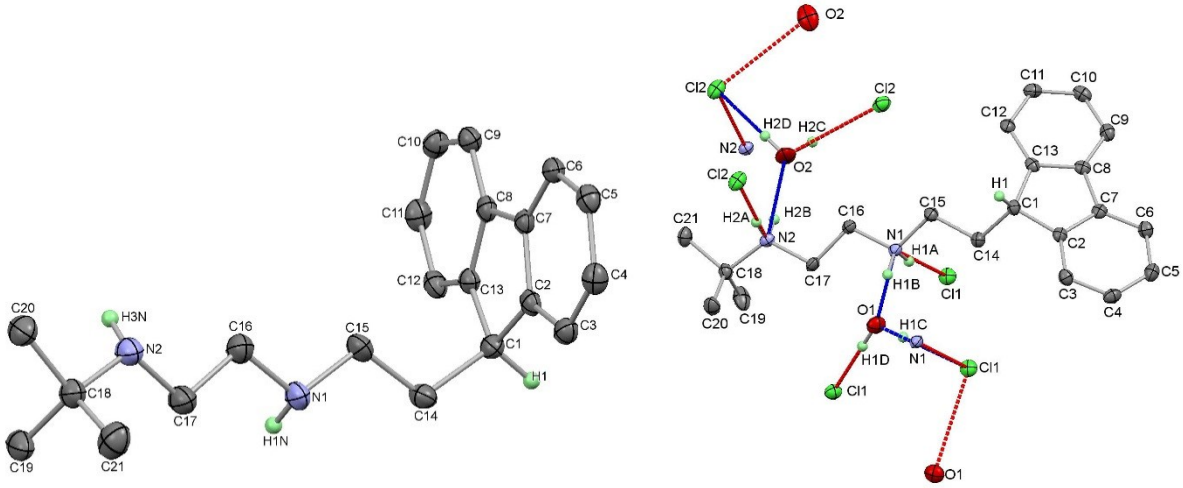


Figure S51. Thermal ellipsoid plot of the molecular structure of **1b** (left) and **1b·2HCl** (right) with thermal ellipsoids at 50% probability. The positions of only the relevant hydrogen atoms are shown with the rest omitted for clarity along with H_2O molecules and partially occupied Cl (1/8 occupancy) for **1b**. Selected bond distances (Å) and angles (°) for **1b**: C1-C2 1.524(3), C1-C13 1.519(3), C1-C14 1.544(3), C2-C7 1.410(3), C7-C8 1.468(3), C8-C13 1.403(3); Σ (angles at C1) 327.9. H-bonding in the asymmetric unit of **1b·2HCl** is highlighted in blue, with additional hydrogen bonding in red.

Additional references

1. I. K. Kormendy, *Acta Chim. Acad. Sci. Hung.*, 1958, **17**, 255.
2. M. Deppner, R. Burger and H. G. Alt, *J. Organomet. Chem.*, 2004, **689**, 1194.