Electronic Supplementary Material (ESI) for Dalton Transactions.

Supporting Information

Dimethylmagnesium revisited

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Table S1. Crystallographic data for compounds **1**, **3** and **4**

	1	**3**	**4**
CCDC#	837386	837387	837388
formula	C_2H_6Mg	$C_{20}H_{60}Ga_4Mg_4O_4$	$C_{16}H_{48}Al_4Mg_2O_4$
M_r [g mol^{-1}]	54.38	740.80	1556.56
color	colorless/plate	colorless/cuboid	colorless/rod
crystal dimensions [mm]	0.132 x 0.075 x 0.060	0.454 x 0.245 x 0.163	0.196 x 0.170 0.163
cryst syst	orthorhombic	tetragonal	monoclinic
space group	*Ibam*	$I4_1/a$	$P2_1/n$
a [Å]	5.8233(9)	17.89(2)	8.063(2)
b [Å]	11.3102(7)	17.89(2)	9.830(2)
c [Å]	5.4324(8)	10.61(1)	18.911(4)
α [°]	90	90	90
β [°]	90	90	97.643(3)
γ [°]	90	90	90
V [Å^3]	357.79(9)	3396(7)	1485.6(6)
Z	4	4	2
T [K]	100(2)	100(2)	100(2)
ρ_{calcd} [g cm^{-3}]	1.009	1.449	1.031
μ [mm^{-1}]	0.214	3.235	0.214
F (000)	120	1536	504
θ range [°]	3.6025/29.99	3.214/26.645	2.1735/28.22
unique reflns	289	2287	2724
observed reflns (I>2σ)	275	2054	2156
R1/*w*R2 (I>2σ)[a]	0.0246/0.0662	0.0182/0.0466	0.0608/0.1491
R1/*w*R2 (all data)[a]	0.0261/0.0671	0.0222/0.0481	0.0798/0.1587
GOF[a]	1.093	1.030	1.113

[a] $R1 = \Sigma(||F_0|-|F_c||)/\Sigma|F_0|$, $F_0 > 4\sigma(F_0)$. $wR2 = \{\Sigma[w(F_0^2-F_c^2)^2/\Sigma[w(F_0^2)^2]\}^{1/2}$.

Notes on NMR spectroscopic characterization.

* residual solvent peaks

Internal standard used for **S3-S5**: dmso-d_6/ethylene glycol (20:80)

NMR Spectra

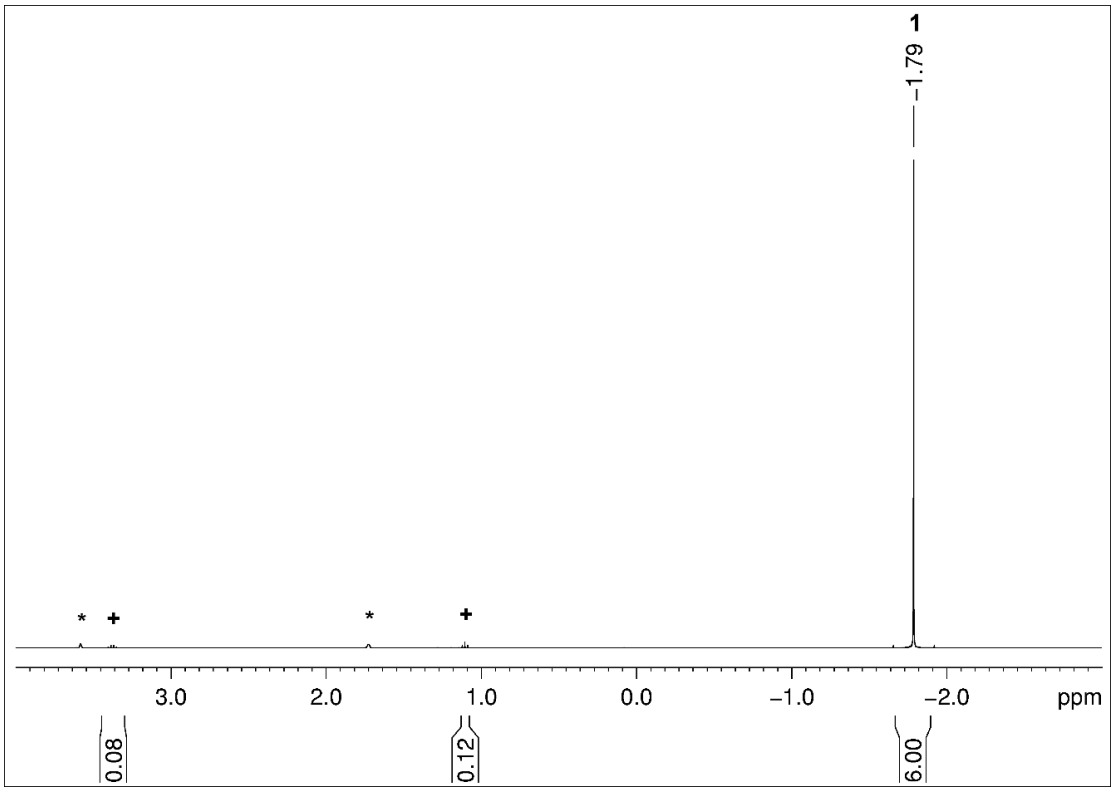

Figure S1. ^{1}H NMR spectrum (400 MHz) of **1** in thf-d_8 at 26 °C. (+ diethyl ether)

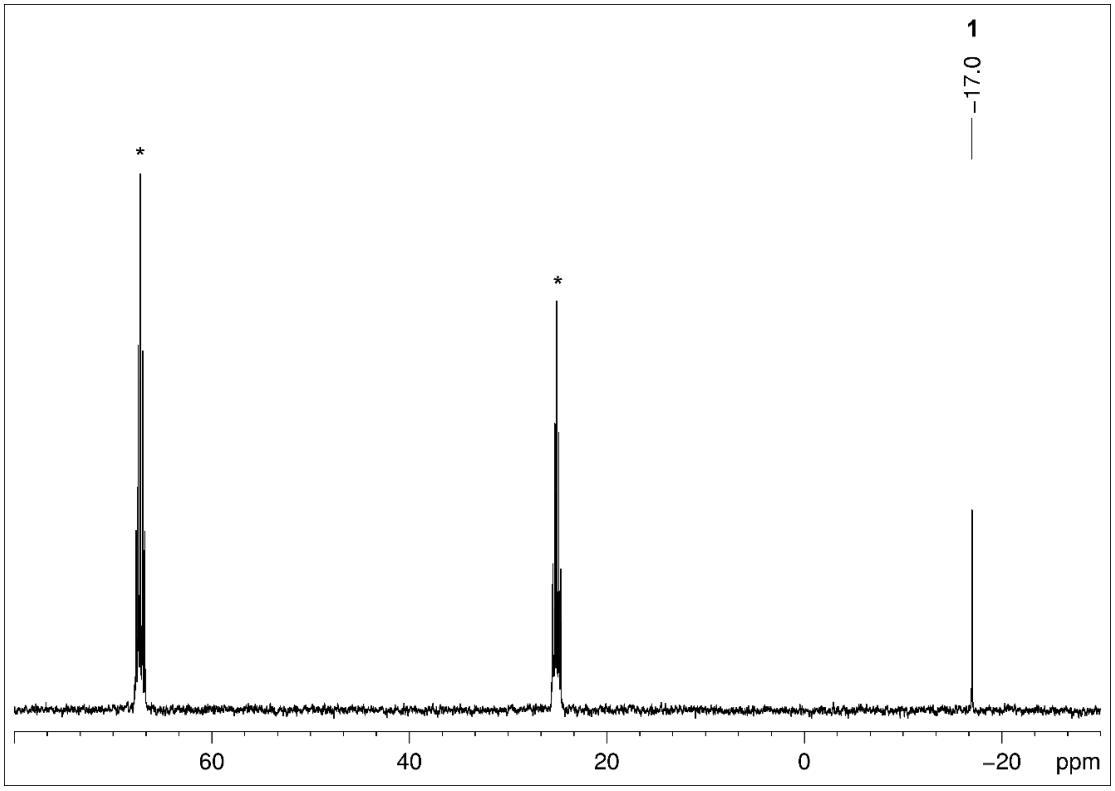

Figure S2. ^{13}C{^{1}H} NMR spectrum (101 MHz) of **1** in thf-d_8 at 26 °C.

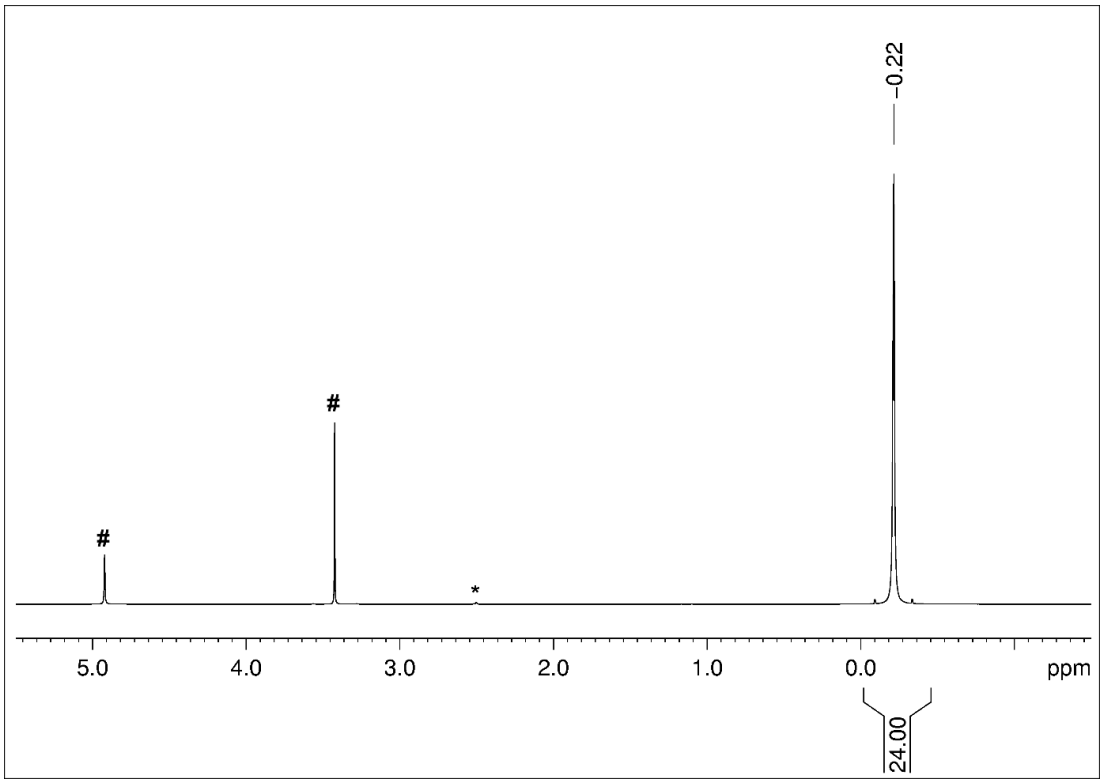

Figure S3. ^{1}H NMR spectrum (400 MHz) of **2** in neat $GaMe_3$ at 26 °C (dmso-d_6 as internal standard, # ethylene glycol).

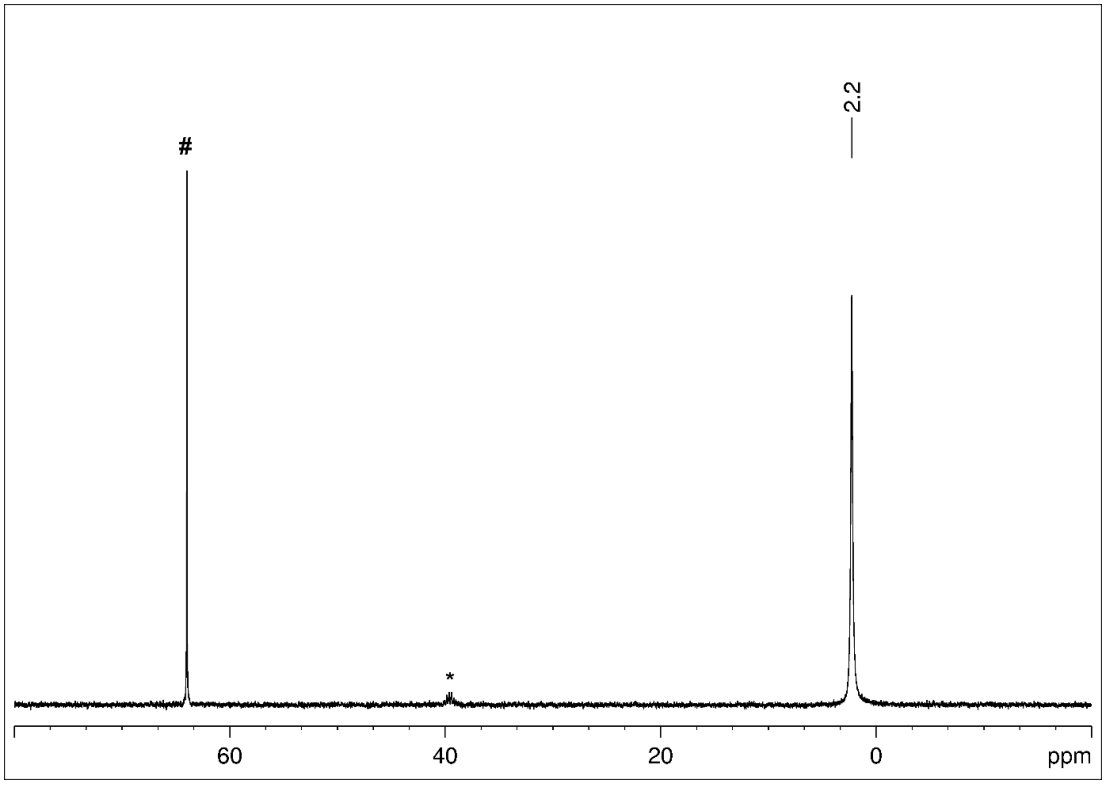

Figure S4. $^{13}C\{^{1}H\}$ NMR spectrum (101 MHz) of **2** in neat $GaMe_3$ at 26 °C (dmso-d_6 as internal standard, # ethylene glycol).

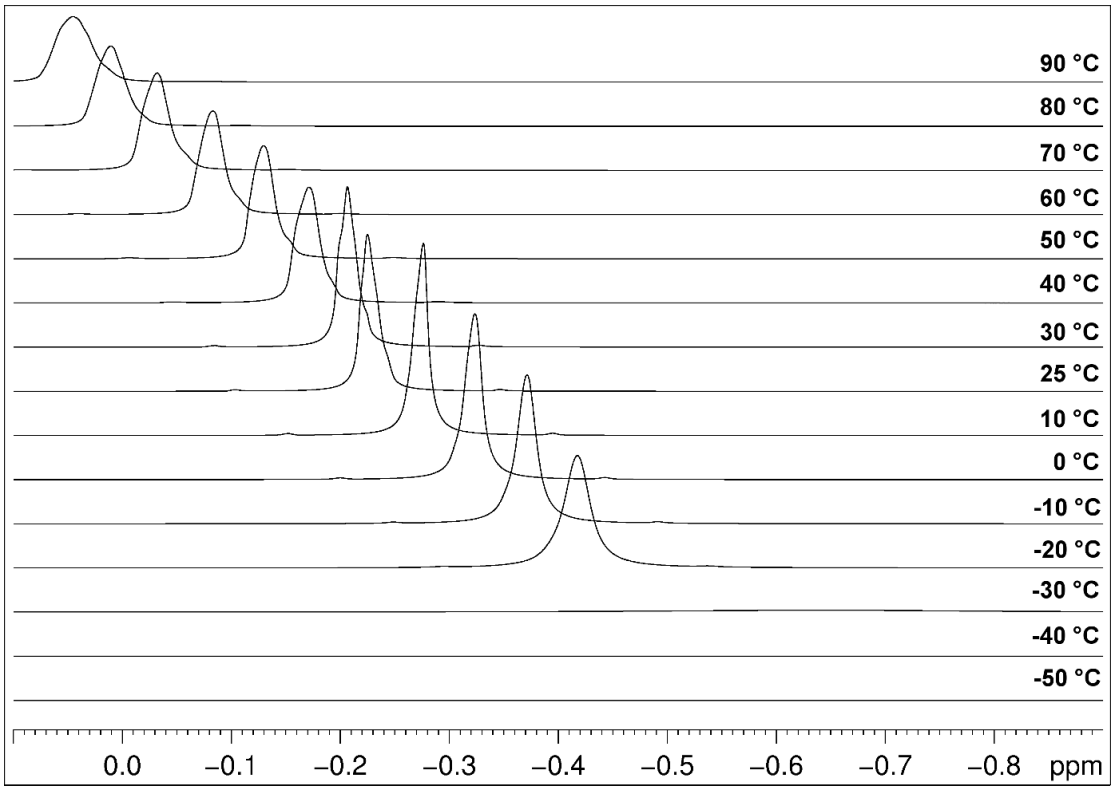

Figure S5. Variable-temperature ^{1}H NMR spectra (400 MHz) of **2** in neat $GaMe_3$ (dmso-d_6 as internal standard, # ethylene glycol).

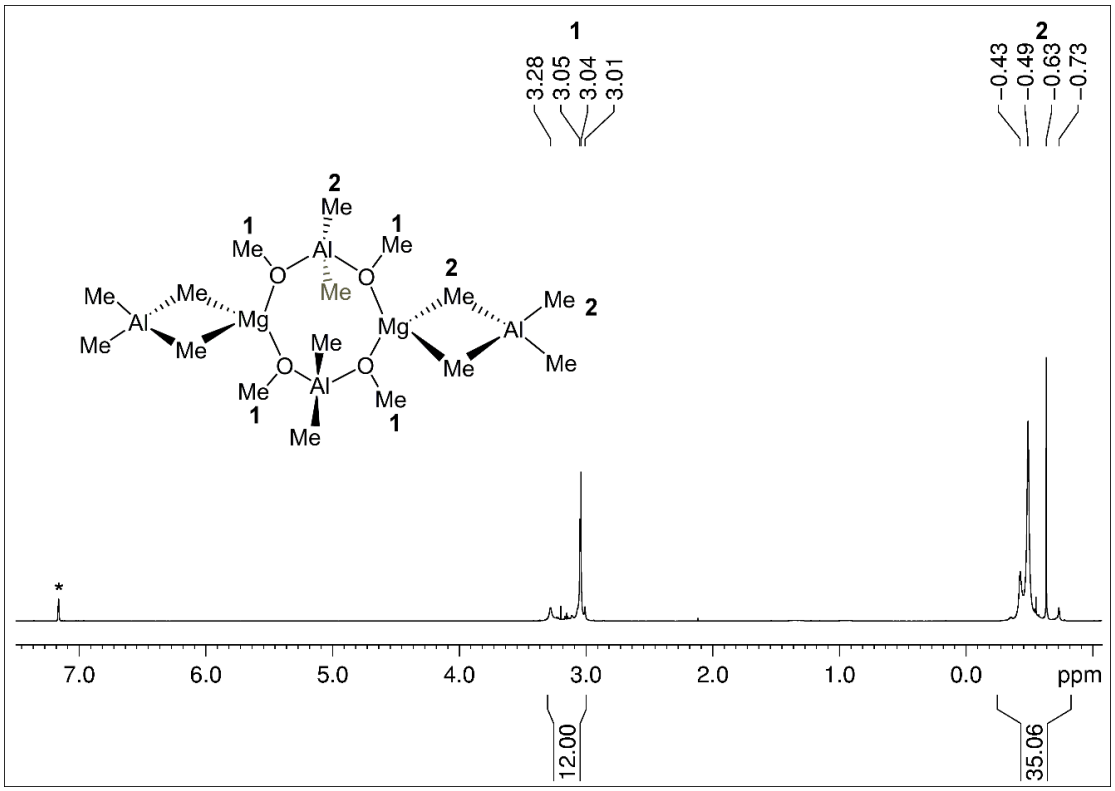

Figure S6. ^{1}H NMR spectrum (400 MHz) of **4** in benzene-d_6 at 26 °C.

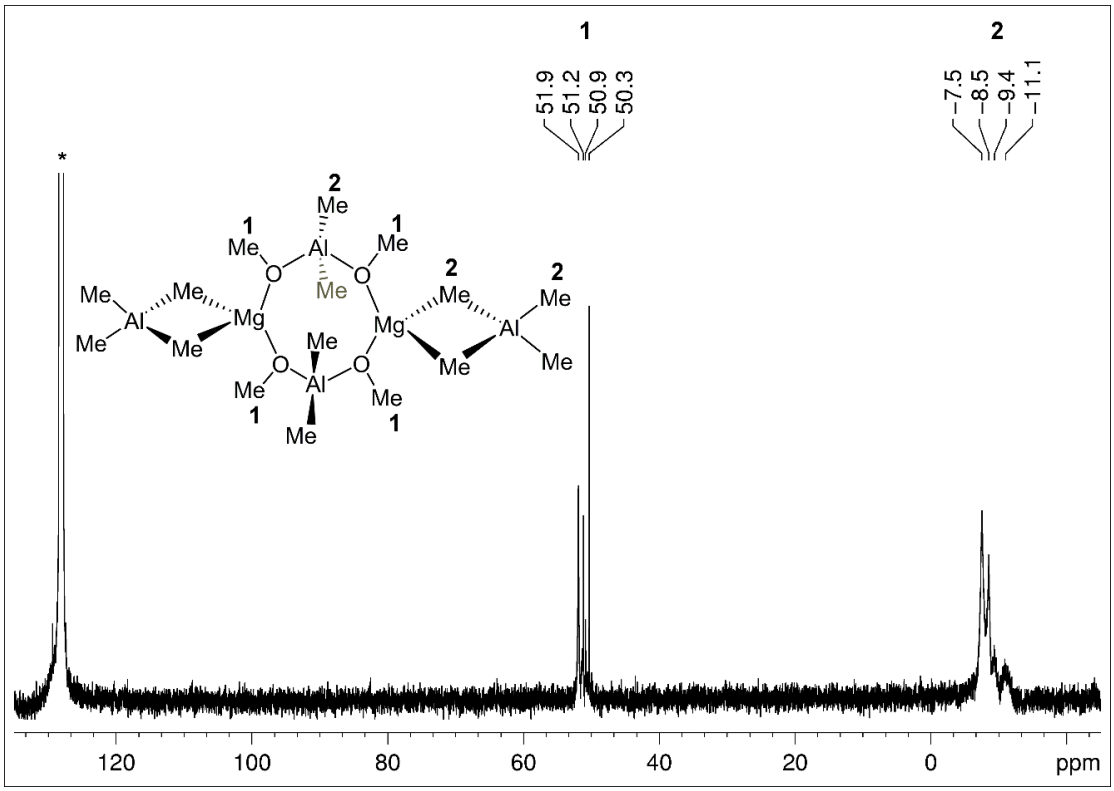

Figure S7. $^{13}C\{^{1}H\}$ NMR spectrum (101 MHz) of **4** in benzene-d_6 at 26 °C.

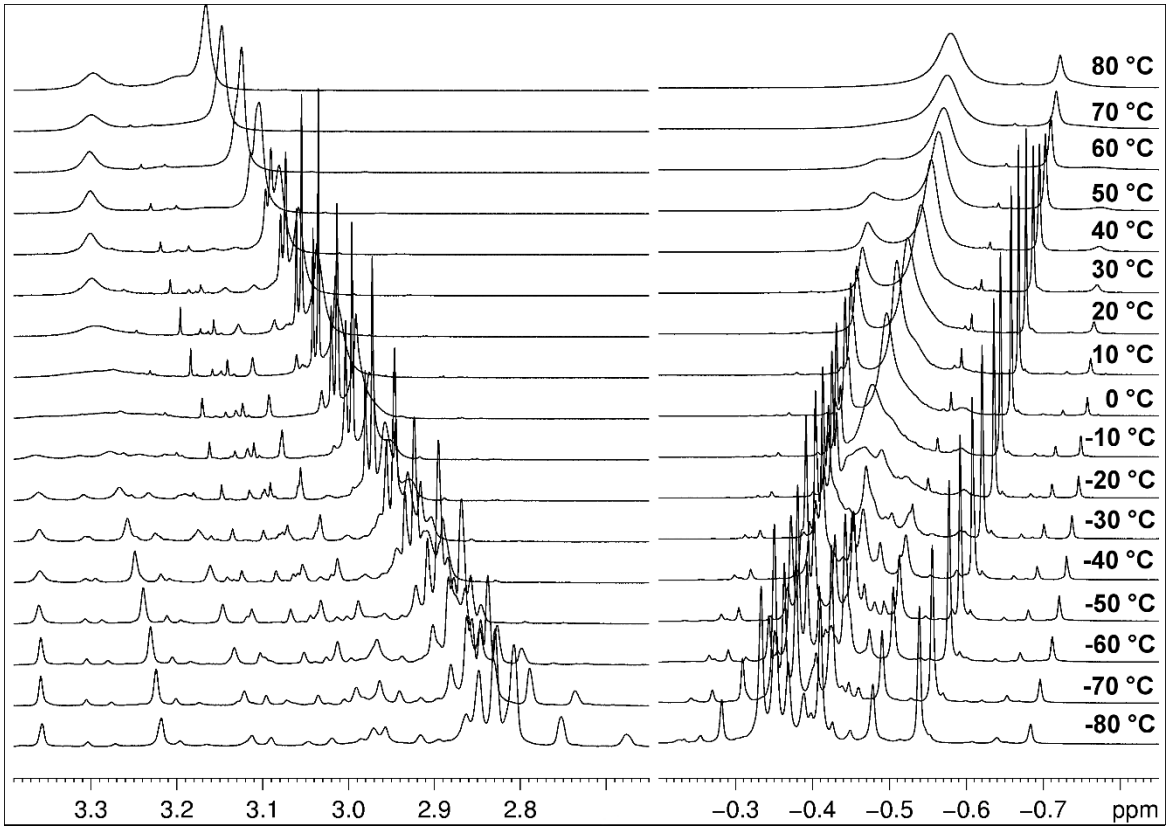

Figure S8. Variable-temperature ^{1}H NMR spectra (400 MHz) of **4** in toluene-d_8.

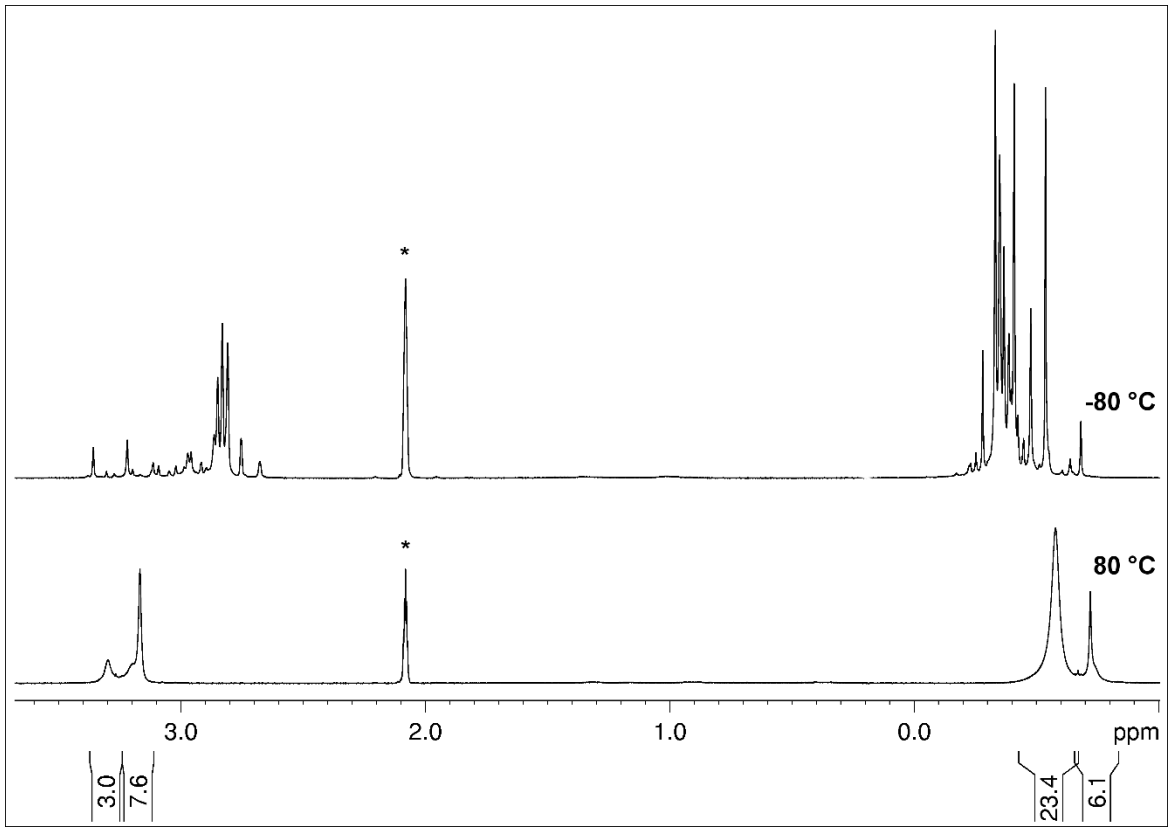

Figure S9. Selected variable-temperature ^{1}H NMR spectra (400 MHz) of **4** in toluene-d_8.

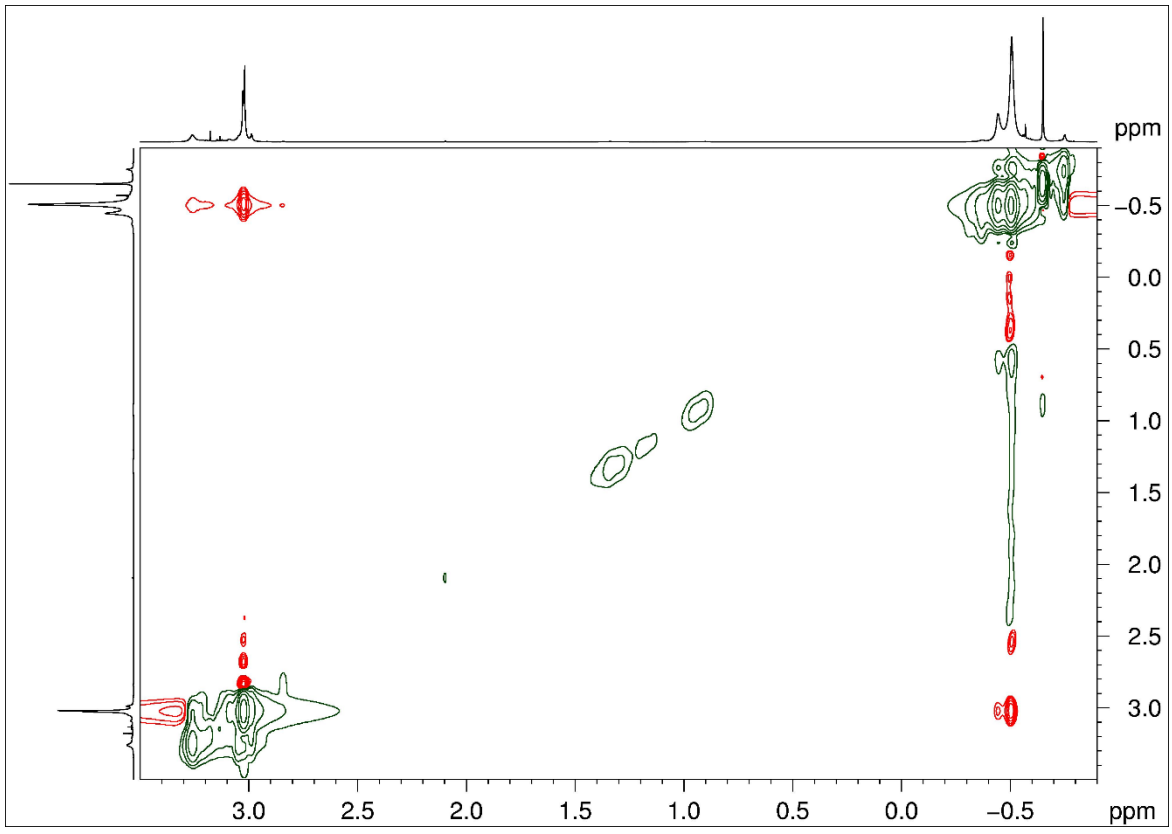

Figure S10. ^{1}H-^{1}H NOESY NMR spectrum (400 MHz) of **4** in benzene-d_6 at 26 °C.

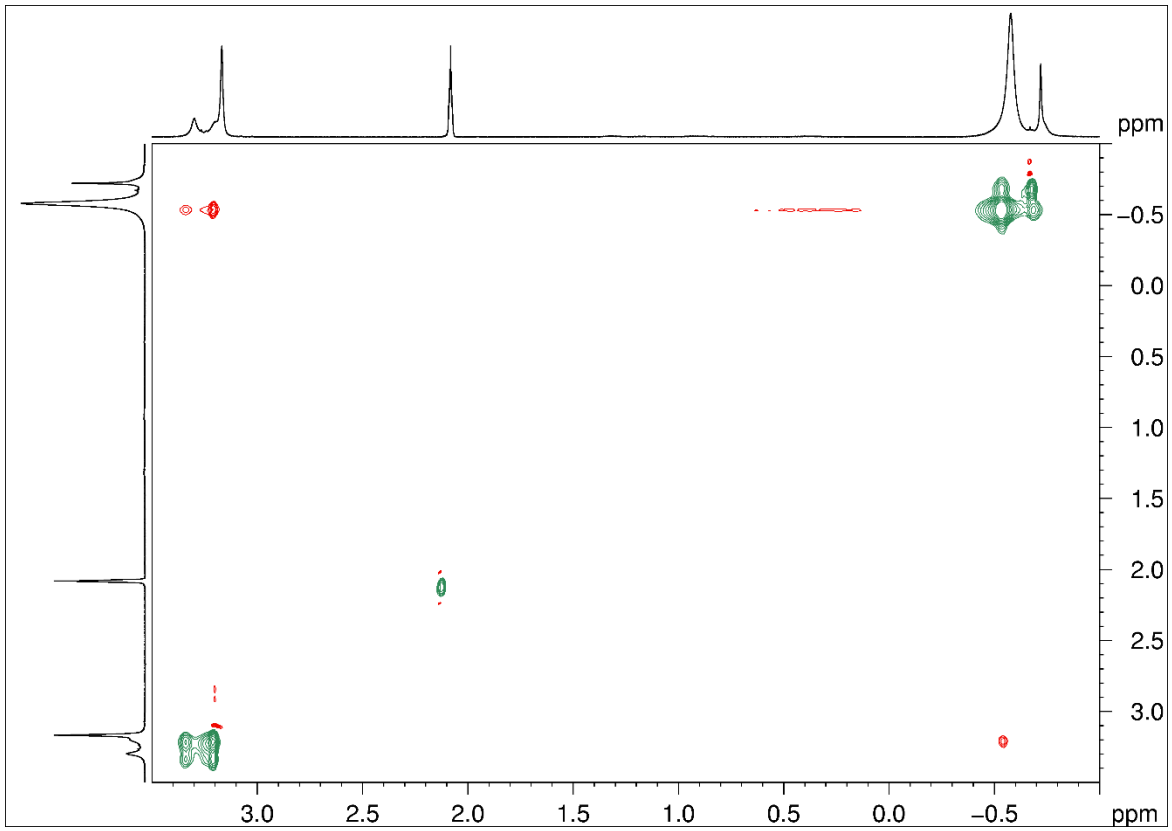

Figure S11. ^{1}H-^{1}H NOESY NMR spectrum (400 MHz) of **4** in toluene-d_8 at 80 °C.