Oligosilanylated Silocanes [†]

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[†] Dedicated to Professor Josef Michl on the occasion of his 80th birthday and in recognition of his numerous exciting contributions to organosilicon chemistry.

Contents

Table S1. Crystallographic data for compounds 5, 8, 8a, 9, 10 and 12.

	5	8	8a	9	10	12
Empirical formula	$C_{28}H_{76}N_2O_4Si_{10}$	$C_{23}H_{65}NO_2Si_9$	$C_{23}H_{65}NO_2Si_7$	$C_{21}H_{59}NO_2Si_9$	$C_{21}H_{59}NO_3Si_9$	$C_{15}H_{38}KNO_3Si_4$
M_w	785.81	640.57	729.57	610.50	626.50	431.92
Temperature [K]	100(2)	100(2)	100(2)	100(2)	100(2)	100(2)
Size [mm]	0.33×0.23×0.18	0.34×0.26×0.20	0.34×0.22×0.17	0.24×0.22×0.09	0.40×0.22×0.20	431.92
Crystal system	monoclinic	orthorhombic	triclinic	orthorhombic	monoclinic	Monoclinic
Space group	P2(1)/c	Pccn	P-1	Pca2(1)	P2(1)/n	P2(1)/c
a [Å]	9.773(4)	43.341(8)	9.952(2)	20.122(5)	9.879(2)	9.762(2)
b [Å]	18.507(4)	12.833(2)	12.433(2)	15.271(4)	18.655(4)	19.313(4)
c [Å]	13.604(3)	14.389(3)	17.836(3)	12.449(3)	21.111(4)	13.327(3)
α [°]	90	90	101.476(3)	90	90	90
β [°]	103.801(3)	90	92.746(3)	90	96.358(3)	100.070(4)
γ [°]	90	90	110.105(3)	90	90	90
V [Å^3]	2389(2)	8003(2)	2015(2)	3825(2)	3867(2)	2473.9(9)
Z	2	8	2	4	4	4
ρ_{calc} [gcm^{-3}]	1.092	1.063	1.203	1.060	1.076	1.160
Absorption coefficient [mm^{-1}]	0.305	0.318	1.719	0.330	0.330	0.421
F(000)	860	2816	776	1336	1368	936
θ range	1.89<θ<26.35	0.94<θ<26.37	1.79<θ<26.36	1.67<θ<26.38	1.46<θ<26.37	1.88<θ<26.37
Reflections collected/unique	18794/4868	55175/8159	16140/8103	29445/7810	30339/7917	19491/5050
Completeness to θ [%]	99.9	99.9	98.4	100	100	99.8
Data/restraints/parameters	4868/0/209	8159/0/335	8103/0/335	7810/0/316	7917/0/319	5050/3/269

Goodness of fit on F^2	1.14	1.11	1.05	1.13	1.25	1.26
Final R indices [I>2σ(I)]	R1=0.049 wR2=0.122	R1=0.072 wR2=0.180	R1=0.025 wR2=0.066	R1=0.046 wR2=0.097	R1=0.075 wR2=0.148	R1=0.065 wR2=0.145
R indices (all data)	R1=0.053 wR2=0.125	R1=0.078 wR2=0.184	R1=0.029 wR2=0.068	R1=0.049 wR2=0.099	R1=0.077 wR2=0.149	R1=0.070 wR2=0.148
Largest diff. Peak/hole [e$^-$/ Å^3]	0.86/-0.30	2.97/-0.54	0.67/-0.31	0.51/-0.23	0.77/-0.76	0.67/-0.28
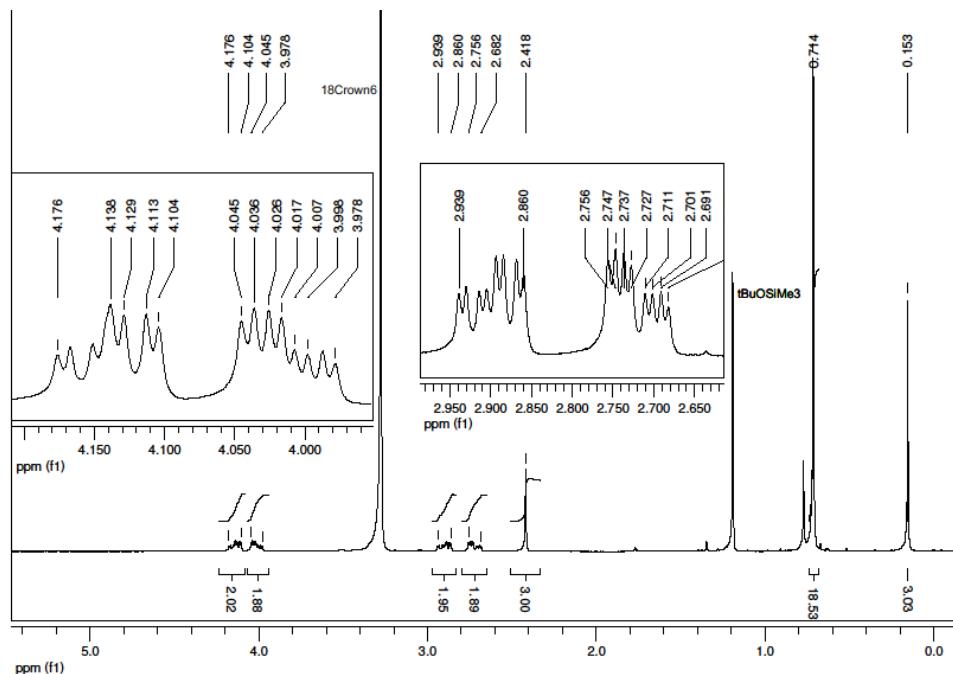
Figure S1. ^{1}H NMR spectrum of **4** in C_6C_6.
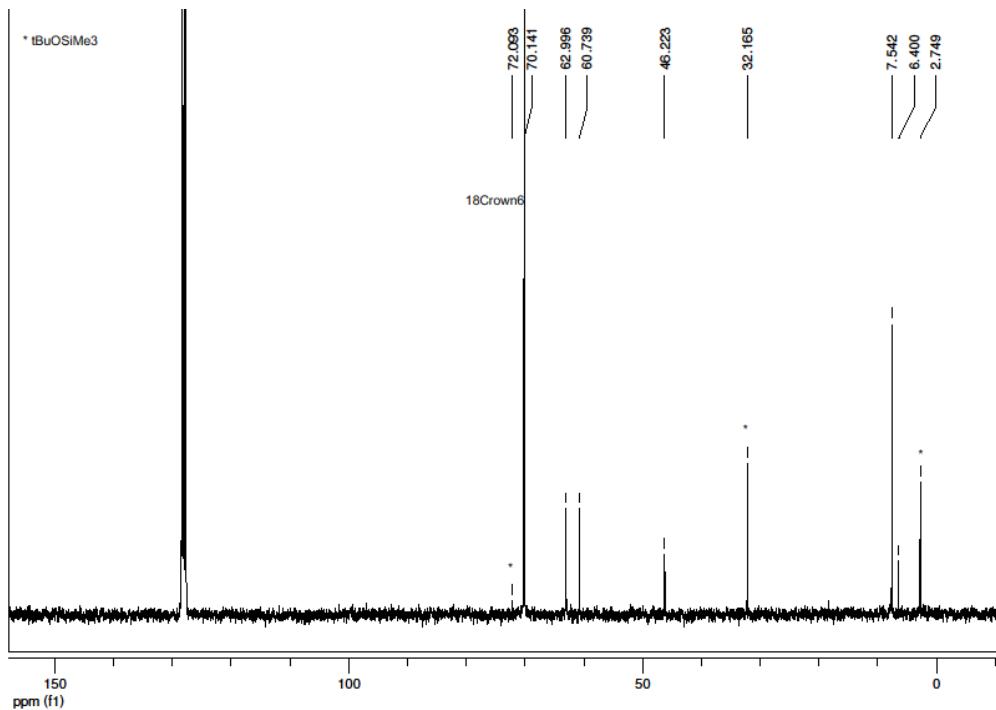
Figure S2. ^{13}C NMR spectrum of **4** in C_6C_6.
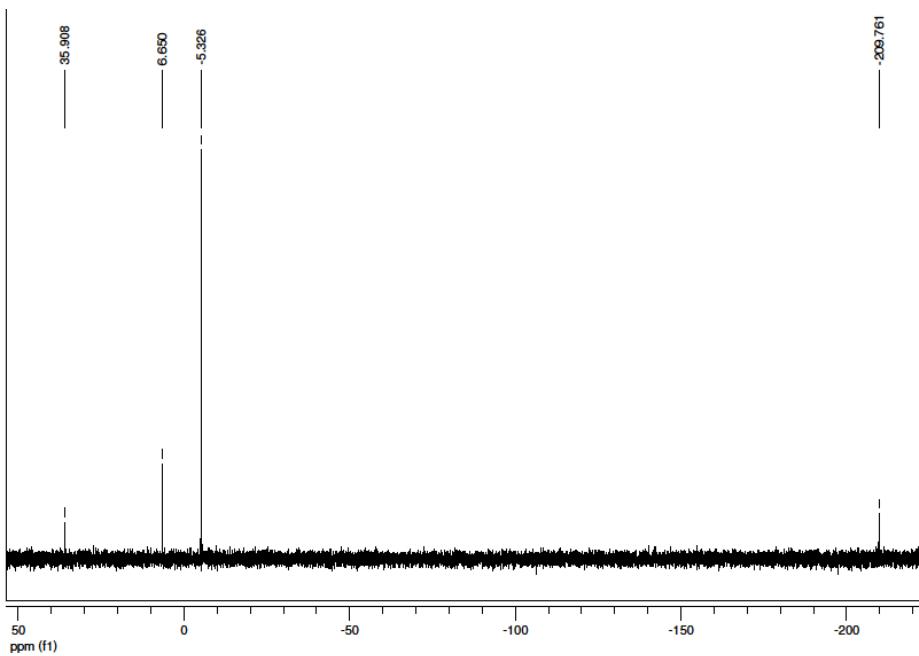
Figure S3. ^{29}Si INEPT NMR spectrum of **4** in C$_6$C$_6$.
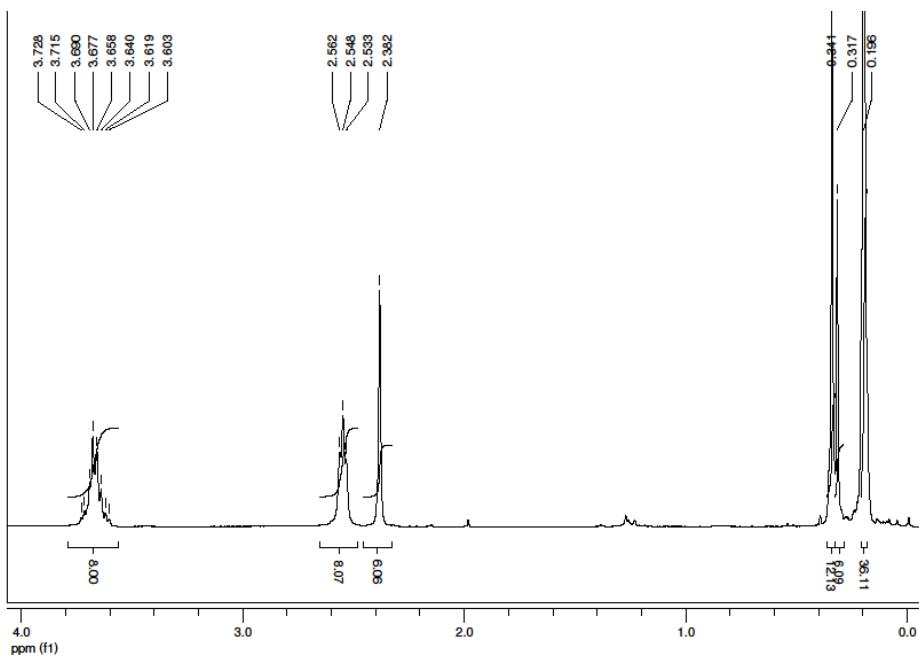
Figure S4. ^{1}H NMR spectrum of **5** in CDCl$_3$.
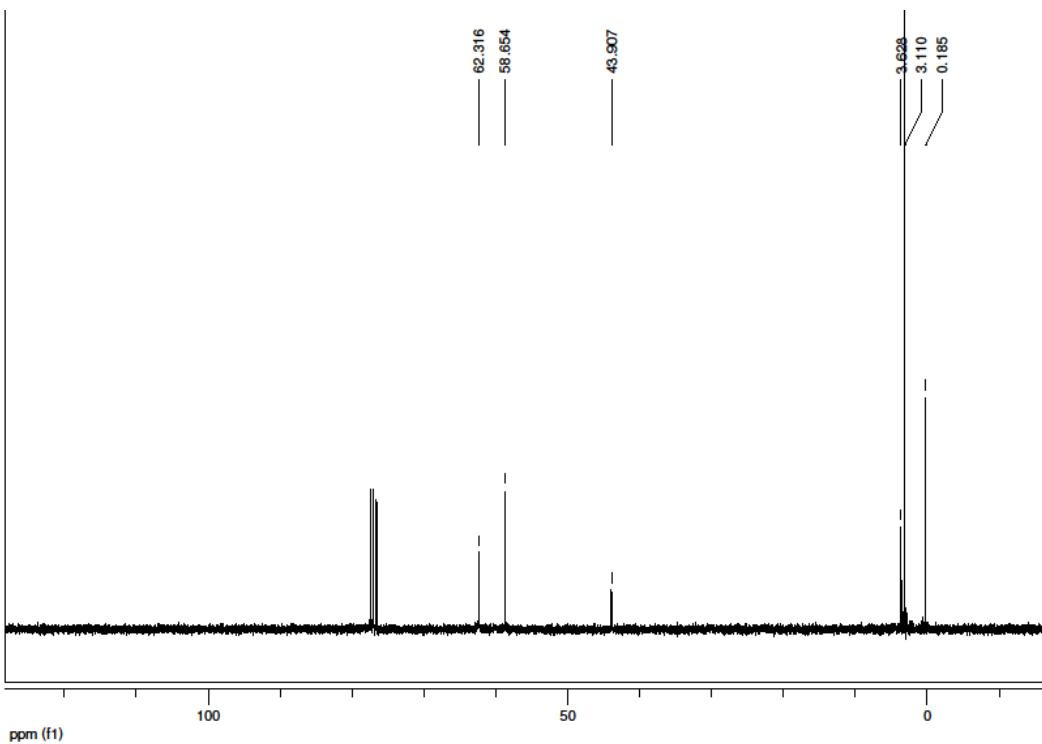
Figure S5. ^{13}C NMR spectrum of **5** in CDCl$_3$.
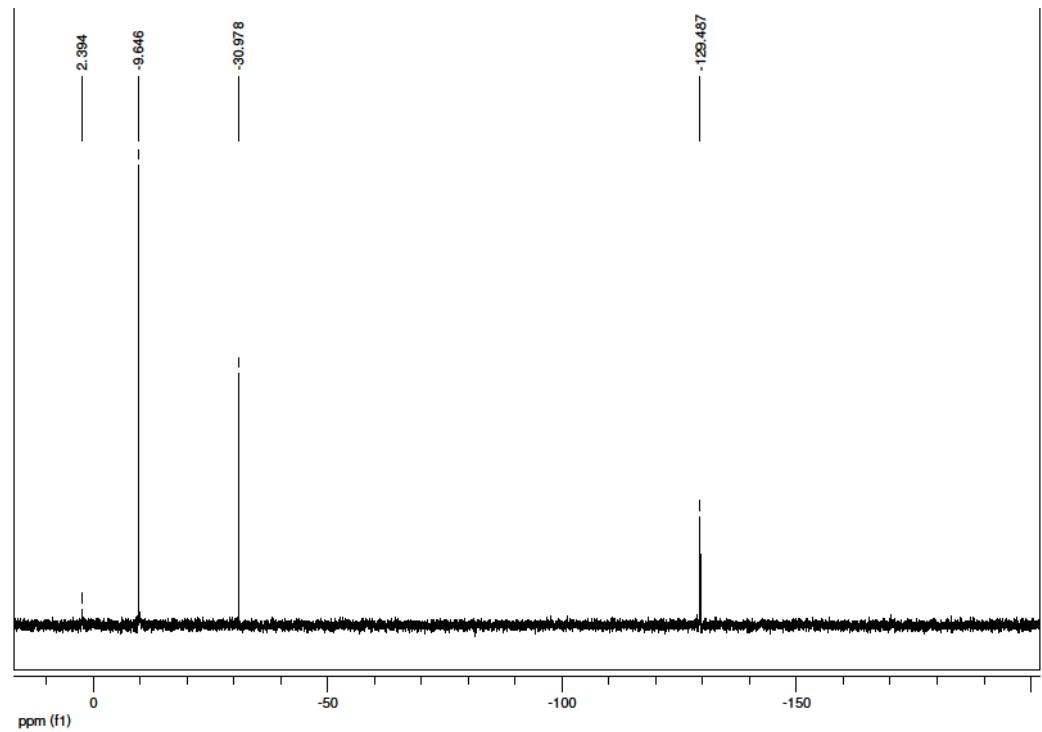
Figure S6. ^{29}Si INEPT NMR spectrum of **5** in CDCl$_3$.
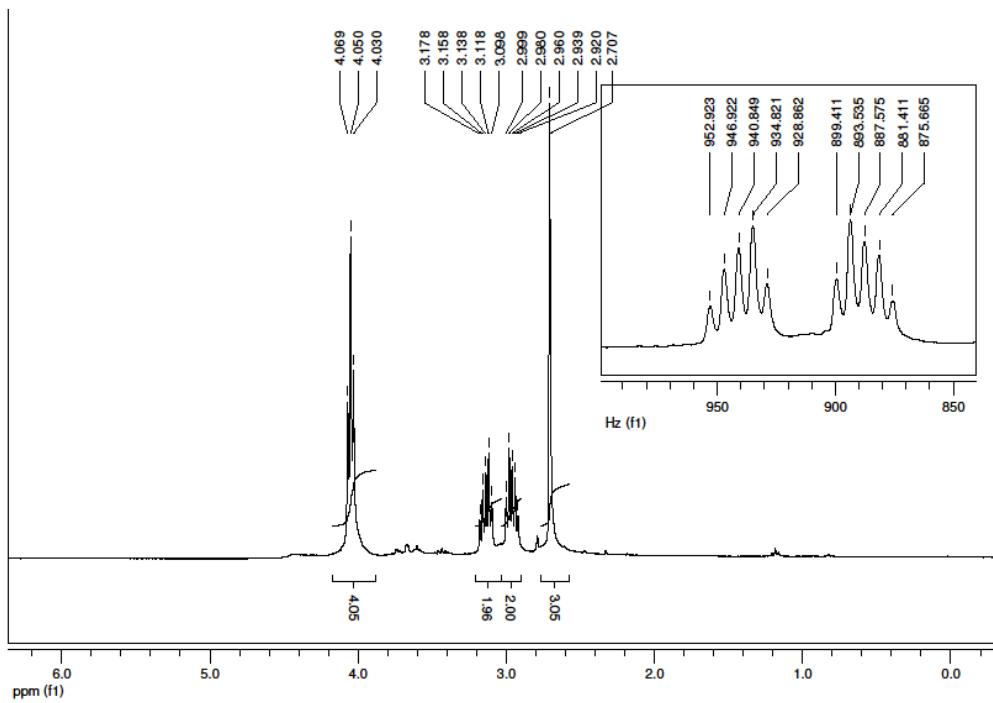
Figure S7. ^{1}H NMR spectrum of **7** at -40°C in CDCl$_3$.
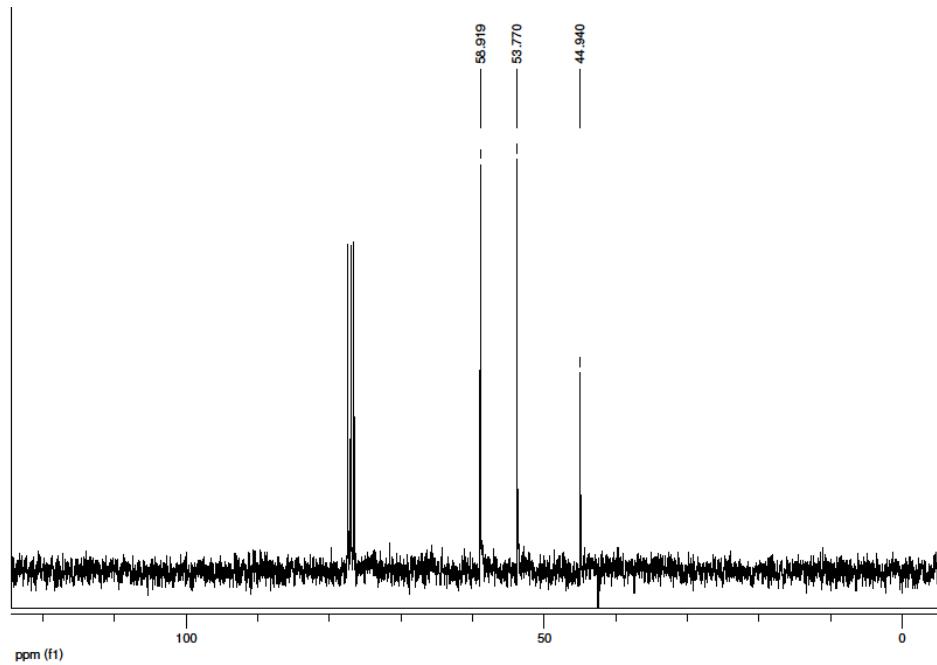

Figure S8. ^{13}C NMR spectrum of **7** in CDCl$_3$.
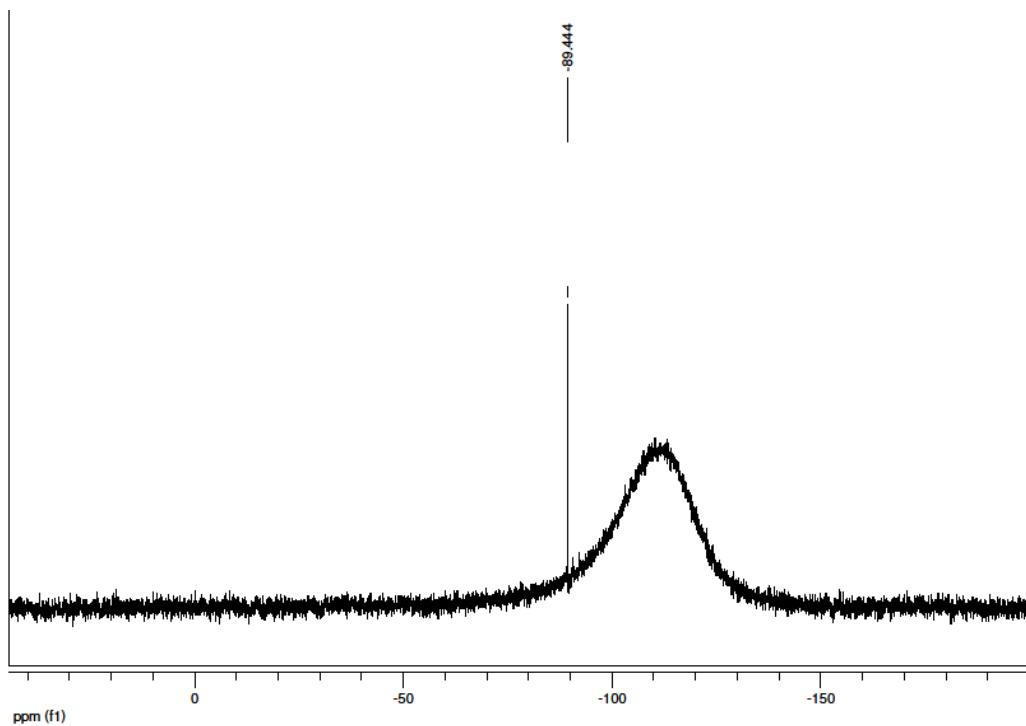
Figure S9. ^{29}Si inversed gated NMR spectrum of **7** in CDCl$_3$.
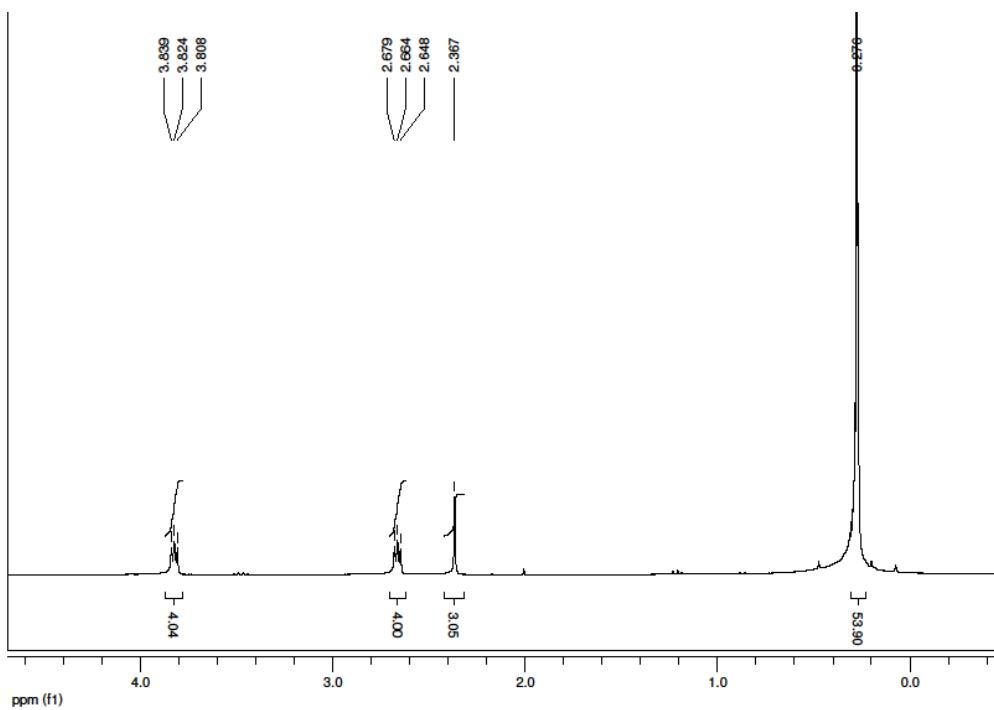

Figure S10. ^{1}H NMR spectrum of **8** in CDCl$_3$.
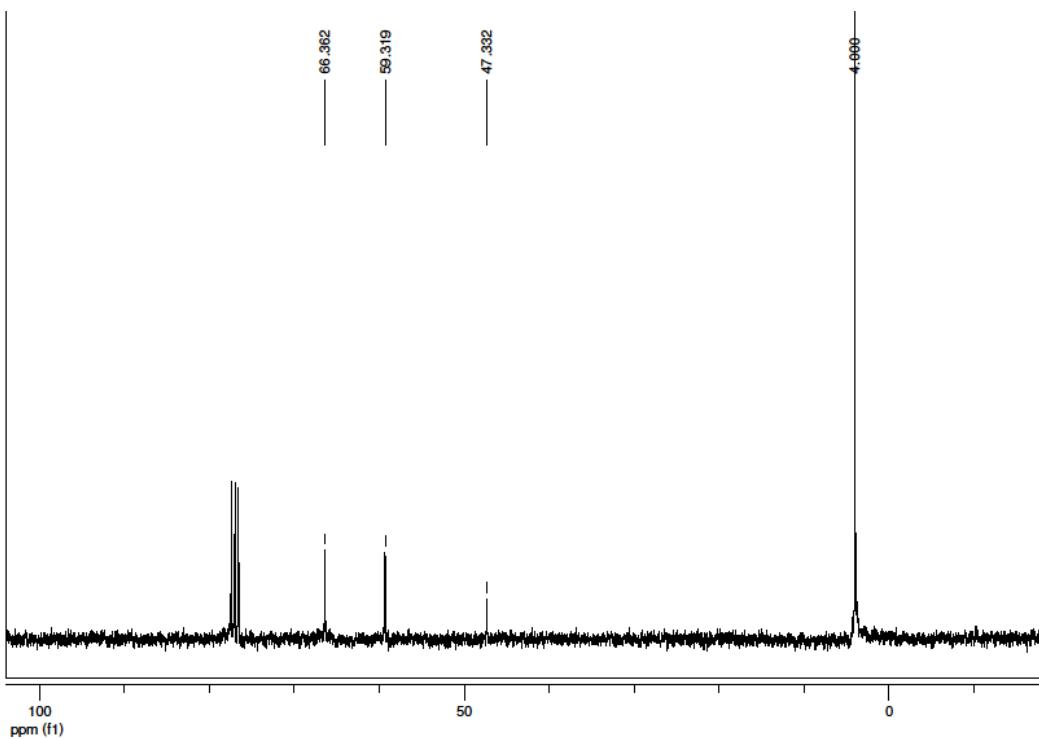
Figure S11. ^{13}C NMR spectrum of **8** in CDCl$_3$.
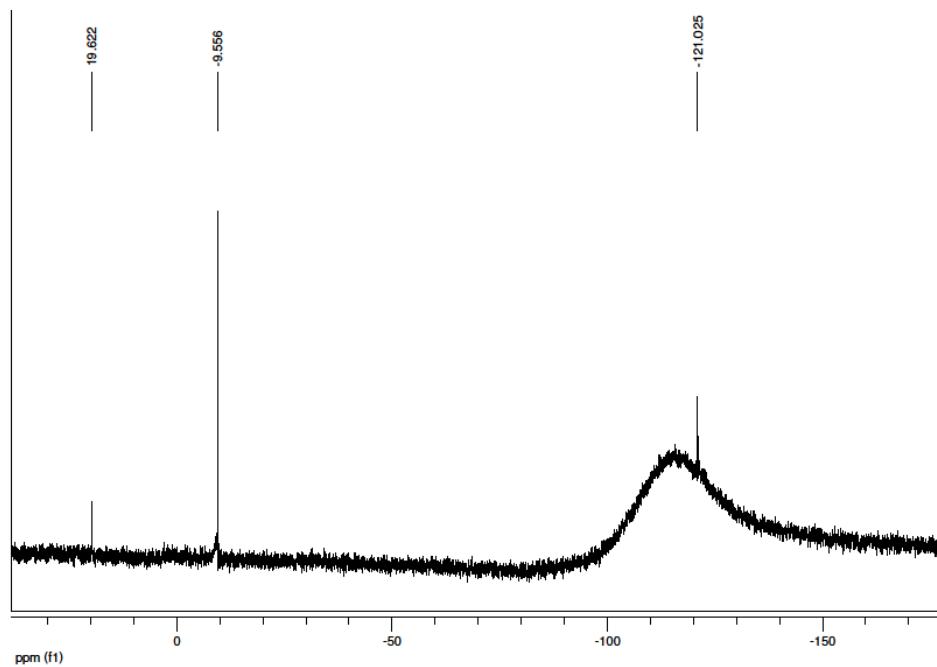
Figure S12. ^{29}Si inversed gated NMR spectrum of **8** in CDCl$_3$.
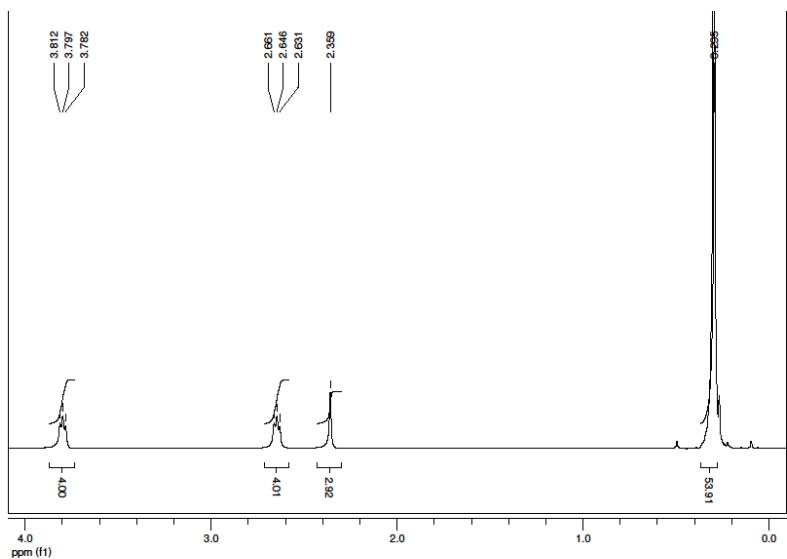
Figure S13. ^{1}H NMR spectrum of **8a** in CDCl$_3$.
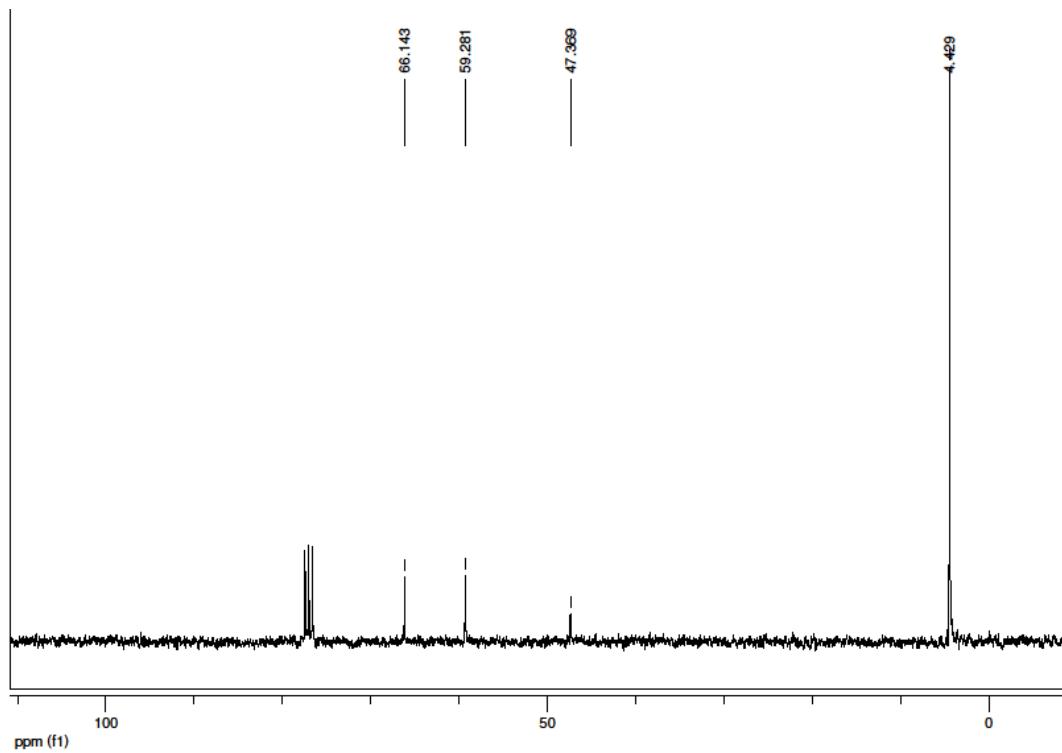
Figure S14. ^{13}C NMR spectrum of **8a** in CDCl$_3$.
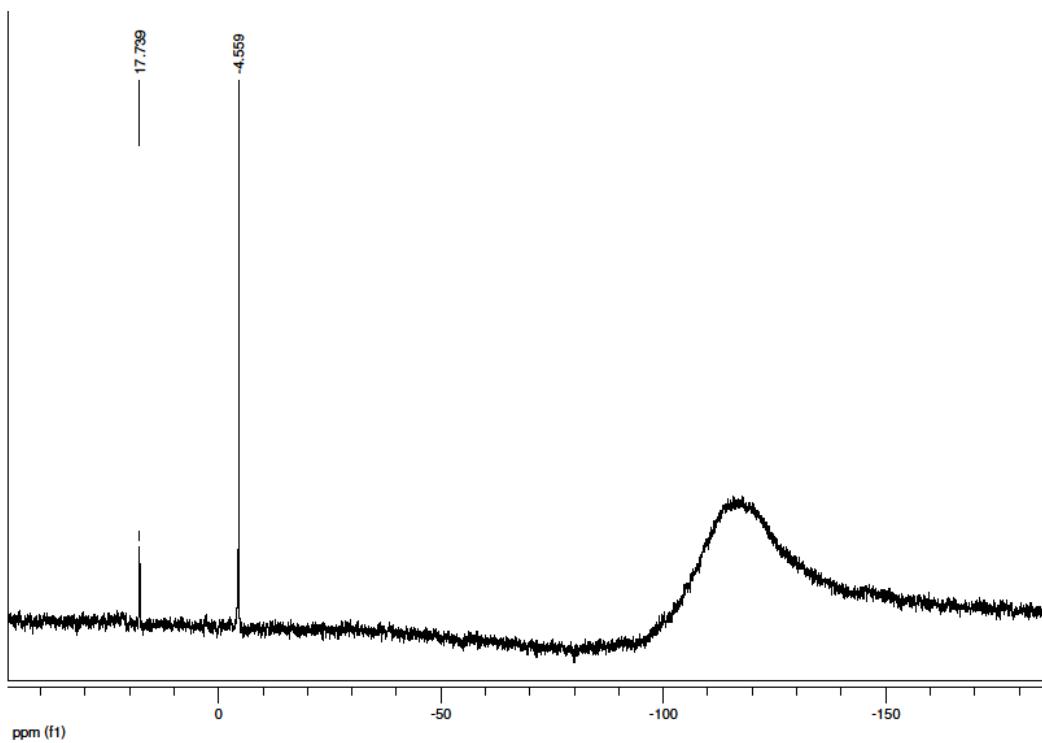
Figure S15. ^{29}Si inversed gated NMR spectrum of **8a** in CDCl$_3$.
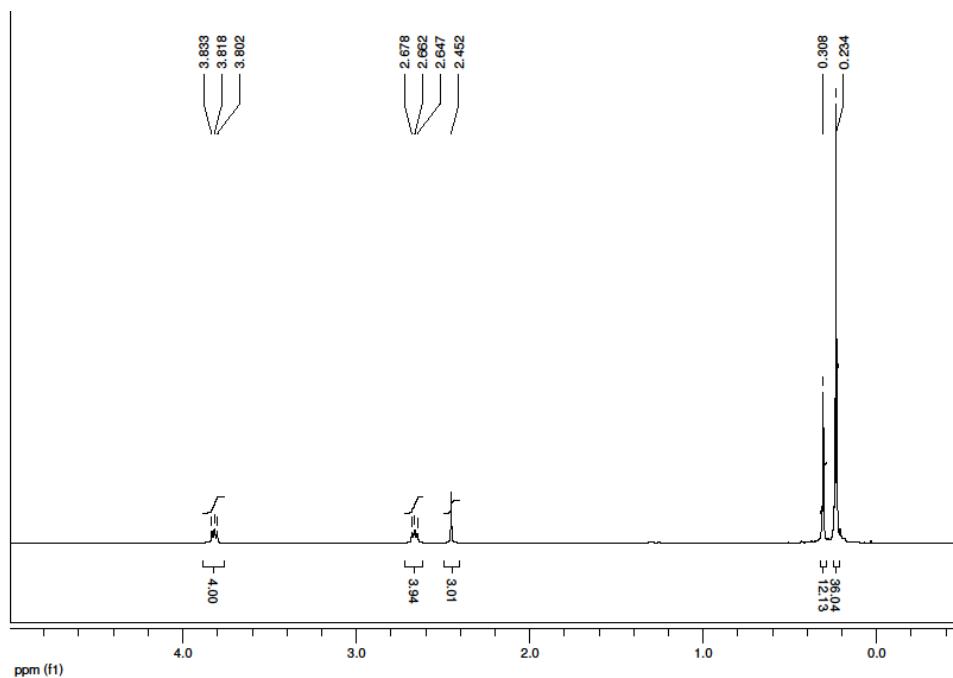
Figure S16. ^{1}H NMR spectrum of **9** in CDCl$_3$.
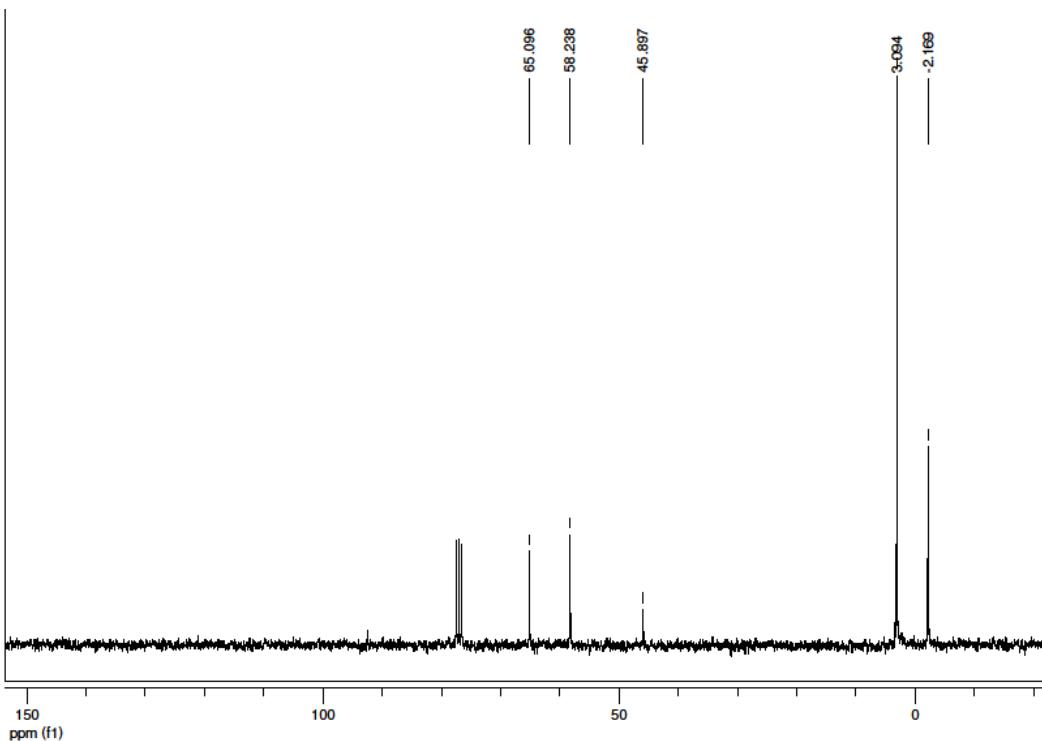
Figure S17. ^{13}C NMR spectrum of **9** in CDCl$_3$.
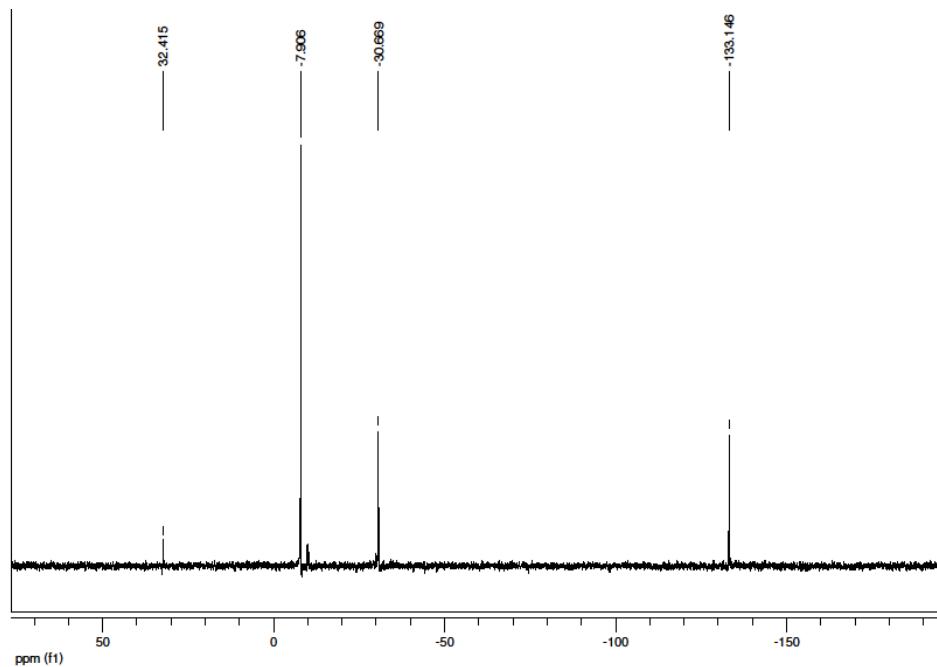
Figure S18. ^{29}Si INEPT NMR spectrum of **9** in CDCl$_3$.

S10
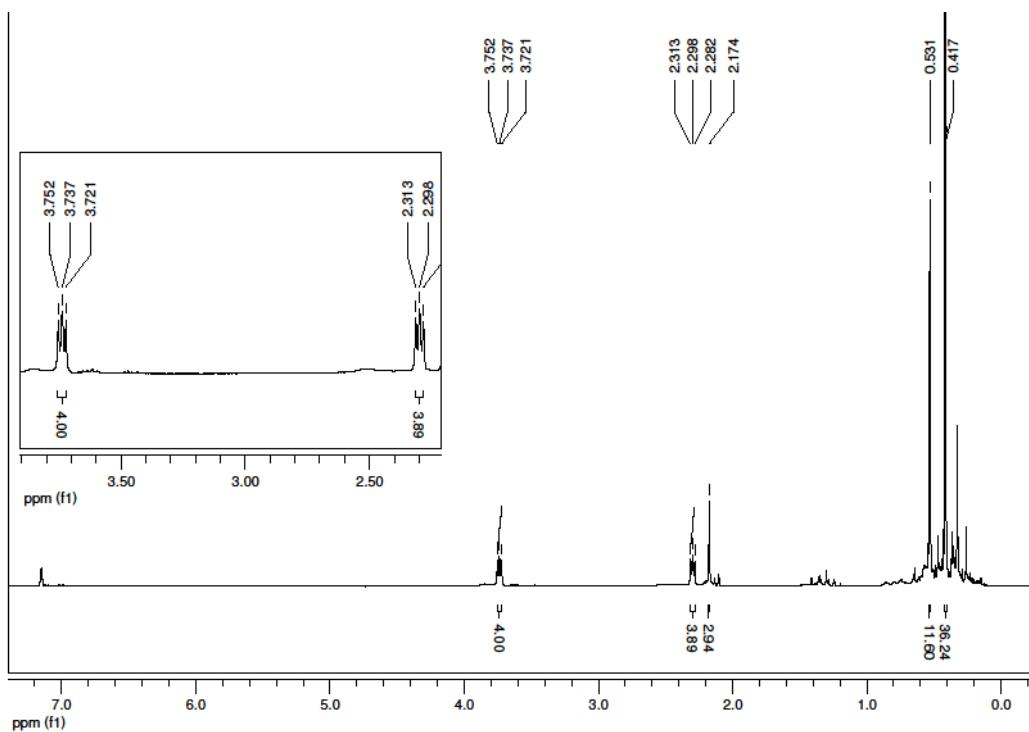
Figure S19. ^1H NMR spectrum of **10** in C_6D_6.
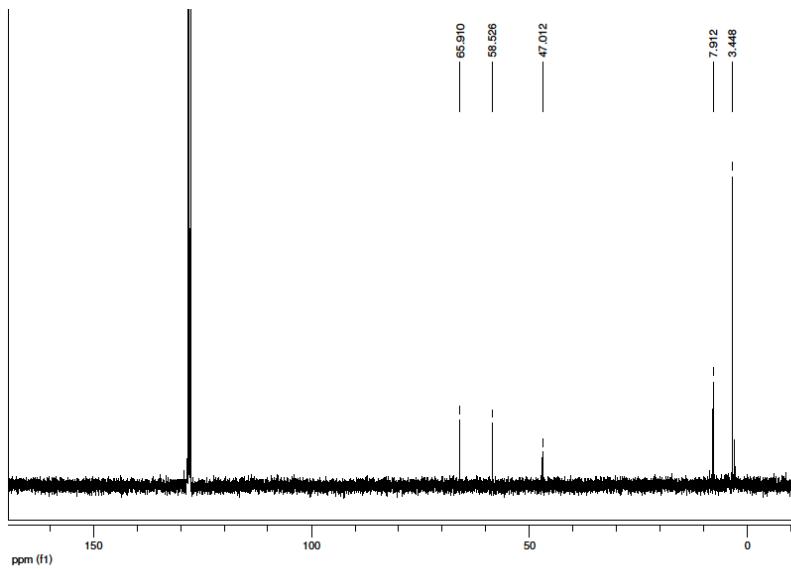
**Figure S20.** ^{13}C NMR spectrum of **10** in C_6D_6.
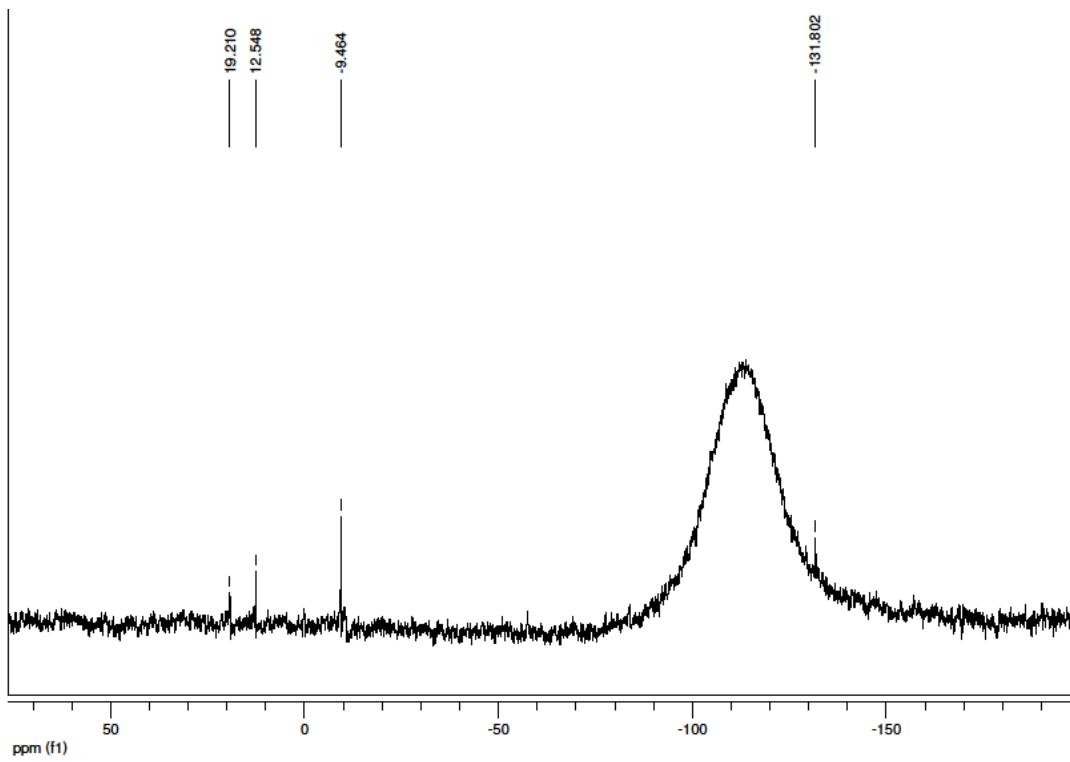
**Figure S21.** ^{29}Si inversed gated NMR spectrum of **10** in C_6D_6.
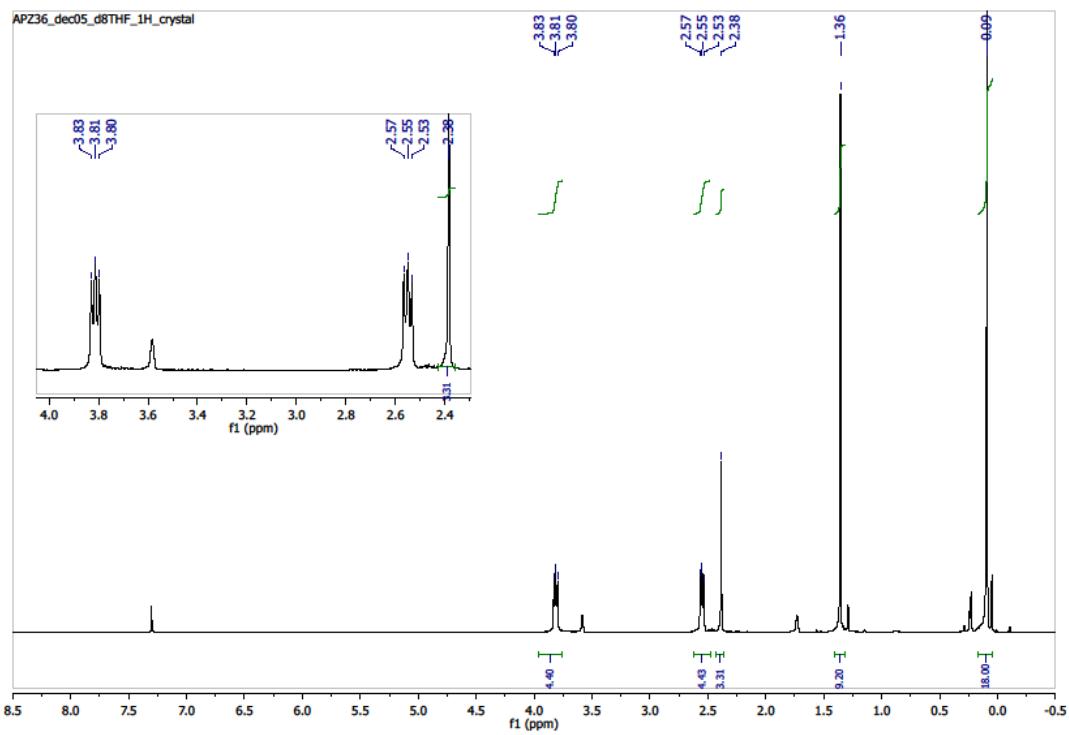


Figure S22. ^1H NMR spectrum of **12** in THF-d_8.

—129.18

—72.52

—62.77
—61.21

—48.13

—33.15

—7.02

C6H6

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
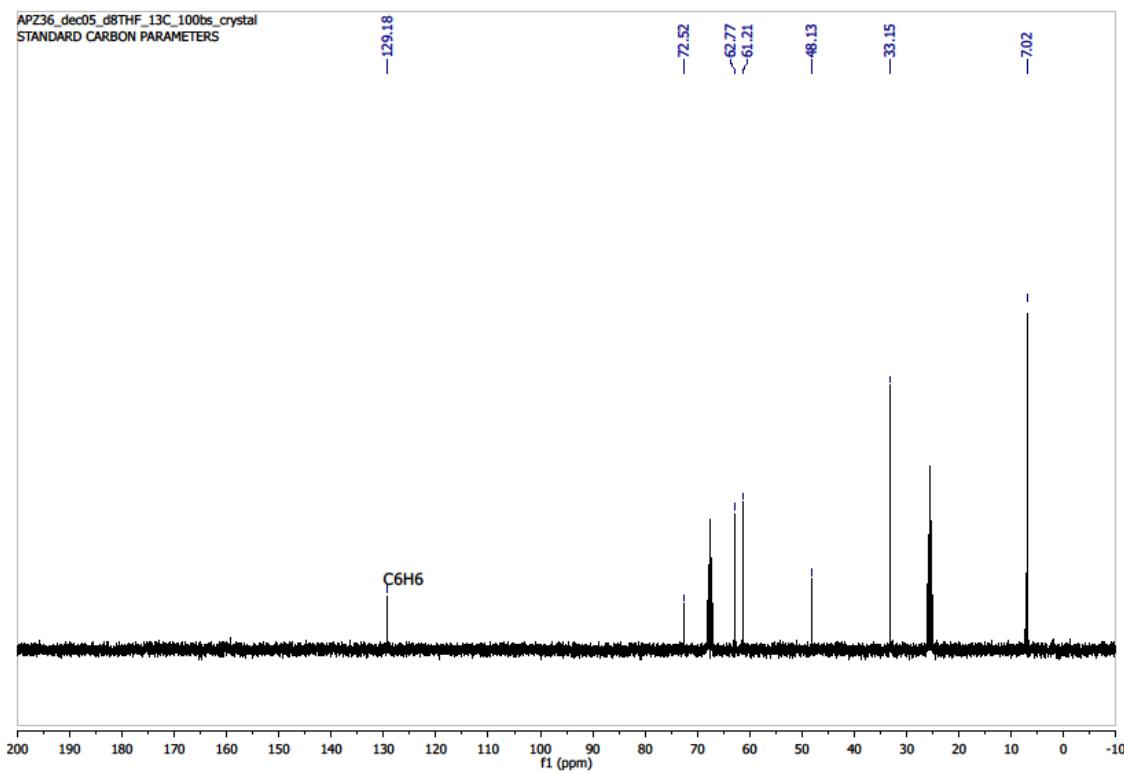
Figure S23. ^{13}C NMR spectrum of **12** in THF-d$_8$.

—5.44
—12.96

—208.67

140 120 100 80 60 40 20 0 -10 -30 -50 -70 -90 -110 -140 -170 -200 -230
f1 (ppm)
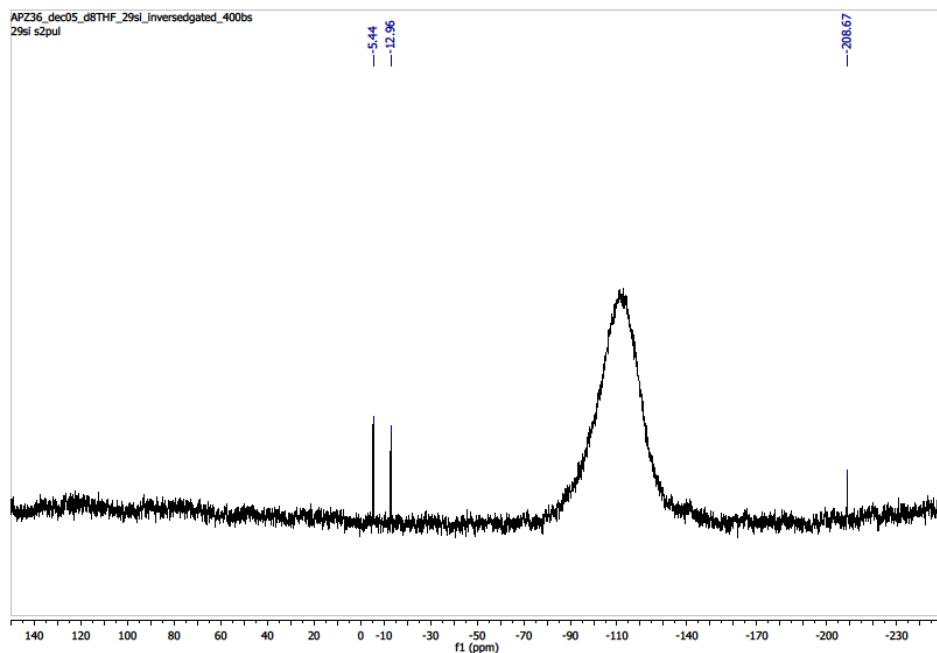
Figure S24. ^{29}Si inversed gated NMR spectrum of **12** in THF-d$_8$..
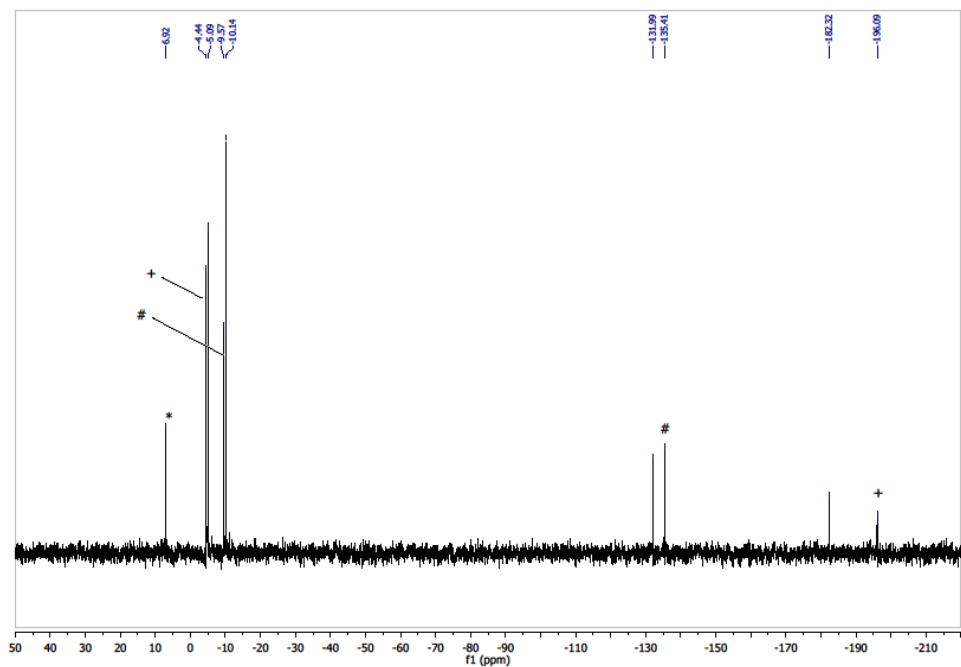
Figure S25. ^{29}Si INEPT NMR of **13** (in the reaction mixture together with Me₃SiOᵗBu (*), (Me₃Si)₄Si (#), and (Me₃Si)₃SiK (+)) in DME.
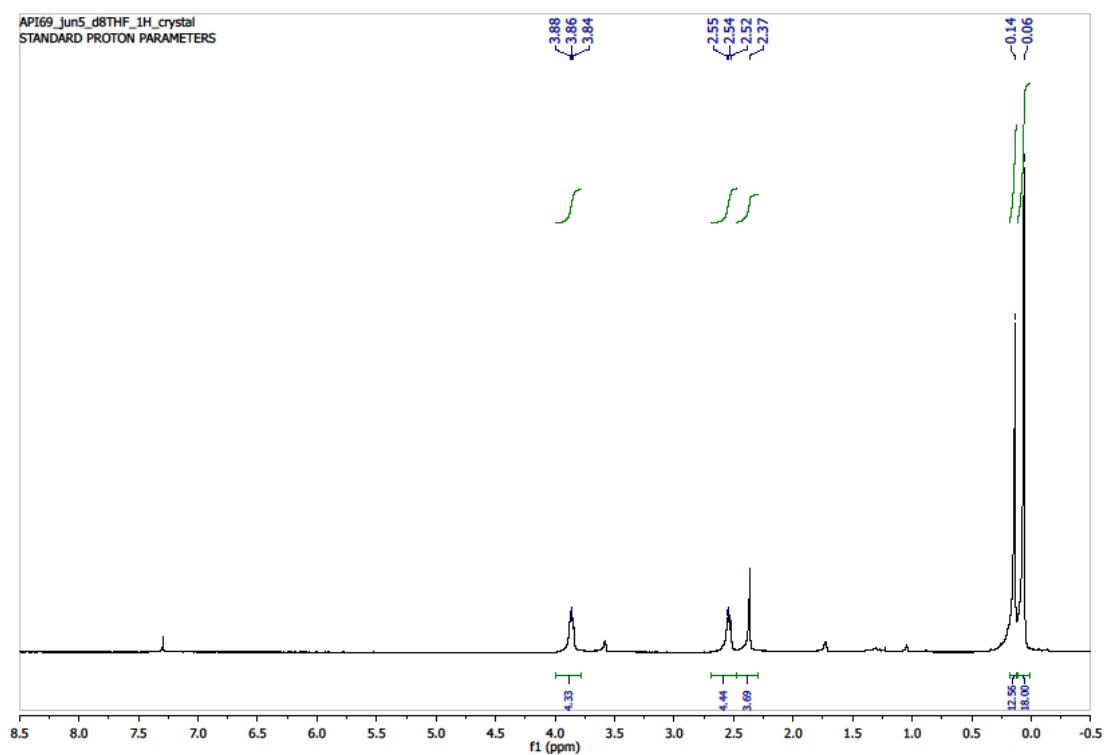

**Figure S26.** ^{1}H NMR spectrum of **14** in THF-d₈.
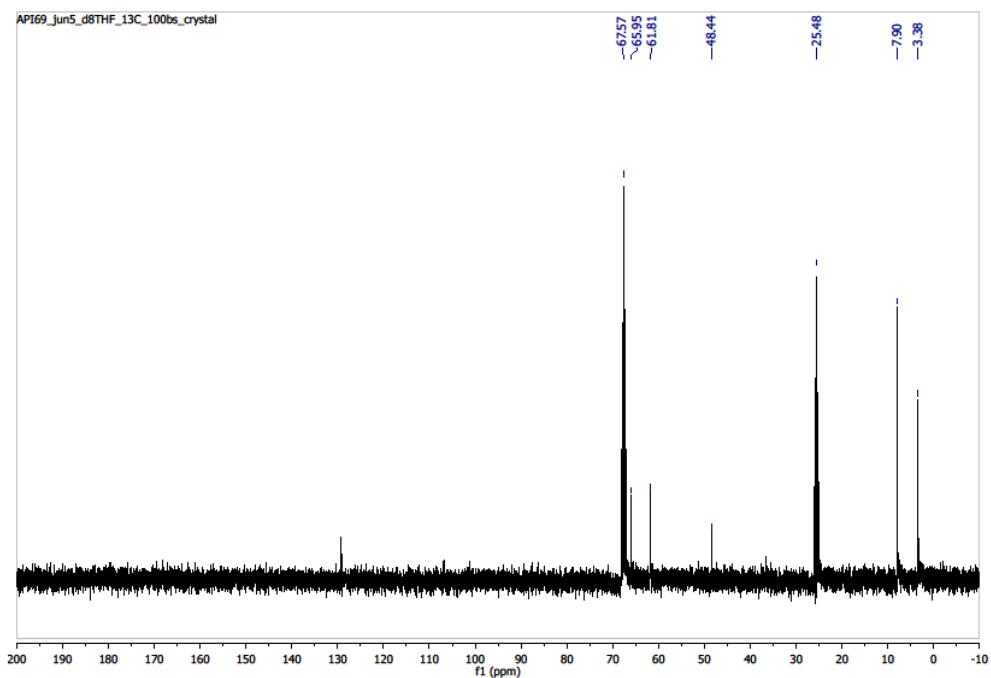
**Figure S27.** ^{13}C NMR spectrum of **14** in THF-d[8].
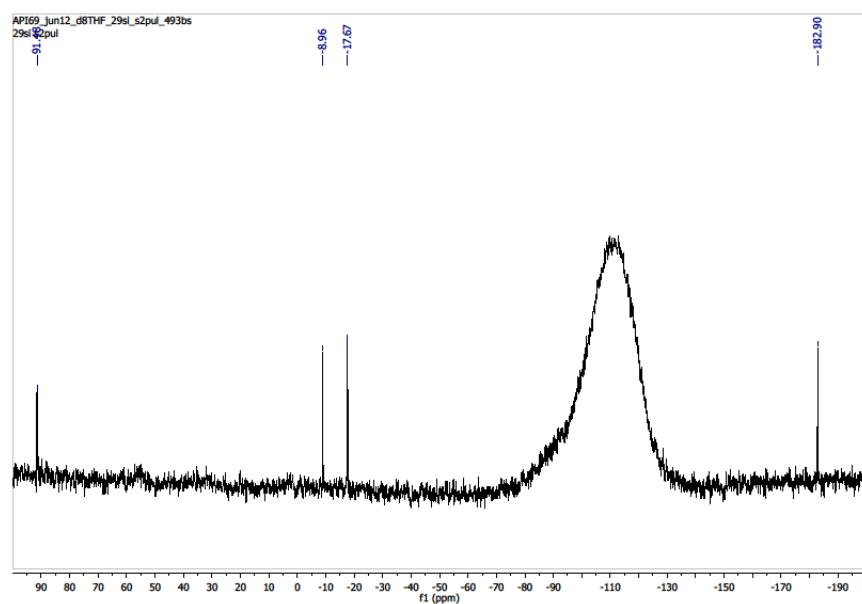
**Figure S28.** ^{29}Si inversed gated NMR spectrum of **14** in THF-d[8].
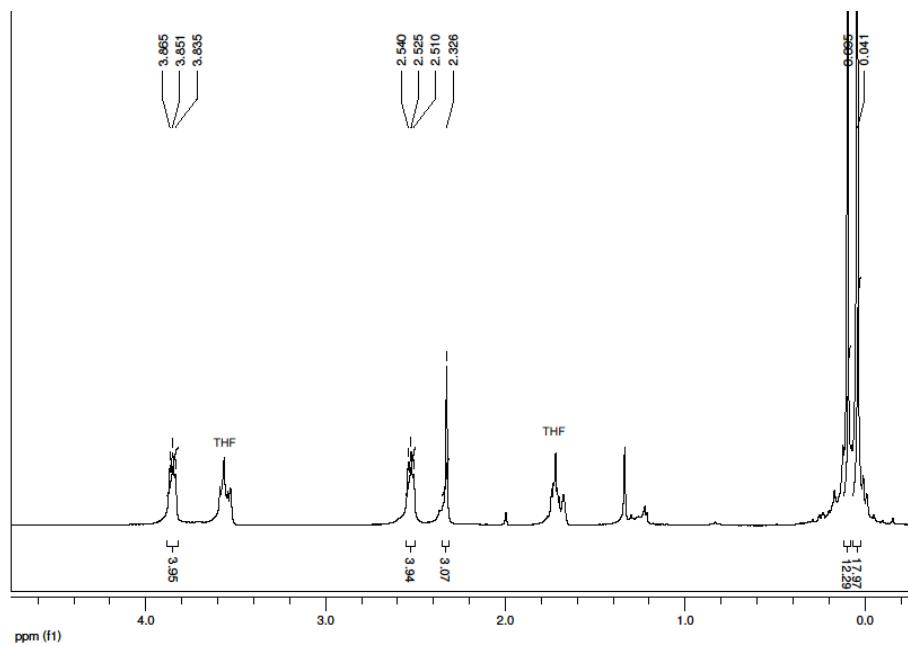
Figure S29. ¹H NMR spectrum of **15** in THF-d₈.
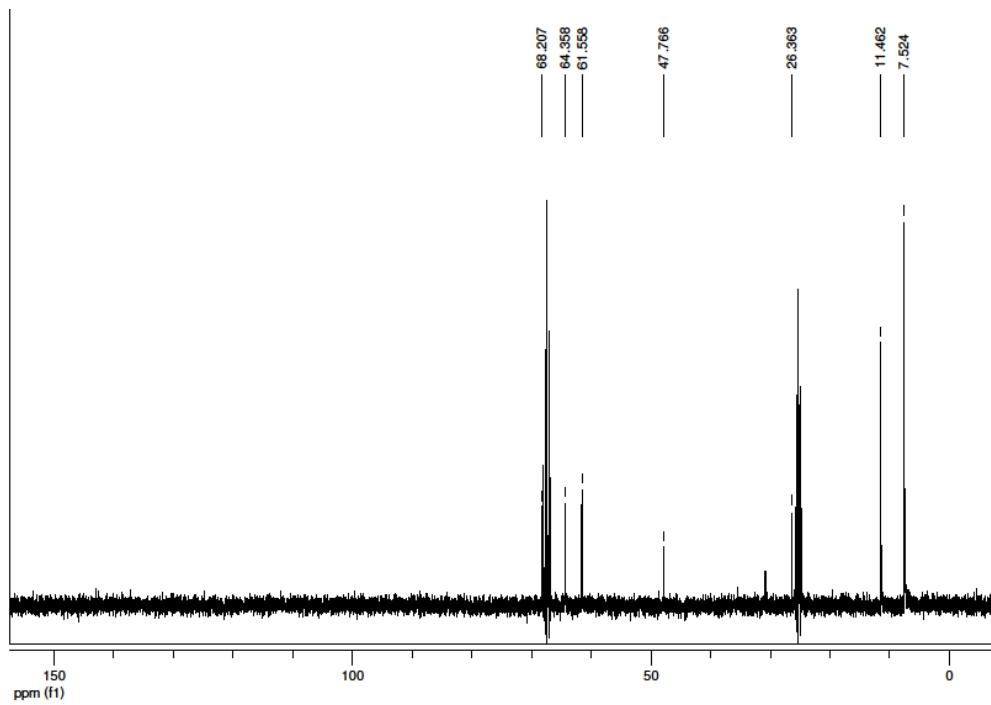
**Figure S30.** ¹³C NMR spectrum of **15** in THF-d₈.
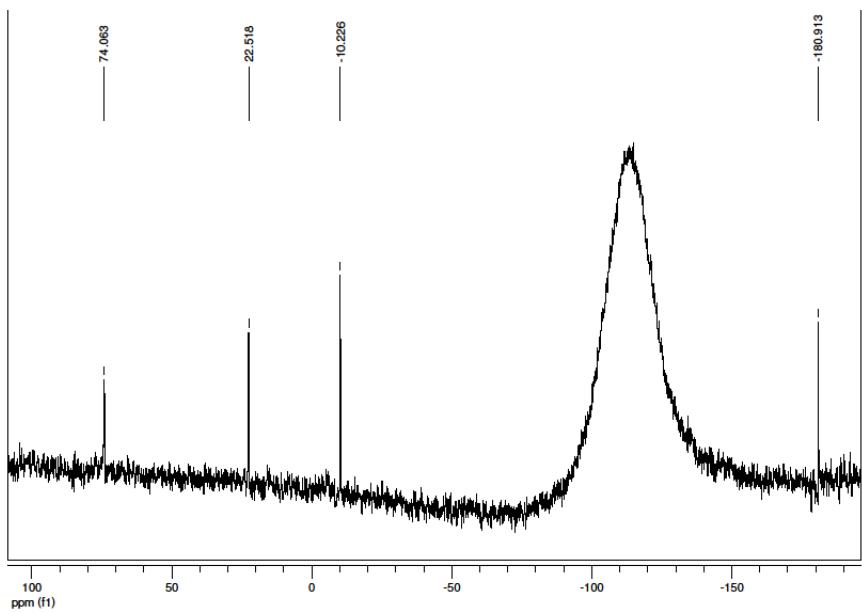
Figure S31. ^{29}Si inversed gated NMR spectrum of **15** in THF-d$_8$.