Electronic Supplementary Material (ESI) for Dalton Transactions.

Table of contents:

General experimental techniques

All manipulations were performed either under solvent vapour pressure or dry argon using glovebox and *Schlenk* techniques. Dichloromethane was dried by refluxing over CaH_2 and CD_2Cl_2 was dried over CaH_2 and vacuum transferred directly into the *J. Young* NMR tubes. $BeCl_2$,[1] 1,2-disila[12]crown-4,[2] 1,2,4,5-tetrasila[12]crown-4,[3] 1,2,7,8-tetrasila[12]crown-4,[2] 1,2-disila[9]crown-3,[4] 1,2-disila[15]crown-5,[2] 1,2-disila-benzo[18]crown-6[5] and 2,2'-[1,2-phenylenebis(oxy)]diethanol[6] were prepared according to literature procedures. Due to the expected extreme toxicity of the beryllium compounds no elemental analysis or mass spectrometry could be performed of these.

NMR spectroscopy

^{1}H, ^{9}Be, ^{13}C and ^{29}Si NMR spectra were recorded on *Bruker* Avance III HD 300 and Avance III 500 NMR spectrometers. ^{1}H NMR (300 / 500 MHz) and ^{13}C NMR (76 / 126 MHz) chemical shifts are given relative to the solvent signal CD_2Cl_2 (5.32 and 53.8 ppm) while ^{9}Be (42 / 70 MHz) used 0.43 [M] $BeSO_4$ in D_2O and ^{29}Si (60 / 99 MHz) used neat $SiMe_4$ as an external standard. NMR spectra were processed with the MestReNova software.[7]

IR spectroscopy

IR spectra were recorded on a *Bruker* alpha FT-IR spectrometer equipped with a diamond ATR unit in an argon filled glovebox. Processing of the spectra was performed with the OPUS software package[8] and OriginPro 2017.[9]

Single crystal X-ray diffraction

Single crystals were selected under exclusion of air in perfluorinated polyether (Fomblin YR 1800, *Solvay Solexis*) and mounted using the *MiTeGen* MicroLoop system. X-ray diffraction data were collected using the monochromated Mo-K_α radiation of a *Stoe* IPDS2 diffractometer equipped with an Image Plate detector or the monochromated Mo-K_α radiation of a *Bruker* D8 Quest diffractometer equipped with a microfocus source and a *CMOS* Photon 100 detector. The diffraction data were reduced with the X-Area software.[10] The structures were solved using Direct Methods (SHELXS-2013/1 & SHELXT-2015) and refined against F^2 (SHELXL-2016/4) using the ShelXle (**4**) and OLEX2 (**5**) software packages.[11] All atoms were located by Difference Fourier synthesis and non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined isotropically.

Mass spectrometry

Mass spectrometry was performed on a *Thermo Fischer Scientific* LTQ-FT Ultra using a electrospray ionization (ESI) source.

Synthesis and characterization

1,2-disila-benzo[12]crown-4 (3):
1.00 g of 2,2'-[1,2-Phenylenebis(oxy)]diethanol (6.02 mmol, 1.00 eq) and 1.67 mL of NEt_3 (12.04 mmol, 2.0 eq) were dissolved in 50 ml of THF. Subsequently, 1.14 ml of $Si_2Me_4Cl_2$ (6.02 mmol, 1.0 eq) dissolved in 50 ml of THF was added over a period of 60 min. The resulting white suspension was then stirred overnight and freed of the solvent in vacuo. The residue was extracted with 50 ml of *n*-pentane followed by filtration. Removing the solvent under reduced pressure yielded the crown ether as a colourless oil (1.32 g, 70%). For purification, the crown ether was sublimated by slowly raising the temperature to 90 °C under fine vacuum ($1 \cdot 10^{-3}$ mbar) and was then obtained as a colourless wax.
^1H NMR: (300 MHz, CD_2Cl_2) $\delta = 0.24$ (s, 12H, $SiCH_3$), 3.96-3.99 (m, 4H, CH_2), 4.04-4.08 (m, 4H, CH_2), 6.92 (s, 4H, CH_{AR}) ppm. $^{13}C\{^1H\}$ NMR: (126 MHz, CD_2Cl_2) $\delta = 0.8$ (s, $SiCH_3$), 63.6 (s, CH_2), 72.5 (s, CH_2), 116.8 (s, C_{AR}), 122.5 (s, C_{AR}), 150.5 (s, C_{ARq}). $^{29}Si\{^1H\}$ NMR (60 MHz, CD_2Cl_2) $\delta = 11.5$ ppm (s, $SiCH_3$). HR-MS(ESI$^+$): m/z calcd. for $[C_{14}H_{24}O_4Si_2+H]^+$: 313.1291; found: 313.1285 (100). Anal. calcd. for $C_{14}H_{24}O_4Si_2$: C, 53.81; H 7.74. Found: C, 53.67; H, 7.76.

***In situ* NMR monitoring:**
5.0 mg (0.06 mmol) $BeCl_2$ and 0.06 mmol of the ligand were placed in a *J. Young* NMR tube and 0.5 ml CD_2Cl_2 were vacuum transferred into the tube. NMR spectra were recorded in regular time intervals (see NMR spectra below).

Crystallisation of compounds 4 and 5:
$BeCl_2$ (5.0 mg, 0.06 mmol) and 0.06 mmol of ligand **2a** or **3** were placed in a *J. Young* NMR tube and 0.5 ml CD_2Cl_2 were vacuum transferred into the tube. After standing at ambient temperature for two months crystals of compounds **4** suitable for single crystal X-ray diffraction analysis grew on the wall of the vessel, while layering of the reaction solution with benzene led to the formation of crystals of compound **5**.

Synthesis of samples for IR spectroscopy:
8.0 mg (0.10 mmol) $BeCl_2$ and 0.10 mmol of ligand **2a**, **2c** or **3** respectively were weighed into a *Schlenk* tube and 5.0 ml dichloromethane was added *via* cannula and the reaction mixture was stirred for two hours at ambient temperature. Afterwards the solvent was removed *in vacuo* and the received colourless oils were analysed via ATR FT-IR spectroscopy.

Spectroscopic data:

$[Be(O(C_2H_4O)_3)]$ (8a): ^1H NMR (300 MHz, CD_2Cl_2) $\delta = 3.75$ (bs, $\omega_{1/2} = 6.3$ Hz, 4H, CH_2), 3.83 (bs, $\omega_{1/2} = 15.0$ Hz, 4H, CH_2), 4.16 (bs, $\omega_{1/2} = 18.7$ Hz, 4H, CH_2). ^9Be NMR (42 MHz, CD_2Cl_2) $\delta = 2.7$ ($\omega_{1/2} = 18.3$ Hz). ^{13}C NMR (126 MHz, CD_2Cl_2) $\delta = 64.2$ (bs, $\omega_{1/2} = 43.8$ Hz, CH_2), 70.0 (bs, $\omega_{1/2} = 62.0$ Hz, CH_2), 70.9 (bs, $\omega_{1/2} = 108.5$ Hz, CH_2).

$[Be(O(C_2H_4O)(SiMe_2SiMe_2O)(C_2H_4O)]$ (8b): ^9Be NMR (70 MHz, CD_2Cl_2) $\delta = 3.4$ ($\omega_{1/2} = 69.0$ Hz). Due to extremely broad signals and the presence of compounds **10b** and **2b** no signal assignment could be performed in the ^1H, ^{29}Si and ^{13}C NMR spectra.

$[Be(O(C_2H_4O)(C_6H_4O)(C_2H_4O))]$ (8Ph): ^9Be NMR (42 MHz, CD_2Cl_2) $\delta = 3.1$ ($\omega_{1/2} = 59.1$ Hz). Due to extremely broad signals and the presence of compounds **10Ph** and **3** no signal assignment could be performed in the ^1H and ^{13}C NMR spectra.

$[\mathrm{BeCl(O(C_2H_4O)_3Me)}]$ (9): ^{1}H NMR (300 MHz, $\mathrm{CD_2Cl_2}$) δ = 3.27 – 3.70 (m, 4H, $\mathrm{C}H_2$), 3.87 (bs, $\omega_{1/2}$ = 25.6 Hz, 7H, $\mathrm{C}H_2$, $\mathrm{C}H_3$), 4.14 (bs, $\omega_{1/2}$ = 15.7 Hz, 4H, $\mathrm{C}H_2$). ^{9}Be NMR (42 MHz, $\mathrm{CD_2Cl_2}$) δ = 3.6 ($\omega_{1/2}$ = 30.1 Hz). ^{13}C NMR (126 MHz, $\mathrm{CD_2Cl_2}$) δ = 59.4 (bs, $\omega_{1/2}$ = 61.1 Hz, $C\mathrm{H_3}$), 63.8 (bs, $\omega_{1/2}$ = 37.9 Hz, $C\mathrm{H_2}$), 70.4 (bs, $\omega_{1/2}$ = 21.7 Hz, $C\mathrm{H_2}$), 71.1 (bs, $\omega_{1/2}$ = 53.6 Hz, $C\mathrm{H_2}$), 72.1 (bs, $\omega_{1/2}$ = 81.8 Hz, $C\mathrm{H_2}$).

$[\mathrm{BeCl(O(C_2H_4O)_3SiMe_2SiMe_2Cl)}]$ (10a): ^{1}H NMR (500 MHz, $\mathrm{CD_2Cl_2}$) δ = 0.31 (s, 6H, $\mathrm{OSi}Me_2\mathrm{SiMe_2Cl}$), 0.51 (s, 6H, $\mathrm{OSiMe_2Si}Me_2\mathrm{Cl}$), 3.49 – 3.53 (m, 4H, $\mathrm{C}H_2$), 3.58 (s, 4H, $\mathrm{C}H_2$), 3.71 – 3.76 (m, 4H, $\mathrm{C}H_2$). ^{9}Be NMR (70 MHz, $\mathrm{CD_2Cl_2}$) δ = 3.4 ($\omega_{1/2}$ = 60.5 Hz). ^{13}C NMR (126 MHz, $\mathrm{CD_2Cl_2}$) δ −1.0 ($\mathrm{OSi}Me_2\mathrm{SiMe_2Cl}$), 2.7 ($\mathrm{OSiMe_2Si}Me_2\mathrm{Cl}$), 63.9 ($C\mathrm{H_2}$), 71.1 ($C\mathrm{H_2}$), 73.0 ($C\mathrm{H_2}$). ^{29}Si NMR (99 MHz, $\mathrm{CD_2Cl_2}$) δ = 11.5 (s, $\mathrm{O}Si\mathrm{Me_2SiMe_2Cl}$), 18.4 (s, $\mathrm{OSiMe_2}Si\mathrm{Me_2Cl}$).

$[\mathrm{BeCl(O(C_2H_4O)(SiMe_2SiMe_2O)(C_2H_4O)SiMe_2SiMe_2Cl)}]$ (10b): ^{1}H NMR (500 MHz, $\mathrm{CD_2Cl_2}$) δ = 0.26 (s, 12H, $\mathrm{O}Si\mathrm{Me_2}Si\mathrm{Me_2O}$), 0.31 (s, 6H, $\mathrm{OSi}Me_2\mathrm{SiMe_2Cl}$), 0.51 (s, 6H, $\mathrm{OSiMe_2Si}Me_2\mathrm{Cl}$), 3.67 (s, 2H, $\mathrm{C}H_2$), 3.82 (s, 6H, $\mathrm{C}H_2$). ^{9}Be NMR (70 MHz, $\mathrm{CD_2Cl_2}$) δ = 2.5 ($\omega_{1/2}$ = 53.2 Hz). ^{13}C NMR (126 MHz, $\mathrm{CD_2Cl_2}$) δ = −1.0 ($\mathrm{OSi}Me_2\mathrm{SiMe_2Cl}$), −0.1 ($\mathrm{O}Si\mathrm{Me_2}Si\mathrm{Me_2O}$), 2.8 ($\mathrm{OSiMe_2Si}Me_2\mathrm{Cl}$), 65.7 ($C\mathrm{H_2}$), 67.0 ($C\mathrm{H_2}$). ^{29}Si NMR (99 MHz, $\mathrm{CD_2Cl_2}$) δ = 11.6 (s, $\mathrm{O}Si\mathrm{Me_2}Si\mathrm{Me_2O}$), 11.8 (s, $\mathrm{O}Si\mathrm{Me_2SiMe_2Cl}$), 18.6 (s, $\mathrm{OSiMe_2}Si\mathrm{Me_2Cl}$).

$[\mathrm{BeCl(O(C_2H_4O)(C_6H_4O)(C_2H_4O)SiMe_2SiMe_2Cl)}]$ (10Ph): ^{1}H NMR (500 MHz, $\mathrm{CD_2Cl_2}$) δ = 0.36 (s, 6H, $\mathrm{OSi}Me_2\mathrm{SiMe_2Cl}$), 0.52 (s, 6H, $\mathrm{OSiMe_2Si}Me_2\mathrm{Cl}$), 3.96 – 4.00 (m, 4H, $\mathrm{C}H_2$), 4.05 – 4.08 (m, 4H, $\mathrm{C}H_2$), 6.86 – 6.98 (m, 2H, H_{Ar}), 7.18 – 7.26 (m, 2H, H_{Ar}). ^{9}Be NMR (42 MHz, $\mathrm{CD_2Cl_2}$) δ = 3.1 ($\omega_{1/2}$ = 59.1 Hz). ^{13}C NMR (126 MHz, $\mathrm{CD_2Cl_2}$) δ = −1.0 ($\mathrm{OSi}Me_2\mathrm{SiMe_2Cl}$), 2.7 ($\mathrm{OSiMe_2Si}Me_2\mathrm{Cl}$), 63.2 ($C\mathrm{H_2}$), 70.7 ($C\mathrm{H_2}$), 114.6 ($C_{\mathrm{Ar}}\mathrm{H}$), 121.8 ($C_{\mathrm{Ar}}\mathrm{H}$), 149.3 ($C_{\mathrm{Ar}}$). ^{29}Si NMR (99 MHz, $\mathrm{CD_2Cl_2}$) δ = 12.2 (s, $\mathrm{O}Si\mathrm{Me_2SiMe_2Cl}$), 18.3 (s, $\mathrm{OSiMe_2}Si\mathrm{Me_2Cl}$).

$[\mathrm{BeCl_2}$(1,2-disila[12]crown-4)] (11a): ^{1}H NMR (500 MHz, $\mathrm{CD_2Cl_2}$) δ = 0.24 (s, 12H, $\mathrm{Si}Me_2$), 3.60 (s, 4H, $\mathrm{C}H_2$), 3.69 – 3.72 (m, 4H, $\mathrm{C}H_2$). ^{13}C NMR (126 MHz, $\mathrm{CD_2Cl_2}$) δ −0.1 ($\mathrm{Si}Me_2$), 0.0 ($\mathrm{Si}Me_2$), 63.6 ($C\mathrm{H_2}$), 71.2 ($C\mathrm{H_2}$), 73.1 ($C\mathrm{H_2}$). ^{29}Si NMR (99 MHz, $\mathrm{CD_2Cl_2}$) δ = 11.4 ($Si\mathrm{Me_2}$). FT-IR (cm^{-1}): 2951 (m), 2884 (m), 1458 (w), 1395 (w), 1350 (w), 1293 (w), 1248 (m, Si-Me), 1136 (m, Si-O/C-O), 1083 (s, Si-O/C-O), 1046 (s, Si-O/C-O), 930 (s), 822 (m), 789 (s, Si-Me), 767 (s, Si-Me), 728 (s), 687 (w), 671 (w), 628 (s), 522 (m), 484 (s, Be-Cl), 404 (m).

$[\mathrm{BeCl_2}$(1,2,4,5-tetrasila[12]crown-4)] (11c): ^{1}H NMR (500 MHz, $\mathrm{CD_2Cl_2}$) δ = 0.19 (s, 6H, $\mathrm{Si}Me_2$), 0.20 (s, 6H, $\mathrm{Si}Me_2$), 3.58 – 3.64 (m, 4H, $\mathrm{C}H_2$), 3.74 – 3.77 (m, 4H, $\mathrm{C}H_2$). ^{13}C NMR (126 MHz, $\mathrm{CD_2Cl_2}$) δ = 0.41 ($\mathrm{Si}Me_2\mathrm{OSi}Me_2$), 2.5 ($\mathrm{Si}Me_2\mathrm{OCH_2}$), 63.2 ($C\mathrm{H_2OSiMe_2}$), 73.1 ($C\mathrm{H_2O}C\mathrm{H_2}$). ^{29}Si NMR (99 MHz, $\mathrm{CD_2Cl_2}$) δ = 3.8 (s, $Si\mathrm{O}Si$), 10.7 ($Si\mathrm{OC}$). FT-IR (cm^{-1}): 2949 (m), 2882 (w), 1593 (w), 1501 (m), 1454 (m), 1399 (w), 1248 (s, Si-Me), 1218 (m), 1126 (m, Si-O/C-O), 1091 (s, Si-O/C-O), 1042 (s, Si-O/C-O), 926 (s), 822 (m), 791 (m), 763 (s, Si-Me), 742 (s, Si-Me), 683 (m), 634 (s), 598 (m), 481 (s, Be-Cl), 406 (m).

$[\mathrm{BeCl_2}$(1,2-disila-benzo[12]crown-4)] (11Ph): FT-IR (cm^{-1}): 2945 (m), 2880 (m), 1593 (w), 1501 (m), 1452 (m), 1399 (w), 1356 (w), 1328 (w), 1289 (w), 1250 (s, Si-Me), 1216 (m), 1126 (m, Si-O/C-O), 1089 (s, Si-O/C-O), 1046 (s, Si-O/C-O), 938 (s), 830 (s), 787 (s), 767 (s, Si-Me), 738 (s, Si-Me), 632 (m), 528 (m, Be-Cl), 483 (m).

Crystallographic details

Table S1: Crystallographic details of compounds **4** & **5**

	4 · CH_2Cl_2	**5** · 2 C_6H_6
empirical formula	$C_{25}H_{50}Be_6Cl_6O_{16}$	$C_{48}H_{52}Be_6Cl_4O_{14}$
color and appearance	colourless block	colourless block
molecular mass / g mol^{-1}	873.41	1048.75
cryst. syst.	monoclinic	triclinic
space group type (No.)	$C2/c$ (15)	$P\bar{1}$ (2)
a / Å	19.481(4)	9.8872(5)
b / Å	9.549(2)	12.0013(6)
c / Å	21.762(4)	12.2122(6)
α / °	90	108.235(2)
β / °	106.72(3)	103.883(2)
γ / °	90	106.623(2)
volume / Å^3	3877.2(15)	1229.35(11)
Z	4	1
λ / Å	0.71073 (Mo-K$_\alpha$)	0.71073 (Mo-K$_\alpha$)
T / K	100(2)	100(1)
μ / mm^{-1}	0.509	0.307
Θ_{max}	26.371	24.91
hkl_{max}	$-24 \le h \le 24$	$-11 \le h \le 11$
	$-11 \le k \le 11$	$-14 \le k \le 14$
	$-19 \le l \le 27$	$-14 \le l \le 14$
crystal size / mm^3	0.11 · 0.10 · 0.09	0.26 · 0.16 · 0.09
R_{int}, R_σ	0.081, 0.057	0.022, 0.039
$R(F)$ ($I \ge 2\sigma(I)$, all data)	0.053, 0.064	0.040, 0.049
$wR(F^2)$ ($I \ge 2\sigma(I)$, all data)	0.140, 0.148	0.096, 0.100
S (all data)	1.069	1.046
data, parameter, restraints	3895, 226, 0	4297, 325, 0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ / e Å^{-3}	0.693 / −0.488	0.552 / −0.310

NMR spectra

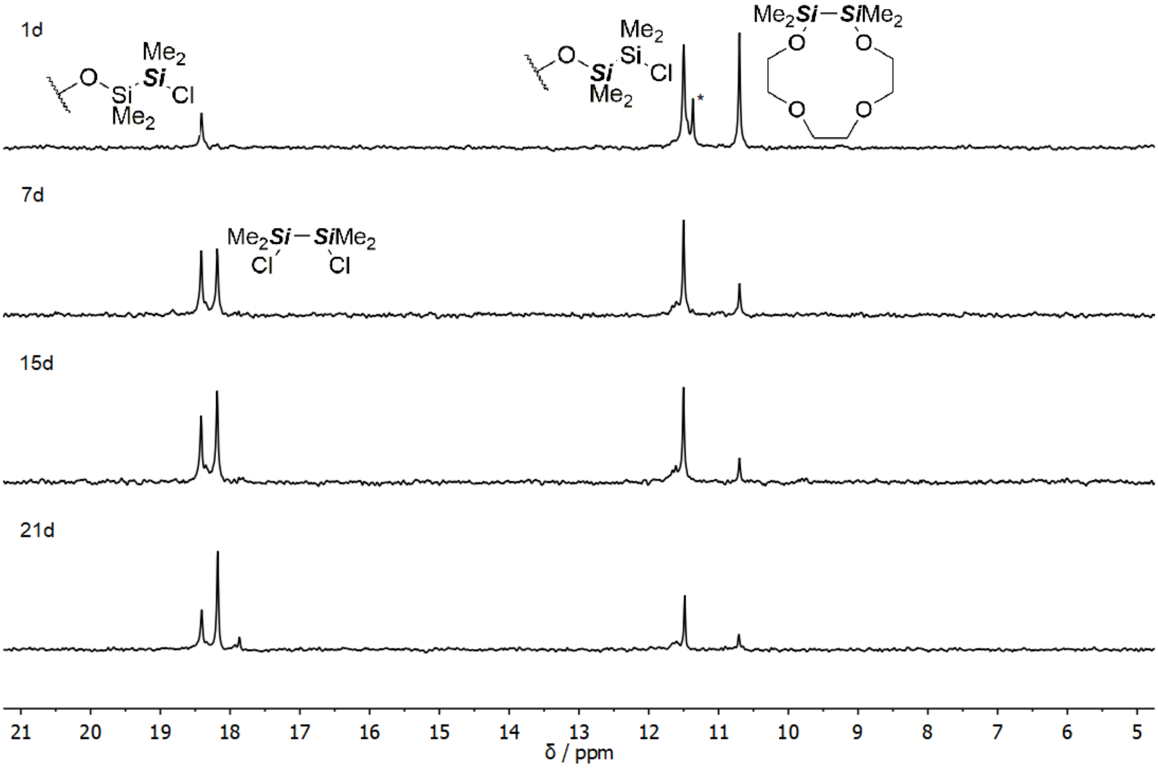

Figure S1: ^{29}Si NMR spectra of the reaction of BeCl$_2$ with ligand **2a** after different reaction times in CD$_2$Cl$_2$. The signal marked with an asterisk presumably originates from compound **11a**.

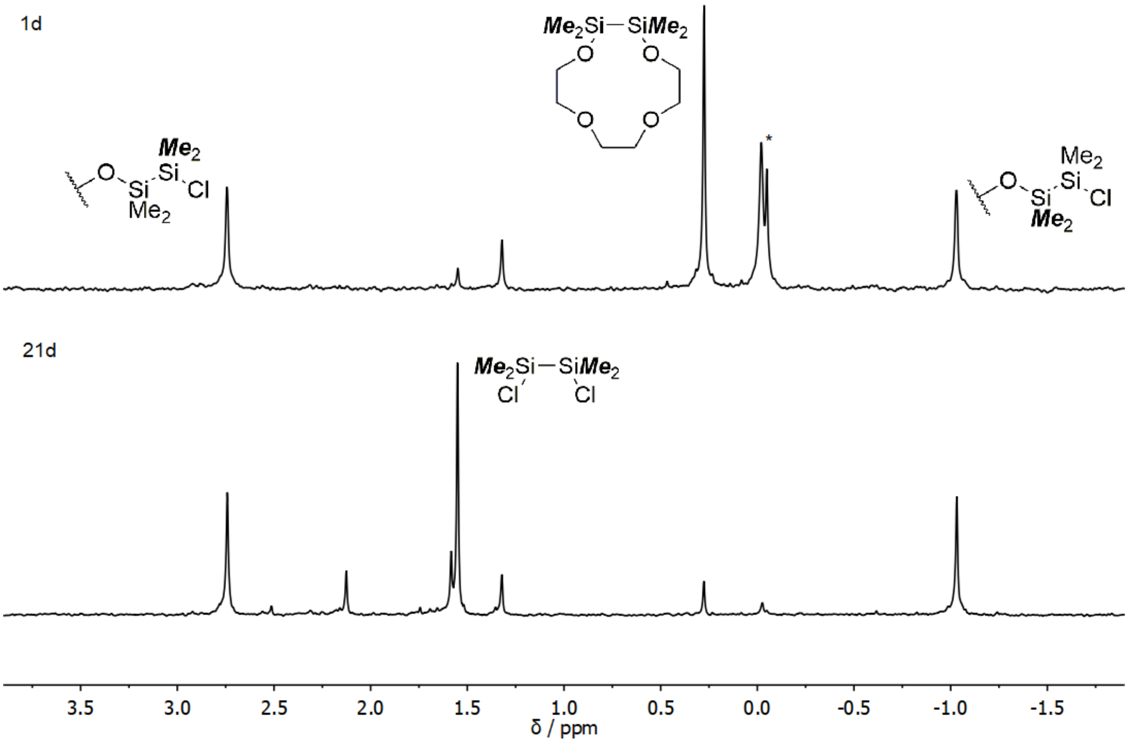

Figure S2: ^{13}C NMR spectra of the reaction of BeCl$_2$ with ligand **2a** after different reaction times in CD$_2$Cl$_2$. The signal marked with an asterisk presumably originates from compound **11a**.

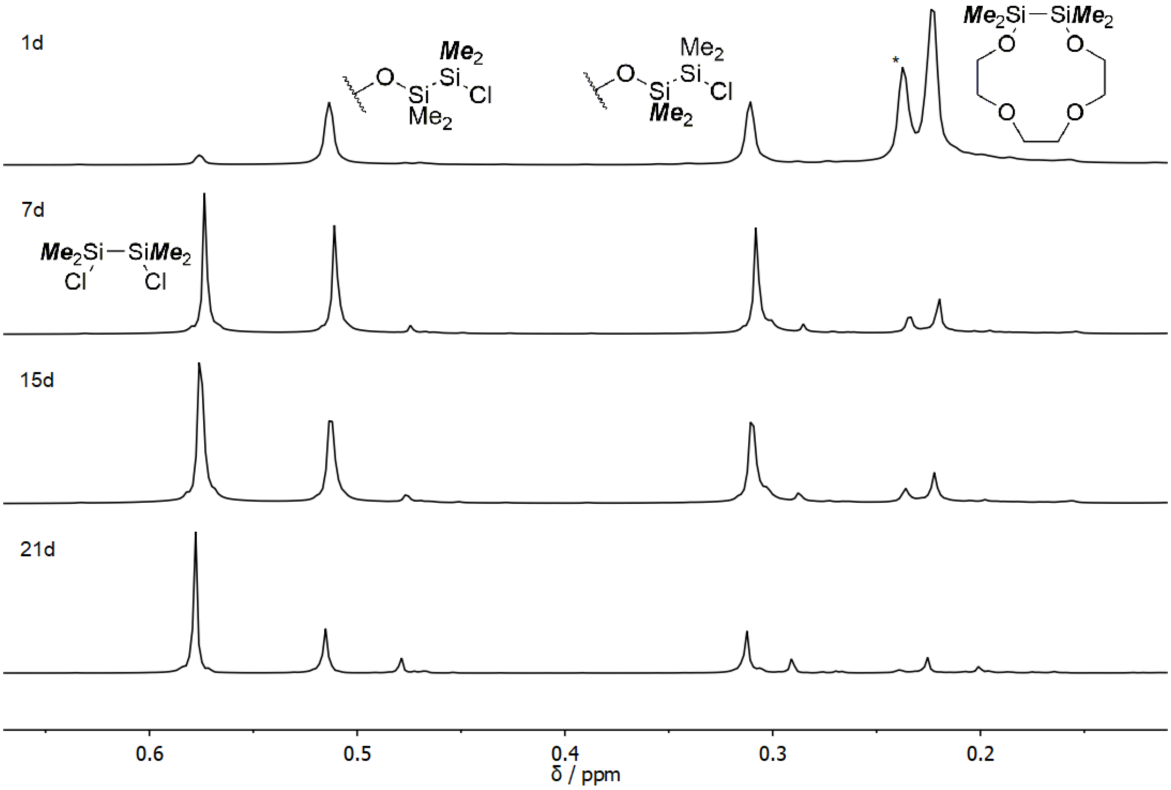

Figure S3: ^{1}H NMR spectra of the reaction of $BeCl_2$ with ligand **2a** after different reaction times in CD_2Cl_2. The signal marked with an asterisk presumably originates from compound **11a**.

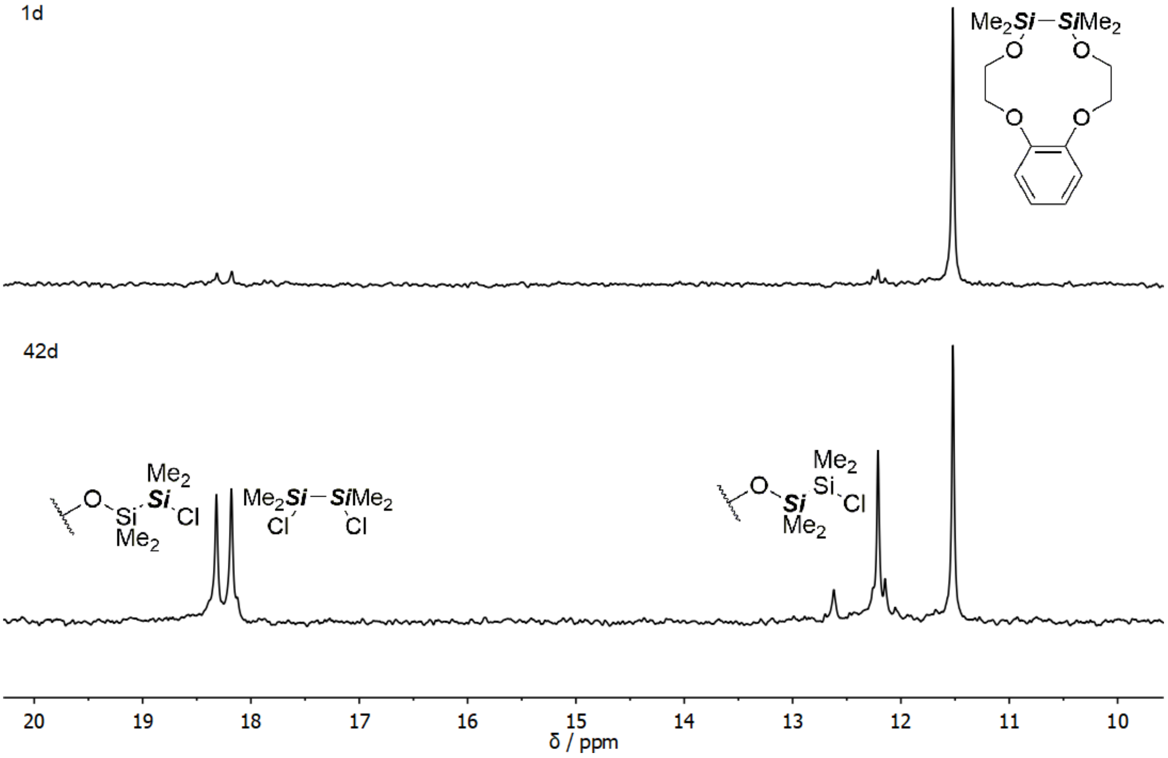

Figure S4: ^{29}Si NMR spectra of the reaction of $BeCl_2$ with ligand **3** after different reaction times in CD_2Cl_2.

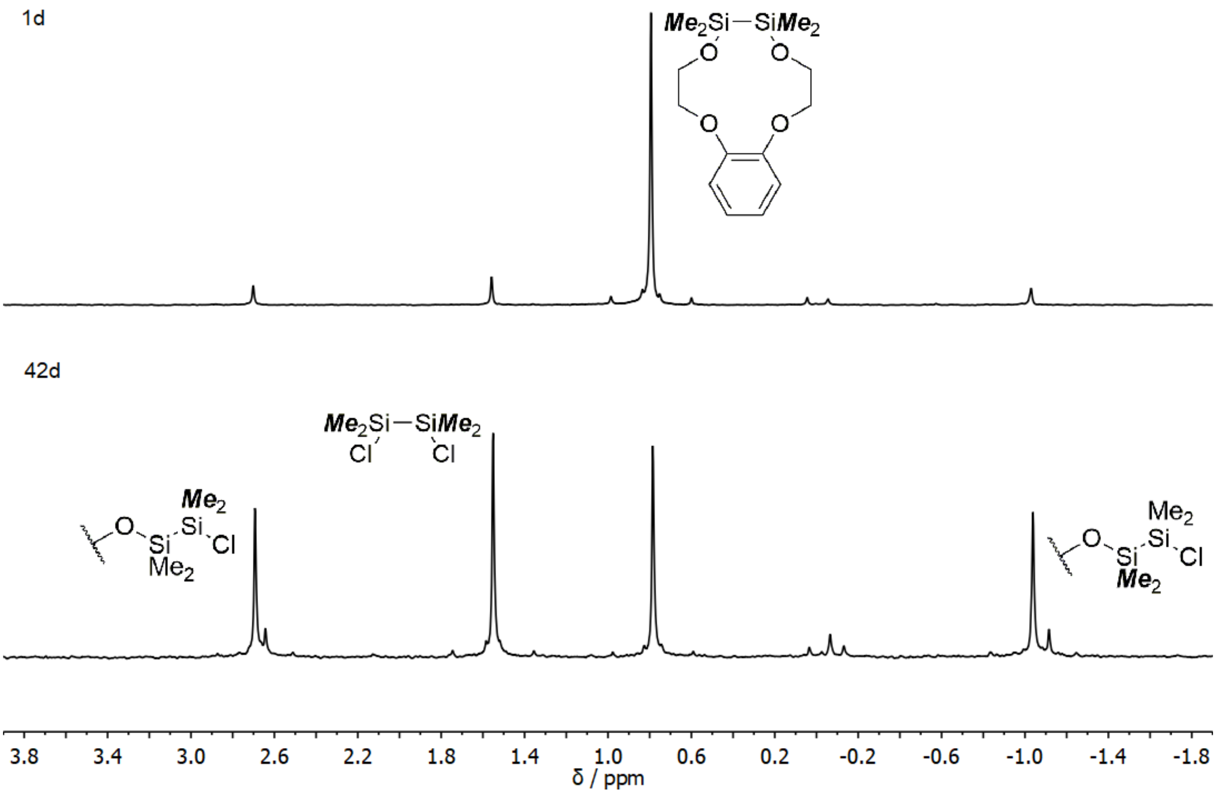

Figure S5: ^{13}C NMR spectra of the reaction of $BeCl_2$ with ligand **3** after different reaction times in CD_2Cl_2.

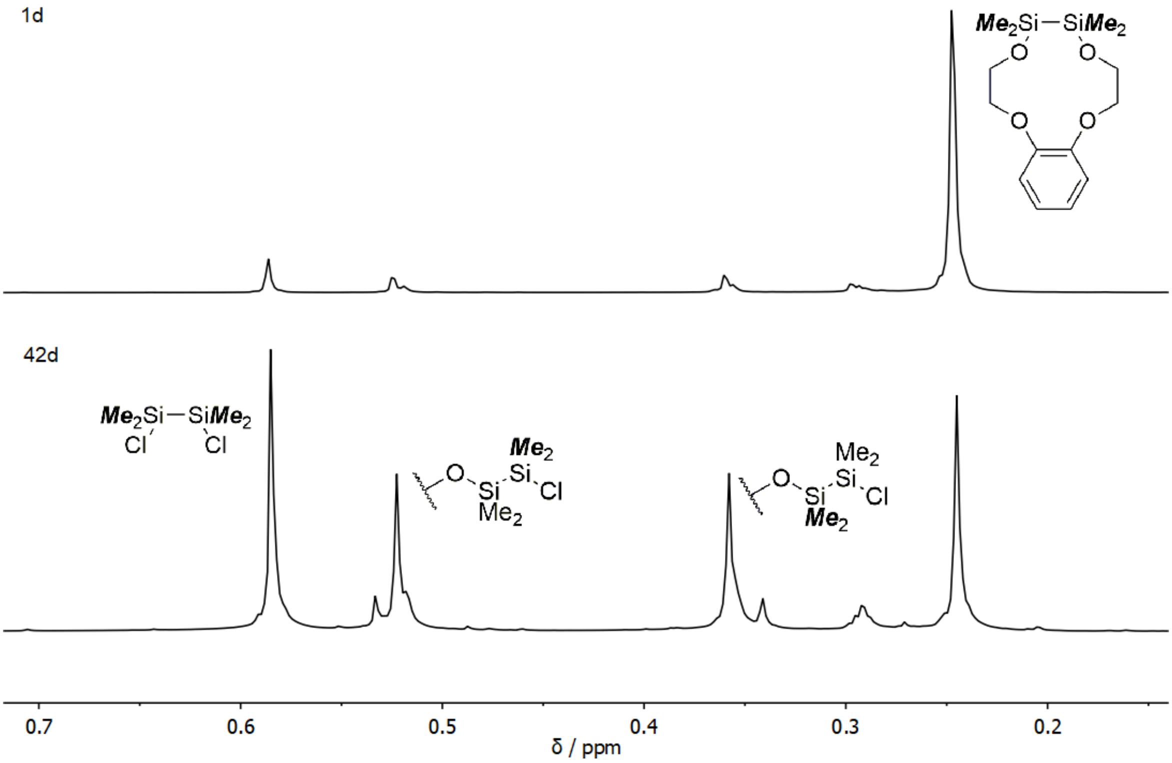

Figure S6: ^{1}H NMR spectra of the reaction of $BeCl_2$ with ligand **3** after different reaction times in CD_2Cl_2.

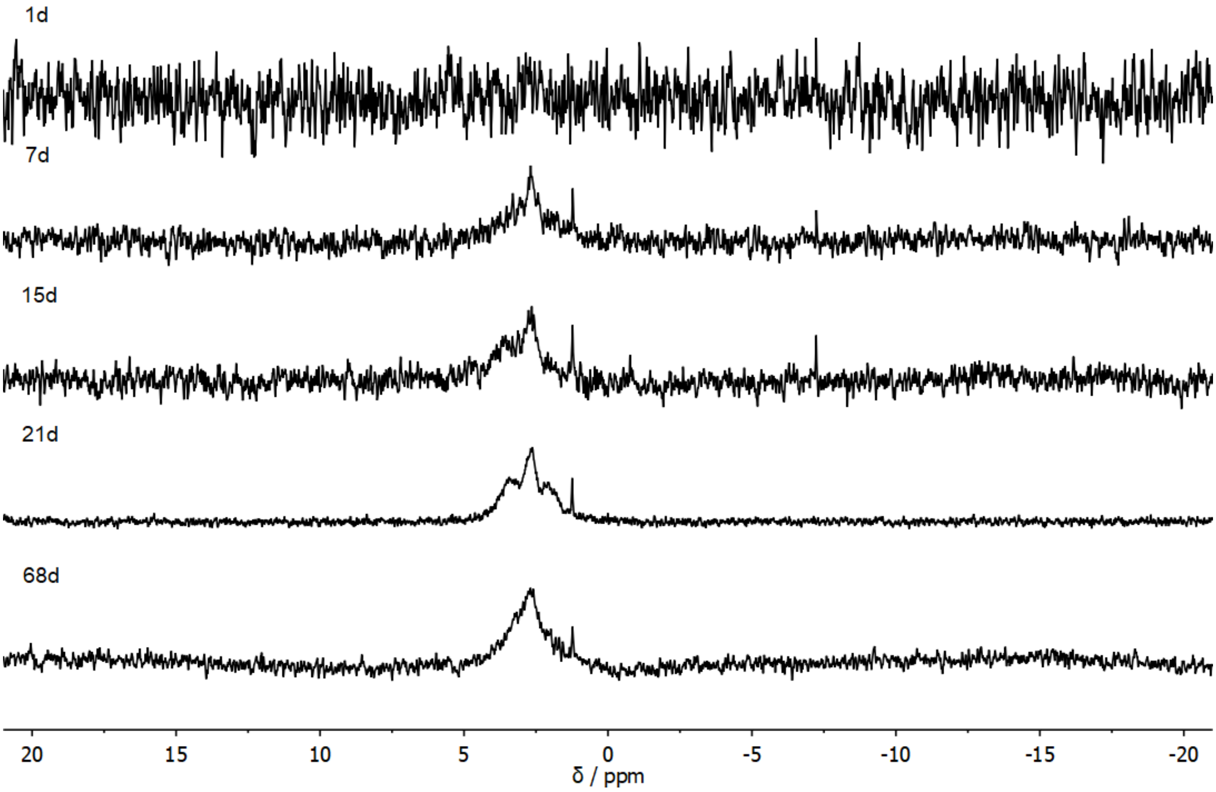

Figure S7: ^{9}Be NMR spectra of the reaction of $BeCl_2$ with ligand **2a** after different reaction times in CD_2Cl_2.

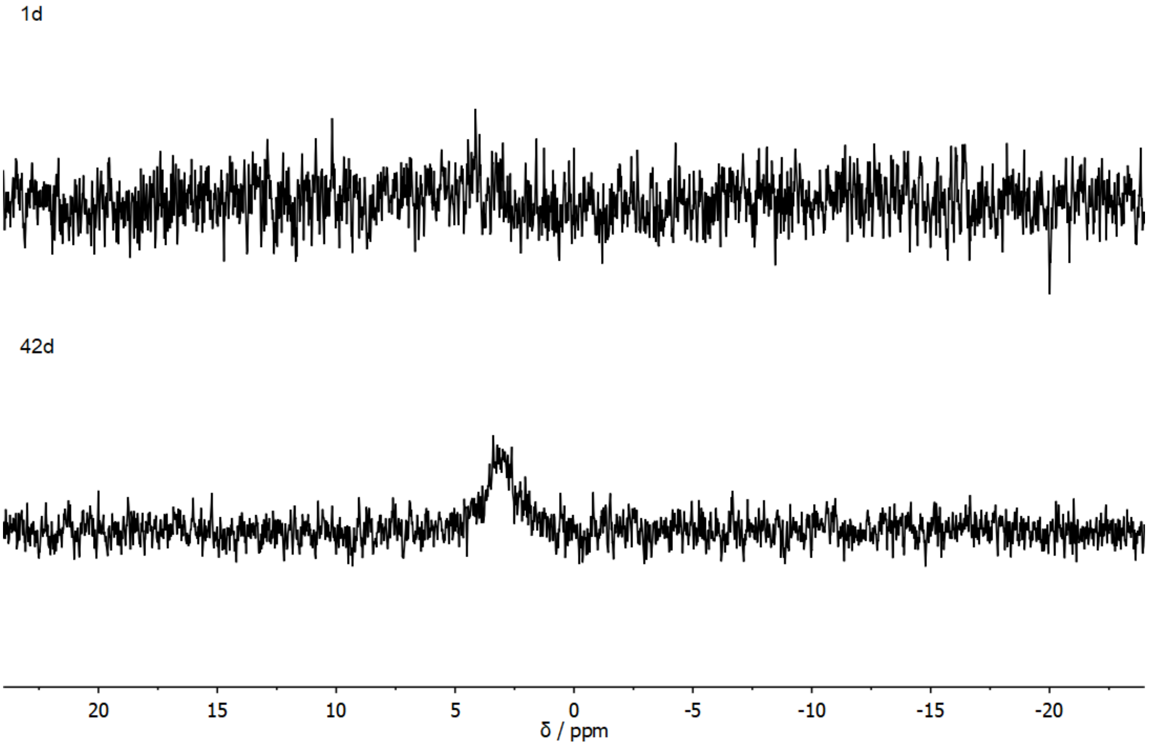

Figure S8: ^{9}Be NMR spectra of the reaction of $BeCl_2$ with ligand **3** after different reaction times in CD_2Cl_2.

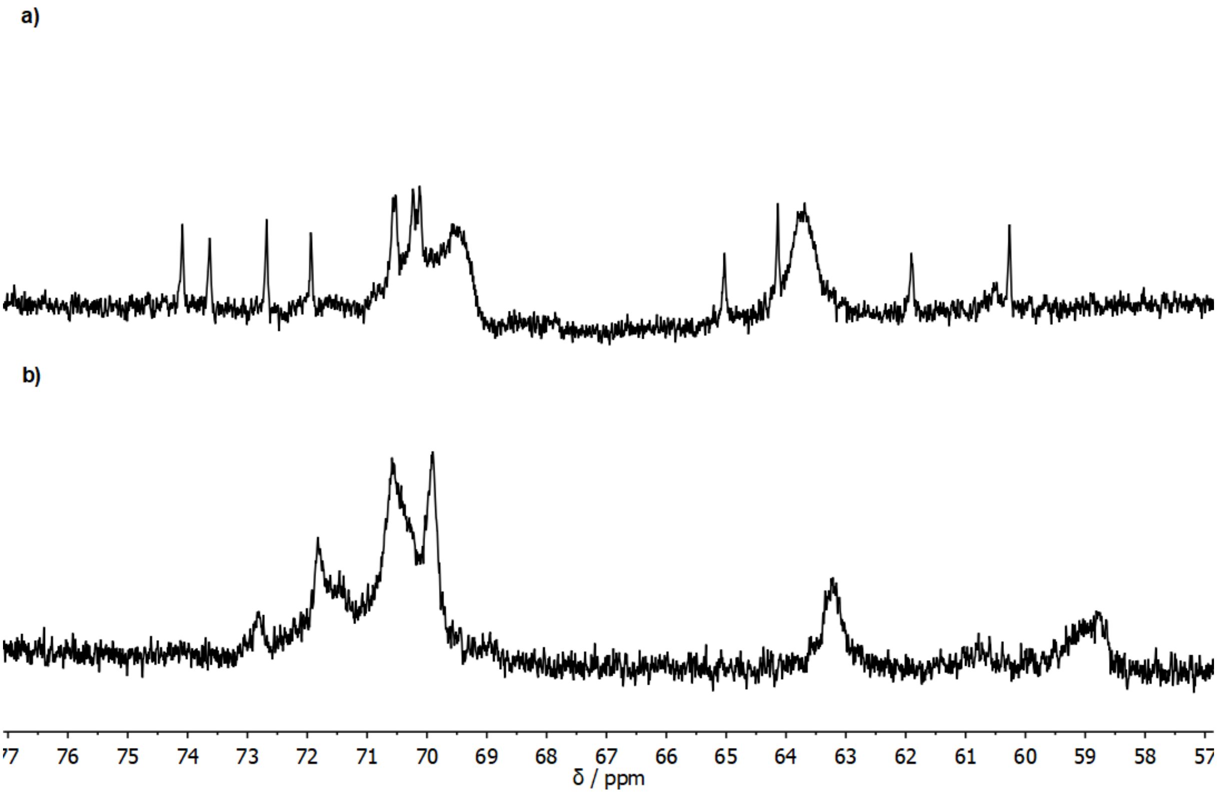

Figure S9: ^{13}C NMR spectra of the reaction of $BeCl_2$ with a) ligand **6** and b) ligand **7** in CD_2Cl_2.

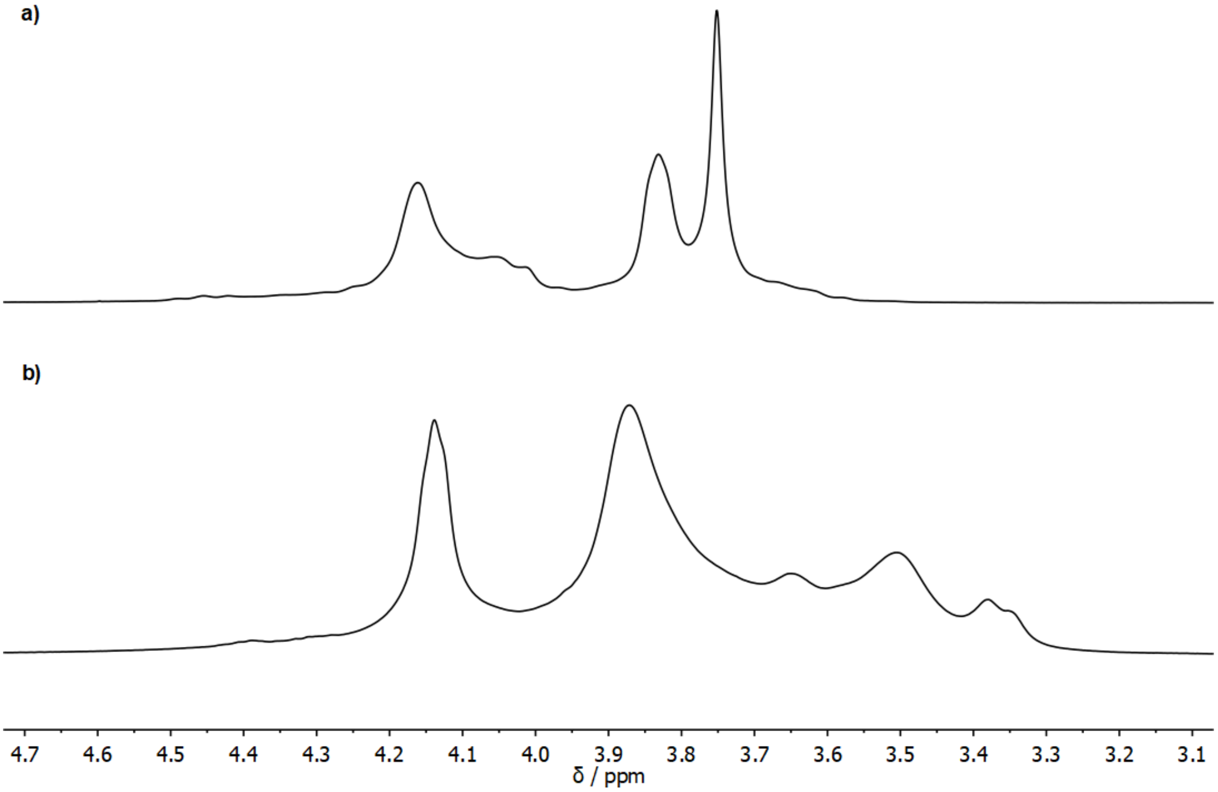

Figure S10: ^{1}H NMR spectra of the reaction of $BeCl_2$ with a) ligand **6** and b) ligand **7** in CD_2Cl_2.

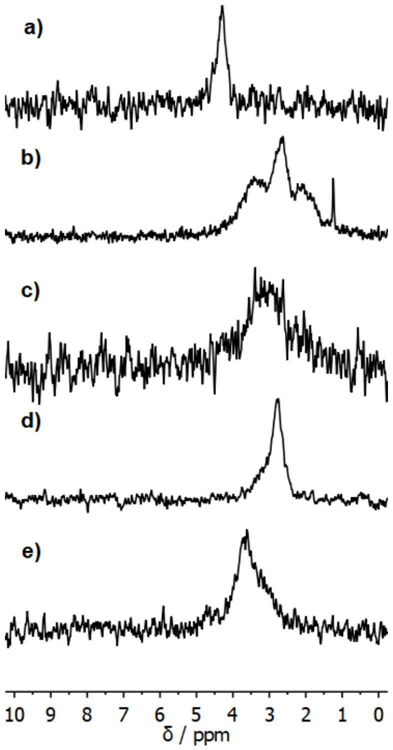

Figure S11: ^{9}Be NMR spectra of $BeCl_2$ with [12]crown-4 (a), ligands **2a** (b), **3** (c), **6** (d) or **7** (e) in CD_2Cl_2 respectively.

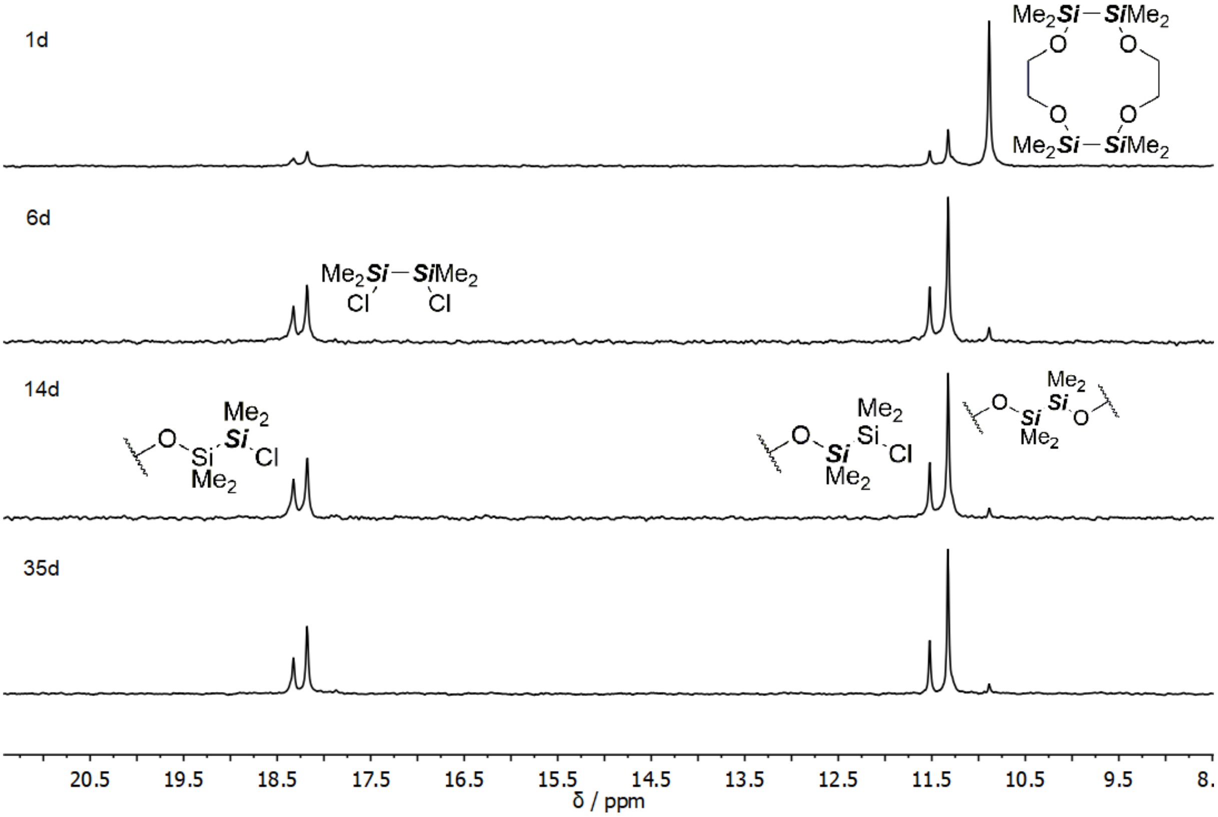

Figure S12: ^{29}Si NMR spectra of the reaction of $BeCl_2$ with ligand **2b** after different reaction times in CD_2Cl_2.

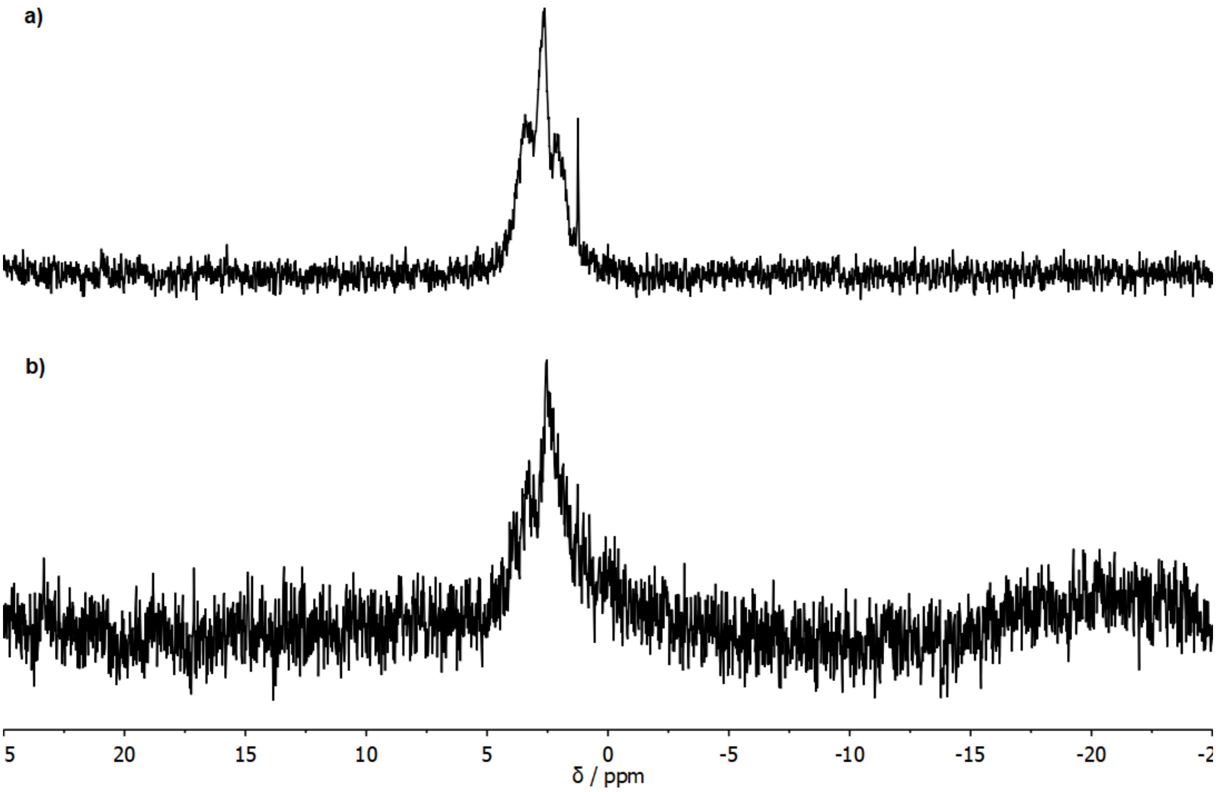

Figure S13: ^{9}Be NMR spectra of the reaction of $BeCl_2$ with a) ligand **2a** and b) ligand **2b** in CD_2Cl_2.

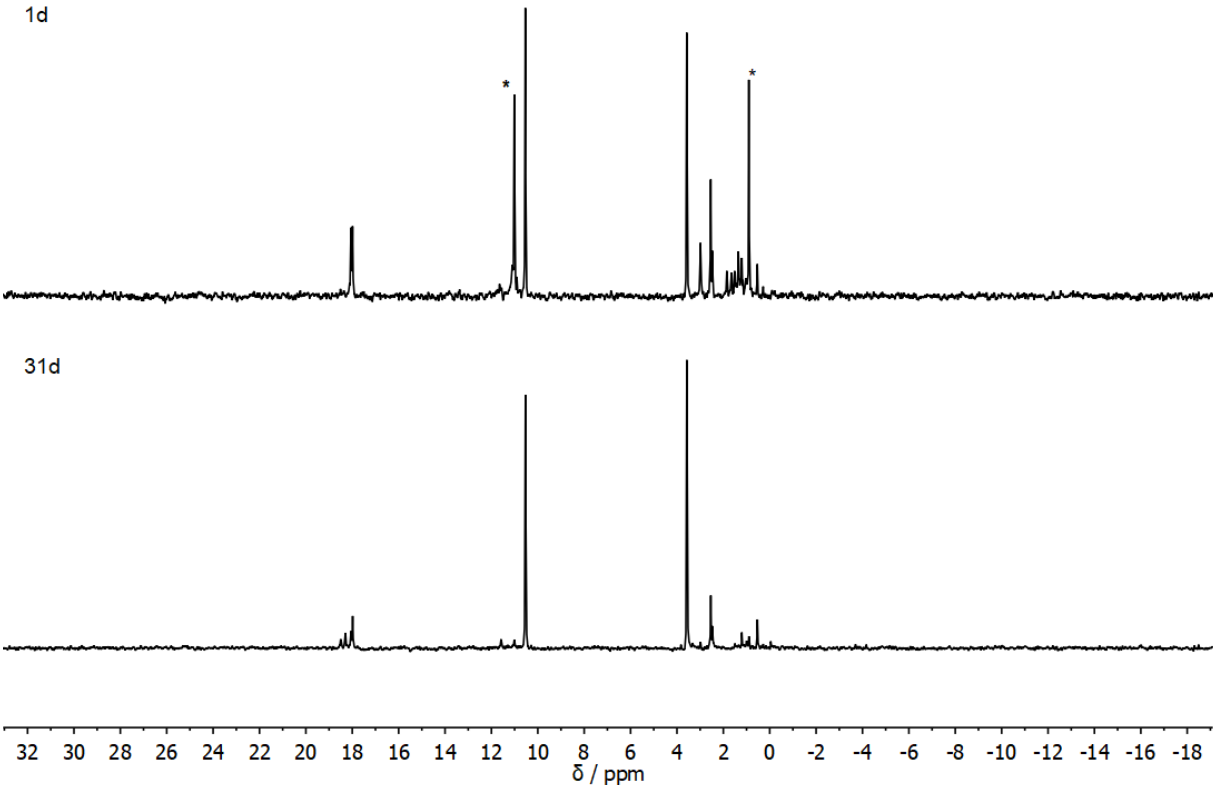

Figure S14: ^{29}Si NMR spectra of the reaction of $BeCl_2$ with ligand **2c** after different reaction times in CD_2Cl_2. Free ligand is marked with an asterisk.

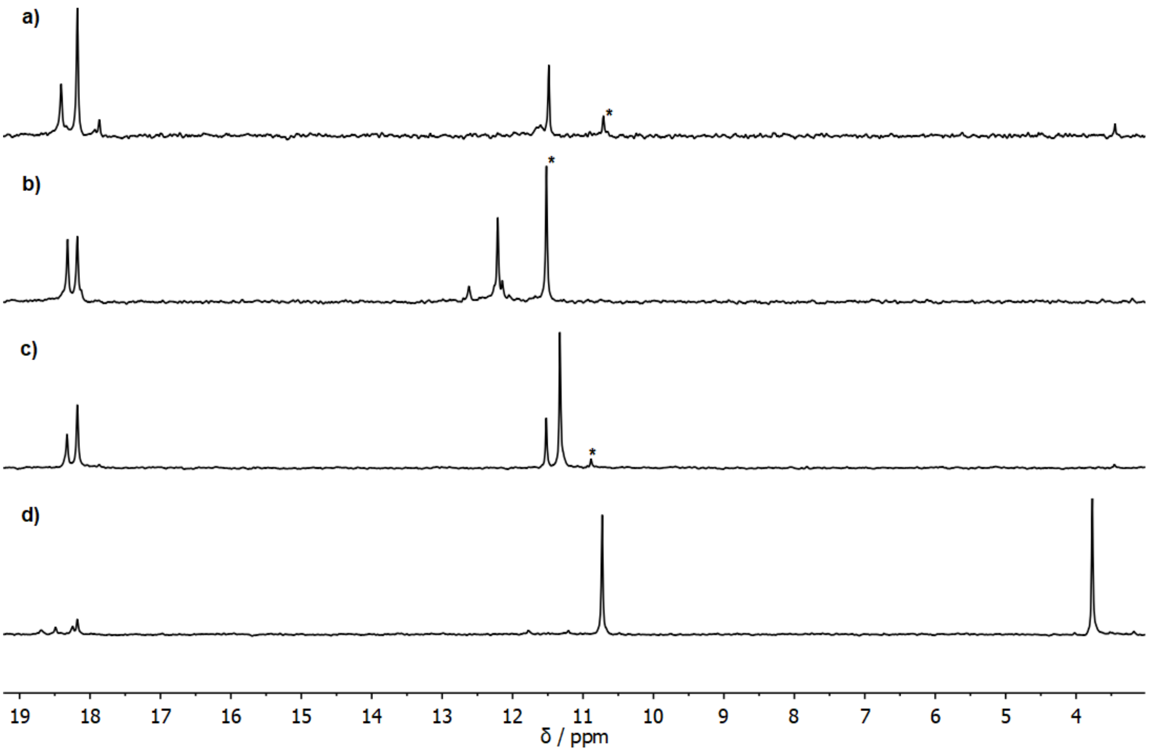

Figure S15: ^{29}Si NMR spectra of the reaction of $BeCl_2$ with ligands a) **2a**, b) **3**, c) **2b** and d) **2c** in CD_2Cl_2. Free ligand is marked with an asterisk.

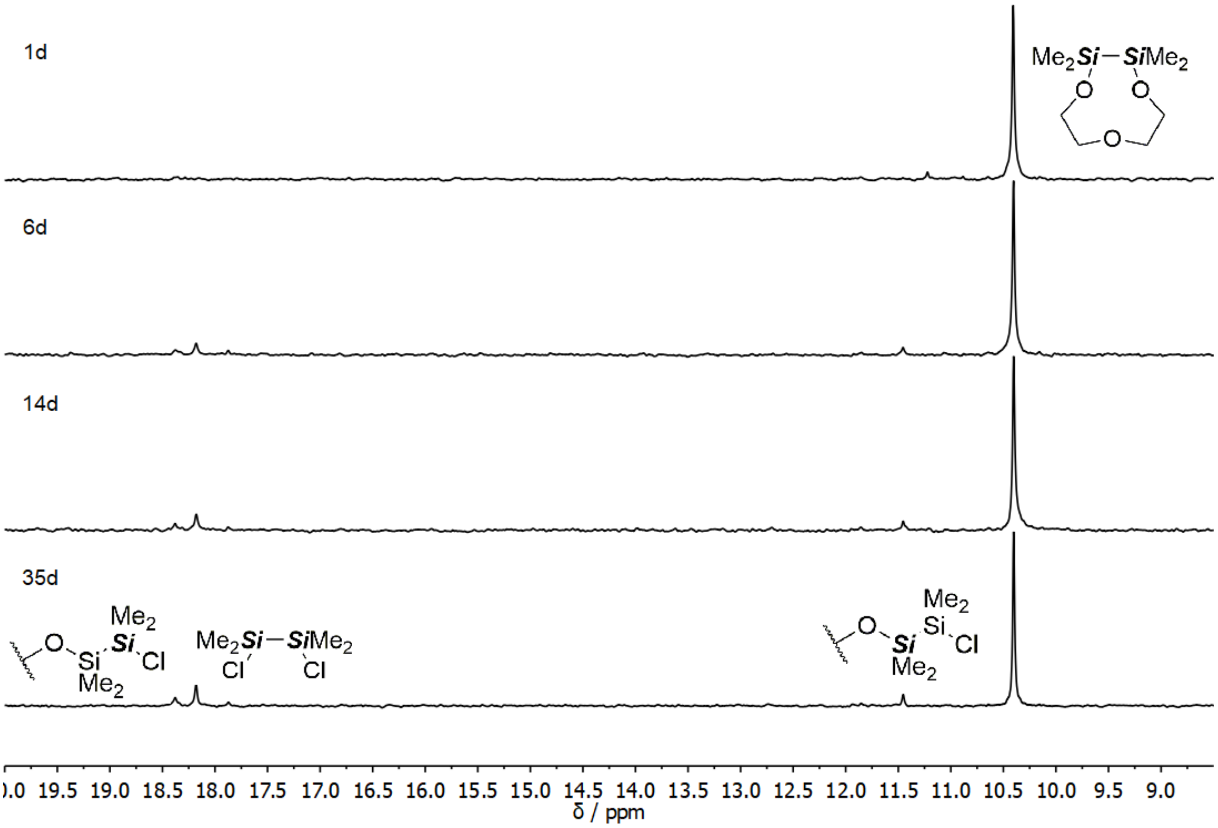

Figure S16: ^{29}Si NMR spectra of the reaction of $BeCl_2$ with ligand **15** after different reaction times in CD_2Cl_2.

IR spectra

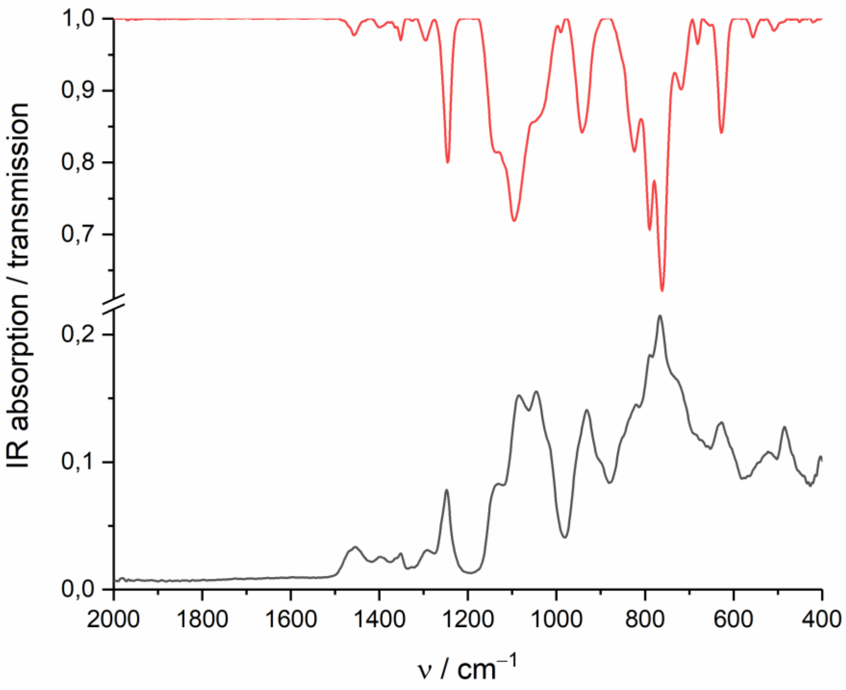

Figure S17: Detail of the FT-IR spectra of 1,2-disila[12]crown-4 (**2a**) before (red) and after reaction with $BeCl_2$ (black).

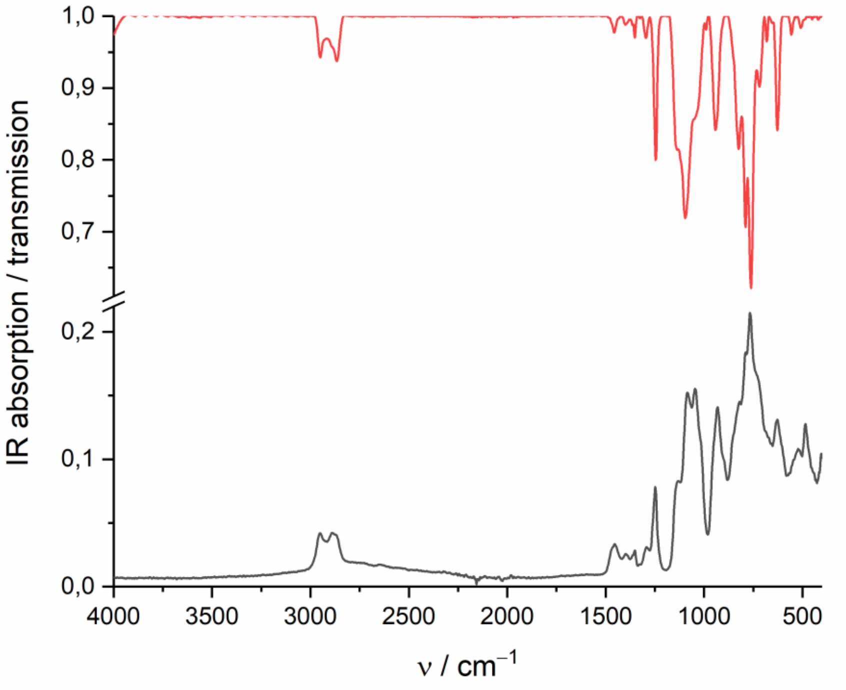

Figure S18: FT-IR spectra of 1,2-disila[12]crown-4 (**2a**) before (red) and after reaction with $BeCl_2$ (black).

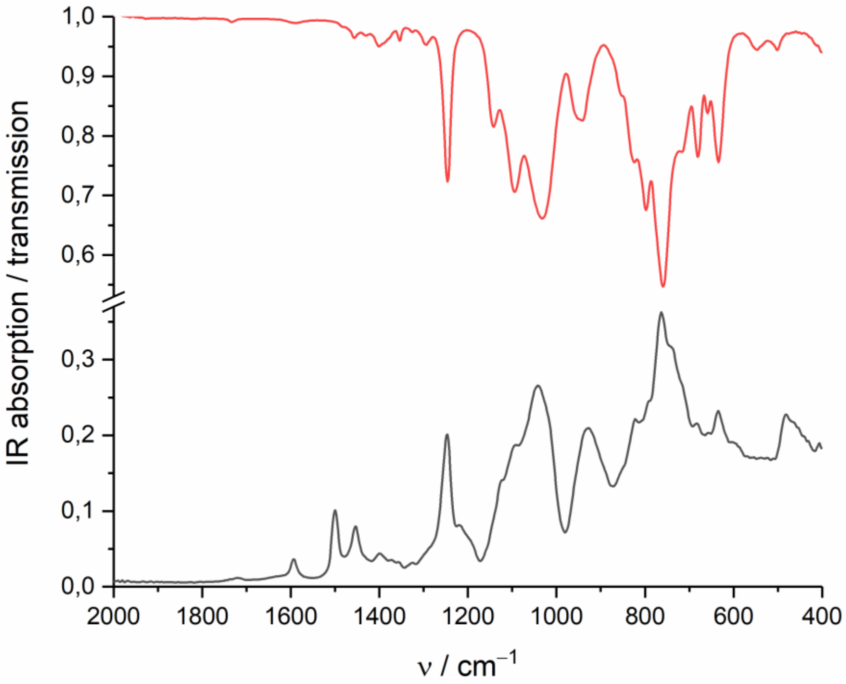

Figure S19: Detail of the FT-IR spectra of 1,2,4,5-tetrasila[12]crown-4 (**2c**) before (red) and after reaction with $BeCl_2$ (black).

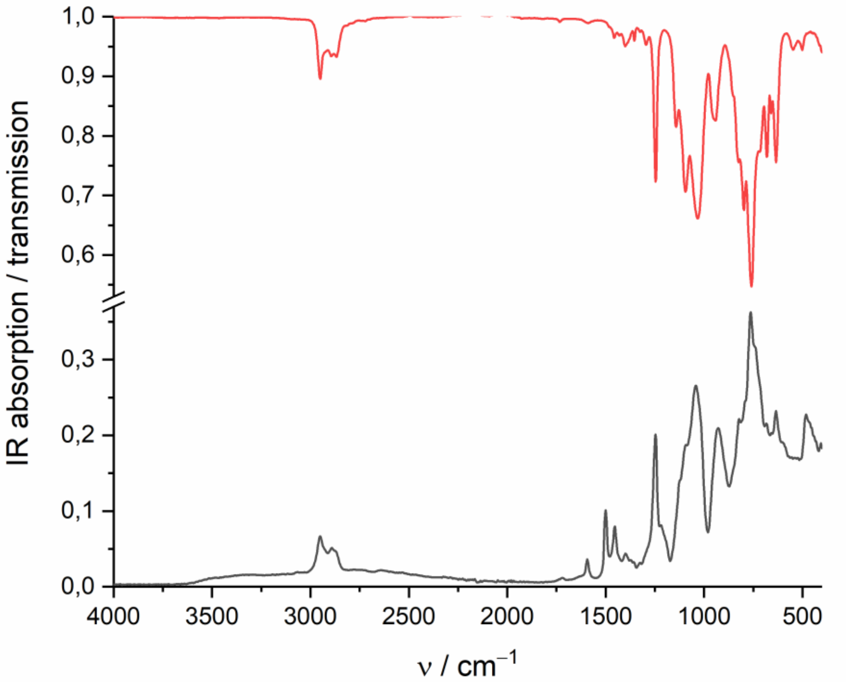

Figure S20: FT-IR spectra of 1,2,4,5-tetrasila[12]crown-4 (**2c**) before (red) and after reaction with $BeCl_2$ (black).

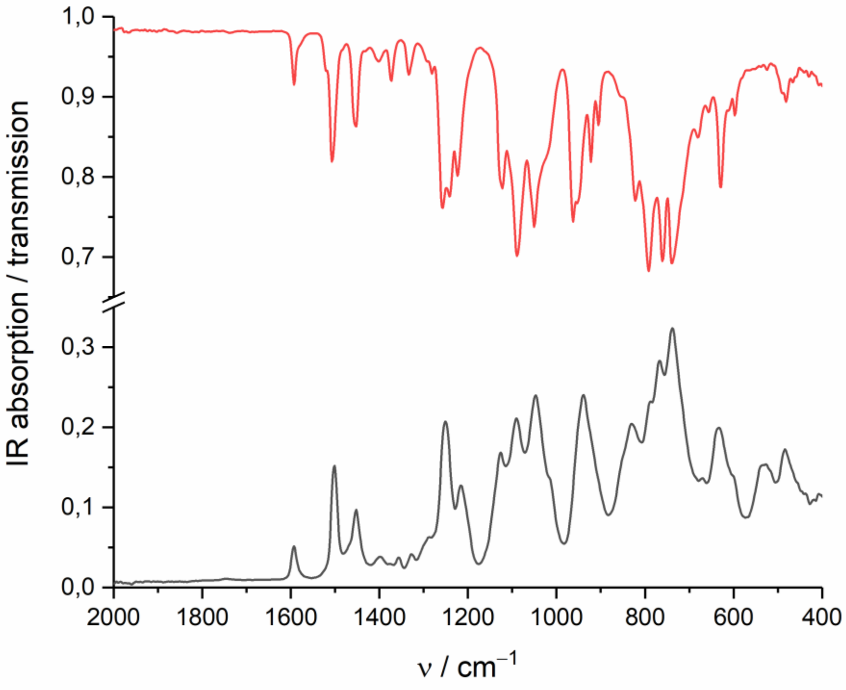

Figure S21: Detail of the FT-IR spectra of 1,2-disila-benzo[12]crown-4 (**3**) before (red) and after reaction with $BeCl_2$ (black).

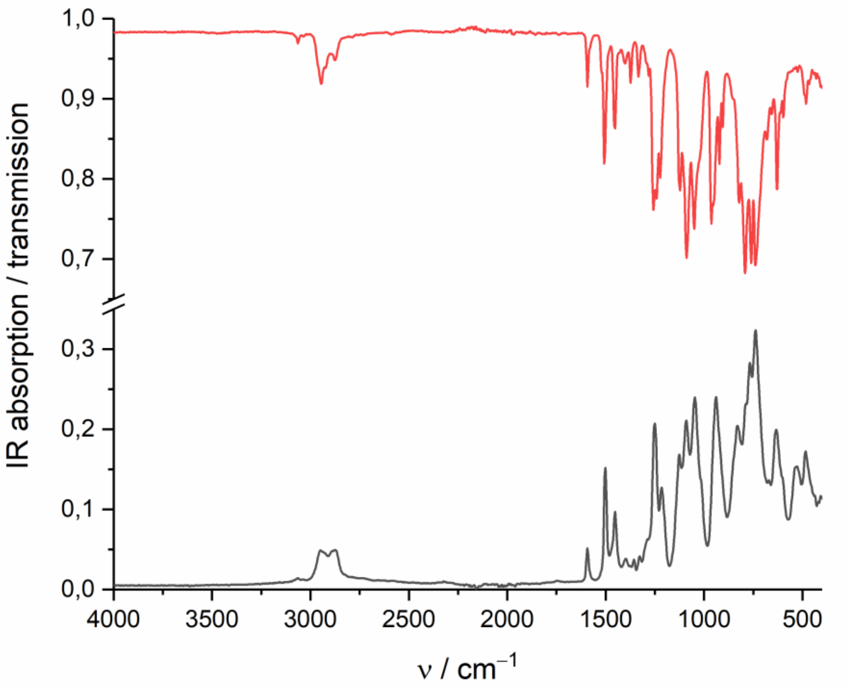

Figure S22: FT-IR spectra of 1,2-disila-benzo[12]crown-4 (**3**) before (red) and after reaction with $BeCl_2$ (black).

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