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Electronic Supporting Information (ESI)

Hydroxyalkyl-substituted double-decker silsesquioxanes: effective separation of *cis* and *trans* isomers

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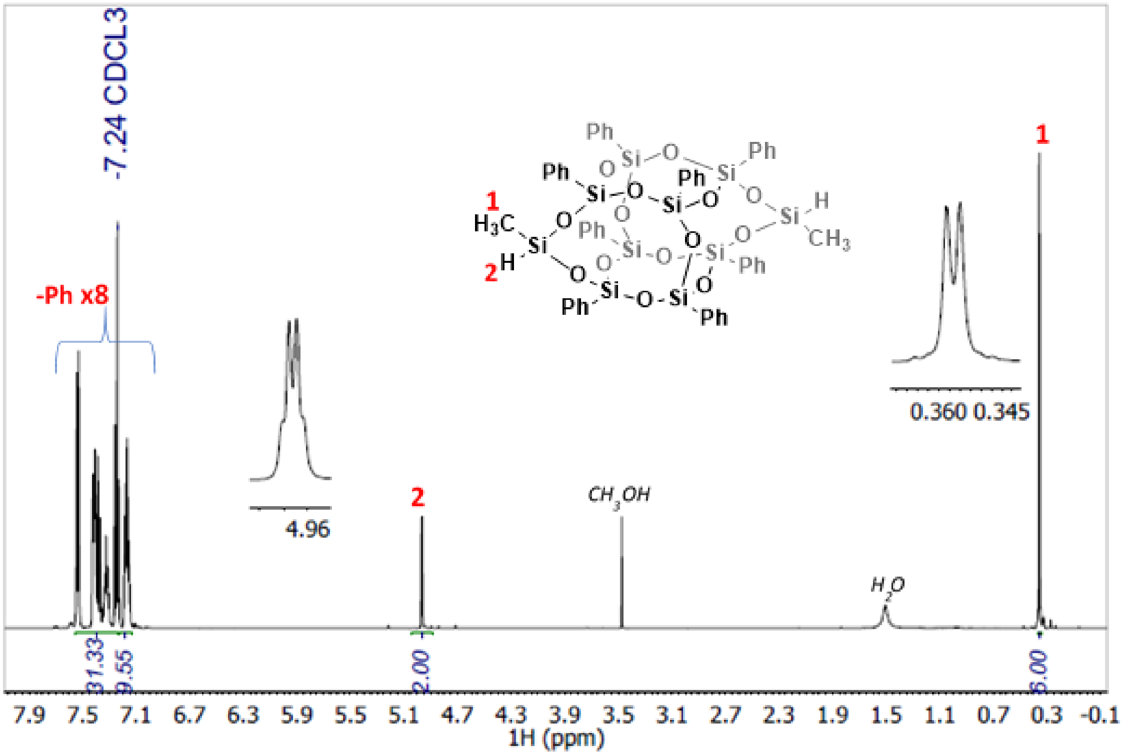
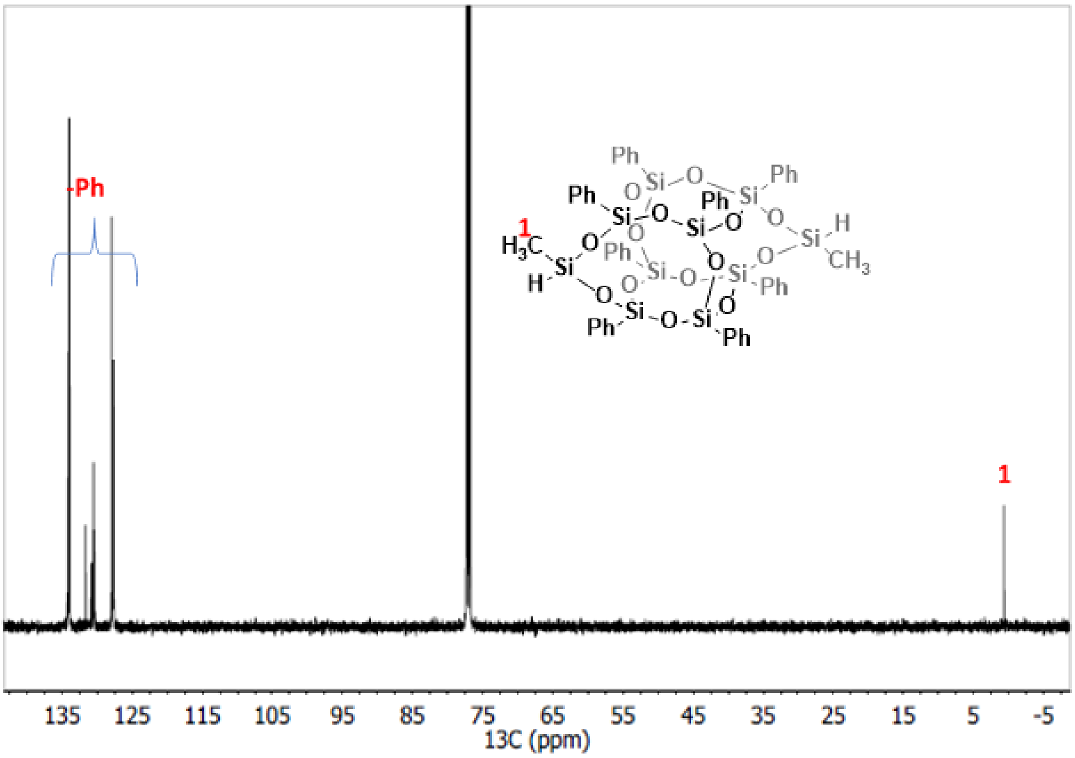


Figure S1. ^{1}H NMR (500 MHz, $CDCl_3$, 300 K) spectrum of **1**.



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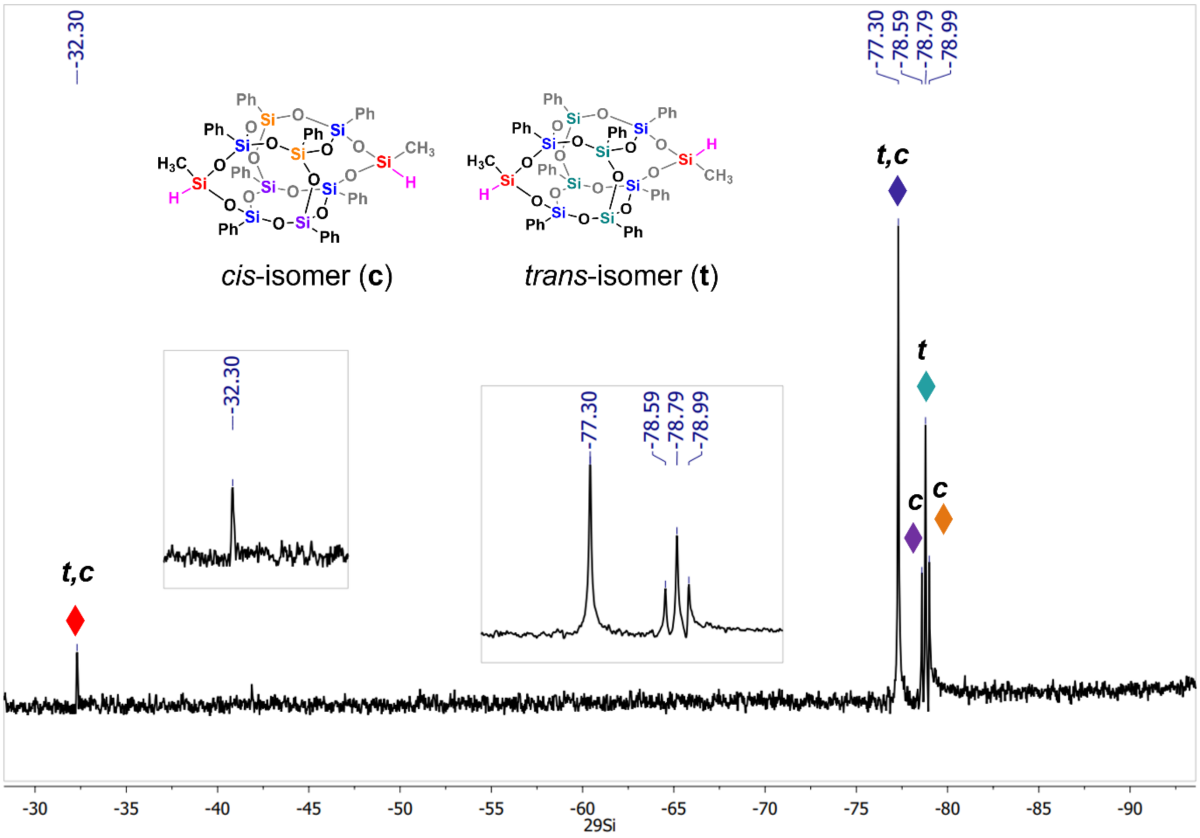


Figure S3. ^{29}Si NMR (119 MHz, $CDCl_3$, 300 K) spectrum of **1**.

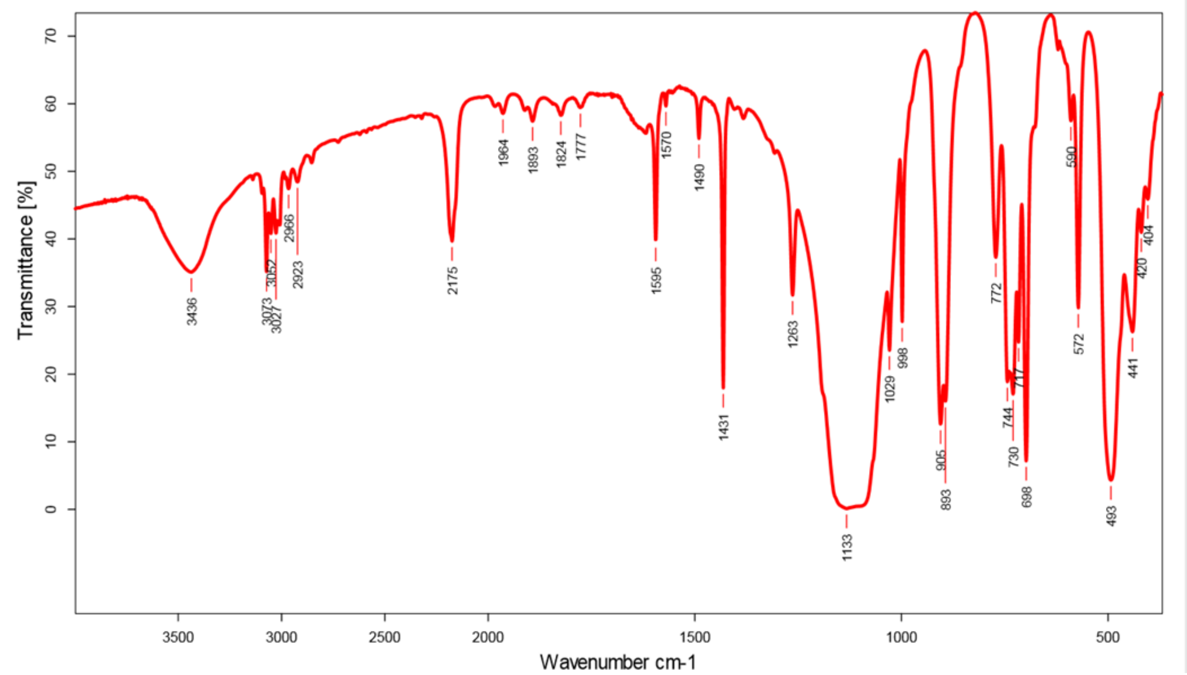
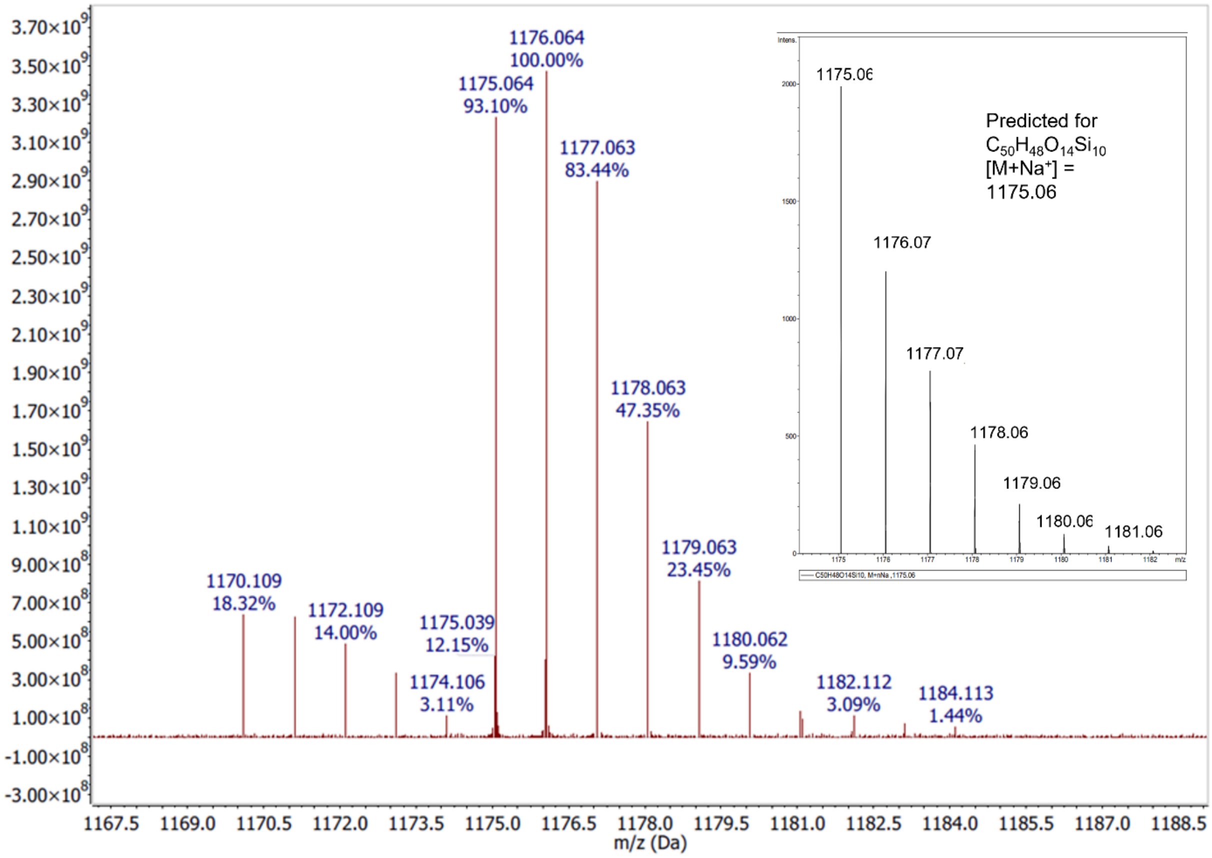


Figure S4. FT-IR spectrum (KBr pellet) of **1**.



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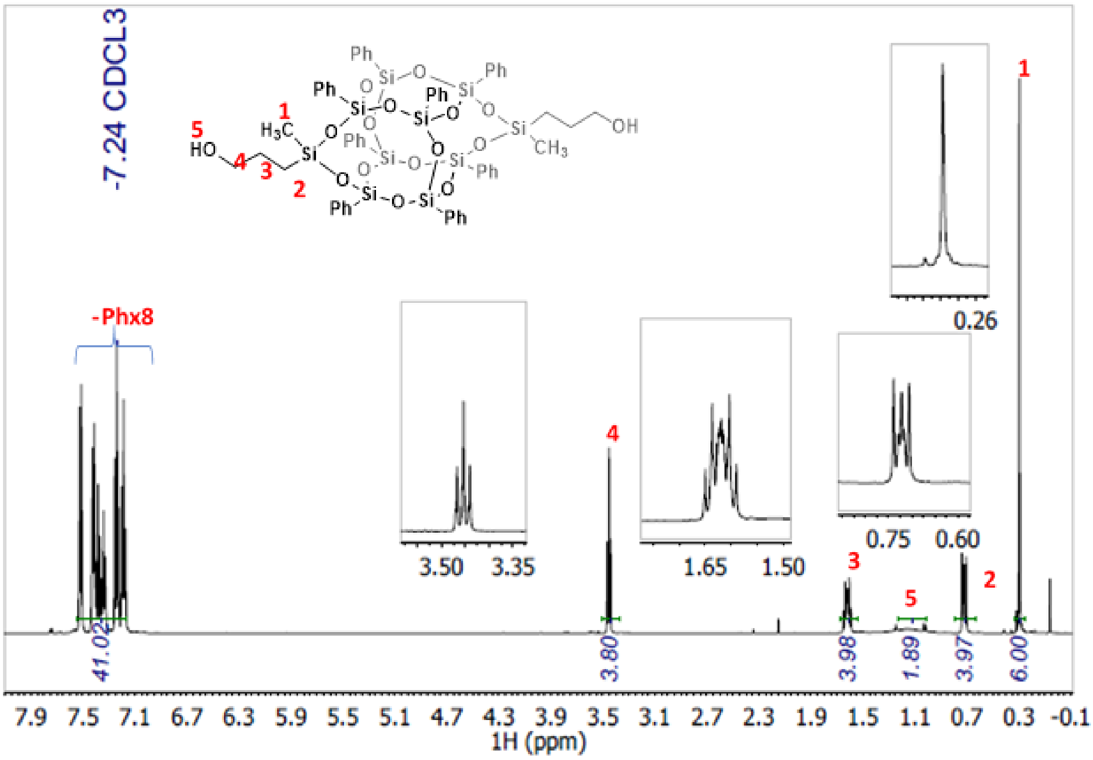


Figure S6. ^{1}H NMR (500 MHz, CDCl$_3$, 300 K) spectrum of **2**.

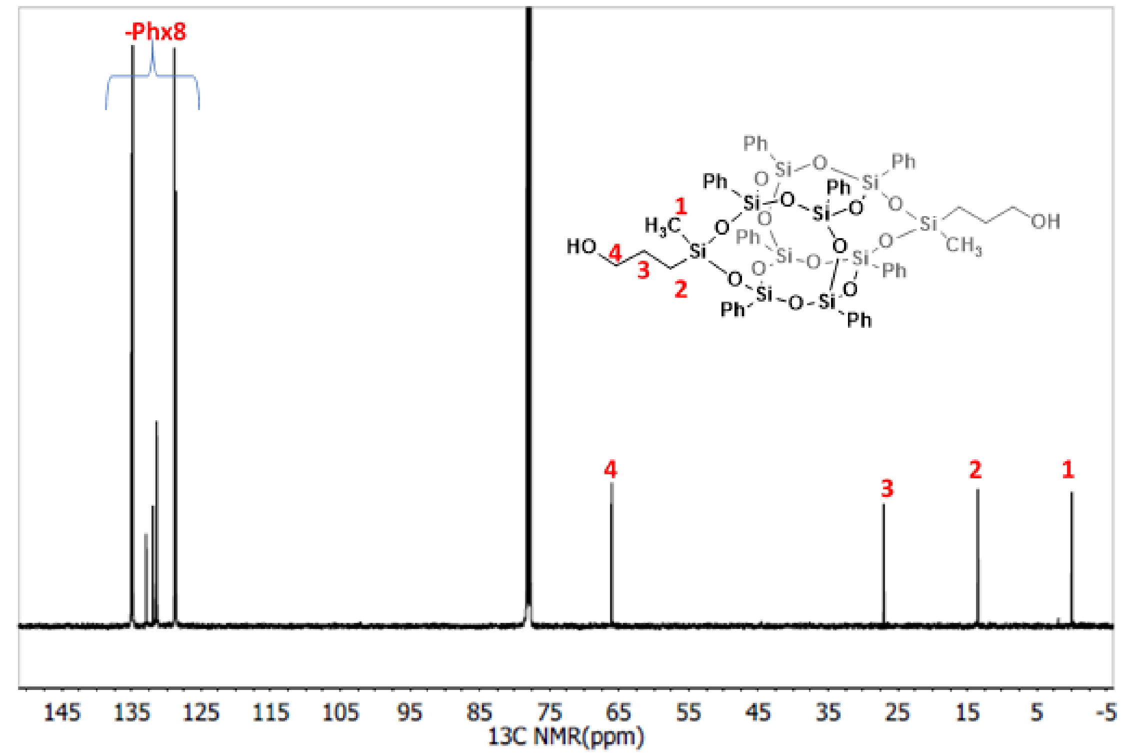


Figure S7. ^{13}C NMR (500 MHz, $CDCl_3$, 300 K) spectrum of **2**.

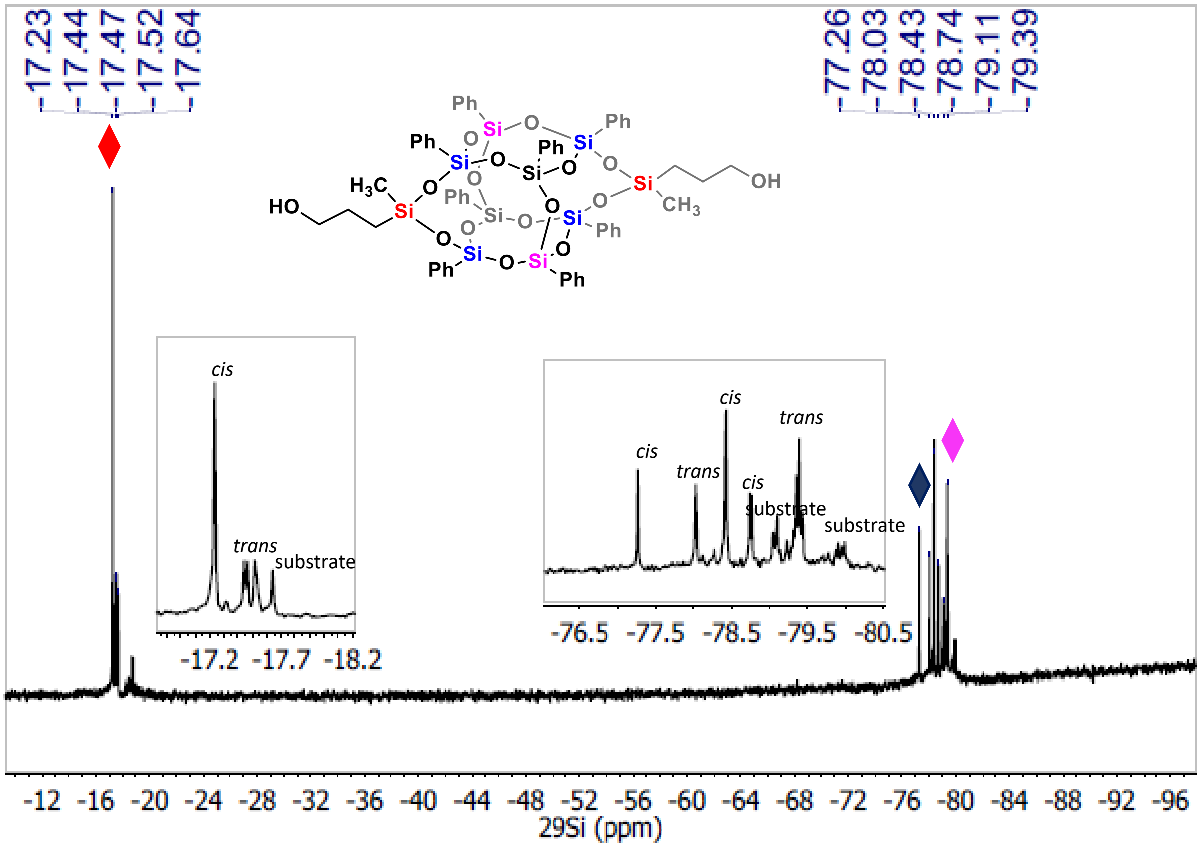
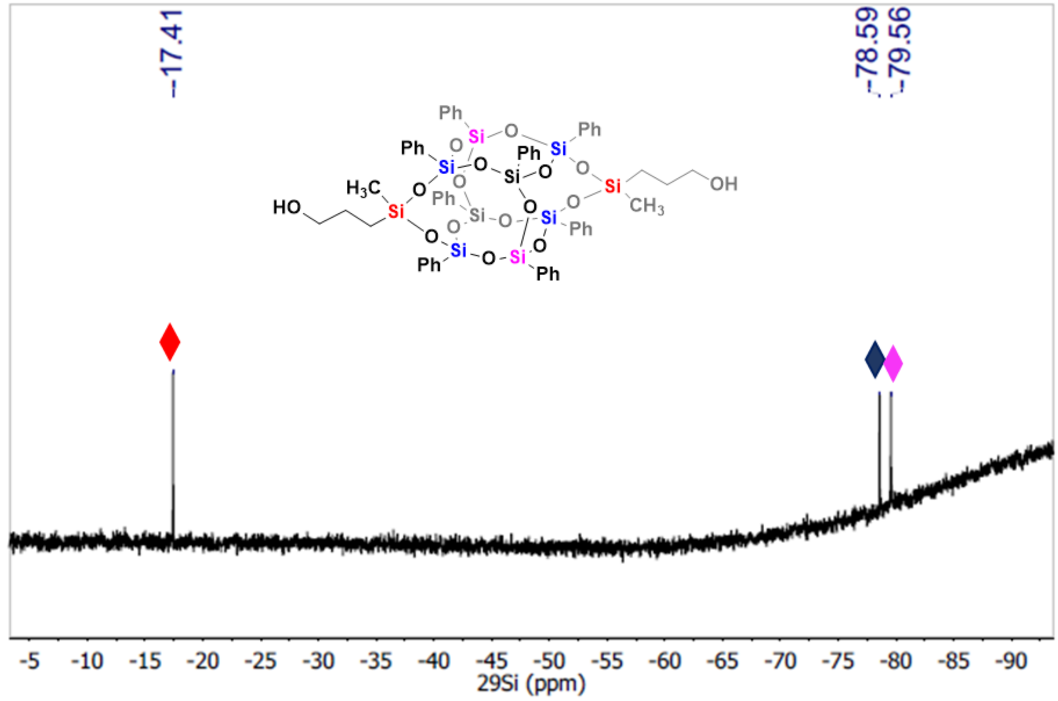


Figure S8. ^{29}Si NMR (126 MHz, $CDCl_3$, 300 K) spectrum of **2** (*cis*/*trans* mixture). Silsesquioxane substrate: DDSQ-Me_2H_2 (**1**).



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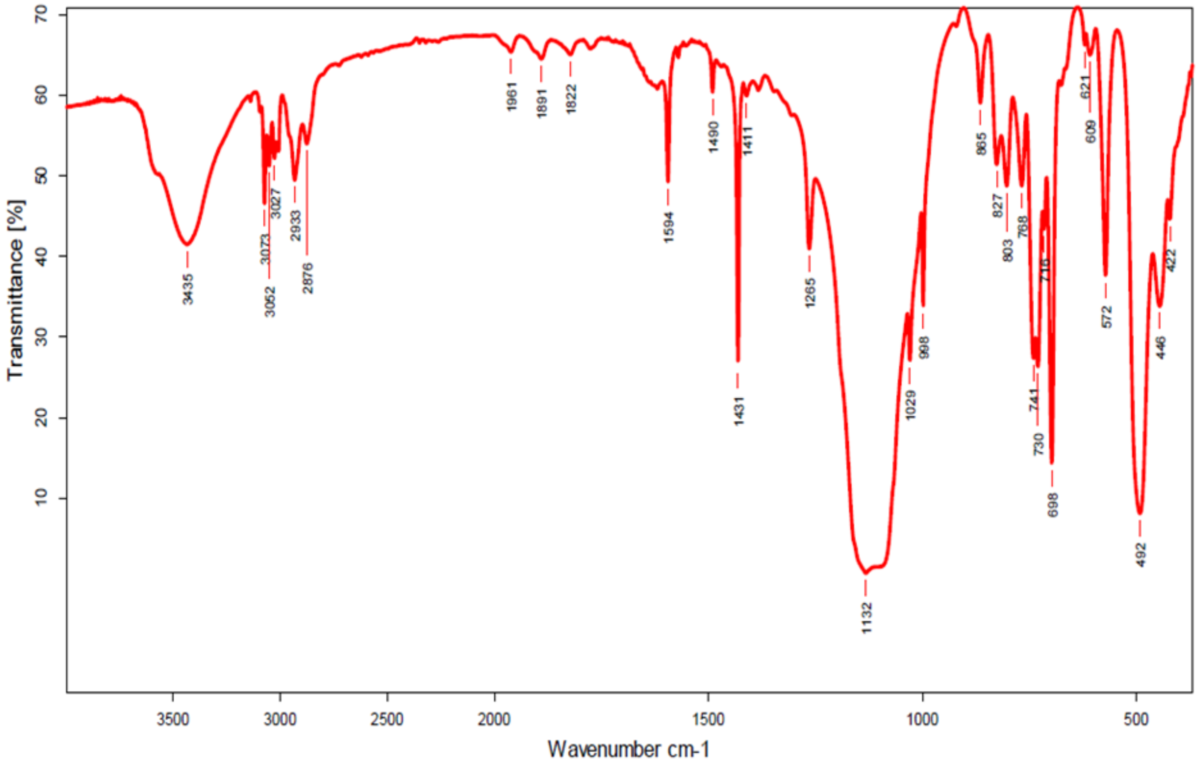

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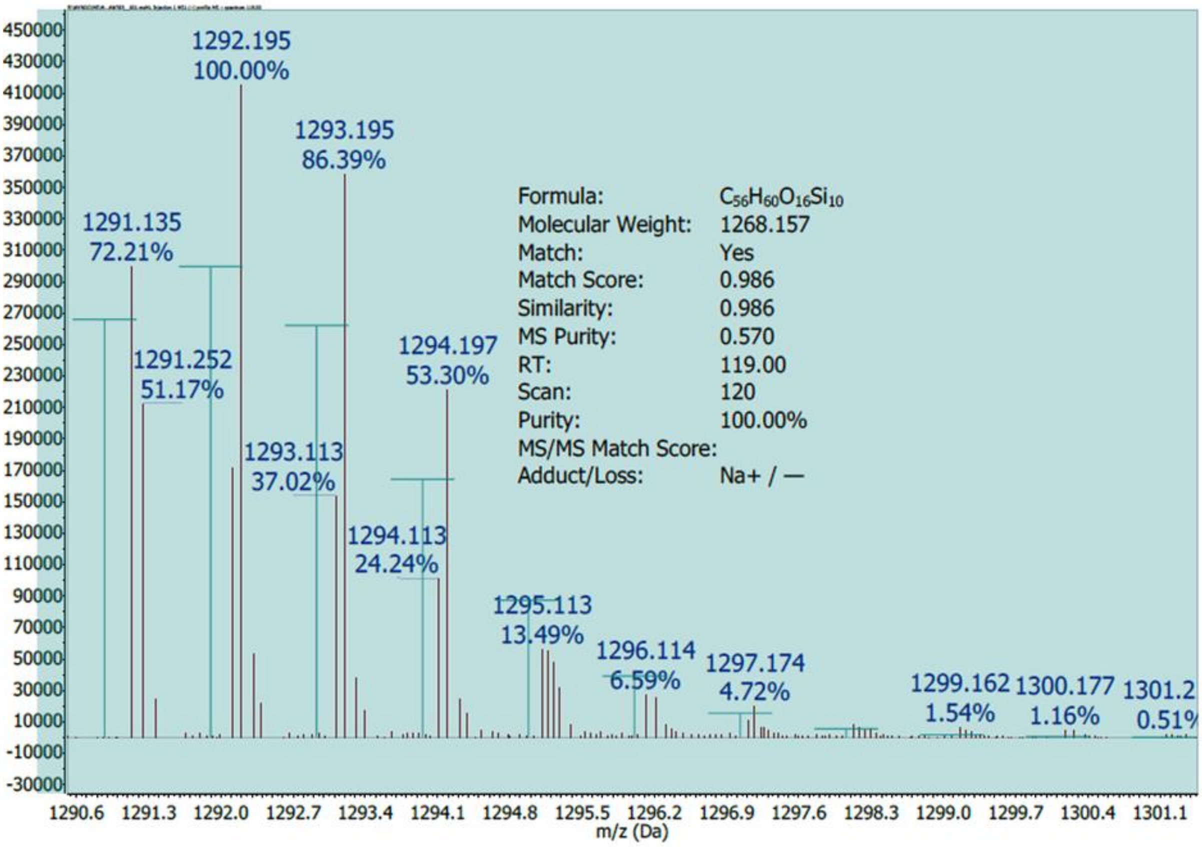


Figure S11. HRMS (ESI-TOF) of **2** ([M + Na]$^+$) with comparison with the calculated value.

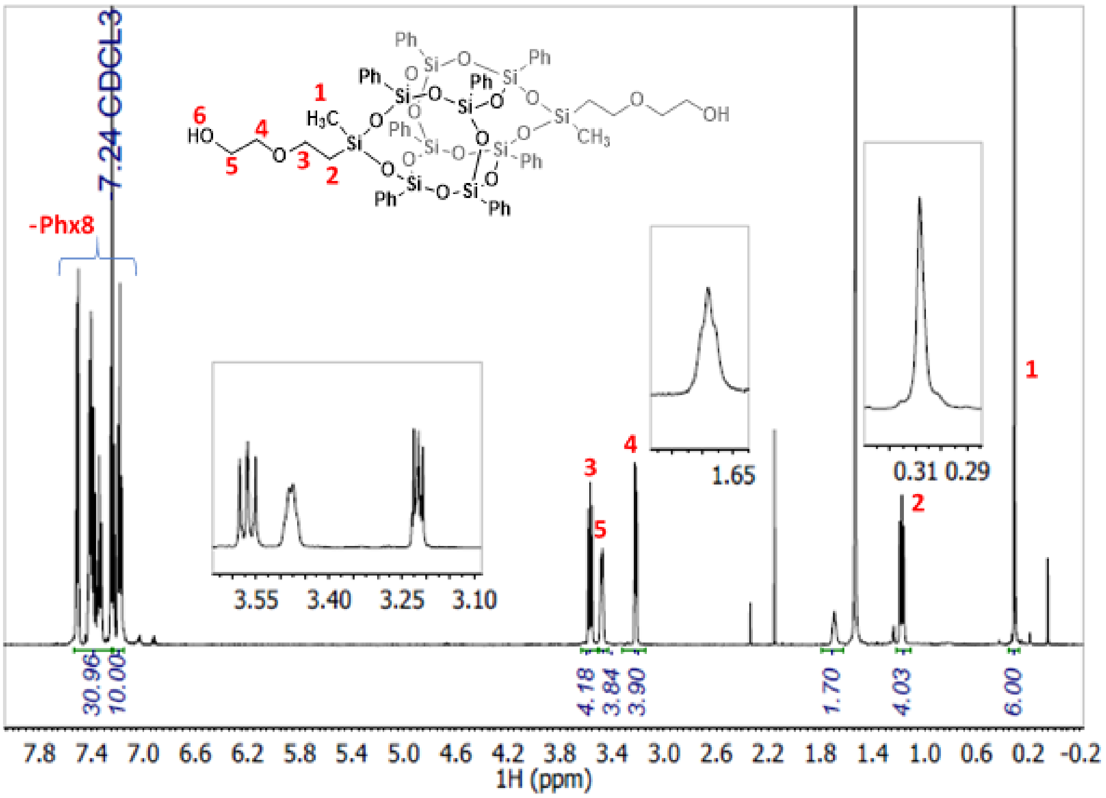


Figure S12. ^{1}H NMR (500 MHz, $CDCl_3$, 300 K) spectrum of **3**.

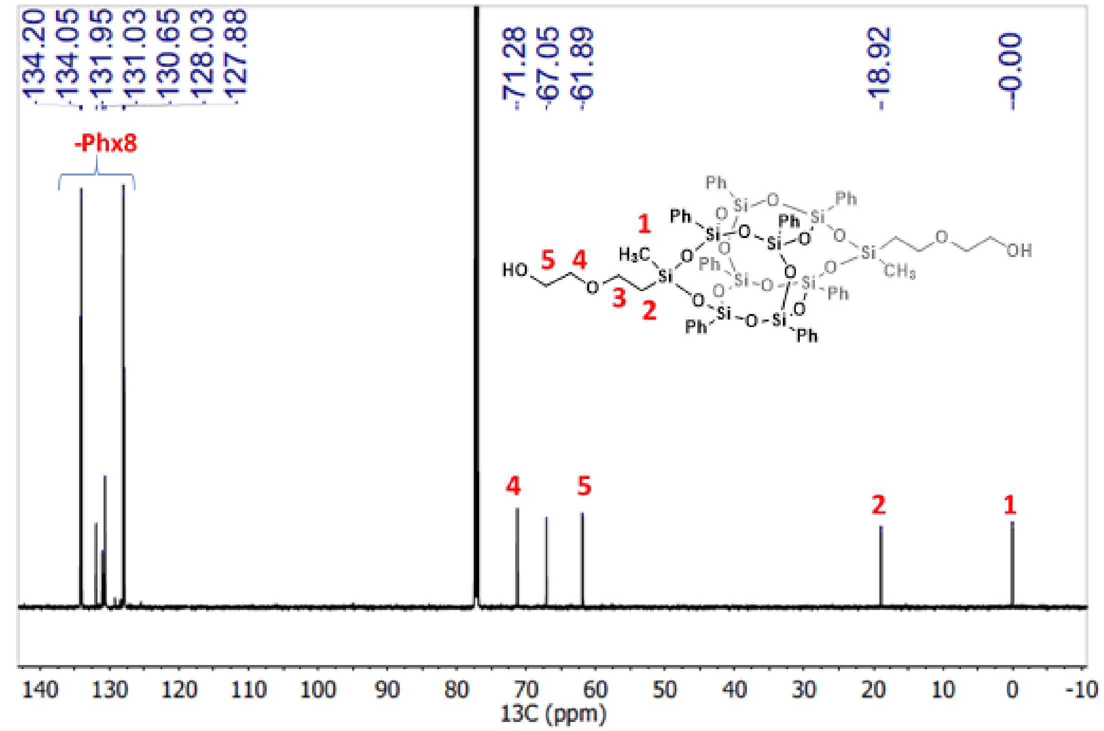
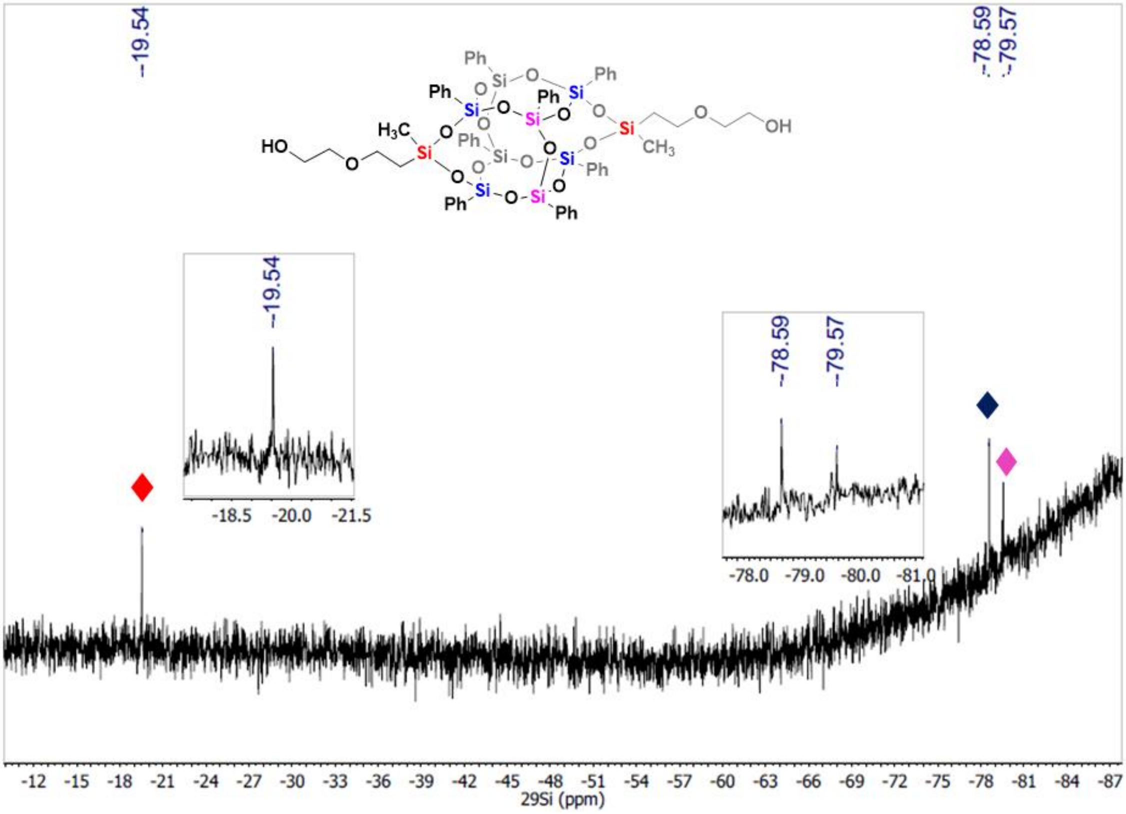


Figure S13. ^{13}C NMR (500 MHz, $CDCl_3$, 300 K) spectrum of **3**.



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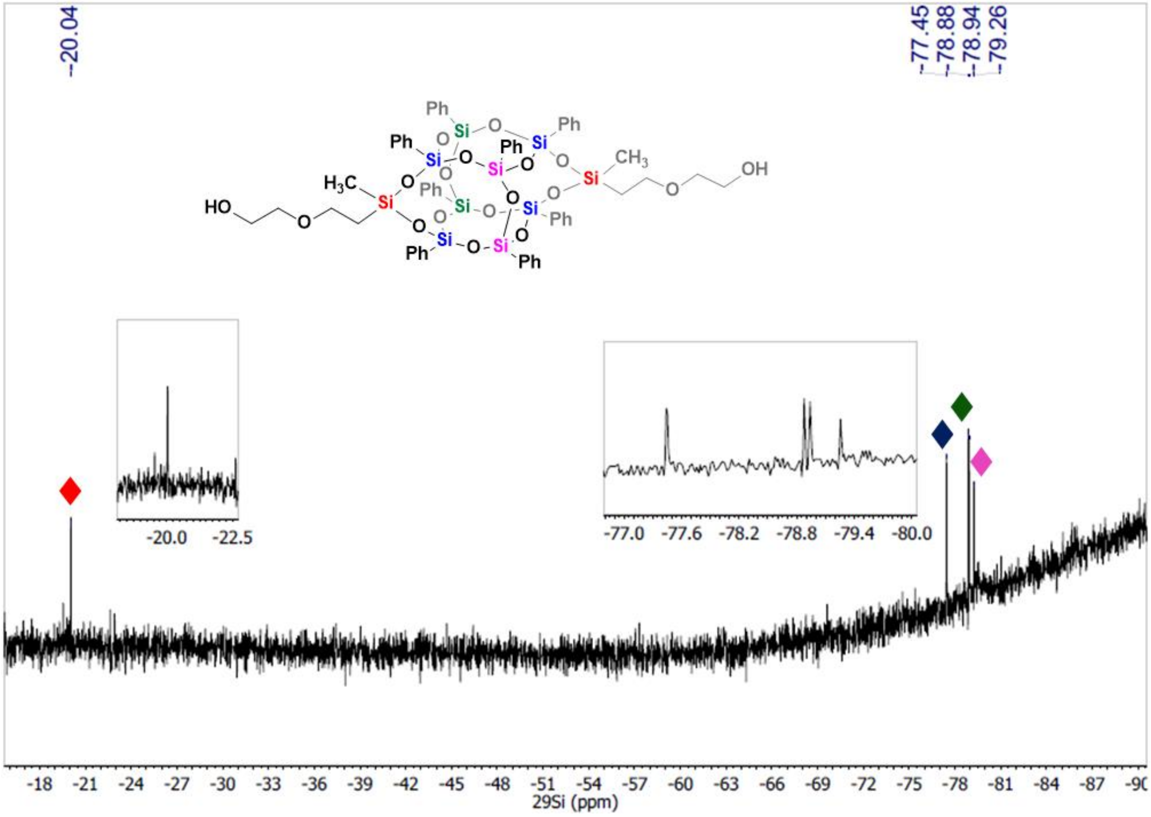


Figure S15. ^{29}Si NMR (99 MHz, CDCl$_3$, 300 K) spectrum of *cis*-**3**.

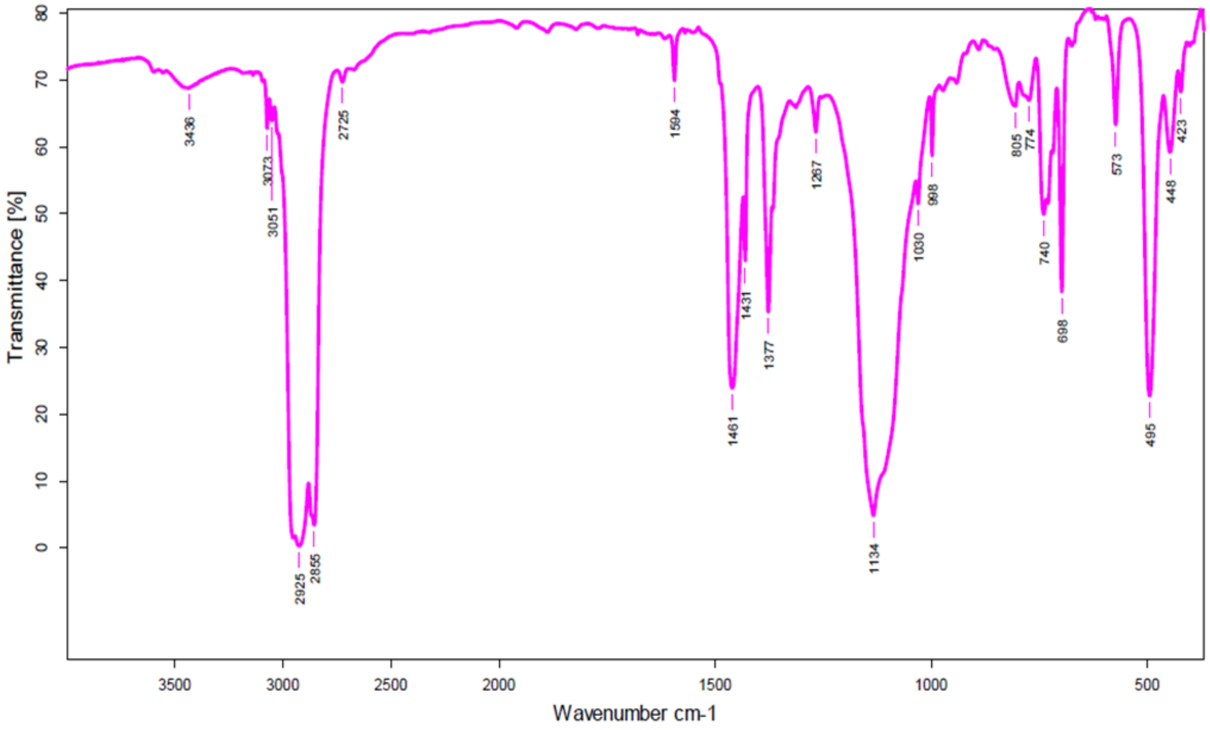


Figure S16. FT-IR spectrum (KBr pellet) of **3**.

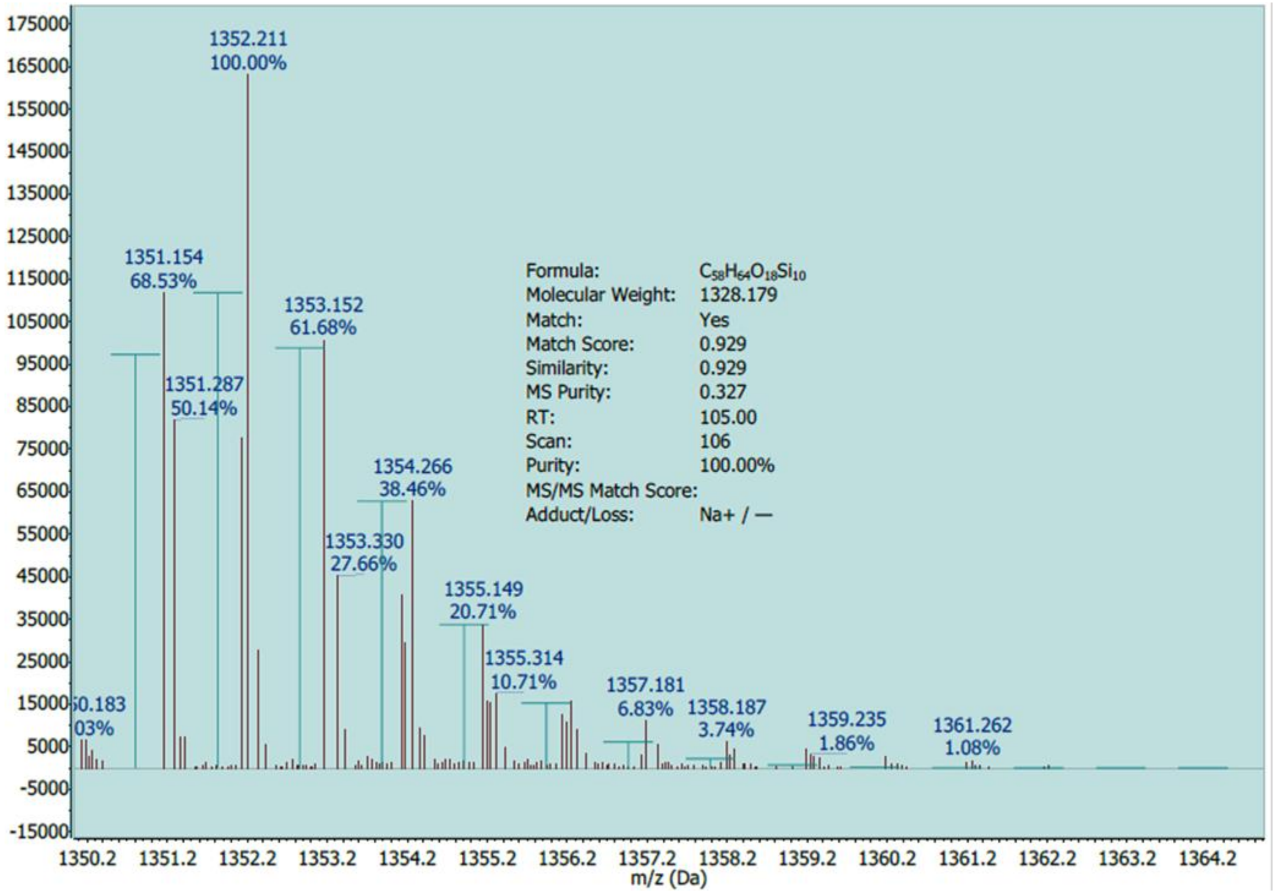


Figure S17. HRMS (ESI-TOF) of **3** ([M + Na]$^+$) with comparison with the calculated value.

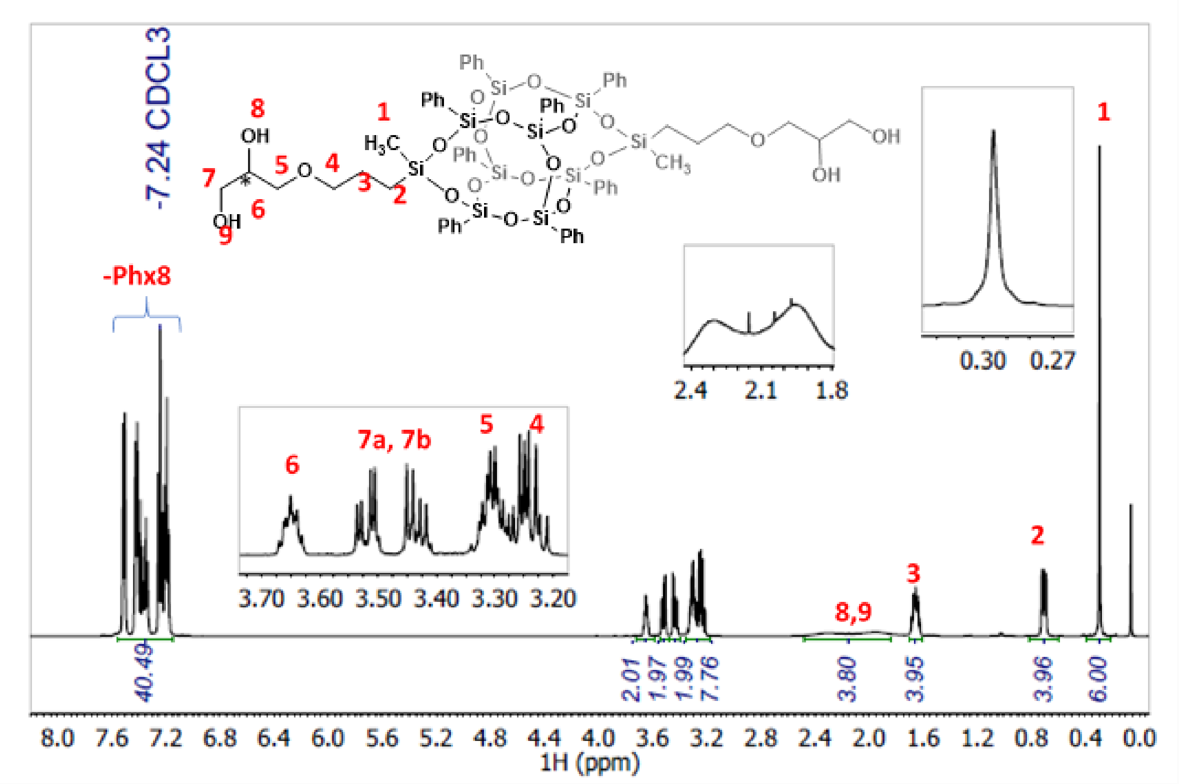


Figure S18. ^{1}H NMR (500 MHz, $CDCl_3$, 300 K) spectrum of **4**.

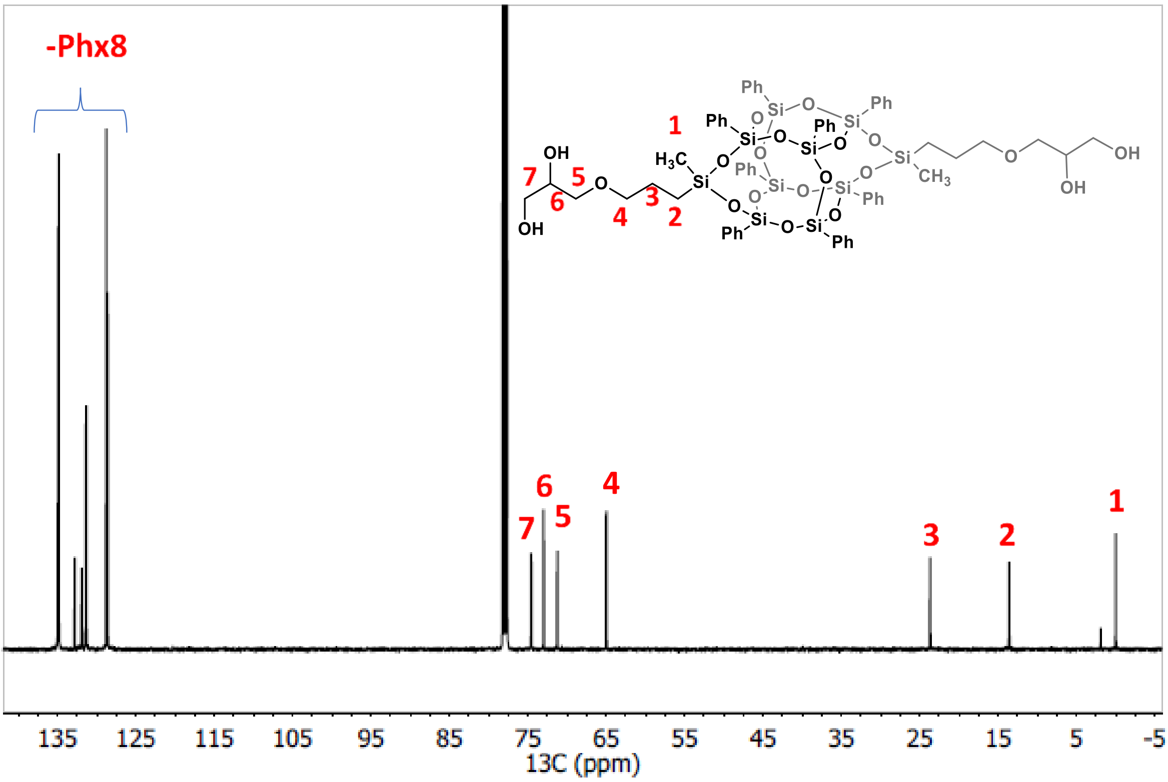
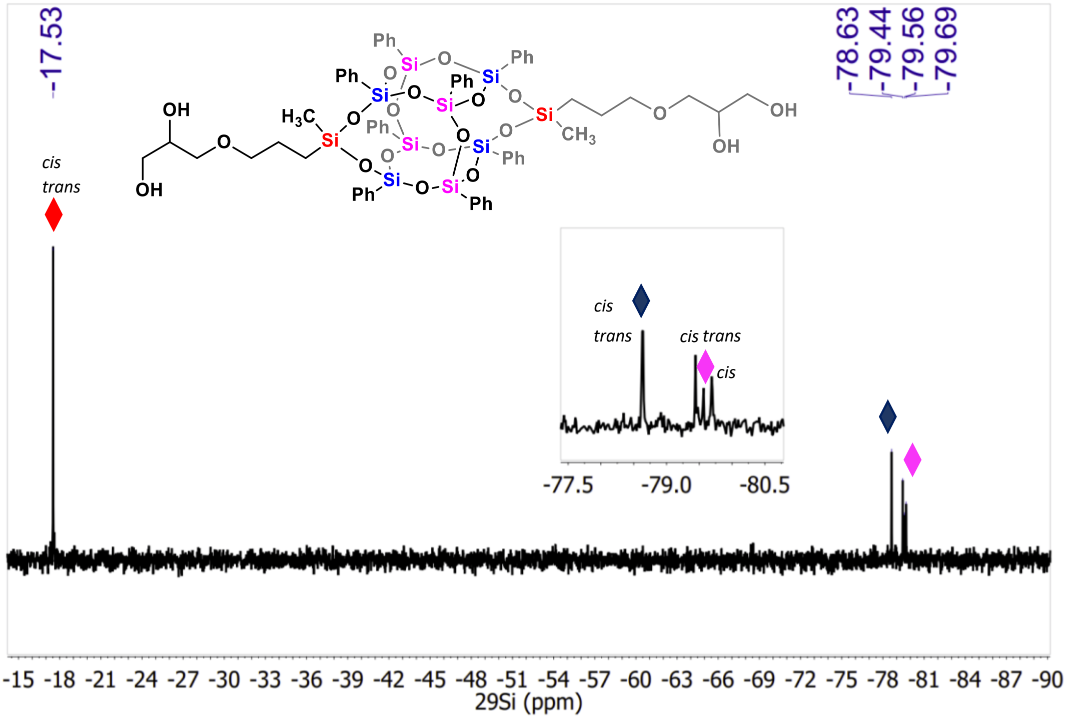


Figure S19. ^{13}C NMR (500 MHz, CDCl$_3$, 300 K) spectrum of **4**.



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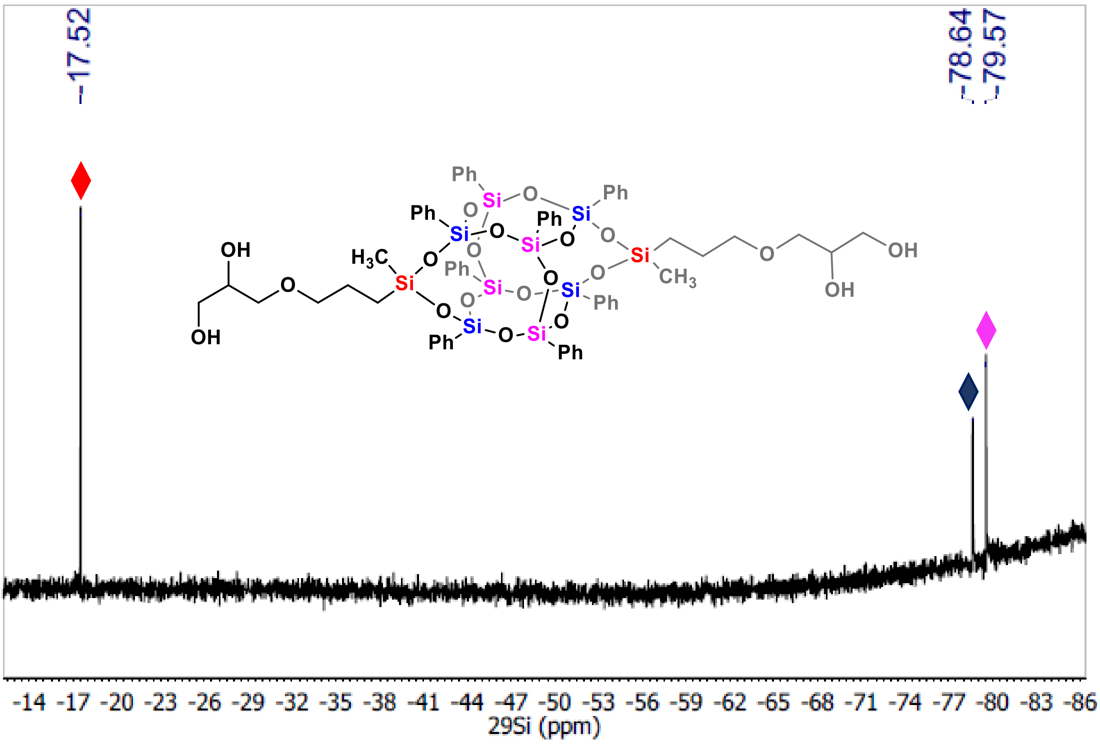

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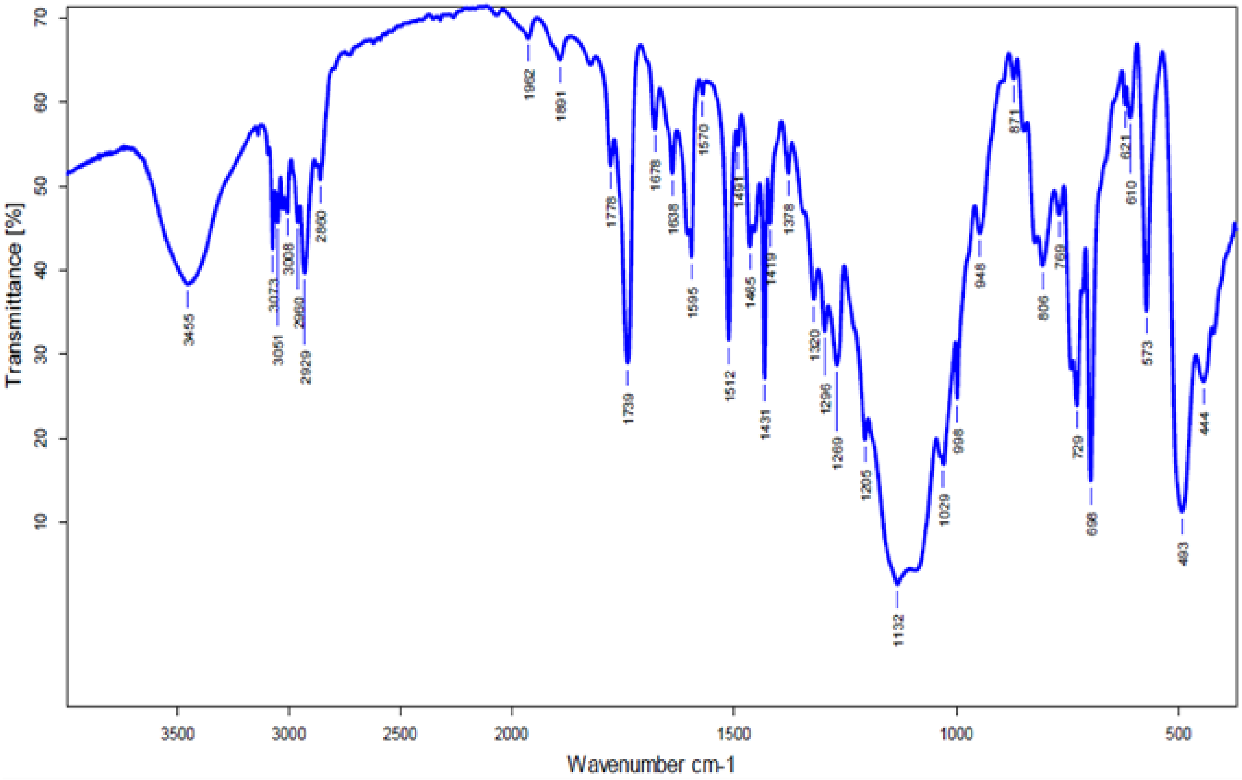

Figure S22. FT-IR spectrum (KBr pellet) of **4**.

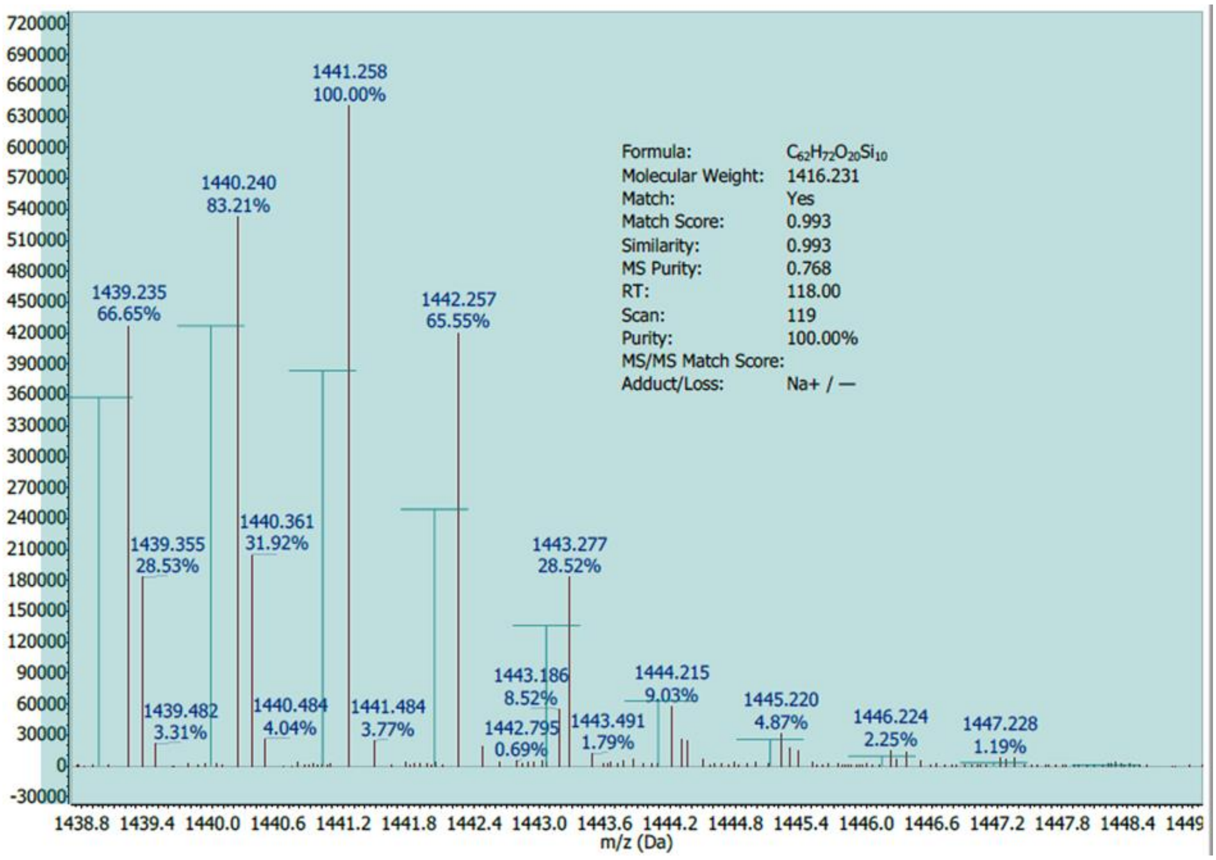

Figure S23. HRMS (ESI-TOF) of **4** ($[M + Na]^+$) with comparison with the calculated value.

X-ray crystallographic data

X-ray crystallographic data for *trans*-1

X-ray quality crystals were prepared by slow evaporation from saturated toluene. X-ray diffraction data for the crystal of *trans*-**1** were collected at 100(2) K on a κ-geometry Rigaku XtaLAB Synergy DW four-circle diffractometer (ω scans) with Mo*K*α radiation (rotating-anode) and Hybrid Photon Counting (HPC) HyPix-Arc 150° detector. Data collection, cell refinement, data reduction, and analysis, including empirical (multi-scan) absorption correction, were carried out with *CrysAlisPRO*.[1] Using Olex2[6], the structure was solved with the SHELXS97[2] program using direct methods and refined on F^2 by a full-matrix least-squares technique using SHELXL-2014 program[3] with anisotropic displacement parameters for all disordered non-H atoms.

There are two symmetry-independent molecules in a unit cell. For each of them, O-$Si(CH_3)(H)$-O fragments were found to be disordered and were refined in three positions, each with SOFs = (1) 0.25 and 0.3 and 0.45 and (2) 0.16, 0.3, and 0.54. Restraints on anisotropic displacement parameters (SIMU, ISOR) were applied to get an acceptable and appropriate model of the disorder.

All H atoms were included using geometrical considerations and were refined using a riding model, with C–H = 0.95–0.99 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ for CH and CH_2 or $U_{iso}(H) = 1.5U_{eq}(C)$ for CH_3 groups.

Figures presenting X-ray structures were made using the *Diamond* program.[5] The details of structure refinement are given in Table S1. The crystallographic information file (CIF) is deposited at the Cambridge Crystallographic Data Centre (CCDC No. 2158320) and provided as Electronic Supporting Information (ESI).

X-ray crystallographic data for *trans*-2

X-ray quality crystals were prepared by slow evaporation from a saturated mixture of THF and methanol. X-ray diffraction data for the crystal of *trans*-**2** were collected at 100(2) K on Xcalibur, Ruby, Gemini ultra (ω scans) with Cu*K*α radiation (rotating-anode) and CCD plate detector. Data collection, cell refinement, data reduction, and analysis, including empirical (multi-scan) absorption correction, were carried out with *CrysAlisPRO*.[1] Using Olex2[6], the structure was solved with the SHELXS97[2] program using direct methods and refined on F^2 by a full-matrix least-squares technique using SHELXL-2014 program[3] with anisotropic displacement parameters for all disordered non-H atoms.

Both $CH_2CH_2CH_2OH$ fragments were found to be disordered and were refined in two positions, each with SOFs = 0.5. Additionally, two of the phenyl rings were modeled in two positions with SOFs refined to: 1) 0.5 and 0.5, 2) 0.56(2) and 0.44(2) (position with lower occupancy was refined as a rigid group with AFIX 66 instruction in the *SHELXL-2014*). Some geometrical restraints (DFIX, SADI, SAME) on anisotropic displacement parameters (SIMU, ISOR) were applied to get an acceptable and appropriate model of the disorder.

All H atoms were included using geometrical considerations and were refined using a riding model, with O–H = 0.84 and C–H = 0.95–0.99 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ for CH and CH_2 or $U_{iso}(H) = 1.5U_{eq}(O,C)$ for OH and CH_3 groups.

Figures presenting X-ray structures were made using the *Diamond* program.[4] The details of structure refinement are given in Table S2. The crystallographic information file (CIF) is deposited at the Cambridge Crystallographic Data Centre (CCDC No. 2158321) and provided as Electronic Supporting Information (ESI).

X-ray crystallographic data for *cis*-3^1, *cis*-3^2, *cis*-3^3

X-ray quality crystals were prepared by slow evaporation from saturated solvents. X-ray diffraction data for the crystal of *cis*-$\mathbf{3}^1$ and *cis*-$\mathbf{3}^2$ were collected at 100 K and 300 K on a κ-geometry Rigaku XtaLAB Synergy DW four-circle diffractometer (ω scans) with Mo*K*α radiation (rotating-anode) and Hybrid Photon Counting (HPC) HyPix-Arc 150° detector. X-ray diffraction data for the crystal of *cis*-$\mathbf{3}^3$ were collected at 100 K on Kuma KM4-CCD, Oxford Diffraction Xcalibur PX, Agilent Technologies Xcalibur R with CuKα radiation. Data collection, cell refinement, data reduction, and analysis, including empirical (multi-scan) absorption correction, were carried out with *CrysAlisPRO*.[1] Using Olex2[6], the structure was solved with the SHELXS97[2] program using direct methods and refined on F^2 by a full-matrix least-squares technique using SHELXL-2014 program[3] with anisotropic displacement parameters for all disordered non-H atoms.

At room temperature, the molecule (*cis*-$\mathbf{3}^2$) is located at a special position (with .m. symmetry); the asymmetric part contains methanol molecule (with SOFs refined to 0.5). The mirror symmetry generates the disorder of $CH_2CH_2OCH_2CH_2OH$ chains, which occupy two equivalent positions with 0.5 occupancies. Some geometrical restraints (DFIX, SADI, SAME) on anisotropic displacement parameters (SIMU, ISOR) were applied to get an acceptable disorder model. The disorder of the side chains is also present at 100 K (*cis*-$\mathbf{3}^1$). At low temperatures (below 220K), the symmetry decreases from monoclinic to triclinic, introducing the domain structure. Thus, the crystal structure at 100 K was refined based on the diffraction patterns from two twin domains of 0.6/0.4 ratio. Due to the twinning and smeared diffraction peaks coming from poor crystallinity of the *cis*-**3** crystals, the overlapped peaks result in the moderate quality of the refinements. Methanol molecule could not be modelled satisfactorily in high temperature structure (*cis*-$\mathbf{3}^2$) and was therefore removed from the electron density map using the OLEX^2 solvent mask command. Voids values (104 Å^3) in asymmetric unit) indicate its presence.

All H atoms were included using geometrical considerations and were refined using a riding model, with O–H = 0.84 and C–H = 0.95–0.99 Å, with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$ for CH and CH_2 or $U_{\mathrm{iso}}(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{O,C})$ for OH and CH_3 groups.

Figures presenting X-ray structures were made using the *Diamond* program.[4] The details of structure refinement are given in Table S4. The crystallographic information file (CIF) is deposited at the Cambridge Crystallographic Data Centre (CCDC Nos. 2158323, 2158968) and provided as Electronic Supporting Information (ESI).

X-ray crystallographic data for *trans*-**3**

X-ray quality crystals were prepared by slow evaporation from a saturated mixture of toluene and THF. X-ray diffraction data for the crystal of *trans*-**3** were collected at 100(2) K on a κ-geometry Rigaku XtaLAB Synergy DW four-circle diffractometer (ω scans) with Mo*K*α radiation (rotating-anode) and Hybrid Photon Counting (HPC) HyPix-Arc 150° detector. Data collection, cell refinement, data reduction, and analysis, including empirical (multi-scan) absorption correction, were carried out with *CrysAlisPRO*.[1] The structure was solved using a dual-space algorithm with *SHELXT* program[2] and refined on F^2 by a full-matrix least-squares technique using *SHELXL-2014* program[3] with anisotropic displacement parameters for all disordered non-H atoms and those with site occupation factors (SOFs) > 0.5. Disordered O–Si–O fragments were refined anisotropically regardless of the SOFs of the O and Si atoms.

Both $O(O)Si(CH_3)(CH_2CH_2OCH_2CH_2OH)$ fragments were found to be disordered and were refined in two positions, each with SOFs = 0.688(4) and 0.312(4). Additionally, one of the phenyl rings was modeled in two positions with SOFs refined to 0.774(11) and 0.226(11) (position with lower occupancy was refined as a rigid group with AFIX 66 instruction in the *SHELXL-2014*). Some geometrical restraints (DFIX, SADI, SAME), restraints on anisotropic displacement parameters (SIMU, ISOR), constraints on the coordinates, and U_{ij} (EXYZ and EADP), and anti-bumping restraints (BUMP) were applied to get an acceptable and appropriate model of the disorder.

All H atoms were included using geometrical considerations and were refined using a riding model, with O–H = 0.84 and C–H = 0.95–0.99 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ for CH and CH_2 or $U_{iso}(H) = 1.5U_{eq}(O,C)$ for OH and CH_3 groups.

Figures presenting X-ray structures were made using the *Diamond* program.[4] The details of structure refinement are given in Table S3. The crystallographic information file (CIF) is deposited at the Cambridge Crystallographic Data Centre (CCDC No. 2158322) and provided as Electronic Supporting Information (ESI).

Table S1. Crystal data and structure refinement parameters for *trans*-**1**

CCDC	2158320
Chemical formula	$C_{51}H_{48}O_{14}Si_{10}\cdot C_7H_8$
$\boldsymbol{M_r}$	1246.45
Crystal system, space group	Triclinic, *P*-1
Temperature (K)	100
***a*, *b*, *c* (Å)**	9.9670(18), 14.266(2), 21.913(4)
α, β, γ (°)	101.96(2), 95.17(2), 95.54(2)
***V* ($Å^3$)**	3014.5(9)
Z	2
Radiation type	Cu *K*α
μ (mm^{-1})	2.60
Crystal size (mm)	0.32 × 0.14 × 0.12
Data collection	
Diffractometer	XtaLAB Synergy R, DW system, HyPix-Arc 150
Absorption correction	Multi-scan
$\boldsymbol{T_{min}, T_{max}}$	0.715, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	129327, 12380, 11600
$\boldsymbol{R_{int}}$	0.028
$(\sin \theta/\lambda)_{max}$ ($Å^{-1}$)	0.629
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.090, 1.05
No. of reflections	12380
No. of parameters	845
No. of restraints	667
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement.
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e $Å^{-3}$)	0.56, -0.37

Computer programs: CrysAlis PRO 1.171.39.46 (Rigaku OD, 2018), SHELXS97 (Sheldrick, 1990), SHELXL 2018/3 (Sheldrick, 2015), Olex2 1.3 (Dolomanov et al., 2009).

Table S2. Crystal data and structure refinement parameters for *trans*-**2**

CCDC	2158321
Chemical formula	$C_{56}H_{60}O_{16}Si_{10}$
M_r	1269.94
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
***a*, *b*, *c* (Å)**	10.091(3), 24.272(5), 25.791(4)
***V* ($Å^3$)**	6317(2)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	0.27
Crystal size (mm)	0.40 × 0.15 × 0.10
Data collection	
Diffractometer	Xcalibur, Ruby, Gemini ultra
Absorption correction	Analytical
T_{min}, T_{max}	0.218, 0.424
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	53772, 11399, 10833
R_{int}	0.059
$(\sin \theta/\lambda)_{max}$ ($Å^{-1}$)	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.172, 1.09
No. of reflections	0.063, 0.172, 1.09
No. of parameters	897
No. of restraints	515
H-atom treatment	H-atom parameters constrained.
$\Delta\rho_{max}$, $\Delta\rho_{min}$ ($e\ Å^{-3}$)	0.64, −0.63
Absolute structure	Flack x determined using 4421 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259)
Absolute structure parameter	−1.57 (6)

Computer programs: CrysAlis PRO 1.171.39.46 (Rigaku OD, 2018), SHELXS97 (Sheldrick, 1990), SHELXL 2018/3 (Sheldrick, 2015), Olex2 1.3 (Dolomanov et al., 2009).

Table S3. Crystal data and structure refinement parameters for *trans*-**3**

CCDC	2158322
Chemical formula	$C_{58}H_{64}O_{18}Si_{10}$
***M*r**	1329.99
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
***a*, *b*, *c* (Å)**	10.019(2), 24.275(3), 25.908(4)
***V* (Å^3)**	6301.1(18)
Z	4
Radiation type	Mo Kα
μ (mm^{-1})	0.28
Crystal size (mm)	0.40 × 0.15 × 0.10
Data collection	
Diffractometer	XtaLAB Synergy R, DW system, HyPix-Arc 150
Absorption correction	Multi-scan
T_{min}, T_{max}	0.673, 1.00
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	89692, 17429, 14381
R_{int}	0.032
(sin θ/λ)$_{max}$ (Å^{-1})	0.712
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.059, 0.172, 1.05
No. of reflections	17429
No. of parameters	898
No. of restraints	321
H-atom treatment	H-atom parameters constrained.
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.57, −0.48
Absolute structure	Flack x determined using 5683 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259)
Absolute structure parameter	0.019(19)

Computer programs: CrysAlis PRO (Rigaku OD, 2020), SHELXT-2014/7 (Sheldrick, 2015), SHELXL2014/7 (Sheldrick, 2015)

Table S4. Crystal data and structure refinement parameters for *cis*-$\mathbf{3^1}\cdot 0.5(CH_3OH)$ (100K) and *cis*-$\mathbf{3^2}\cdot 0.5(CH_3OH)$ (300K)

CCDC	2158323	2158968
Chemical formula	$C_{58}H_{64}O_{18}.Si_{10}\cdot 0.5(CH_3OH)$	$C_{58}H_{64}O_{18}.Si_{10}\cdot 0.5(CH_3OH)$
M_r	1346.01	1346.01
Crystal system, space group	Monoclinic, C2/m	Triclinic, P-1
Temperature (K)	300	100
a, b, c **(Å)**	19.753(4), 20.266(4), 20.202(4)	13.808(4), 13.953(6), 20.069(7)
α,β,γ(°)	90, 110.22(20), 90	101.52(4), 105.35(4), 92.32(4)
V **(Å^3)**	75899(3)	3636(2)
Z	4	2
Radiation type	Cu Kα	Cu Kα
μ (mm^{-1})	2.14	2.24
Crystal size (mm)	0.37 × 0.14 × 0.10	0.37 × 0.14 × 0.10
Data collection		
Diffractometer	XtaLAB Synergy R, DW system, HyPix-Arc 150	XtaLAB Synergy R, DW system, HyPix-Arc 150
Absorption correction	Multi-scan	Multi-scan
T_{min}, T_{max}	0.897, 0.973	0.760, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37983, 7714, 3910	23898, 23898, 11134
R_{int}	0.059	Refined as a 2-component twin
(sin θ/λ)$_{max}$ (Å^{-1})	0.625	0.629
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.116, 0.387, 1.28	0.148, 0.432, 1.36
No. of reflections	7714	23898
No. of parameters	432	756
No. of restraints	208	638
H-atom treatment	H-atom parameters constrained.	H-atom parameters constrained.
$\Delta\rho_{max}$, $\Delta\rho_{min}$ **(e Å^{-3})**	0.42, −0.59	0.73, −0.48

Computer programs: CrysAlis PRO 1.171.39.46 (Rigaku OD, 2018), SHELXS97 (Sheldrick, 1990), SHELXL 2018/3 (Sheldrick, 2015), Olex2 1.3 (Dolomanov et al., 2009).

Table S5. Hydrogen-bond geometry (Å, º) for *trans*-**2**

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*1B'*—H*1B'*···O1*B'*^{i}	0.84	1.89	2.65(4)	150.9
O*1B*—H*1B*···O*1B*^{ii}	0.84	1.94	2.65(3)	141.4

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −z+1; (ii) *x*+1/2, −*y*+1/2, −*z*+1.

Table S6. Hydrogen-bond geometry (Å, º) *for trans*-**3**

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2*A*—H2*A*···O1*B*^{i}	0.84	1.99	2.66 (3)	136
O2*B*—H2*B*···O1*A*^{ii}	0.84	2.40	3.16(3)	153
O2*B′*—H2*B′*···O2*A′*^{iii}	0.84	2.24	2.97(8)	145
C4*B*—H4*BA*···O2*A*^{iii}	0.99	2.17	2.96(4)	136
C4*B′*—H4*BD*···O2*A′*^{ii}	0.99	1.54	2.31(7)	131
C63—H63···O2*B*^{iv}	0.95	2.56	3.36(2)	142

Symmetry codes: : (i) −x+1/2, −y+1, z+1/2; (ii) −x+1/2, −y+1, z−1/2; (iii) −x+1, y−1/2, −z+1/2; (iv) x−1/2, −y+1/2, −z.

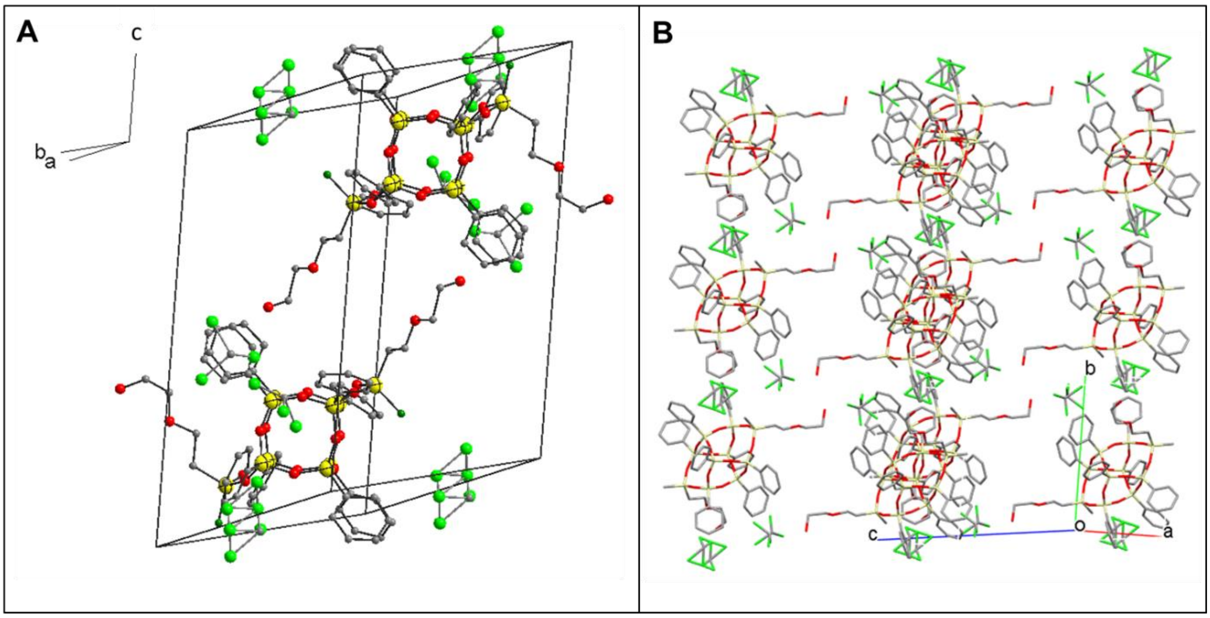

Figure S24. A: Crystal unit cell of *cis*-$\mathbf{3^3}$ with two symmetry-dependent molecules. B: Crystal packing of *cis*-$\mathbf{3^3}$. Hydrogen atoms are omitted for clarity. Representation model: “balls and sticks”.

Color code: C (grey), Si (yellow), O (red), methyl group (dark green), Cl (bright green). Hydrogen atoms are omitted for clarity.

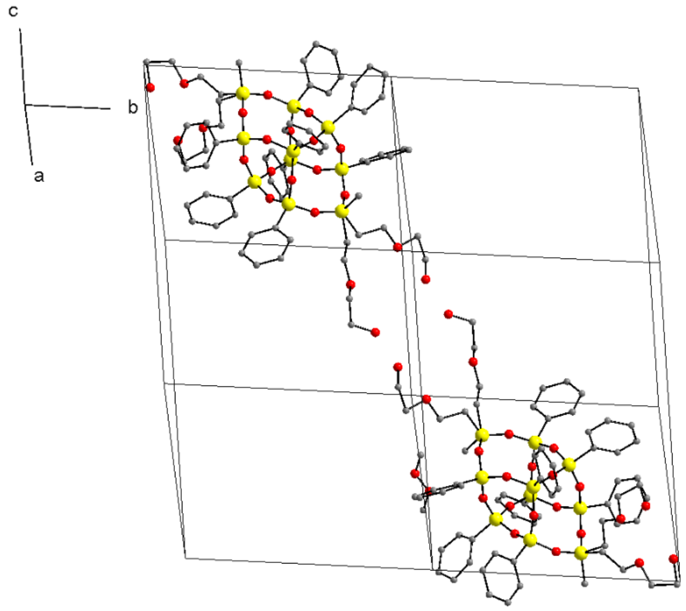

Figure S25. Fragment of molecular packing; two molecules organization for *cis*-$\mathbf{3^1}$.

Color code: C (grey), Si (yellow), O (red).
Hydrogen atoms are omitted for clarity. Representation model: “balls and sticks”.
Both $CH_2CH_2OCH_2CH_2OH$ substituents are disordered and were defined in two positions, each with SOFs = 1) 0.5 and 0.5; 2) 0.6 and 0.4 (at 100 K).
Methanol molecule is not fully occupied - its SOFs is 0.5.

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Dielectric Studies

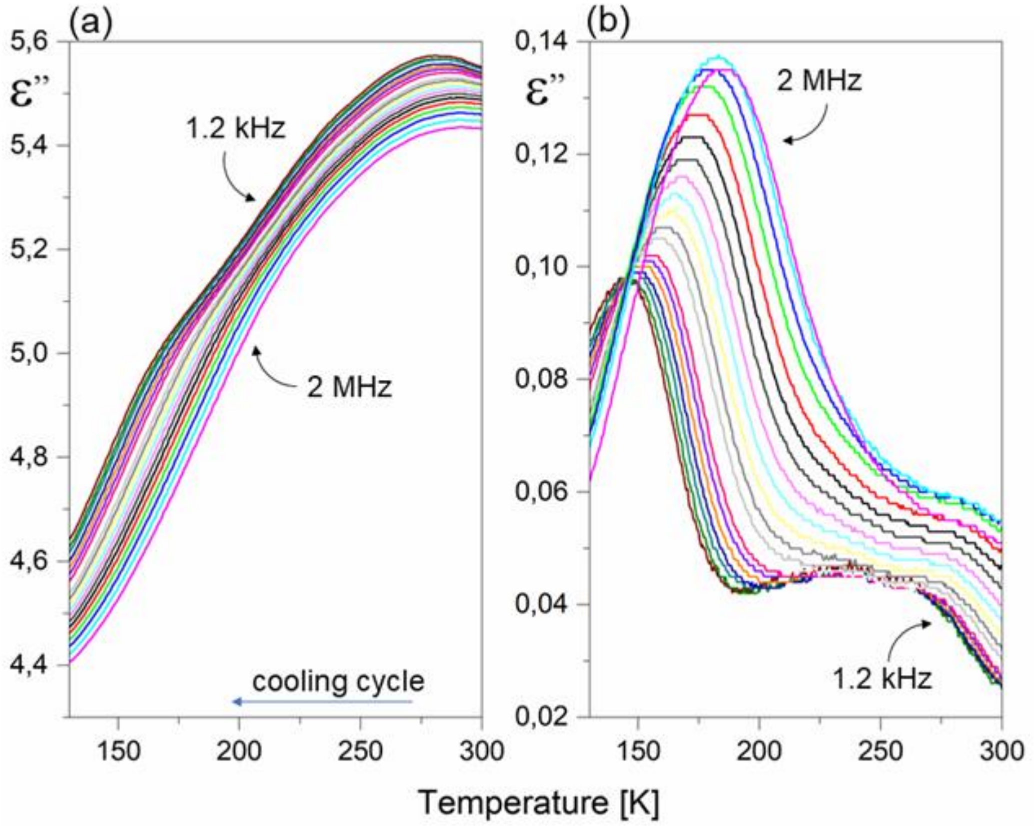

Figure S26. Temperature dependence of the (a) real and (b) imaginary part of the complex dielectric permittivity (pellet sample) for *cis*-**3**.

It has been found that the dielectric response is well described by the Cole-Cole relation:

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_o - \varepsilon_\infty}{1+(i\omega\tau)^{1-\alpha}} \qquad (1),$$

where ε_0 and ε_∞ are the low and high-frequency limits of the electric permittivity, respectively, ω is the angular frequency, τ is the macroscopic relaxation time. The parameter α characterizes the distribution of the macroscopic relaxation time. The Cole-Cole diagrams at selected temperatures are presented in **Figure S27**. In all cases, the Cole-Cole plots deviate from semi-circles over this temperature region (α- ranges between 0.22 and 0.41), which means that the relaxation process has a polydispersive nature.

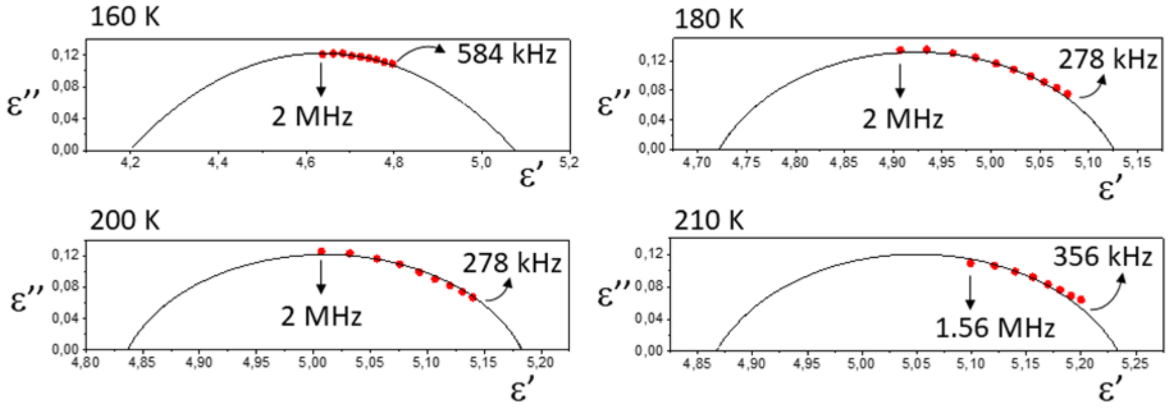

Figure S27. Cole-Cole diagrams at selected temperatures for *cis*-**3**.

We have fitted the experimental Cole-Cole plots at several temperatures with the equation (1) and determined the fitting parameters ε_0, ε_∞ and τ. The activation energy, E_a, can be estimated from the Arrhenius relation (eq. 2):

$$\tau = C\exp\left(\frac{E_a}{kT}\right) \qquad (2)$$

The E_a magnitudes being *ca.* **16.1 kJ/mol.**