Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information for

Tetra-Functional Double-Decker Silsesquioxanes as anchors for Reactive Functional Groups and potential synthons for hybrid materials

Katarzyna Mituła,[a] Julia Duszczak,[a] Dariusz Brząkalski,[a] Beata Dudziec,*[a] Maciej Kubicki,[a]
Bogdan Marciniec[a,b]

[a]*Faculty of Chemistry, Adam Mickiewicz University in Poznan, Umultowska 89B, 61-614 Poznan, Poland*
e-mail: beata.dudziec@gmail.com

[b]*Centre for Advanced Technologies, Adam Mickiewicz University in Poznan,*
Umultowska 89C, 61-614 Poznan, Poland

Table of contents:

1. General Considerations

The chemicals were purchased from the following sources: Hybrid Plastics for DDSQ tetrasilanol form ($C_{48}H_{44}O_{14}Si_8$) (DDSQ-4OH); Sigma-Aldrich for dichloromethane (DCM), tetrahydrofuran (THF), diethyl ether, toluene, *n*-hexane, chloroform-d, hexamethylphosphoramide (HMPA), Karstedt's catalyst – 2% xylene solution, chlorosilanes, ethynylmagnesium bromide, 1,5-hexadiene, 1,9-decadiene, calcium hydride, molecular sieves and anhydrous magnesium sulfate, trimethylamine and silica gel 60. Chloroethynyldimethylsilane was prepared according to the literature procedure.[1] All solvents were dried over sodium or CaH_2 prior to use and stored under argon over type 4A molecular sieves. All liquid substrates were also dried and degassed by bulb-to-bulb distillation. All syntheses were conducted under argon atmosphere using standard Schlenk-line and vacuum techniques.

1.1 Measurements

Nuclear Magnetic Resonance (NMR)

^1H, ^{13}C, and ^{29}Si Nuclear Magnetic Resonance (NMR) were performed on Brucker Ultra Shield 600, 400 and 300 spectrometers using $CDCl_3$ as a solvent. Chemical shifts are reported in ppm with reference to the residual solvent ($CHCl_3$) peaks for ^1H and ^{13}C and to TMS for ^{29}Si.

Matrix-assisted ultraviolet laser desorption/ionization time-of-flight mass spectroscopy (MALDI-TOF-MS)

Matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF-MS) mass spectra were recorded on a UltrafleXtreme mass spectrometer (Bruker Daltonics), equipped with a SmartBeam II laser (355 nm) in 500-4000 m/z range. 2,5-Dihydroxybenzoic acid (DHB, Bruker Daltonics, Bremen, Germany) served as matrix and was prepared in TA30 solvent (30:70 v/v acetonitrile:0.1% TFA in water) at 20 mg/mL concentration. Studied samples were dissolved in dichloromethane (2 mg/mL) and then mixed in a ratio 1:1 v/v with matrix solution. Matrix/sample mixtures (1 µL) were spotted onto the MALDI target and air-dried. Mass spectra were measured in reflection mode. The data were analysed using the software provided with the Ultraflex instrument - FlexAnalysis (version 3.4). Mass calibration (cubic calibration based on five to seven points) was performed using external standards (Peptide Calibration Standard).

Fourier Transform-Infrared (FT-IR)

Fourier Transform-Infrared (FT-IR) spectra were performed on a Bruker Tensor 27 Fourier transform spectrophotometer equipped with a SPECAC Golden Gate, diamond ATR unit with $2 cm^{-1}$resolution.

Thermogravimetric Analysis (TGA)

TGA analyses were performed using a TGA4000 (Perkin Elmer) with thermal gravimetric analyzer. The measurements were conducted in nitrogen atmosphere (flow of 20 mL/min) or air (flow of 60 mL/min), from 30°C to 995°C, at the heating rate of 10°C/min. The temperature of initial degradation (T_d) was taken as the onset temperature at which 5 wt% of mass loss occurs.

X-ray crystallography

X-ray diffraction data were collected at 100(1)K, by the ω-scan technique on an Agilent Technologies four-circle Xcalibur diffractometer (Eos detector) with graphite-monochromatized MoK$_\alpha$ radiation (λ=0.71073Å). The data were corrected for Lorentz-polarization and absorption effects.[2] The structures were solved with SIR92[3] and refined with the full-matrix least-squares procedure on F^2 by SHELXL97.[4] Scattering factors incorporated in SHELXL97 were used. All non-hydrogen atoms were refined anisotropically, hydrogen atoms bonded to Si atoms in **1-Me** were found in the difference Fourier maps and their positions were further refined, all other hydrogen atoms were placed in the calculated positions, and refined as 'riding model' with the isotropic displacement parameters set at 1.2 (1.5 for methyl groups) times the U$_{eq}$ value for appropriate non-hydrogen atom. Crystallographic data (excluding structure factors) for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, No. CCDC-1503391 (**1-Me**), CCDC- 967454 (**2-Me**) and CCDC-1544710 (**4-Me**). Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK. Fax: +44(1223)336-033, e-mail:deposit@ccdc.cam.ac.uk, or www: www.ccdc.cam.ac.uk.

Crystal data:

1-Me: $C_{56}H_{68}O_{14}Si_{12}$, M$_r$=1302.18, triclinic, P-1, a = 12.6679(11) Å, b = 12.7069(10) Å, c = 13.3265(10) Å, α = 77.041(6)°, β = 62.164(8)°, γ = 62.244(8)°, V = 1678.6(3) Å^3, Z = 1, F(000) = 684, d$_x$ = 1.29 g·cm^{-3}, μ = 0.29 mm^{-1}, 12905 reflections collected up to 2θ = 50°, 5893 unique (R$_{int}$ = 0.034), 4650 with I>2σ(I). Final R(I>2σ(I)) = 4.16%, wR2(I>2σ(I)) = 10.24%, R(all reflections) = 5.98%, wR2(all reflections) = 11.07%, S = 1.04, Δρ$_{max}$ = 0.44 e·Å^{-3}, Δρ$_{maxmin}$ =-0.32 e·Å^{-3}.

2-Me: $C_{64}H_{76}O_{14}Si_{12}$, M$_r$=1406.32, orthorhombic, P2$_1$2$_1$2$_1$, a = 12.927(2) Å, b = 21.826(3) Å, c = 26.008(4) Å, V = 7338(2) Å^3, Z = 4, F(000) = 2960, d$_x$ = 1.27 g·cm^{-3}, μ = 0.27mm^{-1}, 20636 reflections collected up to 2θ = 50°, 12187 unique (R$_{int}$ = 0.076), 10117 with I>2σ(I). Final R(I>2σ(I)) = 4.68%, wR2(I>2σ(I)) = 8.63%, R(all reflections) = 6.43%, wR2(all reflections) = 9.28%, S = 1.03, Δρ$_{max}$ = 0.31 e·Å^{-3}, Δρ$_{min}$ =-0.34 e·Å^{-3}.

4-Me: $C_{64}H_{68}O_{14}Si_{12}$, M$_r$=1398.26, monoclinic, P2$_1$/c, a = 22.8530(3) Å, b = 14.1212(2) Å, c = 22.8872(3) Å, β = 98.244(2)°, V = 7309.65(17) Å^3, Z = 4, F(000) = 2928, d$_x$ = 1.27 g·cm^{-3}, μ = 0.27 mm^{-1}, 68833 reflections collected up to 2θ = 53.2°, 14368 unique (R$_{int}$ = 0.040), 12007 with I>2σ(I). Final R(I>2σ(I)) = 3.94%, wR2(I>2σ(I)) = 10.47%, R(all reflections) = 4.98%, wR2(all reflections) = 11.11%, S = 1.03, Δρ$_{max}$ = 1.14 e·Å^{-3}, Δρ$_{min}$ = -0.44 e·Å^{-3}.

Figure S1. Three dimensional network of **(1-Me)**

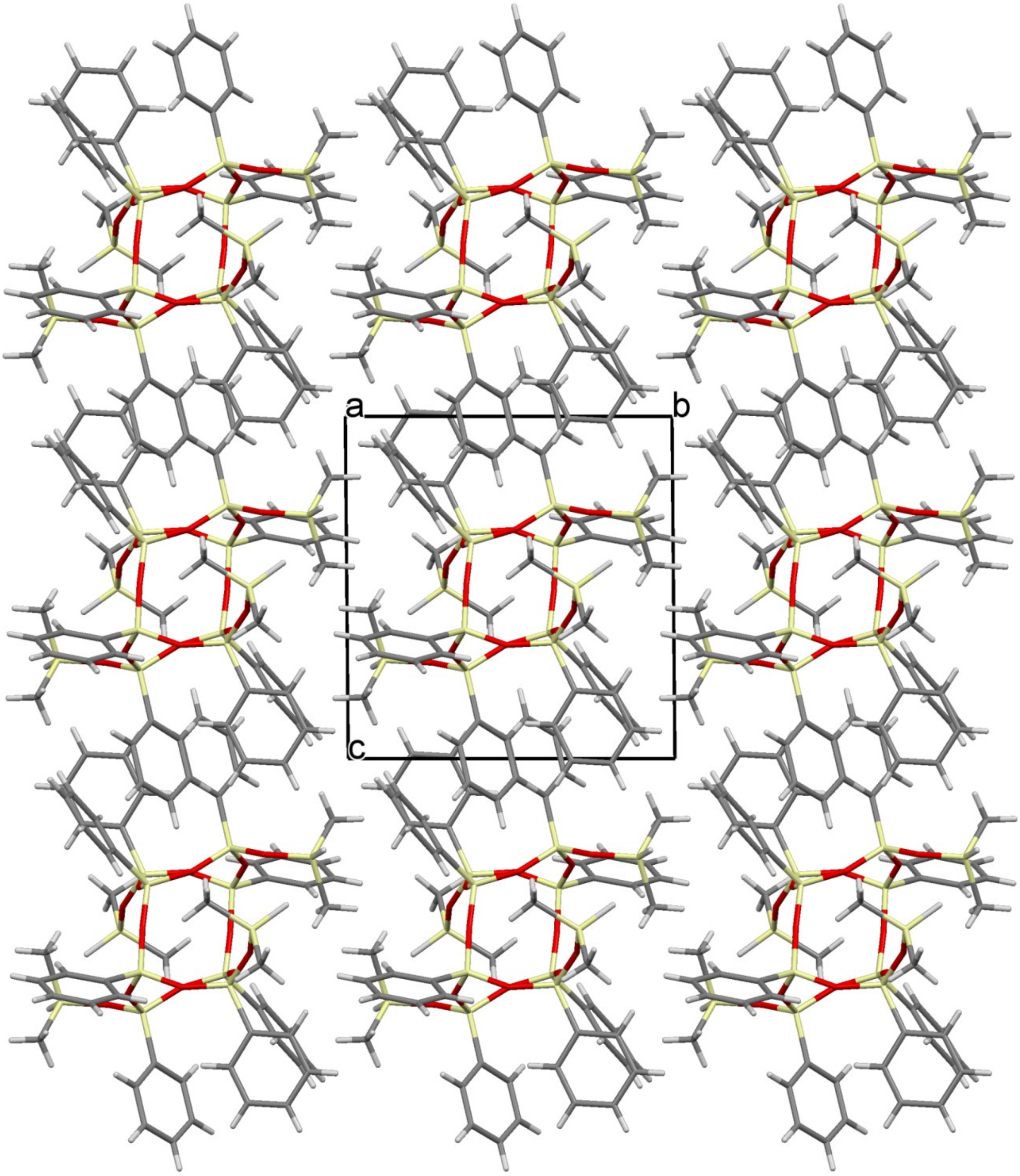


Figure S2. Three dimensional network of **(2-Me)**

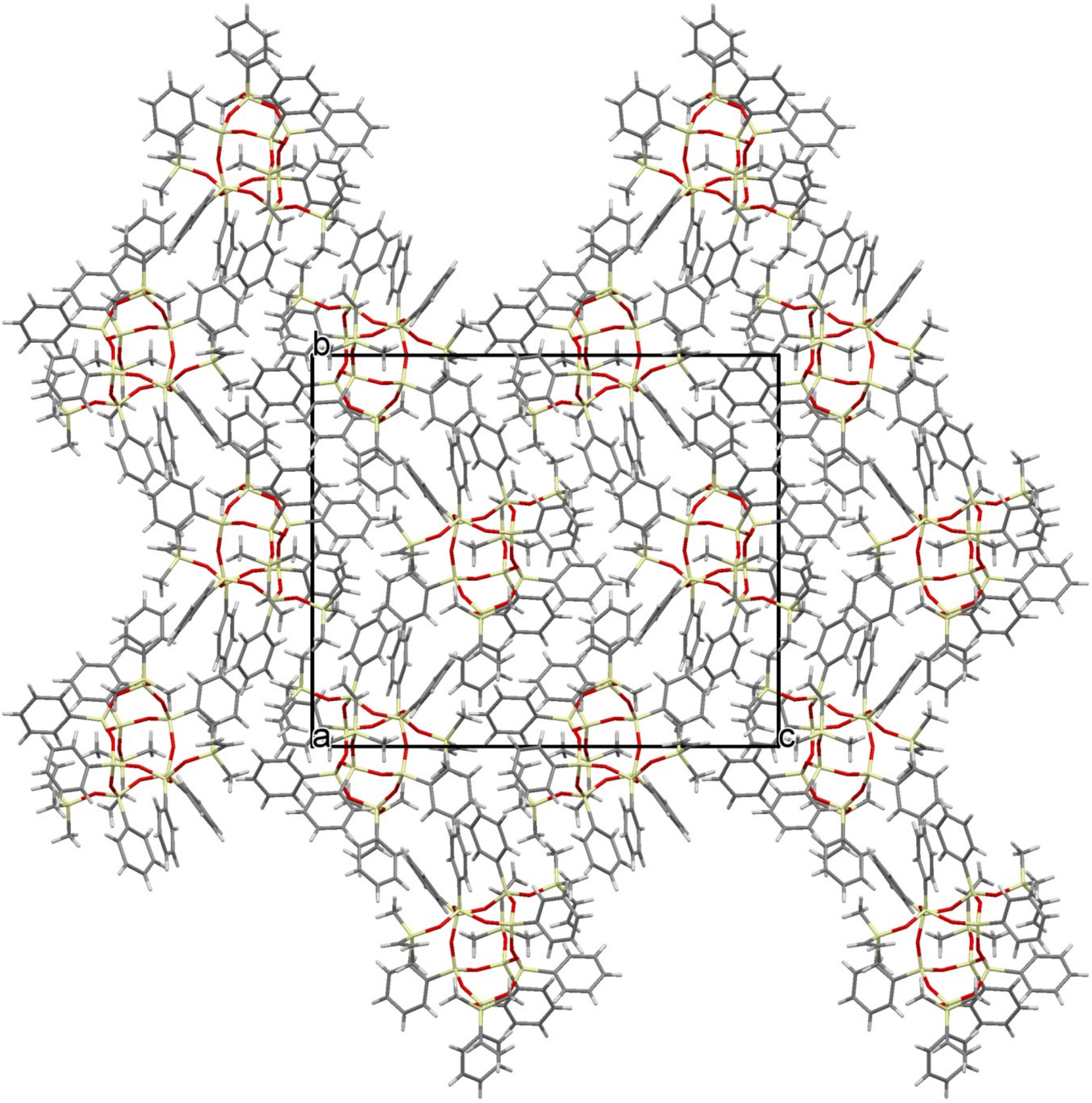
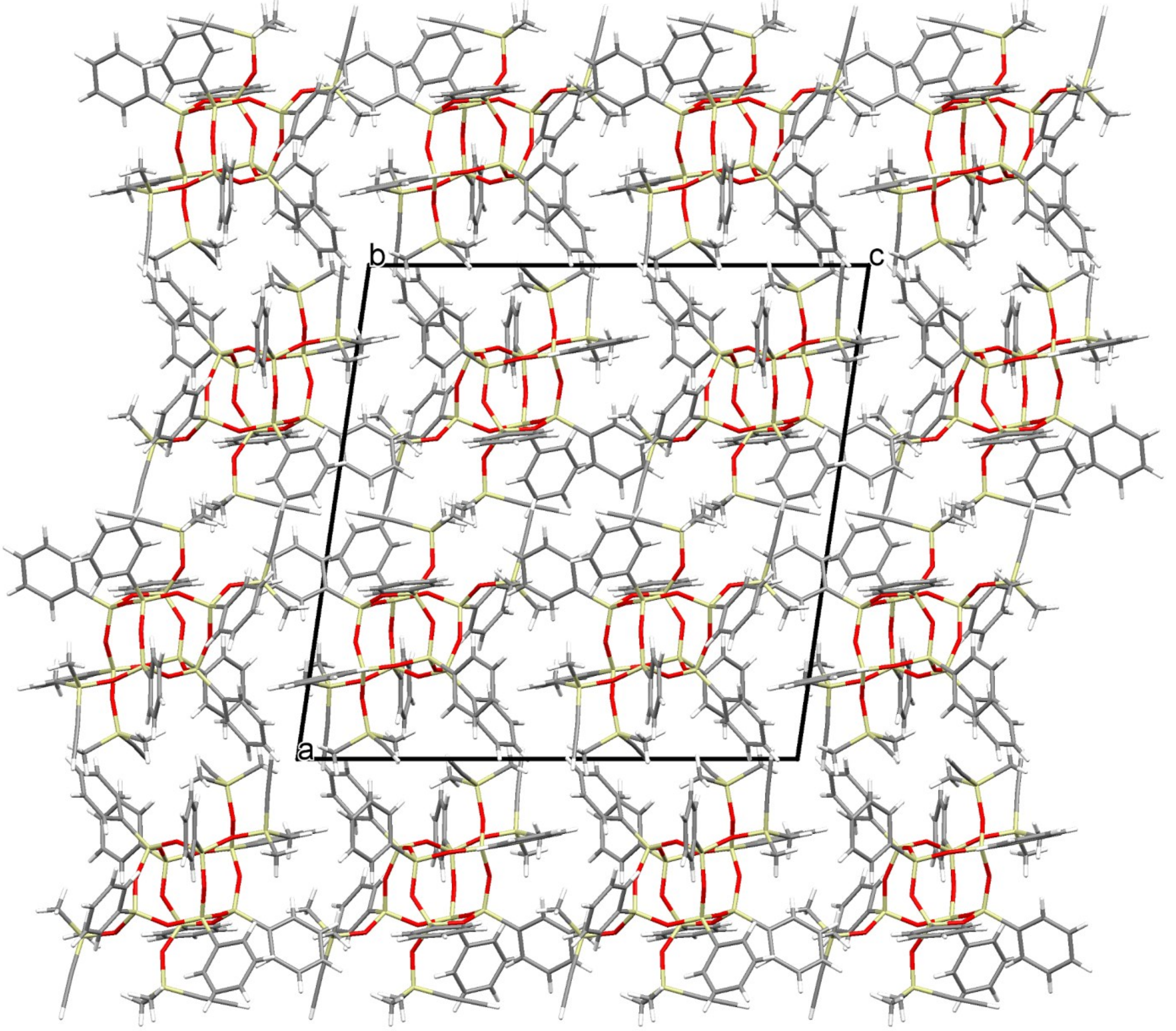
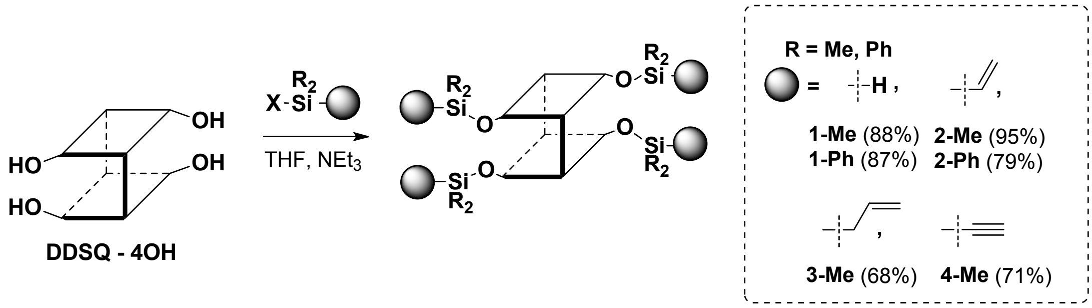


Figure S3. Three dimensional network of **(4-Me)**:



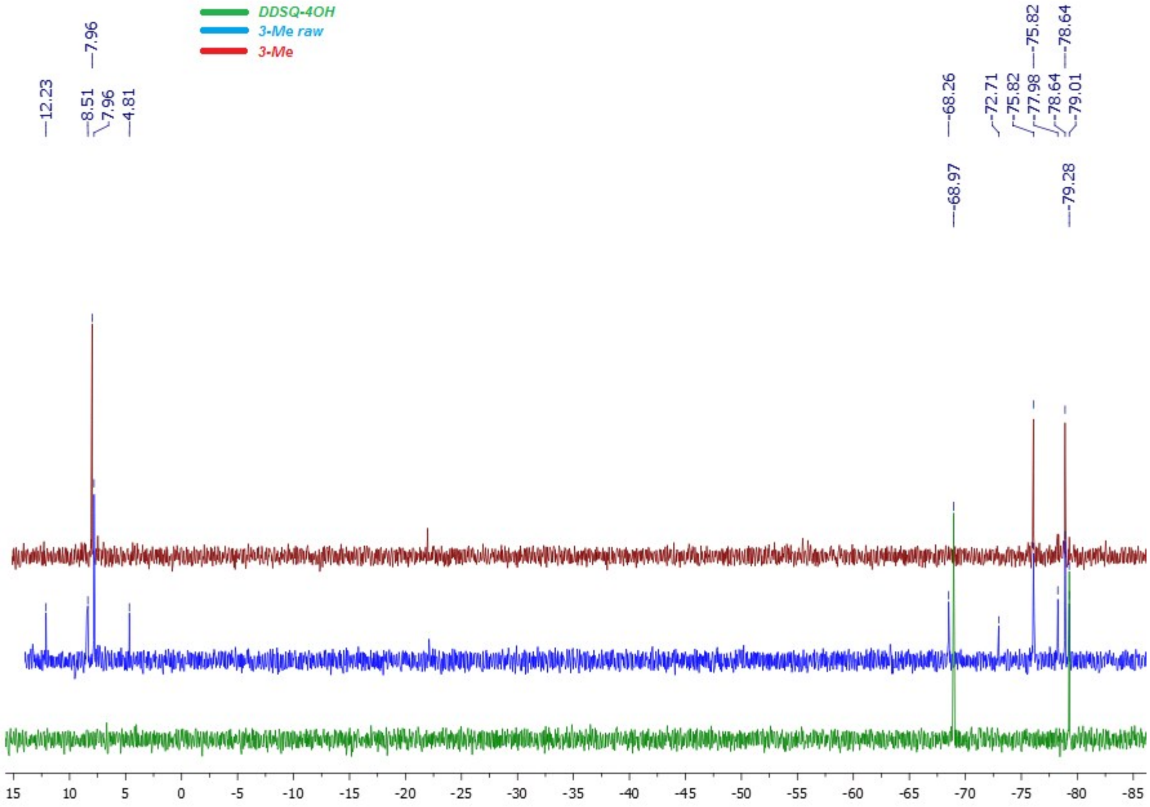
1.3 Synthetic procedures

General synthetic procedure for tetrafunctional DDSQ (1-4) compounds obtained *via* condensation reaction



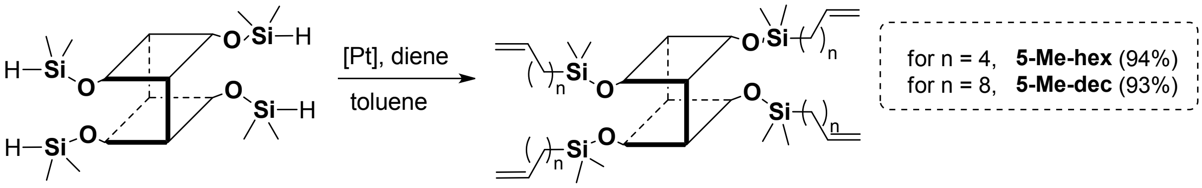
The synthesis was based on the hydrolytic condensation of DDSQ tetrasilanol (DDSQ-4OH) with respective chlorodiorganosilane carried out in the presence of triethylamine under argon atmosphere. Herein, a procedure for the synthesis of **1-Me** is presented. A Schlenk flask, placed on ice bath, was charged with dry THF (130 mL), DDSQ-4OH (2.12 g, 1.97 mmol), triethylamine (1.66 mL, 11.9 mmol), followed chlorodimethylsilane (0.94 mL, 9.31 mmol) introduced by dropping to the obtained solution. The reaction mixture became cloudy as a result of hydrochloride formation and consequently [$HNEt_3$]Cl was observed as a white solid. After one hour, the mixture was warmed to room temperature and kept at stirring for 23 hours. Then the post-reaction mixture was subjected to filtration in order to remove the formed triethylamine hydrochloride and the filtrate was evaporated under reduced pressure. The crude product was dissolved in DCM and precipitated in MeOH, to remove any possible hydrochloride residue. After evaporation of MeOH, **1-Me** was isolated as white solid, with 88% yield and subjected to spectroscopic and thermal analysis. For the synthesis of **3-Me**, the raw product was purified by chromatographic column (silica gel 60) using DCM : hexane 3:2 as an eluent.

Figure S4. ^{29}Si NMR stacked spectra of DDSQ-4OH, **3-Me raw** (sample from the reaction mixture) and **3-Me** purified by column chromatography.



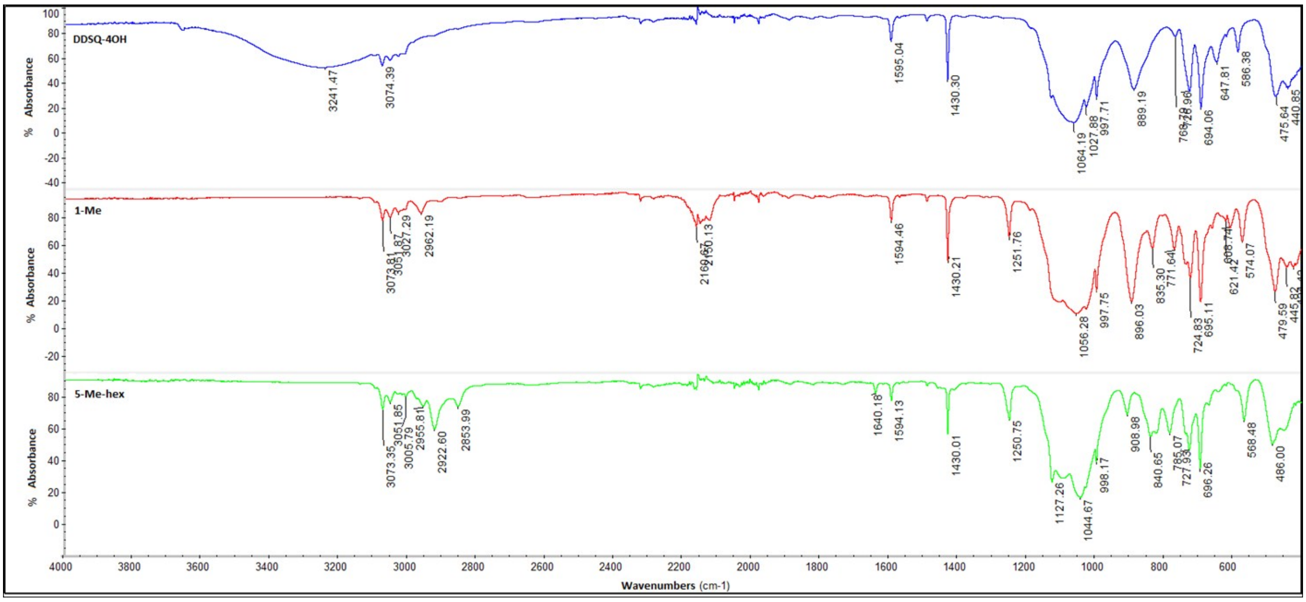
The additional signals (12.23, 8.51, 4.81ppm) most probably come from not fully substituted product **3-Me**, i.e. with one, two or even three remaining Si-OH groups. In this way the resonance lines of the FG are slightly shifted in comparison with the final, fully, i.e. tetra-substituted product 3-Me. In addition, there are more signals in the region of Si-O-Si core atoms (-68.26, -72.71, -77.98, -79.01), due to the abovementioned reasons (there is also visible signal at -68.26 ppm, deriving from the Si-OH of remaining tetrasilanol). The purified (after column chromatography) **3-Me** product spectrum reflects presence of the pure **3-Me** product with only 3 signals.

General synthetic procedure for tetrafunctional DDSQ (5) compounds obtained *via* hydrosilylation reaction of 1-Me with diene

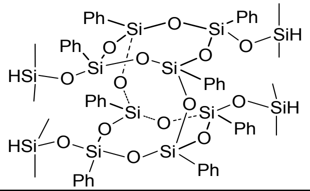
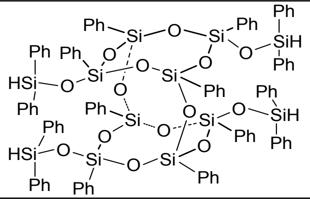
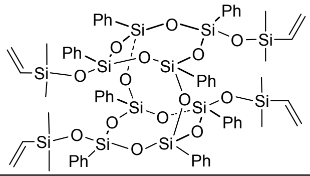
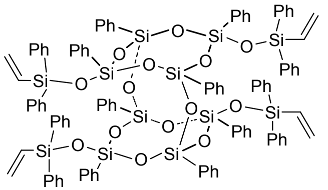
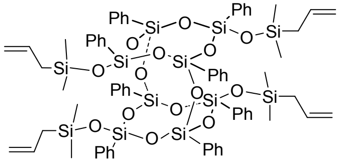
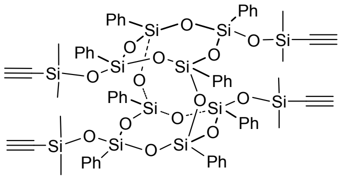
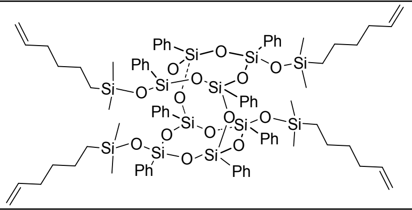
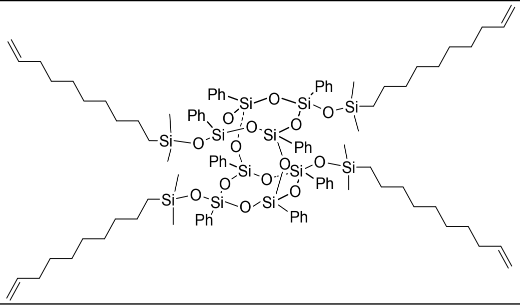


The procedure for the synthesis of **5-Me-hex** is described as an example. To a 50 mL glass reactor equipped with a condenser and magnetic stirrer, **1-Me** (0.508 g, 0.39 mmol) was placed in argon atmosphere along with toluene (1.5 mL), 1,5-hexadiene (0.955 mL, 7.81 mmol) and Pt(dvs) (1.78 uL 1.56×10^{-7} mol). The reaction mixture was kept at 65°C to obtain complete Si-H consumption (usually 20h). Then, the volatiles were evaporated under vacuum and the crude product was transferred onto chromatographic column (silica gel 60) using DCM : hexane 3:2 as an eluent. Evaporation of eluent gave an analytically pure sample with 94% yield.

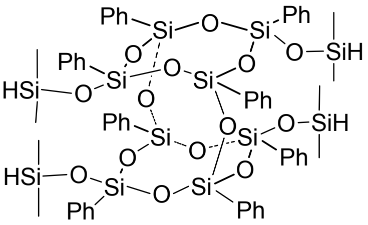
Figure S5. FT- IR spectra of DDSQ-4OH, **1-Me** and **5-Me-hex** after completion of hydrosilylation reaction.



2. Table of isolated compounds:

Structure	Compound Abbrev.	NMR spectra page:
	**1-Me**	S – 10 –
	**1-Ph**	S – 12 –
	**2-Me**	S – 14 –
	**2-Ph**	S – 16 –
	**3-Me**	S – 18 –
	**4-Me**	S – 20 –
	**5-Me-hex**	S – 22 –
	**5-Me-dec**	S – 24 –

3. Characterization data of the hydrosilylation products:



1-Me. White solid. Isolated Yield 89%. R_f=0.92.

^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 0.16 (d, 24H, $Si(CH_3)_2$), 4.83-4.87 (m, 4H, Si-H), 7.08-7.44 (m, 40H, Ph).

^{13}C NMR (101 MHz, $CDCl_3$, ppm): δ = 0.59 ($SiCH_3$), 127.44-127.51 (Ph), 129.85-130.11 (Ph), 131.46-132.90 (Ph), 133.99-134.23 (Ph).

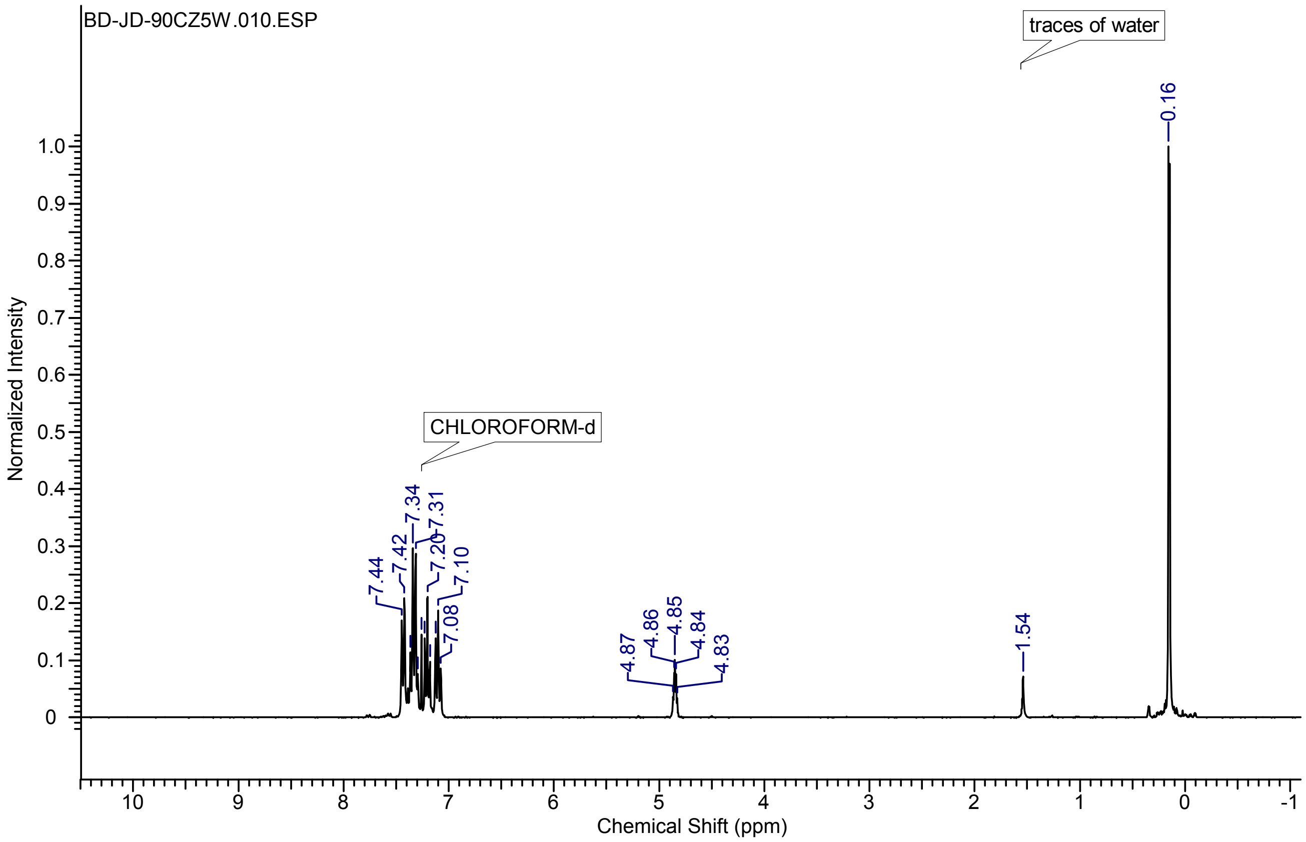
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = -3.88 ($-Si-(Me)_2H$), -78.96, -75.81(-O-Si-Ph).

IR (ATR, cm^{-1}): 3073.59, 3051.99 (C-H phenyl), 2962.47 (C-H), 2161.02, 2121.34 (Si-H), 1594.41, 1430.43 (C=C phenyl), 1251.61 (Si-C), 1120.32, 1055.81, 1026.65 (Si-O-Si), 997.92 (C-H phenyl).

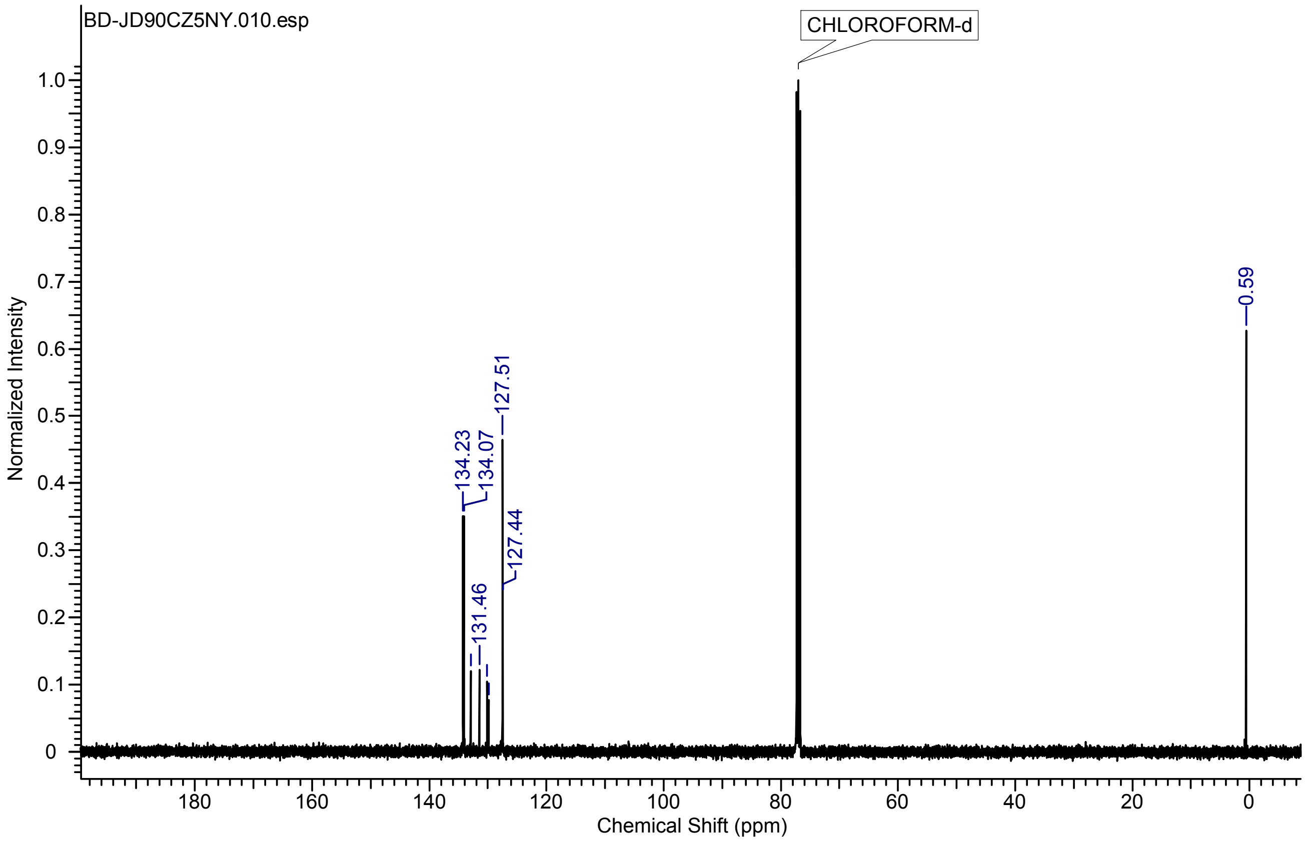
MALDI-ToF MS: Calcd. for $C_{56}H_{68}Na^+O_{14}Si_{12}$: *m/z* 1323.1732 [M + Na$^+$]. Found: 1323.1707.

EA: Anal. calcd for $C_{56}H_{68}O_{14}Si_{12}$ (%): C, 51.65; H, 5.26; found: C, 51.67; H, 5.28.

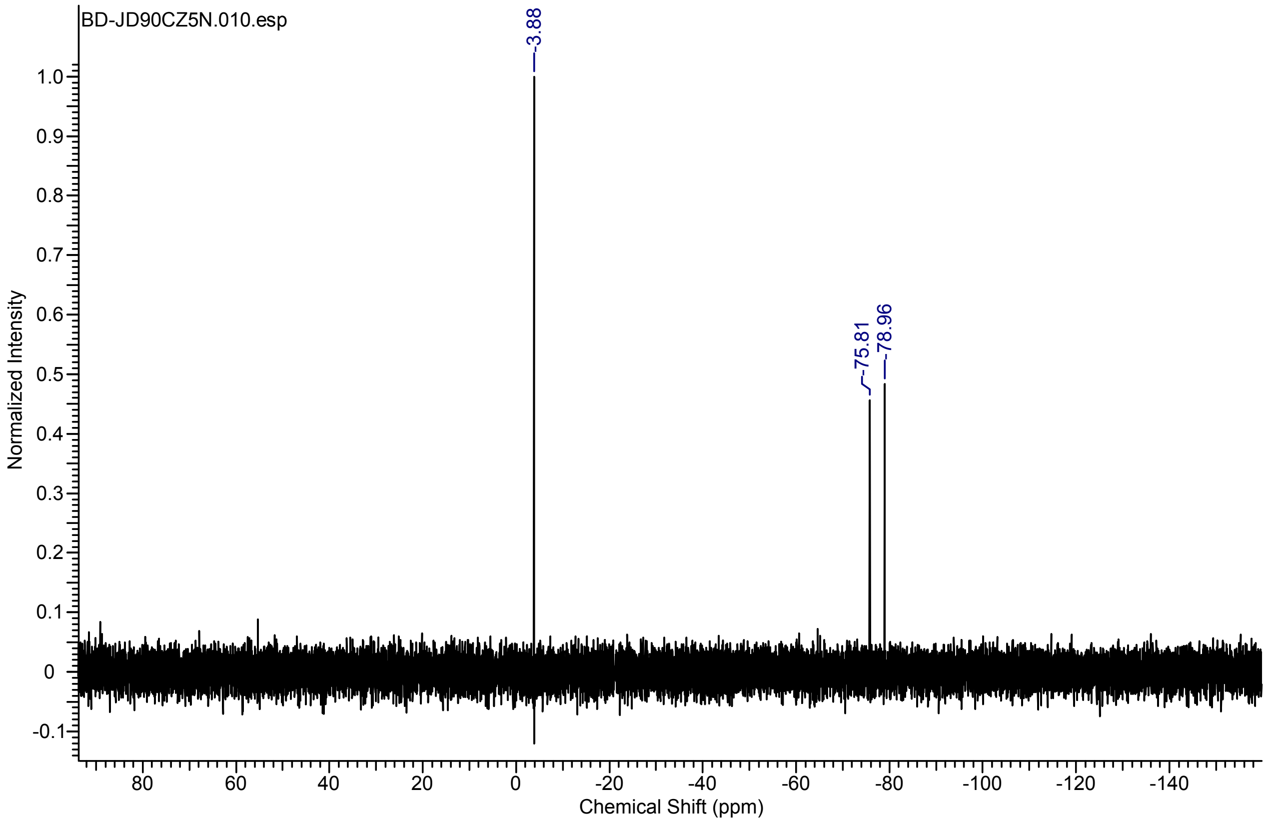
^{1}H NMR (300 MHz, $CDCl_3$)

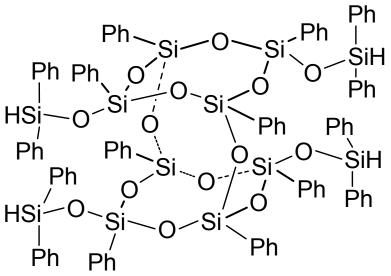


^{13}C *NMR (101 MHz,* $CDCl_3$*)*



^{29}Si *NMR (79.5 MHz,* $CDCl_3$*)*





1-Ph. White solid. Isolated Yield 88%. R_f=0.83.

^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 5.53 (s, 4H, Si-H), 6.96-7.70 (m, 80H, Ph).

^{13}C NMR (101 MHz, $CDCl_3$, ppm): δ = 127.33-127.81 (Ph), 130.09-130.39 (Ph), 134.02-134.57 (Ph).

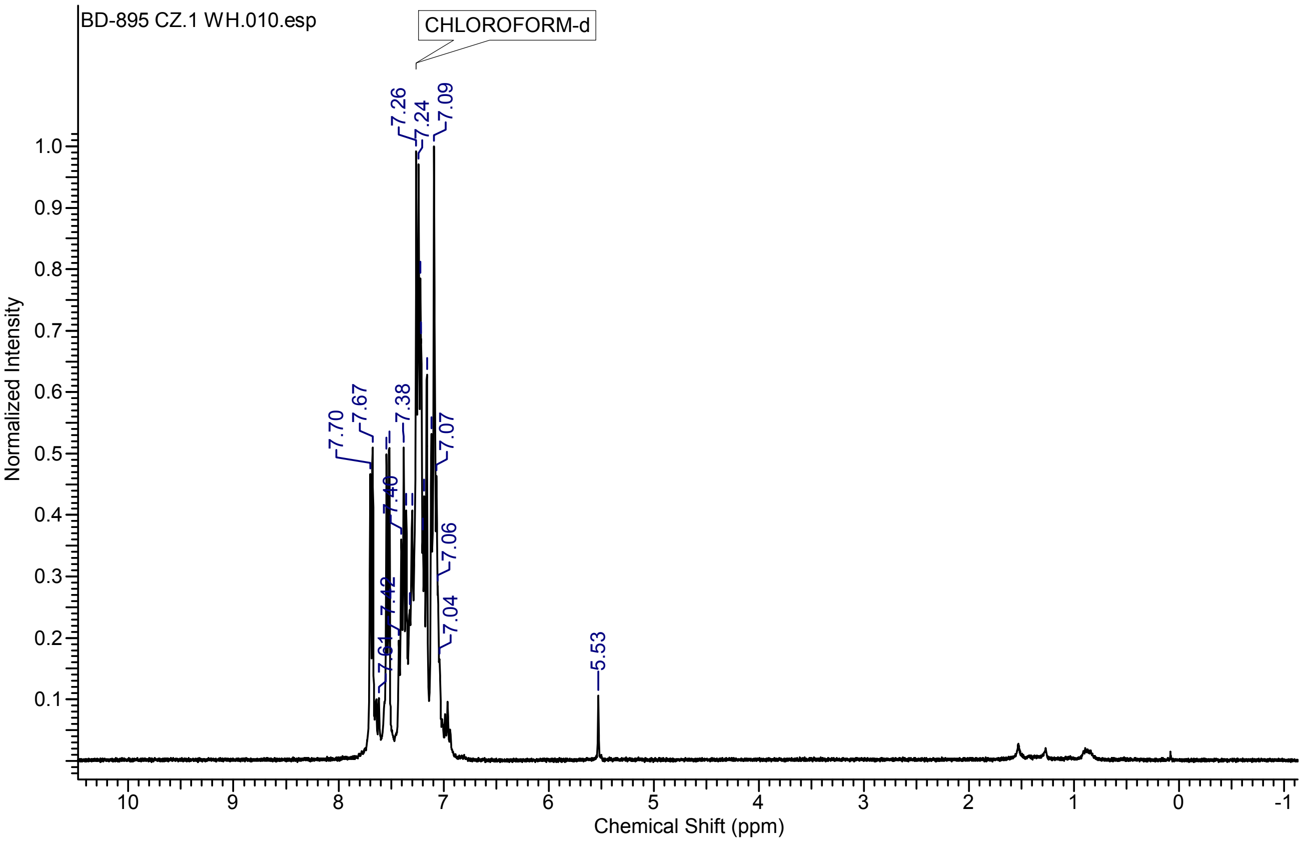
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = -45.44 (-Si-$(Ph)_2$-H), -78.37, -75.12 (-Si-Ph).

IR (ATR, cm^{-1}): 3071.62, 3049.92, 3026.37 (C-H phenyl), 2151.84 (Si-H), 1593.38, 1429.35 (C=C phenyl), 1078.34, 1027.92 (Si-O-Si), 997.86 (C-H phenyl).

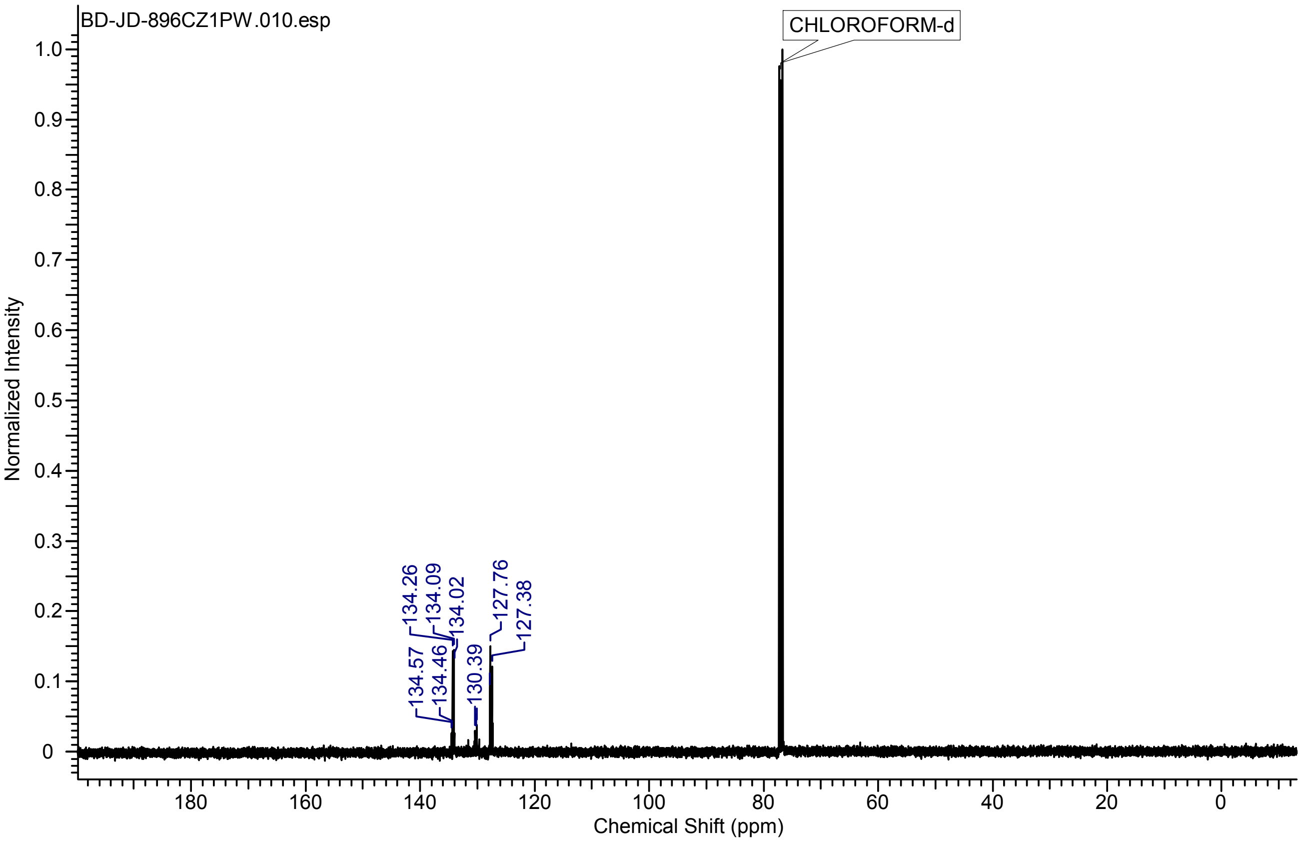
MALDI-ToF MS: Calcd. for $C_{96}H_{84}Na^+O_{14}Si_{12}$: *m/z* 1819.2984 [M + Na$^+$]. Found: 1819.3063.

EA: Anal. calcd for $C_{96}H_{84}O_{14}Si_{12}$ (%): C, 64.10; H, 4.71; found: C, 64.11; H, 4.72.

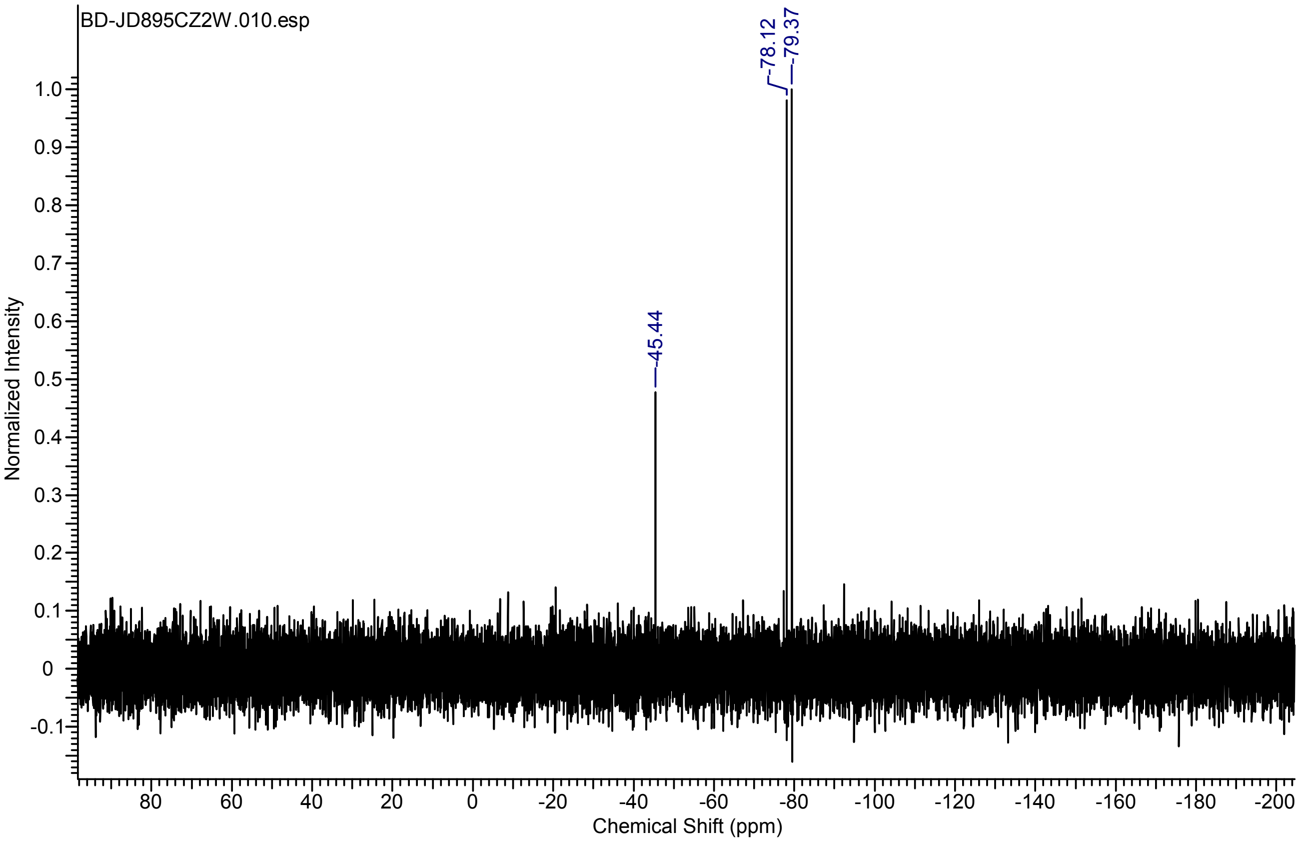
^{1}H NMR (300 MHz, $CDCl_3$)

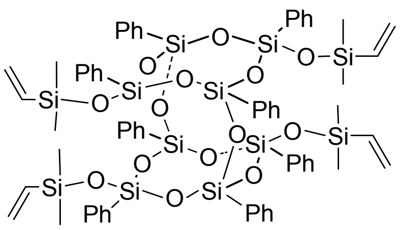


^{13}C NMR (101 MHz, CDCl$_3$)



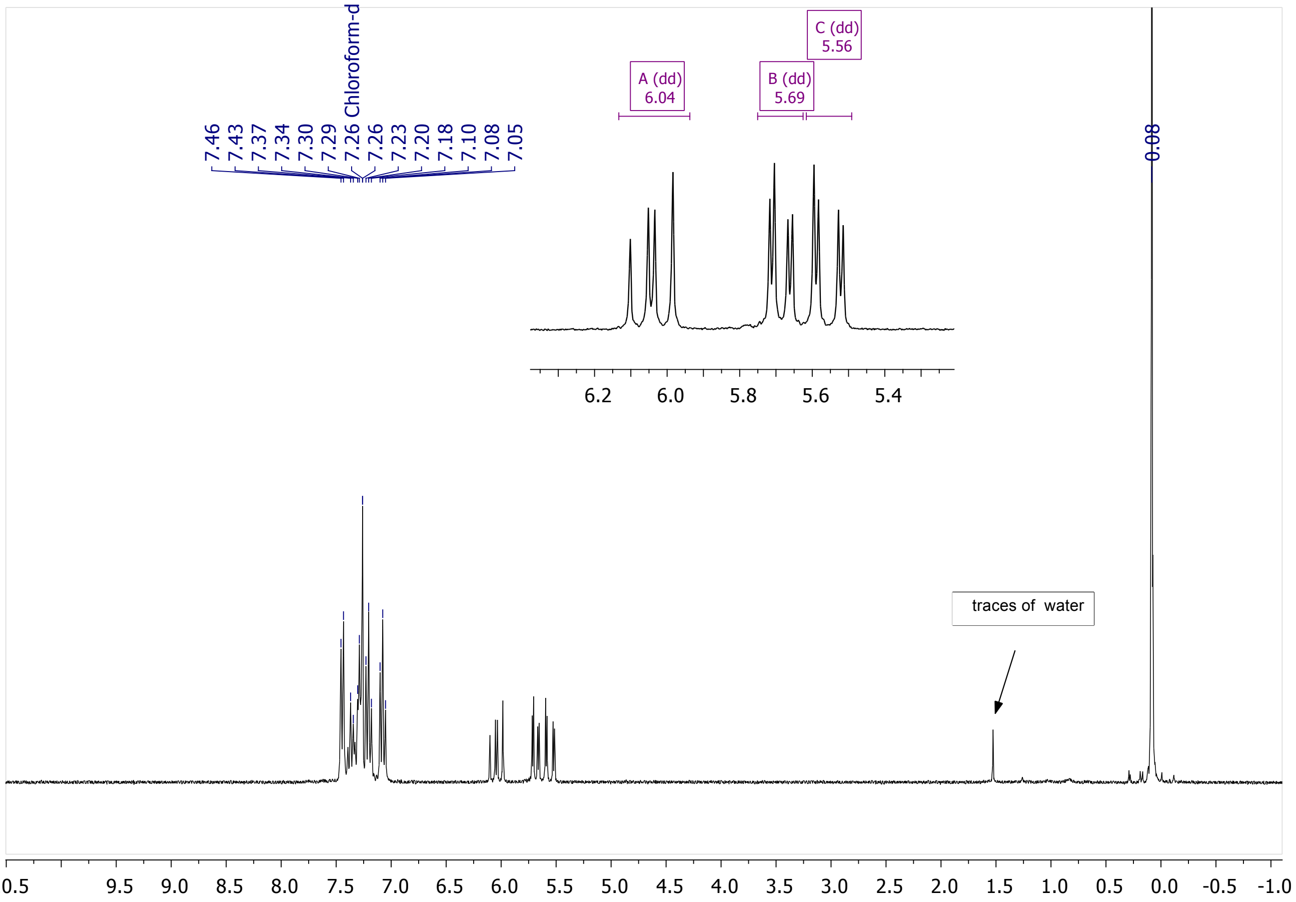
^{29}Si NMR (79.5 MHz, CDCl$_3$)



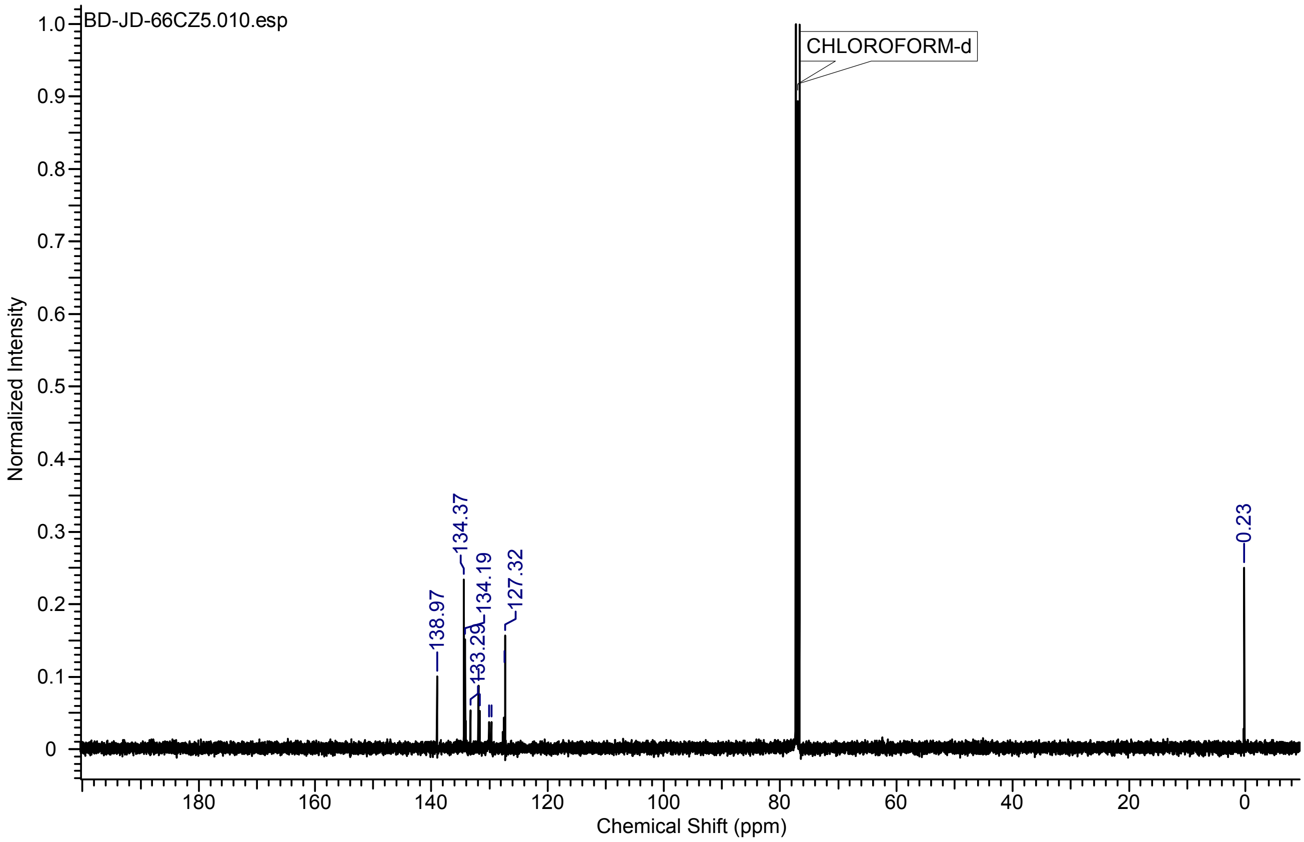


2-Me. White solid. Isolated Yield 95%. R_f=0.95.
^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 0.08 (s,24H,$SiCH_3$), 5.56 (dd, $^3J_{H-H}$ = 20.3, 3.8 Hz, 4H), 5.69 (dd, $^3J_{H-H}$ = 14.9, 3.8 Hz, 4H), 6.04 (dd, $^3J_{H-H}$ = 20.4, 14.9 Hz, 4H), 7.05-7.46 (m, 40H, Ph).
^{13}C NMR (101 MHz, $CDCl_3$, ppm): δ = 0.23 ($SiCH_3$), 127.32-127.50 (Ph), 129.69-130.04 (Ph), 131.63 (Ph), 131.87 (H_2C=CH-), 133.29 (Ph), 134.19-134.37 (Ph), 138.97 (H_2C=CH-).
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = -0.84 ($Si(Me)_2Vi$), -75.83,-78.86 (-Si-Ph).
IR (ATR, cm-1): 3073.04, 3050.94 (C-H phenyl), 2960.09 (C-H), 1594.03, 1430.10 (C=C phenyl), 1251.44 (Si-C), 1127.80, 1047.84 (Si-O-Si), 998.59 (C-H phenyl).
MALDI-ToF MS: Calcd. for $C_{64}H_{76}Na^+O_{14}Si_{12}$: *m/z* 1427.2358 [M + Na^+]. Found: 1427.2550.
EA: Anal. calcd for $C_{64}H_{76}O_{14}Si_8$ (%):C ,54.66; H, 5.45; found: C, 54.67; H, 5.47.

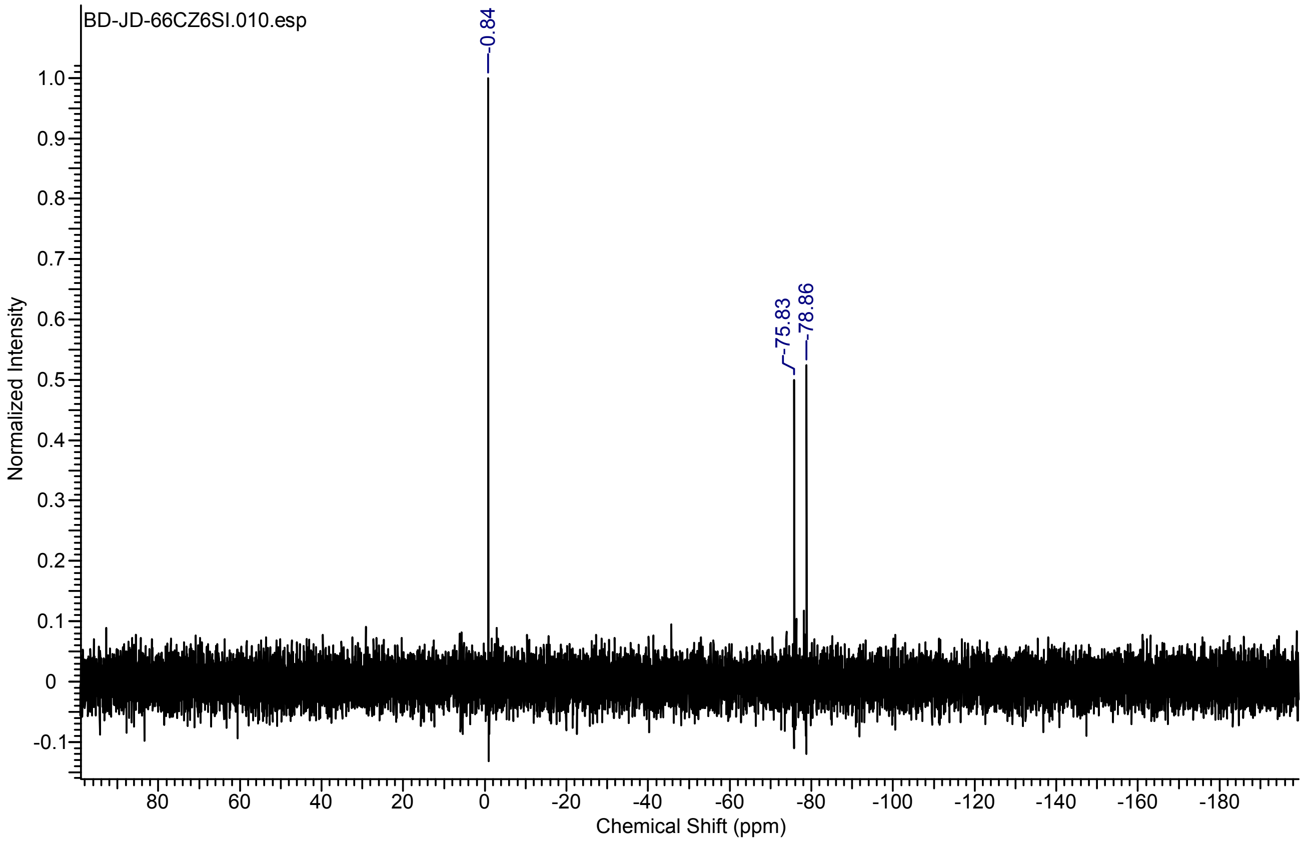
^{1}H NMR (300 MHz, $CDCl_3$)

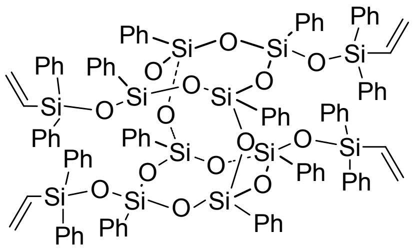


^{13}C NMR (101 MHz, CDCl$_3$)



^{29}Si NMR (79.5 MHz, CDCl$_3$)





2-Ph. White solid. Isolated Yield 79%. R_f=0.89.

^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 5.88 (dd, $^3J_{H\text{-}H}$ = 20.3, 3.6 Hz, 4H), 6.09 (dd, $^3J_{H\text{-}H}$ = 14.8, 3.6 Hz, 4H), 6.32 (dd, $^3J_{H\text{-}H}$ = 20.2, 14.9 Hz, 4H), 7.09-7.77 (m, 80H, Ph).

^{13}C NMR (101MHz, $CDCl_3$, ppm): δ = 127.42-127.86 (Ph), 129.60-130.60 (Ph), 132.63 (H_2C=CH-), 134.07 (Ph), 134.80-135.00 (Ph), 136.69 (H_2C=CH-).

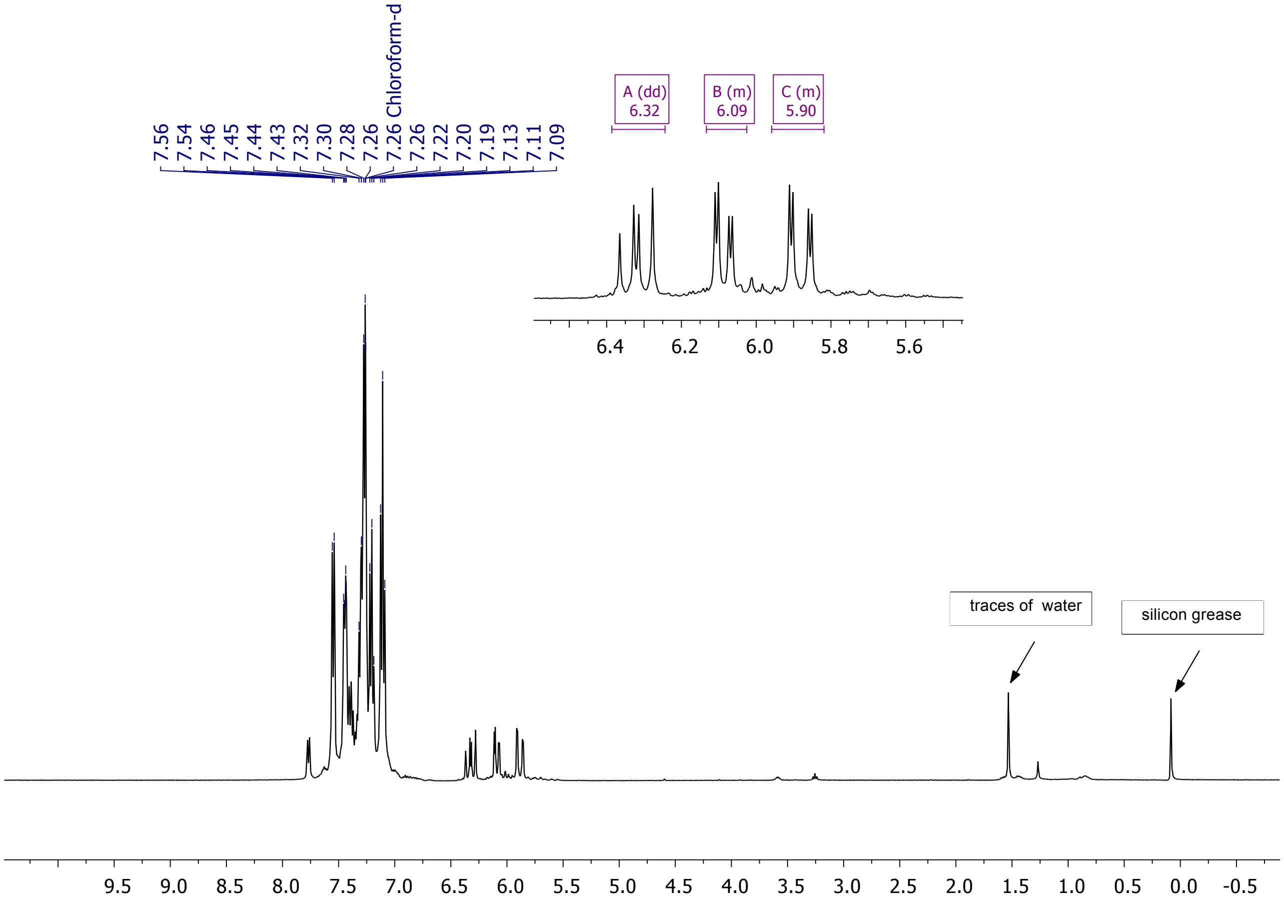
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = -20.33 ($Si(Ph)_2Vi$), -76.21, -77.74 (-Si-Ph).

IR (ATR, cm^{-1}): 3071.84, 3050.34, 3007.22 (C-H phenyl), 1594.08, 1429.64 (C=C phenyl), 1107.12, 1070.97, 1028.26 (Si-O-Si), 997.85 (C-H phenyl).

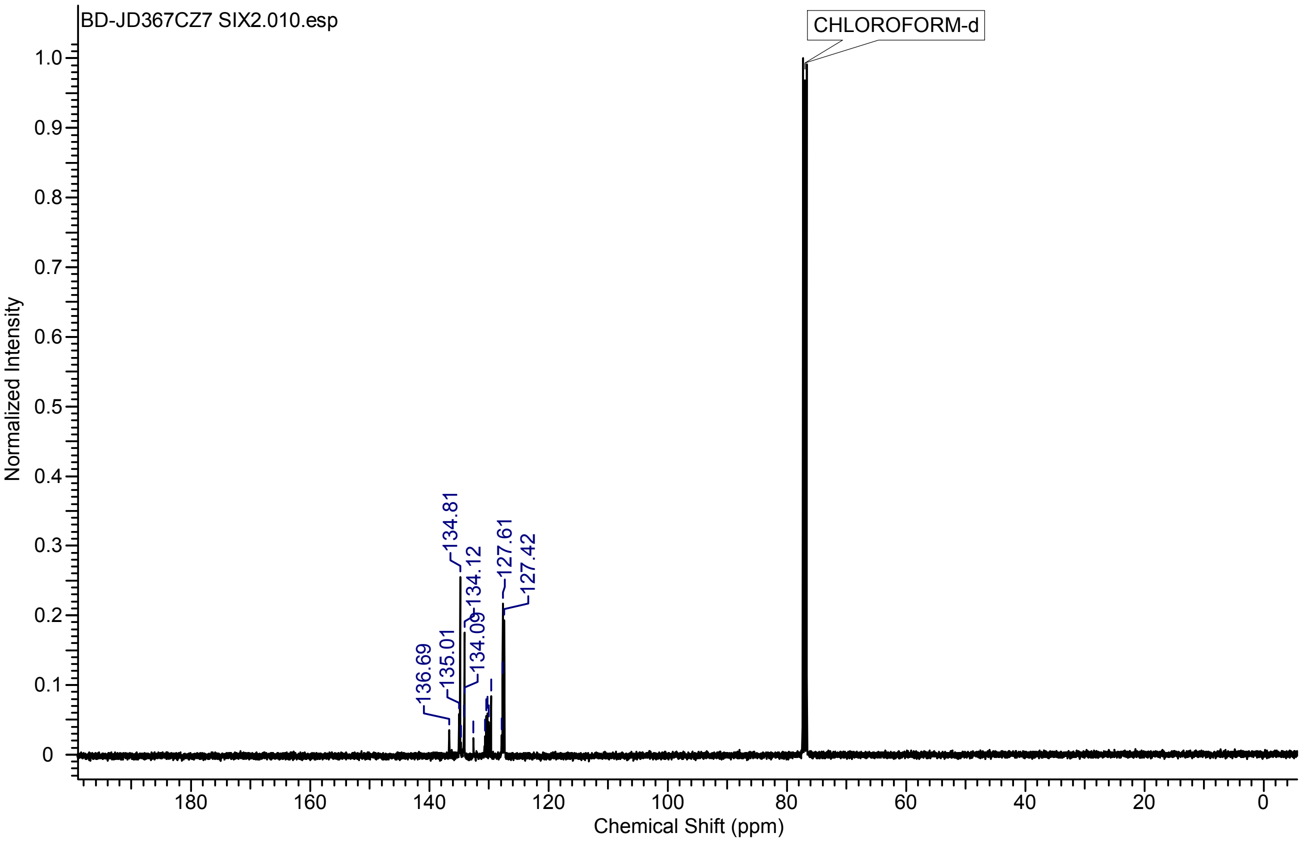
MALDI-ToF MS: Calcd. for $C_{104}H_{92}Na^+O_{14}Si_{12}$: *m/z* 1923.3610 [M + Na^+]. Found: 1923.3673.

EA: Anal. calcd for $C_{104}H_{92}O_{14}Si_{12}$ (%): C, 65.64; H, 4.87; found: C, 65.67; H, 4.89.

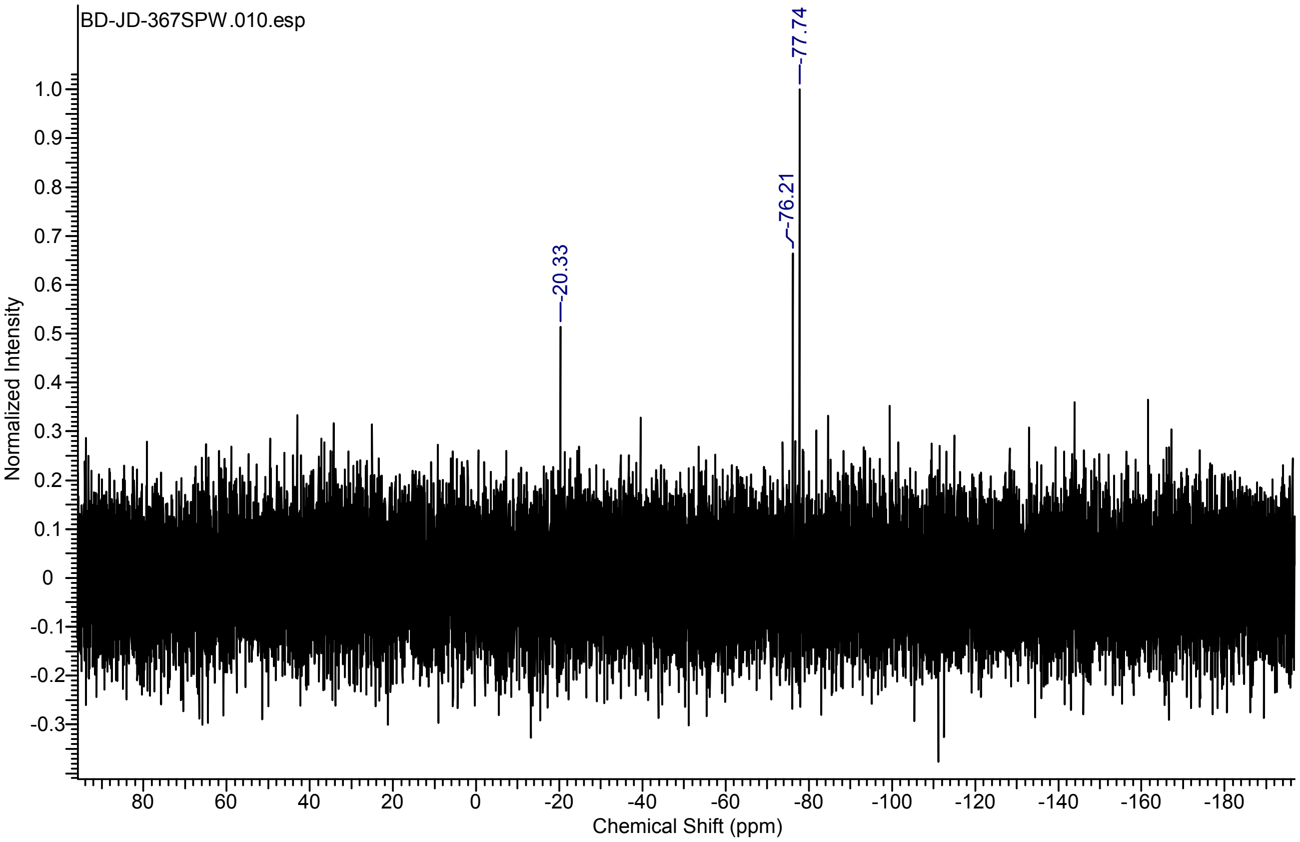
^{1}H NMR (400 MHz, $CDCl_3$)

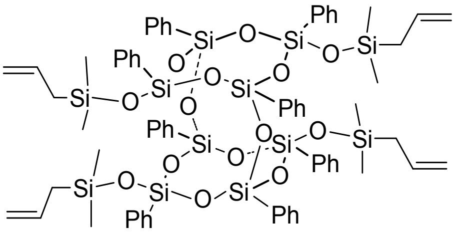


^{13}C NMR (101 MHz, $CDCl_3$)



^{29}Si NMR (79.5 MHz, $CDCl_3$)





3-Me. Waxy white solid. Isolated Yield 68%. R_f=0.85.

^{1}H NMR (300 MHz, CDCl$_3$, ppm): δ = 0.02 (s, 24H, Si(CH$_3$)$_2$-), 1.50-1.52 (m, 8H,-CH$_2$-), 4.58-4.63 (m, 8H, H$_2$C=CH-), 5.54-5.58 (m,4H, -CH=CH$_2$), 7.10-7.46 (m, 40H, Ph).

^{13}C NMR (101 MHz, CDCl$_3$, ppm): δ = -0.36 (SiCH$_3$), 26.14 (-CH$_2$-), 113.33 (H$_2$C=CH-), 127.46-127.53 (Ph), 129.85-130.20 (Ph), 131.43 (Ph), 133.16 (Ph), 134.05 (H$_2$C=CH-), 134.20-134.34 (Ph).

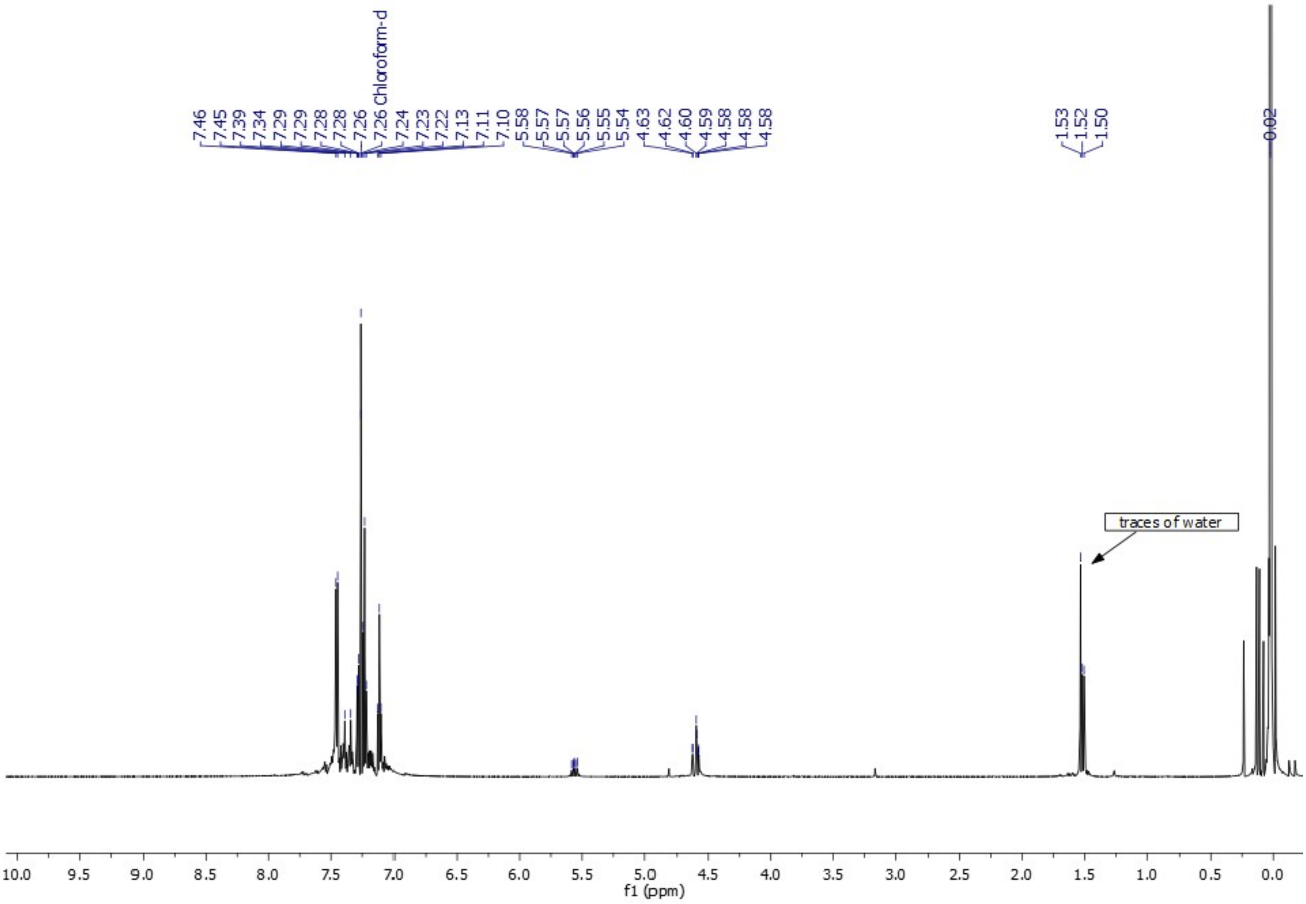
^{29}Si NMR (79.5 MHz, CDCl$_3$, ppm): δ = 7.96 (Si(Me)$_2$-), -75.84,-78.66 (-Si-Ph-).

IR (ATR, cm^{-1}): 3073.94, 3052.73 (C-H phenyl), 2961.31 (C-H), 1630.27 (C=C), 1594.71, 1430.22 (C=C phenyl), 1255.20 (Si-C), 1127.65, 1026.82 (Si-O-Si), 997.88 (C-H phenyl).

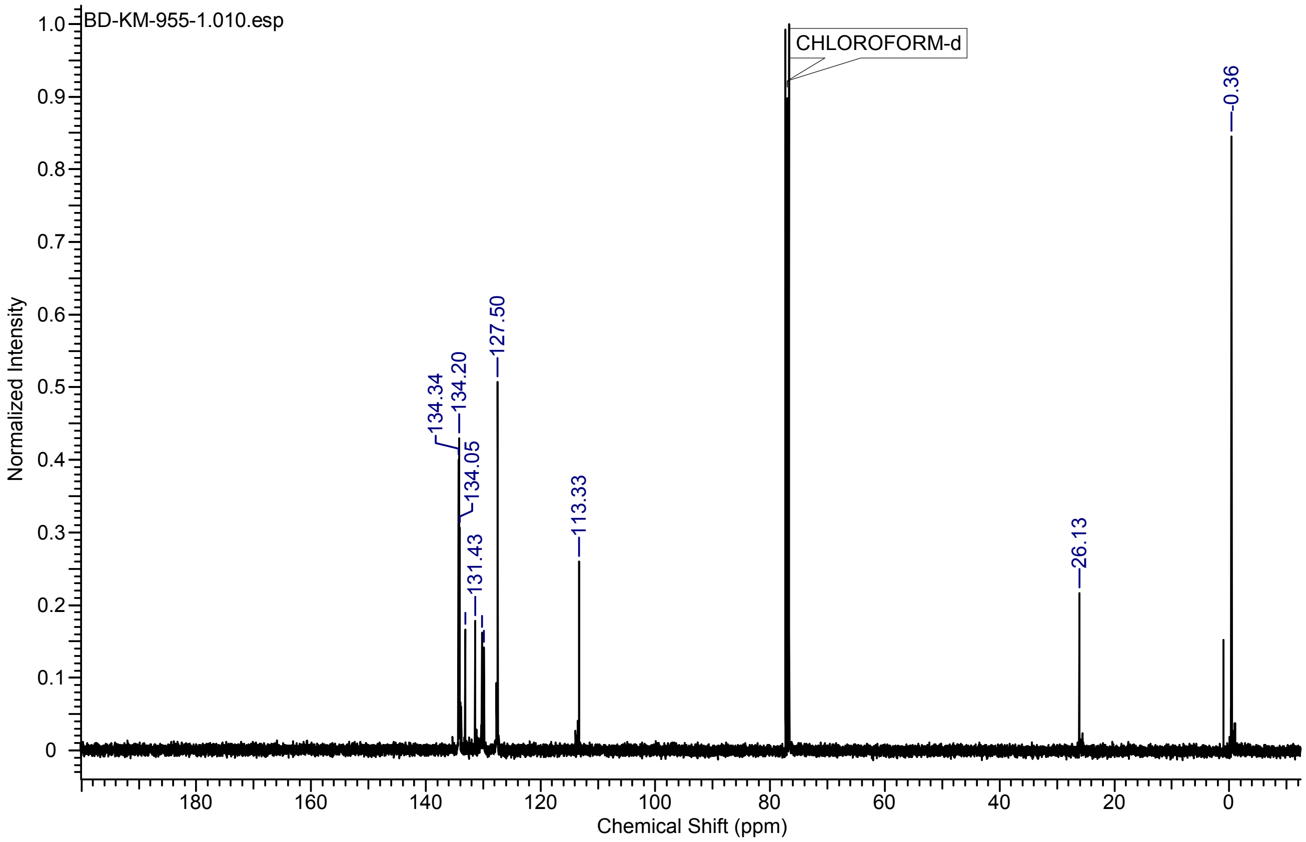
MALDI-ToF MS: Calcd. for $C_{68}H_{84}Na^+O_{14}Si_{12}$: *m/z* 1483.2984 [M + Na$^+$]. Found: 1483.2958.

EA: Anal. calcd for $C_{68}H_{84}O_{14}Si_{12}$ (%):C, 55.85, H, 5.79; found: C, 55.89; H, 5.83.

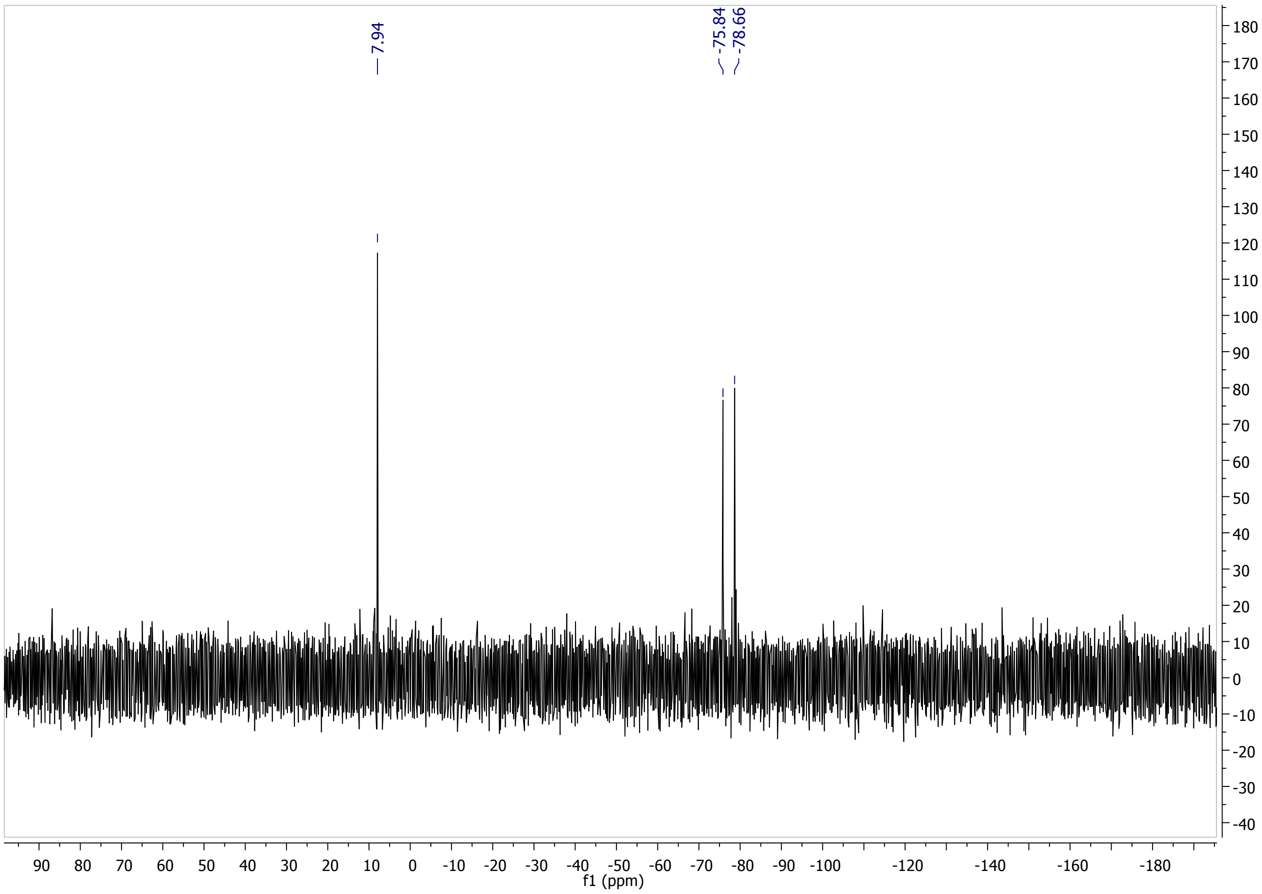
^{1}H NMR (300 MHz, CDCl$_3$)



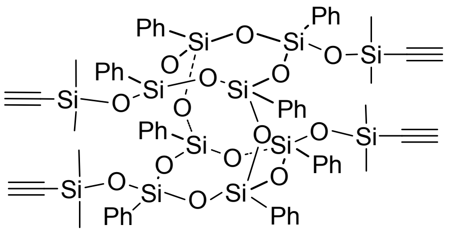
^{13}C NMR (101 MHz, CDCl$_3$)



^{29}Si NMR (79.5 MHz, CDCl$_3$)



7.94
-75.84
-78.66
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180
f1 (ppm)



4-Me. White solid. Isolated Yield 71%. R_f=0.78.

^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 0.24 (s,24H, $Si(CH_3)_2$),2.05 (s,4H,-CC-H), 7.10-7.53 (m,40H,Ph).

^{13}C NMR (101 MHz, $CDCl_3$, ppm): δ = 2.06 ($SiCH_3$), 88.87, 92.68 (-CC-H), 127.29-127.49 (Ph), 129.90-130.11 (Ph), 131.46-132.90 (Ph), 133.99-134.23 (Ph).

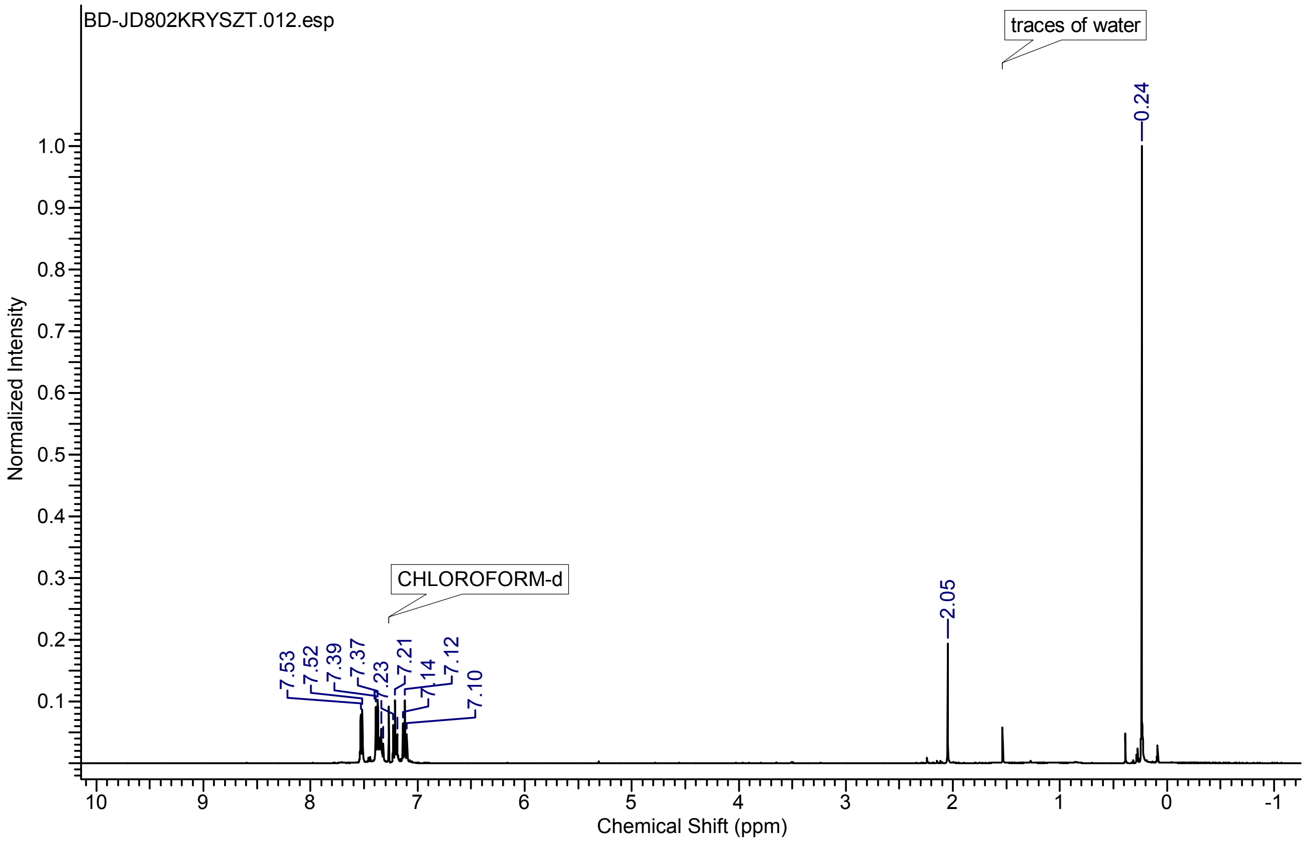
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = -16.45 (Si-$(CH_3)_2$), -75.84, -78.64 (-Si-Ph-).

IR (ATR, cm^{-1}): 3279.85 (-CC-H), 3075.59 (C-H phenyl), 2967.66 (C-H), 2036.57 (-CC-H), 1594.29, 1430.29 (C=C phenyl), 1257.17 (Si-C), 115.94, 1044.32 (Si-O-Si), 998.39 (C-H phenyl).

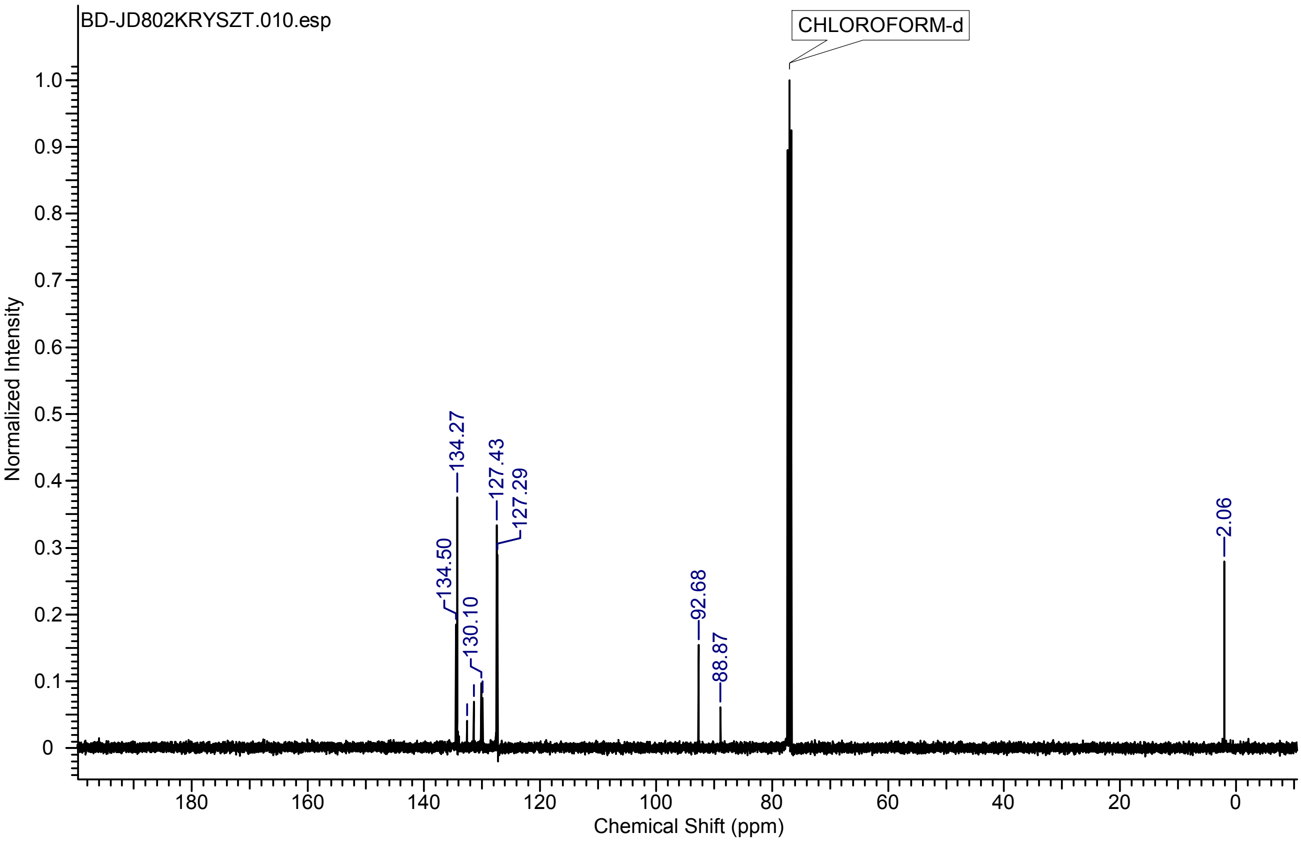
MALDI-ToF MS: Calcd. for $C_{64}H_{68}Na^+O_{14}Si_{12}$: *m/z* 1419.1732 [M + Na$^+$]. Found: 1419.1677.

EA: Anal. calcd for $C_{64}H_{68}O_{14}Si_{12}$ (%): C, 54.98; H,4.90; found: C, 55.00; H, 4.92.

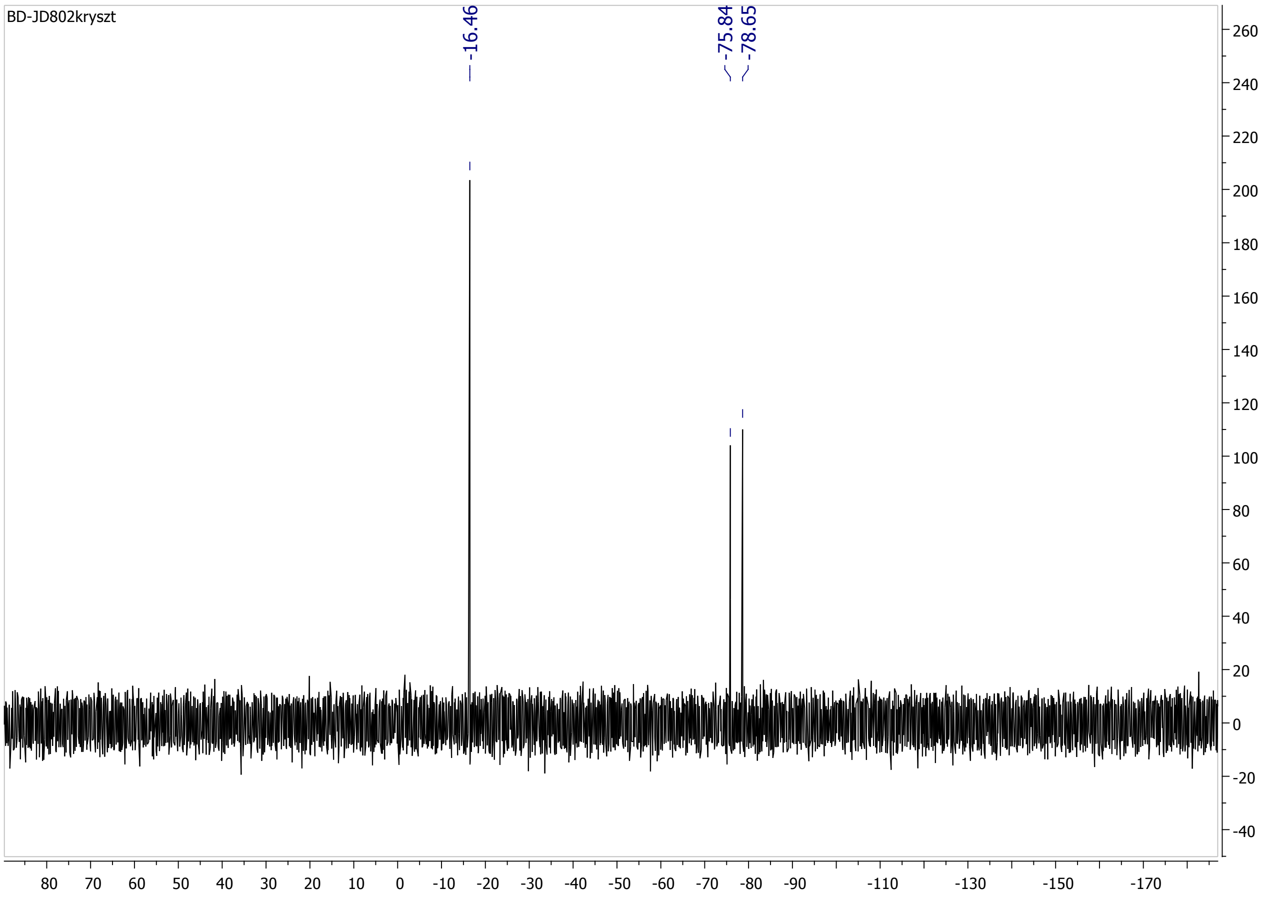
^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (101 MHz, $CDCl_3$)



^{29}Si NMR (79.5 MHz, $CDCl_3$)


BD-JD802kryszt
-16.46
-75.84
-78.65
260
240
220
200
180
160
140
120
100
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60
40
20
0
-20
-40
80
70
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50
40
30
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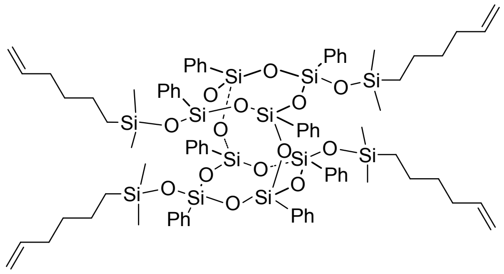
-170



5-Me-hex. Waxy, white solid. Isolated Yield 94%. R_f=0.85.

^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = -0.01 (s, 24H, $Si(CH_3)_2$-), 0.43-0.48 (m, 8H, -CH_2-), 1.03-1.07 (m, 16H, -CH_2-CH_2-), 1.72-1.74 (m, 8H, -CH_2-), 5.58-5.67 (m, 4H, H_2C=CH-), 4.81-4.86 (m,8H, -CH=CH_2), 7.05-7.49 (m, 40H, Ph).

^{13}C NMR (101 MHz, $CDCl_3$, ppm): δ = 0.12 ($SiCH_3$), 18.03 (-H_2C-), 22.57 (-H_2C-), 32.48 (-H_2C-), 33.28 (-H_2C-), 113.78 (H_2C=CH-), 127.40-127.51 (Ph), 129.71-130.10 (Ph), 133.55 (Ph), 134.10-134.36 (Ph), 139.17 (H_2C=CH).

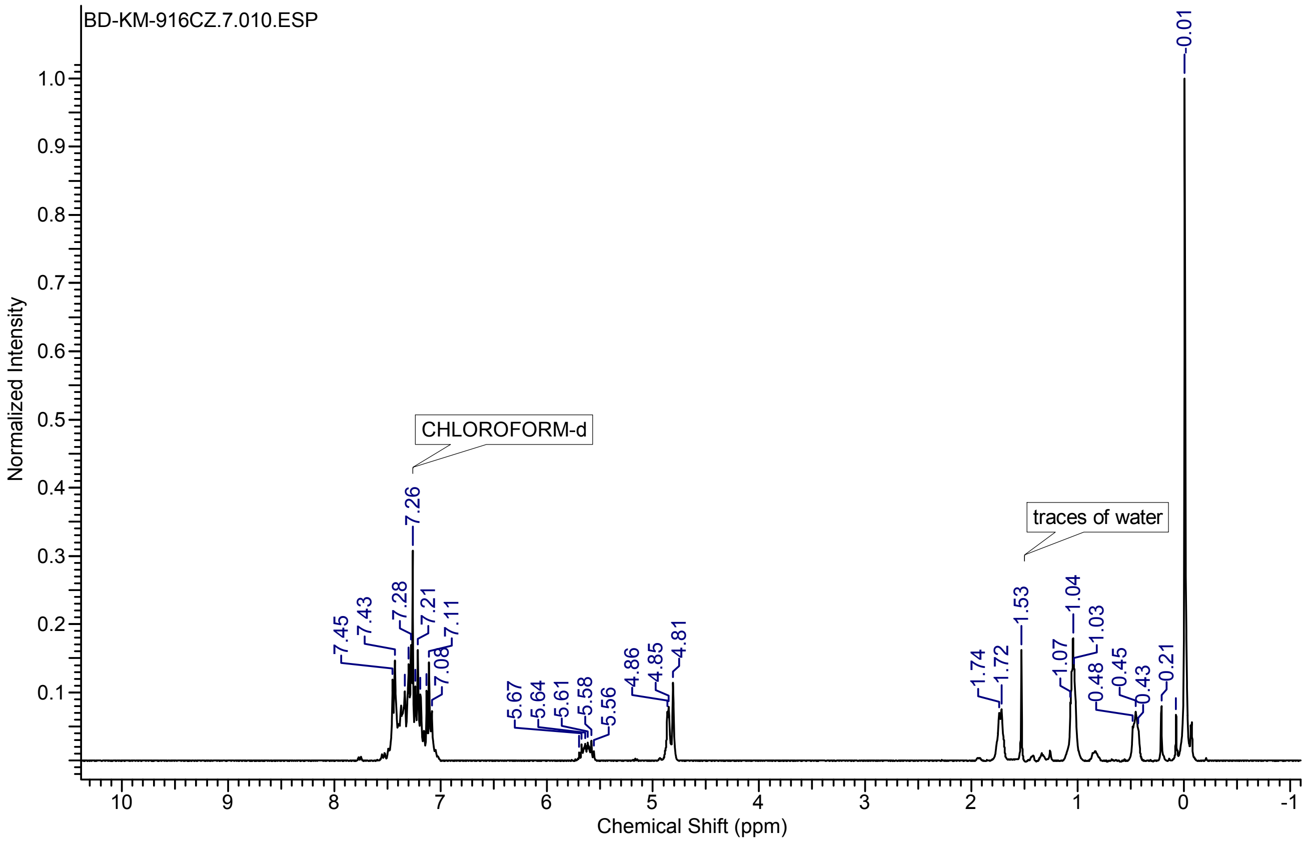
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = 10.95 (-$Si(Me)_2$-),-76.13, -78.77 (-Si-Ph-).

IR (ATR, cm^{-1}): 3073.35. 3051.85 (C-H phenyl), 2922.60, 2853.99 (C-H), 1640.18 (C=C), 1594.13, 1430.01 (C=C phenyl), 1250.75 (Si-C), 1127.26, 1044.67 (Si-O-Si), 998.17 (C-H phenyl).

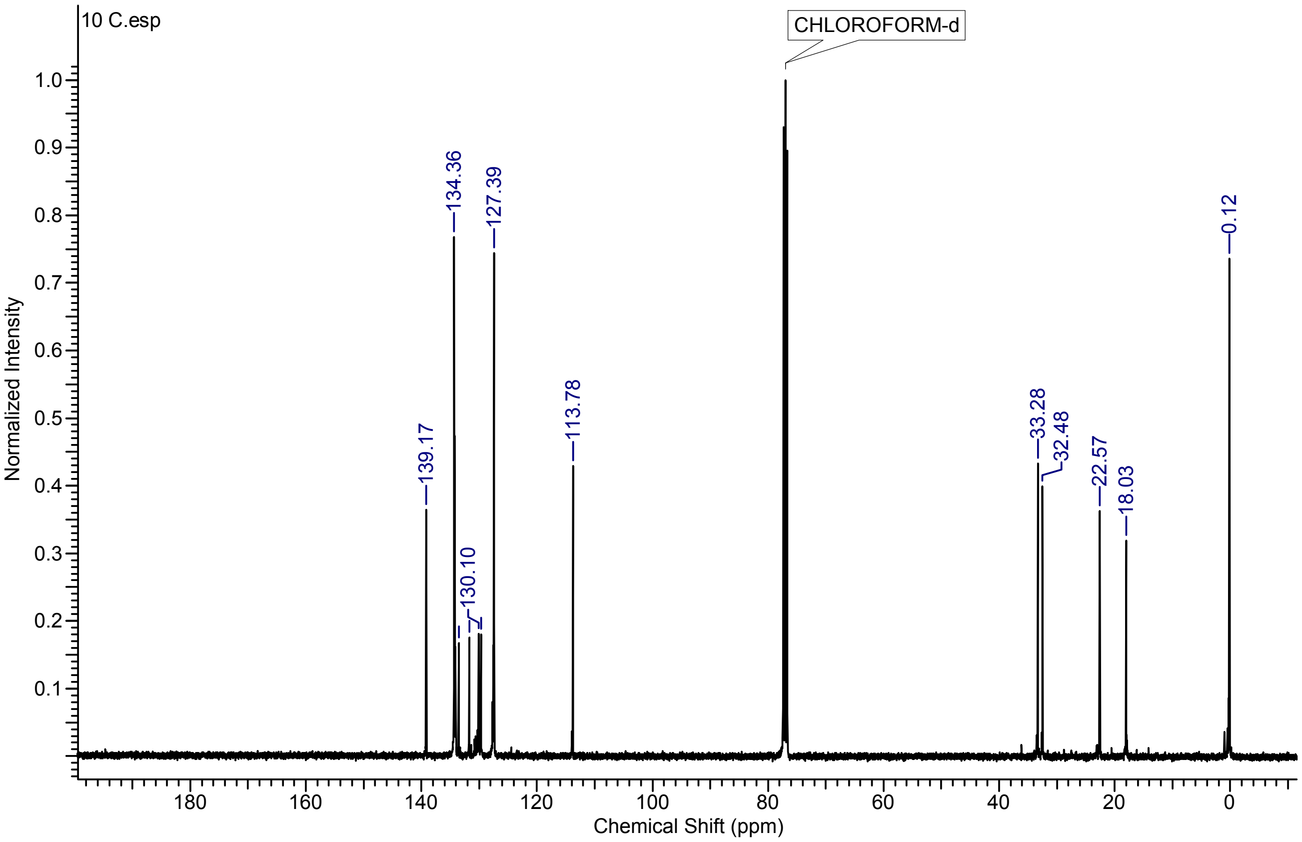
MALDI-ToF MS: Calcd. for $C_{80}H_{108}Na^+O_{14}Si_{12}$: *m/z* 1651.4862 [M + Na^+]. Found: 1651.4849.

EA: Anal. calcd for $C_{80}H_{108}O_{14}Si_{12}$ (%): C, 58.92; H,6.68; found: C, 58.93; H, 6.71.

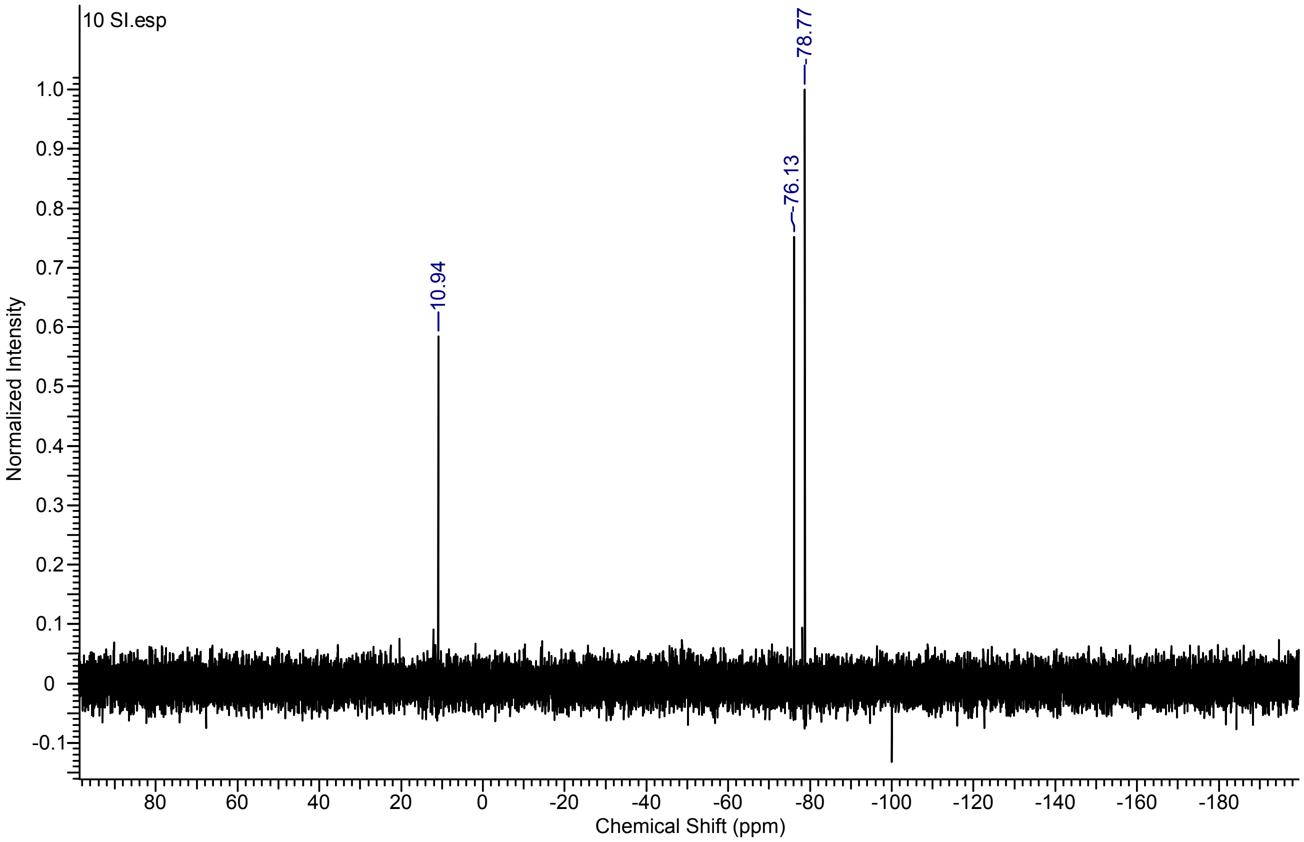
^{1}H NMR (400 MHz, $CDCl_3$)

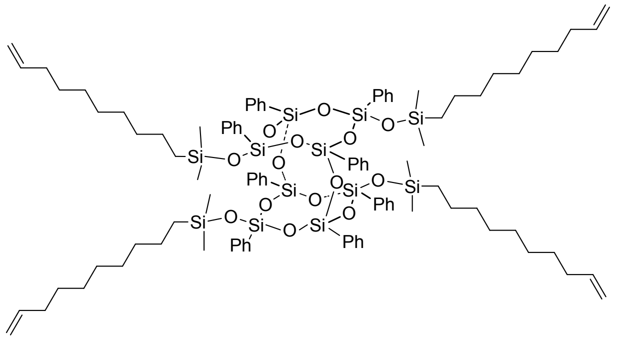


^{13}C NMR (101 MHz, $CDCl_3$)



^{29}Si NMR (79.5 MHz, $CDCl_3$)





5-Me-dec. Waxy, white solid. Isolated Yield 93%. R_f=0.86.

^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 0.03 (s, 24H, $Si(CH_3)_2$-), 0.41-0.46 (m, 8H, $Si-CH_2$-), 0.95-1.32 (m, 48H, $-CH_2$-), 1.95-2.03 (m, 8H, $-CH_2$-), 4.89-4.99 (m, 8H, $H_2C=CH$-), 5.72-5.85 (m, 4H, $-CH=CH_2$), 7.07-7.44 (m, 40H, Ph).

^{13}C NMR (101 MHz, $CDCl_3$, ppm): δ = 0.14 ($SiCH_3$), 18.24 ($-H_2C$-), 23.06 ($-H_2C$-), 28.97 ($-H_2C$-), 29.15 ($-H_2C$-), 29.37 ($-H_2C$-), 29.44 ($-H_2C$-), 33.40 ($-H_2C$-), 33.84 ($-H_2C$-), 114.06 ($H_2C=CH$-), 127.34-127.73 (Ph), 129.64-130.02 (Ph), 131.78 (Ph), 133.66 (Ph), 134.10-134.38 (Ph), 139.27 ($H_2C=CH$-).

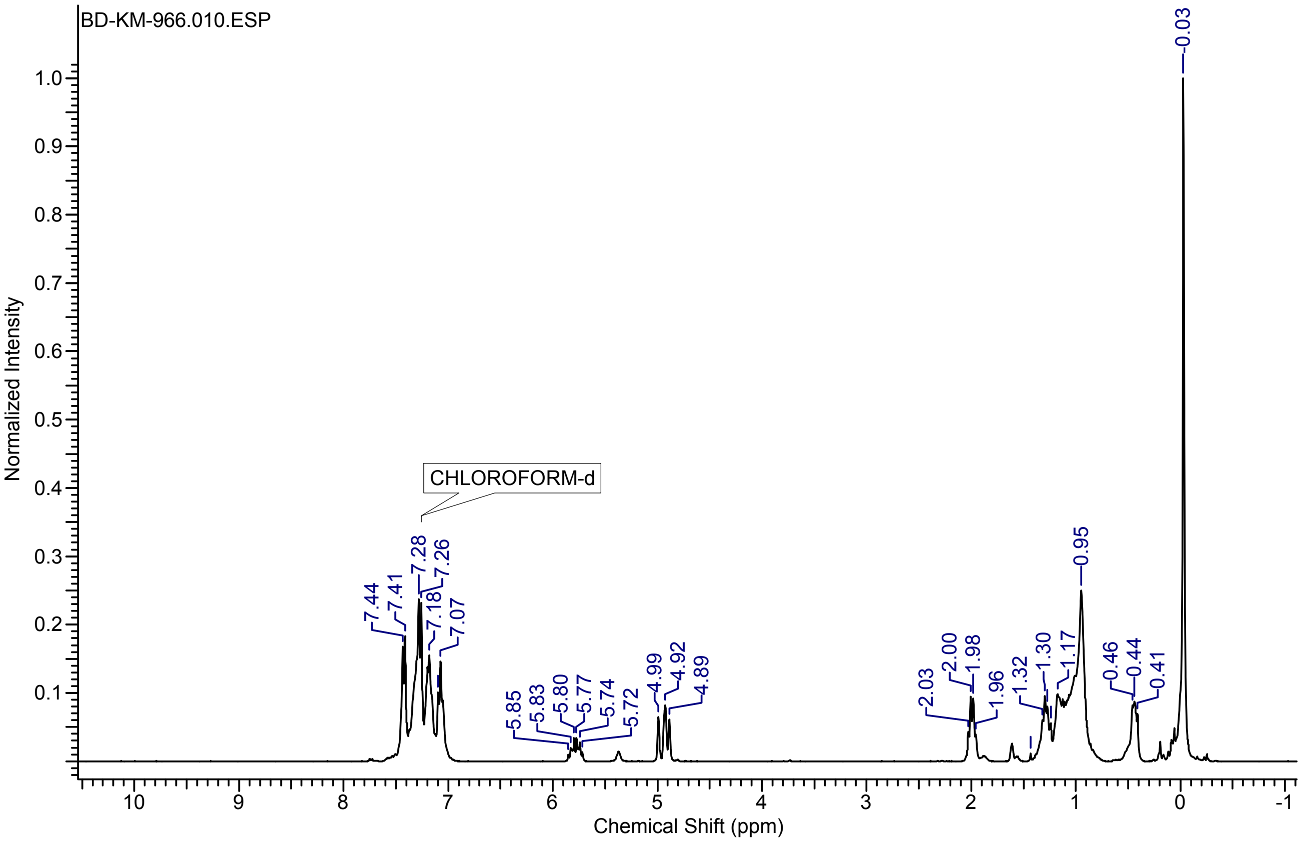
^{29}Si NMR (79.5 MHz, $CDCl_3$, ppm): δ = 11.01 ($-Si(Me)_2$-), -76.15, -78.77 (-Si-Ph).

IR (ATR, cm^{-1}): 3073.25, 3051.36 (C-H phenyl), 2922.86, 2852.96 (C-H), 1640.60 (C=C), 1594.32, 1429.95 (C=C phenyl), 1250.45 (Si-C), 1127.50, 1047.51 (Si-O-Si), 998.18 (C-H phenyl).

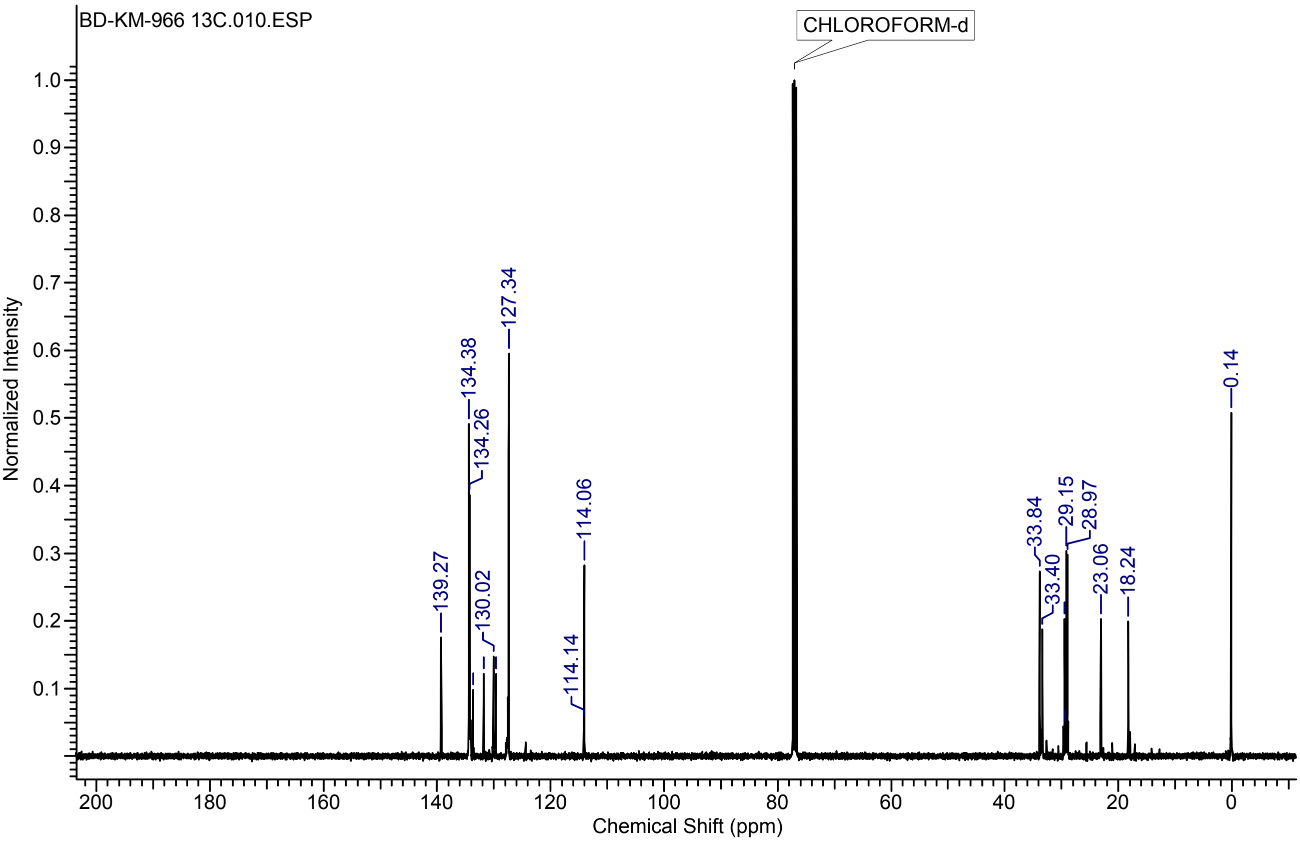
MALDI-ToF MS: Calcd. for $C_{96}H_{140}Na^+O_{14}Si_{12}$: *m/z* 1875.7366 [M + Na^+]. Found: 1875.7329.

EA: Anal. calcd for $C_{96}H_{140}O_{14}Si_{12}$ (%): C, 62.15; H,7.61; found: C, 62.18; H, 7.65.

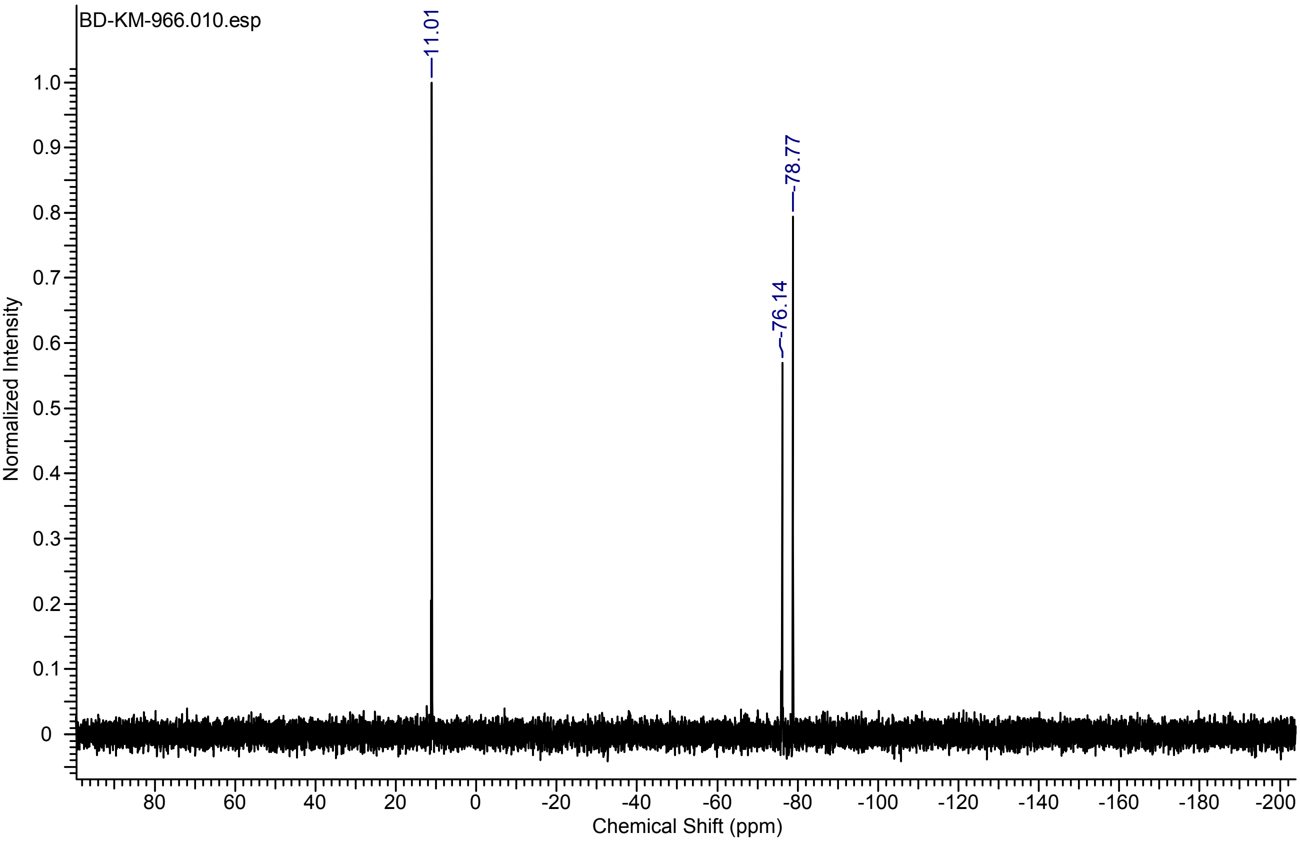
^{1}H NMR (300 MHz, $CDCl_3$)



^{13}*C NMR (101 MHz, CDCl$_3$)*



^{29}*Si NMR (79.5 MHz, CDCl$_3$)*



4. TGA curves of obtained products 1-5

Figure S6. TGA curves (performed in N_2) of obtained products 1-5

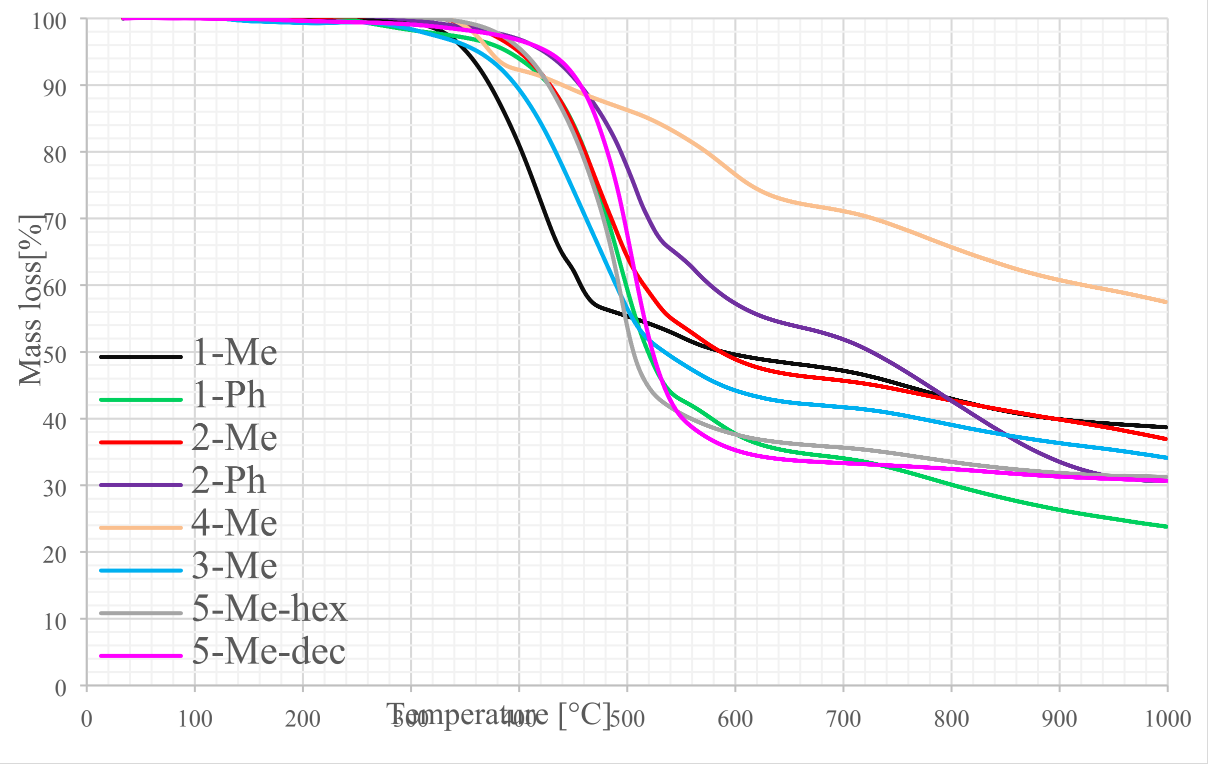
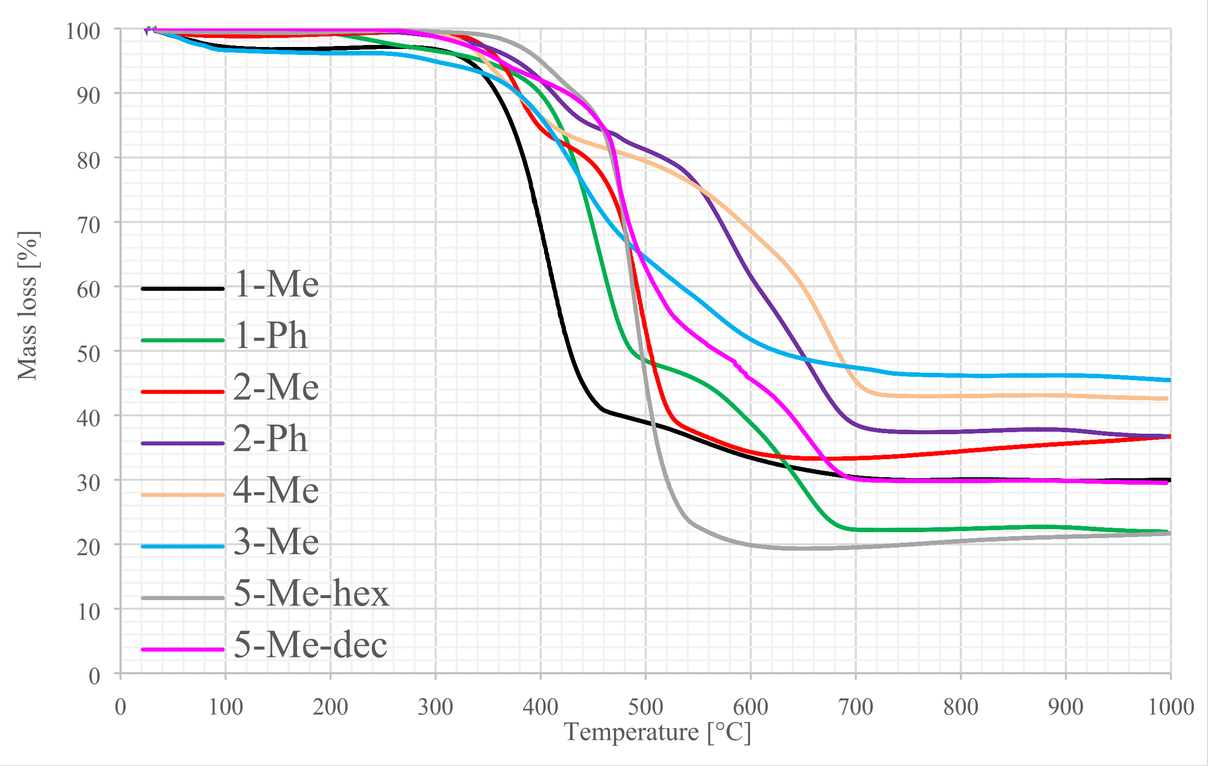


Figure S7. TGA curves (performed in air) of obtained products 1-5



5. References

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