Supplementary Information (SI) for ChemComm.

Supplementary Information

Light-driven silane functionalization using disulfide and dichloromethane

Jiin Lee,[a,b‡] Hyunji Kim,[a‡] Sunggi Lee*[a,b]

[a]Department of physics and Chemistry, DGIST, 333 Techno Jungang-daero, Dalseong-gun, Daegu 42988

[b]Center for Basic Science, DGIST, 333 Techno Jungang-daero, Dalseong-gun, Daegu 42988

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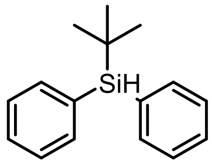
1. General Consideration

All chemicals and solvents were used without further purifications. ^{1}H, ^{13}C spectra were recorded on a Bruker Avance III-400 spectrometer. The chemical shifts reported in ppm relative to residual solvent $CDCl_3$, CD_2Cl_2 or added TMS as an internal reference. Splitting patterns are designated as follows: br; broad, s; singlet, d; doublet, t; triplet, q; quartet, m; multiplet, dt; double of triplet, td; triple of doublet, dd; double of doublet. Flash chromatography was carried out on Merck silica 60 (230-400 mesh ASTM). Analytical thin-layer chromatography (TLC) was performed on E. Merck precoated silica gel 60 F_{254} plates. Mass data acquired on a Supercritical Fluid Chromatograph combined with Xevo G2-XS QTOF Mass Spectrometer(Waters, Milford, MA, U.S.A., NFEC-2022-12-283850) at the Chiral Material Core Facility Center of Sungkyunkwan University. Blue light LEDs were purchased from Kessil (A160WE, PR 160 390 nm).

2. Experimental Section

2-1. Preparation of Substrate

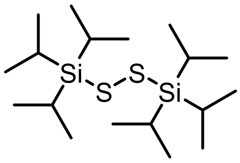
***tert*-butyldiphenylsilane (1b)**



In a 25 mL round-bottom flask, diphenylsilane (50 uL, 0.27 mmol, 1.0 equiv.) was added in dry THF (0.5 mL) with a magnetic stirrer bar. A pentane solution of *tert*-butyllithium (1.6 M, 170 uL, 0.27 mmol, 1.0 equiv.) was added to the reaction mixture at -78 ºC and stirred for 1 hour. Reaction was quenched with H_2O, than extracted with diethyl ether and dried over $MgSO_4$. Solvent was removed under reduced pressure. Colorless solid was obtained (57.6 mg, 89%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.64 (m, 4H), 7.38 (qdt, J = 8.5, 4.1, 2.1 Hz, 6H), 4.63 (s, 1H), 1.07 (s, 10H).

2-2. Preparation of Disulfide

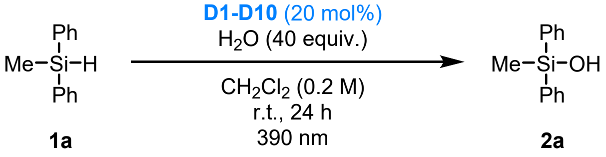
1,2-bis(triisopropylsilyl)disulfane (D7)[1]

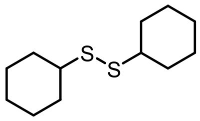


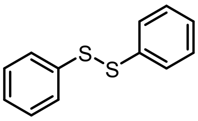
A vial was charged with triisopropylsilanethiol (122 μL, 0.57 mmol, 1.14 equiv.), of Et_3N (75 μL, 0.54 mmol, 1.08 equiv.), and 1.0 mL of toluene. Then iodine (63.8 mg, 0.50 mmol, 1.00 equiv.) was added in one portion. The resulting mixture was allowed to stir at room temperature for 15 minutes. The residual solid was filtered through a pad of Celite to afford a pale brown solution. The solution was passed through a plug of silica gel eluting with additional toluene to afford a colorless solution. Solvent was evaporated under reduced pressure. The crude residue was dissolved in pentane, and the solvent was removed to afford the purified solid. Colorless solid was obtained (103 mg, 51%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.48 – 1.33 (m, 6H), 1.16 (d, J = 7.4 Hz, 36H).

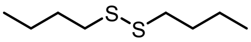
2-3. Reaction Optimization

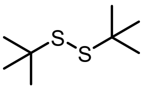
Table S1. Disulfide screening[a]

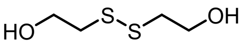


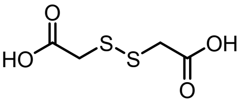

D1, 97%

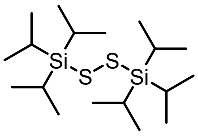

D2[b], 8%

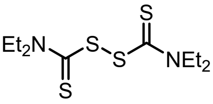

D3, 78%

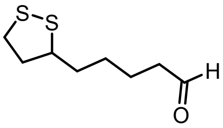

D4, 76%

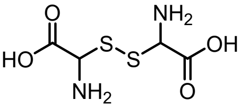

D54, 45%


D6, 6%


D7, 78%

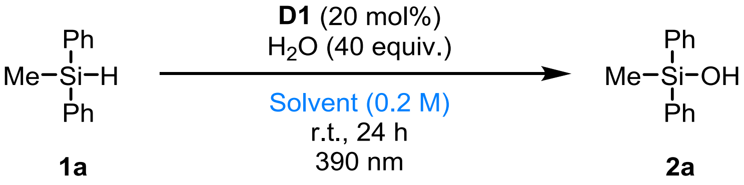

D8, 5%


D9, trace


D10, 8%

[a] Reaction conditions (0.2 mmol scale): **1a** (0.2 mmol, 1.0 equiv.), disulfide (20 mol%), H_2O (40 equiv.) in 1.0 mL DCM (0.2 M), 390 nm irradiation for 24 h under Ar. [b] 440 nm irradiation. Yield was determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard.

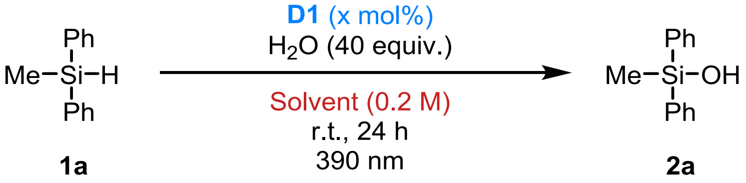
Table S2. Solvent screening[a]



Entry	Solvent	Remained starting material (%)[b]	Yield (%)[c]
1	*n*-BuCl	82	10
2	DCE	46	38
3	DCM	< 5	91
4	$CHCl_3$	< 5	95

[a] Reaction conditions (0.2 mmol scale): **1a** (0.2 mmol, 1.0 equiv.), **D1** (20 mol%), H_2O (40 equiv.) in 1.0 mL solvent (0.2 M), 390 nm irradiation for 24 h under Ar. [b, c] Yield and remained starting material determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard. DCE=1,2-dichloroethane.

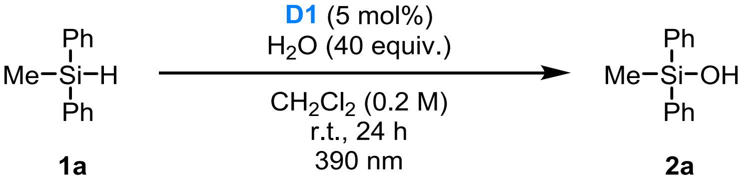
Table S3. Disulfide loading screening[a]



Entry	Solvent	Disulfide (x mol%)	Remained starting material (%)[b]	Yield (%)[c]
1	DCM	0.1	88	3
2	DCM	1	77	15
3	DCM	5	< 5	97
4	DCM	10	< 5	96
5	DCM	20	< 5	91
6	$CHCl_3$	0.1	84	7
7	$CHCl_3$	1	82	8
8	$CHCl_3$	5	52	43
9	$CHCl_3$	10	16	74
10	$CHCl_3$	20	< 5	95

[a]Reaction conditions (0.2 mmol scale): **1a** (0.2 mmol, 1.0 equiv.), **D1** (0.1-20 mol%), H_2O (40 equiv.) in 1.0 mL solvent (0.2 M), 390 nm irradiation for 24 h under Ar. [b, c]Yield and remained starting material determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard.

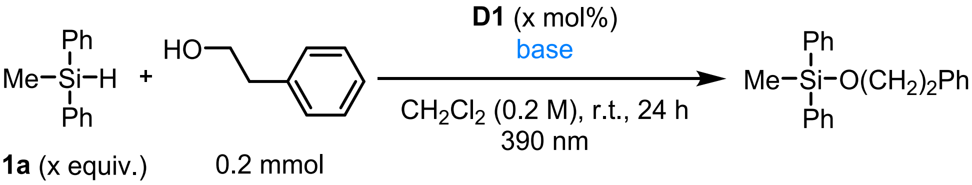
Table S4. Control experiment[a]



Entry	Variation	Remained starting material (%)[b]	Yield (%)[c]	Yield of bypdt (%)[d]
1	Without H_2O	11	< 5	82 (silyl chloride)
2	440 nm instead of 390 nm	> 95	< 5	-
3	456 nm instead of 390 nm	97	< 5	-
4	Under air	< 5	93	-

[a]Reaction conditions (0.2 mmol scale): **1a** (0.2 mmol, 1.0 equiv.), **D1** (5 mol%), H_2O (40 equiv.) in 1.0 mL DCM (0.2 M), 390 nm irradiation for 24 h under Ar. [b, c]Yield and remained starting material determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard.

Table S5. **Base screening**[a]



Entry	Additive	Silane	Disulfide	Wavelength	Remained starting material (%)[b]	Yield (%)[c]
1	-	1.2	5	390	49	50
2	**Imidazole (1 equiv.)**	1.2	5	390	19	80
3	**TEA (1 equiv.)**	1.2	5	390	> 95	< 5
4	-	1.5	10	390	60	52
5	**Imidazole (1.5 equiv.)**	1.5	10	390	< 5	> 95

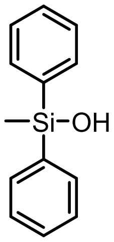
[a] Reaction conditions: 0.2 mmol of 2-phenylethyl alcohol, x equiv of diphenylmethyl silane, y mol% $(c\text{HxS})_2$ in 1.0 mL of DCM under 390 nm irradiation. [b,c] Yield and remained starting material determined by ^1H NMR spectroscopy using CH_2Br_2 as an internal standard.

2-4. Synthesis of silanol

All reactions were monitored by thin-layer chromatography (TLC). NMR yield was measured with dibromomethane as an internal standard and isolated yield was obtained by flash chromatography using silica gel.

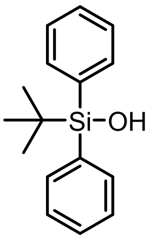
General Procedure for synthesis of silanols (**General Procedure A**). In an oven-dried 4 ml vial, silane (0.2 mmol, 1.0 equiv.), dicyclohexyl disulfide (**D1**) (2.2 μL, 5 mol%), H_2O (40 equiv.), CH_2Cl_2 (1.0 mL, 0.2 M) were added under argon atmosphere. The vial was sealed with electric tape and stirred for 24 hours at room temperature (~30 ˚C) under 390 nm irradiation (390 nm Kessil LEDs lamp). After the reaction was completed, the solvent was evaporated under reduced pressure. And the crude mixture was purified by silica gel column chromatography. NMR yield was determined by ^{1}H NMR spectroscopy using CH_2Br_2 as an internal standard.

methyldiphenylsilanol (2a)



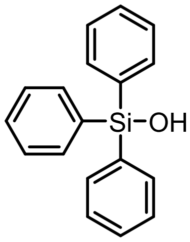
Following General Procedure A, **2a** was obtained (42.9 mg, 72%) as a colorless oil by silica gel column chromatography (10% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67 – 7.57 (m, 4H), 7.48 – 7.34 (m, 6H), 0.68 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 137.20, 134.09, 130.00, 128.04, -1.13.

***tert*-butyldiphenylsilanol (2b)**



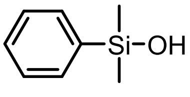
Following General Procedure A, the reaction was conducted for 48 hours. **2b** was obtained (38.2 mg, 74%) as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 – 7.76 (m, 4H), 7.50 – 7.37 (m, 6H), 1.17 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 135.21, 134.85, 129.69, 127.77, 26.61, 19.05.

triphenylsilanol (2c)



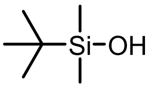
Following General Procedure A, **2c** was obtained (55.3 mg, 89%) as a colorless oil by silica gel column chromatography (10% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68 – 7.62 (m, 6H), 7.50 – 7.37 (m, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 135.26, 135.13, 130.23, 128.05.

dimethylphenylsilanol (2d)



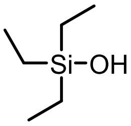
Following General Procedure A, **2d** was obtained (23.1 mg, 76%) as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 – 7.53 (m, 2H), 7.44 – 7.32 (m, 3H), 0.35 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.98, 133.15, 129.40, 127.85, 1.00.

tert-butyldimethylsilanol (2e)



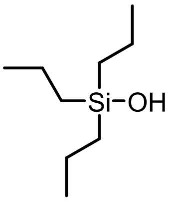
Following General Procedure A, **2e** was obtained (72% NMR yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.90 (s, 9H), 0.08 (s, 6H). Spectrum in accordance with literature: *J. Chem. Lett.,* **2015**, *44*, 1062–1064.

triethylsilanol (2f)



Following General Procedure A, **2f** was obtained (97% NMR yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.98 (t, *J* = 8.0 Hz, 9H), 0.65 – 0.55 (m, 6H). Spectrum in accordance with literature: *Synlett,* **2016***, 27,* 1649–1652.

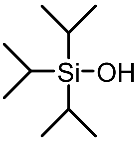
tripropylsilanol (2g)



Following General Procedure A, **2g** was obtained (96% NMR yield).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.43 – 1.32 (m, 6H), 0.96 (t, *J* = 7.4 Hz, 9H), 0.57 (dt, *J* = 11.5, 4.2 Hz, 6H).

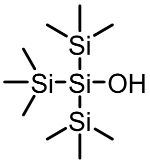
Spectrum in accordance with literature: *ACS Catal.*, **2013**, *3*, 2239–2243.

triisoproylsilanol (2h)



Following General Procedure A, the reaction was conducted for 72 hours with imidazole (20.4 mg, 1.5 equiv.). **2h** was obtained (22.3 mg, 64%) as a colorless oil by silica gel column chromatography (10% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.05 (s, 21H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 17.84, 12.44.

tris(trimethylsilyl)silanol (2i)

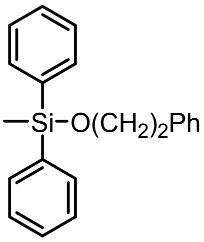


Following General Procedure A, **2i** was obtained (23.7 mg, 45%) as a colorless oil by silica gel column chromatography (10% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.20 (d, *J* = 17.0 Hz, 27H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ -0.57.

2-5. Synthesis of silyl ether

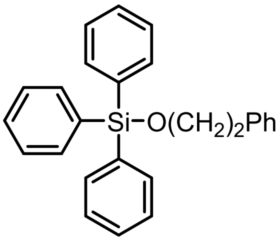
General Procedure for synthesis of tri-substituted silyl ethers (**General Procedure B**). In an oven-dried 4 ml vial, 2-phenylethyl alcohol (24 μL, 0.2 mmol, 1.0 equiv.), silane (0.3 mmol, 1.5 equiv.). dicyclohexyl disulfide (**D1**) (4.4 μL, 10 mol%) and imidazole (20.4 mg, 1.5 equiv.) were added to 1.0 mL of CH_2Cl_2 (0.2 M) under argon atmosphere. The vial was sealed with electric tape and stirred for 24 hours at room temperature (~30 ˚C) under 390 nm irradiation (390 nm Kessil LEDs lamp). After the reaction was completed, the solvent was evaporated under reduced pressure. And the crude mixture was purified by silica gel column chromatography. NMR yield was determined by ^{1}H NMR spectroscopy using CH_2Br_2 as an internal standard.

methyl(phenethoxy)diphenylsilane (3a)



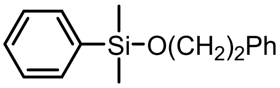
Following General Procedure C, **3a** (48.4 mg, 76%) was obtained as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67 – 7.53 (m, 4H), 7.49 – 7.34 (m, 6H), 7.34 – 7.13 (m, 5H), 3.94 (t, *J* = 7.1 Hz, 2H), 2.92 (t, *J* = 7.1 Hz, 2H), 0.62 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.90, 136.03, 134.37, 129.81, 129.19, 128.28, 127.86, 126.20, 64.73, 39.32, -3.13.

phenethoxytriphenylsilane (3b)



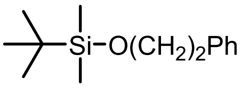
Following General Procedure C, **3b** (70.5 mg, 93%) was obtained as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 – 7.51 (m, 6H), 7.45 – 7.40 (m, 3H), 7.38 – 7.32 (m, 6H), 7.26 – 7.17 (m, 3H), 7.15 – 7.09 (m, 2H), 3.98 (t, *J* = 7.2 Hz, 2H), 2.88 (t, *J* = 7.1 Hz, 2H).**^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.82, 135.39, 134.19, 130.00, 129.19, 128.26, 127.86, 126.16, 65.07, 39.24.

dimethyl(phenethoxy)(phenyl)silane (3c)



Following General Procedure C, **3c** (26.1 mg, 51%) was obtained. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 – 7.50 (m, 2H), 7.44 – 7.32 (m, 3H), 7.30 – 7.25 (m, 2H), 7.23 – 7.15 (m, 3H), 3.81 (t, *J* = 7.2 Hz, 2H), 2.85 (t, *J* = 7.2 Hz, 2H), 0.34 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.76, 139.67, 135.34, 131.45, 130.98, 130.14, 129.71, 128.05, 66.20, 41.23, -0.00.

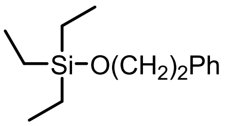
***tert*-butyldimethyl(phenethoxy)silane (3d)**



Following General Procedure C, the reaction was conducted for 48 hours. **3d** (39.4 mg, 83%) was obtained as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.25 (m, 2H), 7.22 (d, *J*

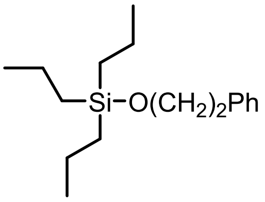
= 7.1 Hz, 3H), 3.82 (t, *J* = 7.1 Hz, 2H), 2.84 (t, *J* = 7.1 Hz, 2H), 0.89 (s, 9H), 0.00 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.21, 129.19, 128.24, 126.14, 64.61, 39.69, 25.97, 18.38, -5.38.

triethyl(phenethoxy)silane (3e)



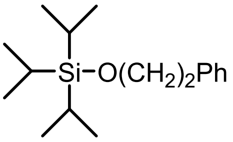
Following General Procedure C, **3e** (36.1 mg, 76%) was obtained as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.25 (m, 2H), 7.22 (d, *J* = 7.3 Hz, 3H), 3.82 (t, *J* = 7.3 Hz, 2H), 2.86 (t, *J* = 7.3 Hz, 2H), 0.95 (t, *J* = 7.9 Hz, 9H), 0.59 (q, *J* = 8.0 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.04, 129.10, 128.25, 126.14, 64.25, 39.70, 6.73, 4.41.

phenethoxytripropylsilane (3f)



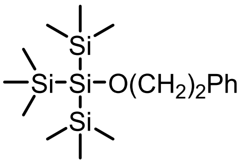
Following General Procedure C, **3f** was obtained (31.1 mg, 56%) as a colorless oil by silica gel column chromatography (3% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 2H), 7.31 – 7.25 (m, 3H), 3.87 (t, *J* = 7.3 Hz, 2H), 2.91 (t, *J* = 7.3 Hz, 2H), 1.54 – 1.34 (m, 6H), 1.10 – 0.99 (m, 9H), 0.74 – 0.55 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.07, 129.09, 128.24, 126.12, 64.23, 39.67, 18.37, 16.74, 16.39. **HRMS (ESI)** m/z: $[M+Na]^+$: calcd. for $C_{17}H_{30}OSi$ 301.1958; Found 301.1958.

phenethoxytriisopropylsilane (3g)



Following General Procedure C, the reaction was conducted for 72 hours. **3g** (46.3 mg, 83%) was obtained as a colorless oil by silica gel column chromatography (5% EtOAc in hexanes). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 2H), 7.24 – 7.17 (m, 3H), 3.88 (t, *J* = 7.3 Hz, 2H), 2.86 (t, *J* = 7.3 Hz, 2H), 1.10 – 1.00 (m, 21H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.23, 129.16, 128.20, 126.07, 64.87, 39.81, 17.98, 12.00.

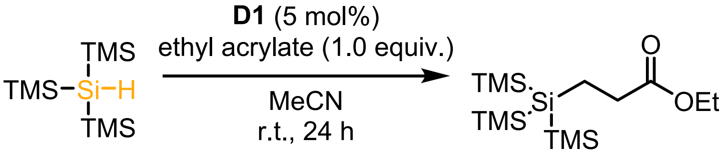
1-[tris(trimethylsilyl)silyl]-2-phenylethanol (3h)



Following General Procedure C, the reaction was conducted for 48 hours to afford **3h** (43% NMR yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (ddd, *J* = 8.8, 5.9, 1.8 Hz, 2H), 7.37 – 7.28 (m, 3H), 3.79 (t, *J* = 7.0 Hz, 2H), 2.91 (t, *J* = 7.0 Hz, 2H), 0.30 (s, 27H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.02, 128.89, 127.88, 125.73, 68.76, 39.49, -0.00. **HRMS (APCI)** m/z: $[M+]^+$: calcd. for $C_{16}H_{33}OSi_4$ 353.1602; Found 353.1609.

2-6. Mechanistic studies

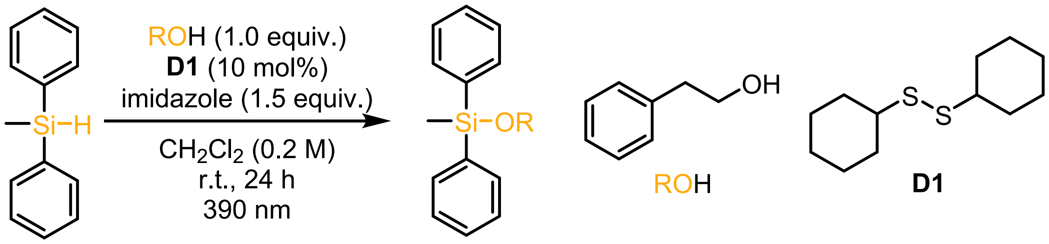
Radical trapping experiments:



5a, 73%

In an oven-dried 4 ml vial, tris(Trimethylsilyl)silane (49.2 mg, 0.2 mmol, 1.0 equiv.). dicyclohexyl disulfide (**D1**) (2.2 μL, 5 mol%) and ethyl acrylate (20.0 mg, 0.2 mmol, 1.0 equiv.) were added to 1.0 mL of MeCN (0.2 M) under argon atmosphere. The vial was sealed with electric tape and stirred for 24 hours at room temperature (~30 ˚C) under 390 nm irradiation (390 nm Kessil LEDs lamp). After the reaction was completed, the solvent was evaporated under reduced pressure. And the crude mixture was purified by silica gel column chromatography to give **5a** as a colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.14 – 7.09 (m, 2H), 7.06 – 6.99 (m, 3H), 3.49 (t, *J* = 7.0 Hz, 2H), 2.61 (t, *J* = 6.9 Hz, 2H), 0.00 (s, 27H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.02, 128.89, 127.88, 125.73, 68.76, 39.49, -0.00.

On/off experiments:



(1.5 equiv.)

In NMR tube, 2-phenylethyl alcohol (12 μL, 0.1 mmol, 1.0 equiv.), methyldiphenylsilane (0.15 mmol, 1.5 equiv.). dicyclohexyl disulfide (**D1**) (2.2 μL, 10 mol%) and imidazole (10.2 mg, 1.5 equiv.) were added to 0.5 mL of CD_2Cl_2 (0.2 M) under argon atmosphere. NMR yield was determined by ^{1}H NMR spectroscopy using mesitylene as an internal standard. The reaction was monitored by on/off irradiation cycles (0.5 h each) and the NMR spectra were recorded immediately after each interval without isolating the reaction mixture.

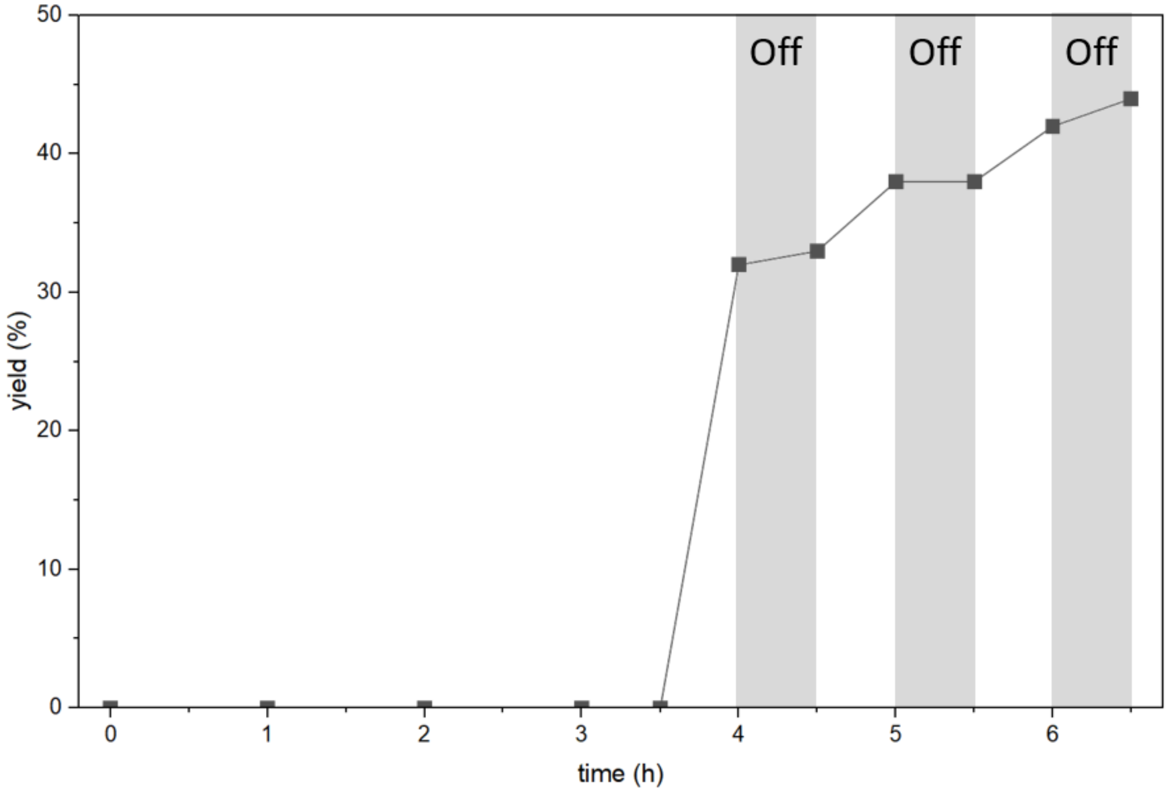
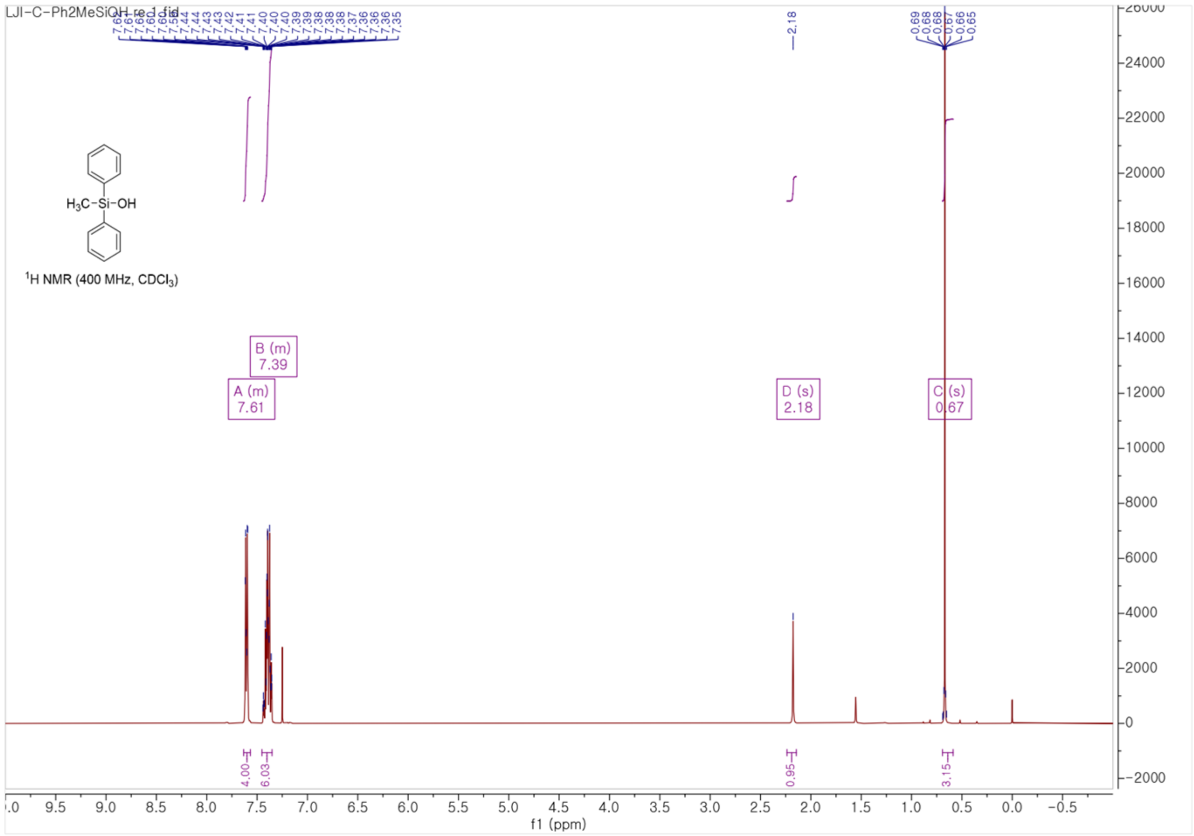
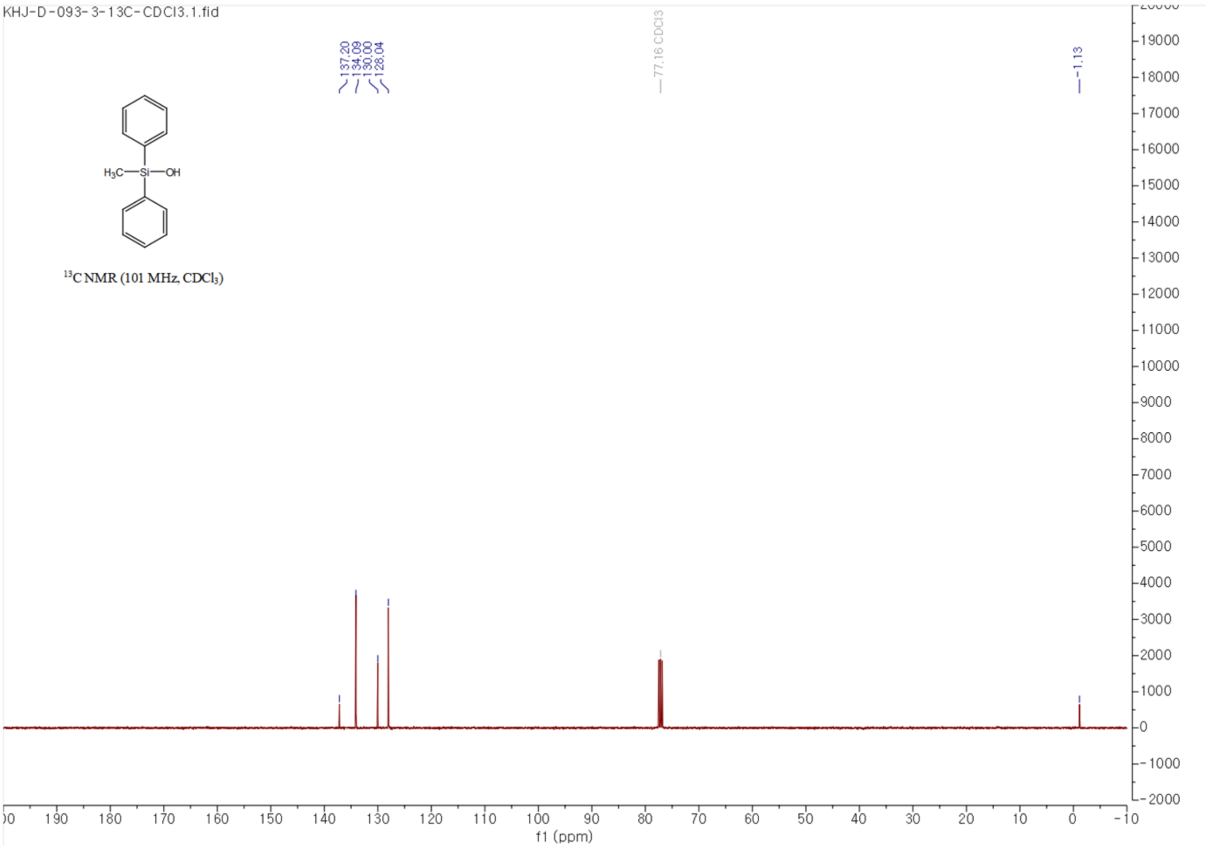
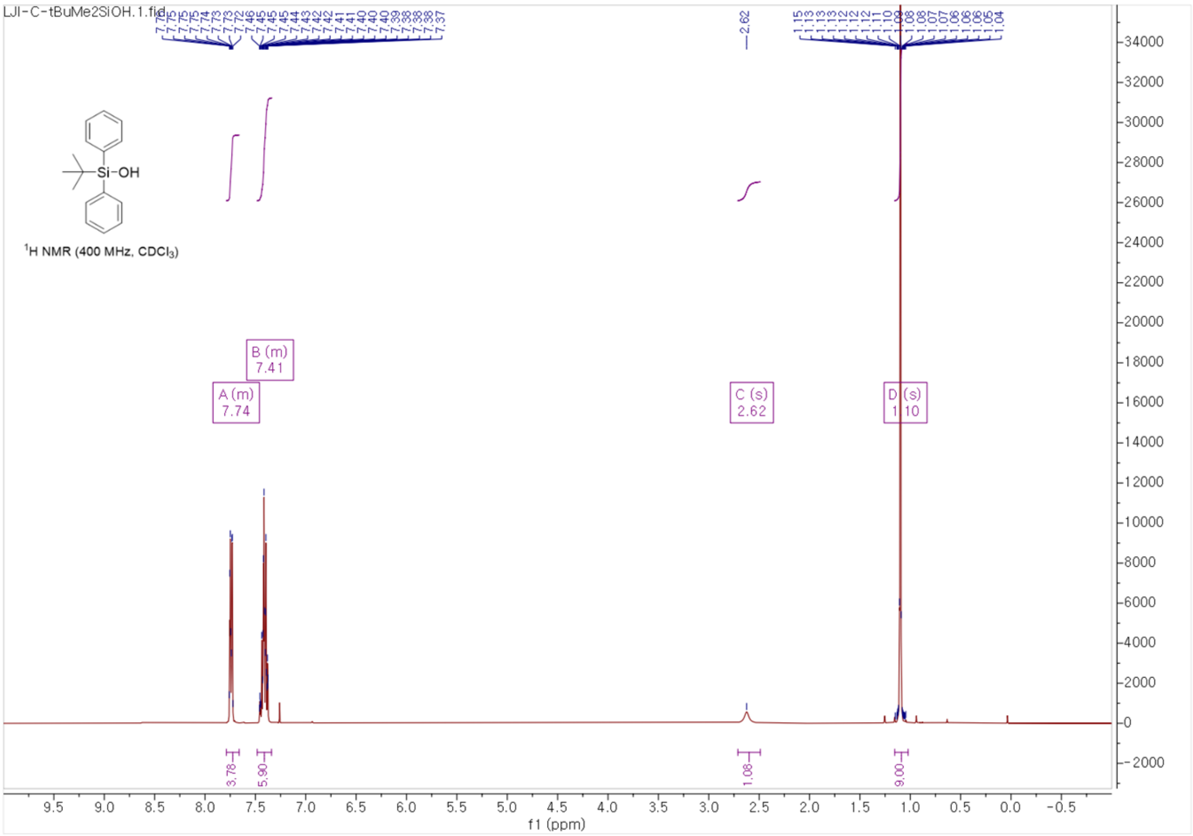


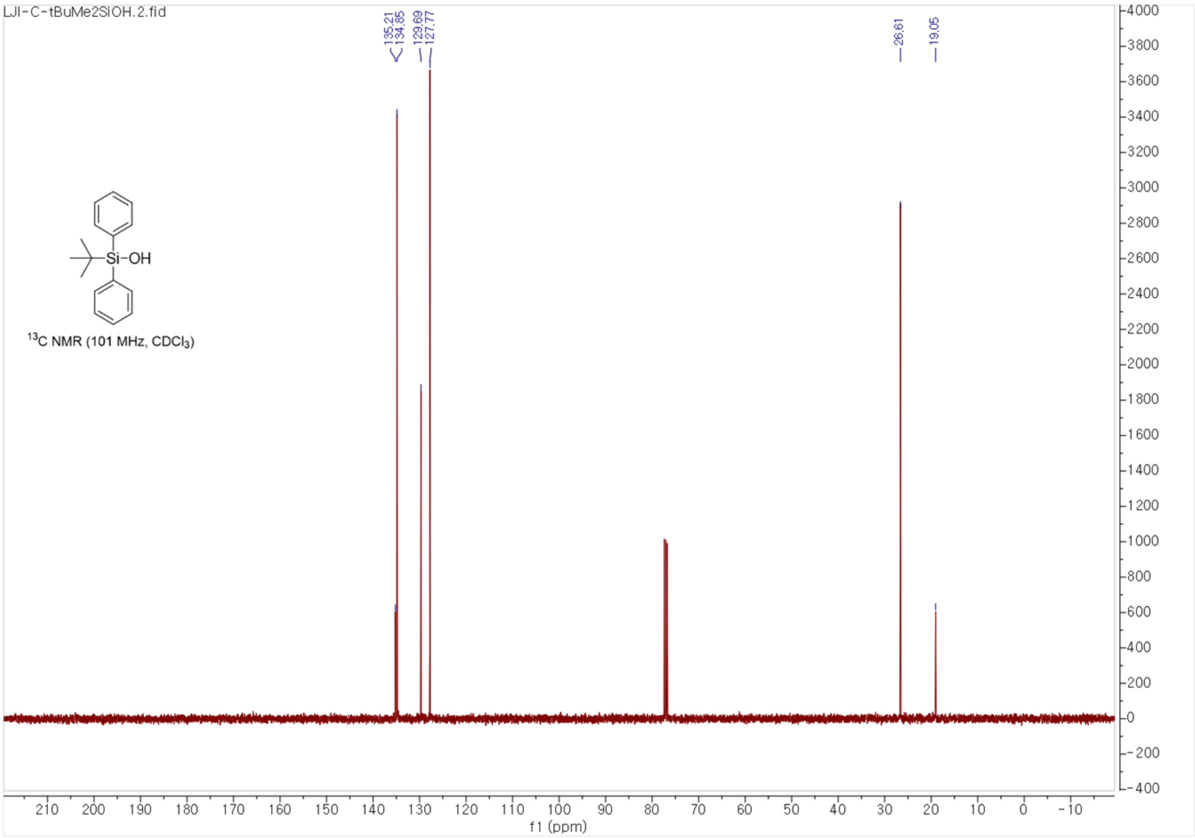
Figure S1. On/off experiments.

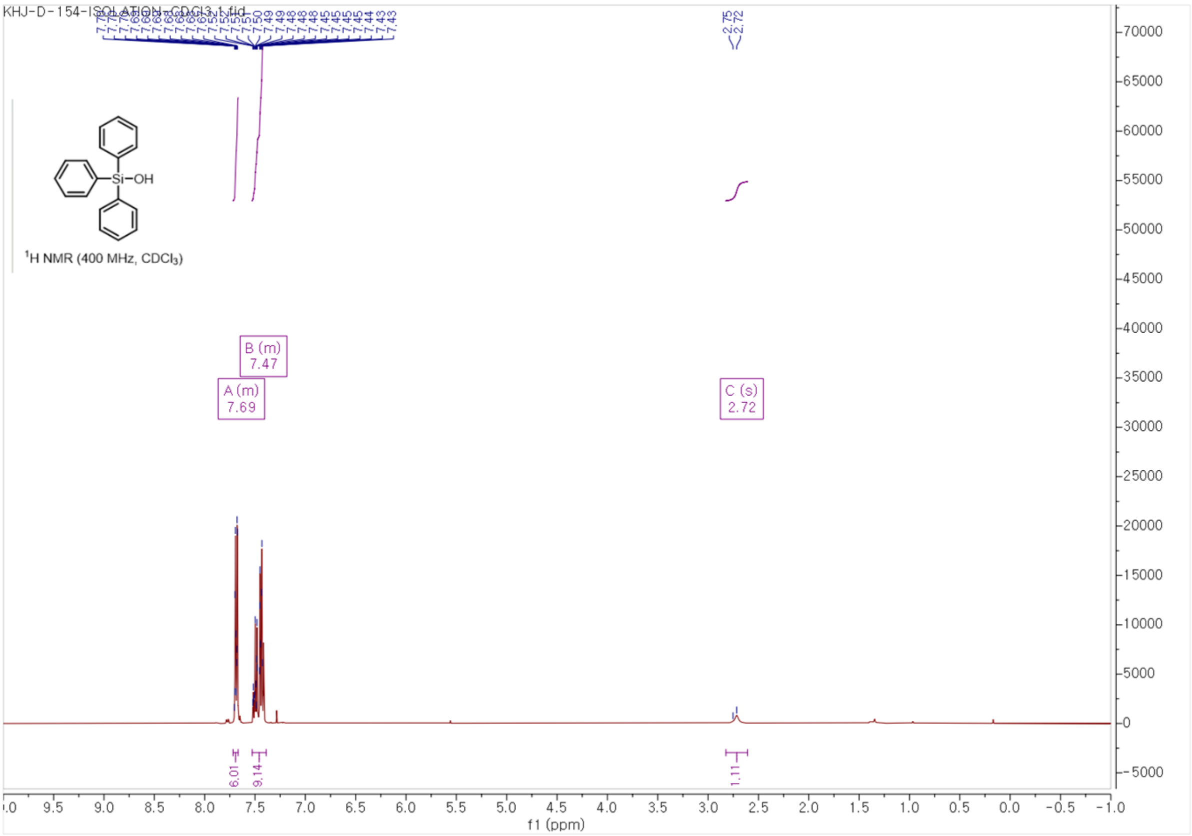
3. NMR spectra

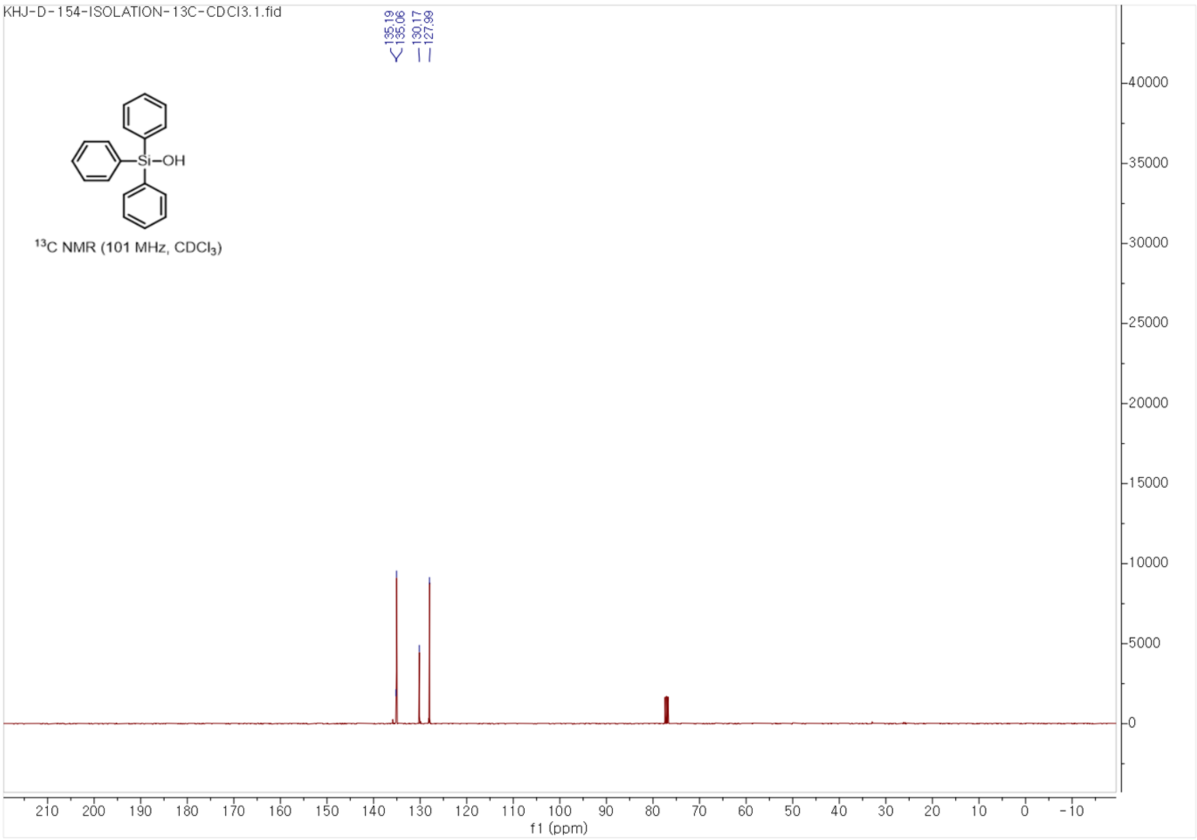


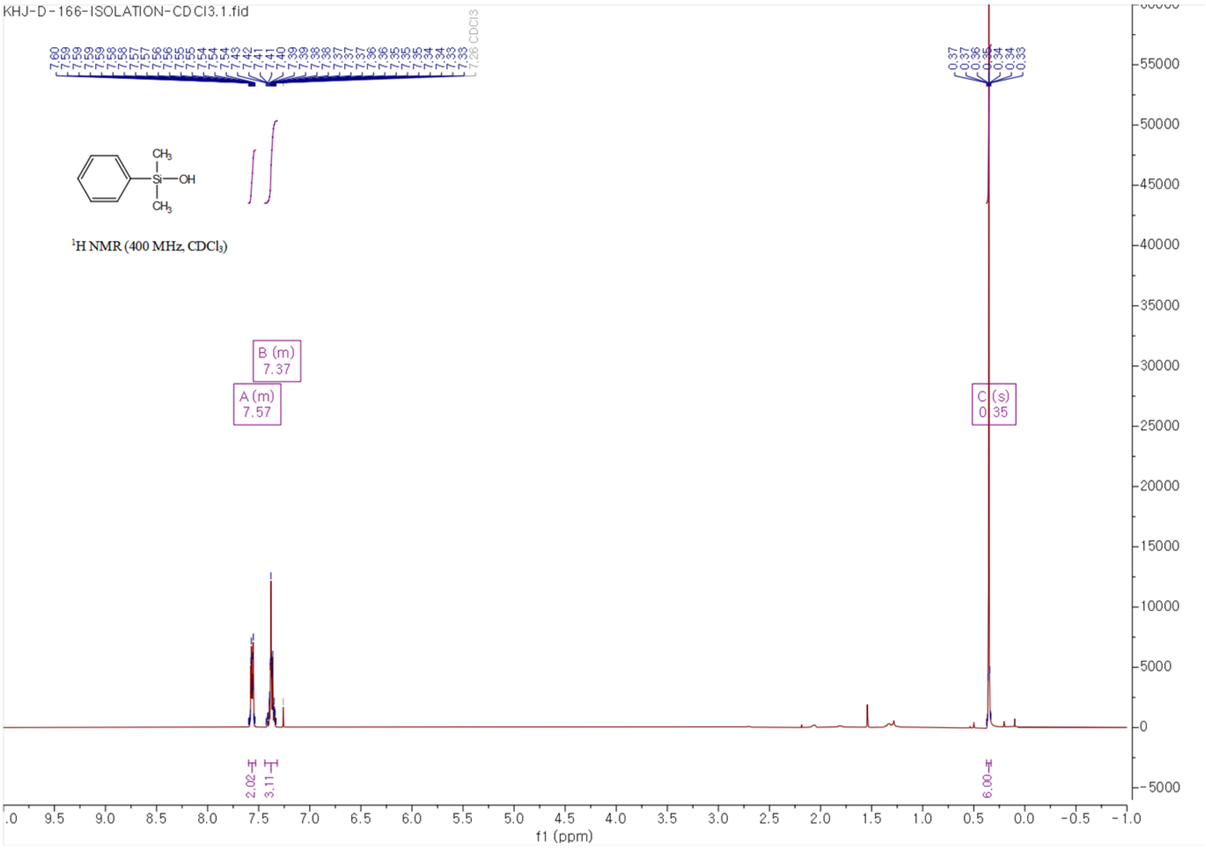


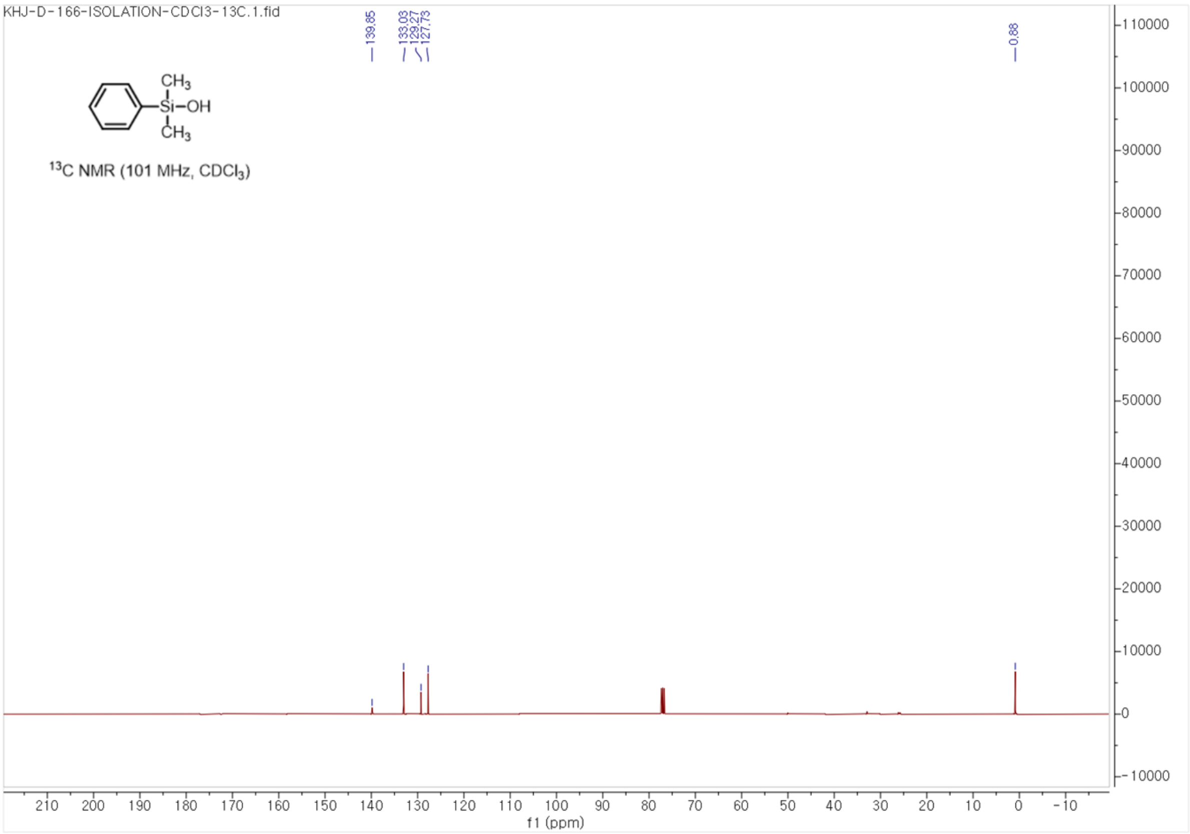

LJI-C-tBuMe2SiOH.1.fid
7.75
7.75
7.75
7.75
7.74
7.73
7.73
7.72
7.46
7.45
7.45
7.45
7.44
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.40
7.39
7.38
7.38
7.38
7.37
2.62
1.15
1.13
1.13
1.13
1.12
1.12
1.12
1.11
1.10
1.09
1.08
1.08
1.07
1.07
1.06
1.06
1.06
1.05
1.04
Si-OH
^{1}H NMR (400 MHz, CDCl$_3$)
B (m)
7.41
A (m)
7.74
C (s)
2.62
D (s)
1.10
3.78
5.90
1.08
9.00
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

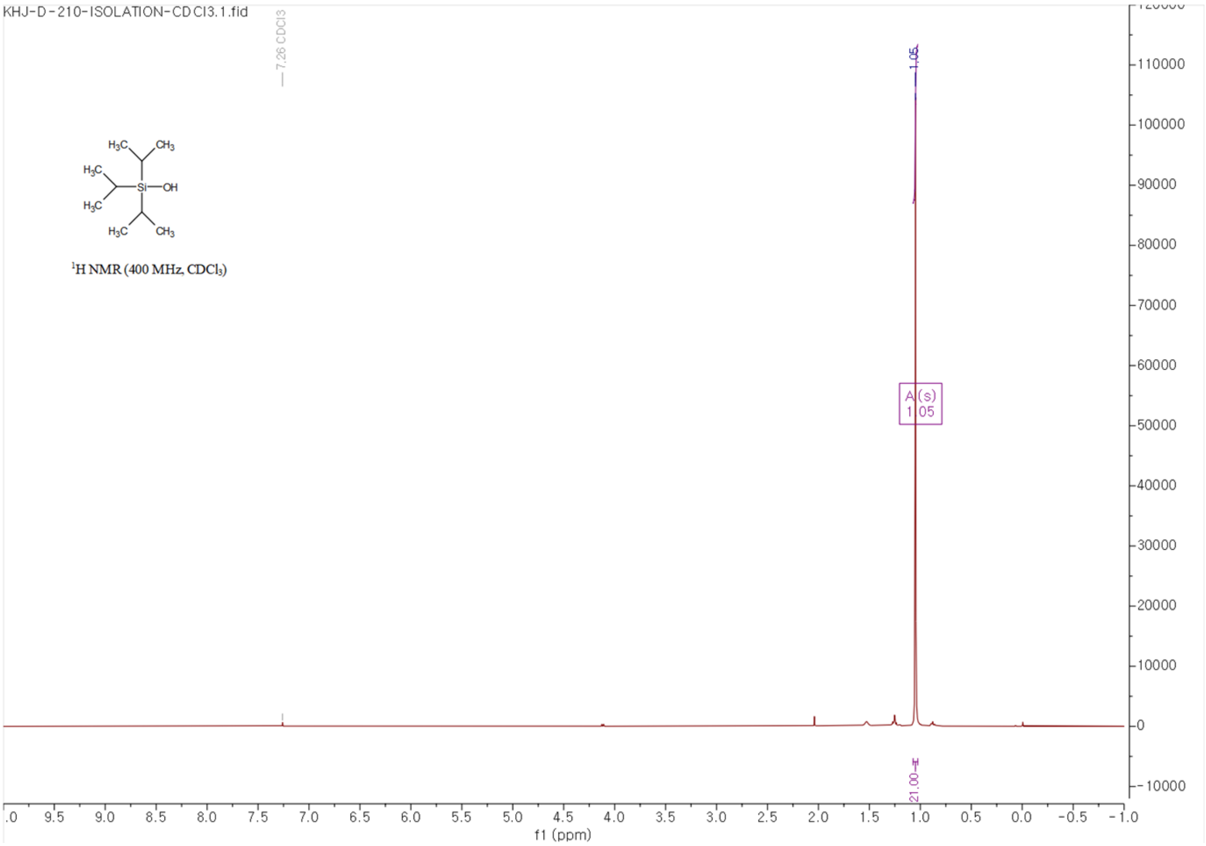

LJI-C-tBuMe2SiOH.2.fid
135.21
134.85
129.69
127.77
26.61
19.05
Si-OH
^{13}C NMR (101 MHz, CDCl$_3$)
4000
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
-400
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

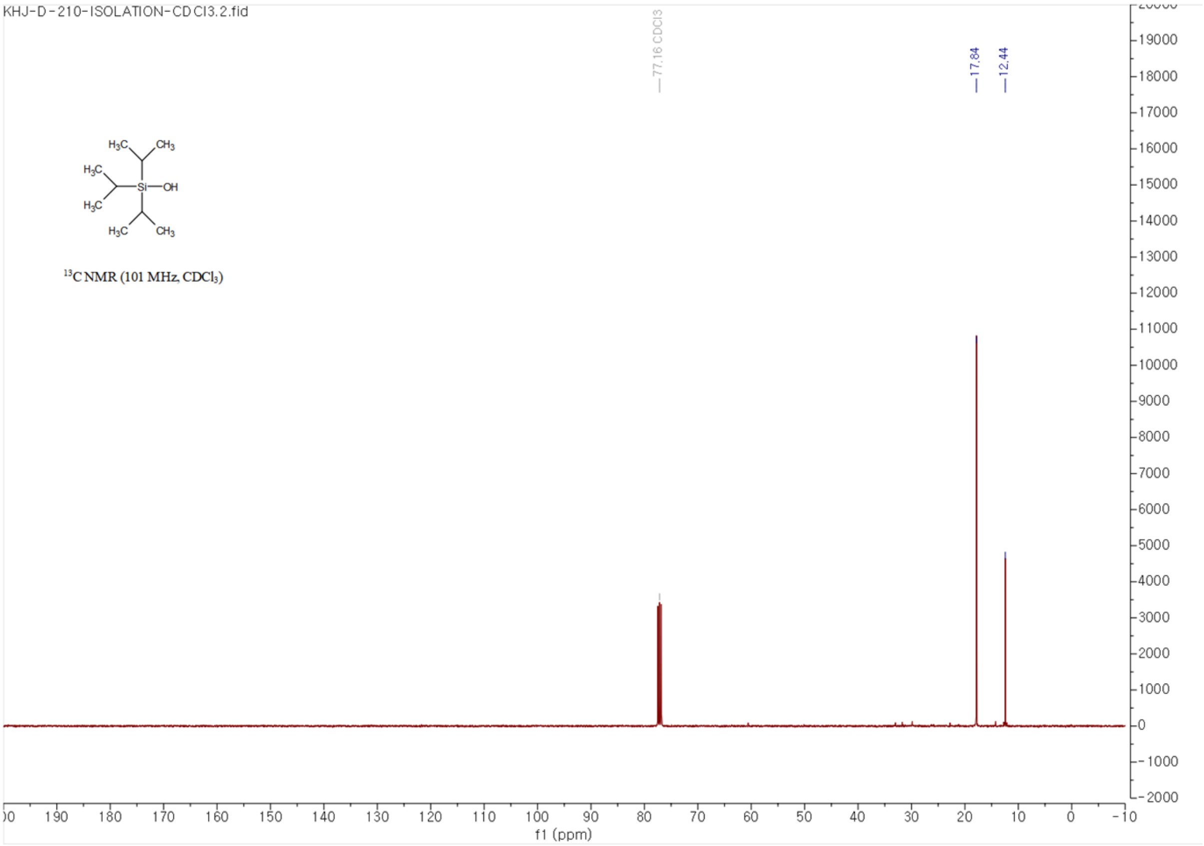

KHJ-D-154-ISOLATION-CDCl3.1.fid
7.50
7.49
7.49
7.48
7.48
7.48
7.45
7.45
7.45
7.45
7.44
7.43
7.43
2.75
2.72
Si–OH
^{1}H NMR (400 MHz, CDCl$_3$)
B (m) 7.47
A (m) 7.69
C (s) 2.72
6.01
9.14
1.11
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

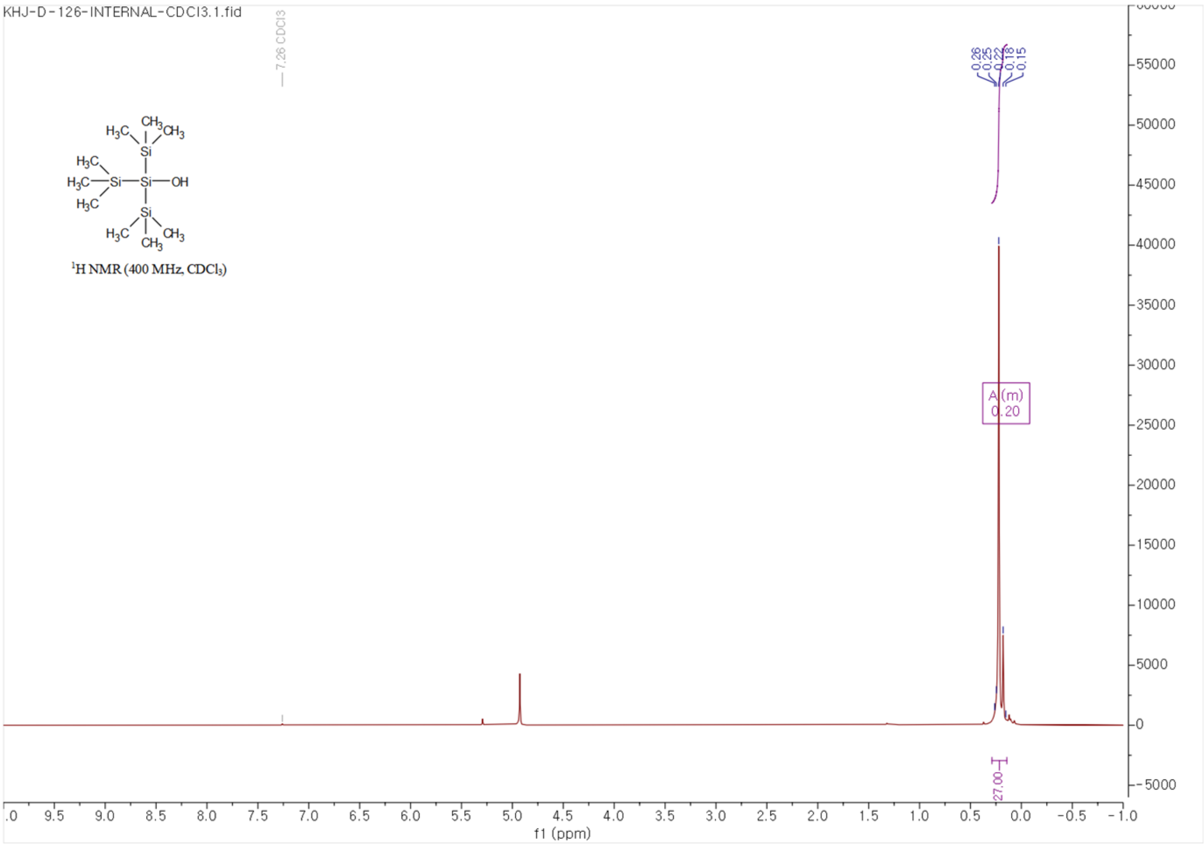

KHJ-D-154-ISOLATION-13C-CDCl3.1.fid
135.19
135.06
130.17
127.99
Si–OH
^{13}C NMR (101 MHz, CDCl$_3$)
40000
35000
30000
25000
20000
15000
10000
5000
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

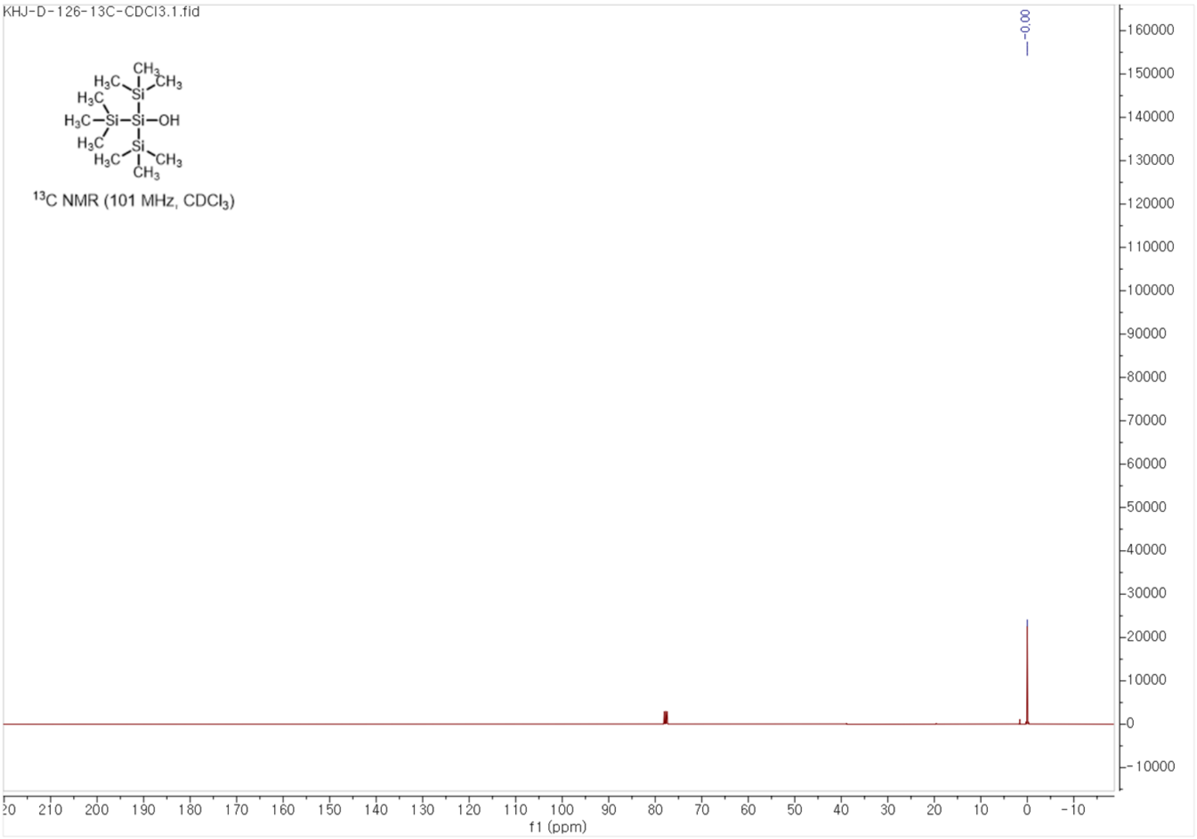

KHJ-D-166-ISOLATION-CDCl3.1.fid
7.60 7.59 7.59 7.59 7.59 7.58 7.58 7.57 7.57 7.56 7.56 7.55 7.55 7.54 7.54 7.54 7.43 7.42 7.41 7.41 7.40 7.39 7.39 7.38 7.38 7.37 7.37 7.37 7.36 7.36 7.35 7.35 7.35 7.34 7.34 7.33 7.33
7.26 CDCl3
0.37 0.37 0.36 0.35 0.34 0.34 0.33
CH3
Si
OH
CH3
1H NMR (400 MHz, CDCl3)
B (m) 7.37
A (m) 7.57
C (s) 0.35
2.02
3.11
6.00
f1 (ppm)

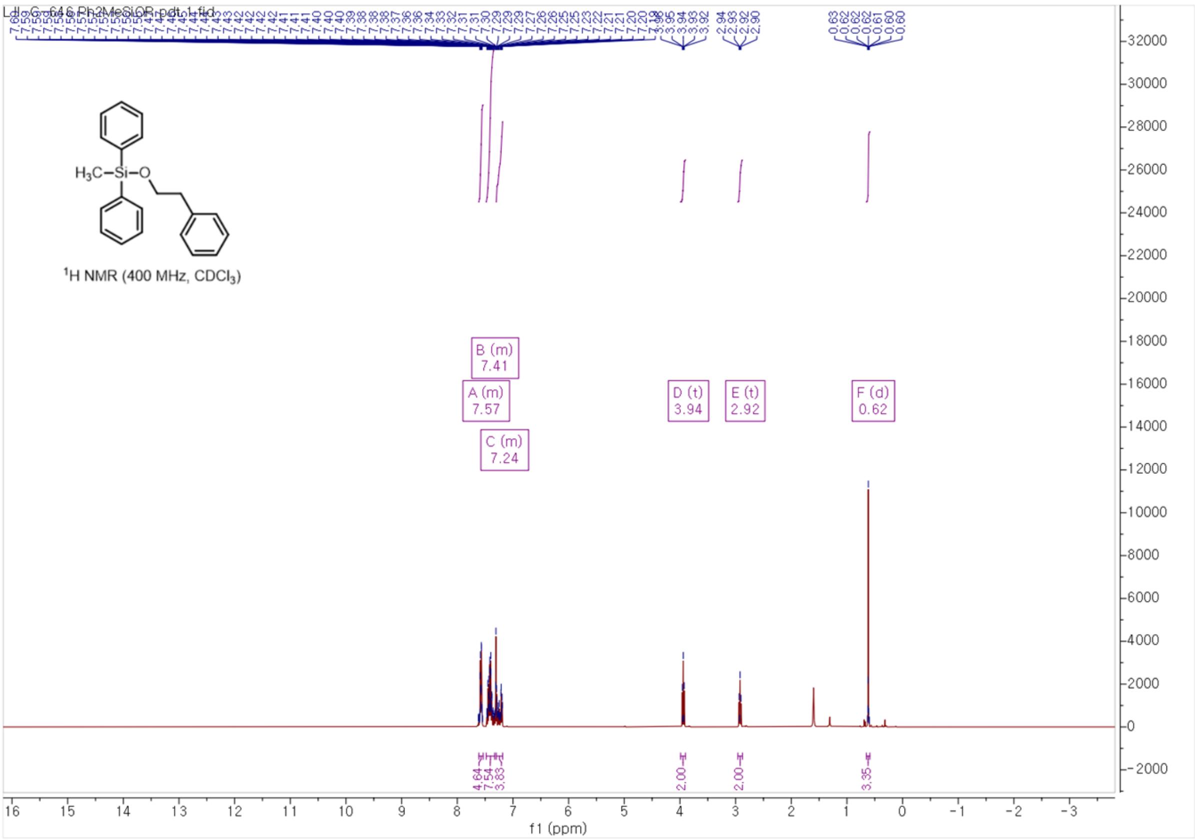

KHJ-D-166-ISOLATION-CDCl3-13C.1.fid
139.85
133.03
129.27
127.73
0.88
CH3
Si-OH
CH3
13C NMR (101 MHz, CDCl3)
f1 (ppm)


KHJ-D-210-ISOLATION-CDCl3.1.fid
7.26 CDCl3
1.05
^{1}H NMR (400 MHz, CDCl$_3$)
A (s)
1.05
21.00
f1 (ppm)
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
110000 100000 90000 80000 70000 60000 50000 40000 30000 20000 10000 0 -10000
Si
OH
H$_3$C
CH$_3$

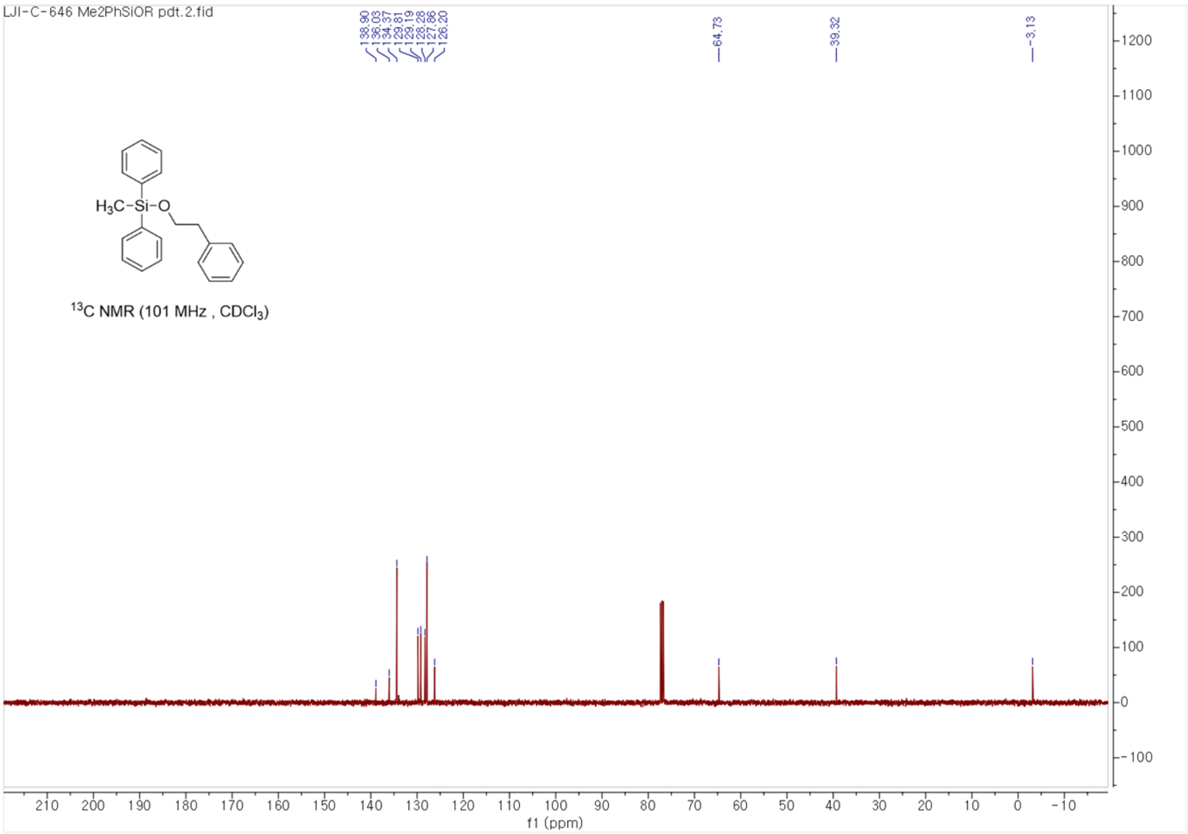

KHJ-D-210-ISOLATION-CDCl3.2.fid
77.16 CDCl3
17.84
12.44
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000
Si
OH
H$_3$C
CH$_3$


KHJ-D-126-INTERNAL-CDCl3.1.fid
7.26 CDCl3
0.26
0.25
0.22
0.18
0.15
A (m)
0.20
27.00
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)

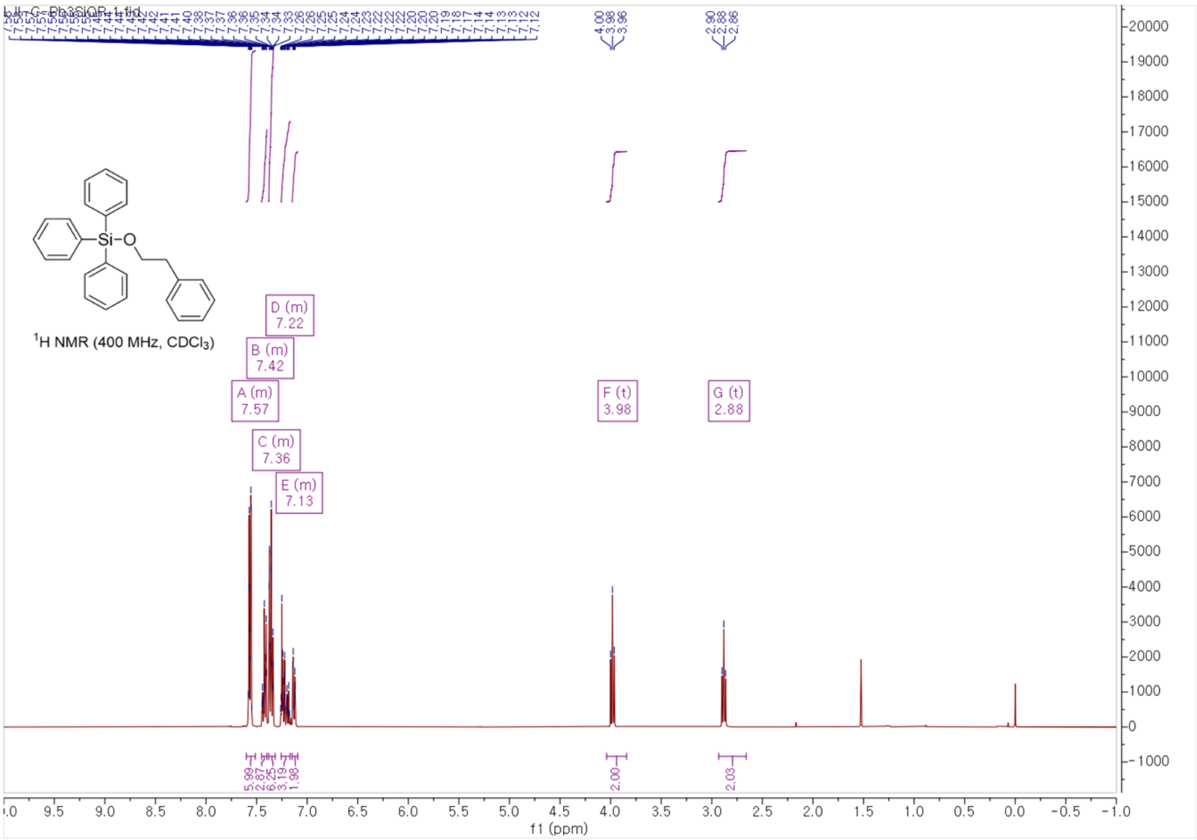

KHJ-D-126-13C-CDCl3.1.fid
-0.00
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)



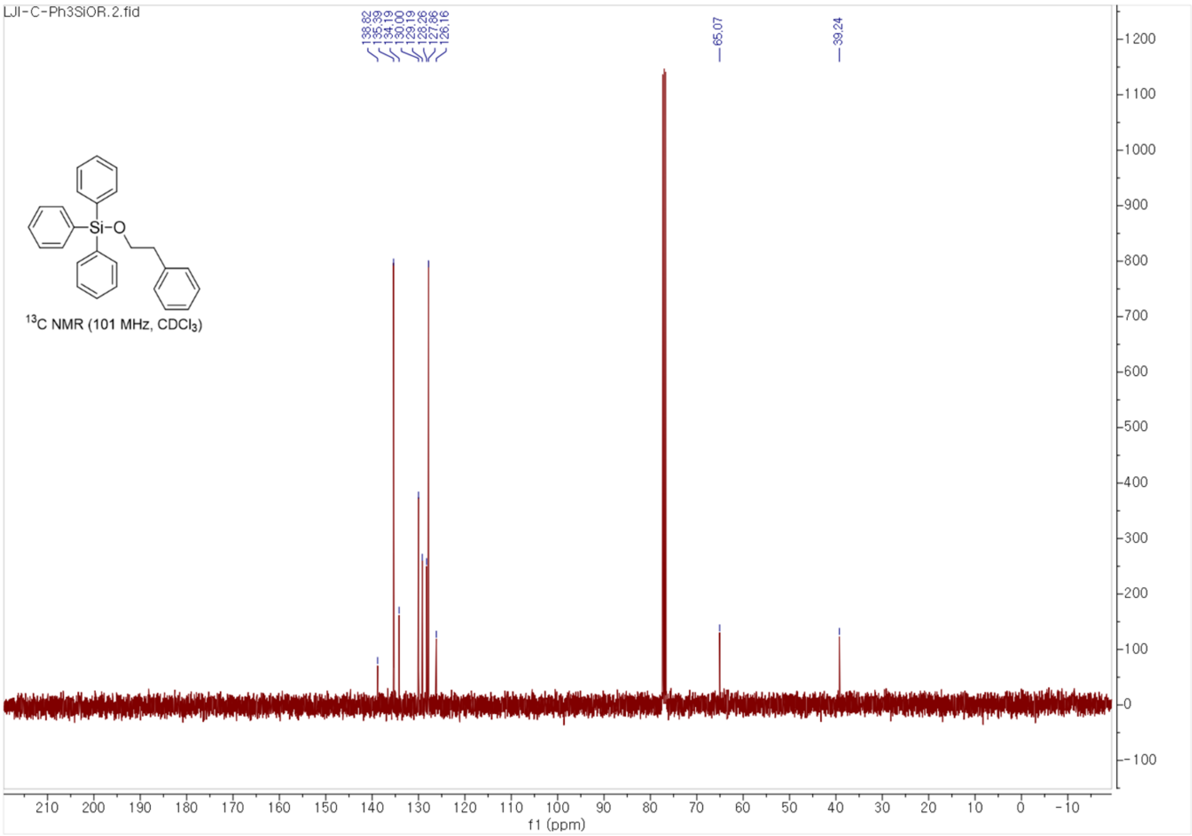
LJI-C-646 Ph2MeSiOR pdt.1.fid
3.95
3.94
3.93
3.92
2.94
2.93
2.92
2.90
0.63
0.62
0.62
0.62
0.61
0.60
0.60
$H_3C-Si-O$
^1H NMR (400 MHz, $CDCl_3$)
B (m) 7.41
A (m) 7.57
C (m) 7.24
D (t) 3.94
E (t) 2.92
F (d) 0.62
4.64
7.54
3.83
2.00
2.00
3.35
f1 (ppm)



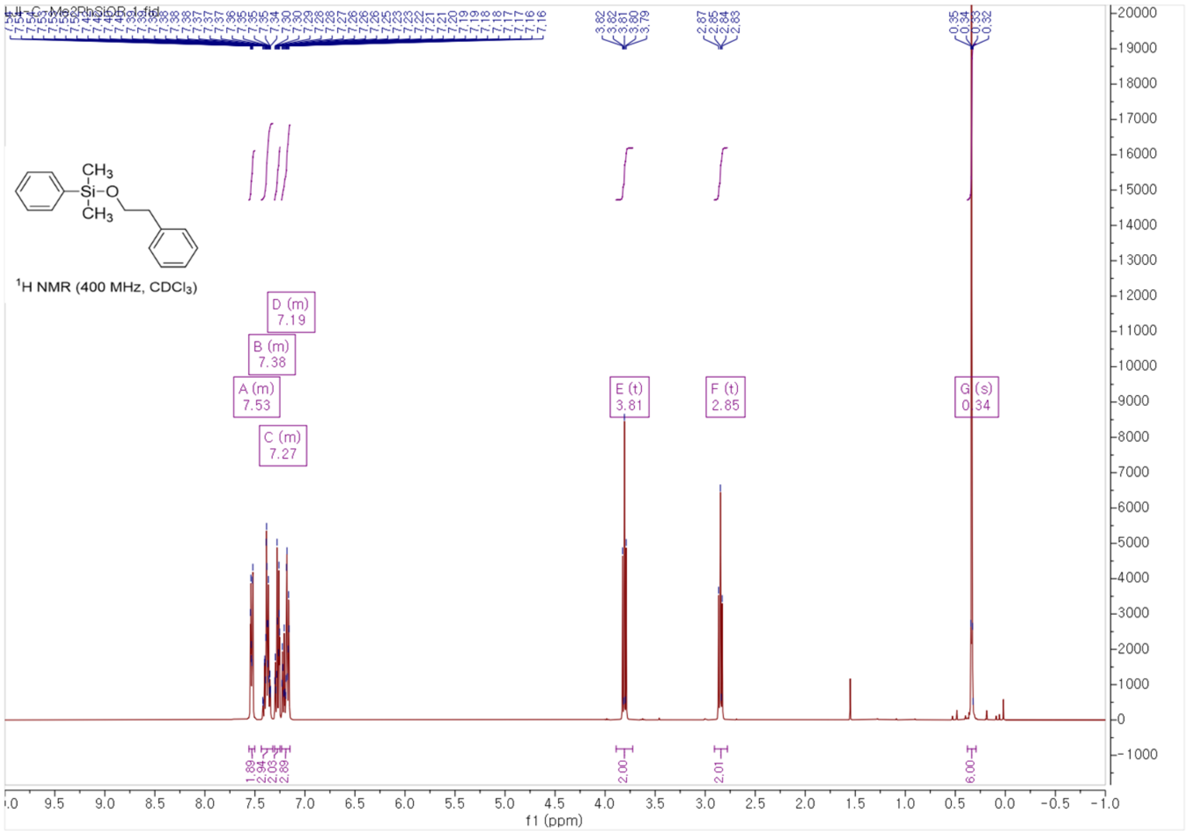
LJI-C-646 Me2PhSiOR pdt.2.fid
138.90
136.03
134.37
129.81
129.19
128.28
127.86
126.20
64.73
39.32
-3.13
$H_3C-Si-O$
^{13}C NMR (101 MHz , $CDCl_3$)
f1 (ppm)

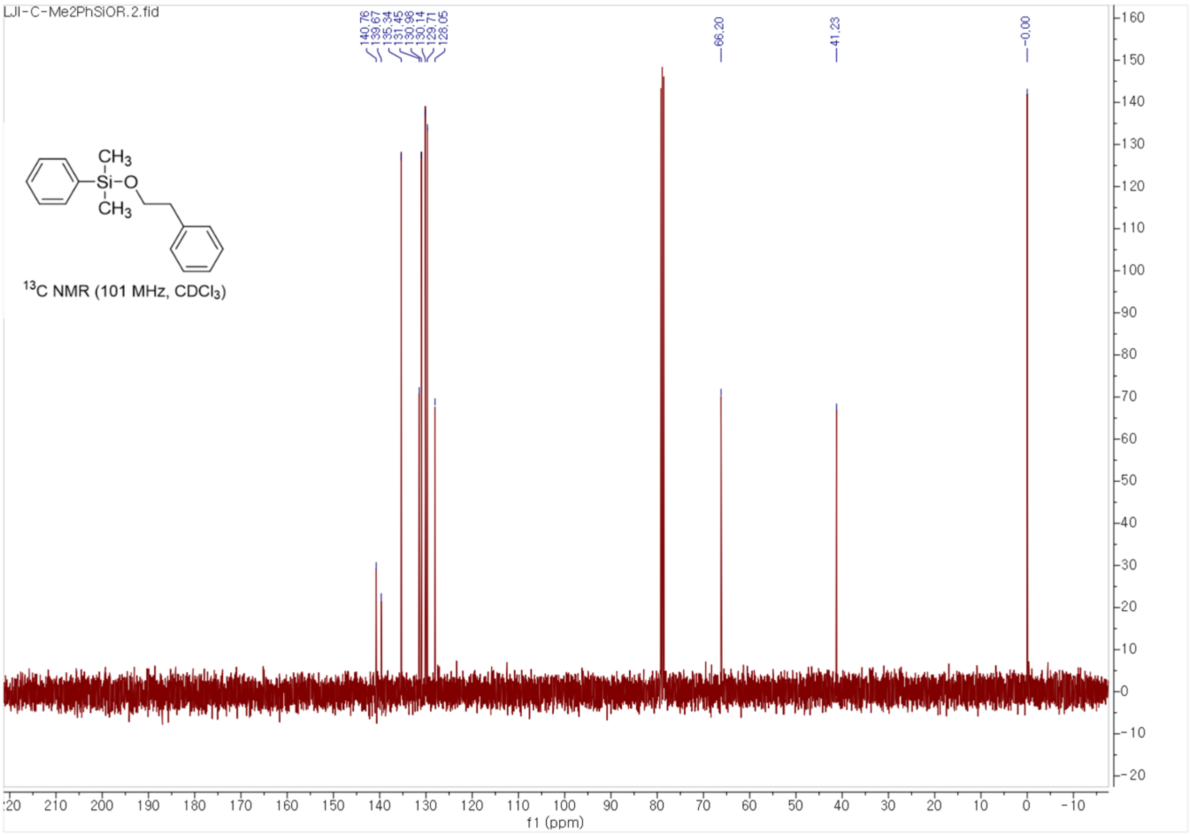


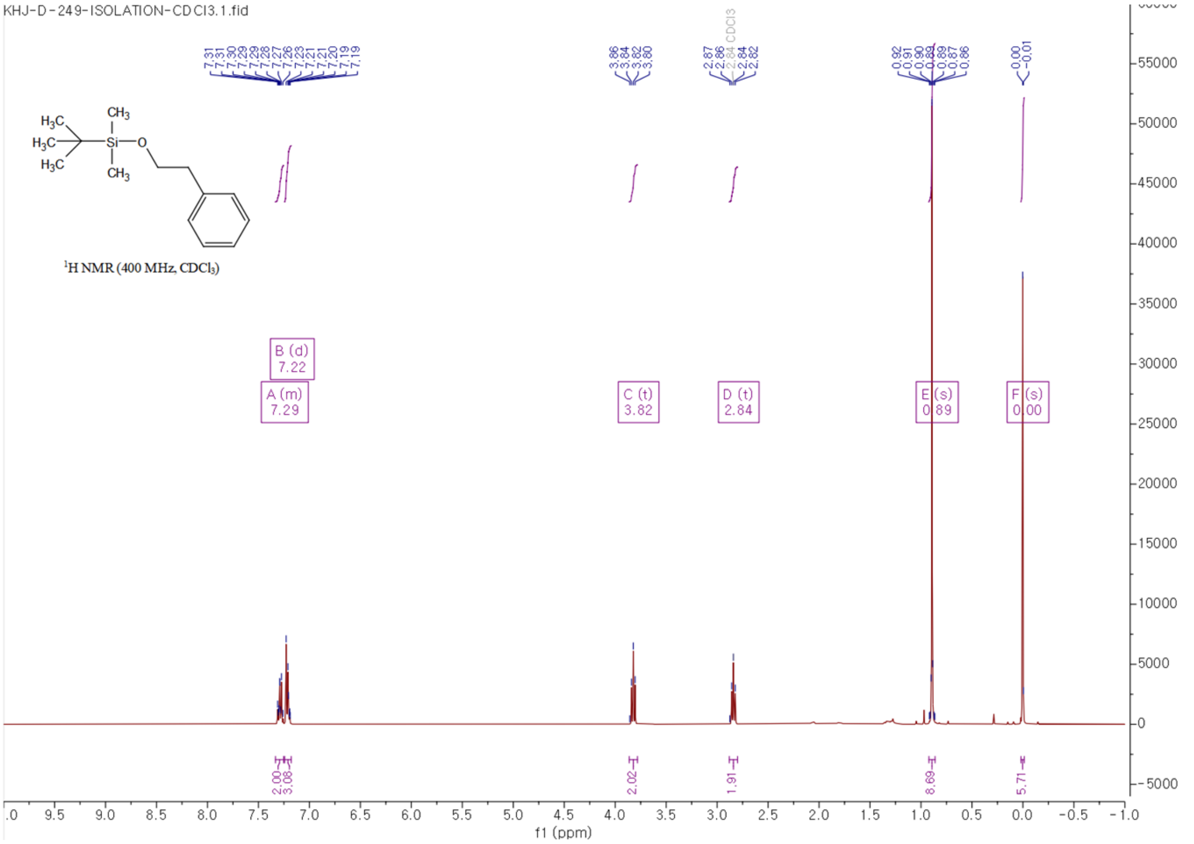
LJI-C-Ph3SiOR.1.fid
7.44
7.44
7.42
7.42
7.41
7.41
7.40
7.38
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.33
7.26
7.26
7.25
7.25
7.24
7.24
7.23
7.22
7.22
7.22
7.20
7.20
7.20
7.19
7.18
7.17
7.14
7.14
7.13
7.13
7.12
7.12
4.00
3.98
3.96
2.90
2.88
2.86
Si
O
^{1}H NMR (400 MHz, CDCl$_3$)
D (m) 7.22
B (m) 7.42
A (m) 7.57
C (m) 7.36
E (m) 7.13
F (t) 3.98
G (t) 2.88
5.99
2.87
6.25
3.19
1.98
2.00
2.03
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

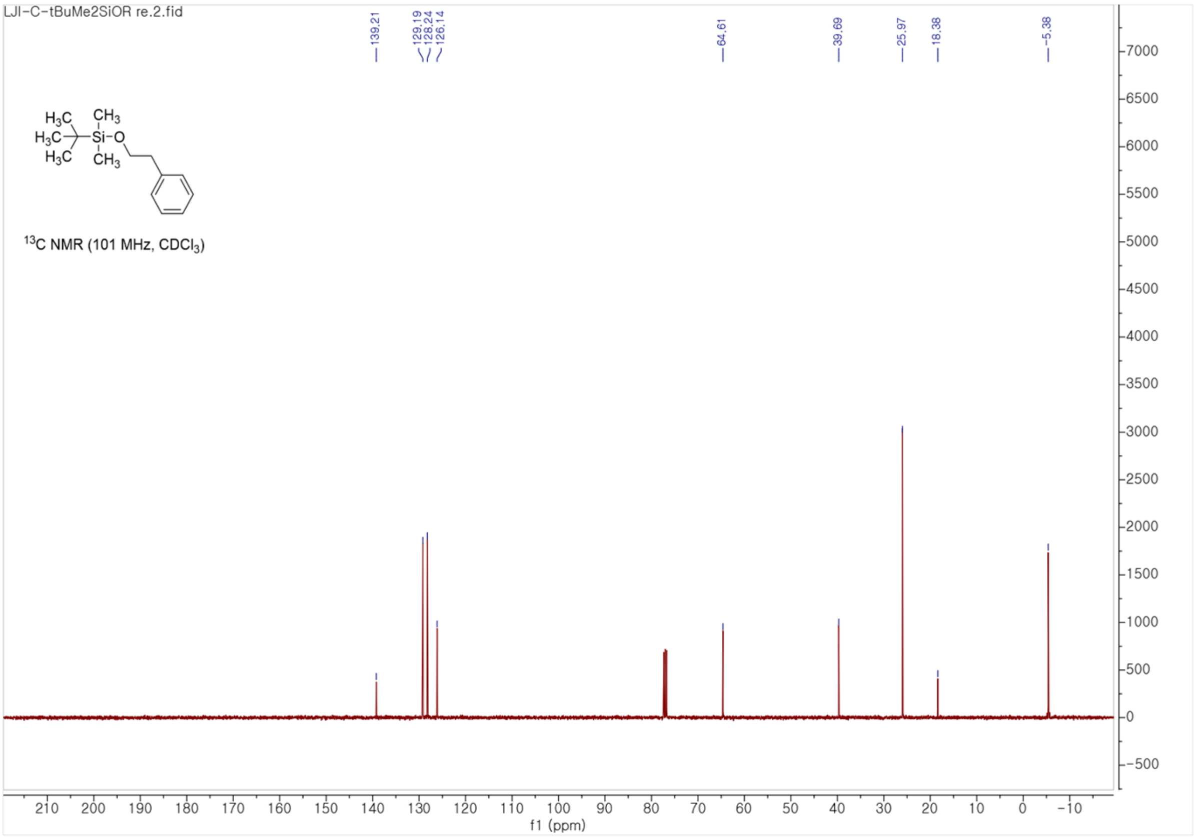


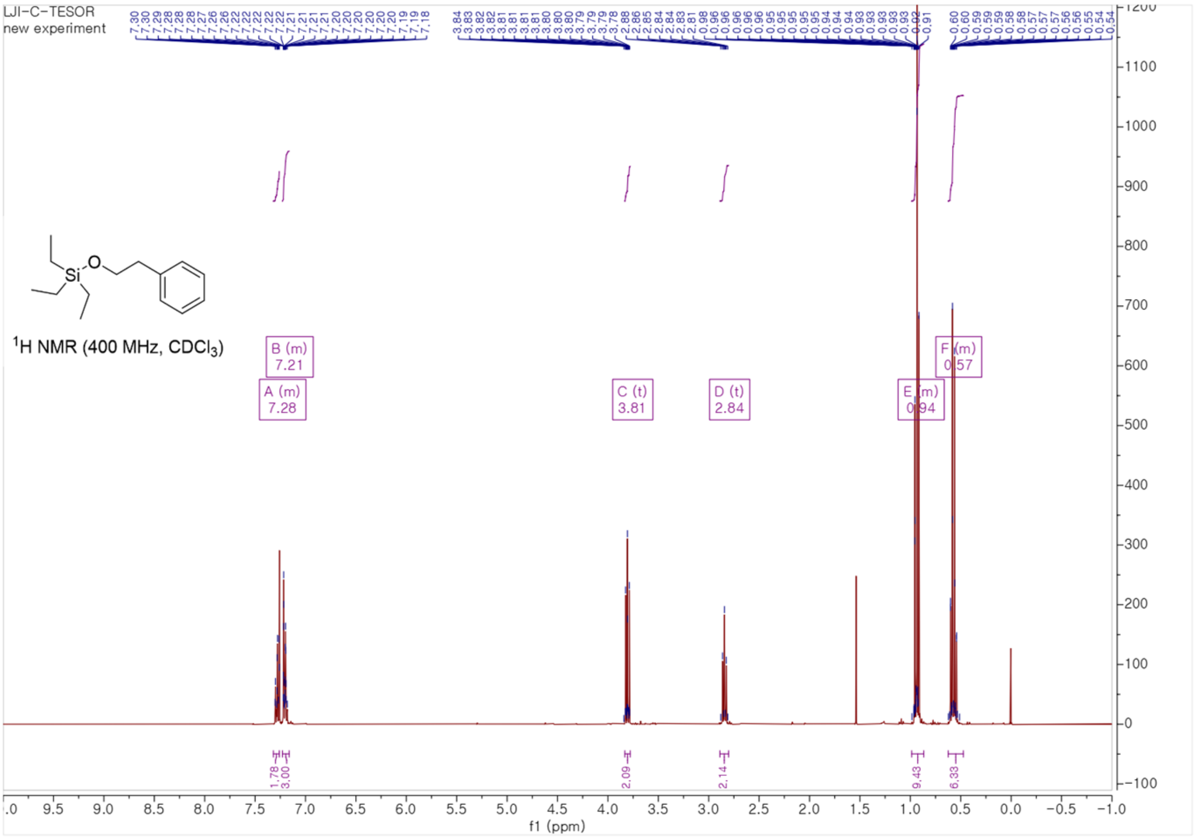
LJI-C-Ph3SiOR.2.fid
138.82
135.39
134.19
130.00
129.19
128.26
127.86
126.16
65.07
39.24
Si
O
^{13}C NMR (101 MHz, CDCl$_3$)
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

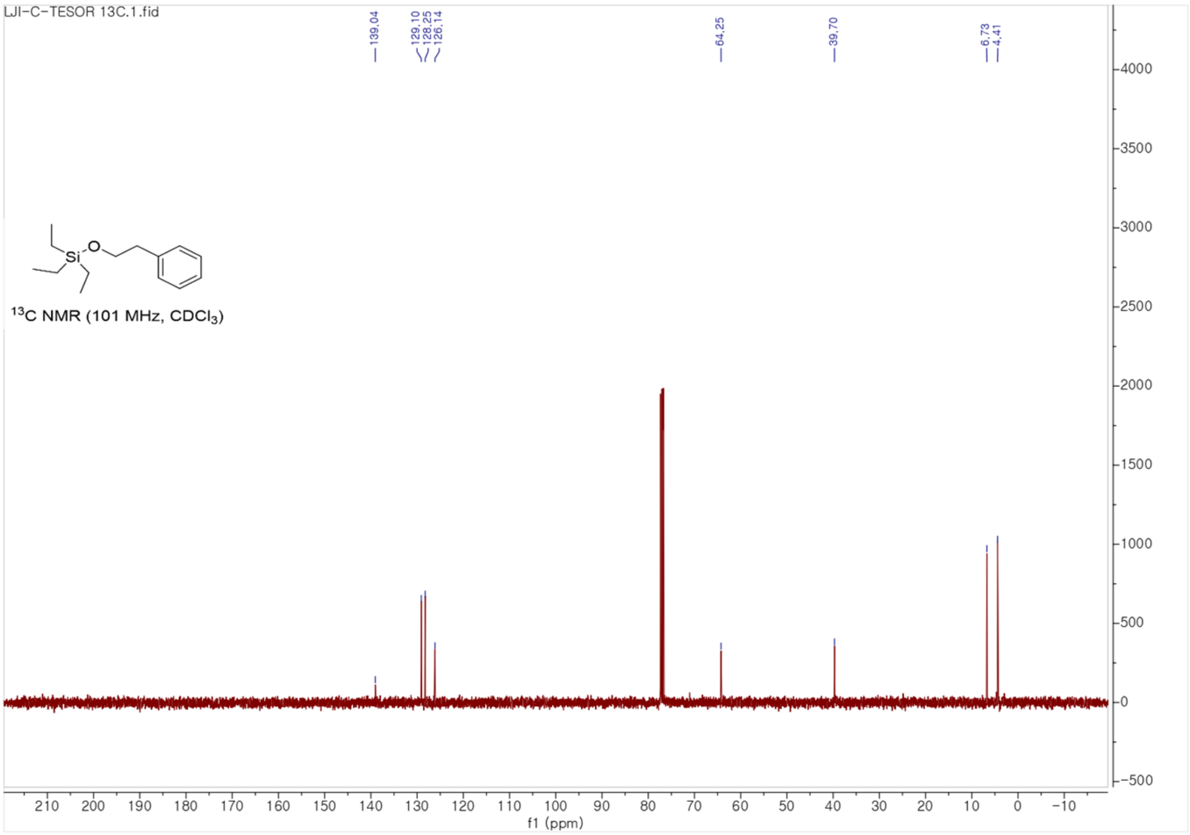

LJI-C-Me2PhSiOR.1.fid
7.39 7.39 7.38 7.38 7.38 7.38 7.37 7.37 7.37 7.36 7.35 7.35 7.35 7.34 7.30 7.30 7.29 7.28 7.28 7.27 7.26 7.26 7.26 7.25 7.23 7.23 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.18 7.17 7.17 7.16 7.16
3.82 3.82 3.81 3.80 3.79
2.87 2.85 2.84 2.83
0.35 0.34 0.33 0.32
CH3
Si
O
CH3
^{1}H NMR (400 MHz, CDCl$_3$)
D (m) 7.19
B (m) 7.38
A (m) 7.53
C (m) 7.27
E (t) 3.81
F (t) 2.85
G (s) 0.34
1.89 2.94 2.03 2.89
2.00
2.01
6.00
f1 (ppm)

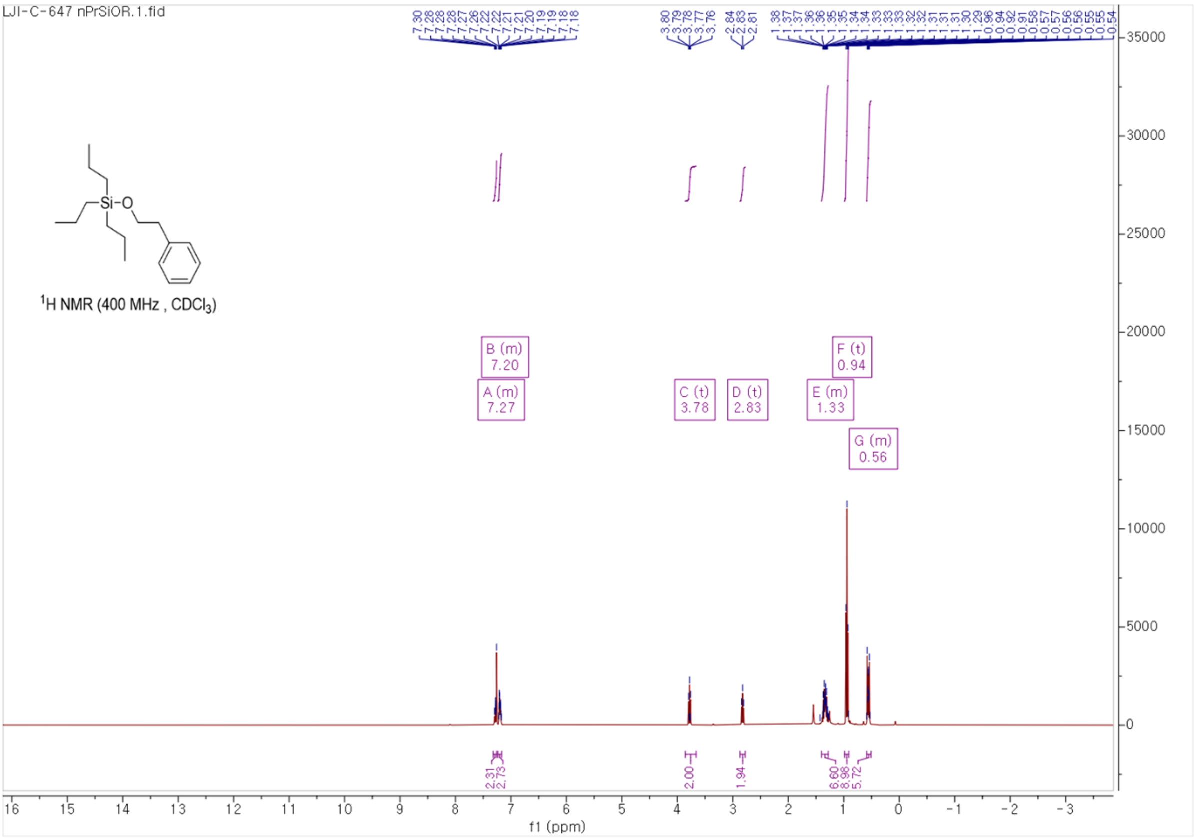

LJI-C-Me2PhSiOR.2.fid
140.76 139.67 135.34 131.45 130.98 130.14 129.71 128.05
66.20
41.23
-0.00
CH3
Si
O
CH3
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

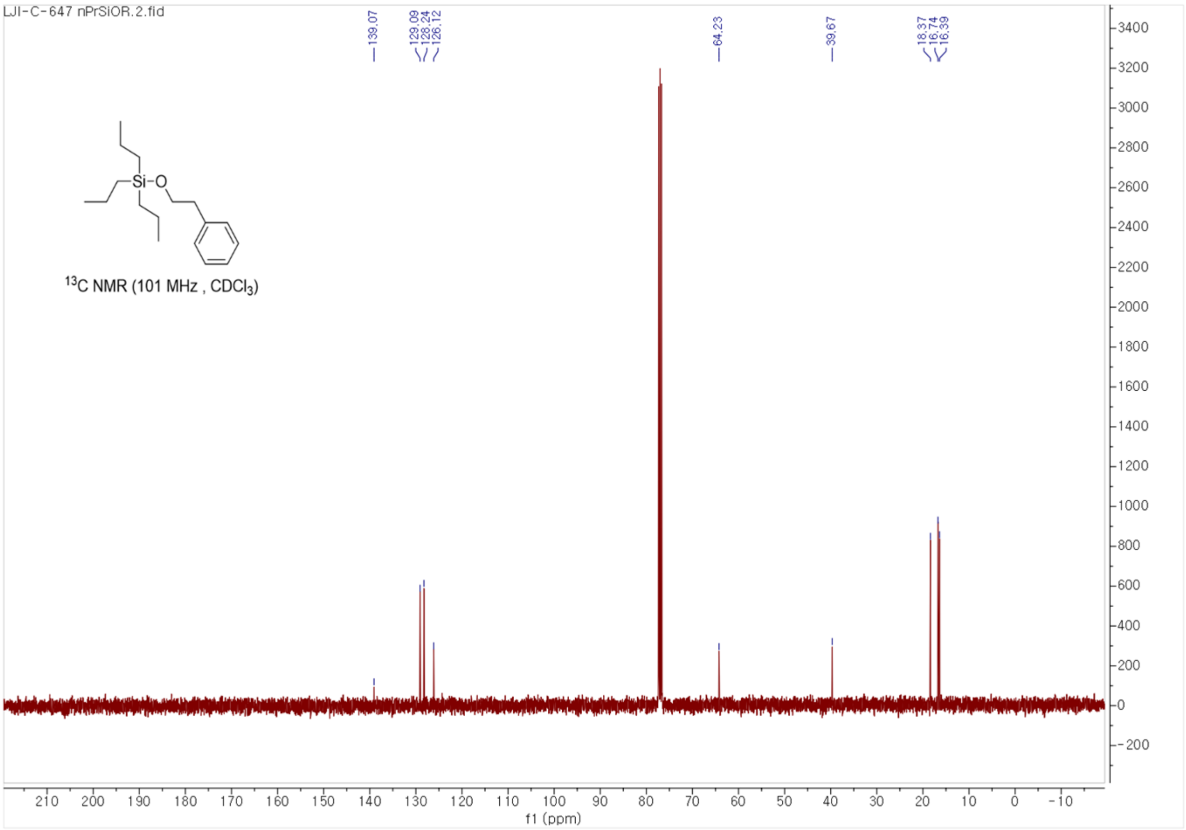

KHJ-D-249-ISOLATION-CDCl3.1.fid
7.31 7.31 7.30 7.29 7.28 7.27 7.26 7.23 7.21 7.21 7.20 7.19 7.19
3.86 3.84 3.82 3.80
2.87 2.86 2.84 CDCl3 2.84 2.82
0.92 0.91 0.90 0.89 0.89 0.89 0.87 0.86
0.00 −0.01
H3C H3C H3C CH3 Si O CH3
1H NMR (400 MHz, CDCl3)
B (d) 7.22
A (m) 7.29
C (t) 3.82
D (t) 2.84
E (s) 0.89
F (s) 0.00
2.00 3.08
2.02
1.91
8.69
5.71
f1 (ppm)

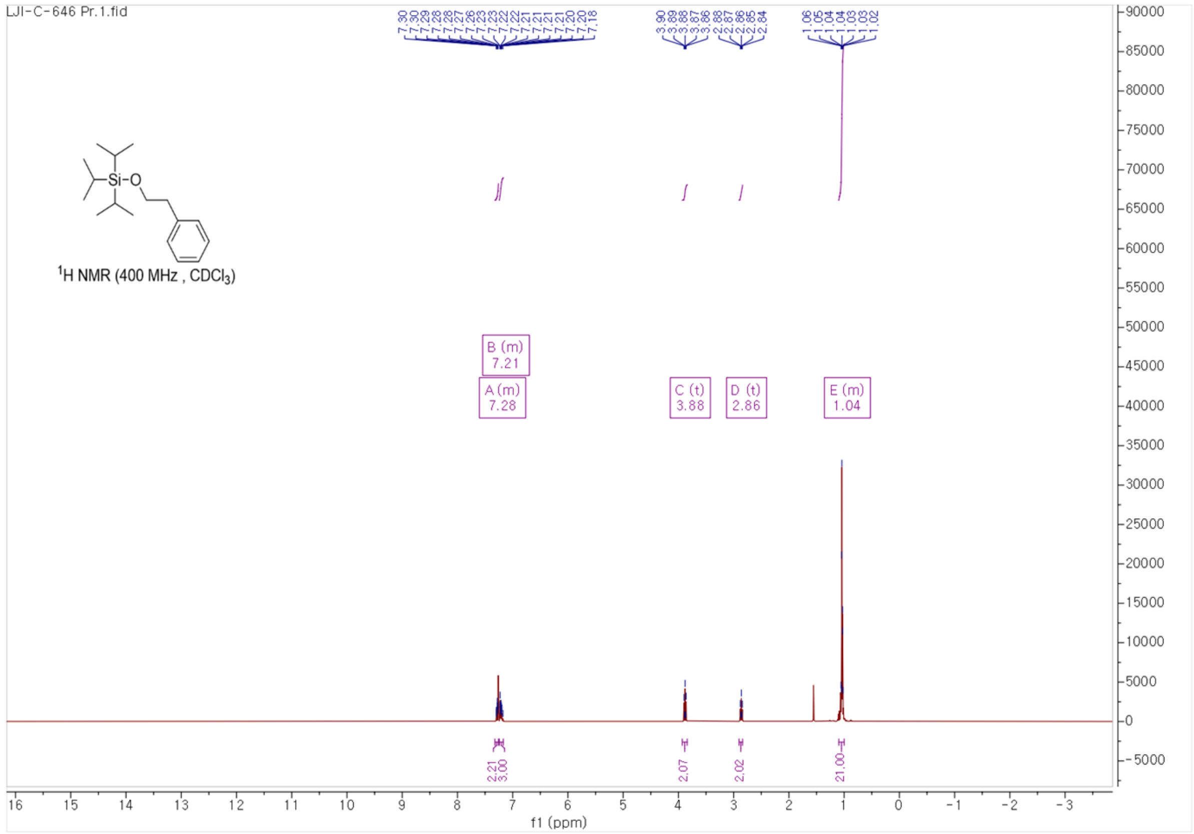

LJI-C-tBuMe2SiOR re.2.fid
139.21
129.19 128.24 126.14
64.61
39.69
25.97
18.38
−5.38
H3C H3C H3C CH3 Si O CH3
13C NMR (101 MHz, CDCl3)
f1 (ppm)

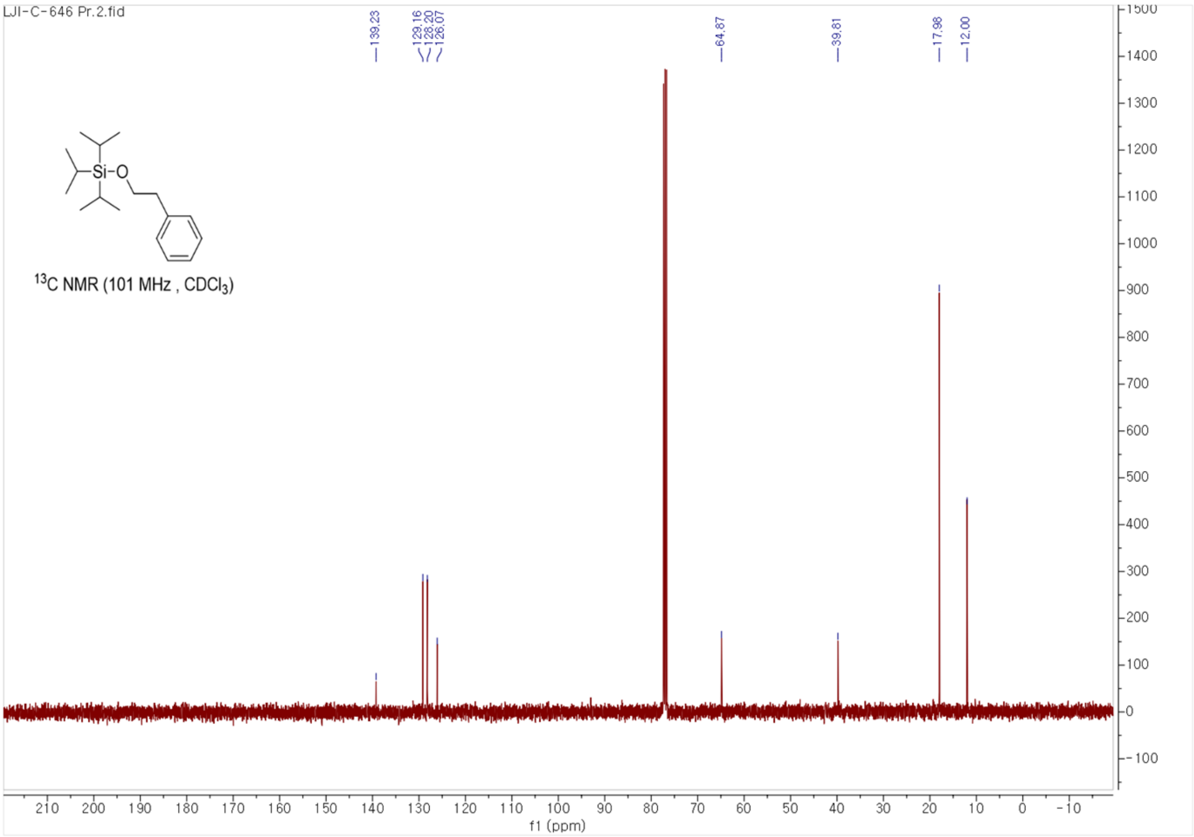

LJI-C-TESOR
new experiment
7.30 7.30 7.29 7.28 7.28 7.28 7.27 7.26 7.26 7.22 7.22 7.22 7.22 7.22 7.21 7.21 7.21 7.21 7.20 7.20 7.20 7.20 7.20 7.20 7.19 7.19 7.18
3.84 3.83 3.82 3.81 3.81 3.81 3.81 3.80 3.80 3.80 3.79 3.79 3.79 3.78
2.88 2.86 2.85 2.84 2.84 2.83 2.81
0.98 0.96 0.96 0.96 0.96 0.96 0.95 0.95 0.95 0.95 0.94 0.94 0.94 0.93 0.93 0.93 0.93 0.92 0.91
0.60 0.60 0.59 0.59 0.59 0.58 0.58 0.57 0.57 0.57 0.56 0.56 0.55 0.54 0.54
Si
O
^{1}H NMR (400 MHz, CDCl$_3$)
B (m) 7.21
A (m) 7.28
C (t) 3.81
D (t) 2.84
E (m) 0.94
F (m) 0.57
1.78
3.00
2.09
2.14
9.43
6.33
f1 (ppm)

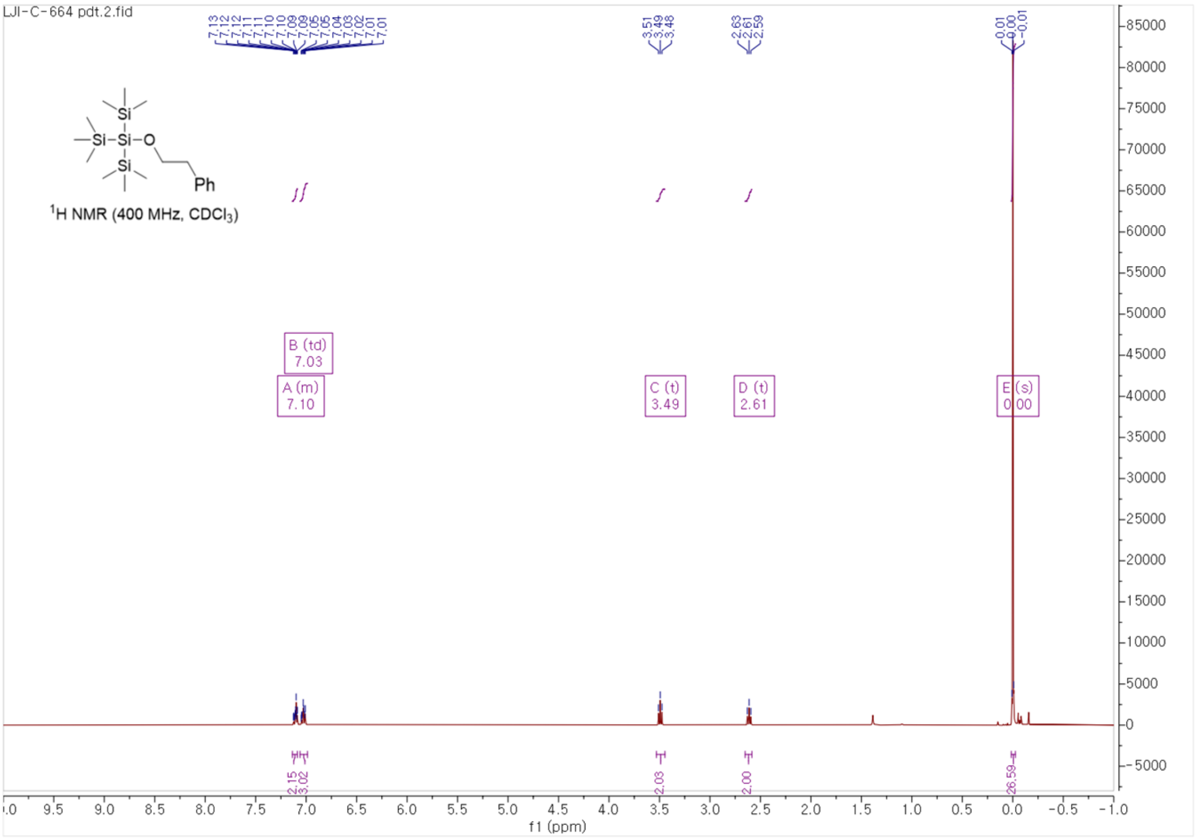

LJI-C-TESOR 13C.1.fid
139.04
129.10
128.25
126.14
64.25
39.70
6.73
4.41
Si
O
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

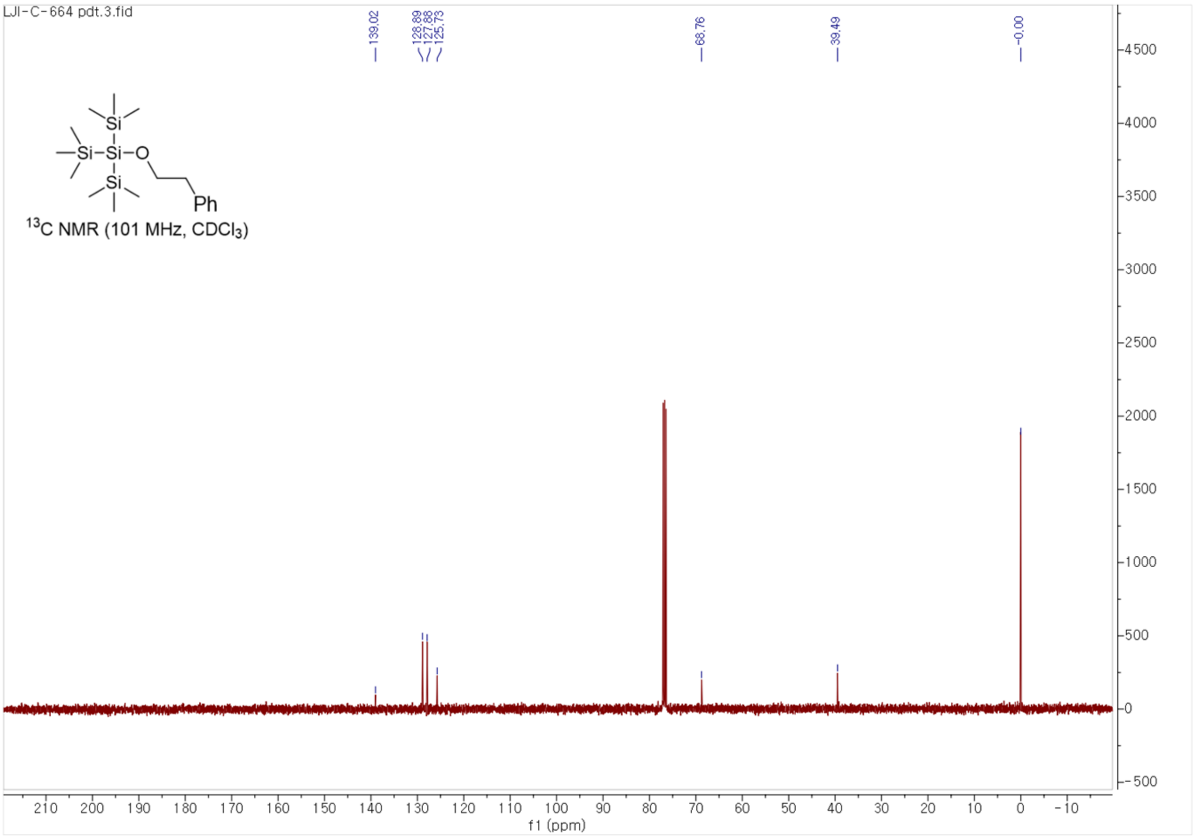

LJI-C-647 nPrSiOR.1.fid
7.30 7.28 7.28 7.28 7.27 7.26 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.18
3.80 3.79 3.78 3.77 3.76
2.84 2.83 2.81
1.38 1.37 1.37 1.36 1.36 1.35 1.35 1.34 1.34 1.33 1.33 1.33 1.32 1.32 1.31 1.31 1.31 1.30 1.29 0.96 0.94 0.92 0.91 0.58 0.57 0.57 0.56 0.56 0.55 0.55 0.54
Si
O
^{1}H NMR (400 MHz , CDCl$_3$)
B (m) 7.20
A (m) 7.27
C (t) 3.78
D (t) 2.83
E (m) 1.33
F (t) 0.94
G (m) 0.56
2.31
2.73
2.00
1.94
6.60
8.98
5.72
35000
30000
25000
20000
15000
10000
5000
0
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


LJI-C-647 nPrSiOR.2.fid
139.07
129.09
128.24
126.12
64.23
39.67
18.37
16.74
16.39
Si
O
^{13}C NMR (101 MHz , CDCl$_3$)
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


LJI-C-646 Pr.1.fid
7.30
7.30
7.29
7.28
7.28
7.27
7.26
7.23
7.23
7.22
7.22
7.21
7.21
7.21
7.21
7.20
7.20
7.18
3.90
3.89
3.88
3.87
3.86
2.88
2.87
2.86
2.85
2.84
1.06
1.05
1.04
1.04
1.03
1.03
1.02
Si
O
^{1}H NMR (400 MHz , CDCl$_3$)
B (m) 7.21
A (m) 7.28
C (t) 3.88
D (t) 2.86
E (m) 1.04
2.21
3.00
2.07
2.02
21.00
90000
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


LJI-C-646 Pr.2.fid
139.23
129.16
128.20
126.07
64.87
39.81
17.98
12.00
Si
O
^{13}C NMR (101 MHz , CDCl$_3$)
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


LJI-C-664 pdt.2.fid
7.13
7.12
7.12
7.11
7.11
7.10
7.10
7.09
7.09
7.05
7.05
7.04
7.03
7.02
7.01
7.01
3.51
3.49
3.48
2.63
2.61
2.59
0.01
0.00
-0.01
Si
Si
Si
O
Si
Ph
^{1}H NMR (400 MHz, CDCl$_3$)
B (td)
7.03
A (m)
7.10
C (t)
3.49
D (t)
2.61
E (s)
0.00
2.15
3.02
2.03
2.00
26.59
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


LJI-C-664 pdt.3.fid
139.02
128.89
127.88
125.73
68.76
39.49
-0.00
Si
Si
Si
O
Si
Ph
^{13}C NMR (101 MHz, CDCl$_3$)
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

5. Reference

1. D. J. Meininger, Z. Kasrawi, H. D. Arman and Z. J. Tonzetich, Synthesis of tetraphenylporphyrinate manganese(III) siloxides by silyl group transfer from silanethiols, *J. Coord. Chem.*, **2016**, *69*, 1970-1978.