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Supporting information

Electrochemically Driven Silicon-Carbon Bond Cleavage of

Silacyclobutanes: A Transition Metal-Free Approach

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1. General Information

All reagents were purchased from Macklin, Sigma-Aldrich, Adamas-beta, and Energy Chemical of the highest purity grade and used without further purification, unless otherwise indicated. Analytical thin-layer chromatography (TLC) was performed on hexane, visualized by irradiation with UV light. For column chromatography, 300-400 mesh silica gel was used. ¹H-NMR and¹³C-NMR were recorded on a BRUKER 600 MHz spectrometer in CDCl₃. ¹⁹F-NMR spectra were recorded at BRUKER 400 MHz spectrometer in CDCl₃. The residual solvent peak was used as an internal reference: proton (CDCl₃ δ 7.26) and carbon (CDCl₃ δ 77.16). Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, dd = double doublet, dt = double triplet, m = multiplet), coupling constants (*J*) are in Hertz (Hz). HRMS spectra were recorded on a Xevo G2-XS QTof (Waters Corporation) using electrospray ionization. DC power supply MT-152D was used for all experiments.

2. Experimental Procedures

2.1 Preparation of Silacyclobutanes (SCBs)

Preparation of 1a-1i

To the suspension of magnesium (40 mmol, 4.0 equiv.) and a grain of I₂ in dry THF (50 mL) was added a small amount (about 20 drops) solution of the 3-chloropropyltrichlorosilane (10 mmol, 1.0 equiv.) in THF (20 mL) by syringe at ambient temperature under argon. The reaction was initiated and heated to reflux with addition the rest of the 3-chloropropyltrichlorosilane solution dropwise over 1 h. The resulting mixture was refluxed for additional 4 h before addition of a solution of RBr (22 mmol, 2.2 equiv.) in THF (40 mL) dropwise over 3 h. The resulting mixture was refluxed overnight. The reaction was cooled to room temperature before quenching with aq. HCl (15 mL, 2 M). The mixture was extracted with n-hexane (3×50 mL). The combined organic layers were dried over Na₂SO₄, filtered, and evaporated under reduced pressure. The crude residue was purified by column chromatography on silica gel to afford the silacyclobutanes.

Preparation of 1j-1y, 1ab

To the suspension of magnesium (30 mmol, 3.0 equiv.) and a grain of I₂ in dry THF (50 mL) was added a small amount (about 20 drops) solution of the 3-chloropropylmethyldichlorosilane (10 mmol, 1.0 equiv.) in THF (20 mL) by syringe at ambient temperature under argon. The reaction was initiated and heated to reflux with addition the rest of the THF solution of 3-chloropropylmethyldichlorosilane dropwise over 1 h. The resulting mixture was refluxed for additional 4 h before addition of a solution of the RBr (15 mmol, 1.5 equiv.) in THF (40 mL) dropwise over 3 h. The resulting mixture was refluxed overnight. The reaction was cooled to room temperature before quenching with aq. HCl (15 mL, 2 M). The mixture was extracted with n-hexane (3×50 mL). The combined organic layers were dried over Na₂SO₄, filtered, and evaporated under reduced pressure. The crude residue was purified by column chromatography on silica gel to afford the silacyclobutanes.

Preparation of 1z and 1aa

$$H_{\text{Br}} + \frac{R_1}{R_2} CI \qquad \frac{\text{Mg (4.0 eq), I}_2}{\text{Et}_2 \text{O, reflux, 24 h}} \rightarrow \frac{\text{Si}-R}{R_2}$$

To a suspension of magnesium (1.92 g, 80 mmol) and a grain of I₂ in dry Et₂O (2.0 mL), the 2-bromobenzyl bromide solution (2-bromobenzyl bromide (5.0 g, 20 mmol) dissolved in 20.0 mL Et₂O) and dichlorodialkylsilane or dichlorodiarylsilane were added dropwise simultaneously. Then the reaction mixture was refluxed for 24 h. After cooling to room temperature, the reaction was quenched with water and extracted with Et₂O (3×30 mL). The combined organic phase was washed with water and brine, and dried over anhydrous Na₂SO₄. After removal of the solvent under reduced pressure, the residue was purified by silica gel flash column chromatography to afford the corresponding benzosilacyclobutanes.

All silacyclobutanes are known compounds and were prepared according to reported procedures. ^[1-12]

Ph Ph Si + 1a	MeOH MeOH ⁿ Bu ₄ NI, MeCN, NEt ₃ 16 mA, 30 min	Ph Si-"Pr Ph 2a
Entry	Electrode	Yield (%) b
1	GF (+) GF (-)	89
2	Mg (+) GF (-)	70
3	Zn (+) GF (-)	Trace
4	Cu (+) GF (-)	62
5	GF (+) Pt (-)	79
6	Pt (+) Pt (-)	66
7	SST (+) Pt (-)	68
8	carbon cloth $(+)$ carbon cloth $(-)$	69

2.2 Experimental Optimization

Table S1. Screening of electrode^a

^a Unless otherwise noted, the conditions were as follows: **electrode**, constant current = 16 mA, **1a** (0.2 mmol), MeOH (0.4 mL), MeCN (4.0 mL), NEt₃ (0.5 equiv.), "Bu₄NI (1.0 equiv.), room temperature, 30 min, undivided cell, N₂.^{*b*} yield of isolated product.

Ph Ph ⁻ Si + 1a	MeOH GF(+) GF(-) electrolyte MeCN, NEt ₃ 16 mA, 30 min	Ph. OMe Si− ⁿ Pr Ph 2a
Entry	Electrolyte	Yield (%) b
1	ⁿ Bu ₄ NBF ₄	64
2	"Bu4NCl	Trace
3	"Bu4NBr	45
4	"Bu4NOAc	Trace

Table S2. Screening of electrolyte ^a

^a Unless otherwise noted, the conditions were as follows: graphite felt anode (10 mm \times 15 mm \times 0.3 mm), graphite felt cathode (10 mm \times 15 mm \times 0.3 mm), constant current = 16 mA, **1a** (0.2 mmol), MeOH (0.4 mL), MeCN (4.0 mL), NEt₃ (0.5 equiv.), **electrolyte** (1.0 equiv.), room temperature, 30 min, undivided cell, N₂. ^{*b*} yield of isolated product.

Table S3. Screening of solvent^a

Ph _{Ph} Si + Ph ⁻ Si 1a	GF(+) GF(-) ™Bu₄NI, NEt₃ solvent 16 mA, 30 min	Ph.Si- ⁿ Pr Ph 2a
Entry	Solvent	Yield (%) b
1	DMSO	36
2	DMF	58
3	DMA	54
4	THF	66
5	NMP	47
6	MeOH	Trace

^a Unless otherwise noted, the conditions were as follows: graphite felt anode (10 mm \times 15 mm \times 0.3 mm), graphite felt cathode (10 mm \times 15 mm \times 0.3 mm), constant current = 16 mA, **1a** (0.2 mmol), MeOH (0.4 mL), **solvent** (4.0 mL), NEt₃ (0.5 equiv.), "Bu₄NI (1.0 equiv.), room temperature, 30 min, undivided cell, N₂.^{*b*} yield of isolated product.

Table S4. Screening of current^a

Ph Ph ^{>} Si 1a	+ MeOH	GF(+) GF(-) [™] Bu₄NI, MeCN, NEt ₃ current 30 min	Ph, OMe Si−″Pr Ph' 2a
Entry		Current	Yield (%) b
1		8 mA	60
2		12 mA	66
3		No electricity	N.R.

^a Unless otherwise noted, the conditions were as follows: graphite felt anode (10 mm \times 15 mm \times 0.3 mm), graphite felt cathode (10 mm \times 15 mm \times 0.3 mm), **constant current**, **1a** (0.2 mmol), MeOH (0.4 mL), MeCN (4.0 mL), NEt₃ (0.5 equiv.), "Bu₄NI (1.0 equiv.), room temperature, 30 min, undivided cell, N₂.^{*b*} yield of isolated product. N. R. = no reaction.

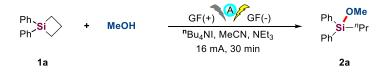
Table S5. Screening of base ^a

Ph ² V	MeOH GF(+) GF(-) ⁿ Bu ₄ NI, MeCN base	Ph, OMe Si− ⁿ Pr Ph
1a	16 mA, 30 min	2a
Entry	Base	Yield (%) <i>b</i>
1	w/o NEt ₃	83
2	CsCO ₃	40
3	DABCO	66
4	DBU	41

^a Unless otherwise noted, the conditions were as follows: graphite felt anode (10 mm \times 15 mm \times 0.3 mm), graphite felt cathode (10 mm \times 15 mm \times 0.3 mm), constant current = 16 mA, **1a** (0.2 mmol), MeOH (0.4 mL), MeCN (4.0 mL), **base** (0.5 equiv.), "Bu₄NI (1.0 equiv.), room temperature, 30 min, undivided cell, N₂.^{*b*} yield of isolated product.

3. General procedure for the synthesis of products

3.1 Synthesis of silicon ether by electrochemically induced Si-C bond fracture in silicon ring



Under N₂ atmosphere, 1,1-diphenylsiletane **1a** (44.8 mg. 0.2 mmol), MeOH (0.4 mL), NEt₃ (0.5 equiv.), "Bu₄NI (1.0 equiv.) and MeCN (4.0 mL) were added in a boiling flask-2-neck (10.0 mL). The flask was equipped with graphite felt (10 mm \times 15 mm \times 0.3 mm) as anode and graphite felt (10 mm \times 15 mm \times 0.3 mm) as cathode. Then the reaction mixture was stirred and electrolyzed at a constant current of 16 mA for 30 min. When the reaction was finished, the mixture extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄. Part of the organics was concentrated and the crude residue was purified by preparative TLC on silica gel to afford the product.

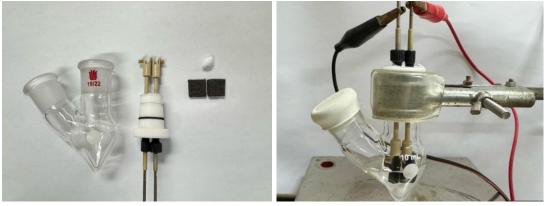
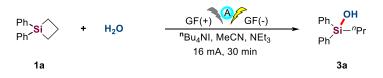


Figure S1. Electrolysis setup for the small scale experiment.

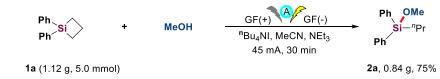
3.2 Synthesis of silanol by electrochemically induced Si-C bond fracture in silicon ring



Under N₂ atmosphere, 1,1-diphenylsiletane **1a** (44.8 mg. 0.2 mmol), H₂O (0.4 mL), NEt₃ (0.5 equiv.), "Bu₄NI (1.0 equiv.) and MeCN (4.0 mL) were added in a boiling flask-2-neck (10.0 mL). The flask was equipped with graphite felt (10 mm \times 15 mm \times 0.3 mm) as anode and graphite felt (10 mm \times 15 mm \times 0.3 mm) as cathode. Then the reaction mixture was stirred and electrolyzed at a constant current of 16 mA for 30 min. When the reaction was finished, the mixture extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄. Part of the organics was concentrated and the crude residue was purified by preparative TLC on silica gel to afford the product.

3.3 Gram-scale synthesis

3.3.1 Gram-scale synthesis of alkoxysilane 2



Under N₂ atmosphere, 1,1-diphenylsiletane **1a** (1.12g. 5 mmol), MeOH (1.2 mL), NEt₃ (50.0 μ L) and MeCN (20.0 mL) were added in a bottle (50.0 mL). The bottle was equipped with graphite felt (40 mm × 40 mm × 0.3 mm) as anode and graphite felt (40 mm × 40 mm × 0.3 mm) as cathode. Then the reaction mixture was stirred and electrolyzed at a constant current of 45 mA for 30 min. When the reaction was finished, the mixture extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄. Part of the organics was concentrated and the crude residue was purified by column chromatography on silica gel (*n*-hexane/EtOAc = 40: 1, v/v) to afford the product **2a**.

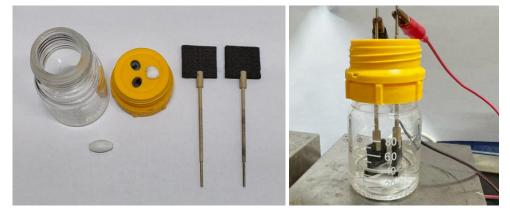
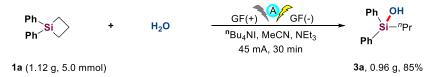


Figure S2. Electrolysis setup for the gram scale experiment.

3.3.2 Gram-scale synthesis of silanol 32



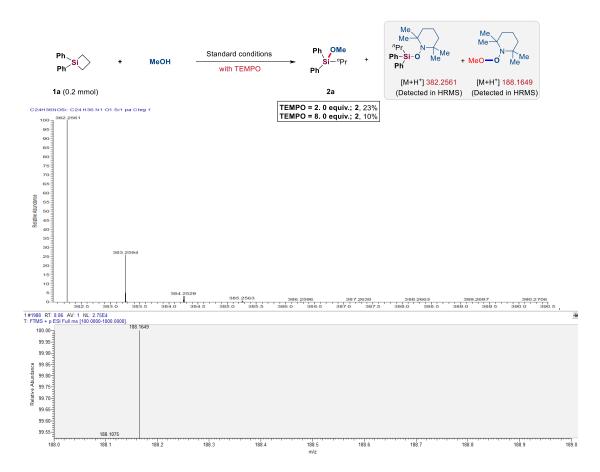
Under N₂ atmosphere, 1,1-diphenylsiletane **1a** (1.12g. 5 mmol), H₂O (1.2 mL), NEt₃ (50.0 μ L) and MeCN (20.0 mL) were added in a bottle (50.0 mL). The bottle was equipped with graphite felt (40 mm ×40 mm × 0.3 mm) as anode and graphite felt (40 mm ×40 mm × 0.3 mm) as cathode. Then the reaction mixture was stirred and electrolyzed at a constant current of 45 mA for 30 min. When the reaction was finished, the mixture extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄. Part of the organics was concentrated and the crude residue was purified by column chromatography on silica gel (*n*-hexane/EtOAc = 10: 1, v/v) to afford the product **3a**.

4. Mechanistic studies

4.1 radical trapping experiments ^a

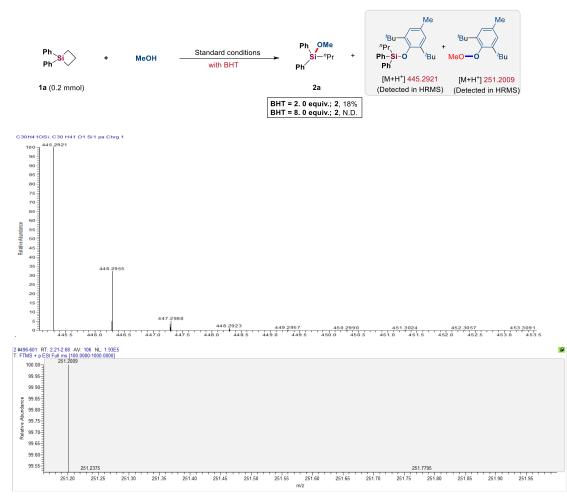
Ph Ph `si〉 +	MeOH standard conditions radical scavenger	Ph, OMe Si−″Pr Ph
1a		2a
Entry	Radical scavenger	Yield (%) ^b
1	none	88
2	TEMPO (2.0 equiv.) 23	
3	TEMPO (8.0 equiv.) 10	
4	BHT (2.0 equiv.) 18	
5	BHT (8.0 equiv.)	N.D.

^{*a*} Standard reaction conditions: graphite felt anode (10 mm \times 15 mm \times 0.3 mm), graphite felt cathode (10 mm \times 15 mm \times 0.3 mm), constant current = 16 mA, **1a** (0.2 mmol), MeOH (0.4 mL), MeCN (4.0 mL), NEt₃ (0.5 equiv.), ^{*n*}Bu₄NI (1.0 equiv.), room temperature, 30 min, undivided cell, N₂. ^{*b*} yield of isolated product. N. D. = not detected.



An undivided cell (10 mL pearbottle) was equipped with a magnet stirrer (3 mm x 5 mm), graphite felt (10 mm \times 15 mm \times 0.3 mm) as cathode, and graphite felt (10 mm \times 15 mm \times 0.3 mm) as anode, and then evacuated and refilled with nitrogen gas for 3 cycles. The electrolyte "Bu₄NI (73.9 mg, 0.2 mmol, 1.0 equiv.), MeCN (4.0 mL), followed by the addition of the substrate **1a** (44.8 mg, 0.2 mmol), MeOH (0.4 mL), a solution of NEt₃ (0.5 equiv.) and 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO). The reaction mixture was then stirred (1000 rpm) and electrolyzed at a constant current of 16 mA flow for 30 min at room temperature. The aqueous layer extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄.

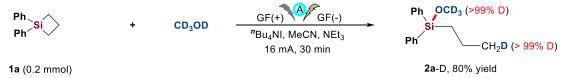
When TEMPO was introduced into the model reactions, the desired 1,1-diphenylsiletane was isolated in 23% yield after column chromatography separation. Then the TEMPO was improving to 8.0 equiv. at the standard condition the yield of **2a** reduced from 23% to 10%. These results suggest the involvement of radical process in this transformation. The TEMPO-adduct was detected by HRMS (ESI, m/z), calculated for $[C_{24}H_{35}NOSi + H]^+$: 382.2561; Found: 382.2561; calculated for $[C_{10}H_{22}NO_2Si + H]^+$: 188.1645; Found: 188.1649.



An undivided cell (10 mL pearbottle) was equipped with a magnet stirrer (3 mm x 5 mm), graphite felt (10 mm \times 15 mm \times 0.3 mm) as cathode, and graphite felt (10 mm \times 15 mm \times 0.3 mm) as anode, and then evacuated and re-filled with nitrogen gas for 3 cycles. The electrolyte "Bu₄NI (73.9 mg, 0.2 mmol, 1.0 equiv.), MeCN (4.0 mL), followed by the addition of the substrate **1a** (44.8 mg, 0.2 mmol), MeOH (0.4 mL), a solution of NEt₃ (0.5 equiv.) and 2,6-Di-tert-butyl-4-methylphenol (BHT). The reaction mixture was then stirred (1000 rpm) and electrolyzed at a constant current of 16 mA flow for 30 min at room temperature. The aqueous layer extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄.

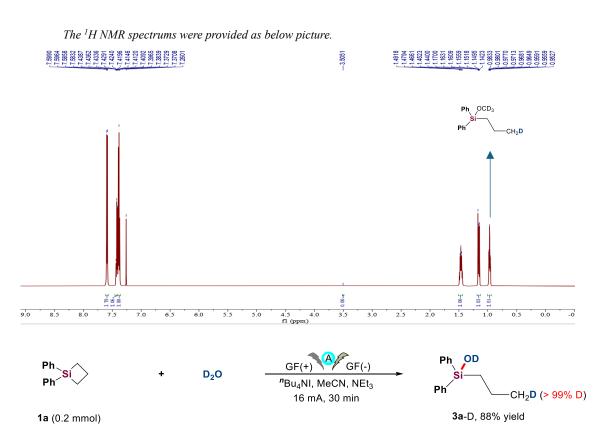
When BHT was introduced into the model reactions, the desired 1,1-diphenylsiletane was isolated in 18% yield after column chromatography separation. Then the BHT was improving to 8.0 equiv. at the standard condition the yield of **2a** reduced from 18% to no product was detected. These results suggest the involvement of radical process in this transformation. The BHT-adduct was detected by HRMS (ESI, m/z), calculated for $[C_{30}H_{40}OSi + H]^+$: 445.2921; Found: 445.2921; calculated for $[C_{16}H_{27}O_2Si + H]^+$: 251.2006; Found: 251.2009.

4.2 D-labeling experiments



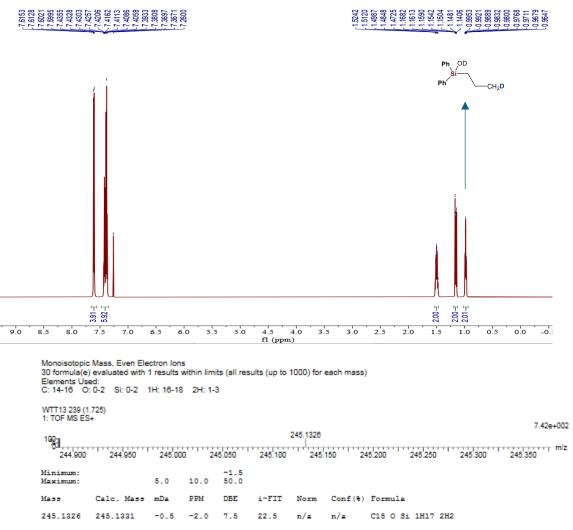
In an oven-dried undivided two-necked bottle (10 mL) equipped with a stir bar, 1,1-diphenylsiletane (1a) (44.8 mg, 0.2 mmol), and "Bu₄NI (1.0 equiv.) were combined and added. The bottle was equipped with graphite felt (10 mm \times 15 mm \times 0.3 mm) as the anode and graphite felt (10 mm \times 15 mm \times 0.3 mm) as the cathode and was then

charged with nitrogen. Under the protection of N₂, CD₃OD (0.8 mL) and MeCN (8.0 mL) were injected respectively into the tubes via syringes. The reaction mixture was stirred and electrolyzed at a constant current of 16 mA at 25°C for 30 min. The aqueous layer extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄. The volatile was removed under reduced pressure and the crude residue was purified by preparative TLC on silica gel using *n*-Hexane/EtOAc (40: 1, v/v) as the eluent to afford the desired product (isolated yield: 80%).



In an oven-dried undivided two-necked bottle (10 mL) equipped with a stir bar, 1,1-diphenylsiletane (1a) (44.8 mg, 0.2 mmol), and "Bu₄NI (1.0 equiv.) were combined and added. The bottle was equipped with graphite felt (10 mm × 15 mm × 0.3 mm) as the anode and graphite felt (10 mm × 15 mm × 0.3 mm) as the cathode and was then charged with nitrogen. Under the protection of N₂, D₂O (0.8 mL) and MeCN (8.0 mL) were injected respectively into the tubes via syringes. The reaction mixture was stirred and electrolyzed at a constant current of 16 mA at 25°C for 30 min. The aqueous layer extracted with EtOAc (3 x 10 mL) and the combined organics were washed with H₂O (10 mL), brine (10 mL) and dried over Na₂SO₄. The volatile was removed under reduced pressure and the crude residue was purified by preparative TLC on silica gel using *n*-Hexane/EtOAc (10: 1, v/v) as the eluent to afford the desired product (isolated yield: 88%). The **3a-D** was detected by HRMS (ESI, m/z), calculated for [C₁₅H₁₆D₂OSi + H] +: 245.1325; Found: 245.1326.

The ¹*H NMR spectrums were provided as below picture.*



5. Characterization



methoxydiphenyl(propyl)silane (2a). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 45.6 mg, 89%. ¹H NMR (600 MHz, CDCl₃) δ 7.60 (dd, *J* = 7.9, 1.5 Hz, 4H), 7.46-7.41 (m, 2H), 7.41-7.38 (m, 4H), 3.55 (s, 3H), 1.52-1.45 (m, 2H), 1.19-1.15 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 135.03, 134.80, 129.94, 127.98,

51.50, 18.31, 16.75, 16.19.; HRMS (ESI): calculated for $C_{16}H_{21}OSi^+$ [M+H]⁺ : 257.1356; found: 257.1358.



OMe

-ⁿPr

OMe

OMe

Si-ⁿPr

Si—ⁿPr

Me

Me

MeO

MeÓ

MeS

MeS

bis(4-(tert-butyl)phenyl)(methoxy)(propyl)silane (2b). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 63.4 mg, 82%. ¹H NMR (600 MHz, CDCl₃) δ 7.54 (d, *J* = 8.2 Hz, 4H), 7.41 (d, *J* = 8.2 Hz, 4H), 3.54 (s, 3H), 1.52-1.46 (m, 2H), 1.33 (s, 18H), 1.15-1.12 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 152.75, 134.71, 131.70, 124.90, 51.52, 34.85, 31.38, 18.41, 16.85, 16.49.; HRMS (ESI): calculated for C₂₄H₃₇OSi⁺[M+H]⁺: 369.2608; found: 369.2608.

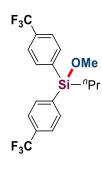
methoxy(propyl)di-p-tolylsilane (2c). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 30.1 mg, 53%. ¹H NMR (600 MHz, CDCl₃) δ 7.49 (d, *J* = 7.8 Hz, 4H), 7.21 (d, *J* = 7.5 Hz, 4H), 3.52 (s, 3H), 2.37 (s, 6H), 1.49-1.42 (m, 2H), 1.14-1.11 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H). ¹³C NMR (151 MHz, CDCl₃) δ 139.81, 134.90, 131.56, 128.81, 51.42, 21.71, 18.34, 16.81, 16.39.; HRMS (ESI): calculated for C₁₈H₂₅OSi⁺ [M+H]⁺: 285.1669; found: 285.1674.

methoxybis(4-methoxyphenyl)(propyl)silane (2d). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 36.7 mg, 58%. ¹H NMR (600 MHz, CDCl₃) δ 7.48 (d, J = 7.9 Hz, 4H), 7.21 (d, J = 8.1 Hz, 4H), 3.51 (s, 3H), 2.37 (s, 6H), 1.48-1.42 (m, 2H), 1.13-1.11 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 139.82, 134.90, 131.55, 128.80, 51.42, 21.71, 18.34, 16.80, 16.38.; HRMS (ESI): calculated for C₁₈H₂₅O₃Si⁺ [M+H]⁺: 317.1567; found: 317.1568.

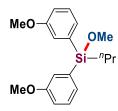
methoxybis(4-(methylthio)phenyl)(propyl)silane (2e). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 61.9 mg, 89%. ¹H NMR (600 MHz, CDCl₃) δ 7.47 (d, J = 8.2 Hz, 4H), 7.25 (d, J = 8.0 Hz, 4H), 3.51 (s, 3H), 2.49 (s, 6H), 1.48-1.41 (m, 2H), 1.13-1.09 (m, 2H), 0.98 (t, J = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 141.02, 135.16, 130.74, 125.52, 51.42, 18.28, 16.73, 16.18, 15.23.; HRMS (ESI): calculated for C₁₈H₂₅OS₂Si⁺ [M+H]⁺ : 349.1111; found: 349.1111.



bis(4-chlorophenyl)(methoxy)(propyl)silane (2f). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 43.6 mg, 67%. ¹H NMR (600 MHz, CDCl₃) δ 7.48 (d, J = 8.2 Hz, 4H), 7.37 (d, J = 8.2 Hz, 4H), 3.51 (s, 3H), 1.45- 1.40 (m, 2H), 1.14- 1.11 (m, 2H), 0.98 (t, J = 7.2 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 136.59, 136.07, 132.96, 128.42, 51.49, 18.21, 16.64, 15.95.; HRMS (ESI): calculated for C₁₆H₁₉Cl₂OSi⁺ [M+H]⁺ : 325.0577; found: 325.0577.



methoxy(propyl)bis(4-(trifluoromethyl)phenyl)silane (2g). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 48.6 mg, 62%. ¹H NMR (600 MHz, Chloroform-*d*) δ 7.68 (d, J = 7.8 Hz, 4H), 7.64 (d, J = 7.9 Hz, 4H), 3.56 (s, 3H), 1.48-1.44 (m, 2H), 1.22-1.19 (m, 2H), 1.00 (t, J = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 139.24, 134.97, 132.17 (q, J = 32.4 Hz), 124.72 (q, J = 3.9 Hz,), 124.26 (q, J = 272.6 Hz,), 51.65, 18.17, 16.57, 15.71.; ¹⁹F NMR (377 MHz, CDCl₃) δ -63.08; HRMS (ESI): calculated for C₁₈H₁₉F₆OSi⁺ [M+H]⁺: 393.1104; found: 393.1108.



methoxybis(3-methoxyphenyl)(propyl)silane (2h). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 37.9 mg, 60%. ¹**H NMR** (600 MHz, CDCl₃) δ 7.35-7.30 (m, 2H), 7.16 (d, *J* = 7.2 Hz, 2H), 7.13 (d, *J* = 2.6 Hz, 2H), 6.95 (ddd, *J* = 8.3, 2.8, 1.0 Hz, 2H), 3.80 (s, 6H), 3.54 (s, 3H), 1.50-1.42 (m, 2H), 1.16-1.11 (m,

2H), 0.98 (t, J = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 159.17, 136.54, 129.24, 127.08, 120.07, 115.35, 55.27, 51.58, 18.32, 16.77, 16.21.; HRMS (ESI): calculated for C₁₈H₂₅O₃Si⁺ [M+H]⁺: 317.1567; found: 317.1569.



methoxy(propyl)di(thiophen-2-yl)silane (2i). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 39.1 mg, 73%. ¹H NMR (600 MHz, CDCl₃) δ 7.71 (dd, *J* = 4.6, 0.9 Hz, 2H), 7.45 (dd, *J* = 3.4, 0.9 Hz, 2H), 7.25 (dd, *J* = 4.6, 3.4 Hz, 2H), 3.55 (s, 3H), 1.55-1.50 (m, 2H), 1.18-1.14 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 136.96, 133.77, 132.23, 128.38, 51.63, 18.42, 18.12, 16.68.; HRMS (ESI): calculated

for $C_{12}H_{17}OS_2Si^+$ [M+H]⁺ : 269.0485; found: 269.0487.



methoxy(methyl)(phenyl)(propyl)silane (2j). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 33.0 mg, 85%. ¹**H NMR** (600 MHz, CDCl₃) δ 7.59-7.55 (m, 2H), 7.40 (t, *J* = 6.7 Hz, 3H), 3.46 (s, 3H), 1.43 (dq, *J* = 14.8, 7.5 Hz, 2H), 0.98 (t, *J* = 7.3 Hz,

3H), 0.93-0.81 (m, 2H), 0.38 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 137.07, 133.81, 129.70, 127.98, 50.94, 18.24, 17.51, 16.76, -4.12.; HRMS (ESI): calculated for C₁₁H₁₉OSi⁺ [M+H]⁺ : 195.1200; found: 195.1200.



methoxy(methyl)(propyl)(m-tolyl)silane (2k). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 30.0 mg, 72%. ¹**H NMR** (600 MHz, CDCl₃) δ 7.36 (d, *J* = 8.6 Hz, 2H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.22 (d, *J* = 7.5 Hz, 1H), 3.46 (s, 3H), 2.37 (s, 3H), 1.45-1.40 (m, 2H), 0.98 (d, *J* = 7.3 Hz, 3H), 0.90-0.81 (m, 2H), 0.36 (s, 3H); ¹³**C NMR** (151

MHz, CDCl₃) δ 137.33, 136.92, 134.43, 130.84, 130.52, 127.90, 50.95, 21.69, 18.27, 17.54, 16.78, -4.09.; HRMS (ESI): calculated for C₁₂H₂₁OSi⁺ [M+H]⁺: 209.1356; found: 209.1357.



(3,5-dimethylphenyl)(methoxy)(methyl)(propyl)silane (21). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 33.7 mg, 76%. ¹H NMR (600 MHz, CDCl₃) δ 7.17 (s, 2H), 7.04 (s, 1H), 3.46 (s, 3H), 2.33 (s, 6H), 1.47-1.40 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.90-0.80 (m, 2H), 0.36 (s, 3H); ¹³C

NMR (151 MHz, CDCl₃) δ 137.28, 136.72, 131.48, 130.72, 50.95, 21.53, 18.29, 17.57, 16.80, -4.07.; **HRMS** (ESI): calculated for C₁₃H₂₃OSi⁺ [M+H]⁺: 223.1513; found: 223.1513.



Me

^tRı

(3,4-dimethoxyphenyl)(methoxy)(methyl)(propyl)silane (2m). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 20/1) as a colorless oil. Isolated yield: 41.4 mg, 81%. ¹H NMR (600 MHz, CDCl₃) δ 7.12 (dd, J = 7.8, 1.4 Hz, 1H), 7.04 (d, J = 1.4 Hz, 1H), 6.91 (d, J = 7.8 Hz, 1H), 3.90 (d, J = 10.3 Hz, 6H), 3.44 (s, 3H), 1.45-1.39

(m, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.89-0.79 (m, 2H), 0.36 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 150.51, 148.79, 128.48, 127.16, 116.00, 111.07, 56.01, 55.82, 50.88, 18.25, 17.62, 16.81, -4.04.; HRMS (ESI): calculated for C₁₃H₂₃O₃Si⁺ [M+H]⁺ : 255.1411; found: 255.1406.

OMe (4-(tert-butyl)phenyl)(methoxy)(methyl)(propyl)silane (2n). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 43.0 mg, 86%. ¹H NMR (600 MHz, CDCl₃) δ 7.50 (d, J = 8.2 Hz, 2H), 7.41 (d, J = 8.2 Hz, 2H), 3.45 (s, 3H), 1.46-1.41 (m, 2H), 1.33 (s, 9H), 0.98 (t, J = 7.3 Hz, 3H), 0.89-0.82 (m, 2H), 0.36 (s, 3H); ¹³C NMR (151 MHz,

 $CDCl_{3}) \ \delta \ 152.66, \ 133.73, \ 133.51, \ 124.94, \ 50.95, \ 34.84, \ 31.37, \ 18.29, \ 17.59, \ 16.81, \ -4.05.; \ \textbf{HRMS} \ (ESI): \\ calculated \ for \ C_{15}H_{27}OSi^{+} \ [M+H]^{+}: \ 251.1826; \ found: \ 251.1826. \\ \end{cases}$



methoxy(4-methoxyphenyl)(methyl)(propyl)silane (20). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 31.8 mg, 71%. ¹H NMR (600 MHz, CDCl₃) δ 7.49 (d, J = 8.7 Hz, 2H), 6.93 (d, J = 8.6 Hz, 2H), 3.82 (s, 3H), 3.43 (s, 3H), 1.45-1.38 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.89-0.79(m, 2H), 0.35 (s, 3H); ¹³C NMR (151

MHz, CDCl₃) δ 160.96, 135.38, 127.97, 113.73, 55.15, 50.84, 18.26, 17.61, 16.80, -4.02.; **HRMS** (ESI): calculated for C₁₂H₂₁O₂Si⁺ [M+H]⁺: 225.1305; found: 225.1301.



methoxy(methyl)(4-(methylthio)phenyl)(propyl)silane (2p). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 43.2 mg, 90%. ¹H NMR (600 MHz, CDCl₃) δ 7.46 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 3.44 (s, 3H), 2.49 (s, 3H), 1.44-1.38 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.89-0.79 (m, 2H), 0.35 (s, 3H);

¹³C NMR (151 MHz, CDCl₃) δ 140.59, 134.22, 132.92, 125.64, 50.90, 18.22, 17.51, 16.74, 15.34, -4.11.; **HRMS** (ESI): calculated for C₁₂H₂₁OSSi⁺ [M+H]⁺: 241.1077; found: 241.1077.



[1,1'-biphenyl]-4-yl(methoxy)(methyl)(propyl)silane (2q). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 49.1 mg, 91%. ¹H NMR (600 MHz, CDCl₃) δ 7.68-7.61 (m, 6H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.37 (d, *J* = 7.3 Hz, 1H), 3.50 (s, 3H), 1.51-1.44 (m, 2H), 1.01 (td, *J* = 7.3, 2.9 Hz, 3H), 0.94-0.87 (m, 2H), 0.42 (s, 3H); ¹³C NMR

 $(151 \text{ MHz}, \text{CDCl}_3) \\ \delta 142.44, 141.18, 135.78, 134.34, 128.92, 127.59, 127.32, 126.74, 51.00, 18.27, 17.58, 16.79, -4.02.; \text{ HRMS} (ESI): calculated for C_{17}H_{23}OSi^+ \ [M+H]^+: 271.1513; found: 271.1515.$



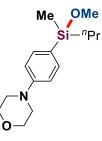
4-(methoxy(methyl)(propyl)silyl)-N,N-dimethylaniline (2**r).** The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 20/1) as a colorless oil. Isolated yield: 30.3 mg, 64%. ¹**H** NMR (600 MHz, CDCl₃) δ 7.44 (d, *J* = 8.4 Hz, 2H), 6.75 (d, *J* = 8.0 Hz, 2H), 3.42 (s, 3H), 2.98 (s, 6H), 1.46-1,39 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.87-0.80 (m, 2H), 0.33 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 134.95, 111.76, 50.64, 40.15, 18.17, 17.53, 16.74, -4.17.;

HRMS (ESI): calculated for $C_{13}H_{24}NOSi^+$ [M+H]⁺: 238.1622; found: 238.1627.



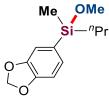
(4-fluorophenyl)(methoxy)(methyl)(propyl)silane (2s). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 17.0 mg, 40%. ¹H NMR (600 MHz, CDCl₃) δ 7.53 (dd, *J* = 8.4, 6.3 Hz, 2H), 7.10-7.05 (m, 2H), 3.44 (s, 3H), 1.44-1.37 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.89-0.81 (m, 2H), 0.36 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 164.17 (d, *J* = 248.8 = 7.4 Hz) 132.63 (d, *L* = 3.8 Hz) 115.17 (d, *L* = 19.8 Hz) 50.90 18.20 17.56 16.72

Hz), 135.80 (d, J = 7.4 Hz), 132.63 (d, J = 3.8 Hz), 115.17 (d, J = 19.8 Hz), 50.90, 18.20, 17.56, 16.72, -4.02.; ¹⁹**F NMR** (377 MHz, CDCl₃) δ -111.12; **HRMS** (ESI): calculated for C₁₁H₁₈FOSi⁺ [M+H]⁺ : 213.1105; found: 213.1104.



4-(4-(methoxy(methyl)(propyl)silyl)phenyl)morpholine (2t). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 33.5 mg, 60%. ¹H NMR (600 MHz, CDCl₃) δ 7.47 (d, *J* = 8.6 Hz, 2H), 6.92 (d, *J* = 8.1 Hz, 2H), 3.86 (d, *J* = 4.9 Hz, 4H), 3.42 (s, 3H), 3.21 (d, *J* = 5.0 Hz, 4H), 1.45-1.38 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.88-0.80 (m, 2H), 0.34 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 134.98, 114.67, 66.84, 50.69, 48.62, 18.14, 17.46, 16.68, -4.20.; HRMS (ESI): calculated

for $C_{15}H_{26}NO_2Si^+$ [M+H]⁺: 280.1727; found: 280.1727.



benzo[d][1,3]dioxol-5-yl(methoxy)(methyl)(propyl)silane (2u). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 43.3 mg, 91%. ¹H NMR (600 MHz, CDCl₃) δ 7.04 (dd, J = 7.6, 1.1 Hz, 1H), 7.01 (s, 1H), 6.86 (d, J = 7.6 Hz, 1H), 5.95 (s, 2H), 3.43 (s, 3H), 1.44-1.37 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.87-0.79

(m, 2H), 0.33 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 148.96, 147.59, 130.12, 128.03, 113.11, 108.78, 100.71, 50.87, 18.23, 17.61, 16.77, -4.00.; **HRMS** (ESI): calculated for C₁₂H₁₉O₄Si⁺ [M+H]⁺: 239.1098; found: 239.1093.



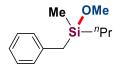
methoxy(methyl)(naphthalen-1-yl)(propyl)silane (2v). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 40.5 mg, 83%. ¹H NMR (600 MHz, CDCl₃) δ 8.31 (d, *J* = 8.6 Hz, 1H), 7.92-7.85 (m, 2H), 7.73 (dd, *J* = 6.8, 1.3 Hz, 1H), 7.55-7.46 (m, 3H), 3.47 (s, 3H), 1.50-1.40 (m, 2H), 1.11-1.00 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.55 (s, 3H); ¹³C NMR

(151 MHz, CDCl₃) δ 137.22, 135.16, 134.27, 133.42, 130.52, 128.99, 128.21, 126.24, 125.66, 125.63, 125.14, 50.86, 18.62, 18.24, 16.97, -2.93.; **HRMS** (ESI): calculated for C₁₅H₂₁OSi⁺ [M+H]⁺:245.1356; found: 245.1354.



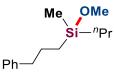
methoxy(methyl)(propyl)(o-tolyl)silane (2w). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 26.2 mg, 63%. ¹H NMR (600 MHz, CDCl₃) δ 7.49 (d, *J* = 7.3 Hz, 1H), 7.29 (td, *J* = 7.5, 1.5 Hz, 1H), 7.18 (t, *J* = 8.0 Hz, 2H), 3.44 (s, 3H), 2.48 (s, 3H), 1.45-1.37

(m, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.94- 0.86 (m, 2H), 0.41 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 143.92, 135.52, 135.04, 130.02, 129.88, 124.98, 50.70, 22.56, 18.26, 18.20, 16.90, -3.09.; HRMS (ESI): calculated for C₁₂H₂₁OSi⁺ [M+H]⁺ : 209.1356; found: 209.1360.



benzyl(methoxy)(methyl)(propyl)silane (2x). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 21.6 mg, 52%. ¹H NMR (600 MHz, CDCl₃) δ 7.22 (t, *J* = 7.7 Hz, 2H), 7.10-7.05 (m, 3H), 3.42 (s, 3H), 2.23-2.13 (m, 2H), 1.42-1.33 (m, 2H), 0.95

(t, J = 7.3 Hz, 3H), 0.67- 0.54 (m, 2H), 0.06 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 139.24, 128.49, 128.42, 124.31, 50.89, 25.14, 18.24, 16.85, 16.70, -4.52.; HRMS (ESI): calculated for C₁₂H₂₁OSi⁺ [M+H]⁺ : 209.1356; found: 209.1356.



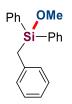
Ph

methoxy(methyl)(3-phenylpropyl)(propyl)silane (2y). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 40.1 mg, 85%. ¹H NMR (600 MHz, CDCl₃) δ 7.25-7.19 (m, 2H), 7.10-7.05 (m, 3H), 3.42 (s, 3H), 2.23-2.13 (m, 2H), 1.41-1.34

(m, 2H), 0.95 (t, J = 7.3 Hz, 3H), 0.66-0.55 (m, 2H), 0.06 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 142.66, 128.63, 128.37, 125.80, 50.60, 39.87, 25.39, 18.32, 17.31, 16.80, 14.67, -4.26.; HRMS (ESI): calculated for C₁₄H₂₅OSi⁺ [M+H]⁺ : 237.1669; found: 237.1669.

OMe methoxydiphenyl(o-tolyl)silane (2z). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 40.1 mg, 66%. ¹H NMR (600 MHz, CDCl₃) δ 7.62 (dd, J = 8.0, 1.4 Hz, 4H), 7.54 (dd, J = 7.4, 1.5 Hz, 1H), 7.45 (t, J = 7.4 Hz, 2H), 7.41-7.35 (m, 5H), 7.21-7.18 (m, 2H),

3.64 (s, 3H), 2.30 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 145.02, 136.95, 135.48, 134.51, 132.65, 130.50, 130.23, 130.06, 128.06, 125.01, 51.92, 23.24.; **HRMS** (ESI): calculated for C₂₀H₂₁OSi⁺ [M+H]⁺ : 305.1356; found: 305.1355.



benzyl(methoxy)diphenylsilane (2z'). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 17.0 mg, 28%. ¹H NMR (600 MHz, CDCl₃) δ 7.50 (dd, *J* = 8.0, 1.4 Hz, 4H), 7.42 (t, *J* = 7.4 Hz, 2H), 7.38-7.32 (m, 5H), 7.13 (t, J = 7.5 Hz, 2H), 6.97 (d, J = 6.5 Hz, 2H), 3.52 (s, 3H), 2.71 (s, 2H); ¹³C NMR (151 MHz, CDCl₃) δ 137.71, 135.02, 134.04, 130.13, 129.18,

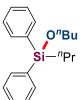
128.25, 127.96, 124.62, 51.83, 23.88.; HRMS (ESI): calculated for C₂₀H₂₁OSi⁺ [M+H]⁺ : 305.1356; found: 305.1355.

methoxy(methyl)(phenyl)(o-tolyl)silane (2aa). The title compound was isolated through OMe Ph preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: Si-Me 38.7 mg, 80%. ¹H NMR (600 MHz, CDCl₃) δ 7.60-7.53 (m, 3H), 7.44-7.39 (m, 1H), Me 7.39-7.35 (m, 2H), 7.33 (td, J = 7.5, 1.5 Hz, 1H), 7.24-7.19 (m, 1H), 7.17 (d, J = 7.6 Hz, 1H), 3.54 (s, 3H), 2.33 (s, 3H), 0.68 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 144.30, 136.53, 135.73,

134.29, 130.28, 130.10, 129.85, 128.04, 125.04, 51.15, 22.83, -2.70.; HRMS (ESI): calculated for $C_{15}H_{19}OSi^+$ [M+H]⁺: 243.1200; found: 243.1199.

ethoxydiphenyl(propyl)silane (2ab). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: Si-"Pr 35.1 mg, 65. ¹H NMR (600 MHz, CDCl₃) δ 7.62-7.57 (m, 4H), 7.43-7.36 (m, 6H), 3.77 (q, J = 7.0 Hz, 2H), 1.47 (dd, J = 16.5, 7.3 Hz, 2H), 1.21 (t, J = 6.9 Hz, 3H), 1.17-1.13 (m, 2H), 0.98 (t, J = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 135.60, 134.81,

129.82, 127.92, 59.36, 18.54, 18.36, 16.83, 16.65.; **HRMS** (ESI): calculated for $C_{17}H_{23}OSi^+$ [M+H]⁺: 271.1513; found: 271.1514.



OEt

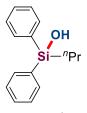
butoxydiphenyl(propyl)silane (2ac). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 28.1 mg, 47%. ¹H NMR (600 MHz, CDCl₃) δ 7.59 (dd, *J* = 7.9, 1.4 Hz, 4H), 7.42-7.35 (m, 6H), 3.68 (t, J = 6.6 Hz, 2H), 1.58 (s, 2H), 1.54 (dd, J = 14.8, 6.8 Hz, 2H), 1.49-1.43 (m, 2H), 1.39-1.33 (m, 2H), 1.25 (t, J = 7.0 Hz, 2H), 1.16-1.13 (m, 2H), 0.98 (t, J = 7.3 Hz,

3H), 0.88 (t, J = 7.4 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 135.70, 134.82, 129.79, 127.89, 63.40, 34.89, 19.12, 18.37, 16.83, 16.56, 14.01.; **HRMS** (ESI): calculated for C₁₉H₂₇OSi⁺ [M+H]⁺: 299.1826; found: 299.1826.



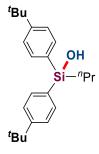
isopropoxydiphenyl(propyl)silane (2ad). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 40/1) as a colorless oil. Isolated yield: 42.0 mg, 74%. ¹H NMR (600 MHz, CDCl₃) δ 7.64-7.57 (m, 4H), 7.42-7.34 (m, 6H), 4.08 (p, J = 6.1 Hz, 1H), 1.49-1.41 (m, 2H), 1.16 (d, J = 6.1 Hz, 8H), 0.98 (t, J = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 136.18, 134.89, 129.71, 127.84, 65.89, 25.84,

18.46, 17.10, 16.91.; **HRMS** (ESI): calculated for $C_{18}H_{25}OSi^+$ [M+H]⁺: 285.1669; found: 285.1663.

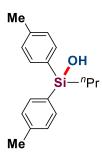


diphenyl(propyl)silanol (3a). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 45.0 mg, 93%. ¹H NMR (600 MHz, CDCl₃) δ 7.61 (dd, *J* = 7.9, 1.4 Hz, 4H), 7.46-7.35 (m, 6H), 1.54-1.46 (m, 2H), 1.18-1.13 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 136.57, 134.31, 129.96, 128.03, 18.27, 17.80, 16.78.; HRMS (ESI): calculated for +HI⁺ : 242 1200; found: 243 1200

 $C_{15}H_{19}OSi^{+} \ \ [M+H]^{+}: 243.1200; \ found: 243.1200.$



bis(4-(tert-butyl)phenyl)(propyl)silanol (3b). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 32.6 mg, 46%. ¹H NMR (600 MHz, CDCl₃) δ 7.55 (d, *J* = 8.2 Hz, 4H), 7.41 (d, *J* = 8.2 Hz, 4H), 1.55-1.48 (m, 2H), 1.32 (s, 18H), 1.18-1.10 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 152.86, 134.23, 133.27, 124.97, 34.86, 31.36, 18.36, 18.01, 16.85.; **HRMS** (ESI): calculated for C₂₃H₃₅OSi⁺ [M+H]⁺: 355.2453; found: 355.2454.

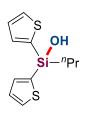


propyldi-p-tolylsilanol (3c). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 49.7 mg, 92%. ¹**H NMR** (600 MHz, CDCl₃) δ 7.50 (d, *J* = 7.9 Hz, 4H), 7.21 (d, *J* = 7.6 Hz, 4H), 2.37 (s, 6H), 1.56-1.42 (m, 2H), 1.16-1.10 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 139.83, 134.38, 133.16, 128.82, 21.68, 18.30, 17.95, 16.83.; **HRMS** (ESI): calculated for C₁₇H₂₃OSi⁺ [M+H]⁺ : 271.1513; found: 271.1516.

CI OH Si-"Pr **bis(4-chlorophenyl)(propyl)silanol (3d).** The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 47.1 mg, 76%. ¹H NMR (600 MHz, CDCl₃) δ 7.49 (d, *J* = 8.3 Hz, 4H), 7.36 (d, *J* = 8.3 Hz, 4H), 1.50-1.42 (m, 2H), 1.14-1.08 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 136.60, 135.62, 134.38, 128.43, 18.18, 17.64, 16.67.; HRMS (ESI): calculated for C₁₅H₁₇Cl₂OSi⁺ [M+H]⁺:325.0577; found: 325.0577



propylbis(4-(trifluoromethyl)phenyl)silanol (3e). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 46.1 mg, 61%. ¹H NMR (600 MHz, CDCl₃) δ 7.71 (d, *J* = 7.8 Hz, 4H), 7.64 (d, *J* = 7.8 Hz, 4H), 1.52-1.50 (m, 2H), 1.21-1.17 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 140.56, 134.54, 132.20 (q, *J* = 32.2 Hz), 124.76 (q, *J* = 3.8 Hz), 124.18 (q, *J* = 272.4 Hz), 18.13, 17.43, 16.59; ¹⁹F NMR (377 MHz, CDCl₃) δ -63.08; HRMS (ESI): calculated for C₁₇H₁₇F₆OSi⁺ [M+H]⁺ : 379.0947; found: 379.0949.



propyldi(thiophen-2-yl)silanol (3f). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 48.3 mg, 95%. ¹H NMR (600 MHz, CDCl₃) δ 7.64 (d, *J* = 4.5 Hz, 1H), 7.37 (d, *J* = 3.2 Hz, 1H), 7.22 (t, *J* = 3.8 Hz, 1H), 1.51-1.46 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H), 0.90 (dd, *J* = 10.1, 6.3 Hz, 2H), 0.45 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 136.56, 135.49,

132.15, 128.42, 19.74, 18.08, 16.67.; **HRMS** (ESI): calculated for $C_{11}H_{15}OS_2Si^+$ [M+H]⁺ : 255.0328; found: 255.0328.



methyl(propyl)(thiophen-2-yl)silanol (3g). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield:31.2 mg, 84%. ¹H NMR (600 MHz, CDCl₃) δ 7.64 (d, *J* = 4.5 Hz, 1H), 7.37 (dd, *J* = 3.2, 1.0 Hz, 1H), 7.22 (t, *J* = 3.8 Hz, 1H), 1.52-1.45 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H),

0.90 (dd, J = 10.1, 6.3 Hz, 2H), 0.45 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 138.00, 134.90, 131.21, 128.32, 19.98, 18.12, 16.73, -0.43.; **HRMS** (ESI): calculated for C₈H₁₅OSSi⁺ [M+H]⁺: 187.0607; found: 187.0611.



DH methyl(phenyl)(propyl)silanol (3h). The title compound was isolated through $-^{n}$ Pr preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield:19.8 mg, 55%. ¹H NMR (600 MHz, CDCl₃) δ 7.59 (dd, *J* = 7.4, 1.9 Hz, 2H), 7.44-7.34 (m, 3H), 1.48-1.41 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.88-0.83 (m, 2H),

 $\begin{array}{l} 0.39\ (s,\, 3H); {}^{13}C\ \textbf{NMR}\ (151\ \text{MHz},\ \text{CDCl}_3)\ \delta\ 138.69,\ 133.37,\ 129.71,\ 128.01,\ 19.23,\ 18.21,\ 16.79,\ -1.45.\\ ;\ \textbf{HRMS}\ (ESI):\ \text{calculated for}\ C_{10}H_{17}OSi^+\ \ [M+H]^+:\ 181.1043;\ \text{found:}\ 181.1039. \end{array}$



(4-(tert-butyl)phenyl)(methyl)(propyl)silanol (3i). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 28.3 mg, 60%. ¹H NMR (600 MHz, CDCl₃) δ 7.53 (d, J = 8.2 Hz, 2H), 7.42 (d, J = 8.1 Hz, 2H), 1.49-1.43 (m, 2H), 1.33 (s, 9H), 0.99 (t, J = 7.3 Hz, 3H), 0.87-0.83 (m, 2H), 0.38 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ

152.75, 135.19, 133.33, 124.99, 34.84, 31.36, 19.27, 18.27, 16.83, -1.44.; **HRMS** (ESI): calculated for $C_{14}H_{25}OSi^+$ [M+H]⁺: 237.1669; found: 237.1667.



(4-methoxyphenyl)(methyl)(propyl)silanol (3j). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 34.4 mg, 82%. ¹H NMR (600 MHz, CDCl₃) δ 7.51 (d, J = 8.4 Hz, 2H), 6.93 (d, J = 8.3 Hz, 2H), 3.82 (s, 3H), 1.46-1.40 (m, 2H), 0.98 (t, J = 7.3 Hz, 3H), 0.84 (dd, J = 10.0, 6.6 Hz, 2H), 0.37 (s, 3H); ¹³C NMR (151

MHz, CDCl₃) δ 160.95, 134.94, 129.68, 113.75, 55.18, 19.37, 18.23, 16.84, -1.35.; **HRMS** (ESI): calculated for C₁₁H₁₉O₂Si⁺ [M+H]⁺: 211.1149; found: 211.1149.



methyl(4-(methylthio)phenyl)(propyl)silanol (3k). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 21.7 mg, 48%. ¹H NMR (600 MHz, CDCl₃) δ 7.48 (d, J = 8.2 Hz, 2H), 7.25 (d, J = 8.1 Hz, 2H), 2.49 (s, 3H), 1.46-1.39 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H), 0.89-0.78 (m, 2H), 0.37 (s, 3H); ¹³C NMR (151 MHz, CDCl₃)

δ 140.60, 134.57, 133.80, 125.69, 19.25, 18.20, 16.79, 15.39, -1.42.; **HRMS** (ESI): calculated for C₁₁H₁₉OSSi⁺ [M+H]⁺: 227.0920; found: 227.0919.



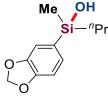
(4-fluorophenyl)(methyl)(propyl)silanol (3l). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 32.9 mg, 83%. ¹H NMR (600 MHz, CDCl₃) δ 7.55 (dd, *J* = 8.4, 6.3 Hz, 2H), 7.07 (dd, *J* = 9.3, 8.6 Hz, 2H), 1.45-1.39 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.87-0.80 (m, 2H), 0.38 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 164.14 (d, *J* = 248.9 Hz),

135.40 (d, J = 7.3 Hz), 134.22 (d, J = 3.8 Hz), 115.16 (d, J = 19.5 Hz), 19.31, 18.17, 16.84, 16.76, -1.33.; ¹⁹F NMR (377 MHz, CDCl₃) δ -111.15; **HRMS** (ESI): calculated for C₁₀H₁₆FOSi⁺ [M+H]⁺: 199.0949; found: 199.0953.



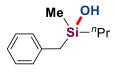
methyl(naphthalen-1-yl)(propyl)silanol (3m). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield:37.7 mg, 82%. ¹H NMR (600 MHz, CDCl₃) δ 8.26 (d, *J* = 8.3 Hz, 1H), 7.90-7.86 (m, 2H), 7.78 (dd, *J* = 6.8, 1.3 Hz, 1H), 7.54-7.46 (m, 3H), 1.52-1.42 (m, 2H), 1.07-1.03 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H), 0.57 (s, 3H); ¹³C NMR (151 MHz,

CDCl₃) δ 136.89, 136.55, 133.61, 133.53, 130.46, 129.15, 128.26, 128.02, 126.11, 125.96, 125.64, 125.16, 20.28, 18.21, 17.01, -0.06.; **HRMS** (ESI): calculated for C₁₄H₁₉OSi⁺ [M+H]⁺: 231.1200; found: 231.1203.



benzo[d][1,3]dioxol-5-yl(methyl)(propyl)silanol (3n). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 40.8 mg, 91%. ¹H NMR (600 MHz, CDCl₃) δ 7.06 (d, *J* = 7.6 Hz, 1H), 7.03 (s, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 5.94 (s, 2H), 1.45-1.39 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.84-0.79 (m, 2H), 0.35 (s, 3H); ¹³C NMR (151

MHz, CDCl₃) δ 148.92, 147.55, 131.78, 127.55, 112.76, 108.76, 100.70, 19.33, 18.19, 16.80, -1.34.; **HRMS** (ESI): calculated for C₁₁H₁₇O₃Si⁺ [M+H]⁺ : 225.0941; found: 225.0943.



benzyl(methyl)(propyl)silanol (30). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 24.1 mg, 62%. ¹H NMR (600 MHz, CDCl₃) δ 7.24 (dd, *J* = 14.8, 7.1 Hz, 2H), 7.10 (t, *J* = 7.2 Hz, 1H), 7.06 (d, *J* = 7.4 Hz, 2H), 2.24-2.12 (m, 2H), 1.44-

1.36 (m, 2H), 0.97 (t, J = 7.2 Hz, 3H), 0.62 (td, J = 7.7, 3.3 Hz, 2H), 0.11 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 139.16, 128.58, 128.33, 124.42, 26.87, 18.48, 18.21, 16.68, -2.11.; HRMS (ESI): calculated for C₁₁H₁₉OSi⁺ [M+H]⁺:195.1200; found: 195.1200.

Ph OH diphenyl(o-tolyl)silanol (3p). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 53.4 mg, 92%. ¹H NMR (600 MHz, CDCl₃) ¹H NMR (600 MHz, CDCl₃) δ 7.65-7.59 (m, 4H), 7.49-7.43 (m, 3H), 7.41-7.36 (m, 5H), 7.22 (d, J = 7.6 Hz, 1H), 7.21-7.14 (m, 1H), 2.34 (s, 3H); ¹³C NMR (151 MHz, CDCl₃) δ 144.73, 136.72, 135.81, 135.02, 134.49, 133.67, 130.61, 130.22, 130.15, 128.95, 128.51, 128.12, 128.02, 125.03, 23.45.; HRMS (ESI): calculated for C₁₉H₁₉OSi⁺ [M+H]⁺ : 291.1200; found: 291.1200.



methyl(phenyl)(o-tolyl)silanol (3q). The title compound was isolated through preparative TLC on silica gel (*n*-Hexane/EtOAc = 10/1) as a colorless oil. Isolated yield: 42.0 mg, 94%. ¹H NMR (600 MHz, CDCl₃) δ 7.62 (d, *J* = 7.3 Hz, 1H), 7.60-7.56 (m, 2H), 7.46-7.41 (m, 1H), 7.40-7.33 (m, 3H), 7.25-7.17 (m, 2H), 2.36 (s, 3H), 0.72 (s,

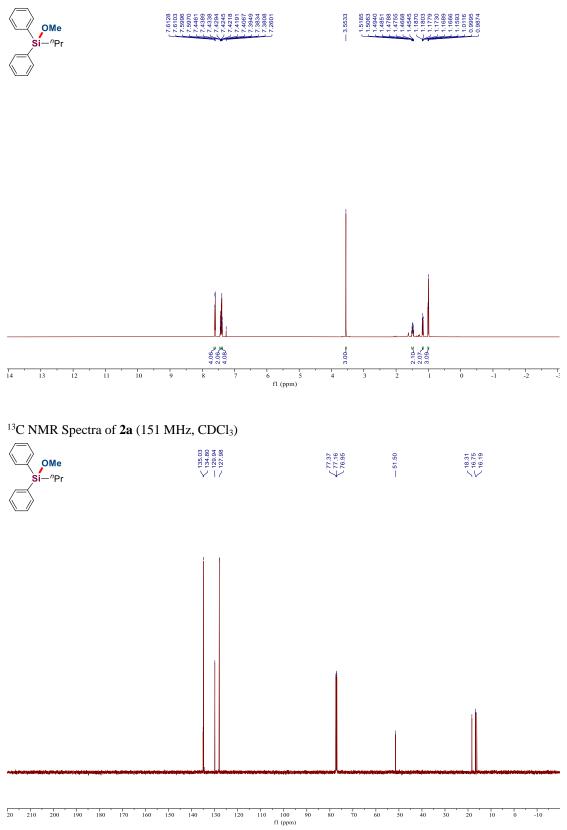
3H); ¹³C NMR (151 MHz, CDCl₃) δ 143.96, 137.94, 135.39, 135.31, 133.92, 130.34, 130.11, 129.92, 128.10, 125.11, 23.10, -0.24.; HRMS (ESI): calculated for C₁₄H₁₇OSi⁺ [M+H]⁺ : 229.1043; found: 229.1046.

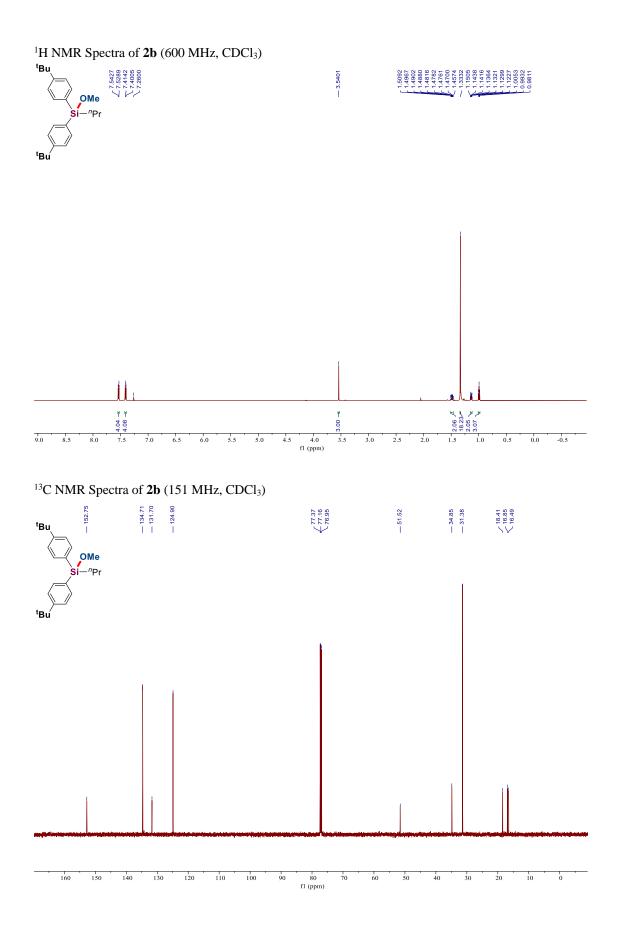
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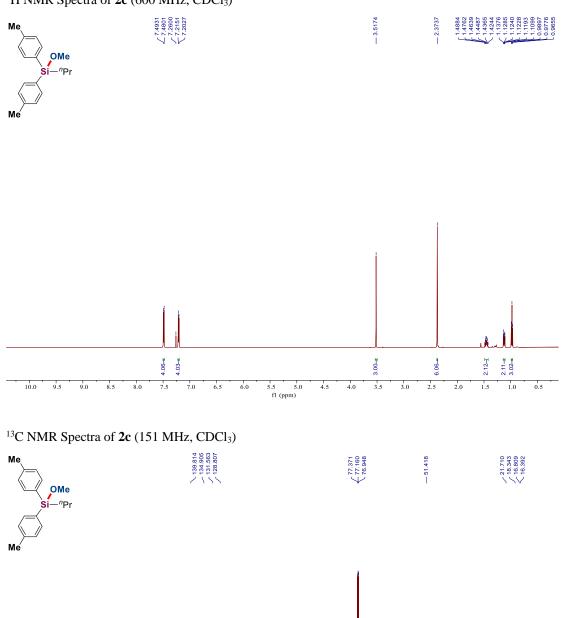
7. NMR Spectra

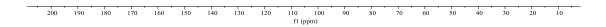
¹H NMR Spectra of **2a** (600 MHz, CDCl₃)

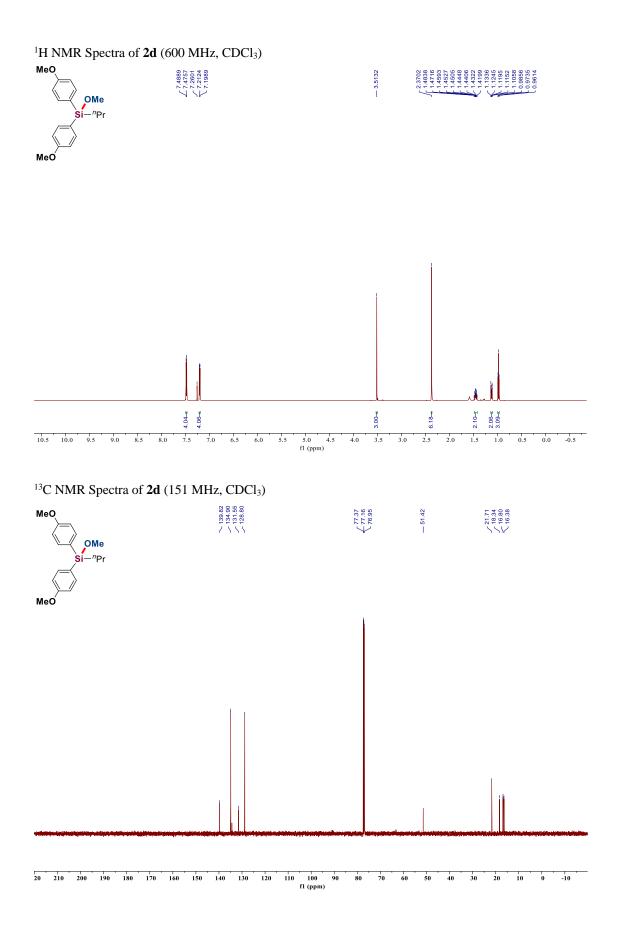


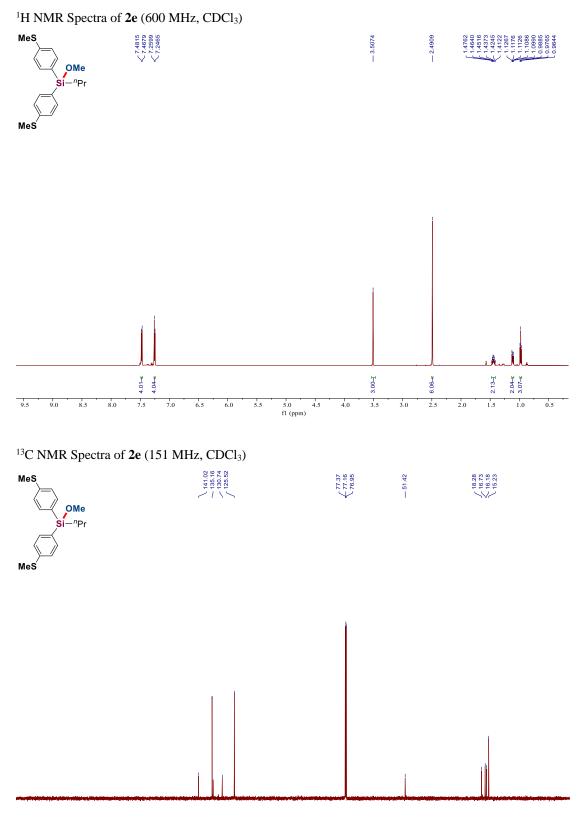


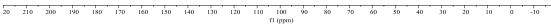
¹H NMR Spectra of **2c** (600 MHz, CDCl₃)

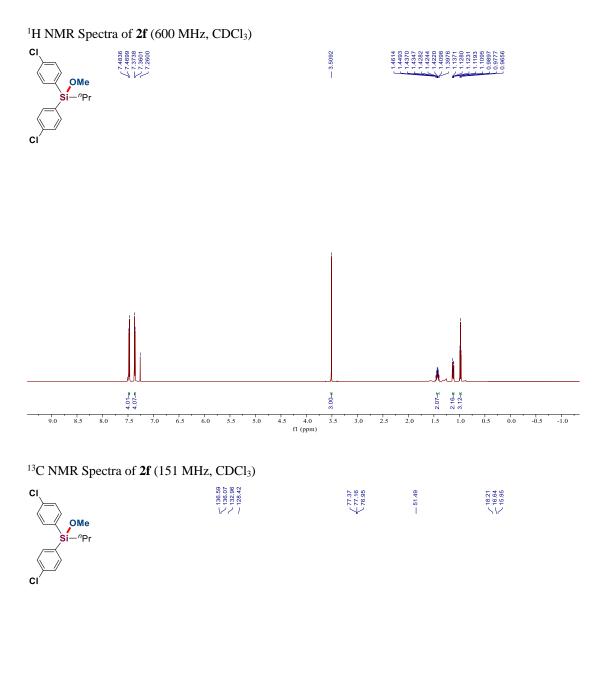


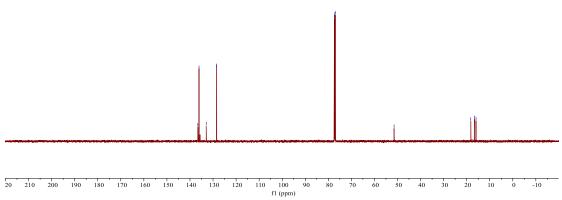






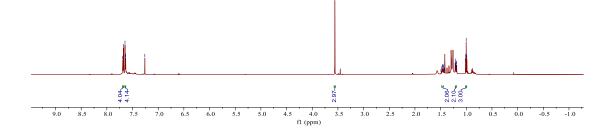




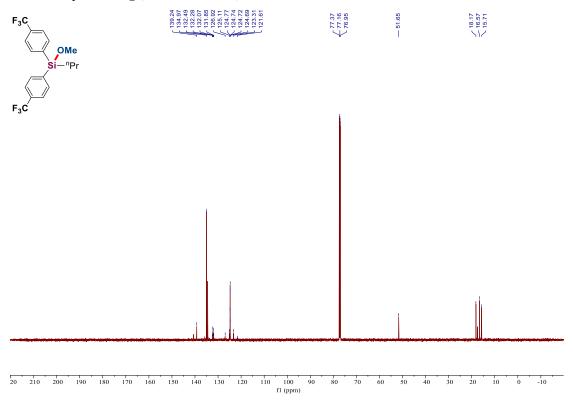


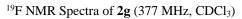
¹H NMR Spectra of **2g** (600 MHz, CDCl₃)

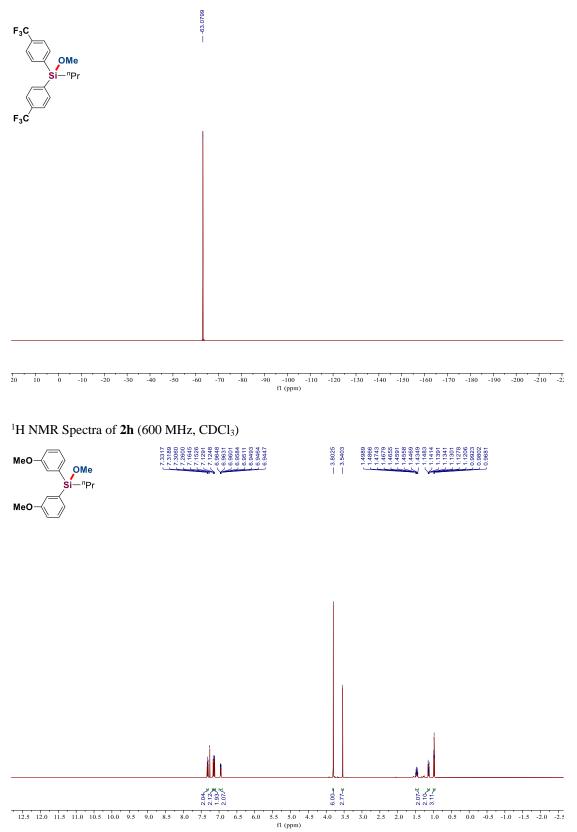


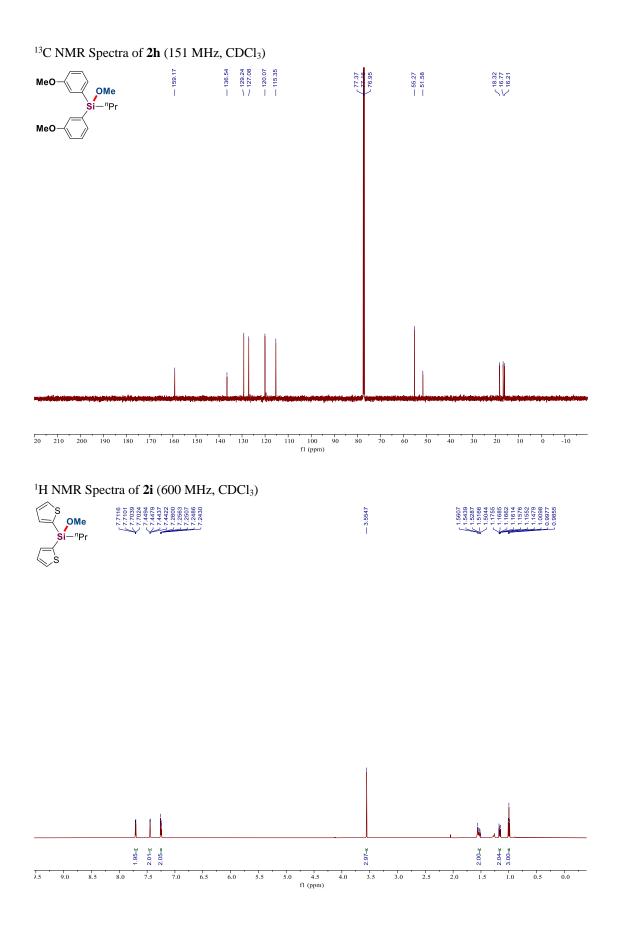


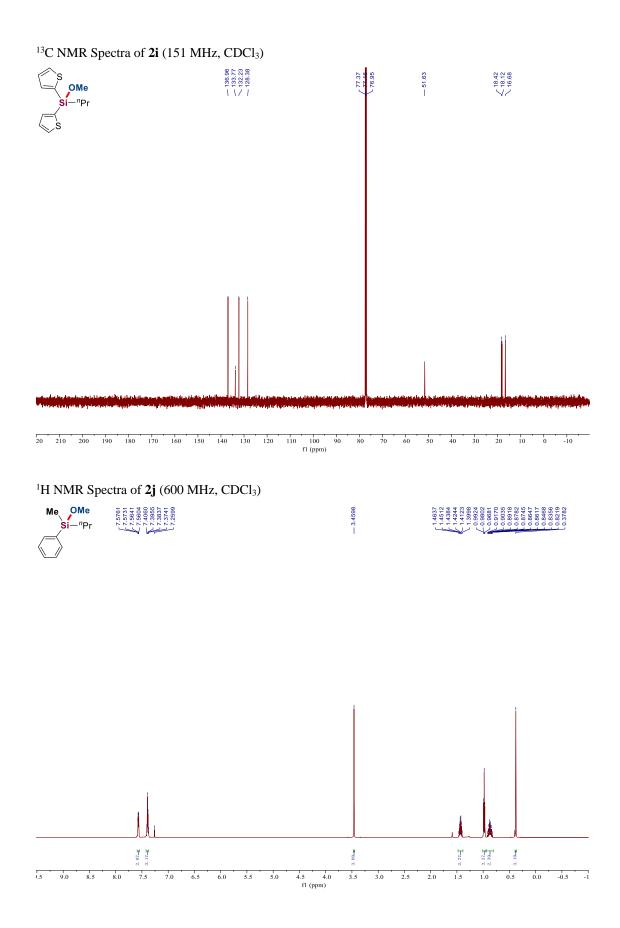
 ^{13}C NMR Spectra of 2g (151 MHz, CDCl₃)

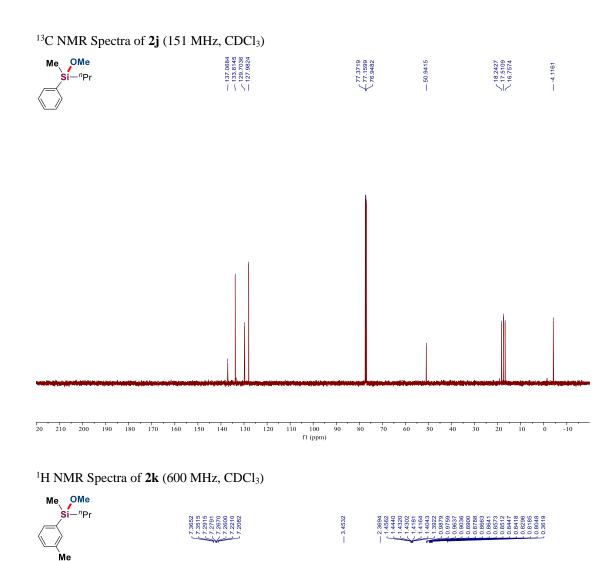


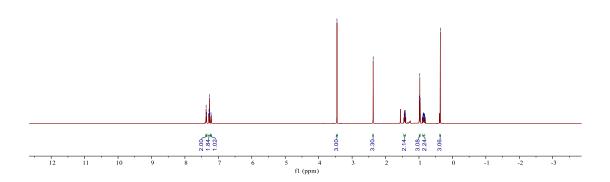


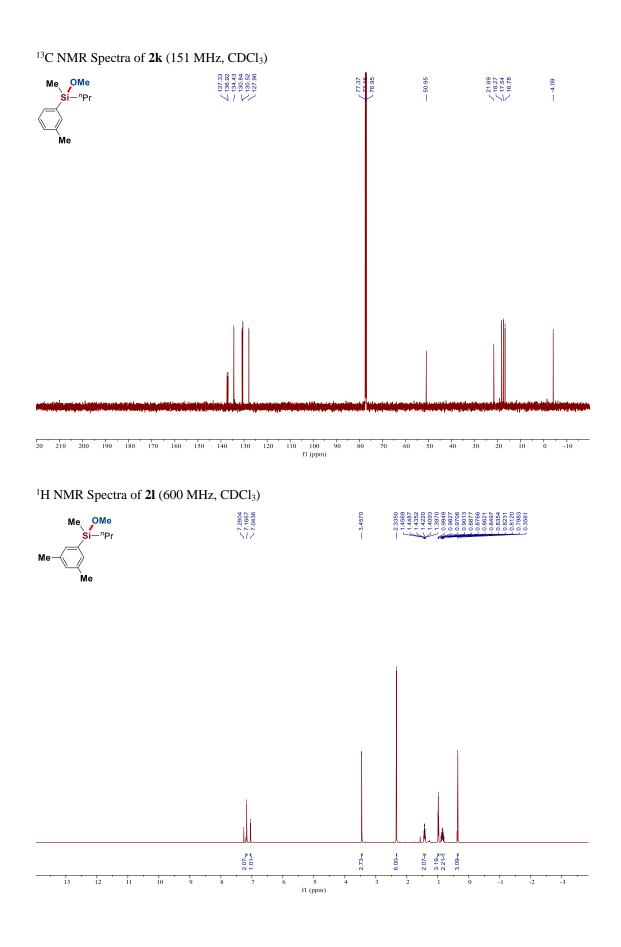


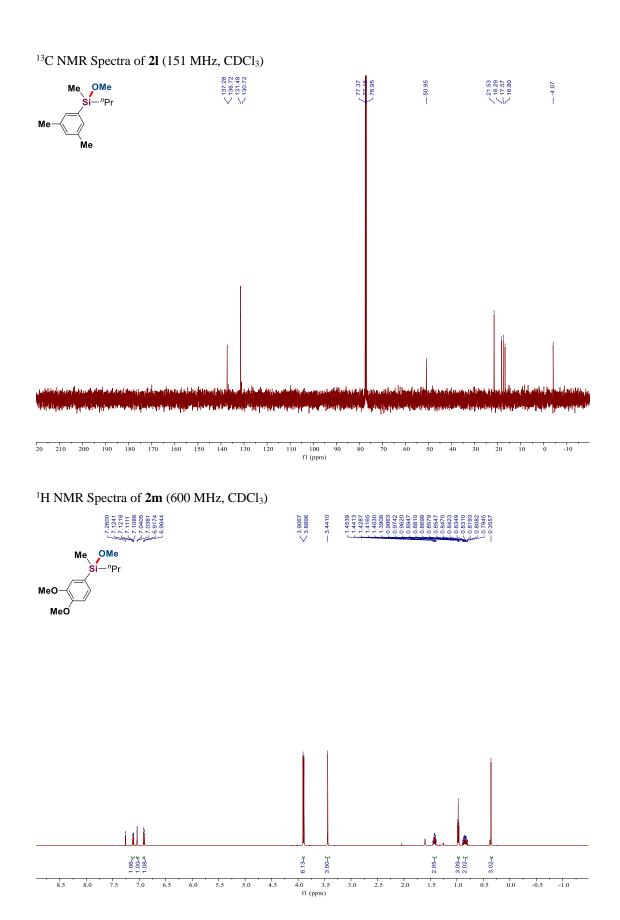




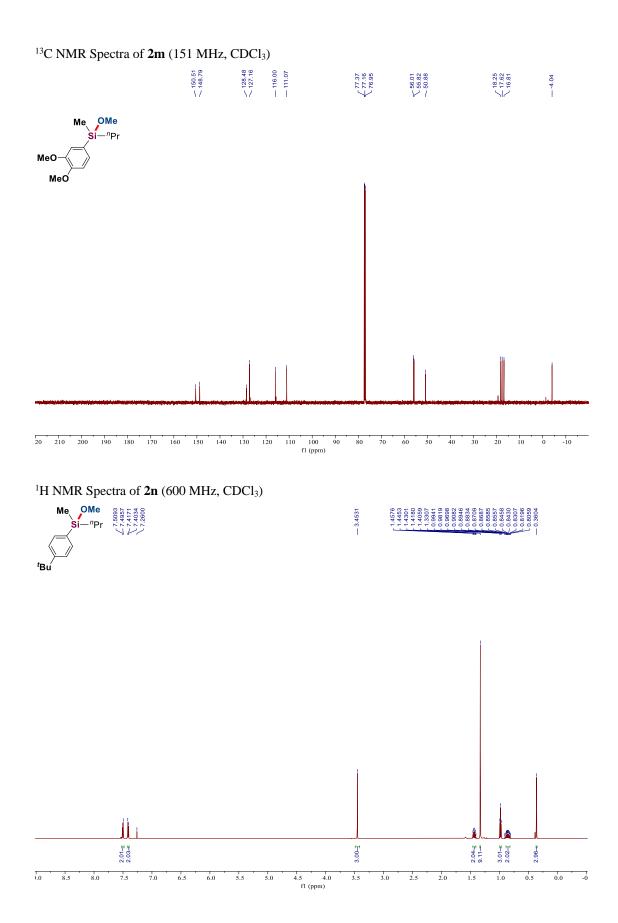




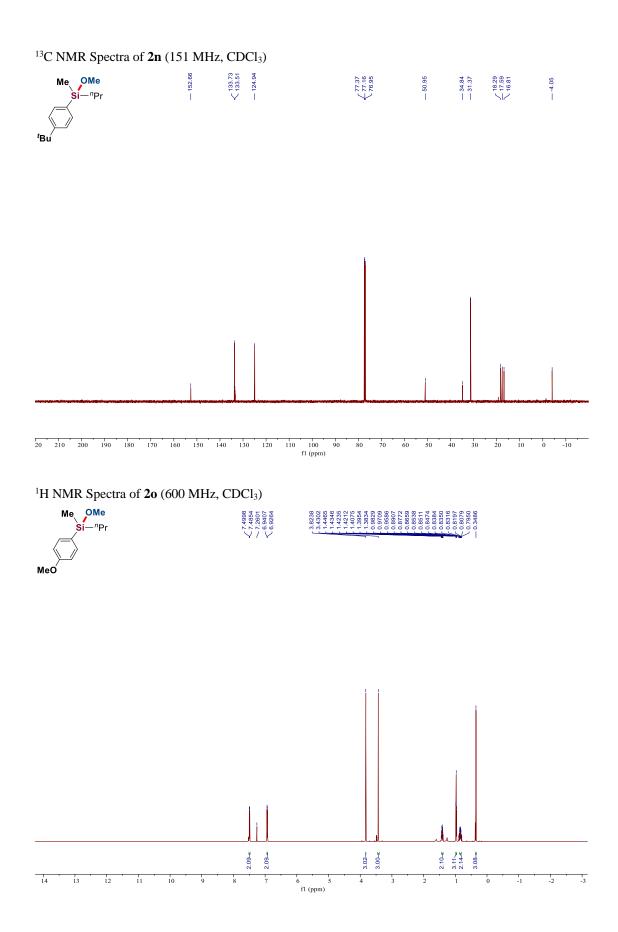


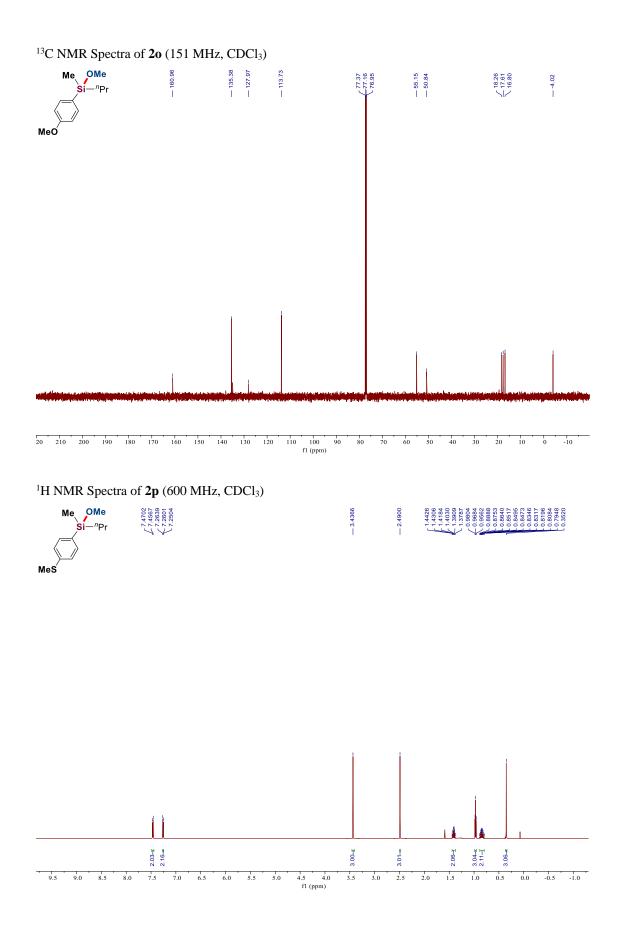


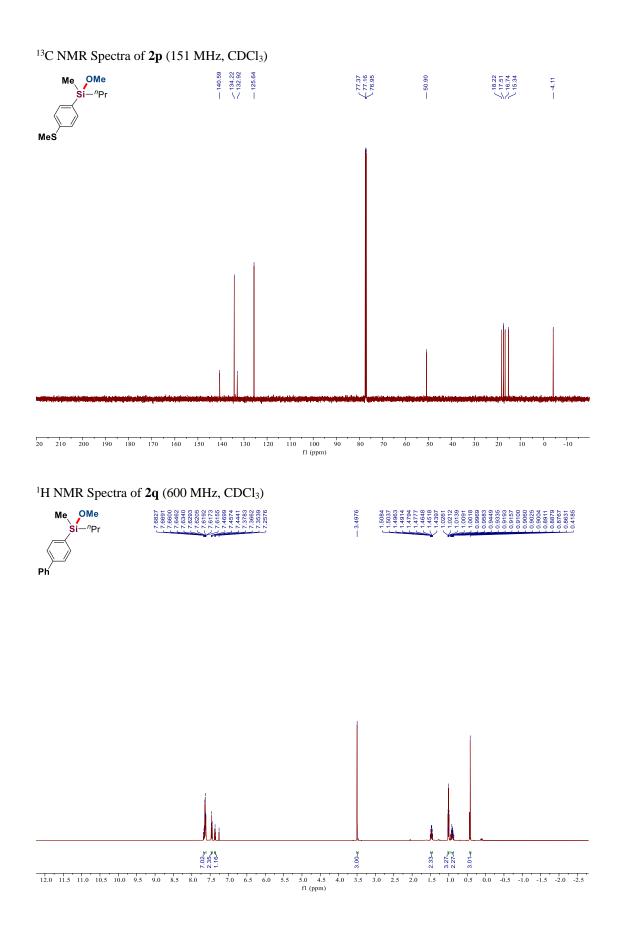
S32

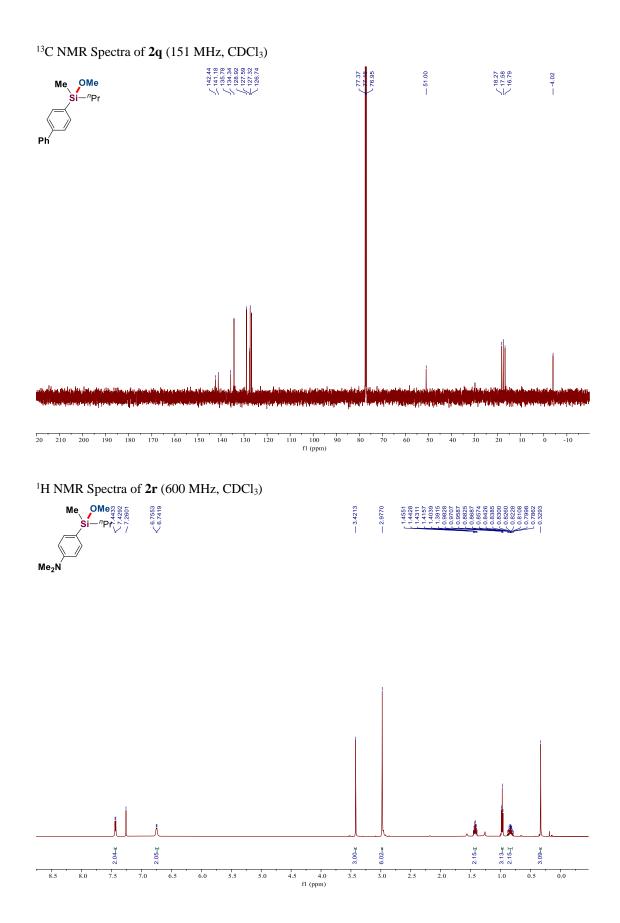


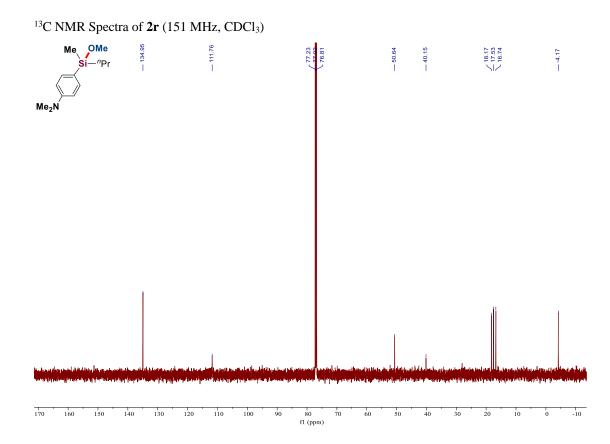




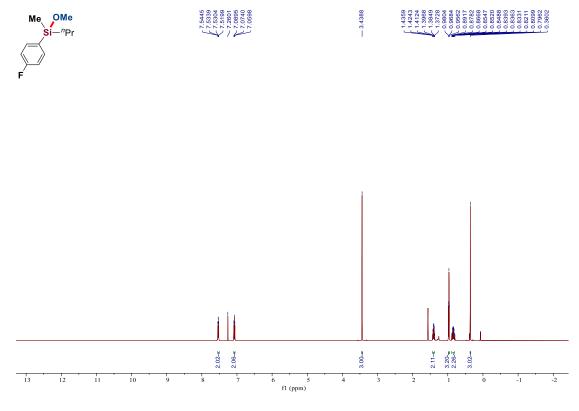


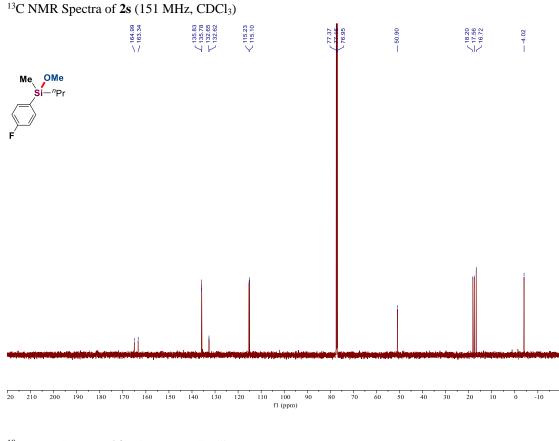






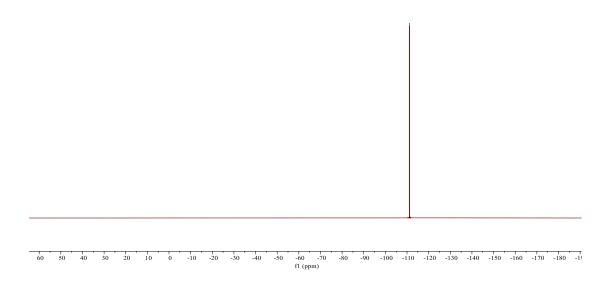
¹H NMR Spectra of **2s** (600 MHz, CDCl₃)

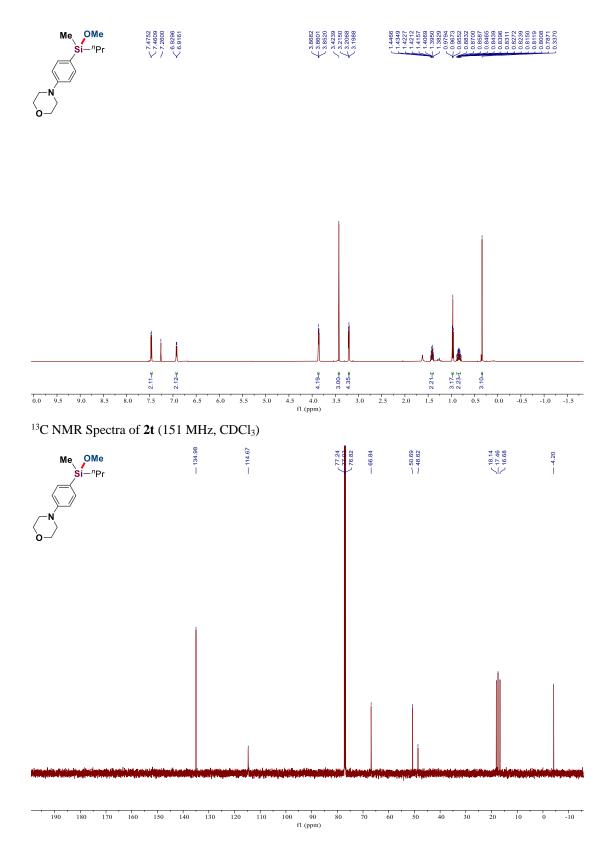




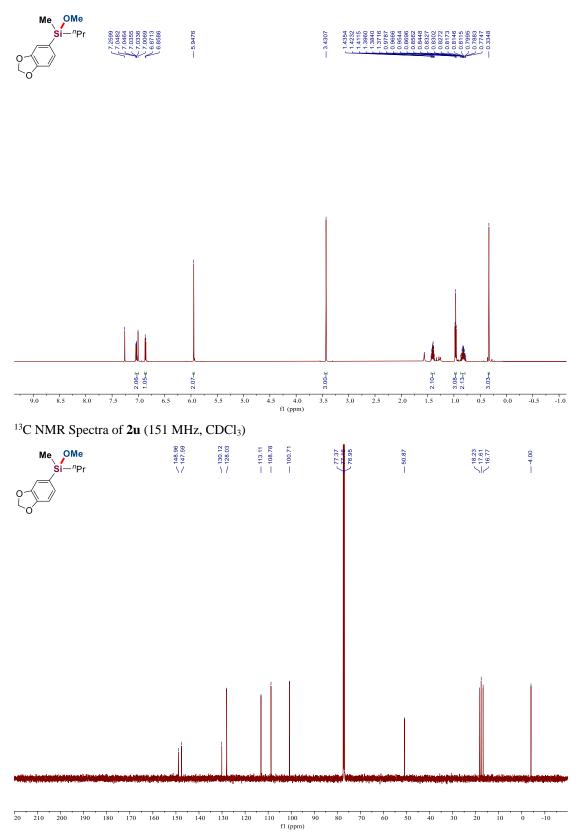
¹⁹F NMR Spectra of **2s** (377 MHz, CDCl₃)

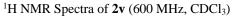


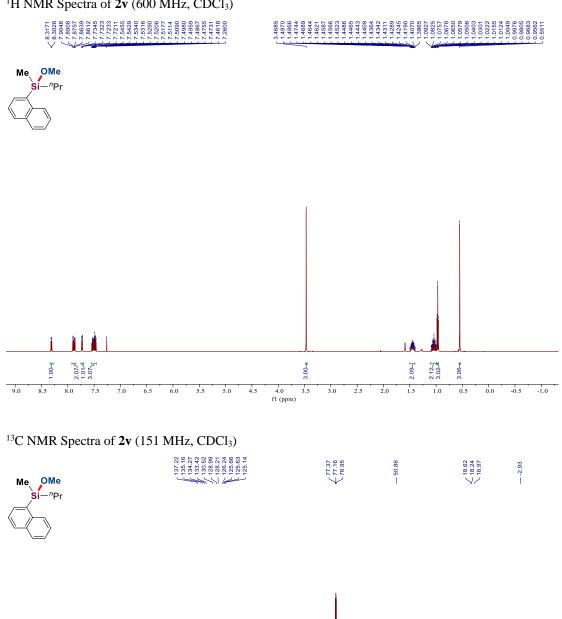


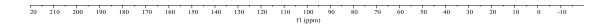


¹H NMR Spectra of **2u** (600 MHz, CDCl₃)

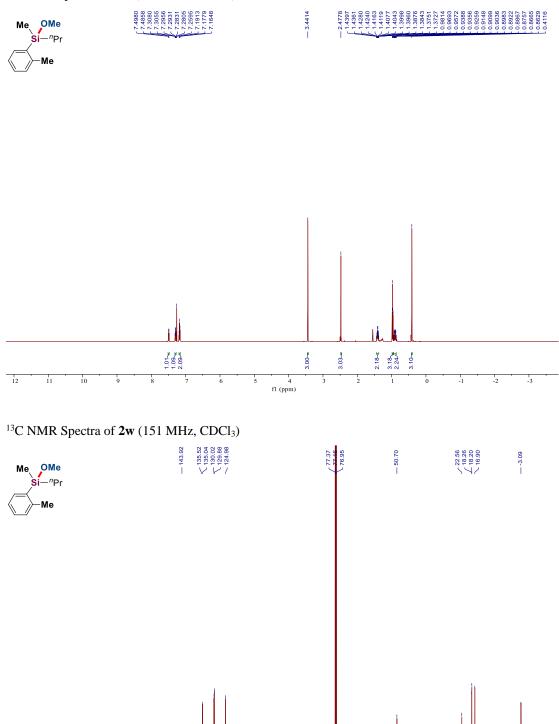




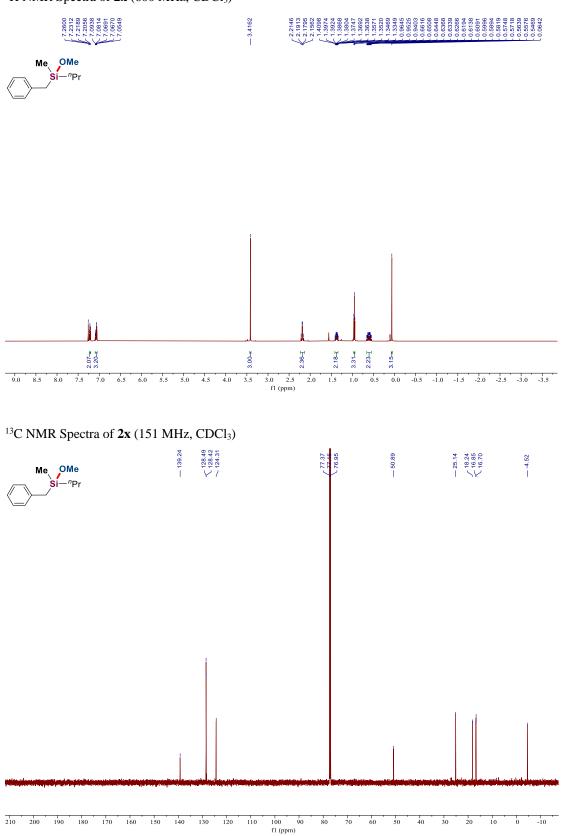




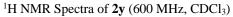
¹H NMR Spectra of **2w** (600 MHz, CDCl₃)

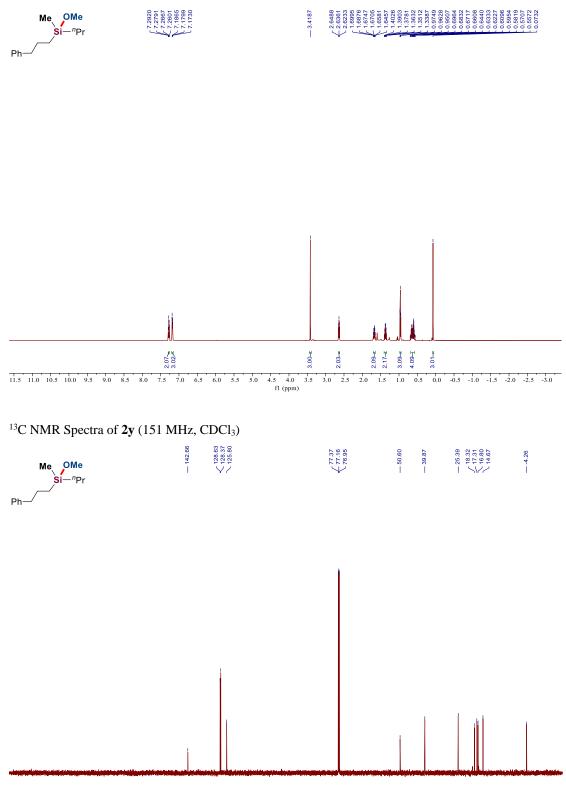


¹H NMR Spectra of **2x** (600 MHz, CDCl₃)

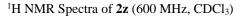


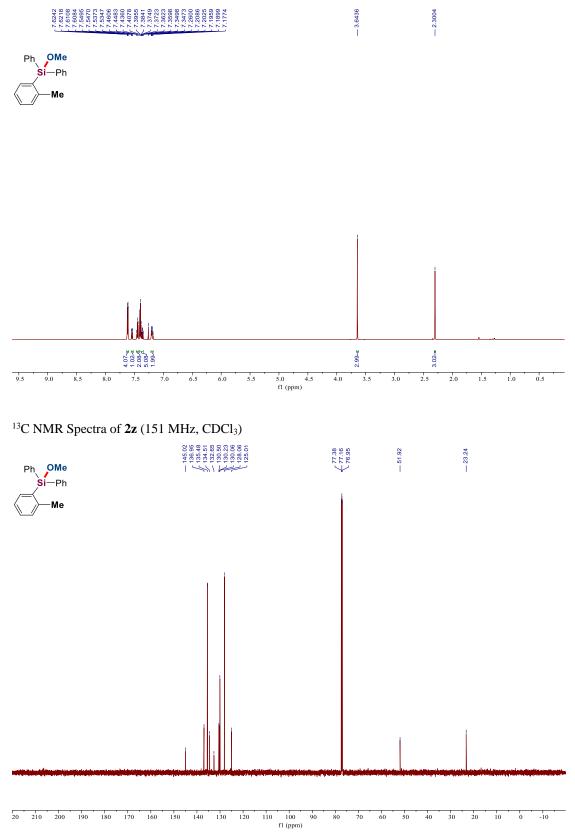
S44



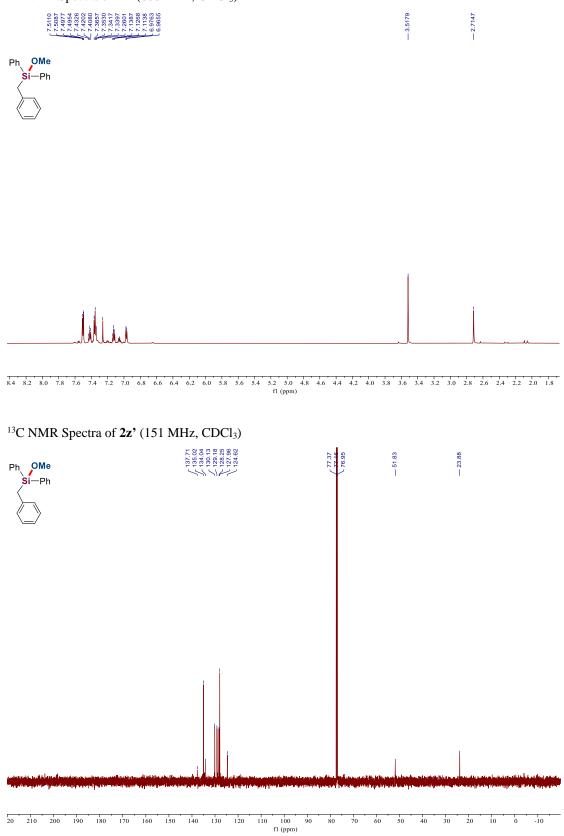


20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm)

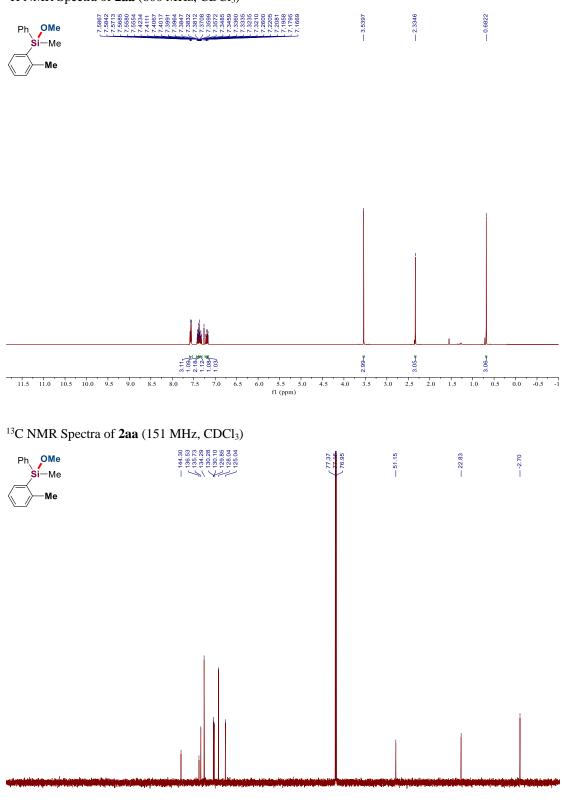




¹H NMR Spectra of **2z'** (600 MHz, CDCl₃)

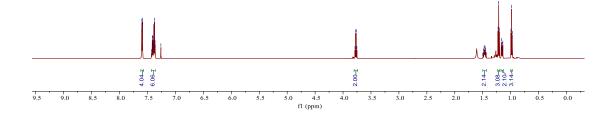


¹H NMR Spectra of **2aa** (600 MHz, CDCl₃)



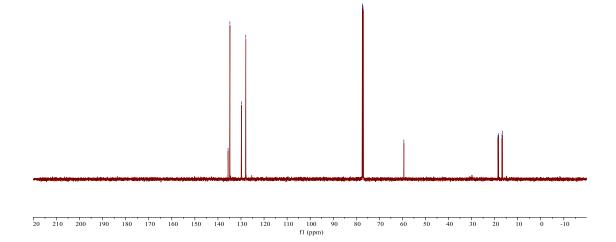
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 fl (ppm) ¹H NMR Spectra of **2ab** (600 MHz, CDCl₃)



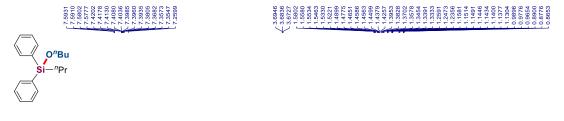


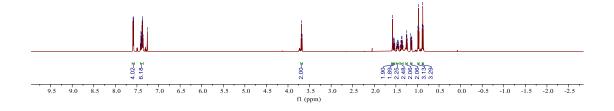
¹³C NMR Spectra of **2ab** (151 MHz, CDCl₃)



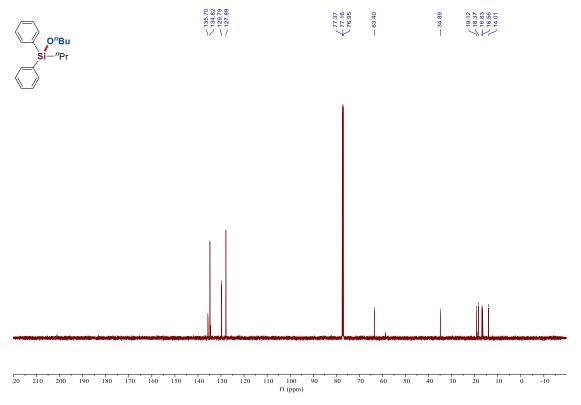


¹H NMR Spectra of **2ac** (600 MHz, CDCl₃)

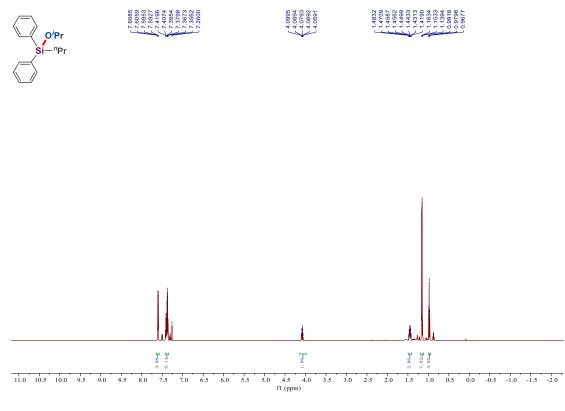




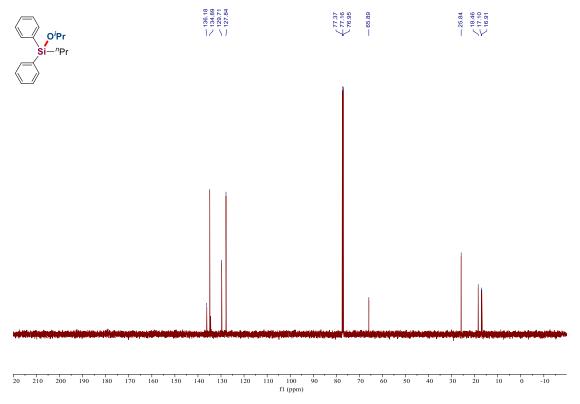
¹³C NMR Spectra of **2ac** (151 MHz, CDCl₃)



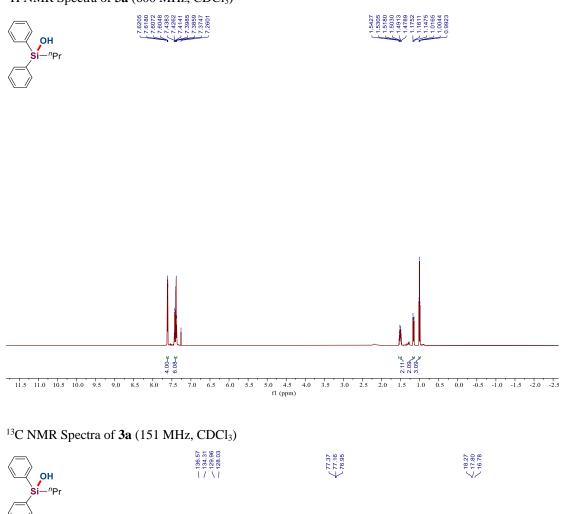
¹H NMR Spectra of **2ad** (600 MHz, CDCl₃)

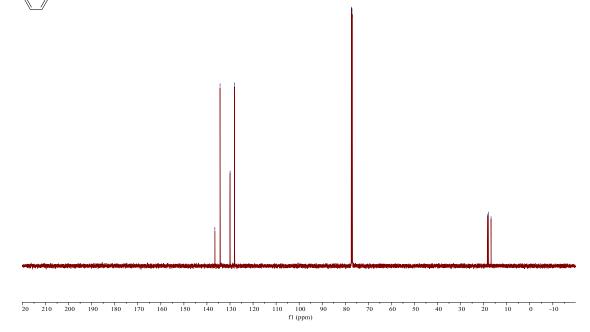


¹³C NMR Spectra of 2ad (151 MHz, CDCl₃)

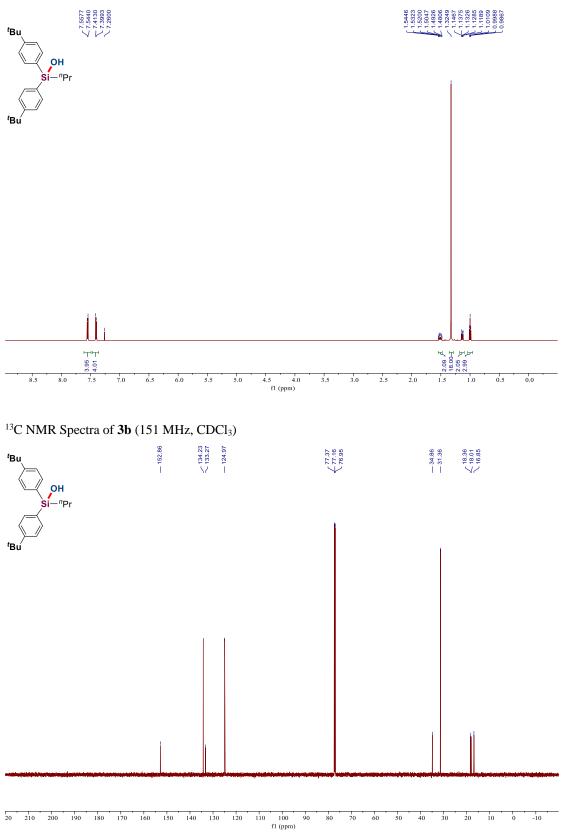


¹H NMR Spectra of **3a** (600 MHz, CDCl₃)

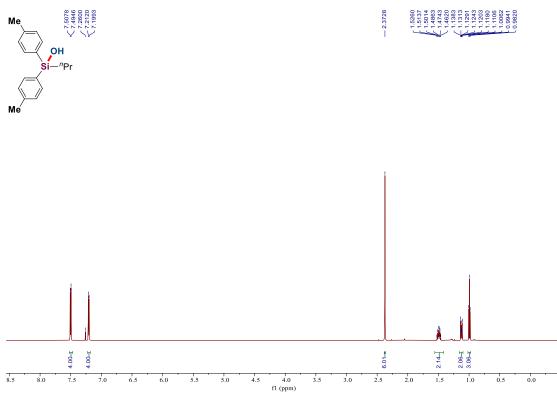




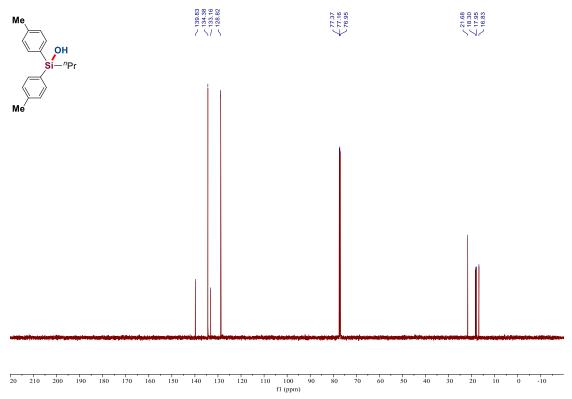
¹H NMR Spectra of **3b** (600 MHz, CDCl₃)



¹H NMR Spectra of **3c** (600 MHz, CDCl₃)

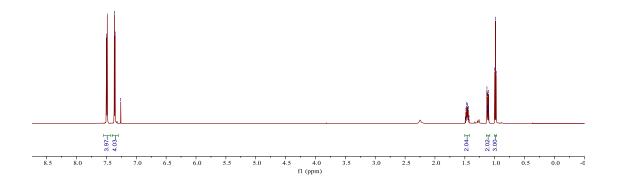


¹³C NMR Spectra of **3c** (151 MHz, CDCl₃)

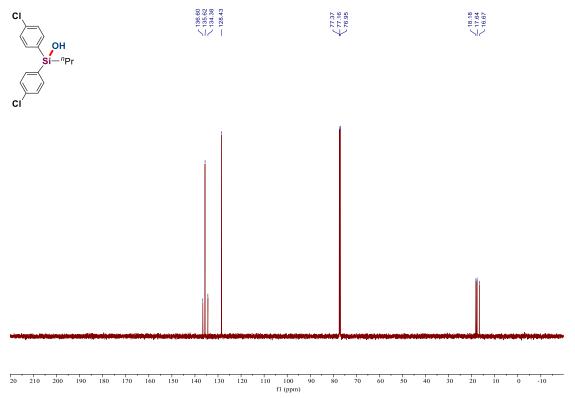


¹H NMR Spectra of **3d** (600 MHz, CDCl₃)



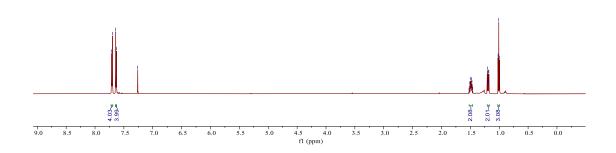


¹³C NMR Spectra of **3d** (151 MHz, CDCl₃)



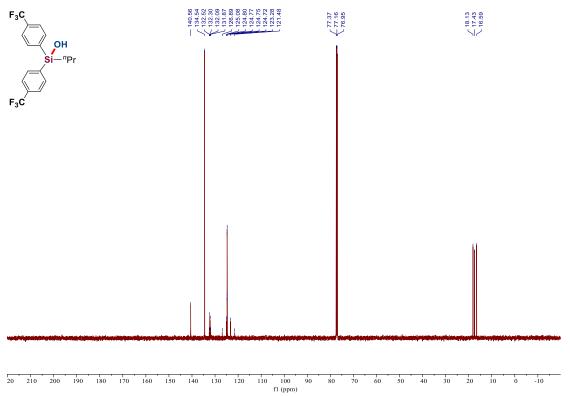
¹H NMR Spectra of **3e** (600 MHz, CDCl₃)

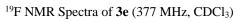


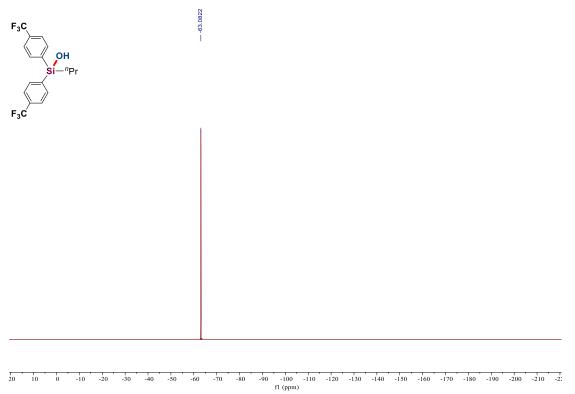


1.5235 1.5113 1.5113 1.4987 1.4839 1.4719 1.4719 1.2668 1.1974 1.1927 1.1927 1.1927 1.1793 1.1793 0.09957 0.9957

¹³C NMR Spectra of **3e** (151 MHz, CDCl₃)





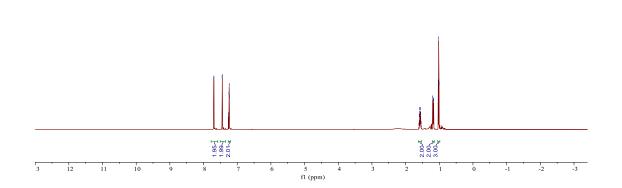


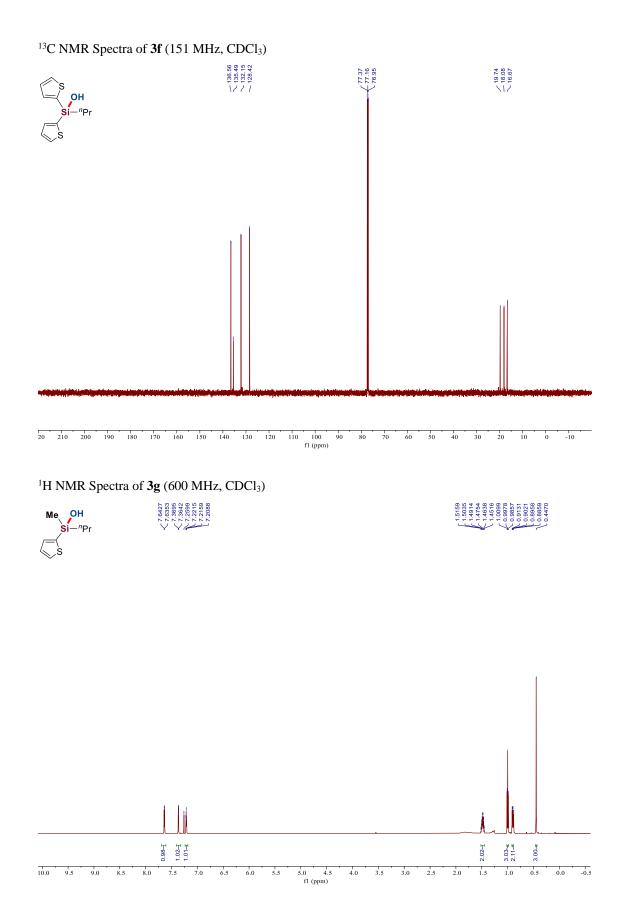
¹H NMR Spectra of **3f** (600 MHz, CDCl₃)

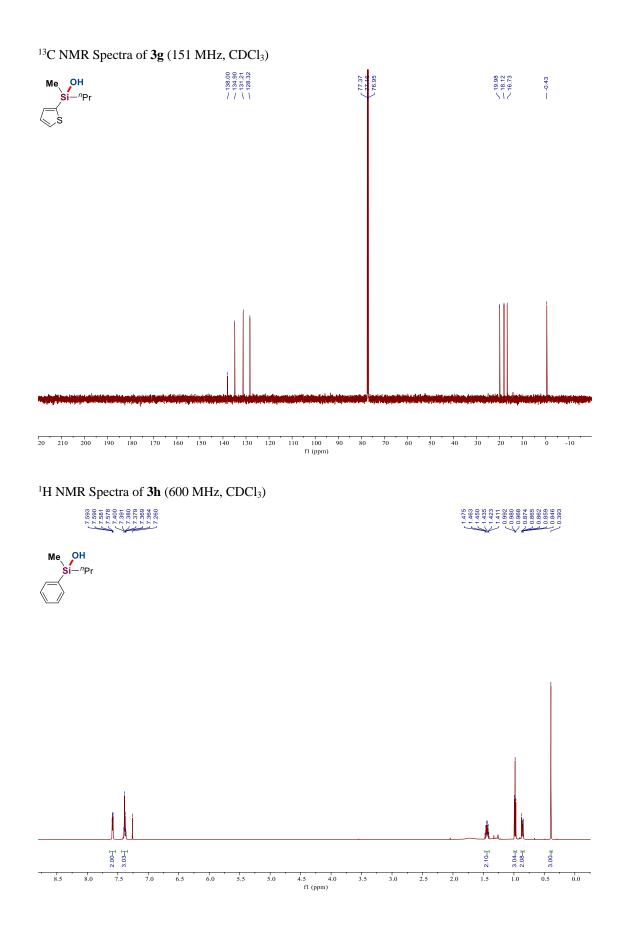


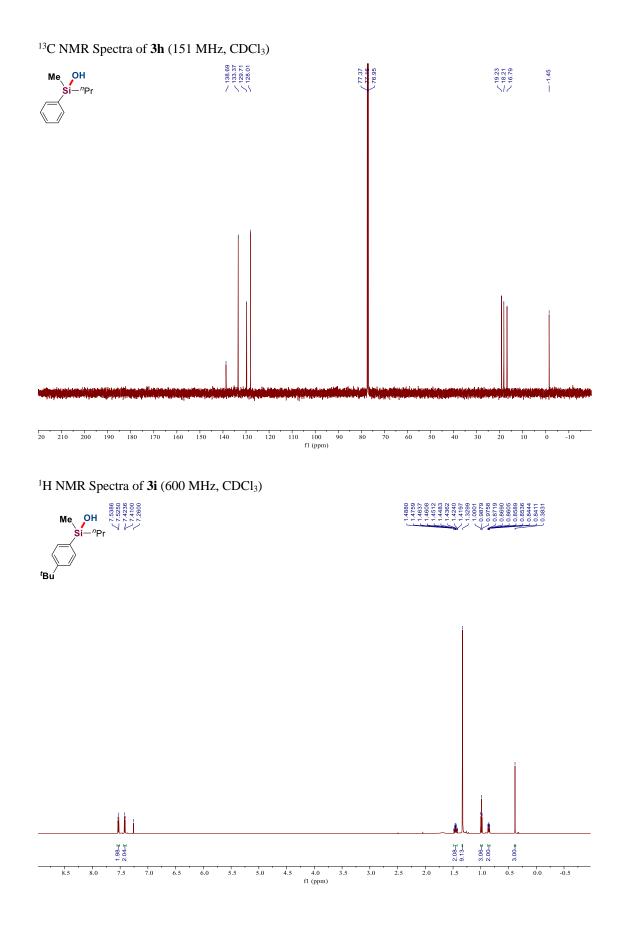
7.6983 7.4491 7.4491 7.2405 7.2449 7.2382

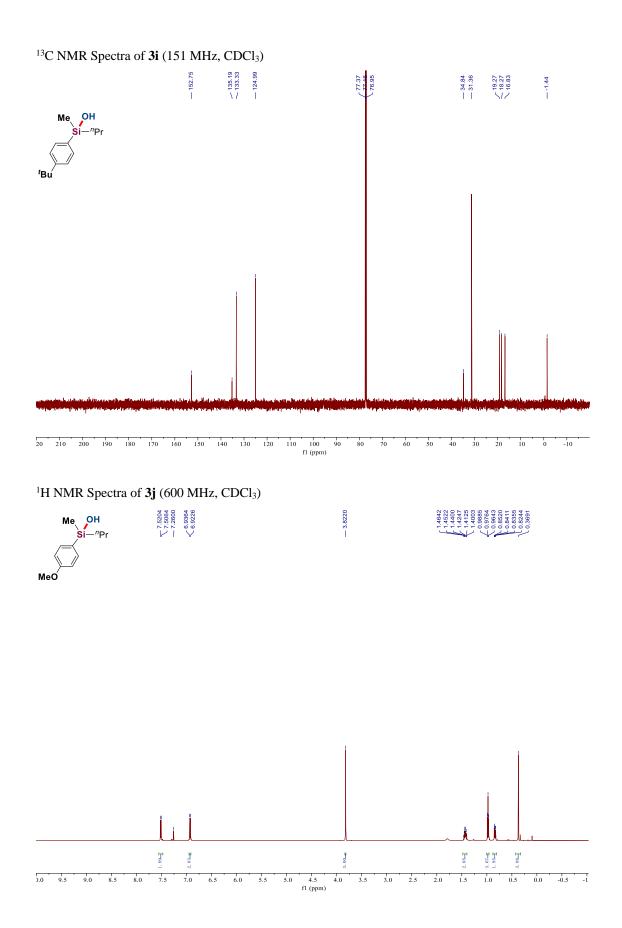




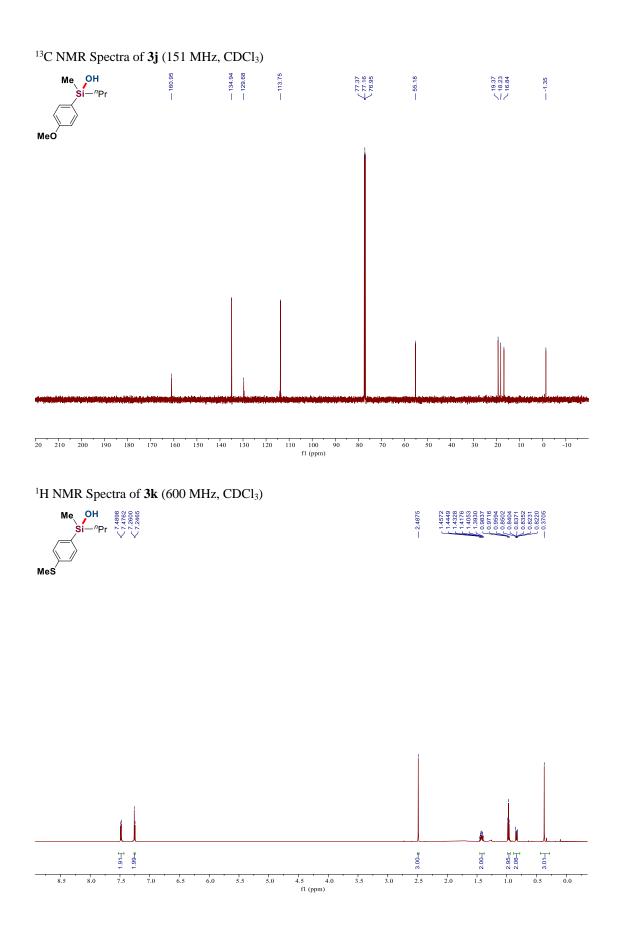


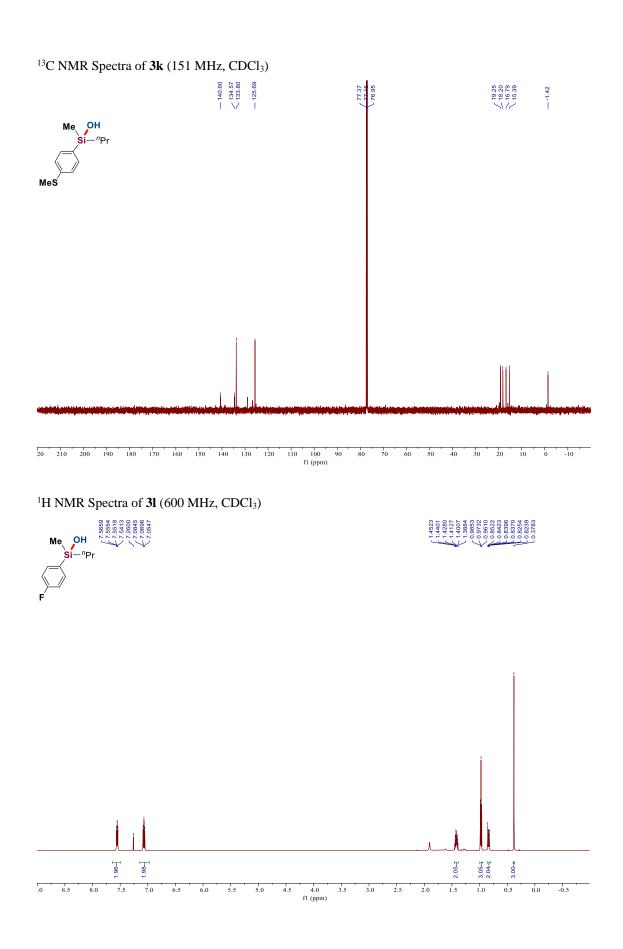


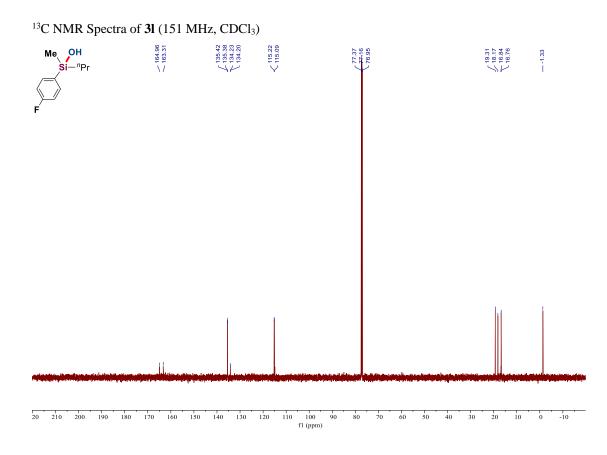




S61

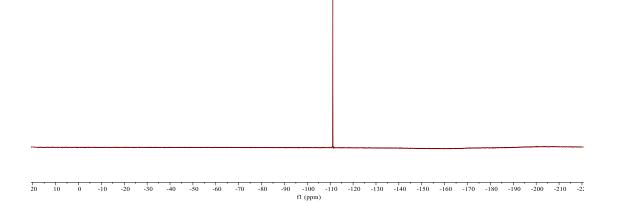






¹⁹F NMR Spectra of **3l** (377 MHz, CDCl₃)

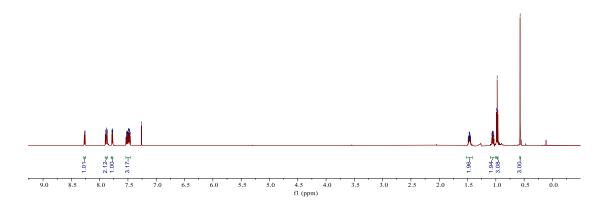




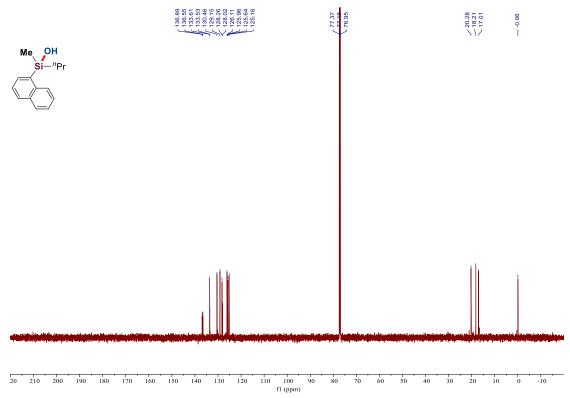
¹H NMR Spectra of **3m** (600 MHz, CDCl₃)

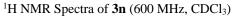


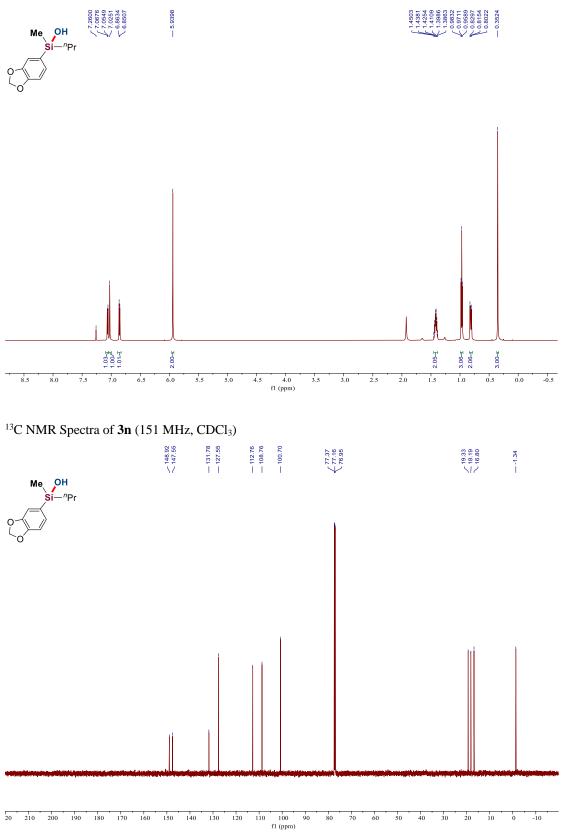




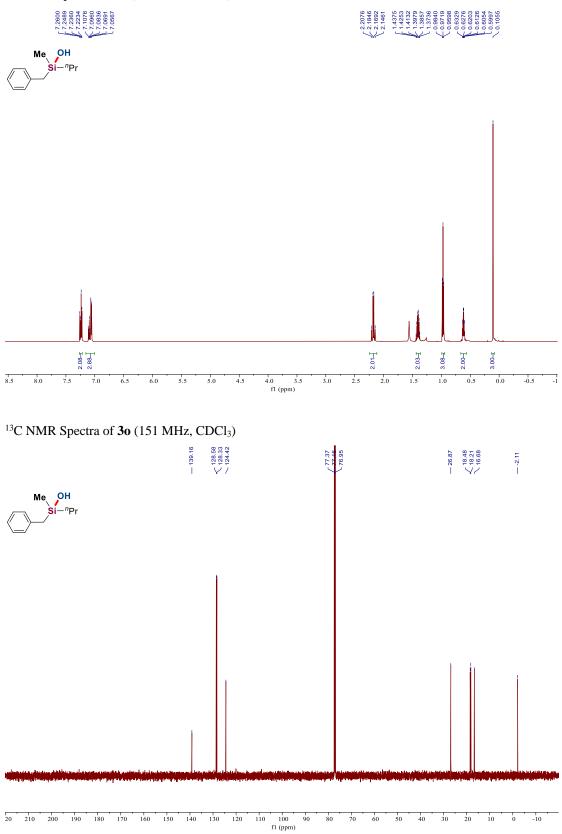
¹³C NMR Spectra of **3m** (151 MHz, CDCl₃)



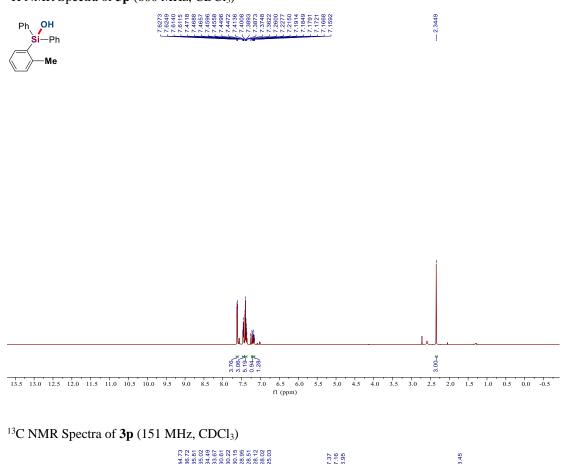


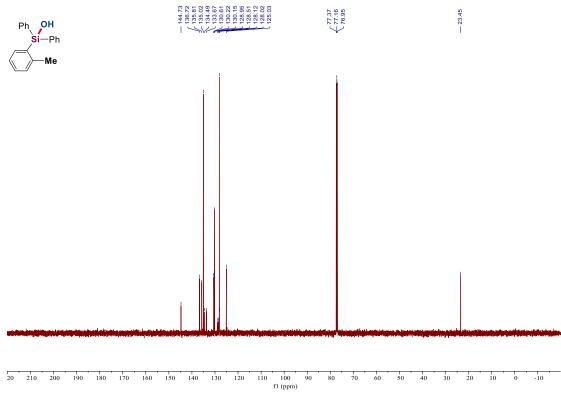


¹H NMR Spectra of **30** (600 MHz, CDCl₃)



¹H NMR Spectra of **3p** (600 MHz, CDCl₃)





¹H NMR Spectra of **3q** (600 MHz, CDCl₃)



