Supporting Information for

Cu-catalyzed efficient construction of S (Se)-containing functional organosilicon compounds

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Contents

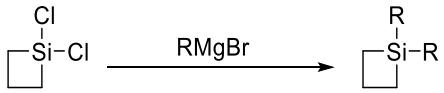
Ⅰ. General Information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents for chromatography were analytical grade and used without further purification. Anhydrous DMF, was purchased from Beijing InnoChem Science & Technology Co., Ltd. Analytical thin-layer chromatography (TLC) was performed on silica gel, visualized by irradiation with UV light. For column chromatography, 300-400 mesh silica gel was used. ^{1}H-NMR and ^{13}C-NMR were recorded on a BRUKER 400 MHz spectrometer in $CDCl_3$. Chemical shifts (δ) were reported referenced to an internal tetramethylsilane standard or the $CDCl_3$ residual peak (δ 7.26) for ^{1}H NMR. Chemical shifts of ^{13}C NMR are reported relative to $CDCl_3$ (δ 77.16). Data are reported in the following order: chemical shift (δ) in ppm; multiplicities are indicated s (singlet), bs (broad singlet), d (doublet), t (triplet), m (multiplet); coupling constants (J) are in Hertz (Hz). IR spectra were recorded on a BRUKER VERTEX 70 spectrophotometer and are reported in terms of frequency of absorption (cm^{-1}). HRMS spectra were obtained by using GCT Premier TOF-MS with CI source. The starting materials were isolated by SepaBean machine Flash Chromatography, which purchased from Santai Technologies Inc.

II. Synthesis of Substrates

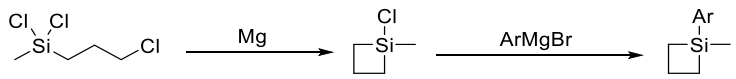
General procedure for the synthesis of Silacyclobutanes (SCBs).

1. Synthesis of symmetric SCB 1a-1i, 1l [1]



To an oven-dried 50 mL of round bottom flask equipped with a stirring bar under Ar atmosphere, the solution of 1,1-dichlorosiletane (3.0 mmol, 1.0 equiv) in anhydrous THF (15 mL) was added followed by the addition of commercial available Grignard reagent (6.3 mmol, 2.1 equiv) at 0 oC. The mixture was stirred at ambient temperature for 4 h. The mixture was then quenched by water (5 mL) and saturated aqueous NH_4Cl solution (15 mL). The organic phase phase was separated, and the aqueous layer was extracted with EtOAc (20 mL × 2). The combined organic layers were dried over MgSO4 and concentrated under reduced pressure to afford crude product. The residue was purified by flash column chromatography on silica gel using petroleum ether or petroleum ether/EtOAc to afford corresponding SCB **1a-1i, 1l**.

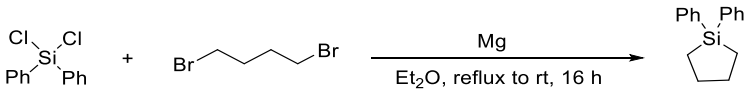
2. Synthesis of unsymmetric SCB 1j, 1k [1, 2]



In a 100-mL round-bottom flask, 1,2-dibromoethane (3 drops), magnesium (1.90 g, 80 mmol), and dry THF (5 mL) were added and stirred at ambient temperature for 30 min under nitrogen. Then 3-chloropropylmethyldichlorosilane (9.50 mL, 60 mmol) in THF

(45 mL) was added dropwise over a period of 15 min and the reaction mixture was heated to 50 ºC for another 2 h followed by the slow addition of commercial available RMgBr (3.3 mmol, 1.1 equiv) at 0 ºC. The mixture was stirred for 4 h at ambienttemperature. The mixture was then quenched by water (5 mL) and saturated aqueous NH_4Cl solution (15 mL). The organic phase phase was separated, and the aqueous layer was extracted with EtOAc (20 mL×2). The combined organic layers were dried over MgSO4 and concentrated under reduced pressure to afford crude product. The residue was purified by flash column chromatography on silica gel using petroleum ether or petroleum ether/EtOAc to afford corresponding SCB **1j**, **1k**.

3. Synthesis of 1,1-diphenylsilolane and 1,1-diphenylsilinane. [3]



A portion of 1 mL of a solution of 1,4-dibromobutane (1.54 mL, 13 mmol) in diethyl ether (5 mL) was added to a stirred suspension of magnesium turnings (729 mg, 30 mmol) in diethyl ether (5 mL), and the reaction was started by gentle heating. Subsequently, the remaining 1,4-dibromobutane solution was added within 2 h, causing the mixture to boil under reflux. After the addition was complete, the mixture was heated under reflux for a further 90 min and then cooled to 20 ℃ within 1 h. The resulting two-phase Grignard reagent was added dropwise within 2 h to a solution of dichlorodiphenylsilane (2.08 mL, 10 mmol) in diethyl ether (5 mL), causing the mixture to boil under reflux. The reaction mixture was stirred at 20 ℃ for 16 h and acidified with 1N HCl. The resulting mixture was extracted with diethylether, the organic layer was washed with $NaHCO_3$ aq., brine, and dried over Na_2SO_4, filtered and concentrated. The residue was chromatographed on silica gel (Hexane) to afford the target product as a colorless oil.

General procedure for the synthesis of $PhSO_2SNa$. [4]

Sodium benzenesulfinate (10 g, 61 mmol) and sulfur (1.95 g, 61 mmol) were dissolved in anhydrous pyridine (60 mL) to give a yellow solution. The reaction was stirred under argon and after 1 h gave a white suspension. Et_2O was added to the suspension, and the reaction was filtered and washed with anhydrous diethyl ether. Recrystallization from anhydrous ethanol afforded $PhSO_2SNa$ (10.5 g, 88%) as a white crystalline solid.

General procedure for the synthesis of $PhSO_2SAr$ or $PhSO_2SeAr$.[4]

A mixture of $PhSO_2Na$ (4 equiv), disulphide (1 equiv) and NBS (2 equiv) in MeCN was stirred at room temperature. After the completion of the reaction, as monitored by TLC, the reaction mixture was washed with water and extracted with ethyl acetate. The organic phase was separated and dried over anhydrous Na_2SO_4 and filtered. The filtrate was concentrated and the resulting residue was purified by column chromatography to provide the desired aryl-thiosulfonates.

General procedure for the synthesis of $PhSO_2SAlkyl$.

To a solution of $PhSO_2SNa$ (1 equiv) in DMF was added Alkyl bromide (2 equiv) and the reaction mixture was stirred at room temperature. After the completion of the reaction, as monitored by TLC, the reaction mixture was diluted with ethyl acetate and washed with water. The organic phase was separated and dried over anhydrous Na_2SO_4 and filtered. The filtrate was concentrated and the resulting residue was purified by column chromatography to provide the desired alkylthiosulfonates.

III .General Procedure

A representative procedure synthesis of diphenyl(3-(phenylthio)propyl)silanol (3a) is shown below.

An oven-dried screw-capped 8-mL vial equipped with a magnetic stir bar was charged with 1,1-diphenylsiletane **1a** (0.6 mmol, 1.5 equiv), *S*-phenyl benzenesulfonothioate **2a** (0.4 mmol, 1.0 equiv), $Cu(MeCN)_4PF_6$ (5 mol %), DMA (1.0 mL) was added via syringe under N_2 atmosphere and the mixture was stirred at 40 °C (oil bath). After 12 h, cooled to r.t. The system was extracted with EA (3 times). The combined organic layers were dried over Na_2SO_4 and solvent was removed under reduced pressure. The residue was purified by flash column chromatography with ethyl acetate and petroleum ether (EA : PE = 1 : 20) as eluents to afford pure product **3a**. Yield 68% (95.2 mg) (Note: The products obtained in this paper are perishable and need to be stored at low temperature).

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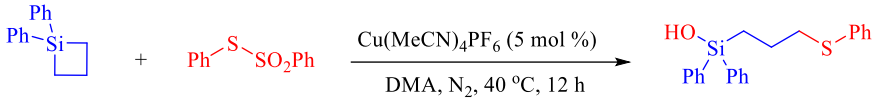
The procedure scale-up synthesis of diphenyl(3-(phenylthio)propyl)silanol (3a) is shown below.

An oven-dried screw-capped 8-mL vial equipped with a magnetic stir bar was charged with 1,1-diphenylsiletane **1a** (1.5 mmol, 1.5 equiv), *S*-phenyl benzenesulfonothioate **2a** (1.0 mmol, 1.0 equiv), $Cu(MeCN)_4PF_6$ (5 mol %), DMA (3.0 mL) was added via syringe under N_2 atmosphere and the mixture was stirred at 40 °C (oil bath). After 12 h, cooled to r.t. The system was extracted with EA (3 times). The combined organic layers were dried over Na_2SO_4 and solvent was removed under reduced pressure. The residue was purified by flash column chromatography with ethyl acetate and petroleum ether (EA : PE = 1 : 20) as eluents to afford pure product **3a**. Yield 75% (0.2684 g).

IV. Optimization of the reaction conditions



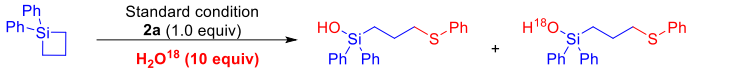
1a **2a** **3a**

entry	Devation from standard conditions	3a(%)
1	none	70 (68)[c]
2	DMF, DMSO	51, 42
3	DCE, MeCN	N.R
4	$Cu(OTf)_2$ instead of $Cu(MeCN)_4PF_6$	30
5	CuI instead of $Cu(MeCN)_4PF_6$	49
6	CuTc instead of $Cu(MeCN)_4PF_6$	57
7	CuBr instead of $Cu(MeCN)_4PF_6$	39
8	$CuBr_2$ instead of $Cu(MeCN)_4PF_6$	trace
9	10 mol% instead of 5 mol%	52
10	20 °C, 60 °C instead of 40 °C	68, 46
11	add bipyridine, phenanthroline	33-54
12	**1a** : **2a** = 1.0 : 1.0, 2.0 : 1.0	59, 48

Initially, we tested the reaction of 1,1-diphenylsiletane **1a** with S-phenyl benzenesulfonothioate **2a** and $Cu(OAc)_2$ (5 mol %) in DMF at 40 °C for 12 hours under a N_2 atmosphere. Gratifyingly, the reaction proceeded selectively to give the target product diphenyl(3-(phenylthio)propyl)silanol **3a**. With this promising result in hand, we tried to further optimize the reaction conditions. As briefly illustrated in Table 1, first, we probed the reactions in several different solvents, such as DMF, DMA, DMSO, and MeCN. DMA was crucial for the successful transformation of substrate **1a** (Table 1, entries 2-3). After systematic testing with copper catalysts, $Cu(MeCN)_4PF_6$ was chosen as the catalyst because of its highest yield (Table 1, entries 4-8). Next, we conducted the amount of catalyst. $Cu(MeCN)_4PF_6$ (5 mol %) provided inferior results (Table 1, entry 9). We investigated the effect of temperature on the reaction. It was found that neither raising the temperature nor lowering the temperature increased the yield (Table 1, entry 10). Finally, we tried to add ligands to increase the yield, but disappointingly, the yield of **3a** could not be increased (Table 1, entry 11). Finally, we investigated the ratio effects of **1a** and **2a**. When the ratio of **1a** to **2a** was 1.5:1.0, **3a** could be obtained in 70% LC yield (Table 1, entry 12).

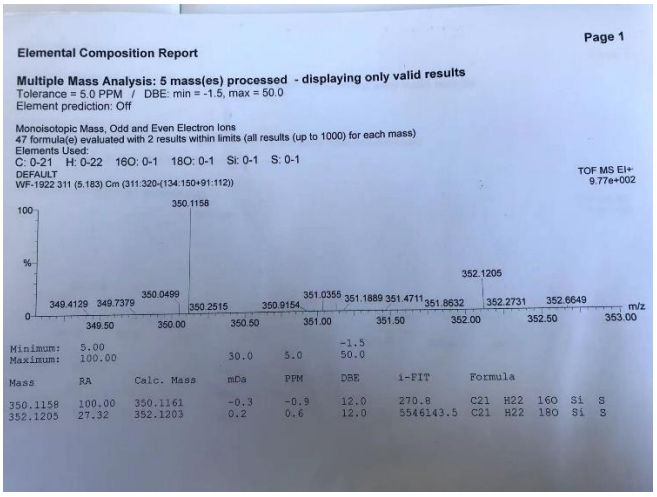
V. Mechanism Investigation

1. Heavy-oxygen water experiment



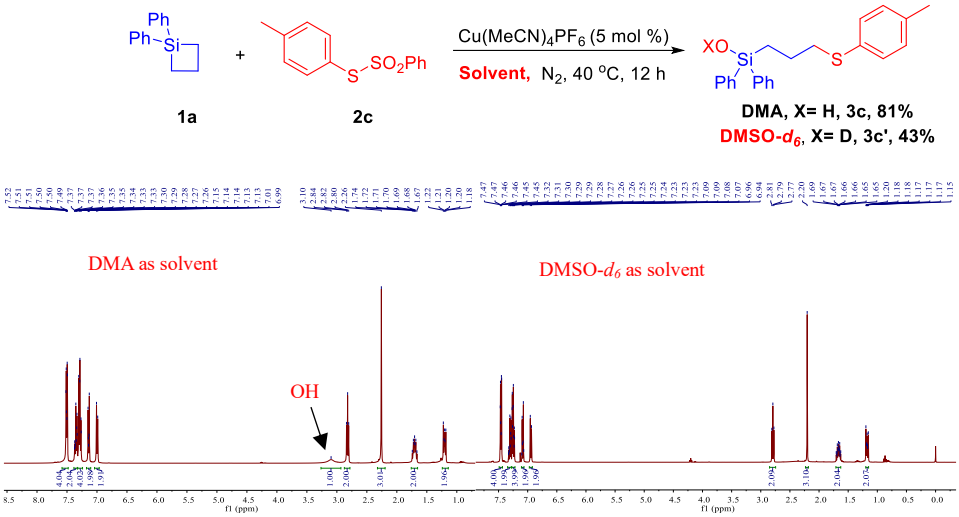
1a **3a**, 350.1161, found 350.1170 **3a**, 352.1203, found 352.1205

3a : 3a' = 100 : 27



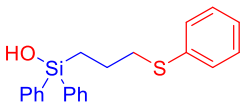
When heavy-oxygen water ($H_2{}^{18}O$) was added, only a small part of the ^{18}O-labeled product [^{18}O]-3a' could be detected by HRMS.

2. Deuteration experiment



We investigated the source of hydroxyl hydrogen. The deuterated product **3c'** (D-inc. 100%) was successfully obtained when deuterated DMSO replaced DMA as the solvent, indicating that the hydroxyl hydrogen may come from the solvent.

VI. Product Characterization

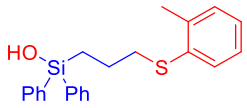


diphenyl(3-(phenylthio)propyl)silanol (3a)

Yield: 68% (95.2mg). (EA : PE = 1 : 20). Pale yellow oil.

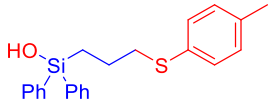
IR (neat, ν, cm^{-1}): 3003, 1710, 1358, 1220, 1092, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 – 7.47 (m, 4H), 7.40 – 7.34 (m, 2H), 7.31 (m, *J* = 7.8, 6.3 Hz, 4H), 7.26 – 7.17 (m, 4H), 7.14 – 7.07 (m, 1H), 2.89 (t, *J* = 7.1 Hz, 3H), 1.83 –

1.70 (m, 2H), 1.29 – 1.20 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.6, 135.9, 134.2, 130.0, 129.2, 128.9, 128.0, 125.8, 36.9, 22.8, 14.5. **HRMS** (CI^+) m/z calcd for $C_{22}H_{24}OSSi$ $(M)^+$: 350.1161, found 350.1170.



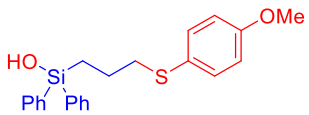
diphenyl(3-(*o*-tolylthio)propyl)silanol (3b)

Yield: 87% (127.2mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1359, 1220, 871, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 – 7.47 (m, 4H), 7.38 – 7.32 (m, 2H), 7.29 (m, J = 7.9, 6.4 Hz, 4H), 7.06 (m, J = 29.3, 14.1, 7.4, 1.7 Hz, 4H), 2.83 (t, J = 7.2 Hz, 3H), 2.30 (s, 3H), 1.83 – 1.69 (m, 2H), 1.27 – 1.19 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.3, 136.0, 135.9, 134.2, 130.1, 130.0, 128.0, 127.7, 126.4, 125.4, 36.0, 22.7, 20.4, 14.7. **HRMS** (CI) m/z calcd for $C_{22}H_{24}OSSi$ $(M)^+$:364.1317, found 364.1320.



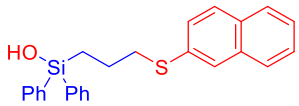
diphenyl(3-(*p*-tolylthio)propyl)silanol (3c)

Yield: 81% (117.3mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1358, 1220, 1092, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 4H), 7.38 – 7.33 (m, 2H), 7.29 (m, J = 7.9, 6.5 Hz, 4H), 7.17 – 7.10 (m, 2H), 7.00 (m, J = 8.0 Hz, 2H), 3.10 (s, 1H), 2.82 (t, J = 7.1 Hz, 2H), 2.26 (s, 3H), 1.76 – 1.65 (m, 2H), 1.24 – 1.14 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.0, 135.9, 134.2, 132.7, 130.0, 129.9, 129.7, 128.0, 37.6, 22.8, 21.1, 14.4. **^{29}Si NMR** (79 MHz, $CDCl_3$) δ -4.22. **HRMS** (CI) m/z calcd for $C_{22}H_{24}OSSi$ $(M)^+$:364.1317, found 364.1323.



(3-((4-methoxyphenyl)thio)propyl)diphenylsilanol (3d)

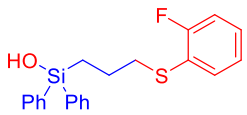
Yield: 56% (85.8mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1428, 1358, 1220, 881, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 – 7.58 (m, 4H), 7.49 – 7.43 (m, 2H), 7.40 (m, J = 7.8, 6.3 Hz, 4H), 7.33 – 7.27 (m, 2H), 6.87 – 6.79 (m, 2H), 3.81 (s, 3H), 2.88 (t, J = 7.0 Hz, 3H), 1.84 – 1.71 (m, 2H), 1.35 – 1.28 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.8, 136.0, 134.2, 133.2, 130.0, 128.0, 126.5, 114.6, 55.4, 39.1, 22.9, 14.3. **HRMS** (CI) m/z calcd for $C_{22}H_{24}O_2SSi$ $(M)^+$: 380.1266, found 380.1274.



(3-(naphthalen-2-ylthio)propyl)diphenylsilanol (3e)

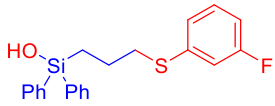
Yield: 70% (111.4mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1709, 1420, 1358, 1220, 1092, 785, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 (m, J = 7.7, 1.6 Hz, 1H), 7.67 – 7.61 (m, 3H), 7.50 (m, J = 7.9, 1.2 Hz, 4H), 7.43 – 7.36 (m, 2H), 7.32 (m, J = 6.3, 4.8, 1.7 Hz, 3H), 7.26 (m, J = 7.8, 6.5 Hz, 4H), 2.96 (t, J = 7.1 Hz, 2H), 2.62 (s, 1H), 1.86 – 1.74 (m, 2H), 1.29 – 1.23 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.9, 134.2, 133.8, 131.7, 130.0, 128.4, 128.0, 127.8, 127.4, 127.1, 126.8, 126.5, 125.6, 36.7, 22.8, 14.5. **HRMS** (CI)

m/z calcd for $C_{25}H_{24}OSSi$ $(M)^+$: 400.1317, found 400.1323.



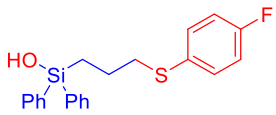
(3-((2-fluorophenyl)thio)propyl)diphenylsilanol (3f)

Yield: 90% (131.8mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1428, 1359, 1220, 1116, 1046, 871, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 – 7.46 (m, 4H), 7.38 – 7.32 (m, 2H), 7.32 – 7.25 (m, 4H), 7.21 (m, *J* = 7.5, 1.8 Hz, 1H), 7.10 (m, *J* = 7.8, 5.1, 1.7 Hz, 1H), 7.00 – 6.92 (m, 2H), 3.19 (s, 1H), 2.84 (t, *J* = 7.1 Hz, 2H), 1.69 (ddt, *J* = 11.8, 8.8, 6.1 Hz, 2H), 1.24 – 1.15 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.3 (*J* = 243 Hz), 135.8, 134.2, 131.8 (*J* = 2.6 Hz), 120.0, 128.1, 128.0, 124.4 (*J* = 38.4 Hz), 123.3 (*J* = 17.4 Hz), 115.6 (*J* = 22.4 Hz), 36.4 (*J* = 2.3 Hz), 22.9, 14.3. **HRMS** (CI) m/z calcd for $C_{21}H_{21}FOSSi$ $(M)^+$: 368.1066, found 368.1063.



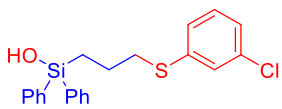
(3-((3-fluorophenyl)thio)propyl)diphenylsilanol (3g)

Yield: 73% (107.5mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1710, 1473, 1359, 1092, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 (m, *J* = 6.6, 1.5 Hz, 4H), 7.41 – 7.35 (m, 2H), 7.34 – 7.28 (m, 4H), 7.13 (m, *J* = 8.0, 6.0 Hz, 1H), 6.98 – 6.87 (m, 2H), 6.82 – 6.75 (m, 1H), 2.87 (t, *J* = 7.1 Hz, 3H), 1.75 (ddt, *J* = 11.9, 8.7, 6.3 Hz, 2H), 1.26 – 1.19 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) 162.9 (*J* = 245.9 Hz), 139.3 (*J* = 7.5 Hz), 135.8, 134.2, 130.2, 130.1, 128.1, 124.0 (*J* = 2.9 Hz), 115.1 (*J* = 23.0 Hz), 112.5 (*J* = 21.0 Hz), 36.3, 22.7, 14.5. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OSSi$ $(M)^+$: 368.1066, found 368.1068.



(3-((4-fluorophenyl)thio)propyl)diphenylsilanol (3h)

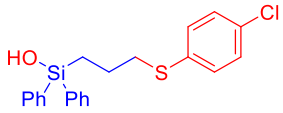
Yield: 72% (106.2mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1428, 1359, 1220, 1092, 871, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.64 – 7.59 (m, 4H), 7.50 – 7.44 (m, 2H), 7.41 (m, *J* = 7.9, 6.4 Hz, 4H), 7.32 – 7.27 (m, 2H), 7.02 – 6.94 (m, 2H), 3.02 (s, 1H), 2.91 (t, *J* = 7.1 Hz, 2H), 1.83 – 1.76 (m, 2H), 1.34 – 1.28 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 161.7 (*J* = 244.3 Hz), 135.9, 134.2, 132.3 (*J* = 8.0 Hz), 131.2 (*J* = 3.3 Hz), 130.1, 128.0, 116.0 (*J* = 21.7 Hz), 38.2, 22.8, 14.3. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OSSi$ $(M)^+$: 368.1066, found 368.1061.



(3-((3-chlorophenyl)thio)propyl)diphenylsilanol (3i)

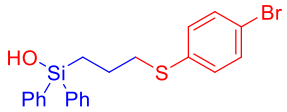
Yield: 75% (115.9mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1710, 1428, 1359, 1220, 1116, 870, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 4H), 7.40 – 7.34 (m, 2H), 7.33 – 7.27 (m, 4H), 7.22 – 7.18 (m, 1H), 7.13 – 7.00 (m, 3H), 2.85 (t, *J* = 7.1 Hz, 3H), 1.80 – 1.68 (m, 2H), 1.25 – 1.17 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.0, 135.8, 134.6,

134.2, 130.1, 129.9, 128.1, 128.1, 126.6, 125.7, 36.4, 22.7, 14.5. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OSSi$ $(M)^+$: 384.0771, found 384.0768.



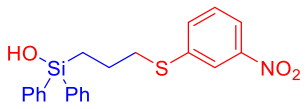
(3-((4-chlorophenyl)thio)propyl)diphenylsilanol (3j)

Yield: 72% (111.0mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1428, 1358, 1220, 1094, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (m, *J* = 6.7, 1.5 Hz, 4H), 7.41 – 7.35 (m, 2H), 7.31 (m, *J* = 7.9, 6.4 Hz, 4H), 7.17 – 7.08 (m, 4H), 3.07 (s, 1H), 2.84 (t, *J* = 7.1 Hz, 2H), 1.76 – 1.67 (m, 2H), 1.25 – 1.19 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.8, 135.1, 134.2, 131.7, 130.5, 130.1, 129.0, 128.1, 37.1, 22.7, 14.4. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OSSi$ $(M)^+$: 384.0771, found 384.0763.



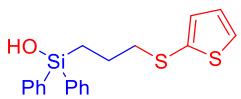
(3-((4-bromophenyl)thio)propyl)diphenylsilanol (3k)

Yield: 79% (135.9mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1358, 1094, 818, 743, 511. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 – 7.45 (m, 4H), 7.40 – 7.34 (m, 2H), 7.34 – 7.22 (m, 6H), 7.07 – 6.95 (m, 2H), 3.14 (s, 1H), 2.81 (t, *J* = 7.1 Hz, 2H), 1.78 – 1.63 (m, 2H), 1.25 – 1.09 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.8, 135.7, 134.2, 131.9, 130.6, 130.1, 128.0, 119.5, 36.8, 22.6, 14.4. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OBrSSi$ $(M)^+$: 428.0266, found 428.0271.



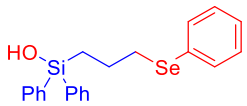
(3-((3-nitrophenyl)thio)propyl)diphenylsilanol (3l)

Yield: 74% (116.3mg). (EA : PE = 1 : 10). Yellow oil. **IR** (neat, ν, cm^{-1}):3011, 1702, 1523, 1346, 1113, 844, 729, 698. **^{1}H NMR** (400 MHz, $CDCl_3$)δ 7.99 (m, *J* = 2.1 Hz, 1H), 7.87 (m, *J* = 8.2, 2.3, 1.0 Hz, 1H), 7.59 – 7.49 (m, 4H), 7.42 (m, *J* = 7.9, 1.2 Hz, 1H), 7.39 – 7.34 (m, 2H), 7.29 (m, *J* = 7.7, 6.0 Hz, 5H), 3.28 (s, 1H), 2.93 (t, *J* = 7.2 Hz, 2H), 1.83 – 1.72 (m, 2H), 1.29 – 1.21 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 148.5, 140.0, 135.7, 134.1, 133.7, 130.1, 129.4, 128.0, 122.1, 120.1, 36.0, 22.5, 14.5. **HRMS** (CI) m/z calcd for $C_{21}H_{21}NO_3SSi$ $(M)^+$: 395.1011, found 395.1012.



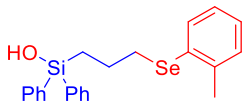
diphenyl(3-(thiophen-2-ylthio)propyl)silanol (3m)

Yield: 76% (108.6mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1428, 1359, 1220, 1116, 870, 703, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 – 7.47 (m, 4H), 7.42 – 7.35 (m, 2H), 7.35 – 7.28 (m, 4H), 7.22 (m, *J* = 5.3, 1.3 Hz, 1H), 6.96 (m, *J* = 3.5, 1.3 Hz, 1H), 6.86 (m, *J* = 5.4, 3.5 Hz, 1H), 2.99 (s, 1H), 2.74 (t, *J* = 7.0 Hz, 2H), 1.78 – 1.64 (m, 2H), 1.24 – 1.13 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.9, 134.5, 134.3, 133.5, 130.0, 129.0, 128.0, 127.5, 42.1, 22.9, 14.0. **HRMS** (CI) m/z calcd for $C_{19}H_{20}OS_2Si$ $(M)^+$: 356.0725, found 356.0734.



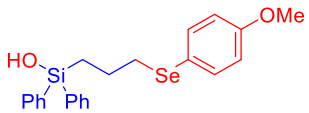
diphenyl(3-(phenylselanyl)propyl)silanol (3n)

Yield: 47% (71.1mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1428, 1358, 1220, 1092, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 – 7.57 (m, 4H), 7.50 – 7.35 (m, 8H), 7.40 (m, *J* = 7.6 Hz, 4H), 7.26 (m, *J* = 5.6, 3.7, 1.8 Hz, 3H), 2.98 (t, *J* = 7.2 Hz, 2H), 2.76 (s, 1H), 1.89 (dt, *J* = 15.3, 6.2 Hz, 2H), 1.35 – 1.30 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.9, 134.2, 132.6, 130.4, 130.0, 129.1, 128.0, 126.8, 31.6, 23.8, 15.6. **HRMS** (CI) m/z calcd for $C_{21}H_{22}OSeSi$ $(M)^+$: 398.0605, found 398.0611.



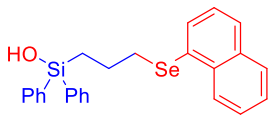
diphenyl(3-(*o*-tolylselanyl)propyl)silanol (3o)

Yield: 78% (128.9mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}):3002, 1710, 1428, 1358, 1220, 1092, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 – 7.48 (m, 4H), 7.39 – 7.33 (m, 2H), 7.32 – 7.25 (m, 5H), 7.13 – 7.04 (m, 2H), 7.00 (m, *J* = 7.4, 1.9 Hz, 1H), 2.85 (t, *J* = 7.3 Hz, 3H), 2.33 (s, 3H), 1.86 – 1.74 (m, 2H), 1.26 – 1.18 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.2, 135.9, 134.2, 131.6, 131.2, 130.0, 129.9(8), 129.9(7), 128.0, 126.5, 30.3, 23.6, 22.4, 15.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 238.0. **^{29}Si NMR** (79 MHz, $CDCl_3$) δ -4.35. **HRMS** (CI) m/z calcd for $C_{22}H_{24}OSeSi$ $(M)^+$: 412.0762, found 412.0760.



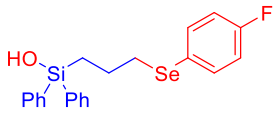
(3-((4-methoxyphenyl)selanyl)propyl)diphenylsilanol(3p)

Yield: 68% (116.3mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1358, 1220, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 – 7.48 (m, 4H), 7.40 – 7.34 (m, 3H), 7.34 – 7.27 (m, 5H), 6.75 – 6.68 (m, 2H), 3.72 (s, 3H), 2.80 (t, *J* = 7.1 Hz, 3H), 1.80 – 1.68 (m, 2H), 1.25 – 1.17 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.2, 136.0, 135.6, 134.2, 130.0, 128.0, 120.0, 114.8, 55.3, 32.8, 23.7, 15.5. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 273.6. **HRMS** (CI) m/z calcd for $C_{22}H_{24}O_2SeSi$ $(M)^+$: 428.0711, found 428.0721.



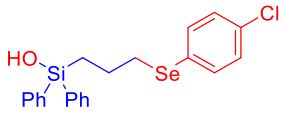
(3-(naphthalen-1-ylselanyl)propyl)diphenylsilanol (3q)

Yield: 78% (139.2mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1710, 1358, 1220, 745, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.33 (m, *J* = 8.5, 3.4 Hz, 1H), 7.75 (m, *J* = 7.6, 2.1 Hz, 1H), 7.67 (m, *J* = 8.2, 2.1 Hz, 1H), 7.60 (m, *J* = 7.3, 1.7 Hz, 1H), 7.52 – 7.38 (m, 6H), 7.36 – 7.28 (m, 2H), 7.24 (m, *J* = 7.2, 1.7 Hz, 5H), 2.87 (t, *J* = 7.1 Hz, 3H), 1.73 (ddt, *J* = 9.3, 7.0, 4.8 Hz, 2H), 1.22 – 1.13 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.9, 134.3, 134.2, 134.0, 132.0, 130.0, 129.6, 128.7, 128.1, 128.0, 127.6, 126.6, 126.2, 125.8, 31.9, 23.7, 15.6. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 223.5. **HRMS** (CI) m/z calcd for $C_{25}H_{24}OSeSi$ $(M)^+$: 448.0762, found 448.0764.



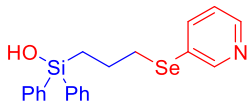
(3-((4-fluorophenyl)selanyl)propyl)diphenylsilanol (3r)

Yield: 72% (120.6mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1710, 1359, 1219, 1115, 868, 829, 702, 509. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 – 7.49 (m, 4H), 7.41 – 7.28 (m, 8H), 6.86 (m, *J* = 8.7 Hz, 2H), 2.89 (t, *J* = 7.1 Hz, 1H), 2.82 (t, *J* = 7.2 Hz, 2H), 1.80 – 1.69 (m, 2H), 1.25 – 1.15 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.3 (*J* = 244.9 Hz), 135.9, 135.3 (*J* = 7.9 Hz), 134.2, 130.1, 129.0 (*J* = 25.3 Hz), 128.0, 116.2 (*J* = 21.1 Hz), 32.5, 23.7, 15.5. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 282.3. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OFSeSi$ $(M)^+$:416.0511, found 416.0520.



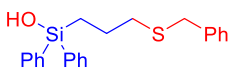
(3-((4-chlorophenyl)selanyl)propyl)diphenylsilanol (3s)

Yield: 57% (98.3mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1709, 1428, 1359, 1220, 1090, 869, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 (m, *J* = 7.7, 1.1 Hz, 4H), 7.42 – 7.35 (m, 2H), 7.35 – 7.23 (m, 6H), 7.15 – 7.09 (m, 2H), 2.85 (t, *J* = 7.2 Hz, 3H), 1.81 – 1.73 (m, 2H), 1.25 – 1.16 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.8, 134.2, 134.1, 132.9, 130.1, 129.2, 128.4, 128.1, 31.9, 23.7, 15.6. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 285.6. **HRMS** (CI) m/z calcd for $C_{21}H_{21}OClSeSi$ $(M)^+$: 432.0215, found 432.0221.



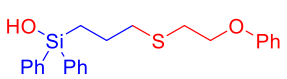
diphenyl(3-(pyridin-3-ylselanyl)propyl)silanol (3t)

Yield: 39% (62.5mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1709, 1414, 1220, 1111, 701, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.41 (m, *J* = 5.0, 1.9, 0.9 Hz, 1H), 7.66 – 7.56 (m, 4H), 7.45 – 7.24 (m, 8H), 6.98 (m, *J* = 7.3, 5.0, 1.2 Hz, 1H), 5.56 (s, 1H), 3.27 – 3.12 (m, 2H), 2.01 (p, *J* = 7.3 Hz, 2H), 1.31 (t, *J* = 7.3 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 155.6, 149.9, 137.0, 136.3, 134.2, 129.7, 127.9, 126.0, 120.4, 27.9, 27.9, 24.8, 14.9. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 348.8. **HRMS** (CI) m/z calcd for $C_{20}H_{21}OSeSiN$ $(M)^+$:399.0558 found 399.0563.



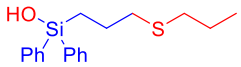
(3-(benzylthio)propyl)diphenylsilanol (5a)

Yield: 56% (80.9mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1710, 1428, 1359, 1220, 1092, 703, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.66 – 7.61 (m, 4H), 7.48 – 7.40 (m, 6H), 7.36 – 7.27 (m, 5H), 3.67 (s, 2H), 2.87 (s, 1H), 2.49 (t, *J* = 7.2 Hz, 2H), 1.81 – 1.71 (m, 2H), 1.29 – 1.22 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.6, 136.0, 134.2, 130.0, 128.9, 128.5, 128.0, 127.0, 36.0, 34.6, 22.8, 14.5. **HRMS** (CI) m/z calcd for $C_{22}H_{24}SSi$ $(M)^+$: 364.1317 found 364.1323.



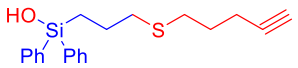
(3-((2-phenoxyethyl)thio)propyl)diphenylsilanol (5b)

Yield: 68% (107.5mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1420, 1358, 1220, 1092, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.71 – 7.64 (m, 4H), 7.51 – 7.41 (m, 6H), 7.38 – 7.32 (m, 2H), 7.04 (m, *J* = 7.3 Hz, 1H), 6.98 – 6.91 (m, 2H), 4.13 (t, *J* = 6.9 Hz, 2H), 3.24 (s, 1H), 2.87 (t, *J* = 6.9 Hz, 2H), 2.69 (t, *J* = 7.2 Hz, 2H), 1.88 – 1.79 (m, 2H), 1.35 – 1.27 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.4, 136.0, 134.2, 130.0, 129.5, 128.0, 121.0, 114.6, 67.7, 35.9, 30.8, 23.4, 14.5. **HRMS** (CI) m/z calcd for $C_{23}H_{26}O_2SSi$ $(M)^+$: 394.1423 found 394.1416.



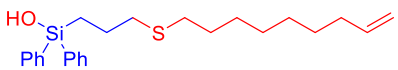
diphenyl(3-(propylthio)propyl)silanol (5c)

Yield: 24% (30.8mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1419, 1359, 1220, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67 – 7.61 (m, 4H), 7.47 – 7.38 (m, 6H), 2.97 (s, 1H), 2.57 (t, *J* = 7.1 Hz, 2H), 2.47 – 2.40 (m, 2H), 1.82 – 1.72 (m, 2H), 1.59 (h, *J* = 7.4 Hz, 2H), 1.31 – 1.26 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.1, 134.3, 130.0, 128.0, 35.4, 34.1, 23.3, 23.1, 14.6, 13.6. **^{29}Si NMR** (79 MHz, $CDCl_3$) δ -4.10. **HRMS** (CI) m/z calcd for $C_{18}H_{24}OSSi$ $(M)^+$: 316.1317 found 316.1306.



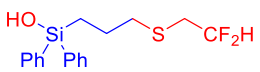
(3-(pent-4-yn-1-ylthio)propyl)diphenylsilanol (5d)

Yield: 42% (57.1mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1358, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 – 7.60 (m, 4H), 7.48 – 7.38 (m, 6H), 2.64 (s, 1H), 2.57 (td, *J* = 7.2, 1.8 Hz, 4H), 2.30 (td, *J* = 7.0, 2.6 Hz, 2H), 1.96 (t, *J* = 2.7 Hz, 1H), 1.77 (pd, *J* = 7.2, 3.3 Hz, 4H), 1.31 – 1.26 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.1, 134.3, 130.1, 128.1, 83.7, 69.0, 35.4, 30.7, 28.4, 23.3, 17.6, 14.6. **HRMS** (CI) m/z calcd for $C_{20}H_{24}OSSi$ $(M)^+$: 340.1317 found 340.1327.



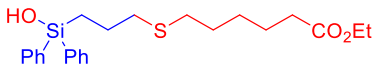
(3-(non-8-en-1-ylthio)propyl)diphenylsilanol (5e)

Yield: 49% (77.7mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1359, 1221, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.68 – 7.62 (m, 4H), 7.48 – 7.44 (m, 1H), 7.44 (m, *J* = 1.7 Hz, 1H), 7.41 (m, *J* = 7.8, 6.1 Hz, 4H), 5.86 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.08 – 4.96 (m, 2H), 2.95 (s, 1H), 2.57 (t, *J* = 7.1 Hz, 2H), 2.49 – 2.42 (m, 2H), 2.09 (tdd, *J* = 6.6, 5.3, 1.4 Hz, 2H), 1.83 – 1.74 (m, 2H), 1.60 – 1.52 (m, 2H), 1.47 – 1.40 (m, 2H), 1.40 – 1.32 (m, 6H), 1.31 – 1.28 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.2, 136.1, 134.3, 130.0, 128.0, 114.3, 35.5, 33.9, 32.0, 29.7, 29.2, 29.1, 29.0, 28.9, 23.2, 14.6. **HRMS** (CI) m/z calcd for $C_{24}H_{34}OSSi$ $(M)^+$: 398.2100 found 398.2093.



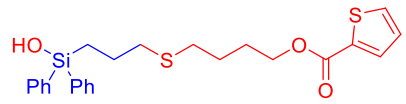
(3-((2,2-difluoroethyl)thio)propyl)diphenylsilanol (5f)

Yield: 32% (42.6mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1419, 1358, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 – 7.48 (m, 4H), 7.37 – 7.26 (m, 6H), 5.92 – 5.50 (m, 1H), 2.67 (td, *J* = 15.6, 4.6 Hz, 2H), 2.58 – 2.51 (m, 2H), 1.70 – 1.62 (m, 2H), 1.18 – 1.13 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.9, 134.3, 130.2, 128.1, 116.7 (t, *J* = 241.5 Hz), 36.1, 34.6 (t, *J* = 23.8 Hz), 23.2, 14.4. **HRMS** (CI) m/z calcd for $C_{17}H_{20}OSSiF_2$ $(M)^+$: 338.0972 found 338.0969.



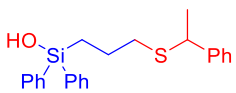
ethyl 6-((3-(hydroxydiphenylsilyl)propyl)thio)hexanoate (5g)

Yield: 42% (70.3mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3069, 2930, 1731, 1710, 1428, 1362, 1220, 1114, 864, 701, 509. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 – 7.60 (m, 4H), 7.43 – 7.36 (m, 6H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.10 – 2.71 (m, 1H), 2.55 (t, *J* = 7.2 Hz, 2H), 2.44 (t, *J* = 7.3 Hz, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.81 – 1.71 (m, 2H), 1.66 – 1.60 (m, 2H), 1.55 (q, *J* = 7.7 Hz, 2H), 1.43 – 1.36 (m, 2H), 1.29 – 1.25 (m, 5H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 136.3, 134.2, 129.9, 127.9, 60.4, 35.4, 34.2, 31.7, 29.3, 28.3, 24.6, 23.3, 14.6, 14.3. **HRMS** (CI) m/z calcd for $C_{23}H_{32}O_3SSi$ $(M)^+$: 416.1841 found 416.1837.



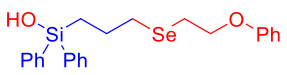
1-4-((3(hydroxydiphenylsilyl)propyl)thio)butyl thiophene-2-carboxylate (5h)

Yield: 42% (84.7mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 2924, 1706, 1419, 1359, 1221, 1096, 861, 703, 510. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76 (m, *J* = 3.7, 1.3 Hz, 1H), 7.66 – 7.54 (m, 4H), 7.50 (m, *J* = 5.0, 1.3 Hz, 1H), 7.43 – 7.29 (m, 6H), 7.05 (m, *J* = 5.0, 3.8 Hz, 1H), 4.24 (t, *J* = 6.4 Hz, 2H), 3.22 (s, 1H), 2.52 (t, *J* = 7.2 Hz, 2H), 2.46 (t, *J* = 7.3 Hz, 2H), 1.84 – 1.68 (m, 4H), 1.68 – 1.59 (m, 2H), 1.25 – 1.19 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.5, 136.2, 134.2, 133.8, 133.6, 132.5, 129.9, 128.0, 127.8, 64.8, 35.3, 31.4, 27.9, 26.0, 23.2, 14.6. **HRMS** (CI) m/z calcd for $C_{24}H_{28}O_3S_2Si$ $(M)^+$: 456.1249 found 456.1239.

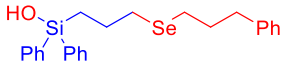


diphenyl(3-((1-phenylethyl)thio)propyl)silanol (5j)

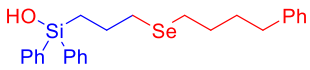
Yield: 89% (111.2mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1709, 1359, 1220, 1115, 871, 702, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 – 7.52 (m, 3H), 7.39 – 7.35 (m, 2H), 7.35 – 7.29 (m, 4H), 7.25 – 7.21 (m, 3H), 7.18 (ddd, *J* = 9.2, 4.4, 2.4 Hz, 1H), 3.81 (q, *J* = 7.0 Hz, 1H), 2.77 (s, 1H), 2.37 – 2.21 (m, 2H), 1.69 – 1.53 (m, 2H), 1.49 (d, *J* = 7.1 Hz, 3H), 1.18 – 1.04 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.1, 136.1, 134.2, 130.0, 128.5, 128.0, 127.3, 127.0, 43.9, 34.6, 23.0, 22.7, 14.6. **HRMS** (CI) m/z calcd for $C_{23}H_{36}OSSi$ $(M)^+$: 378.1474 found 378.1475.



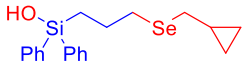
(3-((2-phenoxyethyl)selanyl)propyl)diphenylsilanol (5m)
Yield: 64% (113.0mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1428, 1359, 1220, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 (m, *J* = 8.0, 1.6 Hz, 4H), 7.39 – 7.29 (m, 6H), 7.26 – 7.21 (m, 2H), 6.94 – 6.89 (m, 1H), 6.86 – 6.80 (m, 2H), 4.09 (t, *J* = 7.2 Hz, 2H), 2.77 (t, *J* = 7.2 Hz, 2H), 2.64 (t, *J* = 7.3 Hz, 2H), 1.86 – 1.74 (m, 2H), 1.25 – 1.19 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.4, 136.1, 134.2, 130.0, 129.6, 128.0, 121.0, 114.7, 68.4, 28.4, 24.4, 22.0, 15.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 137.6. **HRMS** (CI) m/z calcd for $C_{23}H_{26}O_2SeSi$ $(M)^+$: 442.0867 found 442.0873.



diphenyl(3-((3-phenylpropyl)selanyl)propyl)silanol (5n)
Yield: 65% (114.6mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1709, 1428, 1359, 1220, 703, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 – 7.46 (m, 4H), 7.36 – 7.25 (m, 6H), 7.18 (m, *J* = 8.0, 6.6 Hz, 2H), 7.13 – 7.04 (m, 3H), 2.61 – 2.56 (m, 2H), 2.54 – 2.39 (m, 4H), 1.84 (p, *J* = 7.5 Hz, 2H), 1.75 – 1.66 (m, 2H), 1.56 (s, 1H), 1.19 – 1.14 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 141.6, 136.1, 134.3, 130.1, 128.6, 128.5, 128.1, 126.0, 36.0, 32.2, 27.7, 24.2, 23.3, 15.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 151.6. **HRMS** (CI) m/z calcd for $C_{24}H_{28}OSeSi$ $(M)^+$: 440.1075 found 440.1074.

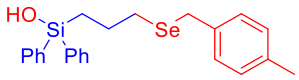


diphenyl(3-((4-phenylbutyl)selanyl)propyl)silanol (5o)
Yield: 66% (120.4mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1709, 1420, 1358, 1220, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 – 7.49 (m, 4H), 7.35 – 7.26 (m, 6H), 7.20 – 7.16 (m, 2H), 7.12 – 7.04 (m, 3H), 2.54 – 2.36 (m, 6H), 1.71 (ddt, *J* = 12.1, 8.9, 6.1 Hz, 2H), 1.62 – 1.52 (m, 4H), 1.17 (dd, *J* = 8.3, 2.9 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 142.4, 136.1, 134.3, 130.1, 128.5, 128.4, 128.1, 125.9, 35.5, 31.7, 30.3, 27.7, 24.2, 23.7, 15.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 153.3. **HRMS** (CI) m/z calcd for $C_{26}H_{32}OSeSi$ $(M)^+$: 454.1231 found 454.1232.



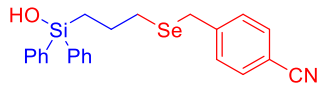
(3-((cyclopropylmethyl)selanyl)propyl)diphenylsilanol (5p)
Yield: 67% (101.0mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1428, 1358, 1220, 1092, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 – 7.42 (m, 4H), 7.29 – 7.19 (m, 6H), 2.51 (d, *J* = 7.2 Hz, 2H), 2.27 (d, *J* = 7.1 Hz, 2H), 1.71 – 1.63 (m, 2H), 1.15 – 1.09 (m, 2H), 0.88 – 0.76 (m, 1H), 0.45 – 0.36 (m, 2H), -0.01 (dt, *J* = 6.1, 4.5 Hz, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.2, 134.3, 130.0, 128.1, 29.4, 27.6, 24.4, 15.9, 12.2, 6.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 168.0. **HRMS** (CI) m/z calcd for $C_{19}H_{24}OSeSi$ $(M)^+$: 376.0762 found

376.0768.



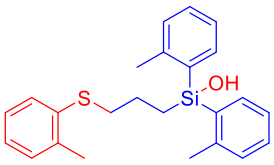
(3-((4-methylbenzyl)selanyl)propyl)diphenylsilanol (5q)

Yield: 72% (121.9mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1421, 1358, 1220, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 4H), 7.36 – 7.24 (m, 6H), 7.03 – 6.94 (m, 4H), 3.57 (s, 2H), 2.44 (d, *J* = 7.2 Hz, 2H), 2.38 (s, 1H), 2.22 (s, 3H), 1.73 – 1.62 (m, 2H), 1.15 – 1.08 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.5, 136.3, 136.1, 134.3, 130.1, 129.3, 128.8, 128.1, 27.7, 26.7, 23.9, 21.2, 15.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 245.8. **^{29}Si NMR** (79 MHz, $CDCl_3$) δ -4.31. **HRMS** (CI) m/z calcd for $C_{23}H_{26}OSeSi$ $(M)^+$: 426.0918 found 426.0921.



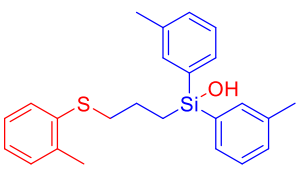
4-(((3-(hydroxydiphenylsilyl)propyl)selanyl)methyl)benzonitrile (5r)

Yield: 45% (79.5mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 2228, 1710, 1427, 1358, 1220, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 – 7.47 (m, 4H), 7.44 – 7.40 (m, 2H), 7.38 – 7.28 (m, 6H), 7.21 – 7.17 (m, 2H), 3.60 (s, 2H), 2.44 (t, *J* = 7.3 Hz, 2H), 1.98 (s, 1H), 1.74 – 1.62 (m, 2H), 1.15 – 1.07 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.6, 136.0, 134.2, 132.4, 130.2, 129.6, 128.1, 119.0, 110.4, 28.0, 26.3, 23.9, 15.8. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 263.3. **HRMS** (CI) m/z calcd for $C_{23}H_{23}ONSeSi$ $(M)^+$: 437.0714 found 437.0706.



di-*o*-tolyl(3-(*o*-tolylthio)propyl)silanol (6a)

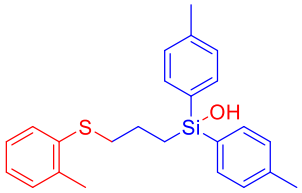
Yield: 57% (90.2mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3005, 1710, 1420, 1359, 1220, 1112, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.47 (m, *J* = 7.4, 1.5 Hz, 2H), 7.18 (m, *J* = 7.5, 1.5 Hz, 2H), 7.06 (m, *J* = 7.8, 1.3 Hz, 3H), 7.03 – 6.97 (m, 4H), 6.97 – 6.93 (m, 1H), 2.82 (t, *J* = 7.1 Hz, 2H), 2.23 (s, 3H), 2.14 (s, 6H), 1.71 – 1.62 (m, 2H), 1.31 – 1.24 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 143.7, 137.4, 136.0, 135.2, 135.1, 130.1, 130.1, 130.1, 127.7, 126.4, 125.5, 125.2, 36.1, 23.0, 22.9, 20.4, 15.3. **HRMS** (CI) m/z calcd for $C_{24}H_{28}OSSi$ $(M)^+$: 392.1630 found 392.1638.



di-*m*-tolyl(3-(*o*-tolylthio)propyl)silanol (6b)

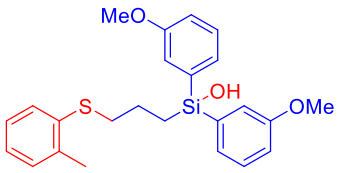
Yield: 64% (100.8mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1359, 1220, 705, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.49 (s, 2H), 7.45 (m, *J* = 6.8 Hz, 2H), 7.36 – 7.28 (m, 4H), 7.25 (m, *J* = 7.6, 1.6 Hz, 1H), 7.20 (m, *J* = 11.9, 7.2, 1.9 Hz, 2H), 7.16 – 7.11 (m, 1H), 3.00 (t, *J* = 7.2 Hz, 2H), 2.42 (d, *J* = 4.8 Hz, 10H), 1.97 – 1.85 (m, 2H), 1.41 – 1.35 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$)

δ 137.4, 137.3, 136.1, 135.9, 134.8, 131.3, 130.8, 130.1, 128.0, 127.6, 126.4, 125.4, 36.1, 22.9, 21.6, 20.4, 14.8. **HRMS** (CI) m/z calcd for $C_{24}H_{28}OSSi$ $(M)^+$: 392.1630 found 392.1638.



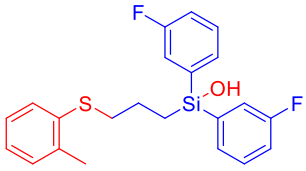
di-*p*-tolyl(3-(*o*-tolylthio)propyl)silanol (6c)

Yield: 71% (111.0mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3005, 1710, 1359, 1220, 1092, 803, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (m, *J* = 7.9 Hz, 4H), 7.07 – 7.00 (m, 6H), 6.95 (m, *J* = 8.8, 7.1, 1.8 Hz, 2H), 2.77 (t, *J* = 7.2 Hz, 2H), 2.50 (s, 1H), 2.23 (s, 6H), 2.22 (s, 3H), 1.68 (ddt, *J* = 11.9, 8.7, 6.2 Hz, 2H), 1.17 – 1.10 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.9, 137.3, 136.2, 134.3, 132.6, 130.1, 128.8, 127.6, 126.4, 125.4, 36.1, 22.9, 21.6, 20.4, 14.9. **^{29}Si NMR** (79 MHz, $CDCl_3$) δ -4.02. **HRMS** (CI) m/z calcd for $C_{24}H_{28}OSSi$ $(M)^+$: 392.1630 found 392.1636.



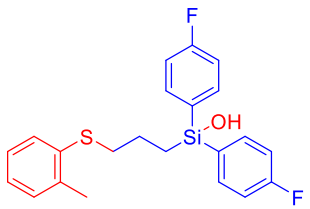
bis(3-methoxyphenyl)(3-(*o*-tolylthio)propyl)silanol (6d)

Yield: 53% (89.3mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1418, 1359, 1220, 1046, 701, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.20 – 7.15 (m, 2H), 7.03 (m, *J* = 7.1, 5.8, 1.7 Hz, 6H), 6.99 – 6.91 (m, 2H), 6.82 (m, *J* = 8.2, 2.7, 1.1 Hz, 2H), 3.64 (s, 6H), 2.79 (t, *J* = 7.2 Hz, 2H), 2.22 (s, 3H), 1.76 – 1.65 (m, 2H), 1.20 – 1.14 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 159.1, 137.5, 137.3, 136.0, 130.1, 129.3, 127.6, 126.5, 126.4, 125.4, 119.4, 115.6, 55.2, 36.0, 22.8, 20.4, 14.7. **HRMS** (CI) m/z calcd for $C_{24}H_{28}O_3SSi$ $(M)^+$: 424.1528 found 424.1535.



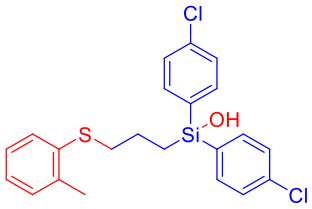
bis(3-fluorophenyl)(3-(*o*-tolylthio)propyl)silanol (6e)

Yield: 60% (96.3mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1416, 1358, 1219, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.26 – 7.16 (m, 4H), 7.12 (m, *J* = 8.8, 2.8 Hz, 2H), 7.09 – 6.92 (m, 6H), 2.80 (t, *J* = 7.1 Hz, 2H), 2.59 (s, 1H), 2.23 (s, 3H), 1.75 – 1.59 (m, 2H), 1.23 – 1.13 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.7 (*J* = 247.1 Hz), 138.4 (*J* = 4.3 Hz), 137.6, 135.6, 130.2, 130.1, 130.1, 129.7 (*J* = 3.0 Hz), 127.9, 126.1 (*J* = 73.4 Hz), 120. 5 (*J* = 18.8 Hz), 117.3 (*J* = 20.9 Hz), 36.0, 22.6, 20.4, 14.4. **HRMS** (CI) m/z calcd for $C_{22}H_{22}OF_2SSi$ $(M)^+$: 400.1129 found 400.1135.



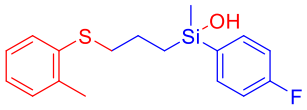
bis(4-fluorophenyl)(3-(*o*-tolylthio)propyl)silanol (6f)

Yield: 64% (103.1mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1420, 1358, 1220, 1092, 824, 528. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.34 (m, 4H), 7.07 – 7.01 (m, 2H), 7.01 – 6.88 (m, 6H), 2.79 (t, *J* = 7.1 Hz, 2H), 2.54 (s, 1H), 2.22 (s, 3H), 1.65 (ddt, *J* = 11.2, 8.6, 6.2 Hz, 2H), 1.19 – 1.12 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.4 (*J* = 248.4 Hz), 137.5, 136.3, 136.3, 135.7, 131.3 (*J* = 3.7 Hz), 130.2, 127.8, 126.1 (*J* = 76.5 Hz), 115.4 (*J* = 19.6 Hz), 36.0, 22.7, 20.4, 14.7. **HRMS** (CI) m/z calcd for $C_{22}H_{22}OF_2SSi$ $(M)^+$: 400.1129 found 400.1139.



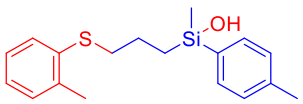
bis(4-chlorophenyl)(3-(*o*-tolylthio)propyl)silanol (6g)

Yield: 69% (118.6mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1420, 1358, 1220, 1084, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 (m, *J* = 7.8 Hz, 4H), 7.25 (m, *J* = 7.8 Hz, 4H), 7.04 (m, *J* = 25.4, 5.8, 2.4 Hz, 4H), 2.83 (t, *J* = 7.0 Hz, 2H), 2.49 (s, 1H), 2.25 (s, 3H), 1.73 – 1.65 (m, 2H), 1.23 – 1.16 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.6, 136.7, 135.7, 135.6, 133.8, 130.3, 128.5, 128.0, 126.5, 125.8, 36.0, 22.6, 20.5, 14.5. **HRMS** (CI) m/z calcd for $C_{22}H_{22}OCl_2SSi$ $(M)^+$: 432.0538 found 432.0544.



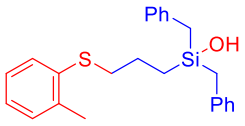
(4-fluorophenyl)(methyl)(3-(*o*-tolylthio)propyl)silanol (6h)

Yield: 32% (40.9mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1358, 1220, 826, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (m, *J* = 8.6, 6.2, 2.4 Hz, 2H), 7.08 (m, *J* = 7.6, 1.5 Hz, 1H), 7.05 – 6.90 (m, 5H), 2.78 (t, *J* = 7.2 Hz, 2H), 2.23 (s, 3H), 1.91 (s, 1H), 1.66 – 1.57 (m, 2H), 0.88 (d, *J* = 16.7 Hz, 2H), 0.25 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 164.2 (*J* = 247.6 Hz), 137.5, 136.0, 135.4(2), 135.3(5), 133.6 (*J* = 3.7 Hz), 130.2, 127.8, 126.0 (*J* = 83.2 Hz), 115.2 (*J* = 19.5 Hz), 115.1, 36.2, 22.9, 20.5, 16.3, -1.4. **HRMS** (CI) m/z calcd for $C_{17}H_{21}OFSSi$ $(M)^+$: 320.1066 found 320.1073.



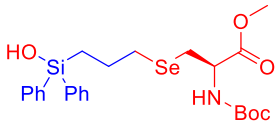
methyl(*p*-tolyl)(3-(*o*-tolylthio)propyl)silanol (6i)

Yield: 24% (30.0mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3011, 1717, 1456, 1253, 1110.9, 848, 742, 555. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.33 (m, 2H), 7.14 – 7.09 (m, 3H), 7.02 (m, *J* = 20.9, 7.1, 1.6 Hz, 3H), 2.82 (t, *J* = 7.3 Hz, 2H), 2.27 (d, *J* = 3.6 Hz, 6H), 1.84 (s, 1H), 1.73 – 1.64 (m, 2H), 0.97 – 0.89 (m, 2H), 0.28 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.8, 137. 5, 136.2, 134.4, 133.4, 130.2, 128.9, 127.7, 126.4, 125.5, 36.2, 23.0, 21.6, 20.5, 16.4, -1.5. **HRMS** (CI) m/z calcd for $C_{18}H_{24}OSSi$ $(M)^+$: 316.1317 found 316.1321.



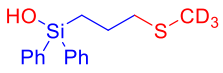
dibenzyl(3-(*o*-tolylthio)propyl)silanol (6j)

Yield: 56% (88.4mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3003, 1709, 1359, 1220, 1092, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 (m, *J* = 7.6 Hz, 5H), 7.25 – 7.14 (m, 5H), 7.11 – 7.05 (m, 4H), 2.90 (t, *J* = 7.3 Hz, 2H), 2.43 (s, 3H), 2.24 (s, 4H), 1.90 (s, 1H), 1.77 – 1.67 (m, 2H), 0.85 – 0.69 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.2, 137.4, 136.0, 130.2, 128.6, 128.4, 127.6, 126.4, 125.5, 124.7, 36.0, 24.6, 22.7, 20.5, 13.7. **HRMS** (CI) m/z calcd for $C_{24}H_{28}OSSi$ $(M)^+$: 392.1630 found 392.1628.



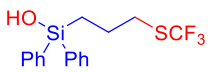
methyl (*R*)-1-hydroxy-11,11-dimethyl-9-oxo-1,1-diphenyl-10-oxa-5-selena-8-aza-1-siladodecane-7-carboxylate (5s)

Yield: 76% (154.1mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3000, 2929, 1743, 1706, 1499, 1364, 1219, 1161, 1114, 1048, 858, 700, 507. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 – 7.46 (m, 4H), 7.30 – 7.22 (m, 6H), 5.31 (d, *J* = 8.2 Hz, 1H), 4.43 (q, *J* = 6.0 Hz, 1H), 3.57 (s, 3H), 2.81 – 2.67 (m, 2H), 2.50 (td, *J* = 7.2, 2.9 Hz, 2H), 1.72 – 1.63 (m, 2H), 1.32 (s, 9H), 1.15 – 1.08 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.8, 155.3, 136.4, 134.2, 129.8, 127.9, 80.4, 53.6, 52.6, 28.6, 28.4, 25.7, 24.0, 15.6. **^{77}Se NMR** (76 MHz, $CDCl_3$) δ 121.6. **HRMS** (CI) m/z calcd for $C_{24}H_{33}O_5NSeSi$ $(M)^+$: 523.1293 found 523.1298.



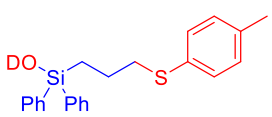
(3-((methyl-*d*$_3$)thio)propyl)diphenylsilanol (5t)

Yield: 61% (71.3mg). (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3001, 2922, 2127, 1705, 1428, 1360, 1220, 1114, 862, 701, 509. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.65 – 7.55 (m, 4H), 7.41 – 7.32 (m, 6H), 3.04 (s, 1H), 2.48 (t, *J* = 7.1 Hz, 2H), 1.77 – 1.66 (m, 2H), 1.25 – 1.18 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.1, 134.2, 130.0, 128.0, 37.4, 22.7, 14.4. **HRMS** (CI) m/z calcd for $C_{16}H_{17}OD_3SSi$ $(M)^+$: 291.1192; found 291.1183.



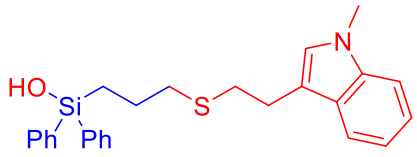
diphenyl(3-((trifluoromethyl)thio)propyl)silanol (5u)

Yield: 65% (44.5mg). (EA : PE = 1 : 20). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1710, 1428, 1359, 1220, 1118, 873, 704, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48 (m, *J* = 6.5, 1.6 Hz, 4H), 7.36 – 7.26 (m, 6H), 2.79 (t, *J* = 7.2 Hz, 2H), 2.53 (s, 1H), 1.79 – 1.68 (m, 2H), 1.16 – 1.09 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 135.6, 134.3, 131.2 (q, *J* = 284.5 Hz), 130.3, 128.2, 33.1, 23.6, 14.5. **^{19}F NMR** (377 MHz, $CDCl_3$) δ -40.9. **HRMS** (CI) m/z calcd for $C_{16}H_{17}F_3OSSi$ $(M)^+$: 342.0721; found 342.0714.



diphenyl(3-(*p*-tolylthio)propyl)silanol-d (3c')

Yield: 43% (63.2mg). (EA : PE = 1 : 20). D-inc. 100% (determined by **^{1}H NMR**). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3004, 1709, 1358, 1220, 529. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.49 – 7.43 (m, 4H), 7.34 – 7.28 (m, 2H), 7.27 – 7.22 (m, 4H), 7.11 – 7.06 (m, 2H), 6.95 (m, *J* = 7.9 Hz, 2H), 2.79 (t, *J* = 7.1 Hz, 2H), 2.20 (s, 3H), 1.67 (ddt, *J* = 11.3, 8.7, 6.3 Hz, 2H), 1.20 – 1.15 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.1, 136.0, 134.3, 132.7, 130.2, 130.0, 129.7, 128.0, 37.8, 22.9, 21.1, 14.5. **HRMS** (CI) m/z calcd for $C_{22}H_{23}ODSSi$ $(M)^+$: 365.1380; found 365.1379.



(3-((2-(1-methyl-1H-indol-3-yl)ethyl)thio)propyl)diphenylsilanol (5s)

Yield: 48% . (EA : PE = 1 : 10). Pale yellow oil. **IR** (neat, ν, cm^{-1}): 3002, 1710, 1359, 1220, 742, 704. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51 – 7.46 (m, 4H), 7.45 – 7.41 (m, 1H), 7.28 – 7.21 (m, 6H), 7.16 (d, *J* = 8.1 Hz, 1H), 7.10 (m, *J* = 8.1, 1.1 Hz, 1H), 6.99 (m, *J* = 8.0, 6.8, 1.2 Hz, 1H), 6.71 (s, 1H), 3.57 (s, 3H), 2.91 – 2.84 (m, 2H), 2.76 (d, *J* = 13.3 Hz, 1H), 2.66 (dd, *J* = 8.8, 6.8 Hz, 2H), 2.48 (t, *J* = 7.1 Hz, 2H), 1.70 – 1.60 (m, 2H), 1.21 – 1.16 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.1, 136.3, 134.3, 130.0, 128.0, 127.7, 126.6, 121.6, 118.8(9), 118.8(5), 113.6, 109.3, 35.6, 32.8, 32.7, 26.0, 23.4, 14.7. **HRMS** (CI) m/z calcd for $C_{26}H_{29}NOSSi$ $(M)^+$: 431.1739; found 431.1736.

VI. References

(1) (a) Barday, M.; Janot, C.; Halcovitch, N. R.; Muir, J.; Aïssa, C. *Angew. Chem. Int. Ed.*, **2017**, *56*, 13117. (b) Lu, L. Q.; Cao, Y. J.; Liu, X. P.; An, J.; Yao, C. J.; Ming, Z. H.; Xiao, W. J. *J. Am. Chem. Soc.* **2008**, *130*, 6946. (c) Ratts, K. W.; Yao, A. N. *J. Org. Chem.* **1966**, *31*, 1185.

(2) Bogonda, G.; Patil, D. V.; Kim, H. Y.; Oh, K. *Org. Lett*. **2019**, *21*, 3774.

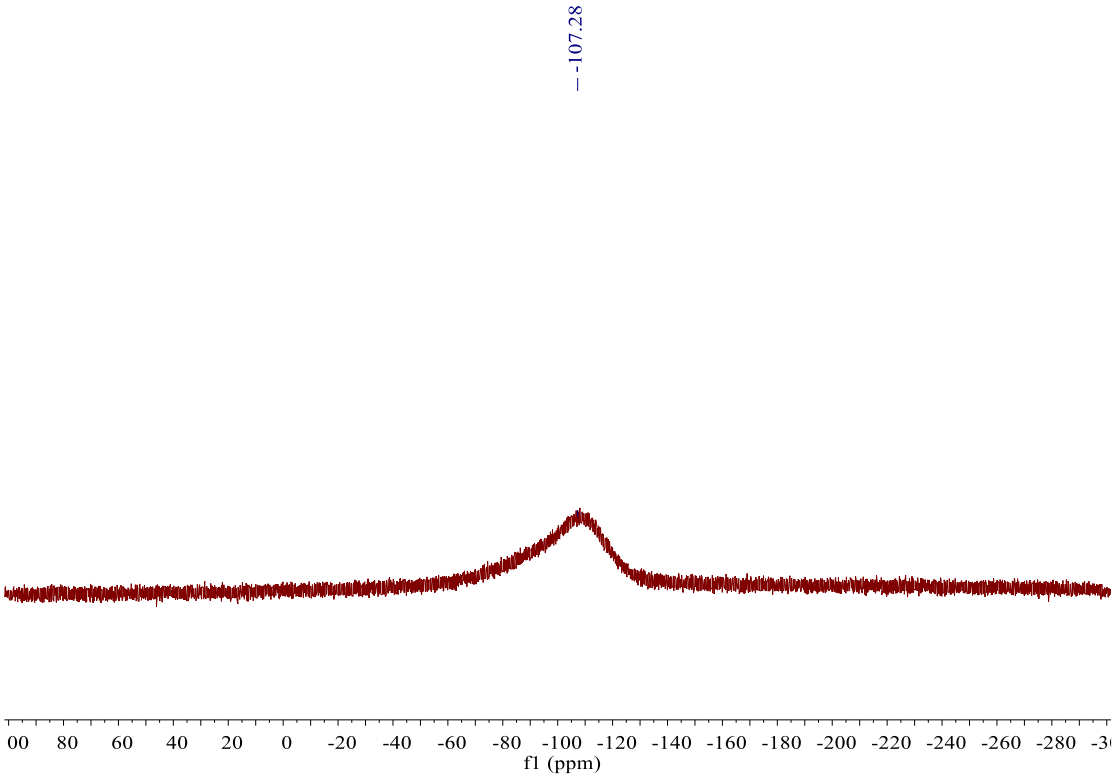
(3) Fang, Y.; Rogge, T.; Ackermann, L.; Wang, S. -Y.; Ji, S.-J. *Nat Commun*. **2018**, *9*, 2240.

(4) Cai, L.-B; Zhu, X.-Y; Chen, J.-Y.; Lin, A.-J.; Yao, H.-Q. *Org. Chem. Front*. **2019**, **6**, 3688.

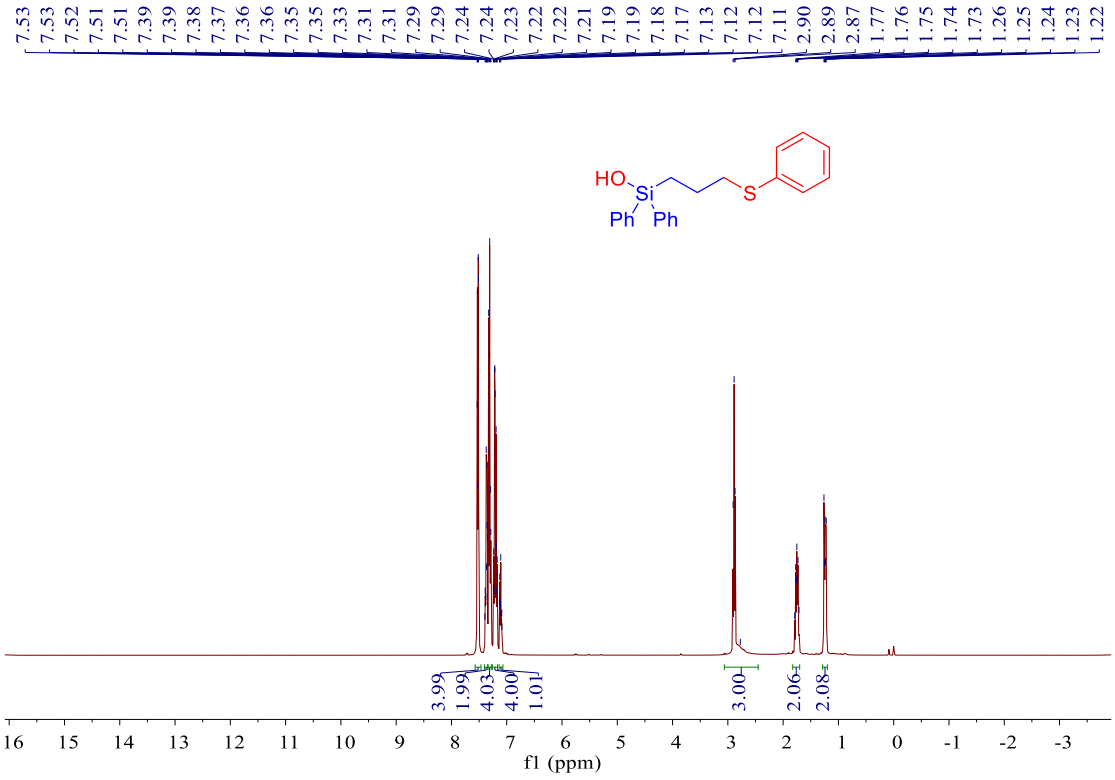
(5) (a) Jiang, M.; Li, H.-F.; Yang, H.-J.; Fu, H. *Angew. Chem.* **2017**, *129*, 892 –897.

VII. Copies of ^{1}H NMR ^{13}C NMR and ^{29}Si NMR Spectra

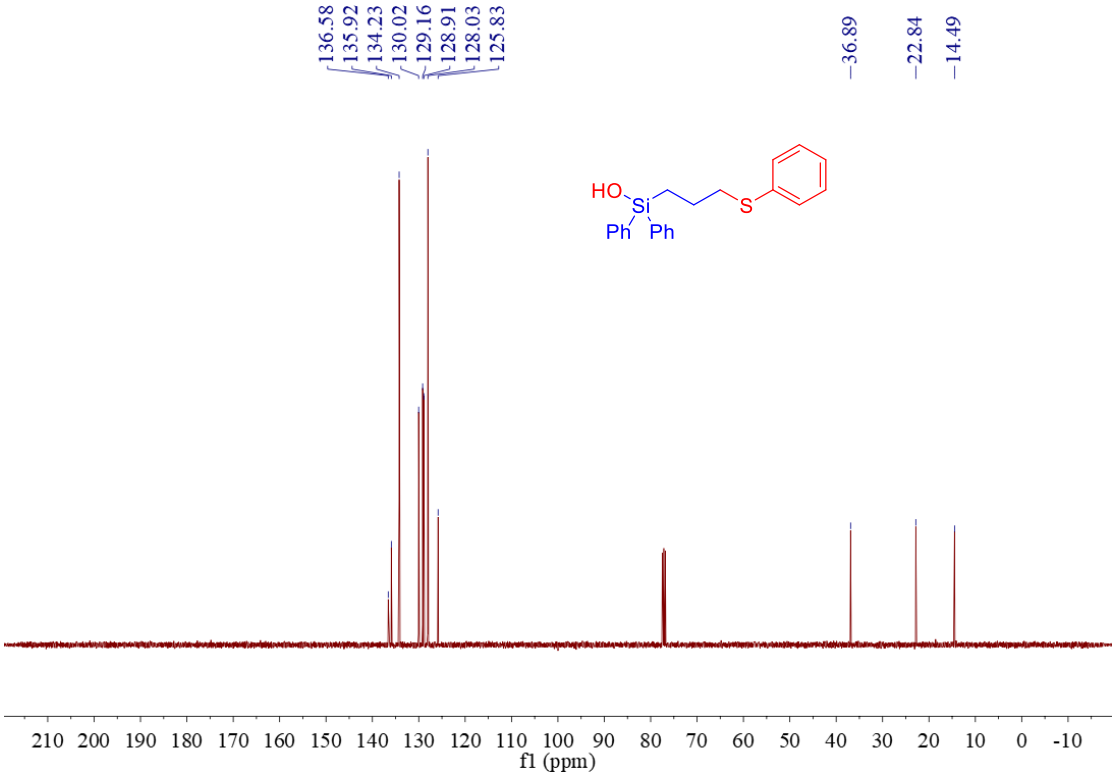
^{29}Si NMR Spectra of Nuclear magnetic tube (79 MHz, $CDCl_3$)



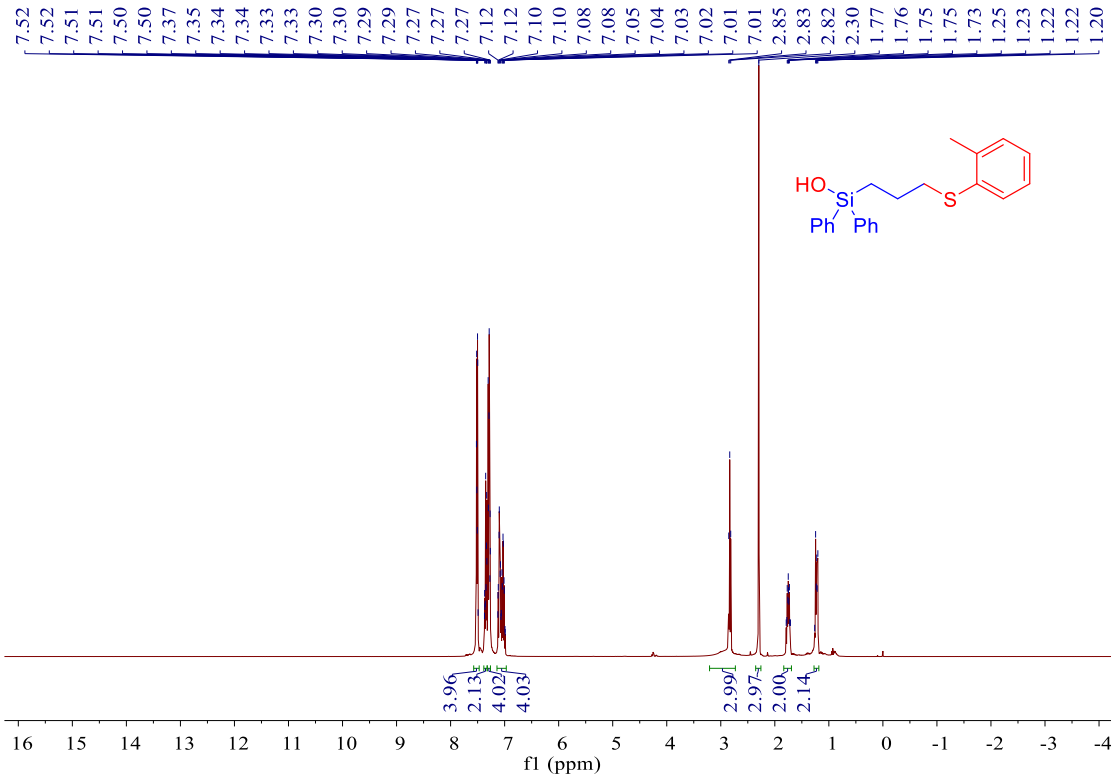
^{1}H NMR Spectra of **3a** (400 MHz, $CDCl_3$)



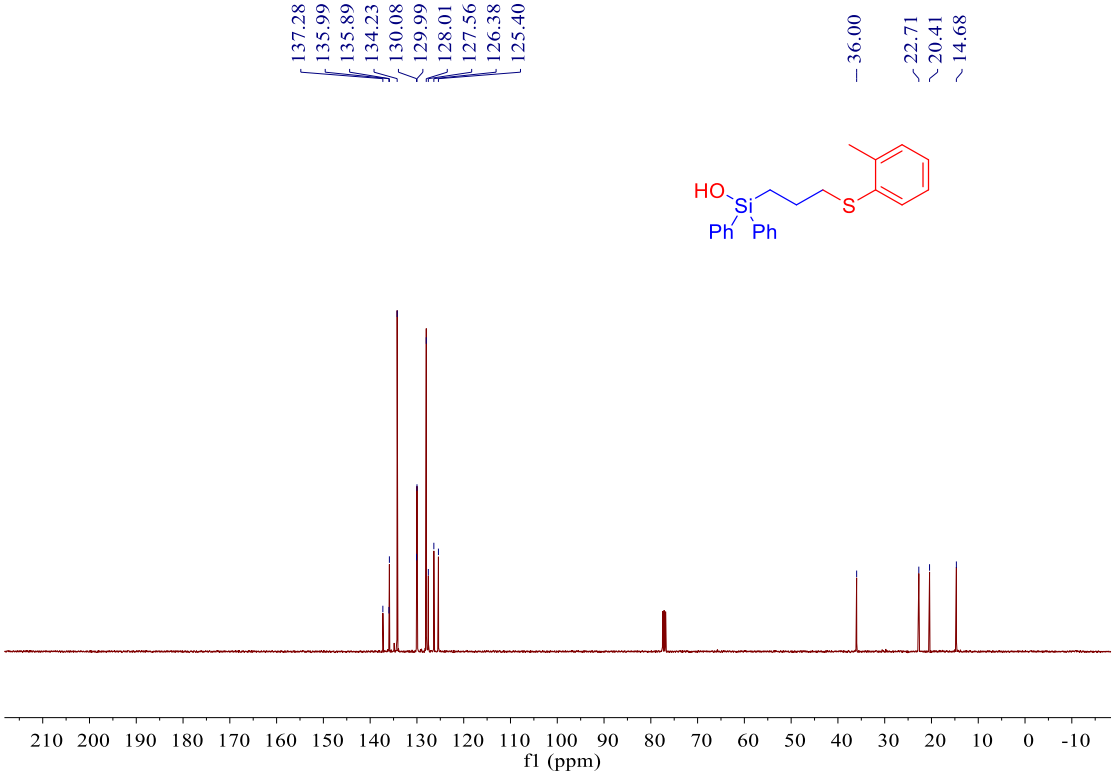
^{13}C NMR Spectra of **3a** (100 MHz, $CDCl_3$)



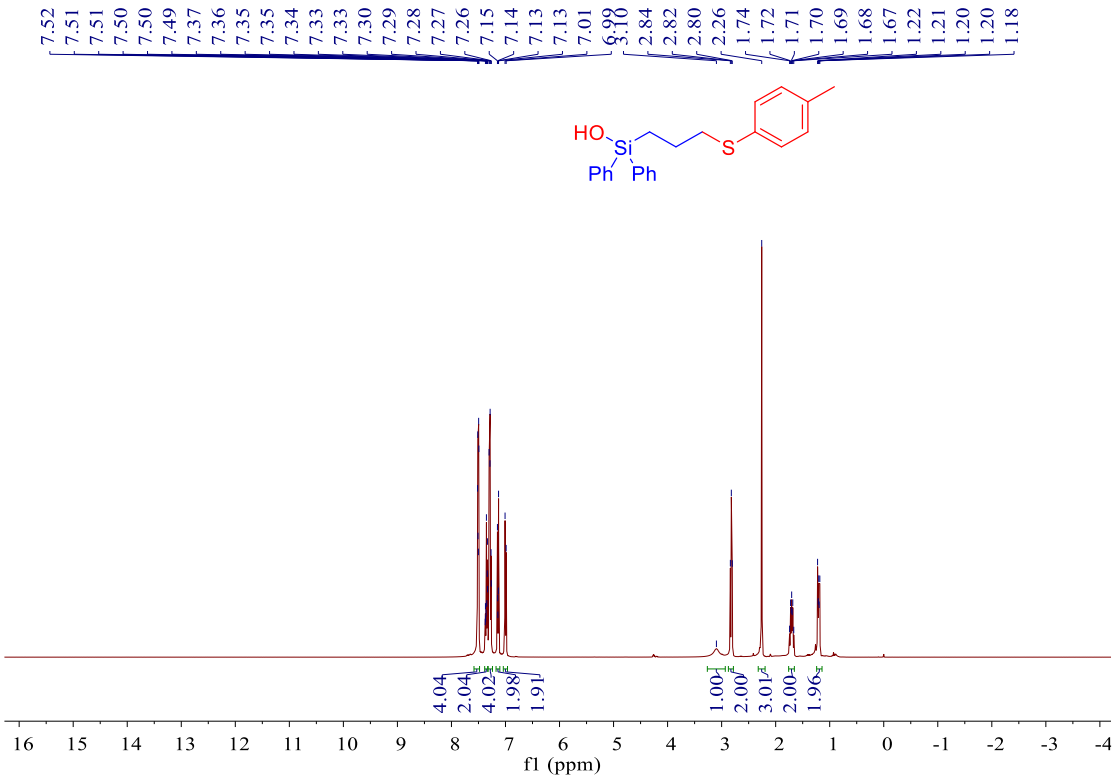
^{1}H NMR Spectra of **3b** (400 MHz, $CDCl_3$)



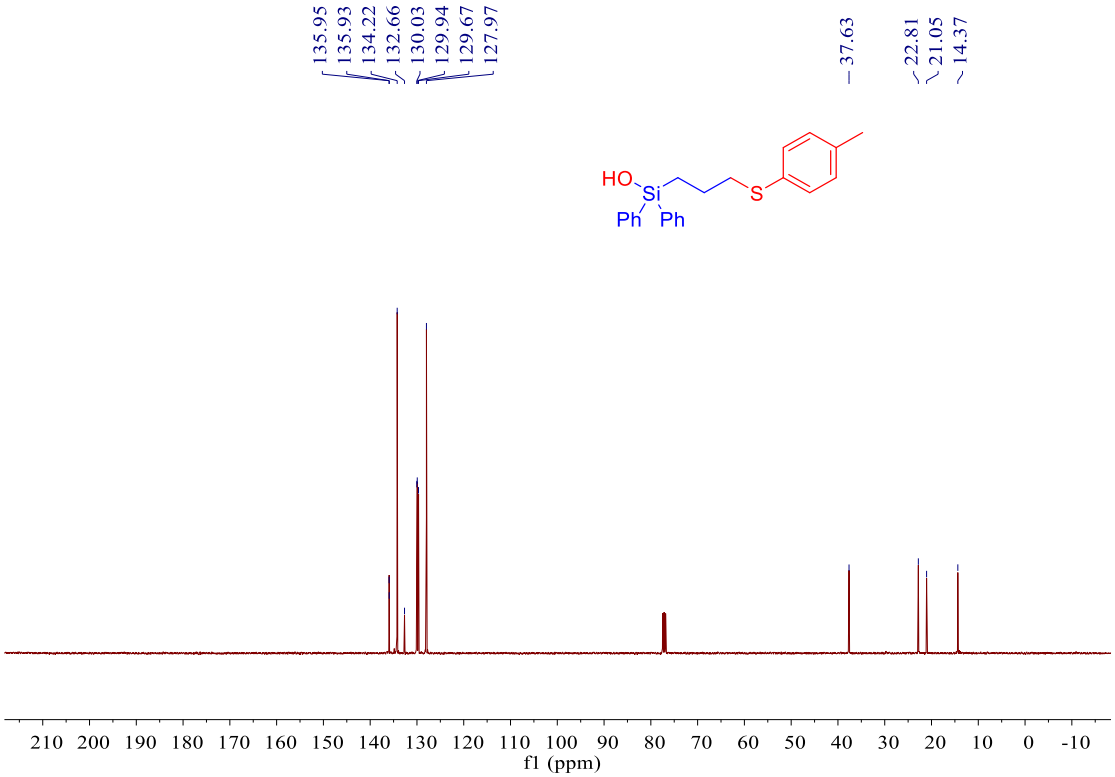
^{13}C NMR Spectra of **3b** (100 MHz, $CDCl_3$)



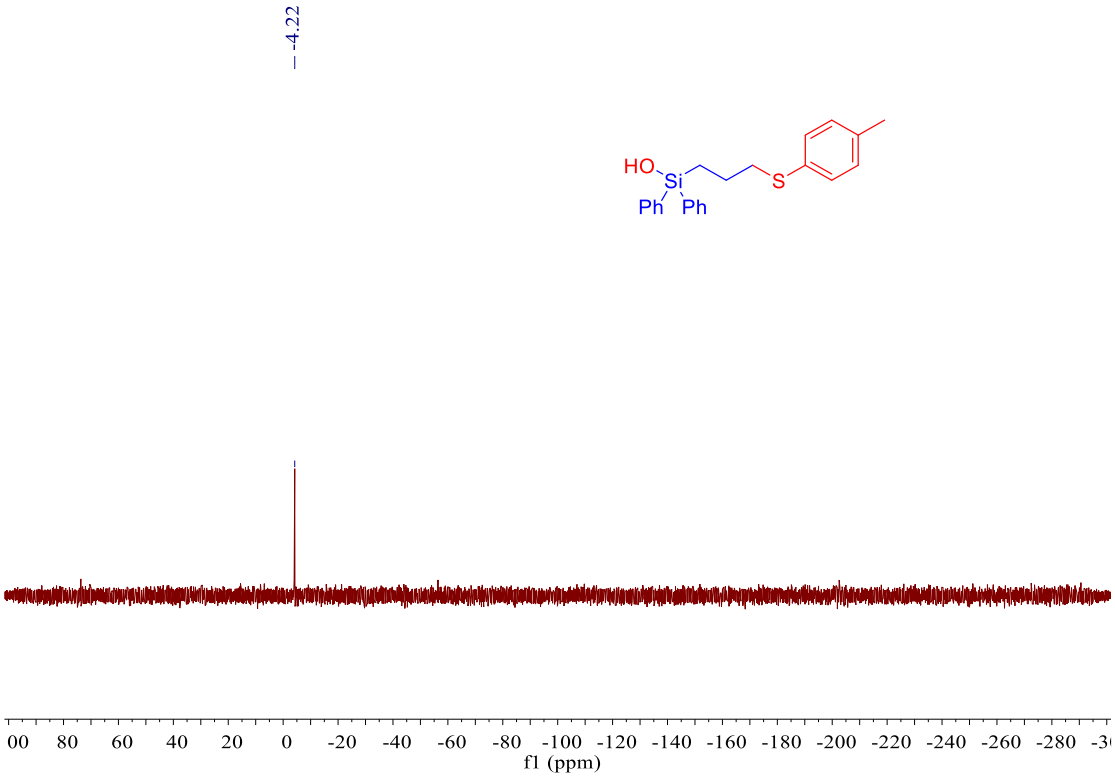
^{1}H NMR Spectra of **3c** (400 MHz, $CDCl_3$)



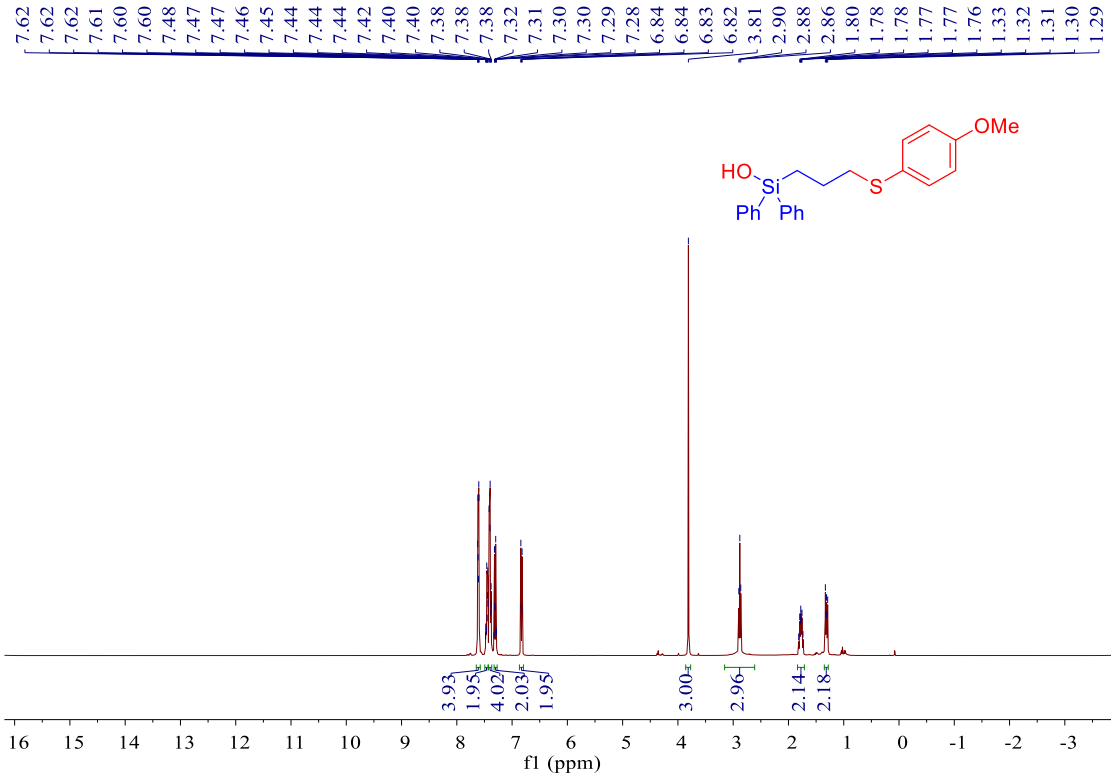
^{13}C NMR Spectra of **3c** (100 MHz, $CDCl_3$)



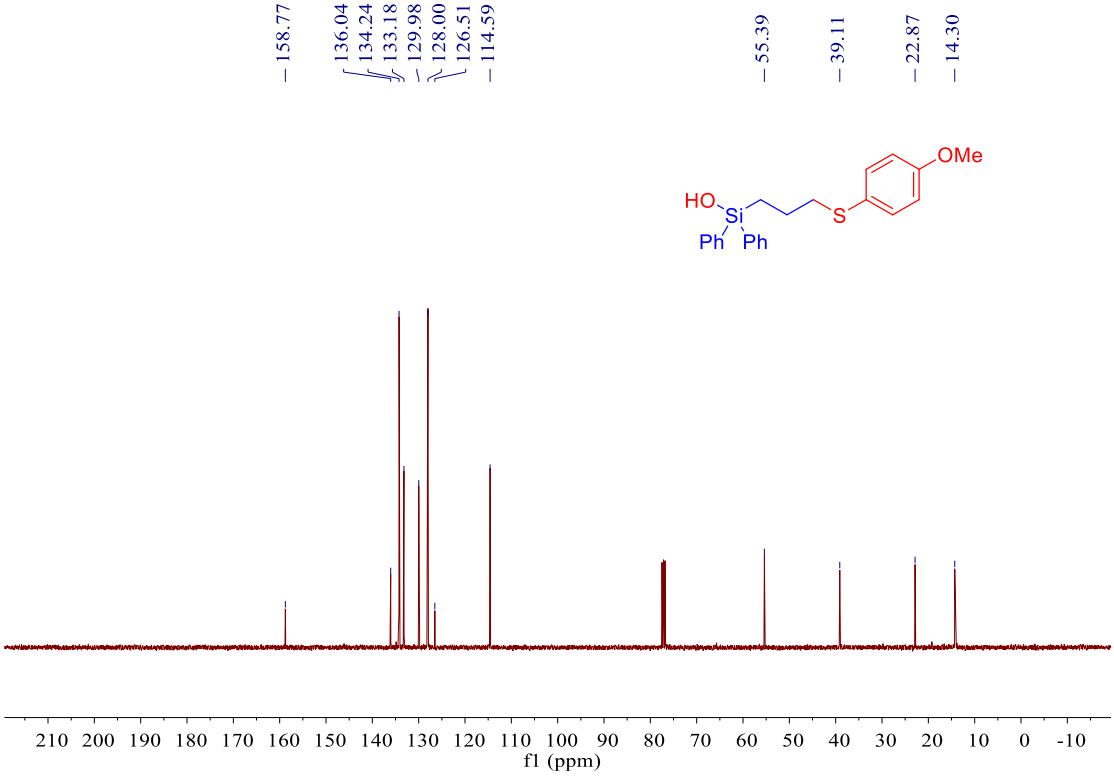
^{29}Si NMR Spectra of **3c** (79 MHz, $CDCl_3$)



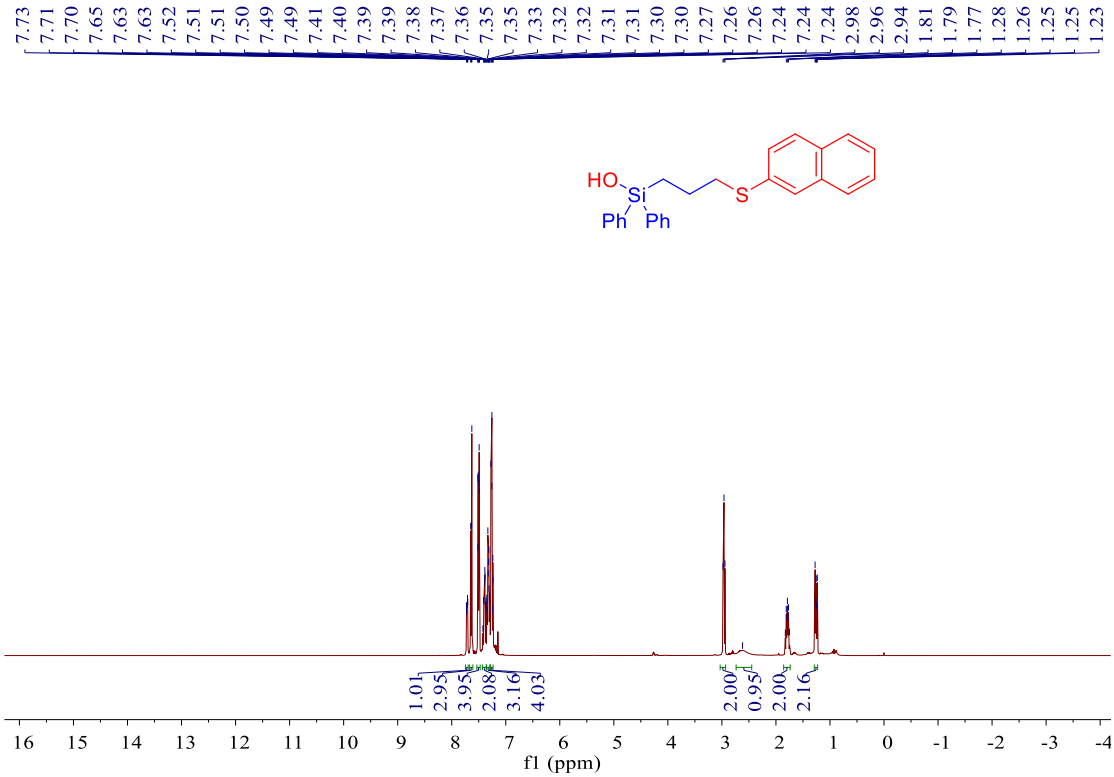
^{1}H NMR Spectra of **3d** (400 MHz, $CDCl_3$)



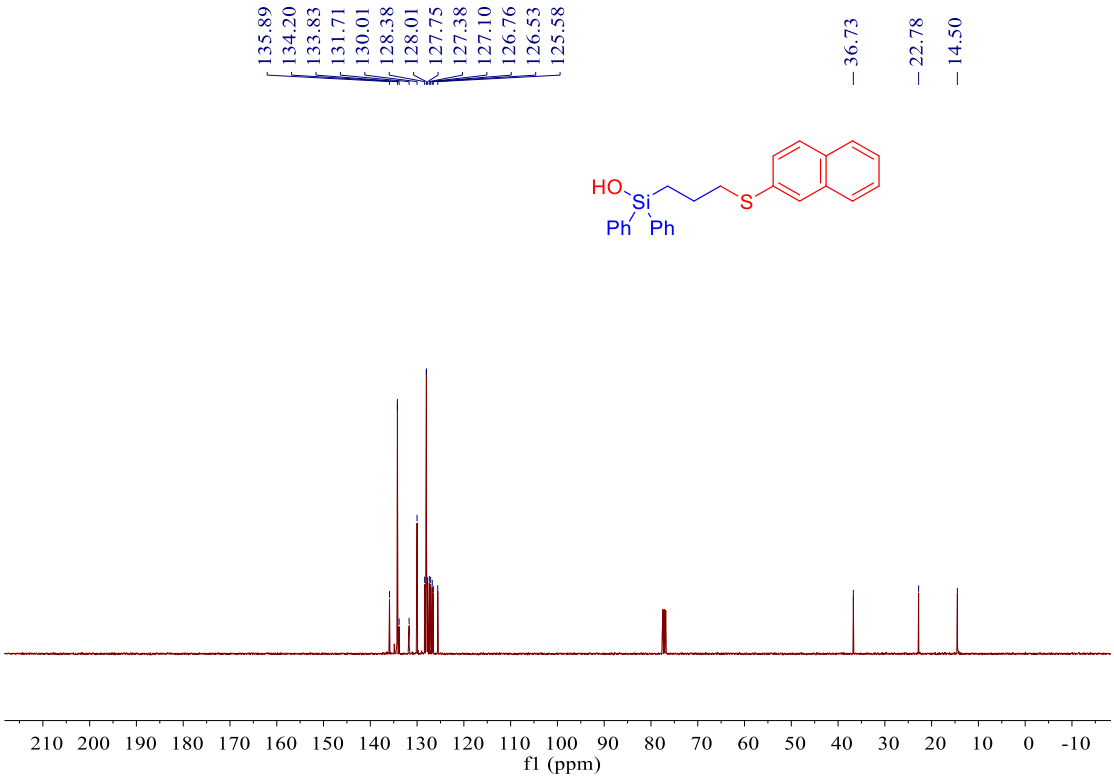
^{13}C NMR Spectra of **3d** (100 MHz, $CDCl_3$)



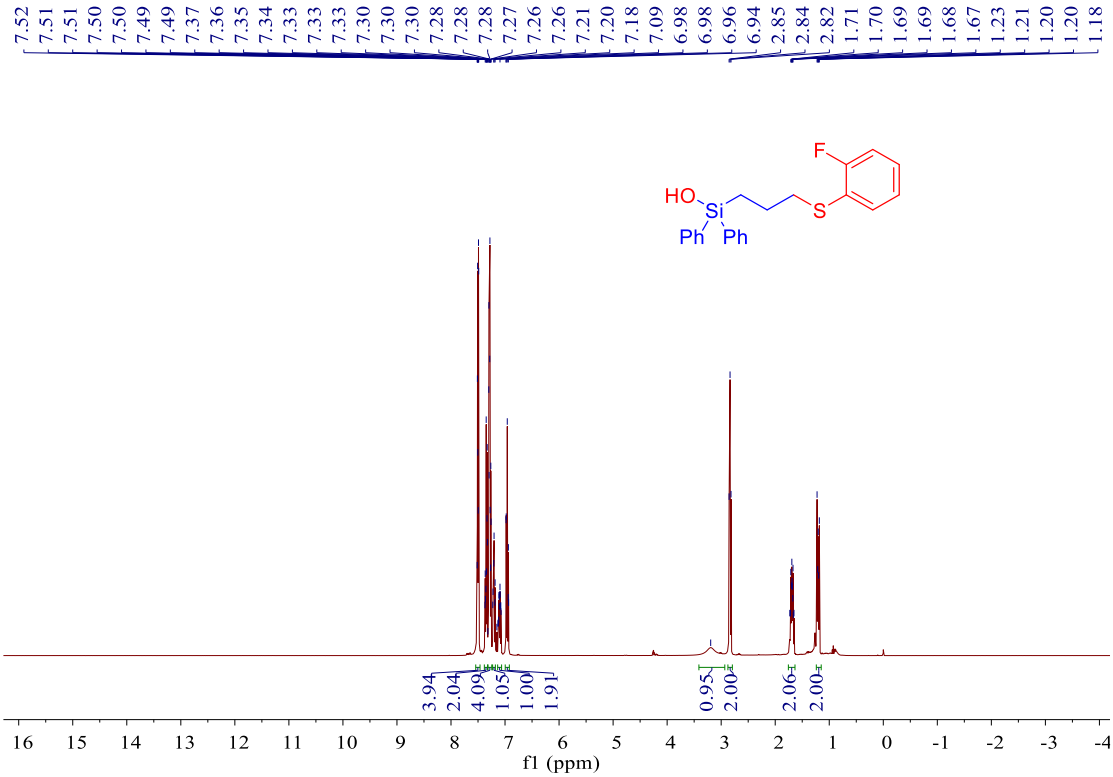
^{1}H NMR Spectra of **3e** (400 MHz, $CDCl_3$)



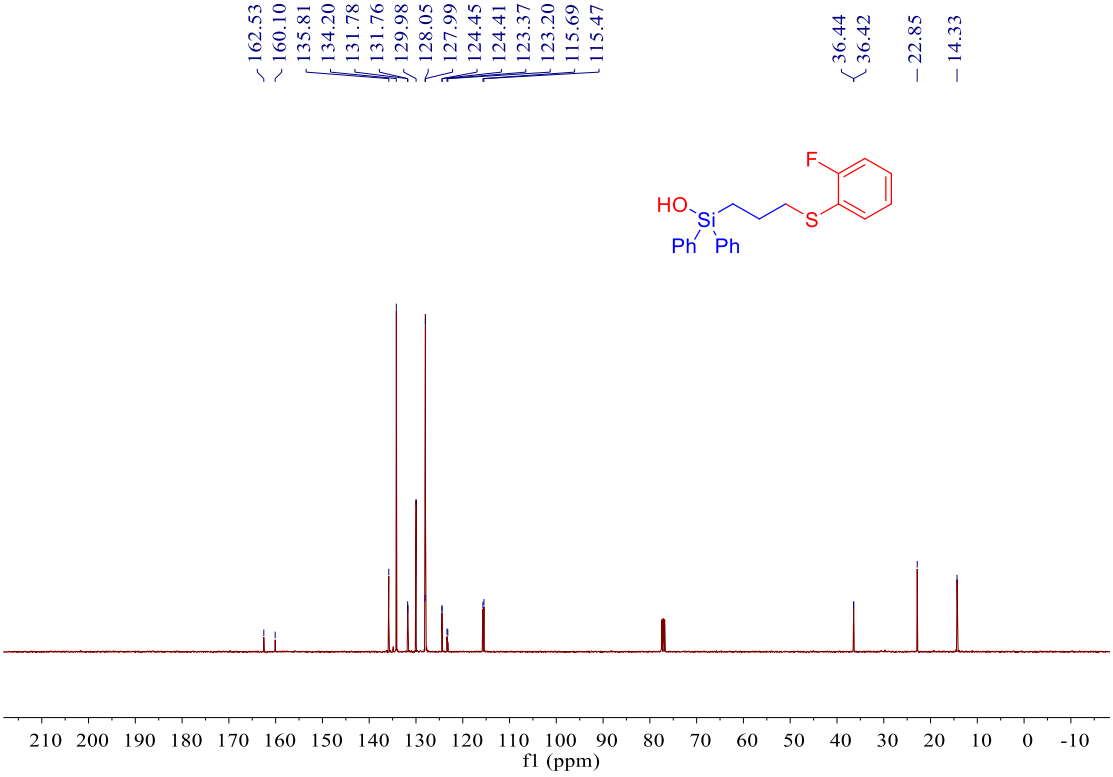
^{13}C NMR Spectra of **3e** (100 MHz, $CDCl_3$)



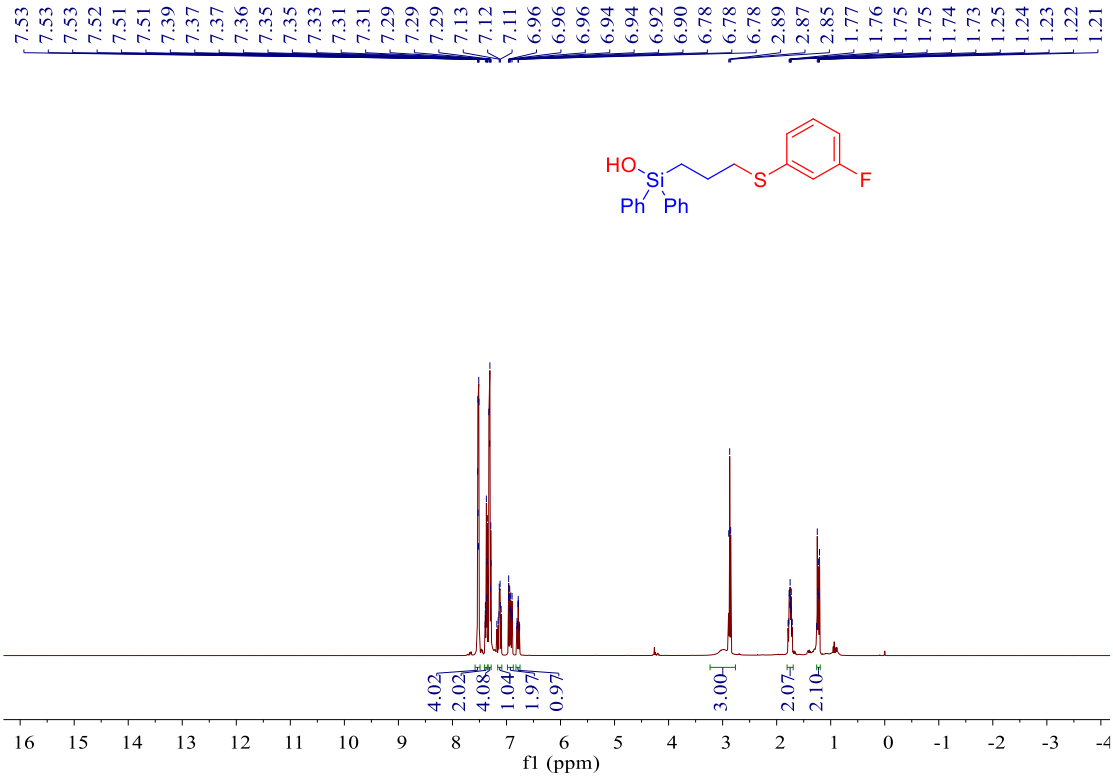
^{1}H NMR Spectra of **3f** (400 MHz, $CDCl_3$)



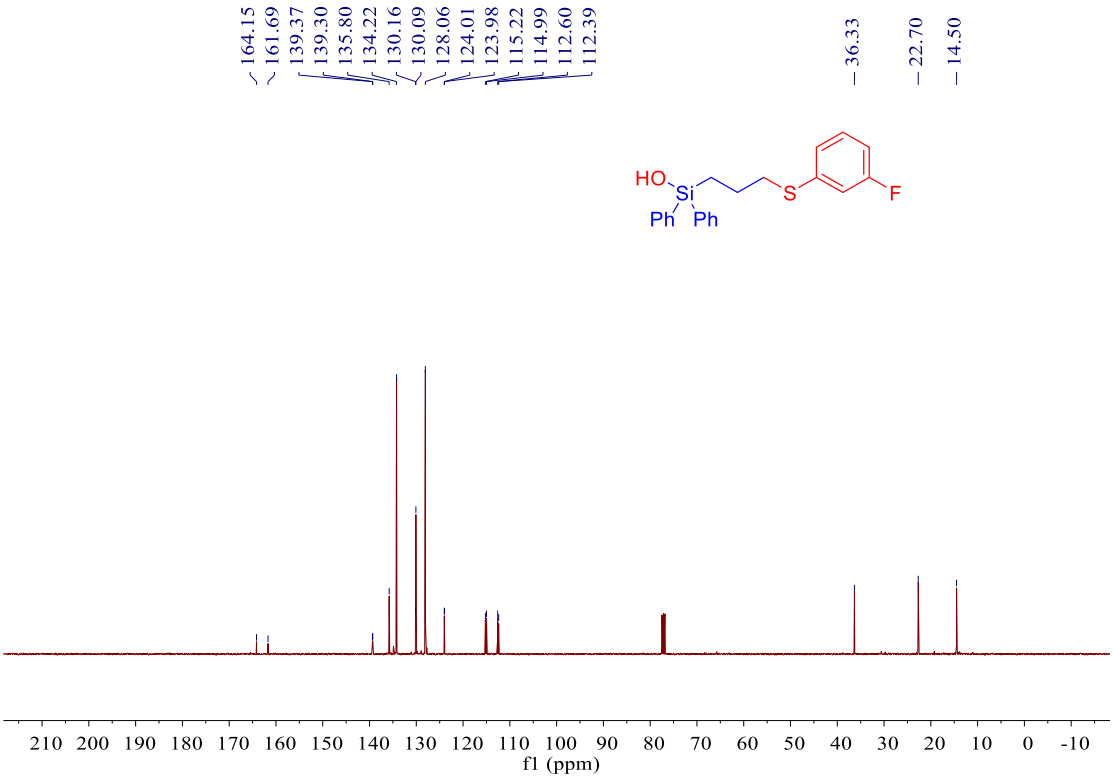
^{13}C NMR Spectra of **3f** (100 MHz, $CDCl_3$)



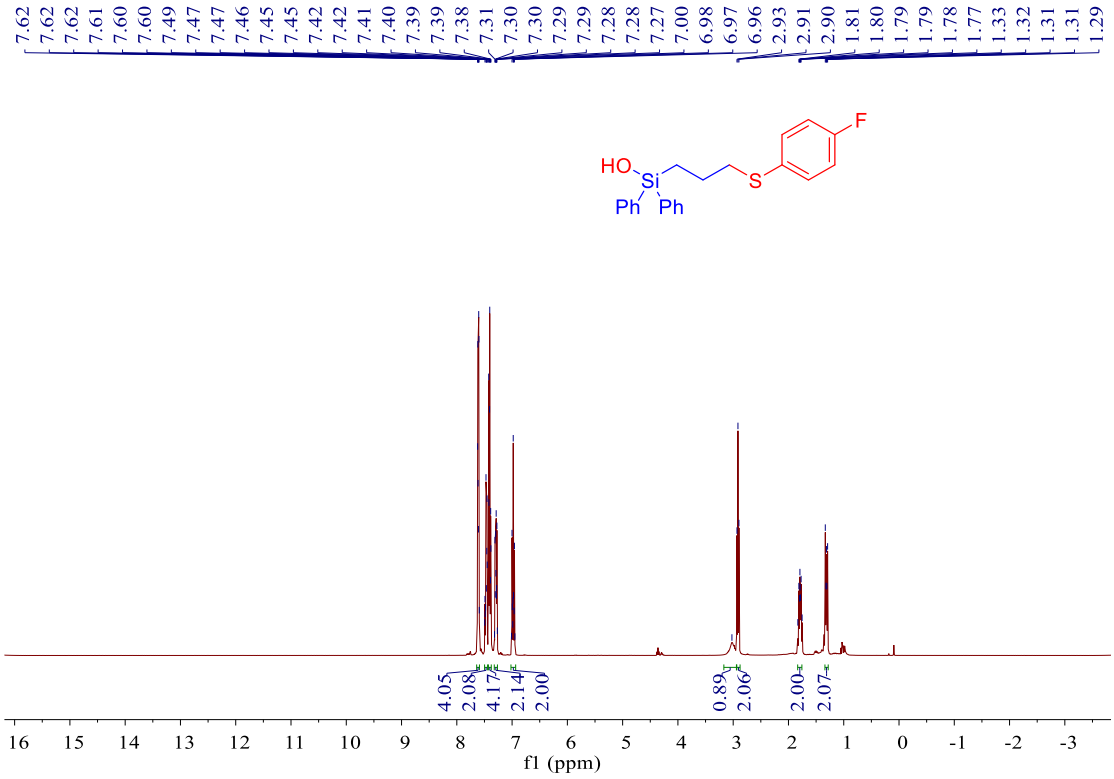
^{1}H NMR Spectra of **3g** (400 MHz, $CDCl_3$)



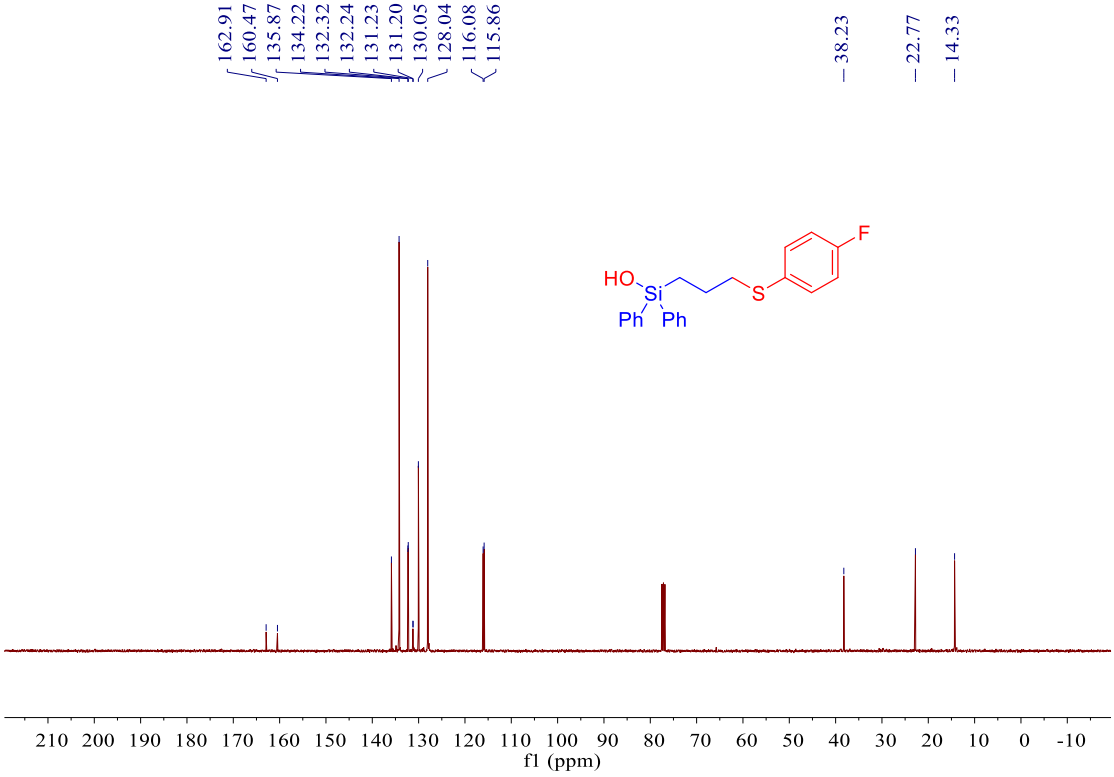
^{13}C NMR Spectra of **3g** (100 MHz, $CDCl_3$)



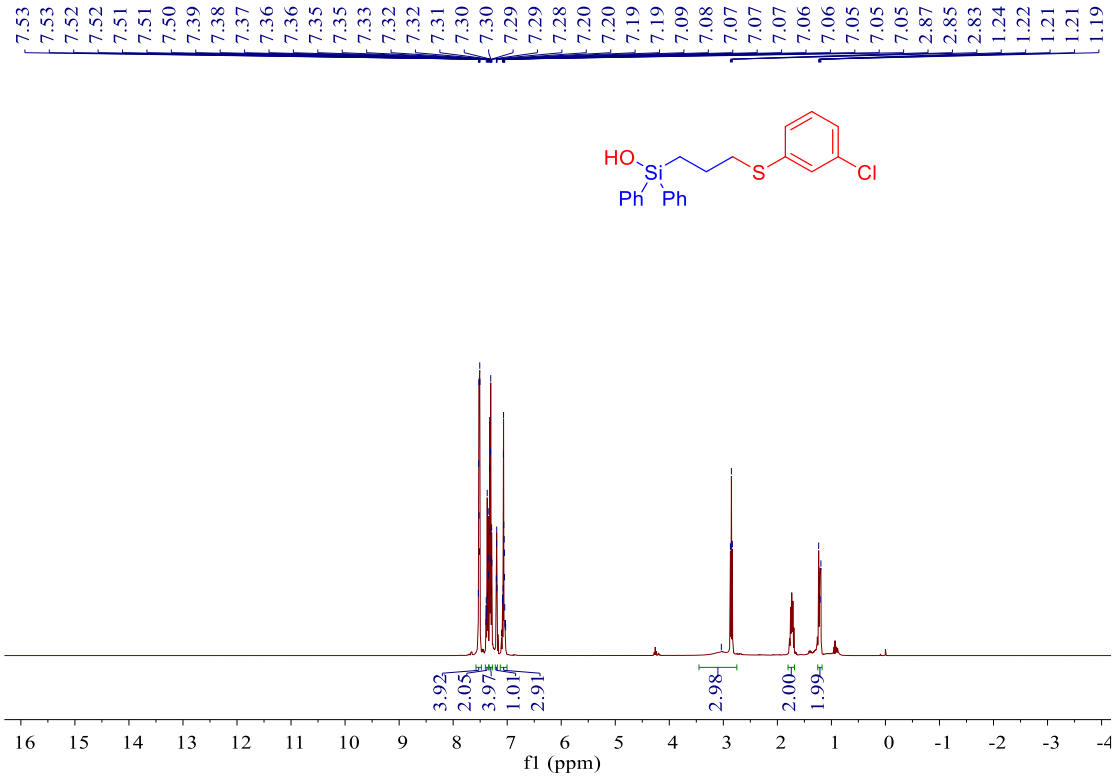
^{1}H NMR Spectra of **3h** (400 MHz, $CDCl_3$)



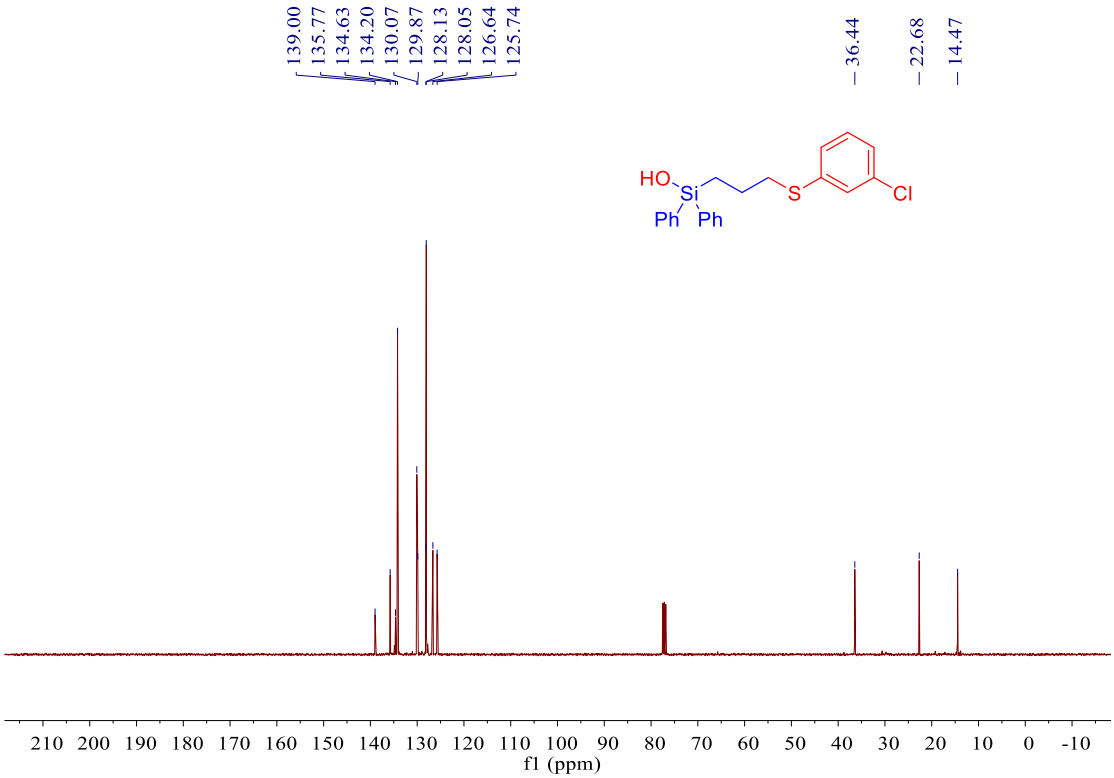
^{13}C NMR Spectra of **3h** (100 MHz, $CDCl_3$)



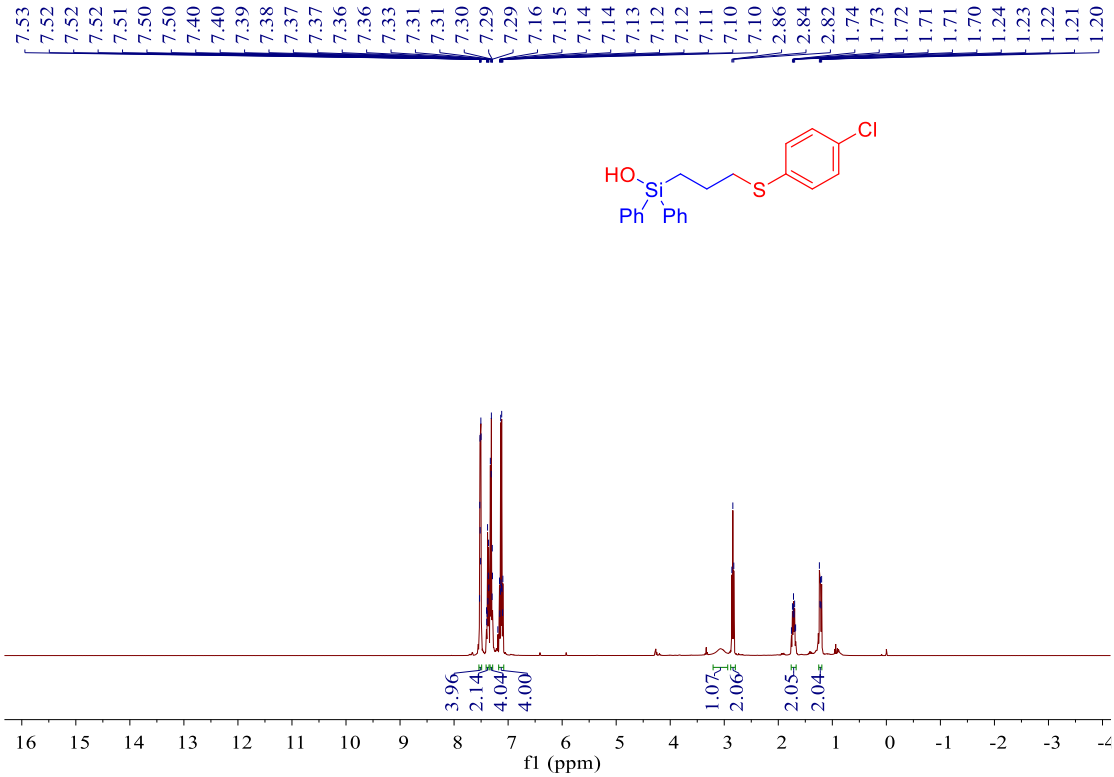
^{1}H NMR Spectra of **3i** (400 MHz, $CDCl_3$)



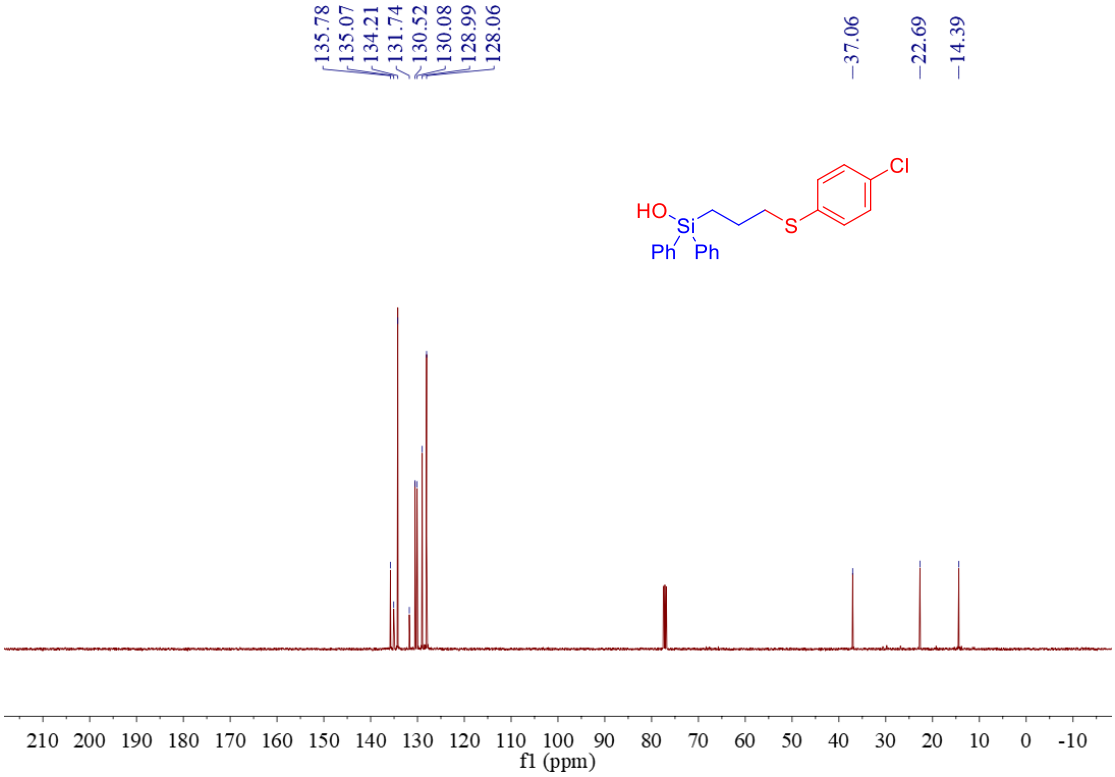
^{13}C NMR Spectra of **3i** (100 MHz, $CDCl_3$)



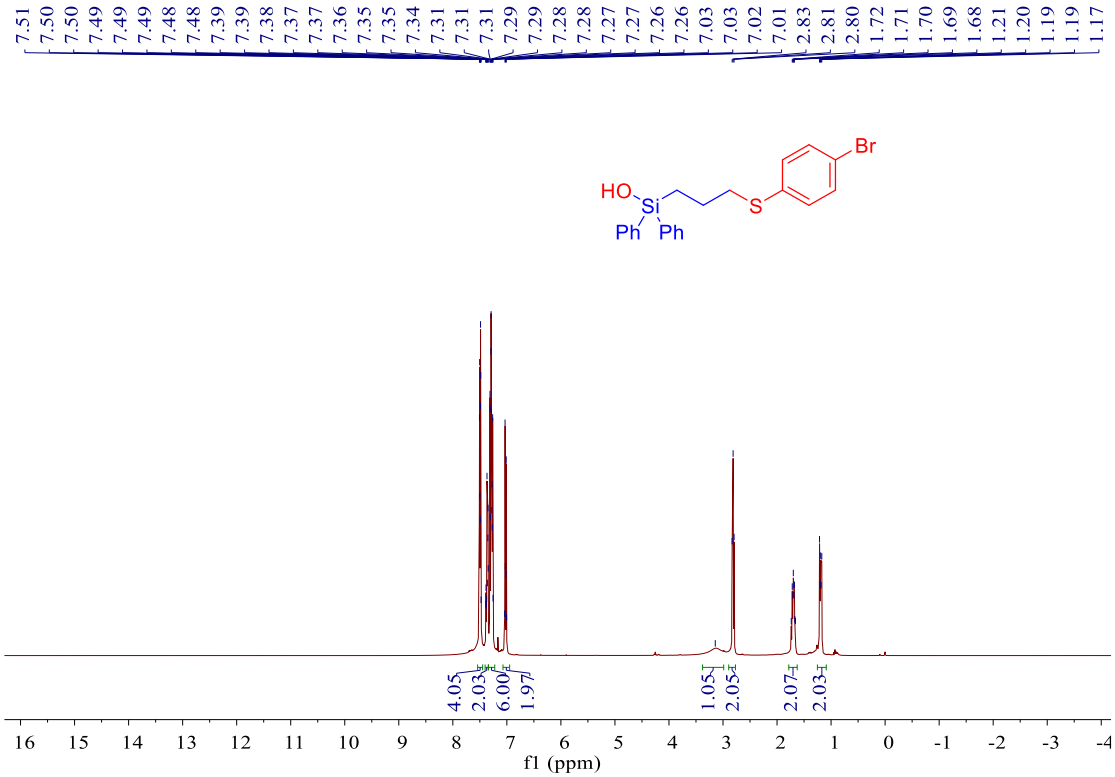
^{1}H NMR Spectra of **3j** (400 MHz, $CDCl_3$)



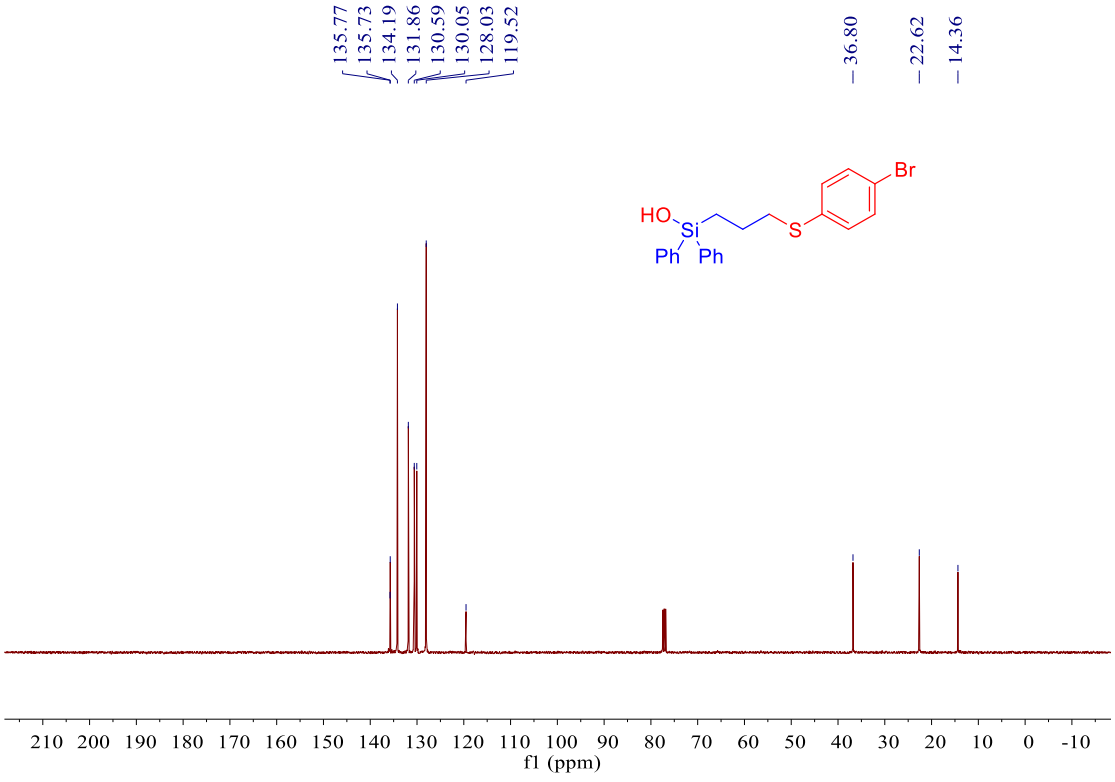
^{13}C NMR Spectra of **3j** (100 MHz, $CDCl_3$)



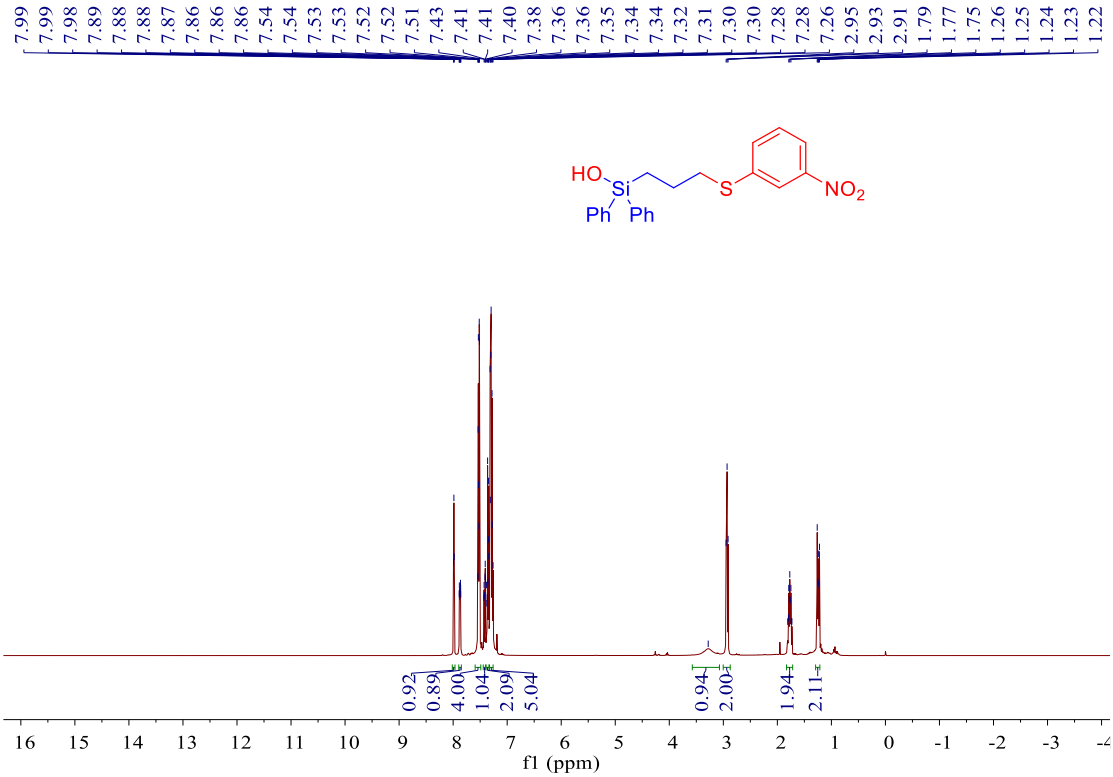
^{1}H NMR Spectra of **3k** (400 MHz, $CDCl_3$)



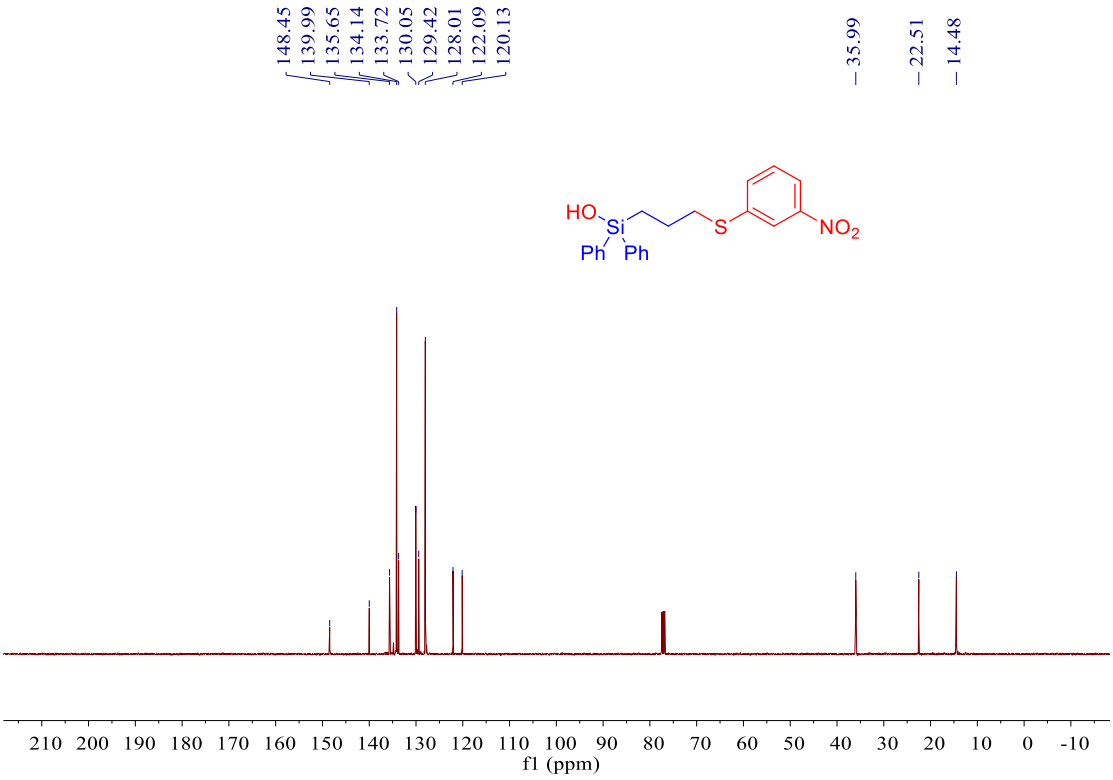
^{13}C NMR Spectra of **3k** (100 MHz, $CDCl_3$)



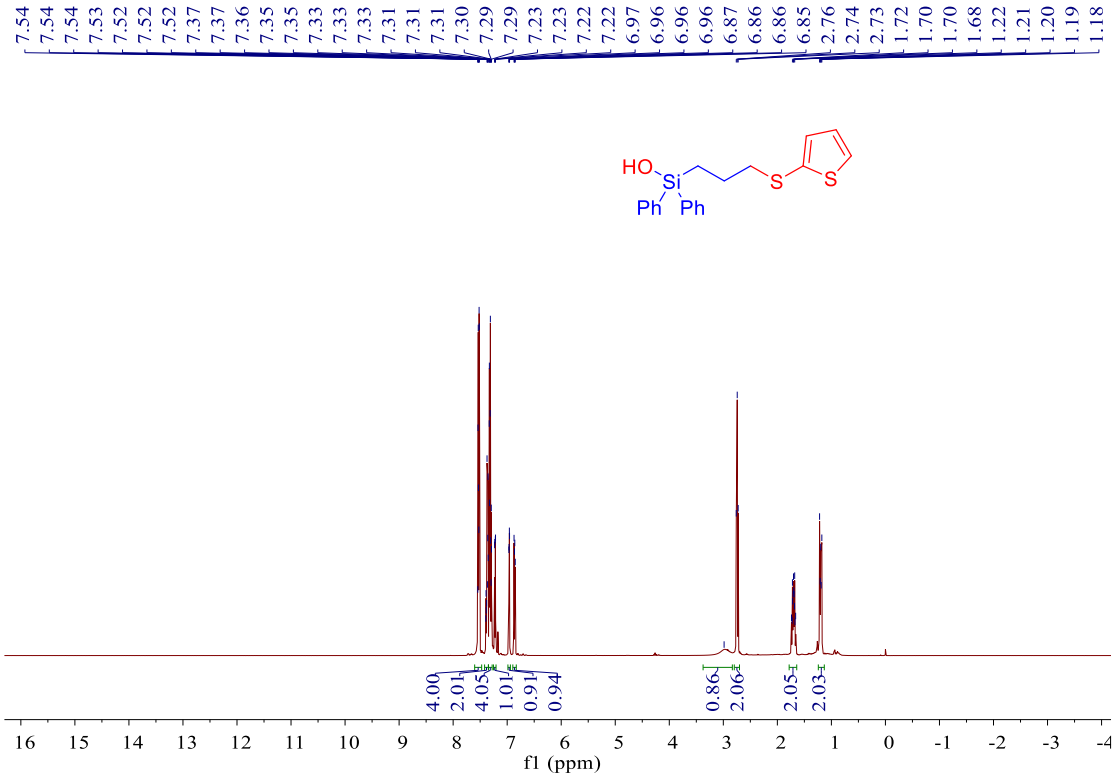
^{1}H NMR Spectra of **3l** (400 MHz, $CDCl_3$)



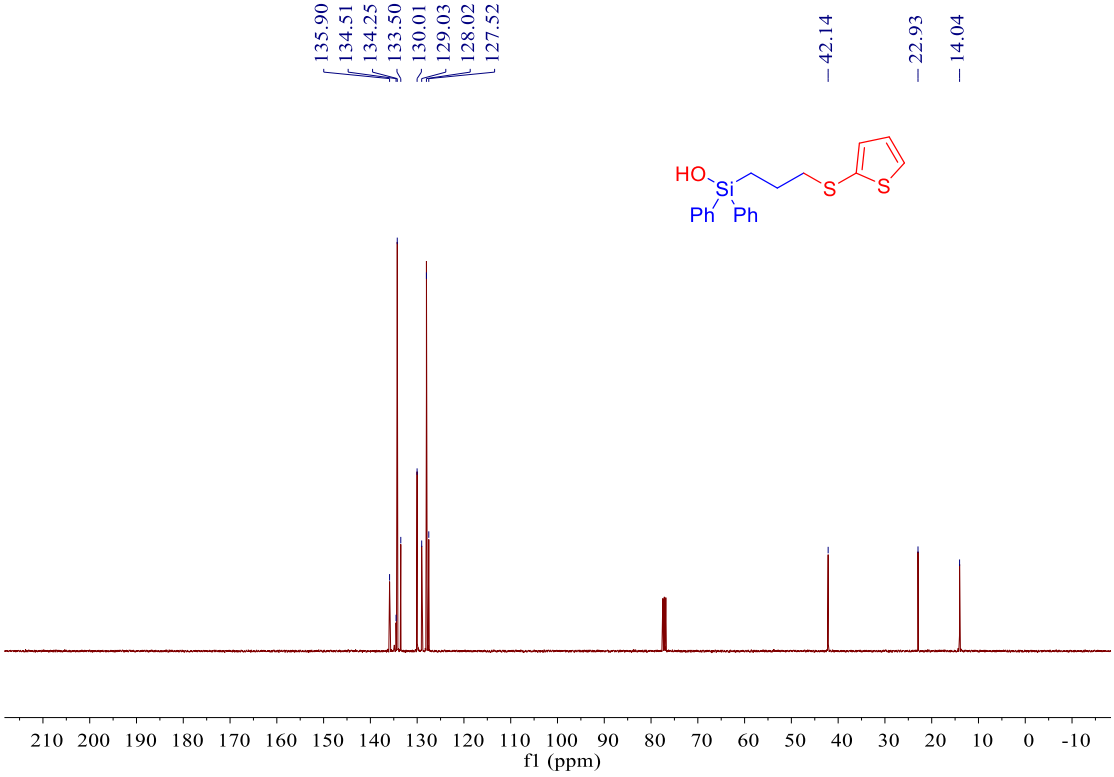
^{13}C NMR Spectra of **3l** (100 MHz, $CDCl_3$)



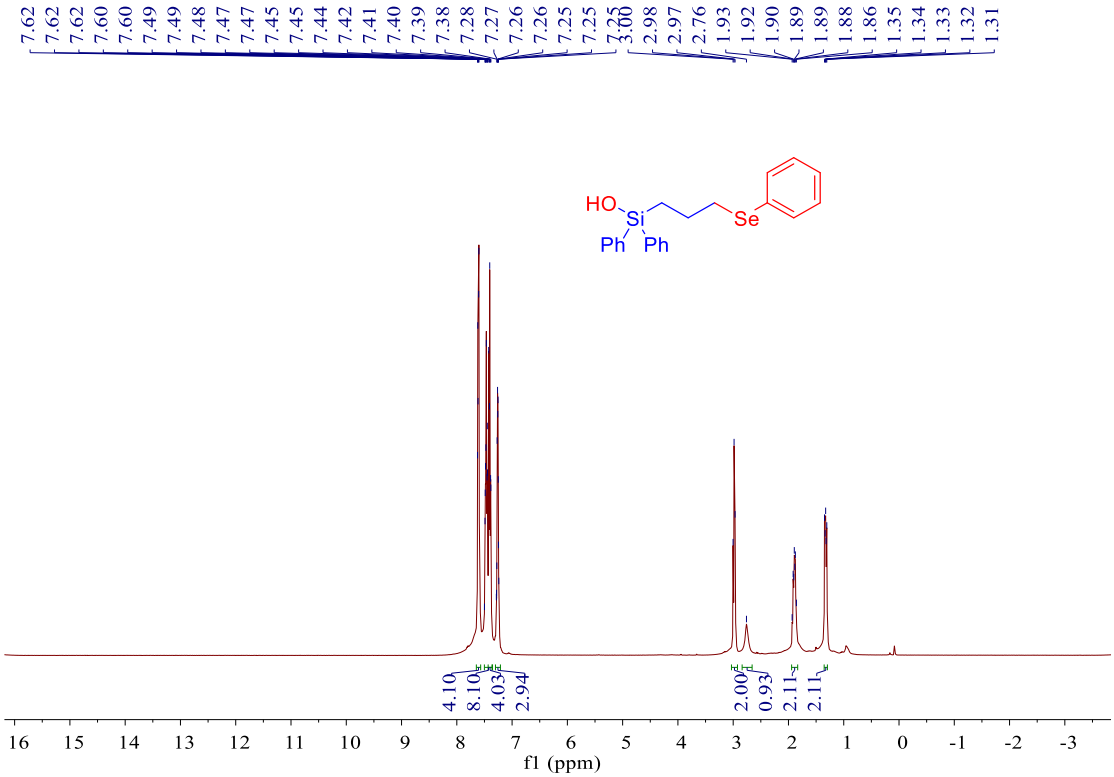
^{1}H NMR Spectra of **3m** (400 MHz, $CDCl_3$)



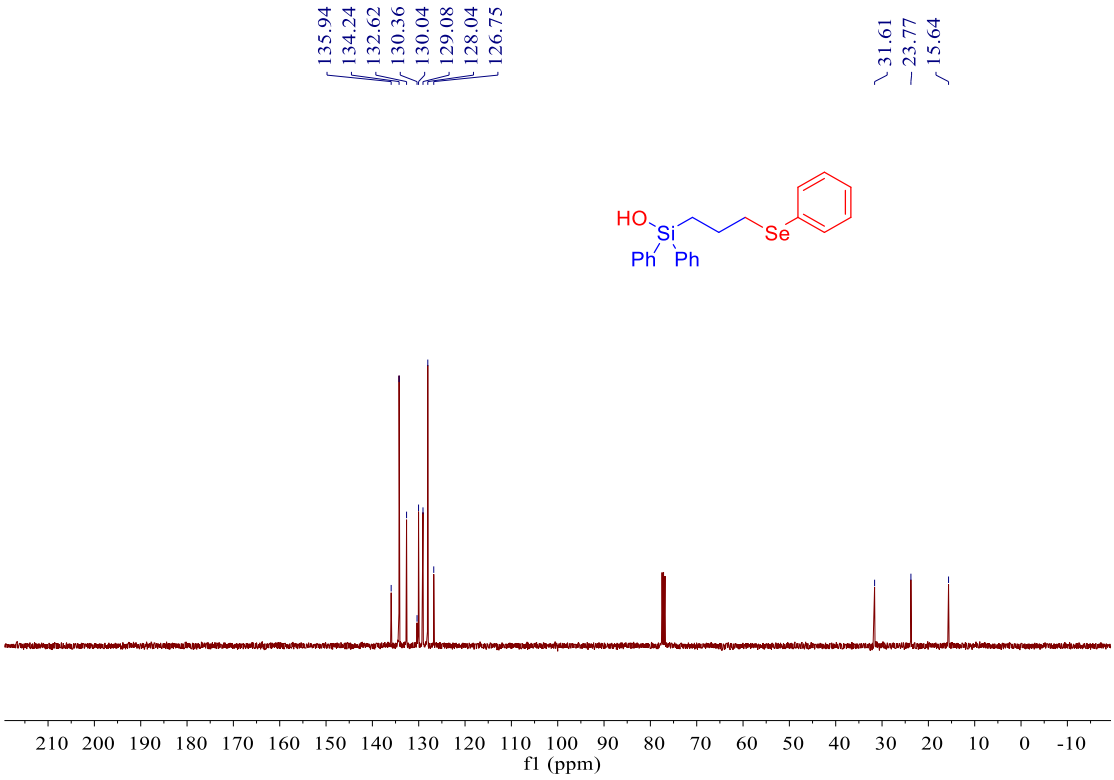
^{13}C NMR Spectra of **3m** (100 MHz, $CDCl_3$)



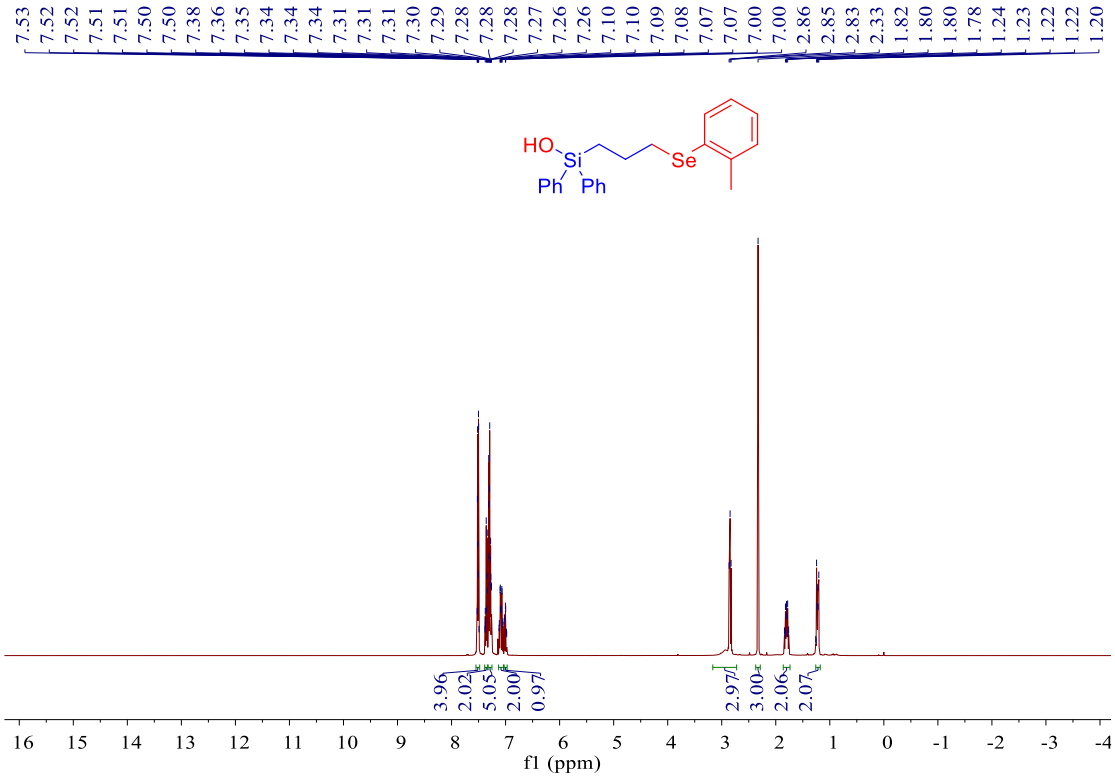
^{1}H NMR Spectra of **3n** (400 MHz, $CDCl_3$)



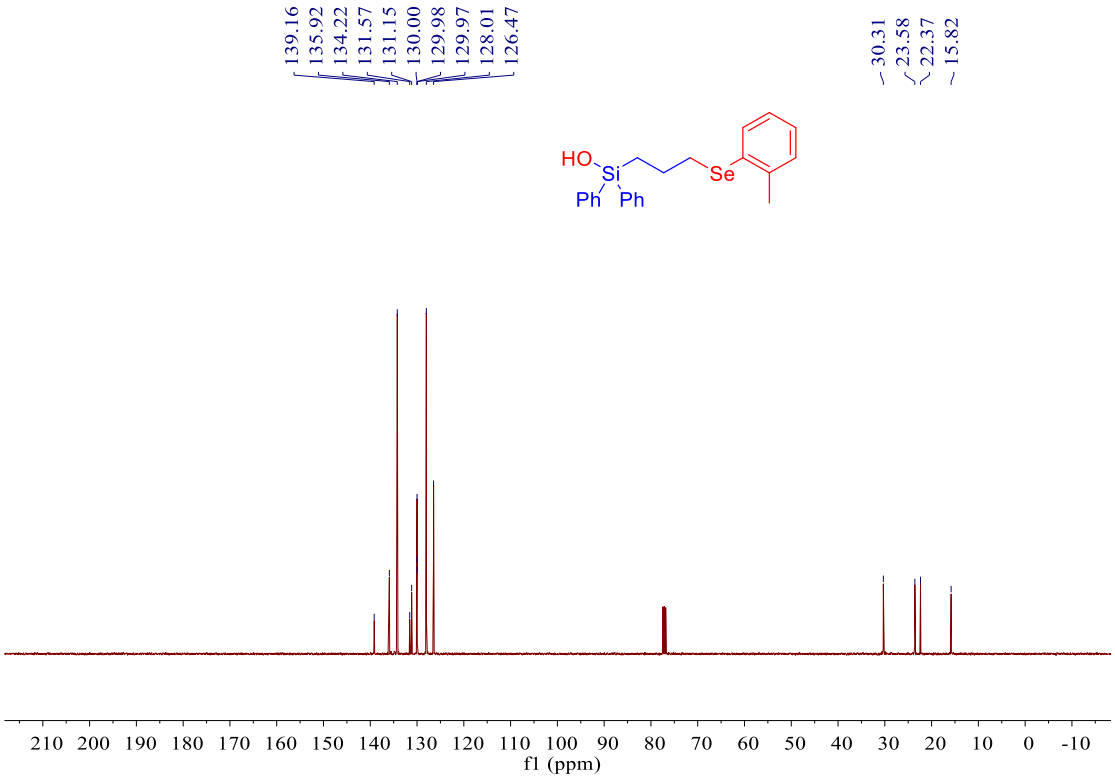
^{13}C NMR Spectra of **3n** (100 MHz, $CDCl_3$)



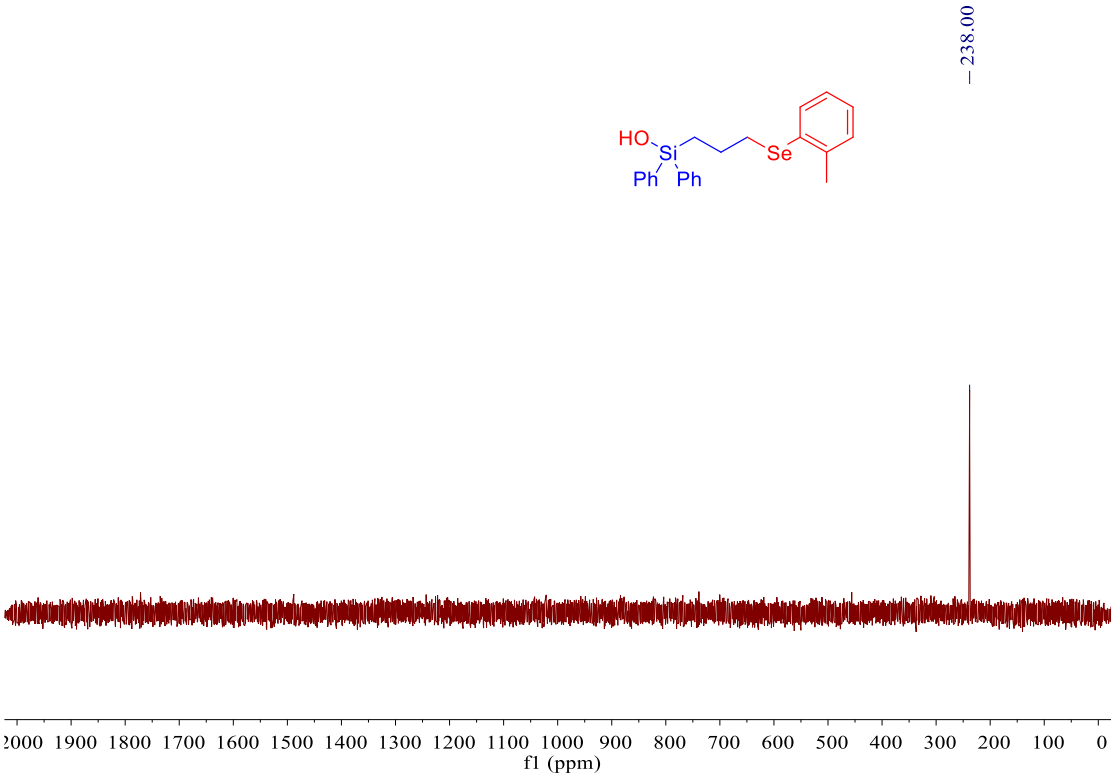
^{1}H NMR Spectra of **3o** (400 MHz, $CDCl_3$)



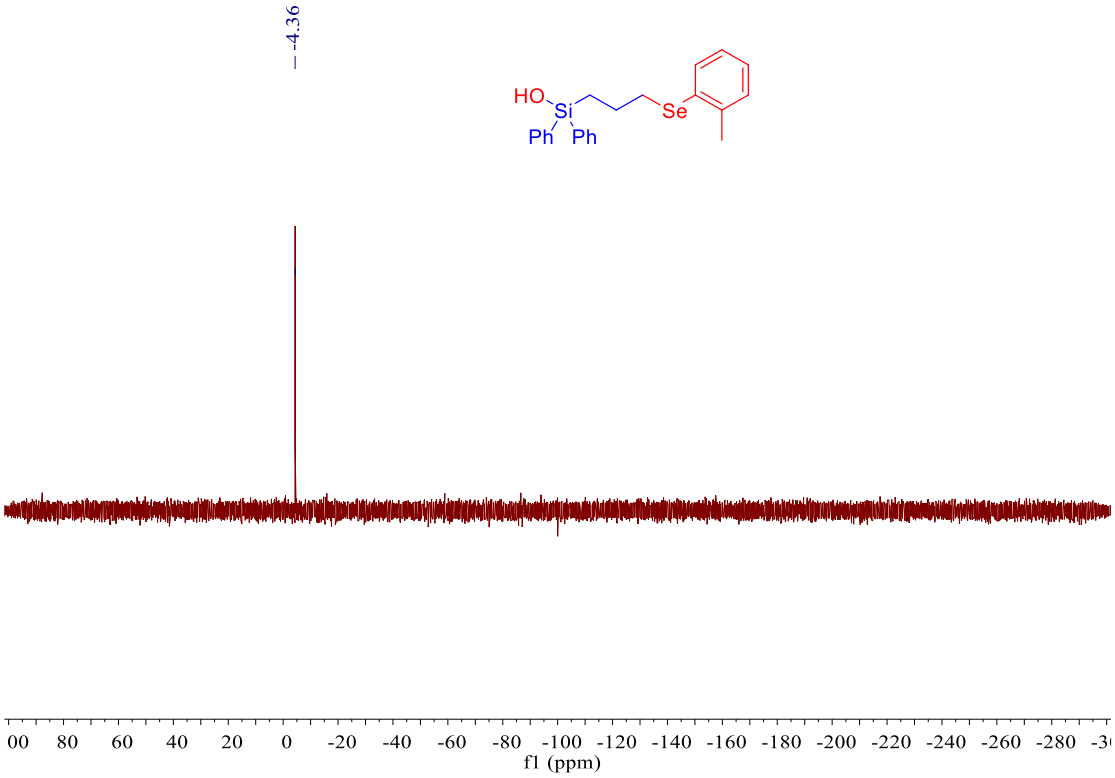
^{13}C NMR Spectra of **3o** (100 MHz, $CDCl_3$)



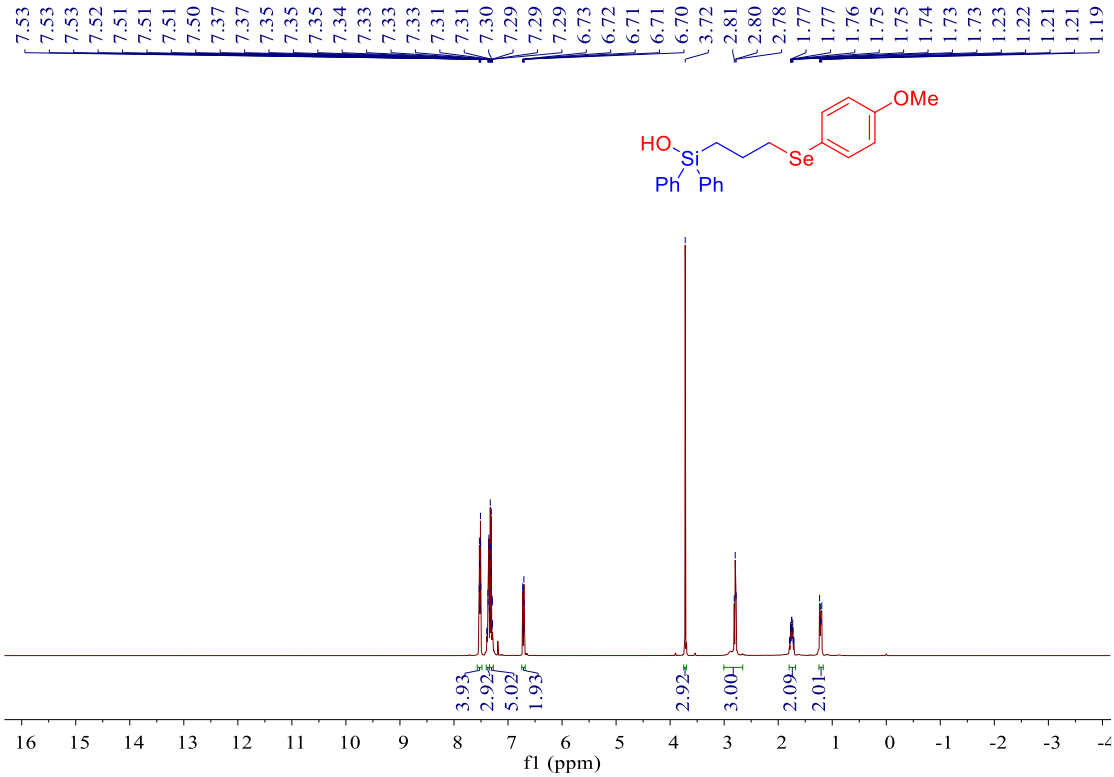
^{77}Se NMR Spectra of **3o** (400 MHz, $CDCl_3$)



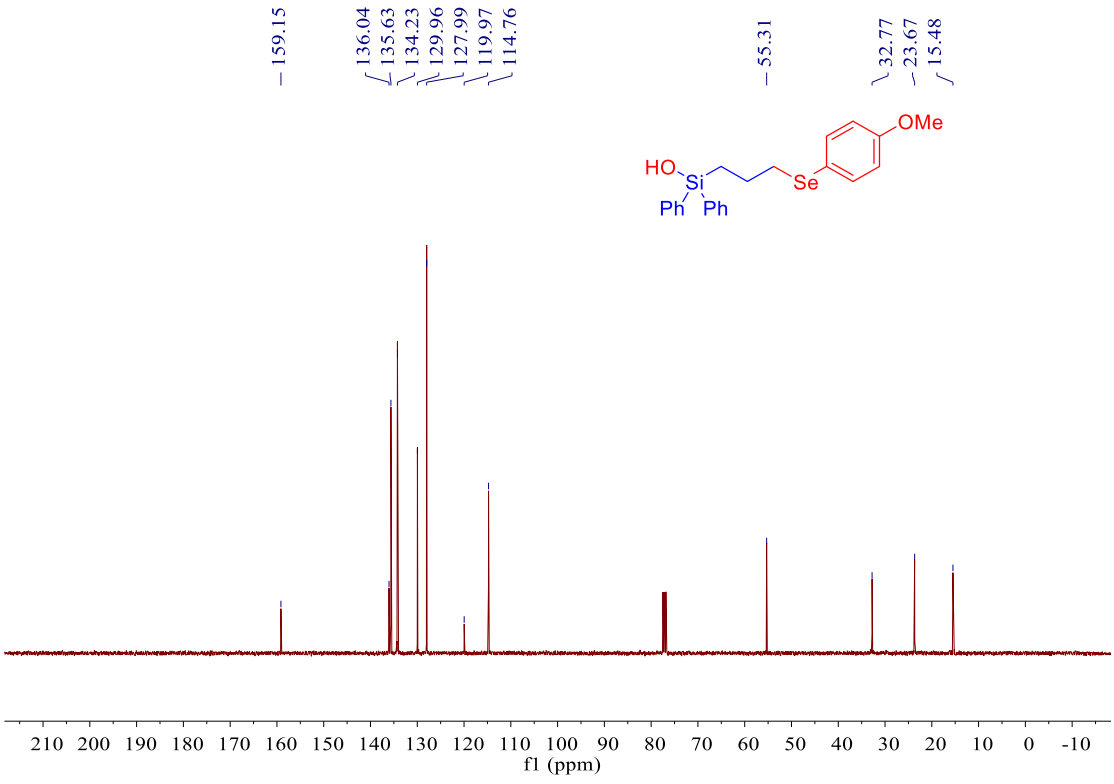
^{29}Si NMR Spectra of **3o** (79 MHz, $CDCl_3$)



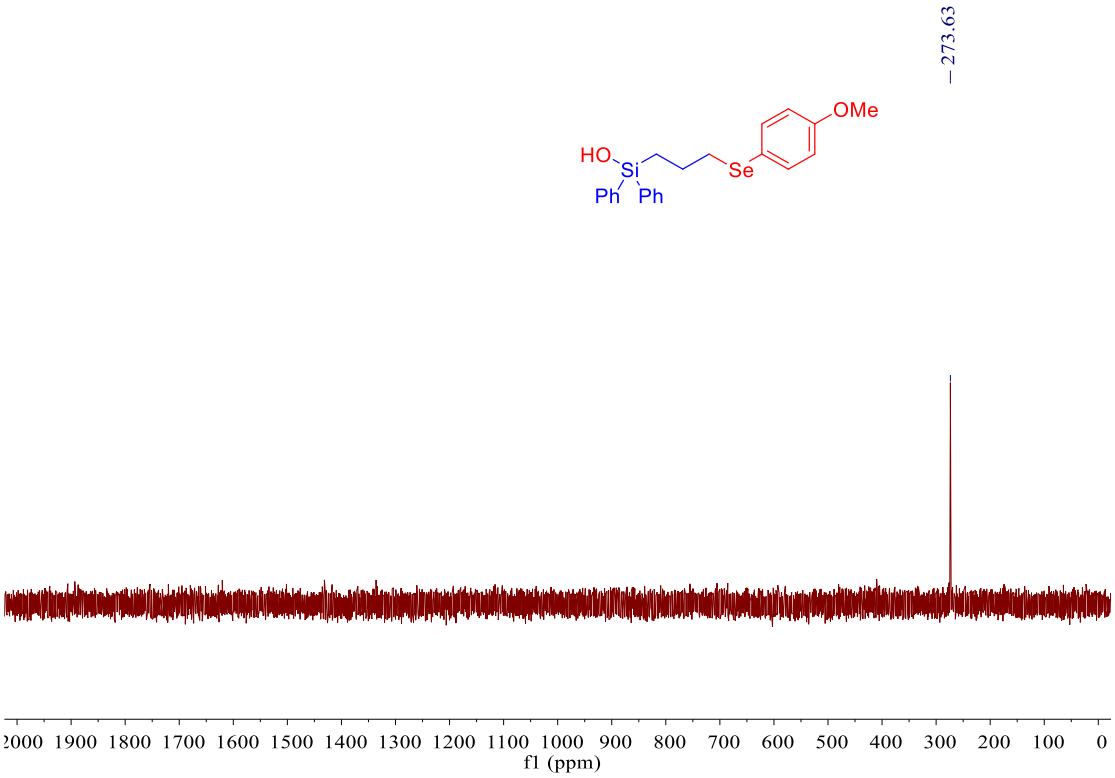
^{1}H NMR Spectra of **3p** (400 MHz, $CDCl_3$)



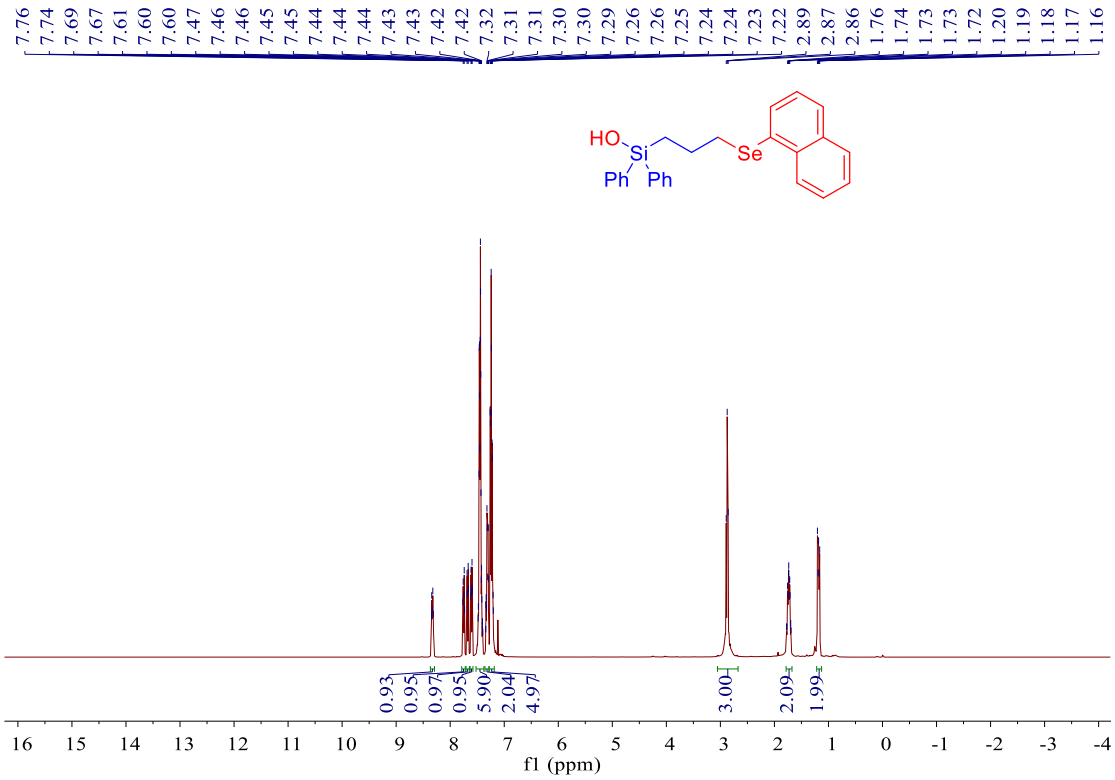
^{13}C NMR Spectra of **3p** (100 MHz, $CDCl_3$)



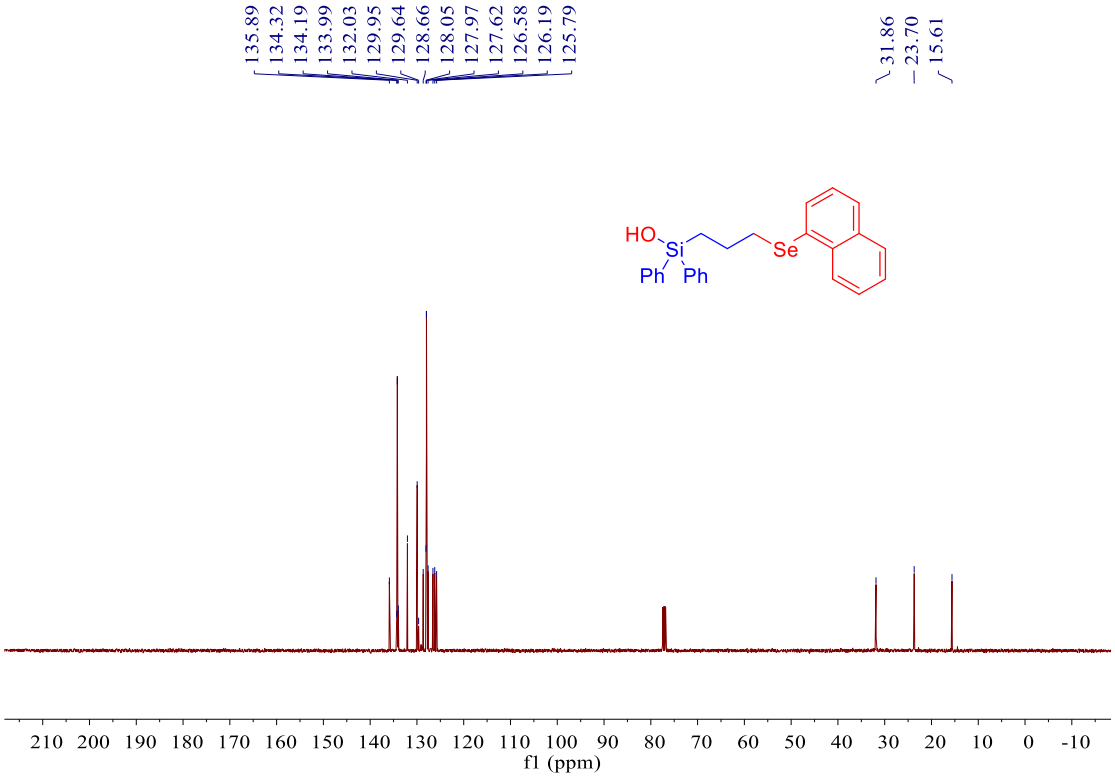
^{77} Se NMR Spectra of **3p** (100 MHz, $CDCl_3$)



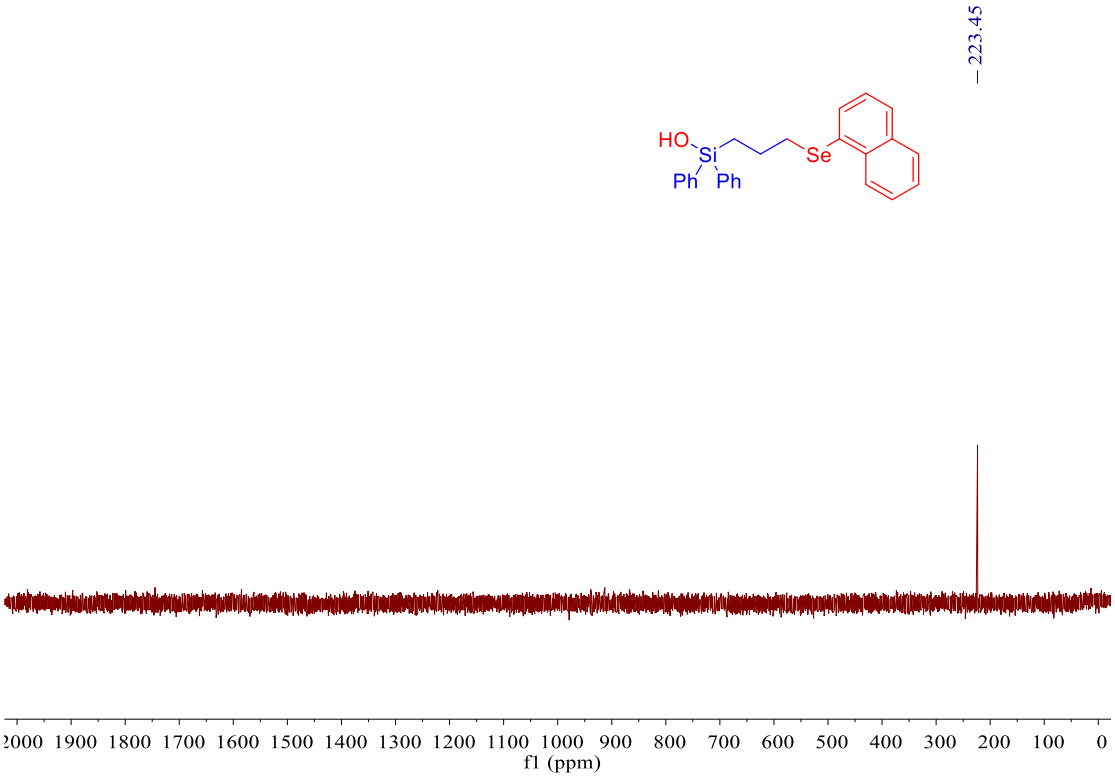
^{1}H NMR Spectra of **3q** (400 MHz, $CDCl_3$)



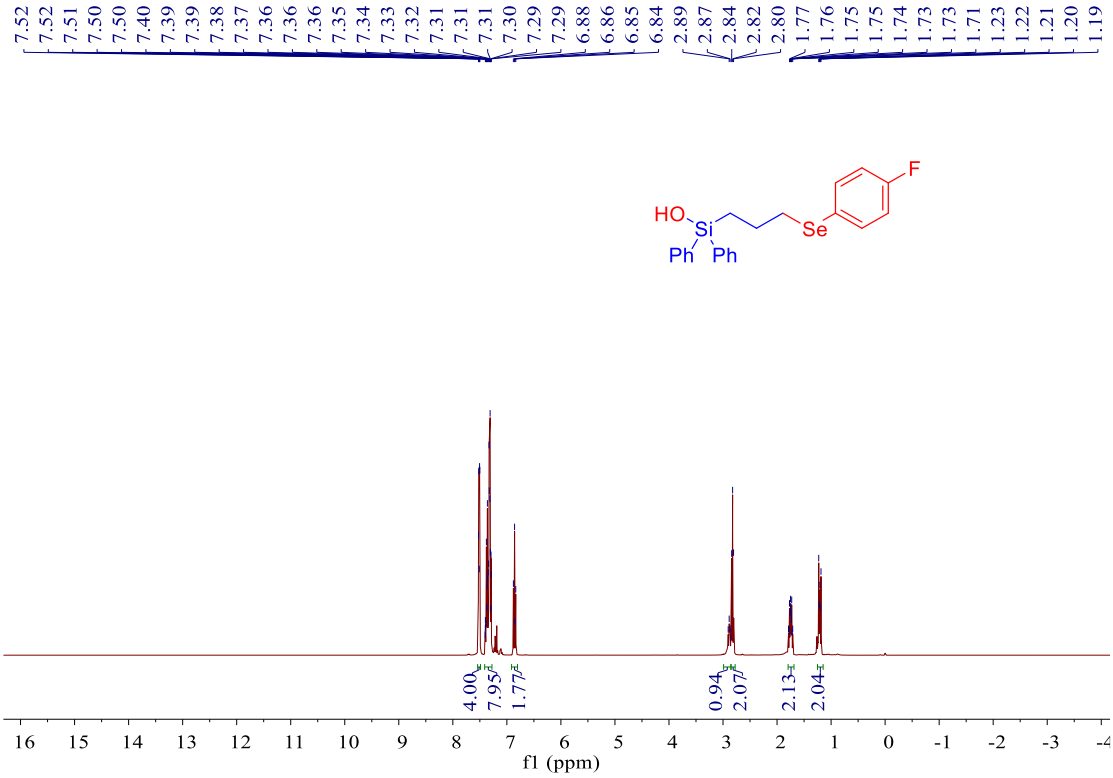
^{13}C NMR Spectra of **3q** (100 MHz, $CDCl_3$)



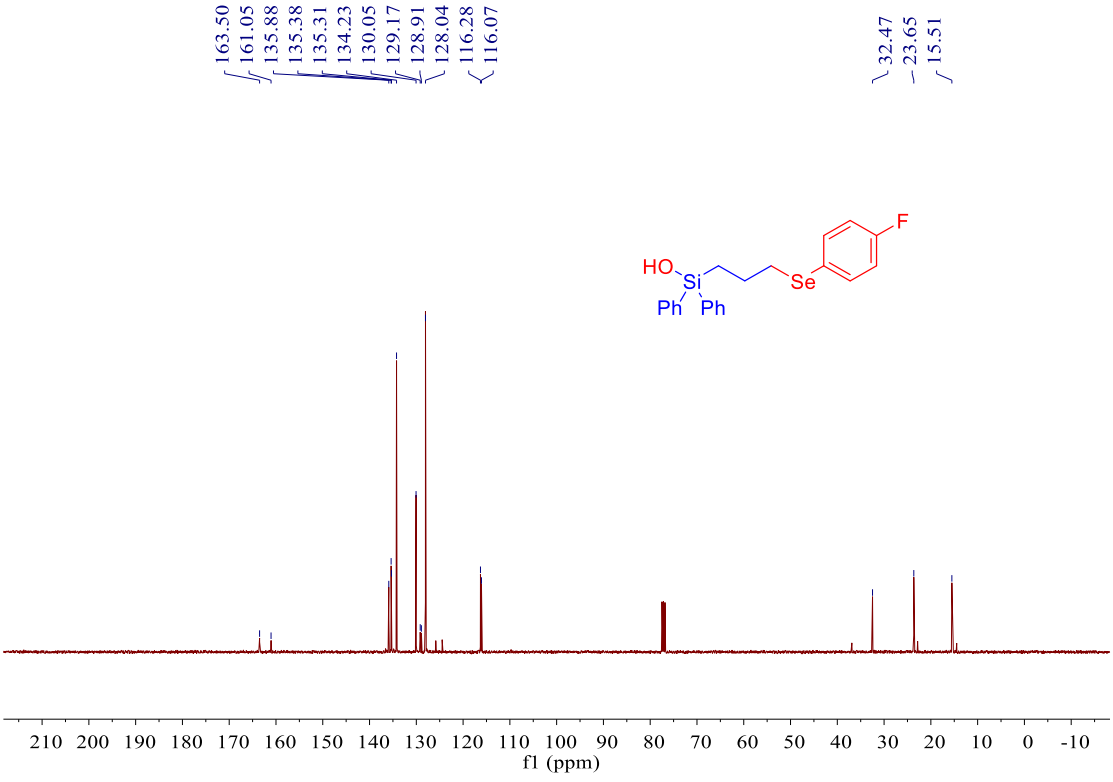
^{75}Se NMR Spectra of **3q** (400 MHz, $CDCl_3$)



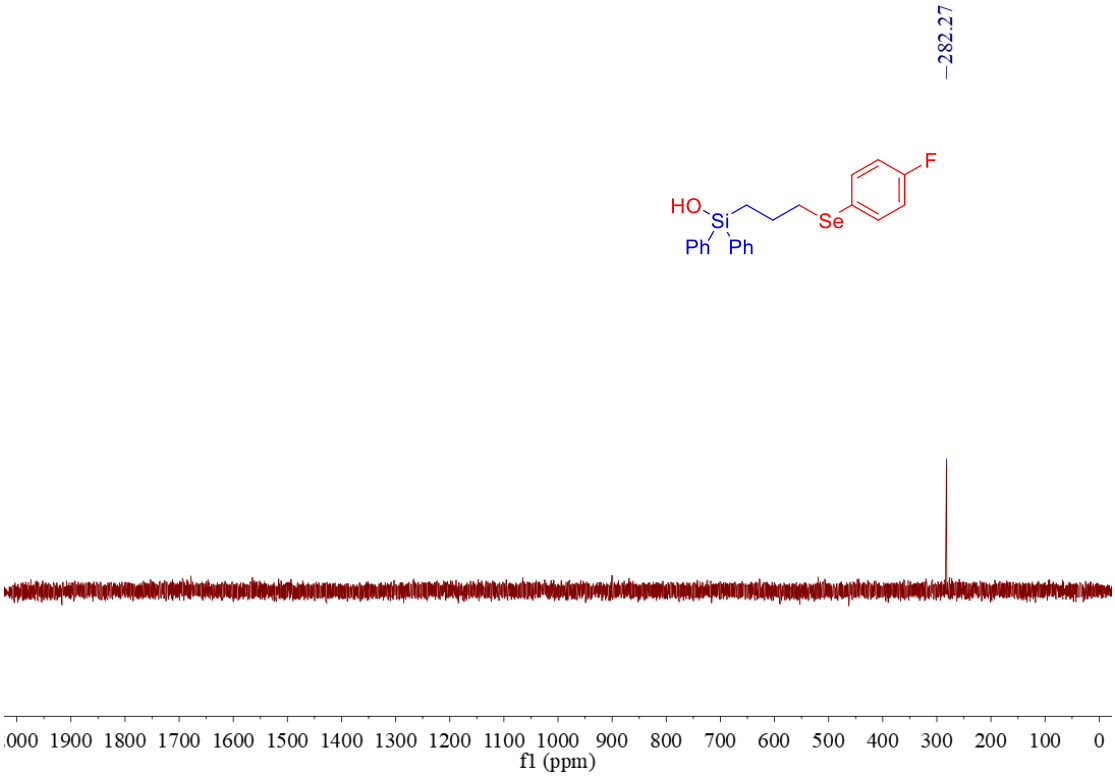
^{1}H NMR Spectra of **3r** (400 MHz, $CDCl_3$)



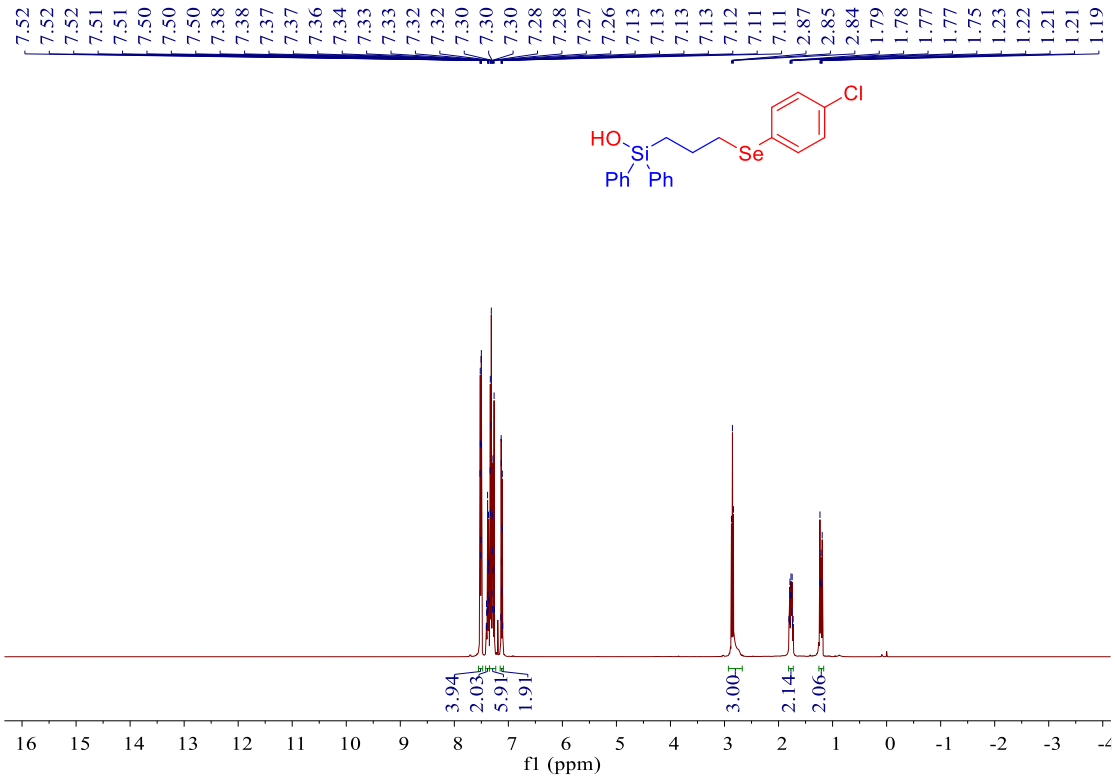
^{13}C NMR Spectra of **3r** (100 MHz, $CDCl_3$)



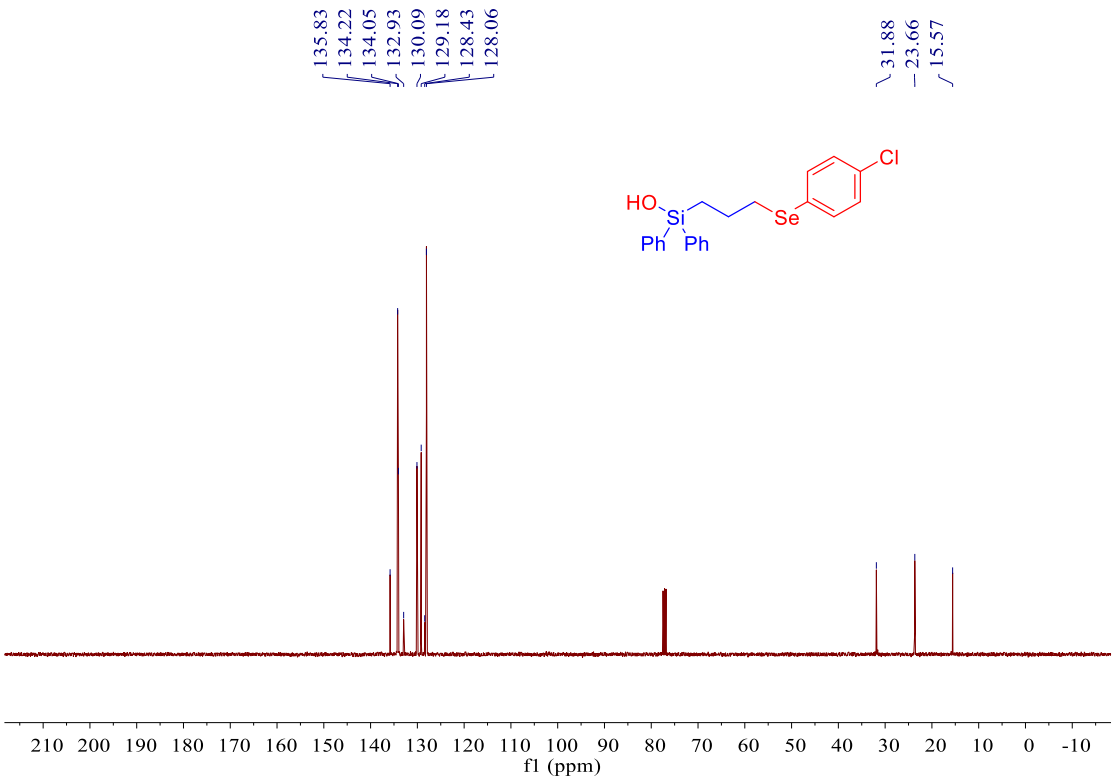
^{77}Se NMR Spectra of **3r** (400 MHz, $CDCl_3$)



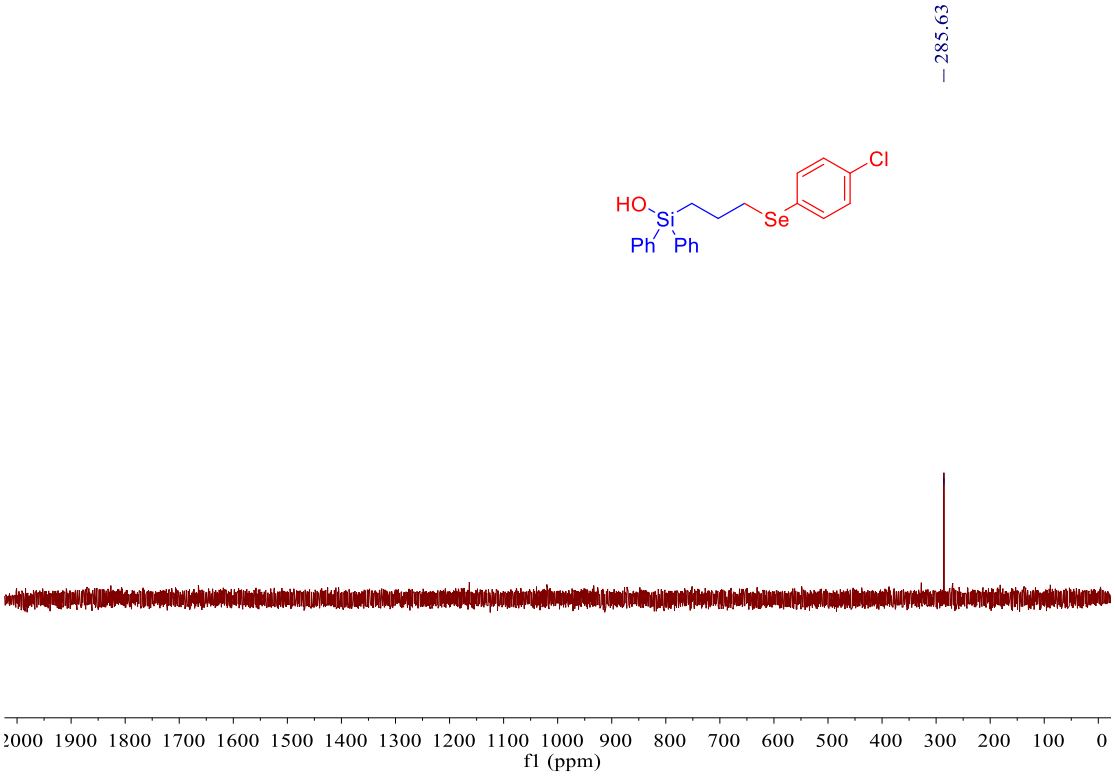
^{1}H NMR Spectra of **3s** (400 MHz, $CDCl_3$)



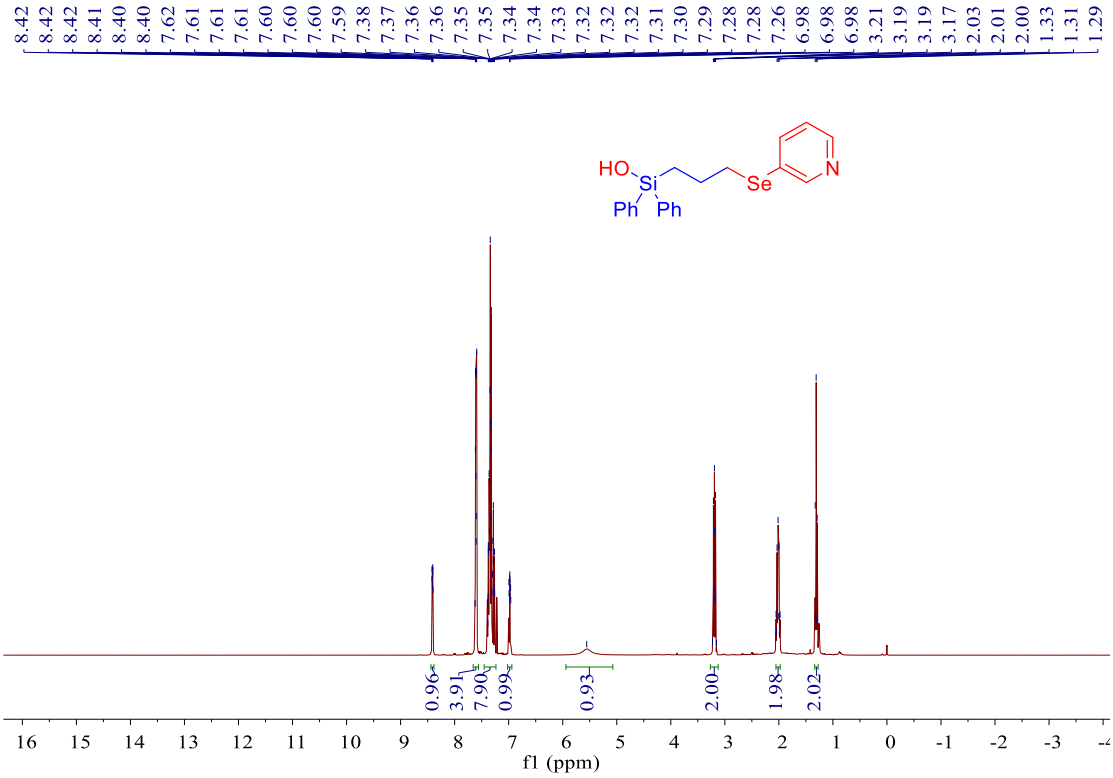
^{13}C NMR Spectra of **3s** (100 MHz, $CDCl_3$)



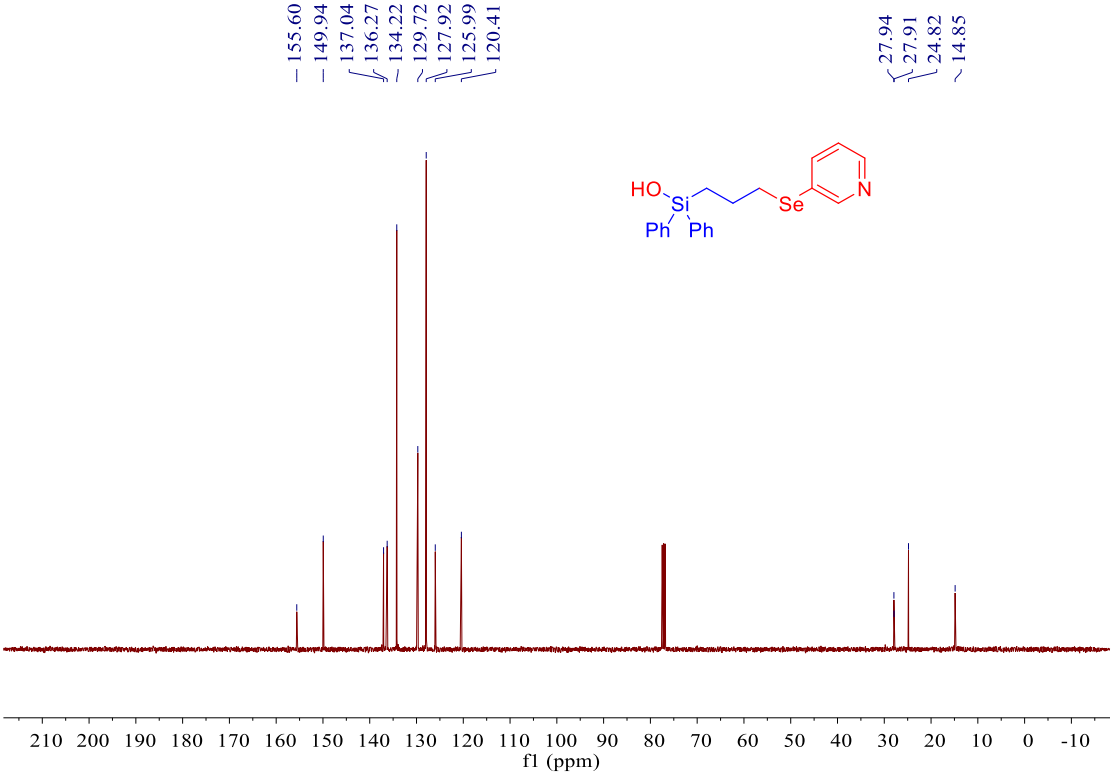
^{77}Se NMR Spectra of **3s** (100 MHz, $CDCl_3$)



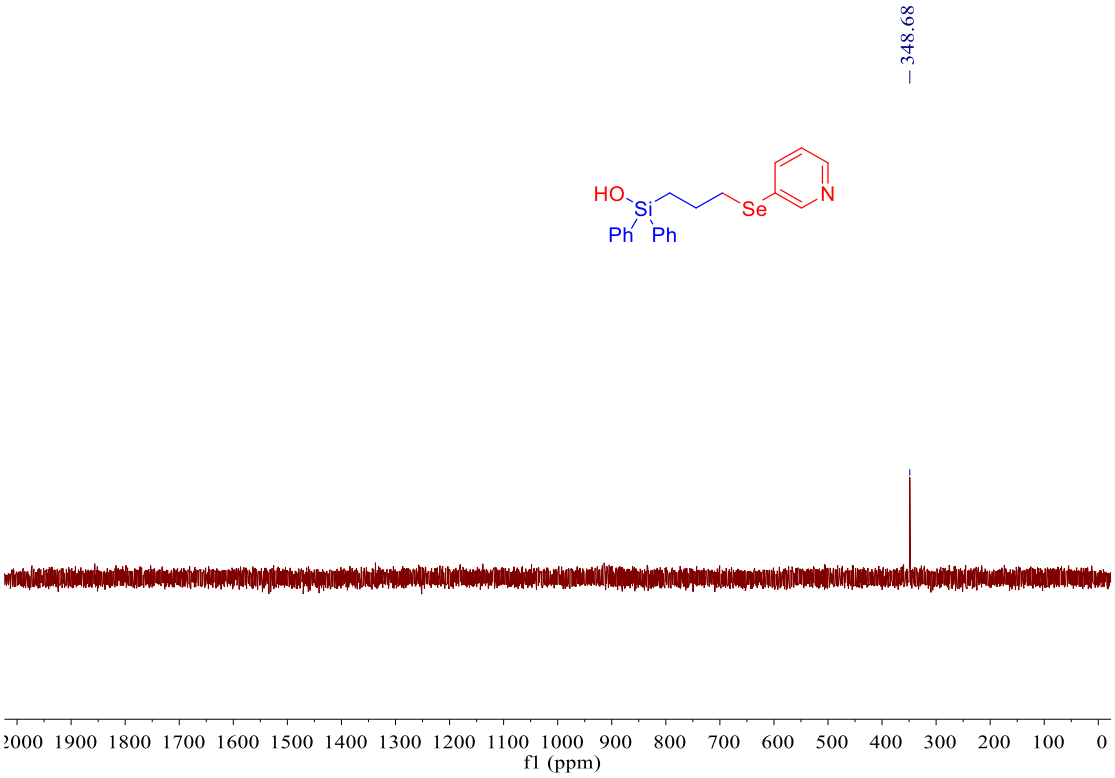
^{1}H NMR Spectra of **3t** (400 MHz, $CDCl_3$)



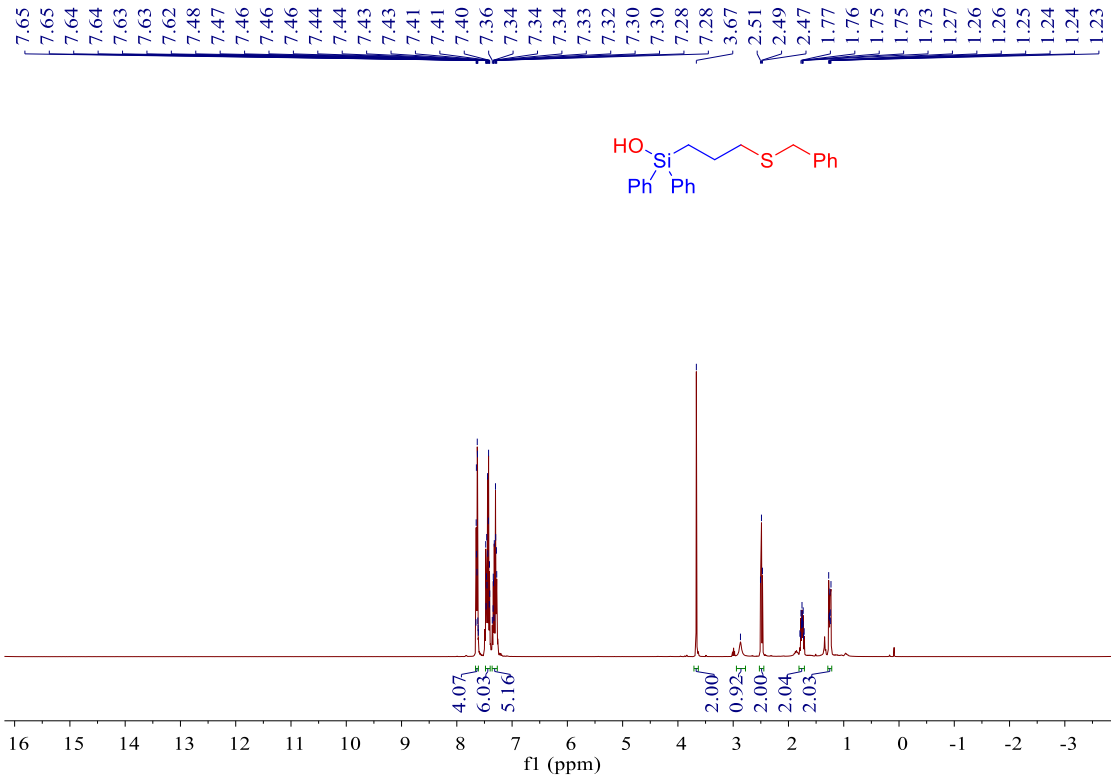
^{13}C NMR Spectra of **3t** (100 MHz, $CDCl_3$)



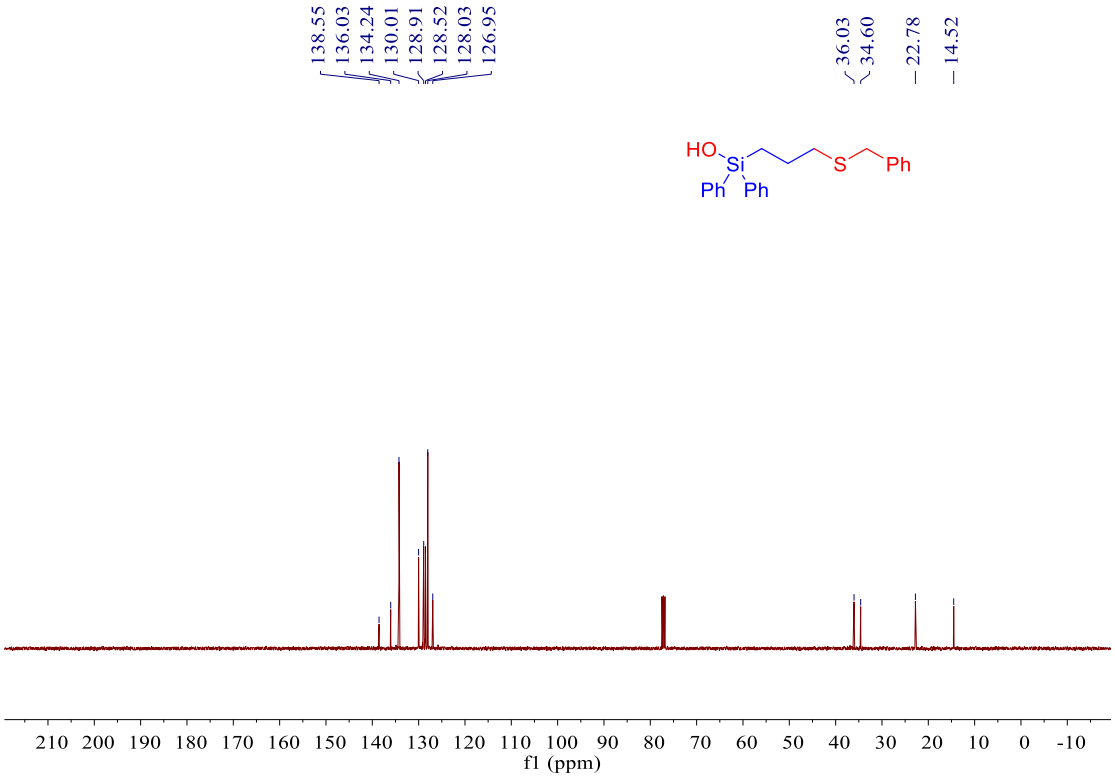
^{77}Se NMR Spectra of **3t** (100 MHz, $CDCl_3$)



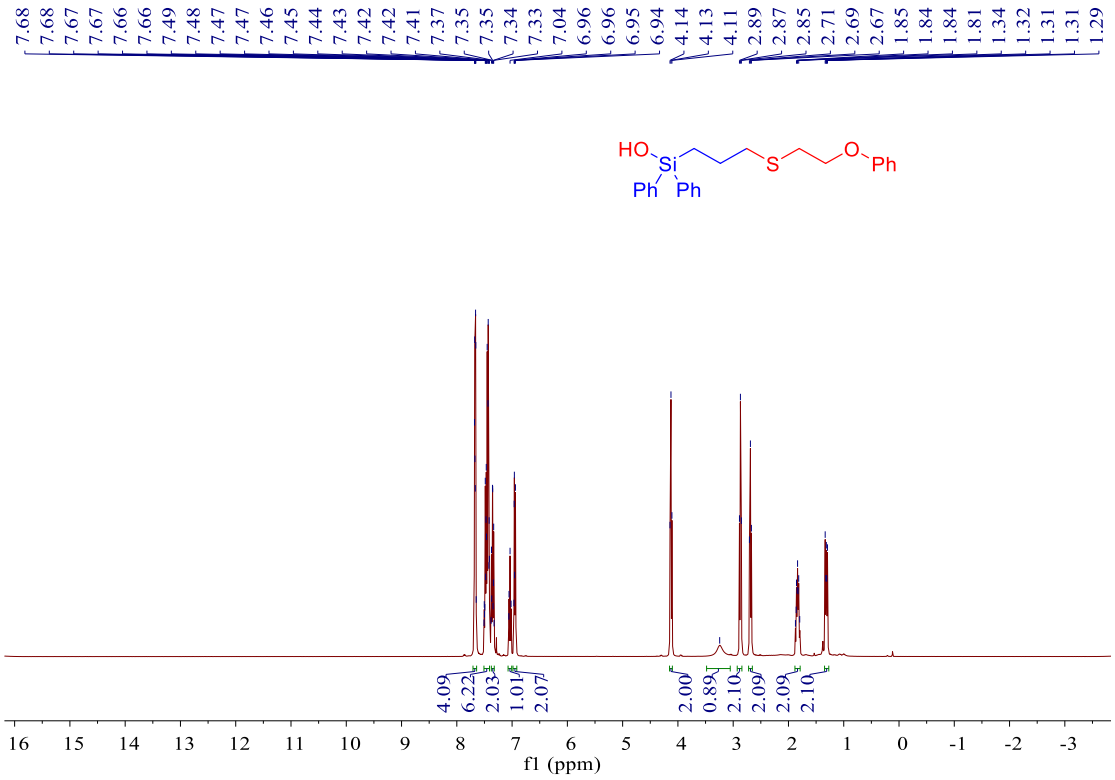
^{1}H NMR Spectra of **5a** (400 MHz, $CDCl_3$)



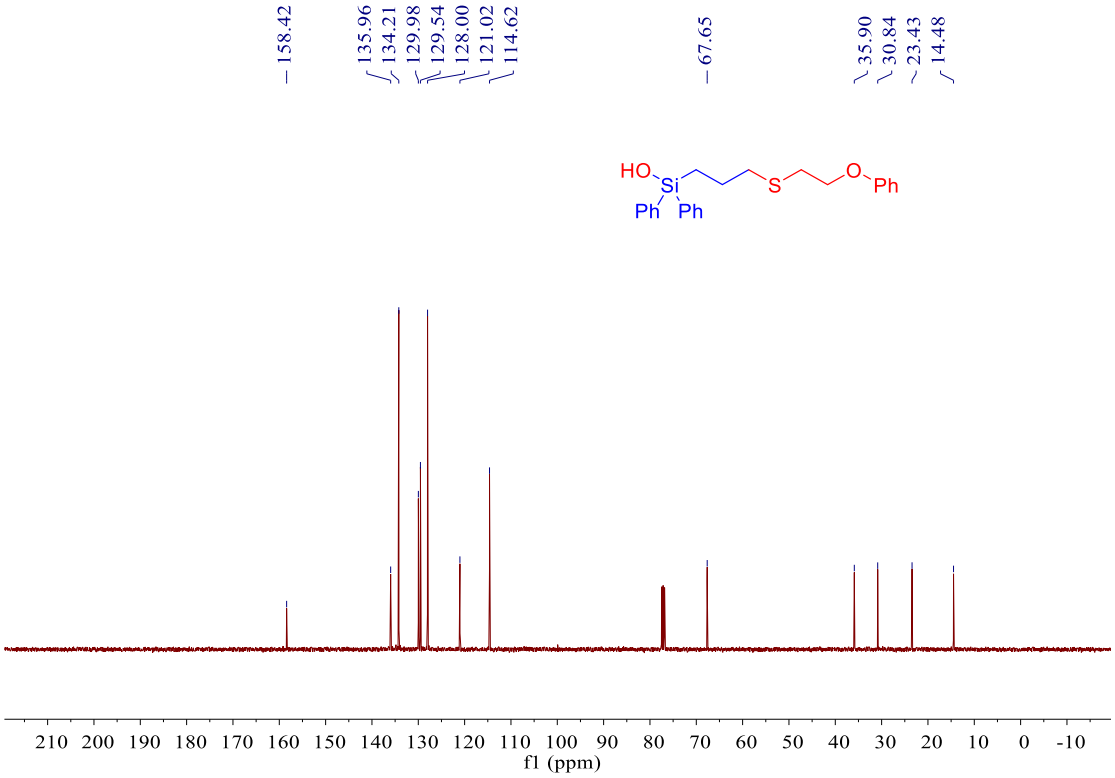
^{13}C NMR Spectra of **5a** (100 MHz, $CDCl_3$)



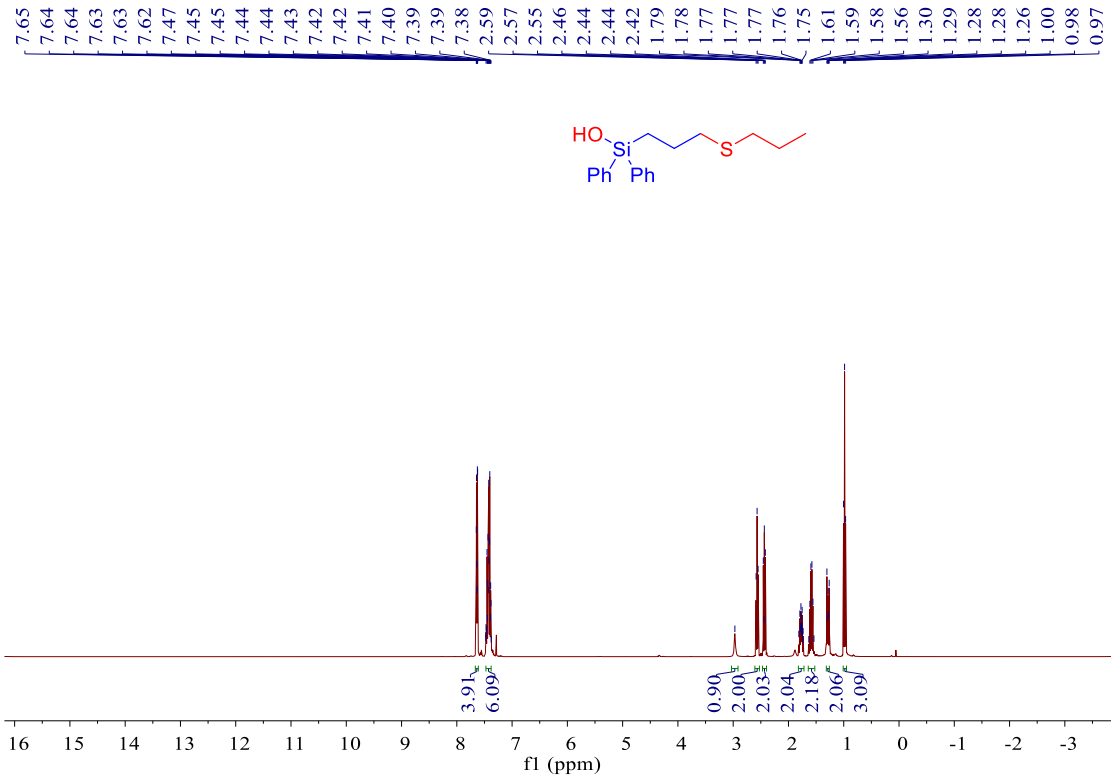
^{1}H NMR Spectra of **5b** (400 MHz, $CDCl_3$)



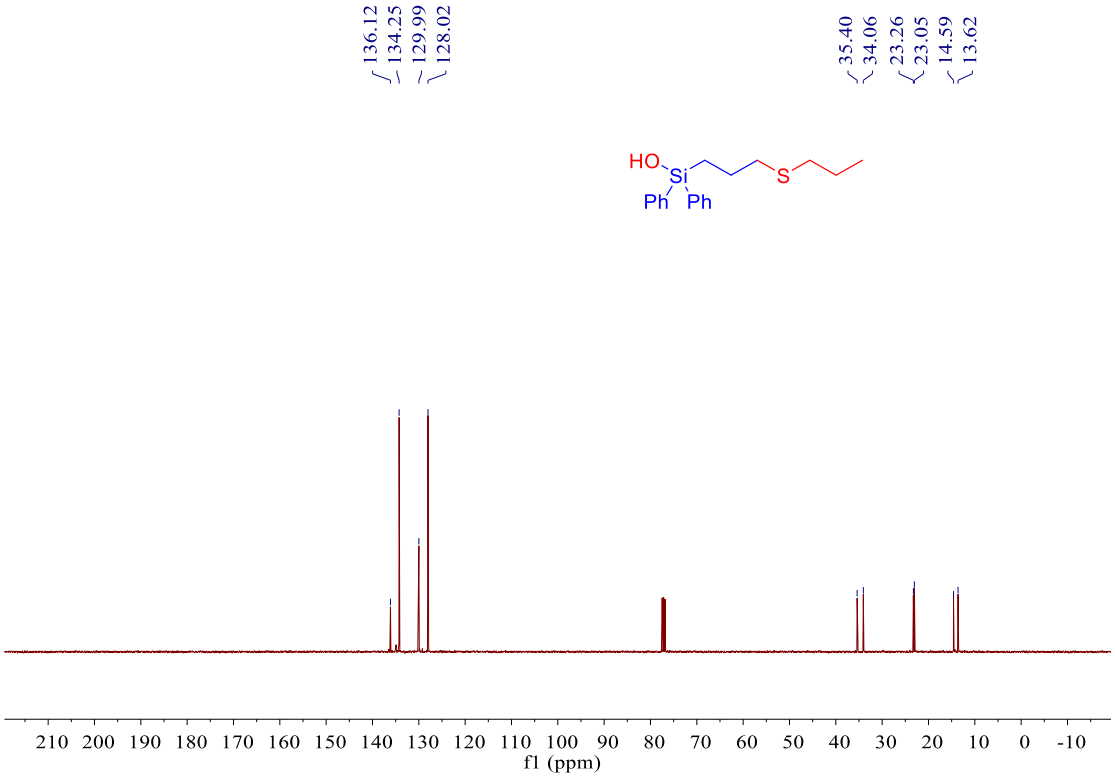
^{13}C NMR Spectra of **5b** (100 MHz, $CDCl_3$)



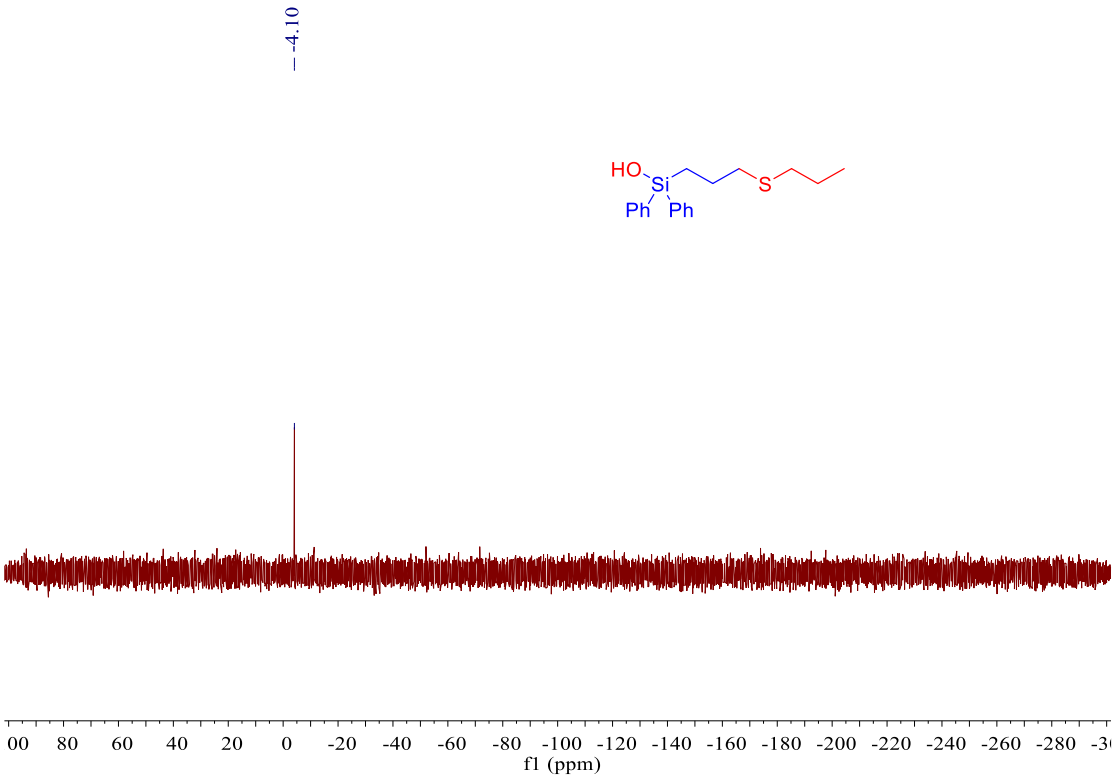
^{1}H NMR Spectra of **5c** (400 MHz, $CDCl_3$)



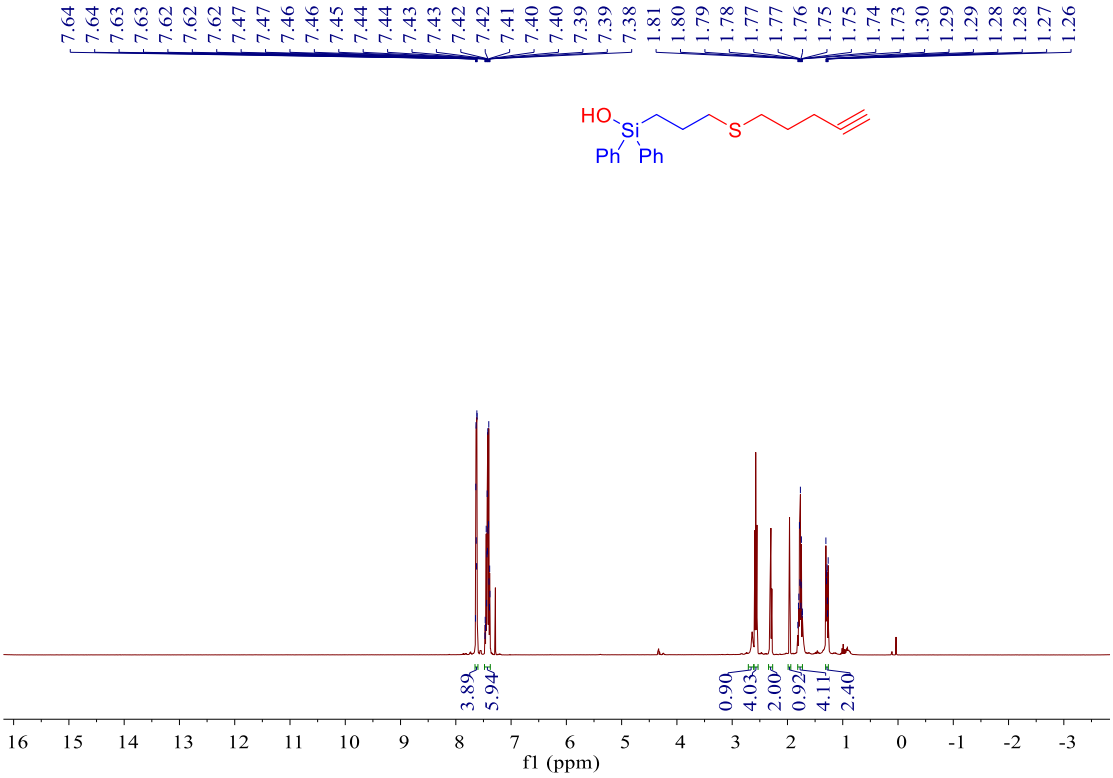
^{13}C NMR Spectra of **5c** (100 MHz, $CDCl_3$)



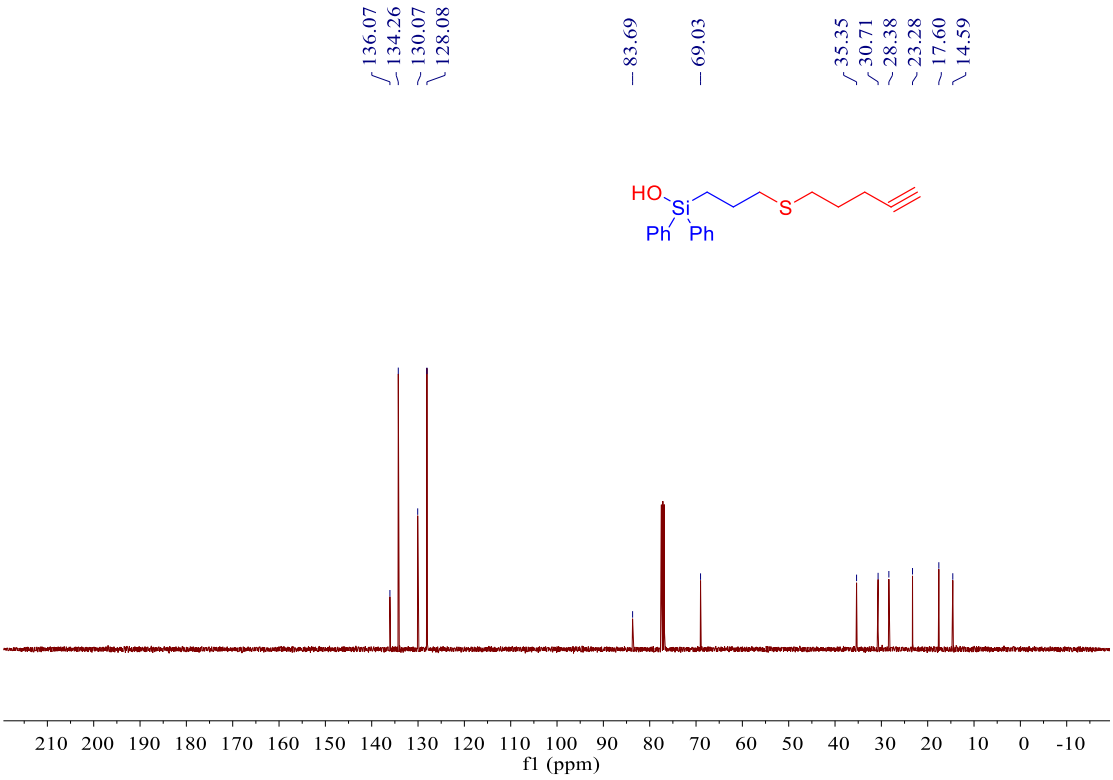
^{29}Si NMR Spectra of **5c** (79 MHz, $CDCl_3$)



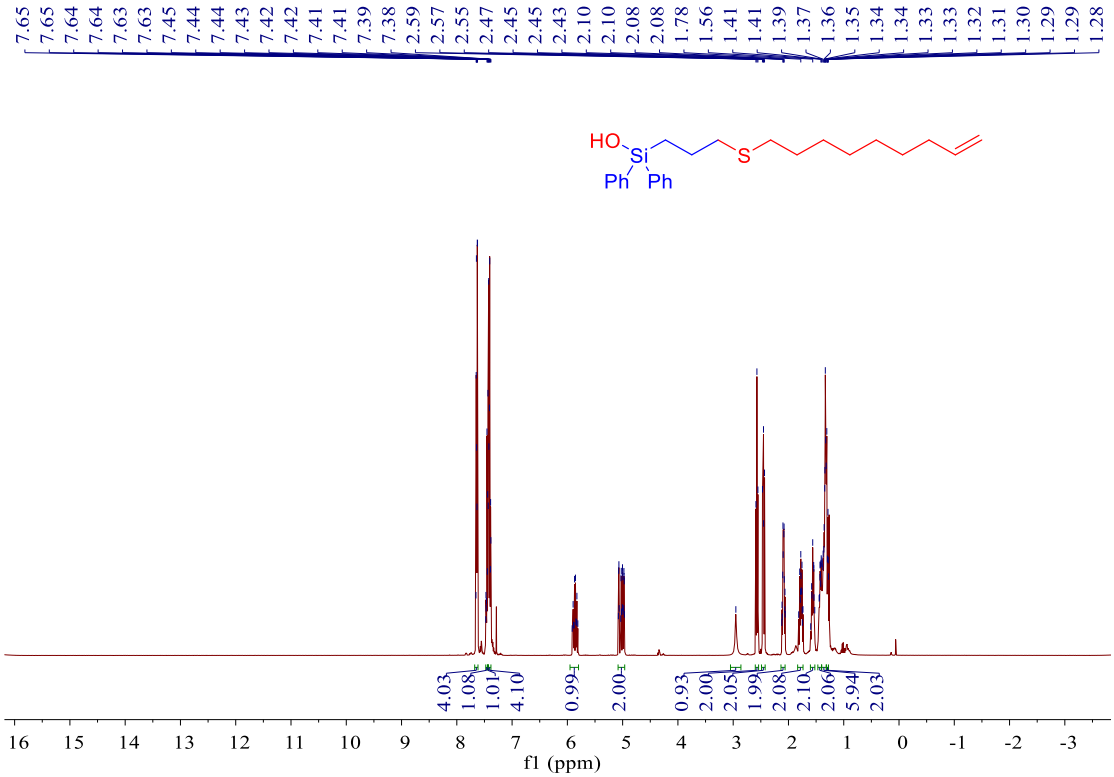
^{1}H NMR Spectra of **5d** (400 MHz, $CDCl_3$)



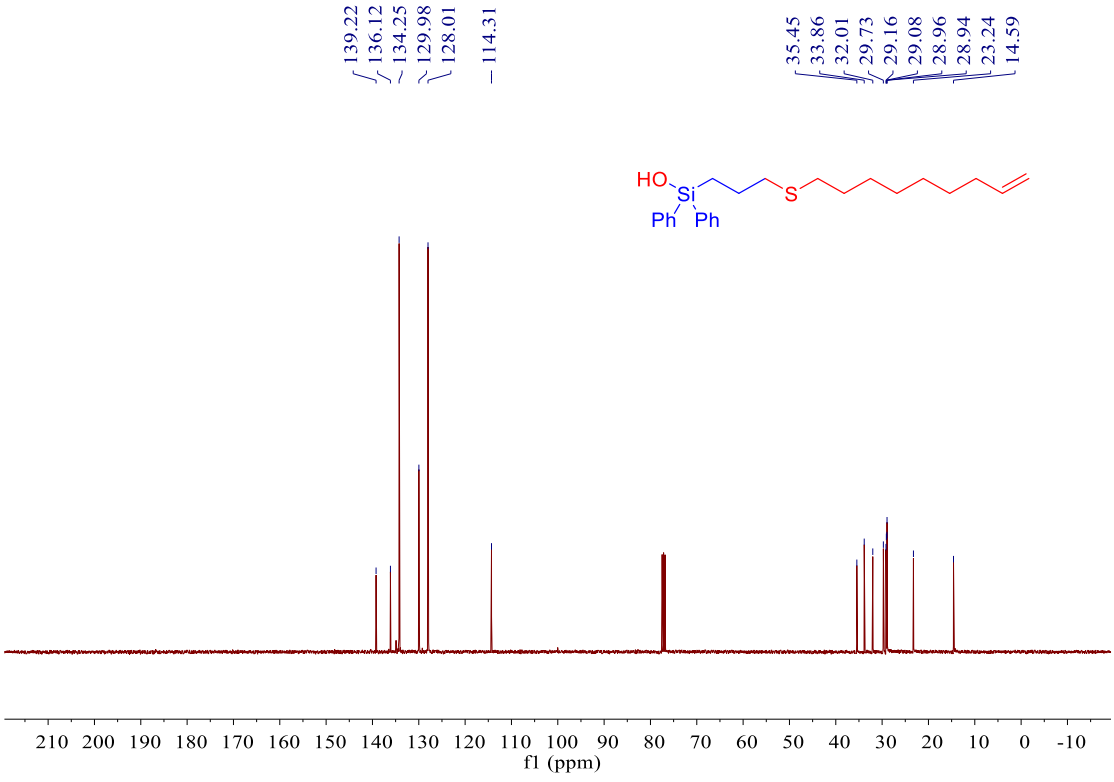
^{13}C NMR Spectra of **5d** (100 MHz, $CDCl_3$)



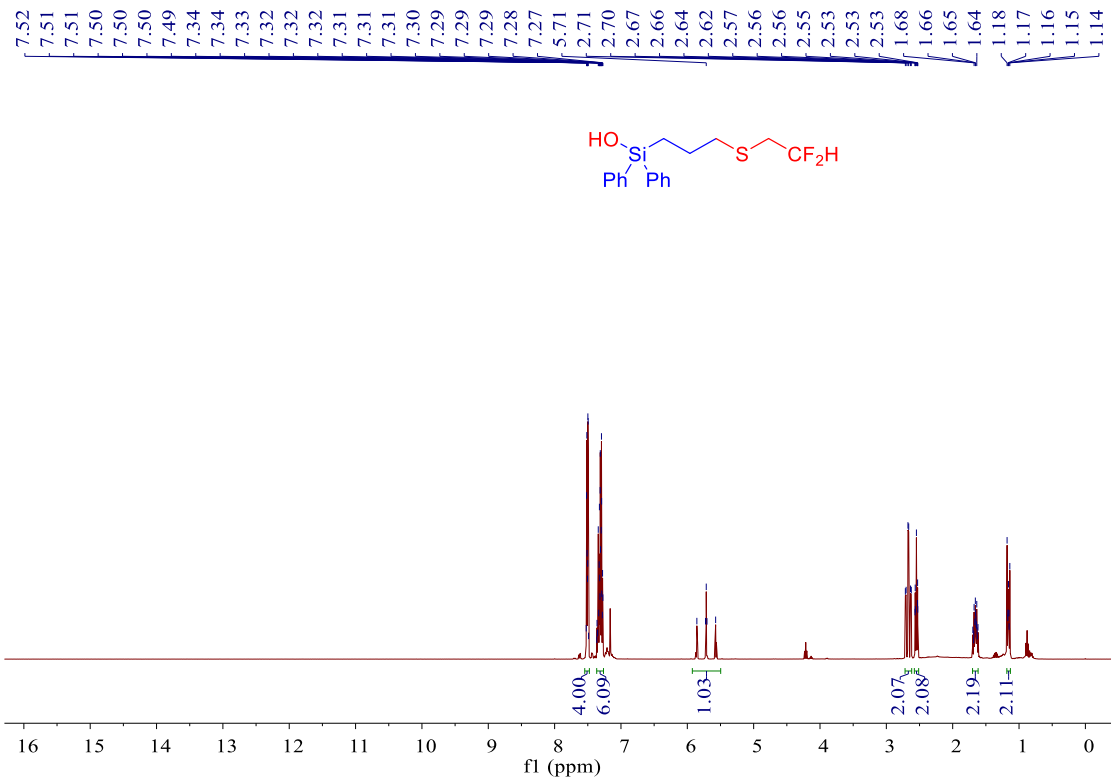
^{1}H NMR Spectra of **5e** (400 MHz, $CDCl_3$)



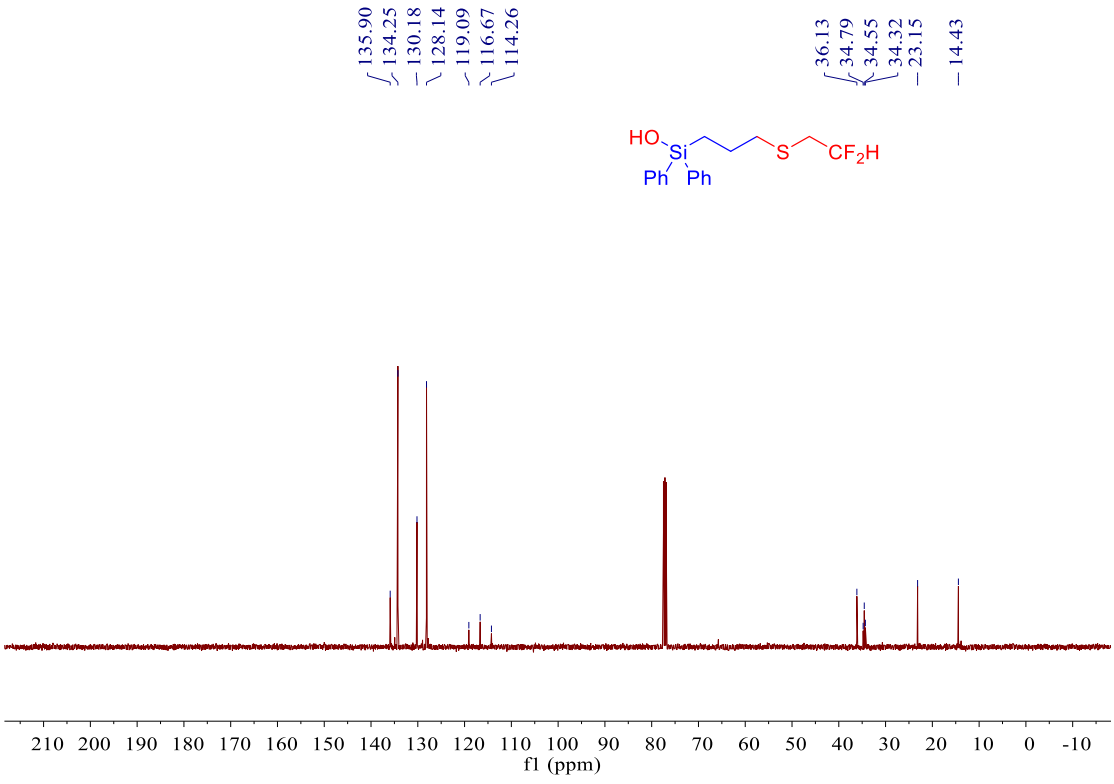
^{13}C NMR Spectra of **5e** (100 MHz, $CDCl_3$)



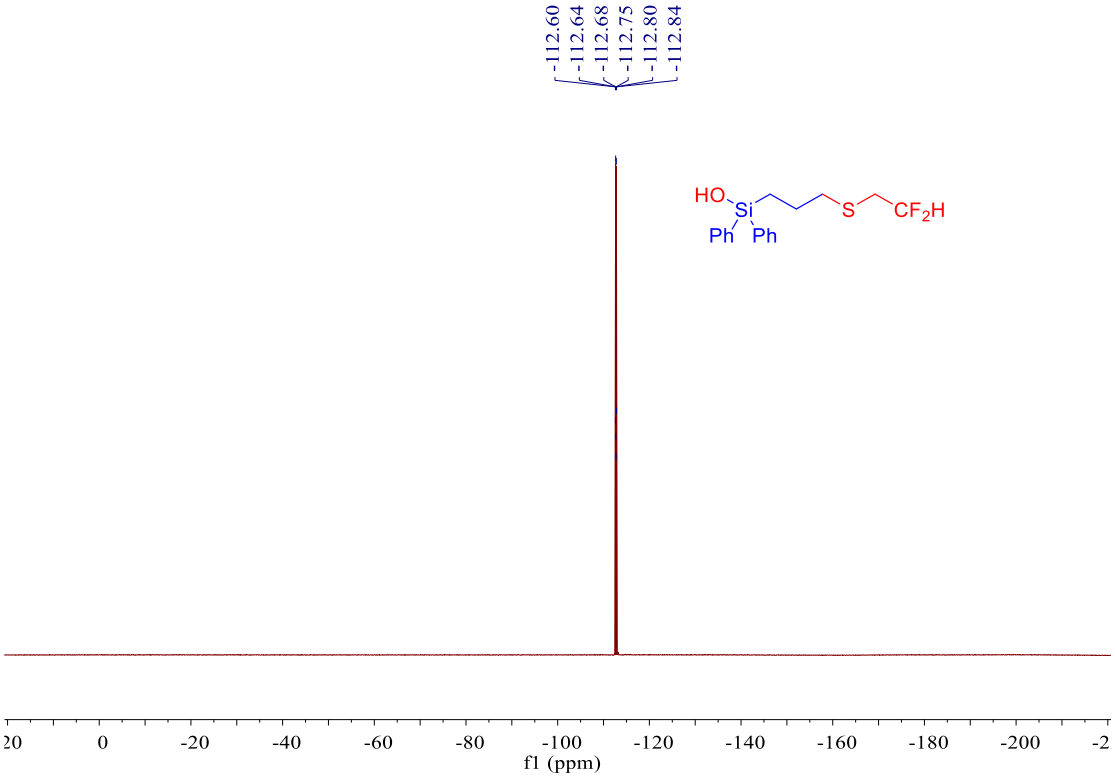
^{1}H NMR Spectra of **5f** (400 MHz, $CDCl_3$)



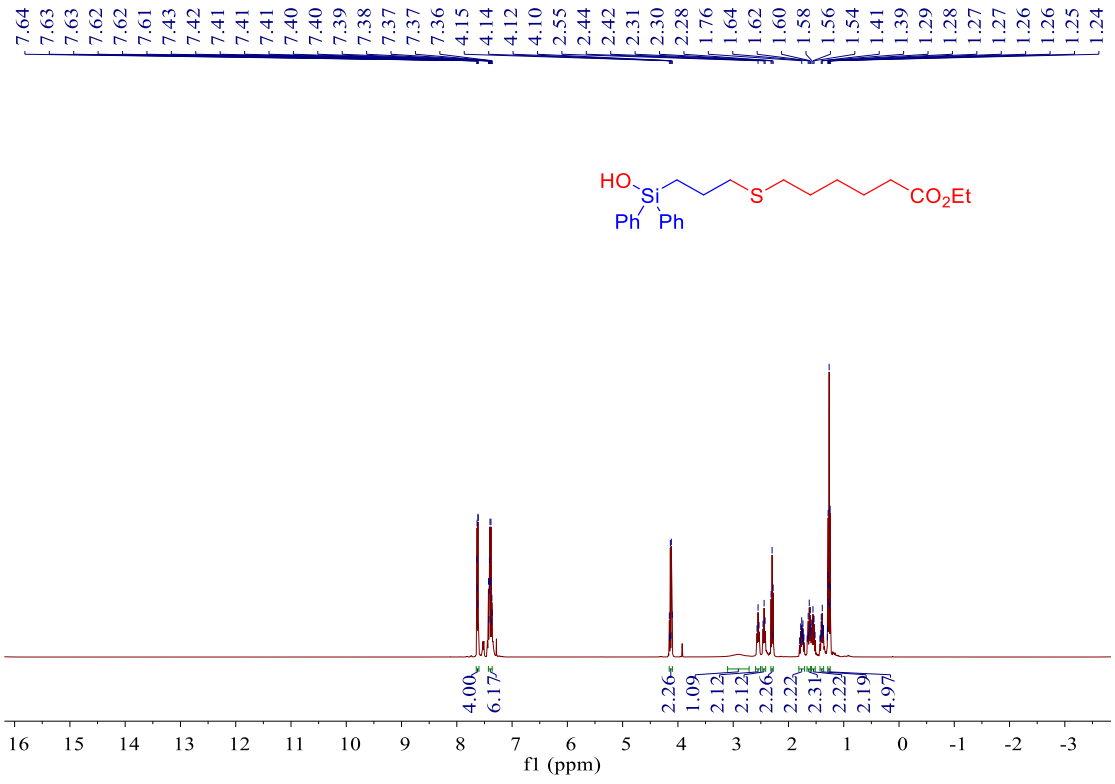
^{13}C NMR Spectra of **5f** (100 MHz, $CDCl_3$)



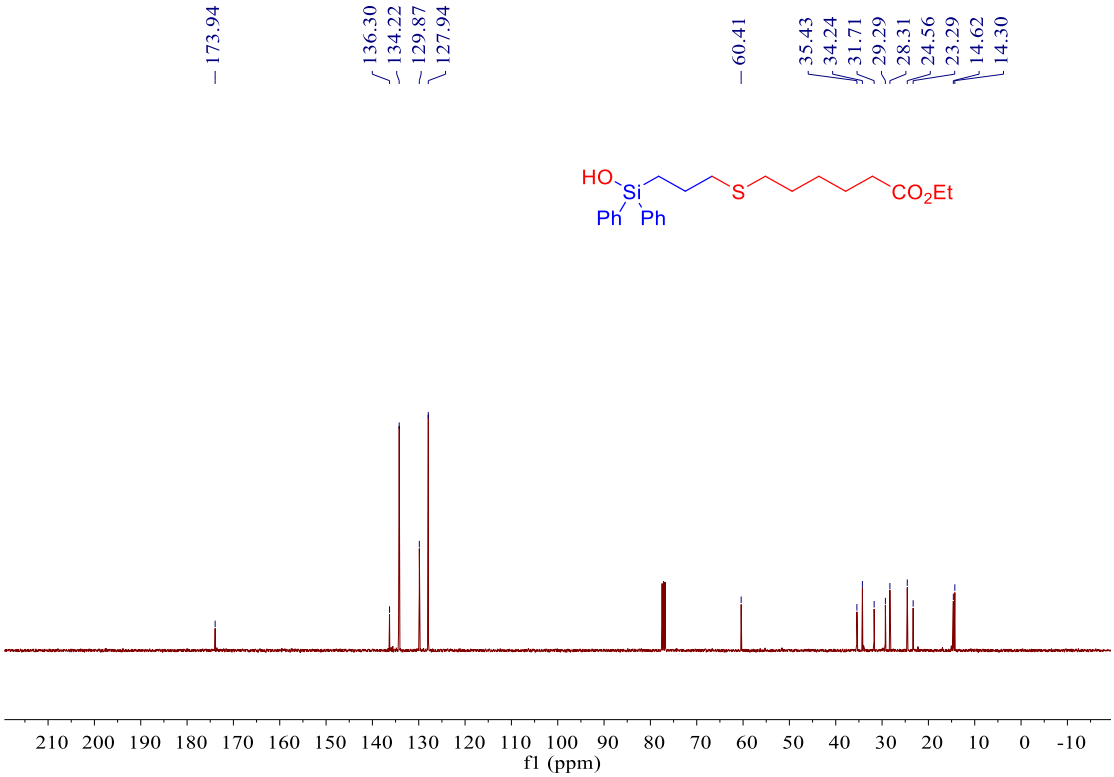
^{19}F NMR Spectra of **5f** (100 MHz, $CDCl_3$)



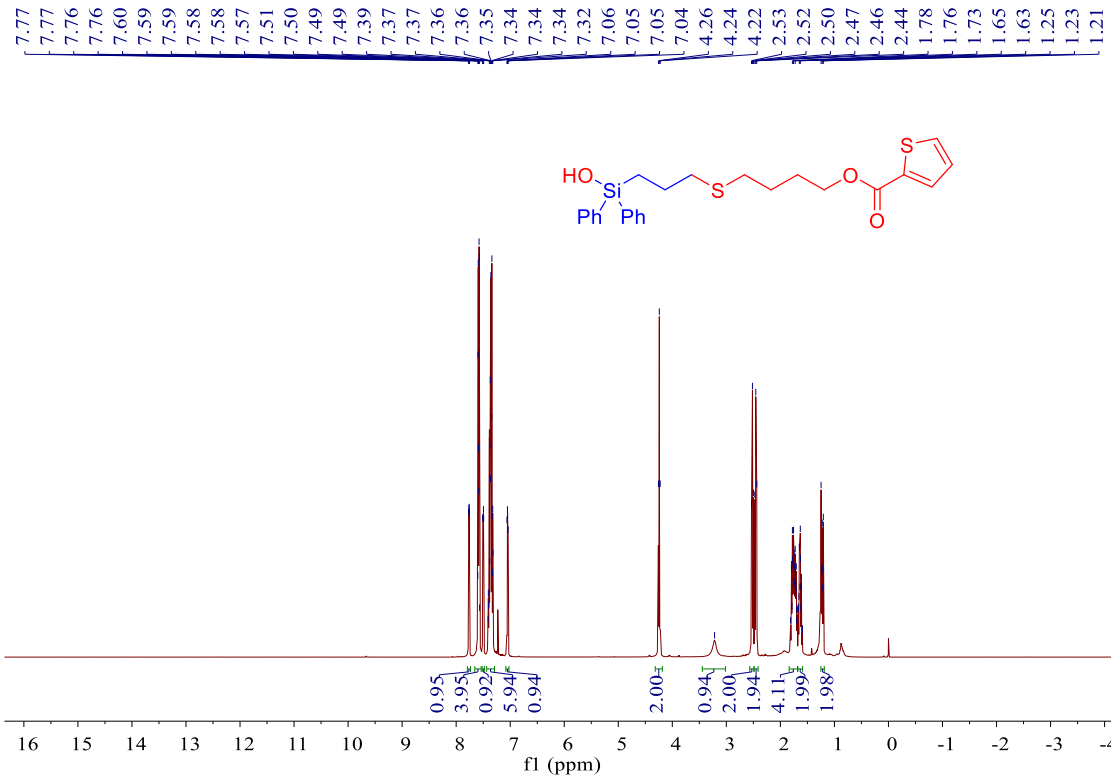
^{1}H NMR Spectra of **5g** (400 MHz, $CDCl_3$)



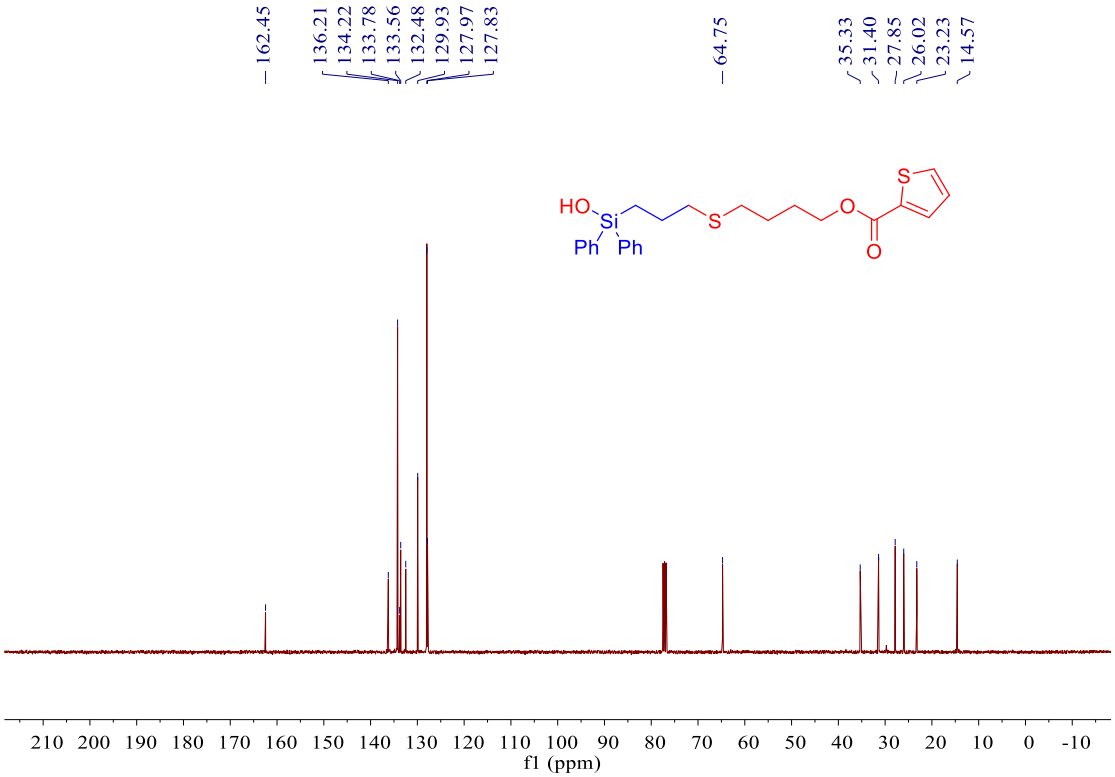
^{13}C NMR Spectra of **5g** (100 MHz, $CDCl_3$)



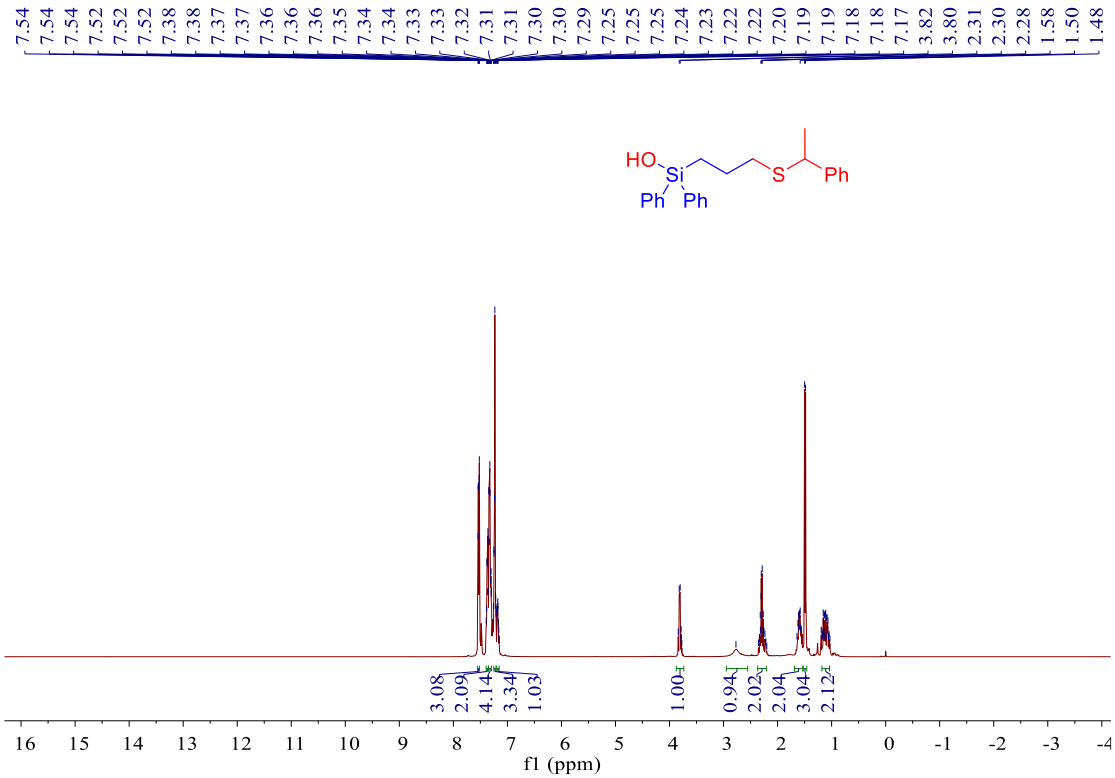
^{1}H NMR Spectra of **5h** (400 MHz, $CDCl_3$)



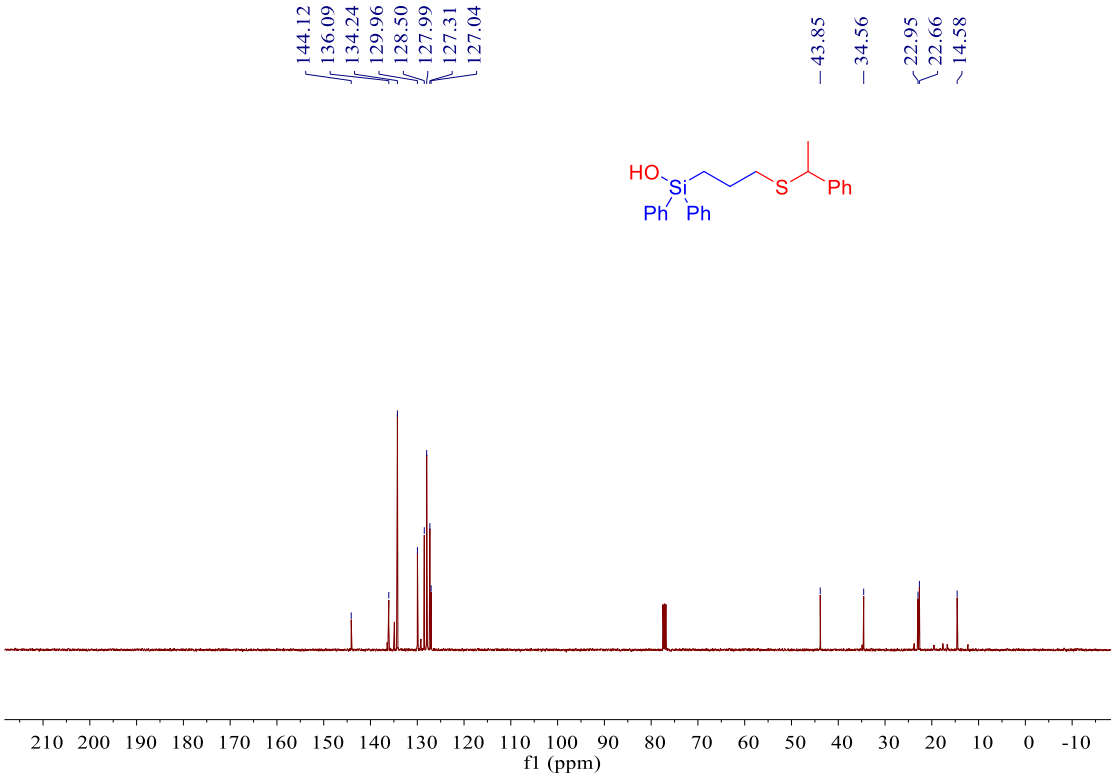
^{13}C NMR Spectra of **5h** (100 MHz, $CDCl_3$)



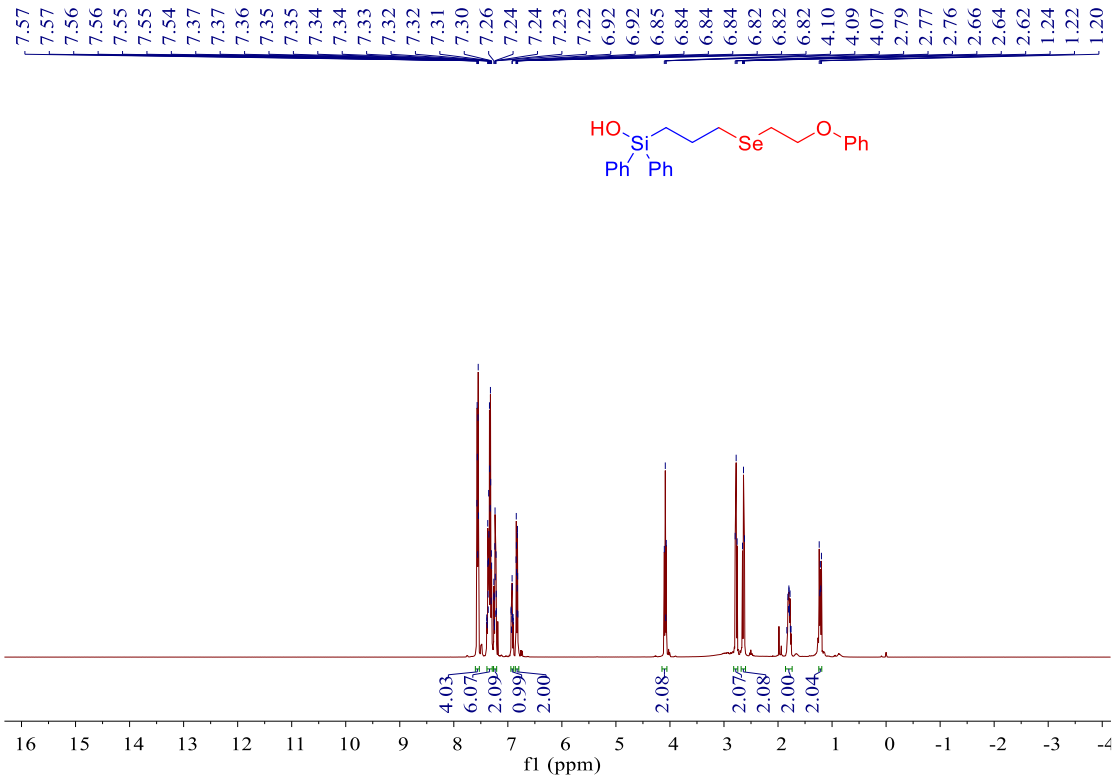
^{1}H NMR Spectra of **5j** (400 MHz, $CDCl_3$)



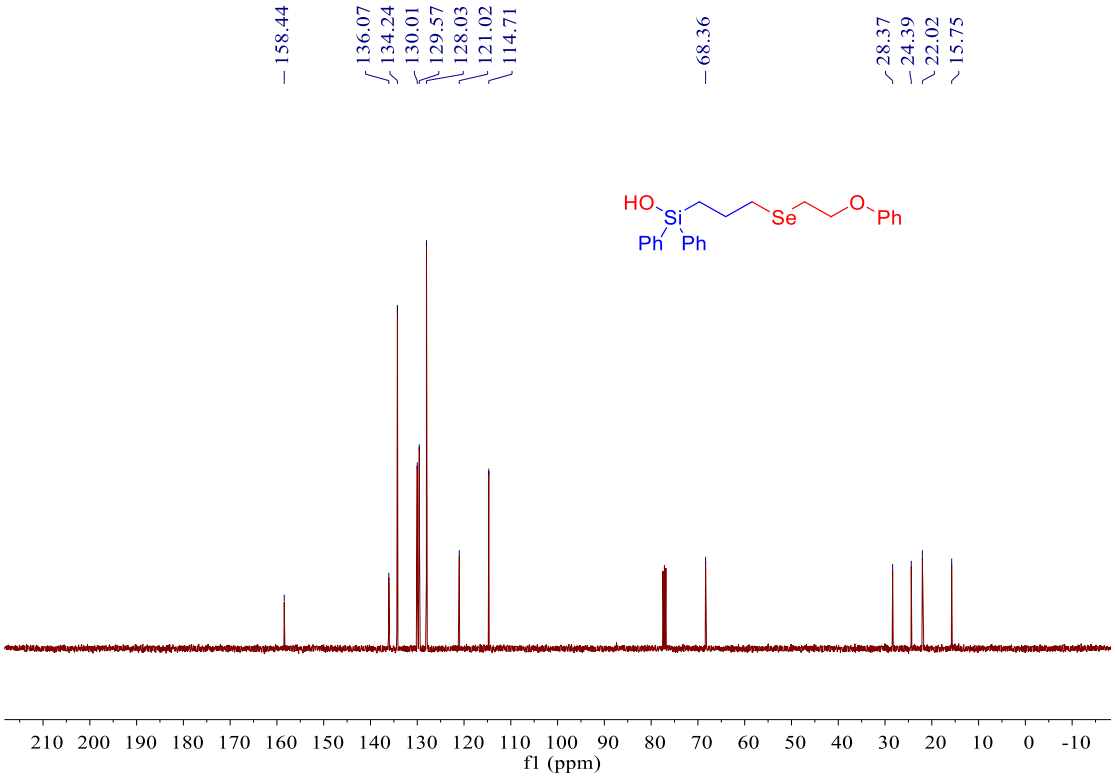
^{13}C NMR Spectra of **5j** (100 MHz, $CDCl_3$)



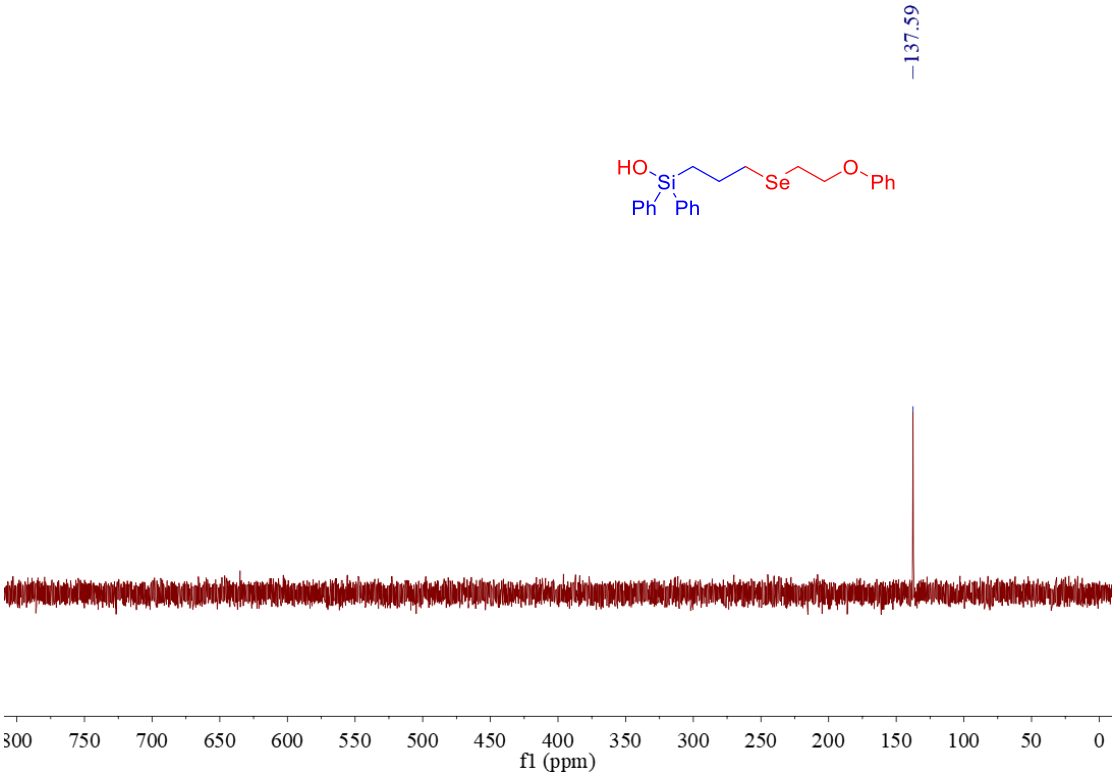
^{1}H NMR Spectra of **5m** (400 MHz, $CDCl_3$)



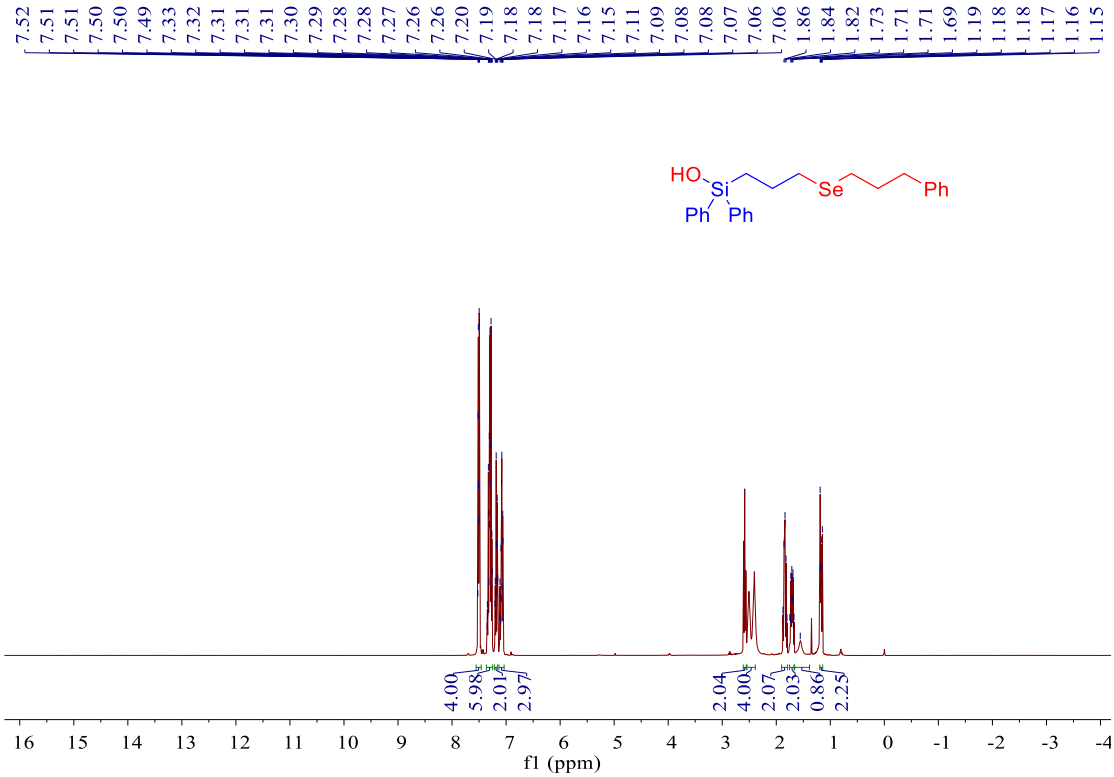
^{13}C NMR Spectra of **5m** (100 MHz, $CDCl_3$)



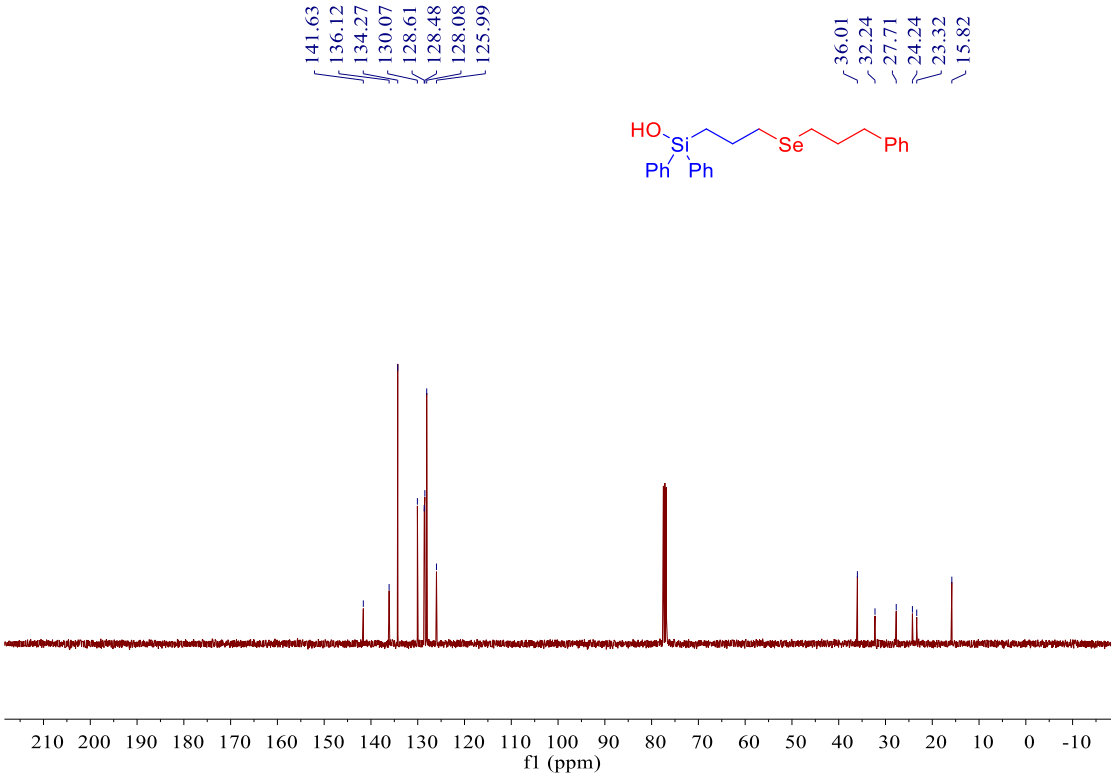
^{77}Se NMR Spectra of **5m** (100 MHz, $CDCl_3$)



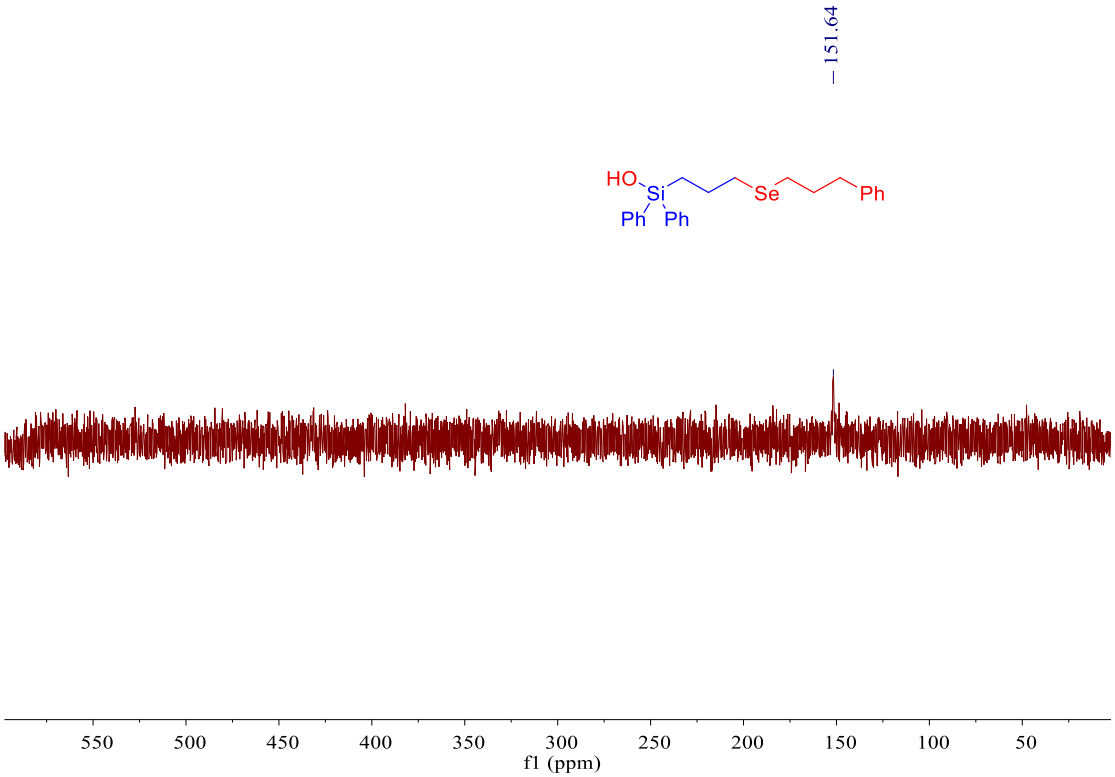
^{1}H NMR Spectra of **5n** (400 MHz, $CDCl_3$)



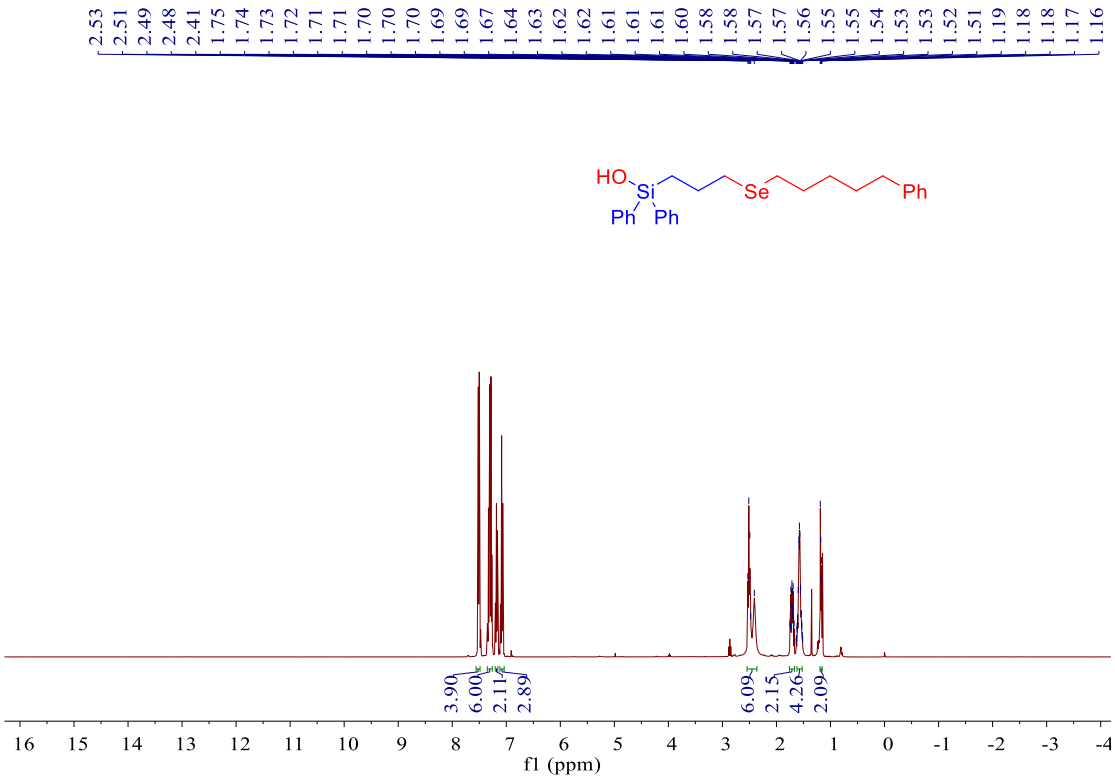
^{13}C NMR Spectra of **5n** (100 MHz, $CDCl_3$)



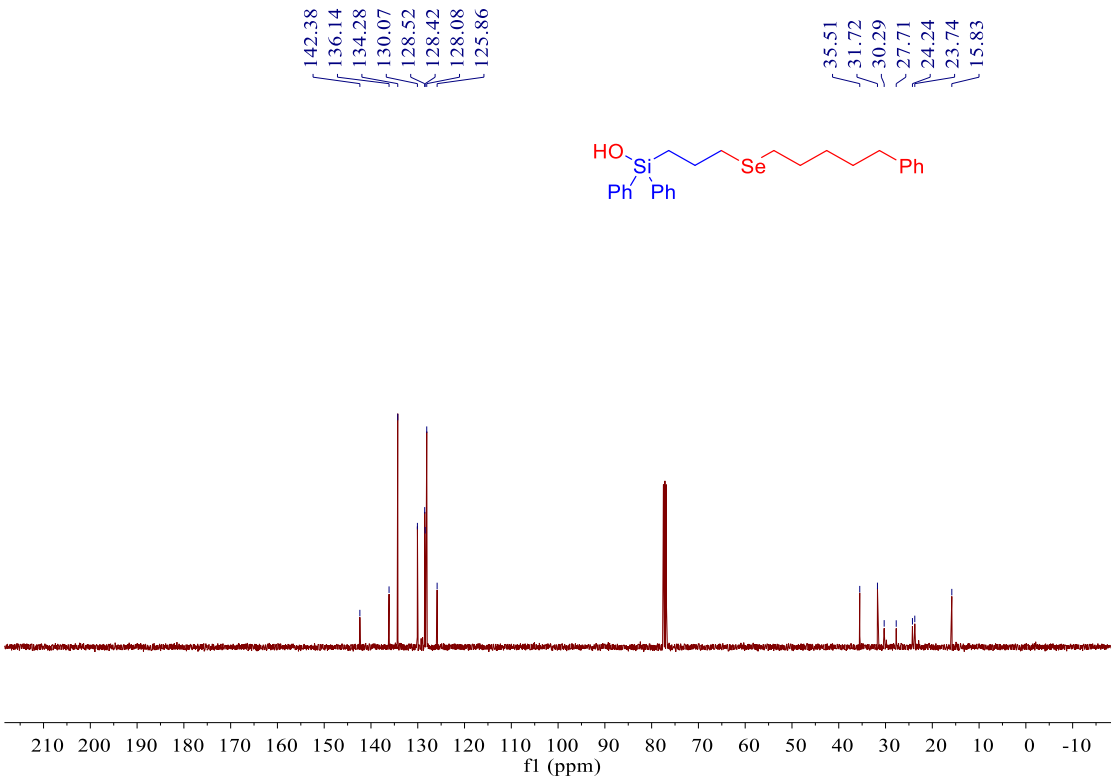
^{77}Se NMR Spectra of **5n** (100 MHz, $CDCl_3$)



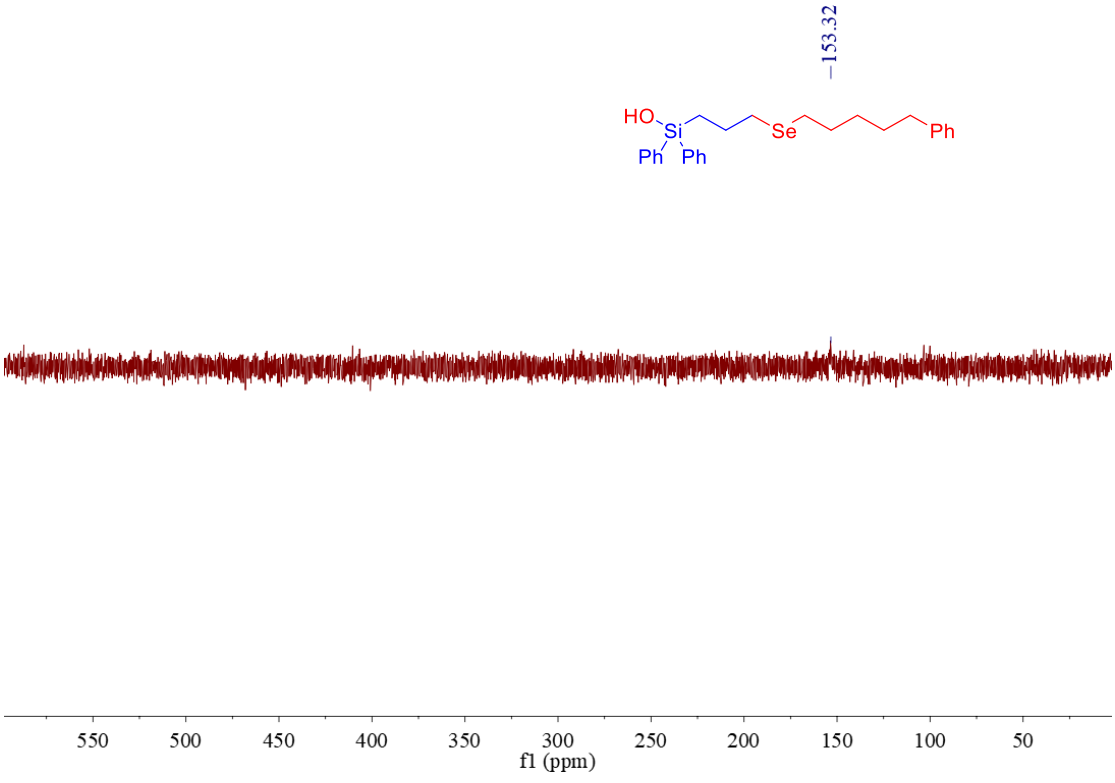
^{1}H NMR Spectra of **5o** (400 MHz, $CDCl_3$)



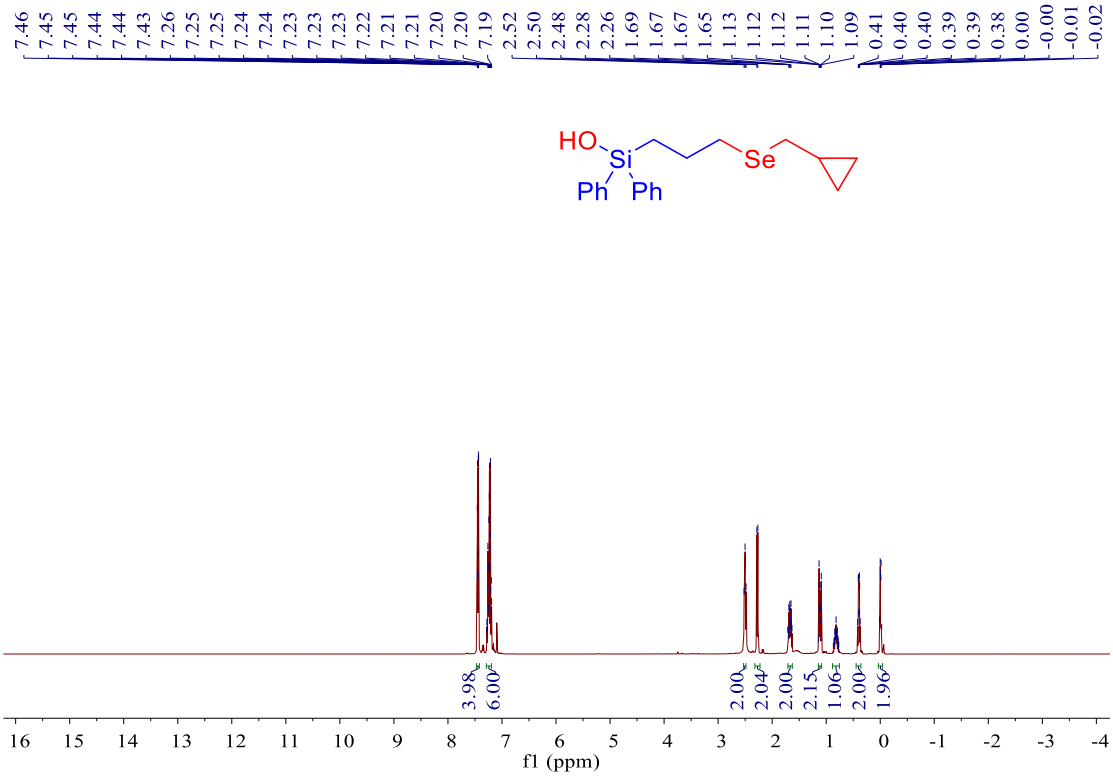
^{13}C NMR Spectra of **5o** (100 MHz, $CDCl_3$)



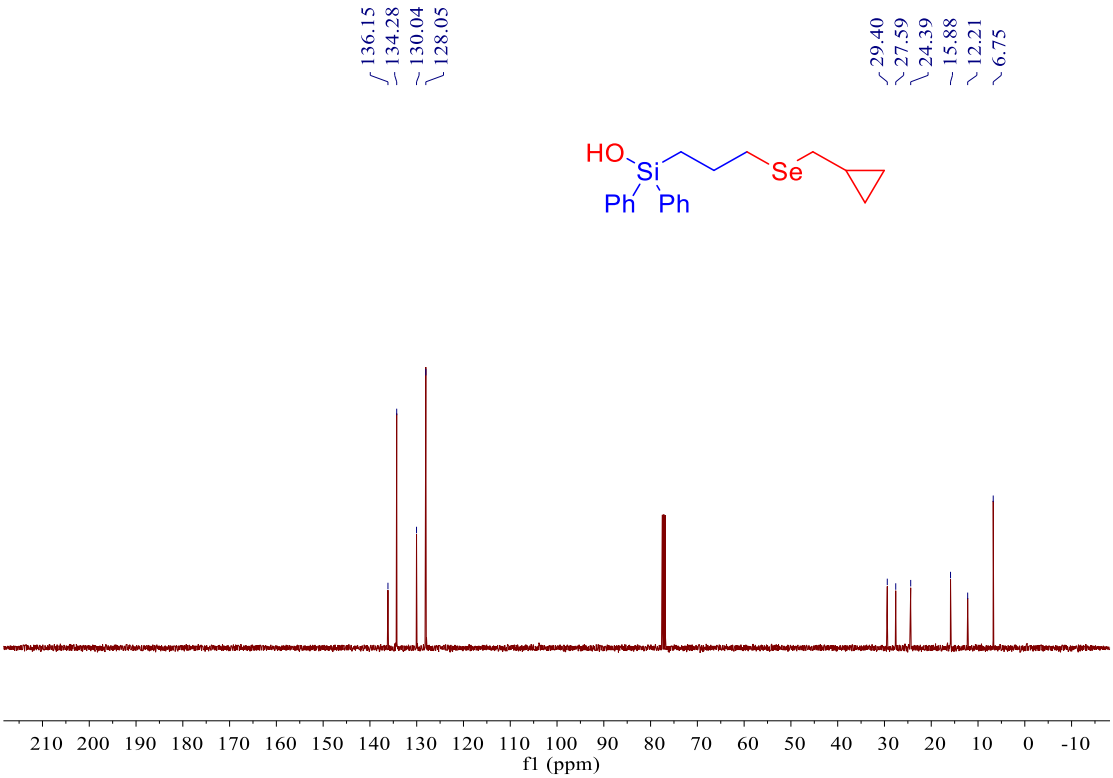
^{77}Se NMR Spectra of **5o** (100 MHz, $CDCl_3$)



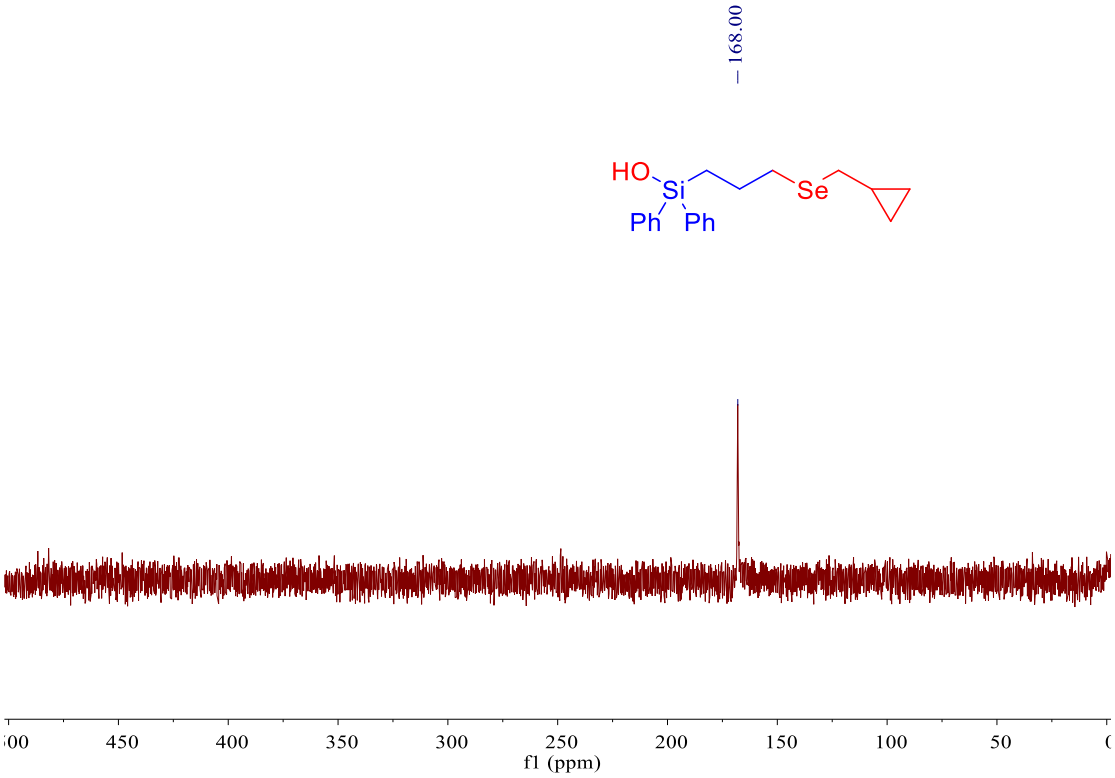
^{1}H NMR Spectra of **5p** (400 MHz, $CDCl_3$)



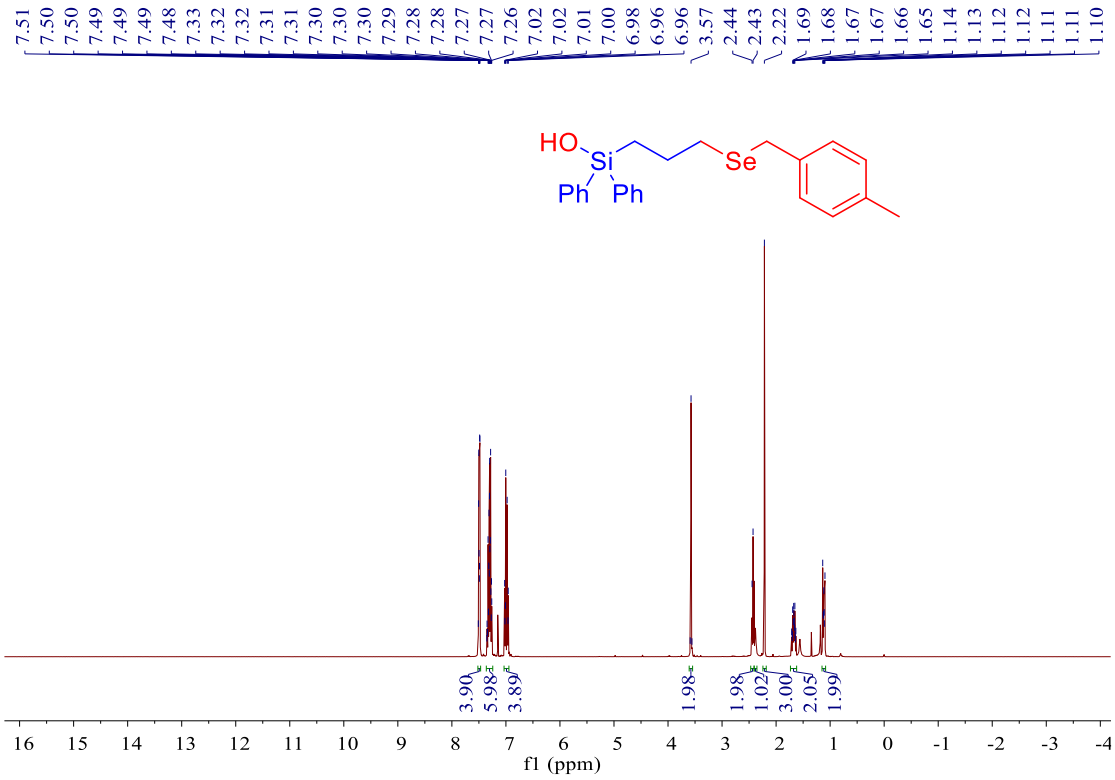
^{13}C NMR Spectra of **5p** (100 MHz, $CDCl_3$)



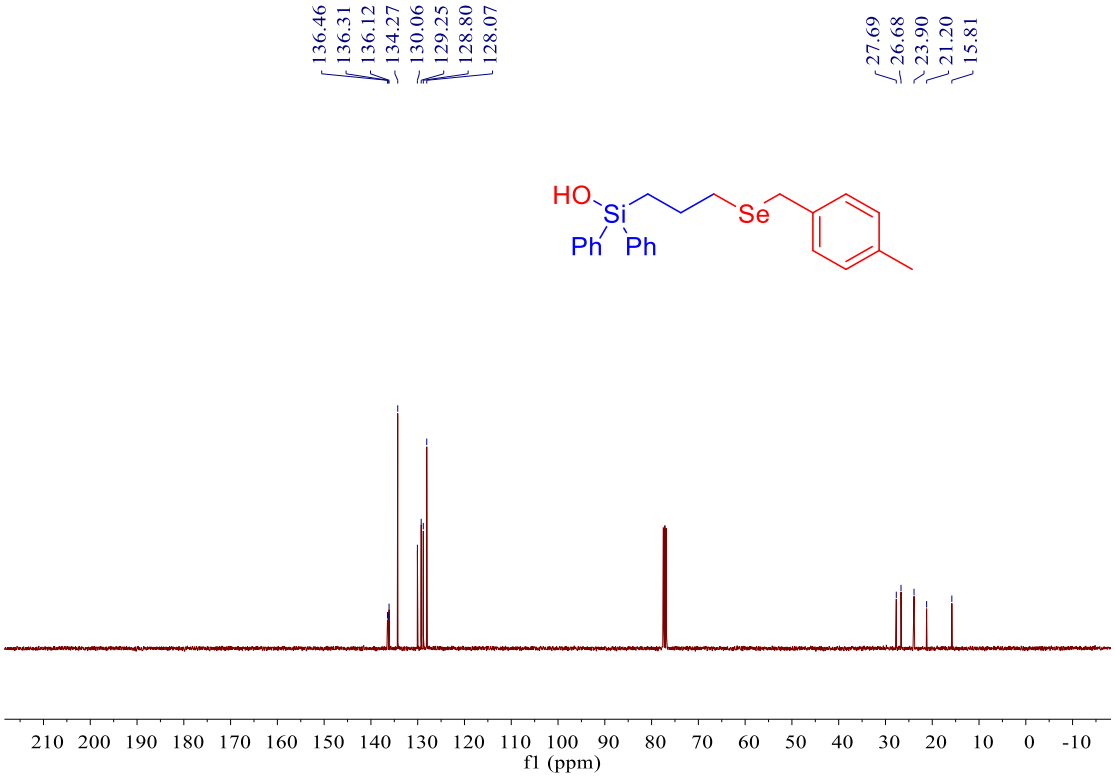
^{77}Se NMR Spectra of **5p** (100 MHz, $CDCl_3$)



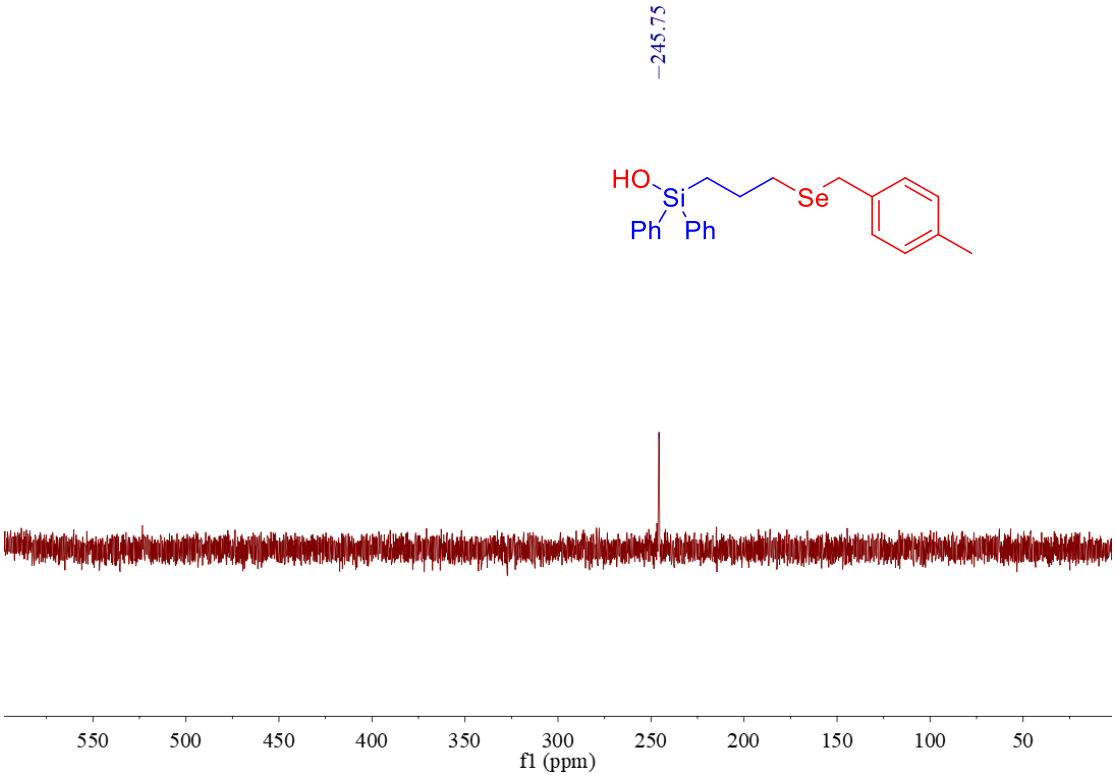
^{1}H NMR Spectra of **5q** (400 MHz, $CDCl_3$)



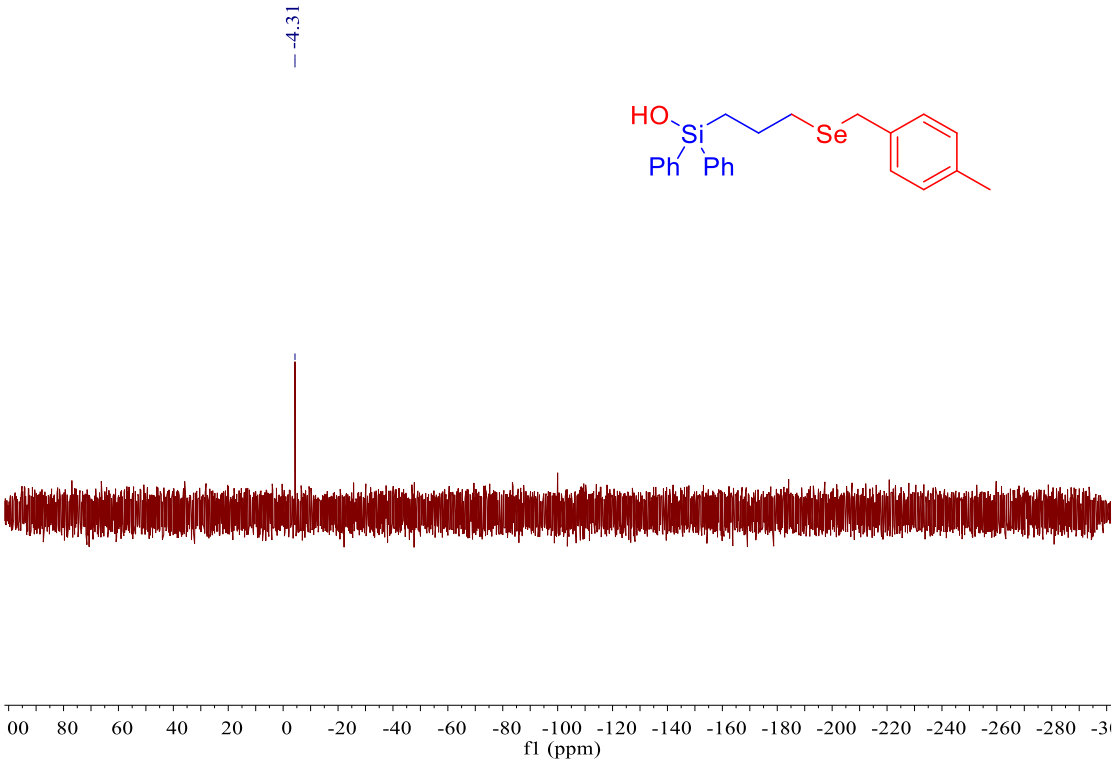
^{13}C NMR Spectra of **5q** (100 MHz, $CDCl_3$)



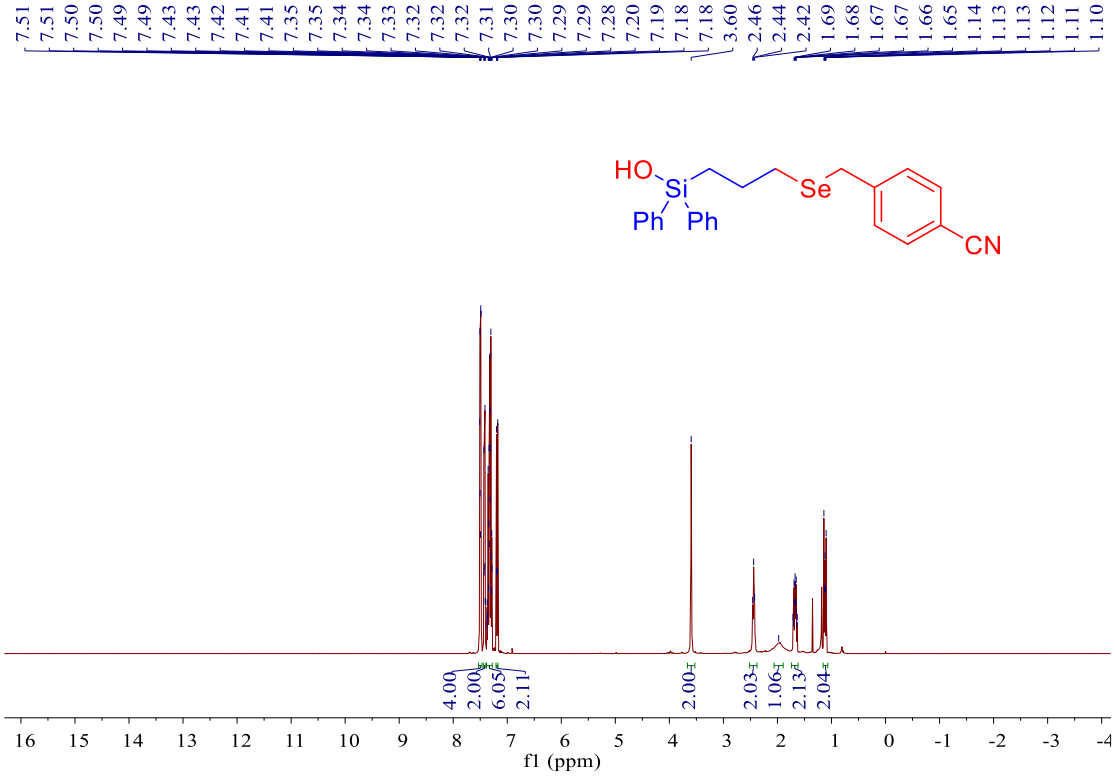
^{77}Se NMR Spectra of **5q** (100 MHz, $CDCl_3$)



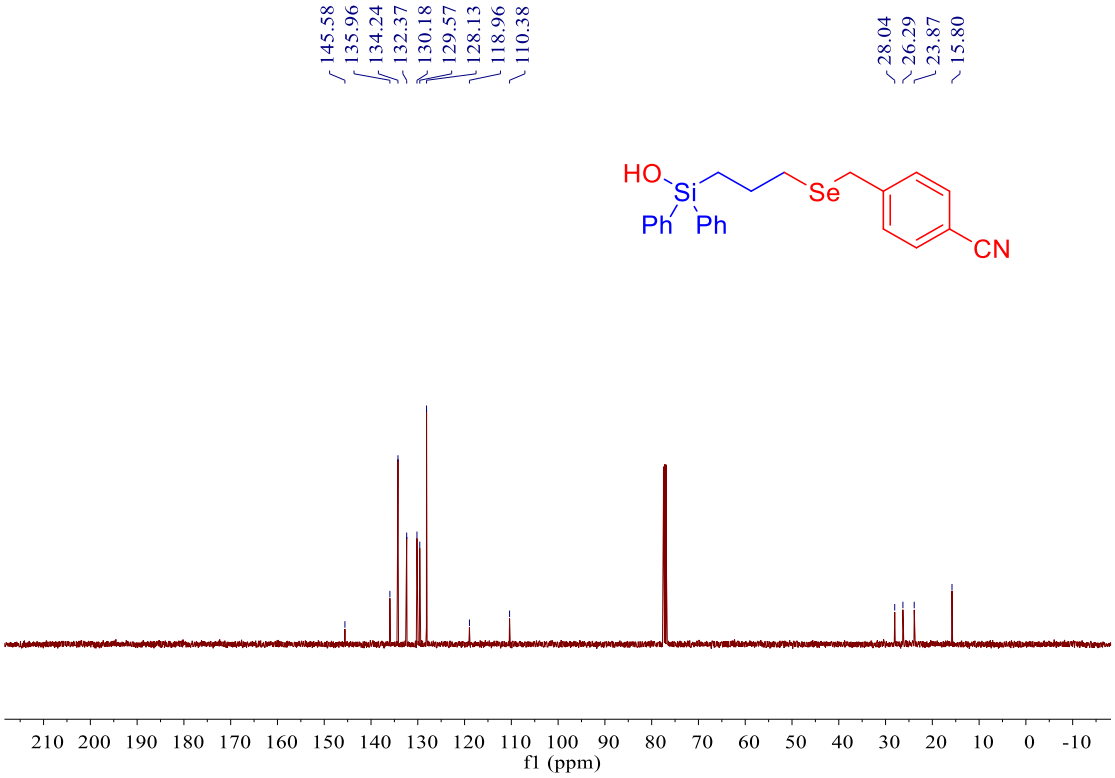
^{29}Si NMR Spectra of **5q** (79 MHz, $CDCl_3$)



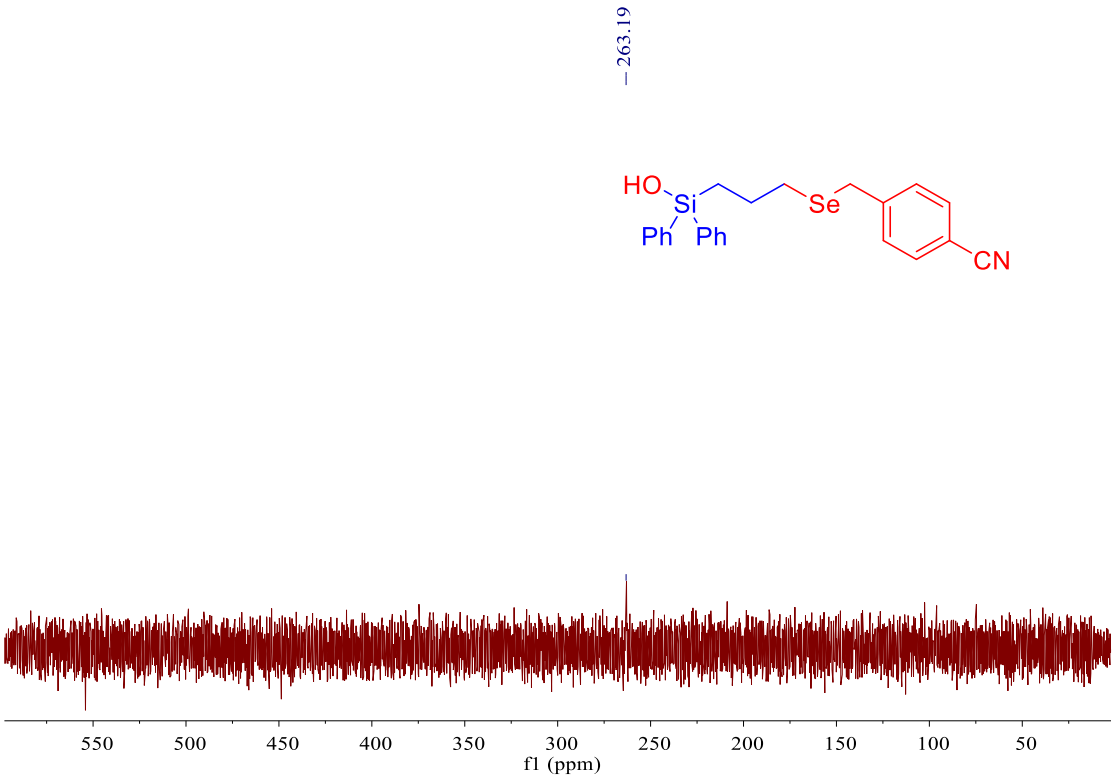
^{1}H NMR Spectra of **5r** (400 MHz, $CDCl_3$)



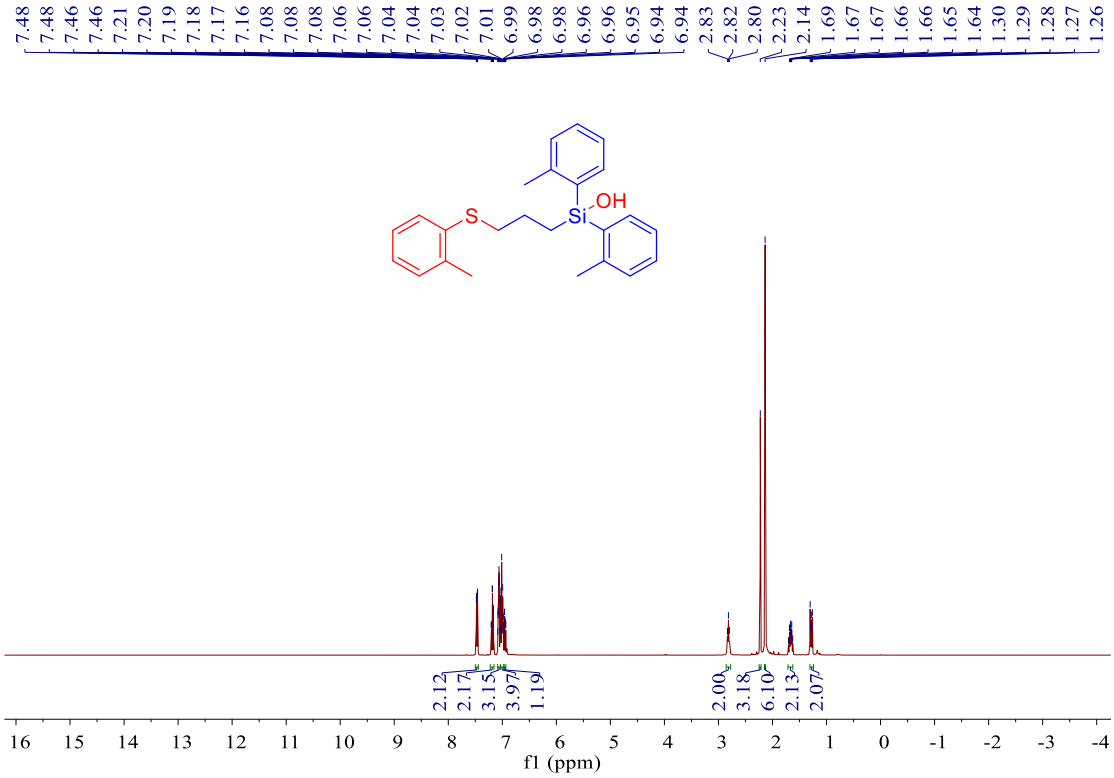
^{13}C NMR Spectra of **5r** (100 MHz, $CDCl_3$)



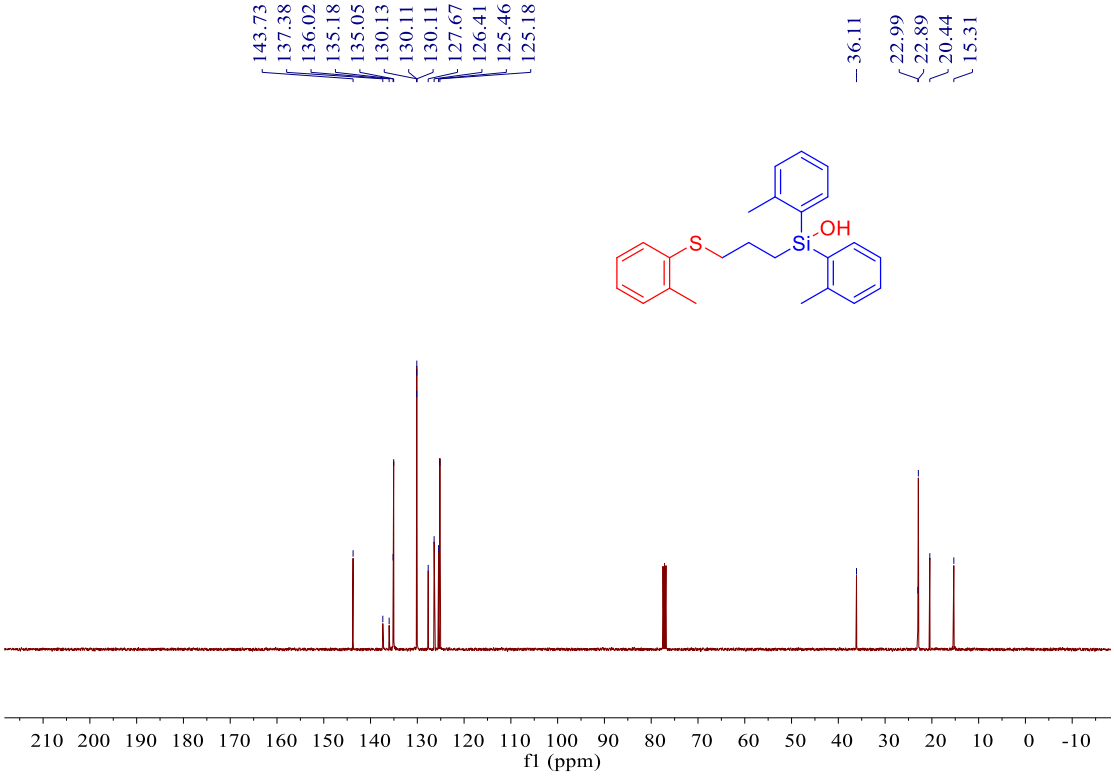
^{77}Se NMR Spectra of **5r** (100 MHz, $CDCl_3$)



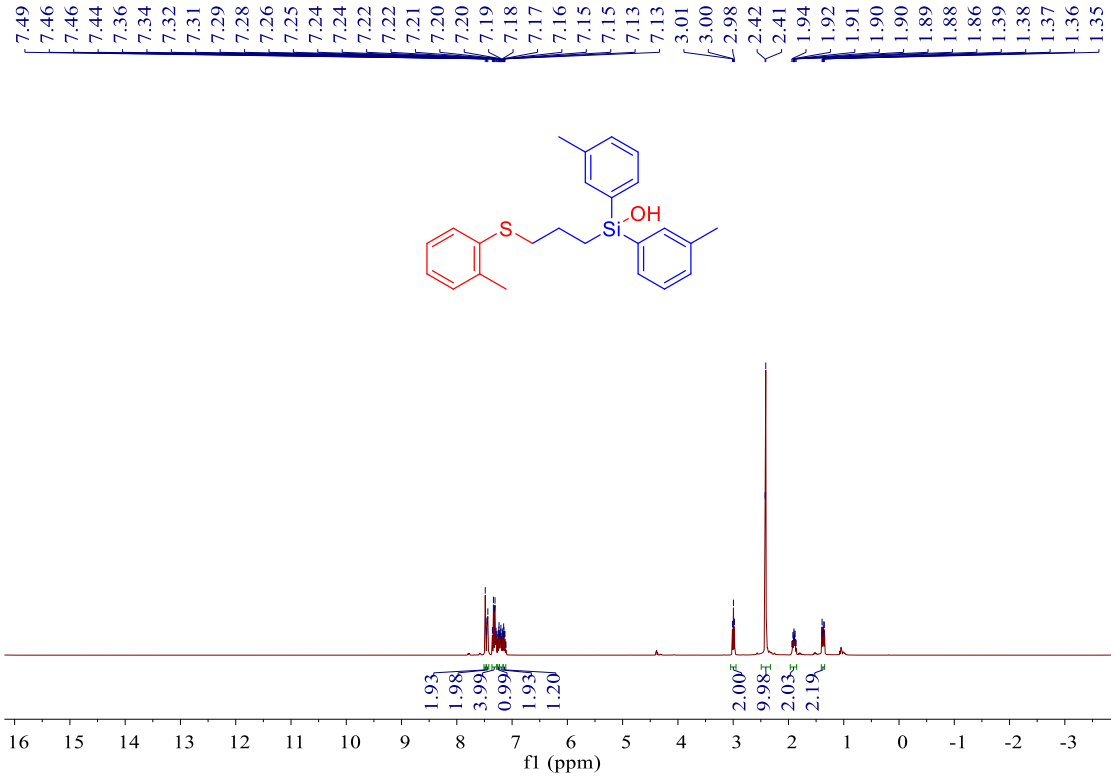
^{1}H NMR Spectra of **6a** (400 MHz, $CDCl_3$)



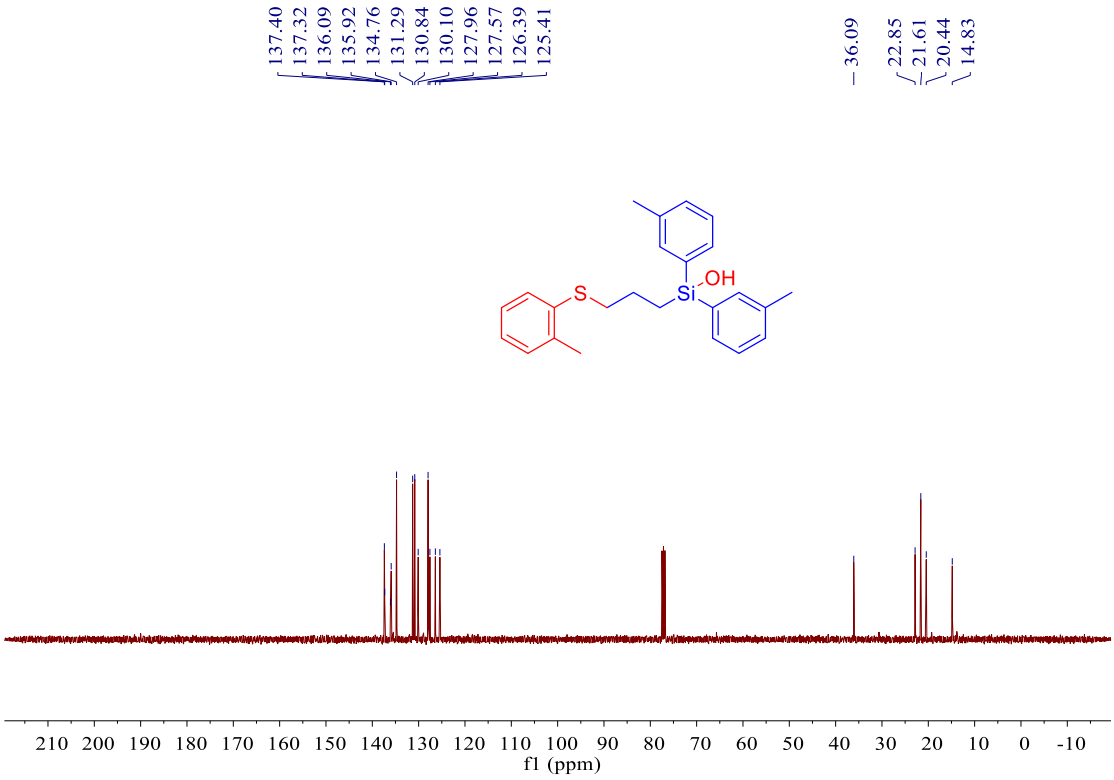
^{13}C NMR Spectra of **6a** (100 MHz, $CDCl_3$)



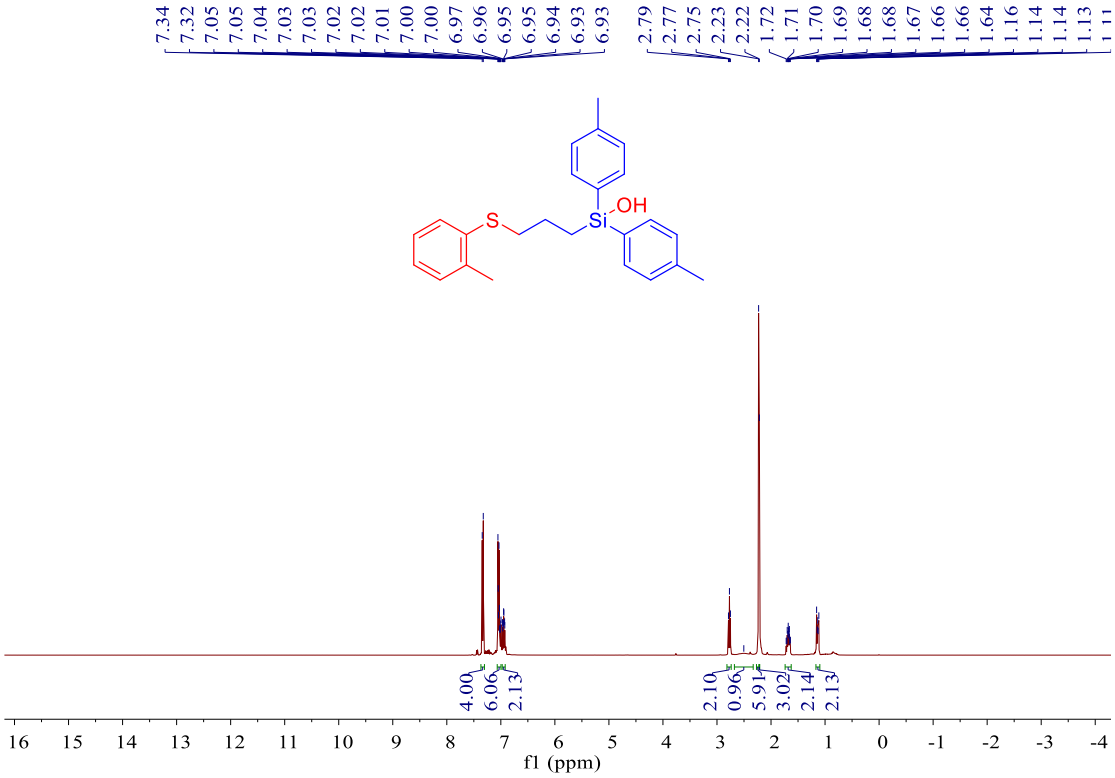
^{1}H NMR Spectra of **6b** (400 MHz, $CDCl_3$)



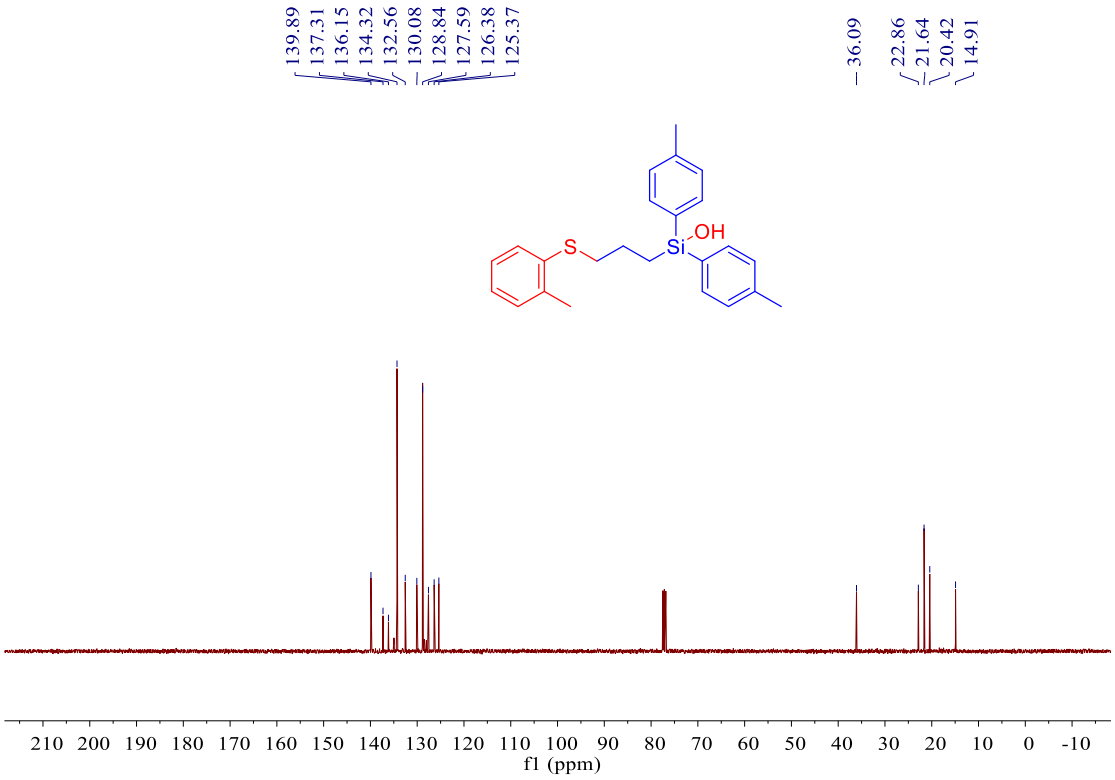
^{13}C NMR Spectra of **6p** (100 MHz, $CDCl_3$)



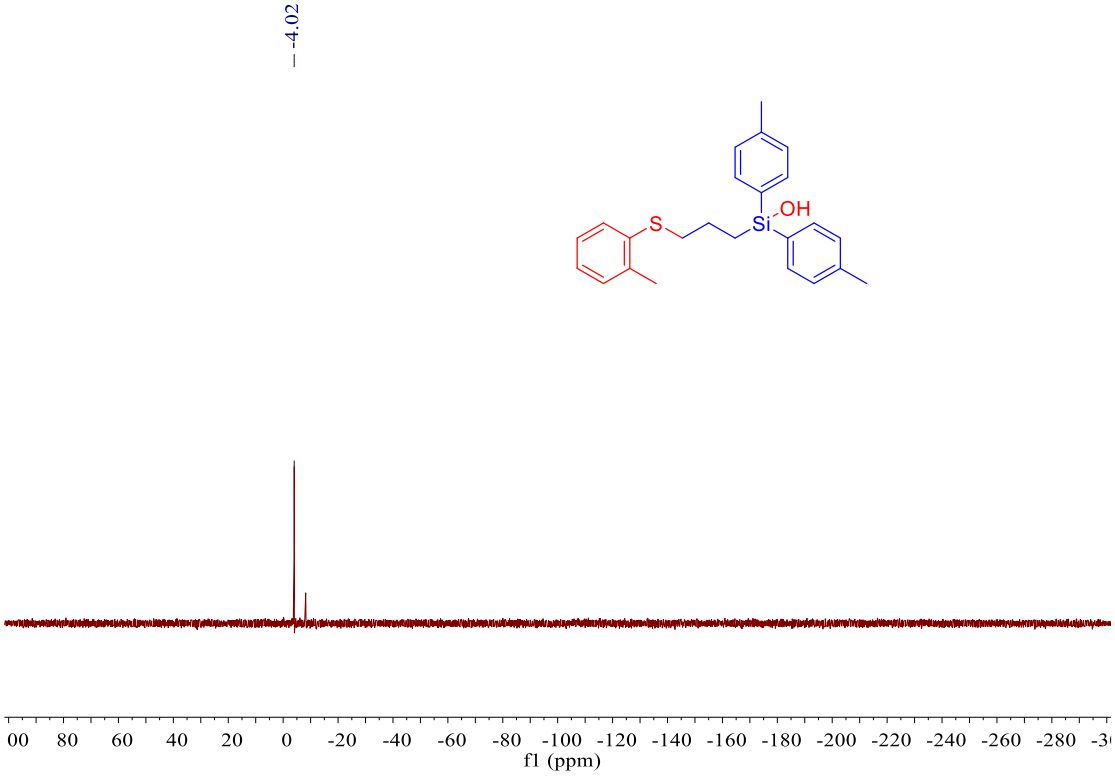
^{1}H NMR Spectra of **6c** (400 MHz, $CDCl_3$)



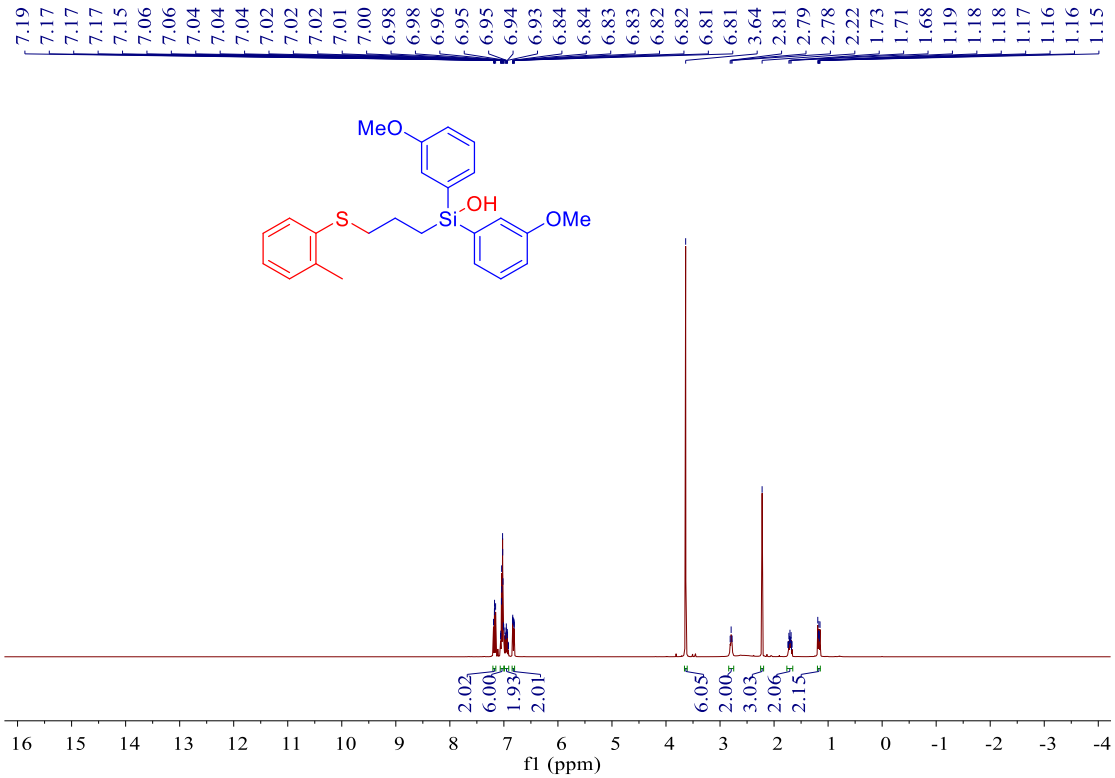
^{13}C NMR Spectra of **6c** (100 MHz, $CDCl_3$)



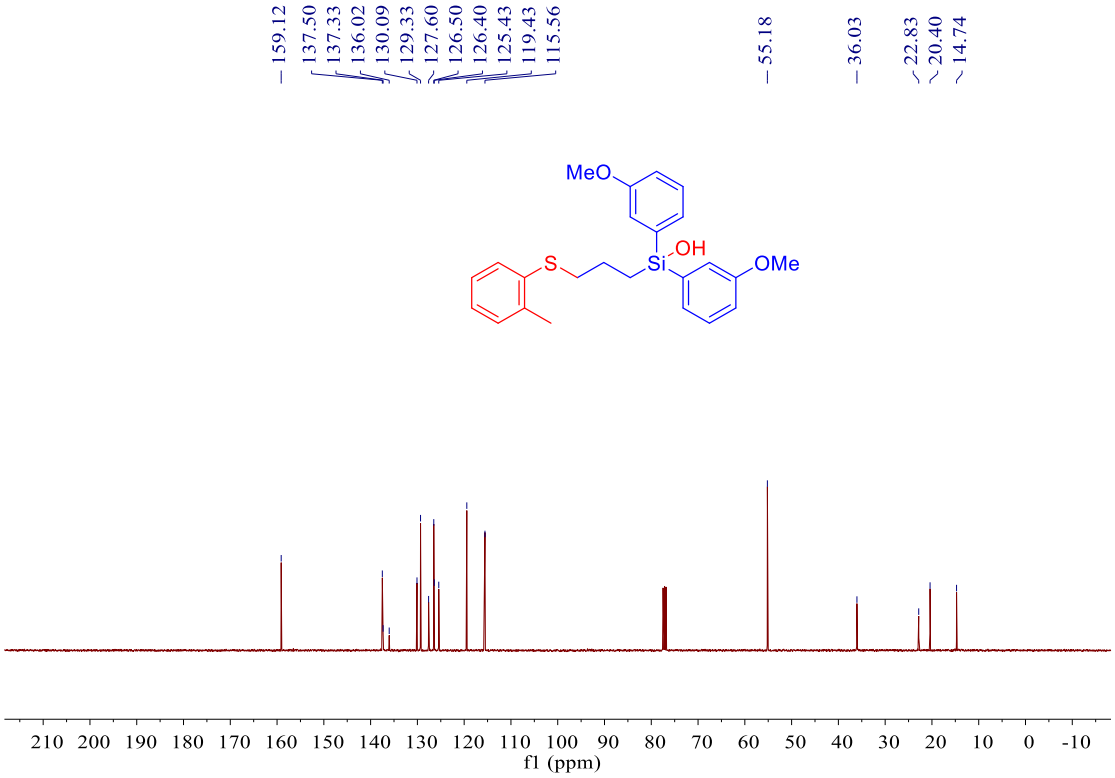
^{29}Si NMR Spectra of **6c** (79 MHz, $CDCl_3$)



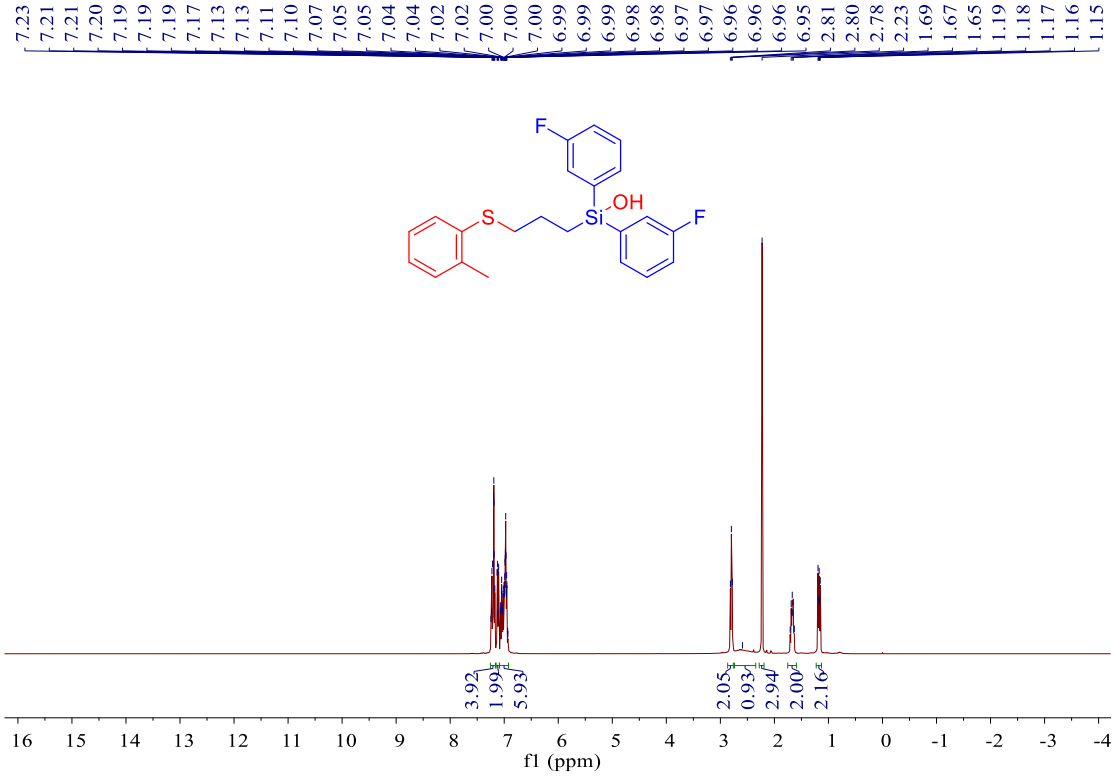
^{1}H NMR Spectra of **6d** (400 MHz, $CDCl_3$)



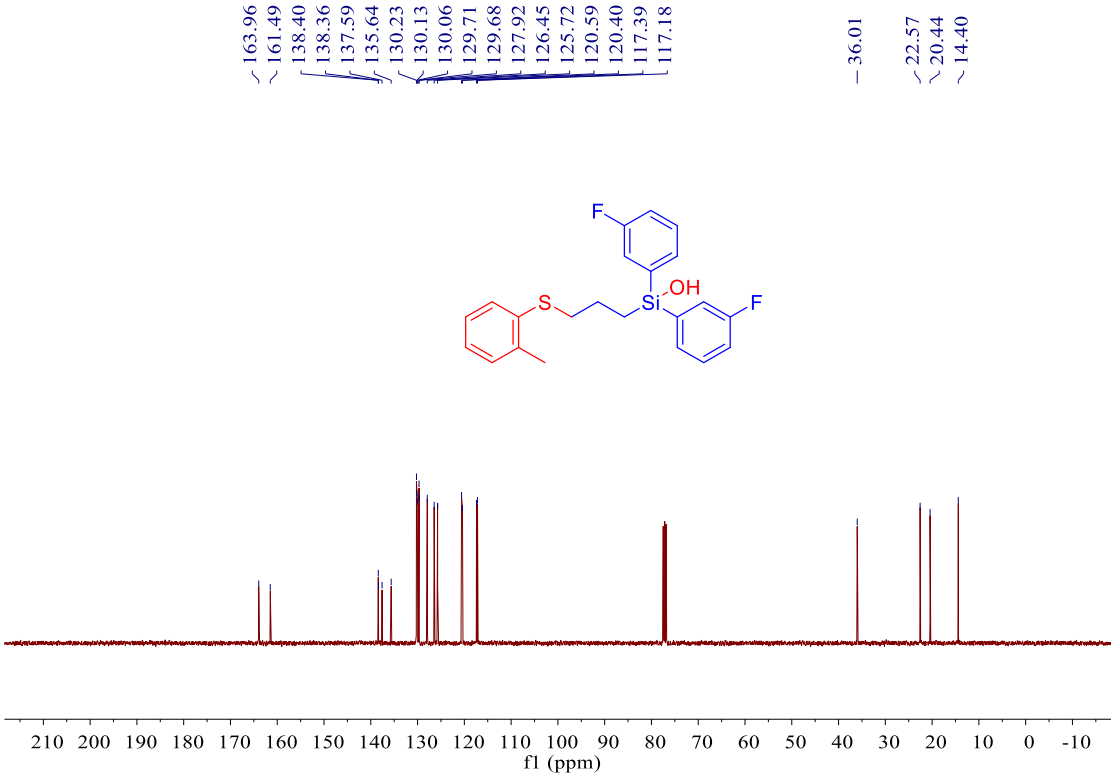
^{13}C NMR Spectra of **6d** (100 MHz, $CDCl_3$)



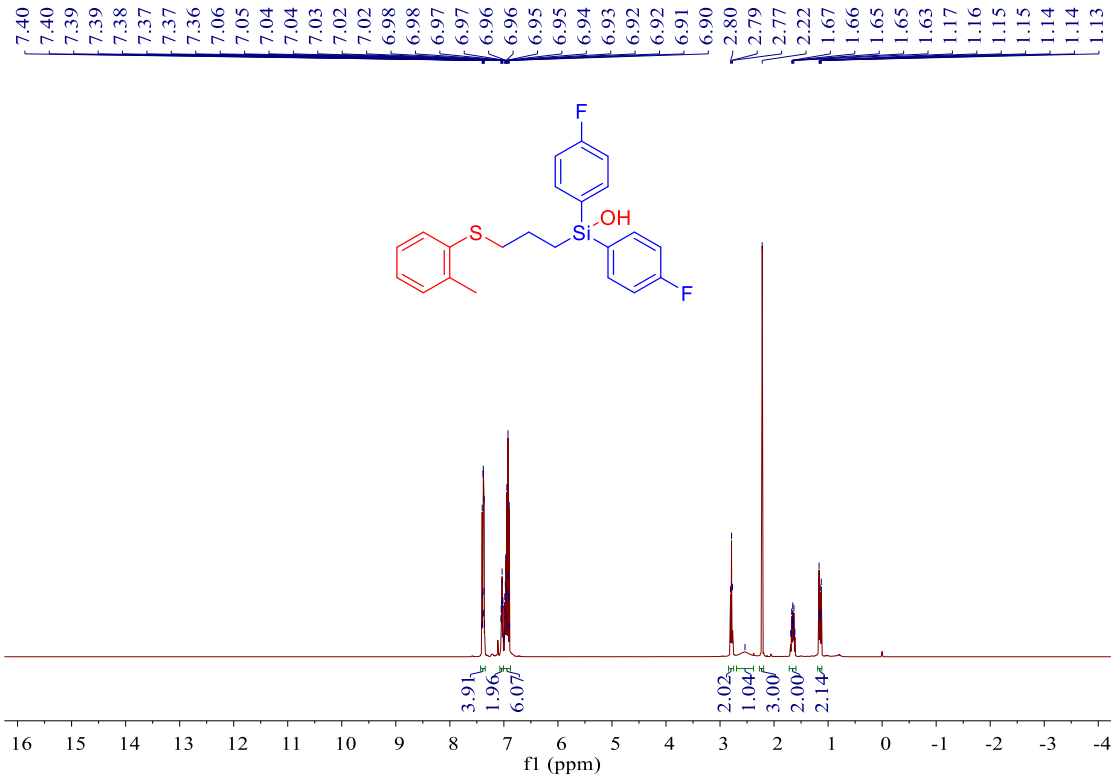
^{1}H NMR Spectra of **6e** (400 MHz, $CDCl_3$)



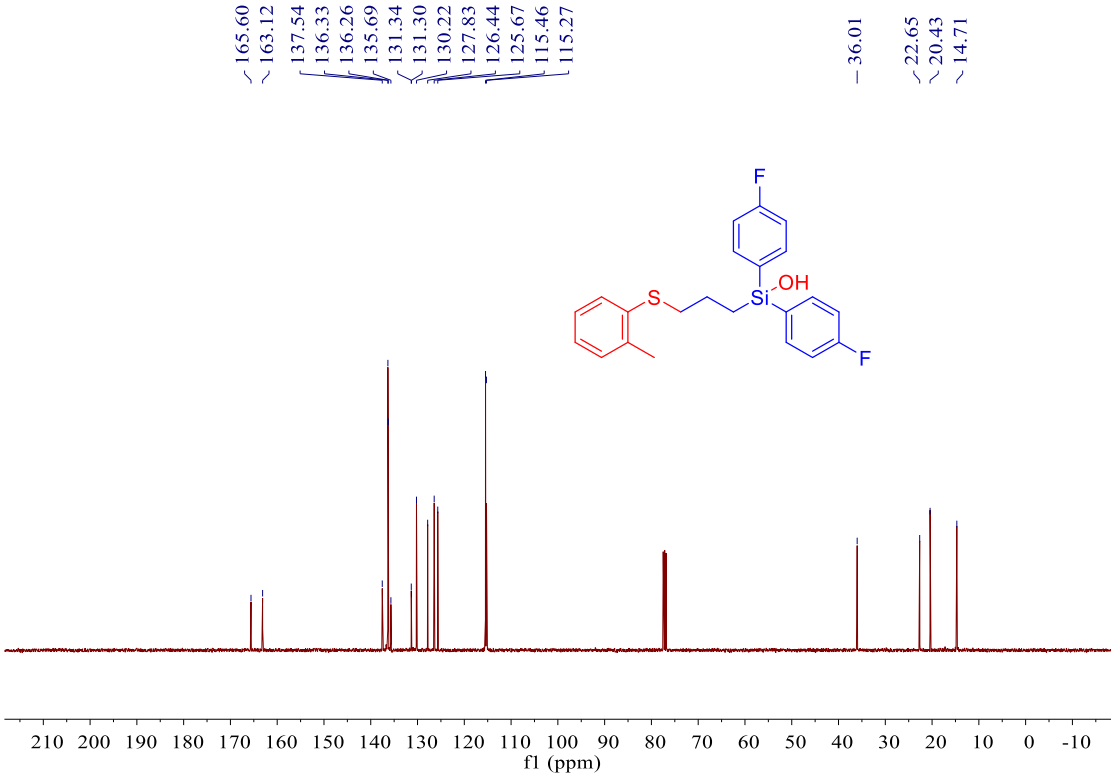
^{13}C NMR Spectra of **6e** (100 MHz, $CDCl_3$)



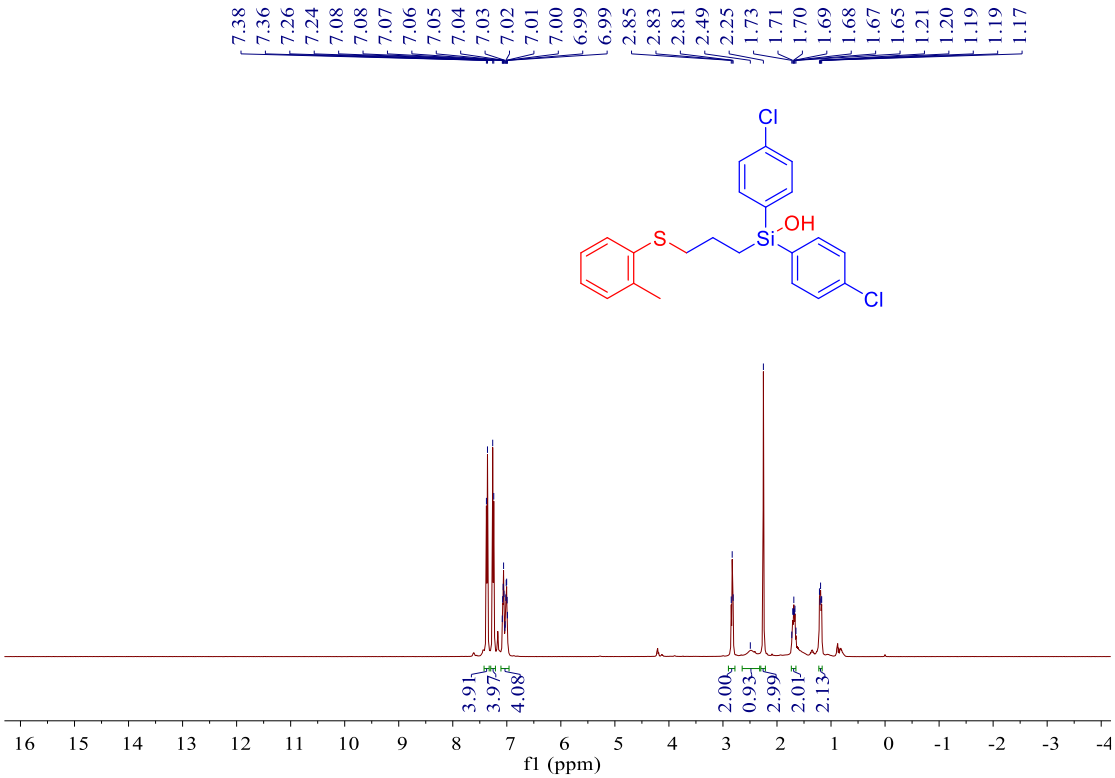
^{1}H NMR Spectra of **6f** (400 MHz, $CDCl_3$)



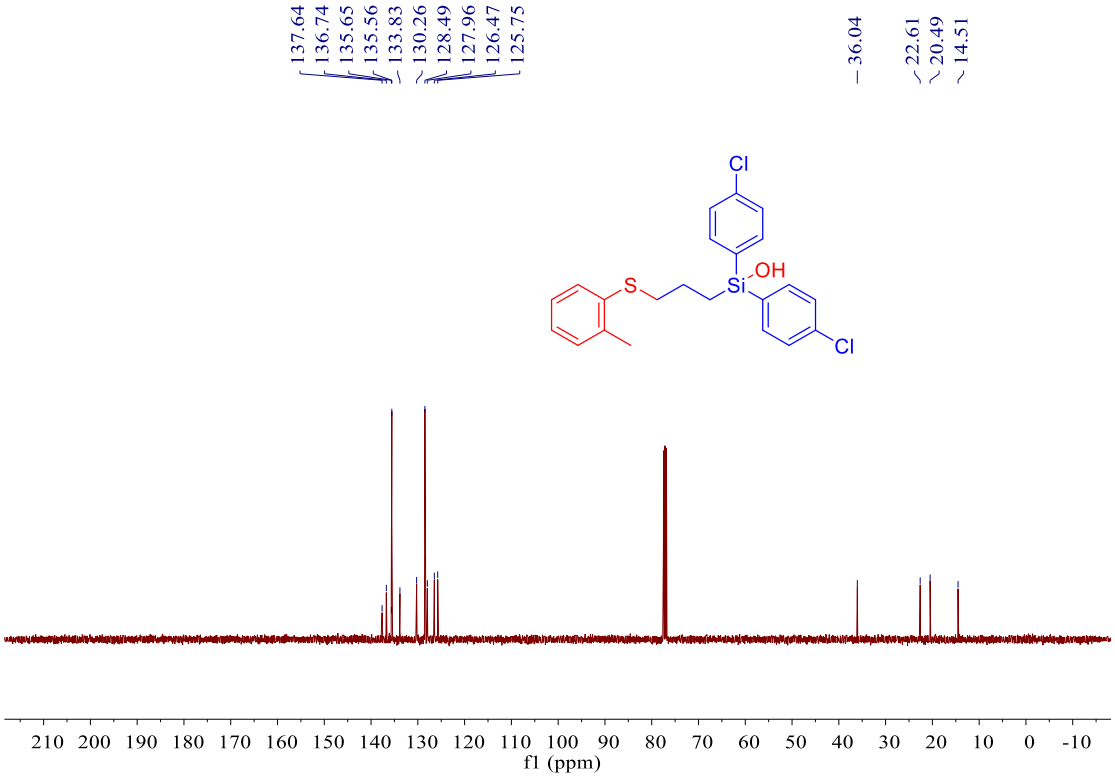
^{13}C NMR Spectra of **6f** (100 MHz, $CDCl_3$)



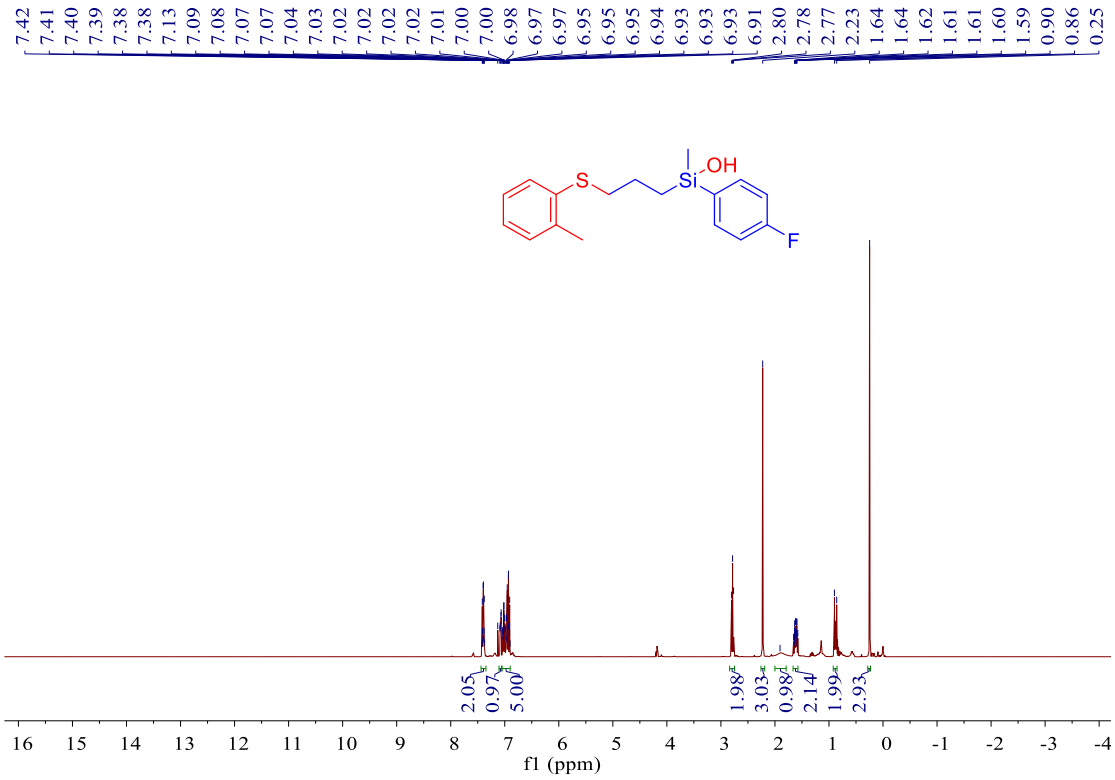
^{1}H NMR Spectra of **6g** (400 MHz, $CDCl_3$)



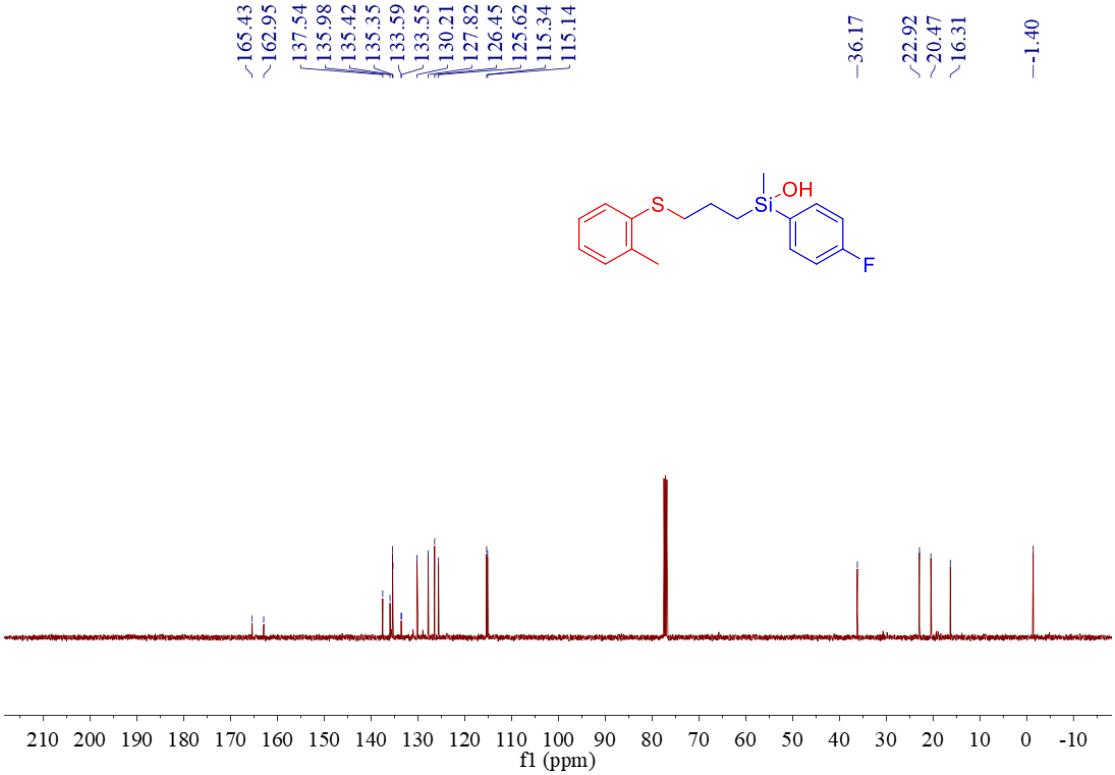
^{13}C NMR Spectra of **6g** (100 MHz, $CDCl_3$)



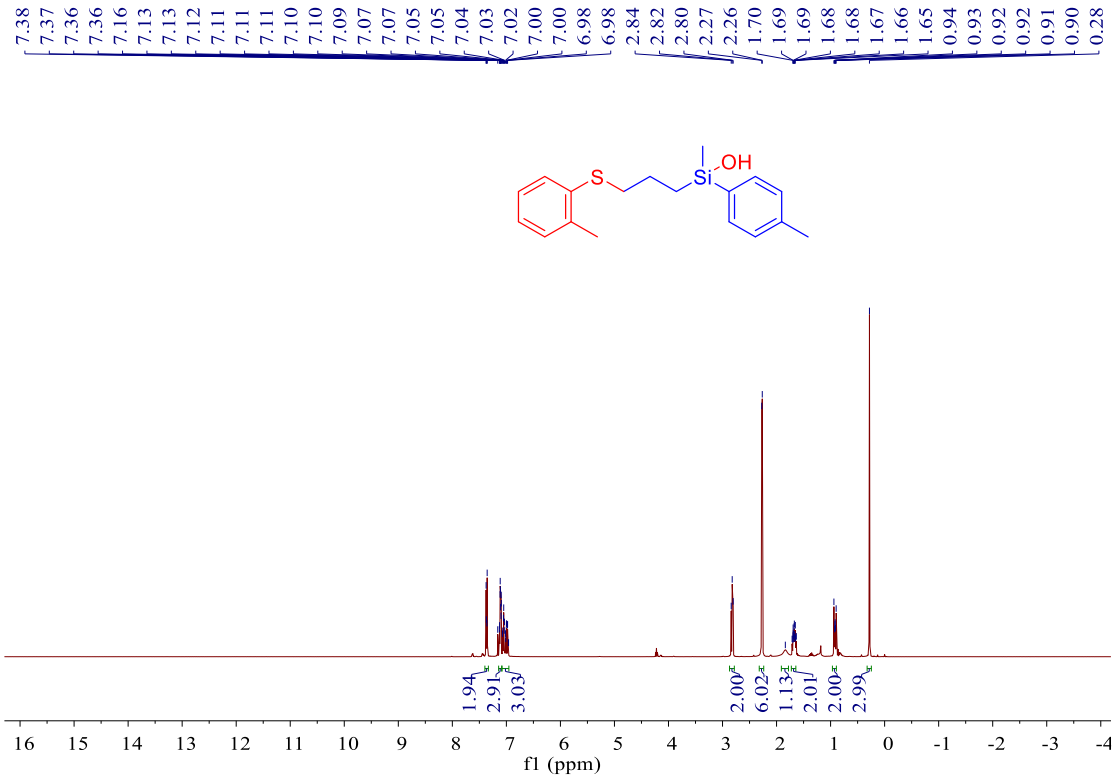
^{1}H NMR Spectra of **6h** (400 MHz, $CDCl_3$)



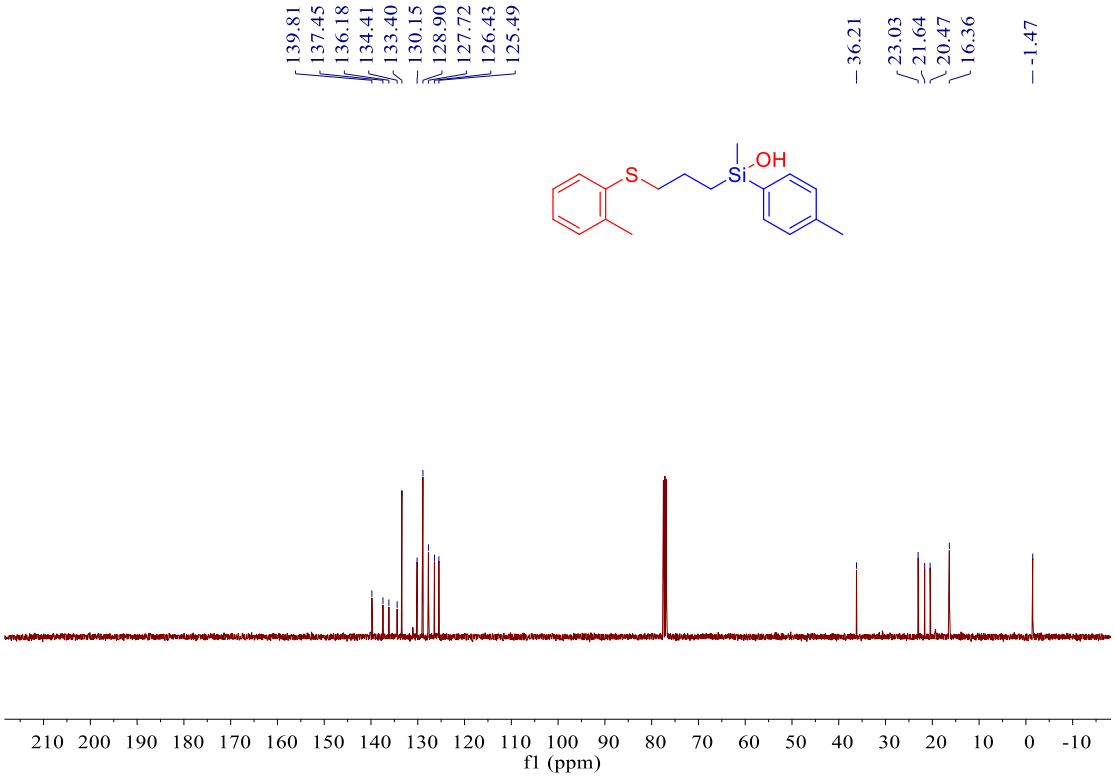
^{13}C NMR Spectra of **6h** (100 MHz, $CDCl_3$)



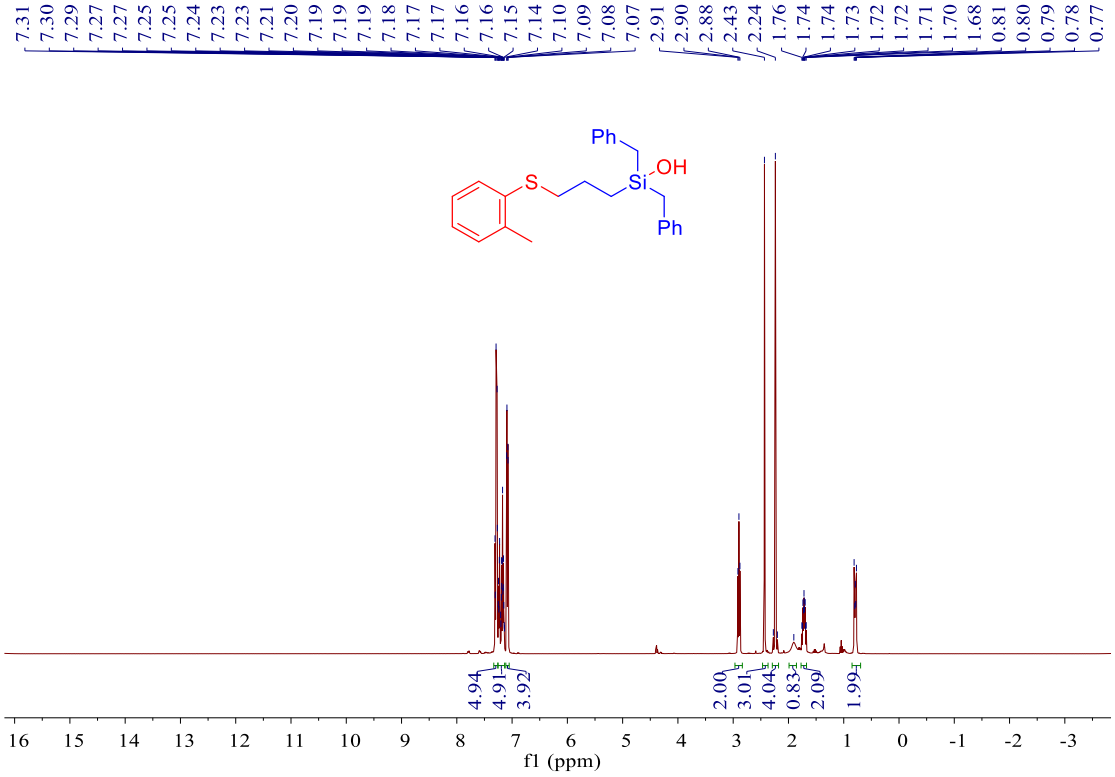
^{1}H NMR Spectra of **6i** (400 MHz, $CDCl_3$)



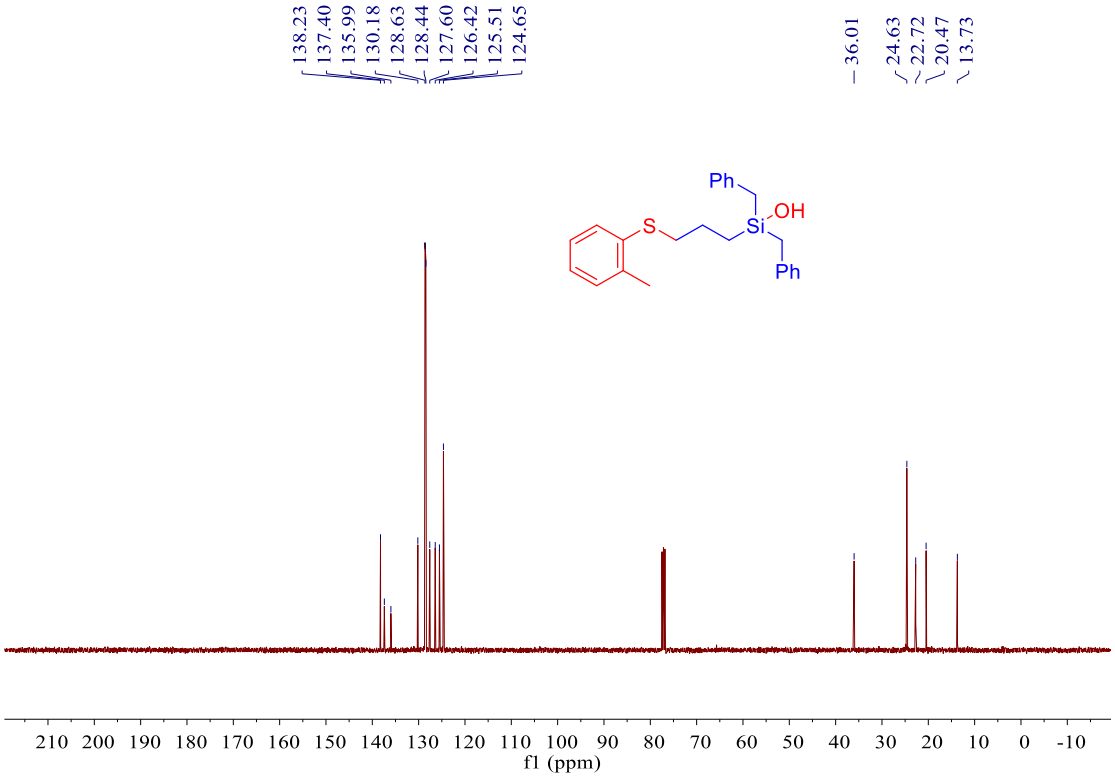
^{13}C NMR Spectra of **6i** (100 MHz, $CDCl_3$)



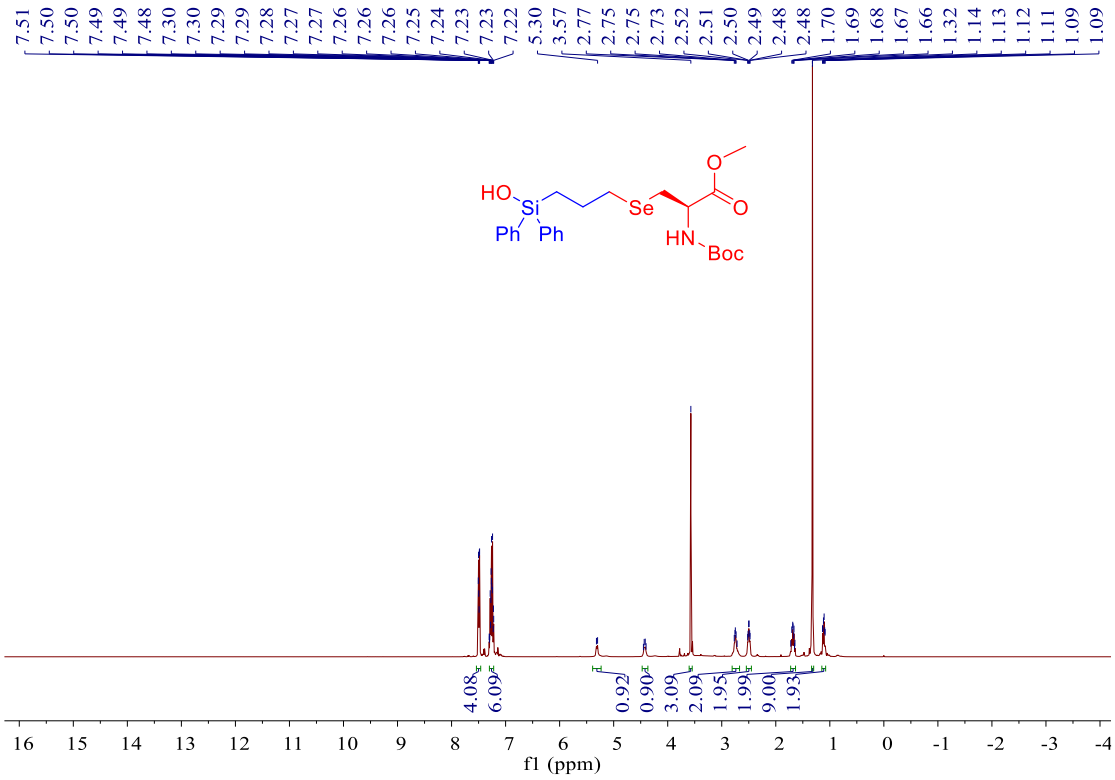
^{1}H NMR Spectra of **6j** (400 MHz, $CDCl_3$)



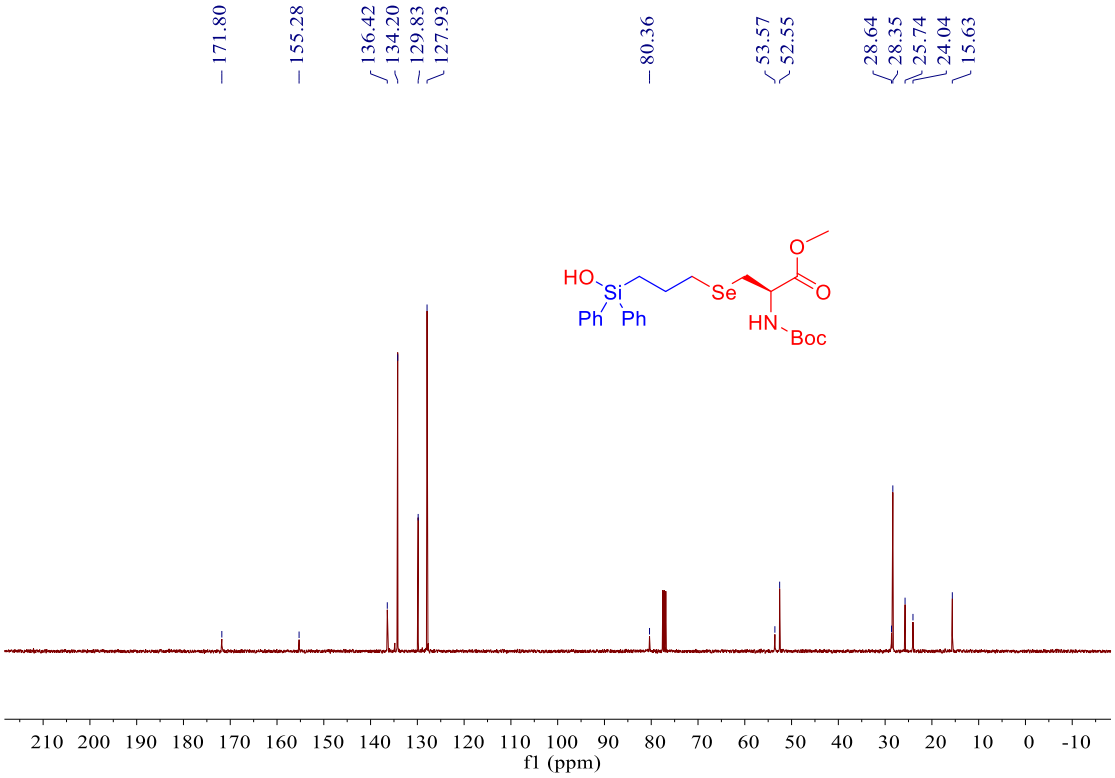
^{13}C NMR Spectra of **6j** (100 MHz, $CDCl_3$)



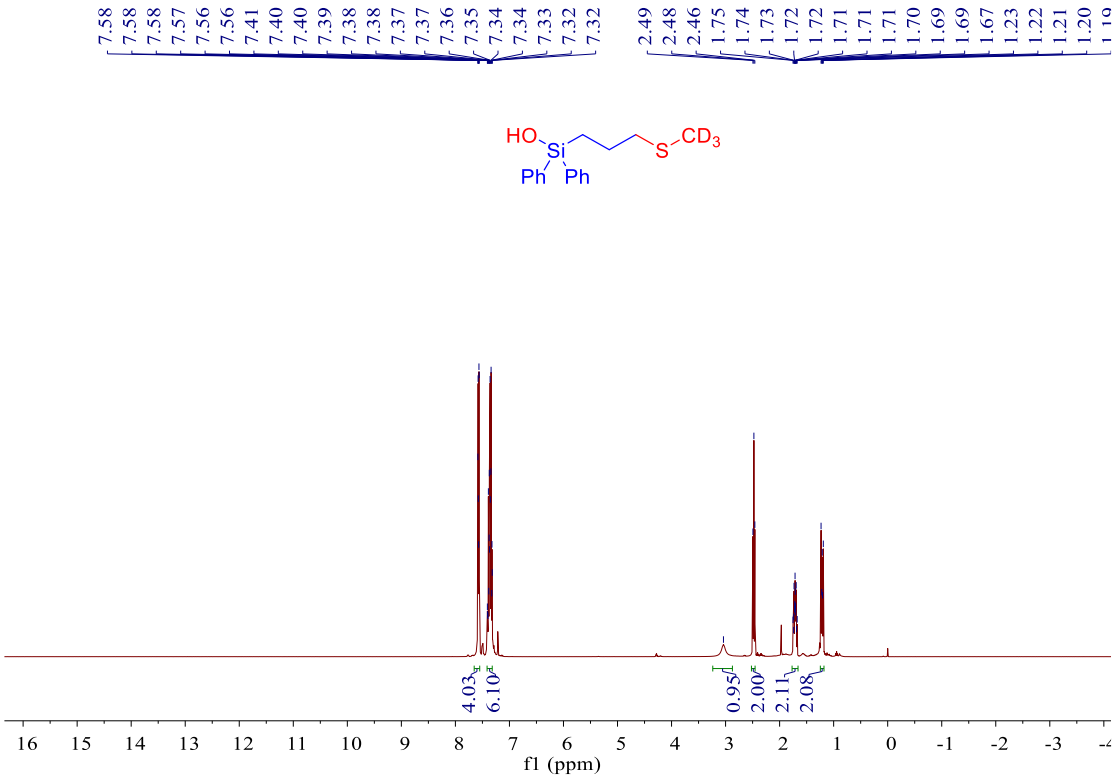
^{1}H NMR Spectra of **5s** (400 MHz, $CDCl_3$)



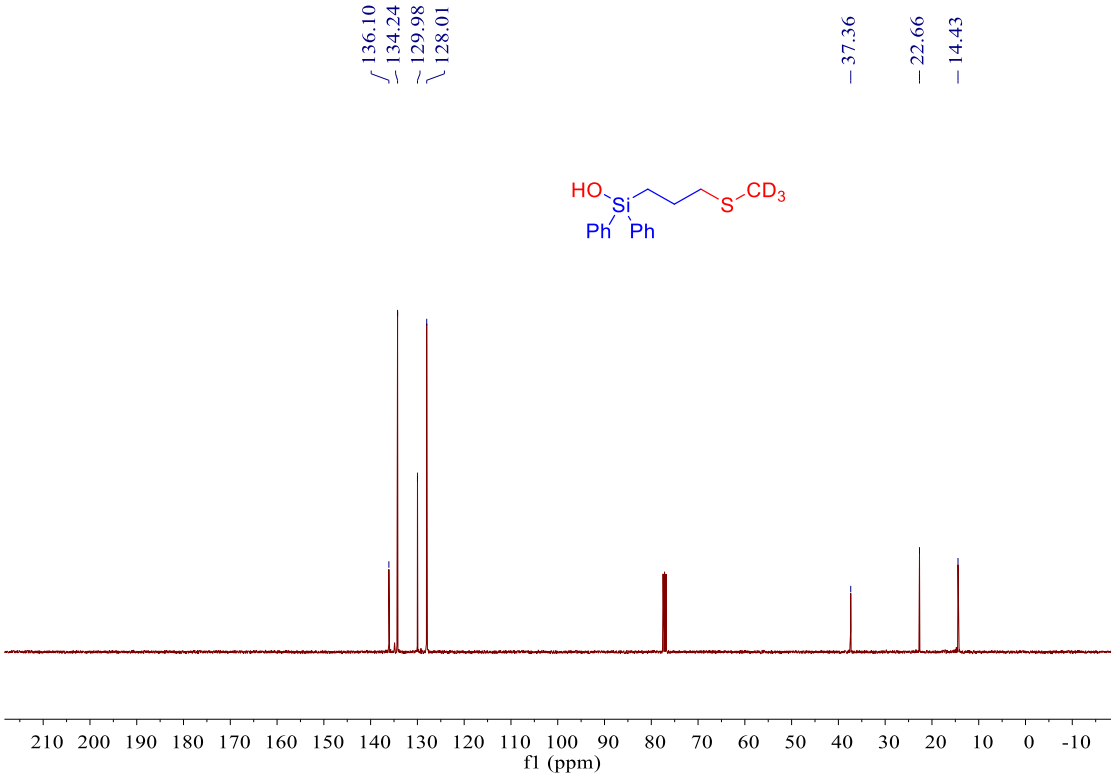
^{13}C NMR Spectra of **5s** (100 MHz, $CDCl_3$)



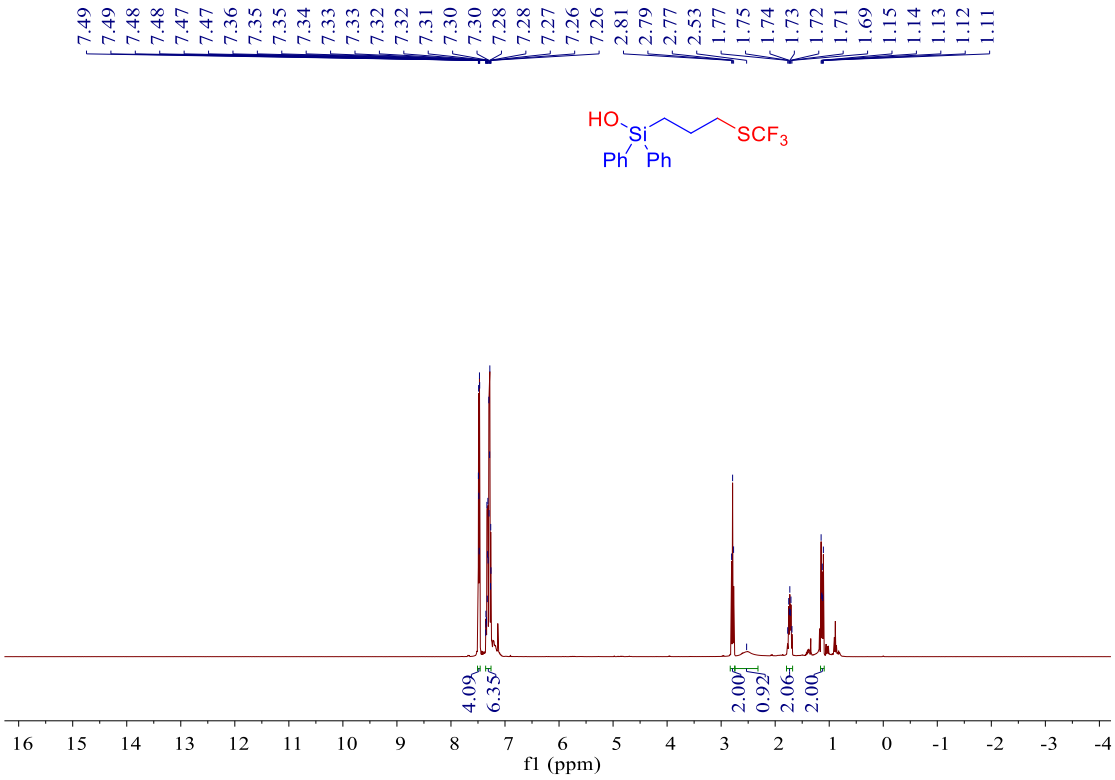
^{1}H NMR Spectra of **5t** (400 MHz, $CDCl_3$)



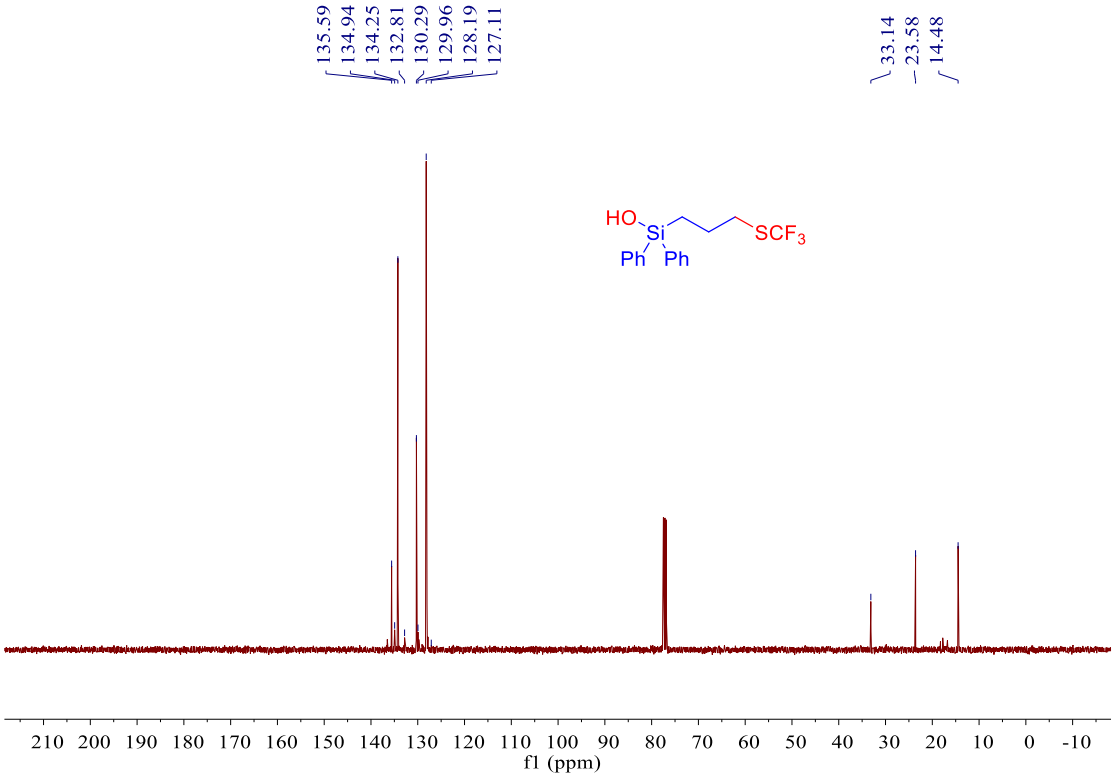
^{13}C NMR Spectra of **5t** (100 MHz, $CDCl_3$)



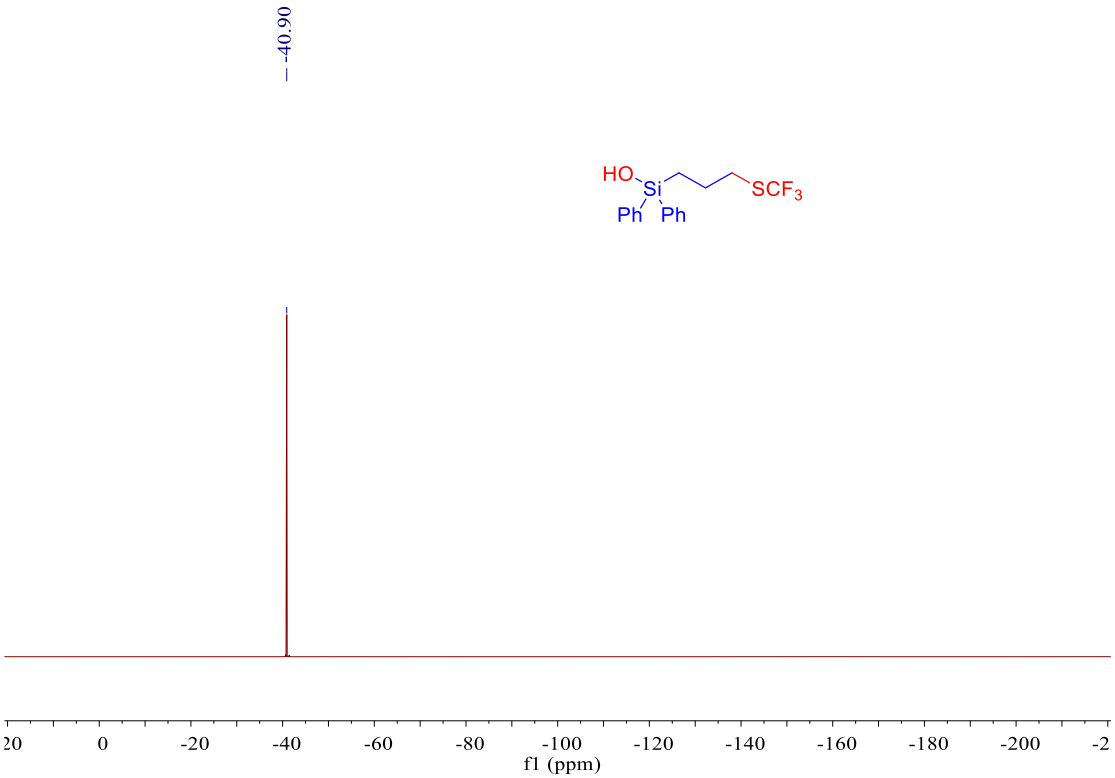
^{1}H NMR Spectra of **5u** (400 MHz, $CDCl_3$)



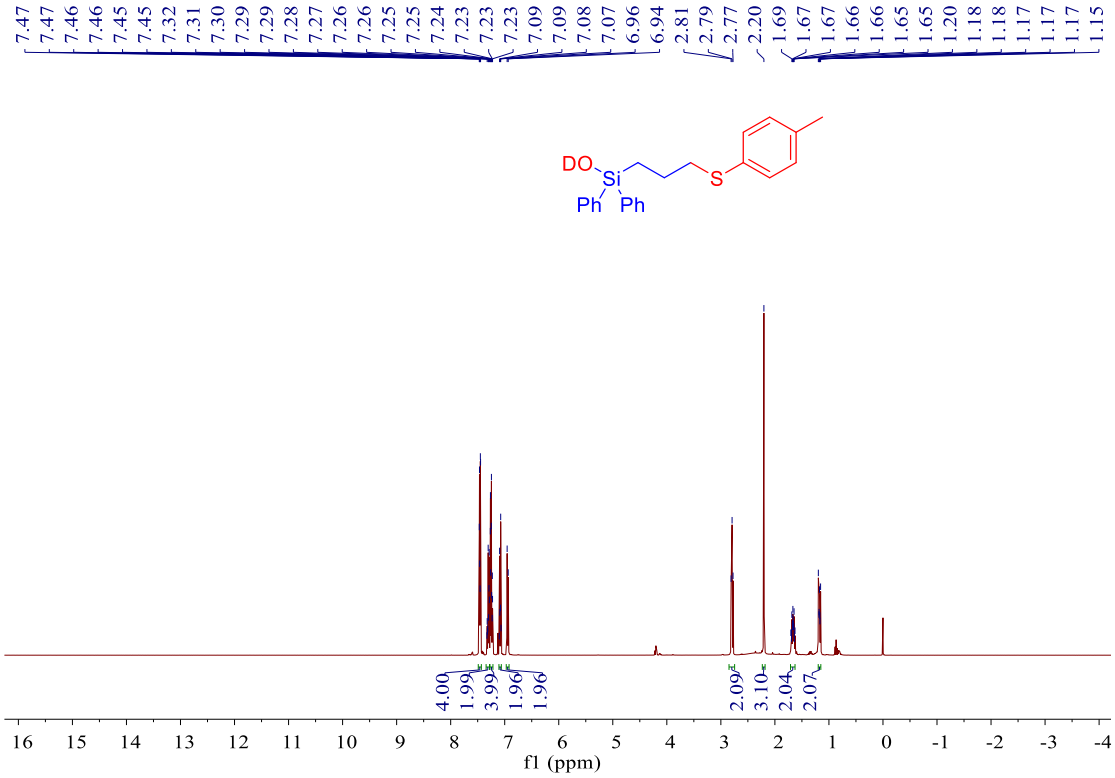
^{13}C NMR Spectra of **5u** (100 MHz, $CDCl_3$)



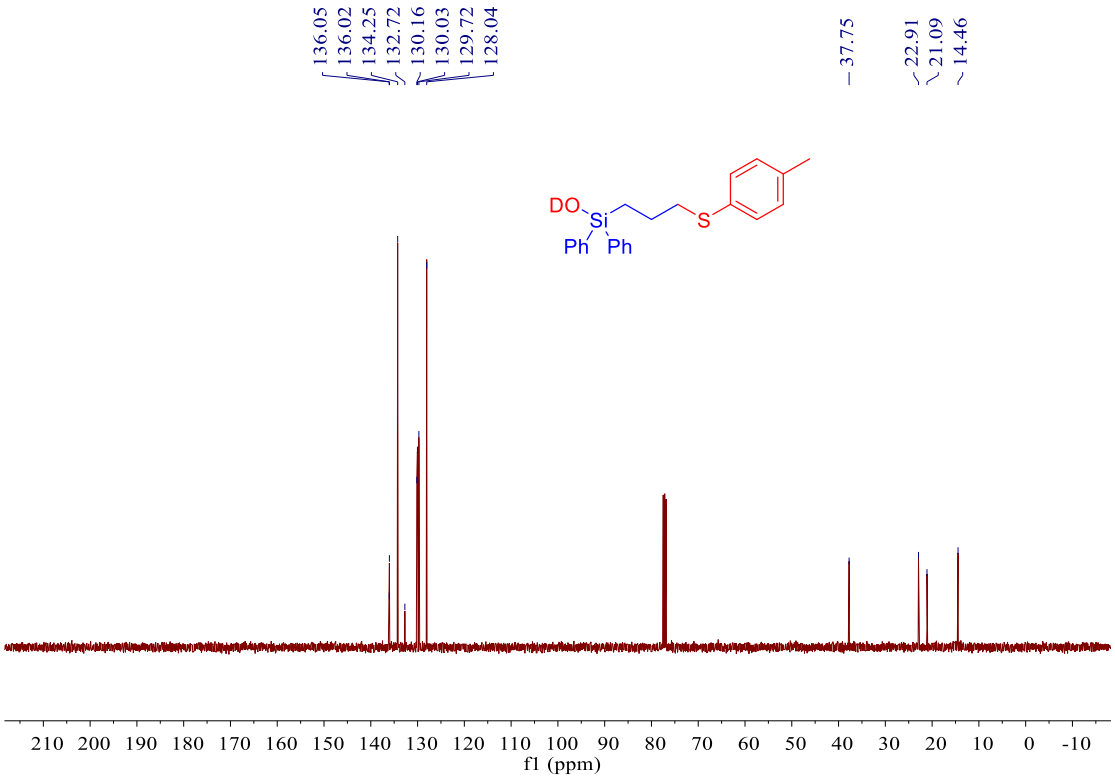
^{19}F NMR Spectra of **5u** (100 MHz, $CDCl_3$)



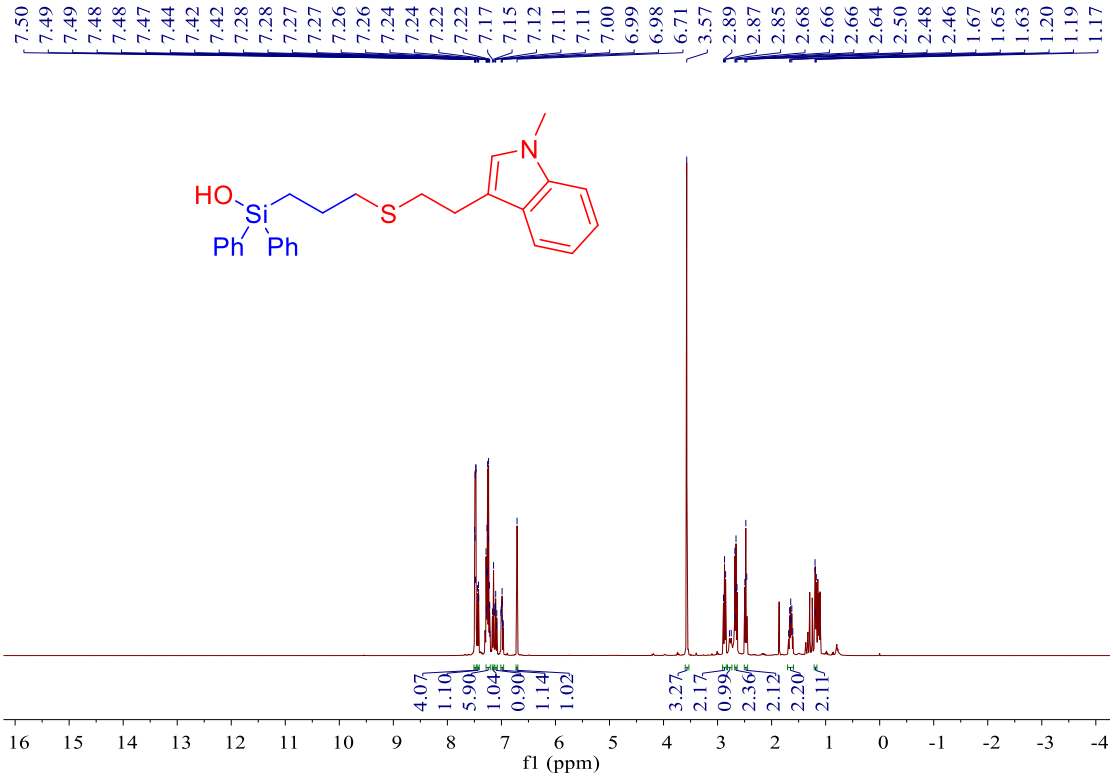
^{1}H NMR Spectra of **3c'** (400 MHz, $CDCl_3$)



^{13}C NMR Spectra of **3c'** (100 MHz, $CDCl_3$)



^{1}H NMR Spectra of **5s** (400 MHz, $CDCl_3$)



^{13}C NMR Spectra of **5s** (100 MHz, $CDCl_3$)