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Two Methods for Selective Deprotection of Diphenylmethylsilyl Protecting Group under Aqueous Conditions

A Thesis submitted in partial satisfaction of the

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in Chemistry

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ABSTRACT

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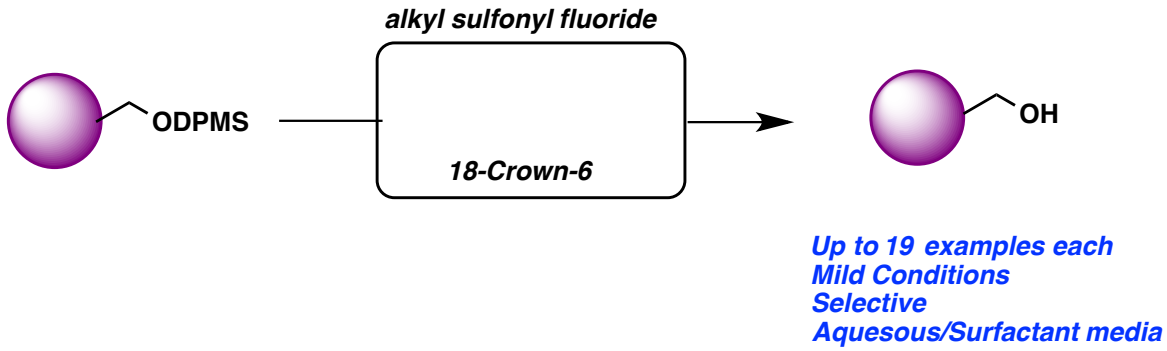

Two, new, methods for chemo-selective deprotection of diphenylmethylsilylether of allylic, benzylic, and alkyl alcohols is achieved. Catalytic amounts of Perfluoro-1-butanesulfonyl fluoride or stoichiometric amounts of 18-crown-6 ether are employed for successful deprotection. All selective deprotection can be attained under mild aqueous conditions.

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Introduction

Silyl ethers are ubiquitous in the realm of protecting group chemistry. Before their importance in protecting group chemistry is brought to light, a general overview of these compounds will be discussed. Silyl ethers can be described as a group of chemical compounds which contain a silicon atom covalently bonded to an alkoxy group. A general structure to these molecules is denoted as $R^1R^2R^3Si\text{-}O\text{-}R^4$ where each R group can be an alkyl or aryl group. The most commonly used silyl groups can be found in Figure 1.

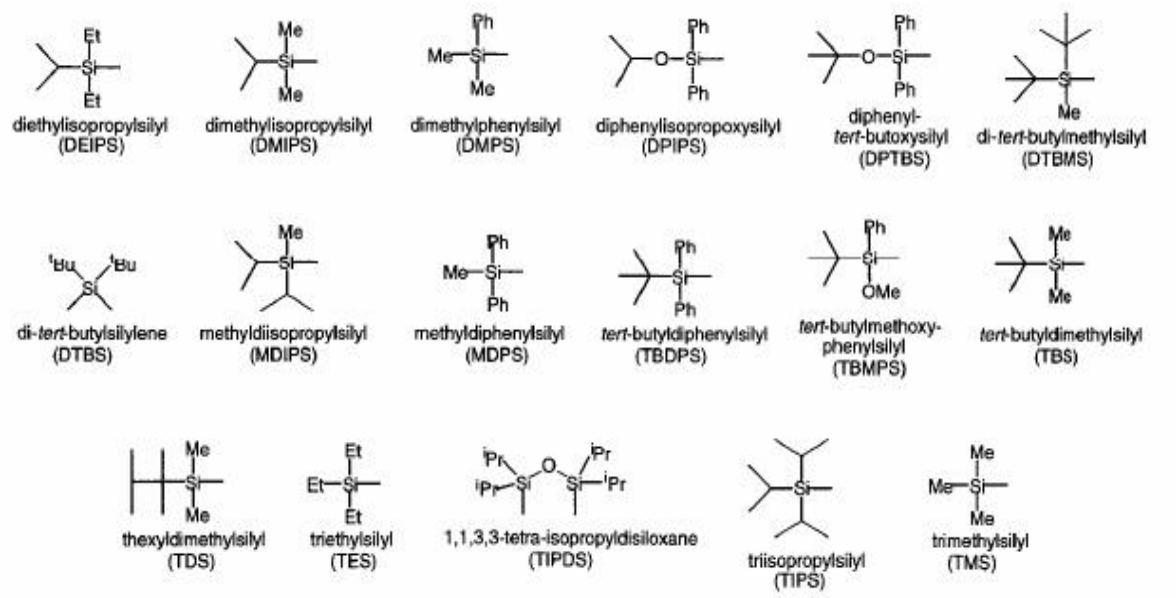
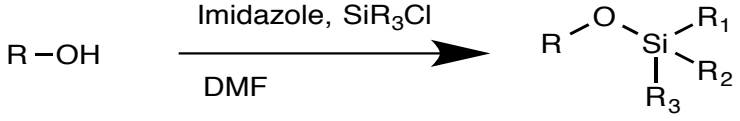


Figure 1[1]**:** *Commonly used silyl ether protecting groups in the last 20 years.*

There are a multitude of synthetic routes for successful silylation of alcohols; however, the most common is the Corey protocol. This achieves silylation by the addition of an alcohol to a solution of a silyl chloride derivative and an amine base, the most common being imidazole or triethylamine, dissolved in DMF at a high concentration (Scheme 1).[2]

Scheme 1



When protection of the alcohol is no longer needed, addition of an acid or fluoride source, the most common being tetra-*n*-butylammounium fluoride (TBAF), removes most silyl groups even if more than one is present. However, when the protection of more than one hydroxyl group is necessary, specific silyl ethers are selected so that each silyl ether can be selectively removed at a later stage of the synthetic scheme,[1] thus, allowing unlike transformations of the various hydroxyl groups in the same molecule. As the complexity of target molecules increase, the need for chemo- and regio-selective deprotection remains crucial in synthetic organic chemistry. Known procedures and new procedures for selective deprotection of various silyl ether groups will be explored throughout this thesis.

Background

Silyl ether protecting groups comprise one of the most convenient and efficient methods to protect any hydroxyl functionality.[2] The most commonly used of the silyl ethers include tert-butyldimethylsilyl (TBS), triisopropylsilyl (TIPS), tert-butyldiphenylsilyl (TBDPS) and diphenylmethylsilyl (DPMS). Many have been employed in the total synthesis of biologically active compounds such as, taxol, brevetoxin, rapamycin, zaragozic acid, and the avermectins.[1] The synthetic routes to these biologically active compounds requires the existence of multiple silyl ether protecting groups at a given time. This demands careful planning and execution of the deprotection conditions for each silyl group (acidic, basic, temperature, reaction time, etc.). Fortunately, a plethora of literature exists that allows for discrimination against different silyl ether groups and selective deprotection of one can be achieved while leaving the other intact.[1] The more distinguished the steric and electronic environments of the two silyl ethers, the more successful the selective deprotection.[3]

Guindon and Gillard, et al. showcased how electronic effects of silyl ethers can be exploited to increase selectivity between certain silyl ether groups.[4,5] Each argued that the

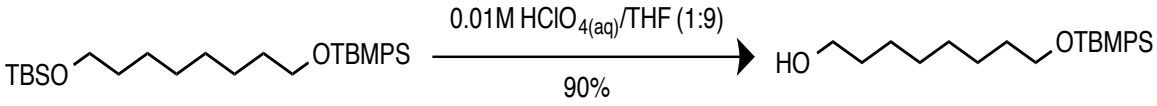
added influence of the electron-withdrawing alkoxy groups increases the difference in reactivity between the silyl ethers and allows for selective deprotection under acidic conditions.[5]

***Table 1**: Half-Lives of Hydrolysis of 1° Trialkylsilyl vs. Alkoxysilyl Ethers with 0.01M $HClO_4$*[5]

Trialkylsilyl Ethers		Alkoxysilyl Ethers	
n-$C_{12}H_{25}$OTBS	1.4 h	*n*-$C_{12}H_{25}$ODPIPS	0.7 h
n-$C_{12}H_{25}$OTBDPS	> 200 h	*n*-$C_{12}H_{25}$ODPTBS	17.5 h
		n-$C_{12}H_{25}$OTBMPS	200 h

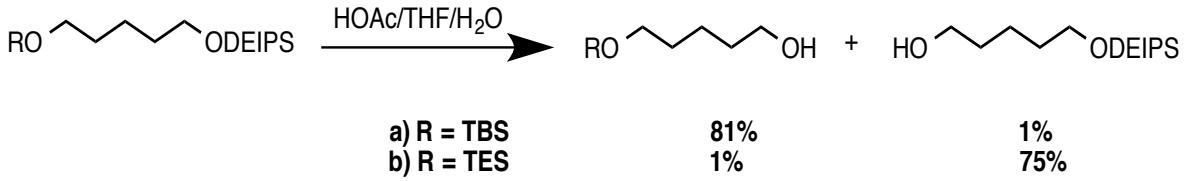
Gillard and Guindon et al. demonstrate the potential of selective cleavage of one 1° silyl ether in the presence of another using the protecting groups mentioned in Table 1. Scheme 2 provides the selective deprotection of a 1° TBS group in the presence of a 1° TBMPS ether, which coincides with the hydrolysis data found in Table 1.[5]

Scheme 2



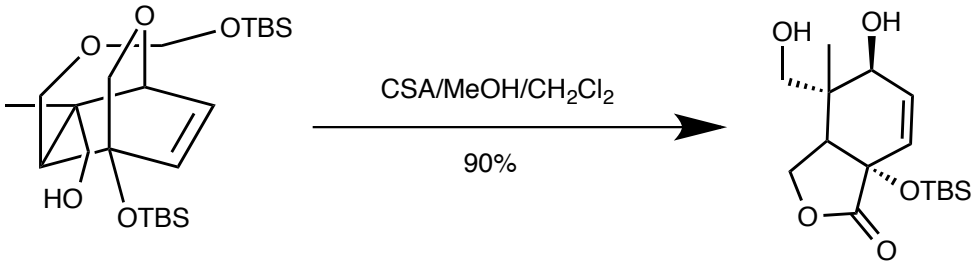
Toshima et al. has tackled the other side of the argument, sterics, with the introduction of a "new" silyl ether protecting group DEIPS.[6] Utilizing the traditional acid-catalyzed hydrolysis, acetic acid in THF/H_2O, Toshima et al. was able to distinguish the DEIPS group from the larger TBS group and the smaller TES group (Scheme 3).[6]

Scheme 3



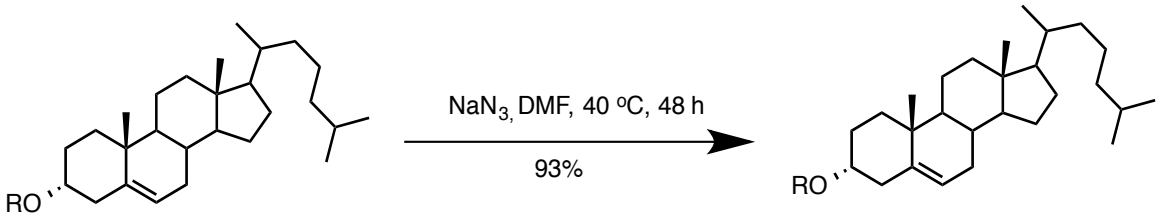
In certain circumstances, if two identical protecting groups are present in the same molecule, it is possible to achieve the mono-deprotected product. As stated above, this can only be possible if there exists sufficient differences within the environments (sterically and/or electronically) in which the silyl ether groups reside.[3] In the synthesis of the taxol precursor, prepared by Nicolaou et al. (Scheme 4), the TBS at the position of the *ortho*-ester was hydrolyzed with acid while the tertiary TBS group was untouched to give excellent yield of the ring opened product.[7]

Scheme 4



While exploring the literature, it was apparent that selective deprotection of many silyl ethers has been studied and successfully demonstrated. However, selective deprotection of diphenylmethylsilyl ether was significantly less represented. The available literature for selective deprotection of the DPMS ether employs harsh reaction conditions that do not favor a lot of functionality.[8-10] Monger et al. reported the use of sodium azide in DMF at elevated temperatures will result in the selective removal of the diphenylmethylsilyl ether in the presence of TBDMS and TBDPS with yields as high as 93% (Scheme 5).[10]

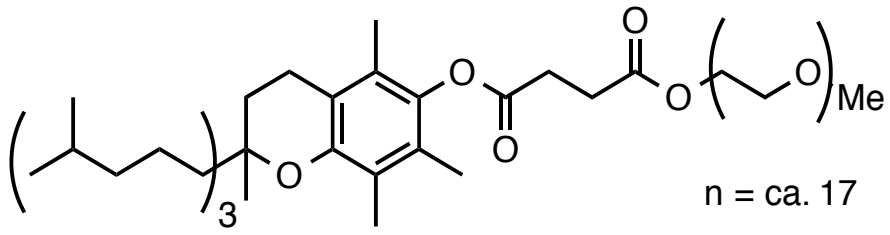
Scheme 5



(1) R = DPMS
(2) R = TBDPS
(3) R = TBDMS

(1) 93% Yield
(2) N/R
(3) N/R

Due to the limited scope of selective deprotection of the DPMS ether and the importance of selectivity in organic synthesis, we decided to investigate the phenomenon of selective deprotection under mild aqueous conditions. Herein, we report two new methods for the selective deprotection of primary and secondary DPMS ethers. The deprotections were facilitated by catalytic amounts of perfluoro-1-butanesulfonyl fluoride in 2 wt% TPGS-750-M/ H_2O (Figure 2) with a co-solvent of 15% v/v propanol, or stoichiometric amounts of 18-crown-6 in a 2:1 H_2O/PrOH solvent system.



TPGS-750-M

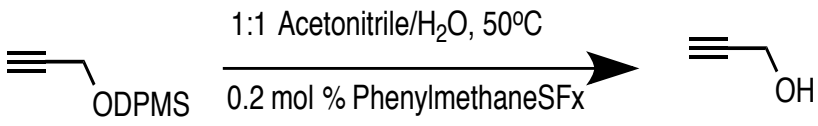
Figure 2: State of the art surfactant developed by the Lipshutz group

Results and Discussion:

Perfluoro-1-butanesulfonyl fluoride Deprotection

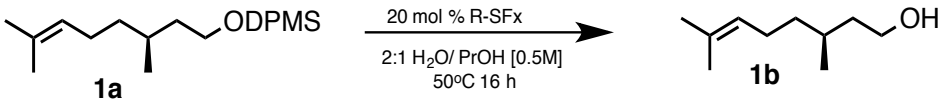
A few years ago, Zarko Boskovic, a member of the Lipshutz group, was investigating aqueous Sharpless SFx chemistry and observed hydrolysis of an activated DPMS protected alcohol in the presence of a catalytic amount of phenylmethane sulfonyl fluoride (Scheme 6).

Scheme 6



To elucidate whether this transformation was general to all sulfonyl fluorides, a variety of alkyl SFx were screened to determine their abilities to enable the deprotection of the DPMS ether (Table 2). The benzylic sulfonyl fluorides as well as sulfonyl fluorides bearing a strong electron-withdrawing component such as 4-nitrobenzenesulfonyl fluoride and perfluoro-1-butanesulfonyl fluoride exhibited the highest desilyating prowess on our model substrate **1a**. The remaining sulfonyl fluorides, consisting of only alkyl or aryl functionality, yielded sluggish to no conversion after 16 hours. From this study, perfluoro-1-butane sulfonyl fluoride was chosen for its greater reactivity and cheaper cost when compared to the 4-nitrophenyl or other benzyl derivatives. The original Sharpless procedure called for a 1:1 acetonitrile and water solvent system; however, after screening various solvent systems, propanol was found to be the most effective organic solvent due to its higher conversion to product, relatively benign nature and lower costs when compared to acetonitrile.

Table 2: *Deprotection of Citronellyl-diphenylmethylsilyl ether* ***1a*** *utilizing various Sulfonyl Fluorides*



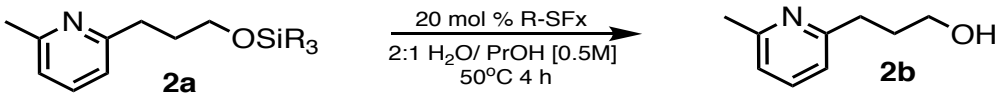
1a **1b**

Entry	R-Sulfonyl Fluoride	Yield (%)[a]
1	butane-SF	0
2	phenylmethane-SF	86
3	benzene-SF	17
4	4-propylpheny-SF	0
5	perfluorobutane-SF	94
6	*m*-nitrophenyl-SF	90

[a] Isolated yields

As the robustness of synthetic and bioactive molecules has increased, the need for selectivity cannot be overstated; therefore, it was crucial to test for the chemoselectivity of this new method. The chemoselectivity was examined with the use of different silyl ethers (Table 3). The trimethylsilyl (TMS) and the DMPS ethers resulted in full deprotection under our conditions in 4 hours while the tert-butyldimethylsilyl (TBS), triisopropylsilyl (TIPS), and tert-butyldiphenylsilyl (TBDPS) ether remained untouched. Due to the labile nature of the TMS ether, its deprotection was not surprising. It should be noted, after 72 hours, the full deprotection of the aforementioned ethers was also observed.

Table 3: *Chemo-selectivity of SFx with various silyl ethers*

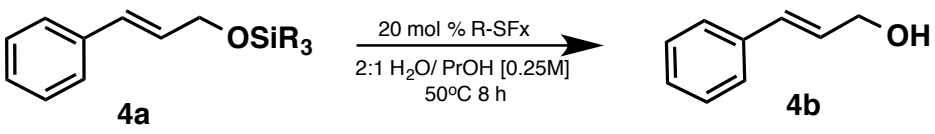


Entry	Silyl Ether	Yield (%)[a]
1	OTMS	95
2	OTIPS	0 (86)[b]
3	ODPMS	91
4	OTBS	0 (89)[b]
5	OTBDPS	0 (84)[b]

[a] Isolated yields
[b] Isolated yields after 72 h

Since the need for selectivity within the realm of organic chemistry is obvious, it was exciting to see the SFx method could discriminate between different alkyl silyl ether groups. Since the original Sharpless method used an activated DPMS ether to illustrate the deprotection, the level of selectivity towards activated DPMS ethers needed to be examined (Table 4). Since the TMS ether was fully deprotected in the previous set of selectivity experiments, it was excluded in the subsequent studies. Full deprotection of the DPMS ether was observed while the TBS and TIPS ethers remained intact. The extent of selectivity was tested further using a protected propargyl alcohol; however, the selectivity drastically diminished. The diminished selectivity could have been foreseen due to the electron-withdrawing capabilities of the acetylene moiety. A silyl ether affected by an electron-withdrawing group is more susceptible to hydrolysis.

Table 4: *Chemo-selectivity of SFx with various allylic silyl ethers*



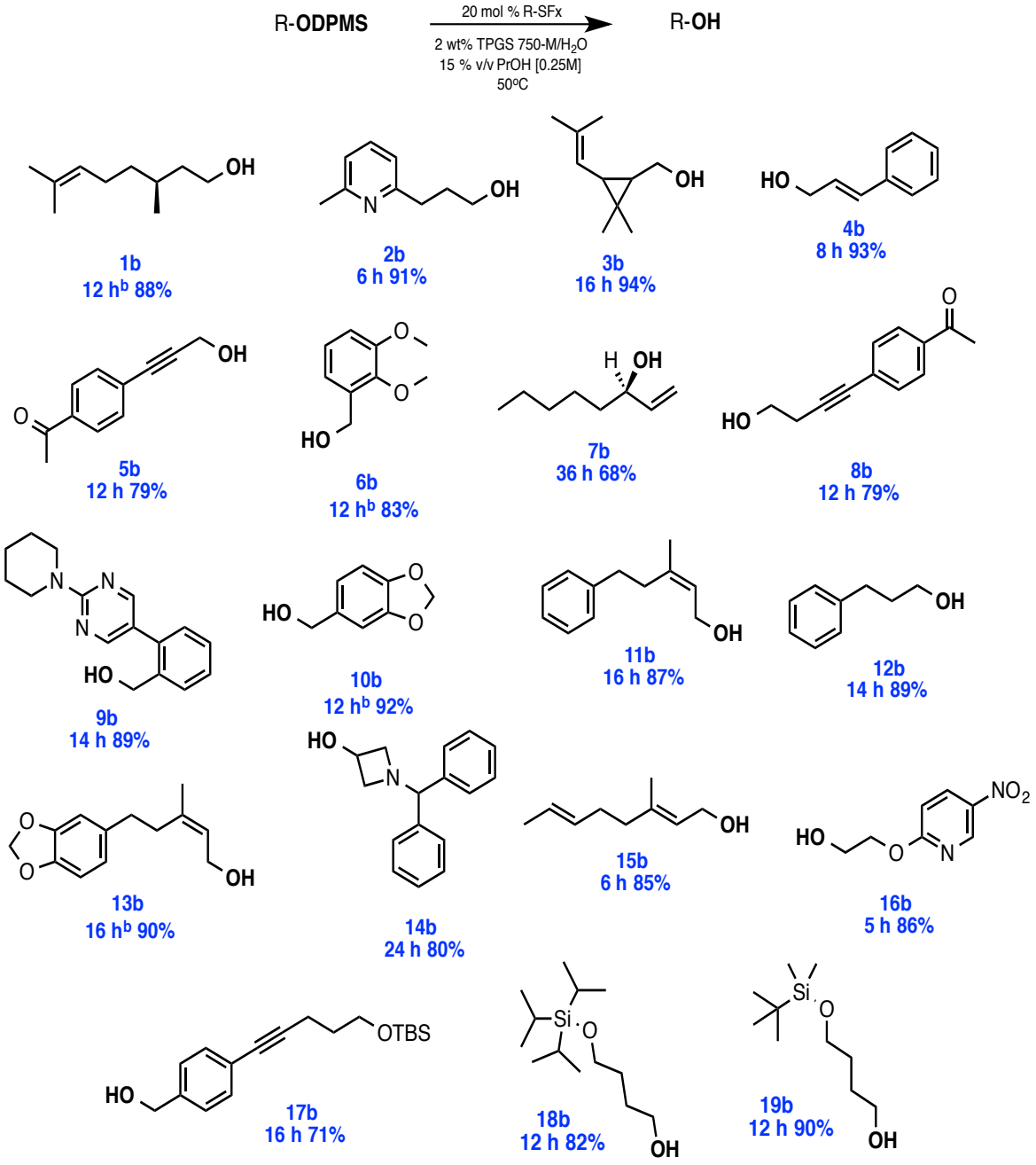
Entry	Silyl Ether	Yield (%)[a]
1	OTIPS	0
2	ODPMS	93
3	OTBS	0

[a] Isolated yields

In looking to expand the scope of the reaction, we started to investigate the effectiveness of the SFx species using our TPGS-750-M surfactant technology (Figure 2).[11] The use of TPGS-750-M enables deprotection of more difficult substrates, as solubility issues and longer reaction times were observed when using water and propanol as the reaction medium.

Utilizing this new medium and the previously mentioned SFx method, an extensive substrate scope was developed (Scheme 7). Primary alcohols containing a variety of functional groups including nitro, benzyl, propargyl, allylic and heteroaromatic systems were tolerated. Secondary alcohols were also deprotected, but a noticeable increase in reaction time was observed. The increased reaction time is likely due to increased steric hinderance. Increasing the mole percent of the sulfonyl fluoride was observed to decrease reaction times. Tertiary alcohols led to no conversion. To gain a better understanding of the mechanism and further investigate selectivity, aryl, enolic and sp^2-based silyl ethers were explored and were found to show no reactivity to this chemistry. Mechanistically, this data suggests that the selectivity of the method could be electronically influenced.

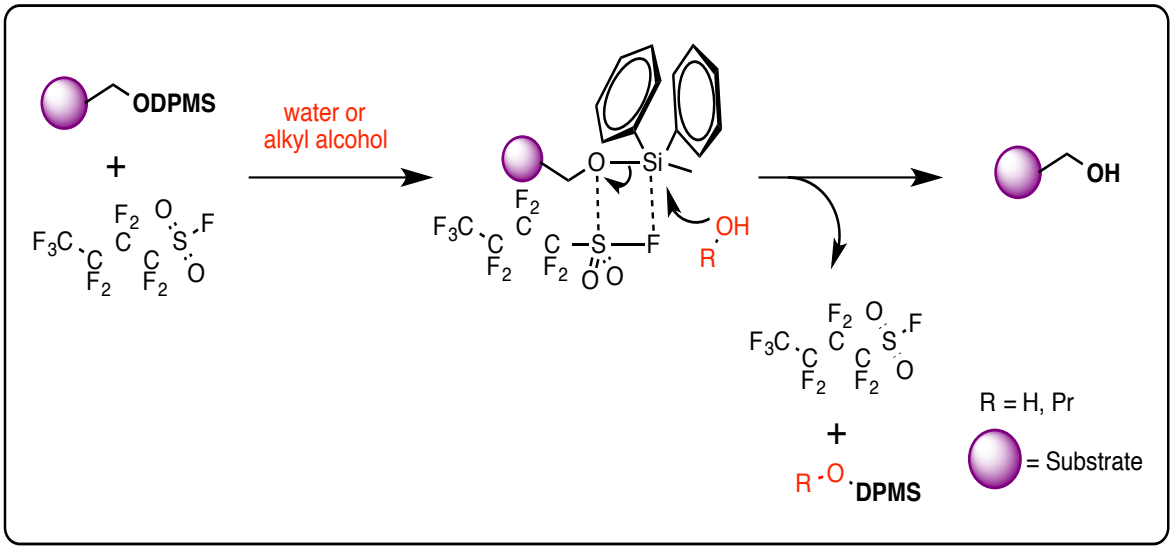
Scheme 7



a. Reaction times not optimzed
b. H_2O/Propanol (2:1) used as solvent system
Reactions were ran at 0.5M

A number of possible mechanisms to describe the cleavage of silyl ethers have been proposed in the literature.[12] The steric and electronic effects around the silicon atom have been studied and it has been concluded that the substitution around silicon plays a crucial role in the reactivity and mechanistic pathway.[13-14] It has also been determined, under certain deprotection conditions, that silicon can become hypervalent.[1] The fluoride ion has also been shown to be responsible for the deprotection of silyl ethers;[15-16] however, the fluoride ion is not believed to be the active species; rather the electronics of the silicon group is believed to be the driving force. Krutak demonstrated the remarkable stability of alkyl sulfonyl fluorides under aqueous conditions by generating SFx derivatives and their use in subsequent reactions.[17] The stability during the derivatization of the alkyl sulfonyl fluoride is suggestive of a fluoride-free deprotection. Krutak showcases the weak reactivity of the -SO_2F moiety by selectively functionalizing other positions in the presence of many functional groups.[17] Furthermore, Gembus et al. describes the high reactivity of sulfonyl fluorides towards silyl groups during their interconversions of silyl ethers to tosylates under basics conditions.[18] This chemistry is believed to follow a similar mechanism, in which the sulfonyl fluoride is weakly coordinated to the silicon-oxygen bond in a 2+2 fashion (Scheme 8). By coordinating, the silicon-oxygen bond is weakened and allows for nucleophilic attack by the protic solvent (i.e. water or propanol). It is postulated that the electron rich nature of the DPMS ether, due to the electron donation of the two phenyl rings, helps facilitate this coordination by stabilizing the electron deficient SO_2F group. The result leads to a free alcohol, a silanol byproduct and the sulfonyl fluoride untouched. In the future, DFT calculations could be performed to confirm if this type of coordination is energetically favorable.

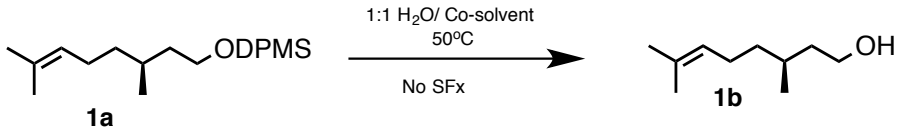
Scheme 8: *Proposed mechanism for SFx deprotection*



18-Crown-6 Ether Deprotection

Upon optimizing the co-solvent for the SFx deprotection, the ability of 18-crown-6 to deprotect DPMS ethers was discovered (Table 5). When testing the model substrate, **1a**, rapid deprotection was observed when utilizing a 1:1 mixture of water and 18-crown-6.

Table 5: *Control studies with various co-solvents*



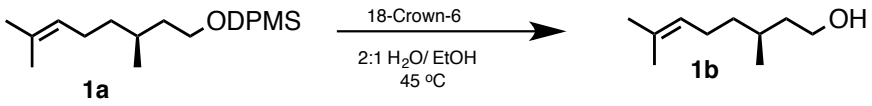
Entry	Co-solvent	Time (h)	Yield (%)[a]
1	None	24	0
2	PEG 200	24	0
3	PEG 400	24	0
4	12-Crown-4	24	71
5	15-Crown-5	24	56
6	**18-Crown-6**	**1.5**	**97**
7	t-Butanol	24	0
8	Propanol	24	0
9	Ethanol	24	0

[a] Isolated Yields
Reactions were ran at 0.25M

The investigation of 18-crown-6 was continued due to its apparent ability to participate in the reaction or facilitate the hydrolysis of the DPMS ether. To fully understand the effectiveness of 18-crown-6, an extensive screen of 18-crown-6 at various equivalents, temperatures, reaction times, and percent of co-solvent was completed (Table 6). As the amount of 18-crown-6 was reduced the reaction times increased. Upon further testing, suitable reaction times were achieved using a 2:1 ratio of water and ethanol. As the solvent system became optimized, stoichiometric amounts of 18-crown-6 were added and complete deprotection was observed

after four hours. Originally, 18-crown-6 was thought to aid in the solubility of the substrate; therefore, it was understandable that an increase in reaction time was consistent with a decrease in the amount of 18-crown-6. Because 18-crown-6 is a hazardous reagent, using catalytic amounts of 18-crown-6 was attractive. However, when sub-stoichiometric amounts of 18-crown-6 were utilized, substantially longer reaction times were observed, but close to complete deprotect was still possible at 0.5 equivalents and greater. Minimal conversion was observed at lower temperatures.

Table 6: *Preliminary screening of 18-crown-6 methodology*



1a **1b**

Entry	Crown (equiv.)	%EtOH	Co-Solvent	T (ºC)	Time	Yield (%)[a]
1	10	0		45	75 min	91
2	5	0		45	45 h	90
3	2	0		45	45 h	20
4	5	33		45	90 min	98
5	2	33		45	2 h	90
6	1	0		45	45 h	18
7	1	33		45	4 h	94
8	1	33		25	16 h	5
9	0.5	33		45	26 h	90
10	0.2	33		45	16 h	6

[a] Isolated Yields
Reactions were ran at 0.25M

As different co-solvents were screened, the use of a primary alcohol as a co-solvent proved to be crucial in this reaction. When solvents such as tetrahydrofuran or ethyl acetate were used, minimal conversion was observed after 24 hours. This gave rise to the question of what role does the primary alcohol play in this mechanism? It is assumed, the primary alcohol

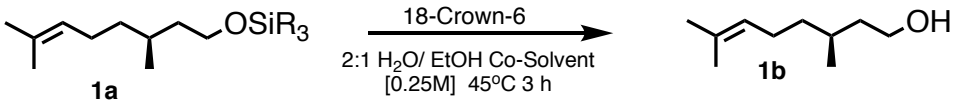
is acting as the nucleophile during the hydrolysis step, similar to the SFx mechanism described in the previous section. One may ask, why is water utilized as the primary solvent versus the surfactant technology developed by this group? Similar solvents systems as the SFx method were tried; however, the surfactant technology was incompatible with 18-crown-6 as it yielded significantly slower reaction times and lower conversion to the free alcohol. It is postulated that due to the extreme hydrophilic nature of 18-crown-6, it does not partition in and out of the micelles, resulting in minimal interaction between 18-crown-6 and the substrate. Therefore, a 2:1 mixture of water and ethanol was deemed the most effective solvent system for this chemistry.

An apparent mechanism for how 18-crown-6 facilitates the deprotection of the DPMS ether is not obvious. Exploration of the literature provided a greater understanding of the known properties and abilities of 18-crown-6. It was discovered that 18-crown-6 is known to accelerate various substitution reactions.[19] In addition, Friesen et al. described the phenomenon of silyl migration from one hydroxyl group to another.[20] Based on this, it was postulated that the oxygens of the 18-crown-6 are weakly coordinating to the electron-rich silicon, therefore, weakening the silicon-oxygen bond enough to initiate a nucleophilic attack by the protic solvent i.e. water or ethanol. It may also be argued that 18-crown-6 is acting as a mediator for the silyl migration of the DPMS ether to the more readily available alkyl alcohol in solution (i.e. ethanol).

Since 18-crown-6 is known for its ability to trap H^+ ions, the idea of trace acid being a factor in this selective deprotection needed to be ruled out. Trace acid was ruled out by referencing Davies et al. and his acid catalyzed hydrolysis of varying silyl ethers. Davies noted all silyl ethers they tested (TMS, TBS, DPMS, TIPS, and TBDPS) hydrolyzed in a 1% HCl/MeOH media.[21] This data confirmed that selectivity cannot be achieved when trace acid

is present. When TBS, TIPS and TBDPS were screened, selectivity towards the DPMS ether was observed. After 24 hours, the TIPS and TBDPS ethers remained untouched while only minimal conversion of the TBS ether was observed. These conditions could not discriminate between the TMS and DPMS ether for similar reasons stated earlier. As related to the SFx studies, allylic and propargyl silyl ethers were also screened for their selectivity (Table 8). The selectivity to the allylic and propargyl silyl ethers followed a similar trend as described for the SFx chemistry.

Table 7: *Chemo-selectivity of 18-crown-6 with various silyl ethers*

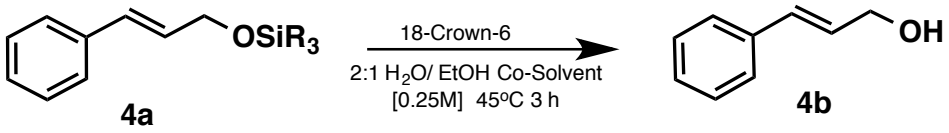


1a **1b**

Entry	Silyl Ether	Yield (%)[a]
1	OTMS	92
2	OTIPS	0 (0)[b]
3	ODPMS	89
4	OTBS	0 (5)[b]
5	OTBDPS	0 (0)[b]

[a] Isolated yields
[b] Isolated yields after 24 h

Table 8: *Chemo-selectivity of 18-crown-6 with various allylic silyl ethers*



4a **4b**

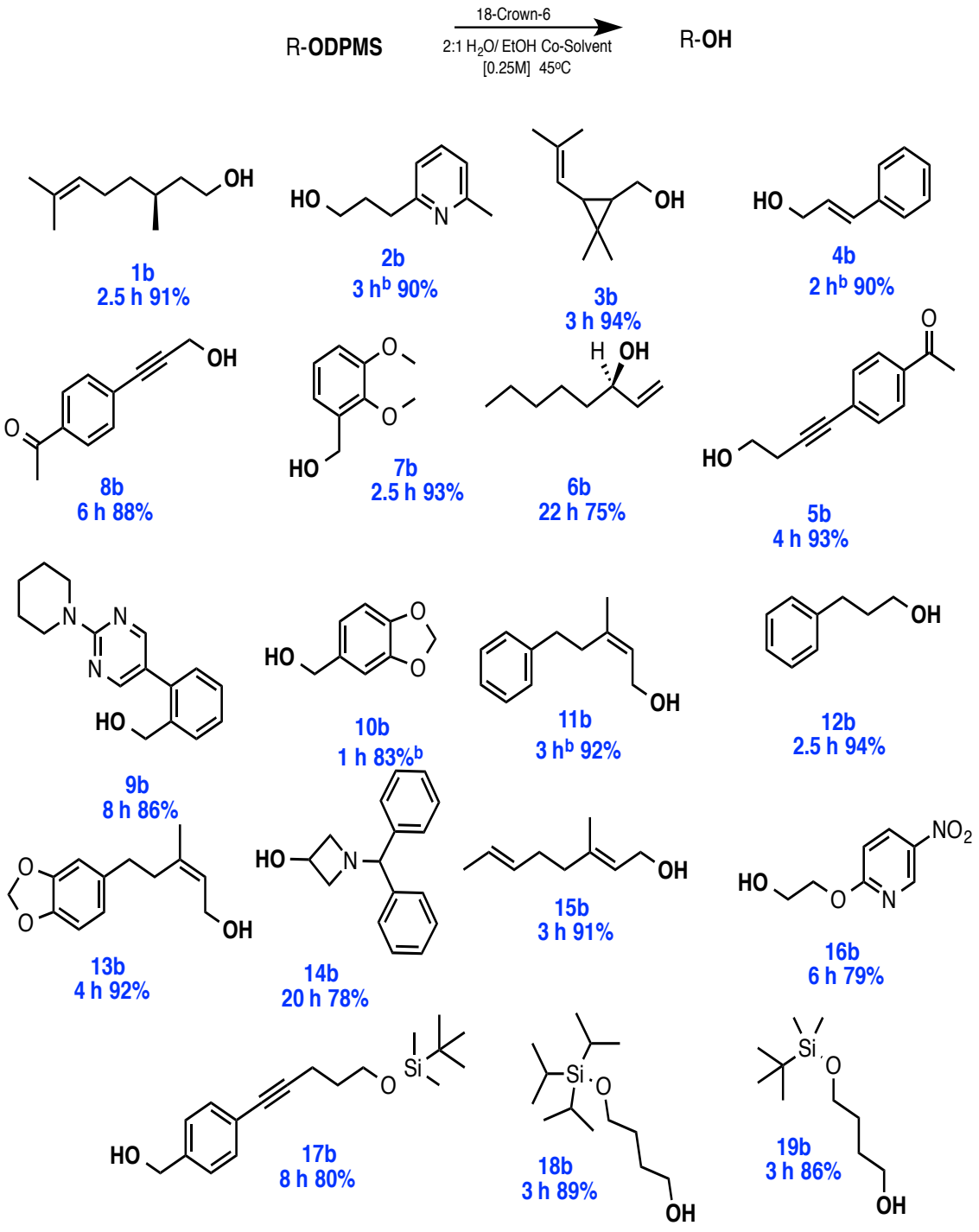
Entry	Silyl Ether	Yield (%)[a]
1	OTIPS	0
2	ODPMS	90
3	OTBS	0

[a] Isolated yields

As with the SFx conditions, aryl and vinyl alcohols as well as enol ethers proved to be nonreactive under these conditions; therefore, bolstering the selectivity of the 18-crown-6 method.

Results from this investigation allowed us to develop a robust substrate scope of mostly primary alcohols (Scheme 9). Faster reaction times were noted when comparing 18-crown-6 to the SFx scope.

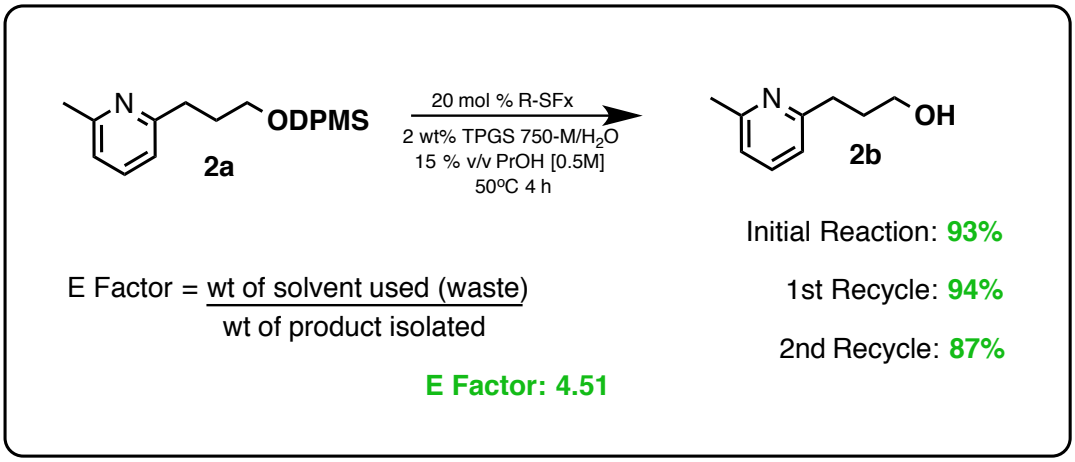
Scheme 9



b. Propanol used as alcohol

A recycling study of the TPGS-750-M reaction mixture was achieved by an in-flask extraction with a 15% ethyl acetate and hexane solution as extraction media (Scheme 10). However, due to the lipophilic nature of propanol, an addition of 15% by volume of propanol was necessary to gain similar reaction times. The yields and reaction times of the two recycles did not vary significantly, while the E factor, defined as the waste produced relative to the amount of product obtained in a chemical process, remained relatively low. The E factor obtained using the SFx method is drastically lower than other deprotection methods found in the literature.

Scheme 10



Conclusion

Two new chemo-selective deprotection methods for the DPMS ether were developed using catalytic amounts of perfluoro-1-butane sulfonyl fluoride or stoichiometric amounts of 18-crown-6. Both methods are simple by design and offer mild deprotection conditions. The cheap commercial availability and selectivity of these compounds make these new conditions affordable and promising. The exact role of 18-crown-6 in this reaction has not yet been fully elucidated and the mechanistic pathway is still under investigation. As it was stated for the SFx mechanism previously, DFT calculations could give more insight into the mechanistic pathway

of the 18-crown-6 method. The ability of 18-crown-6 and the perfluoro-1-butane sulfonyl fluoride to facilitate this type of chemistry may open the door to other methods not yet discovered.

To increase the depth of this study, an investigation regarding different sized crowns, such as 12-crown-4 or 15-crown-5, and their possible selectivity towards certain silyl ether groups could be completed. If different sized crowns are found to be selective towards specific silyl ether groups, then this type of chemistry could be an attractive method for selective deprotection of common silyl ethers due to its mild nature and affordable cost.

Two new sustainable methods for selective deprotection of DPMS Ethers under aqueous conditions

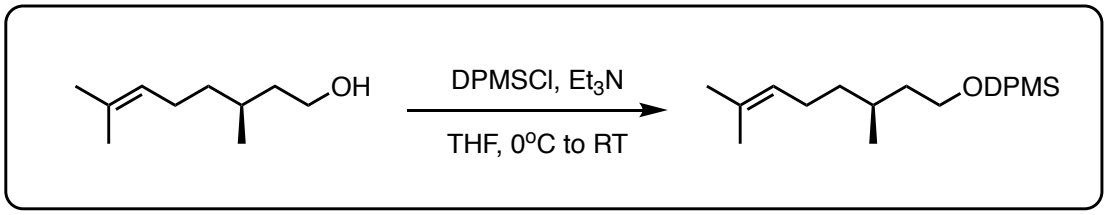
Experimental

1 General Information

A solution of 2 wt % TPGS-750-M/H_2O was prepared by dissolving TPGS-750-M in degassed HPLC grade water from Fisher Chemical. The resulting solution is stored under argon. The synthesis of TPGS-750-M has been described previously in detail and is available from Sigma-Aldrich (catalog #763896 (wax)). 18-crown-6 is commercially available from Sigma-Aldrich (catalog #186651). Perfluoro-1-butanesulfonyl fluoride is also commercially available from Sigma-Aldrich (catalog #319732). Certified Grade 1-propanol was purchased from Fisher Chemical. All commercially available starting alcohols were purchased from Sigma-Aldrich or Fisher Chemical. They were used without further purification.

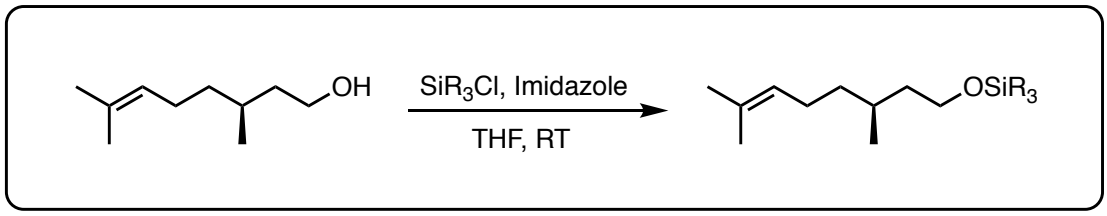
Silica Gel 60 F254 plates (Merck, 0.25nm) were used for thin layer chromatography (TLC). Flash Chromatrogaphy was done with either an Isolera™ One 3.0 Biotage or a standard glass column using Silica Gel 60 (EMD, 40-63µm). ^{1}H and ^{13}C NMR were recorded at 25°C on either a Varian Unity Inova 500MHz or a Varian Unity Inova 600MHz spectrometers in $CDCl_3$ with residual $CHCl_3$ (^{1}H = 7.26ppm, ^{13}C = 77.16ppm) as the internal standard. Chemical shifts are reported in parts per million (ppm). The data presented will be reported as follows; chemical shift, multiplicity (s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, quin = quintet, m = multiplet), coupling constant (if applicable) and integration. HRMS data were recorded on a Waters Micromass LCT TOF ES+ Premier mass spectrometer using ESI ionization.

2 General Procedure for the protection of DPMS ethers



All of the alcohols were protected using a modified literature procedure. A round bottom flask under argon was charged with diphenylmethylchlorosilane and THF was added. The resulting mixture was cooled to 0° C. Triethylamine was added to the flask followed by a slow addition of a solution of the alcohol in THF over a period of 3 minutes, resulting in a heterogenous mixture. The reaction was allowed to warm to room temperature. The reaction progress was monitored by TLC, and upon completion (3-12 h) the reaction solvent was evaporated under reduced pressure. The resulting crude mixture was diluted with ethyl ether and water. The aqueous layer was extracted with ethyl ether and the combined organic layers were washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure. The crude mixture was purified by flash chromatography.

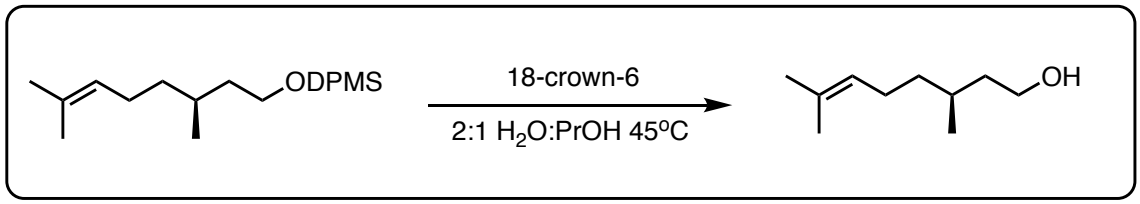
3 General Procedure for the protection of the various SiR_3 ethers



All of the other silyl ethers used for screening selectivity were synthesized using a modified literature procedure. A round bottom flask under argon was charged with silyl chloride and THF was added. To the resulting mixture was added imidazole followed by a solution of the alcohol in THF over a period of 3 minutes resulting in a heterogenous mixture. The reaction progress was monitored by TLC, and upon completion (1-6 h) the reaction solvent was evaporated under reduced pressure. The resulting crude mixture was diluted with ethyl ether and water. The aqueous layer was extracted with ethyl ether and the combined organic

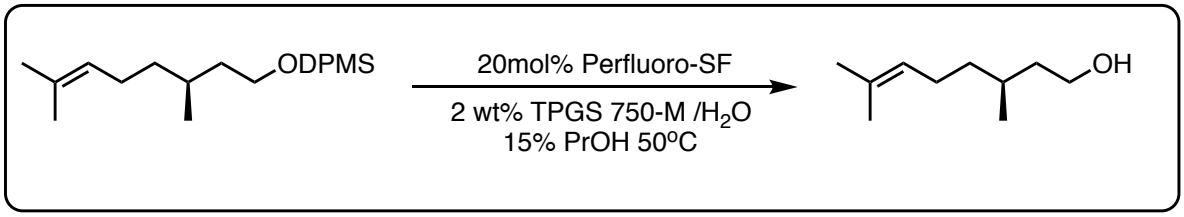
layers were washed with brine and dried over Na_2SO_4. The solvent was removed under reduced pressure. The crude mixture was purified by flash chromatography.

4 Utilization of 18-Crown-6 for DPMS ether deprotections



To a 1 dram reaction vial equipped with a magnetic stir bar was added 18-crown-6 (53 mg, 0.20 mmol, 1.00 equiv.). To this was added ethanol (0.27 mL) and HPLC water (0.53 mL). The mixture was stirred at 45°C giving a clear, homogenous solution. To this was added **1** (70.5 mg, 0.20 mmol, 1.00 equiv.) dropwise *via* microliter syringe, resulting in a heterogenous mixture. The reaction progress was monitored by TLC, and upon complete consumption of starting material was brought to room temperature, then diluted with ethyl ether (2.00 mL). This was shaken and allowed to separate, and the organic layer removed via pipet and filtered over a pad of anhydrous Na_2SO_4 into a round-bottomed-flask. This extraction was repeated an additional two times, and the combined organics were concentrated *in vacuo* to give a crude oil. This was purified by column chromatography to give the pure product.

5 Utilization of Perfluoro-1-butanesulfonyl Fluoride for DPMS ether deprotections



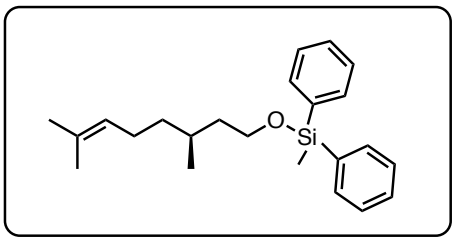
To a 1 dram reaction vial equipped with a magnetic stir bar was added the DPMS ether. To this was added a solution of 2 wt% TPGS 750-M/H_2O (0.85 mL) and Propanol (0.15 mL). Perfluoro-1-butanesulfonyl fluoride was added via a microliter syringe, resulting in a heterogeneous mixture. The reaction progress was monitored by TLC. Upon complete consumption of starting material (6-24 h), the reaction was allowed to cool to room temperature, then diluted with ethyl ether (2.0 mL). The vile was shaken and the solution was allowed to separate. This extraction was repeated an additional two times and the combined organics were concentrated *in vacuo* to give a crude oil. This was purified by column chromatography to give the pure product.

6 Recycle study

The initial reaction was set up according to the general procedure stated above. Upon completion of the reaction, the reaction mixture was extracted three times with MTBE (1 mL total). The organic extractions were placed in a flask and reduced under pressure. The crude product was purified via a plug of silica with a mixture of EtOAc/Hexanes to provide the desired product. The surfactant solution was then charged with additional DPMS alcohol (0.25 mmol) and 15% by volume of propanol. The reaction was sealed and set to stir at 50 ^{0}C. according to the general procedure.

7 Analytical Data for synthesized silyl ether protected products

(*S*)-((3,7-Dimethyloct-6-en-1-yl)oxy(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62-7.59 (dd, 4H), 7.44-7.36 (m, 6H), 5.11-5.06 (m, J = 8.5Hz 1H), 3.79-3.69(m, 2H), 2.04-1.87 (m, 2H), 1.7 (s, 3H) 1.65-1.55 (m, 2H), 1.60 (s, 3H) 1.44-1.36 (m, 1H) 1.34-1.26 (m, 1H) 1.18-1.10 (m, J = 7.6Hz 1H) 0.85 (d, J = 6.6Hz 3H) 0.65 (s, 3H).

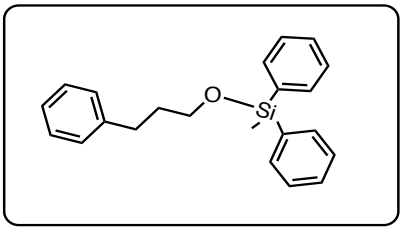
^{13}C NMR (126 MHz, $CDCl_3$) δ 136.45, 134.47, 131.20, 129.86, 127.95, 127.85, 125.00, 61.92, 39.79, 37.30, 29.25, 25.86, 25.59, 19.68, 17.78, -2.88.

Yield: 89% colorless liquid.

R_f: 0.8 (25% Et_2O in hexanes).

HRMS for: $C_{23}H_{32}OSi$ EI-MS $[M^+]$ calcd: 352.2222; found: 337.1988 $[M\text{-}CH_3]^+$

Methyldiphenyl(3-phenylpropoxy)silane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65-7.52 (dd, 4H), 7.47-7.39 (m, 6H), 7.30-7.27 (t, 2H), 7.21-7.17 (m, 3H), 3.78-3.75 (t, 2H), 2.75-2.71 (t, *J* = 7.7Hz, 2H), 1.96-1.89 (m, 2H) 0.69 (s, 3H) .

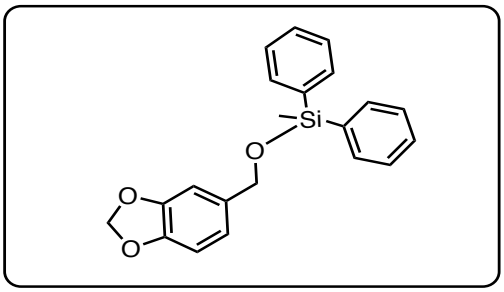
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.19, 136.34, 134.48, 129.91, 128.58, 128.40, 128.00, 127.98, 125.81, 62.86, 34.26, 32.22, -2.90.

Yield: 87% colorless liquid.

R_f: 0.8 (25% Et_2O in hexanes).

HRMS for $C_{22}H_{24}OSi$ EI-MS $[M^+]$ calcd: 332.1596; found: 317.1361 $[M\text{-}CH_3]^+$

(Benzo[d][1,3]dioxol-5-ylmethoxy)(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63-7.61 (dd, J = 6.4Hz, 4H), 7.45-7.37 (m, 6H), 6.84 (s, J = 0.9Hz, 1H), 6.75 (d, J = 1.0Hz, 2H), 5.94 (s, 2H), 4.69 (s, J = 0.7Hz, 2H) 0.69 (s, 3H).

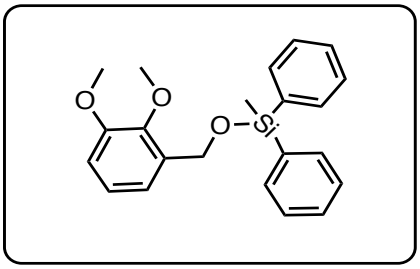
^{13}C NMR (126 MHz, $CDCl_3$) δ 147.75, 146.81, 135.93, 134.76, 134.54, 130.05, 128.05, 120.03, 108.12, 107.69, 101.02, 65.39, -2.74.

Yield: 83% colorless liquid.

R_f: 0.3 (10% Et_2O in hexanes).

HRMS for $C_{21}H_{20}O_3Si$ EI-MS [M^+] calcd: 348.1181; found: 348.1182

((2,3-Dimethoxybenzyl)oxy)(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63-7.61 (dd, 4H), 7.45-7.37 (m, 6H), 7.14-7.13 (d, 1H), 7.09-7.05 (t, 1H) 6.86-6.85 (d, 1H) 4.87 (s, J = 1.3Hz, 2H), 3.86 (s, 3H), 3.75 (s, J = 1.0Hz,3H), 0.70 (s, 3H).

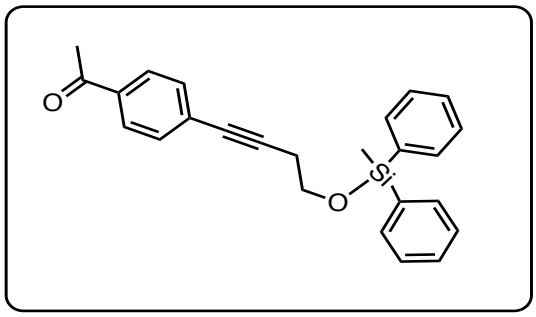
^{13}C NMR (126 MHz, $CDCl_3$) δ 152.43, 136.11, 134.74, 134.54, 129.97, 128.00, 124.07, 120.14, 111.53, 60.75, 60.65, 55.92, -2.86.

Yield: 96% colorless oil.

R_f: 0.33 (15% Et_2O in hexanes).

HRMS for $C_{22}H_{24}O_3Si$ EI-MS [M^+] calcd: 364.1494; found: 364.149

1-(4-(4-((Methyldiphenylsilyl)oxy)but-1-yn-1-yl)phenyl)ethan-1-one



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 – 7.84 (m, 2H), 7.62 – 7.59 (m, 4H), 7.43 – 7.34 (m, 8H), 3.91 – 3.88 (t, 2H), 2.70 (t, *J* = 6.9 Hz, 2H), 2.57 (s, *J* = 0.5 Hz, 3H), 0.67 (s, *J* = 0.6 Hz, 3H).

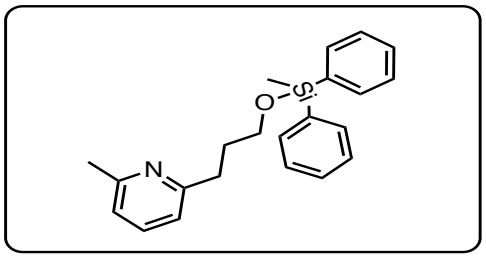
^{13}C NMR (126 MHz, $CDCl_3$) δ 197.50, 136.01, 135.84, 134.51, 134.49, 134.09, 131.86, 130.10, 130.03, 128.85, 128.29, 128.07, 91.10, 81.35, 62.01, 26.73, 23.87, -2.84.

Yield: 76% yellow oil.

R_f: 0.27 (15% EtOAc in hexanes).

HRMS for $C_{25}H_{24}O_2Si$ EI-MS [M^+] calcd: 384.1545; found: 384.1546

2-Methyl-6-(3-((methyldiphenylsilyl)oxy)propyl)pyridine



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 – 7.59 (m, 4H), 7.47 – 7.36 (m, 7H), 6.95 (d, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 7.6 Hz, 1H), 3.77 (t, *J* = 6.4 Hz, 2H), 2.87 – 2.82 (m, 2H), 2.52 (s, 3H), 2.05 – 1.98 (m, 2H), 0.65 (s, 3H).

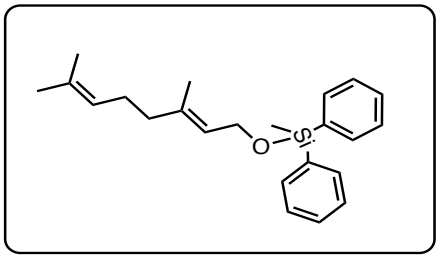
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.32, 157.86, 136.57, 136.34, 134.49, 129.89, 127.96, 120.54, 119.70, 63.07, 34.90, 32.88, 24.69, -2.91.

Yield: 91% brown oil.

R_f: 0.34 (15% EtOAc in hexanes).

HRMS for $C_{22}H_{25}ONSi$ EI-MS [M^+] calcd: 347.1705; found: 347.1711

(*E*)-((3,7-Dimethylocta-2,6-dien-1-yl)oxy)(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 (dd, *J* = 6.3, 1.3 Hz, 4H), 7.45 – 7.36 (m, 6H), 5.42 – 5.37 (m, 1H), 5.06 – 5.01 (m, 1H), 4.24 (dt, *J* = 6.7, 1.1 Hz, 2H), 1.97 (tq, *J* = 9.7, 5.0, 3.6 Hz, 4H), 1.71 (s, *J* = 1.2 Hz, 3H), 1.65 (s, *J* = 1.5 Hz, 3H), 1.55 (s, *J* = 3.0 Hz, 3H), 0.66 (s, *J* = 0.9 Hz, 3H).

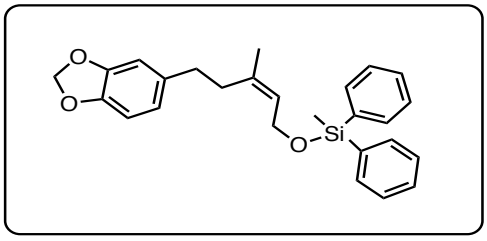
^{13}C NMR (126 MHz, $CDCl_3$) δ 138.46, 136.29, 134.57, 131.97, 129.88, 127.96, 124.61, 124.04, 60.27, 32.31, 26.78, 25.80, 23.57, 17.76, -2.59.

Yield: 81% colorless oil.

R_f: 0.35 (10% Et_2O in hexanes).

HRMS for $C_{23}H_{30}OSi$ EI-MS [M^+] calcd: 350.2065; found: 350.2066

(*Z*)-((5-(Benzo[1,3]dioxol-5-yl)-3-methylpent-2-en-1-yl)oxy)(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 4H), 7.39 (m, *J* = 12.5, 7.6, 5.9 Hz, 6H), 6.72 (d, *J* = 7.8 Hz, 1H), 6.66 (d, *J* = 1.7 Hz, 1H), 6.60 (dd, *J* = 8.0, 1.6 Hz, 1H), 5.91 (s, 2H), 5.38 (td, *J* = 6.5, 1.5 Hz, 1H), 4.26 (d, *J* = 6.5 Hz, 2H), 2.62 – 2.58 (m, 2H), 2.22 (dd, *J* = 9.7, 6.5 Hz, 2H), 1.55 (s, 3H), 0.64 (s, 3H).

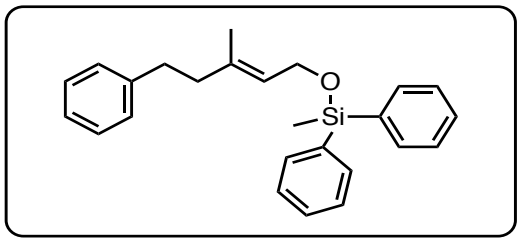
^{13}C NMR (126 MHz, $CDCl_3$) δ 147.62, 145.68, 137.39, 136.28, 136.15, 134.56, 129.91, 127.96, 124.22, 121.14, 108.95, 108.23, 100.86, 60.59, 41.77, 34.15, 16.58, -2.59.

Yield: 82% colorless oil.

R_f: 0.32 (5% EtOAc in hexanes).

HRMS for $C_{26}H_{28}O_3Si$ EI-MS [M^+] calcd: 416.1807; found: 439.1700 [M+Na]$^+$

(*E*)-Methyl(3-methyl-5-phenylpent-2-en-1-yl)oxy)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 6.4, 1.6 Hz, 3H), 7.55 – 7.53 (m, 1H), 7.43 – 7.38 (m, 6H), 7.35 – 7.29 (m, 3H), 7.20 (m, *J* = 9.6, 7.7, 1.3 Hz, 3H), 5.42 (m, *J* = 5.3, 2.6, 1.3 Hz, 1H), 4.30 – 4.27 (d, 2H), 2.70 (t, *J* = 9.6, 6.9 Hz, 2H), 2.30 – 2.27 (d, 2H), 1.59 (s, *J* = 1.5 Hz, 3H), 0.66 (s, *J* = 1.2 Hz, 3H).

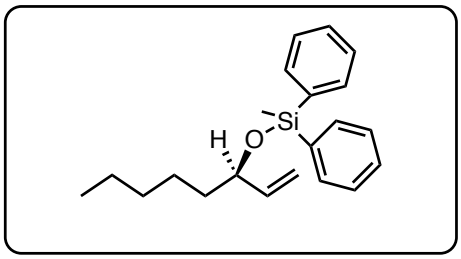
^{13}C NMR (126 MHz, $CDCl_3$) 142.32, 137.55, 136.33, 134.57, 134.55, 134.15, 129.90, 129.71, 128.44, 127.97, 127.87, 125.90, 124.16, 60.62, 41.51, 34.45, 16.61, -2.57.

Yield: 78% colorless oil.

R_f: 0.4 (10% Et_2O in hexanes).

HRMS for $C_{25}H_{28}O_3Si$ EI-MS [M^+] calcd: 404.1807; found: 357.1917 [$M-CH_3$]$^+$

(*R*)-Methyl(oct-1-en-3-yloxy)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.60 (m, *J* = 6.7, 5.2, 1.5 Hz, 4H), 7.41 – 7.34 (m, 6H), 5.83 (ddd, *J* = 17.0, 10.4, 6.5 Hz, 1H), 5.08 (dt, *J* = 17.2, 1.5 Hz, 1H), 5.01 (ddd, *J* = 10.4, 1.7, 1.1 Hz, 1H), 4.20 – 4.15 (m, 1H), 1.61 – 1.55 (m, 1H), 1.51 – 1.45 (m, 1H), 1.34 – 1.15 (m, 7H), 0.84 (t, *J* = 7.1 Hz, 3H), 0.65 (s, 3H).

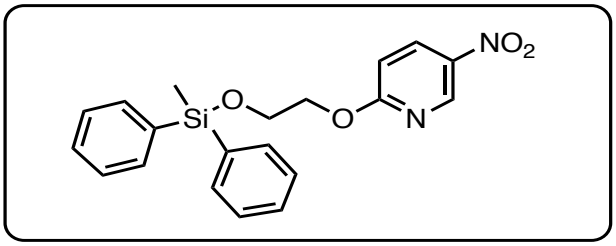
^{13}C NMR (126 MHz, $CDCl_3$) δ 141.41, 136.94, 136.86, 134.69, 134.63, 129.86, 129.83, 127.91, 127.89, 114.42, 74.84, 37.98, 31.93, 24.93, 22.79, 14.23, -1.97.

Yield: 89% colorless liquid.

R_f: 0.42 (hexanes).

HRMS for $C_{21}H_{28}OSi$ EI-MS [M^+] calcd: 324.1909; found: 324.1909

2-(2-((Methyldiphenylsilyl)oxy)ethoxy)-5-nitropyridine



^{1}H NMR (500 MHz, $CDCl_3$) δ 9.01 (d, *J* = 2.8 Hz, 1H), 8.31 (dd, *J* = 9.1, 2.8 Hz, 1H), 7.60 – 7.57 (m, 4H), 7.43 – 7.39 (m, 2H), 7.38 – 7.34 (m, 4H), 6.73 (d, *J* = 9.1 Hz, 1H), 4.57 – 4.54 (m, 2H), 4.08 – 4.06 (m, 2H), 0.67 (s, 3H).

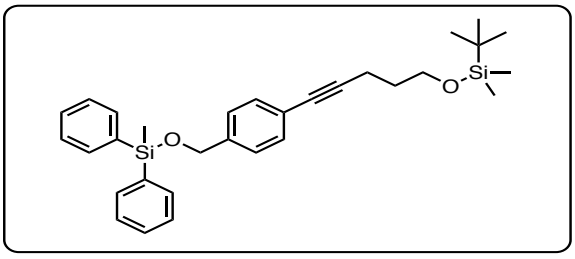
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.06, 144.81, 135.74, 134.48, 133.97, 130.10, 128.04, 111.54, 77.41, 77.16, 76.91, 68.68, 61.87, -2.82.

Yield: 79% yellow oil.

R_f: 0.35 (25% EtOAc in hexanes).

HRMS for $C_{20}H_{20}O_4N_2Si$ EI-MS [M^+] calcd: 380.1192; found: 365.0958 [$M-CH_3$]$^+$

***Tert*-butyldimethyl((5-(4-(((methyldiphenylsilyl)oxy)methyl)phenyl)pent-4-yn-1-yl)oxy)silane**



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 – 7.59 (m, 4H), 7.44 – 7.41 (m, 2H), 7.40 – 7.36 (m, 4H), 7.35 – 7.33 (d, 2H), 7.23 (d, *J* = 7.5, 0.8 Hz, 2H), 4.77 (s, 2H), 3.76 (t, *J* = 6.0 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 1.83 – 1.78 (m, 2H), 0.91 (s, 9H), 0.65 (s, 3H), 0.08 (s, 6H).

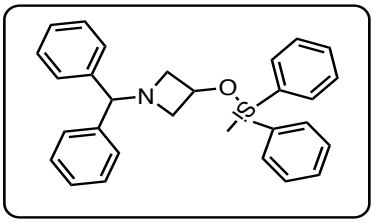
^{13}C NMR (126 MHz, $CDCl_3$) δ 140.21, 135.84, 134.53, 131.58, 130.08, 128.07, 126.39, 105.16, 89.72, 80.81, 77.41, 77.16, 76.90, 65.15, 61.80, 31.93, 26.12, 15.99, -2.78, -5.14.

Yield: 94% colorless oil.

R_f: 0.32 (4% EtOAc in hexanes).

HRMS for $C_{31}H_{40}O_2Si_2$ EI-MS [M^+] calcd: 500.2566; found: 523.2465 [M+Na]$^+$

1-Benzhydryl-3-((methyldiphenylsilyl)oxy)azetidine



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.4 Hz, 4H), 7.39 (dd, *J* = 18.0, 7.2 Hz, 11H), 7.27 (s, 2H), 7.24 (s, 1H), 7.18 (t, *J* = 7.3 Hz, 2H), 4.53 (p, *J* = 6.2 Hz, 1H), 4.35 (s, 1H), 3.47 (td, *J* = 6.2, 2.3 Hz, 2H), 2.95 (td, *J* = 6.3, 2.3 Hz, 2H), 0.60 (s, 3H).

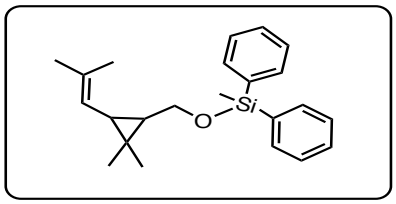
^{13}C NMR (126 MHz, $CDCl_3$) δ 142.40, 135.74, 134.39, 130.09, 128.52, 128.48, 128.05, 127.58, 127.19, 78.62, 63.55, 62.39, -2.48.

Yield: 77% colorless oil.

R_f: 0.32 (15% Et_2O in hexanes).

HRMS for $C_{29}H_{29}ONSi$ EI-MS [M^+] calcd: 435.2018; found: 435.2018

((2,2-Dimethyl-3-(2-methylprop-1-en-1-yl)cyclopropyl)methoxy)(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) major isomer reported **δ** 7.61 – 7.59 (m, 4H), 7.40 – 7.36 (m, 6H), 4.85 (ddq, *J* = 9.7, 7.1, 1.4 Hz, 2H), 3.89 (ddd, *J* = 11.2, 6.2, 1.4 Hz, 1H), 3.72 (ddd, *J* = 7.5, 4.0, 1.4 Hz, 1H), 3.63 (ddd, *J* = 11.2, 8.3, 1.3 Hz, 1H), 1.69 (d, *J* = 1.7 Hz, 3H), 1.64 (d, *J* = 1.3 Hz, 3H), 1.07 (d, *J* = 1.4 Hz, 3H), 1.01 (d, *J* = 1.3 Hz, 3H), 0.81 (ddd, *J* = 8.3, 4.2, 1.3 Hz, 1H), 0.64 (d, *J* = 1.4 Hz, 3H).

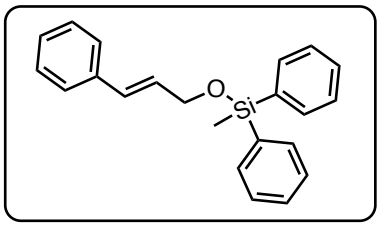
^{13}C NMR (126 MHz, $CDCl_3$) mixture of isomers 136.58, 134.66, 134.55, 134.53, 134.14, 132.84, 129.82, 129.80, 127.92, 127.87, 123.89, 119.58, 77.41, 77.16, 76.91, 64.38, 61.30, 34.99, 30.81, 28.95, 28.68, 26.31, 25.86, 25.78, 22.78, 22.45, 21.57, 18.61, 18.39, 15.62, -2.65, -2.71.

Yield: 85% colorless oil.

R_f: 0.65 (10% EtOAc in hexanes).

HRMS for $C_{23}H_{30}OSi$ EI-MS [M^+] calcd: 350.2065; found: 350.2076

(Cinnamyloxy)(methyl)diphenylsilane



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 – 7.62 (m, 4H), 7.43 – 7.41 (m, 2H), 7.40 – 7.38 (m, 4H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.31 – 7.29 (m, 2H), 7.22 (td, *J* = 7.0, 1.5 Hz, 1H), 6.58 (dd, *J* = 15.9, 1.8 Hz, 1H), 6.31 – 6.27 (m, 1H), 4.42 (dd, *J* = 5.5, 1.8 Hz, 2H), 0.69 (s, *J* = 1.5 Hz, 3H).

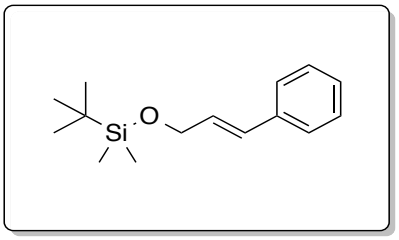
^{13}C NMR (151 MHz, $CDCl_3$) δ 139.76, 138.65, 137.20, 133.09, 132.70, 131.29, 131.19, 130.71, 130.21, 129.23, 67.06, -0.00.

Yield: 85% colorless oil.

R_f: 0.33 (15% Et_2O in hexanes).

HRMS for $C_{22}H_{22}OSi$ EI-MS [M^+] calcd: 330.1440; found: 330.1425

***Tert*-butyl(cinnamyloxy)dimethylsilane**



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.34 (m, 2H), 7.31 – 7.26 (m, 2H), 7.22 – 7.18 (m, 1H), 6.57 (dd, *J* = 15.7, 2.0 Hz, 1H), 6.27 (dtd, *J* = 15.9, 5.1, 0.9 Hz, 1H), 4.34 (dt, *J* = 5.1, 1.2 Hz, 2H), 0.93 (d, *J* = 0.9 Hz, 9H), 0.10 (d, *J* = 0.9 Hz, 6H).

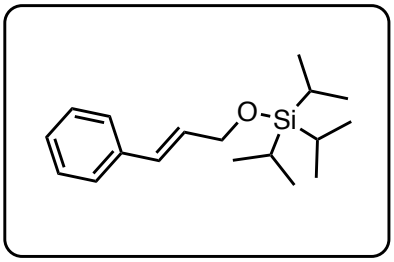
^{13}C NMR (126 MHz, $CDCl_3$) δ 129.46, 129.18, 128.49, 127.29, 126.37, 63.89, 25.98, 18.47, -5.13.

Yield: 75% colorless oil.

R_f: 0.3 (15% Et_2O in hexanes).

HRMS for $C_{15}H_{24}OSi$ EI-MS [M^+] calcd: 248.1596; found: 248.1596

(Cinnamyloxy)triisopropylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.37 (m, 2H), 7.33 – 7.29 (m, 2H), 7.24 – 7.20 (m, 1H), 6.65 (dd, *J* = 15.7, 2.1 Hz, 1H), 6.33 – 6.27 (m, 1H), 4.44 (dd, *J* = 4.8, 1.9, 0.8 Hz, 2H), 1.20 – 1.14 (m, 3H), 1.12 – 1.09 (d, 18H).

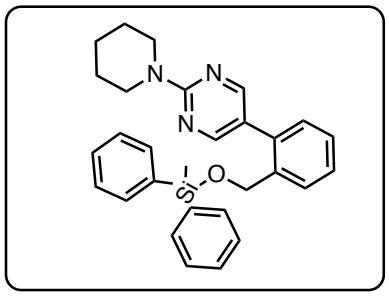
^{13}C NMR (126 MHz, $CDCl_3$) δ 137.41, 129.52, 129.19, 128.63, 127.35, 126.52, 64.07, 18.20, 12.24.

Yield: 89% colorless oil.

R_f: 0.75 (5% EtOAc in hexanes)

HRMS for $C_{13}H_{30}OSi$ EI-MS [M^+] calcd. 290.2066 found 290.2065

5-(2-(((Methyldiphenylsilyl)oxy)methyl)phenyl)-2-(piperidin-1-yl)pyrimidine



^{1}H NMR (600 MHz, $CDCl_3$) δ 8.26 (s, 2H), 7.54 (ddd, *J* = 6.8, 4.0, 1.6 Hz, 5H), 7.40 – 7.36 (m, 2H), 7.33 (qd, *J* = 7.1, 1.3 Hz, 6H), 7.15 (dd, *J* = 7.2, 1.7 Hz, 1H), 4.68 (s, 2H), 3.82 – 3.79 (m, 4H), 1.71 – 1.67 (m, 2H), 1.65 – 1.61 (m, 4H), 0.60 (s, 3H).

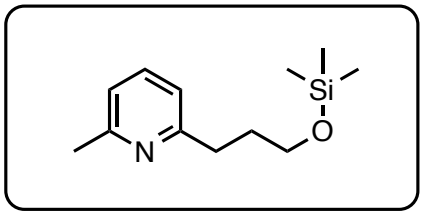
^{13}C NMR (126 MHz, $CDCl_3$) δ 160.83, 157.40, 138.38, 135.72, 135.10, 134.39, 129.91, 129.89, 128.72, 127.92, 127.88, 127.82, 127.70, 121.75, 63.34, 44.94, 25.83, 24.93, -2.94.

Yield: 85% colorless oil.

R_f: 0.30 (7.5% EtOAc in hexanes).

HRMS for $C_{29}H_{31}ON_3Si$ EI-MS [M^+] calcd: 465.2236; found: 466.2315 $[M+H]^+$

2-Methyl-6-(3-((trimethylsilyl)oxy)propyl)pyridine



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (t, *J* = 7.7 Hz, 1H), 6.85 (dd, *J* = 7.7, 2.7 Hz, 2H), 3.53 (t, *J* = 6.5 Hz, 2H), 2.71 – 2.67 (m, 2H), 2.41 (s, 3H), 1.86 – 1.82 (m, 2H), -0.01 (s, 9H).

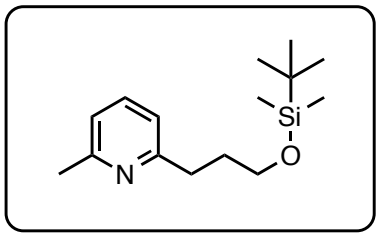
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.70, 158.17, 136.92, 120.88, 120.00, 62.53, 35.24, 33.31, 26.44, 24.98, -0.00.

Yield: 86% light yellow oil.

R_f: 0.34 (10% EtOAc in hexanes).

HRMS for $C_{12}H_{21}NOSi$ EI-MS [M^+] calcd; 223.1392 found; 223.1391

2-Methyl-6-(3-((tert-butyldimethylsilyl)oxy)propyl)pyridine



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (t, *J* = 7.6 Hz, 1H), 6.95 (dd, *J* = 7.6, 2.2 Hz, 2H), 3.66 (t, *J* = 6.4 Hz, 2H), 2.83 – 2.79 (m, 2H), 2.52 (s, 3H), 1.96 – 1.90 (m, 2H), 0.90 (s, 9H), 0.04 (s, 6H).

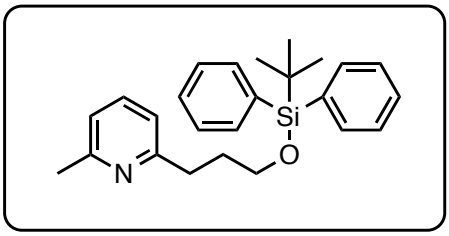
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.72, 158.19, 136.90, 120.86, 119.98, 62.55, 35.25, 33.31, 25.00, -0.00.

Yield: 89% light yellow oil.

R_f: 0.36 (10% EtOAc in hexanes).

HRMS for $C_{15}H_{27}NOSi$ EI-MS [M^+] calcd: 265.1862; found: 265.1867

2-Methyl-6-(3-((tert-butyldiphenylsilyl)oxy)propyl)pyridine



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 – 7.68 (m, 1H), 7.65 – 7.63 (m, 4H), 7.38 – 7.33 (m, 6H), 7.24 (s, 1H), 6.91 (dd, *J* = 14.2, 7.6 Hz, 2H), 3.70 (t, *J* = 6.3 Hz, 2H), 2.85 – 2.82 (m, 2H), 2.49 (s, 3H), 1.99 – 1.94 (m, 2H), 1.04 (s, 9H).

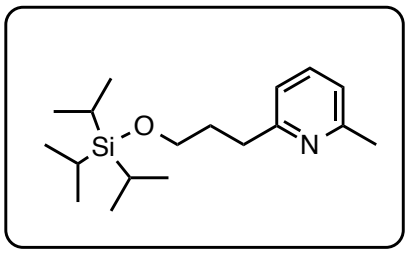
^{13}C NMR (151 MHz, $CDCl_3$) δ 161.27, 157.68, 136.42, 135.54, 134.77, 133.95, 129.59, 129.48, 127.67, 127.56, 120.38, 119.57, 63.30, 34.70, 32.75, 26.85, 24.49, 19.21.

Yield: 83% light yellow oil.

R_f: 0.32 (10% EtOAc in hexanes).

HRMS for $C_{25}H_{31}NOSi$ EI-MS [M^+] calcd: 389.2175; found: 389.2171

2-Methyl-6-(3-((triisopropylsilyl)oxy)propyl)pyridine



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (t, *J* = 7.6 Hz, 1H), 6.93 (dd, *J* = 9.8, 7.7 Hz, 2H), 3.71 (t, *J* = 6.4 Hz, 2H), 2.83 – 2.79 (m, 2H), 2.50 (s, 3H), 1.96 – 1.90 (m, 2H), 1.03 (s, 9H).

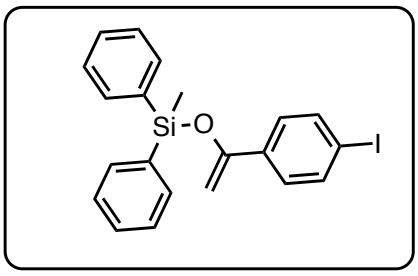
^{13}C NMR (126 MHz, $CDCl_3$) δ 161.45, 157.68, 136.42, 120.36, 119.62, 62.78, 34.75, 33.23, 24.49, 18.03, 12.01.

Yield: 90% light yellow oil.

Rf: 0.28 (10% EtOAc in hexanes).

HRMS for $C_{18}H_{33}ONSi$ EI-MS [M^+] calcd: 307.2332; found: 307.2344

((1-(4-Iodophenyl)vinyl)oxy)(methyl)diphenylsilane



^{1}H NMR (600 MHz, C_6D_6) δ 7.65 – 7.63 (m, 4H), 7.40 – 7.37 (m, 2H), 7.21 – 7.19 (m, 2H), 7.19 – 7.17 (m, 7H), 4.71 (d, *J* = 2.2 Hz, 1H), 4.41 (d, *J* = 2.2 Hz, 1H), 0.65 (s, 3H).

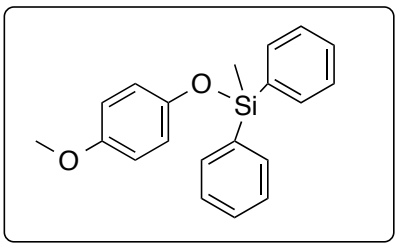
^{13}C NMR (151 MHz, C_6D_6) δ 154.70, 137.22, 135.25, 134.26, 130.03, 127.95, 127.00, 94.03, 92.35, -3.24.

Yield: 78% colorless oil.

R_f: 0.3 (5% Et_2O in hexanes).

HRMS for $C_{21}H_{19}OISi$ EI-MS [M^+] calcd: 442.0249; found: 442.0233

4-((Methyldiphenylsilyl)oxy)anisole



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.65 (m, 4H), 7.46 – 7.39 (m, 6H), 6.79 – 6.71 (m, 4H), 3.73 (s, 3H), 0.75 (s, 3H).

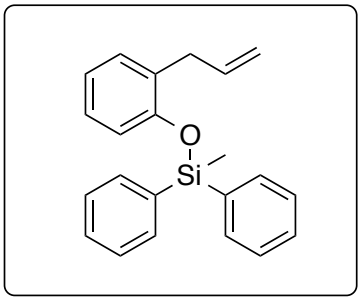
^{13}C NMR (151 MHz, $CDCl_3$) δ 155.89, 150.41, 137.22, 136.04, 131.73, 129.62, 122.19, 116.12, 57.21, -1.01.

Yield: 83% colorless oil.

R_f: 0.31 (5% EtOAc in hexanes)

HRMS for $C_{20}H_{20}O_2Si$ EI-MS [M^+] calcd: 320.1232; found: 320.1235

(2-Allylphenoxy)(methyl)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (dt, *J* = 6.6, 1.5 Hz, 4H), 7.47 – 7.37 (m, 6H), 7.14 (dd, *J* = 7.5, 1.7 Hz, 1H), 6.97 (td, *J* = 7.7, 1.8 Hz, 1H), 6.89 (td, *J* = 7.4, 1.3 Hz, 1H), 6.69 (dd, *J* = 8.0, 1.3 Hz, 1H), 5.97 (ddtd, *J* = 16.8, 10.2, 6.6, 1.4 Hz, 1H), 5.05 – 4.99 (m, 2H), 3.42 (dt, *J* = 6.7, 1.5 Hz, 2H), 0.76 (s, 3H).

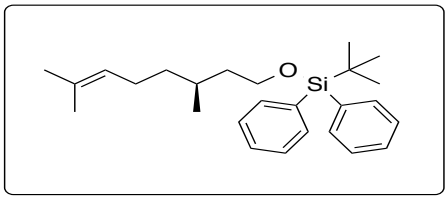
^{13}C NMR (151 MHz, $CDCl_3$) δ 155.89, 150.41, 137.22, 136.04, 131.73, 129.62, 122.19, 116.12, 57.21, -1.01.

Yield: 76% colorless oil.

R_f: 0.3 (5% Et_2O in hexanes).

HRMS for $C_{22}H_{22}OSi$ EI-MS [M^+] calcd: 330.1439; found: 330.1431

(*S*)-*Tert*-butyl-((3,7-dimethyloct-6-en-1-yl)oxy)diphenylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (dd, *J* = 7.9, 1.6 Hz, 4H), 7.43 – 7.36 (m, 6H), 5.09 (dddd, *J* = 7.1, 5.7, 2.9, 1.4 Hz, 1H), 3.73 – 3.67 (m, 2H), 1.99 – 1.92 (m, 2H), 1.68 (q, *J* = 1.3 Hz, 3H), 1.64 – 1.59 (m, 5H), 1.38 – 1.29 (m, 2H), 1.16 – 1.11 (m, 1H), 1.05 (s, 9H), 0.84 (d, *J* = 6.5 Hz, 3H).

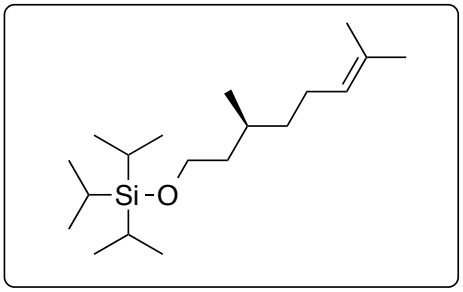
^{13}C NMR (151 MHz, $CDCl_3$) δ 135.57, 134.17, 131.03, 129.47, 127.56, 124.91, 62.20, 39.63, 37.17, 29.05, 26.87, 25.72, 25.49, 19.61, 19.21, 17.65.

Yield: 68% colorless oil.

R_f: 0.75 (10% EtOAc in hexanes)

HRMS for $C_{26}H_{38}OSi$ EI-MS [M^+] calcd: 394.2692 found: 394.2692

(*S*)-*Tert*-butyl-((3,7-dimethyloct-6-en-1-yl)oxy)triisopropylsilane



^{1}H NMR (600 MHz, $CDCl_3$) δ 5.10 (m, J = 7.0, 1.4 Hz, 1H), 3.74 – 3.68 (m, 2H), 2.02 – 1.93 (m, 2H), 1.68 (d, *J* = 1.4 Hz, 3H), 1.61 – 1.57 (m, 5H), 1.37 – 1.31 (m, 2H), 1.16 (dtd, *J* = 9.5, 5.9, 5.1, 2.1 Hz, 1H), 1.07 – 1.05 (m, 19H), 0.89 (d, *J* = 6.5 Hz, 3H).

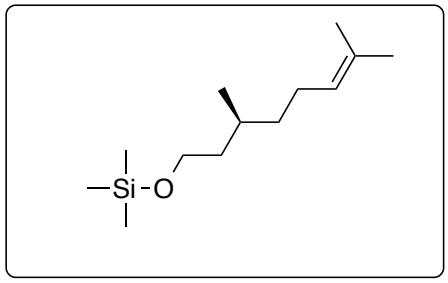
^{13}C NMR (151 MHz, $CDCl_3$) δ 131.19, 125.08, 105.18, 61.84, 40.24, 37.41, 29.30, 25.87, 25.67, 19.85, 18.20, 17.78, 12.19.

Yield: 94% colorless oil.

R_f: 0.75 (10% EtOAc in hexanes).

HRMS for $C_{19}H_{40}OSi$ EI-MS [M^+] calcd 312.6130; found: 269.2297 [$M-C_3H_7$]$^+$

(*S*)-((3,7-Dimethyloct-6-en-1-yl)oxy)trimethylsilane



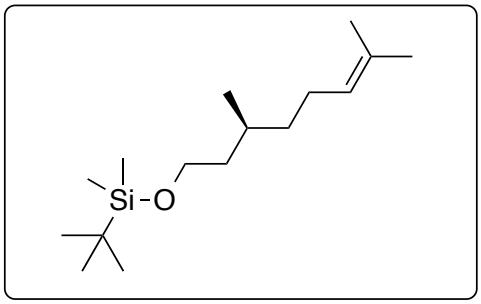
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.09 (ddq, *J* = 8.4, 5.5, 1.4 Hz, 1H), 3.64 – 3.57 (m, 2H), 1.97 (tq, *J* = 14.8, 7.6 Hz, 2H), 1.68 (s, *J* = 1.5 Hz, 3H), 1.60 (s, *J* = 1.3 Hz, 3H), 1.58 – 1.50 (m, 2H), 1.37 – 1.30 (m, 2H), 1.19 – 1.12 (m, 1H), 0.88 (d, *J* = 6.6 Hz, 3H), 0.11 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) 131.25, 125.01, 61.07, 39.96, 37.38, 29.35, 25.87, 25.62, 19.74, 17.78, -0.30.

Yield: 79% colorless oil.

R_f: 0.75 (10% EtOAc in hexanes).

(*S*)-*Tert*-butyl-((3,7-dimethyloct-6-en-1-yl)oxy)dimethylsilane



^{1}H NMR (500 MHz, $CDCl_3$) δ 5.10 (tp, *J* = 7.0, 1.4 Hz, 1H), 3.67 – 3.60 (m, 2H), 2.02 – 1.93 (m, 2H), 1.68 (s, *J* = 1.3 Hz, 3H), 1.60 (s, *J* = 1.2 Hz, 3H), 1.57 – 1.54 (m, 2H), 1.33 (m, *J* = 10.9, 5.6, 2.9, 1.3 Hz, 2H), 1.19 – 1.12 (m, 1H), 0.89 (s, 9H), 0.05 (s, 6H).

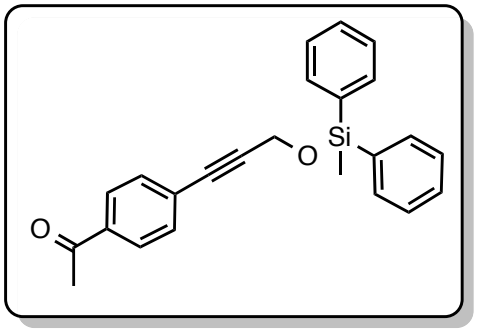
^{13}C NMR (126 MHz, $CDCl_3$) δ 131.22, 125.05, 61.62, 40.10, 37.37, 29.28, 26.13, 25.87, 25.65, 19.79, 18.50, 17.79, -5.11.

Yield: 84% colorless oil.

R_f: 0.75 (10% EtOAc in hexanes).

HRMS for $C_{16}H_{34}OSi$ EI-MS [M^+] calcd. 270.2379 found: 270.2376

1-(4-(3-((Methyldiphenylsilyl)oxy)prop-1-yn-1-yl)phenyl)ethan-1-one



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.90 – 7.86 (m, 2H), 7.69 – 7.66 (m, 4H), 7.41 – 7.39 (m, 8H), 4.63 (s, 2H), 2.59 (s, 3H), 0.77 (s, 3H).

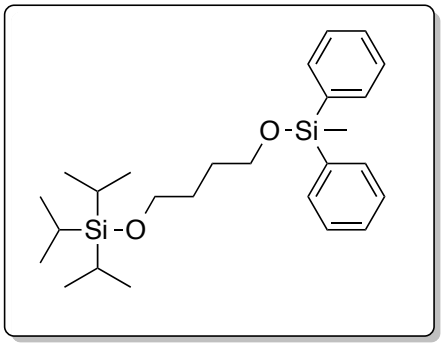
^{13}C NMR (126 MHz, $CDCl_3$) δ 176.69, 171.11, 137.19, 134.63, 134.13, 134.10, 131.94, 131.84, 130.22, 130.04, 129.70, 128.38, 128.27, 128.10, 128.07, 127.86, 51.77, 26.77, -1.08.

Yield: 69% yellow oil.

R_f: 0.65 (15 % EtOAc in hexanes).

HRMS for $C_{24}H_{22}O_2Si$ EI-MS [M^+] calcd: 370.1389; found: 370.1384

9,9-Diisopropyl-10-methyl-2,2-diphenyl-3,8-dioxa-2,9-disilaundecane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.53 (m, 4H), 7.39 – 7.32 (m, 6H), 3.69 (dd, *J* = 6.9, 4.4 Hz, 2H), 3.65 (dt, *J* = 6.3, 3.2 Hz, 2H), 1.60 (tt, *J* = 5.2, 2.9 Hz, 4H), 1.04 (s, *J* = 2.4 Hz, 21H), 0.62 (s, *J* = 2.6 Hz, 3H).

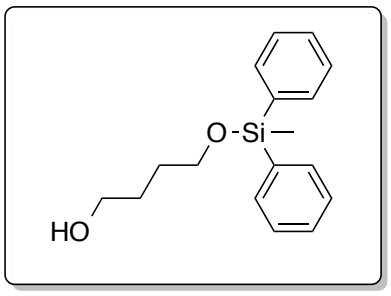
^{13}C NMR (126 MHz, $CDCl_3$) δ 134.32, 129.71, 127.80, 63.47, 63.38, 63.26, 63.18, 29.54, 29.43, 29.10, 18.03, 12.03, 12.00, -3.05.

Yield: 81% light yellow oil.

R_f: 0.45 (100% hexanes).

HRMS for $C_{26}H_{42}O_2Si_2$ EI-MS [M^+] calcd: 442.2723; found: 442.2720

4-((Methyldiphenylsilyl)oxy)butan-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 – 7.57 (m, 4H), 7.44 – 7.36 (m, 6H), 3.76 – 3.73 (m, 2H), 3.65 – 3.61 (m, 2H), 1.68 – 1.64 (m, 4H), 0.65 (s, 3H).

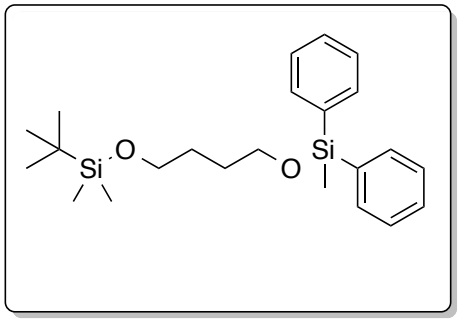
^{13}C NMR (126 MHz, $CDCl_3$) δ 135.81, 134.33, 129.89, 127.90, 63.45, 62.78, 29.75, 29.26, -3.12.

Yield: 85% light yellow oil.

R_f: 0.26 (25% EtOAc in hexanes).

HRMS for $C_{17}H_{22}O_2Si$ EI-MS [M^+] calcd: 286.1389; found: 286.1390

9,9,10,10-Tetramethyl-2,2-diphenyl-3,8-dioxa-2,9-disilaundecane



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 – 7.55 (m, 4H), 7.38 – 7.33 (m, 6H), 3.69 (t, *J* = 6.3 Hz, 2H), 3.57 (t, *J* = 6.2 Hz, 2H), 1.61 – 1.53 (m, 4H), 0.85 (s, 9H), 0.61 (s, 3H), 0.00 (s, 8H).

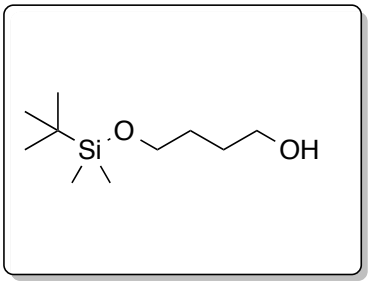
^{13}C NMR (126 MHz, $CDCl_3$) δ 136.40, 134.48, 129.88, 127.96, 63.56, 63.14, 29.41, 29.22, 26.12, 18.49, -2.88, -5.13.

Yield: 85% light yellow oil.

R_f: 0.42 (100% hexanes).

HRMS for $C_{23}H_{36}O_2Si_2$ EI-MS [M^+] calcd: 400.2254; found: 400.2254

4-((*Tert*-butyldimethylsilyl)oxy)butan-1-ol



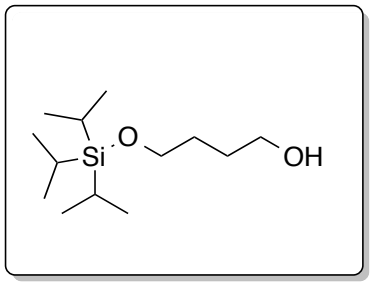
^{1}H NMR (500 MHz, $CDCl_3$) δ 3.63 (dt, *J* = 12.4, 5.7 Hz, 4H), 1.65 – 1.60 (m, 4H), 0.88 (s, 9H), 0.05 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 63.33, 62.77, 30.23, 29.86, 29.69, 25.89, 18.29, -5.40.

Yield: 79% colorless oil.

R_f: 0.25 (25% EtOAc in hexanes).

4-((Triisopropylsilyl)oxy)butan-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 3.73 (q, *J* = 5.1, 4.6 Hz, 2H), 3.64 (q, *J* = 5.2 Hz, 2H), 2.54 (s, 1H), 1.65 (dq, *J* = 9.9, 5.0, 4.4 Hz, 4H), 1.04 (s, *J* = 5.5 Hz, 21H).

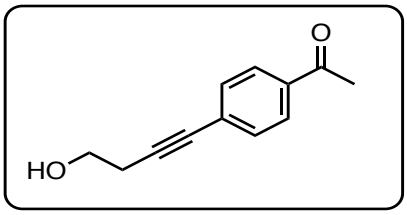
^{13}C NMR (126 MHz, $CDCl_3$) δ 63.59, 62.85, 30.35, 30.06, 17.96, 11.94.

Yield: 89% colorless oil.

R_f: 0.28 (25% EtOAc in hexanes).

8 Analytical Data for Free Alcohols Deprotected under 18-Crown-6 and SFx Conditions

1-(4-(4-Hydroxybut-1-yn-1-yl)phenyl)ethan-1-one



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 – 7.86 (d, 2H), 7.49 – 7.45 (d, 2H), 3.82 (q, *J* = 6.2 Hz, 2H), 2.71 (t, *J* = 6.2 Hz, 2H), 2.57 (s, 3H), 1.75 (t, *J* = 6.3 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 131.80, 128.31, 128.19, 104.99, 90.16, 81.82, 61.03, 26.61, 23.93.

Yield (SFx): 79% tan solid.

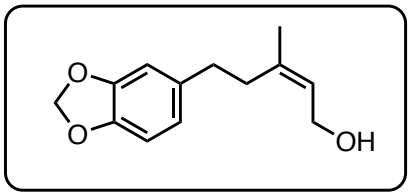
Yield (18-Crown-6): 93% tan solid.

Yield (Syn): 81% tan solid.

R_f: 0.35 (80% Et_2O in hexanes).

HRMS for $C_{12}H_{12}O_2$ EI-MS [M^+] calcd: 188.0837; found: 189.0913 [M+H]$^+$

(*Z*)-5-(Benzo[1,3]dioxol-5-yl)-3-methylpent-2-en-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 6.72 (d, *J* = 7.9 Hz, 1H), 6.67 (d, *J* = 1.7 Hz, 1H), 6.61 (dd, *J* = 7.8, 1.7 Hz, 1H), 5.91 (s, 2H), 5.41 (m, *J* = 6.9, 5.5, 1.3 Hz, 1H), 4.14 (d, *J* = 6.9 Hz, 2H), 2.68 – 2.63 (m, 2H), 2.28 (dd, *J* = 8.9, 7.2 Hz, 2H), 1.71 (s, 3H), 1.25 (s, 1H).

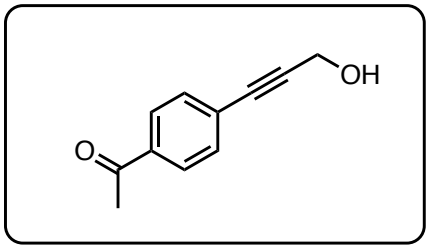
^{13}C NMR (126 MHz, $CDCl_3$) δ 147.65, 145.73, 139.17, 135.95, 124.08, 121.31, 108.94, 108.23, 100.89, 59.48, 41.78, 34.21, 16.53.

Yield (SFx): 90% yellow oil.

Yield (18-Crown-6): 92% yellow oil.

R_f: 0.4 (25% EtOAc in hexanes)

1-(4-(3-Hydroxyprop-1-yn-1-yl)phenyl)ethan-1-one



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.92 – 7.89 (m, 2H), 7.53 – 7.49 (m, 2H), 4.53 (d, *J* = 6.2 Hz, 2H), 2.60 (s, 3H), 1.75 (t, *J* = 6.2 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 197.46, 136.63, 131.94, 128.38, 127.55, 90.62, 85.06, 51.78, 26.78.

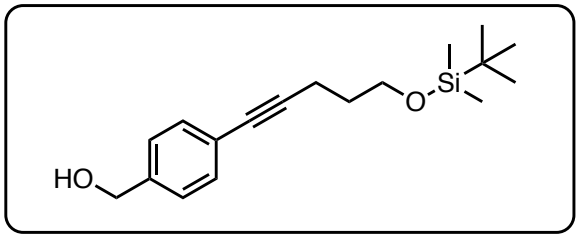
Yield (SFx): 79% red oil.

Yield (18-Crown-6): 88% red oil.

Yield (Syn): 81% red oil.

R_f: 0.37 (50% EtOAc in hexanes)

(4-(5-(*Tert*-butyldimethylsilyl)oxy)pent-1-yn-1-yl)phenyl)methanol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.37 (m, 2H), 7.29 – 7.26 (m, 2H), 4.68 (d, *J* = 5.9 Hz, 2H), 3.76 (t, *J* = 6.0 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 1.84 – 1.78 (m, 2H), 1.67 (t, *J* = 6.0 Hz, 1H), 0.91 (s, 9H), 0.08 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 140.30, 131.85, 126.90, 123.49, 90.16, 80.63, 65.20, 61.79, 31.89, 26.12, 18.53, 15.99, -5.15.

Yield (SFx): 71% brown oil.

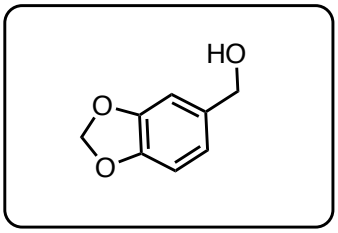
Yield (18-Crown-6): 80% brown oil.

Yield (Syn): 81% brown oil.

R_f: 0.35 (15% EtOAc in hexanes).

HRMS for $C_{18}H_{30}O_2$ EI-MS $[M^+]$ calcd: 278.2245; found: 247.1154 $[M\text{-}C_4H_9]^+$

Benzo[*d*][1,3]dioxol-5-ylmethanol



^{1}H NMR (500 MHz, $CDCl_3$) δ 6.86 (s, *J* = 1.5 Hz, 1H), 6.79 (m, *J* = 7.9, 6.1 Hz, 2H), 5.95 (s, *J* = 1.9 Hz, 2H), 4.57 (s, *J* = 3.4 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 147.90, 147.17, 135.00, 120.59, 120.46, 108.30, 108.21, 108.14, 107.98, 107.84, 101.10, 100.97, 65.29, 65.14.

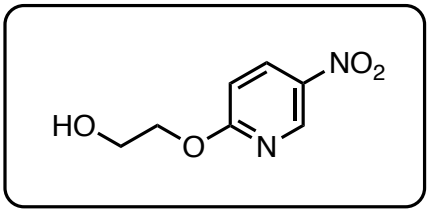
Yield (SFx): 92% white solid.

Yield (18-Crown-6): 83% white solid.

Yield (Syn): 85% white solid.

R_f: 0.33 (40% Et_2O in hexanes)

2-((5-Nitropyridin-2-yl)oxy)ethan-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 9.06 (d, *J* = 2.8 Hz, 1H), 8.38 (dd, *J* = 9.1, 2.9 Hz, 1H), 6.89 (d, *J* = 8.6 Hz, 1H), 4.59 – 4.57 (m, 2H), 4.02 – 3.99 (m, 2H).

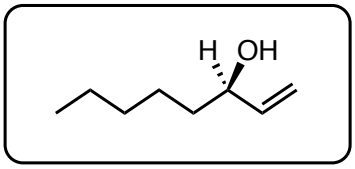
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.06, 144.73, 139.81, 134.30, 111.64, 69.46, 69.45, 61.52.

Yield (SFx): 86% white solid.

Yield (18-Crown-6): 79% white solid.

R_f: 0.35 (80% EtOAc in hexanes).

(*R*)-(-)-1-Octen-3-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 5.85 (ddd, *J* = 16.9, 10.4, 6.2 Hz, 1H), 5.20 (dt, *J* = 17.2, 1.4 Hz, 1H), 5.08 (dt, *J* = 10.4, 1.4 Hz, 1H), 4.07 (tdd, *J* = 7.4, 5.5, 1.3 Hz, 1H), 1.70 – 1.54 (m, 1H), 1.54 – 1.44 (m, 2H), 1.38 (dddd, *J* = 13.6, 8.5, 6.6, 3.0 Hz, 1H), 1.34 – 1.23 (m, 5H), 0.89 – 0.84 (m, 3H).

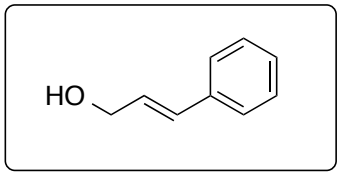
^{13}C NMR (126 MHz, $CDCl_3$) δ 141.32, 114.46, 73.24, 36.97, 31.74, 24.99, 22.58, 14.00.

Yield (SFx): 68% colorless oil.

Yield (18-Crown-6): 75% colorless oil.

Rf: 0.35 (25% EtOAc in hexanes).

(2*E*)-3-Phenyl-2-propen-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.35 (m, 2H), 7.31 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.25 – 7.21 (m, 1H), 6.60 (dt, *J* = 15.9, 1.6 Hz, 1H), 6.35 (dt, *J* = 15.9, 5.8 Hz, 1H), 4.31 (dd, *J* = 5.7, 1.6 Hz, 2H), 1.77 – 1.66 (m, 1H).

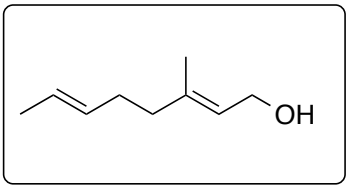
^{13}C NMR (126 MHz, $CDCl_3$) δ 136.71, 131.07, 128.61, 128.55, 127.69, 126.49, 126.48, 63.63.

Yield (SFx): 85% white solid.

Yield (18-Crown-6): 85% white solid.

R_f: 0.40 (25% EtOAc in hexanes).

(*Z*)-3,7-Dimethylocta-2,6-dien-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 5.42 (td, *J* = 7.2, 1.5 Hz, 1H), 5.08 (dddq, *J* = 7.2, 5.8, 2.9, 1.5 Hz, 1H), 4.07 (d, *J* = 7.2 Hz, 2H), 2.11 – 2.03 (m, 4H), 1.73 (d, *J* = 1.3 Hz, 3H), 1.67 (d, *J* = 1.3 Hz, 3H), 1.58 (s, 3H), 1.17 (d, *J* = 6.1 Hz, 1H).

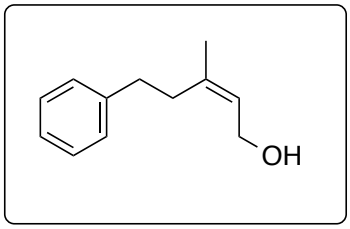
^{13}C NMR (126 MHz, $CDCl_3$) δ 132.31, 124.47, 124.45, 123.81, 123.80, 58.86, 58.84, 31.95, 31.94, 26.53, 26.52, 25.61, 23.37, 17.60.

Yield (SFx): 85% brown oil.

Yield (18-Crown-6): 91% brown oil.

R_f: 0.36 (25% EtOAc in hexanes).

(2*E*)-3-Methyl-5-phenyl-2-penten-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 2H), 7.22 – 7.18 (m, 3H), 5.43 (tp, *J* = 6.9, 1.3 Hz, 1H), 4.16 (d, *J* = 6.9 Hz, 2H), 2.79 – 2.74 (m, 2H), 2.35 (dd, *J* = 9.5, 6.7 Hz, 2H), 1.75 (s, 3H), 1.28 – 1.16 (m, 1H).

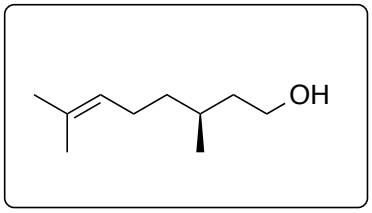
^{13}C NMR (126 MHz, $CDCl_3$) δ 141.98, 128.37, 128.32, 125.85, 123.90, 59.28, 41.38, 34.34, 16.40.

Yield (SFx): 87% yellow oil.

Yield (18-Crown-6): 92% yellow oil.

R_f: 0.26 (25% EtOAc in hexanes).

(*S*)-3,7-Dimethyloct-6-en-1-ol



^{1}H NMR (500 MHz, $CDCl_3$) δ 5.14 – 5.08 (m, 1H), 3.70 (ddddt, *J* = 17.0, 13.4, 10.1, 7.1, 2.7 Hz, 2H), 2.00 (tq, *J* = 14.8, 7.5 Hz, 2H), 1.70 (d, *J* = 3.0 Hz, 3H), 1.62 (d, *J* = 2.8 Hz, 3H), 1.37 (dtdd, *J* = 21.2, 8.4, 5.1, 2.6 Hz, 3H), 1.21 (tdd, *J* = 13.6, 6.7, 3.4 Hz, 2H), 0.92 (dd, *J* = 6.5, 2.7 Hz, 3H).

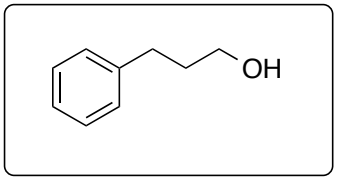
^{13}C NMR (126 MHz, $CDCl_3$) δ 131.25, 124.69, 61.20, 39.91, 37.21, 29.17, 25.70, 25.68, 25.45, 19.52, 17.63.

Yield (SFx): 88% colorless oil.

Yield (18-Crown-6): 91% colorless oil.

R_f: 0.35 (50% Et_2O in hexanes).

3-Phenyl-1-propanol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 7.21 (d, *J* = 7.7 Hz, 3H), 3.68 (td, *J* = 6.5, 1.2 Hz, 2H), 2.74 – 2.69 (m, 2H), 1.90 (dtd, *J* = 9.0, 7.6, 7.0, 5.8 Hz, 2H), 1.55 (d, *J* = 7.1 Hz, 1H).

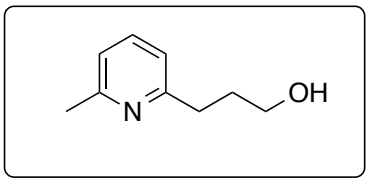
^{13}C NMR (126 MHz, $CDCl_3$) δ 141.85, 128.44, 128.41, 128.40, 125.87, 62.23, 62.04, 34.22, 34.16, 32.09.

Yield (SFx): 89% colorless oil.

Yield (18-Crown-6): 94% colorless oil.

R_f: 0.31 (25% EtOAc in hexanes).

3-(6-Methyl-2-pyridinyl)-1-propanol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 – 7.48 (m, 1H), 6.98 (d, *J* = 7.7 Hz, 2H), 3.72 (td, *J* = 5.6, 2.8 Hz, 2H), 2.94 (td, *J* = 6.9, 2.9 Hz, 2H), 2.51 (d, *J* = 2.8 Hz, 3H), 1.97 (dtq, *J* = 8.7, 5.9, 3.3, 2.5 Hz, 2H).

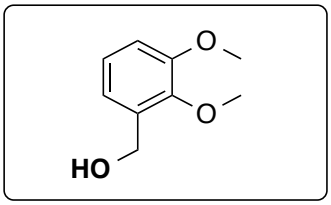
^{13}C NMR (126 MHz, $CDCl_3$) δ 160.69, 157.30, 137.06, 120.66, 120.02, 62.35, 61.91, 35.63, 31.80, 31.55, 24.07.

Yield (SFx): 91% reddish brown oil.

Yield (18-Crown-6): 90% reddish brown oil.

R_f: 0.25 (65% EtOAc in hexanes).

(2,3-Dimethoxyphenyl)methanol



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.03 (td, *J* = 7.9, 1.5 Hz, 1H), 6.92 – 6.86 (m, 2H), 4.68 (dd, *J* = 6.4, 1.5 Hz, 2H), 3.87 (d, *J* = 1.4 Hz, 3H), 3.86 (d, *J* = 1.5 Hz, 3H), 2.18 (tdd, *J* = 6.3, 4.1, 2.3 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 152.50, 134.58, 124.18, 120.64, 112.23, 61.56, 60.87, 55.81.

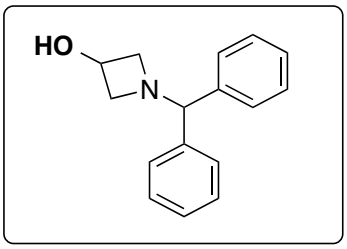
Yield (SFx): 83% white solid.

Yield (18-Crown-6): 93% white solid.

Yield (Syn): 95% white solid.

R_f: 0.32 (25% EtOAc in hexanes).

1-(Diphenylmethyl)-3-azetidinol



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.39 – 7.36 (m, 4H), 7.26 (t, *J* = 7.7 Hz, 4H), 7.19 – 7.16 (m, 2H), 4.44 (p, *J* = 5.8 Hz, 1H), 4.33 (s, 1H), 3.54 – 3.51 (m, 2H), 2.90 – 2.87 (m, 2H).

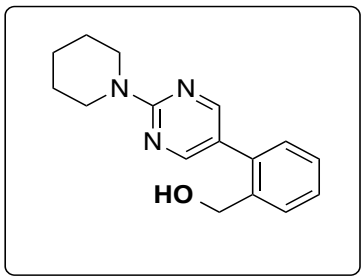
^{13}C NMR (151 MHz, $CDCl_3$) δ 141.87, 128.44, 127.41, 127.16, 78.45, 63.36, 63.34, 62.08, 62.01.

Yield (SFx): 80% white solid.

Yield (18-Crown-6): 78% white solid.

R_f: 0.29 (25% EtOAc in hexanes).

(2-(2-(Piperidin-1-yl)pyrimidin-5-yl)phenyl)methanol



^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (s, 2H), 7.55 – 7.52 (m, 1H), 7.39 – 7.32 (m, 2H), 7.21 – 7.19 (m, 1H), 4.61 (d, *J* = 4.3 Hz, 2H), 3.82 – 3.79 (m, 4H), 1.99 (d, *J* = 5.0 Hz, 1H), 1.68 (dddd, *J* = 8.3, 6.9, 3.9, 2.2 Hz, 2H), 1.64 – 1.59 (m, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 160.83, 148.91, 137.72, 137.10, 129.91, 129.89, 128.72, 127.92, 127.82, 63.34, 44.94, 25.83, 24.93.

Yield (SFx): 89% colorless oil.

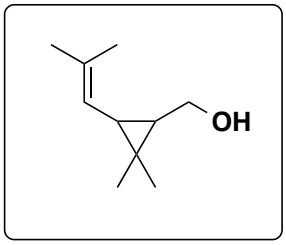
Yield (18-Crown-6): 86% colorless oil.

Yield (Syn): 93% colorless oil.

R_f: 0.27 (60% EtOAc in hexanes).

HRMS for $C_{16}H_{19}ON_3$ EI-MS $[M^+]$ calcd: 269.1528; found: 270.1608 $[M+H]^+$

[2,2-Dimethyl-3-(2-methyl-1-propen-1-yl)cyclopropyl]methanol



^{1}H NMR (500 MHz, $CDCl_3$) δ major isomer 4.87 (1H, d, *J* 8.1), 3.77 (1H, dd, *J* 6.6, 11.4), 3.55 (1H, dd, *J* 8.5, 11.4), 1.70 (3H, s,), 1.67 (3H, s,), 1.15 (3H, s), 1.11 (1H, dd, *J* 5.3, 8.1), 1.06 (3H, s), 0.83 (1H, ddd, *J* 8.5, 6.6, 5.3); minor isomer 4.96 (1H, d, *J* 8.2), 3.67 (1H, dd, *J* 7.6, 11.6), 3.61 (1H, dd, *J* 8.0, 11.6), 1.73 (3H, s), 1.70 (3H, s), 1.38 (1H, dd, *J* 8.2), 1.12 (3H, s), 1.07–1.04 (1H, m) 1.04 (3H, s).

^{13}C NMR (126 MHz, $CDCl_3$) δ major isomer 133.0, 123.5, 63.5, 35.1, 28.6, 25.6, 22.7, 21.3, 18.3, 15.5; minor isomer 135.0, 119.1, 60.4, 31.0, 28.8, 26.2, 25.8, 22.3, 20.8, 18.4.

Yield (SFx): 94% colorless oil.

Yield (18-Crown-6): 94% colorless oil.

R_f: 0.32 (25% EtOAc in hexanes).

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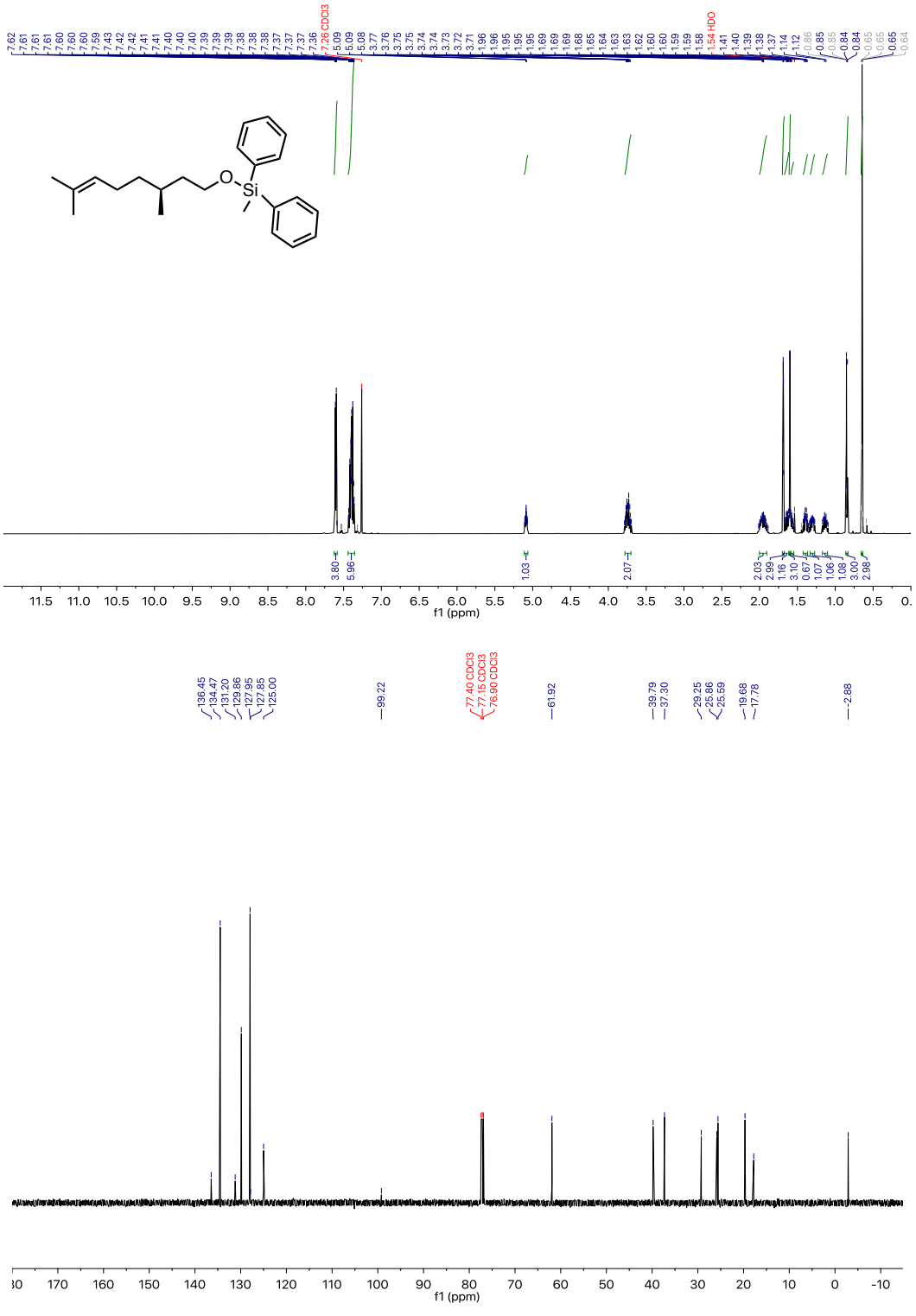
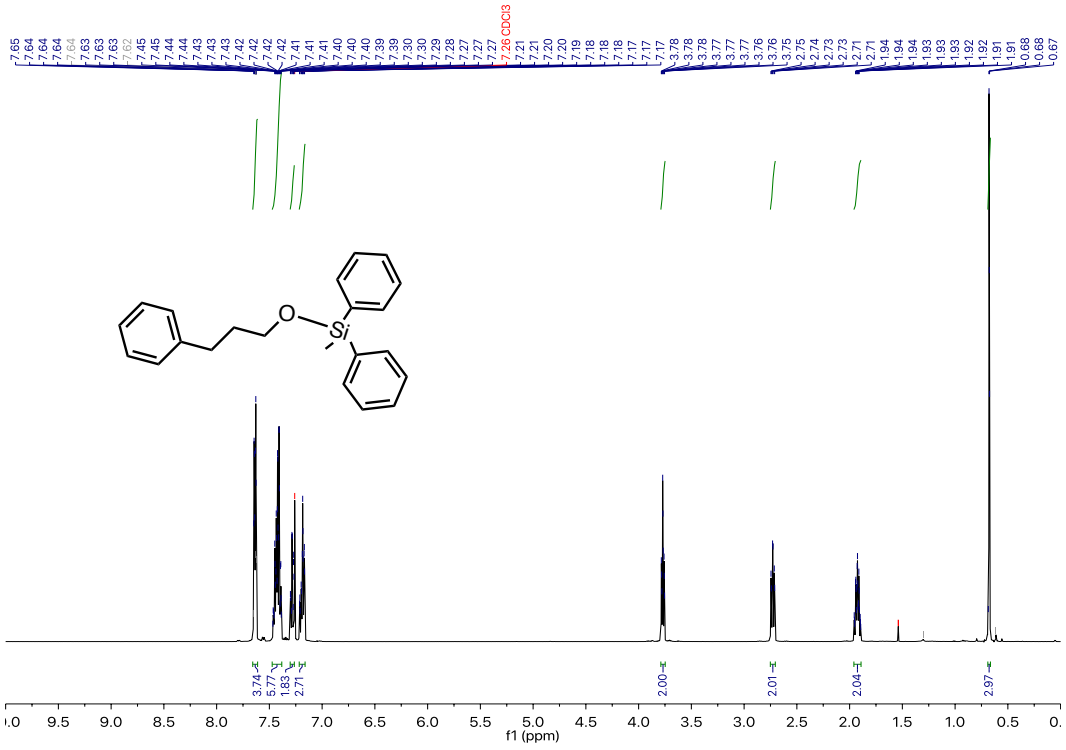
9 1H NMR 13C NMR Spectra for Products

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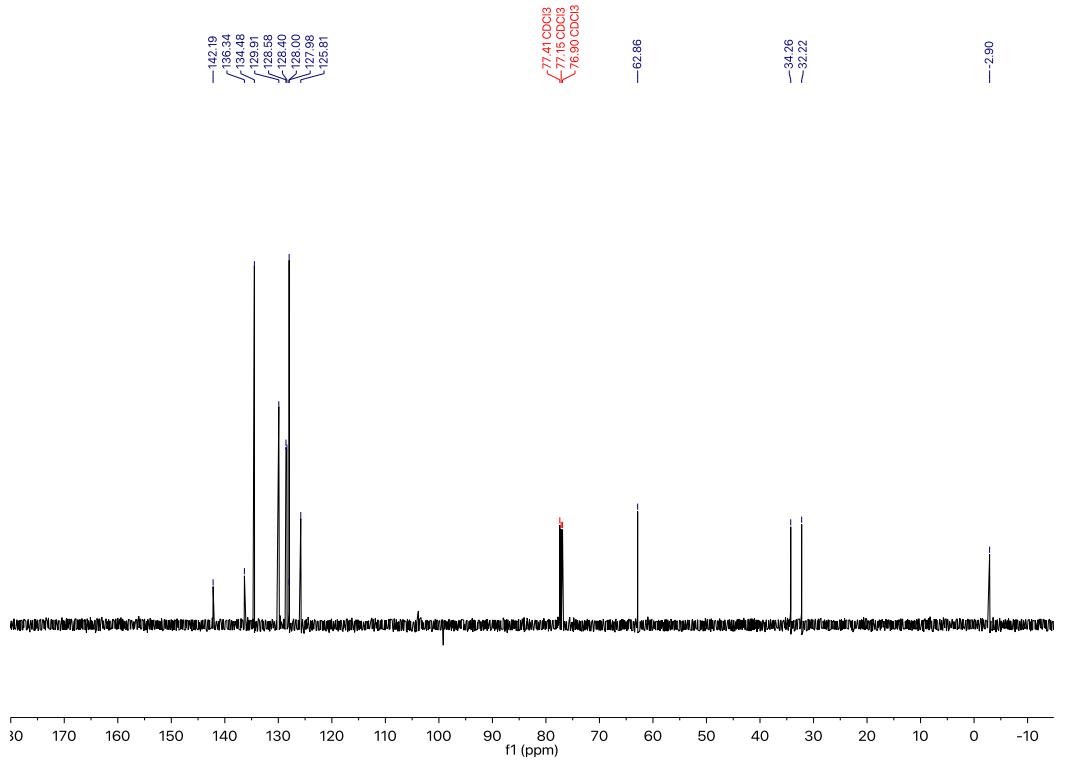
2.04
2.97
f1 (ppm)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127.98
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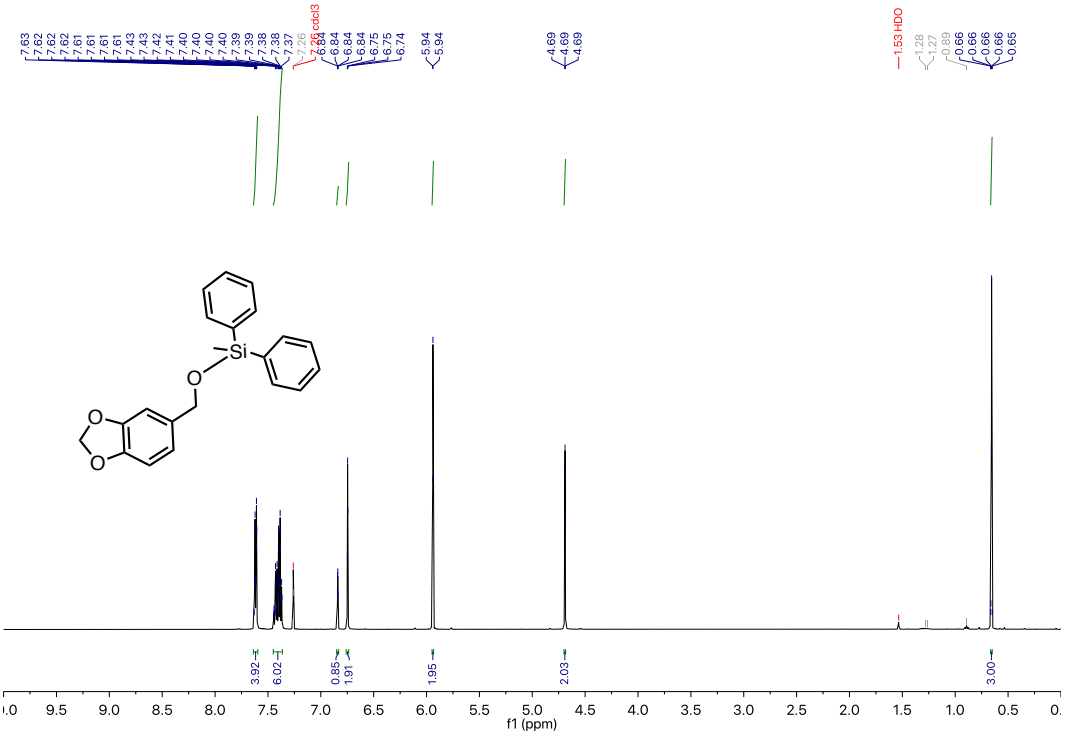
f1 (ppm)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4.69
4.69
4.69
1.53 HDO
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1.27
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0.66
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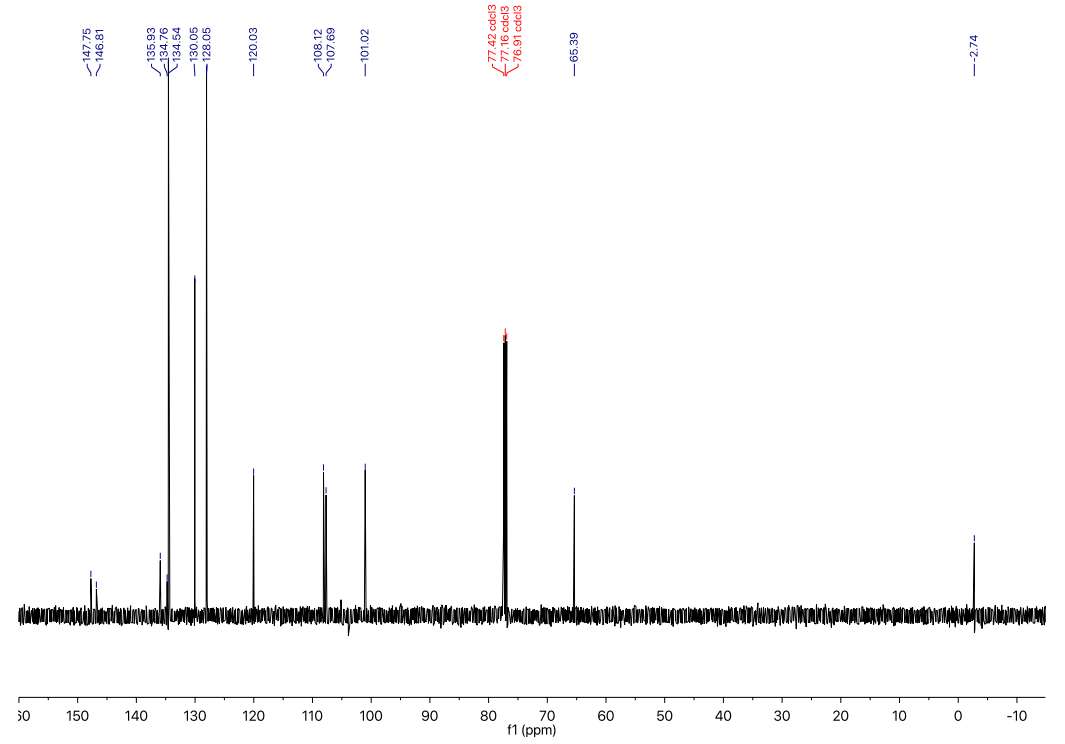
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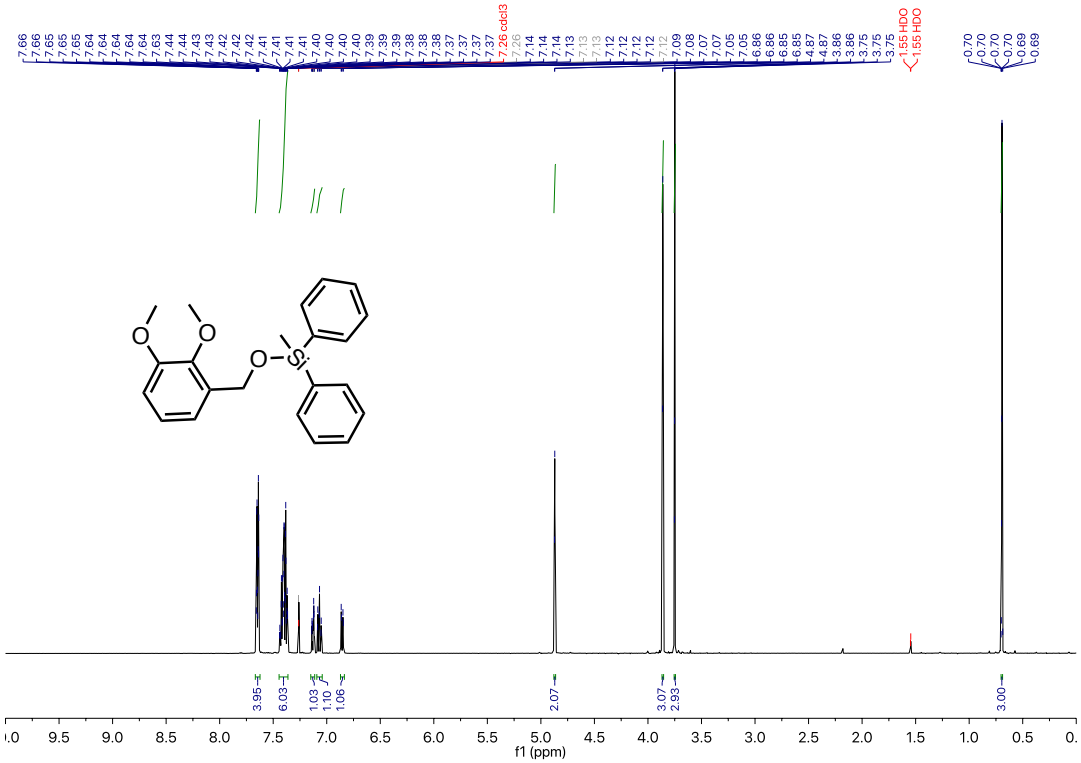
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Si
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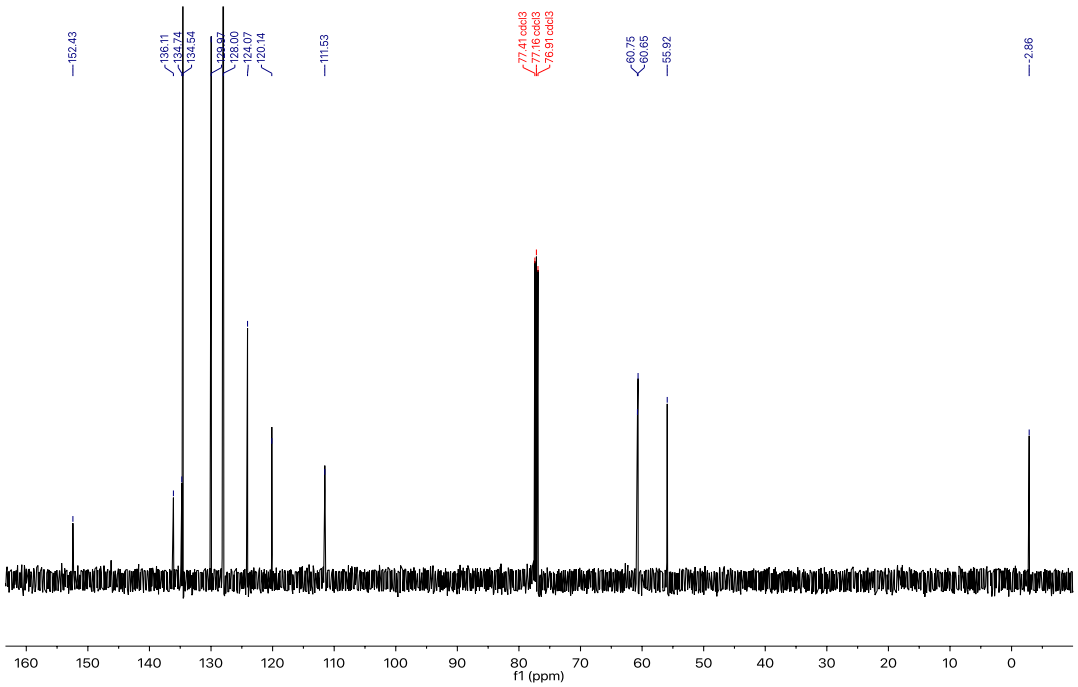
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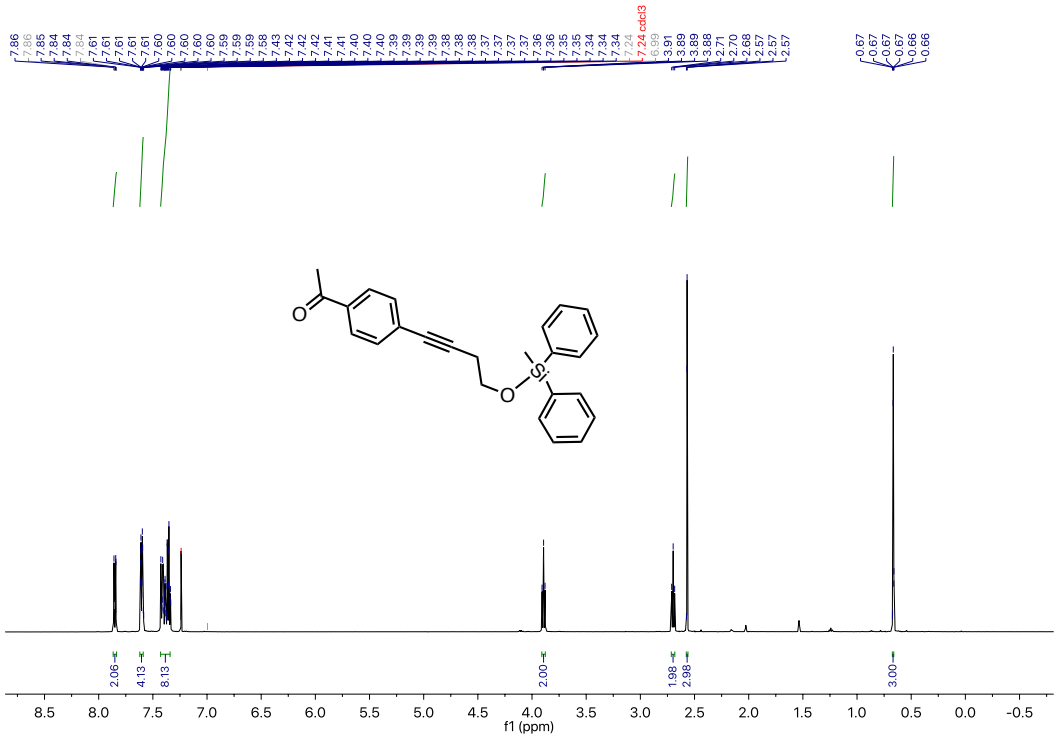
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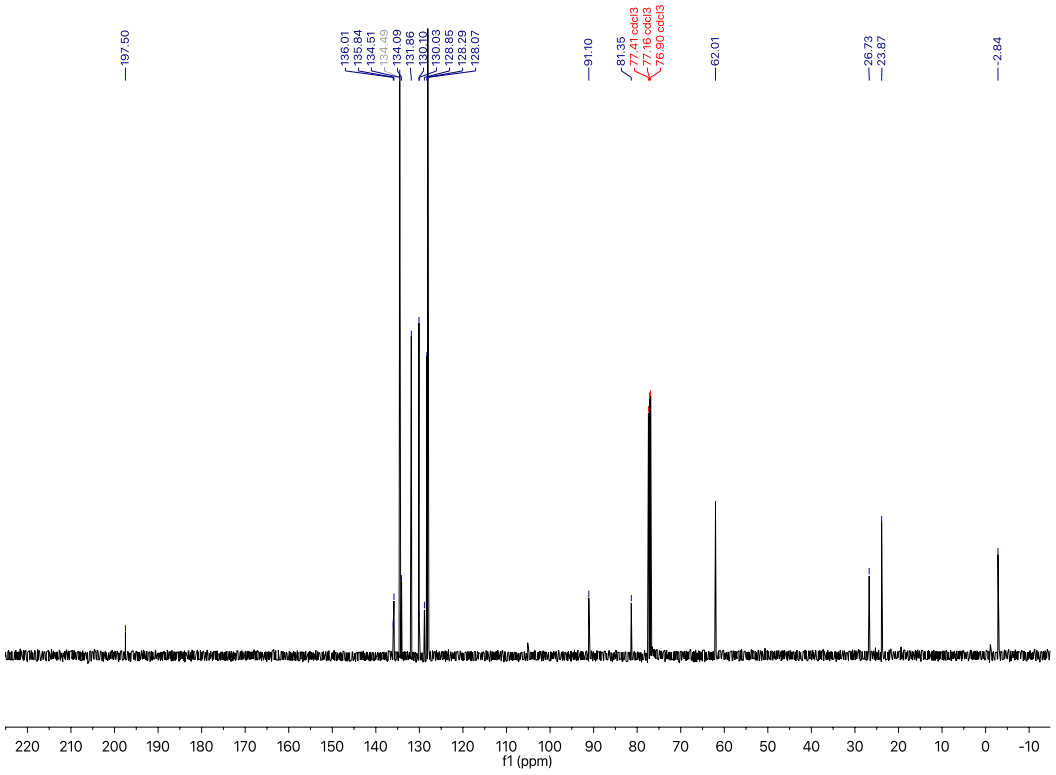
f1 (ppm)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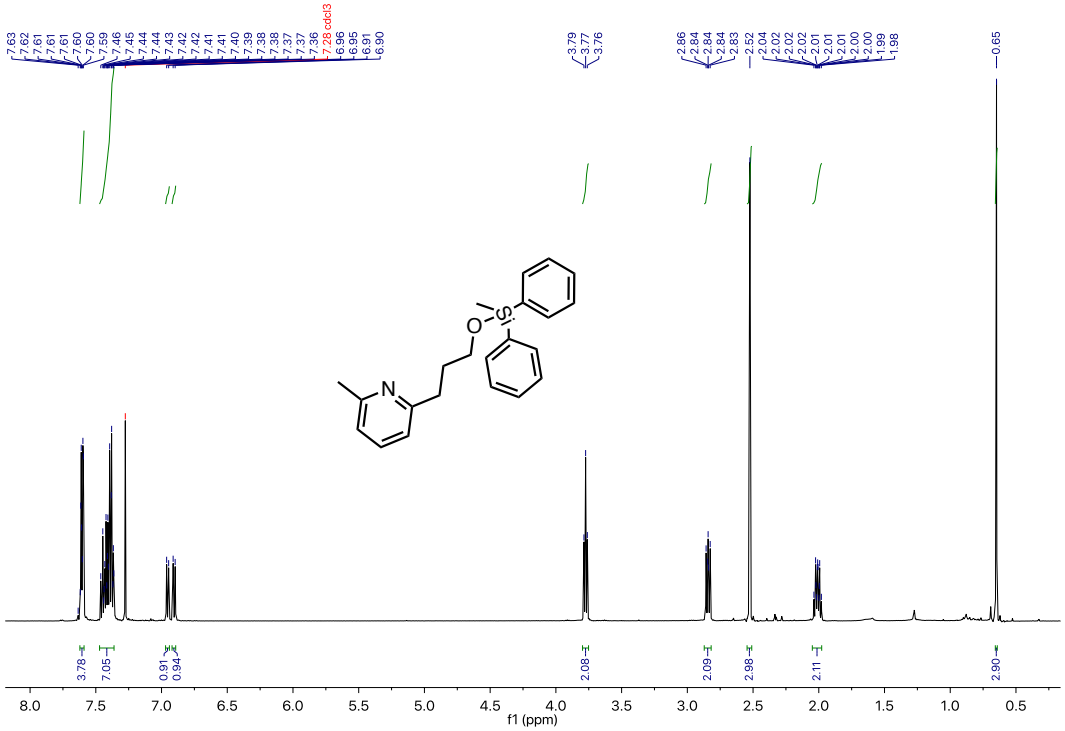
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7.39
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7.38
7.37
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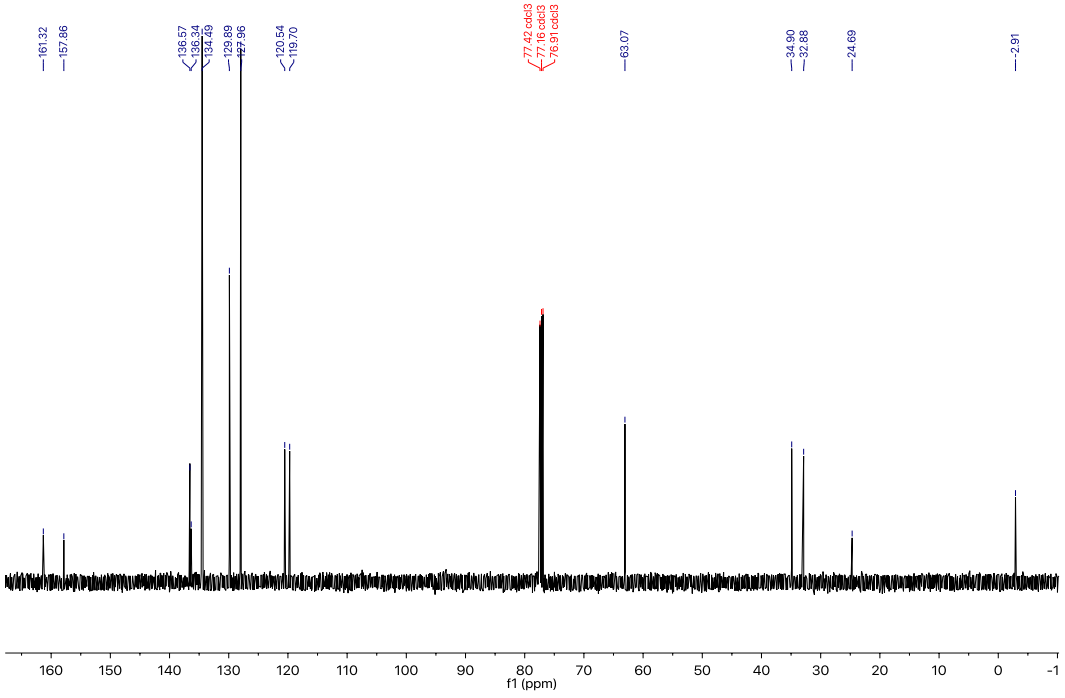
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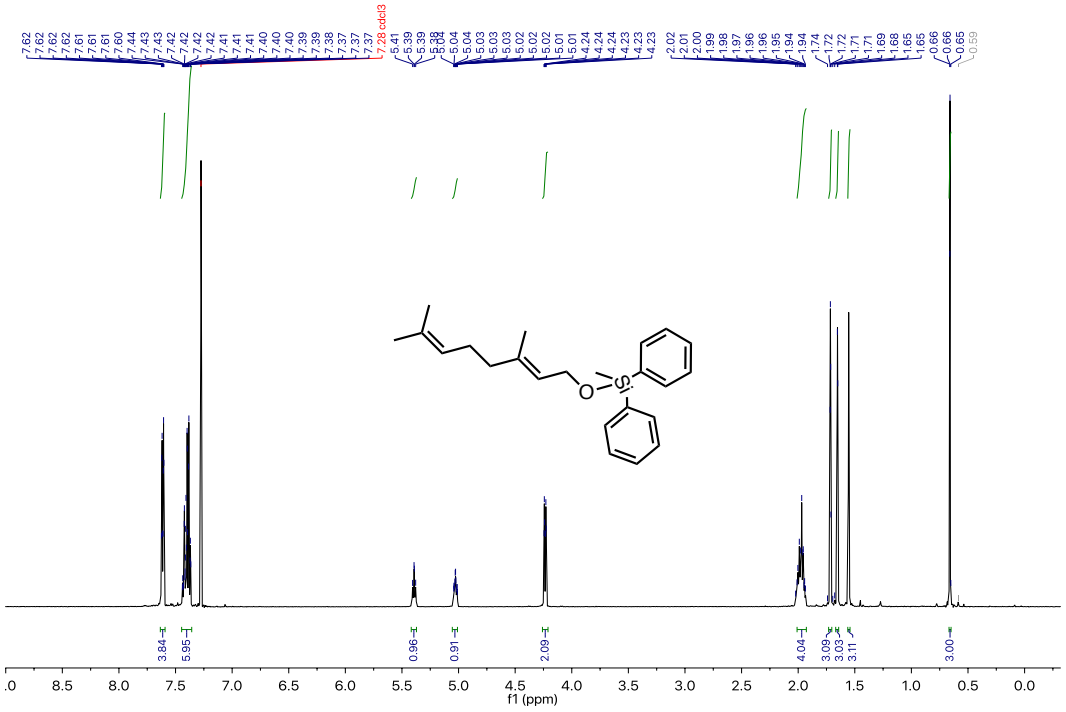
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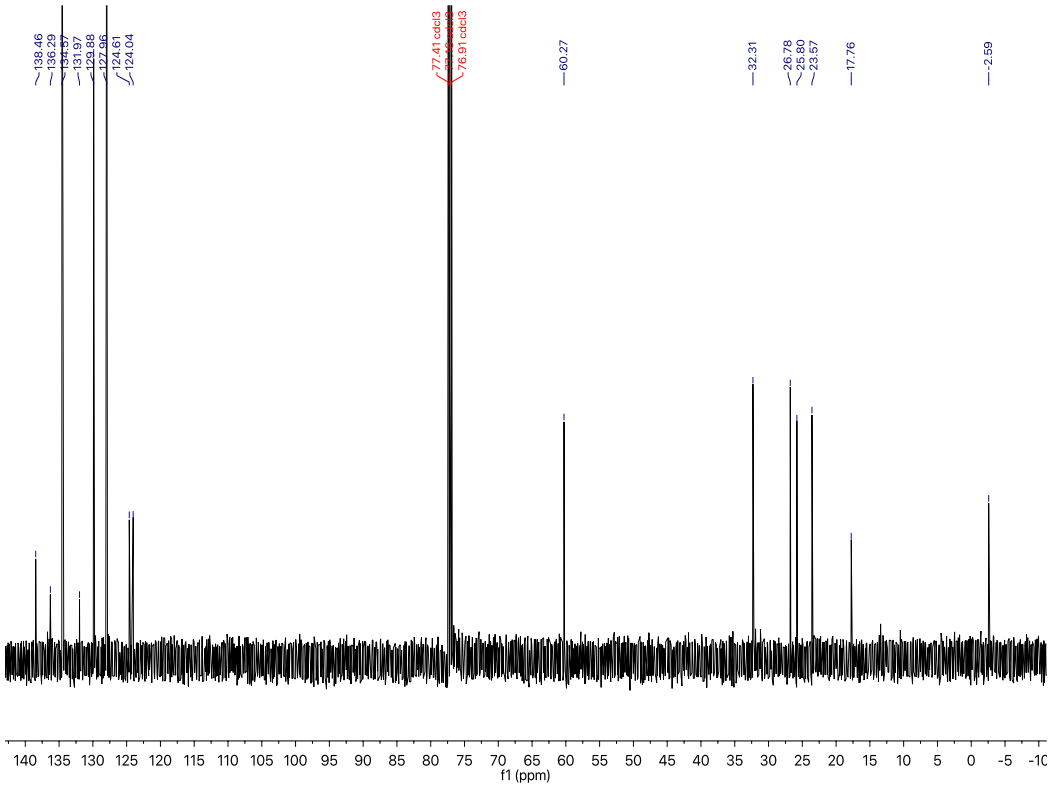
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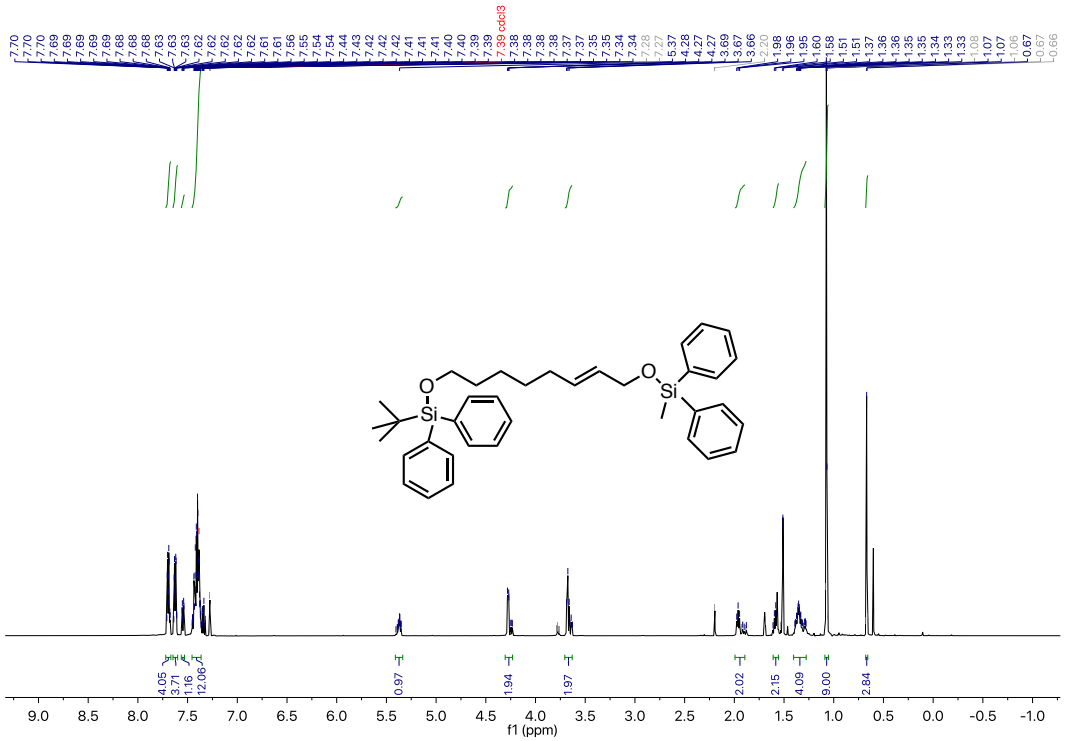
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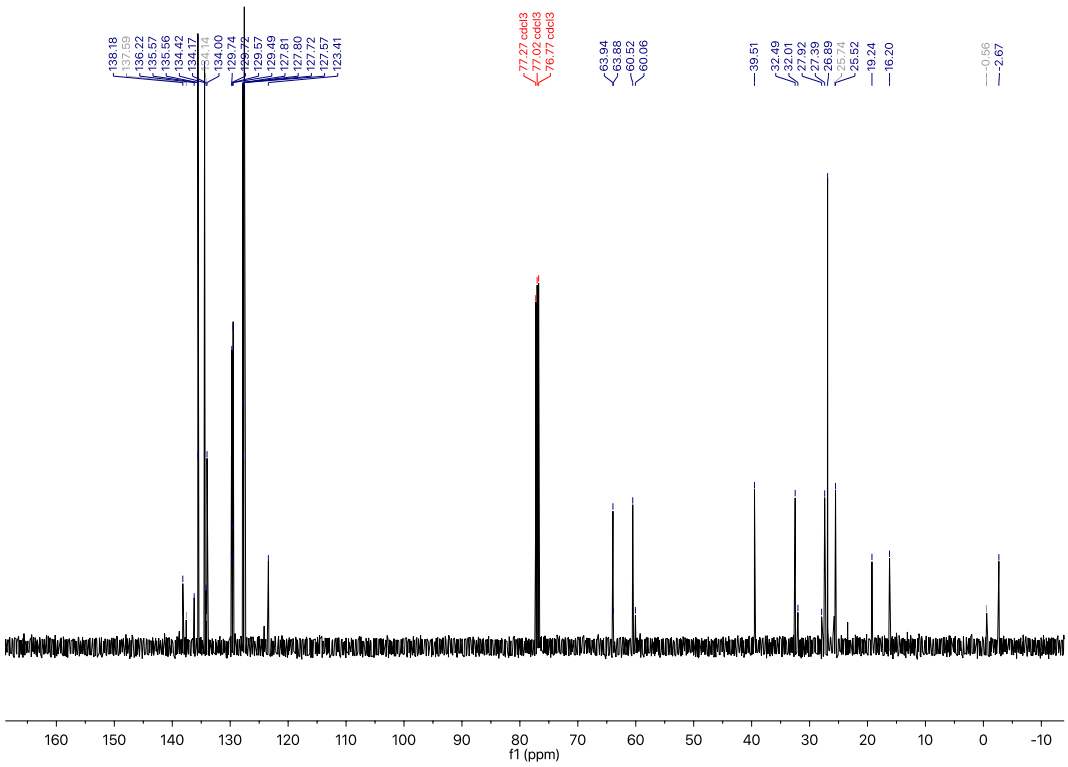
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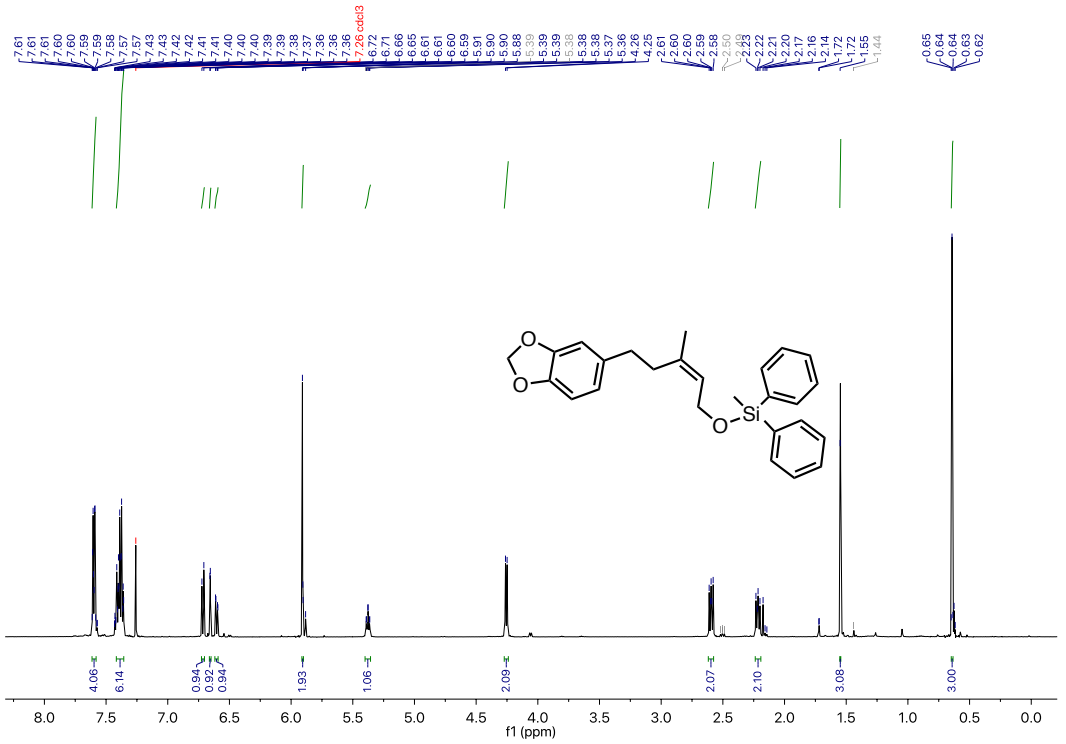
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O
O
O
Si
4.06 6.14 0.94 0.92 0.94 1.93 1.06 2.09 2.07 2.10 3.08 3.00
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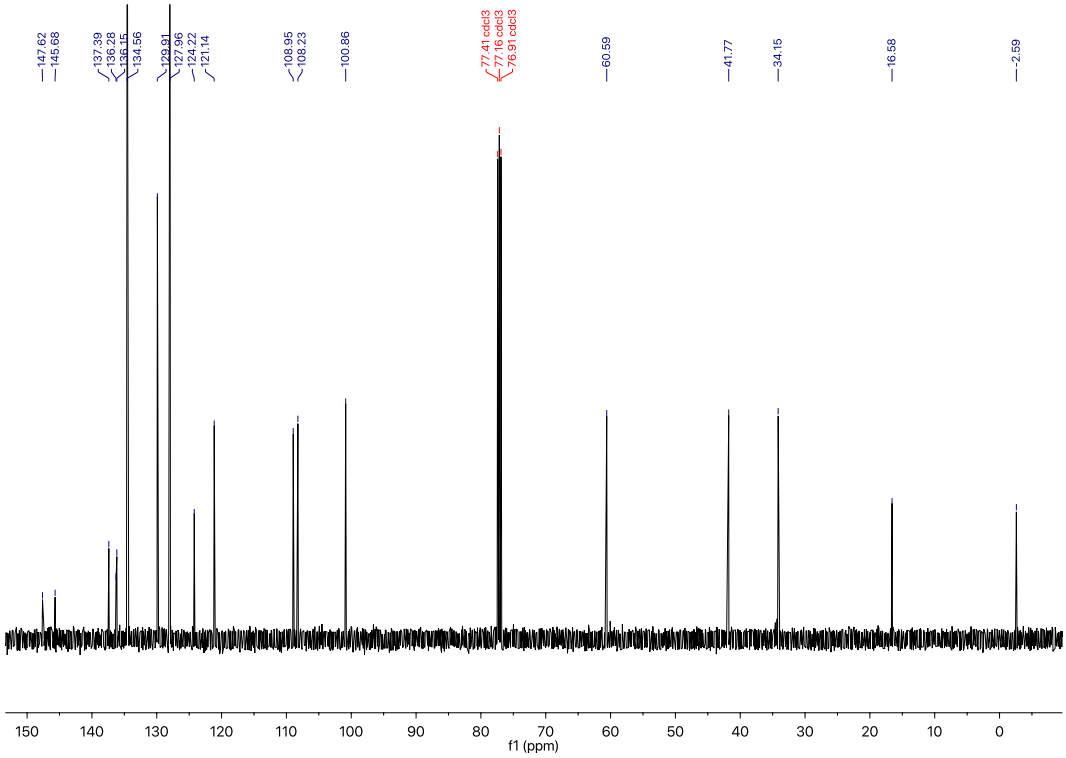
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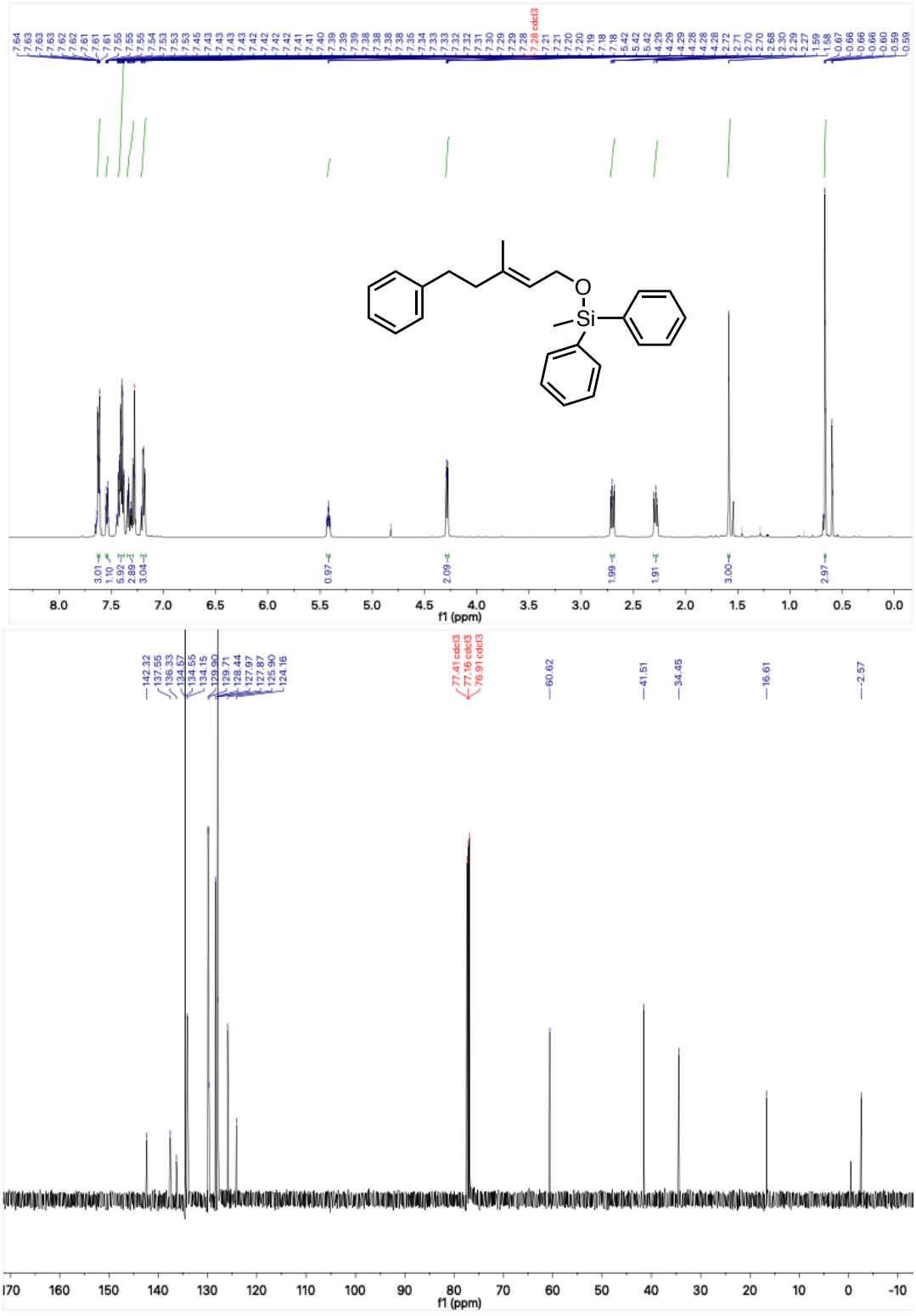
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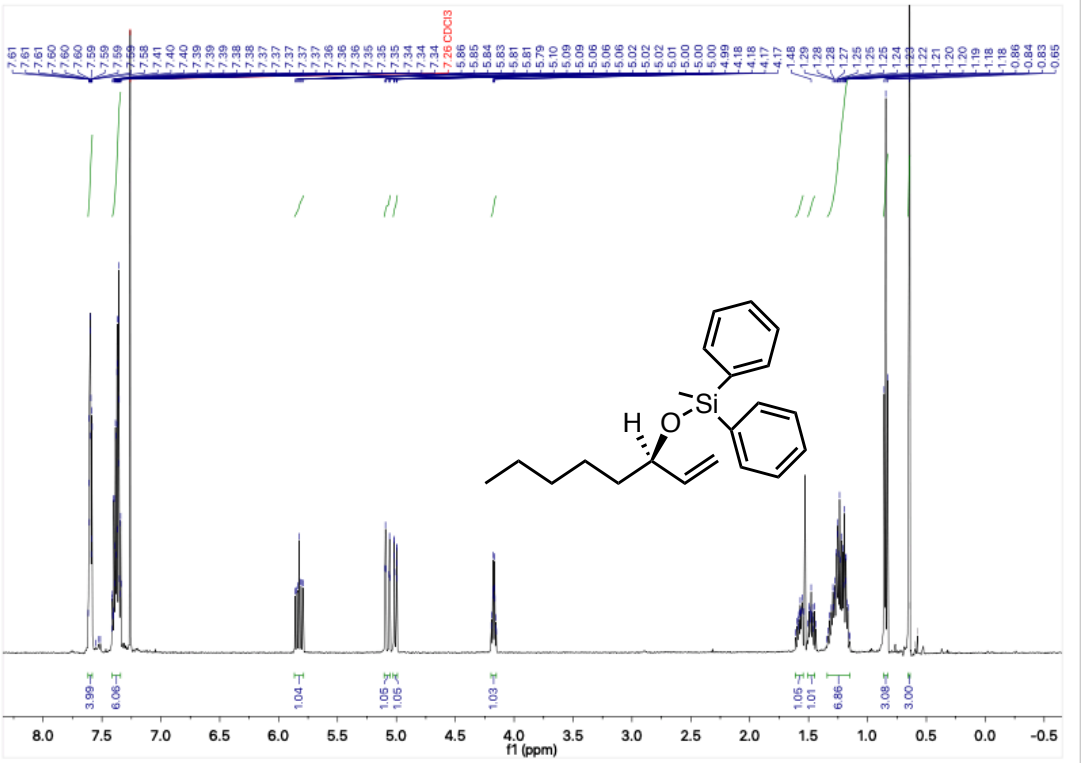
f1 (ppm)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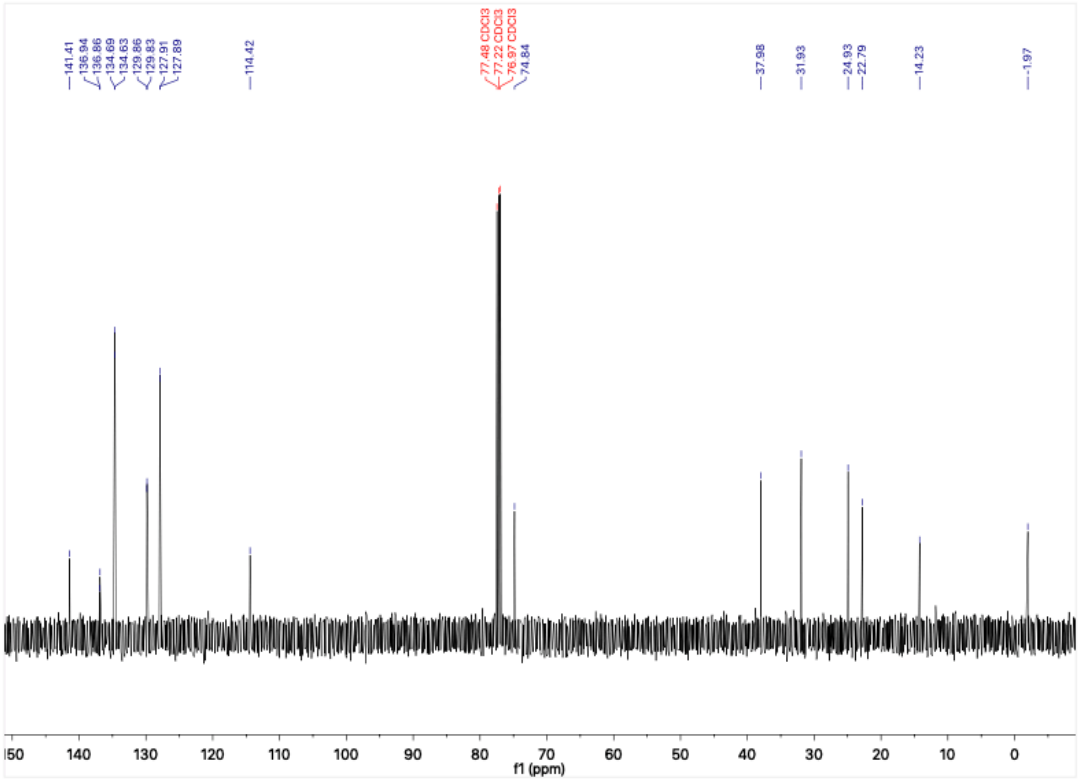
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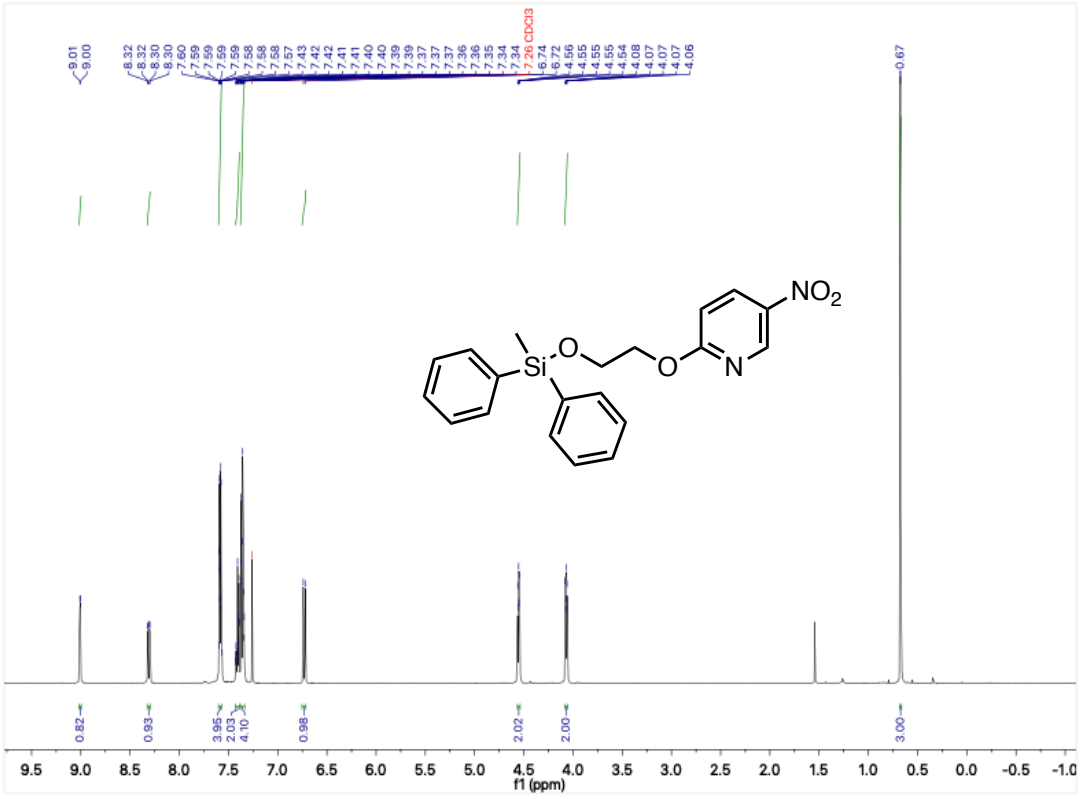
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8.32
8.30
8.30
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7.59
7.59
7.59
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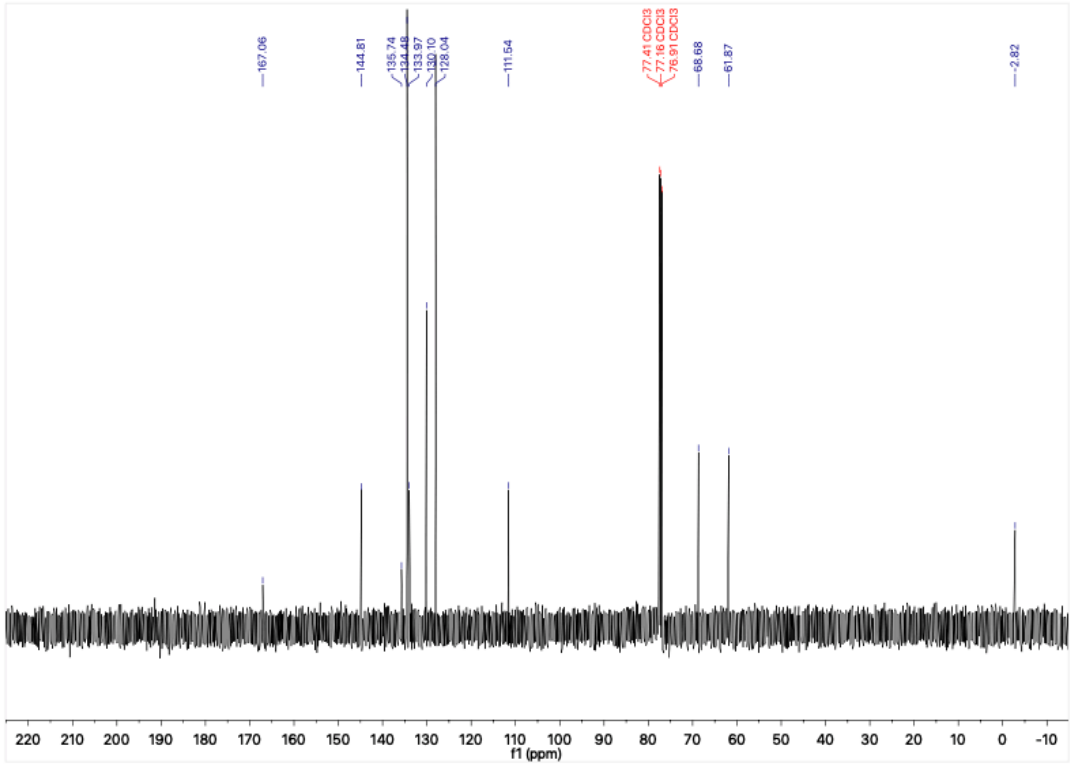
7.58
7.57
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.39
7.37
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.26 CDCl3
6.74
6.72
4.66
4.65
4.65
4.65
4.64
4.08
4.07
4.07
4.07
4.06
0.67
NO2
Si
O
O
N
0.82
0.93
3.95
2.03
4.10
0.98
2.02
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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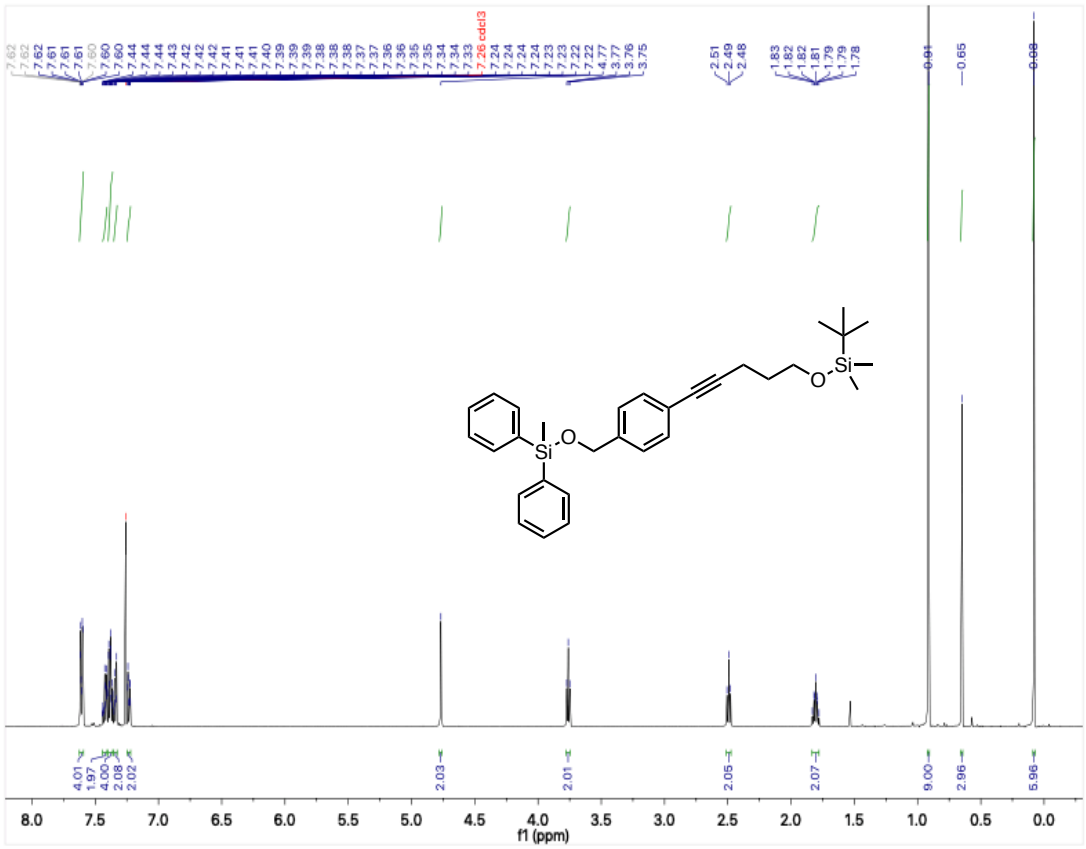
135.74
134.48
133.97
130.10
128.04
111.54
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
68.68
61.87
-2.82
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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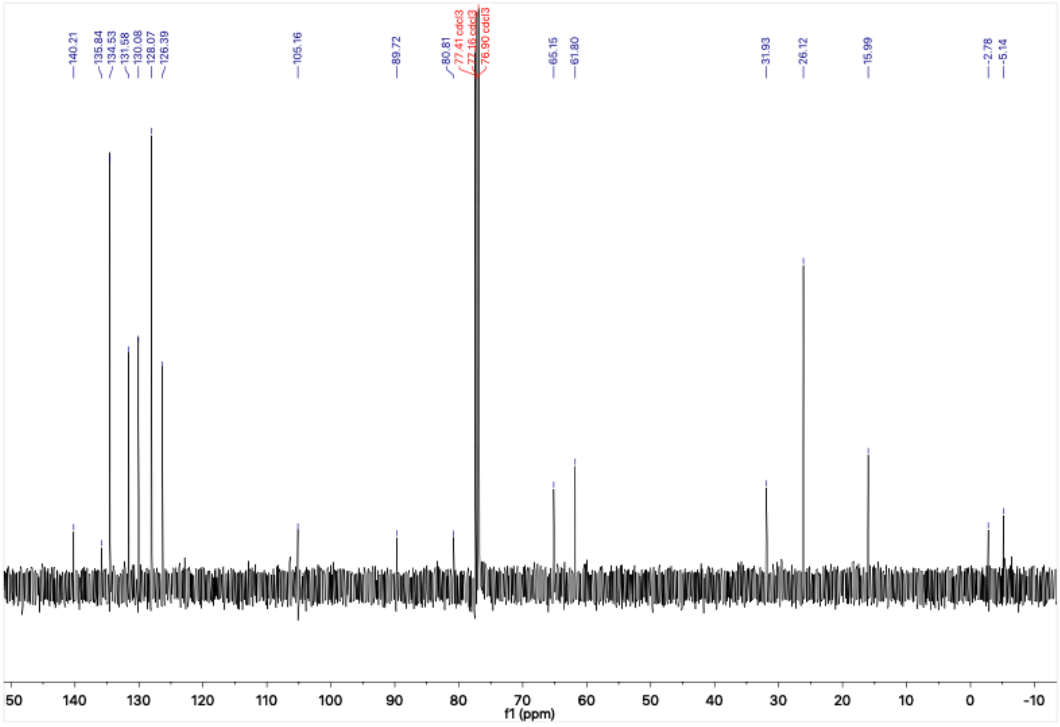
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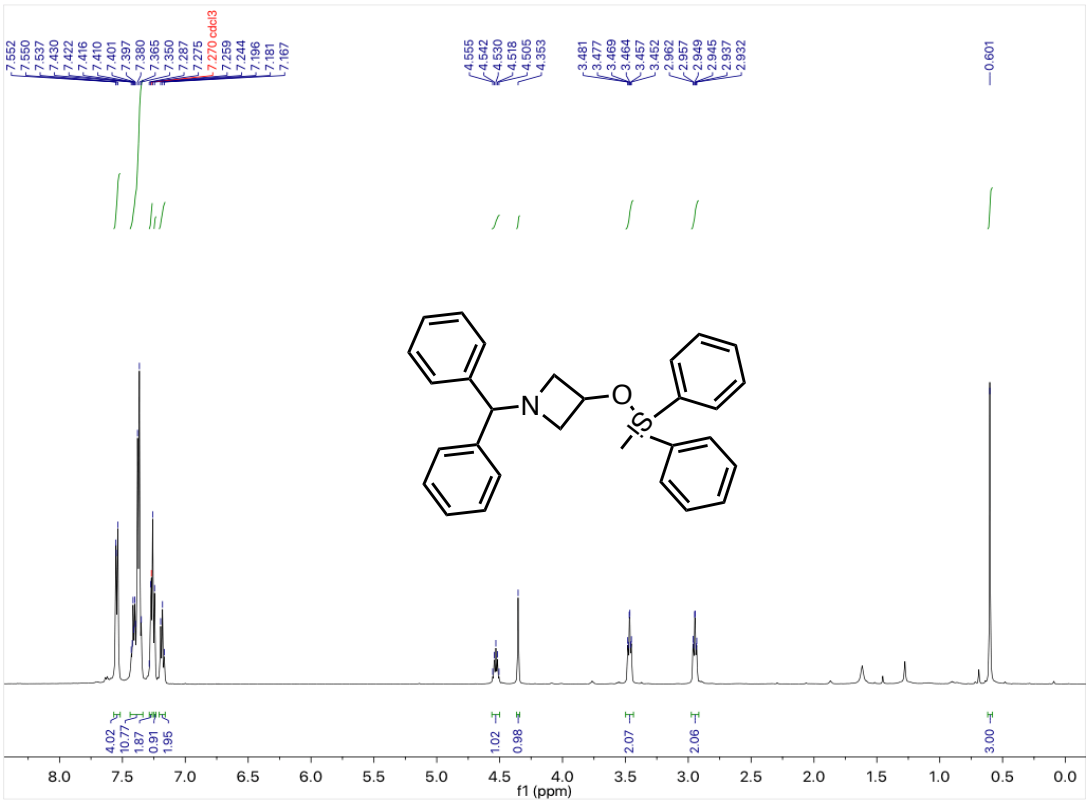
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7.430
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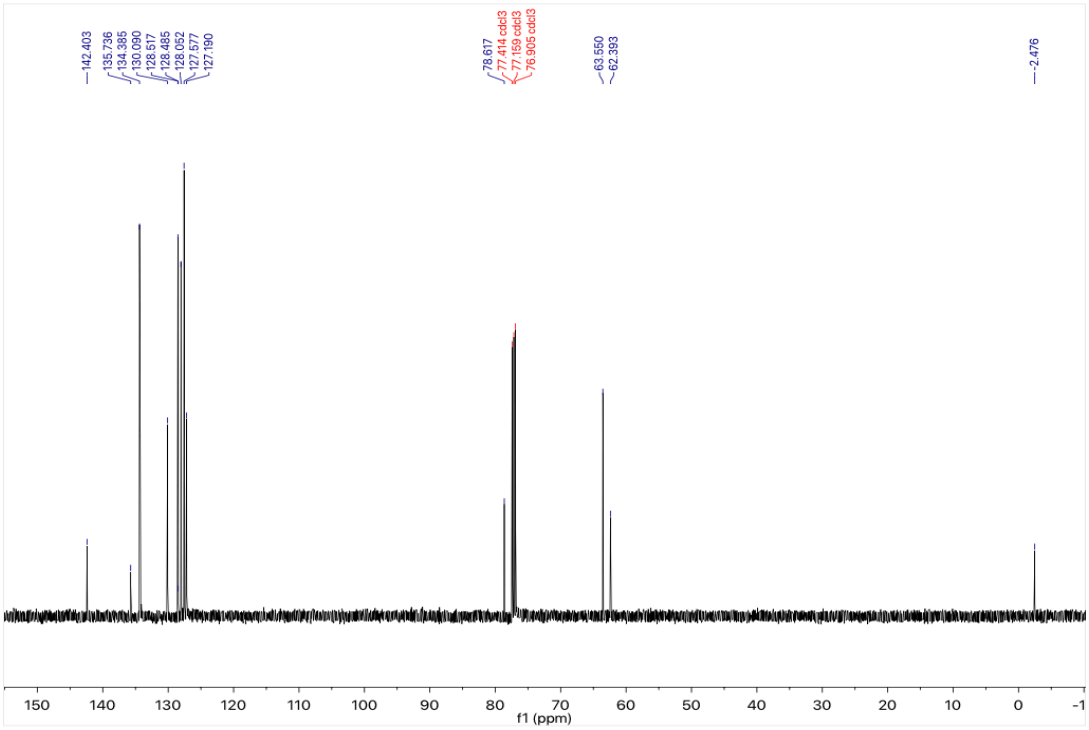
7.416
7.410
7.401
7.397
7.380
7.365
7.350
7.287
7.275
7.270 cdcl3
7.259
7.244
7.196
7.181
7.167
4.555
4.542
4.530
4.518
4.505
4.353
3.481
3.477
3.469
3.464
3.457
3.452
2.962
2.957
2.949
2.945
2.937
2.932
0.601
N
O
Si
4.02
10.77
1.87
0.91
1.95
1.02
0.98
2.07
2.06
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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134.385
130.090
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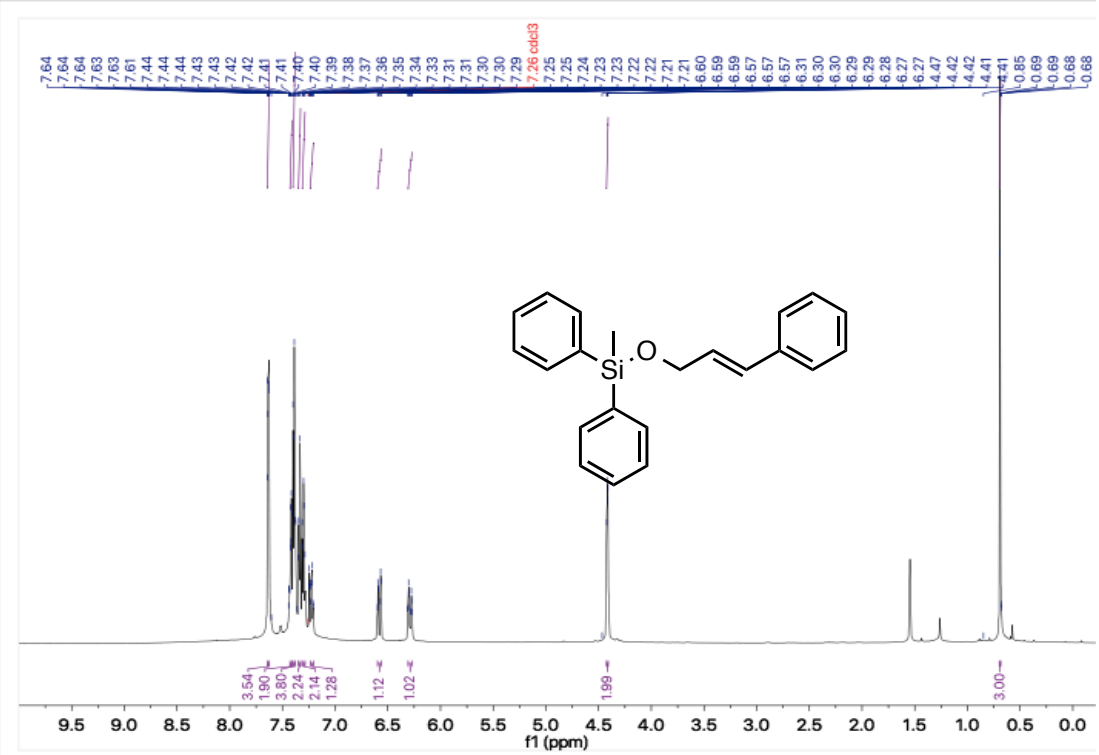
128.485
128.052
127.577
127.190
78.617
77.414 cdcl3
77.159 cdcl3
76.905 cdcl3
63.550
62.393
2.476
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)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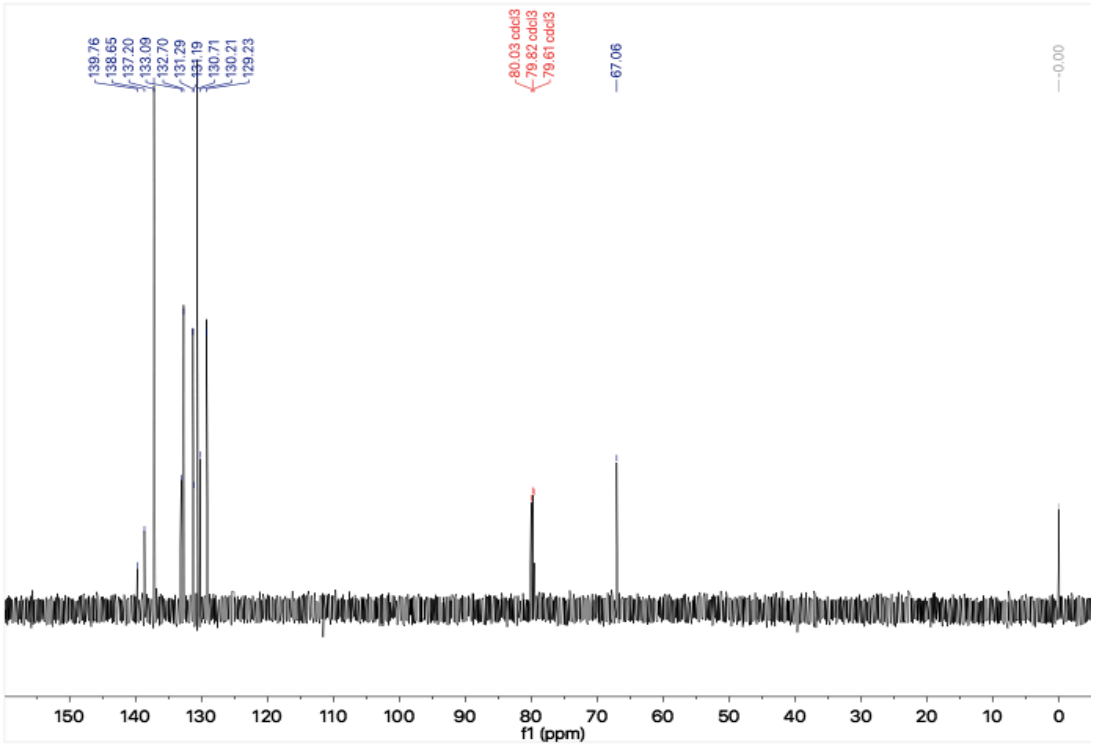
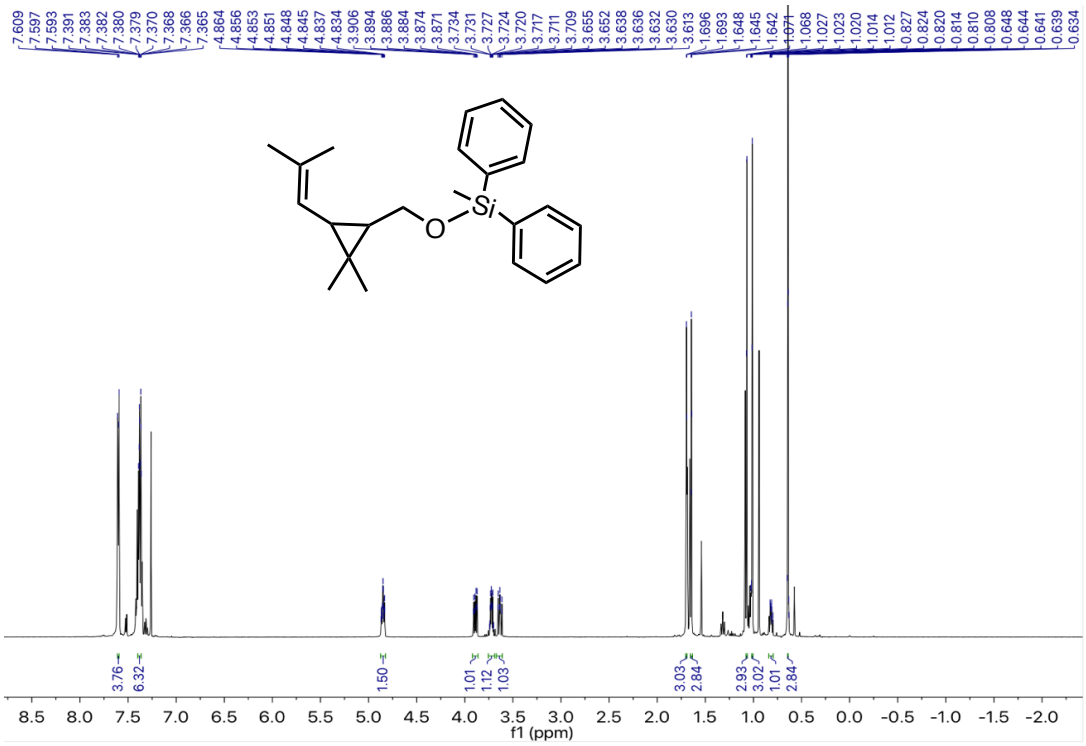
Si
O
3.54 1.90 3.80 2.24 2.14 1.28 1.12 1.02 1.99 3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


139.76 138.65 137.20 133.09 132.70 131.29 131.19 130.71 130.21 129.23
80.03 cdcl3 79.82 cdcl3 79.61 cdcl3
67.06
0.00
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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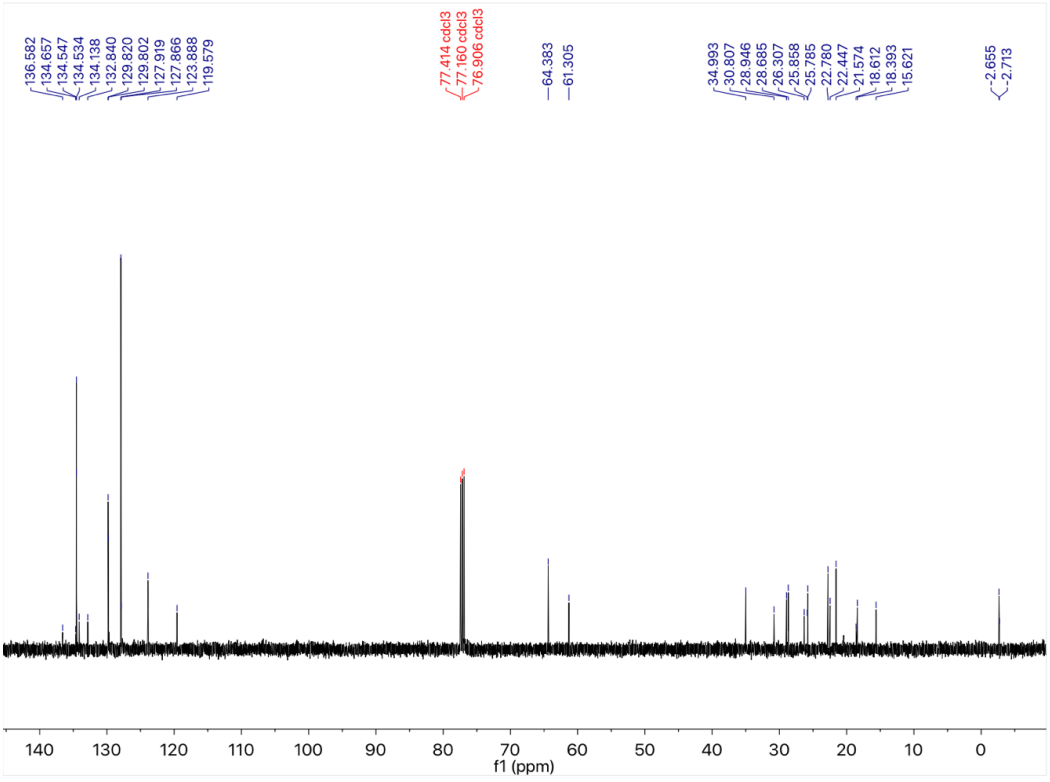
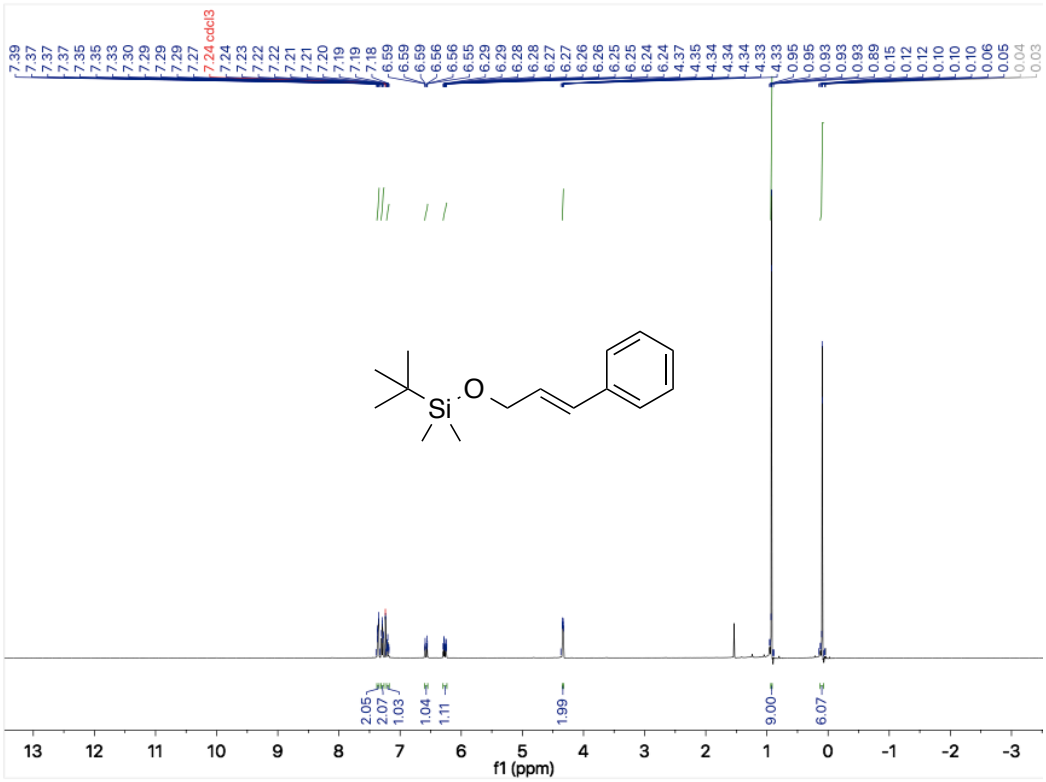
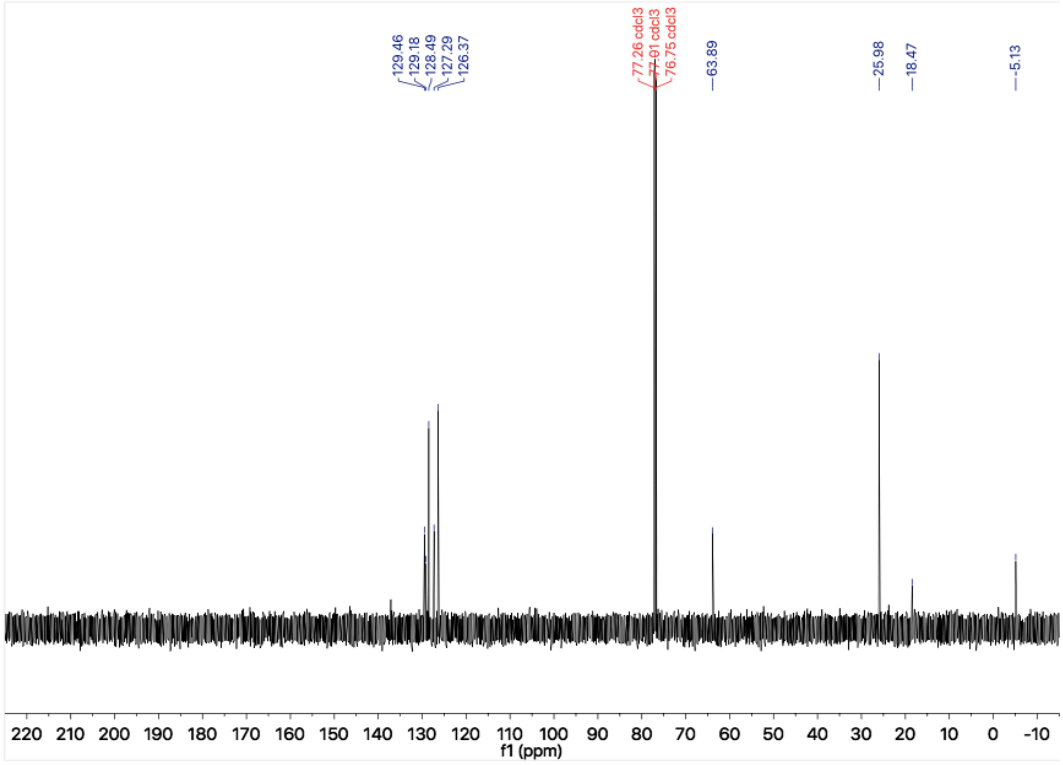
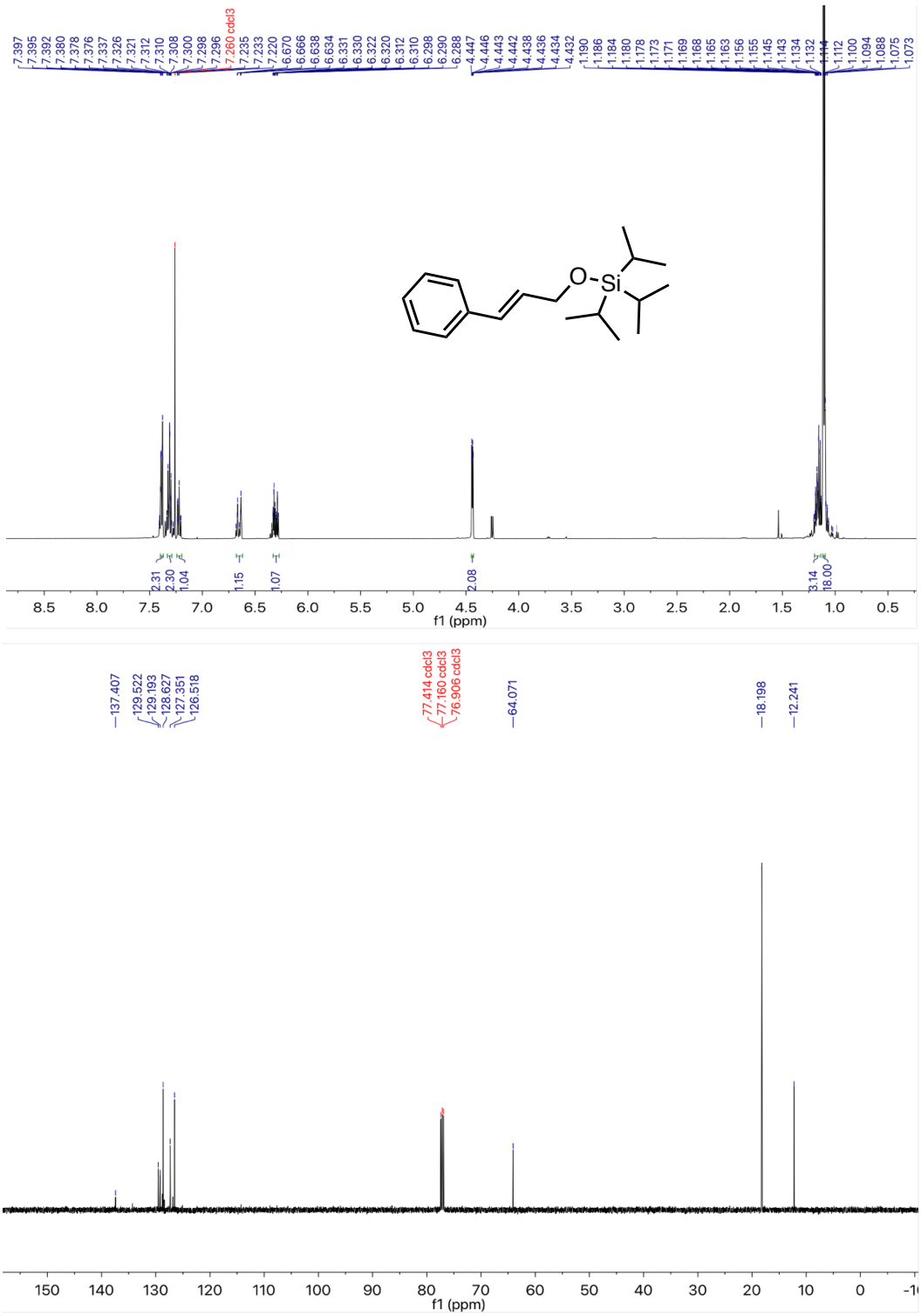
4.864 4.856 4.853 4.851 4.848 4.845 4.837 4.834 3.906 3.894 3.886 3.884 3.874 3.871 3.734 3.731 3.727 3.724 3.720 3.717 3.711 3.709 3.655 3.652 3.638 3.636 3.632 3.630 3.613 1.696 1.693 1.648 1.645 1.642 1.071 1.068 1.027 1.023 1.020 1.014 1.012 0.827 0.824 0.820 0.814 0.810 0.808 0.648 0.644 0.641 0.639 0.634
O
Si
3.76 6.32 1.50 1.01 1.12 1.03 3.03 2.84 2.93 3.02 1.01 2.84
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)


136.582 134.657 134.547 134.534 134.138 132.840 129.820 129.802 127.919 127.866 123.888 119.579
77.414 cdcl3 77.160 cdcl3 76.906 cdcl3
64.383 61.305
34.993 30.807 28.946 28.685 26.307 25.858 25.785 22.780 22.447 21.574 18.612 18.393 15.621
-2.655 -2.713
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.39 7.37 7.37 7.37 7.35 7.35 7.33 7.30 7.29 7.29 7.29 7.27 7.24 cdcl3 7.24 7.23 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.18 6.59 6.59 6.59 6.56 6.56 6.55 6.29 6.29 6.28 6.28 6.27 6.27 6.26 6.26 6.25 6.25 6.24 6.24 4.37 4.35 4.34 4.34 4.33 4.33 0.95 0.95 0.93 0.93 0.93 0.89 0.15 0.12 0.12 0.10 0.10 0.10 0.06 0.05 0.04 0.03
Si
O
2.05 2.07 1.03 1.04 1.11 1.99 9.00 6.07
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


129.46 129.18 128.49 127.29 126.37
77.26 cdcl3 77.01 cdcl3 76.75 cdcl3
63.89
25.98 18.47 5.13
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.310
7.308
7.300
7.298
7.296
7.260 cdcl3
7.235
7.233
7.220
6.670
6.666
6.638
6.634
6.331
6.330
6.322
6.320
6.312
6.310
6.298
6.290
6.288
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4.446
4.443
4.442
4.438
4.436
4.434
4.432
1.190
1.186
1.184
1.180
1.178
1.173
1.171
1.169
1.168
1.165
1.163
1.156
1.155
1.145
1.143
1.134
1.132
1.112
1.100
1.094
1.088
1.075
1.073
O
Si
2.31
2.30
1.04
1.15
1.07
2.08
3.14
18.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
137.407
129.522
129.193
128.627
127.351
126.518
77.414 cdcl3
77.160 cdcl3
76.906 cdcl3
64.071
18.198
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120
110
100
90
80
70
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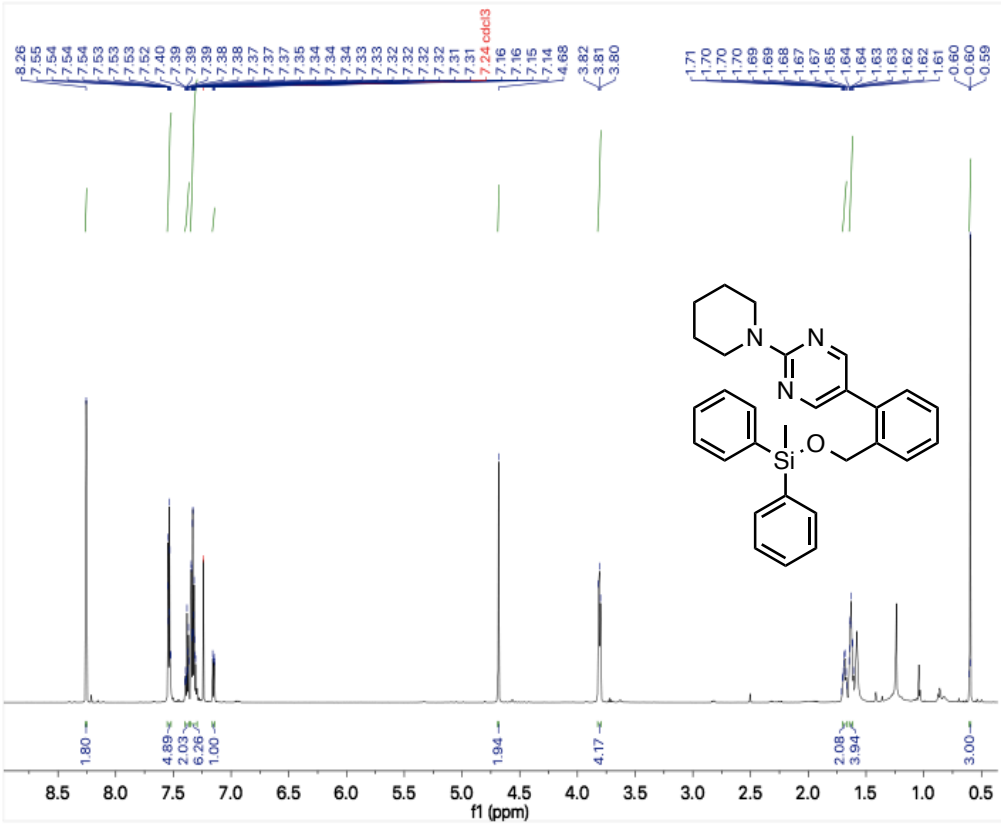
50
40
30
20
10
0
f1 (ppm)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7.53
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7.38
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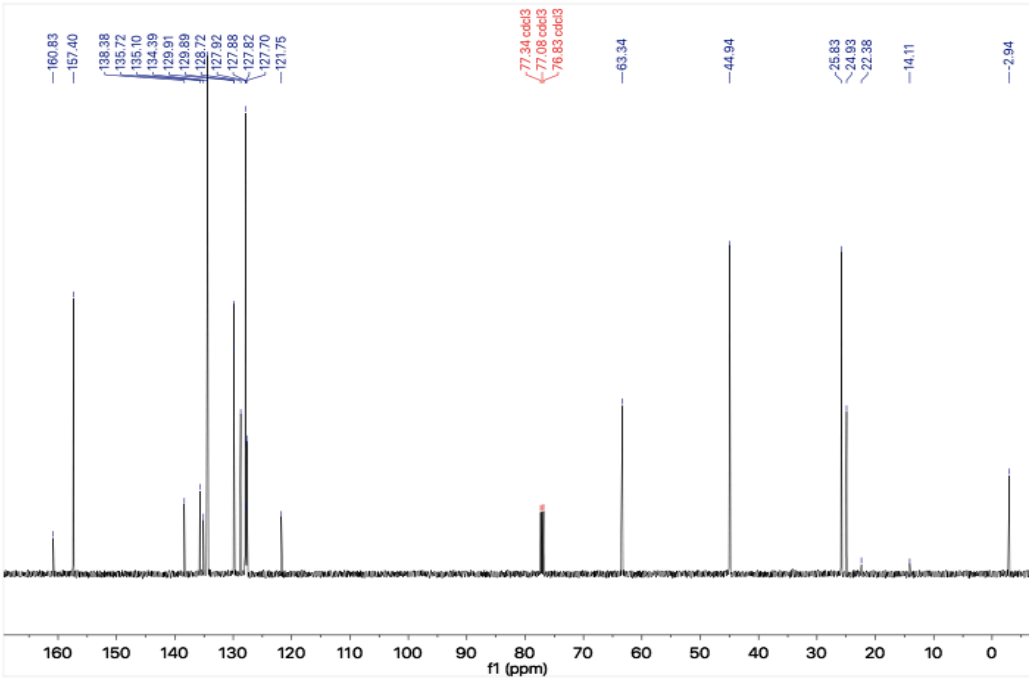
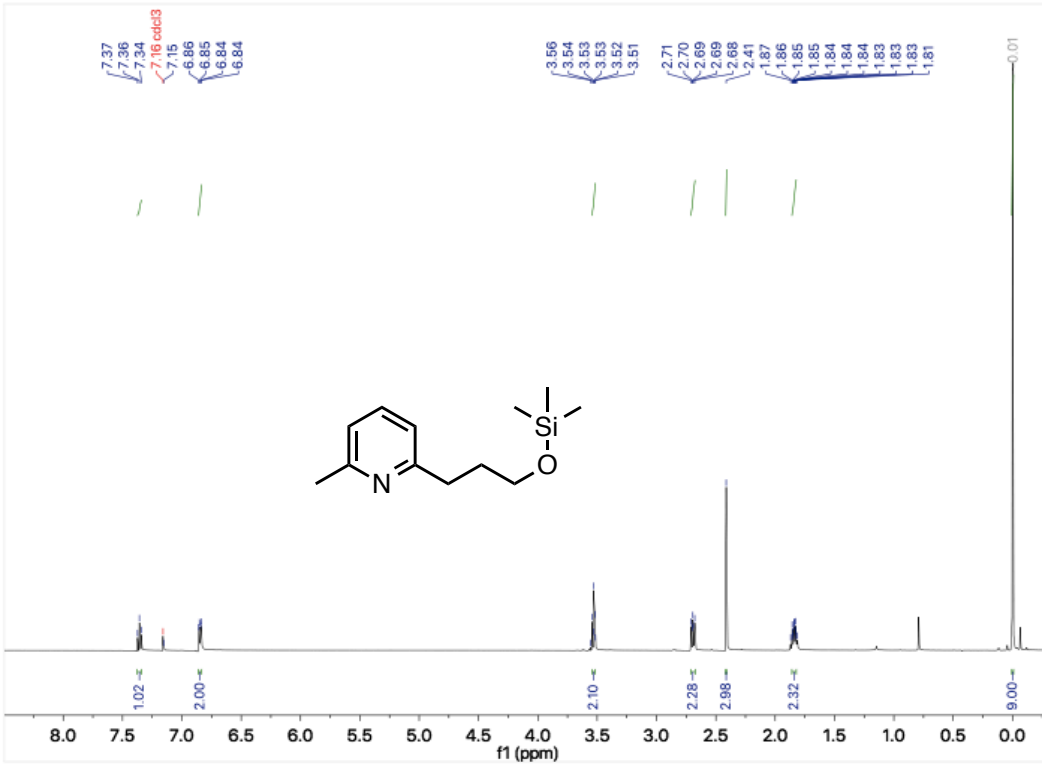
7.37
7.37
7.37
7.35
7.34
7.34
7.34
7.33
7.33
7.32
7.32
7.32
7.31
7.31
7.24 cdcl3
7.16
7.16
7.15
7.14
4.68
3.82
3.81
3.80
1.71
1.70
1.70
1.70
1.69
1.69
1.68
1.67
1.67
1.65
1.64
1.64
1.63
1.63
1.62
1.62
1.61
0.60
0.60
0.59
N
N
N
Si
O
1.80
4.89
2.03
6.28
1.00
1.94
4.17
2.08
3.94
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


160.83
157.40
138.38
135.72
135.10
134.39
129.91
129.89
128.72
127.92
127.88
127.82
127.70
121.75
77.34 cdcl3
77.08 cdcl3
76.83 cdcl3
63.34
44.94
25.83
24.93
22.38
14.11
-2.94
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.37
7.36
7.34
7.16 cdcl3
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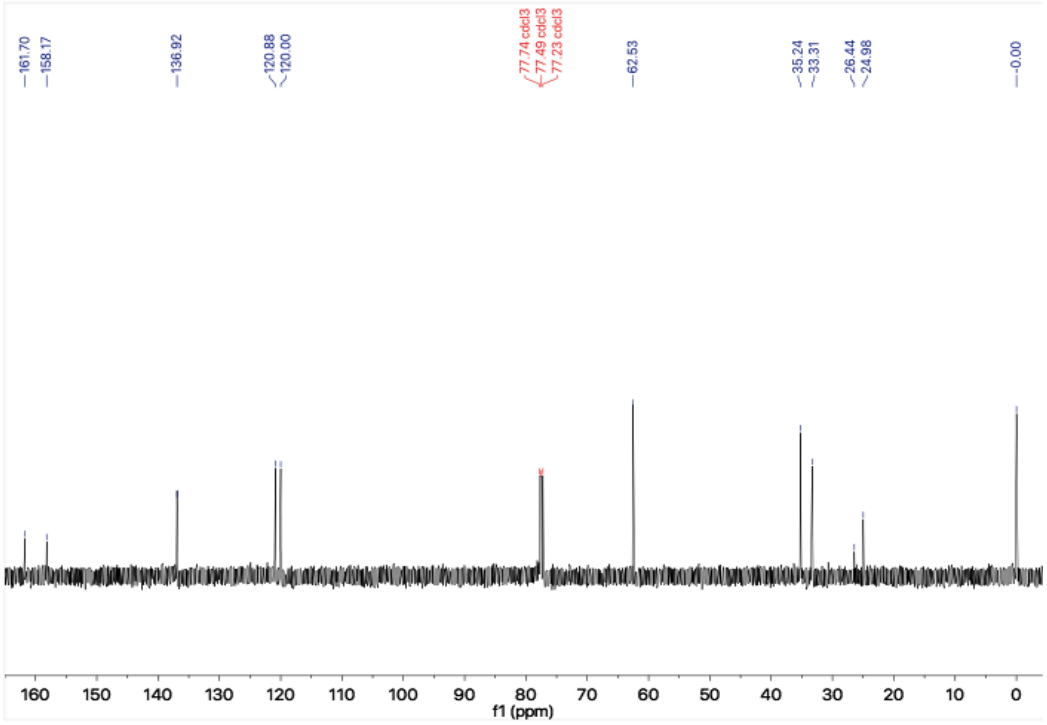
6.85
6.84
6.84
3.56
3.54
3.53
3.53
3.52
3.51
2.71
2.70
2.69
2.69
2.68
2.41
1.87
1.86
1.85
1.85
1.84
1.84
1.84
1.83
1.83
1.81
0.01
Si
O
N
1.02
2.00
2.10
2.28
2.98
2.32
9.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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136.92
120.88
120.00
77.74 cdcl3
77.49 cdcl3
77.23 cdcl3
62.53
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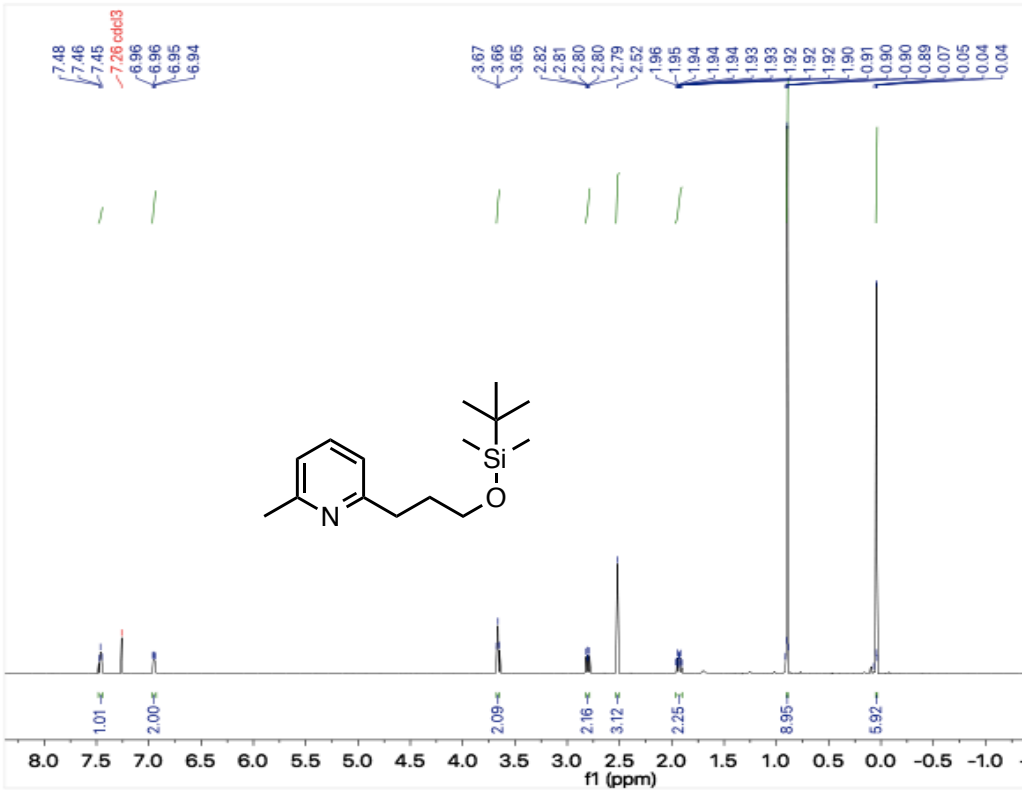
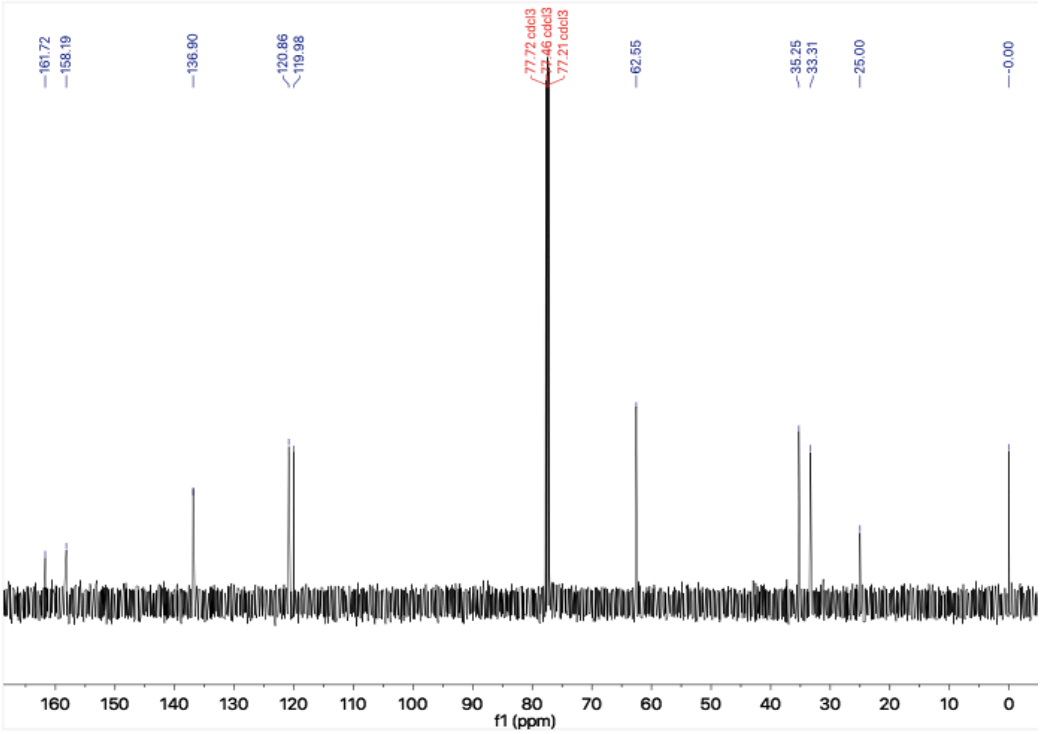
33.31
26.44
24.98
0.00
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.48
7.46
7.45
7.26 cdcl3
6.96
6.96
6.95
6.94
3.67
3.66
3.65
2.82
2.81
2.80
2.80
2.79
2.52
1.96
1.95
1.94
1.94
1.94
1.93
1.93
1.92
1.92
1.92
1.90
0.91
0.90
0.90
0.89
0.07
0.05
0.04
0.04
Si
O
N
1.01
2.00
2.09
2.16
3.12
2.25
8.95
5.92
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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158.19
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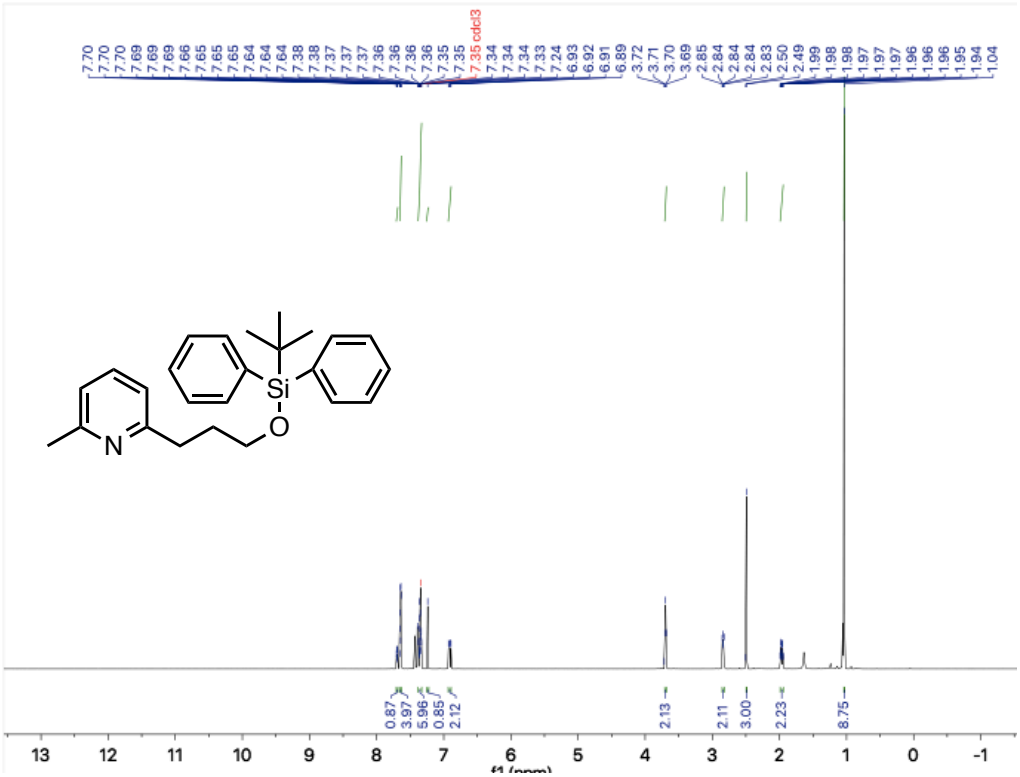
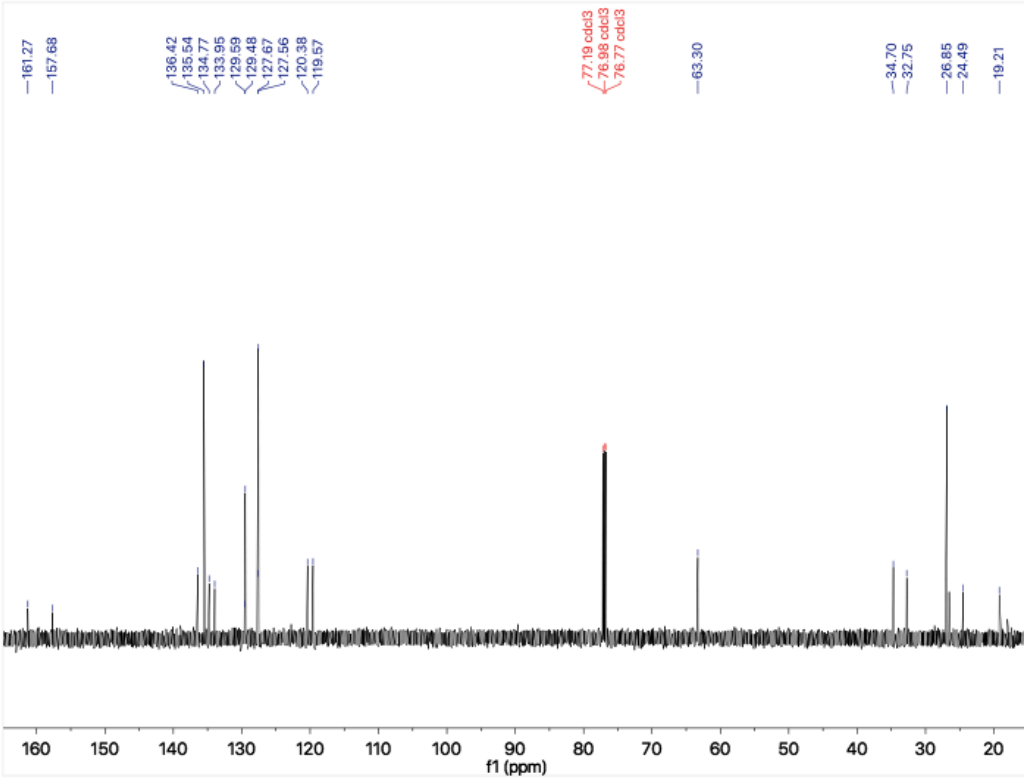
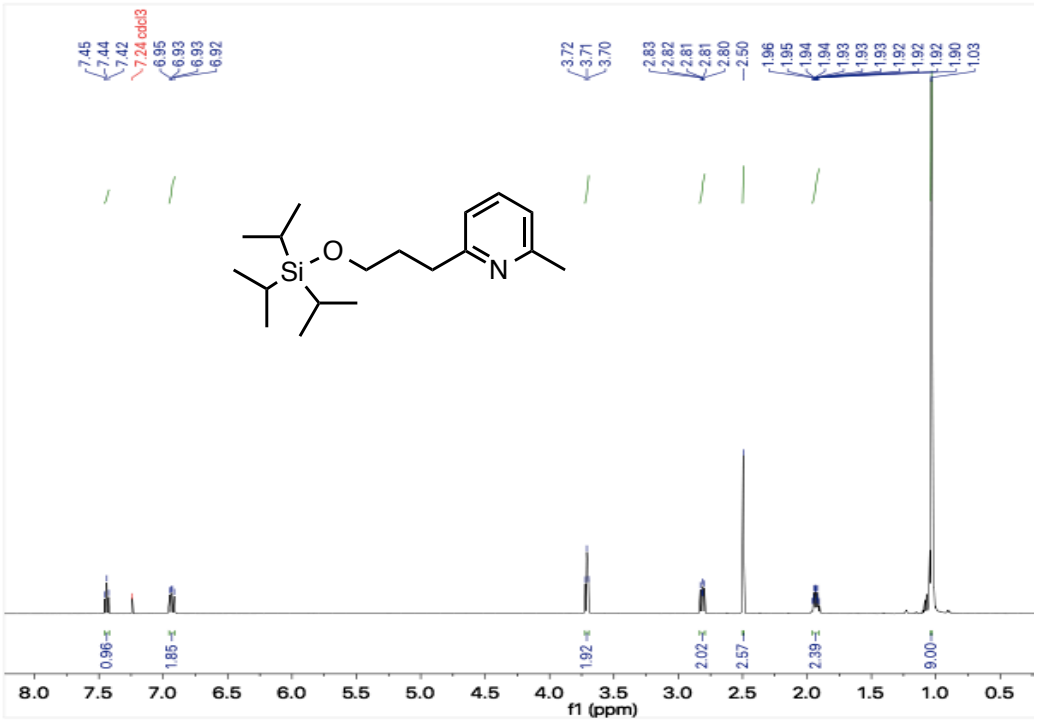
120.86
119.98
77.72 cdcl3
77.46 cdcl3
77.21 cdcl3
62.55
35.25
33.31
25.00
0.00
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.70 7.70 7.70 7.69 7.69 7.69 7.66 7.65 7.65 7.65 7.64 7.64 7.64 7.38 7.38 7.37 7.37 7.37 7.36 7.36 7.36 7.35 7.35 7.35 cdcl3 7.34 7.34 7.34 7.33 7.24 6.93 6.92 6.91 6.89 3.72 3.71 3.70 3.69 2.85 2.84 2.84 2.84 2.83 2.50 2.49 1.99 1.98 1.98 1.97 1.97 1.97 1.96 1.96 1.96 1.95 1.94 1.04
Si
O
N
0.87 3.97 5.96 0.85 2.12 2.13 2.11 3.00 2.23 8.75
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)


161.27 157.68 136.42 135.54 134.77 133.95 129.59 129.48 127.67 127.56 120.38 119.57 77.19 cdcl3 76.98 cdcl3 76.77 cdcl3 63.30 34.70 32.75 26.85 24.49 19.21
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)


7.45
7.44
7.42
7.24 cdcl3
6.95
6.93
6.93
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3.72
3.71
3.70
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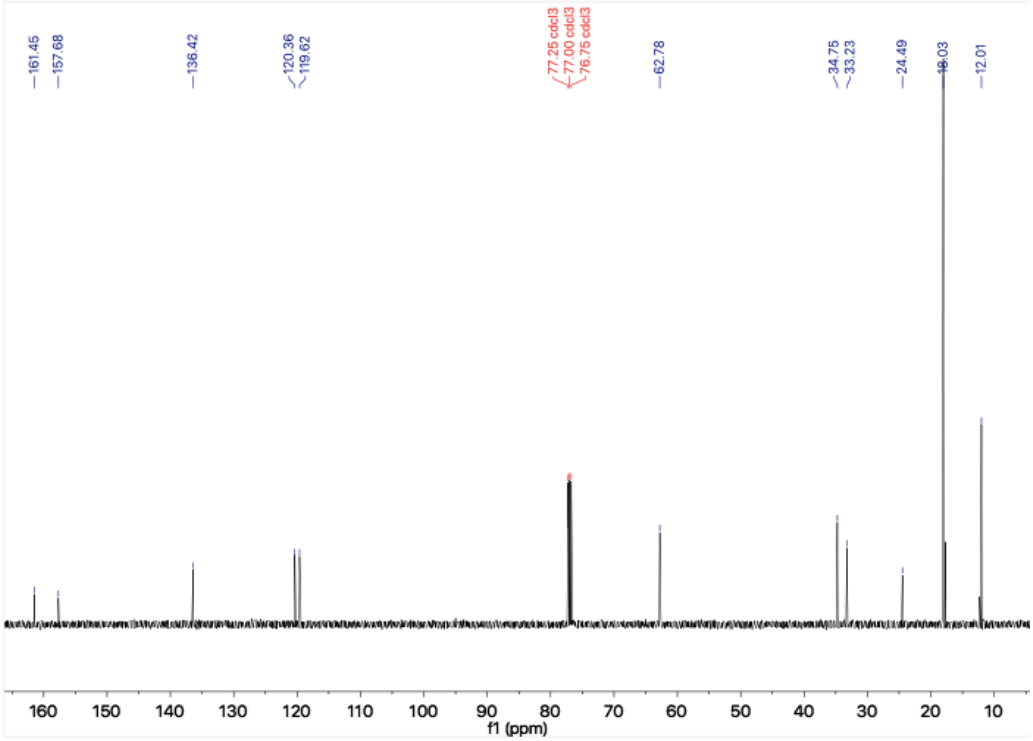
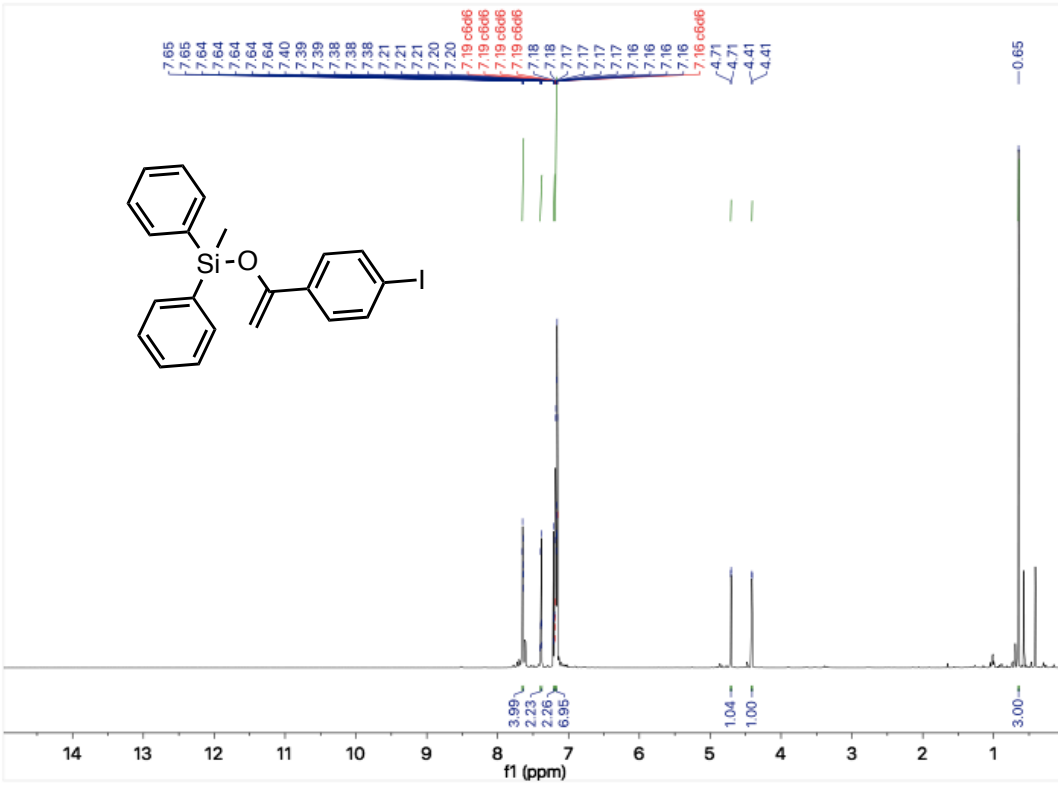
2.82
2.81
2.81
2.80
2.50
1.96
1.95
1.94
1.94
1.93
1.93
1.92
1.92
1.90
1.03
Si
O
N
0.96
1.85
1.92
2.02
2.57
2.39
9.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


161.45
157.68
136.42
120.36
119.62
77.25 cdcl3
77.00 cdcl3
76.75 cdcl3
62.78
34.75
33.23
24.49
18.03
12.01
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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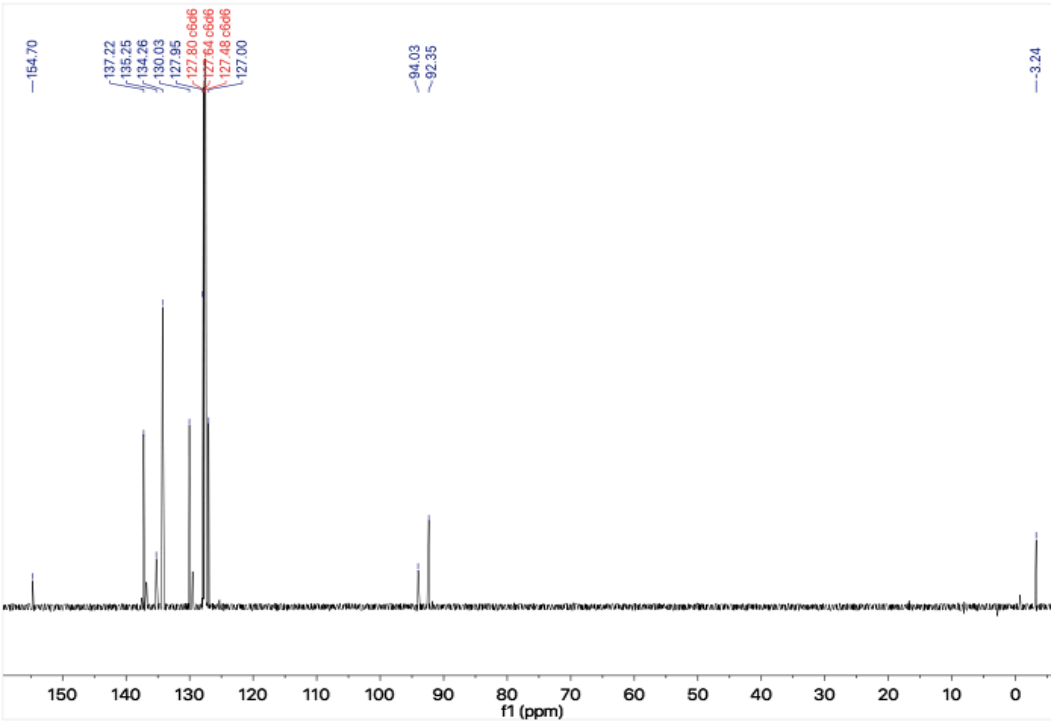
7.64
7.40
7.39
7.38
7.21
7.20
7.19 c6d6
7.18
7.17
7.16
7.16 c6d6
4.71
4.41
0.65
Si
O
I
3.99
2.23
2.26
6.95
1.04
1.00
3.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1
f1 (ppm)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137.22
135.25
134.26
130.03
127.95
127.80 c6d6
127.64 c6d6
127.48 c6d6
127.00
94.03
92.35
3.24
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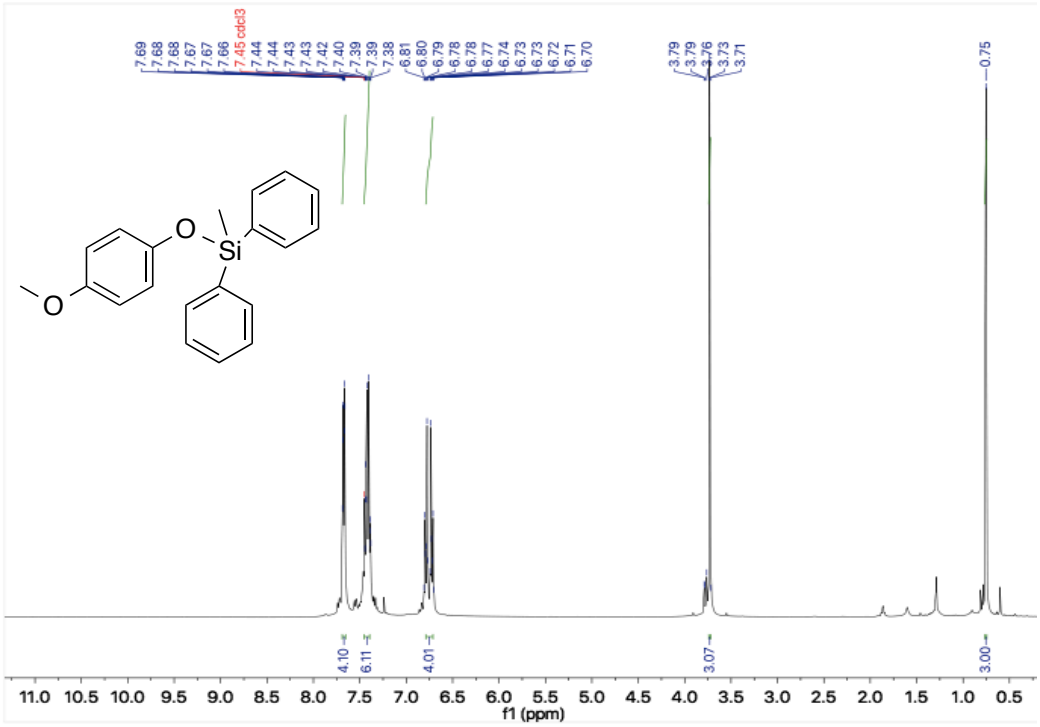
f1 (ppm)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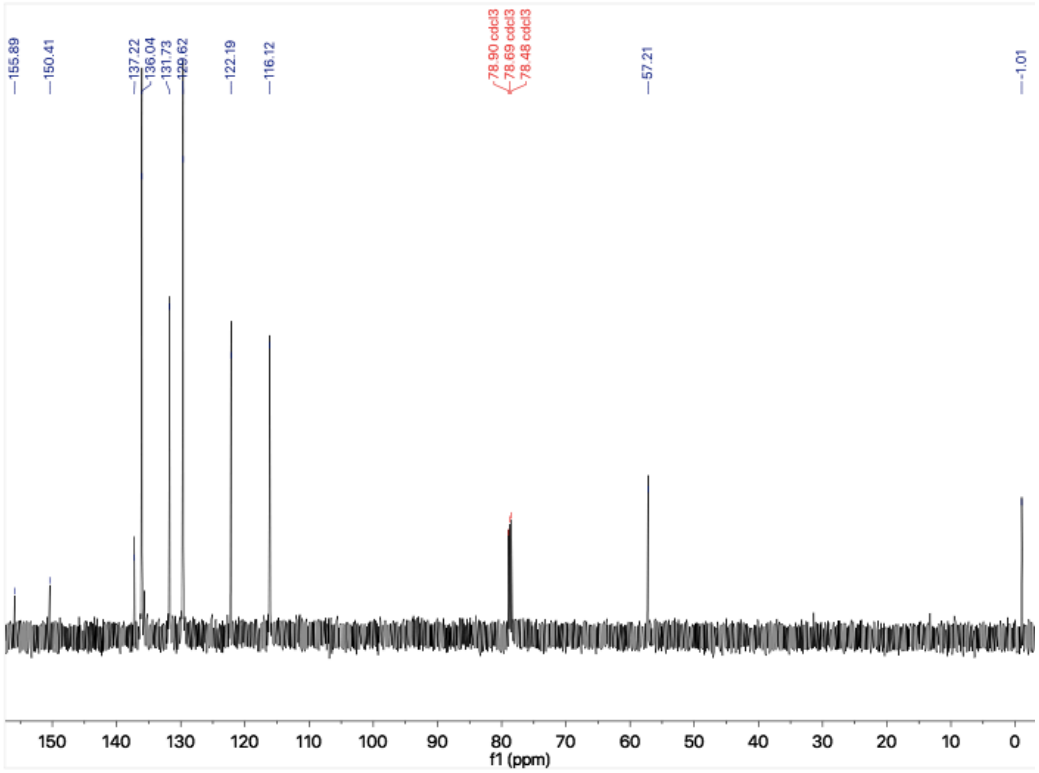
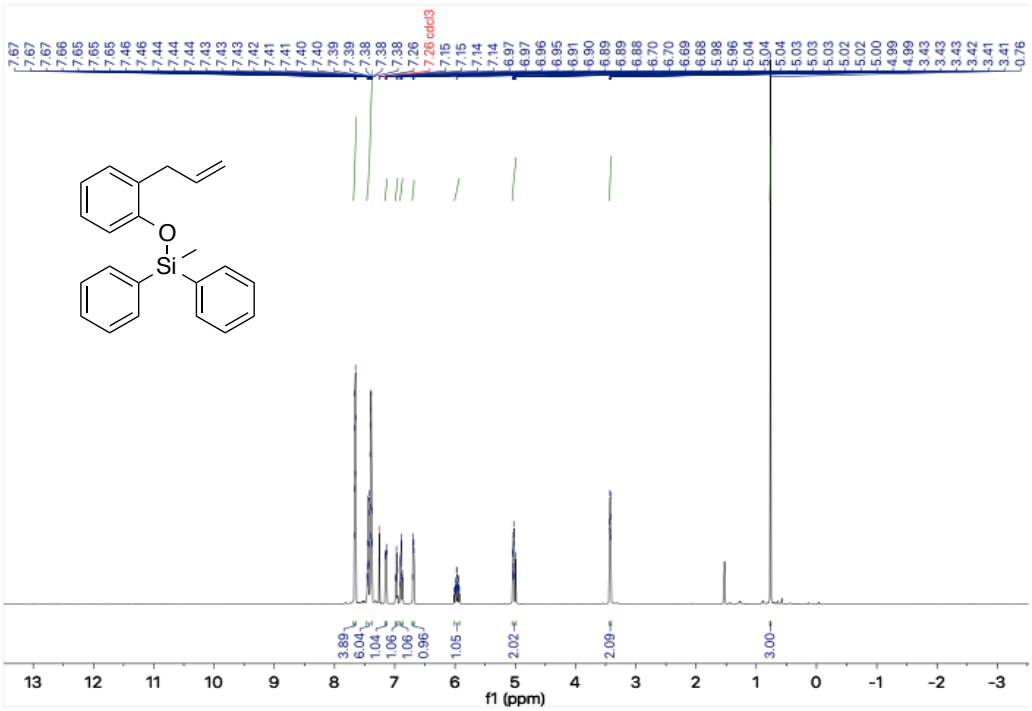
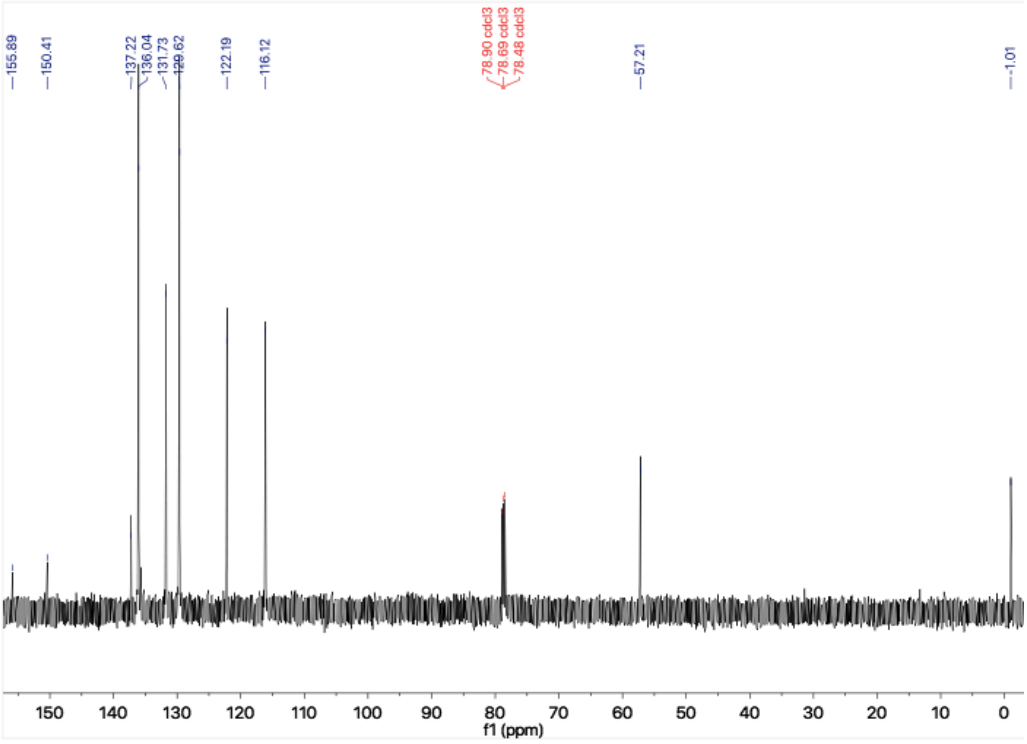
7.68
7.68
7.67
7.67
7.66
7.45 cdcl3
7.44
7.44
7.43
7.43
7.42
7.40
7.39
7.39
7.38
6.81
6.80
6.79
6.78
6.78
6.77
6.74
6.73
6.73
6.72
6.71
6.70
3.79
3.79
3.76
3.73
3.71
0.75
O
Si
O
4.10
6.11
4.01
3.07
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


155.89
150.41
137.22
136.04
131.73
129.62
122.19
116.12
78.90 cdcl3
78.69 cdcl3
78.48 cdcl3
57.21
-1.01
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
Si
f1 (ppm)


155.89
150.41
137.22
136.04
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116.12
78.90 cdcl3
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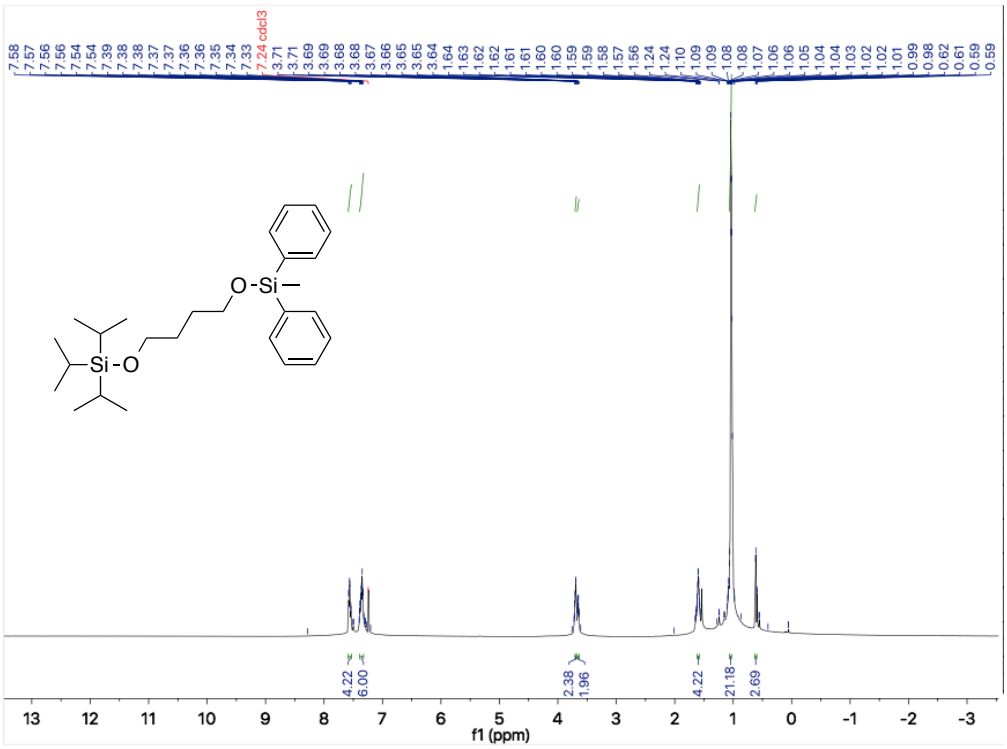
78.48 cdcl3
57.21
-1.01
f1 (ppm)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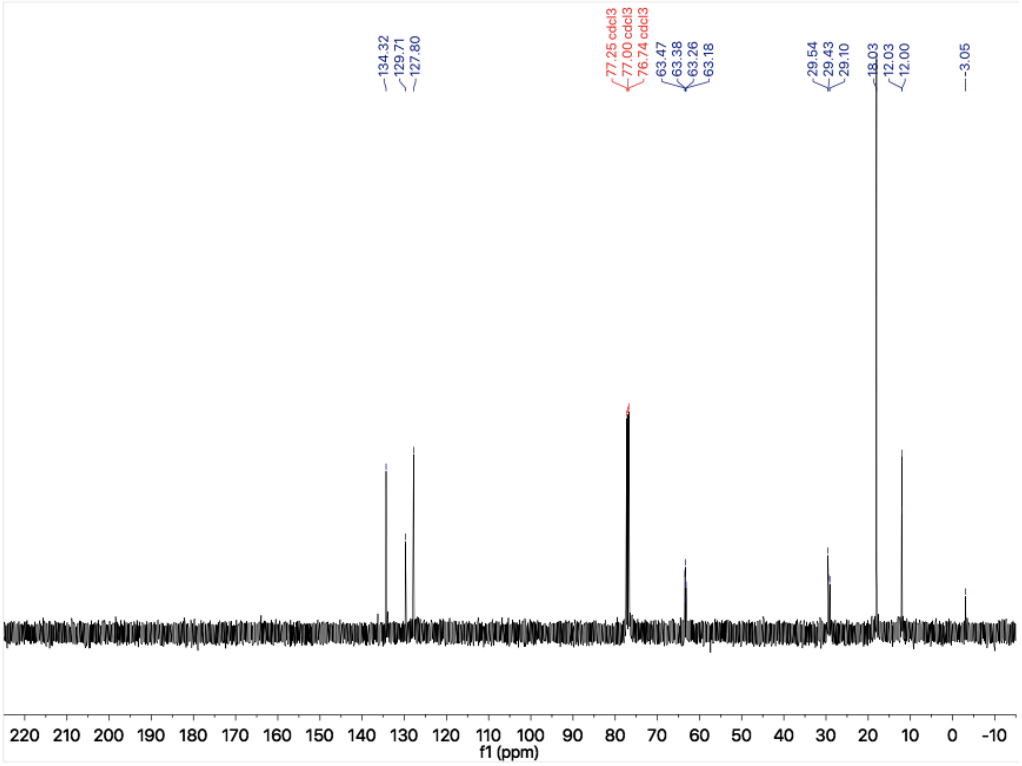
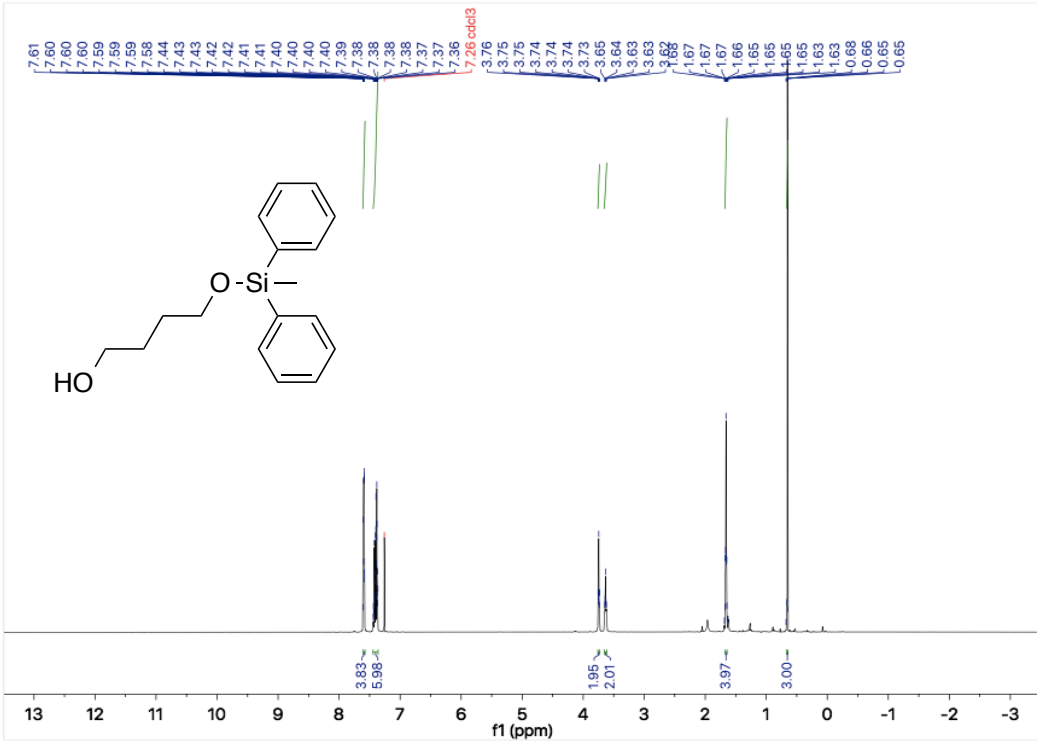
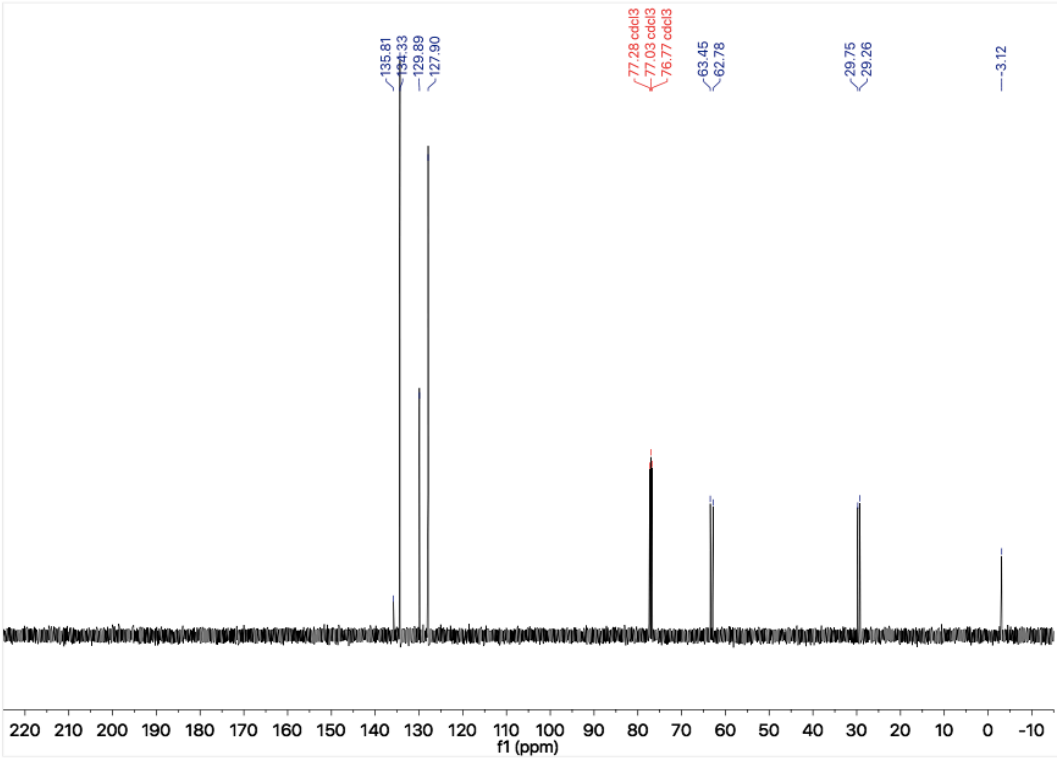
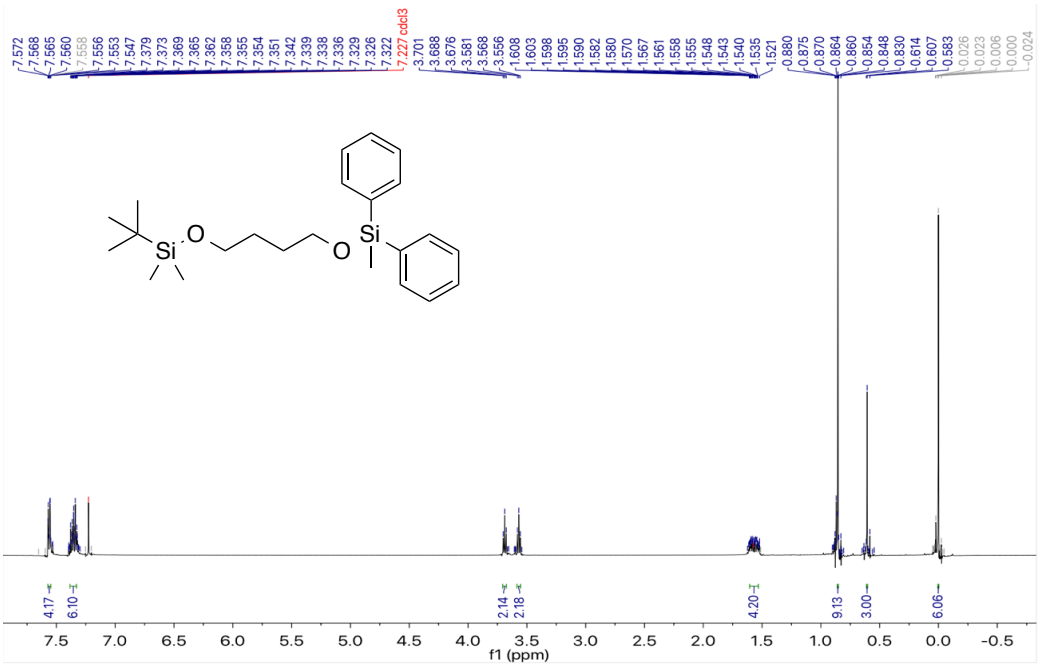
O-Si
Si-O
4.22 6.00 2.38 1.96 4.22 21.18 2.69
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


134.32 129.71 127.80
77.25 cdcl3 77.00 cdcl3 76.74 cdcl3
63.47 63.38 63.26 63.18
29.54 29.43 29.10 18.03 12.03 12.00 -3.05
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


O-Si
HO
f1 (ppm)


f1 (ppm)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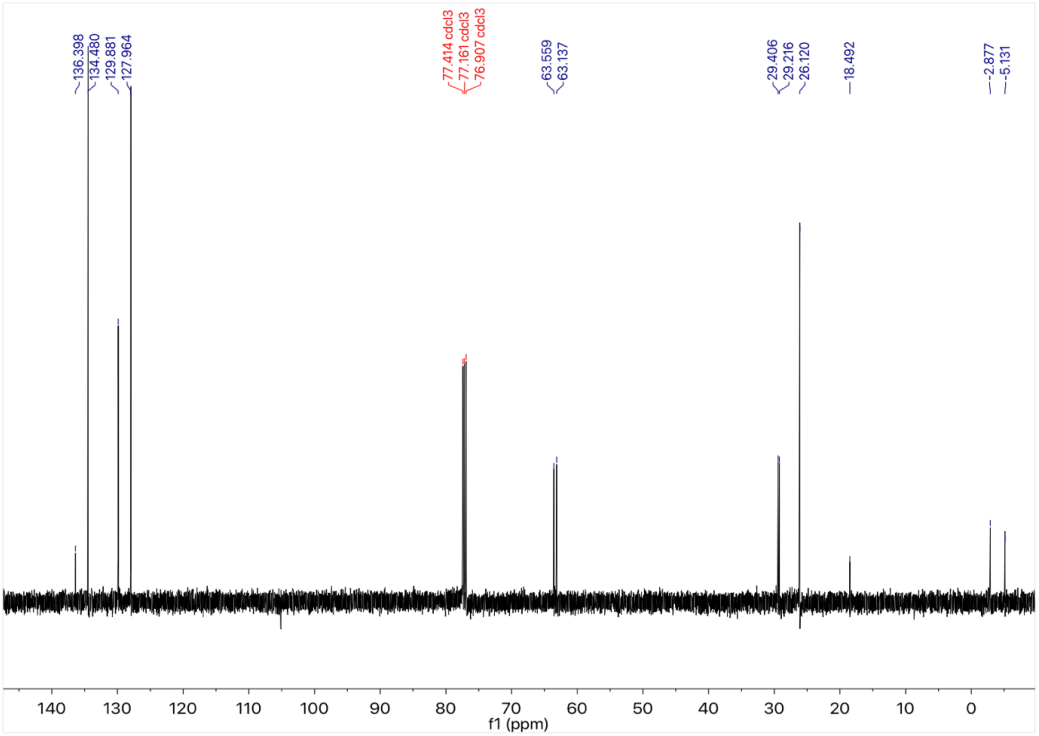
7.556
7.553
7.547
7.379
7.373
7.369
7.365
7.362
7.358
7.355
7.354
7.351
7.342
7.339
7.338
7.336
7.329
7.326
7.322
7.227 cdcl3
3.701
3.688
3.676
3.581
3.568
3.556
1.608
1.603
1.598
1.595
1.590
1.582
1.580
1.570
1.567
1.561
1.558
1.555
1.548
1.543
1.540
1.535
1.521
0.880
0.875
0.870
0.864
0.860
0.854
0.848
0.830
0.614
0.607
0.583
0.026
0.023
0.006
0.000
-0.024
Si
O
O
Si
4.17
6.10
2.14
2.18
4.20
9.13
3.00
6.06
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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129.881
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77.161 cdcl3
76.907 cdcl3
63.559
63.137
29.406
29.216
26.120
18.492
-2.877
-5.131
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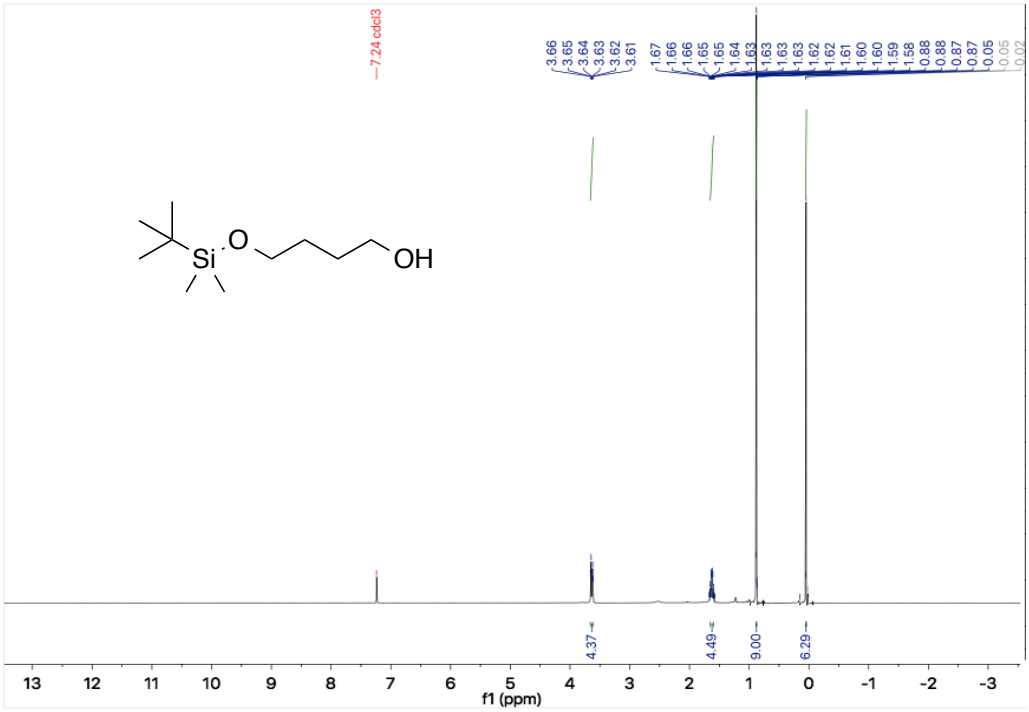
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Si
O
OH
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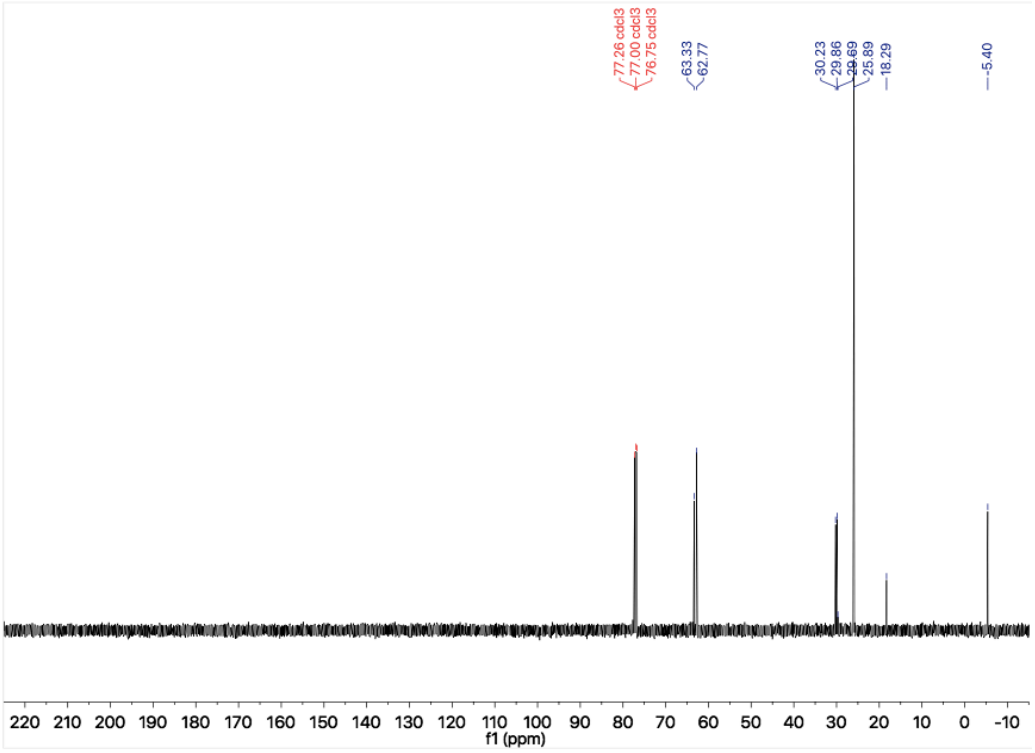
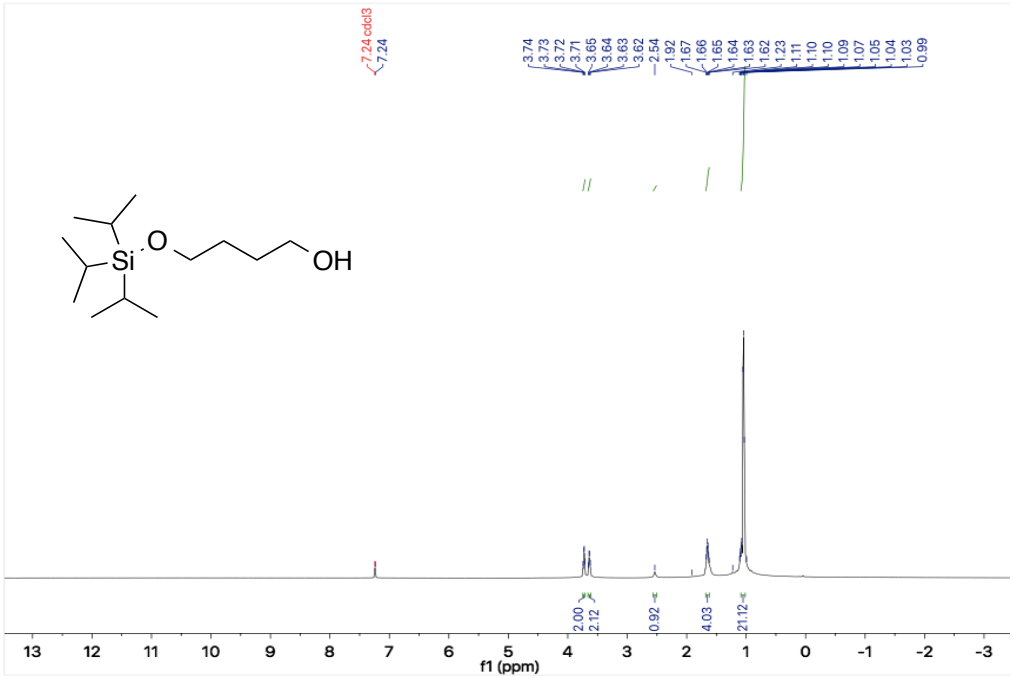
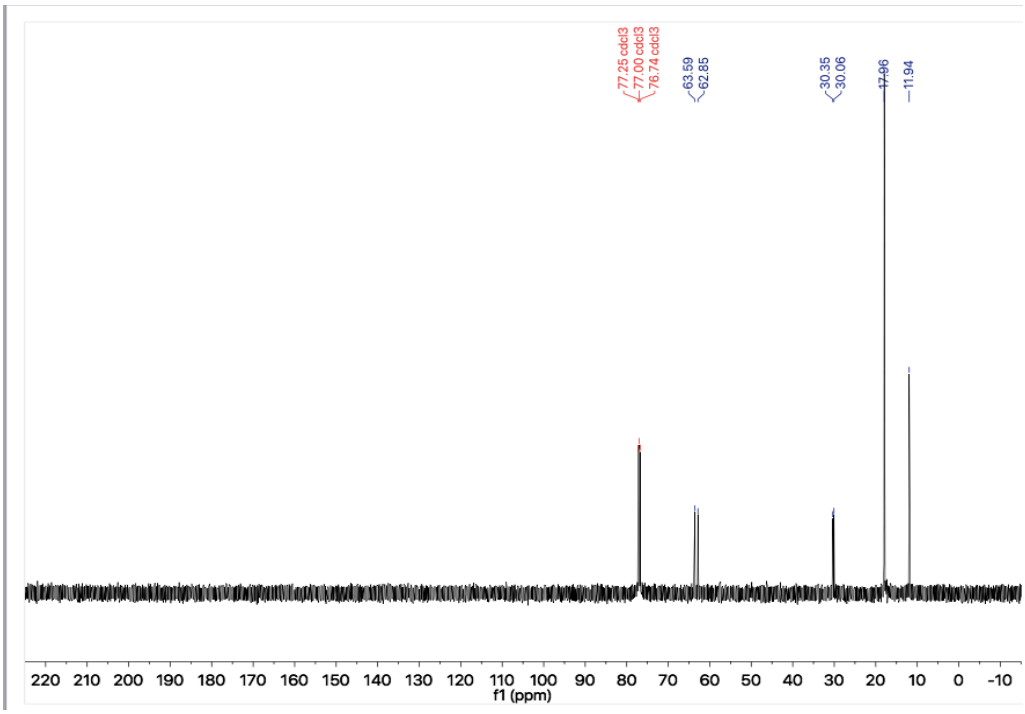
3.66
3.65
3.64
3.63
3.62
3.61
1.67
1.66
1.66
1.65
1.65
1.64
1.63
1.63
1.63
1.63
1.62
1.62
1.61
1.60
1.60
1.59
1.58
0.88
0.88
0.87
0.87
0.05
0.05
0.02
4.37
4.49
9.00
6.29
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


77.26 cdcl3
77.00 cdcl3
76.75 cdcl3
63.33
62.77
30.23
29.86
29.69
25.89
18.29
-5.40
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.24 cdcl3
7.24
3.74
3.73
3.72
3.71
3.65
3.64
3.63
3.62
2.54
1.92
1.67
1.66
1.65
1.64
1.63
1.62
1.23
1.11
1.10
1.10
1.09
1.07
1.05
1.04
1.03
0.99
Si
O
OH
2.00
2.12
0.92
4.03
21.12
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)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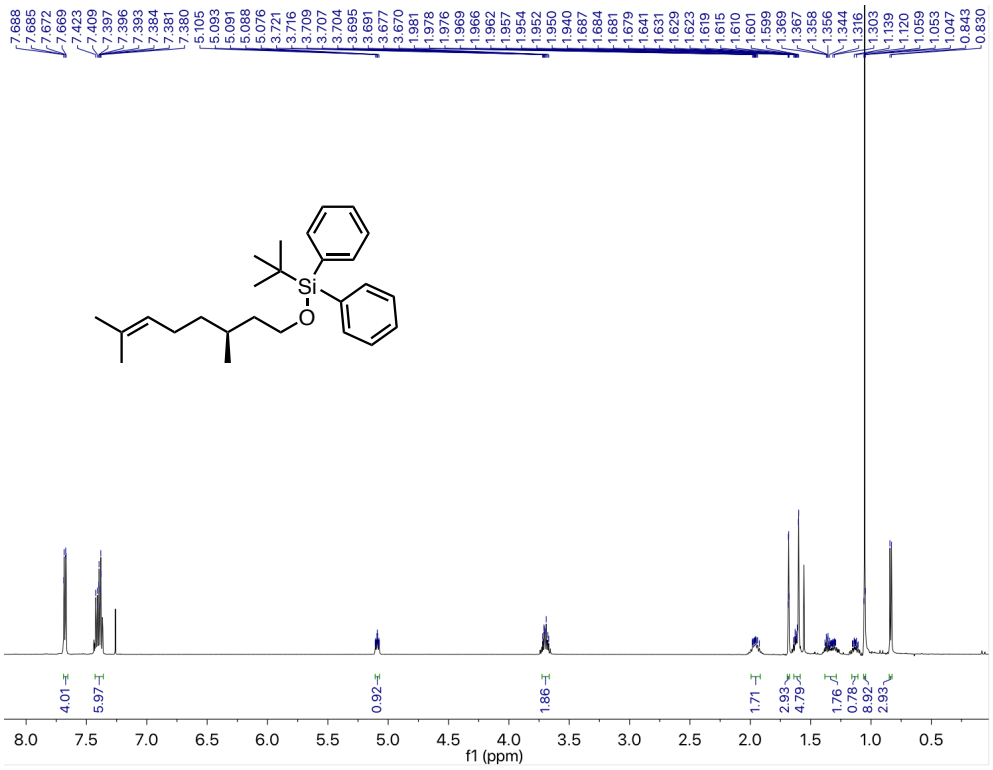
77.00 cdcl3
76.74 cdcl3
63.59
62.85
30.35
30.06
17.06
11.94
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.396
7.393
7.384
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7.380
5.105
5.093
5.091
5.088
5.076
3.721
3.716
3.709
3.707
3.704
3.695
3.691
3.677
3.670
1.981
1.978
1.976
1.969
1.966
1.962
1.957
1.954
1.952
1.950
1.940
1.687
1.684
1.681
1.679
1.641
1.631
1.629
1.623
1.619
1.615
1.610
1.601
1.599
1.369
1.367
1.358
1.356
1.344
1.316
1.303
1.139
1.120
1.059
1.053
1.047
0.843
0.830
Si
O
4.01
5.97
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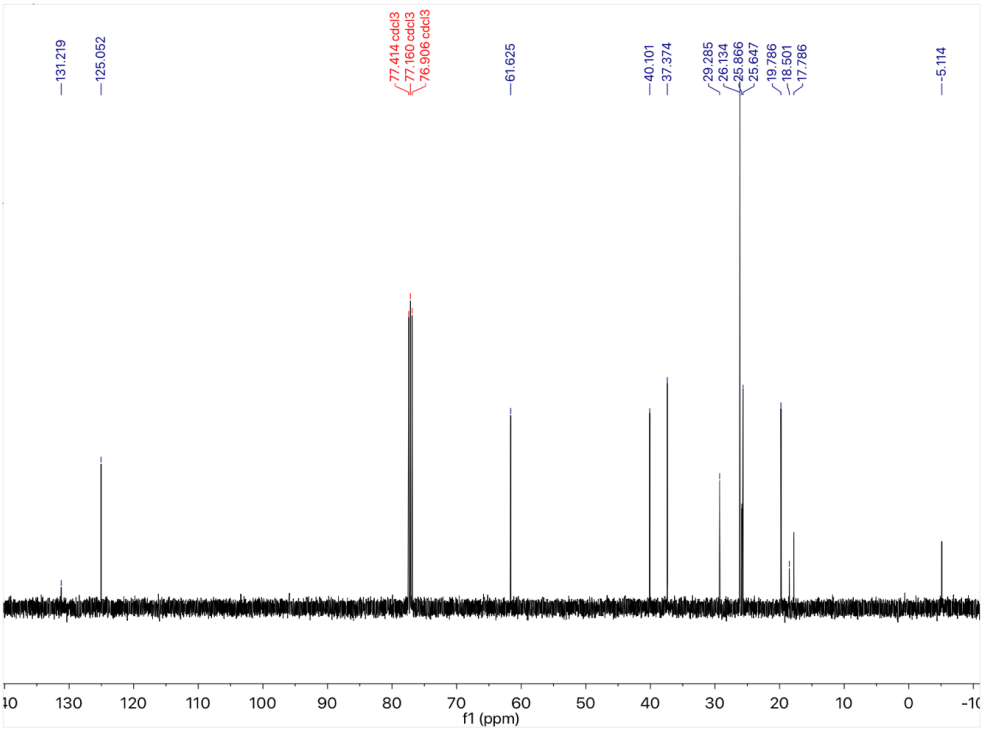
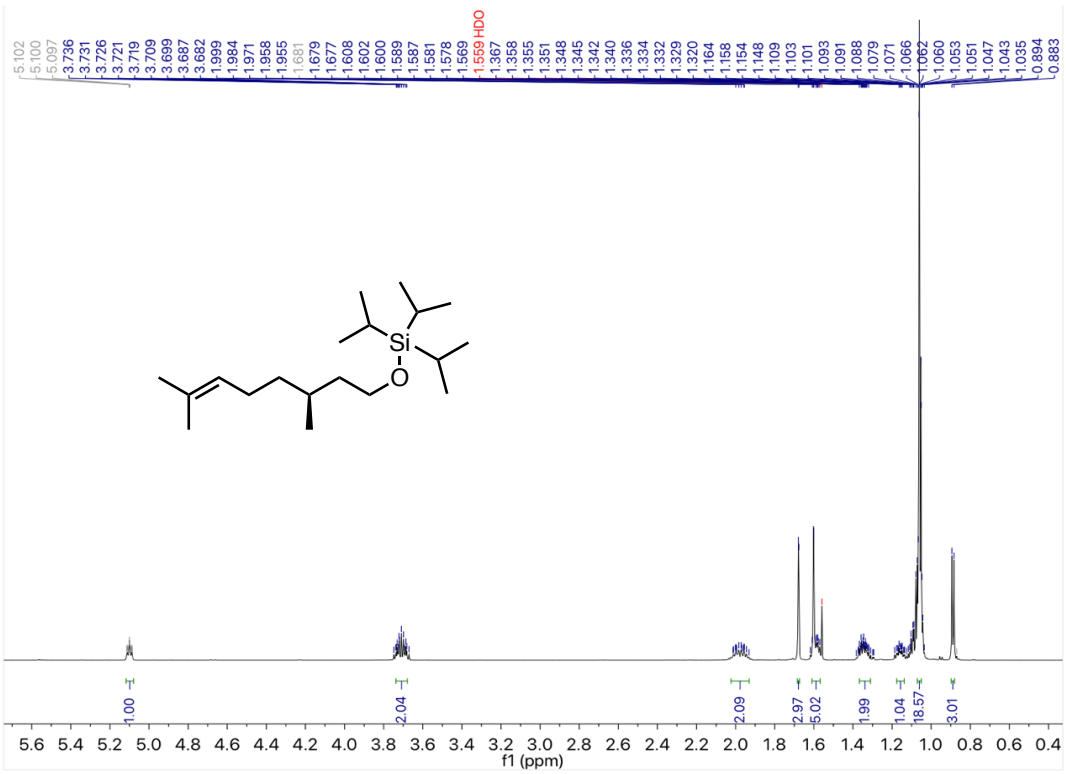
1.71
2.93
4.79
1.76
0.78
8.92
2.93
f1 (ppm)


131.219
125.052
77.414 cdcl3
77.160 cdcl3
76.906 cdcl3
61.625
40.101
37.374
29.285
26.134
25.866
25.647
19.786
18.501
17.786
-5.114
f1 (ppm)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3.726
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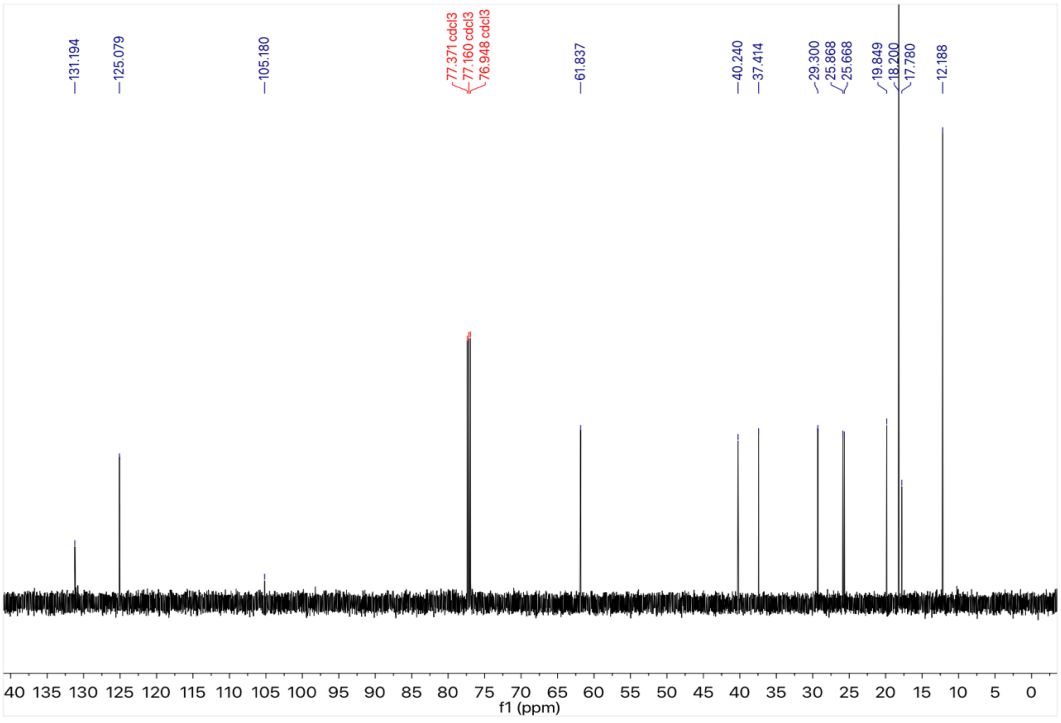
3.719
3.709
3.699
3.687
3.682
1.999
1.984
1.971
1.958
1.955
1.681
1.679
1.677
1.608
1.602
1.600
1.589
1.587
1.581
1.578
1.569
1.559 HDO
1.367
1.358
1.355
1.351
1.348
1.345
1.342
1.340
1.336
1.334
1.332
1.329
1.320
1.164
1.158
1.154
1.148
1.109
1.103
1.101
1.093
1.091
1.088
1.079
1.071
1.066
1.062
1.060
1.053
1.051
1.047
1.043
1.035
0.894
0.883
Si
O
1.00
2.04
2.09
2.97
5.02
1.99
1.04
18.57
3.01
5.6 5.4 5.2 5.0 4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8 0.6 0.4
f1 (ppm)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76.948 cdcl3
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25.868
25.668
19.849
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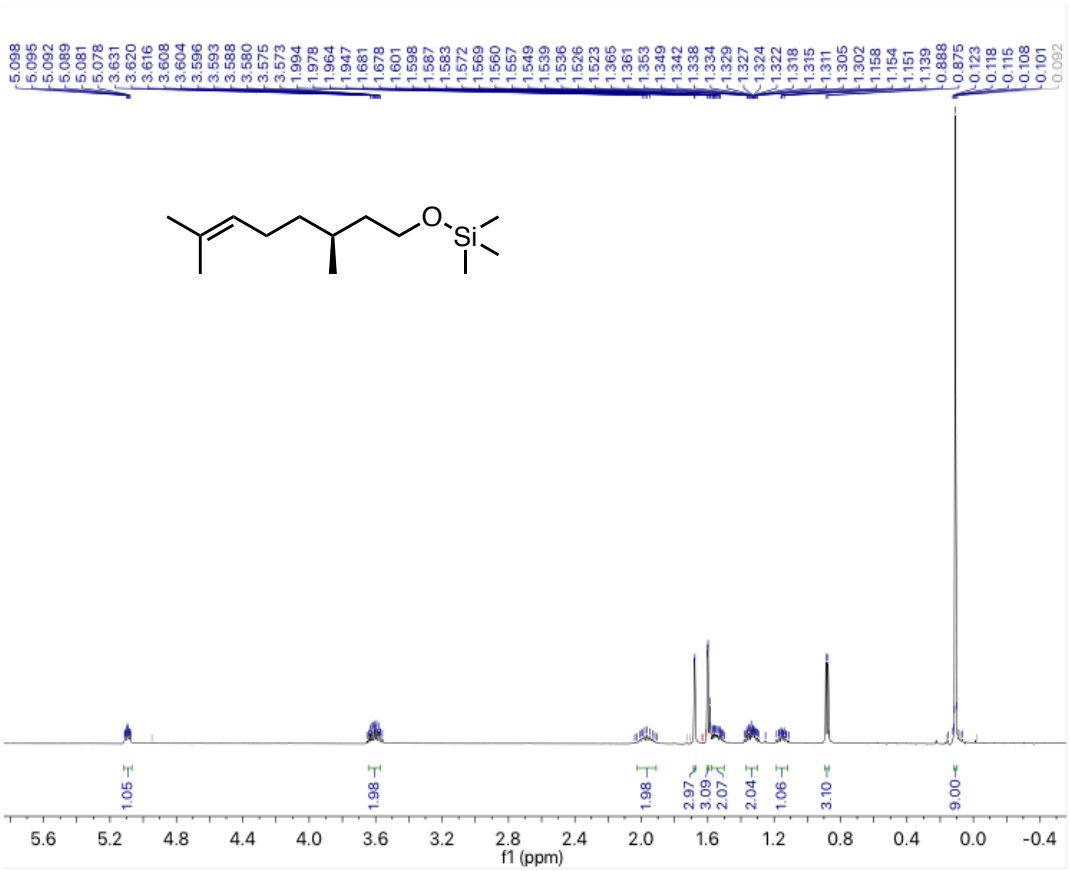
17.780
12.188
40 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)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3.608
3.604
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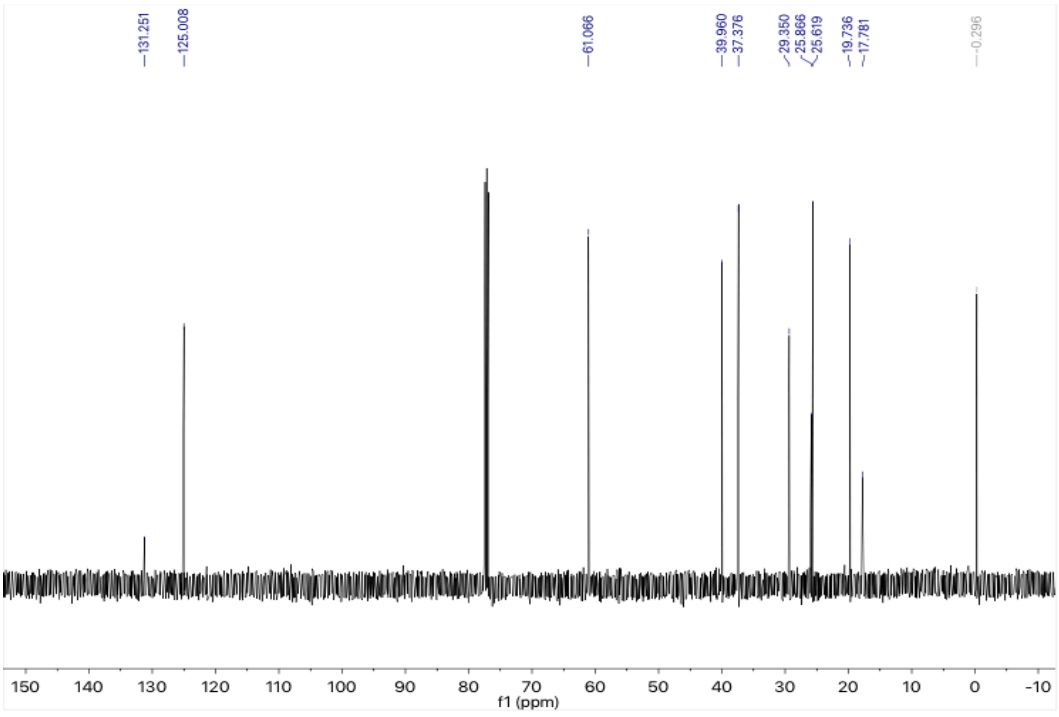
3.593
3.588
3.580
3.575
3.573
1.994
1.978
1.964
1.947
1.681
1.678
1.601
1.598
1.587
1.583
1.572
1.569
1.560
1.557
1.549
1.539
1.536
1.526
1.523
1.365
1.361
1.353
1.349
1.342
1.338
1.334
1.329
1.327
1.324
1.322
1.318
1.315
1.311
1.305
1.302
1.158
1.154
1.151
1.139
0.888
0.875
0.123
0.118
0.115
0.108
0.101
0.092
O
Si
1.05
1.98
1.98
2.97
3.09
2.07
2.04
1.06
3.10
9.00
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0.0
-0.4
f1 (ppm)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140
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120
110
100
90
80
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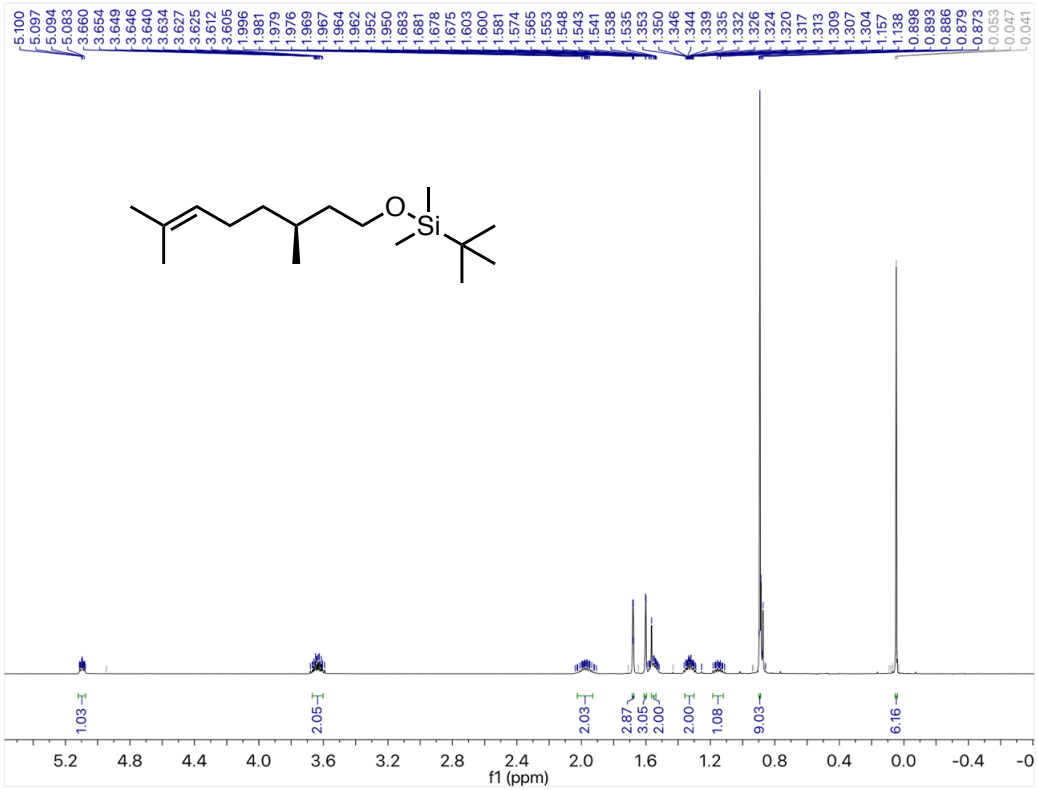
60
50
40
30
20
10
0
-10
f1 (ppm)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3.646
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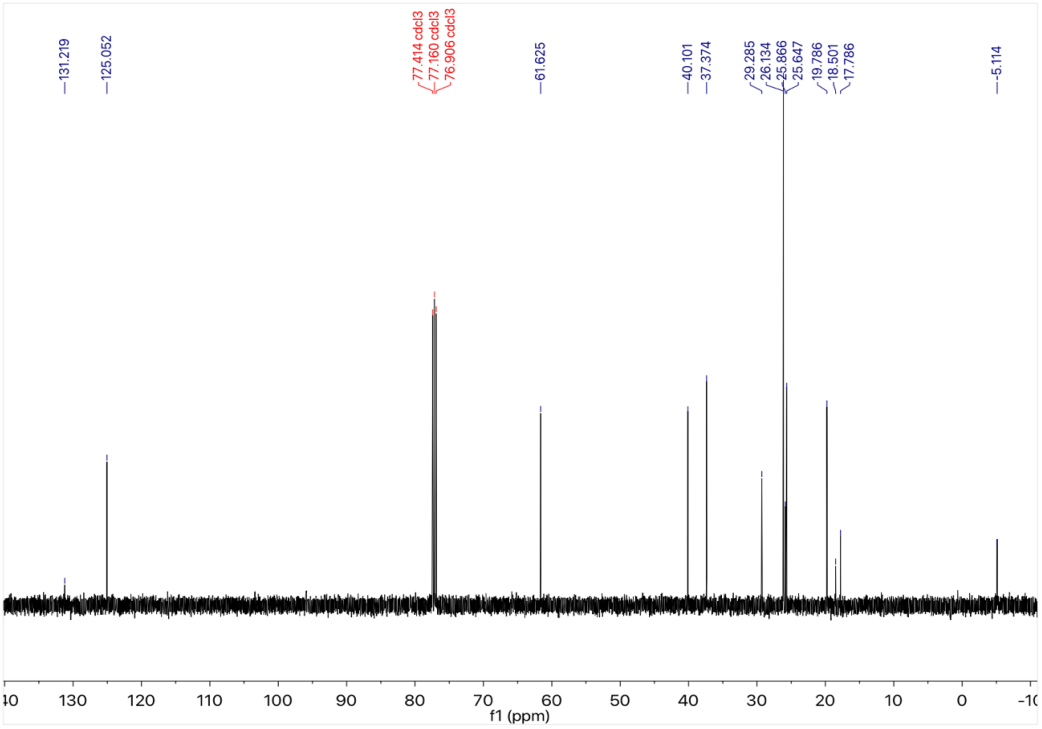
3.634
3.627
3.625
3.612
3.605
1.996
1.981
1.979
1.976
1.969
1.967
1.964
1.962
1.952
1.950
1.683
1.681
1.678
1.675
1.603
1.600
1.581
1.574
1.565
1.553
1.548
1.543
1.541
1.538
1.535
1.353
1.350
1.346
1.344
1.339
1.335
1.332
1.326
1.324
1.320
1.317
1.313
1.309
1.307
1.304
1.157
1.138
0.898
0.893
0.886
0.879
0.873
0.053
0.047
0.041
O
Si
1.03
2.05
2.03
2.87
3.05
2.00
2.00
1.08
9.03
6.16
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0.0
-0.4
f1 (ppm)


131.219
125.052
77.414 cdcl3
77.160 cdcl3
76.906 cdcl3
61.625
40.101
37.374
29.285
26.134
25.866
25.647
19.786
18.501
17.786
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130
120
110
100
90
80
70
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50
40
30
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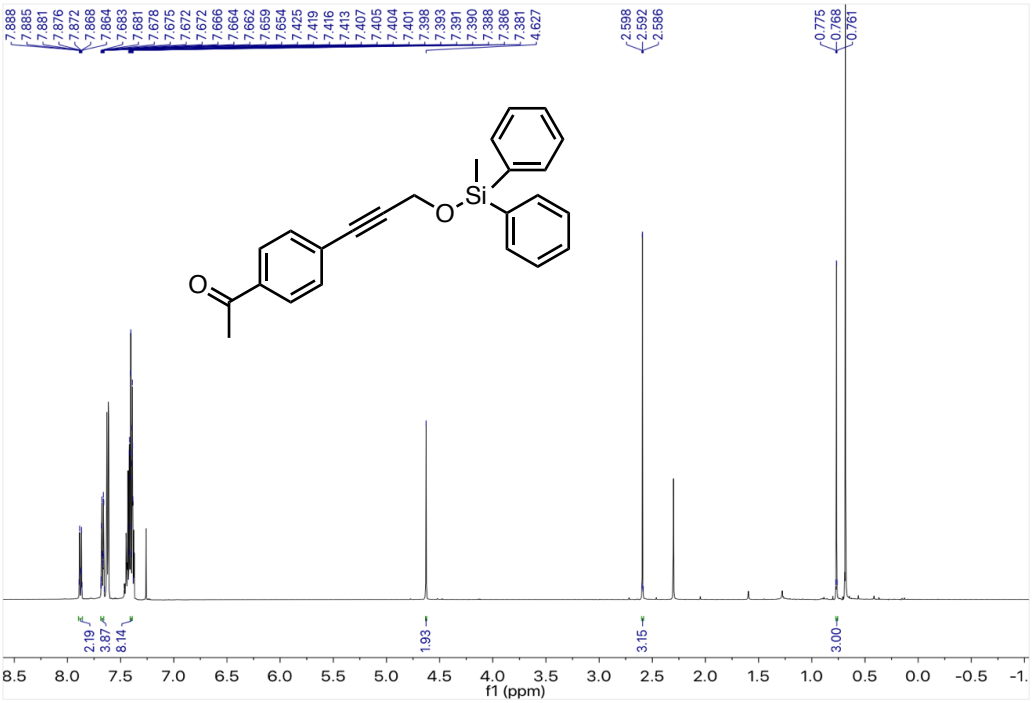
10
0
f1 (ppm)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7.678
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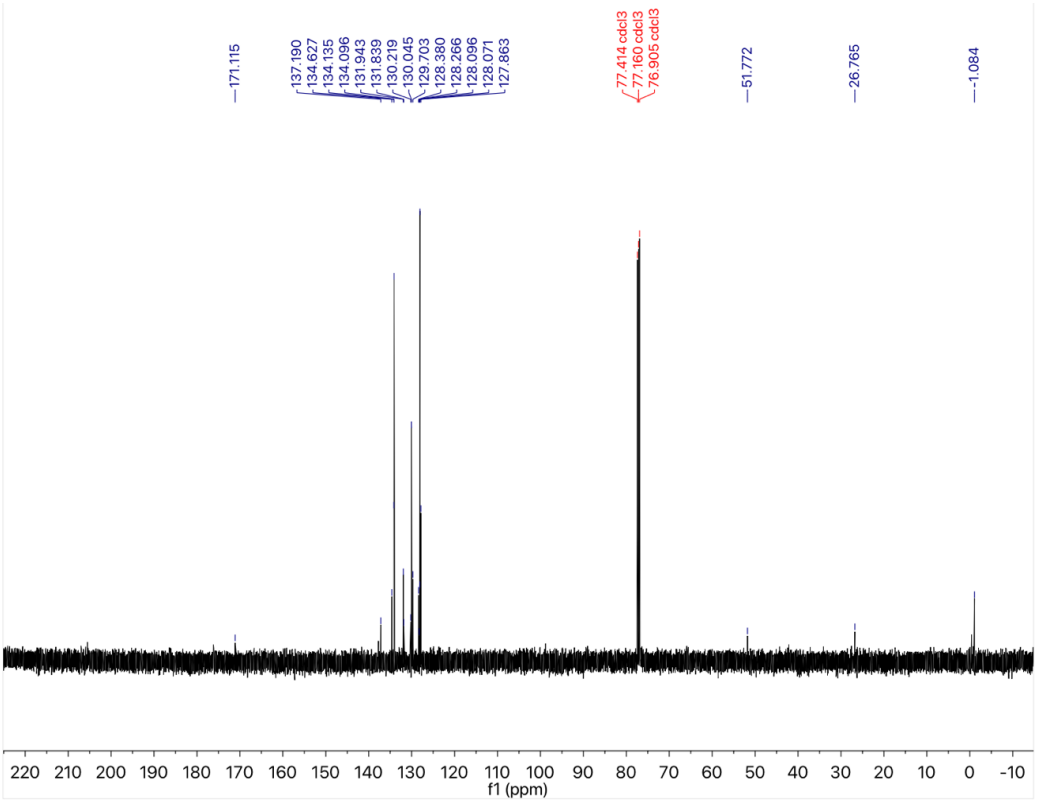
7.672
7.666
7.664
7.662
7.659
7.654
7.425
7.419
7.416
7.413
7.407
7.405
7.404
7.401
7.398
7.393
7.391
7.390
7.388
7.386
7.381
4.627
2.598
2.592
2.586
0.775
0.768
0.761
O
Si
O
2.19
3.87
8.14
1.93
3.15
3.00
f1 (ppm)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131.943
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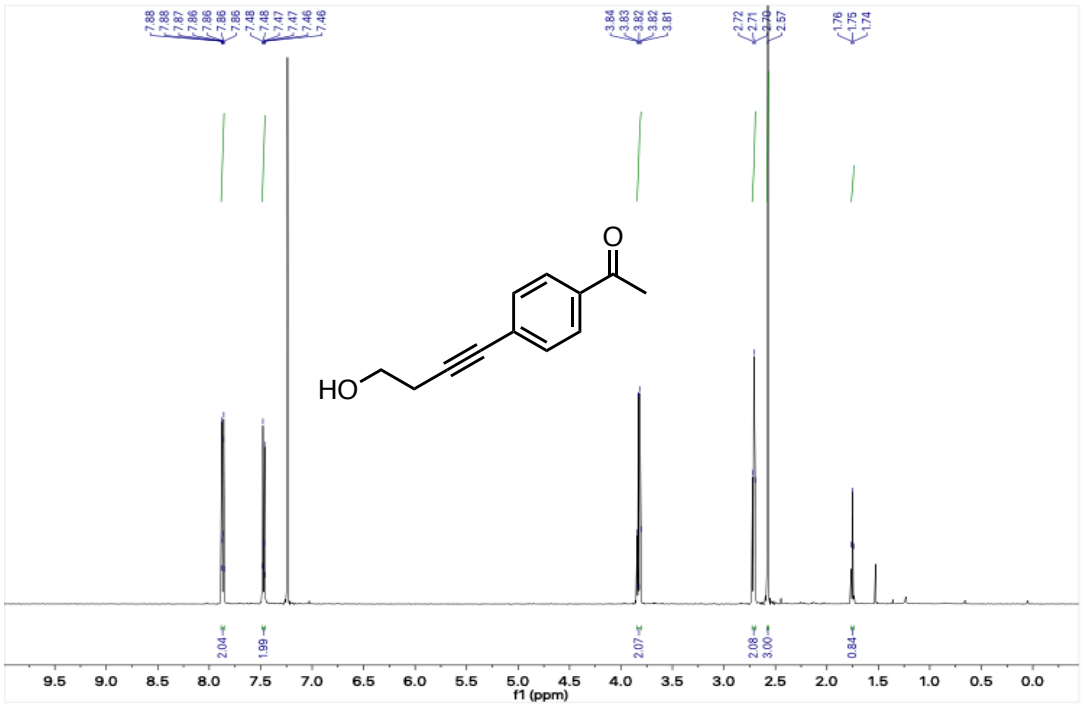
130.045
129.703
128.380
128.266
128.096
128.071
127.863
77.414 cdcl3
77.160 cdcl3
76.905 cdcl3
51.772
26.765
-1.084
f1 (ppm)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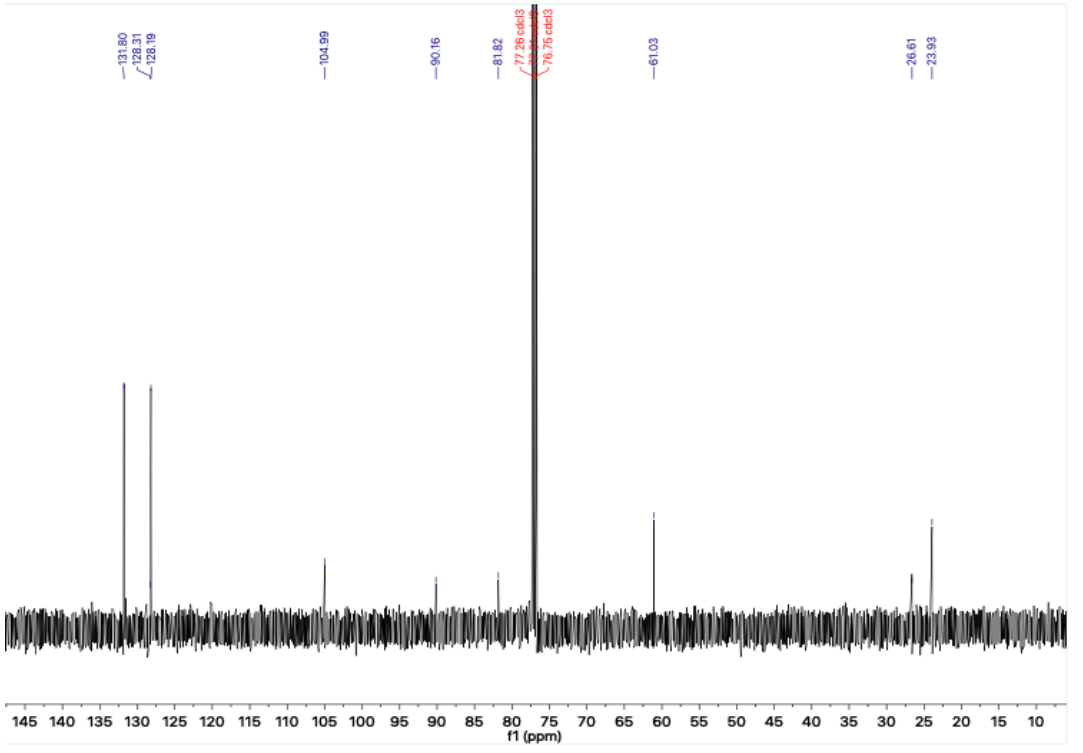
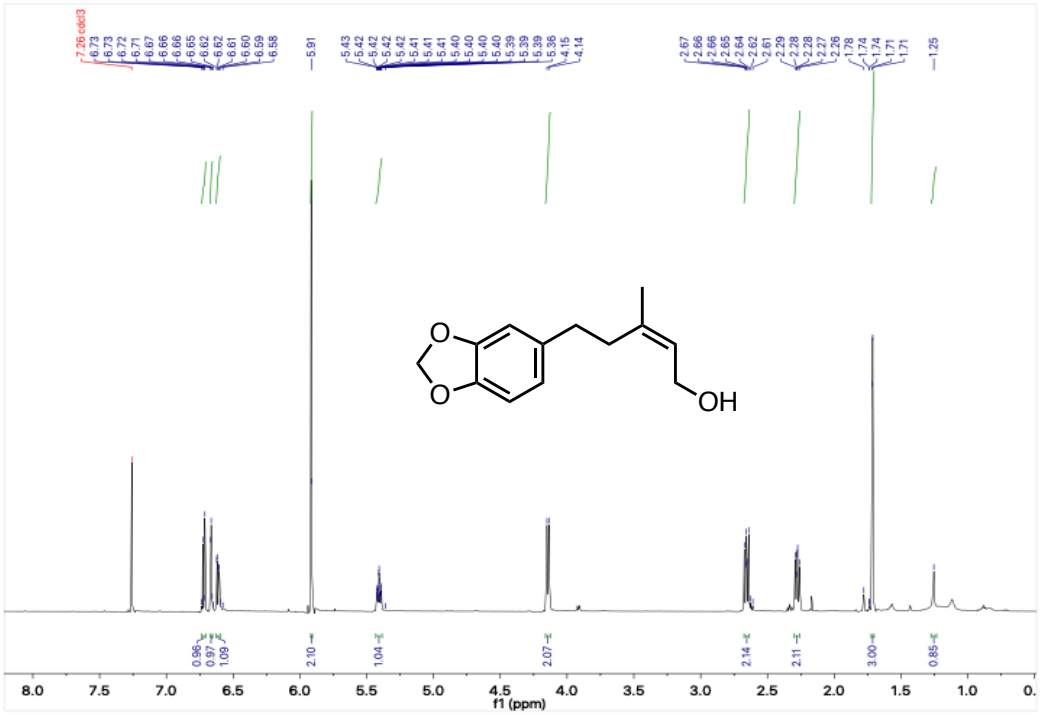
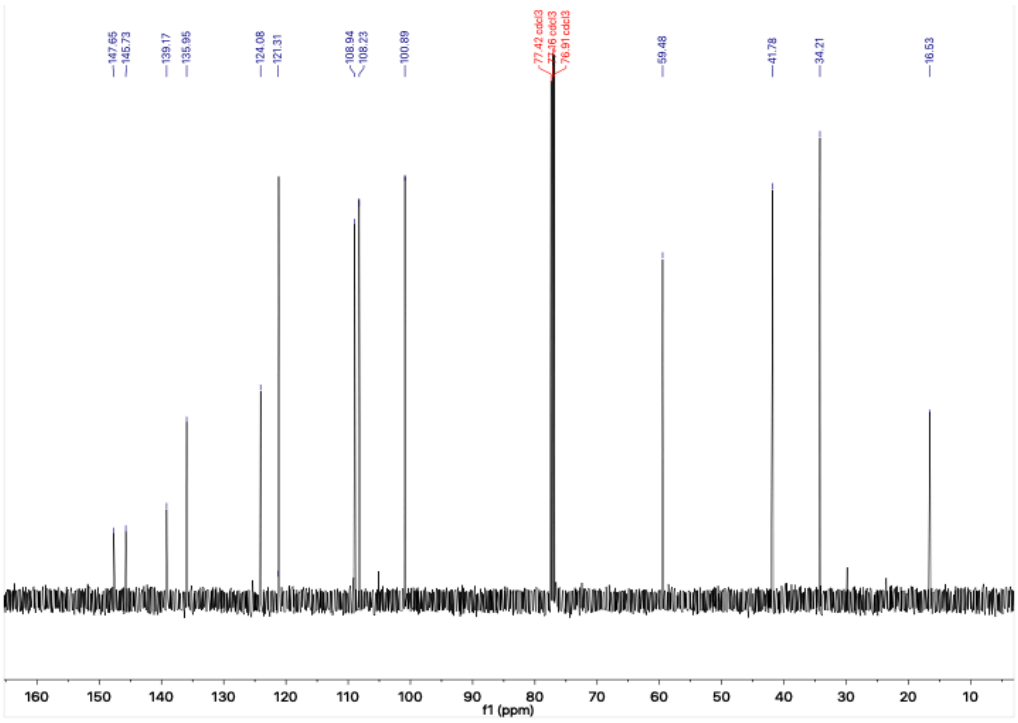
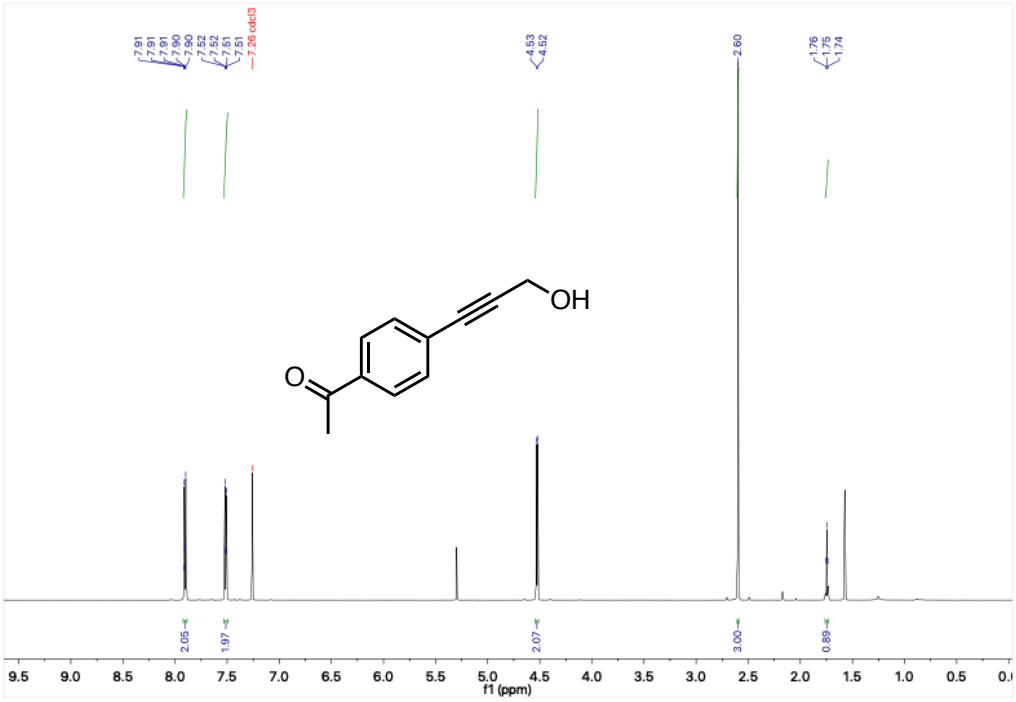
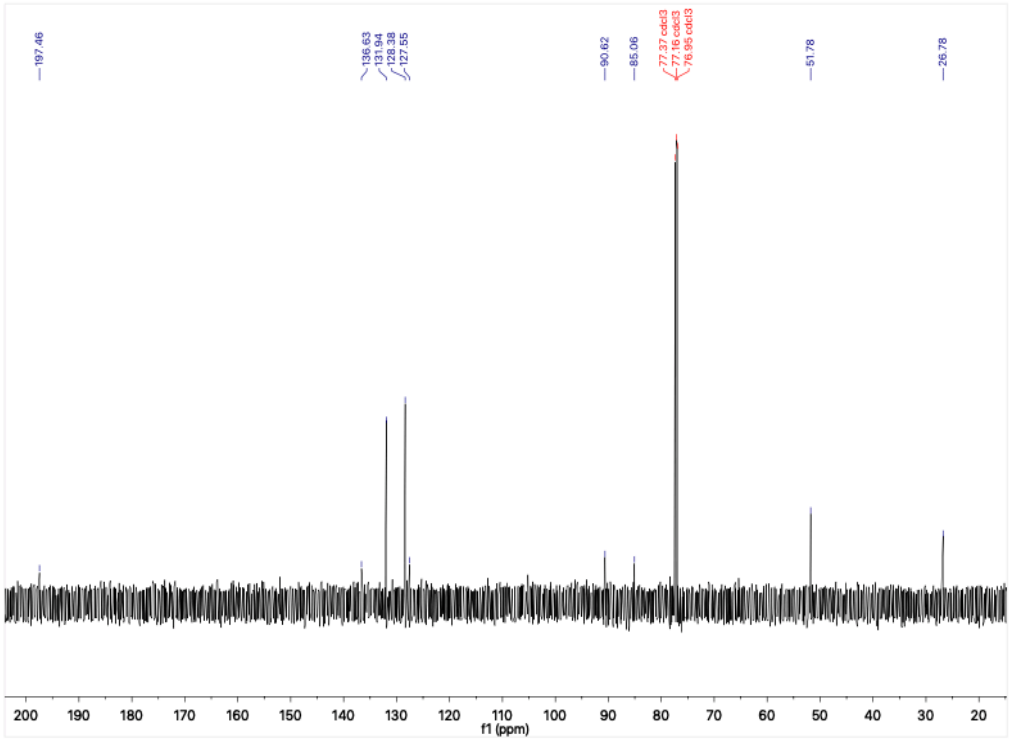
7.88
7.87
7.86
7.86
7.86
7.86
7.48
7.48
7.47
7.47
7.46
7.46
3.84
3.83
3.82
3.82
3.81
2.72
2.71
2.70
2.57
1.76
1.75
1.74
O
HO
2.04
1.99
2.07
2.08
3.00
0.84
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

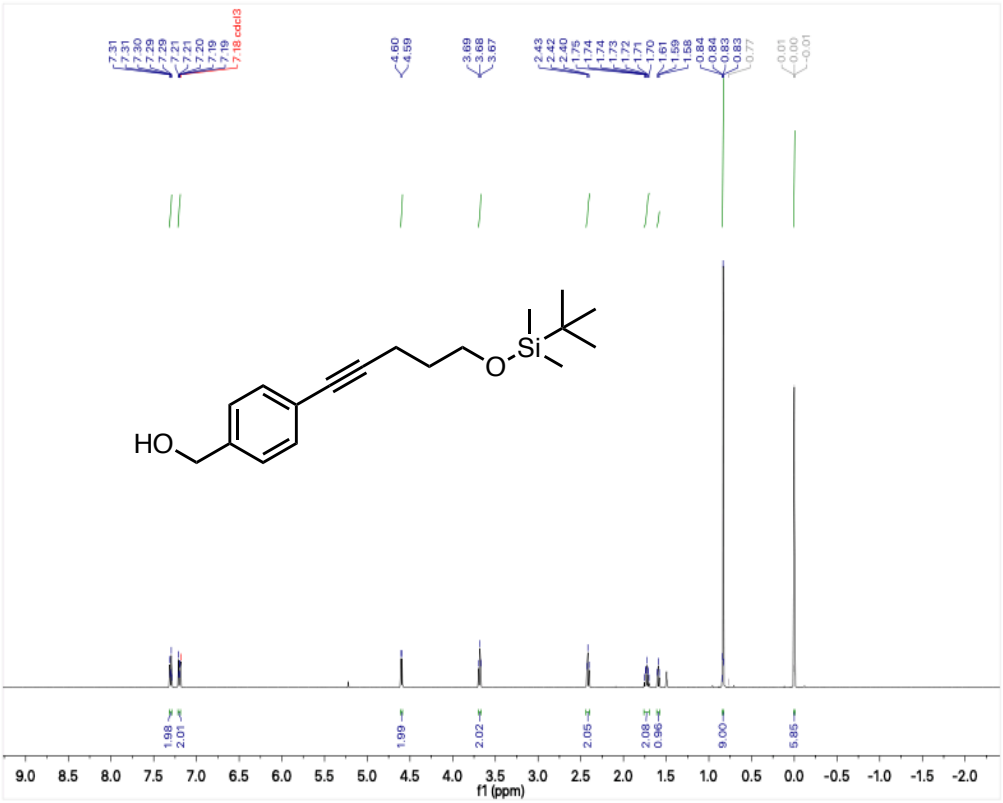

131.80
129.31
128.19
104.99
90.16
81.82
77.26 cdcl3
76.75 cdcl3
61.03
26.61
23.93
145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
f1 (ppm)


7.26 cdcl3
6.73
6.73
6.72
6.71
6.67
6.66
6.66
6.65
6.62
6.62
6.61
6.60
6.59
6.58
5.91
5.43
5.42
5.42
5.42
5.42
5.41
5.41
5.41
5.40
5.40
5.40
5.40
5.39
5.39
5.39
5.36
4.15
4.14
2.67
2.66
2.66
2.65
2.64
2.62
2.61
2.29
2.28
2.28
2.27
2.26
1.78
1.74
1.74
1.71
1.71
1.25
O
O
OH
0.96
0.97
1.09
2.10
1.04
2.07
2.14
2.11
3.00
0.85
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)


147.65
145.73
139.17
135.95
124.08
121.31
108.94
108.23
100.89
77.42 cdcl3
77.16 cdcl3
76.91 cdcl3
59.48
41.78
34.21
16.53
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.91
7.91
7.91
7.90
7.90
7.52
7.52
7.51
7.51
7.26 cdcl3
4.53
4.52
2.60
1.76
1.75
1.74
OH
O
2.05
1.97
2.07
3.00
0.89
f1 (ppm)


197.46
136.63
131.94
128.38
127.55
90.62
85.06
77.37 cdcl3
77.16 cdcl3
76.95 cdcl3
51.78
26.78
f1 (ppm)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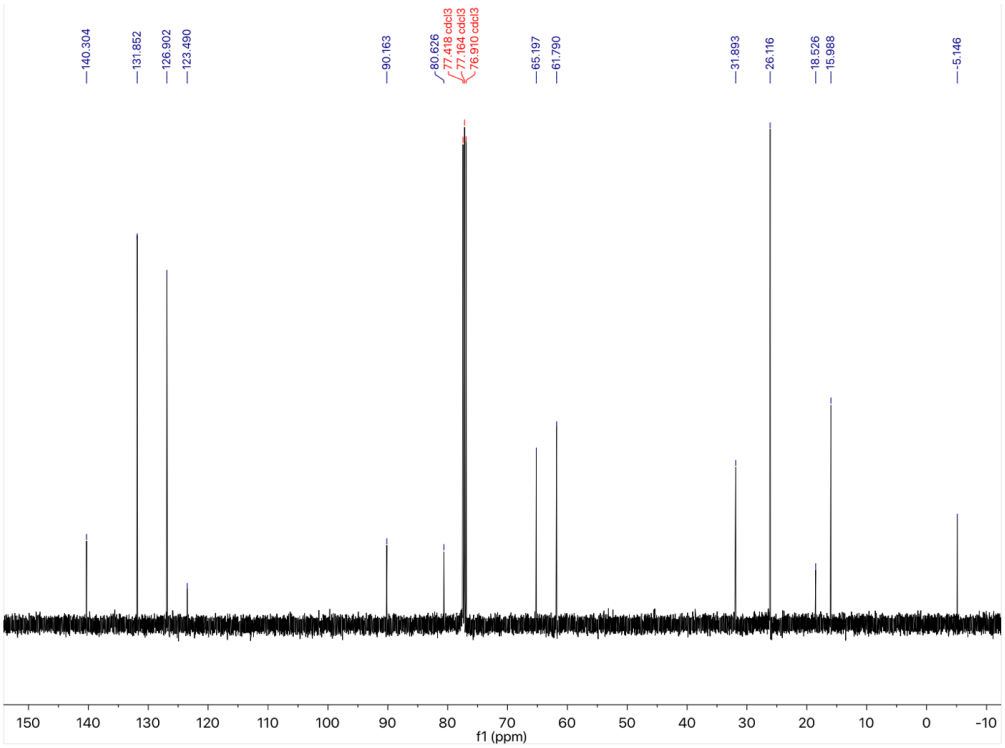
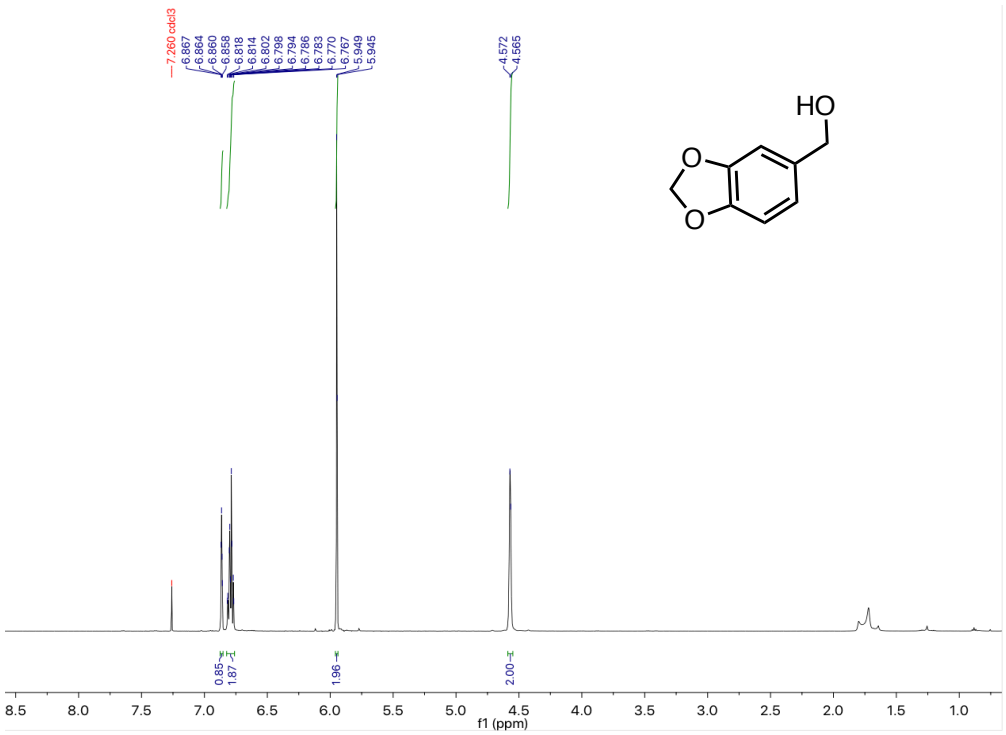
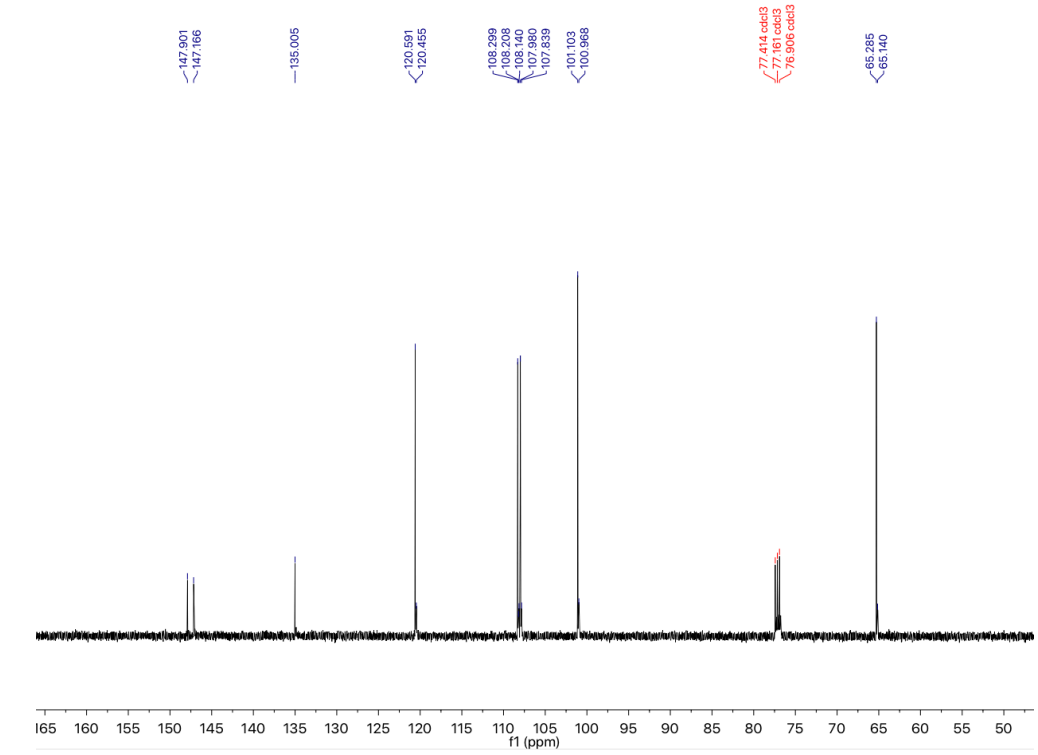
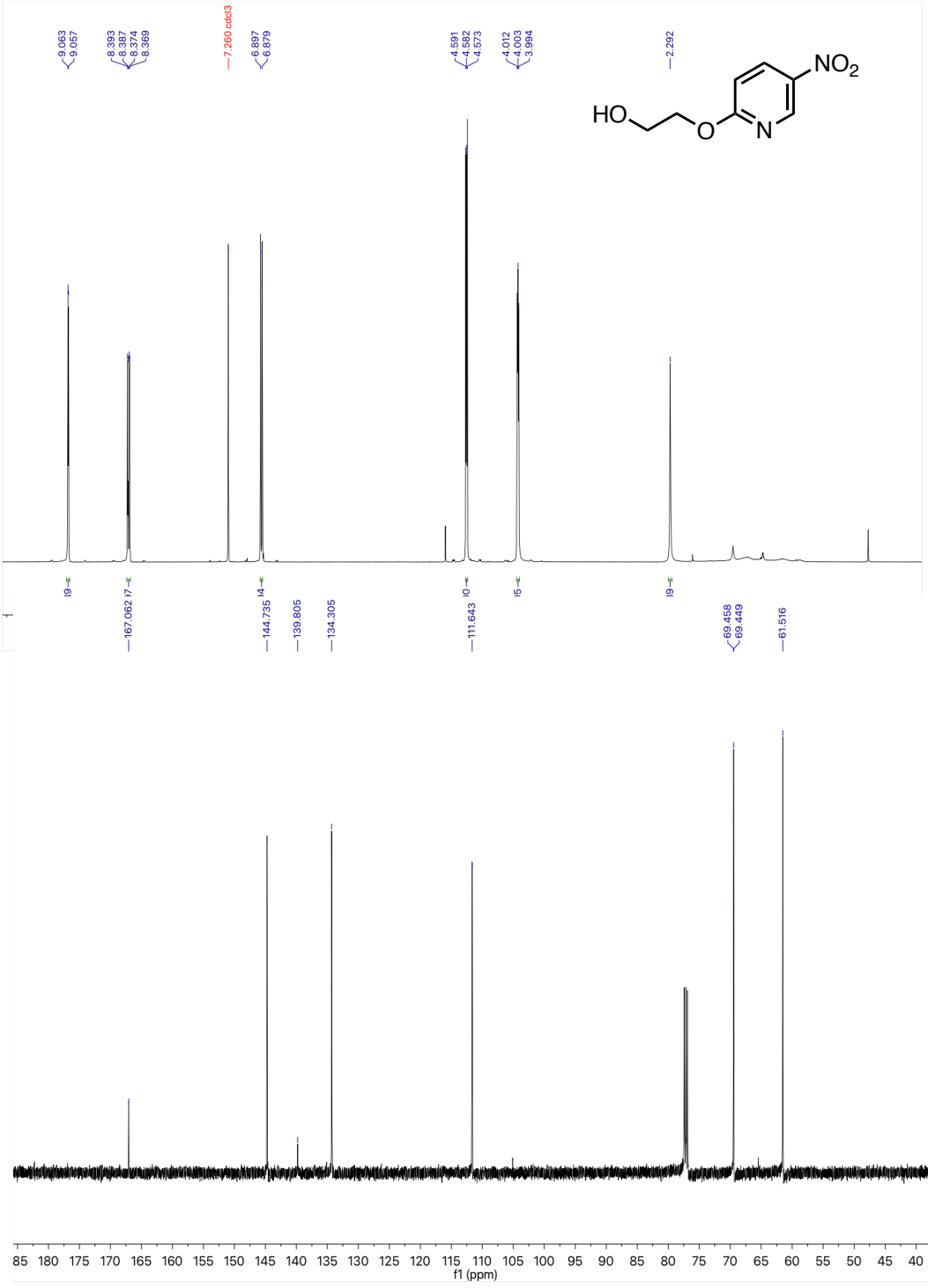
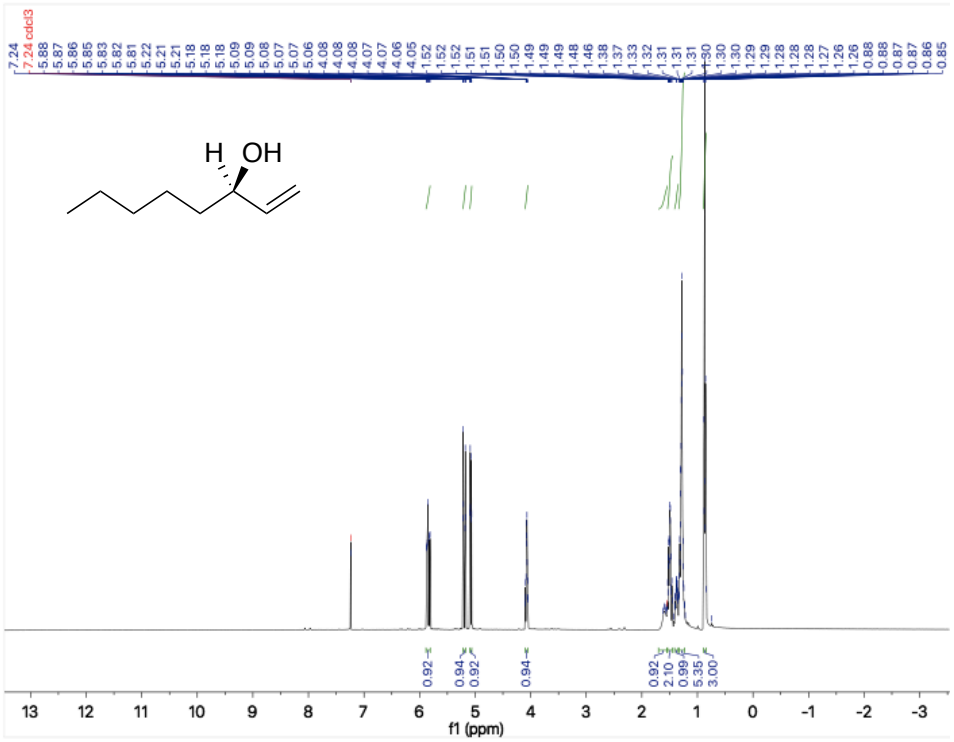
7.31
7.30
7.29
7.29
7.21
7.21
7.20
7.19
7.19
7.18 cdcl3
4.60
4.59
3.69
3.68
3.67
2.43
2.42
2.40
1.75
1.74
1.74
1.73
1.72
1.71
1.70
1.61
1.59
1.58
0.84
0.84
0.83
0.83
0.77
0.01
0.00
-0.01
HO
Si
O
1.98
2.01
1.99
2.02
2.05
2.08
0.96
9.00
5.85
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)

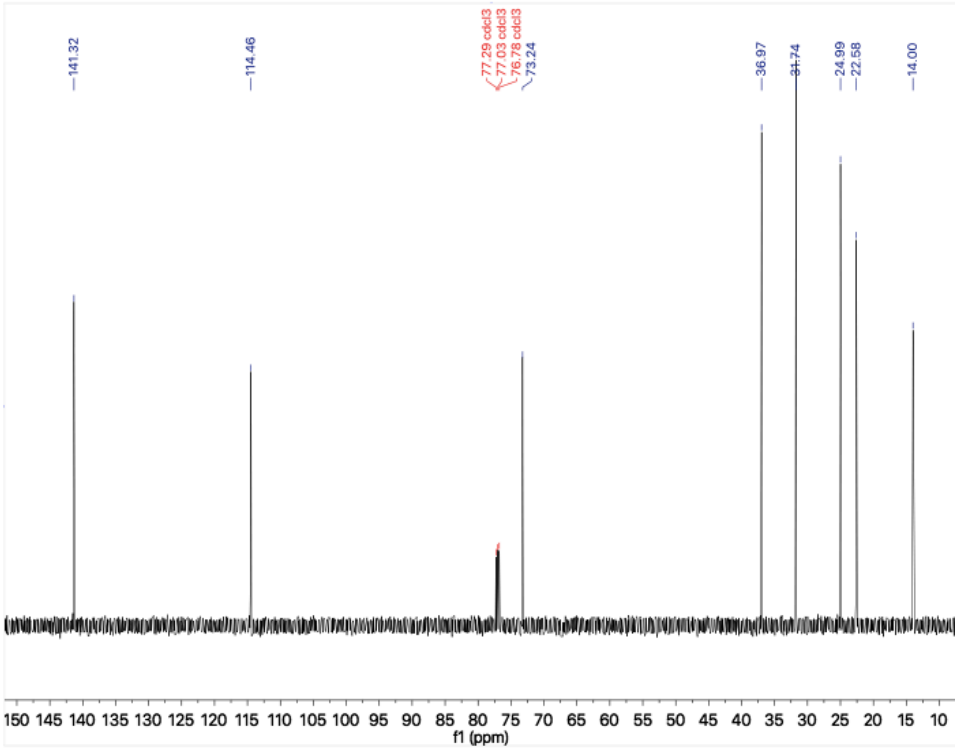

140.304
131.852
126.902
123.490
90.163
80.626
77.418 cdcl3
77.164 cdcl3
76.910 cdcl3
65.197
61.790
31.893
26.116
18.526
15.988
-5.146
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

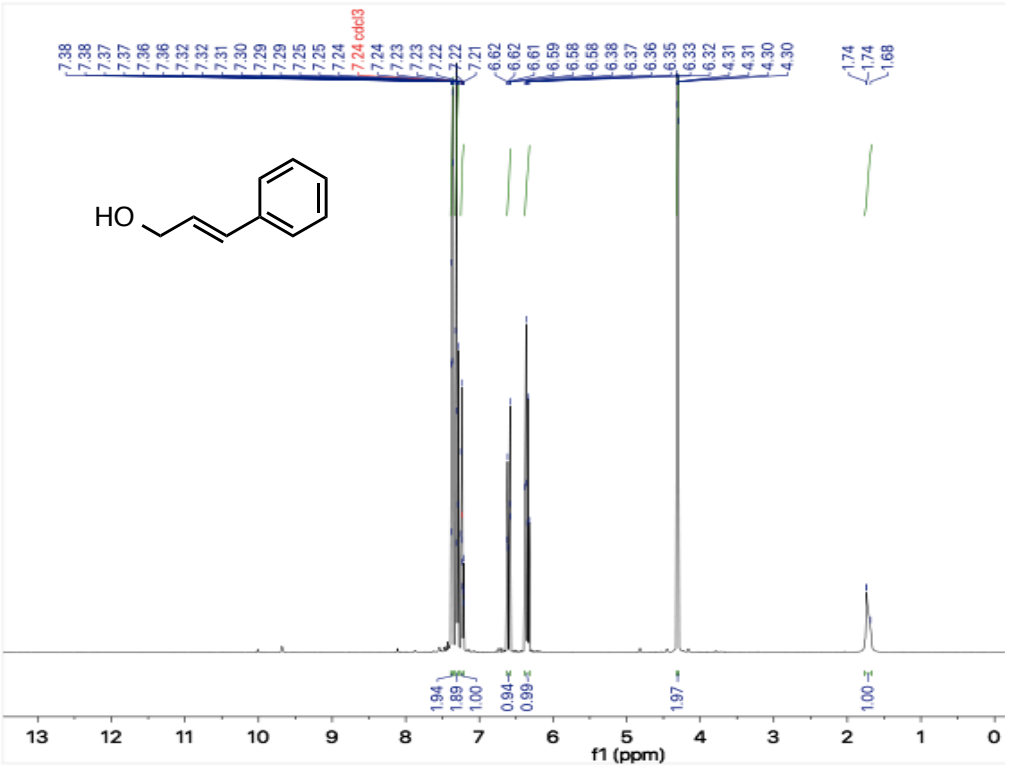

7.260 cdcl3
6.867
6.864
6.860
6.858
6.818
6.814
6.802
6.798
6.794
6.786
6.783
6.770
6.767
5.949
5.945
4.572
4.565
HO
O
O
0.85
1.87
1.96
2.00
f1 (ppm)

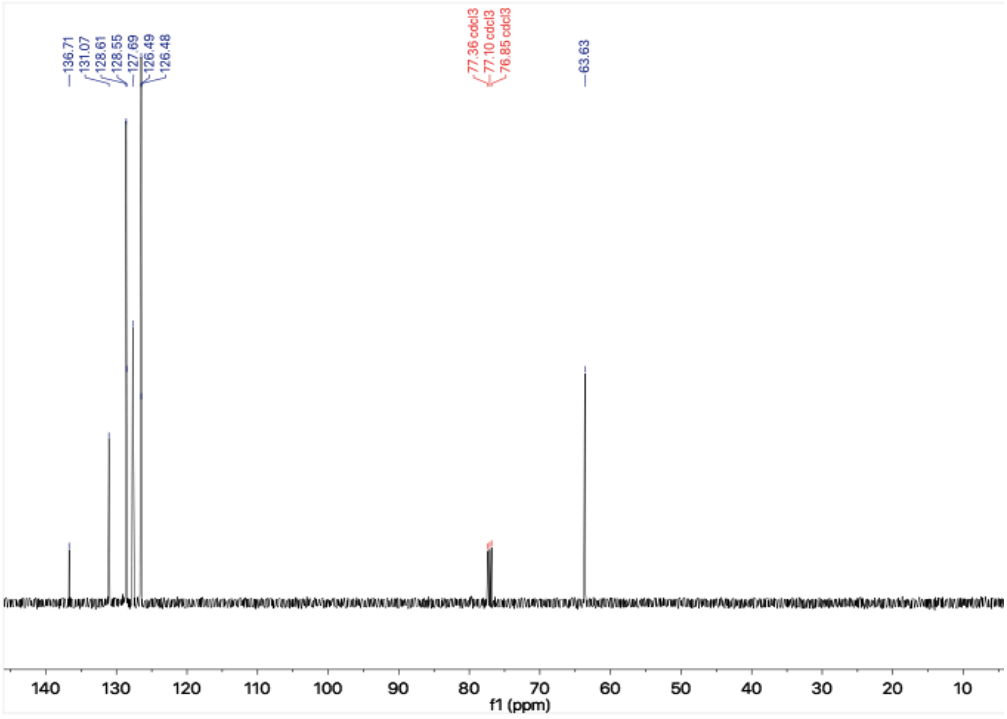

147.901
147.166
135.005
120.591
120.455
108.299
108.208
108.140
107.980
107.839
101.103
100.968
77.414 cdcl3
77.161 cdcl3
76.906 cdcl3
65.285
65.140
f1 (ppm)

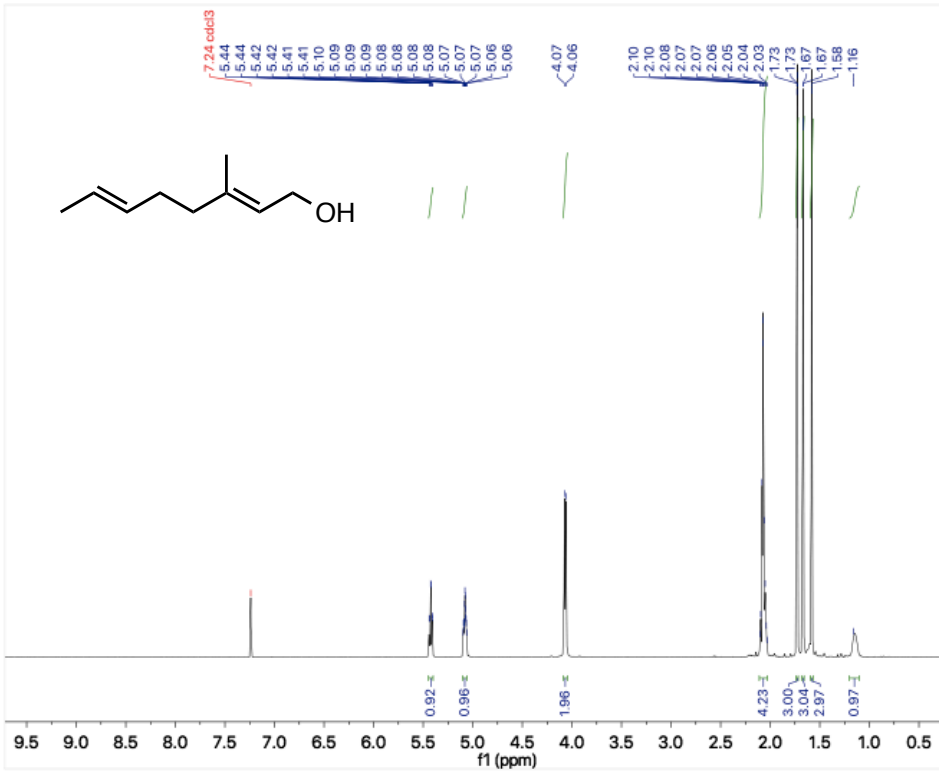

9.063
9.057
8.393
8.387
8.377
8.374
8.369
7.260 cdcl3
6.897
6.879
4.591
4.582
4.573
4.012
4.003
3.994
2.292
HO
O
N
NO2
1.9
2.17
1.4
1.0
1.5
1.9
167.062
144.735
139.805
134.305
111.643
69.458
69.449
61.516
85 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40
f1 (ppm)

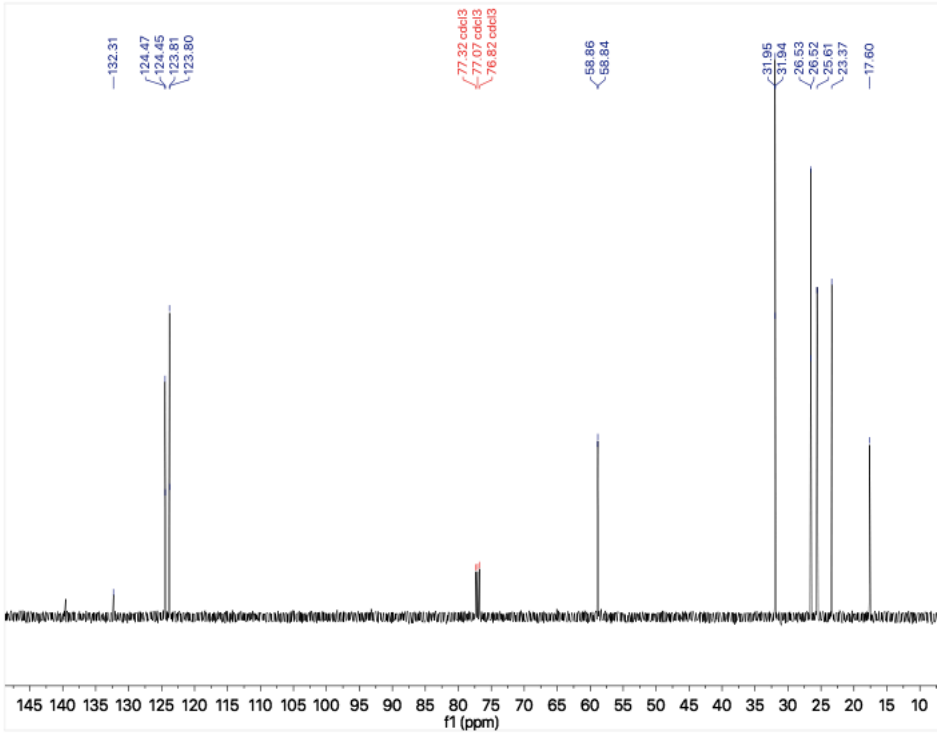

H
OH
f1 (ppm)

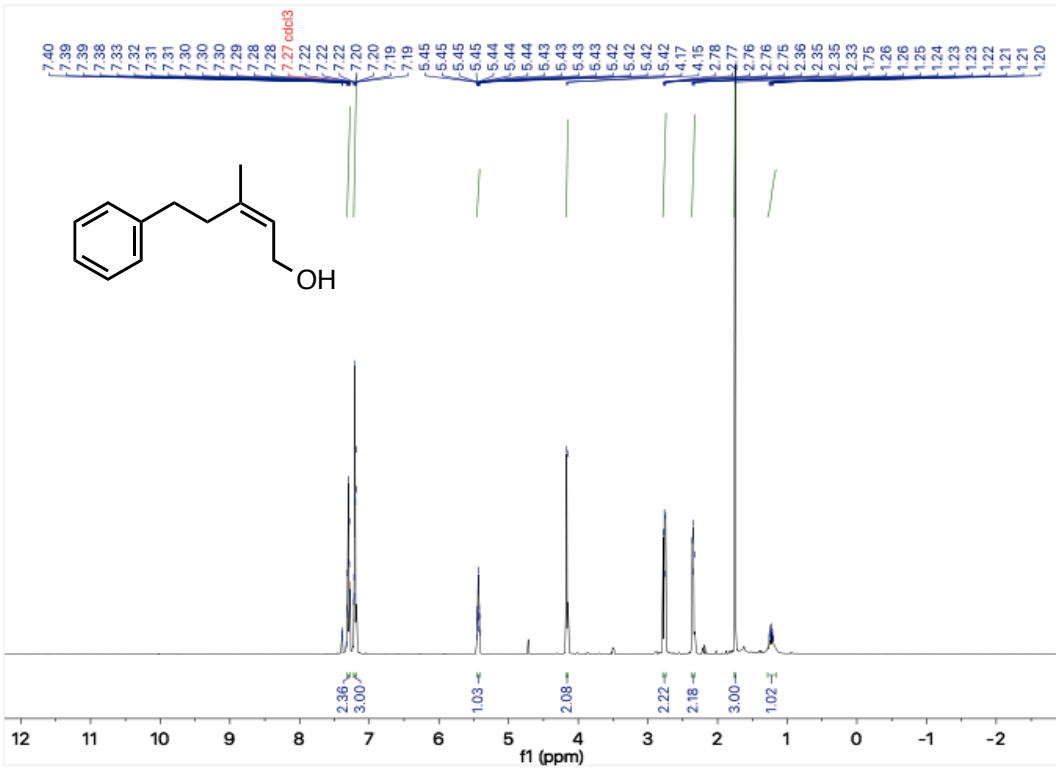

141.32
114.46
77.29 cdcl3
77.03 cdcl3
76.78 cdcl3
73.24
36.97
31.74
24.99
22.58
14.00
f1 (ppm)

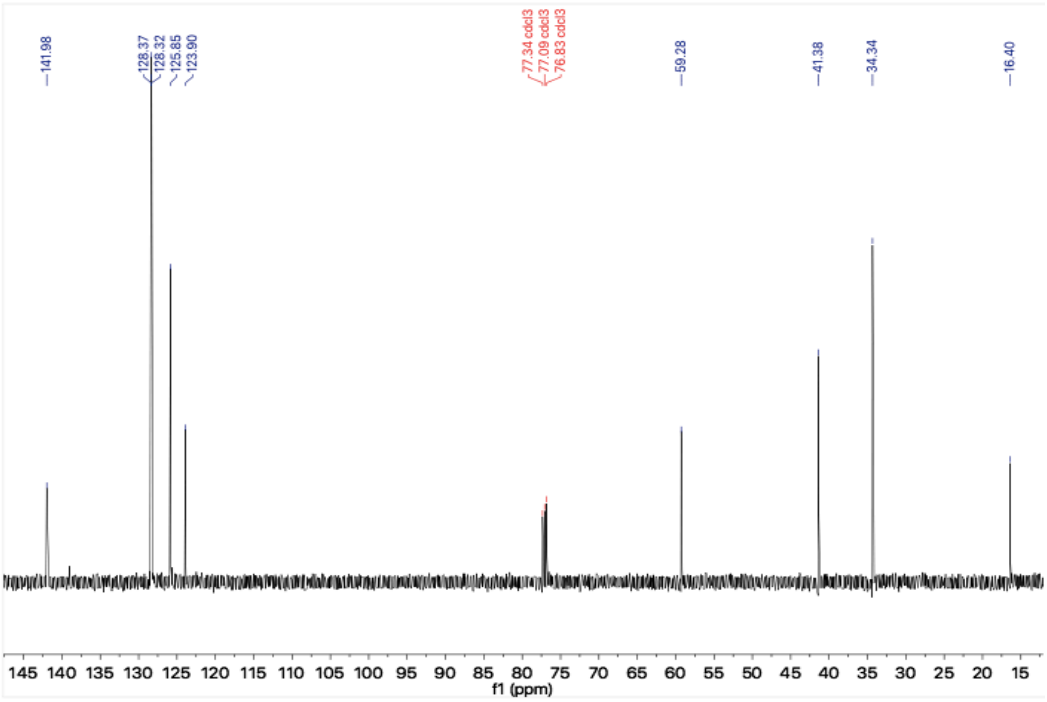

HO
7.38
7.38
7.37
7.37
7.36
7.36
7.32
7.32
7.31
7.30
7.29
7.29
7.25
7.25
7.24
7.24 cdcl3
7.24
7.23
7.23
7.22
7.22
7.21
6.62
6.62
6.61
6.59
6.58
6.58
6.38
6.37
6.36
6.35
6.33
6.32
4.31
4.31
4.30
4.30
1.74
1.74
1.68
1.94
1.89
1.00
0.94
0.99
1.97
1.00
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

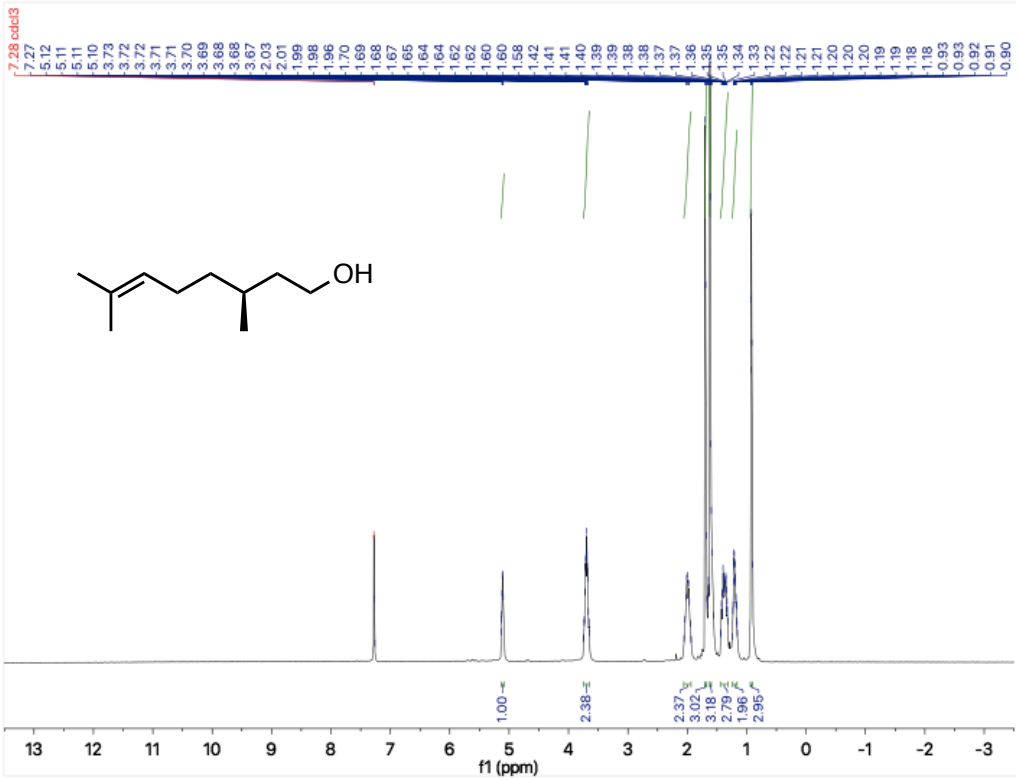

136.71
131.07
128.61
128.55
127.69
126.49
126.48
77.36 cdcl3
77.10 cdcl3
76.85 cdcl3
63.63
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

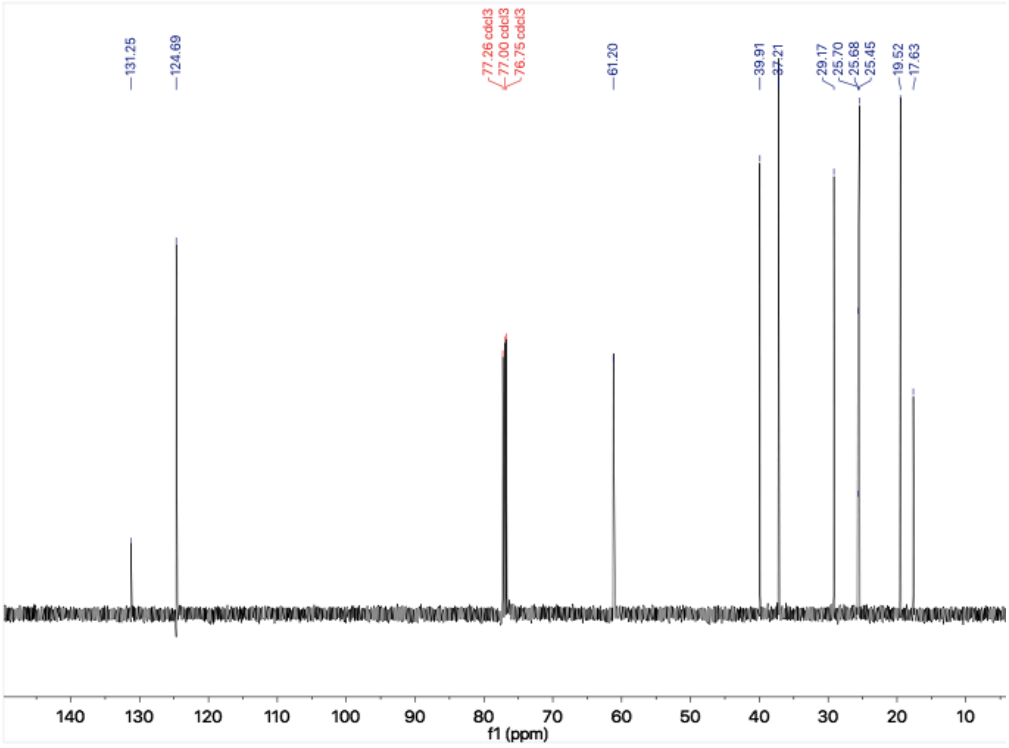

OH
f1 (ppm)

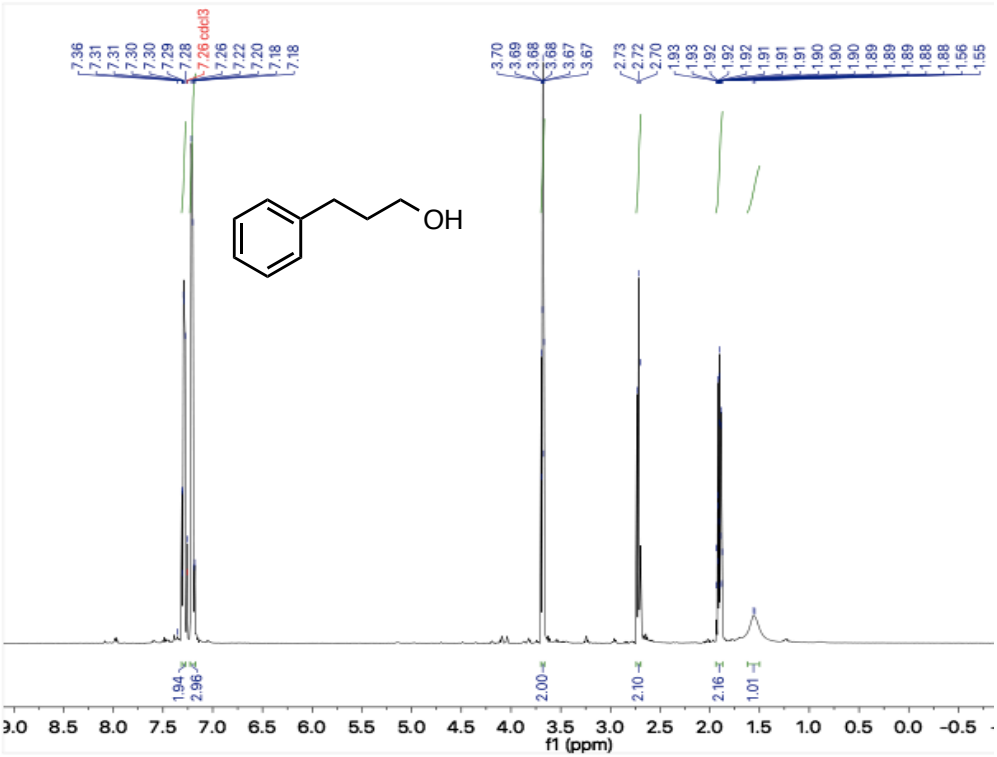

f1 (ppm)

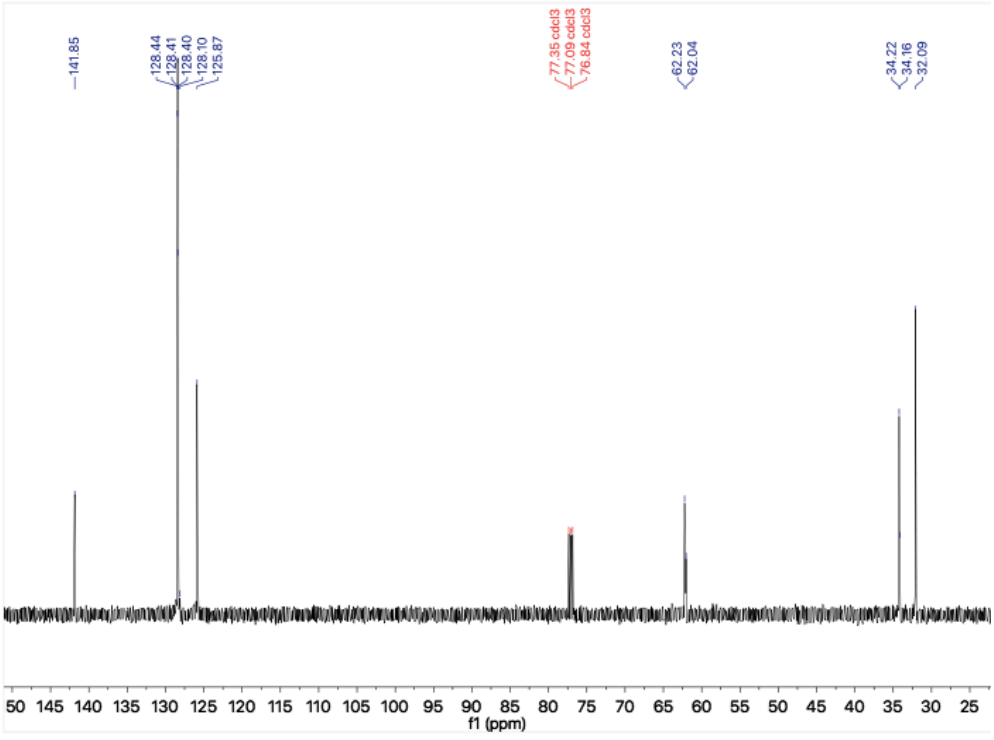

OH
f1 (ppm)

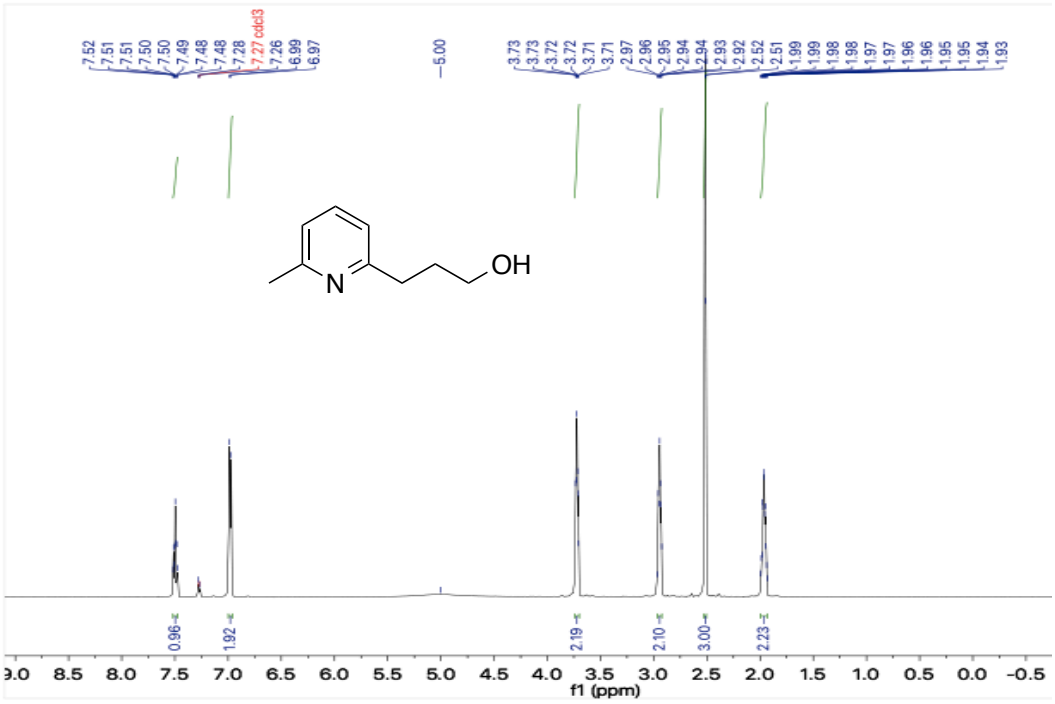

f1 (ppm)


OH
f1 (ppm)


f1 (ppm)


OH
f1 (ppm)


f1 (ppm)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7.50
7.49
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7.48
7.28
7.27 cdcl3
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6.97
5.00
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3.73
3.72
3.72
3.71
3.71
2.97
2.96
2.96
2.95
2.94
2.94
2.93
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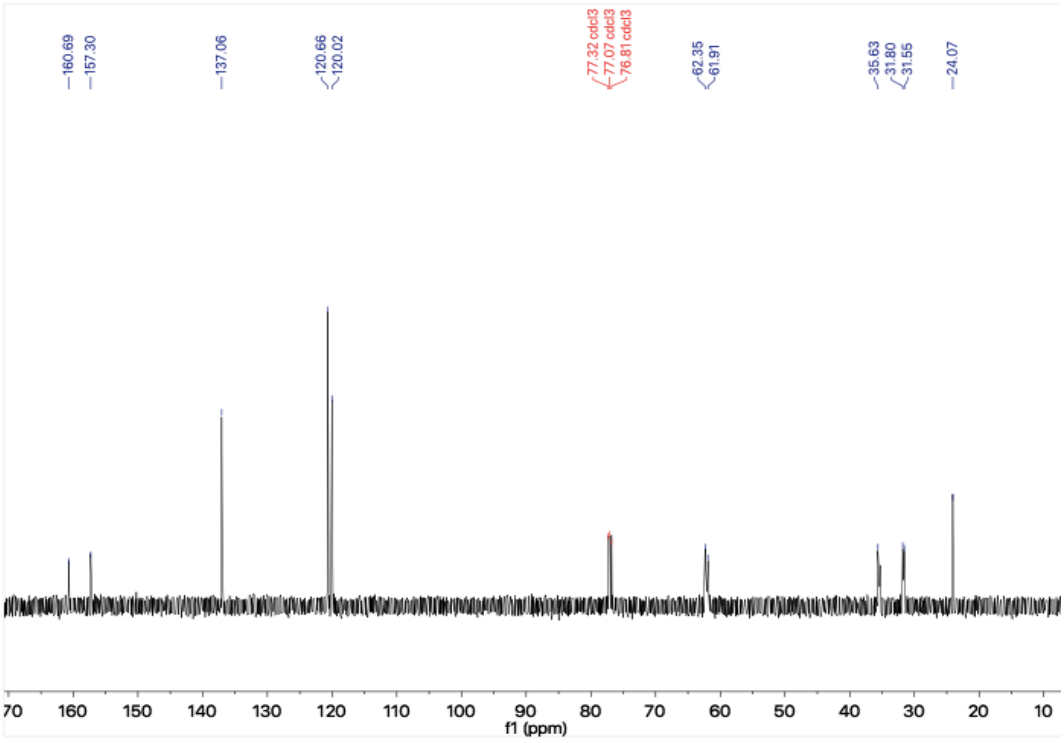
2.51
1.99
1.99
1.98
1.98
1.97
1.97
1.96
1.96
1.95
1.95
1.94
1.93
OH
N
0.96
1.92
2.19
2.10
3.00
2.23
f1 (ppm)


160.69
157.30
137.06
120.66
120.02
77.32 cdcl3
77.07 cdcl3
76.81 cdcl3
62.35
61.91
35.63
31.80
31.55
24.07
f1 (ppm)