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A concise and efficient synthetic strategy to silyl-protected terminal alkynyl sulfides from alkyl or benzyl halides

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ABSTRACT

A novel synthetic strategy to silyl-protected terminal alkynyl sulfides via reaction of lithium 2,2,2-tris(trimethylsilyl)ethanedithioate, produced by the reaction of tris(trimethylsilyl)methyllithium with carbon disulfide, and alkyl or benzyl halides has been developed. The scope of the reaction is broad, with a variety of benzylic and aliphatic halides and products were formed in good to excellent yields.

Introduction

Alkynyl sulfides are key structural intermediates in organic synthesis and can be used as versatile building blocks for a variety of chemical purposes, such as cycloaddition and cross-coupling reactions [1–3]. These compounds are a desirable platform for drug diversification considering the variety of existing acetylene chemistry, the extensively used copper-catalyzed azid-alkyn cycloaddition, as well as the powerful position of organosulfur compounds among top-selling pharmaceutical drugs [4].

To our knowledge, few studies [5–9] are focused on the formation of S-Csp bonds and existing methods suffer from strict conditions and limited scopes on the basis of the promising potentialities of alkynyl sulfides, it would be desirable to develop a general method for the synthesis of these compounds.

Due to the significance of thiazolidine-2-thiones as important building blocks in the synthesis of natural products and pharmaceutical and medicinal compounds [10–14], our previous work [15] mainly covered the development of our achievements to a range of functionalized thiazolidine-2-thiones (Scheme 1).

To extend our investigations to alkynyl sulfide compounds, we decided to examine the generality of this reaction with several alkyl and benzyl halides.

Results and discussion

We have recently used bulky tris(trimethylsilyl)methyllithium as a reagent in various reactions such as the preparation of vinylsilanes, epoxy silanes, halovinylsilanes, silyl ethers, etc [16–19]. In addition we have investigated its behavior with carbon disulfide at low temperature [20,21]. In the previous publications we reported the new type of chemistry of carbon disulfide proceeding via nucleophilic attack of tris(trimethylsilyl)methyllithium at carbon of carbon disulfide at −46 °C. In continuous to extend our investigations to organosilicon compounds, we decided to study the reaction of tris(trimethylsilyl)methyllithium with carbon disulfide in the presence of various alkyl or benzyl halides at 0 °C. Silyl-protected alkynyl sulfides procedure utilizing the tris(trimethylsilyl)methyllithium and carbon disulfide as substrates has been developed. This scale reaction proceeds in 10–20 min at 0 °C under dry argon. The scope of the reaction is broad, with a variety of aliphatic, benzylic halides in good to excellent yields.

We began our study with tris(trimethylsilyl)methyllithium as an organolithium compound and benzyl bromide as our model substrate. When carbon disulfide was added to a solution of tris(trimethylsilyl)methyllithium in THF at 0 °C, the color immediately changed from yellow to red, indicating the formation of new anion **A** (Scheme 3). Benzyl bromide was added and the color changed again (dark brown). The mixture was stirred for 10 min at 0 °C and ((benzylthio)ethynyl)trimethylsilane (**3a**) was formed as the sole product in 85% yield (Scheme 2). Its structure was distinguished from FT IR, ^{13}C and ^{1}H NMR spectra. In the FT IR spectrum of this

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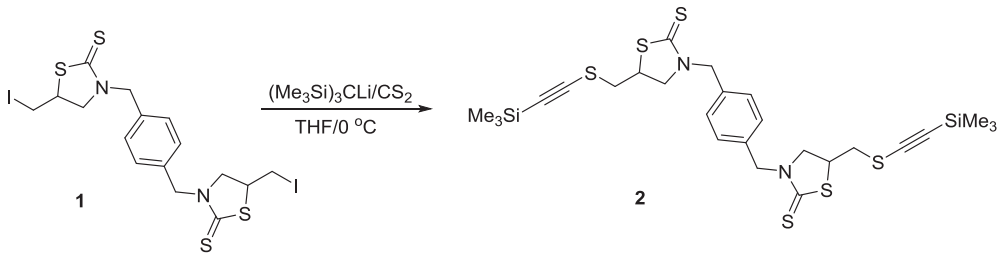
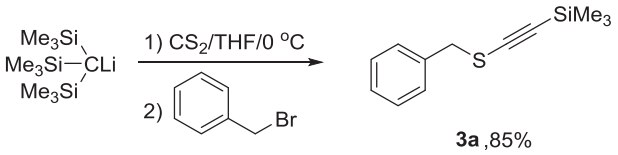


Scheme 1. Reaction of tris(trimethylsilyl)methyllithium and carbon disulfide with thiazolidine-2-thiones.



Scheme 2. Preparation of ((benzylthio)ethynyl)trimethylsilane.

compound a peak at 2092 cm^{-1} indicated the presence of C≡C group. ^{1}H NMR of the compound showed a singlet at 0.08 ppm assigned to $-SiMe_3$ protons. In addition, in the ^{13}C NMR spectrum, two peaks at 93.1 and 101.2 ppm confirmed the presence of the C≡C group.

The precursor tris(trimethylsilyl)methane was prepared by the reaction of chloroform, lithium, and chlorotrimethylsilane in tetrahydrofuran. The organolithium reagent tris(trimethylsilyl) methyllithium was obtained by treatment of tris(trimethylsilyl) methane with methyllithium at reflux in tetrahydrofuran [22].

The reaction of various alkyl or benzyl halides was studied and the results are summarized in Table 1. All the experiments were carried out at 0 °C. All the products are stable for a long time at room temperature.

Generally, alkyl and benzyl iodides reacted better and gave higher yields than alkyl and benzyl bromide or chlorides. Furthermore, reaction of tris(trimethylsilyl)methyllithium and carbon disulfide with benzyl halides proceeded rapidly than its alkyl analogs.

On the basis of the experimental results and the literature [13,23–25], a possible mechanism for the formation of silyl-protected terminal alkynyl sulfides can be proposed as shown in Scheme 3. According to the literature, the reaction of carbon disulfide with organolithium compounds lead to the formation of two different types of intermediates, 1,1-dithioenolates $RHC = C(SLi)_2$ and dithiocarboxylates RC(S)SLi. In the present work, addition of carbon disulfide to tris(trimethylsilyl) methyllithium at 0 °C, produced $(Me_3Si)_3C(S)SLi$ intermediate, which subsequently reacted with alkyl or benzyl halides to give $(Me_3Si)_3C(S)SCH_2R$. This type of reaction observed for all of compounds RCH_2X with X = Cl, Br, I. An E_2 elimination from **B** with attack of X^- at Me_3Si and lose of $XSiMe_3$ group, produced **C** intermediate. The carbanion intermediate **C** can be stabilized with lose of $LiSSiMe_3$ group and produced the final products **3**.

The developed method not only allows a highly efficient synthesis of alkynyl sulfides but also provides access to unique alkynes with the potential for further derivatization.

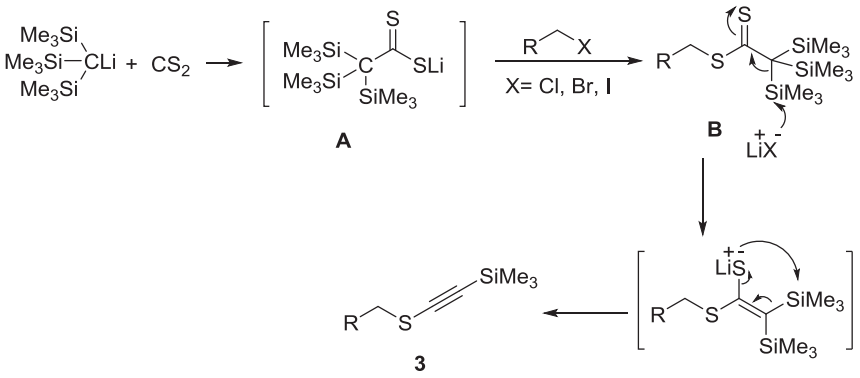
Conclusion

In conclusion, a novel synthetic strategy to silyl-protected terminal alkynyl sulfides from the reaction of the lithium 2,2,2-tris(trimethylsilyl)ethanedithioate intermediate with various alkyl or benzyl halides has been developed. The new method presented herein has the potential to achieve privileged position as a novel tool for various applications in synthetic chemistry, chemical biology, and material science. Ongoing research efforts are aimed at developing this method on various biologically important compounds.

Experimental

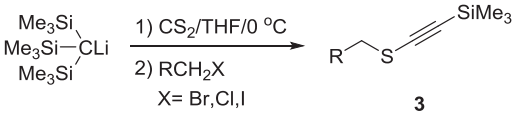
Solvents and reagents

The reactions were carried out under dry argon. Solvents and carbon disulfide were dried by standard methods. Substrates for the preparation of tris(trimethylsilyl)methyllithium, viz. trimethylsilyl chloride (Merck), lithium (Merck), chloroform (Merck), and substrate for preparation of alkynyl sulfide, viz. benzyl halides (Merck) were used as received. Commercially unavailable benzyl iodides were prepared from benzyl chlorides and bromides as described in the literature [26].

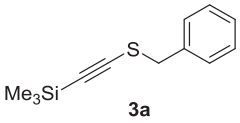
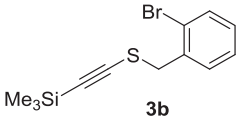
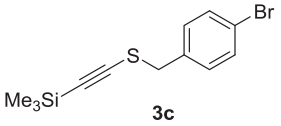
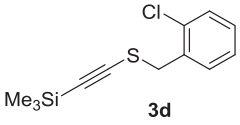
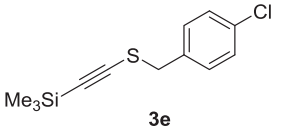
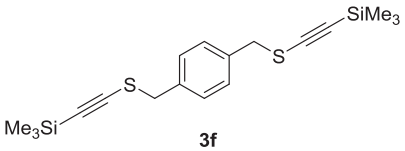
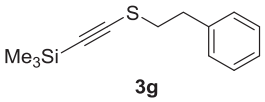
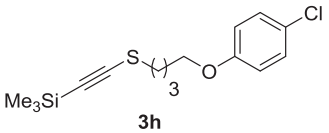
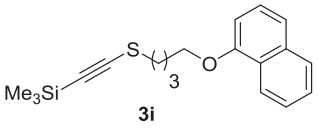


Scheme 3. Proposed mechanism.

Table 1
Synthesis of alkynyl sulfides.



3

Entry	RCH_2X	Product	Time (min)	Yield (%)[a]
1	$C_6H_5CH_2Cl$	 **3a**	15	83
2	$C_6H_5CH_2Br$	**3a**	10	85
3	$C_6H_5CH_2I$	**3a**	8	86
4	2-$BrC_6H_4CH_2Br$	 **3b**	10	87
5	2-$BrC_6H_4CH_2I$	**3b**	8	89
6	4-$BrC_6H_4CH_2Br$	 **3c**	8	90
7	4-$BrC_6H_4CH_2I$	**3c**	5	93
8	2-$ClC_6H_4CH_2Cl$	 **3d**	15	80
9	2-$ClC_6H_4CH_2Br$	**3d**	12	81
10	2-$ClC_6H_4CH_2I$	**3d**	9	83
11	4-$ClC_6H_4CH_2Br$	 **3e**	10	86
12	4-$ClC_6H_4CH_2I$	**3e**	8	88
13	4-$CH_2IC_6H_4CH_2I$	 **3f**	7	82
14	$C_6H_5CH_2CH_2Cl$	 **3g**	20	87
15	$C_6H_5CH_2CH_2Br$	**3g**	16	91
16	$C_6H_5CH_2CH_2I$	**3g**	14	94
17	4-$ClC_6H_4O(CH_2)_4Br$	 **3h**	15	89
18	4-$ClC_6H_4O(CH_2)_4I$	**3h**	12	93
19	1-naphtyl-$O(CH_2)_4Br$	 **3i**	19	85
20	1-naphtyl-$O(CH_2)_4I$	**3i**	15	88

[a] Isolated yield.

Instrumentation

The ^{1}H NMR and ^{13}C NMR were recorded with a Bruker FT-400MHz spectrometer in the indicated solvent at room temperature. The FTIR spectra were recorded on a Bruker-Tensor 270 spectrometer. Elemental analyses were carried out with a Heareus CHN-ORAPID instrument.

Preparation of tris(trimethyl)methyllithium

The reagent was prepared as described by Grobel and coworkers [22].

*Preparation of 3,3′-(1,4-phenylenebis(methylene)) bis(5-(iodomethyl)thiazolidine-2-thione) (**1**)*

This compound was prepared as described by K. D. Safa and coworkers [15].

A yellow solid, 81%, (silicagel, n-hexane/methanol 3:1, $R_f = 0.33$); FTIR (KBr, cm^{-1}): 1478, 1270, 1142 (C═S), 953, 835 cm^{-1}; ^{1}H NMR (400 MHz, DMSO): δ = 3.22 (t, $J = 10.61$ Hz, 2H, CH—I), 3.35 (dd, $J = 4.28, 10.10$ Hz, 2H, CH—I), 3.77–3.83 (m, 2H, CH-5), 3.87 (dd, $J = 2.10, 12.21$ Hz, 2H, CH-4), 4.00 (dd, $J = 12.21, 14.42$ Hz, 2H, CH-4), 4.87 (d, $J = 14.49$ Hz, 2H, CH—N), 5.06 (d, $J = 14.49$ Hz, 2H, CH—N), 7.38 (s, 4H, Ar); ^{13}C NMR (100 MHz, DMSO): δ 34.1, 36.6, 47.5, 59.6, 125.5, 133.1, 194.3 (C═S); Anal. Calcd for $C_{16}H_{18}I_2N_2S_4$: C, 30.98; H, 2.92. Found: C, 40.09; H, 3.11.

*3,3′-(1,4-phenylenebis(methylene))bis(5-((((trimethylsilyl)ethynyl) thio)methyl)thiazolidine-2-thione) (**2**)*

This compound was prepared as described by K. D. Safa and coworkers [15].

A brown sticky solid, 85%, (silicagel, n-hexane/ethylacetate 2:1, $R_f = 0.38$); FTIR (KBr, cm^{-1}): 844, 877, and 1246 (Si—C), 1144 (C═S), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 0.14 (s, 18H, $SiMe_3$), 2.68 (dd, $J = 10.36, 13.06$ Hz, 2H, CH—S), 2.98 (dd, $J = 4.66, 13.06$ Hz, 2H, CH—S), 3.83–3.85 (m, 2H, H-5), 3.91–4.03 (m, 4H, H-4), 4.90 (d, $J = 15.2$ Hz, 2H, CH—N), 4.92 (d, $J = 15.2$ Hz, 2H, CH—N), 7.55 (s, 4H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.2 ($SiMe_3$), 37.9, 41.0, 44.2, 58.2, 90.5, 101.7, 119.7, 134.1, 194.5 (C═S); Anal. Calcd for $C_{26}H_{36}N_2S_6Si_2$: C, 49.95; H, 5.80. Found: C, 50.10; H, 5.92.

*Typical procedure for the preparation of alkynyl sulfides (**3a**–**i**)*

To a stirred solution of tris(trimethylsilyl)methyllithium (1 mmol) in THF, carbon disulfide (1.2 mmol) was added in one portion at 0 °C under argon atmosphere. The mixture is stirred for 5 min and then alkyl or benzyl halides (1 mmol) or bis(halomethyl) benzene (0.5 mmol) is added at this temperature and the stirring is maintained to the end of the reaction that followed by TLC. The mixture is poured into water and extracted with CH_2Cl_2. The organic layer was washed with water, dried with Na_2SO_4 and filtered. The solvent was removed in vacuum and the product was purified by preparative column chromatography using n-hexane as eluant to give the product.

*((Benzylthio)ethynyl)trimethylsilane (**3a**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.40$); FTIR (KBr, cm^{-1}): 841, 881, and 1248 (Si—C), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.08 (s, 9H, $SiMe_3$), 3.87 (s, 2H, CH_2), 7.18–7.29 (m, 5H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.1 ($SiMe_3$), 39.2 (CH_2), 93.1 and 101.2 (C≡C), 126.7, 127.4, 128.1, 135.4; Anal. Calcd for $C_{12}H_{16}SSi$: C, 65.39; H, 7.32. Found: C, 65.51; H, 7.48.

*(((2-Bromobenzyl)thio)ethynyl)trimethylsilane (**3b**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.32$); FTIR (KBr, cm^{-1}): 841, 881, and 1248 (Si—C), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.18 (s, 9H, $SiMe_3$), 4.08 (s, 2H, CH_2), 7.18–7.22 (t, 1H, J = 7.3 Hz, Ar), 7.30–7.34 (t, 1H, J = 7.9 Hz, Ar), 7.40 (d, 1H, J = 6.7 Hz, Ar), 7.62 (d, 1H, J = 7.9 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.1 ($SiMe_3$), 39.4 (CH_2), 92.6 and 101.8 (C≡C), 123.5, 126.2, 128.3, 130.4, 132.0, 134.8; Anal. Calcd for $C_{12}H_{15}BrSSi$: C, 48.15; H, 5.05. Found: C, 48.28; H, 5.18.

*(((4-Bromobenzyl)thio)ethynyl)trimethylsilane (**3c**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.38$); FTIR (KBr, cm^{-1}): 839, 881, and 1249 (Si—C), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.15 (s, 9H, $SiMe_3$), 3.86 (s, 2H, CH_2), 7.19 (d, 2H, J = 8.0 Hz, Ar), 7.46 (d, 2H, J = 8.0 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.2 ($SiMe_3$), 38.3 (CH_2), 92.5 and 101.8 (C≡C), 120.7, 129.7, 130.5, 134.5; Anal. Calcd for $C_{12}H_{15}BrSSi$: C, 48.15; H, 5.05. Found: C, 48.27; H, 5.23.

*(((2-Chlorobenzyl)thio)ethynyl)trimethylsilane (**3d**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.35$); FTIR (KBr, cm^{-1}): 841, 881, and 1249 (Si—C), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.18 (s, 9H, $SiMe_3$), 4.07 (s, 2H, CH_2), 7.27–7.30 (m, 2H, Ar), 7.38–7.40 (m, 1H, Ar), 7.42–7.44 (m, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.2 ($SiMe_3$), 36.6 (CH_2), 92.6 and 101.7 (C≡C), 125.5, 128.2, 128.7, 130.4, 133.1, 133.1; Anal. Calcd for $C_{12}H_{15}ClSSi$: C, 56.55; H, 5.93. Found: C, 56.69; H, 6.09.

*(((4-Chlorobenzyl)thio)ethynyl)trimethylsilane (**3e**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.38$); FTIR (KBr, cm^{-1}): 841, 876, and 1248 (Si—C), 2093 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.10 (s, 9H, $SiMe_3$), 3.84 (s, 2H, CH_2), 7.24 (d, 2H, J = 8.0 Hz, Ar), 7.29 (d, 2H, J = 8.0 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.2 ($SiMe_3$), 37.9 (CH_2), 90.4 and 101.8 (C≡C), 128.1, 128.5, 132.2, 133.2; Anal. Calcd for $C_{12}H_{15}ClSSi$: C, 56.55; H, 5.93. Found: C, 56.72; H, 6.11.

*1,4-Bis((((trimethylsilyl)ethynyl)thio)methyl)benzene (**3f**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.40$); FTIR (KBr, cm^{-1}): 841, 882, and 1248 (Si—C), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.14 (s, 18H, $SiMe_3$), 3.92 (s, 4H, CH_2), 7.33 (s, 4H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.1 ($SiMe_3$), 38.9 (CH_2), 93.1 and 101.6 (C≡C), 128.5, 135.0; Anal. Calcd for $C_{18}H_{26}S_2Si_2$: C, 59.61; H, 7.23. Found: C, 59.78; H, 7.36.

*Trimethyl((phenethylthio)ethynyl)silane (**3g**)*

A yellow liquid (silicagel, n-hexane, $R_f = 0.33$); FTIR (KBr, cm^{-1}): 841, 882, and 1250 (Si—C), 2092 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.19 (s, 9H, $SiMe_3$), 2.90–2.94 (m, 2H, CH_2), 3.01–3.04 (m, 2H, CH_2), 7.19–7.24 (m, 3H, Ar), 7.28–7.31 (m, 2H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.0 ($SiMe_3$), 34.3 (CH_2), 35.6 (CH_2), 93.2 and 100.2 (C≡C), 125.6, 127.5, 127.6, 138.4; Anal. Calcd for $C_{13}H_{18}SSi$: C, 66.60; H, 7.74. Found: C, 66.76; H, 7.85.

*(((4-(4-Chlorophenoxy)butyl)thio)ethynyl)trimethylsilane (**3h**)*

A pale brown sticky solid (silicagel, n-hexane, $R_f = 0.36$); FTIR (KBr, cm^{-1}): 844, 876, and 1243 (Si—C), 1067 (C—O), 2091 (C≡C); ^{1}H NMR (400 MHz, $CDCl_3$): δ −0.01 (s, 9H, $SiMe_3$), 2.18–2.25 (m, 4H, $2CH_2$), 3.01 (t, 2H, J = 7.0 Hz, CH_2—S), 4.07 (t, 2H, J = 6.1 Hz, CH_2—O), 6.83 (d, 2H, J = 8.6 Hz, Ar), 7.11 (d, 2H, J = 8.6 Hz, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −0.4 ($SiMe_3$), 23.3, 26.5, 32.5, 65.4, 92.0 and 99.9 (C≡C), 112.7, 128.2, 140.4, 162.9; Anal. Calcd for $C_{15}H_{21}ClOSSi$: C, 57.57; H, 6.76. Found: C, 57.70; H, 6.82.

*(((4-(Naphthalen-1-yloxy)butyl)thio)ethynyl)trimethylsilane (**3i**)*

A pale brown sticky solid (silicagel, n-hexane, $R_f = 0.34$); FTIR (KBr, cm^{-1}): 840, 880, and 1245 (Si–C), 1099 (C–O), 2090 (C≡C); ^1H NMR (400 MHz, $CDCl_3$): δ 0.22 (s, 9H, $SiMe_3$), 2.09–2.15 (m, 4H, $2CH_2$), 2.91 (t, 2H, J = 6.6 Hz, CH_2–S), 4.23 (t, 2H, J = 5.5 Hz, CH_2–O), 6.84 (d, 1H, J = 7.3 Hz, Ar), 7.41 (t, 1H, J = 7.9 Hz, Ar), 7.46–7.49 (m, 1H, Ar), 7.50–7.56 (m, 2H, Ar), 7.83–7.85 (m, 1H, Ar), 8.30–8.33 (m, 1H, Ar); ^{13}C NMR (100 MHz, $CDCl_3$): δ −1.0 ($SiMe_3$), 25.0, 26.8, 34.3, 66.3, 93.4 and 99.7 (C≡C), 103.5, 119.2, 120.9, 124.1, 124.6, 124.8, 125.4, 133.5, 153.6; Anal. Calcd for $C_{19}H_{24}OSSi$: C, 69.46; H, 7.36. Found: C, 69.55; H, 7.47.

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