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# A direct approach for the expedient synthesis of unsymmetrical ethers by employing bromodimethylsulfonium bromide (BDMS) mediated C-S bond cleavage of naphthalene-2-ol sulfides

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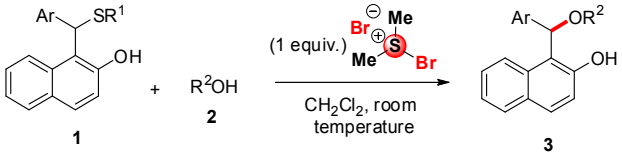
The unsymmetrical ether derivatives 1-(alkoxy(aryl)methyl)naphthalen-2-ols (**3a-q**) were synthesized from 1-[aryl(alkyl/arylthio)methyl)-naphthalene-2-ol derivatives (**1**) and alcohols (**2**) by cleavage of C-S bond using bromodimethylsulfonium bromide (BDMS) at room temperature. Moreover, 1-(hydroxy(aryl)methyl)naphthalen-2-ol derivatives (**7a-d**) were also synthesized from the corresponding sulfide derivatives (**1**) on reaction with water using one equivalent BDMS. The direct synthesis of **3a** was also achieved from β-napthol **5** by tuning the reaction conditions with overall one-pot two-steps sequence. Interestingly, the desired products **3a** are not accessible directly from 2-naphthol, aromatic aldehyde and alcohol in the presence of BDMS at room temperature. Some of the salient features of this protocol are mild reaction conditions, good yields, operational simplicity and with a wide substrate scope.

## Introduction

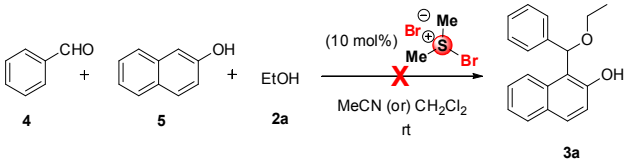
The construction of an ether linkage adjacent to a sterically hindered carbon center is an important synthetic step for the synthesis of many biologically active compounds.[1] Ethers are important structural motifs for life sciences and polymer industries.[2] The conventional method for ether synthesis is the direct $SN_2$-type *O*-alkylation also known as Williamson ether synthesis.[3] However, this protocol is sometimes synthetically impractical owing to the strong basicity of the alkoxide anion, which may be incompatible with other functional groups present in the system.[4] A variety of transition metal[5] or Lewis acid[6] catalyzed cross-coupling reactions of different alcohols have also been used for the synthesis of ethers. It is well-known that substituted 2-naphthoquinone-1-methide intermediates can be generated easily from 2-naphthol and aromatic aldehydes in the presence of a suitable catalyst and it has been utilized for the synthesis of carbamate derivatives using aza-Michael reaction,[7a] which is better known as Betti reaction.[7b] Recently we have demonstrated that synthesis of 1-[(alkyl/arylthio)(phenyl)methyl]-naphthalene-2-ol derivatives (**1**) from *in situ* generated 2-naphthoquinone-1-methide intermediate involving thia-Michael reaction.[8] From this result, we inspired to investigate whether *in situ* generated substituted 2-naphthoquinone-1-methide[9] intermediates used for the synthesis of ethers with alcohols by employing oxa-Michael reaction.[10] In this paper we would like to disclose the synthesis of hitherto unreported ether derivatives (**3**) from 1-[aryl(alkyl/arylthio)methyl)-naphthalene-2-ol derivatives (**1**) by cleavage of C-S bond using one equivalent BDMS followed by oxa-Michael type reaction with alcohols (**2**) using alternate pathway as shown in Scheme 1.




**Scheme 1**. Synthesis of 1-(alkoxy(aryl)methyl)naphthalen-2-ol derivatives (**3**)

## Results and Discussion

Initial attempts with a mixture of benzaldehyde (**4**, 1 mmol), 2-naphthol (**5**, 1 mmol) and ethyl alcohol (**2a**, 2 mmol) was examined in the presence of 10 mol % of BDMS in 2 mL of $CH_3CN$ or DCM at room temperature. Unfortunately, the attempt was unsuccessful as shown in Scheme 2 and the expected unsymmetrical ether (**3a**) is not formed instead of that we have isolated 1-bromo-2-naphthol in trace amount.




**Scheme 2**.Unsuccessful attempt for the synthesis of ether (**3a**)

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Electronic Supplementary Information (ESI) available: [X-ray crystallographic data of **3p** and **7a** CCDC no. is913509 and 913508and copies of the $^1H$ and $^{13}C$ NMR spectra for all compounds]. See DOI: 10.1039/x0xx00000x

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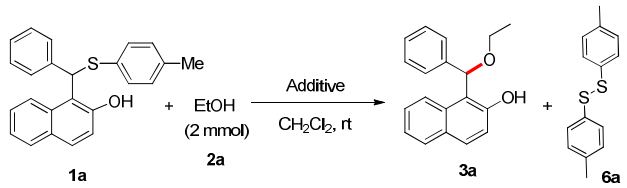
From the results above, we turned our attention for devising an alternate strategy. Recently our research group demonstrated that organic ammonium tribromides (OATB) and *in situ* generated bromonium ion can be utilized for C-S bond cleavage in deprotection of dithioacetals[11] and in hydrolysis of 1-thioglycosides.[12] Therefore, we thought that it might be possible to synthesize 1-(alkoxy(aryl)methyl)naphthalen-2-ol derivatives (**3**) from 1-[aryl(alkyl/arylthio)methyl)-naphthalene-2-olderivatives (**1**) by cleavage of C-S bond through activation with bromonium ion generated from BDMS followed by nucleophilic attack with alcohols (**2**).

**Table 1**. Optimization of reaction conditions for the synthesis of 1-(ethoxy(phenyl)methyl)-naphthalen-2-ol (**3a**)[a]



1a 2a 3a 6a

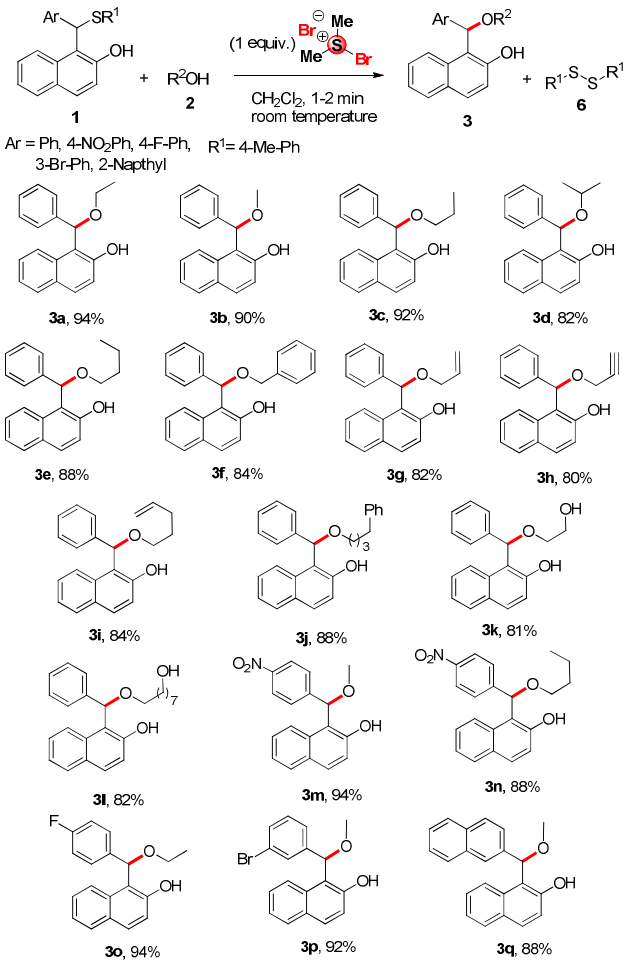
| Entry | Additive | Amount (equiv.) | Reaction time | **3a** Yield[b] (%) |
|---|---|---|---|---|
| 1 | BDMS | 0.10 | 2 h | trace |
| 2 | BDMS | 0.50 | 2 h | 44 |
| 3 | BDMS | 0.75 | 2 h | 61 |
| 4 | BDMS | 1.00 | 2 min | 94 |
| 5 | $Br_2$ | 1.00 | 2 min | 80 |
| 6 | NBS | 1.00 | 1 h | 86 |
| 7 | TBATB | 1.00 | 1 h | 72 |

[a]All the reactions were performed with **1a** (1.0 mmol). [b]Isolated yield.

Bromodimethylsulfonium bromide (BDMS) is a useful brominating agent as well as highly efficient pre-catalyst, which has been used extensively by us[13] and others[14] for numerous organic transformations. We have prepared the unsymmetrical sulfide (**1a**) from benzaldehyde (**4**), 2-naphthol (**5**) and 4-methylthiophenol by following our earlier reported procedure.[8a] Then, various trial reactions were examined with unsymmetrical sulfide **1a** using different amount of BDMS as shown in Table 1. We have isolated the desired product **3a** along with unreacted starting material **1a** (entries 1-3, Table 1) in the presence of 0.10, 0.50 and 0.75 equivalent of BDMS. We have noted the complete conversion with 1.0 equivalent of BDMS using 2 mmol of EtOH in DCM (2 mL) at room temperature and the desired product **3a** was isolated in 94% (entry 4, Table 1). The required product **3a** and the by-product **6a**[15] were characterized from $^1$H NMR and $^{13}$C NMR analysis, which encouraged us to further investigate the reaction. We have also noted that similar transformation is also possible using molecular bromine, NBS and TBATB (entries 5-7, Table 1) however, they provide the desired product in lower yield as compared to BDMS.

After optimizing the reaction conditions, we prepared various sulfide derivatives (**1a-f**) from aromatic aldehydes, 2-naphthol (**5**) and 4-methylthiophenol using 10 mol% BDMS as catalyst in acetonitrile at room temperature (entries 1-6, Table SI-1, Supporting Information). Similarly, other sulfides (**1g-i**) were also prepared using different thiols such as 4-chlorothiophenol, ethane thiol and propanethiol under identical reaction conditions (entries 7-9, Table SI-1, Supporting Information).

**Table 2**. Substrate scope of substituted 1-(alkoxy(aryl)methyl) naphthalen-2-ol derivatives **3**[a]




[a]The reactions were carried out with **1** (1.0 mmol) and different aliphatic alcohols **2** (2.0mmol) in the presence of one equiv. of BDMS in 2 mL of DCM at room temperature.

Next, several reactions were performed with compound **1a** with various alcohols like methanol (**2b**), *n*-propanol (**2c**), *iso*-propanol (**2d**), *n*-butanol (**2e**), benzyl alcohol (**2f**) and the desired products **3b-f** were obtained in good yields on treatment with one equivalent of BDMS at room temperature. The reactions completed instantaneously and the percentage yields of the products are shown in Table 2 (entries 2-6).

Subsequently, the reactions were carried out with different alcohols such as allyl alcohol (**2g**), propargyl alcohol (**2h**), 4-pentene-1-ol (**2i**), 4-phenylbutan-1-ol (**2j**), 1,2-ethanediol (**2k**) and 1,8-octanediol (**2l**) with **1a** under identical reaction conditions and the desired products **3g-l** were isolated in good yields (Table 2, entries 7-12). The product **3k** was further

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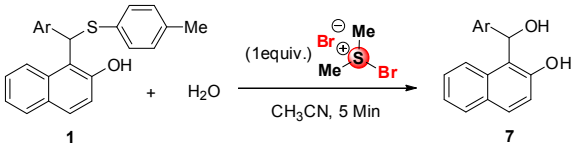
confirmed by acetylation, which was characterized from $^{1}H$ NMR and $^{13}C$ NMR spectra.

To explore the synthetic scope and generality of the present protocol, other sulfides **1b-d** containing different substituents in the aromatic ring such as $NO_2$, F, and Br were treated with methanol (**2b**), ethanol (**2a**) and butanol (**2b**), in the presence of one equivalent BDMS and the desired products **3m-p** were isolated in good yields as shown in Table 2 (entries 13-16). Similarly, 1-(naphthalen-2-yl(*p*-tolylthio)methyl)naphthalen-2-ol(**1e**) react with methanol under similar reaction condition and the expected products **3q** obtained in 88 % yield respectively.

It is worthwhile to mention that sulfides synthesized from aliphatic thiols such as **1h** and **1i** can be transformed into the product **3b** and **3m** with 88% and 90% in the presence of methanol using one equivalent of BDMS at room temperature. However, the expected product is not formed in case of phenol under similar reaction condition.

**Table 3**. Synthesis of substituted 1-(hydroxy(aryl)methyl) naphthalen-2-ol derivatives (**7**) using $H_2O$ as nucleophile[a]




1 7

| Entry | Ar (**1**) | Product (**7**) | Yield[b] (%) |
|---|---|---|---|
| 1 | $C_6H_5$ | **7a** | 84 |
| 2 | $4NO_2$-$C_6H_5$ | **7b** | 88 |
| 3 | 4F-$C_6H_5$ | **7c** | 86 |
| 4 | 4Cl-$C_6H_5$ | **7d** | 84 |

[a]The reactions were carried out with **1** (1.0 mmol) and water (40 µL) in the presence of one equiv. of BDMS in 2 mL of $CH_3CN$ at room temperature. [b]Isolated Yield.

Encouraged by these successful results, we thought to explore the synthesis of 1-(hydroxy(aryl)methyl)naphthalen-2-ol (**7**) derivatives using unsymmetrical sulfide **1** with water as a nucleophile.[16] Various other aromatic sulfide derivatives **1a-c** and **1f** on treatment with one equivalent of BDMS in acetonitrile solvent at room temperature provided the expected products **7a-d** in good to excellent yield as shown in Table 3.

We have also carried out the reaction with different substituted sulphur moiety such as 1-(((4-chlorophenyl)thio)(*p*-tolyl)methyl)naphthalen-2-ol **1g** with ethanol or water under identical reaction condition which afford the required product **8** and **9** with 90% and 82% yield as shown in Scheme 3.

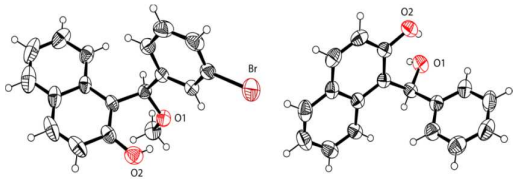
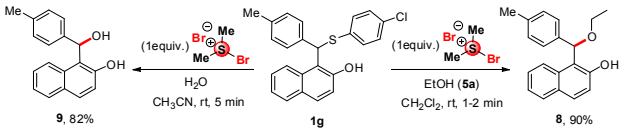



**Fig. 1**. Crystal structures of **3p** and **7a**

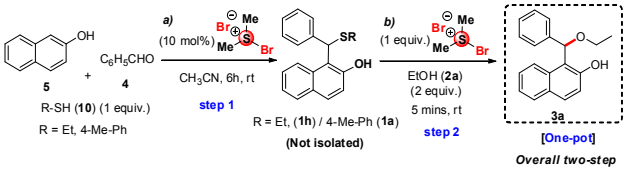
Finally, the structures of the representative compounds 1-(alkoxy(aryl)methyl) naphthalen-2-ol such as (**3p**) and 1-(hydroxy(aryl)methyl)naphthalen-2-ol (**7a**) were also ascertained by single crystal X-ray diffraction data as shown in Figure 1.




**Scheme 3**. Cleavage of 4-chlorophenylthio derivative for the synthesis of ether **8** and hydrins **9**

The compounds **3p** and **7a** were crystallized in P21/n monoclinic and P21/c space group, respectively. The selected bond lengths and angles are given in Table SI-2 (See Supporting Information). The non-covalent strong and weak interactions such as O-H•••O, C-H•••O and C-H•••π facilitate the orientation of molecules in lattice arrangement in both of crystals. The weak hydrogen bonding interactions C-H•••O play the constructive role in stabilizing the solid-state structure of **3p**. In **7a**, intra- and inter-molecular O-H•••O hydrogen bonding forms one dimensional (1D) zigzag chains which further undergo C-H•••π interactions to furnish 2D sheets in the *ac* plane as shown in Figure SI-1 (Supporting Information). The hydrogen bond parameters for both the compounds are given in Table SI-4 (See Supporting Information).

Next, we thought whether it is possible to achieve the transformation in a one-pot manner, by trapping 2-naphthoquinone-1-methide intermediate with thiol using 10 mol% BDMS followed by cleavage of it with another equivalent of BDMS. For this purpose, two set of reactions were conducted using benzaldehyde, 2-naphthol and ethane thiol or 4-methylthiophenol followed by addition of 2 equiv. of ethanol as shown in Scheme 4 and the desired product **3a** was isolated in 64% and 70% yield, respectively. It is noteworthy to mention here that when similar reaction was carried out without consequent addition of BDMS (*i.e*) 1.1 equivalent of BDMS at once, it gave exclusively 1-bromo-2-naphthol instead of the expected product **3a**.




**Scheme 4**. One-pot synthesis of 1-(alkoxy(aryl)methyl) naphthalen-2-ol (**3a**)

To understand the mechanism of the reaction, we have carried out four different type of reactions as shown in Scheme 5. At first, the substrate benzyl(*p*-tolyl)sulfane **11** was treated with treatment of 1 equiv. BDMS in the presence of 2 mmol of ethanol in 2 mL of DCM to obtain desired product **12.** Unfortunately, we didn't get the expected benzyl ethyl ether. Next we thought by putting a hydroxyl group at the *ortho*-

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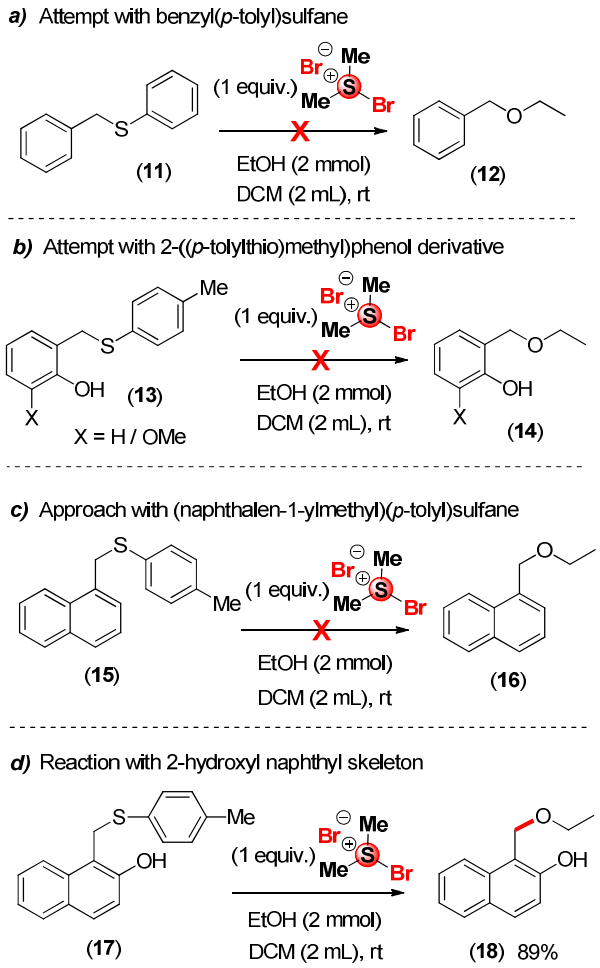
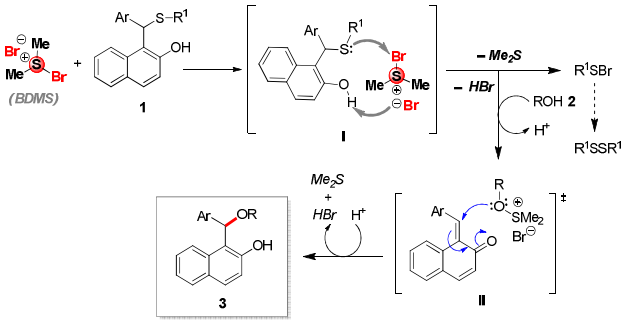
position of the phenyl ring, we may get the expected ether **14** from cleavage of thioether **13**. In this case, the reaction failed. The failure of the reaction may be due to *ortho*-quinone-1-methide intermediate is very much less stable as compared to 2-naphthoquinone-1-methide intermediate. Subsequently, another reaction was carried out with fused aromatic ring of (naphthalen-1-ylmethyl)(*p*-tolyl)sulfane **15** under similar reaction condition and the product **16** was not formed as shown in Scheme 5. Interestingly, the installation of hydroxyl group in 2-position of (naphthalen-1-ylmethyl)(*p*-tolyl)sulfane **17** on treatment with 1 equivalent of BDMS in the presence of 2 mmol of ethanol in 2 mL of DCM which afford successfully 1-(ethoxymethyl)naphthalen-2-ol **18** in 89% yield as shown in Scheme 5. The obtained results indicated that presence of hydroxyl group in *ortho*-position of naphthalene ring in sulphide plays a crucial role for carrying out the transformations for the synthesis of unsymmetrical ether using alcohols.




**Scheme 5**. Investigation on reactant profiles

The plausible mechanism for the formation of the product may be explained as follows: Initially, BDMS can activate the sulphur atom on **1** for cleaving C-S bond and it tend to form an intermediate **I**, then it prefer to release dimethylsulfide, HBr and $R^1SBr$. The incoming alcohol **2** may react with dimethyl sulphide and HBr to undergo oxa-Michael type reaction which proceed through the intermediate **II**. Subsequently, the incoming $H^+$ ion intend to release dimethylsulfide and HBr which lead to the product **3** as shown in Scheme 6. The failure of the two reactions in Scheme 5 (**a** and **c**) evidently discard the possibility of the reaction going through direct nucleophilic substitution. The presence of -OH group at the *ortho*-position of naphthalene ring in intermediate **I** plays a prime role to facilitate the cleavage of C-S bond.




**Scheme 6**. Mechanism for the formation of 1-(alkoxy(aryl) methyl)naphthalen-2-ol **3**

## Conclusions

In short, we have developed a new methodology for the synthesis of unreported unsymmetrical ether derivatives 1-(alkoxy(aryl)methyl)naphthalen-2-ols using 1-[aryl(alkyl/aryl thio)methyl)-naphthalene-2-ol and alcohols in the presence of BDMS. This protocol might be useful for the preparation of highly substituted ethers and in addition, the reaction is simple, fast and transformation is quite effective for a wide range of substrates. Moreover, we have also demonstrated that direct transformation of **3a** can be achieved in a one-pot two-step manner through consequent addition of 1.1 equiv. amount of BDMS.

## General Procedure

### I. Synthesis of 1-(alkoxy(aryl)methyl)naphthalen-2-ol (3)

In a 25 mL round bottom flask a mixture of compound **1** (1.0 mmol) and alcohol (2.0 mmol) was taken in 2 mL of DCM. Then, 1 equivalent of BDMS was added to the reaction mixture and the reaction was completed instantaneously. After completion of reaction, the reaction mixture was extracted with DCM (1 x 25 mL) and washed with aqueous sodium bicarbonate. The organic layer was dried over $Na_2SO_4$ and it was concentrated in a rotatory evaporator. After purification through a silica gel (60-120 mesh) column chromatography, the pure product **3** was obtained in good yield. Similar reaction procedure was followed for the preparation of compounds **8**.

### II. One-pot synthesis of 1-(ethoxy(phenyl)methyl)naphthalen-2-ol derivatives (3a)

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Bromodimethylsulfonium bromide BDMS (0.2 mmol) was added to a mixture of benzaldehyde (2.0 mmol) and 2-naphthol (2.0 mmol) in 5 mL of acetonitrile and the reaction mixture was kept for stirring at room temperature. Then, 4-methylthiophenol (2.0 mmol) was added to it and the progress of the reaction was monitored by TLC. After 6 h of stirring, 2.0 mmol of ethanol (120 µL) and 1 equivalent BDMS were added into it and the reaction completed instantly. Then, acetonitrile was removed in a rotatory evaporator and the residue was extracted with dichloromethane (1 x 25 mL) and washed with aqueous sodium bicarbonate. The organic layer was washed with water and dried over anhydrous sodium sulfate. On concentration followed by purification through a silica gel column, the desired product **3a** was obtained.

#### III. Synthesis of 1-(hydroxy(aryl)methyl)naphthalen-2-ol (7)

Into a 25 mL round bottom flask was taken 1 mmol of **1** in 2 mL of $CH_3CN$. To this reaction mixture, 40 µL of water and 1 equivalent of BDMS were added successively and the reaction mixture was stirred at room temperature for 5 minutes and the progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was concentrated in a rotatory evaporator and the crude residue was extracted with DCM (1 x 25 mL) and washed with water. The organic layer was dried over $Na_2SO_4$ and it was concentrated *in vacuo*. The pure product **7** was obtained in good yield after purification through a silica gel (60-120 mesh) column chromatography. Similarly, **9** were obtained by following identical reaction procedure.

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