Tetrabutylammonium Phthalimide-*N*-oxyl: An Efficient Organocatalyst for Trimethylsilylation of Alcohols and Phenols with Hexamethyldisilazane

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Tetrabutylammonium phthalimide-*N*-oxyl (TBAPINO) was found to be an effective organocatalyst for rapid, simple and chemoselective protection of the hydroxyl group of alcohols and phenols using hexamethyldisilazane (HMDS) under mild conditions. The low catalyst loading, high to quantitative yields and simple removal of the catalyst from the reaction mixture illustrate the other attractive features of this protocol.

Keywords: Trimethylsilylation, Alcohols, Phenols, Hexamethyldisilazane, Tetrabutylammonium phthalimide-*N*-oxyl, Organocatalysis

INTRODUCTION

At the dawn of the 21st century, organic chemistry witnessed a revolution in catalysis using small organic compounds-Organocatalysis. Nowadays, organocatalytic protocols are extensively pursued to replace the traditional catalytic approaches based on the use of toxic or precious transition-metals for the synthesis of organic compounds. Organocatalysts have several significant advantages over traditional metal catalysis. They are usually robust, inexpensive, readily available and non-toxic. Many organocatalysts are inert towards moisture and oxygen. Because of these unique features, no reaction conditions such as inert atmosphere, absolute solvents, *etc.* are to be observed in many instances. Furthermore, due to the absence of transition metals, organocatalytic methods seem to be especially attractive for the preparation of compounds that do not tolerate metal contamination, especially pharmaceuticals.

Thus, organocatalysts are making a valuable contribution to green chemistry [1,2].

Protection of hydroxyl groups is a common practice in synthetic organic and analytical chemistry. Various protecting methods such as tetrahydropyranylation, esterification, esterification and trialkylsilylation have been used for this purpose [3,4]. Among these methods, trimethylsilylation of hydroxyl group has received a lot of attention due to thermal stability, solubility in non-polar solvents, and enhanced stability of the resulting trimethylsilyl (TMS) ether derivatives under a variety of conditions. Furthermore, the removal of the protecting group can be easily accomplished by acid- or base-induced hydrolysis [3,5]. Trimethylsilylation is also extensively used for the derivatization of hydroxyl compounds to increase their volatility for gas chromatography and mass spectrometry [6]. Hence, different silylating agents such as chlorotrimethylsilane [7-9], trimethylsilyl triflate [10], hexamethyldisilane [11] and hexamethyldisiloxane have been used in the presence of a broad range of catalysts and reagents for the introduction of TMS group into a variety of alcohols or

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phenols [12].

Although these methods demonstrate improvements in many cases, they suffer from the use of expensive, uncommon or highly moisture sensitive silylating agents and catalysts, the lack of reactivity and difficulty in the removal of by-products produced during the reaction.

Among different silylating agents, hexamethyldisilazane (HMDS) is a stable, inexpensive, commercially available and easy-to-handle compound which can be used for the preparation of TMS ethers from alcohols and phenols. The work-up of the reaction is not time consuming since the only by-product of the reaction is ammonia which can be easily removed from the reaction media. However, this reagent requires to be activated by a catalytic system for its reaction. Therefore, varieties of catalytic systems including inorganic catalysts have been developed for activating this reagent [12-23].

Recently, we have introduced tetrabutylammonium phthalimide-*N*-oxyl (TBAPINO) or potassium phthalimide-*N*-oxyl (PPINO) as efficient organocatalysts for cyanosilylation of carbonyl compounds and cyclotrimerization of isocyanates under mild conditions [24-27]. In continuation of our efforts to develop the catalytic scope of the phthalimide-*N*-oxyl (PINO) anion, we herein disclose the application of TBAPINO **1** as a new and efficient metal-free catalyst for a chemoselective and very simple protection of alcohols and phenols using HMDS to afford corresponding trimethylsilyl ethers under mild conditions (Scheme 1).

EXPERIMENTAL

Chemicals and Apparatus

All chemicals were purchased from Merck or Aldrich and used as received. EtOAc was purchased from Merck and kept over 4 Å molecular sieve supplied by Carl Roth GmbH + Co, 76185 Karlsruhe, Germany. The catalyst was powdered and dried at 70 °C for 1 h under reduced pressure. All reactions were protected from air moisture using a $CaCl_2$ guard tube. Analytical TLC was carried out using Merck 0.2 mm silica gel 60 F-254 Al-plates.

FT IR spectra were recorded as KBr pellets on a Shimadzu FT IR-8400S spectrometer. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were obtained using a Bruker DRX-500 AVANCE spectrometer. All NMR spectra were determined in CDCl$_3$ or DMSO at ambient temperature. GC chromatograms were recorded on a Shimadzu 2010 instrument.

General Procedure for the Preparation of Trimethylsilyl Ethers (3a-u or 5a-j)

To a mixture of alcohol or phenol (1 mmol), TBAPINO (8.1 mg, 0.02 mmol) and EtOAc (1 ml) was added HMDS (0.55 mmol) and the mixture was refluxed for an appropriate length of time indicated in Tables 2 and 3. Upon completion of the reaction as judged by TLC or GC, water (2.5 ml) was added and the organic materials were extracted with EtOAc. The organic phase was washed with brine followed by water (2.5 ml) and dried over MgSO$_4$. The solvent was evaporated under reduced pressure to afford the product, in almost pure form, which was further purified by column chromatography on silica gel (Hexane/Ethyl acetate 8:2). All compounds were well characterized by GC analysis, IR and NMR spectral data as compared with those obtained from authentic samples or reported in the literature [13,14,20,22].

RESULTS AND DISCUSSION

Review of the literature shows that only few
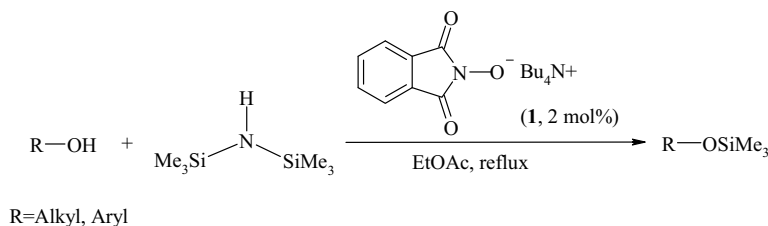
Scheme 1. Trimethylsilylation of alcohol and phenols catalyzed by TBAPINO **1**

organocatalytic protocols using Trichloroisocyanuric acid (TCCA) [28], *N*-bromosuccinimide (NBS) [29], 1,3-dibromo-5,5-diethylbarbutric acid [30], *N,N,N',N'*-tetrabromobenzene-1,3-disulfonamide [31,32] and tetrabutylammonium bromide (TBAB) [33] have been developed for the protection of hydroxyl functional group in recent years. It is noteworthy that all of the above-mentioned catalytic systems fall into Lewis acid-activation of HMDS category, except TBAB. Therefore, the development of new and alternative Lewis basic catalysts which operate under milder conditions would be desirable. On the basis of our experiences with PINO-catalyzed cyanide addition reactions with trimethylsilyl cyanide (TMSCN) [24-25], we postulated that the PINO nucleophile should activate HMDS in an analogous fashion.

First, the catalytic applicability of TBAPINO was examined in the activation of HMDS for the protection of benzyl alcohol **2a** as a model reaction to optimize the required amount of HMDS, reaction media, substrate to catalyst mole ratio and temperature (Table 1). The progress of the reaction was easily monitored by TLC. The best result in terms of turnover number (TON) and turnover frequency (TOF) was achieved by using 2 mol% of the catalyst **1** in EtOAc under reflux conditions (entry 3, Table 1). Indeed, when a mixture of **2a** and 2 mol% of **1** in EtOAc was treated with 0.55 equivalent of HMDS, the trimethylsilylation of benzyl alcohol occurred smoothly under reflux conditions after 20 min to afford the desired trimethylsilyl ether (**3a**) in 98% isolated yield. In the absence of catalyst, the reaction was much less efficient for the conversion of benzyl alcohol to the corresponding TMS ether and only a trace amount of the product was formed after 24 h (Table 1, entry 9).

Then, the optimal conditions were applied to the trimethylsilylation of other alcohols. The protection of a wide range of primary and secondary alcohols was successfully achieved in high to quantitative yields. The obtained results have been summarized in Table 2. In the presence of this catalyst, benzylic alcohols and primary alcohols bearing acid-sensitive heteroaryl substituents were readily transformed into their corresponding TMS ethers (**3a-k**) in excellent yields (Table 2, entries 1-11). However, benzylic alcohols containing electron-donating substituents (entries 2-5) demonstrated shorter reaction times compared to alcohols with electron-
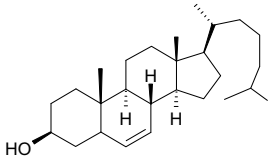
Table 1. Optimization of the Trimethylsilylation of Benzyl Alcohol Catalyzed by TBAPINO[a]

$$PhCH_2OH \xrightarrow{\text{HMDS (0.55 equiv.), TBAPINO (1)}} PhCH_2OTMS$$
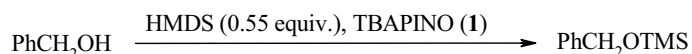
Entry	Mol (%)	Solvent	Time (min)	Temp. (°C)	Yield (%)[b]	TON[c]	TOF (h[-1])[d]
1	2.0	CH$_3$CN	25	reflux	91	46	110
2	2.0	DMSO	25	70 °C	93	46	110
3	2.0	EtOAc	20	reflux	98	49	147
4	2.0	CH$_2$Cl$_2$	30	reflux	98	49	98
5	1.5	EtOAc	60	reflux	94	63	63
6	1.0	EtOAc	120	reflux	95	95	48
7	3.0	EtOAc	25	reflux	96	32	77
8	2.0	EtOAc	60	rt	94	47	47
9	-	EtOAc	24 h	reflux	15	-	-

[a]HMDS (0.55 mmol) was added to a mixture of benzyl alcohol (1.0 mmol) and the required amount of TBAPINO in 1 ml of the solvent. [b]Isolated yields (average of at least 2 runs) after complete conversion of the substrate. [c]Turnover number: moles of product per mole of catalyst. [d]Turnover frequency: moles of product per mole of catalyst per hour.

Dekamin *et al.*

Table 2. Protection of Alcohols Catalyzed by TBAPINO under Optimized Reaction Conditions[a]
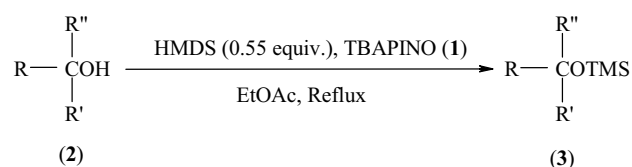
Entry	R	R'	R"	Product	Time (min)	Yield (%)[b,c]
1	Ph	H	H	**3a**	20	94
2	4-MeC$_6$H$_4$	H	H	**3b**	15	96
3	4-MeOC$_6$H$_4$	H	H	**3c**	15	98
4	3,4-(MeO)$_2$C$_6$H$_3$	H	H	**3d**	30	91
5	2-MeOC$_6$H$_4$	H	H	**3e**	45	85
6	4-ClC$_6$H$_4$	H	H	**3f**	25	95
7	2-ClC$_6$H$_4$	H	H	**3g**	30	97
8	4-NO$_2$C$_6$H$_4$	H	H	**3h**	60	94
9	2-NO$_2$C$_6$H$_4$	H	H	**3i**	75	93
10	2-Furyl	H	H	**3j**	20	90
11	2-Thienyl	H	H	**3k**	25	87
12	Me	H	H	**3l**	60	96
13	1-Hexyl	H	H	**3m**	150	90
14	CH$_3$(CH$_2$)$_{16}$-	H	H	**3n**	120	95
15	Vinyl	H	H	**3o**	60	96
16	Ph	Me	H	**3p**	45	94
17	Ph	Ph	H	**3q**	45	95
18	PhCO	Ph	H	**3r**	45	94
19	Cylohexyl		H	**3s**	150	92
20				**3t**	120	92
21	Ph	Ph	Ph	**3u**	180	0

[a]HMDS (0.55 mmol) was added to a mixture of alcohol or phenol (1.0 mmol) and 8.1 mg of TBAPINO in EtOAc (1 ml). [b]Isolated yields (average of at least 2 runs) after complete conversion of the substrates. [c]All compounds were characterized on the basis of spectroscopic data (IR, ^{1}H NMR, ^{13}C NMR) by comparison with those reported in the literature [13,14,20,22].

540

withdrawing substituents (entries 6-9). Aliphatic primary alcohols were also subjected to the same conditions. As a result, the corresponding TMS ethers of these substrates (**3l-o**) were obtained in high yields within longer time periods (Table 2, entries 12-15). It is noteworthy that acid sensitive, allyl and saturated alcohols react under the optimized reaction conditions without the formation of any side products such as polymerisation, rearrangement or dehydration (entries 10-15) [12]. Secondary alcohols also underwent trimethyl-silylation under the optimized reaction conditions (Table 2, entries 16-20). However, secondary alcohols required longer reaction times compared to similar primary alcohols. Interestingly, cholesterol **2t** was involved smoothly in the optimized reaction conditions to afford the desired protected product (**3t**) in 92% yield (entry 20) [12]. On the other hand, tertiary alcohols such as triphenyl carbinol remained intact even after 3 h under the optimized reaction conditions (Table 2, entry 21) [33].

Furthermore, trimethysilylation of different phenols and naphthols with HMDS under the optimized conditions was also performed smoothly and cleanly in high yields. The obtained results have been summarized in Table 3. In general, phenols and naphthols underwent silylation at longer reaction times compared to benzylic alcohols while the same trend of substituent effects was observed. Also, no reactivity was observed for the thiophenolic substrate in the optimized reaction conditions even in the presence of activating amino group at the *ortho*- position (Table 3, entry 10).

To investigate the chemoselectivity of the protocol, some competitive reactions were designed. The related data clearly demonstrate exclusive protection of phenolic functional group in the presence of its aromatic amine competitor. Indeed, only the product of protection of the phenolic functionality (**5b**) was produced by using both 0.55 and 1.1 equivalent of HMDS in the reaction conditions (Sch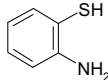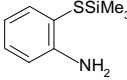eme 2, Table 3, entry 2). On the other hand, in a mixture of benzyl alcohol **2a** and phenol **4a**, the primary alcohol was preferentially converted to the corresponding TMS ether (78% **3a** *vs*. 22% **5a**). The same preference was also observed when the primary benzyl alcohol **2a** was subjected to reaction with HMDS in the presence of secondary or tertiary hydroxyl groups such as benzhydrol (72% **3a** *vs*. 28% **3q**) and triphenyl carbinol (100% **3a** *vs*. 0% **3q**), respectively.

The hypercoordinate silicon species are well known due to their role in developing alternative pathways in organic synthesis [34-37]. Accordingly, significant progress has been made during the recent years for the Lewis base-catalyzed reactions using silylated reagents. Among the Lewis base catalysts, oxygen-containing nucleophiles are more efficient due to the high bond strength as well as the kinetic lability of the Si-O bond. Therefore, oxygen-containing nucleophiles are suitable activators in silylated reagents chemistry such as HMDS [38-39]. This activity could be interpreted in terms of appropriate hardness of the oxygen-containing nucleophiles, as hard bases, to interact efficiently with the trimethylsilyl moiety as a hard acid [40-42]. According to the literature and reactivity of different alcohols or phenols in this study, the mechanism purposed in Scheme 3 can be considered for the conversion of alcohols or phenols to their corresponding trimethylsilyl ether in the presence of TBAPINO **1** [25,34-44]. Accordingly, the initial attack of the more nucleophilic PINO anion on trimethylsilyl groups of **6a** or **6b** produces the pentacoordinated intermediate **I**. The intermediate **I** can form the hexacoordinated intermediate **II** by the subsequent attack of less nucleophilic alcohol (**2**) or phenol (**4**) to expand the valency of silicon, which has a low-lying empty *d*- orbital. Then, an intramolecular acid-base reaction produces the hexacoordinated intermediate **III** which can then decompose to give the product (**3** or **5**) and regenerate the catalyst **1**
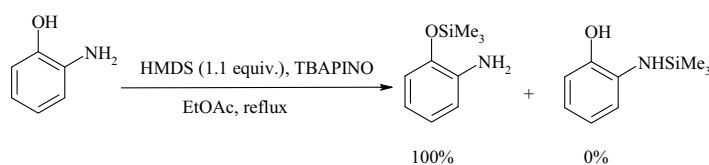
Scheme 2. Chemoselective protection of phenolic functional group in the presence of its aromatic amine competitor

Table 3. Protection of Phenols Catalyzed by TBAPINO under Optimized Reaction Conditions[a]

$$ArOH \xrightarrow[\text{EtOAc, Reflux}]{\text{HMDS (0.55 equiv.), TBAPINO (1)}} ArOTMS$$

(4) (5)

Entry	Ar	Product	Time (min)	Yield (%)[b,c]
1	Ph	**5a**	60	91
2[d]	2-$NH_2C_6H_4$	**5b**	45	94
3	3,4-$(MeO)_2C_6H_3$	**5c**	45	90
4	2-Benzyl-4-ClC_6H_4	**5d**	25	95
5	2-EtC_6H_4	**5e**	45	94
6	4-EtC_6H_4	**5f**	40	92
7	2-$NO_2C_6H_4$	**5g**	75	95
8	1-Naphthyl	**5h**	60	86
9	2-Naphthyl	**5i**	60	82
10	(2-aminophenyl)-SH	(2-aminophenyl)-$SSiMe_3$ **5j**	180	N.R

[a]HMDS (0.55 mmol) was added to a mixture of alcohol or phenol (1.0 mmol) and 8.1 mg of TBAPINO in EtOAc (1 ml). [b]Isolated yields (average of at least 2 runs) after complete conversion of the substrates. [c]All compounds were characterized on the basis of spectroscopic data (IR, ^{1}H NMR, ^{13}C NMR) by comparison with those reported in the literature [13,14,20,22]. [d]The reaction was performed using both 0.55 and 1.1 equivalent of HMDS.
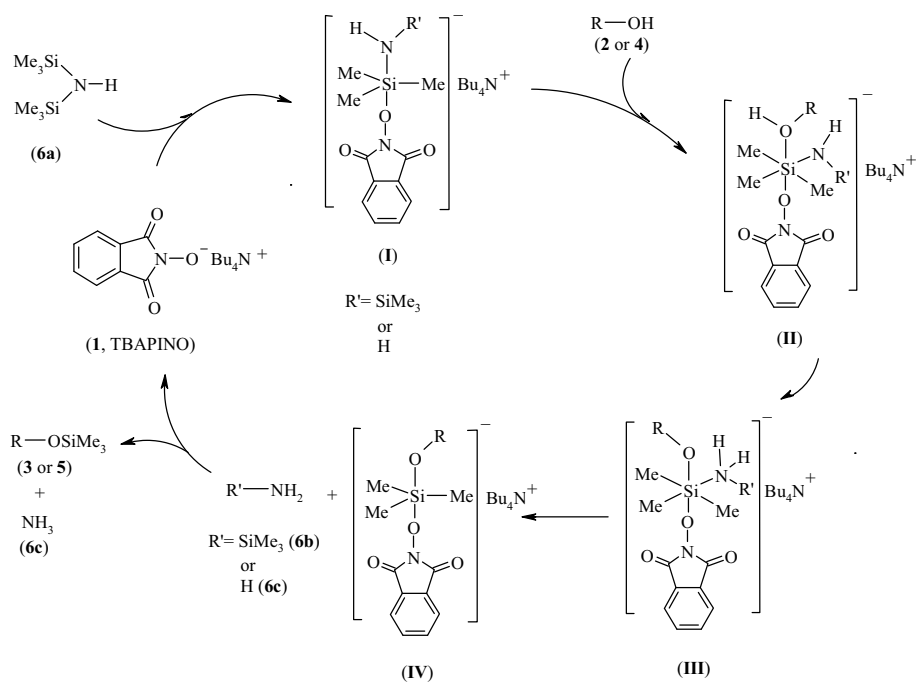

Scheme 3. Plausible mechanism for the trimethylsilylation of alcohol and phenols catalyzed by TBAPINO

Table 4. Comparison of some of the Results Obtained by Trimethylsilylation of Alcohols and Phenols with HMDS in the Presence of TBAPINO (1), with some of those Reported by Lanthanum Trichloride (LaCl$_3$, 2), Lithium Perchlorate (LiClO$_4$, 3), Trichloroisocyanuric Acid (TCCA, 4), 1,3-Dibromo-5,5-diethylbarbituric Acid (5), Tetrabutylammonium Bromide (TBAB, 6)

Entry	Substrate	Method [Equivalent of HMDS/TOF (h^{-1})]					
		1[a]	2[13]b	3[19]c	4[28]b	5[30]b	6[33]d
1	C$_6$H$_5$CH$_2$OH	0.55/147 0.55/47[e]	0.7/3.0	0.7/4.8	0.8/3.8	0.7/19	-
2	4-MeC$_6$H$_4$CH$_2$OH	0.55/196	-	-	0.8/6.2	-	-
3	4-ClC$_6$H$_4$CH$_2$OH	0.55/114	-	-	-	-	0.7/37
4	C$_6$H$_5$OH	0.55/46	-	0.7/5.8	0.8/6.4	-	-
5	1-Naphthol	0.55/43	0.7/3.2	0.7/4.8	-	-	-
6	(C$_6$H$_5$)$_2$CHOH	0.55/63	0.7/2.7	-	1.0/3.2	0.7/3.0	0.7/54
7	Cholesterol	0.55/23	-	-	-	-	-

[a]EtOAc as solvent under reflux conditions otherwise it has been noted. [b]CH$_2$Cl$_2$ as solvent. [c]Solvent-free conditions. [d]CH$_3$CN as solvent. [e]At room temperature.

through the pentacoordinated intermediate **IV**.

To illustrate the efficiency of the proposed method, Table 4 summarizes the comparison of the results obtained by our protocol with some of those reported in the literature [13,19,28,30,33].

CONCLUSIONS

The catalytic activity scope of tetrabutylammonium phthalimide-*N*-oxyl (TBAPINO), as a novel and powerful organocatalyst has been developed for the efficient and chemoselective trimethylsilylation of alcohols and phenols with hexamethyldisilazane (HMDS). The mild reaction conditions, low catalyst loading, low required HMDS for complete conversion of substrates, high to quantitative yield and simple removal of the catalyst from the reaction mixture requiring no subsequent purification by chromatographic methods illustrate some other attractive features of this protocol.

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