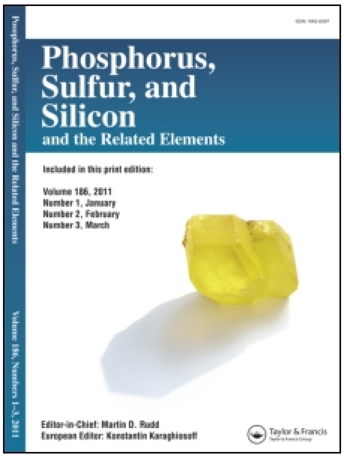

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Heterogeneous Thiocyanation of Benzylic Alcohols and Silyl and THP Ethers, and Deprotection of Silyl and THP-Ethers by $[PCl_{3-n}(SiO_2)_n]$ (Silphos)

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HETEROGENEOUS THIOCYANATION OF BENZYLIC ALCOHOLS AND SILYL AND THP ETHERS, AND DEPROTECTION OF SILYL AND THP-ETHERS BY [$PCl_{3-n}(SiO_2)_n$] (SILPHOS)

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Silicaphosphite (silphos), [$PCl_{3-n}(SiO_2)_n$], as a heterogeneous phosphorous compound, catalyzes the thiocyanation of benzylic alcohols and silyl and THP ethers in the presence of I_2 and NH_4SCN in refluxing CH_3CN. The produced silphos oxide byproduct can be easily separated by a simple filtration. Silphos is also used for the efficient and selective deprotection of silyl and THP-ethers to their corresponding alcohols.

KEYWORDS Alcohol; deprotection; silyl ether, silphos; thiocyanation; THP-ether

INTRODUCTION

The preparation of alkyl halides and thiocyanates from alcohols through the Mitsunobu reaction has been widely studied with tertiary phosphines.[1–3] The use of $PPh_3/Br_2/SCN^-$, phenylene alkylphosphite/Br_2/AgSCN[4], or in situ–generated $PPh_3(SCN)_2$[5] is well studied. The difficulty of removing the phosphine oxide byproduct is usually the major disadvantage encountered with these systems. Heterogeneous phosphorous-containing reagents such as diphenyl(2-pyridyl)phosphine,[6] (*p*-dimethylaminophenyl)-diphenyl phosphine,[7] 1,2-bis(diphenylphosphino)ethane (diphos),[8] tris[4-(1H,1perfluorooctyloxy-phenyl)-phosphine,[9] and 4-diphenyl phosphanyl benzoic acid 2-trimethylsilanylethylester (DPPBE),[10] or the use of crosslinked[11] or non-crosslinked polystyrene[12] and PEG-bound reagents[13] carrying a phosphine group have been applied to carry on the reactions under heterogeneous conditions for easy removal of the phosphine oxide byproduct.

Recently, we have introduced a cheap and easily prepared heterogeneous phosphite reagent, silphos [$PCl_{3-n}(SiO_2)_n$], and applied it in various synthetic transformations.[14] The use of silphos in these synthetic methods offers the possibility of easy removal of silphos oxide by filtration.

The ease of preparation and stability of tetrahydropyranyl (THP) and trimethylsilyl (TMS) groups under a variety of reaction conditions, and also their easy removal, offer

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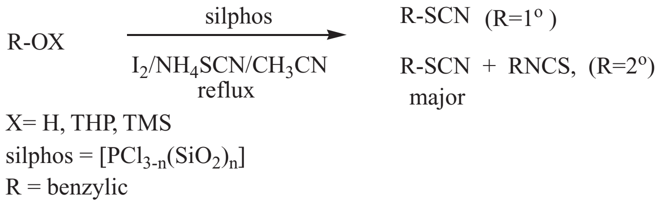
great applicability for their use in protection of hydroxyl groups in multistep synthesis. A wide range of catalysts and reagents have been used for deprotection of tetrahydropyranyl ethers and trimethylsilyl ethers.[15–19] In comparison, the reports on selective deprotection reactions and also deprotection thiocyanation are rare, and as far as we know, there is no report on selective thiocyanation of these compounds.[3,5a–b] In this article, we report an efficient and selective method for the conversion of benzylic alcohols and their trimethylsilyl and tetrahydropyranyl ethers to their corresponding thiocyanates or isothiocyanates by using silphos/I_2/NH_4SCN reagent system. We also report on the efficient use of silphos for deprotection of TMS- and THP-ethers and also highly selective deprotection of TMS-ethers to their corresponding hydroxyl compounds in the presence of THP-ethers.

RESULTS AND DISCUSSION

In order to avoid the problems of using homogeneous phosphorous compounds, and in continuation of our work on thiocyanation reactions,[3–5,19,20] we used the heterogeneous silica-based phosphorous compound (silphos) in conjunction with ammonium thiocyanate and iodine for thiocyanation of benzylic alcohols and their protected derivatives (Scheme 1).



Scheme 1

The use of Br_2 or compounds carrying electrophilic halogens, such as *N*-bromosuccinimide (NBS) or *N*-chloro succinimide (NCS), for this conversion was also studied. We first optimized the reaction conditions using 4-methoxybenzyl alcohol as a model compound. The optimized conditions for the reaction of silphos and ROH/I_2/NH_4SCN with stoichiometry of 1/1.2/2.8 are shown in Table I.

It was observed the reaction of 4-methoxybenzyl alcohol with silphos/I_2/NH_4SCN was completed after 45 min and produced 4-methoxybenzyl thiocyanate in quantitative

Table I Conversion of 4-methoxybenzyl alcohol (1.0 mmol) to 4-methoxy benzyl thiocyanate using silphos (1.2 g) in the presence of NH_4SCN (2.8 mmol) and different source of halogens (1.2 mmol) in refluxing acetonitrile

Entry	Halogen compounds	Conversion[a] %	Time
1	I_2	100	45 min
2	Br_2	10	30 h
3	NBS	5	30 h
4	NCS	0	30 h

[a] Conversion yield is based on GC analysis using *n*-octane as internal standard.

yield. The use of Br_2, NBS, and NCS instead of I_2 is not successful for this transformation. The results are shown in Table I.

When we applied the optimized reaction conditions to other alcohols, it was observed that this reagent system is more efficient for benzylic alcohols (Scheme 1, Table I), and the reaction of nonbenzylic alcohols and deactivated benzylic alcohols do not go to completion. Using this method, primary benzylalcohols are converted to their corresponding alkyl thiocyanates without the formation of any isothiocyanates, but as is the case with other reagent systems, the secondary ones give also some minor amounts of isothiocyanates as a side product. The results are shown in Table II.

We then studied the possibility of applying this selective reagent system to THP- and TMS-ethers. The results obtained from this study (Table II) show that the combination of silphos/I_2/NH_4SCN in the same stoichiometry as used for alcohols provides the most suitable reaction conditions for the quantitative conversion of only protected benzylic alcohols to their corresponding thiocyanates in refluxing acetonitrile.

The order of addition of reagents is also important. The mixture of silphos and I_2 is first prepared in acetonitrile and NH_4SCN is added to this mixture followed by the addition of alcohol or the protected alcohol.

We also prepared a wide variety of structurally different THP-[22] and TMS-ethers[23] according to the procedures in the literature and studied their deprotection to their corresponding alcohols using silphos $[(SiO_2)_n\ PCl_{3\text{-}n}]$. We optimized the conditions for the reaction of benzyltrimethylsilyl ethers and found that the reaction in the presence of silphos (1.2 g) and silyl ether (1 mmol) proceeds at room temperature in CH_3CN (5 mL). The desired alcohol was produced in high yield almost immediately (Table III). In order to show the effect of silphos on this reaction, we replaced silphos with the same amount of silica gel for the deprotection of benzyltrimethylsilyl ether in acetonitrile. This reaction, after 5 h, showed the formation of trace amounts of alcohol. In comparison with the reaction of silphos, which completes almost immediately, the efficient role of silphos

Table II Conversion of benzylalcohols, and silyl and THP ethers to thiocyanates or isothiocyanates

Entry	Substrate	Time	Conversion	RSCN/RNCS[a]	% Isolated yield[e]
1	$4\text{-}MeOC_6H_4CH_2OH$	45 min	100	100/0	90
2	$2\text{-}MeOC_6H_4CH_2OH$	2 h	100	100/0	92
3	$C_6H_5CH(OH)CH_3$	7 h	100	70/30[b]	85
4	$(C_6H_5)_2CHOH$	14 h	80	60/20	88
5	$2\text{-}MeC_6H_4CH_2OH$	12 h	90	100/0	90
6	$C_6H_5CH_2OH$	24 h	100	100/0	90
7	$C_6H_5CH_2CH_2OH$	35 h	40[c]	100/0	—
8	$CH_3(CH_2)_5CH(OH)CH_3$	35 h	30[c]	65/25[b]	—
9	$4\text{-}MeOC_6H_4CH_2OSi(Me)_3$	50 min	100	100/0	90
10	$C_6H_5CH(OSi(Me)_3)CH_3$	9 h	100	65/35[d]	85
11	$2\text{-}MeOC_6H_4CH_2OTHP$	10 h	100	100/0	88
12	$C_6H_5CH(OTHP)CH_3$	12 h	100	70/10[d]	82

[a] GC and NMR yield using *n*-octane as internal standard.

[b] Yield% of thiocyanates and isothiocyanates were determined by NMR analysis.

[c] Gc yield.

[d] The reaction was done with 2 equivalents of silphos.

[e] All the products are known compounds and were identified by comparison of their physical and spectral data with those of authentic samples.[1,2]

Table III Conversion of silyl ethers to alcohols after 2 min

$$\mathrm{ROSiMe_3} \xrightarrow[\mathrm{CH_3CN,\ r.t}]{\text{Silphos}} \mathrm{ROH}$$

Entry	R	Conversion (%)	Isolated yield (%)
1	*p*-Methoxybenzyl	100	90
2	benzyl	100	85
3	1-Phenyl-ethyl	100	90
4	4-Nitrobenzyl	100	90
5	4-Chlorobenzyl	100	92
6	1,1-Diphenylmethyl	100	88
7	2-Phenyl-ethyl	100	88
8	1-Adamantyl	100	95
9	2-Cyclohexylethyl	100	92
10	2-Octyl	100	90

is well demonstrated. The recyclability of silphos was also examined. In the reaction of benzyltrimethylsilyl ether, the catalyst was filtered and washed with ether and dried. After drying in a vacuum oven (40°C), it was reused for the same reaction. This process was repeated five times, and the recovered silphos was found to have almost the same reactivity.

We observed that deprotection of different THP-ethers with silphos as a heterogeneous reagent occurs in acetonitrile at reflux conditions. We optimized the conditions for cleavage of the THP-ethers to obtain the parent hydroxyl compounds. The results of the optimization reactions show that silphos (1.2 g) in refluxing acetonitrile (5 mL) converts benzyl tetrahydropyranyl ether (1.0 mmol) to benzyl alcohol (90%) after 40 min. In comparison to the silphos, SiO_2 is not effective for this deprotection. The results for the reactions of THP-ethers with this reagent showed that this system is suitable for the conversion of 1°, 2°, 3°, and benzylic THP-ethers to their corresponding alcohols (Table IV). Various types of benzylic THP-ethers with electron-donating and electron-withdrawing groups were converted to their corresponding alcohols in the presence of silphos with excellent yields at room temperature. Our studies showed that this method is also applicable for deprotection of nonbenzylic THP-ethers (Table IV).

Table IV Conversion of THP ethers to alcohols

$$\mathrm{ROTHP} \xrightarrow[\mathrm{CH_3CN,\ reflux}]{\text{Silphos}} \mathrm{ROH}$$

Entry	R	Time (min)	Conversion	Isolated yield (%)
1	4-Methoxybenzyl	40	100	90
2	Benzyl	100	100	88
3	1-Phenyl-ethyl	85	100	92
4	4-Nitrobenzyl	90	100	90
5	4-Chlorobenzyl	70	100	85
6	1,1-Diphenylmethyl	90	100	88
7	2-Phenyl-ethyl	80	100	85
8	1-Adamantyl	90	100	90
9	2-Cyclohexylethyl	100	100	92
10	2-Octyl	90	100	90

Table V Selective reactions of different binary mixtures of THP and silyl ethers

Entry	Substrate	Time	Conversion	% Isolate yield[a]
1	$PhCH_2OSiMe_3$	1 min	100	85
	$PhCH_2OTHP$		0	0
2	$4\text{-}MeOPhCH_2OSiMe_3$	1 min	100	90
	$4\text{-}MeOPhCH_2OSi(i\text{-}Pr)_3$		0	0
3	$4\text{-}MeOPhCH_2OSiMe_3$	1 min	100	92
	$4\text{-}MeOPhCH_2OSi(Me)_2t\text{-}Bu$		0	0
4	$PhCH_2OTHP$	100 min[b]	80	75
	$4\text{-}MeOPhCH_2OSi(i\text{-}Pr)_3$		20	7
5	$PhCH_2OTHP$	100 min[b]	100	88
	$PhCH_2OSi(Me)_2t\text{-}Bu$		0	0

[a] GC yield using *n*-octane as internal standard.
[b] Reflux condition.

In comparison with THP-ethers, the deprotection reactions of TMS-ethers with silphos were found to be much quicker, and occurred almost immediately to give their corresponding alcohols (Table V).

Deprotection reactions of silyl ethers with silphos were found to be highly selective in the presence of THP-ethers. In a binary mixture of benzylsilyl ethers and benzyl-THP-ethers, the silyl ethers were converted quantitatively to their corresponding alcohols, while the THP-ethers remained intact (Table V). Excellent selectivity was also observed for the conversion of TMS-ethers in the presence of bulky trialkylsilyl ethers such as *t*-butyldimethylsilyl (TBS) and triisopropylsilyl ethers (Table V). Similarly, this method showed excellent selectivity for the conversion of tTHP-ethers in the presence of a *t*-butyldimethylsilyl (TBS) and triisopropylsilyl ethers to the corresponding alcohols.

In addition to the selective nature of silphos for the conversion of only active benzylic alcohols and their protected derivatives to their corresponding thiocyanates, silphos is also very efficient for deprotection reactions. In Table VI, the present method for deprotection of trimethylsilyl ethers was compared with some of the reported methods in the literature.

In conclusion, the use of silphos provides a very selective method for thiocyanation of only active benzylic alcohols. In comparison, none of the reported methods for this transformation has shown any selectivity between alcohols. The use of silphos offers the possibility

Table VI Comparison of this method with some of the methods in the literature

Entry	Substrates	Reagent or catalyst[ref]	Condition	Time	Isolated yield%
1	$4\text{-}MeOC_6H_4CH_2OTMS$	Silphos	rt	2 min	90
2	$4\text{-}MeOC_6H_4CH_2OTMS$	[bmim]Cl[21]	r.t	40 min	95
3	$Ph_2CHOTMS$	Silphos	rt	2 min	88
4	$Ph_2CHOTMS$	[bmim]Cl[21]	rt	1.5 h	90
5	$Ph_2CHOTMS$	$Fe(NO_3)_3.9H_2O/Fe(HSO_4)_3$[22]	r.t	2 h	95
6	$4\text{-}NO_2C_6H_4CH_2OTMS$	Silphos	rt	2 min	90
7	$4\text{-}NO_2C_6H_4CH_2OTMS$	[bmim]Cl[21]	rt	2 h	90
8	$4\text{-}ClC_6H_4CH_2OTHP$	Silphos	reflux	70 min	85
9	$4\text{-}ClC_6H_4CH_2OTHP$	$Fe(NO_3)_3.9H_2O/Fe(HSO_4)_3$[22]	rt	3 h	90
10	$4\text{-}MeOC_6H_4CH_2OTHP$	Silphos	reflux	40 min	90
11	$4\text{-}MeOC_6H_4CH_2OTHP$	β-CD[23]	60°C	9 h	85

of efficient deprotection of TMS- and THP-ethers to their corresponding alcohols. In addition, the highly selective use of silphos for the high yielding deprotection of TMS-ethers in the presence of THP-ethers and in the presence of more bulky trialkylsilyl ethers can be considered as strong points in favor of using this compound. In addition, the ease of separation of silphos oxide in the thiocyanation reactions and silphos in the deprotection reactions can be regarded as another advantage of using this heterogeneous method.

EXPERIMENTAL

Chemicals were either prepared in our laboratories or were purchased from chemical companies. The purity determination of the products was accomplished by GC on a Shimadzu model GC-14A instrument or by TLC on silica gel polygram SIL G/UV 254 plates. The IR spectra were recorded on a Shimadzu FTIR-8300 spectrophotometer. The NMR spectra were recorded on a Bruker Avance DPX-250 MHz spectrometer. All products are known compounds, and their analytical and spectral data were compared with those of authentic samples.

Typical Procedure for the Conversion of 4-Methoxybenzyl Alcohol or 4-Methoxybenzyl TMS-Ether to 4-Methoxybenzyl Thiocyanate

To a flask containing a stirring mixture of silphos (1.2 g) and I_2 (0.3 g, 1.2 mmol) in refluxing dry acetonitrile (5 mL), NH_4SCN (0.21 g, 2.8 mmol) was added. Then 4-methoxybenzyl alcohol (0.13 g, 1 mmol) or 4-methoxybenzyl trimethylsilyl ether (0.21 g, 1 mmol) was added. Monitoring of the reaction by TLC and GC analysis showed the completion of the reaction after 45–50 min. After evaporation of the solvent on a rotary evaporator, the obtained solid phase was washed by diethyl ether (3 × 15 mL). The combined organic solution was washed with saturated solution of sodium thiosulfate (10 mL) to remove the unreacted iodine. The organic layer was separated and dried with anhydrous Na_2SO_4. After filtration, the solvent was evaporated under vacuum. The product was purified on a short column of silica gel using *n*-hexane-ethyl acetate (2:1) as eluent to give the pure 4-methoxybenzyl thiocyanate (90–92%). IR ($-$SCN) in CCl_4 2158 cm^{-1}; ^1H NMR ($CDCl_3$) δ (ppm) $=$ 3.2 (3H, s), 3.9 (2H, s), 7.1 (4H, m); ^{13}C NMR ($CDCl_3$) δ (ppm) $=$ 159.5, 132.8, 127.5, 115.9, 111.2, 38.8.

Typical Procedure for the Conversion of Benzyltetrahydropyranyl Ether or Benzyltrimethylsiliyl Ether to Benzyl Alcohol in the Presence of Silphos

Silphos (1.2 g) and benzyltetrahydropyranyl ether (0.19 g, 1 mmol) or benzyltrimethylsiliyl ether (0.18 g, 1 mmol) and dry acetonitrile (5 mL) were placed in a round bottom flask. The reaction benzyltetrahydropyranyl ether was refluxed for 40 min, and the reaction of benzyltrimethylsiliyl ether was stirred at room temperature for 1 min. TLC monitoring showed the completion of the reaction. The reaction mixture was filtered and washed by ethanol (50 mL). The solvent was evaporated on a rotary evaporator. Column chromatography of the crude mixture on silica-gel using *n*-hexane-ethyl acetate (3:1) as eluent gave pure benzyl alcohol in 88% (0.095 g) and 85% (0.092 g) yields, respectively.

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