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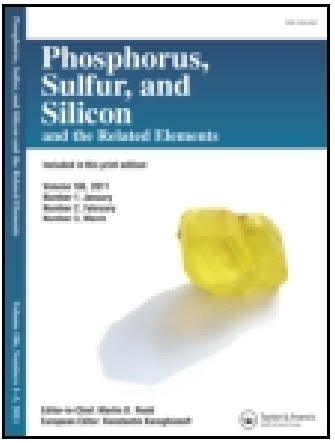

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Oxidative Deprotection Of Trimethylsilyl and Tetrahydropyranyl Ethers and Ethylene Acetals With Benzyltriphenylphosphonium Peroxomonosulfate in the Presence of Bismuth Chloride Under Non-Aqueous Conditions

A. R. Hajipour [a c], S. E. Mallakpour [a c], I. Mohammadpoor-baltork [b] & H. Adibi [a]

[a] Pharmaceutical Research Laboratory, College of Chemistry, Isfahan University, of Technology, Isfahan, 84156, IR, Iran

[b] Department of Chemistry, Isfahan University, Isfahan, 81744, IR, Iran

[c] E-mail:

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OXIDATIVE DEPROTECTION OF TRIMETHYLSILYL AND TETRAHYDROPYRANYL ETHERS AND ETHYLENE ACETALS WITH BENZYLTRIPHENYLPHOSPHONIUM PEROXOMONOSULFATE IN THE PRESENCE OF BISMUTH CHLORIDE UNDER NON-AQUEOUS CONDITIONS

A.R. HAJIPOUR[a*], S.E. MALLAKPOUR[a*], I. MOHAMMADPOOR-BALTORK[b] and H. ADIBI[a]

[a]*Pharmaceutical Research Laboratory, College of Chemistry, Isfahan University of Technology, Isfahan, 84156, IR, Iran and* [b]*Department of Chemistry, Isfahan University, Isfahan 81744, IR, Iran*

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Benzyltriphenylphosphonium peroxomonosulfate ($BnPh_3P^+HSO_5^-$) (1) is used as a new reagent for oxidative deprotection of trimethylsilyl and tetrahydropyranyl ethers and ethylene acetals to afford carbonyl compounds in refluxing acetonitrile in the presence of bismuth chloride.

Keywords: Deprotection; Oxidation; Oxone; Trimethylsilyl ethers; Tetrahydropyranyl ethers; Ethylene acetals

INTRODUCTION

The protection and deprotection of the hydroxy functional groups can play an essential role in synthetic strategy.[1] The transformation of alcohols to the corresponding trimethylsilyl ethers is a very common way to protect hydroxy groups.[1-3] In recent years a wide variety of methods have been

* Correspondence Author: Email: haji@cc.iut.ac.ir

reported for oxidative cleavage of trimethylsilyl ethers to the corresponding carbonyl compounds,[4–7] but some of the mentioned methods encounter drawbacks such as the requirement for aqueous reaction conditions, use of expensive reagents, long reaction times, low yields of the products and tedious work-up. Therefore, introduction of new methods and inexpensive reagents for such functional group transformations is still in demand. The tetrahydropyranyl (THP) group is one of the most frequently used groups to protect alcohols and phenols,[1,8] Owing to the outstanding stability of tetrahydropyranyl ether under a variety of reaction conditions, 3,4-dihydro-2H-pyran is still a reagent of choice for protection of the alcohol group in peptide, nucleotide and carbohydrate chemistry.[9] Since direct transformation of tetrahydropyranyl ethers to their carbonyl compounds is rare in the literature,[5,7,10–13] introduction of new methods is desirable. The carbonyl groups are one of the most encountered functionalities in organic chemistry and a great deal of synthetic work has been performed on the preparation of carbonyl derivatives such as cyclic acetals and ketals.[1] However, deprotection of acetals and ketals to the corresponding carbonyl compound is a useful transformation in organic synthesis.[13–18]

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RESULTS AND DISCUSSION

Oxone ($2KHSO_5.KHSO_4.K_2SO_4$) is an inexpensive, water-soluble and stable oxidizing reagent that is commercially available. This reagent is insoluble in non-aqueous solvents; therefore, the requirement of aqueous conditions had to be overcome. Very recently, we have introduced new reagents, which are able to oxidize different substrates under non-aqueous conditions.[19,20] In this paper we wish to report a novel and efficient method for oxidative deprotection of trimethylsilyl and tetrahydropyranyl ethers and ethylene acetals to the corresponding carbonyl compounds in refluxing acetonitrile in the presence of catalytic amount of bismuth chloride. Benzyltriphenylphosphonium peroxomonosulfate ($BnPh_3P^+HSO_5^-$) (**1**) in the presence of bismuth chloride is able to oxidize trimethylsilyl and tetrahydropyranyl ethers to the corresponding carbonyl compounds. This compound can also react as an efficient reagent in the presence of bismuth chloride in refluxing acetonitrile to transform ethylene acetals to the corresponding carbonyl compounds in high yield.

TABLE I Oxidative Deprotection of Trimethylsilyl and Tetrahydropyranyl Ethers with $BnPh_3P^+HSO_5^-/BiCl_3$ in refluxing CH_3CN

Entry[a]	*Substrate*	*Product*	*Time (min)*	*Yield (%)*[b]
1	$PhCH_2OTMS$	PCHO	45	90
2	$2\text{-}MeOC_6H_4CH_2OTMS$	$2\text{-}MeOC_6H_4CHO$	75	80
3	$3\text{-}MeOC_6H_4CH_2OTMS$	$3\text{-}MeOC_6H_4CHO$	45	90
4	$3\text{-}O_2NC_6H_4CH_2OTMS$	$3\text{-}O_2NC_6H_4CHO$	150	95
5	$2,5\text{-}(MeO)_2\ C_6H_3CH_2OTMS$	$2,5\text{-}(Me)_2C_6H_3CHO$	30	75
6	PhCH(Me)OTMS	PhCOMe	45	98
7	$4\text{-}ClC_6H_4CH(Me)OTMS$	$4\text{-}ClC_6H_4COMe$	60	99
8	$Ph_2CHOTMS$	Ph_2CO	60	95
9	$4\text{-}PhC_6H_4CH(Me)OTMS$	$4\text{-}PhC_6H_4COMe$	75	90
10	α-tetralolTMS	α-tetralone	45	95
11	$PhCH_2OTHP$	PhCHO	60	95
12	$2\text{-}MeOC_6H_4CH_2OTHP$	$2\text{-}MeOC_6H_4CHO$	90	70
13	$3\text{-}MeOC_6H_4CH_2OTHP$	$3\text{-}MeOC_6H_4CHO$ 30		88
14	$4\text{-}MeOC_6H_4CH_2OTHP$	$4\text{-}MeOC_6H_4CHO$	30	85
15	$3,4\text{-}(MeO)_2C_6H_3CH_2OTHP$	$3,4\text{-}(MeO)_2C_6H_3CHO$	45	80
16	$2,5\text{-}(MeO)_2C_6H_3CH_2OTHP$	$2,5\text{-}(MeO)_2C_6H_3CHO$	90	78
17	PhCH(Me)OTHP	PhCOMe	40	90
18	$4\text{-}ClC_6H_4CH(Me)OTHP$	$4\text{-}ClC_6H_4COMe$	60	85
19	$Ph_2CHOTHP$	Ph_2CO	90	94
20	α-tetralolTHP	α-tetralone	60	82

a. Substrate/Oxidant/$BiCl_3$ (1:1:0.4).
b. Yields based on the isolated products after column chromatography.

Benzyltriphenylphosphonium peroxomonosulfate ($BnPh_3P^+HSO_5^-$) (**1**) is a mild, efficient, stable and cheap reagent, which has been used for our studies under non-aqueous conditions. This reagent is a white powder, which is prepared by the dropwise addition of an aqueous solution of oxone, to an aqueous solution of benzyltriphenylphosphonium chloride at room temperature. Filtration and drying of the precipitates resulted in a white powder, which could be stored for months without losing its oxida-

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tion ability. The amounts of HSO_5^- in this reagent have been determined by an iodometric titration method.[21] The measurements are consistent with almost 99% by weight of active oxidizing agent. This reagent is quite soluble in methylene chloride, chloroform, acetone, and acetonitrile and insoluble in non-polar solvents such as carbon tetrachloride, n-hexane, and ether. At first, we decided to investigate the oxidative deprotection of benzyltrimethylsilyl ether in refluxing acetonitrile without catalyst with this reagent, and observed that in the absence of catalyst the reaction does not proceed at all. For this aim, the catalytic effects of several Lewis acids such as $ZnCl_2$, $FeCl_3$ $6H_2O$, $AlCl_3$ and $BiCl_3$were thoroughly investigated. Surprisingly, only $BiCl_3$ was found to be an effective catalyst during this investigation. The optimum molar ratio of bismuth chloride for this purpose was 0.4 molar ratio. Primary and secondary trimethylsilyl ethers were converted to their corresponding carbonyl compounds with benzyltriphenylphosphonium peroxomonosulfate in the presence of 0.4 molar ratio of bismuth chloride in refluxing acetonitrile in high yields and short reaction times (Table I). Interestingly, overoxidation of the products was not observed by this method. In order to evaluate the ability of this reagent, we also decided to convert tetrahydropyranyl ethers to their carbonyl compounds with benzyltriphenylphosphonium peroxomonosulfate. The reaction of primary and secondary tetrahydropyranyl ethers was completed in acetonitrile under refluxing conditions and the corresponding products were obtained in good to excellent yields (Table I).

TABLE II Deprotection of Ethylene Acetals with $BnPh_3P^+HSO_5^-/BiCl_3$ in Refluxing CH_3CN

Entry[a]	*Substrate*	*Product*	*Time (min)*	*Yield (%)*[b]
1	actophenoneethylene acetal	acetophenone	35	90
2	2-methoxybenzaldeethylene acetal	2-methoxybenzaldehyde	60	87
3	2-nitrobenzaldehydeethylene acetal	2-nitrobenzaldehyde	180	78
4	2-chloroactophenoneethylene acetal	2-chloroactophenone	75	80
5	α-tetralonecthylene acetal	α-tetralone	60	90
6	4-phenylacetopheno ethylene acetal	4-phenylacetophenone	150	80

a. Substrate/Oxidant/BiCl3 (1:1:0.4).
b. Yields based on the isolated products after purification with column chromatography.

Benzyltriphenylphosphonium peroxomonosulfate was also used to transform ethylene acetals to the corresponding carbonyl compounds in acetonitrile under refluxing conditions in the presence of 0.4 molar ratio of bismuth chloride in good to high yields (Table II). The cinnamaldehyde acetal as an allylic carbonyl compound derivative was not converted to its corresponding aldehyde properly with this reagent, and many by-products were obtained.

TABLE III Competitive Deprotection with $BnPh_3P^+HSO_5^-/BiCl_3$ in Refluxing CH_3CN

Entry[a]	*Substrate*	*Product*	*Time (min)*	*Yield (%)*[b]
1	3-nitrobenzaldehydeethylene acetal acetophenoneethylene acetal	3-nitrobenzaldehyde ace- tophenone	75	0 90
2	$3\text{-}MeOC_6H_4CH_2OTMS$ $4\text{-}ClC_6H_4CH(Me)OTHP$	$3\text{-}MeOC_6H_4CHO$ $4\text{-}ClC_6H_4CH(Me)OH$	60	50 50
3	$3\text{-}MeOC_6H_4CH_2OTHP$ $4\text{-}ClC_6H_4cH(Me)OTMS$	$3\text{-}MeOC_6H_4CH_2OH$ $4\text{-}ClC_6H_4COMe$	60	40 60
4	4-phenylacetophenoneethylene acetal $3\text{-}MeOC_6H_4CH_2OTMS$	4-phenylacetophenone	40	0
		$3\text{-}MeOC_6H_4CHO$		90
5	4-phenylacetophenoneethylene acetal $3\text{-}MeOC_6H_4CH_2OTHP$	4-phenylacetophenone $3\text{-}MeOC_6H_4CHO$	60	10 90
6	$PhCH_2OH$ PhCH(Me)OTMS	PhCHO PhCOMe	45	0 100
7	$PhCH_2OH$ PhCH(Me)OTHP	PhCHO PhCOMe	55	0 100
8	$PhCH_2OH$	PhCHO	60	20
	acetophenoneethylene acetal	PhCOMe		80
9	PHCH(Me)OH 2-methoxyacetophenoneethylene acetal	PhCOMe $2\text{-}MeOC_6H_4CHO$	60	10

a. Substrated/Oxidant $BiCl_3$ (1:1:0.4).
b. Evaluated by TLC analysis.

A noteworthy advantage of this reagent lies in its selectivity. We have tried competitive reactions between trimethylsilyl ethers and tetrahydropyranyl ethers or ethylene ketals, tetrahydropyranyl ethers and ethylene ketals and also between ethylene ketals and ethylene acetals. We have

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found that trimethylsilyl ethers are selectively oxidized in the presence tetrahydropyranyl ethers or ethylene ketals. Tetrahydropyranyl ethers were also selectively oxidized in the presence of ethylene ketals. We have also observed that ethylene ketals in the presence of ethylene acetals are selectively deprotected. All of the mentioned competitive reactions were carried out in the presence of bismuth chloride (0.4 molar ratio) in refluxing acetonitrile. We have also performed competitive oxidative reaction between alcohols and other compounds such as trimethylsilyl ethers, tetrahydropyranyl ethers and ethylene acetals with this reagent in the presence of 0.4 molar ratio of bismuth chloride in refluxing acetonitrile. We have found that alcohols in the presence of trimethylsily ethers or tetrahydropyranyl ethers are not oxidized at all. Ethylene acetals or ketals in the presence of alcohols selectively deprotected to their corresponding carbonyl compounds. Table III shows the experimental results.

In order to show the oxidative ability of this reagent we compared some of our results with some of those reported for silvet bromate[13] and 3-carboxypyridinum chlorochromate[5] with respect to their yields and the reaction time (Table IV). This reagent is superior to silver bromate in terms of selectivity, high yields, low cost, and purity of products and facile work-up.

TABLE IV Comparison of Oxidative Deprotection of some Trimethylsilyl and Tetrahydropyranyl Ethers with $BnPh_3P^+$ $HSO_5^-/BiCl_3$, 3-Carboxypyridinium Chlorochromate (CPCC)[5] and Silver Bromate[13]

Entry	*Substrate*	*Product*	*Yield % (h)*		
			Reagent (1)	*CPCC*	*$AgBrO_3$*
1	PhCH(Me)OTMS	PhCOMe	98(0.75)	93(1.15)	–
2	Ph_2CHOTMS	Ph_2CO	95(1)	80(2)	–
3	4-PhC_6H_4CH(Me)OTMS	4-PhC_6H_4COMe	90(1.25)	98(2)	–
4	PhCH(Me)OTHP	PhCOMe	90(0.66)	–	90(0.6)
5	3-$MeOC_6H_4CH_2$OTHP	3-$MeOC_6H_4$CHO	88(0.5)	93(0.1)	82(2.5)

In summary, in this study we introduced a new methodology for the oxidative cleavage of different types of protected functional groups under non-aqueous and homogeneous conditions. The stability, easy preparation,

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and high selectivity of the reagent, easy work-up, mild reaction conditions, and high yields of the products, make this method a novel and useful one relative to the present methodologies for oxidative deprotection of trimethylsilyl ethers, tetrahydropyranyl ethers and ethylene acetals to afford carbonyl compounds in refluxing acetonitrile in the presence of bismuth chloride.

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EXPERIMENTAL

General

Trimethylsilyl ethers, tetrahydropyranyl ethers and ethylene acetals were prepared according to described procedures.[3a,14,22,23] All of the yields refer to isolated products after column chromatography. All of the products were characterized by comparison of their spectral (IR, ^{1}H-NMR and TLC) and physical data (melting and boiling point) with those of authentic samples.[5,13,19]All ^{1}H-NMR spectra were recorded at 90 MHz in $CDCl_3$ and CCl_4 relative to TMS (0.00 ppm). Elemental analysis was performed by the Research Institute of Petrolium Industry, Tehran, I.R. Iran.

Preparation of Reagent (1)

A solution of benzyltriphenylphosphonium chloride (19.0 g, 49 mmol) in 100 ml of water was prepared, then oxone ($2KHSO_5.KHSO_4.K_2SO_4$, 30.06 g, 49 mmol) in water (100 ml) was added dropwise to the above solution and stirred for 1 hour at room temperature. The resulting precipitate was filtered and washed with cooled distilled water (50 ml), and dried in a desiccator under vacuum over calcium chloride to afford a white powder (21.7 g, 95% yield), which decomposed at 144–146 °C to a dark-brown material. ^{1}H-NMR: δ 7.90–6.85 (m, 20H), 4.7 (d, J = 25.6 Hz, $\underline{CH_2}$P). IR (KBr): 1298, 1269, 1098, 1060, 700, 658, 590 cm^{-1}. The white solid was then titrated three times as following procedure.[21]: To a 0.244 g sample was added 0.5 ml of glacial acetic acid and 1 ml of 10 % aqueous NaI. After dilution to 5 ml with THF, it was titrated with 5.20 ml of 0.1012 M solution of sodium sulfite to the yellow endpoint. The average of the three trials gave 99 % (24.24±0.6% by weight) of active oxidizing agent

(HSO_5^-). Anal Calcd for $C_{25}H_{23}O_5PS$: C, 64.37; H 4.97; S, 6.87%. Found: 64.20; H, 5.20; S. 6.60%.

Oxidative Deprotection of Trimethylsilyl and Tetrahydropyranyl Ethers with $BnPh_3P^+HSO_5^-/BiCl_3$

General Procedure

In a round-bottomed flask equipped with condenser and magnetic stirrer, a solution of trimethylsilyl ether or tetrahydropyranyl ether (1 mmol) and $BiCl_3$ (0.126 g, 0.4 mmol) in acetonitrile (5 ml) was prepared. Benzyltriphenylphosphonium peroxomonosulfate (0.466 g, 1 mmol) was added to the solution and refluxed for 30–150 minutes. The reaction progress was followed by TLC (eluent: cyclohexane/EtOAc, 8:2). The reaction mixture was fillered off and the solid material was washed with acctonitrile (15 ml). The filtrate was evaporated and the resulting crude material was purified by column chromatography on silica gel using a mixture of ethyl acetate/hexane (10:90) as eluent. Pure carbonyl compounds were obtained in 70–99% yields (Table I).

Oxidative Deprotection of Ethylene Acetals with $BnPh_3P^+HSO_5^-/BiCl_3$

General Procedure

In a round-bottomed flask, a solution of ethylene acetal or ketal (1 mmol) in CH_3CN (5 ml) was treated with $BnPh_3P^+HSO_5^-$(0.466 g, 1 mmol) and bismuth chloride (0.126 g, 0.4 mmol) and the reaction mixture was refluxed for 35–180 minutes. TLC (cyclohexane/EtOAc, 8:2) monitored the reaction progress. The reaction mixture was cooled to room temperature, and was then filtered off and washed with CH_3CN (15 ml). The filtrates were evaporated on a rotary evaporator and the resulting crude material was purified by column chromatography on silica gel with a mixture of hexane/ethyl acetate (90:10) as eluent to afford pure carbonyl compounds in 78–90% yields (Table II).

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