Antimony(V) Chloride as an Efficient Reagent for Deprotection of Methyl Ethers

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Received: 21.05.2013; Accepted after revision: 29.05.2013

Abstract: This paper proposes a new and efficient method for the deprotection of methyl ethers using antimony pentachloride at ambient temperature. The procedure described here is a facile and practical method for the removal of the methyl group from aryl and benzyl methyl ethers. High selectivity was observed for the removal of the methyl group from dimethoxyarenes. The notable advantages of this protocol are mild reaction conditions, high yields, and the facility of workup procedure. The mechanism of the ether cleavage is proposed to explain this new reaction.

Key words: demethylation, methyl ether, antimony pentachloride, selectivity, dimethoxyarenes

Ethers as a masked hydroxyl group are versatile intermediates of valuable compounds in organic chemistry from both academic and industrial perspectives,[1] so the preparation or deprotection of ether groups is an important organic transformation.

A useful method for the protection of hydroxyl groups in aliphatic and aromatic compounds is O-methylation with dimethyl sulfate (DMS),[2] methyl halides,[3] dimethyl carbonate (DMC),[4] and methanol in the presence of strong acid[5] or zeolite[6] as catalyst.

The ether groups have been traditionally considered as inert and highly stable to a variety of reagents and experimental conditions. Hence, deblocking of ethers is laborious and requires highly challenging transformation and deep study. Numerous cleavage methods are available,[7] such as using bases,[8] oxidative deprotection,[9] reductive cleavages,[10] and alkali metal agents.[11] Another well-documented ones are the use of microwave irradiation[12] and improving biological methods in this area.[13] The most practical and widely employed strategy is the deprotection of aryl methyl ethers using Lewis acids and strong acids. Examples include BBr_3,[14] the combination of $AlCl_3$/pyridine,[15] as well as $AlBr_3$/NaI,[16] $BeCl_2$,[17] $NiCl_2$/Zn,[18] ionic liquids (IL) such as 3-methylimidazolium bromohydrogenate (HmimBr–HBr),[19] pyridine–hydrochloride system,[20] and iodocyclohexane.[21]

However, some of these methods restricted by low reaction yields and high temperatures, which are not suitable for the synthesis of thermolabile compounds, result in undesired products because of the harsh reaction conditions. Therefore, more efficient methods are desired to deprotect methyl ethers under ambient conditions.

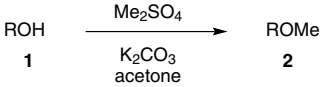
Antimony pentachloride is a commercially available and inexpensive reagent that exhibits a variety of interesting and useful properties. It has been successfully used as a standard Lewis acid[22] and a strong oxidant in organic synthesis.[23] This led researchers to develop this reagent in several organic process, such as O-desilylation reaction,[24] Friedel–Crafts alkylation,[25] polymerization,[26] electrophilic additions to alkenes,[27] aromatization of enamines,[28] and chlorination of organic compounds.[29]

We report herein a general method for the effective deprotection of methyl ethers under mild reaction conditions using antimony pentachloride as a high selective and efficient reagent.

To our knowledge, selective deprotection of ethers has scarcely been investigated. Demethylation of polymethoxyarene derivatives is a very attractive route for the preparation of fine chemicals and intermediates. A disappointingly high proportion of the prior works focuses on high-temperature conditions on which deprotections are relatively easy to promote.

We now wish to report our development of selective and room-temperature methodology with minimal waste streams and good yields. Reactions are possible with aryl methyl ethers, benzyl methyl ether, and dimethoxyarenes. This method is also superior to the synthesis of 2-methoxy-2′-hydroxy-1,1′-binaphtyl, monodentate BINOL monoether ligand.

A series of methoxy-substituted compounds were conveniently prepared by O-methylation of di- and monohydroxybenzene and naphthol derivatives **1b–j**, 1,1′-binaphthyl-2,2′-diol (**1k**, BINOL), and phenyl methanol (**1g**). The purification was performed according to the procedure described in the experimental section below without using column chromatography. Of all the protection systems tested, the most effective turned out to be the couple of potassium carbonate (base)/dimethyl sulfate (DMS) in anhydrous acetone at reflux conditions. Once the optimal reaction conditions were established, the cor-



Scheme 1 Synthesis of methyl ether derivatives

SYNLETT 2013, 24, 1702–1706
Advanced online publication: 16.07.2013
DOI: 10.1055/s-0033-1339310; Art ID: ST-2013-D0470-L

responding methyl ethers were successfully obtained in quantitative yields (Table 1, Scheme 1).

In order to find a suitable procedure for the selective demethylation, a model reaction of anisole (**2a**) was initially performed. In the presence of anisole, the solvent, temperature, molar ratio, and atmosphere conditions were examined as the common factors affecting the reaction. The results are summarized in Table 2.

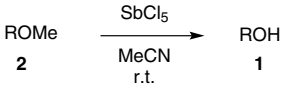
As for the solvent effect, the cleavage efficiency was highly solvent-dependent (Table 2, entries 1–5). The reaction was carried out using as low as 1.5 equivalents $SbCl_5$ in anhydrous tetrahydrofuran, toluene, dichloromethane, acetonitrile, and wet acetonitrile (5% w/v) at ambient temperature under inert atmosphere. In the presence of toluene and THF, low yields of 29% and 35% were produced with several byproducts within 48 and 24 hours, respectively. Since acetonitrile produced the best conversion and yield, it was chosen as the preferred solvent for the reaction. It is worth noting that the reaction consistently produced low yields under reflux conditions with the above-mentioned solvents.

The corresponding phenol was obtained in good yields at ambient temperature (Table 2, entries 5–8). The reaction continued with the dropwise addition of $SbCl_5$ to the stirred mixture of methyl ether and anhydrous acetonitrile at 0 °C under nitrogen atmosphere, and the mixed temperature became ambient. This method result in minimal waste streams and higher yields (Table 2, entries 5 and 6).

Table 2 shows that the reaction was not very sensitive to the amount of $SbCl_5$. Under the same conditions, the variation of the molar ratio of anisole to $SbCl_5$ (1:0.5, 1:1.5, 1:2, and 1:5) in acetonitrile produced different yields (65–92%).

The reaction under air was compared with that under an inert atmosphere (Table 2, entry 13 vs. entry 5) and indicates that air is responsible for lowering the yield. This is probably due to the fact that antimony pentachloride is a hygroscopic agent which reacts explosively with H_2O.

The above-described investigations prompted us to use $SbCl_5$ (1.5 equiv) in anhydrous acetonitrile at ambient temperature under nitrogen atmosphere as the optimized reaction conditions to probe the scope of deprotection of various types of methyl ethers (Scheme 2).



Scheme 2 Deprotection of methyl ether derivatives

As can be seen in Table 3, deprotection of various types of aryl methyl ethers is completed by $SbCl_5$ under desired reaction conditions with good to excellent yields. In addition, the reaction of methoxy methyl benzene **2g** in the presence of $SbCl_5$ gave the benzyl alcohol in good yield (85%). An important point in this method was the selective monodeprotection of dimethoxy aryl compounds.

Table 1 Methylation of Alcohol and Phenol Derivatives with Potassium Carbonate/Dimethyl Sulfate in Acetone

Entry	Substrate	Product	Time (h)	Yield (%)[a]
1	4-bromophenol **1b**	1-bromo-4-methoxybenzene **2b**	12	95
2	3-bromophenol **1c**	1-bromo-3-methoxybenzene **2c**	10	95
3	2-chlorophenol **1d**	1-chloro-2-methoxybenzene **2d**	10	96
4	1-naphthol **1e**	1-methoxynaphthalene **2e**	8	90
5	2-naphthol **1f**	2-methoxynaphthalene **2f**	7	97
6	phenyl methanol **1g**	1-(methoxymethyl)benzene **2g**	15	97
7	benzene 1,2-diol **1h**	1,2-dimethoxybenzene **2h**	24	90
8	benzene-1,3-diol **1i**	1,3-dimethoxybenzene **2i**	24	91
9	benzene-1,4-diol **1j**	1,4-dimethoxybenzene **2j**	24	92
10	(S)-1,1′-bi-2-naphthol **1k**	(S)-2,2′-dimethoxy-1,1′-binaphthyl **2k**	18	95

[a] Isolated yield.

Table 2 Optimization of the Reaction Conditions in the Demethylation of Anisole **2a** with $SbCl_5$[a]

Entry	$SbCl_5$ (equiv)	Solvent	Atmosphere	Temp (°C)	Time (h)	Yield (%)[c]
1	1.5	CH_2Cl_2	N_2	r.t.	16	65
2	1.5	toluene	N_2	r.t.	48	29[d]
3	1.5	THF	N_2	r.t.	24	35[d]
4	1.5	MeCN (5% H_2O)	N_2	r.t.	24	75
5	1.5	MeCN	N_2	r.t.	16	90
6	1.5	MeCN	N_2	r.t.[b]	16	82
7	1.5	MeCN	N_2	55	16	80
8	1.5	MeCN	N_2	30	19	70
9	0.5	MeCN	N_2	r.t.	16	65
10	1	MeCN	N_2	r.t.	16	75
11	2	MeCN	N_2	r.t.	16	90
12	5	MeCN	N_2	r.t.	16	92
13	1.5	MeCN	air	r.t.	16	75

[a] $SbCl_5$ was added dropwise at 0 °C.
[b] $SbCl_5$ was added dropwise at r.t.
[c] Isolated yield.
[d] Isolated yield by column chromatography.

1,1′-Bi-(2-naphthol) (**1k**, BINOL) derivatives, an axially chiral aromatic molecule, with C_2-symmetry have been found to be useful in various fields of chemistry, especially in stoichiometric and catalytic asymmetric reactions.[30] In experiments involving O-methylated (S)-BINOL (**2k**) which contained dimethoxy groups, this compound is selectively monodeprotected to provide the corresponding product, 2-methoxy-2′-hydroxy-1,1′-binaphtyl (**3k**, BINOL-Me), in excellent yields (Table 3, Scheme 3). The monodentate BINOL monoether ligand is well demonstrated for a variety of asymmetric reactions.[31]

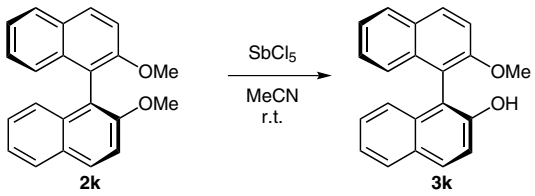
Table 3 Demethylation of Methyl Ethers Using $SbCl_5$[a]

Entry	Substrate	Product	Time (h)	Yield (%)[b]
1	anisole **2a**	phenol **1a**	16	90
2	1-bromo-4-methoxybenzene **2b**	4-bromophenol **1b**	24	80
3	1-bromo-3-methoxybenzene **2c**	3-bromophenol **1c**	24	78
4	1-chloro-2-methoxybenzene **2d**	2-chlorophenol **1d**	24	80
5	1-methoxynaphthalene **2e**	1-naphthol **1e**	48	90
6	2-methoxynaphthalene **2f**	2-naphthol **1f**	48	82
7	1-(methoxymethyl)benzene **2g**	phenylmethanol **1g**	30	85
8	1,2-dimethoxybenzene **2h**	2-methoxyphenol **3h**	24	95
9	1,3-dimethoxybenzene **2i**	3-methoxyphenol **3i**	24	88

Table 3 Demethylation of Methyl Ethers Using $SbCl_5$[a] (continued)

Entry	Substrate	Product	Time (h)	Yield (%)[b]
10	1,4-dimethoxybenzene **2j**	4-methoxyphenol **3j**	15	94
11	(S)-2,2′-dimethoxy-1,1′-binaphthyl **2k**	(S)-2-methoxy-2′-hydroxy-1,1′-binaphthyl **3k**	24	95
12	2-methoxyphenol **3h**	benzene-1,2-diol **1h**	72	–
13	3-methoxyphenol **3i**	benzene-1,3-diol **1i**	72	–
14	4-methoxyphenol **3j**	benzene-1,4-diol **1j**	48	47

[a] Reaction conditions: molar ratio substrate/$SbCl_5$ = 1:1.5; MeCN as solvent; r.t.; N_2 atmosphere.
[b] Isolated yield.

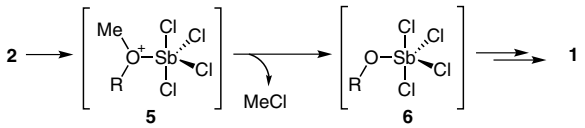


Scheme 3 Synthesis of monodentate BINOL

An interesting feature of this method was that the selectivity results were the same in the presence of higher amounts of $SbCl_5$ in the ratio of 2, 3, and 5 mmol to the substrate. Application of this new method to monodemethylation of dimethoxybenzene derivatives was also successful (Table 3).

It is worth noting that the demethylation of 2-methoxyphenol (**3h**) did not proceed after 72 hours. This observation can be ascribed to steric hindrance by the preferred coordination of $SbCl_5$ to the OH moiety of the substrate. It is also interesting to note that the 3-methoxyphenol (**3i**) produced similar results. It should be noted that 4-methoxyphenol (**3j**) was deprotected and changed to the corresponding dihydroxy compound under these conditions within 48 hours, and that no yield better than 47% was obtained (Table 3, entries 11–13). It is worth mentioning that the preferred coordination of $SbCl_5$ to the OH moiety of **3j** can cause the low yield but it has less steric hindrance in comparison with *ortho* and *meta* derivatives **3h** and **3i**, respectively.

The mechanistic illustration of these reactions prompted us to envision the formation of Sb(V) species of type **5** and then removal of MeCl to obtain type **6**. The Sb–O bond



Scheme 4 Proposed mechanism for the deprotection in the presence of $SbCl_5$

was dissociated after the addition of a saturated sodium hydrogen carbonate solution during the isolation step (Scheme 4).

In conclusion, $SbCl_5$ in acetonitrile was proposed as a very efficient demethylating agent in the deprotection reaction of methyl ethers. The generality of the procedure, high selectivity, and mild reaction conditions, good to excellent yields, and purification with a very simple workup procedure could make this method a very useful addition to the present reported processes. In view of exploiting the process, selective monodemethylation of poly aryl methyl ethers was established.

All chemicals were purchased from commercial suppliers and were distilled or recrystallized before use. NMR spectra (^1H NMR, ^{13}C NMR) were obtained using a 250 Bruker Avance instrument with the chemical shifts reported as δ (ppm) and the couplings expressed in hertz. The IR spectra were recorded using an FTIR Bruker Vector spectrophotometer with samples prepared as a KBr pellet or in a mull matrix. Microanalysis of the products was carried out on a CHN analyzer, PerkinElmer model 2400. Optical rotations were determined with a JASCO-D1P-370 digital polarimeter. Column chromatography was performed using Merck silica gel 60 (230–400 mesh). Merck silica gel 60 F254 plates (no. 5744) were used to monitor the reaction progress on TLC.

Preparation of Methyl Ether

A mixture of hydroxyl compound (1 mmol) and anhyd K_2CO_3 (0.21 g, 1.5 mmol) in anhyd acetone (2 mL) was stirred for 15 min. The dimethyl sulfate (1 mmol for monohydroxy compound and 1.5 mmol for dihydroxy aryl compound) was added to the mixture and refluxed to complete the reaction as monitored by TLC. Volatile compounds were removed under vacuum, and then CH_2Cl_2 (15 mL) was added to the reaction mixture under stirring for 5 min. The resulting mixture was filtered, and the solution was extracted with distilled H_2O (4 × 25 mL) and washed with brine solution. The combined organic layers were dried over Na_2SO_4 and were evaporated under reduced pressure to afford the corresponding methyl ether. The analytical spectral data were identical with literature references.[32b]

Demethylation of Methyl Ethers

The distilled $SbCl_5$ (0.2 mL, 1.5 mmol) was added dropwise to the stirred mixture of methyl ether (1 mmol) and anhyd MeCN (2.0 mL) at 0 °C under nitrogen atmosphere over 15 min. The temperature of

the resulting mixture was brought to the ambient temperature. Once the reaction was complete, the solvent was evaporated under reduced pressure. The complex was treated with sat. $NaHCO_3$ for 2 h (**CAUTION:** Antimony pentachloride reacts explosively with H_2O and this operation should be performed in a well-ventilated fume cupboard), and the mixture was extracted using CH_2Cl_2 (4 × 25 mL). The aqueous mixture was acidified to pH 2 by adding 1 M HCl. Then, the extraction with CH_2Cl_2 (4 × 25 mL), drying with Na_2SO_4, and evaporation under reduced pressure afforded the corresponding product (78–95% yield). Analytical and spectroscopic data were identical with literature references for known demethylated products.[32]

(*S*)-2-Methoxy-2′-hydroxy-1,1′-binaphthyl (3k)
Yield 95%; white solid; mp 83–85 °C; $[\alpha]_D^{27}$ –38.7 (*c* 0.67, THF). IR (KBr): 3431.9 cm^{-1}. ^1H NMR (250 MHz, $CDCl_3$): δ = 3.68 (1 H, s), 5.05 (1 H, s), 7.11 (1 H, d, *J* = 8.5 Hz), 7.18–7.41 (6 H, m), 7.47 (1 H, d, *J* = 9.0 Hz), 7.87 (1 H, d, *J* = 8.2 Hz), 7.90 (2 H, d, *J* = 8.0 Hz), 7.98 (1 H, d, *J* = 9.0 Hz). ^{13}C NMR (62.9 MHz, $CDCl_3$): δ = 53.17, 107.06, 110.52, 114.00, 119.75, 120.30, 120.46, 121.51, 122.54, 123.75, 124.17, 124.67, 125.48, 125.64, 125.72, 127.70, 130.27, 149.01, 151.23. Anal. Calcd for $C_{21}H_{16}O_2$: C,83.66; H, 5.72. Found: C, 83.77; H, 5.83.

Acknowledgment

We are grateful to the Research Council of Zanjan University and INSF for their financial support.

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