A Mild and Versatile Method for the Synthesis of Alkyl Ethers from Methoxymethyl Ethers and Application to the Preparation of Sterically Crowded Ethers

Yutaka Minamitsuji,[a] Atsuhisa Kawaguchi,[a] Ozora Kubo,[a] Yoshifumi Ueyama,[a] Tomohiro Maegawa,[a] and Hiromichi Fujioka[a,*]

[a] Graduate School of Pharmaceutical Sciences, Osaka University, 1–6 Yamada-oka, Suita, Osaka 565-0871, Japan
   Fax: (+81)-6-6879-8229; e-mail: fujioka@phs.osaka-u.ac.jp

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Abstract: A mild and divergent route for the synthesis of alkyl ethers from methoxymethyl (MOM) and methoxyethyl (ME) ether derivatives *via* pyridinium-type salt intermediates has been developed. The addition of organocuprates to the salts afforded the corresponding alkyl ethers, including highly crowded ones, in high yields even in the presence of acid- or base-sensitive functional groups.
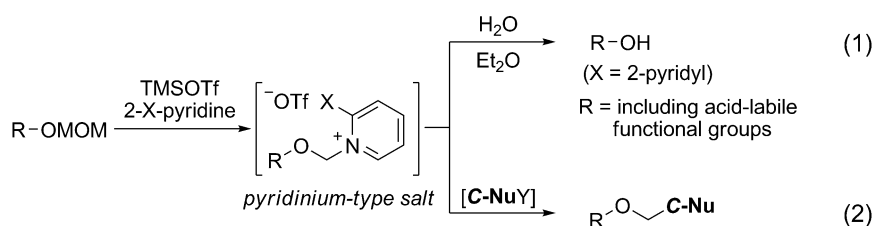
Keywords: alkyl ethers; alkylation; crowded ethers; nucleophilic substitution; organocuprates; pyridinium-type salts

Alkyl ethers are fundamentally important compounds in organic chemistry that are often chemically and metabolically stable.[1] Ether moieties are also found in many pharmaceutical agents and biologically active compounds.[2] Therefore, the development of efficient methods to prepare ethers is of general interest. The Williamson ether synthesis protocol, involving reactions between an alkoxide and alkyl halide, is perhaps the best known of the traditional methods used for this purpose.[3,4] However, this protocol has critical limitations associated with the use of strongly basic nucleophiles. Furthermore, Williamson ether syntheses are sensitive to steric hindrance in alkyl halide substrates and, as a result, cannot be applied to preparation of the crowded ethers. In order to overcome these drawbacks, various alternative methods for the preparation of alkyl ethers have been developed.[5,6] However, many of these methods often involve harsh conditions, have severe substrate limitations, or require the use of complicated procedures. Recently, a few alkyl ether-forming reactions have been described which utilize potassium organotrifluorobo-

rates as *C*-nucleophiles.[7,8] Bode and co-workers have developed an efficient method for the preparation of alkyl ethers that utilizes methoxymethyl (MOM) ethers or their derivatives, exemplified by hydroxamic acid-derived acetals.[7] However, sp^3 *C*-nucleophiles (e.g., alkyl groups) cannot be employed in this process. Molander and co-workers have devised a direct route for alkyl ether formation in heterocyclic substrates that involves treatment of alkoxytrifluoroborates in the presence of $Mn(OTf)_3$.[8] However, excess amounts of the Mn(III) reagent are needed and substrate limitations exist in this process. Therefore, the discovery of new, versatile and efficient procedures for alkyl ether synthesis remains a worthy goal in organic chemistry. Below, we describe the results of an investigation that led to the development of a mild and divergent route for the synthesis of alkyl ethers.

In a prior effort, we devised a mild and efficient method for the deprotection of MOM ethers.[9] This reaction [Scheme 1, Eq. (1)], which proceeds under mild conditions *via* a substituted pyridinium salt intermediate, is promoted by treatment of the ethers with TMSOTf and 2,2′-bipyridyl. The key feature of the reaction is its high chemoselectivity, which enables the survival of various functional groups including halogens, esters, amides, and acid-labile trityl (Tr) and *tert*-butyldimethylsilyl (TBS) ethers.

An analysis of the mechanistic route followed in the MOM ether deprotection reaction led to the proposal that appropriate *C*-nucleophiles might participate in reactions with the pyridinium salt intermediate and, if so, the overall process would serve as a mild and highly general method for the preparation of alkyl ethers [Scheme 1, Eq. (2)]. In order to explore this proposal, we treated MOM ether **1a** in CH_2Cl_2 with a combination of TMSOTf and 2,2′-bipyridyl under the conditions that were optimized in previous studies of the MOM ether deprotection pro-

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$$R-OMOM \xrightarrow[\text{2-X-pyridine}]{\text{TMSOTf}} \left[\begin{array}{c} {}^{-}OTf \quad X \\ R^{\diagdown O\diagdown}{}^{+}N \end{array} \right]$$

pyridinium-type salt

$$\xrightarrow[\text{Et}_2\text{O}]{\text{H}_2\text{O}} \quad R-OH \qquad (1)$$
(X = 2-pyridyl)
R = including acid-labile functional groups

$$\xrightarrow{[\textbf{C-Nu}Y]} \quad R^{\diagdown O\diagdown}\textbf{C-Nu} \qquad (2)$$

Scheme 1. The previously developed mild and chemoselective method for deprotection of MOM ethers [Eq. (1)] and the new strategy for synthesis of alkyl ethers [Eq. (2)].

Table 1. Optimization of reaction conditions for the alkyl ether-forming reaction.[a]

$$\text{Ph}\diagdown\diagup\diagdown\text{OMOM} \xrightarrow[\text{CH}_2\text{Cl}_2,\ 0\,°\text{C},\ 30\ \text{min}]{\substack{\text{TMSOTf (2.0 equiv.)} \\ \text{pyridine derivative (3.0 equiv.)}}} \xrightarrow[0\,°\text{C, time}]{\substack{\text{Ph}_2\text{CuMgBr} \\ \text{(3.0 equiv.)}}} \text{Ph}\diagdown\diagup\diagdown\text{O}\diagdown\text{Ph}$$

1a → **2aa**

Entry	Pyridine derivative	Reaction time [h]	Yield [%][b]
1	2,2′-bipyridyl	5	trace
2	2-methoxypyridine	24	n.r.[c]
3	2-(dimethylamino)pyridine	3	52
4	2-bromopyridine	1	58
5	2-chloropyridine	1	62
6	2-chloro-4-picoline	1	72
7[d]	2-chloro-4-picoline	1	92

[a] *Reaction conditions:* MOM ether **1a** was treated with pyridine derivative and TMSOTf in CH$_2$Cl$_2$ (0.2 M) at 0 °C for 30 min. The *C*-nucleophile was then added and the resulting mixture was stirred at 0 °C.
[b] Isolated yield.
[c] No reaction.
[d] THF was used as solvent instead of CH$_2$Cl$_2$.

cess. The *in situ* generated pyridinium salt was then reacted with 3.0 equivalents of Ph$_2$CuMgBr (from PhMgBr and stoichiometric CuI).[10] Indeed this two-step, one-pot reaction was found to generate a trace amount of the desired benzyl ether **2aa** (Table 1, entry 1). Studies probing the efficiencies of the ether-forming reaction taking place *via* salts derived from a variety of 2-substituted pyridines[11] showed that the electron-donating substituted 2-methoxypyridine did not promote alkyl ether formation and the process using 2-dimethylaminopyridine gave **2aa** in only a moderate yield (entries 2 and 3). In contrast, 2-bromo- and 2-chloropyridines, both of which contain weak electron-withdrawing groups, were suitable reagents for this reaction, which yielded **2aa** in better yields (entries 4 and 5). 2-Chloro-4-picoline was found to be the optimal pyridine for the alkyl ether-forming process (**2aa** formed in 72% yield, entry 6). Furthermore, replacement of CH$_2$Cl$_2$ by THF as solvent was found to lead to a further increase in the efficiency of this process (entry 7).[12,13]

The generality of the ether-forming reaction, carried out under the optimized conditions described above, was explored using several different cuprates (R$_2$CuMgX) derived from the corresponding Grignard reagents and CuI (Table 2). Reactions employing phenyl and *p*-tolyl cuprate were observed to produce the corresponding benzyl ethers in high yields (entries 1 and 2). The aryl organocuprate bearing an electron-withdrawing fluoride substituent also underwent this reaction efficiently to afford the corresponding ether (entry 3) and electron-donating group substituted (4-methoxy and 2,4-dimethoxy) aryl cuprates also reacted to form the respective ethers in excellent yields (entries 4 and 5). Moreover, the bulky mesityl, the further conjugated naphthyl, and a variety of heteroaromatic cuprates also reacted with the pyridinium salt intermediate to produce ethers in excellent yields (entries 6–10). The scope of this process was found to include vinyl, 2-methyl-1-propenyl, and phenylethynyl cuprates, which all added to the salt to form the corresponding products in moderate yields (entries 11–13). In order to extend the range of this process, we explored reactions of *sp*^{3} carbon-bonded organocuprates, such as ethyl, trimethylsilylmethyl, benzyl, isopropyl, cyclohexyl, and *tert*-butyl cuprates. The results demonstrated that, in each case, a reaction occurred to form the alkyl ether product in an excellent yield (entries 14–19). This observation contrasts with those made earlier, which showed that Bode's

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Table 2. Reactions of the pyridinium salt formed from MOM ether **1a** with various organocuprates.[a]

Ph⌒⌒OMOM **1a** → TMSOTf (2.0 equiv.) / 2-Cl-4-picoline (3.0 equiv.), THF, 0 °C, 30 min → R_2CuMgX (3.0 equiv.), 0 °C, time → Ph⌒⌒O⌒R **2a**

Entry	R	**2a**	Reaction time [h]	Yield [%][b]
1	Ph	**2aa**	1	92
2	p-MeC$_6$H$_4$	**2ab**	1	90
3	p-FC$_6$H$_4$	**2ac**	1	92
4	p-MeOC$_6$H$_4$	**2ad**	3	88
5	2,4-(MeO)$_2$C$_6$H$_3$	**2ae**	2	84
6	2,4,6-Me$_3$C$_6$H$_2$	**2af**	2	87
7	2-naphthyl	**2ag**	1	62
8	2-thienyl	**2ah**	1	94
9	2-benzothienyl	**2ai**	1	91
10	2-furyl	**2aj**	1	92
11	vinyl	**2ak**	2	73
12	(CH$_3$)$_2$C=CH−	**2al**	2	68
13	Ph-C≡C−	**2am**	2	68
14	Et	**2an**	3	75
15	TMSCH$_2$	**2ao**	2	82
16	Bn	**2ap**	2	73
17	i-Pr	**2aq**	1	81
18	cyclohexyl	**2ar**	2	75
19	t-Bu	**2as**	1	90

[a] *Reaction conditions:* MOM ether **1a** was treated with 2-chloro-4-picoline (3.0 equiv.) and TMSOTf (2.0 equiv.) in THF (0.2 M) at 0 °C for 30 min. Organocuprate (3.0 equiv.) was then added and the resulting mixture was stirred at 0 °C.
[b] Isolated yield.

method could not be applied to sp^3 C-nucleophiles. It should be emphasized that bulky *tert*-butyl ethers that are typically difficult to generate by using other methods could be prepared in excellent yields using the organocuprate-pyridinium salt protocol (entry 19).

Further investigations revealed that various types of MOM ethers served as starting materials in the new ether-forming process (Table 3). For example, reaction of (t-Bu)$_2$CuMgCl with the terminal olefin containing MOM ether **1b**-derived salt took place smoothly to produce the corresponding ether product **2b** (entry 2). The results of this effort also showed that the process displayed a high chemoselectivity in that a host of functional groups, such as methyl ester, acetoxy (AcO), benzoyloxy (BzO), benzyloxy (BnO), *tert*-butyldimethylsilyloxy (TBSO), and trityloxy (TrO) groups, were unreactive under the reaction conditions (entries 3–8). It is especially noteworthy that base-labile ester moieties were not affected in this process (entries 3–5) because they are typically sensitive to conditions employed in the Williamson ether synthesis protocol. Furthermore, Lewis and Brønsted acid sensitive TBS and Tr ethers tolerated the conditions used in new ether-forming reaction (entries 7 and 8). Finally, this study showed that the method could be used to prepare the bulky ethers **2i–k**, starting with the 1-adamantanol derivative **1i** and secondary or primary MOM ethers, **1j** or **1k**, respectively (entries 9–11).

The new ether-forming process was next probed in context of more crowded α,α′-disubstituted and multi-substituted ethers, which are difficult to synthesize by using the Williamson ether synthesis method. For example, the highly substituted ether **4a**, which could not be generated using the classical method owing to steric hindrance encountered in reactions between the appropriate alkoxide and halides or triflates [Scheme 2, Eq. (4)], was efficiently generated by reaction of the methoxyethyl (ME) ether **3a** utilizing 2,4,6-collidine in place of 2-chloro-4-picoline followed by treatment with (t-Bu)$_2$CuMgCl [Scheme 2, Eq. (3)].[14]

Table 4 contains a summary of results obtained in the effort of exploring applications of the new method to the synthesis of several crowded ethers. In all processes, the desired alkyl ethers **4** were obtained in excellent yields. In addition, *tert*-butyl cuprate could also be used in these processes to generate the desired alkyl ethers (entries 1, 4, and 5).[15]

Further investigations revealed that catalytic amounts of CuI were as effective as stoichiometric amounts in promoting reactions that afforded alkyl ethers **2c**, **2h**, and **4c** in high yields, even in cases (e.g., **2c**, **2h**) where methyl ether and trityl ether moieties
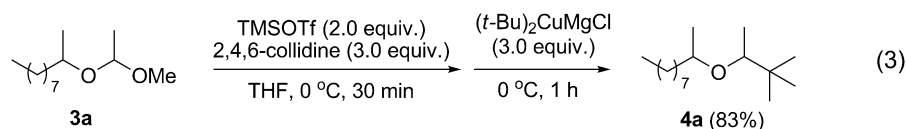
Table 3. Reactions of various MOM ethers **1** with (*t*-Bu)$_2$CuMgCl.[a]

$$R-OMOM \xrightarrow[\text{THF, 0 °C, 30 min}]{\substack{\text{TMSOTf (2.0 equiv.)} \\ \text{2-Cl-4-picoline (3.0 equiv.)}}} \xrightarrow[\text{0 °C, 1–3 h}]{\substack{(t\text{-Bu})_2\text{CuMgCl} \\ \text{(3.0 equiv.)}}} R^{O}\diagdown$$

1   **2**

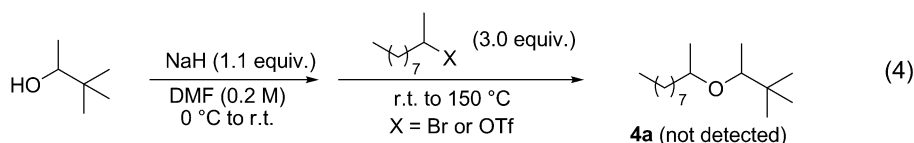| Entry	MOM ether (**1**)		Alkyl ether (**2**)		Yield [%][b]
1	**1a**	Ph⌒⌒OMOM	**2as**	Ph⌒⌒O⌒	90
2	**1b**	⌒⌒⌒₈OMOM	**2b**	⌒⌒⌒₈O⌒	90
3	**1c**	MeO(C=O)⌒₈OMOM	**2c**	MeO(C=O)⌒₈O⌒	93
4	**1d**	RO⌒₉OMOM, R=Ac	**2d**	RO⌒₉O⌒, R=Ac	89
5	**1e**	R=Bz	**2e**	R=Bz	90
6	**1f**	R=Bn	**2f**	R=Bn	86
7	**1g**	R=TBS	**2g**	R=TBS	81
8	**1h**	R=Tr	**2h**	R=Tr	85
9	**1i**	(adamantyl)OMOM	**2i**	(adamantyl)O⌒	81
10	**1j**	⌒⌒₇OMOM	**2j**	⌒⌒₇O⌒	82
11	**1k**	⌒⌒₈OMOM	**2k**	⌒⌒₈O⌒	81

[a] *Reaction conditions:* MOM ethers **1** were treated with 2-chloro-4-picoline (3.0 equiv.) and TMSOTf (2.0 equiv.) in THF (0.2 M) at 0 °C for 30 min. (*t*-Bu)$_2$CuMgCl (3.0 equiv.) was then added and the resulting mixture was stirred at 0 °C.
[b] Isolated yield.

i) Our method

$$\underset{\textbf{3a}}{⌒⌒_7\text{O}\text{CH(OMe)}} \xrightarrow[\text{THF, 0 °C, 30 min}]{\substack{\text{TMSOTf (2.0 equiv.)} \\ \text{2,4,6-collidine (3.0 equiv.)}}} \xrightarrow[\text{0 °C, 1 h}]{\substack{(t\text{-Bu})_2\text{CuMgCl} \\ \text{(3.0 equiv.)}}} \underset{\textbf{4a (83\%)}}{⌒⌒_7\text{O}C(\text{CH}_3)_3} \quad (3)$$

ii) Usual method (Williamson ether synthesis)

$$\underset{}{\text{HO}-C(\text{CH}_3)_3} \xrightarrow[\substack{\text{DMF (0.2 M)} \\ \text{0 °C to r.t.}}]{\text{NaH (1.1 equiv.)}} \xrightarrow[\substack{\text{r.t. to 150 °C} \\ X = \text{Br or OTf}}]{⌒⌒_7 X \;(3.0 \text{ equiv.})} \underset{\textbf{4a (not detected)}}{⌒⌒_7\text{O}C(\text{CH}_3)_3} \quad (4)$$
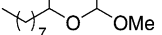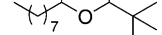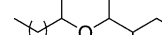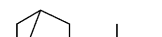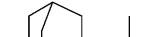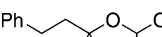
Scheme 2. Synthesis of crowded ether **4a** using the new method and the Williamson ether synthesis protocol.

were present in the substrates. Moreover, utilization of substrate **1c** on a gram-scale was successful in this reaction to afford the corresponding alkyl ether **2c** in high yield (Scheme 3).

In summary, the current work has led to the development of a mild and versatile method for the synthesis of alkyl ethers. The observations made in this study show that reactions of MOM and ME ethers,

proceeding through addition of organocuprate to the corresponding pyridinium salt, take place efficiently to produce sterically crowded alkyl ethers bearing various functional groups. Significantly, these ether targets could not be generated using the classical Williamson ether synthesis or the Bode or Molander protocols.
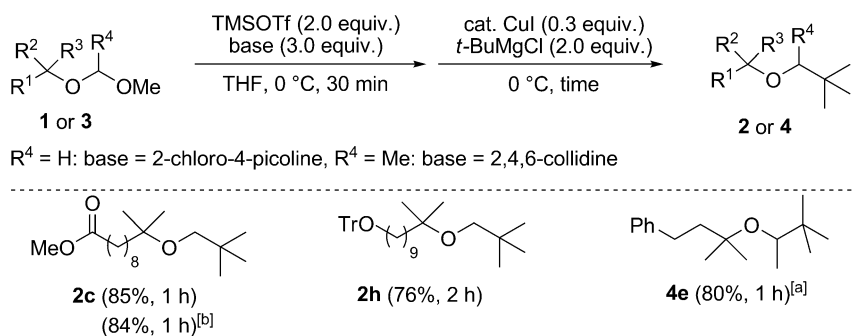
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Table 4. Synthesis of crowded ethers **4** using several ME ethers **3** and organocuprates.[a]
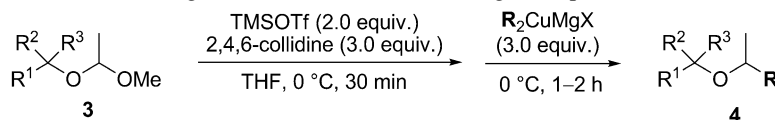
Entry	ME ether (**3**)		R	Alkyl ether (**4**)		Yield [%][b]
1	**3a**		*t*-Bu	**4a**		83
2	**3a**		Cy	**4b**		84
3	**3a**		Ph	**4c**		88
4	**3b**		*t*-Bu	**4d**		83
5[c]	**3c**		*t*-Bu	**4e**		84

[a] *Reaction conditions:* acetals **3** were treated with 2,4,6-collidine (3.0 equiv.) and TMSOTf (2.0 equiv.) in THF (0.2 M) at 0 °C for 30 min. Organocuprate (3.0 equiv.) was then added and the resulting mixture was stirred at 0 °C.
[b] Isolated yield.
[c] The reaction was carried out at −30 °C to −20 °C.

R^4 = H: base = 2-chloro-4-picoline, R^4 = Me: base = 2,4,6-collidine

2c (85%, 1 h)
(84%, 1 h)[b]

2h (76%, 2 h)

4e (80%, 1 h)[a]

[a] The reaction was carried out at −30 °C to −20 °C.
[b] Gram-scale preparation: 1.1 g of **1c** were used.

Scheme 3. Alkyl ether-forming reactions by use of a catalytic amount of CuI.

Experimental Section

Representative Procedure for the Conversion of MOM Ether 1a to Ether 2aa

TMSOTf (95 μL, 0.524 mmol) was added dropwise to a solution of MOM ether **1a** (54.5 mg, 0.262 mmol) and 2-chloro-4-picoline (86 μL, 0.786 mmol) in THF (1.2 mL, 0.2 M) at 0 °C under argon. The reaction mixture was stirred at 0 °C for 30 min, at which time **1a** had disappeared (by TLC). A solution of Ph₂CuMgBr, prepared by reaction of phenylmagnesium bromide (1.10 M in THF, 1.43 mL, 1.57 mmol) and

CuI (149.7 mg, 0.786 mmol) in THF (2.8 mL), was added to the mixture by cannula, and the resulting mixture was vigorously stirred at 0 °C until all polar components had disappeared by TLC. The reaction mixture was then diluted with saturated aqueous NH₄Cl, stirred for 20 min at 0 °C, and extracted with CH₂Cl₂. The combined organic layer was washed with brine, dried over Na₂SO₄, and concentrated under vacuum, giving a residue that was subjected to flash SiO₂ column chromatography (*n*-hexane/benzene = 1/1) to give alkyl ether **2aa** as a colorless oil; yield: 61.0 mg (92%). See the Supporting Information for characterization details.

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Acknowledgements

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[11] In the deprotection of MOM ethers, the salt from 2-substituted pyridines was subjected to hydrolysis, but the salt from 2,4,6-collidine did not undergo the hydrolysis, see ref.[9]

[12] PhLi or PhMgBr caused decomposition of pyridinium salt intermediate and no reaction occurred under treatment with Ph_2Zn.

[13] As described later (Scheme 3), the reaction with a catalytic amount of CuI produced an about 10% lower yield than the stoichiometric reaction. Therefore, we decided to use the stoichiometric reaction as our optimized conditions. Reduction of organocuprate also decreased the yield of **2aa** (89% with 2.0 equiv. of $Ph_2CuMgBr$; 83% with 1.5 equiv. of $Ph_2CuMgBr$).

[14] In our previous work, we have disclosed that 2,4,6-collidine was an efficient base in the mild deprotection of tetrahydropyranyl (THP) ethers, see: a) H. Fujioka, T. Okitsu, T. Ohnaka, Y. Sawama, O. Kubo, K. Okamoto, Y. Kita, *Adv. Synth. Catal.* **2007**, *349*, 636–646; b) H. Fujioka, T. Ohnaka, T. Okitsu, O. Kubo, K. Okamoto, Y. Sawama, Y. Kita, *Heterocycles* **2007**, *72*, 529–540; c) H. Fujioka, O. Kubo, K. Okamoto, K. Senami, T. Okitsu, T. Ohnaka, Y. Sawama, Y. Kita, *Heterocycles* **2009**, *77*, 1089–1103.

[15] When the reaction of **3c** was conducted at 0°C, β-elimination occurred as a side reaction and a decreased yield of **4e** (62%) was obtained.

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