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ORIGINAL PAPER

Microwave-assisted rapid and efficient deprotection and direct esterification and silylation of MOM and EOM ethers catalyzed by [Hmim][HSO_4] as a Brønsted acidic ionic liquid

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Abstract 1-Methylimidazolium hydrogensulfate, [Hmim][HSO_4], a Brønsted acidic room temperature ionic liquid, is used as a catalyst and reaction medium for facile and eco-friendly deprotection of methoxymethyl (MOM) and ethoxymethyl (EOM) ethers to their corresponding alcohols under thermal conditions (Δ) and microwave irradiation (MW). Furthermore, one-pot interconversion to the respective acetates and trimethylsilyl (TMS) ethers was also achieved.

Keywords [Hmim][HSO_4] · Acetate · Silyl ether · Microwave irradiation

Introduction

The protection of hydroxyl groups is often a necessary step in organic synthesis, especially in the total synthesis of natural products and multi-functional organic compounds [1]. Among them, methoxymethyl (MOM) and ethoxymethyl (EOM) ethers are commonly used as protected forms of alcohols due to their easy access and high stability under both basic and acidic conditions [2]. Furthermore, the deprotection of these ethers to the corresponding alcohols also plays an important role in organic synthesis. Many catalysts and reagents, such as HCl [3], BBr_3 [4], pyridinium *p*-toluenesulfonate [5], silica-supported sodium hydrogen sulfate [6], CBr_4/PPh_3 [7], $Sc(OTf)_3$ [8], Ce$(OTf)_4$ [9], $Bi(OTf)_3$ [10], $CeCl_3$ [11], Montmorillonite K-10 [12], $ZrCl_4$ [13], $ZnBr_2$ [14], and catechol boron bromide [15] have been reported for deprotection of MOM or EOM ethers.

Direct interconversion of functional groups plays a critical role in successful synthesis of multi-functional complex molecules. Esters and trimethylsilyl ethers are the most versatile protected forms of hydroxyl groups, due to their reasonably better stability to the acidic media widely utilized in organic synthesis [1, 2]. In some cases, besides simple deprotection, it becomes desirable to convert ether into ester protecting groups in a one-pot synthesis.

Design of organic synthesis in ionic liquids (ILs), particularly those based on the imidazolium cation, has attracted much attention in recent years. Some of the unique physical and chemical properties of ILs—such as high thermal stability, non-measurable vapor pressure, recyclability, ability to dissolve a large range of organic, inorganic, and organometallic compounds, increasing reaction rate, selectivity, and tendency to immobilize starting materials and catalyst—make them an attractive substitute to various volatile organic compounds [16, 17]. In fact, room temperature IL-catalyzed reactions provide excellent results with higher selectivity for the required products. In addition, ionic liquids have been demonstrated to couple very effectively with microwaves through an ionic conduction mechanism [18]. Also small amounts of ionic liquids can be used in order to increase temperature of reactions noticeably in microwave-assisted reactions. However, moisture sensitivity and decomposition of some ionic liquids under normal atmospheric conditions are two major drawbacks of ILs which limit their practical use [19]. On the other hand, the high cost of most conventional room

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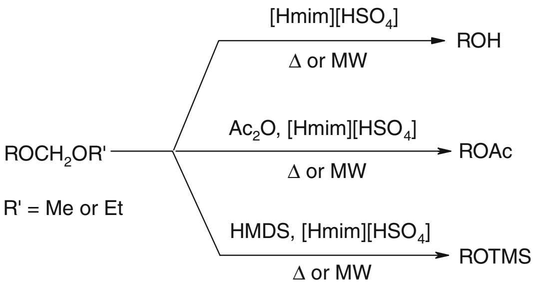

Scheme 1

temperature ionic liquids and apprehension about their toxicity have led us to explore the use of more benign salts as practical alternatives. Recently, it was reported that 1-methylimidazolium hydrogensulfate, [Hmim][HSO_4], a powerful Brønsted acidic room temperature ionic liquid, is relatively non-toxic, readily available at low cost, and fairly stable to water, unlike some ionic liquids (e.g., [bpy][$AlCl_4$]) which decompose readily in aqueous media [20]. These results in combination with our recent examination to design new synthetic methodologies [21, 22], especially in room temperature ILs [23, 24], led us to develop a simple and efficient procedure for deprotection of MOM or EOM ethers and their interconversion to the corresponding acetates and TMS ethers in the presence of [Hmim][HSO_4] as a Brønsted acidic room temperature ionic liquid (Scheme 1).

Results and discussion

First, the amount of catalyst and microwave power was optimized in the deprotection of benzyl methoxymethyl ether (Tables 1, 2). The results showed that the best yield was obtained with 1 mmol of benzyl methoxymethyl ether and 0.5 mmol of [Hmim][HSO_4] under microwave irradiation conditions at 150 W (120 °C, method A). Under the optimized conditions, a wide range of substituted and structurally diverse benzylic and aliphatic MOM and EOM ethers were deprotected to the corresponding alcohols in high to excellent yields and in very short reaction times (Table 3). Interestingly, in compounds containing both MOM or EOM ether, and benzyl or methyl ether, only MOM or EOM ether was deprotected and benzyl and methyl ethers remained intact during the reaction (Table 3, entries 3–6). These results clearly show that MOM and EOM ethers are more reactive than benzyl and methyl ethers under the reaction conditions studied.

Table 1 Optimization of ionic liquid amount for the deprotection of benzyl methoxymethyl ether

Entry	Amount of IL (mmol)	Yield (%)[a]	MW power (W)	*T* (°C)
1	0.2	<10	150	87
2	0.3	34	150	98
3	0.4	77	150	111
4	0.5	90	150	120
5	0.6	90	150	135

[a] Isolated yield

Table 2 Optimization of MW power for deprotection of benzyl methoxymethyl ether

Entry	MW power (W)	Yield (%)[a]	*T* (°C)
1	100	69	67
2	125	82	99
3	145	88	110
4	150	92	120
5	175	92	151

Reaction conditions: benzyl methoxymethyl ether (1 mmol) and ionic liquid (0.5 mmol)

[a] Isolated yield

For examination of the effect of microwave irradiation on this transformation, all reactions were performed under traditional thermal conditions (method B, Table 3). As illustrated in Table 4, the highest yields and lowest reaction times under thermal conditions were obtained at 50 °C.

The recovery and reusability of the catalyst, which is important from a cost and efficiency point of view, was also investigated in order to develop a ‘green’ protocol. The results showed that in the microwave-assisted reaction (method A) the catalytic activity of the ionic liquid decreased, but in the reaction under traditional conditions (method B) [Hmim][HSO_4] can be reused five times with only small loss of its activity (Table 5).

In order to demonstrate the chemoselectivity of the presented method, a set of competitive reactions was conducted between primary, secondary, or tertiary alkoxymethyl ether of alcohols and phenols (Table 6). The results indicated that the present protocol is potentially applicable for the chemoselective conversion of alkoxymethyl ethers of primary and secondary alcohols in the presence of ethers of tertiary alcohols and phenols. On the other hand, the competitive deprotection of alkoxymethyl ethers in the presence of acetates and TMS ethers were also investigated. The results summarized in Table 7 show that alkoxymethyl ethers are deprotected selectively in the presence of acetates and TMS ethers.

To develop the generality of this protocol, we investigated the one-pot conversion of these ethers into their acetates or TMS ethers with acetic anhydride and hexamethyldisilazide (HMDS), under microwave irradiation (150 W, 120 °C) or thermal conditions (50 °C) (Scheme 1).

Table 3 Deprotection of MOM and EOM ethers in the presence of [Hmim][HSO_4] under microwave irradiation (method A) and traditional method (method B)

Entry	R	MOM ether				EOM ether			
		MW irradiation (method A)		Traditional method (method B)		MW irradiation (method A)		Traditional method (method B)	
		Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]
1	$C_6H_5CH_2$	3	92	60	91	3	92	60	90
2	4-$(CH_3)_3CC_6H_4CH_2$	2.5	96	45	95	2	92	45	93
3	4-$PhCH_2OC_6H_4CH_2$	2.5	91	60	89	3	91	75	87
4	4-$CH_3OC_6H_4CH_2$	3	93	60	91	3	91	75	92
5	3-$CH_3OC_6H_4CH_2$	3	93	75	90	3	91	75	92
6	3-CH_3O-4-(HO) $C_6H_3CH_2$	1.5	95	75	90	2	93	75	91
7	2-$HOC_6H_4CH_2$	2.5	90	75	89	2	94	60	91
8	2-$ClC_6H_4CH_2$	1.5	92	45	90	1.5	94	45	93
9	4-$ClC_6H_4CH_2$	1	97	45	95	1	95	45	95
10	2,4-$Cl_2C_6H_3CH_2$	1	97	45	90	1	93	30	91
11	4-$BrC_6H_4CH_2$	1	96	45	92	1	95	45	90
12	2-$BrC_6H_4CH_2$	1.5	89	45	91	1	89	45	88
13	4-$NO_2C_6H_4CH_2$	5.5	83	90	81	5	89	90	83
14	$(C_6H_5)_2CH$	5	92	120	87	6	90	120	84
15	$C_6H_5CH_2CH_2$	1.5	95	45	94	2	94	45	91
16	$C_6H_5CHCH_3$	4	88	105	90	4.5	88	90	90
17	$CH_3(CH_2)_6CH_2$	2	91	45	88	2	93	45	89
18	(+)-Menthyl	7	89	105	86	6	83	105	84
19	2-Adamantyl	6	85	120	81	6.5	84	135	88

[a] Isolated yield

Table 4 Optimization of temperature for the deprotection of benzyl methoxymethyl ether under traditional thermal conditions

Entry	*T* (°C)	Yield (%)[a]	Time (min)
1	25	45	600
2	45	78	180
3	50	91	60
4	80	91	60
6	120	91	60

[a] Isolated yield

Table 5 Results obtained using recycled [Hmim][HSO_4] in the deprotection reactions of benzyl methoxymethyl ether

Run	Method A		Method B	
	Time (min)	Yield (%)	Time (min)	Yield (%)
1	3	92	60	91
2	3	87	60	89
3	3	78	60	86
4	3	64	60	83
5	3	42	60	81

Initially, the amounts of acetic anhydride and HMDS were optimized in the reaction of 1 mmol of benzyl methoxymethyl ether in the presence of 0.5 mmol of [Hmim][HSO_4] and the best results were obtained by using 3 mmol of acetic anhydride or HMDS (Table 8). Under these conditions, a wide range of MOM and EOM ethers were subjected to acetylation and trimethylsilylation in the presence of [Hmim][HSO_4] under both MW irradiation and thermal conditions. The results are shown in Tables 9 and 10. A wide range of aliphatic and benzylic MOM and EOM ethers containing electron-withdrawing as well as electron-donating groups on the aromatic ring were easily converted into the corresponding acetates or TMS ethers in high yields in this one-pot procedure. It is also important to note that Friedel–Crafts acetylation was not observed in conversion of benzylic alkoxymethyl ethers to their corresponding acetates (Table 9, entries 1–16).

Conclusions

In conclusion, we demonstrated that [Hmim][HSO_4] is an efficient, green, and recyclable catalyst for deprotection of

Table 6 Competitive deprotection of alkoxymethyl ether of alcohols and phenols

Entry	Alkoxymethyl ether	Product	Traditional method		MW irradiation	
			Yield (%)	Time (min)	Yield (%)	Time (min)
1	$C_6H_5CH_2OMOM$	$C_6H_5CH_2OH$	91	60	92	3
	$4\text{-}ClC_6H_4CH_2OMOM$	$4\text{-}ClC_6H_4CH_2OH$	11		20	
2	$C_6H_5CH_2OMOM$	$C_6H_5CH_2OH$	91	60	92	3
	$C_6H_5C(Me)_2OMOM$	$C_6H_5C(Me)_2OH$	13		21	
3	$C_6H_5CH(Me)OMOM$	$C_6H_5CH(Me)OH$	90	105	88	4
	$4\text{-}ClC_6H_4CH_2OMOM$	$4\text{-}ClC_6H_4CH_2OH$	18		28	
4	$C_6H_5CH(Me)OMOM$	$C_6H_5CH(Me)OH$	90	60	88	4
	$C_6H_5C(Me)_2OMOM$	$C_6H_5C(Me)_2OH$	13		21	
5	$C_6H_5CH_2OEOM$	$C_6H_5CH_2OH$	90	60	92	3
	$C_6H_5C(Me)_2OEOM$	$C_6H_5C(Me)_2OH$	10		18	
6	$C_6H_5CH(Me)OMOM$	$C_6H_5CH(Me)OH$	90	90	88	4.5
	$4\text{-}ClC_6H_4CH_2OEOM$	$4\text{-}ClC_6H_4CH_2OH$	12		20	

Table 7 Competitive deprotection of alkoxymethyl ethers in the presence of acetates and TMS ethers

Entry	Protected group	Product	Traditional method		MW irradiation	
			Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)
1	$4\text{-}CH_3OC_6H_4CH_2OMOM$	$4\text{-}CH_3OC_6H_4CH_2OH$	91	60	93	3
	$C_6H_5CH_2OAc$	$C_6H_5CH_2OH$	14		24	
2	$4\text{-}CH_3OC_6H_4CH_2OMOM$	$4\text{-}CH_3OC_6H_4CH_2OH$	91	60	93	3
	$C_6H_5CH_2OTMS$	$C_6H_5CH_2OH$	6		12	
3	$4\text{-}ClC_6H_4CH_2OEOM$	$4\text{-}ClC_6H_4CH_2OH$	95	45	95	1
	$C_6H_5CH_2OAc$	$C_6H_5CH_2OH$	11		20	
4	$4\text{-}ClC_6H_4CH_2OEOM$	$4\text{-}ClC_6H_4CH_2OH$	95	45	95	1
	$C_6H_5CH_2OTMS$	$C_6H_5CH_2OH$	5		10	

[a] Isolated yield

Table 8 Optimization of Ac_2O and HMDS amounts in transformation of benzyl methoxymethyl ether to its acetate and TMS ether under the MW irradiation (method A)

Reagent (mmol)	Ac_2O		HMDS	
	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]
1	4	45	3.5	51
1.5	4	54	3.5	57
2	4	69	3.5	73
2.5	4	82	3.5	86
3	4	90	3.5	91
3.5	4	90	3.5	91
4	4	90	3.5	91

Reaction conditions: benzyl methoxymethyl ether (1 mmol) and ionic liquid (0.5 mmol)

[a] Isolated yield

a variety of MOM and EOM ethers under different reaction conditions. Furthermore, direct transformation of these ethers into the acetates and TMS ethers was performed by using this catalytic system. The notable special features of this methodology are the simple reaction procedure, excellent yields, chemoselectivity, and easy work-up.

Experimental

All products were identified by comparison of their physical and spectroscopic data with those of authentic samples [25–28]. [Hmim][HSO_4] [20] and MOM and EOM ethers [29] were prepared according to reported procedures. The microwave system used for these experiments includes the following items: Micro-SYNTH labstation, complete with glass door, dual magnetron system with pyramid-shaped diffuser, 1,000-W delivered power, exhaust system, magnetic stirrer, "quality pressure" sensor for flammable organic solvents, ATCFO fiber optic system for automatic temperature. During experiments, power, temperature, time, and pressure were monitored and controlled with the easyCONTROL software. Temperature was monitored

Table 9 Interconversion of MOM and EOM ethers to their corresponding acetates in the presence of [Hmim][HSO_4] under microwave irradiation (method A) and traditional method (method B)

Entry	R	MOM ether				EOM ether			
		MW irradiation (method A)		Traditional method (method B)		MW irradiation (method A)		Traditional method (method B)	
		Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]
1	$C_6H_5CH_2$	4	90	75	85	4	89	75	83
2	4-$(CH_3)_3CC_6H_4CH_2$	2.5	93	60	85	2.5	92	60	83
3	4-$PhCH_2OC_6H_4CH_2$	4	91	75	82	4	90	90	80
4	4-$CH_3OC_6H_4CH_2$	3	95	75	86	3	93	75	88
5	3-$CH_3OC_6H_4CH_2$	3.5	91	90	84	3.5	90	90	82
6	3-CH_3O-4-$(HO)C_6H_3$ CH_2	2.5	92	75	83	2.5	94	75	85
7	2-$HOC_6H_4CH_2$	3	89	75	86	3	89	60	85
8	2-$ClC_6H_4CH_2$	2.5	90	75	84	2.5	88	75	82
9	4-$ClC_6H_4CH_2$	2	95	60	87	2	93	60	86
10	2,4-$Cl_2C_6H_3CH_2$	2.5	90	60	82	2.5	90	60	81
11	2-$BrC_6H_4CH_2$	3.5	89	60	80	3	87	75	87
12	4-$BrC_6H_4CH_2$	2	96	60	87	2	93	60	85
13	4-$NO_2C_6H_4CH_2$	8	83	135	72	7.5	80	135	74
14	$(C_6H_5)_2CH$	6	83	135	76	6	84	150	77
15	$C_6H_5CH_2CH_2$	2.5	90	60	84	3	90	60	82
16	$C_6H_5CHCH_3$	6.5	88	135	75	6.5	85	135	77
17	$CH_3(CH_2)_6CH_2$	3	92	60	82	2.5	90	60	80
18	(+)-Menthyl	7.5	89	135	80	6.5	88	135	79
19	2-Adamantyl	9	85	180	70	9	82	195	71

[a] Isolated yield

Table 10 Interconversion of MOM and EOM ethers to their corresponding TMS ethers in the presence of [Hmim][HSO_4] under microwave irradiation (method A) and traditional method (method B)

Entry	R	MOM ether				EOM ether			
		MW irradiation (method A)		Traditional method (method B)		MW irradiation (method A)		Traditional method (method B)	
		Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]
1	$C_6H_5CH_2$	3.5	91	75	84	3.5	87	75	85
2	4-$(CH_3)_3CC_6H_4CH_2$	2.5	92	60	84	2.5	91	60	86
3	4-$PhCH_2OC_6H_4CH_2$	3.5	93	75	84	3.5	91	75	82
4	4-$CH_3OC_6H_4CH_2$	3	95	60	86	3	93	60	88
5	3-$CH_3OC_6H_4CH_2$	3.5	90	75	83	3.5	89	75	84
6	3-CH_3O-4-$(HO)C_6H_3CH_2$	2.5	94	75	89	2.5	93	75	87
7	2-$HOC_6H_4CH_2$	3	89	60	81	3	89	60	83
8	2-$ClC_6H_4CH_2$	2.5	90	75	81	2.5	88	75	82
9	4-$ClC_6H_4CH_2$	2	96	60	89	2	94	60	89
10	2,4-$Cl_2C_6H_3CH_2$	2.5	93	60	86	2.5	91	60	84
11	2-$BrC_6H_4CH_2$	3	93	60	87	3	92	60	85
12	4-$BrC_6H_4CH_2$	2	96	60	89	2	95	60	89
13	4-$NO_2C_6H_4CH_2$	7.5	84	135	76	7.5	82	135	74
14	$(C_6H_5)_2CH$	6	85	135	79	6	84	135	80
15	$C_6H_5CH_2CH_2$	2.5	92	60	88	2.5	90	60	87
16	$C_6H_5CHCH_3$	6.5	89	135	77	6.5	89	135	78

Table 10 continued

Entry	R	MOM ether				EOM ether			
		MW irradiation (method A)		Traditional method (method B)		MW irradiation (method A)		Traditional method (method B)	
		Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]	Time (min)	Yield (%)[a]
17	$CH_3(CH_2)_6CH_2$	2.5	94	60	86	2.5	91	60	84
18	(+)-Menthyl	7	90	135	83	6.5	88	135	80
19	2-Adamantyl	8	87	180	74	8	85	180	75

[a] Isolated yield

with the aid of ATCFO sensor TS3517 inserted directly into the corresponding reaction container.

Deprotection of 4-chlorobenzyl methoxymethyl ether to 4-chlorobenzyl alcohol: typical procedure

A mixture of 1 mmol 4-chlorobenzyl methoxymethyl ether and 0.5 mmol [Hmim][HSO_4] was stirred at 50 °C or exposed to microwave irradiation (150 W). After the reaction was completed (detected by TLC), the reaction mixture was cooled to room temperature and Et_2O (2×10 cm^3) was added. The organic layer was washed with 10 cm^3 H_2O and dried over anhydrous Na_2SO_4. The solvent was evaporated and the crude product was purified by column chromatography on neutral alumina (eluent: *n*-hexane/ethyl acetate, 4:1) to afford the pure 4-chlorobenzyl alcohol [30] (Table 3, entry 9).

Direct transformation of 4-chlorobenzyl methoxymethyl ether to 4-chlorobenzyl acetate and (4-chlorobenzyloxy)trimethylsilane: typical procedure

A mixture of 1 mmol 4-chlorobenzyl methoxymethyl ether, 3 mmol Ac_2O or HMDS, and 0.5 mmol [Hmim][HSO_4] was stirred at 50 °C or exposed to microwave irradiation (150 W). The progress of the reaction was monitored by TLC (eluent: *n*-hexane/ethyl acetate, 4:1). After completion of the reaction, the excess amounts of Ac_2O or HMDS were evaporated and Et_2O (2×10 cm^3) was added. The organic layer was washed with 10 cm^3 H_2O and dried over anhydrous Na_2SO_4. Evaporation of the solvent followed by column chromatography on neutral alumina afforded the pure 4-chlorobenzyl acetate or (4-chlorobenzyloxy)trimethylsilane (Tables 9, 10, entry 9).

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