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Melamine trisulfonic acid: A new, efficient and reusable catalyst for the protection of alcohols, phenols, aldehydes and amines

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ABSTRACT

Tetrahydropyranylation of alcohols and phenols, acetylation of aldehydes and N-*Boc* protection of amines are efficiently catalyzed by melamine trisulfonic acid (MTSA). All reactions were performed at room temperature in good to high yields.

***Keywords**: Melamine trisulfonic acid, Alcohols, Phenols, Aldehydes, Amines.*

1. Introduction

The hydroxyl, formyl and amino groups are present in a number of compounds of biological and synthetic interest. Protections of these functional groups during multi-step synthesis are important processes, which are under considerable attention of organic chemists [1].

Because of the remarkable stability of tetrahydropyranyl ethers towards a variety of conditions, such as strongly basic media, Grignard reagents and alkyllithiums, reduction with hydride, oxidation, oxidative alkylation and acylation reactions, tetrahydropyranylation has found a wide variety of applications in protecting the hydroxyl group of alcohols. Different types of reagents have been developed for the promotion of the tetrahydropyranylation of the hydroxyl functions [2-10]. Although these methods are suitable for many synthetic conditions, many of these are associated with several drawbacks, which include use of expensive and toxic reagents, tedious and time consuming work up procedures, high temperature, long reaction times, incompatibility with other acid-sensitive functional groups, formation of polymeric by-products of the dihydropyran (DHP) and isomerization. Thus there is still a need for mild and selective methods, especially using heterogeneous catalysts for this purpose.

Conversion of aldehydes to their corresponding 1,1-diacetates is one of the most important methods used for the protection of this type of compounds. The importance of this method can be attributed to: i) relative stability of 1,1-diacetates in neutral, acidic and basic media [11], ii) use of acylals as cross-linking reagents for cellulose in cotton [12] and bleaching activators in wine-stained fabrics [13], and iii) usefulness of acylals as intermediates for nucleophilic substitution reactions [14].

Generally, the formation of acylals carried out by the reaction of aldehydes with acetic anhydride under catalysis of reagents such as H_2SO_4 and H_3PO_4 [15], PCl_3 [16], NBS [17], $ZrCl_4$ [18], $Fe_2(SO_4)_3.xH_2O$ [19], $FeCl_3$-SiO_2 [20], *p*-toluene sulfonic acid [21], $SbCl_3$ [22], $HClO_4$-SiO_2 [23], silica sulfuric acid [24, 25], copper methanesulfonate / HOAc [26], Amberlyst®-15 [27]. Although these methods are an improvement most of them suffer from disadvantages such as long reaction times, harsh reaction conditions, low yields, use of corrosive, expensive or moisture-sensitive reagents, use of excess amounts of Ac_2O, strictly reactive condition (N_2 atmosphere), special efforts required to prepare the catalyst and tedious work-up procedure. Thus, the search for new reagents and methods is still of practical importance.

Due to the remarkable stability of *tert*-butyl carbamates towards a variety of reactions such as base-catalyzed nucleophilic substitutions and catalytic hydrogenation [28], and due to its low cost, availability and ease with which it can be removed [29], di-*tert*-butoxypyrocarbonate [$(Boc)_2O$] can be selected as one of the best candidates for the protection of amines in multi-step organic synthesis [30-32]. There are several reagents available for the promotion of the N-Boc protection of amines with $(Boc)_2O$, which of them aq. NaOH [33], $Cu(BH_4)_2.xH_2O$ [34], $H_3PW_{12}O_{40}$ [35], indium (III) halides [36], sulfamic acid [37], sulfonic acid functionalized silica [38], I_2 [39], and $HClO_4$ / silica [40] are examples. Although these methods are suitable for many synthetic conditions, many of these are associated with several drawbacks, which include use of corrosive or moisture sensitive reagents, long reaction times, tedious work-up, formation of side-products during base-catalyzed reactions, difficulty in the preparation of reagent and using excess of reagent in the case of *Lewis* acid catalyzed reactions. Therefore, introduction of new methods and catalysts for the preparation of *tert*-butyl carbamates is still in demand.

2. Experimental

Chemicals were purchased from Fluka, Merck and Aldrich Chemical Companies. All yields refer to the isolated products. The purity determination of the substrate and reaction monitoring were

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accompanied 1by thin-layer chromatography (TLC) on a silica-gel polygram SILG / UV 254 plates. The IR spectra were recorded on a Perkin Elmer 781 Spectrophotometer. In all the cases the ^1H NMR spectra were recorded with Bruker Avance 300 MHz instrument. Chemical shifts are reported in parts per million in $CDCl_3$ with tetramethylsilane as an internal standard. ^{13}C NMR data were collected on Bruker Avance 75 MHz instrument.

2.1. Preparation of MTSA

A 250 mL suction flask charged with chlorosulfonic acid (5 mL, 75.2 mmol) was equipped with a gas inlet tube for conducting HCl gas overran adsorbing solution i. e. water. Melamine (3.16 g, 25.07 mmol) was added in small portions over a period of 30min at room temperature. HCl gas evolved from reaction vessel immediatel. After completion of the addition of melamine, the mixture was shaken for 30 min, meanwhile, the residual HCl was exhausted by suction. The mixture was triturated with n-hexane (10 mL) and then filtered. The solid residue was washed with n-hexane (10 mL) and dried under vacuum. Melamine trisulfonic acid (7.9 g, 87%) was obtained as a white solid, which was stored in a capped bottle. M.p.: 142-144 ^OC; IR: υ= 3133, 2621, 1654, 1509, 1175, 1069 cm $^{-1}$; Anal calcd for $C_3H_6N_6O_9S_3$ (366.3): C, 9.83 %; N, 22.95 %; H, 1.64 %. Found: C, 9.81 %; N, 22.95 %; H, 1.64 %.

2.2 General procedure for the tetrahydropyranylation of alcohols and phenols

A mixture of the substrate (1 mmol), 3,4-dihydro-2H-pyran (1.1 mmol, 0.094 g) and MTSA (0.03 mmol, 0.011 g) in CH_2Cl_2 (3 mL) was stirred at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was filtered through a silica gel pad, and the solid residue was washed with CH_2Cl_2 (2 × 5 mL). Evaporation of the solvent gave the desired products in high purity.

2.3 General procedure for the synthesis of 1,1-diacetates

Aldehyde (1 mmol), acetic anhydride (2 mmol), and MTSA (0.03 mmol, 0.011g) were added in a flask and stirred for the appropriate time (Table 1). The reaction was monitored by TLC. On completion, CH_2Cl_2 (5 mL) was added to the mixture and filtered. The catalyst washed with CH_2Cl_2 (5 mL) and then dried; the recovered catalyst can be used for two reactions again. The organic layer was washed with 5% solution of $NaHCO_3$, then with water and dried over $MgSO_4$. Evaporation of the solvent followed by column chromatography on silica gel afforded the pure 1,1-diacete.

2.4 General procedure for the N-Boc protection of amines

1 mmol of the substrate was added to a mixture of MTSA (0.05 mmol, 0.02 g) and $(Boc)_2O$ (1.1 mmol, 0.24 g) and stirred at room temperature for the appropriate time (Table 1). The progress of the reaction was monitored by TLC. On completion, CH_3CN (5 mL) was added to the mixture and filtered. The solid residue was washed with CH_3CN (5 mL). Evaporation of the solvent followed by column chromatography (silica-gel) eluting with *n*-hexane-EtOAc (1:1) followed by evaporation of the solvent gave the desired product in good to high yields. On each occasion the spectral data of known compounds were found to be identical with those reported in the literature.

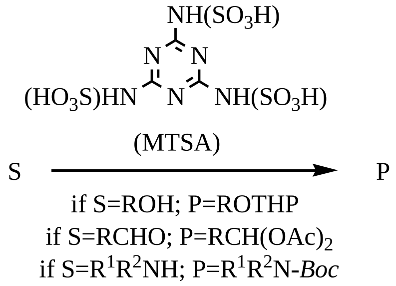


Fig. 1. Protection of alcohols, phenols, aldehydes and amines catalyzed by MTSA

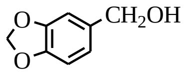
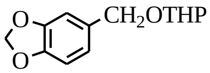
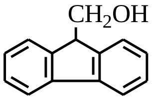
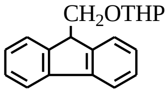
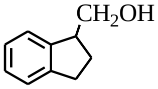
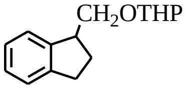
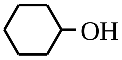
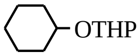
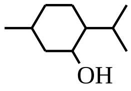
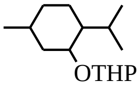
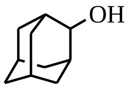
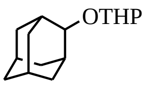
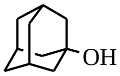
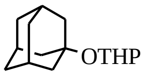
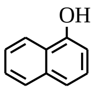
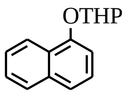
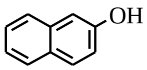
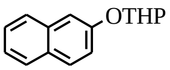
The Selected spectral data:

Table 2, entry 2: White solid, m.p. 81-83 °C; IR (KBr): υ 3100, 3050, 1760, 1740, 1587, 1454, 1430, 1370, 1350, 1232, 1198, 1095, 1060, 1010, 970, 938, 908, 786, 744 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.15 (s, 6 H), 7.57-7.61 (m, 1 H), 7.67-7.74 (m, 2 H), 8.05 (dd, *J* = 0.8, *J* = 7.6 Hz, 1 H), 8.20 (s, 1 H).
Table 2, entry 8 : White solid: m.p. 80 °C. IR(KBr): υ 3090, 2990, 2920 1760, 1738, 1592, 1490, 1376, 1242, 1200, 1084, 1060, 1010, 993, 970, 938, 910, 840, 820, 608, 540 c^{m-1}. ; ^1H NMR (CDC_{l3}): δ 7.64 (s, 1H), 7.44 (d, *J* = 7.2 Hz, 2H), 7.38 (d, *J* = 7.2 Hz, 2H), 2.11 (s, 6H).
Table 2, entry 10: White solid, mp 86-88 °C; IR (KBr): υ 3100, 2992, 2930, 1743, 1590, 1482, 1370, 1230, 1210, 1060, 1010, 968, 940, 820, 717 cm^{-1}. ^1H NMR (400 MHz, CDC_{l3}): δ 2.10 (s, 6H), 7.39 (d, 2H, *J* = 8.4 Hz), 7.53 (d, 2H, *J* = 8.8 Hz), 7.61 (s, 1H).
Table 2, entry 13: White solid, m.p. 67-69 °C; ^1H NMR (400 MHz, CDC_{l3}) δ 8.02 (s, 1H), 7.49 (d, J=8.4 Hz, 2H), 7.36 (t, J=7.6 Hz, 1H), 7.00 (t, J=7.6 Hz, 1H), 6.91 (d, J=8.4 Hz, 1H), 3.83 (s, 3H), 2.11 (s, 6H).
Table 3, entry 1: Yellow solid; mp: 188 °C; IR (KBr): 3356, 2980, 1690, 1550 cm $^{-1}$. ^{1} H NMR (CDC_{l3}, 90 MHz): δ 1.4 (s, 18 H), 3.8 (s, 2 H), 6.4 (br, 2 H), 7.2 (m, 8 H).
Table 3, entry 16: White solid; Mp: 163 °C; IR (KBr): 2976, 1690 cm $^{-1}$.
^{1} H NMR ($CDCl_3$, 90 MHz): δ 1.4 (s, 18 H), 3.3 (s, 8 H).
Table 1, entry, 18: Colorless oil: IR (Nujol): 3450, 2976, 1700 cm $^{-1}$.
^{1} H NMR ($CDCl_3$, 90 MHz): δ 1.3 (s, 9 H), 2.3 (m, 6 H), 3 (s, 1 H), 3.3 (m, 6 H).
Table 3, entry 19: Colorless oil: IR (Nujol): 2930, 1660, 1167 cm $^{-1}$.
^{1} H NMR ($CDCl_3$, 90 MHz): δ= 1.4 (s, 9 H), 3.4-3.7 (m, 24 H).
Table 3, entry 20: Colorless solid: mp: 80 °C; IR (KBr): 2978, 1650, 1200 cm $^{-1}$. ^{1} H NMR ($CDCl_3$, 90 MHz): δ= 1.4 (s, 18 H), 3.2-3.7 (m, 24 H).

3. Results and Discussion

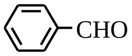
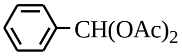
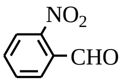
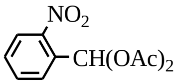
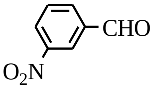
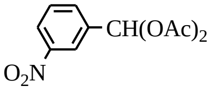
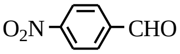
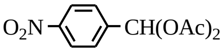
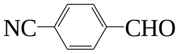
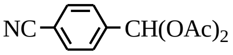
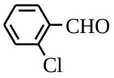
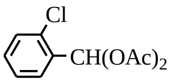
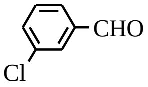
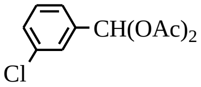
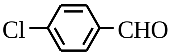
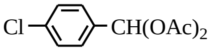
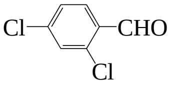
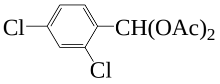
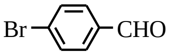
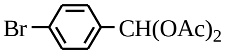
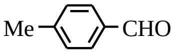
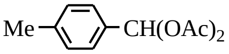
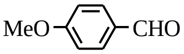
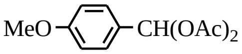
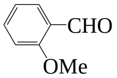
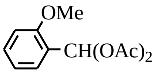
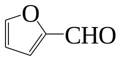
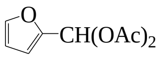
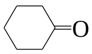
Recently, we have reported the preparation of melamine trisulfonic acid (MTSA) and its application in the promotion of the acetylation of alcohols, phenols and amines, oxathioacetalyzation of aldehydes and methoxymethylation of alcohols [41-43]. In continuation of

Table 1.
Tetrahydropyranylation of alcohols and phenols.[a, b, c]

Entry	Substrate	Product	Time (min)	Yield (%)
1	$PhCH_2OH$	$PhCH_2OTHP$	35	95
2	$4\text{-}ClC_6H_4CH_2OH$	$4\text{-}ClC_6H_4CH_2OTHP$	8	90
3	$2\text{-}ClC_6H_4CH_2OH$	$2\text{-}ClC_6H_4CH_2OTHP$	10	92
4	$2\text{-}BrC_6H_4CH_2OH$	$2\text{-}BrC_6H_4CH_2OTHP$	13	90
5	$2\text{-}NO_2C_6H_4CH_2OH$	$2\text{-}NO_2C_6H_4CH_2OTHP$	18	87
6	$3\text{-}NO_2C_6H_4CH_2OH$	$3\text{-}NO_2C_6H_4CH_2OTHP$	40	90
7	$4\text{-}NO_2C_6H_4CH_2OH$	$4\text{-}NO_2C_6H_4CH_2OTHP$	35	95
8	$2\text{-}MeC_6H_4CH_2OH$	$2\text{-}MeC_6H_4CH_2OTHP$	18	92
9	$4\text{-}Me_3CC_6H_4CH_2OH$	$4\text{-}Me_3CC_6H_4CH_2OTHP$	26	92
10	$2\text{-}MeOC_6H_4CH_2OH$	$2\text{-}MeOC_6H_4CH_2OTHP$	20	90
11	$3\text{-}MeOC_6H_4CH_2OH$	$3\text{-}MeOC_6H_4CH_2OTHP$	15	85
12	$4\text{-}MeOC_6H_4CH_2OH$	$4\text{-}MeOC_6H_4CH_2OTHP$	14	91
13			32	90
14			30	95
15	$PhCH_2CH_2OH$	$PhCH_2CH_2OTHP$	36	92
16	$PhCH_2CH_2CH_2CH_2OH$	$PhCH_2CH_2CH_2CH_2OTHP$	35	92
17	$PhCH(Me)CH_2OH$	$PhCH(Me)CH_2OTHP$	30	92
18	$MeCH_2CH_2CH_2OH$	$MeCH_2CH_2CH_2OTHP$	30	87
19	$PhCH(OH)Me$	$PhCH(OTHP)Me$	18	95
20	Ph_2CHOH	$Ph_2CH(OTHP)$	19	92
21			19	90
22	$PhCH_2CH(OH)Me$	$PhCH_2CH(OTHP)Me$	28	90
23	$MeCH_2CH(OH)Me$	$MeCH_2CH(OTHP)Me$	44	95
24			60	90
25			105	82
26			120	85
27			150	75
28	$4\text{-}MeOC_6H_4OH$	$4\text{-}MeOC_6H_4OTHP$	5	80
29	$4\text{-}ClC_6H_4OH$	$4\text{-}ClC_6H_4OTHP$	10	80
30			5	82
31			7	75

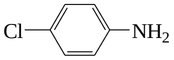
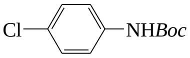
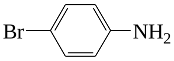
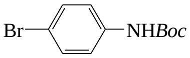
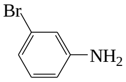
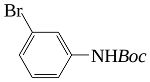
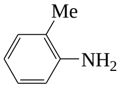
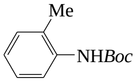
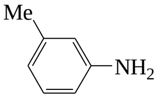
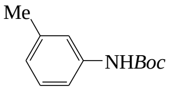
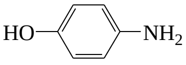
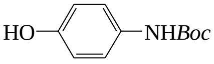
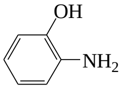
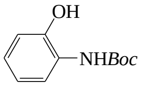
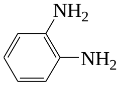
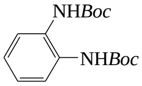
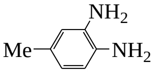
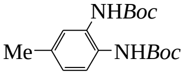
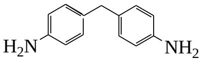
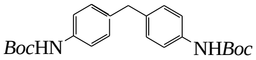
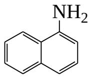
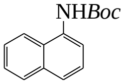
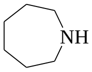
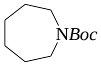
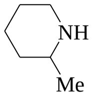
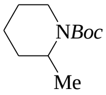
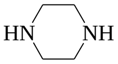
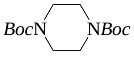
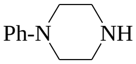
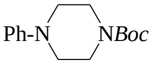
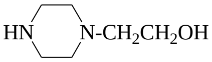
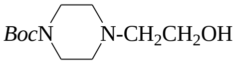
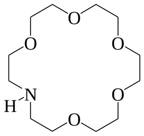
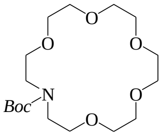
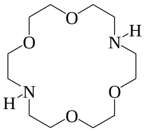
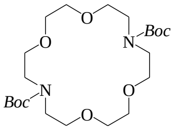
[a] Products were characterized by their physical constants, comparison with authentic samples, IR and NMR spectroscopy.
[b] Isolated yield.
[c] Reaction conditions: substrate (1 mmol) / DHP (1.1 mmol) / MTSA (0.03 mmol) / CH_2Cl_2 (3 mL) / r.t.

Table 2.
MTSA catalyzed formation of 1,1-diacetates.[a, b, c]

Entry	Substrate	Product	Time (min)	Yield (%)
1			1	95
2			1	92
3			1 (1, 2) [d]	90 (87, 82) [d]
4			1	92
5			1	85
6			1	90
7			1	90
8			1	87
9			1	85
10			1	90
11			2 (2, 4) [d]	90 (85, 85) [d]
12			1	90
13			3	85
14			2	90
15	$PhCH_2CH_2CHO$	$PhCH_2CH_2CH(OAc)_2$	1	90
16	$PhCOCH_3$	---	15	0 [e]
17	Ph_2CO	---	10	0 [e]
18		---	30	0 [e]

[a] Products were characterized by their physical constants, comparison with authentic samples, IR and NMR spectroscopy.
[b] Isolated yield.
[c] Reaction conditions: substrate (1 mmol) / Ac_2O (2 mmol) / MTSA (0.03 mmol) / r.t.
[d] Results obtained using recycled catalyst for the second and third times, respectively.
[e] Starting material recovered intact.

Table 3.
Boc protection of amines catalyzed by MTSA.[a, b, c]

Entry	Substrate	Product	Time (h)	Yield (%)
1			2	76
2			3	70
3			8	87
4			1.5	96
5			2	80
6			2	92
7			3	84
8			1	90 [d]
9			30 (min)	92 [d]
10			3	75 [d]
11			3	84
12	$PhCH_2NH_2$	$PhCH_2NH$*Boc*	1	85
13	$(PhCH_2)_2NH$	$(PhCH_2)_2N$*Boc*	0.5	92
14			3 (min)	95
15			30 (min)	85
16			20 (min)	80 [d]
17			5 (min)	88
18			5 (min)	85
19			40 (min)	90
20			1	85 [d]

[a] Products were identified spectroscopically.
[b] Isolated yield.
[c] Reaction conditions: Substrate (1 mmol) / $(Boc)_2O$ (1.1 mmol) / MTSA (0.05 mmol) / r.t.
[d] Reaction was performed in the presence of 2.1 mmol $(Boc)_2O$.

Table 4.
Comparison of some of the results obtained by our method (1), with some of those reported by *p*-toluene sulfonic acid (2) [21], copper methanesulfonate / HOAc (3) [26] and silica sulfuric acid (4) [24].

Entry	Product	Time (min)/ Yield (%)			
		(1)	(2)	(3)	(4)
1	$PhCH(OAc)_2$	1/95	25/96	75/94	30/84
2	$4\text{-}NO_2C_6H_4CH(OAc)_2$	1/92	40/92	12(h)/84	25/86
3	$4\text{-}MeOC_6H_4CH(OAc)_2$	1/90	30/90	150/78	20/94

these studies, herein, we wish to report the applicability of this reagent in the promotion of the preparation of the tetrahydropyranylation of alcohols and phenols, acetylation of aldehydes and N-*Boc* protection of amines (Fig. 1).

Different types of benzylic alcohols having both electron-withdrawing and –donating groups were tetrahydropyranylated with DHP in the presence of catalytic amounts of MTSA in CH_2Cl_2 at room temperature in good to high yields (Table 1, Entries 1-13). Primary and secondary aliphatic alcohols were also efficiently converted to their corresponding tetrahydropyranyl ethers under the same reaction conditions (Table 1, Entries 14-23). This method was found to be useful for the protection of hindered secondary and tertiary alcohols (Table 1, Entries 24-27). This method is also useful for the protection of phenols as their corresponding tetrahydropyranyl ethers (Table 1, Entries 28-31). Using this method, by-products resulting from polymerization of dihydropyran, isomerization and dehydration of secondary and tertiary alcohols were not observed

The acetylation of aldehydes with acetic anhydride was performed in the presence of catalytic amounts of MTSA in the absence of solvent at room temperature, to produce the desired 1,1-diacetate in good to high yields (Table 2). Aliphatic and aromatic aldehydes, including different types of substituents, were protected without the formation of any other side products in the presence of 0.03 mol equiv of the catalyst. Ketones remain intact under the same reaction conditions (Table 2, Entries 17-19). We then investigated the possibility for the selective acetylation of the aldehydes in the presence of ketones. This is exemplified by the competitive reaction between 2-nitrobenzaldehyde and benzophenone (Table 2, Entry 20).

We have also investigated the N-*Boc* protection of amines in the presence of MTSA. Optimization of the reaction conditions showed that the best results were obtained in the absence of solvent at room temperature when the relative ratio of the substrate, $(Boc)_2O$ and MTSA was 1:1.1:0.05, respectively. After that different types of amines including aliphatic and aromatic ones were subjected to N-*Boc* protection under the determined conditions.

As shown in Table 3, all types of the above mentioned substrates times. It is very important to note that by-products such as isocyanates, urea and N,N-di-*Boc* derivatives were not observed were efficiently converted to their corresponding *tert*-butyl carbamates in good to high yields during the acceptable reaction using this method. Because of the stability of phenols and alcohols under the above mentioned conditions, the method can be used for the chemoselective *Boc* protection of amines in the presence of phenols and alcohols (Table 3, Entries 6, 7, 18).

To illustrate the efficiency of the proposed method, Table 4 compares some of the results obtained from the acetylation of aldehydes with some of those reported in the literature.

4. Conclusion

In conclusion, we have introduced melamine trisulfonic acid as a novel recyclable catalyst for the promotion of the tetrahydropyranylation of alcohols and phenols, acetylation of aldehydes and N-Boc protection of amines. High yields of the products, short reaction times, easy work-up and high chemoselectivity are among the other advantages of this method, which make this procedure a useful and attractive addition to the available methods.

Acknowledgments

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