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Zirconyl triflate: A New, Highly Efficient and Reusable Catalyst for Acetylation and Benzoylation of Alcohols, Phenols, Amines and Thiols with Acetic and Benzoic Anhydrides

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Highly efficient acetylation and benzoylation of alcohols, phenols, amines and thiols with acetic and benzoic anhydrides catalyzed by new and reusable zirconyl triflate, $ZrO(OTf)_2$, is reported. The high catalytic activity of electron deficient $ZrO(OTf)_2$ can be used for the acetylation and benzoylation of not only primary alcohols but also sterically-hindered secondary and tertiary alcohols with acetic and benzoic anhydrides. Acetylation of phenols with acetic and benzoic anhydrides was achieved to afford the desired acetates and benzoates efficiently. This catalyst also efficiently catalyzed the acetylation and benzoylation of amines and thiols whereby the corresponding amides and thioesters were obtained in good to excellent yields. This catalyst can be reused several times without loss of its activity.

Keywords: Zirconyl triflate, Reusable catalyst, Acetylation, Benzoylation, Esterification

INTRODUCTION

Protection of alcohols, phenols, amines and thiols *via* acetylation and benzoylation is a commonly used transformation in synthetic organic chemistry. The protection of such functional groups is often necessary during the course of various transformations in a synthetic sequence, especially in the construction of polyfunctional molecules such as nucleosides, carbohydrates, steroids, and natural products [1].

A variety of procedures are routinely followed for the preparation of acetyl derivatives, including homogeneous or heterogeneous catalysts such as iodine [2], *p*-toluenesulfonic acid [3], alumina [4], zinc chloride [5], cobalt chloride [6], montmorillonit K-10 and KSF [7], zeolite HSZ-360 [8], zirconium sulfophenyl phosphonate [9], $[Sc(OTf)_3]$ [10], $TaCl_5$ [11], TMSOTf [12], $[Cu(OTf)_2]$ [13], $[In(OTf)_3]$ [14], magnesium bromide [15], bismuth(III) salts [16], ferric perchlorate adsorbed on silica-gel [17], $RuCl_3$ [18], $InCl_3$ [19], $Ce(OTf)_3$ [20], $Mg(ClO_4)_2$ [21], $ZrCl_4$ [22], Cp_2ZrCl_2 [23], $ZrOCl_2.8H_2O$ [24], $Al(OTf)_3$ [25], $NaHSO_4{\cdot}SiO_2$ [26], $La(NO_3)_3.6H_2O$ [27], $NbCl_5$ [28], $Gd(OTf)_3$ [29], Alumina supported MoO_3 [30] cerium polyoxometalate [31] $Ce(OTf)_4$ [32], $(SiO_2)_n$-PCl_{3-n} [33], copper salts [34,35], $AlPW_{12}O_{40}$ [36, 37] and electron-deficient tin(IV) porphyrins [38-40]. However, some of the reported methods for the acetylation of alcohols suffer from one or more of the following disadvantages such as high temperature and drastic reaction conditions, formation of undesirable or toxic by-products, expensive reagents, hygroscopicity and thermal instability of the reagents, long reaction times, low yields of the desired products and bulk requirement of solid bed. Therefore, introduction of new methods and catalysts for the preparation

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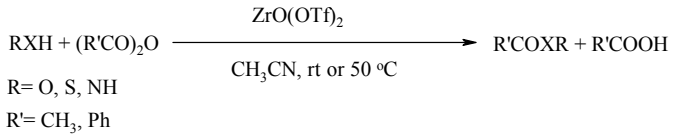


Scheme 1

of esters is warranted as ever.

Zirconium(IV) salts have recently attracted much attention due to their low cost, high catalytic activity, easy availability and low toxicity. A variety of Zr(IV) salts have been used for several organic transformations [41]. In this paper, we report a rapid and efficient acetylation and benzoylation of alcohols, phenols, amines and thiols with acetic and benzoic anhydrides catalyzed by $ZrO(OTf)_2$ (Scheme 1).

EXPERIMENTAL

Chemicals were purchased from Fluka and Merck chemical companies. ^1H NMR spectra were recorded in $CDCl_3$ solvent on a Bruker AM 80 MHz or a Bruker AC 500 MHz spectrometer using TMS as an internal standard. Infrared spectra were run on a Philips PU9716 or Shimadzu IR-435 spectrophotometer. Analyses were performed on a Shimadzu GC-16A instrument with a flame ionization detector using silicon DC-200 or Carbowax 20M columns. $ZrO(OTf)_2$ was prepared as we have previously reported [42].

General Procedure for Acetylation Reactions

To a solution of alcohol, phenol, amine or thiol (1 mmol) and Ac_2O (2 mmol) in CH_3CN (3 ml) was added $ZrO(OTf)_2$ (0.5-2 mol%). The mixture was stirred at room temperature. The reaction progress was monitored by GC or TLC. After the reaction was completed, the solvent was evaporated, Et_2O (20 ml) was added, and the catalyst was filtered. The filtrate was washed with 5% aqueous solution of $NaHCO_3$ and dried (Na_2SO_4). Evaporation of the solvent followed by column chromatography on silica-gel gave the pure product.

General Procedure for Benzoylation Reaction

A solution of alcohol, phenol, amine or thiol (1 mmol), $(PhCO)_2O$ (2 mmol), CH_3CN (3 ml) and $ZrO(OTf)_2$ (0.5-2 mol%) was prepared and stirred at 50 °C. The reaction progress was monitored by GC or TLC. At the end of the reaction, the solvent was evaporated, Et_2O (20 ml) was added and the catalyst was filtered. The filtrate was washed with 5% aqueous solution of $NaHCO_3$ and dried (Na_2SO_4). Evaporation of the solvent followed by column chromatography on silica-gel gave the product.

RESULTS AND DISCUSSION

Acetylation and Benzoylation of Alcohols with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$

First, the ability of $ZrO(OTf)_2$ in the acetylation of 4-methoxybenzyl alcohol (1 mmol) was investigated in the presence of 0.5 mol% of catalyst, where the alcohol was converted completely into the desired acylated product with acetic anhydride (2 equivalents), and in CH_3CN as solvent. The obtained results for acetylation of different primary, secondary (including aliphatic and aromatic alcohols) and tertiary alcohols showed that the reaction was immediately completed for nearly all alcohols at room temperature and no alcohol was detected by TLC or GC. However, indanol, 1-adamantanol, *t*-butyl alcohol and 9-anthracenylmethanol required somewhat longer reaction time (Table 1). In the absence of the catalyst, the reaction was much less efficient for the conversion of alcohols to esters.

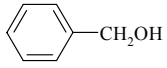
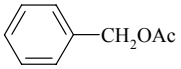
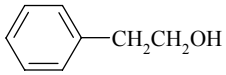
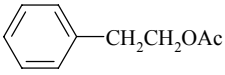
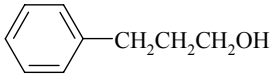
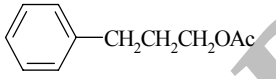
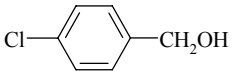
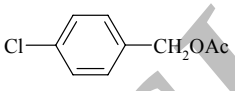
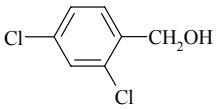
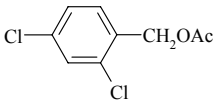
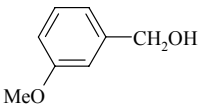
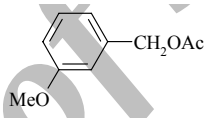
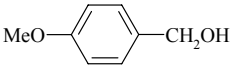
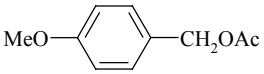
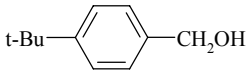
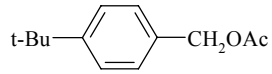
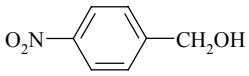
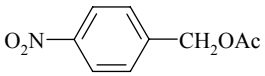
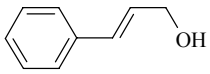
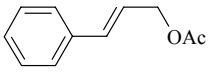
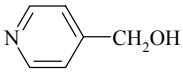
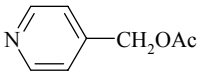
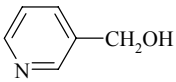
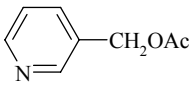
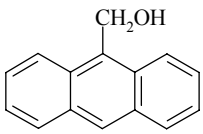
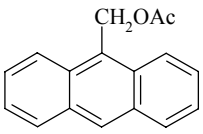
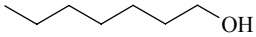
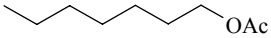
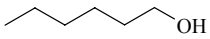
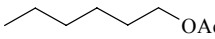
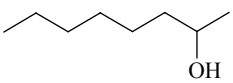
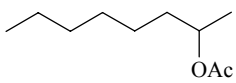
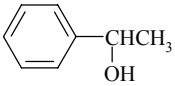
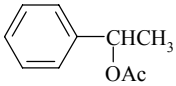
It is noteworthy that in the acetylation of tertiary alcohols such as 1-adamantanol and *t*-butyl alcohol no dehydration products were observed (entries 21 and 22). In the case of benzylic alcohols, the nature of substituents had no significant effect on the acetylation yields. In comparison with other zirconium salts [22-24], $ZrO(OTf)_2$ was superior in product yields, catalyst amount and reaction times.

Some of the above mentioned alcohols were subjected to benzoylation with benzoic anhydride, a less reactive anhydride, in the presence of 0.5 mol% of $ZrO(OTf)_2$ at 50 °C. The results which are summarized in Table 1 (entries 23-36), showed that $ZrO(OTf)_2$ is an efficient catalyst for the preparation of benzoates from alcohols.

The exact mechanism of these reactions is not clear at present. However, it has been reported that in the presence of triflates, a mixed anhydride (CF_3SO_2OCOR') is produced and then alcohol attacks it to give the corresponding ester (Scheme 2) [13].

The reusability of this catalyst was investigated in the acetylation of benzyl alcohol with acetic anhydride. After each run, the catalyst was filtered, dried and used in the next run.

Table 1. Acetylation and Benzoylation of Alcohols with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$[a]

Entry	Alcohol	Product	Time (min)	Yield (%)[b]
1			1	100
2			1	100
3			1	100
4			1	100
5			1	100
6			1	100
7			1	100
8			1	100
9			1	100
10			1	98
11			1	100
12			1	100
13			15	99
14			1	100
15			1	100
16			1	99
17			1	98

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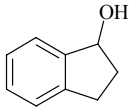
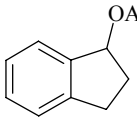
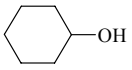
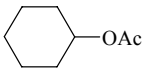
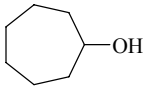
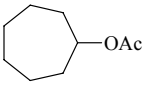
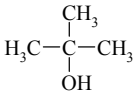
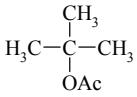
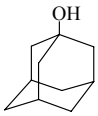
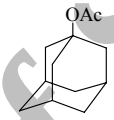
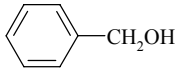
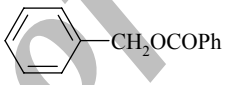
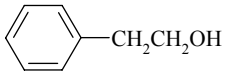
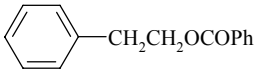
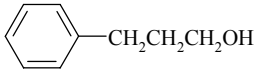
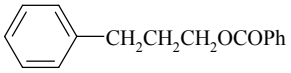
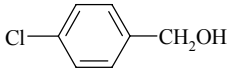
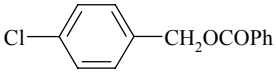
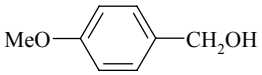
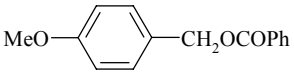
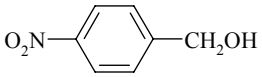
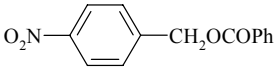
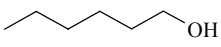
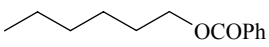
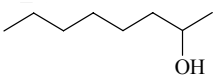
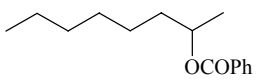
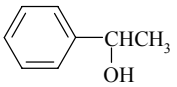
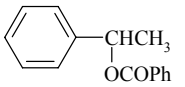
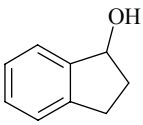
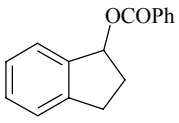
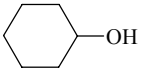
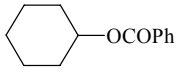
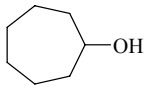
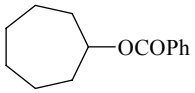
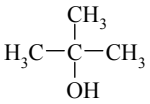
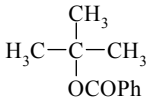
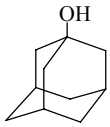
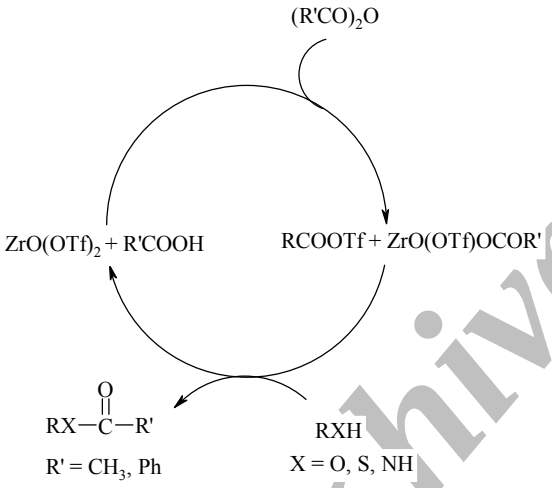
18			10	100
19			1	84
20			1	100
21			10	99
22			5	97
23			5	100
24			5	95
25			5	90
26			5	95
27			5	100
28			5	95
29			5	100
30			5	100
31			5	100
32			10	100
33			5	100

Table 1. Continued

34			5	100
35			10	100
36			10	100

[a]Reaction conditions: alcohol (1.0 mmol), catalyst (0.5 mol%), Ac_2O or $(PhCO)_2O$ (2 mmol), CH_3CN (2 ml). [b]GC yield.



Scheme 2

The results showed that $ZrO(OTf)_2$ could be reused several times without loss of its catalytic activity.

Acetylation and Benzoylation of Phenols with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$

The ability of this catalyst in the acetylation of phenols with acetic anhydride was investigated. The reaction of phenols with acetic anhydride in the presence of 0.005 M equivalent of the catalyst in CH_3CN, as solvent, was carried out and the desired acetates were obtained in excellent yields at room temperature (Table 2, entries1-14). The acetylation of dihydroxybenzenes such as hydroquinone, pyrocatechol and resorcinol, and trihydroxybenzene such as pyrogallol was also achieved. The results showed that all hydroxyl groups were acetylated and the desired polyacetates were obtained in 99% yields (Table 2, entries 5-8). In the absence of catalyst and under the same reaction conditions, only 5-20% of acetylated products were obtained in the acetylation of phenols.

Benzoylation of phenols with benzoic anhydride was also carried out in the presence of catalytic amounts of $ZrO(OTf)_2$ (0.5 mol%) at 50 °C and the corresponding benzoates were obtained in excellent yields (Table 2, entries 15-19).

Acetylation and Benzoylation of Amines with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$

The high catalytic activity of $ZrO(OTf)_2$ in the acetylation and benzoylation of alcohols and phenols, prompted us to investigate its catalytic activity in the acetylation of amines. In this manner the acetylation of different amines with acetic anhydride was carried out in the presence of catalytic amounts of $ZrO(OTf)_2$ (1 mol%) at room temperature and the corresponding acetamides were obtained in excellent yields (Table 3, entries 1-12). Benzoylation of amines with benzoic anhydride was also performed at 50 °C in the presence of 2 mol% of $ZrO(OTf)_2$ and the corresponding benzamides were produced in excellent yields (Table 3, entries 13-20).

Acetylation and Benzoylation of Thiols with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$

Finally, we investigated the acetylation and benzoylation of thiols in the presence of $ZrO(OTf)_2$. The results, which are summarized in Table 4, showed that $ZrO(OTf)_2$ (2 mol%) was also an efficient catalyst for acetylation of thiols with acetic anhydride at room temperature and short reaction times (entries 1-7). The corresponding thiobenzoates were obtained

Table 2. Acetylation and Benzoylation of Phenols with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$[a]

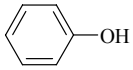
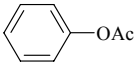
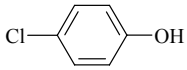
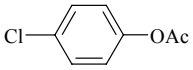
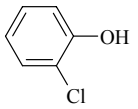
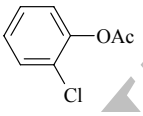
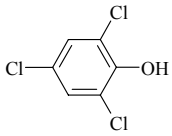
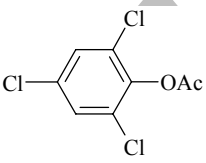
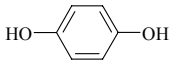
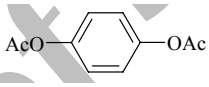
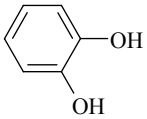
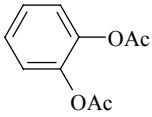
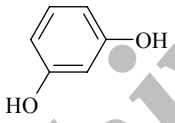
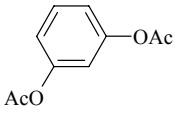
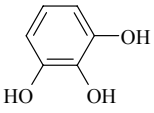
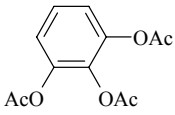
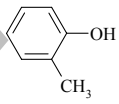
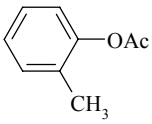
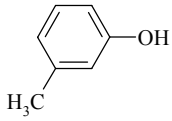
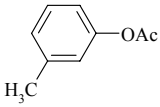
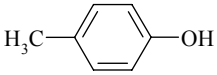
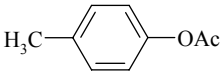
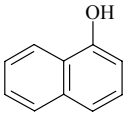
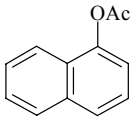
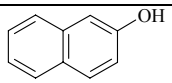
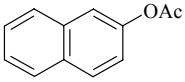
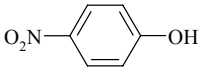
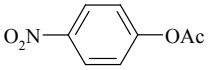
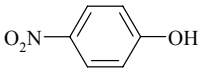
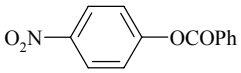
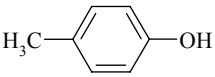
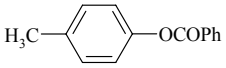
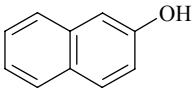
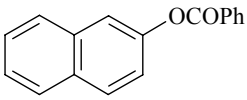
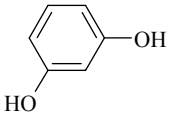
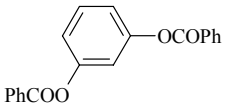
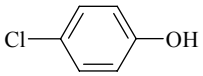
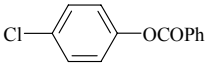
Entry	Phenol	Product	Time (min)	Yield (%)[b]
1			1	100
2			1	100
3			1	100
4			10	97
5			1	100
6			1	100
7			1	100
8			1	100
9			1	100
10			1	100
11			1	100
12			1	100

Table 2. Continued

13			1	100
14			1	100
15			15	85
16			5	94
17			5	90
18			5	95
19			5	95

[a]Reaction conditions: phenol (1.0 mmol), catalyst (0.5 mol%), Ac_2O or $(PhCO)_2O$ (2 equivalents per OH group), CH_3CN (2 ml). [b]GC yield.

Table 3. Acetylation and Benzoylation of Amines with Acetic and Benzoic Anhydrides Catalyzed by $ZrO(OTf)_2$[a]

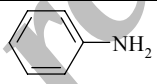
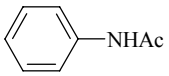
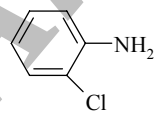
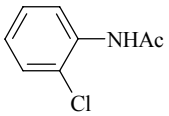
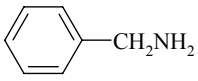
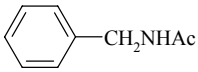
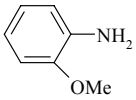
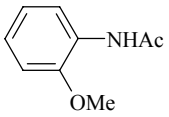
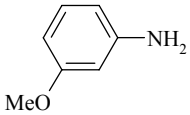
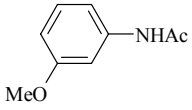
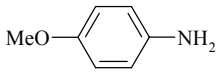
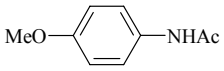
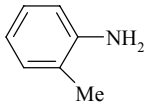
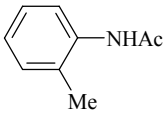
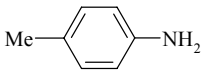
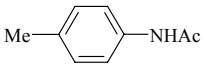
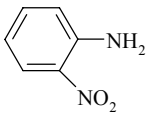
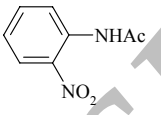
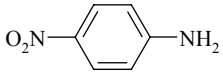
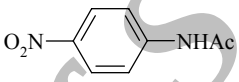
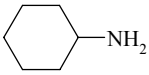
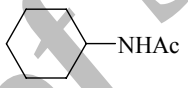
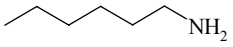
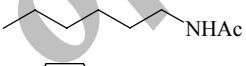
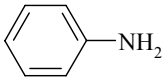
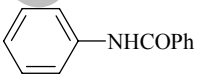
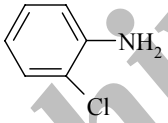
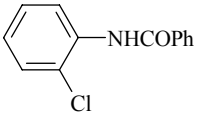
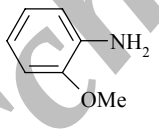
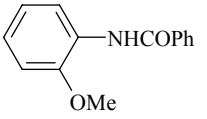
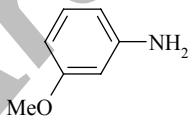
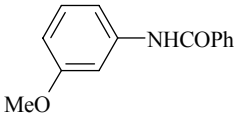
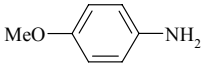
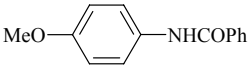
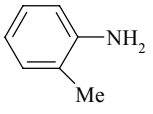
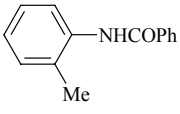
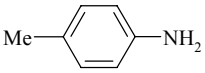
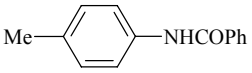
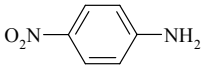
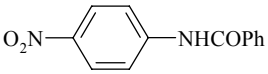
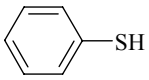
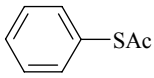
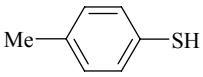
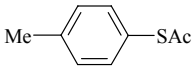
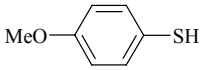
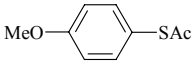
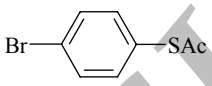
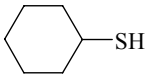
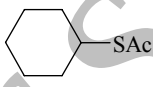
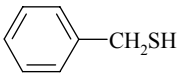
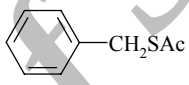
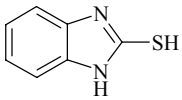
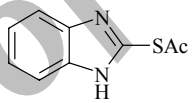
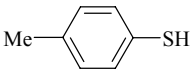
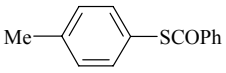
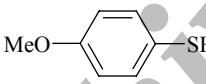
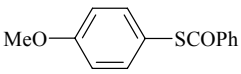
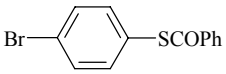
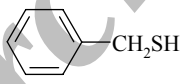
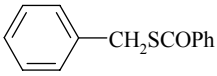
Entry	Amine	Product	Time (min)	Yield (%)[b]
1			1	100
2			1	100
3			1	100
4			1	100
5			1	100

Table 3. Continued

6			1	100
7			1	100
8			1	100
9			3	100
10			1	86
11			5	100
12			5	100
13			1	100
14			1	100
15			60	100
16			30	100
17			1	100
18			80	100
19			35	100
20			5	90

[a]Reaction conditions: amine (1.0 mmol), catalyst (1-2 mol%), Ac_2O or $(PhCO)_2O$ (2 equivalents), CH_3CN (2 ml). [b]GC yield.

Table 4. Acetylation and Benzoylation of Thiols with Acetic and Benzoic Anhydride Catalyzed by $ZrO(OTf)_2$[a]

Entry	Thiol	Product	Time (min)	Yield (%)[b]
1			3	100
2			3	100
3			3	100
4			4	100
5			8	88
6			1	100
7			15	80
8			15	100
9			15	100
10			15	100
11			15	100

[a]Reaction conditions: thiol (1.0 mmol), catalyst (2 mol%), Ac_2O or $(PhCO)_2O$ (2 equivalents), CH_3CN (2 ml).
[b]GC yield.

at 50 °C and in the presence of 2 mol% of $ZrO(OTf)_2$.

CONCLUSIONS

In this paper, we have developed a highly efficient method for acetylation and benzoylation of alcohols, phenols, amines and thiols with acetic and benzoic anhydrides in the presence of catalytic amounts of $ZrO(OTf)_2$. Excellent yields, very short reaction times, reusability and easy work-up are the advantages of the present method.

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