



Electron-deficient $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$: A new and highly efficient catalyst for the acetylation of alcohols and phenols with acetic anhydride

Maryam Yadegari^a, Majid Moghadam^{b,*}, Shahram Tangestaninejad^b, Valiollah Mirkhani^b, Iraj Mohammadpoor-Baltork^b

^a Department of Chemistry, Science & Research Branch, Islamic Azad University, Tehran, Iran

^b Department of Chemistry, Catalysis Division, University of Isfahan, Isfahan 81746-7344, Iran

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ABSTRACT

In the present work, a highly efficient method for acetylation of alcohols and phenols with acetic anhydride catalyzed by high-valent $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ is reported. Under these conditions, primary, secondary and tertiary alcohols as well as phenols were acetylated with short reaction times and high yields. The catalyst was reused several times without loss of its catalytic activity.

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1. Introduction

Schiff base complexes of Mn, Fe, Ru, Cr, Co and V have found many applications in organic chemistry [1]. These compounds have been used as catalysts in the oxidation of alkenes, alkanes, sulfides, amines and alcohols. Titanium Schiff bases have been used as catalysts for the polymerization of ethylene and propene [2], regio- and stereoselective epoxidation of allylic alcohols [3], asymmetric ring-opening of epoxides by dithiophosphorus acid [4], enantioselective catalytic ring-opening of epoxides with carboxylic acids [5], efficient kinetic resolution of terminal epoxides by means of catalytic hydrolysis [6], enantioselective trimethylsilylcyanation of aldehyde [7,8], oxidation of sulfides to sulfoxides with hydrogen peroxide [9], enantioselective ring-opening of *meso*-epoxides with ArSH [10], asymmetric alkynylation of aldehydes [11] and enantioselective Pinacol coupling of aryl aldehydes [12].

The protection of hydroxyl groups is often necessary during the course of various transformations in a synthetic sequence, especially in the synthesis of fine chemicals and natural products. Several methods, such as acetylation, tetrahydropyranylation, methoxymethylation and trimethylacetylation, have been reported for the protection of hydroxyl groups [13,14]. A variety of procedures using homogeneous or heterogeneous catalysts such as iodine [15], *p*-toluenesulfonic acid [16], alumina [17], zinc chloride [18], cobalt chloride [19], montmorillonit K-10 and KSF [20], zeolite HSZ-360

[21], zirconium sulfophenyl phosphonate [22], $\text{Sc}(\text{OTf})_3$ [23], TaCl_5 [24], TMSOTf [25], $\text{Cu}(\text{OTf})_2$ [26], $\text{In}(\text{OTf})_3$ [27], magnesium bromide [28], bismuth(III) salts [29], ferric perchlorate adsorbed on silica-gel [30], RuCl_3 [31], InCl_3 [32], $\text{Ce}(\text{OTf})_3$ [33], $\text{Mg}(\text{ClO}_4)_2$ [34], ZrCl_4 [35], Cp_2ZrCl_2 [36], $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ [37], $\text{Al}(\text{OTf})_3$ [38], $\text{NaHSO}_4 \cdot \text{SiO}_2$ [39], $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ [40], NbCl_5 [41], $\text{Gd}(\text{OTf})_3$ [42], Alumina supported MoO_3 [43] cerium polyoxometallate [44], $\text{Zn}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ [45], $\text{Mg}(\text{NTf}_2)_2$ [46], $\text{Cu}(\text{BF}_4)_2$ [47], $\text{BiO}(\text{ClO}_4)_2$ [48], $\text{HClO}_4 \cdot \text{SiO}_2$ [49], $\text{HBF}_4 \cdot \text{SiO}_2$ [50], $\text{ZrO}(\text{OTf})_2$ [51], $\text{Cp}_2\text{Ti}(\text{OSO}_2\text{C}_8\text{F}_{17})_2$ [52], $\text{Cp}_2\text{Zr}(\text{O}-\text{SO}_2\text{C}_8\text{F}_{17})_2$ [53] and electron-deficient tin (IV) porphyrins [54–56] and have been routinely reported for the acetylation of alcohols and phenols with Ac_2O . Although these procedures provide an improvement, many of these catalysts or activators need long reaction times, drastic reaction conditions or tedious workups, and the catalysts are moisture sensitive or expensive. Hence new procedures to circumvent these problems are still in demand.

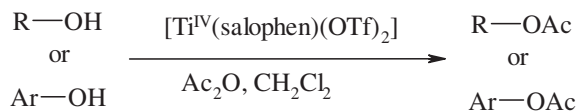
Electron-deficient complexes of Fe, Cr and Sn have been used as mild Lewis acids in organic transformations [54–70]. In this paper we report a rapid and highly efficient method for the acetylation of alcohols and phenols with acetic anhydride catalyzed by high-valent titanium(IV) salophen trifluoromethanesulfonate, $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$, at room temperature (Scheme 1).

2. Experimental

Chemicals were purchased from Merck or Fluka chemical companies. FT-IR spectra were obtained with potassium bromide pel-

* Corresponding author.

E-mail address: moghadamm@sci.ui.ac.ir (M. Moghadam).



Scheme 1. Acetylation of alcohols and phenols with Ac_2O catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$.

lets in the range 400–4000 cm^{-1} with a Nicolet Impact 400D spectrometer. Gas chromatography experiments (GC) were performed with a Shimadzu GC-16A instrument using a 2 m column packed with silicon DC-200 or Carbowax 20M. In the GC experiments, *n*-decane was used as an internal standard. ^1H NMR spectra were recorded on a Bruker-Avance AQS 400 MHz spectrometer.

2.1. The preparation of the catalyst

2.1.1. Preparation of $[\text{Ti}^{\text{IV}}(\text{salophen})\text{Cl}_2]$

In a typical procedure, a solution of $\text{TiCl}_4(\text{THF})_2$ (669 mg, 2.0 mmol) in dry THF (10 mL) was added slowly to a solution of $\text{H}_2\text{Salophen}$ (633 mg, 2.0 mmol) in THF (10 mL), resulting in a red-brown solution. The reaction mixture was stirred and refluxed at 70 °C for 1 h, then cooled to room temperature, and the solvent was evaporated. The solid was slurried with Et_2O (20 mL), filtered through a fine-fritted funnel, washed with additional Et_2O , and dried under vacuum at 80 °C for 2 h [71].

2.1.2. Preparation of $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$

To a solution of $[\text{Ti}^{\text{IV}}(\text{salophen})\text{Cl}_2]$ (434 mg, 1 mmol) in CH_2Cl_2 (15 mL) was added an acetonitrile solution (15 mL) of AgOTf (513.9 mg, 2 mmol), producing a brown precipitate. The AgCl that formed was filtered off through a fine fritted funnel, and the filtrate was concentrated to dryness. The solid was extracted with CH_2Cl_2 and the resulting solid was isolated [71].

^1H NMR (400 MHz, CD_3OD) δ : 9.44 (s, 2H, H-C=N), 8.10 (dd, 2H, Ar, $^1J = 6.2$, $^2J = 3.2$ Hz), 7.95 (d, 2H, Ar, $J = 7.6$ Hz), 7.72 (m, 2H, Ar), 7.64 (dd, 2H, Ar, $^1J = 6.4$, $^2J = 3.2$ Hz), 7.18 (m, 2H, Ar), 6.96 (d, 2H, Ar, $J = 8$ Hz); ^{13}C NMR (CD_3OD) δ : 116.53, 117.24 (CH), 117.58

(O-C-C), 121.24 (CF_3), 123.62, 130.41, 136.37, 138.10 (CH), 141.46 (N-C), 160.56 (O-C), 161.64 (N=CH).

2.2. General procedure for the acetylation of alcohols and phenols with Ac_2O catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$

To a solution of alcohol or phenol (1 mmol) and Ac_2O (3 mmol per OH group) in CH_2Cl_2 (1 mL) was added $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ (2 mol%) and the resulting mixture was stirred at room temperature. The progress of the reaction was monitored by GC. After completion of the reaction, the solvent was evaporated, Et_2O (10 mL)

Table 1

Optimization of the catalyst amount in the acetylation of 4-chlorobenzyl alcohol with Ac_2O catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ at room temperature.^a

Entry	Catalyst amount (%mol)	Solvent	Time (min)	Yield (%) ^b
1	1	CH_3CN	8	56
2	2	CH_3CN	8	71
3	3	CH_3CN	8	95
4	1	CH_2Cl_2	1	77
5	2	CH_2Cl_2	1	100
6	3	CH_2Cl_2	1	100
7	2	<i>n</i> -Hexane	1	18
8	2	CHCl_3	1	25

^a Reaction conditions: alcohol (1 mmol), acetic anhydride (3 mmol), solvent (1 mL).

^b GC yield.

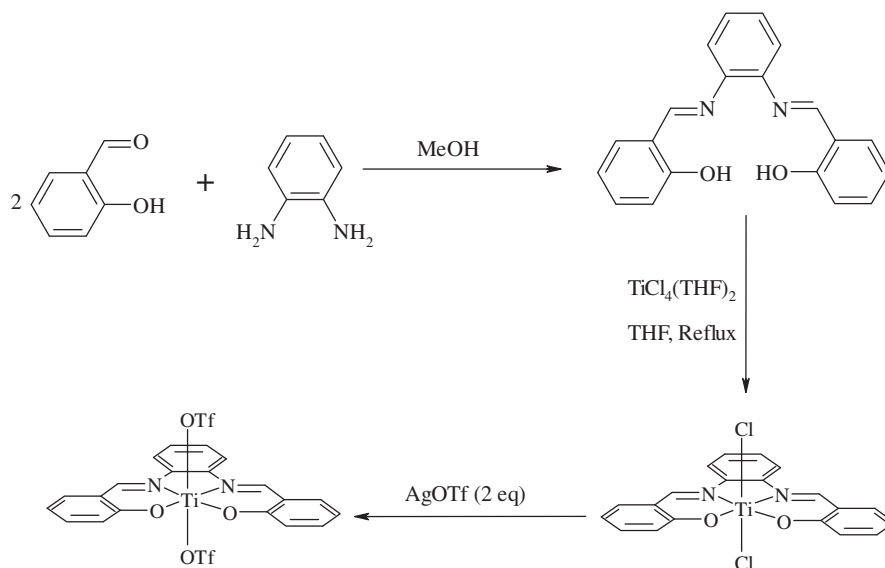
Table 2

Optimization of the Ac_2O amount in the acetylation of 4-chlorobenzyl alcohol.^a

Entry	Time (min)	Ac_2O (mmol)	Yield (%) ^b
1	1	0.5	18
2	1	1.0	34
3	1	1.5	54
4	1	2.0	72
5	1	2.5	91
6	1	3.0	100

^a Reaction conditions: alcohol (1 mmol), Ac_2O , catalyst (2 mol%), CH_2Cl_2 (1 mL).

^b GC yield.



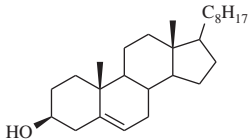
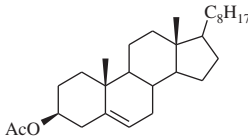
Scheme 2. The preparation route for the catalyst.

Table 3
Acetylation of alcohols with acetic anhydride catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ at room temperature.^a

Entry	Hydroxy compound	Ester	Time (min)	Yield (%) ^b
1			1	100
2			1	100
3			1	100
4			1	97
5			1	100
6			1	100
7			1	100
8 ^c			1	93
9 ^c			1	95
10			1	100
11			1	98
12			1	98
13			1	95
14			1	100
15			1	100
16			1	100
17			1	100
18			1	100
19			10	100
20			1	100
21			1	90
22			1	100

(continued on next page)

Table 3 (continued)

Entry	Hydroxy compound	Ester	Time (min)	Yield (%) ^b
23			5	81

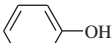
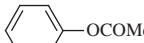
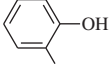
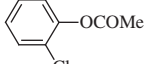
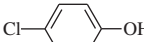
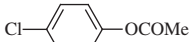
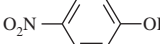
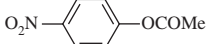
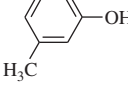
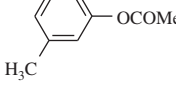
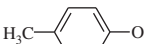
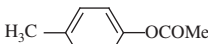
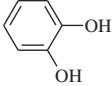
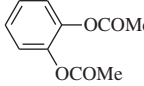

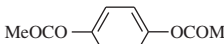
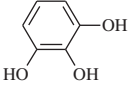
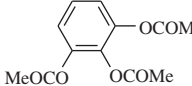


^a Reaction conditions: alcohol (1 mmol), acetic anhydride (3 mmol), catalyst (2 mol%), CH₂Cl₂ (1 mL).

^b GC yield.

^c The reaction was performed with 6 mmol of acetic anhydride.

Table 4

Acetylation of phenols with acetic anhydride catalyzed by [Ti^{IV}(salophen)(OTf)₂] at room temperature.^a

Entry	Phenol	Ester	Time (min)	Yield (%) ^b
1			1	100
2			1	100
3			1	100
4			7	98
5			1	97
6			1	99
7 ^c			1	100
9 ^c			1	100
10 ^c			4	95
11			2	100

^a Reaction conditions: phenol (1 mmol), acetic anhydride (3 mmol), catalyst (2 mol%), CH₂Cl₂ (1 mL).

^b GC yield.

^c The reaction was performed with 3 mmol of acetic anhydride per OH group.

was added and the catalyst was filtered. The filtrates were washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure to afford the crude product.

2.3. Selected spectral data

Acetic acid 4-acetoxy-benzyl ester (Table 3, entry 8), ¹H NMR (CDCl₃), δ: 7.41 (d, 2H, Ar, *J* = 8.8 Hz), 7.11 (d, 2H, Ar, *J* = 8.4 Hz), 5.11 (s, 2H, CH₂), 2.30 (s, 3H, CH₃), 2.10 (s, 3H, CH₃); IR (liquid) ν_{C=O}: 1740 and 1746 cm⁻¹.

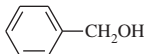
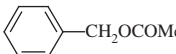
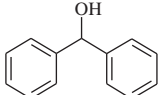
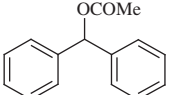
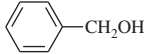
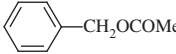
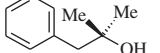
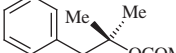
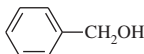
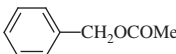
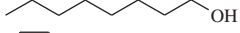

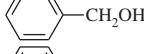
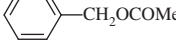
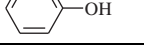
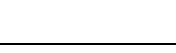
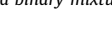
Acetic acid 2-acetoxymethyl-phenyl ester (Table 3, entry 9), ¹H NMR (CDCl₃), δ: 7.46 (d, 1H, Ar, *J* = 7.2 Hz), 7.385 (t, 1H, Ar, *J* = 7.8 Hz), 7.27 (t, 1H, Ar, *J* = 7.4 Hz), 7.12 (d, 1H, Ar, *J* = 8.0 Hz), 5.10 (s, 2H, CH₂), 2.34 (s, 3H, CH₃), 2.81 (s, 3H, CH₃); IR (liquid) ν_{C=O}: 1738 and 1745 cm⁻¹.

3. Results and discussion

3.1. Acetylation of alcohols and phenols with Ac₂O catalyzed by [Ti^{IV}(salophen)(OTf)₂]

The preparation route for the [Ti^{IV}(salophen)(OTf)₂] catalyst is shown in Scheme 2. First, in order to show the effect of the OTf groups on the catalytic activity of [Ti^{IV}(salophen)(OTf)₂], the catalytic activities of [Ti^{IV}(salophen)Cl₂], [Ti^{IV}(salophen)(OPh)₂] and [Ti^{IV}(salophen)(OTf)₂] were investigated for the acetylation of 4-chlorobenzyl alcohol with acetic anhydride. The results showed that the catalytic activity of these catalysts is in the following order: [Ti^{IV}(salophen)(OTf)₂] (100%) > [Ti^{IV}(salophen)Cl₂] (57%) > [Ti^{IV}(salophen)(OPh)₂] (32%). The results clearly show that introducing the OTf groups on Ti(salophen) increases the electron-deficiency of

Table 5
Selective acetylation of alcohols and phenols catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ in CH_2Cl_2 .^a

Row	ROH	ROCOMe	Time (min)	Yield (%) ^b
1			1	100
				0
2			1	100
				0
3			1	90
				17
				100
4			1	0
				

^a Reaction conditions for a binary mixture: 1 mmol of each alcohol or phenol, acetic anhydride (3 mmol), catalyst (2 mol%), CH_2Cl_2 (1 mL).

^b GC yield.

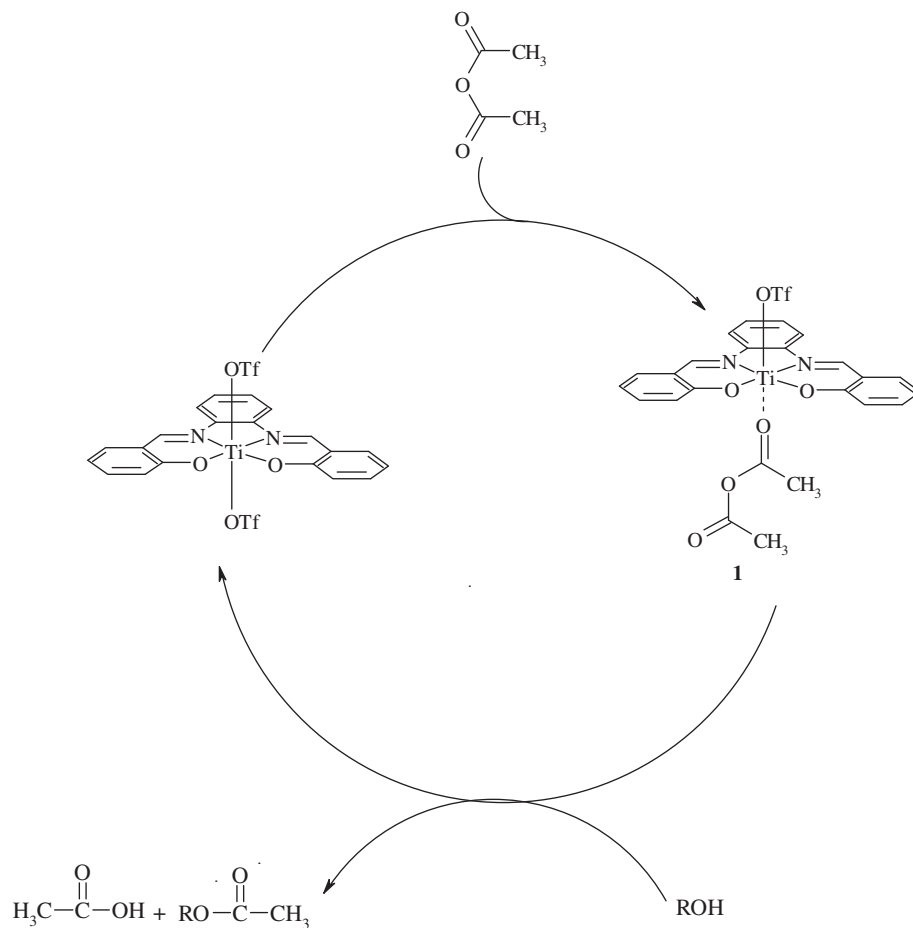
Table 6
Comparison of the results obtained for the acetylation of benzyl alcohol catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ with those obtained by recently reported catalysts.

Entry	Catalyst	Catalyst (mol%)	T (°C)	Time (min)	Yield (%)	Ref.
1	$[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$	2	r.t.	1	100	
2	I_2	10	r.t.	1	99	[15]
3	CoCl_2	0.5	r.t.	240	98	[19]
4	Montmorillonite KSF	20 mg	r.t.	60	90	[20]
5	Zeolite HSZ-360	20 mg	60	60	84	[21]
6	TaCl_5	10	r.t.		77	[24]
7	$\text{Cu}(\text{OTf})_2$	2	r.t.	30	97	[26]
8	$\text{In}(\text{OTf})_3$	0.1	r.t.	15	97	[27]
9	BiCl_3	10	r.t.	35	98	[29]
	$\text{Bi}(\text{TFA})_3$	5	r.t.	60	96	
	$\text{Bi}(\text{OTf})_3$	1	r.t.	5	99	
10	RuCl_3	5	r.t.	10	95	[31]
11	InCl_3	0.1	r.t.	30	85	[32]
12	$\text{Ce}(\text{OTf})_3$	1	r.t.	12	98	[33]
13	$\text{Mg}(\text{ClO}_4)_2$	1	r.t.	15	100	[34]
14	Cp_2ZrCl_2	1	r.t.	600	93	[36]
15	$\text{ZrO}(\text{OTf})_2$	1	r.t.	0.5	100	[51]
16	$[\text{Sn}(\text{TPP})(\text{OTf})_2]$	1	r.t.	1	99	[53]
17	$[\text{Sn}(\text{TPP})(\text{BF}_4)_2]$	1	r.t.	1	99	[54]

the catalyst, which in turn increases the catalytic activity of $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ in the acetylation of alcohols with Ac_2O . The reaction parameters, such as the amount of catalyst and kind of solvent, were optimized in the reaction of 4-chlorobenzyl alcohol with acetic anhydride (Table 1). In the case the catalyst amount, the results showed that highest yield was obtained in the presence of 2 mol% of $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$. For optimization of the reaction media, the same reaction was performed in different solvents. Among CH_2Cl_2 , CH_3CN , *n*-hexane and chloroform, the highest yield was observed in CH_2Cl_2 . Since the catalyst has a heterogeneous nature in CH_2Cl_2 , the catalyst reusability in this solvent is simpler, so dichloromethane was used as the solvent. The amount of acetic anhydride was also optimized and the best result was obtained with a 3:1 M ratio of acetic anhydride to alcohol, whereas with lower amounts of Ac_2O , lower yields were obtained (Table 2). The model reaction was also carried out with acetic acid and ethyl acetate as acylating agents, and only 47% and 21% of the corresponding acetate were produced, respectively.

Under the optimized reaction conditions, a wide range of alcohols, including primary (benzylic and linear ones), secondary and tertiary alcohols, were successfully acetylated with Ac_2O in the presence of $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$. In the case of benzylic alcohols, the nature of the substituent has no significant effect on the yields and reaction times (Table 3). In the case of bifunctional compounds, such as 2-hydroxybenzyl alcohol and 4-hydroxybenzyl alcohol, both hydroxy groups were acetylated completely (Table 3, entries 8 and 9). As an example of a complicated alcohol, the acetylation of cholesterol was investigated and the corresponding acetate was obtained in 81% yield (Table 3, entry 23). The acetylation of phenols was also investigated and the corresponding acetates were produced in high yields (Table 4).

Since this catalyst was highly active in the acetylation of alcohols and phenols, we decided to investigate the chemoselectivity of the presented method. With this in mind, a set of competitive reactions was conducted between a primary alcohol and a secondary or tertiary alcohols, or phenol (Table 5). The results indicated



Scheme 3. The proposed mechanism for acetylation of alcohols and phenols with Ac_2O catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$.

that the primary alcohol is more reactive in the presence of secondary and tertiary alcohols and phenol. It is important to note that in the competitive acetylation of benzyl alcohol and phenol (Table 5, entry 4), after a very short reaction time (1 min) only benzyl alcohol was acetylated, whereas with longer reaction times (5 min), the phenol was acetylated in 53%.

In order to show the advantage of the presented method in the acetylation reactions, we have compared the obtained results in the acetylation of benzyl alcohol with acetic anhydride catalyzed by $[\text{Ti}^{\text{IV}}(\text{salophen})(\text{OTf})_2]$ with some results reported in the literature (Table 6). It is clear that the presented method can be considered as an efficient method for acetylation reactions.

The actual mechanism is not clear at present. However, due to the high oxophilicity of Ti, a plausible explanation is that acetic anhydride is first activated by the catalyst to afford **1**. The hydroxy compound attacks **1** which in turn converts to the final product and releases the catalyst for the next catalytic cycle (Scheme 3).

3.2. Catalyst reusability

Finally, the reusability of the catalyst was tested in the reaction of 4-chlorobenzyl alcohol as a model substrate. It was observed that the activity of catalyst did not decrease after four consecutive times.

4. Conclusion

In conclusion, in this paper a mild and efficient method for the acetylation of alcohols and phenols is reported. This catalytic

system showed a good catalytic activity in these reactions. Other advantages of this catalyst are the easy preparation of the catalyst, short reaction times, high yields and the reusability of the catalyst.

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