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Highly efficient and selective acetylation of alcohols and phenols with acetic anhydride catalyzed by a high-valent tin(IV) porphyrin, $Sn(TPP)(BF_4)_2$

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Abstract

Rapid and efficient acetylation of alcohols and phenols with acetic anhydride in the presence of tin(IV) tetraphenylporphyrinato tetrafluoroborate, $Sn^{IV}(tpp)(BF_4)_2$, as catalyst is reported. The high catalytic activity of this electron-deficient tin(IV) porphyrin can be used to assist the acetylation of not only primary alcohols but also sterically hindered secondary and tertiary alcohols with acetic anhydride. Acetylation of phenols with acetic anhydride was achieved to afford the desired esters efficiently. This catalyst selectively acetylates the alcohols and phenols in the presence of acetals and silyl ethers.

Keywords: Acetylation; Alcohol; Phenol; Electron-deficient porphyrin; Acetic anhydride

1. Introduction

The protection of hydroxyl groups of alcohols and phenols by the formation of esters is one of the most important and widely used transformations in organic chemistry [1]. 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. A variety of procedures are routinely performed for the preparation of acetyl derivatives, including homogeneous or heterogeneous catalysts such as 4-(dimethylamino)pyridine and 4-pyrolidinopyridine [2], *N*,*N*,*N*′,*N*′-tetramethylethylenediamine [3], tributylphosphine [4], iodine [5], *p*-toluenesulfonic acid [6], alumina [7], zinc chloride [8], cobalt chloride [9], montmorillonit K-10 and KSF [10], zeolite HSZ-360 [11], zirconium sulfophenyl phosphonate [12], $[Sc(OTf)_3]$ [13], $TaCl_5$ [14], trimethylsilyl trifluoromethanesulfonate (TMSOTf) [15], $[Cu(OTf)_2]$ [16], $[In(OTf)_3]$ [17], magnesium bromide [18], bismuth(III) salts [19], ferric perchlorate adsorbed on silica-gel [20], $RuCl_3$ [21], $InCl_3$ [22], $Ce(OTf)_3$ [23], $Mg(ClO_4)_2$ [24], $ZrCl_4$ [25], Cp_2ZrCl_2 [26], and cerium polyoxometalate [27]. 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 of esters is still in demand.

Electron-deficient metalloporphyrins have been used as mild Lewis acids catalysts [28]. Recently, the Suda group reported the use of chromium and iron porphyrins in organic synthesis. They used Cr(tpp)Cl for regioselective [3,3] rearrangement of aliphatic allyl vinyl ethers and for Claisen rearrangement of simple aliphatic allyl vinyl ethers, Fe(tpp)OTf for rearrangement of α,β-epoxy ketones into 1,2-diketones and Cr(tpp)OTf for highly regio- and stereoselective rearrangement of epoxides to aldehydes [29].

This features prompted us to explore the ability of new electron-deficient metalloporphyrins in organic transformations. Previously, we reported the use of tin(IV) tetraphenylporphyrin perchlorate [30] and tin(IV) tetraphenylporphyrin trifluorome-

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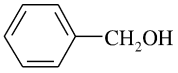
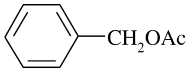
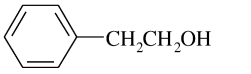
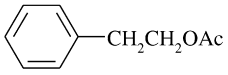
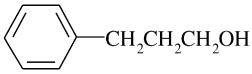
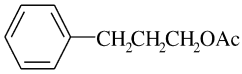
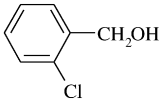
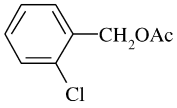
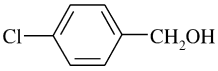
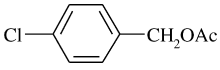
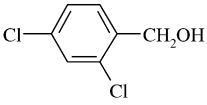
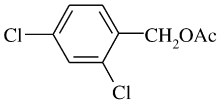
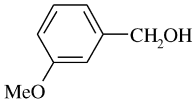
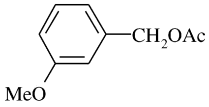
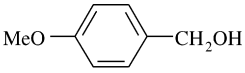
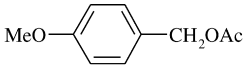
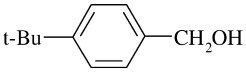
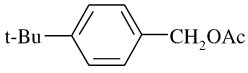
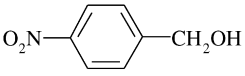
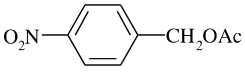
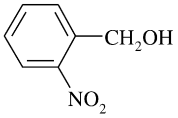
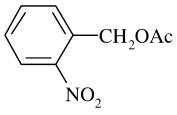
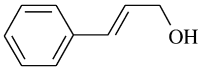
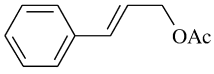
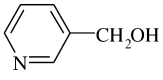
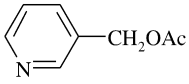
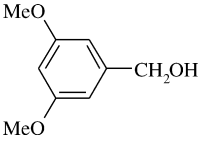
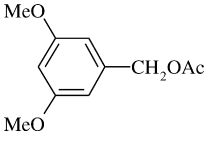
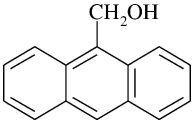
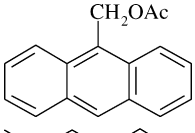
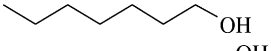
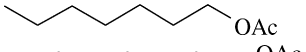
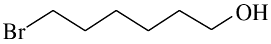
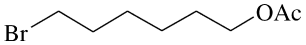
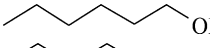
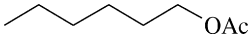
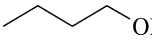
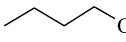
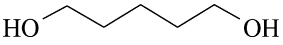
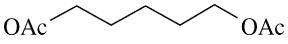
Table 1
Acetylation of alcohols with Ac_2O catalyzed by $Sn^{IV}(tpp)(BF_4)_2$ at room temperature[a]

Entry	Substrate	Product	Time (min)	Yield (%)[b]
1			1	99
2			1	95
3			1	99
4			1.5	99
5			1.5	99
6			2	99
7			1	98
8			1	97
9			2	97
10			1	99
11			1	99
12			2	96
13			1	99
14			1	98
15			5	99
16			1	99
17			1	97
18			1	99
19			1	99
20			2	97
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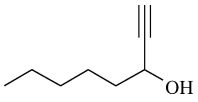
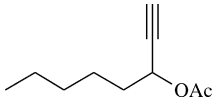
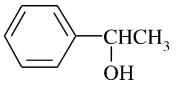
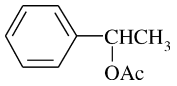
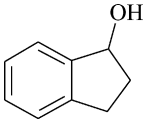
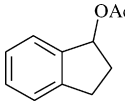
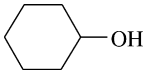
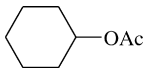
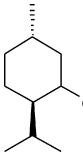
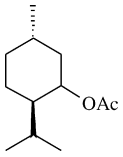
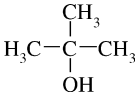
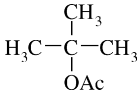
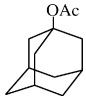
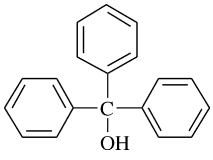
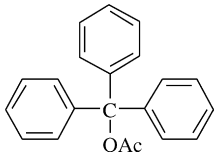
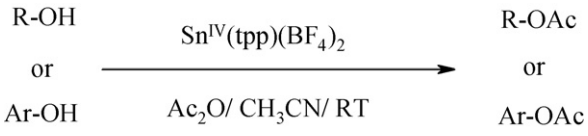
22			2	95
23			2	96
24			10	99
25			1	99
26			5	98
27			1	99
28			10	99
29			15	99

[a] Reaction conditions: alcohol, 1.0 mmol; catalyst, 0.01 mmol, 10 mg; Ac_2O, 2 equiv. per OH group; CH_3CN, 2 mL.
[b] GC yield based on the starting alcohol.



Scheme 1.

thanesulfonate [31] in the acetylation of alcohols and phenols.

Here, we report the use of $Sn^{IV}(tpp)(BF_4)_2$ for selective acetylation of alcohols and phenols with acetic anhydride at room temperature (Scheme 1).

2. Results and discussions

2.1. Acetylation of alcohols with acetic anhydride catalyzed by $Sn^{IV}(tpp)(BF_4)_2$

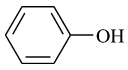
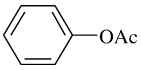
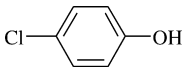
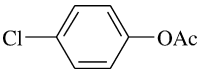
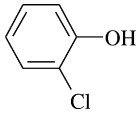
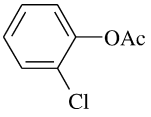
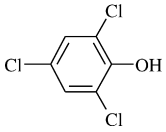
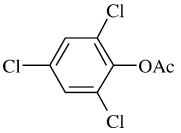
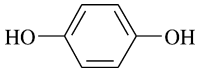
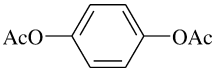
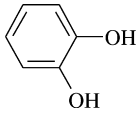
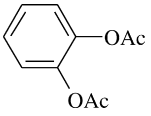
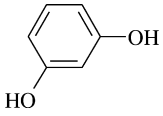
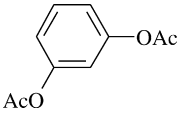
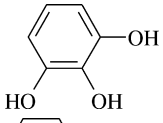
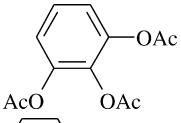
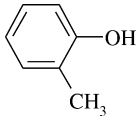
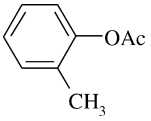
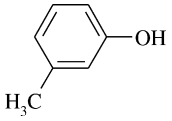
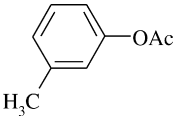
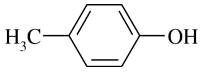
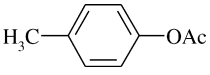
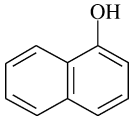
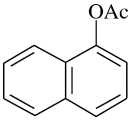
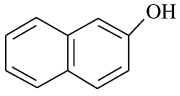
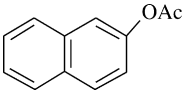
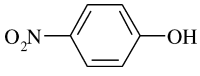
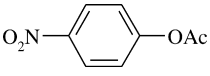
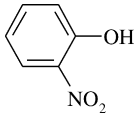
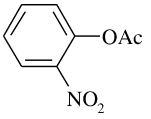
First, we investigated the ability of $Sn^{IV}(tpp)(BF_4)_2$ in the acetylation of 4-methoxybenzyl alcohol (1 mmol), in which this alcohol was converted completely to the desired acylated product with acetic anhydride (2 equiv.), at room temperature, in the presence of 1 mol% of $Sn^{IV}(tpp)(BF_4)_2$ as catalyst in the CH_3CN as solvent. The reaction was immediately completed for nearly all alcohols and no alcohol was detected by TLC or GC. However, indanol, 1-adamantanol, triphenylmethanol and 9-anthracenylmethanol required somewhat longer reaction time. In the absence of $Sn^{IV}(tpp)(BF_4)_2$ catalyst, the reaction was much less efficient for the conversion of alcohols to esters. As shown in Table 1, this catalyst can be used for the acetylation of a wide range of primary (including aliphatic and aromatic alcohols) and secondary alcohols with acetic anhydride in the presence of 0.01 molar equivalent of $Sn^{IV}(tpp)(BF_4)_2$ at room temperature to afford the desired acetates in high yields. Tertiary alcohols such as 1-adamantanol, *t*-butyl alcohol, and triphenylmethanol were also acetylated in high yields, without the formation of dehydration products (entries 27–29).

In the case of benzylic alcohols, the substituents had no significant effect on the acetylation yield.

2.2. Acetylation of phenols with acetic anhydride catalyzed by $Sn^{IV}(tpp)(BF_4)_2$

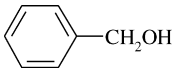
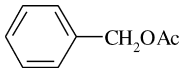
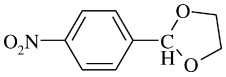
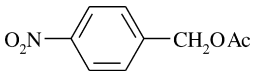
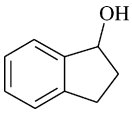
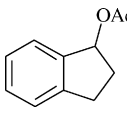
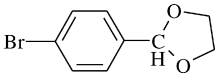
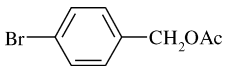
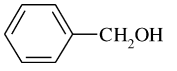
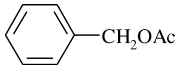
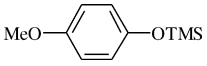
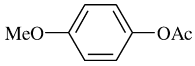
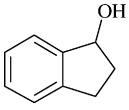
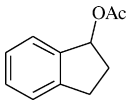
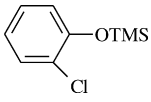
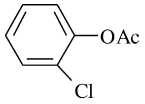
Then, the ability of this catalyst in the acetylation of phenols with acetic anhydride was investigated. The reaction of phenols

Table 2
Acetylation of phenols with Ac_2O catalyzed by $Sn^{IV}(tpp)(BF_4)_2$ [a]

Entry	Substrate	Product	Time (min)	Yield (%)[b]
1			2	99
2			1	99
3			1	99
4			1	99
5			1	99
6			2	99
7			2	99
8			1	99
9			1	99
10			1	99
11			1	99
12			1	99
13			1	99
14			1	99
15			20	88

[a] Reaction conditions: phenol, 1.0 mmol; catalyst, 0.01 mmol, 10 mg; Ac_2O, 2 equiv. per OH group; CH_3CN, 2 mL.
[b] GC yield based on the starting phenol.

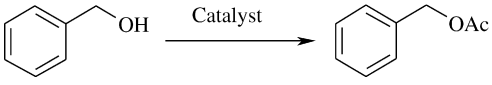
Table 3
Competitive acetylation of alcohols, acetals and silyl ethers catalyzed by $Sn^{IV}(tpp)(BF_4)_2$ [a]

Run	Substrate	Product	Time (min)	Yield (%)[b]
1			1	99
			1	0
2			10	99
			10	0
3			1	99
			1	0
4			10	99
			10	0

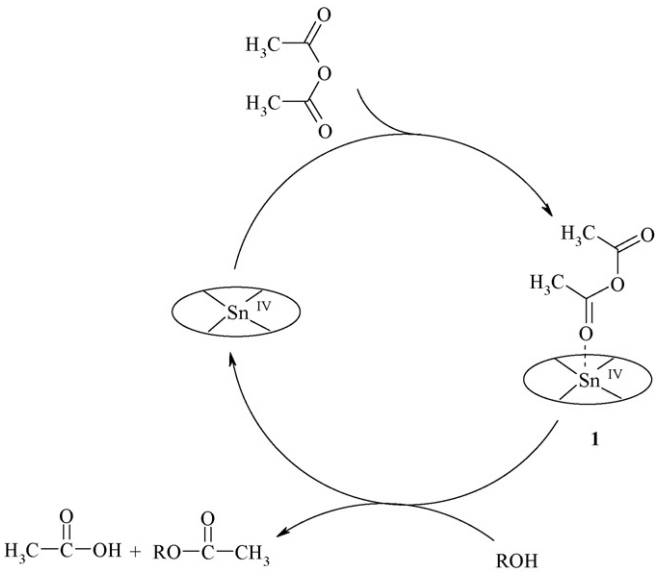
[a] Reaction conditions: substrate, 1.0 mmol; catalyst, 0.01 mmol, 10 mg; Ac_2O, 2 equiv. per OH group; CH_3CN, 2 mL.
[b] GC yield based on the starting substrate.

with acetic anhydride (2 equiv. for each hydroxyl group) in the presence of 0.01 molar equivalent of the catalyst in acetonitrile (2 mL), as solvent, was carried out, and the desired acetates were obtained in excellent yields at 50 °C (Table 2). The acetylation of polyhydroxybenzenes such as hydroquinone, pyrocatechol, resorcinol and pyrogallol was also achieved 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.

Table 4
Comparison of the results obtained for the acetylation of benzyl alcohol catalyzed by $Sn(tpp)(BF_4)_2$ with those obtained by the recently reported catalysts



Entry	Catalyst	Catalyst (mol%)	Temperature	Time (min)	Yield (%)	Ref.
1	$Sn(tpp)(BF_4)_2$	1	r.t.	1	99	–
2	I_2	10	r.t.	1	99	[5]
3	$CoCl_2$	0.5	r.t.	240	98	[9]
4	Montmorillonite KSF	20 mg	r.t.	60	90	[10]
5	Zeolite HSZ-360	20 mg	60 °C	60	84	[11]
6	$TaCl_5$	10	r.t.	–	77	[14]
7	$Cu(OTf)_2$	2	r.t.	30	97	[16]
8	$In(OTf)_3$	0.1	r.t.	15	97	[17]
	$BiCl_3$	10	r.t.	35	98	
9	$Bi(TFA)_3$	5	r.t.	60	96	[19]
	$Bi(OTf)_3$	1	r.t.	5	99	
10	$RuCl_3$	5	r.t.	10	95	[21]
11	$InCl_3$	0.1	r.t.	30	85	[22]
12	$Ce(OTf)_3$	1	r.t.	12	98	[23]
13	$Mg(ClO_4)_2$	1	r.t.	15	100	[24]
14	Cp_2ZrCl_2	1	r.t.	600	93	[26]



Scheme 2.

The actual mechanism is not clear at present. However, a plausible explanation is that acetic anhydride is first activated by catalyst to afford **1**. Alcohol or phenol attacks **1** which in turn converts to the final product and releases the catalyst for the next catalytic cycle (Scheme 2).

In comparison with the data reported with $Sn^{IV}(tpp)(ClO_4)_2$ catalyst [30], these results exhibit that $Sn^{IV}(tpp)(BF_4)_2$ is a more powerful catalyst for the acetylation of alcohols and phenols, and the data is comparable with those of $Sn^{IV}(tpp)(CF_3SO_3)_2$ [30]. The selectivity of this method was checked by competitive acetylation of alcohols with acetals and silyl ethers. The results showed that alcohols were acetylated selectively in the presence of acetals and silyl ethers (Table 3). This may be considered as a useful practical achievement in esterification reactions.

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 $Sn^{IV}(tpp)(BF_4)_2$ with some of those reported in the literature (Table 4). It is clear that the presented method is superior in terms of reaction time, catalyst amount, or product yield.

3. Conclusion

Although metalloporphyrins are widely used in the oxidation reactions as catalysts, there have been few studies on their catalytic activity as Lewis acids. In this report, we have demonstrated that the tin(IV) tetraphenylporphyrinato tetrafluoroborate, $Sn^{IV}(tpp)(BF_4)_2$, which is a stable Sn(IV) compound, can be considered as a mild Lewis acid for efficient and catalytic acetylation of alcohols and phenols. The advantage of this system is that even hindered substrates can be acetylated with acetic anhydride in high yields at room temperature. These points clearly indicate that replacement of Cl^- with BF_4^- converts the $Sn(tpp)Cl_2$ to an electron-deficient catalyst, which can acts as a super Lewis acid in organic transformations. In addition, short reaction times and ease of preparation of the catalyst make this method a useful catalytic system for acetylation reactions.

4. Experimental

Chemicals were purchased from Fluka and Merck chemical companies. ^1H NMR spectra were recorded in $CHCl_3$ on a Bruker AM 80 MHz spectrometer using TMS as an internal standard. Infrared spectra were run on a Philips PU9716 or Shimadzu IR-435 spectrophotometer. All analyses were performed on a Shimadzu GC-16A instrument with a flame ionization detector using silicon DC-200 or Carbowax 20-M columns.

Tetraphenylporphyrin was prepared and metallated according to the literature [32,33].

4.1. Preparation of tin(IV) tetraphenylporphyrinato tetrafluoroborate, $Sn^{IV}(tpp)(BF_4)_2$

To a solution of $Sn(tpp)Cl_2$ (1.03 g, 1.0 mmol) in 100 mL of THF, at 55 °C, $AgBF_4$ (0.39 g, 2.0 mmol) was added. The solution was stirred at 55 °C for 30 min. The AgCl precipitate was filtered through a 0.45 μm filter. The resulting solution was evaporated moderately. $Sn^{IV}(tpp)(BF_4)_2$ was then extracted with CH_2Cl_2. The $Sn^{IV}(tpp)(BF_4)_2$ crystals was obtained by evaporation of solvent at room temperature. Visible spectrum: 418 (Soret), 553, 592, 623, 654 nm; CHN analyses: Calcd. C, 58.39; H, 3.11; N, 6.19; found: C, 57.45; H, 3.24; N, 6.35.

4.2. General procedure for the acetylation reactions

In a round-bottom flask (25 mL) equipped with a magnetic stirrer, a solution of alcohol or phenol (1.0 mmol) in Ac_2O (2 equiv. for each OH group of alcohol or phenol) and CH_3CN (2 mL) was prepared. $Sn^{IV}(tpp)(BF_4)_2$ (0.010 g, 0.01 mmol) was added to this solution and the reaction mixture was stirred at room temperature (the reaction of phenols carried out at 50 °C). The reaction progress was monitored by GC. After completion of the reaction, the mixture was directly passed through a short column of silica-gel (hexane:ethyl acetate = 1:1) to remove the catalyst. The eluate was evaporated under reduced pressure and the remaining residue was purified by silica-gel plate chromatography (eluted with CCl_4:Et_2O = 9:1) to afford the desired ester without any elimination products.

Acknowledgements

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