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Note

Investigation of catalytic activity of high-valent vanadium(IV) tetraphenylporphyrin: A new, highly efficient and reusable catalyst for acetylation of alcohols and phenols with acetic anhydride

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ABSTRACT

In the present work, the catalytic activity of high-valent tetraphenylporphyrinatovanadium(IV) trifluoromethanesulfonate, $[V^{IV}(TPP)(OTf)_2]$, in the acetylation of alcohols and phenols with Ac₂O is reported. This new V(IV) catalyst was used as an efficient catalyst for not only primary alcohols (benzylic and aliphatic) but also sterically-hindered secondary and tertiary alcohols with acetic anhydride and the corresponding acetates were obtained in 85–99% yield and 0.5–15 min. Acetylation of phenols with acetic anhydride was also performed to afford the desired acetates in 88–99% and 1.5–20 min. This catalyst can be reused several times without loss of its catalytic activity.

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1. Introduction

In the last decade, electron-deficient metalloporphyrins have been used as mild Lewis acid catalysts. Suda and coworkers 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 rearrangement of monoalkyl1substituted epoxides into aldehydes, and Cr(tpp)OTf for highly regio- and stereoselective rearrangement of epoxides to aldehydes [1–7].

Recently, we reported the application of tin(IV) porphyrins in organic transformations. These catalysts were used as efficient catalysts for ring-opening of epoxides, and for conversion of alcohols and phenols to trimethylsilyl ethers, tetrahydropyranyl ethers, methoxymethyl ethers and acetates [8–19].

Developing mild and efficient methods for the protection of hydroxyl group of alcohols and phenols is of great importance in synthetic organic chemistry. One of the most common methods for the protection of these compounds is the formation of acetyl derivatives [20,21]. These reactions are of great importance during the

course of various transformations in a synthetic sequence, especially in the synthesis of fine chemicals and natural products. A variety of homogeneous or heterogeneous catalysts such as iodine [22], *p*-toluenesulfonic acid [23], alumina [24], zinc chloride [25], cobalt chloride [26], montmorillonite K-10 and KSF [27], zeolite HSZ-360 [28], zirconium sulfophenyl phosphonate [29], Sc(OTf)₃ [30], TaCl₅ [31], TMSOTf [32], Cu(OTf)₂ [33], In(OTf)₃ [34], magnesium bromide [35], bismuth(III) salts [36], ferric perchlorate adsorbed on silica-gel [37], RuCl₃ [38], InCl₃ [39], Ce(OTf)₃ [40], Mg(ClO₄)₂ [41], ZrCl₄ [42], Cp₂ZrCl₂ [43], ZrOCl₂·8H₂O [44], Al (OTf)₃ [45], NaHSO₄·SiO₂ [46], La(NO₃)₃·6H₂O [47], NbCl₅ [48], Gd(OTf)₃ [49], MoO₃@Al₂O₃ [50] cerium polyoxometalate [51], Zn(ClO₄)₂·6H₂O [52], Mg(NTf₂)₂ [53], Cu(BF₄)₂ [54], BiO(ClO₄)₂ [55], HClO₄-SiO₂ [56], HBF₄-SiO₂ [57], ZrO(OTf)₂ [58], cobalt(II) salen complex [59], zirconocene *bis*(perfluorooctanesulfonate)s [60], CBr₄ [61], phosphomolybdic acid [62], O-benzenedisulfonimide [63] and iron(III) chloride [64] have been routinely reported for acetylation of alcohols and phenols with Ac₂O. Although these procedures provide an improvement, many of these catalysts or activators need long reaction times, drastic reaction conditions or tedious work up, moisture sensitive or expensive of the catalyst. Hence, introducing of new procedures to overcome these problems is still in demand.

Vanadium is required for normal health, and could act *in vivo* either as a metal cation or as a phosphate analog, depending on the oxidation state, V(IV) or V(V), respectively. Vanadium in a sea





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

squirt (tunicate), a primitive vertebrate, is concentrated in blood cells, apparently as the major cellular transition metal, but whether it participates in the transport of dioxygen (as iron and copper do) is not known. In proteins, vanadium is a cofactor in an algal bromoperoxidase and in certain prokaryotic nitrogenases. However, its oxidation state within organisms seems to be highly variable. The biochemistry of vanadium potentially involves four oxidation states that are relatively stable in aqueous solution. These are V^{2+} , V^{3+} , VO^{2+} , and VO_2^+ (the oxidation states 2, 3, 4, and 5, respectively) [65].

Here, we wish to report an efficient method for acetylation of alcohols and phenols with acetic anhydride in the presence of reusable high-valent tetraphenylporphyrinatovanadium(IV) trifluoromethane sulfonate, $[V^{IV}(TPP)(OTf)_2]$, at room temperature (Scheme 1).

2. Experimental

Chemicals were purchased from Fluka and Merck chemical companies. ¹HNMR spectra were recorded in CDCl₃ solvent on a Bruker-Avance 400 MHz spectrometer using TMS as an internal standard. Infrared spectra were run on a Philips PU9716 or Shima-dzu IR-435 spectrophotometer. All analyses were performed on a Shimadzu GC-16A instrument with a flame ionization detector using silicon DC-200 or Carbowax 20M columns. Tetraphenylpor-phyrin, [VO(TPP)] and [V(TPP)Cl₂] were prepared according to the literature [66–68].

2.1. Preparation of tetraphenylporphyrinatovanadium(IV) trifluoromethanesulfonate, $[V^{IV}(TPP)(OTf)_2]$

To a solution of V(TPP)Cl₂ (0.97 g, 1 mmol) in THF (50 mL), at 55 °C, AgCF₃SO₃ (0.54 g, 2 mmol) was added. The solution was stirred at 55 °C for 30 min. The AgCl precipitate was filtered through a 0.45 μ M filter. The resulting solution was evaporated at room temperature. Then, the [V^{IV}(TPP)(OTf)₂] was extracted with CH₂Cl₂ and the [V^{IV}(TPP)(OTf)₂] crystals were obtained after evaporation of solvent at room temperature.

Visible spectrum: 456 (Soret), 585, 628 nm; v (KBr): 1030, 1168, 1224, 1298 cm⁻¹ (belong to SO₃ groups and porphyrin ring); CHNS analyses: Calc. C, 57.44; H, 2.93; N, 5.83; S, 6.67. Found: C, 57.35; H, 2.88; N, 5.91, S, 6.65.

2.2. General procedure for acetylation of alcohol and phenols with Ac_2O catalyzed by $[V^{IV}(TPP)(OTf)_2]$

To a solution of alcohol or phenol (1 mmol) and Ac₂O (3 mmol per OH group) in CH₃CN (0.5 mL) was added $[V^{IV}(TPP)(OTf)_2]$ (0.5 mol% for alcohols and 1 mol% for phenols) and stirred at room temperature. The progress of the reaction was monitored by GC. After completion of the reaction, the solvent was evaporated, Et₂O (10 mL) 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. Catalyst reusability

The reusability of catalyst was checked in the acetylation of 4chlorobenzyl alcohol. At the end of each reaction, the solvent



Scheme 2. Preparation route for catalyst.



Fig. 1. UV-Vis spectrum of [V^{IV}(TPP)(OTf)₂].

was evaporated, $\text{Et}_2\text{O}~(10\text{ mL})$ was added, the catalyst was filtered and reused.

3. Results and discussion

3.1. Acetylation of alcohols and phenols with Ac_2O catalyzed by $[V^{V}(TPP)(OTf)_2]$

Scheme 2 shows the preparation procedure of $[V^{IV}(TPP)(OTf)_2]$. As can be seen, first the $[V^{IV}O(TPP)]$, which is commercially available, was converted to $[V^{IV}(TPP)Cl_2]$ with SOCl₂. The $[V^{IV}(TPP)Cl_2]$ was converted to electron-deficient $[V^{IV}(TPP)(OTf)_2]$ compound.

160

Table 1
Acetylation of alcohols and phenols with acetic anhydride catalyzed by $[V^{IV}(TPP)(OTf)_2]$. ^a

Entry	Substrate	Product	Time (min)	Yield (%) ^{b,c}
1	CH ₂ OH	CH ₂ OAc	0.5	90
2	CH ₂ CH ₂ OH	CH ₂ CH ₂ OAc	0.5	95 (91)
3	CH ₂ CH ₂ CH ₂ OH	CH ₂ CH ₂ CH ₂ OAc	0.5	99 (96)
4	СН_ОН	CH_OAc	0.75	99 (95)
5	CI ————————————————————————————————————	Cl-CH ₂ OAc	0.75	99
6	СІ—СН2ОН	Cl-CH ₂ OAc	1.5	95
7	CH ₂ OH	CH ₂ OAc MeO	0.5	85
8	MeO-CH ₂ OH	MeO-CH ₂ OAc	1	96 (93)
9	t-Bu—CH ₂ OH	t-Bu-CH ₂ OAc	1.5	95
10	O ₂ N — CH ₂ OH	O ₂ N-CH ₂ OAc	1.5	90
11	CH ₂ OH	CH ₂ OAc	1.5	90
12	ОН	OAc	2	95 (90)
13	СH ₂ OH	CH ₂ OAc	1.5	99
14 ^d	CH ₂ OH	CH ₂ OAc	5	99 (94)
15	<i>С С С С С С С С С С</i>	OAc	1	98
16	ОН	OAc	1	99
17	ОН	OAc	1	98
18	CHCH ₃		2	95
19	OH	OAc	6	99
20	~ ОН	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	1	99
21	H ₃ C-C-CH ₃	$H_{3}C - C - CH_{3}$	2	95

(continued on next page)

Table 1 (continued)

Entry	Substrate	Product	Time (min)	Yield (%) ^{b,c}
22 ^d	OH	OAc	1	98 (96)
23 ^d			15	95

^a Reaction conditions: alcohol (1 mmol), catalyst (0.005 mmol, 5 mg), Ac₂O (3 equivalents per OH group), CH₃CN (0.5 mL).
 ^b GC yield based on the starting alcohol.
 ^c The yields in the parentheses refer to isolated products.

^d These reactions were carried out at 50 °C.

Table 2

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Acetylation of phenols with Ac₂O catalyzed by $[V^{IV}(TPP)(OTf)_2]$.^a

Entry	Substrate	Product	Time (min)	Yield (%) ^b
1	ОН	OAc	3	97
2	СІ—ОН	Cl-OAc	2	99
3	СІ	OAc	3	98
5	но-Он	AcO-OAc	2	99
6	ОН	OAc	3	99
7	НО	AcO	3	99
8	НО ОН	AcO OAc	1.5	99
9	ОН СН ₃	OAc CH ₃	2	98
10	н3С	OAc H ₃ C	2	99
11	Н ₃ СОН	H ₃ C-OAc	2	99
12	OH	OAc	2	99
13	ОН	OAc	2	96
14	02N-ОН		2	97
15	С——ОН NO ₂		20	88

 a Reaction conditions: phenol (1 mmol), catalyst (0.01 mmol, 10 mg), Ac₂O (3 equivalents per OH group), CH₃CN (0.5 mL). b GC yield based on the starting alcohol.

Fig. 1 shows the UV–Vis spectrum of this catalyst in which three bands at 456 (Soret band), 585 and 628 nm (Q bands) were observed.

The Lewis acidity of this high-valent V(IV) species was investigated in acetylation of alcohols and phenols with Ac₂O. First, in order to show the effect of OTf⁻ groups on the catalytic activity of [V^{IV}(TPP)(OTf)₂], different vanadium(IV) porphyrins such as [V^{IV}O(TPP)], [V^{IV}(TPP)Cl₂] and [V^{IV}(TPP)(OTf)₂] were used as catalyst in the acetylation of 4-chlorobenzyl alcohol with Ac₂O. The results showed that the catalytic activity of these catalysts is as following: [V^{IV}(TPP)(OTf)₂] (99%) > [V^{IV}(TPP)Cl₂] (55%) > [V^{IV}O(TPP)] (21%). These observations show that introducing of triflate groups on the prophyrin moiety increases the electron-deficiency of V(IV) species which in turns increases the catalytic activity of [V^{IV}(TPP)(OTf)₂] in the acetylation reactions.

Therefore, acetylation of alcohols with Ac_2O was carried out in the presence of catalytic amounts of $[V^{IV}(TPP)(OTf)_2]$ at room tem-



Scheme 3. The proposed mechanism for acetylation of alcohols and phenols with Ac₂O.

Table 3

perature and in CH₃CN as solvent. The results, which are summarized in Table 1, showed that this catalytic system efficiently converted different primary, secondary and tertiary alcohols to their corresponding acetates in 85–99% and short reaction times at room temperature except for bulky alcohols such as anthracene-9-methanol, 1-adamantanol and triphenylmethanol. In these cases, the reactions were carried out at 50 °C. This may due to the higher solubility of these compounds in CH₃CN at 50 °C or may be due to the steric hindrance of the compounds which prevents these compounds to interact with the catalyst. In the case of benzylic alcohols, the nature of substituent, electron-withdrawing or electron-donating, has no significant effect on the yield and reaction time.

Under the same reaction conditions, which was described for acetylation of alcohols, the acetylation of phenols with Ac_2O in the presence of $[V^{IV}(TPP)(OTf)_2]$ at 50 °C was also investigated. As can be seen in Table 2, different phenols were efficiently acetylated by this catalytic system and the corresponding acetates were produced in 88–99%.

The actual mechanism is not clear at present. However, a plausible explanation is that acetic anhydride is first activated by 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).

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 $[V^{IV}(TPP)(OTf)_2]$ with some of those reported in the literature (Table 3). It is clear that the presented method is superior in terms of reaction time, catalyst amount, or product yield.

3.2. Catalyst reusability

The reusability of a catalyst is of great importance from economical and environmental points of view.

The reusability of $[V^{IV}(TPP)(OTf)_2]$ was checked in the acetylation of 4-chlorobenzyl alcohol with acetic anhydride. At the end of each reaction, the solvent was evaporated, Et₂O was added and the catalyst was filtered. Then, the catalyst was used with fresh 4-chlorobenzyl alcohol and Ac₂O. The obtained results showed that after using the catalyst for several times (five consecutive times

Comparison of the results obtained for the acetylation of benzyl alcohol catalyzed by [V^{IV}(TPP)(OTf)₂] with those obtained by the recently reported catalysts.

OH Catalyst OAc						
Entry	Catalyst	Catalyst (mol%)	T (°C)	Time (min)	Yield (%)	Refs.
1	[V ^{IV} (TPP)(OTf) ₂]	0.5	r.t.	0.5	90	_
2	[Sn(TPP)(OTf) ₂]	1	r.t.	1	99	[11]
3	$[Sn(TPP)(BF_4)_2]$	1	r.t.	1	99	[18]
4	I ₂	10	r.t.	1	99	[22]
5	CoCl ₂	0.5	r.t.	240	98	[26]
6	Montmorillonite KSF	20 mg	r.t.	60	90	[27]
7	Zeolite HSZ-360	20 mg	60 °C	60	84	[28]
8	TaCl ₅	10	r.t.	-	77	[31]
9	Cu(OTf) ₂	2	r.t.	30	97	[33]
10	In(OTf) ₃	0.1	r.t.	15	97	[34]
11	BiCl ₃	10	r.t.	35	98	
	Bi(TFA) ₃	5	r.t.	60	96	[36]
	Bi(OTf) ₃	1	r.t.	5	99	
12	RuCl ₃	5	r.t.	10	95	[38]
13	InCl ₃	0.1	r.t.	30	85	[39]
14	Ce(OTf) ₃	1	r.t.	12	98	[40]
15	$Mg(ClO_4)_2$	1	r.t.	15	100	[41]
16	Cp_2ZrCl_2	1	r.t.	600	93	[43]
17	ZrO(OTf) ₂	1	r.t.	0.5	100	[58]

were checked), the corresponding acetate was obtained without any decrease in its yield.

4. Conclusion

Electron-deficient metalloporphyrins have been used as mild Lewis acids. In this report, we demonstrated for the first time that the tetraphenylporphyrinatovanadium(IV) trifuoromethanesulfonate, $[V^{IV}(TPP)(OTf)_2]$, can be considered as an efficient and mild Lewis acid for 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. These points clearly indicate that replacement of Cl⁻ with OTf⁻ converts the [V(TPP)Cl₂] to an electron deficient catalyst, which can acts as a super Lewis acid in organic transformations. In addition, short reaction times and catalyst reusability make this catalytic system as a useful method for acetylation reactions.

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