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Efficient and selective olefination of aldehydes with ethyl diazoacetate catalyzed by high-valent tin(IV) porphyrins

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ABSTRACT

Tetraphenylporphyrinatotin(IV) trifluoromethanesulfonate, [Sn^{IV}(TPP)(OTf)$_2$], and tetraphenylporphyrinatotin(IV) tetrafluoroborate, [Sn^{IV}(TPP)(BF_4)$_2$], were used as efficient catalysts for olefination of aldehydes with EDA in the presence of PPh_3. These high-valent tin porphyrins catalyzed olefination of aldehydes in high yields and short reaction times at room temperature. The reaction rate depended on the nature of substituents on aldehyde; electron poor aldehydes reacted faster than electron rich aldehydes. The olefination of aldehydes indicated that the reactions are very selective and all products are *trans*-isomer. Both catalysts were reused four consecutive times without loss of their catalytic activity.

1. Introduction

Constructive carbon–carbon double bond of carbonyl compounds is one of the most important reactions in organic synthesis, especially in the areas of polymer and natural products synthesis [1,2]. Generally, the used methods were included the Wittig reaction and its modified reactions, the Peterson reaction, and the Julia reaction [3]. In the past two decades a variety of organometallic compounds have been used for this reaction and some of them was done successfully [4,5]. One of the other procedures for olefin formation is the reaction of carbonyl compounds with diazo compounds in the presence of a reducing agent such as triphenylphosphine and trialkylstibines [6]. In contrast to the tradition Wittig method, the metal-mediated syntheses are carried out in one simple reaction. However, some drawbacks such as need high reaction temperature especially when diazo compounds are used to increase safety risks, using high catalyst amount, high dependence of stereoselectivites to the nature of aldehyde and low yields. In 1998, Fujimura reported one-pot olefination of carbonyl compounds by a ruthenium complex as catalyst. In this system, for some aldehydes, the yields were not particularly high [7]. Several transition metal complexes including Mo, Re, Ru, Rh, Fe and Co, have been used as catalysts for olefination of aldehydes with diazo compounds [8–16].

Electron-deficient metalloporphyrins have been used as mild Lewis acids catalysts [17–19]. Suda group has 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 [20–25].

Recently, we have reported the use of tin(IV)tetraphenylporphyrinato perchlorate [26,27], tin(IV)tetraphenylporphyrinato trifluoromethanesulfonate [28–32], tin(IV)tetraphenylporphyrinato tetrafluoroborate [33–35], *tetrakis*(*p*-aminophenyl)porphyrinatotin(IV) trifluoromethanesulfonate, [Sn^{IV}(TNH_2PP)(OTf)$_2$] supported on polystyrene [36–38] and vanadium(IV)tetraphenylporphyrinato trifluoromethanesulfonate [39–42] in organic transformations.

In this paper, we wish to report the application of high-valent [Sn^{IV}(TPP)(OTf)$_2$] and [Sn^{IV}(TPP)(BF_4)$_2$] catalysts for efficient and selective olefination of a wide variety of aldehydes with ethyl diazoacetate (EDA) in the presence of triphenylphosphine at room temperature (Scheme 1).

2. Experimental

Chemicals were purchased from Merck or Fluka chemical companies. All reactions were performed under nitrogen atmosphere

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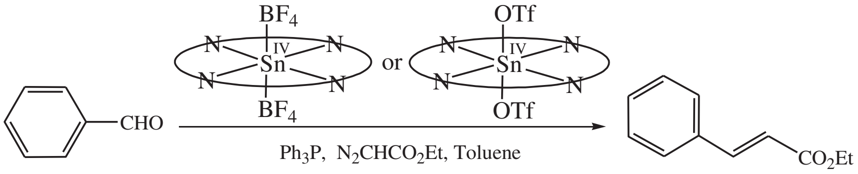


Scheme 1. Olefination of aldehydes with EDA catalyzed by [Sn^{IV}(TPP)(OTf)$_2$] or [Sn^{IV}(TPP)(BF_4)$_2$].

using a glove box equipped with a M040H Dri-Train gas purification system. Toluene and THF were dried before use. NMR spectra were recorded on Bruker-Avance 400 MHz spectrometer in $CDCl_3$ as solvent. 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 20M columns and *n*-decane was used as internal standard. The tetraphenylporphyrin was prepared and metallated according to the literature [43]. The catalysts, [Sn^{IV}(TPP)(OTf)$_2$] [31] and [Sn^{IV}(TPP)(BF_4)$_2$] [32], were prepared as reported previously.

2.1. General procedure for olefination of aldehydes

A mixture of aldehyde (1 mmol), [Sn^{IV}(TPP)(OTf)$_2$] (0.01 mmol) or [Sn^{IV}(TPP)(BF_4)$_2$] (0.02 mmol) and PPh_3 (2 mmol) in toluene (1 mL) was prepared. Then a solution of ethyl diazoacetate (1.5 mmol mL) in toluene (0.5 mL) was added drop-wise to this solution and stirred at room temperature for appropriate time. The progress of the reaction was monitored by GC or TLC. After completion of the reaction, the solvent was evaporated, *n*-hexane (10 mL) was added and the catalyst was filtered. The filtrates were concentrated under reduced pressure to afford the crude product, which was confirmed by ^{1}H NMR and ^{13}C NMR spectral data.

2.2. Spectral data

2.2.1. (E)-Ethyl cinnamate (entry 1, Table 2)

^{1}H NMR ($CDCl_3$, 400 MHz): $\delta = 7.70$ (d, $J = 16.0$ Hz, 1H), 7.53–7.51 (m, 2H), 7.39–7.36 (m, 3H), 6.45 (d, $J = 16.0$ Hz, 1H), 4.27 (q, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 166.8$, 144.5, 137.3, 134.4, 130.1, 128.6, 118.4, 60.5, 14.5 ppm.

2.2.2. (E)-Ethyl 3-(4-chlorophenyl)acrylate (entry 2, Table 2)

^{1}H NMR ($CDCl_3$, 400 MHz): $\delta = 7.64$ (d, $J = 16.0$ Hz, 1H), 7.46 (d, $J = 8.7$ Hz, 2H), 7.36 (d, $J = 8.7$ Hz, 2H), 6.41 (d, $J = 16.0$ Hz, 1H), 4.27 (q, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 160.6$, 142.6, 133.6, 133.4, 128.8, 127.8, 180.8, 61.5, 14.6 ppm.

2.2.3. (E)-Ethyl 3-(4-bromophenyl)acrylate (entry 3, Table 2)

^{1}H NMR ($CDCl_3$, 400 MHz): $\delta = 7.63$ (d, $J = 16.2$ Hz, 1H), 7.53 (d, $J = 8.4$ Hz, 2H), 7.40 (d, $J = 8.4$ Hz, 2H), 6.44 (d, $J = 16.2$ Hz, 1H), 4.28 (q, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 160.6$, 144.1, 134.3, 131.7, 128.6, 122.4, 119.1, 61.5, 14.35 ppm.

2.2.4. (E)-Ethyl 3-p-tolylacrylate (entry 4, Table 2)

^{1}HNMR ($CDCl_3$, 400 MHz): $\delta = 7.66$ (d, $J = 15.9$ Hz, 1H), 7.42 (d, $J = 8.1$ Hz, 2H), 7.18 (d, $J = 8.1$ Hz, 2H), 6.39 (d, $J = 15.9$ Hz, 1H), 4.25 (q, $J = 7.2$ Hz, 2H), 2.36 (s, 3H), 1.33 (t, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 166.1$, 142.7, 137.7, 132.6, 129.1, 126.3, 120.4, 61.4, 21.1, 14.3 ppm.

2.2.5. (E)-Ethyl 3-(4-nitrophenyl)acrylate (entry 5, Table 2)

^{1}HNMR ($CDCl_3$, 400 MHz): $\delta = 8.26$ (d, $J = 8.0$ Hz, 2H), 7.72 (d, $J = 18.0$ Hz, 1H), 7.69 (d, $J = 8.0$ Hz, 2H), 6.57 (d, $J = 18.0$ Hz, 1H), 4.31 (q, $J = 7.2$ Hz, 2H), 1.37 (t, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 166.0$, 148.4, 141.6, 140.6, 128.6, 124.1, 122.6, 61.0, 14.3 ppm.

2.2.6. (E)-Ethyl 3-(4-methoxyphenyl)acrylate (entry 6, Table 2)

^{1}H NMR ($CDCl_3$, 400 MHz): $\delta = 7.64$ (d, $J = 15.6$ Hz, 1H), 7.48 (d, $J = 8.7$ Hz, 2H), 6.90 (d, $J = 8.7$ Hz, 2H), 6.31 (d, $J = 15.6$ Hz, 1H), 4.25 (q, $J = 7.2$ Hz, 2H), 3.83 (s, 3H), 1.33 (t, $J = 7.2$ Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): $\delta = 160.1$, 159.6, 143.2, 127.6, 127.4, 121.9, 114.1,61.9, 55.1, 14.3 ppm.

3. Results and discussion

3.1. Olefination of aldehydes with ethyl diazoacetate (EDA) catalyzed by [Sn^{IV}(TPP)(OTf)$_2$]

First, in order to indicate the effect of OTf groups on the electron deficiency of tin(IV) porphyrin, the olefination of benzaldehyde with EDA in present of PPh_3 was carried out with 1 mol% of [Sn^{IV}(TPP)(OTf)$_2$] or [Sn^{IV}(TPP)Cl_2] catalyst at room temperature. The results showed that the amount of the corresponding olefin in the presence of [Sn^{IV}(TPP)Cl_2] was 30% after 4 h, while in the presence of [Sn^{IV}(TPP)(OTf)$_2$] after 1 h, the corresponding olefin was 97%. Then, to obtain optimal reaction conditions, the amount of catalyst and PPh_3 was optimized in the olefination of benzaldehyde (Table 1). These results showed that the highest yield of ethyl cinnamate was produced in the presence of 1 mol% of [Sn^{IV}(TPP)(OTf)$_2$] in toluene (Table 1, entry 3). Increasing the amount of catalyst did not affect the yield but decreasing it, reduced the yield. In the case of PPh_3, when the amount of PPh_3 increased from 1 mmol to 2 mmol, an increase in the yield from 56% to 97% was observed, but with 3 mmol of PPh_3, the yield dropped to 79%. It seems that higher amounts of PPh_3 can deactivate the [Sn^{IV}(TPP)(OTf)$_2$]. In addition, when the same reaction was performed in THF as solvent, the yield decreased to 44% (Table 1, entry 7). A decrease in yield was dramatically observed when the reaction was carried out under air; therefore, all reactions were carried out under N_2 atmosphere.

Table 1
Optimization of reaction conditions in the olefination of aldehyde with tin porphyrins.[a]

Entry	PPh_3 (mmol)	[Sn^{IV}(TPP)(OTf)$_2$] after 60 min		[Sn^{IV}(TPP)(BF_4)$_2$] after 100 min	
		Catalyst amount (mmol)	Yield (%)[b]	Catalyst amount (mmol)	Yield (%)[b]
1	2	0.005	74	0.005	58
2	2	0.007	85	0.01	77
3	2	0.01	97	0.02	98
4	2	0.02	97	0.03	98
5	1	0.01	56	0.02	48
6	3	0.01	79	0.02	64
7[c]	2	0.01	44	0.02	49

[a] Reaction conditions: benzaldehyde (1 mmol), EDA (1.5 mmol), toluene (1.5 mL).
[b] GC yield.
[c] The reaction was performed in THF.

The optimized reaction conditions, which obtained for olefination of aldehydes, were aldehyde, EDA, PPh_3 and catalyst in a molar ratio of 100:150:200:1, using toluene as solvent. Under the optimized reaction conditions, different aldehydes were converted to their corresponding olefins in excellent yields and short reaction times (Table 2). As can be seen, electron-rich benzaldehydes such as 4-methyl and 4-methoxy benzaldehydes require longer reaction times. On the other hand, electron-poor benzaldehydes, such as 4-chloro, 4-bromo and 4-nitrobenzaldehydes produced the corresponding olefins in shorter times.

The *E*/*Z* ratio of olefins after their purification was determined by their ^1H NMR spectra. The chemical shift of alkenyl hydrogen groups are present in 6.31–6.57 ppm and 7.63–7.72 ppm and the coupling constant of about 16 Hz (18 Hz for 4-nitro derivative) in ^1H NMR spectra indicated that all products were *trans*-isomer [44] and no *cis* isomer was engendered in the presence of electron-deficient of tin(IV) porphyrin.

The Sn=C double bond is now well established, some of them structurally characterized and the Sn=C bond length and the environment of the respective tin atoms. An example of these tin-carbene complexes is formed by the reaction of imidazole-2-yiledene with SnR_2Cl_2 in which a square pyramidal or a trigonal bipyramidal complex is obtained [45–48]. Another example of these complexes is produced by the reaction of the stannylene (R_2Sn:) acting as Lewis acids, with the isocyanide (:C=N–Ar), acting as a Lewis base [49,50]. The third class of tin-carbene complexes is cyclopropenylidene complexes of divalent tin [51].

A plausible mechanism is shown in Scheme 2. Based on this mechanism, tin(IV) porphyrin interacts with diazo compound to affords the tin-carbene intermediate (**A**) (this intermediate can be formed according to the above mentioned explanations) and releases the nitrogen gas. Consecutively, the intermediate (**A**) reacts with PPh_3 and produces intermediate (**B**) (ylide form) and upon a Wittig reaction, the corresponding olefin is produced from ylide and aldehyde. No olefin was produced in the absence of PPh_3 or catalyst and these are good reasons for the proposed mechanism [2]. It is important to note that these results are in accordance with the previously reported results in which stated that the stereoselectivity of the Wittig reaction depends strongly to the structure of the ylide; unstabilized ylides give *Z*-alkenes while stabilized ylides produce *E*-alkenes [52]. In these reactions, the produced yield is stabilized by ester group and therefore results in *E*-alkenes.

3.2. Olefination of aldehydes with ethyl diazoacetate (EDA) catalyzed by $[Sn^{IV}(TPP)(BF_4)_2]$

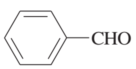
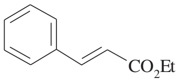
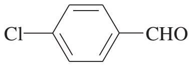
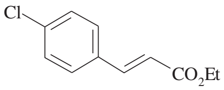
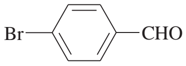
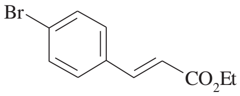
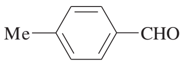
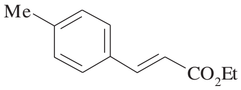
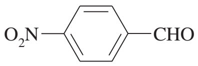
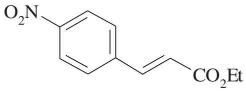
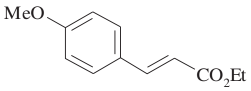
Electron-deficient tin(IV) porphyrin, $[Sn^{IV}(TPP)(BF_4)_2]$, was also used as catalyst in the olefination of aldehydes with EDA in presence of PPh_3. The optimized conditions, which obtained for olefination of benzaldehyde, were benzaldehyde, EDA, PPh_3 and catalyst in a molar ratio of 100:150:200:2 at room temperature (Table 1). Under these conditions, different benzaldehydes including electron-rich and electron-poor ones were converted to their corresponding olefins in excellent yields (Table 2). In the case of $[Sn^{IV}(TPP)(BF_4)_2]$, electron-poor benzaldehydes were converted to their corresponding olefins in shorter times too.

The evaluation of the results in Table 2 indicates that $[Sn^{IV}(TPP)(OTf)_2]$ is more reactive than $[Sn^{IV}(TPP)(BF_4)_2]$ and the higher TOFs were observed for tin(IV) trifluoromethanesulfonate in comparison with tin(IV) tetrafluoroborate. This can be attributed to the higher electron deficiency of $[Sn^{IV}(TPP)(OTf)_2]$ compared to $[Sn^{IV}(TPP)(BF_4)_2]$.

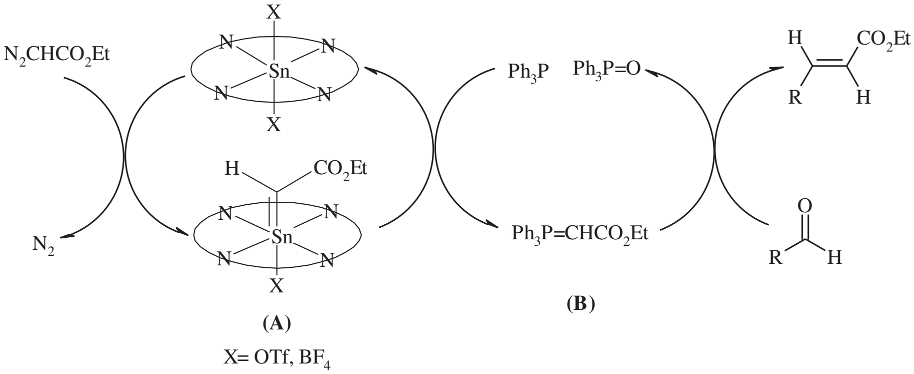
3.3. Catalyst reutilization

The catalyst reusability was investigated in the reaction of benzaldehyde, EDA and PPh_3 in the presence of both tin(IV)

Table 2
Olefination of aldehydes with EDA catalyzed by tin(IV) porphyrins at room temperature.[a]

Entry	Aldehyde	Olefin	$[Sn^{IV}(TPP)(OTf)_2]$ (1 mol%)			$[Sn^{IV}(TPP)(BF_4)_2]$ (2 mol%)		
			Time (min)	Yield (%)[b]	TOF (h^{-1})	Time (min)	Yield (%)[b]	TOF (h^{-1})
1			60	97	97	100	98	29
2			50	97	116	70	97	42
3			50	97	116	70	97	42
4			80	94	70	115	93	24
5			30	99	198	30	98	98
6			120	95	47	140	92	20

[a] Reaction conditions: aldehyde (1 mmol), EDA (1.5 mmol), PPh_3 (2 mmol), toluene (1.5 mL).
[b] GC yield.



Scheme 2. Proposed mechanism for olefination of aldehydes with EDA catalyzed by tin(IV) porphyrins.

porphyrins as catalyst. At the end of each reaction, the solvent was evaporated, *n*-hexane was added, the catalyst was filtered and washed with *n*-hexane. The results showed that both catalysts were reused four consecutive times without loss of their catalytic activity.

4. Conclusion

In this paper, another application of electron-deficient tin(IV) tetraphenylporphyrinato trifluoromethanesulfonate, $[Sn^{IV}(TPP)(OTf)_2]$, and tin(IV)tetraphenylporphyrinato tetrafluoroborate, $[Sn^{IV}(TPP)(BF_4)_2]$, which are stable Sn(IV) compounds was investigated. Selective olefination of different aldehydes with EDA and PPh_3 in the presence of these catalysts was carried out; the pure *trans*-isomer was obtained in excellent yield and short reaction time under mild conditions (room temperature). The both catalyst were reused several times without loss of their catalytic activity.

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