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# SnCl<sub>4</sub>–Zn: a novel reductive system for deoxygenative coupling of aliphatic, aromatic, chalcone epoxide, and indanone carbonyl compounds to olefins

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# Introduction

In the carbon-carbon bonds formation, the McMurry reaction plays an important role to obtain homo- and cross-coupled alkenes from aliphatic and aromatic aldehydes and ketones in the presence of in situ generated low valent titanium (LVT) reagents at reflux temperature.<sup>1</sup> However, the reaction gave a moderate yield of cross-coupled product due to homo-coupled product formation. To enhance the yield of the cross-coupled products under mild reaction conditions, the different reagents are explored for the McMurry reaction. For example, magnesium-mercury couple, NbCl<sub>5</sub>/NaAlH<sub>4</sub><sup>2</sup> zinc-copper couple,<sup>3</sup> LiAlH<sub>4</sub><sup>4</sup> dicyclopentadienyl titaniumdichloride,<sup>5</sup> trimethyl aluminum.<sup>6</sup> Although, these procedures have drawbacks like costly reagents, low yield, the longer reaction time, and/or less functional group tolerance. In recent years, tin tetrahalides  $(SnX_4, X = Cl, Br)$  have been widely used as Lewis acids in numerous organic syntheses.<sup>7</sup> In many cases, they have been reported as efficient catalysts and easy to handle as compared to other metal halides such as  $TiX_4 AIX_3$ ,  $ZnX_2$ , and  $ZrX_4$ .<sup>8</sup>

Generally, the metal alloys are prepared by the Rieke's method and used as a reductive deoxygenating agent for the carbonyl compounds in the organic synthesis.<sup>9</sup> For example, in the McMurry reaction Ti(IV) reagent reduced to Ti(II) in the presence of Zn as

# ABSTRACT

SnCl<sub>4</sub>–Zn complex provided a novel reductive system in the deoxygenative cross-coupling of aliphatic, aromatic, chalcone epoxide and indanone carbonyl compounds to olefins in high yield (55–86%) at reflux temperature in THF. The advantage of this reagent is inexpensive, short reaction time, and high yield compared to the reagents used in the McMurry cross-coupling reaction.

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reducing agent in THF. Thus, a complex of TiCl<sub>4</sub>–Zn–(THF)<sub>2</sub> generated in situ known as low valent titanium (LVT) has been used in the reductive deoxygenating coupling of aldehydes and/or ketones to titanium pinacolates, followed by the removal of TiO<sub>2</sub> gave the olefins, but the exact reaction mechanism is still debatable.<sup>10,11</sup> Likewise, we expect that it might be taking place in SnCl<sub>4</sub>–Zn (used in 1:2 ratio) and THF to form a complex of SnCl<sub>4</sub>–Zn–(THF)<sub>2</sub> in situ low valent tin (LVT) as Sn(II) which converted carbonyl oxygen to tin pinacolate, followed by the removal of SnO<sub>2</sub> to give the olefins.

Therefore, in continuation of our interest to develop new methods in the organic synthesis and the acid catalysis reactions<sup>12</sup> herein, we report a novel and efficient reagent, SnCl<sub>4</sub>–Zn system for the McMurry cross-coupling reaction in the conversion of aliphatic and aromatic ketones, aldehydes, chalcone epoxides, and indanones into olefins and also useful in the synthesis of molecules like tamoxifen analogs in good yield within 4–4.5 h at reflux temperature.

# **Results and discussion**

Initially, we optimized the cross-coupling reaction conditions in the reaction of chalcone epoxide **1** and propiophenone **2** used in 1:1.5 ratio and varying the equivalents of SnCl<sub>4</sub>–Zn (used in 1:2 ratio). We obtained the cross-coupled product in 50% and 55% yields in 4 h using 1 and 2 equiv of SnCl<sub>4</sub>–Zn, respectively (Table 1, entries 1 & 2). Then, SnCl<sub>4</sub>–Zn was used in 3 equiv, the yield was serendipitously improved up to 75% in 4 h (Table 1, entry 3).





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Optimized condition for the cross-coupling reaction using different equivalents of  ${\rm SnCl}_4{\rm -Zn}$ 



Entry	SnCl <sub>4</sub> –Zn	Time (h)	Yield <sup>a</sup> (%)
1	SnCl <sub>4</sub> -Zn (1 equiv)	4	50
2	SnCl <sub>4</sub> -Zn (2 equiv)	4	55
3	SnCl <sub>4</sub> –Zn (3 equiv)	4	75
4	SnCl <sub>4</sub> -Zn (3.5 equiv)	4	60
5	SnCl <sub>4</sub> -Zn (4 equiv)	4	35

Bold entries are indicated the most favored optimized reaction condition.

<sup>a</sup> Isolated yield of cross-product.

Further, an increase in  $SnCl_4$ -Zn equivalent decreased the yields of the cross-coupled product and increased the homo-coupled products (Table 1, entries 4 & 5).

Similarly, we optimized the reaction time, by monitoring the progress of reaction between 1 h and 3 h at reflux temperature, which gave maximum 60% yields (Table 2, entries 1–3). Further, an increase in the reaction time to 4 h gave a very good yield up to 75% (Table 2, entry 4). However, a further increase in the reaction time to 5 h, decreased the product yield (35%) (Table 2, entry 5). After separating the homo-coupled (minor) products, we also determined the ratio of *E* and *Z* isomers in the cross-coupled product, where *E*-isomer and *Z*-isomer were found as major and minor products, respectively. Due to the close  $R_{f}$ -values of these isomers, we were unable to separate the *Z*-isomers using the column chromatography. However, the yields of *Z*-isomers (2–5%) were confirmed by <sup>1</sup>H NMR and GC spectral analysis.<sup>13</sup>

Under optimal reaction conditions, the efficiency of different McMurry reagents was compared (Table 2). Aluminum and Indium complexes gave poor products yield (20%) at reflux in 14 h (Table 2, entries 1, 2). However, the titanium complex (TiCl<sub>4</sub>–Zn–THF) gave the yield (40%) at reflux temperature in 6 h (Table 2, entry 3), while the tin complex (SnCl<sub>4</sub>–Zn–THF) gave the optimal yield (75%) at reflux temperature within 4 h (Table 2, entry 4). Other reagents gave poor yield (traces to 20–25%) in acetonitrile solvent (Table 2, entries 5, 6, 7) (Table 3).

#### Table 2

Optimized condition for cross-coupling reaction by varying reaction times



SnCl <sub>4</sub> –Zn	Time (h)	Yield <sup>a</sup> (%)
SnCl <sub>4</sub> -Zn (3equiv)	1	20
SnCl <sub>4</sub> –Zn (3 equiv)	2	35
SnCl <sub>4</sub> –Zn (3 equiv)	3	60
SnCl <sub>4</sub> –Zn (3 equiv)	4	75
SnCl <sub>4</sub> –Zn (3 equiv)	5	65
	SnCl <sub>4</sub> -Zn   SnCl <sub>4</sub> -Zn (3equiv)   SnCl <sub>4</sub> -Zn (3 equiv)   SnCl <sub>4</sub> -Zn (3 equiv)   SnCl <sub>4</sub> -Zn (3 equiv)   SnCl <sub>4</sub> -Zn (3 equiv)	SnCl <sub>4</sub> -Zn Time (h)   SnCl <sub>4</sub> -Zn (3equiv) 1   SnCl <sub>4</sub> -Zn (3 equiv) 2   SnCl <sub>4</sub> -Zn (3 equiv) 3   SnCl <sub>4</sub> -Zn (3 equiv) 4   SnCl <sub>4</sub> -Zn (3 equiv) 5

Bold entries are indicated the most favored optimized reaction condition. <sup>a</sup> Isolated yield of cross-product.

#### Table 3

Comparison of the McMurry reagents in cross-coupling of chalcone epoxide and propiophenone



Entry	McMurry reagents	Time (h)	Yield <sup>a</sup> (%)
1	AlCl <sub>3</sub> -Zn (3 equiv)	14	20
2	InCl <sub>3</sub> –Zn (3 equiv)	14	20
3	TiCl <sub>4</sub> -Zn (3 equiv)	6	60
4	SnCl <sub>4</sub> –Zn (3 equiv)	4	75
5	AlCl <sub>3</sub> –Zn (3 equiv)	14	Trace
6	InCl <sub>3</sub> –Zn (3 equiv)	14	Trace
7	TiCl <sub>4</sub> –Zn (3 equiv)	14	20

Bold entries are indicated the most favored optimized reaction condition.

Isolated yield of cross-product at 64-66 °C.



**Scheme 1.** SnCl<sub>4</sub>–Zn mediated deoxygenative cross-coupling of aromatic ketone and aldehyde with acetone under optimized conditions.

In the case of aromatic aldehydes or aromatic ketones **6a–6c** with acetone (1:1.5 ratio) and SnCl<sub>4</sub>–Zn (1:2 ratio) gave the corresponding cross-coupled olefin **2ab–2ad** in excellent yield 80–85% along with minor homo-coupled product **3aa–3cc** and **4aa** in yield 8–10% at reflux temperature within 1 h (Scheme 1). The reaction of aromatic aldehydes or aromatic ketones **7a–7g** with SnCl<sub>4</sub>–Zn gave the homo-coupled olefins **8a–8g** in excellent yield 70–86% at reflux temperature within 1 h. Products **8a–8g** were characterized on the basis of their spectral data IR, <sup>1</sup>H NMR, and melting point and comparing it with those of authentic samples (Scheme 2).

R F	R <sup>'</sup> <u>SnC</u> 6	SnCl <sub>4</sub> -Zn-THF 64-66 <sup>0</sup> C		K'
7a-7g			IX.	8a-8g
Entry	Product	R	R′	Yield(%) <sup>a</sup>
1	8a	Me	Me	85
2	8b	Ph	Ph	82
3	8c	Ph	Н	85
4	8d	$4\text{-}FC_6H_4$	Н	83
5	8e	$4\text{-}Cl\ C_6H_4$	Н	80
6	8f	$4\text{-}Me\ C_6H_4$	Н	70
7	8g	$4\text{-}OMe\ C_6H_4$	Η	86

<sup>a</sup>Isolated product yield

Scheme 2. Deoxygenation of simple carbonyl compound to olefin by using SnCl<sub>4</sub>-Zn.



Scheme 3. Under optimized conditions, SnCl<sub>4</sub>-Zn mediated deoxygenative cross-coupling of aromatic ketone with propiophenone.

SnCl<sub>4</sub>-Zn mediated deoxygenative cross-coupling of chalcone epoxides with propiophenone



Entry	4a-4j	5b	Cross-coupled products ( <b>3ab–3ak</b> )	Homo-coupled products		Time (h)	Yield <sup>a</sup> (%)
				4aa–4jj	5bb		
1	CI CI CI			8	10	4	66
2	CI C		CI 3ac OMe OMe	9	8	4	60
3	Br C Me	C C	Br 3ad Me	10	9	4	64

(continued on next page)

### Table 4 (continued)

Entry	4a-4j	5b	Cross-coupled products (3ab-3ak)	Homo-coupled products		Time (h)	Yield <sup>a</sup> (%)
				4aa-4jj	5bb		
4	CI CI Me		CI 3ae Me	8	8	4.5	58
5	Br O Br	°	Br O Br	10	10	4.5	55
6		Č,	o 3ag	9	8	4	75
7	F C C			10	9	4	72
8	C C C C C C C C C C C C C C C C C C C		3ai F	9	9	4	70
9	F C C F	Č,		10	10	4.5	68
10	CI CI			10	9	4.5	67

<sup>a</sup> Isolated yield of cross-product at 64–66 °C.

Further, the reaction of aromatic aldehyde or aromatic ketone **9a–9c** with propiophenone **10** (1:1.5 ratio) and SnCl<sub>4</sub>–Zn (1:2 ratio) gave the cross-coupled olefins **11ab**, **11ac**, and **11ad** in 80, 77, and 70% yields respectively along with homo-coupled products **9aa–9cc** and **10aa** in 5–8% yields at reflux temperature in 2 h (Scheme 3).

The tin reagent (SnCl<sub>4</sub>–Zn) was further applied as an alternate reagent in the McMurry reaction for the synthesis of various classes of carbonyl compounds. For example, the cross-coupled products **3ab–3ak** were obtained in good yield (55–75%) from different chalcone epoxides **4a–4j** and propiophenone **5b** under optimized

reaction conditions (Table 4). Along with major cross-coupled product some minor homo-coupled products **4aa–4jj** and **5bb** were obtained in 8–10% yields. These homo-coupled products were isolated and the yields of product were written as isolated yields. The products were characterized on the basis of their spectral data. In <sup>1</sup>H NMR spectra, the characteristic doublet signal appeared for **3ab–3ak** in the range of  $\delta$  3.85–3.20 ppm, whereas for the compounds **4a–4j** was obtained in the range of  $\delta$  4.30–4.20 ppm, the characteristic quartet and triplet signal of –CH<sub>2</sub>CH<sub>3</sub> appeared in between  $\delta$  0.80 and 2.5 ppm, indicates the cross-coupling reactions (experimental section).

Synthesis of tamoxifen analogs (*E* isomers) of indanone using novel SnCl<sub>4</sub>–Zn reagent



$R^{1}=$	Η,	$R^{2}=$	С
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<sup>a</sup> Isolated yield of cross-product at 64–66 °C.

The tin reagent  $(SnCl_4-Zn)$  was also successfully used in the synthesis of tamoxifen and tamoxifen analogs (Table 5). Products **3al-3ao** were synthesized in good yield 58–72% at reflux temperature in 4–5 h using  $SnCl_4/Zn$  (1:2 equiv) and the products were confirmed on the basis of their spectral data (Supporting information). For example, product **3al**, the <sup>1</sup>H NMR spectra showed the

characteristic double doublet peaks at  $\delta$  4.70–4.60 ppm for *CH–CH–* which shifted from  $\delta$  5.30 to 5.23 ppm in the indanone molecule and the characteristic quartet and triplet peaks of –*CH*<sub>2</sub>*CH*<sub>3</sub> protons appeared at  $\delta$  0.90–2.30 ppm, indicated the cross-coupling product.<sup>14</sup> The structures of all other compounds were further confirmed by IR and HRMS.

Determination of E and Z tamoxifen analogs by using novel SnCl<sub>4</sub>-Zn reagent





<sup>a</sup> Isolated yield of *E* and *Z* isomers.

Compounds 4ab-4ac and 5ab-5ac were synthesized as a mixture of *E* and *Z* isomers which can be separated by using column chromatography and by comparing their spectral values in the literature. We observed that the *E* isomer is the major isomer with 52–55% yields and Z isomer is the minor product with 8–10% yields in 5 h, using SnCl<sub>4</sub>/Zn (1:2 equiv) in indanone and propiophenone (1:1.5 equiv). The  ${}^{1}$ H NMR chemical shift ( $\delta$ ) 1.0–1.3 ppm for -CH<sub>3</sub> and 2.0-2.3 ppm for -CH<sub>2</sub> indicated the *E* isomer of products **4ab–4ac** and  $\delta$  0.6–0.7 ppm for –CH<sub>3</sub> and 1.6–1.9 ppm for  $-CH_2$  gave the **Z** isomer for products **5ab**-**5ac**. Similarly, <sup>13</sup>C NMR chemical shift ( $\delta$ ) 13–15 ppm for –CH<sub>3</sub> and 27–28 ppm for -CH<sub>2</sub> indicated the **E** isomer for products **4ab**-**4ac** and  $\delta$  10-12 ppm for  $-CH_3$  and 23–25 ppm for  $-CH_2$  gave the Z isomer in 5ab-5ac.<sup>15</sup> Similarly, the products 3ab-3ao were characterized as *E*-isomer. The <sup>1</sup>H NMR chemical shift ( $\delta$ ) values of –CH<sub>2</sub>CH<sub>3</sub> in products 3ab-3ao are matched with the 4ab-4ac (E-isomer) and not with 5ab-5ac (Z-isomer). We were unable to isolate the Z-isomer due to close R<sub>f</sub> values. However, the yields of Z-isomers (2-5%) were further confirmed by GC analysis (Table 6).

# Conclusion

In conclusion, we have reported a novel, efficient, and economical tin-reagent for the reductive cross-coupling reaction of carbonyl compounds under the McMurry coupling with aliphatic and aromatic ketones, aldehydes, chalcone epoxides, and indanones in high yield with minor homo-coupling products. To our knowledge, no studies exploiting this reagent for such conversion have previously been reported.

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# Supplementary data

Supplementary data (experimental procedures, characterization data and <sup>1</sup>H, <sup>13</sup>C NMR and HRMS spectra) associated with this article can be found, in the online version, at http://dx.doi.org/10. 1016/j.tetlet.2015.01.194.

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- 15. General procedure for the synthesis compounds 2ab-2ad/8a-8g/11ab-11ad/ 3ab-3ao/4ab-4ac & 5ab-5ac: Under N₂ atmosphere, a three neck flask equipped with magnetic stirrer was charged with Zn-powder (1.5gm, 12 mmol) and 50 mL THF solvent. The mixture was cooled at 0 °C and SnCl<sub>4</sub> (2.3 mL, 6 mmol) was added drop wise at 0 °C. The suspension was warmed to

room temperature and stirred for 15 min and then heated at 64-66 °C for 1.5 h. The solution of aromatic aldehyde or ketone **6a-6c**/chalcone epoxide **4a-4j**/ indanone and propiophenone (1:1.5 molar ratio, 2 mmol) dissolved in THF (30 mL) was added slowly at same temperature. TLC monitoring, the reaction mixture was stirred at same temperature until the carbonyl compound was consumed in the reaction. Then, the reaction mixture was cooled and quenched with 10% aqueous NaHCO3 solution and extracted in EtOAc. The organic layer was washed with brine solution, dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo. The crude material was purified by column chromatography to give the desired products 2ab-2ad/8a-8g/11ab-11ad/ 3ab-3ao/4ab-4ac & 5ab-5ac in 55-86% yields. 1(dimethylamino)ethoxy) phenyl)propylidene)-3-phenyl-2,3-dihydro-1H-inden-2-ol (3am). Light yellow semi solid; yield: (268 mg, 65%); IR v<sub>max</sub> (KBr, cm<sup>-1</sup>): 3452 (OH str), 2963 (aromatic C-H str), 1599 (aromatic, C=C str), 1451, 1419, 1262, 1021, 933, 868, 799 and 704; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ (ppm): 7.92 (d, J = 8 Hz, 2H), 7.83 (d, J = 7.5 Hz, 2H), 7.60 (d, J = 7 Hz, 1H), 7.49-7.45 (m, 2H), 7.41 (dd, J = 7.5, 2.5 Hz, 2H), 7.27-7.25 (m, 2H), 6.92 (d, J = 7.5 Hz, 2H) 4.73 (d, J = 3.5 Hz, 1H), 4.62 (d, J = 3.5 Hz, 1H), 4.15 (t, J = 1.5 Hz, 2H), 2.90 (s, 6H), 2.61 (t, J = 1.5 Hz, 2H), 2.25  $(q, J = 8.0, 2.0 \text{ Hz}, 2\text{H}), 1.18 (t, J = 7 \text{ Hz}, 3\text{H}), 3.60 (s, br, D_2O exchangeable, 1\text{H});$ C NMR (CDCl<sub>3</sub>, 125 MHz) δ (ppm): 160.25, 157.05, 137.76, 132.57, 132.42, 131.77, 130.65, 130.49, 130.33, 129.65, 128.97, 128.68, 128.66, 127.05, 116.56, 116.32, 114.65, 71.65, 67.73, 61.35, 52.65, 48.35, 28.27, 14.03; MS (EI, 70 eV): *m*/*z* = 413 [M]<sup>+</sup> for C<sub>28</sub>H<sub>31</sub>NO<sub>2</sub>; HRMS (ES-TOF) calcd for C<sub>28</sub>H<sub>31</sub>NO<sub>2</sub> 413.2355, found 413.2354. 2-(4-chlorophenyl)-3-(1-(4-chlorophenyl)-2-phenylbut-1-en-1yl) oxirane (3ab). Light brown semi solid; yield: (260 mg, 66%); IR v<sub>max</sub> (KBr, cm<sup>-1</sup>): 2937, 2878 (aromatic C–H str), 1588 (aromatic, C=C str), 1268, 1090, 868, 735 <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ (ppm): 8.18-8.06 (m, 3H), 7.87(t, J = 8.0 Hz, 2H), 7.48 (dd, J = 1.5, 8.5 Hz, 2H), 7.39 (d, J = 8.5 Hz, 2H), 7.30 (m, 2H), (q, J = 2.5, 8.0 Hz, 2H), 1.07 (t, J = 7.0 Hz, 3H);<sup>13</sup>C-(CDCl<sub>3</sub>, 125 MHz)  $\delta$ (ppm):144.35, 143.27, 137.95, 132.55, 132.49, 131.65, 130.49, 130.47, 130.05, 129.65, 128.97, 128.77, 128.66, 127.66, 67.72, 61.05, 27.05, 12.95;MS (EI, 70 eV): m/z (%) = 394[M]<sup>+</sup>, for C<sub>24</sub>H<sub>20</sub>Cl<sub>2</sub>O, 396[M+2H]<sup>+</sup>; HRMS (ES-TOF) calcd for C<sub>24</sub>H<sub>20</sub>Cl<sub>2</sub>O 394.0891, found 394.0893.